NETZSCH

Proven Excellence.



Laser Flash Apparatus LFA 427

Thermal Diffusivity and Thermal Conductivity between -120°C and 2800°C
Method, Techniques and Applications

Analyzing & Testing

Thermal Conductivity/Thermal Diffusivity

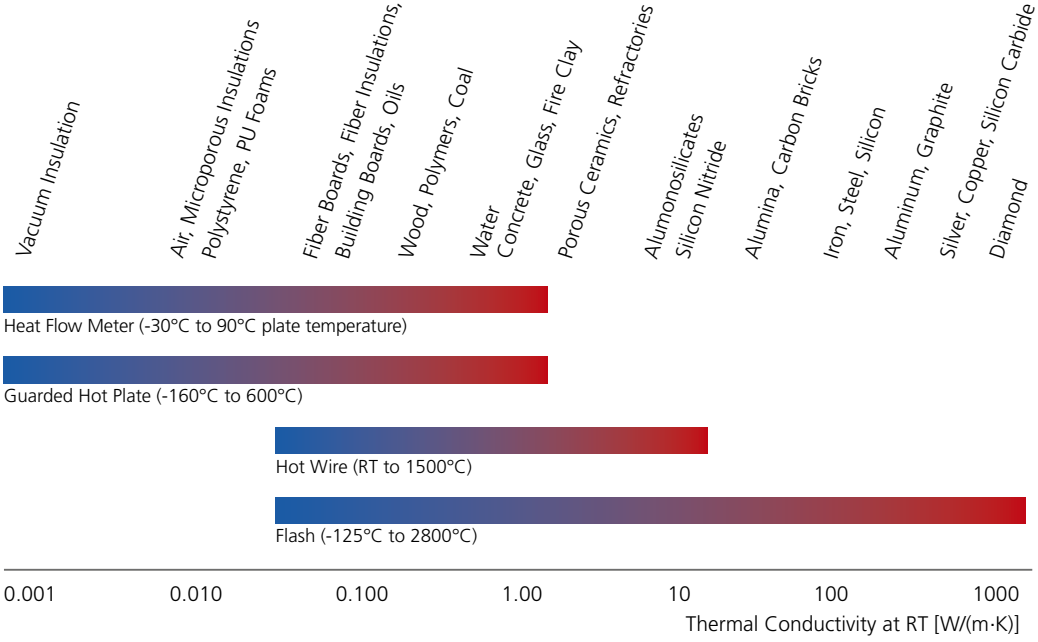
THE LASER FLASH METHOD

How much heat is being transferred, and how fast?

The thermal characterization of highly conductive materials at cryogenic and moderate temperatures – or of ceramics and refractories at elevated temperatures – is of paramount interest in today's milieu of analytical challenges. Many questions can only be answered when two fundamental thermal properties are precisely known: diffusivity and conductivity. One accurate, reliable and elegant solution to this is offered by the Laser Flash method, which allows for addressing questions typically arising in heat transfer processes such as:

- Determining how quickly an aluminum ingot solidifies
- Assessing how quickly the ceramic components of a catalytic converter heat up
- Figuring the temperature gradient in a ceramic brake during use
- Selecting the correct heat exchanger material for the thermal control of a processor

Over the past three decades, NETZSCH has led the way in this technology, expanding our application range to cover a span now extending from -120°C to 2800°C. We never stop innovating, anticipating, and meeting our customers' needs. Once again, true to our tradition of excellence, the LFA 427 has set the industry standard.



The Laser Flash (LFA) technique is a fast, non-destructive and non-contact method for determining thermal diffusivity and specific heat capacity.

Principle

The sample is mounted on a carrier system which is located in a furnace. After the sample reaches a predetermined temperature, a burst of energy emanating from a pulsed laser is absorbed on the front face of the sample, resulting in homogeneous heating. From the resulting temperature excursion of the rear face measured with an infrared (IR) detector, thermal diffusivity and, if a reference specimen is used, specific heat capacity are both determined. Combining these thermophysical properties with the density value allows for calculation of the thermal conductivity as follows:

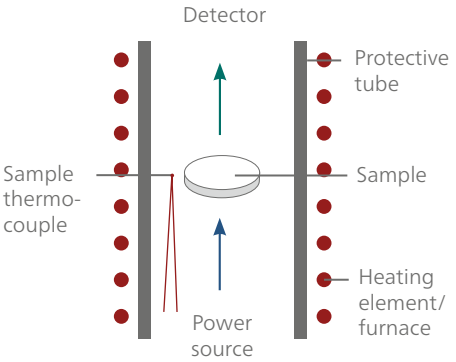
$$\lambda(T) = a(T) \cdot c_p(T) \cdot \rho(T)$$

where
λ = thermal conductivity [W/(m·K)]
a = thermal diffusivity [mm^2/s]
c_p = specific heat [J/(g·K)]
ρ = bulk density [g/cm^3].

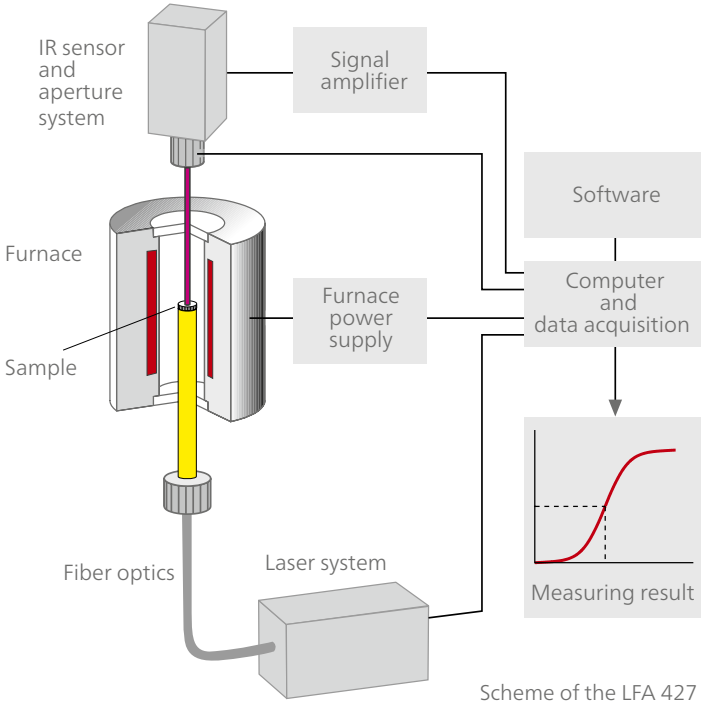
The thermal diffusivity is computed by the software using data on time and relative temperature increase. For adiabatic conditions, the thermal diffusivity (a) is determined by the simple equation:

$$\alpha = 0.1388 \frac{l^2}{t_{0,5}}$$

where
l = sample thickness
$t_{0.5}$ = time at 50% of the temperature increase.



Flash Technique



Scheme of the LFA 427

Elegant Accuracy

The elegance of the LFA method lies in its accurate and direct measurement of time and relative temperature increase. In contrast, methods which attempt to measure the absolute quantity of laser energy absorbed by the sample and the resulting absolute temperature increase can be troublesome.

LFA 427

Definitive Progress in Laser Flash Technology

Ultimate flexibility from subambient temperatures to 2800°C



LFA 427 with power supply P8 incl. laser system

Made in Germany

Optimum Setup for a Broad Range of Temperatures and Specimen Dimensions

A vertical setup with the laser system located on the bottom, the specimen in the center and the detector on top is very sensitive and especially advantageous if high temperatures are applied or larger samples are used. The short distance to the surface of the specimen results in an increase in sensitivity and a reduction of the signal-to-noise ratio.

A Single Instrument for Tests between -120°C and 2800°C

Only the furnace and detector need to be changed. No additional footprint is necessary. The liquid nitrogen (LN_2) controller of the low-temperature furnace is optimized for fast temperature stabilization with reduced LN_2 consumption.

Effectiveness – Double Hoist for Low Thermal Mass Furnaces

The furnace is located on a motorized hoist for easy and safe operation. An optional double hoist allows for the simultaneous connection of two furnaces and detectors. Temperature stabilization is rapid thanks to the low thermal mass of all of the furnaces.

Optimum Laser Power Helps Prevent Specimen Damage

Due to the short distance between the sample and detector, the laser power can be reduced to a minimum. For most applications, 5 J to 8 J is sufficient (only a few mK temperature increase!). Typical problems due to the overheating of specimens during a measurement can be avoided and the risk of chipped sample coatings or other damage to the sample is minimized.

Patented Pulse Mapping

The LFA 427 includes an independent beam mapping system for beam characterization. At least 1000 points are recorded for describing the shape of the pulse, resulting in a sharp and defined pulse width.

Comprehensive Software with a Variety of Models

The software includes countless models and mathematical corrections that can be combined with one another as desired.

VERTICAL SETUP

VARIOUS SAMPLE HOLDERS

FURNACE MADE OF NON-POROUS MATERIAL

VACUUM-TIGHT TO 10^{-5} MBAR

MEASUREMENTS IN GRAPHITE-FREE ATMOSPHERE

SPECIFIC HEAT DETERMINATION

IN-PLANE TESTS

ONE IR DETECTOR ABOVE AMBIENT

SINGLE PYROMETER FOR TESTS TO 2800°C

SENSITIVE MCT DETECTOR FOR SUBAMBIENT TESTS

PATENTED PULSE MAPPING

VARIABLE LASER ENERGY

SOFTWARE-CONTROLLED LASER ENERGY REDUCTION

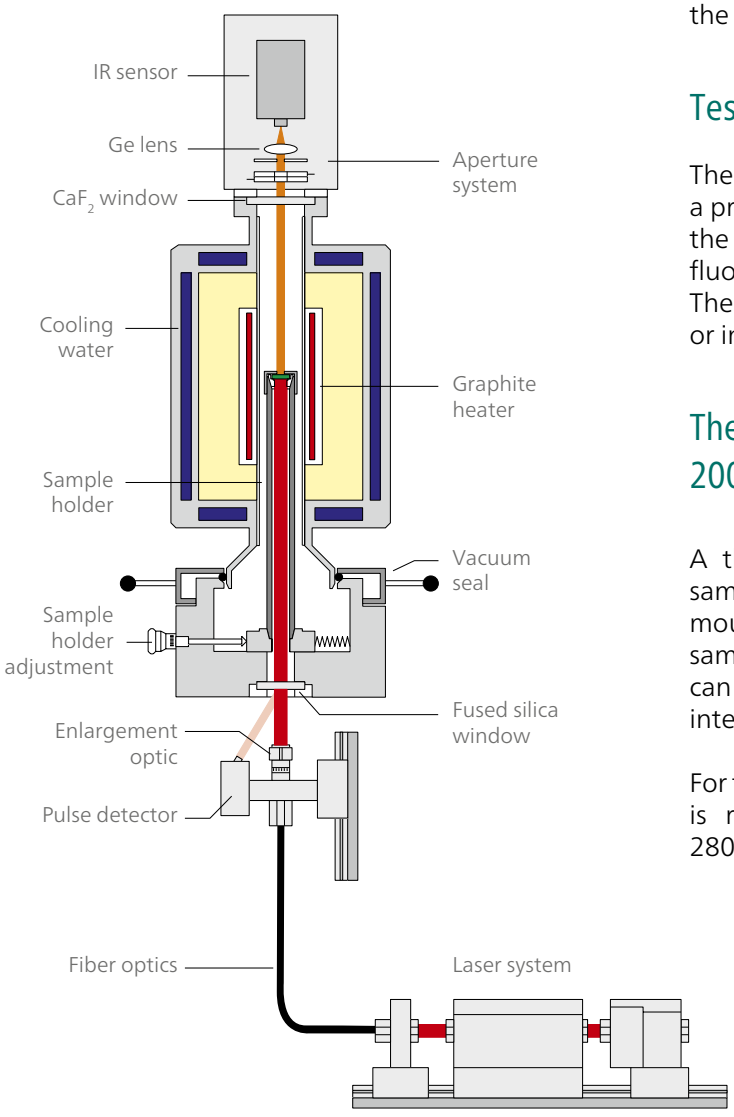
AUTOMATIC ENLARGEMENT OPTICS FOR LASER SPOT ADJUSTMENT

INTEGRATED DATABASE

Trendsetting Technology

The NETZSCH Laser System

- Easy servicing
- Optimized pulse shape
- Short pulse length
- Optimized pulse energy



The Proven Hardware Design

The laser system is connected to the measurement part by a shielded glass fiber. The beam emerges from the outlet of the fiber optics. A recipient block is located above the laser optics. The tube-shaped sample holder and its adjusting device are mounted on this block. A motor-driven hoist raises and lowers the furnace.

Tests under High Vacuum

The furnace is separated from the sample chamber by a protective glass carbon tube. The top and bottom of the sample chamber are sealed by means of calcium fluoride and fused silica windows, respectively. Therefore, tests can be conducted under high vacuum, or in static or dynamic inert gas atmospheres.

Thermocouple and Pyrometer Operation to 2000°C / 2800°C

A thermocouple measures the temperature of the sample (-120°C to 2000°C). This thermocouple is mounted laterally on the sample carrier tube at the same height as the sample. Different thermocouples can be used, depending on the temperature range of interest.

For the highest temperature system, the thermocouple is replaced with a wide-range pyrometer (RT to 2800°C).

The Laser System Built In-House

The Nd:Glass laser system, self-developed and built in-house, has a maximum pulse energy of 25 J and a uniform pulse width over the entire pulse form between 0.1 and 1.5 ms. This produces sharp and defined peaks with negligible tailing. The software-controlled power output can be easily adjusted to the required application.

Laser Beam Enlargement

The emission wavelength of the solid-state laser amounts to 1054 nm (infrared range). The pulse width of the laser is variable. An integrated laser beam enlargement system allows for homogeneous illumination of the specimen (Ø 6 mm to 20 mm).

Completely Enclosed System for Safety

The laser is connected to a sophisticated interlock system allowing it to fire only when the entire system is fully closed. This safety system closes the shutter of the laser system immediately if the hoist is moved or the front door opened, thus preventing release of the laser pulse.

Two Exchangeable IR Detector Systems – A Profitable Investment

Either of the IR detectors (MCT or InSb, both can be equipped with a liquid nitrogen refilling system) is capable of measuring specimens which are highly conductive, inhomogeneous or closed in a container without any consideration to further aspects. Potential problems caused by embedded thermocouples, punctual temperature determination or contact reaction between the specimen and the detector can no longer occur.
As with the furnaces, either detector can be changed out in a matter of minutes.

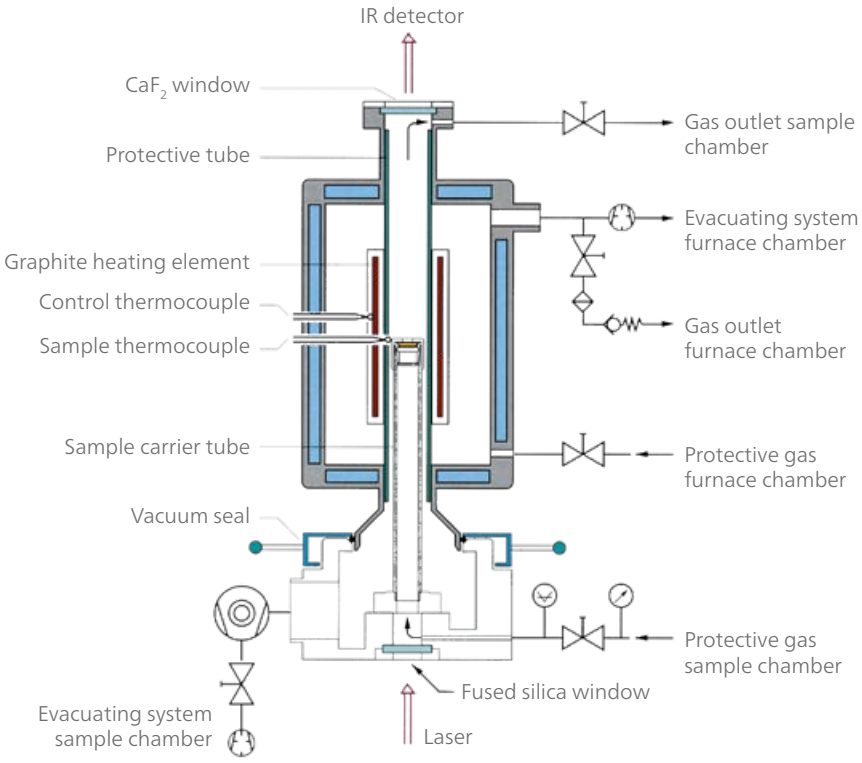
Advanced Laser System – Optimum Pulse Energy and Width for Defined Pulses with Nearly No Tailing Effect

KNOWLEDGE SHEET

High Pulse Energies

The thermal diffusivity of graphites and ceramics is often heavily dependent on temperature. The pulse energy can also significantly influence the experimental data: The mean temperature on the front face of the specimen is shifted toward higher temperatures by the energy input of the laser.
→ If the thermal diffusivity of a material decreases with temperature, calculation of its thermal diffusivity may yield lower values. To obtain the highest level of accuracy, this effect should be taken into account.

Low Thermal Mass Furnaces



Schematic of the 2000°C furnace

From Low to the Highest Temperature Furnaces

A liquid-nitrogen-cooled furnace allows for tests between -120°C and 400°C. A Kanthal furnace enables tests between room temperature and 1300°C, a forced-air-cooled SiC furnace between room temperature and 1575°C. The 2000°C/2800°C furnace consists of a graphite heating element and graphite insulation mounted in a water-cooled housing. All furnaces can be quickly interchanged by the user.

Protective Tubes for Separation of the Sample Chamber and Furnace

The Al_2O_3 tube (max. 1700°C) allows measurements to be conducted under high vacuum or in static or dynamic inert gas or oxidizing atmospheres, while the glass carbon tube (max. 2000°C) allows measurements under high vacuum or in static or dynamic inert gas atmospheres only.

Non-Porous Test Chamber for Pure Test Atmospheres

The thermal masses of both the low- and high-temperature furnaces are lower than those in other conventional thermal conductivity testers, allowing for fast temperature stabilization and short measurement times. The temperature equilibrium is determined from both the sample temperature signal and the stability of the detector signal.

NETZSCH LFA 427 Furnaces			
Temperature Range	**Furnace Type**	**Cooling System**	**TC/Pyrometer**
-120°C to 400°C	Metal	Liquid nitrogen	E
25°C to 1300°C	Kanthal		S
25°C to 1575°C	SiC	Forced air	S
25°C to 2000°C	Graphite	Water-cooled housing	W
25°C to 2800°C	Graphite	Water-cooled housing	Pyrometer

VERSATILITY AND INTER-CHANGEABILITY



For a Clear View – Removable Windows

The top and bottom of the sample chamber are sealed off by CaF_2 and fused silica windows, respectively. The IR detector – directly on top of the furnace – has "visual" contact with the rear face of the specimen, allowing for measurement of the temperature increase. The end of the fiber optics, located directly under the furnace, fires through the fused silica window and heats the front face of the specimen. The windows can easily be removed for cleaning purposes.

Sample Holders for Standard and



Sample holder for testing specimens ("slags") in the liquid state



"Platinum" holder for testing liquids and polymer melts at higher temperatures



Holder for in-plane tests



"Sapphire-S" holder for tests on liquid metals and powdery specimens

Specimen Types and Dimensions

Various sample carriers are available for solid circular or square specimens between 6 mm and 20 mm, including sample carriers for in-plane measurements and tests under pressure as well as ones for special geometries. Of course, sample carriers for tests on laminates, fibers, pastes, liquids, and specimens which crumble or shrink upon heating are also available.

Reference Materials

A number of different reference materials can be supplied in various shapes and diameters. Single reference materials and sets are available. For each material class, a certificate is included.

The special design of the „Sapphire-S" holder has eliminated the requirement of multi-layer analysis!

Special Applications

Sample Holder Systems – Easily Handled

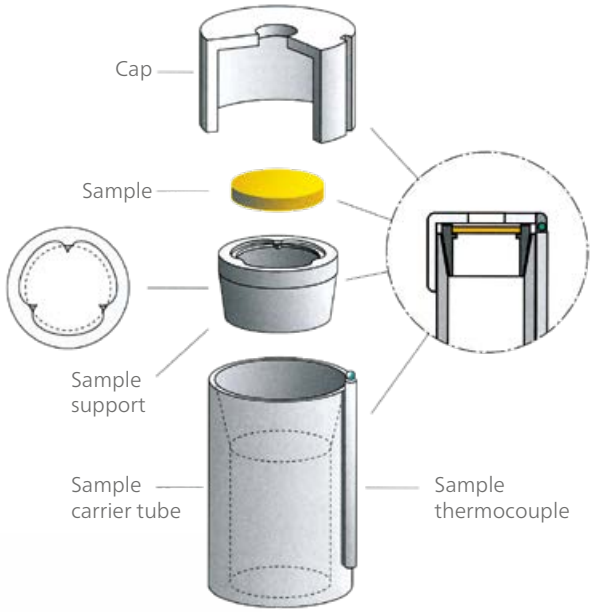
The specimen is in a horizontally stable, well-defined position. Once the furnace has been raised and swung to the side, the sample is directly accessible and can be easily inserted or removed. An Al_2O_3 or graphite sample carrier tube mounted in a metallic adjusting socket carries the sample support, specimen and cap. The sample support is mounted directly into the cone-shaped opening of the carrier tube.

Prongs for Minimized Contact

The sample support holds and centers the specimen on three prongs. This design minimizes the amount of contact between the specimen and the sample support, thereby reducing heat loss and allowing for uniform laser irradiation of almost the entire specimen surface. The inside diameter of the sample support is constructed as a limiting diaphragm below the specimen and corresponds exactly to the diameter of the specimen.

Prevention of Detector Damage

The impinging laser beam with a larger diameter is masked exactly to the specimen diameter. A cap which is placed over both the sample and sample holder prevents the laser energy from impinging directly on the detector. This prevents detector damage as well as signal disturbance.



Sample holder system

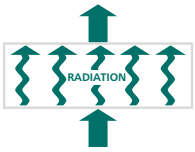


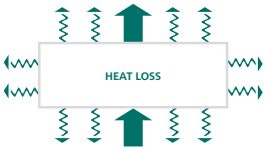
Centering cones (here made of Al_2O_3, shown in white, and graphite, in grey) and caps are available for specimen diameters of 6 mm to 12.7 mm

Software *Proteus*®

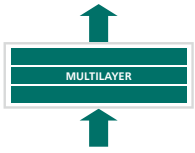
Intelligent Operation – Just a Click Away

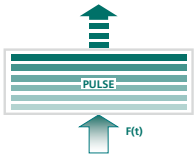




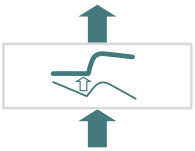






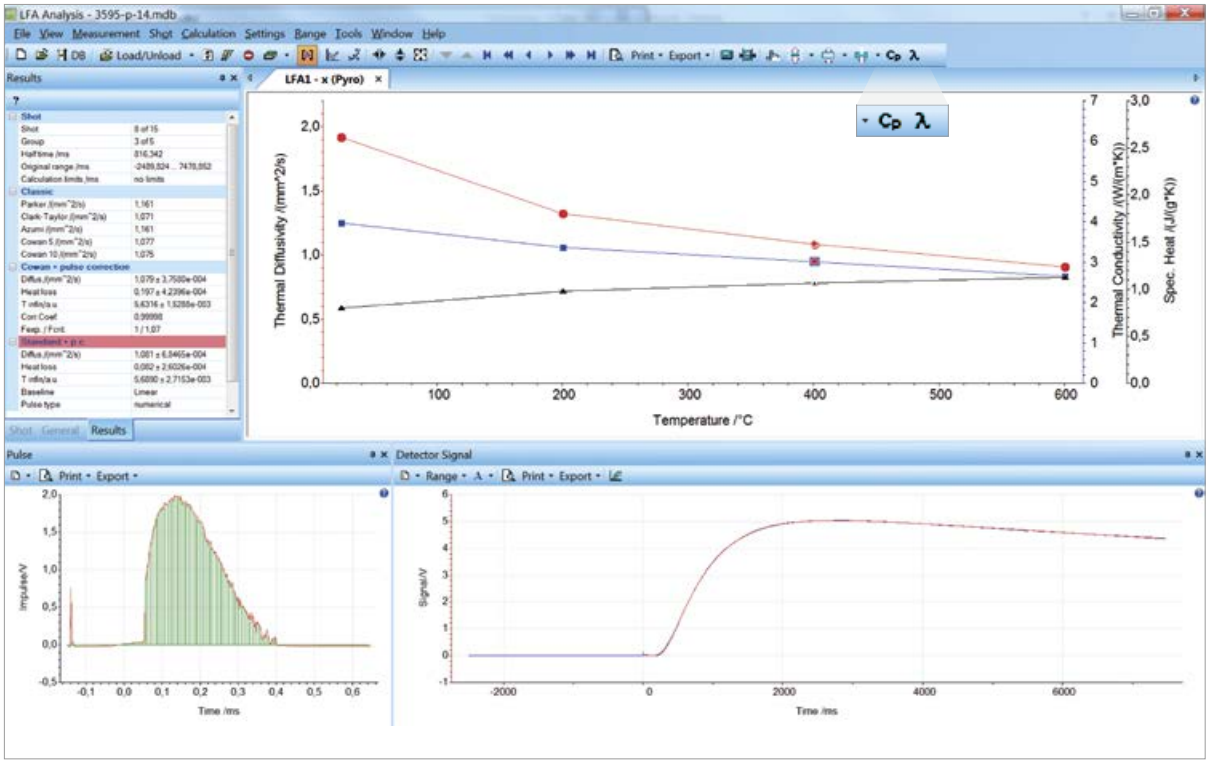






The *Proteus*® software runs on Windows® XP Professional or on Windows® 7 32-/64-bit Professional, Enterprise or Ultimate operating systems. User-friendly menus combined with automated routines make this software very easy to use while still providing sophisticated analysis.
The *Proteus*® software is licensed with the instrument and can, of course, be installed on other computer systems.

General Software Features
Multiple-window technique for clear presentation
Drag-and-drop software functions
Database-oriented saving of series of shots
Fast export routines of all loaded measurements at once
Loading of series of single shots with a preview of parameters and temperature program
Model wizard for selection of the best model
Comparative analysis for up to 32 series of shots from the same database
Ability to average shots at the same temperature level
Definition of an arbitrary number of temperature setpoints and number of shots per setpoint
Determination of the specific heat capacity with the comparative method incl. c_p graph
Integrated database
Determination of the contact resistance in multi-layer systems
Graph of the measurement curves with up to 3 scalable Y axes
Fast zoom function for X and Y segments
Presentation of temperature increase curve, theoretical model curve
Measurement values shown as a Tool-Tip when hovering the mouse over the measurement points
Thermal diffusivity graphs as a function of temperature or time
Combined graph of raw data and theoretical model



Special Software Features

Standard models including
- Improved Cape-Lehman (considers multi-dimensional heat loss and non-linear regression)
- Radiation for transparent and translucent specimens
- Penetration for fibrous and porous specimens

All standard models allow for the combination of heat loss, pulse correction and various baseline types. All factors are freely selectable; R^2-fit and residuals for calculating the Goodness of Fit.

Adiabatic

Cowan

2-/3-layer models (non-linear regression and consideration of heat loss)

Accurate pulse length correction, patented pulse mapping (patent no.: US7038209B2; US20040079886; DE1024241)

Heat-loss corrections

Baseline corrections

In-plane

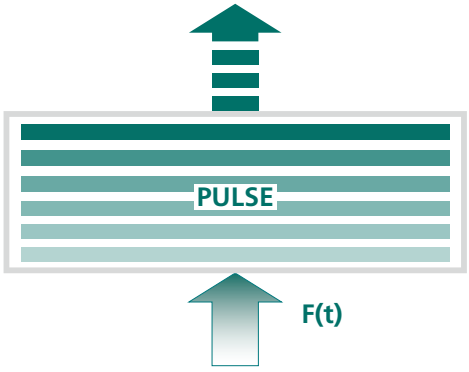
Multiple-shots averaging

Shot approximation via various mathematical functions (polynomials, splines, etc.)

Classical models: Parker, Cowan 5, Cowan 10, Azumi, Clark-Taylor

Calculation Models, Corrections and Mathematical Operations

Software Models, Corrections



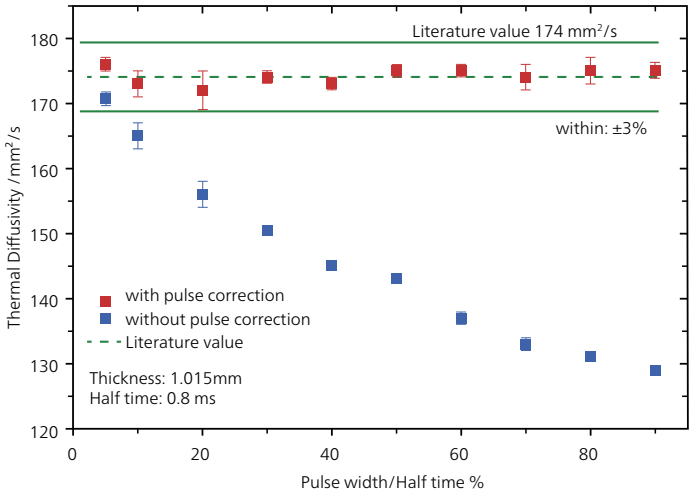
Unrivaled Pulse Correction for Thin and Highly Conductive Materials

Pulse mapping (patent no. US7038209, US20040079886, DE10242741) enables finite pulse correction, improved thermal diffusivity and c_p determination. It considers acquisition of the real laser pulse at each individual measurement and the mathematical description of the real pulse by verifying all calculation models included in the software.

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Laser Pulse Length

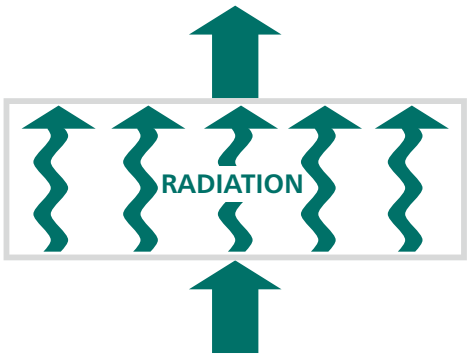
Without pulse length correction, significantly lower thermal diffusivity values are obtained. For short measurement times (thin specimens and/or high thermal diffusivity materials), it is crucial to consider the temporal length of the pulse. This causes a laser-induced shift of the detector signal towards longer half times. According to Parker (page 2), this results in lower thermal diffusivity values.



Measurements on a silver plate comparing the influence of pulse correction on the thermal diffusivity results

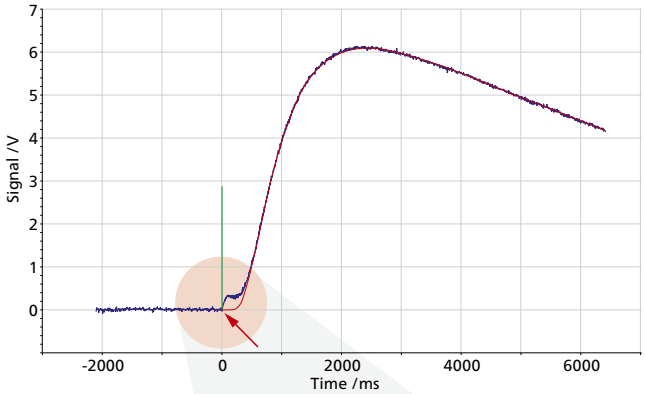
The influence of pulse correction is demonstrated with measurements on a 1.015-mm-thick silver plate at 25°C. This example proves that accurate measurement results are obtained within ± 3% of the literature value when an intelligent pulse correction method is used.

and Wizards

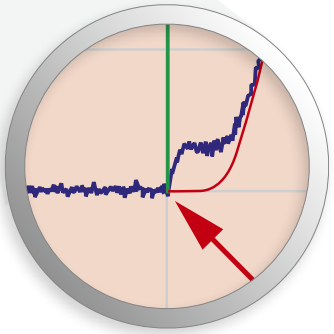


Perfectly Treating Translucent Materials – Transparent (Radiation) Model

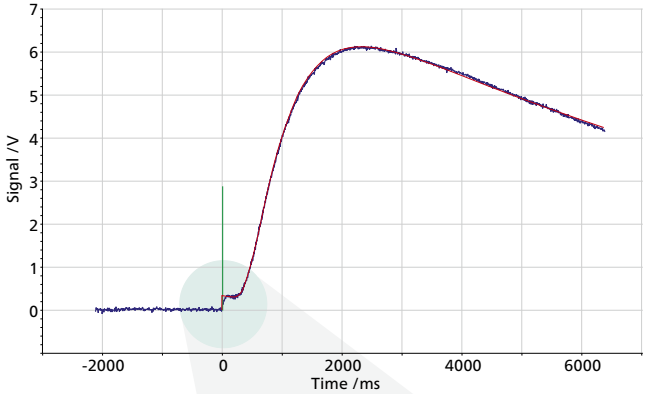
The Transparent Model (patent no. DE102015118856, JP6382912, ZL2016109515017, US10180358) is available for translucent specimens, where the light pulse results in an immediate temperature increase on the rear side of the specimen. Conventional models cannot correctly describe the initial temperature rise. The use of a model (plots below) dedicated to radiation allows for a proper fit (red) of the detector signal (blue).
This measurement on a glass ceramic demonstrates the effectiveness of the radiation model. The improved fit leads to a lower thermal diffusivity value (0.877 mm²/s) than that of the poor fit (0.974 mm²/s) obtained by using the conventional model.



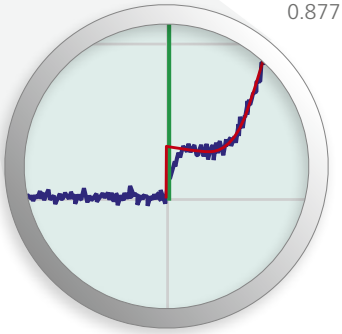
Conventional heat loss model (standard): 0.974 mm²/s

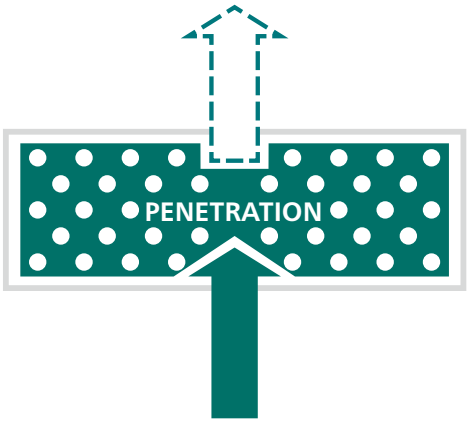


blue: detector signal
red: model fit
green: pulse signal



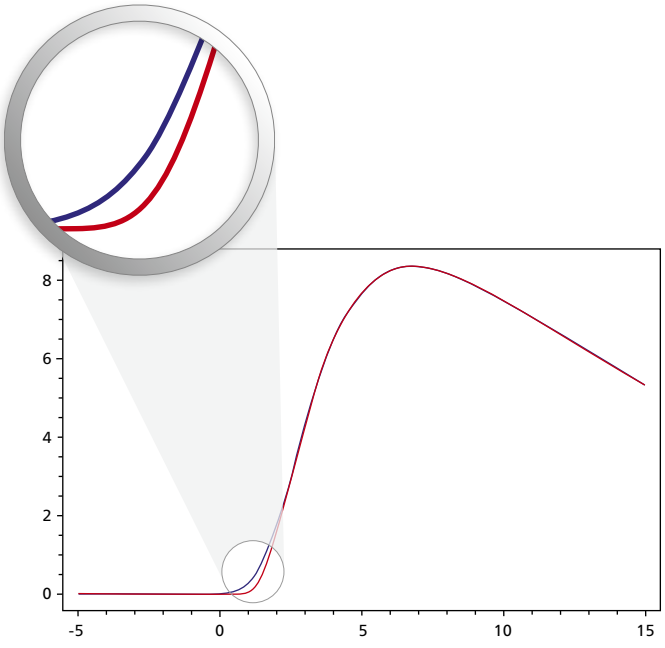
Radiation model: 0.877 mm²/s



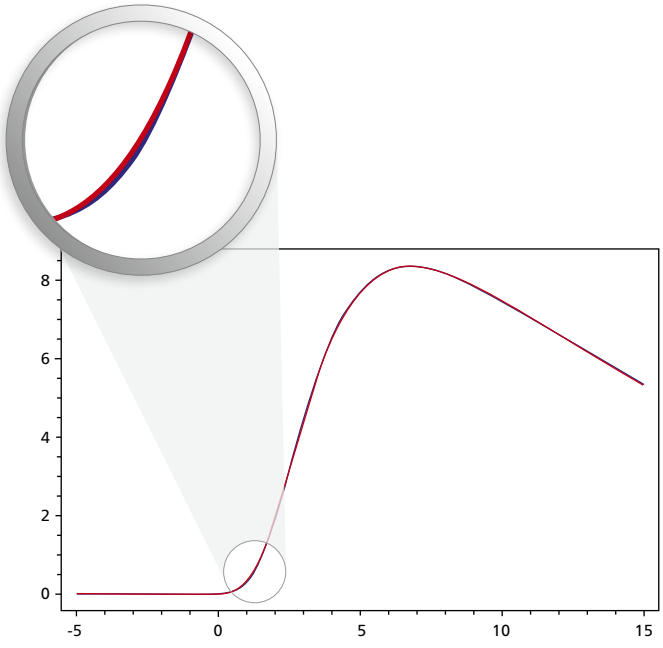


Beam Penetration Model for Porous and Rough Materials

In a slightly porous material or a material with a rough surface, absorption of the pulse energy is no longer limited to the front face, but extends over a thin layer into the specimen thickness. The absorption layer can be considered as the mean free path of photon in the material. This results in an exponentially decaying initial temperature distribution within the specimen.



Laser shot evaluated using the standard model: 0.753 mm²/s



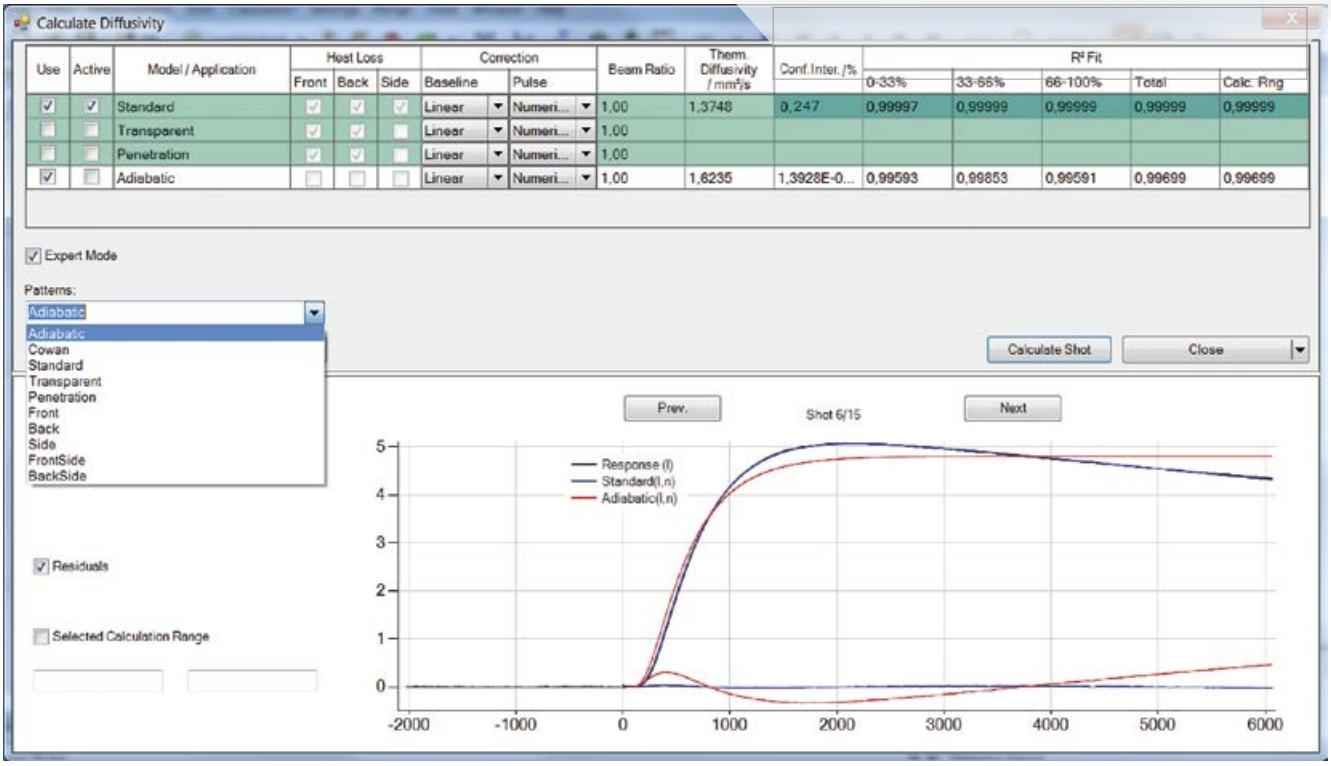
Laser shot evaluated using the penetration model: 0.626 mm²/s



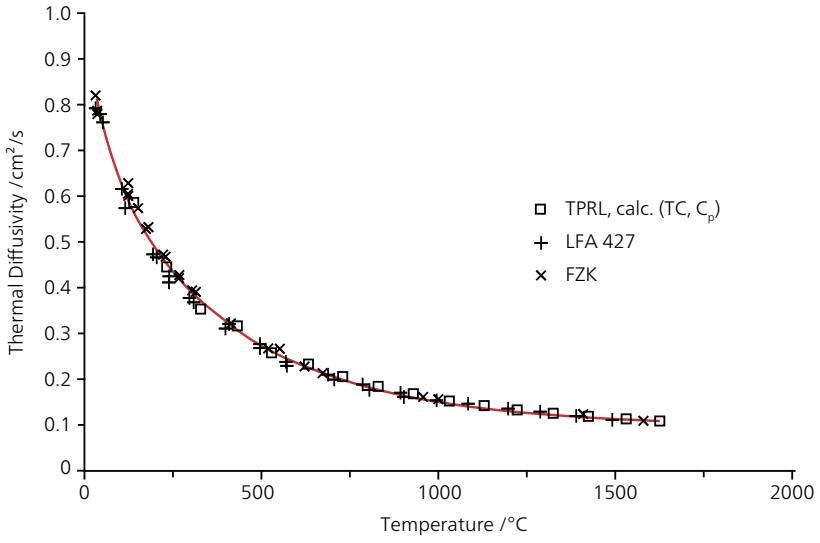
Model Wizard – Best Fit for Best Result

Use of the comprehensive correction models and mathematical operations is facilitated by a smart model wizard integrated into the *Proteus*® software of the LFA system. This powerful model wizard detects the best model fit. By using the wizard to display the data obtained through the selected model, any deviations in the calculated parameters become evident. In the example, the model fit for the standard model (D-2) is almost congruent with the detector curve while for the adiabatic model, large deviations are visible due to the missing heat-loss correction.

	Conf. Inter. /%	R² Fit				
		0-33%	33-66%	66-100%	Total	Calc. Rng
Standard	0,247	0,99997	0,99999	0,99999	0,99999	0,99999
Adiabatic	1,3928E-0...	0,99593	0,99853	0,99591	0,99699	0,99699



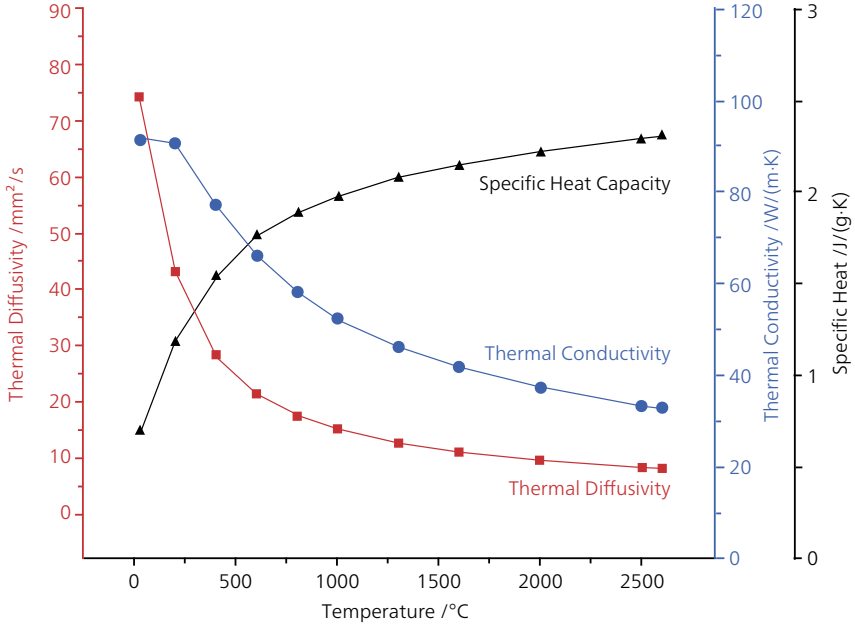
Graphite – At highest temperatures



Comparison of graphite measurements on the same material

Comparison of Measured and Calculated Values

This plot shows a measurement on the common reference material, POCO graphite (carried out at Forschungszentrum Karlsruhe, IMF1). The thermal diffusivity values are compared to values calculated from measured thermal conductivity and specific heat capacity data (obtained by Thermophysical Properties Research Laboratory, Purdue University, USA).



The thermophysical properties by means of LFA 427 (2800°C)

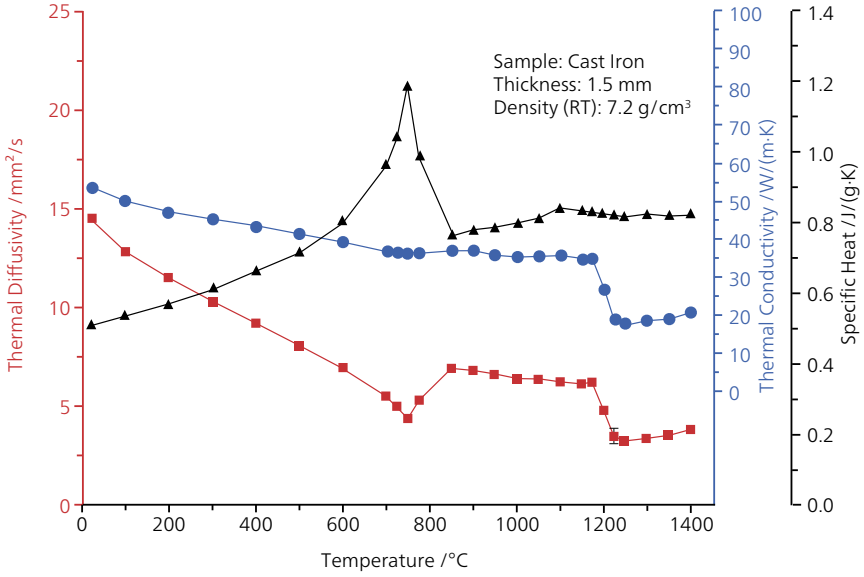
Measurement to 2800°C

This plot shows a measurement on graphite of the thermal diffusivity and specific heat capacity up to the highest possible temperature on the LFA 427. The thermal conductivity was calculated on the basis of the LFA measurement. Only small deviations from literature for thermal diffusivity (a) and specific heat capacity (c_p) are observed.

Tests on metals within the liquid phase

Thermophysical Properties up to the Melt of Cast Iron

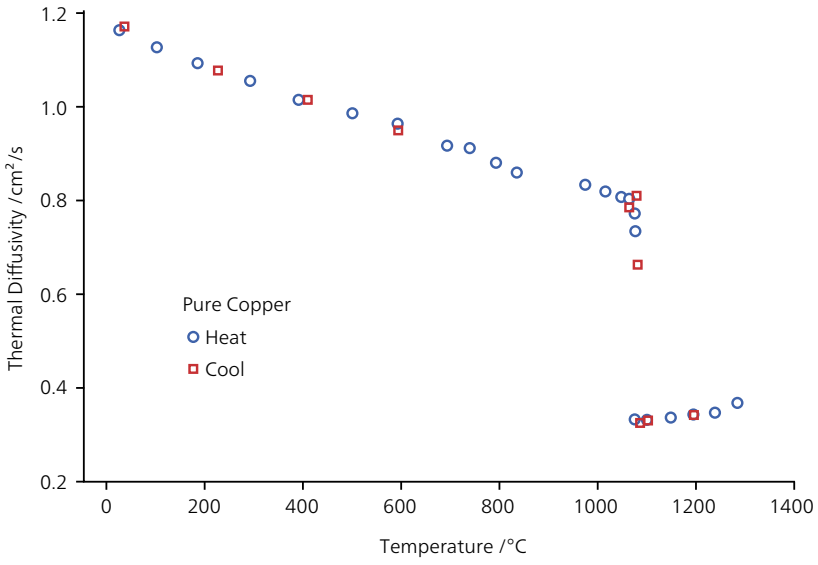
The thermal diffusivity (measured by LFA) and the specific heat capacity (measured by DSC) exhibit typical behavior with peaks at the Curie transition (≈770°C). The thermal conductivity decreases nearly continuously up to the melt. The step above 1150°C represents the phase transition solid → liquid. The reason is that the lattice structure collapses during the phase transition (no heat transfer of phonons in the melt).
This LFA measurement was carried out using the "sapphire" holder. It ensures defined dimensions for the molten material.



Cast iron between RT and 1400°C; sandblasted specimen surface, thickness 1.5 mm, density (RT) 7.2 g/cm³; specific heat capacity obtained by means of DSC.

Pure Copper – From Solid to Liquid

The thermal diffusivity of pure copper was measured for both the heating and cooling cycles. The large change in the thermal diffusivity at approximately 1080°C is primarily due to the change in thermal conductivity upon melting or solidification. The fact that there is almost no difference in the thermal diffusivity values between the heating and cooling cycles indicates that no significant microstructural changes occur. The measured values of the thermal diffusivity for both the solid and liquid regions deviate from those found in literature by less than 2.5%.

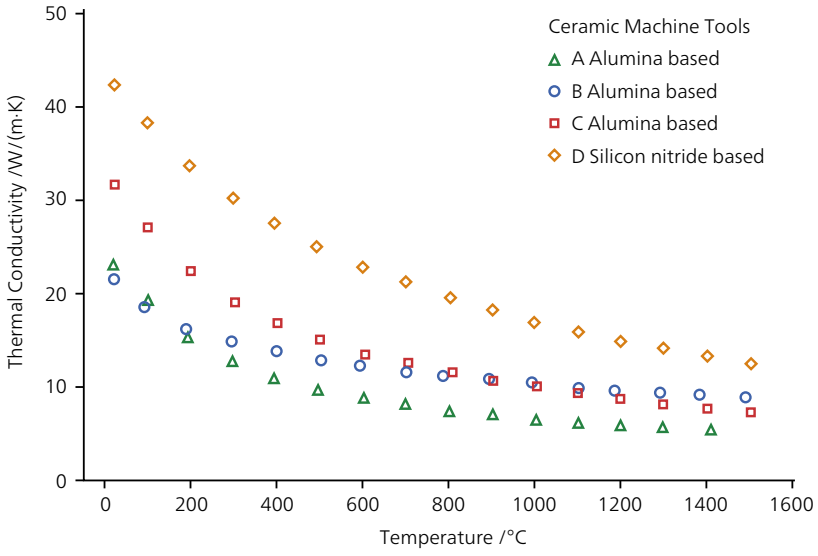


Thermal diusivity of copper during heating and cooling cycles; the measurements were carried out in a special sample holder for liquids

Salient Performance

Machine Tools Based on Different Ceramics

Since ceramics generally have lower thermal shock resistance than metals, the temperature gradients which develop in the tools during use are of paramount importance. As a result, the thermal diffusivity (→ thermal conductivity) of these materials is a primary design consideration. This plot shows the thermal conductivity of four ceramic machine tools. Ceramics A, B and C are all Al_2O_3-based, each with different secondary components, while sample D is Si_3N4- based. The higher thermal conductivity of the Si_3N_4 allows it to be liquid-cooled, while samples A and B must be used without liquid cooling. Sample C can be liquid-cooled at lower temperatures.

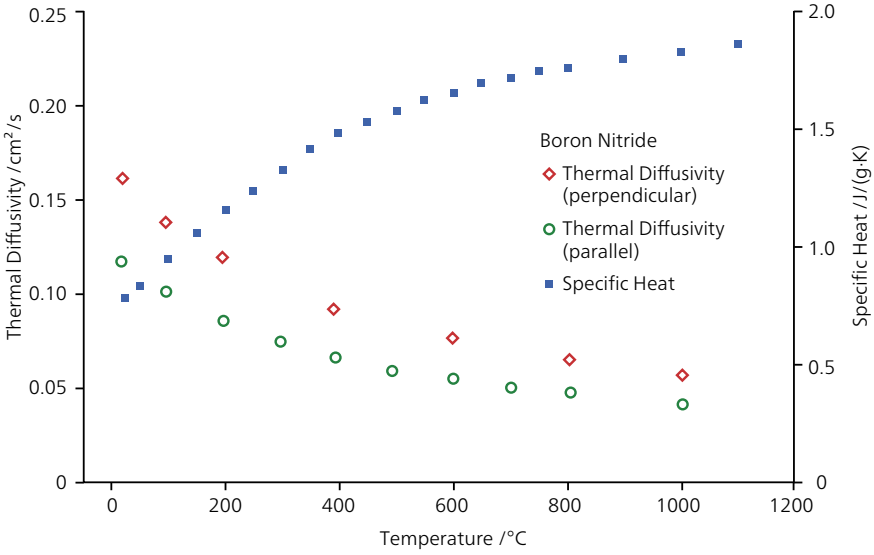


Thermal conductivity of ceramics. Here, the thermal diffusivity and specific heat capacity for all application examples were measured with the LFA 427 and DSC 404 *Pegasus*, respectively. The thermal conductivity values (see page 3, top) were computed from the thermal diffusivity, specific heat capacity and bulk density data.

Ceramics have replaced metals in many applications due to features such as increased machining speed, better wear and chemical resistance

Boron Nitride

Pure hexagonal boron nitride is a high-temperature insulating and soft material similar to graphite; in fact, it is also called "white graphite". This plot depicts the specific heat capacity and thermal diffusivity of hot pressed boron nitride (bulk density ≈ 2.1 g/cm³). Because of its structure, the thermal diffusivity parallel to the direction of press is lower than that in the perpendicular direction. This is, of course, the expected behavior.



LFA measurement between RT and 1000°C on specimens perpendicular and parallel to the press direction. The graphite-like structure of boron nitride causes different thermal diffusivity behavior in the two directions.



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LFA 427 in a mockup glove box

LFA 427 Glove Box Version

The glove box version of the vacuum-tight LFA 427 can be used in a glove box either with a slight negative pressure or in one that works with a low over-pressure. In addition, the LFA 427 can be placed in a hot cell. For these cases, the LFA 427 has separate electronics and can be handled by using gloves or manipulators; feedthroughs allow for the connection of cables, water and gas supply, vacuum pump, etc.

LFA 427	
Furnaces	▪ -120°C to 400°C (metal, connection to liquid nitrogen cooling possible) ▪ 25°C to 1300°C (Kanthal) or to 1575°C (SiC furnace) or 2000°C/2800°C (graphite furnaces)
Heating rates	0.01 K/min to 50 K/min (furnace-dependent)
Isothermal stability	0.02 K/min
Laser system	▪ Nd:Glass; wavelength 1054 nm ▪ Variable energy up to 25 J/pulse and pulse width between 0.1 ms and 1.5 ms ▪ Patented pulse mapping for finite pulse correction (patent no.: US7038209B2; US20040079886; DE1024241)
Sensors	▪ MCT (-120°C to 500°C, recommended), LN_2-cooled, optional LN_2 refill system including 35 liter dewar ▪ InSb (RT to 2800°C), optional LN_2 refill system including 35 liter dewar
Measuring range	▪ Thermal diffusivity: 0.01 mm^2/s to 1000 mm^2/s ▪ Thermal conductivity: 0.1 W/(m·K) to 2000 W/(m·K)
Accuracy	▪ Thermal diffusivity: ± 3% (over the entire temperature range, for most materials) ▪ Specific heat capacity: ± 5% (for most materials)
Measurement atmospheres	Inert, oxidizing or vacuum (<2x 10^{-5} mbar; turbo molecular pump)
Specimen dimensions and shapes	Ø: 6 mm, 8 mm, 10 mm, 12.7 mm*, 20 mm; 0.1 mm to 6 mm thickness □: 8 mm x 8 mm, 10 mm x 10 mm; 0.1 mm to 6 mm thickness
Sample holder inserts	Al_2O_3 (max. 1700°C), Graphite (max. 2800°C), Pt, Sapphire, Al
Special sample holder systems	▪ Types: liquid organics (incl. low viscosity materials such as water) and liquid metals, fibers, laminates, slags, powders ▪ Methods: testing in-plane, mechanical pressure (“pressure sample holder”)
Reference materials	Various sets and individual reference materials in different dimensions and shapes
Software including calculation and correction models	Each model can be combined with 4 different baseline corrections (incl. shifted baseline) and w/o pulse correction; model wizard, display of detector signal and model fit, data export; various special and extended models
Display of detector signal and model fit	▪ Quality check of the model fit (same plot) ▪ Automatic storage of both curves for each shot
Specialty	Glove box version
Power	15 kW, max. power consumption at 2800°C

* 12.7 mm recommended; additional sample holders upon request

Technical Specifications

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