Analyzing & Testing
Business Unit

NETZSCH

LFA for Thermal Diffusivity and Conductivity of Metals, Ceramics and Polymers

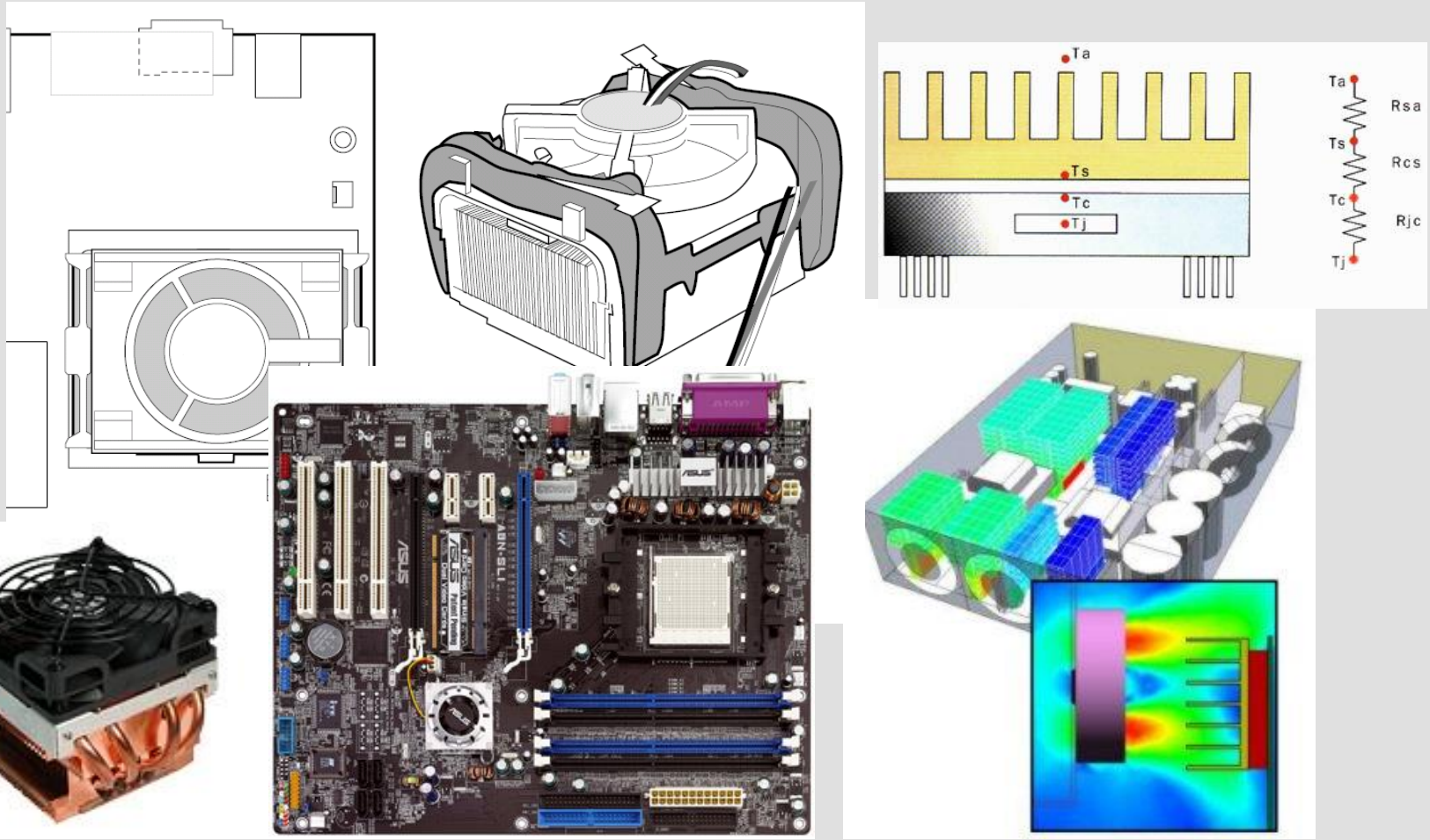
Ramón Arauz Lombardia, Service & Applications
NETZSCH-Gerätebau GmbH, Branch Office Barcelona, Spain

Agenda

1. Heat Transfer - Basics
2. LFA - Method
3. LFA - Instrumentation
4. Application Examples
5. Summary

Examples of Heat Transfer From Everyday Life

..cooling the microprocessor chip in a laptop computer



Heat Transfer – Electronics

Examples of Heat Transfer From Everyday Life

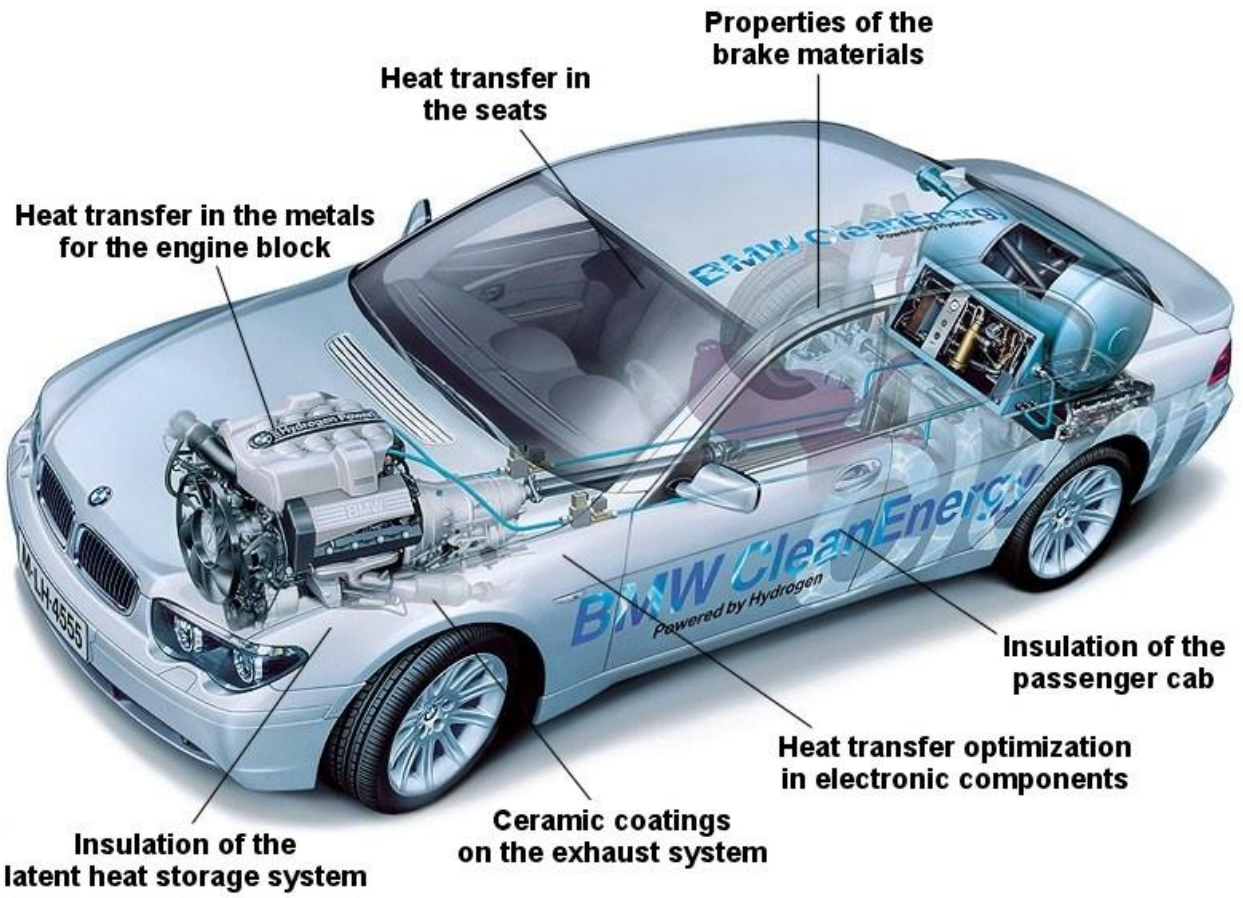


Photo: BMW AG – Photo-Gallery: 3 Series

Heat Transfer - Basics

Jean Baptiste Joseph Fourier

Heat transfer (or heat) is the energy in transit (energy moving through a material) due to a temperature difference.

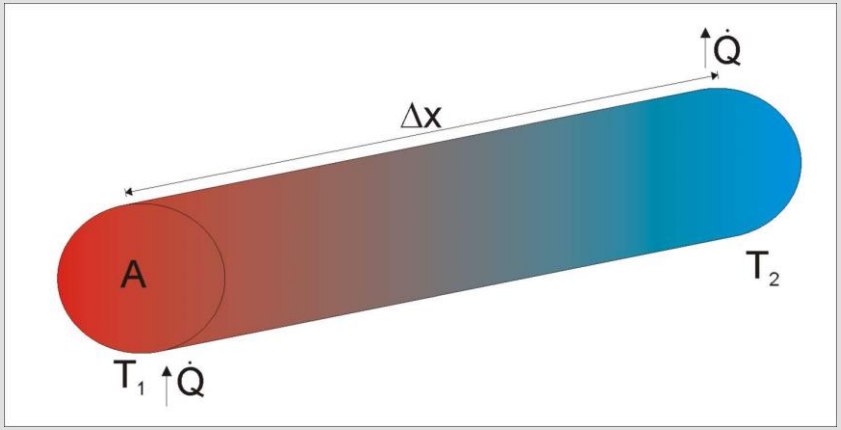
A quantitative expression relating the rate of heat transfer, the temperature gradient and the nature of the conducting medium is attributed to Fourier (1822):

$$\dot{Q} = -\lambda A \frac{T_2 - T_1}{\Delta x} \quad ; \quad \dot{q} = -\lambda \frac{\Delta T}{\Delta x}$$

A – area normal to heat flow

Heat flux = heat flow rate per unit area in direction x

λ - thermal conductivity of material



Heat Transfer - Basics

There are three main modes of **heat transfer**:

- Conduction: molecular interactions or free electrons
- Convection: exchange of heat between a fluid and a surface or an interface.
- Radiation: no medium required, electromagnetic phenomenon

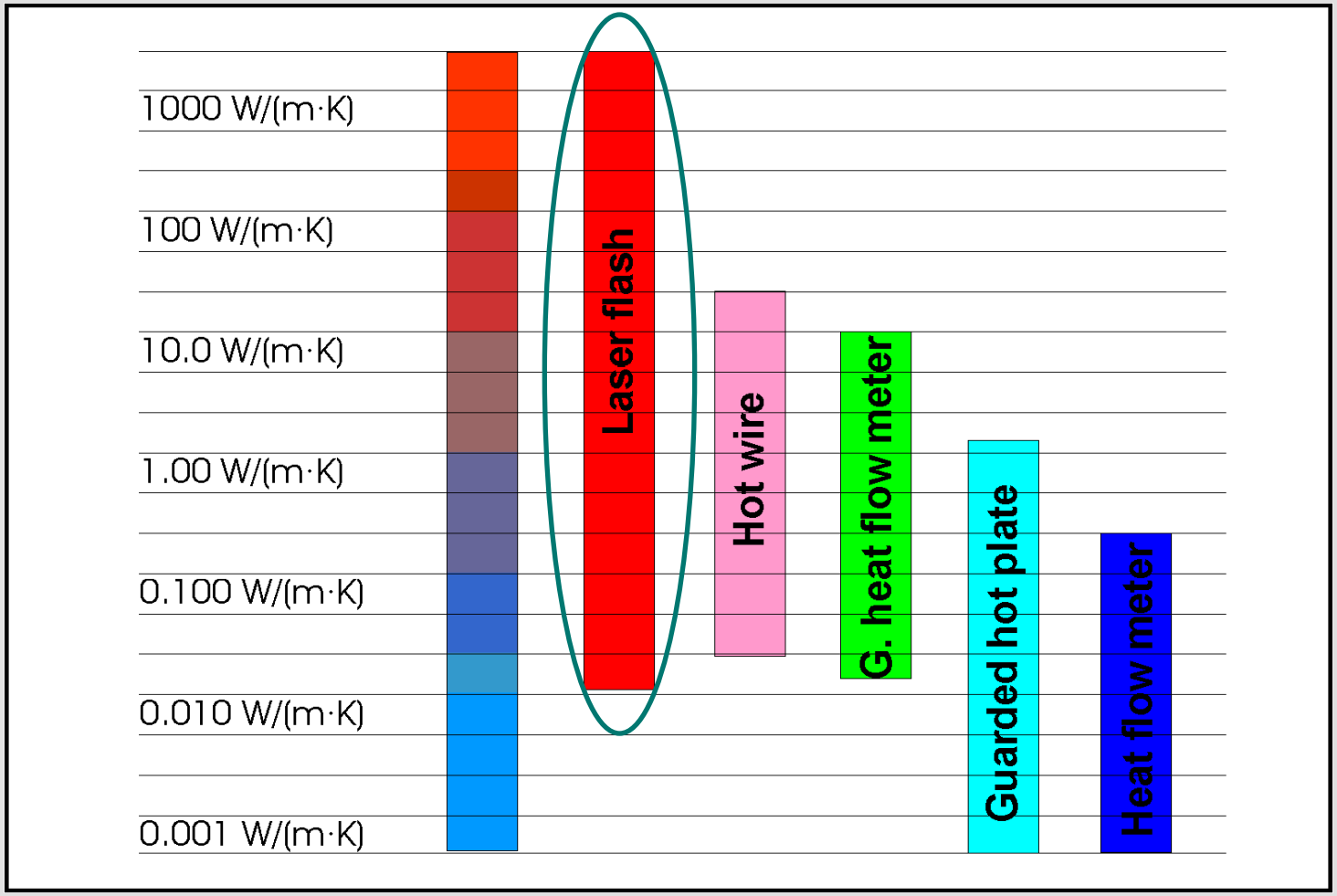
Stefan-Boltzmann constant

$$\frac{q}{A} = \sigma T^4$$

Thermal conductance varies for each mode of heat transfer

Heat Transfer
Basics

Thermal Conductivity
Measurement Methods



Agenda

1. Heat Transfer - Basics
2. LFA - Method
3. LFA - Instrumentation
4. Application Examples
5. Summary

Heat Transfer – LFA Method

The measurement of the **thermal diffusivity** of a material is usually carried out by rapidly heating one side of a sample and measuring the temperature rise curve on the opposite side.

The time that it takes for the heat to travel through the sample and cause the temperature to rise on the rear face can be used to measure the through-plane diffusivity and calculate the through-plane thermal conductivity if the specific heat and density are known.

All three quantities as well as the thermal diffusivity can be functions of temperature.

$$\lambda(T) = a(T) \cdot c_p(T) \cdot \rho(T)$$

Heat Transfer – LFA Method

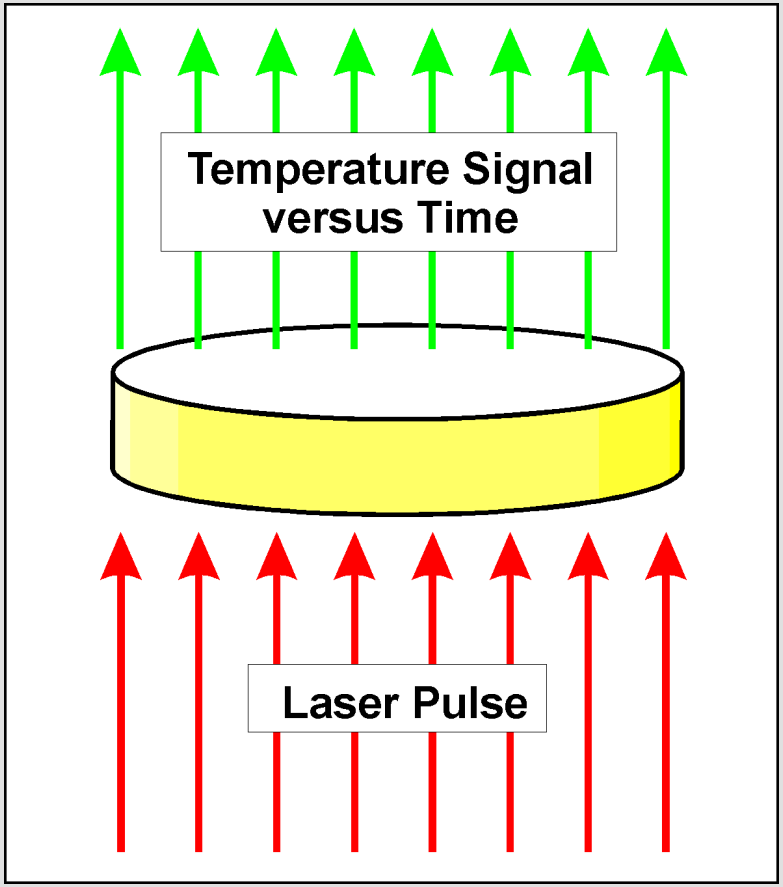
NETZSCH

Flash Method:

- Measurement Principle
  Introduced by Parker et al. 1961

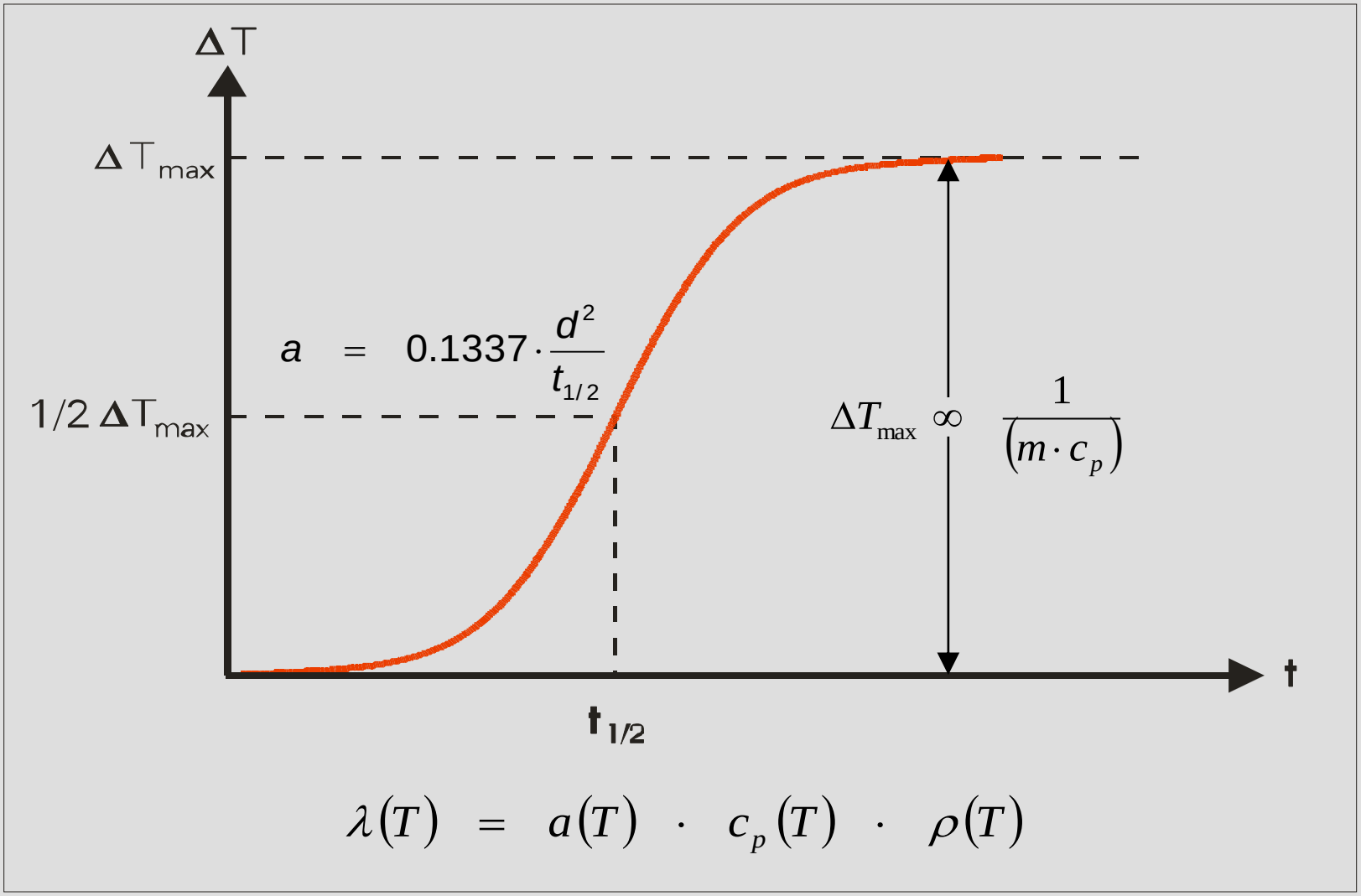
The front surface of a plan-parallel sample is heated by a short light or laser pulse.

The temperature rise on the rear surface is measured versus time using an IR detector.



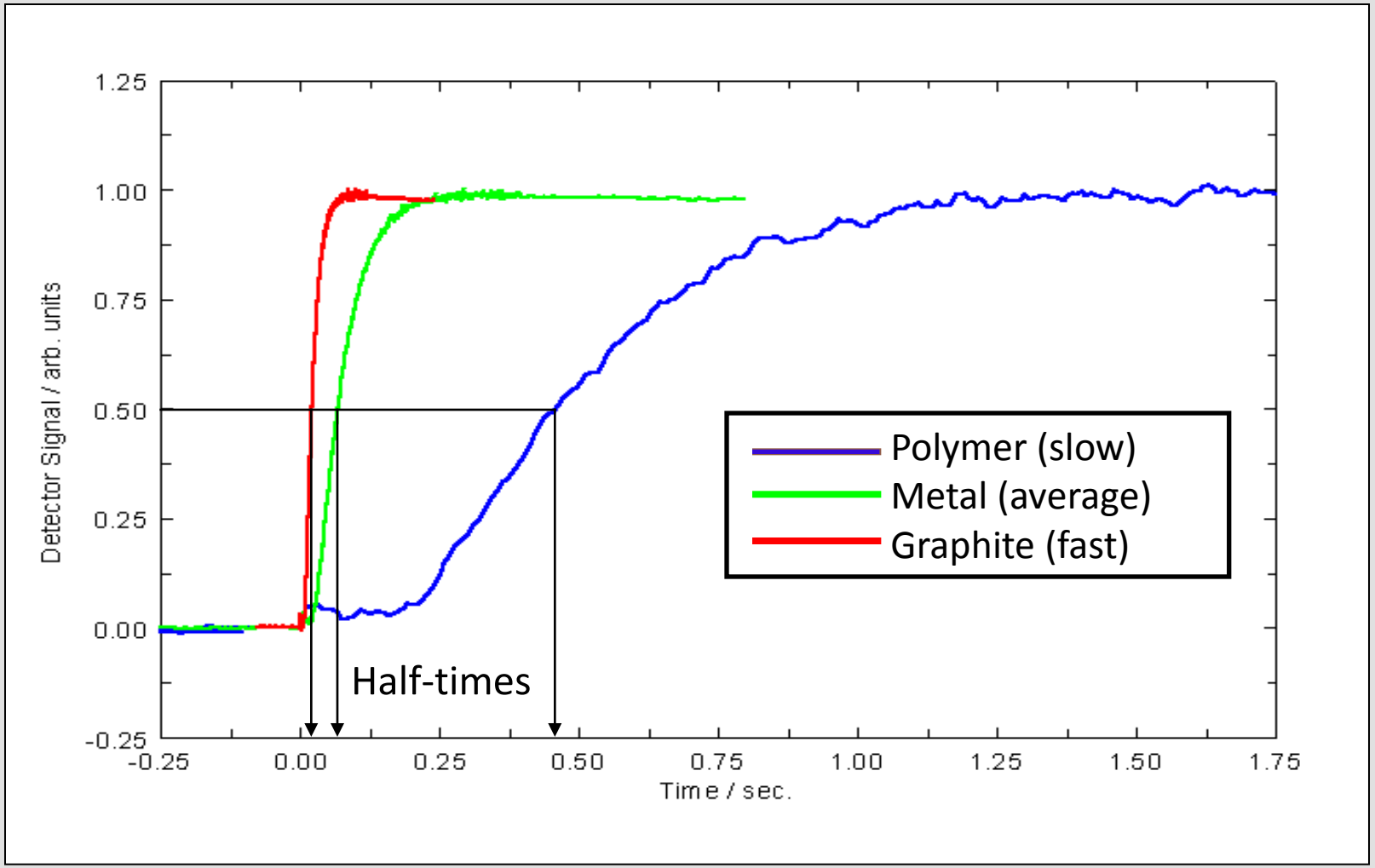
The sample has been stabilized at the desired temperature, the laser fired several times over a span of a few minutes and the necessary data is recorded for each laser „shot“. The laser beam energy strikes and is absorbed by the front surface of the sample, causing a heat pulse to travel through the samples‘ thickness. The resulting sample temperature rise is fairly small, ranging from 0.5 to 2 °C. The temperature rise is amplified and recorded.

Heat Transfer – LFA Method



$$\lambda(T) = a(T) \cdot c_p(T) \cdot \rho(T)$$

Heat Transfer: LFA Method



Agenda

1. Heat Transfer - Basics
2. LFA - Method
3. LFA - Instrumentation
4. Application Examples
5. Summary

Flash Technique – Introduction

Netzsch LFA Systems for almost all application ranges

LFA 467 *HyperFlash*

- -100°C to 500°C
- Sample changer for 16 samples
- *ZoomOptics* - IR detector
- Fastest data acquisition

LFA 457 *MicroFlash*®

- -125°C to 1100°C
- Sample changer for 3 samples

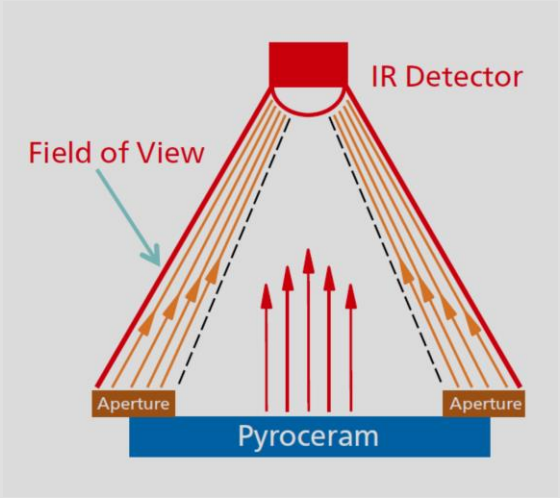
LFA 427

- -120°C to 2800°C

NETZSCH LFA 467 *HyperFlash*

- Table-top instrument
- Works with a high power Xenon-Flash lamp
- Temperature range: -100 to 500°C
- Integrated sample changer for 16 samples
- Different cooling options
- *ZoomOptics* for optimized field of view

NETZSCH LFA 467 *HyperFlash – ZoomOptics*



Field of view in standard LFA systems

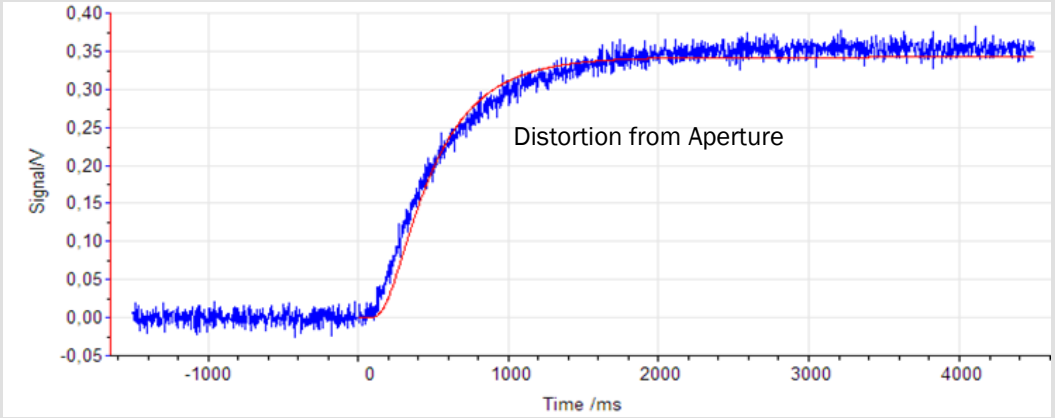
No Zoom Optics — Distortion from Aperture

→ Field of view is fixed, large enough to cover big diameter samples.

→ Testing smaller diameter samples, apertures are used minimizing the influence of the surroundings.

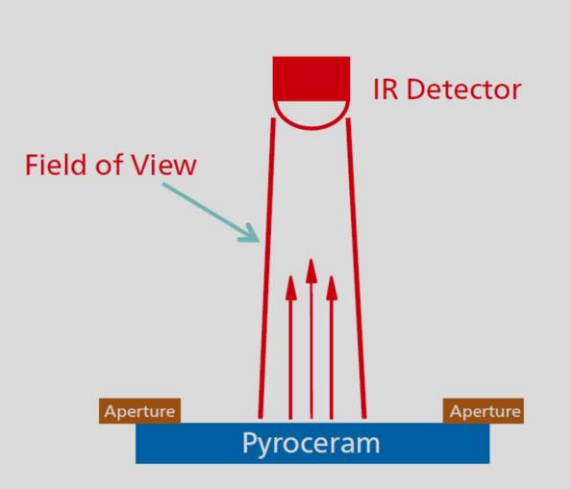
→ Significant distortion of the thermal curve: detector not only senses the temperature rise of the sample, but also any fluctuations from the aperture.

→ Thermal curve shows either a continuously increasing trend or an extended leveling-off period.



Standard LFA system without zoom optics leads to measurements with distortions from the aperture

NETZSCH LFA 467 *HyperFlash – ZoomOptics*



Field of view by using a zoom optics, no aperture influences occur

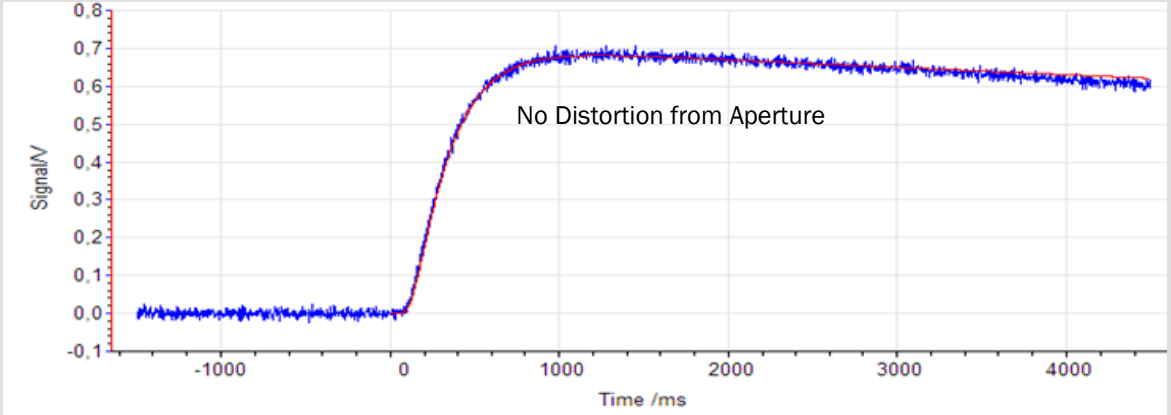
Zoom Optics — No Distortion from Aperture

→IR signal originates solely from the sample surface

→Large and small samples can be tested with an optimal sensing area.

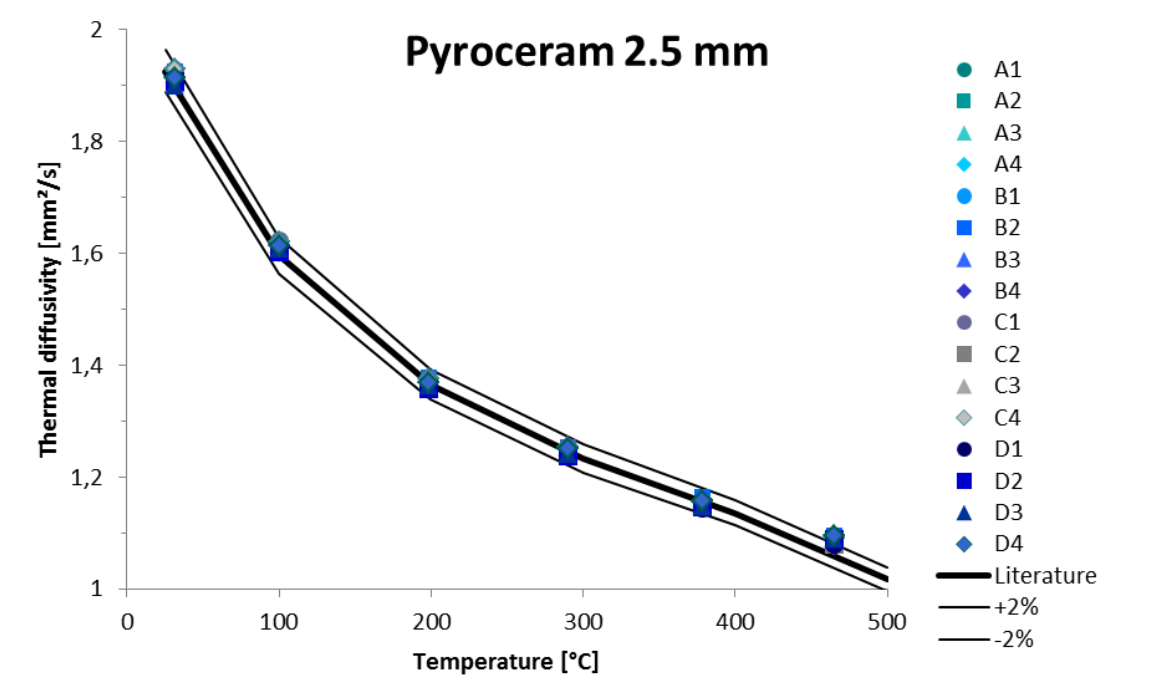
→ No contributions from the aperture

→ Thermal curve conforms to the theoretical model, yielding correct diffusivity values.



Using the zoom optics the measurement signal shows no distortion from the aperture

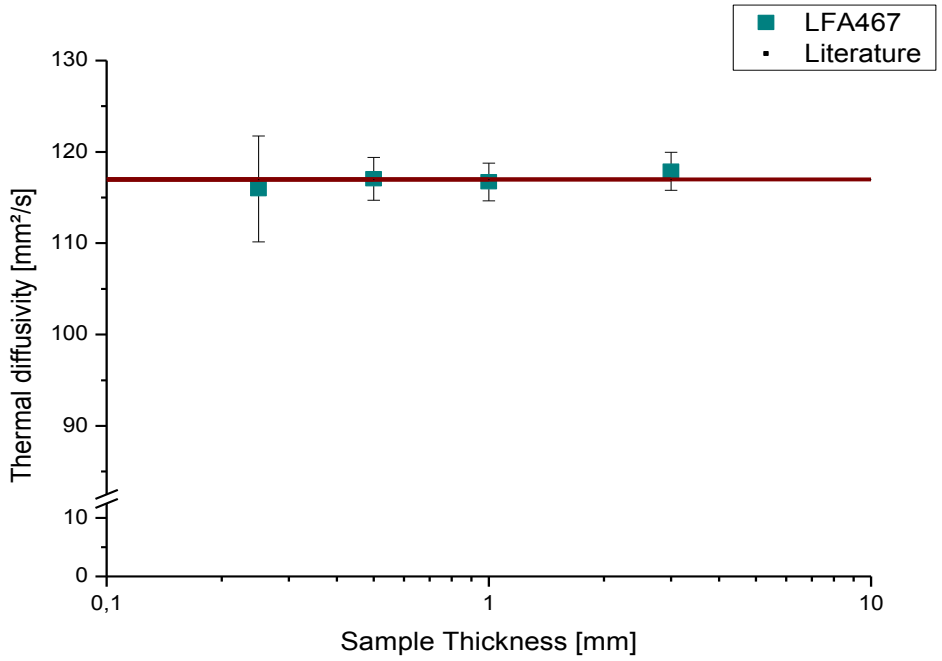
NETZSCH LFA 467 *HyperFlash* – High Sample Throughput



Highest Sample Throughput – High Efficiency

- → 16 Pyroceram samples (2.5 mm thick, ∅ 12.7 mm)
- → Thermal diffusivity shows a deviation of ±2% from literature data.
- → Optimum position for each of the 16 samples in the furnace over the entire temperature range.
- → Reduced measurement time
- → High throughput possible without operator‘s intervention

NETZSCH LFA 467 *HyperFlash* – Thin and High Conductive Materials



Thermal diffusivity of the copper samples are in very good accordance with literature data independent from the sample thickness.

Thin and High Conductive—Copper

→ Copper samples with different thicknesses from 3.0 mm to 0.25 mm.

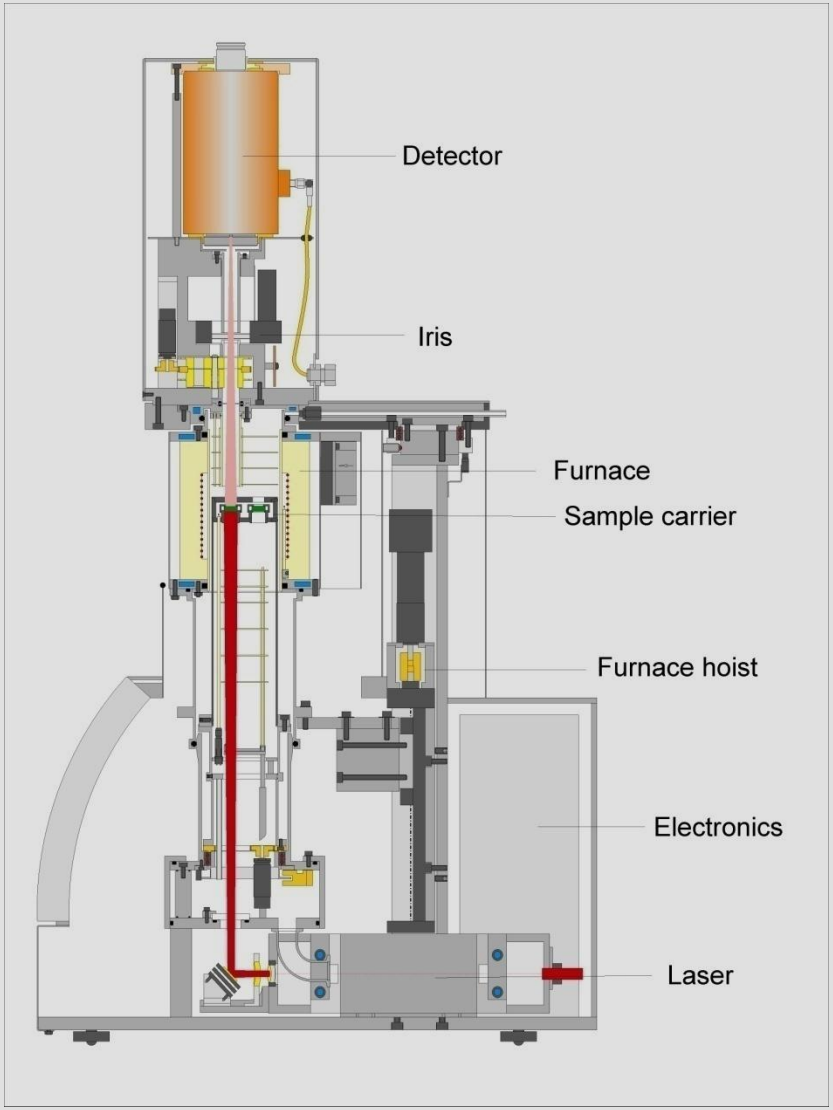
→ Measurements confirm that even very thin samples can be tested with very high accuracy.

→ Sample preparation and thickness determination have to be carefully considered when measuring thin samples. Especially graphite coating can lead to lower thermal diffusivity values.

NETZSCH LFA 457 *MicroFlash*®

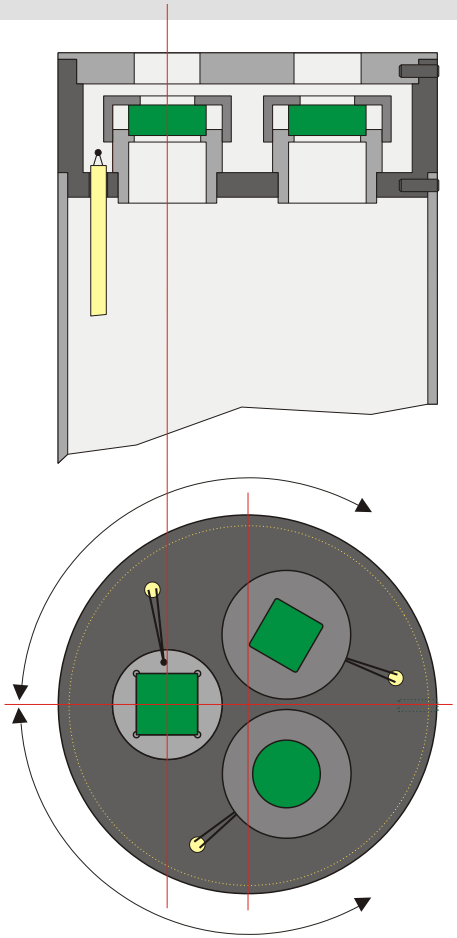
- Table-top unit with laser
- Temperature range: -125 to 1100 °C
  (with two furnaces)
- Integrated sample changer for 3 samples

LFA 457 *MicroFlash*® – Design

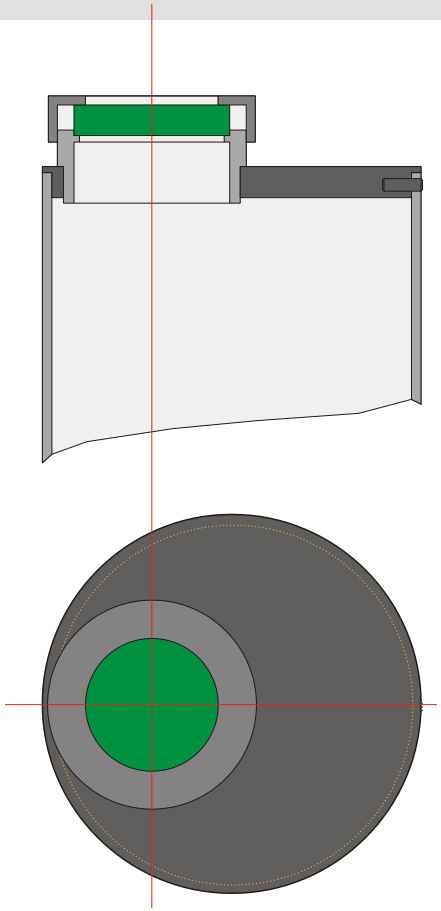


- Vertical system
- Two exchangeable furnaces
  - -125 … 500°C
  - RT … 1100°C
- Vacuum-tight (1 Pa)
- Laser pulse length ~ 0.33 ms

LFA 457 *MicroFlash*® – Sample Changer

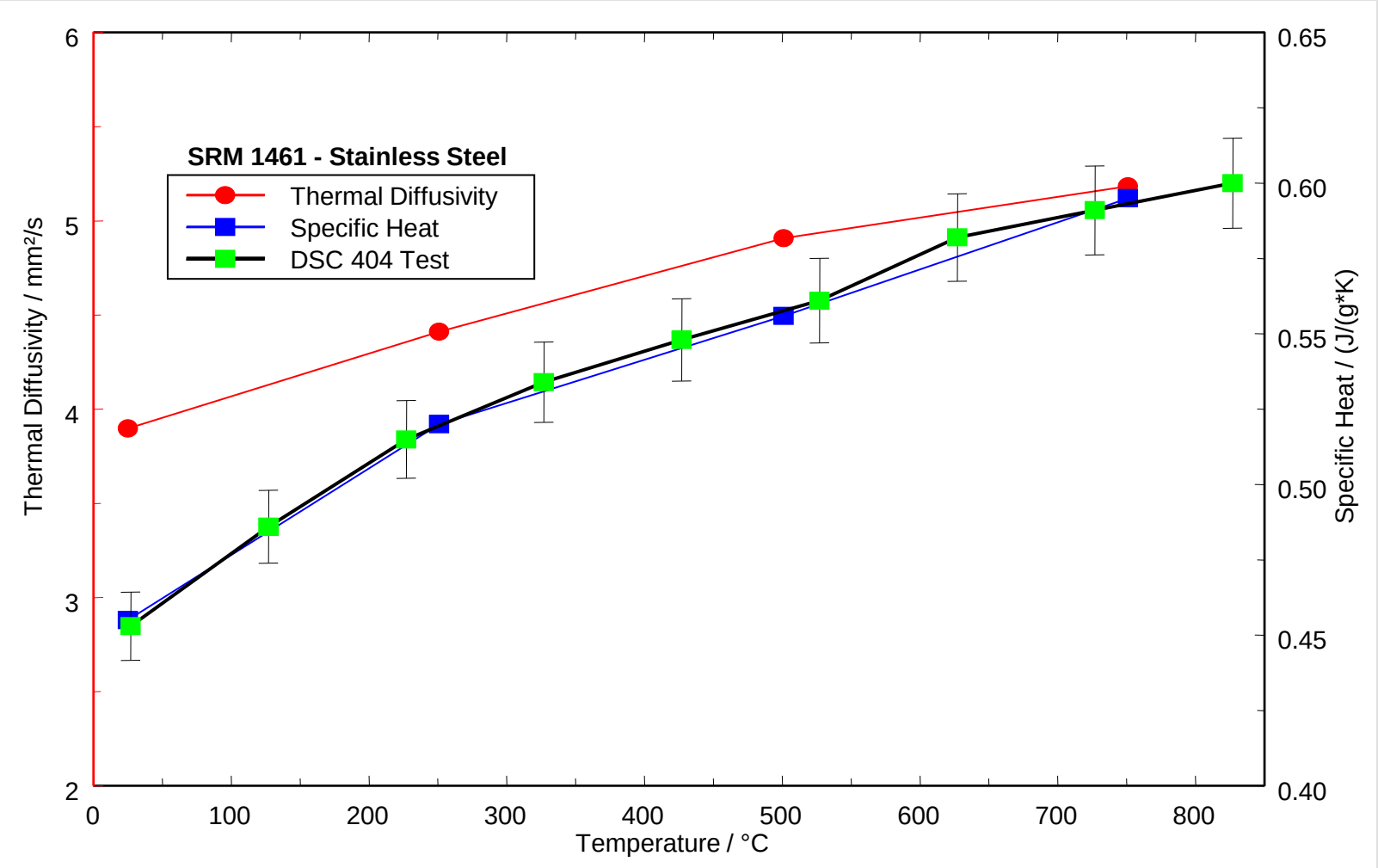


Multiple Sample Arrangement
12.7 mm dia. and 10.0 mm square

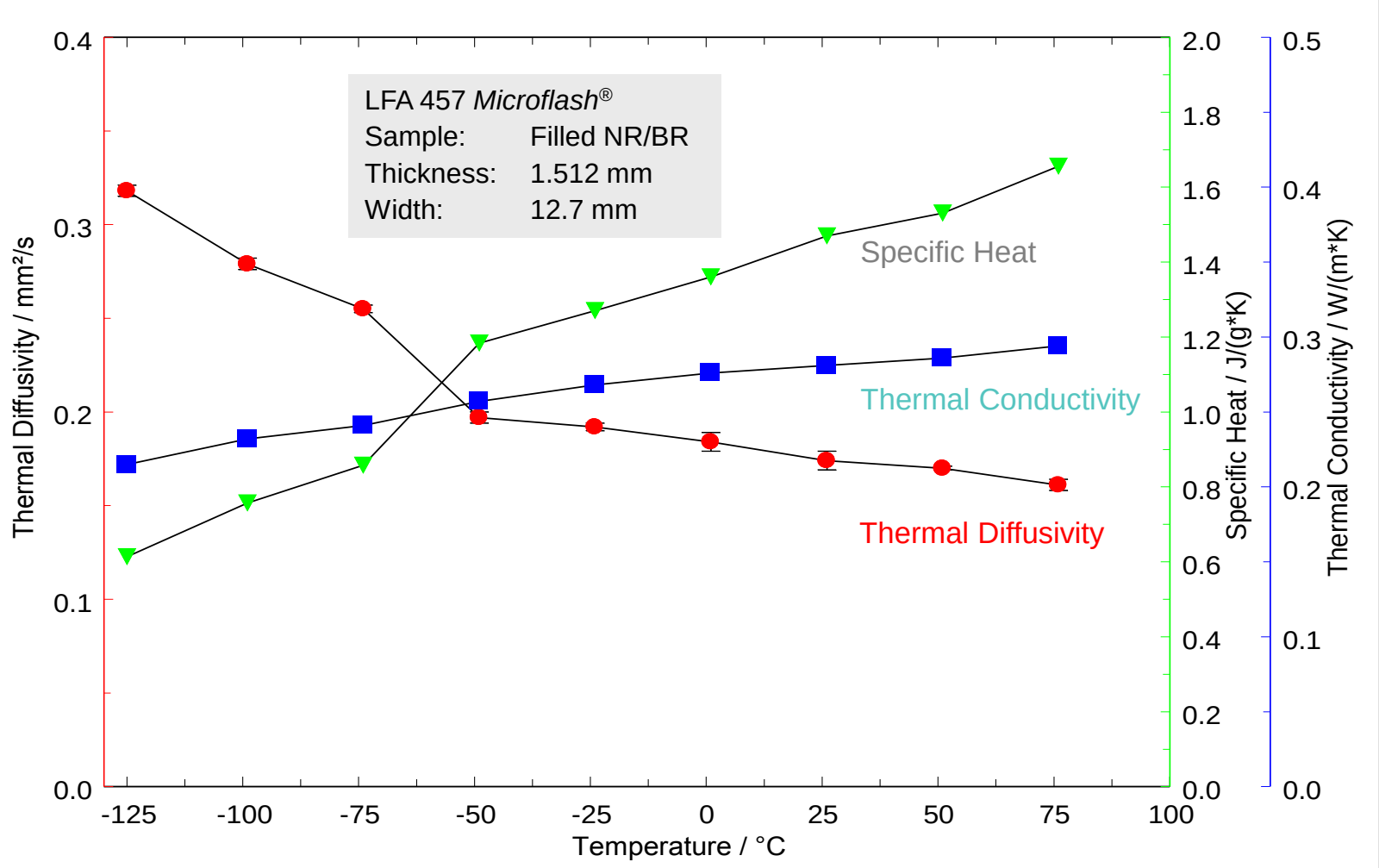


Single Sample Arrangement
25.4 mm dia.

Standard Application – Stainless Steel



Application – Filled Rubber

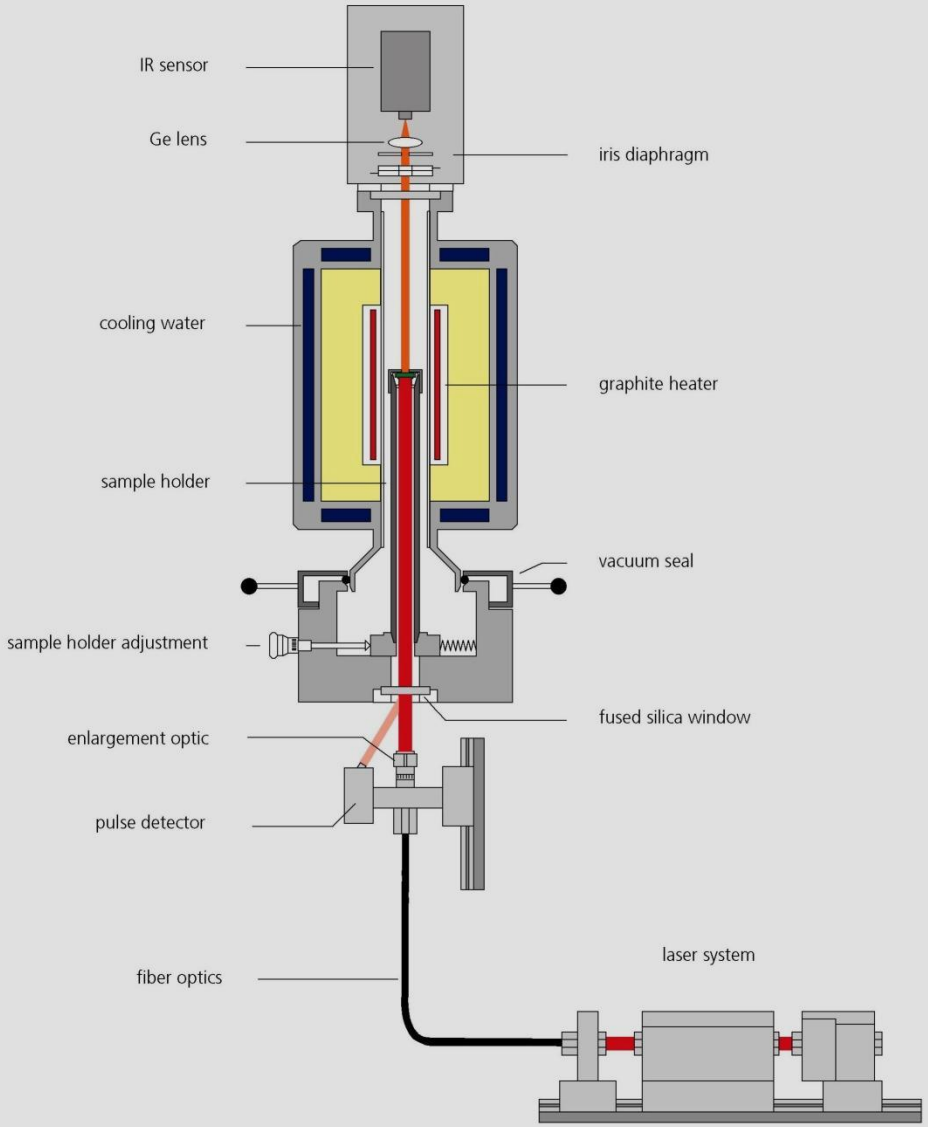


Thermophysical Properties Testing
NETZSCH LFA 427

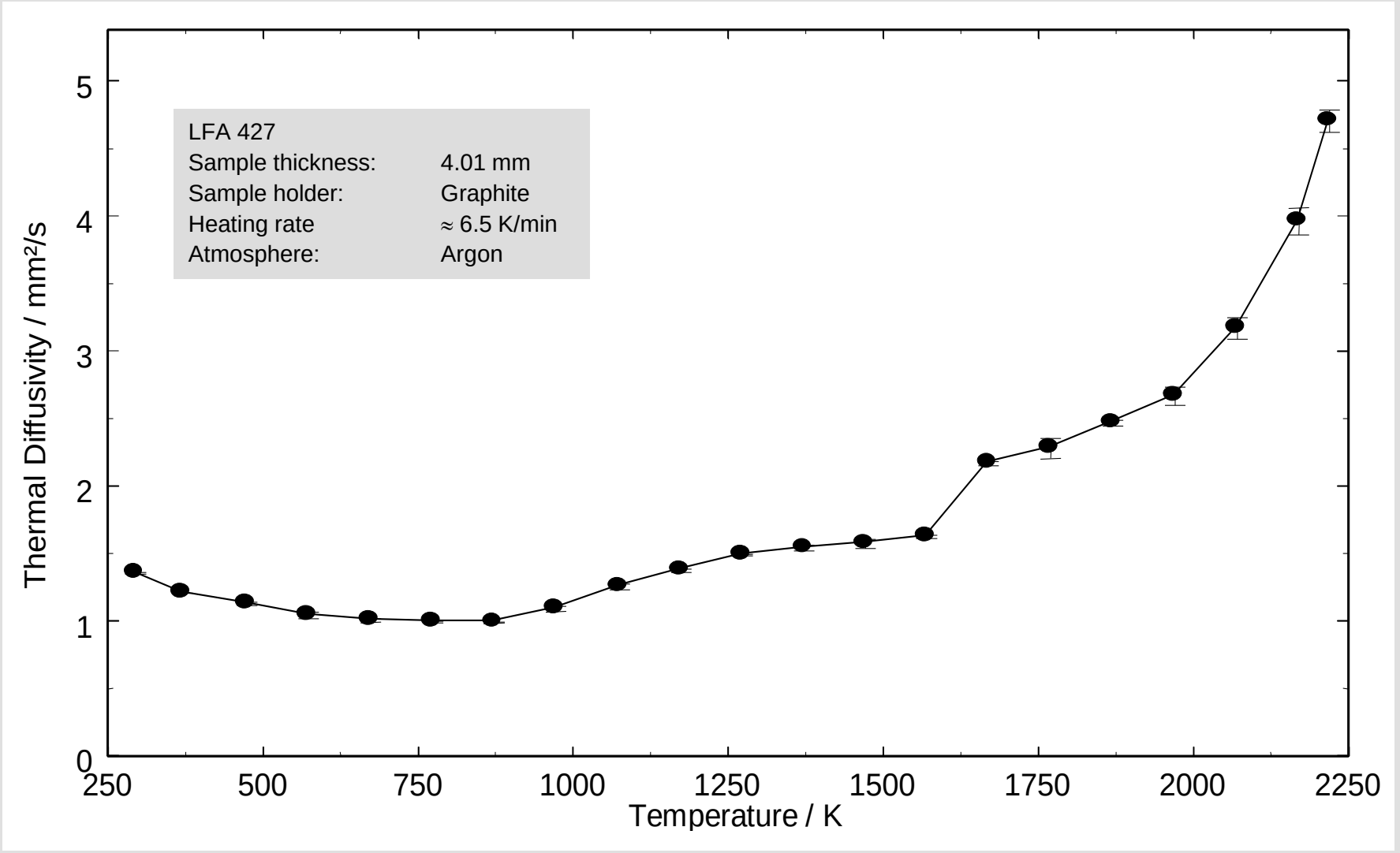
- Temperature range -120 to 2800°C
- Vacuum-tight design $1*10^{-5}$ mbar
- Different sample holders
  - standard sample holder
  - square sample holder
  - laminate sample holder
  - fiber sample holder
  - slag sample holder
  - liquid metal sample holder

NETZSCH LFA 427

- The system is vacuum-tight by design.
- Laser, sample and detector are in line (No mirrors are used)
  ⇒ Improved signal-to-noise ratio.
- One single system covers the temperature range from room temperature to 2000°C



Application: Silicon Carbide



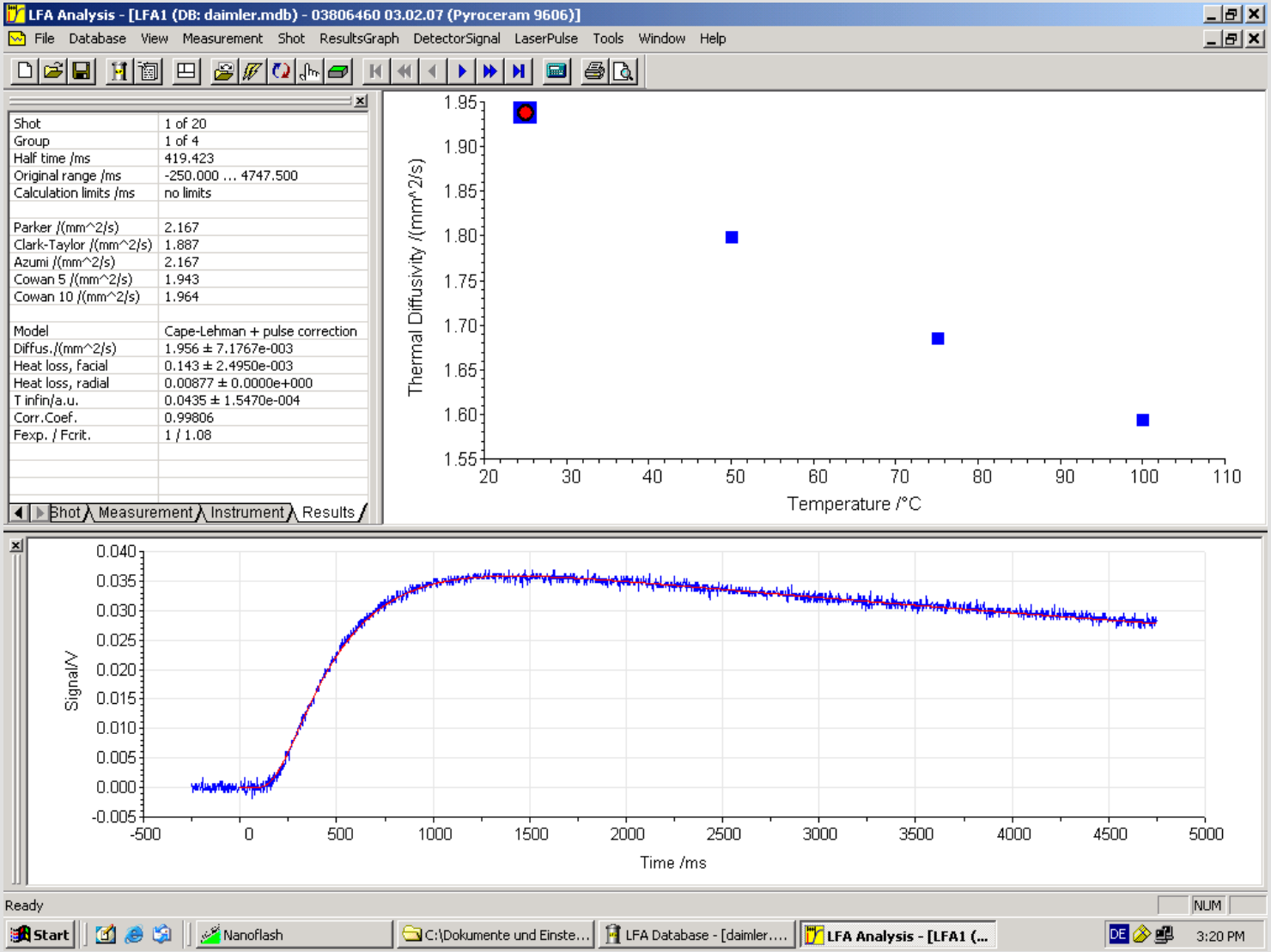
Laser Flash – Instrument Setup: NETZSCH LFA 427/8/G Pyro

Temperature range: RT to 2800°C

Conclusion

- NETZSCH offers a full range of flash systems (Xenon and laser based) for a broad application range.
- The LFA 447 *NanoFlash*® allows for scanning the sample's surface -> MTX.
- The LFA 467 *HyperFlash* offers optimum performance for thin and high conductive materials in the temperature range from -100°C to 500°C. The integrated *ZoomOptics* guarantees for an optimized field of view and precise results.
- The LFA 457 *MicroFlash*® is the right research instrument for applications from -125°C up to 1100°C.
- The LFA 427 fulfills the requirements of extreme temperatures and special application ranges.

LFA Software



Some Reasons for the Success of the Flash Technique:

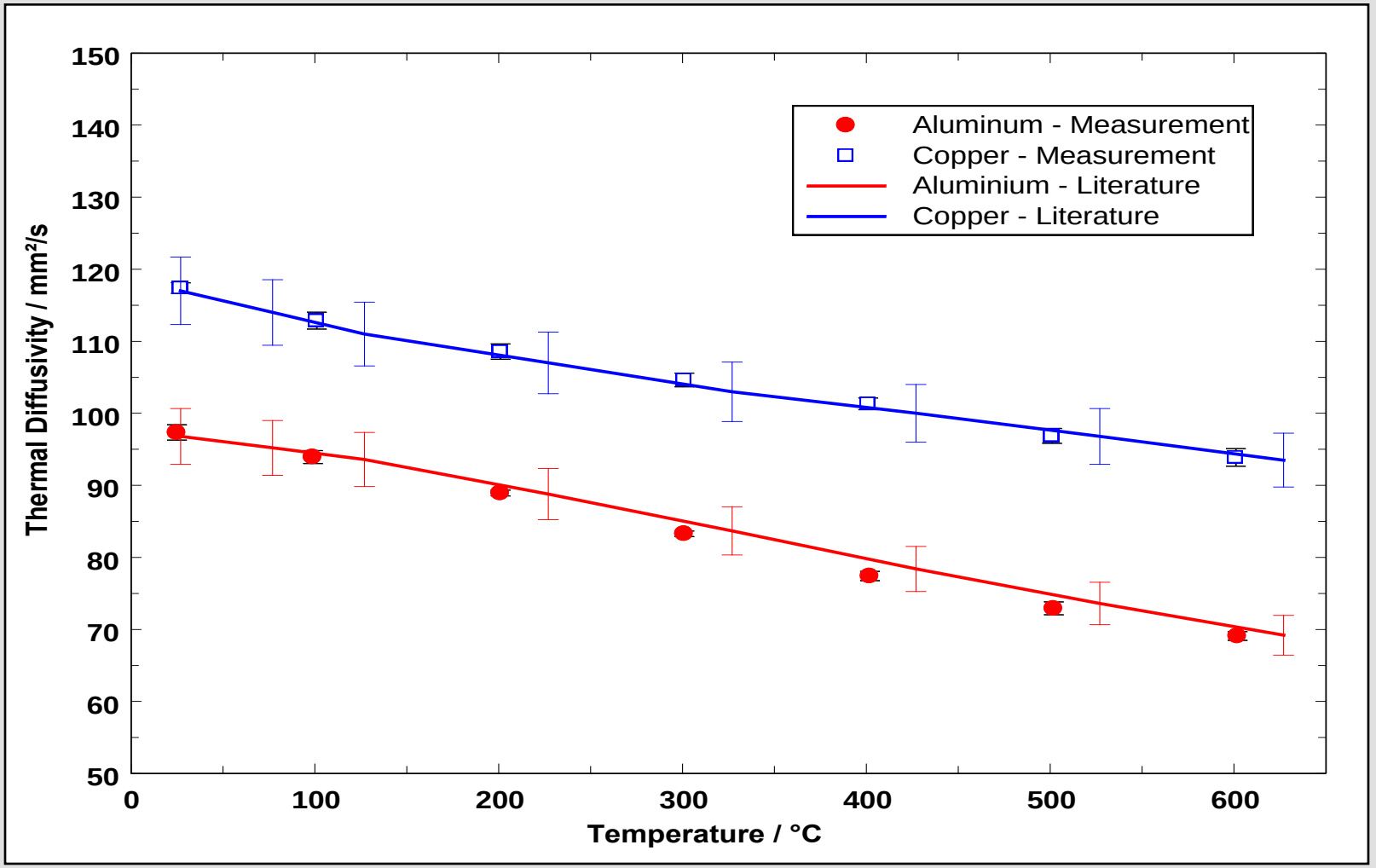
1. The method covers a broad measurement range (0.01 … 1000 mm²/s, **0.05 … 2000 W/(m*K)**)
2. The laser flash technique can easily be adapted to temperature-dependent tests (**-125 … 2800°C**)
3. Sample preparation is easy
4. It is a **non-contact, non-destructive** measurement technique
5. It can be employed on **multi-layer systems**
6. **Fast testing times** (generally a few seconds)

Agenda

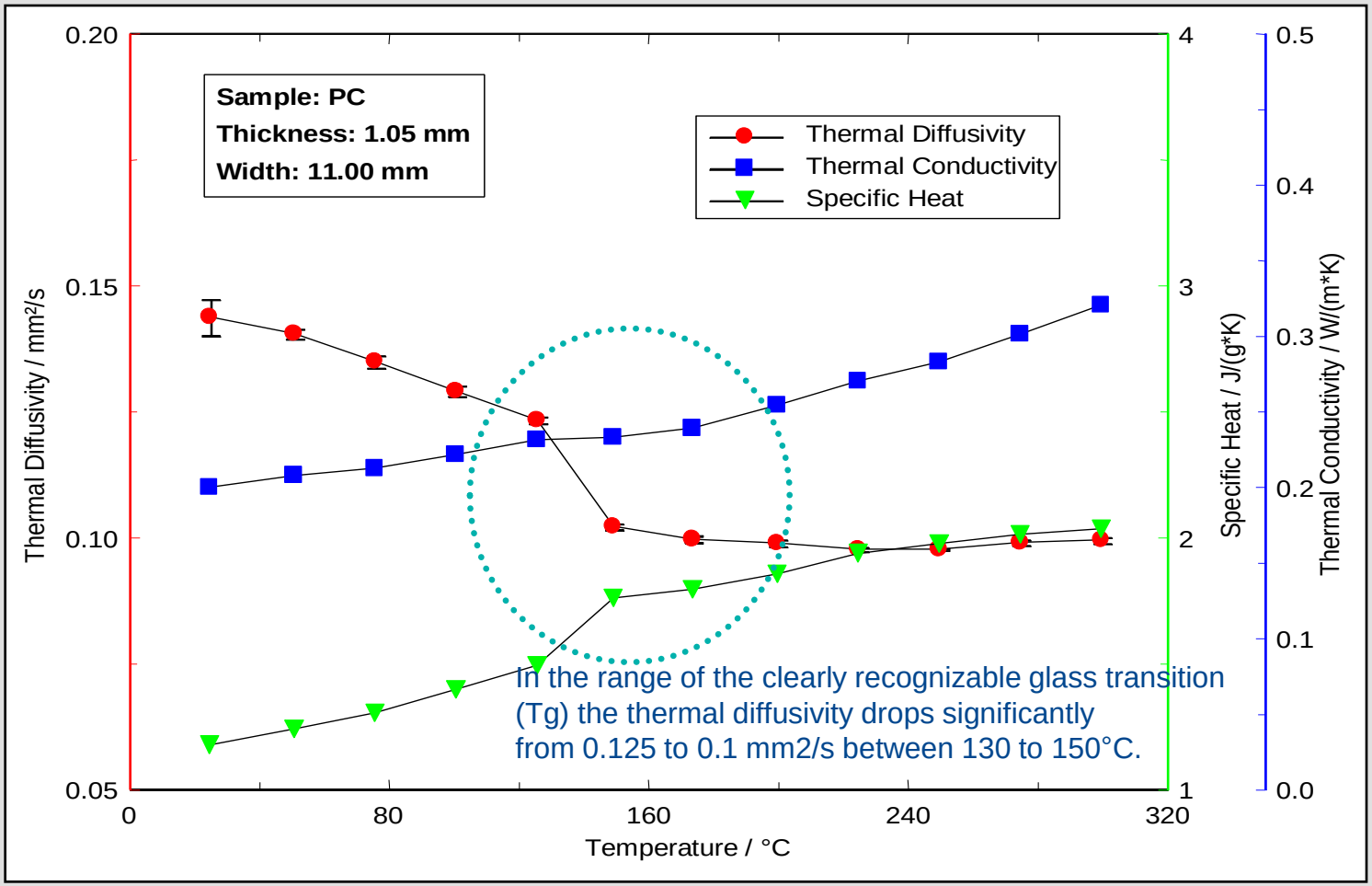
1. Heat Transfer - Basics
2. LFA - Method
3. LFA - Instrumentation
4. Application Examples
5. Summary

Thermal Diffusivity of Metals: Cu and Al

Comparison to Literature Values



Polycarbonate (PC) in an Aluminum Container

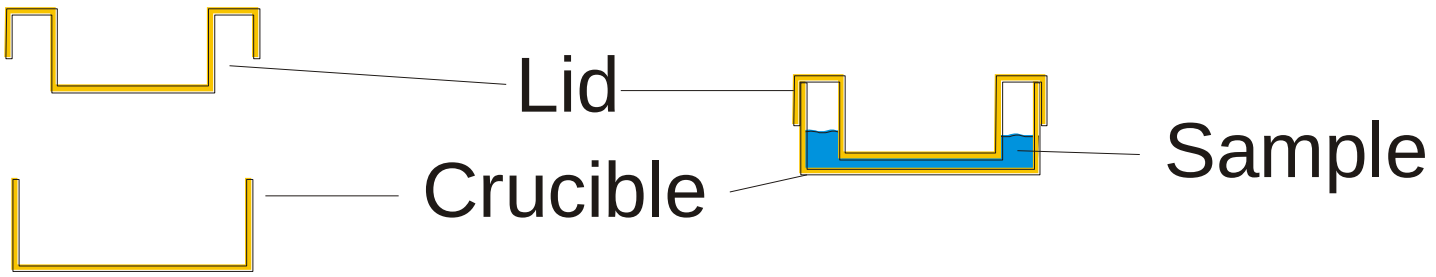


In the Tg range the specific heat (Cp) rises from 1.5 to 1.8 J/gK (typical for 2nd order transitions). The thermal conductivity (λ) shows a continuous progression. Although the thermal diffusivity (a) decreases constantly after the Tg, λ of the amorphous material continues to increase due to the increasing Cp and amounts to 0.3 W/mK at 300 °C (thus at injection moulding temperature).

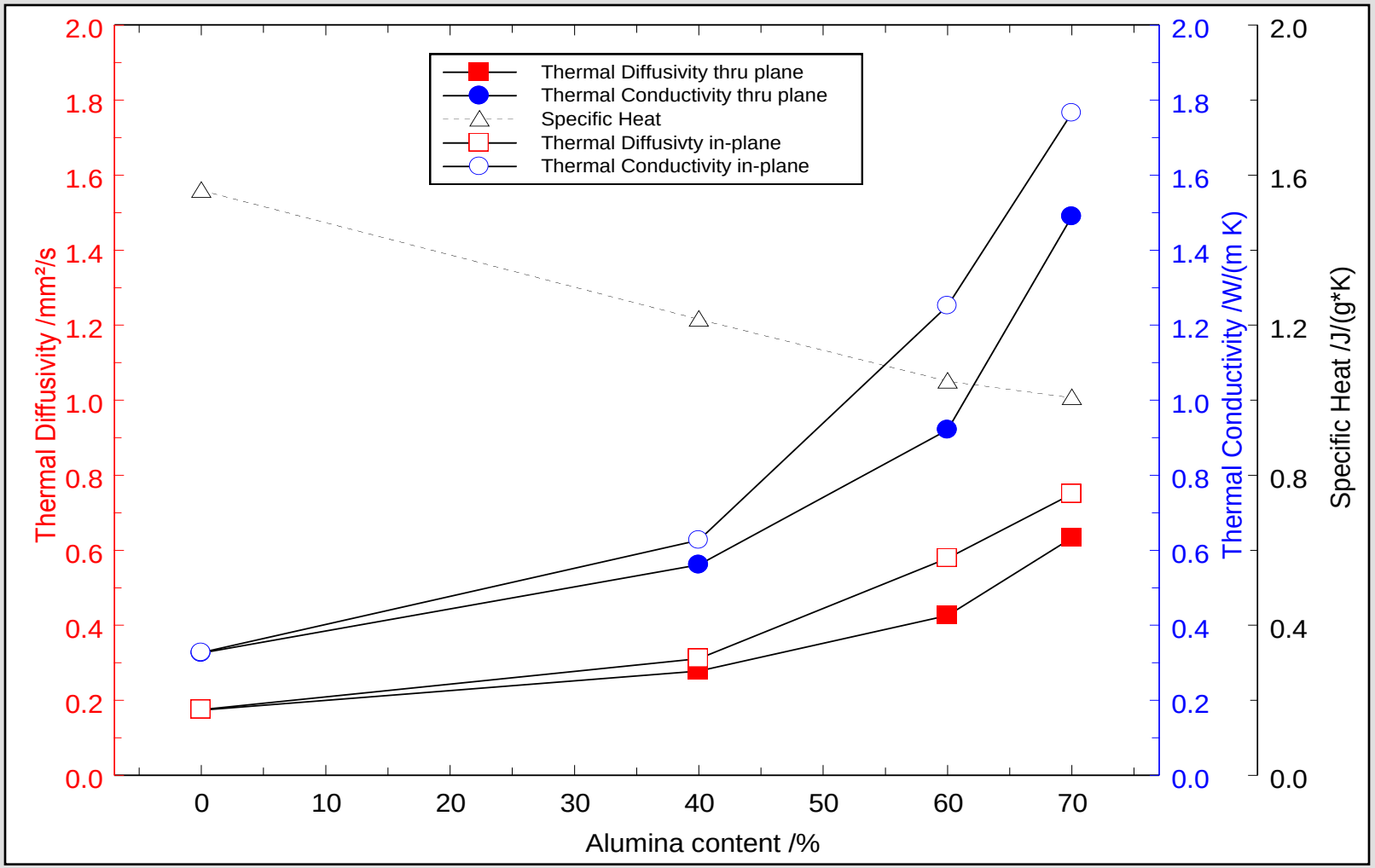
Liquids and Molten Polymers in an Aluminum Container

NETZSCH

Aluminum Sample holder for Low-Viscosity Liquids



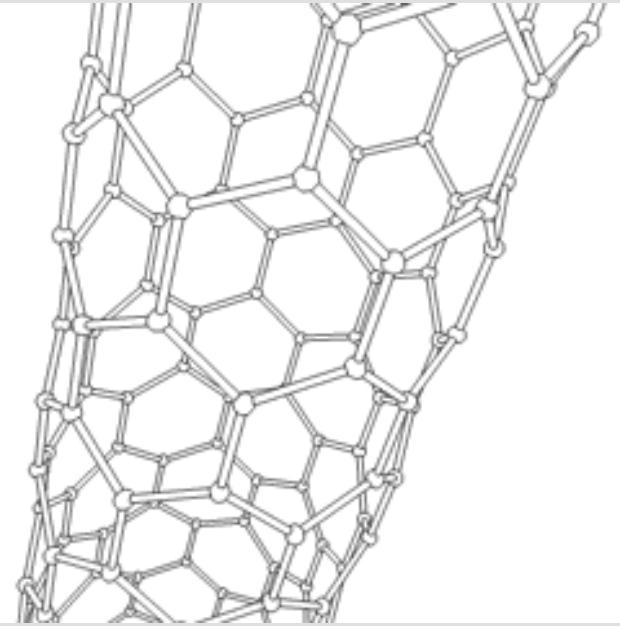
Polyamide PA 46
Influence of the Filler Content

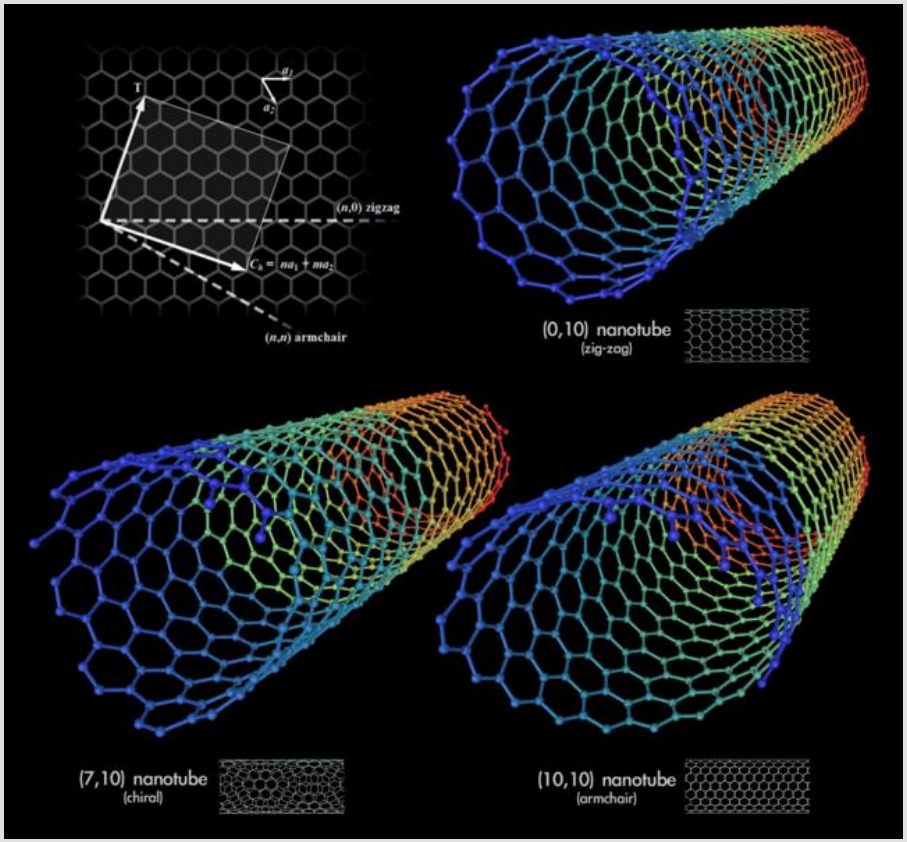


PA 46 alone has a very low **thermal conductivity.** With increasing filler (Al_2O_3) content in the polymer the **specific heat** decreases while the **thermal diffusivity** and **conductivity** increase.

Polyetheretherketone (PEEK) Filled with Carbon Nanotubes (CNT)

NETZSCH



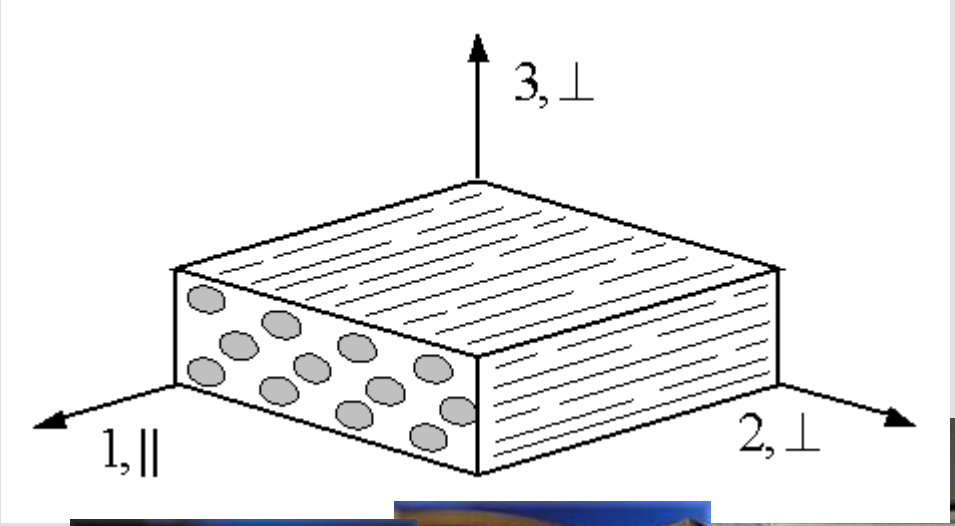


Prepared in Cooperation with:

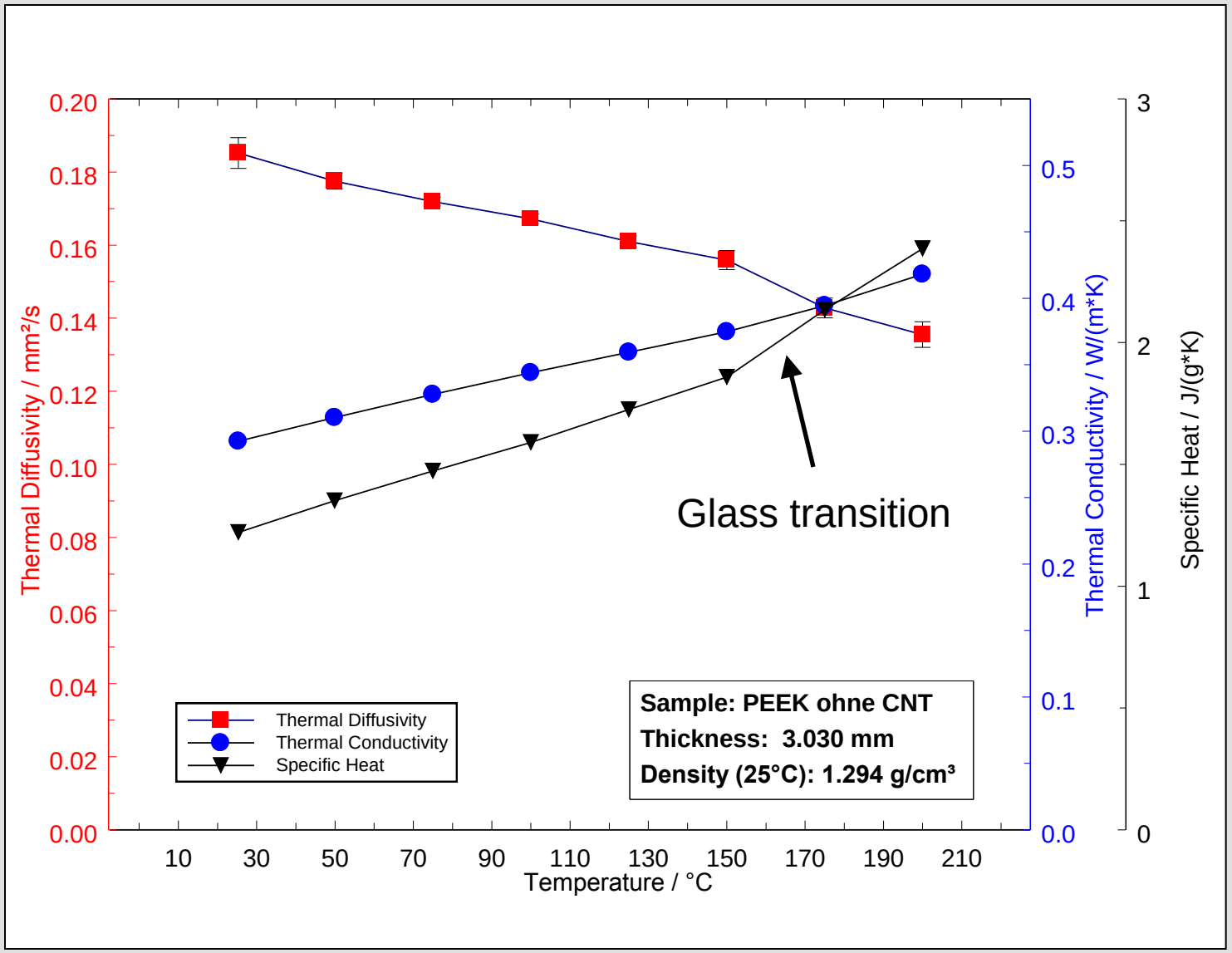
ENSINGER
ASK. THINK. SUCCEED.

PEEK filled with CNT - Anisotropy

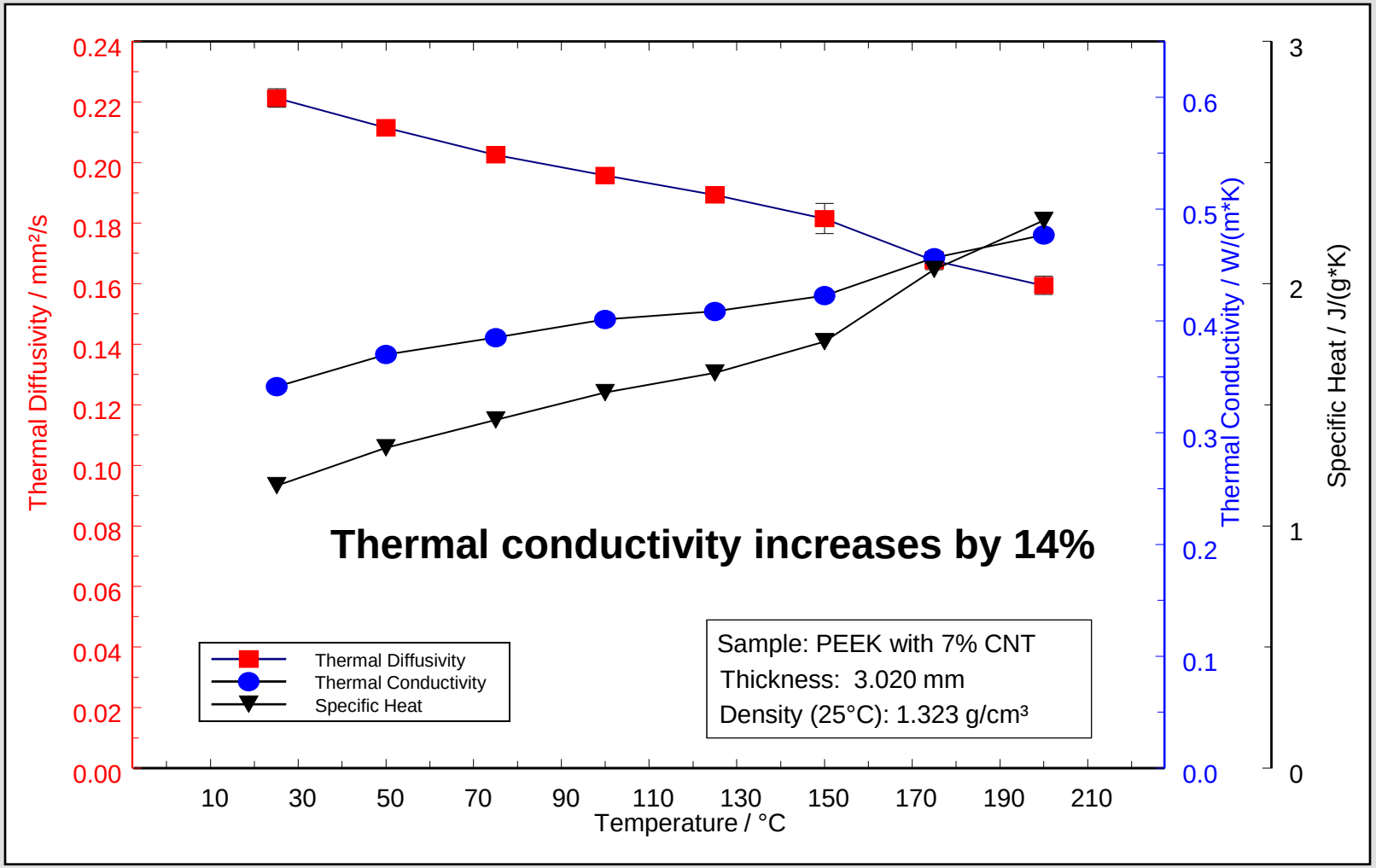
→ **Orientation of Carbon Nanotubes due to injection moulding**



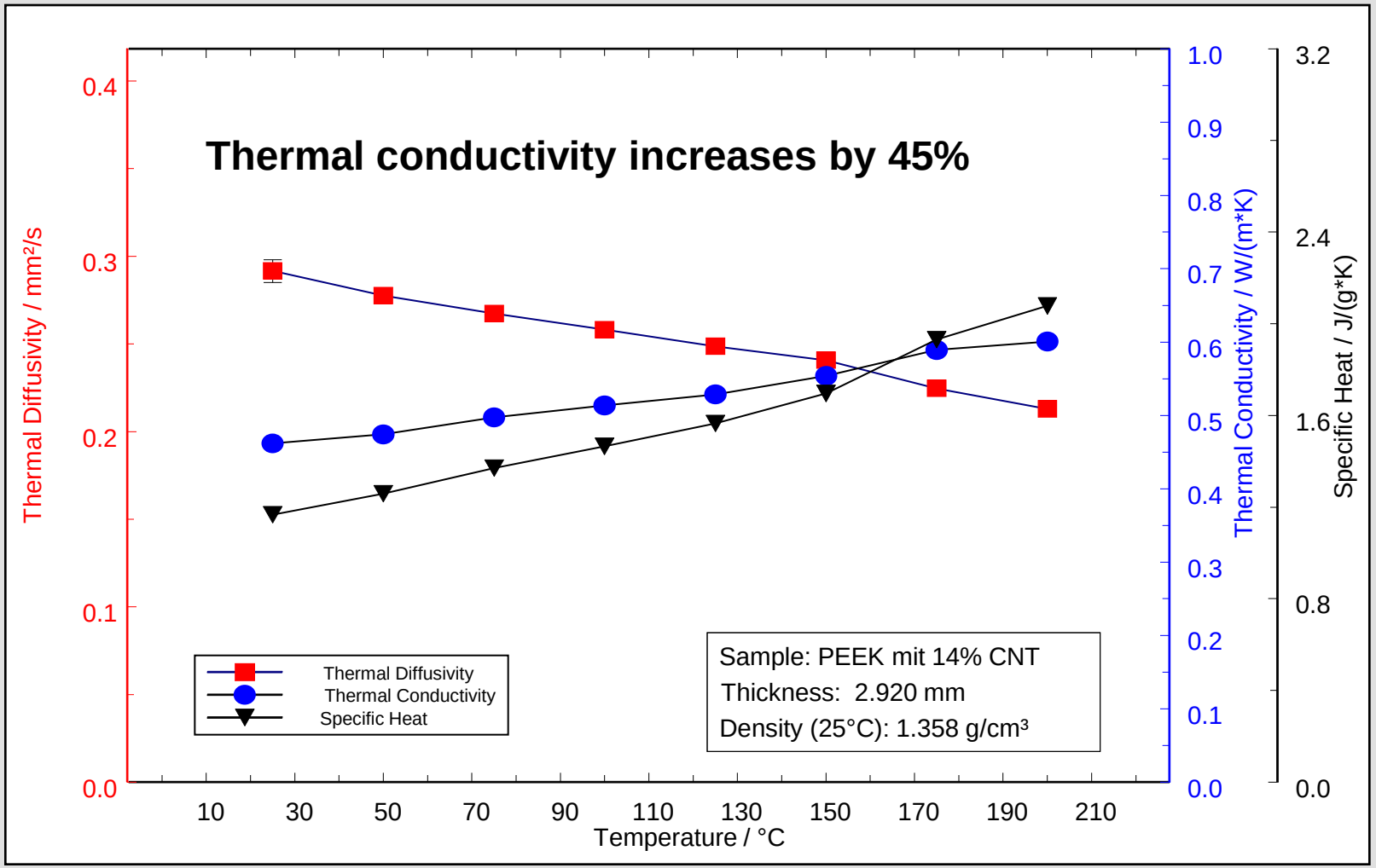
Pure PEEK Matrix



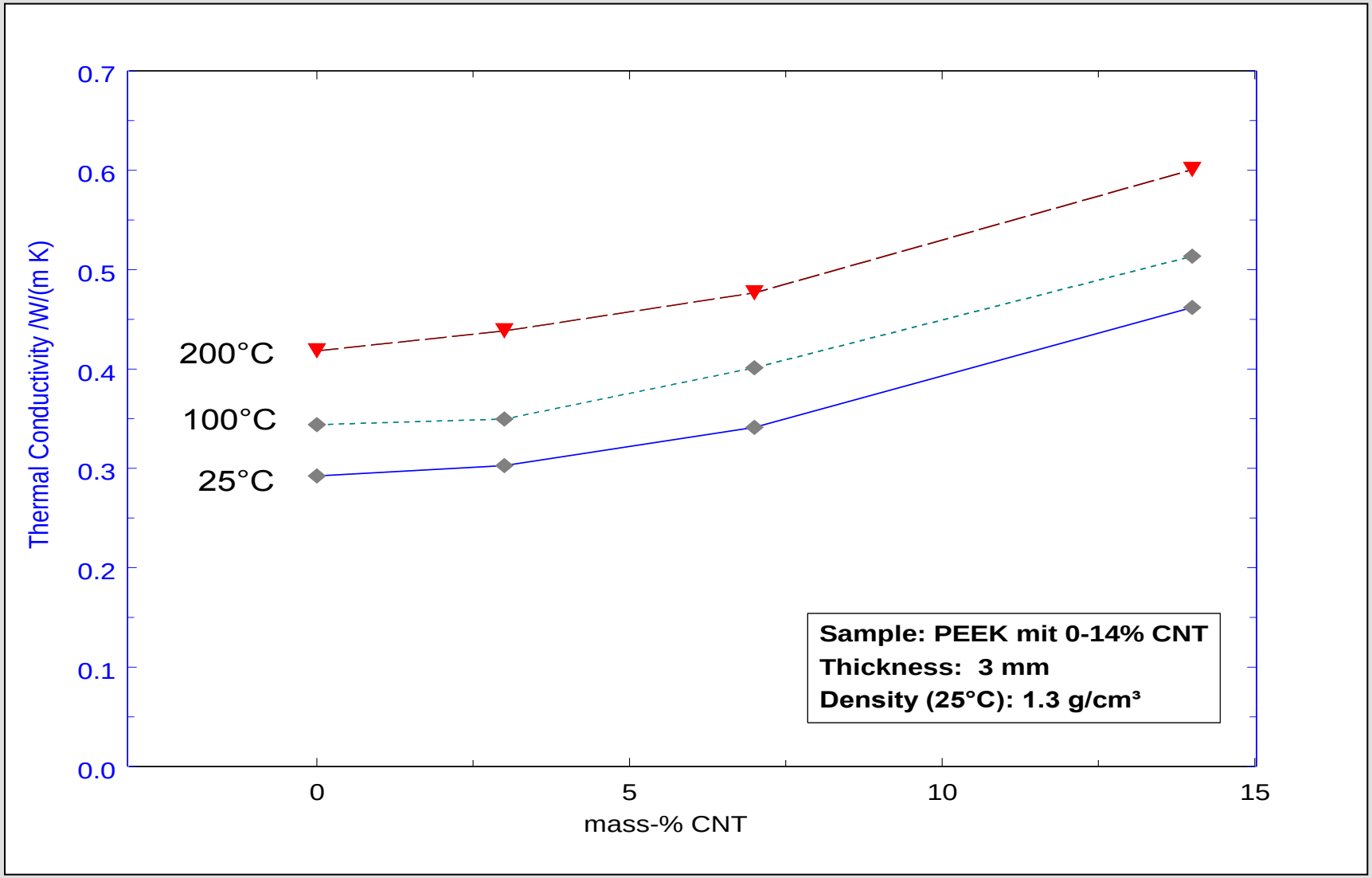
PEEK Filled with 7% CNT



PEEK Filled with 14% CNT

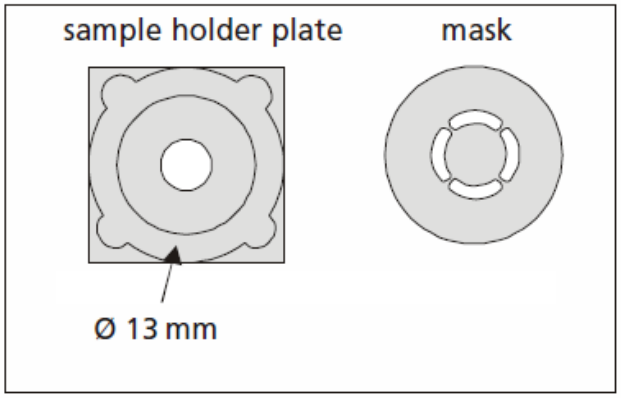


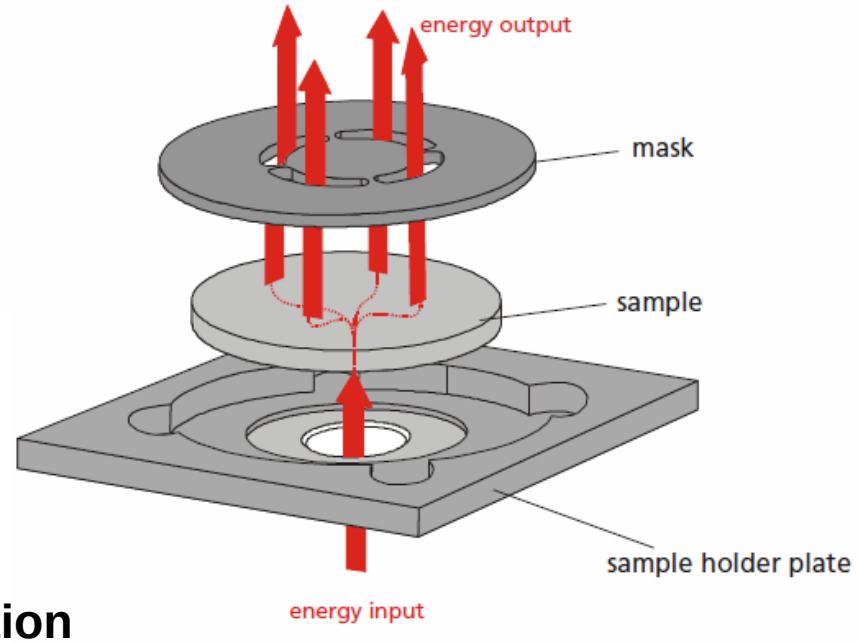
Increasing Thermal Conductivity with Increasing CNT Content



Anisotropy: In-Plane Sample Holder

In-plane sample holder





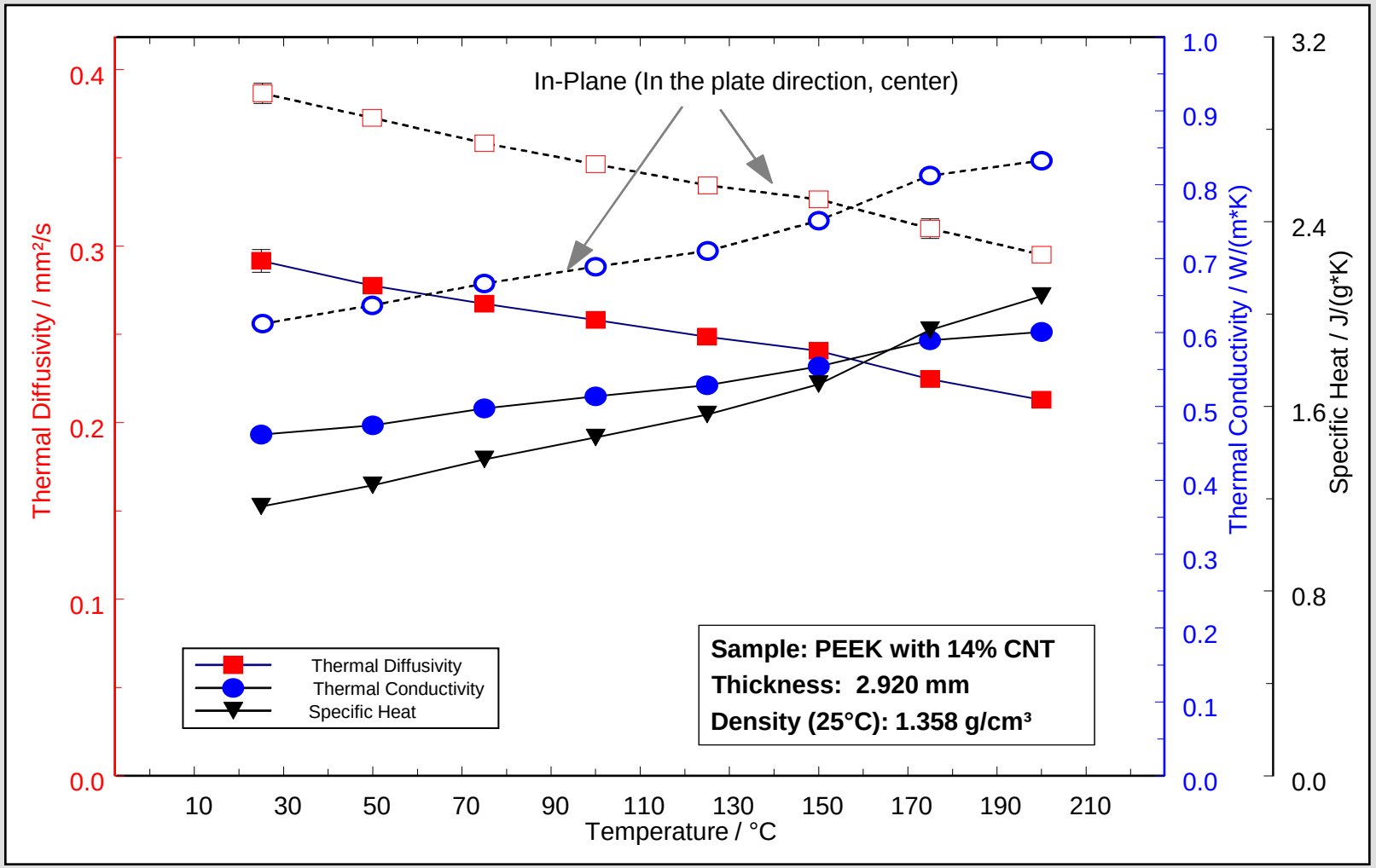
Vertical direction

through-plane

Horizontal direction

In-plane

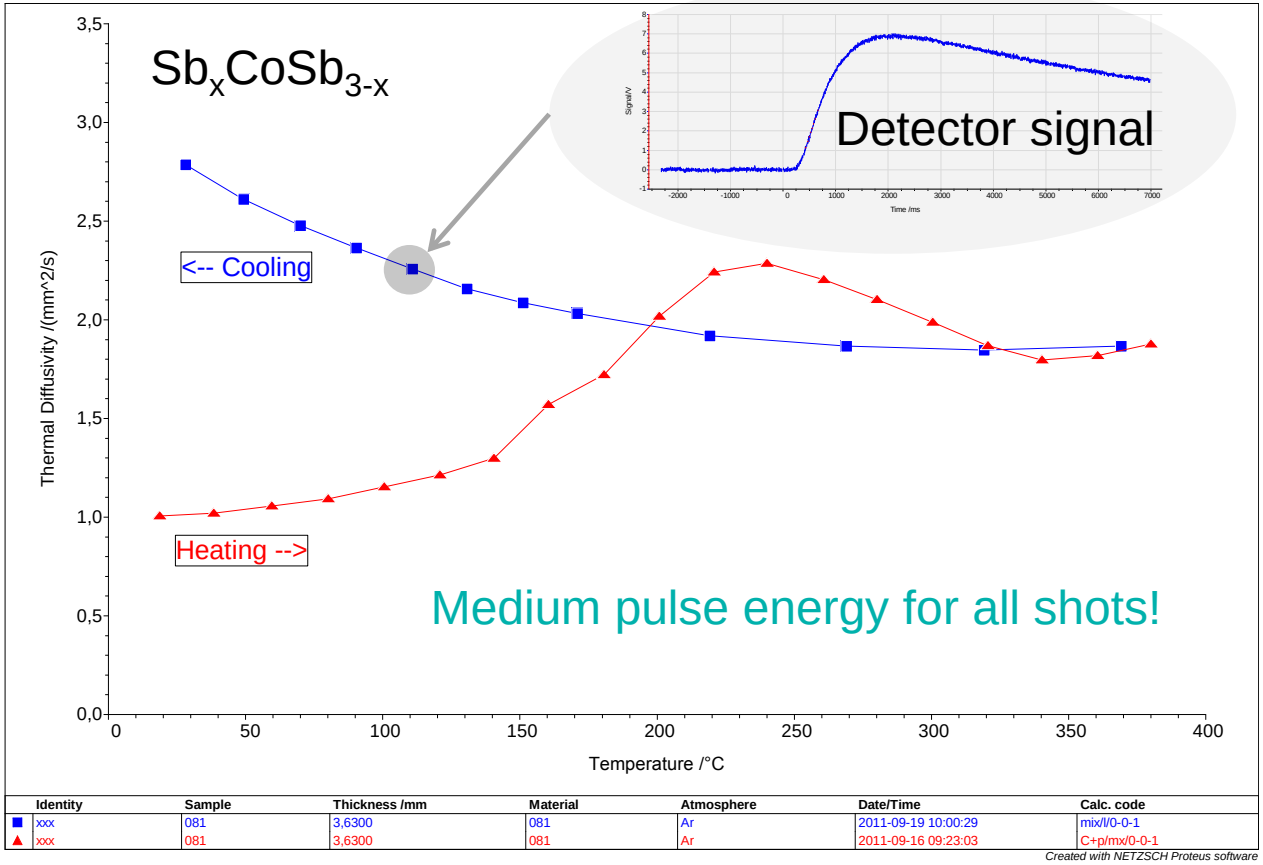
PEEK with CNT – Anisotropy



Key Advantages of Netzsch LFA Systems

High sensitivity opens the doors for smallest sample dimension

- Low conducting thermoelectric material with bad dimension
- Diameter = 6 mm; Thickness = 3.6 mm (recommended: 12.6 / 2 mm)



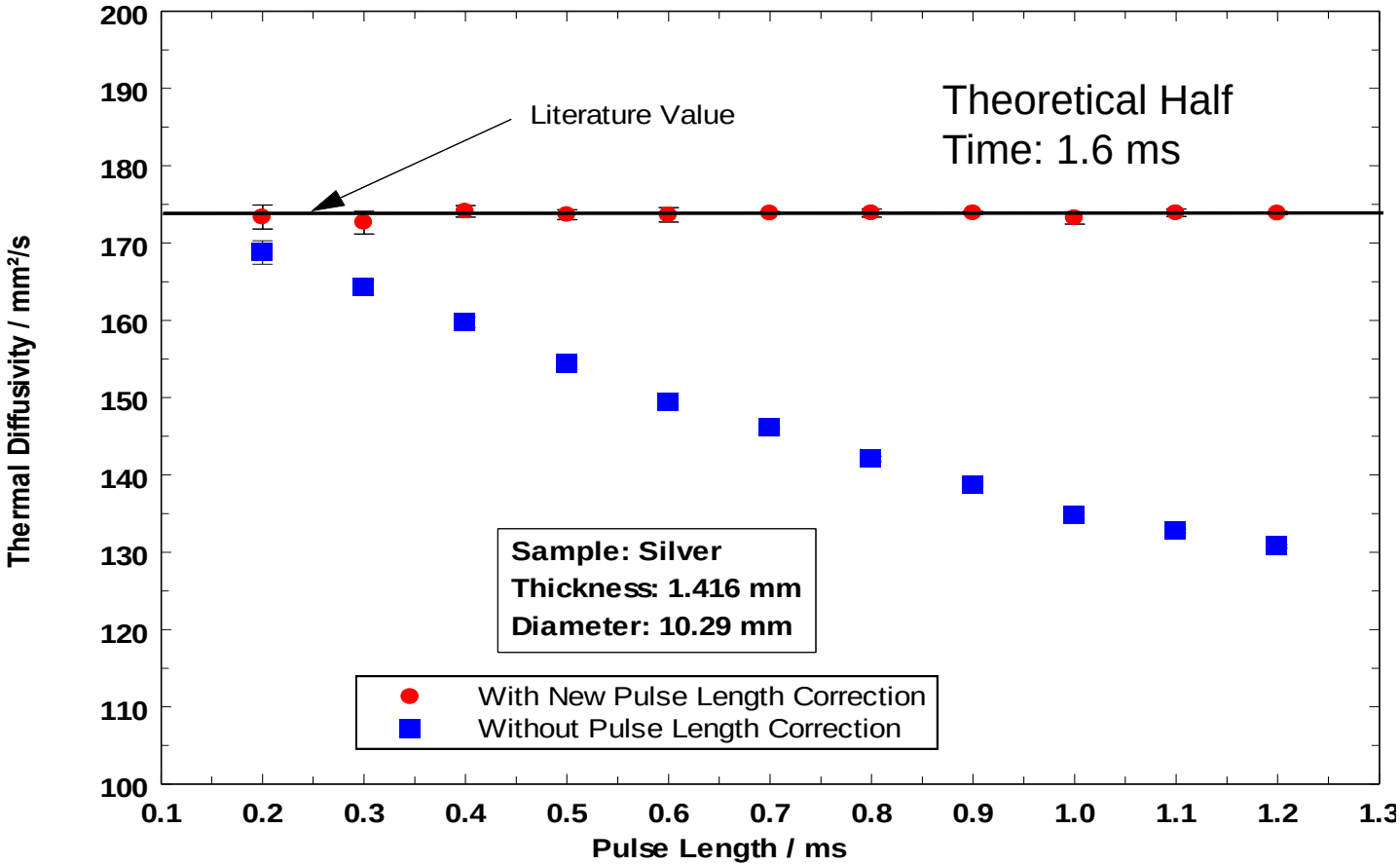
	Identity	Sample	Thickness /mm	Material	Atmosphere	Date/Time	Calc. code
■	xxx	081	3,6300	081	Ar	2011-09-19 10:00:29	mix/l/0-0-1
▲	xxx	081	3,6300	081	Ar	2011-09-16 09:23:03	C+p/mx/0-0-1

Created with NETZSCH Proteus software

- Small noise level at bad conditions
- ~5 Joule sufficient
- No overheating of sample
- Reliable results

Key Advantages of Netzsch LFA Systems

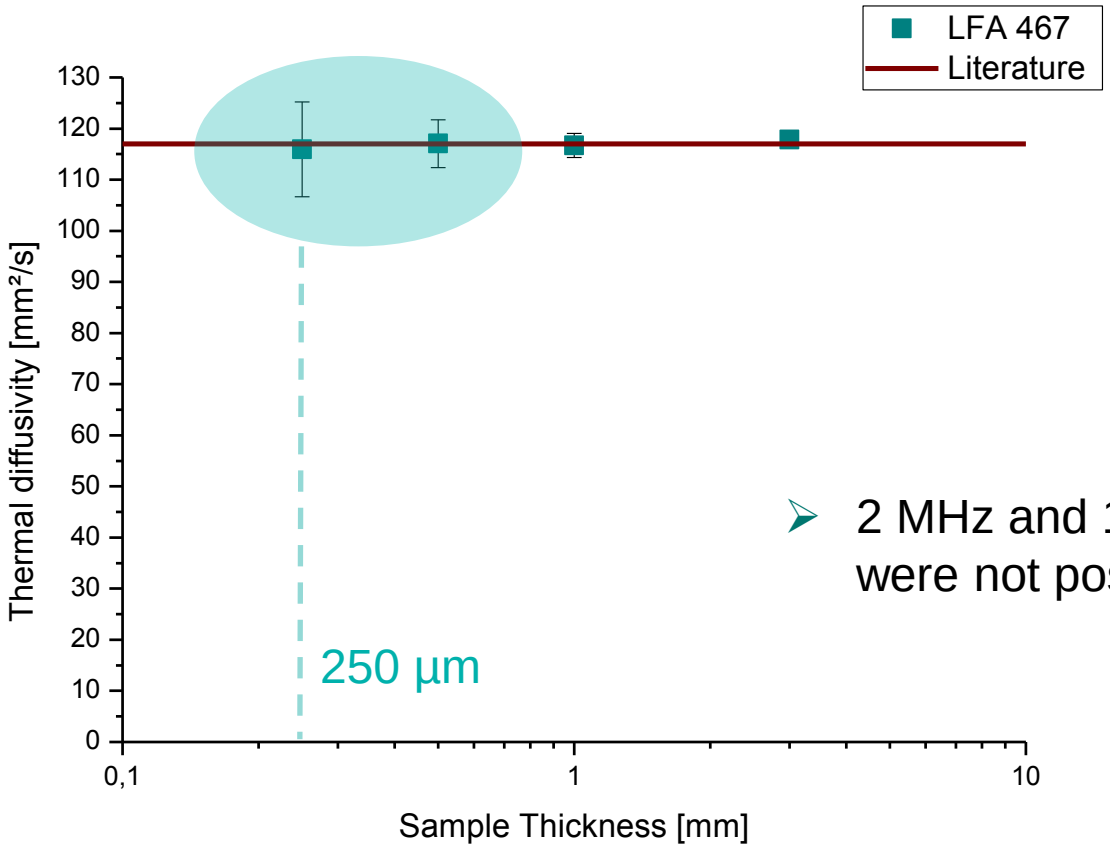
Wrong results without pulse length correction



Key Advantages of Netzsch LFA Systems

Now we measure thinner samples thanks to 2 MHz data acquisition

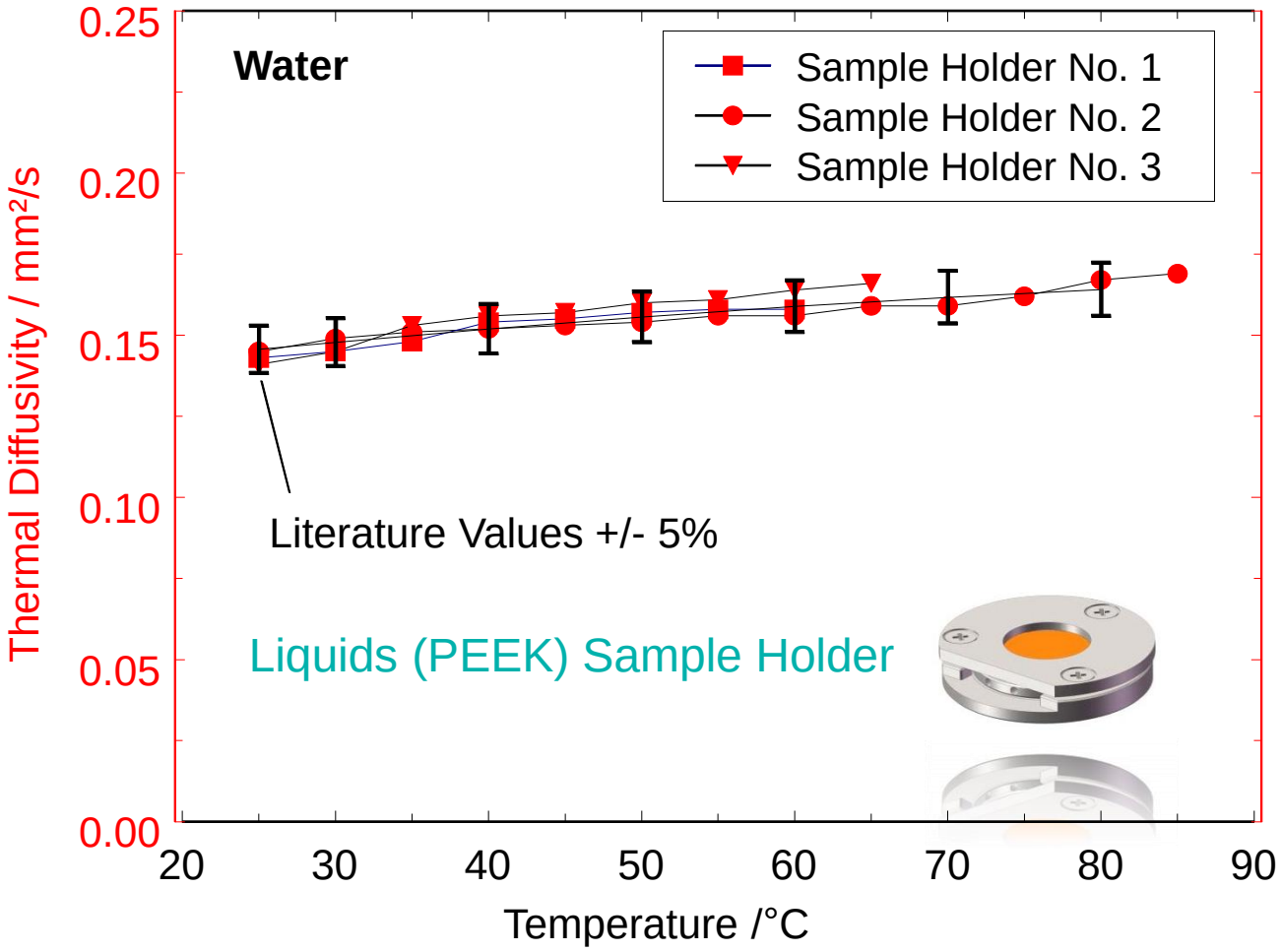
- Copper with a thickness < 1mm is possible now



- 2 MHz and 10 µs pulse length were not possible in the past!

Key Advantages of Netzsch LFA Systems

High accuracy at low viscosity liquids like water, oils …

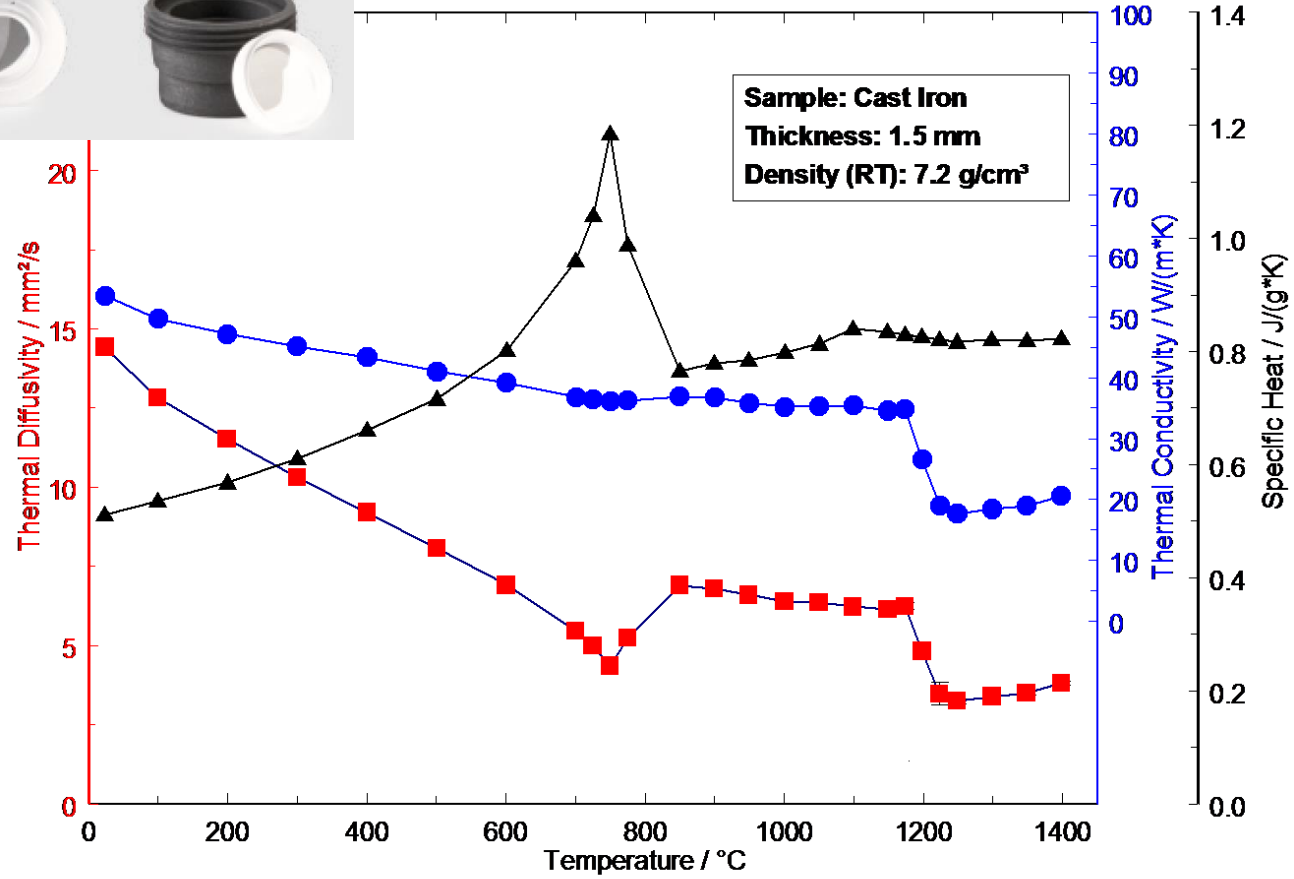


Key Advantages of Netzsch LFA Systems

Liquid metals (Sapphire) sample holder

NETZSCH

Liquid Metals (Sapphire) Sample Holder

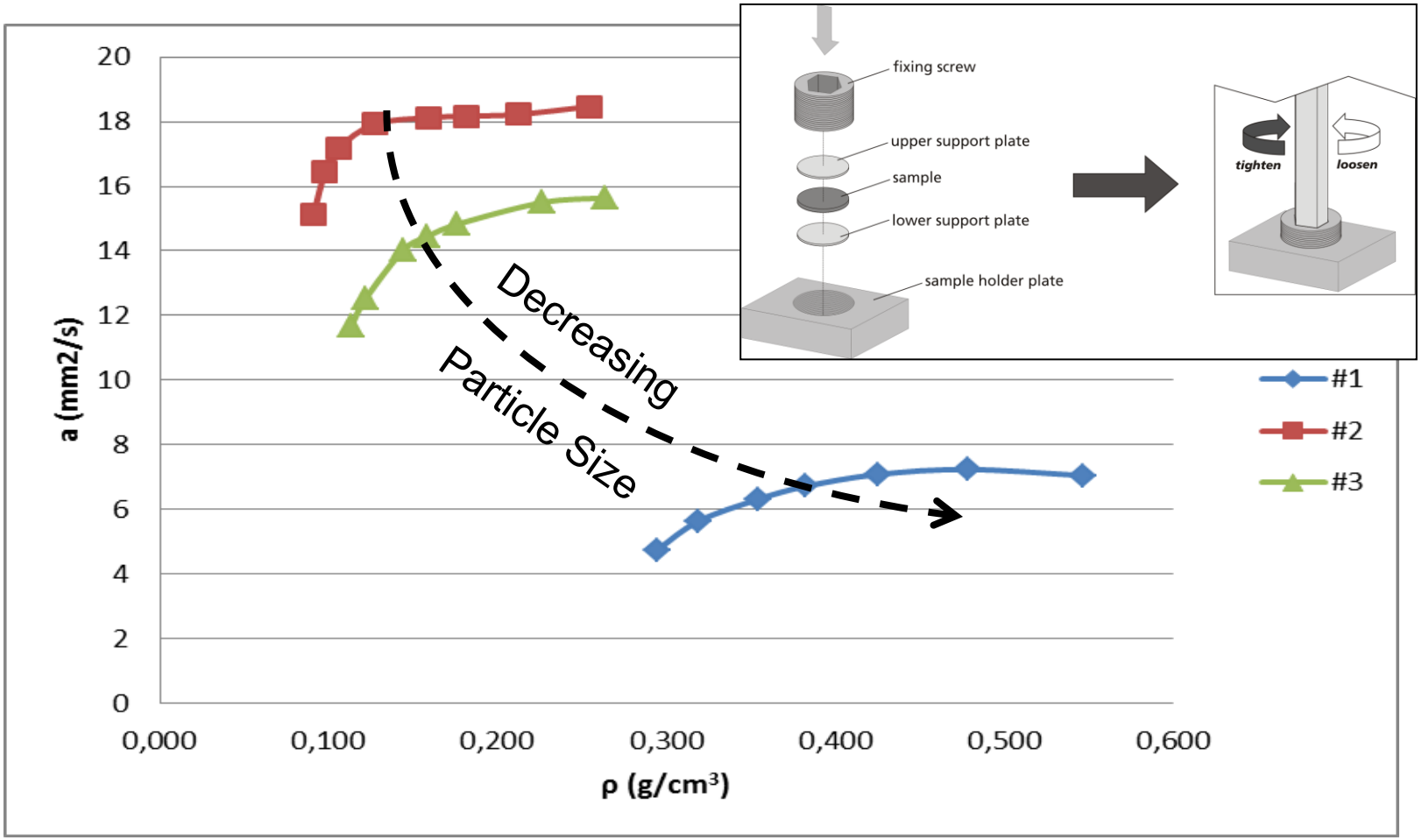


Key Advantages of Netzsch LFA Systems

Pressure sample holder for compressible samples

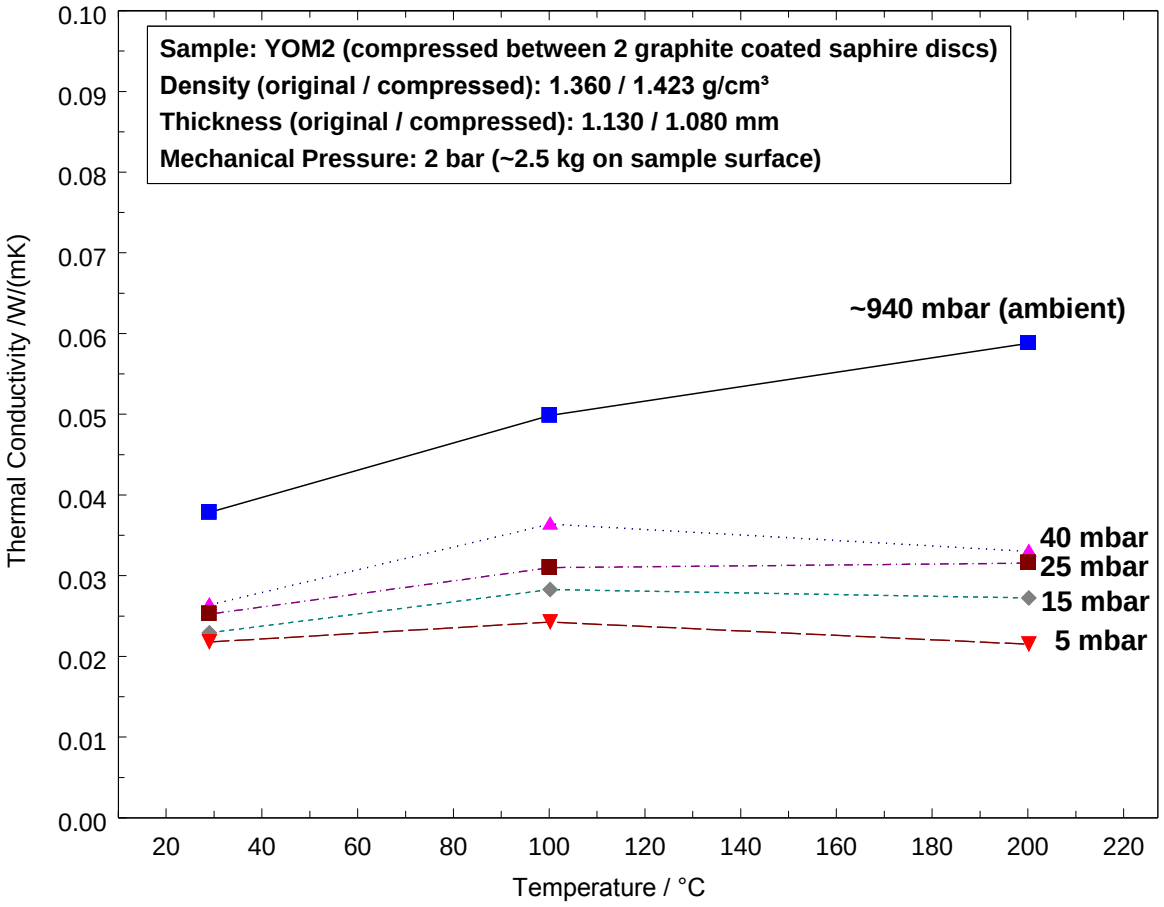
- CNT Powders with different particle sizes

Pressure Sample Holder



Key Advantages of Netzsch LFA Systems

LFA must be gas tight for pressure depending measurements

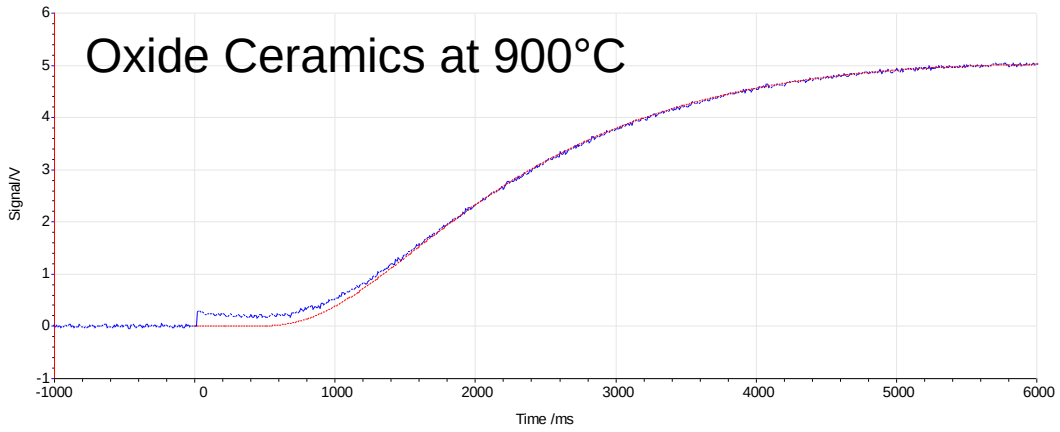


Why it must be gas tight?

- for low pressure measurements
- Temp. < 10°C (to avoid condensation)
- Temp. > 300°C (to save graphite coating)

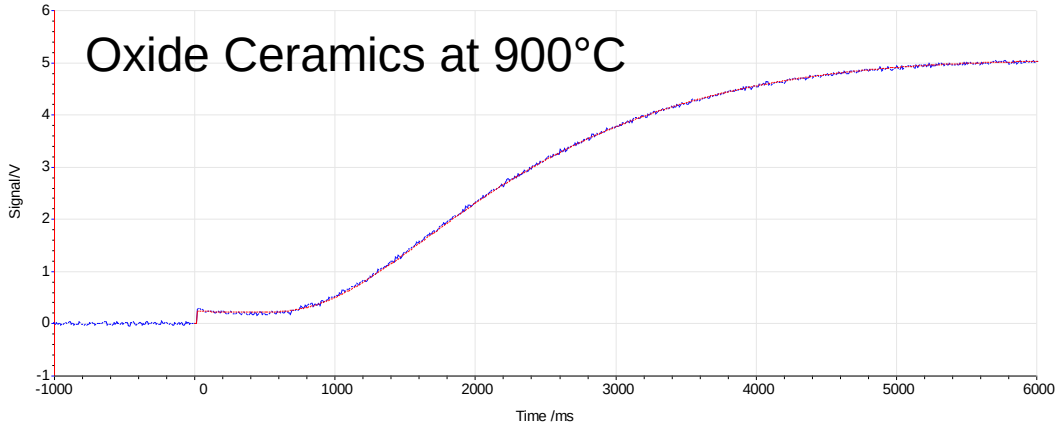
Key Advantages of Netzsch LFA Systems

Automatic model wizard for detection of best model fit



- Standard model
- Deviations after shot due to radiation

Model Wizard

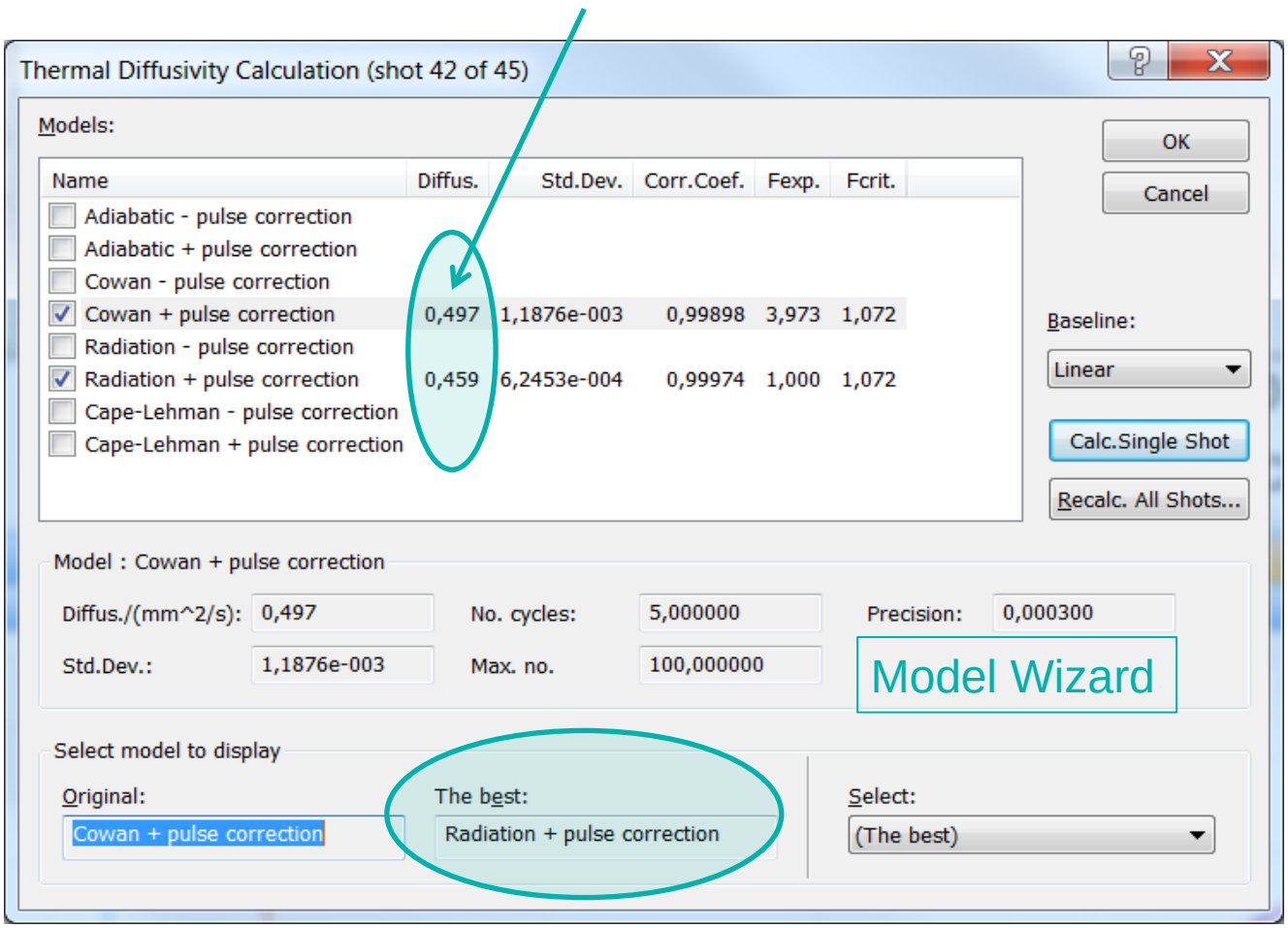


- Radiation model
- Excellent fit after shot
- Consideration of radiation

Key Advantages of Netzsch LFA Systems

Automatic model wizard for detection of best model fit

- More than 8% error if standard (wrong) model is used!



Agenda

1. Heat Transfer - Basics
2. LFA - Method
3. LFA - Instrumentation
4. Application Examples
5. Summary

Summary

- The LFA method allows fast and reliable measurement of the thermal diffusivity/conductivity of solids and liquids over a broad temperature range.

- Due to the speed of the method and the high accuracy, more and more researchers replace existing stationary methods with the flash technique.

- Even critical sample geometries such as liquid metals or multi-layer samples can be analyzed using this method.

Thank you very much for your attention !