LINSEIS

THERMAL ANALYSIS

LASER FLASH ANALYSIS

LFA 1000
1250
1600

Since 1957 LINSEIS Corporation has been delivering outstanding service, know how and leading innovative products in the field of thermal analysis and thermo physical properties.

Customer satisfaction, innovation, flexibility and high quality are what LINSEIS represents. Thanks to these fundamentals, our company enjoys an exceptional reputation among the leading scientific and industrial organizations. LINSEIS has been offering highly innovative benchmark products for many years.

The LINSEIS business unit of thermal analysis is involved in the complete range of thermo analytical equipment for R&D as well as quality control. We support applications in sectors such as polymers, chemical industry, inorganic building materials and environmental analytics. In addition, thermo physical properties of solids, liquids and melts can be analyzed.

LINSEIS provides technological leadership. We develop and manufacture thermo analytic and thermo physical testing equipment to the highest standards and precision. Due to our innovative drive and precision, we are a leading manufacturer of thermal Analysis equipment.

The development of thermo analytical testing machines requires significant research and a high degree of precision. LINSEIS Corp. invests in this research to the benefit of our customers.
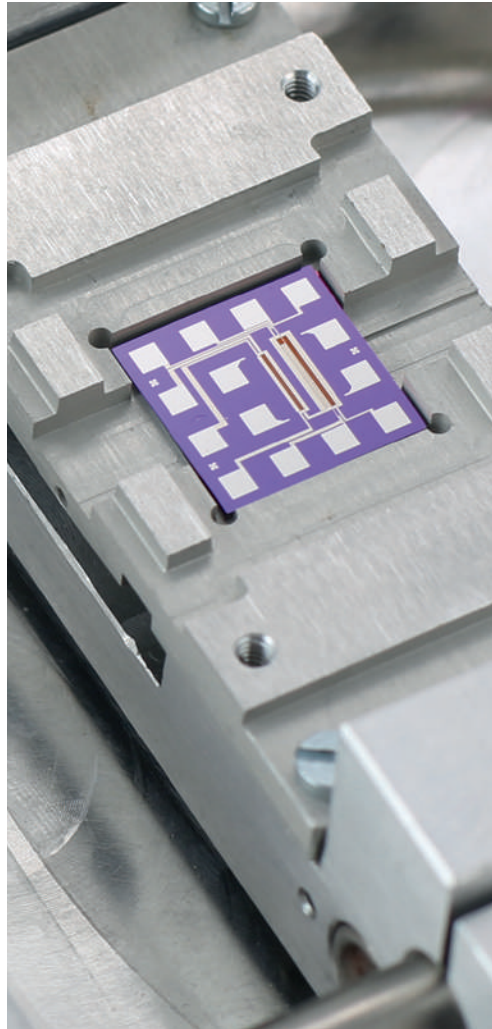
Claus Linseis
Managing Director
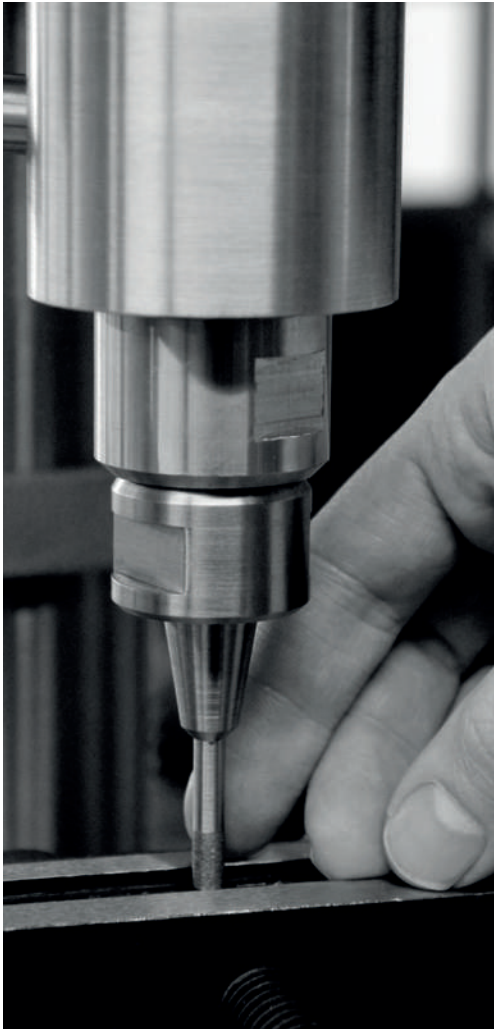
German engineering

The strive for the best due diligence and accountability is part of our DNA. Our history is affected by German engineering and strict quality control.

Innovation

We want to deliver the latest and best technology for our customers. LINSEIS continues to innovate and enhance our existing thermal analyzers. Our goal is constantly develop new technologies to enable continued discovery in science.

THE TECHNIQUE

Information about the thermophysical properties of materials and heat transfer optimization of final products is becoming more and more vital for industrial applications. Over the past few decades, the flash method has developed into a commonly used technique for the measurement of the thermal diffusivity and thermal conductivity of various kinds of solids, powders, pastes and liquids. Application areas are electronic packaging, heat sinks, brackets, reactor cooling, heat exchangers, thermal insulators and many others. Trouble-free sample preparation, small required sample dimensions, fast measurement times and high accuracy are only a few of the advantages of this non-contact and non-destructive measurement technique.

LINSEIS offers a variety of instruments to measure the Thermal Diffusivity/Conductivity. The LFA 1000 Laser Flash series provides a cost effective solution for the temperature range from -125 up to 1600°C.

MEASUREMENT CONCEPT

The sample is positioned on a sample robot, which is surrounded by a furnace. For the measurement, the furnace is held at a predetermined temperature and a programmable energy pulse irrdiate the back side of the sample, resulting in a homogeneous temperature rise at the sample surface. The resulting temperature rise of the surface of the sample is measured by a very sensitive high speed IR detector. Both, thermal diffusiviy and specific heat can be determined from the temperature vs. time data. If the density (ρ) is identified, the thermal conductivity can be calculated:

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

λ=Thermal Conductivity [W/mK]
α=Thermal Diffusivity [mm^2/s]
Cp=Specific Heat [J/g·K]
ρ=Density [g/cm^3]

Principle oF LFA measurement

Temperature rise on sample surface
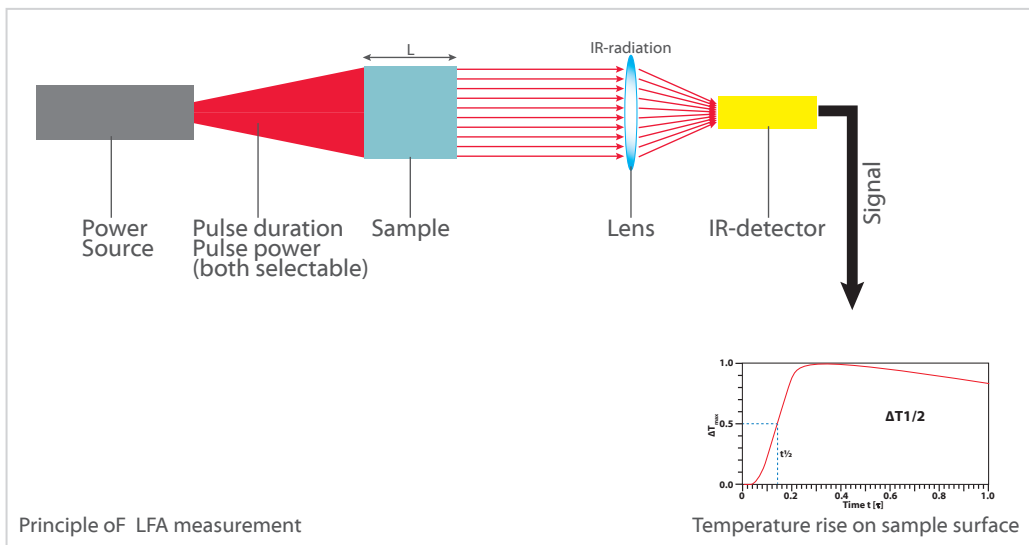
Calculation of thermal diffusivity

- Determine the baseline and maximum rise to give the temperature difference, ΔT_{max}
- Determine the time required from the initiation of the pulse for the rear face temperature to reach $\Delta T_{\frac{1}{2}}$. This is the half time, $t_{\frac{1}{2}}$.

- Calculate the thermal diffusivity, α, from the specimen thickness, samples hight L squared and the halftime $t_{\frac{1}{2}}$, as follows:

$$\alpha=0.13879 \, L^2/t_{\frac{1}{2}}$$

L=Sample height
$t_{\frac{1}{2}}$=Half time rise
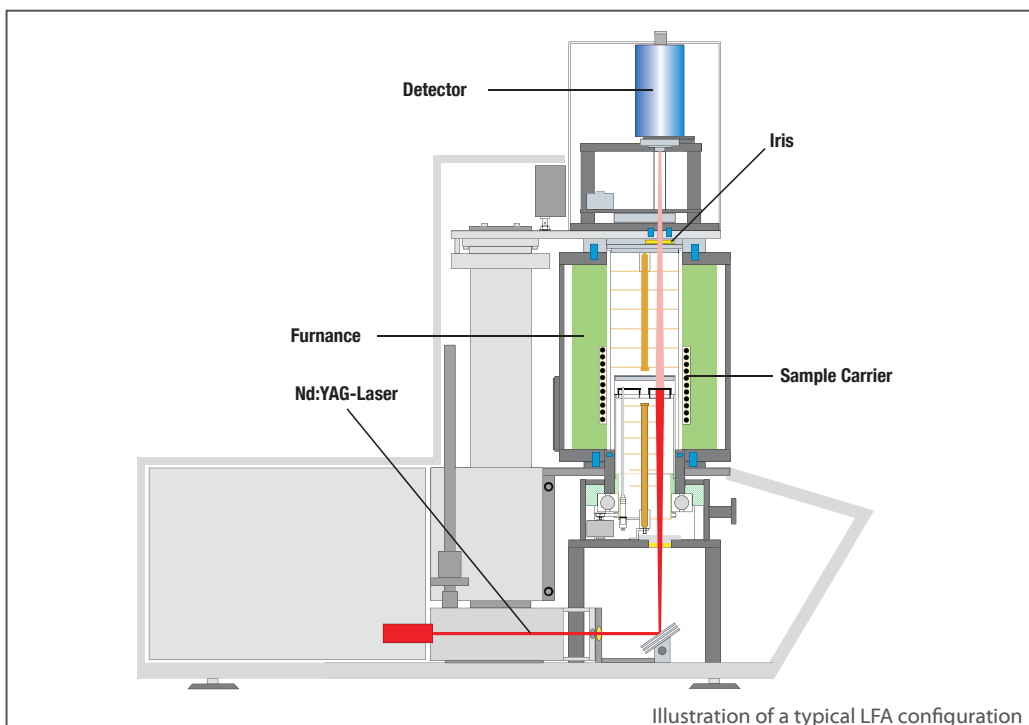α=Thermal Diffusivity

ABSOLUTE METHOD

The method used is an absolute measurement technique (for thermal diffusivity), hence there is no need to calibrate the system. The LFA 1000 Laser Flash operate in agreement with natio- nal and international standards, such as ASTM E-1461, DIN 30905 and DIN EN 821.

SYSTEM DESIGN

The vertical arrangement with sensor on top, sample in the middle and Laser Flash sour- ce on the bottom ensure easy handling and best possible measurement results. The pulse energy is adjustable in the range of 0.05 to 25 Joule/pulse. In addition the pulse duration can be adjusted. Due to this flexibility all kinds of demanding samples (thin film or ultra-low thermal conductivity) can be analyzed.

Detector

Iris

Furnance

Nd:YAG-Laser

Sample Carrier

Illustration of a typical LFA configuration

FUTURE UPGRADES

LINSEIS is offering an unparalleled modular system design. It is possible to upgrade the temperature range (exchangeable furnaces) and the detector (InSb/MCT). This enables the user to start with a cost effective solution and upgrade the system whenever the budget allows or the measurement task requires it.

DETECTORS

The system can be either equipped with an InSb detector or with a MCT detector , covering the complete temperature range from sub-ambient up to 1600°C. Both are easily user exchangeable.

An automatic LN_2 refilling accessory with Dewar can be ordered for prolonged measurement cycles.

ENVIRONMENTAL OPTIONS

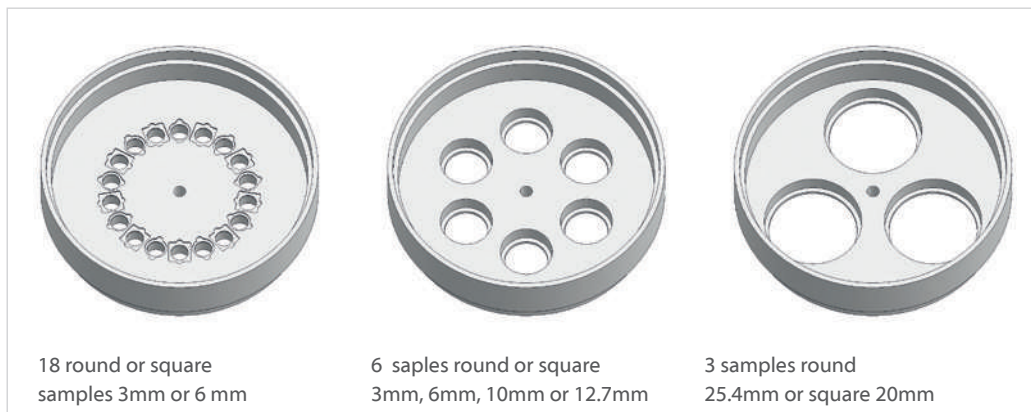The instrument can be operated under defined atmospheric conditions. It is either possible to attach a vacuum pump, in order to minimize heat loss effects or to attach an additional gas dosing systems to measure under specific atmospheres.
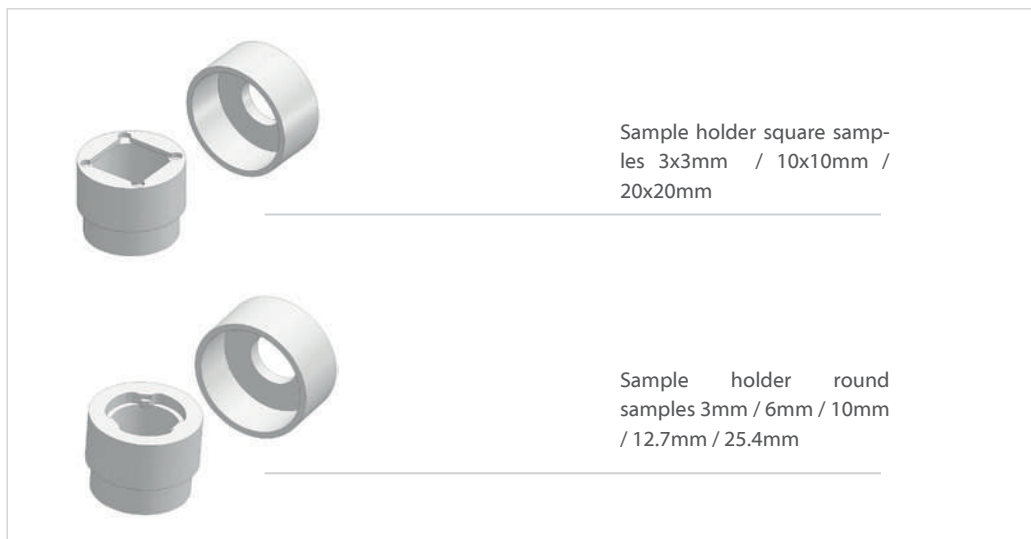
UNMATCHED SAMPLE THROUGHPUT

Highest throughput in the market. The combination of sample robot and i nfrared furnace allows unbeaten measurement turnaround time. A typical measurement for up to 18 samples takes only a few hours.
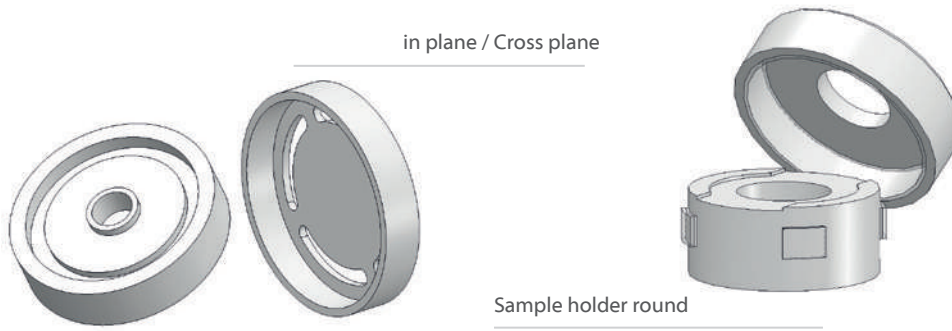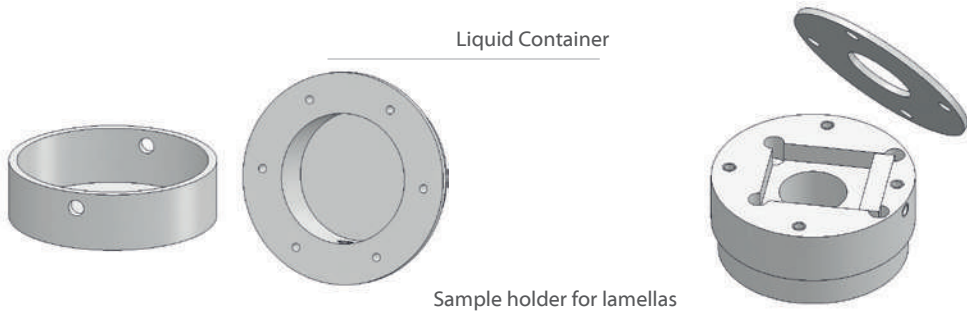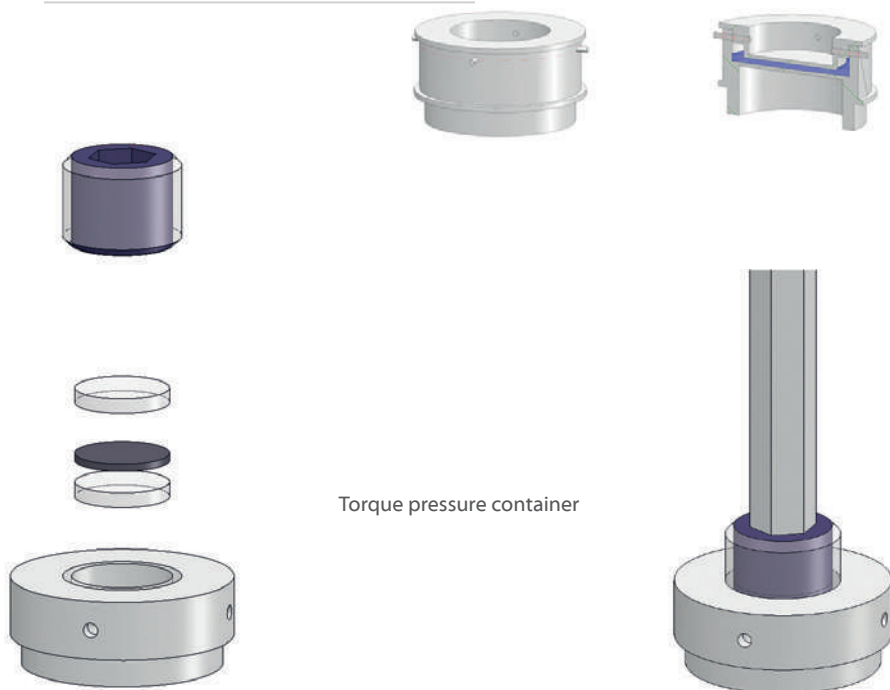
Sample carriers

18 round or square
samples 3mm or 6 mm

6 saples round or square
3mm, 6mm, 10mm or 12.7mm

3 samples round
25.4mm or square 20mm

Sample holder

Sample holder square samples 3x3mm / 10x10mm / 20x20mm

Sample   holder   round samples 3mm / 6mm / 10mm / 12.7mm / 25.4mm

Liquid Container

Sample holder for lamellas

in plane / Cross plane

Sample holder round

Sample holder for liquids and pastes

Torque pressure container

SOFTWARE

- Fully compatible MS®Windows™ software
- Data security in case of power failure
- Safety Features (Thermocouple break protection, power failure, etc.)
- Online and offline Evaluation of current measurement
- Curve comparison
- Storage and export of evaluations
- Export and import of data in ASCII format
- Data export to MS Excel
- Multi - method analysis (DSC TG, TMA, DIL, etc.)
- Programmable gas control

Measurement Software

- Easy and user-friendly data input for temperature segments, gases etc.
- Controllable sample robot
- Software automatically displays corrected measurements after the energy pulse
- Fully automated measurement procedure for multi sample measurements

Evaluation Software

- Automatic or manual input of related measurement data: (density), Cp (Specific Heat)

- Model wizard for selection of appropriate model:

Evaluation Models

- Dusza combined model
- Cowan
- 2/3 layer models
- Parker
- Cowan5
- Cowan 10
- Azumi
- Clark-Taylor
- Degiovanni
- Finite pulse correction
- Heat loss correction
- Baseline correction
- Multilayer model
- Determination of contact resistance
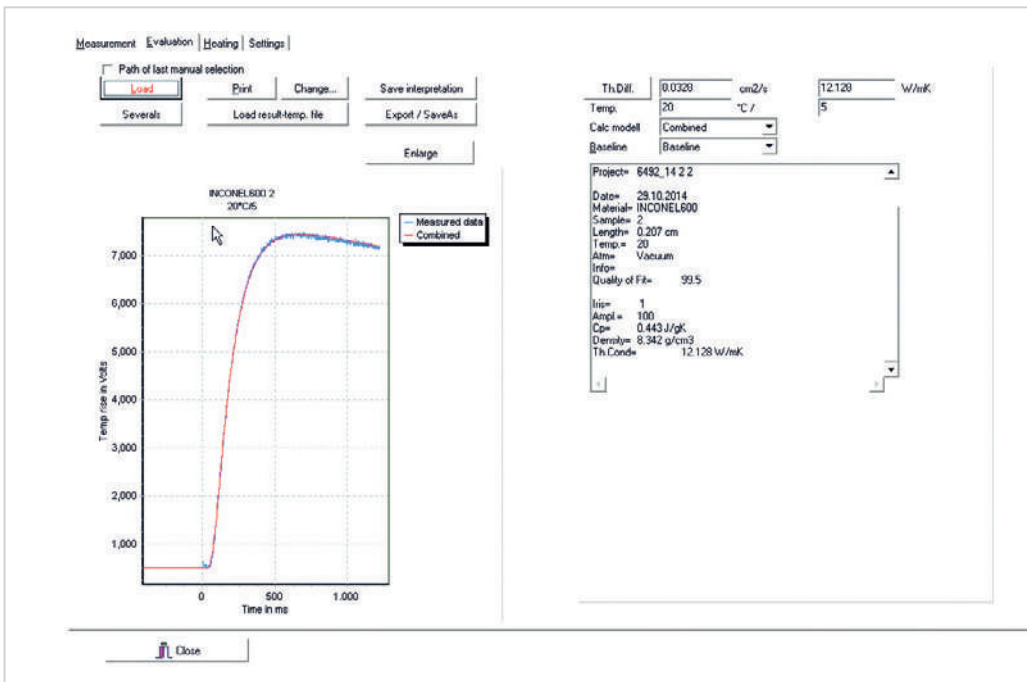
Cp (Specific Heat) determination by comparative method

To calculate the specific heat capacity, the maximum of the temperature raise in the sample is compared to the maximum of the temperature raise of a reference sample. Both, the unknown and the reference sample are measured under the same conditions in a single run, using the sample robot. So, the energy of the laser pulse and the sensitivity of the infrared detector are the same for both measurements. The temperature raise in the sample can be calculated according the following equation:

$$\Delta T = \frac{E}{m \cdot cp}$$

ΔT = Raise in temperature [K]

E = Energy [J]

m = Weight [g]

cp = Heat capacity [$\frac{J}{gK}$]

Since the energy is the same for sample and reference, the Cp of the sample can be calculated according the following equation:

$$cp_{Sample} = \frac{cp_{Reference} \cdot \Delta T_{Reference} \cdot m_{Reference}}{\Delta T_{Sample} \cdot m_{Sample}}$$

To achieve a good accuracy, the absorbability and emissivity of sample and reference must be the same (same coating), and the absolute heat capacity (cp * Weight) of sample and reference must be similar.

DUSZA SOFTWARE COMBINED MODEL

Combined solution of the simultaneous heat loss and finite pulse corrections with the laser flash method
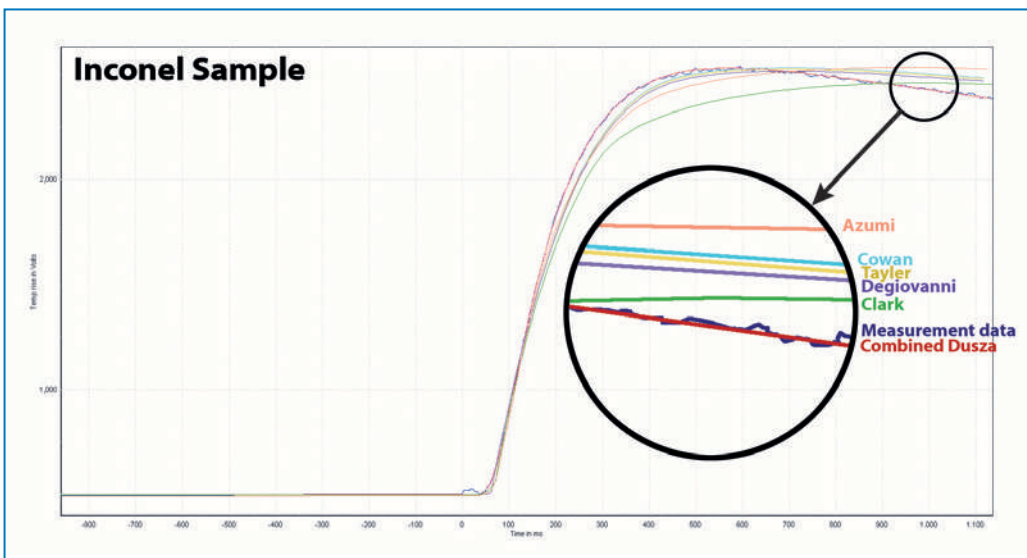

The plot illustrates a measurement on an Inconel sample. The raw data was then evaluated using different common models. It can clearly be observed, that the combined model provides the best fit, resulting in the most accurate measurement result.

Conclusion

The combined model method with nonlinear parameter estimation has been proven for more than 100 samples. In all cases it worked reliably and its results gave the correct adiabatic, finite pulse, and/or heat loss corrected values. The two main advantages of the method are that no operator choice between the different models and correction is necessary, and the fit can be checked by plotting the model curve.

Low Thermal Conductive PMMA
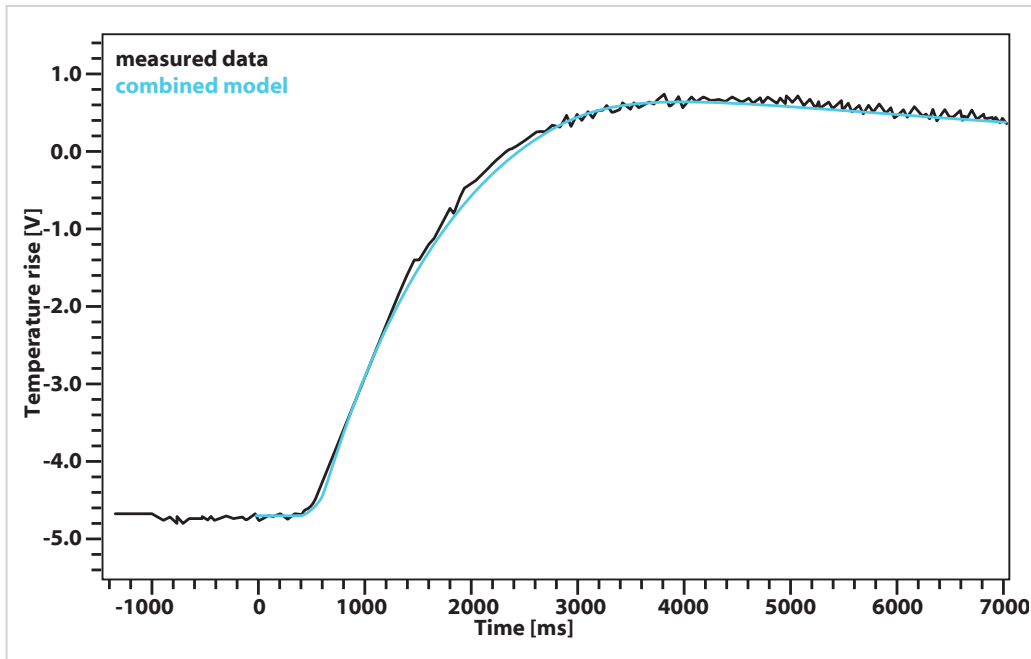
Temperature rise of the PMMA sample (length 0.1005 cm, half maximum time 1218.75 ms). The combined model illustrates a perfect fit to the data points.
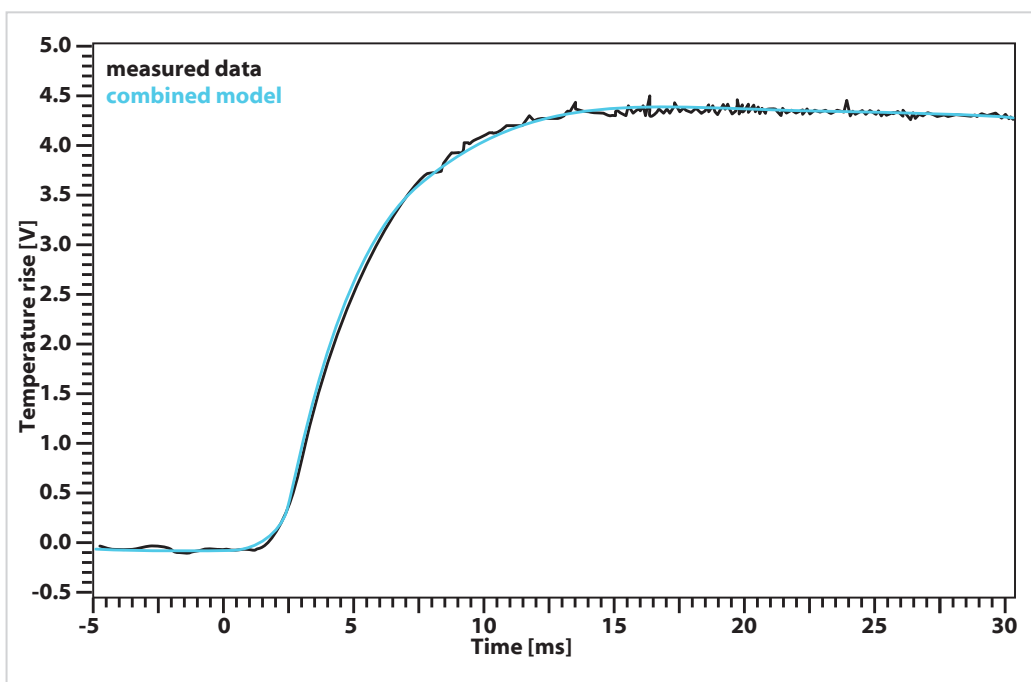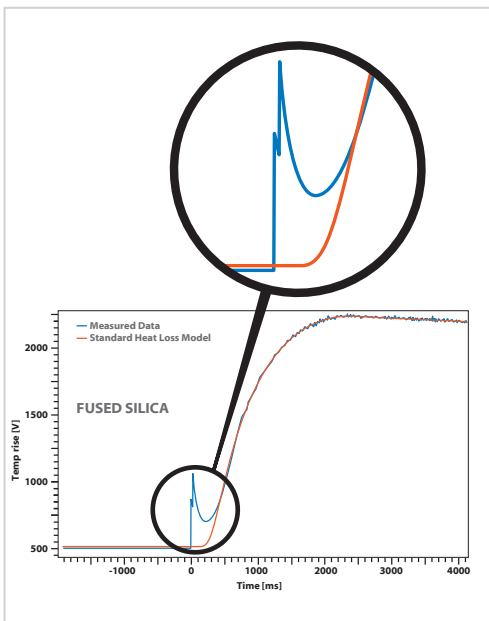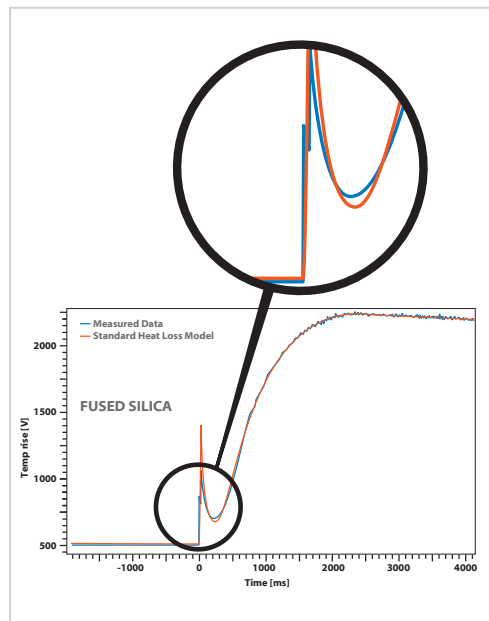
High Thermal Conductive Graphite

Figure 1. Temperature rise of an SiC sample (length 0.1108 cm, half maximum time 4.37 ms). The combined model curve is fitted to the data points.

MODIFIED COMBINED MODEL / SPECIAL MODEL FOR TRANSLUCENT SAMPLES

Standard Heat Loss Model

Modified Combined Model

As illustrated in the graph, the temperature rise for translucent samples, generated by the induced energy pulse, results in an immediate signal increase of the detector. This initial signal has to be considered and corrected, as it distorts the measurement result to a seemingly higher thermal diffusivity. Up to now, existing models could not provide a sufficiently good fit for this immediate temperature rise phenomena. Our unique combined model enables the correction of the sample data and provides an adjusted fit, leading to significantly improved measurement results.

TECHNICAL DATA

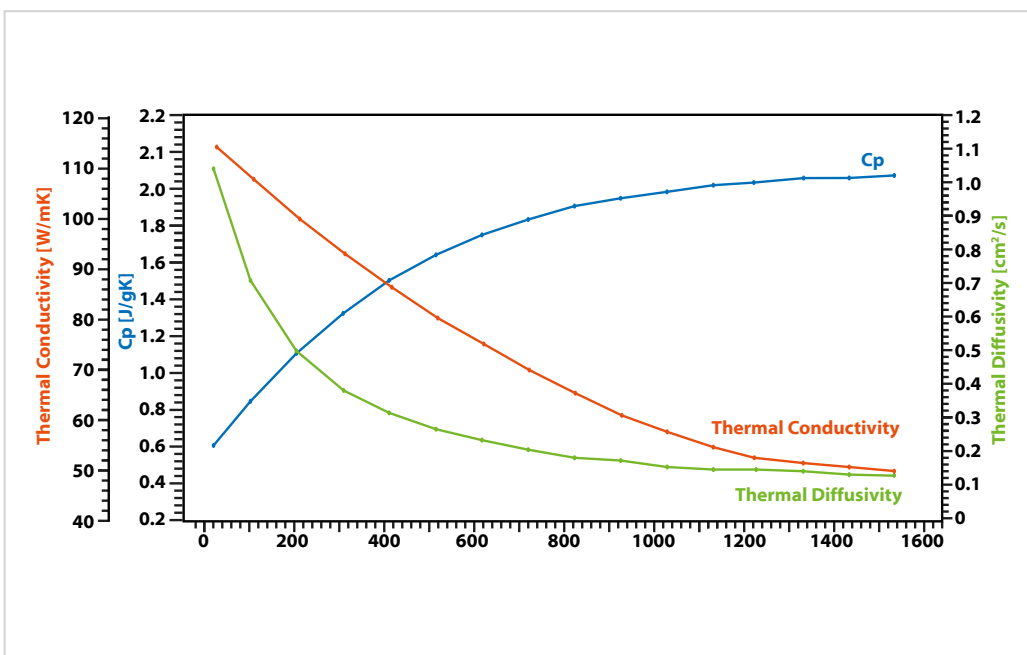|	LFA 1000
Temperature range	−100 up to 500°C RT up to 1250 / 1600°C
Heating rate	0.01 up to 20°C
Thermal Diffusivity	0.01 up to 2000 mm^2/s
Thermal Conductivity	0.1 up to 4000 W/(m·K)
Accuracy	Thermal diffusivity ± 2.4% Specific Heat ± 5%
Repeatability	Thermal diffusivity ± 1.9% Specific Heat ± 3%
Flash source	Laser Nd:YAG 25 J/pulse Pulse width: 0.05 up to 5 ms variable pulse energy: software controlled
IR-detector	InSb: RT up to 1250 / 1600°C MCT: −100 up to 500°C
Atmosphere	inert, oxidizing, reducing, vacuum
Vacuum	up to 10^{-5}mbar
Data aquisition	2 MHz
Gas control	manual or MFC gas dosing systems
Sample holders	for round, square, powders, pastes, laminates, samples and mechanic pressure
Sample numbers	up to 18 samples

APPLICATIONS

Thermal conductivity, thermal diffusivity and specific heat capacity of glass ceramics
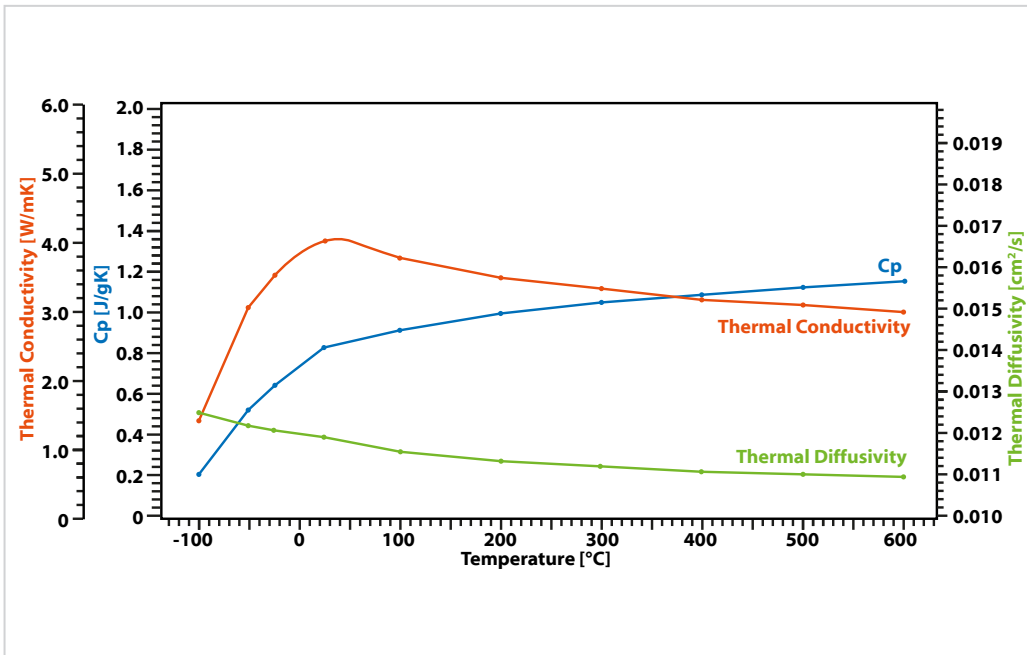
BCR 724, a standard glass ceramic has been measured using LFA 1000. Therefore, a small disc of 1mm thickness and 25mm diameter was cut out of a plate of bulk material and coated with graphite for the measurement. The LFA 500 gives the thermal diffusivity as a direct function of temperature. The Cp data was obtained in a comparative way by measuring a known ceramic standard under the same conditions in a second sample position of the same sample holder. Using this, the thermal conductivity was calculated out of the product of density, specific heat and thermal diffusivity. The result shows a slightly decreasing thermal diffusivity and conductivity while the Cp value increases over temperature.

Thermal conductivity of graphite

A graphite sample has been investigated using the LFA 1000. Thermal diffusivity has been determined directly at several temperature between RT and 1600°C. Specific heat capacity has been determined using a known graphite standard in a second sample position as a reference in the same measurement. The product out of diffusivity, specific heat and density gives the corresponding thermal conductivity.

The result shows a linear decreasing thermal conductivity which is typical and a thermal diffusivity that is showing a plateau above 500°C. The Cp is slightly increasing over temperature.

Thermal diffusivity of glass ceramic
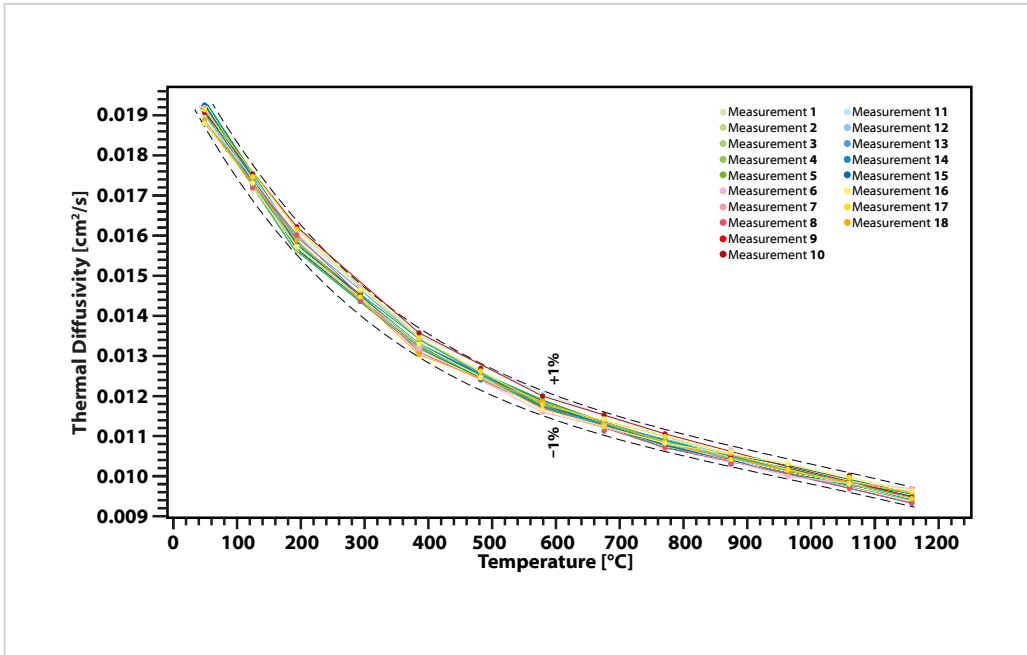
Pyroceram, a glass ceramic trademark of Corning used as a standard material in various applications, has been measured using the LFA 1000 to show the reproducibility of thermal diffusivity values. In total 18 measurements were performed with 18 samples that were cut out of one bulk block. Each sample was measured separately and the result shows a spread in the result that is in a range of +/- 1 % in a temperature range up to 1250°C.

Influence of sample thickness on thermal conductivity accuracy of LFA 1000
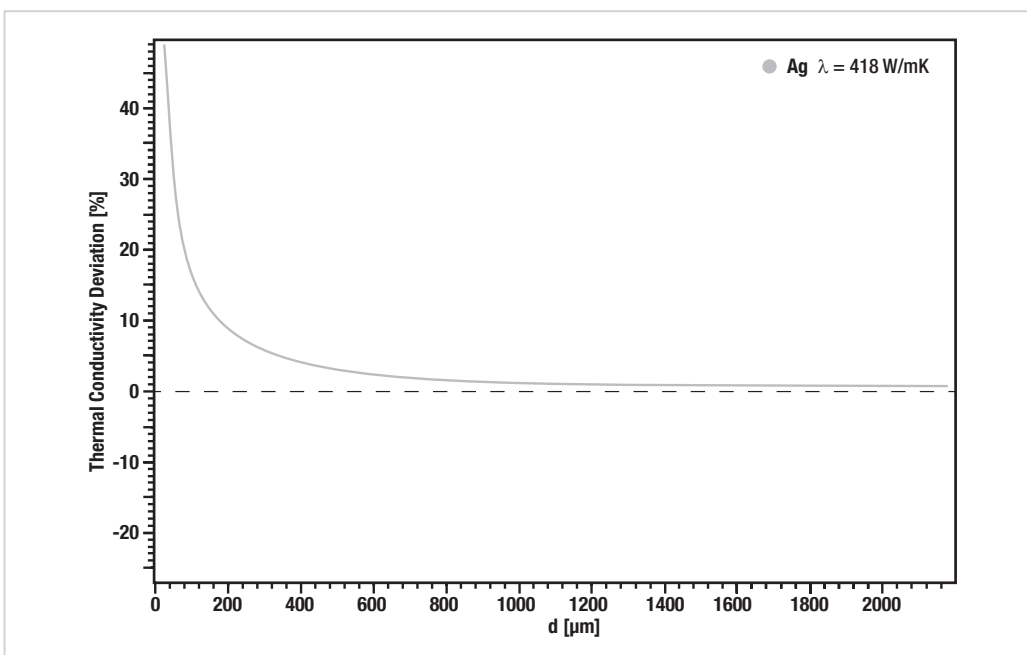
The accuracy of thermal conductivity values depending on sample thickness was investigated using a silver standard.

To get an idea which sample thickness is ideal for the laser flash method, silver samples with different thickness were measured at room temperature. The thermal conductivity was calculated out of thermal diffusivity, density and heat capacity. The scheme shows that the accuracy (deviation from literature value) grows exponential the smaller the diameter gets. The limit for an accurate value is around 200 micrometers. Below that "barrier" the values are dramatically different. However this is not only because of the limitations of the method, but also due to the fact that thin layers show different behavior like bulk materials what can be investigated using the THIN FILM LFA or other thin film techniques.

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Products: DIL, TG, STA, DSC, HDSC, DTA, TMA, MS/FTIR, In-Situ EGA, Laser Flash, Seebeck Effect, Thin Film Analyzer, Hall-Effect

Services: Service Lab, Calibration Service

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