NETZSCH

Proven Excellence.

Laser Flash Apparatus LFA 457 *MicroFlash*®

Thermal Diffusivity and Thermal Conductivity
Method, Techniques and Applications

Analyzing & Testing

Thermal Conductivity/Thermal Diffusivity

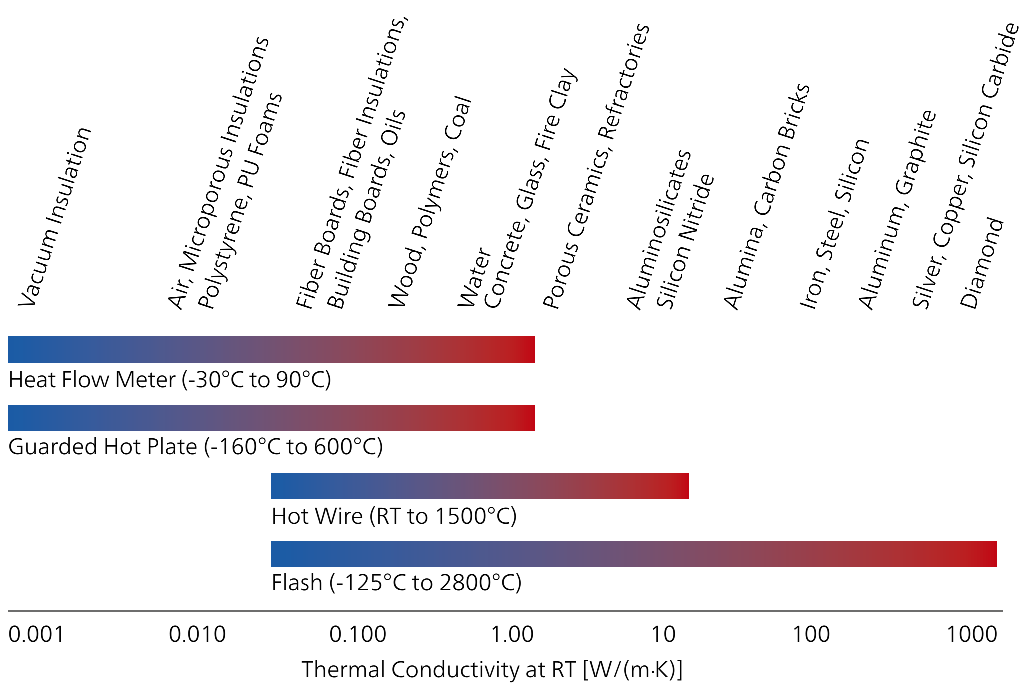
THE FLASH METHOD

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

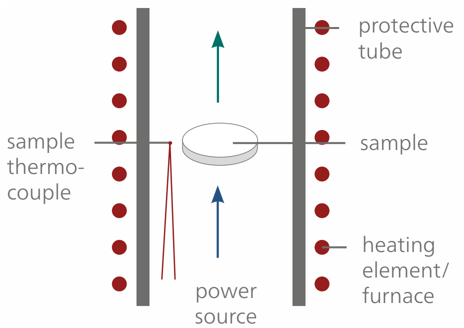
Over the past three decades, NETZSCH has led the way in this technology, expanding our application range to cover a span now extending from -125°C to 2800°C. We never stop innovating, anticipating, and meeting our customers' needs. Once again, true to our tradition of excellence, the LFA 457 *MicroFlash*® 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. The front surface of a plane-parallel sample is heated by a short energy light pulse. 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 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 [mm²/s]
c_p = specific heat [J/(g·K)]
ρ = bulk density [g/cm³].



Flash Technique

This set of thermophysical properties can be the base of development for new materials, process optimization and numerical simulations.

Data yielded by LFA analysis thus allows values for the following properties to be obtained either directly or calculated:

- Thermal diffusivity (a)
- Specific heat capacity (c_p)
- Thermal conductivity (λ) – via calculation
- Thermal effusivity b – via calculation
- k-value – via calculation

The Laser Flash method is an absolute test technique for thermal diffusivity. No calibration is required for the determination of this thermophysical property. Tests at a given temperature generally take only a few seconds. Measurements over the entire temperature range can be done within a few hours (usually three tests per day; morning, afternoon and overnight).

The LFA 457 *MicroFlash®* is based on national and international standards such as ASTM E1461, DIN EN 821-2, DIN 30905, ISO 22007-4 and ISO 18755.

Thermal Conductivity
Thermal Diffusivity
Thermal Effusivity
Specific Heat Capacity
k-Value

Laser Flash – An Efficient Method for the Determination of Thermophysical Properties

LFA 457 *MicroFlash*®

Advancements in Laser Flash Technology

This table-top instrument with small footprint lends tremendous flexibility!

Made in Germany

Temperature-Dependent Tests in a Broad Temperature Range

Two user-interchangeable furnaces are available which are moved with a motorized hoist. The low-temperature furnace comes with a controlled liquid nitrogen cooling device and allows for measurements between -125°C and 500°C, while the air-cooled high-temperature furnace allows for measurements between room temperature and 1100°C.

Broadest Measurement Range

The technique can be used for materials with thermal diffusivities between 0.01 mm^2/s and 1000 mm^2/s (thermal conductivities between 0.1 W/(m·K) and 2000 W/(m·K)).

Interchangeable Detectors to Accommodate Any Future Application

In general, the standard InSb (Indium Antimonide) detector offers the best performance at high temperatures. For the lower temperature range between -125°C and 500°C, the highly sensitive MCT (Mercury Cadmium Telluride) IR-detector is available.

High Accuracy and Repeatability

Measurements on standard materials prove that the thermal diffusivity can be determined with an accuracy of within ±3%; the specific heat capacity can be directly determined with an accuracy of within ±5% for most materials.

LOW-TEMPERATURE FURNACE

VARIOUS SAMPLE HOLDERS

FURNACE MADE OF NON-POROUS MATERIAL

VACUUM-TIGHT BY DESIGN

IN-PLANE TESTS

SPECIFIC HEAT DETERMINATION

PATENTED PULSE MAPPING

CONFORM LASER CLASS 1

0.3 MS LASER PULSE WIDTH

BROAD TEMPERATURE RANGE

VARIABLE LASER ENERGY

BUILT-IN LASER

SOFTWARE-CONTROLLED LASER ENERGY REDUCTION

AUTOMATIC ENLARGEMENT OPTICS FOR LASER SPOT ADJUSTMENT

INTELLIGENT CALCULATION AND CORRECTION MODELS

Trendsetting Technology

Optimum Laser Power in Combination with High-Sensitivity Detector System

The laser system built into the instrument requires no external optical fiber delivery. Due to the small distance between the sample and detector, the laser power can be reduced to a minimum. For most applications, 5 to 8 J is sufficient (a temperature increase of only a few mK!). Thanks to the detector, the sample will not overheat during measurement and the risk of chipped sample coatings and sample damage is minimized.

Laser – Defined Pulse Energy for Optimum Pulses

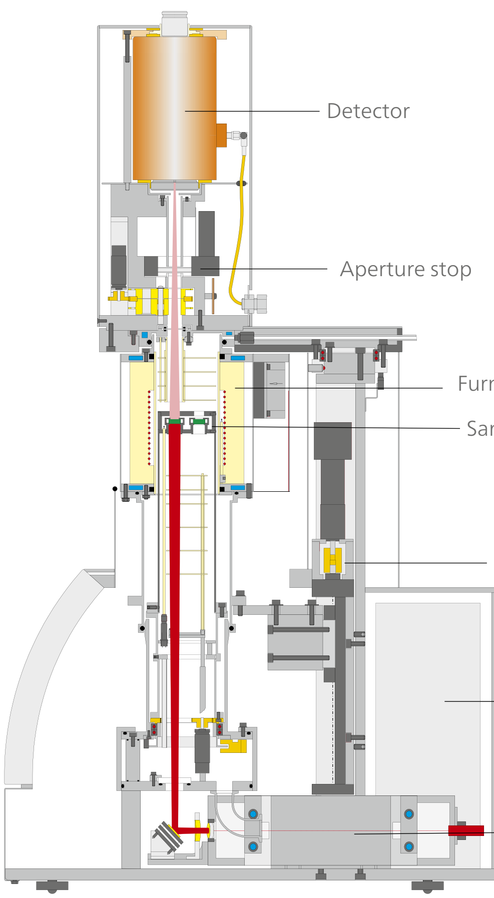
The Nd:Glass has a maximum pulse energy of 18 J and a uniform pulse width over the entire pulse form of 0.3 ms. This produces sharp and defined peaks with negligible tailing. The power output is controlled by the software and can be easily adjusted to the required application. The laser is connected to a sophisticated interlock system allowing the laser to fire only when the entire system is fully closed (Laser Class 1).

Fast Temperature Stabilization in a Pure Test Chamber

The thermal masses of both the low- and high-temperature furnaces are lower than those of 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. The test chamber is made of non-porous material in order to provide a pure test atmosphere.

Laser Beam Enlargement

An integrated laser beam enlargement system allows homogeneous illumination of the sample at different sizes between 6 mm and 25.4 mm.



LFA 457 *MicroFlash®* – 1100°C version

Patented* Pulse Mapping for Finite Pulse Correction and Improved c_p Determination

The acquisition of the real laser pulse at each individual measurement is allowed. The mathematical description of the real laser pulse and consideration in all calculation models pave the way for investigations on thin foils and materials with the highest of conductivities. In addition, the real pulse energy is taken into consideration in the calculation of specific heat to improve accuracy.

* Patent No.: US 7,038,209 B2 from 9/2003; DE 1024241 from 9/2002.

Two Changeable IR Detector Systems – One Profitable Investment

Both IR detectors (MCT and InSb) are capable of measuring specimens which are highly conductive, inhomogeneous or closed in a container without any consideration to further aspects. Problems caused by embedded thermocouples, punctual temperature determination or contact reaction between sample and detector no longer occur. The IR detector systems can be equipped with a liquid nitrogen refilling system including a 35-liter Dewar. Like the furnaces, both detectors can be changed by the operator within minutes.

A very elaborate LFA – from design to execution

ACCESSORIES

Automatic Sample Changer

The integrated motorized sample changer allows measurement of up to three samples at the same time. The sample carriers are arranged on a robust sample carrier tube which rotates during sample change. For large sample sizes, the system can be equipped with a carrier plate for single-sample operation (up to Ø 25.4 mm).

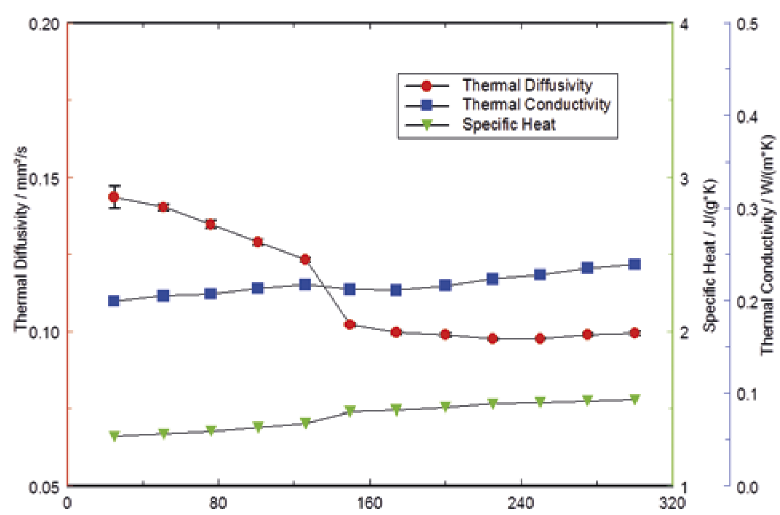
Sample Carriers

Various sample carriers for circular or square solid samples between 6 mm and 25.4 mm are available including sample carriers for special geometries, in-plane measurements and tests under pressure. Of course, we offer sample carriers for tests on laminates, fibers, pastes, liquids, and samples which crumble or shrink upon heating.

Polycarbonate (PC) – LFA Tests in the Liquid Region

Optimization of the molding process of PC by finite element simulations requires knowledge of the thermo-physical properties — even above glass transition (> 140°C) – and thus also requires a sample carrier for molten materials. The thermal conductivity is calculated from the density, temperature conductivity and specific heat capacity. Its slight increase versus temperature is typical for a 100% amorphous material.

The glass transition is clearly visible in the specific heat capacity and in the thermal diffusivity curve. In the thermal conductivity result, this 2nd order transition cannot be seen.



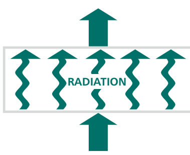
LFA 457 *MicroFlash*®	
Furnaces/Temperature range	■ -125°C to 500°C (helium atmosphere recommended) ■ RT to 1100°C → One instrument and two easily exchangeable furnaces and detectors
Laser	■ Nd:Glass ■ Laser class 1 ■ Wavelength 1054 nm ■ Energy up to 18 J/pulse (variable, software-controlled) ■ Pulse width 0.3 ms ■ Patented pulse mapping (US7038209, DE10242741), for finite pulse correction ■ Automatic enlargement optics for laser spot adjustment (for large samples)
Sensors	■ MCT (-125°C to 500°C, recommended), LN_2-cooled, optional LN_2 refill system ■ InSb (RT to 1100°C), optional LN_2 refill system
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% (for most materials) ■ Specific heat capacity: ± 5% (for most materials)
Repeatability	■ Thermal diffusivity: ± 2% (for most materials) ■ Specific heat capacity: ± 3% (for most materials)
Measurement atmospheres	Inert, oxidizing or vacuum ($<10^{-2}$ mbar)
Automatic sample changer	Up to three samples simultaneously
Specimen dimensions/ shapes*	Ø: 6 mm, 8 mm, 10 mm, 12.7 mm, 25.4 mm; 0.1 mm to 6 mm thickness □: 6 mm x 6mm; 8 mm x 8 mm, 10 mm x 10 mm; 0.1 mm to 6 mm thickness
Reference materials	Various sets and individual reference materials in different dimensions and shapes
Software	Various calculation and correction models, model wizard, display of detector signal and model fit, data export
Utilities	110/230 V, 50/60 Hz, 16 A (one 230 V line is required for the PU), water: 1 liter/week, LN_2: 2 liters/day
Instrument dimensions	Width: 570 mm, depth: 550 mm, height: 880 mm

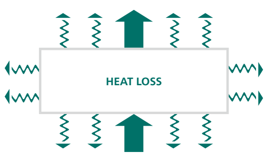
* 12.7 mm recommended; further sample holders upon request

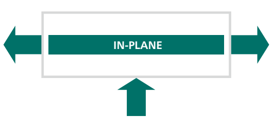
Technical Specifications

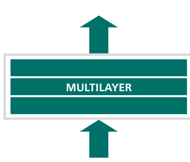
Software *Proteus*®

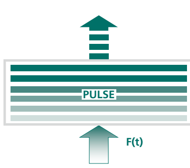
Intelligent Operation – Just a Click Away

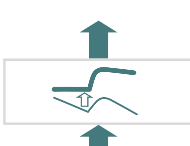






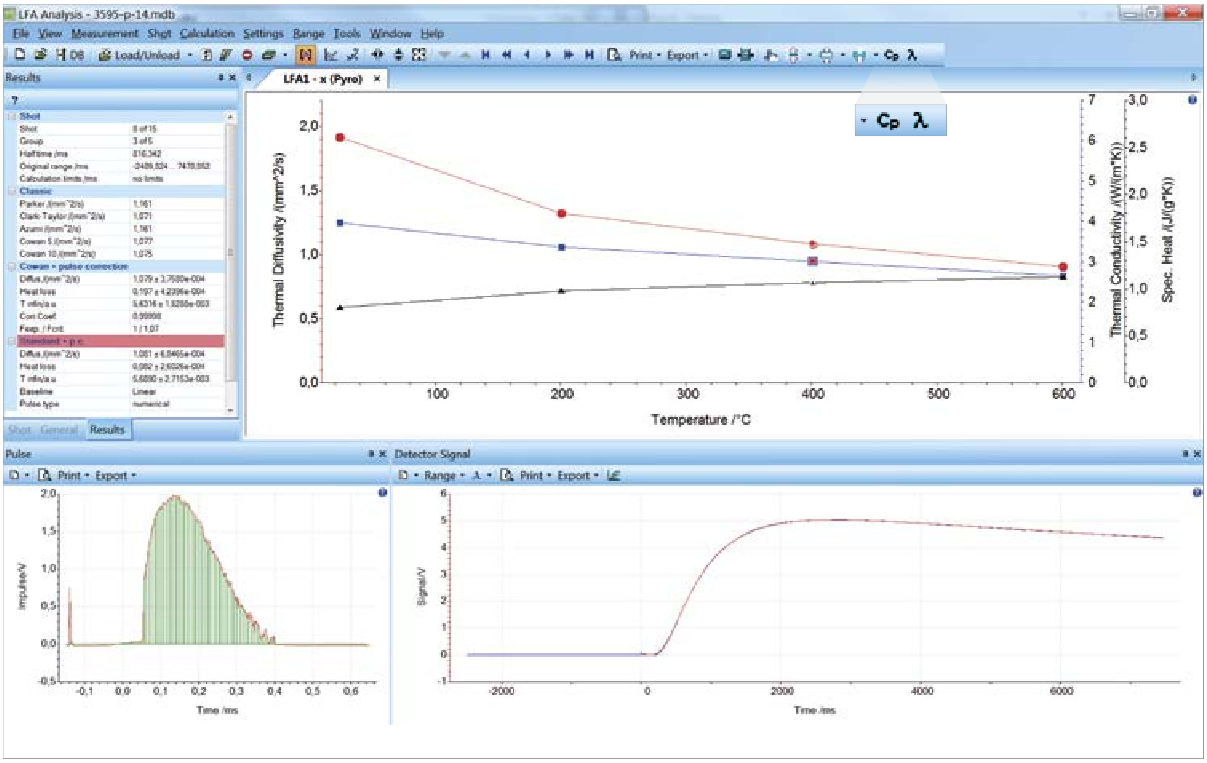






The *Proteus*® software runs on Windows® XP Professional or on Windows® 7 32-/64-bit Professional, Enterprise or Ultimate operating systems. User-friendly menus combined with automated routines make this software very easy to use while still providing sophisticated analysis.
The *Proteus*® software is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

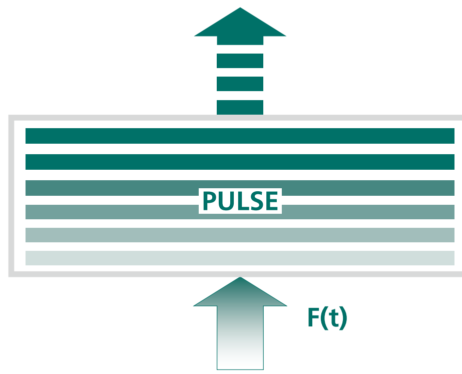
General Software Features
Multiple-window technique for clear presentation
Drag-and-drop software functions
Database-oriented saving of series of shots
Fast export routines of all loaded measurements at once
Loading of series of single shots with a preview of parameters and temperature program
Model 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



Calculation Models, Corrections and Mathematical Operations

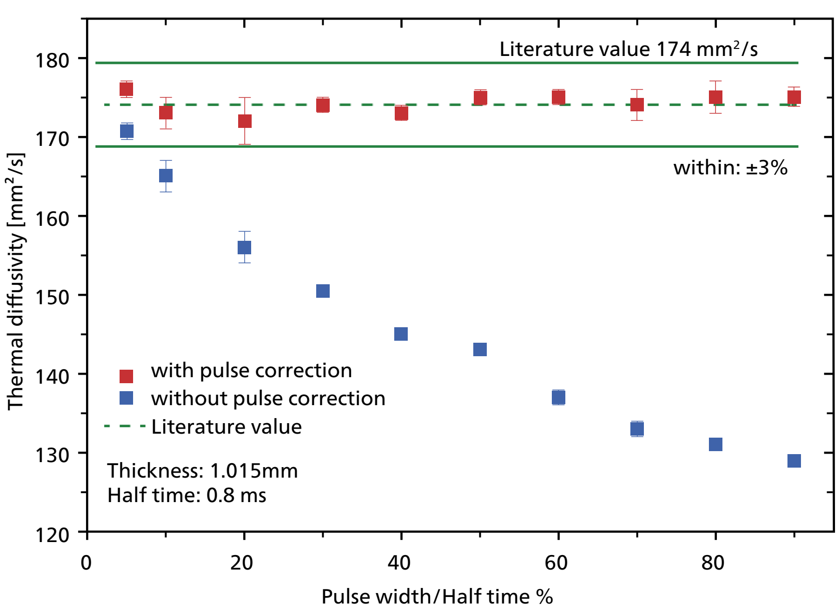
Special Software Features
Standard models including ▪ Improved Cape-Lehman (considers multi-dimensional heat loss and non-linear regression) ▪ Radiation for transparent and translucent specimens ▪ Penetration All standard models allow for the combination of heat loss, pulse correction and various baseline 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

Software Models, Corrections



Unrivaled Pulse Correction for Thin and Highly Conductive Materials

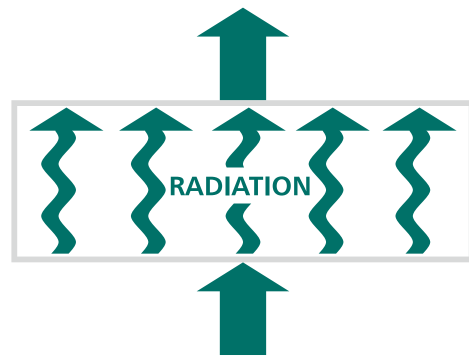
Pulse mapping (patent no. US7038209, US20040079886, DE10242741) enables finite pulse correction, improved thermal diffusivity and c_p determination. It considers acquisition of the real laser pulse at each individual measurement and the mathematical description of the real pulse by verifying all calculation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



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

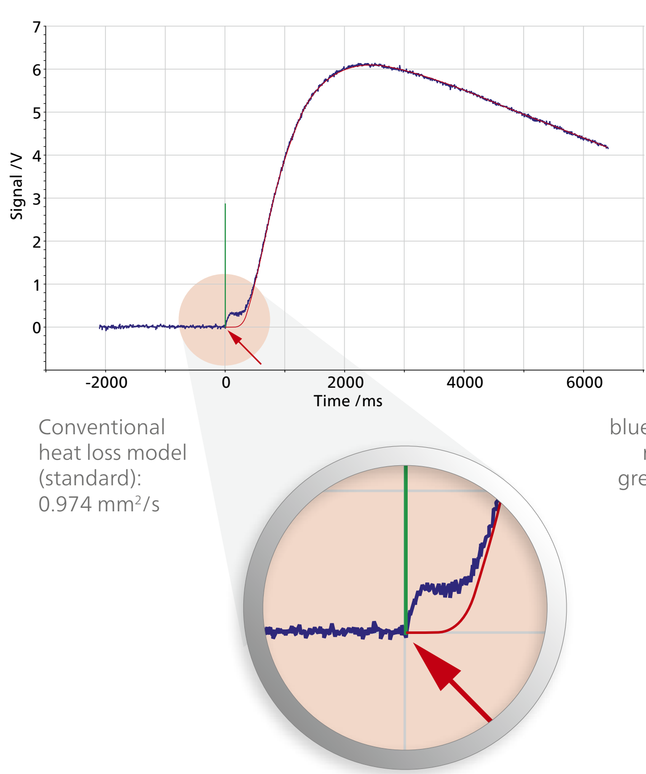
The influence of pulse correction is demonstrated with measurements on a 1.015-mm-thick silver plate at 25°C. This example proves that accurate measurement results are obtained within ± 3% of the literature value when an intelligent pulse correction method is used.

and Wizards



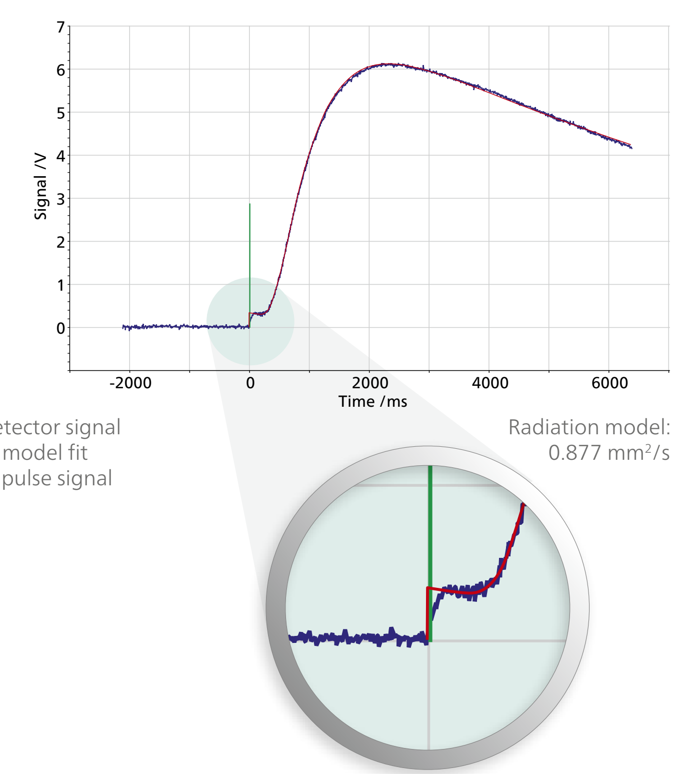
Perfectly Treating Translucent Materials – Transparent (Radiation) Model

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

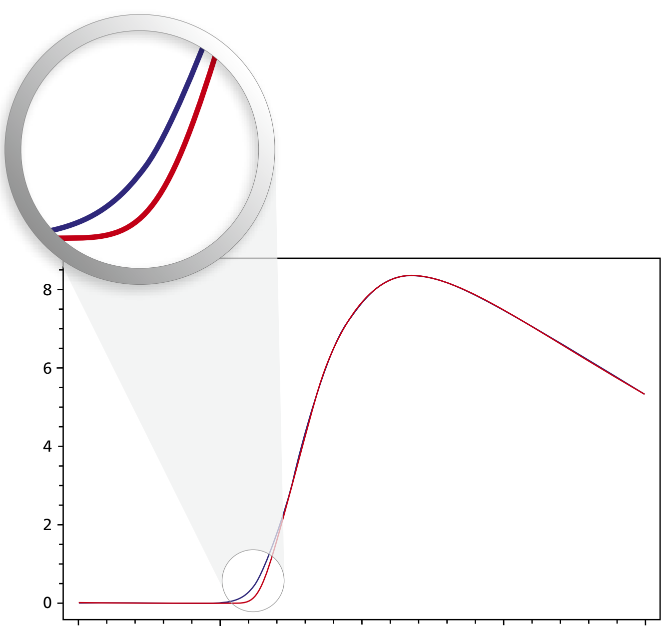
blue: detector signal
red: model fit
green: pulse signal



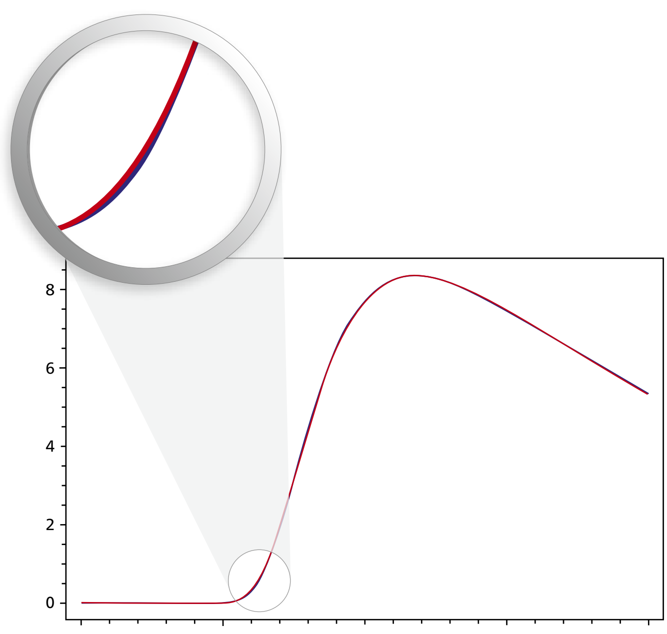
Radiation model: 0.877 mm²/s

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 without penetration: 0.753 mm²/s



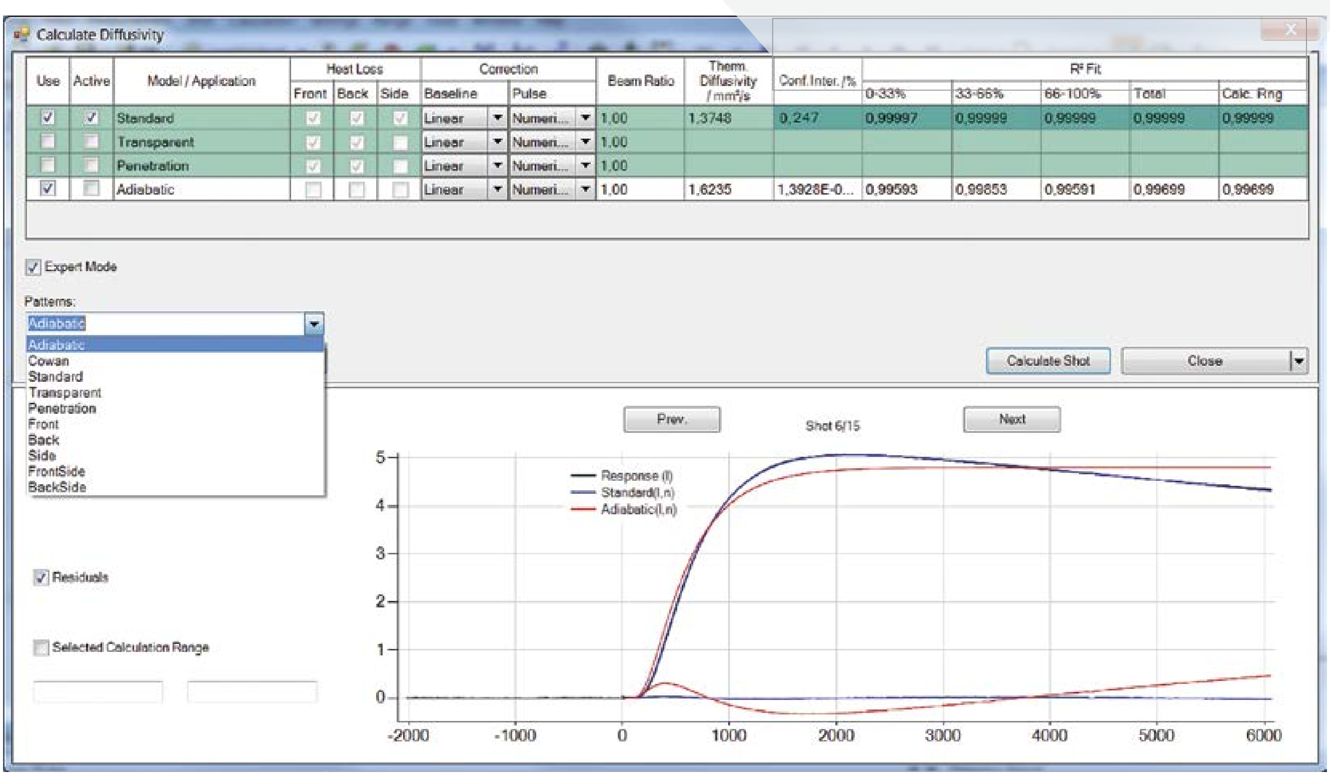
Laser shot with penetration: 0.626 mm²/s

MODEL WIZARD

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. /%	R^2 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

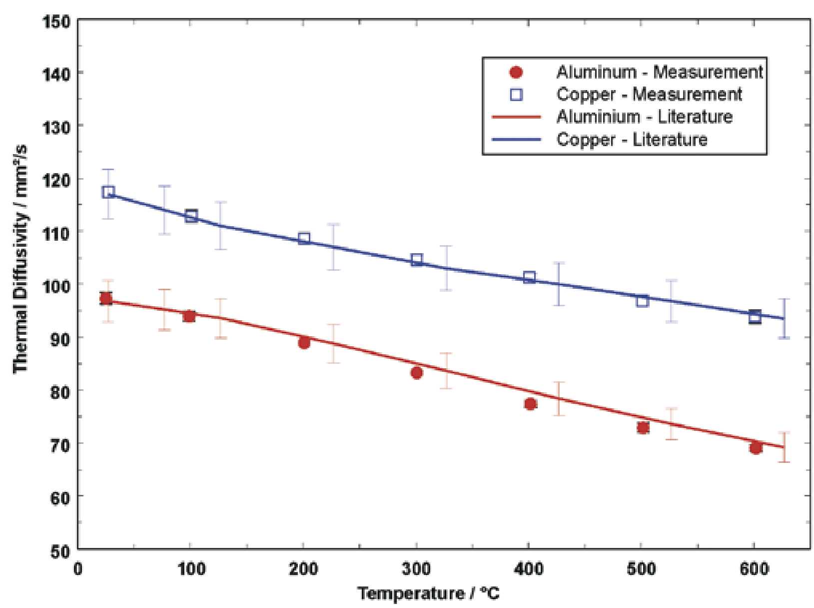


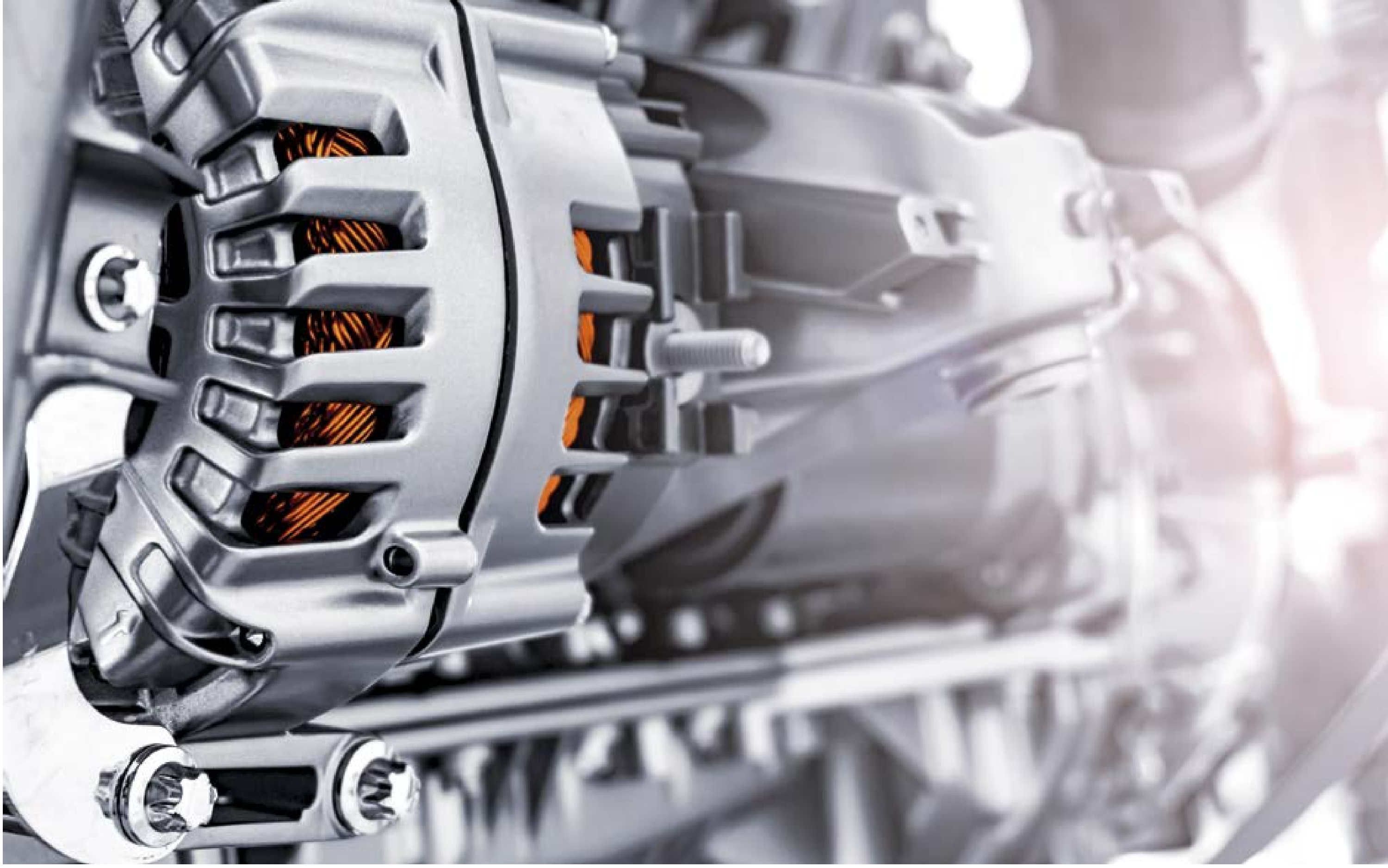
Salient Performance

Pure metals and stainless steel are excellent for checking the performance of a laser flash device up to high temperatures.

Copper and Aluminum

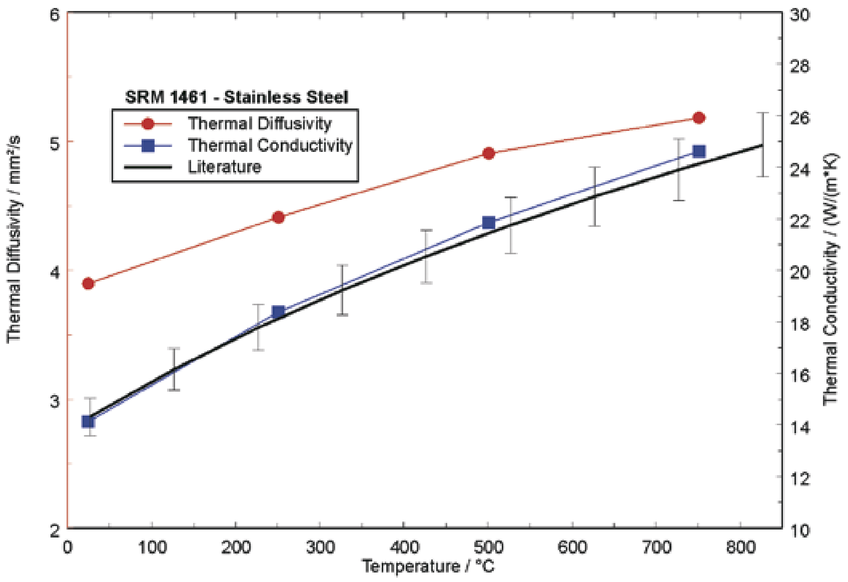
Presented in the plot are the thermal diffusivity results measured on pure copper (99.999%) and pure aluminum (99.9%) between room temperature and 600°C. The results are compared with literature values taken from the TPRC database (lines).
The literature values have a stated uncertainty of ±4% (error bars). However, the measurement results achieved with the NETZSCH LFA 457 *MicroFlash*® are generally in agreement to within ±2% with the values given in literature.





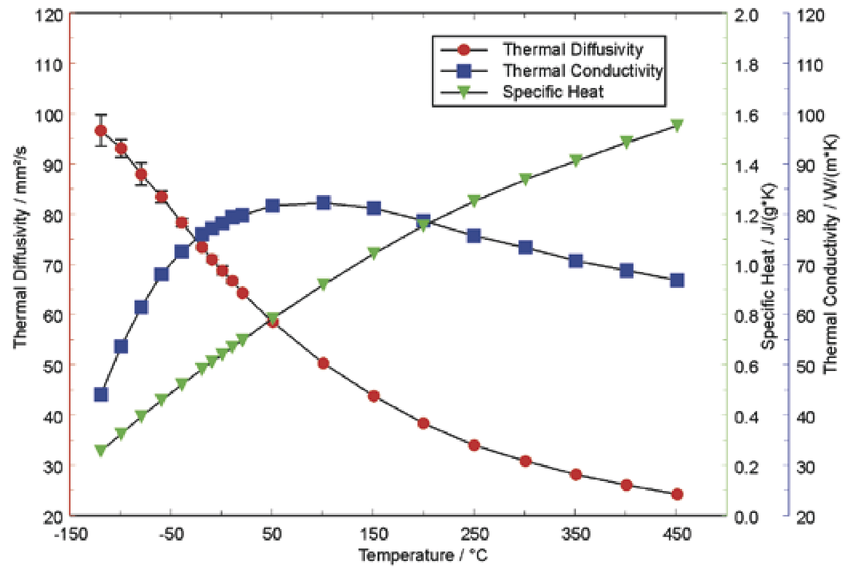
Stainless Steel (SRM 1461)

This plot displays the measured thermal diffusivity and thermal conductivity of the NIST thermal conductivity standard reference material SRM 1461.
In addition, the thermal conductivity values of the corresponding NIST certificate are shown together with the stated uncertainty. The results are clearly well within the given uncertainty range.



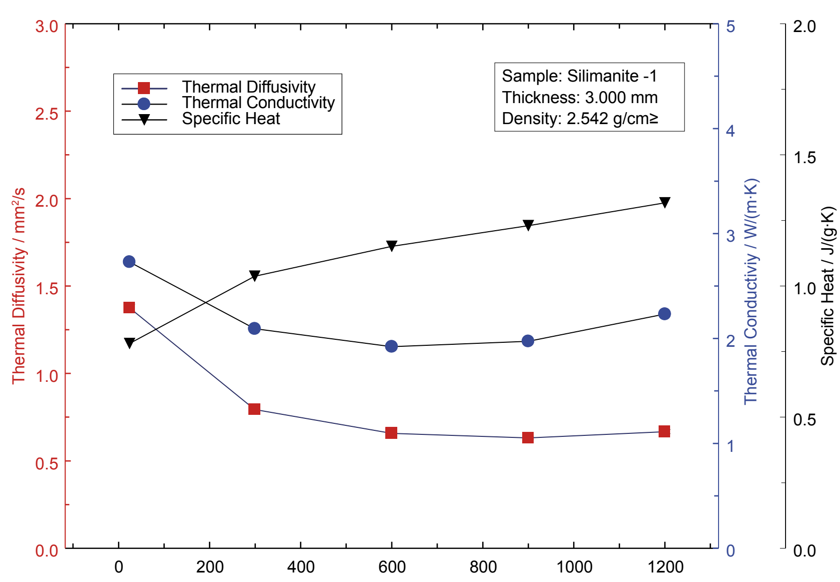
Polycrystalline Graphite

Graphite materials exhibit maximum thermal conductivity around room temperature. This can easily be analyzed using the low-temperature version of the LFA 457 *MicroFlash*®. The physical explanation for this maximum is the high Debye temperature of this material (>1000 K). As temperature increases, it is the dependence of the thermal diffusivity on temperature that dominates during the course of thermal conductivity. Below room temperature, the specific heat capacity decreases sharply and the gradient of the thermal diffusivity decreases as well. The specific heat capacity dominates during the course of thermal conductivity.



Coarse Ceramics

This plot exhibits the measured thermal diffusivity and specific heat capacity of a sillimanite sample. For calculation of the thermal conductivity, a bulk density of 2.542 g/cm^3 (at RT) is used.
The weak temperature dependence as well as the low thermal conductivity results are typical for alumino-silicate ceramics: thermal diffusivity decreases with temperature up to 800°C (phonon conductors); above 800°C, the values increase again which is most probably due to the increasing contribution of radiation heat transfer. On the other hand, the specific heat capacity increases over the entire temperature range.



Expertise in Service

All over the world, the name NETZSCH stands for comprehensive support and expert, reliable service, before and after sale. Our qualified personnel from the technical service and application departments are always available for consultation.

In special training programs tailored for you and your employees, you will learn to tap the full potential of your instrument. To maintain and protect your investment, you will be accompanied by our experienced service team over the entire life span of your instrument.

The NETZSCH applications laboratories are a proficient partner for nearly any Thermal Analysis issue. You will receive high-precision measurement results and valuable interpretations from us in the shortest possible time. This will enable you to precisely characterize new materials and components before actual installation, minimize risks of failure, and gain decisive advantages over your competitors. For production problems, we can work with you to analyze concerns and develop solutions.

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- Training
- Hotline service
- Preventive maintenance
- Individual maintenance services
- Calibration service
- On-site repairs with emergency service for NETZSCH components
- PC supported diagnostics
- E-mail reporting
- Moving / exchange service
- Technical information service
- Spare parts assistance
- Accessories catalogue
- Software update service
- Application support
- Environmental instrument recycling

The NETZSCH Group is an owner-managed, international technology company with headquarters in Germany. The Business Units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems represent customized solutions at the highest level. More than 3,700 employees in 36 countries and a worldwide sales and service network ensure customer proximity and competent service.

Our performance standards are high. We promise our customers Proven Excellence – exceptional performance in everything we do, proven time and again since 1873.

When it comes to Thermal Analysis, Calorimetry (adiabatic & reaction), the determination of Thermophysical Properties, Rheology and Fire Testing, NETZSCH has it covered. Our 50 years of applications experience, broad state-of-the-art product line and comprehensive service offerings ensure that our solutions will not only meet your every requirement but also exceed your every expectation.

Proven Excellence.

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