THERMAL ANALYSIS

THERMAL CONDUCTIVITY

LFA
TF-LFA
TFA
THB

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

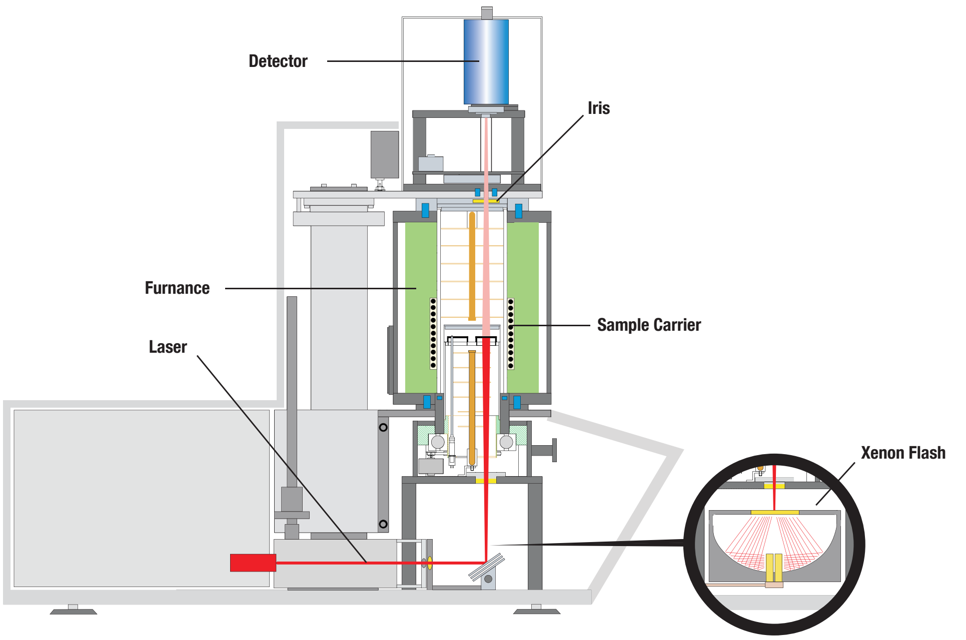
LFA

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 others. 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 500/1000/2000 Flash series provides a cost effective solution for the temperature range from -125 up to 2800°C.

SYSTEM DESIGN

The vertical arrangement with sensor on top, sample in the middle and Laser flash source on the bottom ensure easy handling and best possible measurement results. The pulse energy is adjustable in the range of 0.05 to 25 Joule/pulse. In addition the pulse duration can be adjusted. Due to this flexibility all kinds of demanding samples (even thin film or ultra-low thermal conductivity) can be analyzed.

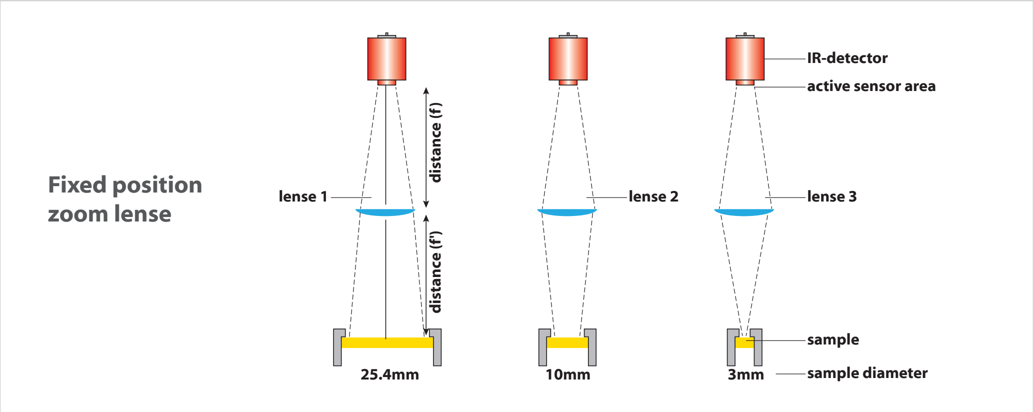


ABSOLUTE METHOD

The method used is an absolute measurement technique (for thermal diffusivity), hence there is no need to calibrate the system. The LFA 500/1000/2000 Flash Analyzers operate in agreement with national and international standards, such as ASTM E-1461, DIN 30905 and DIN EN 821.

VISION CONTROL

The vision control feature provides best signal quality for any sample dimension. The lens selection tool ensures superior signal quality for big and small samples. In addition, the arrangement overcomes positioning accuracy problems with existing zoom lens systems by ensuring best possible specific heat determination capabilities.



DETECTORS

The system can be either equipped with an InSb or with a MCT detector, covering the complete temperature range from sub-ambient up to 2800°C. Both are easily user exchangeable. An automatic LN_2 refilling accessory with Dewar can be ordered for prolonged measurement cycles.

ENVIRONMENTAL OPTIONS

The instrument can be operated under defined atmospheric conditions. It is either possible to attach a vacuum pump, in order to minimize heat loss effects or to attach an additional gas dosing systems to measure under specific atmospheres (argon, nitrogen, air, etc.).

UNMATCHED SAMPLE THROUGHPUT

Highest throughput in the market. The combination of sample robot and infrared furnace allows unbeaten measurement turnaround time. A typical measurement of up to 18 samples, as needed for quality control, takes only a few hours even for measurements over the complete temperature range.

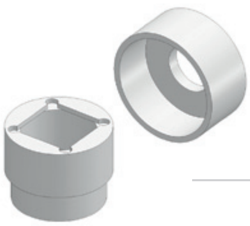
Sample carriers

18 round or square samples 3mm or 6 mm

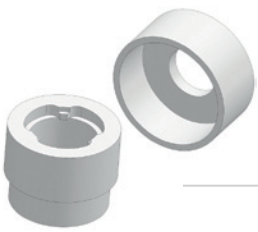
6 samples round or square 3mm, 6mm, 10mm or 12.7mm

3 samples round 25.4mm or square 20mm

Sample holder



Sample holder square samples 3x3mm / 10x10mm / 20x20mm

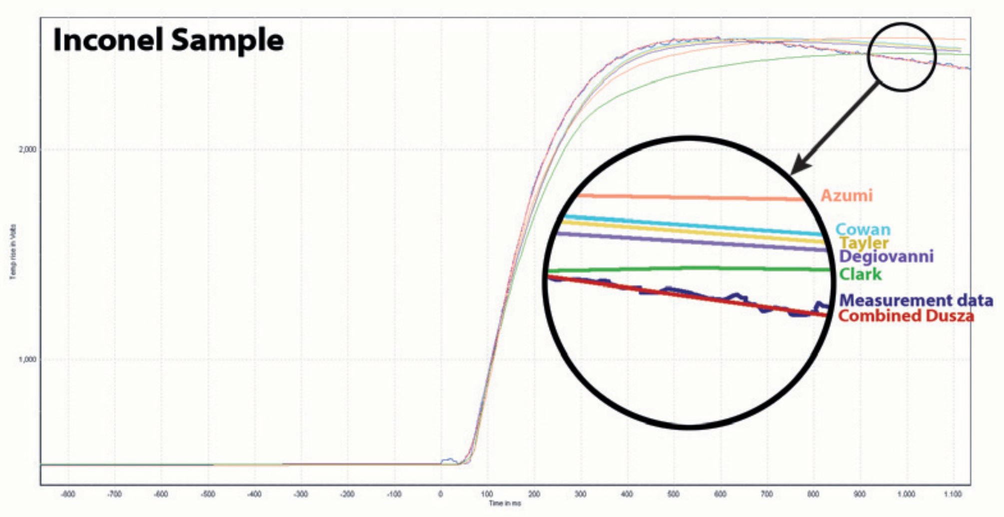


Sample holder round samples 3mm / 6mm / 10mm / 12.7mm / 25.4mm

More sample holders available!

SOFTWARE: COMBINED MODEL ACCORDING TO DUSZA

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



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 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, 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

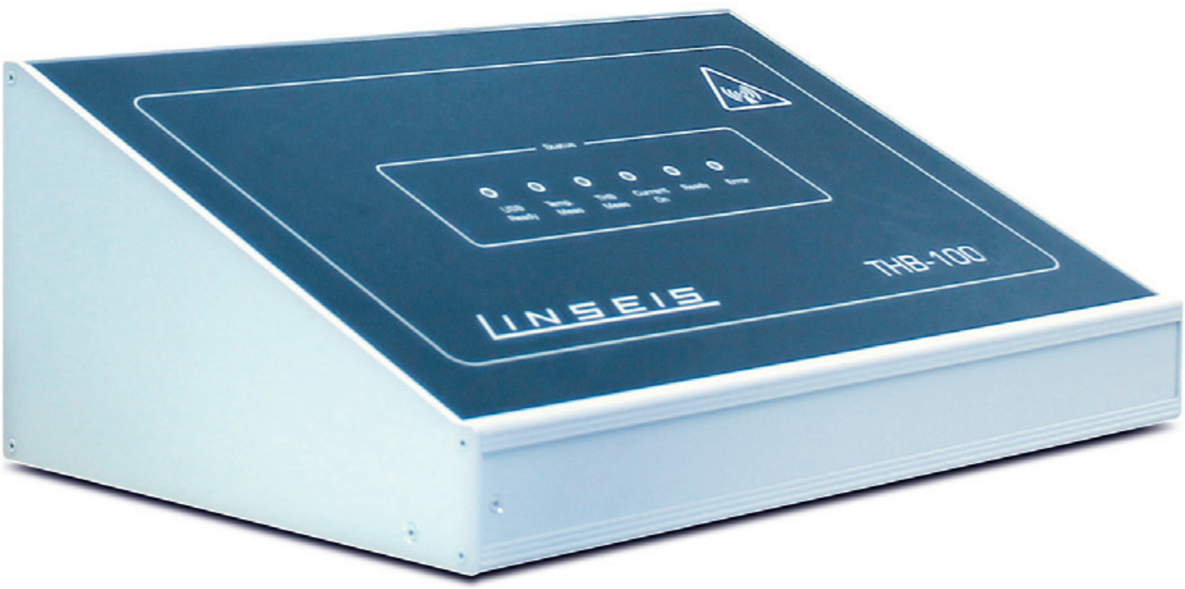
	LFA 500	LFA 1000/2000
Temperature range	–100 / –50°C up to 500°C RT up to 500 / 1000 / 1125°C	–125 up to 500°C RT up to 1250 / 1600 / 2000 / 2800°C
Heating rate	0.01 up to 100°C	0.01 up to 20°C
Thermal Diffusivity	0.01 up to 2000 mm^2/s	0.01 up to 2000 mm^2/s
Thermal Conductivity	0.1 up to 4000 W/(m•K)	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
Vision control	Perfect field of view	Perfect field of view
Pulse width	20 up to 2000 µs	0.05 up to 5 ms
IR-detector	InSb: RT up to 500 / 1000°C MCT: –100 up to 500°C	InSb: RT up to 1250 / 1600°C MCT: –100 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 aquisition	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 (option)	round or square - solid samples, powders, pastes, liquids, laminates - special holder for measurement under mechanical pressure (option)
sample numbers	up to 18 samples	up to 18 samples

THB – TRANSIENT HOT BRIDGE

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

Two flat surfaces of the specimens are put in contact with both sensor sides in order to assure a good thermal contact without air inclusions. Either the delivered sample holder or a weight can be used. The shapes of the faces which are not in contact with the sensor are of no importance so that sample preparation is reduced to a minimum. Minimum sample thicknesses depend on their thermal diffusivity. In most cases some mms are sufficient. After adjusting the heating power and heating time the measurement and the evaluation run automatically. Results 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/THB-500). Multiple measurements with automatic calculation of the mean values are possible as well as the storage of measurement programs.

Measurement can be done either at room temperature (no further equipment required) or at high or low temperatures. The set-up including the sensor can be placed in a normal lab oven or climatic chamber often available in laboratories. LINSEIS offers specially adapted furnaces covering temperatures from -150 up to 700°C.



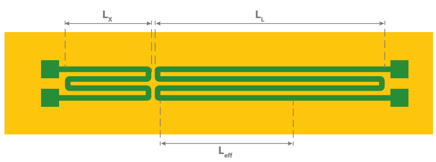
THE MEASUREMENT SET-UP

Advantages

- Short measurement times (seconds to few minutes)
- High accuracy due to patented sensor design
- Easy set-up and sample preparation
- Nondestructive measurement
- Absolut 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.

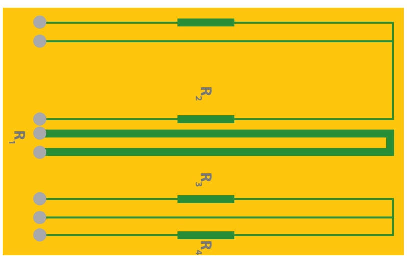


Patented meander-shaped tandem hot wire

SENSORS

For the THB-100 and THB-500 additional hot wire measurement techniques are available based on innovative and patented new 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 3x3x1 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

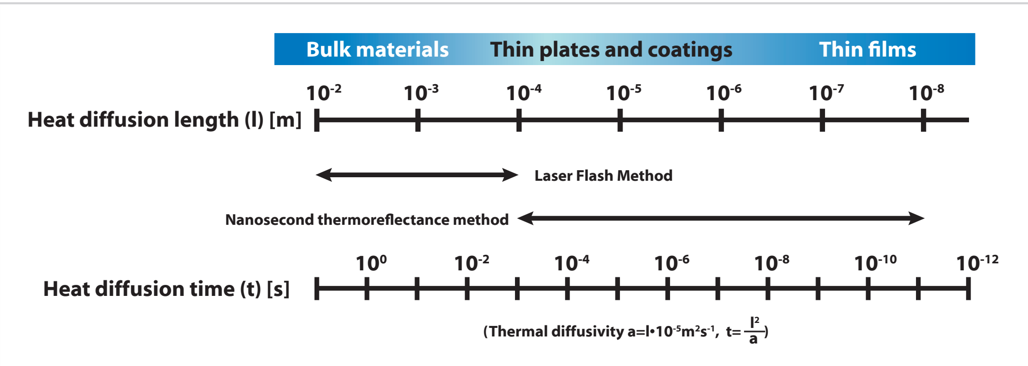
Sensor type	Sensor size	Min. sample size	Temperature range	Measuring range	suitable for...
THB/Sensor/A/B 	82 x 42 mm 42 x 22 mm	20 x 40 x 5 mm 10 x 20 x 3 mm	-150 up to 200°C	0.01 – 1 W/m·K	solids, powders, gases
THB/Sensor/A/B/Metal 	105 x 42 mm 54 x 22 mm	20 x 40 x 5 mm 10 x 20 x 3 mm	-150 up to 200°C	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

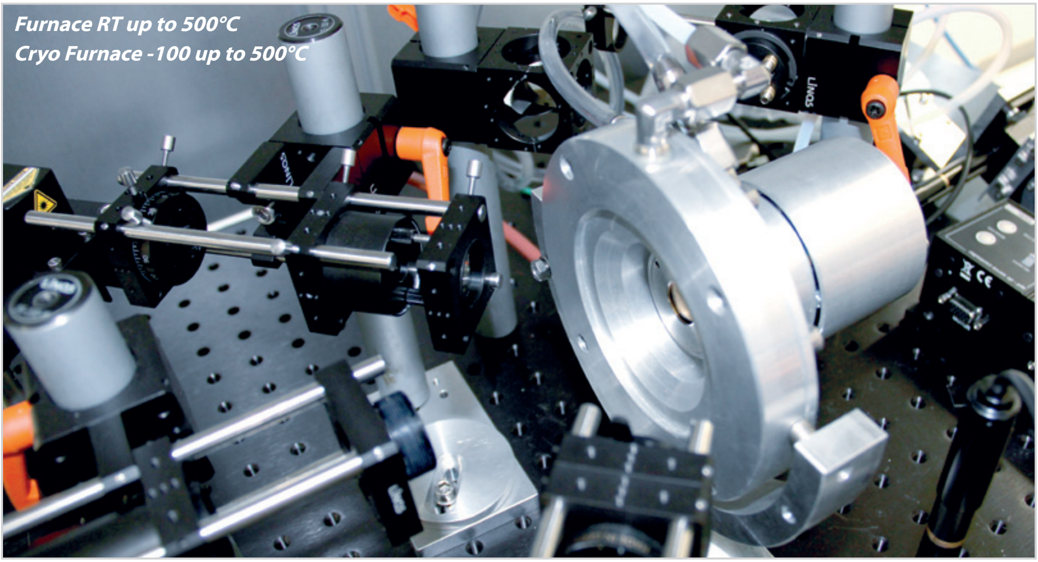
TF-LFA

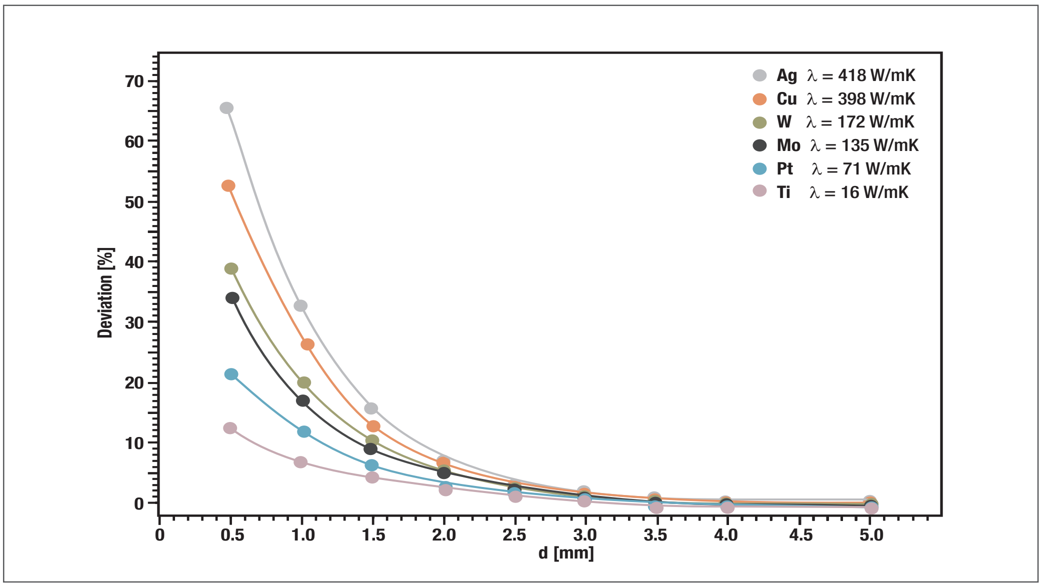
Thermophysical properties of thin films are becoming more and more important in industries such as, phase-change optical disk media, thermo-electric materials, light emitting diodes (LEDs), phase change memories, flat panel displays, and the semiconductor industry. All these industries deposit a film on a substrate in order to give a device a particular function. Since the physical properties of these films significantely 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-Analyzer" now offers a whole range of new possibilities to analyze thermophysical properties of thin films from 80nm up to 20µm 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.

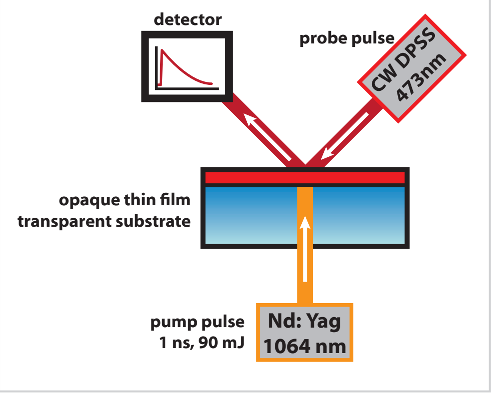
DESCRIPTION OF THE LASERFLASH TECHNIQUE

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)

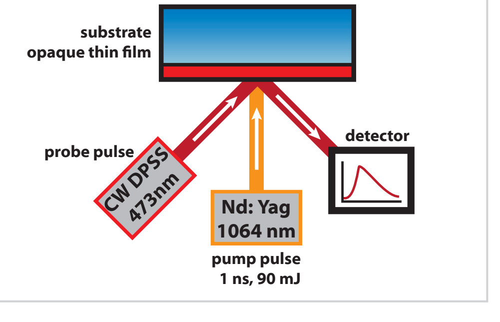
The measurement geometry is the same as for the standard Laserflash technique: detector and laser are on opposite sides of the samples. Because IR-detectors are to slow for measurement of thin layers, detection is done by the so called thermoreflectance method. The idea behind this technique is that 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 side. The thermal diffusivity of the sample layer can be calculated by using the falling edge of the normalized temperature rize in combination with a multilayer modell developed by Linseis in cooperation with Prof. David G. Cahill of the Univerity of Illinois.



SPECIFICATIONS

	Thin Film Laser Flash Analyzer TF-LFA
Sample dimensions	Round with a diameter of 10mm to 20mm
Film thickness	80nm up to 20µm (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°C/min
Vacuum	up to 10^{-4}mbar
Atmosphere	inert, oxidizing or reducing
Thermal diffusivity measuring range	0,01mm^2/s up to 1000mm^2/s
Pump-Laser	Nd:YAG Laser (1064 nm), maximum pulse current: 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 firmware and software package.

Motivation

Due to new research efforts in the field of semiconducting 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 organics. Therefore many different deposition methods like PVD or spin coating and drop casting can be used.

PACKAGING OPTIONS

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

1. Basic device (incl. transient package)

Consists of measurement chamber, vacuum pump, basic sample holder with included heater, system integrated lock-in amplifier for the 3ω-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

• ε - emissivity (depends on material)

2. Thermoelectric package

Consisting of extended measurment 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

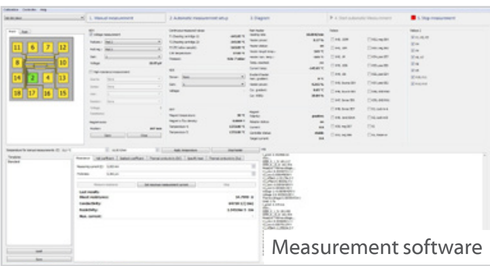
4. Low temperature option for controlled cooling down to 100 K

• TFA/KREG controlled cooling unit

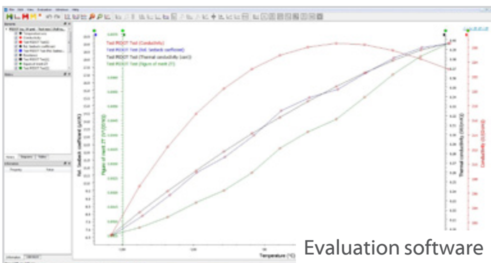
• TFA/KRYO Dewar 25l

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 dircet 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 3ω 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.

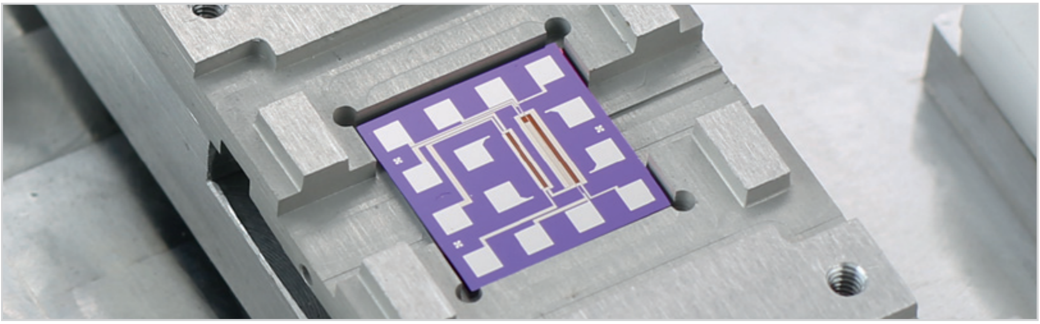
$$\exp\left(-\frac{\pi d}{\rho} \cdot R_{AB,CD}\right) + \exp\left(-\frac{\pi d}{\rho} \cdot R_{BC,DA}\right) = 1$$

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 measurment of the thermovoltage at different temperature gradients wich 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 few nm to µm range (depends on sample)
Measurement principle	chip based (pre-structured measurement chips)
Desposition techiques	include: PVD (sputtering, evaporation), ALD, spin coating, ink-jet printing and more
Measured parameters	thermal conductivity (3ω) specific heat
Optional	Seebeck coefficient electrical resisitivity / 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

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Services: Service Lab, Calibration Service

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