Book of Abstracts

2009 E-MRS Fall Meeting & Exhibit

Book of Abstracts: 2009 E-MRS Fall Meeting & Exhibit

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Table of Contents

Welcome 1
- Organisers 2
- Sponsors 2

Programme 5

Plenary Session, Symposia and Exhibit 7
- Plenary Session 9
- Symposium A 11
- Symposium C 37
- Symposium D 75
- Symposium E 97
- Symposium F 117
- Symposium G 135
- Symposium H 151
- Symposium I 187
- Exhibition 231

Satellite events 233
- Carbon Dioxide: a new material for energy storage and a challenge for European industry 235
- Homeland Security Workshop: role of Materials sciences and technologies 237
- FP7 RAINBOW Training workshop 239
- Materials Brokerage Event 241

List of Participants 243

Index 283

Welcome

The European Materials Research Society
Fall Meeting

***

Warsaw University of Technology

Warsaw (Poland)

14th-18th September, 2009

Introduction

The Fall Meeting of E-MRS was established in 2002 as a twin conference to the annual Spring Meeting held in Strasbourg, France. Initially the Fall Meetings were intended to develop and enhance contacts between scientists from Central and Eastern European countries with those from Western Europe, Asia and America. The inaugural Fall Meeting consisted of three symposia and was held in three locations:

- Moscow - Software Development for Process and Materials Design;
- Warsaw- Interfacial Effects and Novel Properties of Nanomaterials
- Zakopane - Solid Solutions of the II-VI Compounds, Growth, Characterization and Applications

The success of the first meeting in Warsaw has led to the E-MRS Fall Meeting being based entirely in Warsaw since 2003. In this period the conference has grown from 3 to the 12 symposia presented in 2008. The Conference is held in the Central Campus of Warsaw University of Technology. The Fall Meeting organizers follow the procedures developed over the years by the Society to host wide ranging multi-disciplinary conference to bring established scientists with a world reputation together to share their state-of-the-art knowledge and experience. The most important philosophy of E-MRS is to facilitate scientific and social contacts between established researchers, young scientists and Ph.D. students and to promote technology transfer from public institutions to industry.

The Czochralski Award

One of the most important and prestigious events which take place during the Fall Meetings is the Presentation Ceremony of the annual "Jan Czochralski" Award. The Award in the name of Professor Jan Czochralski was instituted by the E-MRS, to commemorate his life long contribution to materials science, technology transfer to industry and international collaboration. The Society has formally legalised the Czochralski Award for Achievements in Materials Science.

In 2006 - the European Materials Research Society, the Polish Academy of Sciences, the Polish Materials Science Society, and the Polish Society for Crystal Growth jointly established the International Chapter of the Jan Czochralski Award which together select the Award Laureate.

To date, five distinguished scientists have been presented with the Czochralski Award:

- 2004 - Professor Walter Heywang (former Director of Research of Siemens);
- 2005 - Proessor Boris Paton (the long term President of the National Academy of Sciences of Ukraine);
- 2006 - Professor Thaddeus B. Massalski (Carnegie-Mellon University in Pittsburgh);
- 2007 - Professor Shuji Nakamura (ERATO, JST, UCSB group, Santa Barbara; the inventor of the Blue Laser Diode);
- 2008 - Professor Kurt Heinz Juergen Buschow (University of Amsterdam)

In 2009 the laureate will be **Professor Hermann Grimmeiss**. Hermann Georg Grimmeiss was born in Hamburg, Germany, in 1930. He was educated in Germany and was awarded his doctorate at the University of Munich in 1957. He joined the Philips Research Laboratory in Aachen the same year and his first task was to find suitable materials to produce“cold” light as possible replacements for conventional light bulbs. His immediate interest (with his colleague H. Koelmans) was focussed on gallium nitride being a III-V compound with the right band gap energy. At that time, very little, was known about the physical and chemical properties of GaN.

In 1965 Hermann Grimmeiss left the Philips Company and Germany develop the Department of Solid State Physics at the newly established Technical University in Lund in Sweden. He held the chair almost continuously until 1981 and was Emiritus Professor of solid state physics from 1983 to 1996. However, he spent a year as a Professor of Physics at the University of Frankfurt from 1973-74 and from 1981-83 he was v.p. RIFA in the Erricsson Group in Stockholm. At Lund His interest focused on materials characterization, particularly on point defect characterization in semiconductors. He is the author and co-author of over 200 scientific papers in this field and has delivered many invited and plenary presentations world wide. He is the founder of the biannual international conference on the characterization of point defects in semiconductors, now called the Lund Conference, because in the beginning the meeting was always organized in Lund.

In the late 80s his interest moved to silicon-germanium, as a substitute of III-V compound materials for the development of fast electronic devices. In 1991, after the reunification of Germany, he was invited by the German Research Minister to build up the new Institute of Semiconductor Physics (IHP) in Frankfurt (Oder) with 160 employees. He convinced the German authorities, that the IHP (as the first institute) should concentrate its efforts on the development of a silicon-germanium technology. The IHP has now about 200 employees and produces and licenses since several years the world’s fastest silicon based transistor.

He held many advisory positions including special advisor to the Swedish Prime minister 1988-89, Wissenschaftsrat of Germany

1990, Danish Ministry of Education 1991, Committee for Science and Research, Land Brandenburg 1993 to 1996. He wasPresident of E-MRS from 2004-2007 and since 2004 he has been Vice-President of the European Materials Forum and chairman of the scientific advisory board at IHP. He has been editor and co-editor of several scientific journals and is a member of several boards: Swedish-German Research association from 1987 and the Einstein Forum, Potsdam, Germany, from 1992, He is a member of the Royal Swedish Academy of Sciences (KVA), the Royal Swedish Academy of Engineering Sciences (IVA), Finish Society of Science and Letters, and the Royal Physiographic Society of Lund. He is a fellow of the APS and honorary member of the A.F.Ioffee Institute, St. Petersburg, and the Roland Götvös Physical Soc., Hungary.

Hermann Grimmeiss has been decorated Bundesverdienstkreuz 1.Klasse 1993, and is recipient of the King's medal of 8th Dimension with blue ribbon, Stockholm 1998, and the order of the North Star, 1969.
He has been chairman of several international conferences and symposia (ICPS 1986, EPS CMD 1986, Nobel-Symposium on "Heterostructures in Semiconductors", Arild, Sweden, 1996, and been chairman of many E-MRS Spring Meeting symposia on Nanosciences and several summer schools (NATO ASI, Erice, Sicily, 1990 and 1995, on "SiGe-from basic physics to applications" etc).

Organisers

Conference Organisers:

- Polish Materials Science Society
- Warsaw University of Technology (*)
- Institute of Physics, Polish Academy of Sciences (**)
- European Materials Research Society (***)

Organising Committee:

- J. Franc (Prague, Czech Republic)
- K. Kurzydłowski (*)
- M. Lewandowska (*)
- A. Mycielski (**)
- P. Siffert (***)
- F. Tuomisto (Helsinki, Finland)

Conference Chairpersons:

- Krzystzof J. Kurzydłowski
- Małgorzata Lewandowska
- Giuseppina Padeletti
- Abdelilah Slaoui

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- **F. Priolo** E-MRS President
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Sponsors

Polish Ministry of Science and Higher Education

European Materials Research Society

(EMRS) a non profit scientific association founded in 1983 focuses on creating the synergy between interdisciplinary, innovative technologies, spreading and exchanging information and promoting technology transfer from public institutions to industry. The primary objective of E-MRS is to promote and enhance the efficiency of research in Europe in the field of Advanced Materials. E-MRS seeks to quickly inform researchers of the scientific and technological developments in their field from the rest of the world through the society's links with other MRS societies belonging to the International Union (IUMRS). In 2002 E-MRS decided to launch in parallel to the well established spring meetings in Strasbourg also the fall meetings in Warsaw.

The Polish Materials Science Society

The Polish Materials Science Society (PMSS) was foundedin December 2004. PMSS promotes interdisciplinary cooperation between scientific working in different scientific areas - materials science and engineering, chemistry, physics, biology, medicine etc. PMSS works in close collaboration with E-MRS.

The main aims of the PMSS are:

- provide a platform facilitating interdisciplinary cooperation between materials scientists from different fields
- strengthening the interdisciplinary meetings, conferences, workshops and schools important for the PMSS members
- distribution of information among scientists working in the field of materials research

Faculty of Materials Science and Engineering, Warsaw University of Technology

The Warsaw University of Technology Faculty of Materials Science and Engineering, FMSE, is the leading research and higher education institution in Poland as it was ranked first in the Ministry of Science Evaluation Exercise. The Faculty has a strong interest in new materials and technologies and has made significant advances and achievements in both fundamental and applied activities in the field.

The teaching of materials science and engineering at Warsaw Uni-

versity of Technology can be trace back to the beginning of twentieth century and is closely related to Professor Jan Czochralski - the inventor of the widely used method of growing single crystals. However, the present Faculty of Materials Science and Engineering, as an independent unit at the University, was created in 1975, initially as the Institute of Materials Science and Engineering and then as the Faculty in 1991.

The Faculty offers materials science education at 3 levels for approximately 350 undergraduate students and about 80 post-graduate PhD students. Degrees are awarded at Licentiate - the equivalent of Bachelors, Masters and PhD level. The Faculty has about 80 permanent academic members of staff supported by the PhD students. These two figures make the Faculty one of the largest academic groups of materials scientists in Europe and ensures that the Faculty possesses a critical mass which provides an excellent opportunity for participation in ambitious international research projects.

Currently, the main research topics include: nanomaterials and nanotechnologies, bioengineering, functional and tailored materials, degradation of engineering materials and surface engineering. The extensive research programmes undertaken at the Faculty are financed by the Ministry of Science and Higher Education, the European Commission, NATO and many industrial partners.
The FMSE has also realised significant achievements in materials characterization and modelling. The Faculty operates a Materials Characterization Laboratory equipped with an ultra high resolution TEM (Jeol JEM 3010), STEM (Hitachi HD 2700 with a C_s correction system and a Hitachi S 5500), X-ray Micro/Nano Tomography and a Focus Ion Beam. The available equipment and staff expertise can provide the key to the understanding and control of the structure of processed materials. Materials characterization is combined with a quantitative description of materials microstructures using the methods of stereology and image analysis. In recent years a team has been developed which successfully undertakes the modelling of materials' structures and properties by ab-initio, molecular dynamic, Monte Carlo and FEM modelling methods. For higher computation power the Faculty cooperates with the Interdisciplinary Centre for Mathematics of Warsaw University which operates a super-computer.

Institute of Physics PAS

The Institute of Physics was established on 24th September 1953 by a special Government decision. It was rooted in the physics faculties of seven universities: Warsaw University and Warsaw Technical University, Jagiellonian University in Kraków, Adam Mickiewicz University in Poznań, Nicolaus Copernicus University in Toruń, as well as the University and Technical University of Wrocław. The Institute was relying on their research staff, premises and laboratory facilities. The Institute was established as a country-wide scientific institution conducting research in all areas of experimental and theoretical physics, including educational obligations. The first director of the Institute, Prof. Stefan Pieńkowski, and the first chairman of its Scientific Council, Prof. Leopold Infeld, were the most prominent physicists of that time in Poland. Since the establishment of the National Agency for Nuclear Energy in 1955, the nuclear physics has ceased to constitute a part of the research program of the Institute. Those units of the Institute which were specifically active in this field (the Departments of Radioactive Isotopes, Elementary Particles, and Nuclear Physics in Krakow) formed the kernel of Nuclear Research Institutes. The fast rate of development of the Institute has led, in the course of years, to the formation of independent research units grown on its basis. In 1975 the Department of Ferromagnetics, Department of Dielectrics, together with the Radiospectroscopy Laboratory created in 1966 in Poznań, became incorporated into the Institute of Molecular Physics of the Polish Academy of Sciences. The Wrocław branch of the Institute became a part of the Institute of Low Temperature and Structural Research, of the Polish Academy of Sciences. The Institute of Physics was also the founder of the Department of Solid State Physics in Zabrze. The industrial, high-tech centers of the Academy of Sciences, such as "UNIPAN" (specializing in scientific electronic instruments) and "WILMER" (humidity meters), as well as the High Pressure Research Center "UNIPRESS", are also rooted in the Institute. In September 2003 the Institute celebrated its 50th anniversary. On this occasion the achievements of the Institute were recognized by the Academy and State authorities: 28 of our scientific and technical staff were decorated by various state medals.

NT - MDT

Nanotechnology is exploding. The world of small sizes has never been as vital for so many directions in science and industry. With this new direction comes a greater need for measurement on the atomic scale and new demands for reliable, precise, and multipurpose instrumentation. **NT-MDT** enjoys a 17-year history in instrumentation created specifically for nanotechnology research, leading the field in originality, quality, and high tech development.

NT-MDT
Integrated solutions for NanoTechnology

Programme

Plenary Session:

1. Monday, September 14th , 10:30 - 12:30 (including Czochralski Award Ceremony)

Poster Sessions (both obligatory for all authors):

1. Monday, September 14th, 16:00 - 17:30
2. Wednesday, September 16th, 16:00 - 17:30

Symposia:

- SYMPOSIUM A, Room 226: InN material and alloys
- SYMPOSIUM C, Room 231: Wide band gap II-VI and III-V semiconductors
- SYMPOSIUM D, Room 144: IR and THz electronics: from materials to devices
- SYMPOSIUM E, Room 213: New materials for spintronics
- SYMPOSIUM F, Room 208: Novel bio&chemosensing materials for health, safety and security applications
- SYMPOSIUM G, Room 206: Materials for membranes in energy applications
- SYMPOSIUM H, Room 134: Bulk amorphous and nanocrystalline materials
- SYMPOSIUM I, Room 219: Multifunctional advanced composite materials: manufacturing, structure, properties, characterization

Satellite event:

- **Workshop on CdTe based crystals for nucleor radiation spectrometry**

(12th - 13th September) Room 123

- **Carbon Dioxide: a new material for energy storage and a challenge for European industry**

(14th September, 2pm - 7pm) Room 123

- **Homeland Security: role of Materials sciences and technology**

(16th September, 10am - 5pm) Room 123

- **RAINBOW Training Workshop "Investigation of the properties of nitride semiconductors"**

(16th-17th September, 2009) Room 226

- **EuMat - FP7 NMP/INCO Brokerage Event**

(17th-18th September, 2009) Rooms 306-309 & 315

Registration

Please note the Main Reception will be open according to the hours given below:

Day	Date	From	To
Sunday	September 13 th	12:00	19:00
Monday	September 14 th	08:00	18:00
Tuesday	September 15 th	08:00	18:00
Wednesday	September 16 th	08:00	18:00
Thursday	September 17 th	08:00	18:00
Friday	September 18 th	08:00	16:00

Exhibition

- Mon (Sept. 14) 12:00 - 17:00
- Tue (Sept. 15) 09:00 - 17:00
- Wed (Sept. 16) 09:00 - 17:00
- Thu (Sept. 17) 09:00 - 12:00

This year, following companies will present their offer during Fall Exhibition event:

- NT-MDT (Russia) as the Main Sponsor of 2009 Fall Meeting & Exhibition
- COMEF (Poland)
- SCHAEFER TECHNOLOGIEGmbH (Germany)
- PREVAC Ltd. (Poland)
- L.O.T. - ORIEL GmbH & Co KG (Germany)
- INTERNATIONAL PUBLISHING SERVICES Ltd. (Poland)

Plenary Session, Symposia and Exhibit

Plenary Session

Programme

Monday, 14 September

Opening & Plenary Session

Czochralski Award Ceremony
Monday morning, 14 September, 10:30
Small Hall (237)

11:00	Invited oral

The World of Materials Science - Recollections and Expectations

Hermann G. Grimmeiss

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Materials science is a multidisciplinary area that strongly interacts with technology. By providing faster and more reliable means of communication through new electronic, optical, and magnetic materials, materials science plays a crucial role in improving economic performances, quality of life and employment, particulary in areas such as living environments, health, communication and transport. Materials Science and Engineering is also a well-recognized discipline with its dual grounding in basic science and engineering, and by virtue of its own intellectual cohesiveness. The history of Materials Science is highlighted by exciting and often unexpected developments, which were not foreseen by classical roadmaps. The paper will focus its discussion on three such examples: Light Emitting Diodes, Microelectronics and Solar Cells.

12:00	Invited oral

Organic Spin Electronics

Mike Coey

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Organic Spin Electronics

Spintronics is a branch of electronics taking advantage of both charge and spin of the electrons. The field started in the 1980s with the discovery of giant magnetoresistance in metallic multilayers. It is actively developing and has been expanded to various materials including semiconductors, nanotubes and graphene, molecular and polymeric materials.

Spin dependent transport has been the object of intense research since the demonstration of magnetoresistive effects in metallic multilayers and magnetic tunnel junctions. The field has evolved to the extent that commercial applications for magnetic recording and electronic memory are now available. This was celebrated in 2007 with the Nobel prize to Albert Fert and Peter Grunberg. In the last few years the range of materials used for spin-devices has witnessed a dramatic broadening. Among others silicon, graphene and organic semiconductors have recently focused considerable efforts of the community. This symposium will be focused on the experimental and theoretical aspects of various spintronic phenomena with particular emphasis on material issues.

Lunch break

(maps available at the Reception desk)
Monday afternoon, 14 September, 12:45
building of the Faculty of Physics

Symposium A

InN material and alloys

Welcome

The small band gap energy, together with the small effective mass, the large electron mobility and high saturation velocities accompanied by the very high resistance to radiation damage of InN have the potential to lead to a variety of device applications, including high-efficiency multi-junction solar cells, high speed transistors, and light emitters the visible to near-infrared spectral region. Furthermore, the high electron concentrations in the surface accumulation layer can be exploited for gas, chemical and biological sensors and for generation of THz radiation. This symposium covers the most recent developments related to growth and characterization of InN materials and its alloys with GaN and AIN as well as InN heterostructures and device applications.

Topics do be covered by the symposium include:

- p-type doping of InN and In-rich nitrides
- Dislocation reduction in heteroepitaxy
- Reduction of free electron concentrations
- Radiation damage, point defects & doping
- Different growth approaches: MBE, MOCVD, HVPE, ELO
- InN quantum wells and dots
- InN surfaces and heterostructure interfaces
- Growth of In(Al)N alloys
- InN based devices (e.g. HEMTs, diodes, solar cells)

Organisers

- **Dr. Filip Tuomisto**, Department of Applied Physics, Helsinki University of Technology, Finland
- **Dr. Pierre Ruterana**, CIMAP-ENSICAEN, Caen, France
- **Prof. Michael Kneissl**, Institut für Festkörperphysik, Technische Universität Berlin, Germany
- **Prof. Yasushi Nanishi**, Department of Photonics, School of Science and Engineering, Ritsumeikan University, Japan

Proceedings

The proceedings of this symposium will be published in physica status solidi (c), with selected papers in physica status solidi (a) and (b).

Sponsors

The symposium is partially supported by the EU through the RAINBOW ITN Grant agreement N°: PITN-GA-2008-213238. Website: http://rainbow.ensicaen.fr

INITIAL TRAINING NETWORK

RAINBOW

Programme

Monday, 14 September

LUNCH

(maps available at the Reception desk)
Monday afternoon, 14 September, 12:30
building of the Faculty of Physics

Session A1

Devices
Monday afternoon, 14 September, 14:00
Room 226
Chair: F. Tuomisto/P. Ruterana

14:00	Keynote lecture

Applications of group III-nitride alloys for multijunction solar cells

Wladek Walukiewicz

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The direct band gaps of the group III-nitride alloys can be continuously tuned in a very large energy range from 0.65 eV in InN to 6.2 eV in AlN. In addition, changing the composition from InN to AlN allows for varying the electron affinity of the alloys from 5.8 to 2.1 eV. These unique properties offer a possibility of using group III-nitride alloys for high efficiency tandem cells. A successful implementation of group III-nitrides for multijunction PV devices requires p- and n-type doping in the whole alloy composition range. As grown, undoped InGaN films are always n-type. Reproducible p-type doping with Mg acceptors in the whole composition range has been reported by several groups. Any design of the multijunction cells based on InGaN or InAlN alloys must consider the n-type surface inversion layer that is experimentally found for p- $In_xGa_{1-x}N$ with $x > 0.4$. A number of possible cell designs will be presented, including structures with graded band gaps. In addition results on a hybrid InGaN/Si double junction (2J) cell will be discussed in detail. Our experimental work has established that for x=0.5 the conduction band of $In_xGa_{1-x}N$ aligns with the valence band of Si. Thus, a low resistance ohmic contact is expected at a junction between n-type $In_{0.5}Ga_{0.5}N$ and p-Si, eliminating the requirement of heavily doped layers needed for the tunnel junctions in the current multijunction cells. This greatly simplifies the design of a 2J hybrid cell. Efficiencies exceeding 30% are predicted for an optimized p-InGaN/n-InGaN/p-Si/n-Si cell based on realistic absorption and trans-

port parameters for InGaN. It should be noted that similar considerations apply to the $In_yAl_{1-y}N$/Si system with y=0.7. Latest results on the performance of the InGaN based tandem cells will be presented and the remaining obstacles in achieving higher power conversion efficiencies will be discussed.

This work was supported by the US DOE and Rose Street Labs Energy, Phoenix, Arizona

14:45	Oral

Low frequency noise measurements in InN films

Jean-Marc Routoure[1], Laurence Mechin[1], Bruno Guillet[1], Arantxa Vilalta Clemente[2], Javier Grandal[3], Miguel Ángel Sánchez-García3, Sara Martin[3], Fernando Calle[3], Pierre Ruterana[2]

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We report DC and low frequency noise measurements at room temperature in InN films patterned using “transmission line method” structures. Two samples with thicknesses 250 nm (sample A) and 345 nm (sample B) were investigated.

The InN films were grown using MBE on a 5 µm thick GaN template on top of a 300 µm thick Al_2O_3 substrate. The width of the devices was 94 µm and the length varies from 38 µm to 210 µm. DC and Noise measurements were performed on more than 25 devices. A mapping of the two samples was performed as well as a study as a function of the length in each sample. The mean resistivity for sample A is 3.8x10□□Ω□m(+/-15%) and $12.7x10^{-6}$Ω□m(+/-4%) for sample B. These values are in the range of those reported in the literature.

The noise spectral density consists of a white noise contribution due to the thermal noise and in the low frequency range, mainly a 1/f contribution. The 1/f noise level varies with the square of the DC voltage V across the sample. A Hooge parameter αcan then be estimated according to the calssical empirical : $S_V/V^2=\alpha/(fxN)$ with Sv the 1/f noise spectral density level estimated at the frequency f and N the total number of free carriers in the device. For samples with carrier concentrations in the range of 10^{18}- 10^{19}at/cm³ the Hooge parameter is found to lie between $4x10^{-5}$- $4x10^{-4}$. The Hooge parameter is a normalized figure of merit for the device quality. Values reported for different kind of materials goes from $10^{-7}$for pure and defect-less material up to more than unity. The values obtained in this instance are in the same order of magnitude than the Hooge parameter of standard CMOS silicon processed devices which may be an indication that the studied devices are of substantially good quality.

Finally, the study versus the length of the devices reveals that the metal-InN contact contribution is not negligible for either DC and noise measurements. More statistics are needed for an elaborate identification.

Acknowledgment: This work is supported by the EU under the Grant agreement N°: PITN-GA-2008-213238, Initial training network RAINBOW of the 7 RTD Framework

15:00	Invited oral

LPMOCVD growth of InAlN/GaN HEMT heterostructures. Comparison of composite SiCopSiC and bulk SiC substrates for HEMT device applications

Marie-Antoinette Di Forte Poisson, Nicolas Sarazin

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InAlN material is a very attractive material as compared to the conventional GaAlN material, for electronics devices. InAlN with 17% of In is lattice matched on GaN, and a high spontaneous polarisation is predicted from theoretical studies. Therefore, material with low strain and low defect density induced is expected together with a very high two dimensional electron gas density at the InAlN/GaN heterojunction.

The optimisation of the MOCVD growth of InAlN/GaN HEMT structures on bulk SiC and SiCopSiC composite substrates and the related device performances are reported in this paper. The InAlN/GaN HEMT heterostructures grown on composite substrates are successfully compared in terms of material quality to the standard InAlN/GaN HEMT Heterostructures grown on bulk SiC substrates.

HEMT devices based on InAlN/GaN heterostructures grown on SiC substrates exhibited an output power density of 10.4W/mm, with a Power Added Efficiency PAE of 52% at 10 GHz. Compared to the published results, that represents the state of the art power performances obtained from AlInN based HEMT. This result strengthens the performance projections made about InAlN based technology, which predicted AlInN based heterostructures as a potential successor to GaAlN/GaN HEMTs for microwave power applications.

Present research work has been supported by the French Ministry of Defence (DGA / STTC, Contract 01/34/050, Korrigan project RTP n° 102.052) and the European Community (Hyphen and Ultragan projects).

COFFEE

Monday afternoon, 14 September, 15:30
Main Hall

Session A2

Devices and Structural Properties
Monday afternoon, 14 September, 15:50
Room 226
Chair: M. Kneissl/Y. Nanishi

15:50 Invited oral

Wavelength-Stable Green Light Emitting Diodes in Non-Polar GaInN/GaN Quantum Well Growth

Christian Wetzel, Theeradetch Detchprohm

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Extending the efficiency sweet spot of 460 nm AlGaInN LEDs all across the visible spectrum proves a formidable challenge. Successive elimination of the most-likely hindrances seems the most prudent approach and reduction of macroscopic line defects certainly requires the highest priority. Next, the role of piezoelectric polarization, a most prominent uniqueness in the group-III nitrides, must be considered. All conventional high-efficiency LEDs and laser diodes are grown along the polar *c*-axis. Consequently, polarization control by growth along other axes, such as non-polar *m*-, and *a*-axes or various semi-polar axes are indicated.

Naturally, such approaches require substantial epitaxy development to explore the ultimate capabilities of the system. By employing homoepitaxy on thick HVPE grown bulk GaN, we achieve significant reduction of threading dislocation densities from the 10^9 cm^{-2} to the 10^7 cm^{-2}. We also succeeded to grow along various crystal orientations after such surfaces have been prepared from the thick bulk GaN.

We have learned to grow blue and even green LEDs with very low line defect densities $<10^8$ cm^{-2}. now in non-polar *a*- and *m*-plane growth as well as semi-polar directions. Unlike other approaches, stacking faults are not a concern ($<10^5$ cm^{-1}). Pushing the emission wavelength beyond 500 nm in the non-polar orientations, yet, proves a particularly large challenge. In the absence of piezoelectric polarization, the wavelength extending aspects of the piezo-dipole cannot be employed. In turn, however, once a certain wavelength has been achieved, an LED will perform with high stability at that wavelength up to high current densities.

What remains to be seen is whether devices of such growth geometry can outperform conventional c-plane material in terms of total output power, differential, and absolute efficiency.

This work was supported by a DOE/NETL Solid-State Lighting Contract of Directed Research under DE-FC26-06NT42860.

Joint Poster Session

Monday & Wednesday
Monday afternoon, 14 September, 16:00
Main Hall

Lectures

Monday afternoon, 14 September, 16:20

16:20 Oral

Microstructure of InN grown on Si (111) by plasma-assisted MBE using a double buffer layer

George P. Dimitrakopulos[1], Thomas Kehagias[1], Adebowale O. Ajagunna[2], Joseph Kioseoglou[1], Gerard Nouet[3], Iordanis Kerasiotis[1], Alexandros Georgakilas[2], Theodoros Karakostas[1], Philomela Komninou[1]

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InN offers important advantages such as a 0.7eV direct bandgap, large electron mobility, small effective mass and high saturation velocity, thus making it a promising material for various optoelectronics and microelectronics applications. It is thus highly desirable to achieve good quality epilayers, and heteroepitaxial growth of InN on Si(111) substrates could reduce the lattice mismatch to ~8%, in comparison to ~11% for growth on GaN(0001) buffer layers. However, previous experimental work showed that a thin AlN/GaN buffer layer on Si improved the InN structural and electrical properties compared to InN nucleation directly on Si.

We report on the structural properties of a 2mm thick InN film grown on Si(111) by nitrogen radio-frequency plasma source MBE, using a buffer layer comprising 20nm AlN and 40nm GaN. Structural characterization was performed in cross section geometry using transmission electron microscopy (TEM), both conventional (XTEM) and high-resolution (HRTEM). The film was single crystalline, and exhibited N-polarity. The double buffer layer and dislocation annihilation interactions in the first ~200nm reduced the threading dislocation density inside the film to $\sim 2.3 \times 10^{10} cm^{-2}$. At the AlN/GaN and GaN/InN interfaces, the roughness is enhanced by threading dislocations terminating at V-defects. This causes the introduction into the film of threading dislocations with **c**-type Burgers vector components in order to accommodate the interfacial roughness. Therefore the majority of threading dislocations propagating into the epilayer were of **a**+**c** mixed type. Strain measurements showed that all the layers were relaxed. The GaN/InN interface was found to comprise a regular array of misfit dislocations, and the InN lattice constants were found as follows: a_{InN} = 0.3529nm, c_{InN} = 0.5708nm. This structure can be considered as an appropriate III-N system for further developments in device fabrication.

Acknowledgement: Work supported under MRTN-CT-2004-005583 (PARSEM)

16:35 Oral

Effects of threading dislocations and other defects on reduction of band-edge photoluminescence in n-InN films

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Recent progress of InN growth led to the decrease in residual electron density n_{e0} to 10^{17} cm^{-3}. As a material of light emitting devices, radiative efficiency and non-radiative recombination processes are the most important issues to be uncovered. There are a lot of defects such as edge-type threading vacancies and dislocations producing residual electrons. Although carrier recombination processes were studied by several groups, defects inducing non-radiative recombination or recombination via deep levels have not been uncovered yet. In this report we show two those non–band edge recombination (NBER) processes for InN samples of the state of the art.

The samples grown by RF-MBE had In-polarity and n-type conductivity. We have reported the importance of edge type dislocations and point defects in carrier scattering in these samples by IR-reflectance analysis, which also revealed n_{e0} ((2 – 10)´10^{17} cm^{-3}) in the bulk region except for the surface or interface accumulation region. The edge and screw type dislocation densities (n_{ed} and n_{sc}) were obtained from the peak widths in XRC of (0002) and (10-10) diffractions, respectively. The temperature dependence of PL intensity, I_{PL}, was obtained by spectral integration in a region of 0.54 – 0.85 eV.

I_{PL} was normalized as I_{PL}/n_{e0}, since I_{PL} is proportional to n_{e0} under weak carrier excitation density of 10^{14} – 10^{15} cm^{-3}. We have found that I_{PL}/n_{e0} at 16 K decreases with n_{ed} and n_{e0}. A sample with large n_{ed} of 2.5´10^{10} cm^{-2} had small I_{PL}/n_{e0} at 16 K in spite of small n_{e0} (2.5´10^{17} cm^{-3}), indicating the existence of an NBER process taking place around edge-type dislocations without threshold energy. On the other hand I_{PL}/n_{e0} decreased with temperature without correlation with n_{ed} or n_{sc}; the parameters obtained from the successful fitting of a model function of I_{PL} with thermal activation to the experimental data had no correlation with n_{ed}, n_{sc}, or n_{e0}. There is another process with threshold energy and originating from defects such as vacancies or impurities except for dislocations. PLE spectra also supported the two recombination processes without and with threshold energy (< 100meV). From consideration on configuration coordinate in NBER, we conclude the importance of the defects except for the dislocations and processes without threshold energy around edge-type dislocations.

16:50 Oral

The microstructure and properties of InN layers

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Recently, due to its smallest band gap among the nitride semiconductors, InN has attracted great interest as a promising material for THz emission, high frequency communication electronics, solar cells and various sensors. Indeed good quality InN layers have been grown by molecular beam epitaxy (MBE), but such material is still not commercially available. In addition, results on HVPE growth of InN and InGaN materials are still limited, and the reported material does not reach the device-quality level. In this work, we have carried out transmission electron microscopy of high-quality HVPE-grown InN layers with good optical, and substantially improved electrical properties. For comparison, we have also investigated InN layers grown on HVPE GaN templates by MBE.

The HVPE InN layers were grown in a temperature range from 500 to 600ºC on GaN/sapphire template substrates using $InCl_3$ –NH_3 gas system. Ar is used as a carrier gas. InN growth rate ranged from 0.3 to 0.5 µm/hour. Layers with thickness from 0.02 to 2.5 microns have been investigated.

In all the InN layers grown on GaN/sapphire substrates, the high density of threading dislocations in InN close to InN/GaN interface decreases markedly with the distance from the interface. High resolution TEM and analytical microscopy show that the interface with GaN is sharp at atomic scale and no In inclusions have been detected. By both techniques the InN layers exhibit good crystalline quality and their polarity has been determined when the thickness was above 0.4-0.5 µm. The MBE layers exhibit a PL edge around 0.8 eV at room temperature. For the HVPE layers, the PL edge position changes from one sample to the next, in the range 1-2 eV. These results will be discussed in correlation with SIMS and Raman investigations.

This work is supported by the EU under Contract RAINBOW: PITN-GA-2008-213238

17:05 Oral

Inclined Dislocation Pair Formation as a Mechanism of Partial Strain Relaxation in GaInN/GaN Quantum Wells on Low-Dislocation Density Bulk GaN

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Approaching the deep green and yellow spectral region in electroluminescence of GaInN/GaN quantum well light emitting diodes (LEDs) requires high alloy fractions of InN which induces a high level of biaxial strain of up to 3%. While in typical sapphire-based epitaxy, among a high density network of threading dislocations (10^8 - 10^9 cm^{-2}), an inclination of such defects is seldom seen, we find those to occur reliably and high density in homoepitaxy on low-dislocation density (~10^6 cm^{-2}) *c*-plane bulk GaN. Their observation revises the long-held interpretation that the smallest available Burgers vector was too large to be likely in this hexagonal crystal structure. We find an inclination angle of 18 - 23° whenever pairs of two dislocations emanate from the GaInN layers and branch off in opposite directions or a 120° pattern. Both dislocations are edge-type and have the Burgers vectors in the growth plane. We don't find any precursor defects suggesting that both Burgers vectors sum up to zero. Comparing various models of the acting forces, we find that the partial strain relaxation induced by the separation of both branches in the pair is the most likely process at work. We find that an open core structure that includes alternate translations of *a*-lattice vectors can very well describe our observations. This strain relaxation mechanism is thought to play a relevant role in the light output performance of wavelength-stabilized green LEDs.

This work was supported by a DOE/NETL Solid-State Lighting Contract of Directed Research under DE-FC26-06NT42860.

17:20 Invited oral

InGaN/GaN nanocolumn LEDs and selective area growth of GaN nano-crystals by rf-plasma assisted molecular beam epitaxy

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III-nitride nano-crystals, such as nanocolumns [1] are attractive candidate for opto-electronic devices, for the sake of their dislocation free-nature, strain-relaxation effect and high light extraction efficiency. For the application of nano-crystals, suitable device structure and accurate shape and position control are required.

Recently we have fabricated InGaN/GaN nanocolumn LEDs on flexible metal sheet. After the growth of n-GaN nanocolumn and InGaN/GaN quantum disks on (111) Si substrate by rf-plasma assisted molecular beam epitaxy, the lateral growth of p-GaN was enhanced to make continuous film at the top surface [2]. Pt/Au (50mm) electrode was formed on the p-side surface and then Si was selectively removed. After filling the interstice of n-GaN nanocolumns with spin on glass, tips of the nanocolumns were exposed in circular (f=400mm) then ITO transparent electrodes were deposited. The nanocolumn LED showed red (663nm) emission under 10mA DC drive.

The size and position fluctuation of self-assembled InGaN/GaN nanocolumns cause broadening of emission spectra. Selective area growth (SAG) of GaN nano-crystals is a key technology to overcome this problem. For the SAG of nano-crystals, (0001) GaN templates coated with nano-patterned Ti (~5nm) were used as substrate. Holes or stripe windows with diameter or width of around 100~400nm were open to appear the GaN surface. When the growth temperature was above 900°C, GaN nano-crystals were grown only on the window area with side facets normal to the substrate surface. Well aligned nanocolumns [3] and plate-like thin nano-crystals (nanowalls) [4] were grown.

This study was partly supported by a Grant-in-Aid for Scientific Research on Priority Areas #18069010 and (B) #21310087 from MEXT.

[1] M. Yoshizawa et al, Jpn. J. Appl. Phys. 36 (1997) L459. [2] A. Kikuchi et al, Jpn. J. Appl. Phys. 43 (2004) L1524. [3] H. Sekiguchi et al, Appl. Phys. Express **1**, (2008) 124002. [4] A. Kikuchi et al, MRS Fall Meeting, Q4.6, USA (2007).

Tuesday, 15 September

Session A3

Optical Properties I
Tuesday morning, 15 September, 8:30
Room 226
Chair: A. Yoshikawa/R. Goldhahn

8:30 Keynote lecture

InN/In nanocomposites: plasmonic effects and optical properties

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Nanocomposites, comprising metallic nano-clusters inside a semiconductor matrix, are promising materials for rapidly developed plasmonics which suggests new concept of light manipulation and generation [1]. Optical properties of the nanocomposites are controlled by collective electron excitations in metallic inclusions or localized plasmons (Mie resonances) [2]. These materials can exhibit a huge discrepancy between the characteristic energies of optical

processes, which match well in a semiconductor, because they occur in spatially different areas which undergo the different influence of the plasmons [3]. The basic properties of the nanocomposites are studied using InN/In with metallic clusters formed both spontaneously and by periodic deposition of In insertions. It is found that the spots of intense cathodoluminescence (CL) is strongly correlated with the agglomerations of the In clusters due to plasmon-induced enhancement. With increasing In amount, the peaks of thermally detected optical absorption (TDOA) and CL move in the opposite directions because of different plasmonic effects in the well-separated clusters and their dense agglomerations. Furthermore, a strong difference is discovered between the onsets of TDOA and photocurrent. This feature is a key to distinguish InN/In from InN; without that, optimization of a structure using absorption or emission data does not guarantee its applicability, e.g., for full-spectrum solar cells. To simulate the plasmonic effects in the nanocomposites, a model has been proposed, which considers the clusters as spheroids of random shapes and orientations. The calculated spectra of the averaged enhancement are in reasonable agreement with optical data. The conditions of terahertz generation in the plasmonic nanocomposites are analyzed and compared with experimental findings as well.

[1] E. Ozbay, Science **311**, 189 (2006).

[2] T.V. Shubina *et al.,* Phys. Rev. Letters **92**, 117407 (2004).

[3] T.V. Shubina et al., Phys. Rev. B **79**, 153105 (2009).

Lectures

Tuesday morning, 15 September, 9:15
Room 226

9:15 Oral

Investigation of InN layers grown by molecular beam epitaxy on Si or GaN templates

Arantxa Vilalta Clemente[1], Marie P. Chauvat[1], Javier Grandal[2], Miguel A. Sanchez-Garcia[2], Fernando Calle[2], Magali Morales[1], Evgenia Valcheva[3], Kiril Kirilov[3], Jean Louis Doualan[1], Pierre Ruterana[1]

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The evolution of surface morphology and stress of molecular beam epitaxy InN/(Si,GaN) heteroepitaxial deposited layers have been investigated by X-ray Diffraction, Raman spectroscopy and Transmission Electron Microscopy. The nominal thickness of the InN layers was between 150 and 750 nm. Atomic force microscopy (AFM) observations show that under specific growth conditions, step flow growth has taken place, independent of the layer thickness. For the surface morphology and roughness in 1µm x 1µm areas, we attain values of root-mean-square (rms) as low as 0.393 nm for the best sample. XRD investigations of the in plain compressive biaxial stress versus layer thickness indicate that the relaxation may not follow a conventional trend as expected with a complete relaxation within the largest thickness layers, and as evidenced for InN films epitaxially grown by radiofrequency magnetron sputtering. The results show that, in the studied samples, the strain does not appear to have a monotonic evolution versus thickness. The structural properties of these layers will be correlated with Raman, TEM and photoluminescence measurements.

Acknowledgment: This work is supported by the EU under the Grant agreement N°: PITN-GA-2008-213238, Initial training network RAINBOW of the 7 RTD Framework and the French-Bulgarian bilateral Programme PAI-RILA, project No DOO2-25/2008/NIS 2536

9:30 Oral

Optical anisotropy of a- and m-plane InN grown on free-standing GaN substrates

Jochen Raethel[1], Pascal Schley[1], Egidijus Sakalauskas[1], Gerhard Gobsch[1], René Müller[1], Thomas A. Klar[1], J. Pezoldt[2], Ruediger Goldhahn[1], Gregor Koblmueller[3], James S. Speck[3], Matthias Wieneke[4], Jürger Bläsing[4], Alois Krost[4]

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The optical response of wurtzite nitride semiconductors at the band gap and in the spectral region of high-energy critical points differs appreciably for light polarization perpendicular (ordinary configuration) and parallel (extraordinary) to the *c*-axis. In order to demonstrate this effect, films (or bulk crystals) are required for which the optical axis lies in the surface plane (*a*- or *m*-plane). For InN, the only studies reported so far employed an *a*-plane layer deposited on *r*-plane sapphire with an *a*-plane GaN buffer. Considerable improvement of the film quality was recently achieved by growing *a*- and *m*-plane InN directly on free-standing *a*- and *m*-plane GaN by plasma-assisted molecular beam epitaxy. In this contribution, we present a comprehensive characterization of these higher-quality non-polar InN materials by polarization-dependent Raman spectroscopy and ellipsometry. The influence of stress on the transition energies is discussed based on the lattice parameters obtained by high-resolution X-ray diffraction.

Raman measurements yielded two strong phonon lines with an intensity ratio strongly dependent on the polarization conditions, but being consistent to the results found for GaN. In comparison to the material grown on sapphire, we observed here a clear shift of the phonon lines to lower wave numbers. No evidence was found for strong phonon-plasmon coupling.

Ellipsometry reveals a clear energy splitting between the ordinary and extraordinary absorption edges for both samples. This splitting amounts to 19 and 39 meV for the *m*- and *a*-plane films, while the transition energy at the Fermi wave vectors was found at 680 and 720 meV, respectively, in the ordinary configuration. The different

values are a clear indication for the influence of stress on the valence band structure at the Γ-point of the Brillouin zone and for band filling. Finally, the polarization behaviour at the high-energy van Hove singularities was almost identical to the previous findings.

9:45 Oral

Optical properties of InN grown on Si(111) substrate

Egidijus Sakalauskas[1], Pascal Schley[1], Ruediger Goldhahn[1], Javier Grandal[2], Miguel A. Sanchez-Garcia[2], Enrique Calleja[2], Arantxa Vilalta Clemente[3], Pierre Ruterana[3]

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A comprehensive characterization of the optical properties of wurtzite InN films grown on Si(111) is presented. InN layers were deposited by plasma assisted molecular beam epitaxy. Two types of InN grown on Si(111) substrate samples are investigated in this work: InN on AlN/Si(111) and InN on GaN/AlN/Si(111). The crystalline quality of the layers was investigated by means of X-ray diffraction. Surface morphology was measured by atomic force microscope (AFM). Spectroscopic ellipsometry between 0.56 eV and 9.8 eV (synchrotron radiation) is applied in order to determine the dielectric function (DF), i.e. the absorption related properties, while conventional photoluminescence yields the emission characteristics. For InN grown on Si(111) substrate, the absorption onset is slightly shifted to higher energies with respect to films on GaN/sapphire substrates studied for comparison. The electron density is obtained from the size of the Burstein-Moss shift. The analysis includes the non-parabolicity of the conduction band and the influence of strain on the band gap.

Despite the higher carrier concentration for InN on Si(111) samples, strongly pronounced optical transitions due to critical points of the band structure are found in the high-energy part of the spectra. The fitted transition energies are in excellent agreement with the results for films grown on sapphire. It emphasizes the already promising quality of the films on silicon and makes feasible that Si with its several advantages such as large size, high quality, low cost and high potential for integration with well-developed Si-based devices can serve as a substrate for InN epitaxy.

We acknowledge the support of the EU under Grant agreement N°:PITN-GA-2008-213238 (RAINBOW project: Initial Training Network of the 7th RTD Framework).

10:00 Invited oral

MOVPE growth and characterization of InN and related heterostructures

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Among the Group III-nitrides, InN has been an enigma, with excellent material properties predicted theoretically, which have been difficult to realize experimentally, primarily limited by poor material quality. I will present an overview of research at TIFR into the synthesis and characterization of InN epilayers.

We have performed a detailed investigation of the microstructure of InN deposited under various MOVPE conditions in a showerhead reactor, allowing us to optimize the InN epilayers. We obtain InN layers that are highly mosaic, with an intrinsic bandgap of ~ 0.7eV, whose measured absorption edge is strongly influenced by background carrier concentration and residual stress. By a careful evaluation of the lattice constants we find that the films are under primarily hydrostatic stress which results in a shift in the band edge to higher energy. The key factor influencing the microstructure is the total amount of ammonia dissociation which is governed by a combination of growth parameters. Inserting an InGaN interlayer between the GaN buffer layer and InN films is helpful in reducing the edge dislocation density, which is otherwise not very sensitive to the growth parameters for a given layer thickness.

We found signatures of superconductivity in a few of our InN samples. However, by a careful correlation of the temperature dependences of both resistivity and magnetic susceptibility with structural information, we conclusively established that the superconductivity seen is not intrinsic to the InN, and arises from the presence of trace amounts of indium oxide impurity in the sample.

We have grown GaN/InN/GaN single- and multi-quantum well structures by MOVPE, although with very rough interfaces. From internal photoemission measurements we can determine the band offsets at the GaN/InN heterojunction. Recently, we have also synthesized a-plane InN layers via MOVPE, and are in the process of optimizing the layer quality.

COFFEE

Tuesday morning, 15 September, 10:30
Main Hall

Session A4

Advances in Growth and Devices
Tuesday morning, 15 September, 10:50
Room 226
Chair: W. Walukiewicz/A. Kikuchi

10:50 Invited oral

Recent advances in the MOVPE growth of Indium Nitride

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Within the last decade, a lot of research efforts have been devoted to Indium Nitride (InN), the least known of the semiconducting group-III nitrides.
Most of the samples available today have been grown using the Mo-

lecular Beam Epitaxy technique, and fewer using the Metal Organic Vapor Phase Epitaxy (MOVPE) method. Whatever the method, InN is extremely chalenging, in particular due to the fact that no lattice matched substrate is available. The use of low temperature InN buffer layers was investigated, in order to transpose the successful process for GaN growth to the epitaxy of InN. The growth of low temperature InN buffer layers on both sapphire and GaN layers will be discussed here, along with the use of surfactants (indium halides) in the growth ambient to improve morphology. Furthermore, we will show that post-growth thermal treatment of InN films improves the layers properties. Regarding the growth of InN quantum dots, we present an alternative approach consisting of the thermal annealing of a very thin InN layer deposited at low temperature. The recrystallization process which occurs, leads to the formation of dots with an improved orientation distribution, compared to as-grown InN dots

11:20 Oral

Luminescence and structural studies of In-containing III-nitrides

Carol Trager-Cowan[1], Naresh Kumar[1], Paul R. Edwards[1], Robert W. Martin[1], Kevin P. O'Donnell[1], Francis Sweeney[2], Angus J. Wilkinson[3], Austin P. Day[4], Tao Wang[5], Peter J. Parbrook[6], Ian M. Watson[7]

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The scanning electron microscopy techniques of wavelength-dispersive X-ray (WDX) mapping, hyperspectral cathodoluminescence (CL), electron backscatter diffraction (EBSD) and electron channelling contrast imaging (ECCI) allow the investigation and correlation of the structural, compositional and luminescence properties of III-nitrides on the nanoscale.

WDX provides a quantitative measure of the composition of the material under study. CL provides information on a sample's luminescence properties, where the luminescence spectrum is sensitive to strain, crystallinity, defects, doping and free carrier concentration. To directly correlate composition and luminescence properties, high-sensitivity WDX maps are acquired at the same time as CL spectra [1]. InN fluctuations in $In_xGa_{1-x}N$ thin films have been measured on a scale of tens of nanometres [2].

To obtain a direct measure of the structural properties of III-nitrides we use the techniques of EBSD and ECCI [3]. An EBSD pattern is recorded by placing a phosphor screen in front of a highly tilted sample so that it intercepts electrons backscattered from the sample. An EBSD pattern provides a direct measurement of a sample's crystal structure as it is a 2-D projection of the crystal structure. EBSD patterns acquired from a mesh of points on a sample can be used to produce maps of crystal structure, tilt, rotation and strain with a spatial resolution of order 20 nm.

ECCI exploits the fact that electrons will channel down the crystal planes of a suitably tilted sample. Images derived from these channelled electrons reveal defects which distort the crystal planes. Images with a resolution of tens of nanometres are obtainable.

We will report on recent results from In-containing III-nitrides produced by the RAINBOW Network [4].

1. R. W. Martin et al., phys. stat. sol. (a), **201** 665 (2004).

2. P. R. Edwards et al., phys. stat. sol. (c), **0** 2474 (2003).

3. C. Trager-Cowan et al., Phys. Rev. B **75**, 085301 (2007).

4. http://rainbow.ensicaen.fr/

11:35 Oral

Non-linear absorption measurements of InN/InGaN multiple-quantum-wells structures at 1.5 µm using the Z-Scan method

Fernando B. Naranjo[1], Prem K. Kandaswamy[2], Sirona Valdueza-Felip[1], Lise Lahourcade[2], Vincent Calvo[2], Miguel González-Herráez[1], Sonia Martín-López[3], Pedro Corredera[3], Eva Monroy[2]

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Non-linear optical phenomena, like self focusing, phase conjugation and four-wave mixing (FWM) have direct applications in coherent control of optical communications. InN is a semiconductor particularly interesting for telecommunications, since it has direct band-gap with a room-temperature energy of 0.65-0.7 eV, and predicted high optical nonlinear coefficients. These nonlinear properties can be enhanced using low dimensional structures, like InN/InGaN multiple quantum wells (MQWs). At the same time, these structures offer the possibility of tuning the resonance wavelength, making them very attractive for all-optical devices in communications applications, like optically controlled delay lines and wavelength converters.

In this work, we have measured the non-linear absorption of InN/$In_xGa_{1-x}N$ MQWs with $0.9>x>0.7$ grown by plasma-assisted molecular beam epitaxy on GaN-template substrates. The structural quality of the samples were confirmed from high-resolution X-ray diffraction data, which reveal well-defined multiple satellite peaks for all samples, confirming the existence of the MQW structure with abrupt interfaces. Low temperature (5 K) photoluminescence measurements show a redshift of the emission energy when increasing the Ga content in the barriers. This behaviour is explained in terms of an increase of the piezoelectric fields present in the well. The Z-scan method is used to estimate the non-linear optical absorption coefficient, a_2, of the samples at 1.5 µm. The nonlinear behaviour of the MQWs shows a dependence on the Ga content in the QW barrier.

Saturable absorption, with a_2~-8.9x10^3 cm/GW, is obtained for samples with low Ga content. On the other hand, Two Photon Absorption (TPA) mechanism is observed for samples with high Ga contents, obtaining values of a_2~3.6x10^3 cm/GW. The observed TPA is attributed to photoexcitation of photogenerated carriers from the confined level in the QWs.

11:50 Keynote lecture

Recent Advances in InN-based Solar Cells; Status and Challenges in InGaN and InAlN Solar Cells

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The III-nitrides InGaN, InAlN are very attractive materials for the application to super-high efficiency solar cells, since bandgap energies in the range of 0.7-2.5 eV, which are the essential requirement for the fabrication of multijunction tandem solar cells with a efficiency higher than 50%, can be obtaied only by adjusting their In composition. In this paper, we review the recent advances in InN-based solar cells and discuss the main issues in their fabrication.

The studies on InGaN cells have been started using Ga-rich materials since the growth and Mg-doping of GaN were widely studied. By using MOVPE growth, InGaN films with an In content up to 0.4 have been successfully grown without both phase separation [1]. By doping with Mg, p-type conduction and an n^+-p homo-junction have been successfully realized for $In_{0.2}Ga_{0.8}N$ (Eg=2.65eV) on a GaN template. The fabricated device shows a Voc =1.5 V and Jsc=0.5 mA/cm^2 under AM1.5 illumination [1]. For InGaN with a higher In composition, a higher CP_2Mg flow rate is needed to get p-type conduction [2]. This is due to a higher defect density in InGaN with a higher In composition. The crystalline quality of InGaN is also deteriorated with increasing Mg doping level. The insertion of step-graded $In_xGa_{1-x}N$ interlayer between the GaN and InGaN epilayer has been found to be effective to improve the epilayer quality [3].

The study on InAlN growth has been started from In-rich side. Single-crystalline InAlN films with an Al content from 0 (Eg=0.7 eV) to 0.43 (2 eV) have been successfully grown by employing atmospheric-pressure MOVPE [4]. These films show a strong PL at room temperature. However, the Al composition in InAlN and its distribution are found to be markedly influenced by the parasitic reaction between TMA and NH_3. The reduced pressure growth at around 76 Torr is effective to suppress the parasitic reaction, giving a reduced compositional gradient along the gas flow direction [5]. The study on Mg-doping for InAlN will be the next step.

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LUNCH

(maps available at the Reception desk)
Tuesday afternoon, 15 September, 12:30
building of the Faculty of Physics

Session A5

Growth and Properties I
Tuesday afternoon, 15 September, 14:00
Room 226
Chair: T. Shubina/C. Van de Walle

14:00 Invited oral

Growth of InN and Related Alloys Using Droplet Elimination by Radical Beam Irradiation

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InN is the most difficult binary material to grow among group III nitride semiconductors. It is known that the growth under a N-rich condition results in a poor-quality film with a rough surface. The growth under an In-rich condition leads to a high-quality InN film, but In droplets are formed on the surface. Once In droplets are formed on the surface during growth, the elimination of droplets is essentially impossible by thermal treatment, since InN decomposes before In droplets are evaporated.

In this talk, a new method using droplet elimination by radical beam irradiation (DERI) is proposed for a reproducible growth of high-quality InN. This growth method is composed of In-droplet formation and elimination steps. In In-droplet formation step, In droplets are formed on a surface by either InN growth under an In-rich condition or simple In irradiation. These droplets are eliminated by transforming to InN epitaxially on the underlayer in In-droplet elimination step, consisting of simple nitrogen radical beam irradiaiton. This method supplies simple and reproducible growth of high-quality InN film, since DERI process can be monitored in situ by RHEED intensity variation. This method can be useful for the growth of not only polar c-plane but also non-polar m-plane InN.

The growth of InGaN using DERI process is also demonstrated. In the case of InGaN, Ga is preferentially captured from Ga and In mixture into the growing InGaN layer and In is preferentially swept out to the surface, especially when they are grown under a metal-rich condition. The swept In was transformed to InN epitaxially on an InGaN underlayer during the DERI process.

This work was supported by the MEXT through Grant-in-Aids for Scientific Research in Priority Areas “Optoelectronics Frontier by Nitride Semiconductor” #18069012 and Scientific Research (A) #21246004. One of the authors (T. Y.) was also supported by Grant-in-Aid for Young Scientist (B) #21760237 from the MEXT.

14:30 Oral

Study on Indium Gallium Nitride Quantum Wells: A Comparison between MBE grown and MOVPE grown Quantum Wells

Nils A K. Kaufmann, A. Dussaigne, Pierre Corfdir, Eric Feltin, Denis Martin, Jean-daniel Ganière, Nicolas Grandjean

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Group III Nitrides offer the possibility to elaborate laser diodes (LDs) and high brightness light emitting diodes (LEDs) that can range from ultraviolet to blue (400 to 490 nm) due to their active zone which is composed of InGaN/GaN quantum wells (QWs). The green range is missing in the color palette of LDs and high brightness LEDs. It is due to both the difficulty to increase the In content above 20-25% (low miscibility of In in GaN and difference in lattice parameter) and to the degradation of high In content QWs at high growth temperature (above 750°C). Metalorganic vapour phase epitaxy (MOVPE) is the proof growth technique which guaranties high efficiency blue LEDs. However, for high In content QWs, the p-type GaN layer, which is grown on top of the active zone at 1000°C, degrades significantly the device efficiency. Although LEDs grown by molecular beam epitaxy (MBE) have still not demonstrated high efficiency InGaN/GaN QWs, this technique offers the possibility to grow p-type GaN at low temperature (740°C) and would thus be of interest for the elaboration of high brightness green LEDs. However, understanding the low radiative efficiency of InGaN/GaN QWs grown by this technique is still a challenge. Different works, such as those of Hangleiter et al., Chichibu et al., or Sonderegger et al., have proposed different possible explanations accounting for the high efficiency of MOCVD QWs. In this work, we propose to study the quantum efficiency discrepancies of InGaN/GaN QWs grown by both MBE and MOVPE techniques. Two InGaN/GaN QWs with 25% In content but with different well width (1,5nm and 2,5nm) were prepared. The four samples are analyzed by photoluminescence (PL), micro-photoluminescence, atomic force microscopy and cathodoluminescence. Comparing the PL intensity for different excitation wavelength and as a function of the temperature allows us to extract the internal quantum efficiency.

14:45 Oral

In-vacancies in Si-doped InN

Christian Rauch[1], Floris Reurings[1], Filip Tuomisto[1], Tim Veal[2], Chris F. McConville[2], Hai Lu[3], William J. Schaff[3], Chad S. Gallinat[4], Gregor Koblmueller[4], James S. Speck[4]

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Despite of remarkable improvements in crystal quality during the last years, as-grown InN still exhibits a strong n-type tendency and considerably high defect concentrations [1]. Concerning native defects, recent calculations [2] predict In-vacancies to be the most abundant defect type in n-type InN and possess the tendency for forming complexes with other intrinsic defects or impurities [3], which raises the question of their impact on the materials properties. In this contribution we present results obtained with positron annihilation spectroscopy in MBE-grown Si-doped InN with electron concentrations from 1 x 10^{18} cm^{-3} to 6.6 x 10^{20} cm^{-3}. Temperature dependent Doppler Broadening measurements show In-vacancies in concentrations from 2 x 10^{16} cm^{-3} to 7 x 10^{17} cm^{-3} which increase with increasing electron concentration in the material. These values are remarkably higher than what can be estimated from theoretically predicted formation energies of In-vacancies in InN based on experimentally determined Fermi levels in the investigated samples [4]. Additionally, a strong inhomogeneity of the defect distribution with an increasing vacancy concentration towards the layer/substrate-interface can be observed. These results suggest that the formation energy of In-vacancies is locally reduced in the vicinity of extended defects, which are especially abundant in the interface area - possibly through the formation of defect complexes. From the observed threshold for the formation of In-vacancies, which is seen by a comparison with earlier results in He-irradiated InN [5], it can be deduced that in spite of their still relatively low absolute concentrations In-vacancies play an important role in reducing the electron mobility in the material.

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15:00 Invited oral

Meissner effect of superconducting InN

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Since the first report on the superconducting InN with a narrow band gap in 2000, several reports on it have been published. C. T. Liang et al. reported the superconductivity of InN as huge positive magnetoresistance (APL 90, 172101), D. C. Ling et al. reported it as intrinsic properties (pss(b) 244, 4594), and Kadir et al. reported it as extrinsic properties (SSC 146, 361). Recently Tiras et al. reported that p-type InN becomes a superconductor at T_c=3.97 K (APL 94, 142108). It indeed should be decided whether the superconductivity is intrinsic or extrinsic. The superconductivity of InN is characterized by its low carrier concentration. Diamond and Si which are boron doped become superconductors when the carrier concentration exceeds ~5x$10^{21}$$cm^{-3}$. On the other hand InN with a carrier concentration of 4x$10^{17}$$cm^{-3}$ shows superconductivity below 3 K, which indicates that the electronic structure of InN is the key issue to understand the origin of the superconductivity.

Here we report on the Meissner effect of InN. Samples with a carrier concentration as low as $4x10^{17}cm^{-3}$ show Meissner effect. They show an upward curvature of the upper critical field expressed by $H_{c2}(T)\sim\exp(-T/T_0)$, where T_0 is the specific temperature for the superconductive transition. This curvature is explained by a micro-Josephson junction mechanism, which was introduced for the explanation of irreversible line of high T_c superconductors. The excellent fitting of the theoretical curve to the experimental data corroborates the conclusion that the transition is Josephson coupling due to the presence of superconducting clusters with a transition temperature T_{c1} higher than the bulk transition temperature T_{c0}. The transition at T_{c1} (~3 K) is attributed to the resistive onset temperature where the superconductivity occurs in clusters separated from each other by ~1 μm, which is estimated from T_0, while T_{c0} (~0.5 K) is the temperature at which superconducting coherence forms through Josephson coupling between the superconducting clusters. It is found that the flux motion between T_{c1} and T_{c0} is reversible, which means that no vortices have formed within the clusters.

From these results we propose a mechanism of the superconductivity in terms of residual carriers produced by 4*d* semicore electrons originating from the second nearest neighbor In-In interactions, which is the cause of the micro-Josephson junctions.

COFFEE

Tuesday afternoon, 15 September, 15:30
Main Hall

Session A6

Growth and Properties II
Tuesday afternoon, 15 September, 15:50
Room 226
Chair: J. Speck /A. Yamamoto

15:50 Invited oral

Cross-sectional scanning photoelectron microscopy/ spectroscopy studies on the electronic structures of InN surface and interface

Chung-Lin Wu[1,2], Hong-Mao Lee[2], Cheng-Tai Kuo[2], Chia-Hao Chen[3], Shangjr Gwo[2]

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Indium nitride (InN), like other group-III nitrides, is a polar semiconductor in wurtzite structure, which exhibits strong orientation-dependent electronic and optical properties. It is now well recognized that the surface and interface properties of III-nitride films grown in the polar directions are drastically different from their counterparts grown in the nonpolar directions. In this talk, a cross-sectional view of wurtzite III-nitrides will be presented for studying the electronic properties of nonpolar InN surface and interface. For these studies, a wurtzite N-polar (–*c*-axis), undoped InN/GaN/AlN heterostructure, consisting of a InN top layer, a GaN intermediate layer, and a AlN bottom layer, was grown by plasma-assisted molecular beam epitaxy (PA-MBE) on a Si (111) substrate with relaxed lattices. Using this III-nitride heterostructure sample grown on Si substrates, *insitu* cleavage becomes a viable approach to expose the clean, nonpolar {-1-120} surface (i.e., the *a*-plane) under ultra-high vacuum conditions. To investigate the cleaved surfaces which present a small cross section on the order of a few mm, a special experimental technique, cross-sectional scanning photoelectron microscopy and spectroscopy (XSPEM/S), was applied to achieve the required spatial resolution. The unique features of XSPEM/S for studying InN surface and interface are: (1) Elimination of the polarization effects associated with the interface charges/dipoles normal to the cleaved cross-sectional surfaces of III-nitride heterojunctions, and (2) Formation of nonpolar InN surface without the influence of indium metal adlayers.

At the III-nitride heterojunctions, the spontaneous and piezoelectric polarizations [1] manifest themselves with bound interface charges and built-in electrostatic fields. It has been found that the discontinuities of spontaneous and piezoelectric polarizations play an important role in the "apparent" band lineups at the III-nitride heterojunctions. Martin *et al*. reported that the strong asymmetry of measured valence band offset (VBO) vs. growth sequence is due to the built-in piezoelectric fields with opposite directions [2]. Very recently, we also reported that, under nearly strain-free conditions, large variation in VBO can still be found for InN/GaN heterojunctions grown with different crystal polarities (cation-polar vs. anion-polar) [3]. This phenomenon was explained by the presence of discontinuity of spontaneous polarizations across the III-nitride heterojunction. Therefore, the "intrinsic" VBOs of III-nitride heterojunctions can be obtained only after eliminating the effects of polarization fields. The XSPEM/S measurement of "intrinsic" VBO of InN/GaN heterojunction by using photoelectron emissions from the *insitu* cleaved *a*-plane surface [4] will be presented here as an example to demonstrate its unique feature.

In addition, indium nitride (InN), due to its narrow direct bandgap and superior electron transport properties, has recently emerged as a technologically important semiconductor for use in near-infrared optoelectronics, high-efficiency solar cells, and high-speed electronics. The current obstacle for extensive fundamental studies and widespread applications of InN is mostly related to its unique surface electronic properties [5], resulting from the exceptionally large electron affinity. In particular, it has been found that the growth surfaces of InN exhibit a striking phenomenon that an intrinsic electron accumulation layer can form in the near-surface region of as-grown, unintentional *n*-type InN. This phenomenon has been experimentally confirmed by a variety of different techniques [6,7] and all results indicate that the Fermi level at InN growth surfaces is pinned well above the conduction band edge. Based on the first-principles calculations, Segev and Van de Walle suggested that the microscopic origin of donor-type surface states is mostly associated with In-In bonding states on the InN surfaces with In adlayers. Furthermore, they have predicted that the phenomenon of surface electron accumulation will be absent on reconstructed nonpolar InN surfaces without In adlayers [8]. Very recently, using the technique of XSPEM/S, we have provided definitive evidence of unpinned Fermi

level for *insitu* cleaved *a*-plane InN surfaces [9]. To confirm the presence or absence of band bending, the surface Fermi level relative to the valence band edge was precisely measured by using both the Fermi edge of Au reference sample and the core level of ultrathin Au overlayer. Experimental data unambiguously show the absence of band bending for the *insitu* cleaved *a*-plane InN surface. In contrast, all the growth surfaces of InN (polar or nonpolar) with In adlayers exhibit a large downward surface band bending.

Acknowledgment:

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16:20 Oral

Changes in the valence band structure of as-grown InN(0001) surfaces upon exposure to oxygen and water

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We investigated the surface chemistry and valence band structure of as-grown thin InN films as well as their change upon exposure to oxygen and water in a combined UHV molecular beam epitaxy (MBE) and surface analysis system (base pressure < $2x10^{-10}$ mbar). Thin InN(0001) films were grown on GaN(0001)/Al_2O_3 substrates by plasma assisted MBE and in-situ characterised by reflection high energy electron diffraction (RHEED) and photoelectron spectroscopy (XPS, UPS). In dependence of the growth parameters, we observed different surface reconstructions and corresponding electron states above the valence band maximum. The oxygen and water exposure was directly performed on the as-grown, contamination-free InN surfaces without any further preparation. Changes of the chemical surface states as well as the electronic properties upon continuous exposure to oxygen or water were detected. The surface states observed on clean InN films disappeared in both cases and two similar adsorbate-related electron states at around 5 eV and 10 eV were formed in the valence band in combination with changes in the work function and surface band bending. Furthermore, the reactivity of the different molecules was compared and revealed, that the reactivity of water is much higher than that of oxygen. Additionally, these molecules have different impact on the work function f and surface band bending. In the case of oxygen exposure, an increase in f and a reduction of the downward band bending was found. On the other hand, water decreases f and leads to a further downward shift of the bands at the surface. The details of the coverage-dependent changes will be discussed with respect to surface dipole formation, charge transfer and changes in the surface accumulation layer. Furthermore during oxidation, different chemical oxygen bonds are formed, while a direct assignment to In-O or N-O bonds is difficult due to the change in core level peak shape.

16:35 Oral

Vacancies in InN and In rich InGaN

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InN is surprisingly radiation tolerant, motivating the modeling of the simplest native defects with electronic structure theory. Several groups have investigated the two types of native vacancies in InN, despite the known issues of the problematic band-gap. To partly alleviate the effect of the metallic (LDA) or very narrow gap (GGA) electronic structure, we have instead considered extreme indium-rich (>90%) InGaN. The recent work of Zunger *et al.* suggests that, at least for cubic material, the high concentration limit is well represented by random alloys. Hence we present the results of a new extension of the special quasirandom structure method to consider large numbers of atoms suitable to study defects. Focusing on the nitrogen vacancy system, we discuss the local structure, estimated electronic levels and diffusion barriers in addition to qualitatively discussing the effects on the band-structure.

16:50 Oral

Polarity determination of InN by using circular photogalvanic effect

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To determine lattice polarity is an important issue in InN research

since it leads to various differences in growth behaviours and properties such as crystal growth modes, surface stability, and impurity incorporation. In this presentation, a novel way was proposed to determine lattice polarity by using circular photogalvanic effect (CPGE).

In CPGE, a net current was generated without adding any external bias under the irradiation of a circular polarized light. This CPGE current was observed in as-grown InN layers in a magnitude of several nA, Origin of the CPGE can be attributed to lack of spatial inversion symmetry. The former degeneration band splits into two bands with opposite spin indices as symmetry reduced. When the InN layer is radiated by circular polarized light, an asymmetry distribution of excited electrons in momentum space is generated due to optical selection rules, which leads to a net current in the InN.

As a wurtzite semiconductor, InN is lacking of inversion symmetry. Along c-direction, the lattice of In-polarity can be regarded as a complete inversion of that of N-polarity. Thus the spin-orbit interaction in InN with contrary polarities should be opposite, resulting in opposite spin splitting. Therefore, the sign of photocurrent due to CPGE should be opposite for InN layers with different polarities because of the opposite spin splitting. This prediction was then confirmed by the experimental observation that both CPGE photocurrents and their dependences on the incident phase angle are opposite for InN layers with different polarities. Furthermore, it was found that the above phenomenon is independent of conduction type, i.e. both n- and p-type InN shows the same sign of CPGE current and the same dependence on the incident phase angle as well. Therefore, polarity of InN can be easily determined by investigating their CPGE photocurrents.
It should be noted that this method is operated under ambient, technically simple and sample-nondestructive.

17:05 Oral

Preparation and surface structure of InN(0001) and $In_xGa_{1-x}N$(0001) surfaces

Christian Friedrich[1], Marcel Ewald[1], Amelie Biermann[1], Brendan Holland[2], Veit Hoffmann[3], Michael A. Kneissl[1], Norbert Esser[2], Patrick Vogt[1]

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InN and InGaN alloys still belong to the least understood materials among the group III-nitrides. However, due to the promising applications, e.g. blue, green and infrared light emitters, solarcells and high frequency transistors it is crucial to obtain more information on the atomic surface structure, binding configuration and electronic properties such as surface charge accumulation and band bending. Furthermore, the existence of metallic bilayers has been proposed recently, which require more detailed investigation [1].

$In_xGa_{1-x}N$ layers with different In-content (0.07<x<0.3) and film thicknesses ranging between 15nm and 30nm were grown by metallorganic vapour phase epitaxy (MOVPE) on (0001) GaN/sapphire. Within this work the preparation of clean surfaces was investigated by annealing the $In_xGa_{1-x}N$ samples under ultrahigh vacuum (UHV) conditions at temperatures between 350°C and 800°C and the results are compared to results obtained on 300nm thick InN films on sapphire substrate. The stoichiometry, atomic structure and binding configuration were elucidated with Atomic Force Microscopy (AFM), Electron Energy Diffraction methods (LEED, RHEED), Scanning Tunneling Microscopy (STM), Auger Electron Spectroscopy (AES) and Synchrotron based Photoemission Spectroscopy (SXPS).

The post-growth surface morphology was determined by AFM and reveals a surface roughness of approximately 2nm for InGaN (15% In-content) and 25nm for InN. Also typical defects like screw dislocations and surface pits can be observed. After transfer into UHV the samples were thermally deoxidized by radiative heating. AES and SXPS measurements of the chemical surface composition confirm residual oxide components such as carbon and oxygen even after annealing up to 600°C on both, InN and InGaN. The surface symmetry as observed by RHEED and LEED reveals a (1x1) pattern and does not change during these annealing steps. However, further annealing of InGaN at higher temperatures leads to a strong reduction of carbon and oxygen and a change of the diffraction pattern. LEED measurements reveal clearly a (1+1/6)-surface periodicity as reported for GaN(0001) in [2]. Depending on the preparation conditions a (2x2) symmetry can be observed. STM images on such prepared clean surfaces show atomically flat terraces. Annealing at higher temperatures leads to a destructive change of the surface morphology and chemical composition. Further, LEED reveals faceting structures on the surface.

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17:20 Invited oral

MBE growth and characterization of InN-based layers and nanostructures for infrared applications

Miguel A. Sanchez-Garcia[1], Javier Grandal[1], Fernando Calle[1], Enrique Calleja[1], Eva Gallardo[2], Jose M. Calleja[2], Esperanza Luna[3], Achim Trampert[3]

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The InN band gap (0.7 eV) and its predicted high electron mobility have generated a great interest on InN-based heterostructures for photovoltaic devices (multi-junction solar cells) and high power/ high speed electronic devices. InN-based alloys are also promising materials for high-efficiency optoelectronic devices (laser diodes and detectors) for optical communications in the near infrared (IR) range.

This work presents a study and revision on the growth and characterization of InN-based layers and nanostructures by plasma-assisted

molecular beam epitaxy (MBE) using different substrates (i.e. Si(111), Si(l00) and GaN templates). The effective III/V ratio employed during the growth clearly determines the morphology of the InN layers. N-rich conditions lead to InN nanorods, while the growth under slightly In-rich conditions yields a compact morphology. Structural characterization is performed by electron microscopy techniques (HRTEM, SEM), while optical properties are analyzed with photoluminescence (PL) and Raman techniques.

This work also focuses on the structural and optical characterization of InN/InGaN MQWs heterostructures designed to emit in the 1.5 um window. Finally, electrical transport properties and the issue of charge accumulation at the surface of InN layers are discussed in order to assess the viability to fabricate practical InN-based electronic devices.

This work was partially funded by the Initial training network RAINBOW of the 7 RTD Framework and the Community of Madrid through the project FUTURSEN, CAM S-0505/AMB-0374.

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Wednesday, 16 September

Joint Session A7 and RAINBOW Worshop -RW1

InN Properties
Wednesday morning, 16 September, 8:30
Room 226
Chair: C. McConville/C. Giesen

8:30 Keynote lecture

Spectral properties of InN and related compounds from first principles

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Electronic single- and two-particle excitations and their influence on electron, optical, and X-ray spectra are studied by *ab initio* methods and discussed in the light of experiments for InN and its eigenoxide In_2O_3. The theoretical approaches are based on iterative solution of the quasiparticle equation [1]. The resulting gaps 0.47 eV (zinc blende) and 0.71 eV (wurtzite) are close to the experimental data [2]. The total density of states (DOS) explains the XPS measurements [3]. There is excellent agreement between site- and orbital-projected DOS and X-ray absorption spectra [4]. The rigid shift between theory and experiment is identified as core exciton binding energy. The inclusion of excitonic effects yields optical spectra with details of the measured peak structure [5]. Our computational method [6] possesses an accuracy that allows the prediction of binding energies E_B for Wannier-Mott excitons. For InN we predict $E_B < 7$ meV. A pseudomorphic growth of the eigenoxide In_2O_3 on InN seems to be possible. Our new methods give the energy gaps of the different In_2O_3 polymorphs and explain the so-called indirect gap by interconduction band transitions [7]. The band alignment using the branch-point energy yields band discontinuities of a type-I heterostructure [8].

Lectures

Wednesday morning, 16 September, 9:00
Room 226

9:15 Invited oral

In-segregation induced anomalous behavior of band gap and its pressure coefficient in InAlN and InGaN. Theory and Experiment

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It is believed that indium cations play a specific role in In containing nitride compounds. It was proved that In-addition, even in a small amount to nitride compounds leads to an enhancement of light emission intensity in LEDs and LDs and many works were devoted to explain this phenomenon. The most popular model dealing with this subject was proposed by Chichibu and Nakamura. It relates an enhancement of light emission with In-segregation effect. There exists a variety of results that confirm effects of fluctuations in In distribution in nitride alloys. Transmission electron microscopy, cathodoluminescence, and microphotoluminescence demonstrate wide spread of the In-segregation scale, from few nanometers to micrometers. In spite of a very intensive studies, the issue of the spatial and energetic scale of In-fluctuations is still not well understood.

In this work we are interested in InAlN and InGaN alloys. We present results of ab-initio calculations showing a strong modification of the band gap, E_G and its pressure coefficient, dE_G/dp, as a function of In-content and In-segregation rate (in nanometer scale). The unusual decrease in both the band gaps and their pressure coefficients with In content is observed in the case of clustered In arrangements. We compare theoretical results with the experimental data on E_G and dE_G/dp variations with In content.

An explanation of the observed phenomena is proposed on the basis of density of states and bond lengths analysis. We found that the unusual behavior of E_G and dE_G/dp as functions of In concentration comes from the In-induced changes of the states at the valence band top. It relates to the increase of the valence band width due to (hybridization) admixture of In *p* and *d* states into the uppermost N-originating valence states. The detailed analysis of the lattice relaxation shows that strong interaction of In and N- states is related to the bonds between In and N-atoms, being shorter in the clustered InAlN

and InGaN alloys than in InN itself.

9:45 Keynote lecture

Sources of doping for InN bulk and surfaces

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InN exhibits a high tendency for unintentional *n*-type conductivity, both in the bulk and on the surface. We have used first-principles calculations based on density functional theory to investigate various possible causes for the doping. Our results indicate that native point defects are unlikely to be the source of conductivity. Instead, attentions should be focused on unintentional incorporation of impurities.

In particular, hydrogen has a low formation energy in InN and acts as a shallow donor [1]. Interstitial hydrogen is stable in the bond-center configuration, with a H-N stretching vibration at 3050 cm^{-1}. Hydrogen can also substitute for nitrogen in InN, bonding equally to the four In nearest neighbors in a multicenter-bond configuration (a highly unusual type of chemical bond). Substitutional hydrogen, somewhat counterintuitively, is a double donor. In addition to the bulk conductivity, accumulation of electrons has been almost universally observed on InN surfaces. While donor impurities adsorbed on the surface could of course contribute to this conductivity, we have proposed that the accumulation layers are an intrinsic property of the material that can be attributed to the fact that on polar surfaces occupied surface states are located above the conduction-band minimum (CBM). Fermi-level pinning occurs due to occupied surface states above the CBM, for all In/N ratios, thus explaining the observed electron accumulation [2]. Interestingly, we have found an absence of electron accumulation on *nonpolar* surfaces of InN at moderate In/N ratios, a prediction that has been experimentally confirmed. Consequences for growth as well as for devices will be discussed.

This work was performed in collaboration with A. Janotti, J. L. Lyons, and D. Segev.

[1] A. Janotti and C. G. Van de Walle, Appl. Phys. Lett. **92**, 032104 (2008).

[2] D. Segev and C. G. Van de Walle, EuroPhys. Lett. **76**, 306 (2006).

COFFEE

Wednesday morning, 16 September, 10:30
Main Hall

Session A8/RW2

Growth and Properties III
Wednesday morning, 16 September, 10:50
Room 226
Chair: M.A. Poisson/C. Wetzel

10:50 Keynote lecture

Recent advances and challenges in p-type doping of InN and InN-based novel nanostructures

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InN is still the least studied material among III-nitrides and there are several problems to be overcome for better understanding of its material properties and also for its device application. Bonding between In and N is weaker and longer than those for GaN and/or AlN, and this results in large mismatches for InN in both lattice parameters and optimum epitaxy temperatures among III-nitrides. Another important issue for InN epitaxy is a big expected difficulty in achieving p-type conduction, because the Fermi level stabilized energy for InN is located inside the conduction band, resulting in easy introduction of donor levels together with high density surface electrons

In this paper, we report the latest advances in InN epitaxy and material control paying special attention to the following two important issues:

1) P-type doping of InN and InGaN: Reduction in the residual donors/defects concentration and successful p-type doping, together with accompanying problems arising from Mg-acceptor doping as well as their very special characterization methods inherent to InN properties.

2) Novel InN/GaN MQWs and very detailed in-situ epitaxy analyses with SE (spectroscopic ellipsometry): Proposal, realization and characterization of novel InN/GaN MQWs consisting of around 1ML-thick InN wells in GaN matrix, together with in-situ and real-time SE characterization of self-limiting and self-ordering deposition processes resulting in 2ML, 1ML, and fractional ML InN wells in GaN matrix at extremely high temperatures under the In-polarity growth regime.

For achieving p-type conductivity in InN and In-rich InGaN ternary alloys, on the basis of careful understanding on the residual donors in unintentionally n-type doped high-quality InN, we have achieved successful p-type doping of InN after a systematic doping study for very wide range [Mg]s. Edge-component threading dislocation density in typical InN epilayers grown on GaN is about 10^{10} cm^{-2} which is originating from their large lattice mismatch. Edge-type dislocations induce donor levels above the conduction band bottom in InN and they are one of the residual donors in the orders of 3.5×10^{17} cm^{-3} density. Successful p-type doping of InN was achieved for Mg doping levels from 10^{18} to about 3×10^{19} cm^{-3}. It was found that overdoped Mg's introduce new donors in InN resulting in n-type conduction. We analyzed the behaviors of vacancy-type defects in those InN samples with different Mg concentrations by using positron annihilation experiments/analyses, and it was confirmed that complex defects including In and multiple-N vacancies are responsible for newly introduced donors in Mg-overdoped InN samples. It was also found that the polarity inversion from In-

polarity to N-polarity took place for Mg-overdoped InN samples, though the polarity was kept unchanged in N-polarity growth in very wide Mg concentrations.

As for the novel InN/GaN MQWs, InN-based QWs consisting of coherent 1ML-thick InN wells embedded in GaN matrix were proposed and fabricated. It was confirmed that very fine structure InN QWs were fabricated by self-limiting growth mode under In-polarity growth regime at remarkably higher growth temperatures up to 690 ^{o}C. From the detailed deposition-analyses of In metal as well as InN with using SE in wavelengths from 250 nm to 1700 nm, it was found that both In and/or InN deposition was self-limited to 2ML or below at such high temperatures. Their optical properties are determined by GaN-excitons localized at InN wells. Experimental LED/LD structures were fabricated and it was confirmed that QCSE was ultimately reduced due to ultimately thin 1ML InN wells.

LECTURE

Wednesday morning, 16 September, 11:35
Room 226

11:35 Invited oral

Interface, bulk and surface electronic properties of InN

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A three-region model of the high *n*-type conductivity in InN will be presented, including contributions from the bulk, interface with the buffer layer and surface of the InN films. In particular, a parallel conduction analysis is used to show that this model can account for the variation of both the Hall effect-measured electron concentration and the mobility with film thickness. For a set of In-polarity InN samples grown on GaN buffer layers under the same conditions (V/III ratio and temperature), as the film thickness is varied from 200 to 12000 nm, the electron density changes from 2×10^{19} cm^{-3} to 3×10^{17} cm^{-3} and the mobility from 300 cm^{2}V^{-1}s^{-1} to 2000 cm^{2}V^{-1}s^{-1} [1,2]. Similar results have also been observed for InN films grown on AlN buffer layers [3]. The findings of additional studies of the effects of the V/III ratio during growth on the interface-related electron density will also be presented. Finally, our results will be placed in the context of recent data from other groups and the origins of the interface, surface and bulk conductivity will be discussed.

[1] L. F. J. Piper, T. D. Veal, C. F. McConville, H. Lu, and W. J. Schaff, Appl. Phys. Lett. 88 (2006) 252109.

[2] P. D. C. King, T. D. Veal, C. F. McConville, J. Phys.: Condens. Matter 21 (2009) 174201.

[3] V. Cimalla, V. Lebedev, F. M. Morales, R. Goldhahn, and O. Ambacher, Appl. Phys. Lett. 89 (2006) 172109.

12:05 Invited oral

Progress in the MBE Growth of InN

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We studied the effect of different deposition conditions on the properties of multiple InN orientations grown by plasma-assisted molecular beam epitaxy (PA-MBE). We found that the optimized growth conditions for In-face, N-face, m-plane, and a-plane oriented InN films generally include an In-wetting layer on the InN surface during growth (III/V ratio ≥ 1). Optimized InN growth temperatures for the different orientations, however, were somewhat different. N-face InN had the lowest thermal decomposition and was deposited at the highest growth temperatures (up to 620°C) compared to the other InN orientations. In-face InN had optimal deposition temperatures somewhat lower than N-face InN (~ 470°C), with complete thermal decomposition around 500°C. Finally, both the non-polar m- and a-plane InN orientations were grown at about 400°C and showed no growth beyond 430°C.

The origin of bulk electron concentrations in In-face InN was studied by considering the effects of both unintentionally incorporated impurities and threading dislocation densities on the electron transport properties of InN. The concentrations of the unintentionally incorporated impurities oxygen and hydrogen scaled with the bulk electron concentrations while TDs had no observed effect on the electron concentrations. We conclude that impurities were the significant source of electrons and TDs acted only as scattering centers limiting the mobility of electrons in InN. Using the optimized growth conditions, unintentionally doped In-face InN films have been realized with record room temperature as-measured electron mobilities as high as 2900 cm^{2}/Vs (3750 cm^{2}/Vs bulk electron mobility as determined by 2-layer Hall analysis) and free electron concentrations as low as 2.3 x 10^{17} cm^{-3} (6.6 x 10^{16} cm^{-3} bulk concentration by 2-layer Hall).

The optical properties of the different InN orientations were measured by reflectance, transmission, and photoluminescence (PL). The measured band gap values for the different InN orientations were in good agreement and ranged from 0.65 eV to 0.70 eV. The PL peak wavelength was red-shifted by ~70meV with respect to the band gap energy for m- and a-plane orientations, and only ~25meV for the c-plane orientations. This disparity is most likely due to increased band tail states from stacking faults and increased impurity incorporation for the non-polar samples due to the lower growth temperatures.

LUNCH

(maps available at the Reception desk)
Wednesday afternoon, 16 September, 12:30
building of the Faculty of Physics

Session A9/RW3

Devices and Properties of nitrides semiconductors
Wednesday afternoon, 16 September, 14:00
Room 226
Chair: A. Cavallini/P. Ruterana

14:00 Keynote lecture

New Twists in LEDs and HFETs based on Nitride Semiconductors

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Despite notable progress in light emitting and transport based devices in GaN heterostructures, there is still controversy as to the light emission characteristics at high injection levels in LEDs and hot carrier scattering in FETs. For LEDs to be inserted into conventional lighting systems, reasonably high efficiencies would have to be maintained at high injection levels to meet intensity requirements which is not borne out by experiments. The efficiency degradation at high injection levels, beyond that which is expected due to heating, has been attributed to mainly Auger recombination and carrier spill over. In FETs, the conventional wisdom of increased carrier concentrations leading to better devices does not seem to hold beyond a certain point. This is due to strong electron LO phonon coupling in this highly ionic material which leads to hot phonon population. Hot phonon lifetime decreases with increasing carrier concentration up to a point owing to Plasmon phonon interaction. But beyond the frequency at which the plasma frequency and phonon frequency resonate, the phonon lifetime begins to increase again. Increased phonon lifetimes lead to reduced carrier velocity and thus performance degradation ensues. Another intriguing feature is that the aforementioned phenomenon is electric field dependent at least because increased field in FETs means widening of the channel and thus for the same volume density the resonance occurs at higher sheet densities.

The details of carrier recombination in the context of LEDs at high injection levels, and hot carrier scattering related physics in the context of HFETs based on the GaN material family will be elucidated.

14:45 Keynote lecture

Capacitance and surface voltage charge techniques. Surface Photovoltage Spectroscopy. Method and Applications.

Daniela Cavalcoli

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The investigation of surface space-charge properties (charge depletion, accumulation or inversion etc..), defect states (which have a detrimental effect on doping) and electronic properties of InN-based quantum wells and heterostructures (electronic transport at interfaces, quantum confinement) are among the main objectives of the RAINBOW Project.

Surface photovoltage spectroscopy (SPS) is a powerful method for obtaining a detailed picture of the electronic structure of surfaces, defects and interfaces [1]. In SPS, changes in band bending (both at the free semiconductor surface and at buried interfaces) are monitored as a function of external illumination. Surface photovoltage spectroscopy can provide detailed, quantitative information on bulk properties (e.g. bandgap and type, minority carrier diffusion length and lifetime) and can be used for a complete construction of surface and interface band diagrams, including the measurement of energy levels in quantum structures. A particular strength is that a comprehensive analysis of surface and bulk defect state distributions and properties is made possible. Measurements using SPS are contactless and non-destructive.

In the present contribution the relevant surface and interface theory, the physical principles of the method and the experimental details will be discussed, together with several applications concerning a wide variety of materials, alloys and low dimensional semiconductor structures.

[1] L Kronik and Y Shapira, *Surface photovoltage spectroscopy of semiconductor structures: at the crossroads of physics, chemistry and electrical engineering*, Surf. Interface Anal. 2001; 31: 954–965.

COFFEE

Wednesday afternoon, 16 September, 15:30
Main Hall

Session A10/RW4

Optical Properties of nitride semiconductors
Wednesday afternoon, 16 September, 15:50
Room 226
Chair: F. Bechstedt/C. Trager Cowan

15:50 Keynote lecture

Optical properties of low-dimensional nitride semiconductors

Raphaël Butté

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The main optical properties of III-nitride (III-N) heterostructures will be reviewed. We shall first define spontaneous and piezoelectric polarizations which are inherent to III-N semiconductors crystallizing with the wurtzite phase [1]. It will be shown that polarization discontinuities are responsible for the built-in electric field present in III-N heterostructures grown along the *c*-axis [2]. The physics will be illustrated by considering low Al content GaN/AlGaN quantum wells (QWs) [3], which exhibit a quality allowing probing their intrinsic properties. In particular, we shall describe the experimental signature of the quantum confined Stark effect (QCSE) [4] and the subsequent decrease of the radiative efficiency of QWs with increas-

ing well thickness. The observation of well-resolved A and B excitonic resonances in the reflectivity spectra of high quality GaN QWs [5] and the biexcitonic properties of the latter [6] will be treated. The possible origins explaining the high quantum efficiency of In-based III-N QWs, which are presently at the heart of the huge success of UV-blue III-N optoelectronic devices, will also be discussed [7]. Finally, the concept of non- and semi-polar QWs, where the QCSE is either suppressed or decreased, for the realization of more efficient light-emitters will be introduced [8].

[1] F. Bernardini, V. Fiorentini, and D. Vanderbilt, Phys. Rev. B **56**, R10024 (1997)

[2] See, e.g., R. Butté and N. Grandjean, in *Polarization Effects in Semiconductors*, ed. by C. Wood and D. Jena (Springer, New York, 2008)

[3] E. Feltin *et al.*, Appl. Phys. Lett. **90**, 021905 (2007)

[4] M. Leroux *et al.*, Phys. Rev. B **58**, R13371 (1998)

[5] F. Stokker-Cheregi *et al.*, Phys. Rev. B **79**, 245316 (2009)

[6] F. Stokker-Cheregi *et al.*, Phys. Rev. B **77**, 125342 (2008); *ibid.*, Appl. Phys. Lett. **93**, 152105 (2008)

[7] See, e.g., S. Nakamura, Science **281**, 956 (1998); S. Pimputkar *et al.*, , Nature. Photon. **3**, 180 (2009)

[8] P. Waltereit *et al.*, Nature (London) **406**, 865 (2000)

Joint Poster Session

Monday & Wednesday
Wednesday afternoon, 16 September, 16:00
Main Hall

LECTURE

Wednesday afternoon, 16 September, 16:35
Room 226

16:35	Keynote lecture	

Optical properties of bulk-like nitride semiconductors

Ruediger Goldhahn

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The first part of the talk provides an overview how to determine a reliable complex dielectric function (DF) of a material from spectroscopic ellipsometry studies covering the whole spectral range between infrared and vacuum-ultraviolet. Then, the peculiarities of the spectral dependence of the DF for nitride semiconductors with zinc-blende and wurtzite crystal structure will be discussed in detail. It is demonstrated that the inclusion of electron-hole interaction becomes very important for correlating the experimental results with one-electron band-structure calculations, i.e. for determining transition energies for critical points of the band structure. The valence-band ordering at the center of the Brillouin zone has a strong impact on the optical anisotropy of hexagonal compounds around the band gap. Recent results, obtained from measurements on non-polar surface orientations, demonstrate the main aspects. Many-particle effects such as band-gap renormalization and band-filling lead to an energy shift of the absorption edge for high carrier concentrations. Examples for the DF analysis under those conditions will be presented in the final part.

Thursday, 17 September

RAINBOW Worshop - 2nd day

Thursday morning, 17 September, 9:00

Posters

Monday, 14 September

Joint Poster Session

Monday & Wednesday
Monday afternoon, 14 September, 16:00
Main Hall

16:00	Poster	A-1

Thickness dependence of electrical properties of c- and a-plane InN films

Adebowale O. Ajagunna, Eleftherios Iliopoulos, George Tsiakatouras, Katerina Tsagaraki, Maria G. Androulidaki, Alexandros Georgakilas

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The structural and electrical properties of c-plane and *a*-plane InN thin films, as a function of thickness, are investigated and compared in this work. All samples were grown under same conditions on c- and r-plane Al_2O_3 substrates with RF-MBE, using GaN buffer layers. The epitaxial growth of a-plane InN proceeded through the nucleation, growth and coalescence of 3D islands, resulting to increased surface roughness with increasing film thickness. Step-flow growth mode was maintained during the growth of c-plane films. The rms roughness determined for AFM 2 x 2 μm^2 scans was 0.3 nm and 5.9 nm for 500 nm thick c- and a-plane films, respectively. However, the a-plane InN films did not exhibit any structural or surface morphology anisotropy, which is typical for a-plane GaN. The lowest to date XRD (11-20) rocking curve (RC) FWHM of 1440 arcsec is reported for a-plane InN. The sheet electron density (N_s) and mobility were determined by single magnetic field Hall-effect measurements and exhibited a similar dependence on thickness for both c- and a-plane InN films. The measured N_s exhibited a minimum of ~ 2 x 10^{14} cm^{-2} for 100 nm thickness. It increased linearly with increasing thickness above 100 nm but it also increased with decreasing thickness below 100 nm. The analysis of the Hall-effect measurements, by considering the contribution of two conducting layers, indicates a

similar accumulation of low mobility electrons with $N_s > 10^{14}$ cm^{-2} at the films' surface/interfacial region for both the a- and c-plane InN films. The actual electron mobility of the InN bulk increases with thickness for both orientations as a result of improving structural quality.

Acknowledgement: Work supported under MRTN-CT-2004-005583 (PARSEM) and the Greek State Scholarships Foundation (IKY).

16:00	Poster	A-2

The influence of surfactant and the nucleation process to InN growth by metal-organic vapor phase epitaxy

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InN and group III-nitride materials have attracted great interest due to applications for optoelectronic devices because their band gaps cover range from ultra-violet to near infrared spectrum. The growth of high quality InN is still a challenge due to the lack of suitable lattice-matched substrates and low growth temperature required. The high equilibrium nitrogen vapor pressure and the low decomposition rate of ammonia result in a narrow growth window. Antimony (Sb) as a surfactant for InN growth may help to relief some of these constrains. Due to the low solubility of nitrogen in InSb and the low melting point of InSb (about 527 ^{o}C), a floating InSb layer can act as a protective layer on the InN surface to prevent nitrogen desorption. Simultaneously, the InSb layer may improve lateral transport and therefore smoothen the surface.

We have studied Sb incorporations during metal-organic vapor phase epitaxy (MOVPE) InN growth. A three-step growth process was employed to produce InN layers: nitridation, nucleation, and buffer growth. The nucleation process is a key to reduce defect densities in InN layers and thus was investigated before without Sb. The influence of Sb on these processes was systematically studied, especially for the optimum trimethylantimony (TMSb) fluxes and the most suitable sequence to introduce TMSb. Structural, optical, and electrical properties of the grown InN layers were investigated using high-resolution X-ray diffractometry, atomic force microscopy, and photoluminescence.

16:00	Poster	A-3

InN dielectric function from the midinfrared to the visible range

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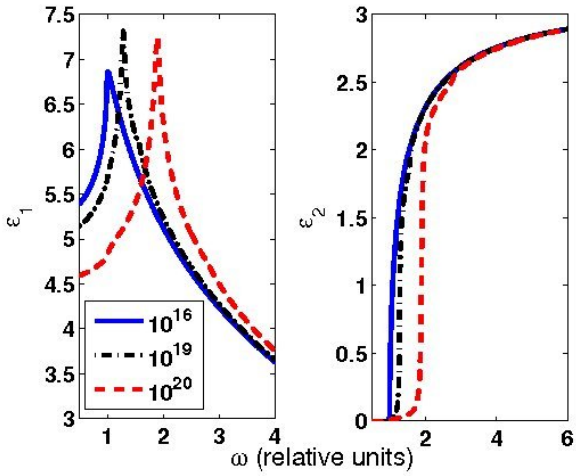
For semiconductors and semimetals possessing a narrow gap between bands with different parity, the dispersion of the dielectric function (DF) is analytically evaluated in the region near the fundamental energy gap. The pristine as well as doped semiconductors with degenerate carriers are considered. The imaginary part ε_2 of the dielectric function (shown in Fig.) has a plateau far away from the absorption onset for the interband electron transitions. For doped samples with degenerate carriers, the real part ε_1 of the DF is logarithmically divergent at the absorption threshold. This divergence is smeared with the temperature or the collision rate. The singularity results in the large contribution into the dielectric constant for pristine semiconductors. Without any fitting parameters, our results agree very well with the ab initio calculations and with available experimental results for wurtzite InN.



The real and imaginary parts of the dielectric function of wurtzite InN versus the photon frequency (in units of the gap ε_g = 0.69 eV), for various free-electron concentrations, corresponding values of the chemical potential μare 1.01, 1.57, 2.79 (in units of ε_g); relaxation rate is 0,01μ.

16:00	Poster	A-4

Theoretical study of the adsorption of InN on the GaN (0001) surfaces

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From more than ten years ago, nitrides have been attracting the attention of reserachers because of their applications; they have been used to fabricate light emmiting diodes and lasers, among others devices. In general, the nitrides are grown in the wurtzite phase, however it is also possible to find metastable zinc blende strucutre. Recently, special attention has been paid to InN due to its potential applications.However, nowadays it is a bit diffuclt to grow InN of high quality because current groth procedure are still not fully satisfactory. In this sense, further detailed investigations of the growth mechanisms as well as the exploration of new apporaches, can both contribute to a better understanding and control of the growth characteristics of this material. Motivated by these facts, in this workwe used first principles density functional theory (DFT) calculations to investigate the basic mechanisms of the growth of InN, in particular the adsorption and disffusion of In and N atoms on the GaN (0001) surface. The is modeled with the repeated slab geometry, each slab

consisting of four GaN bilayers and an atom on top of the surface. The botton surface was saturated by fractional pseudohydrogen atoms, while consecutive slabs were separated by an empty space. The saturating hydrogen atoms and the lowest GaN bilayer were fixed to simulate a bulklike environment. The other three bilayers and the adsorbed atoms are set free to move. The most stable atomic structure corresponds to the T4 configuration.

16:00	Poster	A-5

Electron microscopy of InGaN nanorods spontaneously grown on Si (111) substrates

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InGaN nanorods (NRs) for optoelectronic applications were grown on Si(111) by nitrogen radio-frequency plasma source molecular-beam epitaxy (rf-MBE). In order to detect their structural features, nanostructures were characterised by transmission electron microscopy (TEM) methods. Samples were prepared in cross-section orientation with the standard mechanical grinding process, followed by ion-thinning to electron transparency, without harvesting the NRs from the substrate.

Observations showed that the initial nanostructures starting from the GaN/Si interface were narrow NRs, having widths of the order of 10-30 nm, where two or three or more of them later merge to finally form NRs with widths in the range of 20-120 nm, the majority being at 50-70 nm. A few of the narrow NRs continued to grow higher than the merged ones and did not join together to form wider NRs, but they were actually linked to the wide NRs with low-angle grain boundaries. These NRs reached heights of nearly 1 µm, over twice the heights of merged NRs that were measured within 300-430 nm. A trapezoid morphology of the tips of NRs was observed. Basal stacking faults (SFs) with a density of 2.5×10^5 cm^{-1}, were the major structural defects detected in the majority of NRs, whereas they seem to be far less in the long NRs.

Electron diffraction analysis showed an average (110)Si/(11-20)InGaN, [1-11]Si/[0001]InGaN orientation relationship between the two lattices, where the axial direction of most of the NRs deviated $\pm 3\text{-}4^{o}$ around the growth direction. The influence of the interfacial structure on the divergence from the exact epitaxial orientation between the two crystal structures is examined.

Acknowledgement: Work supported under the MRTN-CT-2004-005583 "PARSEM" project.

16:00	Poster	A-6

TEM investigation of processed InGaN based laser grown by PAMBE on bulk GaN substrate

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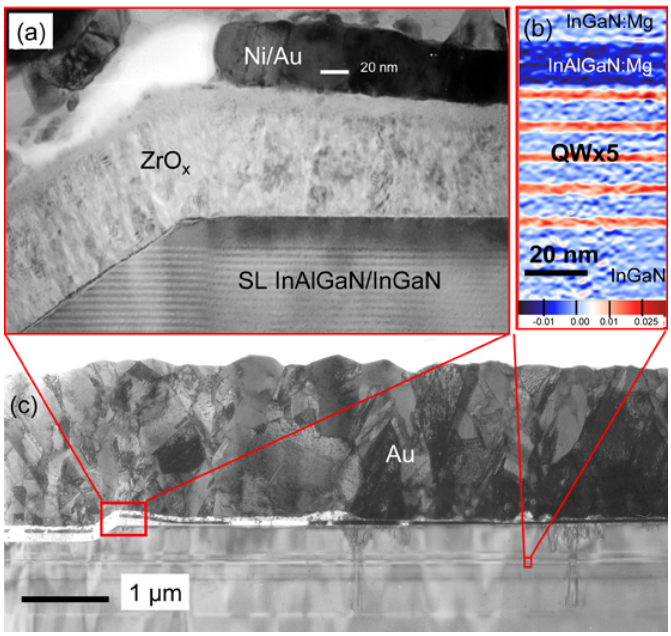
The structural investigation of the processed InGaN/GaN LDs and LEDs structures are performed by the Transmission Electron Microscopy. A PEMBE process is used to fabricate laser structures on high pressure bulk GaN substrates [1]. The indium composition inside the active MQW region is measured by the analysis of the local lattice distortion using high resolution HRTEM Images. The characterization of different types of structural defects of the structures is given in this paper.



The figures above show the cross-section of a laser structure with the top ohmic contact, after the functional tests. Over the mesa region the remaining ZrO_x mask can be seen (figure a). Local lattice distortion maps in the active region of $In_{x1}Ga_{1-x1}N/In_{x2}Ga_{1-x2}N$ MQW lasers (figure b) show high homogeneity of the indium composition along individual QWs, as well as between them. The low magnification TEM image (figure c) shows a part of the processed laser structure. The threading dislocations, which start at the interface between the bulk GaN and the Si doped GaN and the defects in the Mg doped top cladding are detected. These defects can be related to the discontinuities of the Ni layer. The local heating caused by the local non-uniform density of the current can be the origin of this defect creation.

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16:00	Poster	A-7

The instability of the MOVPE InGaN in the 20at% indium range studied by transmission electron microscopy.

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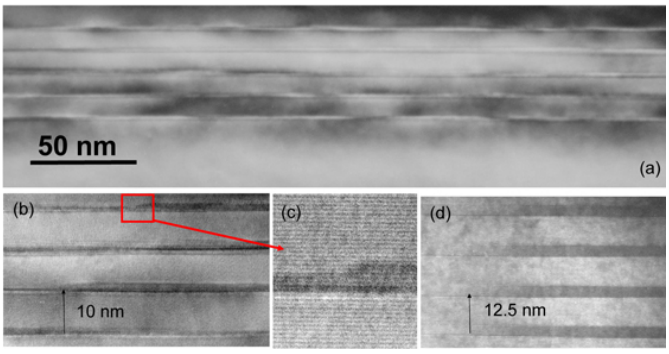
The InGaN/GaN:Si multiple quantum well (MQW) samples used in this study were grown by low-pressure metal-organic chemical vapor deposition (LP-MOCVD) on high-pressure grown bulk GaN mono-crystalline substrates. Two InGaN/GaN samples were investigated. One of them was grown at 780°C (sample A) . In the case of the second (sample B), the GaN barriers were grown at 900°C, but InGaN at 730°C.

For both samples the indium composition in the QWs reaches the level of about 20at%. The local indium composition was measured through strain measurements by digital processing from the lattice fringes' images of Transmission Electron Microscopy. We compare the analysis performed for two zone axis [11-10] and [11-20] with the use of axial and off-axis illumination. During TEM investigations the formation of "false indium clusters " of the size of 2-4 nm was observed for both samples just after 2 minutes of LaB_6 electron beam illumination at 200kV. Figures (a) and (b) show the cross-section of sample B.



These images have been taken in the first seconds of illumination and they clearly show the inhomogeneous thickness of the QWs. These fluctuations have a length of 53 nm and the QW thickness changes from 2.5 nm to 0.5 nm .The maximum composition reaches 23% of indium, but can drop below 5% in the "valleys". The abrupt 1 nm high step of the width QW is shown in figure (c). Sample A, which barriers and wells were grown at 730°C, shows a perfect lateral homogeneity with 3 nm thick QWs and an indium composition of 19.5at%. The QW with the mentioned modulations for sample A, can be explained by the desorption of indium during the 30s rise of the temperature necessary for the growth of the GaN barriers.

The work was partly supported by the EU project ITN RAINBOW 213238-2 and Polish Scientific Network project No. 71/E-64/BWSN-0108/2008.

16:00	Poster	A-8

Role of threading dislocations on Indium distribution in InGaN alloys

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Despite the presence of high density threading dislocations, InGaN alloys and quantum wells still exhibit high efficiency light emission, and this has been subject of research interest from the scientific and industry fields for the last few years. This peculiar behavior has been related to the properties of the threading dislocations and the localization of excitons. One of the explanations to the exciton localization has been the possible formation of In-rich clusters. Theoretical investigations have pointed out that a miscibility gap exists in the InGaN ternary alloys grown under equilibrium conditions. Therefore, if the growth temperature is below a critical temperature, InGaN alloys are due to be unstable over most of the composition range and In-rich and Ga-rich regions may form in the quantum well areas. However, number experimental reports have shown that down to the nanometric scale, In-rich clusters may not exist in InGaN high quality QWs. In fact, it appears that In clustering can be suppressed by strain in InGaN/GaN heterostructures as reported in recent experimental and theoretical investigations. However, phase separation has been found to nucleate with the aid of the threading dislocations having screw component in InGaN films [1], whereas the nucleation of InN quantum dots grown by MBE [2] and MOCVD [3] are associated with threading dislocations with edge component from the GaN buffer layer. In this work, as the In-rich clusters and threading dislocations involve hundreds to thousands of atoms, the empirical Stillinger-Weber potential is used to investigate the energetic stability of In-rich clusters in InGaN QWs and the role of the screw dislocations on the phase separation. The results indicate that In-rich clusters are not energetically favorable; however, indium atoms are found to be able to aggregate towards the dislocation core and to form In-rich regions by the screw dislocation with a full core configuration. This is explained based on the relaxation of the dislocation core in GaN by indium atoms.

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16:00	Poster	A-9

Electrical and optical properties of Mg-doped InN

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The ability to reproducibly achieve *p*-type doping is a prerequisite for many device applications of InN. Straightforward characterization of *p*-type conductivity of Mg-doped samples using single-field Hall effect is prevented by the presence of a surface inversion space-charge layer, whereby the surface electron-rich region dominates the measurements. However, several different groups have recently reported that Mg doping during growth by molecular-beam epitaxy produces *p*-type conductivity [1-3]. The techniques that have so far been applied to studying Mg-doping of InN include electrochemical capacitance-voltage (ECV) profiling [1-3], variable magnetic field Hall effect [2], and thermopower measurements [4].

Here, an investigation of Mg-doped InN samples grown by plasma-assisted molecular beam epitaxy is reported. The Mg concentration ranged from 3×10^{17} to 1×10^{20} cm^{-3}, as determined by secondary ion mass spectrometry. The samples have been characterized by Hall effect, thermopower, ECV measurements, x-ray photoemission spectroscopy, infrared absorption and reflectivity. The dependence of the experimental data on the Mg concentration will be reported, giving insight into the impact of the surface inversion layer on characterization of Mg-doped InN.

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16:00	Poster	A-10

An effective numerical computation of hot-electron transport in wurtzite indium nitride

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For a long time, the band gap width of around 2 eV has been assumed for wurtzite InN. However, recent experimental and theoretical investigations (see [1]) have provided convincing evidence that the band gap of InN is actually close to 0.7 eV. Thus, the alloy $In_xGa_{1-x}N$ formed of InN and wideband GaN is promising for the use in optoelectronic applications [2]. The hot-electron transport in bulk *n*-InN was numerically studied several times using the Monte-Carlo approach (for example see papers [3,4]) for the free electron concentrations near to $N=10^{-17}$ cm^{-3}. However, no comparison with experimental data was presented in these papers.

In our work a new iterative method for solution of the Boltzmann transport equation is introduced. Using this method one can find the stationary distribution function as well as it evolution. The method can be used in the wide range of conditions (including the degenerated electron gas statistics), it has no fitting parameters, it can take all relevant scattering mechanisms into account. In this paper the introduced method is applied to the hot-electron transport in *n*- InN. According to the experiment [5], we used in our numerical simulation the lattice temperature $T = 77$ K and the free electron concentration $N = 9 \cdot 10^{18}$ cm^{-3} so that the electron gas degenerates. Calculations are performed for all relevant sets of InN material parameters and the results are compared with the experimental data [5]. It is worth noting, that the "old" set of the material parameters (i.e. with the wide band gap) provides best fit of the experimental data.

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16:00	Poster	A-11

Investigation of optical nonlinearities in InN layers by using time-resolved differential transmission and light-induced transient grating techniques

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Picosecond dynamics of light induced optical nonlinearities was investigated in InN layers over wide range of temperature and excitation using complementary time-resolved pump-probe techniques. Picosecond light-induced transient grating (LITG) technique has been applied for investigation of nonequilibrium carrier diffusion and recombination in T=15K-300K range. Femtosecond differential transmission (DT) technique provided kinetics of induced bleaching and free carrier absorption in 1550-2300 nm spectral range. The measurement were carried out in two (2.3 μm and 0.6 μm thick) InN lay-

ers grown on sapphire with background electron density of 1.4×10^{18} cm^{-3} and 4.7×10^{18} cm^{-3}.
LITG experiments provided novel features of non-equilibrium carrier recombination in highly excited InN ($\Delta n>10^{18}$ cm^{-3}). The recombination rate was found strongly nonlinear and followed the dependence $1/\tau_R$ □B*(n0+N) which was nearly independent on temperature down to 40K. These features allowed us to suggest a trap-assisted Auger recombination being the dominant recombination mechanism in InN. The modelling provided a value of trap-assisted Auger recombination coefficient $B^*=8\times10^{-10}$ cm^3s^{-1}.
DT kinetics at fixed pump energy and various probe wavelengths revealed the spectral range, where the induced bleaching was dominating (for wavelengths above the absorption edge at ~0.7eV). By increasing probe wavelength, we observed a gradual transfer from the optical bleaching ($\Delta\alpha<0$) to the induced free carrier absorption ($\Delta\alpha>0$ at $\lambda p>2260$ nm) as well as the intermediate spectral region when these DT effects nearly cancelled each other. Measurements of free carrier absorption kinetics and determined carrier lifetimes at various excitation energy densities confirmed the inverse dependence of lifetime vs total carrier density. Moreover, the latter data provided a value of $B^*=4\times10^{-10}$ cm^3s^{-1}, which is close to the one obtained by LITG technique.

16:00	Poster	A-12

Irradiation-induced defects in InN and GaN studied with positron annihilation

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Positron annihilation is an effective method for the investigation of vacancy-type defects in semiconductors. Positrons can get trapped at vacancies, resulting in observable changes in the characteristics of the annihilation radiation. The annihilation data can be used to determine vacancy concentrations as well as to distinguish between different vacancy types. In this work, we apply positron annihilation to investigate radiation-induced defects in InN and GaN grown by molecular-beam epitaxy. InN and GaN samples were irradiated with 2-MeV $^4He^+$ ions to fluences in the range 5×10^{14}–2×10^{16} cm^{-2}, and subsequently subjected to rapid thermal-annealing below 500°C. This has previously been found to result in increased electron concentrations and mobilities in the InN films.

Our results show that in InN, negative In vacancies are introduced in the irradiation at a significantly low rate of 100 cm^{-1}, with their concentration saturating in the mid-10^{17} cm^{-3} at an irradiation fluence of 2×10^{15} cm^{-2}. The annealing is found to result in efficient clustering of the In vacancies near the film-substrate interface, in good agreement with results on void formation obtained by transmission electron microscopy. This is completely opposite to the behaviour observed in GaN, where Ga vacancies are introduced in the irradiation at a much higher rate of 3600 cm^{-1} showing no saturation, but where no clustering or recovery is seen after annealing. The cluster formation suggests that a large number of N vacancies need to be present in the InN films after irradiation, possibly being the cause of the increased *n*-type conductivity. Also, it is likely that the introduction rate of In vacancies is much higher than observed, which could be evidence of a similar acceptor-to-donor transition as the $V_{Ga} \leftrightarrow (V_{As} + As_{Ga})$ transition in GaAs.

16:00	Poster	A-13

Could positrons be used to study interface properties in nitride devices?

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Semiconductor heterostructures are the core elements of electronic and especially opto-electronic semiconductor devices, such as light emitting diodes (LEDs) and laser diodes (LDs). Ideally it is the design of the structure that defines the properties of the device at hand, but in practice the structural quality of both the semiconductor materials themselves and the heterointerfaces between them often limit significantly the output. This is the case especially in device structures fabricated out of III-nitrides (AlN, GaN and InN) that are the material family of choice for blue and ultraviolet (UV) optoelectronics with a potential for covering the whole spectral range down to infrared (IR), due to the unfortunate aspect of having lattice constants that differ considerably one from another. Hence even if it was possible to grow the thin layers of materials, or more specifically their ternary alloys such as $In_{0.13}Ga_{0.87}N$, without defects in the bulk of the layer, the interface e.g. between the quantum well and the barrier in a LD is likely to have a complicated structure and will be prone to generate extended defects detrimental to the functioning of the device.

We explore the potential of positron annihilation spectroscopy in studying nitride heterostructures. In addition to the bandgap modulation that is essential for the electronic device, the use of different nitrides creates electric fields in the structure induced by spontaneous polarization due the non-inversionsymmetric wurtzite structure of the materials. We show that depending on the design of the heterostructure, both the interface regions and the defects inside the bulk materials in these structures can be studied with positrons. To this end we combine positron experiments in nitride materials and LED structures with state-of-the-art ab initio computational methods. without defects in the bulk of the layer, the interface between e.g. the quantum well and the barrier in a LD is likely to have a complicated structure and will be prone to generate extended defects detrimental to the functioning of the device.

16:00 Poster A-14

Morphological, structural and optical properties of high-quality InN on GaN-template films deposited by RF sputtering

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Poly-crystalline Indium Nitride deposited by reactive sputtering presents a room-temperature direct apparent optical bandgap around 1.8 eV, making it very attractive for optoelectronic applications like high-efficency low cost solar cells, optical coatings and optical sensors. In this work, we report on a detailed study of the influence of deposition parameters on the crystalline quality, surface morphology and optical properties of InN films grown on GaN-on-sapphire substrates by reactive radio frequency (RF) sputtering. InN samples present a wurzite crystallographic structure and a highly *c*-axis preferred growth orientation, as confirmed by X-ray diffraction measurements. In a pure N_2 atmosphere the growth conditions under study are the substrate temperature, RF power and N_2 pressure. The optical properties were studied through transmission measurements. The apparent optical bandgap was estimated close to 690 nm (1.79 eV) for InN samples deposited at 450ºC and 3.5 mTorr. For samples deposited at temperatures above 450ºC a slight blueshift of 0.02 eV of the bandgap was observed. The sputtering pressure seems to strongly influence the InN absorption edge due to a change of the species in the plasma composition with a damage of the InN surface. However, RF power does not appear to affect the bandgap position. The surface morphology of the InN films was analyzed by atomic force microscopy in a surface of 5x5 μm^2. The morphological quality of the InN samples was highly improved at the optimized conditions. At low substrate temperatures (400°C) and low RF powers (20W and 30W) high surface quality InN films were achieved, with root-mean-square surface roughness as low as ~0.4 nm, comparable to the surface roughness of the GaN-template substrate. Our results indicate that the reactive RF sputtering is a promising deposition technique for obtaining high quality InN layers on GaN-template substrates with low cost and good compatibility with other optoelectronic devices.

16:00 Poster A-15

Time-resolved differential transmission and photoluminescence studies of recombination mechanisms in Mg-doped InN

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InN is a narrow-gap (~0.65 eV) semiconductor which has generated interest for its potential in applications ranging from photovoltaics to terahertz detectors to high-speed transistors. The realization of p-type InN through in-situ Mg doping has recently been reported by a number of different groups [1-3]. Two distinct and unrelated phenomena initially interfered with attempts to determine whether doping experiments yielded p-type conductivity: a high density surface electron accumulation layer [4], and unexpected quenching of the photoluminescence [1, 2]. Verification of p-type material has since been performed using a variety of techniques including electrochemical capacitance-voltage, valence band x-ray photoelectron spectroscopy, and variable magnetic field Hall effect. The origin of the apparent photoluminescence quenching, however, has remained an open question. We have investigated recombination dynamics of Mg-doped InN epilayers grown by plasma-assisted molecular beam epitaxy using a combination of photoluminescence and time-resolved differential transmission (TRDT) measurements. The Mg concentration was determined by secondary ion mass spectrometry using an ion-implanted standard, and found to be in the range of 3×10^{17} to 1×10^{20} cm^{-3}. Photoluminescence was performed using an argon ion laser and either an InSb or InGaAs detector, resulting in detectable signals from all samples. TRDT was performed at room temperature using a pump-probe technique on three separate samples, with doping densities of 6.2×10^{17} cm^{-3} (SL), 3.6×10^{19} cm^{-3} (SM) and 1.0×10^{20} cm^{-3} (SH), respectively.As has been reported by others, the photoluminescence of the films generally consisted of a single peak, with lightly doped films typically exhibiting a single feature near the bandedge. Quantum efficiency decreased markedly with increased Mg content, and many of the moderately doped films were characterized by a lower energy peak near 0.6 eV. For TRDT sample SL, the decay was single-exponential, as would be expected from a combination of radiative and Shockley-Read-Hall recombination. Samples SM and SH, however, were characterized by a different type of decay, indicating that a different recombination mechanism dominates. Sample SL, which in many respects has the same characteristics of undoped (n-type) InN, does not exhibit this different decay behaviour until higher excitation.

Thus, Mg doping leads to a dramatic shift in the dominant recombination pathway, which has profound implications for devices.

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16:00	Poster	A-16

Structural properties of InAlN thin layers for HEMT applications

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The $In_xAl_{1-x}N$ material system is highly attractive for possible applications in high electron mobility transistor. Heterostructures InAlN/GaN, grown by metal organic chemical vapour deposition (MOCVD), have been investigated by transmission electron microscopy and atomic force microscopy and X-ray diffraction for determination of the residual strain. The TEM samples were prepared by standard mechanical polishing and Ar ion beam thinning at 5 KeV and 5° angle incidence. The nominal composition of the investigated samples is around the strain free 0.17% In composition. In these materials where the lattice parameters changes are so steep, the control of the growth parameters is always a challenge. A tight iterative characterisation of the growth process is still necessary in order to optimize it. Observations show that in this instance, in a same growth sequence, the composition of the layers may oscillate between 13-21% In composition. So, the control of the growth parameters is may not be so straightforward.

In this work, we have carried out extensive investigation, by XRD and TEM, of lattice matched AlInN thin and QWs layers grown by MOVPE. We will report on the changes in the structural properties at and around the lattice matched In fraction. The results show that above In=0.2, the MOVPE layers are not only polycrystalline, but also that complete phase separation may take place with XRD detecting pure InN.

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Symposium C

Wide band gap II-VI and III-V semiconductors

Welcome

The Symposium will cover the most important developments in this booming field of the basic and applied research. The main emphasis will be given to recent progress in growth methods and understanding of material properties, which allow wide spread applications of wide band gap semiconductors based on group-III nitrides and ZnO in a new generation of electronic and optoelectronic devices.

High scientific interest continues also in another application of wide band gap semiconductors, such as ZnO and GaN. These materials, when doped with transition metal ions, often show ferromagnetic response. During the Symposium the recent developments in this hot topic will also be discussed.

- GaN for short and long wavelength LD applications (far UV and green LDs)
- GaN for electronic applications and biological sensors
- GaN-based LEDs for white light sources
- Perspectives of spintronics based on wide band gap compounds
- Low temperature ZnO for electronic applications
- Growth techniques of wide band gap oxides and material characterization
- II-VI-based wide band gap semiconductors for optoelectronic and spintronics applicationsProgramme

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Proceedings

The proceedings of this symposium will be published in physica status solidi (c), with selected papers in physica status solidi (a) and (b).

Programme

Monday, 14 September

LUNCH

(maps available at the Reception desk)
Monday afternoon, 14 September, 12:30
building of the Faculty of Physics

Opening
Monday afternoon, 14 September, 14:30

Session 1
Monday afternoon, 14 September, 14:45
Room 231

14:45 Invited oral

Room temperature polariton lasing and condensation effects in III-nitride microcavities

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The recent demonstration of room temperature (RT) polariton lasing in a bulk GaN microcavity (MC) has established the possibility to use nitride semiconductors for the realization of polariton-based devices. Indeed, those structures could be used as novel low threshold coherent light emitting sources or micro optical parametric amplifiers. Unfortunately, achieving an efficient electrical injection within a bulk GaN active region is difficult. As a result the realization of an electrically pumped polariton laser based on such bulk samples should reveal highly challenging. A way to push forward polariton-based devices consists in achieving a well-grounded RT strong coupling regime (SCR) and demonstrating nonlinear effects in quantum well (QW) MCs for which the realization of an electrically pumped device should be more realistic. This would add to the recent demonstration of GaAs-based polariton light-emitting diodes. In this context, we have recently developed a structure using a GaN/AlGaN multiple quantum well (MQW) active region which allowed the demonstration of the SCR at RT with a record vacuum Rabi splitting value for a semiconductor QW MC (~ 56meV).

Here we report on the emission properties of this type of MCs under nonresonant optical pumping. It will be shown that the samples exhibit a nonlinear emission threshold at RT while remaining in the SCR. Experimental results will be depicted within the picture of polariton lasing and prospects regarding the realization of an electrically pumped polariton laser will be given. The behavior of the nonlinear emission threshold versus polariton trap depth and temperature, leading to the polariton condensation phase diagram, as well as that of the polarization of the emitted light below and above threshold will also be discussed. For the sake of comparison, if time permits, these results will be connected to those obtained on "standard" vertical cavity surface emitting lasers using InGaN/GaN QWs as an active region.

COFFEE
Monday afternoon, 14 September, 15:30
Main Hall

Joint Poster Session
Monday & Wednesday
Monday afternoon, 14 September, 16:00
Main Hall

Tuesday, 15 September

Session 2
Tuesday morning, 15 September, 9:00
Room 231

9:00 Invited oral

Cathodoluminescence spectroscopy of Lithium in-diffusion in ZnO

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Li doped ZnO has been investigated using temperature, depth and power-density resolved cathodoluminescence (CL) spectroscopy. The incorporation of Li was achieved by evaporating 50 nm of Li metal at ~ 1 mPa onto the surface of 1 mm thick, 5 x 5 mm a-plane ZnO sections (hydrothermally grown by the MTI Corporation) and then heating to 950ºC for 30 min without breaking vacuum. CL spectra of the as-received ZnO specimens consisted of the near band edge at 3.25 eV as well as three broad defect luminescence bands centred at 1.95 eV (orange, OL), 2.15 eV (yellow, YL) and 2.42 eV (green, GL). As the temperature was decreased from 300 K to 80 K the dominant GL disappeared giving rise to an OL around 170 K followed by an increase of the YL and reappearance of the GL with further cooling. This behaviour is in agreement with the repopulation of thermally ionised shallow donors in donor-acceptor pair (DAP) transitions and also implies that the GL can be attributed to a number of different defect centres. Following heat treatment, an intense broad OL peak centred at 1.96 eV was observed on the Li metal coated side but not the opposite face, which exhibited a weak GL peak. Depth-resolved CL demonstrated that Li in-diffusion had occurred to at least 3 µm (the electron range in ZnO at 30 kV). When cooling to 80K, the Li-related OL peak increased in intensity and no other CL peaks emerged. These results confirm that Li in-diffusion incorporates Li on Zn cation sites (Li_{Zn}^{O}) and that the OL originates from a radiative recombination of a free electron with a bound hole at a neutral Li acceptor. This assignment is consistent with the results of power-density resolved CL experiments where OL saturation and a slight red shift were observed with increasing beam current. The Li in-diffusion data also indicates that the OL in the as-received specimen arises from a DAP involving a Lithium acceptor and a shallow donor.

9:30 Invited oral

Zinc oxide grown by Atomic Layer Deposition - a material for novel 3D electronics

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Last years miniaturization of semiconductor devices encountered obstacles to scale below the 65 nm mode, because of a high parasitic current in a dominant floating gate architecture. Therefore in 2007 for the first time high-k oxides replaced silicon oxide, which enables scaling of the device to the 45 nm mode. However, further scaling below 22 nm mode requires changing of the device architecture from planar to three-dimensional. This imposes critical restrictions on growth and processing temperature as using of metal passes at the back of the line (BEOL architecture) limit processing temperature to 350 ^{0}C. The silicon technology requires processing temperature at the level of 1000 ^{0}C and cannot be used in this case. Therefore we observe a booming interest in semiconductor materials, which can be successfully grown at low temperature.

Our investigations show that zinc oxide is a very good candidate for a low temperature electronics. ZnO thin films are grown by Atomic Layer Deposition using organic compounds as zinc precursors. Thanks to these volatile precursors we were able to investigate a low growth temperature range between 1000C and 3000C. The obtained zinc oxide films are monocrystalline when deposited on gallium nitride substrate and growth temperature is higher than 2500C. We show that even without external doping a free carrier concentration in these films can be reproducibly controlled in the wide range (form 10^{15}cm^{-3} to 10^{21}cm^{-3}), which is a basic requirement for electronic applications. Our investigations show that ZnO grown at low temperature by ALD is a prospective material for novel low temperature electronic applications such as 3D structures and organic/inorganic heterojunctions.

The work was partially supported by EU within the European Regional Development Found through grant Innovative Economy No. POIG.01.01.02-00-008/08.

10:00 Oral

Unipolar transparent vertical diode based on ZnMgO/ ZnO heterostructure films

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Recently, oxide semiconductor materials have attracted much attention for their potential application in optoelectronic devices and transparent electronics. ZnO is one of the promising material because of its direct band gap (~ 3.3 eV) and large exciton binding energy (~60 meV). Although there have been many reports demonstrating ZnO based devices such as p-n junction ultraviolet-blue light emitting diodes and UV photodetectors, ZnO-based transparent thin film diodes has been less studied probably due to the difficulties in achieving p-type ZnO film. Here, we report on the fabrication of a unipolar transparent thin film diode based on ZnO through a heterojunction implementation, without invoking to p-type materials.

Since unipolar diodes, which are one of majority carrier devices, have a high switching speed, unipolar transparent thin film diodes can have many device applications in transparent electronics and nonvolatile resistive random access memories. Our ZnO-based unipolar vertical diodes utilize a heterostructure of ZnO and $Zn_{1-x}Mg_xO$ (ZnMgO) film of graded-composition. ZnO and ZnMgO heterostructure films were deposited on indium-tin-oxide coated glass substrate by using ultrasonic spray pyrolysis. The diodes showed excellent rectifying characteristics with the rectification ratio exeeding 10^3 in the range of applied volages between -3.0 and +3.0 V and turn-on voltage about 0.8-1.0 V. Potential barriers produced in the diode structures and controllability of the diode characteristics will be discussed in terms of the graded band gap of ZnMgO films.

10:15 Oral

Structural characterization of Indium-acceptor complexes in ZnO by PAC

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Difficulties in achieving reliable and reproducible p-type ZnO hamper its use for optoelectronic applications in the blue and UV region. A solution to the challenge of p-type doping might be the theoretically proposed codoping [1] or cluster doping [2] of donors and acceptors. The former method has been confirmed by Hall effect measurements in the case of In-N codoping of ZnO-films [3]. From theory the formation of In-acceptor complexes is predicted, thereby enhancing the solubility of the acceptor.

On an atomic scale, these defect complexes are distinguished by characteristic electrical field gradients (EFG) at the lattice site of the indium donor. Using perturbed γγ angular correlation spectroscopy (PAC), the presence of these EFG is detected at the site of the radioactive probe atom ^{111}In/^{111}Cd [4]. After applying different codoping procedures (diffusion and/or implantation) in case of the combinations In-N and In-P, the formation of In-acceptor complexes has indeed been observed via the occurence of additional EFG besides the $EFG_{lattice}$.

Each complex is characterized by two slightly different EFG as is expected because of the wurtzite structure of ZnO and the different nearest neighbor distances along the c axis and in the basal plane. The efficiency of the different co-doping procedures and their dependencies on the diffusion and annealing conditions will be discussed.

This work has been supported by the Bundesministerium für Bildung und Forschung (BMBF) under Contract No. 05 KK7TS2.

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[4] Th. Wichert, in: Stavola (ed.), *Identification of Defects in Semiconductors* (Semiconductors and Semimetals, Vol. 51B, Academic Press, London 1999) p. 297

COFFEE

Tuesday morning, 15 September, 10:30
Main Hall

Session 3

Tuesday morning, 15 September, 11:00
Room 231

11:00 Invited oral

Inter-subband optics at 1.55 µm in GaN-based nanostructures - Physics and applications

Maria Tchernycheva[1], Houssaine Macchadani[1], Laurent Nevou[1], Juliette Mangeney[1], François H Julien[1], Prem K. Kandaswamy[2], Alexander Wirthmüller[2], Eva Monroy[2], Alon Vardi[3], Shmuel Schacham[3], Gad Bahir[3], Gianmauro Pozzovivo[4], Sebastien Golka[4], Gottfried Strasser[4]

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Thanks to their large conduction band offset (up to 1.75 eV for GaN/AlN), III-nitride nanostructures in the form of quantum dots (QD) or quantum wells (QW) offer great prospects for ultrafast intersubband devices operating at room temperature in the near-infrared spectral range. Nitride intersubband devices are intrinsically very fast because of the subpicosecond absorption recovery time due to enhanced electron-LO phonon interaction in these highly ionic materials. In this talk, we will report on recent achievements in terms of GaN/AlGaN-based intersubband physics, devices and technology.

As a first example of QW intersubband device, we will show how electron tunneling between coupled quantum wells can be applied to the fabrication of fast electro-optical modulators at 1.3-1.55 µm wavelengths. Then, we will present recent experiments on a new type of ultrafast detectors based on the quantum cascade concept. Finally, we will discuss the progress towards near-infrared lasers and amplifiers based on the observation of intersubband light emission from GaN/AlN quantum wells at a wavelength as short as 2 µm. GaN/AlN QDs are also of great interest for intersubband devices, namely because of the achievable large QD density (a few 10^{12} cm^{-2}) and of the expected narrow linewidth of intraband transitions due to the slow-down of the dephasing mechanisms. We will describe recent resonant Raman scattering experiments which reveal an intraband light emission at a record short wavelength of 1.5 µm and allow estimating the intraband linewidth of single QDs. We have also performed pump-probe femtosecond measurements which show the extremely short intraband lifetime (165 fs) while the intraband saturation intensity (0.27 pJ/μm^2) is one order of magnitude smaller than that measured in QWs. These results open prospects for ultrafast saturable absorbers and all-optical switches based on GaN QDs for telecommunication applications.

11:30 Invited oral

Spontaneous and ordered growth of III-N nanorods on polar and non-polar substrates

Enrique Calleja[1], Javier Grandal[1], Sergio Fernandez[1], Miguel A. Sanchez[1], Jose M. Calleja[2], Eva Gallardo[2], Esperanza Luna[3], Achim Trampert[3], Uwe Jahn[3]

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The growth of self-assembled and ordered III-N nanorods by Plasma-Assisted MBE is addressed. The nucleation stage and the subsequent nanorod growth are explained considering the growth conditions and the lattice mismatch between the substrate and the nanorods. A strong lateral to vertical growth rate anisotropy is caused by a very fast ad-atom diffusion along the nanorods sidewalls. A significant change of this anisotropy occurs when Al atoms are incorporated to the growth (AlGaN nanorods), as it is clearly distinguished in TEM pictures. This effect, related to a slower Al ad-atom diffusion along the nanorod sidewalls, allow the formation of Quantum Dots (QDs) embedded within the nanorod (GaN/AlGaN).

Nucleation may proceed via Volmer-Weber or Stranski-Krastanov mechanisms depending on the initial lattice mismatch between the nanorod material and substrate. A high initial mismatch (i.e. GaN on bare Si) leads to isolated, well defined nanorods, whereas a smaller one (i.e. GaN on AlN buffered Si) produces a mixture of nanorods and a “faceted matrix” of continuous, rough material. A description of the process to be followed in order to avoid this “faceted matrix” is also presented.

Wurtzite InN nanorods have been grown along the c-plane, on Si(111) substrates, and along the a-plane on m-plane GaN/r-plane sapphire templates. In the second case, a well defined a-plane orientation is determined by HR-TEM and from the dependence of the E2 Raman mode with the polarization angle. HR-SEM pictures reveal top nanorod surfaces showing a- and m- plane facets. PL measurements at low temperature give an emission energy of 0.69 eV, a typical signature of a high quality material. As in the case of c-plane oriented III-N nanorods, no wetting layer is observed, in marked contrast with the nucleation and growth of QDs.

12:00 Oral

Indium segregation in $Al_{1-x}In_xN$ investigated by photoluminescence under hydrostatic pressure

Agata Kamińska[1], Grzegorz Staszczak[2], Izabela Gorczyca[2], Andrzej Suchocki[1], Tadeusz Suski[2], David P. Schenk[3]

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The progress in the growth of $Al_{1-x}In_xN$ layers has enabled detailed investigation of this material, which is of considerable interest because its band gap E_g covers a very wide spectral range, from the infrared (InN with E_g = 0.7 eV) to the ultraviolet (AlN with E_g = 6.2 eV).

Introducing In to GaN or to AlN improves efficiency of light emission from these materials. Segregation of In seems to play a crucial role in this improvement. Photoluminescence and absorption measurements of $Al_{1-x}In_xN$ revealed surprisingly large bowing in the E_g vs. x [1]. Recently I. Gorczyca et al. have performed theoretical calculations of the electronic band structure of $Al_{1-x}In_xN$ alloys and their pressure dependence [2]. The calculated band gaps and their pressure coefficients dE_g/dp show a decrease for increasing x with especially pronounced bowing of dE_g/dp. Moreover, taking into account effect of In segregation causes a decrease in E_g, reduction of dE_g/dp and an increase of bowing with respect to In distributed uniformly. Predicted pressure coefficients of E_g for $Al_{1-x}In_xN$ with x = 0.25 change drastically from 35 meV/GPa to 15 meV/GPa for uniform and entirely segregated cases, respectively.

In this work we compare these predictions with our experimental results. We analyze the pressure behaviour of light emission for series of $Al_{1-x}In_xN$ layers. The measured values of PL peak pressure coefficient are shown to decrease with increasing indium content in accordance with theoretical predictions and are situated between theoretical values calculated for uniform and entirely In-segregated cases. This likely reflects the role of different growth conditions in modifying a rate of In-segregation in $Al_{1-x}In_xN$ layers. These results can be useful in understanding and controlling the influence of growth conditions on basic properties of these materials.

References:

[1] K. Wang et al., *J. Appl. Phys.* **103**, 073510 (2008);

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12:15 Oral

Strain control in GaInN/GaN multiple quantum wells for high-performance green-light emitters

Motoaki Iwaya, Daisuke Iida, Tetsuya Matsubara, Satoshi Kamiyama, Hiroshi Amano, Isamu Akasaki

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High-brightness blue- and green-LEDs and high-power violet LDs have been achieved. In these devices, GaInN-based QWs are usually used as an active layer grown on a thick GaN template layer. However, the efficiency of green LEDs is not satisfactory and green LDs have not been realized yet. Many reports have been published on LEDs grown on nonpolar nitride planes, such as a- and m-plane, because the problem of the internal electric field can be solved in principle. However, regarding the efficiency of green LEDs and the realization of green LDs, the generation of mismatch-induced defects is another severe problem. In this report, we discuss about the importance of the strain control in GaInN/GaN MQWs for achieving high-performance green-light emitters. We fabricated GaInN/GaN MQWs on a high-crystalline-quality thick m-plane GaInN underlying layer, and the integral PL intensity was compared with that of identical GaInN/GaN MQWs on a high-crystalline-quality m-plane GaN underlying layer. The dislocation and stacking fault densities of thick GaInN on m-plane grooved GaN and m-plane GaN are of the same order: 10^7 cm^{-2} and 10^4 cm^{-1}, respectively. The light emission wavelengths from the MQWs were violet (~396 nm), blue (~450 nm), and green (~535 nm). The integral PL intensities obtained from the GaInN/GaN violet, blue, and green MQWs on the high-quality thick GaInN underlying layer were 1.1, 1.5, and 2.1 times higher than these obtained from the MQWs on the m-plane GaN underlying layer, respectively. This result clearly shows that the use of an almost perfectly relaxed GaInN template is essential for improving the PL efficiency in the long-wavelength (green) region, which is expected to lead to the improved performance of green-light emitters.

Acknowledgement: This study was partly supported by the Knowledge Cluster Initiative (2nd stage), Tokai region and the Ministry of Education, Culture, Sports, Science and Technology (Grant-in-Aid for Young Scientists (A), # 21686034).

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Session 4

Tuesday afternoon, 15 September, 14:00
Room 231

14:00 Oral

Effect of the oxygen on the optical, structural and electrical properties of ZnF_2 thin films prepared by DC reactive sputtering

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Zinc fluoride (ZnF_2) is an insulating wide band gap crystal has been recently demonstrated that this material is attractive for applications as efficient light emitter in the UV.

Crystalline ZnF_2 films of a rutile-type structure were obtained on corning-glass and silicon substrates kept at 100 and 300°C. A film growth along [110] direction was found. An amorphous ZnF_2 film was obtained when the substrate was kept at 100°C.

The ZnF_2 thin films were elaborated on silicon and corning glass substrates by using a reactive sputtering with an argon/oxygen/fluorine mixture from a metallic Zn target at 100°C and at constant working total pressure of 100 Pa. The ZnF_2 thin films were investigated by optical transmission, X-ray diffraction (XRD), scanning electron microscopy (SEM), Rutherford backscattering spectrometry (RBS) and electrical conductivity measurements. The transmittance measurements have shown that all films exhibit high transmittance in the 400–2500 nm range. The averaged transmittance of the ZnF_2 film increased from 80 to 94% upon vacuum-annealing at 300°C for 1 hour (Fig.1). The crystalline structure of ZnF_2 thin films indexed by XRD reveals tetragonal lattice with cell parameters a =b= 4.7034 Å and c = 3.1335 (Fig.2). The RBS measurements reveal that ZnF_2 thin films are almost stoichiometric, with an atomic composition close to Zn(34%) F(66%). The surfaces morphology of ZnF_2 thin films deposited at 100°C, from the scanning electron microscopy images, shows significant changes due to the oxygen partial pressure used during the film deposition. A porous surface consisting of circular craters, with an average diameter ranging between 0.2 and 20nm is revealed. The dark conductivity s_d versus temperature, in the Arrhenius plot, from a ZnF_2 thin films deposited at 100°C and after vacuum-annealing at 300°C for one hour clearly evidenced a very large increase in electrical conductivity, from 10^{-10} to 10 $W^{-1}.cm^{-1}$ (Fig.3).

14:15 Oral

Surface and bulk electronic properties of significantly cation-anion mismatched oxide semiconductors

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Group II- and III-oxides, such as CdO, ZnO, and In_2O_3, have a significant size and electronegativity mismatch between the large cation and the small electronegative anion oxygen: these materials can be described as significantly cation-anion mismatched oxide semiconductors (SCAMOS). Due to this mismatch, their electronic structure is characterised by a particularly low Γ-point conduction band minimum which lies below the charge neutrality level [1,2]. We show that this ultimately determines the charge character of native defects, hydrogen impurities and surface states, unifying bulk and surface electronic properties via a single energy level.

In particular, native defects and hydrogen impurities are both shown to be donors, even in already heavily n-type material [1], in contrast to the behaviour observed in most semiconductors, and explaining the propensity for SCAMOS to exhibit high unintentional conductivity. Further, the formation energy for compensating native defects to form when n-type doping these materials is low compared to in conventional semiconductors and so these materials can be easily doped n-type: SCAMOS can therefore be made highly conducting despite their generally large optical band gaps, allowing them to be used as transparent conducting oxides. These materials are also shown to support a build up of charge (that is, an electron accumulation) at their surfaces [1,2], in contrast to the surface carrier depletion of most semiconductors. This is associated with positively charged donor surface states and a downward bending of the bands at the surface, which further results in a quantization of the conduction band states [3]. This electron accumulation has important implications for metal-SCAMOS contacts.

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14:30 Oral

Influence of exciton-phonon interaction on the absorption properties of ZnMgO alloys

Maciej Neumann[1], Gerhard Gobsch[1], Ruediger Goldhahn[1], Thomas A. Wassner[2], Bernhard Laumer[2], Martin Eickhoff[3]

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ZnO and the related ternary ZnMgO alloys are known to exhibit a

very high exciton binding energy and a strong polar character. Due to these properties, the characteristic features in their absorption spectra cannot be fitted alone by the exciton line shape suggested by Elliott. We present a comprehensive study of the so-called exciton-phonon absorption in ZnMgO alloys based on the analysis of the complex dielectric function (DF). Wurtzite $Zn_{1-x}Mg_xO$ films with Mg contents between x=0 and x=0.37 were grown on C-plane sapphire substrates by plasma-assisted molecular beam epitaxy using a MgO/ZnMgO buffer layer. High-resolution X-ray diffraction measurements indicated composition-independent in-plane lattice parameters, i.e. pseudomorphic growth of the films on the buffer. The optical properties of the films were investigated by temperature-dependent spectroscopic ellipsometry, yielding the DF, photoreflectance (PR) as well as photoluminescence (PL) spectroscopy. In order to determine the characteristic transition energies from the imaginary part of the DF, a model is applied which comprises three parts: free excitonic lines, the exciton continuum and enhanced absorption above the band gap due to exciton-phonon interaction. The fitted free excitonic transition energies are in excellent agreement with the PR data while the corresponding emission peak of the alloys is found at slightly lower energy indicating weak exciton localization. The broadening of the exciton-phonon contribution increases slightly with the Mg content but decreases with temperature. The analysis yields data on the compositional dependence of the Huang-Rhys parameter. The full DF analysis yields the band gaps with considerably improved reliability. In the final part, the influence of the in-plane tensile strain on the determined band gaps and the valence band ordering is discussed. Strain-free values are obtained from k·p calculations using XRD-results as input parameters.

14:45 Oral

Low Temperature Synthesis of ZnO Nanowires with Very High Aspect Ratio: The Effect of Solution Concentration on Nanowire Morphology Grown by Wet Chemical Technique.

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Single-crystalline ZnO nanowires with diameters of about 20 nm and lengths of over 2 µm (aspect ratio >100:1) have been successfully synthesized by a simple hydrothermal treatment of zincnitrate and hexamethylenetetramine aqueous solution at relatively low temperature, i.e. 95 oC. It has been found that solution concentrations of 5 mM or above were favourable for nanorod synthesis, while lower concentrations resulted in formation of thin nanowires. The mechanism of this kind of growth will be discussed and suggested for localization of the nanowire growth. The crystal structure, morphology and the electrical properties of the so grown ZnO nanowires will also be characterized by transmission electron microscopy, scanning electron microscopy and current-voltage measurement, respectively.

15:00 Oral

Extraction of diode parameters of Mo-CdSe-ZnS-Cu_xS structure from dark I-V curve

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We present the results of studying of current – voltage (I-V) characteristics of the structure based on II-VI materials including wide band gap zinc sulfide (Eg = 3.58 eV). The examined structures are prepared by continuous thermal evaporation of CdSe and ZnS powders with films growth in quasi-closed space. The presented structure works as ultraviolet detectors.

Taking into account the facts that the upper conductive layer Cu_xS is degenerative semiconductor material, CdSe thick layer serves as a base layer for formation of ohmic contact to Mo, we consider that the main process for the charge carrier transport take place in thin film of ZnS and in the junction of ZnS - Cu_xS and examine our structure as a Schottky diode. The I-V characteristics are measured from 183 to 343 K using the measurement setup described in [1]. The procedure of extraction of diode parameters is fulfilled on the base of the method presented in [1] applying the Lambert function. The simulated and experimental I-V curves show the good fitting and it allows extracting the diode parameters and examines their temperature dependences. The temperature curves show the follows results: the series resistance linear drops with temperature increase; monotonic and quasi-linear dependence of the Schottky barrier height that increase from 0.3 to 0.8 eV with temperature increasing; the ideality factor has the linear temperature dependence in temperature range (180 – 280 K) with dependence n ~ -0.094*T. The big value of the ideality factor points to the non - ideal of the junction and big influence of multistep tunnel - recombination currents through the levels in the space-charge region.

The mathematical simulation shows that contribution from shunt resistance is negligible.

1. W. Jung, M. Guziewicz, Mater. Sci. Eng. B (2009), doi:10.1016/j.mseb.2009.02.013

15:15 Oral

Photoluminescence studies of ZnO nanocolumns and nanocones grown by PLD on various substrates

Marco A. Peres[1], Manuel J. Soares[1], A. J. Neves[1], Teresa Monteiro[1], Vinod E. Sandana[2], Ferechteh Teherani[2], David J. Rogers[2]

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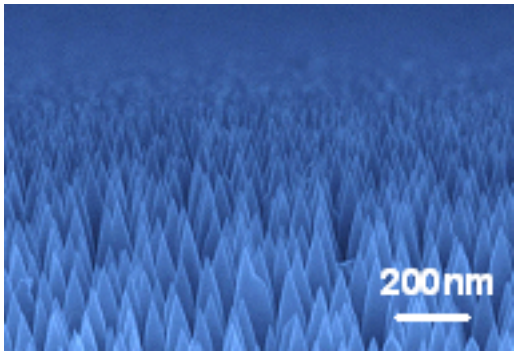
ZnO nanostructures were grown by PLD on c-sapphire, Si (111) (n-type and p-type), steel and ZnO substrates and analysed using

SEM, XRD and PL techniques. Under optimised growth conditions, self-forming arrays of vertically aligned nanostructures were obtained. SEM studies revealed two main structures: nanocolumns and a "moth-eye type" array of nanocones, which gave a graded effective refractive index [2].

XRD measurements indicate that both types of structures were well crystallized and highly oriented along the c-axis. nZnO/pSi heterojunction LEDs formed using the nanocone arrays gave rectification and blueish-white electroluminescence [2]:



The sample photoluminescence was investigated using above band gap excitation. Low temperature PL spectra for nanocolumns grown on sapphire and n-type Si (111) show an emission which is similar to that observed for bulk ZnO with a spectrum dominated by donor bound exciton recombination at ~3.36 eV and a structured green band. The PL intensity was particularly enhanced for nanocone structures grown on the cubic Si substrate. For these samples, besides the usual PL lines observed both in ZnO bulk and in nanocolumns grown on sapphire, additional features were observed at 3.12 eV, 3.02 eV and 2.92 eV which follow a vibronic progression of 100 meV. The analysis of these new lines and a correlation with the growth parameters and structural analysis will be discussed.

[1] Sandana et al., accepted for publication J. Vac. Sci. Technol. (2009)

[2] Rogers et al. Proc. SPIE Vol. 7217 08 pp1-8 (2009)

COFFEE

Tuesday afternoon, 15 September, 15:30
Main Hall

Session 5

Tuesday afternoon, 15 September, 16:00
Room 231

16:00 Invited oral

Nanowires and 2D modulation doped nanostructures based on tellurides *,**

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We will review recent progress in the molecular beam epitaxy of telluride nanostructures achieved in the Institute of Physics. This will cover two areas of nanostructures: nanowires and n-type modulation doped quantum wells.

The growth of ZnTe, ZnMgTe and ZnMnTe nanowires was performed on the GaAs substrates covered with Au nanocatalysts. Raman experiments revealed the shift and splitting of the observed lines which is comparable to that expected for the 3D mixed crystal with composition equal to the composition of NWs estimated based on their lattice constant. The temperature variation of the internal Mn^{2+} luminescence transition provides strong evidence that Mn^{2+} ions in our $Zn_{1-x}Mn_xTe$ NWs are located at tetrahedral lattice sites.

Careful optimization of the growth procedures and structure design have lead to a significant improvement of the quality of CdTe-based structures containing 2DEG. Low temperature Hall mobility as high as 400 000 cm^2/Vs was observed after the illumination. The exceptional quality of nanostructures was also proven by the observation of fractional quantum Hall effect (FQHE) at mK temperatures and high magnetic fields not only in CdTe- but also in $Cd_{1-x}Mn_xTe$-QWs, with effective electron g-factor as high as 300. To our knowledge this is the very first observation of FQHE in II-VI semiconductor materials.

*Work done in collaboration with E. Janik, W. Zaleszczyk, G. Karczewski, P. Dłużewski, S. Kret, W. Szuszkiewicz, E. Dynowska, J. Domagała, M. Aleszkiewicz, L.T. Baczewski, A. Petroutchik, A. Presz, J. F. Morhange, H. Kirmse, W. Neumann, W. Caliebe, M. Wiater, V. Kolkovski, T. Wojciechowski, C. Betthausen, A. Voge, W. Wegscheider, D. Weiss, J. Kunc and M. Potemski.

** The research was partially supported by the European Union within European Regional Development Fund, through grant Innovative Economy (POIG.01.01.02-00-008/08), by the MNiSW (Poland) through grant N515 015 32/0997, and by the Foundation for Polish Science through subsidy 12/2007.

16:30 Invited oral

New concepts for ZnTe-based microcavities

Wojciech Pacuski[1,2], Carsten Kruse[1], Stephan Figge[1], Kristian Frank[1], Marco Schowalter[1], Andreas Rosenauer[1], Matthias Florian[1], Frank Jahnke[1], Tomasz Jakubczyk[2], Jan A. Gaj[2], Mihai Stoica[3], Mariuca Gartner[3], Detlef Hommel[1]

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We report on the realization and a detailed characterization of photonic structures based on II-VI compounds and designed for ZnTe substrates. Our motivation is to enhance CdTe quantum dots (QDs) emission using cavity effects. Distributed Bragg reflectors (DBRs) lattice matched to ZnTe were developed in order to create a microcavity and to confine light in direction perpendicular to the sample plane [1]. Micropillars were etched using a focused ion beam (FIB) in order to confine light in the sample plane by internal reflection. Inside the microcavity, a sheet of CdTe QDs was introduced. Our structures were grown using molecular beam epitaxy (MBE) on

GaAs substrate followed by a 1 μm thick fully relaxed ZnTe buffer layer. The requirements of the lattice matching and of a high refractive index step are fulfilled using a novel method. Instead of using ternary or quaternary compounds, the low refractive index layer is made of a short period triple superlattice based only on the binary compounds: ZnTe, MgTe, and MgSe. Layer thicknesses in the superlattice are chosen to balance strain and fit to ZnTe. The high refractive index layer is ZnTe. Pseudomorphic growth of the structure on the ZnTe buffer was confirmed using high resolution X-ray diffraction (HRXRD). Images of the main components of the structure, i.e. cavity, QDs, and DBR pairs with triple supperlattice were obtained using scanning transmission electron microscopy (STEM). The reflectivity spectrum reveals a large width of the stopband (over 60 nm) and a cavity mode at about 620 nm with a linewidth corresponding to Q factor of 1000. Ellipsometry studies were performed separately for all materials used in the structure in order to determine both real and imaginary part of refractive indices. The determined parameters are used for calculation of optical properties of photonic structures.

[1] W. Pacuski, C. Kruse, S. Figge, D. Hommel, Appl. Phys. Lett. 94, 191108 (2009).

17:00 Oral

Selective Multiterminal Sensor on Individual ZnO Nanotetrapod

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Sensors based on semiconducting oxides nanostructures such as zinc oxide nanowires, nanotubes and nanorods have attracted vast and persistent attention for detecting ultraviolet (UV) radiation, gas and chemicals [1-3]. Low-cost, nonpoisonous and highly sensitive and selective sensors are very desirable for detection of various gases and chemicals in the environment. Here we present a sensitive and selective sensor based on individual ZnO nano-tetrapods for detection of UV light and H_2 gas at room temperature. The ZnO nano-tetrapod sensor was synthesized by a solution method [4].

For the sensor fabrication, focused ion beam technique is employed. The ZnO nano-tetrapods were first transferred to another SiO_2 coated silicon substrate using an ultrasonic bath. After transferr, we greatly reduce the density of nano-tetrapods on the substrate surface and make the individual manipulation of the ZnO nano-tetrapod possible. The sensitivity of the ZnO nano-tetrapod sensor versus different types of test gases, like H_2, CO, LPG and CH_4 are presented. It was observed that the developed sensor shows different sensitivity values to different gases.

References

[1] E. Comini, G. Faglia, G. Sberraglieri, Z. Pan, and Z. L. Wang, Appl. Phys. Lett. 81, 1869 (2002).

[2] O. Lupan, G. Chai, L. Chow, Microelectronics Journal 38, 1211, 2007.

[3] M. C. Newton, S. Firth, P. A. Warburton, Appl. Phys. Lett. 89, 072104, 2006.

[4] O. Lupan, L. Chow, and G. Chai, Sensors & Actuators, B. To appear.

17:15 Oral

II-VI compound phosphors for modern X-Ray applications

Oleksandr I. Lalaiants, Sergiy M. Galkin, Vladimir D. Ryzhikov, Iryna A. Rybalka, Yevgen F. Voronkin, Igor A. Breslavskiy

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It is known that several A^2B^6 compounds have high temperature stability, radiation hardness, phosphor properties and therefore scintillation and optoelectronic applications. Thus, ZnS crystals use as phosphor in light-emitting and detective devices. The ZnSe crystals apply as X-Ray- and gamma- detectors in medical homographs and airport security systems. The optical and electrical parameters of these crystals depend on structure of defective complexes. These complexes have the spatially-periodic structure.

We have studied the optical and scintillation parameters of ZnSe crystals grown by Bridgman method. We have studied the structure of luminescence centers and the emission mechanism in ZnSe with donor dopants. Thus luminescence at 300 K is conditioned by defective complexes Donor-V_{Zn}-Zn_i in ZnSe crystals.

The donor-doped ZnSe crystals are characterized of following parameters:

- Presence of n-type conductivity with electron concentration 10^{17}-10^{18} cm^{-3};
- Low level of self-indemnification of the generated vacancies;
- Introduction into a crystal of the interstitial element with formation of the dipole complexes "vacancy - interstitial atom" in amount 10^{17}-10^{18} cm^{-3}.

The “scintillator-photodiode” detectors are applied in the majority of the modern computer tomographs. A broad dynamic range (10^3-10^4) and a small aperture are typical for these detectors.

These parameters determine the high spatial resolution of tomography systems. The short decay time and low afterglow of scintillators are necessary for rapid signal counting from great number of detectors (1000 channel and more). The afterglow must be within (0.001-0.01) % after 10 ms of X-Ray pulse.

These research results have allowed to develop reproducible technology of ZnSe scintillator production for application in inspection and medical equipments.

This work was supported bythe U.S. Civilian Research and Development Foundation (CRDF), and NATO Science for Peace and Security Program (Project SfP-982823).

Wednesday, 16 September

Session 6

Wednesday morning, 16 September, 9:00
Room 231

9:00 Invited oral

Present Status and New Challenges of Nitride Semiconductors for Advanced Electronic Devices

Yasushi Nanishi, Tomohiro Yamaguchi

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AlGaN/GaN hetero-structure attracted much attention as a promising material system for high power and high frequency transistors. Remarkable progress in high power and high frequency operation has been made in the last 10 years. Power performances at high frequency obtained from this structure confirmed the high potential of this system and production of these devices for high frequency power amplifier has been started.

Focused attention on InN has been made as a material for even higher frequency operation with its high mobility, small effective mass, large inter-valley separation and large optical phonon energy. After new finding of InN narrow band gap, material parameters of InN have been totally revised including effective mass and non parabolicity coefficient. Using these revised material parameters, carrier transport in InN is re-estimated and even higher performances in high frequency operation have been predicted.

In spite of these remarkable physical potentials, almost no reports on experimental demonstration of InN electronic devices have been made so far. Main reason for this comes from poor quality of InN, band bending and accumulation of carriers at the surface.

In this talk, we will introduce newly developed RF-MBE growth method which made it possible to grow high-quality InN very reproducibly. This new growth method is composed of In-rich growth process and consecutive In droplet elimination by nitrogen radical beam irradiation (DERI) process. Then, the results on studies on ohmic and MIS contacts to InN will be shown. InN re-growth technology applied to AlGaN/GaN HFET will also be introduced and show possibility to make new type of functional device using large band offset between InN and other nitride semiconductor system.

This work was supported by the MEXT through Grant-in Aids for Scientific Research in Priority Areas “Optoelectronics Frontier by Nitride Semiconductor” #18069012 and Scientific Research (A) #21246004.

9:30 Invited oral

Catalyst- and mask-free grown GaN nanocolumns

Timo Aschenbrenner[1], Gerd Kunert[1], Carsten Kruse[1], Stephan Figge[1], Joachim Kalden[2], Kathrin Sebald[2], Knut Müller[3], Marco Schowalter[3], Jürgen Gutowski[2], Andreas Rosenauer[3], Detlef Hommel[1]

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In recent years, the self-organized growth of III-nitride based nanocolumns attracted considerable interest since it allows for the preparation of defect-free single-crystalline epitaxial structures for various applications. We focus on a straightforward catalyst- and mask-free deposition on r-plane sapphire [1].

Prior to the growth, a nitridation of the substrate is performed in a MOVPE reactor using NH3, which leads to the formation of AlN islands of approx. 30nm diameter on the Al_2O_3surface. These substrates are transferred into the MBE chamber where growth of the GaN takes place. Almost immediately, the RHEED diffraction pattern indicates the development of two different crystal phases, namely a compact two-dimensional a-plane GaN layer and an ensemble of nanocolumns with diameters of 50-200nm, which are oriented with an average angle of 62° towards the surface plane. The nanocolumns show a hexagonal cross-section, they are highly ordered and well aligned. The length of the nanocolumns depends nearly linearly on the deposition time, while their formation is very sensitive to the growth temperature (range of $\Delta T=10K$) but insensitive to the III/V-ratio.The crystallographic orientations of both phases were analyzed by HRXRD polar-figures and the polarity of the rods was determined by convergent beam electron diffraction measurements to be N polar. TEM darkfield measurements indicate a very low defect density in the rods. This is in good agreement with µ-PL measurements, which show no yellow or stacking-fault related luminescence. The spectrum of a single GaN column is dominated by a very sharp D^0X line with a FWHM of 1.2meV. Doping of the rods with silicon (n-type) and magnesium (p-type) was carried out. Depening on the concentration an increase of the D^0X intensity was observed. Furthermore, first results concerning nanocolumn-based heterostructures, e.g. InGaN-QWs and AlN-interlayers will be discussed.

[1] T. Aschenbrenner et al. Nanotechnology 20, 75604 (2009)

10:00 Oral

Optical properties of high-quality cubic AlN, GaN, and AlN/GaN MQWs grown on 3C-SiC

Marcus Roeppischer[1], Christoph Cobet[1], Norbert Esser[1], Georg Rossbach[2], Ruediger Goldhahn[2], Martin Feneberg[3], Benjamin Neuschl[3], Klaus Thonke[3], Thorsten Schupp[4], Klaus Lischka[4], Donat J. As[4]

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It was recently demonstrated, that the quality of zincblende group-III nitrides can be considerably improved if bulk 3C-SiC(001) is used as the substrate for the deposition of the films by molecular beam epitaxy. For example, phase-pure cubic GaN and AlN have been achieved, and intersubband absorption of short-period GaN/AlN MQWs in the infra-red was demonstrated. Despite this progress, fundamental optical properties of these materials have not been reported so far. In this contribution, we present a comprehensive characterization of cubic AlN, GaN and related MQWs. The shape of the dielectric functions (DF) from 0.56 eV up to 9.8 eV, as obtained by ellipsometry, as well as photoreflectance (PR), photo- (PL) and cathodoluminescence (CL) spectra will be discussed in detail.

The DF of AlN for example shows pronounced features emphasizing the excellent quality of the material. In comparison to the hexagonal counterpart, the characteristic interband transitions are appreciably shifted to lower energies. In particular, we find a sharp excitonic onset of absorption at 5.88 eV, from which, taking into account the exciton binding energy, a lowest direct gap at 5.93 eV is obtained. From the pronounced absorption tail we estimate the indirect gap with about 5.2 eV. By low temperature CL we observe for the first time a clear signal originating unambiguously from the cubic AlN layer. Broad and unstructured defect related luminescence bands are detected above the energy position of the bandgap of 3C-SiC extending up to ~ 4.2 eV. Temperature-dependent PR and PL measurements yield an unambiguous indication for strain-induced heavy-light hole splitting in GaN.

Finally, interband transition energies of AlN/GaN MQWs for various well widths, as obtained by ellipsometry, PR, and PL are discussed. A comparison with theoretical calculations will be given.

10:15 Oral

High quality (0001) GaN films grown on diamond substrates by molecular beam epitaxy

Alexandros Georgakilas, George Tsiakatouras, Adebowale O. Ajagunna, Katerina Tsagaraki, Maria G. Androulidaki

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The growth of wide bandgap III-nitrides on diamond substrates is very interesting since it would provide the highest thermal conductivity for efficient extraction of the large amounts of heat produced in the active region of high power light emitters or microwave transistors. This paper reports on the growth and properties of high quality single crystalline (0001) GaN films on diamond substrates by nitrogen RF plasma source molecular beam epitaxy (RFMBE). A range of diamond surface orientations have been investigated including (100), (110) and (111) to determine the dependence of GaN crystal quality. FE-SEM and AFM observations of the GaN-on-diamond surfaces revealed the growth of compact GaN films with adequately smooth surfaces. Micro-cracks appeared for the 2.0mm GaN films. A low rms roughness value of 3.5nm was determined for $10x10mm^2$ AFM scans. XRD (q-2q) scans covering an extensive range of diffraction angles revealed that single crystalline (0001) GaN films were grown for all the different orientations of diamond substrates. However, the crystalline quality of the GaN films, as evidenced by the FWHM of XRD rocking curves (RC) around the symmetric (0002) and asymmetric (10-15) reflections of GaN exhibited a dependence on the diamond orientation, with the (100) orientation clearly resulting to inferior structural quality. The c lattice constant was also measured by XRD using the extended Bond method. The 1.4mm GaN films were under tensile strain of 1.74×10^{-3}, which dropped to 0.96×10^{-3} in the 2.0mm GaN film with formation of micro-cracks. Low temperature PL spectra exhibited excitonic emission as well as defect or impurities related emission peaks. Bandedge luminescence was recorded at room temperature, exhibiting a red-shift of the PL peak as expected for the present tensile stress. The

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COFFEE
Wednesday morning, 16 September, 10:30
Main Hall

Session 7
Wednesday morning, 16 September, 11:00
Room 231

11:00 Invited oral

Elaboration and physical properties of a-plane ZnO layers and nonpolar quantum wells

Jean-Michel Chauveau[1,2], Monique Teisseire[1], Christian Morhain[1], Christiane Deparis[1], Hyonju Kim-Chauveau[1], Borge Vinter[1,2]

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ZnO-based quantum wells have attracted much attention in the last few years due to their opportunity of combining band gap engineering, along with large excitonic binding energies. Indeed theoretical works suggest that the 60meV-binding energy of excitons in ZnO, could be further doubled in quantum wells (QWs). Studies on ZnO have mainly focused on films grown in (0001) orientation. In this configuration, the wurtzite ZnO layers exhibit built-in electric fields along the growth direction. Non-polar surfaces are thus of a particular interest since the c-axis lies within the growth plane. As a result it is expected that quantum well structures can be grown without any screening of the exciton binding energies.

In this communication, we report on the growth, structural and photoluminescence properties of a-oriented (non-polar) (Zn,Mg)O/ZnO quantum wells (QW). The heterostructures are grown by plasma assisted Molecular Beam Epitaxy (MBE) on sapphire and a-plane ZnO substrate. A series of QWs with different widths and different Mg content demonstrates the absence of Quantum Confined Stark Effect [1]. The strain relaxation process exhibits a strongly anisotropic behavior when a sapphire substrate is used [2]. The anisotropy of the lattice parameters gives rise to an unusual in-plane strain state on ZnO QWs: tensile strain along [1-100], compressive strain along [0001]. Due to this strain state, the excitonic transitions are strongly blue-shifted, and the quantum-well confined exciton energies measured by photoluminescence excitation are very satisfactorily explained by the theory that takes this into account [3]. Finally, we show the drastic improvement of the structural and optical properties when the QWs are grown on non-polar ZnO substrates.

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[2] J.-M. Chauveau et al., J. Appl. Phys. 104, 073535 (2008).
[3] J.-M. Chauveau et al., Appl. Phys. Lett. 23 (3), 035005 (2008).

11:30 Invited oral

Spectroscopy of transition metal ion doped GaN and ZnO

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In the context of spintronic applications we studied Mn, Fe and Gd doped GaN and ZnO. Samples with different transition metal concentrations and n or p co-doping were investigated by means of optical and magnetic experiments. The results allow us to elucidate the following issues that are of crucial significance on the way to eventually realize a ferromagnetic coupling at room temperature: (1) Which charge states the transition metals are found in, (2) whether and where they from levels within the band gap, (3) the formation of bound states consisting of a hole localized at a transition metal ion in the charge state 2+. We also assess the influence of the transition metal concentration on the structural, electronic and optical properties of GaN and ZnO. The origin of room temperature ferromagnetism of Gd doped GaN is still an unsolved puzzle. In this contribution we present a comprehensive array of experimental results that help to shed light on this issue. While the incorporation of Gd barely affects the structural and electrical properties of GaN, it leads to a magnetization of 20 emu/cm3 as determined by vibrating sample magnetometry.

12:00 Oral

Photovoltaic cells based on nickel phthalocyanine and zinc oxide formed by atomic layer deposition

Pavlo Stakhira[1], Grzegorz Łuka[2], Georgiy Pakhomov[3], Dmytro Volynyuk[1], Vladyslav Cherpak[1], Marek Godlewski[2,4], Elżbieta Guziewicz[2], Zenon Hotra[1,5]

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Recent progress in organic solar cells is associated, in most cases, with utilization of the layered (or bulk) heterojunctions, as well as with development of packaging technologies. This particularly concerns various prototypes of solar cells employing phthalocyanines (Pc) as photoactive materials. Here, due to high sensitivity of these compounds to absorbed gases (sensitivity of electrical parameters of solids or thin films, but not molecular destruction), isolation from atmosphere is highly desired. Additionally, problems arise at the top interface between Pc layer and metallic electrode. The device life time suffers much from instability of this interface, which can roughly be divided into chemical (possible reactions, diffusion of components *etc.*) and physical degradation, *i.e.* alteration in barrier properties.

One of the ways to solve these problems is insertion of an ultrathin layer, organic or inorganic, before depositing top contact. This may either prevent penetration of admixtures into Pc layer or facilitate injection of charge carriers from the contact, or both. The present work investigates the effect of introduction of zinc oxide (ZnO) ultrathin layer on the efficiency of photovoltaic cells based on nickel phthalocyanine (NiPc). Introduction of ultrathin ZnO layer formed by the atomic layer deposition (ALD) technique was found to markedly improve the parameters of nickel phthalocyanine (NiPc) based photovoltaic cells. This improvement is attributed to an increase in both rectifying and photovoltaic activity in the ITO/NiPc/ZnO/Al cells incorporating a hybrid heterojunction as compared with the single-layer ITO/NiPc/Al cells, to enhancement of the overall spectral re-

sponse in the double-layer cells and to extension of their long-term operational stability.

12:15	Oral

A membrane supported dual-band acoustic device for biological applications

Alexandros K. Pantazis[1,2], Electra Gizeli[2,3], George Konstantinidis[1]

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A variety of microfabricated devices are used in biotechnology and biomedicine, mostly for interaction studies and analysis between different biomolecules (i.e. antigen – antibody) or for the detection of specific biotargets, especially when used in human health or environmental monitoring. Such biotargets can be proteins, liposomes, DNA, bacteria etc. Consequently, it is well understood that the sensitivity of these devices is very essential. Among the acoustic devices, Lamb-wave has the greater sensitivity. The reason for this is the membrane configuration that this device has. Until now, mostly of the Lamb-type devices were based on sputtered AlN or ZnO material and a few on MOCVD AlN. No reports have been made for using GaN as the piezoelectric material in these devices, just in a couple of Surface Acoustic Wave (SAW) devices. In this paper, we present the first GaN Lamb-wave device (Fig. 1). Due to the fact that when the membrane thickness is quite smaller than the wavelength of the device, only the two fundamental modes of the acoustic wave are supported (Anti-symmetrical A_0 and Symmetrical S_0). Thus, two different operation frequencies exist, a low one and a high. Therefore, interaction studies and analysis can be performed simultaneously for a low and a high frequency. Additionally, the chosen material offers the potential for monolithic integration with other semiconductor devices or systems.

A variety of design considerations were taken into account before the fabrication of the device. These considerations included the application of the device, the materials to be used, the experimental set-up, as well as the geometry of device and the fabrication method.

The Lamb-wave devices were fabricated by using conventional photolithographic techniques. The procedure has 3 main steps: 1) Front etch of 2 μm, in order eventually to dice the devices, 2) The electric circuit, which consists of the pads and the Interdigital Transducers (IDTs), were formed by the deposition of 20 nm Cr followed by 200 nm Au and 3) A back-etch of ~500-650 μm (depending on the thickness of the substrate) for forming the membrane. The wavelength of the device is 40 μm, while the two acoustic modes that are produced, the A_0 and S_0, were measured by a Network Analyzer, with operation frequencies 10.56 MHz and 192 MHz respectively, while the amplitude in S_{21} parameter was -79 dB and -43 dB respectively (Fig.2).

The evaluation of the sensitivity of the Lamb-wave devices to a variety of biomolecules is currently under investigation and will be presented.

LUNCH

(maps available at the Reception desk)
Wednesday afternoon, 16 September, 12:30
building of the Faculty of Physics

Session 8

Wednesday afternoon, 16 September, 14:00
Room 231

14:00	Oral

Efficiency „droop" in nitride light emitters

Szymon Grzanka[1], Piotr Perlin[1,2], Robert Czernecki[2], Łucja Marona[2], Michał Boćkowski[1,2], Mike Leszczynski[1,2], Tadeusz Suski[2]

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The decrease of the light emission efficiency at high currents in the nitride based light emitting diodes (LEDs) – nicknamed as "droop" attracts a lot of attention because of its importance for the production of very bright white LEDs which could in perspective completely replace the incandescent bulbs. The gradual decrease of the diode efficiency at high currents density was reported by many groups and interpreted in various ways. The dominant interpretations is the contribution of Auger effect which indeed should manifest at high currents. However, a conventional Auger effect is hardly expected for wide band-gap semiconductor like GaN or InGaN with low indium content. The other, non direct Auger mechanisms were proposed like defect-assisted Auger, or phonon-assisted Auger.

We have measured the electroluminescence from LEDs and laser diodes fabricated on top of various types of substrates like sapphire, HVPE-GaN, high pressure grown GaN. These structures were characterized by the density of dislocations ranging from $10^9 cm^{-2}$ down to $10^5 cm^{-2}$. In all cases the similar efficiency droop was observed. We also studied the structures with the different emission wavelength (between 410 and 440 nm). All these structures showed similar "droop". Finally, we investigated the droop effect in nitride laser diodes. We observed a strong competition between the stimulated emission and the nonradiative droop-mechanism. In some cases, the shortening of the recombination time in laser diodes below the lasing threshold caused by the stimulated recombination may somehow suppress the Auger-like nonradiative recombination.

14:15 Oral

Surface analysis of polar GaN surfaces with photoelectron and high resolution electron energy loss spectroscopy

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Group III-nitrides with wurtzite crystal structure are chemically stable semiconductors with high carrier mobilities, a high internal spontaneous and piezoelectric polarization. These properties make them suitable materials for very sensitive and robust chemical sensors as wel as transistors for high frequency and high power applications. The polar GaN surfaces N-face (000-1) and Ga-face (0001) exhibit significant differences in the electrical and chemical properties. The understanding and controlling of these properties is mandatory for further successful processing for electronic devices.

We studied the properties of N-face and Ga-face GaN surfaces with X-ray and ultraviolet photoelectron spectroscopy (XPS, UPS), atomic force microscopy (AFM), reflection of high energy electron diffraction (RHEED) as well as high resolution electron energy loss spectroscopy (HREELS).

The Ga-face GaN was grown on 6H-SiC(0001) and the N-face GaN directly on a-Al_2O_3 (0001) by plasma assisted molecular beam epitaxy (PAMBE). The growth was controlled by in-situ RHEED measurements. After finishing the growth the samples were immediately transfered under UHV conditions to the directly attached analytic chamber (base pressure below $2x10^{-10}$ mbar). There the samples were in-situ examined by photoelectron spectroscopy (XPS and UPS) and atomic force microscopy (AFM).

The HREELS measurements were performed after sample transfer with a high vacuum transport system (base pressure below 10^{-6} mbar) into the HREELS system.
The Ga-face GaN surfaces show a 2x2 reconstruction measured with RHEED and a roughness below 1 nm (AFM). From the analysis of the HREELS- and XPS-spectra a surface depletion layer and a surface upward band bending of 0.4 eV results. Furthermore the UPS spectra exhibit the two surface states at 2 eV and 3 eV below the Fermi level, respectively.

14:30 Oral

Influence of thermal annealing on the morphological, structural and optical properties of GaN/AlN quantum dots

Marco A. Peres[1], A. J. Neves[1], Teresa Monteiro[1], Sérgio Magalhães[2], E. Alves[2], Katharina Lorenz[2], Hanako Okuno-Vila[3], Catherine Bougerol[4], Vincent Fellmann[4], Bruno Daudin[4]

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We present morphological, structural and optical properties of superlattices of MBE- grown self assembled GaN QDs in AlN. At low temperature the recombination of the smallest as-grown dots is dominated by a broad ultraviolet PL band with maximum at around 3.8 eV. When increasing the dot size, the main PL emission is shifted to lower energies with its maximum ranging between 2.9 eV and 2.7 eV in the studied samples. In both cases the QD recombination is detected up to RT. The grown samples were further submitted to thermal annealing treatments in nitrogen atmosphere at $1100^{o}C$. After annealing the emission of the GaN QDs embedded in the AlN spacer layers was found to be substantially modified: for the samples with smaller and medium sized GaN QDs a high energy shift of the PL peak position was detected. For the sample with larger dots an opposite trend was observed. In this case a low energy shift of the main emission was measured after thermal annealing. The structural properties of the multilayers were analysed using X-ray diffraction (XRD), X-ray reflection (XRR) and Rutherford backscattering spectrometry (RBS) showing excellent crystalline quality and interface properties. XRR indicates that after annealing intermixing effects are enhanced, which was confirmed by scanning transmission electron microscopy measurements for one of the samples with small dots. The influence of the intermixing and possible strain relaxation effects on the PL of differently sized GaN QDs will be discussed.

14:45 Oral

High p-type conductivity in Phosphorus-doped ZnO films.

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ZnO is a crucial member in the arena of exciton-based UV emitters because of its wide band gap (3.37eV) and large exciton binding energy (60meV) at room temperature. Fabrication of p-type ZnO is a big challenge as undoped ZnO is unintentionally n-type due to the presence of native point defects (oxygen vacancies and zinc interstitials). In this work, ZnO thin films were deposited on semi insulating <100>InP by Pulsed Laser Deposition (PLD) technique at 400ºC in

an oxygen ambient of 75mTorr followed by Rapid Thermal Annealing (RTA) of the samples at 500ºC, 550ºC, 600ºC and 650ºC. Atomic force microscopy (AFM) images revealed the growth of ZnO with roughness varying between 9.26nm to 22.28nm (root-mean-square) within a scanned area of 1µm^2. It was observed from the XRD results of grown ZnO <002> that the full width at half maxima (FWHM) of the annealed samples were narrower (~0.1428º) compared to as grown one (~0.2652º) due to improved crystallinity. Absorption coefficients calculated from the Reflectance data of UV/VIS spectroscopy decreased for the annealed samples (from 2.56um^{-1} to 1.02 um^{-1} at 360 nm). Temperature dependent (80K to 310K) Van Der Pauw Hall measurements confirmed stable p-type conductivity for the ZnO samples (resistivity~ .024 Ω-cm) annealed at 550ºC, 600ºC and 650ºC. The highest hole concentrations and Hall mobilities were measured to be 2.52x10^{18}cm^{-3}and 397cm^2/V-sec at 80K while their values at 310K were 5.41x10^{20}cm^{-3} and 19.8cm^2/V-sec respectively. *This is the best combination of hole concentration and Hall mobility reported so far for any phosphorus doped p-type ZnO films.* At 80K, the hole diffusivity calculated was 2.73 cm^2/s for the phosphorus impurities. From Arrhenius plots, Acceptor activation energy of 25.8±0.7 meV was calculated for P acceptors . Our future works are paving towards the fabrication and characterization of UV LEDs. We acknowledge Central SPM Facility of IIT Bombay for AFM study and DST for financial assistance.

15:15	Oral

Cathodoluminescence spectroscopy of silica capped Li doped ZnO nanoparticles

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Lithium doped ZnO nanoparticles have been prepared using wet chemical methods and capped with silica by the addition of TEOS (tetraethylorthosilicate) to the reaction mixture. Solutions of zinc acetate in methanol were prepared with a nominal proportion of lithium acetate added to achieve a molar percentage of 0% to 10% Li to Zn. A solution of potassium hydroxide (KOH) in methanol was then added dropwise to these solutions, under stirring, to achieve a pH of 10. The solutions were then left to react for an hour before the addition of TEOS and milliQ water, after which the resulting powders were harvested by centrifugation, rinsed several times and re-suspended in water. Each experiment was repeated without the addition of TEOS to yield uncapped particles as well.

The specimens were characterized using cathodoluminescence (CL) spectroscopy at 80 K and 300 K. The CL spectra of the uncapped ZnO powders all display a near band-edge (NBE) emission centred at 3.27 eV at 300 K, which shifts to 3.35 eV upon cooling to 80 K. The nominally 5% Li doped ZnO powder was the only specimen that displayed any defect emission, a broad peak centred at 1.95 eV at both 300 K and 80 K with the appearance of another peak centred at 2.42 eV at 80 K.

There was no ZnO NBE emission observed for any of the silica capped ZnO particles even at 30 kV. All spectra showed only a broad emission centred at 2.95 eV attributed to an intrinsic emission in silica. Analysis of the capped specimens by energy dispersive X-ray spectroscopy (EDS) confirmed the presence of Si in addition to Zn and O.

COFFEE

Wednesday afternoon, 16 September, 15:30
Main Hall

Joint Poster Session

Monday & Wednesday
Wednesday afternoon, 16 September, 16:00
Main Hall

Thursday, 17 September

Session 9

Thursday morning, 17 September, 9:00
Room 231

9:00	Invited oral

Nitrides lasers after BluRay

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The GaN-based BluRay laser diodes emitting light at 405 nm and powers up to 100 mW are manufactured in large quantities and for down-going prices. The presentation will deal with prospects of manufacturing new-generation laser diodes of higher powers, wide spectral range (UV- green), and reliability.

In the first part, I will discuss the most important issues related to the GaN substrate preparation: its size, orientation, misorientation, doping level. I will compare the properties of substrates available from other vendors with those of Unipress/TopGaN crystals.

In the second part, I will focus on the growth modes of InGaN layers (quantum wells) which are active parts of the light emitters. New results, as influence of the substrate misorientation on indium incorporation, and influence of growth parameters on the layer morphology, will be shown.

Then, such problems as layer cracking, creation of defects, sample bowing, all related to AlGaN/GaN lattice mismatch will be discussed. A new method of fabricating non-planar, laterally patterned laser diode structures will be proposed.

In the final part, the main limitations of introducing new devices, as droop phenomenon, low-growth temperature of green emitters, built-in electric fields, will be discussed and ways of overcoming those problems will be proposed

9:30 Invited oral

Application of modulated spectroscopies to nitride structures

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Electomodulated spectroscopy [photoreflectance (PR) and contactless electroreflectance (CER)] is known as a very sensitive and nondestructive absorption-like technique to investigate the optical transitions between the ground and excited states in various semiconductor structures including bulklike layers, quantum wells (QW) and quantum dots. Due to the Franz-Keldysh effect this technique can be also used to investigate the built-in electric field as well as the Fermi-level position at semiconductor surface and/or inside semiconductor structures. In this presentation the application of PR and CER techniques to study III-nitride structures will be presented. Two kind of semiconductor heterostructures will be discussed in this presentation. The first structures are GaN-based quantum wells which are dedicated for green/blue lasers and Ga(In)N/Al(In)N QW-based intersubband modulators. For these structures the optical transitions between the ground and excited states have been measured by contactless electroreflectance and compared with theoretical calculations performed within the electron effective mass approximation. For the second kind of GaN-based heterostructures (AlGaN/GaN transistor structures and so called Van Hoof structures) Franz-Keldysh oscillations were measured by PR and CER techniques. From the period of these oscillations the value of built-in eclectic field and/or the Fermi level position have been determined as well as the type of surface band bending has been identified. Obtained results will be discussed in the context of strong polarization effects in III-nitrides and their screening by the intentional and unintentional doping.

10:00 Oral

Structural models of GaN quantum dots in semipolar AlN

Efterpi Kalesaki[1], Joseph Kioseoglou[1], Thomas Kehagias[1], Antiopi Lotsari[1], Lise Lahourcade[2], Eva Monroy[2], Holm Kirmse[3], Wolfgang Neumann[3], George P. Dimitrakopulos[1], Philomela Komninou[1]

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The growth of nitride heterostructures along semipolar directions can help to partially alleviate the problem of carrier separation due to polarization-induced electrostatic fields, as well as the problem of the quantum-confined Stark effect. Further enhancement of the internal quantum efficiency of optoelectronic device active regions can be achieved by combining semipolar orientations with quantum dot (QD) nanostructures. Their morphology and strain state affect the band structure and internal efficiency of optoelectronic devices significantly, and it is thus required to study them in detail. In the present work, semipolar (11-22) and (10-11)-oriented GaN QDs were grown on (11-22) AlN by plasma-assisted molecular-beam epitaxy, following a modified Stranski–Krastanow growth mode [1]. Cross sectional and plan-view high-resolution transmission and scanning transmission electron microscopy (HRTEM and STEM) observations were employed in order to elucidate the nanostructures and their morphologies. The dominant QD morphology was that of truncated trigonal or tetragonal pyramid with chemically sharp interfaces. Three-dimensional structural models consistent with the experimental observations were constructed indicating that both types of QDs are bounded by (11-2n) and (10-1n) pyramidal planes. The energetically degenerate variants consistent with bicrystal symmetry were identified. Geometrical phase analysis [2] was employed for the elucidation of the internal strain state of the QDs and structural models of the pyramidal interfaces bounding the QDs were obtained using *ab initio* DFT.

Acknowledgement: Support under the 7th European Framework Project DOTSENSE (Grant No. STREP 224212) and the MRTN-CT-2004-005583 "PARSEM" project is gratefully acknowledged.

References

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[2] M.J. Hÿtch, E. Snoeck, and R. Kilaas, Ultramicroscopy **74**, 131 (1998)

10:15 Oral

Impact of stress on the optical properties of AlN layers

Georg Rossbach[1], Gerhard Gobsch[1], Ruediger Goldhahn[1], Marcus Roeppischer[2], Christoph Werner[2], Christoph Cobet[2], Norbert Esser[2], Armin Dadgar[3], Matthias Wieneke[3], Alois Krost[3], Barbara Bastek[3], Frank Bertram[3], Juergen H. Christen[3]

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The reversed valence band ordering of wurtzite AlN with respect to GaN leads to a very strong dependence of the lowest free excitonic transition energy on stress. The magnitude of strain depends on the epitaxial and thermal mismatch and can be influenced by the substrate choice, i.e. sapphire, SiC or silicon. So far, most studies focussed on the emission properties only, i.e. the analysis of photo- and cathodoluminescence (CL) spectra.

Here, we present for the first time a comprehensive study of the strain influence on the absorption properties, in particular on the energy spacing of the three valence bands at the center of the Brillouin zone and the corresponding oscillator strengths of the excitonic transitions. We compare the shape of the dielectric function (DF) of undoped and Si-doped AlN layers grown on different substrates

(sapphire, SiC, Si). The DF from infrared (0.9 eV) up to the vacuum UV (9.8 eV) spectral range is obtained from spectroscopic ellipsometry studies. The use of synchrotron radiation at low temperatures provides superior spectral resolution around the fundamental band gap of about 6 eV. As a result of the valence band ordering, the influence of the extraordinary DF on the ellipsometry spectra of the C-plane layers is more pronounced than for other semiconductors (like GaN). In order to analyze the optical response, we employ an anisotropic optical model for the AlN layers. Its validity is confirmed by measurements of an A-plane AlN film grown on R-plane sapphire substrate.

The excitonic transition energies, extracted from the DF; and the relative oscillator strengths are consistent with the results of strain-dependent **k·p** calculations for which the XRD-results serve as input parameters. Finally, the values for the lowest-energetic transition are in excellent agreement with the results of CL studies.

COFFEE

Thursday morning, 17 September, 10:30
Main Hall

Session 10

Thursday morning, 17 September, 11:00
Room 231

11:00 Oral

Acceptor behavior of N2O in MOCVD-grown ZnO thin-film transistors

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ZnO grown by metalorganic chemical vapor deposition (MOCVD) usually shows high density of n-type defects, and we showed that it could be improved by employing growth interruptions [1]. In this work, we studied the effect of nitrogen doping in ZnO films grown by MOCVD. Nitrogen doping was achieved by introducing N2O during growth. Nitrogen concentration was investigated by x-ray photoelectron spectroscopy (XPS) and secondary ion mass spectroscopy (SIMS). We observed that N2O doping shifted threshold voltage of thin-film transistor (TFT) by a large amount to positive direction. We explain that this positive shift is evidence of acceptor behavior of N2O. Our MOCVD system has a horizontal reactor operating at atmospheric pressure, using diethylzinc (DEZ) and oxygen for sources. XPS results showed a broad peak at 399.8 eV (N-H bonding), and the amount of nitrogen was estimated to be 0.47%. The presence of nitrogen was also confirmed by SIMS. We fabricated TFT structure (channel length= 15 μm, width= 500 μm, gate oxide thickness= 110 nm) by evaporating Al through a shadow mask. Current-voltage characteristics of two consecutively grown TFT's were compared. The difference between the two TFT's is the presence of N2O doping. Before annealing N2O did not cause much difference. After vacuum annealing at 450 deg C, threshold voltage of N2O doped TFT was shifted by +70 V. In contrast, the shift in TFT without N2O was only +30 V. We explain the large shift as a result of hydrogen escape during vacuum annealing. It is well known that N2O induces high concentration of hydrogen, which is n-type dopant. Acceptor behavior of nitrogen would be shielded by the hydrogen. During vacuum annealing, the excess hydrogen will escape, and underlying p-type dopants would show up. We conclude that N2O can be used as p-type dopants in MOCVD grown ZnO TFT's.

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11:15 Oral

Dimethylzinc and diethylzinc as precursors for monocrystalline zinc oxide grown by Atomic Layer Deposition

Łukasz Wachnicki[1], Małgorzata Łukasiewicz[1], Tomasz A. Krajewski[1], Grzegorz Łuka[1], Bogdan J. Kowalski[1], Bartłomiej Witkowski[2], Krzysztof Kopalko[1], Ewa Przezdziecka[1], Jaroslaw Domagala[1], Marek Godlewski[1,2], Elżbieta Guziewicz[1]

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A new area in the semiconductor technology is related to the change of the device architecture from planar to three-dimensional [1]. This imposes critical restrictions on temperature of the semiconductor processing as in 3D structures metal passes are placed not only at the top, but also below of the device. Typical temperature limit here is 350 ^{0}C. This temperature is much lower than typically used in the silicon technology, therefore a lot of efforts is focused now on new materials, which can be grown inside the above temperature limit.

One of them is zinc oxide, which is a II-VI semiconductor with a direct energy gap of 3.37 eV at room temperature. At present, ZnO has many potential applications as solar cells, piezoelectric transducers, ultraviolet light emitters or gas sensors. We show that zinc oxide is also an appropriate material for 3D applications, because monocrystalline ZnO layers can be obtained at temperature below 300^{0}C.

We present structural, morphological and optical properties of zinc oxide thin films grown by the Atomic Layer Deposition method at the temperature range between 200 ^{0}C and 350 ^{0}C. ZnO layers were deposited on a gallium nitride substrate using water as an oxygen precursor and diethylzinc or dimethylzinc as a zinc precursor. The obtained ZnO films show bright luminescence of excitonic origin. Defect-related luminescence bands observed in red and green spectral regions are correlated with the composition measured by the EDX analysis. A high resolution X-ray diffraction proves that our zinc oxide films are monocrystalline with FWHM of 00.2 peak equal to230 arcsec.

The work was partially supported by EU within the European Regional Development Found thorough grant Innovative Economy No. POIG.01.01.02-00-008/08.

[1] M. Godlewski et al., Microel.Eng. **85,** 2434 (2008)

11:30 Oral

Study of the flat band voltage shift in Metal/Insulator/n-GaN cacitors by annealing

Junjiroh Kikawa[1], Masamitsu Kaneko[1], Hirotake Otake[2], Tetsuya Fukushima[2], Kentaro Chikamatsu[2], Atsushi Yamaguchi[2], Yasushi Nanishi[1]

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The flat-band-voltage (Vfb) in metal/insulator (SiN or SiO_2)/n-GaN capacitors has been investigated focusing on an annealing temperature for post-deposit-annealing (PDA). We have found that the Vfb shifts to the negative bias direction as increasing the annealing temperature. Generally, a Vfb shift is known to be proportional to both the spatial trapped and interfacial charge density. If this shift was due to a change of traps distributed in an insulator bulk, PDA should cause a change of an insulator property. Refractive index of as-deposited SiN was 1.98 and it turned to 1.96 by annealing at 600 °C. This change exhibits that the annealing treatment pushes SiN to N-rich side slightly and N-rich SiN turns the Vfb shift to a positive direction. Thus, it is considerable that the negative shift of Vfb is due to the trapped charge mainly exists at interface but SiN bulk.

Estimated activation energies of a trap related to the Vfb shift are around 160 meV for SiN and 130 meV for SiO_2. The difference of these energies is small. This implies the each reaction of SiN/GaN and SiO_2/GaN interface is promoted by a same mechanism. In addition, these energies are too small compared with the formation energy of SiN, SiO_2, or GaN. This result suggests that the PDA does not produce new defect by breaking a bond for the Vfb shift. At the interface, a vacancy defect and an interstitial defect might form a dipole by Coulomb potential. In a deposit process of insulator, each dipole is faced at random direction because the process temperature is room temperature. It is considered PDA might work to face the random direction of dipoles to the same direction. As a result, they affect to the Vfb shift .

We also performed Kelvin force microscopy measurement to observe a charged state of SiN. The measured potential of 600 °C annealed SiN is around 1 eV lower in electron energy scale than as-deposited SiN one. The sign of this is reasonable to shift the Vfb for negative direction.

11:45 Oral

Electrochemical Analyses of Multi-Walled Carbon Nanotubes Composited Zinc Oxide Electrodes

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Multi-walled carbon nanotubes (MWCNTs) are one-dimensional nanostructured materials with many interesting properties such as high thermal and electrical conductivities. Wide band gap semiconductor material such as zinc oxide (ZnO) is also of interest. The ZnO-MWCNTs composite is introduced in the field of photocatalysis, sensor and solar cell, mainly focus on the improvement of ZnO properties. Electron transfer of redox species with electrode is the essential prerequisite for the application of ZnO-MWCNTs composite electrode for sensor and solar cell. Here, we investigated the electrochemical properties of ZnO-MWCNTs composite electrode. The techniques utilized for the characterization of the carbon composite semiconductor electrode were cyclic voltammetry and electrochemical impedance spectroscopy. The experiments were carried out in a conventional three-electrode system using home-made ZnO-MWCNTs composite film, platinum rod, and Ag/AgCl as working, auxiliary, and reference electrodes, respectively. The oxidation-reduction behaviors were studied using two different redox couples. The aqueous solution of $Fe(CN)_6^{3-}/Fe(CN)_6^{4-}$ has been used as a general model while the acetonitrile solution of I^-/I_3^- has been employed as a representative system for photovoltaic cell. Cyclic voltammogram of ZnO electrode containing 0.5 wt.% MWCNTs illustrated an increase of anodic peak current and a decrease of anodic peak potential for both $Fe(CN)_6^{3-}/Fe(CN)_6^{4-}$ and I^-/I_3^-. The electron transfer resistances at ZnO-MWCNTs electrode for both redox couples were significantly smaller than that of conventional ZnO electrode, indicating its much improved conductivity.

12:00 Oral

Radiation effects on ZnO thin films; observation of green emission

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Six ZnO thin films were grown under different oxygen flow rates by electrochemically onto the commercial indium tin oxide (ITO) substrate. X-ray diffraction (XRD), optical absorption and photoluminescence (PL) measurements were performed on all films. XRD measurements showed that the films are highly (0002) c-axis oriented. It has been observed that the growth rates of the films are highly dependent on the oxygen flow rates. High growth rate is obtained for the mid oxygen flow rates in the cell. Calculated crystallite size values have increasing trend as the oxygen flow rate increases. Absorption measurements have revealed that band gap energy of ZnO thin films are about 3.4 eV. PL measurements showed that the two emissions are observed in all films, which are band to band emission at about 3.37 eV and the so called blue emission at 2.66 eV in ZnO.

Relatively low dose ($5x10^{12}$ e^-/cm^2) and high energy electron irradiation (HEEI) (12 MeV) experiments were performed on all films. Its effects on the optical and structural characteristics were discussed. So called green emission in ZnO is observed as the effects of HEEI. In addition, annealing process was applied in order to understand the effect on the recovery of the HEEI induced defects.

Closing
Thursday afternoon, 17 September, 12:15

LUNCH
(maps available at the Reception desk)
Thursday afternoon, 17 September, 12:30
building of the Faculty of Physics

Posters

Monday, 14 September

Joint Poster Session
Monday & Wednesday
Monday afternoon, 14 September, 16:00
Main Hall

16:00 Poster C-1

Investigation of p-Si/$Cd_{0.3}Zn_{0.7}S_{0.8}Se_{0.2}$ heterostructures with intermediate nano-granular layer of CdTe

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Thin film p-Si/CdTe/$Cd_{0.3}Zn_{0.7}S_{0.8}Se_{0.2}$ heterostructures were fabricated using an electrochemical deposition method. Nano-granular thin films of CdTe with thickness 50-200 nm were deposited at room temperature on p-Si substrates from an aqueous solution containing $CdSO_4$ and TeO_2. Electrodepositing of the $Cd_{0.3}Zn_{0.7}S_{0.8}Se_{0.2}$ films onto the p-Si/CdTe substrates was carried out at room temperature from aqueous solution containing cadmium ($CdCl_2$), zinc ($ZnCl_2$), sodium ($Na_2S_2O_3$) and selenium (SeO_2 or Na_2SeO_3) salts. The deposition potential was controlled at– 0.82 V. The thickness of the films was 0.5 ¸ 1 mm. As ohmic contacts we used Al for the silicon and ZnO for the films $Cd_{0.3}Zn_{0.7}S_{0.8}Se_{0.2}$. The active area of heterostructures was 0.4 ¸ 0.8 cm^2. The electrical and photoelectrical properties of structures were investigated for various regimes of annealing in argon atmosphere. Atomic force microscopy (AFM) was used to evaluate the resulting surface morphology. X-ray diffraction analysis reveals that the heat treatment enhances the recrystallisation and shifts the CdTe peaks towards a smaller lattice parameter. It is established, that films CdTe that are deposited in the space charge region of structures reduce the concentration of defects and increase the device efficiency up to 11%.

16:00 Poster C-2

Electrochemical deposition of ZnO films on silicon with Ni buffer layer

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In the recent years, ZnO was accepted as one of the most promising material for optoelectronic and sensor applications. It is common knowledge that modern microelectronic technology utilizes exclusively silicon. A high-quality epitaxial deposition of ZnO on Si substrates would make it possible to combine various devises based on compound semiconductor with silicon circuitry.

In the present paper, we investigate the process of electrochemical deposition of continuous ZnO films on silicon wafers with thin Ni buffer from aqueous acidic nitrate solutions by the voltammetry method. Experimental data was compared with the data obtained on reproducible bulk platinum electrode. ZnO layers were deposited at 80°C in galvanostatic mode. Structure and composition of the deposited ZnO layers were studied by X-ray diffractometry, and X-ray microanalysis and SEM. Electrodeposited ZnO films were shown to have stoichiometric composition but polycrystalline structure with preferential crystallographic orientation (0002).

Optical properties of the deposited ZnO films were also investigated. ZnO films demonstrated strong photoluminescence in the visible range with maximum at 610 nm. Maximum of the excitation spectra is located at 375 nm (3,3 eV). This value corresponds to ZnO energy band gap. Hence, excitation of photoluminescence occurs via direct band-to-band transition. Potential applications in optoelectronics of the fabricated ZnO films are discussed.

16:00 Poster C-3

Parameter-free calculations of electronic properties and optical transitions of MgO, ZnO, and CdO

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Since Moore's law still holds, not only do computational capabilities evolute rapidly but at the same time the cost per GHz drops dramatically. This development allows the implementation of computationally expensive high-quality ab-initio methods that were out of reach only a few years ago. Undoubtedly, ab-initio methods are an inevitable tool for materials science and even high-end materials design.

We briefly introduce the theoretical concepts of one of the currently most reliable ab-initio approaches to the computation of electronic band structures. We use the nonlocal hybrid HSE03 functional as an approximation to exchange and correlation in a generalized Kohn-Sham equation to compute the starting point for the solution of the quasiparticle equation. Using this starting electronic structure we

compute quasiparticle energies within the GW approximation which involves screening from the fully frequency-dependent dielectric function. Spin-orbit coupling is included in our calculations.

We present highly accurate electronic band structures of MgO, ZnO, and CdO, which we use to derive fundamental gaps, effective electron and hole masses, crystal-field and spin-orbit splittings, optical transition matrix elements, and even exciton binding energies. Chemical trends are pointed out. Since we are not limited to the equilibrium lattice structures of these materials, we predict and compare to values for the wurtzite polymorphs of MgO and CdO. Both become important in alloys and heterostructures of MgO and CdO together with *wz*-ZnO, e.g., for band-gap tailoring. However, it is practically impossible to fabricate samples of sufficient quality for experimental studies.

16:00	Poster	C-4

Physicochemical approach to chalcogenide passivation processing of III-Vsemiconductor surfaces

Larisa V. Fomina[2], Serge A. Beznosyuk[1]

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Today II-V semiconductors find application for manufacturing of microwave electronics and optoelectronics devices. Their properties are in many respects defined by interfaces. Some of perspective directions of updating of the interface is chalcogenide passivation of the semiconductor surface. Using of water solutions of chalcogenide compounds at room temperatures allows to carry out passivation processing in soft conditions.

In report the comparative thermodynamic analysis of passivation processing semiconductor surfaces of gallium nitred, gallium phosphide and gallium arsenide is presented. For equilibrium chemical reactions between the semiconductor and water solutions of chalcogenide reagents some equations of electrode potentials are worked out and standard emf-values are calculated. It is shown that a variety of products of chalcogenide processing are always defined by the nature of elements of V group and oxidation degree characteristics of them. In all cases gallium passes into Ga_nX_m compounds, where X – sulphur or selenium. Components of V group pass typically into next products, as follows: in case of GaN into molecular nitrogen and ammonia, for GaP into soluble compounds of quinquivalent phosphorus, for gallium arsenide are possible arsenic chalcogenide formation.

Taking into account crystal chemistry characteristics of contacting substances and physical-chemistry properties of products the thermodynamical analysis of chalcogenide passivation allow to conclude that processing of the semiconductor surface promotes perfection of heterostructure of metal-semiconductor interface in comparison with the ordinary one containing oxide films. Being based on results of the carrying out physicochemical analysis of the semiconductor's surface passivation processes in water solutions of chalcogenide compounds some new regimes of chalcogenide processing are picked up and diode contacts Ni/GaAs, Ni/GaP with the improved characteristics are received.

16:00	Poster	C-5

Computer simulation of stability of gallium nitride nanofilm

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Today investigators take intense interest in gallium nitride nanofilms as to a perspective material for semi-conductor nanoelectronics. At 300 K its band-gap width is equal to 3,39 eV. It is a wide band gap III-V semiconductor. GaN bulk crystal is a strong chemical compound, as experimental values of atomization energy and bond length are equal, accordingly, 2,20 eV/at and 0,17 nm. Thin films on its basis are applied to creation of light-emitting diodes and the lasers radiating in dark blue area of a spectrum, for manufacturing of the electronic devices working at high frequencies, temperatures and in excited environments. Gallium nitride thin films crystallize in stable hexagonal (wurtzite) or meta-stable cubic (blende) crystal structures. It depends on substrate type, on which there is a growth of gallium nitride films. But the nature of their stability in diamond like lattices is not quite clear till now, as atoms of nitrogen and gallium have the big distinction in atomic radiuses and electronegatives.The purpose of presented work is investigation of gallium nitride nanofilm's stability. Using a wurtzite crystal lattice data initial GaN nanofilms with various number (from 160 up to 1200) atoms was constructed. After that variation procedure of energy minimization was used by nonlocal density functional method. The nanostructure optimization by algorithmic steepest descent method along the binding potential energy surface in a configuration space of nanosystem was performed. The main results were as following. 1. At small number of atoms values of nanofilm's atomization energy (1.82 eV/at) is essential below crystal one. 2. Value of nanofilm's atomization energy is increased with increasing of number of atoms, passing through a local maximum (2.14 eV/at) at 1000 atoms.3. Energy of lattice optimization counting per atom has also a maximum (0.037 eV/at) at 1000 atoms. 4. Equilibrium bond lengths (~0.20 nm) in all synthesized nanofilms are greater than bulk one.

16:00	Poster	C-6

Analytical potential for atomistic study of hydrogen bonding and diffusion in ZnO

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A new analytical potential and a bonding model were developed for O-H bond in ZnO, the model is based on the weekness of O-H bond in ZnO caused by the surrounding Zn-O covalent bond compared to O-H bond in OH molecule. In this model the OH interaction is de-

scribed by analytical bond order potential (ABOP) developed for OH free molecule where ZnH interaction is described by leannard jones potential since there is no evidence of ZnH bond formation in this material. The OH potential reproduce the structural, energetic and vibrational properties of OH and H_2O molecules in good way. Our model give a binding energy of 1.24 eV for H in BC site and 1.08 eV for AB site which appear to be very close to the hydrogen binding energy observed by N H Nickel in ZnO single crystal[1]. In the other hand, the barrier height for H hop between tow interstitial site is about 0.5 eV which is very close to DFT results[2,3].

References:

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16:00 Poster C-7

Optical, structural, and magnetic properties of V-implanted ZnO

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Optical, structural, and magnetic properties of V-implanted ZnO (ZnO:V) have been investigated by photoluminescence (PL), x-ray diffraction (XRD), and superconducting quantum interference devices (SQUID). The ZnO films of 100 nm thickness were implanted with 100 keV V ions to fluences (n_V) of 1.0 x 10^{15}, 2.5 x 10^{15}, 5 x 10^{15}, and 1.0 x 10^{16} cm^{-2} at room temperature and subsequently annealed at 300 ~ 900 ^oC for 20 min in an oxygen ambient. Near-band-edge (NBE) PL emission from ZnO:V was observed at ~ 380 nm, irrespective of n_V and the annealing temperature (T_A), and its intensity increased gradually with increasing n_V from 0 to 2.5 x 10^{15} cm^{-2}, but at $n_V \geq 5$ x 10^{15} cm^{-2}, it decreased. The NBE PL intensity of ZnO:V films at T_A = 900 ^oC was almost 6 times enhanced at n_V = 2.5 x 10^{15} cm^{-2}, compared to that of the unimplanted ZnO. The XRD peak of the ZnO:V films with n_V = 2.5 x 10^{15} cm^{-2} was observed dominantly at ~ 34° and was attributed to the ZnO (002) plane. Only the ZnO (002) peak was identified up to T_A = 800 ^oC, but at T_A = 900 ^oC, additional XRD peak corresponding to V clusters or V oxide phases was also observed. The M-H curves measured at 10 K exhibited strong ferromagnetism at T_A = 500 ^oC for the ZnO:V films with n_V = 2.5 x 10^{15} cm^{-2}. Possible physical mechanisms will be discussed to explain the experimental results.

16:00 Poster C-8

Effects of growth interruption of ZnO buffer layer on properties of ZnO epitaxial layer on Si (100)

Min Young Cho[1], Su Min Jeon[1], Hyun Young Choi[1], Ghun Sik Kim[1], Do Yeob Kim[1], Min Su Kim[1], Jin Soo Kim[2], Jong Su Kim[3], Joo In Lee[4], Jae-Young Leem[1]

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ZnO epitaxial layers with low-temperature (LT) ZnO buffer layers were grown on p-type Si (100) substrates by plasma-assisted molecular beam epitaxy (PA-MBE). The buffer layers with the thickness of 500 Å were grown by two step growth. The first (350 Å) and second (150 Å) step of the buffer layer growth were without and with interrupts, respectively. The growth of the second step was interrupted for 10 s, after the deposition of 7.5Å, 5 Å, and 3.75 Å that were repeated 20, 30, and 40 times respectively, while the O_2 plasma was kept constant during the growth. The structural and the optical properties of the ZnO epitaxial layers have been investigated by X-ray diffraction (XRD) and photoluminescence (PL). The full width at half maximum (FWHM) of the ZnO (002) diffraction peak was affected by the growth condition. The strain and the grain size were also changed. The PL intensity ratio of near-band-edge emission (NBEE) to deep level emission (DLE) of the ZnO epitaxial layers was enhanced with increasing the interrupt times. The FWHM of the NBEE and the DLE were decreased from 176 to 131 meV and from 868 to 573 meV, respectively. The DLE were blue shifted from 2.07 to 2.21 eV.

16:00 Poster C-9

Quantum computations in ZnO:Fe^{3+} system

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The last years a variety of quantum systems have been proposed as a possible quantum bits for quantum computers. In the search for new solid state materials zinc oxide can be an interesting alternative to silicon. In this case it is easy to eliminate the superhyperfine interactions in ZnO since nuclear spin-free isotopes of Zn are highly abundant (~96%). ZnO is thus one of the rare semiconductors that can be isotopically purified and that offers the possibility to combine electrical and optical methods to control the quantum state of spin qubits. The spin state of a single Fe^{3+} spin qubit could be readout by optical methods and it could by coherently manipulated using pulsed electron spin resonanse methods.

In present work, we assume an Ising-type interactions between nearest and second-nearest neighbors. This system must be subjected

to a magnetic field, constant in time with a sufficiently strong variation along the spin chain. Additional radio-frequency(rf) pulses allow the coherent control of the state of the system such that a quantum protocol can be realized. These rf-perturbations are used to implement universal quantum gates from which any unitary quantum algorithm can be constructed. We implemented Fredkin universal quantum gate on an Ising chain of length 4, taking into account first and second neighbor couplings. The coupling strength of second neighbors is much weaker than that of first neighbors which means that the dominant source for errors are near resonant transitions. The accumulated errors in the course of the quantum algorithm are quantified by the fidelity. We investigate its temporal behavior as well as its final value, as a function of the Rabi frequency and the coupling parameter. We performed also some preliminary studies using fluctuating Larmor frequencies, simulating tiny variations of the external magnetic field. This is a simple way to introduce decoherence into our model.

16:00	Poster	C-10

In-situ catalytic growth of Gallium Nitride nanowires using an environmental transmission electron microscope

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Group III-Nitride semiconductors have attracted special interest for applications in several optoelectronics devices. Multi-layer epitaxial structures required for various devices present high density of dislocations, which in turn reduces the efficiency of the device. One possible solution for this problem is to substitute semiconductor nanowires for epitaxial films. Nanowires often have stress-free surfaces and better electrical and optical properties. Vapor-liquid-solid (VLS) catalytic growth is one of the most common methods used to synthesize 1-D nanostructures. Although ex-situ catalytic growth of gallium nitride (GaN) nanowires has been achieved before, there is not a complete understanding of the role of the catalyst, the growth mechanism and the interface dynamic. This work presents a dynamic observation of the nucleation and growth mechanisms of GaN nanowires which were formed by direct reaction of ammonia (NH_3) with gold (Au) - gallium (Ga) alloy liquid particle.

An environmental scanning/transmission electron microscope (E(S)TEM), Tecnai F20, was used for in-situ observations. Au was deposited ex-situ on porous silicon membrane TEM ready grids by physical vapor deposition (sputering of a Au target). These grids were introduced into the E(S)TEM column and heted up to 400C using a GATAN heating holder. Next, 60 mTorr of Trimethylgallium (TMG) was introduced into the E(S)TEM column for 3 minutes to deposite Ga particles. The sample area and delivery lines were evacuated after closing the inlet of the TMG to the E(S)TEM column. Subsequently, the sample temperature was increased to 800C and 40-70 mTorr of NH3 was introduced into the column. Low and high magnification images and digital videos (15 frames per second) were recorded using Gatan Orious 600SC camera. JEOL 2010F TEM/STEM was used for ex-situ imaging (High Resolution TEM (HR-TEM) and STEM) and for chemical analysis (Energy Dispersive Spectrometry (EDS)) of the samples.

GaN nanowires nucleation started at 800C after 40mTorr of NH3 was introduced. They grew over the whole substrate, thus growth due to electron beam effect is discarded. Ex-situ analysis confirms the presence of Ga and N, as well as the wurzitic structure for GaN.

16:00	Poster	C-11

Interfacial structure of semipolar AlN grown on m-plane sapphire by PAMBE

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Interfacial properties of AlN grown on *m*-plane sapphire by plasma-assisted MBE have been investigated in the context of achieving high-quality GaN/AlN heterostructures with reduced internal polarization. We investigate ~200 nm-thick epilayers deposited either directly on *m*-sapphire or after 30 min of sapphire nitridation at the growth temperature (~720°C). The active nitrogen flux was tuned to obtain a growth rate of 0.3 ML/s (~280 nm/h) under Al-rich conditions. AlN was synthesized under a III/V ratio of ~0.95 to achieve two-dimensional layers. High-resolution transmission electron microscopy (HRTEM) identified the dominant film orientation as (11-22) semipolar, with the AlN basal planes aligned parallel to the (1-104) plane of sapphire. This result is consistent with *in situ* RHEED observations and x-ray diffraction studies. An interfacial zone comprising additional AlN nanocrystals with the *m*-plane orientation was observed. In the case of growth without nitridation, the average *m*-plane nanocrystal size was 30 nm, but it was increased significantly in the case of nitridated sapphire, with some nonpolar crystallites exceeding 100 nm. A good epitaxial relationship with the substrate was observed both for the case of semipolar as well as for nonpolar AlN, comprising patches of regularly spaced misfit dislocations which were characterized using circuit mapping. However, the periodicity of the interfacial structure was perturbed by interfacial roughness comprising nanofacets on the (1-104), (1-10-2) and {1-210} sapphire planes. Often these nanofacets were found arranged in v-shaped formations, and were correlated to the introduction of misoriented nanocrystals. The nonpolar and semipolar orientations are interrelated by a 90°<11-20> relative rotation leading to the formation of low-energy grain boundaries between them.

Acknowledgement: Work supported under the 7th European Framework Project DOTSENSE (Grant No. STREP 224212).

16:00	Poster	C-12

Binding energy of a hydrogenic impurity in vertical-coupled wurtzite GaN/AlGaN quantum-dots under hydrostatic pressure and external applied electric field

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In this work, we perform a theoretical study, of the binding energy of the ground state of a hydrogenic donor impurity in a vertical-coupled quantum-dot structure. We have considered the effects of hydrostatic pressure and external applied electric field and we have used a variational method and the effective mass approximation. The low dimensional structure consists of three cylindrical shaped GaN quantum-dots, grown in the *z*-direction and separated by $Al_xGa_{1-x}N$ barriers. Our results are reported for several sizes of the structure (well width, barrier thickness, and dot radius), and takes into account the strong built-in electric fields caused by the piezoelectricity and spontaneous polarizations [1,2], the variation of the impurity position along the *z*-direction, the effect of external applied electric field, and hydrostatic pressure. We have found that, for both, symmetrical and asymmetrical dimensions of the structures, the binding energy, as a function of the impurity position, has a similar behavior to what is shown by the electron wave function without the Coulomb interaction. Additionally, it is obtained that the presence of the electric field changes dramatically the profile of the binding energy, destroying (favoring) the symmetry in symmetric (asymmetric) structures. Regarding the effect of hydrostatic pressure, our results show that the binding energy increases due to the increasing of the Coulomb interaction mainly related to changes with the hydrostatic pressure of the dielectric constant. Also, depending on the impurity position, the binding energy can increase or decrease with the hydrostatic pressure mainly due to increases or decreases of the carrier-wave function's symmetry.

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16:00	Poster	C-13

Mn effect on structral, optical and magnetic properties of ZnO nanopowders

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ZnO and $Zn_{0.98}Mn_{0.02}O$ nanopowders were synthesized by the sol gel technique. Mn effect on lattice parameters was investigated by X-ray diffraction analysis. The size of nanoparticles was estimated 30-40 nm by SEM observation. Optical characterization and the effect of doping were carried out by means of Fourier Transform Infrared (FTIR) spectroscopy. Kramers-Kronig analysis was employed to evaluate the optical constants in mid-IR range and optical phonons frequencies of ZnO and ZnO:Mn powders. Refractive indices and extinction coefficients in UV region were measured from transmittance spectrum. Optical bandgap energy showed a redshift in Mn doped samples in comparison with pure ZnO. The Curie temperature of $Zn_{0.98}Mn_{0.02}O$ was determined and ferromagnetism state were observed below 140K.

16:00	Poster	C-14

CdTe homojunction nanowire diodes

Ionut Marius Enculescu[1], Elena Matei[1], Lucian Ion[2], Stefan Antohe[2]

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Electrodeposition in nanoporous templates represents an interesting method for the preparation of semiconducting nanostructures such as nanowires, mainly due to the possibility of up-scaling with relatively low costs, a quality important for applications. In the present paper we report our results regarding the preparation of CdTe homojunctions by using a single electrodeposition bath. In order to achieve this result we exploited the possibility of varying the electrical behaviour of the semiconducting segment by varying the deposition potential. This property was exploited in fabricating multisegment nanowires with taylored transport properties. Such an ingenious approach is much more convenient from the experimental point of view, opening possibilities to fabricate a new generation of ultraminiaturized devices. The wires were characterized for basic properties using scanning electron microscopy, energy dispersive X ray analysis, optical spectroscopy and X-ray diffraction. Measurement of the transport properties showed that we were able to tune the junction behaviour for both metal – semiconductor and semiconductor – semiconductor from ohmic to rectifying.

16:00	Poster	C-15

Electrical properties of CdTe:Br single crystals at high temperature

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CdTe crystals, doped by chlorine, have found an application for gamma- and X-ray detector production. The possibility of CdTe:I films to "feel" radiation was shown. Bromine also can be a very in-

teresting dopant for this purpose but there are few information concerning its behavior in CdTe. The goal of this work was to study the electrical properties and self-compensation phenomena in CdTe:Br single crystals at high temperature.

The crystals were grown by the Bridgman method and contained $3*10^{19}$ at/cm^3 of Bromine in the melt. Hall effect measurements were performed under well-defined Cd/Te vapor pressure at ~600-1200 K using the 6 contacts method. Temperature, component vapor pressure and temporal dependencies of specific conductivity, charge carrier density and their mobility were studied.

The electron density ([e^-]) at RT in as-grown samples varied from 10^{10} till 10^{17} cm^{-3} but after heating above 800 K it became essentially higher than in undoped CdTe, sometimes exceeding 10^{18} cm^{-3} at T>1100 K. It didn't depend on the Cd vapor pressure (P_{Cd}) till 1000 K and increased at higher temperature with P_{Cd} increasing. In heavily doped samples after heating above 900 K the [e^-] was above 10^{17} cm^{-3} and constant in a wide temperature range (500-1100 K). The electron mobility in CdTe:Br crystals usually was lower comparing with undoped CdTe due to the high concentration of dopants atoms. It was impossible to reduce the [e^-] sufficiently by P_{Cd} reducing or quenching. Only the treatment at Te saturation allowed decreasing the [e^-] more than 6 orders of magnitude (at RT). The observed strong self-compensation phenomena are discussed in the framework of the Krogers' quasichemical point defect theory assuming the presence in the samples of a large quantity of dopant acceptors (Bromine associates with Cd vacancies) and dopant donors (Br_{Te}).

16:00	Poster	C-16

ZnO Nanostructures for Photovoltaic Cells

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Organic and hybrid solar cells have received attention due to their potential to provide environmentally friendly and cheap source of energy [1]. Making use of nanostructures in such devices looks attractive as they provide percolative pathways for charge carriers by quantum confinement, helping in the improvement of the efficiency of the devices [2]. In this work the use of different ZnO nanostructures has been studied to assess the effect of morphology and surface passivation on the performance of photovoltaic devices. ZnO nanostructures (nanoparticles, nanowires and nanofibers) have been produced by different techniques, were functionalized by pyrene-1-carboxylic acid and characterized by XRD, UV-Visible spectroscopy and SEM. ZnO nanoparticles and ZnO nanowires were prepared by wet chemical synthesis and thermal decomposition of zinc acetate dihydrate respectively. ZnO nanofibers were prepared by calcination of polyvinyl alcohol/zinc acetate composite nanofiber, which had been produced by the electrospinning process. The photovoltaic devices have been prepared in two different configurations - glass/ITO/PEDOT:PSS/photoactive layer/Al and glass/ ITO/ZnO/photoactive layer/ PEDOT:PSS/Ag paste. The PEDOT:PSS and the photoactive layers were deposited by spin coating of the suspension of PEDOT:PSS in water and the suspension of the ZnO nanostructures in the poly-3-hexylthiophene solution respectively. The photovoltaic cells were finally characterized by current-voltage characteristics measurement under simulated standard illumination conditions.

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16:00	Poster	C-17

Physical properties of pure and Co-doped ZnO nanoparticules

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Pure ZnO and doped with Co (2%, 6%, 10%) nanopowders were elaborated by sol-gel method. As a starting material, acetate of zinc and cobalt, citric acid, ethylene-glycol and bi-distilled water were used to synthesize the powder. The prepared solutions were stirred at different temperature (100°C, 130°C, 140°C, 150°C) and calcinated at 400°C and 500°C for 4 hours.

The obtained powders were characterized by XRD in order to identify their structure and the grain size as a function of different elaboration parameters: temperature, duration of the gel in the solution, started material concentration and dopant concentration and by IR.

The obtained results show on one hand that the grain size varies between 20 nm and 55 nm for pure ZnO and 46 nm for doped powders and on the other hand that the crystalline structure is wurtzite for both pure and doped powders.

Further results were obtained by IR analysis confirming the previous ones and showing some new effects.

16:00	Poster	C-18

Structural and optical studies of pure and Al-doped ZnO nanopowders

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Structural and optical properties of pure and Al-doped ZnO were investigated using X-Ray diffraction (XRD), IR transmittance and Photoluminescence. The nanopowders were obtained by means of sol-gel method: the pure ZnO and Al- doped (Al: 1%, 3%, 5%, 7% and 10%) were the chosen materials for our study. The prepared

solutions were stirred at 60°C for 2 hours and calcinated at 500°C for 4 hours. The crystalline structure is wurtzite for both pure and doped powders and crystallites sizes become larger with doping. More characteristics were obtained by optical methods confirming the previous results and adding some effects as a function of different parameters.

16:00 Poster C-19

Structural and optical properties of ZnO epitaxial layers grown on ZnO buffer layers with different interrupts by molecular beam epitaxy

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ZnO epitaxial layers were grown on low temperature (LT) ZnO buffer layers/p-type Si (100) substrates by plasma-assisted molecular beam epitaxy (PA-MBE). The buffer layers were grown with different interrupts (4, 6, 9, and 19 times) in oxygen plasma. The interrupts time for all the samples was kept constant in 2 min. X-ray diffraction (XRD), photoluminescence (PL), scanning electron microscopy (SEM) were carried out to investigate the structural and optical properties of the ZnO epitaxial layers. The XRD rocking curves of all the samples show the typical peaks such as ZnO (002), Si, and ZnO (004). The XRD peak position was dependent on the interrupts times. The (002) diffraction peak position and the lattice constant of the ZnO epitaxial layer with the interrupts of 9 times were almost in agreement with the unstressed ZnO powder value of 34.43° and 5.2066 Å. The ZnO epitaxial layer with the 9 times of the interrupts also shows a narrower and more symmetric near band edge emission (NBEE) peak shape with a linewidth 111 meV. The SEM observation shows that the as-grown ZnO epitaxial layer grew in columnar shape. The surface morphologies were changed from worm-like shape to columnar shape with decreasing the interrupts times of LT-ZnO buffer layers. The thickness of the ZnO epitaxial layers were also affected by the interrupts times of LT-ZnO buffer layers.

16:00 Poster C-20

Structural, optical and electrical properties of a-$Si_{1-x}C_x$:H films synthesized by DC-magnetron co-sputtering technique

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In this contribution, SiC layers were fabricated in DC magnetron sputtering system on corning glass and p type Si(100)by co-sputtering of intrinsic silicon as target coated by 10 x 5 mm^2 sprigs (32 sprigs) of polycrystalline 6H-SiC in argon and hydrogen plasma mixture. The a-$Si_{1-x}C_x$:H thin films have been characterized by UV-Visible-IR spectrophotometry, Fourier transform infrared spectroscopy (FT-IR), secondary ions mass spectrometry (SIMS), electrical conductivity measurements and photoluminescence (PL).

The band gap energy values of a-SiC:H films, for thicknesses ranging from 0.18 to 2.5 µm, increases until to reach a constant value around 2.70 eV. This behaviour was observed as well in undoped material as in doped a-Si:H, and can be explained by the influence of hydrogen bonding on the valence band edge through the transforming of Si-Si weak bonds to Si-C bonds those correspond to the deep energetic levels in valence band.

FT-IR measurements clearly evidenced the absorption bands at around 660, 769 and 1108 cm^{-1}, which correspond to Si-C transitional phase ($Si_{1-x}C_x$ alloys, $x < 0.5$), SiC transverse optical phonons and Si-O stretching vibration, respectively. The Si-C absorption band is shifted to higher wave number than those for the 6H-SiC phonon mode (located at 780 cm^{-1}). The peak shift was attributed to the size effects of the precipitates.

The effect of a-$Si_{1-x}C_x$:H/pSi film thickness on interface and composition was investigated using dynamic SIMS. The presence of ^{16}O profiles with a high signal at the interface can be ascribed to the possible formation of silicon oxide during deposition; this result confirms the presence of Si-O band in the FT-IR spectra. The film composition is close to a structure as a-$Si_{0.75}C_{0.25}$:H noticeably identical from all thickness layers.

Photoluminescence spectra of the samples were observed in the visible range at room temperature with two peaks centred at 470 nm and 550 nm. The origin of the PL was discussed.

16:00 Poster C-21

Low-temperature deposition of ZnO thin films by DC reactive sputtering at high oxygen partial pressure

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Transparent conducting zinc oxide (ZnO) has received a vast amount of attention in the last few years. This material is both transparent and conductive with a wide variety of applications (solar cells, microelectronics, sensors). ZnO can be deposited by a variety of techniques such as radio frequency sputtering and direct current magnetron sputtering, pulsed laser deposition and molecular beam epitaxy. The material properties (structural, optical and electrical properties) of the ZnO thin film are critically related to the specific growth technique employed and to the growth parameters used.

Zinc oxide (ZnO) films were grown on corning glass and crystalline silicon substrates at room temperature and 100°C using DC reactive sputtering with an argon/oxygen mixture at constant working total pressure of 100 Pa. The ZnO thin films were investigated by optical transmission, X-ray diffraction (XRD), scanning electron microscopy (SEM), Rutherford backscattering spectrometry (RBS) and electrical conductivity measurements. The transmission measurements have shown that all films exhibit high transmittance in the 400–2500 nm range. The optical transmission is in an increasing order of oxygen content in the as-grown films. The crystalline structure of ZnO thin films indexed by XRD reveals hexagonal ZnO phase with cell parameters a=3.2499 Å and c=5.2066 Å. The XRD spectra consisted of a ZnO phase oriented with its c-axis with a strong (002) peak, indicating that the films are highly texturized along the c-axis perpendicular to the substrate surface. No diffraction from randomly oriented grains or impurity phases can be observed from the X-ray patterns related to the as-grown and annealed samples. The thermal annealing at 300°C have the effects of narrowing the diffraction peak, indicating that grain growth has occurred, and shifting the (002) peaks to higher 2q angles.V From the scanning electron microscopy images, significant changes are observed according to the oxygen partial pressure used during the film deposition. The elementary chemical analysis (EDX) reveals the presence of ^{66}Zn and ^{16}O in the ZnO layer. A very large increase in electrical conductivity, up to nine orders of magnitude, was observed in as-grown ZnO films upon vacuum-annealing at 300°C for one hour, reaching as high as 0.2 $W^{-1}.cm^{-1}$.

16:00 Poster C-22

Annealing effects of the initial Zn layers on properties of ZnO thin films grown by plasma-assisted molecular beam epitaxy

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ZnO thin films with initial Zn layers were grown on p-type Si (100) by plasma-assisted molecular beam epitaxy (PA-MBE). Before the growth of the ZnO thin films, the initial Zn layers were deposited on the Si substrates for 4 minutes and then annealed at the different substrate temperature ranging from 500 to 700 ^{o}C in oxygen plasma. The annealing effects of the initial Zn layers on properties of the ZnO thin films have been investigated by room temperature (RT) photoluminescence (PL), X-ray diffraction (XRD), atomic force microscopy (AFM), and scanning electron microscopy (SEM). In the UV and visible range, near-band edge emission (NBEE) at 3.3 eV and trap-state or deep-level emission (DLE) around 1.9 to 2.1 eV were observed. The as-grown sample showed a broad and asymmetric NBEE. The NBEE peaks of the samples with the annealed initial Zn layers (from 500 to 700 ^{o}C) showed narrower and higher symmetric shape. The PL intensity ratio of the NBEE to DLE for the sample annealed at 500 ^{o}C was the largest. The XRD rocking curves of all the samples showed the typical peaks such as ZnO (002), Si, and ZnO (004). The ZnO (002) peak intensity of the XRD spectra was relatively dominant. The PL spectra, the XRD spectra, the strain, and the surface morphology for the ZnO thin films were affected by the annealing process.

16:00 Poster C-23

The (10-10)/(2-1-12) grain boundary in nonpolar GaN:Atomic structure and influence on defect introduction

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In III-Nitride compound semiconductor nanotechnology, a lot of emphasis is placed on achieving good quality epilayers grown along nonpolar and semipolar orientations, in order to reduce the polarization-induced internal electrostatic fields and the quantum-confined Stark effect. During growth of nonpolar or semipolar nitrides on *r*-

plane or *m*-plane sapphire respectively, misoriented nanocrystals are introduced at the epilayer/substrate interface causing formation of threading dislocations (TDs) [1]. This phenomenon is facilitated by the high order of common symmetry conserved by the 90°<11-20> rotation which characterizes the misorientation between the nanocrystals and the matrix epilayer. In the present work we investigate by atomistic simulations, combined with high resolution transmission electron microscopy (HRTEM), the (10-10)/(2-1-12) grain boundary (GB) which was observed in *a*-plane GaN grown on *r*-plane sapphire. For the atomistic simulations a modified Stillinger –Weber empirical interatomic potential was employed. Two principal structural configurations were compared, in particular one comprising interfacial dislocations and a GB with interfacial disconnections i.e. dislocations with step character. It was found that the bicrystal comprising dislocations exhibits strain due to curvature at the points where the defects are located. On the other hand the GB comprising disconnections exhibits no curvature and is consistent the HRTEM observations. The Burgers vector of the disconnections is 1/6<2-203> as verified using circuit mapping. Due to nodal balance, two such defects can combine to give a TD emanating into the epilayer, in agreement with the experimental observations.

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16:00	Poster	C-24

Electrical and optical properties of zinc oxide layers obtained by Atomic Layer Deposition

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Due to its unique electrical and optical properties, zinc oxide is regarded as a very prospective material in electronic, photovoltaic and optoelectronic applications (including gas sensors, piezoelectric transducers, solar cells and cross-bar memories). All of these applications require stable and controllable in time electrical parameters of obtained zinc oxide thin films, possibly achieved without any post-growth treatment of the deposited ZnO layers.

We present the results of Hall effect measurements on zinc oxide thin films grown at low temperature (LT) by Atomic Layer Deposition (ALD) method using diethylzinc (DEZn) and deionized water as precursors. Our films were deposited at the temperature range between 60°C and 200°C. Electrical parameters of such ZnO layers may be scaled by adjusting the basic growth conditions, such as growth temperature and reciprocal relations between precursors' pulse/purging times [1]. In this way by introducing slight changes of deposition parameters we are able to obtain ZnO layers with substantially varied *n* concentration (from 10^{16} – $10^{20}cm^{-3}$) and Hall mobility (up to 100cm^2/Vs).

The present investigations' aim is to examine and compare the changes in electrical and optical parameters of ZnO films after the appropriate measurements performed at low temperature range. The relations between structural, optical, and electrical parameters of zinc oxide films will be shown, provided by composition analysis, cathodoluminescence, pholuminescence and Hall effect measurements. This helps to understand the nature of *n* type conductivity observed in ZnO thin films.

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16:00	Poster	C-25

Contactless electroreflectance of ZnSe layers grown by atomic layer epitaxy

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ZnSe is a wide band gap II-VI semiconductor which can be easily integrated with GaAs (III-V semiconductor) due to a small lattice mismatch between the two materials. This material can be also quite easily grown by atomic layer deposition (ALD), which is a very cheap growth technique in comparison to molecular beam epitaxy (MBE) or metalorganic chemical vapor deposition (MOCVD) techniques. ALD method provides uniform films with reproducible thickness, low stress and low defect density. Last years we observed a booming interest in this method, since it was applied for deposition of high-k oxides in 45nm generation processors. ALD can be also used for a monocrystalline growth and then is called atomic layer epitaxy (ALE). In this work we studied monocrystalline zinc selenide films grown by ALE. We applied contactless electroreflectance (CER) to study the optical quality of ZnSe layers grown on GaAs and glass substrates. So far CER spectroscopy has never been used to study semiconductor films obtained by ALE. In this measurement technique, instead of measuring the optical reflectance of the material, the derivative with respect to a modulating electric field is evaluated. This approach leads to resonance-like lines in optical spectra at energies corresponding to the singularities in the total optical density of states (TODS). The sharpness of CER resonances is directly associated with the shape of the TODS, i.e. the quality of the material. In the case of ZnSe layers studied in this work, clear CER resonances were observed at the energy of ~2.7 eV. It has been observed that the intensity of these resonances and their broadening depend on the growth conditions and the kind of substrate. In this work obtained CER results will be analyzed in the context of structural investigations for these materials.

16:00 Poster C-26

Structural and optical properties of $Mg_{0.1}Zn_{0.9}O$/ZnO multilayer grown by Pulsed Laser Deposition

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ZnO-based semiconductors are recognized as promising materials for optoelectronic application in ultraviolet (UV) light emtting diodes (LEDs) and laser diode (LDs). Because ZnO has a larger exciton binding energy (60 meV) which could lead to lasing action based on exciton recombination even at room temperature than other wide band gap materials. Another advantage is that it is easily controllable band gap from 3.3 to 4.0 eV by alloying ZnO with MgO. Additionally, In order to obtain high-performance UV LED devices, a heterojunction is one of the key structures to realize double confinement actions for electrons and photons in optoelectronic devices. MgZnO alloy is considered to be suitable potential barrier layer for ZnO. Recently, there are many studies on forming the ternary alloy of $Mg_xZn_{1-x}O$. However, most of the reports were focused on the fabrication of MgZnO alloy film and MgZnO/ZnO quantum wells (QWs) by molecular beam epitaxy (MBE).

In this study, $Mg_{0.1}Zn_{0.9}O$/ZnO multilayers were grown on p-GaN/sapphire (0001) by Pulsed Laser Deposition (PLD) with different thickness. Multilayer MgZnO design is expected to improve optical and thermal characteristics of LEDs with ZnO-based active regions. The structure properties of the $Mg_{0.1}Zn_{0.9}O$ multilayer were investigated by XRD, AFM and SEM and the optical properties were studied by PL.

16:00 Poster C-27

Growth and characteristics of annealed n-ZnO/p-6H-SiC heterostructure

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ZnO is under consideration for a variety of potential uses for optoelectronic devices due to its wide band gap of 3.37 eV and large exciton binding energy of 60 meV at room temperature. However, ZnO has difficult to obtain the high quality and reproducible p-type material. In an alternative approach, there have been some attempts to grow n-ZnO on other p-type materials fore realizing ZnO based p-n heterojunctions. One of the important factors of heterostructures is the close lattice match of the components. In this respect, 6H-SiC is a good candidate for ZnO based heterostructure devices. Considering the ZnO lattice, lattice mismatch has been estimated to be ~5 % between ZnO and SiC. Although SiC substrate has these many advantages, there have been only a few reports on the growth n-type ZnO on p-type 6H-SiC heterostructure diodes and these reports were mostly studied on growth by MOCVD or MBE. In this letter, we report on the growth and post-thermal annealing of n-ZnO/p-6H-SiC heterostructure by sputtering system and on their properties. The n-type ZnO films were grown on 6H-SiC substrate and post thermal annealed in the oxidation furnace. The surface morphology was observed with atomic force microscopy (AFM) and X-Ray diffraction (XRD) measurements were carried out for the ZnO films. Moreover optical properties of the ZnO films were studies with Photoluminescence (PL). We observed that the structural properties and crystallinity of n-ZnO/p-SiC are improved by post thermal annealing and obtained high quality of the ZnO films on SiC substrate, which might be attributed to the small lattice mismatch.

16:00 Poster C-28

Surface morphology of InGaN layers

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The aim of this work was to give new experimental information on how the growth conditions in metalorganic chemical vapour phase epitaxy (MOVPE) influence the morphology and microstructure of InGaN layers in order to find optimum growth conditions of light emitting diodes and laser diodes.

InGaN (In content up to 25%) and GaN layers (up to 30 nm) grown on GaN bulk substrates (with ultra-low dislocation density) were examined using the atomic force microscopy (AFM), X-ray diffraction (XRD) in order to learn about the atomic step morphology and microstructure influenced by the substrate misorientation (up to 2 degrees with respect to c-axis), flow of indium, of hydrogen and by growth temperatures. Concerning the step morphology, the following observations were made:

i) at low growth temperatures, when N2 is used as a carrier gas, GaN layers contain pinholes,

ii) the pinholes disappear when hydrogen is added to the carrier gas and then the step-flow-like morphology of the surface is observed (Fig. 1),

iii) when InGaN layers are grown using N2 carrier gas, no pinholes were observed, but the steps become jaggy,

iv) when adding hydrogen, the steps become linear, but the incorporation of indium is much smaller, as observed in previous papers.

The explanation of the experimental results is proposed based on higher oxygen incorporation (without hydrogen in the carrier gas) at low temperatures causing jaggy steps and pinholes, however, in all cases the oxygen content was below the detection limit in secondary

ion mass spectroscopy (SIMS).

Concerning microstructure, the following new data were obtained:

i) the substrate misorientation is accompanied by smaller indium incorporation into InGaN layers caused by a smaller step velocity in the step-flow growth mode (Fig. 2).

ii) This phenomenon gives rise to an additional surface roughening as the length of the terraces is not uniform.

iii) the indium content increases with the layer thickness.

16:00	Poster	C-29

High temperature defect equilibrium in ZnS:Cu single crystals

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This report presents high temperature defect equilibrium (HTDE) model for Cu-doped ZnS crystals based on high temperature electrical conductivity (HTEC) measurements. HTEC data were obtained under defined conditions of component vapour pressure and of sample temperature. The appearance of different slopes on HTEC isotherms can be explained by a two-carrier conduction mechanism involving electrons and holes. All HTEC isotherms investigated can be divided into four regions. The high Zn vapor pressure region characterized by positive HTEC isotherm slope caused by native donors changes into negative slope caused by the decrease of Cu solubility. The slope of HTEC isotherm change negative again at low Zn vapor pressures. It is caused by increasing part of holes in bipolar conductivity. Analogous behavior of HTEC isotherms is observed also at low sulphur vapor pressures. The result is surprisingly similar to HTDE models for Ag-doped CdS [1] and for doped CdTe [2]. The method for solving the system of quasi-chemical reactions without approximation [3] was used. Our experimental data can be explained only by the inclusion of abnormal site occupation i.e. by antistructural disorder. This involves the occurance of sulfur at a zinc lattice, which must be expected to be a donor. Association of copper with this antistructure defect occurs also. Antistructure disorder disappears with increasing of Zn vapor pressure. Preliminary HTDE isotherms and isobars containing antistructure disorder and Cu solubility limitation are proposed.

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16:00	Poster	C-30

Effects related to deposition temperature of ZnCoO films grown by Atomic Layer Deposition – uniformity of Co distribution, structural, electric and magnetic properties

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ZnCoO films are the promising semiconductor material for spintronic applications [1]. For these applications films should be highly conductive and ferromagnetic (FM). The present situation is however very confusing. Ferromagnetic phase was observed by some groups and not by others. In most of the cases ZnCoO films showing FM phase were deposited at temperatures well above 300°C. This indicates that the growth temperature and deposition method (as shown by Ueda et al. [2]) may affect magnetic properties of the obtained films. Moreover, it is still controversial if high conductivity can be achieved in films containing Co. A similar situation was observed for ZnMnO and was related by us to nonuniform Mn distribution, formation of foreign phases [3], and carriers trapping by Mn ions.

In the present study we report properties of ZnCoO films grown at relatively low temperature by Atomic Layer Deposition (ALD), using two reactive organic precursors (DEZn and DMZn). The use of these precursors allowed us the significant reduction of a growth temperature to 300 ^{o}C and below. The so-obtained ZnCoO layers were studied as as-grown and after a thermal treatment. The influence of growth conditions on the Co distribution, structure and magnetic properties were investigated using the SIMS, SEM, CL, EDX, XRD and SQUID methods. We demonstrate that the films grown at low temperature are uniform. Such films are paramagnetic. For non-paramagnetic films formation of foreign phases was evidenced using the high resolution EDX method.

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16:00 Poster C-31

Nitrogen-doped and Antimony-doped ZnO thin layers grown by MO-VPE

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ZnO, the wide band gap semiconductor, is a very promising material for blue and UV applications meanwhile the p–type conductivity in ZnO is difficult to obtain routinely. In general, the p-type doping is obtained from the substitution of oxygen atoms by column V elements.

In this study the Nitrogen-doped and Antimony-doped ZnO layers have been grown by metal organic vapour phase epitaxy (MO-VPE) on sapphire and ZnO substrates at high temperature (950 °C) and low pressure conditions (50 torr). Nitrous oxide and diethyl-zinc as the oxygen and zinc precursors have been used. The incorporation of nitrogen and antimony has been obtained from the decomposition of tributhyl-nitrogen and triethyl-antimony molecules respectively. According to the growth parameters, the layers show a flat surface or a structured surface and sometimes with some nano-structurations. The nitrogen and antimony incorporation was checked by secondary ion mass spectroscopy (SIMS), the impurities are ranged from 10^{18} to 10^{20} at/cm^{-3}. The doped layers were investigated by optical characterisations: Raman scattering and photoluminescence spectroscopy. In N-doped layers we have observed several Raman lines from local vibrational modes related to nitrogen. Low temperature photoluminescence spectra exhibit donor-acceptor pair (DAP) transitions around 3.23 eV on both N-doped and Sb-doped layers. The DAP transitions have been investigated from temperature-dependant or excitation power-dependant experiments.

16:00 Poster C-32

Charge carrier mobility in ZnCdTe and ZnSe

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Usually the charge carrier scattering processes in ZnCdTe and ZnSe are considered in relaxation time approximation. However, these models have essential shortcoming – they are long-range which contradict special relativity. From the other side in [1,2] the short-range models of electron scattering were proposed for CdHgTe and CdHgSe in which the above mentioned shortcomings were absent. The purpose of the present work is to use this approach for description of the charge carrier scattering in ZnCdTe and ZnSe.

For the charge carrier scattering on the nonpolar optical and acoustic phonons, neutral defects, disorder and static strain potential the interaction radius of the short-range potential is limited by one unit cell. For the charge carrier scattering on the ionized impurity, polar optical and piezoelectric (piezoacoustic and piezooptic) phonons the interaction radius of the short-range potential is founded in a form $R=g\ a$ (a - lattice constant, g – the respective adjusting parameters).

To calculate the conductivity tensor components the method of a precise solution of the stationary Boltzmann equation was used [3]. The temperature dependences of the charge carrier mobility in the range 4.2 – 370 K in $Zn_xCd_{1-x}Te$ ($0\leq x\leq 1$) and ZnSe crystals are calculated. The influence of the different scattering mechanisms on the charge carrier mobility is considered. A good agreement between theory and experiment in all investigated temperature range is established. The scattering parameters g for different scattering modes are determined.

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16:00 Poster C-33

Thermostimulated electron-hole processes in GaN and AlGaN thin films.

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The AlGaN, GaN and InGaN based devices are composed of layers having both the distinctive lattice parameters and the different donor or acceptor doping. The characteristics of corresponding localized states are affected by the layer tensions, formation of the compensating point defects, the threading dislocations as well as by the eventual interface region between layers. Actual problem is experimental analysis of the nature, characteristics and electron recombination mechanism of defects as well as understanding of their formation related to the layer composition and growth conditions. Due to the semi-insulating character of these materials most electrical characterisation methods, e.g. DLTS, are not applicable. In order to get an experimental access to the defect states of strongly compensated wide band gap materials we applied thermostimulated luminescence (TL) and thermally stimulated current (TSC) techniques on the MOCVD grown GaN and $Al_{0.18}Ga_{0.82}N$ samples deposited on sapphire. The TL (emission band at E_{em}=1.95 eV) of GaN excited by E=3.5 eV at 10K showed five, typical for different samples peaks at 32 K, (E=9 meV), 82 K (E=29 meV), broad unresolved peak at 125-160 K and two overlapping peaks at 238 K (E=0.53 eV) and 273 K. The TL peaks at 32–82 K and 238–273 K was accompanied by a broad unresolved TSC peaks whereas no TSC was detected for the 125- 160 K TL peak. Observed essential change of the TL/TSC peak intensities ratio indicates that the released charge carriers have different mobility. Thus the peak at 125-160 K is due to the release of holes from Ga vacancy related centres. TL of the AlGaN (emission band at E_{em}=2.1 eV) excited by E=3.88 eV (absorption edge E=3,78 eV) showed intense broad peak from 15 K to 120 K

$_{max}$=75 K), and two broad peaks at 150 K and 250 K indicating the compositional disorder. The TL of GaN underlying AlGaN due to presence of layer tensions showed broadening of all peaks compared to the freestanding GaN.

16:00	Poster	C-34

Growth of non-polar (10.0) ZnO thin films on MgO (100) using rf-magnetron sputtering

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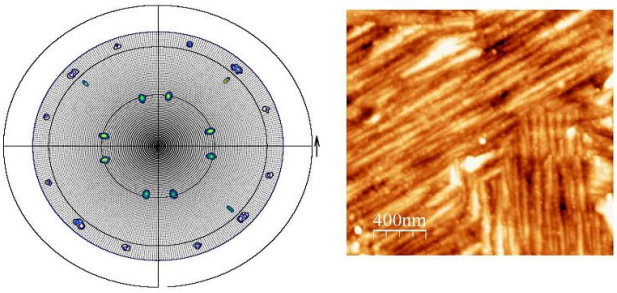
We report the deposition and growth of *m*-plane non-polar (10.0) ZnO oriented thin films on MgO (100) substrates using the rf-magnetron sputtering technique. The key parameters temperature, oxygen partial pressure and substrate/film lattice mismatch must be combined to yield a preferred growth orientation. HRXRD and AFM microscopy reveals macroscopic single domain structures with 13º rotations relative to the substrate due to in-plane MgO [011] alignment with the ZnO [01̲1]. PL emission and Raman spectroscopy were the techniques used for thin film optical characterization.



a) b)

a) X-ray diffraction pole figure of the (10.1) ZnO reflection at 2Θ=36,21° for the m-plane ZnO epilayer on MgO.
b) AFM image indicating different orientations.

16:00	Poster	C-35

Electronic structure of sputtering deposited undoped and Ga-doped ZnO thin films on a commercial Cz-Si solar cell substrate.

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ZnO is a promising material for use in solar cell applications requiring antireflective coatings and transparent conducting materials in front contacts. Its resistivity can be reduced by appropriate doping with different group III elements, which act as donors, without sacrificing optical transmission. These characteristics makes it one of the most promising and cheap transparent conducting oxides (TCOs).The interface between Si and a TCO like ZnO is a critical part of Si-based solar cells, since the conversion efficiency may be profoundly altered by the presence of recombination centres formed at the interface. We have carried out a thorough analysis using photoelectron spectroscopy (PES) and ultraviolet photoelectron spectroscopy (UPS) of the chemistry of the ZnO:Ga/Si interface formed when a TCO film is deposited by RF magnetron sputtering on a wafer Si solar cell.

16:00	Poster	C-36

SURFACE MORPHOLOGY OF ZnO/ZnS bilayer obtained by Chemical Spray Pyrolysis

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The efficiency of solar cells dramatically depends on light that can arrive to the active layer. Due to reflection losses, the photo generated current density (and consequently, the efficiency) reaches a level much lower than he maximum for the standard terrestrial spectrums of light coming for the sun To reduce the reflection of incident light at the surface of the cell, an antireflecting coating (ARC) can be used over the cell. This coating can range from a simple layer to a multilayer system of many layers having almost zero reflectance over a wide range of wavelengths. ZnO/ZnS bilayers to antireflection coatings for solar cells have been prepared by spray pyrolysis using mixed aqueous solutions of zinc acetate dehydrated and thiourea or zinc chloride and thiourea. The surface morphology of the bilayer are investigated as a function of the initial solution.

In general, ZnS thin films prepared from both solutions have smooth, homogeneous and dense surfaces, without cracks or hollows. ZnO/ZnS bilayers show a less smooth surfaces produced by the ZnO thin film prepared over the ZnS thin film. The ZnO layer covers the ZnS film completely; the surface of the ZnO film shows a surface with a slow ruggedness without cracks or hollows. When the ZnS inner film of the ZnO/ZnS bilayer has been annealing at 773 K in N_2 atmosphere during 1 hour, it shows the ruggednest surface.

16:00	Poster	C-37

Far-infrared and Raman Spectroscopy of $CdTe_{0.97}Se_{0.03}$(In)

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Far-infrared reflectivity and Raman spectra of $CdTe_{0.97}Se_{0.03}$(In)

single crystals were measured in 40-500cm^{-1} spectral range at 80 and 300K temperatures. Studied material had been produced with the Bridgman method at the Institute of Physics, Polish Academy of Science, Warsaw. The analysis of the far-infrared spectra was made by a fitting procedure based on the dielectric function which includes spacious distribution of free carrier influence on plasmon-phonon interaction. In undoped sample ($CdTe_{0.97}Se_{0.03}$) two optical mode frequencies (TO/LO pairs) at about 147/169 and 173/177 cm^{-1} were obtained. Plasmon-phonon interaction is weak.

In doped sample concentration of free carriers increasing and plasmon-phonon interaction is stronger. In that case, like in some other II-VI semiconductors[1], with high free carrier concentration, a surface layer with a low concentration of free carriers (depleted region) could be formed. Then, from this surface layer we can clearly see phonons. While the light penetration depth is greater than the surface layer thickness, we see both, coupled and non-coupled modes of the mixed crystals in the reflection spectra. In the Raman spectra we did not registered any influence of doping. The excellent agreement between the experimental data and theoretical spectra supports this assumption.

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16:00 Poster C-38

Raman scattering from ZnO doped with Fe, Mn and Co nanoparticles

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The purpose of this work was to study optical properties of nanosized powders of ZnO doped with Fe_2O_3, MnO and CoO. Nanocrystaline samples were synthesized by wet chemical method. Samples were characterized by X-ray diffraction to determine composition of the samples. The mean crystalline size varied from 8-52 nm for ZnO doped with Fe_2O_3, between 9-100 nm for ZnO doped with MnO and between 14-156 nm for ZnO doped with CoO.

In this paper we report the experimental spectra of micro-Raman measurements, at different laser powers. With increases of laser power, the peaks shift to lower frequencies and broaden. In all samples Raman peak at 436 cm^{-1} is clearly visible. This peak is typical for undoped ZnO nanoparticles [1].

For the samples of ZnO doped with Fe_2O_3 the Raman spectrum shows the band at ~ 642 cm^{-1}. The presence of this band is typical for Fe doped ZnO nanoparticles [1]. In these samples, especially in case of high doping level, bands from various crystal structures are identified (Fe_2O_3, $ZnFe_2O_4$) from about 514 to 660 cm^{-1}.

For the samples of ZnO doped with MnO Raman peak at ~ 660 cm^{-1} is clearly visible. This peak is typical for spinel structure. In these samples especially in case of high doping level, bands from various crystal structures are identified ($ZnMnO_3$, Mn_3O_4) at about 315 and 525 cm^{-1}.

For the samples of ZnO doped with CoO the Raman spectrum shows the band at ~ 691 cm^{-1}. The presence of this band is typical for Co_3O_4. We also observed bands at ~ 194, 482, 521 and 618 cm^{-1}. These bands are typical for Co_3O_4, too.

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16:00 Poster C-39

Electronic and magnetic properties of Co-doped ZnO: first principles study

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In order to investigate the electronic and magnetic properties of Co-ZnO alloys, we used a full-augmented-plane-wave (FPLAPW) method within the density functional theory (DFT), as implement in the WIEN2K package. This work is carried out within the LSDA approximation as the exchange-correlation potential. We have modelled ZnO doped with 6.25%, 12.5%, and 18.75% of Co. It pointed out that the band gap and the magnstic moments increase with the atomic fraction of Cobalt. The $Zn_xCo_{1-x}O$ is found to be a semiconductor, where the filled-states are located in the valence bands and the empty ones above the conduction dand edge. The filled and empty d-states are also shown to shoft downwards and upwards in the valence and the conduction bands, respectevly, with increase in the U potential. The total density of states (TDOS) and local density of states (LDOS) of the d electrons shed light on the evolution of the elctronic properties.

16:00 Poster C-40

Influence of structural defects upon electrophysical and scintillation characteristics of wide band gap ZnSe(Te) crystals for UV sensors and X-ray detectors

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ZnSe crystals are widely applied in the modern multichannel low-energy radiation devices of X-ray image visualization (medical tomographs, customs introscopes, devices of antiterrorist purpose, etc.), and in UV sensors as Shottky diode.

The work is devoted to research of possibility of obtaining of ZnSe

crystals by Bridgman method with a high level of the structural perfection, the increased mechanical strength and the established electrophysical and scintillation parameters for use in the radiation detectors.

In this work it is investigated the impurity composition of ZnSe(Te) charge and crystals grown up in the graphite crucible at isovalent doping by ZnTe, and the analysis of possible ways of their contamination by chemical impurities is carried out. Influence of the chemical purity of initial raw material, ZnSe(Te) crystal growth conditions on the formation of structural defects of the different type (dislocations, twin boundaries, etc.) is determined.

Influence of tellurium doping on the structural perfection, electric properties and scintillation parameters of ZnSe(Te) crystals is studied. The optimum mode of thermal treatment of ZnSe crystals is determined, that on condition of tellurium doping with 0,3...0,6 % concentration range has allowed to obtain scintillators with a high level of structural perfection (blende free of packing defects), the increased mechanical strength (increase in breaking point on ~ 20 % in comparison with undoped ZnSe), the specified light output (within of 110-120 % relatively CsI(Tl)), time decay 5-10 ms and afterglow less 0,005 % through 10 ms after X-ray action. It has enabled in Institute for Scintillation Materials to create pilot production of ZnSe(Te) scintillation crystals corresponding to the best world samples for the introscopes and medical tomographic equipments.

This work was supported by the U.S. Civilian Research and Development Foundation (CRDF) and NATO Science for Peace and Security Program (Project SfP-982823).

16:00	Poster	C-41

Surface morphology studies of ZnO layers grown using MOCVD

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ZnO is an attractive material, especially due to exciting optical properties. Therefore, its application fields can be expanding from the SAW devices to the light emitting diodes (LEDs).

In this work, we first investigated the surface morphology of ZnO epilayers grown on C-, M-, A- and R-plane sapphire by metalorganic chemical vapour deposition (MOCVD) at low pressure and high temperature. Diethylzinc and N_2O were used as the zinc and oxygen precursors respectively. The carrier gas was helium. The varying growth parameters were the substrate temperature, the reactor pressure and the VI/II partial pressure ratio. ZnO layers were characterised by atomic force microscopy (AFM), scanning electron microscopy (SEM), X- ray diffraction (rocking curve), Raman spectroscopy and photoluminescence.

Figure 1 shows different surface morphologies of ZnO layers grown on sapphire substrates. On R-plane, SEM image exhibits a rather smooth surface, and the mechanism of growth of ZnO is "quasi 2D". On C- plane, the surface is rough and shows 3D islands, and, on M-plane, a high density of defects can be observed.

Subsequently, we studied the surfactant effect of a triethyl-antimony (TESb) flow on the surface morphology. Whereas the ZnO film grown on C-sapphire substrate exhibits a rough surface, the ZnO:Sb layer shows a smooth surface.

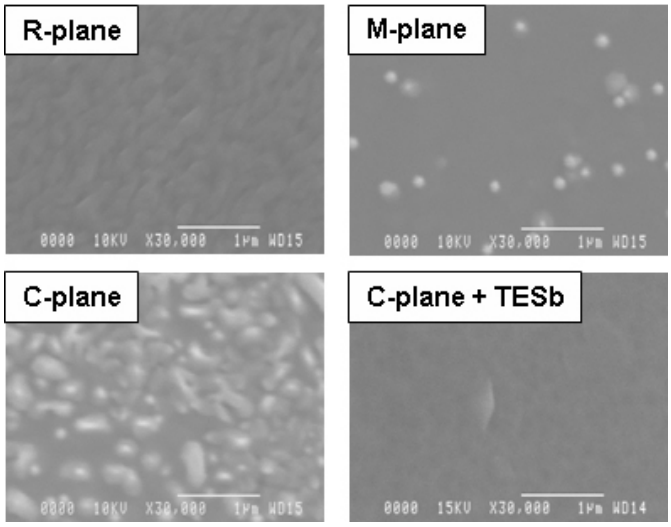

Figure 1 : SEM images of ZnO layers grown on R-plane sapphire, M-plane sapphire,C-plane sapphire, and ZnO:Sb grown on C-plane sapphire.

16:00	Poster	C-42

Comparative study of Mn-doped II-VI semiconductors in form of bulk crystals, thin films and nanocrystals

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In last decades there has been a great deal of study focused on transition metal doped II-VI wide band gap semiconductors because of their attractive fundamental properties and numerous application possibilities. Among the most spectacular of these properties is the giant Faraday rotation, which in $II_{1-x}Mn_xVI$ alloys is two orders greater than that in conventional semiconductors. The Faraday rotation in bulk crystals can be significantly changed by varying kind and content of magnetic ions, temperature and external magnetic field. Recent progress in nanotechnology opens new way for further flexibility in such "Faraday rotation engineering". In this work, we present results of optical and magneto-optical studies of $II_{1-x}Mn_xVI$ bulk crystals, thin films and nanocrystals. The $Cd_{1-x}Mn_xS$ single crystals were grown using vapor transport method. For growth of $Cd_{1-x}Mn_xTe$ single crystals Bridgman method has been applied. Growth of thin layers of both materials has been performed by pulsed laser deposition and RF-diode sputtering technique. Pulsed laser deposition technique has been also modified for fabrication of $Cd_{1-x}Mn_xTe$ nanocrystals embedded in SiO_2 matrix. The synthesis of $Cd_{1-x}Mn_xS$ nanoparticles was performed by colloidal chemistry technique at room temperature and under ambient conditions. Optical absorption and Faraday rotation measurements

were carried out in range of (320-900) nm at temperatures of (5-300) K. The observed spectral dependence of the Faraday rotation for $Cd_{0.97}Mn_{0.03}S$ colloidal nanoparticles in polyvinylalcohol solution suggests of exhibition of a negative part due to s p-d exchange interaction contribution. This is case typical for semimagnetic semiconductors with large exchange interaction between manganese ions and band carriers. Possible mechanisms for the observed difference between spectral, magnetic field and temperature dependence of the Faraday rotation for the studied materials in form of bulk and nano will be discussed.

16:00	Poster	C-43

Hybridization and magnetic anisotropy of S-state ions in wurtzite DMS

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Diluted magnetic semiconductors (DMS) have become recently the subject of considerable interest as promising materials for spin electronics. In particular, Mn-doped II-VI and III-V DMS is extensively explored both experimentally and theoretically. The control of magnetic anisotropy is proved to be essential in fabricating spintronic devices. In the present communication we propose a theory for single ion anisotropy (SIA) of S-state ions in DMS with the wurtzite structure. For the calculation of the spin Hamiltonian (SH) parameters we start from a realistic p-d model. The model includes the Hamiltonian of isolated S-ion (Coulomb repulsion, spin-orbit coupling and p-d hopping). A canonical Schrieffer-Wolff-like transformation reduces this model to an effective single ion Hamiltonian. The ligand field parameters are expressed in function of ligands coordinates and p-d hybridization. Then, this Hamiltonian is numerically diagonalized and treated by pertubative approach which enable us to obtain explicit analytic formula for SH parameters, a-F and D. The results of the calculation are found to be in good agreement with our EPR measurements on ZnO:Mn thin films and the available EPR data on Mn2+ and Fe3+ for other II-VI compounds (ZnX and CdX, X = S, Se, Te).

16:00	Poster	C-44

Synthesis of aluminum nitride nanopowder; compare methods for determination of the grain size in nanopowders

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Ultrafine grain and nanocrystalline materials show attractive properties. Aluminum nitride, well known semiconductor, as a nanopowder has got unique physical and chemical properities. It is attractive not only for applications in laser diodes, solar blind Photodiodes (PD), UV Light Emitting Diodes (LED) and high- temperature electronic devices but also for future light emitting systems based on aluminum nitride nanopowder and polymers.

The main goal of our research was to develop a method to synthesize nanopowder of aluminum nitride, which will be free from carbon. The reaction was carried out in horizontal tube furnace. As a starting material we used a mixture of nanopowder α-Al_2O_3 (produced by Alfa-Aesar, particle size less than 60nm) and aluminum powder (3-5% by weight). As a source of nitrogen gaseous ammonia was used. The reaction can be expressed as follows:

$$Al_2O_3 + 2NH_3 \rightarrow 2AlN + 3H_2O$$

The process was carried out in the temperature range 1050 -1350°C for time 1- 5h. During our experiment we validated influence of parameters like: temperature, ammonia rate, duration of reaction, the regime of heating and cooling. The samples were investigated by: powder X-ray diffraction (PXRD), Brunner-Emmett-Teller method (BET), scanning electron microscopy (SEM) and ramman studies.

16:00	Poster	C-45

The electronic energy band spectrum of A_2B_6 crystals evaluated with the density gradient corrected exchange-correlation potential derived from the new functional

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Electronic energy bands of ZnS, ZnSe, and ZnTe crystals have been evaluated within the mixed basis approach [1-3] with using the core Bloch states and plane waves. The exchange potential has been derived from the new exchange-energy functional including the density gradient corrections.

The aim of this paper consists: (1) in the employing more accurate generalized gradient approximation (GGA) for solids, suggested in [4], within the mixed basis approach; (2) in using the simplest generalization of GGA (denoted GGGA in [5]); (3) in applying for crystal the new exchange-energy functional, derived in [5] and successfully employed to atoms; (4) in verifying of the parameter β found to be good for atoms in application in crystals; (5) in searching for the parameter β reliable for crystals.

The electronic energy bands have been calculated with exchange and correlation potentials suggested in [6], and also with their versions named revPBE and RPBE.

The semicore states energies obtained within the mixed basis approach by means of the new exchange-energy functional, suggested in [5], are in better agreement to experiment than ones calculated with pseudopotentials in LDA approach.

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16:00 Poster C-46

Properties of Zinc Oxide Nanoparticles Synthesized by Electrical Discharge Technique in Liquids

Nikolai V. Tarasenko, Alena A. Nevar, Mikhail I. Nedelko, Dmitri A. Lopatik

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The capabilities of plasma assisted technique based on electrical discharge in liquids for synthesis of N-doped and In-N codoped ZnO nanocrystals and fabrication of p-type ZnO have been analyzed. The synthesis was carried out in a reactor containing the aqueous ammonium nitrate solution of 0.01M concentration. The pulsed spark discharges between Zn-Zn and Zn-In electrodes have been used. The diameter of the rod electrodes was 6 mm and the gap distance between the electrodes was several hundreds micrometers. The discharge was initiated by applying a high-frequency voltage of 3.5 kV with a repetition rate of 100 Hz. The peak current of the discharge was 60 A with a pulse duration of 30 μs.

The optical properties, morphology and composition of the nanoparticles formed were investigated by means of UV/VIS absorption spectroscopy, TEM and XRD. Optical absorption spectra of as-prepared colloidal solutions exhibited a broad absorption band edge in the UV spectral range. For a better crystallinity and lower electronic conduction, undoped ZnO showed the highest transmittance. There was a distinct red shift for the doped ZnO samples at the sharp fundamental absorption edge as compared with the undoped ZnO particles.

As it followed from the XRD patterns the synthesized product was composed of hexagonal ZnO nanocrystals. The position of the reflection peaks were observed to be varied with the composition of dopants. The influence of doping level on the electrical and optical properties of synthesized nanoparticles has been discussed.

16:00 Poster C-47

Electron Channelling Contrast Imaging of Defects in Nitride Semiconductor Thin Films

Carol Trager-Cowan[1], Naresh Kumar[1], Francis Sweeney[2], Paul R. Edwards[1], Angus J. Wilkinson[3], Austin P. Day[4], Tao Wang[6], Gordon England[5], Peter J. Parbrook[7], Ian M. Watson[8]

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Nitride semiconductor thin films are used in the manufacture of UV and blue laser diodes, UV and visible LEDs and white LEDs. Present applications of nitride LEDs extend from automotive and street lighting, to back lighting in mobile phones, to traffic lights. Future applications include replacing the light bulb in your home.

The performance of existing devices is limited by the presence of defects such as dislocations in the films. For example dislocations present in aluminium containing nitride thin films are thought to be responsible for the present low efficiencies of UV-emitting LEDs. Developing the capability to analyse dislocation densities rapidly with negligible sample preparation would represent a real step forward in the development of more efficient nitride semiconductor based devices.

In this presentation I will describe the use of electron channelling contrast imaging – in a field emission scanning electron microscope – to reveal and identify dislocations in nitride semiconductor thin films [1].

In electron channelling contrast imaging the intensity of electrons backscattered from a suitably oriented sample depends on the relative orientation of planes in a crystal. Changes in crystallographic orientation (tilt) or changes in lattice constant due to strainare revealed by changes in grey scale of an image constructed by monitoring the intensity of backscattered electrons as an electron beam is scanned over the sample. Extremely small orientation changes are detectable, enabling dislocations to be imaged due to lattice plane tilting and strain in the vicinity of the dislocations.

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16:00 Poster C-48

Epitaxial growth of semipolar $Al_xGa_{1-x}N$(1-103) films on ZnO substrates using room temperature buffer layer

Kohei Ueno[1], Atsushi Kobayashi[1], Jitsuo Ohta[1], Hiroshi Fujioka[1,2]

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Group III nitride light emitting devices fabricated on semipolar planes have attracted increasing attention due to the potential for significant reduction in the undesirable effects of the built-in field. As the substrates for growth of high quality semipolar $Al_xGa_{1-x}N$ films, ZnO is a promising candidate because ZnO has the same crystal structure and the small lattice mismatch with $Al_xGa_{1-x}N$. However, there still remains one serious problem with the use of the ZnO substrate, which is formation of the interfacial layers at the heterointerface that negate all the advantages of the nearly lattice-matched substrates. This problem stems from the high growth temperatures for $Al_xGa_{1-x}N$ by the conventional growth techniques. Therefore, it can be solved by development of a low temperature growth technique for $Al_xGa_{1-x}N$. Recently, we have found that the use of pulsed laser deposition (PLD) leads to dramatic reduction in the growth temperature of nitrides.

We have found that RHEED patterns during the growth of AlGaN on ZnO (1-103) at room temperature (RT) remain nearly unchanged from the initial stages, indicating the epitaxial growth of high quality $Al_xGa_{1-x}N$ (1-103) on this substrate. X-ray reflectivity measurements have revealed that the heterointerface between $Al_xGa_{1-x}N$ and ZnO is atomically abrupt. This result indicates that the reduction in growth temperature down to RT is inherently important for suppression of the interfacial reactions. Figure 1 shows XRD 2θ/ω curves of $Al_xGa_{1-x}N$ with various x values grown at 850°C on the RT AlGaN layers. One can clearly see 1-103 diffraction for all the samples, indicating that high quality semipolar AlGaN can be grown at all the Al concentrations with this technique. We also found the small tilt of the $[1\text{-}103]_{AlGaN}$ relative to that of ZnO from reciprocal space map near 1-103 diffraction shown in Fig. 2. This phenomenon can be attributed to the formation of the small tilt angle boundary at the heterointerface.

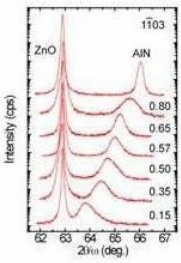

Fig. 1 XRD 2θ/ω scans for semipolar $Al_xGa_{1-x}N$ (1-103) with various x values.

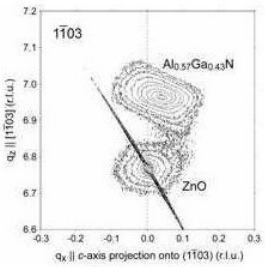

Fig. 2 A typical reciprocal space map near 1-103 diffraction for $Al_{0.57}Ga_{0.43}N$/ZnO (1-103).

16:00 Poster C-49

Influence of heavily doped n-Si(111) substrate on optical properties of the AlN thin films

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In this work the influence heavily doped n-Si(111) substrates on the growth and optical properties AlN thin films is investigated, because of AlN is perspective material for optoelectronic. AlN films with the thickness of 0.45-0.85 μm were grown by means of high frequency magnetron sputtering method. Deposition rate of films was 1.5-3 nm/min. For substrates used n-Si(111) (electron concentration $N_e = 2.5\cdot10^{19}$ cm^{-3}). The reflection spectra of n-Si(111) substrates and AlN/n-Si(111) thin films in range 200-750 nm and in range 1.4 - 25 μm were measured and also the elipsometric investigations were performed for the information about optical characteristics of thin films. The value of refractive index in range 505-675 nm (nearby maximums) of the AlN/n-Si(111) films is equal 2.13. Results of the elipsometric investigations are shown a value 2.11 of refractive index AlN films, which are typical for policrystals. In infrared reflection spectra AlN/n-Si(111) films there is a characteristic stripe of absorption conditioned by TO-vibrations of AL-N bonds. From data of the reflection spectra following physical parameters have been obtained: plasma frequency ω_p, electron mobility μ_e, phenomenological broadening parameter Γ, energy relaxation time τ_i, relaxation time of free carriers over the pulse τ_p .

16:00 Poster C-50

Thin films of ZnS and ZnSe by Atomic Layer Deposition for light sensor applications

Krzysztof Kopalko[1], Łukasz Wachnicki[1], Tetyana V. Semikina[2], Elżbieta Lusakowska[1], Wojciech Paszkowicz[1], Elżbieta Guziewicz[1], Marek Godlewski[1,3]

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Thin films of ZnS and ZnSe were grown by the Atomic Layer Deposition (ALD) method using three different procedures. In the ALD process we used either: a) a double exchange reaction (for ZnS, using $ZnCl_2$ as inorganic zinc precursor and H_2S as sulphur precursor); b) single exchange reaction (for ZnS, using atomic Zn and H_2S as sulphur precursor); or c) synthesis (for ZnS and ZnSe, using atomic zinc and sulphur or selenium). ZnS and ZnSe films obtained on this way were deposited on various substrates, including lattice matched ones (GaAs for ZnSe). For device applications ZnS and ZnSe were deposited on plated pyroceramic substrates covered with CdS or CdSe by the sublimation quasi-closed method. In this case ZnS and ZnSe layers were deposited to form heterostructures of II-VI semi-

conductors for light sensor applications, for highly efficient photodetectors, of the type proposed in the reference [1].

In this report we analyze structural, optical and electrical properties of ALD deposited films. In particular, we compare properties of ZnS films obtained by the three different ALD approaches, as mentioned above. We demonstrate that films grown at increased temperature and lattice matched substrates are crystalline.

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The research was partially supported by the European Union within European Regional Development Fund, through grant Innovative Economy (POIG.01.01.02-00-008/08).

16:00	Poster	C-51

Electrodeposition of n-type Zinc Oxide Thin Films on Flexible Plastics

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Thin films of transparent zinc oxide (ZnO) have been grown by potentiostatic cathodic deposition onto copper-coated PET (Polyethylene terephthalate) from a simple aqueous zinc nitrate electrolyte. The various optimum deposition parameters like potential, pH and bath temperature are investigated. Structural, morphological, compositional and optical characterizations of the films developed under different conditions were carried out to study such effects. The films were also tested for their potential use in photovoltaics.

16:00	Poster	C-52

Hybrid functional applied to the study of properties of narrow and wide band gap semiconductors

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One shortcoming of Density Functional Theory (DFT) calculations based on the Local Density Approximation (LDA) or Generalized Gradient Approximation (GGA) is its failure to reproduce well the band gap of semiconductors and insulators. This deficiency of DFT is attributed to a large part to discontinuities in the derivative of the exchange-correlation energy. A way to overcome this problem is the application of a certain fraction of exact exchange (Hartree-Fock exchange) within DFT via a so-called hybrid functional. The present paper will focus on DFT calculations based on the hybrid functional HSE06 [1]. This method gives better estimate than standard approaches to DFT for properties of narrow and wide band gap semiconductors like Ge and ZnO [2]. As the example the structure optimizations, calculations of electronic band gap, phonon dispersions and formation and migration energies of intrinsic point defects are performed and compared with LDA and/or GGA calculations and available experimental results.

[1] J. Heyd G. E. Scuseria and M. Ernzerhof J. Chem. Phys. 118 8207 (2003); and erratum ibid. 124 219906 (2006). [2] J. Wróbel, J. Piechota, phys. stat. sol. (b) 244, No. 5, 1538 (2007); 244, No. 12, 4688 (2007)

16:00	Poster	C-53

Towards single-source alkylzinc alkoxy and hydroxy precursors for ZnO nanoparticles.

Michał Dutkiewicz[1], Janusz Lewiński[1,2], Elżbieta Krajewska[1], Wojciech Bury[1], Iwona Justyniak[2]

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The nanochemistry of ZnO has been the focus of a number of research efforts for decades due to the unusual physical properties that are expected upon entering this size regime. Present big challenge for chemists is to develop tools for controlling nucleation, growth and morphology of nanoscaled ZnO particles. Various different physical and chemical synthetic approaches for preparation of ZnO nanoparticles were developed ranging from vapour phase synthesis, sol–gel chemistry, co-precipitation, solvothermal treatment to template syntheses. We are particularly interested in organometallic routes[1,2] which have recently been developed[3] and herein we communicate about the well-defined alkylzinc alkoxides $[^tBuZn(O^tBu)]$ and related hydroxides $[^tBuZn(O^tBu)]_x[^tBuZn(OH)]_y$ as novel efficient precursors for ZnO nanoparticles. We have discovered a new trimeric form of *tert*-butylzinc *tert*-butoxide and developed an effective method for separation of its various aggregates in the solid state. This achievement opens unique opportunities to study the effect of aggregation degree of the organometallic precursor on the character of ZnO particles prepared by thermolytic procedure under oxidative or inert atmosphere. In addition, the results of solution pyrolysis of the novel organometallic precursors under hydrolytic and rigorously anhydrous conditions will be presented. The resulting ZnO particles have been characterized by UV/VIS, PXRD and TEM measurements.

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[3] M.L. Kahn, A. Glaria, C. Pages, M. Monge, L.S. Macary, A. Maisonnat, B. Chaudret, *J. Mater. Chem.*, **2009**, *19*, 4044.

16:00	Poster	C-54

Structural characterizations of ZnO based one-dimensional nanostructures grown by MOCVD

Vincent Sallet[1], Said Agouram[2], Farid Falyouni[1], Corinne Sartel[1], Nadia Hanèche[1], Alain Lusson[1], Vicente Muńoz-Sanjosé[2], Pierre Galtier[1]

1. *CNRS-Université de Versailles St Quentin (GEMAC), 1 place A. Briand, Meudon 92195, France* **2.** *University of Valencia , Department of Applied Physics, c/ Dr. Moliner no 50, Valčncia 46100, Spain*

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ZnO one-dimensional (1D) nanostructures have been grown on sapphire substrates, and characterized by scanning electron microscopy and transmission electron microscopy (TEM). Samples were realized by metalorganic chemical vapor deposition (MOCVD) using diethyl-zinc, N_2O, ditertiarybutyl-selenide, and bismethylcyclopentadienyl-magnesium, as the zinc, oxygen, selenium and magnesium sources, respectively. Samples include : ZnO hexagonal rods and conical nanoneedles, ZnO/ZnSe core/shell 1D structures, ZnMgO nanowires, and "Vapor-Liquid-Solid (VLS) grown" ZnO nanowires using gold droplets as the catalyst.

Structural characterizations are presented for the different ZnO based nanostructures. Details of their microstructure are given by TEM analysis. In particular, we observed : i) excellent structural properties of the ZnO needles from their base up to the end of the tip, ii) core/shell structures exhibiting wurzite (ZnO) and zincblend (ZnSe) crystals with a well-defined interface (figure1), and iii) the presence of gold in the VLS grown ZnO wires, as revealed by X-ray energy dispersive spectroscopy.

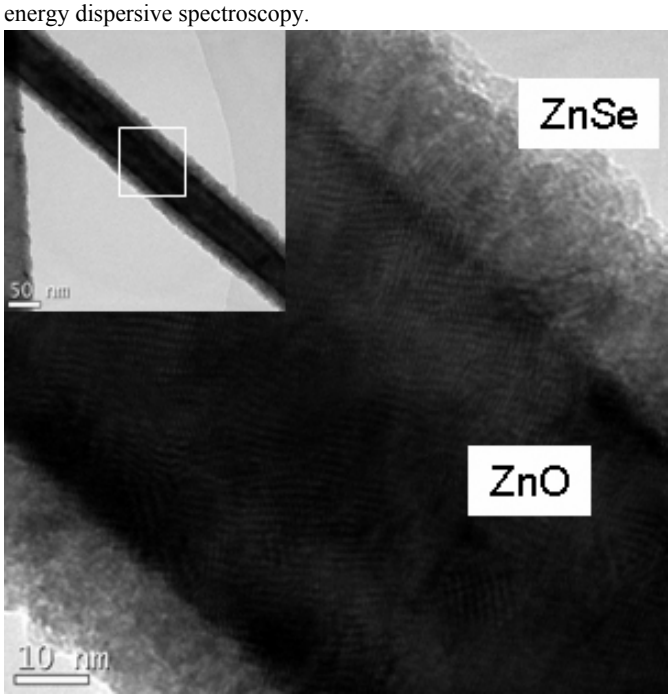

Figure 1 : TEM image of a ZnO/ZnSe core/shell nanowires

Symposium D

IR and THz electronics: from materials to devices

Welcome

The symposium will be focused on materials properties, and their functionality for IR and THz Electronics. IR and THz detectors, and also THz sources, as cooled and uncooled, are the parts which are the key-points that define the ultimate performance of IR and THz imaging and spectroscopic systems. Also, it is supposed to engage to the Meeting the specialists in modeling and simulation of these components. Further development physics based modeling and simulation is important for subsequent development of costly save IR and THz vision and spectroscopic systems. Development of such high performance systems, modeling and simulation of their parts will continue to be of interest to wide audience for several reasons: First, these parts are important systems components for standoff detection of objects with different chemical composition, security applications, etc., as they qualify the system parameters. Second, development in the fields mentioned, is supposed, will promote design of more advanced IR and THz systems. Development of IR and THz e.g. imaging and spectroscopic systems to provide the detectors and associated electronics capabilities is complex and costly. Advanced devices, including the low-dimensional structures, their design and manufacturing especially of uncooled or slightly cooled parts, must be used to reduce cost and extend application capabilities of IR and THz systems.

Objectives: The objectives of this Meeting are to review the current state of the art of materials for IR and THz detectors, and also THz sources. The aim of the Meeting is to provide a clear view on the current technology and the required advances to achieve more efficient systems. Also the aim is to establish the baseline of current uncertainty estimations in physics-based modeling and simulation and to identify key areas requiring further research and development.

Topics to be covered: Topics will address material properties, detectors physics, detectors design and technology based manufacturing, their modeling and simulations. The program will be completed with a discussion on the experience acquired over the past years in developing of the advanced detectors in Europe and USA.

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International Program Committee: S. Dvoretskii, Russia; I. Izhnin, Ukraine; S. Krishna, USA; H.C. Liu, Canada; A.G.U. Perera, USA; R. Rehm, Germany; V. Shul'ga, Ukraine; H. Zogg, Switzerland

Proceedings

Papers presented at the Symposium (after peer review) will be published in Optoelectronics Review.

Programme

Monday, 14 September

LUNCH

(maps available at the Reception desk)
Monday afternoon, 14 September, 12:30
building of the Faculty of Physics

Tuesday, 15 September

Lectures
Tuesday morning, 15 September, 9:00
Room 144

9:00	Keynote lecture

Superconducting hot-electron bolometer based on NbN nanostructures as THz mixer, direct detector and IR single-photon counter

Gregory N. Gol'tsman

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Recently, a new generation of hot-electron superconducting sensors:

terahertz mixers, direct detectors and single-photon counters have been developed.These sensors have already demonstrated a performance that makes them devices-of-choice for many terahertz and optical applications.

The superconducting hot electron bolometer(HEB) mixers based on ultrathin films of NbN combine the best sensitivity at the frequencies well above 1 THz and a gain bandwidth of about 5-6 GHz which make them suitable for most sensitive instruments being developed in the far IR region for astronomical and atmospheric studies.

Direct detectors made from NbN and MoRe films are operated in 0.3-3 THz range exhibit response time as low as 50 ps and 1 ns respectively with noise equivalent power (NEP) of 3x10□13 W Hz-1/2(NbN) and 5x10-14 W Hz-1/2(MoRe). Another versions of these detectors have a broadband sensitivity in 0.1-30 THz range with the same response time and exhibit higher NEP but much higher dynamic range. A promising type of the photon counting detector is superconducting single-photon detector (SSPD). The SSPD is patterned from 4-nm-thick NbN film as 120-nm-wide and meander-shaped strip that covers a square area of 10 x 10 μm2. At wavelength λ≤1.3 μm quantum efficiency (QE) of our best devices approaches 30% at 2 K with 35 ps timing jitter. The single-photon counting was observed at wavelength up to 5.6 μm with QE of ~1%. Simultaneously, at 2K the SSPD has negligibly low dark counts of 2x10□4 s□1. It provides NEP value of 10-20 W/Hz1/2 at λ≤1.3 μm and 10-18 W/Hz1/2 at 5 μm. Our recent investigations allowed us to develop an SSPD with photon-number resolving capability. Single-, two-, three- and four-photon absorptions were clearly observed. Finally, successful coupling of the SSPD with a single mode optical fiber makes SSPD a device of choice for wide range of applications including study of single-photon sources, telecom systems, quantum key distribution systems and quantum computing.

9:30	Invited oral

Comparison of Silicon Versus III-V Semiconductor Material Choice for Terahertz Imaging with Fast Field Effect Transistors Based Detectors

Wojciech Knap[1], Maciej Sakowicz[1], Hadley Videlier[1], Frederic Teppe[1], Dominique Coquillat[1], Jerzy Łusakowski[2], Krzysztof Karpierz[2]

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Resonant frequencies of the two-dimensional plasma in field effect transistors (FETs) increase with the reduction of the channel dimensions and can reach the THz range for micrometer and submicrometer channel lengths [1,2]. Non linear properties of the electron gas in the transistor channel can be used for the detection of THz radiation. The possibility of tunable narrow band detection in sub-THz and THz range, related to plasma resonances, has been demonstrated for short gate transistors at cryogenic temperatures. At room temperatures - the plasma oscillations are usually strongly damped, but FETs can still operate as an efficient broadband detector in the THz range. We present the main theoretical and experimental results on THz detection by FETs in the context of their possible application for THz imaging. We discuss in details the importance of the material issue – comparing III-V GaAs [3-5] and GaN HEMTs with Silicon MOSFET technology [6].

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[2] W. Knap, V. Kachorovskii, Y. Deng, S. Rumyantsev, J.-Q. Lu¨ , R. Gaska, M. S. Shur, G. Simin, X. Hu, M. Asif Khan, C. A. Saylor and L. C. Brunel, “Nonresonant detection of terahertz radiation in field effect transistors”, J. Appl. Phys. **91**, 9346 (2002)

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10:00	Invited oral

THz Quantum Devices

Hui Chun Liu

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Terahertz (THz) science and technology are widely investigated nowadays. To have a wide spread application, the key components, namely detectors and lasers, must be available, with the following properties: compact, convenient, and mass producible. Unfortunately today’s commercial THz detectors have various drawbacks such as low sensitivity, slow speed, and needing deep cryogenic cooling. THz quantum cascade lasers (QCL) have only been realized in 2002. Though impressive advances in THz QCLs have been achieved, the operating temperature is still in the cryogenic regime. Quantum structures based on semiconductors offer new possibilities. This talk starts with a general overview of various quantum structure THz detectors and then list some applications where THz lasers are needed. I will review the current status of THz QCLs. The main part of the

talk is on new directions in using either quantum dots and impurities for new THz lasers with potentially superior performance.

COFFEE

Tuesday morning, 15 September, 10:30
Main Hall

Lectures

Tuesday morning, 15 September, 11:00
Room 144

11:00 Invited oral

All electrical detection of the Stokes parameters of infrared/terahertz radiation

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Terahertz (THz) physics and technology are on the frontiers of physics holding great promise for progress in various fields of science such as solid state physics, astrophysics, plasma physics and others. Furthermore, THz physics holds a great potential for applications in medicine, environmental monitoring, high-speed communication, security, spectroscopy of different materials, including explosives, etc. Areas of THz physics of high current interest are the development and application of coherent semiconductor sources, molecular gas lasers, ultrafast time-domain spectroscopy based on femtosecond near-infrared radiation pulses, as well as the development of novel detectors of laser radiation. An important characteristic of THz laser radiation is its state of polarization. The detection of the polarization state, in particular the orientation of the electric field vector of linear polarized radiation and/or the ellipticity of transmitted, reflected or scattered, light represents a powerful technique for analyzing the optical anisotropy of various media such as solids, solid surfaces, plasmas, and biological tissues. The established way to gain information about the polarization state is the use of optical elements, which allow to determine optical path differences.

Here we review a new technique which allows one all-electric fast room temperature detection of the Stokes parameters of laser radiation, with a bandwidth from the infrared to the terahertz range [1-3]. The method is based on the simultaneous measurements of circular and linear photogalvanic effects in semiconductor quantum well (QWs) structures of suitably low symmetry and provides the full characterization of laser beams. The time constant of photogalvanic currents is determined by the momentum relaxation time of electrons, which is in the range of picoseconds at room temperature. This allows to measure the ellipticity of THz laser radiation with sub-nanosecond time resolution. The detection principle has been shown applying doped GaAs-, SiGe- and, most recently, HgTe-based QWs at room temperature. The detection of radiation polarization state is demonstrated for various low power and high power laser systems. A large spectral range has been covered applying cw and pulsed optically pumped molecular terahertz lasers, as well as low-pressure cw, pulsed, and Q-switched CO2 lasers and high-power pulsed transverse excited atmospheric pressure (TEA) CO2 lasers. In addition measurements were carried out with a free-electron-laser making use of its tunability and short pulses.

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11:30 Invited oral

Porous and oxide materials in the terahertz frequency range

Alexander Shkurinov

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Metamaterials can be used for obtaining the optical properties "on-demand". This study was focused on NOA based materials possessing very low density ~ 0.04 g/cm^3), high specific surface area (300 – 800 m^2/g) and fine fibrous structure. The particular interest of this work was terahertz properties of porous NOA samples after physical and chemical treatment. The chemical modification by using gaseous or liquid absorption at the NAO surface allows predictable changes in the optical properties. The preliminary results of the studies of the native, thermally treated and chemically modified NAO in the terahertz frequency range are presented. The first application of NAO based materials for photonic band gap (PBG) and a waveguide application was demonstrated. We also report an attempt to use NOA based composites with TiO_2 and ZrO_2 nanocrystals for making light driving terahertz devices. The dielectric properties of the NOA samples were studied using terahertz time-domain spectroscopy (THz-TDS).The essential part of the work is concerned with the detection of OH^- and H_2O molecules absorbed on the NAO surface and bonded inside phase structure. At room temperature the NAO structure consists of amorphous nanofibrils of aluminum oxihydroxide with composition $Al_2O_3 \cdot 3.6H_2O$. In the work the possibility of the detection and separation of the different state of OH^- and H_2O molecules are discussed. We show for the first time the THz detection of molecular monolayers at the NAO surface and also the properties of NAO composites with TiO_2 and ZrO_2 nanocrystals and we observed the real-time condensation and desorption of the atmospheric water on and out of the NAO surface.

12:00 Oral

Design of IF extra-low power consumption amplifiers at ≤ 1 K for terahertz matrix receivers, bolometer arrays and quantum devices

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An efficient method for drastic reducing the power consumption of the intermediate frequency (RF-/microwave) amplifiers for multi-channel devices (such as bolometer arrays, SIS matrixes) is theoretically grounded and experimentally demonstrated. The relevant technique is based on the HEMTs with excessive-high (initially, in saturated I-V region) cut-off-frequency and sacrificing their gain after changing to linear (unsaturated) I-V region, where transistor operates with very low drain voltages (10…100 mV) and current (30…100 mA). According to the approach the concept of low-noise amplifier on commercially available pHEMT (exactly, Agilent ATF36055) is proposed. One-stage amplifiers with associated gain of 10…20 dB were designed with emphasis on minimizing the power consumption and tested at ambient temperatures from room level to 300 mK. It has been founded that the power consumption of a few microwatts may be achieved at room temperature at frequencies up to L-band. The power consumption minimum level of 950 nanowatt per 10-dB-gain-version of the amplifier (frequency band 490…510 MHz) has been obtained at 300 mK ambient temperature. That is at least the order of magnitude better then was the case to date. The obtained both theoretical and experimental results permit predicting the feasibility to brought to the "nanowatt supply level" for RF/microwave amplifiers on InP HEMTs (with cut-off-frequency of hundreds gigahertz) and forward-looking InSb HEMTs.

Via comparison with state of-the-art amplifiers, it is confirmed that proposed technique has a high potential to design amplifiers especially for multi-channel ultra-deep-cooled (in future – down to 1 mK) cryoelectronic devices when the DC power consumption budget is critical requirement for the system.

12:15 Oral

GaBiAs layers for terahertz optoelectronic devices activated by 1 µm wavelength laser pulses

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Majority of pulsed THz systems are presently using Ti:sapphire lasers emitting at the wavelengths around 800 nm and photoconductive components made from low-temperature-grown GaAs (LTG GaAs) epitaxial layers. Ti: sapphire lasers are quite bulky and complicated, thus using of compact, efficient, and cost-effective solid-state and fiber laser systems that are directly pumped by diode-laser bars and are generating femtosecond pulses at wavelengths of 1 µm and 1.5 µm would be advantageous for THz applications.

In this contribution we will present the research on technology and physical characteristics of a novel semiconductor compound – LTG GaBiAs. The energy bandgap of this ternary compound can be shifted to narrower values by adding Bi much faster than, e.g., the bandgap of InGaAs when Ga-atoms are exchanged by In-atoms. Moreover, electron trapping times in GaBiAs shorter than ~0.5 ps can be easily achieved during the low-temperature growth of this material.

Epitaxial $GaBi_xAs_{1-x}$ layers were grown on semi-insulating (100)-oriented GaAs substrates in a solid-state molecular-beam-epitaxy (MBE) system at substrate temperatures ranging from 240 to 350° C; the Bi-content in the layers growth at the lowest temperatures has reached x=0.11, which has corresponded to the energy bandgap of the alloy of 0.9 eV. The majority of the investigations were performed on the layers with x=0.08 that were sensitive to the radiation of femtosecond Yb:KGW laser (the wavelength of 1030 nm) used in the experiments of THz radiation emission and detection.

As-grown $GaBi_xAs_{1-x}$ layers were of a p-type conduction with dark resistivity exceeding 1000 Ωcm. Electron mobility in the layers as measured by using the optical pump – THz probe technique was larger than 2000 cm^2/Vs. The same technique was employed also for the determination of the electron lifetimes, which, depending on the growth conditions, ranged from several picoseconds to less than 1 ps. All these material parameters satisfy the requirements that are set when manufacturing photoconductive switches used for optoelectronic THz radiation emission and detection.

Both THz emitters and THz detectors were made from $GaBi_xAs_{1-x}$ layers. When activated by 70-fs duration Yb:KGW laser pulses the system has produced THz transients with a bandwidth reaching 5 THz and signal-to-noise ratio of 60 dB.

LUNCH

(maps available at the Reception desk)
Tuesday afternoon, 15 September, 12:30
building of the Faculty of Physics

Lectures

Tuesday afternoon, 15 September, 14:00
Room 144

14:00 Invited oral

Physics, Technology and Performance of Strained Layer Superlattice Infrared Detectors

Robert Rehm[1], Martin Walther[1], Johannes Schmitz[1], Frank Rutz[1], Joachim Fleissner[1], Ralf Scheibner[2], Johann Ziegler[2]

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Over the last years, superlattice (SL) related heterostructures based on the 6.1 Å materials family (GaSb, InAs and AlSb) have proven their great potential for high performance infrared (IR) detectors. The most interest is focused on short-period InAs/GaSb SLs, which offer a widely adjustable effective band gap (3-30 µm). A high effective mass and a low Auger recombination rate result in low dark currents in p-i-n InAs/GaSb SL photodiodes. A long minority carrier diffusion length enables IR-detectors with high quantum efficiency comparable to $Cd_xHg_{1-x}Te$.

At the Fraunhofer IAF a mature manufacturing process for mid-wavelength IR (MWIR, 3-5 µm) InAs/GaSb SL detector arrays has been established. The small volume production process is based on commercial 3”-GaSb substrates, a solid source multi-wafer molecular beam epitaxy system, a fully automated stepper lithography environment and inductively coupled plasma dry etching.

For the integration of SL detectors into closed-cycle cooled IR modules or cameras, Fraunhofer IAF cooperates with AIM Infrarot-Module GmbH. Monospectral as well as bispectral, dual-color SL IR-imagers with a noise equivalent temperature difference (NETD) down to around 10 mK have been realized.

Via a spatially and temporally coherent detection of two different wavelength ranges in the MWIR, the dual-color superlattice imagers enable fast and highly sensitive remote detection of carbon dioxide. Applications range from missile warning for airborne platforms to the study of gas exchange between the ocean and the atmosphere and its influence on climate change.

14:30 Invited oral

Infrared Detectors with Quantum Dots and Type II InAs/GaSb Strained Layer Superlattices

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There is an increasing need for mid infrared (3-25□) detectors and focal plane arrays for applications ranging from night vision surveillance to non-invasive medical diagnostics. This presentation will be focused on the fabrication of focal plane arrays using two material systems, which are emerging as promising technologies for mid infrared sensors. These are (i) InAs/InGaAs self assembled quantum dots in well (DWELL) Detectors and InAs/(In,Ga)Sb strain layer superlattices (SLS) Detectors

In the DWELL heterostructure, InAs quantum dots are placed in a thin InGaAs quantum well that is in turn placed in a GaAs matrix. Three-color DWELL detectors, operating at 78K, with spectral response in the MWIR (λ_{p1} ~ 4□), LWIR (λ_{p2} ~ 8□) and VLWIR (λ_{p3} ~ 23□) regime have been fabricated in our group. Recently, we have fabricated the first long wave infrared quantum dot focal plane array (320x256 pixels). Images from this FPA along with the details of our research will be discussed in the presentation. The second material system we are investigating is the type II InAs/InGaSb superlattice for MWIR/LWIR detection. We have recently fabricated a high performance InAs/GaSb SLS detector with a 5□ cut-off operating at room temperature and 320x 256 FPA based on a novel nBn design. This will be discussed in the presentation.

Acknowledgements: I wish to acknowledge my collaborators (Dr. Cardimona’s group at AFRL, Prof. Perera’s group at Georgia State University, Prof. Painter’s group at Caltech, Dr. Toni Taylor’s group at Los Alamos National Laboratory and researchers from ARL) and co-workers (Prof. L.R. Dawson, Prof. A. Stintz, , Dr. S.J. Lee, Dr. E. Plis, Dr. Y.D. Sharma, H.S. Kim, J. Shao, D. Ramirez, A. Barve, N.W. Bernstein, S. Myers, A. Khoshakhlagh, J. Montoya, M. Kutty, E. Jang and R. Shenoi). Work supported by AFRL, ARL, ARO, MDA, KOSEF-GRL, DARPA, and NSF

BIO-SKETCH OF THE SPEAKER:

Sanjay Krishna is the Associate Director of the Center for High Technology Materials and an Associate Professor of Electrical and Computer Engineering at the Center for High Technology Materials at University of New Mexico. Sanjay received his Masters in Physics from the Indian Institute of Technology, Madras in 1996, MS in Electrical Engineering in 1999 and PhD in Applied Physics in 2001 from the University of Michigan, Ann Arbor. He joined the University of New Mexico as a tenure track faculty member in 2001. His present research interests include growth, fabrication and characterization of self-assembled quantum dots and type_II InAs/InGaSb based strain layer superlattices for mid infrared detectors. Dr. Krishna received the Gold Medal from IIT, Madras in 1996. He received the best student paper award at the 16th NAMBE Conference in Banff in 1999, the 2002 Ralph E Powe Junior Faculty Award from Oak Ridge Associated Universities, the 2003 IEEE Outstanding Engineering Award, 2004 Outstanding Researcher Award from the ECE Department, the 2005 School of Engineering Junior Faculty Teaching Excellence Award, 2007 NCMR-DIA Chief Scientist Award for Excellence, the 2007 NAMBE Young Investigator Award and the 2008 SPIE and IEEE-NTC Early Career Achievement Awards. Dr. Krishna has authored/co-authored more than 60 peer-reviewed journal articles, over 40 conference presentations, two book chapters and has two issued and six pending paten

14:45 Oral

Photoconductive Properties of GaAs-AlGaAs Broad Band Quantum Well Infrared Photodetectoers

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In this work includes the design of the material for infrared photodetectors based on GaAs/AlGaAs multiple quantum wells and the investigation of the electrical and optical properties of the main materials for QWIP (Quantum Well Infared Photodetector) which works in 8-12 micrometer atmospheric window. In this scope, 5 (five) different wafers (from Rev-3 to Rev-7) had been grown and both electrical and optical studies have been performed. Under high electric field, Franz-Keldish oscillation were observed in photo conduction experiments; it was seen that the observed and calculated electron-hole transition energies are in good aggrement for voltage values up to 12V. Calculated activation energy values are are in the range of 80 meV to 120 meV (at +- 1.5V); therefore, the dark current values

obtained for our measurements are in aggrement with the values for QWIPs in the literature. Finally, in the photo response experiments it was shown that the infrared detection above 7.2 micrometer is possible with these structures. On the other hand, TEM spectroscopy technique had been used to understand sample quality of grown materials by obtaining cross section images.

15:00 Oral

Two materials studies on residual background carriers and density dependent carrier mobilities in InAs/GaSb superlattices

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Material properties of InAs/GaSb type-II superlattices (SLs) are sensitive indicators of SL growth quality and of the eventual performance of devices made from these materials. In InAs/GaSb SLs, electrons move predominantly in the InAs layer, where they are affected by intrinsic and extrinsic scattering mechanisms, including interface roughness scattering (IRS).. The mid-infrared 7 ML InAs/8 ML GaSb superlattices (SLs) were grown by molecular beam epitaxy at growth temperatures between 370 and 430 C in order to study the intrinsic characteristic of background carriers. Grown SLs were all residual p-type with carrier densities in the low 10^{11} cm^{-2}, and a minimum density of $1.8x10^{11}$ cm^{-2} was obtained from the SL grown at 400 C. With increasing growth temperature, the in-plane carrier mobility decreased from 8740 to 1400 cm^2/Vs due to increased interfacial roughness, while the
photoluminescence intensity increased six fold due to a decrease in the nonradiative defect densities. Next, we report on a study of the in-plane carrier mobility in InAs/GaSb superlattices as a function of carrier density. Instead of using a number of differently doped samples, we use the persistent photoconductivity-effect to vary the carrier density over a wide range from n- to p-type in single samples and perform Hall effect measurements. Hence, our data are not obscured by sample to sample non-uniformities. We demonstrate that low-temperature in-plane mobilities are limited by screened interface roughness scattering (IRS), although present models of two-dimensional carrier screening of IRS lead to limited agreement with our data.

COFFEE

Tuesday afternoon, 15 September, 15:30
Main Hall

Lectures

Tuesday afternoon, 15 September, 16:00
Room 144

16:00 Oral

High pulse power mid-infrared AlGaAs/GaAs quantum cascade lasers

Kamil Kosiel, Maciej Bugajski, Anna Szerling, Justyna Kubacka-Traczyk, Piotr Karbownik, Emilia Pruszyńska-Karbownik, Kamil Pierściński

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High pulse power, mid-infrared AlGaAs/GaAs-quantum cascade lasers (QCLs) operation with no cryogenic cooling is reported. The laser design followed 3QW anticrossed-diagonal scheme [1]. The structures used double-plasmon Al-free waveguide for planar optical confinement. The injector doping was ~ $2.2x10^{12}cm^{-2}$ per period.

The electronic band structure of QCL has been calculated by solving Schroedinger equation with position dependent effective mass by finite difference method. The laser structures were grown by solid source MBE. The (100) oriented GaAs n+ substrates were used. The substrate temperature T_s was kept at 580 ^{0}C. The value of V/III ratio was at least 35 for growth of both GaAs and $Al_{0.45}Ga_{0.55}As$ layers [3][4].

For obtaining information about thickness and a composition of component layers of both test structures and a complete QCL structures, the analysis of double-crystal rocking curves was carried out. The dynamical diffraction theory has been used for simulating the symmetric (004) reflections, leading to extraction of structural parameters of periodic structures. The better than 1% thickness accuracy has been routinely achieved for lasing structures.

The double trench lasers were fabricated using standard processing technology, i.e., wet etching and Si_3N_4 for electrical insulation. The low resistivity Ni/AuGe/Ni/Au ohmic contacts, alloyed in 430^{0}C, were used at the top of the devices. After the wafer was thinned down to about 100 mm, an alloyed AuGe/Ni/Au contact was deposited on the backside. For current injection, windows were opened through the insulator with width 25, 35, and 50 mm. The lasers were cleaved into bars of 1 and 2.5 mm length and soldered with Au/Sn eutectic, epilayer down on diamond heatspreader and copper submounts. The devices emitted at 9.4 mm and operated up to 240 K, in pulsed mode with powers ~ 1W (77 K) and few mW (240 K) with slope efficiency h»0.70 W/A per uncoated facet.

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Letters Poland, vol..1, 16 (2009)

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15th European Molecular beam Epitaxy Workshop, March 8-11, 2009, Zakopane, Poland

16:15 Oral

A comparative study of single layer and bi-layer InAs/ GaAs quantum dots (QDs) with higher InAs monolayer coverage

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InAs monolayer (ML) plays a critical role in controlling the density and size of dots in QDs heterostructures grown by S-K mode using MBE. The density of QDs increses very sharply up to an InAs deposition of 2.0 ML and gets saturated over a 3.0 ML thickness. It is kown that for single layer QDs (SQD) the wavelength of emission peak associated with QDs gets extended as the thickness of InAs layer increases from 2.0 ML to 3.5 ML, with a considerable reduction in the full-width-at-half-maximum (FWHM), indicating formation of larger dots with homogenius size.

In this paper, we have shown that by using bi-layer QDs (BQD), which are grown having two layers of InAs coverage with a spacer layer of GaAs inserted in between them, the emission wavelength can be extended further, with a more uniform distribution of dots as compared to SQD. We have comapered structural and optical properties of an SQD with BQD samples, with higher monolayer coverage, and proved the superioty of BQD sample over the SQD sample in terms of optical and structural quality.

From AFM images we have found that the dots in the BQD show greater homogeniety, with lower size variation, as compared to SQD, which shows a bimodal distribution. The dot density as estimated from AFM are $1.5x10^{10}/cm^2$ and $1.4x10^{10}/cm^2$ for the SQD and BQD samples respectively. PL experiment is done at 8K, under an excitation of 40mW. The ground state (GS) peak of SQD is found at 1157 nm with an FWHM of 34.5meV while for the BQD, the GS peak is at 1229 nm with an FWHM 16.7 meV. The greater homogeneity in BQD sample is indiacted by lower line width of emission peak. It is attributable to the uniform formation of dots in top layer under the strained field produced by the seed layer, being a separation of only 8.5 nm spacer thickness between two layers. The red shift of emission peak for BQD sample indicates the growth of larger dots in comparison to SQD sample.

DST, India and SPM,IITB is gratefully acknowledge for their support.

16:30 Oral

Stimulated and spontaneous far infra-red emission from uniaxially strained gapless Hg1-xCdxTe

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The intensive far IR irradiation in the range of 80-l00 mkm was observed in uniaxially strained gapless p-Hg1-xCdxTe in the strong electric field. The transformation of energy spectrum of gapless semiconductor (GS) occuring under uniaxial strain P is following: the energy gap is formed between the c- and v-bands at k=0. The gap width is proportional to P. The v-band has loop-like top in the plane, transverse to deformation direction. The resonance acceptor level in GS splits under strain also. The lower of two split levels moves in the gap, and then, at P=2.5 kbar, it becomes resonance in the v-band. The inverse occupation in strained Hg1-xCdxTe is created because the hot electrons distribution occurs in the c-band under impact ionization, while the holes are localized near the v-band top. The band-to-band radiation transition occurs only at phonon participation, therefore its probability is low. The probability of this transition increases dramatically (by orders of magnitude) when the acceptor level becomes resonance in the v-band. Therefore, the stimulated irradiation can take place. Experimentally this effect was observed in p-Hg1-xCdxTe (x=0.l0-0.14), at T=4.2 K in the pulse electrical fields E<60 V/cm and at P<3 kbar. At threshold values of strain and electric field (P=2.5-2.7 kbar, E=50-55 V/cm), the dramatic increase of irradiation (by 3 orders of magnitude) and the increase of current (by 4-6 times) occurs. The existence of thresholds in E and P values, the great intensity of signal, and the correlation between radiation and current signals, points to stimulated character of the emission.

16:45 Oral

Thermal analysis of GaAs/AlGaAs quantum – cascade lasers

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The quantum cascade lasers (QCLs) are the most advanced class of semiconductor sources operating in the midinfrared wavelengths (3.5 – 24 μm)[1] and also in the terahertz range (1.2– 4.9 Thz)[2][3]. QCLs are unipolar devices based on transitions between intersubband states created by quantum confinement in ultrathin alternating layers of semiconductors.

The first demonstration of emissions in GaAs/ AlGaAs QCL was reported in 1998 by C. Sirtori group[4]. From this time QCL based on this materials have undergone a rapid development over the last few years. However still, the highest operating temperature in continuous wave are 150 K. The main limiting factors are the large electrical power density required for operation, and the low thermal con-

ductivity characteristic of ternary alloys and complex multilayer heterostructure. Those factors contribute to high temperature gradients in the device. Additionally, temperature causes the leakage of the electrons into delocalized continuum states, lowering the population inversion. These effects are the main limiting factors of the high temperature operation of the devices.

In this paper we use spatially resolved thermoreflectance (SRTR) to measure temperature distribution over the facet of pulsed operated quantum cascade lasers[5] . The method is based on the measurement of the change in the refractive index caused by current-induced heating of working device. The technique has a spatial resolution of about ~1 µm and temperature resolution better than 1 K. It has been previously applied to study facet heating in edge emitting lasers[6],[7]. In conventional junction lasers facet temperature can be significantly higher than the temperature inside device because of nonradiative surface recombination. This process is eliminated in unipolar lasers, which allows one to use facet temperature as an estimate of true temperature inside device. The presented method gives an insight into distribution and relative importance of heat sources within the laser. It also allows for determination of thermal resistance of the laser and to evaluate the in-plane k_{II} and the cross-plane k□ thermal conductivities of the active region which enables validation of a two-dimensional model for the anisotropic heat diffusion in QCLs.

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Wednesday, 16 September

Lectures

Wednesday morning, 16 September, 9:00
Room 144

9:00	Keynote lecture

Electron transport in nanostructures: the key to high temperature superconductivity

Hans-Peter Roeser

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The electron transport in nanostructured Schottky barrier diodes - used as low noise THz mixers up to 5 THz – is ballistic. Best signal-to-noise performance is achieved when the phase coherent electron movement is characterized by an optimum barrier depletion thickness D_{depl} which has the thickness of twice the doping distance D_{depl} = 2x. Detailed investigations on different Schottky diodes show a linear relation between $(2x)^2$ and the mobility µ of the device: $(2x)^2 = h/2e \cdot \mu$. Since the mobility µ is proportional to $(\text{energy})^{-1}$ and because of many similarities compared to high temperature superconductors (HTSC) it has been investigated if the doping distance in HTSCs is connected with $(kT_c)^{-1}$. By analysing published data of very well studied HTSCs we found out that P-type as well as N-type HTSCs with single (n = 1) and multilayered superconducting CuO_2 layers and even recently discovered iron-arsen based HTSCs show a linear correlation between the doping distance x and the critical transition temperature T_c. It turns out that the correlation equation $(2x)^2 \cdot n^{-2/3} \cdot 2M_{eff} \cdot \pi kT_c = h^2$ can be derived theoretically from the density of states in a one-dimensional quantum wire. This suggests that the superconducting CuO_2 plane consists of superconducting quantum wires. The correlation equation has the prospect to calculate the transition temperature for HTSCs for the first time. The paper will describe the electron transport phenomena in nanostructured Schottky diodes in the THz range and the detailed investigation of the different HTSC families.

9:30	Oral

$La_{0.7}Sr0.3MnO_3$ thin films for uncooled bolometers

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We report our results about uncooled $La_{0.7}Sr_{0.3}MnO_3$ (LSMO) bolometers: measurements of the temperature coefficient of the resistance (TCR), low-frequency noise measurements in epitaxial LSMO thin films deposited on buffered Silicon (Si), and $SrTiO_3$ (STO) substrates, and micromachining techniques for suspended structure fabrication.

A normalized Hooge parameter of 9×10^{-31} m^3 was measured at 300 K in the case of a 10 µm-wide, 575 µm-long line patterned in the 200-nm-thick film grown on STO substrate. This value is among the lowest reported values for manganites and close to values measured in standard metals and semiconductors. The corresponding noise

equivalent temperature (NET) was constant in the 300–340 K range and equal to 6×10^{-7} K.Hz$^{-1/2}$ at 10 Hz and 0.15 mA for a 10 µm-wide, 575 µm long line patterned in a 200-nm-thick LSMO film [1]. This very low NET value is comparable to the best published results for manganites and was even found to be lower than the NET of other uncooled thermometers such as semiconductors (a-Si, a-Si:H, a-Ge, poly SiGe) and other oxide materials (semiconducting YBCO, VOx, other manganite compounds). The results show that despite a TCR of only 0.017 K^{-1} at 300 K, and thanks to a very low-noise level, LSMO thin films are real potential material for uncooled thermometry and bolometry. Optical responses of a LSMO/STO sample at a wavelength of 533 nm in the 300–400 K range has been investigated [2]. We measured an optical sensitivity at I=5 mA of 10.4 V W^{-1} and corresponding Noise Equivalent Power (NEP) values of 8.1×10^{-10} W.Hz$^{-1/2}$ and 3.3×10^{-10} W.Hz$^{-1/2}$ at 30 Hz and above 1 kHz, respectively. In this case, the thermal conductance G was about 10^{-3} W.K^{-1}.

Further improvements are expected thanks to the fabrication of suspended structures using standard silicon micromachining techniques. Preliminary results showing suspended LSMO bridges of various widths (4 to 10 µm) and lengths (50 to 200 µm) will be presented and their performances as mid-infrared bolometers will be discussed. The effective time response could be less than 1 ms and the electrical sensitivity as well as the thermal conductance could be improved by a factor 1000.

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9:45 Invited oral

Mid infrared resonant cavity detectors and lasers with epitaxial lead-chalcogenides

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Wavelength tunable emitters and detectors in the mid-IR wavelength region allow applications including thermal imaging and gas spectroscopy. One way to realize such tunable devices is by using a resonant cavity. By mechanically changing the cavity length with MEMS mirror techniques, the wavelengths may be tuned over a considerable range. - We report the first room temperature mid-IR VECSEL (Vertical External Cavity Surface Emitting Laser) with a wavelength above 3 um. The active region is just 850 nm PbSe, followed by a 2.5 pair Bragg mirror. Output power is >10 mW.

Resonant cavity enhanced detectors (RCED) are sensitive at the cavity resonance only. They may be applied for low resolution spectroscopy, and, when arrays of such detectors are realized, as multicolor IR-FPA or "IR-AFPA", adaptive focal plane arrays.

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10:15 Oral

Transparent electroactive films for optical applications

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Electrostrictive polyurethane elastomer films were prepared using the solvent casting technique with tetrahydrofuran (THF) as a solvent followed by annealing at 95°C in vacuum for 45 minutes. Sample films of 50mm diameter were rigidly attached to circular frames and tested under applied field in the range of 0-25 kV/mm and tested under various environmental conditions. To investigate the electroactive response and optical properties of these lens-type films, single and two-layer lenses were made of these films. Each film was stretched and coated on both sides by depositing conductive and transparent polypyrrole polymer. Various film thicknesses were tested for transmission and reflection. 83% light transmittance was achieved for each individual coated film. The deformation response in the single layer is 0.2mm when subjected to a DC voltage of 17.73kV. This translates into a lens focal length of 1.871 m and a power of +0.534 DS. However, the two layer lens offers deformation in the order of 0.59 mm, which translates into a 0.317 m focal length and a +3.152 DS power when subjected to the same voltage. Further suggestions for improvement are proposed.

COFFEE

Wednesday morning, 16 September, 10:30
Main Hall

Lectures

Wednesday morning, 16 September, 11:00
Room 144

11:00 Invited oral

Room temperature infrared photodetectors

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The common believe was that infrared photodetectors of the mid and long wavelength infrared radiation need to be cooled to achieve a high sensitivity. Cooling is a straightforward way to suppress thermal generation of charge carriers and reduce related noise. But, at the same time, cooling requirements make infrared systems bulky, heavy and inconvenient in use.

A number of concepts to improve performance of photodetectors operating at near room temperatures have been proposed. Recent considerations of the fundamental detector mechanisms suggest that near perfect detection can be achieved in the MWIR and LWIR range without the need for cryogenic cooling.

This paper, to a large degree, is based on the research, development and fabrication of uncooled HgCdTe detectors in Poland. The devices are based on 3D-variable band gap and doping level structures, that integrate optical, detection and electric functions in a monolithic chip. The architecture of the structures is optimized for the best compromise between requirements of a high quantum efficiency, efficient and fast collection of photogenerated charge carriers, minimized thermal generation, reduced parasitic impedances, wide linear range, wide acceptance angles and other device features.

Recent refinements in the devices design and technology have lead to sensitivities close to fundamental limits due to quantum noise of the background thermal radiation, extension of useful spectral range to >16 μm wavelength and picosecond range response times. The devices have found numerous applications in various optoelectronic systems.

11:30 Oral

History of HgTe-based photodetectors in Poland

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The purpose of this paper is to review the main achievements in the investigations of HgTe-based ternary alloys and point out the Polish contributions in development of the middle and long wavelength infrared photodetectors. Research and development efforts in Poland were concentrated mostly on uncooled market niche.

At the beginning, a modified isothermal vapor phase epitaxy has been used for research and commercial fabrication of photoconductive, photoelectromagnetic, and other HgCdTe devices. Bulk growth and liquid phase epitaxy were also used. Recently, the fabrication of infrared devices relies on low temperature epitaxial technique, namely metalorganic vapor phase deposition.

At present stage of development, the photoconductive and photoelectromagnetic (PEM) detectors are gradually replaced with photovoltaic devices which offer inherent advantages of no electric or magnetic bias, no heat load and no flicker noise. Potentially, photodiodes offer high performance and very fast response. However, conventional photovoltaic uncooled detectors suffer from low quantum efficiency and very low junction resistance. The problems have been solved with advanced band gap engineered architecture, multiple cell heterojunction devices connected in series, and monolithic integration of the detectors with microoptics.

In final part of the paper, the Polish achievements in technology and performance of HgMnTe and HgZnTe photodetectors are presented

11:45 Oral

Acceptor doping control in MOCVD grown HgCdTe layers.

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Basic aspects of acceptor doped HgCdTe layers grown by MOCVD are presented. HgCdTe (111) layers were grown on GaAs (100) substrates in 350°C horizontal reactor using IMP deposition method. TDMAAs and AsH_3 were alternatively used as a effective p-type doping precursors. Incorporation and activation rates of arsenic were studied. Over a wide range of $Hg_{1-x}Cd_xTe$ compositions ($0.17<x<0.4$) arsenic doping control from $5\cdot10^{15}cm^{-3}$ to $5\cdot10^{17}cm^{-3}$ was obtained without post growth anneal. SIMS profiles, Hall and minority carrier lifetime measurement results are presented for chemical and electrical carrier concentrations characterization. Presented device quality p-type layers show Auger-7 process as a dominant lifetime limiting mechanism.

12:00 Oral

Far-infrared reflectivity as a probe of point defects in Zn- and Cd-doped HgTe

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Intrinsic point defects often play a significant role in determining the properties of mercury-containing solid solutions such as HgCdTe or HgZnTe, which are fundamental materials for infrared (IR) devices. An IR spectrum may provide useful information concerning the presence of defects and their influence on phonon spectra, as already demonstrated in CdHgTe and ZnHgTe[1] alloys as well as in hydrogenated CdTe-monocrystals[2]. In this paper we report and discuss far-infrared (FIR) experimental spectra of both HgCdTe and HgZnTe ternary alloys where additional modes induced by Hg-vacancies have been clearly detected.

FIR reflectivity experiments were performed at the DAFNE-light laboratory at Frascati (Italy). We used a BRUKER Equinox 55 FT-IR interferometer modified to collect spectra in vacuum. We used as IR sources both the synchrotron radiation from the DAFNE storage ring, and a mercury lamp. Measurements were performed over a temperature range of 20-300 K and for frequencies ranging from 50 to 600 cm^{-1}.

We present a non-destructive method for quantitative determining the vacancy concentration in the lattice of Hg-based semiconductor alloys with tetrahedral structure. The method is based on the identification of additional vibrational modes (AVM) induced by lattice

deformations in the far infrared (FIR) reflectivity spectrum. Although the method is restricted by sensitivity limitations, recent FIR experimental data carried out on HgZnTe and HgCdTe samples containing Hg-vacancies confirmed the presence of AVMs induced by Hg-vacancies in as-grown crystals.

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(maps available at the Reception desk)
Wednesday afternoon, 16 September, 12:30
building of the Faculty of Physics

Lectures

Wednesday afternoon, 16 September, 14:00
Room 144

14:00 Oral

The study of HgCdTe MBE-grown structure with ion milling

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Ion milling (IM) is emerging as a unique tool to reveal electrically active defects and complexes at MCT. It is known that IM is capable of strongly affecting electrical properties of MCT, up to conductivity type conversion in *p*–type material. It appears, that strongly non-equilibrium processes which take place under IM, when material is oversaturated with interstitial mercury atoms Hg_I generated near the surface, lead to the appearance of specific defect complexes, which may not form under other conditions. These complexes may also comprise intrinsic point and extended defects that normally do not show their presence due to their electrical neutrality or compensation. By measuring the parameters of the crystal before and after the milling, and following the disintegration of defects with time after IM ('relaxation'), one can detect and identify these defects.

The applying IM for the study of defects in LWIR and MWIR MCT films grown by MBE on GaAs substrates with ZnTe/CdTe buffer layers is reported. The results obtained on the MBE films grown on GaAs are compared to those acquired on the MBE films grown on CdZnTe substrates, and on MCT bulk crystals and films grown by liquid (LPE) and isothermal vapor (ISOVPE) phase epitaxy.

Nominally un-doped MBE films with composition x=0.22 (LWIR) and x=0.30 (MWIR) have been studied, and those *in situ* doped with an acceptor (As with concentration 10^{15}–10^{16} cm^{-3}) or a donor (In with concentration $5\cdot10^{14}$–10^{17} cm^{-3}), both as-grown and after various kinds of post-growth annealing. The thickness of the MBE films was 7 to 11 μm, and they were protected by ~ 0.3 mm–thick (top) and ~ 1.0 mm–thick (bottom) graded–gap films with x increasing up to ~ 0.5 near the both interfaces. Straight after IM the electron concentration at T=77 K $n_{77}(i)$ in all the MBE films (~10^{17} cm^{-3} for LWIR and ~$5\cdot10^{16}$ cm^{-3} for MWIR films) greatly exceeded that in the bulk, LPE and ISOVPE samples.

The $n_{77}(i)$ in the MBE films depended only on x (i.e., growth conditions), and neither on substrate type, nor on the type and concentration of dopants, nor on the terms of post-growth annealing. Since the $n_{77}(i)$ in the MBE films was 1 to 2 orders higher than the concentration of residual/intentionally introduced dopants in the samples. It's high value could not be explained solely by the interaction of Hg_I with electrically active species, which is typical for IM. It was concluded that in our MBE films IM led to the formation of a donor center, which resulted from the interaction of Hg_I with a certain defect that had been electrically neutral before the milling. The analysis showed that the most probable candidate was an initially neutral impurity (e.g., oxygen), or a defect complex involving excessive tellurium atoms. The exact nature of the defect and its relation to the MBE growth conditions will be discussed in details.

Using IM, was established donor and acceptor background doping level in the MBE films and made estimates of their electrical compensation. The electron concentration as measured after the 'relaxation' of defects formed under the milling, $n_{77}(f)$, appears to be similar in all the HgCdTe samples, except for those strongly doped with In, ~(1□4)×10^{15} cm^{-3}. The other exception was MWIR MBE films, where the $n_{77}(f)$ comprised (4□7)×10^{15} cm^{-3}. This feature is related to the MBE growth conditions that allow for a variety of Te-related defects, which will be discussed in the paper. Important is that $n_{77}(f)$ in un-doped samples was much higher than the n_{77} in the as-grown samples (the latter comprised ~10^{13} cm^{-3} in the MWIR films and ~10^{14} cm^{-3} in the LWIR films). This testifies to the fact that some of the as-grown samples were heavily compensated for, and the reason of such a high compensation degree will be also discuss.

14:15 Oral

The determination of solubility limits of gallium impurity in PbTe films doped with Ga on Si substrates

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The determination of solubility limits of gallium impurity in PbTe films doped with Ga on Si substrates,

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The thin films and single crystals of PbTe doped with Ga are the perspective materials for detecting IR-radiation in middle wave range. The IR-sensitivity of these materials is similar to $Cd_{1-x}Hg_xTe$ solid solutions but process of fabrication is much less demanding.

The main purposes of this study are to discuss the experimental results, which have been received during the examination of the chemical quantitative composition, phase composition, and the real crystal structure of PbTe<Ga>/Si and PbTe<Ga>/SiO_2/Si heterostructures, and to evaluate the solubility of Ga atoms in lead telluride thin films at different temperatures.

In this work the method of fabrication of PbTe<Ga>/Si and PbTe<Ga>/SiO_2/Si heterostructures offers the direct HWE one stage synthesis, in which the doping and the layer condensation process proceeds simultaneously. On the base of SEM observations, XRD, and EPMA results the boundary of gallium's limited solubility region in lead telluride matrix has been drawn. From the point of view of Gibbs's compositional triangle of Pb – Ga - Te ternary system, one can make a conclusion that the analysis of the PbTe – GaTe pseudobinary cross section only, as it have been done earlier, does not explain, in general, the process of gallium's solubility in PbTe matrix. For the fundamental understanding the formation process of Ga solid solutions it is necessary to consider the PbTe – Ga_2Te_3 and PbTe – Ga_2Te_5 polythermic cross sections in this ternary system also.

14:30 Oral

Linear 288×4 long wavelength infrared mercury cadmium telluride detector

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The application of linear 288×4 LWIR MCT detectors with time delay and integration (TDI) capability in thermal imaging systems allows to realize standard 576×768 TV format, to increase detectivity and to decrease defect channels. We used MCT heteroepitaxial structures (HS) grown by MBE with wide gap graded layers at the interface and surface of absorber for fabrication 288×4 diode FPA. The formation of n-p junction was carried out by B^+ implantation into p-type MCT HS MBE in planar technology. The special MCT composition throughout the layer together with developed planar technology gives the possibility to eliminate surface leakage current that leads to diode parameters limited by diffusion current. The typical diode dark current is equal to I_{dark} = 2-5 nA with maximum differential resistance R_{dmax} = (5-7)×10^8 Ohm at cut-off wavelength lambdac = 10,0-10,5 mkm. CMOS silicon read-out integration circuit (ROIC) provides the following function as TDI regime of each tetra diodes channel, bidirectional scanning, linear charge-to-voltage transformation, deselecting of each 1152 diodes and operating by sequential and parallel interface. The ROIC output voltage is over 2.5 V for information charge 2 pC. The dynamic range is 80 dB with nonlinearity 1%. Linear 288×4 LWIR MCT detectors were fabricated by hybridization of FPA and ROIC through the indium bumps at controlled pressures. The detectors parameters (78K, 30^0, 300K) at maximum sensitivity are the following: the response Sv = (4,0-4,5)×10^8 ± 9% V/W; the detectivity D* = (1,5-1,9)×10^{11}± 16% cm×$Hz^{1/2}$×W^{-1}.

COFFEE
Wednesday afternoon, 16 September, 15:30
Main Hall

Poster Session
Wednesday afternoon, 16 September, 16:00
Main Hall

Posters

Wednesday, 16 September

Poster Session
Wednesday afternoon, 16 September, 16:00
Main Hall

16:00 Poster D-1

The influence of laser shock waves on anodic oxide-HgCdTe interface

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Anodic oxide is widely used to passivate HgCdTe-based photoconductive detectors. However structures on anodic oxide have not sufficient stability. This makes necessary developing a new method to improve thermal stability of such structures. The laser shock wave treatment seems a promising option. Neodymium-glass laser pulses with 30 ns width were used to induce a shock wave in the samples under room temperature. The experiment has shown that the induced positive charge value an the anodic oxide- HgCdTe interface approached to its constant value under influence of laser shock waves. This kind of treatment is found to decrease electron concentration in a converted n-type layer by one order of magnitude in annealed anodic oxide- HgCdTe structures. Additionally, the findings show that the annealing time needed to have this layer disappeared decreases as well. The most relevant mechanism of the interaction between laser shock waves and the anodic oxide- HgCdTe interface in believed to involve the interface rebuilding which implies that elemental mercury and related charge is pressed back from the $CdTeO_3$ layer being right on the interface, and complexes with oxygen vacancies are formed.

16:00 Poster D-2

Effect of low ion beam milling and anodic oxidation on electrical properties of HgCdTe subsurface layers

Nicolas N. Berchenko[1,2], Ihor I. Izhnin[3], V. A. Yudenkov[3]

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Two techniques are used to fabricate n-layers on differently doped HgCdTe: (1) the ion beam milling (IBM) or (2) anodic oxidation followed by annealing (AO/A). Comparative analysis was performed to get additional knowledge about how n-layers are formed. HgCdTe samples with the hole conductivity caused by either intrinsic points defects (metal vacancy) or impurities (As, Au, Cu and Ag) were studied. The IBM etching system with Ar+ ions was used to perform IBM under ion energy E = 500 eV. The samples were anodised in standard conditions and then they were annealed in air at 428 K. The Hall measurements were conducted at 77 K in the fields 0.01–1.5 T and analysed to make the initial and step-by-step characterisation. It was found that the IBM forms the n-layer in all HgCdTe. In contrast, the AO/A causes the formation of the n-layer in Hg-vacancy doped HgCdTe only. A common reason of the n-layer formation in HgCdTe under the IM and AO/A is the superfast diffusion of excess Hg interstitials and their interaction with Hg vacancies and extrinsic acceptors. But the post-anneal excess Hg concentration at the oxide-semiconductor interface is much lower than that after IBM. Though this concentration is sufficient to convert the Hg vacancy-doped HgCdTe, its value is too small to ensure the conversion of impurity doped materials. Thermodynamic calculations allow predicting the conditions necessary to form the n-layer and evaluating its stability.

16:00 Poster D-3

Metal-semiconductor-metal photodetector on silicon on insulating wafers based on nanoscale interdigitated electrodes E. Budianu, M. Purica, A. Dinescu, E. Manea

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Recently, an increased interest in metal-semiconductor-metal photodetector (MSM) is observed due to high performances regarding bandwidth and responsivity that can be achieved by the application in fabrication of advanced technologies such as electron beam lithography (EBL) which can offer the possibility to realize nanoscale devices and a new concept such as nanoscale grating.

The paper presents the fabrication of an MSM type photodetector with an active area of 100x100 μm^2 on silicon on insulating wafers (SOI) with an absorbing layer thickness of 4 µm and oxide box of 2-4 µm. The interdigitated electrodes with 100µm length and 80 nm finger widths and three different fingers spacing of 80 nm, 100 nm, and 200 nm have been experimented in order to analyze the enhancement of the response due to subwavelength grating. The oxide box limits the thickness of optically active region of the structure for a high speed response and nanoscale electrodes leads to an efficient coupling of the light in the absorption layer.

The fabrication steps consist in:

- configuration of contacting pads over an insulating layer of SiO_2 layer by conventional photolithography;

- direct electron beam writing in polymethylmethacrylate (PMMA) of nanoscale fingers;

- Ti/Au evaporation and lift-off process;

Intrinsec transit time and R-C time constant calculated taking into account the experimented finger spacing evidenced that MSM on SOI substrate can be achieved values less than 1ps for transit time and less than 2 pF for structure capacitance. A good Schottky contact was obtained for Au/n-Si with Ti as interlayer for a good adherence. The responsivity of the structure is improved compared with conventional MSM structure.

16:00 Poster D-4

Increasing the size of InAs/GaAs multilayer coupled quantum dots with low defect density by using a InAlGaAs quaternary capping layer

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The emission wavelength of multilayer InAs/GaAs QDs using GaAs capping is typically around 1.1 µm, which is short compared to the 1.3-1.55 µm used in communication devices. In this experiment we report a new method for tuning 1.33 µm emission by increasing the size of QDs while maintaining a low defect density and fast grow rate by adding InAlGaAs quaternary capping (same lattice constant with GaAs) beside GaAs layer. The MBE grown heterostructures consisted of 10 layers of 2.7 ML InAs QDs with various thickness of capping. sample A and B have GaAs capping of thickness of 65Å and 70Å, while the quaternary capping layer thickness is increased from 20Å to 30Å. Sample C has 30Å of InAlGaAs capping and 150Å of GaAs capping. The samples were characterized by AFM, TEM and Photoluminescence spectroscopy. The TEM micrographs of A and B reveal that the number of complete Qd stacks has decreased and defect density has increased in B. Threading dislocations which reach the surface have caused some large dots (dia ~ 100 nm) to form on the surface in both samples, as seen in the AFM image. The Pl spectra illustrate the ground state emission peak at 1300 nm in B is red shifted by about 65 nm as compared to A, an indication of large dot formation. The QDs are enlarged because of strain driven migration of Indium atoms into the dots from thicker quaternary InAlGaAs capping in B compared to sample A. The PL spectrum shows that the emission peaks for both B and C are formed at same wavelength, indicative of dots of equal size. The integrated PL intensity in C is much higher which is because of the absence of defects. sample C can better accommodate the increased strain arising

from larger dots because of a thicker GaAs capping layer. Thus to achieve the desired red shift, an InAlGaAs quaternary capping layer can be added to increase the effective size of QDs and thick GaAs layer maintaining defect free heterostructure. DST India and SPM facility IIT Bombay are being acknowledged.

16:00	Poster	D-5

Silicon planar source of modulated infrared radiation

Tadeusz Piotrowski[1], Maciej Węgrzecki[1], Henryk Polakowski[2], Vladimir K. Malyutenko[3], Andrzej Czerwinski[1]

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This paper presents results of the theoretical analysis and the experimental verification of influence of geometrical parameters of a planar silicon structure emitting the modulated infrared radiation in a wide and continuous spectral range 3-12 μm.The approach is based on the modulation of the thermal emission power of semiconductors in the spectral range of intraband electron transitions. The free-carrier absorption-emission processes are utilized in techniques of dynamic emissivity enhancement. The emitting area was constituted by the base of a *p-i-n* structure operating well above room temperatures. The signal modulation was realized by the contact injection of free carriers. The changes of current-carrier concentration in the base of *p-n* diode influence the absorption coefficient (α), therefore also the emissivity (ε), thus allowing for modulation of infrared radiation emitted from the heated structure. Optimization of the structure parameters was aimed at maximal difference between emissivity of this structure heated to about 450K, for cases with and without forward bias applied to the *p-n* junction. Nearly 1.0 mW output power was achieved at $T \sim 450$ K with ~0.8 % external power conversion efficiency. The difference of emissivity can be described by: $\Delta\varepsilon_R = (1-R_1)(R_2\eta_2+1)(1-\eta_2)/(1-R_1R_2\eta_2^2)-(1-R_1)(R_2\eta_1+1)(1-\eta_1)/(1-R_1R_2\eta_1^2)$, where $\eta = \exp(-\alpha_\lambda d)$, R - reflection coefficient, α_λ - absorption coefficient at wavelength λ. This equation was applied for calculation of thickness d that enables maximal $\Delta\varepsilon_R$ at specific values of R and α. It was shown that high $\Delta\varepsilon_R$ for wafers with typical thickness (0.05 cm) requires an application of mirrors with R>0.8. This planar device based on the transparency modulation technique enhanced by the reflecting mirror on the back facet is easy to produce using standard silicon technology. It can be applied for diode matrix used for testing of dynamic characteristics of thermal cameras and generation of infrared imagery.

16:00	Poster	D-6

Matrix 320×256 infrared long wavelength detector with inner short wavelength cut-off filter

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Usually the producers of LWIR detectors (8-14 mkm) used external short wavelength cut-off filter (lower 7 mkm) to decrease background illumination. We developed the design and growth procedure in MBE technology of MCT heteroepi-taxial structure (HS) with built-in inner short wavelength cut-off filter. The additional high level wider gap layer before absorber at MCT HS MBE was grown to decrease sequence resistance and realize short wavelength cut-off filter. This layer creates the conditions same voltage biases applied to each diode of FPA and needed for planned spectral response range. For elimination interference between cut-off filter layer and absorber additionally barrier layer was grown. The In-doping cut-off filter layer in range concentration 2×10^{14} cm^{-3} - 5×10^{17} cm^{-3} did not change the LWIR spectral response. The numerical calculations of spectral response of photodiode arrays for different thick-ness and composition of cut-off filter and barrier layers were carried out. The NETD formula for IR CMOS detectors with cut-off layer was deduced. Matrix 320×256 IR CMOS LWIR detectors with built-in inner In-doped cut-off filter layer were fabricated. This layer allows to decrease the background illumination and realize storage time up to 200 mks. The detectors parameters (78K, 300, 300K) at maximum sensitivity are the following: sensitivity Sv ~ $(1\div3)\times10^8$ V/W, threshold power P ~ $(1,5\div2,5)\times10^{-7}$ W/cm^2.

16:00	Poster	D-7

Bolometer detectors for registration of terahertz radiation

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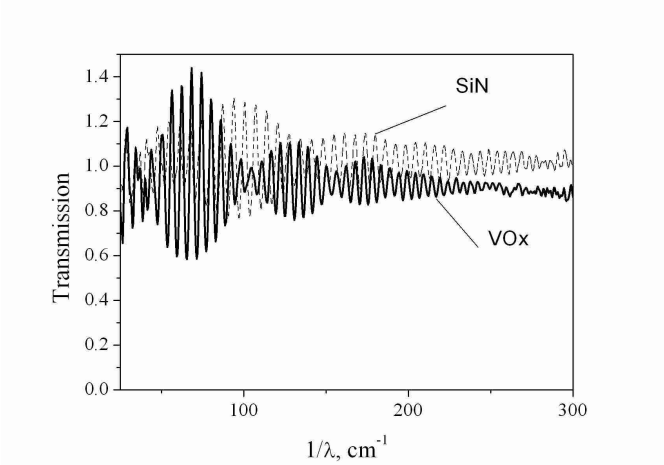
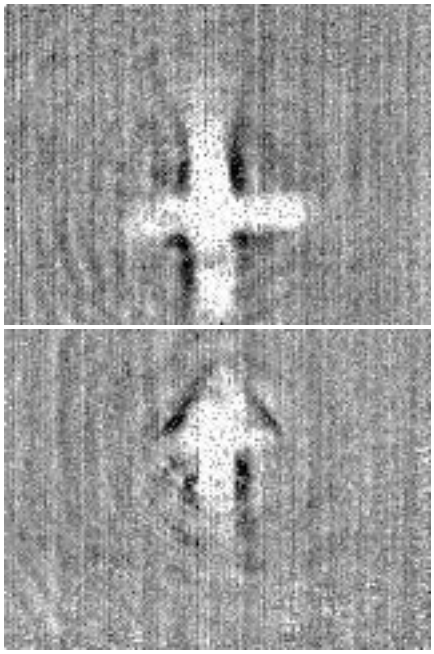
An uncooled microbolometer focal plane array (FPA) has been developed and used for imaging of objects illuminated by monochromatic coherent submillimeter radiation. The temperature of the thermosensitive element increases under the illuminated radiation and the change of the resistance were detected.

IR matrix microbolometer detector (120x160 elements) for a spectral range 7-14 micron, designed and manufactured in the Institute of Semiconductor Physics SB RAS, was used for registration submillimeter radiation. The transmission of the main elements of the microbolometer detector – silicon oxinitride (air bridge) and vanadium oxide (thermosensitive film) deposited on the sapphire wafer are shown in the figure below. The absorption in the vanadium oxide

film is about 10% which is enough for registration terahertz radiation.



Figures are shown the images of the model objects illuminated by the radiation from the laser on the ethanol vapor with wavelength of 115 micron (2.6 THz).



The sample of the image detected by the microbolometer array and produced by radiation of Novosibirsk free electron laser (Institute of Nuclear Physics SB RAS) at a wavelength 130 micron (2.3 THz) is shown on the figure below

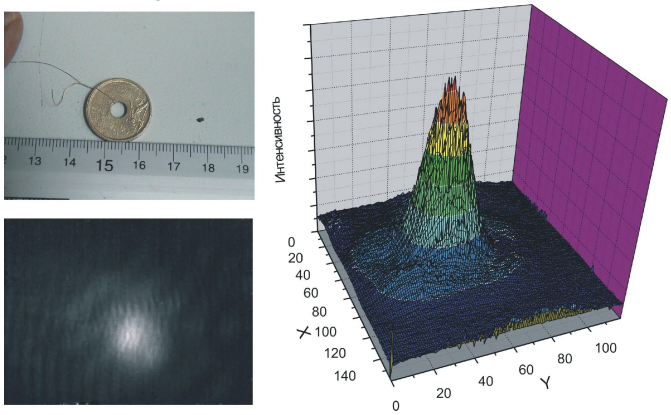


Image of a hole (4 mm) formed by a kinoform lens and detected by a matrix of microbolometer detectors.

The moving object can be detecte at the rate of 90 frame per second.

Noise equivalent power at wavelength 130 micron is NEP = $2x10^{-10}$ $W/Hz^{1/2.}$

16:00	Poster	D-8

Energy-band diagrams and capacity-voltage characteristic of $Cd_xHG_{1-x}Te$-based variband structures calculated with taking into account the dependence of electron affinity on the composition

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At present technology of growing of herteroepitaxial structures with variable composition based on CdxHg1-xTe solid solution by the molecular-beam epitaxy method is significantly developed. Now similar semiconductor structures are applied often in the photodetectors of middle and far infrared ranges of spectra.

The subject of this scientific work is the research of energy-band diagrams and capacity-voltage characteristics of CdxHg1-xTe-based variband heteroepitaxial structures with taking into account the dependence of electron affinity on the composition.

In this work numerical simulations of energy-band diagrams and capacity-voltage characteristics for metal-insulator-semiconductor structures based on CdxHg1-xTe-variable composition layers with wide quantum wells (~200 nm) inside the variband layer are carried out. Energy diagrams are calculated with taking into account the dependence of electron affinity on the composition x of CdxHg1-xTe [1, 2]:

$\chi(x)=5.56-1.23*+0.79*x2-0.83*x3$

This dependence was obtained in terms of dependence of local electroneutrality level on the composition for CdxHg1-xTe (see [1]).

It is shown in this work that the taking into account of the dependence of electron affinity on the composition for CdxHg1-xTe in some cases strongly influence on the view of energy structure and capacity-voltage characteristics of variband layers. So, the most influence is observed in depletion and inversion modes of operating of metal-insulator-semiconductor structure.

Also, comparison of calculated and experimental capacity-voltage characteristics is carried out.

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16:00 Poster D-9

Shallow acceptors in quantum wells of the $Si/Si_{1-X}Ge_X$ stressed heterostructures: application for detectors and sources of the Terahertz radiation

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The semiconductor heterostructures with shallow impurities in quantum wells (QW) are fruitfully applied lately for development of photodetectors and sources of the Terahertz spectral range. Their attractiveness is caused by the fact that an impurity state energy in heterostructures may vary with changing QW parameters (such as composition of components, QW width, spatial position of an impurity layer) in contrast to bulk doped semiconductors in which this energy is unchangeable. Such advantage may be used for variation of the THz laser emission spectrum and THz photodetector spectral sensitivity.

In the present work we have studied the dependence of the ground state energy, E_0, of the boron impurity in QW (stressed layers of $Si_{0.88}Ge_{0.12}$ in the $Si/Si_{0.88}Ge_{0.12}$ heterostructures) on the spatial position in the well and its width. The energy was determined from the temperature dependence of the Hall coefficient in the range of impurity deionization and from the long wavelength edge of the impurity photoconduction.

It has been found that the magnitude of E_0 in QW is less as compared to the bulk $Si_{0.88}Ge_{0.12}$ and it decreases with increasing a QW width and shifting the impurity d-layer from the QW center to its edge. For example, the magnitude of E_0 decreases by ~ 50 % with variation of the QW width from 150 to 200 A. For the QW width of 200 A and a shift of impurity from the QW center to its edge it varies by 1.5 to 2 times. It has been shown that variation of E_0 in QW as compared to the bulk material is caused by the change of the effective mass of free carriers, which connected basically with splitting of the light and heavy hole subbands due to internal stress and the effect of size quantization on the energy spectrum, rather than by spatial restriction of wave functions of the carriers localized on impurities by the barrier potential, which leads only to a stronger localization near an impurity center and, consequently, to an increase of E_0.

From measurements of conduction via the impurity band in the range from 30 down to 2 K under electric fields heating up charge carriers we have determined the magnitude of carrier localization radius *r* (the Bohr radius of an impurity state). It has been found that its value increases with shifting of impurity position to the QW edge. It has been shown that relationship between *r* and an impurity state energy in QW does not obey the formula $r \sim (E_0)^{-1/2}$ following from the Bohr model for the shallow impurities in bulk semiconductors.

16:00 Poster D-10

Ion milling-assisted study of defect structure of HgCdTe layers grown by liquid phase epitaxy on CdZnTe substrates

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HgCdTe, the material of choice for infrared (IR) detectors, has a low energy of defect formation due to a weak Hg–Te bond. While many defects are common for HgCdTe in general, some of them are specific to the growth method, conditions and substrates used.

Of the many techniques, which are used to characterize the defect structure of HgCdTe, ion milling (IM) is emerging as a unique means to study various defects and complexes. Interstitial mercury atoms, which are released during IM, interact with intrinsic defects and impurities, and by studying electrical properties of HgCdTe before and after IM, one can obtain a pattern of the defect structure of the material. In this paper, we report on the results of applying IM to the study of defects in HgCdTe grown by liquid phase epitaxy (LPE) on CdZnTe substrates. Studied were thick (up to 32 μm) layers for long– (x~0.2) and midwave (x~0.3) IR structures, grown in a closed system LPE machine from Te-rich melts. After the growth the layers were annealed in saturated Hg vapors at 230 ^{0}C for 48 hours or at 350^{0}C for 5 hours, ion-milled with 500 eV Ar ions with current density 0.2 mA/cm^2 for 20 min, and left for relaxation of the milling-induced defects at 300 K for 10^5 min.

The electron concentration in the LPE layers straight after the milling comprised $(3\square7)\times10^{16}$ cm$^{\square3}$. This value is typical for concentration of donor defect complexes formed by IM in high-quality HgCdTe, and is substantially less than that in HgCdTe grown by molecular beam epitaxy, where IM activates specific Te-related defects, yielding very high electron concentration. Upon relaxation, the electron concentration leveled at background doping that differed for layers grown on different CdZnTe substrates and comprised $(1–10)\times10^{14}$ cm$^{\square3}$, which, as a whole, spoke of the high purity of the layers. We discuss the properties of the layers and possible ways, in which the parameters of the CdZnTe substrates affect the electrical properties of the epitaxial material.

16:00	Poster	D-11

Calculation of radiation defect profiles in CMT subjected to high power laser beams

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At present time the development of infrared photo detectors with the use of the technique of high power radiation is an important branch of modern optoelectronics. Thus the major task of this research is to analyze the influence of intensive laser pulses on physicochemical properties of semiconductor crystals. As the object of study a semiconductor material, mercury cadmium telluride or MCT, is chosen. It can vary its spectral characteristics in wide range by changing molar percentage.

Improvement of a physico-mathematical model for radiation-induced defect formation in MCT under shot laser pulse influence was made. This model includes heat conduction equation, wave equation, the set of diffusion and complex formation equations for each type of radiation defects and neutrality equation. A function describing the generation of primary defects is built in such a way, that the process of defect formation occurs at the excess of melting temperature.

Heating patterns, distributions of temperature gradient and dynamic stresses which maxima are reached in near - surface area, are obtained.

Spatial profiles of the charge carrier concentration are calculated varying relations of interstitial and vacancy diffuse coefficients of mercury atoms, recombination coefficients of primary defects, and time constants of complex formation. Thus if the diffusion coefficient of interstitial atoms is sufficiently large their profiles will move into the sample and form complexes, increasing electron concentration. It is possible to observe inversion of conductivity type owing to the intensive generation of vacancy or interstitial atom complexes changing lifetime of primary radiation-induced defects. Variation of mutual recombination coefficient of vacancies and interstitial atoms has an effect on concentration profile of electrons in case of comparable diffusion rates of vacancies and interstitial atoms.
The comparison of calculation results with experimental data is made and their agreement is shown.

16:00	Poster	D-12

Changes in 8-12 mm CdHgTe photodiode arrays caused by fast neutron radiation

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It is important to ascertain the functionality $Cd_XHg_{1-X}Te$ arrays manufactured in MBE grown epitaxial layers under exposure to the radiation environment and try to disclose the physics of the process. The stability of CdHgTe photodiodes iv various radiation environments has been studied in [1,2]. In the present work the changes in $Cd_XHg_{1-X}Te$ photodiodes' characteristics after fast neutron radiation were studied. The photodiode arrays were formed on $Cd_{0,215}Hg_{0.785}Te$ film x=0.215 by implantation of B^+ with 150 keV ion energy and dose $3´10^{13}$ см$^{-2}$. The film was produced on the GaAs substrate by molecular-beam epitaxy. Current-voltage characteristics were measured before and after the fast neutron radiation treatment with the dose $5´10^{13}$ см$^{-2}$ at the room temperature. According [1] we should expect no changes in photodiodes, because defects that caused by neutrons should be annealed at the room temperature. Quite the opposite, we observed increasing of the dark currents in the reverse bias range U<0.2 V and decreasing one in the range U>0.6 V in most photodiodes. In the other photodiodes group dark current increasing has been observed in the whole range of bias voltage. For the analysis of the changes of photodiode's generation-recombination processes the model of the balance of the carriers on the traps in the band gap [3] was applied. The modelling shows that the dark currents increase at low bias voltage and decrease at high bias voltage only by acceptor concentration decreasing in the *p* – quasi neutral region and in the *p–n* junction. Decreasing of the acceptor –like Hg^{2-} vacancies concentration could be explained by the generation of vacancy-interstitial pairs of mercury under neutron radiation and diffusion of the high mobile mercury interstitials to the *p–n* junction with the recombination with the mercury vacancies in this region.

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16:00	Poster	D-13

Use of $Cd_xHg_{1-x}Te$ compound as a material for THz and sub-THz detector

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The characteristics of $Cd_xHg_{1-x}Te$ hot-electron microbolometer as a detector for THz and sub-THz radiation are studied and discussed. The device operation is grounded on the phenomena in semiconductor bipolar plasma and hot-carrier effect. In this device, the radiation heats only electron gas without the semiconductor lattice heating. Sensitive elements of microbolometer are the epitaxial layers with following parameters: n- and p-type $Cd_xHg_{1-x}Te$ with 50x50 μm and 10x50 μm sizes. Samples have been irradiated by radiation

with 37 GHz, 78 GHz, 95 GHz, 129 GHz, 0.89 THz and 1.58 THz frequencies. Temperature dependencies of photoresponse in 68÷300K temperature region were investigated. Investigations have shown that detector is sensitive to radiation in mentioned wavelength region. In the temperature range T=68÷300 K volt-watt sensitivity and calculated noise-equivalent power of n-type sample at 8 mm wavelength are as following: S =10 V/W at T=300 K and S =4·10^2 V/W at T=77 K, NEP ~10^{-10} W/Hz$^{1/2}$ at T=300 K and ~ 10^{-12} W/Hz$^{1/2}$ at T=77 K. The model of hot-electron detector is discussed.

16:00 Poster D-14

Nature of Ga deep centers in lead telluride based semiconductors

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Lead telluride based semiconductors doped with group-III impurities are promising materials for possible applications in VLIR and submillimeter regions. Still up to now the nature of its photosensitivity is not clear. Doped with Ga lead telluride, in which high photoresponse in MWIR region was revealed, was taken as a model object to explain the nature of group-III deep levels in IV-VI semiconductors and to elucidate the doping mechanism.

Vapor phase doping technique when prefabricated PbTe thin films interact with the various gallium-containing molecules was considered. Formation energies for defects created in PbTe as a result of interaction the Ga_2Te molecules, Ga_2 dimers and Ga atoms with a host crystal were calculated using density functional theory. Particularly Ga_{Pb} and Ga_i together with formation of accompanied Pb_i and Te_i self interstitials were examined. We propose the new type of defects – the impurity complex $(Ga_2)_{Pb}$ which looks like <111>-oriented gallium dumbbell. Calculations suggest that $(Ga_2)_{Pb}$ centers are preferably formed under Ga_2Te doping while $(Ga_2)_{Pb}$+Pbi ones are formed under Ga_2 or Ga doping.

The $(2Ga)_{Pb}$ center has negative value of formation energy and resulting defect concentration is determined by the reaction kinetics only. It is a double donor with DX-like properties, 'negative-U' behavior and metastable paramagnetic state corresponding to the positive defect charge. The neutral state has C_{3V} symmetry and the deep electronic level while positive state is characterized by the shallow level and D_{3d} symmetry. After ionization of the neutral defect under infrared illumination the large atomic relaxation (~0.6 Å) occurs. This sets conditions for the observed phenomenon of persistent photoconductivity.

The $(2Ga)_{Pb}$ complex may be regarded as a new type of DX-like centers in the ionic semiconductors. Performed EPR experiments confirm the model proposed as well.

16:00 Poster D-15

electric-
al properties of lead telluride single crystals doped with bromine

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Electrical properties of lead telluride single crystals doped with bromine, M.K. Sharov,A.M. Samoylov, O.B. Yatsenko, Voronezh State University, Universitetskaya Sq., 1, 394006 Voronezh, Russian Federation.

PbTe single crystals doped with VII A Periodic system group elements will continue to attract a widespread attention both for their fundamental electronic properties and for the applications in infrared (IR) optoelectronic. Under influence of the presence of bromine the electrical parameters of PbTe and its solid solutions can change significantly. However some fundamental problems of the formation, saturation and influence of quasi-local impurity levels in Br-doped PbTe have not been resolved yet.

The dependence of thermo-power factor and electrical conductivity upon the concentration of bromine atoms in the single crystals of $PbTe_{1-x}Br_x$ solid solutions has been studied in this work. The electron density relating to the content of bromine atoms in $PbTe_{1-x}Br_x$ samples has been estimated on the basis of experimental results. The values of thermo-power factor tend to reduction with increasing in concentration of bromine. On the contrary, the electrical conductivity and electron densities increase sharply after injecting the bromine atoms in PbTe matrix. It has been established that at x > 0.03 the all studied electrical parameters do not undergo the changes.

16:00 Poster D-16

THz/sub-THz bolometer based on electron heating in the semiconductor waveguide

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Direct detection THz/sub-THz bolometer is proposed, in which electromagnetic wave propagates in the bipolar semiconductor waveguide, heats electrons and holes there, and therefore creates their excess concentrations, as well as electromotive force. Both the carrier excesses at the supplied voltage switching on and electromotive force cause the bolometer response voltage. Semiconductor lattice heating is absent, and the minimal response time equals to the charge carrier lifetime. A bolometer theoretical model is developed. The possibility without cooling or moderate cooling to get the noise equivalent power (NEP) less than that one in the existing direct detection detectors and even close to NEP of cryogenic coherent systems is the proposed bolometer important peculiarity. Experiments on the $Cd_{0.2}Hg_{0.8}Te$ structures confirm the model main conclusions.

16:00 Poster D-17

Modeling of an IR variable-gap photodiode with the trapezoid-like profile of energy band diagram

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The paper presents results of modelling the photosensitivity of an IR photodiode based on HgCdTe of spatially variable composition. It is assumed that in the front p-base region energy gap linearly decreases in the direction to metallurgical edge of the p-n junction, whereas in the n-base energy gap firstly does not change with coordinate and then increases towards the rear contact.

For the photodiode with the proposed form of energy band diagram we have developed an analytical model of photocarrier transport that allows one to calculate the photosensitivity spectra depending on the band gap gradients in both base regions, thicknesses of base regions and photocarrier surface recombination velocity at the illuminated plane of the photodiode.

It is shown that due to internal quasi-electric field, which acts in the front base region there takes place a reduction of influence of photocarrier surface recombination upon the photosensitivity, as well as an increase of the collection coefficient of photocarriers. Presence of homogeneous region adjoining the p-n junction gives rise to increasing the absorption of photons what results in increasing the quantum efficiency of the photodiode. Besides, such a photodiode is characterized by widen range of the photosensitivity and reduced dark current.

16:00 Poster D-19

Electrical and optical properties of HgTe/CdTe superstructures

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Recently, much attention has been given to the potential of superlattices and heterostructures employment as the far infrared detectors [1]. In this work we investigated optical and electrical properties of HgTe/CdTe superstructures. Superstructures in the form of HgTe/CdTe thin films were obtained by the laser-beam successive evaporation of HgTe and CdTe targets using YAG:Nd laser (λ=1064 nm) with repetition rate of f=0.5 Hz. The films were evaporated at different temperatures and laser irradiation parameters onto Al_2O_3 and BaF_2 substrates with pre-deposited Ag contacts. The change of electrical resistance of the growing layers was measured by METEX multimeter connected with PC. Films resistivities were calculated and their time dependences during deposition process were determined for substrate temperatures of 30 and 200ºC. High resistivities were observed for the case of very thin films (lower than 40 nm). It was determined that the increasing of metal adhesion coefficient is the prevailing process during initial stage of films growth. Extrinsic carrier's concentrations were estimated from the results of optical measurements. Correlation between films thickness and their electric and optical properties was established.

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16:00 Poster D-20

Capacitance-voltage characteristics of MIS-structures in base on graded-band MBE $Hg_{1-x}Cd_xTe$ at passivation epitaxial grown in situ CdTe

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The important problem of creators of photodiode matrix in base on $Hg_{1-x}Cd_xTe$ is a passivation of semiconductor surface . The passivation coating must have low density of surface states in boundary, low densities of fixed and moved charges in insulators also passivation coating must have a stable and homogeneity of properties of boundary and insulator. For passivation surface of $Hg_{1-x}Cd_xTe$ proposed use of various insulators: native oxide and sulphide, SiO_2, ZnS, CdTe. In this work results of investigation capacitance-voltage characteristics MIS-structures in base on graded-band n(p)-$Hg_{1-x}Cd_xTe$ (x=0.22 and x=0.32-0.36) with grown in situ CdTe are submitted. For improvement stable and electrical strength of insulator coating for several samples on top CdTe formed additional protective layers SiO_2-Si_3N_4 for x=0.22 and ZnTe for x=0.32-0.36.

The methods of determination of main parameters of boundary and insulator from capacitance-voltage characteristics of MIS-structures in base on graded-band $Hg_{1-x}Cd_xTe$ are modified. It is shown that for correct calculation of ideal low-frequency capacity-voltsge characteristics necessary take account of presence of graded-band layers. For various samples the density of fixed and moved charges, density of surface states near middle of forbidden gap are calculated. For all investigated MIS-structures observed little moved charge (less than 10^{-5}-10^{-6} Cl/m^2). For structures with x=0.22 at used as passivation coating only CdTe the density of fixed charge vary from 4.4×10^{-5} Cl/m^2 to1.83×10^{-4} Cl/m^2. For structures with x=0.32-0.36 with single layer CdTe as passivation coating density of fixed charge total 1.9×10^{-4} Cl/m^2, formed of additional layer ZnTe is not lead to increase of value of density of fixed charge. For structures with x=0.32-0.36 formation of ZnTe lead to increase of density of surface states near middle of forbidden gap from 8.7×10^{10} eV^{-1}cm^{-2} to (4-5) $\times10^{11}$ eV^{-1}cm^{-2}.

16:00	Poster	D-21

The influence of near-surface graded-band layers on electrical properties of MIS-structures in base on MBE HgCdTe

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The development of molecular beam epitaxy methods give to possibility of optimization of parameters of infrared photodetectors in base on $Hg_{1-x}Cd_xTe$ used control of distribution of composition Cd along thickness of epitaxial film. In this wotk the influence of graded-band layers on value of differential resistance of space charge region in MIS-structures in base on MBE HgCdTe (x=0.225) are investigated. For structures in base on n-$Hg_{0.78}Cd_{0.22}Te$ product of differential resistance of space charge region by area amount about 15 Ohm×cm^2. Confirm that tunnel recombination via deep levels in near-surface layer of semiconductor severely influence on value of differential resistance of space charge region in MIS-structures in base on n-$Hg_{0.78}Cd_{0.22}Te$ without graded-band layers. It is shown that in MIS-structures creation of near-surface graded-band layers lead to suppress tunnel recombination via deep levels. The probability reason of limited of differential resistance in MIS-structures with graded-band layers is a background photogeneration and diffusion of minority carriers in sub-electrode region. The electro-physical and photo-electrical properties of MIS-structures in base on p-$Hg_{0.78}Cd_{0.22}Te$ with various distributions of composition in near-surface graded-band layers are investigated. It is shown that increase of content Cd at surface from 0.33 to 0.48-0.58 lead to suppress of interband generation-recombination and increase of differential resistance of space charge region. It is shown that small-siganl photo-emf give information about differential resistance of space charge region and dependencies of photo-emf graded-band MIS-structures in base on n-HgCdTe (x=0.225 and x=0.292) and p-HgCdTe (x=0.225) versus temperature and voltage bias are investigated, the mechanisms of limitation of value of differential resistance of space charge region at various conditions are analysed.

16:00	Poster	D-22

Dynamic heterodyned polarization imaging: studying polarization dynamics in materials at gigahertz frequencies

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We report the observation of surface acoustic waves (SAWs) launched from an interdigital transducer (IDT) at gigahertz frequencies, employing dynamic heterodyned electrostatic force microscopy (EFM). Dynamic heterodyned EFM is a relatively simple technique for the imaging of the polarization dynamics at the surface of the materials caused by complex interdigital transducer radiation patterns or any other excitation factor, with nanometer lateral resolution [1].

In this demonstration of the heterodyned EFM technique, the polarization at the substrate surface is induced by a passing surface acoustic wave. Amplitude modulation at the probe enables this method to be extended to frequencies well above the mechanical resonant frequency of the probe cantilever. This increases the bandwidth of the measurement and eliminates the need to an interference pattern in order to recover the high-frequency behavior of the SAW [2-4]. The electrostatic attraction between the probe and the piezoelectric material generated by the polarization dipoles in the surface layers of the material can be easily detected using techniques common in non-contact force microscopy. In earlier work using dynamic heterodyned EFM [1], the very small signals from the SAW were used to provide the dynamic equivalent of a grating to calibrate the technique.

In this paper, we study new samples (Fig. 1) which reflect the SAW across its path creating an interference pattern. We demonstrate sub-micron lateral resolution and high sensitivity by investigating the SAW generation form a set of metallic interdigitated electrodes fabricated on a weakly coupled substrate (quartz), visualizing the launched wave. In addition to the high resolution imaging of the mechanical waves, the SAW reflection pattern from a metal reflector on the surface has also been investigated (Fig. 2). A mathematical model for the SAW generation coupled with the force interactions between the probe and the substrate has been used to verify the experimental data.

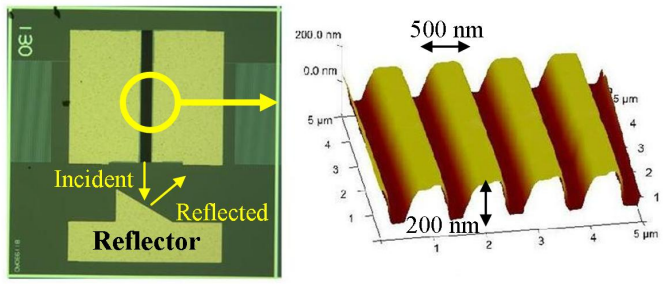

Fig. 1. Test device and reflector configuration (left); a topographic AFM image of the main IDT structure (right). The average height and width of each electrode is 200 nm and 500 nm respectively.

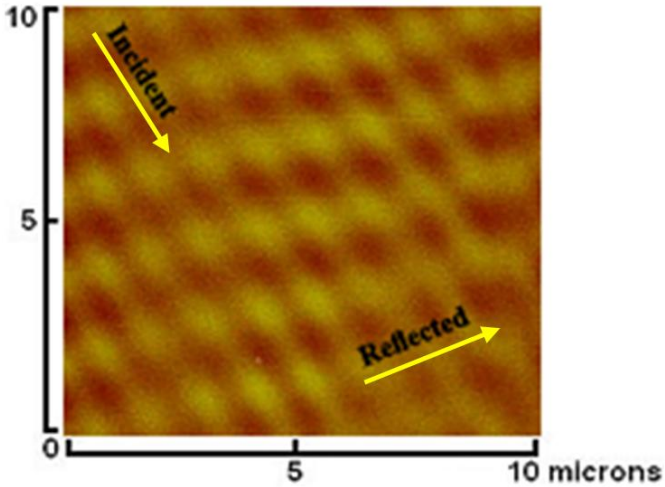

Fig. 2. 10 μm × 10 μm image of the raw response of the EFM probe to the polarization due to the SAW propagation on the substrate surface. This image was obtained in lift mode 300 nm above the substrate, near the reflector area. It clearly shows the main wave (up-down) and reflected wave (left-right).

References:

[1] D. R. Oliver et al., *Appl.Phys. Lett.***79**p. 3729, [2001]

[2] T. Hesjedal et al., *Appl.Phys.Lett.***78** p. 1948, [2001]

[3] G. Behme et al., *J. Appl. Phys.***89** p. 4850, [2001]

[4] T. Hesjedal et al., *Appl. Phys. Lett.***79** p. 1054, [2001]

Symposium E

New materials for spintronics

Welcome

Spintronics is a branch of electronics taking advantage of both charge and spin of the electrons. The field started in the 1980s with the discovery of giant magnetoresistance in metallic multilayers. It is actively developing and has been expanded to various materials including semiconductors, nanotubes and graphene, molecular and polymeric materials.

Spin dependent transport has been the object of intense research since the demonstration of magnetoresistive effects in metallic multilayers and magnetic tunnel junctions. The field has evolved to the extent that commercial applications for magnetic recording and electronic memory are now available. This was celebrated in 2007 with the Nobel prize to Albert Fert and Peter Grunberg. In the last few years the range of materials used for spin-devices has witnessed a dramatic broadening. Among others silicon, graphene and organic semiconductors have recently focused considerable efforts of the community. This symposium will be focused on the experimental and theoretical aspects of various spintronic phenomena with particular emphasis on material issues.

Selected topics:

- Fundamentals: new achievements and tendencies
- Semiconductor Spintronics
- Organic and Molecular Spintronics
- Multi-functional effects
- Spin dynamics
- Spin-torque
- Interface effects
- Devices and applications
- Industrial trends

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Programme

Monday, 14 September

Opening & Plenary Session

Czochralski Award Ceremony
Monday morning, 14 September, 10:30
Small Hall (237)

LUNCH

(maps available at the Reception desk)
Monday afternoon, 14 September, 12:30
building of the Faculty of Physics

Lectures
Monday afternoon, 14 September, 14:00
Room 213

14:00	Invited oral

Spintronics in graphene

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I will give an overview of electron spin injection, spin transport, spin precession and spin manipulation in graphene. The focus will be on recent experiments on single graphene field effect devices with ferromagnetic contacts. The use of the so-called non-local geometry allows a detailed investigation of various aspects of spin injection and spin transport.

I will first give a basic introduction into the “standard model” for spin transport and show how it can be applied to carbon systems, in particular graphene. The Bloch equations will be explained, which describe the processes of spin diffusion, drift, precession and relaxation. Following that will discuss that:

a) Spins can be transported through a graphene layer with a spin relaxation length of about 1.5 micrometer. By applying a perpendicular magnetic field Hanle spin precession can be studied and information about spin relaxation and the carrier diffusion can be obtained [1].

b) By applying a large DC electric field the transport of spins between injector and detector can be manipulated (sped up or slowed down) using carrier drift [2].

c) The spin relaxation is found to be slightly anisotropic, with spins directed perpendicular to the graphene plane relaxing faster than spins directed in the plane [3].

d) Spins can be injected into graphene with an injection efficiency up to 20 percent. This injection efficiency can be enhanced by a current bias which takes the carriers away from the injecting contacts. In this way injection efficiencies up to 38% have been achieved [4].

e) We have observed a scaling between the spin relaxation times and lengths and the carrier mobility in graphene [5,6]. I will discuss the possibility that in intrinsic graphene (where the carriers are only scattered by electron-phonon interaction) spin relaxation lenghts of 100 micrometer in graphene at room temperature might be possible, and even longer ones at lower temperatures. Related to that I will discuss the potential of graphene for future spintronics applications.

[1] N. Tombros et al., Nature 448, 571 (2007)

[2] N. Tombros et al., Phys. Rev. Lett. 101, 046601 (2008)

[3] C. Jozsa et al., Phys. Rev. Lett. 100, 236603 (2008)

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[5] M. Popinciuc et al., submitted to Phys. Rev. B.

[6] C. Jozsa et al, in preparation.

14:30 Oral

Impact of tunnel junctions for spin injection in graphene.

Bruno Dlubak[1], Pierre Seneor[1], Abdelmadjid Anane[1], Clement Barraud[1], Stéphane Fusil[1], Karim Bouzehouane[1], Cyril Deranlot[1], Bernard Servet[2], Stéphane Xavier[2], Richard Mattana[1], Frédéric Petroff[1], Albert Fert[1]

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Graphene, whereas in sole or few layers, has aroused a considerable interest for spintronics. This is mainly due to its high mobility and long spin diffusion length expected up to room temperature. In line with the early results of spintronics, conventional tunneling barriers of MgO or alumina have been used in order to inject spins into the graphene/graphite layer up to now.We studied the influence of both spin dependent barriers on the exfoliated graphene properties. We will first present the results of studies (Raman spectroscopy, TEM...) on the chemical compatibility of graphene with spin-dependant tunnel barrier (MgO, Al2O3). In the case of alumina, a 0.6nm Al film is deposited and then oxidized in pure O2. In the case of MgO, the sputtering is done directly from a MgO polycristaline target. This will be followed by a presentation of transport properties.

14:45 Invited oral

Giant tunnel magnetoresistance in organic magnetic tunnel junctions: A new insight into molecular spintronics

Clement Barraud[1], Pierre Seneor[1], Richard Mattana[1], Stéphane Fusil[1], Karim Bouzehouane[1], Cyrile Deranlot[1], Frédéric Petroff[1], Albert Fert[1], Patrizio Graziosi[2], Luis E. Hueso[2], Ilaria Bergenti[2], Valentin A. Dediu[2]

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Whereas spintronics brings the spin degree of freedom to electronic devices, molecular/organic electronics adds the opportunity to play with the chemical versatility. Here we show how, as a contender to commonly used inorganic materials, organic/molecular based spintronics devices can exhibit very large magnetoresistance and lead to tailored spin polarizations. We report on giant tunnel magnetoresistance of up to 300% in a (La,Sr)MnO_3/Alq_3/Co nanometer size magnetic tunnel junction. Moreover, we propose a spin dependent transport model giving a new understanding of spin injection into organic materials/molecules. Our findings bring a new insight on how to tune spin injection by molecular engineering and paves the way to chemical tailoring of the properties of spintronics devices.

15:15 Oral

Rubrene single-crystal FETs with spin-polarized electrodes

Wouter Naber[1], Jasper Lemmens[1], Anna Molinari[2], Anne Arkenbout[3], Chuan Du[4], Alberto Morpurgo[5], Wilfred G. Van der Wiel[1]

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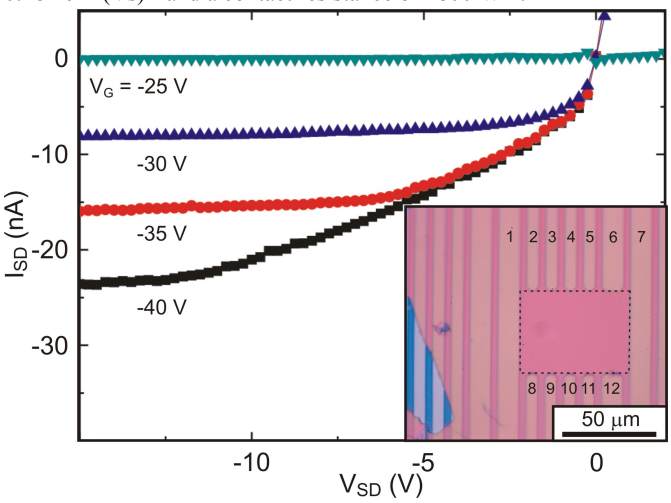
Organic materials are very interesting for spintronics, since the low spin-orbit coupling and small hyperfine interactions in these materials lead to potentially very long spin relaxation times [1]. The drawback of using organic materials for spintronic applications is the low mobility in this kind of materials. As a solution to this problem, we investigated rubrene single-crystal, with very large mobilities compared to other organic materials. For the first time, we have measured FET structures with ferromagnetic electrodes and a rubrene single-crystal spacer [2]. Our electrodes consist of an aluminum oxide tunnel barrier on top of cobalt. The tunnel barrier can be used to overcome the conductivity mismatch between the metal electrodes and the semiconducting organic spacer and prevents the oxidation of

cobalt. Another important feature of this device is that it explores the possibility of fabricating devices with both FET and spin-valve functionality. Such a device has not been realized yet.

We have fabricated several devices with Co/Al_2O_3 electrodes. With our electrode configuration, which is shown in the inset of the figure with electrodes numbered from 1 to 12, we are able to perform 2 and 4-point measurements in the same device. Using the 2-point measurement setup we observe clean FET behaviour in our devices, as can be seen in the figure. The current is contact dominated, as we expect from the tunnel barrier in our contacts. With the 4-point measurement setup we are able to study the length dependence of the resistance. From these measurements, we extract a mobility of 0.281 $cm^2(Vs)^{-1}$ and a contact resistance of 2800 Wm.



Small electrode separations will be used to perform magnetoresistance measurements to study the spin injection efficiency and spin relaxation in rubrene single-crystal. Our devices are suitable to perform non-local spin-dependent measurements [3].

[1] W.J.M. Naber, S. Faez and W.G. van der Wiel, *Organic Spintronics*, J. Phys. D: Appl. Phys. **40**, R205-R228 (2007).

[2] W.J.M. Naber *et al.*, to be submitted

[3] M. Johnson and R.H. Silsbee, Phys. Rev. Lett. **55**, 1790 (1985).

COFFEE
Monday afternoon, 14 September, 15:30
Main Hall

Joint Poster Session
Monday & Wednesday
Monday afternoon, 14 September, 16:00
Main Hall

Tuesday, 15 September

LECTURE
Tuesday morning, 15 September, 9:00
Room 213

9:00 Invited oral

Tailoring the spin properties of metal/organic interfaces by alkali metal doping

Mirko Cinchetti, Sabine Neuschwander, Jan-Peter Wüstenberg, Alexander Fischer, Martin Aeschlimann

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Doping of organic semiconductors (OSCs) has proven to be an efficient strategy to adjust their electronic properties for implementation in organic-based electronic devices. The recent success in the application of OSCs for spintronics applications raises the fundamental question if doping can be used as well to tailor the spin properties of OSCs, thus enhancing their performance in spintronics devices. In this contribution, we will first introduce a recently developed purpose-made technique based on spin resolved two-photon photoemission [1]. It allows obtaining direct and quantitative information about interfacial effects on spin-injection and on the spin transport properties of OSCs, as it has been demonstrated for the model system consisting of the interface between the ferromagnet cobalt and the OSC copper phthalocyanine (CuPc). In a further step, we have studied the influence of alkali metal doping on the spin properties of the cobalt/CuPc interface. We show that doping of CuPc with caesium or natrium atoms allows to achieve enhanced spin injection efficiency across the FM/OSC interface and to obtain quasi-elastic spin-flip lengths in the OSC of up to 50 nm at room temperature, a factor of five higher than the values obtained without doping [1]. These observations indicate the suitability of doping for an effective tuning of the spin properties of organic-based spintronics devices.

[1] M. Cinchetti et al., Nature Materials 8, 115-119 (2009)

9:30 Invited oral

Organic semiconductors for spintronic applications: key factors for the improvement of vertical hybrid devices

Ilaria Bergenti[1], Valentin A. Dediu[1], Alberto Riminucci[1], Patrizio Graziosi[1], Mirko Prezioso[1], Francesco Borgatti[1], Francesca Casoli2, Donald MacLaren[3], John Chapman[3]

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π-conjugated organic semiconductors have emerged as suitable candidates in spintronic devices mainly thanks to their low spin orbit interaction. One of the most reproduced effects in organic spintronics is the inverse magnetoresistive effect in vertical Spin Valves (SV) based on archetypal organic materials Alq_3 and containing half metallic manganite (LSMO) as bottom electrode and Cobalt as the top one. LSMO/Alq_3 and Alq_3/Co interfaces play a key role in the spin injection process; the occurrence of perturbing effects such as intermixing, chemical reactions, formation of dipole layers or molecular disruption affects both charge and spin injection and thus the

device performance.

Reproducible room temperature operation has been attained on Alq_3 based SV trough the insertion of thin insulating tunnel barriers (usually 1-2 nm of Al_2O_3 or LiF) at the Alq_3/Co interface.

We present a detailed investigation of LSMO and Co ferromagnetic films and their interfaces with Alq3 in presence of buffer insulating layer and we correlate the results with the operation of spin valve device.

In particular, structural information obtained from X-ray reflectivity measurements and High resolution TEM indicates that the presence of the tunnel barrier layer limits the Alq3/Co intermixing. The chemical states at the interfaces has been investigated by High Energy Photoemission Spectroscopy (HAXPES): the insertion of the tunnel barrier reduces the Co-Alq3 interaction, preventing very efficiently changes of the molecular electronic structure within the intermixed region.

10:00 Oral

Magnetic, structural and transport properties of perylene-based spin valves

Jean-Francois Bobo[1], Isabelle Séguy[2], Elena Bedel[2], Bénédicte Warot-Fonrose[1], Christina Villeneuve[2]

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Organic spintronic devices like spin-valves (SV) are attractive because of large spin relaxation time in π-conjugated molecules and simple device fabrication. Most of the studies of spin injection in organic semiconductors focused on two materials: T6 and Alq_3. We present here results on SV based on a perylene derivative: PTCTE. First the charge drift mobility in films of PTCTE deposited on silicon has been determined by time-of-flight technique in order to check the nature of majority carriers in this material. PTCTE has an electron mobility of $2.56.10^{-5}$ cm^2/Vs at $E=2.5.10^5$ V/cm which is about the same order of magnitude as the one of the widely used electron transport material Alq_3. Magnetic properties of NiFe and Co were investigated by MOKE and VSM. Our samples have SV behavior (two distinct coercive fields) with magnetically uncoupled electrodes down to 50 nm PTCTE, for thinner organic layers, we observe a loss of SV behavior due to coupling through pinholes or intermixing and degradation of the organic material. Nonlinear I(V) characteristics were observed at 10 K, as well as I(T) curves indicative of semiconducting behavior. The results were correlated with a morphological study by AFM of each layer and with tunneling atomic-force microscopy (TUNA) to probe the inhomogeneity of current flow through the organic spacer.

Acknowledgments

We would like to thank Dr. H. Bock (CRPP) for his support in PTCTE synthesis.

10:15 Oral

PPB as a transport material for spintronic devises

Alexey A. Lachinov, Natalia V. Vorobjeva, Victor M. Kornilov

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In this study the non-conjugated polyarylenephthalides class polymer are offered for applying it instead conventional conjugated polymers. Polymer is a poly(3,3'-phthalydiliden-4,4'biphenylen) (PPB). It is the non-conjugated polymer with wide band gap (~4eV). In other words it is insulator substance under normal conditions. But this polymer is able to change its conductive state from insulator to metal or vice versa. High conductive state can be reached in thin films. Thickness of the film should be less 1 μm. Sometimes it is necessary to apply any external fields. These fields are pressure, electric field, magnetic field, etc. High conductive polymer film has the following properties: specific resistance is 10-4 Ohm*cm; local current density is up to 106 A/cm2. Conductivity is the metallic type one. It is necessary to notice that film surface stays safe and isn't damaged. There are different factors provides this interesting features of PAP-films. But the main are following. The absence of the electron-hole symmetry in these polymers is an important particularity of their electron structure. Quantum-chemical calculations showed that the electron states forming the highest occupied molecular orbital (HOMO) are localized on the molecule backbone. As well as the electron states corresponding to the lowest unoccupied molecular orbital (LUMO) are localized on the side fragment of the molecule. In connection with this fact one can propose the electron-hole excitation absence. So this will further spin coherent transport through the polymer film. Correspond to last fact it is promising to design spintronic devise based on polyarylenephthalides film. Giant magnetoresistance phenomenon was observed in ferromagnetic/polymer/metal structure. Nickel substrate was used as ferromagnetic layer which can inject spinpolarized charge carriers into PPB transport layer. Relative resistance changing was up to 10 orders of magnitude. Magnetic field required for switching didn't exceed 200mT. GMR can be both positive and negative. It depends on the resistance of sample initial state. All measurements were realized at room temperature and in air atmosphere. Thus these advantages of GMR phenomenon in ferromagnetic/polymer structure are very useful for spintronic devices and its can be put into practice recently. This work is supported by fundamental investigation fund of PSD RAS "Physics of new materials and structures" and RFBR grant № 08-02-12042-ofi.

COFFEE

Tuesday morning, 15 September, 10:30
Main Hall

Lectures

Tuesday morning, 15 September, 11:00
Room 213

11:00 Invited oral

Quantum information processing with electron spin resonance

Arzhang Ardavan

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Electron spin systems were among the earliest proposed physical embodiments of quantum information processors. We have addressed a range of basic questions surrounding the practicalities of exploiting electron spins as qubits. We have shown that electron spin resonance can be used to perform quantum gates with a very high fidelity. We have studied the prospects for application of various candidate spin systems including N@C60 (a nitrogen atom encapsulated in a fullerene cage), molecular nanomagnets and phosphorus donors in silicon (P:Si). While in molecular nanomagnets the presence of magnetic nuclei in the vicinity of the electron qubit provides the dominant decoherence path, we have found that in N@C60 and P:Si nuclear moments can provide a valuable subsidiary resource in a quantum information processor.

11:30 Invited oral

Hybrid spintronic structures and half-metallic magnetic materials

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Magnetic/semiconductor hybrid structure is one of the most promising approaches for the development of the second generation spintronics controlling both spin and charge in a spin device. In this talk we will report the growth, interface magnetism and magneto-transport of several important magnetic/semiconductor hybrid spintronic structures, in particular, with half metallic magnetic oxides and Heusler alloys. The hybrid spintronic structures integrating half-metallic magnetic oxides and Heusler alloys are particularly exciting for the second generation spintronics as a 100% spin polarisation is expected for high efficient spin injection [1]. In the metal based system, while our previous XMCD study [2]. demonstrated that the Fe atoms on GaAs (100) are ferromagnetic, our further measurements of Co and Ni show that the Co atoms are similar to that of Fe with bulk-like spin moments down to submonolayer, but the Ni atoms lost their magnetic moments on the GaAs surface. We have synthesized for the first time single crystal half metallic Fe_3O_4 on GaAs [3], and further found a moderate Schottky barrier in this system ideal for spin-injection [4]. It is also encouraging that the ultrathin Fe_3O_4 films of 3-4nm have a large magnetic moments close to that of the bulk value [5]. Our recent pump probing measurements show that the half metallic Fe_3O_4 has a much larger damping constant than that of the metallic magnetic films. Very recently, we have shown the growth of half magnetic oxides on Si and with high quality epitaxial MgO [6]. We have also carried out detailed studies of the interface magnetic moments of various Heusler alloys on top of GaAs, where a large spin polarization is expected in the Heusler alloys. We have found that, however, the magnetic moments in the ultrathin Heusler alloy films are sharply reduced when the thicknesses were reduced down to 5-10nm. Using the elementary specific XMCD technique, we have found that the reduced spin moments of the Heusler alloy films on GaAs come from the Mn rather than the Co, but the orbital moments are not reduced. For device applications, we have fabricated a novel vertical magnetic/semiconductor spintronic device with a GaAs membrane, and a change of MR was observed, showing unambiguously the room temperature electric spin injection and detection in the hybrid spintronic structures.

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12:00 Oral

Time resolved magnetooptical Kerr effect in $(Pr,Ca)MnO_3$ thin films

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Time resolved Magnetooptical Kerr effect (TRMOKE) in insulating ferromagnetic phase in $(Pr_{0.6}Ca_{0.4})MnO_3$ thin films on $LaAlO_3$ and $SrTiO_3$ substrates was measured upon photoexcitation in magnetic fields up to 1.1T. The photoinduced Kerr rotation and ellipticity show remarkably different magnetic-field dependence. From comparison with the static Kerr rotation and ellipticity we conclude that two different magnetic phases are present in the samples at low temperatures. According to small angle neutron scattering results in bulk one of the phases originates from nanoscopic ferromagnetic metallic clusters. Comparison of temporal dependence of the photoinduced Kerr signals with the photoinduced reflectivity indicates that upon photoexcitation changes of the volume fractions of these phases take place on a timescale of ten picoseconds.

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12:15 Oral

Thermal diffusion of a thin Mn film into ZnO(000-1) probed by High Energy X-ray Photoelectron Spectroscopy

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A sounder understanding of the structural and electronic properties of ZnO-based diluted magnetic semiconductors (DMS) is required in order to achieve a global picture including all the physical processes involved in the origin of ferromagnetism in these materials[1].

The goal of this work is to gain insight into the electronic and structural properties of (Zn,Mn)O obtained by thermal diffusion of a thin (□1nm) Mn film grown on ZnO polar faces. The sample is characterazed in-situ by high-energy X-ray photoelectron spectroscopy (hv=3500eV, HEXPS) at BW2, Hasylab.

We followed the evolution of the Mn *2p* and the valence band photoelectron spectra of the Mn/ZnO(000-1) system respectively at the different stages of the experiment. During Mn deposition, most of the Mn atoms constituting the 0.25 nm thick film are oxidised by the surface oxygen atoms. The complete overlayer becomes metallic in the multilayer regime and, as a result, Mn related states are observed up to the Fermi level. Upon annealing Mn/ZnO to 660 K, Mn is fully oxidised to the *2+* state due to the formation of a thin MnO layer whose valence band edge appears at a binding energy around 2 eV below the Fermi level. Further annealing to 850K restore the valence electronic structure of ZnO. The simultaneous change in the shape of the Mn *2p* spectrum is attributed to a better dilution of Mn into the ZnO lattice and to the formation of (Zn,Mn)O. These interpretations are supported by the evolution of the intensity of the Mn *2p*, Zn *3s*, and O *1s* lines revealing that upon annealing to 850 K the substitution of Zn by Mn and diffusion of Mn into ZnO take place.

Reference:

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We acknowlodge

- C. Moisson (NOVASiC, Savoie Technolac, B.P. 267, Cedex 73375, Le Bourget-du-Lac, France), for the MCP polishing of the ZnO single crystals

- the support of CTB-BTC, CERUNA-University of Namur, the European Commission (contract RII3-CT 2004-506008 (IA-SFS)), and FSR-F.N.R.S.

LUNCH

(maps available at the Reception desk)
Tuesday afternoon, 15 September, 12:30
building of the Faculty of Physics

LECTURE

Tuesday afternoon, 15 September, 14:00
Room 213

14:00 Invited oral

The aggregation of magnetic cations in a semiconductor

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Recent theoretical and experimental studies indicate that semiconductor/ferromagnetic metal nanocomposite systems may exhibit novel and hitherto unexplored functionalities in spintronics, but also in photonics and thermoelectrics. We summarise our studies [1-5] demonstrating that it is possible to fabricate (Ga,Fe)N where the Fe ions are distributed in the nitride matrix in a controllable way giving rise either to a diluted random alloy or to chemical decomposition into (Ga,Fe)N regions more or less rich in the magnetic component or to ferromagnetic FeN_x nanocrystals buried in the GaN host. The aggregation of the Fe cations exhibits a strong dependence on the growth-rate and -temperature, allowing – when appropriately mastered – a control of the solubility limit. Moreover, the formation of the Fe-rich nanocrystals and, hence, the ferromagnetic response of (Ga,Fe)N can be affected by doping with donors and acceptors. Our findings confirm the hypothesis that the attractive force between magnetic cations can be adjusted by varying their charge state [6].

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14:30 Oral

Microstructure of silicon implanted with transition metals

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Silicon doped with manganese is a potential material for future spintronic device application. Due to limited solubility of Mn in silicon [1], various non-equilibrium techniques [2] have been used to introduce Mn into silicon beyond its solubility limit. Ion implantation is a technique for introduction of magnetic atoms into semiconductors that can, in principle, result in the high dopant densities and to control the dopant placement. Here we report our investigation on the microstructure of single crystalline silicon implanted with transition metal ions by transmission electron microscopy (TEM) and Secondary Ion Mass Spectrometry (SIMS).

The implantation of transition metals into silicon with subsequent annealing results in dopant re-distribution [3] strongly influenced not only by the implantation conditions and implantation-related-damage but alsoby microstructural changes, precipitation, surface segregation, and silicide formation. Our implantation has been carried out with Mn, Cr, and V ions with varying beam energy and substrate temperatures. The subsequent thermal anneals were done at temperatures from 300 °C to 1000 °C under flow of Ar gas. The results obtained by SIMS and TEM analysis will be discussed and correlated with magnetic characterization.

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14:45 Invited oral

Dilute-moment ferromagnetic semiconductor materials and devices

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In the lecture we will review basic electronic, magneto-transport, and magneto-optical properties of bulk (Ga,Mn)As and related ferromagnetic semiconductors, and devices based on these material systems. The discussion will be based on complementary theories based on the k · p kinetic-exchange model, microscopic tight-binding model, and ab initio calculations, and on extensive comparisons of the theory results to experiment. We will discuss incorporation of magnetic ions into the semiconductor host lattice, the nature of the insulator-to-metal transition and onset of ferromagnetism, and the Curie temperature trends. Transport effects covered in the lecture will include the Curie point singularity in the temperature derivative of the resistivity and extraordinary magne-toresistance coefficients, namely the anisotropic magnetoresistance magneto-optics phenomena. We will also demonstrate how the more direct relation between the spin-orbit coupled exchange-split band structure and transport realized in more complex tunneling or single-electron transistor devices leads to a large enhancement of the magnitude and tunability of the magneto-transport effects. Finally we will discuss the electric-field control of ferromagnetism in DMS transistor structures and implications of (Ga,Mn)As studies for spintronics operating at high temperatures.

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15:15 Oral

Ferromagnetic transition induced by re-crystallization of amorphous (Ge,Mn)Te layers

Tomasz Story, Wojciech Knoff, Tomasz Andrearczyk, Wiktor Domuchowski, Piotr Dziawa, Leszek Kowalczyk, Elżbieta Łusakowska, Krzysztof Świątek, Badri Taliashvili, Jerzy Wróbel

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(Ge,Mn)Te is a IV-VI diluted magnetic (semimagnetic) semiconductor exhibiting ferromagnetic transition induced by a very high conducting hole concentration. The Curie temperature in crystalline (Ge,Mn)Te layers can be increased up to Tc=190 K by independently optimizing the Mn content and the carrier concentration. Amorphous (Ge,Mn)Te layers exhibit very high resistivity and show paramagnetic properties. In GeTe and (Ge,Mn)Te layers a very fast switching between amorphous and crystalline phases can be induced locally by laser beam, electron beam or electric current flow. We examine magnetic and structural properties of amorphous, polycrystalline (re-crystallized), and monocrystalline (Ge,Mn)Te layers with thickness of about one micron and Mn content up to 20 at. % grown

on insulating BaF2 (111) substrates by molecular beam epitaxy technique. The local re-crystallization of amorphous layer was successfully achieved under the influence of light (YAG:Nd pulsed laser) and electron beam of a scanning electron microscope. Various micron-size wire-like patterns of a polycrystalline (Ge,Mn)Te embedded in an amorphous (Ge,Mn)Te matrix were prepared. Magnetic properties of (Ge,Mn)Te layers and wires were studied by superconducting magnetometry and ferromagnetic resonance (FMR) methods. The analysis of the temperature and magnetic field dependence of magnetization confirmed paramagnetic properties of amorphous layers and revealed a sharp ferromagnetic transition in polycrystalline (Ge,Mn)Te wires re-crystallized either by laser or by electron beam action. In re-crystallized (Ge,Mn)Te samples with Mn content of 10 at. %. the ferromagnetic Curie temperature is Tc=72 K. The analysis of the temperature and angular dependence of the FMR resonant field showed pronounced changes between the normal to the layer plane and various in-plane orientations of the magnetic field pointing out to the dominant role of magnetic shape anisotropy.

Work supported by the EC via ITN SemiSpinNet.

COFFEE

Tuesday afternoon, 15 September, 15:30
Main Hall

Lectures

Tuesday afternoon, 15 September, 16:00
Room 213

16:00 Oral

Computer simulation of nonequilibrium states of gallium arsenide nanolayer doped by atoms of manganese

Serge A. Beznosyuk[1], Mark S. Zhukovskiy[2], Yuliya V. Zemtsova[1], Larisa V. Fomina[3]

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It is well-known that alloying of Mn atoms into gallium arsenide is capable to provide transition of a material to a ferromagnetic state with relatively high Curie point on preservation of the basic semiconductor parameters as the result of its zoned structure change. That brings the given compound to a perspective spintronics material. But there is a problem. At achievement of concentration of the impurity over 5 % the lattice of the compound loses the stability. Some researchers supposed that the reason of this instability is nucleation of local crystal structure of manganese arsenide, which essentially differs from an initial lattice of gallium arsenide. The purpose of presented work is a simulation of this nanostructure instability of gallium arsenide nanolayer system doped by atoms of manganese. Using a crystal lattice data initial GaAs nanolayer structure of 20x20x2 elementary cells with total number of 6400 atoms was constructed. Alloying of gallium arsenide nanolayer was fulfilled by the replacement of gallium atoms with 100, 200, 248, 264, 288, 320, 400 atoms of manganese. It corresponds to small concentration of Mn impurity. After that variation procedure of energy minimization was used by nonlocal density functional method. The nanostructure optimization by algorithmic steepest descent method along the binding potential energy surface in a configuration space of nanosystem was performed. The basic results of the computer experiment were as following. 1. At alloying impurity manganese atoms are homogeneously distributed in a diamondlike lattice of gallium arsenide. 2 Increasing concentration of the impurity multiplies the probability of occurrence in an initial lattice of the semiconductor of local fluctuations when four atoms of manganese occupy the first coordination sphere of arsenide atom. In this place there is a local instability of nanostructure and as result lattice distortions the local fragment of a crystal lattice of manganese arsenide arises.

16:15 Oral

Ferromagnetic Resonance Study of (Ga,Mn)(As,P) thin films with variable P concentrations

Murat Cubukcu[1], Kh Khazen[1], Hans Jurgen Von Bardeleben[1], Jean Louis Cantin[1], Aristide Lemaître[2], Audrey Miard[2], Laurent Travers[2], Olivia Mauguin[2], Ludovic Largeau[2]

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The magnetic properties of ferromagnetic $Ga_{1-x}Mn_xAs$ thin films with Mn concentration between x=0.03 and 0.08 have been extensively studied in the recent years. These films are characterized by strong magnetocrystalline anisotropy due to strain induced by the lattice mismatch. Recently, it has been shown additional alloying with P allows reducing the epitaxial strain [1]; it is further expected that the Curie temperature will be increased above 180K for such alloys [2].

We have investigated a series of 50 nm thick $Ga_{1-0.06}Mn_{0.06}As_{1-y}P_y$ films prepared by MBE on (100) GaAs substrates by X-band and Q-band ferromagnetic resonance spectroscopy. The concentration of phosphorus was varied from y=0 to 10%. We observe a reorientation of the easy magnetization axis perpendicular to the plane for high concentrations of phosphorus. For intermediate P doping levels strain free layers can be obtained. The anisotropy constants have been studied as a function of temperature with special attention to the layers with low anisotropy. The influence of the phosphorus alloying on the magnetic relaxation via the Gilbert damping factor has equally been investigated.

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16:30 Oral

Magnetic Exchange Calculations for Porphyrin based Structures

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Current interest in transition metal-based molecular magnets like Fe-Porphyrin stems from the tantalizing prospect of future molecular-based magnetic storage devices. Porphyrins, which have been for long time at the center of a large research activity in biology, are now taking an important place in spintronics. In general, organic materials are interesting since the weak spin-orbit coupling and very long spin-relaxation times make them ideal media to propagate spin information. In addition to this, magnetic-Porphyrins carry intrinsic magnetic moment that can be exploited for constructing all-organic spin-devices. Finally and equally important Porphirins can be arranged in large molecular-superstructures with magnetic properties that can be largely tailored. In this contribution we investigate theoretically the magnetic ground state of various Fe-porphirins complexes by using ab-initio density functional theory as numerically implemented in the SIESTA code [1].

In particular we will present results for three different structures all derived from Fe-porphyrins, namely Fe-porphyrin dimers, Fe-porphyrin deposited on Ni substrates and Fe-porphyrin double decker with TCNE bridges. In all the three cases we perform a detailed analysis of the local density of state with the aim of identifying the mechanism for the magnetic coupling or for the absence of it. Finally we will discuss the difficulties in obtaining the desired exchange coupling in some structures and the possible solutions to overcome these hurdles.

References

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16:45 Oral

Low magnetization of thick substrates – the origin of giant magnetization of thin films on top of them?

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We present ferromagnetic properties of common substrate materials for oxide thin film growth, i.e. ZnO and yttria stabilized zirconia. Those were induced by a combination of high temperature vacumm processing and ion implantation simulating common preparation methods for the creation of diluted magnetic oxides. Moreover, we will focus on the coexistance of ferromagnetism induced by defect and metallic nanoclusters.

17:00 Oral

Magnetic Biomimetic Scaffolds for Bone Tissue Engineering

Valentin A. Dediu[1], Nathalie Bock[1], Alberto Riminucci[1], Chiara Dionigi[1], Alessandro Russo[2], Anna Tampieri[3], Elena Landi[3], Vitaly Goranov[4], Maurilio Marcacci[2]

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The use of scaffolds for tissue engineering is increasing due to their efficacy in helping the body rebuild damaged tissue. One of the current issues concerning this technique is how to improve the regrowth of damaged tissue by supplying growth factors; these have to be readily available to the live tissue that is growing in the scaffold. In the present work we address this problem by magnetizing the scaffolds. We show an example of our magnetizing technique on a bone graft substitute, which is used to replace damaged or diseased bone tissue. The susceptibility of the scaffold is sufficiently high that, if placed in a magnetic field, it acts as magnet. In our vision this will allow growth factors, which will be attached to magnetic nanoparticles, to be attracted by the scaffold and hence be available to the growing live tissue.

Wednesday, 16 September

LECTURE

Wednesday morning, 16 September, 9:00
Room 213

9:00 Invited oral

Single Molecule Magnets On Metallic Surfaces

Roberta Sessoli[1], Matteo Mannini[1], Francesco Pineider[1], Ludovica Margheriti[1], Dante Gatteschi[1], Andrea Cornia[2], Chiara Danieli[2], Philippe Sainctavit[3], Francesco Buatier de Mongeot[4], Daniele Chiappe[4]

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Magnetic molecules are among the last to have joined the list of molecular materials, but have already attracted great attention and are extremely promising candidates as nanoscale memory cells. In fact a peculiar class of molecules, ubiquitously known with the evocative term of Single Molecule Magnet (SMM), has an intrinsic magnetic

bistability that is not associated to cooperativity. The "memory effect" of SMM, which is reminiscent of the behavior of single domain magnetic particles, is also accompanied by fascinating quantum effects tanks to the intrinsic quantum nature of these small objects and also to the rigorous mono-dispersity in shape, dimension, and structure.

To fully exploit the advantage of the bottom-up approach typical of molecular sciences organization of SMM on surfaces, is a necessary condition to address the magnetic state of a single molecule. Rather unsurprisingly many studies have been devoted to the Mn_{12} family of magnetic molecules, $[Mn_{12}O_{12}(RCOO)_{16}(H_2O)_4]$, the archetype of SMM behaviour. However, the complex nature of SMM and the peculiar origin of their magnetic bistability have for half a decade represented an obstacle for the development of this research field.

Thanks to the surface sensitivity, element, and oxidation state selectivity of some synchrotron radiation techniques, i.e. X-ray absorption and X-ray Magnetic Circular Dichroism, it has been shown that the Mn_{12} clusters are strongly reduced during the process of grafting to a conducting surface. Fortunately another class of polynuclear SMM clusters, based on a propeller-like tetranuclear iron(III) core, Fe_4, demonstrated a more "robust" SMM behaviour, with the typical hysteresis persisting also when the molecules are chemically grafted to on a conducting substrate, like a Au(111) surface, through "alligator clips" (flexible and non-conjugated aliphatic chains terminated with a sulphur-containing group).

These recent findings, combined with an exceptional robustness of these SMM that can be evaporated in high vacuum, open the perspective of using mono or multilayer of SMM in spintronic applications thereby permitting to play with the elementary interactions between electron transport and magnetism degrees of freedom at the molecular scale.

9:30 Oral

Investigations of metallo-phthalocyanines grown on ferromagnetic Co((001) thin films.

Stefan Lach[1], Kathrin Müller[3], Kathrin Goedel[1], Felix Schmitt[1], Ehesan Ali[2], Peter M. Oppeneer[2], Thomas Jung[3], Martin Aeschlimann[1], Christiane Ziegler[1]

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Due to their chemical tunability and weak spin-orbit coupling organic semiconducting materials are promising candidates for future spintronic devices.

This motivates detailed investigations of the interface between metallo-phthalocyanines and ferromagnetic metallic substrates. Here, the morphology of ultra thin films of CuPc and FePc on a magnetized epitaxially grown Co(001) substrate were studied. Cobalt with its high degree of spin polarization at the Fermi level is a well-suited substrate for the investigation of potential spin selective and hence magnetic interaction with organic molecules as recently shown by the magnetic ordering of manganese(III)–tetraphenylporphyrinchloride on Co(001) [1]. The STM data reveal that both molecules are lying flat on the smooth Co films up to the range of just 1 monolayer. To discuss the differences in morphology for the multilayer range as shown especially for the domain structure of CuPc, theoretical calculations were performed. As it was shown recently for the interaction of porphyrin molecules with ferromagnetic substrates, the predictions of DFT+U calculations with a Coulomb U term are in good accordance with the experiments [2]. Using a structure of Fe/Cu-phthalocyanine adsorbed on a 3-layered cobalt surface, the calculations indicate a distinctly stronger interaction for the FePc, corroborating the interpretation of the STM data and the different magnetic coupling between the central atom in MPc and magnetized Co-films.

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9:45 Oral

Experimental and theoretical investigations of the electronic and magnetic coupling of iron and copper phthalocyanine to ferromagnetic Co(100) surfaces

Christiane Ziegler[1], Stefan Lach[1], Felix Schmitt[1], Jens Sauther[1], Ehesan Ali[2], Peter M. Oppeneer[2]

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Organic semiconductors with an open shell structure at the central metal atom are known to show promising magnetic properties, advertising them for use in organic spintronics [1]. For the reported investigations thin films of copper (CuPc) and iron phthalocyanine (FePc) on ultra thin layers of Co(100) were utilized. The investigation of the electronic interaction was performed by means of photoelectron spectroscopy (XPS, UPS and IPES). XPS reveals interactions between the pyrolic nitrogen atoms and Co which are different for the two Pcs. The analysis of the multiplet structures appearing for the nitrogen core levels in the submonolayer regime indicates a stronger electronic coupling of FePc to the Co surface as in the case of CuPc. UPS investigations of the valence band electronic structure with a detailed analysis of the Co dominated region near the Fermi level, indicate a rehybridization for both molecules in the sub monolayer regime.

In order to clarify the influence of the two different central atoms on the electronic and the magnetic coupling to the Co substrate, theoretical calculations were performed. DFT+U calculations with a Coulomb U term [2] using a structure of Fe/Cu-Pc adsorbed on a 3-layered Co surface indicate a distinctly stronger interaction for FePc, corroborating the spectroscopic data. Ferromagnetic coupling between FePc and Co is favored over an antiferromagnetic coupling. Comparing the theoretically found local density of states for the two different organic/inorganic hybrid systems, the spectroscopically detected features near the Fermi level will be discussed in terms of dif-

ferent rehybridization mechanisms depending on the nature of the central metal.

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[2] P.M. Oppeneer, P.M. Panchmatia, B. Sanyal, O. Eriksson and Md. E.Ali, Progress in Surf. Science, 84, 18, (2009)

10:00 Oral

Interface Effects On A Ultrathin Co Film In Multilayered Stacks Based On The Organic Semiconductor Alq3

Chiara Pernechele[1], Andrey A. Sidorenko[1], Ilaria Bergenti[2], Giuseppe Allodi[1], Roberto DeRenzi[1], Pierpaolo Lupo[1], Massimo Ghidini[1], Massimo Solzi[1], Luis E. Hueso[2], Valentin A. Dediu[2]

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Interfaces and dimensionality effects play an important role in the magnetic and transport properties of spintronic devices [1]. They are generally multilayers with two thin ferromagnetic films (cobalt or/ and manganite) separated by a third material (insulator or (in-)organic semiconductor).

In this work we study by means of zero-field ^{59}Co NMR and Squid magnetometry the correlation between microstructure and magnetic properties of thin (5-15 nm) Co layers deposited on top of organic semiconductor 50-nm thick Alq3. ^{59}Co NMR is sensitive enough to obtain detailed information on interfaces (e.g topology) and individual layers with the thickness of a few monolayers embedded in the multilayer structure. The local field at the ^{59}Co nucleus easily distinguishes different local environment, transforming NMR into a sensitive local structural probe. The crystalline phase (i.e fcc, hcp or bcc Co), the presence of impurities and the type of interface can also be identified in the cobalt spectra Uuing NMR. Effects of an insulating barrier Alox with thickness varying from 0 to 5 nm in Co/ (Alox)/Alq3/Si multilayers are investigated too. These results are compared to the morphological properties of the same heterostructures investigated by means of atomic force microscopy and to the magnetic properties measured by Squid magnetometry. The measured RT hysteresis loops show reproducible and comparable coercive field of the oredr of 15 Oe with no strong dependence of the barrier thickness. Combining magnetic and morphological data it was found that 2 nm thick barrier can assure a good quality of the Co layer.

[1] Dediu et al. Phys. rev. B 78 115203 (2008)

10:15 Oral

Efficient spin injection through exchange coupling at organic semiconductor/ferromagnet heterojunction

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Efficient injection of spin-polarized electrons from a ferromagnetic contact into a semiconductor is an important ingredient in many currently proposed spintronic devices. Such injection has been demonstrated by analyzing the circular polarization of photons that are generated after recombination of the injected electrons with holes in a quantum-well layer inside the semiconductor. The difficulty to achieve coherent spin injection and spin transport above the nanometer scale in normal metals and semiconductors, however, has stimulated the search for new materials. In this context, π-conjugated organic semiconductors (OSCs) have a decisive role, as they are mainly constituted by atoms with low atomic number Z, leading to a low spin–orbit coupling and thus to extremely long spin relaxation times. Despite the advances made in device fabrication, the fundamental knowledge about spin injection at OSCs/ferromagnet heterojunction is still missing. Here we show that the amplified spin polarized charge injection through OSCs/ferromagnet heterojunction by exchange coupling effect. Our results demonstrate that charge transfer (CT) from Fe (or Co) to a π-conjugated orbital of Alq3 at the interfaces not only creates an Ohmic contact, but also induces exchange coupling between Alq3 and the substrate as well as creates spin polarization of a π-conjugated orbital of Alq3. We anticipate our results to be a starting point for fully understanding spin injection and the behavior of organic spin valves, guiding the design of such devices and stimulating new type of applications.

COFFEE

Wednesday morning, 16 September, 10:30
Main Hall

Lectures

Wednesday morning, 16 September, 11:00
Room 213

11:00 Invited oral

Current-driven domain wall depinning and resonance in notched Permalloy nanowires

Christopher Marrows[1], Serban Lepadatu[1], Mark Hickey[1], Antoine Vanhaverbeke[2], Ola Wessely[3], Alessandro Potenza[4], Helder Marchetto[4], Timothy Charlton[5], Del Atkinson[6], Sarnjeet S. Dhesi[4], Sean Langridge[5], David Edwards[3], Rolf Allenspach[2]

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Current-driven domain wall motion not only offers fascinating basic physics but offers the prospect of storage-class non-volatile solid state memories. The fruitfly sample used in experimental studies to date is the lithographically patterned permalloy nanowire. Here we describe our recent work on such structures, with patterned pinning centres, notches, halfway along their length. Our key techniques have been high-resolution magnetic imaging by XMCD PEEM and SEMPA, as well as magnetotransport measurements. We have measured the spin polarisation of the current (P~0.4) and the degree of non-adiabaticity of the spin torque (beta~0.04) in Permalloy by studying the depinning boundary of the DWs pinned in triangular notches. We have demonstrated that the depinning current at zero field may be engineered by controlling the half-angle of the triangular notch, which varies the restoring force the pinning centre applies to the wall as it attempts to leave. We have also excited pinned walls with RF currents: in a triangular notch the behaviour is highly anharmonic, whilst a well-defined eigenfrequency is found for parabolic notches, which provide an almost harmonic pinning potential.

This work was supported by the ESF (SpinCurrent consortium), the EPSRC (Spin@RT Consortium), and Diamond Light Source.

11:30 Oral

Magnetic properties of cobalt nanowires and nanotubes

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In this work, ferromagnetic Co nanowires have been synthesized via low voltage electrodeposition method. High resolution TEM and XRD results show that the nanowires are uniform in size, and consist predominantly *hcp* structure with the magnetocrystalline easy axis (*c*-axis) perpendicular to the wire axis. SQUID measurement illustrates the dominance of shape anisotropy, manifested by the weak temperature dependence of the enhanced coercive field along the wire axis. MFM shows a strong dipole at the ends of the wire, together with a spatial magnetization modulation along the wire with a period around 700 nm. Based on theoretical modeling, such intrinsic modulation originates from the competition between the magnetocrystalline along the easy axis and the shape anisotropy along the wire axis. Low temperature magnetoresistance measurement shows irreversible magnetization switching in addition to the anisotropic behavior due to magnetization orientation dependent electron scattering. Such array could develop new microwave devices such as voltage oscillator and current-tunable microwave band stop filters.

In addition, Co nanotube arrays are fabricated by direct electrodeposition with low current density under several mA/cm^2. The wall thickness is about 15 nm and outer diameters are in accordance with pore diameter around 80 nm. TEM and selected-area electron diffraction are performed to show that the nanotubes are predominantly *hcp* single crystals with c-axis (easy axis) perpendicular to the tube axis. SQUID and MFM results indicate that the magnetization follows a circumferential direction around the tube, which is manifested in the weak magnetic signal in the MFM imaging as well as a sheared hysteresis dependence for field applied along the tube axis. This has been confirmed by theoretical modeling taking into account the magnetocrystalline, shape demagnetization and magnetic exchange energies.

11:45 Oral

Co-relation between organic spin-valves and magnetoresistance in organic diodes (OMAR)

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Study of spin-injection and -transport in pi-conjugated organics is a relatively new and less explored territory. It provides a whole new scope of physics along with immense potential for application. Major challenges for spintronics, namely optimization of spin lifetimes across relevant length scales and spin-transport through hetero-interfaces are of importance for organic spintronics as well. In this respect, organic semiconductors (OS) are envisioned to be better than their inorganic counterparts. OS comprise of mainly light molecules like carbon, hydrogen etc and possess low spin-orbit interaction and hyperfine interaction leading to longer spin-correlation length. Polymers are especially the better choice as their conjugation lead to better transport properties than non-crystalline small molecules and oligomers in films made with spin-coating and/or various printing methods. The ability to manipulate electron spin in organic molecules offers an alternative route to conventional electronics, both from fundamental and technological points of view.

Magnetic field effects on OS were demonstrated already in the early '90s. However, demonstration of successful spin injection and transport in lateral [1] and vertical [2] organic spin-valves, with inorganic metals and half-metals as the ferromagnetic (FM) electrodes and OS or oligomers as spacers, caused resurgence of interest. Successful demonstration of efficient spin injection and transport in organic spin–valve structures at room temperature has been done [3] even though the magnitude of response is small (~ 1-2%). Room temper-

ature magnetoresistance in organic diodes (OMAR) have also been demonstrated [4]. The magnitude of OMAR at room temperature is considerably higher (~20%) than the spin-valves and have potential for immediate application.

In our presentation we will co-relate the OMAR and the spin-valve effect in light of the new experimental results on the magnetic behaviour of the ITO electrode and ferromagnetism in OS. Contrary to popular beliefs the ITO electrode and OS used in the diodes were found to have ferromagnetic signatures. We will discuss about these results especially in context of "d^0 ferromagnetism" observed in oxide semiconductors. We will further discuss whether the OMAR effect is the namesake of spin-valve operation or if it is a completely different physical phenomenon.

*Corresponding author: himadri.majumdar@abo.fi

References:

[1] V. Dediu et. al., Solid State Communication **122**, 181 (2002).

[2] Z. H. Xiong et. al., Nature (London) **427**, 821 (2004).

[3] S. Majumdar et. al., Appl. Phys. Lett **89**, 122114 (2006).

[4] T. L. Francis et. al., New J. Phys. **6**, 185 (2004).

12:00	Invited oral

Magnetoresistance and spin transport in organic semiconductors

Peter A. Bobbert[1], Tho Nguyen[2], Frank W. Van Oost[1], Wiebe Wagemans[1], Bert Koopmans[1], Markus Wohlgenannt[2]

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The physics of spin dynamics and spin-spin interactions in organic semiconductors is completely different from their inorganic counterparts and this makes these semiconductors very interesting as new materials for spintronics. The most important differences are the following. 1) In contrast to inorganic semiconductors, the spin-orbit coupling in organic semiconductors appears to play an insignificant role, and it is in fact the hyperfine interaction with the hydrogen nuclear spins that dominates the dynamics of the electron spin. 2) Whereas inorganic semiconductors display band conduction, charge transport in organic semiconductors generally takes place by hopping conduction between localized states, due to disorder and small electron-transfer integrals. 3) While electron-phonon coupling in inorganic semiconductors is small and can be treated as a perturbation, such coupling is very important in organic semiconductors, leading to polaron formation and the possibility of bipolaron formation. New spin-related phenomena will be discussed in this presentation, such as the large intrinsic magnetoresistance of organic semiconductors and the peculiar behavior of the spin diffusion length as a function of temperature and external magnetic field. Novel theoretical frameworks will be presented in which these phenomena can be understood.

LUNCH

(maps available at the Reception desk)
Wednesday afternoon, 16 September, 12:30
building of the Faculty of Physics

Posters

Monday, 14 September

Joint Poster Session

Monday & Wednesday
Monday afternoon, 14 September, 16:00
Main Hall

16:00	Poster	E-1

Magnetic tunnel junctions grown on organic template for flexible electronics

Clement Barraud, Cyrile Deranlot, Pierre Seneor, Richard Mattana, Stephane Fusil, Karim Bouzehouane, Frédéric Petroff, Albert Fert

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Among the promising ideas emerging from the organic electronic, one is to develop electronic
devices with both metals and organic materials which are grown on flexible substrates [1]. The
range of such industrial applications is huge. It concerns, for instance, flexible screens but also
flexible sensors. In 1996, Parkin [2] showed that "giant magnetoresistive" sensors can be grown of organic substrates or on thin organic films deposited on silicon substrates. Another kind of system commonly used for spintronic applications is the magnetic tunnel junction. Two ferromagnetic metals are then separated by a very thin insulator which acts as a tunnel barrier. An important change in the electrical resistance is expected when the two magnetizations switch from a parallel to an anti-parallel configuration. It is the so-called tunnel magnetoresistance (TMR) effect. We report on the fabrication and electrical characterization of micrometric MTJs grown on organic thin films spin-coated on silicon. The polymer used as organic buffer layer is the widespread PEDOT-PSS. PEDOT-PSS (Poly(3,4-ethylenedioxythiophene)poly-styrenesulfonate) is a flexible organic layer widely used in organic electronics. We have grown by sputtering technique standard Co/Al_2O_3/Co magnetic tunnel junctions on the PEDOT:PSS organic buffer layer and on a reference Si/SiO2substate. MTJs with large area (0.3*0.3 mm²) have been studied in order to test the homogeneity of the thin Al_2O_3 (2.5 nm) tunnel barrier. First the roughness and local conductance of Co/Al_2O_3 bilayers have been characterized by a conductive tip AFM in order check the low roughness and the absence of short-circuit required for working MTJs. Co/Al_2O_3 bilayers grown on PEDOT:PSS and Si/SiO_2 substrate show a very low roughness about 0.6 nm and

similar local conductance and without pinholes. Studies of the spin dependent transport show similar TMR for MTJ grown on PEDOT:PSS and on Si/SiO_2. This study shows the ability to conserve spin dependent transport properties of spintronics devices grown on an organic substrate.

[1] S.R. Forrest, Nature, Vol. 428, 911 (2004)
[2] S.S. Parkin, Appl. Phys. Lett. 69, 20 (1996)

16:00 Poster E-2

Magnetoresistance effects in phthalocyanine based magnetic tunnel junctions

Clement Barraud[1], Richard Mattana[1], Pierre Seneor[1], Raphael Guillemet[1], Stephane Fusil[1], Karim Bouzehouane[1], Cyrile Deranlot[1], Frédéric Petroff[1], Albert Fert[1], Jean Batiste Beaufrand[2], Dong Jik Kim[2], Jacek Arabski[2], Samy Boukari[2], Martin Bowen[2], Eric Beaurepaire[2]

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Organic spintronics has been the focus of a growing interest since 2002 and the first
observation of magnetoresistance effects through sexithiophene (T_6) films [1]. Since then, most of the successful studies have dealt with aluminium tris(8-hydroxyquinoline) (Alq_3) [2]. Here we report on the fabrication and magneto-transport properties of nanometer size organic magnetic tunnel junctions based on the cobalt-phthalocyanine organic semiconductor.
In order to elaborate these nanometer size tunnel junctions, the fabrication is carried out using a combination of UV lithography and conductive tip AFM nanoindentation process where the tip-sample resistance is monitored in real time [3]. With this technique, we are able to control the section (about few tenth nm^2) of the nanocontact. Moreover, by indenting further, we can also vary the organic tunnel barrier thickness. As a final step, cobalt is deposited into the nanocontact forming a magnetic tunnel junction enabling the study spin dependent tunnelling through the CoPc layer.
We have performed spin dependent transport measurements in Co/CoPc/Co magnetic tunnel
junctions where the thickness of the organic semiconductor is only few nm. We have observed a significant magnetoresistance effect (>30 %) at low temperature. Two contributions to the magnetoresistance are isolated: a tunnel anisotropic magnetoresistance (TAMR) and a spin valve effect associated to the magnetic configuration of Co electrodes (parallel and antiparallel magnetic configurations).

[1] V. Dediu *et al.*, Solid State Communication, 122, 181 (2002)
[2] Z.H. Xiong *et al.*, Nature, 427, 821 (2004); L.E. Hueso *et al.*, Adv. Mater. 19, 2639 (2007); T.S Santos *et al.*, Phys. Rev. Lett. 98, 016601 (2007); H. Vinzelberg *et al.*, J.Appl. Phys. 103, 093720 (2008)
[3] K. Bouzehouane *et al.* Nanolett. 3 1599 (2003)

16:00 Poster E-3

Pentacene thin films for organic spintronics applications

Patrizio Graziosi[1], Mirko Prezioso[1], Ilaria Bergenti[1], Alberto Riminucci[1], David Brunel[1], Carol Newby, Fenghong Li[2], Yiqiang Zhan[2], Mats Fahlman[2], Massimo Ghidini[3], Valentin A. Dediu[1]

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The utilization of high mobility organic semiconductors in spintronics is expected to provide a breakthrough improvement in the device operation. Pentacene is nowadays the material of choice for most OFET investigations and thus is among the main candidates for spintronic applications.

We present here the study of the growth dynamics of pentacene on ferromagnetic oxide surfaces, namely on $La_{0,7}Sr_{0,3}MnO_3$ (LSMO) manganites. This work is additionally stimulated by recent report of magnetoresistance in planar devices with two LSMO electrodes and pentacene spacer[1]. It is important to mention that no magnetoresistance has been detected in similar geometry with cobalt electrodes [2].

Pentacene films in the thickness range of 3-100 nm were thermally evaporated in UHV conditions on LSMO films at several deposition temperatures. A strictly diffusion limited, thermically activated (Arrhenius-type) growth was found. The molecule diffusion was found to have a strongly anisotropic character. Pentacene molecules form very wide and extended flat islands having size of a few microns. These large structures are delimited by quite high steps and show terraced features. The step height was estimated independently via AFM characterizations and NEXAFS studies and constitutes 1.5nm, corresponding to 70° angle between molecule axis and surface plane.

The pentacene growth on LSMO is related to the uncommon properties of the surface [3], which determine the growth mode by acting on the molecules mobility.

[1] T. Ikegami et al. Appl. Phys. Lett. **92**. 153304 (2008)

[2] M. Popinciuc, Ph.D. Thesis, University of Groningen (2007)

[3] M. Cavallini et al,. arXiv:cond-mat/0301101v1

16:00 Poster E-4

Electrical and Magnetic switching in Alq3 and Pentacene Organic Spin Valves

Valentin A. Dediu, Mirko Prezioso, Alberto Riminucci, Patrizio Graziosi, David Brunel, Ilaria Bergenti, Carol Newby

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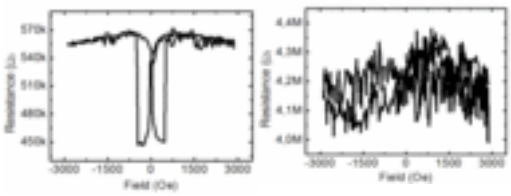
In this work we show the magnetoresisitve and electrical properties of Alq3 based ($La_{0.7}Sr_{0.3}MnO_3$(LSMO)/Alq3/AlOx/Co) and Penta-

cene based ($La_{0.7}Sr_{0.3}MnO_3$/Alq3/Pentacene/AlOx/Co) organic spin valves (OSV).

Alq3 is one of the most studied organic semiconductors (OS), both for organic LEDs (OLED) applications and, more recently, for organic spintronic devices. Pentacene instead is the main OS for organic FETs (OFETs) because of its high mobility. Therefore, these two organic materials are very important for spintronic devices too.

When measuring the Alq3 based OSV with a -100 mV applied bias, we observed the existence of two resistive states, one high resistance state (about 4.2 MΩ) that showed no spin valve effect, and a low resistance state (about 560 kΩ) which showed a spin valve effect of up to 22% at 100 K. The high resistance state was induced by the application of a -1.5 V bias to the LSMO electrode prior to the measurement, whereas we applied a positive bias greater than 2 V to drive the device in the low resistance state. The figures show the magnetoresistance of a OSV in its low resistance (top) and high resistance (bottom) state. Above a positive bias threshold, the spin valve effect increased as the voltage applied before the measurement was increased.



Very interesting results were also obtained with the Pentacene based spin valves. In this case the IVs of the devices showed a switching in the differential resistance at threshold voltage, which increased with temperature. They showed a spin valve MR when the absolute value of the applied voltage was below the threshold. When the absolute value of the applied voltage was greater than the threshold, the spin valve MR was replaced by a negative magnetoresistance.

These results show joint electrical and magnetic effects, which is very interesting both from a fundamental and from an applicative point of view.

16:00	Poster	E-5

Role of Interfacial States in Alq3-based Organic Spin Valves

Valentin A. Dediu[1], Ilaria Bergenti[1], Alberto Riminucci[1], Mirko Prezioso[1], Patrizio Graziosi[1], Donald MacLaren[2], John Chapman[2], Francesca Casoli[3]

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Applications of organic semiconductors into spintronic domain, motivated by unquestionable advantage of weak spin-orbit coupling, have been recently successfully demonstrated; in particular magnetoresistive spin valves (SV) and magnetic tunnel junction (MTJ) based on archetypal organic materials Alq3 have been experimentally obtained.In spite of the different mechanism, injection/transport for SV and tunnelling for MTJ, performances of such devices are affected not just by the properties of its constituent but also by their metal/organic interfaces.Considerable interest has been devoted to the investigation of Alq3 based vertical spin valves containing half metallic manganite (LSMO) as bottom electrode and Cobalt as the top one.While the deposition of Alq3 on LSMO give rise to a well defined interface which energetics has been investigated, it has been claimed by several authors that the Co/alq3 interface is intrinsically "ill defined" and transport properties are strongly affected by the Co clusters that can be found inside the organic material down to 50 nm from the surface.The introduction of a thin (1-2 nm) Al2O3 barrier between the Alq3 and the Co results in a sharper definition of the metal/organic interface. From X-ray reflectivity measurements, the intermixing region at the interface between Alq3 and Co can be estimated to be around 2.5 nm, while the direct interface suffers from a thicker degradation layer. These results have been confirmed by cross Sectional TEM and High Energy Photoemission Spectroscopy (HAXPES) measurements, which indicate that the tunnel barrier prevents also the reaction of Co with the organic molecules. Scarce control of interfacial layer for the direct deposition of Co on top of Alq3 gives rise to a variety of spurious effect on the device operation such as high closure field and not uniform and defined thickness of organic layer. In this latter condition, the estimation of length over which coherent spin transport is achieved is hardly reproducible. We present a full characterization of LSMO/Alq3 and Alq3/Co interfaces in presence of buffer insulating layer and we correlate the results with the operation of spin valve device. The presented results clearly indicate that extension of hybrid spintronic devices to practical application requires the engineering of interfaces between organic and the spin polarized materials.

16:00	Poster	E-6

Transmission of electrons through graphene nanoribbon in a two-dimensional semiconductor structure

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Electronic properties of graphene attracted enormous interest recently due to its real two-dimensionality and very unusual electronic energy spectrum which can be described by the relativistic Weyl model. It is generally believed that graphene may become one of the most important materials for future nanoelectronics.

We have considered transmission of electrons through a graphene nanoribbon being in contact with two-dimensional semiconductor layers. We assumed that the parameters of semiconductor-graphene-semiconductor structure correspond to the quantum regime, where the wavelength of transmitted electrons is of the order

of the nanoribbon width. We formulated a model of the electronic band structure of such a system using a set of band parameters corresponding to the electron energy gap, effective mass, and the potential profile across the structure. This allowed us to find the exact solutions for the electron scattering states in the system. We have calculated the reflection and transmission coefficients of electrons as a function of the parameters corresponding to different semiconducting materials in contact with graphene. The main effect is related to the dependence of transmission on the potential profile, which can be easily controlled by applying the gate voltage, and therefore can be used in graphene-based electronic devices.

16:00	Poster	E-7

Temperature dependence of the Kondo resonance in quantum dot attached to ferromagnetic leads with non-collinear magnetizations

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The equation of motion method for the Green functions has been used to analyze the Kondo resonance in the density of states and conductance of a quantum dot attached to ferromagnetic leads, whose magnetic moments are in general non-collinear. We go out beyond the Meir-Wingreen approximation using the Lacroix decoupling approach. The numerical analysis of the temperature dependence of the Kondo resonance for several magnetic configurations will be presented and discussed.

16:00	Poster	E-8

Induced magnetic phase transitions in ferromagnetic semiconductors under high pressure

Rasul K. Arslanov[1], Ahmedbek Y. Mollaev[1], Ibrahimkhan K. Kamilov[1], Temirlan R. Arslanov[1], Ullubii Z. Zalibekov[1], Vladimir M. Novotorzev[2], Sergey F. Marenkin[2], Irina V. Fedorchenko[2]

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Magnetic phase transitions were found and studied in ferromagnetic semiconductors $Cd_{1-x}Mn_xGeP_2$ and $Cd_{1-x}Mn_xGeAs_2$ according to change of magnetic dynamic penetrability at high hydrostatic pressure up to 9 GPa. Measurement were carried out in high pressure devise of "Toroid" type, dynamic magnetic penetrability m was measured by fluctuation method, and then recalculated into magnetic susceptibility χ according to formula $\chi=(\mu-1)/4\pi$.

Figures show dependencies of relative magnetic susceptibility χ/χ_o (χ_o– value of magnetic susceptibility at atmospheric pressure) for samples $Cd_{1-x}Mn_xGeP_2$ and $Cd_{1-x}Mn_xGeAs_2$. Figure at the right shows dependence $\chi(P)/\chi_o$ for $Cd_{1-x}Mn_xGeAs_2$. It is seen from figure that maximum $\chi(P)/\chi_o$ moves to lower pressures from P=2 GPa for x=0.06 up to P=1.6 GPa for x=0.3 with increase of percent content of manganese (x). The amplitude of maximum on the on the contrary grows with increase of percent content of manganese. The same situation is observed on $Cd_{1-x}Mn_xGeP_2$ with x=0.9 and x=0.18. It follows from analysis of dependence $\chi(P)/\chi_o$ that there takes place spin - reorientation transition ferromagnetic - antiferromagnetic (FM–AFM) in studied samples; the presence of susceptibility shoulder after FM - AFM transition characteristical of antiferromagnetic state testifies it. There were no FM - AFM phase transitions found on base samples $Cd_{1-x}Mn_xGeAs_2$ and $Cd_{1-x}Mn_xGeP_2$.

The works has been carried out under financial support of program of Presidium of the Russian Academy of Science "Heat physics and mechanics of external energy influences and physics of heavily condensed matter".

16:00	Poster	E-9

Preparation of $Zn_{0.90}Co_{0.10}O$ Thin Films by r. f. Magnetron Sputtering

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In this work, $Zn_{0.90}Co_{0,10}O$ diluted magnetic semiconductors (DMS) synthesized by mechanical milling and thermal treatment. The formation of solid state reaction was monitored by differential thermal and thermo gravimetric methods (DT-TGA). Substitution of Co^{+2} ions with Zn^{+2} host atoms in ZnO lattice had been analyzed by using X-Ray Diffractometer (XRD), Fourier Transform Infrared (FT-IR) and X-Ray Photo Spectroscopy (XPS). Additionally, the impurity phases such as CoO, Co_3O_4 and $ZnCo_2O_4$ or Co clusters have not been detected in the samples. However, in the samples, about 1% tungsten (W) impurity from the mortar of the mill was observed in the form of $ZnWO_4$.

The prepared $Zn_{0.90}Co_{0.10}O$ semiconductors pressed under a force of 25tons to form 2inch diameter target. Then, by using this target, thin films were grown on sapphire (α-Al_2O_3) substrate that is ordered in (0001) direction. The structural studies have been done by XRD, Energy Dispersive X-Ray Spectrometry (EDS) and XPS. It is found that ZnO single crystal with (0002) orientation, coherent to the substrate structure, has been obtained. XPS results have shown that there is ~15.6eV energy difference between Co2p1/2 and Co2p3/2. This proves that Zn atoms were replaced by Co atoms in the host ZnO matrix due to this energy difference. XPS results demonstrated that the grown ZnO single crystal film does not have impurity phases such as CoO and Co_3O_4. Also Co clusters have not been observed and homogeneity of Co atoms in the lattice has been shown. The magnetic behavior of the thin films was studied using a vibrating sample magnetometer (VSM). Magnetic field versus magnetization measurements in field range of ±2 T at 10K and 300K temperatures, and temperature versus magnetization measurements between 10K and 300K in 10kOe applied magnetic field was performed. In the light of the results obtained, the suitability of $Zn_{0.90}Co_{0.10}O$ diluted magnetic semiconductors for the spintronic applications were

investigated.

16:00	Poster	E-10

High resolution X-ray diffraction studies of CrO_2 films on Al_2O_3

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Chromium dioxide is an important material for spintronic applications at room temperature due to its ferromagnetic half-metallicity (transport spin polarization of ~100%) and a relatively high Curie temperature of ~ 400 K.

Films were grown on Al_2O_3 (0001) by atmospheric pressure chemical vapour deposition in a wide range of deposition temperatures and oxygen flowrates. The fact that the films are grown on lattice mismatched substrates leads to film strain which induces defects and polycrystalline growth. In particular, a buffer layer of (antiferromagnetic insulator) Cr_2O_3 develops at the CrO_2/Al_2O_3 interface.

Here we use high resolution X-ray diffraction methods to study the crystalline (epitaxial) quality, lattice parameters, orientation relations, strain gradient, thickness and roughness of the layers. These measurements are complemented by concentration depth profiling using ion beam analysis to follow the composition through the layer structure. The results will be correlated with electrical transport and spin polarization data reported elsewhere.

16:00	Poster	E-11

Anomalous behavior of Fe_3O_4 films on Si(100)

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Amongst the ferromagnetic materials that are predicted to be half-metallic, magnetite (Fe_3O_4) is one of the most interesting because of its extremely high Curie temperature of ~ 860 K. Both properties make Fe_3O_4 a very attractive material for application in spintronic devices operating at room temperature.

Fe_3O_4 thin films were grown on Si(100) to ensure compatibility with Si technology. The pulsed laser deposition (PLD) technique was used and the substrate temperature was maintained at a relatively low value of 210 ºC. The background gas consisted on a mixture of oxygen and argon.

The as-grown layers were studied *vis-a-vis* of their phase purity, stoichiometry, magnetic, electrical and magnetotransport properties. The latter show an anomalous behavior in the temperature range 200 – 240 K where a resistivity drop and a signal reversal of the magnetoresistance are observed. These results will be discussed in terms of an inversion layer formed at the interface between SiO_2 and Si in the thinner films. Moreover, Seebeck coefficient data recorded on these films also show abnormal high values.

16:00	Poster	E-12

Ferromagnetism in transition-metal doped ZnS

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We report a first-principles investigation of the electronic properties of ZnXS in the hexagonal-wurtzite phase, where X = Cr, Mn, Fe and Co with 12.5% of concentration. The calculations are made by the full potential linearized augmented plane waves method (FP-LAPW) with the local spin density approximation (LSDA). we study the local density of states of the magnetic ions and the host semiconductor and the magnetic exchange interactions as a function of transition-metal ion. Our results show a half-metallic behavior for Cr, Fe and Co impurities while for Mn the material keeps its semiconducting behavior. The band structure calculations show d-dominated impurities bands in the gap, which hybridize strongly with the p-S states.

16:00	Poster	E-13

Comprehensive study of high temperature ferromagnetic semiconductor $Cd_{0.82}Mn_{0.18}GeAs_2$

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Baric dependencies P≤7 GPa, magnetic field H≤5kOe and dependencies of specific resistivity ρ, Hall coefficient R_H dependencies, dependencies of cross magnetic resistance $\Delta\rho_{xx}/\rho$ and dependencies of relative susceptibility χ/χ_0 in high temperature ferromagnetic semiconductor $Cd_{0.82}Mn_{18}GeAs_2$ have been studied. There were taken several methods for measuring: baric dependencies were measured at rise and fall of hydrostatic pressure using method [1,2], magnetic susceptibility using frequency method [3], magnetic field dependence using standard method. Structural reversible phase transitions semiconductor–metal at P =5.2 GPa were found on baric depend-

ence ρ(P) and R_H(P). One may outline four regions on curve R_H(P): P≤0.6 GPa – region of impurity conductivity, P= 0.6 – 1.9 GPa – region of exhaustion, P=1.9 – 4.5 GPa – region of fall of R_H (reasons are still in discuss) and P=4.5 – 6.5 – region of phase transition. In the region of phase transition P > 5 GPa, curve comes out for saturation, where electric conductivity σ = 3000 Ω^{-1} cm^{-1}, and concentration of charge carriers $5\times10^{20}cm^{-3}$ testifies that there takes place semiconductor–metal transition. Magnetic phase transition ferromagnetic–antiferromagnetic was found for the first time on baric dependence of relative magnetic susceptibility both at rise and fall of pressure. Temperature dependence of phase transition ferromagnetic–antiferromagnetic is measured from temperature, phase transition ferromagnetic–antuferromagnetic with increase of temperature T=310–334 K moves to low pressures, amplitude of phase transition falls.

Cross magnetic resistance was measured in magnetic field up to H≤5 kOe. Magnetic resistance is observed in magnetic field under pressure in $Cd_{0.7}Mn_{0.3}GeAs_2$. The increase of pressure and magnetic field leads to growth of positive magnetic resistance, and the amplitude of positive magnetic resistance reaches maximum in field H = 5 kOe and P≤1 GPa. Further increase of pressure leads to suppression of positive magnetic resistance. In the region of magnetic phase transition ferromagnetic–antiferromagnetic at P □2 GPa, magnetic resistance changes positive sign for negative. Negative magnetic resistance is □ 3% at pressure P >4.5 GPa and field H = 5 kOe. To our opinion, at pressures P < 1GPa a considerable contribution in magnetic resistance gives the scattering of carriers on fluctuations of magnetizing–magnetic resistance is positive. With growth of pressure in magnetic field occur regulating of spins of manganese ions, scattering decreases, and it leads to negative magnetic resistance. Hysteresis of magnetic resistance is found on dependencies $\Delta\rho_{xx}$/ρ(P) at pressure fall. Hysteresis of magnetic resistance, apparently, is a characteristic of anomalous scattering of charge carriers, occurring at transition in magnetic regulated state at forming of ferromagnetic clusters of nano-measuring (ferrons).

The works has been carried out under financial support of program of Presidium of the Russian Academy of Science “Heat physics and mechanics of external energy influences and physics of heavily condensed matter”.

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16:00 Poster E-14

Preculiarities of thin films LaSrMnO prepared by extraction-pyrolysis technique

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The transfer of sistem to granular or qusi-twodimention state can chance the energetic structure of substance and open the addition properties. The usage of solution techniques leads to ultra fine structure of filming materials recieving. We have prepared thin films of La1-xSrxMnO3 by extraction-pyrolysis (EP) method with the aim to investigate their properties. The components of composite oxide La0,7Sr0,3MnO3 have been extracted from inorganic solutions. After amplification of metal concentration the organic extracts have been mixed in correlaton La:Sr:Mn = 0,7:0,3:1. The obtained solution was deposited on quartz substrate by spin coating technique. After drying at 100 – 120 ºC the wetting films are undergoed to pyrolysis in the opened vertical quartz furnace. Films with thick of 300 – 400 nm was sintered at different temperatures and single phase La0,7Sr0,3MnO3 films was obtained at 730 – 800 ºC. The average size of grain is about 70 nm. Magnetic measurements have been made with SCVID magnetometer at the temperatures 4,2 – 350 K in fields H = 0 – 64 kA/m. It was established that magnetic behaviour of films depends on annealing temperature. The films prepared by annealing at 600 ºC shown the thermomagnetic effect and the temperature dependance have viuve like a spine glasses. After high temperature annealing at 730 ºC the dependance σ(T) is closed to ferromagnetic and Kjuri temperature is increased. The transition became more more expressive. At the low temperature we have ansambles of weakly interconnected grains. In this case the magnetic properties are determined by individual properties of grains. As known, at the lowering of grain sizes the Kjuri temperature and magnetic properties are reduced. For this reason, properties of films likes to spine glasses. The high temperature pyrolysis leads to increase of grain sizes and additional oxygen enrichment. Properties of films depending on the annealing conditions are changed from spine glass to ferromagnetic.

16:00 Poster E-15

Effect of cation size and disorder on the structural and transport properties of the manganites.

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We report the results of the magnetic, electrical and thermal transport properties of

mixed-valence with A site cation disordered $La_{0.35}Pr_{0.15}Ca_{0.16}Sr_{0.34}MnO_3$ (ionic radii

$<r_A>$ of 1.237 A^0) manganites. The x-ray diffraction pattern of poly

crystalline $La_{0.35}Pr_{0.15}Ca_{0.16}Sr_{0.34}MnO_3$ manganites infers the orthorhombic

structure. Detailed resistivity measurement shows the semiconducting like behavoir

throughout temperature range. The dc magnetization measurement reveal that sample

undergoes a phase transition from paramanetic to ferromagnetic phase at T_c ~ 275 K

and to an antiferromagnetic phase at T_N ~ 155 K upon further cooling.

The specific heat measurement depict a broadened jump in the vicinity of Tc,

indicating the magnetic ordering and magnetic inhomogeneity in the sample.

16:00	Poster	E-16

Spin injection and detection in ZnO-based structures

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Oxide semiconductors are promising materials for spintronic applications since the performed experiments suggest of ferromagnetic ordering at room temperature. However, there are only a few papers devoted to investigation of the spin injection from the oxide-based ferromagnetic layers into conventionsl semiconductors. In order to detect the spin polarization of the injected carriers one uses as a rule light-emitting diode (LED) technique, which is especially suitable for III-V semiconductors. In this work, multilayer structure consisting of ferromagnetic $Zn_{1-x}Co_xO$ film and diamagnetic ZnO layer has been studied. The oxide layers were grown by pulsed laser deposition and RF-diode sputtering technique. Because of limitation in using LED technique for the studied structures direct mesurements of magtnetoresistance has been applied to study the spin injection. All measurements were performed with the magnetic field directed in plane of semiconductor layers. The obtained results on magnetic field dependence of magnetoresistance in $Zn_{0.95}Co_{0.05}O$ /ZnO structures show spin-valve type behavior and give clear evidence for effective polarization of the injected current.

16:00	Poster	E-17

Magnetic properties of Co doped ZnO nanowires

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We employed template replication by electrochemical deposition as the technique to prepare transition metals doped zinc oxide nanowires. Polycarbonate nanoporous membranes obtained by swift heavy ions irradiation and subsequent etching were used as templates. The pores were filled with the desired material by electrochemical deposition from an aqueous bath. The properties of the nanowires were investigated by scanning electron microscopy, energy dispersive X ray analysis and optical spectroscopy. We investigated the effect of preparation condition (i.e. deposition potential, deposition bath composition) on nanostructures properties. We found that the magnetic properties are extremely sensitive to preparation conditions and accordingly can be easily tuned.

16:00	Poster	E-18

Role of elastic stresses in the magnetic properties of solids

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Physical processes in magnet-containing media determine the regularities of evolution of states, properties and phenomena. The development of the more sensitive methods of investigations led to the specialization of studies in the narrower areas, as a result the possibility to observe and to generalize laws of physical processes was lost. The analysis of results about well studied galvanomagnetic phenomena into magnetodielectrics, semiconductors and metals makes it possible to reveal the generality of the effects of temperature, magnetic field and pressure (T, H, P).

In the present work we consider the mechanism of elastic strains and stresses as the main controlling factor of structure change under the influence of temperature, magnetic field, hydrostatic pressure. We should take into account that the energy of elastic deformation is commensurate to the energy of electric interactions and that is much higher than the rest of the bonds of lower energy value. Besides, the energy elastic stresses are of long range, so it forms the linearity in magnetization and bulk change. These regularities requires a fundamental understanding of the laws of interaction with respect to accepted interpretation of quantum mechanical forces of short range that are attributes of magnetism formation.

The role of elastic stresses in the linear changes of the magnetostriction, magnetization, magnetoelasticity of single-crystal magnetic semiconductors is described. We suggest also the explanation of the colossal magnetoresistance as a consequence of the elasticity laws. Here we state the results of the causal action of the elastic stresses that control the evolution of the structure and the properties and form the phase transformations in magnetic dielectrics and metals.

P.I. Polyakov, T.A. Ryumshyna. Magnetism and Laws of Bulk Elasticity, Transworld Research Network, 2009.

16:00	Poster	E-19

Substrate-dependent electronic structure of single-crystalline multiferroic $DyMnO_3$ thin films

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Magntetoelectric (ME) multiferroics, which exhibit coexisting magnetism and ferroelectricity with cross coupling, are being extensively investigated because of the scientific importance of the electronic origin of magnetoelectrics and relevant prospective applications in

devices, such as the magnetic-field controlled electro-optic or piezoelectric devices, with new functionalities. The multiferroicity is observed in such manganites as $RMnO_3$ (R = Dy, Tb, Gd) and $TbMn_2O_5$. In order to design the devices for the potential applications of $RMnO_3$ manganites, it is essential to prepare the thin films with a well-defined crystalline structure. In this study, we report the fabrication of the single crystalline orthorhombic $DyMnO_3$ thin films on different substrates. We investigated the substrate-dependent electronic structure and anisotropic bonding of the Mn *3d* states in $DyMnO_3$ thin films on $SrTiO_3$(001) and $LaAlO_3$(110) substrates using polarization-dependent x-ray absorption spectroscopy (XAS) at O *K*-, Mn *L*- and Mn *K*-edges for three polarizations, E ∥ *a*, E ∥ *b* and E ∥ *c*. Polarization-dependent x-ray absorption spectra at O *K*-, Mn L_{23}- and Mn K-edges of orthorhombic $DyMnO_3$/$LaAlO_3$(110) thin films show strong polarization dependence for three polarizations, E ∥ *a*, E ∥ *b* and E ∥ *c*, whereas orthorhombic $DyMnO_3$/$SrTiO_3$(001) thin films show nearly isotropic spectral structure. The main peak in polarized Mn $L_{2,3}$-edge XAS spectra of $DyMnO_3$/$LaAlO_3$(110) thin films for the *E* ∥ *b* polarization lies at a lower energy than for polarizations *E* ∥ *a* and *E* ∥ *c*. The disparate spectra for polarizations *E* ∥ *a* and *E* ∥ *b* indicate a great anisotropy in Mn 3*d*-O 2*p* hybridization and charge transfer from O 2*p* to Mn 3*d*, reflecting an orbital ordering and a highly anisotropic coplanar Mn-O bonding in $DyMnO_3$/$LaAlO_3$(110) thin films. These results indicate orbital ordering of e_g-orbital and thus the highly anisotropic in-plane Mn-O bonding which is an indispensable factor to the formation of complicated incommensurate modulated magnetic structures observed in orthorhombic $DyMnO_3$. Utilizing the technique of epitaxial strain stabilization, one can control electronic structure via growing $RMnO_3$ thin films of the same compound on different substrates. The present results provide important implications for the microscopic understanding of the $RMnO_3$ manganites.

16:00	Poster	E-20

Spin polarization at a Co_2MnSi/MgO interface studied by spin resolved photoemission

Jan-Peter Wüstenberg[1], Sabine Neuschwander[1], Takayuki Ishikawa[2], Masafumi Yamamoto[2], Jan Minàr[3], Jürgen Braun[3], Hubert Ebert[3], Martin Aeschlimann[1], Mirko Cinchetti[1]

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Heusler compounds are promising candidates for spintronics applications due to their predicted minority spin gap at the Fermi energy. For the functionality of tunneling magnetoresistance (TMR) devices the interface spin polarization is of utmost importance. However, even though large TMR ratios have been obtained with MgO barriers at low temperatures [1], the predicted bulk half metallicity has not been observed at the clean surface so far. In this contribution we present spin resolved low energy photoemission data as well as results from LEED and Auger spectroscopy of fully epitaxial Co_2MnSi films deposited on MgO. The spectra suggest the presence of a Mn-Si terminated surface after sputter-cleaning and annealing to 600°C. The presence of minority surface states leads to a strong suppression and even inversion of the surface spin polarization at the Fermi energy, in good agreement with our theoretical calculations. We find that these states vanish upon deposition of a 2 nm MgO tunnelling barrier, yielding a maximum spin polarization of 30% after annealing to 500°C.

[1] S. Tsunegi, Y. Sakuraba, M. Oogane, K. Takanashi, and Y. Ando, Appl. Phys. Lett. **93**, 112506 (2008)

Symposium F

Novel bio&chemosensing materials for health, safety and security applications

Welcome

Recent advances in nanotechnology and nanomedicine have brought novel materials and methods to light, which are expected to result in the development of smart sensors and detection systems for bio-hazards and explosives. The session is expected to attract young researchers embarking on careers in bio-chem sensing who will greatly benefit form this event.

Bio- and chemosensing need to develop advanced technology for the rapid detection and early warning of threats to national security, whether this is sniffing bombs in airport security stands or monitoring the environment continuously for abnormal levels of chemical/ biological agents that are potentially hazardous to human health and welfare. The Department of Homeland Security in the US recently came up with the idea of incorporating microsensors in watches equipped with a GPS system which will be wirelessly linked to a centralized monitoring facility and which will trigger an alarm if there is detection of abnormal levels of a particular chemical by many individuals within a certain location, thus enabling the timely identification of both the source and type of the potential threat. The general approach that we propose to follow is to invite experts from the various fields related to bio-chemical sensing of threat agents to give seminars attended primarily by younger professionals embarking on careers in defense and security (who will present contributed talks or posters on their current research activities). This EMRS conference will produce a volume of special issue summarizing the current state of the detection technologies and also identifying future trends and suggesting specific directions for the research and development of micro and nanosensors that meet the current global and regional needs.

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Proceedings

Arrangements for the publication of the *Proceedings of the Symposium* in **SENSOR LETTERS** (American Scientific Publishers) has been done.

Programme

Monday, 14 September

Opening & Plenary Session

Czochralski Award Ceremony
Monday morning, 14 September, 10:30
Small Hall (237)

LUNCH

(maps available at the Reception desk)
Monday afternoon, 14 September, 12:30
building of the Faculty of Physics

Lectures

Security and Safety
Monday afternoon, 14 September, 14:00
Room 208
Chair: Balazsi, Csaba

14:00 Invited oral

Nanomaterials Based Chemical-Biological Sensors and Detectors for Security and Environmental Safety

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The study of nanoscale systems promises to be a catalyst for scientific and technological advances requiring a fundamental understanding and exploration of new physical, chemical, and biological properties of systems in nanoscale dimensions. In recent years, newly developed architectures in nanostructures and nanosystems show improved functionalities. The objective of this presentation is to provide a comprehensive overview of our current research activities and future applications of nanostructured materials for chem.-bio sensors, for potential applications in security and environment safety.

The potential and risk for inadvertent or deliberate contamination of the environment, food and agricultural products has increased due to global threats of terrorism making decentralized sensing an important issue for several federal agencies. A nanotechnology based sensor platform enables direct electrical detection of biological and chemical agents in a label-free, highly multiplexed format over a broad dynamic range during clinical testing. Nucleic acid layers combined with nanomaterials-based electrochemical or optical transducers produce affinity biosensors such as the "DNA Biosensor" or "Genosensor" that are attractive devices for converting the hybridization event into an analytical signal for obtaining sequence-specific information in connection with clinical, environmental, or forensic investigations.

We will present preliminary results of our joint investigations to monitor and mitigate environmental pollution, a leading contributor to chronic and deadly health disorders and diseases affecting millions of people. Using nanotechnology based portable, wireless and web-based gas sensors; pollution is monitored at several ground stations.

14:35 Invited oral

Lab-on-a-Chip: from micro/nanofluidics to biomedical applications

Albert Van den berg

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Over the past 15 years, the concept of Lab on a Chip (LOC) has been developed to a great level of maturity. These microfluidics systems are nowadays employed both for research purposes as for a variety of applications in physics, analytical chemistry and biomedical applications. The first example is the use of a nanofluidic channel to separate DNA. It is found that the movement of DNA fragments is 20 nm high nanoslits in a strong electrical field is not continuous, but in a so-called "stop-and-go" manner. DNA fragments are in the stop-phase for up to 90% of the time, and in the go-phase for only 10% of the time. Moreover, even in the go-phase, the calculated electrophoretic mobility is 10-100 times smaller than that of DNA in microchannels. We suggest two possible explanations for this effect, di-electrophoretic trapping (DEP) or mechanical trapping, both induced by surface roughness effects [1]. (see fig 1). In a second example, the development of a chip for monitoring lithium medication levels in manic-depressive patients. This chip consists of a separation microchannel with integrated conductivity detection electrodes [2]. (see fig 2). Moving towards cell-manipulation and analysis, the fabrication of a million-well petri-dish will be presented, for very high throughput cell-experiments [3]. More focused on the single cell-level, we developed a chip for electroporation and gene transfection of single cells. First, we show that single cells can efficiently be electroporated as indicated by the translocation of dye's into the cell. Then, it is found that using a microfluidic chip we can electroporate and transfect C2C12 cells and human mesenchymal stem cells with high yield (>75%). It is found that GFP-ERK1 constructs transfected in HMSC's translocate form cytosol to nucleus upon stimulus with growth factor bFGF [4]. Finally, the development of silicon nanowires for biosensing will be presented.

15:10 Oral

Development of handheld DNA Sensor for foodborne pathogen detection: Fundamental factors & Challenges

Vijayalakshmi Velusamy, Khalil Arshak, Olga Korostynska, Kamila Oliva-Stasiak, Catherine Adley

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A range of sophisticated technologies to detect the contaminants in food are available. However, foodborne outbreaks from microbial contamination, toxins and chemicals are common in many countries. The electrochemical devices have received significant attention in the development of DNA biosensors. The use of a handheld DNA biosensor platform is essential for the real-time detection of specific pathogens from mixed food matrices.

The current study is aimed at developing a highly sensitive (< 1µg/mL) and stable method for the electrochemical detection of foodborne pathogens. The electrochemical technique cyclic voltammetry was used as a method of transduction and the conducting polymer, while polypyrrole was used as a platform for immobilizing DNA on the gold disc electrodes (2mm diameter).

The electrical conduction in polypyrrole is the result of electron movement within delocalized orbitals and positive charge defects which are known as polarons. Both monovalent (Na, K) and divalent electrolytes (Ca^{2+}, Cu^{2+}, Mg^{2+}, Mn^{2+}) are used as dopant for the polymerisation of polypyrrole. These dopant materials (electrolytes) are naturally found in the bacterial cells and have strong affinity to the DNA backbone. The rate of electropolymerization of pyrrole is varied with the electrolyte concentration (0.1 M – 0.5 M), applied voltage (0.5-1V), and monomer concentration (0.01 M – 0.1 M). The electrochemical entrapment was chosen as an immobilization method because it is rapid, simple, label free and is a one-step technique.

Bonding efficiency and the stability of the DNA immobilised on the polypyrrole film obtained from various electrolytes are studied. Furthermore, the main factors to be considered while designing a DNA biosensor and the challenges for the real time detection of bacterial pathogens is also discussed.

COFFEE

Monday afternoon, 14 September, 15:30
Main Hall

Joint Poster Session

Monday & Wednesday
Monday afternoon, 14 September, 16:00
Main Hall

Tuesday, 15 September

Lectures

Chemical sensing
Tuesday morning, 15 September, 9:00
Room 208
Chair: Barsony, Istvan

9:00 Invited oral

Integrating nanomaterials with nano/micro platforms to achieve enhanced chemical sensing performance

Kurt D. Benkstein, Reit Artzi-Gerlitz, Carlos J. Martinez, Baranidharan Raman, Douglas C. Meier, Steve Semancik

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Real world chemical and biochemical sensing applications present significant challenges related to the required levels of sensitivity, selectivity, speed and reliability. Various types and forms of nanomaterials offer an approach toward meeting these needs, particularly when they are used in conjunction with advanced sensor operating and signal processing modes. In this presentation we emphasize the increased capabilities that can be realized when oxide nanomaterials, used as chemiresistors, are integrated with two types of platforms: anodized aluminum oxide (AAO) nanopore arrays and MEMS microhotplate arrays. The AAO devices employ top-and-bottom contacts, and a flow-through scheme that accesses the high (internal) surface area of an array of parallel WO_3 nanotubes, to greatly enhance the sensitivity (10^2 to 10^3 over planar forms) [1]. We have also integrated a variety of oxide nanomaterials (nanoparticles, porous nanoparticle microshells, nanowires, nano-textured CVD films) into arrays of elements that can be individually temperature programmed [2,3]. Several examples will be used to illustrate how temperature-modulated operation with such nanostructured oxides produce dense data streams for complex monitoring and detection scenarios. Recent results that will be described briefly include sensing of low level chemical hazards in dynamic backgrounds and the recognition of disease-related target analytes in synthetic breath [4].

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Current address (CJM): Purdue University, West Lafayette, Indiana

[1] R. Artzi-Gerlitz, K.D. Benkstein, D.L. Lahr, J.L. Hertz, C.B. Montgomery, J.E. Bonevich, S. Semancik and M.J. Tarlov, Sensors and Actuators B 136, 257-264 (2009).

9:30 Oral

Selective Chemosensor Breathanalyzers – Non-Invasive Disease Diagnostic Tools

Pelagia I. Gouma

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Ancient Greeks physicians already knew the odor of a patient's breath is associated with some diseases. A non-invasive technology, easy to comprehend and operate, especially for those patients who need everyday monitoring and controls, breath analysis has great application potential for human disease diagnosis. Nanotechnology has provided solutions to overcome the problems with selectivity, sensitivity, response time, and sampling size that have stalled the development of diagnostic breathanalyzes to date. Our group has successfully developed chemoresistive probes that are specific to detecting biomarkers such as NO, ammonia, acetone, etc, though the use of nanostructured metal oxide polymorphs with strong physical or chemical affinity to these metabolites. Furthermore, we demonstrate how handheld breath-collection and analysis devices have been developed to assist with the monitoring of diseases such as asthma and diabetes to cholesterol or cancer. These are extremely promising nanomedicine applications that will improve human health and welfare.

9:50 Oral

Membrane based columnar microresonators for mass detection in fluids

Egbert Oesterschulze, Peter Bozek, Elena A. Ilin, Bernhard Radzio, Jenny Kehrbusch

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Microfabricated cantilevers attracted an enormous attention because of their capability to measure an extreme small mass, temperature variation, chemical reaction, or in biological applications single cells. However, operating these sensors in the dynamic mode makes detection in a fluidic environment quite intricate. In this case the quality factor drops down because of the dynamic viscosity of the fluid and possible effects of the surface energy and makes sensitive measurements almost impractical.

We present an adapted sensor design for the detection of single cells in a fluidic environment. It relies on an oscillating column oriented perpendicular to the surface instead of the cantilever geometry with

their parallel orientation to the surface. The column is immersed in a gas and only the top surface is used to interact with the fluid. In this way we propose to improve the quality factor and also the sensitivity of the set-up. For the detection of the resonance frequency of the vibrating column the beam deflection technique known from atomic force microscopy was used. To avoid severe problems with the diffraction of the laser beam at the air/liquid interface we mounted the column on a supporting membrane. With this optimized design the detection beam path and the volume of interaction was separated.

So far we investigated the damping of the oscillation by the viscous properties of pure gases. We observed that the damping is predominantly governed by the molecular mass and only to a minor extent by their viscosity. Furthermore, we were capable to detect a single latex bead with 2.5 µm diameter (12 pg weight). With a sensitivity of approximately 1fg/Hz and an uncertainty of the frequency detection of ca. 5 Hz we end up with a mass detection limit in the order of 10 fg for columnar resonators. First experiments on the operation of membrane sensors will be presented.

10:10 Oral

Functionalized radio-track etched membranes for waste-water analysis

Travis L. Wade, Metlak Al Subaïe, Vincent Lerose, Marie-Claude Clouchard

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Functionalized radio-track etched membranes (FRTEMs) are polymer films with nanopore channels perpendicular to their surface. The nanopore channels are formed by heavy ion irradiation and chemical etching. The heavy ion irradiation produces damage tracks in the form of broken bonds which become radicals. These radical tracks are chemically etched to reveal nanopore channels with a uniform diameter that is proportional to the etching time. After the etching, however, there are still radicals within the pores. These remaining radicals can be reacted with different monomers, such as RCOOH or $RCHNH_2$, to impart chemical functionalization to the nanopore interior without blocking the pores.

A thin gold or metallic layer can then be physically sputtered onto the FRTEMs to make an electrically conductive surface. The metallic layer is thin enough, ~50 nm, that is does not completely cover the pores. The thin metallic layer can function as an electrode for potentiometry.

When these functionalized membrane electrodes are immersed in a liquid sample, such as waste water, they can selectively absorb certain ions depending on their functionalization (ex: metal cations such as Cu2+, Pb2+,

... etc. for polyacrylic acid functionalized pores). They can be removed from the sample, rinsed, and placed in an electrochemical cell for potentiometric analysis.

COFFEE

Tuesday morning, 15 September, 10:30
Main Hall

Lectures

Biosensing
Tuesday morning, 15 September, 11:00
Room 208
Chair: Gouma, Pelagia I.

11:00 Invited oral

Nano/Micro-Technology based Approaches for Biosensing and Cell-Sheet Engineering

Janos Voros

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Micro- and nanotechnology brought new exciting possibilities in life sciences. In this talk, the following novel approaches will be presented:

1. A key challenge in high-throughput screening is to make the methods compatible with the fragile membrane proteins. Membrane proteins are fragile and difficult to handle, but also highly important drug targets. The combination of microfabrication, and DNA-assisted self-assembly enabled the arraying of functional biomembranes opening the possibility for their high-throughput screening.[1,2]

2. An equally important challenge is to push the sensitivity of optical biosensors to the ultimate limit of being able to detect single molecule binding events. Nanotechnology provides new, exciting possibilities through the special physical properties of nanoobjects. For example, gold nanoparticles exhibit special optical properties that can be utilized to monitor single molecule binding events such as DNA hybridization.[3,4]

3. Materials that can response to external electrical stimuli are interesting for a variety of biomedical applications: We have developed electrochemically active polyelectrolyte multilayers that can act as nanoactuators using low potentials in a liquid environment.[5] Other multilayers can be used as a sacrificial film for the harvesting of intact cell-sheets. This enables the creation of controlled 3D cell cultures that are expected to better represent the in vivo situation for e.g. drug testing experiments.[6]

References

[1] M. Bally, K. Bailey, W. Leifert, J. Vörös, T. McMurchie; G-Protein Coupled Receptor array technologies: Site Directed Immobilisation of liposomes containing the H1-histamine or M2-muscarinic receptors; Proteomics, 2009. (In press)

[2] A. Binkert, P. Studer, J. Vörös; A microwell array platform for picoliter membrane protein assays; Small, 2009. (In press)

11:30 Oral

Dual response (fluorimetric and colorimetric) sensor array based on supramolecular host-guest formation.

Laurent A. Baumes, Pedro Montes-Navajas, Mireia Buaki, Avelino Corma, Hermenegildo Garcia

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Amines have been found a challenging compound class in previous works on chemical tongues. Herein, we describe the successful application of libraries based on host-guest inclusion complexes in cyclodextrins (CDs) and cucurbiturils (CBs) for the discrimination of primary, secondary, tertiary, aliphatic, and aromatic as well as linear and branched amines in water. Besides the clear need for new detection, identification and quantification techniques of organic compounds in water, the main advantage of our approach is that an array made by combining6simple basic dyes with7commercial organic capsules allows a perfect discrimination among14amines. The same methodology is employed for other chemicals with applications of industrial interests. We detail the technique and the different algorithms we used in order to obtain the results.

Development of sensor systems that can be of general use for the detection and the determination of organic compounds in water is still a challenge in analytical chemistry. Most approaches remain largely based on the design of selective sensors for individual organic compounds. This type of sensors require dedicated synthesis while frequently showing a very limited solubility in water.Oppositely, Suslick and coworkers shown that common pH indicators, solvathochromic molecules, and coloured metal complexes can be used for the discrimination of organic molecules in water. However they, as well as others, also pointed out that amines are among the most difficult analytes to discriminate. Our previous knowledge on he properties of host-guest complexes, and considering that amines can form strong hydrogen bonds with dyes and organic capsules, and are emission quenchers, let us anticipate that the interactions between amines and host-guest complexes can result in specific variations of the optical spectrum that can serve to sensor these organic compounds in water.

11:50 Oral

Numerical simulations for the dynamics of macromolecular particles in flowing solutions inside mesopores

Antoine Khater[1], Ali Atwi[1], Abbas Hijazi[2]

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The dynamics and motion of macromolecular particles suspended in flowing fluids in mesopore systems, and their diffusive collisions and possible adsorption at surface solid boundaries, is a subject of fundamental importance for physical and biological studies. This theme has also promising new applications in the context of mesopore nanotechnology.

The analysis of the macromolecular motion in a flowing fluid inside mesopores is necessary to understand a variety of complex phenomena, as the motion of red blood cells, of suspended nanoparticles in pipe flow, and in polymer processing and composite manufacturing. The analysis is also necessary to describe the adsorption of suspended nano particles inside mesopore systems in a variety of domains. The treatment of polluted air in human lungs, of selective separation of substances and fuel and energy storage, are just some examples of adsorption applications. Adsorption is also an important effect to consider for successful encapsulation and immobilization for biological systems.

A detailed understanding of the influence of fluid hydrodynamics and Brownian movement on the diffusive collisions of macromolecular particles at the solid surface boundaries of mesopores is, however, still absent. Further, the study of the approach dynamics of the macromolecular particles in the neighbourhood of the solid boundaries, and of their diffusive collisions at these boundaries, should permit calculating with precision their adsorption properties, sorption thermodynamics, as well as their overall mobility. This study could also favour a detailed understanding of the phase separation of mixtures of different species of such macromolecules under hydrodynamic flow inside mesopores.

12:10 Oral

Immunosensors based on a new nanoelectromechanical columnar sensor

Christiane Ziegler, Oliver Köhler

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Quartz crystal microbalances were used for label-free sensing since many years. Their disadvantage is their limited mass sensitivity if compared to optical decetion methods. Usually several thousand cells are needed to get an unequivocal result, e.g. in blood typing [1].

In this context, a nanoelectromechanical sensor system was developed [2], see also abstract by Oesterschulze et al. in the same symposium) which is designed for single cell analysis in liquids. As resonance element, silicon columns are used. The damping in liquid is greatly reduced because they are structured as 'pillars in trenches' so that only the surface of the pillar cap is in contact with liquid, resulting in a high quality factor.

For their utilization as biosensors, the pillar cap surface must be selectively coated. This can be realized by different silanization and lithographic processes which are in the focus of the presented work. In this context, the controlled immobilization of antibodies to the pillar surfaces will be shown. The rest of the surface has to be coated with antisticking layers to avoid unspecific binding. First experiments in which the sensors are used in full blood will be shown, which show that only specific binding of blood cells to the pillar sur-

faces occurs.

[1] Ph.D. thesis F. Gehring, TU Kaiserslautern, 2006

[2] "Verfahren und Vorrichtung zur Detektion von Analyten in fluiden Medien", E. Oesterschulze, Ch. Ziegler, DE 10 2005 027 788

LUNCH

(maps available at the Reception desk)
Tuesday afternoon, 15 September, 12:30
building of the Faculty of Physics

Lectures

Gas and optical sensing
Tuesday afternoon, 15 September, 14:00
Room 208
Chair: Comini, Elisabetta

14:00 Invited oral

Detection of toxic species with defective carbon nanotubes decorated with metal nanoclusters

Radouane Leghrib[1], Zeila Zanolli[2], Alexandre Felten[3], Jean Christophe Charlier[2], Jean-Jacques Pireaux[3], Eduard Llobet[1]

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In the search for gas sensors with enhanced performance low-dimensional nanomaterials such as carbon nanotubes are attracting increasing interest. Their huge surface to volume ratio greatly increases sensitivity. However, selectivity still remains an important issue to be addressed.

In the last few years our approach to enhance selectivity has consisted of using plasma treatments to control interfacial (structural and chemical) defects that rule the deposition and growth of metal nanoclusters deposited on carbon nanotube sidewalls. Metal cluster surfaces act as reactive sites for the adsorption of the target molecules, and a substantial polarisation and accumulation of charge in the region between the metal cluster and the nanotube may result. This charge transfer provides important information regarding the system's electronic response. It affects the ionic component of the bonding, alters the position of the Fermi level and the band alignment (electron transport in the nanotube is affected).

In this lecture, cold plasma methods for cleaning, creating defects, functionalising and decorating with metal nanoclusters the surface of multiwall carbon nanotubes in a single step are reviewed. Details on the fabrication of gas sensors based on the resulting nanohybrid materials are given and the gas sensing properties towards traces of toxic gases such as NO2, CO, hydrocarbons and also humidity in air are shown. In order to better understand the observed gas sensing properties, a realistic description of the gas-nanohybrid material interaction (including electronic transport calculations) is provided based on density functional theory. While the presence of metal clusters favours the detection of some species (case of NO2), it is the presence of oxygenated defects caused by the plasma treatment what favours the detection of other species (case of CO).

Nano2Hybrids project (EC-STREP-033311).

14:30 Oral

Hexagonal tungsten oxide sensors: synthesis and applications

Csaba Balazsi

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Tungsten oxides are among the most used materials in hazardous gas detection. In this work, a soft chemical nanocrystalline processing route was demonstrated for the preparation of hexagonal tungsten oxides. The structure of parent phases was studied by scanning and transmission electron microscopy and by X-Ray diffraction. Electrochemical behavior of the films was determined by atomic force microscopy and cyclic voltammetry. The ion insertion properties of the tungsten-based films show a clear dependence on the presence of structural water and on the close packed structure. Sensing properties of the tungsten oxides were tested toward gaseous ammonia at various temperatures. The layers were found to be sensitive in the concentration range of 50-500 ppm at the temperature of 300 °C.

14:50 Oral

Tunable Fiber-Optic Surface Plasmon Resonance Sensors based on Indium Tin Oxide Coatings

Carlos R. Zamarreño, Miguel Hernáez, Ignacio Del Villar, Ignacio R. Matías, Francisco J. Arregui

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Here, it is studied the surface plasmon resonance phenomenon on indium tin oxide (ITO) coated optical fibers as a function of the ITO thin film properties. These novel fiber-optic SPR devices overcome the major drawbacks of the traditional prism based Kretschmann configuration due to the optical fiber setup. On the other hand, the utilization of ITO improve the traditional disadvantages of the gold or silver layer based configurations, such as quenching of the fluorescence, oxidation or hard to process surfaces. In addition, the utilization of ITO coated optical fibers as the SPR supporting substrates, shifts the resonance wavelength to the IR and near-IR region of the spectrum as well as allows the resonance wavelength tunability by adjusting the ITO fabrication parameters.

These devices were fabricated via a dip-coating process as explained by Ota etal using 200 mm fused silica optical fiber core (Thorlabs Inc.) as substrate. The deposition process was stopped at 8 dips (S1 devices) and 10 dips (S2 devices) in order to obtain ITO coatings with different properties and thicknesses of 85 and 115 nm respectively.

The absorbance spectra collected at the end of the fiber were recorded when the sensitive region was immersed in different glycerin/water concentrations from 0% to 85% corresponding to refractive index from 1.333 and 1.450 respectively.

In conclusion, these novel fiber-optic SPR based on ITO coating have been proven as a useful tool in order to determine the refractive index of the external medium in contact with the ITO layer. Furthermore, the resonance wavelength has been proven to be sensitive to variations in the ITO properties.

The authors acknowledge financial support to the Spanish Ministry of Education and Science-FEDER TEC2006-12170

15:10 Oral

Spin dependent transport

Valentin A. Dediu

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Spin dependent transport has been the object of intense research since the demonstration of magnetoresistive effects in metallic multilayers and magnetic tunnel junctions. The field has evolved to the extent that commercial applications for magnetic recording and electronic memory are now available. This was celebrated in 2007 with the Nobel prize to Albert Fert and Peter Grunberg. In the last few years the range of materials used for spin-devices has witnessed a dramatic broadening. Among others silicon, graphene and organic semiconductors have recently focused considerable efforts of the community. This symposium will be focused on the experimental and theoretical aspects of various spintronic phenomena with particular emphasis on material issues.

COFFEE

Tuesday afternoon, 15 September, 15:30
Main Hall

Lectures

Biomaterials
Tuesday afternoon, 15 September, 16:00
Room 208
Chair: Voros, Janos

16:00 Invited oral

Prethreshold photo- and exoelectron emission to characterize nanostructures for biomedical applications

Yuri Dekhtyar

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Advances in nanotechnologies need non-destructive characterization methods. To reach this, electron emission measurements, when energy to escape an electron from a tested object is not enough to destroy its molecular/atomic couples, are employed as such energies have values ~ 0.1-1 eV for non-destructive characterization. The excited electron has a mean free path (L) ~ 100-10 nm [1], correspondingly within the analysed emitter. These values of L fit sizes of nanostructures. This is a key point to apply electron emission for nanomaterials characterization. The paper is targeted to consider a prethreshold photo- and exo- electron emissions and their capabilities in respect with nanostructures characterization.

The report demonstrates that photo and exo emission measurements are capable to supply sensitive, accurate, and non-contact technique for the needs of nanotechnologies. The techniques are directed to measure the electron work function (to characterise surface potential), distribution of the electron states density; energy gap and charge relaxation; concentration of the point type imperfections, their annealing and migration (EE).
The examples to characterise natural baiomaterials as well as nanoparticles, nanomultilayer systems for biomedical field are considered.

16:30 Oral

A combination of microCT and FEM methods for development of a new scaffold for bone regeneration

Marcin K. Heljak, Jakub Jaroszewicz, Wojciech Swieszkowski, Krzysztof J. Kurzydlowski

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The main aim of the work was to develop a new method for generation and validation of numerical models of 3D porous scaffolds based on in situ micro- CT examination. The validated numerical models of the scaffold could be used in preclinical evaluation how the scaffold fulfill its role in the process of bone tissue regeneration.
The main problem with structural modeling of the scaffolds and the other porous structures is creating of reliable geometrical models, which reflect the details of such complicated structures. In the study, a finite element model of the highly porous polymeric scaffolds fabricated via Solid Free Form Fabrication Method (SFFM) was prepared based on in situ X-ray micro-CT examination. A novelty of the method is that during the micro-CT examination the sample is compressed and at each load step geometry of the scaffold was scannedand the force-displacement characteristic is recorded. Based on the CT dataset of the unloaded sample the 3D the finite element model (FEM) of the scaffold was created. To validate the numerical model FE analysis simulating the compression test in the micro-CT scanner was performed. At each load step the computed displacements of selected points of scaffold domain were compared with the displacements of the corresponding points belong to the experimental data. The created numerical model shows the satisfactory consistence with experiment in linear range of carried out compression test. The next step is to use the created numerical model in inverse analysis for finding nonlinear material properties of the scaffold.

16:50 Oral

A degradable and highly porous polyurethane scaffolds for bone tissue engineering

Monika Bil[1], Krzysztof J. Kurzydlowski[1], Joanna Ryszkowska[1], Piotr Woźniak[2], Małgorzata Lewandowska-Szumieł[2]

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Scaffold-based tissue engineering requires for transplanted or host cells a biodegradable matrix, which provides a specific environment for tissue development. Efficiency of tissue regeneration through cell implantation in scaffolds depends mainly on the architecture of the scaffold and on the properties of the biomaterial used for their fabrication. The scaffold architecture is characterized by the pore shape and size, size distribution, pore interconnectivity and throat size. Among the polymers selected for tissue engineering, polyurethanes (PUR) represent a very important group. By varying the molecular weight of polyol and the composition of the hard segments, properties of PUR can be tuned for use in tissue engineering, either for reconstruction of soft tissue or for cartilage and bone regeneration.

The objective of this study was to characterize polyurethane porous scaffolds fabricated by the salt-leaching/polymer coagulation method. The effect of solution concentration and salt particles size on porosity and pore size distribution was evaluated by mercury-intrusion porosimetry and quantitative analysis of the scanning electron microscopy images. Mechanical properties of the scaffolds were measured in compression tests and by dynamic mechanical analysis (DMA). Growth of Human Bone Derived Cells (HBDCs) cultured in direct contact with the porous polyurethane scaffolds was investigated. *In vitro* studies in simulated body fluid were carried out to determine kinetics of the biodegradation process of polyurethane foams and the bioactivity of PUR-based scaffolds. Structure of the samples after soaking in SBF was characterized by scanning electron microscopy (SEM), EDS analysis and attenuated total reflectance–Fourier transform infrared spectrometry (FTIR–ATR).

Acknowledgements

This work was supported by the Ministry of Science and Higher Education, grant R13 01901.

17:10 Oral

The influence of milling α-TCP in the formation of hydrxyapatite whiskers.

Guinéa Brasil C. Cardoso[1], Sergio L. Ramos[2], Cecilia C. Zavaglia1, Antonio Celso F. Arruda[3]

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The hydroxyapatite is one of the most biocompatible materials owing to similarities with mineral constituents of teeth and bones. They show very good biocompatibility with hard tissues, but unfortunately the mechanical properties of these materials are poor. One approach to improve their reliability is to increase its fracture toughness, which can be accomplished by precise control of the microstructure. A common way is to improve the mechanical brittleness is by the addition of short fibers or whiskers. The size of whiskers influences the mechanical properties; therefore the thinner it is the better the mechanical properties of the whiskers. This can be modified by the milling of α-TCP, which was later used in the hydrolysis, resulting whiskers. In this research, the goal was to observe the influence of the milling α-TCP, in ball mill, for zero, 24 and 48 hours. For these preparation were used α-TCP made in lab, where its shows lowest level of Mg. Then, HA whiskers were made by the alkaline (pH = 11) hydrolysis of α-TCP, as described elsewhere. The samples were characterized by X-Ray Diffractometer (XRD) and SEM. The results appears that the α-TCP milling in 48 hours, were able to form whiskers with thinness crystal, which can be observed in the SEM images. Also, by the results of the XRD analysis was able to observe that the phase of the whiskers was suchlike the hydroxapatite JCPDS 09-0432. However, the peaks were not well defined, primarily, when it is compared without milling. Its characterization shows peaks more defined and crystals more larges in the SEM images, this occurs because the size of α-TCP particles. When milling 24 hours, the results are the same without milling, probity because the similarly of their size. In all of the three cases, the α-TCP were just like the patterns JCPDS 09-034.

Wednesday, 16 September

Lectures

Chemical and biosensing
Wednesday morning, 16 September, 9:00
Room 208
Chair: Vaseashta, Ashok

9:00 Invited oral

New Functional Nanomaterials for Sensing Applications

István Bársony, Róbert Horvath, János Volk, Peter Fürjes, András Deák, Norbert Nagy, Gábor Battistig

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The exploitation of new functionalities provided by different nanomaterials is the main driving force behind the rapid development of nanotechnology. Apart of applications in structural and composite materials or coatings leading to improved mechanical, corrosion protection or wear resistance properties, the exploration of novel physical and chemical characteristics of materials on the nanoscale, their electrical, magnetic, optical, catalytic properties in integrated micro/nanodevices is the most promising from the application point of view. Sensor development will probably profit the most from the utilisation of new nano-functionalities.

The lecture will give an overview of trends and expected developments in the More than Moore domain of nanoelectronics in general and on some examples of ongoing projects at MFA:
Different nanoparticles implemented in catalytic gas sensing for combustible, explosive gas mixtures for transducers and electronic noses
Colloidal nanoparticles are formed and used in Langmuir-Blodget coatings for nanolithography as well as in stamp fabrication for soft nanolithography.
Synthesis of ZnO nanorods for photonic crystals in integrated nano-optics and nanopiezotronics for e.g. energy harvesting.
Use of protein receptors in selective photonic and nanocantilever-type sensing with the prospect of single molecule detection.
In all of these nanotechnology-related research efforts the strong multidisciplinary character, and the application driven character is obvious.

9:30 Oral

Novel Nanocomposite Biosensor for Potassium Ion Sweat Test

Pelagia I. Gouma, Ruipeng Xue

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Potassium ions (K^+) level in the human body is critical for the electrolyte balance of the body and the nerves and muscles functions. So it is very important to accurately and rapidly determine potassium concentrations in human fluids in clinical fields. This paper describes a novel concept for a potassium ion biosensor that accurately, rapidly, and efficiently monitors the presence and records the concentration of potassium ions with high specificity, not only in serum and urine, but also in the sweat or even eye fluid. This specific biosensor design utilizes a nanomanufacturing technique, i.e. electrospinning, to produce advanced nano-bio-composites that specifically trace even minute quantities of potassium ions through the use of selective bio-receptors (ionophores) attached to high surface area nanofibers. Electroactive polymers are then employed as transducers to produce an electronic (rather than ionic) output that changes instantly with the change in K^+ concentration. A convenient, non-invasive, maintenance-free "Band-aid" type configuration of skin patch is demonstrated in this paper.

9:50 Oral

Comparison of single and stacked sensing membrane for pH-ISFET

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The first ion sensitive field effect transistor (ISFET) was with SiO_2 layer as the sensing membrane. Next, to improve sensing performance, Si_3N_4 on a SiO_2 layer was applied. Some stacked metal oxide layers were also investigated. However, for the high-k technology, the intermediate oxide layer is not necessary.
In this study, to compare the sensing properties between single and stacked sensing membranes, Si_3N_4, Ta_2O_5, and TaON layers were deposited on silicon substrates with and without an intermediate oxide layer. For the fabrication process, Si_3N_4 layer was deposited by low pressure chemical vapor deposition (LPCVD); Ta_2O_5 and TaON layers were fabricated by reactive rf sputtering with Ta-target in different gases ambient. Besides, a post deposition annealing (PDA) was used on Ta_2O_5 layer. Before testing of the sensing properties of ISFETs, the electrical parameters of metal insulator field effect transistors (MISFETs) were measured. The on/off current ratio for all samples was higher than 10^6, and all MISFETs with single insulator showed the lower body effect than the stacked one. The sensing properties of single and stacked layer ISFETs were similar. The sensitivity of ISFETs with Ta based gate materials was higher than 50 mV/pH and around 47 mV/pH for Si_3N_4

ISFETs. The low drift coefficients, around 1 mV/h, were observed for all samples. Base on these results, it can be state that the single layer sensing membrane is suitable for ISFET application. The advantages are the following: a simple fabrication process, low cost, and high compatibility with high-k technology.

10:10 Oral

Hydrothermal synthesis of nanocrystalline zeolite materials for application in protein immobilization

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The immobilization of biologically active molecules is an important goal in fine chemical, pharmaceutical and food industries as well as in clinical and chemical analysis because it can offer several advantages including molecule reuse, ease product separation, improvement in stability of the immobilized molecule. Protein-surface interactions are fundamental for the assembly of interfacial protein constructs, such as advanced functional materials, activators and sensors. In biomaterials biochemistry, a basic understanding of protein immobilization on new biomaterials like zeolites may facilitate the design of biocompatible structures and devices, since protein adsorption from body fluids is the first stage that can promote or inhibit the biocompatibility of implanted devices.
Zeolites consist of a three-dimensional arrangement of SiO4 and AlO4 tetrahedra linked to each other by a shared oxygen atom prepared by hydrothermal syntheses. The resulting framework has a system of channels and cavities of molecular dimensions where are cations which can be extracted by ion exchange. Their low toxicity and high biocompatibility make zeolites attractive candidates for new biomaterials for medical applications. The possibility to varied

and modulated acid-base, hydrophobic-hydrophilic and selective adsorption proprieties of these materials are very interesting to study the potential interactions with important biological species.
In this study, new zeolitic nanocrystals and composite membranes by hydrothermal syntheses using different reaction gels to modulate the Brönsted acidity of the microporous structures were synthesized in a fluoride and hydroxide medium and applied to the protein adsorption. In addition, to modulate the surface acidity of the MFI structure, aluminum and vanadium atoms were incorporated into the crystalline framework and the ratios of Si/Al, Si/V, and Al/V were varied by changing the chemical composition of the precursor reaction gels.

COFFEE

Wednesday morning, 16 September, 10:30
Main Hall

Lectures

Chemical and biosensing
Wednesday morning, 16 September, 11:00
Room 208
Chair: Dekhtar, Yuri

11:00 Invited oral

Metal oxide nanowires chemical sensors for security application

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Increasing concern on health hazard due to pollution or terrorist attacks encouraged an increasing research effort on gas sensing for real-time monitoring of all aspects of indoor and outdoor environments. Industrial requirements for a sensor are high sensitivity, high selectivity and good stability, together with low fabrication costs.

Among all possible technological approaches, conductometric gas sensors based on metal oxide semiconductors are the most promising for the development of low cost and reliable sensors. It has been confirmed that the sensitivity of metal oxide is improved as the crystallite size is decreased down to nanometer scale, thanks to the increased surface to volume ratio and the grains carriers' depletion. Unfortunately polycrystalline metal oxides suffer from grain coalescence induced by the high operating temperatures required to enhance chemical reactions. This affects sensor stability over long-term operation. On the contrary single crystalline nanosized metal oxide are stable materials and have the dimensions and surface to volume ratio necessary to obtain high chemical sensitivity. The better stoichiometry and greater crystallinity degree compared to polycrystalline oxides make these materials very promising for a better understanding of sensing principles and the development of a new generation of chemical sensors.

After the first publication demonstrating the capability of nanowires to detect gaseous species, huge research activity was carried out as demonstrated by the high number of publications and conferences on this topic. The challenge is the integration into sensing platforms necessary for the exploitation in the sensor market. Concerning gas sensing application metal oxide nanowires demonstrate performace suited to detect chemical warfare agents (CWAs) simulants at concentrations close or lower than the toxicity values of the real CWAs.

11:30 Oral

The Influence of a Heysler Ferromagnetic Alloy Film on the Spread of Surface Acoustic Waves

Michael M. Karpuk[1], Dmitriy A. Kostiuk[2], Yuri A. Kuzavko[3], Vladimir G. Shavrov[4]

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Spread of surface acoustic waves (SAW) of Rayleigh and SH-modes is examined on the surface medium of (100) and (110) orientations of the cubic crystal (Si, AsGa, etc.) covered with thin (much thinner than SAW wavelength) film of Heysler ferromagnetic alloy (HFA) with shape memory (SM). A 2-partial Rayleigh SAW spreads on the clear (100) cut of a crystal in a sagittal section and a volumetric transverse wave, a SH-mode, also does. A generalized 3-partial Rayleigh SAW spreads on a (110) cut at arbitrary wave vector **k** orientation, and only at **k** || [110] and **k** || [111] there are spoken above SAW.

Film coating with HFA with SM wasn't previously examined from the point of view of SAW spread. Let us note that longitudinal wave (LA) speeds in volumetric Ni_2MnGa HFA are v_l=5540 m/s, fast transverse (TA_1) v_{1t}=3560 m/s and slow one (TA_2) v_{2t}=740 m/s at spread along the **k** || [110] axis with **e** || [1T0] polarization. Measured speed changes at Ni_2MnGa PT are about Δv_l □1.5%, Δv_{1t} □8%, and Δv_{2t} □40% respectfully and under external magnetic field at HFA saturation they are Δ_{vl} □0.2%, Δv_{1t} □0.3% and Δv_{2t} □1%. We have measured 6% v_l change in polycrystalline $Ni_{2.14}Mn_{0.81}Fe_{0.05}Ga$ sample (T_{MA}=40°C) at change from paramagnetic phase to a low-temperature martensitic one. $Ni_{53}Mn_{25}Ga_{22}$ of 3.83x4.06x9.93 mm^3 with combined magnetostructural PT (T_{MA}=105°C) also had 6% at such PT. At HFA film thickness of 0.01 wavelengths there is maximum sensitivity of 0.001° temperature, 1 Oe magnetic field and 1 Pa pressure measurements at 1 Hz stability of the device operation.

11:50 Oral

Electropolymerization of methylene green on gold and platinum electrodes for ascorbic acid determination

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In this paper, results concerning application of a phenothiazine dye - methylene green (MG) as a sensing membrane for amperometric sensors are presented. The MG can act as an electron transfer mediator and reduce the oxidation overpotential of some biologically important compounds.

The MG was electropolymerized on gold and platinum electrodes. The three electrode electrochemical cell consisted of working electrode (WE) of 3.75 mm^2 area, a reference electrode - saturated calomel electrode and a counter one - platinum rod. The electropolymerization was carried out in 1 mM MG/0.1 M KCl solution using a cyclic voltammetry technique: 30 sweeps in potential range from -0.6 V to +1.4 V with scan rate 100 mV/s.
Obtained thin films of poly(methylene green) (PMG) were compared using scanning electron microscopy. It was found that on platinum WE very thin, non-uniform and untight layers were deposited. Electrochemical tests by cyclic voltammetry in potential range from -0.8 V to +0.8 V, starting and finishing at 0.0 V, with scan rate 100 mV/s in 0.1 M KCl solution shown voltammograms of different shape, stability and reproducibility, depending on type of the bare working electrode material used.

Finally, the gold and platinum electrodes modified with PMG were used for the determination of ascorbic acid in concentration range from 1 mM to 20 mM. In case of the platinum WE modified with PMG, the sensitivity to ascorbic acid become worse, while in the case of the gold electrodes sensitivity increased and a reduction peak has appeared.

Summarizing, covering the gold electrode with PMG film makes the electrode sensitive to ascorbic acid. Such modified electrodes can be used in immunoassays with antybodies labelled with alkaline phosphate which uses ascorbic acid phosphate as a substrate. The future research will be oriented towards increase the selectivity to ascorbic acid.

This research is financially supported by Ministry of Science and Higher Education (Grant No. N N518 0926 33).

12:10 Oral

The applicative conjugated copolymers for Sensorics: Synthesis and Electrochemistry

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Conjugated copolymers coordinate the action of a large number of optically active absorbing units with delocalized electronic structures that allow for electronic coupling between optoelectronic segments and efficient energy transfer. Important properties of conjugated polymers such as charge transport, conductivity, emission intensity, and excitation migration, are easily perturbed by external agents, leading to substantional changes in measurable signals. Careful incorporation of functional groups into conjugated polymer yield new materials that possess beneficial properties for even biosensor application.

Among π-conjugated polymers, those containing benzene-fused heteroaromatic rings with nitrogen atom(s) are class of materials that has shown interesting electrical and optical properties. Some compounds containing both, electron donor and acceptor moieties exhibit the intermolecular charge transfer absorbance and fluorescence behavior. In such a donor – acceptor molecules (D-A), very large changes in charge distribution can be induced in the excited state upon absorption of light photons. Therefore, recently these organic D-A compounds have been increasing interest and usually been used as the fluorescent probe to study microenvironments.

Various π-conjugated copolymers of 2,1,3-benzothiadiazole with thiophene, pyrrole, fluorene units have been developed. The introduction of benzothiadiazole into conjugated polyfluorene backbone improved the electron – transporting ability and the electroluminescence efficiency of polymer. Although thianthrenes in general have attracted strong interest due to the fascinating physical and chemical properties of numerous sophisticated derivatives. The electron – rich, heterocyclic thianthrene seems to be a good example for charge transporting materials, since it shows a reversible oxidation behavior at low potential in cyclic voltammetry

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(maps available at the Reception desk)
Wednesday afternoon, 16 September, 12:30
building of the Faculty of Physics

Lectures

Security
Wednesday afternoon, 16 September, 14:00
Room 208
Chair: Llobet, Eduard

14:00 Invited oral

Ultra Sensitive Metal Oxide Gas Sensing Elements

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The structure, functionality and sensing response of metal oxide films will be reviewed with emphasis on In and Zn nanostructures as prepared by Sputtering, PLD, and Spray pyrolysis techniques. Optical, structural and electrical characterization techniques as applied for the in-depth analysis of the film response will be elaborated. Sensing response towards gases such as Ozone, NO_2 and H_2 will be presented utilizing conductometric and Surface Acoustic Waves (SAW) techniques at a range of temperatures and frequencies. It will be shown that sensing responses of the order of a few ppb (6ppb for ozone) at room temperature may be obtained by appropriate control of the film nanostructure.

14:30 Oral

Acousto-electronic Intellectual Sensors Based on Surface Waves

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Conditions are examined of generation, spread and registration of Rayleigh surface acoustic waves (SAW) on a massive substrate of the high Q-factor acoustic crystal, covered with a thin film of the Heysler ferromagnetic alloy with shape memory and combined magneto-structural phase transition (PT) – $Ni_{53}Mn_{25}Ga_{22}$, $Ni_{2.18}Mn_{0.82}Ga$ and $Ni_{2.14}Mn_{0.81}Fe_{0.05}Ga$ with PT at room temperature. SAW are generated and received by interlocking-finger transducer (IFT), mounted on the *yz*-cut of lithium niobate Li_2O_3. 350 mm plates of Si covered with thin 5 µm Ni-Mn-Ga film coating, both of (100) orientation, are placed on these IFT. So experiments were carried out along with the bridge circuit. A 35 Mhz sensor was in the positive frequency feedback with the main high stable oscillator. Placing ferromagnetic film on the silicon substrate substantially increases the selectivity, resolution and sensitivity of thew sensor to hydrocarbons concentration in the air. The amount of admixtures in the air was registered on frequency shift of the setting generator and is about 1 ppm/sm^2.

Chemical substances sensible to some of the gas mixture components been placed on the sensor surface provide a sort of the 'electronic nose' device. Such devices based on chemical sensors are intended to substitute taste and smell receptors of humans. Electronic nose can be successfully used to control the air quality, gas and liquid wastes of industry and agriculture, and for other ecological tasks. Other usage area includes earlier fire alarm systems, determining the volatile burning products, and systems of automated manufacture safety control.

14:50 Oral

Mixed Composition Nanowires Prepared By Electrodeposition

Beata Kalska-Szostko[1], Ewa Brancewicz, Krzysztof Szymański, Wojciech Olszewski[2], Piotr Mazalski

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In last two decades the intensive growth of research in area of nano scale materials takes place. Among many procedures by which the nanomaterials can be obtained electrochemistry is the cheapest and easiest one. This method has no limitation for sample shape and size. Besides that almost any material can be electrochemically deposited.

In our work the preparation of magnetic nanowires with mixed composition in porous matrix will be presented. The obtained structures can have different diameter which is tunable by template material. Length and the inert structure of the wires depends mainly on time of the deposition and electrolyte composition, respectively. The morphology of the prepared wires depends also on deposition mode (alternating current or constant current).

The smallest wires of the diameter 40 nm were prepared in porous anodic alumina oxide anodized in oxalic acid. The largest pores of around 120 nm were produced in phosphorous acid. The length of this nanowires can be as large as the thickness of oxide layer and reach up to 1 µm. The morphology of wires were studied by atomic force microscopy (AFM) and electron scanning microscopy (SEM). The magnetic characterization was done with usage of magnetic force microscopy (MFM) and Mössbauer spectroscopy. The wires show different deposition rate in matrices prepared in oxalic and phosphorous acid. The deposition rate depends also on the elements. The wires show magnetization along their growth direction.

Work is supported by Polish National Grant No N N204 246435

COFFEE

Wednesday afternoon, 16 September, 15:30
Main Hall

Joint Poster Session

Monday & Wednesday
Wednesday afternoon, 16 September, 16:00
Main Hall

Thursday, 17 September

Lectures

Biomaterials
Thursday morning, 17 September, 9:00
Room 208
Chair: Swieszkowski Wojciech

9:00 Invited oral

Bioactive materials as carriers of cells for tissue regeneration

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Artificial and nature-derived materials, such as polymers, metals, ceramics, carbon-based materials and composites of these matrices, are widely used in various biotechnologies, including the construction of replacements of damaged tissues. For creation of advanced tissue replacements, the artificial materials should not be only passively tolerated by cells, but they should promote specific cell responses in a controllable manner. It is generally known that the cell-material interaction is strongly dependent on physical and chemical properties of the material surface, such as its polarity, wettability, roughness and topography, electrical charge and conductivity, rigidity or elasticity, presence of specific chemical functional groups or biomolecules etc. In our studies, we often concentrated on substrates

with roughness and topography in nanoscale, namely materials coated with nanocrystalline diamond films, nanocomposite hydrocarbon plasma polymer films with various concentrations of Ti, gold nanoparticle films, or composites of synthetic polymers with carbon nanotubes or ceramic nanoparticles (hydroxyapatite or tricalciumphosphate). The materials promoted the adhesion, growth and osteogenic differentiation of human bone-derived cells *in vitro*, represented by osteoblast-like MG 63 cells. A possible explanation is a favourable effect of the substrate nanostructure, which promotes the adsorption of cell adhesion-mediating extracellular matrix molecules (e.g., vitronectin, fibronectin, collagen, laminin) in an appropriate spectrum.

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9:30 Oral

Development of in situ ellipsometric methodology and immobilization of proteins in liquid cells

Andrea Németh[1], Tímea Hülber[1], Péter Kozma[1,2], Sándor Kurunczi[1], Peter Petrik[1], Ferenc Vonderviszt[1,2], Csaba Hős[3], Miklos Fried[1], István Bársony[1]

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The in situ measurement of adsorption and immobilization of proteins in liquid cells is a complex task influenced by numerous parameters. Most of these factors (like temperature of the cell, purity of water, rate of injection) are known to have little effect on the repeatability of the measurement (Castro et al., Surf. Sci. 601 [2007] 1795). On the other hand, there are various possible causes of uncertainty that need to be investigated. As an improvement of our standard procedure for protein immobilization (Kozma et al., phys. stat. sol. 5 (2008) 1427), we studied the influence of different substrate materials, including virgin and chemically activated thermal SiO2 and Ta2O5 thin films on Si wafer, in the aspect of the properties of protein adsorption (kinetics, surface coverage and the stability of the deposited material against detergent flow) by ellipsometry and atomic force microscopy. The adsorption of flagellar filaments was investigated using two-layer effective medium models that accounted for the vertical density distribution of the filaments with lengths around 500-1000 nm bound to the surface. We performed measurements also on suprasil/Ta2O5 structures to directly compare the results of ellipsometry with that of optical waveguide lightmode spectroscopy. We also investigated flow cells of different constructions mainly focusing on the capacity. We designed a flow cell with significantly smaller volume for economic measurements and for a better understanding of protein adsorption. The development of the flow cell was supported by finite element flow dynamics calculations. We also made attempts to improve our standard procedure for immobilization with a modified surface treatment, as well as to measure receptor attachment.

9:50 Oral

Optimization of a novel HfTaO membrane for pH sensing and applied to food industry

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Ion sensitive field effect transistor (ISFET) has been developed for more than 30 years. It shows high potential to replace the conventional pH glass electrode, but there are still some drawbacks need to be improved. Therefore, many high-k materials have been investigated to be a perfect sensing membrane. In this paper, a binary metal oxide layer, HfTaO, was first proposed to be the sensing membrane of electrolyte insulator semiconductor (EIS) structure. The HfTaO layer was deposited by reactive rf sputtering with a binary metal target (Hf:Ta = 1:1). Afterwards, a post rapid thermal annealing (RTA) was used at 500, 700, 900°C in O_2 ambient for 1 min. For the evaluation of sensing properties, the pH sensitivity and light immunity were improved with higher RTA temperature.
It could be explained through the further surface oxidation observed from the higher intensity of O_{1s} by X-ray photoelectron spectroscopy (XPS).

For the cleaning-in-process (CIP) test, the HfTaO layer was optimized to 20 cycles at 700°C but degraded to 2 cycles at 900°C. According to the X-ray diffraction (XRD) spectrum, it started to crystallize at 900°C. It results the formation of grain boundary as the weak point to be etched by NaOH and HNO_3 solution. To check the HfTaO-EIS annealed at 700°C after 20 CIP cycles, the surface morphology was observed by optical microscope, and the pH sensitivity was changed from 48.4 mV/pH to 52.0 mV/pH. A slight increase of capacitance could be come from the etching mechanism to decreas the thickness of HfTaO layer. Finally, the HfTaO layer with 700°C RTA is concluded as the best condition of sensing membrane for food industry.

10:10 Oral

New hydroxyl free radical sensor based on coumarin 3 carboxylic acid intercalated into layered double hydroxide hydrotalcite

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The hydroxyl radical is the most reactive oxygen species (ROS), presenting two problems for the construction of sensors: (a) inability to penetrate significantly into any matrix without being destroyed; (b) ability to oxidize (and photobleach) most potential reference dyes (1). Here we present the synthesis and properties of an inorganic-organic hybrid material that overcomes the above two basic problems and serves as an effective hydroxyl free radical sensor. A hydroxyl free radical sensing moiety (2) coumarin-3-carboxylic acid (3CCA), was intercalated into the layered double hydroxide (HTLC) framework using one-pot hydrothermal synthesis (3).

The obtained layered inorganic/organic hybrid material with empirical formula: $[M(II)_{0.65}M(III)_{0.35}(OH)_2](X)_{0.35}.n\ H_2O]$, where M(II) = ($Zn^{2+}$), M(III) = ($Al^{3+}$ with Eu^{3+} substituting the Al^{3+} sites), and X = anionic 3CCA, along with having lanthanide (Eu^{3+}) ion substitution into the layered structure, enhances the luminescence decay lifetime thus enabling easier temporal variation of the luminescence measurements (4). The ZnAlEu(3CCA) HTLC materials with M(II)/M(III) ≈ 2.0 was synthesized and further characterized by XRPD/FTIR/TGA/SEM-EDX/SS-NMR techniques along with solid-state luminescence measurements. The generation of hydroxyl free radical from the aqueous phase reaction of copper chloride and Vitamin C (Ascorbic acid) has already been well studied (5) and henceforth used as a probe for our work.

The synthesized hybrid sensor material is promising because of its observed luminescence in the visible region and high-temperature resistance as indicated by the XRPD measurements and TGA/DTG analysis moreover, the two basic problems in the development of hydroxyl free radical sensor should be overcome.

COFFEE

Thursday morning, 17 September, 10:30
Main Hall

Lectures

Biomaterials
Thursday morning, 17 September, 11:00
Room 208
Chair: Balazsi, Csaba

11:00 Oral

3D characterization of composites, soft tissue, smart materials to sensors with a novel lab based multiscale x-ray nanotomography system.

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Advanced ceramics, composites, biomaterials, polymers, smart materials and sensors are currently being developed for a variety of applications in aerospace, medicine, defense and alternative energy systems. Accurate 3D analysis of their micro and nanostructures, porosity, defects and chemistry are crucial to predict and correlate mechanical properties to reliability, performance and the design of next generation materials. While conventional imaging modalities such as optical microscopy, SEM with FIB, AFMs have good resolution, they are limited to surface imaging in 2D and require destructive sample preparation to reveal buried structures. Existing non destructive techniques such as X-ray/Neutron radiography, acoustic, eddy current techniques lack the resolution for micro and nano structural characterization. Resolution of conventional microCT or nanoCT (micro or nano x-ray computed tomography) are limited to the spot size of the x-ray source, sample size and sample-source working distance. Besides conventional CT systems often lack the contrast to satisfactorily image soft materials, biological tissue or polymer blends. We describe a suite of novel lab-based X-ray CT for 3D imaging of materials across lengthscales and resolutions. Samples with dimension from several cm to microscopic sizes may be imaged at resolution from mm to sub 50 nm. Multilengthscale 3D characterization of composites, polymer blends with flame retardants, high temperature ceramics, biomaterial-soft tissue interaction, biochip and electronic sensors will be illustrated with a focus on porosity, material phase distribution, coating thickness, bonding/joint integrity and cracks/defects after fabrication, stress, impact or thermal mechanical tests. Comparison of results from the novel lab based CT with ceramography, serial sectioning, SEM and synchrotron x-ray tomography will also be discussed

11:20 Oral

Molecular-genetic criteria of efficacy of the use of bioactive nanomaterials in the field of traumatology and orthopaedy.

Kirill A. Nechaev

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Introduction: traumatism and orthopaedic diseases cause huge damage to modern society. Annually more than 12 million men get hurt. More than 10 million men suffer from orthopaedic diseases. The diseases of bone tissue are originally different pathologic conditions, which become apparent with bone structure disturbance. The integral index of bone tissue metabolic activity is processes of active remodeling and renovation of bone structures, which continue the whole life. On the one hand, these processes are an important mechanism of maintenance of mineral homeostasis; on the other hand, they provide structural bone adaptation to changing conditions of functioning. The process of bone tissue remodeling is a mechanism directed on preservation of homeostasis, having a high sensitivity to different regulatory, supervisory mechanisms, as well as endogenous and exogenous influences. The most actual direction nowadays is detection and investigation of the main pathogenetic component of bone tissue pathology (both hereditary induced and multifactorial pathology) and search for ways of leading pathologic sign correction based hereon.

From theoretical point of view, the relevance of the work in this dir-

ection is determined with necessity to get fundamentally new concepts of aetiology and pathogenesis of musculoskeletal systemdiseases, as a complex multifactorial phenotype of individual. At practical perspectives, importance of investigation is determined with a necessity of development and acquisition of new medical materials for treatment of complex orthopaedic pathology.

LUNCH

(maps available at the Reception desk)
Thursday afternoon, 17 September, 12:30
building of the Faculty of Physics

Posters

Monday, 14 September

Joint Poster Session

Monday & Wednesday
Monday afternoon, 14 September, 16:00
Main Hall

16:00	Poster	F-1

Instant Glucose Sensing Using the AD5933 Impedance Converter

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Diabetes is a metabolic disorder, which results from insulin deficiency and hyperglycemia and is reflected by blood glucose concentrations being outside the normal range of 80-120 mg/dL (4.4-6.6 mM). The novel system is presented that utilises change in the value of impedance as an instant indicator of the glucose level of the solution, brought in contact with a sensor. The glucose sensor was developed using the enzyme glucose oxidase (GOx) immobilized within the poly(o-Phenylenediamine) (PPD). O-Phenylenediamine (o-PD) was prepared in an acetate buffer solution with pH5 and then mixed with GOx. The resultant GOx/o-PD mixture was deposited onto Au thick film interdigitated electrodes and electrochemical immobilization of the enzyme was performed using the bulk electrolysis with coulmetry technique at 3 V for 900 s.

Through a catalytic enzymatic reaction between glucose and glucose oxidase the biosensor generates hydrogen peroxide and its response therefore is based on the electrochemical oxidation of H_2O_2 [1]. The electrochemical formation of polymer films enables to make a glucose sensor with a controlled thickness, which is reproducible and could be miniaturized.

The sensor was connected to the Analog devices AD5933 impedance converter system, which measured a change in impedance when different glucose concentrations were deposited onto the sensor within 1-7 mM glucose range.

The well-manifested change in the value of capacitances is directly related to the concentration of the glucose in the solution being tested. Moreover, the response of the sensors to the glucose change was in the order of 10 s, similarly to [2].

[1] S. Cosnier et al, *J. Electroanalytical Chemistry* 469(2), pp. 176-181, 1999.
[2] O. Korostynska et al, Proc. IEEE SAS-2009, pp. 34-37.

16:00	Poster	F-2

Investigation of hysteresis dependent sensitivity on n and p type Si_3N_4-EIS structures

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A differential system on chemical sensors, such as reference field effect transistor (REFET) was proposed as the solution to replace the conventional reference electrode. However, the drawbacks as the complicated fabrication process and short lifetime of organic REFET are still existed. Therefore, an electrolyte insulator semiconductor (EIS) structure with single Si_3N_4 sensing membrane and p-type substrate was proposed to be a possibility for inorganic REFET application. In this paper, the electrical and sensing properties of n-type and p-type EIS with single Si_3N_4 sensing membrane was investigated. The pH sensitivity was measured from accumulation region to inversion region, a low sensitivity as 23.75 mV/pH was shown on p-EIS, but high as 46.37 mV/pH on n-EIS.
However, the sensitivities of both structures were high and similar by measuring from an opposite direction (around 46 mV/pH, from inversion to accumulation). To investigate the different sensitivity between these two structures, hysteresis measurement for typical metal oxide silicon capacitor (MOSCAP) was used. A positive voltage shift was performed on p-EIS, but only a slight negative voltage shift was observed for n-EIS. The voltage shift could be caused by the charging and discharging behavior during bias sweep. For the further study, the hysteresis was measured in different pH buffer solutions. In both type of substrate, the hysteresis was increased with pH value, but the hysteresis is much higher on p-EIS than on n-EIS. Based on the result, the hysteresis of p-EIS could be also the explanation of the large variation of pH sensitivity between different bias sweep directions.

The difference of the hysteresis dependent sensitivity on different substrate will be useful on the application of ISFET/REFET pair. Besides, this mechanism can also be used to explain the sensing properties on other high-k membranes which performed a large hysteresis.

16:00 Poster F-3

The influence of the tannic and gallic acid on the size and monodispersity of synthesized gold and silver nanoparticles

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Metal particles in the nanometer size range have gained considerable interest in recent years. Nanoparticles possess unique optical, electrical, and mechanical properties, which are strongly dependent on the size and shape of the particle. Antibacterial properties of gold and silver nanoparticles have potential applications in the pharmaceutical and cosmetic industries and also for the diagnostics of the biological probes.

Tannic and gallic acids posses antibacterial activity, in this way they increase bioactivity of Au and Ag nanoparticles. These acids act as reducing and stabilizing agents, which ensures high monodispersity of colloids.

We describe a simple method to synthesize silver and gold nanoparticles by chemical reduction. Soluble metal salts ($AgNO_3$, HAuCl4) were used in the particle synthesis. Sodium citrate, tannic and gallic acids were used as reducing and stabilizing agents. The resultant nanoproducts were characterized by Dynamic Light Scattering (DLS) and Atomic Force Microscopy (AFM). We report the preparation of silver nanoparticles in the range of
4–50 nm and gold nanoparticles in the range from 10nm to 100 nm. Synthesized nanoparticles are very stable and monodisperse. Tannic and gallic acids were found to be excellent additives to control the nanoparticles sizes.
This work was partially supported by MNiSW grant nr N N507 350435.

This work was partially supported by grant from Iceland, Liechtenstein
and Norway through the EEA Financial Mechanism and Foundation for
Polish Science - Homing Program.

16:00 Poster F-4

Effect of the increment of a polymeric chain coating over Fe_3O_4 particles used for magnetic separation

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The development of magnetic separation techniques using small magnetic particles has been of great attention for biomedical applications. Such applications involve strict requirements on particles characteristics such as: chemical composition, size and size distribution, crystalline structure, stability of magnetic properties, surface morphology, adsorption properties and low toxicity, among others. The effect that an increased polymeric chain has over this type of application has been studied, however, in only few investigations. Based on this fact, spherical magnetite nanoparticles with size of 200 nm were synthesized by chemical coprecipitation under refluxing and aging conditions, being then coated and functionalized with different types of polymer chains onto particle surface. In this research, silica was the minor polymeric chain used, while aminosilane was the other polymeric material involved. In order to increase the chain length, silica coating was applied followed by a second aminosilane shell. Protein adsorption on magnetite nanoparticles coated with polymeric chains was studied. The protein adhesion for magnetite-silica, magnetite-aminosilane and magnetite-silica-aminosilane arrays was 12.5%, 79.5% and 145.75% higher than pure magnetite, respectively. The result shows a dependence of the protein adsorption with the increased chain. The magnetic properties were slightly modified, having that the saturation magnetization decreased with the application of the coating; however all coated magnetite nanoparticles showed a superparamagnetic behavior translated into a fast response to an external magnetic field applied.

X-Ray Diffraction (XRD) was used to confirm the magnetite (Fe_3O_4) phase. The particle size distribution and morphology of nanoparticles with and without coateing were characterized by Field Emission Scanning Electron Microscope (FE-SEM).

16:00 Poster F-5

Polyurethane-based material with calcite and aragonite for tissue engineering composite scaffolds

Ida O. Dulińska-Molak[1], Joanna Ryszkowska, Piotr Woźniak[2], Małgorzata Lewandowska-Szumieł[2], Krzysztof J. Kurzydlowski[1]

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The tissue engineering scaffolds act as a substrate for cells, and their subsequent growth and proliferation. The basic requirements for the scaffold material are biocompatibility, degradability and osteoconductivity. This paper reports that such requirements are met by novel biodegradable scaffolds made of poly (e-caprolactone) urethane (PCL_PUR) porous matrix and calcium carbonate $CaCO_3$ (calcite and aragonite) developed for bone tissue engineering.

The polyurethanes were prepared by in situ polymerization. Porous structure of the scaffolds was obtained by the salt leaching/polymer coagulation method, using NaCl with the grain size 300-450 um as a porogen. The structures of composite materials were observed by scanning electron microscopy SEM and analysed by micro-CT. The mechanical properties of PUR/$CaCO_3$ composites were evaluated in static and dynamic compression test. The thermal properties of composites were investigated using Thermal Gravimetric Analysis (TGA) and Differential Scanning Calorimeter (DSC).

In vitro studies in simulated body fluid (SBF) were performed to study the influence of calcium carbonate ($CaCO_3$) on a bioactivity. Calcium phosphates formation in the form of the apatite layer on PUR/$CaCO_3$ composites was investigated byX-ray Photoemission Spectroscopy (XPS), Scanning Electron Microscope (SEM) equipped with Energy-Dispersive Spectrometer (EDS) and Fourier transformed infrared spectroscopy-attenuated total reflectance (FTIR-ATR). The structure composites materials was observed by scanning electron microscopy SEM and analysed by micro-CT.

Acknowledgements:The authors thank Dr Zbigniew Jaegermann from Institute of Glass and Ceramics for supplying us calcium carbonate. This scientific work was funded from the finances for education in the years 2006-2009 as research project no. R1301901

16:00 Poster F-6

Investigation of the DLC and novel DLC-polymer hybrid coatings using FIB and SEM techniques

Emilia Choińska[1], Piotr Wieciński[1], Łukasz Ciupiński[1], Wojciech Swieszkowski[1], Veli M. Tiainen[2], Krzysztof J. Kurzydlowski[1]

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The aim of the present study was to characterize the cross-section of the diamond-like carbon (DLC) and novel DLC-polymer hybrid (DLC-p-h) nanocomposite coatings for medical application. These investigations were done to measure the coatings thickness, analyse the distributions of the DLC and polymer in the coatings and observe the interface between substrate and coatings.

The DLC and DLC- polymer hybrid coatings were deposited on the stainless steel (ANSI 316L) substrate using filtered pulsed arc discharge (FPAD) method. Two different DLC-polymer hybrid coatings have been studied – DLC-polytetrafluoroethylene-h (DLC-PTFE-h) and DLC-polydimethylosiloxane-h (DLC-PDMS-h). The cross-section of the coatings for the SEM and STEM observation have been prepared using the focused ion beam (FIB) Hitachi FB2100 by milling i.e removal bulk material. Because the coatings are made of the polymer, the low energy of the ion beam was used to prepare cross-sections views. The observations of the prepared samples were performed using the HR scanning electron microscope Hitachi S-5500. 3 different modes, such as secondary electrons (SE), backscattered electrons (BSE) and scanning transmission electrons STEM were used to investigate the cross-sections.

The results of microscopic investigations show that the distribution of the constituents in the hybrid coatings depends on the used polymer. In the cross-section of the DLC-PTFE-h coating two separate layers were observed: deeper one - DLC and PTFE at the surface, while DLC-PDMS-h coating was composed of alternating thin sublayers of presumable different phase volume fractions of polymer and DLC, forming a multilayer coating. The STEM observation of the DLC coating indicate amorphous character of this material, what was confirmed by X-Ray diffraction (XRD) results.

16:00 Poster F-7

Investigation of nano hydroxyapatite prepared from eggshell and seashell

Gréta Gergely[1], Ferenc Wéber[1], István E. Lukács[1], Katalin Balázsi[1], Levente Illés[1], Attila L. Tóth[1], Judith Mihály[2], Csaba Balazsi[1]

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Bioceramics fulfil an unique function as biomedical materials. The development of biomaterials and manufacturing techniques has broadened the diversity of applications within the human body. Hydroxyapatite (HAP) is chemically similar to the mineral component of bones and hard tissues in mammals. It is one of few materials that are classed as bioactive, meaning that it will support bone ingrowth and osseointegration when used in orthopaedic, dental and maxillofacial applications. Coatings of hydroxyapatite are often applied to metallic implants (most commonly titanium/titanium alloys and stainless steels) to alter the surface properties. Hydroxyapatite may be employed in forms such as powders, porous blocks and hybrid composites to fill bone defects or voids. These may arise when large sections of bone have had to be removed (e.g. bone cancers) or when bone augmentations are required (e.g maxillofacial reconstructions or dental applications). In this work, hydroxyapatite were successfully produced by using recycled eggshell, seashell and phosphoric acid. The observed phases on the powder synthesis process were dependent on the mixing ratio (wt.%) of the calcined eggshell and phosphoric acid, and the calcination temperature. Elaboration processes of the HAP has been developed and compared of the HAPs from different raw materials.The structures of the HAP were characterized by X-ray diffraction and scanning electron micropscopy. Samples were pressed from the HAP powder, and the three and four point beinding strengt was determined.

16:00 Poster F-8

Non-cytotoxic small Carbon Nano-Onions - the first covalent functionalization with biomolecules

Marta E. Płońska-Brzezińska[1], Joanna Łuszczyn[1], Amit Palkar[2], Alina Dubis[1], Agneta Simionescu[2], Dan T. Siomionescu[2], Beata Kalska-Szostko[1], Luis Echegoyen[2]

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Application of *small* (6 to 8 shells) carbon nano-onions modified with the carboxylate groups (ox-CNOs) in biosensor construction were performed. Main attention is paid to study the ability of modified CNOs to interact with other functionalized biologically active compounds.

Carbon nano-onions were prepared and functionalized with carboxylic group by chemical oxidation and by pegylation using method applied by Lieber et al. for surface modification of SWNTs.[1] These nanostructures after functionalization become soluble in aqueous solutions. MTS test showed excellent cytocompatibility of all CNOs tested at 30 and 3000 µg/ml. Therefore these carbon nanostructures can be safely used for intended biological applications. For the first time covalent functionalization of ox-CNOs with biomolecules has been reported. It was used biotin-avidin interaction. The oxidized CNOs particles were immobilized by amidation reaction on Au surface.

Multilayers were prepared on gold surface by layer-by-layer assembly method and the process was monitored by surface plasmon resonance (SPR) and atomic force microscopy (AFM). The Fourier transform infrared spectroscopy with total attenuated reflactance mode (FT-IR/HATR) were also used to characterize the multilayer formation. The FT-IR spectroscopy confirmed covalent linkage between interacting molecules.

In conclusion, functionalized *small* CNOs (5-6nm) can be potentially used for developing chemical sensors and biosensors.

[1] Wong, S.; Woolley, A.; Joselevich, E.; Cheung, C.; Lieber, C. *J. Am.Chem. Soc.***1998**, *120*, 8557

16:00	Poster	F-9

Preliminary studies on degradation of polylactides fibers

Maryla Moczulska[1,3], Wacław Tomaszewski[2], Wojciech Swieszkowski[1], Arie Bruinink[3], Krzysztof J. Kurzydlowski[1]

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In vitro degradation study is essential step in the characterization of a tissue engineering products. The degradation rate of the tissue engineering products should be similar or slower than the rate of tissue repair. Products of degradation should be non toxic and no harmful to the body. They should be also easily secrete through normal physiological pathways. Many various agents can influence on degradation process (polymer molecular weight, polymer polydispersity, polymer crystallinity, pH of degradation medium, degradation condition: static or dynamic and also morphology and porosity). Design of scaffold can also influence on degradation process.

There are many strategies which are used to study scaffold properties during in vitro degradation. Some of them use as a buffer solution a phosphate buffered saline (PBS) to maintain a stable pH and perform the assay at 37°C. The scaffolds are immersed in solution for a certain period. Various parameter such as weight loss, polymer molecular weight and mechanical properties are monitored.

The aim of this work was preliminary studies of degradation of fibrous material produced from degradable polylactide (PLA). Fibers were fabricated via electrospinning. These material was tested using PBS as immersing liquid. Degree of degradation of the materials was estimated on the basis of weight loss. Microstructure of the materials was observed using scanning electron microscope. Molecular mass changes were evaluated by gel permeation chromatography (GPC).

The results obtained in this study indicate that the incubation for a period 18 weeks in PBS caused the weight loss and decrease molecular mass. During incubation in PBS the fibers diameter do not change. It suggest that the fibers degrade through bulk hydrolysis.

16:00	Poster	F-10

Micro Sensor for cell force measurement

Wojciech Swieszkowski[1], Waldemar T. Smolik[1], Norbert Danz[2], Erik Forester[2], Jean-Pierre Kaiser[3], Arie Bruinink[3], Krzysztof J. Kurzydlowski[1]

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Cell elasticity, motility and migration are very important for understanding various biological processes. Currently, monitoring cellular traction forces is desired as new marker to monitor cell functionality. We developed a sensor system for cell force measurement based upon a polymer microchip covered with a micro pillar matrix. The spring constant is chosen to enable pillar bending by forces in the range of 10..100 nN. The pillars bend in response to cellular forces that are transmitted via cellular focal adhesion points. The micro chip is read out optically. Besides a transmission image to analyze pillar bending, two different fluorescence images of dye markers can be taken sequentially. Matrix with the cells is illuminated bys spatially homogenized LED radiation. The focal position during long term experiments can be maintained by means of a piezo stage in order to obtain highly resolved images from the CCD imager. Sophisticated software controls the optical system and performs image registration. First, one transmission (red) as well as two fluorescence images (blue & green) are taken using appropriate CCD gain and integration times. Fluorescence information yields those pillar positions that are at least covered by a cell. The transmission image is used to calculate the magnitude and direction of pillars deflection. Therefore, detection of single pillar position and its deviation from a hexagonal grid is analyzed automatically, enabling a mapping of traction forces.In summary, this system containing transducer polymer chip, optics, and dedicated software analysis is a complete imaging system for image registration, single cell visualization and cellular force measurement. First results of cellular force measurements obtained are presented.

Symposium G

Materials for membranes in energy applications

Welcome

The classical definition of membranes says: Thin skin for different purposes especially for letting something through and other things not.

The symposium cares about membrane materials in energy applications. It especially views the preferred or impeded transport of atoms, molecules, ions, electrons, photons and phonons through a thin freestanding or substrate supported functional layer with selectively acting properties and their combinations. Application areas are e.g. fuel cells and batteries where either pure ionic conductors combined with electronic conductors, or even mixed ionic/electronic conductors, electrically isolating membranes etc. are needed. Thermoelectric converters look for highly electrical and low thermal conducting membranes, photovoltaic's use light translucent membranes. Fossil power plants ask for thermal barrier membranes with high temperature stability and sensoric/optical properties, air separation membranes or even gas separation membranes for selective CO_2-permeation.

The photo catalytic production of hydrogen asks for combined membranes acting catalytically as well as proton conductive.

All these different membranes and their materials should be considered in one symposium to put together the existing knowledge, find out new solutions and discuss the strategies of realising membrane devices for future energy systems.

Organisers

Detlev Stöver

Forschungszentrum Jülich GmbH

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Programme

Monday, 14 September

Opening & Plenary Session

Czochralski Award Ceremony
Monday morning, 14 September, 10:30
Small Hall (237)

LUNCH

(maps available at the Reception desk)
Monday afternoon, 14 September, 12:30
building of the Faculty of Physics

Poster Session
Monday afternoon, 14 September, 16:00
Main Hall

Tuesday, 15 September

Oxygen Mixed Conductor Membrane Materials I
Tuesday morning, 15 September, 9:00
Room 206
Chair: Prof. Dr. Harald Bolt

9:00	Invited oral

Determination of oxygen chemical transport parameters of MIEC materials from electrochemical impedance spectroscopy

Ellen Ivers-Tiffée

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Several mixed ionic-electronic conducting (MIEC) materials, e.g. (La,Sr)(Co,Fe)-based perovskites, exhibit excellent oxygen transport properties, namely fast chemical surface exchange and oxygen bulk diffusion, thus rendering them suitable candidates for applications such as fuel cell catalysts and oxygen permeation membranes. As the reduction of CO_2 emissions in coal or gas power plants is increasingly gaining attention, particularly membranes for oxygen separation based on such MIEC perovskite materials show promise for a future large-scale application in power plants.

In this presentation, several material systems are assessed with regard to their surface exchange kinetics and oxygen ion bulk diffusion properties. Porous perovskite layers with the chemical compositions $La_{0.68}Sr_{0.3}FeO_{3-\delta}$ and $La_{0.68}Sr_{0.3}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (two solid oxide fuel cell (SOFC) cathode compositions) are characterized by electrochemical impedance spectroscopy (EIS). The chemical surface (k^d) and diffusion (D^d) coefficients of both materials are obtained as a function of temperature and pO_2 directly from EIS measurements performed on full anode-supported cells. This is achieved by a combined distribution of relaxation times and equivalent circuit impedance analysis method, which allows a highly resolved identification and deconvolution of each single polarization mechanisms contributing to the overall loss of the cell. This approach can also be extended to further MIEC materials, such as the state-of-the-art membrane perovskite $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$.

9:30 Oral

Oxygen permeation through asymmetric perovskitic membranes

Michael Betz, Stefan Baumann, Wilhelm A. Meulenberg, Detlev Stoever

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The necessity of reducing CO_2-emissions is evident and getting more and more social consensus all over the world. Therefore, carbon capture and storage (CCS) is an important option in order to achieve CO_2 mitigation. One of three options for CCS is making use of oxyfuel technology, where pure oxygen is used to combust a fuel (e.g. coal, gas, biogas) forming primarily CO_2 and H_2O making it much easier and cheaper to capture the CO_2 than by using air in the combustion. The required oxygen can be produced using ceramic oxygen transport membranes (OTM), which are associated with significantly lower efficiency losses compared to conventional separation technologies. OTM consist of gastight mixed ionic electronic conductors (MIEC), which allow oxygen diffusion through vacancies in the crystal lattice and simultaneous transport of electrons in the opposite direction at elevated temperatures, e.g. 800-900°C. Their major advantage is infinite oxygen selectivity, assuming no leakage through the membrane layer or the sealing. Highest oxygen permeation can be obtained using perovskite materials (ABO_3) such as $(Ba,Sr)(Co,Fe)O_3$ or $(La,Sr)(Co,Fe)O_3$. In order to further increase permeation rates, asymmetric thin film membranes with activated surfaces are necessary. The preparation of asymmetric multilayered membrane assemblies is presented. Furthermore, characterization results, e.g. SEM-micrographs, permeation data, are shown, and challenges and prospects are derived. It is shown that permeation rates increase using asymmetric membrane, but the increase is relatively low due to surface exchange kinetics and gas diffusion through porous support.

9:45 Oral

Sintering of $Ba_{0,5}Sr_{0,5}Co_{0,8}Fe_{0,2}O_{3-\varphi}$ Perovskite, the (Grain) Boundary Between Functional and Mechanical Properties?

Markus Zwick, Ewald M. Pfaff, Christoph Broeckmann

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Perovskite structured materials with mixed ionic/electronic conductivity (MIEC) can be used as oxygen transport membrane (OTM) for generating pure oxygen, for syngas processes or oxyfuel combustion. A lot of investigations are dealing only with the functional properties. But for using perovskite components in industrial applications also the thermo-mechanical properties have to be taken into consideration carefully. Because both functional and mechanical properties are depending on microstructure a compromise has to be found.

In the following work the influence of microstructure on the functional and mechanical properties of $Ba_{0,5}Sr_{0,5}Co_{0,8}Fe_{0,2}O_{3-\varphi}$, a material with highest oxygen permeation rate, was analysed.

To verify microstructure of poly crystalline ceramics you have to understand the sintering mechanisms. The sintering mechanisms depending on temperature were analysed and special sintering curves were designed to create different microstructures. Isostatic pressed tubes were sintered using these different sintering curves. Polished and thermal etched samples were analysed by SEM concerning grain size distribution and porosity. The influence on mechanical properties was compared. Oxygen permeation at 800 °C was determined and correlated to the mechanical properties to find the best compromise between functional properties, mechanical properties and microstructure.
The results of this work are used in the German project OXYCOAL-AC with focus on emission free coal combustion.

10:00 Oral

Fabrication and oxygen permeation of asymmetric dual-phase composite membranes

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Asymmetric dual-phase membranes for oxygen permeation, $Ce_{0.85}Sm_{0.15}O_{1.925}/Sm_{0.6}Sr_{0.4}FeO_{3-d}$ (SDC-SSF), were fabricated by an acid-leaching technique, i.e., a thin dense layer which consists of SDC-SSF was remained by controlling acid-leaching degree, and a porous substrate which consists of $Ce_{0.85}Sm_{0.15}O_{1,925}$ (SDC) with fluorite structure was formed with dissolution of $Sm_{0.6}Sr_{0.4}FeO_{3-d}$(SSF) with perovskite structure in HCl. The acid-leaching method can readily adjust the thickness of thin dense layer and porosity of supporting substrate. Meanwhile, traditional shrinkage mismatch and chemical reaction between thin dense layers and porous substrates can be easily avoided. The oxygen permeation experiments showed that the as-prepared asymmetric membranes possessed higher oxygen permeation flux.

10:15 Oral

Electrical and Oxygen Transport Properties of $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$

Mónica Burriel[1,2], Christian Niedrig[1,2], Stefan Baumann[3], Stefan F. Wagner[1,2], Ellen Ivers-Tiffée[1,2]

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In the last years, several mixed ionic-electronic conducting (MIEC) materials, such as $Sr(Co,Fe)O_3$-based perovskites, have attracted increasing interest owing to their excellent ionic and electronic transport properties. In particular, $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ (BSCF) shows

a high oxygen permeation flux, thus making it a promising candidate for intermediate temperature solid oxide fuel cell cathodes and dense ceramic oxygen permeation membranes. However, several issues, such as its long-term behaviour (stability) as a function of oxygen partial pressure, temperature or in the presence of other gas components (e.g., CO_2), are still subject of investigation.

In the present study, polycrystalline BSCF as well as related perovskite compositions of interest for similar applications (e.g., $La_{0.58}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF), $Pr_{0.58}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (PSCF)) were investigated as bulk samples in an oxygen pump setup with respect to the conductivity behaviour as a function of oxygen partial pressure and temperature. Moreover, surface exchange (k^{δ}) and bulk diffusion (D^{δ}) coefficients have been determined from measurements on thick film and thin film samples by means of further electro-chemical techniques (impedance spectroscopy, conductivity relaxation in the frequency domain) and are compared with values taken from a literature survey.

COFFEE

Tuesday morning, 15 September, 10:30
Main Hall

Oxygen Mixed Conductor Membrane Materials II

Tuesday morning, 15 September, 11:00
Room 206
Chair: Prof. Dr. Harald Bolt

11:00 Oral

The Effect of Cation Inter-diffusion on Oxygen Exchange Properties of CGO-LSCF Composites

John W. Druce, John A. Kilner

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Oxyfuelled Carbon Capture and Storage (CCS) is one means of achieving the necessary reductions in the emission of carbon dioxide from fossil fuel-fired power plants. In this approach, a hydrocarbon fuel is burned in pure oxygen, yielding only carbon dioxide and steam as combustion products. Since the steam may be easily condensed out of the exhaust stream, the CO_2 can be easily separated for subsequent sequestration in, for example, geological storage sites.

Clearly, one of the import requirements for implementing this is providing an economical and reliable source of pure oxygen. Mixed electronic – oxygen ionic conducting ceramic membranes can fulfil both of these criteria. However, single phase ceramics do not offer the required chemical and mechanical stability for this application, and so it is thought that composite materials could provide the necessary performance.

The oxygen exchange properties of the pure ionic conductor-mixed electronic conductor $Ce_{0.9}Gd_{0.1}O_{1.95}$-$La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ system were investigated by the Isotope Exchange Diffusion Profiling method. The "macroscopic" (integrating over many grains of each phase) properties were measured with a Duoplasmatron source-based Atomika 6500 Secondary Ion Mass Spectrometer (SIMS), and compared with "microscopic" measurements from within single grains of each phase, obtained using a Gallium liquid metal ion source Focussed Ion Beam (FIB)-SIMS instrument.

Although the variation in the diffusion (D) and surface exchange (k) coefficients measured on the macro-scale appears relatively straightforward, the microscopic oxygen exchange behaviour is more interesting. The surface ^{18}O isotopic fraction measured on grains of the CGO phase is higher than expected from literature values, suggesting the presence of LSCF enhances oxygen exchange in these materials. This will be discussed in terms of the interdiffusion of cations between the two phases on sintering, which has been indicated by both XRD and SEM-EDX.

11:15 Oral

The Role of Gas-Solid Interfaces in Oxygen Permeable Dense Ceramic Membranes

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Mixed oxygen-ion and electron conducting dense ceramic membranes are now on their way to integrated chemical and power plant applications that require a continuous feed of pure oxygen or oxygen-enriched gases. However, the variety of potential membrane applications represent a large span of process conditions a membrane has to meet. In an Oxy-Fuel power plant process, the membrane would be subjected to a rather small gradient of oxygen partial pressure, whereas in a chemical process such as membrane-assisted syngas production the membrane would be exposed to an extremely large partial pressure difference. However, there are a few requirements that are valid for all these applications. Among the most important requirements is that the membrane must be optimized towards a maximum permeation flux. This may be achieved by improving the transport properties of the membrane material and/or by optimizing the membrane geometry. In this paper, a case study of the perovskite-based membrane material Ba(Co,Fe,Zr)O3 [1] will be presented. We have determined the transport properties of this material for a wide range of parameters (oxygen partial pressure, temperature, membrane thickness and membrane geometry). A defect chemical and transport model was applied to this experimental data set, which provides us with an excellent description of the permeation properties of Ba(Co,Fe,Zr)O3 membranes with planar and tubular geometries. The modelling results highlight the important role of oxygen exchange at the membrane surfaces where oxygen is transferred from the gas to the solid phase and vice versa. Furthermore, under certain conditions, the oxygen permeation flux is strongly affected by the membrane microstructure.

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Catal. Today, 104 (2005) 126.

11:30 Oral

Permeation Studies and Chemical Stability of MIEC Membranes for highly efficient Air Separation in Oxy-fuel Power Plant Processes

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To reduce the CO2 emissions of fossil fuel fired power plants by separating CO2 out of the process is one important step to migitate the greenhouse effect. One discussed process is to concentrate carbon dioxide in the power plant process by coal combustion using oxygen instead of air. If the required air separation is realised by a MIEC membrane, it will be possible to reduce the efficiency drop of the power plant. For development of membrane moduls for industrial applications it is necessary to study membrane materials under comparable conditions to the power plant. To reach high enough O2-permeation rates membranes have to be as thin as possible and they have to be mechanically and chemically stable under the operating conditions (T > 800°C, ΔP = 20 bar). Furthermore the MIEC membrane has to withstand all contacting materials and gases without decomposition of the membrane material. Therefore experimental studies are made under various thermal, mechanical and chemical conditions. Main topic is to test the influence of flue gas components CO2, SO2, CO, NO and water each of variable concentrations on the permeation rate, the membrane long time stability and the changings of the material microstructure. The influence of flue gas components on perovskite and non-perovskite types Ba0,5Sr0,5Co0,8Fe0,2O3-d(BSCF), Sr0,5 Ca0,5 Mn0,8 Fe0,2 O3-d (SCMF) and La2NiO4-d has been studied with the result that some types have been shown higher chemical stability combined with much lower permeation rate. In addition the permeation rate as a function of membrane thickness and O2-partial and absolut pressure has been investigated. The permeate pressure has been varied from 40 mbar to 1 bar combined with high feed pressures up to 20 bar at the same time. Permeation rates about 10 mlN/cm2min at temperatures about 900°C have been measured. These experimental results combined with a CFD-based simulation of the experiments provide a basis to fit the materials constants of the wagner-equation and to estimate limitations by surface kinetics.

11:45 Oral

Stability and oxygen transport properties of $BaCo_{0.8-x}Fe_xNb_{0.2}O_{3-\delta}$ perovskite membranes in CO_2-containing atmosphere

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Dense perovskite-type ceramic membranes made from mixed oxygen ionic-electronic conductors (MIEC) have received much attention due to the high selectivity for oxygen separation. Using an MIEC membrane system for the pure oxygen supply benefits an oxy-fuel power plant with a significant increase in the efficiency, compared with the conventional route by cryogenic air separation. For such application, membranes with sufficient long-term stability under the desired working conditions, that is, in atmospheres containing aggressive gases like CO_2 and H_2O etc., and at temperatures typically above 700 °C, are crucial. Some perovskite-type membranes with high oxygen permeability have recently been shown to be susceptible to the presence of these aggressive gases.

In order to better understand how the presence of CO_2 affects the performance of the perovskite oxides and to explore strategy for development of CO_2-resistant materials, $BaCo_{0.8-x}Fe_xNb_{0.2}O_{3-\delta}$ (x=0.2-0.8) perovskite membranes were prepared and their oxygen permeability and stability in CO_2 were investigated. The oxygen permeation fluxes were measured with pure CO_2 as sweep gas and compared with those measured with Ar. Degradation in the oxygen fluxes was observed when CO_2 was used. The stability of the materials was also examined by a series of CO_2 annealing experiments under various conditions and the results of the (micro-)structural analyses for the annealed samples will be presented. The behaviour of the membranes in CO_2 is linked with the B-site cation composition of the perovskites.

12:00 Oral

Module Design for MIEC Membranes in Energy Applications

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In world wide efforts processes are in development for coal or gas combustion with an additional capturing of CO_2 exhaust gas for storage in the underground. Most efficiency is expected by burning in a nitrogen free atmosphere. The oxygen needed for the combustion shall be delivered by so called Oxygen Transport Membranes (OTM) in the combustion process. Perovskite type ceramics with mixed ionic and electronic conductivity at high temperatures can be used as membrane materials.

With improving the oxygen ion conductivity other relevant material properties became critical in particular chemical stability and mechanical strength. The ceramic membranes must be joint gas tight to metal housings and work at 800 – 1000 °C. Concentrating on tubular membranes material behaviour of BSCF-Perovskite will be described as basis for optimization joining techniques and module design. Experimental data for Reactive Air Brazing, Glass soldering and gluing in cold surrounding will be presented and discussed as well as different module concepts.

12:15 Oral

Study of highly oxygen deficient perovskite cathodes

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The critical factor in increasing solid oxide fuel cells (SOFC) reliability and decreasing operating costs is lowering the operating temperature below 800 ^{o}C. Materials with the greatest prospects for application at these temperatures are transition metal perovskites because of a wide range of unique physical and thermal properties, stability against CO poisoning, no need for expensive catalysts, and possibility of use with variety of fuels. Currently used cathode materials such as $La_{1-x}Sr_xMnO_3$ (LSM, x=0.1–0.4) with purely electronic conductivity are limiting the performance. Mixed ionic and electronic conducting cathodes are preferred for utilization because of an increased reaction zone of the three-phase boundary. Superior oxygen ion conductivity requires large amount of hoping sites such as oxygen vacancies. By using x-ray and neutron powder diffraction, thermogravimetric (TGA), conductivity, dilatometery, and area specific resistance (ASR) measurements we have recently studied several highly oxygen deficient perovskites, LSM (x>0.5) [1,2], $SrMn_{1-y}Fe_yO_z$ (SMF), and $La_{1-x}Sr_xFe_{1-y}Co_yO_z$ (LSFC, x>0.5)[3] to explore their potential as SOFC cathodes.

Polycrystalline samples were synthesized from stoichiometric mixtures of La_2O_3, $SrCO_3$, MnO_2, Fe_2O_3, and Co_3O_4. Single-phase perovskite SFM and LSFC were obtained in air, while LSM required special treatment in Ar followed by anneal in air. [4] Oxygen content under various atmospheres was studied with TGA apparatus (Cahn TG171). TGA was also used to obtain samples with desired oxygen content by "in-situ" anneal. Structural details, including oxygen content and ordering were investigated by NPD at the Intense Pulsed Neutron Source at ANL. High-resolution data were collected in situ by varying temperature, time, and oxygen partial pressure, and analyzed using the Rietveld method and the GSAS code. X-ray powder diffraction (Rigaku D/MAX) was used to determine the phases and structure types present after long-term annealing in air at 600, 800, and 1000 ^{o}C. ASR performance was studied using electrochemical impedance spectroscopy and dc polarization curves.

LSM samples with higher Sr content exhibit increasing range of oxygen deficiency, high ionic and electronic conductivity, and good chemical stability with YSZ. ASR measurements showed that compositions x=0.6–0.8 exhibit 2-3 times lower values than the standard LSM materials. While samples with x=0.9-1 decompose in air above 800 ^{o}C, all compositions show good thermal stability against decomposition at low oxygen pressures. [5] Three oxygen-vacancy ordered phases with z=2.71, 2.6, and 2.5, detrimental for both oxygen permeation and electrical conductivity, were found for x=0.6-1 up to 1100 ^{o}C. [6] The LSFC samples exhibit very high oxygen non-stoichiometry, 2.25≤z≤3.00 even at moderately reducing conditions, and excellent ionic and electronic conductivity. However, they show poor stability with YSZ and decomposition at low oxygen pressures. In addition, we observed several LSFC oxygen-vacancy ordered phases at z=2.875, 2.5, and 2.25+x/2, some of them stable to 800 ^{o}C. The SMF samples showed intermediate properties between LSM and LSFC; no oxygen vacancy ordered phases were observed to date. We have determined the compositions of LSM, SMF, and LSFC and their thermal stability ranges that exhibit only the favorable vacancy-disordered phases. Unfortunately, all these compositions exhibit very large thermal ((17-26)10^{-6}/K) and oxygen-chemical (0.024-0.030/mole) expansion coefficients not compatible with the known electrolytes.

Several new perovskite compositions satisfy quite a few requirements for low temperature SOFC cathode materials such as: fast oxygen-ion and high electronic conductivity, structural stability, chemical compatibility with YSZ electrolyte, and improved performance over the present materials. However, frequent presence of oxygen ordered phases at enhanced vacancy content and the a large thermal chemical expansion coefficients may limit their applicability. Requirements of an enhanced vacancy content for high ionic conductivity and the mixed-valency of transition metals for high electronic conductivity go up against the small expansion coefficients. Clever materials design of new compounds is required to overcome these impediments.

Acknowledgments

This work was supported by the NSF-DMR-0706610 and U.S. DOE-BES DE-AC02-06CH11357.

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[4] B. Dabrowski, et al., Acta Physica Polonica A 105, 45 (2004)

[5] L. Suescun, et al., JSSCh 180, 1698 (2007)

[6] L. Suescun, et al., Chem. Mater. 20, 1636 (2008)

LUNCH

(maps available at the Reception desk)
Tuesday afternoon, 15 September, 12:30
building of the Faculty of Physics

Hydrogen Mixed Conductor Membrane Materials

Tuesday afternoon, 15 September, 14:00
Room 206
Chair: Dr. Jose M. Serra

14:00 Oral

Ceramic Hydrogen-Permeable Membranes made of Mixed Proton-Electronic Conducting Materials based on the system Ln_6WO_{12}

Jose M. Serra[1], Sonia Escolastico[1], Maria Balaguer[1], Wilhelm A. Meulenberg[2], Hans Peter Buchkremer[2], Tobias Scherb[3], Gerhard Schumacher[3], Marck W. Lumey[4], Richard Dronskowski[4]

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The tungstates Ln_6WO_{12} are proton conducting crystalline materials exhibiting sufficient mixed conductivity (protonic and electronic) and remarkable stability to consider them as potential candidates for the separation of hydrogen at high temperature. Membranes made of this type of materials can be applied for industrial gas separations such as hydrogen separation from hydrogen containing gases, for the implementation of pre-combustion strategies in power plants, and for the application in ceramic proton conducting fuel cells. This contribution summarizes the recent developments achieved on ceramic hydrogen-permeable membranes in the frame of the MEM-BRAIN Alliance. It is shown the preparation of nanocrystalline mixed conducting materials based in the system Ln_6WO_{12} using a sol-gel complexation synthesis method. This method is based on the citrate-complexation route modified to stabilize W- and Ln-containing ions. The influence of the lanthanide (Ln) nature and the partial substitution in the system $Ln_{6-x}A_xWO_{12}$ (Ln= La and Nd; A= Ce, Eu, Gd, La, Nd, Pr, Sm, Tb, Yb; and x =1 and 3) on the total conductivity and crystalline structure was studied systematically. The electrical conductivity measurements were conducted by standard four-point DC technique on the sintered rectangular bars and silver paste and wire were used for contacting. The measurements were carried out under different atmospheres, i.e., helium and H_2/D_2 saturated with H_2O/D_2O at 20ºC. For selected samples, it was studied the water uptake process followed by thermogravimetry, XRD and Raman spectroscopy. Hydrogen permeation was studied for different disk-shaped Ln_6WO_{12} membranes in the range of 700-1000ºC. The membrane sides were coated by screen printing with a 20 μm layer of a Pt ink aiming to improve surface hydrogen exchange. Furthermore, stability of these materials at 700 and 800ºC has been evaluated in a CO_2-rich gas stream, dry and saturated with water at room temperature. Materials remained unchanged after the treatments, irrespective of its original sintering temperature and lanthanide nature. The experimental work has been supported by *ab-initio* calculations aiming to give more insight into the fundamentals of protonic conduction through the crystalline lattice.

14:15 Oral

Hydrogen in rutile TiO_2

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Rutile TiO_2 is a wide-band gap metal oxide semiconductor with a variety of established and potential applications involving its electrical, optical and catalytic properties. The point defect chemistry governing its properties has been extensively studied since the 1960s, and has recently been reviewed by Nowotny *et al.*[1]. However, hydrogen defects, such as protons are often neglected. In this study, we utilize *ab initio* DFT calculations and molecular dynamics (MD) to elaborate on the behaviour of hydrogen defects in rutile TiO_2, and predict the relative dominance of protons and oxygen vacancies as positive defects.

All calculations were carried out using DFT as implemented in VASP, with GGA-PBE as the exchange-correlation functional and electron-ions interactions described by the PAW method with a cut-off energy of 500 eV. The supercells used for defect calculations consisted of 3×3×3 primitive unit cells, while a 2×2×3 supercell was used for MD simulations.

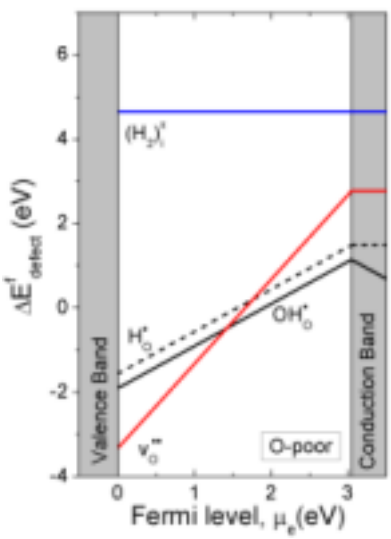
The defect calculations show that OH_O^q, H_O^q and v_O^q form shallow acceptor levels (Fig. 1a). Of the three hydrogen defects, protonic defects have the lowest formation energy, even at low oxygen activities, and will thus be the dominating species. However, the formation energy of $H_O^{\bullet}$ is only about 0.5 eV higher, and may thus form in small quantities. From the formation energy of $OH_O^{\bullet}$ and $v_O^{\bullet\bullet}$, we have determined the enthalpy of hydration of oxygen vacancies:

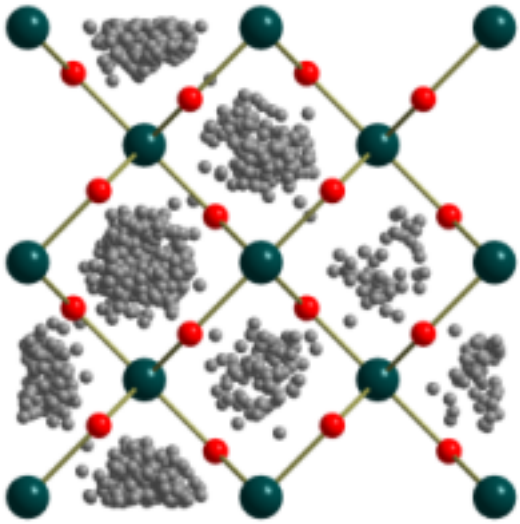
$$H_2O(g) + O_O^{\times} + v_O^{\bullet\bullet} = 2OH_O^{\bullet}$$

to ΔH_{Hydr} = -1.57 eV. As such, rutile TiO_2 can be expected to be dominated by protons at elevated temperatures, even under relatively dry conditions.

A MD simulation in the NVT ensemble at 2000K (Fig.1b and c) shows an anisotropic proton diffusion in rutile, with diffusion along the c-axis dominating over diffusion in the a-b-plane, in good agreement with experiments [2, 3].

(a)





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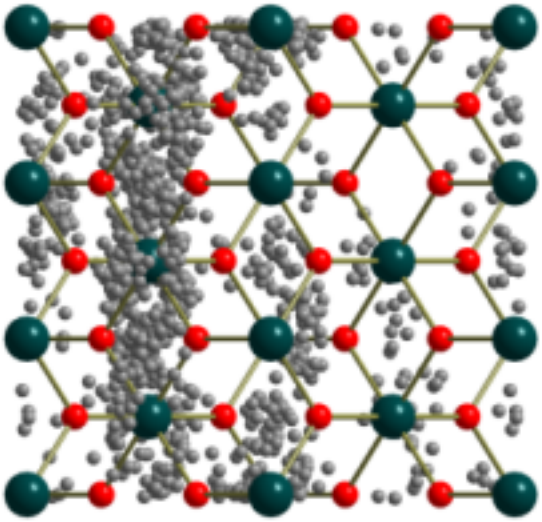

(c)

Figure 1:*(a) Defect formation energies as a function of Fermi level, (b) and (c) Proton positions extracted from a MD simulation of 50 ps at 2000K plotted on a static lattice, viewed along the c- and b-axis, respectively.*

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14:30 Oral

Enhanced sintering of cobalt or nickel doped solid solution of barium cerate and zirconate

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High Temperature Proton Conductors (HTPCs) can be used as electrolytes in a Protonic Ceramic Fuel Cell (PCFC) or, in case of a mixed ionic and electronic conductivity, as membranes for hydrogen separation.

Solid solutions of barium cerate and zirconate have been widely studied because they represent a compromise between the relatively high proton conductivity of cerate and the chemical/mechanical stability of zirconate [1-2]. But the very high sintering temperatures required limit their use from an economic but also practical point of view. Indeed, in case of anode supported cells, potential electrode materials can't resist these high temperatures. Doping with transition metals has been shown to promote sintering, for example, a 400°C decrease of the sintering temperature for Zn doped barium zirconate [3-4].

In this study, nickel and cobalt doping (from 1 to 5% of the B-site) is studied with two compositions: $BaZr_{0.9}Y_{0.1}O_{3-\delta}$ (BZY10) and $BaCe_{0.2}Zr_{0.7}Y_{0.1}O_{3-\delta}$ (BCZY27). The powders were synthesized by a solid state reaction at 1400°C and pellets sintered in air at temperature between 1350°C and 1450°C. Density percentages of about 95% could have been achieved for doped samples, compared to only 80% for the undoped ones. The conductivity of the samples has been determined using impedance spectroscopy. Measurement of the conductivity isotope effect by exchange with heavy water vapour has been used to identify the protonic component of the ionic conductivity.

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14:45 Oral

Raman evaluation of the proton diffusion coefficient in the pressure protonated perovskite electrolytes

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The design of electrolyser for working under moderate water pressure[1] offers new impetus in the study of perovskite ceramics as membranes of the green house gas free devices such as the fuel cells and water electrolysers. We have recently pointed out that the protonation process of perovskite ceramic performed in the autoclave (high temperature, high pressure of H_2O) gives rise to a broad "Raman" component, centred at ~2500cm^{-1} correlated to the protonic species content and the conductivity of a material[2]. The so-called *Raman profilometry* method which permits to map and control the homogeneity of the proton distribution on ceramic section is discussed. The Raman intensity distribution profile is fitted according to Fick's diffusion laws in order to compare the proton diffusion

coefficient at various temperatures and water pressure for different perovskites ((Ba/Sr)(Zr/Ti)O_3).

1. PCT patent WO 2008/152317 A2 (18-12-2008), *Method for optimising the conductivity provided by the displacement of H^+ protons and/or OH- ions in a conductive membrane.*

2. A. Slodczyk, Ph. Colomban, S. Willemin, O. Lacroix, B. Sala, *Indirect Raman identification of the proton insertion in the high-temperature [BA/Sr][Zr/Ti]O_3 modified perovskite protonic conductor*, J. Raman Spectroscopy, 39 (2008) DOI 10.1002/jrs.2157

15:00 Oral

Proton diffusion in perovskites by Elastic and Quasi-Elastic Neutron Scattering measurement

Aneta Slodczyk[1,2], Philippe Colomban[1], Daniel Lamago[3], Olivier Lacroix[2,4], Stephanie Willemin[2,4], Beatrice Sala[2,4]

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Thanks to their relative low functioning temperature the proton conducting perovskite ceramics are the most promising electrolytes for fuel cell and electrolyser applications[1]. Prior to the better industrial application it is necessary to understand the nature, diffusion and conduction mechanism of the protonic species. Neutron scattering experiments are very well suited to study the proton content, structure and dynamics of the proton conductors due to the very large cross-section of the hydrogen element. Elastic/quasi-elastic neutron studies were performed as the function of high temperature and momentum transfer for protonated Ln-modified $BaZrO_3$ using the three axis spectrometers: 1T1 and 4F1 at the Leon Brillouin Laboratory (Saclay, France). Simultaneously, the neutron diffraction patterns were recorded permitting to follow the structural modifications. The results show the presence of the protonic species in the dense ceramic, their successive motion from 400 to 600°C and then the departure above 750°C under high vacuum conditions[2]. The QNS results are compared with the conductivity and thermal expansion measurements.

1. PCT patent WO 2008/152317 A2 (18-12-2008), Method for optimising the conductivity provided by the displacement of H^+ protons and/or OH^- ions in a conductive membrane.

2. A. Slodczyk, Ph. Colomban, D. Lamago, MH. Limage, F. Romain, S. Willemin, B. Sala *Phase transitions in the H^+ -conducting perovskite ceramics by the quasi-elastic neutron and high-pressure Raman scattering,* **Ionics** 14 (2008) 215

15:15 Oral

High pressure phase transitions in proton-conductor $SrZrO_3$–based perovskites

Aneta Slodczyk[1,2], Marie-Helene Limage[1], Philippe Colomban[1], Stephanie Willemin[2,3], Beatrice Sala[2,3]

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The water electrolysis operating at temperature below 700°C is recently considered as one of the most efficient and economical methods to obtain the hydrogen – future energetic carrier[1,2]. Prior to the better industrial application it is crucial to well understand the modifications of the host perovskite behaviour induced by the protonic species[3]. Therefore, the high pressure Raman study of pure, Ln-modified protonated and non-protonated $SrZrO_3$ dense ceramics were performed between 0.1 and 40 GPa using a Diamond Anvil Cell (DAC). The comparison of the Raman spectra shows important differences in the pressure behaviour between the pure and Ln-modified $SrZrO_3$ ceramics. Simultaneously, rather similar sequence of phase transition (orthorhombic Pnma –10 GPa– > pseudo-tetragonal Imma –20 GPa– > tetragonal I4/mcm -35 GPa- > cubic Pm3m) was revealed in the case of Ln-modified samples, both protonated and non-protonated. Note that the lack of abrupt structural differences between protonated and non-protonated samples is very important from the application point of view because preserves the mechanical strength and the long life time. The main differences between the protonated and non-protonated ceramics concern the behaviour of Zr-O stretching mode of the Zr-O_{6-x} octahedron perturbated by neighbouring oxygen vacancies and Ln ions. The high pressure Raman results are compared with the Raman data as the function of high temperature as well as the diffraction and the thermal expansion ones.

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2. PCT patent WO 2008/152317 A2 (18-12-2008).

3. A. Slodczyk, Ph. Colomban, D. Lamago, MH. Limage, F. Romain, S. Willemin, B. Sala *Phase transitions in the H^+ -conducting perovskite ceramics by the quasi-elastic neutron and high-pressure Raman scattering,* Ionics 14 (2008) 215.

COFFEE

Tuesday afternoon, 15 September, 15:30
Main Hall

Membrane Materials for various Applications

Tuesday afternoon, 15 September, 16:00
Room 206
Chair: Dr. Jose M. Serra

16:00 Oral

Materials and processing for high performance solid oxide fuel cells with long-term durability

Robert Mücke, Norbert H. Menzler, Sven Uhlenbruck, Hans Peter Buchkremer, Detlev Stoever

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Solid oxide fuel cells (SOFC) were enhanced at Forschungszentrum Jülich the last five years to yield higher performance and better long-term stability. This development includes all facets of material science: the materials themselves (e.g. Fe perovskite cathodes); the processing of applied layers in terms of their thickness, paste/suspension parameters, and sintering regime; the adaptation of microstructure (porosity, particle sizes); and the combination of different material interfaces in the cell and the stack. The newest generation of cells yield approx. 900 mW/cm² at an operating temperature as low as 700°C (H_2/3%H_2O, air, 0.7 V) in single cell tests. In a stack the performance is still approx. 700 mW/cm² at the same operating conditions, whereas the slight decrease is owed to losses in the cell-interconnect interface.
As there does not exist an unified cell design for all currently investigated applications, three different types of cells are used at Forschungszentrum Jülich: (1) for stationary applications, (2) a cell with high-performance cathode, (3) and a light-weight cell with better redox stability in mobile applications (e.g. auxiliary power units). The differences in these types are presented and specific material problems addressed, e.g. diffusion barrier layers against undesired phase reactions. Furthermore, state-of-the-art fabrication routes are shown that are capable of being scaled-up for industry production with their specific advantages and characteristics.

16:15 Oral

Lanthanum silicate thin films for application as electrolyte of SOFCs

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Nowadays, Solid Oxide Fuel Cells (SOFCs) are seen as a future alternative energy generation source. The commercial SOFCs have been operated at 1000ºC to insure a sufficient ionic conductivity of their yttria-stabilized zirconia (YSZ) electrolyte. However, the high working temperature causes serious problems in terms of materials degradation and lifetime. There is therefore a lot of interest in develop new solid electrolyte materials for lower temperatures applications. Recently, apatite-type lanthanum silicates of general formula La10-x(SiO4)6O3-1,5x have gained considerable attention because they exhibit high ionic conductivity and low activation energies at low temperatures besides they allow a wide range of substitutional possibilities [2, 3]. One key problem is the high processing temperature necessary to prepare them [4].

The aim of this contribution is the production of apatite-like lanthanum silicates thin films by magnetron sputtering. Thin films with the appropriate La/Si atomic ratios were deposited by reactive magnetron sputtering from LaSi and Si targets and subsequently annealed in controlled atmosphere to obtain the targeted lanthanum silicate oxide. The influence of the adding of germanium in the production of lanthanum germanosilicate thin films was also investigated.

The Chemical composition of the coatings was determined by electron probe analysis (EPMA). The structure was studied by X-ray diffraction (XRD) using a Phillips diffractometer operated in Bragg-Brentano configuration with Co (Ka) radiation. The cross section and surface topography of the La-Si films were examined on a JEOL scanning electron microscope (SEM) equipped with an EDAX energy dispersive spectrometer (EDS). The electrical properties of the films were measured by AC impedance spectroscopy (HP4284A precision LCR meter, 20 H z – 1 MHz).

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16:30 Oral

Plasmon enhanced Dye-Sensitized Solar Cells by using Atomic Layer Deposition of TiO2 on gold/silver nanoparticles

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Plasmonic nanoparticles have unique optical properties that lend themselves to unusual optical applications, potentially including use as absorption amplifiers in dye-sensitized solar cells (DSSCs). Atomic layer deposition of TiO2 was examined as a means of not only controlling thickness of films precisely but also protecting particles while simultaneously incorporating them into DSSC functional photoelectrodes. We find that enhanced performance was observed for dye-sensitized solar cells when nanoparticles coated with a thin layer of TiO2 serve as electrodes. An appropriate size of nanoparticles is able to reduce reflection losses because the particles have larger cross-sectional area in the surface plasmon resonance range. The results show that the effects of the surface plasmon resonance of Ag or Au nanoparticles are potential for improve the conversion efficiency of DSSC and the structure and the quantity of particles were very crucial for the application of the surface Plasmon resonance effect to absorption coefficient of electrodes.

16:45 Oral

Growth Mechanism of 3D CuO Nanogrids

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The nanostructure of metal oxides has been a subject of intense research due to their potential applications. CuO, a p-type semiconducting oxide, is having a monoclinic structure and an indirect band gap of 1.4 eV. It's compositional simplicity, strong electron to electron correlation, and monoclinic structure render it an optimal candidate for applications in solar cells, catalysis, etc.

This study investigates the direct oxidation and growth of 3D CuO nanogrids from copper mesh substrates on which organic nanofibers were deposited by electro spinning and act as templates guiding the growth of the novel nanoparticle networks. To study the synthesis of 3-D CuO nanostructures, the experiment was assessed in effects of heating temperature, heating time and heating rate. Novel, self-supported CuO nanostructures in an open 3D configuration formed continuous networks of extreme surface area. The unique CuO nanostructure first occurred by Cu diffusion within the organic nanowires on the copper substrate and subsequent oxidation of the metallic core. The morphology, composition, and structures of the CuO nanostructure were characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and x-ray diffraction(XRD).

17:00 Oral

Characterisation and performance of novel TiO_2-ZrO_2 membranes for gas separation

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The rising content of the greenhouse gas CO_2 in the atmosphere caused by human activities is presumed to be an important contribution to global warming. At present, fossil fired power plants are one of the main producers of CO_2 and an increasing usage of such plants is assumed. A reduction of the CO_2 content in the atmosphere could be achieved by separation of CO_2 using gas separation membranes having molecular sieving properties. In this paper, the preparation and characterisation of novel TiO_2-ZrO_2 membranes are presented.

In a first series of experiments, nano-sized TiO_2-ZrO_2 membrane materials were prepared by a polymeric sol-gel route and their properties were investigated by dynamic light scattering (particle size), N2-adsorption (pore size) and XRD (phase structure). The composition with the TiO_2:ZrO_2 ratio of 1:1 exhibited the most promising characteristics including a particle size of ~3-6 nm, a microporous structure with a pore size <1 nm and an amorphous phase up to 600 °C.

In a second series of experiments, these membrane materials were coated on an alumina membrane carrier consisting of a macroporous α-Al_2O_3 substrate and a mesoporous γ-Al_2O_3 membrane layer with a pore size of 5 nm. Structural examination of the TiO_2-ZrO_2 membranes by high resolution SEM and TEM showed very homogeneous top layers with a thickness of ~30-50 nm. The gas permeation of the top layer was investigated by standard gas permeation tests with He, H_2, CO_2 und N_2. The results were compared with data obtained for microporous SiO_2 membranes which were coated on the same substrate. The SiO_2 membranes showed 100 % H_2/CO_2 separation which indicated the formation of high quality layers by our clean room dip-coating procedure. The novel TiO_2-ZrO_2 layers were much tighter and showed a H_2/CO_2 permselectivity of ~10 with very low fluxes. Current investigations are aimed to enhance this performance by synthesis adaption.

17:15 Oral

Simulation of structural evolution of nanoscopic graphene samples

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At present time a monolayer of honeycomb-like packed carbon atoms, also known as graphene, seems to be one of the most promising materials for technologies dealing with membrane applications. As is well known, uniqueness of graphene is commonly associated with either or both peculiarities of its electronic structure and features of its spatial structure as well as mechanical properties of this material as a flexible membrane. The effect of surface inhomogeneity on the band structure and transport properties of graphene samples is the subject of intense investigations in both experimental and theoretical directions.

In presented work we report on the results of modeling of the graphene's ground state evolution in dissipative mode under temperature-controlled environmental conditions. We estimate spatial extent of the surface corrugations to be of the order of several nanometers (up to 7–8 nm at 300 K), which represents the known experimental and theoretical data to adequate degree of accuracy. The effect of increase in out-of-plane fluctuations with consistent growth of deviations from equilibrium is also appeared in our simulations with the density functional method estimated binding energy potential. The effect however does not result in dangerous crumpling of the layer, keeping the finite height of normal deviations from the plane, which in our model samples reaches the magnitude of ~0.13 nm at high enough temperature. The results obtained confirm available in literature theoretical data concerning the effect of temperature conditions on variation of spatial scale of structural deformations in graphene sheets. We display the tendency of graphene structure to disorder with rising temperature via radial distribution function g(R). By comparing the obtained results with both experimental data and predictions of the other models we conclude on applicability of the used model of dissipative stochastic evolution to nanoscopic graphene structure dynamics.

Posters

Monday, 14 September

Poster Session
Monday afternoon, 14 September, 16:00
Main Hall

16:00	Poster	G-1

Degradation of $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ Membrane Material in CO_2-containing Atmosphere

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$Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ (BSCF5582) is a mixed ionic and electronic conductor which could be used for oxygen separation. The thermochemical behavior of the material is a main factor in the selection of the material for membrane technology. In this work the degradation and the reversibility of degradation is investigated.

It is known from literature that BSCF5582 forms $(Ba,Sr)CO_3$ if it is annealed in CO_2-containing atmosphere.[1] Hence, annealing experiments in 20% CO_2 in air are conducted. Samples are heated up to 800°C and dwelled for 200h. The microstructure is investigated by SEM+EDX. In Fig. 1A a SEM image of the annealed sample is shown. As can be seen a carbonate layer (3.5µm) is formed on the surface. Below the layer needle-like structures occurred (16µm). The reversibility of the degradation is checked by further annealing. As-annealed samples are then annealed in synthetic air at 800°C for 200h. As can be seen in Fig. 1B the carbonate layer is decomposed and SrO is formed. The needle-like structures are partially destroyed.

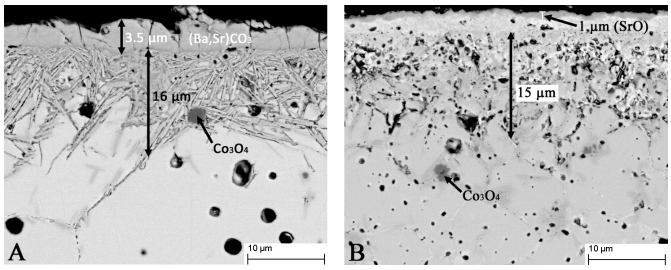

Fig. 1: SEM images of BSCF5582 annealed in 20% CO_2 in air (A) and in 20% CO_2 in air, afterwards annealed in synthetic air (B)

The tendency of BSCF5582 to react with CO_2 and the reversibility of the formation of carbonates is investigated by DTA measurements. Therefore, samples are heated up to 1100°C and cooled down in air, heated up to 1100°C and cooled down in 20% CO_2 in air, and heated up to 1100°C and cooled down in air again. Fig. 2 shows the normalized mass change. After heating up and cooling down in 20% CO_2 in air there is an enduring increase in weight. Anyway, after heating up and cooling down in air again the origin is reached. Hence, it could be assumed that the formation of carbonates is reversible. Our results are in good agreement with literature.[2]

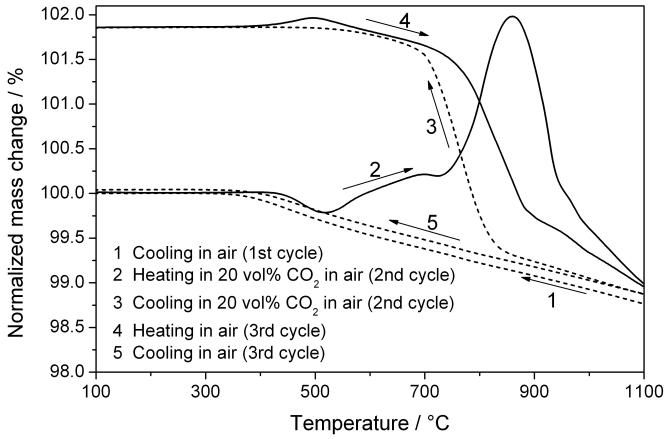

Fig. 2: Normalized mass change of BSCF5582 with and without CO_2

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16:00	Poster	G-2

Ceramic Oxygen Membranes

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Ceramic oxygen membranes operating in the temperature range 600 – 1000ºC will play an important role in future in several industrial fields if different technological issues are solved: (1) thermochemical stability in CO_2 and low oxygen environments, (2) permeability of the material surpassing values of 5 ml/min at moderate temperatures and (3) reliable (mass-scale) manufacturing of thin-films on inexpensive and durable porous supports. In this work it is shown the application of perovskite-based oxygen conducting membranes to oxygen separation and to the catalytic conversion of hydrocarbons into added-value chemicals as olefins. The LnSFC and BaSFC membrane sides were catalytically activated using different oxidic and metallic coatings aiming to improve both (1) oxygen exchange rate and (2) selectivity towards the conversion of hydrocarbons into olefins and syngas. The hydrocarbons studied were methane and ethane whereas the main reaction products were ethylene and syngas. The operating conditions of the catalytic membrane reactor (temperature, gas flows and feed composition) were studied on thick and thin-supported membranes to maximize the catalytic selectivity and productivity. Catalytic stability was assessed for one week under operation with hydrocarbons. The membrane assemblies were postmortem characterised by XRD, SEM while the starting components were studied by XRD, Raman, TPO-TPD.

16:00 Poster G-3

Hydrogen permeation Properties of Pd-coated Ni-Nb-Zr-Ta amorphous membranes

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The widespread of hydrogen-related energies necessitates the production of hydrogen at a large scale and competitive cost. One method of production consists in separating, at high temperature, hydrogen molecules from a gas by means of the permeation technique. Pd-based membranes are currently the most commonly used materials for this application however the price of Pd elements stimulates the search for alternative candidate materials. For that purpose, this study was undertaken to develop new compositions of metallic amorphous alloys with performance at least similar to those of Pd-based membranes. In this paper, we will present the hydrogen permeation properties of Ni-Nb-Zr-Ta amorphous alloy prepared in the form of thin ribbon of about ~30 μm thick. At low temperature, the hydrogen permeability of the Ni-Nb-Zr-Ta alloy was found to be similar to that of Pd-Cu alloy while for temperature larger than 400°C, the hydrogen permeability decreased slightly. The variation in the properties of the Ni-Nb-Zr-Ta amorphous alloy will be discussed based on microstructural modifications detected by XRD, XPS and TEM analyses.

16:00 Poster G-4

Characterization of NiCoCrAlY powders and coating obtained by plasma sparaying for TBC applications

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The thermal barrier coatings (*TBC*) enable to lower temperature (at approx. 170°C) of operating elements in a hot section of gas turbine to a range, which enables to operate for a long time in conditions of high temperature influence and prolongs operation of them even three or four times. Usually, the TBC coverings are constructed of four layers:

- superalloys on a base of nickel;
- outer ceramic zone, from which low thermal conduction is required;
- bond coat of a type Ni(Co)CrAlY or diffusion layer of a type (Ni,Pt)Al;
- layer of barrier oxides, accrueting as a result of temperature growth TGO (thermally grown oxide).

In this article detailed microstructural characterisation of three types of MCrAlY powders and VPS sprayed coatings such as Ni-23Co-17Cr-12.5Al-0.45Y, Co-32Ni-21Cr-8Al-0.5Y and Ni-22Cr-10Al-Y will be presented. The study scope included chemical and phase composition analysis of powders and layers after spraying process. It was stated that e.g. in a case of powder, comprising 23% Co, the dominating phases are as following: NiAl phase with added cobalt in a form of (Ni,Co)Al and a phase on nickel matrix type Ni(Cr,Y). Shape and surface morphology of the tested powders were also characterized. The microstructural assessment of the layers obtained by spraying the analysed powders was performed in the second part of the article. Phase composition, roughness and morphology of top layer of coatings were assessed. Criteria assessment of the obtained layers was performed for their application as the base layer in TBCs.

Financial support of Structural Funds in the Operational Programme - Innovative Economy (IE OP) financed from the European Reginal Development Fund - Project No POIG.0101.02-00-015/08 is gratefully acknowledged.

16:00 Poster G-5

Microstructural characterization of $Gd_2Zr_2O_7$ type of TBC system obtained by APS method

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Generally, the TBC applied in gas turbines is made up of two components: a bond coat created by the vacuum or low pressure plasma-sprayed MCrAlY (M = Ni, Co) and a top coat of yttria and partially stabilized zirconia produced by the atmospheric plasma spraying (APS) or electron beam-physical vapour deposition (EB-PVD). Zirconium oxide, modified with yttrium oxide (YSZ) is a main material of a ceramic layer. Although, the TBC barrier layers, which are based on ceramics type YSZ, have been used for 30 years, their life is a basic problem, because it limits wider application of them. The basic problem depends on production of a ceramic material, provided for an outer layer of thermal barriers (provided to be used in high temperature creep resisting alloys on a base of nickel and niobium as well), and this material is characterized by reduced thermal conduction, comparing to the YSZ, especially in high temperature. The microstructural characteristics of TBC layers on the basis of a new type of $Gd_2Zr_2O_7$ compound was presented in this article. The powder of Co-32Ni-21Cr-8Al-0.5Y type sprayed with the VPS method was used as the base layer. The ceramic layer was sprayed with the APS method. The study scope included standard characteristics of the base and ceramic layer microstructure according to criteria assessment used heretofore for standard TBC layers on the YSZ basis. The study performed showed that the layer of a new type is 250mm thick and uniform in this respect on the whole length (2cm) analysed. No vertical or horizontal cracks were found. No delamination effects of individual layers were either noticed. The porosity observed was at the level of approx. 2.5%, but no massive porosity was found.

This work was supported by the Polish Ministry of Education and Science under the research project No PBZ-MNiSW-01/I/2007

16:00	Poster	G-6

XRD characterization of new type of pyrochlore powders for TBC applications

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Zirconium oxide, modified with yttrium oxide (YSZ) is a main material of a ceramic layer in TBC systems. Although, the TBC layers, which are based on ceramics type YSZ, have been used for 30 years, their life is a basic problem, because it limits wider application of them. Considering very limited possibilities to increase operation temperature of gas turbines, what results from achieving similar operation parameters of turbines to a melting point of superalloys, one can state that development is necessary, from the one hand on high temperature creep resisting materials, on a base of high-melting metals, and on the other hand materials, provided for a new type of thermal barriers, which are used to high temperature creep resisting alloys on a base of nickel and high-melting metals. Zirconates modified with elements of rare-earths type $RE_2Zr_2O_7$ of pyrochlore structure - these materials demonstrate considerably low thermal conduction than the YSZ and high structural stability up-to temperature 1,500°C. Here, zirconates La, Gd, Nd, Sm are the most promising. Diffraction analyses of the powders with pyrochlore structure on the basis of Gd, La, Sm and Nd zirconates were performed in this article. Phase composition was determined in the initial state and after annealing at temperatures within the range 1100-1500°C. In the case of the powder on the basis of Gd, significant changes of phase composition were found. Phase composition analysis of powders after spraying with the APS method was also performed. The above analyses were performed on the basis of the previous differential thermal analysis (DTA) of powders, which made it possible to determine range of temperatures which phase transformations occur at.

This work was supported by the Polish Ministry of Education and Science under the research project No PBZ-MNiSW-01/I/2007

16:00	Poster	G-7

Microstructure and thermal diffusivity of $La_2Zr_2O_7$ powder for TBC aplications

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From the beginning of construction of contemporary aircraft engines, in order to improve life and use properties of applied materials to a maximum, the systems of protective coatings are used. Contemporary constructions of engines, and development in technologies as well, lead to evolution of new protective coatings and to improvement of ones, which were sooner applied. The thermal barrier coatings TBC (*thermal barrier coating*) enable to lower temperature (at approx. 170°C) of operating elements, exposed to creeping, in a hot section of gas turbine (e. g. combustion chambers and directing and rotating blades) to a range, which enables to operate for a long time in conditions of high temperature influence and prolongs operation of them even three or four times, simultaneously reducing consumption of fuel. The results of microstructure tests performed on the powders intended for thermally sprayed TBCs with APS method were presented in this article. The tests of phase and chemical composition of the analysed powder were performed. The carbon, sulphur and gas nitrogen contents were, among other things, determined during those tests. The x-ray powder diffraction phase identification in as received material was determined. The tested material showed the presence of $La_2Zr_2O_7$ compound as the predominant one and ZrO_2 oxides. The surface morphology analysis of the powder was carried out and its internal structure was characterized. The tested material shows porous structure typical for agglomerated powders. The second testing area applied to analysis of the powder thermal properties. The thermal diffusivity of the compressed samples with density similar to the solid material was determined with the laser flash (LF) method. The measurement results show that requirements for the materials used for new generation TBCs are met.

This work was supported by the Polish Ministry of Education and Science under the research project No PBZ-MNiSW-01/I/2007

16:00	Poster	G-8

Microstructural characterization of $La_2Zr_2O_7$ new type of TBC system

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The selection of new TBC materials is restricted by few basic requirements such as: high melting point, no phase transformation between room and the operation temperatures, low thermal conductivity, chemical inertness to the combustion gases and environment, thermal expansion match with the metallic substrate, good adherence to the metallic substrate and low sintering rate of the porous microstructure. Among these properties, one of the most important is thermal diffusivity. The number of material that can be used as TBCs is limited and so far only a few materials have been found to basically satisfy these requirements. Recent research has shown that certain rare-earth zirconates, such as $La_2Zr_2O_7$, have even lower thermal conductivities than 7YSZ, and this has spurred an intensive research in discovering alternative TBC materials. The microstructural characteristics of TBC layers on the basis of a new compound type with pyrochlore structure - $La_2Zr_2O_7$ was presented in this article. The powder of Co-32Ni-21Cr-8Al-0.5Y type sprayed with the VPS method was used as the base layer. The ceramic layer was sprayed with the APS method. The study performed included microstructural characteristics taking into consideration porosity, presence of cracks, contamination, not melted particles and adhesiveness of layers. It was found that the analysed layer is characterized by uniform thickness distribution, which is approx. 250mm. No vertical or horizontal cracks were found. No delamination effects of individual

layers were either noticed. The porosity observed was at the level of approx. 3.5%, but no massive porosity was found. The study of substrate – base layer and base layer – ceramic layer area showed no presence of delamination or oxide contamination in quantities that disqualify the TBC layer.

This work was supported by the Polish Ministry of Education and Science under the research project No PBZ-MNiSW-01/I/2007

16:00	Poster	G-9

Cracks characterization of YSZ and RE zirconates thermal barrier coatings obtained by APS method

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Problems, related to quantity and quality valuation of porosity and cracks in the protective ceramic layers type TBC standards (YZS) and new type (RE2O3-ZrO2) are presented in this article. A procedure of valuation of porosity, which enables not only to carry out quantity valuation of contents of pores, is proposed but it enables also to create a map of porosity. Creation of these maps gives a possibility to valuate the TBC layer from a point of view of identification of "critical areas" i.e. spots of high contents of pores and/or pores of a large area. The results concerning valuation of cracks and micro-cracks in the ceramic layers are presented in the second part of the article. A special emphasis is laid on the quantity and quality valuation of vertical and horizontal components. The results, showing the double-modality of obtained distributions, are presented. A carried out segmentation of cracks points the domination of vertical and horizontal cracks in the ceramic layer. The quantity and quality valuation of pores and cracks has got an essential meaning, from a point of view of characteristics of properties, such as thermal diffusivity.

The application of the quantitative metallography methods, combined with the automatic image analysis presented in described procedures, can form an effective tool for both quantitative and qualitative assessment of such parameters in structural ceramic layers, such as porosity and vertical and horizontal macro- and micro-cracks.

Financial support of Structural Funds in the Operational Programme - Innovative Economy (IE OP) financed from the European Reginal Development Fund - Project No POIG.0101.02-00-015/08 is gratefully acknowledged.

16:00	Poster	G-10

Structural and electronic properties of layered titanate Ln2Ti2O7 (Ln = La or Nd)

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Perovskite structure of rare earth titanate oxides are of growing interest in the recent year. $Ln_2Ti_2O_7$ belongs to the family of perovskite layer structure compounds which is one of interesting ferroelectrics substances characterized by high Curie temperature (Tc > 1500 °C), high coercive filed and a low dielectric constant. In addition, compared with conventional ferroelectric materials, such as lead zirconium titanat or strontium bismuth tantalate, these materials do not contain volatile element which may cause leakage problem due to a possible increase of defect density. Recently, much attention has been focused on the photocatalytic activity in the water splitting with the help of these compounds. It has been shown that the photocatalytic activities are highly dependent on the electronic band structure for these materials. In the present work, we have studied the structural and the electronic properties of the layered titanate photocatalysts by mean of first-principles calculations. These calculations were performed in the framework of density functional theory (DFT), within the generalized gradient approximation (GGA). The total energy calculation and the analysis of the electronic structure are performed within the projector augmented wave (PAW) method as implemented in the Vienna Ab-initio Simulation package (VASP) and the full-potential linearized augmented plane-wave (FPLAPW) as implemented in the WIEN2K package. It is found that the monoclinic $P2_1$ phase of the Ln2Ti2O7 compounds is stable in energy compared to the orthorhombic ones. This phase sustains ferroelectric layers of strongly distorted TiO6 octahedra. The ferroelectric behavior of this phase persists in the orthorhombic Cmc21 one whereas the Cmcm phase becomes paraelectric. We have also predicted the existence of a paraelectric phase, for the $La_2Ti_2O_7$ compound, with the P2/m symmetry. Four the electronic structure of these compounds, we have established that the conduction band of $La_2Ti_2O_7$ consisted mainly of Ti 3d and La 5d, whereas the valence band consisted mainly of O 2p and Ti 3d. The empty La 4f level was found to be located in the conduction band. The occupied and unoccupied Nd 4f level in $Nd_2Ti_2O_7$ was shifted to lower energy. The unoccupied Nd 4f level located between the conduction band and the valence band was found to be detrimental to photocatalytic activity in water splitting because it could act as an electron-trapping site.

16:00	Poster	G-11

MEM-BRAIN Gas separation membranes for zero-emission fossil power plants

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The aim of the MEM-BRAIN project is the development and integration of gas separation membranes for zero-emission fossil power plants. This should be achieved by selective membranes with high permeability for CO_2, O_2 or H_2, so that CO_2 is reached with high

purity in a readily condensable form.

The technical basis is provided by two Research Topics of the Alliance in the field of materials science, one developing ceramic membranes (RT 1), the other polymeric membranes (RT 2). Main scientific challenge is the development of membrane systems with high permeability, specific selectivity and long-term stability. In application these systems have to be included into power plants and an energy system which define additional boundary conditions.

Ceramic membranes operating at intermediate temperatures (≤ 400°C) are developed for H_2/CO_2 separation (pre-combustion process). These include microporous molecular sieving membranes, especially zeolite and sol-gel derived membranes. Dense ceramic mixed proton/electron conducting membranes are developed for operating temperatures above 800°C.

For O_2/N_2 separation (oxyfuel process and hydrogen production for pre-combustion process), dense ceramic mixed oxygen ion-electron conducting membranes are developed for working conditions at 800-1000°C. A demonstration-unit (Proof-of-concept) for testing O_2/N_2 separation membranes is designed and built up. The reference material is $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ (BSCF).

Polymeric and organic/inorganic hybrid membranes are produced for the CO_2/N_2 separation (post-combustion process) at temperature ranges up to 200°C. Beside pure polymeric membranes new hybrid organic/inorganic membranes with inorganic molecular sieves will be prepared.

In RT 3 all power plant processes are modelled and their potential of using membranes for CO_2 capture are analysed.

RT 4 is performing an assessment of specific technical, economic and environmental aspects of CO_2 capture as a component of the whole CCS process chain.

Symposium H

Bulk amorphous and nanocrystalline materials

Welcome

Amorphous and nanocrystalline alloys represent a unique materials group exhibiting better or new properties in comparison with traditional polycrystalline alloys. However, in many cases their application is limited due to small dimensions of the fabricated alloy as a consequence of the technological processes. Recently, a new types of alloys and new processing routes have appeared, enabling the production of amorphous and nanocrystalline alloys and their composites in a bulk form. It gives a new technological potential for novel applications.

Bulk amorphous alloys are produced from the liquid state by various casting techniques (bulk metallic glasses) or by compaction of amorphous powders obtained by atomisation method or mechanical alloying. This symposium covers the most recent developments in this area, including the influence of chemical composition on glass forming ability, thermodynamic aspects of glass formation, phase separation phenomena, possibility of amorphous powders compaction and crystallization processes.

The second main topic of the symposium is fabrication of bulk nanostructured materials by severe plastic deformation techniques or by compaction of nanocrystalline powders.

The symposium is also devoted to the recent progress in properties studies of bulk amorphous and nanocrystalline materials (alloys and composites), covering mechanical, magnetic, corrosion and other properties. Finally, some application aspects will be presented.

Selected topics:

- glass forming ability
- plasticity/ductility of bulk metallic glasses
- corrosion behavior
- soft magnetic properties of bulk metallic glasses
- crystallization processes
- compaction of amorphous powders into bulk form
- nanocrystalline ceramics
- phase separation in amorphous state
- amorphous/nanocrystalline composites

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Proceedings

The proceedings of this symposium will be published in physica status solidi (a), (b) or (c),

after adopting a rather strict reviewing policy.

Programme

Monday, 14 September

Lectures
Monday afternoon, 14 September, 14:00
Room 134

14:00 Invited oral

Shear band melting in metallic glasses

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Metallic glasses (MGs) exhibit high strength, fracture toughness and high elastic limit at ambient temperature, as well as good formability through viscous flow in the supercooled liquid state However, their limited ductility, especially in tension, serves as a major impediment to wider application of metallic glasses. This behavior is related to work-softening and inhomogeneous flow localized in shear bands. One important aspect of the deformation mechanism in metallic glasses is the temperature rise in the shear bands, which may be related to shear softening. A long debate has been raised, with estimates of the local temperature rise ranging from less than 0.1 K to a few thousand Kelvin.

Using high resolution scanning electron microscopy (HRSEM) observations of shear steps on Zr-based bulk metallic glasses (BMG), we have recently reported clear and direct evidence of melting due to elastic energy release in the form of heat in the shear zones. Shear steps were found at several instances to have been formed in successive increments with evident wear and tear on a thickness scale larger than the expected thickness of the shear band itself. The estimated range of attained temperatures and the observed morphologies are consistent with shear steps forming at a subsonic speed limited by a required redistribution of local microscopic stresses. Calculations indicated that a 0.2 μm layer melts in the vicinity of a shear band forming a 1 μm shear step.

Examining a large number of shear steps of several metallic glasses we observed a strong dependence of various factors on the morphology of the shear steps, including the composition of metallic glasses and samples' size. Shear steps morphology differs for metallic glasses with different glass transition and melting temperatures implying that these properties affect the viscosity drop induced by the temperature rise in the shear bands.

14:30 Oral

Molecular dynamics simulation of relaxation, dynamical correlations, and amorphous transition in a rapidly cooled liquid sheet of TiAl alloy

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A significant interest has recently arisen in investigations of titanium aluminides in the form of sheets or thin film forms as thermal protection systems because of their low density, high temperature strength, reasonable oxidation resistance, and low thermal conductivity. Therefore, improving ductility of TiAl alloys by grain refinement to submicron-size levels is a crucial issue to prevent from fracturing and failure of the thermal protection systems for applications[1,2]. The amorphous phase can then be a precursor to a nanocrystalline structure.

For a better understanding of the crystallization kinetics of the amorphous phase and microstructural evolution in the thin sheet, molecular dynamics(MD) simulations have been performed to study structural relaxation within the framework of the embedded atom method(EAM). The time and space correlation of atoms are analyzed in real and reciprocal space for equilibrium liquid, supercooled liquid and amorphous state. The details of atom motion in studied three states are analyzed using three factors including mean square displacement(MSD), non-Gaussian parameter(NGP), and incoherent intermediate scattering function[3,4]. The relaxations of the three factors clearly reveal two relaxation processes involving in slow relaxation and fast relaxation of the cooled liquid TiAl sheet. With increasing undercooling, self-diffusion slows down along with an increasing in the structural relaxation time as compared with the normal liquid state. Accompanying with massive structural rearrangement at a certain supercooling and time, the local structural patterns in the amorphous state are identified by decomposing peaks of pair distribution functions (PDFs) according to the pair analysis(PA) technique[5]. Concerning the studied sheet, the obtained results reveal how quenching rates affect the liquid-amorphous transition as well as the local structure changes.

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14:45 Oral

Stable dispersions of nanocrystalline strontium hexaferrite and composite materials on their base

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Nowadays single domain hexaferrite nanoparticles are in the focus of intense scientific activity. They can be used in highly effective permanent magnets, magnetic fluids and in several biomedical applications. In this study we report on the synthesis of strontium hexaferrite SrFe12O19 nanoparticles with average particle sizes <30nm, their stable dispersions in water and a composite material on their base that exhibits high magnetic anisotropy. The colloids were obtained by dissolving in acetic acid the ceramics synthesized by glass crystallization method. Hysteresis parameters, losses and dependence of the initial susceptibility versus temperature were ex-

plored. Particles size investigations were done by SEM, TEM and AFM. The average particle size and crystal shape were also determined by Rietveld analysis. The diffusion parameters were studied by DLS. The particles exhibit superparamagnetic behavior that allows the colloids to avoid agglomeration and sedimentation. After sustained interaction with a magnetic field the solution splits. One of the liquids, enriched by hexaferrite, behaves like a magnetic fluid: retains its volume and can be drawn by a magnet. If the system is stirred it becomes homogeneous again. The particles have the shape of thin platelets with only two unit cells along the c axis. The composite was obtained by high pressure filtration of the colloids through anodized alumina films and further thermal treatment. The particles embedded in the pores are orientated along the c axis. After sintering they form nanowires of strontium hexaferrite inside the pores. The magnetic properties of the material differ greatly along and normal to the pores. The coercitive power along the pores reaches 6500 Oe and doesn't exceed 4500 Oe in the other direction. The saturation magnetisation is equal in both cases (approx. 1 emu/g) while the remanent magnetisation is two times higher along the pores (approx. 0.7 emu/g).

15:00 Oral

Submicron nucleation kinetics during high-speed laser assisted surface modification.

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Kinetic approach for the particle size distribution function under high-speed laser cooling from the melt is discussed. The Fokker-Planck equation was decided exactly for broadly realized in practice lamellar (dendrite) nucleation in the cases of laser surface coating, micro cladding or amorphization. There were determined expressions for an average size of the crystallizing nucleus, as well as a relative fraction of the solid phase volume. Submicron particle distribution by sizes, average radius and density of new growing phase are evaluated numerically depending of regimes of a laser influence. Influence of morphology, chemical state (catalytic properties) and deformations on the kinetic of phase transformation are discussed. It is shown, that within the framework of considered kinetic approach it is possible to describe different regimes of laser assisted surface modification including nano fabrication. A new criterion of amorphous state after high-speed laser cooling is proposed.

COFFEE

Monday afternoon, 14 September, 15:30
Main Hall

Joint Poster Session

Monday & Wednesday
Monday afternoon, 14 September, 16:00
Main Hall

Tuesday, 15 September

Lectures

Tuesday morning, 15 September, 9:00
Room 134

9:00 Invited oral

FeCoBSiNb bulk metallic glasses with Cu additions

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Recently, (Fe-Co)-B-Si-Nb bulk metallic glasses (BMGs) were produced. Such BMGs exhibit high glass-forming ability (GFA) as well as good mechanical and magnetic properties (s_f: ~4000 MPa; M_s: ~1 T; μ: ~12000; H_c: ~2 A/m). These alloys are regarded as one of the most attractive candidates for application, because they combine the advantages of functional and structural materials. Their good soft magnetic properties can be even enhanced by small addition of Cu. Studies of crystallization and its effect on the magnetic properties in this system are essential for possible commercial application, similar like in the case of FINEMET-type alloys. However, few works have been devoted to this topic so far. In this paper, $\{[(Fe_{0.5}Co_{0.5})_{0.75}Si_{0.05}B_{0.20}]_{0.96}Nb_{0.04}\}_{100-x}Cu_x$ glassy alloys (x = 1, 2 and 3) were chosen for investigation. The GFA and the thermal stability of these alloys were evaluated. The effects of crystallization during heat-treatement processes on the phase evolution, the kinetic parameters and the magnetic properties, including M_s, H_c and T_c, in these alloys were investigated. The phase analyses were done with the help of the X-ray diffraction patterns recorded *in-situ* by using the synchrotron radiation in transmission configuration.

9:30 Oral

Effect of mechanical pretreatment on structure and mechanical properties of bulk metallic glasses

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The recent development of bulk metallic glasses (BMGs) has attracted enormous interest because of their positive combination of high thermal stability against crystallization with remarkable mechanical properties at room temperature. However, the plastic deformation at room temperature of these materials is generally inhomogeneous and occurs via highly localized shear bands, which finally leads to catastrophic failure. Recently, it has been reported that mechanical pretreatment of metallic glasses, such as cold rolling or the application

of an elastostatic stress, may change the mechanical properties of the glass leading to considerable room temperature ductility. These findings suggest that highly deformable metallic glasses may be produced from brittle glassy precursors by simply applying plastic or elastic deformation. The aim of this work is to explore the possibility to ductilize brittle metallic glasses by systematically investigating the effect of mechanical deformation on structure and mechanical properties of different types of glassy materials and to fully evaluate the effectiveness of this approach as a universal ductilization process. In order to achieve this purpose it is essential to understand the fundamental mechanism responsible for the enhanced ductility. To reach this goal, detailed investigations of the structural and thermal properties of the as-cast and the mechanically pretreated materials have been performed to correlate any structural change with the observed changes in the mechanical properties and, finally, to find a model description for their mechanical properties.

9:45 Oral

Phase structure and crystallization of the bulk glassy Fe-CoZrWB alloys

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It was shown in previous studies [1], that $Fe_{61}Co_{10+x}Zr_5W_{4-x}B_{20}$ alloys (where x = 0, 2, 3 at.%) reveal good glass forming abilities, which allow to process bulk glassy samples in a form of rods up to 2 mm diameter, for the x=1 alloy, by suction-casting technique. Furthermore, the suction-casting process, led to produce thin wall tubes of even larger outer diameters up to 4 mm. Magnetic measurements had shown good soft magnetic properties of the investigated alloys. The low fraction of crystalline phase present in the rod samples was impossible to detect by conventional XRD equipment, therefore the synchrotron radiation is due to its advantages, more preferred technique of studying the glass forming abilities. In the present work, the high-energy X-ray diffraction (XRD) measurements performed using monochromatic synchrotron radiation of 112 keV (λ=0.110696 Å) at undulator beamline PETRA 2 of synchrotron facility DESY/ HASYLAB (Hamburg, Germany), were utilized to compare a phase constitution of melt-spun ribbon and suction-cast rods and tubes. The analysis of diffraction patterns revealed fully glassy structure for ribbon samples of all compositions investigated. In case of the bulk samples, some traces of crystallinity were detected for the rod samples, due to lower cooling rates applied for processing bulk glassy alloys. The most evident traces of crystalline phase were observed for the 1mm dia. rods of the x=2 alloy. The X-ray diffraction analysis shown existence of B_6Fe_{23} metastable phase embedded within an amorphous matrix. Similar studies of the thin walled tube samples revealed their fully amorphous nature for all compositions investigated.

The Mössbauer spectroscopy was used to determine hyperfine parameters characteristic for the amorphous phase in rod, tube and ribbon samples. The hyperfine field B_{hf} distributions calculated from the Mössbauer spectra indicated non-equivalent surroundings of Fe atoms in the amorphous phase. Furthermore, dissimilar B_{hf} distributions were observed for samples produced by various rapid solidification techniques.

It was shown that admixture of W into the alloy composition, play important role in the glass forming ability. In the present investigations, the differential scanning calorimetry (DSC) allow to determine the thermal stability parameters of the samples, as well as crystallization processes during continuous heating. For all alloy compositions the activation energy and the crystal growth process parameters were determined using modified Matusita equation [2].

References

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10:00 Oral

Deformation-induced martensitic transformation in Cu-Zr-Al(Ti) bulk metallic glass composites

Simon Pauly[1], Jayanta Das, Jozef Bednarcik, Norbert Mattern, Ki Buem Kim, Do Hyang Kim, Jürgen Eckert[1,2]

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Plastic deformation of Cu-Zr-(Al, Ti) bulk metallic glass (BMG) composites induces a martensitic phase transformation from the B2 to the B19' CuZr phase. Addition of Ti to binary Cu-Zr increases the temperature above which the B2 CuZr phase becomes stable. This affects the phase formation upon quenching in Cu-Zr-Ti BMG composites. The deformation-induced martensitic transformation is believed to cause the strong work hardening and to contribute to the large compressive deformability with plastic strains up to 15%.

10:15 Oral

Nano patterning of glassy carbon surface by focused ion beam at different incident angles and associated applications

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Glassy carbon is a biocompatible carbon material combing glassy and ceramic properties. Nano patterning on glassy carbon surface has been studied by using CrossBeam Focused Ion Beam/Scanning Electron Microscopy (FIB/SEM) system. When the incident angle of Ga ion beam was over 40 degree, ripples started to appear on the irradiated surface. When the incident angle was increased from 60 degree to 80 degree, the induced features gradually changed from

ripples to column-like features. The evolution of surface morphology with incident ion dose has also been studied. FIB induced nano patterning can be used in many areas. Application in microfluidic systems and cell biology will be discussed.

COFFEE

Tuesday morning, 15 September, 10:30
Main Hall

Lectures

Tuesday morning, 15 September, 11:00
Room 134

11:00 Invited oral

Synthesis of novel nanostructures by electrical discharge assisted mechanical milling.

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In this paper we describe the potential of electrical discharge milling as a new route for the synthesis of ultrafine and nanostructural powders.

In our method mechanical milling is assisted by electrical discharges. It might be expected that localized heating in our device generates high temperature plasma, and that the precursor powders are vaporized and then rapidly quenched throughout the condensation process. However by using impulse discharge massive melting can be prevented and the precursor powders in solid form from the beginning up to the end of the process can be maintained. This milling condition leads to formation of novel nanostuctures.

Although the reactions occurring during electric discharge milling are poorly understood, we believe that impulse discharge and vibration of electrodes leads to the formation of what we have termed a "dusty cloud" between the electrodes. The dust particles become suspended in the plasma region between the plunger and the mill base. The interaction between the particle surface and the arriving radicals leads to the nucleation and growth of new phases by deep penetration of ions and subsequent diffusion. We found that this condition is the most effective for formation of nanostructural materials. In this work SEM, TEM and XRD results will be presented and mechanism of formation of nanostructural materis will be discussed.

11:30 Oral

Sintering of nanocrystalline $Y_3Al_5O_{12}$:Nd (3at.%) powders for fabrication transparent ceramics

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The work aims at revealing principle influence of morphology and phase composition of nanopowders YAG:Nd (3 at.%) of various prehistory on their sintering for fabrication of optical ceramics.

YAG:Nd (3at.%) were obtained by chemical co-precipitation method from various initial reagents: $Y(NO_3)_3$ and $Al(NO_3)_3$ (synthesis 1) as well as $Y(NO_3)_3$ and $(NH_4)Al(SO_4)_2$ (synthesis 2) with using as precipitant of $(NH_4)HCO_3$ and by calcination of precursor 1 at 1000^0C, precursor 2 - at 1100^0C as it contains sulfate-group and has higher decomposition temperature. It is established, that at increasing calcination temperature of precursors from 800 up to 1300^0C the average size of garnet particles increases from 20 up to 150 nm (1) and from 20 up to 75 nm (2).

Agglomerates arise both during precipitation and drying and at high-temperature treatment owing to bonding particles with each other. It is established, that insertion of sulfate-ion SO_4^{2-} into reaction mixture provides formation of precipitation particles of spherical form and reduces agglomeration process. Also it is established, that suppression of interparticle agglomeration process promotes garnet powders calcination in oxygen environment.

Sintering process of YAG ceramics from powders of various dispersiveness and agglomeration degree was studied. It is established, that at sintering highdisperse powders there is the largest shrinkage and even ceramics crumbling owing to the phenomenon of zone isolation. At sintering powders with lower specific surface processes of volume diffusion prevail above surface and grain boundary diffusion and the final density of ceramics decreases.

Ceramics YAG:Nd (3 at.%), transparent in the range of visible waves have been fabrication via vacuum sintering at 1750^0C (10h). Average crystallite size of ceramics, character and porous structure depends on morphology of initial powder and the mode of high-temperature sintering. Luminescent, optical and mechanical properties of ceramics were studied.

11:45 Oral

Influence of Fe and Ag addition on the amorphization and thermal stability of CuTiZrNi powders processed by mechanical alloying

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Since 1988 it has been demonstrated that metallic glasses can be made in a bulk form by solidifying the melt at relatively low cooling rates. Over the past decade, BMGs have gained considerable attention due to their properties (high yield strength, hardness and elastic strain limit, relatively high fracture toughness, fatigue resistance and corrosion resistance) and potential as new structural materials. Also Cu-based alloys have been reported to show the enhanced glass forming ability, enabling the preparation of bulk alloys by conventional casting methods. Another possibility of fabrication of bulk amorphous alloys is consolidation of amorphous powders obtained by melt atomisation or by mechanical alloying technique.In the present work, the influence of chemical composition (Fe and Ag ad-

dition) on the amorphization possibility of CuTiZrNi alloys processed by mechanical alloying of a mixture of pure elemental powder was studied. The thermal stability and crystallization behaviour of the amorphous mechanically alloyed powders was determined. X-ray diffraction and differential scanning calorimetry were employed as the experimental techniques for samples characterization.

12:00 Oral

Cu-Ti Base Multicomponent Amorphous and Silver Nanocrystalline Composites

Jan M. Dutkiewicz, Wojciech Maziarz, Lidia Lityńska-Dobrzyńska, Łukasz Rogal, Tomasz Czeppe

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Two easy glass forming alloys based on Cu-Ti binary system of compositions Cu47Ti33Zr11Ni8Si1 (alloy 1) and Ti50Cu20Ni25Co5 (alloy 2) were ball milled for 40 hours to obtain amorphous powders. X-ray diffraction studies confirmed complete amorphisation of both alloys after ball milling. Differential scanning calorimetry DSC measurements detected weak endothermal glass transition effects several degrees below the crystallization peaks at onset equal to 476°C for the alloy 1 and at 457°C for the alloy 2. However, high resolution transmission electron microscopy from powders allowed to identify fine intermatallic particles of a few nm in diameter within the amorphous matrix. Powders were then hot pressed at temperatures between the glass transition and crystallization in vacuum, to form bulk amorphous samples. DSC studies of hot pressed compacts confirmed presence of a similar heat effects as in the milled powders. Composites were prepared in the same way from mixed amorphous powders and nanocrystalline ball milled silver powder. Various ratios of amorphous to nanocrystalline powder were applied with maximum of 60% of the nanocrystalline silver. The microhardness of the amorphous phase was higher than that of nanocrystalline silver and composites has shown lower compression strength than that of the amorphous samples, however of better ductility.

12:15 Oral

Nickel-free austenitic stainless steels and their nanocomposites with hydroxyapatite

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In a newly introduced manufacturing process of nickel-free austenitic stainless steels with nitrogen absorption treatment [1], small devices can be precisely machined in a ferritic phase and than during nitrogenization of their surfaces in nitrogen gas at temperature approx. 1200°C they become nickel-free austenitic stainles steels with better mechanical and corrosion resistance properties.

Using the combination of mechanical alloying (MA), heat treatment and nitrogenation of elemental microcrystalline Fe, Cr, Mn and Mo powders it is possible to syntesize a nanocrystalline nickel-free stainless steels. Nanocomposites with addition of hydroxyapatite have been prepared for the best samples The process parameters, morphology, microhardness and EDX-analysis of obtained powders were determined. Phase transformation from ferritic to austenitic was confirmed by XRD analysis. Changes of size and shape of the mechanically alloyed powder mixtures as a function of milling time was done by the SEM and AFM techniques. Human normal diploid fibroblasts were cultured onto each disk of nanostructured steels. Also the microhardness of the final bulk material was studied using Vickers method. The result is almost two times greater than in austenitic steel obtained by conventional methods. This effect is directly connected with structure refinement and obtaining of nanostructure.

Mechanical alloying is a very effective technology to improve also the corrosion resistance of stainless steel. Decreasing the corrosion current density is a distinct advantage for prevention of ion release and it leads to better cytocompatibility.

According to existing conceptions, decreasing of material's crystallites size to nanometric scale allows to achieve much better mechanical properties (e.g. microhardness) compared to conventional materials [2]. The results show that nanocrystalline nickel-free stainless steels and nickel-free stainless steel/hydroxyapatite nanocomposites could be promising bionanomaterials for use as a hard tissue replacement implants from mechanical and corrosion properties point of view.

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LUNCH

(maps available at the Reception desk)
Tuesday afternoon, 15 September, 12:30
building of the Faculty of Physics

Lectures

Tuesday afternoon, 15 September, 14:00
Room 134

14:00 Invited oral

The influence of heterogeneous nucleation on the prediction of glass formability in metallic materials

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The discovery of numerous bulk glass forming alloys over the past two decades and the possibility of exploiting their high intrinsic mechanical strengths has stimulated a renewed interest in the glass

forming ability (GFA) of metallic systems.

The critical cooling rate for complete vitrification of an alloy, R_c, was shown earlier[1], for high and intermediate values of R_c, to correlate satisfactorily with the reduced glass temperature T_g/T_l, where T_g and T_l are the glass transition and liquidus temperatures, respectively. On the other hand, for easy glass formers having low values of R_c <~ 1K/s, T_g/T_l is, in practice, no longer a useful 'predictive' parameter[2]. The reasons for this are discussed. In particular, the presence of heterogeneous nucleants is shown to have a much greater effect on the predicted R_c for bulk glass forming alloys than for compositions having high and intermediate values of R_c. In other words, the relative importance of homogeneous and heterogeneous nucleation rates is dependent on the magnitude of T_g/T_l[3]. Recent experimental data, indicating large effects on the GFA of small concentrations of specific solutes[4], are cited in support of the arguments presented.

(1) H A Davies, "The Formation of Metallic Glasses", Diamond Jubilee Edition of Phys Chem Glasses, 17, 159-173 (1976).

(2) H A Davies, "Metallic Glass Formation Re-Visited" in "Nanostructured and Non-Crystalline Materials", eds. M Vazquez, A Hernando, pp. 3-14, 1995: Singapore, World Scientific.

(3) B G Lewis, H A Davies, "The Formation of Amorphous Metallic Phases by Continuous Cooling from the Melt", in 'Liquid Metals', eds R Evans, D A Greenwood, pp. 274-82, 1977: London, Inst of Physics,.

(4) I A Figueroa, H A Davies, I Todd, "High Glass Formability for Cu-Hf-Ti Alloys with Small Additions of Y and Si" Phil Mag A, in the press.

14:30 Oral

Formation and properties of the Cu-Zr-Ag-Ti bulk metallic glasses

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Cu – based bulk metallic glasses (BMGs) exhibit interesting mechanical properties since they combine high fracture stress, significant fracture toughness and good corrosion resistance. Quaternary systems with general composition Cu-Zr-Ag-Ti show wide composition ranges in which BMG can be obtain. The aim of this work was to study the influence of silver content on the glass forming ability and on the mechanical properties of the Cu-Zr-Ag-Ti alloys. Multicomponent Cu – based alloys were produced by casting technique into a copper mould, manufacturing rod shape samples with maximum diameter of 3 mm. Structural characterizations were studied by x-ray diffraction. Calorimetric measurements were performed in order to check the stability of the amorphous phase, the presence of a glass transition temperature and the crystallization behavior. Mechanical properties of the investigated alloys were determined by means of Vickers microhardness and room temperature compression tests.

14:45 Oral

How to join heat sensitive materials?

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The nanoeffects like melting point depression are creating new opportunities for joining of advanced materials. Thanks to that phenomenon, heat-sensitive materials like bulk amorphous or nanostructured materials can be brazed without loosing their advantageous properties due to extensive processing temperatures.

The aim of this work is to investigate the principles of the melting point depression on the example of AgCu eutectic nanostructured filler metal.

The magnetron sputtering was used to build a nanomultilayer structure of AgCu layers (2.3nm-30nm) separated by AlN diffusion barrier layer (9nm). The melting point depression of up to 130°C was observed by high-temperature XRD measurements. The experimental results show, on the contrary to the theoretical predictions, no inverse relationship to the layer thickness of the brazing filler. However, the energetic character of the interface that depends on the AgCu layer thickness has to be considered. Thin AgCu layers (≤7nm) show semi-coherent interface resulting in a superlattice, measured by XRD. These interfaces are energetically more stable, than a random orientation interface observed for thicker AgCu layers. In future, designing the diffusion barrier layer would allow tailoring the properties of the interface and maximize the beneficial effect of the melting point depression.

15:00 Oral

Structural transformations and magnetic properties of $Fe_{60}Pt_{25-x}B_{15+x}$ alloys

Agnieszka Grabias[1], Michal Kopcewicz[1], Jerzy Latuch[2], Dariusz Oleszak[2], Maciej Kowalczyk[2], Marek Pękała[3]

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Nanostructured Fe-Pt-B alloys consisting of hard and soft magnetic phases have revealed excellent hard magnetic properties with a potential for fabrication of efficient exchange-coupled spring magnets. In these alloys the ordered tetragonal γ_1-FePt serves as the hard magnetic phase whereas the Fe_2B phase is magnetically soft.

In the present work we compare structural and magnetic properties of two $Fe_{60}Pt_{25-x}B_{15+x}$ (x=0 or 10) alloys. Both alloys were prepared in the form of ribbons by the melt spinning technique. Thermally induced phase transformations in the alloys were studied by differential scanning calorimetry (DSC) measurements. Structural properties

of the as-quenched and annealed alloys were characterised by X-ray diffraction and Mössbauer spectroscopy techniques. Magnetic properties of the samples were investigated by using a vibrating sample magnetometer and a Faraday balance equipment.

The as-quenched $Fe_{60}Pt_{15}B_{25}$ ribbon was fully amorphous whereas the $Fe_{60}Pt_{25}B_{15}$ alloy was partially crystalline with major contribution of the γ-FePt disordered solid solution. Both alloys crystallize revealing a single exothermal peak at the DSC curves with the maximal effect at 847 K and 880 K for x=0 and 10, respectively. The ordered tetragonal γ_1-FePt and Fe_2B phases were formed after the DSC exposure of the ribbons. Magnetic properties of the annealed alloys, studied by hysteresis loop measurements, were related to differences in relative volume fractions of the hard and soft magnetic phases calculated from the Mössbauer spectra. The as-quenched alloys were magnetically soft while the annealed samples exhibited hard magnetic properties. Temperature dependence of magnetization reflected structural transformations of the samples induced by annealing.

15:15 Oral

Correlations Between Structural Differences and Mechanical Properties of Different Forms of Amorphous Silicon

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Pure amorphous silicon (a-Si) is one of the most studied model systems for amorphous networks as it is well known that it undergoes relaxation upon thermal annealing to a new 'state', a state which might be the closest attainable to the perfect continuous random network.

Nanoindentation proved to be a powerful tool to distinguish between these different states of as-prepared and relaxed. For example, as-prepared ion-implanted a-Si will tend to deform via plastic flow under indentation, while relaxed ion-implanted a-Si will undergo a phase transformation to a metallic phase under pressure and further transformation to crystalline phases upon unloading. However, less is known about the mechanical and structural property correlations of other forms of a-Si, pure and deposited, and this study addresses these issues.

Other forms of a-Si have been studied using nanoindentation, Raman microspectroscopy and fluctuation electron microscopy. The first form discussed here is the so-called pressure-induced a-Si, a pure form of a-Si created itself by indentation of crystalline silicon. This pressure-induced a-Si undergoes relaxation in the same manner as ion-implanted a-Si does, resulting in the same phase transformation behaviour and also same short-range order. Interestingly, the medium-range order of the two as-prepared forms, ion-implanted and pressure-induced, proved to be very different reflecting clearly the different formation processes.

Additionally, forms of a-Si created by deposition methods have been studied. The influence of a relaxation anneal on such a deposited forms of a-Si will be compared to the formation of an almost perfect continuous random network upon annealing as exhibited by pure forms of a-Si.

COFFEE

Tuesday afternoon, 15 September, 15:30
Main Hall

Lectures

Tuesday afternoon, 15 September, 16:00
Room 134

16:00 Invited oral

Nanocrystallization kinetics under instantaneous growth approximation: experiments and cellular automata simulations

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Thermally activated nanocrystallization processes in metallic alloys consist of the formation of a partially crystalline material with 40-70 % in volume of nanocrystals embedded in a residual amorphous matrix. This implies the formation of an atypically huge number of crystalline nuclei. The observed constraining in the crystal growth has been explained by the so called soft impingement due to the slow diffusing atoms which pile up at the edge of the growing crystals. A very simplistic idea could describe an initial fast growth of the crystallites till the surface of the crystal is covered by the rejected atoms non soluble in the crystal. Afterwards, the growth is very slow. If this very slow growth is neglected, the time required by the growing crystal to achieve an almost constant size becomes very small in comparison with the time required by the nanocrystallization process to be completed. Under these premises, it is possible to establish an instantaneous growth approximation in which the crystallization kinetics is reduced to the nucleation of crystallites with a constant size (~5 nm). This approach strongly simplifies the kinetic analysis and allows us to obtain the nucleation rate from both isothermal and non-isothermal nanocrystallization processes. Moreover, as no constraining mechanism is considered but the absence of growth, the results could be discussed in the frame of Jhonson-Mehl-Avrami-Kolmogorov theory with a growth index equal to zero. Cellular automata simulations are in agreement with the observed kinetics and microstructure.

16:30 Oral

Glass Forming Ability And Nanocrystallization Kinetics of $Fe_{65}Nb_{10}B_{25}$ Metallic Glasses

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In this work the mechanisms controlling the nanocrystallization kinetics of the $Fe_{65}Nb_{10}B_{25}$ metallic glasses have been determined by the combination of the analysis microstructural data from XRD and TEM, and the kinetic analysis performed using the Master Curve method of the continuous heating and isothermal calorimetric curves. The results show that the transformation starts by the nucleation and interface controlled growth of the $Fe_{23}B_6$-type phase that changes to diffusion controlled growth as the transformation advances until is stopped by the soft-impingement effect.

The transformation is modelled in the framework of the Kolmogorov-Johnson-Mehl-Avrami theory using constant activation energy expressions for the nucleation frequency and interface-controlled growth and taking into account the reduction of those quantities with the transformed fraction due to the change in the matrix composition using a mean-field approximation. The parameters of the modelling are determined from the coupling between the isothermal and constant heating rate calorimetric analysis and from the quantitative analysis of microstructural data. This is the outset for the determination of the viscosity, driving force for crystallization and interfacial energy when replacing the constant activation energy expressions by the classical nucleation and growth ones. Both the glass forming ability in Fe-Nb-B based bulk metallic glasses and the temperature dependence of the interfacial energy are discussed in terms of the influence of the minor alloying elements.

16:45 Oral

Atomistic Models for the Mechanical Response of Nanocrystalline Materials

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As most ordinary solids are polycrystalline, the dependence of mechanical and other properties on the size of their grains is a question of fundamental interest for materials science. The hardness of polycrystalline metals increases with decreasing grain size, in accordance to the Hall-Petch law: their hardness is a linear function of d^{-n}, where d is the average grain size and $n > 0$. On the other hand, when the grain size reaches the nanometer range, several metals have been found to exhibit a so-called "reverse Hall-Petch effect", and become softer at smaller grain sizes. Some very recent experiments show that nanocrystalline ceramics might show the same effect.

In the present work, we present simulations that demonstrate the reverse Hall-Petch effect for a typical metal (Copper) and the ultimate non-metal (Diamond). We consider fully three-dimensional model structures, having several grains of random sizes and orientations, and employ state-of-the-art Monte Carlo simulations for diamond and Molecular Dynamics simulations for Cu. Samples are created in an automated way using the Voronoi construction method, while the grain size is deduced from sophisticated local order analysis. This is necessary in order to get realistic samples for nanocrystalline Cu, where several low-energy metastable structures can exist. The properties of the material are themodynamical average values over several different samples.

We calculate structural properties, elastic constants and estimate the hardness of the materials. Our results compare well with experimental observations. Moreover, we verify that this material becomes softer at small grain sizes. The effect is attributed to the large concentration of grain boundary atoms at smaller grain sizes. Our analysis yields scaling relations for the elastic constants as a function of the average grain size.

17:00 Oral

Growth of CuAg core-shell nanoparticles: silver-enhanced cluster diffusion on amorphous carbon

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Bimetallic nanoparticles offer an additional degree of freedom for modifying their physical properties leading to a wide range of novel applications. We detail here our study on the Cu-Ag system, characterized by a large lattice mismatch (~12%) and non miscibility. Nanoparticles are prepared under ultra-high vacuum (UHV) conditions, either by pulsed laser deposition (PLD) or thermal evaporation (TE). Cu nanoparticles are first synthesized by condensing Cu vapor onto a thin amorphous carbon film, supported by a TEM grid. Ag is then ablated/evaporated and deposited on the pre-existing Cu nanoparticles. For each experiment, two TEM grid fixed on the same heating support are prepared simultaneously, one with Cu and Ag and the other one with only Cu.

Samples have been prepared by PLD with nominal thicknesses of 2 nm and 1 nm for Cu and Ag respectively, at a substrate temperature of 350°C. Core-shell nanoparticles are obtained with a Cu core and Ag shell. It appears that coalescence of many Cu nanoparticles occurred during the Ag deposition, leading to large Cu core inside a bimetallic core-shell nanoparticle. However, with the same thermal history, this coalescence did not occur on the sample with Cu only, and neither on another test sample prepared with the same total nominal thickness (3 nm Cu total nominal thickness).

We demonstrate that this phenomenon depends on thermodynamics rather than kinetics, by considering the results of UHV annealing of 3 specifically designed samples: 1/ using TE instead of PLD, 2/ using samples with Ag deposited at a lower substrate temperature and

3/ using samples covered by a 3 nm thick amorphous Al_2O_3. For the two first samples, identical core-shell configurations are found, very similar to the initial sample described before. For the last sample with Al_2O_3, almost no microstructural changes are observed.

We finally explain how these results support the thermodynamic hypothesis for the rearrangement of Cu nanoparticles under Ag deposition, even if the equilibrium state is not reached.

17:15 Oral

Molecular-Dynamic Research of the Structure of Amorphous Ni

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The paper is devoted to the research by the method of molecular dynamics of the structure of amorphous Ni obtained by superrapid cooling from melted state using the method of molecular dynamics. Main purpose of the research was the determination of the structure of amorphous metal in the dependence on the velocity of cooling by the separation of known structural elements (elementary cells of FCC and HCP, Frank-Kasper polyhedrons, tetrahedrons). The calculated block of Ni contained 13500 atoms in a molecular-dynamic model. The researches were made using two types of potentials of interatomic interaction: Morse pair potential and many-particle Cleri-Rozato potential. Three different velocities of Ni cooling from the melted state: 10^13, 10^14 and 10^15 K/s were considered. The method of cooling was in the corresponding decrease of atomic velocities at every iteration of the computer experiment. It was found in the paper that amorphous metal can have minimum two variants of structure in the dependence on the velocity of cooling from melt. Ni structure at the cooling velocities 10^14 - 10^15 K/s consisted of conjugated and differently deformed tetrahedrons. In that case, the structure had “nuclei of idealness” - clusters formed of two or three conjugated ideal tetrahedrons. Less ideal tetrahedrons were located around ideal ones. Deformation of tetrahedrons increased with the removal from “the nuclei of idealness”. The average distance between “nuclei” was about 10 Å. Frank-Kasper phase consisting of conjugated and opposite deformed icosahedrons formed at the cooling velocity of the melt of 10^13 K/s order. The formation of FCC and HCP nanocrystals competing with Frank-Kasper phase takes place at the given velocity of cooling. Concentration of 14- and 15-vertex polyhedrons of Frank-Kasper was very small and did not exceed 0.1 % 16-vertex polyhedrons were not observed.

Wednesday, 16 September

Lectures

Wednesday morning, 16 September, 9:00
Room 134

9:00 Invited oral

Carbon Nanotubes Supported Bimetallic Palladium-Gold Electrocatalysts for Direct Formic Acid Fuel Cell

Cheng-Han Chen[1], Wei-Jen Liou[1], Wei-Syuan Lin[1], Hong-Ming Lin[1], She-Huang Wu[1], Leszek Stobinski[2,3], Andrzej Borodzinski[2], Piotr Kedzierzawski[2]

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Preparation and characterization of palladium and palladium-gold nanoparticles on multiwall carbon nanotubes (MWCNTs) electrocatalysts towards formic acid electrooxidation are described. Palladium and gold have been deposited on MWCNTs by pol-yol method using $PdCl_2$ and $HAuCl_4 \cdot 4H_2O$ as precursors. The catalysts synthesized after annealing in H_2(5%)/Ar mixture gas are characterized by XRD, TEM, EDS and cyclic voltammetry. According to XRD analysis, annealed Pd-Au/MWCNTs catalyst reveals solid solution structure of Pd-Au nanoparticles. The images of TEM show that: (i) Pd and Pd-Au nanoparticles particle sizes are in the ranges of 5-10 nm and 20-40 nm, respectively, (ii) they are both distributed uniformly on MWCNTs, and (iii) Pd-Au/MWCNTs have core/shell (Pd/Pd-Au) structure. Pd-Au/MWCNTs shows significantly improved catalytic activity and the stability at room temperature for formic acid electrooxidation. The specific catalysis of palladium in Pd-Au/MWCNTs is found to be more than two times higher than that of Pd/MWCNTs catalyst.

9:30 Oral

Effects of impurities in $LiFePO_4$ cathodes on the cycling performance of $LiFePO_4$/MCMB cells

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$LiFePO_4$-based samples with impurities of H_2O, Li_3PO_4, and Fe_2P were prepared from precursors with composition of $LiFePO_4$, $Li_{1+x}Fe(PO_4)_{1+x/3}$ ($0 \le x \le 0.1$), and $LiFe_{1-y}PO_4$ ($0 \le y \le 0.1$) via a solution method. The compositions, crystalline structures, and morphology of the prepared powders were investigated with ICP-AES, XRD, and SEM, respectively. In order to reveal the effects of impurities of H_2O, Li_3PO_4 and Fe_2P on the amount of Fe^{2+} dissolution in $LiPF_6$ electrolyte and on the cycling performance of $LiFePO_4$/$LiPF_6$ electrolyte/MCMB cells, $LiFePO_4$-based electrodes prepared from various prepared powders with various moisture contents were dipped in $LiPF_6$ electrolyte of various temperatures for two weeks. The electrochemical properties of the prepared studied with cyclic voltammetry and capacity retention study. The retention studies

were/will be carried out at various temperatures with both of $LiFePO_4$/Li and $LiFePO_4$/MCMB cells comprised with $LiFePO_4$-based electrodes containing various impurities. The anodes and cathodes obtained from the cycled cells will be studied with XPS and FTIR to uncover the effects of the impurities on the cycling performance of the $LiFePO_4$/$LiPF_6$ electrolyte/MCMB cells at elevated temperatures.

9:45 Oral

Observation and enhancement of visible photoluminescence of PECVD a-SiO_x:H (x<2) thin films by microcavity effect

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a-SiO_x:H (x<2) thin film samples prepared in a PECVD system by using SiH_4+CO_2 gas mixture show strong room temperature visible photoluminescence (PL) at a photon energy of 2.1eV. The PL spectrum of a single layer sample was observed to reshape due to the multiple reflection between two interfaces of the film. The orders of the resonance modes in the PL spectrum range of 1.5eV-2.5eV, were calculated to be within a range of m=8 to m=13. The peak energy of the most intense mode (m=11) was observed at 2.1eV and the half width and the quality factor of the microcavity were calculated as Δn=20THz and Q=20, respectively. Two multi-layered distributed Bragg reflectors (10 and 25 layer pairs) were fabricated with two different refractive indices (n_1=3.2 and n_2=2.4) of the layers with λ/4 (λ_0=580nm) optical thickness. The PL intensity was detected increasing in intensity around the photon energy of 2eV and the other modes were suppressed.

10:00 Oral

Process of transformation nanoparticles cadmium of sulfide on copper of sulfide by the ionic exchange

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Application prospects of nanomaterials is connected with features of their physical and chemical properties. One of the features of nanomaterials is possibility of management of properties nanomaterials by changing their sizes and geometry. In this case it is possible to changing their properties without changing chemical compound of nanomaterials

In the given work it is developed reception method of nanoparticles sulphide of copper from nanoparticles sulphide of cadmium by an ionic exchange.

The technology of reception of nanoparticles is consisted following stages: formation nanoparticles cadmium sulphide in the volume of a polymeric matrix by means of successive ionic layer adsorption and reaction (SILAR) ; transformation nanoparticles CdS→CuS by an exchange of ions of cadmium with ions of copper from solutions $CuSO_4$.

By means of SILAR have been formed CdS nanoparticles in a polymer matrix with 3 and 5 growth cycles. After that samples have been placed in water solution $CuSO_4$ with concentration of 0,05 M and 0,001 M. After ionic exchange samples dried up.

Optical and other characteristics of these samples have been measured. Have been defined the optical band gap of samples depending on time of an ionic exchange. Results of the spent researches have shown, character of change of band gap depends on concentration of solutions. In the changing of time from 0 to 120 minutes at concentration of solutions $CuSO_4$ of 0,001 M the band gap varies in the range of 3,65 eV-2,89 eV, and at concentration of 0,05 M in the range of 3,65 eV-2,97 eV. By means of EDAX the element composition of the received structures depends on time of an ionic exchange has been studied. Results of the spent researches show that for samples of 1 growth cycle (the sizes of particles d ~ 3nm) at the initial stage of an ionic exchange the quantity of ions of copper sharply increases. When the time of sorbtion achieved 3 minutes saturation is reached. Delay of process of an ionic exchange is connected with reduction of atoms of cadmium in shape of nanoparticles. Character of this process depends on the sizes of particles and concentration of solutions.

10:15 Oral

Temperature and frequency investigations of the electrical parameters in the TGS ferroelectric transition

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Pure and d:alanina doped triglycin sulphate (TGS) crystals were grown from aqueous solutions in paraelectric phase, by slow solvent evaporation [1]. Fresh cleaved, polished and silver paste painted samples of ~1cm^2 surfaces and ~1.2 mm thickness were investigated in the -20 ÷ +70 ^oC and 90 Hz ÷ 5 MHz, temperature and frequency ranges. Permittivity e_r and losses, serial and parallel capacity C_s, C_p, serial and parallel resistance R_s, R_p were temperature and frequency investigated using a Hioki 3532-50 type automatic RLC bridge in line with a computer for automatic registering and subsequent processing of the experimental data. Pure crystals show nonreproducible values of the permittivity and dielectric losses crossing up and down the Curie point. More stable and much lower values of the permittivity and dielectric losses could be noticed for the dielectric permittivity of alanina doped crystals as previously shown [2]. The temperature and time evolutions of ferroelectric domains are responsible for this behavior. Both R_s and R_p in the electric equivalent circuit of the crystals shows minima around 49 ^oC, the Curie temperature. Much higher values of R_s, R_p were found for doped crystals due to pined polarization of the crystal induced by the dopant. For pure TGS crystals, losses in the ferroelectric phase show a complicated pattern of time evolution versus temperature. Much smaller and stable values of permittivity and losses, versus temperature and frequency, were found for doped TGS crystals. All investigated parameters show decreasing values with the frequency increase. The ex-

perimental results point out for a more stabilized structure with decreased mobility of the dipoles in the structure of the doped versus pure TGS crystals. Thus, higher figure of merit of doped TGS crystals point to a major advantage for technical applications, as IR detectors, gas sensors, thermal imaging devices, etc.

[1] H.V.Alexandru, C.Berbecaru, Cryst.Res. and Technol., **30** (1995) 307

[2] C.Berbecaru, H.V.Alexandru, Mater.Sci.Eng. B **118** (2005) 141

COFFEE

Wednesday morning, 16 September, 10:30
Main Hall

Lectures

Wednesday morning, 16 September, 11:00
Room 134

11:00 Invited oral

A coupling of rapid crystallization and plastic consolidation as a method for mass-production of bulk nanocrystalline metallic materials.

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Rapid crystallization is a well known method for production of ultrafine grained metallic materials using the overcooling effect. In some cases it is possible to get the amorphous material even. Unfortunately, this method, nevertheless of technical realisation of this process, provide material with very limited size, which must be consolidated to bulk form for technical use. To prevent destabilisation of ultra-fine grain structure the consolidation process should to be quick enough and conducted in reasonable low temperature. Such reasons assure the process of plastic consolidation. In this process plastic deformation of individual particles develops new *in status nascendi* surfaces, which is able to create of strong bounds between collectively deformed particles of dispersed materials and convert the powders to the bulk form. The plastic consolidation is the process of diffusionless agglomeration of plastically deformable particles of dispersed materials. Bulk materials with no measurable porosity are the product of this process, in which the character of interaction among individual particles of powder shows similar character like interaction at grain boundary or interfacial borders in polycrystals. Technical realization of this process can use the different plastic forming methods - drawing, rolling, forging and extrusion. This last process in form of isothermal extrusion as well as cold extrusion by "KoBo" method seems to be the most promising from economical point of view.

11:30 Oral

The Evolution of Internal Energy Released from Nanomaterials during Grain Growth

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The driving force for limited thermal stability of Nano-Poly-Crystals is a high concentration of internal energy. This makes their microstructures and properties to undergo significant changes when exposed to temperatures exceeding 30% of the melting points of their conventional material counterparts. Two processes are expected to take place under such temperatures, which reduce the internal energy: (a) reduction in the total grain boundary (GB) surface area or GB volume fraction and (b) reduction of the GB energy per unit surface area. The first one leads to the so called grain growth, as the reduced surface of grain boundaries divides the material in question into smaller number of grains with larger sizes. The second process equilibrates the grain boundaries and this can be achieved via reduction of the content of linear and point defects in the grain boundaries or by rotation of grains towards orientations implying lower (specific) misorientation configuration.

This present paper couples the knowledge from energy method with that from theory of stochastic differential equations to study the energy released from nanometals during grain growth. The energy method is used in determining, practically, the important stress (and, hence, energy) distributions in loaded materials, while the theory of stochastic differential equations deals with the analysis of the (temporal) evolutions of the constituent structures in nanomaterials.

The proposed model is tested on polycrystalline nano-aluminium samples. Results reveal that the coupling of the work energy method with the theory of the stochastic differential equation offer a reverberating tool for studying the energy evolved from nanomaterials during grain growth. The impact of annealing time, mean grain size, grain size dispersion and annealing temperatures on the on the internal energy concentrations of the Nanocrystalline aluminium samples are also revealed. Results also demonstrate that a considerable amount of energy is released from the nanomaterials during grain growth. It is also revealed that the energy released from nanomaterials as a result of increase in grain size by GBM and GRC mechanisms is smaller than the energy released from the material during grain growth. The energies involved in T2 events where grains translate after exchanging neighbours and the energy involved in rotating the grain before coalescence in which cases the grain sizes do not change have also been dealt. The energy evolved due to T1 events where smaller grains instantaneously disappear is also dealt with. It is also revealed that the rate of release of the energy is initially very high when the grain sizes are small, but this rate reduces approaching zero as grains grow larger. Thus, it may be concluded from the principle of stationary potential energy that the nanomaterial attains energy equilibrium and as such, the driving force

for grain growth at larger micro-sizes is not more the grains' internal energy concentration.

11:45 Oral

Investigation of fatique crack growth rate of Al 5484 ultrafine grained alloy after ECAP process

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During the last decade equal-channel angular pressing (ECAP) has emerged as a widely-used fabrication route of ultrafine-grained (UFG) metals and alloys. Enhanced mechanical properties of UFG materials produced by severe plastic deformation, with a grain size smaller than 1μm, have been reported in a large number of publications. However, the higher strength does not imply higher resistance to fatigue both high- and low-cyclic. In fact, due to reduced plasticity of UFG materials higher fatique crack propagation rates are reported, particularly in low-amplitude range. The aim of this work was to investigate fatigue crack propagation in Al 5483 alloy subjected to ECAP treatment. Because of small dimensions of the coupons processed by ECAP, non standard, mini- samples were used in a crack propagation tests. Two test procedures were used to estimate stress intensity factor (K). The first one was based on optical measurements of crack length from images recorded during the test. The second method was based on Digital Image Correlation (DIC), which allowed to determine displacement field near the crack tip and, subsequently, to determine K value directly from the displacement data. The paper compares both methods of determining the K values. It also reports the relationship between the intensity of ECAP process (measured in terms of the number of ECAP passes) and fatigue crack propagation rate. In addition to fatigue resistance, the results of tensile tests carried out with mini-samples are presented. Applicability of such samples in the investigations of the mechanical properties of UFG materials is discussed.

12:00 Oral

Reduced activation ODS ferritic steel – recent development in high speed hot extrusion processing

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This article presents microstructure and mechanical properties of an oxide dispersion strengthened (ODS) reduced activation ferritic (RAF) Fe-14Cr-2W-0.3Ti-0.3Y_2O_3 steel fabricated by hot isostatic pressing (HIP) followed by high speed hot extrusion (HSHE) and heat treatment (HT) at 1050°C. Transmission electron microscopy (TEM) revealed significant differences in grain size and dislocation density of as-HIPped and as-HSHE materials. Heat treatment performed after HSHE however, did not cause any significant changes in microstructure of the ODS steel.

The HSHE process significantly increased tensile and Charpy impact properties of the ODS steel. Tensile strength at room temperature increased from 950MPa up to 1400MPa and at highest testing temperature of 750°C as-HSHE material has quite good strength of about 400MPa. Also, Charpy impact properties, especially upper shelf energy (USE), significantly increased from 3.0 up to 6.0 J. Ductile-to-brittle transition temperature (DBTT), however, remained relatively high (about 75°C).
These preliminary results showed that high speed hot extrusion process of the ODS ferritic steel is a promising method for grain refinement and mechanical properties improvement.

12:15 Oral

Effect of deformation and strain rate on diminishing of microstructure of aluminium alloys (AlCu4Zr0.5 and AlZn6Mg3CuZr)

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Intensive deformations influence strongly microstructure. The very well known phenomenon is the diminishing dimension of grain size in materials deformed by the Severe Plastic Deformation (SPD) methods. The nanometric features of microstructure were discovered after the SPD deformation of various materials, such as aluminium alloys, iron and others. The observed changes depended on the kind of the deformed material, amount of deformation, strain rate, existence of different phases and stacking fault energy. The influence of the strain and strain rate on the microstructure is commonly investigated nowadays. It was found that the high strain rates activate deformation in shear bands, microbands and adiabatic shear bands and its effect is similar as exertion of very large strains.

The results of two aluminium alloys: AlCu4Zr0.5 and AlZn6Mg3CuZr deformed by hydrostatic extrusion process were presented. The samples were deformed at the strain rate of $7.4\times10^{1}\div3.84\times10^{2}s^{-1}$ and in the range of true strains $\varphi=1.39\div2.4$.

After the deformation process the samples were investigated using an optical and electron microscopy. Also the statistical analysis of geometry of microbands observed in the microstructure was performed and the microhardness was measured. The average width of microbands and microhardness served to outline the Hall - Petch relationship. It was found that the inclination of the diagrams depends on the kind of the alloy. The Hall - Petch relationship is fulfilled only for AlZn6Mg3CuZr alloy.

A characteristic feature of the microstructure was large density of the microbands with a great misorientation to the surrounds. The in-

crease of the strain rate and the deformation caused the reduction of the microbands width in AlZn6Mg3CuZr alloy. In both alloys after true strain φ=2.4 the mean width of the microbands was placed in the range of d=100÷120nm.

The aim of the present study was to establish the influence of the strain rate and the deformation on the possibility of microstructure refinement in the AlCu4Zr0.5 and AlZn6Mg3CuZr alloys.

Acknowledgements

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(maps available at the Reception desk)
Wednesday afternoon, 16 September, 12:30
building of the Faculty of Physics

Lectures

Wednesday afternoon, 16 September, 14:00
Room 134

14:00 Invited oral

Study of the phase composition of Fe_2O_3 nanoparticles using x-ray diffraction and Debye formula

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Important physical properties of nanoparticles are determined mainly by their atomic structure, especially by their phase composition and the presence of structure defects. X-ray diffraction is a good tool for studying the structure of the nanoparticles, its application for very small particles is however limited by very small intensity of the scattered wave. For this reason special experimental setups, like e.g. diffraction with small incidence angle, are used and many experiments have to be done at synchrotrons. Standard methods of the measured data analysis based on the description of the diffraction using instrumental functions and functions of physical broadening of the lines fail in the case of very small particles. An ab-initio calculation method (based on the Debye formula) has to be used instead. In this work the Debye formula is used for the description of the diffraction of iron oxide samples measured at ANKA synchrotron in Karlsruhe. Using this approach we determine basic parameters of the particles such as lattice parameters and the size of the particles, as well as the presence of different phases. During annealing, subsequent phase transitions from γ-Fe_2O_3 to ε-Fe_2O_3 and to α-Fe_2O_3 take place. New phases nucleate at the surface of the nanoparticles and the phase transformation proceeds towards the particle center, so that the structure of the nanoparticles can be described by a core-shell model; this model was used in the Debye-formula based simulation. From the analysis of the experimental data we determined the kinetic parameters of the phase transitions and their dependence on the nanoparticle sizes. The results of the structure studies are compared with the SQUID measurements of the magnetic properties of the iron-oxide phases.

14:30 Oral

Lutetium oxide nanoparticles and ceramics on their basis

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Recently object of steadfast attention of scientists became nanopowders of oxide rare earth elements, used for reception of optical ceramics. Interest to these materials is caused by prospect of their wide application in a science and technics as laser environments for powerful lasers, optical elements, transparent phosphors, magneto-optical materials.

The purpose of work was obtaining and research nanopowders of oxide lutetium. By the method of colloid synthesis in solution has been synthesized precursor, at further which temperature treatment have been received monodisperse, the spherical form nanoparticles in diameter 60-120 nm. The degree of dispersion of nanoparticles Lu_2O_3 does not exceed 10 %. The specific surface nanopowder Lu_2O_3, appreciated with the help of method BET, makes 14-22 m^2/g. By a method of X-ray diffraction it is determined, that received oxide lutetium has cubic symmetry of a crystal lattice with the period 10,39 Å.

One of the major problems for obtaining dense optical nanoceramics is prevention of process of agglomeration nanoparticles which occurs at a stage of drying and annealing precursor. The lead researches on use of various methods of drying precursor (drying on air at room temperature, 60°C, 120°C; azeotropic distillation with use ethyl alcohol; lyophilization; microwave drying) have shown what to prevent agglomeration nanoparticals Lu_2O_3 it is possible at drying precursor on air at room temperature and at drying with the help azeotropic distillation. At the further annealing at temperature 700 °C turn out not agglomerated nanopowders Lu_2O_3.

From isolated monodisperse nanoprticles Lu_2O_3 by colloidal pressure filtration and monoaxial pressing have been received homogeneous dense compacts. The density received compacts makes 45-52 %. The preliminary optical properties of vacuum sintered Lu_2O_3 ceramics have been determined.

14:45 Oral

New spherical nanocrystalline heterostructures on the basis of Lu2O3:Eu

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It is known that lutetium oxide is the effective host for rare-earth dopants. Nowadays nano-sized Lu2O3 doped with Eu3+ ions is considered as one of the most perspective materials for X-ray detection and imaging and also new X-ray phosphors creation due to its high density (□=9.42 g/cm3) and high effective atomic number Zeff.=71. However, preparation of Lu2O3 spherical particles with narrow size distribution by conventional soft chemistry methods meets some difficulties, which is limited of the nanostructured scintillation materials creation on the lutetium oxide basis. Development of the method of the spherical Lu2O3:Eu3+ particles preparation with the given sizes and heterostructures on their basis opens up the possibilities of the creation of the effective X-ray screens, high-resolution displays with improved characteristics and also photonic matrixes preparation with the diffraction enhancement of the luminescence light. Monosized Lu2O3:Eu3+ spherical particles (diameter range from 60 to 300 nm) and core-shell heterostructures on their basis (Lu2O3:Eu3+/SiO2, SiO2/Lu2O3:Eu3+/Lu2O3) were prepared for a first time. The structure, morphology and X-ray luminescent properties were studied in this work. Lu2O3:Eu3+ spherical particles were prepared by the co-precipitation technique from water solutions with subsequent heat treatment. Europium content in the samples was ranged from 1 to 10 at. %. Lu2O3:Eu3+/SiO2 core-shell heterostructures were synthesized according to Stober method. Lu2O3:Eu3+ coverings of the controlled thickness (10-50 nm) were formed on the surface of the SiO2 spheres by the sol-gel technique from water solutions. The shape, sizes, phase composition of the heterostructures prepared were studied by means of electron microscopy methods, X-ray diffraction, X-ray photoelectron and FT-IR spectroscopy. The temperature conditions of the crystallization and morphology properties of the Lu2O3:Eu3+ solid-state solutions in the form of spherical particles and nanolayers onto SiO2 spheres were investigated. Lu2O3:Eu3+→ Lu2SiO5:Eu3+ → Lu2Si2O7:Eu3+ structural transformations in the heterostrucrures prepared in the 900 – 1200 ºC temperature range were observed. The features of Eu3+ entering in the low dimensional systems were also discussed. The X-ray luminescent properties of the Lu2O3:Eu3+ spherical particles and Lu2O3:Eu3+/SiO2, SiO2/Lu2O3:Eu3+/Lu2O3 heterostructures were studied. Under X-ray excitation europium-doped nanophosphors obtained are characterized by effective luminescence in the □=575–725 nm range corresponding to 5D0□7FJ transitions (J=0□4) of Eu3+ ions in the crystalline host. It has been shown that radioluminescence intensity of the heterostructures strongly depends on the phase composition, shells thickness and Eu3+ dopant concentration.

15:00 Oral

Transition phases in nanocrystalline M_2O_3 oxides

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A transition phase can be defined as a structure formed at relative low temperature, irreversibly transforming at higher temperature into an equilibrium phase of the same elemental composition Among the transition aluminas, gamma-alumina, usually produced from boehmite, is an extremely important technological material. It is easy to produce gamma-alumina by sol-gel technique. This alumina, which is formed by firing the xerogel at temperature range between 500 to 800 °C, has a nanocrystalline cubic spinel-like structure entirely different from the rhombohedral hR10 structure of alpha-alumina, that formed by thermal treatment at higher temperatures. This work deals with nanocrystalline M_2O_3 oxides formed by firing xerogels at relatively low temperatures. The following nanocrystalline binary oxide systems were studied: $MgO-TiO_2$, $ZnO-TiO_2$, $NiO-TiO_2$, $ZrO_2-Al_2O_3$, and $Y_2O_3-Ln_2O_3$, and $Y_2O_3-Ln_2O_3$ (Ln = rare earth element). Some pure or doped unary oxides were also studied. The xerogels were heated at constant T (200 to 1600 °C) for 3 to 6 hours. There was a threshold temperature for oxide formation and in many cases the products were transition nanocrystalline phases, depending on grain size and composition, including doping. The binary $MM'O_3$ oxide phases of $NiTiO_3$, $MgTiO_3$, and $ZrTiO_3$ are ilmenite type structures with rhombohedral hR10 structure similar to alpha-alumina, however since they have more than one component it is possible to study nanocrystalline solid solutions. It was found that firing the xerogels at temperature range between 300 and 600 °C transition nanocrystalline phases were obtained. The phases have several cubic types. In contrast to the ilmenite-like compounds ($NiTiO_3$ for example) which are stoichiometric, the transition phases exist at wide range of non-stoichiometric compositions. A model correlating the size effect with the unusual solid solutions and structures is proposed, correlating the size effect with the structure and enhanced solubility.

15:15 Oral

Influence of α-Al_2O_3 seeds on densification of δ-Al_2O_3

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One of the critical factors in advanced ceramics is synthesizing the starting powder. Therefore in the last few years many different methods of synthesis of nanosized alumina powders were developed. Unfortunately, very often the synthesized nanopowder is not the thermodynamically stable phase. Hence, the phase transformations occurs during sintering process. The transformation into the stable α-Al_2O_3 goes through the stages of nucleation, transformation to a phase and coalescence of α-nuclei. During densification a intragranular pores are closed so to achieve fully densification in the final stage of sintering the holding time and temperatures must be increased. But the kinetics of transformation and in the consequence the microstructure can be enhanced by the addition of α-seeds that act as a nucleation sites for phase transformation.

In this work a commercially available δ-alumina powder (NanoTek) with average particle size 47nm was seeded by 1, 1.5 and 2wt% of α-Al_2O_3 (TM-DAR, Taimei Chemicals, Japan). Obtained samples were sintered in different temperatures. Next the influence of addition of α-Al_2O_3 on the transformation, microstructural development

and densification was studied. The researches showed that the unseeded samples have lower sintered densities although the green densities of seeded and unseeded samples were similar. The transformation temperature and the microstructure of seeded samples after sintering also was improved

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COFFEE

Wednesday afternoon, 16 September, 15:30
Main Hall

Joint Poster Session

Monday & Wednesday
Wednesday afternoon, 16 September, 16:00
Main Hall

Thursday, 17 September

Lectures

Thursday morning, 17 September, 9:00
Room 134

9:00 Invited oral

Challenges in the development of light metal (Al and Ti) based high entropy and bulk amorphous alloys.

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BMG's containing great majority Al element are missing in the literature. Only traditional amorphous ribbon could be prepared so far with thicknesses well below 1 mm. Surveying the data from the literature and more then 30 home made compositions, it turned out that in order to obtain ductile Al-based metallic glasses (MG's) containing more then 75-80 at% Al, one have to select alloying elements for which the heats of formation (ΔH) are between -5 and -25 kJ/mole. In addition, we have to exclude those alloying elements which form compounds with high Al content, above 80 at%. We have calculated also the atomic mismatch (Λ parameter of Egami) for these alloys and using the ΔH versus Λ map we tried to assess the GFA. We have found new amorphous Al -based compositions basing on the assumption that the higher the number of stable and meta-stable phases in the surroundings of the selected composition the higher the GFA.

Instead of BMG's the one phase crystalline solid solution of high entropy alloys (HEA) would be a good candidate for a light metal structural material combining the high strength and ductility. In order to obtain a crystalline solid solution both the atomic mismatch (Λ) and the free energy (ΔG) should be small enough to not drive out the solute element in compound like precipitates. The free energy can be diminished by increasing the configurational entropy composing a multi-component alloy with equal atomic concentrations of at least 5 constituents: $\Delta G = \Delta H - T\Sigma c_i \ln c_i$, This idea proved to be valid for the mixing of the late transition elements (Cu, Ni, Co, Fe, Cr) and their derivatives [1]. In the case of Al and Ti based multi-component alloys however, extended solid solution was observed only for components which forms no compound phases.

Electric discharge assisted compaction was applied to obtain BMG and HEA samples as disks of 2 mm thick and 10 mm diameter.

References

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9:30 Oral

Cu/Ag interfaces TEM characterization for core-shell nanoparticles

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Non-miscible bimetallic systems have been widely studied, in the bulk state and more recently in thin film configurations. Cu-Ag is one of these face-centered cubic systems, allowing probing the effects of lattice mismatch and surface and interfacial energies. It is however new to consider the same kind of system in the nanosize range. We can wonder whether the same growth mecanisms apply when the substrate is now a cluster rather than an infinite surface. We detail here our study on the Cu-Ag system, characterized by a large lattice mismatch (~12%) and a non-alloying tendency in the bulk state, through the use of Transmission Electron Microscopy (TEM) techniques corrected from lens aberrations.

Nanoparticles are prepared under ultra-high vacuum conditions by pulsed laser deposition. Cu nanoparticles are first synthesized by condensing Cu vapor onto a thin amorphous carbon film supported by an electron microscopy grid. Ag is then ablated/evaporated and deposited on the pre-existing Cu nanoparticles.

It is worth noting that for all the nanoparticles characterized, Cu and Ag are in a perfect epitaxial relationship with all crystallographic directions corresponding. Moreover, we observe interfacial dislocations accommodating the lattice misfit (a_{Cu}=0.3615 and a_{Ag} = 0.4086). The spacing between the dislocations corresponds nicely to the theoretical one. The other striking result is the observation of Ag nanoislands on the surface of the particles. Since the STEM technique can be thickness sensitive, we believe that in the present case the contrast corresponds to a "low resolution" topographic map somewhat identical to what an STM image would produce. A triangular superstructure is clearly distinguishable on the surface of the particle, that corresponds very likely to the 9x9 Ag on $Cu_{(111)}$ superstructure.

We conclude by underlying the similarities observed for the Cu-Ag system between thin films and nanostructures configurations.

9:45 Oral

Inside the Moiré Patterns: a high resolution TEM study

Mehrnoush Valamanesh[1], Cyril T. Langlois[2], Nadi Braidy, Christian Ricolleau[2]

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The Moiré phenomenon is well known in conventional optics as well as in transmission electron microscopy but since the recent evolutions towards high spatial resolution, the use of the Moiré patterns has lost its importance. We propose here a new insight into this phenomenon and we show how it can be used to obtain the thickness of two superimposed crystals.

We worked with (111) reflections of copper and silver to study the pseudo-lattice fringes appearing inside the Moiré patterns (fig.1a). The high resolution images are simulated using the dynamical mutislice technique on a crystal of two superimposed thin films of copper and silver. Some of the simulations were carried out in kinematic conditions of electron diffraction in order to model the (111) fringes analytically. These pseudo-lattice fringes appear in both cases in the simulations.

In the first place we studied the variation of the periodicity of the pseudo-lattice fringes with the relative thicknesses of the two superimposed films. We can see that when the value of s, the deviation parameter from Bragg reflections, is small (dynamical condition) the variation of this periodicity with the thickness fraction of copper is independent of the total thickness and has a linear form. This result allows us to find the thickness fraction from a single high resolution image.

On the contrary, for the large values of s (kinematics conditions) it has been found that this variation is not linear and depends on the total thickness. This result is confirmed using an analytic equation representing the intensity of the transmitted and diffracted waves superimposed.

In the last part we have used this equation to fit the intensity profile of the simulated images(in the kinematics conditions) and we have used the thickness fraction obtained above as a fixed parameter and the total thickness as a fit variable. In many cases we can find the right total thickness with this fitting method (fig.1b).

We conclude this study with a discussion on the possible application of this technique.

Fig.1 a) Simulated image of the Moiré patterns of a cristal of 4nm of copper and 2nm of silver b) analytic intesity profile fitted into simulated intensity profile

.

10:00 Oral

Application of electron tomography to analysis of nanoparticles in structural alloys

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Transmission electron microscopy (TEM) images are 2-dimensional projections of analyzed 3-dimensional objects. In many cases determination of microstructural parameters using stereological rules may lead to inaccurate conclusions. Electron tomography enables 3-dimensional visualization of a material microstructure by using sets of projections obtained at different tilt angles of the specimen (tilt series). This method provides a possibility to avoid uncertainties in determination of size, shape and 3-dimensional distribution.

In order to utilize electron tomography, applied imaging technique should generate contrast that has a fully monotonic relationship with thickness. For crystalline specimens, bright-field and dark-field imaging are not suitable for tomography due to no monotonic contrast generated by diffraction effects or Fresnel fringes. An alternative technique is Z-contrast imaging, obtained by High Angle Annular Dark-Field Scanning Transmission Electron Microscopy (HAADF-STEM), which shows little or no diffraction effects. Its intensity is approximately proportional to Z^2 and thickness of a specimen. Those images may be used for reconstruction of the 3-dimensional space.

Main goal of the performed investigation was identification and visualization of nanoparticles in two structural materials: oxide dispersion strengthened (ODS) alloy, INCOLOY MA956 and nickel based superalloy, IN718.

For electron tomography investigation of oxide nanoparticles (dispersoids) in INCOLOY MA956 extraction double replicas were used. STEM-HAADF tilt series were applied for investigation of shape and chemical composition of dispersoids. The results showed that MA956 alloy contains several Y-Al oxide phases and the shape of nanoparticles is related to their crystal structure and chemical composition.

IN718 superalloy is strengthened by coherent nanoparticles of g' and g'' intermetallic phases. To investigate 3-dimensional distribution of g' and g'' nanoparticles, energy filtered transmission electron microscopy (EFTEM) was used to collect series of images at different tilt angles. Due to a similarity of chemical composition of g' and g'' phases, elemental maps of Cr, which is dissolved in g matrix, were recorded. Analysis of the particles shape showed two classes of precipitates present in IN718 superalloy: spherical–like and asymmetric shaped, elongated through the XY plane.

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10:15	Oral

Gas suspended ZnO clusters and pulsed laser deposition of ZnO thin film

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We report on spectroscopic investigations of interaction of an expanding laser ablated plume of aluminum, zinc, graphite with ambient atmosphere of oxygen and nitrogen gas and the formation of nitrides and oxides. The ambient pressure and fluence dependence on the reactive process occurring in the ablated plume responsible for formation of oxides and nitride is discussed. Fast photography/imaging technique using ICCD is gaining importance as a diagnostic tool for understanding the dynamics of the expanding plasma plume and the gas phase nanoparticle synthesis, thus providing a novel way to understand the transportation of nanoparticles and of control of particle size. A comprehensive study done on oxide and nitrides films using pulsed laser deposition technique for possible laser oscillations in UV region of electromagnetic spectrum, in particular of ZnO will be presented. Rayleigh scattering and photoluminescence technique are used to confirm the formation of clusters in gaseous phase. The intensity of the PL emission band increases with increase in time delays of probe pulse with respect to ablating pulse, however the intensity of Zn atomic transitions decreases. The observed emission band falls in the UV spectral region of PL of ZnO, and is attributed to excitonic recombination in ZnO clusters formed in vapor phase. The clustering of grains could be the major cause of increased surface roughness of ZnO thin films deposited by PLD at high pressure. We have deposited ZnO films by PLD at moderate pressure of oxygen ambient. X-ray diffraction spectrum of film shows that grains are well oriented along c-axis. The PL profiles of films show strong UV emission band, and weak violet and green emission band.

COFFEE
Thursday morning, 17 September, 10:30
Main Hall

Posters

Monday, 14 September

Joint Poster Session

Monday & Wednesday
Monday afternoon, 14 September, 16:00
Main Hall

16:00	Poster	H-1

Nanocrystalline Ni_3Al-based alloys produced by mechanical alloying of Ni-Al-Co powders and consolidation

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In this work, preparation of nanocrystalline Ni_3Al-based alloys by mechanical alloying process followed by hot-pressing consolidation is reported. Ni75Al20Co5 and Ni70Al25Co5 elemental powder mixtures were subjected to ball milling. Subsequently, the milling products were sintered at 1000 ^oC under a pressure of 7.7 GPa. The powders and the consolidated pellets were investigated by X-ray diffraction. The results obtained show that the milling products were in both cases supersaturated solid solutions Ni(Al,Co) and that during hot-pressing consolidation they had transformed into nanocrystalline Ni_3Al-based intermetallic compounds. The microhardness of the produced Ni_3Al-based alloys is 920 HV0.1 in the case of $Ni_{75}Al_{20}Co_5$ composition and 1030 HV0.1 in the second case. The density of compacted materials is close to the theoretical value and their open porosity is negligible.

16:00	Poster	H-2

Bulk nanocrystalline Al-Si-Fe-mischmetal (Mm) alloys produced by high pressure hot compaction

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Nanocrystalline Al-based alloys containing silicon (Si), rare earth metal (RE) and late transition metal (Fe), are interesting for their excellent mechanical properties, exceeding those of the commercial crystalline Al-based alloys. Rapid solidification and mechanical alloying are well known methods to obtain alloys with nanometer grain size. The nanometer grains provide an increase of mechanical properties of the alloys. However, the possible industrial aplication of these alloys is limited by the reduced dimensions, obtained by the traditional rapid solidification techniques. That is why, many attempts to produce nanocrystalline aluminium alloys in bulk form have been carried out. The aim of this work was to manufacture high strength bulk nanocrystalline alloys from Al-Si-Fe-RE system. Replacement of RE by Mischmetal (Mm) reduces the cost of investigated materials, as Mm is an order of magnitude cheaper than pure rare earth elements. Bulk nanostructured alloys were produced by ball milling of nanocrystalline ribbons followed by high pressure hot compaction.

16:00 Poster H-3

Amorphous $Al_{60}Fe_{20}Ti_{20}$ alloy obtained by consolidation of mechanically alloyed powder

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Bulk amorphous $Al_{60}Fe_{20}Ti_{20}$ alloy was produced by consolidation of mechanically alloyed powder. Mechanical alloying of 60%Al-20%Fe-20%Ti powder mixture yielded the alloy with an amorphous structure. Thermal behaviour of the milling product was examined using differential scanning calorimetry. It was found that heating in a calorimeter causes crystallisation of the amorphous phase at 525 °C. The amorphous powder was compacted under a pressure of 7.7 GPa at a temperature 500 °C for 180 s. X-ray diffraction investigations of the bulk material revealed that the amorphous structure was retained after consolidation process. The microhardness of the bulk amorphous sample is 856 HV0.2 The density of the bulk amorphous material is 4.113 g/cm^3 and its open porosity is negligible. The results obtained show that the quality of compaction preserving amorphous structure is satisfactory and the product's microhardness is relatively high.

16:00 Poster H-4

Structure and magnetic properties of Fe-Nb-B amorphous/nanocrystalline alloys produced by compaction of mechanically alloyed powders

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Compaction of $Fe_{85}Nb_5B_{10}$ and $Fe_{75}Nb_{10}B_{15}$ powders obtained by mechanical alloying in a Spex mill was performed under isostatic conditions at 7.7 GPa at different temperatures (823, 973 and 1273 K). Quality of compaction, microstructure, thermal stability and magnetic properties were studied using different techniques: scanning and transmission electron microscopy, X-ray diffraction, differential scanning calorimetry and vibrating sample magnetometry. The as-milled powder of the alloy with 5 at. % Nb exhibits two phases: bcc□Fe(Nb,B) nanocrystals (~10 nm) and almost pure B inclusions (~100 nm). However, in the case of the alloy with 10 at. % Nb, an amorphous phase is also observed. The presence of this phase enhances the quality of compaction. Compaction at 823 K preserves both microstructure and magnetic properties of the as-milled powder in both alloys. Compaction at 973 K affects mainly the crystalline fraction of the alloy with 10 at. % Nb. Compaction at 1073 K yields the formation of bcc-Nb and fcc-$Fe_{23}B_6$ type crystals which magnetically harden the material.

16:00 Poster H-5

In situ XRD study of the transformation characteristics of severely plastic deformed NiTi SMAs

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Severe plastic deformation (SPD) of NiTi alloys (Ti-rich and Ni-rich) has been performed by High Pressure Torsion (HPT). Aim of the study was to follow the structural evolution in the samples subjected to SPD, in the martensite ↔ austenite phase transformation temperature regions. HPT is carried out at room temperature (RT) on two separate NiTi samples, Ti-rich alloy with martensite finish temperature (M_f) above and Ni-rich with austenite finish temperature (A_f) below RT. The samples have been studied by in situ X-ray diffraction (XRD) in order to get information about (i) the transformation characterisitics and (ii) the structural evolution during annealing. The results showed that the Ti-rich alloy retained the martensite phase and Ni-rich alloy was observed having austenite phase at RT after SPD. However, in the case of Ni-rich alloy subjected to SPD followed by heat treatment, it is seen that the grain size is in the nanoscale region.

16:00 Poster H-6

Modelling the influence of the grain size distribution on the properties of nanometals

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The influence of the average grain size on the mechanical properties of metals is usually described by the Hall-Patch relationship (H-P), which has been experimentally proved for a wide range of grain sizes. It has been postulated in the past, that the flow stress of polycrystalline metals depends also on the diversity of the grain size. This effect is of particular importance when the grain size is reduced to the nanometric level. In the present study finite element model has been developed which explains the effect of the grain size diversity in quantitative way. The results of computations are compared with the experimental data for nano-aluminium.

16:00 Poster H-7

Grain refinement in constrained groove pressing of metals and alloys

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Severe plastic deformation (SPD) is one of the most effective methods for producing nanocrystalline (grain size < 100 nm) or ultrafine-grained (100 nm < grain size < 1000 nm) metals and alloys. There are several SPD techniques to synthesize bulk nano- or ultrafine metals (alloys), including equal channel angular pressing (ECAP) and high-pressure torsion (HPT). However, both of these techniques are discontinuous processes and can be applied to fabrication of samples up to certain sizes. Recently, it has been shown the effectiveness of constrained groove pressing or rolling (CGP/R) in reducing the grain size from tens of micrometers to submicrometers in several materials. In our work the applicability of CGP for grain refinement and improvement in mechanical properties of different metals and alloys was studied. The influence of repeated CGP (different number of passes) on the microstructure refinement was investigated by transmission electron microscopy (TEM) and electron backscatter diffraction (EBSD). Changes in mechanical properties were measured by tensile and hardness tests and then related to microstructure development.

16:00 Poster H-8

Wear resistance of the nanostructured 316 LVM austenitic stainless steel processed by hydrostatic extrusion

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Austenitic stainless steels are widely exploited in industry and are used for biomedical applications. For these reasons, the improvement of their properties is of great importance. One of the efficient ways to increase the mechanical properties is grain size refinement down to nanometer scale. Recent results showed that such a refinement is accompanied by increase of flow stress and hardness. On the other hand little is know about the effect nano-refinement on other properties such as wear resistance. The aim of this work was to investigate wear resistance of the nanostructured 316 LVM austenitic stainless obtained by hydrostatic extrusion (HE).

The tribological tests were performed using a rotating pin-on-disc with fixed pin. The counterpart materials were hardened chromium steel X17CrNi16-2 and ultra-high molecular weight polyethylene. All tests were conducted in normal saline solution (0,9 % NaCl) with sliding speed 0.2 m/s. The applied loads were 1, 3.5, 6 MPa and the sliding distance was 720 m. The wear rate was calculated by weight loss. The morphologies of the worn surfaces were observed with a scanning electron microscope and characterized by optical surface profiler.

The results indicate that hydrostatic extrusion process in addition to flow stress improves also tribological properties of the 316 LVM stainless steel. This finding opens new possibilities for biomedical applications of the nano-structured 316 LVM steel.

Acknowledgment

The HE processes were carried out at the Institute of High Pressure Physics of Polish Academy of Sciences.

16:00 Poster H-9

Recrystallization and Grain Growth in Nano-structured Austenitic Stainless Steel under Electric Current Heating

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Nanometals obtained by severe plastic deformation (SPD) exhibit very high strength combined with satisfactory plasticity. The microstructure of SPD metals can further be optimized by post-deformation heat treatment. In most cases, this heating is performed in radiation furnaces. On the other hand, it has been shown that heating by an electric current may significantly change the course of recrystallization and grain growth, due to the influence of the electric current on the migration of point defects and dislocations.

The aim of this work was to determine the influence of electric current heating on the process of recrystallization and grain growth in nano-structured metals obtained by SPD methods. Austenitic stainless steel 316LVM was used in this context for testing the efficiency of such a heat treatment.

Samples of 316LVM austenitic stainless steel were hydrostatically extruded, in a multi-step process with the total true strain of 1.84 to produce a uniform microstructure consisting of nanotwins. The twins averaged 19 nm in width and 168 nm in length. Earlier results proved that a post-deformation annealing at 700°C results in the formation of a recrystallized structure with nanograins of 68 nm in diameter. In the present study samples were heated to 700°C by alternating current, AC, for different periods of time using Gleeble 3800 thermo-mechanical simulator. Changes in the nanostructure of the AC annealed samples were investigated using transmission and scanning electron microscopes. Microhardness was also measured under the load of 200g.

The results obtained on the SPD samples of 316LVM austenitic stainless steels after conventional and AC heating are compared and discussed.

Acknowledgement

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16:00 Poster H-10

Tailoring mechanical properties of nano-structured Eurofer 97 steel for fusion applications

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EUROFER 97 steel is a candidate structural material for the future fusion reactors and Test Blanket Modules (TBMs). In the present work microstructure of Eurofer 97 was modified by hydrostatic extrusion in multi-step process with total true strain exceeding 3.
TEM observations showed that HE causes significant grain refinement from about 400 to 80 nm. This is accompanied by improvement of the tensile mechanical properties and microhardness. On the other hand, there is a clear decrease in the resistance to brittle fracture as measured in the Charpy impact tests.
In order to improve strength/ductility/fracture toughness balance, the extruded samples were annealed for 1 hour at temperature range of 473-1073K. The results obtained for samples after post-extrusion annealing are discussed in terms of the microstructure (grain size, precipitation, dislocations) relationship to the properties of Eurofer steel.

16:00 Poster H-11

Grain refinement in CuCrZr by SPD processing

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CuCrZr alloys exhibit relatively high strength (due to precipitation hardening) and high thermal and electrical conductivity which make them attractive for variety of applications, e.g. as heat sinks and electrodes. In the present study, a CuCrZr alloy was subjected to severe plastic deformation (SPD) to increase its strength by grain refinement without modification of the chemical composition. A combination of the multi-pass equal channel angular pressing (ECAP) followed by cumulative hydrostatic extrusion (HE) was applied with a total true strain of ~10. The microstructure of SPD samples was evaluated by transmission and scanning electron microscopy. The grains and particle size distributions were quantitatively described. The resulting mechanical properties were determined in tensile tests and via microhardness measurements. The results show that the applied SPD route, combining ECAP and HE, leads to high grain size refinement accompanied by high increase of strength. The possible improvement in life-time of electrodes made of CuCrZr alloy is discussed.

16:00 Poster H-12

Structure and properties of copper after large strain deformation

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Structure and properties of Cu in dependence on strain (from e~ 0,9 to e~ 15) during multi-axial compression processing at room temperature was investigated. The evolution of dislocation structure, misorientation distribution and crystallite size were observed by using transmission electron microscopy (TEM) and scanning electron microscopy (SEM) equipment with electron back scattered diffraction (EBSD) facility. The structure–flow stress relationship of multi-axial compression processing material at strains e~ 3,5 and e~ 5,5 is discussed. At strain e~ 3,5 the deformation microstructure indicated a mixture of elongated geometrically necessary boundaries (GNB) and dislocation cell structures with dense dislocation tangles. Some regions revealed the wide bands which probably corresponded with the elongated initial grains, while other regions revealed fine dislocation bands (DBs). After e~ 5,5 the spacing of the DBs structure become smaller and microstructure has evolved into an approximately equiaxed subgrain structure, and the dislocation density of grain interiors seemed lower than those after e~ 3,5. It is found that processing does not produce any drastic changes in deformation structure and the microstructural refinement is slow. These results indicate that dynamic recovery and recrystallization play an important role during multi-axial compression process in this range of deformation. For this reason, a slight changes in the mechanical properties was observed. Due to small volume materials available for testing one way of measuring mechanical properties was using mini tensile test samples. In this work minisamples with total length of 2,2mm were used. Digital Image Correlation method (DIC) was employed for the strain measurements in a uniaxial tensile test.

16:00 Poster H-13

The influence of grain refinement on the corrosion mechanism of 7475 aluminium alloy

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The 7xxx series aluminium alloys are widely use in the aerospace industry due to their high strength. The grain refinement down to nanometre scale offers the possibility to further increase their mechanical properties which was already confirmed in a number of stud-

ies. However until now, there is a lack of comprehensive studies of the influence of grain size refinement on the corrosion mechanisms of aluminium alloys.

The aim of this study was to compare the electrochemical behaviour of polycrystalline and nanocrystalline 7475 aluminium alloy. The corrosion resistance and susceptibility to localized attack was investigated by means of potentiostatic and potentiodynamic polarization in solutions containing chloride ions of different pH (0.05M NaCl of pH2, pH8.4 and pH12). The extended microstructural characterization was carried out using SEM, TEM, EDS and EBSD techniques to determine the morphology of the corrosion attack.

The polarization tests showed that in strong alkaline environment, the electrochemical behaviour of poly- and nanocrystalline material was very similar. In strong acid solution, the nanostructured material was characterized by higher dissolution rate whereas in the solution of pH 8.4, the breakdown potential and the open circuit potential were shifted to more negative values which indicated poorer corrosion resistance of nanocrystalline material. The microscopic investigations indicated that in both cases the corrosion attack was strongly related with intermetallic particles, however, the morphology of this attack is different. In the case of polycrystalline material the peripherical pitting and intergranular corrosion along high angle grain boundaries was observed while in nanocrystalline material the corrosion damage was accumulated only in the region near the intermetallic particles.

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16:00 Poster H-14

Bulk nanostructured titanium fabricated by hydrostatic extrusion

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This paper is concerned with Hydrostatic Extrusion (HE) as a one of Severe Plastic Deformation (SPD) methods. The material investigated was pure titanium grade 2. Titanium in the form of rod was subjected to multi-stage extrusion with the aim to grain refine and improve the mechanical properties. During subsequent operations of extrusion the rod diameter was gradually reduced. HE is a technique, which permits to accumulate the strain necessary for grain refinement and obtain the nanostructure. The important and basic problem concerned with nanometals after SPD is their relative small volume. HE emerged recently as an effective method of the production of bulk nanostructured metals.

The aim of this study was to optimize the HE process in order to increase the nanostructured titanium rods diameter. The one of the elementary condition of the nanostructure receiving is to accumulate the strain above three ($\varepsilon > 3$). As our studies show, it is possible to obtain nanostructured Ti rods with diameter above 3mm and exceeded 5mm also. The homogeneity of the microstructure and the grain size were examined on transverse and longitudinal cross-sections of the rods. The average grain size on the transverse cross-sections appeared to be below 100nm. The grains observed on the longitudinal cross-sections were elongated and their average grain size exceeded 100nm. According with changes of structure the significant improvement of mechanical properties was observed. The tensile strength was more than 1000MPa and hardness increased over 50%.

Acknowledgement:

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16:00 Poster H-15

Effect of grain size on reactive diffusion between Ti-Al

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Nanocrystalline alloys represent a unique materials group exhibiting better or new properties in comparison with traditional polycrystalline alloys. One of this kind of materials is nanocrystalline titanium obtained by hydrostatic extrusion. Hydrostatic extrusion is a method which allows to achieve very large imposed strains in bulk samples without fracture, to realize grain refining and significant hardening. The performance properties of nanomaterials can be improved using surface treatment. Ti-Al layers increase the wear and corrosion resistance of titanium alloys. The aim of this paper was to examine the effect of titanium substrate structure refinement (to the nanometric scale) on the chemical and phase composition of Ti-Al intermetallic layers. An accent was placed on evaluation of surface hardness and microstructural aspects. The material examined in the study was commercially available pure titanium with average grain size of 90 um in as-received state. The titanium samples in the form of 30 mm rods were extruded to a diameter of 5 mm, which corresponds to the true strain of 3.8. HE resulted in a strong refinement of the microstructure. After HE the nano-titanium with grain size about 60 nm was obtained. In the next step, the aluminium coatings have been deposited on the titanium in initial state and after hydrostatic extrusion using magnetron sputtering technique. The samples with aluminium coatings have been annealed at 600°C in argon atmosphere. The microstructures of the titanium with aluminium coatings, before and after annealing, have been investigated using scanning transmission electron microscope (Hitachi STEM 5500) and transmission electron microscope (JEOL 1200EX). The samples for the observations have

been prepared using Focus Ion Beam microscope (Hitachi FB 2100). The performed investigations allow to determined the role of the nanostructure on the diffusion and intermettalic phase occurrence rates, and on the layers mechanical properties.

16:00 Poster H-16

Titania nanotubes as an effective support for metal nanoparticles

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Materials of high specific surface area are promising for catalysis and photocatalysis as an efficient support for metal nanoparticles which often demonstrate the tendency to agglomeration on heating. Inorganic nanotubes are one of rather new types of materials which are intensively studied during last ten years. Such materials pose the unique types of structure and special porosity because of inner hollows and grain boundaries. The specific surface area of titania nanotubes reaches 400 m^2/g which is not the record value but could be enough. The diameter of inner cavities is about 5 nm, the outer diameter is about 10 nm. Titania nanotubes degrade above 400°C as a result of dehydratation process leading to nanocrystalline anatase.

Platinum, gold and silver nanoparticles were deposited onto TiO_2 nanotubes using deposition-precipitation and wet-impregnation techniques. Nanocomposites were characterized using TEM, XPS, BET, TPR-H_2 methods. All the experiments we performed in an excess of oxygen to CO. Catalytic activity of materials was studied in CO oxidation model process. Photocatalytic activity of the probes was analyzed by methyl-orange and brilliant green decomposition.

We showed that the deposition technique influences strongly on the catalytic and photocatalytic activities of nanocomposites. Sodium percentage in TiO_2 nanotubes is critical for both catalytic and photocatalytic activities of materials.

16:00 Poster H-17

Synthesis of gallium nitride nanowires by Sublimation Sandwich Method

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Recently the research on obtaining nanosize materials such as nanowires, nanorods or nanoballs is one of the most prospective topics in the semiconductor technology. We present results of experiments concerning obtaining gallium nitride nanocrystals by Sublimation Sandwich Method. As starting materials we used a mixture of gallium and gallium nitride powder and gaseous ammonia as source of nitrogen. The GaN nanocrystals grow on various substrates with gold nanoballs (a catalyst agent) by the vapor- liquid- solid mechanism. The process reactions are as follows:

$$Ga + Au \rightarrow GaAu_{(alloy)}$$

$$GaAu_{(alloy)} + NH_3 \rightarrow GaN + 1.5\ H_2 + Au$$

The obtained gallium nitride nanocrystals were examined by scanning electron microscopy (SEM), Raman studies and Powder X-ray diffraction (PXRD).

This work has been supported by the European Union in the framework of European Social Fund through the Warsaw University of Technology Development Programme.

16:00 Poster H-18

Microscopic and surface analytical characterization of TiO_2 nanotubes produced by anodic oxidation and thermal treatment

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Titanium oxide nanotube arrays were fabricated in a mixture of glycerol, deionized water and low concentration solution of NH_4F (0.86 wt. %). Well-ordered nanotube arrays of titania ~1.0 μm high and about 100-120 nm in diameter have been prepared via electrochem-

ical oxidation at constant voltage (25V). SEM, X-ray diffraction, Raman spectroscopy, AES and XPS surface analytical techniques were used to characterize the self-organized porous layers, and the effect of annealing at 200 – 600°C on their morphology, structure and mechanical stability. Our results show, that the nanotubular structure was stable within the temperature range from 200°C up to 600°C, but their crystallographic structure changed from amorphous, via anatase to the rutile phase.

16:00	Poster	H-19

Modelling of the Cluster-Nature Solvent Features of Single-Wall Carbon Nanohorns

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The existence of single-wall carbon nanotubes (SWNTs) in organic solvents, in the form of clusters, is discussed. A theory is developed based on a bundlet model for clusters, describing the distribution function of clusters by size. The phenomena have a unified explanation in the bundlet model of clusters, in accordance with which the free energy of an SWNT, involved in a cluster, is combined from two components: a volume one, proportional to the number of molecules n in a cluster, and a surface one proportional to n^(1/2). The bundlet model for clusters enables describing the distribution function of SWNT clusters by size. The droplet model is formally analogous to the one for fullerene clusters. From purely geometrical differences the models predict different behaviours. Single-wall carbon nanocones (SWNCs) of various disclinations are investigated via energetic and structural analyses. Several SWNC's terminations are studied, which are different among each other because of the type of closing structure and the arrangement of them. The SWNC packing efficiencies, and interaction-energy parameters, are intermediate between the ones of fullerene and SWNT clusters; an in-between behaviour is expected. However, SWNC properties are calculated closer to those of fullerene and more distant from those of SWNT. Provisional conclusions follow. (1) Close packings are the tightest way to pack spheres. Atoms and fullerenes being nothing but tiny spheres often arrange in this way. Furthermore it is also possible to deduce atomic structures of metal alloys, salts and oxides by fitting the voids of close-packed spheres. (2) The single-wall carbon nanocone-nanohorn packing efficiencies, and interaction-energy parameters, are intermediate between those of fullerene and single-wall carbon nanotube clusters. Therefore an in-between behaviour is expected.

16:00	Poster	H-20

Soft – chemistry preparation of porous globules of nanocrystalline TiO_2 with improved photocatalytic properties

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Nanomaterials based on TiO_2 promise a wide range of unique properties and highly attractive applications. They are of great interest in photocatalysis, as electrode materials, oxidation catalysts, sensors, self-cleaning surface components.

Most of TiO_2 preparation methods such as sol-gel, hydrothermal, aerosol pyrolysis, are usually not enough flexible and do not allow to control key functional properties of produced materials. In particular, various works focused on ultrasonic spray pyrolysis apply the only method of thermal decomposition of aerosols of titanium alkoxides precursors. In the present work we developed a novel flexible and effective approach employing directly inexpensive and readily available $TiCl_4$ in the route closely related to low-temperature soft-chemistry methods. In the method, aerosol of hydrated titania in submicron water droplets was obtained directly in an experimental setup using controllable hydrolysis $TiCl_4$ vapors in aerosols of various aqueous solution of organic and inorganic compounds; thus formed mist was decomposed in a hot zone of a tubular furnace with controlled temperature of 500-1100^{0}C in air or oxygen. It allows to control composition and properties of thus obtained materials, easily introduce dopants, influence on morphology, specific surface area etc. Moreover all the process are in fact united in one experimental cycle. Using solutions of block copolymer, urea, H_2O_2 aqueous solutions, we can tune porosity, doping and then the photocatalytic activity of the obtained titania.

The obtained titania particles were found to have a spherical shape with a diameter ranging from 50 to 2000 nm and a complex surface structure. In the case of precursors containing organic or inorganic gas-forming component, porous particles were obtained. Using urea solution has resulted to nitrogen-doped TiO_2, adding H_2O_2 to the hydrolyzing solution has demonstrated a drastic change of titania morphology. All the samples produced at temperatures above 600°C possessed an anatase structure and a different degree of crystallinity, then an amount of rutile becomes higher with a temperature increase.

All the samples obtained were analyzed with XRD, REM, TEM, nitrogen sorption (surface area analysis), DTA, TGA, IR and UV-vis spectroscopy, ESR.

16:00	Poster	H-21

Metal oxides doped with silver nanoparticles as tunable plasmonic materials

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New composite materials containing noble metal nanoparticles are presently actively developed. In such composites drastic changes of the host media properties can take place on account of the plasmonic activity of the metal nanoparticles. Electric field of the excitation light is enhanced nearby metal nanoparticles at the plasmonic resonance wavelength. Such a local field with enhanced strength is used for the resonance amplification of light absorption, scattering, Raman scattering and fluorescence [1] or improving non-linear optical

properties of materials [2].

The aim of the present work is creation of a composite material with adjustable wavelength of plasmonic resonance based on metal oxides doped with silver nanoparticles. The host oxides TiO_2 and ZrO_2 were prepared by sol-gel method. The chosen hosts possess two advantages: i) their permittivities are high enough for shifting resonance of silver nanoparticles to longer wave-lengths; ii) they allow by changing of porosity to provide an additional tuning of the resonance wavelengths. Silver nanoparticles in the form of colloidal solution (5-15 nm) or powder (30-50 nm) was used as a dopant. To get optically transparent in visible range material thermal treatment in air was needed. To recover plasmonic properties of silver after annealing, reduction of samples in the H_2:Ar atmosphere was performed. As a result, the silver-containing TiO_2 and ZrO_2 films of good optical quality were obtained. Steering of plasmonics resonance in spectral range from 380 nm up to 430 nm was achieved for films with 4-8 wt % of nanoparticles. Potential of embedding of luminescent markers (like rare earth ions) into considered films or possible applications of the obtained films as surfaces for enhanced Raman scattering is discussed.

1. Nanomaterials and nanochemistry / Eds. C. Brechignac, P. Houdy, M. Lahmani. Springer (2006).

2. Smith D.D., Snow L.A., Sibille L., Ignont E. // J. Non-Cryst. Solids, V. 285, P. 256-263 (2001).

16:00	Poster	H-22

Evolution of Platinum Palladium nanoparticles supported on Silicon Dioxide Amorphous by High Resolution Electron Microscopy

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Platinum (Pt) Palladium (Pd) nanoparticles supported on silicon dioxide (SiO_2) were prepared by wetness impregnation techniques at same concentrations of Pt(0.5) and Pd(0.5) ≈ 1 metallic wt %. The particle size distribution were measured as function of reduction temperature, in the range of 200 ºC - 400 ºC to evaluate the nucleation phenomena. In addition morphology and cristallinity under various reactor temperature were investigated by physisorption Brunauer-Emmett-Teller-(BET), X-Ray Diffraction (XRD), High Resolution Electron Microscopy (HREM) and Transmission Electron Microscopy (TEM). In this work, we observed the distribution of Platinum and Palladium in nanoparticles. By XRD we found Pt-Pd bimetallic nanoparticles with ffc structure and particles diameter in the last stage was about 4.2 nm was estimated by HREM and Bright field image allowed to determine that Pt-Pd nanoparticles were found mainly to have cube octahedral shape.

These results clearly demonstrated that particle size distribution is affected by chemical rate as a function of temperature. At high temperature, the particle size increased. The reaction temperature greatly affected the crystallinity of nanoparticles. It was found that particle generation process consist of the nuclei control, in which the concentration of nuclei is low, (200ºC) the second is the nucleation surface reaction which the concentration of nuclei is enough large(300ºC), and the last (350ºC-400ºC) when the chemical reaction are complete. In the last stage Pt_x-$Pd_{(1-x)}$ Bimetallic nanoparticles presented cubo-octahedral shape.

16:00	Poster	H-23

Fabrication of Nanocomposite Thin Films of Silicon Nanoparticles and Amorphous SiO_2

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Semiconductor nanoparticles have received much attention due to the quantum confinement effect and potential applications in the biological, electrical or photonic fields. As the luminescent nanoparticles are embedded in the supporting media, the semiconductor nanoparticles could be used widely in photonics research area.

The amorphous SiO_2 films filled with Si nanoparticles were prepared from SiO_2 sol and Si nanoparticles colloid. Si nanoparticles in the diameter of 2-5 nm were prepared from Zintl salt sodium silicide (NaSi) by solution routs. The surface-passivating ligand, chlorine, was replaced with hydroxyl group by the reaction of chlorine-capped Si nanoparticles with distilled water. The prepared water-soluble Si nanoparticles were dispersed to ethanol and incorporated into SiO_2 sol by hydrolysis of TEOS. The freshly prepared Si nanoparticle-sol mixture was spin-coated on Si subatrate. The substrates were annealed at 300, 400, 500, and 700 ^oC in a furnace under air.

The PL spectrum of colloidal Si nanoparticles covered a wide range 340-700 nm with a peak maximum near 420 nm upon 325 nm excitation. As for the nanocomposite thin films, the peak of PL was more blue-shifted and the intensity decreased with the higher annealing temperature, possibly due to the oxidation of Si nanoparticles into SiO_2. Some other optical and material analysis will be discussed.

16:00	Poster	H-24

ZrO_2:Tb nanopowders obtained by coprecipitation method

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Nanopowders with luminescent properties are of great interest since low energy-consuming light source is still in demand. The main purpose of the work is to obtain luminescent material based on terbium doped zirconia, via coprecipitation route. Quality of nanopowder de-

pends on many factors such as temperature and time of calcination, temperature of solution, concentration of precipitation agent, intensity of agitation, etc. The content of terbium in obtained ZrO_2:Tb samples varied from 0,1 wt.% to 5 wt.%. Powders were prepared starting from solutions of Zr and Tb nitrates, using ammonia solution as coprecipitation agent. Corresponding metal hydroxides were precipitated at the pH value of 10, then filtered and washed with water. The residue was next dried at the temperature of 70°C, and ground in agate mortar. The effect of preparation conditions and of chemical composition on properties of obtained nanopowders was described. Phase composition was determined using X'Pert X-Ray diffractometer. SEM observations were performed using LEO 1530 scanning electron microscope. Multipoint BET measurements were performed using Micromeritics Gemini instrument. Density was measured using Micromeritics AccuPyc 1330 helium pycnometer. Crystallite size in obtained powders varied from 4 nm to few micrometers, depending on preparing method. It was concluded that precipitation conditions strongly affect resulting zirconia nanopowder.

16:00 Poster H-25

$ZnFe_2O_4$/ZnO core-shell particles obtained by coprecipitation route

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The paper deals with preparation of nanocrystalline zinc oxide doped with iron oxide obtained via calcination process. Mixture of iron and zinc hydroxides of various composition was used as precursor of nanopowders. The zinc ferrite samples were prepared by wet chemistry method - precipitation from the corresponding metals nitrates solutions using ammonia. The obtained hydroxides were filtered, dried and calcined at 300°C for 1 hour. Series of the samples containing 5 to 95 wt.% of Fe_2O_3 was obtained. The phase compositions of the samples were determined using XRD. Depending on the chemical composition the phases of hexagonal ZnO, and/or romboedric Fe_2O_3 and/or cubic $ZnFe_2O_4$ were identified. The mean crystallite size of $ZnFe_2O_4$ phase present in all the samples, determined using Scherrer's formula, varied from 8 to 13 nm. The morphology of the samples was investigated using SEM and HRTEM. XPS measurements were conducted using Prevac UHV system. X-ray photoelectron spectra of all samples were analysed. Charging was reduced using neutralization cannon. Detailed spectroscopic and morphological analysis of the samples suggests core-shell model of the produced grains.

16:00 Poster H-26

Synthesis of Nanocrystalline hydroxyapatite facilitated by the presence of an oligosaccharide

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Nanocrystalline hydroxyapatite was prepared by chemical synthesis using α and β cyclodextrin (CD) as template. The particles were characterized by Field Emision Scanning Electron Microscope (FE-SEM),X-ray powder diffraction (XRD) Fourier Transform Infrared Spectroscopy (FTIR); The Thermal Analysis was measured by thermogravimetric and differential thermal analysis (TGA-DTA). The size of the particles was reduced considerably with the addition with a little amount of CD (0.5-2%) as consequence of HA-CD interaction. It was showed that nanocrystalline hydroxyapatite can be obtained by chemical synthesis with the presence of CD, without CD only particles of the order of microns were acquiered.

16:00 Poster H-27

Photoluminescence properties of Aluminum oxide Nanoparticles

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We report the photoluminescence (PL) properties of aluminum oxide nanoparticles synthesized in a liquid confined ambient using pulsed laser ablation of aluminum. The colloidal suspension was dried on a quartz substrate using an IR heat lamp prior to further structural and optical characterization. TEM measurement confirms the particles of nano dimension with average size of ~23nm and of spherical shape. XRD measurement shows a strong peak at ~ 31.5^{0} indicating the presence of aluminum oxide. The possibility of oxide formation is strong because of the laser induced breakdown of the water, which used as a confining medium for the plasma. The PL measurement using excitation source of Xenon lamp shows a broad peak which could be resolved using different excitation wavelengths.

The peak position shifts towards longer wavelength as the excitation wavelength is changed to longer wavelengths. The shift may be due to the presence of intermediate compounds like AlO and Al_2O in addition to Al_2O_3 .The amalgamation of some the excited electronic - vibrational bands of the intermediate compounds may contribute to the broad observed PL peak. The appearance of the luminescence bands may be associated with the defects formed in the non equilibrium phases and the oxygen vacancy defects in the sub-lattice which are specific features of the nanostructured state in nanostructures formed during the laser ablation inside the liquid confined ambient. A comparative study of laser ablated aluminum plasma in air and water to elucidate the various processes is also presented.

16:00	Poster	H-28

Nanocrystalline WC with non-toxic binder Fe-Mn

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A cemented carbides, base on the tungsten carbides (WC), are very popular and useful in an industry. The most important metal which is using as a binder in this kind of materials is cobalt. It has many advantages as a binder: very good wettability, favorable solubility with WC and thermal conduction similar to WC. However, costs of cobalt is very height because natural resources aren't sufficient. Cobalt isn't also neutral for health it is known as an allergen and same research shown that it could cause a cancer. This paper presents results of sintering the tungsten carbides with Fe-Mn alloys as the binder.

16:00	Poster	H-29

Study of the mechanical and magnetic properties of $Fe_{61}Co_{10}Zr_{5-x}Hf_xW_2Y_2B_{20}$ (x = 0, 3) bulk amorphous and crystalline alloys

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Bulk amorphous alloys are group of materials with large number of better properties then their crystalline equivalents (like mechanical, environmental etc.). In addition some of these alloys display good soft magnetic properties and high Curie temperature what make them perfect materials for parts of engines and transformers working in bad environmental conditions.

In this work the mechanical and magnetic properties of $Fe_{61}Co_{10}Zr_{2+x}Hf_{3-x}W_2Y_2B_{20}$ (x = 0, 3) bulk amorphous and crystalline alloys were investigated. The crystalline materials were smelted on copper mould using electric arc, their amorphous equivalents were prepared using induction suction method. All investigated samples had shape of 10/10mm length and 0,5mm thickness plates. The density of the samples were derived using Archimedes law by dipping method. The study of the microstructure were performed using X-ray diffraction and mössbauer spectroscopy. The X-ray diffraction were made using cobalt source with moderate wave length 1,79021 Å. The Mössbauer spectra were recorded using the conventional constant-acceleration spectrometer with the ^{57}Co of g-ray source in a rhodium matrix. The spectrometer was calibrated by means of Fe foil. The microstructure measurements proved that samples made by induction suction method have amorphous and arc smelted crystalline structure.

The magnetic measurements were performed using LakeShore VSM, with maximum magnetic field up to 2T. From the analysis of static hysteresis loops it was found that $Fe_{65}Fe_{61}Co_{10}Zr_{2+x}Hf_{3-x}W_2Y_2B_{20}$ (x = 0, 3) amorphous alloy have better soft magnetic properties then their crystalline equivalents.

The mechanical properties of all investigated samples were obtained using micro hardness system FutureTech 710 with 0,2 kG load and wear resistance measurement system "Kulotester" using 20mm balls made from stainless steel. It was shown that amorphous alloys have higher density, micro hardness and better wear resistance.

16:00	Poster	H-30

Study of the microstructure and magnetic properties of $Fe_{60}Co_5Zr_{10}Mo_5B_{20}$ bulk amorphous and partially crystallized soft magnetic alloy.

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During the few past years a great quantity of data has been published about bulk amorphous alloys [1-2]. One of the main studying aims is to find alloys with high glass-forming ability (GFA), purposed for controlled crystallization process to obtain nanocrystalline materials with fine grains proportionally displaced in amorphous matrix. The other aim is to obtain magnetic material with good soft magnetic properties.

In this work microstructure and magnetic properties of $Fe_{60}Co_5Zr_{10}Mo_5B_{20}$ bulk amorphous and partially crystallized alloys has been investigated. All investigated samples were prepared using induction suction method, in a protective atmosphere, as a plates of 1 and 0,5 mm thickness. The study of the microstructure were performed using X-ray diffraction and mössbauer spectroscopy. The magnetic properties were investigated using LakeShore VSM magnetometer and Ferrotester. From analysis of hysteresis loops and initial magnetization curve the magnetic parameters were derived. The Ferrotester was used to determine the dependence of hysteresis losses in function of frequency. All microstructure and magnetic measurement were performed in room temperature. The thermal stability after solidification process and characteristic coefficients of the alloys with amorphous matrix were obtained using differential scanning calorymetry (DSC).

It was shown that presence of few percent of fine grains in amorphous matrix lead to improvement of soft magnetic properties. The coercivity of the sample decrease from 210 A/m to 60 A/m, and saturation of the magnetization rise about 20 kA/m. It was also shown that partially crystallized alloy have lover hysteresis losses then amorphous one.

[1] Akihisa Inoue, Tao Zhang, and Hisato Koshiba; J. Appl. Phys. 83, 6326 (1998); DOI:10.1063/1.367811

[2] M. Xu, M. X. Quana, Z. Q. Hua, L. Z. Chengb and K. Y. Heb; Journal of Alloys and Compounds; Vol 334, Issues 1-2, 28 February 2002, Pages 238-242

16:00 Poster H-31

Amorphous $Ni_{59}Zr_{20}Ti_{16}M_5$ (M = Cu, Ag) alloys obtained by melt spinning and mechanical alloying

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Nowadays Ni-based bulk metallic glasses (BMG) are considered as potential engineering materials due to their good mechanical properties (high strength, relatively low Young's modulus and large elastic limit). Several Ni-based alloys have been reported to show the enhanced glass forming ability (GFA), enabling the preparation of BMG by Cu-mould injection casting. Another possibility of fabrication of amorphous powders is mechanical alloying (MA) method.

The main purpose of this work was to produce amorphous $Ni_{59}Zr_{20}Ti_{16}M_5$ (M = Cu, Ag) alloys by mechanical alloying of a mixture of the powders of pure crystalline elements. The studied alloys exhibit high GFA, however, the compositional ranges of amorphisation for MA and rapid quenching are usually different. Then, the thermal stability of all investigated amorphous alloys were studied and compared.

X-ray diffraction (XRD) and differential scanning calorimetry (DSC) were employed as the experimental techniques for samples characterization, both melt spun ribbons and MA powders at different stages of processing.

16:00 Poster H-32

On phase constitution, microstructure and magnetic properties of the directly quenched $Pr_9Fe_{60}Co_{13}Zr_1Ti_3B_{14}$ alloy

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It was shown in [1] that bulk nanocrystalline Pr-Fe-Co-Zr-Ti-B alloys produced directly by various rapid solidification techniques were hard magnetic in as-cast state. However, values of magnetic parameters depend on the geometric dimensions of the samples. In the present work the changes of microstructure and phase constitution of the $Pr_9Fe_{60}Co_{13}Zr_1Ti_3B_{14}$ alloy samples of various sizes were investigated. The nanocrystalline 1 mm diameter rods and 3 mm outer diameter tubes were produced by suction-casting of the melt into the water cooled copper die under an Ar atmosphere. For comparison the nanocrystalline ribbon in as-cast state of the thickness t_r~140μm, were produced by controlled atmosphere melt-spinning. The X-ray difractometry and quantitative analysis of the Mössbauer spectra, shown differences in phase constitution of the samples of various shapes. Furthermore, the transmission electron microscopy revealed differences in microstructure of the rod, tube and ribbon samples. The aim of the work was to determine the correlations between the cooling rate, microstructure, phase constitution and magnetic properties of the $Pr_9Fe_{60}Co_{13}Zr_1Ti_3B_{14}$ alloy samples.

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[1] P. Pawlik, K. Pawlik, H.A. Davies, W. Kaszuwara, J.J. Wysłocki, N. Harrison, I. Todd, Journal of Alloys and Compounds 423 (2006) 99–101

16:00 Poster H-33

Microstructure, thermal stability and magnetic properties of bulk amorphous $Fe_{73-x}Co_xNb_5Y_3B_{19}$ (x = 0 or 10)

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Iron-based bulk amorphous alloys are interesting for basic studies because they have promising magnetic properties for technical applications. These alloys usually contain more than three elements and show better thermal stability of magnetic properties than classical amorphous ribbons.

In this paper we present the influence of iron replacement by cobalt on the microstructure, thermal stability and magnetic properties of the bulk amorphous $Fe_{73-x}Co_xNb_5Y_3B_{19}$.

Ingots of the alloys were prepared by arc melting of elements in a protective argon atmosphere. Plates 10 mm long with a cross section of about 0.5 mm x 10 mm were obtained by a rapid solidification of molten materials in a copper mold cooled with water (a suction casting method).

The amorphicity of the samples was tested by a X-ray diffractometer with Co K_α radiation and Mössbauer spectrometer. These investigations were carried out at room temperature for the powdered samples. The thermal stability of the alloys was examined by differential scanning calorimetry (DSC). DSC curves were recorded at the heating rate of 10 K/min. The Curie temperature was found from thermomagnetic curves obtained by a force magnetometer. The hysteresis loops and magnetization curves versus the strength of the magnetizing field were measured by a vibrating sample magnetometer.

The as-cast samples were fully amorphous. No traces of peaks originating from crystalline phases were seen in X-ray diffraction patterns and Mössbauer spectra consist of broad and overlapping lines characteristic of the amorphous state. In the DSC curves the pronounced peak at about 900 K corresponding to the primary crystallization is visible. The investigated alloys are soft magnetic materials exhibiting low coercivity and relatively large magnetic saturation polarization. The magnetization process at high magnetic field is connected with rotation of magnetic moments near the structural defects called quasi-dislocation dipoles.

16:00 Poster H-34

Corrosion behaviour of amorphous and crystalline $Fe_{59}Co_{10}Zr_2Y_4Nb_5Ni_5B_{15}$ alloy in acidified sulphate and phosphate environments

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The electrochemical corrosion characteristics are discussed for amorphous and crystalline $Fe_{59}Co_{10}Zr_2Y_4Nb_5Ni_5B_{15}$ alloy in acidified (pH = 3), deaerated, 0.5 M sulphate- and phosphate solutions at 25°C. Potentiokinetic polarisation curves have been measured for both structural states of the tested material and compared with those determined for pure, crystalline iron. Both in acidified sulphate- and in phosphate solutions the alloy corrodes in active state and its anodic external currents are clearly smaller than those for pure iron. The anodic active dissolution of the amorphous alloy occurs *ca* one order of magnitude slower than that of the crystalline alloy. The effect of strong inhibition of corrosion in passive state is especially distinct for amorphous alloy in phosphate solution. In spite of considerable presence of cobalt, the amorphous alloy shows distinctly lower anodic currents in passive state as compared to pure iron. The beneficial role of alloy amorphisation in corrosion behaviour can be explained in terms of limiting number of structural deffects and lack of grain boundaries in amorphous state. The alloy amorphous structure favours better continuity of oxide layers formed in passive region and decreases number of active sites in active range.

16:00 Poster H-35

Nanocrystallization of Al-based glasses via nucleation and growth under "soft impingement" conditions

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The nanocrystallization behaviour of $Al_{86}(TM)_8(RE)_6$ (where TM – Ni,Co,Fe; RE – Gd,Y) amorphous alloys has been studied by X-ray diffraction methods involving small angle scattering (SAXS), resistance measurements and differential scanning calorimetry. It has been established that the transformation in amorphous alloy investigated occurs mainly by the growth process at initial state of transformation while at final stage nucleation contributes into formation of nanocomposite structure. In order to describe the nanocrystallization kinetics Al- based glasses the analytical kinetic equations in the frames of Kolmogorov-Johnson-Mehl-Avrami model have been proposed and the effective volume diffusion coefficients values which govern the diffusion-limited growth nanocrystals have been determined.

16:00 Poster H-36

Influence of Mn on the magnetocaloric effect in Nanoperm-type alloys

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Magnetic refrigeration is currently gaining an increasing interest due to the discovery of materials with remarkable magnetocaloric response close to room temperature. Among others, the main advantages of the refrigerators based on the magnetocaloric effect (MCE) with respect to the systems based on the compression-expansion gas cycle are an improvement in the energetic efficiency and the avoidance of ozone depleting and green-house effect gasses. The characterization of the magnetocaloric materials is based on two parameters: the peak of the magnetic entropy change (ΔS_M^{pk}) and the refrigerant capacity (RC). The study of amorphous materials for use in magnetic refrigeration has been motivated by finding a compromise between the aforementioned parameters: a modest ΔS_M^{pk} with a high RC. These quantities together with their reduced magnetic and thermal hysteresis, high electrical resistivity, acceptable mechanical properties and corrosion resistance make them good candidates as magnetic refrigerators. The aim of this work is to study the influence of the manganese content on the magnetocaloric response of the $Fe_{80-x}Mn_xB_{20}$ alloy series (x = 10, 15, 18, 20 and 24). The magnetic entropy change has been calculated from the numerical derivative of the magnetization curves with respect to temperature and subsequent integration in field, and the RC have been obtained from the product of ΔS_M^{pk} times the full temperature width at half maximum of the peak (RC_{FWHM}). The results indicate a nearly linear dependence on the manganese content of the Curie temperature Tc, the maximum magnetic entropy change ΔS_M^{pk}, and the magnetic refrigeration RC_{FWHM}, with slopes of -20 K/at. % Mn, -0.04 $J \cdot K^{-1} \cdot kg^{-1}$/at. % Mn and □3.5 $J \cdot kg^{-1}$/at. % Mn, respectively.

16:00 Poster H-37

Influence of rare earth (Gd, Dy or Sm) dopants on the magnetic properties and microstructure of $Nd_{60-x}RE_xFe_{30}Al_{10}$ alloys

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The effect of partial substitution of Gd, Dy and Sm for Nd in $Nd_{60-x}RE_xFe_{30}Al_{10}$ alloys for the magnetic properties and microstructure was studied. Each of the elements used has different influence on the magnetic properties of the (Nd, RE)-Fe-Al alloys. Gd substantially increases the coercivity, even at as high concentrations as 20%. Addition of Gd does not change neither the initial magnetiz-

ation curve nor the initial susceptibility. Similarly high coercivity increase can be achieved by addition of up to 5% of Dy. For higher Dy contents a dramatic drop of the coercivity was observed. Introduction of Sm leads to deterioration of the magnetic properties, even at as small concentrations as 1%. All the additional elements studied decrease the saturation magnetization and remanence. Additions of Dy, Gd and Sm, which partly substitute Nd in the $Nd_{60}Fe_{30}Al_{10}$ alloy, leads to formation of crystalline precipitates. In the case of Dy these precipitates are formed by $MgCu_2$ type phase. In the Sm doped alloy the size of the crystalline precipitates, visible in the amorphous matrix, are about twice of those, which are present in the dopant free alloy.

16:00 Poster H-38

Microstructure of rapidly solidified Nd-Fe-Al alloys

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Hard magnetic, rapidly solidified Nd-Fe-Al alloys have been studied since 1996 y. Nevertheless, a uniform, widely accepted structural model of these alloys still does not exists. The phase constitution of these alloys consists of nanostructured crystalline phases and amorphous phases. Another difficulty of structural characterisation comes from inhomogeneity of the material in respect to the chemical composition and phase constitution. The phase constitution may change along the cross section of the material due to the gradient of quenching rate. Inhomogeneities of the chemical and phase composition are also observed in areas as small as few micrometers. All these factors make interpretation of the data of structural investigations on the nanometeric level very difficult, especially for the material state of which is fare from equilibrium.

In the current work microstructure and local chemical composition were studied for the $Nd_{60}Fe_{30}Al_{10}$ alloy. The analyses were done on the nanometric level (HRTEM) and also completed with the studies with application of SEM and Mossbauer spectroscopy. The specimens studied were either in the form of 1 mm rod or rapidly solidified ribbon, obtained with the roll speed of 30 m/s. It has been shown that the alloy contains in its volume nanometric size crystallites, partly agglomerated and exhibiting crystallographic texture. Size of the crystallites depends on the quenching rate, which also influence the composition of the amorphous phase. The HRTEM studies revealed that the crystalline grains are frequently separated by narrow layer of intermediate phase.

16:00 Poster H-39

Structure and approach to magnetic saturation in the $Fe_{36}Co_{36}B_{19}Si_5Nb_4$ bulk amorphous alloy

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The idea of the paper is to study the structure and approach to magnetic saturation in the $Fe_{36}Co_{36}B_{19}Si_5Nb_4$ bulk amorphous alloy in the shape rods with diameter of 1 and 2 mm. Investigated material were produced by the pressure die casting method. The ingot alloy was prepared by repeatedly inductive melting under a protective argon atmosphere using high purity elements. Subsequently, liquid melt was push out from quartz capillary to suitable rod forming copper die with using inert gas. The structure of obtained samples in solidified state was examined by X-ray diffraction (XRD) method and Mössbauer spectrometry.

From these studies we have stated that samples are fully amorphous. The regions with low and high density in the bulk amorphous alloys occur and due to magnetoelastic coupling influence the magnetization behavior in the vicinity of the ferromagnetic saturation. Any departure from the atoms system in liquid state be in thermodynamic equilibrium can be considered a defect in amorphous material [1].

Defects occurs in studied bulk ferromagnetic amorphous materials presenting source of short range stresses were investigated by indirectly method. These method was based on measurement of magnetization in high magnetic fields [2-4] by a vibrating sample magnetometer (VSM). In accordance to [1-4] magnetic polarization in high magnetic fields can be described by the equation:

$$m_0M(H)=m_0M_S[1-\Sigma_i(a_i)/(\mu_0H)^i]+b(\mu_0H)^{1/2},$$

where:

m_0M_S - magnetic saturation polarization

μ_0 - magnetic permeability of free space,

H – magnetic intensity,

M_S – magnetization intensity,

a, b – coefficents.

On the basis of results obtained by VSM method in high magnetic field it was observed that the diameter of rods (of the amorphous materials) has significant influence on size of defects. For all diameters of the analyzed alloy, important role in magnetization process in the vicinity of the ferromagnetic saturation plays sets of conglomeration of point-like defects, referred as quasi-dislocation dipoles. For magnetic field above 1T, where the relation $(\mu_0H)^{1/2}$ is valid for every diameter of the rod made of $Fe_{36}Co_{36}B_{19}Si_5Nb_4$ alloy, the Holstein – Primakoff paraprocess connected with damping of thermal activation spine wave [5] is observed.

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16:00 Poster H-40

Thermal stability and soft magnetic properties of $Fe_{61}Co_{10}Zr_{2,5}Hf_{2,5}Me_{4-x}W_2B_{20}$ (Me = Nb, Mo, Ti, Y, Ni) bulk amorphous alloys obtained by induction suction method.

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The bulk amorphous alloys based on iron or cobalt display good soft magnetic properties (low value of coercivity and high saturation of the magnetization). These features lead to wide range of application of these materials. In this work the microstructure, thermal stability and magnetic properties of $Fe_{61}Co_{10}Zr_{2,5}Hf_{2,5}Me_{4-x}W_2B_{20}$ (where Me = Nb, Mo, Ti, Y, Ni) bulk amorphous alloys were investigated. Samples were obtained using induction suction method as a 0,5 mm thickness and 1 cm^2 area plates. The study of the microstructure were performed using X-ray diffraction and mössbauer spectroscopy. It was shown that the $Fe_{61}Co_{10}Zr_{2,5}Hf_{2,5}Me_{4-x}W_2B_{20}$ (Me = Nb, Mo, Ti, Y, Ni) alloys have amorphous structure in all volume of the samples. The magnetic properties were investigated using LakeShore VSM magnetometer. From analysis of hysteresis loops magnetic parameters were derived. It was found that coercivity field is less then few teens A/m and saturation of the magnetization is about 106 A/m for all measured samples. Thermal stability of the investigated alloys were obtained using differential scanning calorymetry (DSC). From DSC plots several characteristic temperatures like T_g the glass transition temperature, T_x the crystallization temperature, $\Delta T_x = T_x - T_g$ extent of the supercooled liquid region, T_1 the primary crystallization temperature, T_2 the secondary crystallization temperature, $\Delta T_l = T_l - T_x$ undercooled temperature, T_m the melting temperature, T_l the liquidus temperature, $T_{rg} = T_g / T_l$ reduced glass transition temperature, $\Delta T_m = T_l - T_m$ interval temperature, modified GFA parameter defined as $\gamma m = (2T_x - T_g)/T_l$ were derived. For all measured samples reduced glass transition temperature parametr T_{rg} have values higher then 0,6 and GFA parameter γ_m higher then 0,7 what prove good glass formability of investigated alloys.

16:00 Poster H-41

Study of the magnetic interaction in nanocrystalline Pr-Fe-Co-Nb-B permanent magnets.

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The magnetic properties of an isotropic, epoxy resin bonded magnets made from Pr-Fe-Co-Nb-B powder produced by using the rapid solidification method were investigated. The magnetization reversal process and magnetic parameters were examined by measurements of virgin magnetization curve, major and minor hysteresis loops and sets of recoil curves using LakeShore VSM mounted in 2T electromagnet. The virgin magnetization curve was used in order to determine the differential susceptibility as a function of an applied magnetic field. From the minor hysteresis loops the field dependencies of the coercivity and remanence were derived. It was found that the magnetization reversal process is the combination of irreversible pinning of domain walls and reversible rotation of magnetization vector in single domain grains.

16:00 Poster H-42

The effect of heat treatment on the phase constitution and magnetic properties of the $Pr_9Fe_{60}Co_{13}Zr_1Ti_3B_{14}$ alloy ribbons

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Fully amorphous samples of the $Pr_9Fe_{60}Co_{13}Zr_1Ti_3B_{14}$ alloy were produced by the melt-spinning technique under an Ar atmosphere. In as-cast state the ribbon samples were soft magnetic with the coercivity field $_JH_c$ of ~ 10 A/m and saturation magnetization J_r of ~1 T. Subsequent annealing at various temperatures T_a (from 923K to 973K for 10 min), resulted in evolution of the phase constitution together with significant change of their magnetic properties. X-ray diffractometry (XRD) revealed presence of the hard magnetic $Pr_2(Fe,Co)_{14}B$ phase precipitated during annealing. However, the quantitative analysis of Mössbauer spectra, shown multiphase constitution of the alloy with relatively low volume fraction of this crystalline phase. Furthermore, the temperature dependences of the magnetization confirmed differences in constitution of the samples annealed at various temperatures. The room temperature measurements of the hysteresis loops indicated very good hard magnetic properties of annealed ribbons. The coercivity field increased from 28 kA/m to 1360 kA/m for the ribbons annealed respectively at 923K and 973

K.

The aim of present work was to determine correlations between the phase constitution and magnetic properties of ribbon samples.

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16:00 Poster H-43

Phase decomposition and some magnetic properties in bulk amorphous multicomponent Fe based alloys

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Bulk amorphous samples consist of more than three elements and are prepared by a rapid solidification of melted materials in a copper mold or on rotating roller. In order to obtain bulk amorphous alloys atomic radii of main components should differ more than 12 % and exhibit negative heat of mixing. Depending on the preparation method, they have a form of rods, tubes, plates, cores of different shapes or ribbons with the thickness larger than 40 µm. The bulk amorphous alloys are obtained at a relatively low quenching rate and it is possible decomposition of the amorphous structure during their preparation.

The aim of this paper is to study the microstructure and magnetic properties of the multicomponent $Fe_{59}Co_{10}Zr_2Y_4M_5Ni_5B_{15}$ (where M denotes late transition metals such as Mo, Ti or Nb). Ingots of the alloys were obtained from high purity elements by arc melting. To obtain homogenous alloys, the ingots were remelted four times. The samples in the form of rods 1 mm in diameter and 2 cm long were prepared by a suction casting method in a protective argon atmosphere. The microstructure of the alloys was studied using X-ray diffractometer and Mössbauer spectrometer. However, the magnetic properties i.e. saturation magnetization, coercive field and Curie temperature were obtained from measurements performed by a vibrating sample and force magnetometers. From X-ray and Mössbauer studies it was stated that samples are fully amorphous but show inhomogeneous structure with regions with different concentration of magnetic elements. From the thermomagnetic curves two Curie temperatures corresponding to the different magnetic phases can be obtained. The investigated alloys exhibit relatively high effective magnetic saturation polarization which is equal to about 1 T at room temperature. The coercive field determined from the hysteresis loops indicates that the investigated alloys are relatively magnetically hard.

16:00 Poster H-44

Lean neodymium Nd-Fe-B magnets containing minor addition of titanium

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The effect of titanium addition on the structure and magnetic properties was studied for nanocomposite $Nd_8Fe_{78-x}B_{14}Ti_x$ and $Nd_7Fe_{79-x}B_{14}Ti_x$ systems. The alloys were rapidly solidified by melt-spinning with the roll speed of 20 m/s. The overquenched, amorphous ribbons were subsequently crystallization annealed at 680-700 ºC for 20 min. It was found that addition of 2 and 4 at% of Ti leads to substantial increase of the coercivity and maximum energy product of the nanocomposite magnets while maintaining the remanence unchanged. The highest properties were obtained for the alloys containing 4 at% of Ti. The maximum energy product of 122 kJ/m^3 was obtained for the $Nd_8Fe_{74}B_{14}Ti_4$ alloy. The x-ray phase analysis revealed a presence of the hard magnetic $Nd_2Fe_{14}B$ and soft magnetic phases (Fe, Fe_2B) in all the alloys studied. For the alloys containing Ti additional boride phases, such as: TiB_2 could also be detected. More detailed information on the phase constitution was obtained from the Mössbauer spectroscopy. Comparison of the Ti-free and 4 at% Ti alloys revealed that the amount of the hard magnetic $Nd_2Fe_{14}B$ phase grows up from 66,60% to 82,82% for $Nd_8Fe_{78-x}B_{14}Ti_x$ system and decreases from 84,53 % to 69,88% for the $Nd_7Fe_{79-x}B_{14}Ti_x$ system. The TEM studies showed substantial differences in the morphology of the alloys. The NdFeB alloys contain grains of various sizes about hundred nanometers. The alloys containing 4 at% of Ti exhibit more homogeneous structure with the crystal sizes of up to 40 nm.

16:00 Poster H-45

Characterization of TiO_2 thin films deposed on Ti and SnO_2 (F) by a dip-coating MF.BENCHARIF, A.CHARI, T.KERBACH, E.TAYAR abelhamidchari@gmail.com benmedfa@gmail.com

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TiO_2 thin films have been obtained by a dip-coating method on Ti and SnO_2 (F). Structural and physicals properties of the films have been examined for different number of dips and annealing at a temperature of 723 K.

Scanning Electron Microscopy (SEM) and X rays diffraction (XRD have been used to characterize the films morphology and composition. These films are anatase phase and nano-metric with a grain size of 18 to40 nm.

The photo catalytic activity of the nano-structured TiO_2 thin films

were evaluated by the degradation of solo phenyl red 3BL and compared with that obtained using P25 TiO_2.

16:00 Poster H-46

Characterization of nanocrystalline Ni surface layers electrodeposited in Watts bath with additions of ionic liquids

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The aim of the present paper is to report results of performed investigations on the influence of additions of different quantities of ionic liquids into Watts bath on properties of nanocrystalline Ni layers prepared by the electrodeposition method. The nanocrystalline nickel layers were electrodeposited in Watts bath with saccharine and different ionic liquids additions. The results of analyses of the topography and morphology and hardness properties of nanocrystalline Ni layers prepared with and without liquid ions additives are also presented. The structural analyses are performed by using the X-Ray diffractometer PW -1830. The morphology and topography of produced layers were performed by scanning electron microscopy S-3600N and transmission electron microscopy JOEL 1200. Results of performed investigations have showed that the introduction of ionic liquids into the bath leads to important improvements of mechanical properties of the nanocrystalline Ni surface layers produced by the method of electrocrystalizations

16:00 Poster H-47

Structure and properties of thermal sprayed coatings

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Nowadays the coating technologies are more often applied for covering surfaces of intermediate products and industrial goods. The plasma sprayed technologies are very useful which do not require expensive devices such as example PVD and CVD methods. Plasma sprayed thermal coating is the method which produces multi-layers with the micrometer size of microstructure elements. The microstructure diminishing of plasma sprayed coats is possible by using the innovative methods as High Velocity Oxygen Fuel (HVOF) process. Nanometric structure of layers considerably improved the coating properties. The main aim of the work is investigations of the HVOF carbide Cr_3C_2 coats and its comparison to the plasma sprayed layers. The multilayer coats consisted from two layers: intermediate NiAl layer and the external Cr_3C_2 layer were prepared at the Al-Si substrate. The increase of microhardness HVOF coats with the nanometric features was found in comparison to the plasma sprayed coats with the micrometric structure.

16:00 Poster H-48

Effect of palladium addition on the electrochemical properties of an amorphous 2Mg+ 3d alloys doped by nickel atoms (3d = Fe, Ni)

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Magnesium-based alloys are promising hydrogen storage materials because of their high storage capacity and low cost. It was found that the electrochemical activity of nanocrystalline hydrogen storage alloys can be improved in many ways, by alloying with other elements, by ball-milling the alloy powders with a small amount of nickel or graphite powders. The surface modification of nanocrystalline hydrogen storage alloys with graphite by ball-milling leads to an improvement in both discharge capacity and charge-discharge cycle. In this work, an amorphous 2Mg+ 3d/x wt% Ni materials were prepared by mechanical alloying (MA) of Mg and 3d elemental powders (3d = Fe, Ni x=0, 100 and 200%) under high purity argon atmosphere in SPEX 8000 Mixer Mill. The effect of the Ni addition on the electrochemical properties of the synthesized nanostructured alloys was investigated in details. The discharge capacity of amorphous 2Mg+Fe (Ni) high energy ball milled with Ni was improved. With increasing nickel content in the studied 2Mg+Fe (Ni) materials, at first cycle, the discharge capacity increases first and then decreases, and for x = 100 in 2Mg+Fe reaches a maximum value of 155 mAh g^{-1}. The elemental nickel was distributed inside of mechanically alloyed Mg-Fe (Ni) particles homogenously. It is generally agreed that the role of nickel particles is to catalyze the electrochemical reaction and/or reduce the diffusion resistance of hydrogen. Additionally, when coated with palladium, the discharge capacity of an amorphous 2Mg+Fe (Ni) powders was increased. The elemental palladium was distributed on the surface of ball milled alloy particles homogenously and role of these particles is to catalyse the dissociation of molecular hydrogen on the surface of studied alloy. Mechanical coating with palladium effectively reduced the degradation rate of the studied electrode materials. Compared to that of the uncoated powders, the degradation of the coated was suppressed. The amorphous Mg-based hydrides offer a breakthrough in prospects for practical applications.

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16:00 Poster H-49

Hybrid Ti-ceramic bionanomaterials for medical engineering

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In the last decade a great interest has been observed in the field of nanoscale materials. Commercially pure titanium as well as titanium alloys have become predominant in implantology. Low hardness and poor tribiological properties of titanium alloys may become critical factor when wear phenomena are involved. One of the methods that allow the change of properties of Ti alloys is to the production of nanocomposite, which will exhibit the favorable mechanical properties of titanium and excellent biocompatibility and bioactivity of ceramics. In this work hybrid Ti-*x* vol% ceramic (hydroxyapatite (HA), 45S5 Bioglass (SiO_2-45 wt%, P_2O_5-6 wt%, CaO-24.5 wt%, Na_2O-24.5 wt%), SiO_2, Al_2O_3) bionanocomposites (*x*=0, 3 and 10) were produced by the combination of mechanical alloying (MA) and powder metallurgical process. The average size of produced nanocomposites was about 30 nm. Reinforced by HA, 45S5 Bioglass, SiO_2 or Al_2O_3 particles, Vickers hardness of Ti-based nanocomposite is higher from two to five times from that of pure microcrystalline Ti. Additionally, the experimental results show that in Ringer's solution at 37°C, Ti-based nanocomposites have good corrosion resistance. For example, the Ti-10 vol% SiO_2 nanocomposite possesses higher corrosion resistance and thus higher corrosion current densities ($I_c = 3.74\cdot10^{-8}$ A/cm^2) than microcrystalline titanium ($I_c = 1.31\cdot10^{-5}$ A/cm^2). On the other hand, our results of *in vitro* studies show that these bionanocomposites have excellent biocompatibility and could integrate with bone.

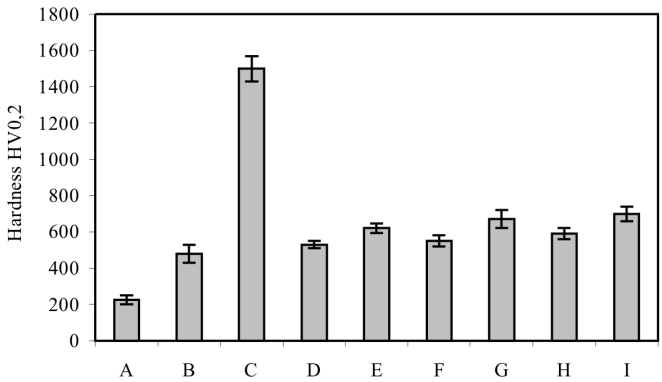

Fig. 1. Mean values of hardness of studied Ti-ceramic nanocomposites:

A - Ti (microcrystalline),
B - Ti-3 vol% HA,
C - Ti-10 vol% HA,
D - Ti-3 vol% 45S5 Bioglass,
E - Ti-10 vol% 45S5 Bioglass
, F - Ti-3 vol% SiO_2,
G - Ti-10 vol% SiO_2,
H - Ti-3 vol% Al_2O_3,I - Ti-10 vol% Al_2O_3.

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16:00 Poster H-50

Hydriding properties of Mg-3d/M type nanocomposites (3d=Cu, Ni; M=C, Ni, Cu, Pd)

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Magnesium-based hydrogen storage alloys have been also considered to be possible candidates for electrodes in Ni-MH batteries. In this work, we have studied experimentally the structure and properties of Mg_2Ni(Cu)/M-type hydrogen storage nanocomposite materials (M=C, Ni, Pd). These materials were prepared by mechanical alloying (MA) followed by annealing and ball milling, respectively. In nanocrystalline Mg_2Cu powder, discharge capacities up to 26 mA h g^{-1} were measured. It was found that nickel substituting copper in Mg_2(Cu-Ni) alloy greatly improved the discharge capacity of studied material. In nanocrystalline Mg_2Ni powder, discharge capacities up to 100 mA h g^{-1} were measured. Additionally, mechanically coated Mg_2Ni-based alloys with graphite, nickel, copper or palladium have effectively reduced the degradation rate of the studied electrode materials. They show substantially enhanced cycle life, at room temperature. Results also showed that the strong modifications of the electronic structure of the nanocrystalline alloys could significantly influence on their hydrogenation properties.

16:00 Poster H-51

Synthesis and photocatalytic properties of the compounds in the system Ag_2O-WO_3

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Photocatalysts with higher photocatalytic performance under visible light irradiation have been desired for many applications, such as water and air purification, solar cell and water splitting into H_2 and O_2. In the present study, $Ag_2W_2O_7$ and Ag_2WO_4 are synthesized and investigated. Ag_2O (Kanto Chemical: 99.0%) and WO_3 (Kanto Chemical: 99.0%) were mixed with ethanol in a stoichiometric ratio. The mixtures were pressed into pellets at 400kg/cm^2 and calcined at temperatures from 400 to 650□ in air for 20h. The calcines were grounded and pelletized again for heating at the same calcination temperatures from 400 to 650□. Characterization of the compounds was carried out by XRD, SEM. Photocatalytic activity of the compounds was evaluated from measurements of decomposition of mechylene blue and acetaldehyde under irradiation of black light, fluorescent light and in dark. For the methylene blue decomposition ex-

periment, 0.10g of photocatalyst was suspended in 50 mL mechylene blue solution with concentration 10mg/dm^3. And for acetaldehyde decomposition experiment, 0.5g of photocatalyst was put in pyrex glass container with 1000 cm^3 in volume. And acetaldehyde was injected into the container with microsyringe so that concentration of acetaldehyde became 40 ppm. The solution and the container were exposed to irradiation of lights for various times.

XRD measurement revealed that $Ag_2W_2O_7$ and Ag_2WO_4 were synthesized as single phase by calcination at temperatures from 450 to 650□ and at 400-600□, respectively. $Ag_2W_2O_7$ synthesized at 600□ and 550□showed excellent methylene blue decomposition performance under irradiation of black light and fluorescent light. The compounds decomposed acetaldehyde from 40ppm to 18ppm and from 40ppm to 28ppm under black light and fluorescent light irradiations, respectively. Ag_2WO_4 showed poor performance for methylene blue decomposition. The compounds decomposed acetaldehyde from 40ppm to 28ppm and from 40ppm to 29ppm under black light and fluorescent light irradiations, respectively.

16:00 Poster H-52

Molecular dynamics study on different melting behaviours of Cu_N(N=51-53) clusters with icosahedron-based geometry

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Melting studies of metal nanoclusters with three to thousands of atoms has been grown steadily for a long time due to their key roles in constructing new electronic nanodevices and novel nanocatalysis materials, and these studies have provided a deep understanding of thermodynamical behaviors in these systems[1-5]. Owing to the high surface to volume ratio for these nanoparticles, melting behaviors present their own special characteristics, and issues pertaining to melting differences in small size clusters containing tens of atoms with icosahedron-based geometry continue to be a subject of current interest.

The structural changes of three Cu clusters respectively containing 51-53 atoms during their melting processes have been studied by employing molecular-dynamics simulations. The local structures in the atomic density shells are presented according to the pair index of Honeycutt and Andersen[6,7]. Structural transformation temperatures of the three clusters are increased with increasing the cluster size. Owing to structural differences in these three clusters, their different melting behaviors can be observed. The simulations provide the implications for us to understand the effect of atomic packing in these clusters with icosahedron-based geometries on causing the structural change differences at elevated temperature.

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16:00 Poster H-53

Featurs Of electric Properties Of nanocomposites On The Basis Of Copper Sulfide and Polyvinil Alcohol

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By means of successive ionic layer adsorption and reaction (SILAR) have been received nanoparticles copper of sulphide in volume of a polymeric matrix. As a matrix polyvinyl alcohol(PVA) has been used. Composites with different growth cycles (different size of particles) have been obtained. Research changing of weight of films CuS:PVA from quantity of cycles has shown that at initial cycles weight of films change considerably. After 3 growth cycles change of weight of films depending on quantity of cycles becomes insignificant. This results showed, that at initial cycles the film volume is filled with nanoparticles. At the further increase of quantity of cycles growth process occurs basically in surface region areas.

Dependence of dielectric permeability and specific resistance for different samples depending on frequency has been investigated. Results of researches have shown that at initial cycles (1-3 growth cycles) specific resistance of samples decreases. The further increase in quantity of cycles leads to increase in specific resistance of samples.

Research of distribution of particles in cross-section of films, has shown, after 3 growth cycles there is a redistribution of particles. There is a migration of nanoparticles from volume on surface area. As a result of such migration resistance in a cross-section direction increases

16:00 Poster H-54

Ab-Initio Modeling of a-Si and a-Si:H

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Amorphous silicon (a-Si) has several important technological applications. The simulation of a-Si is complicated because there is no direct experimental data, as there are for crystals. Our approach to simulate a-Si is to build several small samples, which can easily ran in a personal computer and the calculated properties are statically obtained.

We applied the Wooten, Winer and Weaire algorithm to build 15 samples of 64 atoms supercells of continuous random network of silicon. For each supercell volume and atomic relaxation was allowed in order to minimize the total energy, using a density functional-

pseudopotential code.

The radial and angular distributions, the electronic and vibrational density of states (VDOS), the Raman and the Infrared spectra were calculated. The radial distribution agrees very well with experimental data. The experimental positions and relative intensities of the Transverse Optical (TO) and Transverse Acoustic of the VDOS are well reproduced, with 14 and 25 cm^{-1} peak deviations, respectively. The shape of the experimental Raman spectra agrees well with experimental data, being the intense TO peak shifted by 50 cm^{-1}. The TO width at half-weight is very well reproduced.

When hydrogen passivates the dangling bonds and, initially positioned in the middle of floating bonds, their vibrational frequencies agree very well with experimental data.

16:00	Poster	H-55

TEM investigations of the nanostructure, multilayer CrN/Cr coatings obtained by vacuum-arc method

Piotr Wieciński[1], Halina Garbacz[1], Jerzy A. Smolik[2], Krzysztof J. Kurzydlowski[1]

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The aim of this study was to characterized of the nanostructured, multilayer coatings (CrN/Cr)x8 on Ti6Al4V titanium alloy obtained using vacuum-arc surface engineering method. The investigations were focused on microstructure, chemical and phase composition and crystallographic orientation of the CrN and Cr sub-layers. The special attention has been paid on the transition layer between CrN and Cr phases. This layer, several tens of nanometer in thickness, ensures good coherence, adhesion, wear and corrosion resistance of the CrN/Cr multilayer coatings. The samples for the observation have been prepared using Focus Ion Beam (Hitachi FB2100) microscope followed by low-energy argon ion polishing using Gentle Mill Technoorg Linda to achieve desired film thickness and artifact- and damage-free surface. The microstructure investigations have been performed using scanning electron microscope SEM/STEM Hitachi 5500, transmission electron microscope JEOL 1200EX and Cs-corrected dedicated STEM Hitachi HD2700. The thickness of the nanostructure, multilayer coating CrN/Cr was of 8,5 µm. These composite coatings consist of 8 sub-layers of CrN 700 nm thick and 8 sub-layers of Cr 400 nm in thickness. The performed investigations will help to understand the structure – properties relationship, which can be crucial in designing and modeling nanostructure, multilayer coatings CrN/Cr.

Symposium I

Multifunctional Advanced Composite Materials: manufacturing, structure, properties, characterization

Welcome

Composite materials have been developed to meet increasing demands for materials possessing a broad array of desirable properties. In general, the reinforcing material and the matrix material comprise any combination of metals, ceramics, polymers, nanomaterials. The utility and versatility of composites has resulted in their use within a wide variety of applications, ranging from aircraft and marine structural components to sporting goods.

The development of materials science during last 50-60 years is inseparably linked with the creation of composite materials consist of two or more distinct components with interfaces to be distinguished by chemical composition, structure and physical and mechanical characteristics but at the same time together forming the new structure of material with the principally new useful complex of physical and mechanical and performance properties.

Only on the base of composite materials (natural, for example, metal eutectic and synthetic such as reinforced heterogenous inorganic and organic materials) it became possible the production of high temperature and high-strength details and constructions with high thermal and electrical conductivity, unique magnetic and tribotechnical properties. Composite materials can be characterized by various complex of functional properties. Manufacturing of the products from composite materials is realized by the means of solid and liquid phase technologies, chemical and physical sputtering and various ways of deposition and etc.

The program of our symposium will cover the detailed discussion of the problems concerning the composite materials development and the manufacturing of the products from these materials: from the theory to the ways of their practical realization.

Organisers

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Proceedings

Powder Metallurgy and Metal Ceramic (Springer)

Sponsors

- **National Academy of Sciences of Ukraine**
- **Institute for Problems of Materials Science of NASU**
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Programme

Monday, 14 September

Lectures
Monday afternoon, 14 September, 14:00
Room 219

14:00	Invited oral

Multi-layer composites as perspective structural and functional materials

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The major properties of multi-layer composites as perspective structural and functional materials are reviewed in connection with the geometrical peculiarities of their structure. The structural-geometrical classification of one-dimensional composites is reduced. Heat conductivity and thermal expansion of laminated ceramic-metal composites taking in account the "heritable" thermal stresses are reviewed and the conditions of dividing into layers on inter-laminar boundaries are analysed. The special attention is given to anisotropy of laminated structures. The methods of calculation of effective values of electrical, thermal and elastic properties of one-dimensional composites along and across of layers are described, the estimations of top and bottom values of properties are reduced, the cases of deflection from an additivity of these properties are analysed. A special attention is paid to physical properties of multi-layer composites

with nano-scale thickness of layers. Some features of fracture of ceramic-metal composites at static and impact loading and the perspectives of their using as impact-resistant and thermally stable refractory materials for parts of constructions under extreme thermal and mechanical conditions are reviewed.

14:30	Invited oral

State -of-Art in ICT applications for nanomaterials and technologies of nanomaterials education

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Research and development in nanomaterials and technologies of nanomaterials involves manipulating and controlling individual atoms and molecules to model, simulate, design and create new materials, nano-machines, and nano-devices for application in all aspects of our lives. This branch belongs to that where strong academic background of researchers and engineers, new ideas and intellectual efforts are the most crucial factors of the overall success of innovations.

To prepare future professionals for emerging opportunities in nanomaterials and technologies of nanomaterials academic communities shall provide students an interdisciplinary education to with a broad understanding of natural, engineering and information sciences, develop methodology for integrating nanomaterials and technologies of nanomaterials education into mainstream curricula as well as offer programs for refresher training to specialists and instructors.

All above-listed problems can't be solved without wide usage of ICT. As well as distant forms of education. National Science Foundation (USA) estimated that by the year 2015 there will be a need for 2 million workers worldwide in the fields of nanoscience and nanotechnology. According to Academician Y.D.Tretiakov this number is higher - about 5-6 millions.

This "army" needs to be given second education or to be provided with advanced training that can't be implemented in such volume without distant forms of education. But also "first education" that physician students, material scientists etc. will get today in universities in the field of nanomaterials and technologies of nanomaterials can't be provided without ICT, without Internet-resources, without electronic libraries.

The report gives an overview of ICT (information and communication technologies) applications which can be used effectively both for education (and for research activities) in nanomaterials and technologies of nanomaterials in different countries and world regions.

15:00	Oral

Scientometric study of composite materials: global trends, current status and future outlook.

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Global trends in development of technologies of production and synthesis and methods of investigations and fields of applications for various groups of composite materials such as metal and polymer and ceramic matrix composites and carbon containing composites have been analyzed.

The results of scientometrics investigations of composite materials to be produced and investigated in various institutes of Europe, America and Asia have been presented. The investigations is based on INSPEC database of the Institute of Electrical and Electronics Engineers and SCOPUS database (Elsevier). The composite materials publications have been analyzed with respect to the most productive authors, research organizations, countries of authors, and the leading journals.

COFFEE
Monday afternoon, 14 September, 15:30
Main Hall

Joint Poster Session
Monday & Wednesday
Monday afternoon, 14 September, 16:00
Main Hall

Tuesday, 15 September

Lectures
Tuesday morning, 15 September, 9:00
Room 219

9:00	Invited oral

On magnetic behaviour of new bio-metamaterials

Oksana Kasyutich[1], Johan Van Lierop[2], Walther Schwarzacher[1]

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Three-dimensional (3D) superstructures of individual magnetic nanocrystals that form artificial solids provide a new way to study collective behaviour. These innovative magnetic metamaterials are the basis of a new paradigm in magnetism where a nanoparticle can be considered to be the "atom" of a crystalline solid. Clear evidence of this is provided by comparing the magnetism of a disordered and 3D FCC ordered array, both made with the same nanoparticle "atom".

Recently [1] we have demonstrated a successful route to make 3D crystals of nanoparticles of magnetite. We use cage like proteins to carry nanoparticles and to crystallize them into periodic 3D array. We studied [2] the structure of these bio-metamaterials in comparison with non –crystalline counterpart and have shown that the interparticle (centre-to-centre) separation is different in both systems by only 0.5 nm. Thus it is a viable to suggest that the only structural difference between two studied systems is special ordering of nano-

particles in the FCC array in comparison to absence of long range order in the non-crystalline closed packed assembly.

The results reported here show a dramatic change in coercivity and remanence when short range order/disorder is replaced by long range FCC-order and there is only a small change in interparticle separation. The magnetization measurements of the disordered nanoparticle system reveal typical nanomagnetism. Dynamical freezing of the single domain magnetization when cooling from temperatures where the nanoparticles are superparamagnetic through to the nanoparticle magnetization being blocked (around 50 K) is observed. When the nanoparticles are superparamagnetic, no coercivity is observed (as expected). By contrast, the magnetism of the 3D periodic array of nanoparticles exhibits no dynamical freezing and coercivity and remnant magnetization are measured at all temperatures from 2 to 400 K, manifesting dipolar ferromagnetism of FCC system of magnetic nanoparticles.

[1] O. Kasyutich et.al. J. of Physics D: Applied Physics, **41,** 134022 (2008)

[2] O. Kasyutich et.al. J. Appl. Phys. 105 (2009)

9:30 Oral

Investigation of transition metal nanoparticle self-assembling in nanopores of multifunctional composite materials by Monte-Carlo computer simulation

Mark S. Zhukovskiy[2], Serge A. Beznosyuk[1], Michael S. Grishko[1]

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A Monte-Carlo method is applied to the research of self-assembling processes of transition metal nanoparticle aggregation in nanopores of multifunctional composite materials . Method was adapted for non-equilibrium self-assembling processes in nano-sized pore to take account of two opposite quantum statistical correlation modes of corporate particles motion. The first mode is a model of "highly-correlation state" where nanosystem is characterized by highly-correlated wave function. Contrariwise, the second mode is a model of "lowly-correlation state" where nanosystem is characterized by multiplicative wave function.In original research the simulation program randomly generates a set of particles located inside a cubic cell with edge length of 1 nanometer. This allows us to acquire highly non-equilibrium system. After that the structural relaxation is fulfilled at temperature T=298K. Using two correlation modes described above there is a Markov process, when chain length is equal to 100000 steps. For each step the nanosystem's energy is calculated using a pairwise potential, which was calculated by means of non-local-density functional method. As a result it is gained extremely different nanosystem morphology in each way of quantum correlation statistic. In the "highly-correlation" mode nanoparticles assembling into one cluster of atoms, but in "lowly-correlation" mode a number of aggregates with various nuclearity are reaveled. Thermodynamics equilibrium of last system is described by huge contribution of entropy factor. Analyzing the energy functional relation of relaxation process reveals regularity in aggregation of small structures into more compact clusters with greater number of bonds. Discovered regularities of self-assembling in each correlation mode may be used for controlling functional nanomaterials shaping processes with specified properties. This way adaptively morphed nanostructure of highly-active multifunctional composite material could be produced.

9:45 Oral

Features of shock-wave processes and destructions in anisotropic materials

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Materials with primary orientation of physicomechanical properties are widely used in various areas of engineering, including as the equipment for the launch of projectiles, the projectiles and barrier. At the same time properties of such materials at intensive dynamic processes poorly studied. In work results of research of behaviour of anisotropic materials and designs are submitted at impact and pulse loads. For the description of the behaviour of anisotropic materials within the framework of the phenomenological approach the model which is taking into account distinctions in strength on compression and a stretching is offered. The model as allows carrying out researches with any orientation of properties of a material concerning system of coordinates. Numerical modeling is carried out in three-dimensional statement. The range of speeds of interaction up to 3000 m/sec is considered. Evolution of wave processes and destructions is investigated on orientation physicomechanical a material, kinematic and geometrical parameters. Orientations of properties providing optimum use of anisotropic materials are determined.

10:00 Oral

From monolithic advanced ceramics to nanostructured ceramic matrix composites. Structural features and tribological advantages

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The paper analyzes the advantages and limitations of the major types of monolithic structural ceramics currently in use for tribological applications in abrasive and aggressive media.

Limitations inherent to the material, as well as processing and geometry limitations narrowing the scope of possible designs and dimensions of the wear-resistant tribological structural ceramics are discussed.

Transition from monolithic ceramics to fiber composites with ceramic matrix enables a number of advances, which are demonstrated through comparing the physical, mechanical, and tribological

parameters of the two classes of materials: monolithic SiC ceramics and fiber-reinforced C_f/n/- SiC composite.

Two key structural factors influencing the optimization of the physical, mechanical and tribological behaviour of the composite are discussed:

· Nanostructured barrier coating on carbon fibre

· Special 'working' tribological layer (functionally graded structure of the composite).

Hierarchical structural features of the nanostructured ceramic matrix composite (NCMC) were characterized by means of light and scanning electron microscopy.

Major tribological parameters of the both classes of materials are diagnosed, and feasibility of using NCMC in tribological assemblies of slide bearings is demonstrated. Correlations between NCMC structure and critical specific load on tribological assembly are discussed.
The efficiency of NCMC is exemplified by large radial slide bearings designed for extreme service conditions.

COFFEE
Tuesday morning, 15 September, 10:30
Main Hall

Lectures
Tuesday morning, 15 September, 11:00
Room 219

11:00 Invited oral

Manufacturing of ceramic-ceramic composites by colloidal routes

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Ceramic-based composites have received great attention due to their wide number of applications. It is desirable to produce materials with the suitable combination of properties of various components. The addition of a low concentration of secondary phase to a ceramic matrix has a great effect on the behavioural properties of the composite. Nevertheless, the final properties of the composites are a function of the characteristics of the starting powders used in their manufacture. This work deals with the manufacture of ceramic-ceramic composites by using suspensions of oxides with different characteristics, such as particles size distribution and surface area. In addition to the synthesis routes employed for each powder, the dispersion of the mixtures is the key point to produce homogeneous materials. This can be achieved by manipulating the interaction potentials between the components through heterocoagulation and dispersion mechanisms. The stability of the suspensions can be studied in terms of zeta potential measurements and interparticle potential calculations. The manufacture of bulk bodies needs the preparation of concentrated suspensions in order to reduce the amount of liquid to be removed during green processing. This work focuses on the manufacture of alumina-based composites including compatible oxides or incompatible ones that develop through a reaction sintering process.

11:30 Oral

Phase relations in the systems based on ceria with lanthanides and zirconia

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Phase equilibria in the ZrO_2-CeO_2-Ln_2O_3 systems are important for the development of alternative ceramics for thermal barrier coatings (TBCs) other than state of the art yttria-stabilized zirconia (YSZ), aiming to lower thermal conductivity and to improve high temperature performance and durability. Ceria doped with aleovalent cations, such as rare earth oxides, has been considered as one of the most promising candidate materials for intermediate temperature solid oxide fuel cells (SOFCs) because of its much higher ionic conductivity at lower temperatures in comparison with that of stabilized zirconia. In addition, ceria finds its application in optical polishing, petroleum cracking catalyst, nuclear reactors components etc.

Present work is about phase equilibria in the ternary ZrO_2-CeO_2-Ln_2O_3 systems and in the boundary binary systems CeO_2-Ln_2O_3, ZrO_2-Ln_2O_3 (where Ln= La, Sm, Gd) at 1100-1500 °C in air in the whole concentration range. X-ray diffraction, petrography and electron microprobe X-ray diffraction were used to determine phase contents. The microstructures of the sintered ceramic samples were examined by using the scanning electron microscopy (SEM).

The isothermal sections at 1100, 1500 °C for the ternary systems ZrO_2-CeO_2-La_2O_3, ZrO_2-CeO_2-Sm_2O_3 and ZrO_2-CeO_2-Gd_2O_3 were developed. The main regularities of the phase diagram state (topology) have been revealed in both binary and ternary systems as a function of dopant ion radius. It was shown that the character of the phase equilibrium in the ternary system is determined by the binary phase diagram.

In the subsolidus area of the phase diagrams ZrO_2-CeO_2-Ln_2O_3 solid solutions based on tetragonal (T) ZrO_2, cubic with fluorite-type structure (F) CeO_2 and ZrO_2, hexagonal (A) La_2O_3, cubic form of rare-earth oxides (C-type) and monoclinic (B) forms Sm_2O_3 and Gd_2O_3 as well as intermediate phases with pyrochlore-type structure $Ln_2Zr_2O_7$ were found.

11:45 Oral

SnO_2, TiO_2, and TiO_2/ZnO thick film pressure sensor with interdigitated electrodes

Olga Korostynska, Arshak Arous, Khalil Arshak, Martin O'Sullivan, Deirdre Morris

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The pressure sensing properties of SnO_2, TiO_2, and TiO_2/ZnO thick film sensors are investigated.

To form the dielectric layers, the metal oxides powders were respectively mixed with isopropanol and wet ball milled for 24 hours. The mixtures were dried at 120 °C to evaporate the alcohol. The powders were then placed under 2 tons of pressure to form pellets, which were fired at 1250 °C (rate of 5 °C per min) in a vacuum of 6 x 10^{-3} mbar for five hours, followed by cooling (rate of 3 °C per min). After firing, the powders were mixed with 7 wt.% of polyvinyl butyral (binder) and suitable amount of Ethylenglycolmonobutylether (solvent) to form the pastes. These were screen-printed over the Ag electrodes on alumina substrates to form SnO_2, TiO_2, and TiO_2/ZnO capacitor pressure sensors accordingly.

The capacitance of the sensors with interdigitated electrodes is largely dependant on the number of fingers (N), their length (L) and width (W), in addition to the gap between electrodes (G), spatial wavelength ($\lambda = 2(W+G)$) and the metallization ratio ($\eta = 2W/\lambda$) [1]. Any changes in capacitance are due to deformation of the dielectric layer, deposited over the electrodes. The overall capacitance of the device is generally determined by using a conformal mapping technique [2].

The evaluation of pressure sensing properties of these sensors was performed using a HP 4192A Impedance Analyser, which recorded the changes in the values of the capacitances under different mechanical stresses. At the applied load of 5 kPa, the response times of 2.5 s, 5.6 s and 4 s were recorded for SnO_2, TiO_2, and TiO_2/ZnO sensors respectively.

In addition to instant response times, these pressure sensors have the advantage of being reusable, as their electrical properties were restored to the original value after annealing for 2 hours at 80 °C.

12:00	Oral

Application of monosaccharides derivatives in gelcasting of alumina

Paulina Bednarek[1], Mikolaj Szafran[1], Yoshio Sakka[2], Tadeusz Mizerski[1]

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Recently colloidal processes are willingly applied in fabrication of high-quality ceramic elements of complicated shape among which we can distinguish gelcasting, a method which combines conventional moulding from slips with polymer chemistry. The research made by authors concentrate on synthesis of new low-toxic monomers, which could be applied in gelcasting instead of toxic and commonly used acrylic substances. The interesting alternative for commercially monomers can be compounds synthesized on the basis of monosaccharides. The first synthesized monomer is 3-acrylic-D-glucopyranose. The application of this monomer in gelcasting process allowed to obtain rigid green ceramic specimens without adding an external cross-linking agent to a slurry, what allowed reducing amount of gases released to the atmosphere during polymer burnout.

The work reports the research on application of new monomer in gelcasting of alumina powders of different grain size 0.21mm and 0.70mm. The rheological behaviour of ceramic slurries has been first investigated. Then the properties of received green and sintered elements have been compared with those obtained with application of commercially available monomer 2-hydroxyethyl acrylate.

This work was financially supported by Ministry of Science and Higher Education of Poland, (Grant No. N N209 150236)

LUNCH

(maps available at the Reception desk)
Tuesday afternoon, 15 September, 12:30
building of the Faculty of Physics

Lectures

Tuesday afternoon, 15 September, 14:00
Room 219

14:00	Oral

Silicon carbide synthesized from plants and its potential applications

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Silicon carbide can be synthesized by a gas-solid reaction between silicon vapor and carbon. After such reaction, the resultant silicon carbide preserved the morphology of starting carbon. Using this process, silicon carbide powder can be synthesized from a powder mixture of Si and carbon black in argon atmosphere. Furthermore, bulk porous silicon carbide can also be synthesized from plant-derived bio-carbon. These bulk porous silicon carbides can be applied as reinforcing skeletons of ceramic-metal composites, or they can be used as free-standing catalyst supports.

In this presentation, I will present our work on the synthesizing of porous silicon carbide from bamboo-derived, rattan-derived and vegetable sponge-derived bio-carbon. The synthesizing parameters and the microstructures will be correlated. I will also describe the results of our attempts in fabricating Al-SiC, Cu-SiC composites using these bulk porous SiC and the catalytic performance of Ni catalysts supported on vegetable sponge-derived SiC.

14:15 Oral

Methods of hydroxyaptite preparation from catle bone tissue in order to create artificial biomaterials for bone tissue replacement.

Lauris Rupeks, Viktor Filipenkov, Ivars Knets, Zilgma Irbe, Liga Mezmale, Natalija Borodajenko

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Created method for obtaining natural hydroxyaptie (NHAp) from bone tissue of cattle. The NHAp was obtained at temperature 300-320 ^{o}C (first mode) and 260-280 ^{o}C (second mode).

First mode – NHAp was obtained from bone tissue of catle. Before deproteinization, the bone was freed from soft tissue and fat aftertwards cutted into 2-3 mm thick layers. Then the bone specimens were placed in a furnace and heat trated in a suspended state in a stream of air at a temperature gradually increasing from room temperature to 300-320 ^{o}C for 6.0 h., after which it was kept at a constatnt temperature for 37-40 h.

Second mode – The NHAp was obtained also from bone tissue of catle, but at temperature gradually increasing from room temperature to 260-280 ^{o}C for 1.5 h., after which it was kept at a constant temperature for 7-10 h.

In the present study the structure of bone tissue before and after deproteinization was investigated by the method of scanning electron microscopy.

The results of experiment, performed by the method of infrared spectroscopy, show that the protein was removed from the heat treated specimens of bone tissue practically compleately in both cases.

This method of obtaining of NHAp was designed for further creation of biomaterilas for bone tissue replacement.

14:30 Oral

Synthesis and investigation of the structural properties highly porous silica with large surface area

Natalia Kondrashova, Ekaterina Saenko, Viktor A. Valtsifer, Vladimir N. Strelnikov

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Mesoporous mesophase materials (MMM) with well-expressed porous hexagonal structures and dimension range 2-10 nm are used as inorganic sorbents, catalysts and catalyst carriers. Therefore, the production of mesoporous synthetic materials with controllable pore dimensions, large surface area is of current importance. One of the most effective methods for the production of nano-scale materials as a basis of metal oxide catalysts is the chemical liquid-phase condensation due to various conditions. Literary sources on the matter are inconsistent, therefore the optimization of technological procedures for the production of mesoporous silica appeared necessary. In this paper the influence of synthesis conditions, of the ratio between reaction components CTAB/TEOS, NaOH/TEOS, water/TEOS, of the use of organic additives such as 2-cyanoethyltriethoxysilane, 2-aminopropyltriethoxysilane and for the first time tris-trimethylsiloxysilane on textural and structural characteristics of the finished material during production of mesoporous silica has been studied. The structural characteristics have been investigated by BET measurement, Raman spectroscopy, Fourier-transform infrared spectroscopy and X-ray diffraction techniques. Optimization of technological procedures allows to produce the material with high specific surface (1400 m^2/g), total pore volume over 1 cm^3/g and average pore diameter approximately 4 nm.

14:45 Oral

Water-soluble photosensitizer-iron oxide nanoparticle conjugates for intracellular photodynamic therapy

Wen-Chang Chen[1], Fu-Kuo Hwang[1], Sheng-Feng Lai[1], Chi-Jen Liu[2], Cheng-Liang Wang[2], Hsiang-Hsin Chen[2], Tzu-En Hua[2], Yeu-Kuang Hwu[2,3,4,5]

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Water-soluble conjugates of protoporphyrin IX (PPix) and cross-linked dextran- coated iron oxide nanoparticles (CLIO NPs) have been designed and synthesized for the delivery of the hydrophobic photosensitizer for photodynamic therapy (PDT) of cancer. The PPix-CLIO NPs conjugates have an average shell-diameter of ca. 77.8 ± 1.6 nm with a magnetite core of ca. 4.03 ± 0.55 nm. When the PPix-CLIO NPs conjugates (0 - 40 μg Fe/mL) were incubated with HeLa cells at 37 □ for 12 h, they were taken up, delivering the PPix photosensitizer directly to the intracellular environment. The highly internalizing efficiency of the NPs-conjugates may be attributed to their positive surface potential (12.7 mV) at pH 7.4. Besides, the HeLa cells showed no observable dark toxicity. However, irradiation of the NPs conjugates within the cells by using 312 nm UV-light for 10 min (at co-cultured concentration of 5 μg Fe/mL of PPix-CLIO NPs) causes significant cell mortality through the photodynamic effect. These results suggest that PPix-CLIO NPs conjugates are an excellent vehicle for the delivery of surface-bound hydrophobic photosensitizers for efficient PDT of cultured tumor cells.

15:00 Oral

Shape memory effect in porous nitinol, synthesized during SLS/M process: Experimental approach

Igor V. Shishkovsky[1], Vladimir I. Sherbakov[1], Igor Yadroitsev[2], Igor Smurov[2]

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At present paper the existence of the shape memory effect in the porous titanium nickelide (NiTi intermetallic phase, named as *nitinol*), which was synthesized by the laser-controlled combination of the processes of the Selective Laser Sintering/Melting (SLS/M) and Self-propagated High-temperature Synthesis (SHS) are described, comparative with direct SLS/M of powdered nitinol. Optical metallography, SEM with EDX and X-ray analysis were used. The phase-structure content, thermal dependences of electrical resistivity are presented depends of laser influence parameters. Advantages and disadvantages perspectives of bio - MEMS fabrication from this porous material are discussed.

COFFEE
Tuesday afternoon, 15 September, 15:30
Main Hall

Wednesday, 16 September

Lectures
Wednesday morning, 16 September, 9:00
Room 219

9:00 Oral

Hot forging of P/M TiC reinforced and high speed steels

Gennadii A. Bagliuk, Dmitro Goncharuk

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Hot forging of porous preforms is the alternative method for manufacturing of piece preforms from tool steels powders in relation to liquid phase sintering, providing, at the same time, possibility for production of materials fine-grained structure which is approximating to characteristics of powder steels, produced by technology of hot isostatic pressing at solid-phase temperature interval with the subsequent hot hammering and (or) rolling.

Among the variety of wearproof iron based materials which are produced by methods of powder metallurgy, the carbide reinforced steels - composites on the basis of, as a rule, alloyed steels with disperse inclusions of transitive metals carbides particles (mainly – titanium carbide) with their mass composition from 15 to 70 % to take up special position.

The research of the basic technological parameters influence on structure features of hot forged materials produced from high speed steel – TiC powders mixture was the purpose of the present work.

Technological process for production of TiC reinforced steels with application of hot forging technology included following basic operations: miling of high speed steel and 20 % (weights.) titanium carbide powders mixture, compaction of porous preforms at pressure of 500 MPa, its heating in argon to temperature 1150 ¸1160 ^{0}C and hot forging in a closed matrix on a crank press.

Comparative analysis of produced by liquid phase sintering or hot forged composites microstructures has shown, that in a structure of sintered material the TiC grains, settling down mainly on borders of matrix grains, create rather coarse-dispersion grid structure. The average size of TiC grains makes about 1,8 microns, and matrix grains - 13 microns. Application of hot forging, practically not changing the average size of TiC grains (about 1,7 microns), at the same time allows to crush essentially alloy matrix structure which sizes of grains decrease in 2 ¸ 2,5 times.

The effect of alloy formation activation is found out at hot plastic working in high speed steel – titanium carbide powder system. Quality standard of elements distribution between matrix and titanium carbide particles has shown, that in a composites, produced by liquid phase sintering, TiC grains are enriched by vanadium, tungsten and molybdenum with distinctly expressed circular-type structure on grain borders. In a case of application hot forging technology, the concentration of elements is substantially equalized, therefore essentially more uniform distribution of alloying elements across the field of microsection is observed.

9:15 Oral

Structure and properties of Fe-C-B sintered steels

Gennadii A. Bagliuk, Svietlana G. Napara-Volgina, Vladimir Volfman

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The application of boron opens up possibilities for production of low-alloyed steels which operational characteristics in many cases are not inferior, but also surpass the properties level of steels produced with application of traditional alloying methods. The ample opportunities on working out of boron-containing materials of different functions are opened by the powder metallurgy, which application allows to produce both wearprof steels containing raised quantity of boron (up to several percent), and high-strength lean alloy steels of constructional appointment.

The various modes of boron insertion in powder steels have been studied: boron carbide, amorphous boron powder and powder Fe-B-C master alloy, produced by thermal synthesis from a mixture of iron and boron carbide powders had been applied as boron-containing components. The boront content in the steels were varied within 0,4 - 1,6 %.

In the issue of powder mixtures pressing investigation it is established, that compactibility of mixtures with the identical boron con-

tent at pressing essentially worsens with increase of boron master alloy component quantity.

The investigation of sintered materials properties has shown, that application of boron-containing master alloys allows to obtain the higher level of sintered and thermoprocessed materials physicomechanical properties in comparison with the steels produced from a mixture with boron or boron carbide powders.

The boront content in sintered materials, providing optimum combination of their properties at application of boron-containing master alloy of different composition, makes about 1,2 %: such boron content corresponds with the maximum values of hardness, durability and wear resistance as well as minimum porosity of both sintered and thermoprocessed materials.

The wear resistance tests had shown, that durability of sintered boron-containing steels substantially exceed the same for the cast and rolled bearing steel with 1% C and 1% Cr.

9:30 Oral

Structure and properties of ceramet composites Hf – HfB: experiment and theory

Volodymyr A Makara[1], Serhiy Chornobuk[1], Valeriy V. Kartuzov[2], Aik Khachatryan[2]

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In this work a structure and mechanical properties of composition materials Hf – HfB, got the method of a vacuum argon-arc melting is studied. Composition of the got specimens was studied by a X-ray analysis. A structure was probed the method of optical microscopy. A microhardness was measured on the device of PMT-3 at loading on indentor 1 N.

It is set that at the melting of powders of Hf and HfB_2 a chemical reaction the result of which is disappearance of phase of HfB_2 and formation of new phase of HfB flows between them. Thus, composition material of composition of Hf– HfB is formed.

At maintenance of HfB at the level of 25 % (molar concentration) formed materials with an eutecticum structure (T_{eut} = 2163 K). At the increase of maintenance of HfB the structure of matrix type is formed: the needle-shaped including of ceramic phase of HfB is evenly placed in the metallic matrix of Hf.

The microhardness of the got materials substantially depends on composition of compos, evenly rising from 2 GPa (Hf) to 9,5 GPa (Hf – 75%(molar concentration) HfB)

For proof of existence of eutecticum in the system of Hf – HfB by quantum mechanical calculations a calculable experiment is conducted

On the basis of method of a priori pseudopotential calculated energy of co-operation between the atoms of Hf – Hf, between the molecules of HfB – HfB and similarly between Hf – HfB. The parameters of grates of Hf and HfB are calculated.

It is got, that energy of mixing of components, consequently, Hf and HfB in the system of Hf – HfB does not dissolve, and from a condition (T is a temperature of existence of the system) follows, that Hf and HfB form an eutecticum alloy. From the condition of extremum of thermodynamics potential determine the characteristic parameters of the system in the point of eutecticum (temperature, concentration).

It is got, that a concentration of HfB is in the system of Hf–HfB = 26.3% (molar concentration) in the point of eutecticum, and temperature is T = 2173 K.

9:45 Oral

Structure and properties of ceramet composites Hf - HfB

Volodymyr A Makara, Serhiy Chornobuk

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Hafnium is characterized high mechanical durability and plasticity, and has high resistance and high corrosive firmness to the radiation damages. Such complex of descriptions does him suitable for the use in quality the absorber of neutrons. One of effective methods to increase of strenght descriptions of hafnium without worsening of suctive ability there is creation of composition ceramet materials on the basis of hafnium and boron, that it is related to specific nuclear properties of the indicated elements.

In this work a structure and mechanical properties of composition materials Hf – HfB, got the method of a vacuum argon-arc melting is studied. Composition of the got specimens was studied by a X-ray analysis. A structure was probed the method of optical microscopy. A microhardness was measured on the device of PMT-3 at loading on indentor 1 N.

It is set that at the melting of powders of Hf and HfB_2 a chemical reaction the result of which is disappearance of phase of HfB_2 and formation of new phase of HfB flows between them. Thus, composition material of composition of Hf– HfB is formed.

At maintenance of HfB at the level of 5 %(wt.) formed materials with an eutecticum structure (T_{eut} = 1890 0C). At the increase of maintenance of HfB the structure of matrix type is formed: the needle-shaped including of ceramic phase of HfB is evenly placed in the metallic matrix of Hf.

The microhardness of the got materials substantially depends on composition of compos, evenly rising from 2 GPa (Hf) to 9,5 GPa (Hf – 15%(wt.) HfB).

10:00 Oral

Film-forming metallic materials

Elena Zhdanuk[1], Elvira Krutko[1], Ta Zharskaya[1], Ja Galeeva[2]

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The present research is devoted to physical-chemical, technological aspects and material authority of order to produce nanostructured paint and vanish composites.

In capacity of research objects are chosen stove paint and varnish materials such as semi-manufactured vanish ML-0136, enamel ML-12 black produced by Joint Stock Company "Lakokraska" and metallic nanomaterial.

Film-forming compositions are obtained by bringing in vanish ML-0136 and enamel ML-12 of calculated amount of nanofiller which should be crushed in ball grinder before and kneaded. Solutions are poured on clean and degreased surfaces from steel, tin ang soda-lime glass. Obtained compositions are hardened in the oven at elevated temperature (130-140 °C) within 30 minutes till the insiccation extent 3.

From analysis of physical, mechanical and adhesion coating characteristics it follows that nanofillers make it possible to improve hardness of coating from 0.35 to 0.42 standard units. Investigated nanofiller show it's the greatest efficiency if it is brought in small amount (0.5 % content to solid residual mass). In this connection it is observed optimal values of physical, mechanical and adhesion perfomance.

Compositions on basis of enamel ML-12 black are obtained similarly. Investigation showed that metallic nanofiller have an influence on coating properties based on enamel ML -12. Hardness of coatings, bending strength and adhesion depend on content of nanosize particles. Optimal value of concentration is 0,5 %. If modifier content is more than 0,5%, coating elasticity becomes worse.

Consequently it is induced law of influence of superdispersed filler content on breaking point and adhesion durability of investigated film-forming composites based on melamine-formaldehyde oligomers. It is specified optimal concentration of nanofiller when maximum breaking point (more than 100 centimetres) and maximum adhesion durability of coating to steel (0 point) can be reached.

COFFEE

Wednesday morning, 16 September, 10:30
Main Hall

Lectures

Wednesday morning, 16 September, 11:00
Room 219

11:00 Oral

Atom balance based on a carbon nanotube resonator

Yuan-Chih Chang[1], Der-Hsien Lien[1], Shau-Chieh Wang[1], Tung Hsu[2], Chia-Seng Chang[1], Tien-Tzou Tsong[1]

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We demonstrate a carbon-nanotube-based resonator with atomic mass resolution. We tailored a multiwalled carbon nanotube (CNT) to possess a resonant frequency over 130 MHz and a Q factor of ~1000. Calculated with the mass of this tube, the mass sensitivity is estimated about 10^{-22} g. By detecting the resonance frequency shift of this CNT using the STM@TEM system, the mass of a 3 nm Ag particle was estimated to be 0.18×10^{-18} g, close to the mass of 1000 Ag atoms. Our tailored CNT is thus capable to resolve a single Ag atom and can be named an ''atom-balance''. It can also be found that the resonance frequency shift due to the Ag particle is sensitive to the Ag particle's adsorption position on a CNT, as shown in Fig.1. The results are confirmed with the FEM simulation. In addition, when a CNT is driven into resonance under the evaporation of Ag atoms, a gradient distribution of Ag particles along the CNT axis can be assembled (Fig. 2). By combining these techniques, further improvements may conceivably achieve the mass resolution of a light element, which could then enable the elemental or chemical identification.

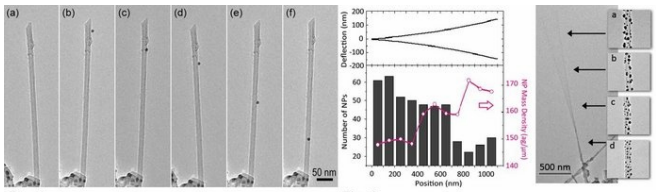

Fig. 1 Fig. 2

11:15 Oral

Carbon- and metal-based nanocomposite films as substrates for the adhesion and growth of bone cells

Lucie Bacakova[1], Lubica Grausova[1], Marta Vandrovcova[1], Katarina Novotna[1], Alexander Kromka[2], Jiri Vacik[3], Hynek Biederman4

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Nowadays, there is an increasing demand for artificial body implants, which support the regeneration of the damaged tissues and are well-integrated with the surrounding environments. Metallic bone implants often suffer for mechanical and biochemical incompatibility with the surrounding bone tissue, which can lead to the

aseptic loosening of the bone tissue and the implant failure. Their anchorage in the bone tissue can be improved with mechanically resistant, chemically stable and biocompatible layers, especially those nanostructured. The nanoscale roughness and topography of the material surface is believed to mimic the nanoarchitecture of the natural extracellular matrix as well as the cell membrane, and thus such types of surfaces are usually attractive for the cell adhesion and growth. Moreover, these surfaces adsorb preferentially vitronectin, i.e. an extracellular matrix protein recognized preferentially by osteoblasts, and thus they can prevent a fibrous encapsulation of the bone implant. In our studies, we investigated the adhesion, growth, viability and osteogenic maturation of human bone-derived cells (represented by a cell line MG 63) in cultures on undoped and boron-doped nanocrystalline diamond films, and nanocomposite hydrocarbon plasma polymer films with various concentrations of Ti. We found that the cell number, cell spreading area, formation of focal adhesion plaques, concentration of integrin adhesion molecules with alpha V or beta 1 chain, integrin-acssociated proteins talin and vinculin, as well as the concentration of osteocalcin and osteopontin, markers of osteogenic cell differentiation, in cells on these layers were similar or even higher than on standard polystyrene cell culture dishes. Even nanocomposite hydrocarbon plasma polymer films with 3 at% of Ag promoted cell adhesion, growth and formation of focal adhesion plaques without considerable cytotoxicity. At the same time, these layers attenuated growth of the bacterium E. coli. Also the layers of fullerenes C_{60} or binary composited or C_{60}/Ti gave good supports for the adhesion and growth of MG 63 cells. Micropatterned layers of C_{60} or C_{60}/Ti, i.e. deposited on the substrates through a metallic grid, promoted regionally-selective cell adhesion and growth in grooves among the prominences formed bellow the grid openings. Therefore, nanostructured and hierarchically micro- and nanostructured hard biocompatible and bioactive films are applicable not only for biomaterial coating but also as an interesting tool for investigating mechanisms controlling the cell behavior by cell-material interaction.

Supported by the Academy of Sciences of the Czech Republic (Grants No. KAN101120701, KAN400480701, IAA400320901, IAAX00100902).

11:30 Oral

Effect of carbon contents on the structural and mechanical properties of sputtered (AlCrTaTiZr)C_x coatings

Keng-Hao Cheng, Ting-Wei Liu, Su-Jien Lin, Jien-Wei Yeh

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The (AlCrTaTiZr)C_x films are deposited onto Si substrates and cemented carbide plates by dual-gun RF magnetron sputtering. The carbon contents are controlled by tuning the applied power of each target, and ranged from 0 to nearly 50 at.% determined by EPMA analysis. The influence on the crystal structure, cross-sectional microstructure, hardness and other mechanical properties is also investigated. Without the carbon additions, the AlCrTaTiZr alloy film exhibits an amorphous structure and shows a glass-like microstructure by SEM observation. Another distinguished peak in XRD pattern is found as the carbon atoms are incorporated, which is considered as the multi-element carbide phase. These (AlCrTaTiZr)C_x films perform a nanocomposite structure with FCC carbide nanocrystallites embedded in an amorphous alloy matrix. The hardness and modulus are increasing with the raised carbon contents. The friction coefficient and wear rate of coatings are also improved by carbon addition.

11:45 Oral

High-resolution eddy current sensor system for multiaxial carbon material characterisation

Henning Heuer, Martin Schulze, Martin Kuettner

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Carbon fibre materials become more and more important in many applications. Unlike to metal the technological parameters and certificated quality control mechanisms are not developed at present. There is no efficient and reliable testing system for an inline inspection and a consecutively manual inspection of the Raw Carbon Fibre materials (RCF) and the post laminated Carbon Fibre Reinforced Plastics (CFRP). Hidden defects during the production can cause far-reaching consequences with high-risk about material fatigue and even higher costs due to scrap coming out after machining. Based upon the multi-frequency eddy-current device developed at the Fraunhofer IZFP it is possible to detect structural an hidden defects e.g. missing carbon fibre bundles, lanes, suspensions, fringes, missing sewing threads and angle errors. With the help of an optimized sensor array and an intelligent image pre-processing algorithm it is possible to allocate the complex impedance signal to different carbon fibre layers. This technique enables the possibility to detect defects in the depth of 5 layers with the option of free scale resolution and measure frequency. Appropriate parameter lists for an optimal error classification are available. The sizes of the smallest detectable defects are in the range of few millimetres. A prototype of a special single sensor and even an eddy-current sensor array are developed and establish the chance to move the prototype into an industrial application.

12:00 Oral

Spin-depended processes of carbon nanotube hydrogen accumulation

Olga A. Maslova[1], Mark S. Zhukovskiy[2], Serge A. Beznosyuk[1]

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Description of state and quantum nature of hydrogen subsystem, represented by supramolecular polycondensate on nanotubular surface, is the problem of particular interest. One has to consider that theoretical approaches based on both physical sorption due to Van der Waals interaction and chemisorption with covalent bonding

seem to be unsatisfactory when dealing with high-performance reversible hydrogen polycondensation. The main consequence obtained under such circumstances is inapplicability of molecular orbital approach to description of interaction between hydrogen and adsorbing surface. Our consideration of the state and of the nature of bonding of subsurface molecules H2 proceeds from the results of non-empirical calculations via approximating quasiparticle density functional method. The state of hydrogen is described by active biradical state of dimer H-H. There are quasielectron density has a localized one-centered distribution in hydrogen molecule. Evidently, this fact is determined for hydrogen intermolecular interaction the examination of two cases of spin multiplicity: singlet and triplet. The selection of an optimal spin configuration for the hydrogen layer is carried out by iteration procedure of the total binding energy minimization within the framework of spin-flip dynamics. The nature of contact exchange interaction, typical for the systems with finite density distribution, is revealed within the framework of quantum-field concepts of matter structure. We model a relaxation phase being comprised in evolution of hydrogen polycondensate from the position of the approach to stochastic dissipative dynamics. It is taking into account, that decoherence frequency depends on parameters of polycondensate quantum plasma and on temperature. Several cases of adsorption contact of hydrogen with both external and internal nanotubular surfaces are presented. On the basis of received we forecast the possibility of using carbon nanotubes as hydrogen medium storage.

LUNCH

(maps available at the Reception desk)
Wednesday afternoon, 16 September, 12:30
building of the Faculty of Physics

Lectures

Wednesday afternoon, 16 September, 14:00
Room 219

14:00 Invited oral

Structure and adsorption abilities of nanoporous Ti,V-(N, He) coatings.

Alexsey Guglya[1], Aleksander Goncharov[1], Victor Kolobrodov[1], Ruslan Vasilenko[1], Yuriy Nikitenko[2]

1. *Kharkov Institute of Physics and Technology (KIPT), Akademiskaja 1, Kharkov 61108, Ukraine* **2.** *Joint Institute of Nuclear Research, Dubna, Russian Federation*

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The basic idea of this work is to create nanocrystalline composite materials with high level of porosity and good adsorption properties. It was investigated structure and adsorption characteristics Ti,V-(N, He) coatings deposited onto Ti substrate using the ion-beam assisted deposition technology. The results of the research show that the ion beam assisted deposition can be used to obtain nanoporous structures. It was shown that Ti and V deposition during high energy N_2 and He ions bombardment leads to nanocrystalline fcc structures formation with equiaxed and fine-dispersed pores. The average size of grains in V-(N_2, He) and Ti-(N_2, He) coatings is equal 20 nm, porous – 15 nm. Beside equiaxed pores there are many long, presumably gas-vacancy, 100-200 nm length formations. These formations create the system of inner cannels in coatings. The average size of grains in Ti,V-(N_2, He) coatings is equal
60 nm. Only equiaxed pores are present. The electric resistance of such materials exceeds that of powder vanadium nitrides by factor of 10. A sign of the temperature coefficient of resistance changes from a negative to the positive one in the temperature range of 250 to 350°C.

The results of adsorption studies showed that the obtained structures have a rather developed inner specific surface: Ti-(N_2, He) – 20.3 m2/g, V-(N_2, He) – 38.3 m^2/g, Ti,V-(N_2, He) - 69 m^2/g. From studying of isotherm of adsorption it has been established that the main contribution to the process of hydrogen storage at low temperatures is made by the created porous structure. For all coatings took place the increasing of hydrogen adsorption from the pressure ~ 0.15 MPa. The largest specific quantity hydrogen was adsorbed by Ti,V – (N_2, He) structure - ~ 350 cm^2/g. At elevated temperatures the absorption capacity of the intermetallide and nitride phases may increase.

14:30 Oral

Structural characterization and capacitance measurement of TiO_2 / Al_2O_3 nano-composite fabricated by atomic layer deposition (ALD)

Chun-Lin Wu, Jyun-Yi Wu, Chia-Cheng Chang, Wen-Kuang Hsu, Su-Jien Lin

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In the past decades, nanowires attracted lots of attentions due to their unique properties, so they are predicted to be the candidates for nano-devices in the next generation. Among nano-materails, titaninm dioxide is a well known photocatalyst, which possesses excellent photoelectric transformation efficiency. Alumina is a common gate oxide material with high dielectric constant (k) and low leakage current. We coated a extremely thin sheath of titaninm dioxide on the surface of alumina nanowire by atomic layer deposition (ALD). From the selected area electron diffraction (SAED) image and energy dispersion spectrum (EDS) results by TEM, structure and composition of the nano-composite was identified to be single crystalline γ-alumina and titanium oxide. An exceptional epitaxy effect of ALD on alumina nanowire was found by HR-TEM analysis. The effect may be resulted from the high crystallinity of Al_2O_3 nanowires and minimization of lattice distortion energy. C-V curve under light emission was also measured to discuss the feasibility of photocatalytic characteristics and carrier density of TiO_2/Al_2O_3 nano-composite materials. The described fabrication and characterization technique enables a systematic investigation of the characteristic of capacitance in the nanowires as well as of the quality of the nanodevices.

14:45 Oral

Low energy plasma spraying of thick-biocomposite coatings: a step towards bone scaffolds

Diana Garcia-Alonso[1], Maria Parco[2], Joseph Stokes[1], Lisa Looney1

1. *Dublin City University (DCU), Dublin 9, Dublin Dublin 9, Ireland* **2.** *INASMET-TECNALIA, San Sebastián 20009, Spain*

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Hydroxyapatite, (HA), is a calcium phosphate bioceramic that has been widely used in orthopaedics and dentistry due to its biocompatibility and osteoconductivity. However, its poor bulk mechanical properties have limited its use to coating on metallic surfaces. The plasma spraying technique is the most commonly used method for the production of HA coatings. Specifically, low energy versions of it have been appointed as most suitable techniques to spray HA without major phase decomposition and crystallinity decrease. However, HA coatings have small non-interconnected porosity which would hinder the posterior cellular ingrowth.

The present work aims to produce thick coatings as a step further towards the bone scaffolds. Aiming to produce bone scaffolds will require addition of second phases to the matrix material (HA) to improve both the final mechanical properties and porous structure of the components.

A reference HA series was produced. Statistically designed experiments (DoE) were used to determine the effect of three process parameters on four HA coating properties (layer thickness, roughness, crystallinity and phase purity). Models describing the effects of the variables on these coating properties were developed and the optimal parameters were selected to produce the biocomposite coatings. Three mechanical properties (Vicker's microhardness (H_v), Young's modulus (E) and fracture toughness (K_{IC})) and porosity were measured as reference on the sample produced with the selected parameter set.

In order to improve the structure, a bioreabsorbable polymer (Polycaprolactone (PCL)) was selected. The reinforcement material selected was a bioinert ceramic, Titanium Oxide (TiO_2). The addition of these materials was also optimised using DoE. Early results suggest an improvement in both structure and mechanical properties.

Further research will involve the characterization of the dissolution behaviour of the components and determination of cell behaviour.

15:00 Oral

Laser induced microrelief superstructure of Ag/ITO seed-mediated nanocomposites

Rafał Miedziński[1], Jean Ebothe[2], Richard Kleismit[3], Gregory Kozlowski[3], Julita Ozga[4], Jacek J. Kasperczyk[1], Ivan V. Kityk[5], Izabela Fuks-Janczarek[1], Khalid Nouneh[6], Munetaka Oyama[6], Marek Matusiewicz[4]

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The topography of indium tin oxide (ITO) films with incorporated silver nanoparticles and irradiated by single pulses of 18 ns Nd:YAG laser has been investigated. The study was carried out with two Ag/ITO films having resistances of 50W/ITO sheet and 4W/ITO sheet. The periodic structures in both samples were created after the laser treatment. The photo-induced periodic structures have a different character while the sheet resistance plays a major role in the growth process of these structures. The results of optical and non-linear optical investigations lead us to the conclusion that the temporary polarization of samples during laser treatment is responsible for the shape of periodic structures. This conclusion has been also confirmed by local permittivity measurements using evanescent microwave microscope.

COFFEE
Wednesday afternoon, 16 September, 15:30
Main Hall

Joint Poster Session
Monday & Wednesday
Wednesday afternoon, 16 September, 16:00
Main Hall

Thursday, 17 September

Lectures
Thursday morning, 17 September, 9:00
Room 219

9:00 Invited oral

Deagglomeration process of nanoceramic powders-challenges and practice

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The results of studies on application of selected mono-, di- and oligosaccharides in the deflocculation process of nanometric alumina and zirconia slurries are presented. In the research nanometric alumina powder of average grain size 47 nm and specific surface area measured by BET method S_{BET} = 35 m^2/g was used. In the research of nanometric zirconia powders two kinds of powders were used: average grain size 17 and 44 nm with specific surface area measured by BET method S_{BET} = 35 m^2/g and 65 m^2/g respectively. To explain the influence of the individual hydroxyl groups of saccharide on the adsorption process on the n-Al_2O_3 and n-ZrO_2 surface and the reduction process of the particle agglomeration were performed. The influence of D-fructose, D-glucose, L-sorbose; and disaccharids: maltose and saccharose on nanometric alumina and zirconia slurry have been carried out. The results obtained with mono- and disaccharides have been compared with the results on inulin. Inulin is oligosaccharide which consists of fructose units connected in chain (about 12 units) and ended by a glucose molecule from one side. It has been found that not only the amount of hydroxyl groups but also their orientation in saccharides molecule and their orientation to the surface of nanopowder are the main factors which decide about the amount of water connected with the nanometric alumina and zirconia particles and directly influenced oh the rheological properties of alumina slurry.

The work was supported by the Polish Ministry of Science and Higher Education nr N205 009 31/053

9:30 Oral

Effects of reverse polarity on electrospun fiber nanocomposites

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This work examines the effects of polarity on electrospun nanocomposite fibers made of polymer and metal oxide sol solutions. Molybdenum trioxide and Polyvinylpyrrolidone nanocomposites were studied as-spun under straight polarity, i.e., keeping the needle negatively charged and collector grounded and on reverse polarity. The former configuration produced “core-shell” type nanofibers with the polymer encasing the amorphous gel. This was an unexpected result as there was nothing (but electrostatic forces acting on the composite solution within the metallic needle tip) to separate the two materials in such a clear and aligned manner. On studying the effects of reverse polarity on the electrospun nanocomposites, i.e., keeping the needle grounded and the collector negatively charged, the nanofibers morphology was opposite too, by keeping the PVP embedded into the MoO3 matrix, as it was concluded by obtaining a similar diameter wire under as-spun and after a heat treatment to remove the polymer. Parameters such as the relative Zeta potential and electrostatic force appear to play an important role in electrospinning composite nanofibers. Knowing the parameters and their effects will allow tailored processing of biocomposite nanofibers. A model is proposed to explain the findings.

9:45 Oral

Synthesis and Characterization of Bifunctional TiO_2/SnO_2 Nanocomposites

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Uniform TiO_2/SnO_2 composite nanopowders with different Ti/Sn molar ratio have been successfully prepared via the sol-gel route. The samples were characterized using X-ray diffraction (XRD), thermogravimetric and differential thermal analysis (TG-DTA), transmission electron microscopy (TEM) and infrared spectrum (IR). The crystal size of the as-synthesized TiO_2/SnO_2 nanopowders is about 80 nm according to the XRD calculation. XRD analysis shows that the diffraction peaks associated with SnO_2 can’t be found in XRD patterns when the molar ratio of TiO_2/SnO_2 is less than 10/1. The photocatalytic degradation of methyl orange (MeO) in TiO_2/SnO_2 suspension was investigated. The results indicate that the TiO_2/SnO_2 nanopowders show higher photocatalytic activity than pure TiO_2 when the molar ratio of TiO_2/SnO_2 is 10/1, but it will remarkably decrease with TiO_2/SnO_2 molar ratio of 1/1. The photocatalytic mechanism of TiO_2/SnO_2 nanopowders was discussed. In addition, the electric property of TiO_2/SnO_2 nanopowders suggests an inhibition electric effect at higher TiO_2 content. TiO_2/SnO_2 nanopowders with Ti/Sn molar ratio of 1/10 shows a resistivity of 1.4×10^3 Ω·cm. The TiO_2/SnO_2 nanopowders indicate a potential application in photocatalytic and electronic materials based on its bifunctional characteristics.

10:00 Oral

Synthesis of hybrid nanomaterials RuO_2@SiO_2

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Nanoparticles of metals or metal oxides are a subject of great interest in current research because of their unique physical and chemical properties which come from their high surface/volume ratio and from quantum size effects.[1] Because of their particular properties, nanoparticles find applications in various fields such as electronics,

optics, catalysis and magnetic storage media. Metal oxides nanoparticles are often used as sensitive layers in gas sensors,[2] but sometimes display a lack of selectivity towards a target gas. To overcome these problems, membrane filters can be employed, but then comes the problem of saturation. Thus, the use of catalytic filters involving a porous material as a catalyst support is an alternative that can increase the selectivity of gas sensors.[3] In this communication, an original way for the preparation of hybrid RuO_2@SiO_2 nanomaterials will be presented. The key point of this synthesis method is the use of ruthenium nanoparticles stabilized by a bifunctional ligand as building blocks. Such a ligand allows the nanoparticles stabilization in the first stage and then their assembly through the formation of silica by sol-gel process (Figure 1). Composite nanomaterials so-obtained can contain a high content of metallic nanoparticles dispersed in a matrix of silica. After calcination, hybrid RuO_2@SiO_2 nanomaterials are obtained. Such nanomaterials were used as catalytic filters for selective detection gaz[4]

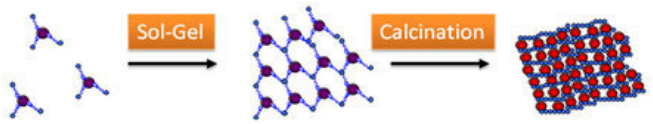

Figure 1. Synthesis of hybrid nanomaterials

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COFFEE

Thursday morning, 17 September, 10:30
Main Hall

Lectures

Thursday morning, 17 September, 11:00
Room 219

11:00 Invited oral

Features of obtaining composite powders and alloys in the $CrSi_2$–Ti (Ta)Si_2 systems in a nanodisperse state under mechanosynthesis of mechanical activation

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Refractory materials are traditionally synthesized via solid phase reactions between elements at high temperature. The achievement of a nanosized state for ceramics in this way is, however, practically impossible because of high temperatures of diffusion interaction, which causes coalescence and aggregation of fine refractory particles. Under intense milling, formation of new phases is possible:

- at room temperature thanks to activation of one or several chemical reactions, formation of nuclei in the course of mechanical treatment or growth from amorphous state, decomposition and phase transitions (mechanosynthesis);
- at reduced temperatures of subsequent synthesis thanks to prior activation of initial components (mechanical activation).

In both cases, synthesis includes milling. It is milling that provides nanoscale of initial powders and formation of high concentration of interfaces, at which numerous defects arise.

In this work, mechanosynthesis was studied depending on the process intensity, which was controlled by varying the duration of milling (60 and 90 min) in an impulse regime and the mass balls–to–powder ratio (BPR) of 20:1 and 30:1. Mechanical activation of the initial mixtures was performed under softer conditions (lower milling intensity, BPR 10:1, process duration 60 and 90 min), which leads to smaller particle sizes and to accumulation of structural defects, but this degree of activation does not provide running of the chemical reaction with formation of refractory compounds.

Numerous researches devoted to synthesis, structure and physicochemical properties of complex compounds on the basis of transition metal silicides make it possible to conclude that the level of physicochemical properties of solid solutions is higher as compared to that of individual silicides. When used, solid solutions provide high reproducibility of operation parameters thanks to wide regions of their existence. Materials based on solid solutions of higher silicide phases possess most stable properties.

To develop protective coatings operating at high temperatures, the $CrSi_2$–(Ti)$TaSi_2$ system was selected, in which two solid solutions, (Cr,Ti(Ta)Si_2 and (Ti(Ta),Cr)Si_2, exist in a wide homogeneity region. The solid solutions are formed by a reaction diffusion mechanism through subsequent formation of individual silicides from lower to higher ones followed by their interaction (homogenization). At the latter stage diffusion processes prevail.

The application of mechanosynthesis to these systems failed: a solid solution was not obtained. By the XRD data, the products of solid phase interaction under mechanosynthesis were dispersed composites composed of both higher and lower silicide phases. Mechanism

of their formation is reaction diffusion characterized by prevailing of the rate of chemical reaction over that of diffusion processes. In this case, it is impossible to produce solid solutions of silicides because the process of their formation, that is, mutual solution of disilicides requires long–term homogenization owing to diffusion. The use of low temperature synthesis of prior activated initial components permits one to produce a nanosized powder of solid solution with high enough reproducibility of the element composition. Composite systems prepared via mechanosynthesis are characterized by a particle size of about 50 nm, whereas solid solutions are characterized by agglomerates of about 300 nm.

11:30 Oral

Elaboration and nanoscale characterization of a Fe-38%atY_2O_3 nanocomposite prepared by reactive ball-milling and annealing

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Reactive ball-milling is a promising technique for producing nanocomposite materials. Indeed, when appropriate reactants and conditions are chosen, ball-milling can involve a chemical reaction which usually promotes nano-structuration. However, it is difficult to maintain the metastable nanostructure once it is formed. Indeed during ball-milling, takes place a competition between mechanically forced processes which disorder the system and thermal activation which tends to bring it back to the equilibrium state. Up to now it is not possible to predict how to control the balance between both. So we study this issue on a specific nanocomposite material.

The Fe-Y_2O_3 system was chosen because of its interest for ODS (Oxide Dispersion strengthened) steels studied for future nuclear power plant. We synthesized the Fe-38%atY_2O_3 metal-oxide nanocomposite by ball-milling YFe_3 and Fe_2O_3 in the stoichiometric proportions of the following chemical reaction: $2YFe_3 + Fe_2O_3$ à $8Fe + Y_2O_3$. Then the material was briefly annealed. The material was characterized at different steps by X-Ray Diffraction, Mössbauer Spectroscopy and Atom Probe Tomography.

We observe that, after few hours of ball-milling which mixes the reactants, the desired chemical reaction propagates but unexpectedly quickly and violently, leading to an unwanted coarse microstructure composed of two phases, α-iron and Y_2O_3. Afterwards, ball-milling gradually refines the microstructure while changing the phase composition and leads to the formation of two nanometric interconnected phases: α-iron super-saturated in yttrium and oxygen, and an yttrium and oxygen rich phase, with a ratio of oxygen over yttrium close to 1. After annealing, phase composition changes while phases coarsen but slightly enough to stay smaller than 100 nm.

Finally, we will explain the mechanism of formation with the concept of competition between thermal and forced processes. We will also identify the processing parameters which control it.

11:45 Oral

Synthesis and hydriding properties of Mg-based nanocomposites

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Mg/AB_5, Mg/AB and Mg/C nanocomposites were synthesized by mechanical milling in planetary type ball mills. The milling conditions were varied with the aim to produce composites with different morphology and microstructure, characterized by electron microscopy and x-ray diffraction. Continuous milling results in grain size refinement and in some cases to amorphization of the material. Milling under hydrogen gas atmosphere results in substantially finer particle size of the Mg/AB_5 and Mg/AB composites compared to the materials milled under argon.

The hydriding and dehydriding of the composites were carried out in a hydrogen gas phase as well as electrochemically. It was proved that the morphology and microstructure of the nanocomposites has an influence on their hydrogen capacity and hydriding/dehydriding kinetics. Finer particle and grain size improves markedly the kinetics of hydriding/dehydriding. Smaller nanocrystallites size generally results in an increase of the electrochemical capacity as well.

The hydrogen sorption kinetics of Mg/AB_5 and Mg/AB nanocomposites with different microstructure has been studied and the mechanism of the absorption process has been determined. A low-temperature (near room temperature) hydrogen absorption has been detected for the Mg/AB_5 nanocomposites. A small difference in the nanocrystalls size of the nanocomposites results also in noticeable difference in the electrochemical behavior (hydrogen capacity and stability at hydrogen charge/discharge cycling).

The hydrogen capacity, hydrogen desorption temperature and absorption/desorption kinetics of MgH_2 mechanically milled with different carbon additives were found to be dependent on the amount of the additives as well as on the milling duration. Considerable decrease of the hydrogen desorption temperature has been obtained for the MgH_2/C nanocomposites. The electrochemical hydriding/dehydriding behavior of Mg/C nanocomposites was also investigated.

12:00 Oral

The research of atomic interaction of substitutional impurities having grain boundaries in Ni3Al intermetallide at nanostructural level

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It is known that grain boundaries (GB) are the areas with declina-

tions of local densities with plus and minus signs at microscopic atomic level. That is why, substitutional impurities (alloying elements) can interact with GB and form complexes with them. Complex elastic-strained state realized at microscopic atomic level near GB leads to the change of the energy of the complex GB-alloying element on the distance between defects, type of alloying atom, type of the knot where alloying atom is located. In the paper, atomic interaction of substitutional impuritiesis is studied by the method of molecular dynamics in the dependence on temperature, type of substitutional impurities, its concentration, tilting angle of symmetric GB. Fully ordered intermetallide Ni3Al was chosen as the object of the research. Cr, Fe, Nb were taken as substitutional impurities. The calculated block containing the definite tilting angle in the center of GB with the combination of periodical and rigid boundary conditions was made at the first stage of computer experiment. The calculated block contained up to 20*10^3 atoms. The interactions between different pairs of atoms were given in the approximation of interatomic Morse potentials, the bonds of atoms in the first seven coordination spheres were taken into account. The procedure of relaxation was made by the heating of the block near 0 K, the following rapid cooling to 0 K. Then the definite concentration of substitutional impurities was introduced in the structure. The following results were obtained. Atoms of Fe were pushed from GB at their distribution over Al sublattice and they were attracted over Ni knots. The interaction energy of Cr and Nb atoms appeared to be negative independently on the knot type. The concentration of dopants near GB was to be above the average. Diffusion of admixtures grew in the direction of GB mainly in the area of low local density of the material with the increase of disorientation angle.

LUNCH

(maps available at the Reception desk)
Thursday afternoon, 17 September, 12:30
building of the Faculty of Physics

Lectures

Thursday afternoon, 17 September, 14:00
Room 219

14:00 Oral

Characterization of diatoms consolidation

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Diatoms are alge which create a variety of amorphous silica skeletons. The diatoms size range from 2 μm up to several millimeters. Tens of thousands of species which inhabit fresh and saltwater and also the possibility to their growth in cultures. The symmetric skeletons with the characteristic nanopores and many more their properties make diatoms as a very important materials for many engineering applications.

Among of the potential applications diatoms are reinforcements for composites and substrate for fabrication of bulk materials. From this point of view description of diatoms consolidation process and characterization of bulk materials are important.

In the work results of consolidation diatoms are presented. The bulk materials were obtained by modeling using a uniaxial (die) pressing method with different additions, then dried and sintered. Scanning electron microscopy (SEM) was used to observation of diatoms and bulk materials. Some properties as density and hardness were measured.

Acknowledgement

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14:15 Oral

Composite material microstructure design by means of T-x-y diagram computer model

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Phase diagram (PD) is the main tool for heterogeneous design (HD) - new approach of solid state chemistry, which opens new opportunities for composite materials engineering [Lutsyk V. Heterogeneous design: phase diagram – microstructure – materials genotype // http://www.matport.com/publishing/pdms/index.shtml P. 304-344.].

By means of PD geometrical elements projecting in the direction of concentration simplex, we divide the phase regions beneath into thermodynamically unstable fragments. PD computer model permits to decipher the horizontal and vertical sections of these fragments, surfaces and phase regions, to simulate the crystallization paths and to calculate the mass-balances for the structural elements and their conglomerates.

Two types of material balances may be shown: vertical – for the given centre of masses (in the temperature interval), and horizontal – an isothermal one, which coincides with the isopleth position.

Any surface is presented as a pseudo-ruled one, when a space curve moves along the analogously given directing curves [Lutsyk V., Zyryanov A. Microstructures design in the ternary systems with the only solubility gap // MRS Proceed. 2004. Vol. 804, P. 349-355].

T-x-y diagram computer model elaboration starts with its geometrical description, when all types of points, lines, surfaces and phase regions are named in special way. As the tabular schemes of phase reactions include the phase trajectories, it helps to designate the boundaries of two-phase regions too.
The term “genotype” is offered to take into account the whole variants of microstructures which can be realized at definite thermodynamic conditions. HD can be used to predict the microstructure of composite materials and to decipher their genotype, which is determined by PD topology.

14:30 Oral

Processing of end-of-life CRT glass and its potential as dielectric in AC-electroluminescence

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The reuse of waste materials in high quality applications is of high economic and ecologic importance. Glassy materials from end-of-life cathode ray tubes (CRT) in this context come in several fractions: panel or screen glass (ca. 2/3 by weight, major content of BaO and SrO), funnel glass (ca. 1/3 by weight, up to 25% PbO), neck glass (up to 40% PbO) and solder glass (up to 85% PbO). The PbO-containing fractions can be processed to metallic Pb, while for screen glass there is no further use at present. Therefore, preprocessing of this fraction (to get rid of the heavy metal content) and its potential use in dielectric binders (e.g. for AC-electroluminescence) are reported in this work.
Preprocessing of the milled glass fraction proceeds at elevated temperature in moist atmosphere and reduces the Ba- and Sr-content in the surface layer of the powder particles. The final activated glass powder is formulated with polymeric binders and sintering aids on borate base to a printable paste. Commercial AC-electroluminescent ZnS powder was worked into this paste, and finally small AC-electroluminescent units were fabricated from this multifunctional composite by tape-casting on ITO glass and application of carbon back electrodes. Morphological as well as optical data of these structures are reported. Future work will concentrate on improvements in homogeneity, area, emission intensity and durability. Since the binder is of inorganic nature, high temperature resistant structures on glass will be fabricated as well.
The results thus show, that a problematic waste material may be transformed into a multifunctional material of high economic value by comparable simple processing.

14:45 Oral

Multifunctional advanced composite materials, based on meso-2,3-dimercaptosuccinic acid modified magnetite nanocomposites.

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The necessity of obtaining new sorbents with medico-biological functions causes the urgency of new methods creation, by which the synthesis of magnetocarried nanocomposites with selective sorbing agent's features may be held. Nanosized magnetic particles coverage by highly effective chelating agent allows the purification of biosystems by nanostructures with functions of nanorobots of the first generation. In order to form polymeric layer on the surface of magnetic nanoparticles, which may act as a ligand, contain free tiol groups, cause transmission of nanoparticles from organic phase to an aqua one and provide for biostability, meso-2,3-dimercaptosuccinic acid (DMSA) was taken. The new technique of chemical modification of the monodomain magnetite surface by DMSA was developed to create nanocomposites for microbiological objects recognition in biological environment. The characteristics of obtained nanocolloid magnetic suspensions were analized. Zeta-potential (-40,3 mV) and average size (40,5 nm) of the magnetic particles suspension was measured by the method of dynamic light scattering. Methods of RPhS and IR spectroscopy were used to prove the the presence of disulfide bridges and –SH groups on the modified surface, while the method of iodometric titration the quantitive content of –SH functional groups was determined.

15:00 Oral

Formation of oligothiophenes from terthiophenes catalysed by gold nanoparticles

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The use of gold nanoparticles has been demonstrated to catalyse the formation of oligothiophenes from terthiophene in solution using ferric chloride as the oxidizing agent. The structures of the resulting core-shell materials have been confirmed by electron microscopy and surface-enhanced Raman spectroscopy. Due to the lengthening of the conjugated backbone upon polymerization the SERS enhancement of the polymer increases as the interband electronic transition approaches resonance with the laser excitation. We believe that it should be possible to perform the polymerization of terthiophene **in solution** on the surface of AuNPs, the very high surface area of nanoparticles allows a much larger amount of material to be prepated.

COFFEE
Thursday afternoon, 17 September, 15:30
Main Hall

Friday, 18 September

LECTURE
Friday morning, 18 September, 9:00

9:00 Invited oral

Features of mechanical behavior of isotropic composites with metallic matrix

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The application of alloys consisting of two and more phases made possible to obtain composite materials in which high temperature strength combines with low temperature ductility and other useful properties. Strength characteristics of composite materials can be estimated from the mixing low, taking into account the dependence of

properties for every phases from the size of structural elements (for example, by Hall-Petch law) and contiguity of phases.

For example hardness of well known composite material WC-Co can be calculated by equation proposed by Lee and Gurland [1]

$H=H_{WC}V_{WC}C+H_m(1-V_{WC}C)$ (1)

In this equation H – hardness of WC-Co, H_{WC} – hardness of binderless polycrystalline WC, H_m – hardness of the binder phase in WC-Co, C – contiguity of the WC grains, V_{WC} – volume fraction of the WC phase (0.9 in WC-6wt%Co).

It was shown that H_{WC} and H_m follow the Hall-Petch relationships of the type:

$H_{WC}=H_{0WC}+K_{0WC}d^{-1/2}$ (2)

$Hm=H_{0m}+K_{0m}l^{-1/2}$ (3)

where d is the mean WC grain size, l the mean free path in the binder phase, and H_{0WC}, K_{0WC}, H_{0m} and K_{0m} are parameters that were determined experimentally.

The following relationship exists between d and l:

$l=V_m d/((1-V_m)(1-C))$ (4)

where V_m is the volume fraction of the binder (0.1 in WC-6wt%Co).

By substituting (2), (3) and (4) into (1) obtains [2]

$H=H_0+K_y d^{-1/2}$ (5)

where:

$H_0=H_{0WC}V_{WC}C+H_{0m}(1-V_{WC}C)$ (6)

$K_y=K_{0WC}V_{WC}C+K_{0m}(1-V_{WC}C)B^{-1/2}$ (7)

Equation (7) suggests that when the cobalt content is constant, a Hall-Petch-type relationship exists between the hardness of composite WC-Co and the WC grain size.

It was shown that equation (5) is true for WC-Co alloys in the wide temperature range.

The estimation of composite plasticity is the more difficult problem.

So, if composite consists of two phases: hard and metallic soft ones, elongation to fracture in tension tests can be calculated using supposition that the soft phase is only deformed.

For characterization of plasticity of brittle at standard mechanical tests composites the indentation method can be used [3]. Plasticity characteristic was introduced as a dimensionless parameter which is determined by the fraction of the plastic strain e_p in the total elastoplastic strain ε_t $\delta_H=\varepsilon_p/\varepsilon_t$.

For Vickers method

$\delta_H = 1-14.3(1-\nu-2\nu^2)HV/E$,

where n is Poisson's ratio, HV is the Vickers hardness and E is Young's modulus. The dependence of d_H on the temperature for different composites is discussed.

Dispersion hardened metallic alloys is the very important class of composite materials. Mechanisms of deformation and fracture of these materials are investigated the most fully and discussed in the presentation. Some new possibilities of dispersion hardening are considered: effectiveness of dispersion hardening metallic alloys by quasicrystalline particles and hardening by two ensembles of disperse particles, possibility of dispersion hardening metallic glasses by nano size crystalline particles etc.

For example in aluminum alloys of Al-Zn-Mg-Sc system two ensembles of disperse particle are used for hardening: h'-phase and intermetallic Al_3Sc. Variation of size and concentration of these particles made possible to find optimal values of strength and plasticity.

It was shown that in metallic glass $Al_{91}Ce_{9-x}Sc_x$ nano size particles of crystalline aluminum which are formed at x = 3 in amorphous matrix increase hardness from 2 to 3.5 GPa.

Eutectic alloys are discussed as the natural composites, which have the most advantageous mutual orientation of phases, high thermal stability and increased high temperature strength. Effectiveness of creating eutectic alloys in quasibinary sections of three components systems (e.g. Al-Ti-Cr) is considered.

In last years it was shown that one component metals of technical purity (in which phase transitions are absent) can behave as many phases composites. At that texture components play a part of phases. Texture components, which are formed in the process of thermo-mechanical treatment, have different dislocation density, different mechanical properties and recrystallization temperature.

The different properties of the texture components can lead to the so called 45^0-britleness of Mo and W alloys.

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9:30 Oral

X-ray and electron backscattered diffractometry of copper nanoparticles grown on porous silicon

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Displacement deposition technique was used to grow copper nanoparticles on porous silicon (PS). PS films were fabricated by electrochemical anodization of monocrystalline Czochralski Si wafers doped with Sb to have 0.01 Ω·cm resistivity. (100) and (111) oriented wafers were used to investigate an effect of crystallographic orientation of silicon on copper nanoparticles growth process. Self-organization into an array of a regular cylindrical pores with mesosized diameter and 1000 nm depth was achieved under 10-150 mA/cm^2 current density during 20 sec. Our previous studies have demonstrated deposition of copper nanoclusters onto the walls of partially dissolved pore channels and well faced copper nano-

particles onto an external surface of PS. The aim of the present work is to obtain crystalografic information about the upper copper nanoparticles for its further application. Using different compositions of plating solution and varying time periods and temperature regimes of displacement deposition we have fabricated copper nanoparticles of wide dimensional range from 5 to 250 nm. Usually XRD provides complete crystallographic data obtaining, but in the case of low-size objects this technique has not enough sensitivity. To study nanoparticles of sizes less than 100 nm we have also applied EBSD. It was shown, that copper nanoparticles are crystalline in nature and have cubic face centering elementary cell. In addition, the traces of Cu_2O cubic primitive crystalline phases were determined. For the first time the orientation of separate copper nanoparticles was discovered to be determined by the PS substrate, i.e. epitaxial growth of copper takes place. Further copper nanoparticles coalescence into continuous film leads to losing of copper crystal ability to inherit PS orientation. Discussing of such copper nanoparticles growth behavior and its probably applications are presented.

9:45	Oral

The mechanisms of surface discharges on C-130 ceramic materials, MOV varistors and glass with silicone rubber cover

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The paper describes mechanisms of surface discharges development on ceramic, glass with silicon rubber cover and between polymer and varistor. Underlying these mechanisms are these materials electrical strength and behavior. The type of discharge on surface or near dielectric surface depends on material on to which the electricity is to be discharge, type of both space charge and charge generated on surface, as well as on type of voltage and shape of electrodes. Flashover voltage of both insulating and power protecting systems can be increase by applying of semi – conducting covers which will carry away the generated surface charge and uniform the distribution of electric field on material surface. The above thesis was confirmed by DC, AC and high impulse voltage tests.
When it comes to glass covered with silicone rubber the surface discharges develop more readily on the glass side, what was recorded on pictures.

Posters

Monday, 14 September

Joint Poster Session

Monday & Wednesday
Monday afternoon, 14 September, 16:00
Main Hall

16:00	Poster	I-1

Development of a three dimensional in vitro model system for nondestructive investigation of osteoblast cell growth in scaffolds

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Three dimensional cell cultures for tissue engineering are possibly limited concerning nutrition and hypoxia inside the scaffolds which may lead to changes in cell behavior. The aim of the present study was to create an *in vitro* three dimensional (3D) model system to investigate the interaction of human osteoblasts with porous scaffolds in the depth of the corpus. A clamping ring was designed, in which two porous discs can be horizontally fixed. Thereby a 3D module with 4 levels with a maximal distance of 10 mm is generated. Porous discs could be made from different materials, e.g. porous tantalum (Ta) (Zimmer, Inc.), ceramic, polymer and composite scaffolds manufactured by rapid prototyping. Human MG63 osteoblastic cells (ATCC) were seeded apical onto the 3D model and placed in DMEM with 10 % FCS. After 7 and 14 days the cell ingrowth from level 1 to level 2-4 by field emission scanning electron microscopy (FESEM) and mRNA expression for proliferative and osteogenic marker genes was analyzed. Preliminary results demonstrated a cell migration through the scaffolds after 7 and 14 days on Ta and ceramic scaffolds. Cell morphology was similar in the different levels of the model system, but mRNA expression indicated an influence of the corpus depth on cell differentiation. Ta scaffolds for *in vitro* cell culture studies were normally used with a maximal height of 5 mm for the ingrowth of cells. In this study we present a 3D model with 4 levels for nondestructive cell analysis, where the maximal distance for ingrowths is 10 mm. This model is a well suited system for the investigation of structurally new designed composite implant materials. Examination of cultured cells from distinct levels will improve our knowledge about the cell growth inside a 3D corpus.

16:00 Poster I-2

Quantification of the inhomogeneity of structure distribution of multiphase materials based on the concept of directional lacunarity

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A quantitative evaluation of the microstructure of a material requires determining characteristic features of objects, such as: their number, size, volume fraction, shape and distribution method. From among the qualities mentioned, the least systematised problem is the evaluation of a mutual distribution of characteristic objects composing the microstructure. A complex quantification of the mutual distribution of structural components should take into account the concept of inhomogeneity comprising not only the reciprocal arrangement, but also the size and shape of objects forming the microstructure, as well as a multiscale methodology of evaluation.

The paper contains the results of a complex quantification of the inhomogeneity of structural components of materials subject to thermal treatment (Ti-6Al-4V after hydriding and cyclic thermal treatment) and plastic processing (analysis of the microstructure grain orientation after deformation and recrystallisation – EBSD measurement), as well as composite materials and engineering plastic materials characterised by a gradient percolation structure. In the research, a methodology was used based on a multiscale measurement and assessment of the moments of inertia in relation to the measuring field sides, and an analytical description based on the Hurst correlation, adequate to the measuring technique. The method applied enables quantifying the inhomogeneity of distribution, i.e. determining the span of local and global scales for each structure type, including percolation structures. Owing to the standardisation procedure applied, it also allows comparing the investigation results, regardless of the size of the sets analysed.

16:00 Poster I-3

Photoacoustic Transformation in Inhomogeneous Structures: Pulse Mode of Laser Irradiation

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Photoacoustic (PA) is a perspective area of the solid state physics that develops intensively. Methods of PA allow investigating a huge amount of optical, thermal and elastic characteristics ant the parameters of materials. They are successfully applied in various areas of modern material research [1]. Lets notice, that overall PA effects description, grounding on propagation and interaction of different types of fields (such as electro-magnetic, thermal, elastic), gives the key to understanding the nature of the phenomena that take place in media (which can be in the different aggregate states). Usage of the power pulse irradiation during the experiment enables to investigate nonlinear phenomena, and as the result, allows to get (for example from the second and the third harmonics) additional information about materials.

Mathematical model for the calculations of the fields of elastic displacements in inhomogeneous structures (such as composite materials) under their irradiation by short (~10 ns) laser pulses is presented in the work. As a result of such irradiation the nonstationary temperature distribution originates in material that defines the form of the acoustic response through the thermoelastic mechanism.

If we know the form of temperature distribution (for example from [2], [3]), that corresponds to definite spatial distribution of the thermal parameters of the material, we have the possibility to use such model to get temporal and spatial distribution of elastic displacements.

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16:00 Poster I-4

Role of phonons on radiation defects formation in silicon and germanium

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The influence of hard radiation on electrical and recombination properties of semiconductors is successfully used in radiation technology of parameters management on silicon and germanium electronic devices. This technology is based on controllable accumulation of radiation induced defects (RD) in Si or Ge crystals structure. Temperature of a crystal under irradiation strongly influences on the generation rate, accumulation efficiency and spectrum of dominant RD [1-2]. The experimental results of RD accumulation in silicon and germanium single crystals under 1 MeV electron irradiation at temperatures 10-650 K are submitted in this report. It is shown, that

the accumulation of most important for technology RD is determined not only balance of generation and annealing processes of secondary RD. It appears, that annihilation and dissociation of genetic Frenkel pairs (primary RD) non monotone depends on crystal temperature under irradiation. The theoretical analysis of experimental results has shown: acoustic and optical phonons influence on generation rate of RD. Phonons brake down the "hot" self interstitial atoms, created by radiation. Braking of interstitial component of genetic Frenkel pair increases the probability it annihilation with vacancy. Efficiency of free vacancy generation under irradiation accordingly is decreased and the secondary RD accumulation is accordingly slowed down too. Besides the influence of intensity of electron irradiation on annealing of dominant secondary RD in Si – A-centre is discovered. The probable mechanisms of this influence are discussed.

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16:00 Poster I-5

Amplification of evanescent acoustic waves by the layer of 2D magnetic composite

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In this work the conditions for resonance amplification by the layer of 2D magnetic composite both bulk and shear evanescent waves incident upon the materials' surface have been determined. As an example of composite medium the system of identical metal-coated ferro- and antiferromagnetic wires parallel to the Z-direction (it's also the easy axes) in the elastically isotropic nonmagnetic matrix have been considered. Analyze of this dynamical problem was carried out in the frame of effective medium method. To this end the acoustic analog of Maxwell, Garnett, symmetrized Maxwell-Garnett, and Sheng approximations that actively used in polariton dynamics of fine-dispersed media have been obtained. Received data was correlated with the results of shear bulk wave scattering by the isolated metal-coated ferro- and antiferromagnetic cylinders.

16:00 Poster I-6

Role of the structural factors in shaping of mechanical properties of galvanic composite coatings

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The abrasive resistant heterogeneous coatings with controlled gradient of the contents structure and structure by principles of determination of dependence of their physico-mechanical, tribotechnical and service properties from the geometric and physico-chemical factors of structure composition material of coating are created. The technology of electrolytic deposition gradient of coatings on working surfaces of materials, for critical conditions of friction without lubrication at presence of abrasive, aggressive mediums, influence of corrosion and temperature are developed and optimized. The condition of electrochemical synthesis of composite electrolytic coatings (CEC) and creation of dimensional effects of a structure before chemical-thermal treatment processing (CTT) CEC is investigated. The influence gradient of structure CEC for shaping of tribotechnical properties such galvanopowder compositions is marked. The couplings between a type of filling, geometry of disposition and nature of interaction of components origin CEC and after CTT are established, and at application of the concentrated power sources also. The role of filling and features of interaction between components of coating and dependence of strength of triping for interaction of galvanic metal of matrix with powder filling, from their nature and conditions of formation adhesion of transitional layers which are capable to improve tribomechanical of properties of material of coating composite as a whole was investigated. The optimization of combined method at obtaining gradient of coatings for materials that is working in critical conditions of friction is executed. The number of technological processes, necessary the equipment which successfully has approbation in production new of galvanopowder method of obtaining CTT CEC gradient type for tribotechnical purpose is developed.

16:00 Poster I-8

Characteristic of surface topography of ancient pottery

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The aim of the paper was to charcterize the surface topography of fragments of ceramic vessels found in the region of Starosiedle in Poland and Rosenhof in Germany and dated from 10th century. The work is realized in frame of the collaboration with archeologists from Institute of Archeology and Ethnology of the Polish Academy of Science (PAN). The main idea of work is to charac-

terization of topography of ancient pottery, especially the attention in concentrate of ceramic matrix, admixtures and bonding with matrix. The results of investigation of ancient pottery with using stereoscopy microscope and profilometer and reported.

For the archeologists results of examination fragments of ancient pottery will permit finding the relations between the ceramic structure and the technique of its manufacturing and, on this basis, to conclude about the development of the technology of ceramic pottery in the relation on the social and cultural background at the times of its manufacture. For the materials science and engineering scientists will permit examinining the effects of the produckt remaining in a given ground for thousand of years. This experience can be supplemented to studes of the materials that were exposed to the action of the extremal envirnment for such a long time.

16:00	Poster	I-9

Fluorescent liquid crystals

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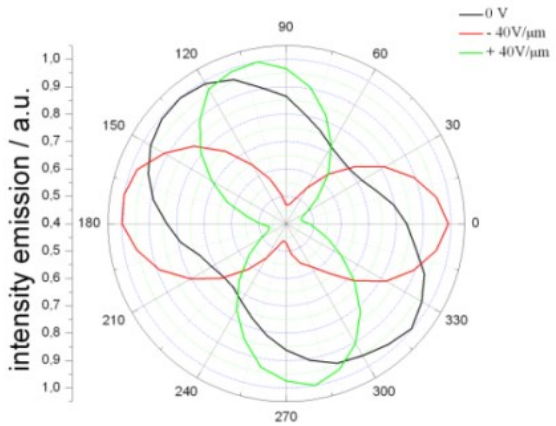
Liquid crystal materials showing strong fluorescence in visible light have been synthesized and studied. Molecules have bent mesogenic core incorporating 2-benzothiophene. Material exhibit Nematic, Smectic A and Smectic C. Due to the strong anisotropy of molecular organization in LC phases the emitted fluorescent light is strongly polarized. In chiral SmC materials polarization direction can be changed by electric field due to the ferroelectric structure of the phase. This behaviour seems promising for the future applications.



16:00	Poster	I-10

Synthesis of functional graded structures and 3D parts on their base from nitinol and titanium powders by the SLS method.

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Goal of this contribution was study of possibilities functional graded structure (FGS) fabrication and 3D parts on their base via layer - by - layered Selective Laser Sintering (SLS) method from nitinol powder (intermetallics phase - NiTi) and/or titanium. Particularities a micro and macro structures, formed under such regimes of syntheses, were determined. It was shown that FGS delamination had been revealed in the NiTi + Ti system, while homogeneity between of layers was in the system - titanium, chemical cladding by the nickel ($NiTi_2$ - phase) + Ti.

16:00	Poster	I-11

Kinetics of the melting front movement in process of centrifugal induction surfacing of powder material

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Kinetics of movement of the powdered layer melting front at the centrifugal induction surfacing makes essential impact on physical-mechanical and operating properties of applying protective coatings. This is due to the fact that the majority of surfacing materials are thermosensitive, i.e. increase surfacing temperature and time of thermal influence, as a rule, has negative influence on formation of the powder layer microstructure.

As a result of development of mathematical model of melting front movement in a powdered material the minimum surfacing time is defined. It allows to minimise negative impact of thermal influence on structure formation of applyed coating, to raise productivity of the process, to lower power inputs and to ensure saving of nonferrous and high alloys by reducing the allowance for machining.

The difference of developed mathematical model of melting front movement from previously known is that the surface temperature from which there is a heat transfer, is a variable and varies in a time under the linear law.

16:00 Poster I-12

Investigation of an optical near field along the fiber needle's surface

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In this work investigation of optical near field along the fiber needle's surface and its action on the different chemical substances are experimentally studied. For that purpose the process of photochemical vapour deposition is conducted in the chamber of scanning electron microscope that allow observe the results of the experiment before and right after the deposition.

Also the possibility of nonresonance chemical vapour deposition and overcoming the diffraction limit on the size of deposited structures are examined.

The process of electromagnetic radiation propagation in the fiber nanoneedle is studied via numerical simulations. The obtained results are in a good agreement with experimental data of nanostructures' deposition on the needle's surface.

16:00 Poster I-13

Electrochemical deposition of advanced materials based on SnO_2 using polymeric additives

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Advanced materials based on tin dioxide are widely studied because of their properties and their multiple applications. These materials can be used as transparent conducting oxide (TCO), but also as a part of photovoltaic cell or in the design of solid state gas sensors. The field of applications depends on the structural and morphological properties of the thin film. A relatively new alternative to the common additives, to control these properties is represented by polymeric additives. .

This paper reports on thin layers of tin dioxide obtained by cathodic electrodeposition on tin substrate. Tin (II) chloride with different concentrations was used as precursors.

The deposition process was optimized by varying the deposition potential, the deposition duration and respectively the additives type and concentration. Copolymers of maleic anhydride with controlled hydrophobia (vinyl acetate/methyl methacrylate) were added in the precursor solution for tailoring the surface morphology.

The structural and surface properties of the films were investigated by X-ray diffraction, atomic force microscopy, contact angle measurements and correlations composition-morphology-tehnological parameters were established.

16:00 Poster I-14

Colloidal processing of alumina-monazite composites

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Rare earth phosphates are known to possess very high melting points in the range 1900–2000 °C and have excellent thermal phase stability. Hence, they are considered excellent candidates to produce high refractory materials. Alumina-$LaPO_4$ composites are thermally stable and $LaPO_4$ successfully controls the grain growth of alumina, thus leading to enhanced thermomechanical properties.

This work deals with the shape forming of alumina-$LaPO_4$ composites by a colloidal filtration route. A $LaPO_4$ powder was synthesised by mechanochemical milling from lanthanum nitrate and dihydrogen ammonium phosphate precursors. A commercial coarse alumina was used to obtain mixtures with 20, 30, and 40 wt.% of monazite phase (A80, A70, and A60, respectively). The starting powders and the mixtures were attrition milled and its effect on the physicochemical properties was studied, focusing on the particle size and shape, as well as the colloidal behaviour as a function of pH and adsorbed polyelectrolytes. The optimisation of the dispersing conditions was studied by measuring the rheological behaviour of concentrated suspensions of the mixtures. Suspensions of alumina and alumina-monazite prepared to a solids loading of 40 vol.% were slip cast in plaster moulds. Green cast bodies were characterized through densities determination and microstructural observations. Sintering treatments were studied in order to obtain dense materials.

16:00 Poster I-15

Electrochemical corrosion properties of hydroxyapatite composite

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Recent years have seen a significant rise in number of research works concerning use of metallic and composite materials in medicine. The progress in investigations concerns in particular a group of materials being a base material for formation of human joint prostheses and dental implants.

Hydroxyapatite, $Ca_{10}(PO_4)_6(OH)_2$, is the primary mineral content of bone and calcified cartilage, representing 43% by weight of bone. It has the desirable physiochemical attributes of stability, inertness, and biocompatibility. The elastic modulus of hydroxyapatite is two orders of magnitude greater than that of collagen. The relatively low strength and toughness of hydroxyapatite, however, produced little

interest among researchers when the focus of attention was on bulk structural samples.

The investigations focused on compositions of ceramic powders based on hydroxyapatite with addition of zirconium dioxide (ZrO_2 + 8%wt. Y_2O_3 and ZrO_2 + 20%wt. Y_2O_3). Implant specimens were prepared by mixing the ceramic. The powders were axially compacted and then sintered at the temperature of 13000C for two hours.

The electrochemical corrosion behavior of ceramic composite was investigated.

16:00	Poster	I-16

Composite materials made on the basis of hydroxyapatite for bone tissue replacement

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Three types of composite implant materials are investigated. Their mechanical characteristics, biocompatibility, and the dynamics of bond strength between the biocomposites and a live rabbit bone tissue are determined in 2, 4, 10, and 25 weeks. The stress-strain state of reconstruction system bone-implant was determined by the finite element method.

The first type of the biomaterials is based on silicate glass (SG) and the synthetic (HAp) hydroxyapatite. The second type of the biomaterials was made of on SG and the natural (NHAp) hydroxyapatite. The third type of the biomaterials was made of on ultrahigh-molecular polyethylene (UHMPE) and the NHAp.

Also in the present study, a procedure is described for obtaining NHAp and HAp, as well as composite materials based on them, namely SG-NHAp, SG-HAp, and UHMPE-NHAp. The structure of bone tissue before and after deproteinization and the structure of composite materials based on UHMPE and NHAp (with different percentage) were investigated by the method of scanning electron microscopy. Some mechanical characteristics of SG-NHAp, SG-HAp, and UHMPE-NHAp biocomposite materials and the bond strength between a live bone tissue of rabbits and SG(60)-HAp(40), SG(60)-NHAp(40), UHMPE(70)-NHAp(30), and UHMPE(50)-NHAp(50) composites were determined.

The NHAp, to prepare composite materials, was obtained from the bone tissue of catle. The results of experiment, performed by the method of infrared spectroscopy, show that the protein was removed from the heat-treated specimens of bone tissue practically completely.

All the specimens implanted were punched out from the bone tissue in 2, 4, 10, and 25 weeks after implantation. The data of experiments show that the bond strength between the UHMPE(50)-NHAp(50), SG(60)-HAp(40) and SG(60)-NHAp(40) composite biomaterials and the surface of the cortical bone increases considerably in four weeks after the implantation, and reaches almost its maximum in 10 weeks.

16:00	Poster	I-17

Filled aluminum foam for impact applications

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Cellular metallic materials, also known as metallic foams, are known for their good capability of absorbing mechanical energy through the progressive deformation of their cellular structure. An open-cell foam based on AA2011 alloy is being developed at FEM/Unicamp, using a SSM technique. In this work, the mechanical properties of compression of such material are analyzed, in both semi-static and impact tests. It is also proposed the use of fluid inside the internal voids of the material, in order to achieve different mechanical properties, especially under impact. The results obtained with and without the fluid filling are compared, and their respective behavior suggest possible applications.

16:00	Poster	I-18

Adsorption properties of nano-size hydroxylapatite particles

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Studying of various cations and anions adsorption on hydroxylapatite crystals (HAP) is carried out for a long time. It is caused by wide isomorphic opportunities of natural apatite regarding two- and trivalent cations. The significant extent of the researches is devoted to the use of apatite and its modifications as matrixes for deduction of radionuclides. Adsorption and deduction of radioactive ^{90}Sr in apatite's structure are especially important in animate nature, both for the environment protection purposes, and the establishment of mechanisms of accumulation of this radionuclide in almost completely consisting of apatite bones of the man and animals.

In this work by complex of structural and spectroscopic methods (AFM, XPS, IRS) and radiometry it is shown, that nano-size crystals of hydroxylapatite have high sorptive capacity of H_2O and Sr-90. It is clarified by the small sizes of particles and non-stoichiometric structure (FIG.1). It is established, that the distribution factor of Kd of radioisotope Sr-90 between the apatite and the solution does not depend on concentration of radionuclide in the solution in an interval 4.2 - 61 Bc/ml while the size of desorption is directly proportional to this concentration. It is established, that the sorption Sr-90 on nano-crystallic HAP with the subsequent annealing at 650 ºC(transfer in a crystal condition) leads to thirtyfold decrease of the parameter desorption of isotope from the structure of apatite at insignificant decrease Kd. It is supposed that by formation of crystals of apatite of greater sizes at calcination, the isomorphic entering in structure of apatite of ions Sr^{2+} , that preliminary have formed weak bonds with a surface of nano-size crystallite apatite at its keeping in

a water solution, occurs.

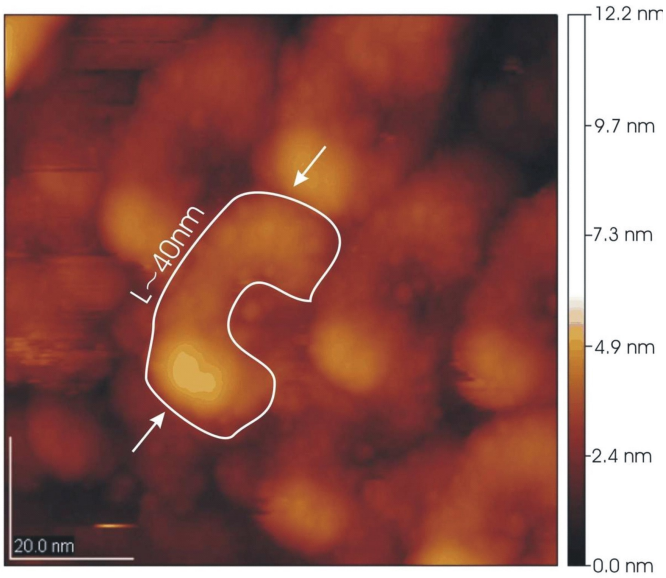

FIG. 1. Morphology of nano-dispersed hydroxylapatite.

16:00	Poster	I-19

Vitrification of lignite fly ash LFA from Polish power plants

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The results of experimental investigation concerned vitrifying of coal fly ash by the vitrification method are presented in the paper. The fly ash used was collected from thermal power plants: firing lignite. After burning the lignite for electricity, lignite fly ash LFA is produced; this is considered a waste product. Currently only a few percent of the LFA that is produced in the lignite fired power plants is used in the building industry. In most countries the LFA is disposed in the lignite mines. This can cause various environmental impacts. Wind can erode LFA deposits, harmful particles are blown into the environment. Furthermore the leaching of polluting substances to the soil and groundwater is considered a problem. The properties of glasses obtained in the vitrification process were investigated. The experimental investigations were carried out in the fixed bed reactor. The laboratory studies were performed using the plasma method of vitrification. The obtained results showed effectiveness of the vitryfication method in vitrifying of coal fly ash. Glasses produced from LFA had amorphous texture and high physical durability. Soluble salts of four heavy metals (cadmium, chromium, lead and zinc) were used for doping the fly ash. Leachability of the heavy metals used from LFA samples and from the products of vitrification was examined according to Polish regulations. Crystallisation of minerals in the vitrufication glass was investigated using the XRD method. The effect of heat treatment was examined by hardness measurement on the glass and glass-ceramic. Using of material as: material in building industry, and building and roads industry, constructional and thermo-insulating material, depend on vitryfication proces.

16:00	Poster	I-20

Laser-stimulated thermoelectronic emission from aluminum with nanotubes

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Thermoelectronic emission from the surface of aluminum with nanotubes under action of millisecond laser pulses is researched. Target (cathode) and collector (anode) were located in the vacuum camera ($p \sim 10^{-2}$ Pa) with quartz window, through which the laser ray was inserted. Cathode heating was carried out by YAG:Nd laser (λ = 1.06 µm , $q \sim 10^5$ Wt/cm^2) in the upward direction from the wire anode side. Cathode was made from Al with multilayer nanotubes. Al and mono-crystal Mo (100) were used as the standard samples. The temperature in Mo-target very center raised to 2990 K under this condition of laser irradiation.

It was found that the main way of electron emission from the cathode was that of thermoelectronic. The smallest amount of laser energy to start thermoelectronic emission was observed for Al with multilayer nanotubes and equaled 0.005 J. For aluminum without nanotubes it is equal to 0.4 J and for molybdenum - 0.8 J.

The samples were produced by inserting carbon nanotubes into aluminum matrix by Q-switched Nd-laser with flux density of energy $5\cdot10^8$ - $2\cdot10^9$ Wt/cm^2. Pressure pulses with duration regulated by the transparent condensed medium were generated.

Microstructure and chemical composition of the aluminum matrix surface with integrated nanotubes was studied by scanning electronic microscope with special block for the energy-dispersion microanalyses. Portion of the carbon varied from 5 % to 16 % and was not distributed homogeneously on the samples surface.

It was revealed that the current of thermoelectronic emission increases in two orders of that in aluminum surface with nanotubes compared with current from aluminum surface without nanotubes at the same parameters of millisecond-laser pulse action. The results of the experiments and its practical application have been analysed.

16:00	Poster	I-21

Phase diagram for the Al_2O_3–HfO_2–Y_2O_3 system

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The isothermal sections at 1250 and 1650 °C, liquidus surface and

solidus surface for the Al_2O_3–HfO_2–Y_2O_3 phase diagram were constructed for first time. Phase equilibria were established at these temperatures. No ternary compounds and appreciable solid solution regions on the base of components or binary compounds were found in the ternary system. As far as HfO_2 interacts with every other phases eutectically, this fact allows to combine in materials the unique properties of HfO_2-based T- and F-phases with the properties of other phases of the Al_2O_3–HfO_2–Y_2O_3 system. The liquidus surface is completed by eight primary crystallization fields. Four four-phase nonvariant eutectic equilibria, one four-phase nonvariant transformation equilibrium and three three-phase nonvariant eutectic equilibria were found in the ternary system. It solidus surface consists of five isothermal fields which correspond to four invariant eutectic equilibria and one invariant transformation equilibrium. The solidus surface includes also six linear surfaces of binary eutectics crystallization end. The highest solidus temperature in the system is 2810 ^oC – the HfO_2 melting point, the lowest – 1755 °C – the ternary eutectic AL+F+Y_3A_5 melting temperature. The diagram of phase equilibria and the equilibrium alloys crystallization scheme for the Al_2O_3–HfO_2–Y_2O_3 system were constructed using data on bounding binary systems, liquidus and solidus surfaces.

16:00 Poster I-22

Structural characteristics of $BiFeO_3/LaNiO_3$ artificial superlattices prepared by rf magnetron sputtering

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Artificial superlattices consisting of multiferroic $BiFeO_3$ (BFO) and conductive $LaNiO_3$ (LNO) sublayers were epitaxial grown on $SrTiO_3$ (001) single crystal substrates by a RF magnetron sputtering system. X-ray reflectivity and high-resolution diffraction measurements were employed to characterize the microstructure of these films. Formation of a superlattice structure was confirmed from the appearance of Bragg peaks separated by Kiessig fringes in x-ray reflectivity curves and a diffraction pattern. The clearly discernible main feature and satellite features on both sides of the substrate about the (002) $SrTiO_3$ Bragg peak indicate the high quality of the BFO/LNO artificial superlattice structure formed on a $SrTiO_3$ substrate. Azimuthal scans of a superlattice film in the vicinity of a surface peak and the substrate Bragg peak that clearly exhibit a fourfold symmetry with the same orientation provide firm evidence for an epitaxial relation between the film and the substrate. The fitted result from x-ray reflectivity curves shows that the densities of the BFO and LNO sublayers are slightly less than their bulk values. X-ray measurements show that these superlattice films become subject to greater tensile stress along the -axis, increased compressive stress parallel to the surface plane and increased crystalline quality with increasing temperature of deposition.

16:00 Poster I-23

Preparation of earth rare diborides and their some properties

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Rare earth diborides MeB_2 (Me=Sc, Y, Sm, Gd÷Lu) are refractory compounds with hexagonal structure AlB_2 type (пр. гр. *P6/mmm*-D^1_{6h}) and may be considered as quasilayer structures. Mechanical properties of these compounds are stipulated by boron sublattice and are similar for other diborides, whereas transport and magnetic properties are determined by electron nature of metal and essentially distinguish for diborides of different metals. Physical properties of these compounds are practically not studied in contrast with d-transition metal doborides. Today the most studied properties are magnetic ones of rare earth diborides (REE = Tb, Dy, Tm, Yb), which are shown their anisotropy connected with anisotropy of structure [1, 2].

Taking into account this feature of diboride properties for REE diboride precision study are necessary single crystal samples. We set a problem to prepare the source materials and to grow diborides single crystals from them. In this work we present the obtained results. As a source materials for further zone melting we received REE diborides (REE = Tb, Dy, Er, Lu) by synthesis from elements by arc melting procedure. As a rule, results of arc melting presents mixture of phases whose base the diboride was. The prepared from these powders rods were melted by induction crucible-free zone melting with “Kristall” unit. Optimization of technological process allowed to obtain the TbB_2 and LuB_2 single crystals. DyB_2 and ErB_2 are obtained in form of texture zone melting rods.

The obtained samples were tested by X-ray method, the Young module is estimated and Raman spectra are studied.
Rare earth diborides can be used in the composite materials.

1 Compounds with Boron: System Number 39, edited by H.Bergman et al., Vol. C11a, b of Gme-lin Handbook of Inorganic Chemistry. Sc, Y, La-Lu Rare Earth Elements. Springer-Verlag, Berlin. - 1990.
2 Buschow K.H. Magnetic properties of borides. In: Boron and Refractory Borides. Ed. V.I.Mat-kovich. Berlin-Heidelberg-New York: Springer-Verlag. 1977. – P.494-516.

16:00 Poster I-24

Preparation and characterization of porous Poly-DL-Lactide/Chitosan Composites

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The synthesis and mechanical properties of a porous PDLA/CH blend by a thermal induced phase separation technique is presented. By the optimization of several parameters like polymer ratio, temperature, solvent concentration a well controlled structures could be achieved. The materials were characterized by thermogravimetric analysis (TGA) and Differential Scanning calorimetry (DSC), evidence of the interaction between both polymers was acquired from FT-IR. The morphology of the materials was studied by Field Emission Scanning Electron Microscopy. The results showed that the TIPS technique is appropriated for the production of Porous composites.

16:00 Poster I-25

Structure and Magnetic Properties of Mn–Zn-Ferrites

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Synthesis conditions, structure and magnetic properties of Mn–Zn-ferrites prepared by spray pyrolysis of solutions of manganese, zinc and iron nitrates have been considered. The applied technique provides low-temperature (650 °C) obtaining highly dispersed (7-8 nm) powder of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ ferrite with a narrow size distribution. Synthesis of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ at low temperature avoids $Mn^{II} \rightarrow Mn^{III}$ oxidation reaction that influences negatively the ferrite properties. IR data collected from the ferrite samples obtained both in air and in N_2 ambient indicate their high structural and concentration homogeneity. Magnetic measurements confirm single-phase structure of the $Mn_{0.5}Zn_{0.5}Fe_2O_4$ powders and give no evidence of the presence of individual iron oxide phases. Curie temperature (375-380 K) is consistent with the theoretically calculated value for $Mn_{1-x}Zn_xFe_2O_4$ (x = 0.5) (T_c = 365÷385 K). Parameters of Moessbauer spectra of the ferrites are typical of Fe^{III} state in oxide solid solutions with a considerable ionicity contribution in Fe–O bonds (δ = 0.33÷0.34 mm/s). Fe^{II} state was not revealed by Moessbauer spectroscopy that indicates the absence of $Fe^{III} \rightarrow Fe^{II}$ reduction accompanying the $Mn^{II} \rightarrow Mn^{III}$ oxidation process.

16:00 Poster I-26

Forming of the structure and properties of composite material system LaB_6-(Ti,Zr)B_2

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Previously, it was found [1] that the use of solid solutions diborides as a hardening phase in self reinforced composites based on lanthanum heksaboride significantly facilitates the formation of fibers phase of diboride in the eutectic alloy and increases the frequency of the eutectic structure and mechanical characteristics of materials.

In order to establish the regularities of formation of the structural state directed crystallized composite materials of this type, a detailed study of the microstructure of a wider on the composition of a set alloy of system LaB_6-$(Ti_xZr_{1-x})B_2$ depending on the ratio of diboride components in the hardening phase (x) using methods of electron microscopy.

The results of investigation of feature of the structure of obtained samples alloy system LaB_6-$(Ti_xZr_{1-x})B_2$ from the ratio Ti/Zr showed that in all cases persist the general trends formation of a regular whisker structure, and otherness is observed only in the degree of uniformity of distribution and fiber diameter of the reinforcing phase. Established, that the most evenly structure with uniform size of fibers formed in the ratio TiB_2/ZrB_2~3/2 in the solid solution.

Using solid solution diborides titanium-zirconium as a reinforcing phase allowed to obtain samples with varying degrees of discrepancy of parameters of crystal lattices of phases and a several different stress state in the composite due to smooth changes in the crystal lattice and thermal expansion coefficient in diboride phase depending on the ratio Ti/Zr.

It was found that the maximum value of the modulus of elasticity, which corresponds to the composition LaB_6-$(Ti_{0,6}Zr_{0,4})B_2$, coincides with a maximum uniformity of the eutectic microstructure.

1.Paderno Yu., Paderno V., Filippov V. Some Peculiarities of Eutectic Crystallization of LaB_6-$(Ti_xZr_{1-x})B_2$ Alloys / Jour. of Solid State Chem. 154, 165-167, 2000.

16:00 Poster I-27

Brazing perovskite ceramics in pure oxygen atmosphere

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The brazing technology for perovskite ceramic materials with metals, and also a method of coatings deposition with high adhesion using metal melts which contain elements with high affinity to electron is developed. The braze alloys based on the Ag–Cu–O system were investigated for use in joining barium titanate ($BaTiO_3$), strontium titanate ($SrTiO_3$) and plumbum tungstate ($PbWO_4$) ceramics. Perovskite ceramic materials were successfully brazed in air and

pure oxygen atmosphere at 700°C and 1000°C. It was shown, that wetting in pure oxygen and adhesion in pure oxygen is more intensively, than in air. The microstructure of contacting phases a ceramics/metal was researched. The variant of silver-copper coating plating and the subsequent brazing of platinum electrode was developed. Silver wire can be successfully used as an electrode. Such joints have been obtained too. The strength of brazed joint was defined in shear condition. It is known, that these values strongly depends on ceramics strength. The strength of brazed samples is 40 – 50 MPa. The strength was relatively constant for all brazing conditions tested.

16:00 Poster I-28

Influence of impact pressure on a structure and properties of AL2O3-ZrO2 nanostructured powders in the process of ceramic production

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For the production of high density ceramic materials in the technician dynamic compaction of powders are used when impulses generated by explosion condense powder blanks. It is possible the proceeding of phase transformations in material, the increase of energetic potential of formed powder milieus owning of defects formation in their structure and organization of interparticle boundaries on atomic level, what increase the durability of parts. Especially use of fibrous nanostructured powders and high-energy method of loading would be very effective. In the present work results of pulse pressing nanostructured oxide powders with application of various schemes of power influence on a material are presented: a method of a running and flat wave, and also research of structure of the preparations pressed by explosion and their physical-mechanical properties after sintering. Partially stabilized zirconia and with alumina addition powders possessed the high specific surface (100-200 m2/g) and consisted of nanograins 10-25 nanometers in the size. The powder samples were treated by explosion impulse loading during which the pressure was up to 5 GPa. The velocity of detonation wave was about 4000 m/s, and time of explosion impulse was some microseconds. X-ray and phase analysis of powders has shown that in the result of influence of energy of running wave, formed during the explosion, the alterations of crystalline structure take place. In samples a crystalline lattice of monoclinic phase of ZrO_2 was distorted and its content after explosion was increased, but after annealing the tetragonal phase of zirconia come into particular prominence.

The high-energy loading made possible to obtain a finer granular structure, to activate the powder material during the following sintering, to accelerate and to intensify processes, proceeding during a thermal treatment at a lower temperature. Annealed ceramic samples possessed with density 97- 99%, micro hardness 13-14 GPa (on the base ZrO_2), and 17-19 GPa (Al_2O_3), crack resistance higher 5,6-9 $MPa \cdot m^{1/2}$.

16:00 Poster I-29

Thermal imidization of polyimide compositions

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Thermal imidization of PAA on the basis of 4,4′-diaminodiphenyloxide and pyromellitic dianhydride, modified by cinnamic, acetylenedicarboxylic acids, 4,4'-bis-(dimethylamine)-benzophenone and active filler (SiO_2) is studied by IR-spectroscopy. The choice of modifying agents is caused by the assumption, that their use will lead to the formation intermolecular cross-linking in polymer and will prevent decrease in its properties eventually. The chosen quality of filler SiO_2 allows to consider positive effect achievement in creation of the filled material at the expense of its physical characteristics, particularly the specific surface and similarity in value factor of thermal expansion to similar parametre of the polymeric binding.

Absorption spectrum of films removed on IR-spectrometer Nicolet 7101.

It is experimentally shown, that if to accept a degree of imidization of polyimide subjected to warming up to 320°C, for 100 % that the degree of imidization of polyimide, received by warming up of not modified film in the inert atmosphere at 200°C during 4 h achieves 82 %. Process of imidization of PAA, modified cinnamon and acetylendicarboxylic acids, practically does not differ from imidization of not modified PAA. At the same time the imidization of PAA, modified 4,4'-bis-(dimethylamine)benzophenone proceeds more quickly, achieving greater degree of completeness. Catalytic effect of this modifier leads to achievement of the same degree of imidization, that for not modified PAA at warming up at 200°C in an atmosphere of the drained nitrogen during1 h. As the experience implies, this routine of imidization of PAA may be using in cases of step rise of temperature up to 300-320°C for 2-3 h, which provide 100 % of imidization, can leads to degradation of properties of a product in which it is used polyimide. It is established, that the higher the quantity of aerosyl enters to the PAA, the higher - will become the speed of the rising temperature to 320°C with formation of strong films.

16:00 Poster I-30

Fire retardant polyurethane elastomers/aluminophosphorate nanocomposites for higher temperature application

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Nanocomposites offer new possibilities in the area of fire retardant polymer-based materials. Traditional flame retardant polymeric

composites, containing aluminum trihydrate and magnesium hydroxide, require a high content of the filler. Volume fractions of more than 60 weight % are needed for sufficient flame resistance in the industrial applications. Compared with micro-composites, the corresponding nano-composites show significantly improved properties at the filler content of 2-10 wt. %.

Boehmites ([Al(O)(OH)]n) are composed of Al-O double layers interconnected by hydrogen bonds between the hydroxyl groups. The size of boehmite crystals used in commercial applications is below 100 nm. An important advantage of such boehmite nanoparticles is commercial availability and possibility to tailor their interfaces (either hydrophobic, hydrophilic, silane treated and organically modified), which is crucial for their dispersion in a polymeric matrix.

This work reports the results obtained in the study aiming at evaluation of the possibility to use of phosphate-modified boehmite particles in fabrication of polyurethane elastomers nanocomposites. Polyurethane elastomers/aluminphosporates composites were prepared via in-situ polymerization from poly(ethylene adipate), 4,4'diphenylmethane diisocyanate, glycol and glycerine and 0,5 wt. % of aluminophosphorates. Two ways of the filler dispersion were employed, by (a) ultrasonic and mechanical mixing and (b) by mechanical mixing alone.

Investigations of the composite nano-structure have been carried out using high-resolution microscopy. Thermally activated phase transformations were studied using differential scanning calorimetry. Thermal stability and thermal degradation were determined by thermogrvimetric analyses. The results are discussed in terms of the possible applications of developed nanocomposites.

Acknowledgments
The authors thanks the Prof. Z. Florjańczyk , dr A.Wolak and K.Łokaj a for supplying modified boehmite.

16:00	Poster	I-31

Influence of silicas and microfillers from recycling materials on strength of epoxypolymer

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The influence of silicas (A-50, A-100, A-300, titanaerosil TAS) and recycling-fillers from polymers (PET, PS, epoxy), wood sawdust, brick dust on strength properties of polymer from epoxyresin ED-20 (+ 12,5 wt% PolyethylenePolyamine) were investigated.

It is shown, that addition of 2 wt% of silicas cause to increase tensile strength of F1 (plastic deformation) as well as F2 (finally destruction). Increasing of F1 is proportional to surface area of silica: F1 grows up to 3% for A-50, up to 4% for A-100 and up to 7% for A-300 (fig.2). It is shown increasing of F2 up to 7-18% without obvious correlation with surface area of silica. The influence of silicas to tearing strength **Q** is various. In the same manner, Q grows proportionally to the surface area of silica. The micro-hardness M demonstrates small increasing after addition of 2 wt% of silicas.

Addition of microfillers may be effective for reinforcing in several cases. Hence, addition of ruthil (TiO_2) does not change values of F1, F2 and Q (growth to 3-3,5%). In the same time addition of epoxy-polymer-poudre may increase tensile strength . Addition of PET may increase micro-hardness M .

Thus, principal possibility to improving of epoxycomposite's properties by addition of unmodified silicas and recycling-fillers was shown.

16:00	Poster	I-32

Polypropylene microfibers with filler in nanostate

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The physics-chemistry features of obtaining of polypropylene microfibers filled by the aerosils in nano state have been investigated. The microfibers are formed by the way of processing of polypropylene/copolyamide (PP/CPA) mixture melts. It has been shown that introduction of aerosil additives do not essentially influence on viscous properties on PP/CPA mixture melts in spite of solid state of aerosil. Capability of a mixture melt of being longitudinally deformed increases under influence of aerosil additives. The most important result is sharp improving of PP fiber-formation in CPA matrix at presence aerosil additives: diameters of microfibers decrease to 0. 30 ÷ 0.15μm, weight part of PP microfibers increases to 97.4% as opposed 55.4% for the binary mixture, the uniformity of distribution of microfibers on their diameters increases. The latter is confirmed by the values of distribution dispersion. This is explained by stabilizing action of aerosil additives on liquid streams of PP by way of suppression of the disturbance wave amplitude. All above-mentioned has ensured of obtaining of PP microfibers with increased in three-four times specific surface.

The stabilizing action of aerosil additives has been confirmed by data about increasing of life time of PP liquid streams in CPA matrix, and essential lowering of interfacial tension. Introduction of aerosils leads to change of phase transitions of polymers in PP/CPA mixtures. Aerosil nano particles play role of the centres of nucleation.

16:00	Poster	I-33

Thermal imidization of polyimide compositions

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Thermal imidization of PAA on the basis of 4,4′-diaminodiphenyloxide and pyromellitic dianhydride, modified by cinnamic, acetylenedicarboxylic acids, 4,4'-bis-(dimethylamine)-benzophenone and active filler (SiO_2) is studied by IR-spectroscopy. The choice of modifying agents is caused by the assumption, that their use will lead to the formation

intermolecular cross-linking in polymer and will prevent decrease in its properties eventually. The chosen quality of filler SiO_2 allows to consider positive effect achievement in creation of the filled material at the expense of its physical characteristics, particularly the specific surface and similarity in value factor of thermal expansion to similar parametre of the polymeric binding.

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16:00 Poster I-34

Cast eutectic (b-NiAl+g-Re) alloys for works in high temperature conditions

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There are represented researching results of phase equilibrium and structure of alloys on basis of ternary system Ni-Al-Re. The main attention was attached to searching of eutectic compounds with taking part of intermetallide phase NiAl. Experimentally researched structure forming peculiarity of alloys of quasi-binary cross of ternary system Ni-Al-Re. On the subsoil of phase equilibrium researching in ternary system Ni-Al-Re an area of two-phased alloys at crystallization is determined (b-NiAl+g-Re).

16:00 Poster I-35

The structure research of sintered in vacuum Ti-5.4Si after which was obtained by sputtering

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The main requirements for SOFC support are high mechanical strength, high porosity, heat-resistance, the agreement of coefficient of thermal expansion (CTE) with other components of fuel cell.

Recently, the great interest focuses on the study of Fe-Cr support for SOFC, due to meeting with almost necessary requirements. But there are some obstacles to use it in practice because of Cr diffusion in other SOFC components. Reduction of the SOFC operating temperatures till 650-700 ^oC allows substituting Fe-Cr alloys for Ti-Si.

This work is devoted to development of manufacture method of Ti-5.4Si supporter and studying of its properties. The powder was obtained with the rotated electrode sputtering method. This method allows making powder with spherical particles, what simplify to get uniform distribution of Si and high porosity in the samples. The butt-end electrode was melted by plasma. The crystallization of spherical powder occurred during flying of the melted drops in gas-cooling (Ar-He, 1:10) chamber. The average particle size was from 315 to 500 µm. The Ti-5.4Si powder was uniaxialy pressed with compaction pressures 624 and 780 MPa. The samples were sintered in vacuum (2×10^{-3} Pa) at different temperatures 1250, 1270, 1300 and 1400 ^oC. It was shown that highest porosity 37% was reached in the samples made with compaction pressure 624 MPa and sintering temperature 1300 ^oC. Three-point bend test was used for definition of mechanical properties of the Ti-5.4Si samples. The structure and fracture mechanisms of the samples were studied with SEM.

16:00 Poster I-36

Study of the the sorption of Cu(II) species on the "TiO_2 / KNO_3" interface.

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The present work is focused on the elucidation of the detailed mechanism of Cu(II) species on TiO_2 surface, which is of paramount importance for the preparation of a composite material "Cu^{2+}/TiO_2" used as catalyst, with desired properties.

The study took place by means of potentiometric titrations, adsorption edges and adsorption isotherms of Cu^{2+}/TiO_2 suspensions at 25^oC. At first, the most appropriate electrolyte was selected (KNO_3) and the pH range, where Cu^{2+} species do not precipitate, was identified. It was illustrated that the point of zero charge (pzc) and surface charge (σ^o) can easily be determined by two potentiometric titration curves, one for a suspension and another for the respective blank solution. It was found that an increase in the pH value brings about an increase to the negatively charged groups of the surface, thus increasing the adsorption of the positively charged Cu^{2+} ions. Moreover, at relatively low pH values (pH<pzc), where the surface is positively charged, an appreciable adsorption of Cu^{2+} species was found. This indicates that not only electrostatic adsorption takes place, but the copper species are deposited on the TiO_2 surface through coordination and hydrogen bonds as well. Furthermore, it was found that the ionic strength does not affect the adsorption. Finally, an increment at the pH value results in an increase in the adsorption constant and in a decrease in the lateral interaction energy (table 1).

pH	E / J mol^{-1}	k / mol^{-1} L	Γ_m / µmol m^{-1}
3	2554	342	0.48
4	211	1344	0.52
4.5	79	1533	0.71
5	15	1989	1.47

16:00 Poster I-37

The structure and properties of Ni-P/CNTs composite layers produced by chemical method

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Composite layers with Ni-P matrix and carbon nanotubes (CNTs) disperse phase have been the subject of the present authors' investigations performed recently. The composite layers were deposited on carbon steel St3Ssubstrate by chemical method.

The investigations reported in the present paper are focused on Ni-P composite layers modified by carbon nanotubes. Due to the incorporation of CNTs into Ni-P matrix it is possible to improve mechanical and tribological properties of produced materials. The Ni-P layers as well as Ni-P-CNTs composite layers have been prepared successfully by chemical method. The plating bath used in performed processes contained following components: $NiSO_4$, NaH_2PO_2, CH_3COONa, $C_3H_6O_3$ and pH value of the bath was maintained at 4.5. The Ni-P layers were produced and also examined for the sake of comparison purposes with composite layers.

Morphology and topography Ni-P layers and Ni-P/CNTs composite layers were investigated by scanning electron microscopy (SEM) S-3500N. The structure of produced layers was analyzed by transmission electron microscopy (TEM) JEOL-1200 and by X-ray diffractometer PW-1830. Tribological properties and adhesion of produced Ni-P layers and Ni-P/CNTs composite layers to the substrate were determined by scratch tester - CMS instruments. The layers microhardness was determined by applying the Vickers hardness indenter, using a load of 20 G.

Results of performed investigations have showed that the introduction of CNTs into the bath leads to important improvements of mechanical properties of Ni-P/CNTs surface layers.

16:00 Poster I-38

A novel light high-entropy alloy $Al_{20}Be_{20}Fe_{10}Si_{15}Ti_{35}$

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High-entropy alloys (HEA) have much higher mixing entropy than those of traditional alloys when compared at the liquid state or random solid solution state. HEAs have many attractive features such as: 1. tend to form simple FCC and/or BCC solid solutions, 2. easily form nano-size precipitates and even amorphous phases, 3. exhibit good thermal stability, 4. possess high hardness, and 5. display excellent corrosion resistance and thus have many potential applications. In view of the demand of high-performance low-density materials for transportation vehicles due to energy shortage and green house issues, this study has developed a new light high-entropy alloy (HEA) $Al_{20}Be_{20}Fe_{10}Si_{15}Ti_{35}$ to have unique mechanical properties and oxidation resistance. This alloy has a simple as-cast microstructure composed of one major and two minor phases with a density 3.91 g/cm^3 and a hardness HV 911. In addition, it has excellent oxidation resistance at 700°C and 900°C, which is superior over that of 304 stainless steels. Its combination of properties is unique as compared with traditional high-strength light alloys and bulk metallic glasses.

16:00 Poster I-39

Oxidation of inorganic graphene-like tungsten disulfide nanoparticles

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The progress of layered 2H□WS_2 nanochemistry opens new prospects for purposeful change of its structural-sensitive properties and multifunctional nanomaterials design: solid, radiation-resistant nanolubricants for space and ground based operation; nanolubricant additives to industrial machine oils and greases; photocatalysis on semiconductor nanoparticles etc.

Inorganic graphene-like nanoparticles 2H□WS_2 (layered nanostructures or ultrathin nanosheets) were synthesized by CVD (nanosynthesis "bottom–up"). Layered 2H□WS_2 nanoparticles have small thickness (~ 1 nm) as a result of limited layer-by-layer self-assembly of ultrathin nanosheets S–W–S. The thickness quantity of nanosheets S–W–S (in Z axis direct) is4–12by evaluation data.

By use of complex thermal analysis and X-ray investigation of oxidation on the air of layered 2H□WS_2 nanoparticles it was established that their thermal stability not change virtually (subject to oxygen desorption at 500–1300 K) as against similar value for micron particles. Oxidation of 2H□WS_2 nanoparticles occurs with formation of WO_3 and SO_2 (570–1000 K). As a result WO_3 – 2H□WS_2 heterostructures ("shell – core") are growing. Their unit cell parameters correlate with oxidation temperatures.

The acceptable termal stability of graphene-like 2H□WS_2 nanoparticles allows using them effectively as solid nanolubricants for different tribological units in modern engineering. Processes of controlled oxidation of graphene-like 2H□WS_2 nanoparticles can be used in nanotechnology of WO_3 – 2H□WS_2 nanoheterojunctions ("shell – core").

16:00 Poster I-40

Functionalized cobalt nanoparticles

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The work is related to cobalt nanoparticles. The method which enable obtaining the particles with small size distribution is presented. The thermal decomposition of octacarbonyldicobalt in solution in the presence of trioctylphosphane oxide (TOPO) and oleic acid (OA) as a coordinating ligands was used. The size was determined by SAXS method. The method provides a clean route for the preparation of the material since elemental cobalt is the only nonvolatile product of the reaction: [Co2(CO)8] -> 2Co + 8CO. The resulting powder is highly susceptible to magnetic fields generated by a small permanent magnet, suggesting that it consisted of metallic cobalt. The particles showed large Voigth and Faraday effect. The different type of nanoparticle coating (by the ligand exchange reaction) on particles organization and solubility in varies organic solvents was studied.

16:00 Poster I-41

Structure and properties of porous preforms made of Saffil fibres for the strengthening of metal alloys

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Porous ceramic preforms for reinforcing of composite materials based on copper or aluminium alloys were produced. Alumina Saffil fibres were mixed with water solution of silica binder, dried and formed to required shape. Then the firing at 950C was applied in order to produce preforms characterised by the good durability, high temperature resistance and open porosity for the infiltration with the liquid metal under high pressure. Firing effected in the forming of joints between ceramic fibres, necessary to get high green strength and at the same time open porosity. Permeability, bending and shear strength were examined to evaluate usefulness of preforms for reinforcing of composite materials by squeeze casting method. It was found out that permeability characterising passing efficiency of gases is sufficient for the passing of liquid metal front and thus infiltration of porous ceramic material. Bending and shear strength sharply decreased with the increasing porosity as a result of lower amount and quantity of joints between fibres. Microscopic observations revealed that they formed rounded bridges, relatively strong though small. They did not create sail or membrane that can disturb metal flow during infiltration. Preliminary attempts to squeeze cast perform confirmed proper strength and sufficient permeability what resulted in full dense, sound casting without deformation of reinforcement.

16:00 Poster I-42

Selective laser sintering of magnesium powders for fabrication of porous structures

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Porous biomaterials, has drawn much attention recently, because of their utility in advanced practical applications in biomedical industry such as bioactive implants and tissue scaffolds. Among different engineering methods for the fabrication of porous structure compacts for biological implants, the direct metal laser sintering (DMLS) technique is of particular interest due to its flexibility in materials, capability of fabricating 3D geometrically complex shapes and, most importantly, controllability of internal porous structures using appropriate processing parameters. In past decades, considerable research effort has been reported in the area of direct metal laser sintering. However, rarely work has previously been found on the laser sintering of magnesium powder, which may mainly due to the restriction of its highly flammable characteristic in ambient environments. Magnesium possess of excellent strength to weight ratio, good mechanical properties and high degree of biocompatibility, making it become a potential candidate for the fabrication of biological implants. The novelty of the present research lies in the fabrication of porous structures by laser sintering of magnesium powder using a continuous wave (CW) fiber laser. The laser sintering of single tracks and single layers of magnesium powders were carried out for the determination of process feasibility and the effects of various processing parameters such as laser power, scan rate, scan line spacing on the microstructural features and mechanical properties of porous structures have also been investigated. These results let us understand the microstructural evolution of magnesium powder under a continuous wave (CW) fiber laser in various processing parameters, which would enable the fabrication of porous structures for biological implants in the cheapest and quickest way, whilst achieving superior quality for the laser sintered parts.

16:00 Poster I-43

Application of a three-dimensional minimum spanning tree for reconstructing the crack pathway on fractures of WC-Co sintered carbides

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Analysis of issues related to the crack process requires a quantitative description of a problem which frequently, due to its complexity, is difficult or impossible to solve. In a number of cases, the deficiencies of a quantitative description made using classical methods are compensated for by unconventional mathematical tools.

In this study, computer simulation and a three-dimensional minim-

um spanning tree (3D MST) have been used for reconstructing the crack pathway on a stereometric measuring file acquired as a result of profilographometric analyses of the surfaces of fractures of WC-Co sintered carbides.

16:00	Poster	I-44

Taking advantage of the percolation phenomenon for simulating the crack process of brittle materials based on the example of sialon ceramics

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In the surrounding reality, we must deal with a vast diversity of material cracking processes and phenomena.

The aim of this study is to take advantage of the parameters and methods of applying percolation phenomena to model the crack process of materials based on an example of a modern engineering material, sialon ceramics. The reconstruction of the crack process and its characterisation were carried out using stereometric measuring files acquired as a result of profilographometric analyses of the surfaces of fractures of the material examined.

16:00	Poster	I-45

The Research of the Dynamic Displacements of Atoms at the Introduction of Interstitial Atoms in Ni3Al Intermetallide Containing Symmetric Tilt Grain Boundaries

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Grain boundaries are the integral part of the structure of polycrystals. Grain boundaries can be the source or flow of vacancies or interstitial atoms due to the presence of free volume. In this connection, absorption and emission of such defects take place. Such behaviour of grain boundaries can lead to the strengthening of the material. In the paper, structural reconstructions of atoms in Ni3Al alloy with symmetric grain boundaries of tilt were studied at different concentration of interstitial atoms Ni and Al. The experiment was made by the method of molecular dynamics using Morse pair potential. The calculated crystal represented parallelepiped and contained 25000-35000 atoms. The grain boundaries were located in the middle of the block. The inculcations were given randomly over the whole volume of the crystal so as stoichiometry of the crystal was not broken. The total amount of inculcated interstitial atoms was equal to 0.1 % - 1 % from the total amount of atoms of the crystal block. Then, low temperature relaxation was made. The displacements near inculcated atom were observed, the crystal energy decreased. Thereupon, dynamic relaxation during 50-100 ps at temperature 0.3 T melt took place. The research showed that inculcated atoms of Ni displaced for considerable big distances than inculcated atoms of Al. If inculcated atoms were located at the distances 5-10 Å between each other, they were able to form complexes of defects. The inculcated defects displaced in the direction of the grain boundary, if the distance between them and the boundary was 10-15 Å. But the grain boundary did not emit inculcated atoms. The following main mechanisms were observed at the displacement of atoms: a chain of the displacements of atoms, cyclic mechanisms, crowdion mechanisms. The dependences of the logarithm of diffusion coefficient on the inverse value of temperature were constructed for different concentrations of the inculcated atoms.

16:00	Poster	I-46

Structural and magnetic properties of multilayer system ferromagnetic/metallic separator/ferromagnetic

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This paper presents the investigations of structural and magnetic properties of multilayer system ferromagnetic/metallic separator/ferromagnetic. The investigation factors included structural properties (by means of X-ray dyifraction), surface and interface topology, roughness (X-ray reflectometry) and magnetic properties. Also the impact of both thickness of metallic separator and ferromagnetic sublayers and a number of bilayers repetition on magnetic properties was also investigated. The presented results of the investigation allow for drawing the following conclusions:

- it was proved on the basics of the investigations of multilayer systems ferromagnetic/metallic separator/ferromagnetic with different thickness of magnetic and non-magnetic layer than presence of ferromagnetic (F-F) and antiferromagnetic (A-F) exchange couplings depend on the quality of layers in artificial superlattice and on the state and type of free surface, superlattice structure and its roughness,
- it was proved that presence of ferromagnetic (F-F) and antiferromagnetic (A-F) exchange couplings depend on the state and type of free surface,
- it was proved that presence of ferromagnetic (F-F) and antiferromagnetic (A-F) exchange couplings depend on the superlattice structure and its roughness,
- antiferromagnetic exchange coupling depends on the thickness of the ferromagnetic layer (t_{Ni} = 14Å; 15Å and 16Å),
- no antiferromagnetic coupling (A-F I and A-F II) was observed for particular thicknesses of non-magnetic layer in multilayer systems, probably due to non-periodical growth of the layers and increased roughness in upper, lower and interface layers.

The results obtained within the work proved that existence of antiferromagnetic exchange coupling in multilayer system (ferromagnetic/metallic separator/ferromagnetic) depends on the quality of manufacturing of the artificial superlattice structure.

16:00 Poster I-47

Wear resistance composite electrolitic coating on base nikels

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Composite electrolytic coatings (CEC) of the compositions Ni+B and Ni+B+Cr3C2 were subjected to heat treatment in vacuum as well as treatment with laser, which markedly improved their tribotechnical properties. The wear resistance increased with increasing friction loads. When the coatings were melted in vacuum, their wear resistance decreased by an order of magnitude for the Ni+B coatings and by twice for the Ni+B+Cr3C2 coatings, whereas upon melting with laser by 7-21 times. This is due to the presence of inclusions of the Ni_3B boride and the Ni-B-Cr-C eutectic, characterized by high mechanical properties.

The Cr3C2 particles markedly increase the wear resistance of melted CEC thanks to additional strengthening of the Ni matrix with Cr and dispersed graphite inclusions. The Ni+B+Cr3C2 coatings have a higher wear resistance compared to that of CEC from Ni+B. The tribotechnical characteristics of coatings treated with laser are better than those of coatings melted in the furnace owing to the formation of a more disperse structure of the components having high mechanical properties. A discrete melting provides higher wear resistance compared to that in the case of a continuous melting. Herein coatings melted under an impulse regime possess a higher wear resistance than coatings melted by stripes. In the viewpoint of the minimal wear, the optimal melted area is equal to 40% for the stripes and to 25% for the impulse regime.

16:00 Poster I-48

Sintered materials for bipolar plates

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Bipolar plates (BPs) are key components of fuel cells. Functions of materials used for fuel cells include equal distribution of gas fuel and air, conduction of electricity between adjacent cells, heat transfer from the cell as well as prevention of gas leakage and cooldown. Moreover, the material must show particular corrosion resistance in cell's working conditions. Due to multifunctional nature of fuel cell plates, choice of materials used for plates is immensely difficult.

This paper presents opportunities of application of a new technology of powder sintering for creation of parts for electricity and heat generators. In this research, a sintering technology was developed to obtained sintering stainless steel bipolar plates/interconnectors.

The surface roughness was measured, and an electron spectroscopy analysis (ESCA) was conducted to verify the chemical composition of the surface layer.

The corrosion rates of the sintered materials were analysed.

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16:00 Poster I-49

Copper plating on carbon nanotube for manufacturing aluminum matrix composites

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Carbon nanotube (CNT) has been attracting much interest as a reinforce material in metal matrix composites due to its outstanding properties such as high strength and high elastic modulus. However, its entanglement due to van deer vaals interaction holds back its uniform distribution in aluminum matrix. In addition, its poor wetting property on aluminum and difference in density between CNT and aluminum have been great obstacles for manufacturing composites. In this study, in order to enhance the uniformity of distribution of CNT in aluminum matrix and increase the density of CNT, the CNT was coated with copper film by means of electroless plating. The effects of the surfactant and preplate process (sensitization and activation) on electroless copper plating on CNT were studied. Ultrasonic irradiation was applied both during dispersion of CNT and electroless plating containing preplate process. The dispersion of CNT and flatness of the plated copper film are discussed based on the changes in surfactant concentration and preplate process time. It was clearly appeared that low concentration of surfactant and short time of preplate process could promote the disentanglement of CNT and even copper plating on CNT.

16:00 Poster I-50

Raman spectroscopy studies of metal - carbon nanotubes composite materials.

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Excellent mechanic and elastic properties of carbon nanotubes (CNT's) make them an attractive candidate as a component of composite materials. Because of the tendency of CNT's to bundle together the main issue is to obtain composite materials with homogeneous distribution of the CNT's in a metal matrix. In our studies we used Raman spectroscopy to identify CNT's in a Ni-CNT's composite. The Ni-CNT's composite were produced by two kind of deposition techniques, i.e., the chemical reduction and the electrodeposition. To achieve well dispersion, functionalised nanotubes were used. Raman spectra of the composite materials were compared with results from scanning electron microscopy (SEM), and transmission electron microscopy (TEM). New peaks obserwd in Raman spectra beetwen 800 cm-1 and 1200 cm-1 can be interpreted as interaction the COOH groups with the Ni atoms or deformation of the nanotubes in Ni matrix.

16:00 Poster I-51

Laser radiation-induced effects in Raman spectra of multiwalled carbon nanotubes

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Carbon nanotubes are interesting objects for scientific investigations not only due to their unique physical properties but also due to the importance of theirs potential applications, e.g., in materials science, chemistry and physics. In this area, functionalized multiwalled carbon nanotubes (MWCNTs) are particularly interesting because they create a good compromise between their price and physicochemical properties. However, fabrication, purification and modification of well dispersible MWCNTs is arduous and sensitive to process details, thus fast and reliable method of their characterization is required. Raman spectroscopy proved to be convenient and useful method for MWCNTs characterization. However, the correct Raman spectra acquisition might be quite tricky. The absorption of laser radiation leads to significant sample heating during the measurement procedure. The temperature effect influences the Raman peak position and can shift it in a similar way as chemical sample functionalization. The coexistence of these two different effects can develop a lot of doubts in a spectra interpretation. Moreover, the laser radiation can chemically modify “as prepared” or functionalized carbon nanotubes. Two different contributions can take place. Firstly, heating can induce the carbon nanotubes transformation1. Secondly, the laser radiation can activate a chemical (and/or photochemical) reaction of functionalized MWCNTs. All these processes can modify the MWCNTs Raman spectra. In our presentation we will try to separate all these effects providing the correct Raman spectra interpretations.

16:00 Poster I-52

New Ti-Si-X eutectic composites: structure and mechanical properties

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New class of the Ti-Si-X system alloys , where X- Al, Ga, Sn, Zr, B is elaborated. Ti-“cast irons” with silicon content of 4-6-wt. % are the in situ composites in which the intermetallic strengthening phases (silicides, silico-borides) are produced during eutectic crystallization. Structure and physical and mechanical properties of a number of Ti–Si–Zr, Ti–Si–B, Ti-Si-Al, Ti–Si–Ga eutectic alloys are studied. It is shown that eutectic Ti–Si–Zr alloys reinforced by dispersed (Ti,Zr)2Si silicides in contrast to alloys based on Ti5Si3 silicides have improved mechanical properties. For the first time the as-cast Ti–Si–Zr–Sn eutectic alloy with RT plasticity of ~2 % was produced. Formation of the super dispersed eutectics based on the ternary Ti6Si2B compound in Ti–Si–B alloys allows to obtain titanium composites with enhanced high- temperature properties and an elasticity modulus of 150÷165 GPa. It may be perspective to develop novel high temperature titanium materials with enhanced rigidity. Study of aluminum, gallium and silicon joint effect in Ti–Si–Al and Ti-Si-Ga alloys showed the possibility to produce high-temperature strength titanium materials based on (a-Ti), (a2-Ti3Al), (a2-Ti3Ga) + Ti5(Si)3 binary eutectics.

16:00 Poster I-53

Composites PLLA/MWNTs produced by twin-screw microcompounder

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Carbon nanotubes (CNTs) have been gathering much attention due to their high aspect ratio, electrical, thermal and mechanical remarkable properties. CNTs can be especially useful to act as high-strength reinforcement and confer electric conductivity to polymeric composites.

Possible advantages of combining CNTs and polymers are strongly dependent on the ability to disperse and orient CNTs in the filler. Overcoming the aggregation due to van der Waals interactions between individual tubes is one of the major challenges for the development of high-performance CNTs/polymer composites. Consistent dispersion of CNTs over the matrix leads to an efficient load transfer to the CNTs and, in addition, helps to create the network for

the electrical conductivity. Several methods have been used to achieve efficient dispersion: dispersion by ultrasonication using a solvent; melt blending process using high temperature and shear forces; functionalization of CNTs; use of surfactants; in situ polymerization.

CNTs can be especially interesting when used in combination with biodegradable and biocompatible polymers, due to the large potential for biomedical applications, especially in bone tissue engineering. Furthermore, it has been shown that CNT-reinforced biocompatible polymer composite can stimulate cell growth and regeneration by transferring the electrical signals to the osteoblasts. Consequently, electrical conductive scaffolds, CNTs/biocompatible polymer, in bone tissue engineering might be a good solution to improve bone regeneration and physical properties of the scaffold. In this work, composites composed by the FDA-approved biocompatible poly(L-lactide) (PLLA) and multi-walled carbon nanotubes (MWNTs) were produced by a melt blending process, using a DSM Xplore micro-compounder. This apparatus is a micro-scale twin-screw compounder, mimicking the mixing behavior of the large twin-screw extruders. Our objective is to study the performance of the micro-compounder to produce PLLA/MWNTs nano-composites in a controllable process without solvents or toxic products involved in method, to be further applied as bone tissue engineering scaffolds. The melting and blending in the compounder was tested for 2 different mixing times; and a MWNTs loading of 3 %wt was used. In order to evaluate the method, specimens were observed by SEM and subjected to tensile tests (ISO 527-2). SEM analyses have shown that very good dispersions are achieved and, changing mixing time from 5 to 8 min, higher dispersions are obtained. Although for 5 min mixing time few 500 nm agglomerations were observed, 8 min of mixing time were necessary to observe no significant agglomerations,. Mechanical tests, comparing pure PLLA samples with composites, revealed that UTS had not increased significantly after the introduction of CNTs; but Young modulus was 7% higher for PLLA/MWNTs composites with 8 min mixing time. It has been also shown that percentage of elongation at brake was reduced in 50%.

Concluding, it was shown that the method tested is very quick and easy, highly controllable, versatile and feasible. Furthermore, the method allows the production of desirable complex shape and does not require the use of toxic substances, which are essential for the production of biomedical devices. Electrical properties will be also studied in order to verify the practical importance of a good dispersion. Further studies should optimize the process parameters in order to obtain higher mechanical and electrical properties.

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16:00	Poster	I-54

Carbon nano-onion composites

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The electrochemical properties of *small* carbon nano-onions (CNOs, 6 to 8 shells) in the solid phase, non-modified, n-CNOs, and CNOs functionalized with carboxyclic acid groups, ox-CNOs were investigated by cyclic voltammetry. The redox properties of CNO films depend on the functionalization of carbon surface. The ox-CNOs are electrochemically active at the negative potentials due to the process of carboxylic groups reduction.

The preparation and characterization of CNOs composites with tetraoctyloammonium bromide (TOABr), chitosan (Chit) and diallydimethyloammonium chloride (DDA) as ionic dopands were performed. In all cases, the amount of CNOs in the film was the same. All films exhibit typical capacitance behavior. The voltammograms show almost pseudo-rectangular cathodic and anodic profiles, a characteristic behavior for an ideal capacitor. In aqueous solutions, the n-CNO/TOABr films show behavior typical for an ideal double layer capacitor with the specific capacitance, close to 9.68 F g^{-1}. Capacitance currents obtained for CNOs/Chit and CNOs/DDA films are much higher in comparison to CNOs/TOABr layer.

16:00	Poster	I-55

Micro-Raman analysis of single-wall carbon nanotubes grown by chemical vapor deposition

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Single-wall carbon nanotubes (SWCNT) can be produced using different processes, including arc discharge, laser ablation, CVD and plasma enhanced CVD. The SWCNT investigated in this study were grown by catalytic CVD (CCVD). The CCVD method represents the most suitable technique for nanoelectronic devices applications and for CNT-polymer nanocomposites deposited as conductive thin films electrodes. The samples were prepared by dilution and sonication for different time (30 min., 1h and 1h3min.) of powders in order to obtain thin films on glass substrate by slow evaporation of the solvent. The Raman spectra in the 50-2500 cm-1 wavenumber range and images were obtained with 632 nm laser excitation through a confocal microscope. The spectra at each image point are the average of 4 s integration acquired through a l00x, 0,9 NA (numerical aperture) objective. The Raman spectra for all sample show a prominent multi-peaks (line intensity>25 000 cnt.) located around 1580 cm-1 which is identified as G mode (or TM) and corresponds to the stretching of the -C-C- bond in the graphite plane. Different G band peaks depend on the electronic properties - semiconducting -G+ at 1592 cm-1 and metallic G- at 1550 cm-1 due to the presence of free electrons. The radial breathing mode (RBM) in low frequency region, 50 -300 cm-1 (line intensity>4000 cnt.), associated of at. vibration of C atoms in radial direction is correlated linearly with the reciprocal of nanotube diameter. An estimated value of SWCNT diameter was about 2 nm which are confirmed by ultra-high resolution FEG-SEM images. The D Raman band which is observed at 1340 cm-1 (line intensity>l000 cant.), is activated in the first order scattering process of sp2 carbon by the presence of in- plane substitu-

tional hetero-atoms, vacancies or other defects. The D band is attributed to the disorder-induced vibration and the low value (below one) of D/G peak ratio implies the formation of good quality nanotubes.

16:00	Poster	I-56

Effects of the fiber/matrix interface on the fracture of C_f - SiC ceramic matrix composite

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The subject of the present study was the fibrous ceramic matrix composite Cf - SiC with nanostructured reinforcement/matrix interface.

Nanostructured barrier layer on the fibers was obtained during heat pretreatment of the applied polymer binder coating with particulate filling. Ceramic SiC matrix was formed though chemical interaction of the molten Si with C in the prepreg during liquid silicon infiltration of the carbon fiber preforms.

Structure of the fiber/matrix interface was characterized in detail by scanning electron microscopy.

The protective effects of the barrier coating on carbon fiber were tested against Si vapors and molten Si, as encountered during siliconizing process.

The effects of the phase composition and structure of the barrier coating on composite strength (flexural and impact) and fracture behaviour were analyzed.

Correlations between the organization of the nanostructured interphase and micromechanics behaviour of the stressed composite were studied.

16:00	Poster	I-57

Carbonaceous particles of onion structures for polymer matrix composites modification

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Carbonaceous particles synthesis method consisting in carbonization of cellulosic materials such as hydrate cellulose, wood chips etc., in the presence of metals of iron group is reported. As the result of pyrolysis and carbonization at the temperatures under 2000 °C of initial materials impregnated previously with iron group metals salts, further grinding of the products of carbonization and metals carbides elimination, carbonaceous particles in the form of hollow onions with outer diameter of 300-500 nm and wall thickness of 20-50 nm were obtained. The onions were characterized by the methods of granulometry, XRD, SEM and TEM. Specific electrical resistance of the nanoparticles obtained at different temperatures and it was found that the resistance of the particles synthesized at above 1500°C has order of 10-3 Ω*cm. The synthesized onions were applied to modify epoxy binder of composites reinforced with glass and carbon high-modulus cloths. It was found that by means of adding of 1-2 weight % of onions it is possible to augment coefficient of elasticity (up to 30-40%) and ultimate strength (almost double) of the composites.

16:00	Poster	I-58

X-ray analysis of thin nanocomposite layers Si(Ti)CN and their mechanical properties

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Thin and super thin TiCN system nanolayers are advanced materials for production of wear resistant coatings for cutting tools, machine components and other instruments.

The service, mechanical and physical-mechanic properties of nano composite coatings are determined substantially by the surface and size effects and also by grain boundary effects.

X-ray investigations of thin nanosized Ti(Si)CN layers, deposited by PECVD technique , have been performed. To be deposited in various conditions nano composite coatings are characterized by four structure types such as :nc-TiN, nc-TiN/a-SiCN nc-TiNC/nc-TiSi2/a-SiCN i nc-TiNC/nc-TiCl2/a-SiCN. The presence of TiN and TiNC peaks have been revealed by X-ray analyses.

It was determined that nanocrystalite sizes are about 10-20 nm. The values of nanohardness and elastic modulus are up to 31 GPa and 350 GPa correspondingly. It was determined that nanohardness of such coatings to be deposited on hard alloy and quick-cutting steel and silicon substrates raises from 10 % till 100 %.

16:00	Poster	I-59

Hydrothermal and microwave-hydrothermal synthesis of vanadia-based nanowires with large aspect ratio

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Recently, evolution of electronic devices requires size reduction of batteries. The main problem for this is complexity of size decreasing without decline of electrochemical properties. One option of the decision is use of closed volumes of devices. “Paper”-like electrodes are capable for solving the problem. In our work we suggest obtaining of 1D materials with large aspect ratio for creation of two types of electrode: composite electrodes in which nanowires could be a conductive reinforcing fibers; the paper-like cathode which consist of disordered in plane nanowires.

The aim of the present study - development of a hydrothermal and microwave-hydrothermal techniques of V_2O_5-based nanowires preparation and investigation of their physical and chemical properties for subsequent use as a cathode material in lithium-ion batteries.

$V_2O_5 \cdot nH_2O$ and $Ba_xV_2O_5 \cdot nH_2O$ xerogels were applied as precursors for obtaining of nanowires. The xerogels underwent hydrothermal or microwave-hydrothermal treatment. VO_2, V_6O_{13}, V_3O_7, $V_3O_7 \cdot nH_2O$ and $Ba_{0.25}V_2O_5 \cdot nH_2O$ phases were synthesized as a product materials. Physical–chemical properties of the materials were investigated by SEM, TEM, SAED, EDX, XRD, XPS, TG, FTIR, CV methods.

According to SEM and TEM data, obtained whiskers had 50-150 nm thickness and up to 5 mm length, that means that aspect ratio reached up to 40000/1.

The main advantage of Ba-containing nanowires was high (~140 mAh/g) discharge capacity which did not change during cycle process.

This work was supported by RFBR (grant No. 07-03-00654).

16:00 Poster I-61

Development and investigation of nanocomposites based on aromatic polyamide

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Nanotechnology opens up new horizons in the field of material science. A peculiar direction of nanotechnology is the investigation, elaboration and application of polymer materials modified with carbon nanostructures.

This work is aimed at the development of new composite materials based on thermo-resistant aromatic polyamide phenilon C-2 (FS-2).

In order to improve the characteristics of the initial material, fullerene C_{60}, fullerene soot (FS), fullerene black (FB) were used as fillers. The content of fillers in compositions made 3 mass %.

The technique of obtaining the composites consists in mixing the press-composition within the rotating electromagnetic field with the further treatment into the goods by means of compression moulding.

The results of the investigations held showed that introducing fullerene C_{60}, FS, FB into the structure of composites increases the thermo-resistance of nanocomposites by 10-15 degrees, decreases the coefficient of friction by 1.1-1.7 times and wear by 1.2 time, while leading to the improvement of strength properties.

As for the heat capacity of the elaborated composites, it increases monotonously within the investigated range of temperatures (173-548°K) until the vitrification temperature, after which it decreases rapidly. The average values of heat capacity of the composites and the initial polymer can be put into the following row:

(1,43)>FS-2(1,38)>FS-2+3%FS(1,
FS-2+3%FB(1,46)>FS-2+3%C_{60}32kJ/kg·K).

The obtained results allow considering the elaborated composites as the promising materials with the improved characteristics, which can be applied in machine engineering field.

16:00 Poster I-62

Deflocculation of nanozirconia powders

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Agglomeration phenomenon seems to be the most serious obstacle which makes difficult the nanopowders technology. Porous and non-nanometric size agglomerates attend every stage of nanopowders processing. To reduce agglomeration process in nanoceramic slurries some additions called deflocculants agents are widely used. Recently, studies on deflocculation of alumina nanopowders have met with great interest and has been studied in detail. There were found some noteworthy deflocculants agent of mentioned nanopowder, particulary monosaccharides. Because zirconia such like alumina is the most fundamental ceramic structural material there is a need to examine its properties in nano-size also.

The purpose of presented studies was to investigate, how selected deflocculants, especially monosacharides, can influence the rheological properties of ceramic slurries made of nanometric zirconia powders. Two powders chosen for this research were stabilized by 3 mol.% of yttria and were obtained by means of wet chemical synthesis using co-precipitation route. Nanozirconia powders with an average particle size of 17 and 44 nm were purchased from CEREL and Inframat Advanced Materials (IAM), respectively.

It was found that addition of monosaccharides decreases the viscosity of ceramic slurries made of nano-ZrO_2 with an average particle size of 17 nm and increases the viscosity of those made of nano-ZrO2 with an average particle size of 44 nm particles, but explanation of mentioned problems requires further studies.

Using both of the nanopowders some samples were prepared by means of slip casting method. Subsequently they were sintered at 1300°C with one- and two-step route. Sintered bodies made of nano-ZrO_2 CEREL were of high open porosity (30-40%) and had rather low relative density (60-75% of TD). Sintered samples made of nano-ZrO_2 IAM were of zero open porosity and had high relative density (>97% of TD).

16:00 Poster I-63

Electrooptic effect studies in nano-silicon doped silica sol-gel thin films

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Thin films with ca. 200 nm nc-Si particles embedded in sol-gel

SiO_x thin films (x<2) were prepared. The films exhibit a sizeable electro-optical effect, for converter devices. Owing to the marked differences in bandgap and band-structure between the nanocrystalline grains and the amorphous matrix, heterojunction like structures are formed in the interface regions, where the band offset effects reduce dramatically the activation energy and the grains act like quantum dots. In the presence of an externally applied field, the activated electrons in the quantum dots participate to conduction through the interface barriers via quantum tunneling. For localizing electrons at or near the Si/SiO_x interface, the SiO_x film needs to have a large bandgap (insulator like). It is the localized electrons and their motion under applied electric field that determine the hysteresis/memory properties and refractive index change in the films.

Ellipsometric measurements performed under electric field (3V/15 mm) displayed a refractive index change Δn = 0.02 - 0.03 (maxing out in UV and IR). Scanning tunneling microscopy (STM) on the films (deposited on conductive p-type silicon) showed for low positive bias (0 V) scans the non-conductive state having an average roughness of R_a = 4.39 nm. Increasing the positive bias to 3V the average roughness diminishes to R_a = 4.34 nm – in good agreement with the ellipsometric result of a higher refraction index under electric field. This fluctuation can be attributed to the motion induced by the injected electrons. Once electric field is removed, the surface characteristics gradually regain their initial values.

The sizeable refraction index change (ca. 20-30% that of organics, but x20-30 larger than other inorganics) makes such films suited for electro-optical modulators.

16:00	Poster	I-64

Ecological nanocomposites based on acrylic copolymers and TiO_2 nanoparticles

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Nowadays the research in the field of wood coatings is loking for new opportunities to replace toxic chemical treatments with alternatives aiming the reduction of volatile organic emissions. The paper presents research concerning a new class of ecological, water based nanomaterials for wood finishing. Designed to exhibit bio-protective and aesthetic characteristics, these new nanomaterials, consisting of a hybrid of organic and inorganic nanomaterials, are based on composites of functionalized acrylic copolymers, in water dispersion, with TiO_2 nanoparticles, enabling a superior combination of properties that cannot be achieved by using any single material. The incorporation of nanomaterials into the wood surface coatings can provide long-term protection from harmful UV radiation without significantly effecting optical clarity, gloss, colour or physical properties.

The composite based on acrylic copolymer with titanium dioxide nanoparticles showed better wood coating properties comparing with the acrylic copolymer as such. This behaviour can be correlated with the interactions of acrylic copolymer segments with the inorganic titanium dioxide, enabling the formation of a crosslinked nanometric network of inorganic and organic domains put into evidence by AFM analysis. The chemistry behind the interaction of acrylic copolymer and titanium dioxide surface was demonstrated by FT-IR analysis. Improved properties are obtained as the result of complex interactions, by esterification, etherification, salt formation reactions, between the hydroxyl groups or titanil ion species from the nanostructured titanium dioxide (inorganic phase) and the acrylic ester, carboxyl and carboxylate groups from the acrylic copolymer (organic phase). The compatibility between titanium dioxide nanoparticles and acrylic copolymer explains the stability and the improved properties of the new wood coating nanomaterials.

16:00	Poster	I-65

Mg-based nanocomposites for room temperature hydrogen storage

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Magnesium-based alloys are promising hydrogen storage materials because of their high storage capacity and low cost. It was found that the electrochemical activity of nanocrystalline hydrogen storage alloys can be improved in many ways, by alloying with other elements, by ball-milling the alloy powders with a small amount of nickel or graphite powders. The surface modification of nanocrystalline hydrogen storage alloys with graphite by ball-milling leads to an improvement in both discharge capacity and charge-discharge cycle. In this work, nanostructured composite $Mg_2Ni/LaNi_5$ materials have been synthesized using the mechanical alloying process. The new composites have been investigated by XRD, TEM, SEM and electron energy dispersion spectrum for their phase compositions, crystal structure, grain size, particle morphology and the distribution of catalyst elements. Hydrogen storage capacities and the hydriding-dehydriding kinetics of the new materials have been measured at different temperatures using a Sieverts apparatus. The results show that nanostructured $Mg_{1.5}Mn_{0.5}Ni/LaNi_{3.75}Mn_{0.75}Al_{0.25}Co_{0.25}$ composite material releases 1.6 wt% hydrogen at 295K. It is believed that the dehydriding temperature is largely controlled by the thermodynamic configuration of magnesium hydride. Additionally, doping Mg_2Ni nanocomposite materials with palladium or graphite reduces the hydriding and dehydriding temperatures, as well. The nanocrystalline metal hydrides offer a breakthrough in prospects for practical applications. Their excellent properties (significantly exceeding traditional hydrides) are a result of the combined engineering of many factors: alloy composition, surface properties, microstructure, grain size and others.

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16:00 Poster I-66

Novel hybrid nanocomposite heterostructures based on ZnO and poly-3-methylthiopen for photovoltaic devices

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Currently hybrid dye sensitized solar cells (DSSC) are considered as an important alternative for silicon based photovoltaics in a sector of cheap devices. Photoactive inorganic anode materials, such as ZnO, TiO_2 are generally used in these cells. But DSSCs have substantial shortcoming hampering their wide application. Specifically, the most effective cells are filled with a liquid electrolyte, and special conditions are needed for their storage and exploitation. Our work is directed on elaboration of composite structures for solid-state photovoltaic cells with ZnO photoanode having highly developed surface, which is interpenetrated with photovoltaically active organic polymer poly-3-methylthiophen (P3MT). The photoanode consisted of two electrochemically deposited ZnO layers: the first one was a dense layer contacting with conducting fluorinated thin oxide glass and the second one was a porous overlayer of ZnO nanorods. Interpenetration of P3MT chains between ZnO nanorods and a good contact of these p-type organic and n-type inorganic counterparts were realized through specific electrochemical polymerization of 3MT at the electrode covered with above-mentioned two ZnO layers. The polymerization was run in solutions of $LiClO_4$ and 3MT in dry acetonitrile. The best ZnO/P3MT heterostructures were obtained at potentials ~1.2 V vs. Ag/Ag^+ reference electrode. These photoactive heterostructures both in oxidized (conducting) and reduced (semiconducting) forms were characterized by UV-Vis and PL spectroscopy, SEM, dark and light I-V measurements. The results confirmed interaction and charge transfer between the inorganic and organic components and, in turn, testified to good perspectives of the developed interpenetrated heterostructures. It should be emphasized here that electrodeposition of all the materials from solutions in the present approach without applying high temperatures thus allows using conducting plastic films to achieve flexible photovoltaic cells also.

16:00 Poster I-67

Surface enhanced size-dependent photoluminescence from silver nanoparticles in silica matrix

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We report the strong photoluminescence from the spherical silver nanoparticles with sizes of 8 – 30 nm incorporated in silica matrix. Two bands were observed in PL spectra of Ag nanoparticles. The high energy band was observed only in the samples with smallest 8 and 11 nm Ag nanoparticles. It was attributed to the radiative recombination of the electrons in sp conduction band of silver with holes in d valence band. The low energy band was observed in all the samples with the nanoparticles of sizes of 8 – 30 nm. The intensity of PL spectrum was found to increase with decrease of nanoparticle size. The quantum yield of the photoluminescence was measured. It was found to be maximum in smallest 8 nm Ag nanoparticles, namely about 10^{-2} that is in 10^8 times higher than in bulk silver. We performed the calculation of the theoretical photoluminescence spectra of Ag nanoparticles with various sizes working in the frame of the model[1] of the local field enhancement by surface plasmons in Ag nanoparticles. The calculations is in good agreement with the experimental results that proves that the strong size dependent enhancement of the luminescence from Ag nanoparticles is the results of the enhancement of the electric fields of exciting and outgoing photons due to coupling of photons to surface plasmon resonance in Ag nanoparticle.

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16:00 Poster I-68

Influence of interparticle interaction on melting of gold nanoparticles in Au/PTFE nanocomposites

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We studied the temperature behaviour of the absorption spectra of Au/PTFE nanocomposites with various filling factors at 77-936 K. The obtained temperature dependences of surface plasmon energy and bandwidth in gold nanoparticles with size of 5 nm proves the aggregation of nanoparticles due to thermal softening of polymer matrix and consequent melting of nanoparticles with formation of isolated large (about 25 nm) nanoparticles. The melting of gold nanoparticles occurs at temperatures considerably lower than the melting point of bulk gold. The interaction between the gold nanoparticles significantly affects the melting of the nanoparticles. When the filling factor increases the melting point of gold nanoparticles decreases and the spectral signature of their melting become stronger.

16:00	Poster	I-69

Properties of transparent elastomer nanocomposites (polyurethane/ ZrO_2:Eu^{3+}) as materials for optoelectronic application

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Polyurethanes are engineering materials with useful physical and chemical properties. Polyurethanes copolymers consist of alternating flexible and rigid segments of different polarity. As a result they exhibit a tendency for phase separation, which depends on the length of segments, tendency for crystallization and the overall composition. This phase separation results in the formation of hard and soft domains in polyurethanes and influences their transparency.

Nanocrystalline powders have recently attracted great interest because of their unique properties, including optical ones. It has been shown that nano-ZrO_2 is an excellent host material to Eu^{3+} ions for luminophors. In the present study nanocomposites consisting of transparent PUR and ZrO_2 powders doped with Eu^{3+} ions were investigated. These composites have luminescence properties which can have various applications including document protection.

The elasticity of the nano-polyurethane composites depend on the chemistry of the matrix and on nanofiller - concentration, size and shape of particles. It should be noted in this context that inorganic nanofillers have a strong inclination to agglomerate in organic polymers. The methods for preventing this agglomeration have been the subject of the present study, which aimed at optimizing nanofiller distribution and properties of polyurethane nanocomposites.

For PUR synthesis the following chemicals were used: polycaprolactone diol, 4,4'-dicyclohexymethane diisocyanate, chains extenders. As a nanofiller zirconium oxide containing 10% Eu^{3+} was used. The nanofiller was added at 0.1% of the polymer weight. Different ways of nanofiller incorporation were investigated.

The microstructure of the obtained materials was examined by Atomic Force Microscopy in a Tapping Mode. The size analysis of nanofillers was investigated with HRSEM and TEM. The mechanical (stretching resistance, Young modulus) and thermal (DSC, TGA) properties of polyurethane nanocomposites were also investigated in addition to the analysis of transmittance and luminescence of obtained materials was performed. The results are discussed in terms of the optimizing the microstructure of the composites in question for opto-electronic applications.

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16:00	Poster	I-70

Influence of microstructure of NiO-ZrO_2 anode composite on its catalytic activity and mechanical behaviour

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The efficient operation of SOFC anode [1, 2] is strongly depends on its microstructural parameters such as particle size, composition and spatial distributions of the constituent phases. In this work microstructure, catalytic activity and mechanical behaviour of Ni-10Sc1CeSZ anode were studied.

NiO-10Sc1CeSZ composites were made with conventional powder technigue with different amount of NiO: 40-, 65-, 75-wt. %. The initial powder of NiO and ZrO_2 were studied with SEM, BET and laser granulometry. Catalytic activity of the reduced samples was characterized by conversion of CH_4 into CO_2 and temperature of achievement of the certain level of the methane conversion. Biaxial strength of reduced and non-reduced anodes was determined at ambient temperature in air by the biaxial flexure test (ball-on-ball) [3]. Anode specimens were reduced in hydrogen at the temperature of 600 °C. The fracture surfaces of non-reduced and reduced samples were studied with SEM.

Degradation of structure of composite results in decrease not only the strength but also its catalytic activity. As result, the anodes with higher nickel content have lower catalytic activity in comparison with 40-wt. % NiO–ZrO_2 anodes due to coagulation of Ni particles. Anodes with 40 wt. % NiO showed 50 % of CH_4 conversion at 500 ^oC, whereas anodes with 75-wt. % reach the same conversion level only at 550 ^oC. Comparing CH_4 conversion for anodes at 600 ^oC, which is the working temperature of intermediate temperature SOFC, it seems that 40-wt. % NiO–ZrO_2 anode reached conversion over 80 %, what is 10 % higher than for anodes with 65-, 75-wt. % NiO content. When temperature is above 700 ^oC the NiO content in anodes has no significant influence on its conversion ability.

16:00 Poster I-71

Computer modeling of nanocables structure like SiC@BN

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In conjunction with intensive development of 1D nanomaterials, an interest to technological design of composite materials, namely, nanocables representing nanotube with inner rod has been raised. Silicon carbide is well known with its semiconductive property that is why a development of nanocables SiC@BN is very promising for microelectronic and sensor equipment. This effort is to perform a theoretical investigation of different models of nanocable SiC@BN, in particular, geometry and structure of nanorod SiC, dimensional effects, electron structure, and energy of atoms bond, distribution of electron charge as well as influence of chirality of nanotube BN on electroconductive properties of nanocable.

16:00 Poster I-72

Electrical response on microstructure evolution of multicomponent CMM

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The properties of composites can be roughly divided into two categories: those that can be predicted from the properties of constituent phases, and those that cannot. In the first category we can include such properties as electrical conductivity, Young modulus, thermal expansion and hardness, and in the second, flexural strength, fracture toughness and others. The maximum toughening effect of the secondary phase in CMC materials is reached with additions of 10 to 20 vol.%, the maximum flexural strength is reached at 25-35 vol.%. At these levels, properties vs. concentrations curves have the appearance of non-monotone lines with unpredictable behavior trends. Disadvantages may result: (i) all investigators were studying materials with step of composition of about 5%. We question the adequacy of this technique; (ii) very often the investigators do not carry out analysis of structure vs. properties correlations, so that there is no possibility to design materials with new properties level; (iii) today no one has a trustworthy model for designing physical properties via microstructure design - all solutions are based on empirical or accidental specific knowledge.

When a multi-component composite is designed with an insulator as a matrix and a conductor (material with different conductivity level) as a secondary phase (filler), then both the mechanical and electrical properties of the composite are governed by a bifurcation behavior due to the formation of a continuous network of filler particles throughout the matrix.

We would like to present a new approach in the development of the CMM's microstructure and improvement in the preparation process of the raw material mixture and optimization of the sintering step. The analysis of the microstructure networks shows two typical patterns: array and random type structures. The morphology, distribution and size of conducting particles, for both structural patterns in a multi component composite, depends on the purity and dispersion of the initial powder and on the temperature and environment of the sintering process. Fluctuation in the sintering behaviour influences the structural pattern evolution. We have shown that particles size and shape and packing density of CMM are changing in a non-monotone manner and at present we have no satisfactory method of monitoring their microstructure.

In addition, the percolation theory approach can be used. This theory was developed with respect to DC conductivity. We have shown that AC conductivity gives a new possibility to understand the microstructure vs. properties correlation. It has been observed that in the multi-component composite with a certain volume fraction of the conductor Xm, there exists a threshold conductor volume fraction, Xc, above which the electrical conduction takes place through the composite. The electrical resistivity of the CMC strongly depends not only on the Xm, but also on the Xc, which in turn strongly relies on the microstructure of the composites. Following this approach, it is possible to determine basic micro structural parameters as particles shape factor (h/D), fractal index t, size factor Rm/Rd, threshold concentration Xc and irregularity of particles contour, due to the agglomeration degree, by a multiplier factor (M).

The goal of this work has been the systematic investigation of the sintering behavior, microstructure, electrical and mechanical properties of multi-component Ceramic Matrix Materials (CMM). The design of both mechanical and electrical properties is possible through the realization of a customized microstructure with controlled size, shape and concentration of additives and phases. We also propose simple methods of monitoring the evolution of microstructure of practicable CMM's and new solutions in the area of designing energy saving heating devices for wider applications.

16:00 Poster I-73

Tailoring the structure and functional properties of composites through reinforcing them with knitted metal meshes

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Reinforcing composite materials with knitted metal meshes having a loop-like structure typical for knitwear enables the use of increased resistance of composites to mechanical and thermal loads. Regardless of the nature of the matrix. and the field of application, these composites have common features in terms of the structure and relate to three-dimension reinforced materials, meshes in which ensure a series of important functional applications. The textile technique when applied to the reinforcing knitted meshes is able to provide the desired anisotropy of mechanical and functional properties that is in-

fluenced by the type of weaving, diameter and number of wires forming loops. High processing capability of the flexible knitted meshes enables formation of composites and products using different methods, including the powder metallurgy techniques, and also the processes used for plastics and ceramics.. As examples of application of composites with knitted meshes in practice one can cite sealing materials based on exfoliated graphite. Another example shall be the use of current conductive lightning protection meshes applied to the surface reinforcement for carbon- and glass-plastics in structures of aircraft and wind power station. High electric conductivity, high flexibility and sufficient strength are the major requirements to such meshes.

16:00	Poster	I-74

Electronic parameters and thermodynamic properties of phases in the Mn-Ni-B system

Nicolay Siman, Yuriy M. Goryachev, Volodymir Dekhtiaruk, Vladislav Y. Malakhov, Boris Rud, Lyudmila Fiyalka

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Manganese, nickel, and their compounds with boron are known to be promising materials as an alternative for precious materials used in microelectronics.

The electronic structure of the six most stable borides in the Mn – B system: Mn_4B, Mn_2B, MnB_2, MnB_4, MnB_6, and MnB_{12} has been investigated. The required calculation was performed using the multicluster version (MV) of group orbitals of liner combination of atomic orbitals (GOLCAO). On the basis of the calculated electronic parameters, the elastic-dynamic and thermodynamic properties as well as the formation heat of the borides under investigation were determined. These data indicate that there is a good correlation between the elastic-dynamic and the electronic-atomic parameters.

The established regularities of correlation between electronic-atomic and elastic-dynamic parameters may be used in optimization of process conditions in synthesis of composite systems based on manganese boride

Exhibition

Welcome

During the 2008 E-MRS Fall Meeting and Exhibition 16 international exhibitors presented their offer as a full spectrum of equipment, instrumentation, products, software, solutions, publications and their wide range of services. This year the exhibit will be held Sept.14 - Sept.17, traditionally - in the historical Main Building of Warsaw University of Technology in the Main Audience Hall, convenient to the technical session rooms and scheduled to coincide with the technical programme.

This paragraph will help you managing your exhibitors tour by presenting a short description of companies that would welcome you at their booths. The exhibition is open for visitors:

- Mon (Sept. 14) 12:00 - 17:00
- Tue (Sept. 15) 09:00 - 17:00
- Wed (Sept. 16) 09:00 - 17:00
- Thu (Sept. 17) 09:00 - 12:00

Company profiles

- LOT-Oriel GmbH & Co. KG

www.lot-oriel.com

We are selling products of L.O.T. and many well-known maufacturers since 1970. L.O.T.-Oriel stands for Laser, Optics, Technology. Get to know us and your contact person for the various products! Even if you are one of our long-established and loyal customers, you will be surprised at the new products in our delivery program. As you know: development never stops and our product line is always state-of-the-art. Since November 2007, the European L.O.T.-Oriel Group belongs to Quantum Design. Together with our partner company from San Diego, USA and also in Asia we provide a woldwide sales and service network for high tech products. Ask at the booth for detailed offer.

- PREVAC Ltd.

www.prevac.eu

Our Company focuses all its efforts on permanent development and implementation of the new solutions and measurement systems based on precision or vacuum technology. Prevac Ltd takes up continual development of new solutions, their implementation and selling on home and foreign markets. As the representative of world-leading vacuum technology companies on Polish, Czech and Slovak territory, PREVAC offers their wide range of components and scientific instrumentations. Our concern for quality of delivered components, systems and provided services obliged us to evolve and implement procedures that improve our quality supervision beginning form materials and subassemblies purchase through designing, manufacturing and delivery control.

- SCHAEFER Technologie GmbH

www.schaefer-tec.com

Schaefer Group has been on the European market for more than forty years with state-of-the-art equipment continuously renovating. Our catalogue of products includes instruments for material characterisation (hardness, elastic modulus, etc.), for surface measurements (topography, electric conductance, magnetic properties, etc.) and much more. In addition to state-of-the-art equipment, Schaefer Group can offer a wide set of services tailored on the customers. Schaefer Holding offers to countries in Continental Europe equipment for: **characterisation and testing** (surface & material); **the measurement of** nanoparticle, vacuum, gas flow and **magnetic fields and thin film growth rate.** Here's some of the products Schaefer can offer:

- Scanning Probe Microscopes (both in air and ultra high vacuum)
- 3D Optical Profilers
- tribo-mechanical testing machines
- flowmeters
- teslameters
- quartz balances

If you need further information on the products Schaefer Group can offer to you, ask our representatives.

- International Publishing Service Ltd.

www.ips.com.pl

Jesteśmy polską firmą działającą na rynku od 1990 roku. Zajmujemy się importem zagranicznych publikacji naukowych oraz eksportem książek i czasopism polskich. Współpracujemy z wiodącymi oficynami wydawniczymi na świecie i w Polsce, dostarczając poszukiwane publikacje zwłaszcza do bibliotek wyższych uczelni, a także instytutów naukowych i badawczych, instytucji rządowych i parlamentarnych, banków oraz przedsiębiorstw przemysłowych i handlowych.

International Publishing Service jest członkiem Polskiej Izby Książki. Firma aktywnie uczestniczy w życiu środowiska księgarskiego - bierzemy udział w warszawskich Międzynarodowych Targach Książki oraz branżowych sympozjach i konferencjach. Organizujemy wiele wystaw w bibliotekach wyższych uczelni oraz instytucjach naukowo-badawczych.

- COMEF

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COMEF is representing in Poland leading French, Japanese, American, Danish and German producers of scientific and research equipment. We exist on the market from 1993. We offer equipment, full service and technical advice concerning methods of measurement.

NEWS! Hitachi Tabletop Microscope TM-1000: The first Scanning Electron Microscope for a favourable price. To complete our range of Scanning Electron Microscopes (SEM) Hitachi has developed its first TABLETOP Electron Microscope, the TM-1000. The TM-1000 is designed for Reasearchers whose want to break through the limits of the Light Microscopy and close up to sophisticated magnifica-

tions up to 10.000X.

- NT-MDT

www.nt-mdt.com

NT-MDT enjoys a 17-year history in instrumentation created specifically for nanotechnology research, leading the field in originality, quality, and high tech development.
We strive for next-generation SPM technology, whether it be in pure modularity that allows a university or industrial lab to start with a cost-effective core product and build to a grand, multi-user research center or the ultimate amalgamation of SPM with related technologies that has resulted in ultramicrotomy for nanotomography and spectroscopy-based instruments that meld the world of imaging with the world of chemical analysis. We believe passionately in pushing the envelope for rapid innovation while still delivering superb customer service.

NT-MDT offers expert service and applications development through more than 20 representative offices and distributor centers around the globe. There are two key branch offices opened in Holland and Ireland. In the past five years, our installed base has grown to over 2000 instruments, promoting growth of both lab and research programs world-wide.

Whether you are engaged every day in nano research or just contemplating it, coupling your specific scientific knowledge and expertise with our competence in instrument creation will produce the highest quality research results currently available.

Our mission is to enable researchers, engineers and developers to conduct nanoscale research by creating ever more perfect nanotechnology instrumentation. Along the way, we maintain a global perspective, always taking into consideration the needs of student in the classroom, the researcher at the cutting edge in the laboratory, and the practicalities of industrial R&D.

Sponsors

The Official Sponsor of 2009 E-MRS Fall Meeting & Exhibit

NT-MDT short view

- NTEGRA Spectra has won an R&D 100 Award which is one of the most prestigious international awards in the field of analytical instrumentation!
- The Diploma for active participation in the second international exhibition and congress "Next-generation technologies - 21st century"
- NT-MDT is awarded for the best development NANOEDUCATOR during the second international exhibition and congress " Next-generation technologies - 21st century"
- NT-MDT Company is the winner of contest "Scientific device - 2008" timed to the second International Exhibition of Scientific Equipement "SIMEXPO - Scientific Instrument Manufacturing"
- NT-MDT is awarded by a diploma for the best nanotechnology developments (Nanotechnology assembly – 2008, Kharkov, Ukraine).
- NT-MDT is the participant of the United Nations and UNESCO project of the world culture development decade.

Satellite events

Carbon Dioxide: a new material for energy storage and a challenge for European industry

Welcome

Carbon dioxide the major product arising from the combustions of fossil fuel sources such as coal, gas, or petroleum is being emitted to the atmosphere at the level of 30 billion tons/year. Its greenhouse effect is responsible for climate change and the increasing of average temperature of the earth atmosphere.

In order to significantly decrease carbon dioxide emissions, the European plan is focussed on three steps for 2020:

- 20 % of renewable energy produced by wind turbines and photovoltaics
- 20 % of energy saving by new combustion plants able to reach 40-45 % of efficiency
- 20 % of CO_2 trapping outside the combustion stream by units operating with NH_3 or MEA absorption.

The "high purity product" : CO_2 99 % does not cost much more than 20 $/T in the new industrial plant supported by the EEC, in term of a large demonstration unit for a coal powered electrical power plant of 1000 MW, but its capital cost is close to 1 billion euro.

Many scientists are hopeful that CO_2 can become the future of oil by the production of synfuel using mixtures CO_2 and H_2 with a specific catalytic chemical reactor.

In this way CO_2 appears as one of the possibilities for high level energy storage including the network regulation from renewable energy production.
However, catalytic processes and new chemical plants are needed to develop this industry. This will need knowledge of new catalytic materials amd electro-catalytic or photocatalytic processes in order to produce synfuel, methane, or methanol with high selectivity mechanisms, and high energy efficiency.

The mixtures such as CO_2/H_2, CO_2/coal, carbon/CO_2/H_2O, direct plasma processes from CO_2 to coal gasification, or biomass transformation appear one of the major goals of the next ten years for sustainable development of energy production and the chemical industry.

Our workshop indicates the catalytic processes and the electro-catalytic processes through plasma which may help to develop this ambitious scientific programme.

PROGRAMME

OPENING SESSION

14:00 Welcome and Opening Remarks

K.J. KURZYDŁOWSKI, Warsaw University of Technology

14:15 Objectives & motivation of the workshop

J. AMOUROUX, co-chair, Professeur Génie Chimie, ENSCP/UPMC, France

SESSION I : CATALYTIC MATERIALS FOR PRODUCTION OF FUELS FROM CO_2

14:45 Methanol synthesis from CO_2/H_2 : preparation of catalysts, reactivity and mechanistic aspects

A. KIENNEMANN, A.C. ROGER University of Strasbourg, Laboratoire des materiaux, surface, procédés pour la catalyse

15:15 Artificial photosynthesis – the way to deal with CO_2 emissions

D. NAZIMEK , Department of Environmental Chemistry, Faculty of Chemistry, UMCS in LublinCEA

15:45 Application of CO_2 in chemistry

J. RYCZKOWSKI, Department of Environmental Chemistry, Faculty of Chemistry, UMCS in Lublin

SESSION II : HIGH TEMPERATURE & PLASMA PROCESSING

16:15 A topical issue for thermal-plasma based technologies : the treatment of waste and biomass

P. FAUCHAIS, A. VARDELLE, SPECTS UMR CNRS 6638, University of Limoges, France

H. LORCET, D. GUEANDOU, CEA St Paul les Durance, France

16:45 Production of syngas by gasification of waste biomass using CO_2 as the oxidant

M. HRABOVSKY, Institute of Plasma Physics, ASCR, Praha, Czech Republic

17:15 Non-equilibrium plasma of carbon dioxide to coal particle for Redox process control

J. AMOUROUX, S.V. DRESVIN, D.V. IVANOV, S.G. ZOREV, State Polytechnical University, St Petersburg, Russia

17:30 Round table and discussion

Organisers

- Jacque Amouroux
- Paul Siffert
- Jean Pierre Massue
- Urszula Narkiewicz

Homeland Security Workshop: role of Materials sciences and technologies

Welcome

9.00 Opening session by : dr Andrzej Siemaszko, Head of the National Contact Point, Poland

Session 1

Chairperson : Jean Pierre Massué France

9.15 Objectives of the Workshop : Homelandsecurity at European level, Jean Pierre Massué EMRS Past President (France)

9.35 EU research priority on Homeland security, Dr Jorge Manual Bento Silva from DGJLS E.C.

9.55 Space Techniques for the benefit of Homeland Security by a Representative of the European Space Agency.

10.15 Programme of the fourth European Security Research Conference - SRC09 - Stockholm 29 and 30 September 2009. Organised under the Swedish Presidency of the European Union by….

10.35 The US policy concerning Homeland Security and the importance attached to materials Sciences by ….US Representative.

10.55 "Security of Population in the Case of Nuclear Accident or Terrorist Act with Radioactive Material, Chernobyl accident lessons" Dr Victor Poyarkov Director TESEC Kiev Ukraine,

11.15 Discussion and Break

Session 2

Chairperson : Paul Siffert EMRS Secretary General France

11.35 "COMPOSITES INSERTS FOR BULLET- AND FRAGMENT-PROOF VESTS", by Jadwiga Polak,Iwona Kucinska, Grazyna Grabowska, Joanna Blaszczyk, Marcin H. Struszczyk

11.55 "Optimisation of Composition and Construction for Anti-trauma Products for Homeland Security", Krystyna Fortuniak, Ewa Obersztyn, Grazyna Redlich

12.15 "Multisepctral Camouflage as a Modern Form of Human Health and Life Protection", G. Redlich, A. Bartczak, Ewa Obersztyn, Elzbieta Maklewska,Magdalena Olejnik, Krystyna Fortuniak

12.35 Discussion and break for lunch.

Session 3

Chairperson : Urszula Narkiewicz Poland

14.30 "Scientific Activity of the Central Forensic Laboratory of the Polish Police: Experiences and Way Forward by: Dr Miroslaw Rosak.

14.50 New detectors of radioactive materials Prof Paul Siffert E-MRS Strasbourg France,

15.10 Polish Platform for Homeland Security - Modern Security: to be confirmed

15.30 Discussion and break

15.45 – 17.00 Round Table: Chairperson: Giuseppina Padeletti, Italy

The main topics to be tackle for the 2010 EMRS Fall Meeting Symposium in Warsaw on Homeland Security: The contribution of Materials sciences and Technologies :toward a European and international cooperation?

17.00 Closing.

FP7 RAINBOW Training workshop

Properties of semiconductor materials and devices: Structural, Spectroscopic, Electrical characterization
Organized within the project: **FP7 RAINBOW ITN**

Welcome

Since October 2009, RAINBOW is an initial Training Network gathering 13 partners in 8 European countries. It aims at training 19 Early stage researchers and 8 Experienced researchers throughout the 13 partner institutions.

The partners are located in the following countries: **France, Germany, Spain, Italy, United-Kingdom, Switzerland, Poland** and **Finland**.

The field of research is indium Nitride and its alloys. The RAINBOW academic and industrial consortium work is focused on 3 main work packages: growth, characterization and modelling of InN, InGaN alloys and heterostructures. In the RAINBOW academic and industrial consortium, the theoretical work encompasses modelling of the atomic structure and properties of the material from empirical potentials to ab initio techniques. Experiments provide correlated structural, electronic, optical and chemical information from the nano to the macroscopic scale. Our work will enable us to determine will determine the best conditions for the growth of highest quality InN and In rich (In,Ga,Al)N alloys by the main growth techniques (MOVPE, PAMBE, and HVPE). This will lead to ensure the production of reliable commercial devices such as solar cells and devices capable of operating in the Terahertz range.

The event aims at sharing knowledge on the subject and training young researchers. It will also enable the RAINBOW partners to state their progress through the project after the first year of collaborative work.

The event is focusing on:

- Experimental results on the structure and optical properties
- Growth mechanisms
- Structure of defects that form inside the layers
- Strategies to grow high quality layers
- **Workshop**: Properties of semiconductor materials and devices: Structural, Spectroscopic, Electrical characterization

The event is directed towards the RAINBOW partners and young researchers but also open to the EMRS attendees.

Programme

Wednesday 16th: Optical and electrical properties of nitride semiconductors

8:30 - 9:15 W1 Spectral properties of InN and related compounds from first principles **Prof F. Bechstedt, FSU Jena**

9:45-10:30 W2 Sources of doping for InN bulk and surfaces **Prof. C. Van de Walle, UCSB, Santa Barbara**

11:30-12:00 W3 Interface, bulk and surface electronic properties of InN **Dr. T. Veal, Univ. Warwick**

14:00-14:45 W4 New twists in carrier recombination and hot carrier scattering in light emitting devices and field effect transistors based on III-V nitride semiconductors **Prof. H. Morkoc, VCU, Virginia**

14:45-15:30 W5 Capacitance and surface voltage charge techniques **Dr. D. Cavalcoli, Univ. Bologna**

15:50-16:35 W6 Optical properties of low-dimensional nitride semiconductors **Dr. R. Butté, EPFL**

16:35-17:20 W7 Optical properties of bulk-like nitride semiconductors **Dr.R. Goldhahn TUIL Ilmenau**

Thursday 17th: Structural characterization by transmission electron microscopy

9:00-10:30 W8 Fundamental principles of Transmission electron microscopy **Prof. Pierre Stadelmann, EPFL Lausanne**

11:00-12:30 Practicals: Simulation and quantitative information retrieval **P. Stadelmann,S. Kret IFPAN Warsaw, P. Ruterana CIMAP Caen**

14:00-15:00 W9 The resolution in aberration corrected microscopy, **Prof. Angus J. Kirkland Oxford University**

15:00-15:45 Practicals

16:00-17:00 W10 Focal and Tilt series reconstructions **Prof. Angus J. Kirkland**

17:15-18:00 Practicals

Organisers

RAINBOW Initial Training Network
http://rainbow.ensicaen.fr

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Materials Brokerage Event

Welcome

The event is directed to the most potential research organisations and enterprises **from European Union and partnering IncoNet EECA Eastern Europe and Central Asia countries (EECA)** (Russia, Ukraine, Kazakhstan, Uzbekistan, Armenia, Azerbejdżan, Moldova, Belarus, and Georgia) interested in active participation in the Seventh Framework Programme (FP7)
The main aim of the event is **to bring together the skills and knowledge** of the potential consortia members from the EU and EECA region and **to present concrete project ideas** for future successful proposals within NMP 2010 calls. The event will also create the opportunity **to transfer the experience, expertise and contacts** between successful Project coordinators from EU to EECA potential partners, who will have the chance to realize their ideas and scientific potential via Framework Programme.
The selection of the event timing was depended on the planned date of FP7-NMP-2010 Work Programme to make the planned bilateral/ mutual meetings of participants **more effective and call oriented**.
Focusing on NMP theme within the FP7, the following specific research areas have been identified for this Brokerage Event:

- Hybrid materials for electronic and photonic applications
- Bio- and chemosensing materials in health, safety and security applications
- materials for energy applications, including energy thermo conversion and energy storage
- Nanocristalline materials with novel functionalities and tools for their development
- Nanomaterials characterization

These above mentioned areas, reflect also new challenges defined by the **Green Car** and **Energy Efficient Building** initiatives, belonging to the Recovery Package for the European Economy recently decided by the European Council.

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Programme

----- Day 1 -----

9:00 - 9:30 Registration

9:30 - 10:00 **Opening session**
*(moderator: Dr***Andrzej Siemaszko***, Director of NCP Poland)*

Openning address
Jerzy Duszyński, Vice-Minister, Ministry of Science and Higher Education of Poland (TBC)
Herbert von Bose, Director of Directoriate G Industrial Technologies, DG Research, European Commission,
Wlodzimierz Kurnik, rector of the Warsaw University of Technology (TBC)
Krzysztof Kurzydłowski, Technical University of Warsaw, Chairman of EMRS Fall Meeting (TBC)

10:00 - 10:30 Plenary Session I
*(moderator:***Zygmunt Krasiński***, NCP Poland)*

10:00 Research on materials as the part of Polish research area. **Krzysztof Kurzydłowski**, Technical University of Warsaw

10:15 European Technological Platform on Advanced Engineering Materials and Technologies (EuMaT): activities and role in defining European strategy on research on materials

10:30 - 11:00 Coffee break

11:00 - 12:30 Plenary Session II
*(moderator:***Jarosław Piekarski***, NCP Poland)*

11:00 NMP-Team Project. **Anastasia Constantinou**, NMP-Team coordinator, Help Forward, Grece (TBC)

11:15 IncoNet EECA project (Project coordinator, TBC)

11:30 Directions of research in NMP: open calls, future plans. **Marcin Sadowski**, EC, DG RTD, D6 Industrial Technologies

12:00 International Cooperation (INCO) activities within FP7

12:30 - 14:00 Lunch

14:00 - 15:30 Partnering sessions (two parallel sessions)
Session A - Materials for electronics and photonics
Session B - Bio- and chemosensing materials in health, safety and security applications

15:30 - 16:00 Coffee break

16:00 - 17:30 Partnering sessions (two parallel sessions)
Session A - Materials for electronics and photonics - cont
Session B - Bio- and chemosensing materials in health, safety and security applications - cont

----- Day 2 -----

9:00 - 10:30 Partnering sessions (two parallel sessions)
Session C - Nanocristalline materials with novel functionalities and tools for their development
Session D - Nnomaterials characterization

10:30 - 11:00 Coffee break

11:00 - 12:30 Partnering sessions (two parallel sessions)
Session E - Materials for energy termoconversion and energy storage
Session F - Materials for improvement of energy efficiency in construction sector

12:30 - 14:00 Lunch

14:00 - 14:45 Partnering sessions (two parallel sessions)
Session E - Materials for energy termoconversion and energy storage - cont
Session F - Materials for improvement of energy efficiency in construction sector - cont

14:50 - 15:30 Repporting session
*(moderators:***Joanna Stalewska**,**Jaroslaw Piekarski***, NCP Poland)*

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IPPT PAN

NMPTeAm

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Index

A

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Abdinov, Ahmed S., 55
Adamkiewicz, Witold, 70, 173
Adamo, Carolina, 82
Adhikary, Sourav, 87
Adley, Catherine, 118
Aeschlimann, Martin, 99, 106, 116
Agouram, Said, 74
Ajagunna, Adebowale O., 13, 28, 47
Akasaki, Isamu, 41
Akselrud, Lev, 217
Alexandru, Horia V., 161
Alexeenko, Alexandr, 226
Ali, Ehesan, 106, 106
Aljerf, Moustafa, 152
Al-Jumaily, Ahmed, 83
Allenspach, Rolf, 108
Allodi, Giuseppe, 107
Al Subaïe, Metlak, 120
Alves, E., 50
Alves, Eduardo, 113
Amano, Hiroshi, 41
Anane, Abdelmadjid, 98
Andrearczyk, Tomasz, 103
Andrievskaya, Elena, 190
Androulidaki, Maria G., 28, 47
Ansermet, Jean-Philippe, 115
Antohe, Stefan, 59
Antolak-Dudka, Anna, 168
Anufrieva, Tatyana A., 173
Arab, Louiza, 60, 60
Arabski, Jacek, 110
Ardavan, Arzhang, 101
Arkenbout, Anne, 98
Arous, Arshak, 131, 190
Arregui, Francisco J., 122
Arroyo-Rojas Dasilva, Yadira, 35
Arruda, Antonio Celso F., 124, 210
Arshak, Khalil, 118, 131, 190
Arslanov, Rasul K., 112, 113
Arslanov, Temirlan R., 112, 113
Artzi-Gerlitz, Reit, 119
As, Donat J., 47
Aschenbrenner, Timo, 46
Asıl, Hatice, 54
Atkinson, Del, 108
Attanasio, Carmine, 204
Attaran kakhki, Ebrahim, 59
Atwi, Ali, 121
Avetisyan, Valeriy, 223

B

Baadji, Nadjib, 105
Bacakova, Lucie, 128, 195
Bacıoğlu, Akın, 161
Bader, Rainer, 205
Bagliuk, Gennadii A., 193, 193
Bahir, Gad, 40
Bala, Henryk, 179
Balaguer, Maria, 140
Balakhonov, Sergey V., 223
Balazsi, Csaba, 122, 133
Balázsi, Katalin, 133
Baluc, Nadine, 163
Balucani, Marco, 55, 204
Bandarenka, Hanna, 55, 204
Barabash, Maksym, 216
Barai, Sergei G., 214
Barnaś, Józef, 112
Baron, Denis, 141
Barraud, Clement, 98, 98, 109, 110
Barros, André, 185
Bársony, István, 124, 129
Bastek, Barbara, 52
Battistig, Gábor, 124
Baumann, Stefan, 136, 136
Baumes, Laurent A., 121
Bałkowiec, Alicja Z., 171
Beaufrand, Jean Batiste, 110
Beaurepaire, Eric, 110
Bechstedt, Friedhelm, 24, 55
Beck, Ulrich, 205
Bedel, Elena, 100
Bednarcik, Jozef, 154, 154
Bednarek, Paulina, 191
Beitelschmidt, Denise, 153
Bekenev, Valeriy L., 228
Belan, Irina, 188
Belenko, Sergey V., 85
Belkacem, Youcef, 61
Bellucci, Stefano, 222
Bencharif, Mohammed Fakhri, 60, 60
Benkstein, Kurt D., 119
Berbecaru, Ciceron A., 161
Berchenko, Nicolas N., 86, 87
Bergemann, Claudia, 205
Bergenti, Ilaria, 98, 99, 107, 110, 110, 111
Bernard, Gil, 17
Bertram, Frank, 52
Bertulis, Klemensas, 78
Betz, Michael, 136
Beznosyuk, Serge A., 56, 56, 104, 144, 189, 196
Bhattacharya, Arnab, 17
Bičiūnas, Andrius, 78
Biederman, Hynek, 195
Biermann, Amelie, 23
Bijani, Shanti, 67
Bil, Monika, 124
Bilyk, Igor S., 93
Bissig, Vinzenz, 157
Bjørheim, Tor S., 140
Bläsing, Jürger, 16

Blazquez, Javier S., 158, 169
Bliznakov, Stoyan, 201
Bobbert, Peter A., 109
Bobo, Jean-Francois, 100
Bobryk, Ewa, 224
Boccaleri, Enrico, 129
Bock, Nathalie, 105
Boćkowski, Michał, 49
Bogatu, Cristina, 209
Boiko, Volodymyr, 72
Bojar, Zbigniew, 170
Bonanni, Alberta, 102
Bonanos, Nikolaos, 141
Borgatti, Francesco, 99
Borodajenko, Natalija, 192
Borodzinski, Andrzej, 160
Borowiak - Paleń, Ewa, 176
Bougerol, Catherine, 50
Boughelout, Abderrahmane, 42, 62
Boukari, Samy, 110
Boukezzata, Assia, 61
Bouwmeester, Henny J., 148
Bouzehouane, Karim, 98, 98, 109, 110
Bowen, Martin, 110
Bozek, Peter, 119
Bradby, J. B., 158
Braidy, Nadi, 167
Bram, Martin, 144
Brancewicz, Ewa, 128
Branford, William R., 113
Braun, Jürgen, 116
Brazda, Petr, 164
Braz Fernandes, Francisco M., 169
Bredol, Michael, 203
Breslavskiy, Igor A., 45
Briddon, Patrick R., 185
Bridges, Greg E., 94
Brighet, Amer, 61
Briot, Olivier, 17
Brocq, Mathilde, 201
Brodnikovskyi, Dmytro, 216
Brodnikovskyi, Iegor, 227
Broeckmann, Christoph, 136, 138
Brown, Gail J., 80
Bruinink, Arie, 134, 134
Brunel, David, 110, 110
Bruyer, Emilie, 148
Brynk, Tomasz, 163, 221
Buaki, Mireia, 121
Buatier de Mongeot, Francesco, 105
Buchanan, Douglas A., 94
Buchkremer, Hans Peter, 140, 143, 144
Budes, Matei, 161
Budianu, Elena N., 87
Budniak, Julia A., 170
Bueno Baques, Dario, 132
Bugajski, Maciej, 80, 81
Burbelo, Roman M., 206
Burriel, Mónica, 136
Bury, Wojciech, 73
Burya, Alexandr, 224
Butler, Les, 130
Butté, Raphaël, 27, 38
Bystrzycki, Jerzy, 170
Byun, Young Chang, 137

C

Cabaj, Joanna, 127
Caballero-Flores, Rafael, 179
Caban, Piotr, 173
Calka, Andrzej, 155
Calle, Fernando, 12, 16, 23
Calleja, Enrique, 17, 23, 40
Calleja, Jose M., 23, 40
Calvo, Vincent, 14, 18
Can, Musa M., 112
Cantin, Jean Louis, 104
Cardoso, Guinéa Brasil C., 124
Cariati, Brunella, 125
Carlegrim, Elin, 107
Carlin, Jean-François, 38
Cartwright, Alex, 34
Casoli, Francesca, 99, 111
Castillo, Nancy, 175
Cavalcoli, Daniela, 27
Cavaleiro, Albano, 143
Cebulski, Józef, 84
Celichowski, Grzegorz, 132
Cempura, Grzegorz, 167
Chacón Nava, Jose G., 213
Chai, Guangyu, 45
Chakrabarti, Subhananda, 50, 81, 87
Chambard, Gaetan, 14
Chang, Chia-Cheng, 197
Chang, Chia-Seng, 195
Chang, Yuan-Chih, 195
Chapman, John, 99, 111
Chari, Abdelhamid, 56, 60, 60, 182
Charlier, Jean Christophe, 122
Charlton, Timothy, 108
Chaudret, Bruno, 199
Chauvat, Marie P., 16, 35
Chauveau, Jean-Michel, 48
Chedryk, Valeryi, 227
Chen, Cheng-Han, 160
Chen, Chia-Hao, 21
Chen, Hsiang-Hsin, 192
Chen, Jin-Ming, 115
Chen, Jun, 31
Chen, Mao-Sung, 160
Chen, Shin-An, 115
Chen, Tien-Hao, 115
Chen, Wen-Chang, 192
Chen, Yonghai, 22
Cheng, Keng-Hao, 196
Cheriet, Abdelhak, 61
Chernyshev, Leonid I., 188
Cherpak, Vladyslav, 48

Cheung, Maurice, 34
Chiappe, Daniele, 105
Chikamatsu, Kentaro, 54, 226
Chin, Hong-Seok, 146
Chin, Tsung-Shune, 217
Chindaduang, Anon, 54
Chmiela, Jerzy, 206
Cho, Min Young, 57, 61, 62
Cho, Sung-Woo, 39
Cho, Woon Jo, 175
Choi, Hyun Young, 57, 61, 62
Choi, Suk-Ho, 57
Choińska, Emilia, 133, 221
Chornobuk, Serhiy, 194, 194
Chou, Ta-Lei, 115
Chow, Lee, 45, 103
Chowdhury, Sauvik, 87
Christen, Juergen H., 52
Christmann, Gabriel, 38
Chubarov, Mikhail, 66
Chubenko, Eugene, 55
Churagulov, Bulat R., 223
Cieślak, Grzegorz, 168
Cinchetti, Mirko, 99, 116
Ciupiński, Łukasz, 133, 221
Ciurzyńska, Wanda, 178, 182
Clavaguera-Mora, MªTeresa, 159
Clouchard, Marie-Claude, 120
Cobet, Christoph, 47, 52
Coey, Mike, 9
Cohen, Lesley F., 113
Colomban, Philippe, 141, 142, 142
Colomer, María T., 209
Comanescu, Florin, 222
Comini, Elisabetta, 126
Conde, Agustin, 175
Conde, Alejandro, 158, 169, 179
Conde, Clara F., 158
Conde, Olinda, 113, 113
Coquillat, Dominique, 76
Corfdir, Pierre, 20
Corma, Avelino, 121
Cornia, Andrea, 105
Corredera, Pedro, 18
Correia, Maria R., 67
Coskun, Cevdet, 54
Couvrat, Mathieu, 201
Croce, Gianluca, 129
Croft, Joseph, 224
Cubukcu, Murat, 104
Cultrera, Luca, 69
Cunha, Antonio F., 60
Cuvilly, Fabien, 201
Cywiński, Grzegorz, 30
Czeppe, Tomasz, 156
Czernecki, Robert, 31, 49, 64
Czerwinski, Andrzej, 88
Czyperek, Michael, 148
Czyrska-Filemonowicz, Aleksandra, 167
Çınar, Kübra, 54

D

D'yachkov, Pavel N., 228
Dabbarh, Sabine, 138
Dąbrowska, Sylwia, 155
Dabrowski, Bogdan, 139
Dadgar, Armin, 52
Danelska, Anna W., 199, 202, 224
Danieli, Chiara, 105
Danz, Norbert, 134
Das, Jayanta, 154
Daudin, Bruno, 50
Davies, Hywel A., 156, 181
Davydov, Volodymyr, 217
Day, Austin P., 18, 71
Deák, András, 43, 124
Dearden, Geoff, 224
Dediu, Valentin A., 98, 99, 105, 107, 110, 110, 111, 123
Deibuk, Vitalij G., 57
Deicher, Manfred, 39
Deineka, Tatyana G., 155
Dekhtiaruk, Volodymir, 229
Dekhtyar, Yuri, 123
Del Villar, Ignacio, 122
Demyanenko, Mikhail A., 88
Denisov, Igor A., 90
Deparis, Christiane, 48
Deranlot, Cyril, 98
Deranlot, Cyrile, 98, 109, 110
DeRenzi, Roberto, 107
Derevyanko, Aleksander V., 207
Derewnicka, Daniela, 182
Derlyukova, Lyudmila E., 173
Detchprohm, Theeradetch, 13, 15
Dhesi, Sarnjeet S., 108
Diaz, Pilar, 67
Diaz, Rosa E., 58
Di Forte Poisson, Marie-Antoinette, 12
Dima, Antonela, 224
Dima, Mihai O., 224
Dimakis, Emmanouil, 32
Dimitrakopulos, George P., 13, 52, 58, 62
Dimitrocenko, Lauris, 66
Dinescu, Adrian, 87, 222
Dinh, Duc V., 29
Dionigi, Chiara, 105
Dlubak, Bruno, 98
Dmitriev, Alexey, 32
Dmitruk, Igor, 226, 226
Dobosz, Romuald, 169
Dobrovolsky, Valentin N., 92
Dobrowolski, Witold, 68
Dolgopolova, Emma A., 85
Dolgov, Leonid, 174
Domagala, Jaroslaw, 39, 53, 64
Dominik, Pawel, 70, 173
Domuchowski, Wiktor, 103
Doroshenko, Andrey G., 155

Dospial, Marcin J., 177, 177, 181, 181
Doualan, Jean Louis, 16
Dronskowski, Richard, 140
Drube, Wolfgang, 102
Druce, John W., 137
Du, Chuan, 98
Duangkaew, Pattasuda, 54
Dubiel, Beata, 167
Dubis, Alina, 133
Dubova, Ksenia L., 173, 174
Dudek, Agata K., 209
Dudina, Dina, 152
Dukhnenko, Anatoliy, 212
Dulina, Nadya A., 164
Dulińska-Molak, Ida O., 132
Dumitrescu, Lucia I., 225
Dumont, Jacques A., 102
Duque, Carlos A., 59
Duque, Carlos M., 59
Durbin, Steven M., 32, 34
Durov, Oleksiy, 213
Dussaigne, A., 20
Duś-Sitek, Marta, 219
Duta, Anca, 209
Dutkiewicz, Jan M., 156
Dutkiewicz, Michał, 73
Dvoretsky, Sergei, 86, 88, 91
Dybiec, Henryk, 162
Dybko, Krzysztof, 63
Dziawa, Piotr, 103
Dzyadukh, Stanislav M., 93, 94

E

Ebert, Hubert, 116
Ebothe, Jean, 198
Ebrahimizadeh Abrishami, Majid, 59
Echegoyen, Luis, 133, 222
Eckert, Jürgen, 153, 153, 154
Edwards, David, 108
Edwards, Paul R., 18, 71
Edwardson, Stuart P., 224
Eickhoff, Martin, 42
Eisenhardt, Anja, 22
Elhamri, Said, 80
Elliman, Robert G, 57
Elter, Patrick, 205
Enculescu, Ionut Marius, 59, 115
Enesca, Alexandru I., 209
Engels, Stefan, 138
England, Gordon, 71
Erdély, Robert, 43
Ergun, Yuksel, 79
Esaev, Dmitriy G., 88
Escolastico, Sonia, 140, 145
Esser, Norbert, 23, 47, 52
Ewald, Marcel, 23
Eyvazova, Goncha M., 161, 185

F

Faglia, Guido, 126
Fahlman, Mats, 107, 110
Falkovsky, Leonid A., 29
Falkowski, Paweł, 165, 199
Falyouni, Farid, 74
Fazakas, Eva, 166
Fediv, Volodymyr I., 69, 115
Fedorchenko, Irina V., 112, 113
Feduniewicz-Żmuda, Anna, 30
Felder, Ferdinand, 83
Fellmann, Vincent, 50
Felten, Alexandre, 122
Feltin, Eric, 20, 38
Feneberg, Martin, 47
Fernandez, Sergio, 40
Fernández, Susana, 34
Ferroni, Matteo, 126
Fert, Albert, 98, 98, 109, 110
Figge, Stephan, 44, 46
Figueroa, Ignacio A., 156
Filipenkov, Viktor, 192, 210
Filippi, Matteo, 101
Filippov, Vladimir, 212, 213
Fill, Matthias, 83
Firstov, Sergey, 216
Firstov, Sergey A., 221
Fischer, Alexander, 99
Fittipaldi, Rosalba, 204
Fitzpatrick, Shane, 131
Fiyalka, Lyudmila, 229
Fleissner, Joachim, 78
Fleury, Eric, 146
Florian, Matthias, 44
Fochuk, Petro, 59
Fomin, Boris I., 88
Fomina, Larisa V., 56, 56, 104
Fong, Tiffany, 130
Forester, Erik, 134
Fraczkiewicz, Anna, 170
Franco, Nuno, 113
Franco, Victorino, 169, 179
Frank, Kristian, 44
Fried, Miklos, 129
Friedrich, Christian, 23
Fronc, Krzysztof, 173
Fuchs, Frank, 55
Fuentes, Antonio F., 209
Fujioka, Hiroshi, 72
Fuks-Janczarek, Izabela, 198
Fukushima, Tetsuya, 54
Fur, Cédric, 82
Fürjes, Peter, 124
Fusil, Stephane, 109, 110
Fusil, Stéphane, 98, 98
Fırat, Tezer, 112

G

Gabás, Mercedes, 67, 67
Gagliardi, Massimo, 224
Gągorowska, Barbara, 219
Gaj, Jan A., 44
Galanis, Nikos V., 159
Galeeva, Ja, 195
Galkin, Sergiy M., 45, 68
Gallardo, Eva, 23, 40
Gallinat, Chad S., 20
Galtier, Pierre, 66, 69, 74
Ganichev, Sergey D., 77
Ganière, Jean-daniel, 20
Gao, Yong, 203
Garbacz, Halina, 172, 172, 186
Garcia, Hermenegildo, 121
Garcia-Alonso, Diana, 198
Garcia-Casillas, Perla E., 132, 176, 213
Gartner, Mariuca, 44
Gasan-zade, Salim, 81
Gatteschi, Dante, 105
Gawron, Waldemar, 84
Gębara, Piotr, 178, 181
Gębicki, Wojciech, 220, 221
Gebicki, Wojtek, 70, 173
Gelb, Jeff, 130
Gentsar, Petro, 72
Georgaka, Aglaia D., 216
Georgakilas, Alexandros, 13, 28, 30, 47
Georgarakis, Konstantinos, 152
Gergely, Gréta, 133
Ghidini, Massimo, 107, 110
Ghijsen, Jacques, 102
Giapintzakis, Ioannis, 113
Giesen, Christoph, 35
Gizeli, Electra, 49
Gobsch, Gerhard, 16, 42, 52
Godlewski, Marek, 39, 48, 53, 63, 63, 65, 72
Goedel, Kathrin, 106
Gol'tsman, Gregory N., 75
Goldhahn, Ruediger, 16, 17, 28, 42, 47, 52
Golenkov, Alexsandr G., 93
Golka, Sebastien, 40
Goltvyanitsa, Sergey K., 221
Goltvyanitsa, Vladimir, 221
Goncharov, Aleksander, 197
Goncharuk, Dmitro, 193
Gondro, Joanna, 178, 182
Gonfa, Belete A., 60
González-Herráez, Miguel, 18, 34
Goodilin, Eugene A., 173, 174
Goranov, Vitaly, 105
Gorbyk, Petro P., 203
Gorczyca, Izabela, 24, 41
Górecka, Ewa, 208
Gorn, Dmitry I., 89
Gornaya, Iryna, 221
Gorokhova, Elena, 65
Goryachev, Yuriy M., 229
Gostomska, Marta, 217, 220
Gouma, Pelagia I., 119, 125, 144, 199
Grabias, Agnieszka, 157
Gradišek, Anton, 101
Grandal, Javier, 12, 16, 17, 23, 40
Grandjean, Nicolas, 20, 38
Granville, Simon, 115
Grausova, Lubica, 195
Graziosi, Patrizio, 98, 99, 110, 110, 111
Grebennik, Andrei, 65
Grigorieva, Anastasia V., 173
Grill, Roman, 59
Grimmeiss, Hermann G., 9
Grishko, Michael S., 189
Grobelny, Jaroslaw, 132
Grygoruk, Valerii I., 209
Grytsenko, Konstantin P., 226
Grzanka, Szymon, 49
Guari, Yannick, 199
Gudenko, Yuliya, 90
Guerbous, Lakhdar, 61
Guergouri, Kamel, 56, 60, 60
Guglya, Alexsey, 197
Guillemet, Raphael, 110
Guillet, Bruno, 12, 82
Gumenjuk-Sichevska, Janna, 91
Gür, Emre, 54
Gurau, Carmela, 169
Gurau, Gheorghe, 169
Gutowski, Jürgen, 46
Gutt, Richard, 50
Guziewicz, Elżbieta, 39, 48, 53, 63, 63, 65, 72
Gwo, Shangjr, 21

H

Haberl, Bianca, 158
Hadzic, Branka, 67, 68
Haensel, Thomas, 50
Hamdellou, Saliha, 60, 60
Han, Jun Hyun, 220
Hanèche, Nadia, 66, 69, 74
Hanser, Drew, 15
Harouni, Sofiane, 60, 60
Hashimoto, Akihiro, 19
Hassan, Sameh, 101
Haugan, Heather J., 80
Hauler, Felix, 144
Heljak, Marcin K., 123
Hernáez, Miguel, 122
Hernandez, Juan C., 167
Hernández Cocoletzi, Gregorio, 29
Hernandez Cocoletzi, Heriberto, 29
Heuer, Henning, 196
Heuken, Michael, 35
Hickey, Mark, 108
Hijazi, Abbas, 121
Himitliiska, Tsveta, 201
Himmerlich, Marcel, 22

Hlado, Volodymyr, 211
Hloba, Nastassia, 214, 215
Ho, Kelman, 115
Hoffmann, Axel, 48
Hoffmann, Veit, 23
Holland, Brendan, 23
Holy, Vaclav, 164
Hommel, Detlef, 44, 46
Horvath, Róbert, 124
Horváth, Zsolt E., 43
Hős, Csaba, 129
Hosseini, Seyyed Mohamad, 59
Hotra, Zenon, 48
Hourahine, Ben, 22
Hsu, Tung, 195
Hsu, Wen-Kuang, 197
Hu, Peiwei, 199
Hu, Qin, 154
Hua, Tzu-En, 192
Huang, Mengbing, 103
Huby, Nolwenn, 39
Hueso, Luis E., 98, 107
Hülber, Tímea, 129
Hunter, Luke, 130
Hwang, Fu-Kuo, 192
Hwu, Yeu-Kuang, 192

I

Iida, Daisuke, 41
Ilin, Elena A., 119
Iliopoulos, Eleftherios, 28
Illés, Levente, 133
Ilyuschenko, Alexandr F., 214
Inoue, Akihisa, 152
Inushima, Takashi, 20
Ion, Lucian, 59
Ipus, Jhon J., 169
Irbe, Zilgma, 192
Isaev, Mykola V., 206
Ishikawa, Takayuki, 116
Ishitani, Yoshihiro, 14, 22, 25
Islam, Md R., 19
Ivaldi, Francesco, 30, 31
Ivanchak, Svitlana A., 69
Ivanovskaya, Maria I., 213
Ivaschenko, Vladimir, 223
Ivers-Tiffée, Ellen, 135, 136
Iwankiewicz, Radosław, 162
Iwaya, Motoaki, 41
Izhnin, Ihor I., 87, 90

J

Jahn, Uwe, 40
Jahnke, Frank, 44
Jakieła, Rafał, 65
Jakubczyk, Tomasz, 44
Janczak-Rusch, Jolanta, 157
Jang, Bo Ra, 64, 64
Janik-Czachor, Maria, 173
Jarasiunas, Kestutis, 32
Jaroszewicz, Bohdan, 125
Jaroszewicz, Jakub, 123
Jarvis, Karalee, 58
Jarzębski, Maciej, 183
Jastrzebski, Cezariusz, 70, 173, 220, 221
Jee, Kwang K., 220
Jeon, Su Min, 57, 61, 62
Jezierska, Elżbieta, 180
Jiang, Xiuqian, 203
Jiang, Xunya, 31
Jo, Jungyol, 53
Jodhani, Gagan, 199
Jomard, François, 66
Jones, Rebecca E., 33
Jorge, Alexandre F., 210
Juan, Chien-Chang, 217
Judek, Jarosław, 173, 220, 221
Julien, François H, 40
Jung, Mi Na, 64, 64
Jung, Thomas, 106
Jung, Wojciech, 43
Jungwirth, Tomas, 103
Jurczyk, Karolina, 156, 184
Jurczyk, Mieczysław, 156, 183, 184, 184, 225
Jurczyk-Kowalska, Magdalena, 214
Justyniak, Iwona, 73

K

Kabaldin, Olexandr, 206
Kaczmar, Jacek, 218
Kadys, Arunas, 32
Kaiser, Jean-Pierre, 134
Kalden, Joachim, 46
Kalesaki, Efterpi, 52
Kallavus, Urve, 65
Kalska-Szostko, Beata, 128, 133
Kamilov, Ibrahimkhan K., 112, 113
Kamińska, Agata, 41
Kamiyama, Satoshi, 41
Kandaswamy, Prem K., 18, 40
Kaneko, Masamitsu, 54
Kang, Ting Ting, 19
Karakostas, Theodoros, 13, 62
Karbivskyy, Volodymyr L., 210
Karbownik, Piotr, 80
Karpierz, Krzysztof, 76
Karpuk, Michael M., 126
Kartuzov, Valeriy V., 194, 228
Kasperczyk, Jacek J., 198
Kasprzyk, Krzysztof, 205, 211
Kasprzyk, Zbigniew, 211
Kasyutich, Oksana, 188
Kaszewski, Jarosław, 175, 176
Kaszuwara, Waldemar, 154, 178, 179, 180
Kato, Kenta, 14
Kaufmann, Nils A K., 20
Kazin, Pavel E., 152

Kechouane, Mohamed, 42, 61, 62
Kedzierzawski, Piotr, 160
Keffous, Aissa, 61
Kehagias, Thomas, 13, 30, 52, 58
Kehrbusch, Jenny, 119
Kelires, Pantelis C., 159
Kendrick, Chito, 32, 34
Kerasiotis, Iordanis, 13, 30
Kerdja, Tahar, 61
Kesanakurthi, Sankar, 105
Khachatryan, Aik, 194
Khánh, Nguyen Q., 43
Khater, Antoine, 121
Khazen, Kh, 104
Kheireddine, Abdel Yazid, 62
Khiar, Amir, 83
Kholmska, Anna D., 188
Khoviv, Alexander M., 85
Kiisk, Valter, 174
Kikawa, Junjiroh, 54
Kikuchi, Akihiko, 15
Kilner, John A., 137
Kim, Chang Oh, 57
Kim, Do Hyang, 154
Kim, Dong Jik, 110
Kim, Do Yeob, 57, 61, 62
Kim, Ghun Sik, 57, 61, 62
Kim, Haemi, 53
Kim, Hong Seung, 64, 64
Kim, Jin Soo, 57, 61, 62
Kim, Jong Su, 57, 61, 62
Kim, Ki Buem, 154
Kim, Min Su, 57, 61, 62
Kim, Sung, 57
Kim, Tae Hoon, 61
Kim, Yoon-Bae, 146
Kim, Yu-Chan, 146
Kim-Chauveau, Hyonju, 48
Kimmel, Giora, 165
King, Phil D., 26, 32, 34, 42
Kioseoglou, Joseph, 13, 52, 62
Kiriakidis, George, 127
Kirilov, Kiril, 14, 16
Kirmse, Holm, 52
Kishino, Katsumi, 15
Kiss, Laszlo F., 179
Kityk, Ivan V., 198
Klar, Thomas A., 16
Kleismit, Richard, 198
Kleto, Gennadiy I., 115
Klimenko, Anatolii, 86, 88
Klinkenberg, Ernst-Dieter, 205
Klyshko, Alexei, 55
Knap, Wojciech, 76
Kneissl, Michael A., 23, 29
Knets, Ivars, 192, 210
Knoff, Wojciech, 103
Kobayashi, Atsushi, 72
Koblmueller, Gregor, 16, 20
Kobylińska, Urszula, 207
Koch, Roland J., 50
Kocherga, Lev, 189, 223
Kodolbaş, Alp O., 161
Kogut, Krzysztof, 205, 211
Köhler, Oliver, 121
Kokhanenko, Andrey P., 89
Kolobrodov, Victor, 197
Kolosyuk, Andriy, 206
Komarovskih, Nina V., 56
Komaschenko, Valeriy N., 43
Komninou, Philomela, 13, 30, 52, 58, 62
Kompany, Ahmad, 59
Kondrashova, Natalia, 192
Konig, Nataliia, 217
Konopka, Katarzyna, 202, 207
Konstantinidis, George, 49
Koopmans, Bert, 109
Kopalko, Krzysztof, 39, 53, 63, 72
Kopcewicz, Michal, 157
Kopidakis, George, 159
Kornilov, Victor M., 100
Korolev, Alexy M., 78
Korostynska, Olga, 118, 131, 190
Korshikova, Tatyana, 164
Kortidis, Ioannis, 127
Kosiel, Kamil, 80, 81
Kossakowska, Agnieszka, 126
Kostic, Radmila, 68
Kostiuk, Dmitriy A., 126
Kotko, Andriy V., 226, 226
Kotlarczyk, Urszula U., 181
Kotsikau, Dzmitry A., 213
Kovalchenko, Mikail, 207
Kovalyuk, Bogdan, 211
Kovarova, Anna, 208
Kowalczyk, Leszek, 103
Kowalczyk, Maciej, 157
Kowalski, Bogdan J., 39, 53, 65
Kozikowski, Pawel M., 171
Kozlowski, Gregory, 198
Kozma, Péter, 129
Kraeusel, Simon, 22
Kraitchinskii, Anatoliy, 206
Krajewska, Elżbieta, 73
Krajewski, Tomasz A., 39, 53, 63, 65
Kras'ko, Mykola, 206
Krasnov, Vladimir Y., 160
Krasnowski, Marek, 168, 169, 169
Krawczyńska, Agnieszka T., 170, 171
Kret, Slawomir, 30, 31
Krischok, Stefan P., 22, 50
Krishna, Sanjay, 79
Kromka, Alexander, 195
Krost, Alois, 16, 52
Krotkus, Arūnas, 78
Kruglenko, Ivanna, 72
Kruk, Adam, 167
Kruk, Jerzy, 126

Krukowski, Stanisław, 64
Kruse, Carsten, 44, 46
Krutko, Elvira, 195, 214, 215
Krysko, Marcin, 64
Książek, Marzanna, 183
Ku, Kap-Ryeol, 64
Kubacka-Traczyk, Justyna, 80
Kud, Irina V., 200
Kudelski, Andrzej, 173
Kudrawiec, Robert, 52, 63
Kuettner, Martin, 196
Kühn, Uta, 153
Kukuła, Iwona, 179
Kulczyk, Mariusz, 171
Kulik, Tadeusz, 155, 157, 168, 168, 169, 169, 178
Kulikov, Leonid, 217
Kumar, Bhupesh, 176
Kumar, Naresh, 18, 71
Kumar, Sumeet, 129
Kunert, Gerd, 46
Kunz, Friederike, 205
Kuo, Cheng-Tai, 21
Kuo, Yk, 114
Kurgan, Nataly A., 210
Kuribayashi, Kiyoshi, 184
Kurowski, Dariusz, 170
Kurunczi, Sándor, 129
Kuryliszyn-Kudelska, Izabela, 68
Kurzydlowski, Krzysztof J., 73, 123, 124, 132, 133, 134, 134, 163, 163, 163, 169, 170, 170, 171, 171, 171, 172, 172, 173, 186, 214, 221, 227
Kutrzeba, Milka, 39
Kuzavko, Yuri A., 126, 128
Kuziak, Roman M., 170
Kuzian, Roman, 70
Kuzmenko, Mykola M., 216
Kuzmich, Andrey G., 206
Kuznetsova, Olga, 224
Kłos, Krzysztof, 84

L

Lach, Stefan, 106, 106
Lachinov, Alexey A., 100
Lacroix, Olivier, 142
Lahmer, Mohamed ali, 56
Lahourcade, Lise, 18, 34, 52, 58
Lai, Chao-Sung, 125, 129, 131
Lai, Sheng-Feng, 192
Lakdja, Abdelaziz, 68, 113
Lakiza, Sergij M., 211
Lalaiants, Oleksandr I., 45, 68
Lamago, Daniel, 142
Landi, Elena, 105
Lange, Regina, 205
Langlois, Cyril T., 159, 166, 167
Langridge, Sean, 108
Laptyeva, Tetyana V., 207
Largeau, Ludovic, 104
Laroche, Jean Michel, 69
Latuch, Jerzy, 157, 157, 168, 179
Lau, Siew H., 130
Laumer, Bernhard, 42
Lazarevic, Zorica, 67
Lazreg, Abdelkader, 68, 113
Lebreton, Jean-Marie, 201
Lee, Hong-Mao, 21
Lee, Hsin-Yi, 212
Lee, Huang-Chia, 131
Lee, Jenn-Min, 115
Lee, Joo In, 57, 61, 62
Lee, Jusang, 144
Lee, Ju-Young, 64, 64
Lee, Kuang Che, 143
Lee, Sang Soo, 220
Lee, Soojin, 175
Lee, Won-Jae, 64, 64
Leem, Jae-Young, 57, 61, 62
Legendre, Fabrice, 201
Leghrib, Radouane, 122
Lei, Huaping, 31
Leinen, Dietmar, 67, 67
Lemaître, Aristide, 104
Lemmens, Jasper, 98
Leonov, Mikhail Y., 91
Leonowicz, Marcin, 182
Leonowicz, Marcin K., 179, 180
Lepadatu, Serban, 108
Lerose, Vincent, 120
Lesz, Sabina T., 180
Leszczyńska-Madej, Beata, 163, 183
Leszczynski, Mike, 31, 49, 51, 64
Levchenko, Ganna, 212
Levrat, Jacques, 38
Lewandowska, Malgorzata, 163, 170, 170, 171, 171, 171, 173
Lewandowska-Szumieł, Małgorzata, 124, 132
Lewiński, Janusz, 70, 73
Li, Fenghong, 107, 110
Li, Jia, 108
Li, Yan, 152
Li, Ziyou, 166
Lien, Der-Hsien, 195
Lilental-Weber, Zuzanna, 33
Limage, Marie-Helene, 142
Lin, Hong-Ming, 160
Lin, Su-Jien, 143, 196, 197
Lin, Wei-Syuan, 160
Lin, Yi-Tin, 125, 129
Lin, Yung-Jen, 191
Ling, Dah-Chin, 103
Linhart, Wojciech M., 32
Liou, Wei-Jen, 160
Lischka, Klaus, 47
Lisenko, Anatoly, 228
Lityńska-Dobrzyńska, Lidia, 156
Liu, Amelia C Y., 158
Liu, Chi-Jen, 192
Liu, Hui Chun, 76
Liu, Shuang, 82

Liu, Ting-Wei, 196
Liu, Xianjie, 107
Liu, Yen-Ting, 212
Llobet, Eduard, 122
Looney, Lisa, 198
Lopatik, Dmitri A., 71
Lopato, Lidiya M., 211
López, MariCruz, 67
Lorenz, Katharina, 50
Lorenz, Pierre, 50
Losytskyy, Mykhaylo, 226
Lotsari, Antiopi, 52, 58
Lott, Kalju, 65
Lourenço, Armando C., 67
Lozano-Perez, Sergio, 169
Lu, Hai, 20
Lu, Kueih-Tzu, 115
Lu, Titova, 214
Luchka, Miron V., 207
Lue, Cheng-En, 125, 129, 131
Lukács, István E., 43, 133
Lumey, Marck W., 140
Luna, Esperanza, 23, 40
Lupan, Oleg, 45
Lupo, Pierpaolo, 107
Lusakowska, Elżbieta, 72
Lusson, Alain, 66, 69, 74
Lutsyk, Vasily, 202
Lyashchenko, Alfred, 212, 213
Lykhodid, Lydmila, 200
Lysiuk, Ihor, 91
Łojkowski, Witold, 175, 176
Łuka, Grzegorz, 39, 48, 53, 63, 65
Łukasiewicz, Małgorzata, 53, 65
Łusakowska, Elżbieta, 103
Łusakowski, Jerzy, 76
Łuszczyn, Joanna, 133, 222
Łyszkowski, Radosław W., 170

M

Maaroos, Aarne, 174
Macchadani, Houssaine, 40
MacLaren, Donald, 99, 111
Madejczyk, Paweł, 84
Madrak, Karolina A., 218
Magalhães, Sérgio, 50
Mahajan, Subhash, 58
Maharramov, Abel M., 161
Mahesh, Karimbi K., 169
Maisonnat, André, 199
Majumdar, Himadri S., 108
Majumdar, Sayani, 108
Mak, Chee Leung, 73
Makara, Volodymyr A, 194, 194, 206
Makhniy, Victor P., 115
Mäki, Jussi-Matti, 33
Makkonen, Ilja, 33
Makoviy, Vitaliy V., 69
Malakhov, Vladislav Y., 229
Malinauskas, T., 32
Malyk, Orest P., 66
Malyutenko, Vladimir K., 88
Mamedov, Huseyn M., 55
Mamedov, Vusal U., 55
Manciulea, Ileana, 225
Mandal, Arjun, 50
Manea, Elena, 87
Mangeney, Juliette, 40
Mannini, Matteo, 105
Manseri, Amar, 62
Mansuri, Irfan, 114
Mantlikova, Alice, 164
Marcacci, Maurilio, 105
Marcelli, Augusto, 84
Marchese, Leonardo, 129
Marchetto, Helder, 108
Marchishin, Igor V., 88, 88
Marcins, Guntis, 66
Marenkin, Sergey F., 112, 113
Margheriti, Ludovica, 105
Markus, Torsten, 145
Marona, Łucja, 49
Marrows, Christopher, 108
Martel Estrada, Adriana, 213
Martin, Denis, 20
Martín, Francisco, 67, 67
Martin, Robert W., 18
Martin, Sara, 12
Martínez, Carlos A., 132, 176, 213
Martinez, Carlos J., 119
Martinez, Lourdes, 67
Martini, Aurela, 125
Martín-López, Sonia, 18
Martino, Guglielmo, 125
Marzouki, Ali, 66
Maslova, Olga A., 196
Masyukov, Nikita, 32
Matei, Elena, 59, 115
Matías, Ignacio R., 122
Matsubara, Tetsuya, 41
Matsura, Victor, 199
Mattana, Richard, 98, 98, 109, 110
Mattern, Norbert, 154
Matulionis, A., 27
Matusiewicz, Marek, 198
Matveevskaya, Neonila A., 164
Mauguin, Olivia, 104
Mazalski, Piotr, 128
Maziarz, Wojciech, 156
Mazurkiewicz, Marta, 221
Małolepszy, Artur, 221
McBean, Katie E., 38, 51
McConville, Chris F., 20, 26, 32, 34, 42
McCredie, Geoffrey M., 38
Mechin, Laurence, 12, 82
Meier, Douglas C., 119
Melnik, Iryna, 215
Menari, Hamid, 42, 61, 62

Menzler, Norbert H., 143
Mertelj, Tomaž, 101
Meulenberg, Wilhelm A., 136, 140, 144, 148
Mezmale, Liga, 192
Miard, Audrey, 104
Michalski, Andrzej, 177
Michalski, Bartosz, 154, 179, 180
Miedziński, Rafał, 198
Mihailovic, Dragan, 101
Mihály, Judith, 133
Mikhailov, N. N., 86, 88
Miklaszewski, Andrzej, 184
Milanesio, Marco, 129
Millan, Maria, 158
Milman, Yuly V., 203
Minàr, Jan, 116
Misiewicz, Jan, 63
Misiuk, Andrzej, 103
Mitchel, William C., 80
Mizerski, Tadeusz, 165, 191, 199
Moczulska, Maryla, 134
Modigell, Michael, 138, 148
Moebius, Annett, 145
Mohanta, Antaryami, 168
Molak, Rafal, 171
Molinari, Anna, 98
Mollaev, Ahmedbek Y., 112
Mollaev, Akhmedbek Y., 113
Momot, Natalia I., 91
Monroy, Eva, 14, 18, 34, 40, 52, 58
Monteiro, Teresa, 43, 50, 67
Montes-Navajas, Pedro, 121
Morales, Magali, 16
Moreno, Rodrigo, 190, 209
Moret, Matthieu, 17
Morhain, Christian, 48
Morkoc, Hadis, 27
Morpurgo, Alberto, 98
Morris, Deirdre, 190
Moschovis, Kostas, 127
Moskal, Grzegorz J., 146, 146, 147, 147, 147, 148
Moszczyński, Piotr, 183
Moszyński, Dariusz, 176
Moustakas, Theodore, 32
Mücke, Robert, 143
Mugumaoderha Cubaka, Mac, 102
Müller, Kathrin, 106
Müller, Knut, 46
Müller, René, 16
Muńoz-Sanjosé, Vicente, 74
Muradov, Mustafa B., 161, 185
Murai, Yuuta, 184
Murakami, Hideyuki, 172
Mycielski, Andrzej, 67
Mydlarz, Tadeusz T., 181
Mynbaev, Karim D., 90

N

Naber, Wouter, 98
Nabiałek, Marcin G., 177, 177, 178, 179, 180, 181, 182, 219
Naboka, Olga V., 209
Nagy, Norbert, 124
Naidich, Yury V., 213
Nakazawa, Masayuki, 184
Nanishi, Yasushi, 19, 46, 54
Napara-Volgina, Svietlana G., 193
Naplocha, Krzysztof, 218
Naranjo, Fernando B., 18, 34
Nargelas, Saulius, 32
Narkiewicz, Urszula, 68, 175, 176
Nebe, Barbara J., 205
Nechaev, Kirill A., 130
Nedelko, Mikhail I., 71
Neimash, Vladimdir, 206
Nejman, Ilona H., 183
Németh, Andrea, 129
Nesmelov, Sergey N., 89, 93, 94
Neumann, Maciej, 42
Neumann, Wolfgang, 52
Neuschl, Benjamin, 47
Neuschwander, Sabine, 99, 116
Nevar, Alena A., 71
Neves, A. J., 43, 50
Nevou, Laurent, 40
Newby, Carol, 110, 110
Ng, Chi Chung, 218
Nguyen, Tho, 109
Ni, Krutko, 214
Niedrig, Christian, 136
Niespodziana, Katarzyna, 184
Nikiforov, Yurij, 211
Nikitenko, Yuriy, 197
Nirk, Tiit, 65
Nischenko, Mihajlo, 211
Nogueira Vieira, Milena M., 143
Norby, Truls E., 140
Nouet, Gerard, 13
Nouneh, Khalid, 198
Novo, Luis F., 221
Novotna, Katarina, 195
Novotorzev, Vladimir M., 112, 113
Nowak, Marek, 225
Nowakowska, Anna, 127
Nowosielski, Ryszard, 180
Nurkenova, Barchagul' D., 201, 219

O

O'Donnell, Kevin P., 18
O'Sullivan, Martin, 190
Oesterschulze, Egbert, 119
Ofe, Stefan, 205
Ogenko, Volodymyr M., 209
Ogiwara, Hitoshi, 14
Ohta, Jitsuo, 72
Okońska, Izabela, 183, 184
Oksiuta, Zbigniew, 163
Öktü, Özcan, 161
Okun, Igor, 216

Okuno-Vila, Hanako, 50
Olejnik, Lech, 163
Oleszak, Dariusz, 155, 157, 178
Oliva-Stasiak, Kamila, 118
Oliveira, João C., 143
Oliver, Derek R., 94
Olszewski, Jacek, 178, 180, 182
Olszewski, Wojciech, 128
Öpik, Andres, 65
Oppeneer, Peter M., 106, 106
Osewski, Paweł, 218
Osmakov, Andrey, 189, 223
Ostash, Orest, 227
Osterbacka, R, 108
Ostrowski, Andrzej, 70, 173
Otake, Hirotake, 54
Oueslati, Meherzi, 66
Oyama, Munetaka, 198
Özcan, Şadan, 112
Ozga, Julita, 198
Ozgur, Umit, 27

P

Pačebutas, Vaidas, 78
Pachla, Waclaw, 172
Pacuski, Wojciech, 44
Paderno, Varvara N., 213
Pakhomov, Georgiy, 48
Pakiela, Zbigniew, 171
Pakieła, Zbigniew, 163
Palanco, Santiago, 67, 67
Palin, Luca, 129
Palkar, Amit, 133, 222
Panchuk, Oleg, 59
Pankov, Vladimir V., 213
Pantazis, Alexandros K., 49
Paramés, Luisa, 113
Parbrook, Peter J., 18, 71
Parco, Maria, 198
Pareige, Philippe, 201
Park, Jae Gwan, 175
Partridge, Ashton, 203
Paskova, Tanya, 15
Paszkowicz, Wojciech, 39, 65, 72
Patrusheva, Tamara N., 114
Paul, Geo, 129
Pauly, Simon, 154
Pawlik, Katarzyna, 154, 178, 181
Pawlik, Piotr, 154, 178, 180, 181
Peinemann, Klaus V., 148
Pękała, Marek, 157
Pereira, Sergio, 67
Peres, Marco, 67
Peres, Marco A., 43, 50
Perez, Ramiro, 175
Perlin, Piotr, 49, 64
Pernechele, Chiara, 107
Perrone, Alessio, 69
Petranovskaya, Alla L., 203
Petrenko, Taras L., 92
Petrik, Peter, 129
Petroff, Frédéric, 98, 98, 109, 110
Petrovsky, Vitaly, 228
Petrow, Alex L., 208
Petrunya, Ruslan, 206
Pezoldt, J., 16
Pfaff, Ewald M., 136, 138
Philippot, Karine, 199
Phillips, Matthew R., 38, 51
Picinini, Maximo, 84
Pierscinska, Dorota, 81
Pierscinski, Kamil, 81
Pierściński, Kamil, 80
Pijanowska, Dorota G., 125, 126
Pinchuk, Anatoliy, 226
Pineider, Francesco, 105
Piotrowski, Adam, 84
Piotrowski, Jozef, 83
Piotrowski, Tadeusz, 88
Pireaux, Jean-Jacques, 122
Piryatinski, Yuriy, 226
Pisarek, Marcin, 173
Plyatsko, Sergiy V., 92
Pociask, Malgorzata M., 85, 90
Pociecha, Damian, 208
Podrezov, Yuriy M., 207
Podsiadło, Sławomir, 70, 173
Poisson, Marie A., 35
Polakowski, Henryk, 88
Poletaev, Gennadiy M., 160
Poliakov, Peter, 115
Polit, Jacek, 84
Polovets, Svitlana E., 213
Poltierova-Vejpravova, Jana, 164
Pong, Way-Faung, 103
Pongrácz, Anita, 43
Ponomarenko, Gennady, 223
Ponzoni, Andrea, 126
Poroshin, Volodymyr, 90
Potenza, Alessandro, 108
Potzger, Kay, 105
Pozzovivo, Gianmauro, 40
Pratontep, Sirapat, 54
Preble, Edward E., 15
Predein, Aleksandr, 86, 88
Prellier, Wilfrid, 101
Prezioso, Mirko, 99, 110, 110, 111
Prikhod'ko, Genadij, 211
Prischepa, Sergei, 204
Pristovsek, Markus, 29
Prochowicz, Daniel, 70
Prokopets, Vadym M., 226
Pruszyńska-Karbownik, Emilia, 80
Prystawko, Paweł, 64
Przezdziecka, Ewa, 39, 53, 63
Pud, A., 226
Purica, Munizer, 87, 222
Puska, Martti J., 33

Pysarevskii, Volodymyr K., 93
Płońska-Brzezińska, Marta E., 133, 222
Płusa, Danuta D., 181

Q

Qi, Yun, 102

R

Radchenko, Andrey V., 189
Radchenko, Pavel A., 189
Radiguet, Bertrand, 201
Radzio, Bernhard, 119
Raethel, Jochen, 16
Rahim, Mohamed, 83
Rakitin, Roman Y., 201, 219
Raman, Baranidharan, 119
Ramos, Sergio L., 124
Ramos-Barrado, Jose R., 67, 67
Rasiński, Marcin, 163
Rassolov, Sergey G., 179
Rauch, Christian, 20
Reedo, Valter, 174
Reeves, Roger J., 34
Rehm, Robert, 78
Reichert, Peter, 39
Remediakis, Ioannis N., 159
Rendina, Ivo, 224
Restrepo, Ricardo L., 59
Reurings, Floris, 20, 33
Reva, Vladimir, 86
Rezanova, Natalya, 215
Ribeiro, Ricardo M., 185
Richert, Maria, 163, 183
Ricolleau, Christian, 159, 166, 167
Ricote, Sandrine, 141
Riegert, Dorota, 207
Riminucci, Alberto, 99, 105, 110, 110, 111
Rodak, Kinga B., 171
Rödl, Claudia, 55
Rodriguez, Evelyn, 209
Rodriguez-Viejo, Javier, 159
Roeppischer, Marcus, 47, 52
Roeser, Hans-Peter, 82
Rogal, Łukasz, 156
Rogalski, Antoni, 84, 84
Rogers, David J., 43
Roguska, Agata, 173
Romcevic, Maja, 67, 68
Romcevic, Nebojsa, 67, 68
Romero, Rocio, 67
Rosenauer, Andreas, 44, 46
Roshchupkina, Olga, 105
Rosinski, Marcin, 177
Rossbach, Georg, 47, 52
Roth, Stefan, 153
Routoure, Jean-Marc, 12, 82
Rozale, Habib, 68, 113
Rozhenko, Natalia N., 228
Rozmysłowska, Aleksandra, 147, 147, 148
Rud, Boris, 229
Ruffell, Simon, 158
Rumyantsev, Vladimir, 189, 223
Rupeks, Lauris, 192, 210
Russo, Alessandro, 105
Ruterana, Pierre, 12, 14, 16, 17, 31, 35, 68, 113
Rutkowski, Jaroslaw, 84
Rutz, Frank, 78
Rybalka, Iryna A., 45, 68
Ryszkowska, Joanna, 124, 132, 214, 227
Ryumshyna, Tatyana A., 115
Ryzhikov, Vladimir D., 45
Ryzhikov, Volodymyr D., 68

S

Sabinina, Irina, 86, 88
Saenko, Ekaterina, 192
Sáfrán, György, 43
Sainctavit, Philippe, 105
Sakalauskas, Egidijus, 16, 17
Sakka, Yoshio, 165, 191
Sakowicz, Maciej, 76
Sala, Beatrice, 142, 142
Salamończyk, Mirosław, 208
Sallet, Vincent, 66, 69, 74
Samoylov, Alexander M., 85, 92
Sanchez, Miguel A., 40
Sanchez-Garcia, Miguel A., 16, 17, 23
Sánchez-García, Miguel Ángel, 12
Sandana, Vinod E., 43
Sandra, Ruffenach, 17
Sanvito, Stefano, 105
Sapronov, Roman, 189, 223
Sarazin, Nicolas, 12
Sartel, Corinne, 66, 69, 74
Sauther, Jens, 106
Savchuk, Andriy I., 69, 115
Savchuk, Serhiy A., 115
Savoyant, Adrien, 70
Sawicki, Maciej, 65
Sayede, Adlane, 148
Sberveglieri, Giorgio, 126
Schacham, Shmuel, 40
Schaefer, Juergen A., 22, 50
Schaff, William J., 20, 33
Scheibner, Ralf, 78
Schenk, David P., 41
Scherb, Tobias, 140
Schleife, André, 55
Schley, Pascal, 16, 17
Schlom, Darrell, 82
Schmitt, Felix, 106, 106
Schmitz, Johannes, 78
Schowalter, Marco, 44, 46
Schrader, Sigurd, 226
Schroeder, Michael, 137, 138
Schulze, Martin, 196
Schulze Dieckhoff, Hubert, 203

Schumacher, Gerhard, 140
Schupp, Thorsten, 47
Schwarzacher, Walther, 188
Scudino, Sergio, 153
Sebald, Kathrin, 46
Séguy, Isabelle, 100
Seitz, Hermann, 205
Sekiguchi, Hiroto, 15
Semancik, Steve, 119
Semikina, Tetyana V., 43, 72
Seneor, Pierre, 98, 98, 109, 110
Sengupta, Saumya, 81
Seo, Ogweon, 53
Seok, Hyun Kwang, 220
Sergienko, Zoya P., 155, 164
Serra, Jose M., 140, 145
Servet, Bernard, 98
Sessoli, Roberta, 105
Shalimov, Artem, 105
Sharma, Renu, 58
Sharov, Mikhail K., 85, 92
Shauchonak, Alexandr A., 214
Shaula, Alexander L., 143
Shavrov, Vladimir G., 126, 207
Shen, Bo, 22
Shepelskii, Georgiy, 81
Sherbakov, Vladimir I., 193, 208
Sheregii, Eugene M., 84
Shevchenko, Mykola, 211
Shin, Dong Hee, 57
Shinkarenko, Svetlana, 65
Shishkovsky, Igor V., 153, 193, 208
Shitsevalova, Natalia, 212
Shkurinov, Alexander, 77
Shon, Yoon, 57
Shpak, Anatoly P., 210
Shubina, Tatiana V., 15
Shulga, Sergey A., 91
Shulga, Valerii V., 78
Siad, Menouar, 42, 62
Sibera, Daniel, 68, 176
Sidorenko, Andrey A., 107
Sidorov, Yu. G., 86, 88
Siekacz, Marcin, 30
Siemiaszko, Dariusz, 177
Sildos, Ilmo, 174
Silenko, Petr M., 228
Silva, Rui J C., 169
Silvestre, António J., 113
Siman, Nicolay, 229
Simionescu, Agneta, 133
Singheiser, Lorenz, 148
Siomionescu, Dan T., 133
Sizov, Fedir F., 86, 92, 93
Skierbiszewski, Czeslaw, 30
Skorokhod, Valeriy V., 187
Slinchenko, Yurii A., 209
Slodczyk, Aneta, 141, 142, 142
Ślusarek, Barbara, 181
Smalc-Koziorowska, Julita, 62
Smertenko, Petro, 226
Smirnova, Natalia A., 90
Smoleń, Dariusz R., 70, 173
Smolik, Jerzy A., 186
Smolik, Waldemar T., 134
Smurov, Igor, 193
Soares, Manuel J., 43
Sobczak, Kamil, 31
Sobczyk, Katarzyna, 178, 182
Sokolovskii, Bogdan S., 93
Solzi, Massimo, 107
Son, Jeong-Sik, 61
Song, Young-Wook, 34
Sosnovsky, Igor A., 208
Sołoducho, Jadwiga, Maria, 127
Spanos, Nikos, 216
Spassov, Tony, 201
Spath, Sebastian, 205
Speck, James S., 16, 20, 26
Śpiewak, Piotr, 73
Sporken, Robert, 102
Spychalski, Maciej M., 173, 207
Spyra, Marzena, 182
Stach, Sebastian M., 218, 219
Stakhira, Pavlo, 48
Starokadomsky, Dmitry L., 215
Starostenkov, Mikhail D., 160, 201, 219
Staszczak, Grzegorz, 41
Stepanov, Anatoli, 70
Stobinski, Leszek, 160, 220, 221
Stoever, Detlev, 136, 143, 148
Stoica, Mihai, 44, 153
Stojanovic, Dusanka, 67
Stokes, Joseph, 198
Story, Tomasz, 103
Strasser, Gottfried, 40
Strelnikov, Vladimir N., 192
Strikha, Maxym, 81
Strupinski, Wlodek, 173
Stølen, Svein, 140
Suchocki, Andrzej, 41
Suescun, Leopoldo, 139
Sukhorukova, Olga S., 207
Sun, Haixia, 185
Surreddi, Kumar Babu, 153
Suski, Tadeusz, 24, 41, 49, 64
Suslyakov, Aleksandr, 86
Suvorov, Stanislav, 189
Svoboda, Jiri, 208
Svyrydova, Kateryna, 179
Sweeney, Francis, 18, 71
Świątek, Krzysztof, 103
Świerczek, Jan, 178, 182
Swierczek, Konrad, 139
Swieszkowski, Wojciech, 123, 133, 134, 134, 221
Świst, Agnieszka, 127
Sydorenko, Sergij I., 188
Sydorenko, Tetiana, 213

Sydorov, Dmytro, 226
Syrkin, Alexander L., 14
Syrotyuk, Stepan V., 70
Szafran, Mikolaj, 165, 191, 199
Szafran, Mikołaj, 202, 224
Szala, Janusz, 206
Szczepański, Tomasz, 111
Szczytko, Jacek, 218
Szerling, Anna, 80, 81
Szmulowicz, Frank, 80
Szota, Michał, 177, 177, 219
Szymański, Krzysztof, 128
Słota, Damian, 206
Słowiński, Grzegorz, 221

T

Taibi, Kamel, 42, 62
Takeuchi, Noboru, 29
Tale, Ivars, 66
Taliashvili, Badri, 103
Tallarida, Grazia, 39
Tampieri, Anna, 105
Tarasenko, Nikolai V., 71
Tarasenko, Sergey V., 207
Tarasov, Alexey, 174
Targowski, Grzegorz, 64
Tavolaro, Adalgisa, 125
Tavolaro, Palmira, 125
Tchernycheva, Maria, 40
Teherani, Ferechteh, 43
Teisseire, Monique, 48
Tengen, Thomas B., 162
Teppe, Frederic, 76
Terrazas Reza, Raul, 132
Thareja, Raj, 168, 176
Thiess, Sebastian, 102
Thomson, Douglas J., 94
Thonke, Klaus, 47
Tiainen, Veli M., 133
Timmons, Ana B., 60
Tkatch, Viktor, 179
Todd, Iain, 156
Todorova, Stanislava, 201
Tolmachev, Alexander, 164
Tolmachev, Alexander V., 155, 164
Tomaszewska, Emilia, 132
Tomaszewski, Wacław, 134
Ton-That, Cuong, 38
Topolski, Krzysztof, 172
Torbicz, Władysław, 126
Torrens, Francisco, 174
Torrens-Serra, Joan, 159
Torres, Vítor J B., 185
Tóth, Attila L., 133
Trager-Cowan, Carol, 18, 71
Trajic, Jelena, 67
Trampert, Achim, 23, 40
Travers, Laurent, 104
Tretyakov, Yuri D., 173, 174
Trindade, Bruno, 143
Tristany, Mar, 199
Trofim, Viorel, 45
Trusov, Lev A., 152
Trzaska, Maria, 183, 217, 220
Tsagaraki, Katerina, 28, 47
Tsebrenko, Iryna, 215
Tsebrenko, Marya, 215
Tsiakatouras, George, 28, 47
Tsmots, Volodymyr M., 206
Tsong, Tien-Tzou, 195
Tsybriy, Zenoviya F., 93
Tuliński, Maciej, 156
Tulupenko, Viktor, 90
Tumcharern, Gamolwan, 54
Tuomisto, Filip, 20, 33, 33
Turelyk, Margarita P., 203
Türker, Muhammed, 39
Türn, Leo, 65
Turski, Henryk, 218
Tüzemen, Sebahattin, 54
Tyschenko, Yana, 211

U

Ueno, Kohei, 72
Uhlenbruck, Sven, 143
Ullrich, Bruno, 80
Ulyanova, Tatyana M., 214
Ura-Bińczyk, Ewa, 171
Usov, Denys G., 203
Uvarova, Iryna V., 200

V

Vacik, Jiri, 195
Vainberg, Viktor, 90
Vajpeyi, Agam P., 30
Valamanesh, Mehrnoush, 167
Valcheva, Evgenia, 14, 16
Valdueza-Felip, Sirona, 18, 34
Vales, Vaclav, 164
Valtsifer, Viktor A., 192
Valuyskaya, Kateryna, 221
Van den berg, Albert, 118
Van der Wiel, Wilfred G., 98
Van de Walle, Chris G., 25
Vandrovcova, Marta, 195
Vanfleet, Richard, 103
Van Gestel, Tim, 144
Vanhaverbeke, Antoine, 108
Van Lierop, Johan, 188
Van Oost, Frank W., 109
Van wees, Bart, 97
Varavin, V. S., 86, 88
Varavin, Vasiliy, 91
Vardi, Alon, 40
Varga, Lajos K., 166
Varshney, Dinesh, 114
Vaseashta, Ashok, 118

Vasilenko, Ruslan, 197
Vasilevskiy, Mikhail I., 185
Vasiliev, Oleg, 224
Vasiliev, Vladimir V., 86, 88
Vasyliv, Bogdan, 227
Vasylyev, Oleksandr, 216, 227
Vaughan, Gavin, 153
Vazhenin, Stanislav V., 144
Veal, Tim, 20, 26, 32, 34, 42
Vecchione, Antonio, 204
Velusamy, Vijayalakshmi, 118
Vengris, Mikas, 32
Vert Belenguer, Vicente B., 145
Verzhak, Yevheniya, 59
Videlier, Hadley, 76
Vilalta Clemente, Arantxa, 12, 16, 17, 35
Villeneuve, Christina, 100
Vinter, Borge, 48
Virt, Ihor S., 93
Vishnjakov, Anatoli, 65
Viskadourakis, Zacharias, 113
Vogt, Patrick, 23
Voigt, Ingolf, 148
Voinea, Mihaela, 209
Voitsekhovskii, Alexander V., 89, 91, 93, 94
Volfman, Vladimir, 193
Volk, János, 43, 124
Volkovynska, Liudmyla S., 209
Voloshko, Svitlana M., 188
Volynyuk, Dmytro, 48
Von Bardeleben, Hans Jurgen, 104
Vonderviszt, Ferenc, 129
Vorobjeva, Natalia V., 100
Vorobjeva, Vera, 202
Voronkin, Yevgen F., 45, 68
Voros, Janos, 120
Vovk, Elena A., 155, 164
Vuichyk, Mykola, 72
Vyshniakov, Leon, 223, 228
Vyshnyakova, Kateryna, 223

W

Wachnicki, Łukasz, 39, 53, 63, 63, 72
Wade, Travis L., 120
Wagemans, Wiebe, 109
Wagner, Stefan F., 136
Wajs, Edward, 207, 220
Walther, Martin, 78
Walukiewicz, Wladek, 11, 33
Wang, Cheng-Liang, 192
Wang, Gang, 153
Wang, Shau-Chieh, 195
Wang, Szu-Chieh, 129, 131
Wang, Tao, 18, 71
Wang, Xinqiang, 22, 25
Wang, Yi-Shun, 131
Warot-Fonrose, Bénédicte, 100
Wassner, Thomas A., 42
Waterland, Mark, 203
Watkins, Ken G., 224
Watson, Ian M., 18, 71
Wawrzyniak, Malgorzata, 112
Wéber, Ferenc, 133
Węgrzecki, Maciej, 88
Weißmann, Volker, 205
Werner, Christoph, 52
Wessely, Ola, 108
Wetzel, Christian, 13, 15
Wexler, D, 155
Wichert, Thomas, 39
Wieciński, Piotr, 133, 172, 186
Wieneke, Matthias, 16, 52
Wilkinson, Angus J., 18, 71
Willemin, Stephanie, 142, 142
Williams, Craig J., 224
Williams, Jim S., 158
Winkler, Krzysztof, 222
Wirthmüller, Alexander, 40
Witkowski, Bartłomiej, 39, 53, 63, 65
Wohlgenannt, Markus, 109
Wojtowicz, Tomasz, 44
Wolf, Herbert, 39
Wong, Ching Hong, 73
Wong, Johnny, 101
Wong, Kin Hung, 73
Woźniak, Piotr, 124, 132
Wróbel, Jan, 73
Wróbel, Jerzy, 103, 173
Wu, Chung-Lin, 21
Wu, Chun-Lin, 197
Wu, Jing, 101
Wu, Jyun-Yi, 197
Wu, She-huang, 160
Wu, She-Huang, 160
Wu, Sheng, 82
Wüstenberg, Jan-Peter, 99, 116
Wysłocki, Jerzy J., 154, 178, 181
Włodarczyk, Renata K., 220

X

Xavier, Stéphane, 98
Xu, Yongbing, 101
Xue, Ruipeng, 125, 144

Y

Yadroitsev, Igor, 193
Yahyaie, Iman, 94
Yakovyna, Vitalij S., 86
Yamaguchi, Atsushi, 54
Yamaguchi, Tomohiro, 19, 46
Yamamoto, Akio, 19
Yamamoto, Masafumi, 116
Yang, Chia-Ming, 125, 129, 131
Yang, Huaming, 199
Yang, Weishen, 136
Yarmatov, Ismat, 207
Yaroshenko, Mykola, 43

Yaroshinskaya, Natalia, 152
Yates, Karen A., 113
Yatsenko, Oleg B., 92
Yavari, Alain R., 152, 153
Yavetskiy, Roman P., 155, 164
Yeh, Jien-Wei, 196, 217
Yeremenko, Lydmila I., 200
Yermolayeva, Yuliya V., 164
Yeshchenko, Oleg, 226, 226
Yi, Jianxin, 138
Yoon, Jong-Gul, 39
Yoshida, Tsukasa, 226
Yoshikawa, Akihiko, 14, 22, 25
You, Shi, 15
Yu, Kin M., 33
Yuan, Jun, 166
Yudenkov, V. A., 87
Yun, Junho, 53
Yun, Wenbing, 130
Yusupov, Roman, 101

Z

Zaafrani, Oumaya, 141
Zabicky, Jacob, 165
Zabudsky, Vyacheslav V., 91
Zalibekov, Ullubii Z., 112, 113
Zamarreño, Carlos R., 122
Zanolli, Zeila, 122
Zapp, Petra, 148
Zavaglia, Cecilia C., 124
Zawadzak, Ewelina A., 221, 227
Zayats, Mykola, 72
Zboromirska-Wnukiewicz, Beata, 205, 211
Zbroszczyk, Józef, 178, 182
Zbrzeźniak, Elżbieta, 178
Zelenaya, Anna, 202
Zemtsova, Yuliya V., 104
Zhan, Yiqiang, 107, 110
Zhang, Lin, 152, 185
Zhang, Qi, 22
Zharskaya, Ta, 195
Zhdanuk, Elena, 195, 215
Zhikharev, Igor, 179
Zhou, Shengqiang, 105
Zhu, Mingwei, 15
Zhukovskiy, Mark S., 104, 144, 189, 196
Ziatkevich, Dmitro, 200
Ziegler, Christiane, 106, 106, 121
Ziegler, Johann, 78
Zlatanova, Zlatina, 201
Zogg, Hans, 83
Zwick, Markus, 136, 138
Zyśk, Beata A., 171