LFA for Thermal Diffusivity and Conductivity of Metals, Ceramics and Polymers

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Agenda

1. Heat Transfer - Basics
2. LFA - Method
3. LFA - Instrumentation
4. Application Examples
5. Summary
Examples of Heat Transfer From Everyday Life

..cooling the microprocessor chip in a laptop computer

Heat Transfer – Electronics
Examples of Heat Transfer From Everyday Life

- Heat transfer in the seats
- Heat transfer in the metals for the engine block
- Properties of the brake materials
- Insulation of the passenger cab
- Insulation of the latent heat storage system
- Ceramic coatings on the exhaust system
- Heat transfer optimization in electronic components

Photo: BMW AG – Photo-Gallery: 3 Series
Heat transfer (or heat) is the energy in transit (energy moving through a material) due to a temperature difference.

A quantitative expression relating the rate of heat transfer, the temperature gradient and the nature of the conducting medium is attributed to Fourier (1822):

\[
\dot{Q} = -\lambda A \frac{T_2 - T_1}{\Delta x} \quad ; \quad \dot{q} = -\lambda \frac{\Delta T}{\Delta x}
\]

A – area normal to heat flow

\(\dot{Q}\) – Heat flux = heat flow rate per unit area in direction x

\(\lambda\) – thermal conductivity of material
There are three main modes of heat transfer:

- **Conduction:** molecular interactions or free electrons
- **Convection:** exchange of heat between a fluid and a surface or an interface.
- **Radiation:** no medium required, electromagnetic phenomenon

Stefan-Boltzmann constant

\[ \frac{q}{A} = \sigma T^4 \]

Thermal conductance varies for each mode of heat transfer
# Heat Transfer Basics

<table>
<thead>
<tr>
<th>Thermal Conductivity (W/(m·K))</th>
<th>Materials and Components</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>Diamond</td>
</tr>
<tr>
<td>100</td>
<td>Silver, Copper, Silicon Carbide, Aluminum, Graphite</td>
</tr>
<tr>
<td>10.0</td>
<td>Silicon, Metals, Iron, Steel, Alumina, Carbon bricks</td>
</tr>
<tr>
<td>1.00</td>
<td>Silicon Nitride, Aluminosilicates, Porous Ceramics, Refractories</td>
</tr>
<tr>
<td>0.100</td>
<td>Concrete, Glass, Fire clay, Water, Wood, Polymers, Coal, Helium</td>
</tr>
<tr>
<td>0.010</td>
<td>Building boards, Oils, Fiber boards, Fiber insulations, Air, Polystyrene (exp.), PUR-foams</td>
</tr>
<tr>
<td>0.001</td>
<td>Krypton, Freons (gaseous), Vacuum insulation</td>
</tr>
</tbody>
</table>

(Termal Conductivity at RT)
Thermal Conductivity Measurement Methods

- Laser flash
- Hot wire
- Guarded hot plate
- Heat flow meter

Thermal Conductivity Measurement Methods by LFA - www.netzsch.com
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Heat Transfer – LFA Method

The measurement of the **thermal diffusivity** of a material is usually carried out by rapidly heating one side of a sample and measuring the temperature rise curve on the opposite side.

The time that it takes for the heat to travel through the sample and cause the temperature to rise on the rear face can be used to measure the through-plane diffusivity and calculate the through-plane thermal conductivity if the specific heat and density are known.

All three quantities as well as the thermal diffusivity can be functions of temperature.

\[
\lambda(T) = a(T) \cdot c_p(T) \cdot \rho(T)
\]
Heat Transfer – LFA Method

Flash Method:
- Measurement Principle
  Introduced by Parker et al. 1961

The front surface of a plan-parallel sample is heated by a short light or laser pulse.

The temperature rise on the rear surface is measured versus time using an IR detector.

The sample has been stabilized at the desired temperature, the laser fired several times over a span of a few minutes and the necessary data is recorded for each laser „shot“. The laser beam energy strikes and is absorbed by the front surface of the sample, causing a heat pulse to travel through the samples' thickness. The resulting sample temperature rise is fairly small, ranging from 0.5 to 2 °C. The temperature rise is amplified and recorded.
Heat Transfer – LFA Method

\[ a = 0.1337 \cdot \frac{d^2}{t_{1/2}} \]

\[ \Delta T_{\text{max}} \]

\[ \frac{1}{2} \Delta T_{\text{max}} \]

\[ \Delta T_{\text{max}} \sim \frac{1}{(m \cdot c_p)} \]

\[ \lambda(T) = a(T) \cdot c_p(T) \cdot \rho(T) \]
Heat Transfer: LFA Method

Detector Signal / arb. units

Polymer (slow)
Metal (average)
Graphite (fast)

Half-times

Thermal Diffusivity and Thermal Conductivity by LFA

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Flash Technique – Introduction
Netzsch LFA Systems for almost all application ranges

**NEW!**

**LFA 467 HyperFlash**
- -100°C to 500°C
- Sample changer for 16 samples
- ZoomOptics - IR detector
- Fastest data acquisition

**LFA 457 MicroFlash®**
- -125°C to 1100°C
- Sample changer for 3 samples

**LFA 427**
- -120°C to 2800°C
NETZSCH LFA 467 *HyperFlash*

- Table-top instrument
- Works with a high power Xenon-Flash lamp
- Temperature range: -100 to 500°C
- Integrated sample changer for 16 samples
- Different cooling options
- *ZoomOptics* for optimized field of view
No Zoom Optics — Distortion from Aperture

→ Field of view is fixed, large enough to cover big diameter samples.

→ Testing smaller diameter samples, apertures are used minimizing the influence of the surroundings.

→ Significant distortion of the thermal curve: detector not only senses the temperature rise of the sample, but also any fluctuations from the aperture.

→ Thermal curve shows either a continuously increasing trend or an extended leveling-off period.

Standard LFA system without zoom optics leads to measurements with distortions from the aperture.
NETZSCH LFA 467 *HyperFlash* – *ZoomOptics*

**Zoom Optics — No Distortion from Aperture**

- IR signal originates solely from the sample surface
- Large and small samples can be tested with an optimal sensing area.
- No contributions from the aperture
- Thermal curve conforms to the theoretical model, yielding correct diffusivity values.

Field of view by using a zoom optics, no aperture influences occur

Using the zoom optics the measurement signal shows no distortion from the aperture
NETZSCH LFA 467 HyperFlash – High Sample Throughput

Highest Sample Throughput – High Efficiency

→ 16 Pyroceram samples (2.5 mm thick, Ø 12.7 mm)

→ Thermal diffusivity shows a deviation of ±2% from literature data.

→ Optimum position for each of the 16 samples in the furnace over the entire temperature range.

→ Reduced measurement time

→ High throughput possible without operator’s intervention
Thin and High Conductive—Copper

→ Copper samples with different thicknesses from 3.0 mm to 0.25 mm.

→ 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. Especially graphite coating can lead to lower thermal diffusivity values.

<table>
<thead>
<tr>
<th>Sample Thickness [mm]</th>
<th>Thermal diffusivity [mm²/s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>120</td>
</tr>
<tr>
<td>1</td>
<td>120</td>
</tr>
<tr>
<td>10</td>
<td>120</td>
</tr>
</tbody>
</table>

Thermal diffusivity of the copper samples are in very good accordance with literature data independent from the sample thickness.
NETZSCH LFA 457 MicroFlash®

- Table-top unit with laser
- Temperature range: -125 to 1100 °C (with two furnaces)
- Integrated sample changer for 3 samples
LFA 457 *MicroFlash*® – Design

- Vertical system
- Two exchangeable furnaces
  - 125 … 500°C
  - RT … 1100°C
- Vacuum-tight (1 Pa)
- Laser pulse length ~ 0.33 ms
LFA 457 MicroFlash® – Sample Changer

Multiple Sample Arrangement
12.7 mm dia. and 10.0 mm square

Single Sample Arrangement
25.4 mm dia.
Standard Application – Stainless Steel

**SRM 1461 - Stainless Steel**

- **Thermal Diffusivity** / mm²/s
- **Specific Heat** / (J/g*K)
- **DSC 404 Test**

![Graph showing Thermal Diffusivity and Specific Heat vs. Temperature](#)
Application – Filled Rubber

LFA 457 Microflash®
Sample: Filled NR/BR
Thickness: 1.512 mm
Width: 12.7 mm

Thermal Diffusivity / mm²/s
Thermal Conductivity / W/(m*K)
Specific Heat / J/(g*K)
Thermophysical Properties Testing
NETZSCH LFA 427

- Temperature range -120 to 2800°C
- Vacuum-tight design $1 \times 10^{-5}$ mbar
- Different sample holders
  - standard sample holder
  - square sample holder
  - laminate sample holder
  - fiber sample holder
  - slag sample holder
  - liquid metal sample holder
NETZSCH LFA 427

- The system is vacuum-tight by design.
- Laser, sample and detector are in line (No mirrors are used) ⇒ Improved signal-to-noise ratio.
- One single system covers the temperature range from room temperature to 2000°C
Application: Silicon Carbide

LFA 427
Sample thickness: 4.01 mm
Sample holder: Graphite
Heating rate ≈ 6.5 K/min
Atmosphere: Argon

Thermal Diffusivity / mm²/s

Temperature / K

Temperature range: RT to 2800°C
NETZSCH offers a full range of flash systems (Xenon and laser based) for a broad application range.

The LFA 447 *NanoFlash*® allows for scanning the sample’s surface -> MTX.

The LFA 467 *HyperFlash* offers optimum performance for thin and high conductive materials in the temperature range from -100°C to 500°C. The integrated *ZoomOptics* guarantees for an optimized field of view and precise results.

The LFA 457 *MicroFlash*® is the right research instrument for applications from -125°C up to 1100°C.

The LFA 427 fulfills the requirements of extreme temperatures and special application ranges.
Some Reasons for the Success of the Flash Technique:

1. The method covers a broad measurement range (0.01 … 1000 mm²/s, 0.05 … 2000 W/(m*K))

2. The laser flash technique can easily be adapted to temperature-dependent tests (-125 … 2800°C)

3. Sample preparation is easy

4. It is a non-contact, non-destructive measurement technique

5. It can be employed on multi-layer systems

6. Fast testing times (generally a few seconds)
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Thermal Diffusivity of Metals: Cu and Al
Comparison to Literature Values
Polycarbonate (PC) in an Aluminum Container

In the Tg range the specific heat \( (C_p) \) rises from 1.5 to 1.8 J/gK (typical for 2\textsuperscript{nd} order transitions). The thermal conductivity \( (\lambda) \) shows a continuous progression. Although the thermal diffusivity \( (a) \) decreases constantly after the Tg, \( \lambda \) of the amorphous material continues to increase due to the increasing \( C_p \) and amounts to 0.3 W/mK at 300 °C (thus at injection moulding temperature).
Liquids and Molten Polymers in an Aluminum Container

Aluminum Sample holder for Low-Viscosity Liquids

Lid

Crucible

Sample
PA 46 alone has a very low thermal conductivity. With increasing filler (Al$_2$O$_3$) content in the polymer the specific heat decreases while the thermal diffusivity and conductivity increase.
Polyetheretherketone (PEEK) Filled with Carbon Nanotubes (CNT)

Prepared in Cooperation with:

ENSINGER
PEEK filled with CNT - Anisotropy

→ Orientation of Carbon Nanotubes due to injection moulding
Pure PEEK Matrix

Thermal Diffusivity and Thermal Conductivity by LFA

Sample: PEEK ohne CNT
Thickness: 3.030 mm
Density (25°C): 1.294 g/cm³

Glass transition
PEEK Filled with 7% CNT

Thermal conductivity increases by 14%

Sample: PEEK with 7% CNT
Thickness: 3.020 mm
Density (25°C): 1.323 g/cm³
**PEEK Filled with 14% CNT**

- **Thermal conductivity increases by 45%**

Sample: PEEK mit 14% CNT
Thickness: 2.920 mm
Density (25°C): 1.358 g/cm³
Increasing Thermal Conductivity with Increasing CNT Content

Sample: PEEK mit 0-14% CNT
Thickness: 3 mm
Density (25°C): 1.3 g/cm³

Thermal Conductivity (W/(m K)) vs. mass-% CNT at 25°C, 100°C, and 200°C.
Anisotropy: In-Plane Sample Holder

Vertical direction through-plane

Horizontal direction In-plane

In-plane sample holder

Thermal Diffusivity and Thermal Conductivity by LFA

www.netzsch.com
PEEK with CNT – Anisotropy

In-Plane (In the plate direction, center)

Sample: PEEK with 14% CNT
Thickness: 2.920 mm
Density (25°C): 1.358 g/cm³
Key Advantages of Netzsch LFA Systems
High sensitivity opens the doors for smallest sample dimension

- Low conducting thermoelectric material with bad dimension
- Diameter = 6 mm; Thickness = 3.6 mm (recommended: 12.6 / 2 mm)

**SbₓCoSb₃₋ₓ**

- Small noise level at bad conditions
- ~5 Joule sufficient
- No overheating of sample
- Reliable results

Medium pulse energy for all shots!
Key Advantages of Netzsch LFA Systems

Wrong results without pulse length correction

Theoretical Half Time: 1.6 ms

Sample: Silver
Thickness: 1.416 mm
Diameter: 10.29 mm

With New Pulse Length Correction
Without Pulse Length Correction

Literature Value
Key Advantages of Netzsch LFA Systems
Now we measure thinner samples thanks to 2 MHz data acquisition

- Copper with a thickness < 1mm is possible now

![Graph showing thermal diffusivity vs sample thickness]

- 2 MHz and 10 µs pulse length were not possible in the past!
Key Advantages of Netzsch LFA Systems

High accuracy at low viscosity liquids like water, oils …

[Graph showing thermal diffusivity vs. temperature for different sample holders.]

- Sample Holder No. 1
- Sample Holder No. 2
- Sample Holder No. 3

Literature Values +/- 5%

Thermal Diffusivity / mm²/s

Temperature / °C

Liquids (PEEK) Sample Holder
Key Advantages of Netzsch LFA Systems

Liquid metals (Sapphire) sample holder

Liquid Metals (Sapphire) Sample Holder

Sample: Cast Iron
Thickness: 1.5 mm
Density (RT): 7.2 g/cm³

Thermal Diffusivity and Thermal Conductivity by LFA

Thermal Diffusivity / mm²/s
Spec. Heat / J/(g*K)
Thermal Conductivity / W/(m*K)

Temperature / °C

0 200 400 600 800 1000 1200 1400
Key Advantages of Netzsch LFA Systems
Pressure sample holder for compressible samples

- CNT Powders with different particle sizes

![Graph showing thermal diffusivity (a) vs. density (ρ) for different particle sizes](image)

- Pressure Sample Holder

Thermal Diffusivity and Thermal Conductivity by LFA
Key Advantages of Netzsch LFA Systems

LFA must be gas tight for pressure depending measurements

Why it must be gas tight?

- for low pressure measurements
- Temp. < 10°C (to avoid condensation)
- Temp. > 300°C (to save graphite coating)

Sample: YOM2 (compressed between 2 graphite coated saphire discs)
Density (original / compressed): 1.360 / 1.423 g/cm³
Thickness (original / compressed): 1.130 / 1.080 mm
Mechanical Pressure: 2 bar (~2.5 kg on sample surface)

0.10
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00

Thermal Conductivity /W/(mK)

20 40 60 80 100 120 140 160 180 200 220
Temperature / °C

~940 mbar (ambient)
40 mbar
25 mbar
15 mbar
5 mbar

Thermal Diffusivity and Thermal Conductivity by LFA

www.netzsch.com
Key Advantages of Netzsch LFA Systems
Automatic model wizard for detection of best model fit

- Standard model
- Deviations after shot due to radiation

Model Wizard

- Radiation model
- Excellent fit after shot
- Consideration of radiation

Oxide Ceramics at 900°C
Key Advantages of Netzsch LFA Systems
Automatic model wizard for detection of best model fit

- More than 8% error if standard (wrong) model is used!
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The LFA method allows fast and reliable measurement of the thermal diffusivity/conductivity of solids and liquids over a broad temperature range.

Due to the speed of the method and the high accuracy, more and more researchers replace existing stationary methods with the flash technique.

Even critical sample geometries such as liquid metals or multi-layer samples can be analyzed using this method.
Thank you very much for your attention!