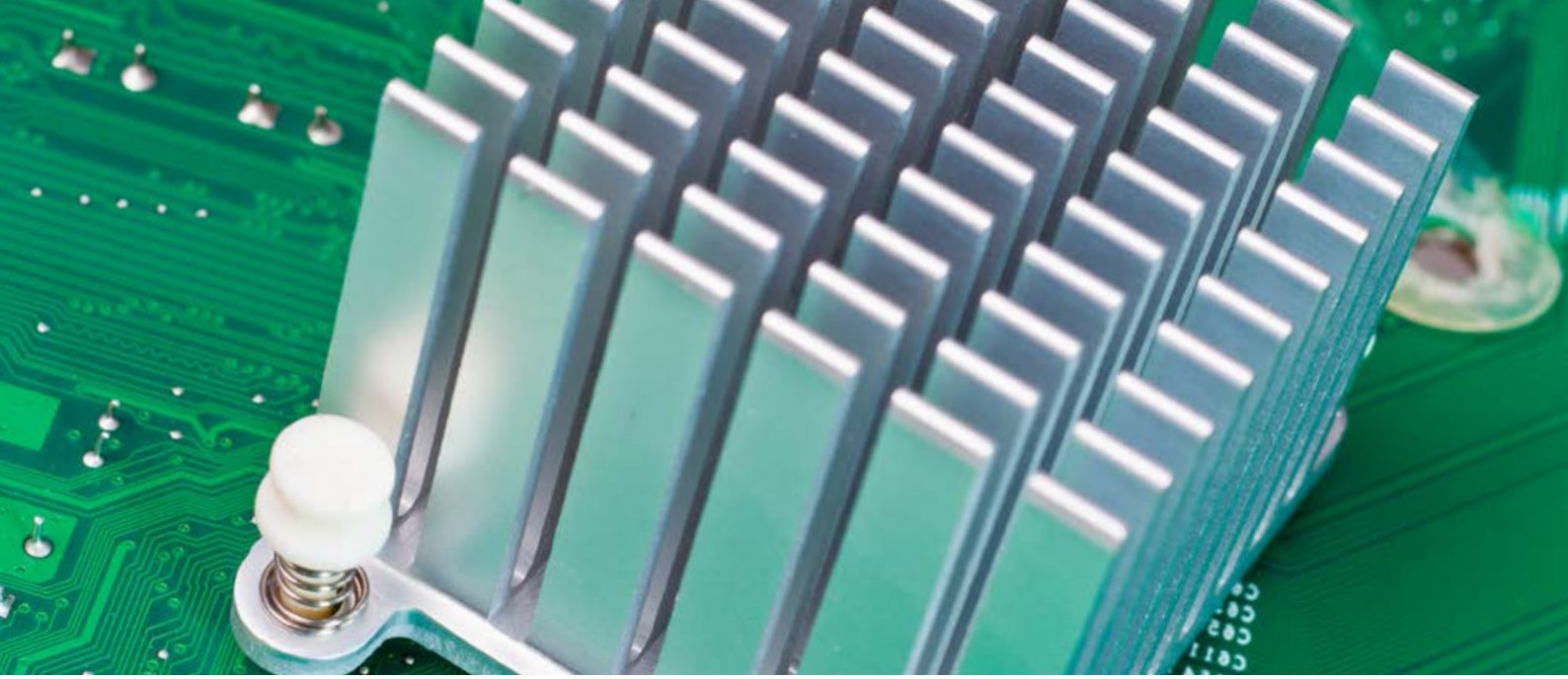
NETZSCH

Proven Excellence.

Simultaneous Determination of the Seebeck Coefficient and Electrical Conductivity – SBA 458 *Nemesis*®

Method and Technique for the Characterization of Thermoelectric Materials

Analyzing & Testing



NETZSCH

Your Partner In Testing Thermoelectrics

Thermoelectric materials with high working temperatures and optimized efficiency are under development for the generation of electrical energy from heat which has been released to the environment. Thereby, precise knowledge of the thermal properties is of paramount importance in order to develop beneficial thermoelectrics with high electrical conductivities, large Seebeck coefficients, and low thermal conductivities.

The relative performance or efficiency of a thermoelectric material is described by the figure of merit (*ZT*):

$$ZT = \left(\frac{S^2\,\sigma}{\lambda}\right) T$$

S = Seebeck coefficient or thermo power of the material [µV/K]
σ = electrical conductivity of the material [S/cm]
λ = total thermal conductivity of the material [W/(m·K)], where $\lambda = \rho \cdot c_p \cdot a$
T = absolute temperature.

ZT already highlights the importance of the Seebeck coefficient with respect to the performance of a thermoelectric material. For this reason, NETZSCH developed the SBA 458 *Nemesis*® which allows for the simultaneous measurement of the Seebeck coefficient (S) and electrical conductivity (σ) under identical conditions.

Using the SBA 458 *Nemesis*® along with our Laser/Light Flash Analyzer, Differential Scanning Calorimeter and Dilatometer systems, it is now possible to determine a complete set of thermophysical properties directly related to the efficiency of thermoelectric materials, including:

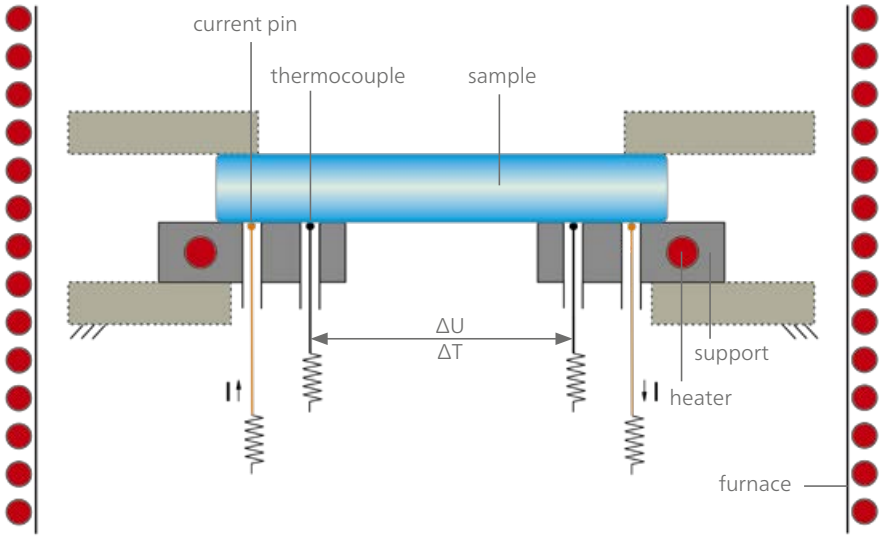
- Seebeck coefficient (S)
- Electrical conductivity (σ)
- Thermal conductivity (λ)/thermal diffusivity (a)
- Density (ρ)
- Specific heat capacity (c_p)

SBA 458 *Nemesis*®

A NEW METHOD WITH INNOVATIVE DESIGN

The SBA 458 *Nemesis*® is a truly new method for determination of the Seebeck coefficient (S) and electrical conductivity (σ). Its novel setup and clever, practical design offer numerous unparalleled advantages.

Perhaps the most significant of these is elimination of the disadvantages inherent in conventional methods – thus allowing for the reliable and precise determination of the two thermophysical properties S and σ.



Measurement setup of the SBA 458 *Nemesis*® for the temperature range between RT and 800°C

A Clever New Setup

Using current pins and thermocouples on the sample's lower surface, the electrical conductivity is determined by the 4-point method. Micro heaters are placed below the two sample edges, creating temperature gradients in both sample directions. The resulting voltage is measured by the thermocouple wires and then used for calculation of the Seebeck coefficient.

SBA 458 *Nemesis*®

A Clever Setup

Yields Advantages

Plug-and-Measure

The new plug-and-measure feature offers convenient sample change. The operator may now use various sample geometries in different dimensions. Since distance determination between the thermocouples is no longer required, a quick start to the measurement can be realized.

Robust, Flexible and Reliable

In every respect, the robust design of the SBA 458 *Nemesis*® allows for unfettered, easy handling. The thermocouples are exceptionally well protected against damage by virtue of Inconel® sheathing, and are additionally mechanically fixated in order to yield high precision and reproducibility of the measurement results.
Nevertheless, thermocouples and current pins can still be easily replaced by the operator.

Integrated Quality Check

The system comes with two micro heaters which operate in alternation. The cyclic heating process is used to perform a quality check for the Seebeck coefficient. A quality check for measurement of electrical conductivity can also be carried out. The user obtains information about the measurement's performance already while it is still in progress.

DETERMINATION OF ZT
NEW METHOD
VARIOUS SAMPLE GEOMETRIES
PLUG-AND-MEASURE
QUICK MEASUREMENT START
CONVENIENT SAMPLE CHANGE
NO DISTANCE DETERMINATION

ROBUSTNESS
FLEXIBLITY
RELIABILITY

INCONEL-SHEATHED THERMOCOUPLES

FIXED THERMOCOUPLES

QUALITY CHECK SEEBECK COEFFICIENT

REPLACEABLE THERMOCOUPLES

CONTACT CHECK

QUALITY CHECK ELECTRICAL CONDUCTIVITY

REPLACEABLE CURRENT PINS
DETECTION OF INHOMOGENEITY
CHECK OF OHMIC CONTACT
DETECTION OF BARRIER LAYER

- Need to measure on a variety of sample geometries – including thin films?
- Tired of complicated sample positioning?
- Prefer to start your measurement quickly?
- Want to eliminate a common source of error by measuring the distance between the thermocouples?

The SBA 458 *Nemesis*®

The Solution!

The One and Only for Different Sample Geometries

Easy and Fast Sample Change

No Tools

No Distance Determination

No Distance Determination Required

Distance determination between fixed thermocouple positions is a cumbersome process which is usually necessary, but this instrument eliminates the need for any operator handling in this regard. By excluding this potential source of error from the determination process for electrical conductivity, the precision and reproducibility of the results are increased.

Measurement Setup for Varied Sample Geometries

The hallmark of the SBA 458 *Nemesis*® is its one-of-a-kind measurement setup which allows more sample geometries than usual for this technique. Depending on the sensor, measurements can be carried out between room temperature and 800°C or room temperature and 1100°C.

Convenient Sample Change – Quick Measurement Start

With the SBA 458 *Nemesis*®, it is no longer necessary to use tools to insert and remove the sample. This makes changing the sample fast and easy while preventing any sample damage which might have resulted from sticking to the electrodes.

PLUG-AND-MEASURE… THAT'S IT!

ROBUST SYSTEM

The SBA 458 *Nemesis*® Has Got It All

- **Prevents**
  contamination of the measuring junction
- **Reduced risk**
  of thermocouple damage
- **No moving**
  of thermocouples
- **Constant contact force**
- **Simple change**
  of thermocouples and current pins

Mechanical Fixation of Thermocouple Positions

The thermocouple positions are fixated, thus eliminating any requirement for distance determination between them. By excluding this potential source of error from the determination process for electrical conductivity, the precision and reproducibility of the results increase considerably.

Inconel-Sheathed Thermocouples

Temperature (voltage) measurements are carried out via two sheathed thermocouples. The Inconel® sheathing prevents chemical contamination of the measuring junction and also prevents any adhesion between the thermocouple and the sample. This minimizes the risk of thermocouples becoming damaged or breaking during sample change.

Constant Contact Force

The constant contact force up to elevated temperatures resulting from mass and cold spring leads to excellent thermal coupling between the sample and thermocouples. This also allows for correct reading of the temperature and thus for accurate and precise determination of the Seebeck coefficient.

Unrestricted Easy Handling

Thermocouples and current pins can be replaced effortlessly by the operator.

Sturdy & Flexible

The unique design of the SBA 458 *Nemesis*® allows for a quality check of both the Seebeck coefficient and electrical conductivity. Even before a time-consuming temperature program is started, this quality check indicates whether a measurement can be performed without any errors. In addition, the quality check is performed at each temperature step, monitoring the correctness of the measurement results for the Seebeck coefficient and the electrical conductivity.

Quality Check Offers

- Contact Check
- Detection of Inhomogeneity
- Detection of Barrier Layer
- Optimized Testing Time

INTEGRATED QUALITY CHECK

The Two Readings of the Quality Check

Seebeck Coefficient

A temperature gradient is generated in both sample directions by two micro heaters operating in alternation. While the cyclic heating is in progress, the resulting voltages (U_A, U_B) are measured. U_A and U_B are the voltages between the two positive and two negative thermocouple wires which are plotted against the temperature difference (ΔT; for both directions). ΔT is determined by two thermocouples (plot A).

The resulting large number of measuring points is the basis for calculation of a regression line. From the slope of the regression line, the Seebeck coefficient can be precisely determined:

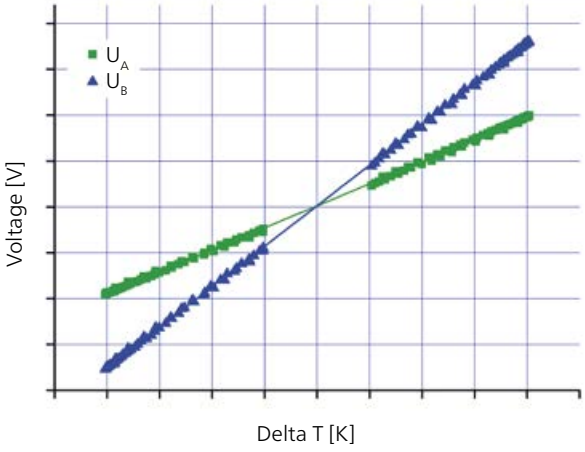
- All measurement points lie on a straight line → well-performed measurement (see plot A)
- Deviation of the measurement points from the straight line, e.g., hysteresis → poor measurement

Electrical Conductivity

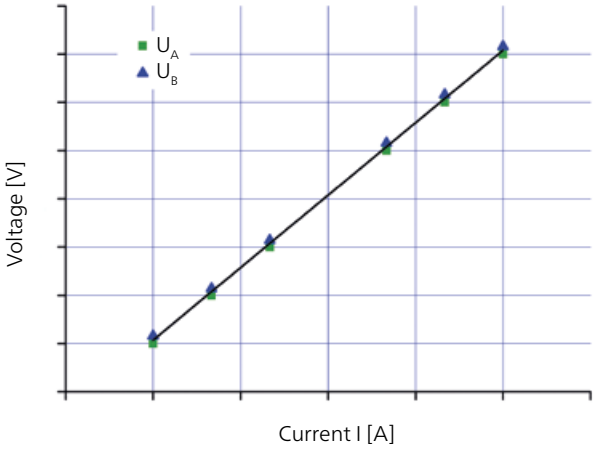
At each testing temperature, different current values will be applied to the sample several times by current pins. These values will be applied in both sample directions and the resulting voltages (U_A, U_B) will be measured (plot B).

This process yields a large number of measurement points, allowing for a quality check prior to and during the measurement of all temperature steps:

- All measurement points lie on a straight line and the two curves are superimposed → well-performed measurement (see plot B)
- Deviation of the measurement points from the straight line or the two curves are not congruent with each other → poor measurement



Plot A: Quality check of the Seebeck coefficient is confirmed by the linear behavior of the voltages.



Plot B: Quality check of the electrical conductivity is confirmed by the linear behavior of the measurement points.

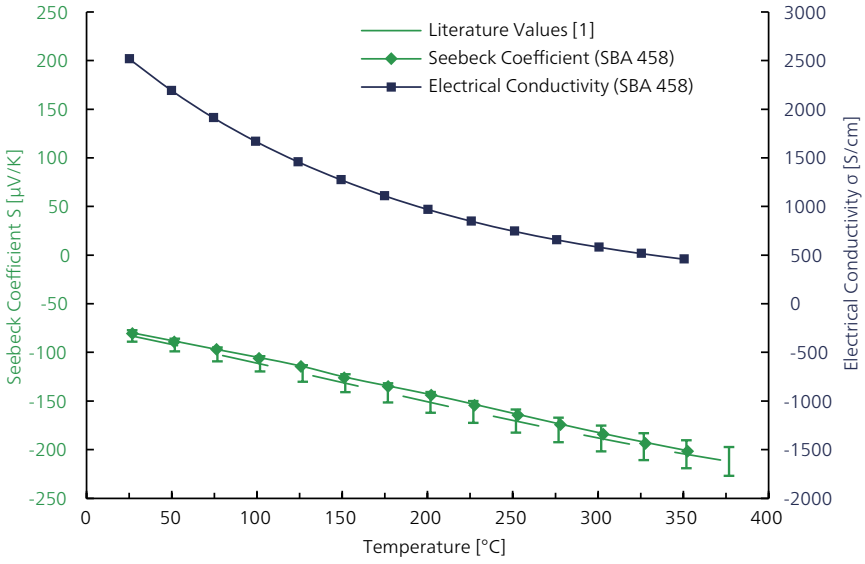
Comparison with Literature Values

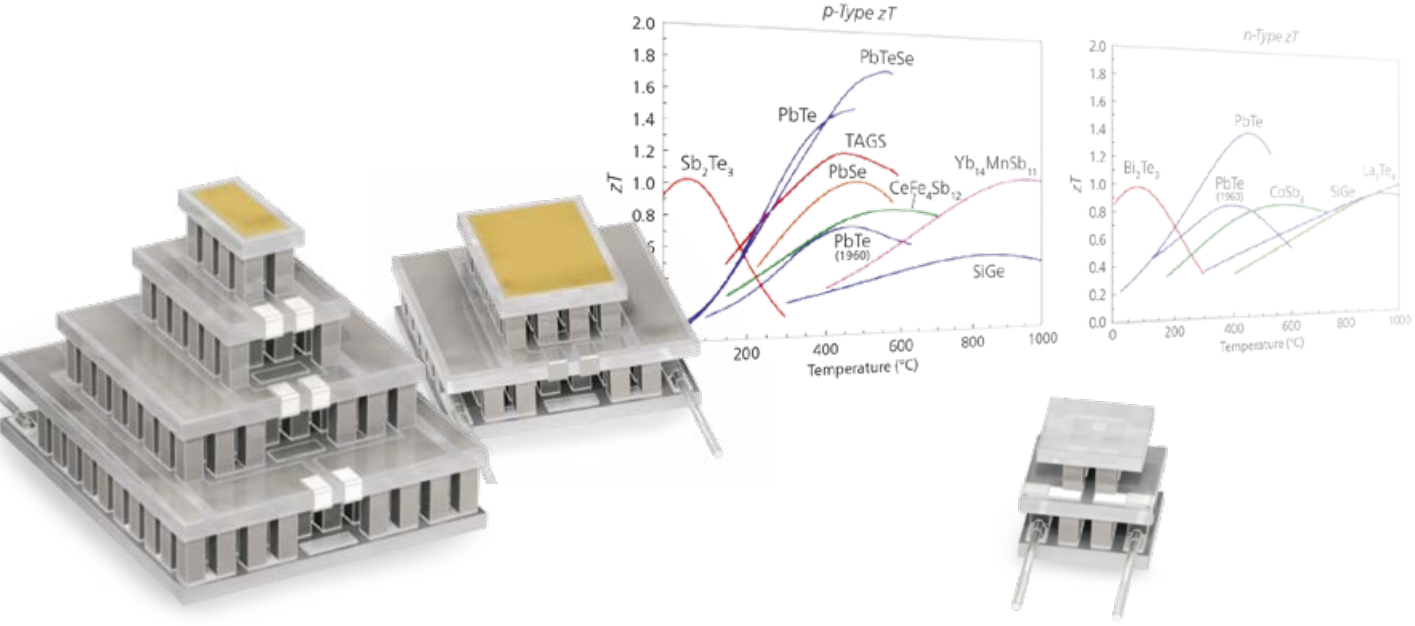
Bi-Doped PbTe

Lead telluride exhibits good performance as a thermoelectric material, due in part to its low thermal conductivity and in part to its electrical properties.

This plots portrays the Seebeck coefficient (green curve) and electrical conductivity (blue curve) between room temperature and 250°C. The dotted green line represents the literature values[1]. Good correlation can be observed, indicating the reliability of the system.

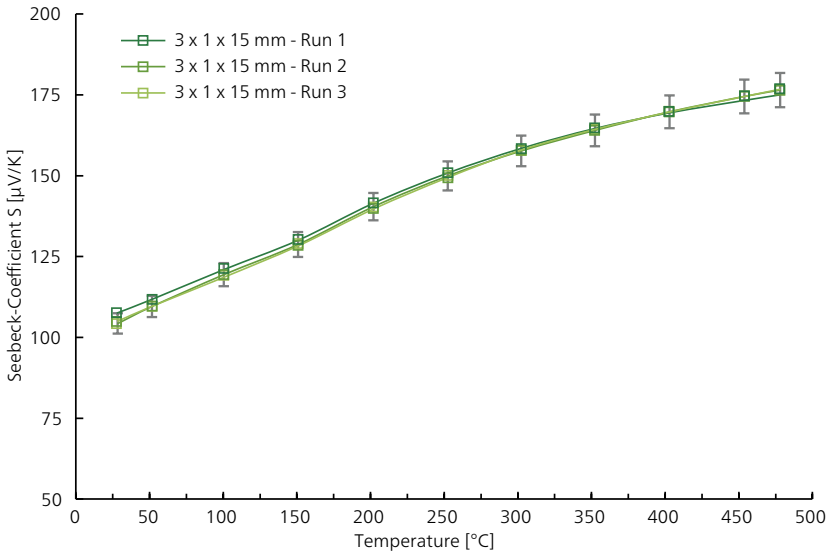
[1] Physikalisch-Technische Bundesanstalt Braunschweig und Berlin (The National Metrology Institute of Germany)

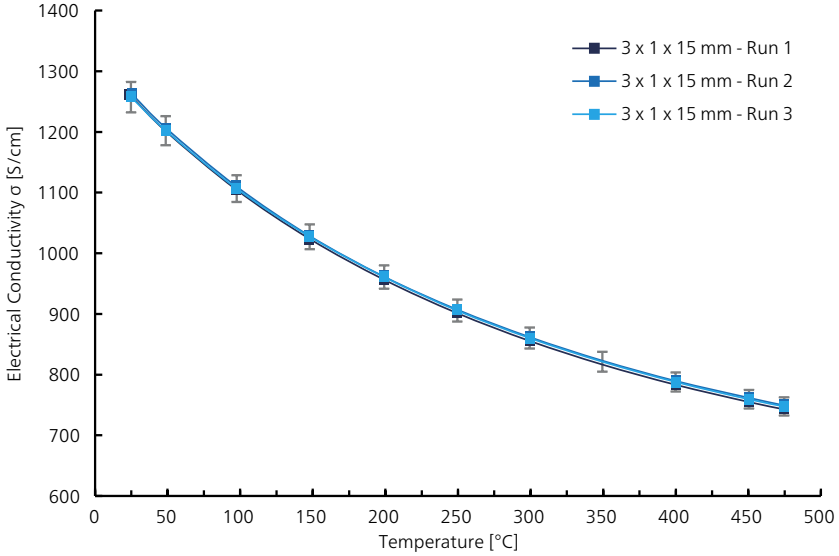




Half-Heusler

Recently, Heusler and Half-Heusler compounds gained significant attention due to their wide range of extraordinary functionalities. They are attractive as half-metallic high-temperature ferri- and ferromagnets, multiferroic shape-memory alloys, etc., and very promising for usage in thermoelectric generators (TEGs) for energy harvesting from any heat source.

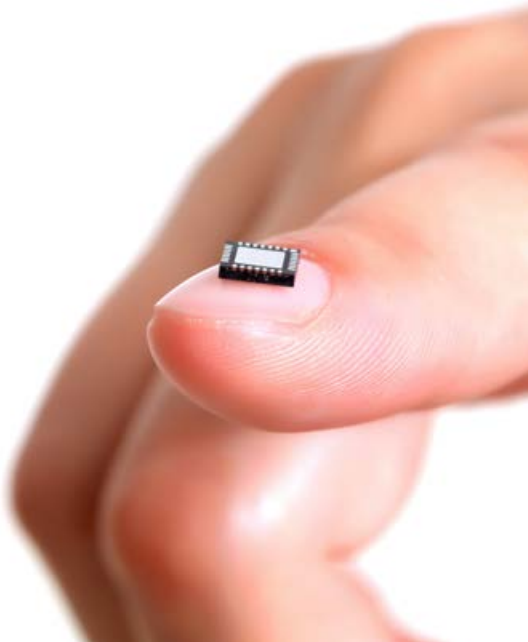




p-ZrHfCoSbSn

In the range between room temperature and 480°C, the Seebeck coefficient of the investigated p-ZrHfCoSbSn material reaches up to 175 µV/K. In the same temperature range, the electrical conductivity drops from 1260 S/cm to 740 S/cm. Both results show a repeatability of ±2%. The measurements were carried out on rectangular samples (3x1x15 mm) in an argon atmosphere at a current of 0.05 A and a voltage of 9 V.

High Repeatability
Reliable!



Radioisotope Thermoelectric Generators (RTG)

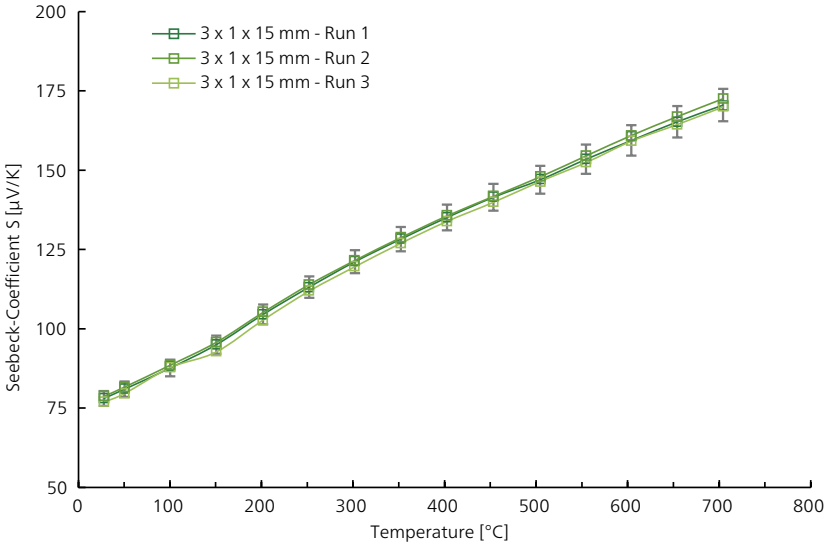
SiGe

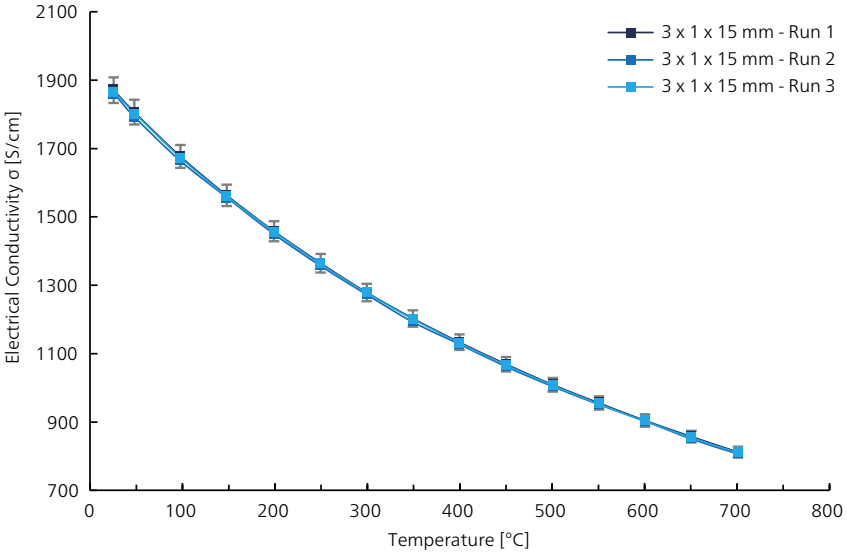
SiGe thermoelectrics ($ZT < 2$) are used in the radioisotope thermoelectric generators (RTGs) that power, for example, spacecraft such as the Galileo, Cassini, and New Horizons. Near the sun, solar cell performance deteriorates due to high incident particle flux and high temperatures from heat flux. However, SiGe thermoelectrics can operate without protection in vacuum and air environments under high temperatures.

The two plots on the right demonstrate the high repeatability of the SBA 458 *Nemesis*® on an SiGe material in a nitrogen atmosphere.

For the Seebeck coefficient, the repeatability amounts to ±3%; for the electrical conductivity, ±2%.

The measurements were performed at a current of 0.01 A and a voltage of 8 V using rectangular samples (3x1x15 mm).

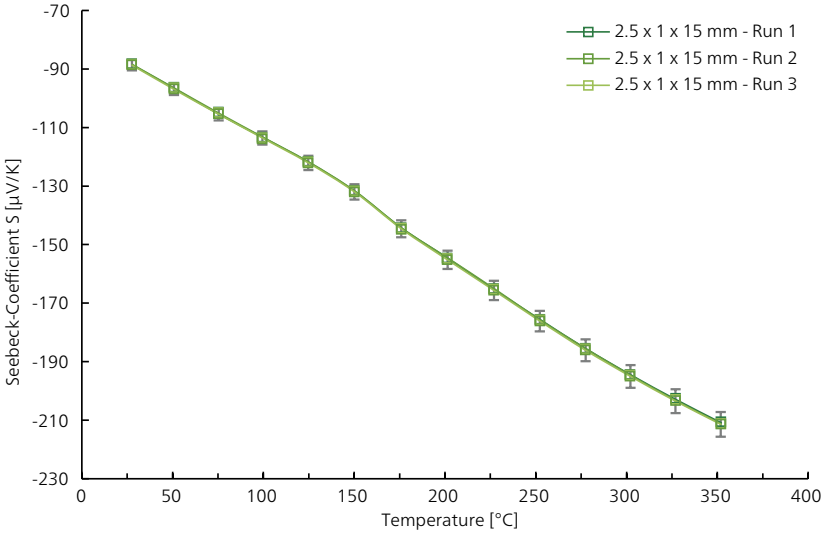


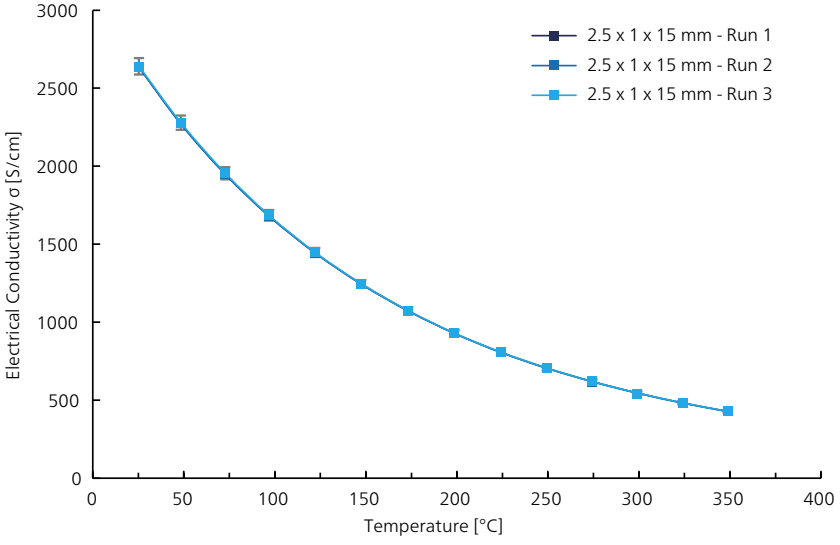


HIGH REPEATABILITY

Lead Telluride – High ZT Material

Recently, enhancements of the figure of merit have been achieved in alloys where *ZT* reaches a value approaching >1.8 for homogeneous PbTe-PbSe materials. This provides incitement for the further development of thermoelectric technologies on Earth.





PbTe

These measurements on PbTe were carried out between room temperature and 350°C in a helium atmosphere at a current of 0.05 A and a voltage of 8 V. The sample dimensions were 2.5x1x1.5 mm.

A repeatability of ±2% was achieved for both the Seebeck coefficient (upper left plot) and the electrical conductivity (lower left plot).

HIGH REPRODUCIBILITY ...

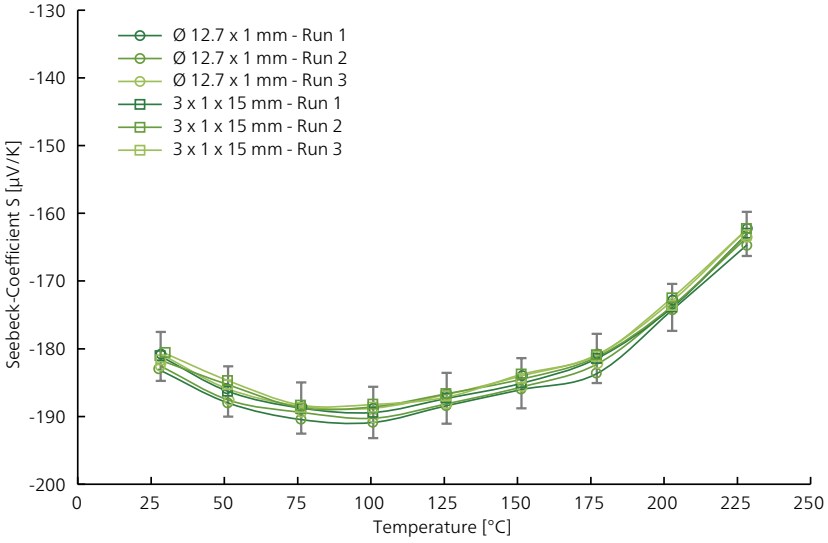
From Chalcogenides to Tellurides

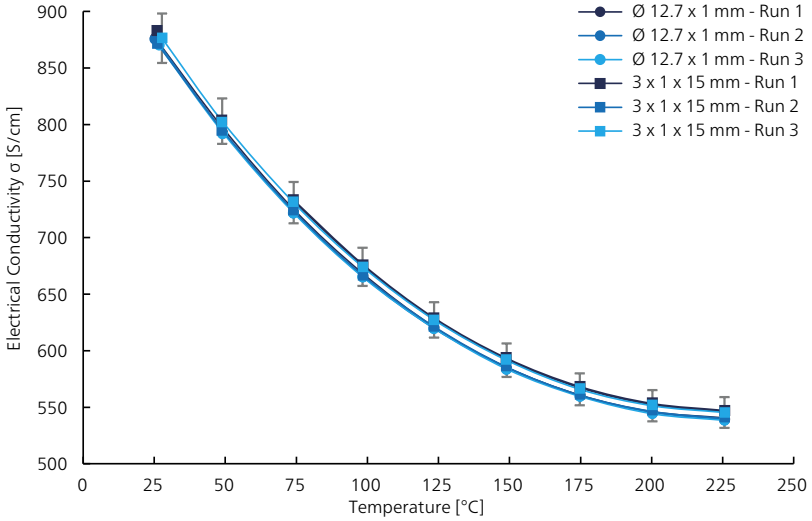
Bi_2Te_3

Bismuth telluride is an efficient thermoelectric material for refrigeration. It is also used in thermoelectric generators when the temperature of the heat source is moderate.

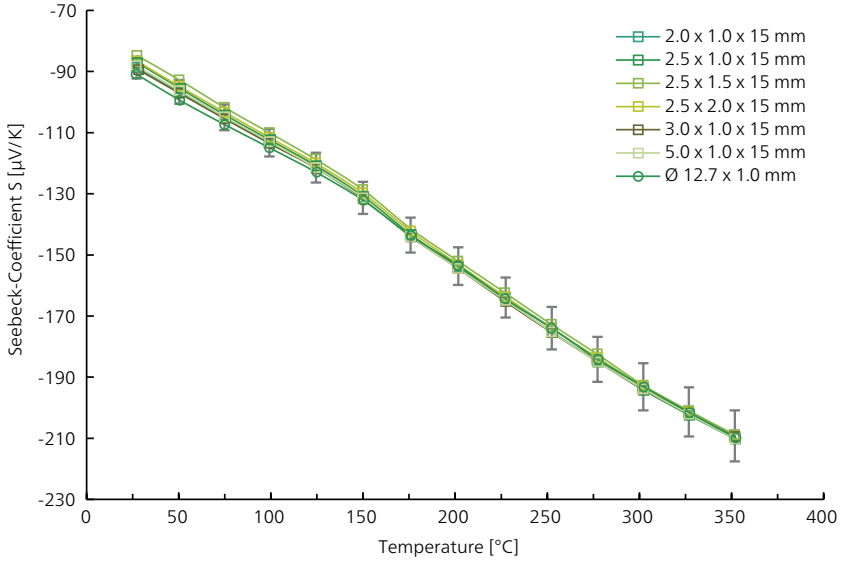
The two plots on the right compare measurements on BiSeTe obtained with round (Ø12.7x1 mm) and square (3x1x15 mm) samples of the same material. The tests were carried out in a nitrogen atmosphere between room temperature and 230°C by applying a voltage of 8 V and a current of 0.01 A.

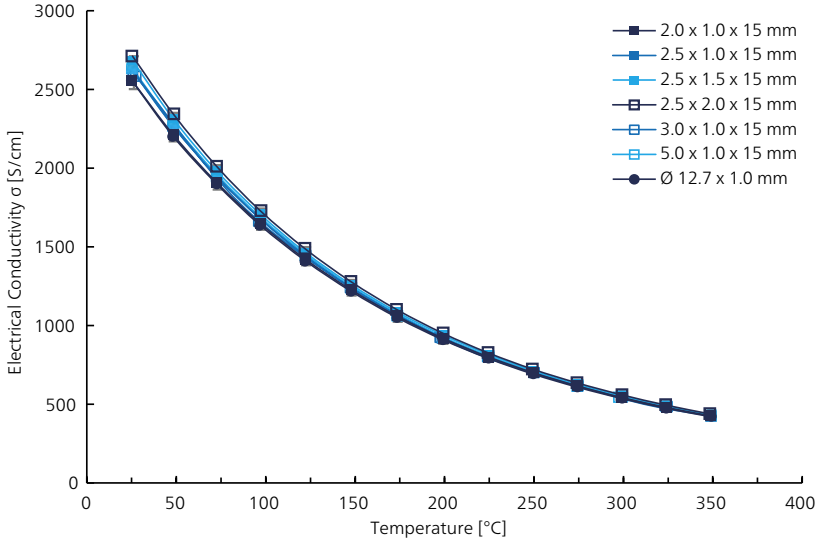
The achieved reproducibility of both the Seebeck coefficient and the electrical conductivity amounted to ±2%.





... EVEN FOR DIFFERENT SAMPLE GEOMETRIES!





PbTe

The high reproducibility of the SBA 458 *Nemesis*® is here demonstrated by various measurements on PbTe in different sample dimensions and shapes.

Under these conditions, the reproducibility of the Seebeck coefficient amounts to ±4% (upper plot) and that of the electrical conductivity also to ±4% (lower plot).

The measurements were carried out between room temperature and 350°C in a helium atmosphere under a current of 0.05 A and a voltage of 8 V.

DETERMINATION OF *ZT*

A single specimen allows for determination of the thermophysical properties including thermal diffusivity and thermal conductivity, specific heat capacity, Seebeck coefficient, and electrical conductivity.

Skutterudite

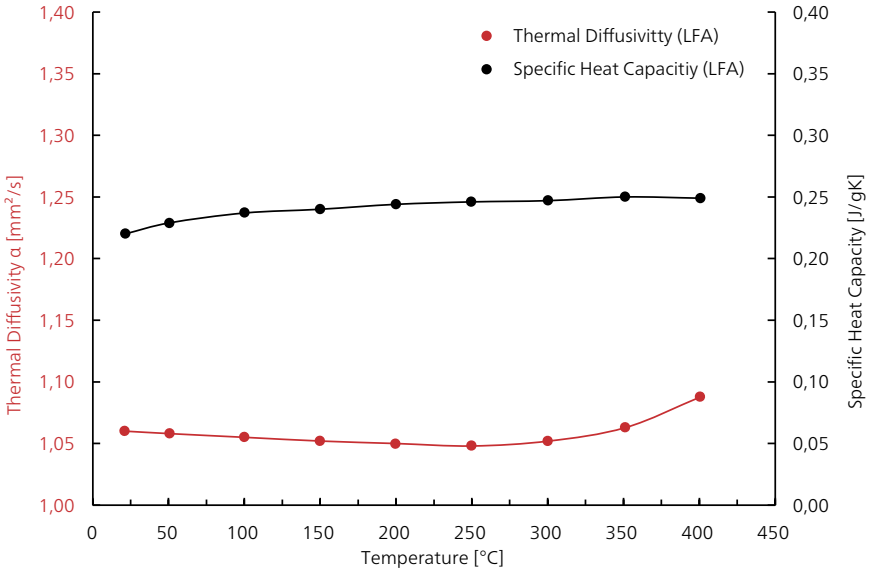
LFA Measurement

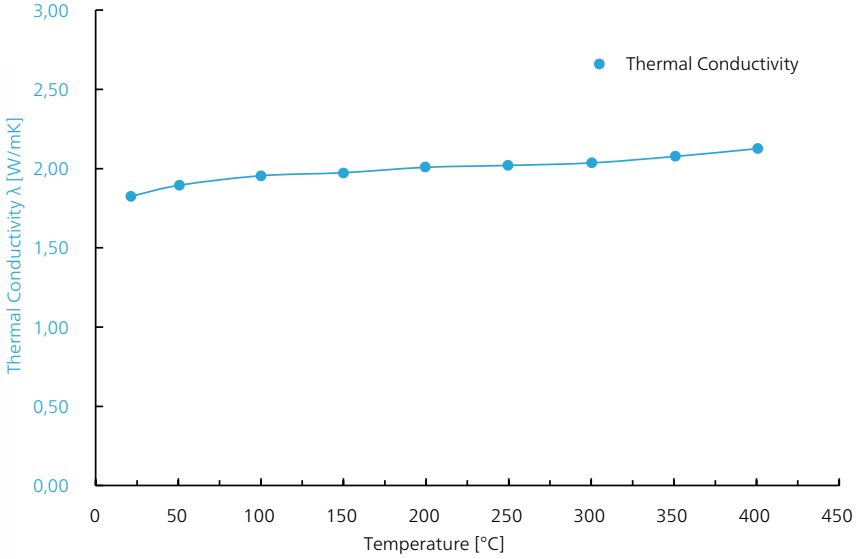
To calculate the dimensionless figure of merit (*ZT*) of skutterudite, the thermal diffusivity (red curve, upper plot) and specific heat capacity (black curve, upper plot) were determined by the LFA method. The test runs were performed between room temperature and 400°C. Calculation of the thermal conductivity (lower plot) is based on the results, using the equation:

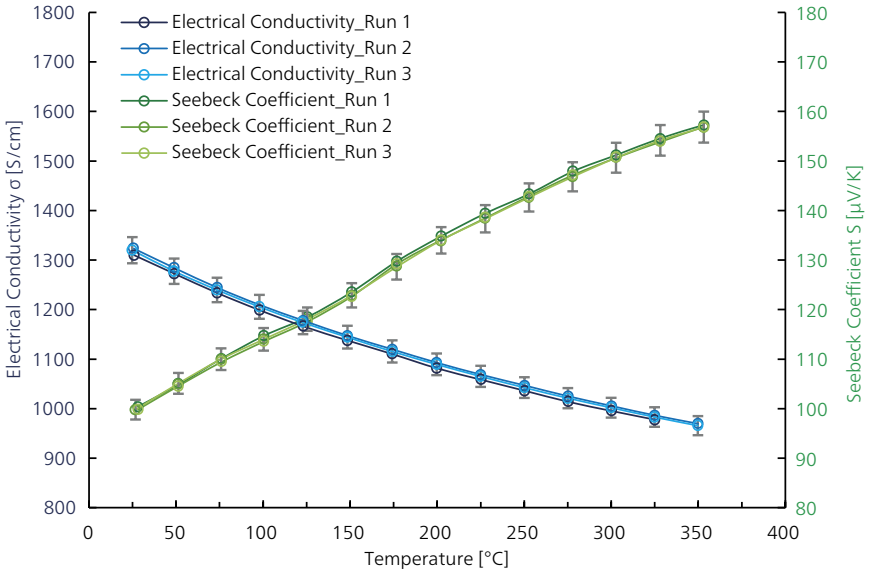
$$\lambda = a \cdot c_p \cdot \rho$$

λ = thermal conductivity
a = thermal diffusivity
c_p = specific heat capacity
ρ = density

LFA tests were carried out with the LFA 467 *HyperFlash*® on a specimen of 12.7 mm diameter.







SBA Measurement

Using the LFA samples, the Seebeck coefficient and electrical conductivity were here determined with the SBA 458 *Nemesis*® between room temperature and 350°C. The Seebeck coefficient increased from 100 µV/K to nearly 160 µV/K, while the electrical conductivity decreased from approximately 1300 S/cm to 1000 S/cm. For both parameters, the measurement results exhibited excellent repeatability (±2%).

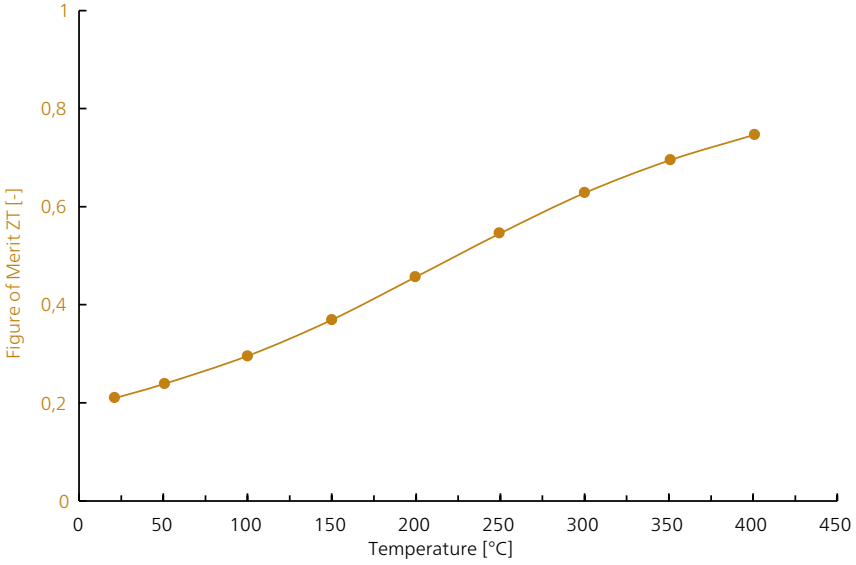


Figure of Merit, *ZT*

ZT is calculated from the results obtained by the LFA and SBA methods using the same sample and applying the following equation:

$$ZT = \left(\frac{S^2 \sigma}{\lambda}\right) T$$

λ = thermal conductivity
S = Seebeck coefficient
σ = electrical conductivity
T = temperature

The plot on the left portrays the increase in *ZT* between room temperature and 400°C, where *ZT* reaches its maximum at 0.75.

Technical Specifications

	SBA 458 *Nemesis*®
Design	Two-heater system, quality check, horizontal sample arrangement
Temperature ranges of the exchangeable furnaces	▪ -125°C ... 500°C ▪ RT ... 1100°C
Temperature setting range	Unlimited number of temperature steps
Thermocouples	▪ Inconel®-sheathed type K (NiCr/NiAl) ▪ Fixed positions
Sample dimensions	▪ □: 10 x 10 mm ▪ Ø: 12.7 to 25.4 mm ▪ ▯: Length x width: 12.7 to 25.4 mm x 2.0 to 25.4 mm ▪ Thickness: 100 nm to 3 mm, depending on the thermophysical properties
Sample geometries	Square, round, rectangular, strips
Seebeck coefficient range	▪ 10 to 2000 µV/K ▪ Accuracy*: ±7% ▪ Repeatability*: ±3%
Electrical conductivity range	▪ 0.05 to 150000 S/cm ▪ Accuracy*: ±5% ▪ Repeatability*: ±3%
Quality check for electrical conductivity	Prior to and during measurement
Quality check for Seebeck measurement	Prior to and during measurement
Number of readings automatically averaged	≥ 100
Atmosphere	Inert, oxidizing, reducing (max. 2% H_2)
Vacuum-tightness	10^{-2} mbar
Software	Based on Windows 7, 32-/64-bit

* for most materials

From A SINGLE Source

for the Thermophysical Properties & Figure of Merit under Defined Conditions

Since the sample geometry is identical to that typically used for Laser/Light Flash Analysis (LFA), there is no longer any need for additional sample preparation. The same sample can be used for determination of the thermal diffusivity/conductivity (LFA) as well as for the Seebeck coefficient and electrical conductivity (SBA), resulting in an improved reproducibility for the thermophysical properties (TPP) and thus, the Figure of Merit (*ZT*).

The gas-tight design of the SBA 458 *Nemesis*® allows measurements to be carried out under defined atmospheres or even reduced pressure – just as with the different NETZSCH LFA systems. Additional information on thermophysical properties is provided by the vacuum-tight DSC 404 *Pegasus*® for precise determination of the specific heat capacity (c_p) and the DIL 402 *Expedis*® dilatometer for the density (ρ).

With our TPP product line, researchers are all set to develop world-best thermoelectric materials at converting waste heat into electricity.

DIL 402 *Expedis*®

LFA 467 *HT HyperFlash*®

DSC 404 ***F1*** *Pegasus*®


NETZSCH Thermokinetics
Curing of Epoxy Resin
Heat flow rate/(W/g)
0
-0.5
-1.0
-1.5
-2.0
-2.5
20.4 K/min
10.1 K/min
5.0 K/min
150
Temperature/°C

Expertise in Service

Our Expertise – Service

All over the world, the name NETZSCH stands for comprehensive support and expert, reliable service, before and after sale. Our qualified personnel from the technical service and application departments are always available for consultation.

In special training programs tailored for you and your employees, you will learn to tap the full potential of your instrument.

To maintain and protect your investment, you will be accompanied by our experienced service team over the entire life span of your instrument.

Summary of Our Services

- Installation and commissioning
- Hotline service
- Preventive maintenance
- On-site repairs with emergency service for NETZSCH components
- Moving / exchange service
- Technical information service
- Spare parts assistance

Our Expertise – Applications Laboratories

The NETZSCH Thermal Analysis applications laboratories are a proficient partner for nearly any Thermal Analysis issue. Our involvement in your projects begins with proper sample preparation and continues through meticulous examination and interpretation of the measurement results. Our diverse methods and over 30 different state-of-the-art measuring stations will provide ready-made solutions for all your thermal needs.

Within the realm of thermal analysis and the measurement of thermo-physical properties, we offer you a comprehensive line of the most diverse analysis techniques for materials characterization.

Measurements can be carried out on samples of the most varied of geometries and configurations. You will receive high-precision measurement results and valuable interpretations from us in the shortest possible time. This will enable you to precisely characterize new materials and components before actual deployment, minimize risks of failure, and gain decisive advantages over your competitors.

The NETZSCH Group is an owner-managed, international technology company with headquarters in Germany. The Business Units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems represent customized solutions at the highest level. More than 3,700 employees in 36 countries and a worldwide sales and service network ensure customer proximity and competent service.

Our performance standards are high. We promise our customers Proven Excellence – exceptional performance in everything we do, proven time and again since 1873.

When it comes to Thermal Analysis, Calorimetry (adiabatic & reaction), the determination of Thermophysical Properties, Rheology and Fire Testing, NETZSCH has it covered. Our 50 years of applications experience, broad state-of-the-art product line and comprehensive service offerings ensure that our solutions will not only meet your every requirement but also exceed your every expectation.

Proven Excellence.

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