LINSEIS

THERMAL ANALYSIS

THERMO-ELECTRIC MATERIALS

Instrumentation for the Characterization of Materials & Modules

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

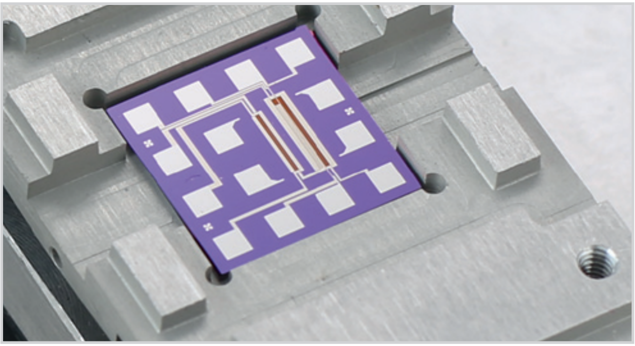
Customer orientation, innovation, flexibility and high quality are what LINSEIS stands for. Thanks to these fundamental characteristics, our company enjoys an exceptional reputation among worldwide leading scientific institutes and industrial companies. LINSEIS has been offering benchmark products in highly innovative branches for many years.

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

LINSEIS thrives for technological leadership. We develop and manufacture thermoanalytic and thermophysical testing equipment to the highest standard and precision. Due to our innovative drive and ultimate precision, we emerged as a leading manufacturer of Thermal Analysis equipment.

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

Claus Linseis
Managing Director



German engineering

The strive for the best due diligence and accountability is part of our DNA. Our history is affected by German engineering and strict quality control.

Innovation

We want to deliver the latest and best technology for our customers. LINSEIS continues to innovate and enhance our existing thermal analyzers. Our goal is constantly develop new technologies to enable continued discovery in Science.

Thermoelectricity describes the reciprocal interaction of temperature and electricity and their conversion into another. There are three different effects which describe the reversible interaction - the Seebeck-Effect, the Peltier-Effect and the Thomson-Effect. Nearly always these effects appear together.

Field of Application

In recent years, thermoelectricity has been increasingly used in applications such as portable refrigerators, beverage coolers, electronic component coolers, and metal alloy sorting devices. Furthermore it is used in thermoelectric generators for waste heat recovery (for example in cars to decrease CO_2 emission) and solid state cooling or Peltier-elements. Thermoelectric generators (TEG) are available since the early 1960s with a power output range from 10 to 550 W. Some advantages of the TEGs are a high reliability, long service intervals, low maintenance and a long durability. One of the most commonly used materials for such applications is Bismuth telluride (Bi_2Te_3), a chemical compound of bismuth and tellurium.

Figure of Merit

Altenkirch (1909, 1911) showed that good thermoelectric materials should possess large Seebeck Coefficients, high Electrical Conductivity and low Thermal Conductivity. Thus, the thermoelectric efficiency of a material is given by the dimensionless figure of merit ZT, which is a combination of these three values and is defined as:

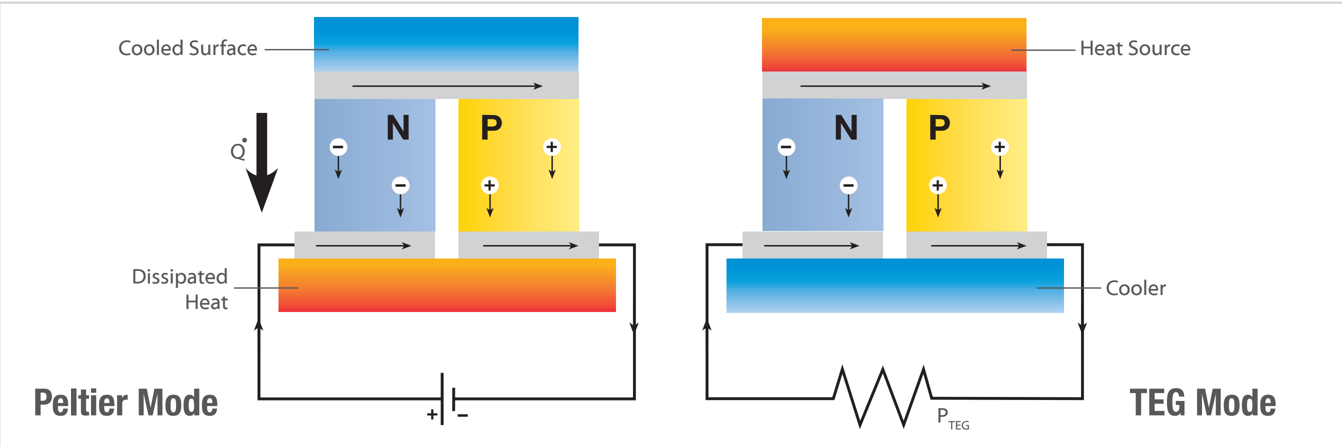
$$ZT = \frac{S^2 \cdot \sigma \cdot T}{\lambda}$$

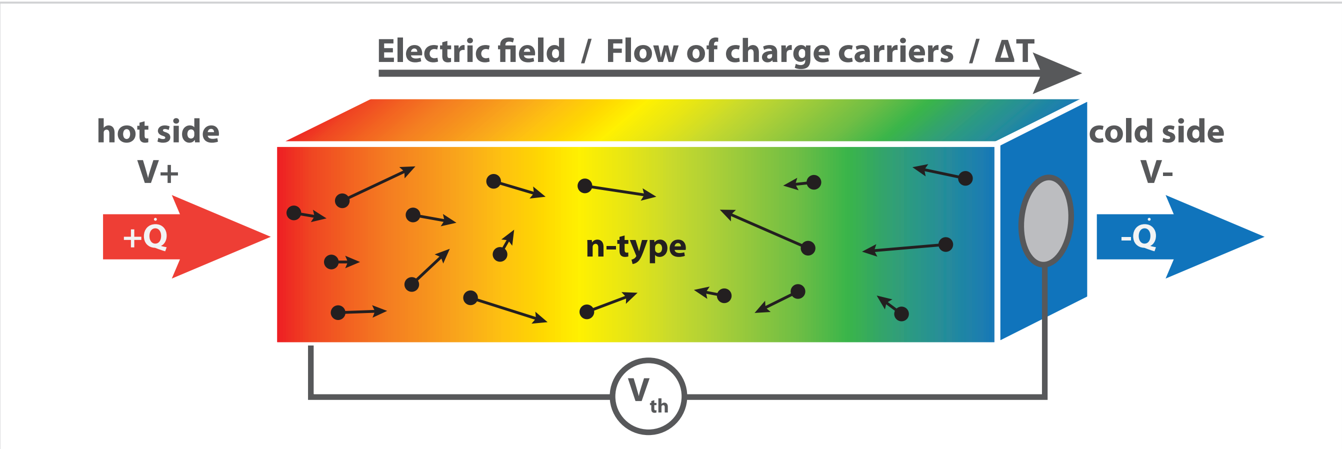
seebeck coefficient; [S] = μV/K

Electrical conductivity; [σ] = 1/Ωm

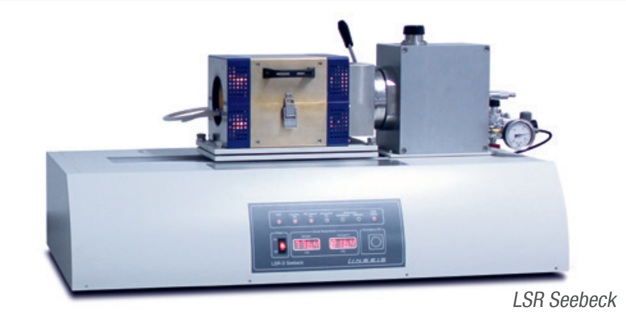
Thermal conductivity; [λ] = W/mK

The figure of merit is an important value for the material science community as well as industry, as it is used for the comparison of the thermoelectric efficiency of materials and modules.
Actually, the highest value of ZT is between 2 to 3. The range of 3 to 4 was considered as a competition to mechanical energy generators.





LSR



LSR Seebeck

Features

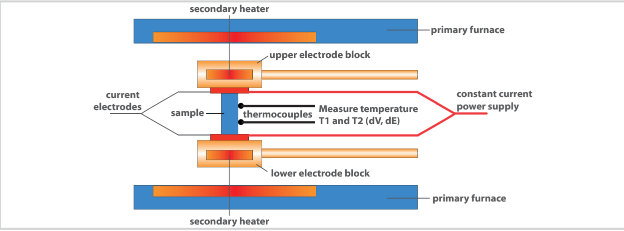
The LSR can simultaneously measure both, Seebeck coefficient and electrical resistance (and optional the thermal conductivity and ZT with the Harman-Method).

- Bar shaped and cylindrical samples with a length between 6 to 23 mm can be analyzed (not for Harman-Method)
- Disc shaped samples with Ø 10, 12.7 or 25.4 mm
- Thin films and foils can be analyzed with a unique measurement adapter
- The design of the sample holder guarantees highest measurement reproducibility (nearly perfect 1-D heat flux through the sample)
- State of the art software enables automatic measurement procedures
- Optional Harman add-on for direct ZT measurement (DC)
- Optional impedance spectroscopy add-on for direct ZT measurement of thermoelectric legs and modules (AC).

Four different exchangeable furnaces cover the temperature range from -100 up to 1500°C. The commonly used infrared furnace enables very fast high heating and cooling rates and the advantage of the most accurate temperature regulation, according to the set temperature profile.

Principles of Measurement

A sample of matching shape is vertically positioned between two electrodes. The lower electrode block contains a heater, while the entire measuring arrangement is located in a primary furnace. The furnace surrounding the measuring arrangement heats the sample to a specified temperature. At this temperature the secondary heater in the electrode block creates a set temperature gradient along the sample. Two contacting thermocouples then measure the temperature gradient $\Delta T = T_{hot} - T_{cold}$ as well as the electromotive force dE at one wire of each of the two thermocouples (thermopower). The DC four-terminal method is used to measure the electric resistance by applying a constant current (I) at both ends of the sample and measuring the corresponding voltage drop between one wire at each of the two thermocouple pairs. A unique thermocouple contact mechanism permits highest measurement accuracy.



Seebeck effect

Electric resistivity

Bulk and thin film samples

Harman method - ZT (300°C)

	LSR
Temperature range	-100 up to 500°C; RT up to 800° / 1100° / 1500°C
Measurement method	Seebeck coefficient: Static DC method / slope method Resistivity: DC four-terminal method ZT-measurement: Harman method (300°C)* ZT of legs and modules: impedance spectroscopy*
Specimen holder	sandwiched between two electrodes / optional thin film adapter
Atmosphere	inert, oxid., red., vac. (typically helium)
Sample size (bar shaped / cylindric)	side lenght/diameter: 2 to 6 mm; height 6 to 23 mm.
Sample size round (disc shape)	10, 12.7, 25.4 mm
Probe distance	4, 6, 8 mm
Cooling water	required

**depends on sample*

LZT-Meter (combined LSR/LFA)

LZT-Meter

Combined LFA and LSR

Thermal Conductivity, Seebeck coeffitient and Electric Resistivity

ZT up to 1100°C

Innovative concept of LZT-Analyzer

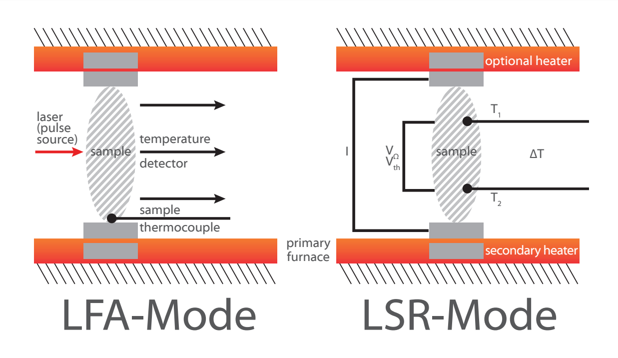
The first commercial instrument worldwide to measure all single parameters for the calculation of the Figure of Merit in only one measurement (combining LSR and LFA). The instrument combines three types of measurement: thermal conductivity, electric resistivity and Seebeck coefficient, what means it can unify the function of a LSR with a LFA.

The analyzer is available with different furnace types. An advanced infrared furnace for most accurate temperature control at very high heating and cooling rates, a low temperature furnace for sub-ambient temperature measurements and a high temperature furnace up to 1500°C. The included software package provides the possibility to evaluate all measured data in the easy-to-handle way the LINSEIS software is known for.

Main advantages of all in one measurement:

Maximum consistence of measurement results due to:

- Same sample
- Same geometry
- Same stoichiometry
- Absolutely identical environmental conditions (humidity, atmosphere)
- Identical temperature profile
- Possible measurement of high ohmic resistance samples

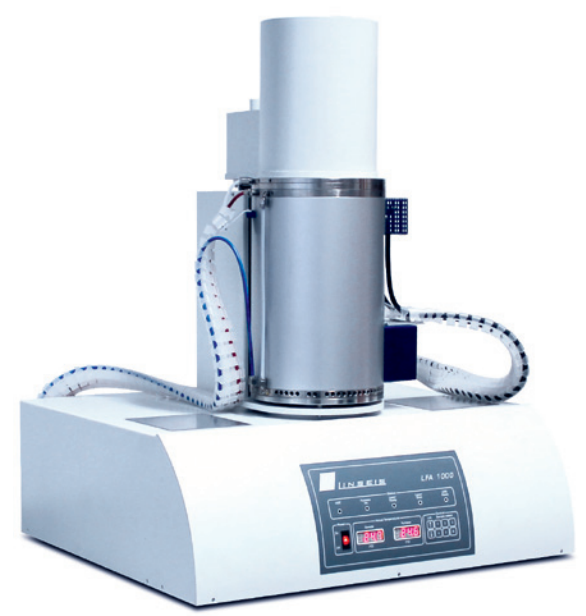


	LZT-Meter
Temperature range	-100°C up to 500°C RT up to 1100°
Specimen holder	sandwiched between two electrodes optional thin film adapter (for LSR part only)
Atmosphere	inert, oxid., red., vac. (typically helium)
Cooling water	required
Seebeck (LSR part)	
Seebeck coefficient	Static DC method / slope method
Electric resistance	four-terminal method
Sample size (LSR only)	2 to 4 mm diameter x 6 to 23 mm long
Thermal conductivity (LFA part)	
Sample size (LFA and LSR)	ø 10, 12.7, 25.4 mm
Lead interval	4, 6, 8 mm
Pulse source	Nd: YAG laser with up to 25 J/pulse
Pulse duration	0.01 up to 5 ms
Detector	InSb
Thermal diffusivity	
Measuring range	0.01 up to 1000 mm²/s

Laser Flash/Light Flash Analyzer – LFA

Thin Film

Bulk



LFA 1000

LINSEIS offers a variety of instruments to measure the thermal diffusivity. The entry level LFA 500 provides a cost effective and powerful solution for the temperature range from -100 up to 1250°C, while the LFA 1000 provides unbeaten sampling rates, up to 6 samples at the same measurement cycle, highest modularity and possibly three different user exchangeable furnaces up to 2800°C.

Absolute Method
Widest temperature range
Advanced software (incl. Dusza-model)
Unique Dusza-model for simultaneous finite pulse-time and heat-loss correction

System Design

LINSEIS is offering an unparalleled modular system design for this thermophysical properties analyzer. It is possible to upgrade the temperature range (exchangeable furnaces / measuring system) and the detector (InSb/MCT). This enables the user to start with a cost effective solution and upgrade the system whenever the budget allows or the measurement task requires it.

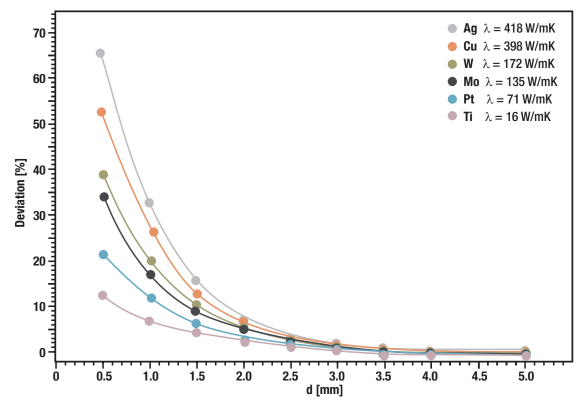
The LINSEIS LFA operates in agreement with national and international standards such as ASTM E-1461, DIN 30905 and DIN EN 821.

Thermal Conductivity λ

Multiple Furnaces/Turntable

Sample Robot

	LFA 500/1000
Sample dimension	Diameter: 6–25.4 mm I Height: 0.1–6.0 mm Square: 6 up to 20 mm
Max. sample number	up to 18 samples
Temperature range	-125 up to 500°C RT up to 500 / 1250 / 1600 / 2800°C
Vacuum	10^{-5} mbar
Atmosphere	inert, oxid., red., vac.
Thermal Diffusivity	0.01 up to 1000 mm²/s
Thermal Conductivity	0.1 up to 2000 W/(m·K)
Pulse source LFA 500 / LFA 1000	Xenon Lamp / Nd: YAG Laser
Pulse enery	15 J/pulse / 25 J/pulse



Thin Film Laser Flash – TF-LFA

TF-LFA

Thermophysical properties from thin-films are becoming more and more important in industries for products such as phase-change optical disk media, thermoelectric materials, light emitting diodes (LEDs), phase change memories, flat panel displays and of course all kinds of semiconductors. In all these cases, a thin film gets deposited on a substrate in order to give a particular function to a device. Since the physical properties of these films 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 Laser Flash Analyzer (TF-LFA) now offers a whole range of new possibilities to analyze thermophysical properties of thin films from 80 nm up to 20 µm thickness.

The perfect choice for smooth coatings and free standing films. Allows a free choice of substrate as well as the characterization of epitactical grown films. Measures cross-plane thermal diffusivity.

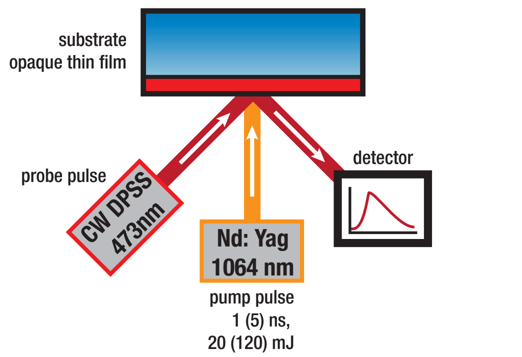
The graph from Schoderböck et. al., Int. J. Thermophys. (2009) illustrates the limitation of the classic Laserflash technique. Samples with a thickness of less than 2mm (depending on the thermal diffusivity of the material) already show a significant deviation from literature values.

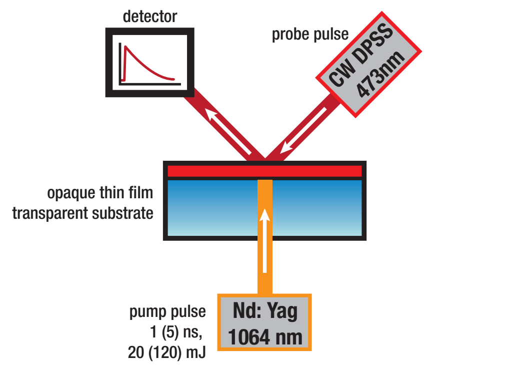
Thermal Conductivity

Thermal Diffusivity

Down to 80 nm layer thickness

-150 up to 500°C

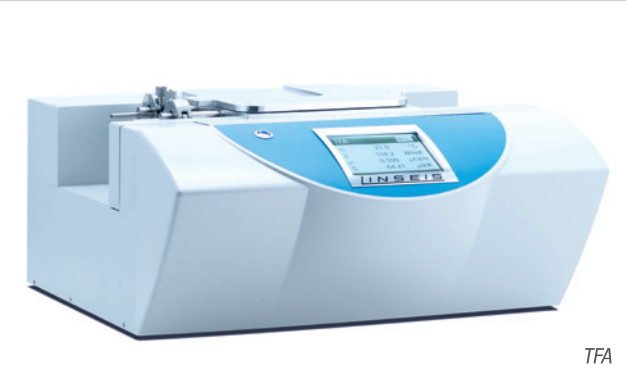




Thin-Film-LFA Specifications	
Sample dimensions	Round with a diameter of 10 mm to 20 mm or square with edges of 10 to 17 mm
Thin film samples	80 nm up to 20 µm*
Temperature range	RT, RT up to 500°C or -100 to 500°C
Heating and cooling rates	0.01 up to 10 K/min
Atmosphere	inert, oxidizing or reducing
Diffusivity measuring range	0,01 mm^2/s up to 1000 mm^2/s

**depends on sample*

Thin Film Analyzer – TFA



TFA

The LINSEIS Thin Film Analyzer 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 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.

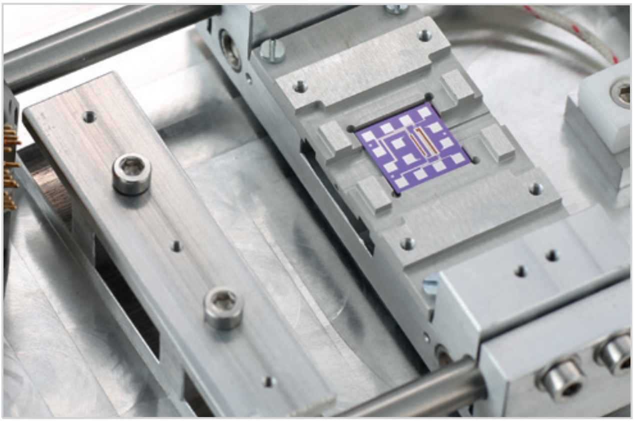
Measurement Setup

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 organic materials. Therefore many different deposition methods like PVD or spin coating and drop casting can be used.

All-in-one Thin Film Characterization

Thermal Conductivity, Seebeck Coefficient, Electrical Conductivity, Hall Constant



	TFA
Temperature range	-170°C up to 280°C
Sample thickness	from few nm to µm range (depends on sample)
Measurement principle	chip based (pre structured measurement chips, 24 pcs. per box)
Deposition techiques	include: PVD (sputtering, evaporation), ALD, CVD, spin coating, ink-jet printing and much more
Measured parameters	thermal conductivity (3 Omega) specific heat
Optional	Electrical resistivity/conductivity Seebeck coefficient hall constant /mobuility / charge carrier concentration Permanent magnet up to 0.5 T or Electromagnet up to 1 T

Hall-Effect

Thin Film

Bulk

HCS-Hall Characterization System

Hall Constant

Mobility

Charge Carrier Concentration

The HCS system permits the characterization of semiconductor devices regarding their electric transport properties, in particular Hall-mobility, charge carrier concentration and resistivity.

The integrated desktop setups offer a complimentary product line-up from a basic, manual operated, Hall characterization stage to an automated high temperature stage up to the innovative Hallbach configuration for the characterization of most challenging samples.

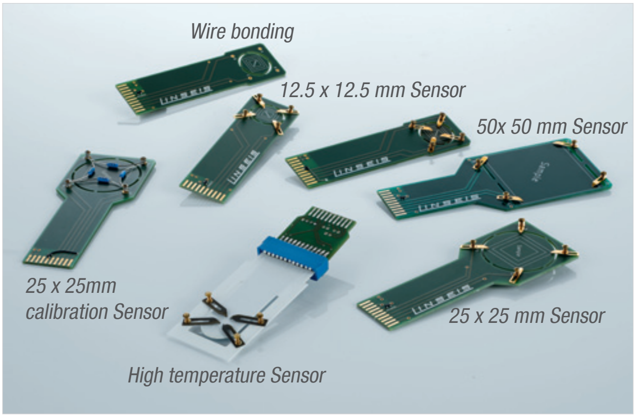
The systems can be equipped with different sample holder for various geometries and temperature requirements. An optional low temperature (LN_2) attachments is available as well as a high temperature version up to 800°C, to ensure that all fields of application can be covered. Depending on the system configuration, either a permanent magnet, a water cooled electromagnet or a Hallbach magnet provide magnetic field strength of up to 1 Tesla.

The comprehensive Windows based software offers an easy to use graphical user interface to control the system parameters, define measurement procedures and temperature profiles as well as allows for an easy data evaluation, presentation and storage

Features

- Carrier concentration
- Resistivity / conductivity
- Mobility
- Alpha (horizontal/vertical ration of resistance)
- Hall constant
- Magneto resistance.

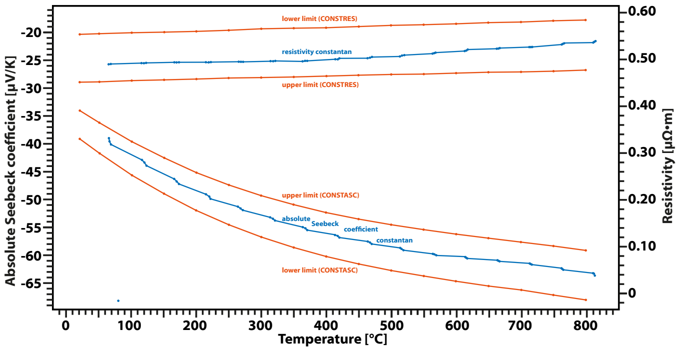
	HCS 1 / 10 / 100
Input current	5 nA up to 125 mA
Hall tension	1 µV up to 2500 µV
Max. resolution	65 pV
Sample geometry	from 5 x 5 mm up to 50 x 50 mm up to 5 mm height
Temperature range	LN2 up to 800°C
Permanent magnet	up to 0.70T
Electro magnet	up to ± 1 T
Hallbach magnet	up to 0.5 T



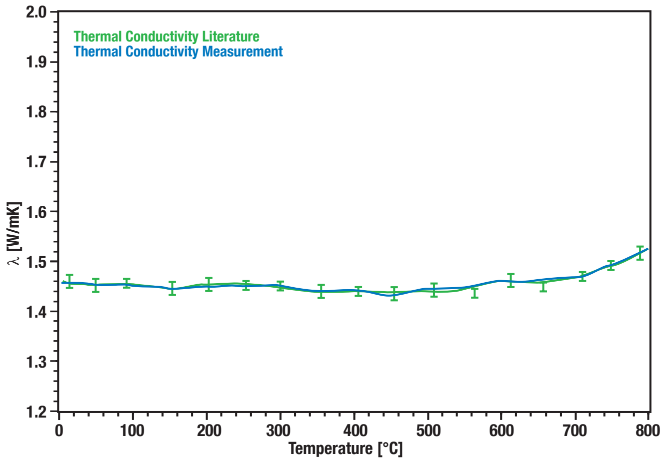
Applications

Measurement of the constantan reference sample

In contrast to the Bi_2Te_3 reference sample provided by NIST (SRM 3451)™, which is only useable in the low temperature range up to 390 K, our constantan reference sample can be used as a high temperature reference sample up to 800°C. The measurement shows a typical result which fits perfectly in the specified tolerances.



LSR LZT

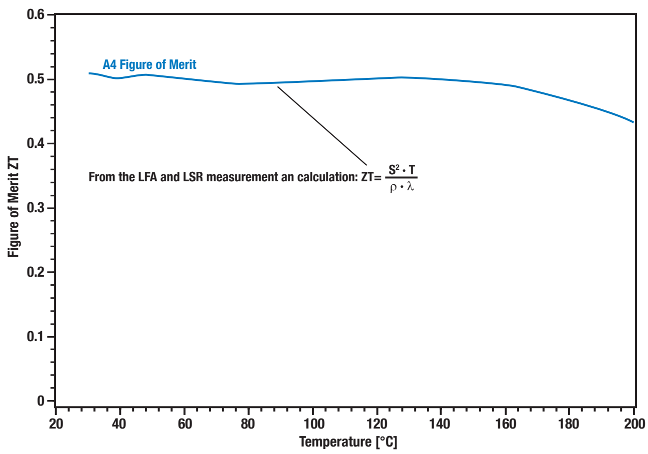


Measurement of thermal conductivity of a ceramic sample using LFA

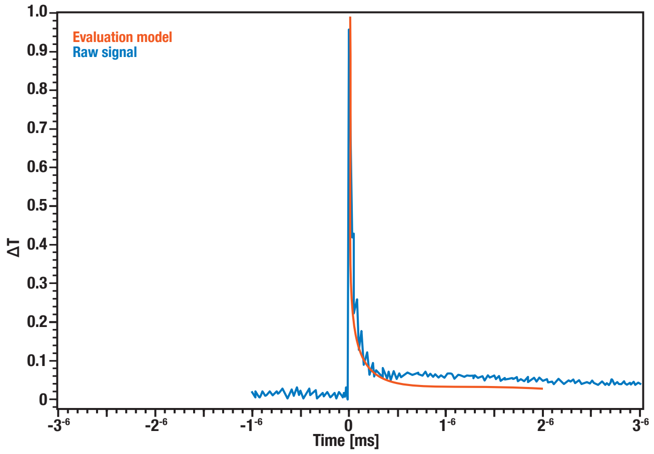
With the LFA, the thermal conductivity of a SiO-containing ceramic sample was measured over temperature. The results show a slightly increasing thermal conductivity over temperature in the range of up to 1.5 W/mK.

LFA LZT

Figure of Merit (calculation) over temperature of $(BiSb)_2Te_3$



LSR LFA LZT

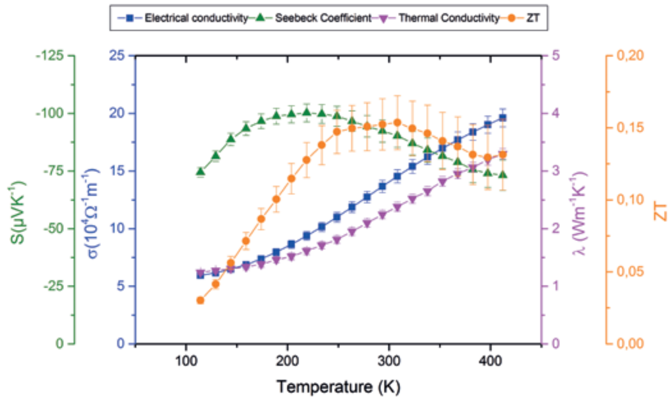


The Thin Film Laser Flash Analyser – TF-LFA was used to measure a 100nm silicon nitride layer, that was covered by a 200nm gold cover-layer and placed on a silicon substrate. The red line shows the evaluation model and its fitting to the detector signal, giving the thermal diffusivity at the corresponding temperature.

TF-LFA

Full ZT characterization of a 142 nm $Bi_{87}Sb_{13}$ thin film

Measured electrical conductivity, thermal conductivity and Seebeck coefficient as well as calculated ZT value of a 142 nm thick $Bi_{87}Sb_{13}$ nanofilm, prepared by thermal evaporation in the temperature range from 120 K up to 400 K.



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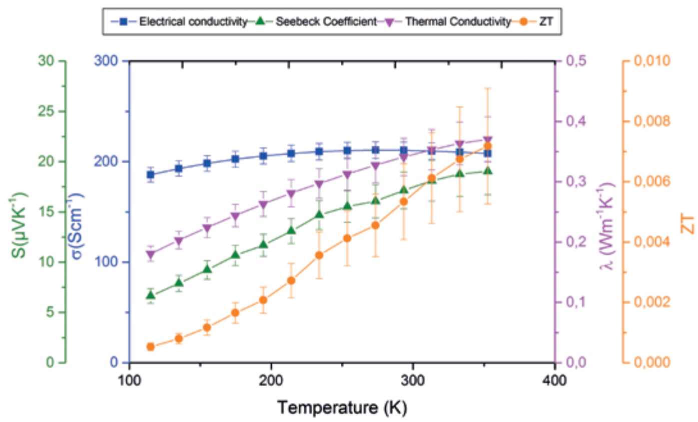

Full ZT Characterization of a PEDOT: PSS layer

Measured electrical conductivity, thermal conductivity and Seebeck coefficient as well as calculated ZT value of a 15 µm thick PEDOT:PSS thin film, prepared by drop casting in the temperature range from 110 K up to 350 K.

TFA

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Services: Service Lab, Calibration Service

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