

## NETZSCH Analyzing & Testing – Advanced Materials Characterization by Thermal Analysis Techniques



Dr. Hilary Smogór

NETZSCH Analyzing & Testing

# The NETZSCH Group and its Globally Acting Business Units

**NETZSCH**

## Erich NETZSCH GmbH & Co. Holding KG

### Analyzing & Testing

The product range comprises instruments for thermal analysis and for determination of thermophysical properties.



### Grinding & Dispersing

Comprehensive product line for a multitude of tasks in wet and dry grinding, mixing, de-aeration and classifying and for the most different industrial applications.



### Pumps & Systems

Comprehensive range of pumps for industrial conveying tasks – manufacturer of the worldwide known NEMO® eccentric pumps



- Foundation:
- Turnover:
- Staff:
- Locations:

1873 by Thomas and Christian Netzscher in Selb  
about € 453 million  
> 3000 worldwide, around 1350 of them in Germany  
163 worldwide in 28 countries

# NETZSCH Analysing & Testing Acts Globally

**NETZSCH**

- 49 Sales and service locations in 37 countries
- Production facilities in Selb - Germany, and Boston - USA, Osaka - Japan



- **Definition (ICTAC):**

Thermal analysis (TA) is a group of techniques in which changes of **physical or chemical properties** of the sample are monitored against **time or temperature**, while the temperature of the sample is programmed.

The temperature program may involve heating or cooling at a fixed rate, holding the temperature constant (isothermal), or any sequence of these.

# The NETZSCH Product Range – Methods

**NETZSCH**

## Materials Characterization by Thermal Analysis Methods

Differential Thermal Analysis, Differential Scanning Calorimetry (DTA/DSC)

Thermogravimetry (TGA), Simultaneous Thermal Analysis (STA=TGA+DSC)

Thermomechanical Analysis (DIL, TMA, DMA, RUL, HMOR)

Thermophysical Properties Measurements (TCT, HFM, GHP, LFA)

Adiabatic Calorimetry (ARC, MMC, APTAC, IBC)

Dielectric Analysis (DEA)

Phase transition temperatures, enthalpy changes, specific heat

-180 ... 2400°C

Mass changes due to evaporation, decomposition and interaction with the atmosphere

-150 ... 2400°C

Dim. changes, deformations, viscoelastic properties, transitions, density

-260 ... 2800°C

Thermal diffusivity, thermal conductivity, transport properties

-125 ... 2800°C

Phase transitions, isothermal/scanning calorimetry, thermal stability, reaction behavior, battery tester

RT ... 500°C

Ion viscosity, curing behavior, dielectric properties

RT ... 375°C

Evolved Gas Analysis (EGA) for the advanced characterization of decomposition/evaporation effects

# The NETZSCH Product Range – Optimized Instruments with Outstanding Features

**NETZSCH**

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(ARC, MMC,  
APTAC, battery tester)

Dielectric Analysis  
(DEA)

DSC 3500 *Sirius*  
DSC 214 *Polyma*  
**DSC 204 F1**  
DSC 204 F1 Photo  
DSC 204 HP  
DSC 404 F1  
DSC 404 F3  
-180 ... 2400°C

TG 209 **F3**  
TG 209 **F1**  
STA 449 **F3**  
STA 449 **F1**  
STA 449 **F5**  
STA 2500 *Regulus*  
STA 409 CD  
STA 429 CD  
-150 ... 2400°C

DIL 402 PC  
DIL 402 C  
DIL 402 CD  
DIL 402 E/Pyro  
TMA 402  
DMA 242 E  
-260 ... 2800°C

HFM 436  
GHP 456  
TCT 426  
LFA 467  
LFA 457  
LFA 427  
SBA 458 *Nemesis*  
-125 ... 2800°C

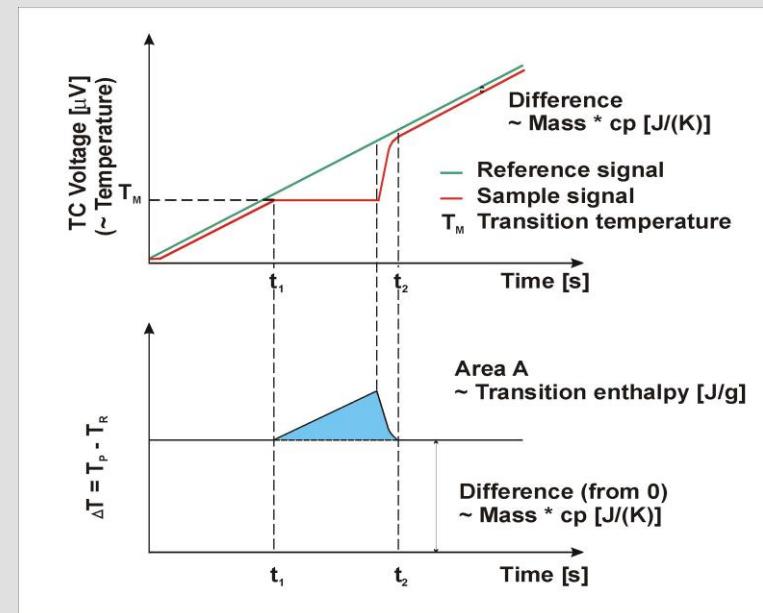
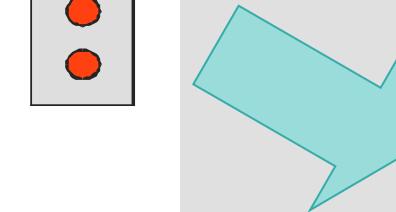
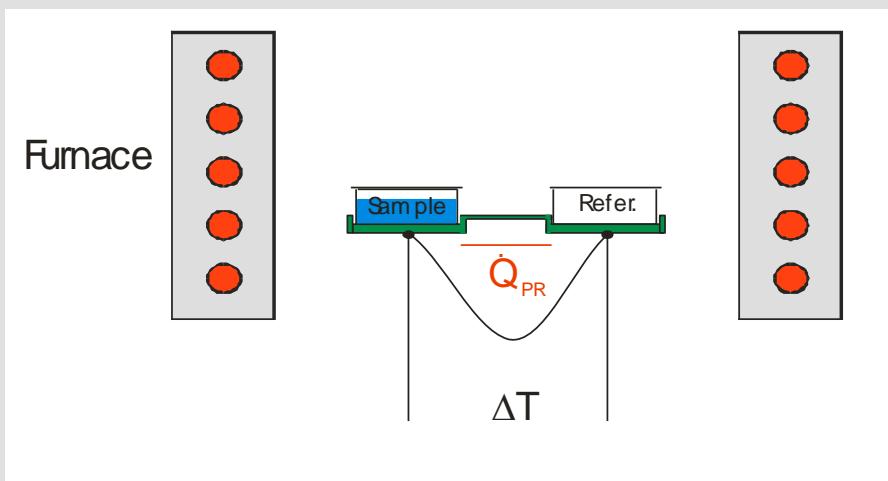
ARC® 244  
ARC® 254  
APTAC™ 264  
MMC 274 *Nexus*®  
IBC 284  
RT ... 500°C

DEA 288  
RT ... 375°C

**Evolved Gas Analysis (EGA)** for the advanced characterization of decomposition/evaporation effects  
QMS 403 *Aëolos*®, STA-MS-Skimmer®, FT-IR, GC-MS

# DSC Method

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- **Thermophysical Properties:**

- Specific Heat

- **Product Identification and Characterization:**

- Melting temperatures

- Transition enthalpies

- Phase transformations, phase diagrams

- Crystallization temperatures

- Degree of crystallinity

- Glass transition temperatures

- **Advanced Material Analysis:**

- Decomposition effects, O.I.T.

- Reaction kinetics

- Purity determinations

# DSC 204 *F1 Phoenix*<sup>®</sup> – Schematic Design

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DSC 204 *F1 Phoenix*<sup>®</sup> - ASC

## Technical data

- Temperature range: -180°C ... 700°C
- Heating and cooling rates: 0.001 K/min ... 200 K/min
- DSC detection limit: 2 µW (0.2 µW RMS; depending on sensor type)
- Exchangeable sensors: fast τ-sensor, high sensitive μ-sensor
- Several cooling devices: compressed air (to RT)  
Vortex tube (compressed air): < 0°C to 700°C  
intracooler (-85°C ... 600°C)  
**liquid nitrogen (-180°C ... 700°C)**
- Electronically controlled gas flow for purge and protective gases
- Specific heat measurements (software option)
- Advanced Software available: *Thermokinetics, Component Kinetics, Peak Separation, Purity Determination*, etc.
- Automatic sample changer (ASC) for 64 samples and all crucibles

# DSC 214 Polyma

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Ideal Combination for Optimum Results

*Arena Furnace ▶ Corona Sensor ▶ Concavus Pans*



Oval, low-mass silver furnace (→ Arena) is able to realize heating and cooling rates up to **500 K/min.**

**Ring sensor** consisting of a core made of NiCr and an external ring made of **constantan**. Both materials are **diffusion-bonded**.

This **ring-shaped contact zone** with the crucible has a positive effect on the measurement **reproducibility** – especially when using the **Concavus pans**.

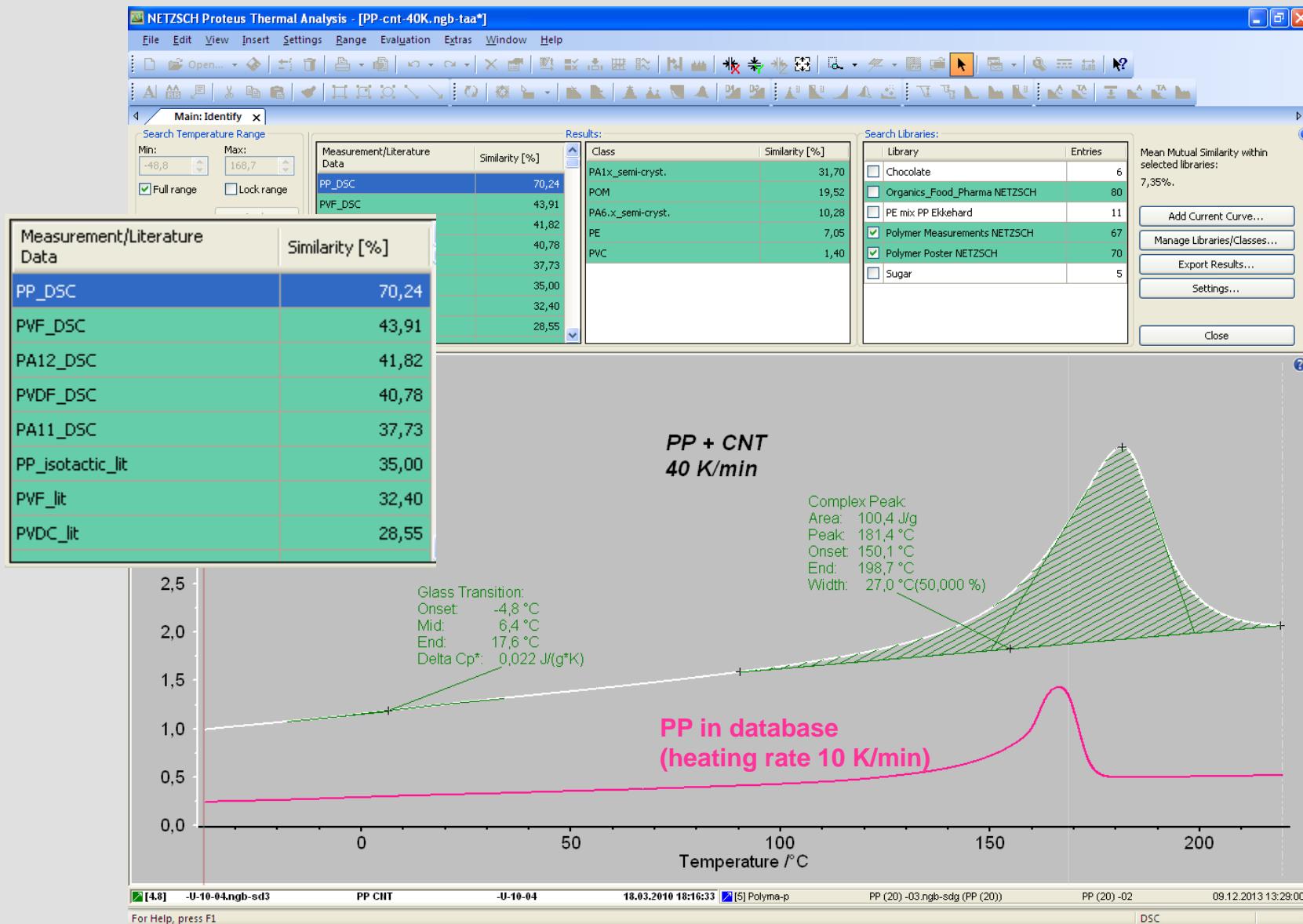
## Technical data

- Temperature range: -170°C ... 600°C
- Heating and cooling rates: 0.001K/min ... 500 K/min
- DSC technical resolution: 0.1 µW (1 µW RMS noise)  
compressed air (to RT);  
Vortex tube (compressed air) < 0°C ... 600°C  
liquid nitrogen (-170 ... 600°C)  
intracooler (-70/-40 ... 600°C)
- Sensitivity: 4 µV/mW (Indium - 156.6°C)
- Time constant: 0.8 s ± 0.1 s (empty system; <5.4 s with Concavus pan and Indium sample)
- Software: *SmartMode, ExpertMode, AutoCalibration, AutoEvaluation, Identify of Polymers....*
- Advanced Software available: *Thermokinetics, Component Kinetics, Peak Separation, Purity Determination*, etc.
- Automatic sample changer: 20 samples and all crucibles

# A PP+CNT Compound verified with *Identify*

PP (Polypropylene) is recognized - even at a heating rate of 40 K/min

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## Technical data

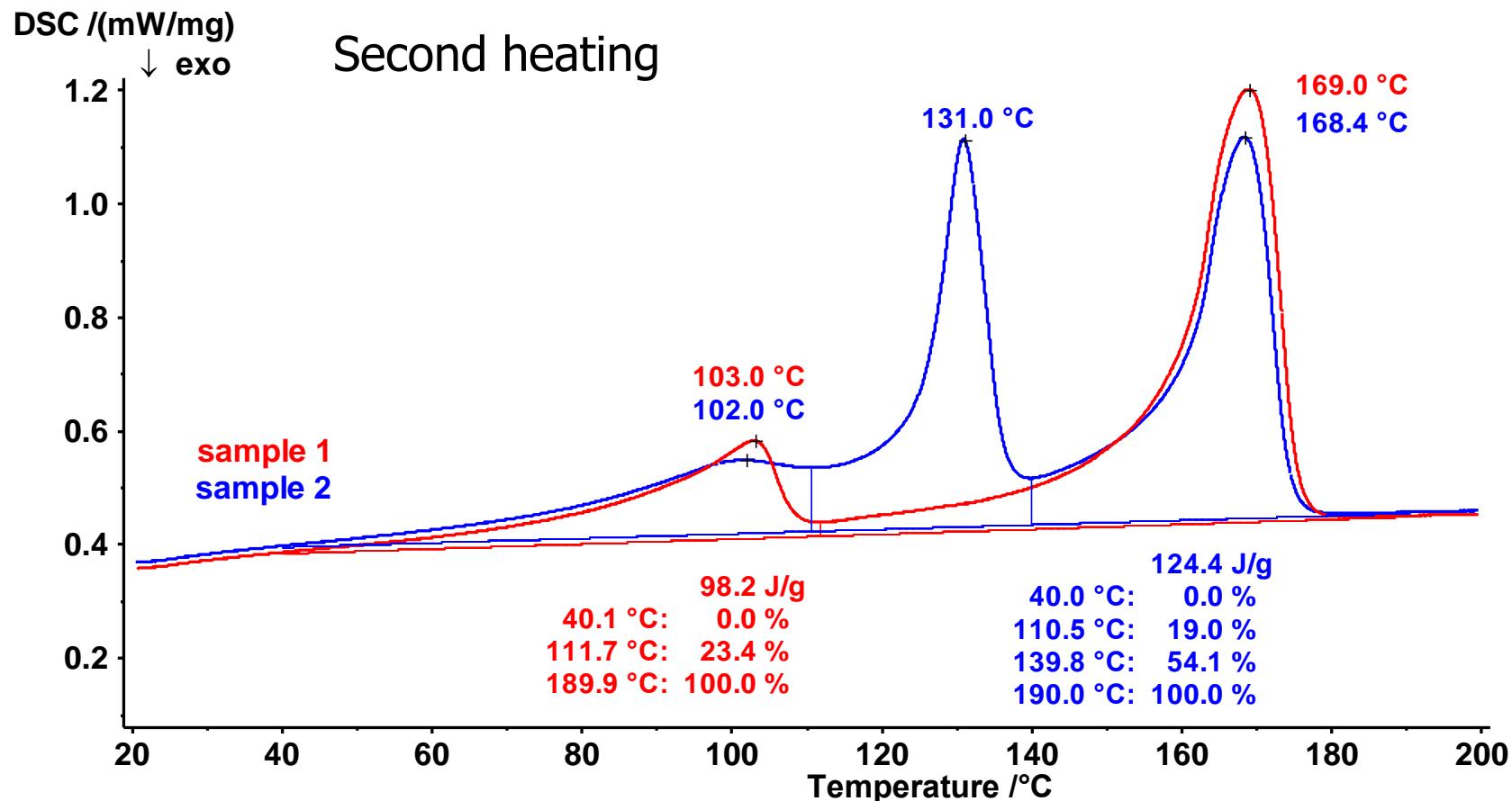
- Temperature range: -170°C ... 600°C
- Heating and cooling rates: 0.001K/min ... 100 K/min
- Digital resolution: 0.2 µW
- Several cooling devices: compressed air (to RT);  
liquid nitrogen (-170 ... 600°C)  
intracooler (-70/-40 ... 600°C)
- Sensitivity: 3.8 µV/mW (Indium)
- Time constant: 2.5 s
- Software: Proteus®
- Advanced Software available: *Thermokinetics, Component Kinetics, Peakseparation, Purity Determination, etc.*
- Automatic sample changer: 20 samples and all crucibles with possibility to change reference sample

# Application – HDPE Packaging

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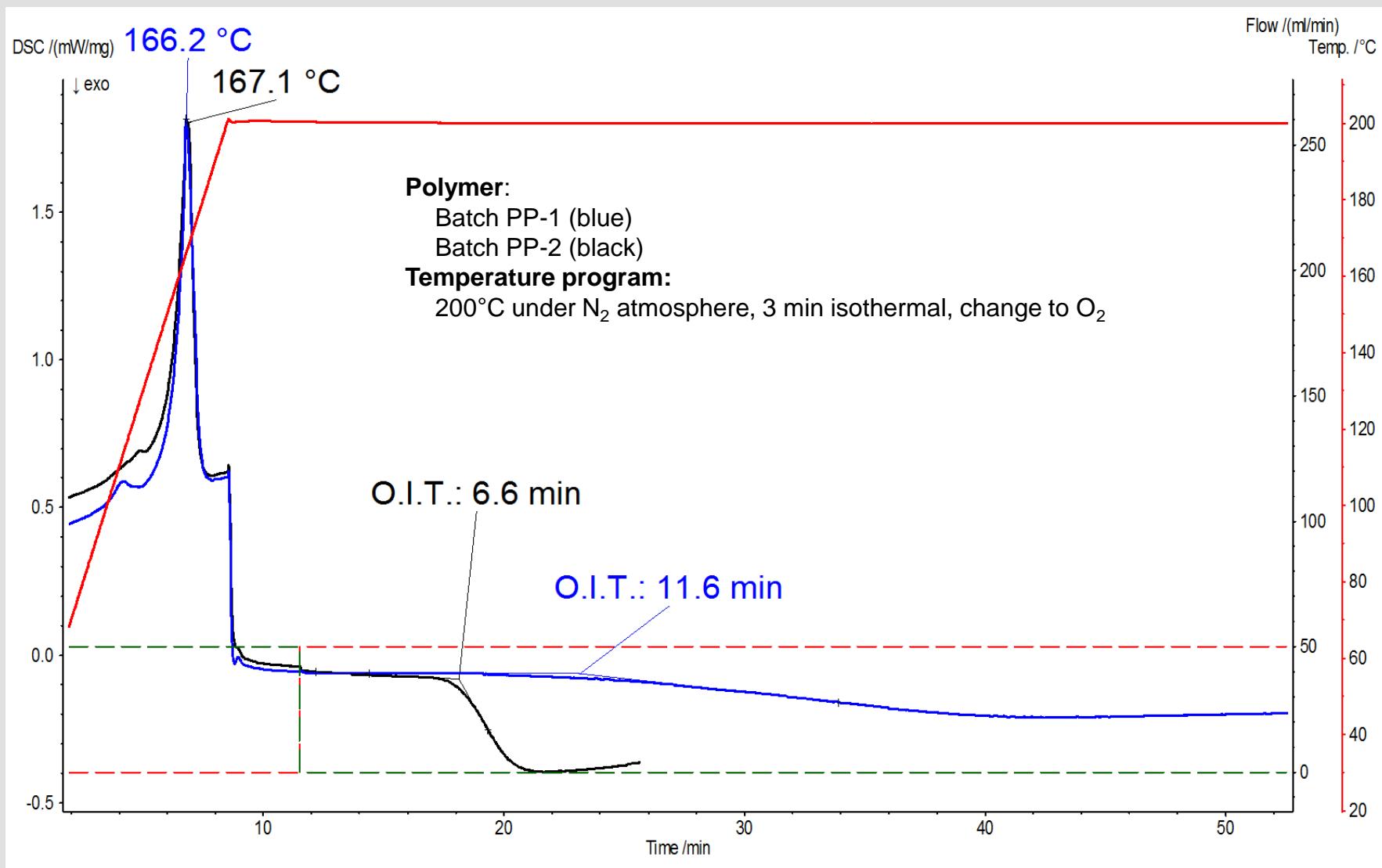
HDPE = High-Density Polyethylene





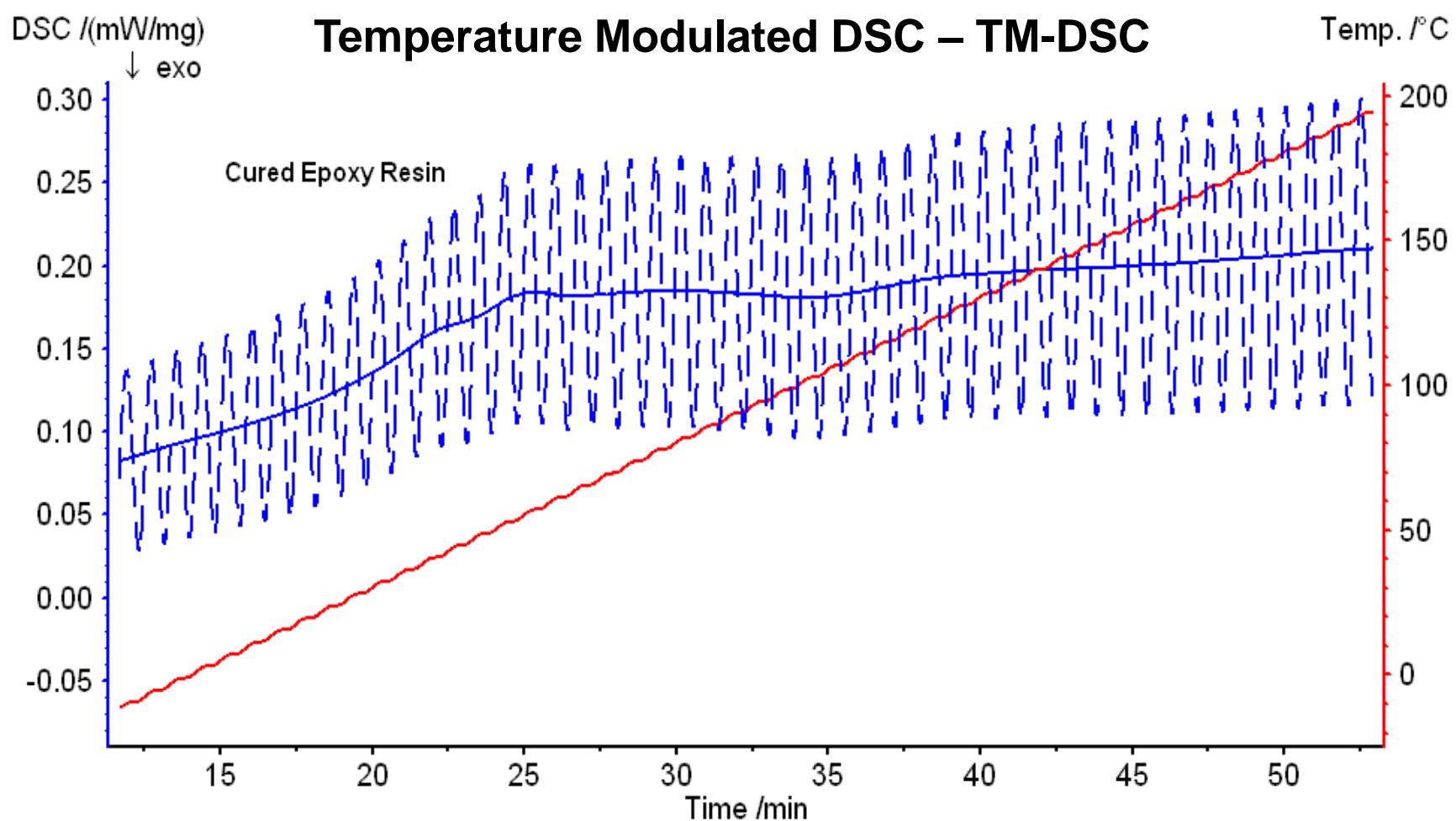
# OIT: Comparison of Oxidation Stability of PP parts

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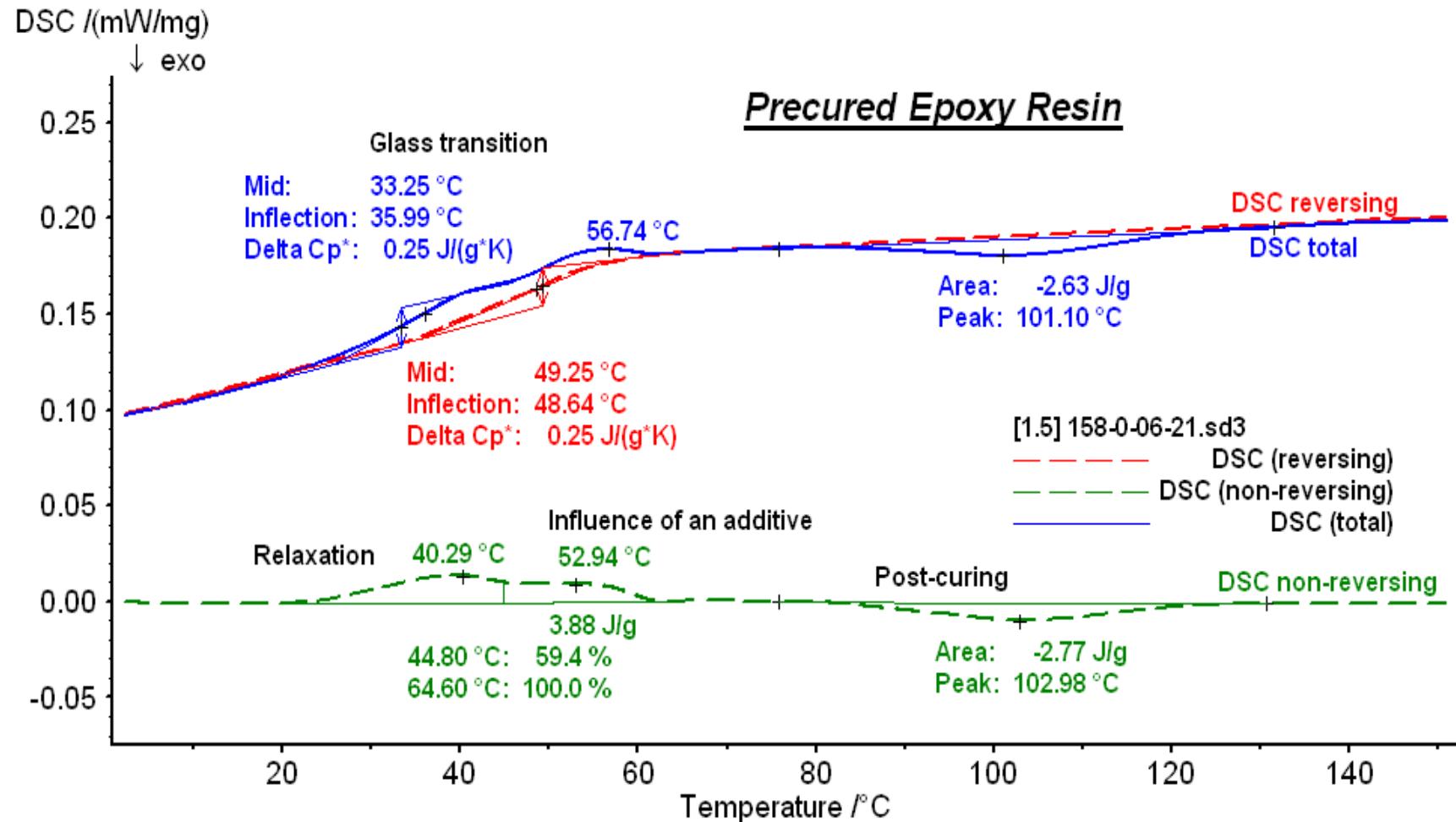


DSC 204 *F1 Phoenix*<sup>®</sup> TM-DSC  
Post-Curing of an Epoxy Resin

**NETZSCH**

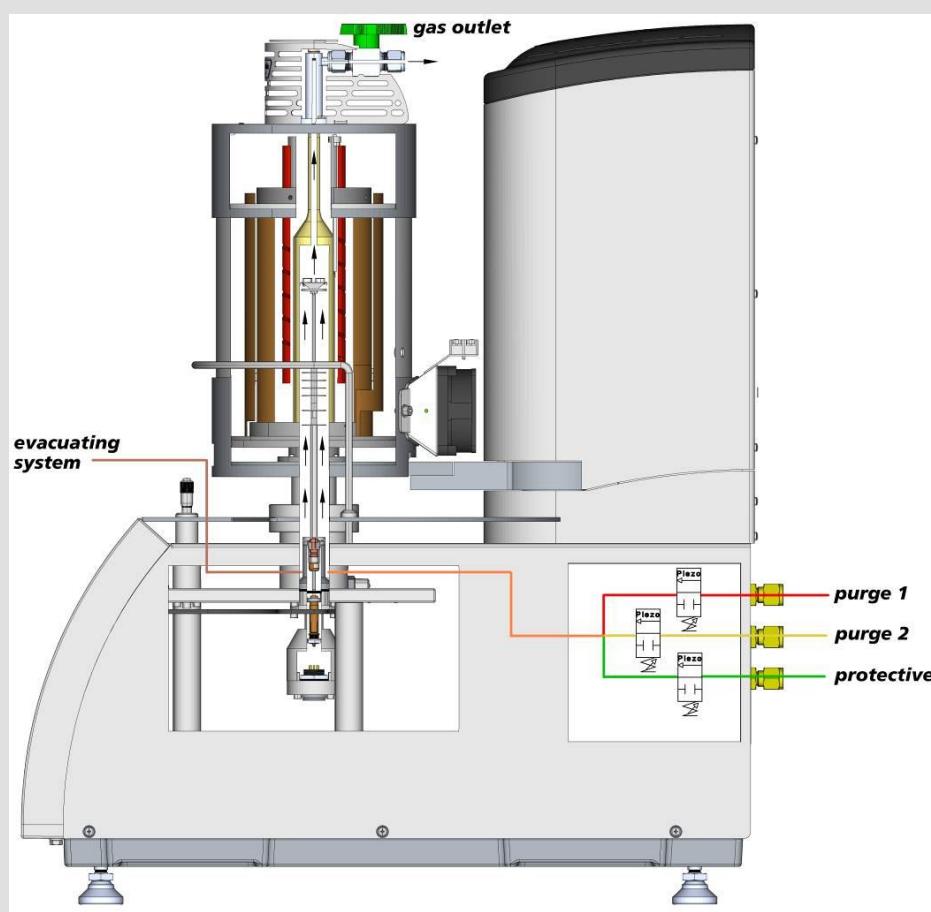


## TM-DSC: Post-Curing of Epoxy Resin



# DSC 404 F1/F3 Pegasus® – High Temp. Base Units

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## Technical data

- Temperature range: -150°C ... 2400°C (depending on the furnace type: **silver, steel, platinum, rhodium, SiC, graphite, tungsten**)
- Vacuum tightness: 10<sup>-4</sup> mbar/10<sup>-2</sup> mbar
- Sensors DTA/DSC/DSCcp: Exchangeable (easy) E, K, S, P, B, W type
- Heating and cooling rates: 0.001 ... 50 (1000) K/min
- DSC resolution: 0.1 µW
- Several cooling devices: compressed air (to RT)  
liquid nitrogen (-150°C ... 1000°C)
- Sensitivity: 16 µV/mW (Indium)
- Time constant: < 7 sec. (including sample)

# DSC Performance – Diopside Glass Powder

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Cp /( $J/(g^{\circ}K)$ )

**Specific heat capability!**

**Weak and strong transitions!**

Glass transition

Onset: 723.8 °C  
Mid: 733.9 °C  
Inflection: 739.5 °C  
End: 744.9 °C  
Delta Cp\*: 0.522 J/(g\*K)

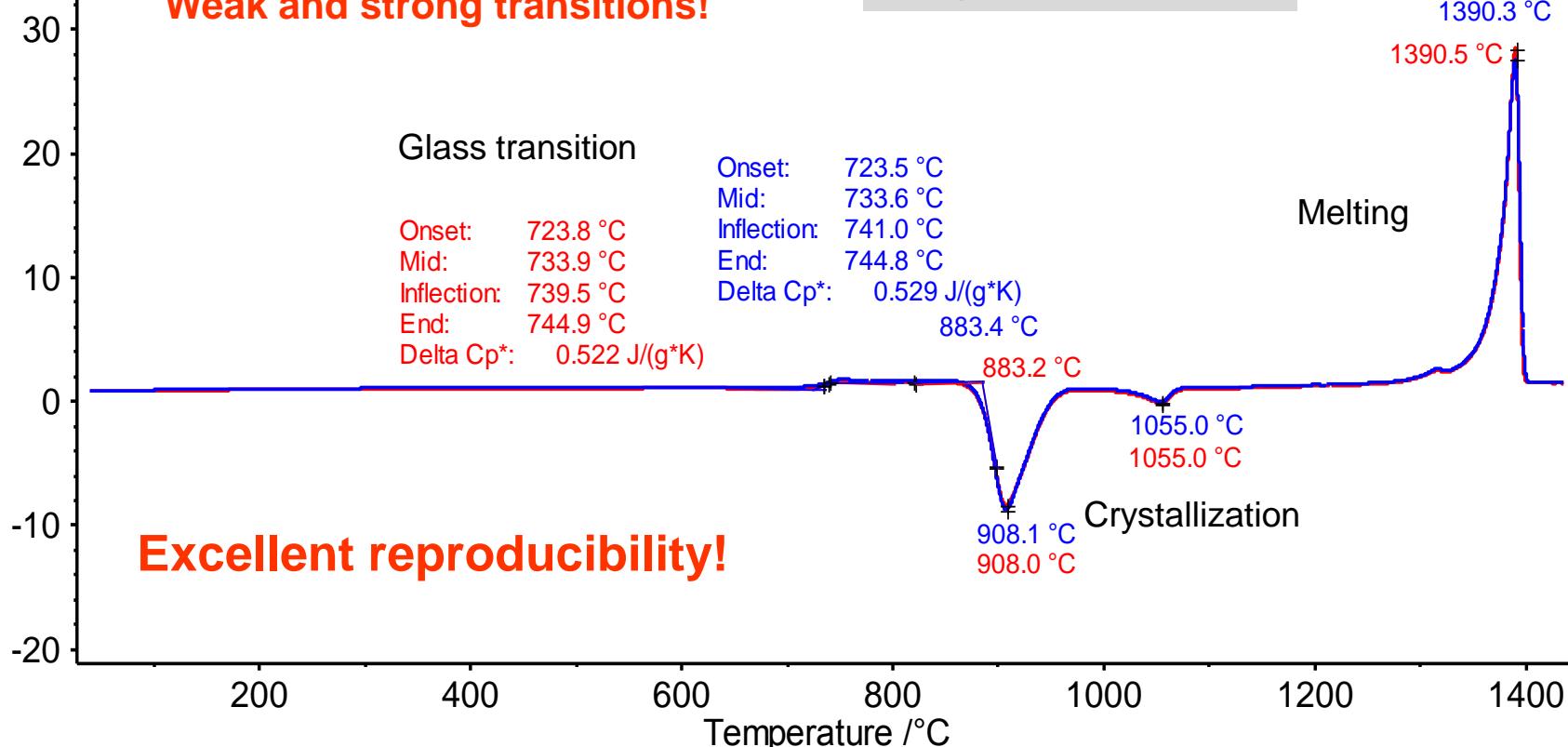
Onset: 723.5 °C  
Mid: 733.6 °C  
Inflection: 741.0 °C  
End: 744.8 °C  
Delta Cp\*: 0.529 J/(g\*K)

883.4 °C

883.2 °C

**Excellent reproducibility!**

Sample: Glass powder  
Sample mass: ~50 mg  
Crucible: Pt+lid  
Sample holder: DSC-c<sub>p</sub>  
Heating rate: 20 K/min  
Atmosphere: Air at 0 ml/min

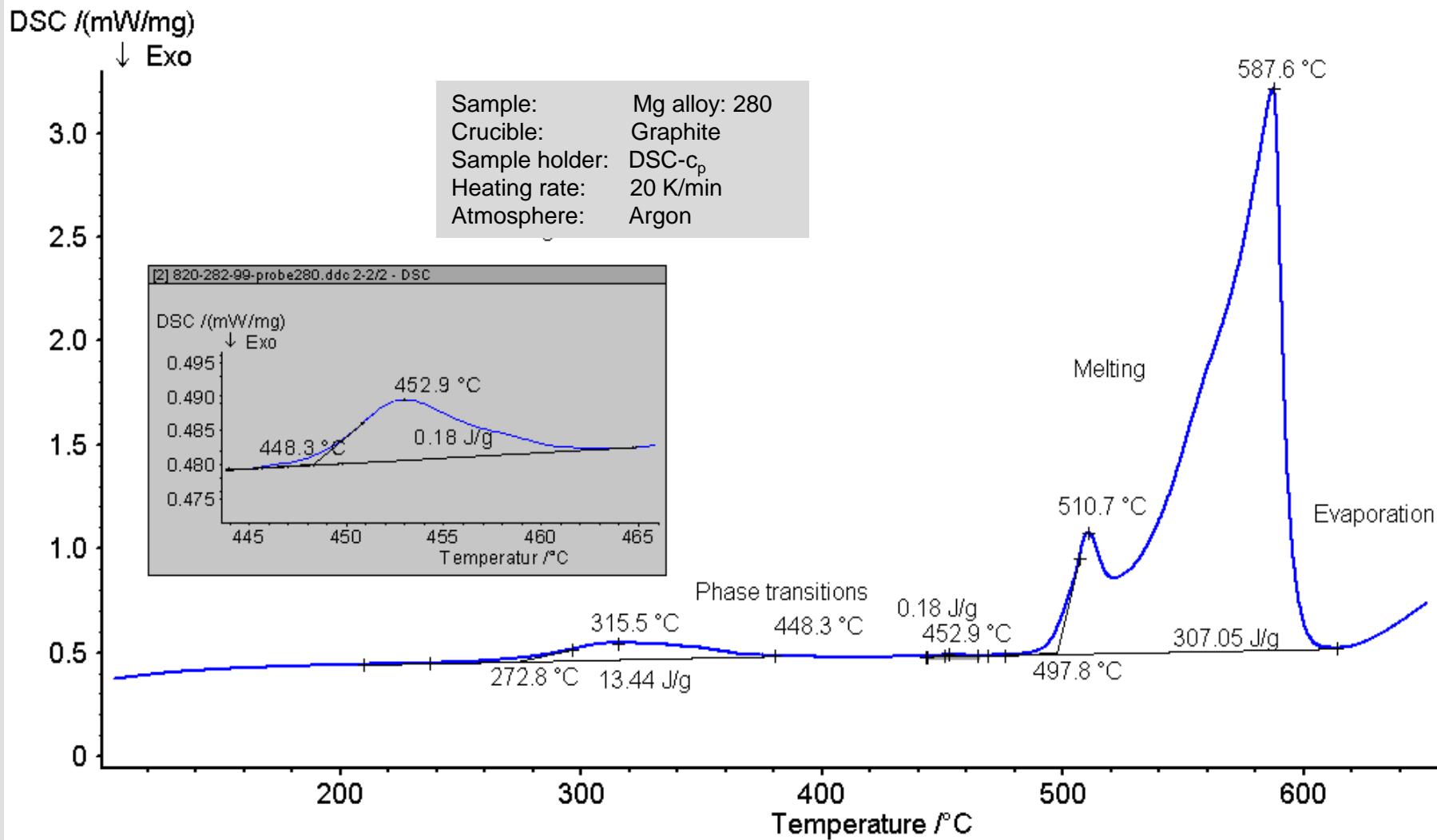


# Magnesium Alloys

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# Magnesium Alloys

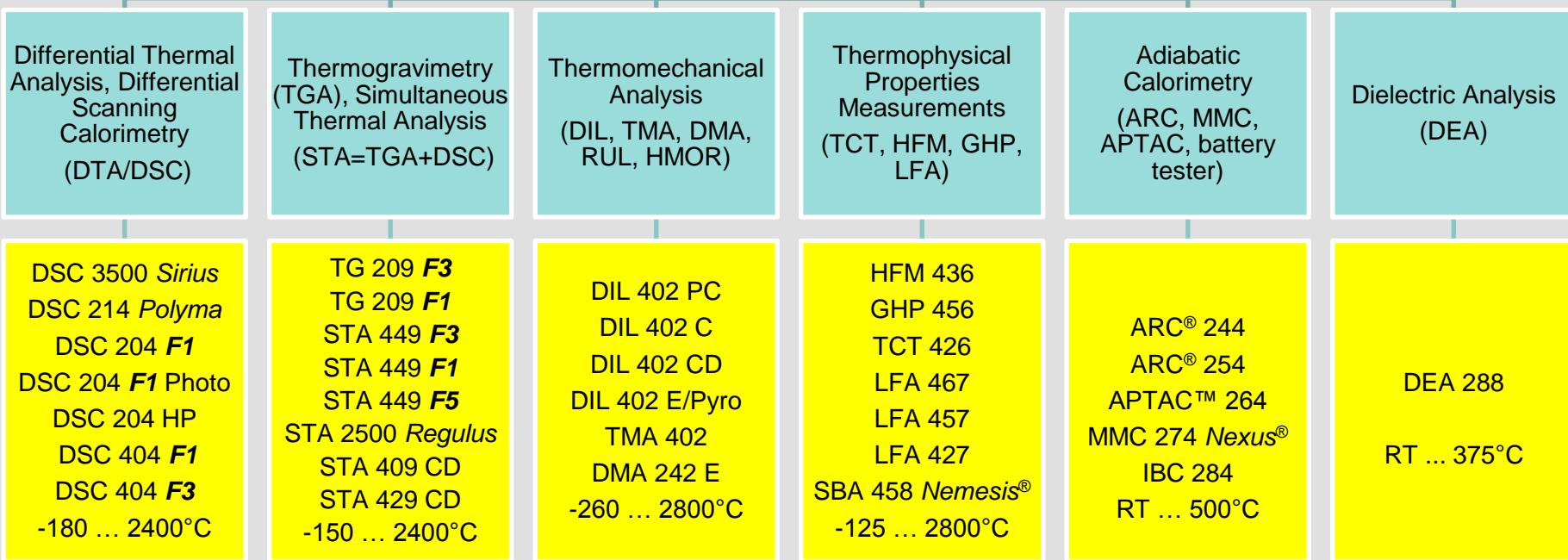


# The NETZSCH Product Range – Optimized Instruments with Outstanding Features

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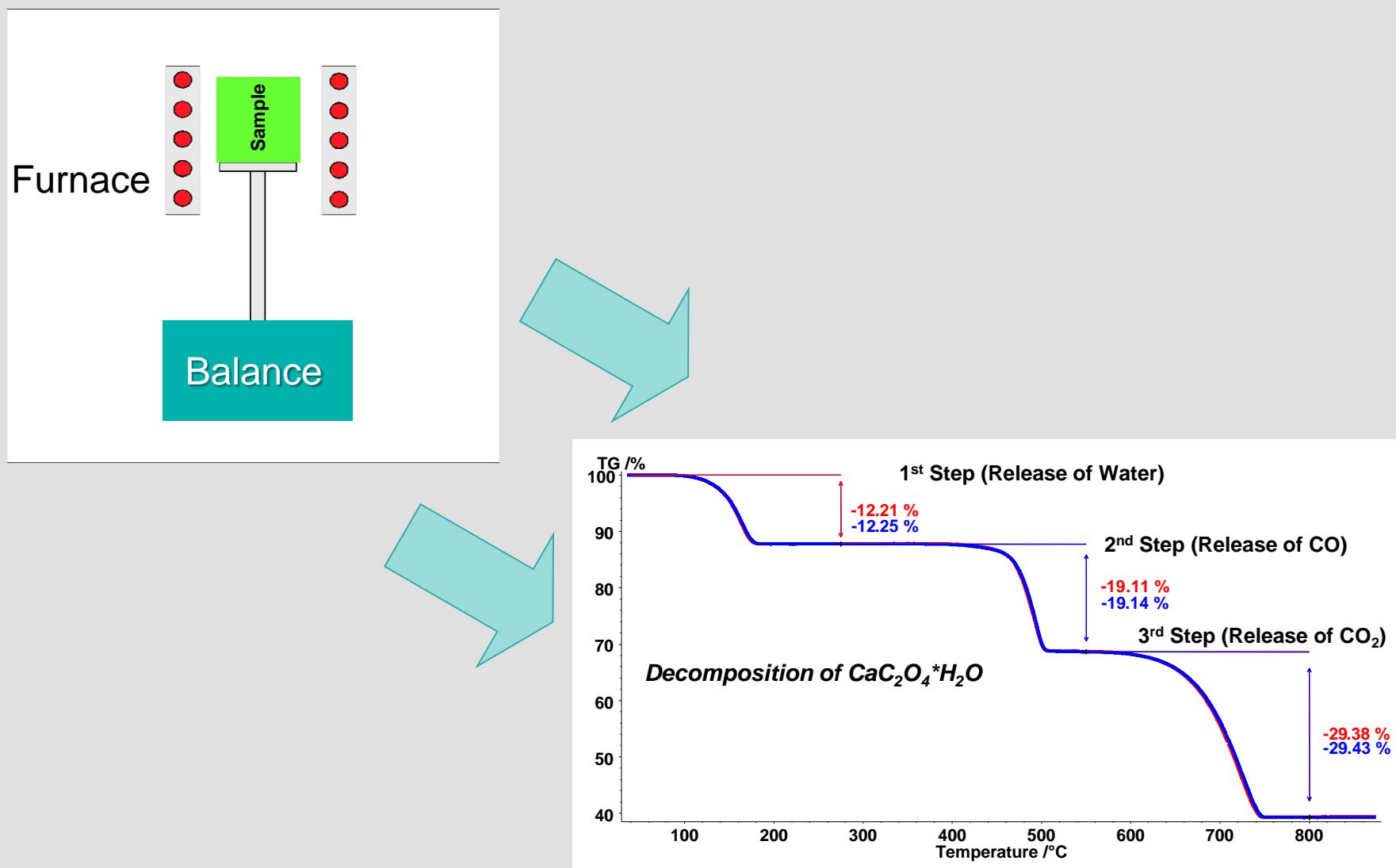
## Materials Characterization by Thermal Analysis Methods



Evolved Gas Analysis (EGA) for the advanced characterization of decomposition/evaporation effects  
QMS 403 *Aëolos*®, STA-MS-Skimmer®, FT-IR, GC-MS

# TGA Method

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- **Thermophysical Properties:**

- Density change

- **Product Identification and Characterization:**

- Mass change

- Decomposition temperatures

- Thermal stability

- Compositional analysis

- Oxidation behavior

- **Advanced Material Analysis:**

- Decomposition kinetics

- Rate-controlled mass loss



- **Technical data**

- Temperature range: RT to 1000°C / 1100°C
- Heating rates: 0.001 ... 100 / 200 K/min
- Wide measuring range: 2000 mg
- Resolution: 0.1 µg
- Atmospheres: inert, oxidizing, static, dynamic
- c-DTA<sup>®</sup> (Option)

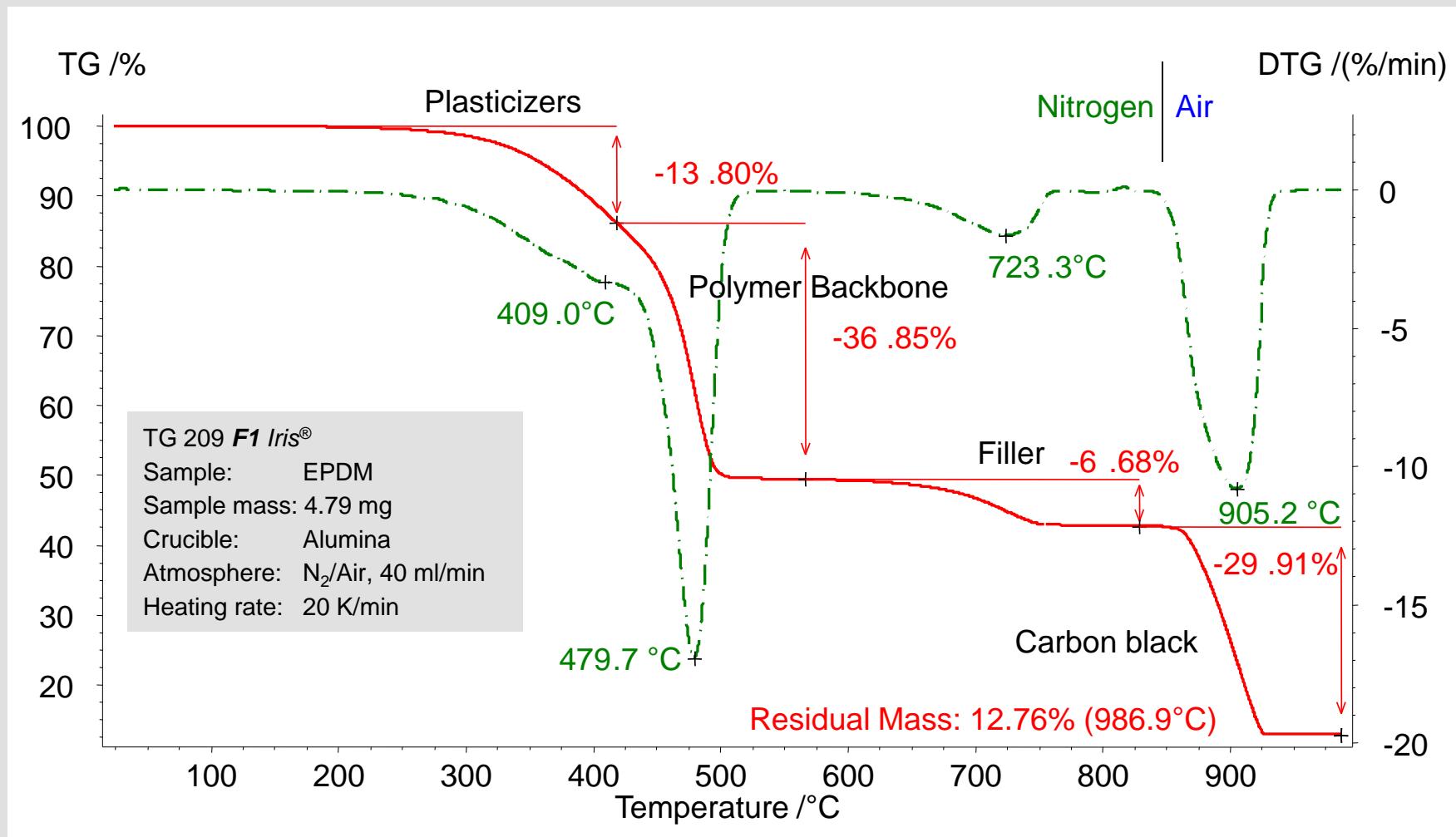
# Application – EPDM

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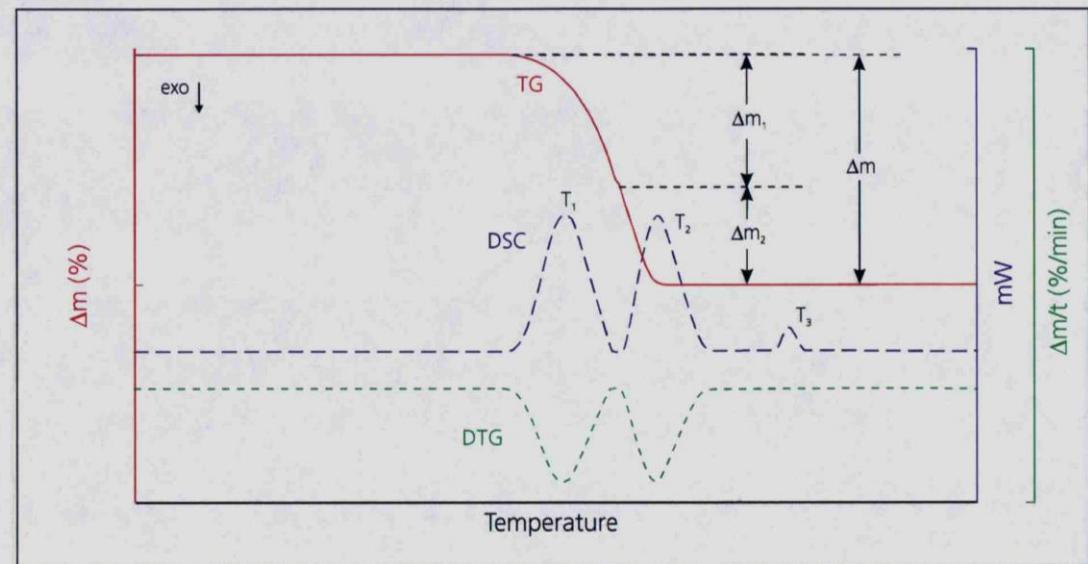
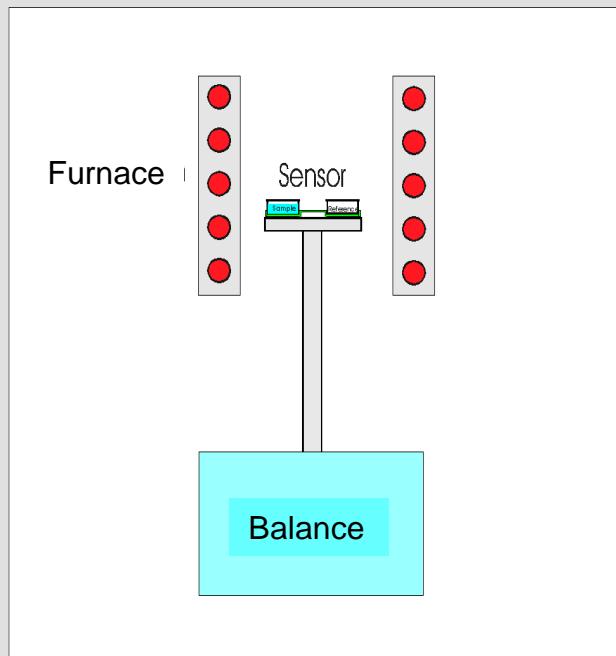
# Application – EPDM (ethylene propylene diene monomer) rubber

**NETZSCH**



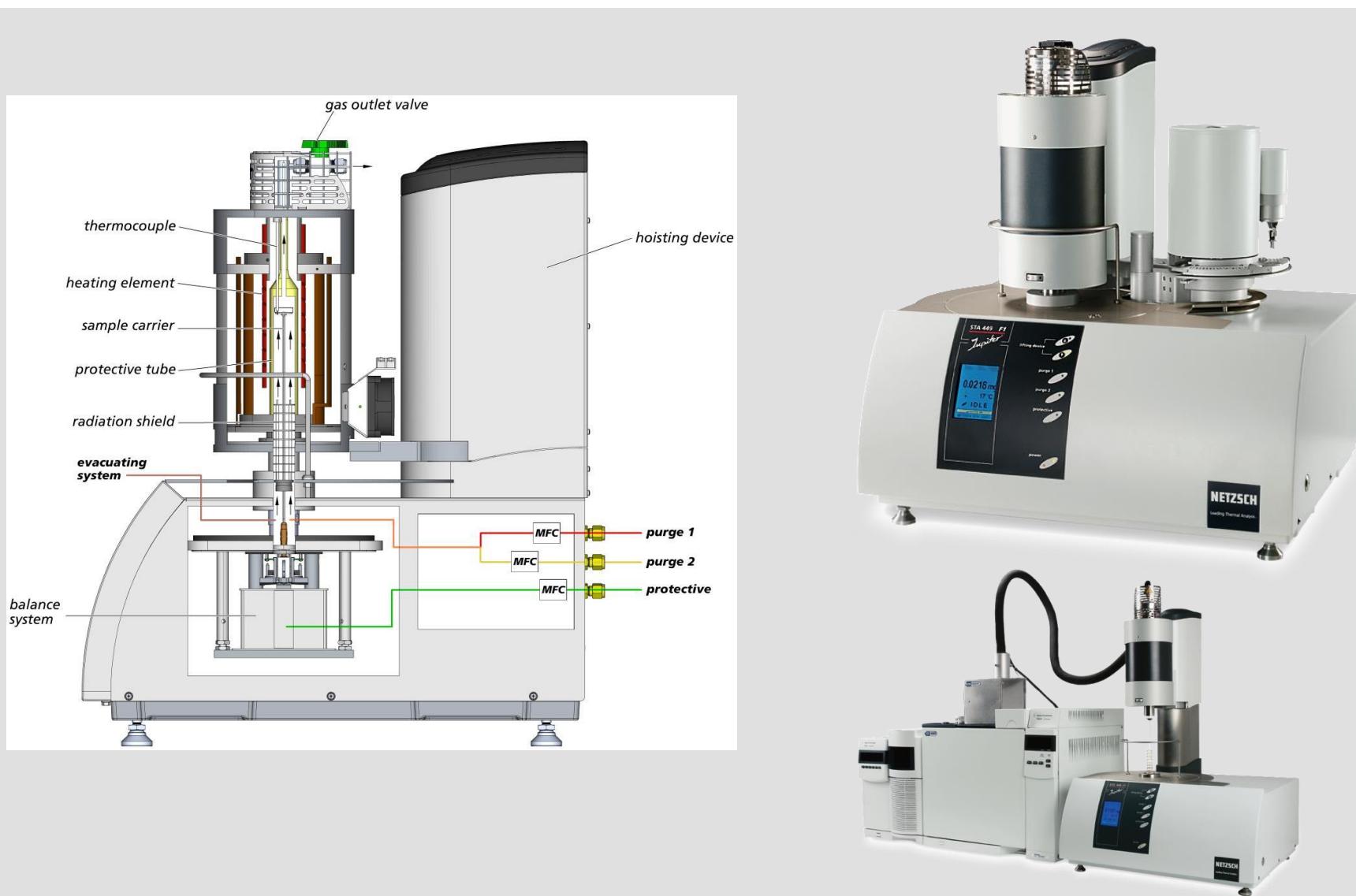
# STA Method

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# STA 449 F1/F3 Jupiter®— Basic Units

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# DSC/STA Body with Automatic Sample Changer

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# STA 449 F5

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# STA 2500 *Regulus*

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STA 2500  
*Regulus*  
coupled to  
GC-MS



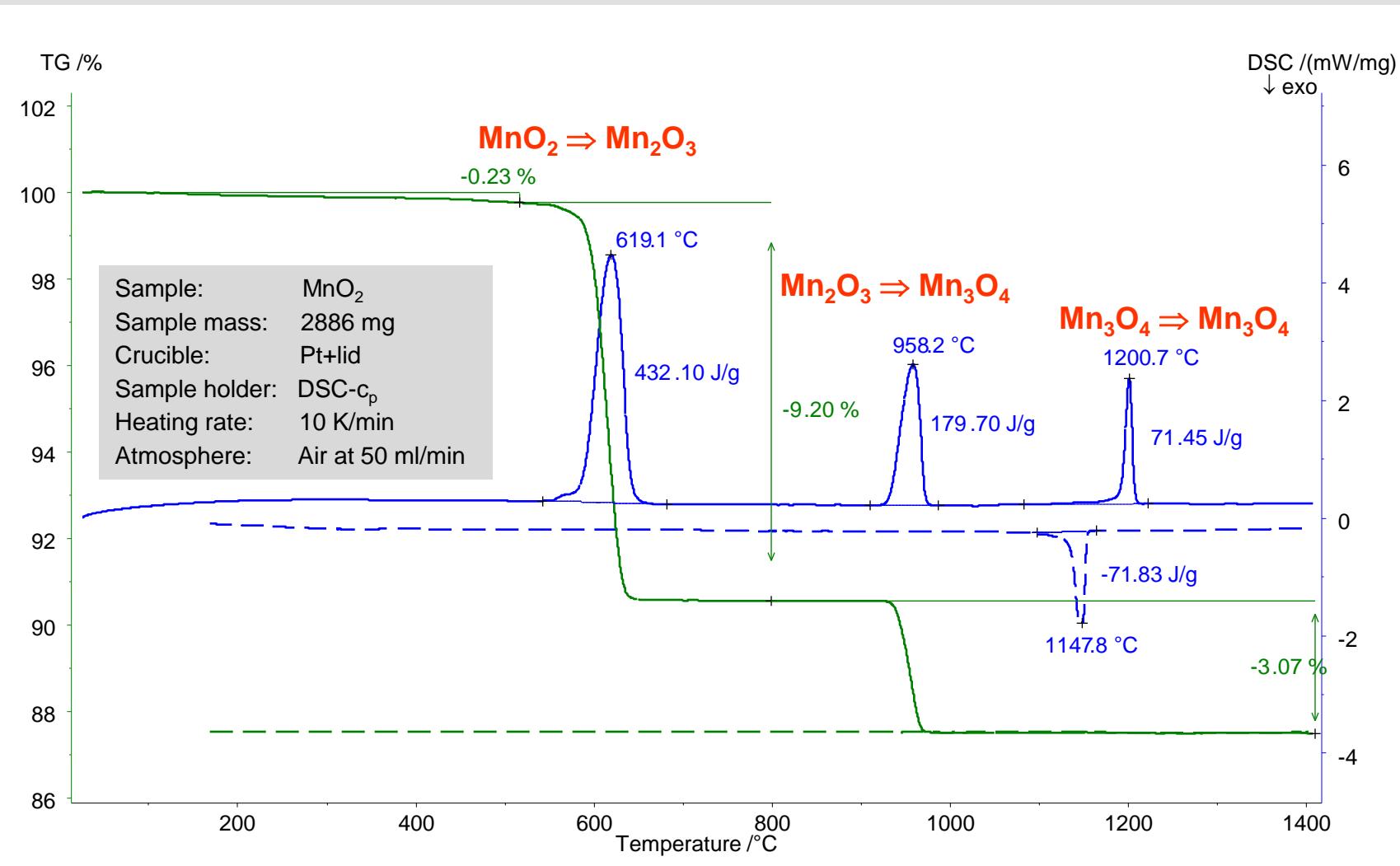
# Technical Specifications

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	STA 449 <b>F5</b> <i>Jupiter®</i>	STA 449 <b>F3</b> <i>Jupiter®</i>	STA 449 <b>F1</b> <i>Jupiter®</i>	STA 2500 <i>Regulus</i>
<b>Temperature range</b>	RT to 1600°C	-150°C ... 2400°C	-150°C ... 2400°C	RT to 1600°C
Vacuum	<10 <sup>-2</sup> mbar (1Pa)	<10 <sup>-4</sup> mbar (0.01Pa)	<10 <sup>-4</sup> mbar (0.01Pa)	<10 <sup>-2</sup> mbar (1Pa)
DSC sensors	standard	standard	standard	TGA-DTA
DSC-c <sub>p</sub> sensors	option	option	standard	n/a
<b>Quick-Change</b>	<b>standard</b>	<b>standard</b>	<b>standard</b>	n/a
<b>Balance resolution</b>	0.1 µg	1 µg	<b>0.025 µg</b>	<b>0.03 µg</b>
<b>Max. sample mass</b>	35 g	35 g	5 g	1 g
Max. sample volume	<b>5 cm<sup>3</sup> (TG mode)</b>	<b>5 cm<sup>3</sup> (TG mode)</b>	<b>5 cm<sup>3</sup> (TG mode)</b>	400 µl
DSC resolution	0.1 µW (sensor dependent)	0.1 µW (sensor dependent)	0.1 µW (sensor dependent)	n/a
<b>OTS® gas purifier</b>	<b>optional</b>	<b>optional</b>	<b>optional</b>	n/a
<b>Double furnace hoist</b>	<b>no</b>	<b>optional</b>	<b>optional</b>	n/a
ASC	Standard in instrument version II	optional	optional	n/a
<b>Metal-housed MFC</b>	<b>standard</b>	optional	<b>standard</b>	<b>standard</b>
TM-DSC	no	no	<b>optional</b>	n/a
<b>BeFlat®, Tau-R Mode™</b>	<b>option</b>	<b>option</b>	<b>standard</b>	Differential balance
Autovac™	standard	optional	optional	optional
CO-Version	no	available	available	available

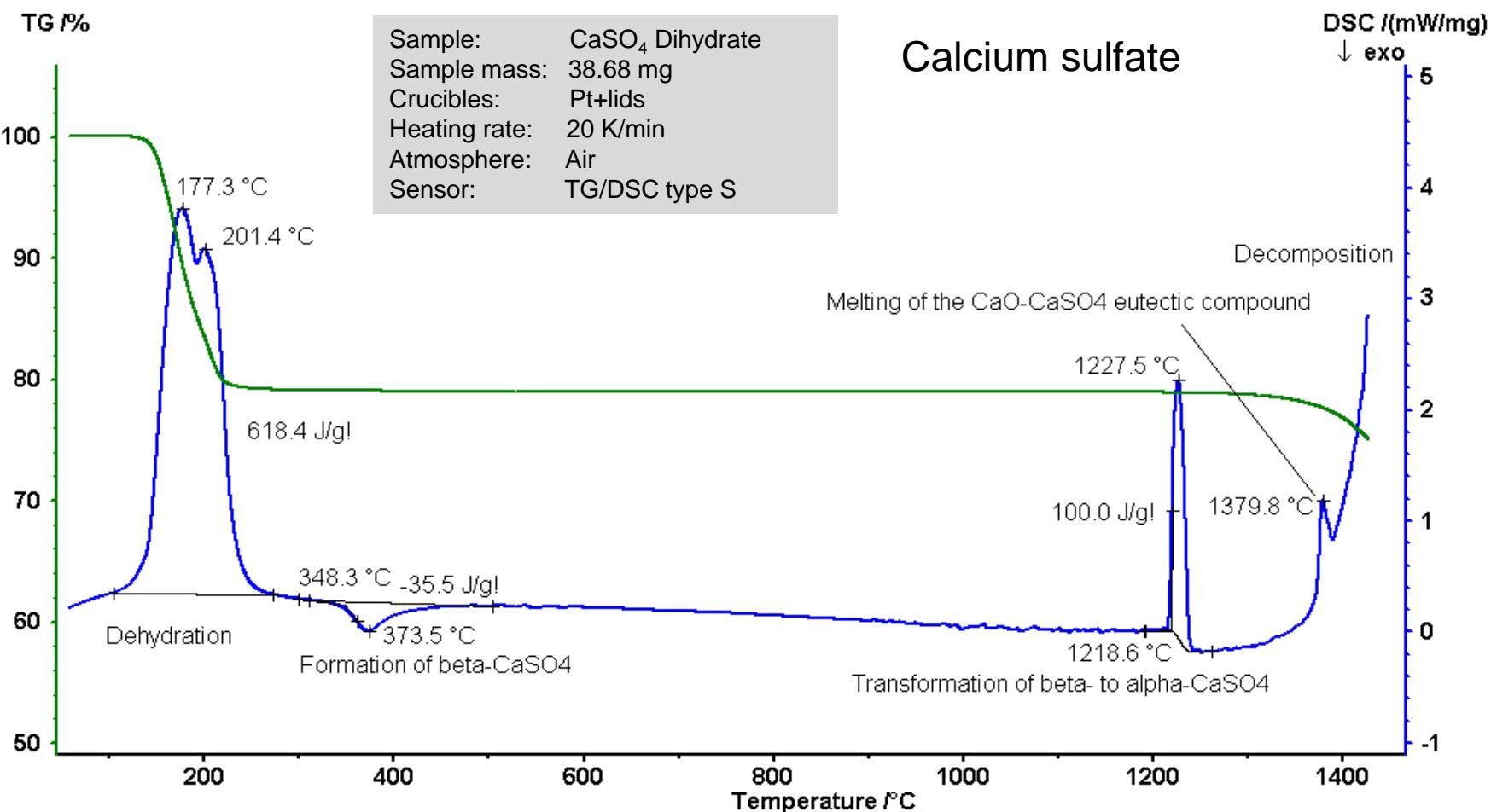
# STA Combined TGA-DSC Performance – Manganese Oxide

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# Gypsum ( $\text{CaSO}_4$ - Dihydrate) – Pt Crucibles

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DSC 204 **F1** Photo  
DSC 204 HP  
DSC 404 **F1**  
DSC 404 **F3**  
-180 ... 2400°C

TG 209 **F3**  
TG 209 **F1**  
STA 449 **F3**  
STA 449 **F1**  
STA 449 **F5**  
STA 2500 *Regulus*  
STA 409 CD  
STA 429 CD  
-150 ... 2400°C

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DIL 402 C  
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TMA 402  
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DEA 288  
RT ... 375°C

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# STA 449 *F1 Jupiter*<sup>®</sup> – QMS 403 Aëolos<sup>®</sup> Capillary Coupling

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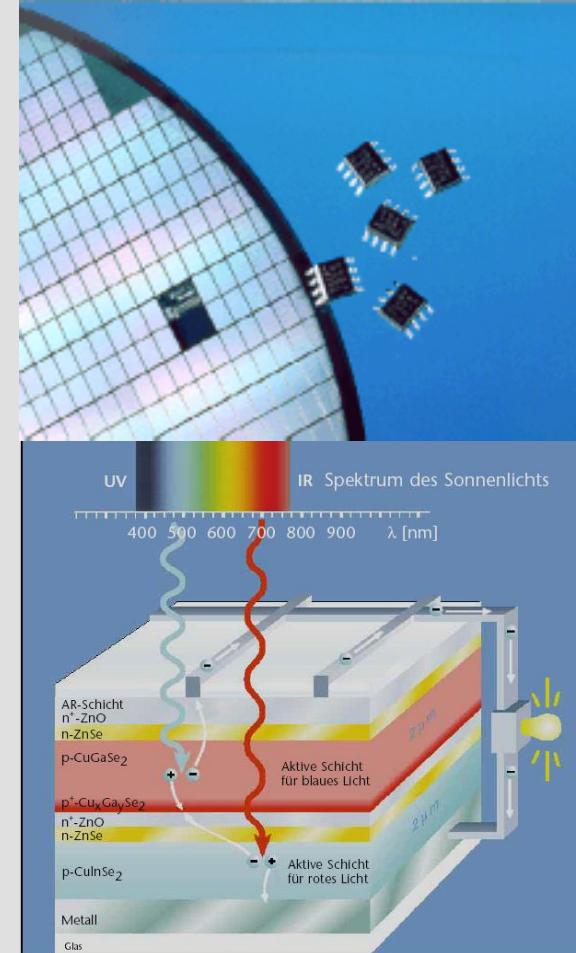
# Example: Organic Contaminations on Si Wafers

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## Silicon wafers and other semiconductor materials:

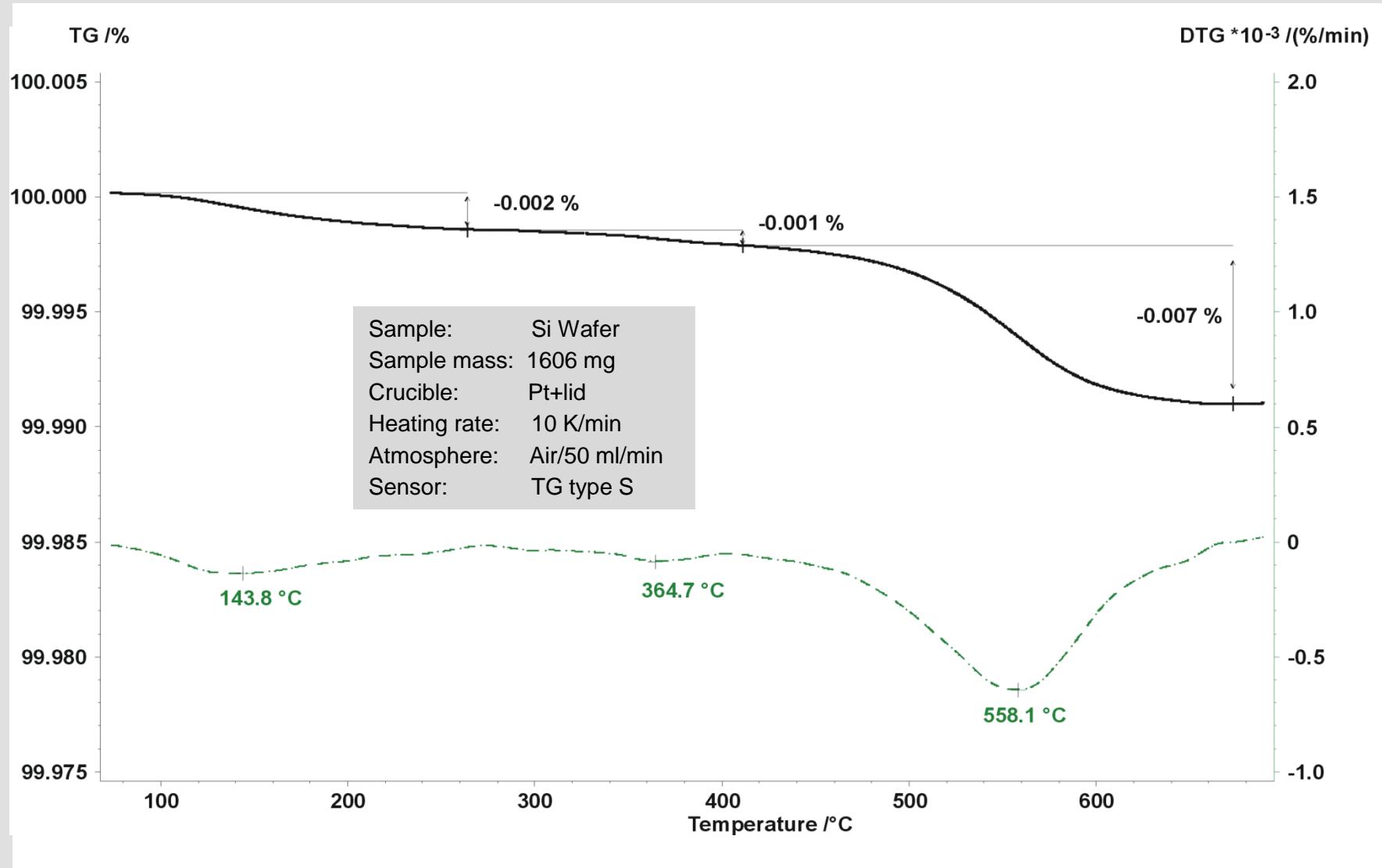
The most important basis of the microelectronics industry are semiconductors.  
No computer would work without silicon.

The purity of silicon wafers used in modern processors is one of the most important quality control parameters.



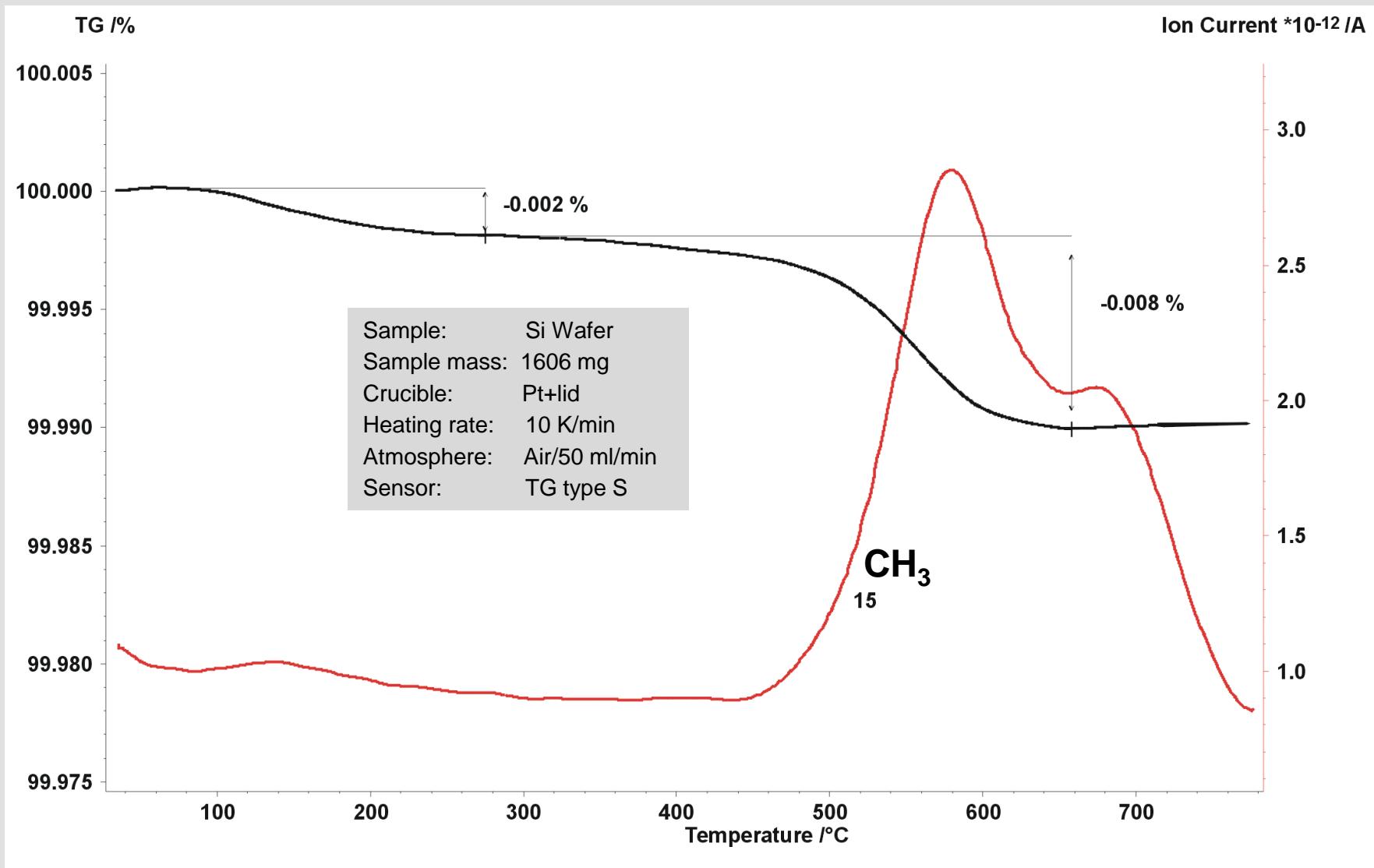
# Organic Contaminations on Si Wafers

**NETZSCH**



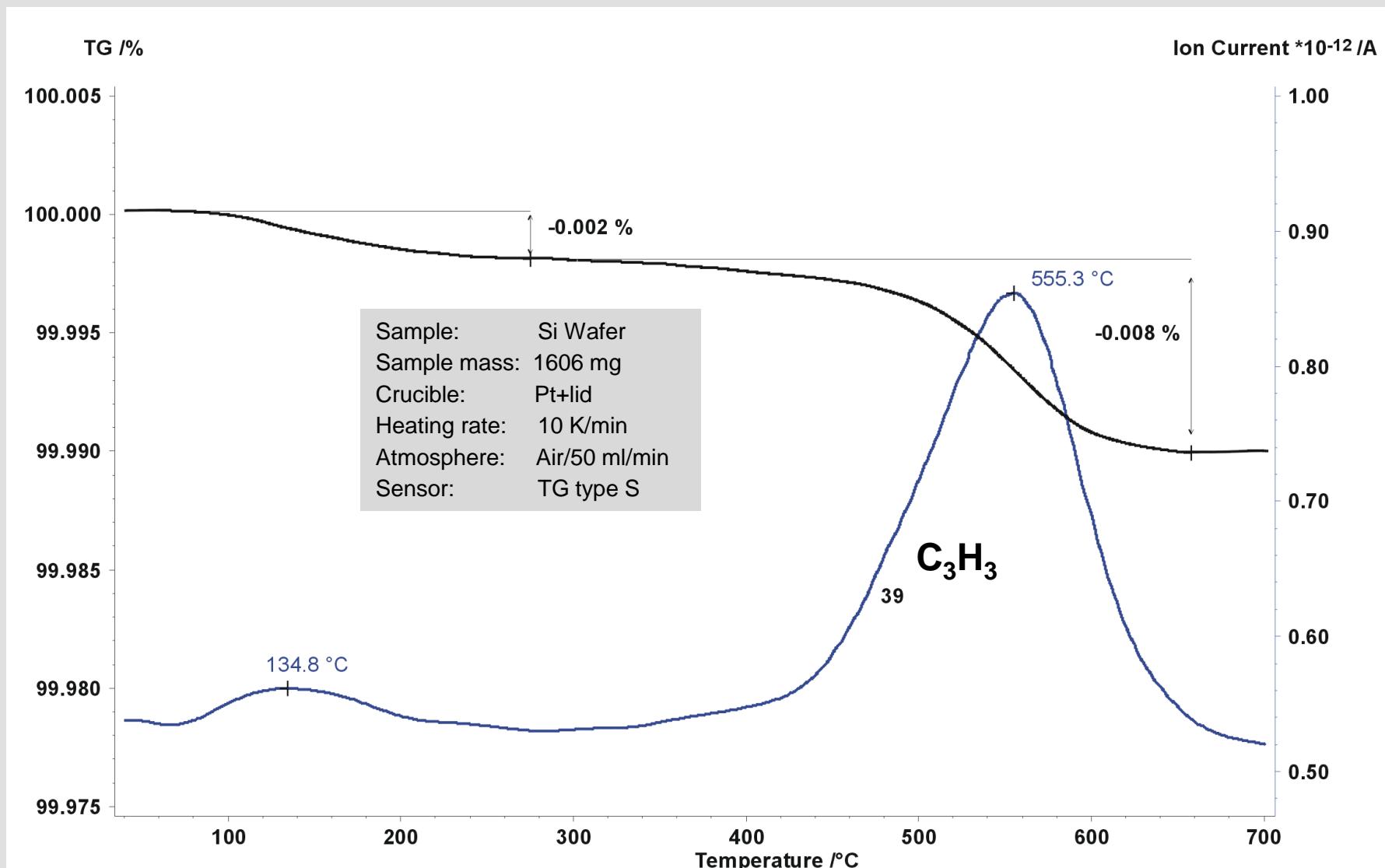
# Organic Contaminations on Si Wafers

**NETZSCH**



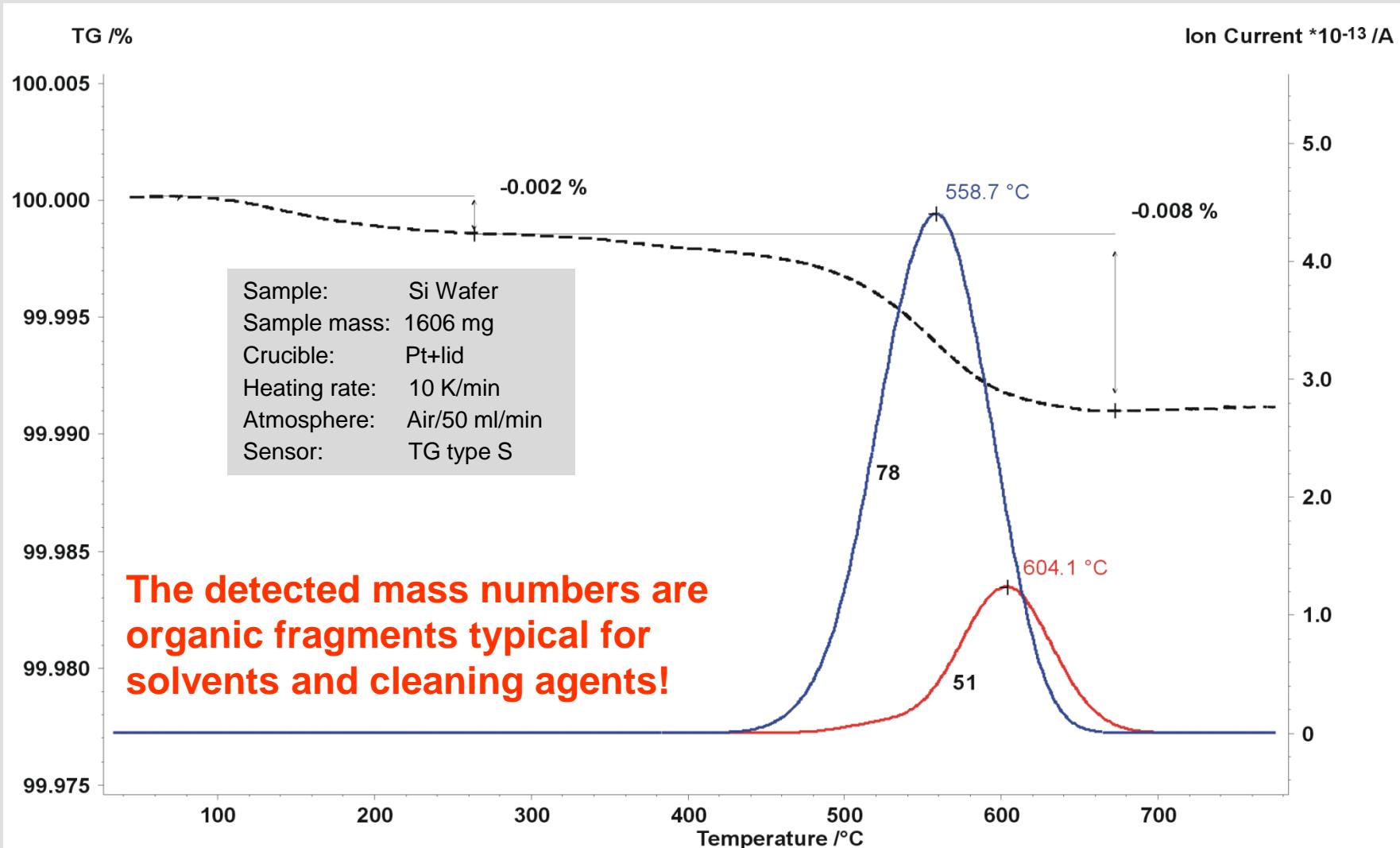
# Organic Contaminations on Si Wafers

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# Organic Contaminations on Si Wafers

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STA 449 **F5**  
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STA 409 CD  
STA 429 CD  
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DIL 402 PC  
DIL 402 C  
DIL 402 CD  
DIL 402 E/Pyro  
TMA 402  
DMA 242 E  
-260 ... 2800°C

HFM 436  
GHP 456  
TCT 426  
LFA 467  
LFA 457  
LFA 427  
SBA 458 *Nemesis*  
-125 ... 2800°C

ARC® 244  
ARC® 254  
APTAC™ 264  
MMC 274 *Nexus*  
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Evolved Gas Analysis (EGA) for the advanced characterization of decomposition/evaporation effects  
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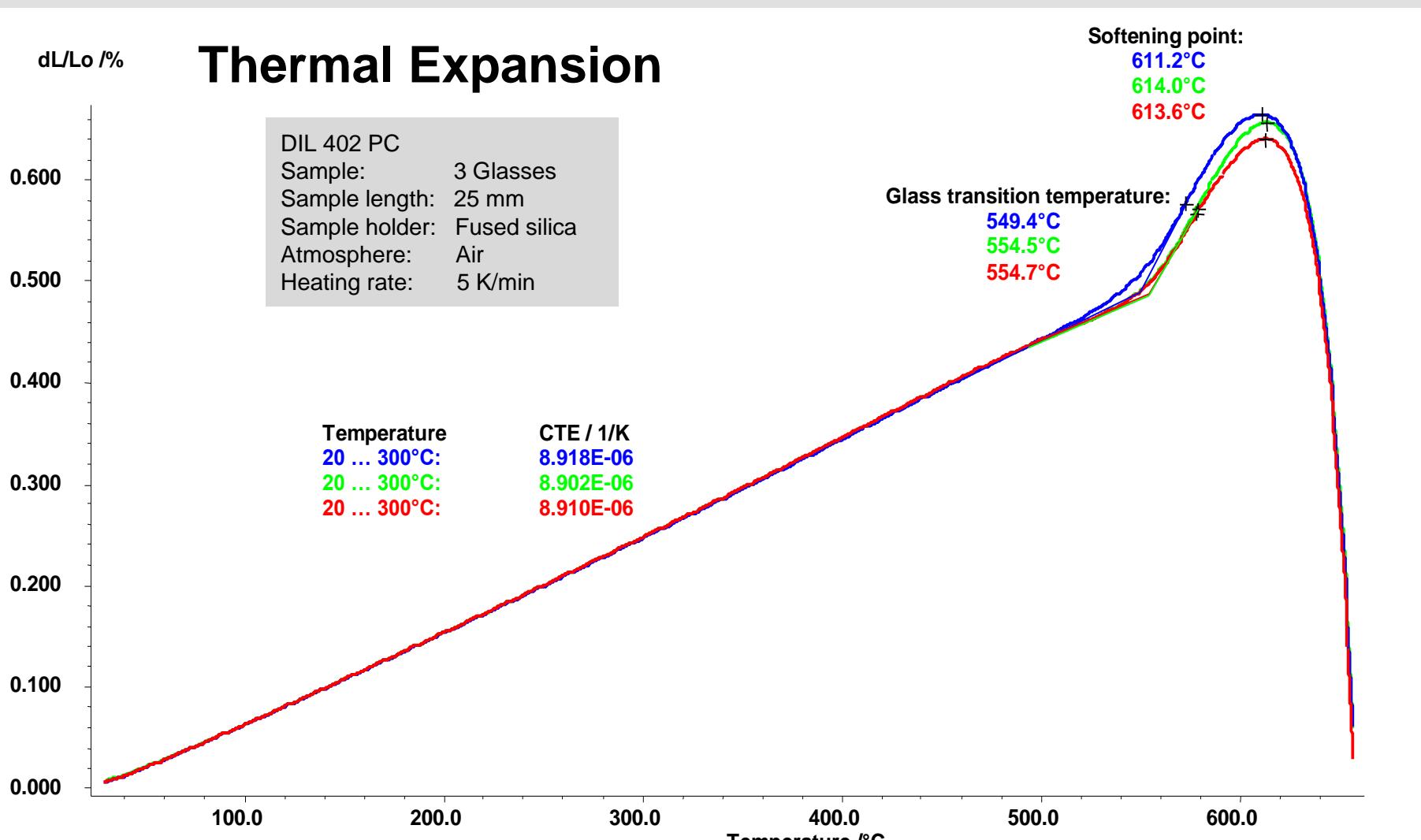
# NETZSCH DIL 402 C/4 (1650°C): Measurement Part

**NETZSCH**

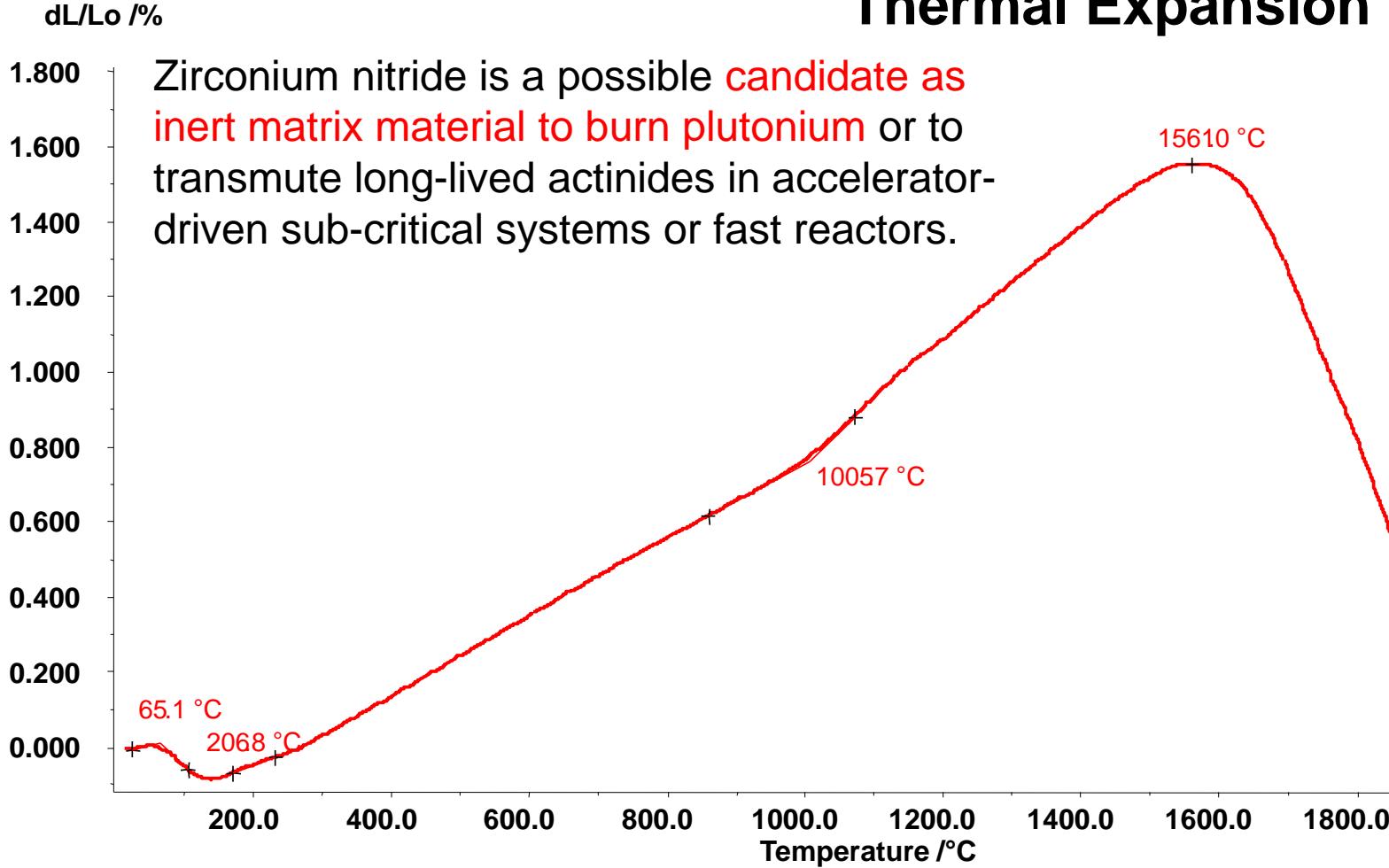


# Application – Glasses

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## Thermal Expansion



# TMA 402 F1/F3 Hyperion®

**NETZSCH**

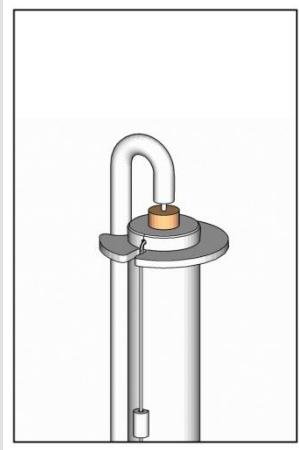


- Linear thermal expansion
- Coefficient of thermal expansion
- Phase transition temperatures
- Sintering temperatures
- Shrinkage steps
- Glass transition temperatures
- Dilatometry softening points
- Volumetric expansion
- Decomposition temperature,  
e.g. of organic binder
- Caloric effects
- Density changes
- Sintering kinetics

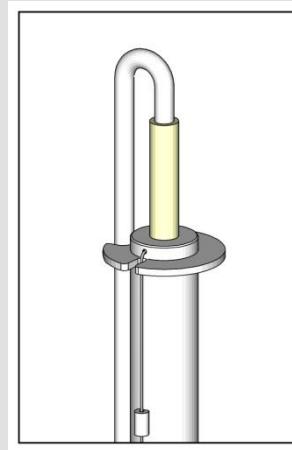
# The New Thermo-mechanical Analyzer TMA 402 Hyperion® – Sample Holder Types

**NETZSCH**

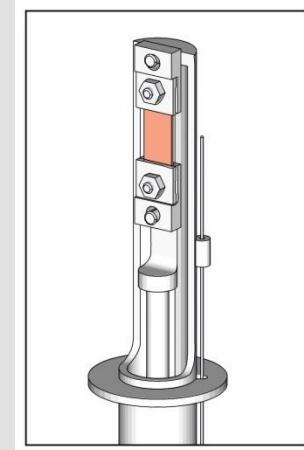
Penetration



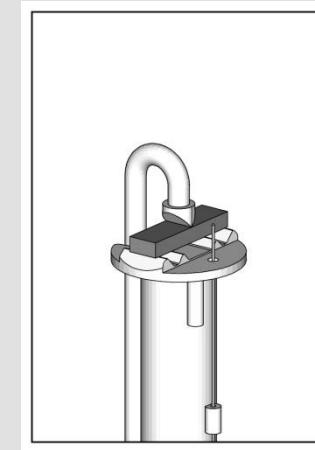
Expansion



Tension

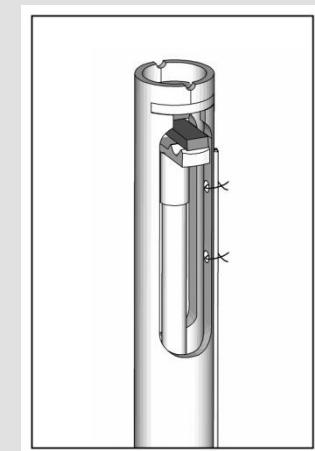
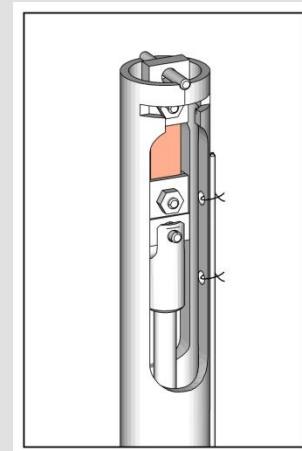
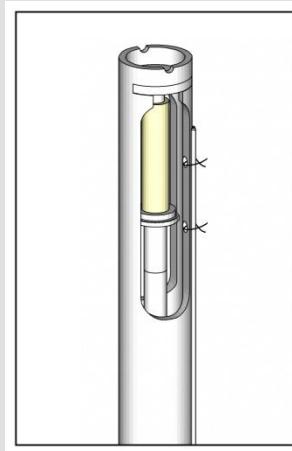


3-point bending



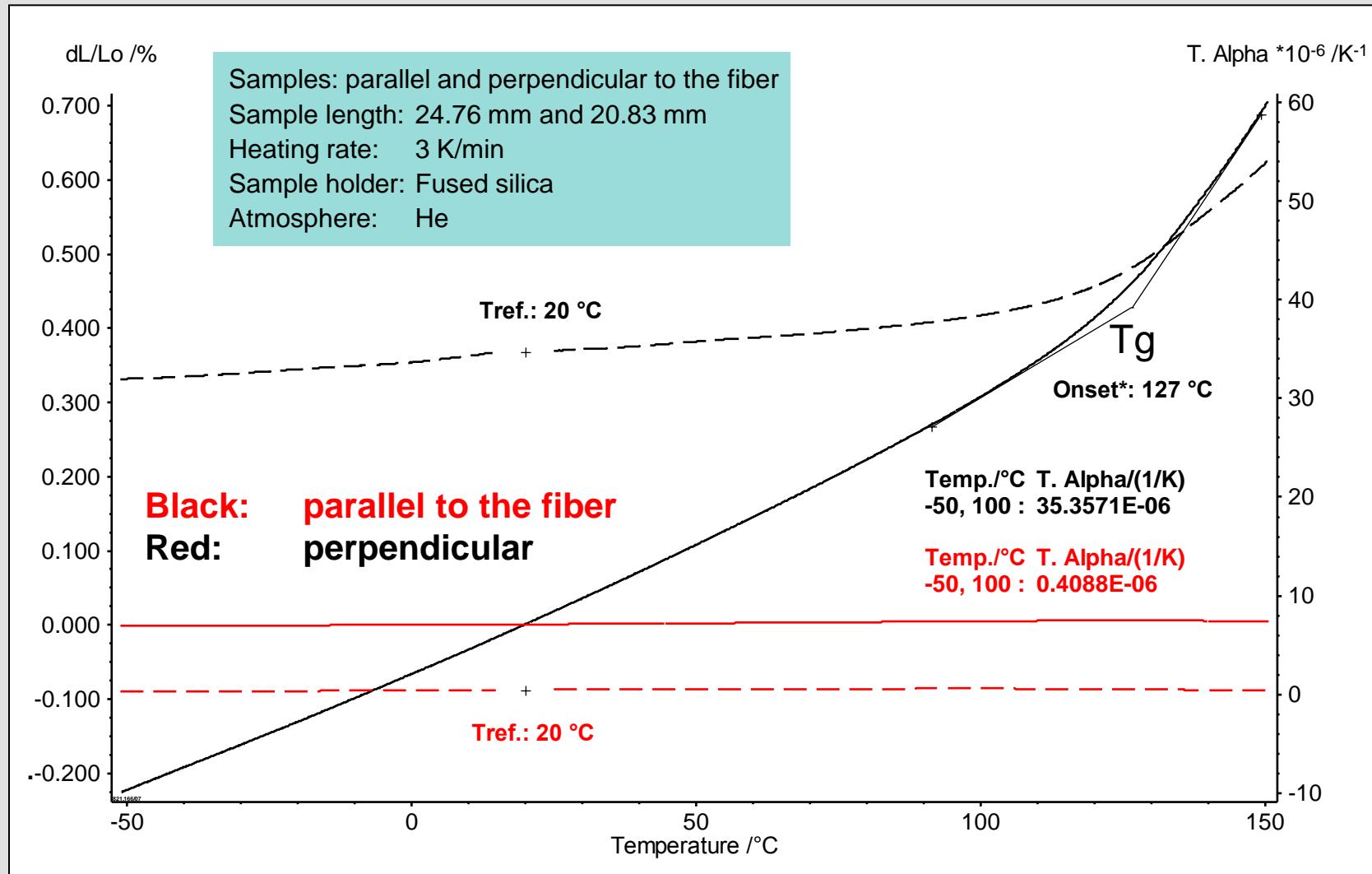
Quartz glass  
-150 ... 1000°C

$\text{Al}_2\text{O}_3$  RT ... 1550°C



# Thermal Expansion of CFRP: Anisotropy Parallel and Perpendicular to the Fiber Orientation

**NETZSCH**



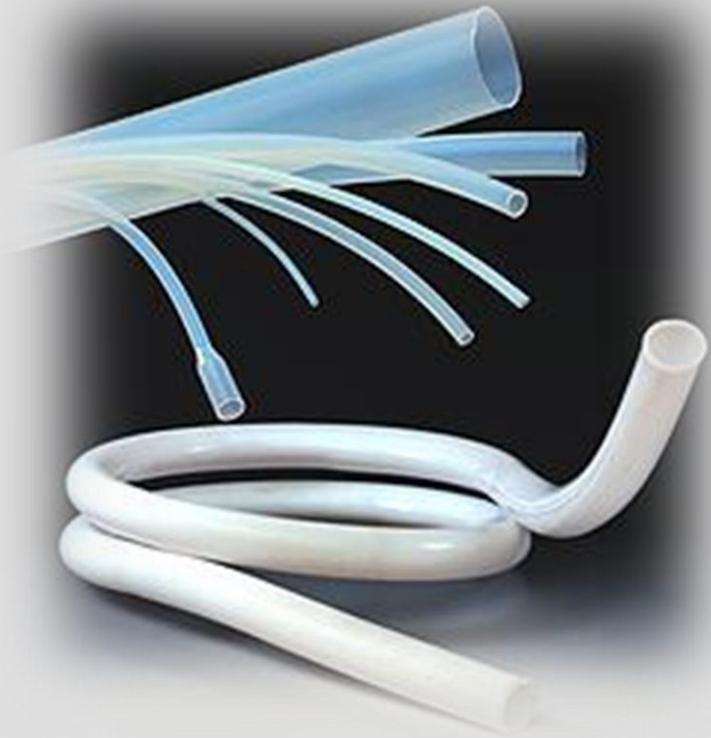


## Technical Data

- Wide temperature range (-170°C ... 600°C)
- Robust design
- Rectangular furnace
- Versatile sample holders
- Large sample geometries
- High dynamic and static forces (up to 24 N)
- High amplitude resolution (up to 0.5 nm)
- Immersion and humid atmosphere tests
- Fourier Analysis
- Comprehensive calibration routines
- User-friendly *Proteus*® software

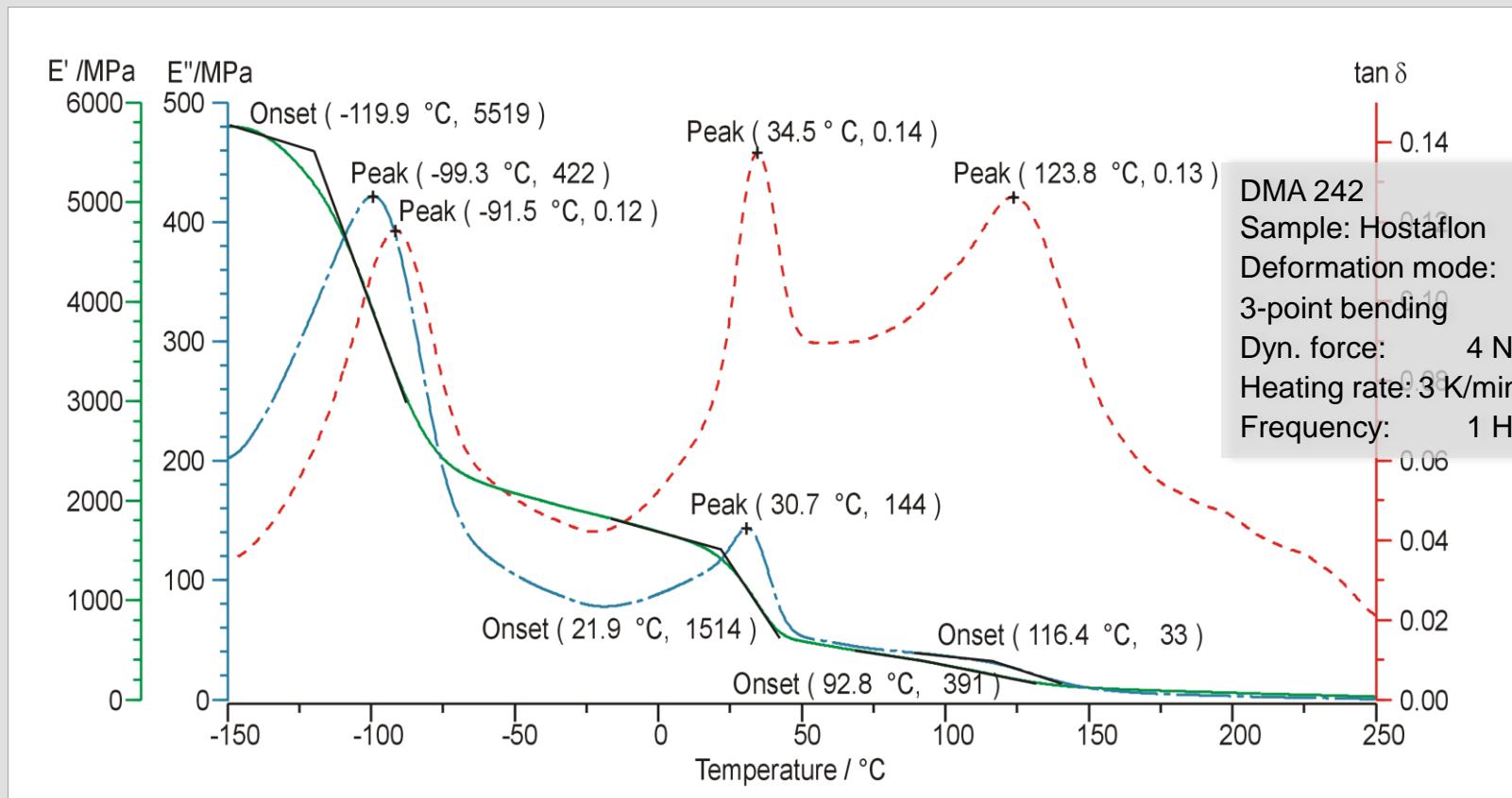
# Application: PTFE

**NETZSCH**



# Friction Ring of PTFE (Polytetrafluoroethylene)

**NETZSCH**



- PTFE clearly shows three transitions in a wide temperature range before the material starts melting.
- PTFE can be used as an internal standard material for temperature and stiffness ( $E'$ )

# The NETZSCH Product Range – Optimized Instruments with Outstanding Features

**NETZSCH**

## Materials Characterization by Thermal Analysis Methods

Differential Thermal Analysis, Differential Scanning Calorimetry (DTA/DSC)

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DSC 3500 *Sirius*  
DSC 214 *Polyma*  
DSC 204 **F1**  
DSC 204 **F1** Photo  
DSC 204 HP  
DSC 404 **F1**  
DSC 404 **F3**  
-180 ... 2400°C

TG 209 **F3**  
TG 209 **F1**  
STA 449 **F3**  
STA 449 **F1**  
STA 449 **F5**  
STA 2500 *Regulus*  
STA 409 CD  
STA 429 CD  
-150 ... 2400°C

DIL 402 PC  
DIL 402 C  
DIL 402 CD  
DIL 402 E/Pyro  
TMA 402  
DMA 242 E  
-260 ... 2800°C

HFM 436  
GHP 456  
TCT 426  
LFA 467  
LFA 457  
LFA 427  
SBA 458 *Nemesis®*  
-125 ... 2800°C

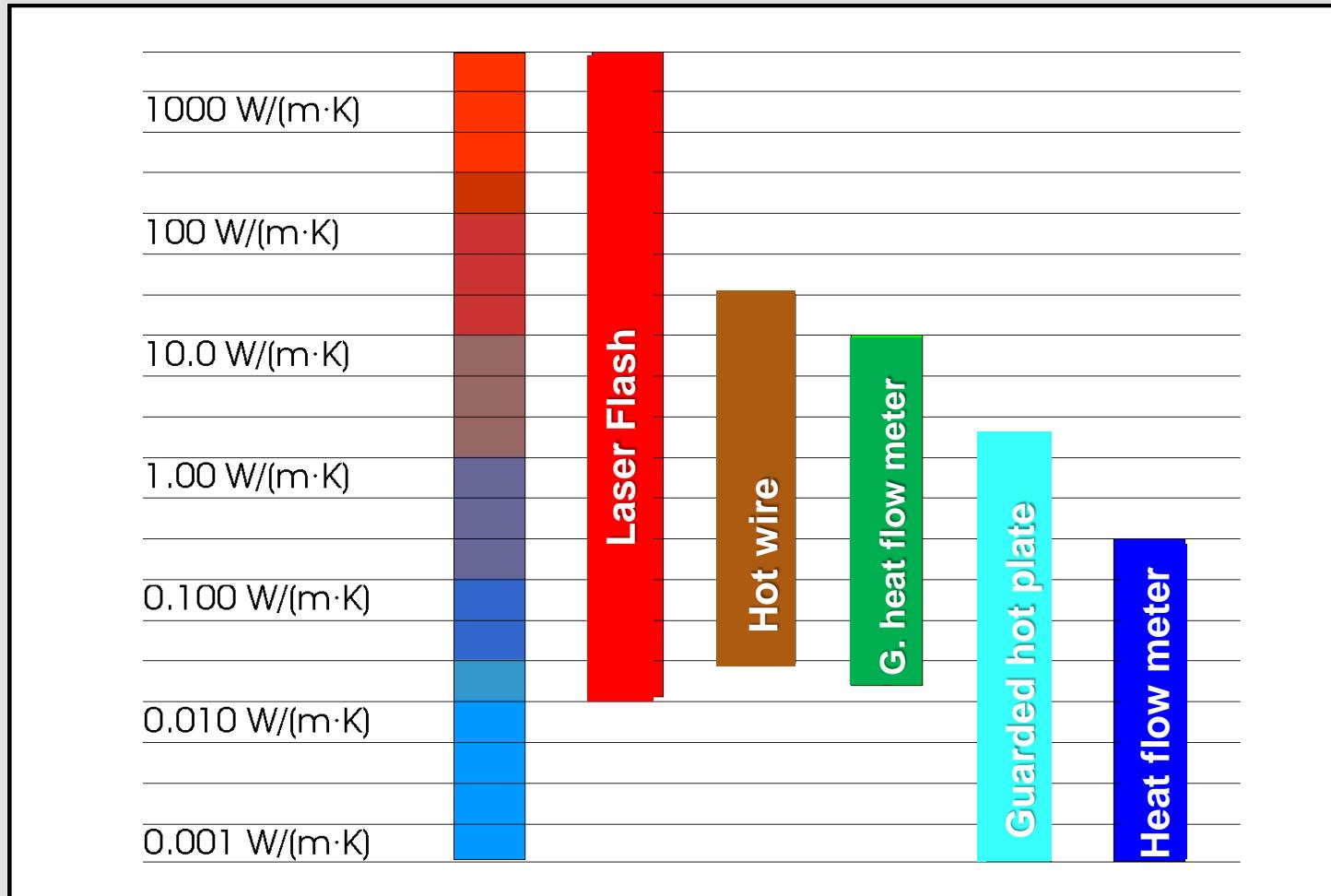
ARC® 244  
ARC® 254  
APTAC™ 264  
MMC 274 *Nexus®*  
IBC 284  
RT ... 500°C

DEA 288  
RT ... 375°C

Evolved Gas Analysis (EGA) for the advanced characterization of decomposition/evaporation effects  
QMS 403 *Aëolos®*, STA-MS-Skimmer®, FT-IR, GC-MS

# Thermal Conductivity – Measurement Methods

**NETZSCH**



# GHP 456 *Titan*<sup>®</sup> and HFM 436 Design

**NETZSCH**

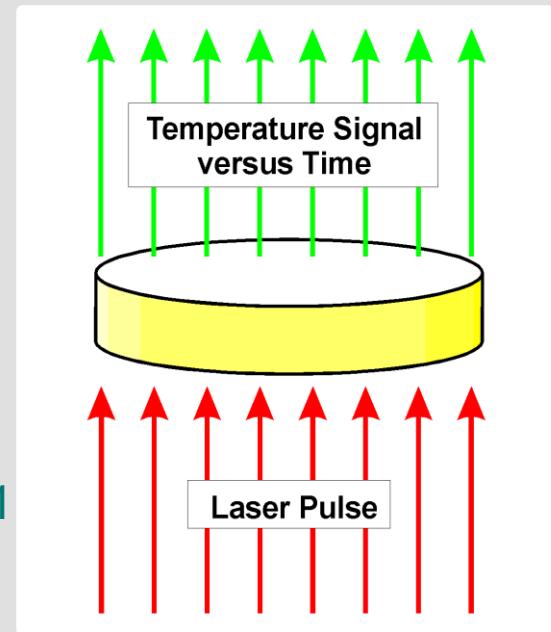


Thermal Conductivity measurements for the large samples – **insulation materials**

## Flash Method:

- The front surface of a plan-parallel sample is heated by a short light or laser pulse.
- The temperature rise on the rear surface is measured versus time using an IR detector.

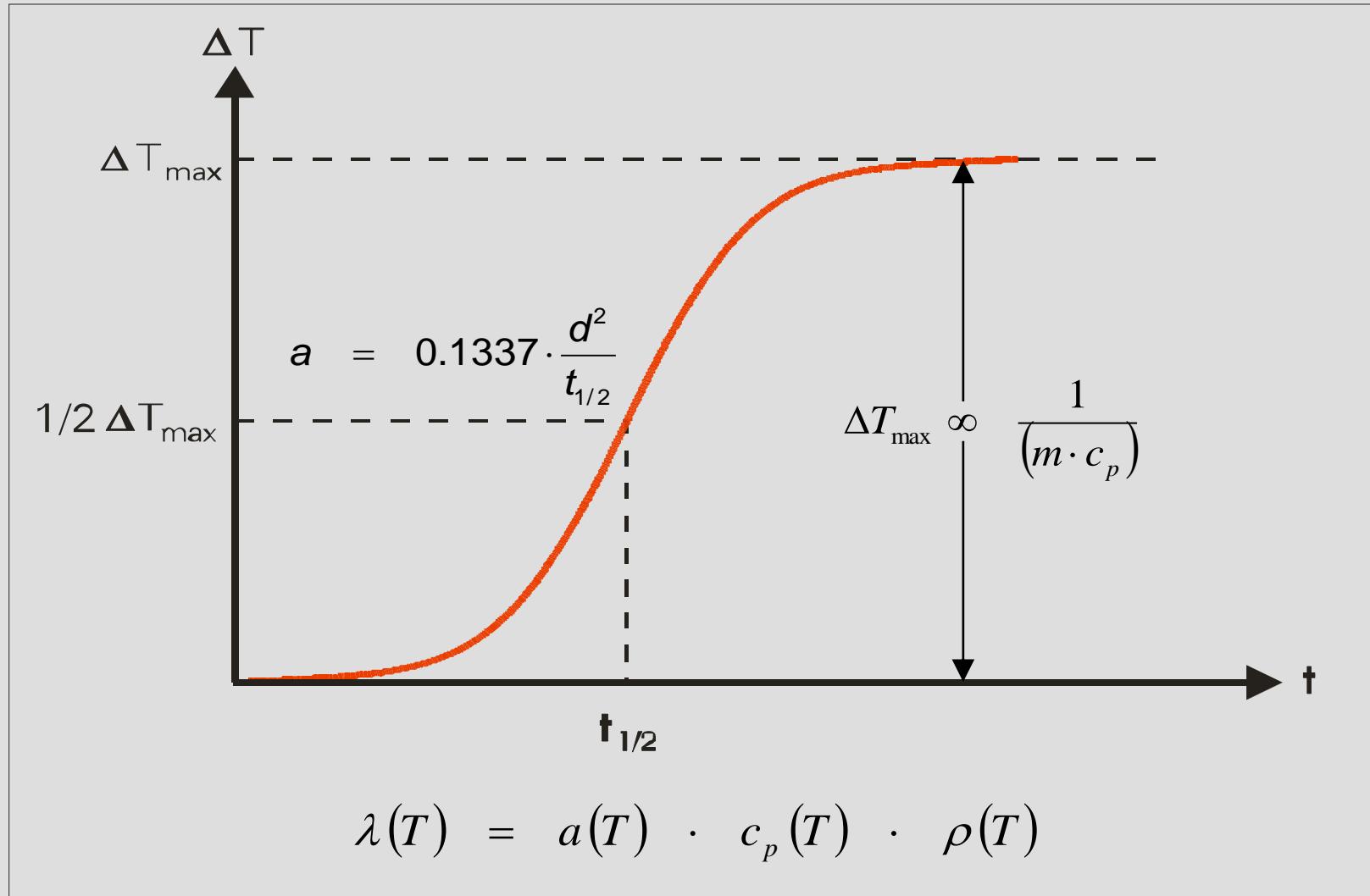
Measurement principle introduced by Parker et al. 1961



The sample has been stabilized at the desired temperature, the laser fired several times over a span of a few minutes and the necessary data is recorded for each laser „shot“.

The laser beam energy strikes and is absorbed by the front surface of the sample, causing a heat pulse to travel through the samples' thickness.

The resulting sample temperature rise is fairly small, ranging from 0.5°C to 2°C. The temperature rise is amplified and recorded.



# NETZSCH LFA Systems

**NETZSCH**



LFA 447 *NanoFlash*®  
for MTX Applications



LFA 467 *HyperFlash*®

-100°C ... 500°C



LFA 457 *MicroFlash*®

-125°C ... 1100°C



LFA 427

-70°C ... 2800°C

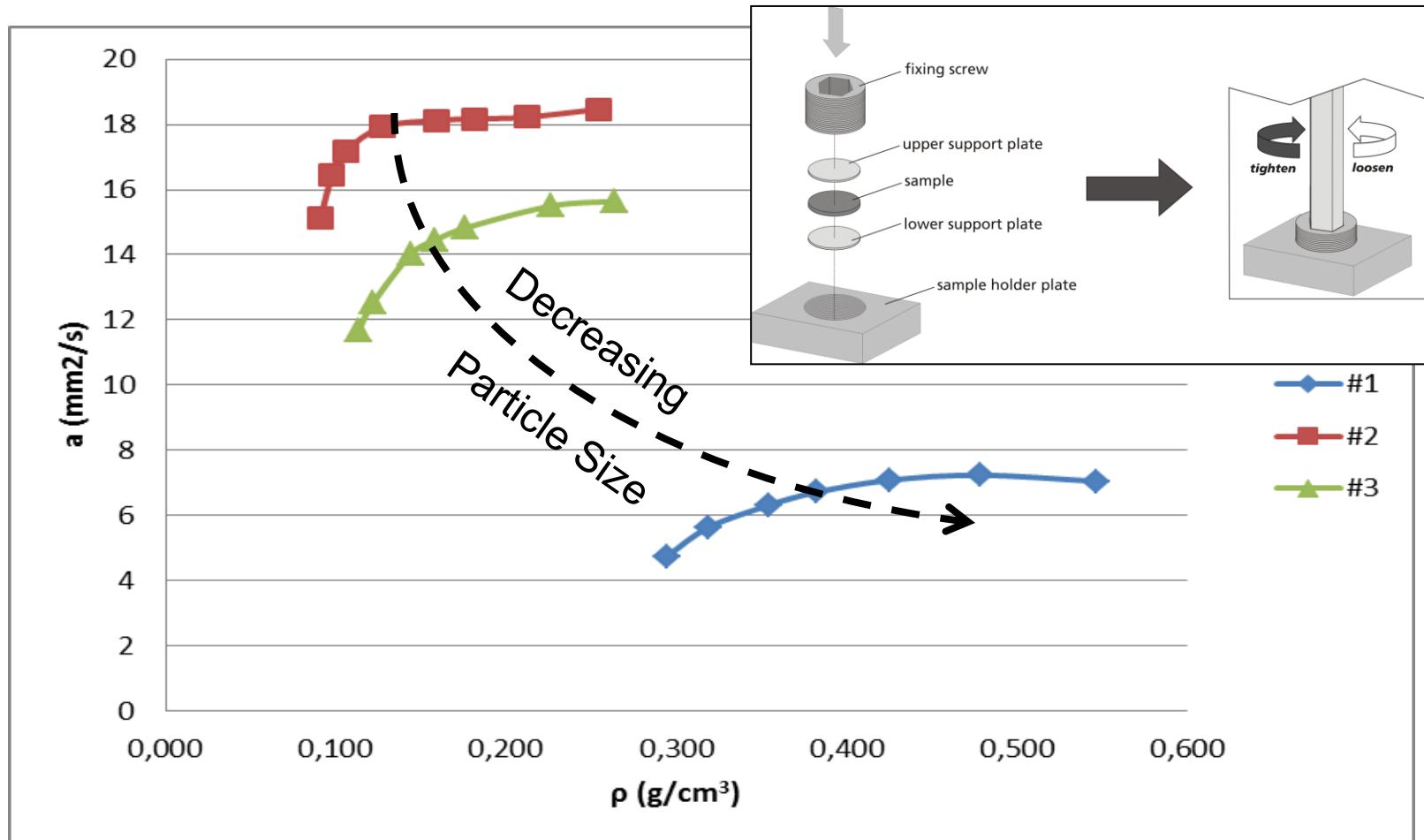
# Key Advantages of Netzscht LFA Systems

**NETZSCH**

## Pressure sample holder for compressible samples

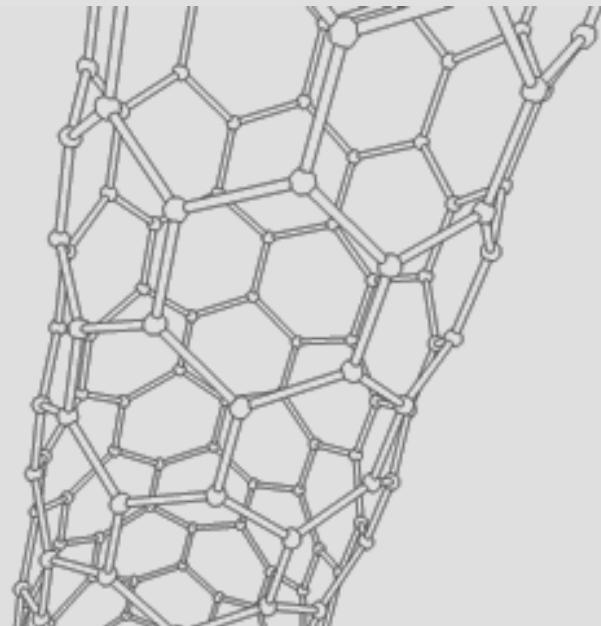
- CNT Powders with different particle sizes

### Pressure Sample Holder

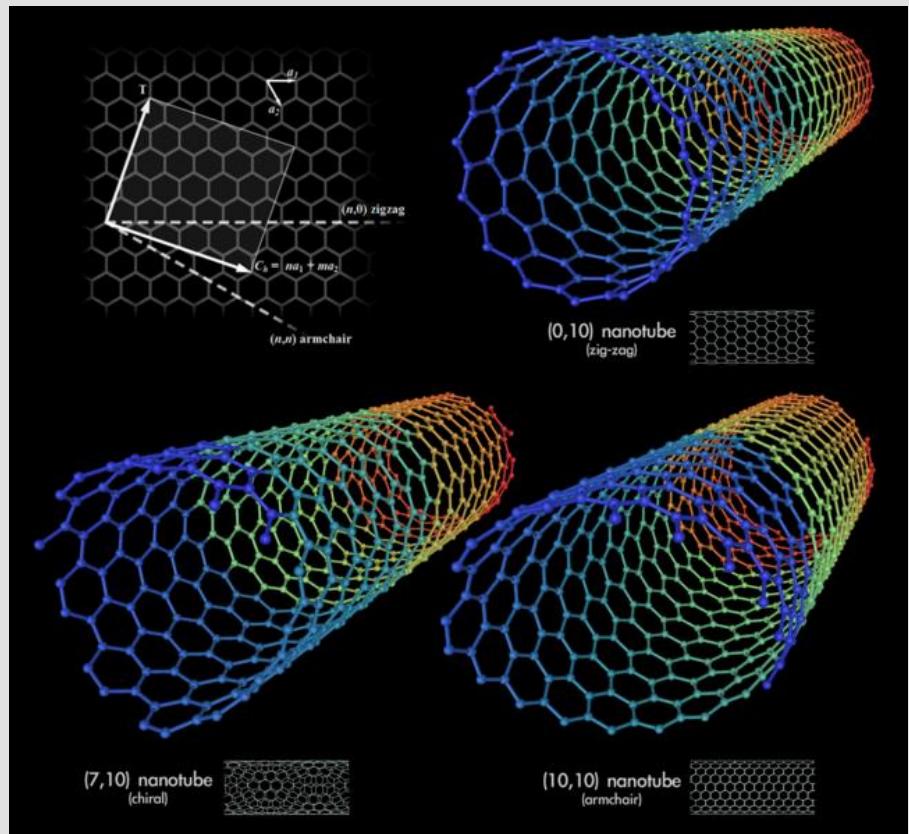


# Application – PEEK filled with CNT

**NETZSCH**



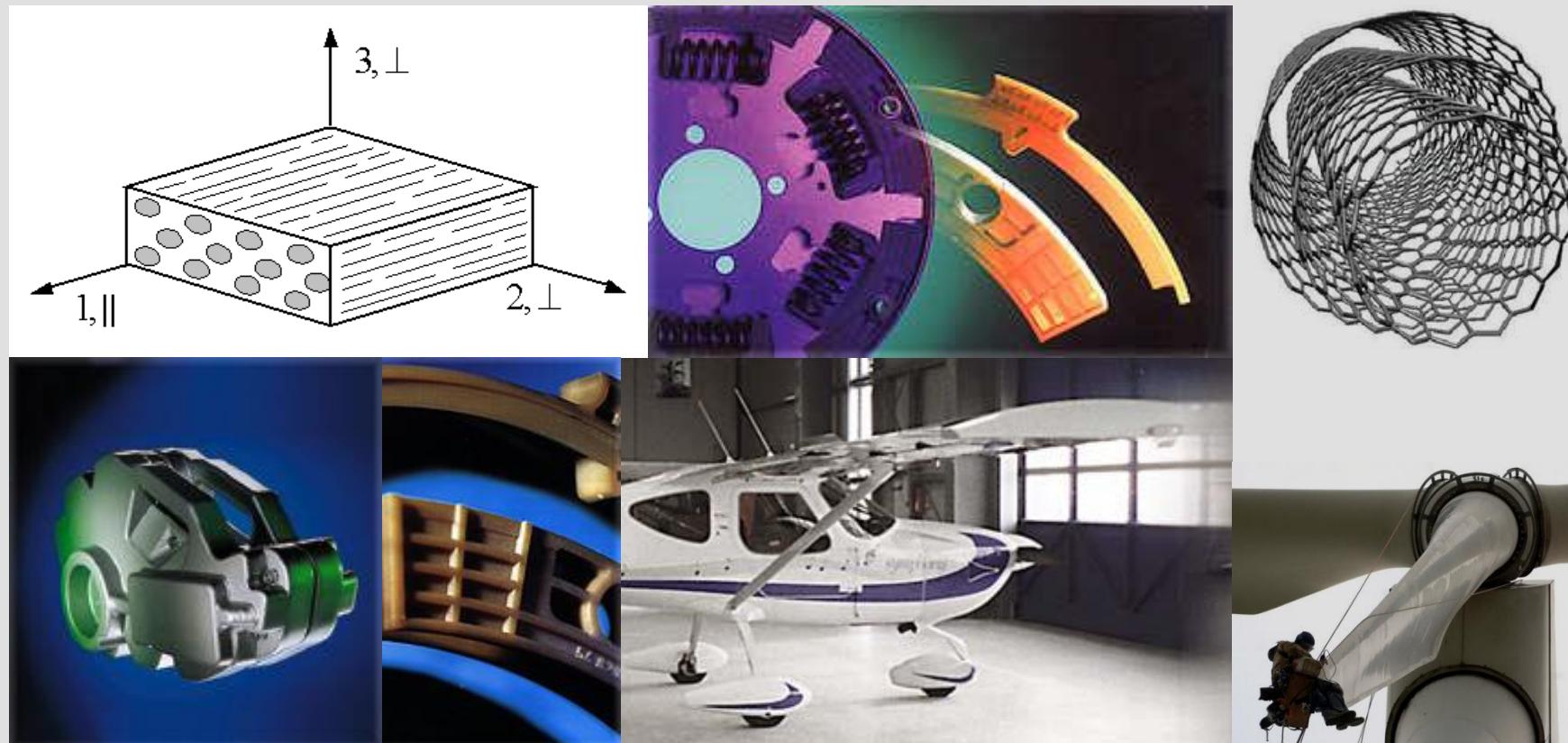
Prepared in Cooperation with:



# PEEK filled with CNT – Anisotropy

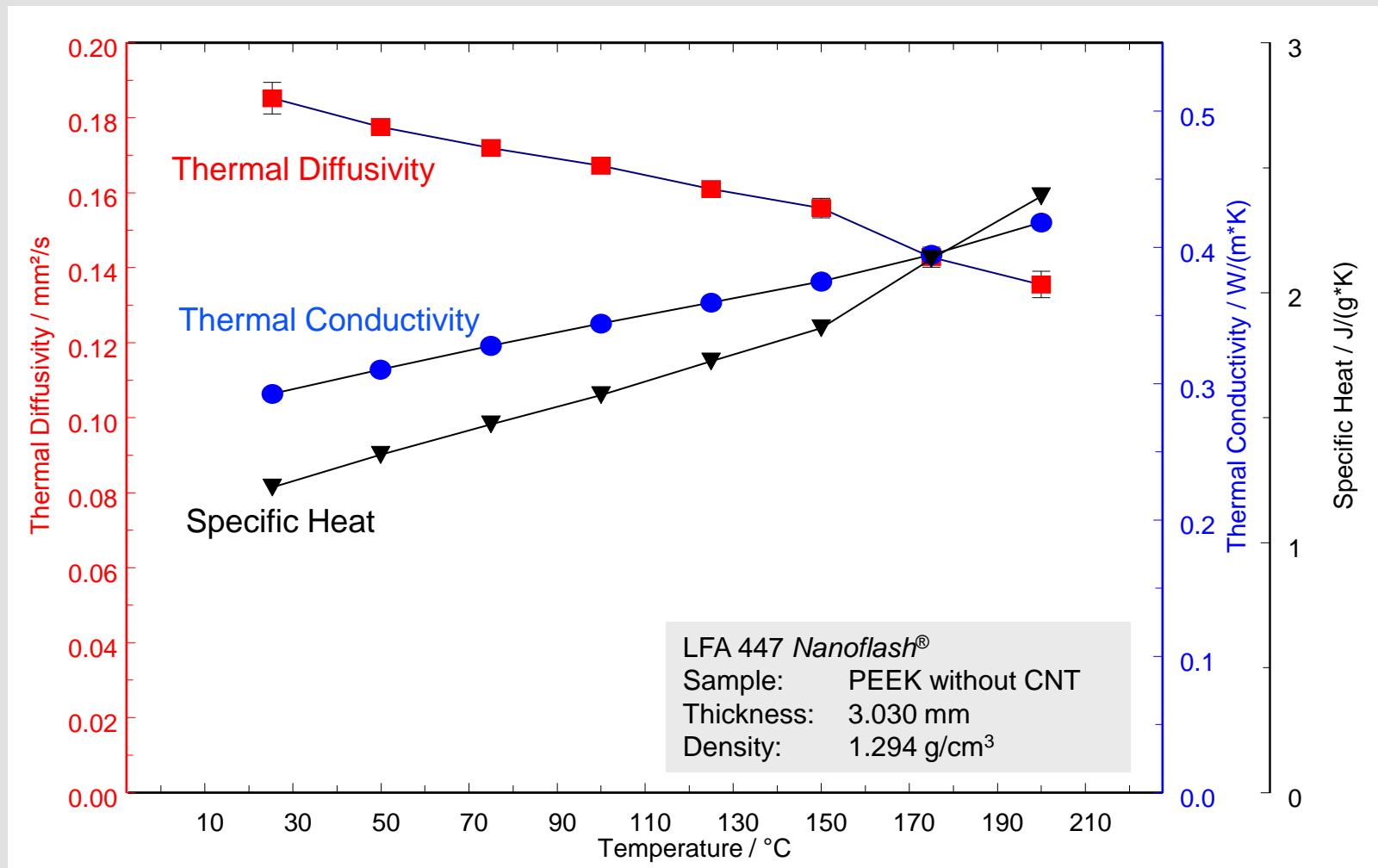
**NETZSCH**

→ Orientation of carbon nanotubes due to injection moulding



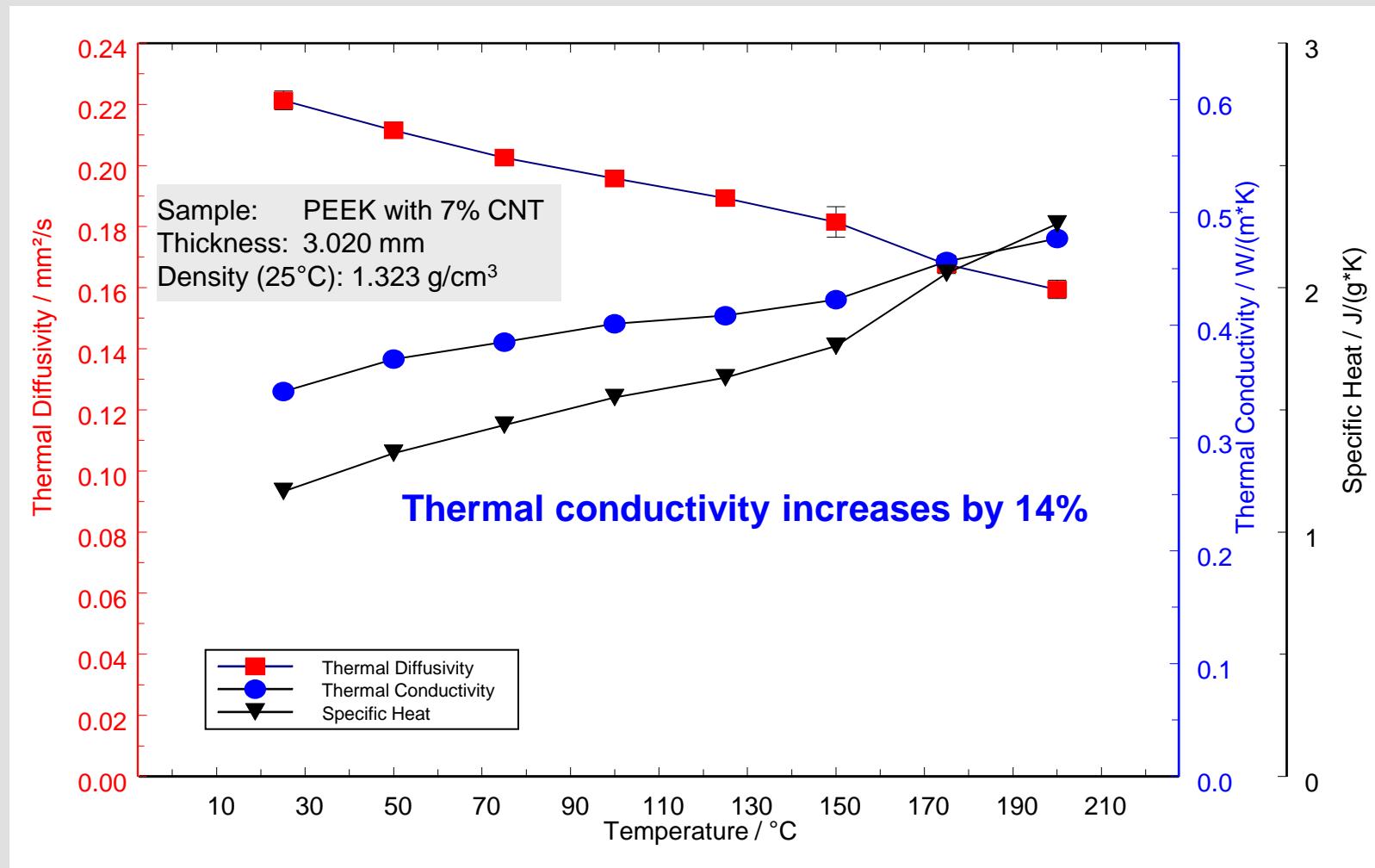
# Application – PEEK filled with CNT 0% - Pure PEEK Matrix

NETZSCH



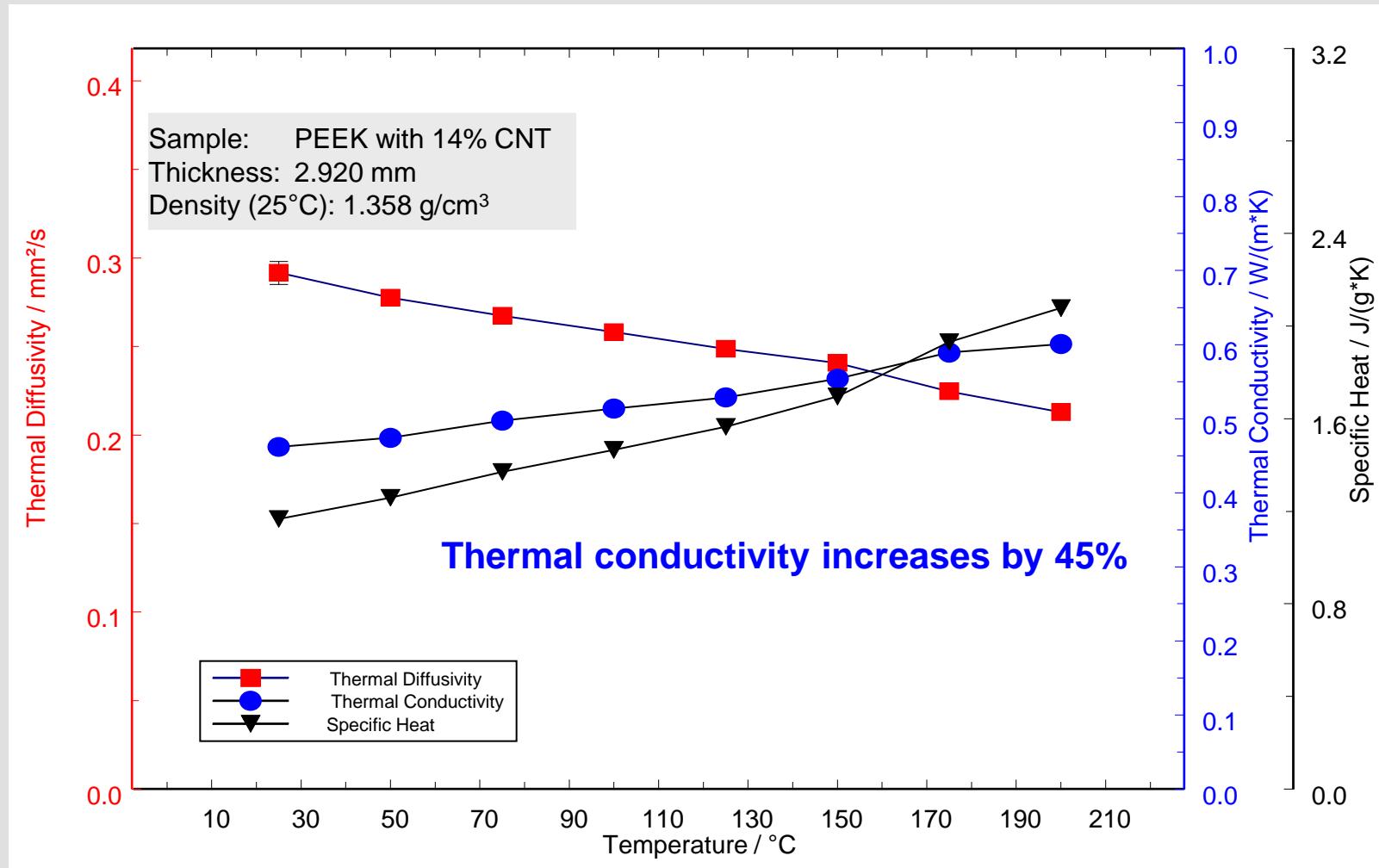
# PEEK Filled with 7% CNT

**NETZSCH**



# PEEK Filled with 14% CNT

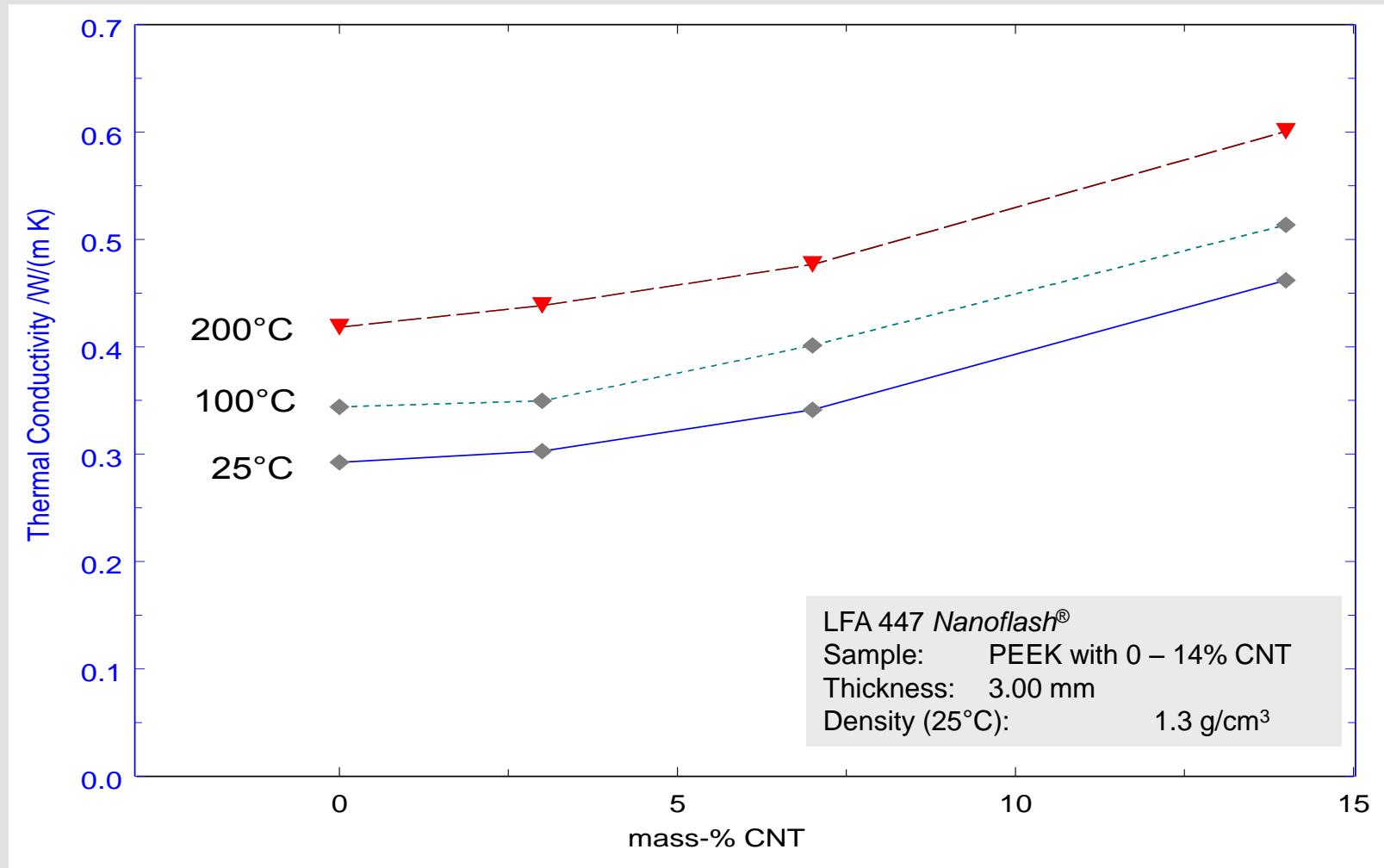
**NETZSCH**



# Application – PEEK filled with CNT

## PEEK with CNT – Thermal Conductivity

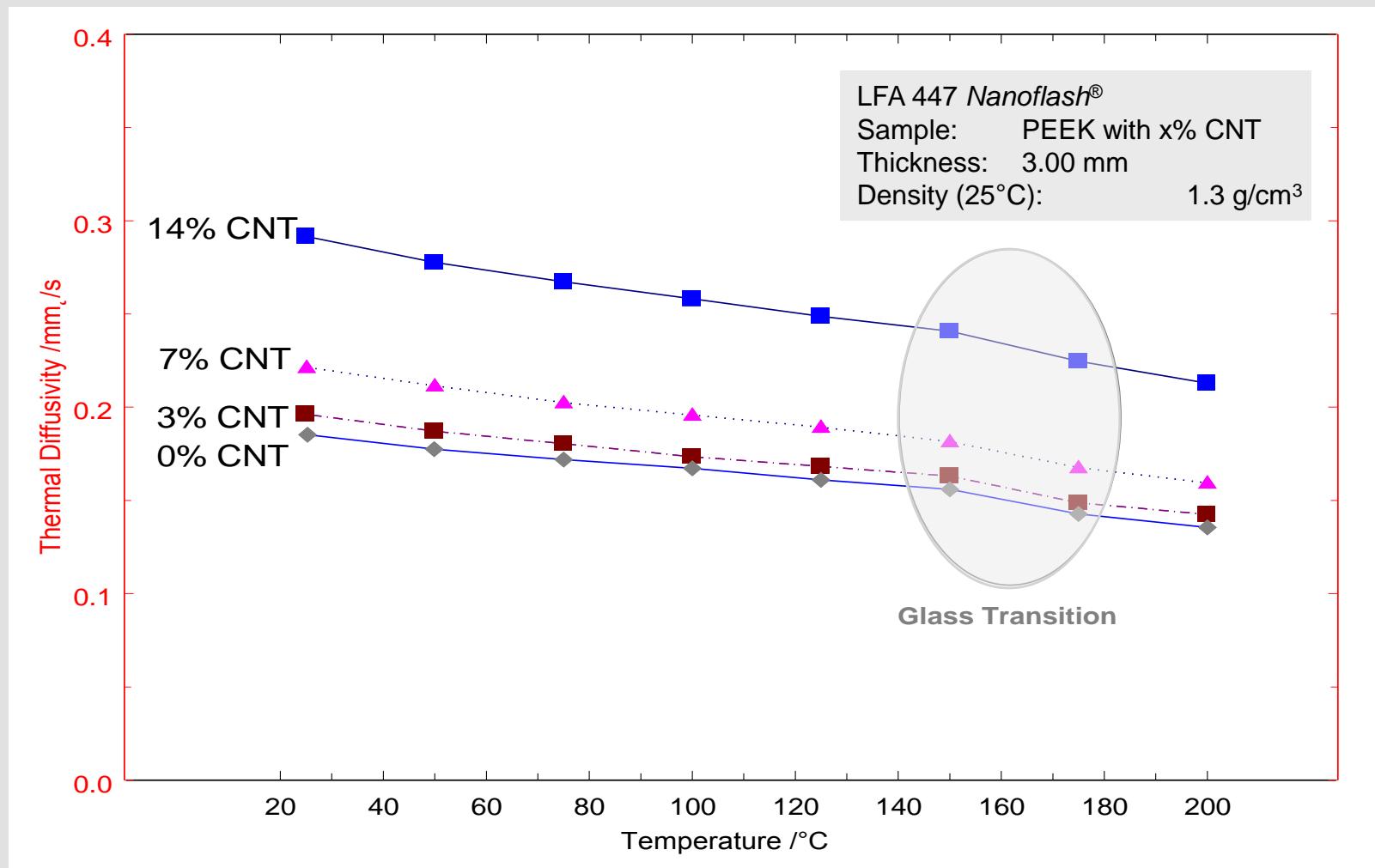
NETZSCH



# Application – PEEK filled with CNT

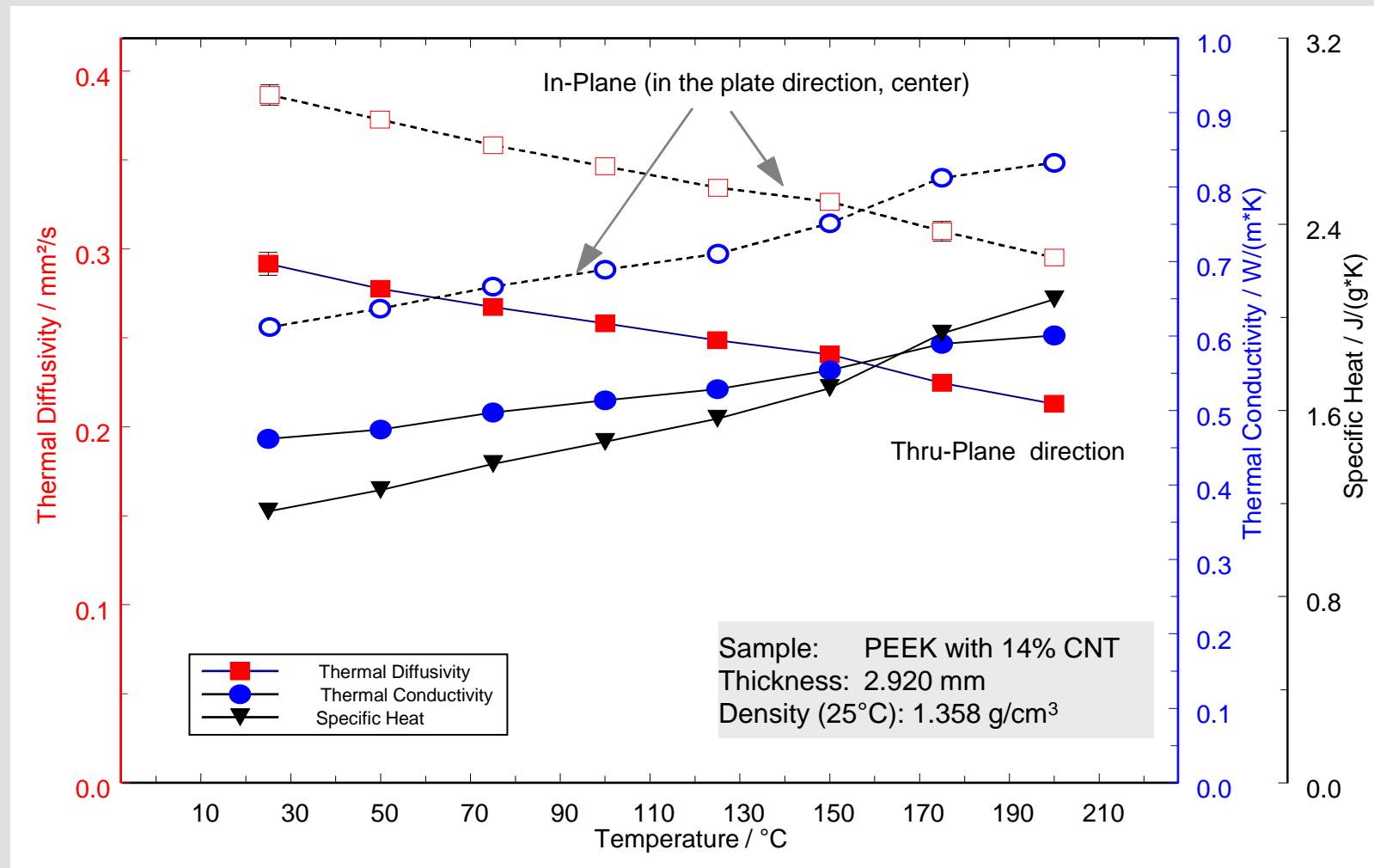
## PEEK with CNT – Thermal Diffusivity

NETZSCH



# PEEK with CNT – Anisotropy

**NETZSCH**



### Technical Data



- Design: Horizontal sample arrangement
- Heater: Two-heater system, integrated quality check
- **Temperature range: RT to 800°C**
- Thermocouples: Inconel®-sheathed type K; fixed position
- Sample dimensions:  
Ø: 12.7 to 25.4 mm; length: 12.7 to 25.4 mm;  
width: 2.0 to 25.4 mm, thickness: ≤ 2 mm
- Sample geometries: Square, rectangular, round, strips
- Seebeck coefficient range: 10 to 500  $\mu\text{V/K}$ ,  
accuracy < 10%; repeatability < 3%
- Electrical conductivity range: 5 to 150000 S/cm,  
accuracy < 10%, repeatability < 7%
- **Atmosphere: Reducing (max. 2% H<sub>2</sub>), inert, oxidizing**
- Vacuum-tightness: 10<sup>-2</sup> mbar

# Smart Solutions in Thermal Analysis

**NETZSCH**

Thank You for Your Attention



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DSC 204 **F1** Photo  
DSC 204 HP  
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DSC 404 **F3**  
-180 ... 2400°C

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TG 209 **F1**  
STA 449 **F3**  
STA 449 **F1**  
STA 449 **F5**  
STA 2500 *Regulus*  
STA 409 CD  
STA 429 CD  
-150 ... 2400°C

DIL 402 PC  
DIL 402 C  
DIL 402 CD  
DIL 402 E/Pyro  
TMA 402  
DMA 242 E  
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HFM 436  
GHP 456  
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ARC® 244  
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APTAC™ 264  
MMC 274 *Nexus*  
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RT ... 500°C

DEA 288  
RT ... 375°C

Evolved Gas Analysis (EGA) for the advanced characterization of decomposition/evaporation effects  
QMS 403 *Aëolos*®, STA-MS-Skimmer®, FT-IR, GC-MS



**The MMC 274 Nexus® is a Table-Top Adiabatic Calorimeter for small sample sizes**

## Technical data

- Sample volume: up to 3 ml
- Temperature range: RT ... 500°C
- Thermal environment: defined; adiabatic environment for Thermal Runaway Reactions
- Pressure changes: automatic detection
- Transitions: temperatures and reaction heats, exo- and endothermal effects
- Specific heat: solids and liquids
- Coin cell module for battery testing

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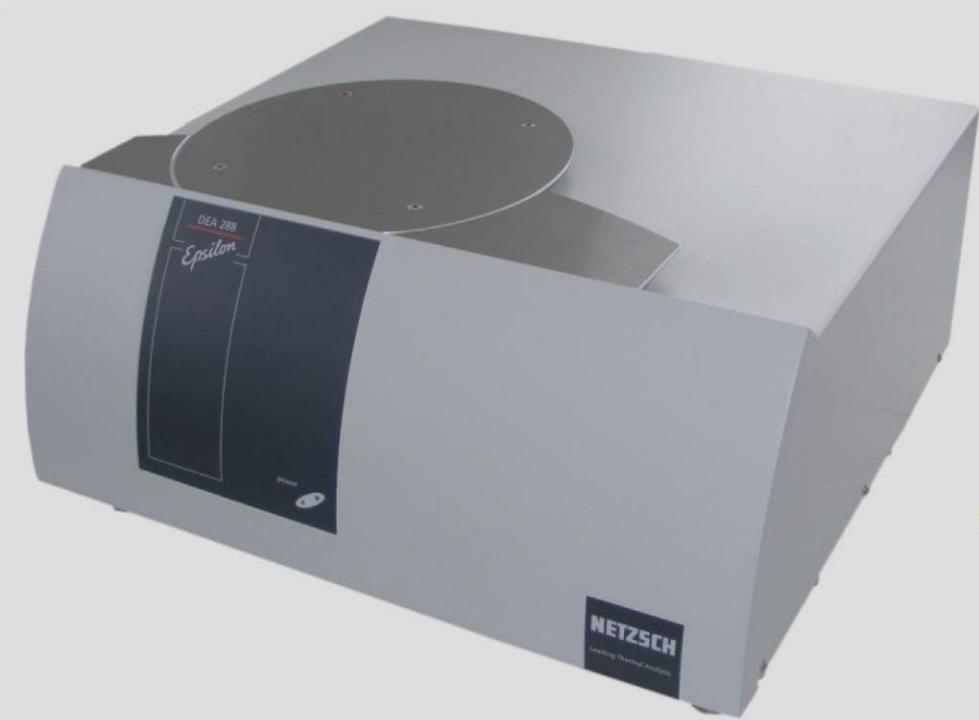
ARC® 244  
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Evolved Gas Analysis (EGA) for the advanced characterization of decomposition/evaporation effects  
QMS 403 *Aëolos*®, STA-MS-Skimmer®, FT-IR, GC-MS

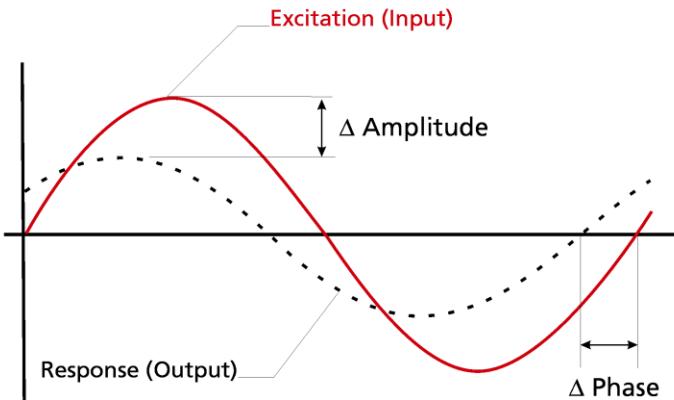
# DEA 288 *Epsilon*<sup>®</sup> – The Ideal Tool for Understanding the Curing Process

**NETZSCH**



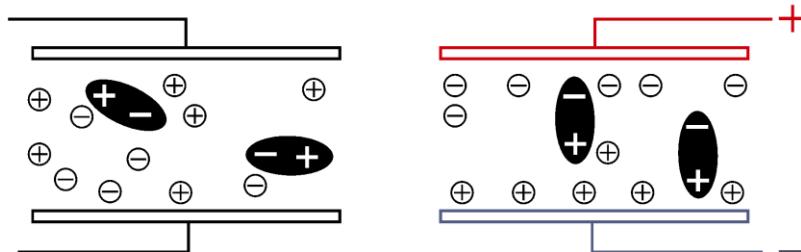
# Fundamental Principle of DEA

**NETZSCH**



A low voltage AC signal (input) is applied at one electrode

The response signal detected at the other electrode (output) is attenuated and phase shifted



Dipolar and Ionic Behavior

Dielectric sensor:

- Alignment of dipoles
- Mobility of ions

$$C = \epsilon_r C_0$$

$$\epsilon_r = \epsilon_r' - i \epsilon_r''$$

# UV Curing of a Flexible Sealant Based on Epoxy for Protection of Organic LEDs and Photovoltaics

**NETZSCH**

Ion visc. / $\text{Ohm}^*\text{cm}$

*The increase in ion viscosity describes the curing process*

