Light Flash Apparatus LFA 467 *HyperFlash*
Method, Technique, Applications of Thermal Diffusivity and Thermal Conductivity
Flash Method – The LFA 467 HyperFlash Pushes the Boundaries

Thermal Conductivity and Thermal Diffusivity: How much heat is being transferred, and how fast?

Now more than ever, in an increasingly competitive technological environment, efficiently devised heat transfer processes are at the heart of success. Researchers and engineers still want to know how fast an aluminum ingot solidifies, how to minimize thermal stresses within a steel block, how best to optimize the manufacturing of glass bottles, how fast the ceramic components of a catalytic converter heat up, how best to select the correct heat exchanger material for the thermal control of a processor, etc.

Such challenges, and many others, can not be met without accurate knowledge about two fundamental thermal properties: diffusivity and conductivity.

Furthermore, what is the best way to thermally characterize high conductivity materials at cryogenic and moderate temperatures, or ceramics and refractories at elevated temperatures? One accurate, reliable and elegant solution: the Flash Method.

Over the past two decades, NETZSCH has led the way in this technology, extending its 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 sets 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 pulse. From the resulting temperature excursion of the rear face measured with an infrared (IR) detector, the thermal diffusivity and, if a reference specimen is also used, also the specific heat is calculated. 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

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

The Flash method is a fast, non-contact, and efficient method for measuring the three fundamental thermal transport properties of thermal diffusivity, specific heat capacity, and thermal conductivity. In contrast to classical steady-state methods, LFA is often the technique of choice when a complete material characterization is desired.
Highest Sample Throughput – 16 Samples Simultaneously

Effective sample throughput in the entire temperature range is guaranteed by the sample changer for up to 16 samples. Your work and cost efficiency are additionally supported by the various available cooling devices.

Wide Temperature Range from -100°C to 500°C

Measurements from -100°C (e.g., 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. Different cooling systems and a variety of sensors complete the base of this true modular concept.

Intelligent Operation – Just a Click Away

Drag-and-drop software functions allow for easy instrument handling from measurement to analysis.

ZoomOptics – For Precise Measurement Results

Between detector and sample, a stepper motor actuated lens optimizes the field of view by software control. This helps to prevent measuring artifacts due to the contributions from the aperture stop, often characterized by a delayed IR signal. The precision of the test results is thereby greatly improved (patent pending).

Thin and High Conducting Materials by Ultra-Fast Sampling Rate

When testing metal (0.3 mm) and polymer (30 μm) foils, 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; DE1024241).

LFA 467 HyperFlash
Unique Concept – Unprecedented Features

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. The flash source is user-exchangeable and has a long lifetime.

One Furnace – Wide Temperature Range

With only a single furnace, measurements can be carried out from -100°C up to 500°C. The design of the furnace enables thermal coupling to different cooling devices. This considerably reduces the measurement times and allows for heating rates up to 50 K/min, while maintaining an excellent thermal stability.

Flexibility – 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, while the optionally available mercury cadmium telluride (MCT) detector allows measurements from -100°C to 500°C.

The instrument design ensures that, even at cryogenic temperatures, the measurements are always based on the infrared energy radiation from the sample’s surface.

Both detectors can be equipped with an optional liquid nitrogen auto-refill system, which comes in handy for long running tests without operator intervention.

Atmospheres

The atmosphere can be controlled via three integrated frits or optionally via mass flow controllers both 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.
ZoomOptics – Simplified Handling Allows for View of Solely the Sample Surface

Between the detector and the sample, a stepper motor (option) actuated lens optimizes the field of view. This patent-pending, software-controlled feature helps to avoid signal distortions from the sample’s immediate surroundings like masks or aperture stops. This feature is particularly valuable for small diameter samples. It also ensures that the detector is always kept within its linear responsivity range.

The Premise for Thin Films – High Data Acquisition

The data acquisition rate of the LFA 467 HyperFlash has been 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 measured.

Cooling Devices – Measurement Flexibility Guaranteed

The cooling devices using liquid nitrogen allow for temperatures as low as -100°C. Depending on the purge gas and the sample, lower temperatures can be achieved.

The cooling systems can be operated with the evacuation system running (below atmospheric pressure). This leads to a further reduction in heat loss and convection. However, 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.
The Sky’s the Limit – Sample Holders

Work Efficiency – Automatic Sample Changer

The LFA 467 HyperFlash has an integrated automatic sample changer for up to 16 samples. A tray for four sample holders (four samples each) can be used for round and square samples. In order to handle all samples without any further intervention by the operator, the Dewar volume of the detector is adapted to long measurement times.

Sample Dimensions

Each of the four sample holders can carry up to four samples with a maximum diameter of 12.7 mm each. The sample dimensions can be round or square. For measurements on large samples, inserts can be chosen to accommodate a sample diameter of up to 25.4 mm. Depending on the sample’s properties, its thickness can vary between 0.01 mm and 6 mm.
Specialties for Special Applications

In addition to the standard sample holders for solid samples of round and square geometries, NETZSCH also offers other sample holders which are designed for special applications on specific materials, including:

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

In the sample holder for liquids, continuous contact between liquid and crucible is guaranteed over the entire temperature range by the design of the sample holder – 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 in-plane direction and ones for tests under mechanical pressure are included in our product line. Customized sample holders are available upon request.
Proteus® Software – Key Features

The LFA 467 HyperFlash system runs under the Proteus® software on Windows® XP Professional, or on Windows® 7 32-/64-bit Professional, Enterprise or Ultimate operating systems. The Proteus® software includes everything you need to carry out a measurement and evaluate the resulting data. User-friendly menus combined with automated routines make this 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 Key Software Features

- Unicode version with support of local character sets
- Multiple-window technique for clear presentation
- Comparative analysis for up to 32 series of shots from the same database
- Convenient shot selection
- Loading of series of single shots with a preview of parameters and temperature program
- Storage of results for subsequent restoration and analysis
- Graphic export: EMF, PNG, BMP, JPG, TIF, or PDF; ASCII-file, export into Excel®-compatible CSV-format
- Model wizard including F-test for selecting of the best model fit
- Definition of an arbitrary number of temperature levels and number of shots per level
- Determination of specific heat with a comparative method
- Integrated database
- Determination of 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
- Calculation and graph of the $c_p$-curve
- Combined graph of raw data and theoretical model
- Thermal resistance calculation (2-layer model)
For thermal diffusivity under various boundary conditions, different baselines/models with correction of the pulse width are used, including:

- Adiabatic
- Cowan
- Improved Cape-Lehman (through consideration of multidimensional heat loss and non-linear regression)
- 2-/3-layer models (analysis by means of non-linear regression and consideration of heat loss)

Calculation Models, Corrections and Mathematical Operations

- In-plane
- Radiation correction (for transparent and semi-transparent samples)
- Heat-loss corrections
- Accurate pulse length correction, patented pulse mapping (patent no.: US7038209B2; US20040079886; DE1024241)
- Baseline correction
- Multiple-shots averaging
- Shot approximation via various mathematical functions (polynomials, splines, etc.)

- Classical models such as
  - Parker
  - Cowan 5
  - Cowan 10
  - Azumi
  - Clark-Taylor

LFA software, analysis includes heat loss and pulse length correction
## Technical Key Data for the LFA 467 HyperFlash

<table>
<thead>
<tr>
<th>Technical Key Data</th>
<th></th>
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</thead>
<tbody>
<tr>
<td><strong>Temperature range</strong></td>
<td>-100°C to 500°C</td>
</tr>
<tr>
<td><strong>Heating rate</strong></td>
<td>up to 50 K/min</td>
</tr>
<tr>
<td><strong>Cooling devices</strong></td>
<td></td>
</tr>
<tr>
<td>✓ CC 200 F3 (liquid nitrogen cooling): -100°C to 500°C; software-controlled</td>
<td></td>
</tr>
<tr>
<td>✓ CC 300 (liquid nitrogen cooling): -100°C to 500°C; optionally with liquid nitrogen re-filling, software-controlled, level monitoring</td>
<td></td>
</tr>
<tr>
<td><strong>Thermal diffusivity</strong></td>
<td>0.01 mm²/s to 1000 mm²/s</td>
</tr>
<tr>
<td><strong>Thermal conductivity</strong></td>
<td>0.1 W/(m·K) to 2000 W/(m·K)</td>
</tr>
<tr>
<td><strong>Accuracy</strong></td>
<td></td>
</tr>
<tr>
<td>✓ Thermal diffusivity: ± 3%</td>
<td></td>
</tr>
<tr>
<td>✓ Specific Heat: ± 5% (for standard materials)</td>
<td></td>
</tr>
<tr>
<td><strong>Repeatability</strong></td>
<td></td>
</tr>
<tr>
<td>✓ Thermal diffusivity: ± 2%</td>
<td></td>
</tr>
<tr>
<td>✓ Specific Heat: ± 3%</td>
<td></td>
</tr>
<tr>
<td><strong>Xenon flash lamp</strong></td>
<td></td>
</tr>
<tr>
<td>✓ Pulse energy: up to 10 Joule/pulse (variable), software-controlled</td>
<td></td>
</tr>
<tr>
<td>✓ Pulse width: 20 to 1200 μs, adjustable</td>
<td></td>
</tr>
<tr>
<td><strong>ZoomOptics</strong></td>
<td>Optimized field of view; requires no mask, patent-pending, option</td>
</tr>
<tr>
<td><strong>Pulse mapping</strong></td>
<td>Patented pulse mapping (US7038209, US20040079886, DE 10242741), for finite pulse correction and improved $c_p$ determination</td>
</tr>
<tr>
<td><strong>IR detectors</strong></td>
<td></td>
</tr>
<tr>
<td>✓ InSb: RT to 500°C</td>
<td></td>
</tr>
<tr>
<td>✓ MCT (Hg-Cd-Te): -100°C to 500°C</td>
<td></td>
</tr>
<tr>
<td>Both detectors are equipped with a 0.5-liter Dewar, 24 h nominal operating time</td>
<td></td>
</tr>
<tr>
<td><strong>Atmosphere</strong></td>
<td>Inert, oxidizing, static and dynamic</td>
</tr>
<tr>
<td><strong>Data acquisition</strong></td>
<td></td>
</tr>
<tr>
<td>✓ up to 2 MHz</td>
<td></td>
</tr>
<tr>
<td>✓ Min. measurement time (10 half times) down to 1 ms → for high conducting and/or thin samples (e.g., Al, Cu plates, thin films, etc.)</td>
<td></td>
</tr>
<tr>
<td>✓ Max. measurement time up to 120 s → for low conducting and/or thick samples (e.g., polymers, refractories, etc.)</td>
<td></td>
</tr>
<tr>
<td><strong>Gas control</strong></td>
<td>Frits with switches and optional reduced pressure and gas cycling system with MFC</td>
</tr>
<tr>
<td><strong>Automatic Sample Changer</strong></td>
<td>Integrated with 4 insets for up to 16 samples, round and square (4 x $\Omega_{\text{max}}$ 25.4 mm; 16 x $\Omega_{\text{max}}$ 12.7 mm)</td>
</tr>
<tr>
<td><strong>Sample holders</strong></td>
<td>For special applications such as molten polymers and low-viscosity liquids, resins during curing, pastes, powders, fibers, laminates, in-plane tests or tests under mechanical pressure</td>
</tr>
</tbody>
</table>
The processes of heat transfer are very important not only in many scientific areas but in many industrial ones as well. For example, when cooling casts, metals, semiconductors, etc., from the liquid phase into a solid state, the heat transfer can have a major impact on the thermophysical properties of the solid.

A variety of materials, whether made of chemicals or mixtures, have a homogenous composition. For those materials, the thermal conductivity is a true physical property, depending only on temperature, pressure and the composition.

However, particularly in the solid state, the thermal diffusivity and thermal conductivity also depend on the direction of the heat flow.

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

In current LFA systems, the field of view is fixed and large enough to accommodate large-diameter samples. When testing smaller-diameter samples, aperture stops are commonly used in an attempt to minimize the influence of the surroundings. This often results in a significant distortion of the thermal curve to the extent that the detector not only senses 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.

**ZoomOptics for an Optimized Field of View**

Field of view in standard LFA systems

**Standard LFA system without ZoomOptics yields measurements with distortions from 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.

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.
Applications – Reproducibility and High Precision over the Entire Temperature Range

Highest Precision over the Entire Temperature Range

This plot portrays the determination of the thermal diffusivity (red symbols), thermal conductivity (blue symbols) and specific heat (black symbols) of a standard material, Inconel 600, over the entire temperature range of the LFA 467 HyperFlash. The thermal diffusivity exhibits precision levels of to ± 3%. The specific heat exhibits a precision level generally even better than ± 3%. The literature data is shown as solid lines.

![Graph showing thermal diffusivity, conductivity, and specific heat of Inconel 600](image)

A single measurement setup can be used for measuring the thermal diffusivity in the temperature range from -100°C to 500°C.

Thin and Highly Conductive Copper

This plot shows measurements on copper samples with different thicknesses. It 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.

Sample preparation and thickness determination have to be carefully considered when measuring thin samples. This is the reason that the uncertainty increases as sample thicknesses decrease.

![Graph showing thermal diffusivity of copper samples](image)

Thermal diffusivity values for the copper samples are in very good accordance with literature data, irrespective of the sample thickness.
Highest Sample Throughput – High Efficiency

This plot shows the very high sample throughput of the LFA 467 HyperFlash. 16 Pyroceram samples (2.5 mm thick, 12.7 mm in diameter) were measured here in one run between room temperature and 500°C. The evaluation of the thermal diffusivity shows a deviation of ±2% from literature data.

This example clearly demonstrates that the design of the furnace with the integrated Automatic Sample Changer guarantees an optimum position for each of the 16 samples over the entire temperature range.

In addition, 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 reduces time and efforts of your tasks in research and/or quality assurance.
When it comes to Thermal Analysis, Adiabatic Reaction Calorimetry and the determination of Thermophysical Properties, 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.

The NETZSCH Group is a mid-sized, family-owned German company engaging in the manufacture of machinery and instrumentation with worldwide production, sales, and service branches.

The three Business Units – Analyzing & Testing, Grinding & Dispersing and Pumps & Systems – provide tailored solutions for highest-level needs. Over 3,000 employees at 163 sales and production centers in 28 countries across the globe guarantee that expert service is never far from our customers.

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