LINSEIS

THERMAL ANALYSIS

THERMO-ELECTRIC MATERIALS

Material & Modules
Characterization
Instrumentation

Introduction

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

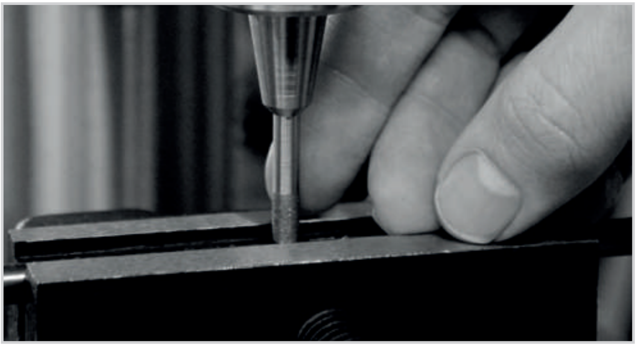
Customer orientation, innovation, flexibility and high quality are what LINSEIS stands for. Thanks to these fundamental characteristics, our company enjoys an exceptional reputation among worldwide leading scientific institutes and industrial companies. LINSEIS has been offering benchmark products in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

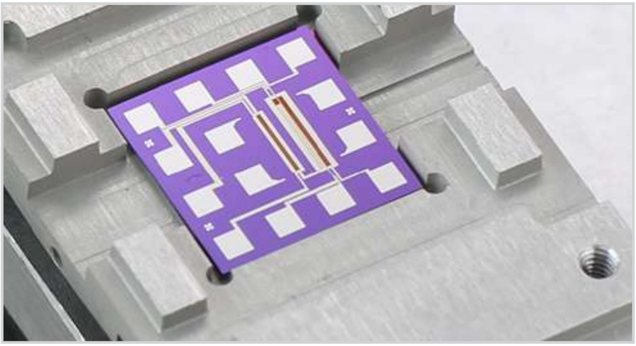
The development of thermo analytical testing machines requires significant research and a high degree of precision. Since many years LINSEIS Corp. invests in this research to the benefit of our customers.

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.

General introduction

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 (Thermoelectric-Effect), the Peltier-Effect and the Thomson-Effect, which describe a reversible interaction between the both values. 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 dimensions figure of merit ZT, which is a combination of these three values and is defined as:

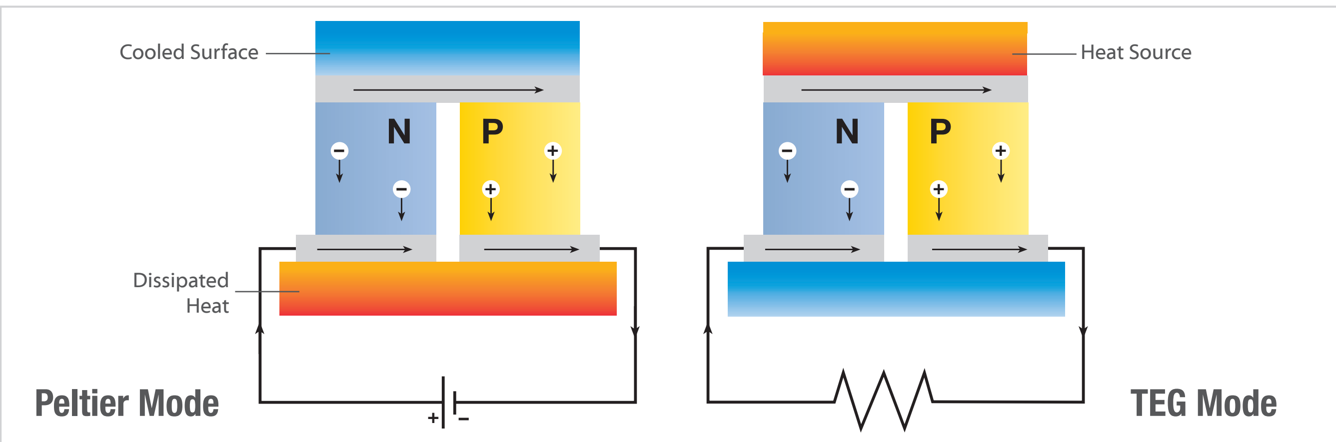
$$ZT = \frac{S^2 \cdot \sigma \cdot T}{\lambda}$$

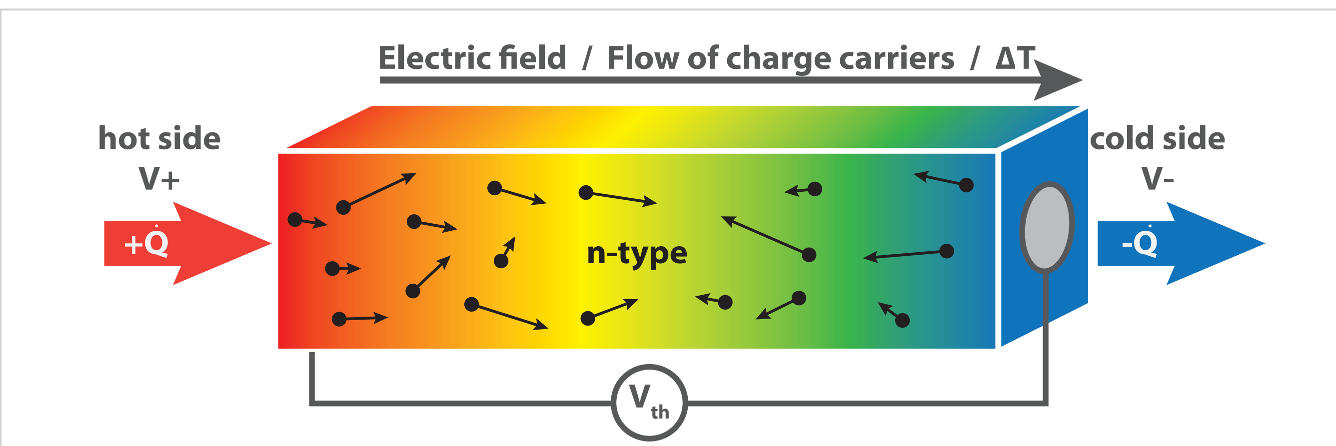
Seebeck Coefficient; [S] = µV/K

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 Z is between 2 to 3. The range of 3 to 4 was considered as a competition to mechanical energy generators.





LSR

LSR Seebeck

Features

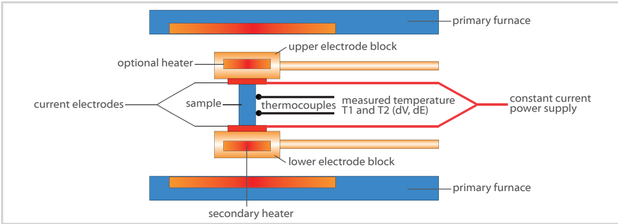
The LSR can simultaneously measure both, Seebeck Coefficient and Electrical Resistance (and optional the Thermal Conductivity and ZT with the Harman-Method).

- Bar shaped and cylindrical samples with a length between 6 to 23mm can be analyzed (Prism samples required 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 addon for direct ZT measurement (DC)
- Optional Impedance Spectroscopy addon 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 furnaces enables very 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 lower electrode block creates a set temperature gradient. Two contacting thermocouples then measure the temperature gradient $\Delta T = T_{hot} - T_{cold}$. A unique thermocouple contact mechanism permits highest temperature accuracy measurements of the electromotive force dE at one wire of each of the two thermocouples. 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 change in voltage (dV) between one wire at each of the two thermocouple pairs.



Seebeck Effect

Electric resistivity

Harman method - ZT (400°C)

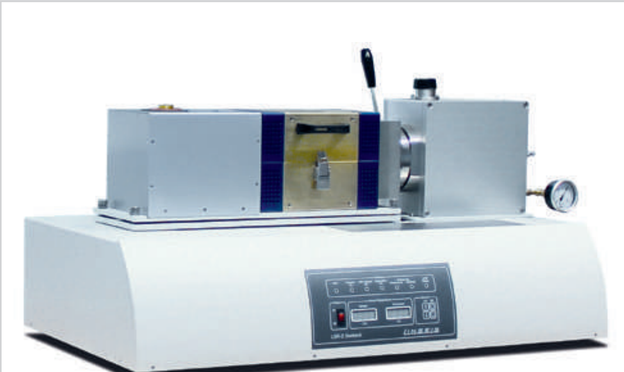
Impedance Spectroscopy

	LSR
Temperature Range	-100 up to 500°C; RT up to 800° / 1100° / 1500°C
Measurement method	Seebeck coefficient: Static DC method / slope method Electric resistance: DC four-terminal method ZT-Measurement: Herman Method (400°C) ZT of legs and modules: Impedance Spectroscopy
Specimen holder	sandwiched between two electrodes / optional thin film adapter
Atmosphere	inert, oxid., red., vac.
Sample size (Bar shaped / Cylindric)	2 to 6 mm width and depth / ø 6 mm x 6 to 23 mm height
Sample size round (Disc shape)	10, 12.7, 25.4 mm
Lead interval	4, 6, 8 mm
Cooling water	required

LZT-Meter (combined LSR/LFA)

Thin Film

Bulk



LZT-Meter

combined LFA and LSR

Thermal Conductivity, Seebeck and Electric Resistivity

ZT up to 1500°C

Innovative concept of LZT-Analyzer

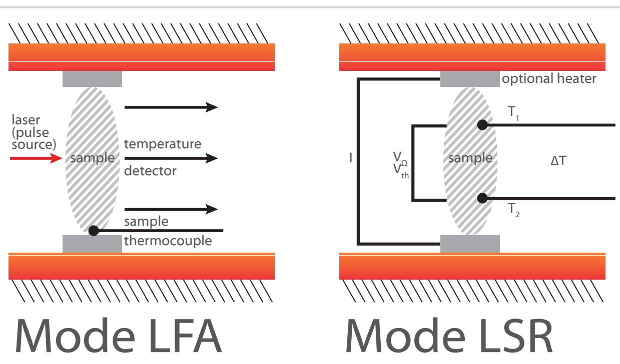
The first commercial instrument worldwide to measure all single parameters for the calculation of the Figure of Merit in only one measurement (combining LSR and LFA). The instrument combines three types of measurement: Thermal Conductivity, Electric Resistivity and Seebeck Coefficient, what means it 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 measurments and a high temperature furnace. 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

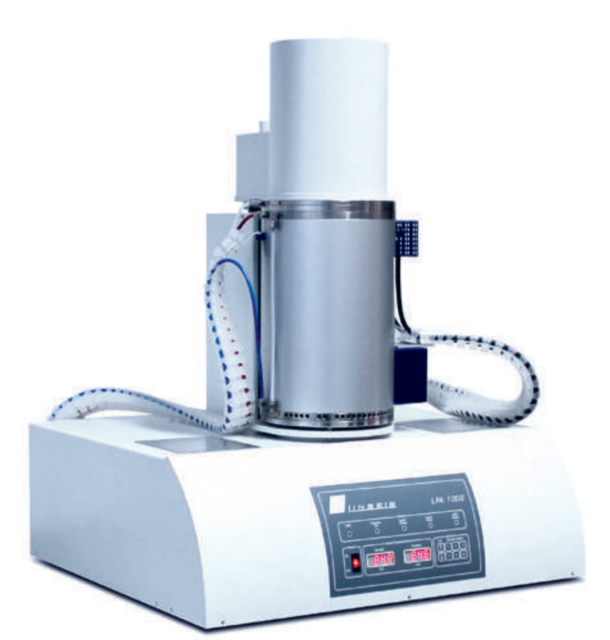


	LZT-Meter
Temperature range	-150 up to 500°C; RT up to 600° / 1100° / 1500°C
Specimen holder	sandwiched between two electrodes
Atmosphere	inert, oxid., red., vac.
Sample size (Disc shape)	10, 12.7, 25.4mm
Lead interval	4, 6, 8mm
Cooling water	required
Seebeck	
Seebeck coefficient	Static DC method / slope method
Electric resistance	four-terminal method
Sample size (LSR only)	2 to 4mm diameter x 6 to 23mm long
Thermal Conductivity	
Pulse source	**Nd: YAG**
Pulse duration	0.01 up to 5ms
Detector	InSb or MCT
Thermal Diffusivity	
Measuring range	0.01 up to 1000mm²/s

Laser Flash/Light Flash – 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 solution for the temperature range -100 up to 1125°C. As a highly modular design, it allows an upgrade to the LFA 1000 system whenever the measurement requires or the budget allows it. The LFA 1000 provides unbeaten sampling rates, up to 6 samples at the same measurement cycle, highest modularity, three different user exchangeable furnaces (-125 up to 2800°C) and two detectors as well as a high vacuum design (10^{-5} mbar).

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.

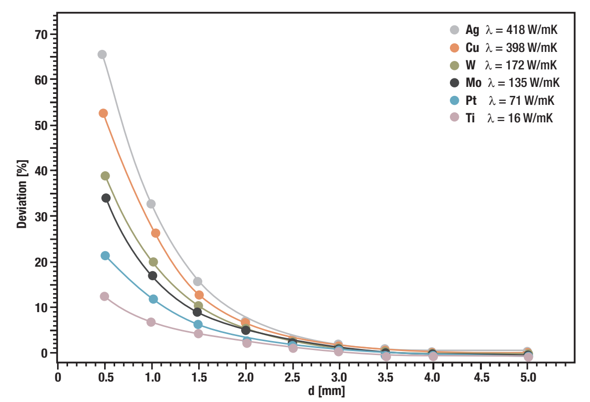
The LINSEIS LFA operates in agreement with national and international standards such as ASTM E-1461, DIN 30905 and DIN EN 821.

Thermal Conductivity λ

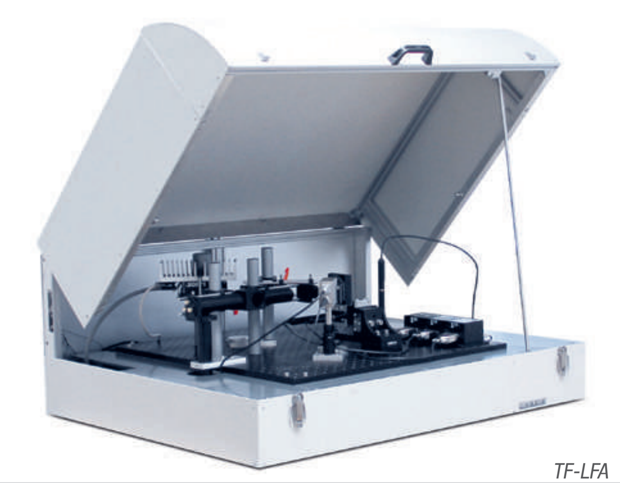
multiple furnaces/turntable

sample robot

	LFA 1000
Sample dimension	Diameter: 6–25.4mm/Height: 0.1–6.0mm
Max. sample number	up to 6 samples
Temperature range	-125 up to 500°C RT up to 1250° / 1600° / 2800°C
Vacuum	10^{-5} mbar
Atmosphere	inert, oxid., red., vac.
Thermal Diffusivity	0.01 up to 1000mm²/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 curse all kinds of semiconductors. In all these cases, a thin film gets deposit on a substrate in order to give a device a particular function. 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 Laserflash for thin films (TF-LFA) now offers a whole range of new possibilities to analyze thermophysical properties of thin films from 80nm 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.

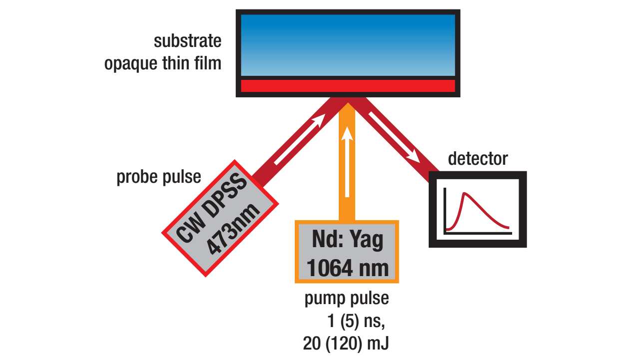
The graph from Schoderböck et. al., Int. J. Thermophys. (2009) illustrates the limitation of the classic Laserflash technique. Samples with a thickness of less than 2mm (depending on the thermal diffusivity of the material) already show a significant deviation from literature values.

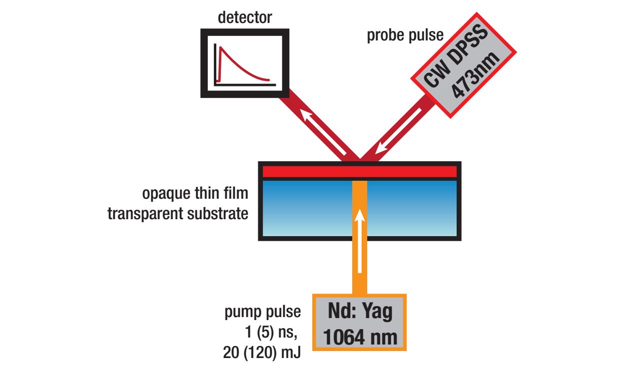
Thermal Conductivity

Thermal Diffusivity

down to 80nm layer thickness

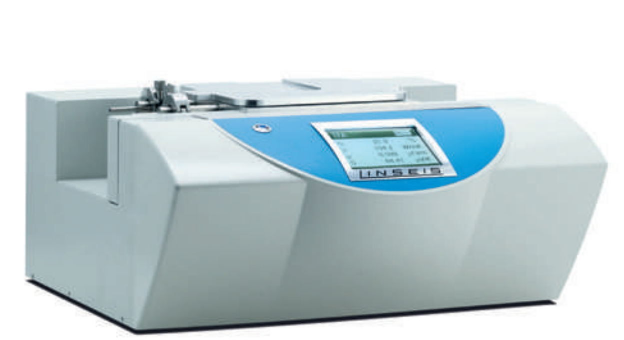
-150 up to 500°C





Thin-Film-LFA Specifications	
Sample dimensions	Round with a diameter of 10mm to 20mm or square with edges of 10 to 17mm
Thin film samples	80nm up to 20µm
Temperature range	RT, RT up to 500°C or -100 to 500°C
Heating and cooling rates	0.01 up to 10°C/min
Atmosphere	inert, oxidizing or reducing
Diffusivity measuring range	0,01mm²/s up to 1000mm²/s

Thin Film Analyzer – TFA



TFA

All-in-one Thin Film Characterization

Thermal Conductivity, Seebeck-Coefficient, Electrical Conductivity, Hall-Coeffiecient

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 Firmware and Software package.

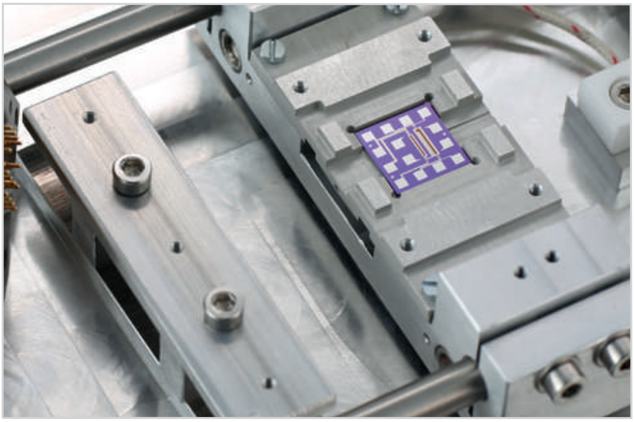
Motivation

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 300°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 semocinducting behaviour as well as metalls, ceramics or organics. Therefore many different deposition methods like PVD or Spin coating and drop casting are possible to use.



	TFA
Temperature range	RT up to 200°C -170°C up to 300°C
Sample thickness	from few nm to µm range (depends on sample)
Measurement principle	chip based (pre structured measurement chips, 24 pcs. per box)
Desposition techiques	include: PVD (sputtering, evaporation), ALD, spin coating, ink-jet printing and more
Measured parameters	Thermal Conductivity (3 Omega) Specific Heat
Optional	Electrical Resisitivity / Conductivity Seebeck Coefficient Hall Constant / Mobility / Charge carrier conc. Permanent magnet up to 0.5 T or Electromagnet up to 1 T

Hall-Effect

Thin Film

Bulk

L79/HCS-Hall Characterization System

Hall constant

Mobility

Charge Carrier Concentration

The L79/HCS System permits the characterization of semiconductor devices, regarding their Hall mobility, charge carrier concentration and resistivity.
The rugged desktop setup offers different sample holders for various geometries and temperature requirements. An optional low temperature (LN2) attachments and a high temperature version up to 800°C ensure that all fields of application can be covered. Different permanent and electric magnets provide fixed or variable magnetic fields up to several tessla. The comprehensive Windows based software provides I-V and I-R Plot.
The system can be used to characterize various materials including Si, SiGe, SiC, GaAs, InGaAs, InP, GaN (N Type & P Type can be measured), metal layers, oxides, etc.. Sample testing can be performed to demonstrate the system´s capability.

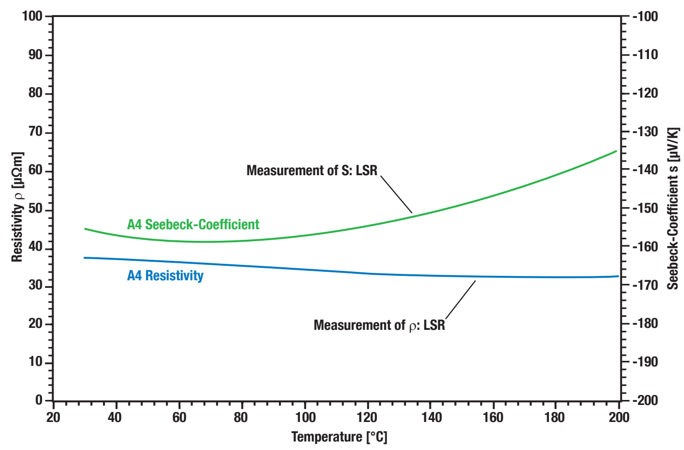
Features

- Carrier Concentration
- Resisitivity
- Mobility
- Conductivity
- Alpha (horizontal/vertical ration of resistance)
- Hall Coefficient
- Megneto resistance

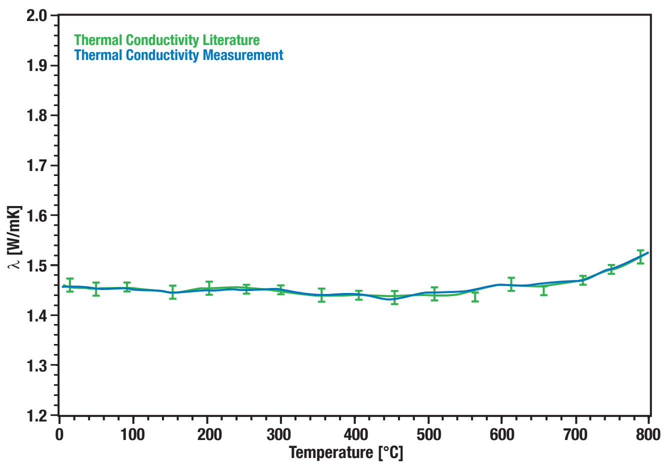
	L79/HCS-Hall
Temperature range	From LN_2 up to 800°C in different configurations
Input current	500nA up to 50mA
Hall tension	1µV up to 2500V
Max. resolution	65pV
Sample geometry	15 x 15, 20 x 20, 25 x 25mm, up to 5mm height
Magnetic field	Permanent magnet 0.75 T Pole diameter 90 mm Two magnet setup for bipolar measurement. Electromagnet up to 1.2 T. Pole diameter up to 76 mm. Power supply 75A / 40V. Current reversal swith for bipolar measurement.
Sensors	different exchangable sensor configurations available
Resistivity Range	10^{-4} up to 10^{7} (Ω/cm)
Mobility range	1 up to 10^{7} (cm²/Volt sec)
Atmospheres	Vaccum, inert, oxidizing, reducing

Applications

Seebeck-Coefficient and Resistivity from the LSR-Measurement above temperature of $(BiSb)_2Te_3$



LSR

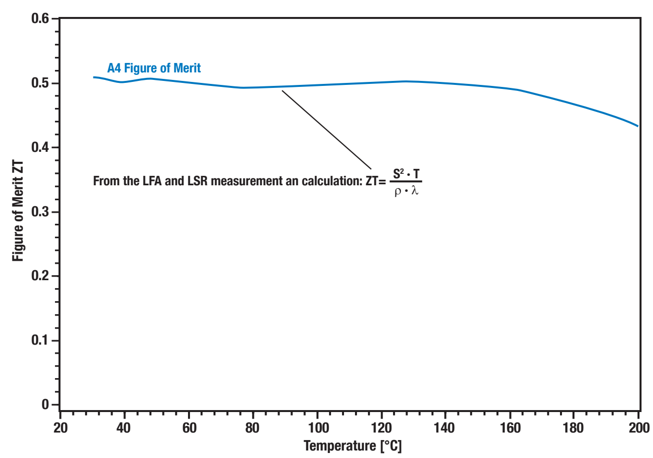


Measurement of thermal conductivity of a ceramic sample using LZT Meter

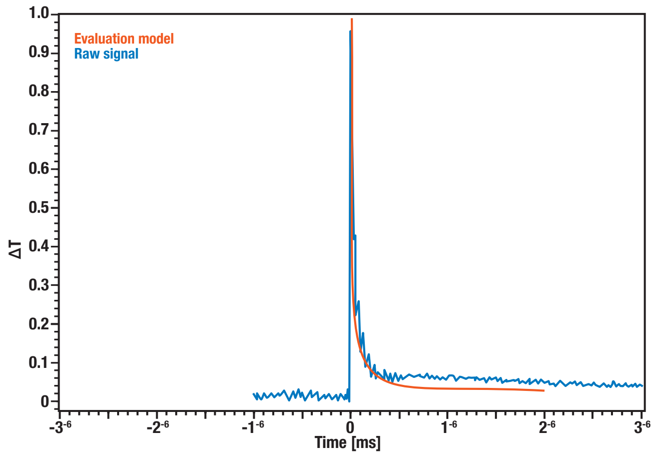
With the LZT, the thermal conductivity of a SiO-containing ceramic sample that showed a light increasing thermal conductivity over temperature up to 1,5 W/mK, was measured over temperature. Next to the standard LFA, the LZT is able to determine electrical properties such as resistivity and Seebeck coefficient but also Thermal Diffusivity and Conductivity by using the integrated pulse laser.

LFA

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



LSR LFA

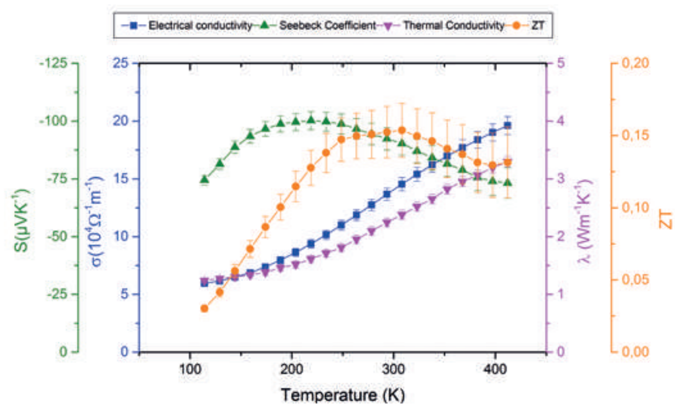


The Thin Film Laser Flash – TF-LFA was used to measure a 100nm Silicon Nitride layer, that was covered by a 200nm gold cover-layer and placed on a silicon substrate. The red line shows the evaluation model and its fitting on the laser pulse, giving the thermal diffusivity at the corresponding temperature.

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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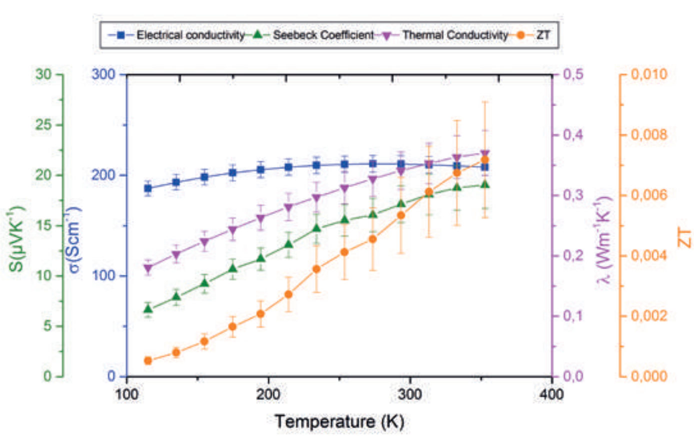

Full ZT Characterization of a PEDOT: PSS layer

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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