

THERMAL ANALYSIS

THIN FILMTFAANALYZER



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.

We are driven by innovation and customer satisfaction.



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.

THIN FILM ANALYZER

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 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 metalls, ceramics or organics. Therefore many different deposition methods like PVD or Spin coating and drop casting are possible to use.



MEASURING SETUP

Components

The basic setup consists of a measurement chip on which the sample can be easily deposited, and the measurement chamber to provide the required environmental conditions. Depending on the application, the setup can be utilized with a Lock-In amplifier and / or a strong electric magnet. The measurements are usually taken under UHV and the samples temperature can be controlled between -170°C and 280°C during the measurement using LN₂ and powerful heaters.

Modular design

Starting with a basic setup to measure the thermal conductivity, the system can easily be upgraded with either the thermoelectric kit to measure the electrical conductivity and Seebeck coefficient or with the magnetic upgrade kit to take Hall constant, mobility and charge carrier concentration measurements.



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
- cp 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:

- $\bullet\,\sigma$ 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 as well as 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.



MEASURING PRINCIPLES

Pre structured measuring chips

The chip is combining the 3 Omega 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 Van-der-Pauw electrodes. For an easy sample preparation either a strip off foil mask or a metal 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.

1. Van-der-Pauw measurement

To determine the electrical conductivity (s) and Hall coefficient (AH) of the sample, the Vander-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 $\left(-\frac{\pi d}{\rho} \cdot \mathbf{R}_{AB,CD}\right) + \exp\left(-\frac{\pi d}{\rho} \cdot \mathbf{R}_{BC,DA}\right)$

By applying a magnetic field and measuring the corresponding change of the diagonal Van-der-Pauw resistance, the Hall coefficient 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=-Vth/ Δ T.

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

2. Hot stripe measurement

For the determination of the in-plane thermal conductivity, a patend pending hot-stripe suspended membrane setup is used. In this setup, a very small wire is used as heater and temperature sensor in one. The sample of interrest will be deposited directly on this membrane.

For the measurement in consequence, a current is applied to the hotwire which is heated up due to Joule heating. Because of the temperature rise, the resistivity of the wire is changing and can be measured easily. From this resistivity change and the knowledge of the exact geometrie of the setup, it is possible to calculate back to the thermal conductivity of the sample.

Depending on the sample, it is also possible to measure the emissivity and specific heat.

In order to get high quality results, the sample thickness times sample thermal conductivity should be equal or bigger than 2×10^{-7} W/K.





Options



TFA with moveable electromagnet (up to 1 Tesla)



VdP Hall coeffitient measurement



TFA with permanent magnet (up to 0.5 Tesla)

TECHNICAL SPECIFICATIONS

General	
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, 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	Seebeck Coefficient Electrical Resisitivity / Conductivity Hall Constant / Mobility / Charge carrier conc. permanent magnet 0.5 T or electromagnet up to 1 T
Vacuum	up to 10 ⁻⁵ bar
Electronics	integrated
Interface	USB
Measurement range	
Thermal Conductivity	0.05 up to 200 W/m·K
Electrical Resistivity	0.05 up to 1 · 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 Coefficient	±9% for most materials

APPLICATIONS

Thermoelectric devices

A typical application is the characterization of thin films made out of thermoelectric materials. A lot of research is done to improve the efficiency of thermoelectric devices.

Integrated devices

For the development and design of new and robust integrated devices like sensors or microchips, the semiconducting industry needs to know a lot about the physical properties of the used films. For example for the heat management or the electric insolation of such devices.

Thermal barrier coatings

Another interesting field for thin films are thermal barrier coatings as they are used in aircraft engines. Many new materials have been developed and characterized, especially in regard to their thermal behavior.

Tribological stress

The last application example is the determination of material parameters for thin films used in tools. The understanding is very important to avoid wear due to tribological stress to ensure an extended product life cycle.









APPLICATIONS

Full ZT Characterization of a 142 nm Bi₈₇Sb₁₃ thin film



Measured electrical conductivity, thermal conductivity and Seebeck coefficient as well as calculated ZT value of a 142 nm thick Bi₈₇Sb₁₃ nanofilm, prepared by thermal evaporation in the temperature range from 120 K up to 400 K.



Thermal annealing effects observed at a Bi₈₇Sb₁₃ thin film



Thermal annealing effects observed at a 282 nm thin $Bi_{87}Sb_{13}$ nanofilm prepared by thermal evaporation. After the first measurement run at elevated temperatures, the thermoelectric properties show strong thermal annealing effects caused by recristallisation and defect healing processes.



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 μ m thick PEDOT:PSS thin film, prepared by drop casting in the temperature range from 110 K up to 350 K.



Full ZT Characterization of a Gold nanofilm



Measured electrical conductivity, thermal conductivity and Seebeck coefficient as well as calculated ZT value of a 100 nm Au thin film, prepared by DC magnetron sputtering in the temperature range from 225 K up to 375 K. The results preflectly agree with the Wiedemann -Franz law and a clear influence of classical size effects has been observed.

LINSEIS GmbH Germany Vielitzerstr. 43 95100 Selb

Tel.: (+49) 9287 880 0

E-mail: info@linseis.de



LINSEIS Inc. USA 109 North Gold Drive

Robbinsville, NJ 08691 Tel.: (+1) 609 223 2070 E-mail: info@linseis.de



LINSEIS China

Kaige Scientific Park 2653 Hunan Road 201315 Shanghai Tel.: (+86) 21 5055 0642 Tel.: (+86) 10 6223 7812 E-mail: info@linseis.de



LINSEIS France Bureaux Paris 52 Boulevard Sébastopol 75003 Paris Tel.: (+33) 1 73.02.82.72 E-mail: info@linseis.de

Bureaux Lyon 2A Chemin des Eglantines 69580 Sathonay Village Tel.: (+33) 4.26.68.70.55 E-mail: info@linseis.de

LINSEIS Poland

ul. Dabrowskiego 1 05-800 Pruszków Tel.: (+48) 692 773 795 E-mail: info@linseis.de

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