Laser Flash Apparatus LFA 427

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

\[
\lambda(T) = \alpha(T) \cdot c_p(T) \cdot \rho(T)
\]

where
- \(\lambda\) = thermal conductivity [W/(m·K)]
- \(\alpha\) = thermal diffusivity [mm²/s]
- \(c_p\) = specific heat [J/(g·K)]
- \(\rho\) = bulk density [g/cm³].

The thermal diffusivity is computed by the software using data on time and relative temperature increase. For adiabatic conditions, the thermal diffusivity \(\alpha\) 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.

**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

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\textsubscript{2}) controller of the low-temperature furnace is optimized for fast temperature stabilization with reduced LN\textsubscript{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.
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

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

**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 Al₂O₃ 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.
VERSATILITY AND INTERCHANGEABILITY

For a Clear View – Removable Windows

The top and bottom of the sample chamber are sealed off by CaF₂ 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 Special Applications

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

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.
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 Al₂O₃ 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.

Centering cones (here made of Al₂O₃, shown in white, and graphite, in grey) and caps are available for specimen diameters of 6 mm to 12.7 mm.
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
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.

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.
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 \(0.877 \text{ mm}^2/\text{s}\) than that of the poor fit \(0.974 \text{ mm}^2/\text{s}\) obtained by using the conventional model.
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.
Graphite – At highest temperatures

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).

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 (cₚ) are observed.
Test 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.

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%.

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 Al₂O₃-based, each with different secondary components, while sample D is Si₃N₄-based. The higher thermal conductivity of the Si₃N₄ 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.

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.
All over the world, the name NETZSCH stands for comprehensive support and expert, reliable service, both 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.

Expertise in SERVICE
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), LN₂-cooled, optional LN, refill system including 35 liter dewar
- InSb (RT to 2800°C), optional LN₂ refill system including 35 liter dewar

**Measuring range**
- Thermal diffusivity: 0.01 mm²/s to 1000 mm²/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**
- Al₂O₃ (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*
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.