

LINSEIS

T H E R M A L A N A L Y S I S

THERMAL CONDUCTIVITY

LFA
TIM-Tester
THB
TFA
TF-LFA
HFM



Since 1957 LINSEIS Corporation has been delivering outstanding service, know how and leading innovative products in the field of thermal analysis and thermo physical properties.

Customer satisfaction, innovation, flexibility and high quality are what LINSEIS represents. Thanks to these fundamentals, our company enjoys an exceptional reputation among the leading scientific and industrial organizations. LINSEIS has been offering highly innovative benchmark products for many years.

The LINSEIS business unit of thermal analysis is involved in the complete range of thermo analytical equipment for R&D as well as quality control. We support applications in sectors such as polymers, chemical industry, inorganic building materials and environmental analytics. In addition, thermo physical properties of solids, liquids and melts can be analyzed.

LINSEIS provides technological leadership. We develop and manufacture thermo analytic and thermo physical testing equipment to the highest standards and precision. Due to our innovative drive and precision, we are a leading manufacturer of thermal Analysis equipment.

The development of thermo analytical testing machines requires significant research and a high degree of precision. LINSEIS Corp. invests in this research to the benefit of our customers.



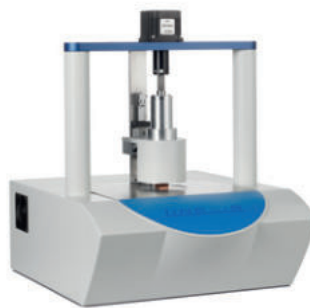
Claus Linseis
Managing Director

PRODUCT OVERVIEW

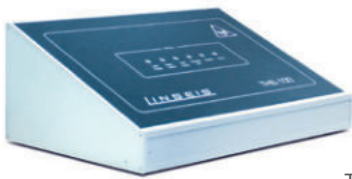
**Laser-Flash Analyser, Thermal Interface Materials Tester,
Transient Hot Bridge, Heat Flow Meter, Thin Film Laser Flash
and Thin Film Analyser**



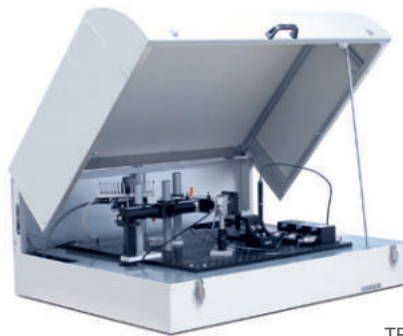
LFA 500/1000



TIM-Tester



THB 100



TF-LFA



TFA



HFM

LFA

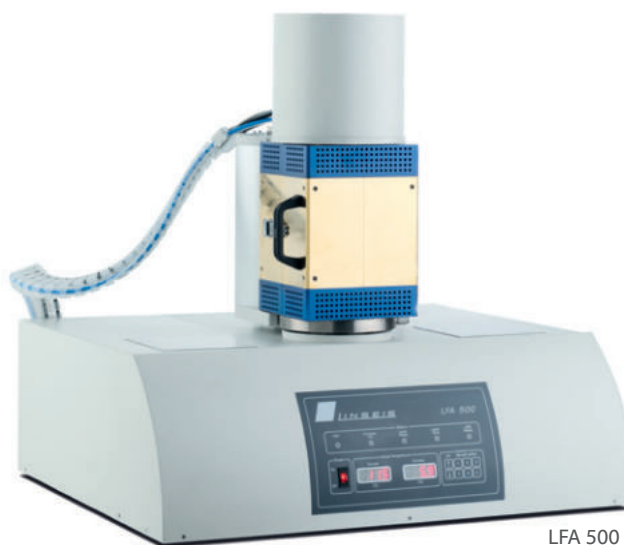
Information about the thermophysical properties of materials and heat transfer optimization of final products is becoming more and more vital for industrial applications. Over the past few decades, the flash method has developed into a commonly used technique for the measurement of the thermal diffusivity and thermal conductivity of various kinds of solids, powders, pastes and liquids. Application areas are electronic packaging, heat sinks, brackets, reactor cooling, heat exchangers, thermal insulators and many

more. Trouble-free sample preparation, small required sample dimensions, fast measurement times and high accuracy are only a few of the advantages of this non-contact and non-destructive measurement technique.

LINSEIS offers a variety of instruments to measure the thermal diffusivity/conductivity. The LFA 500/1000 Flash Analyser series provides a cost effective solution for the temperature range from -125 up to 2800°C.



LFA 1000

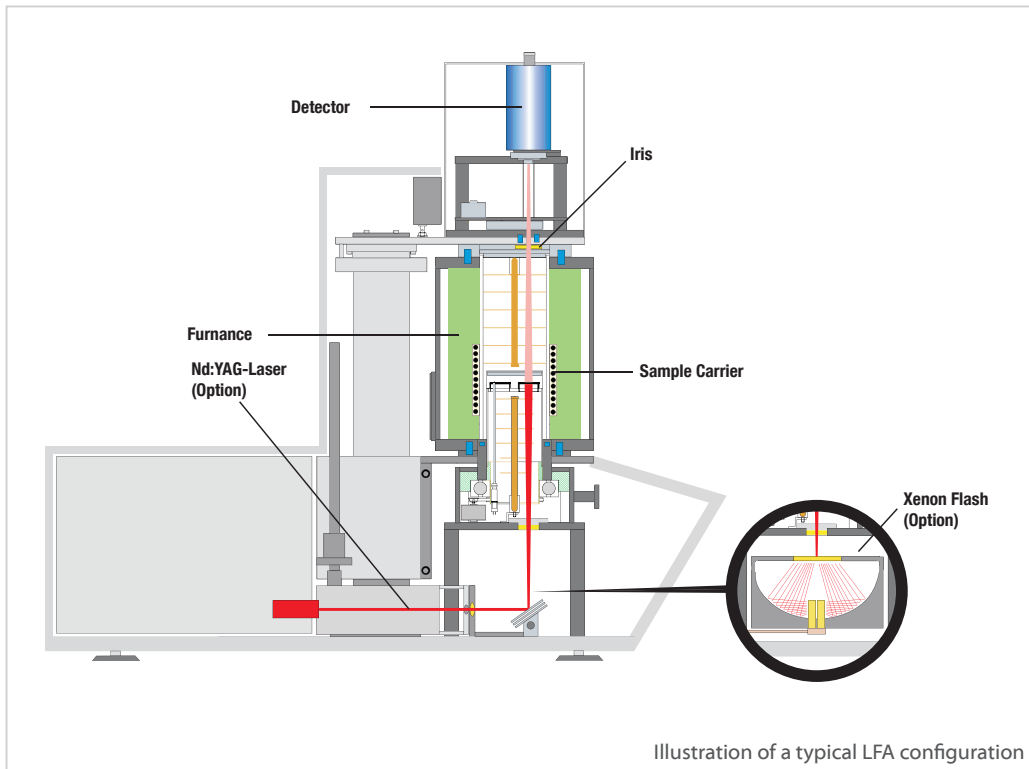


LFA 500

SYSTEM DESIGN

The vertical arrangement with sensor on top, sample in the middle and heat pulse source (Laser/Xenon Lamp) on the bottom, ensures easy handling and best possible measurement results. The pulse energy and duration are adju-

stable in the range of 0.05 to 25 Joule/pulse and 0.05 to 5 ms. Due to this flexibility all kinds of demanding samples (even thin film or ultra-low thermal conductive samples) can be analyzed.



ABSOLUTE METHOD

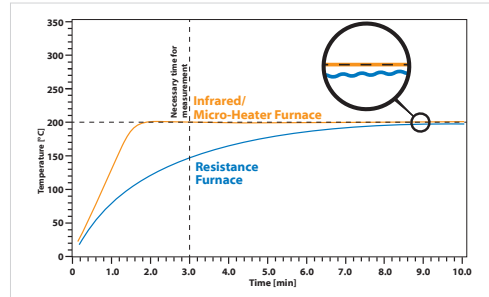
The used method is an absolute measurement technique (for thermal diffusivity), hence there is no need to calibrate the system. The LINSEIS

Flash Analysers operate in agreement with national and international standards, such as ASTM E-1461, DIN 30905 and DIN EN 821.

HIGH SPEED FURNACE

The LFA unit is either equipped with a high speed infrared furnace (LFA 500 - 500/1000) or with an advanced microheater (LFA 500/1250). This technology enables unmatched heating and cooling speed, unmatched temperature control, homogeneity and precision.

Because Time Matters



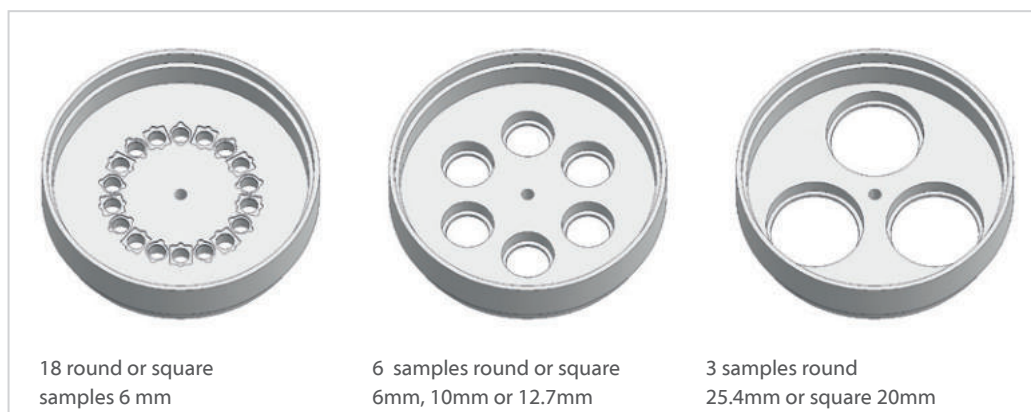
Comparison: time to reach the temperature stability. A high speed IR-micro-heater furnace reaches the set temperature much faster and delivers a superior isothermal temperature stability

DETECTORS

The system can be either equipped with an InSb or with a MCT detector, covering the complete temperature range from sub-ambient temperature up to 2800°C. Both are easily user exchan-

geable. An automatic LN₂ refilling accessory with Dewar flask for prolonged measurement cycles.

SAMPLE CARRIERS



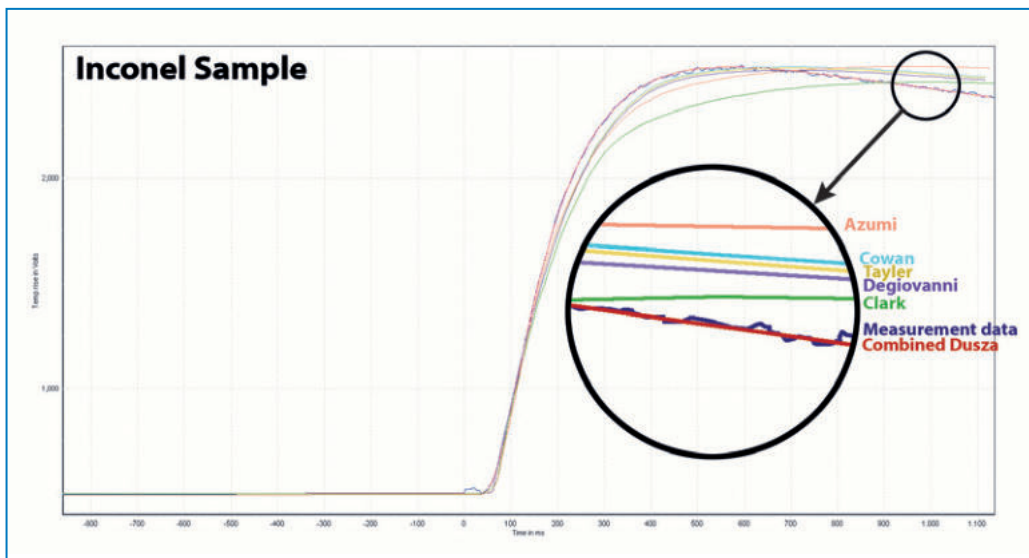
SOFTWARE: COMBINED MODEL ACCORDING TO DUSZA

Combining simultaneous heat loss and finite pulse-time correction in a single evaluation model, providing highest measurement accuracy.

One fits all

Easy controlling

No uncertainty



The plot illustrates a measurement on an Inconel sample. The raw data were evaluated using different common models. It can clearly be observed, that the combined model according to Dusza provides the best fit, resulting in the most accurate measurement result.

Conclusion

The combined model with nonlinear parameter estimation has been proven for more than 100 samples. In all cases it worked reliably and its results gave the correct adiabatic, finite pulse-time and heat loss corrected values. The two main advantages of the method are that no

operator choice between the different models and corrections is necessary, and that it can be applied to any type of sample. The quality of the fit can be checked by plotting the model curve and also as a numeric value.

SPECIFICATIONS

	LFA 500	LFA 1000/2000
Temperature range	–100°C up to 500°C RT up to 500 / 1000 / 1250°C (Boost function: 1450°C)	–125/–100°C up to 500°C RT up to 1250 / 1600 / 2000 / 2400 / 2800°C
Heating rate	0.01 up to 300 K/min	0.01 up to 20 K/min
Thermal Diffusivity	0.01 up to 2000 mm ² /s	0.01 up to 2000 mm ² /s
Thermal Conductivity	0.1 up to 4000 W/(m·K)	0.1 up to 4000 W/(m·K)
Accuracy	Thermal diffusivity ± 2.4% Specific Heat ± 5%	Thermal diffusivity ± 2.4% Specific Heat ± 5%
Repeatability	Thermal diffusivity ± 1.9% specific heat ± 3%	Thermal diffusivity ± 1.9% specific heat ± 3%
Flash source	Light flash 15 J/pulse variable pulse energy: software controlled	Laser Nd:YAG 25 J/pulse variable pulse energy: software controlled
Pulse width	0.05 up to 2 ms	0.05 up to 5 ms
IR-detector	InSb: RT up to 1450°C MCT: –100 up to 500°C	InSb: RT up to 2800°C MCT: –125 up to 500°C
Atmosphere	inert, oxidizing, reducing, vacuum	inert, oxidizing, reducing, vacuum
Vacuum	up to 10 ^{–5} mbar	up to 10 ^{–5} mbar
Data acquisition	2 MHz	2 MHz
Gas control	manual or MFC gas dosing systems	manual or MFC gas dosing systems
Sample holders	round or square - solid samples, powders, pastes, liquids, laminates - special holder for measurement under mechanical pressure	round or square - solid samples, powders, pastes, liquids, laminates - special holder for measurement under mechanical pressure
Sample numbers	up to 18 samples (depends on temperature range)	up to 3, 6 or 18 samples sample robot up to 3 samples

TIM-TESTER

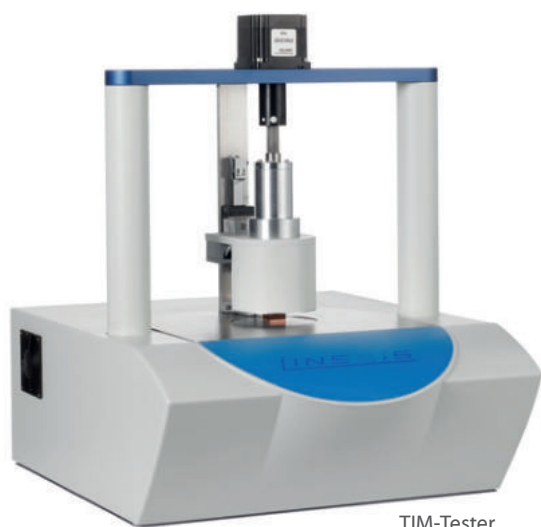
Waste heat management and thermal runaway protection in batteries and electronic packaging is becoming more and more important as power densities of these devices increase. Thermal management of these complex systems is not trivial and requires a fundamental understanding of how components and interface materials work together to shed heat.

Our LINSEIS Thermal Interface Material Tester (TIM-Tester) is the perfect solution for thermal management optimization of these complex systems.

The TIM Tester measures the thermal impedance of sample materials and identifies an apparent thermal conductivity for a wide range of materials from liquid compounds and pastes to hard solid materials. **The approach is consistent with the ASTM D5470 – standard.**

Thermal interface materials such as thermal fluids, thermal pastes (greases), phase change materials (PCM), solders or resilient thermal conductors are tested automatically by applying a pressure of up to 10 MPa (for \varnothing 25mm sample) and temperature of up to 300°C at the hot side.

The software interface allows the instrument to be operated automatically over a wide temperature and pressure range, while all test parameters are recorded in real time. This allows the user the freedom to fully explore an experimental design space for materials optimization. The sample holder is designed with sample size and shape flexibility in mind to accommodate actual size parts.



TIM-Tester



Exchangeable meter bars

SPECIFICATIONS

	TIM-Tester
Sample size	Round: from \varnothing 25 mm to \varnothing 40 mm Rectangular: from 25 mm x 25 mm to 40 mm x 40 mm Other sizes on request. Thickness: 0.01 mm up to 15 mm Other sizes on request.
Sample types	solids, powders, pastes, foils, liquids, adhesives
Resistance range	0.01 K/W – 8 K/W
Temperature range	RT up to 150°C RT up to 300°C (on request) -30°C up to 150°C (intracooler)
Temperature accuracy	0.1 K
Thermal conductivity range	0.1 up to 50 W/m·K Extended range on request
Contact pressure range	0 up to 10 MPa (depending on configurations)
Dimensions	675 mm x 550mm x 680 mm (H x W x D)
Cooling system	external chiller (several options)
Heating system	High performance resistance heater

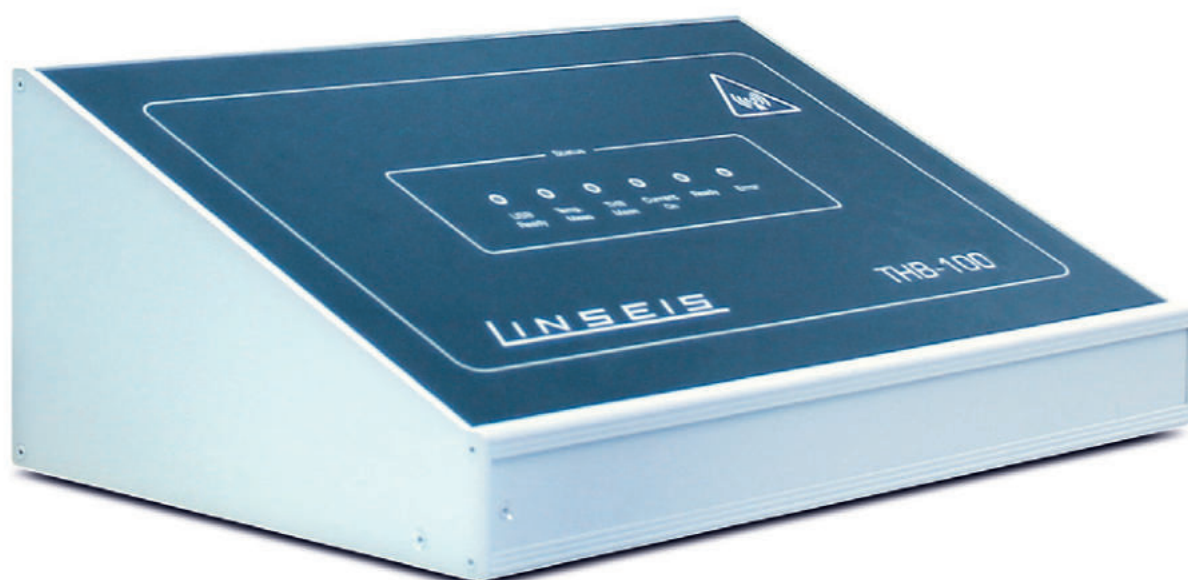
THB – TRANSIENT HOT BRIDGE

Instrument for rapid measurement of thermal conductivity, thermal diffusivity and specific heat

The flexible sensor is sandwiched between two flat surfaces of the specimens in order to assure a good thermal contact without air inclusions. Either the delivered sample holder or a weight can be used to optimize the contact resistance. The shapes of the faces, which are not in contact with the sensor, are of no importance, which helps to reduce the sample preparation to a minimum. There are no limitations regarding the maximum sample size, where else the minimum thickness depends on the samples thermal diffusivity. The measurement and evaluation are completely software controlled, run automatically and can be post-processed if needed. Measurements take only some seconds

up to a few minutes. The sensors are automatically identified by the software (THB-100 and THB-500). Multiple measurements with automatic calculation of the mean values are possible as well as the storage of measurement programs.

Measurements can be done either at room temperature (no further equipment required), elevated or sub-ambient temperatures. The setup, including the sensor, can be placed in a normal lab oven or climatic chamber, which are often available in laboratories. LINSEIS offers specially adapted furnaces covering temperatures from -150 up to 700°C.



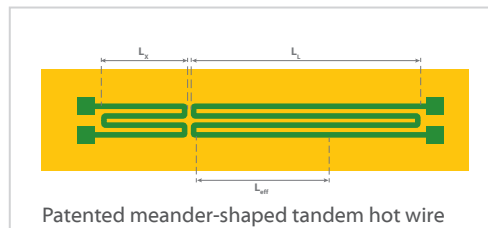
THE MEASUREMENT SET-UP

Advantages

- Short measurement times (seconds to few minutes)
- High accuracy due to patented sensor design
- Easy set-up and sample preparation
- Nondestructive measurement
- Absolute method - no calibration required
- Suitable for solids, liquids, powders, pastes etc.
- Temperature range -150 to 700 °C
- Measurements at different atmospheres, vacuum and pressures up to 150 bar
- Broad thermal conductivity range from 0.01 to 500 W/m·K
- Modular design

Transient Hot Bridge Method

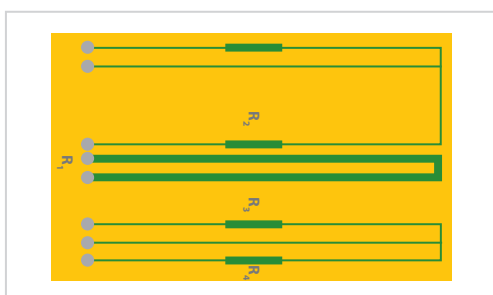
The THB measurement method initially, developed by the National Metrology Institute of Germany, is an optimized hot wire technique to measure all relevant thermal transport properties (thermal conductivity, thermal diffusivity and volumetric specific heat) with the highest possible accuracy.



SENSORS

For the THB-100 and THB-500 additional hot wire measurement techniques are available, based on innovative and patented sensors:

The Quasi-Steady-State (QSS) sensors have been developed for the measurement of high thermal conductivities. It combines the advantages of steady-state and transient measurement techniques and enables measurements in the range between 1 and 500 W/m·K



The LINSEIS Hot Point Sensors (HPS) work according to the transient plane method. They are suitable to measure small samples with dimensions down to 3 x 3 mm² and thicknesses < 1 mm and to measure anisotropic samples. Due to the small amount of heat, which is produced by the hot point sensors, they are a good choice to measure liquids with negligible convection.








SPECIFICATIONS

All sensor types are available as Kapton-foil-sensors and resist to temperatures from -150 up to +200°C. Ceramic sensors are available covering

a temperature range up to +700°C (THB-100 and THB-500).

	THB-1	THB 100	THB 500
Thermal Conductivity range	0 to 5 W/mK	0 to 100 W/mK	0 to 500 W/mK
THB/SENSOR/A	✓	✓	✓
THB/SENSOR/B	✓	✓	✓
THB/SENSOR/C	✗	✓	✓
QSS-Sensors	✗	✓	✓
Hotpoint-Sensors	✗	✓	✓

SENSOR TYPES

Sensor type	Sensor size	Min. sample size	Temperature range	Measuring range	suitable for...
THB/Sensor/A/B 	82 x 42 mm 42 x 22 mm	20 x 40 x 5 mm 10 x 20 x 3 mm	-150 up to 200°C	0.01 – 1 W/m·K	solids, powders, gases
THB/Sensor/A/B/Metal 	105 x 42 mm 54 x 22 mm	20 x 40 x 5 mm 10 x 20 x 3 mm	-150 up to 200°C	0.01 – 1 W/m·K	solids, powders, gases
THB/Sensor/C 	300 x 3 mm	10 x 10 x 10 mm	-150 up to 700°C	0.01 – 1 W/m·K	liquids, powders
THB/Sensor/D/E/QSS 	42 x 22 mm	22 x 42 x 3 mm	-150 up to 200°C	0.2 – 100 W/m·K 0.2 – 500 W/m·K	solids, powders, gases
THB/Sensor/G/HOTPOINT/ Kapton 	65 x 5 mm	3 x 3 x 2 mm	-150 up to 200°C	0.01 – 1 W/m·K	solids, liquids, powders, gases

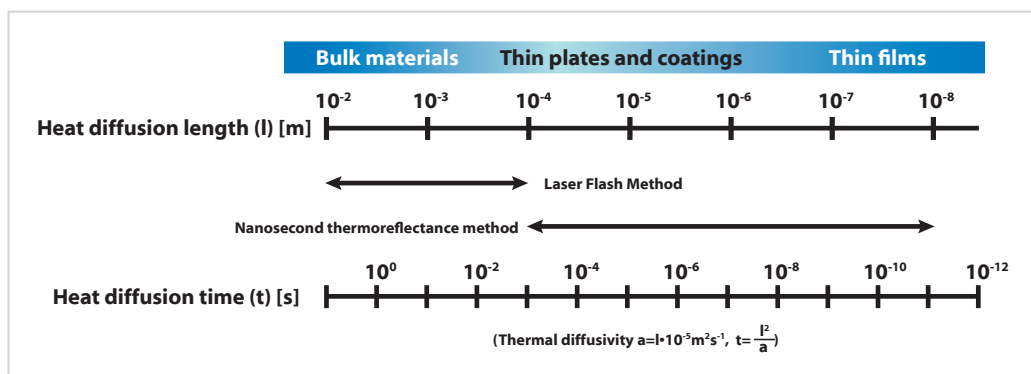
more sensors on request

TF-LFA

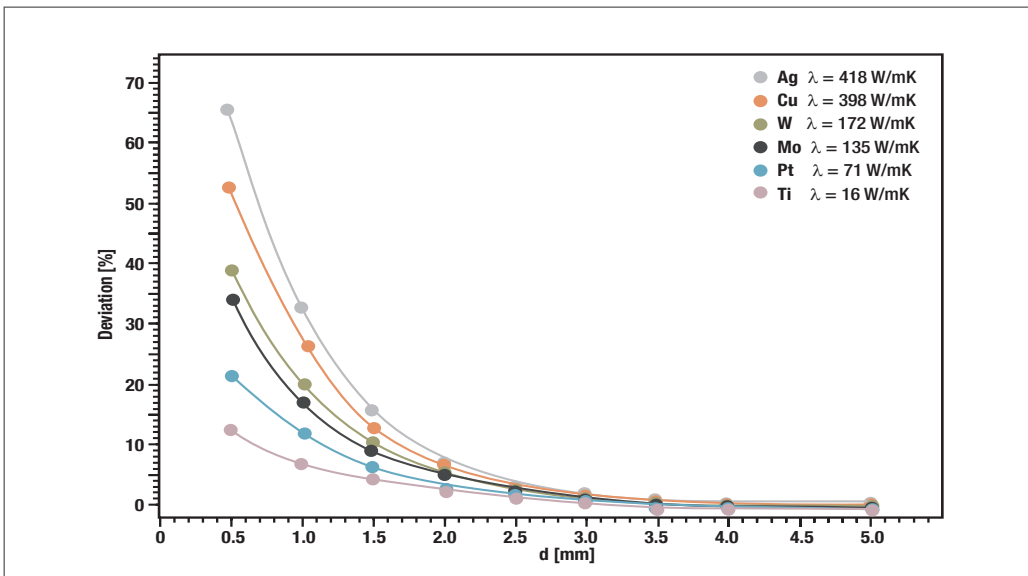
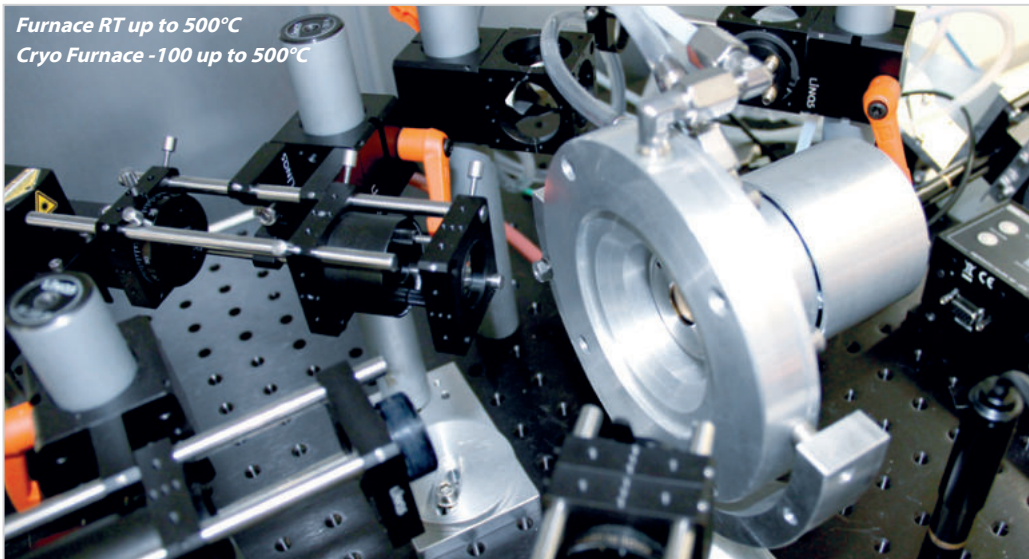
Thermophysical properties of thin films are becoming more and more important in industries such as phase-change optical disk media, thermo-electric materials, light emitting diodes (LEDs), phase change memories, flat panel displays, and the semiconductor industry. All these industries deposit a film on a substrate in order to give a device a particular function. Since the physical properties of these films significantly

differ from bulk material, these data are required for accurate thermal management predictions.

Based on the well established Laser flash technique, the LINSEIS "Thin-Film-Laserflash-Analyser" now offers a whole range of new possibilities to analyse thermophysical properties of thin films from 80 nm up to 20 μm thickness.



FURNACE OPTION



The graph from Schoderböck et. al., *Int. J. Thermophys.* (2009) illustrates the limitation of the classic Laserflash technique. Samples with a

thickness of less than 2mm (depending on the thermal diffusivity of the material) already show a significant deviation from literature values.

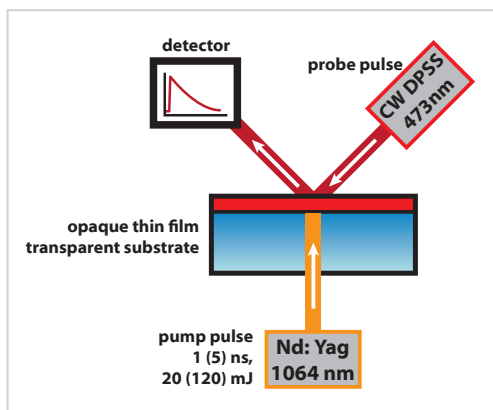
DESCRIPTION OF THE HIGH SPEED LASERFLASH TECHNIQUE

As thermal properties of thin layers and films differ considerably from the properties of the corresponding bulk material, a technique overcoming the limitations of the classical Laserflash method is required: the "High Speed Laserflash Method".

High Speed Laserflash Method

Rear heating Front detection (RF)

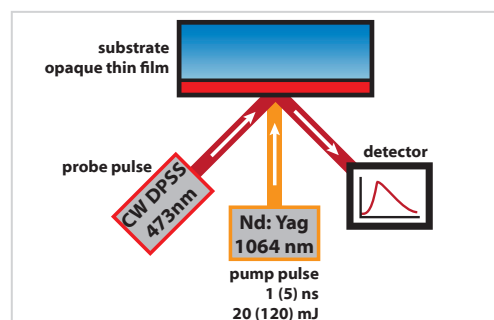
The measurement arrangement is the same as for the standard Laserflash technique: detector and laser are on opposite sides of the sample. Because IR-detectors are too slow for the measurement of thin layers, the detection is done by the so called thermoreflectance method. The idea behind this technique is, that once a material is heated up, the change in the reflectance of the surface can be utilized to derive the temperature change and thus the thermal properties. The reflectivity is measured with respect to time, and the data received can be matched to a model which contains coefficients that correspond to thermal properties.



Time Domain Thermoreflectance Method

Front heating, Front detection (FF)

The measurement geometry is called "front heating front detection (FF)" because detector and laser are on the same side of the sample. This method can be applied to thin layers on various substrates (e.g. non transparent substrates or very thin films) for which the RF technique is not suitable. For the measurement, a heating pulse is applied to the front side of the sample and the temperature rise at this spot is measured with a detection laser coming from the same side. The thermal diffusivity of the sample layer can be calculated by using the falling edge of the normalized temperature rise in combination with a multilayer model developed by LINSEIS in cooperation with Prof. David G. Cahill of the University of Illinois.



SPECIFICATIONS

	Thin Film Laser Flash Analyzer TF-LFA
Sample dimensions	Round with a diameter of 10 mm to 20 mm or square with edges of 10 to 17 mm
Film thickness	80 nm up to 20 μ m (depends on sample)
Temperature range	RT, RT up to 500°C or -100 to 500°C
Heating and cooling rates	0.01 up to 10 K/min
Vacuum	up to 10 ⁻⁴ mbar
Atmosphere	inert, oxidizing or reducing
Thermal diffusivity measuring range	0.01 mm ² /s up to 1000 mm ² /s
Pump-Laser	Nd:YAG Laser (1064 nm), maximum pulse energy: up to 120mJ/pulse (software controlled), pulse width: 1 - 5 ns, spot size 2-4 mm (depends on arrangement)
Probe-Laser	DPSS CW Laser (473 nm, 50 mW)
Photoreceiver	Si-PIN-Photodiode, active diameter: 0.8mm, bandwidth DC ... 400MHz, risetime: 1ns

THIN FILM ANALYZER

The LINSEIS Thin Film Analyzer TFA is the perfect solution to characterize a broad range of thin film samples in a very comfortable and quick way. It is an easy to use, single stand alone system and delivers high quality results using an optimized measurement design as well as the proven LINSEIS firm- and software package.

Motivation

Due to new research efforts in the field of semi-conducting materials, with a focus on size effects, there is a growing need for measurement setups dedicated to samples with small geometrical dimensions like thin films and nanowires, with considerably different physical properties than bulk material. The characterization of these samples is important to learn more about their structure and conduction mechanism but also important for technical applications.

Measurement Setup

The LINSEIS TFA is a chip-based platform to simultaneously measure the in-plane electrical and thermal conductivity, the Seebeck coefficient as well as the Hall constant of a thin film sample in the temperature range from -170°C up to 280°C and in a magnetic field of up to 1 T. Due to the design of the setup, time consuming preparation steps can be omitted and a nearly simultaneous measurement of the sample properties is achieved. Typical errors caused by different sample compositions, varying sample geometries and different heat profiles are avoided with this measurement method.

The system can handle a broad range of different materials. It is possible to measure samples with semiconducting behaviour as well as metals, ceramics or organic materials. Therefore many different deposition methods like PVD, CVD, spin coating or drop casting can be used.



PACKAGING OPTIONS

The following packaging options are available for the LINSEIS Thin Film Analyzer (TFA):

1. Basic device

Consists of measurement chamber, vacuum pump, basic sample holder with included heater, system integrated lock-in amplifier for the 3 ω -method, PC and LINSEIS software package including measurement and evaluation software. The design is optimized to measure following physical properties:

- λ - thermal conductivity
- c_p - specific heat

2. Thermoelectric package

Consisting of extended measurement electronics (DC) and evaluation software for thermoelectric experiments. The design is optimized for measuring the following parameters:

- σ - electrical conductivity / electrical resistivity
- S - Seebeck coefficient

3. Magnetic package

The design is optimized for measuring the following parameters:

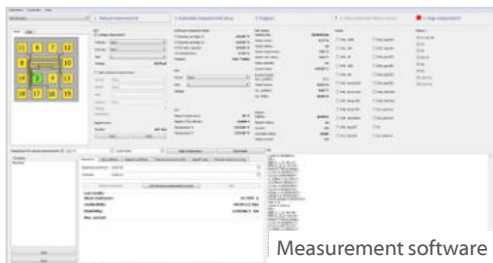
- A_H - Hall constant
- μ - Mobility
- n - charge carrier concentration

4. Low temperature option for controlled cooling down to 100 K

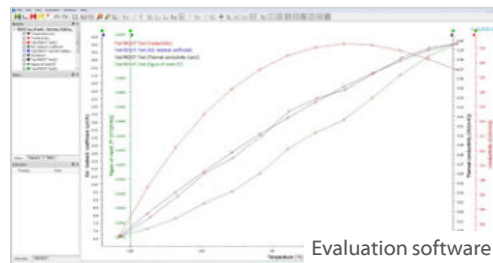
- TFA/KREG controlled cooling unit
- TFA/KRYO Dewar 25l

Software

The TFA software package consists of two parts. A measurement software which displays the actual values and which allows to define a measurement routine and the direct control of the setup. And an additional evaluation software for the post processing of the measured raw data.



Measurement software



Evaluation software

MEASURING PRINCIPLES

Pre-structured measuring chips

The chip is combining the 3 ω measurement technique for the thermal conductivity measurement with a 4-point Van-der-Pauw setup for the determination of the electrical transport properties. The Seebeck coefficient can be measured using additional resistance thermometers located near the sample. For an easy sample preparation either a strip-off foil mask or a metallic shadow mask can be used. This configuration allows for a nearly simultaneous characterization of a sample which has been prepared by either PVD (e.g. thermal evaporation, sputtering, MBE), CVD (e.g. ALD), spin coating, drop casting or ink-jet printing in one step.

Van-der-Pauw measurement

To determine the electrical conductivity (σ) and Hall constant (A_H) of the sample, the Van-der-Pauw method is used. After depositing the sample on the chip, it is already connected to four electrodes A, B, C & D at their edge. For the measurement, a current is applied between two of the contacts and the corresponding voltage

between the remaining two is measured. By clockwise changing of the contacts and repeating of the procedure, the resistivity of the sample can be calculated using the Van-der-Pauw equation.

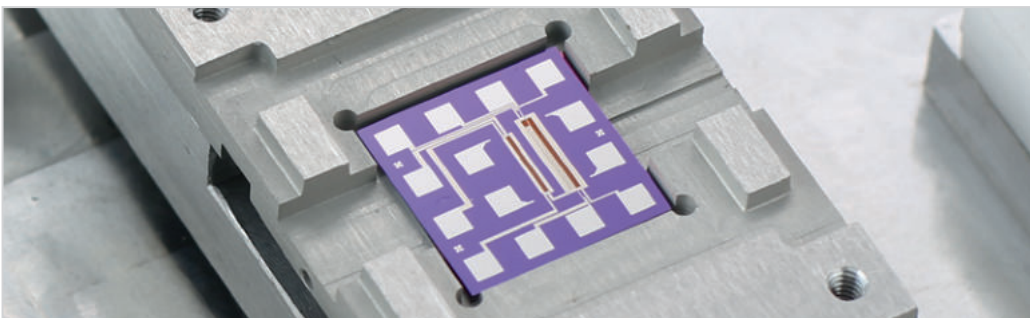
$$\exp\left(-\frac{\pi d}{\rho} \cdot R_{AB,CD}\right) + \exp\left(-\frac{\pi d}{\rho} \cdot R_{BC,DA}\right) = 1$$

By applying a magnetic field and measuring the corresponding change of the diagonal Van-der-Pauw resistance, the Hall constant of the sample can be calculated.

$$A_H = \frac{d}{B} \cdot \Delta R_{AC,BD}$$

For the determination of the Seebeck coefficient, an additional thermometer and heater is placed on the chip near the sample. This configuration allows for the measurement of the thermovoltage at different temperature gradients which can be used in order to calculate the Seebeck coefficient $S = -V_{th} / \Delta T$.

$$S = -V_{th} / \Delta T$$



SPECIFICATIONS

	TFA
Temperature range	RT up to 280°C -170°C up to 280°C
Sample thickness	from only a 5 nm to 25 µm (range depends on sample)
Measurement principle	chip based (pre-structured measurement chips, 24 pcs. per box)
Desposition techniques	include: PVD (sputtering, evaporation), ALD, spin coating, ink-jet printing and more
Measured parameters	Thermal conductivity (3ω) Specific heat
Optional	Seebeck coefficient Electrical resistivity / conductivity Hall constant / Mobility / Charge carrier concentration Permanent magnet 0.5 T or Electromagnet up to 1 T
Vacuum	up to 10^{-5} mbar
Electronics	integrated
Interface	USB
Measurement range	
Thermal conductivity	0.05 up to 200 W/m·K
Electrical resistivity	0.05 up to $1 \cdot 10^6$ S/cm
Seebeck coefficient	1 up to 2500 µV/K
Hall mobility	1 up to 10^7 (cm ² /Volt sec)
Charge carrier concentration	10^7 up to 10^{21} (1/cm ³)
Repeatability & accuracy	
Thermal conductivity	± 10% (for most materials)
Electrical resistivity	± 6% (for most materials)
Seebeck coefficient	± 7% (for most materials)
Hall constant	± 9 % for most materials

HFM

The Heat Flow Meter provides a rapid and easy to use instrument to determine the thermal conductivity properties of low thermal conductivity materials (e.g. like insulating materials) with a high level of accuracy. The instrument is designed for ASTM C518, JIS A1412, ISO 8301 and DIN 12667. The principle of measurement

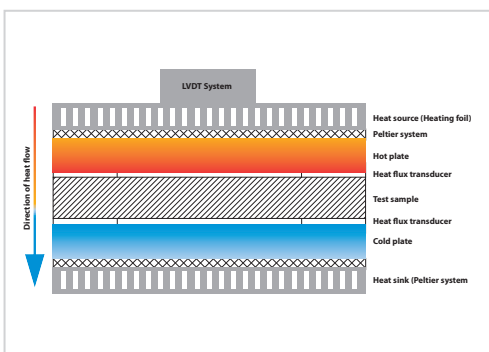
is to position a sample between a hot and a cold plate, and to measure the heat flow.

Service and maintenance

The robust system design and the unique “zero maintenance” Peltier heating and cooling cycle ensure a minimum of cost.

Test cycles

The double heat flux sensor configuration ensures shortest possible measurement cycles. A typical measurement for most samples can take as little as 15 minutes until the temperature stabilizes. Two heat flux sensors then measure the heat flow which is precisely defined between the hot and cold plate.



MEASUREMENT AND SAMPLE OVERVIEW

Below you will find an overview of the different measuring instruments for thermal conductivity. This should serve as an orientation. If you have any questions about a measurement or a material, please contact us.

Devices	LFA	TIM-Tester	THB	HFM	TFA	TF-LFA
Info	Most universal tool	For thermal inter-face materials	For fast and easy measurement	For insulating materials	Thin films on LINSEIS chip	For nm to μm films
Measurement						
Thermal Conductivity						
Thermal Diffusivity						
Specific Heat Capacity						
Thermal Resistivity						
Defined Pressure on Sample						
Atmosphere						
Temperature range	-125 to 2800°C	-20 to +300°C	-150 to +700°C	-40 to +90°C	-170 to +300°C	-100 to +500°C
Price	\$\$-\$\$\$	\$\$	\$	\$\$	\$\$\$	\$\$\$
Sample Type						
Solid						
Liquid						
Powder						
Paste						
Pad						
Thin film						



Measurement is possible



Measurement is probably possible



Measurement is not possible

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Products: DIL, TG, STA, DSC, HDSC, DTA, TMA, MS/FTIR, In-Situ EGA, Laser Flash, Seebeck Effect, Thin Film Analyzer, Hall-Effect

Services: Service Lab, Calibration Service

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