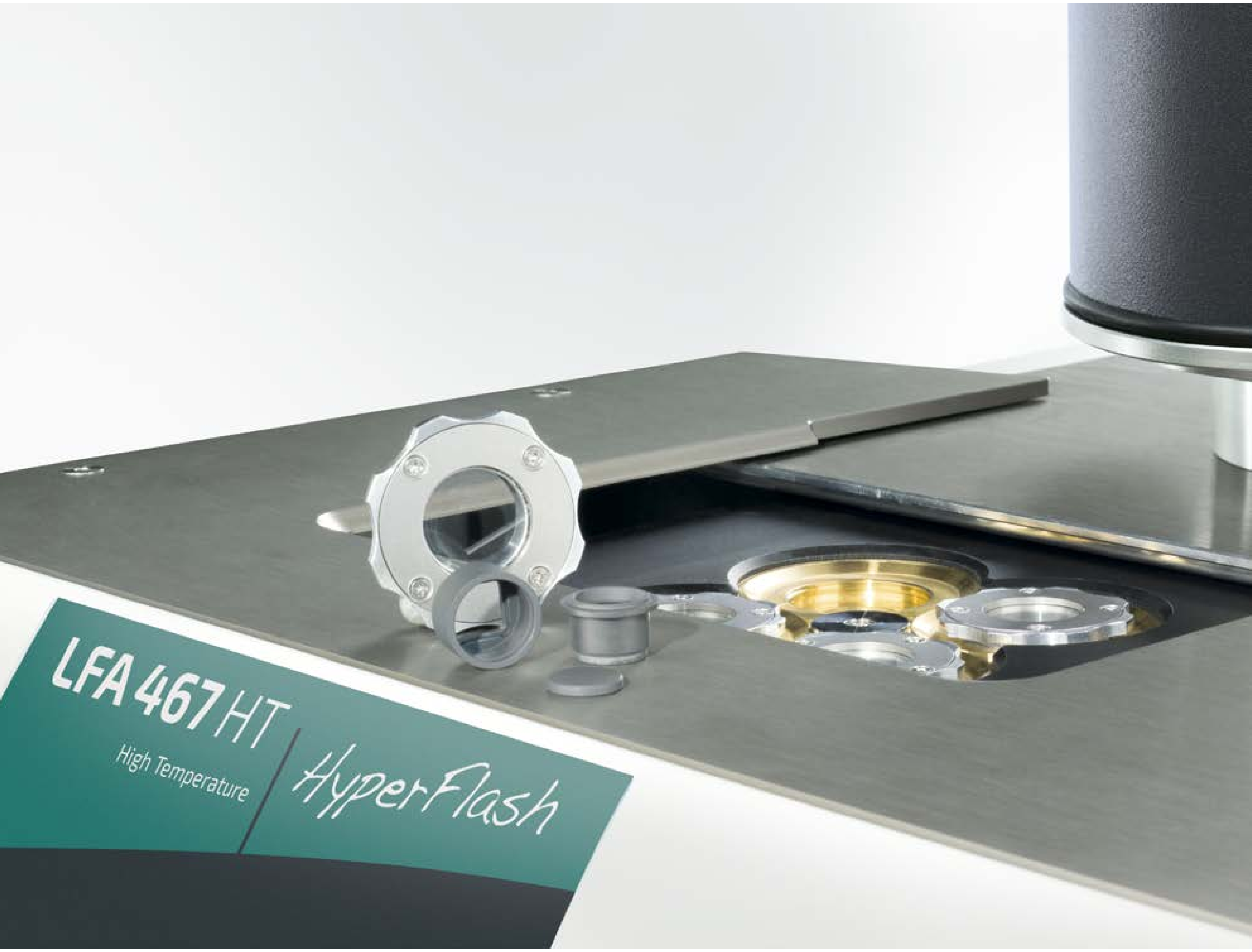
NETZSCH
Proven Excellence.



Light Flash Apparatus LFA 467 *HyperFlash*® Series

Method, Technique, Applications of Thermal Diffusivity and Thermal Conductivity

Analyzing & Testing

THE FLASH METHOD

Thermal Conductivity/Thermal Diffusivity

How much heat is being transferred, and how fast?

Researchers and engineers are interested in the best way to thermally characterize highly conductive materials at cryogenic and moderate temperatures or ceramics and refractories at elevated temperatures. Many challenges can only be met with precise knowledge of two fundamental thermal properties: diffusivity and conductivity. One accurate, reliable and elegant solution is offered by the Flash Method. This method allows for the meeting of challenges typically arising in heat transfer processes such as:

- Determining how quickly an aluminum ingot solidifies
- Assessing how quickly 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 two decades, NETZSCH has led the way in this technology, extending our application range 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 467 *HyperFlash*® and the LFA 467 *HT HyperFlash*® set the standard.

Light Flash

An Efficient Method for Determination of Thermophysical Properties

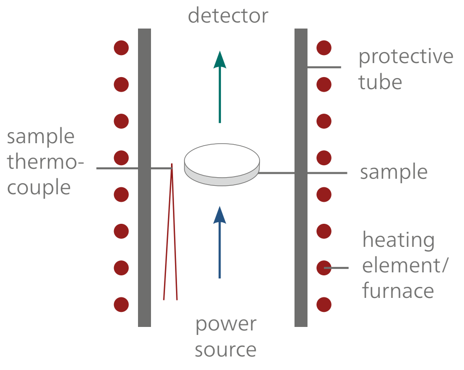
The front surface of a plane-parallel sample is heated by a short energy light pulse. From the resulting temperature excursion of the rear face measured with an infrared (IR) detector, thermal diffusivity and, if a reference specimen is used, specific heat are both determined. Combining these thermophysical properties with the density value results in 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 Light Flash (LFA) technique is a fast, non-destructive, non-contact, and absolute method for determining these thermophysical properties, including specific heat. This data can then be used for:

- Complete set of thermophysical properties such as thermal diffusivity (a), specific heat capacity (c_p) and thermal conductivity (λ) as input data for numerical simulations.
- Material optimization according to the desired thermal performance.



Flash Technique

LFA 467 *HyperFlash*®

DEFINED ATMOSPHERES

The atmosphere can be controlled via three integrated frits or optionally via mass flow controllers for one protective and two purge gases. All gas controls offer operation in oxidizing, inert, dynamic or static atmospheres. Additionally, pumping allows for measurements under reduced pressure.

INTELLIGENT INSTRUMENT SETUP AND FLASH SOURCE

The LFA 467 *HyperFlash*® is designed as a vertical system with the flash source at the bottom, the sample in the center and the detector on top. A xenon lamp serves as the flash source. The variable pulse energy is software-controlled and an optional filter wheel can be used to further adjust it. The pulse width is adjustable in the range of 20 µs to 1200 µs.

HIGHEST SAMPLE THROUGHPUT – 16 SAMPLES SIMULTANEOUSLY

The LFA 467 *HyperFlash®* has an integrated automatic sample changer for up to 16 samples. A tray for four holders, each containing four samples, can be used with round and square samples. A considerable reduction in operator intervention is also effectuated through the use of a high volume liquid nitrogen Dewar.

ONE FURNACE – WIDE TEMPERATURE RANGE

Measurements from -100°C (i.e., below the glass transition temperature of rubber materials) to 500°C can be carried out with a single instrument setup. Neither the furnace nor the detector has to be swapped. The design enables thermal coupling to different cooling devices. This considerably reduces measurement times and allows for heating rates up to 50 K/min, while maintaining an excellent thermal stability.

COOLING – GUARANTEES FLEXIBILITY

The cooling devices using liquid nitrogen allow for temperatures as low as -100°C (depending on the purge gas and the sample, even lower temperatures can also be achieved). They can be operated in conjunction with the evacuation system (below atmospheric pressure). This leads to a further reduction in heat losses. In addition, a pressurized air device is available for measurements between 0°C and 500°C. All cooling systems can also be operated during measurements under a defined atmosphere using a purge gas. This is advantageous when testing oxygen-sensitive samples.

Unique Concept

UNPRECEDENTED FEATURES

LFA 467 *HT HyperFlash®*

HIGH TEMPERATURES WITH XENON FLASH

The LFA 467 *HT HyperFlash®* is based on the already-established LFA 467 *HyperFlash®* technology and requires no laser class due to the innovative light source system. The long lifetime of the xenon lamp provides cost-effective measurements up to 1250°C without costly consumables.

WIDEST TEMPERATURE RANGE WITH THE SMALLEST FOOTPRINT

The LFA 467 *HT HyperFlash*® is the first flash-lamp based LFA system to reach temperatures up to 1250°C. A single furnace with an integrated sample changer covers the entire temperature range, providing the small footprint for which the LFA 467 *HyperFlash*® series is well known. Even at elevated temperatures, an efficient internal water cooling circuit keeps the temperature of the surrounding components within a safe range, thereby reducing the liquid nitrogen consumption of the IR detector.

UP TO 1250°C

DEFINED ATMOSPHERES PREVENT OXIDATION — VACUUM-TIGHT FURNACE

An internal pump device supports defined atmospheres by an automatic evacuation function prior to each measurement. Additional connections for external pump devices are available. The vacuum-tight platinum furnace allows for heating rates up to 50 K/min.

MINI-TUBE FURNACES FOR UNMATCHED TESTING SPEED

Effective sample throughput over the entire temperature range is realized guaranteed by the high-speed furnace. Each of the four specimen positions is equipped with its own thermocouple. This results in short stabilizing times. Within one hour, ten temperature steps can be measured up to 1250°C. The ASC is designed for sample dimensions of 12.7 mm (round) and 10 mm (round and square).

The Solution for Thin Films – High Data Acquisition

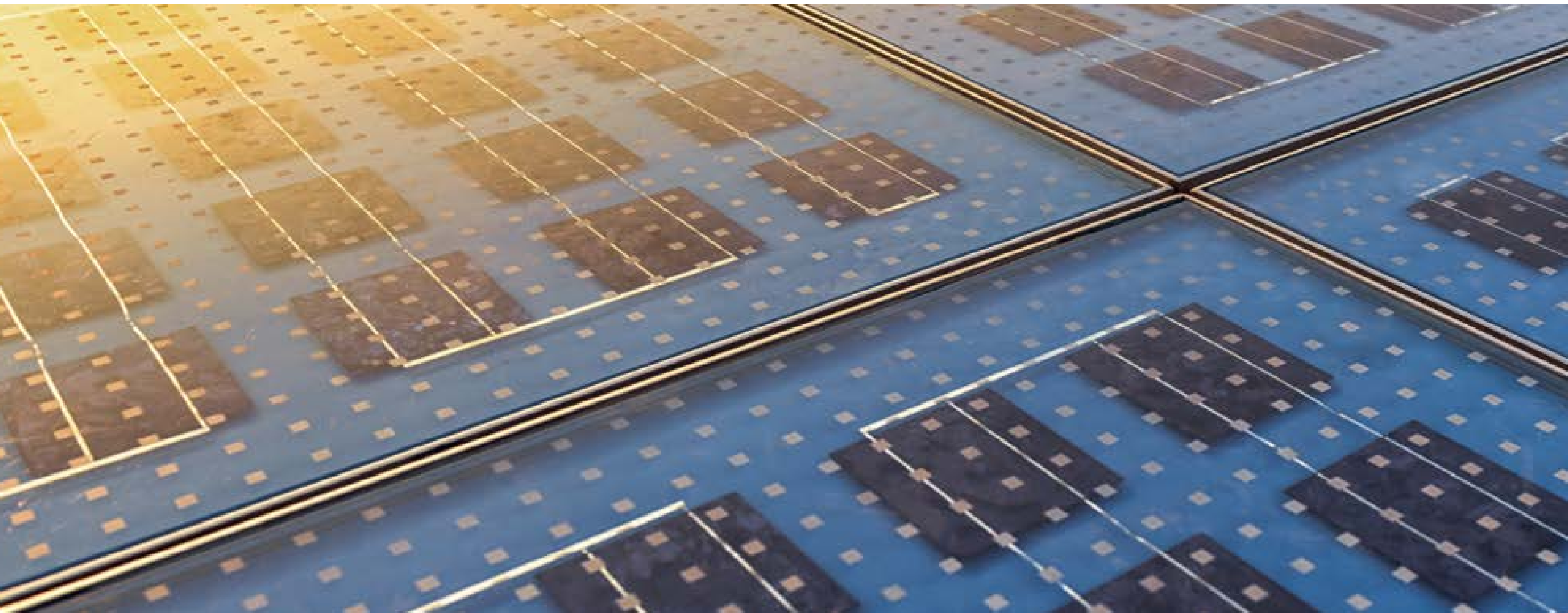
The data acquisition rate of the LFA 467 *HyperFlash®* series was increased to 2 MHz. This acquisition rate applies to both the IR detector and the pulse mapping channels. Thereby, highly conductive and/or thin materials requiring very short test times can be reliably tested.

Thin and Highly Conducting Materials By Ultra-Fast Sampling Rate

When testing metal (0.3 mm) and polymer foils (30 µm), an optimum sampling rate and pulse width can be selected. The patented pulse mapping system accounts for the finite pulse width effect and heat losses (patent no.: US7038209 B2; US20040079886; DE10242741).

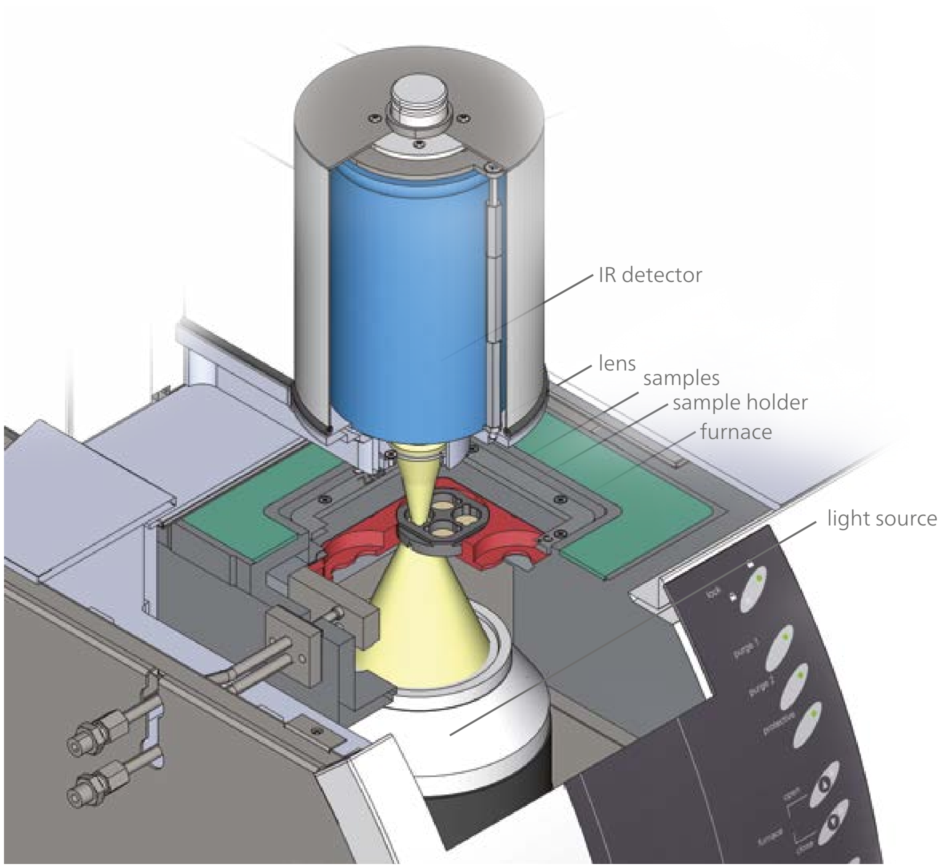
LFA 467 *HyperFlash®* Series

PUSHING THE BOUNDARIES



ZoomOptics

FOR PRECISE MEASUREMENT RESULTS



Simplified Handling Allows for View of Just the Sample Surface

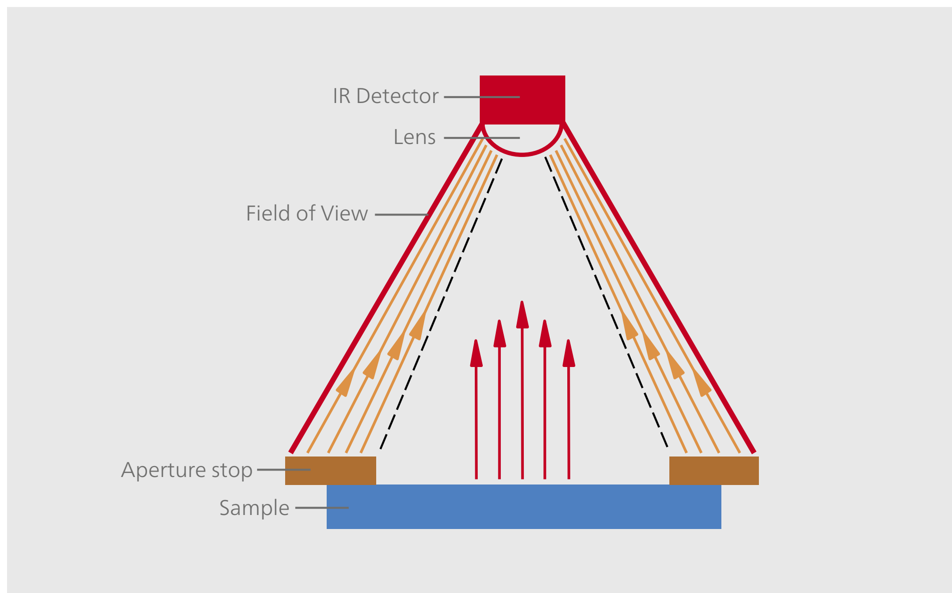
Between the detector and sample, a stepper-motor-actuated lens optimizes the field of view by software control. This helps to prevent measuring artifacts due to contributions from the aperture stop, often characterized by a delayed IR signal. Signal distortions from the sample's immediate surroundings like masks or aperture stops are avoided. The precision of the test results is thus greatly improved.

This feature is particularly valuable for small-diameter samples. It also ensures that the detector is always kept within its linear responsivity range.

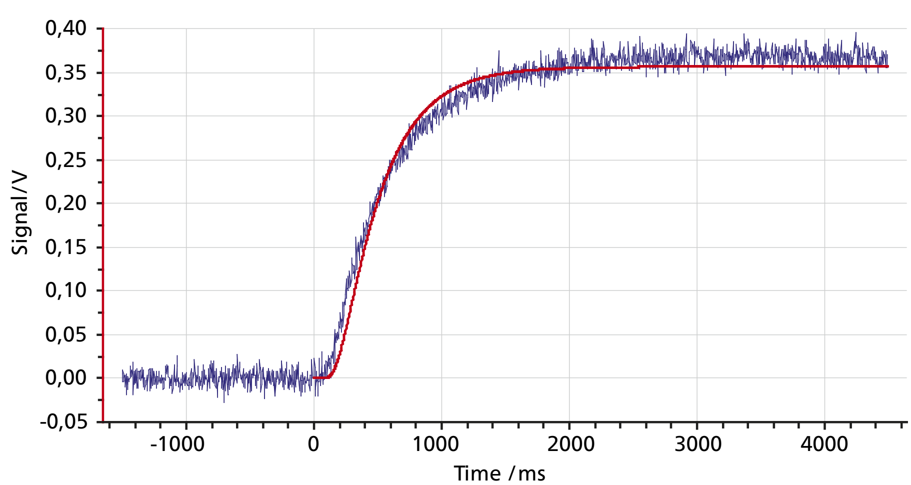
OPTIMIZED FIELD

Lack of *ZoomOptics* Allows for Distortion from the Aperture Stop

In current LFA systems, the field of view is fixed and wide enough to accommodate large-diameter samples. When testing smaller-diameter samples, aperture stops are commonly used in an attempt to minimize influence of the surroundings. This often results in a significant distortion of the thermal curve to the extent that the detector senses not only the temperature excursion of the sample, but also any fluctuations from the aperture stop. Consequently, the thermal curve would show either a continuously increasing trend or, as depicted below, an extended leveling-off period.

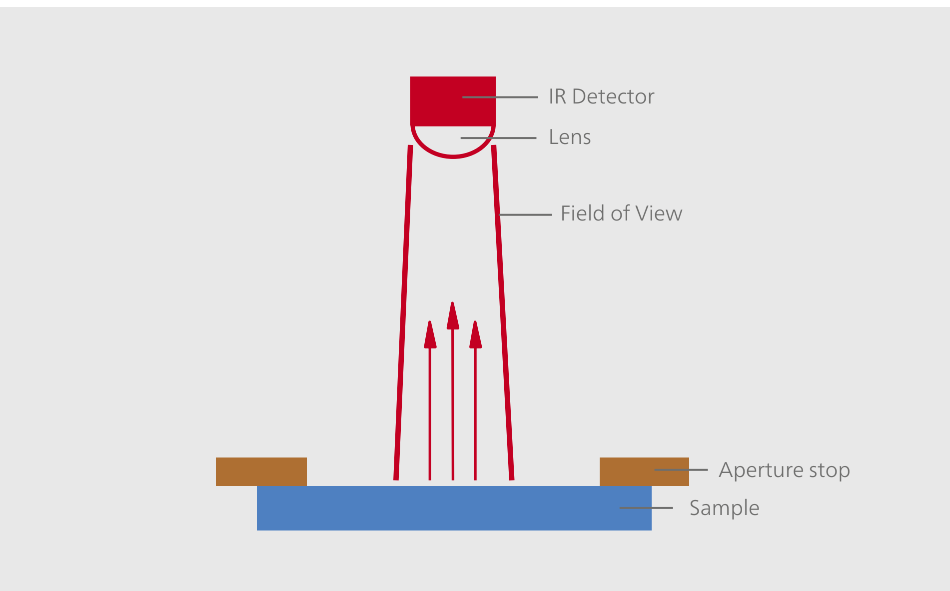


Field of view in standard LFA systems

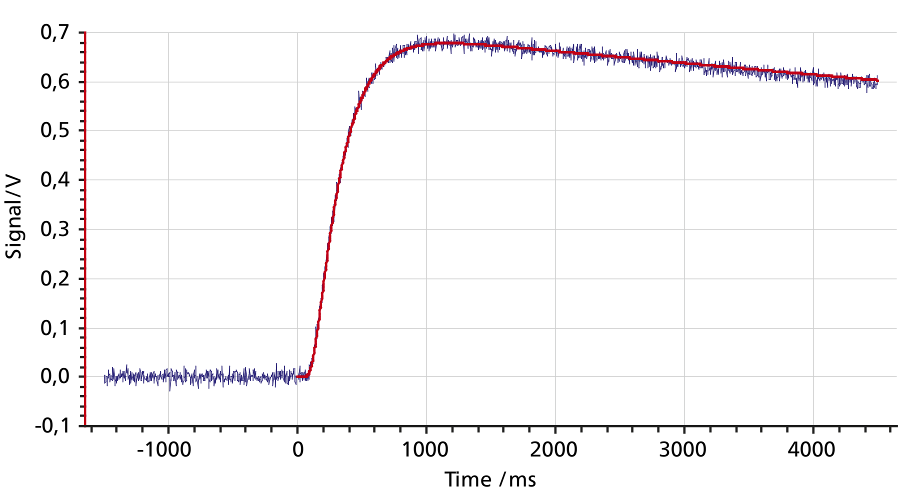


Standard LFA system without *ZoomOptics* yields measurements with distortions from the aperture stop

OF VIEW
ZoomOptics



Field of view when using *ZoomOptics*; no influences from the aperture stop occur



When using *ZoomOptics*, the measurement signal no longer exhibits any distortion caused by the aperture stop

ZoomOptics Prevents Any Distortion from the Aperture Stop

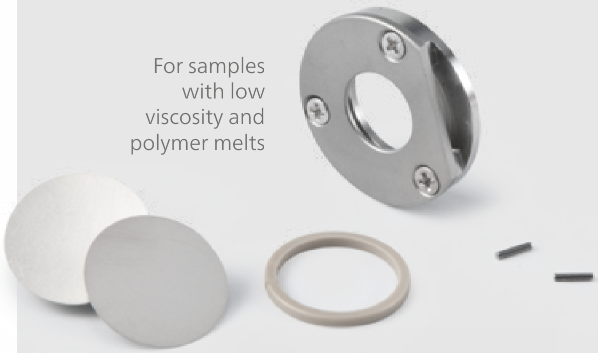
By using the new *ZoomOptics* of the LFA 467 *HyperFlash*®, it can be ensured that the IR signal originates solely from the sample surface and not from any surrounding parts. Therefore, both large and small samples can be tested with an optimal sensing area.

In contrast with the previous configuration, the lens has been shifted for an adequate field of view. The aperture stop no longer produces any noticeable effects.

As expected, the thermal curve now conforms to the theoretical model, yielding correct diffusivity values. In addition, the signal-to-noise ratio is improved.

LFA 467 *HyperFlash®* – Sample Holders for Special Appliations

For samples with low viscosity and polymer melts



In-plane sample holder

For liquids

For lamellar samples

Pressure sample holder

Flexible & Efficient

Two Detectors – Always Remote Sensing

Two user-exchangeable detectors are available. The standard indium antimonide (InSb) detector is suitable between room temperature and 500°C (LFA 467 *HyperFlash®*) or 1250°C (LFA 467 *HT HyperFlash®*), respectively, while the optionally available mercury cadmium telluride (MCT) detector allows for measurements from -100°C to 500°C. The detectors can be equipped with a liquid nitrogen auto-refill system – which operates without operator intervention. Many samples using multiple temperature steps can be conveniently tested. The instrument design ensures that, even at cryogenic temperatures, the measurements are always based on the infrared energy radiation from the sample's surface.

Sample Dimensions

Each of the four sample holder positions in the base plate of the LFA 467 *HyperFlash®* can carry up to four round or square sample specimens with a maximum diameter of 12.7 mm each.
For measurements on large samples, inserts can be selected to accommodate sample diameters up to 25.4 mm. Depending on the sample's properties, the thickness can vary between 0.01 mm and 6 mm.

The LFA 467 *HT HyperFlash®* accommodates sample dimensions of max. 12.7 mm (round) and 10 mm (square/round).

The Sky's the Limit

SAMPLE HOLDERS

Cleverly-Designed Accessories for Special Applications

In addition to the standard sample holders for solid samples of round and square geometries, dedicated sample holders for special applications are available for the LFA 467 *HyperFlash*®:

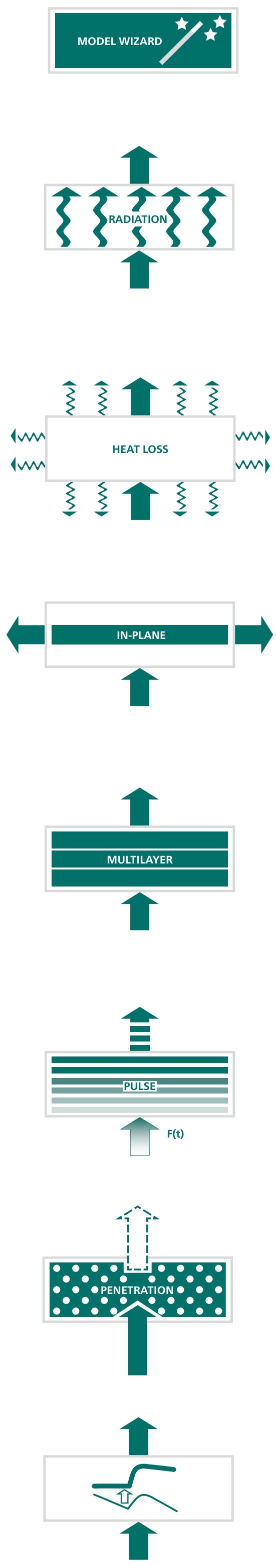
- Molten polymers and low-viscosity liquids
- Resins during curing
- Pastes and powders
- Fibers
- Laminates
- In-plane

The design of the sample holder for liquids ensures continuous contact between the liquid and crucible over the entire temperature range – even at freezing temperatures. The heat transfer through the container wall is minimized.

A special sample holder made of cost-effective consumables is available for measurements on resins during the curing process. In addition, sample holders for measurements in the in-plane direction and ones for tests under mechanical pressure are included in our product line. Customized sample holders are available upon request.

Software *Proteus*®

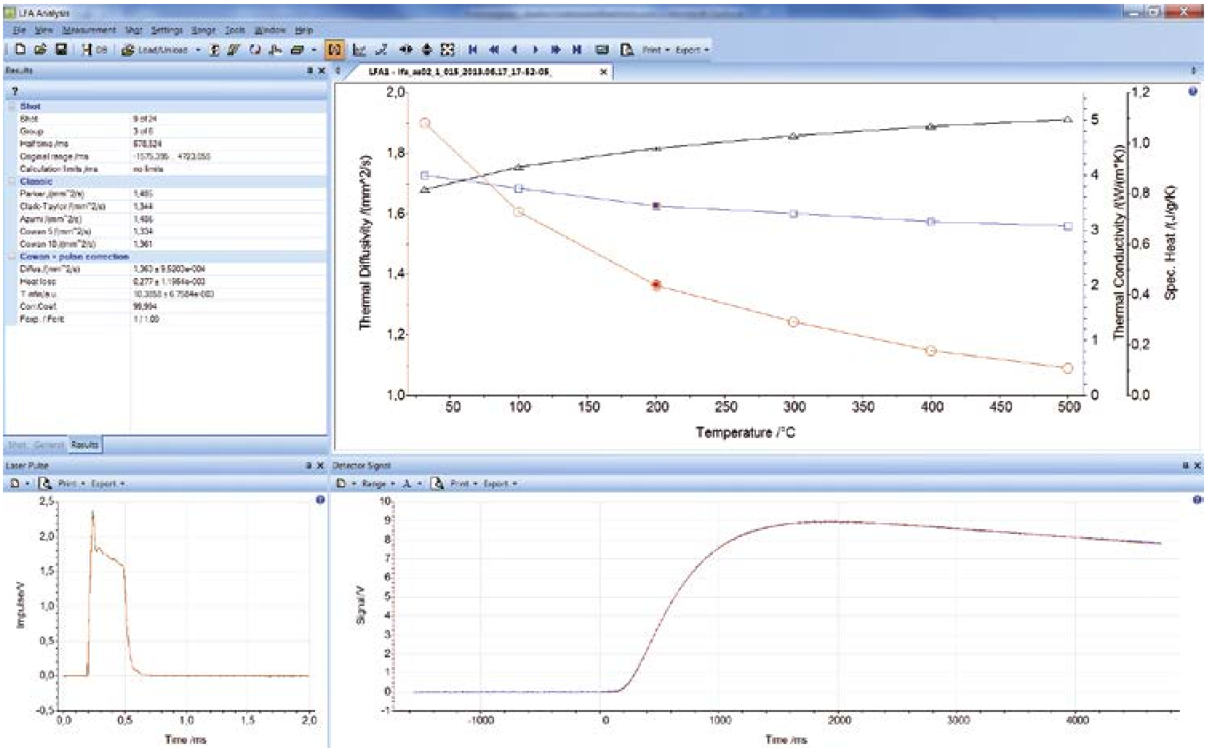
Intelligent Operation – Just a Click Away



The *Proteus*® software combines user-friendly menus with automated routines. This makes 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.

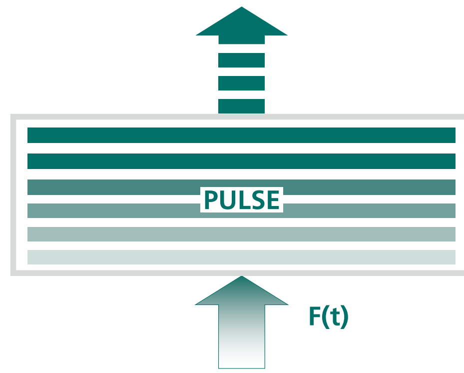
General Software Features
Multiple-window technique for clear presentation
Drag-and-drop software functions
Comparative analysis for up to 32 series of shots from the same database
Loading of series of single shots with a preview of parameters and temperature program
Model wizard for selection of the best model
Definition of an arbitrary number of temperature setpoints and number of shots per setpoint
Determination of the specific heat 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
Measurement values shown as a Tool-Tip when moving 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 All standard models allow for the combination of heat loss, pulse correction and various baseline types. All factors are freely selectable; a 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; DE10242741)
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 Fast-Conducting Materials

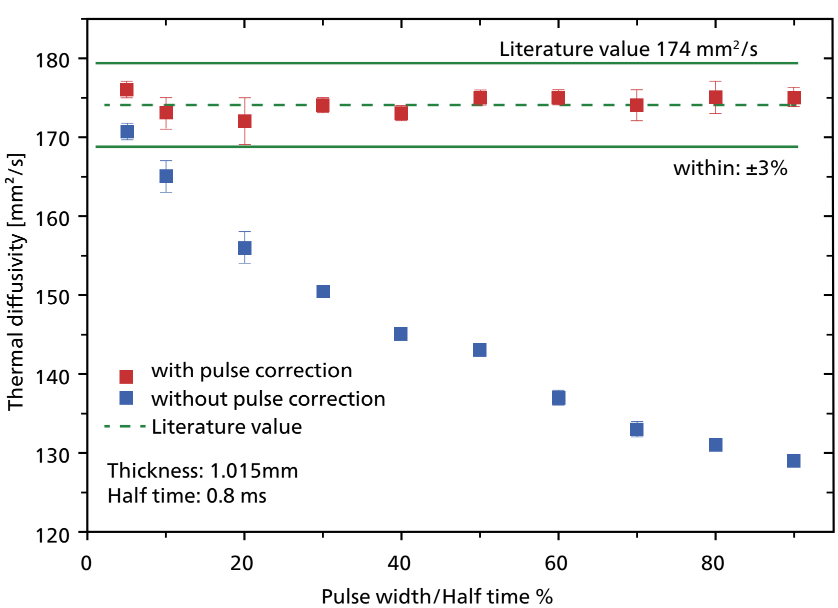


Finite Pulse Correction

The unique pulse mapping (patent no. US7038209, US20040079886, DE10242741) enables finite pulse correction and improved thermal diffusivity and c_p determination. This feature is implemented in the standard software of the LFA *HyperFlash®* series.

It considers the 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.

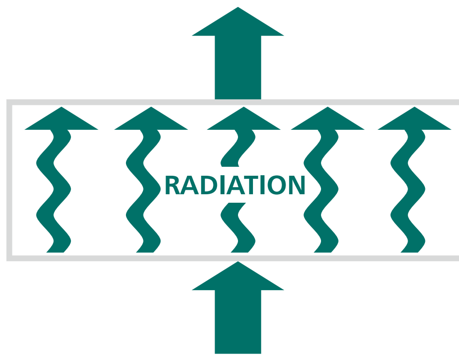
This feature is specifically essential for thin and fast-conducting samples.



LFA 467 *HyperFlash®*: 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.

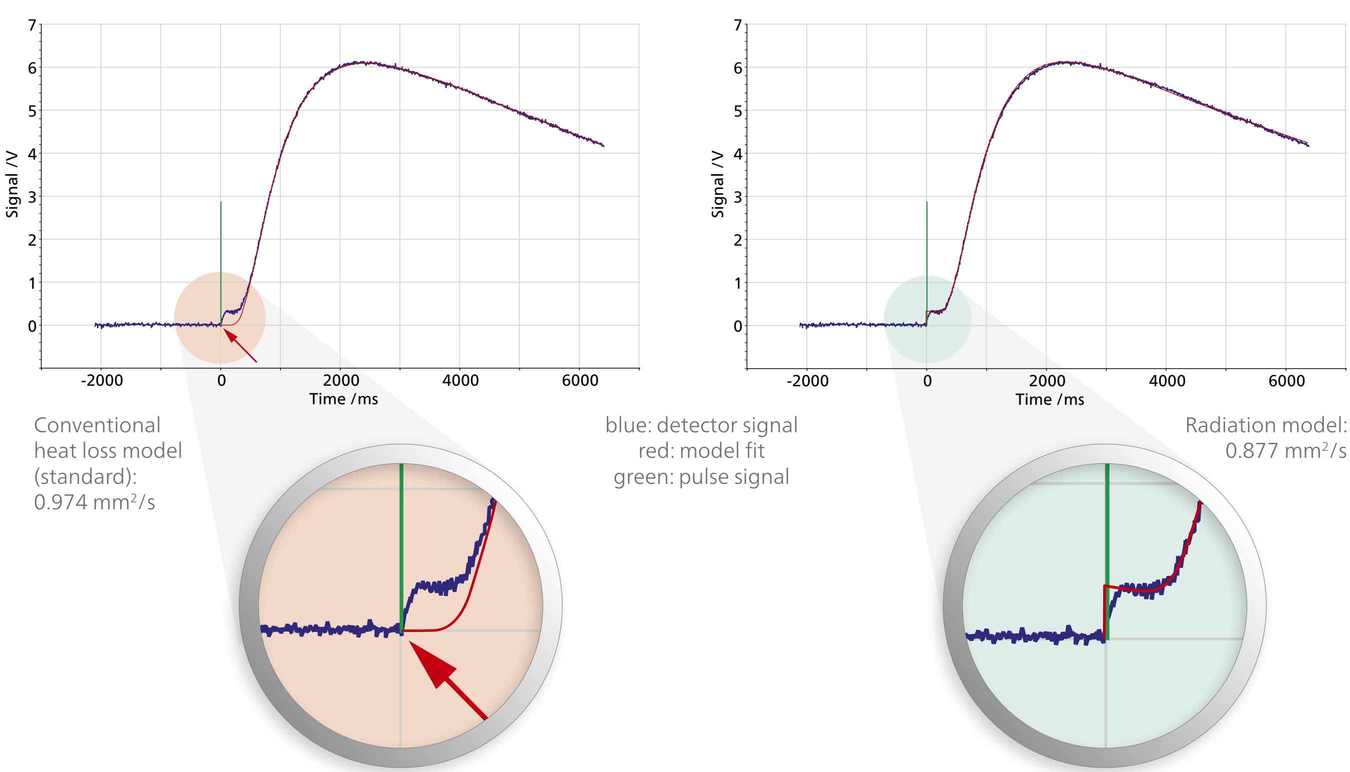
Perfectly Treating Translucent Samples



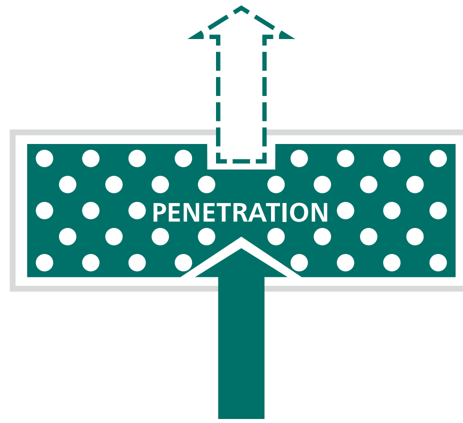
Transparent (Radiation) Model

The Transparent Model (patent no. DE102015118856, JP6382912, ZL2016109515017, US10180358) is based on advanced mathematics accounting for non-conductive heat transfer effects.

For translucent samples, the light pulse immediately leads to a temperature increase on the rear side of the specimen. Conventional models cannot correctly describe the initial temperature rise. The use of a model dedicated to radiation allows for a proper fit (red) of the detector signal (blue). The 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) compared to the poor fit (0.974 mm²/s) obtained by using the conventional model.

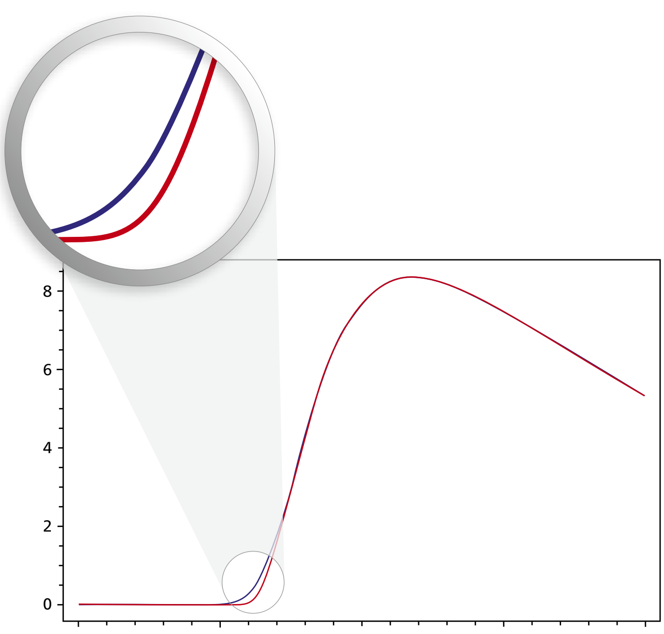


New Model Dedicated to Porous and Rough Materials

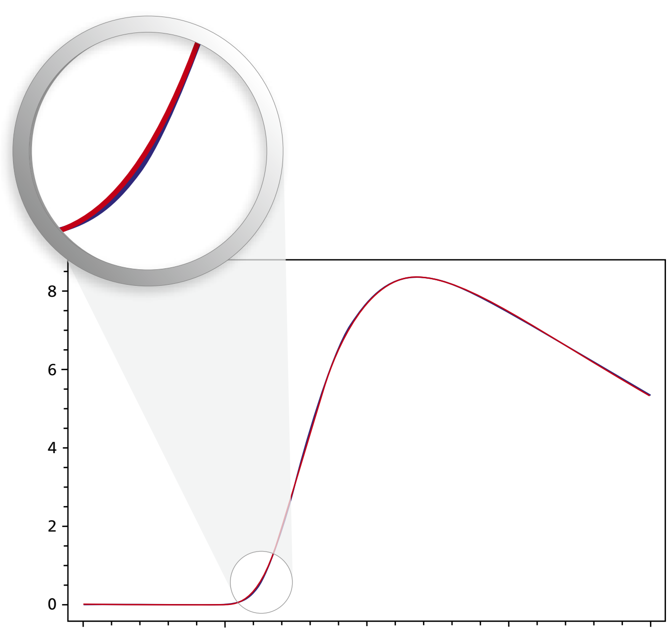


Beam Penetration

The standard flash method (Parker et al.) assumes that the pulse energy is totally absorbed on the front face of the specimen, then migrates as a thermal wave through the thickness of the specimen before finally arriving at the opposite face.
However, in a slightly porous material or a material with a rough surface, the 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

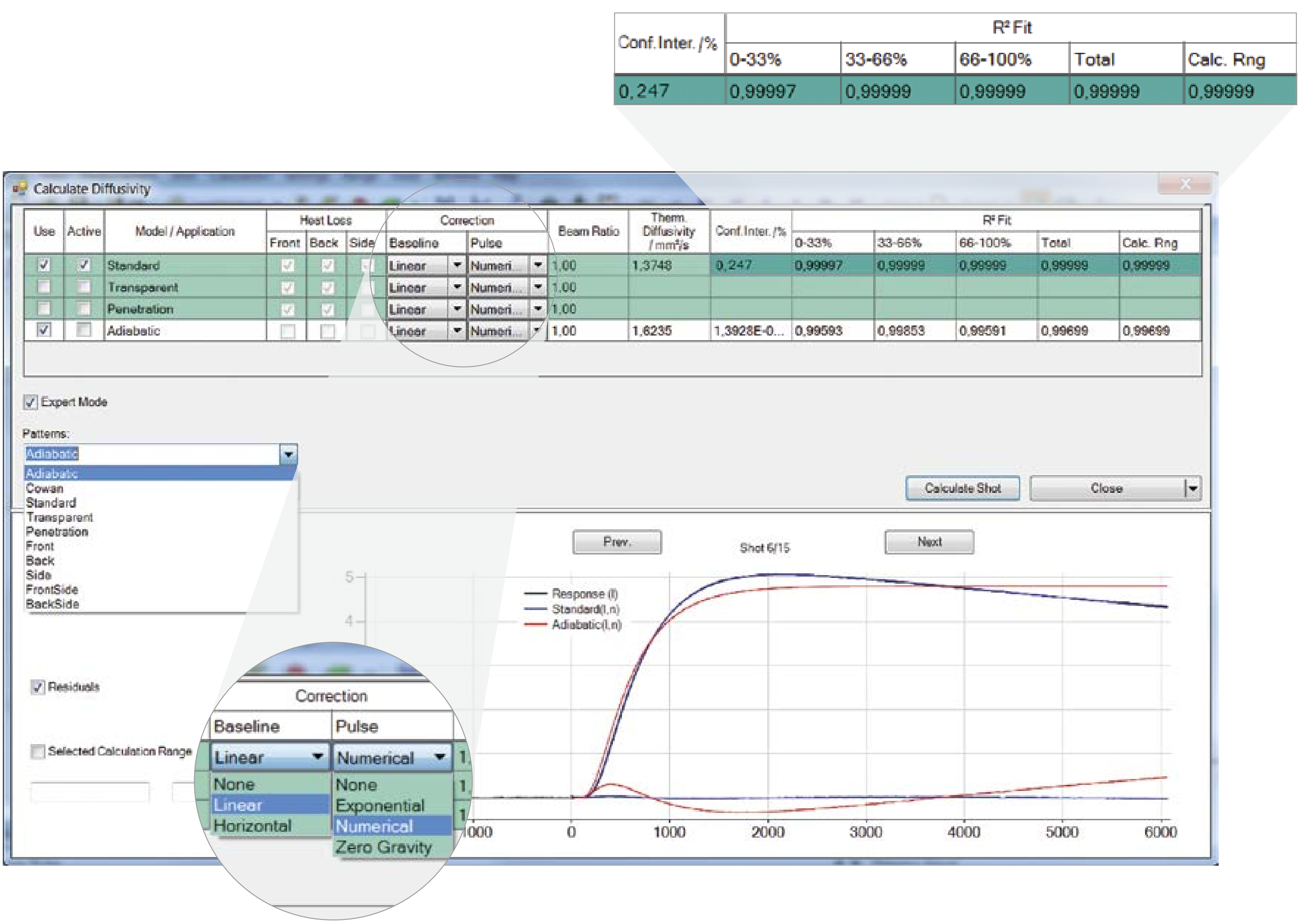


Laser shot with penetration: 0.626 mm²/s

Model Wizard
Always on Your Side

Best Fit for Best Result

Use of the implemented 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 automatically 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.



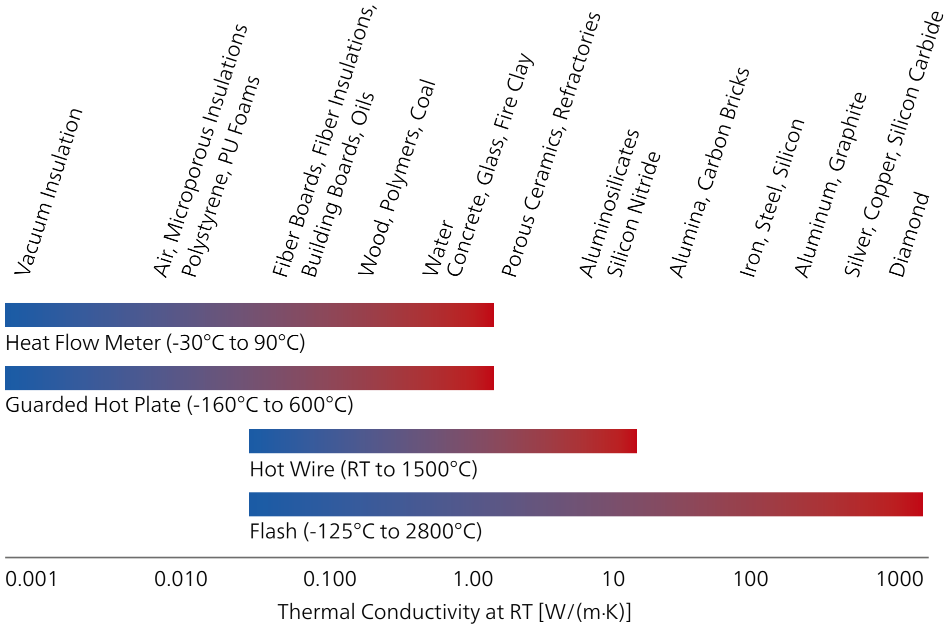
Technical Specifications

	LFA 467 *HyperFlash*®	LFA 467 *HT HyperFlash*®
Temperature range	-100°C ... 500°C room temperature version available	RT ... 1250°C (furnace temperature 1500°C)
Heating rate (max.)	50 K/min	50 K/min
Furnace cooling device	External chiller (RT… 500°C), Optional: ▪ Liquid nitrogen cooling (-100 … 500°C) ▪ Pressurized air (0°C … 500°C)	External chiller
Thermal diffusivity	0.01 mm^2/s ... 2000 mm^2/s	0.01 mm^2/s ... 2000 mm^2/s
Thermal conductivity	0.1 W/(m·K) ... 4000 W/(m·K)	0.1 W/(m·K) ... 4000 W/(m·K)
Accuracy	▪ Thermal diffusivity[1]: ± 3% ▪ Specific heat[2]: ± 5%	▪ Thermal diffusivity[1]: ± 3% ▪ Specific heat[2]: ± 5%
Repeatability	▪ Thermal diffusivity[1]: ± 2% ▪ Specific heat capacity[2]: ± 3%	▪ Thermal diffusivity[1]: ± 2% ▪ Specific heat capacity[2]: ± 3%
Xenon flash lamp	▪ Pulse energy[3]: up to 10 Joules/pulse (variable), software-controlled ▪ Pulse width[4]: 10 to 1500 µs	▪ Pulse energy[3]: up to 10 Joules/pulse (variable), software-controlled ▪ Pulse width[4]: 10 to 1500 µs
ZoomOptics	Patented (EP2693205, DE102012106955); optimized field of view (optional, requires no mask)	Patented (EP2693205, DE102012106955); optimized field of view (optional, requires no mask)
Pulse mapping	Patented pulse mapping (US7038209, DE10242741), for finite pulse correction and improved c_p determination	Patented pulse mapping (US7038209, DE10242741), for finite pulse correction and improved c_p determination
IR detectors	▪ InSb: RT ... 500°C ▪ MCT: -100°C ... 500°C ▪ Detector refill device (option)	▪ InSb: RT ... 1250°C ▪ Detector refill device (option)
Atmosphere	Inert, oxidizing, static and dynamic	Inert, oxidizing, static and dynamic
Vacuum	< 150 mbar	10^{-4} mbar (with turbo pump)
Data acquisition	2 MHz ▪ Min. measurement time (10 half times) down to 1 ms → for highly conducting and/or thin samples (e.g., Al, Cu plates, thin films, etc.) ▪ Max. measurement time up to 120 s → for low-conducting and/or thick samples (e.g., polymers, refractories, etc.)	2 MHz ▪ Min. measurement time (10 half times) down to 1 ms → for highly conducting and/or thin samples (e.g., Al, Cu plates, thin films, etc.) ▪ Max. measurement time up to 120 s → for low-conducting and/or thick samples (e.g., polymers, refractories, etc.)
Gas control	Frits or optional MFC; measurements under reduced pressure possible	MFC + internal pump
Sample holders	▪ For round and square samples ▪ For liquids, pastes, resins, powders, fibers, laminates, anisotropic samples ▪ For tests under mechanical pressure	For round and square samples
Integrated automatic sample changer	4 insets for up to 4 samples each: ▪ 4x $Ø_{max.}$ 25.4 mm ▪ 16x up to $Ø_{max.}$ 12.7 mm ▪ 16x up to $□_{max.}$ 10 mm	4 insets for 1 sample each: ▪ Ø 12.7 mm ▪ □ 10 mm ▪ Ø 10 mm

1 Accuracy of thermal diffusivity amounts to ±1.5% and repeatability to ±1%, based on 900 tests on Cu (high diffusive) and Pyrex (low diffusive) specimens (dia. 12.7mm, thickness 2.0mm) with at least 3 different devices at room temperature.
2 Accuracy of the specific heat capacity amounts to ± 4% and repeatability to ±2% when using 4 different reference materials, 550 shots, averaging for 5 shots, RT, recommended dimension, recommended shot parameters.
3 Pulse energy limited to 10 J to prevent non-linearity effects due to sample overheating and a detector signal not proportional to the temperature changes. Combining lower pulse energy with high detector sensitivity ensures accurate results.
4 Adjustable in steps of 1 µs

APPLICATIONS

The table below gives an overview of the thermal conductivity range of many material groups. As can be seen from the table, the laser flash method covers the broadest thermal conductivity range across the widest temperature spectrum.

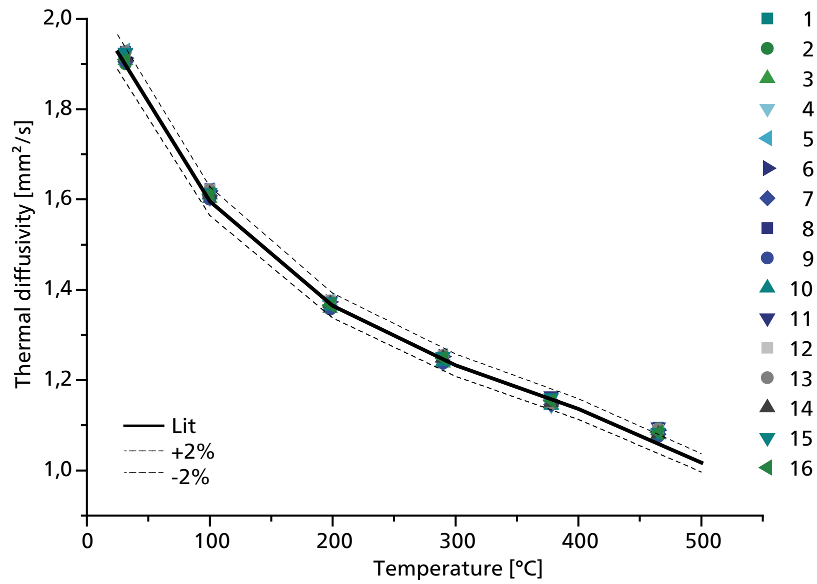


Made for World Class

Highest Efficiency by High Sample Throughput

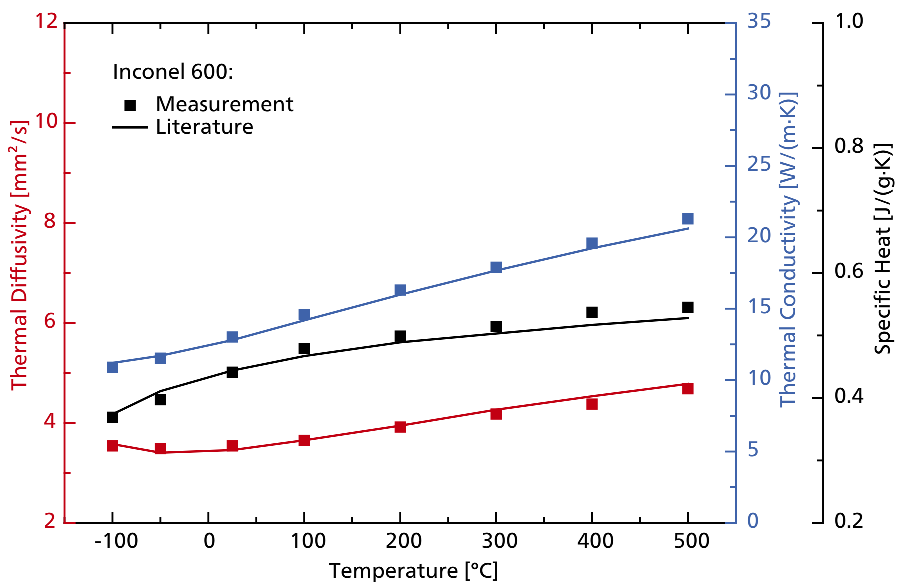
The design of the integrated automatic sample changer guarantees an optimum position for each of the 16 possible samples over the entire temperature range. The total measurement time is drastically reduced due to the fact that heating and cooling occur at the same time.

The high sample throughput of the LFA 467 *HyperFlash*® allows for efficient operation and minimizes time and effort in research and/or quality assurance.

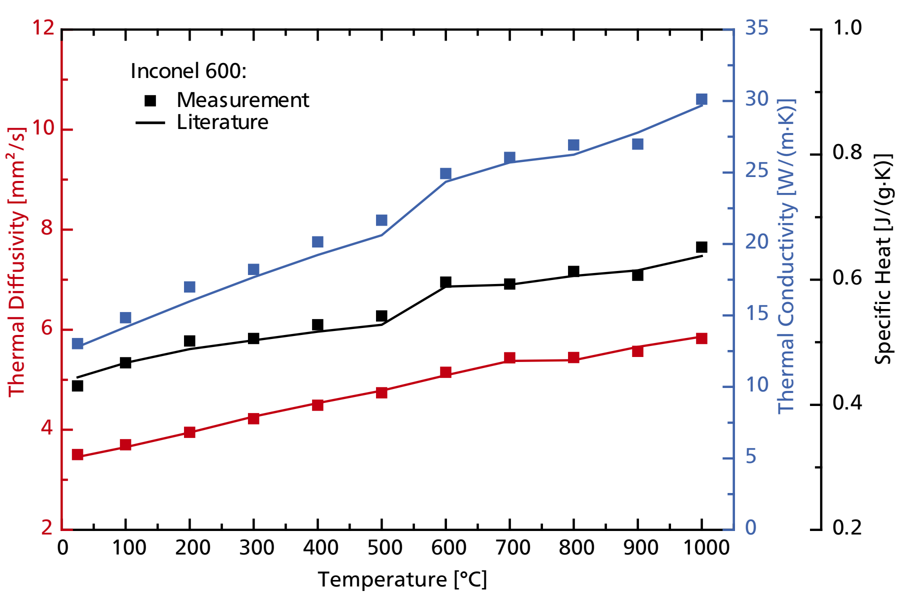


LFA 467 *HyperFlash*®: Measurement results of 16 Pyroceram samples (2.5 mm thick, 12.7 mm in Ø) between room temperature and 500°C obtained by one run. The evaluation of the thermal diffusivity shows a deviation of ±2% from literature data.

Applications



LFA 467 *HyperFlash*®: A single measurement setup can be used for measuring the thermal diffusivity in the temperature range from -100°C to 500°C. The literature data is represented by the solid lines.



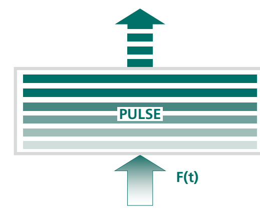
LFA 467 *HT HyperFlash*®: Measurement was carried out between RT and 1000°C for the determination of thermal diffusivity (red), thermal conductivity (blue) and specific heat (black); literature values are represented by the solid lines.

Highest Precision Over the Entire Temperature Range

Both plots on the left portray the determined thermal diffusivity (red symbols), thermal conductivity (blue symbols) and specific heat (black symbols) of Inconel 600 (reference material) over the entire temperature range of the LFA 467 *HyperFlash*® (upper plot) and the LFA 467 *HT HyperFlash*® (lower plot).

For all determined properties, the accuracy levels are below ± 3% at a precision level generally even better than ± 3%.

Thin and Highly Conductive Materials

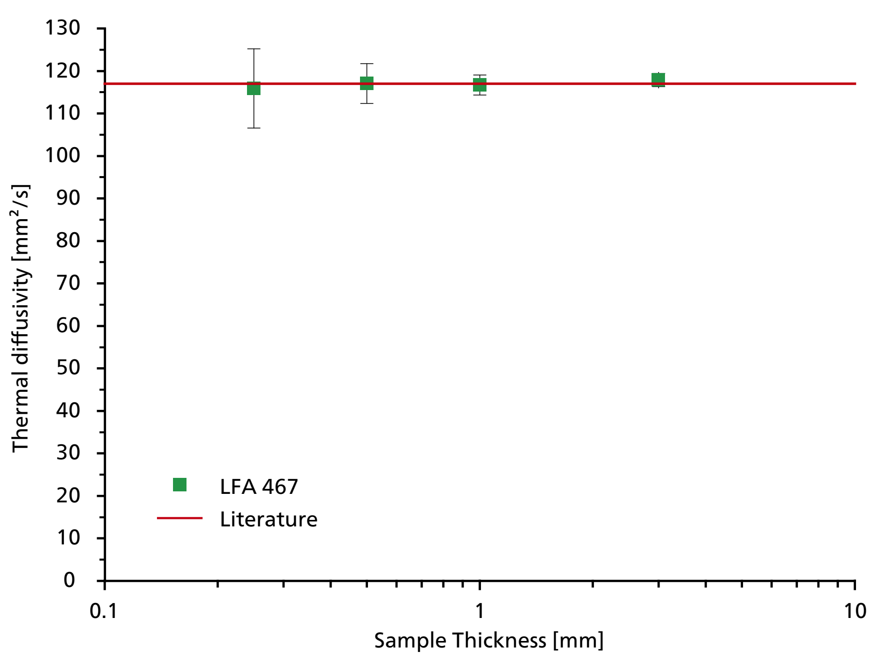


Copper

This plot shows measurements on copper samples with different thicknesses.
This example clearly proves that the system can successfully measure samples with very high diffusivities. In addition, by decreasing the sample thickness from 3.0 mm to 0.25 mm, these measurements confirm that even very thin samples can be tested with very high accuracy.

These measurements are only possible thanks to the 2 MHz data acquisition rate and 20 µs pulse length.

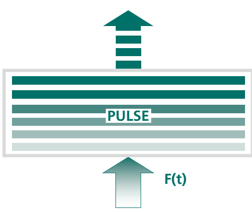
Sample preparation and thickness determination have to be carefully considered when measuring thin samples. This explains the increased uncertainties with decreasing sample thicknesses.



LFA 467 *HyperFlash*®: Thermal diffusivity values for the copper samples are in very good accordance with literature data, irrespective of the sample thickness.

High Data Acquisition and Short Pulse Length

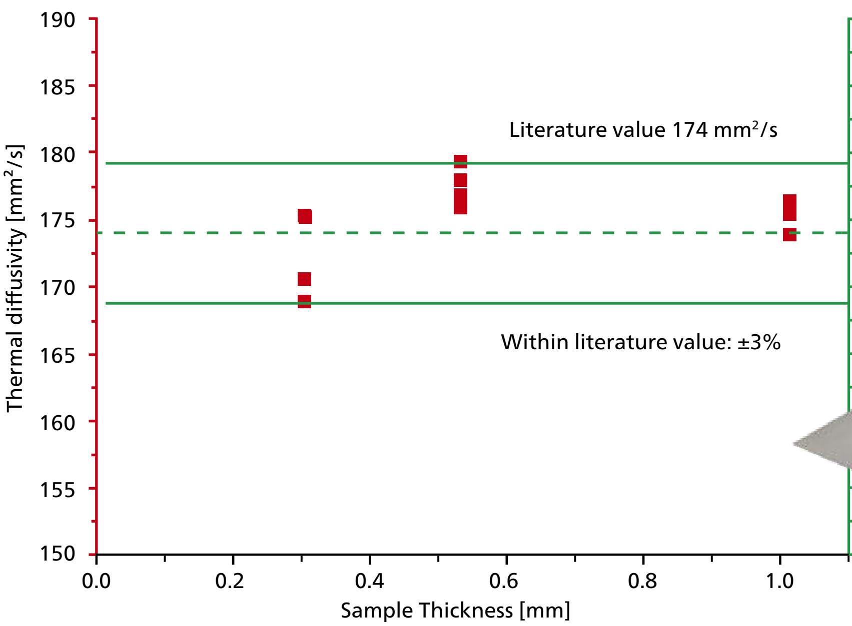
PREREQUISITE FOR THIN FILM MEASUREMENTS



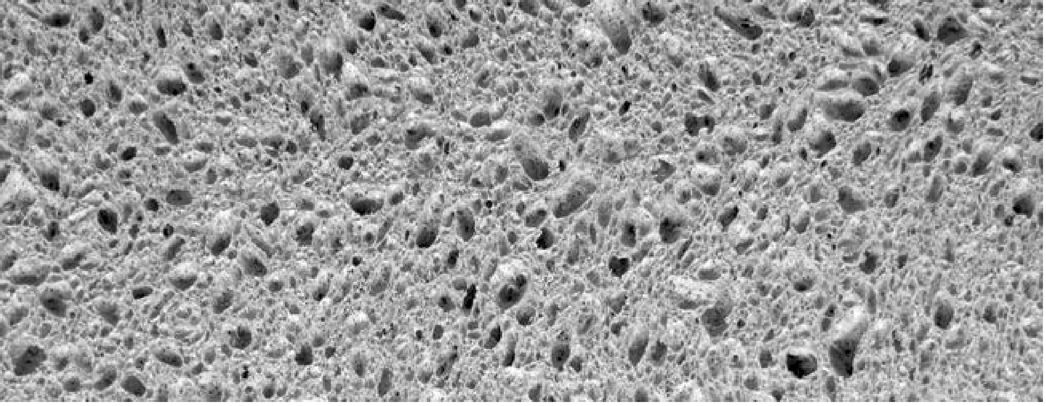
Silver

Silver shows a very high conductivity and is able to reduce the resistance of plated wire. This is particularly beneficial in high-frequency applications because the surface effect will result in increased current flow through the silver.

The thermal diffusivity was determined as a function of the silver plate thickness. The results for the different thicknesses – from low to high – are all within ±3% of the literature value for silver at 300 K.

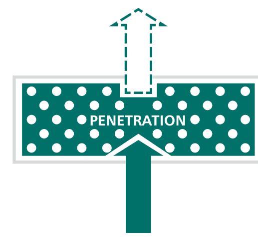


LFA 467 *HyperFlash*®: Thermal diffusivity values of silver specimens of different thicknesses are in very good accordance with literature data.

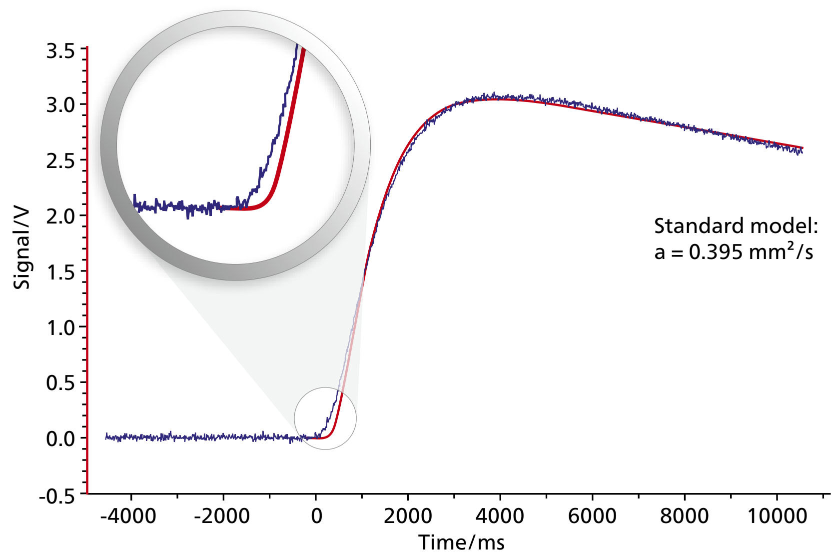


Porous Materials

PENETRATION FOR BEST MODEL FIT

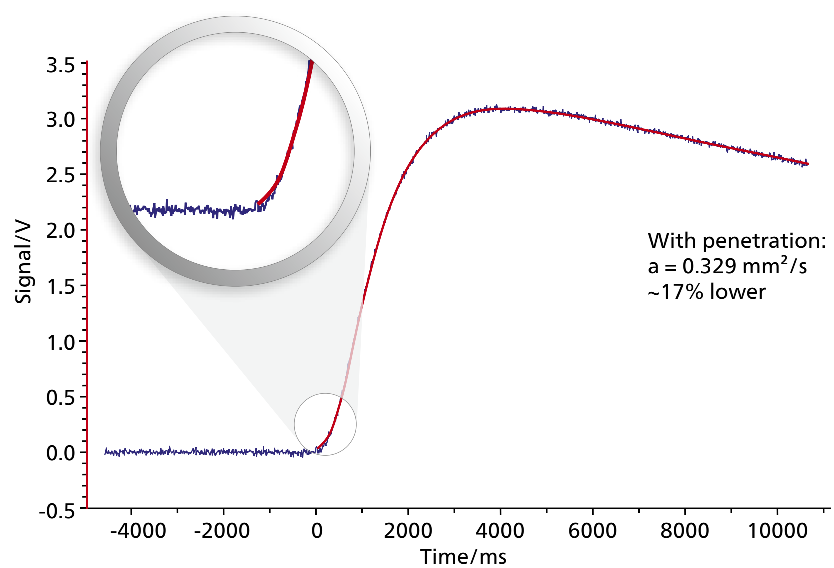


In standard flash method measurements, the pulse energy is totally absorbed on the front face of the specimen. A thermal wave results, traveling through the specimen's thickness before reaching the opposite face. However, in porous materials the absorption of the pulse energy is no longer limited to the front face – it is extended over a thin layer into the specimen thickness. The model for porous materials takes the penetration effect and the resulting decaying temperature distribution into consideration.



Filled Polymer Disc

In the example on the right, the thermal diffusivity of a filled polymer disc with holes was calculated using the standard (upper plot) and contrasted with the penetration model results (lower plot). The thermal diffusivity value yielded by the penetration model was approximately 17% lower than that of the standard model. Correctness of the result can be proven by measuring the same material without holes.



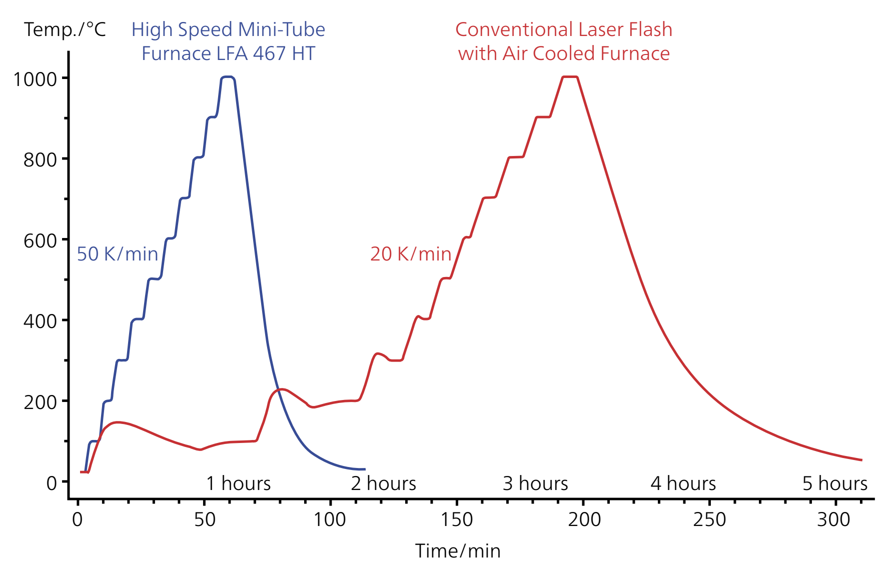
Excellent Stabilizing Behavior at High Temperature

SHORTEST MEASUREMENT TIMES AND HIGH SAMPLE THROUGHPUT

High sample throughput is essential for extensive usage of conventional LFA systems. It can be achieved by using an automatic sample changer, a fast furnace, or a combination of the two.
The LFA 467 *HT HyperFlash*® offers such a combination: Its four individual fast-responding mini-tube furnaces (for four specimens in total) are characterized by low thermal mass and superior stabilizing behavior.

The design offers homogeneous temperature distribution across all samples, which positively affects specific heat (c_p) determination. The combination of these specific features not only guarantees increasing sample throughput but is also a prerequisite for achieving short measurement times.

This figure shows the course of the sample temperature over time for two laser flash systems. A reference sample was measured from room temperature to 1000°C in steps of 100 K. The measurement time for the LFA 467 *HT* was only a third of that compared to the measurement with a conventional laser apparatus with air-cooled furnace.



Course of the sample temperature of an LFA measurement with mini-tube furnace (LFA 467 *HT*) and a furnace with standard air cooling

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