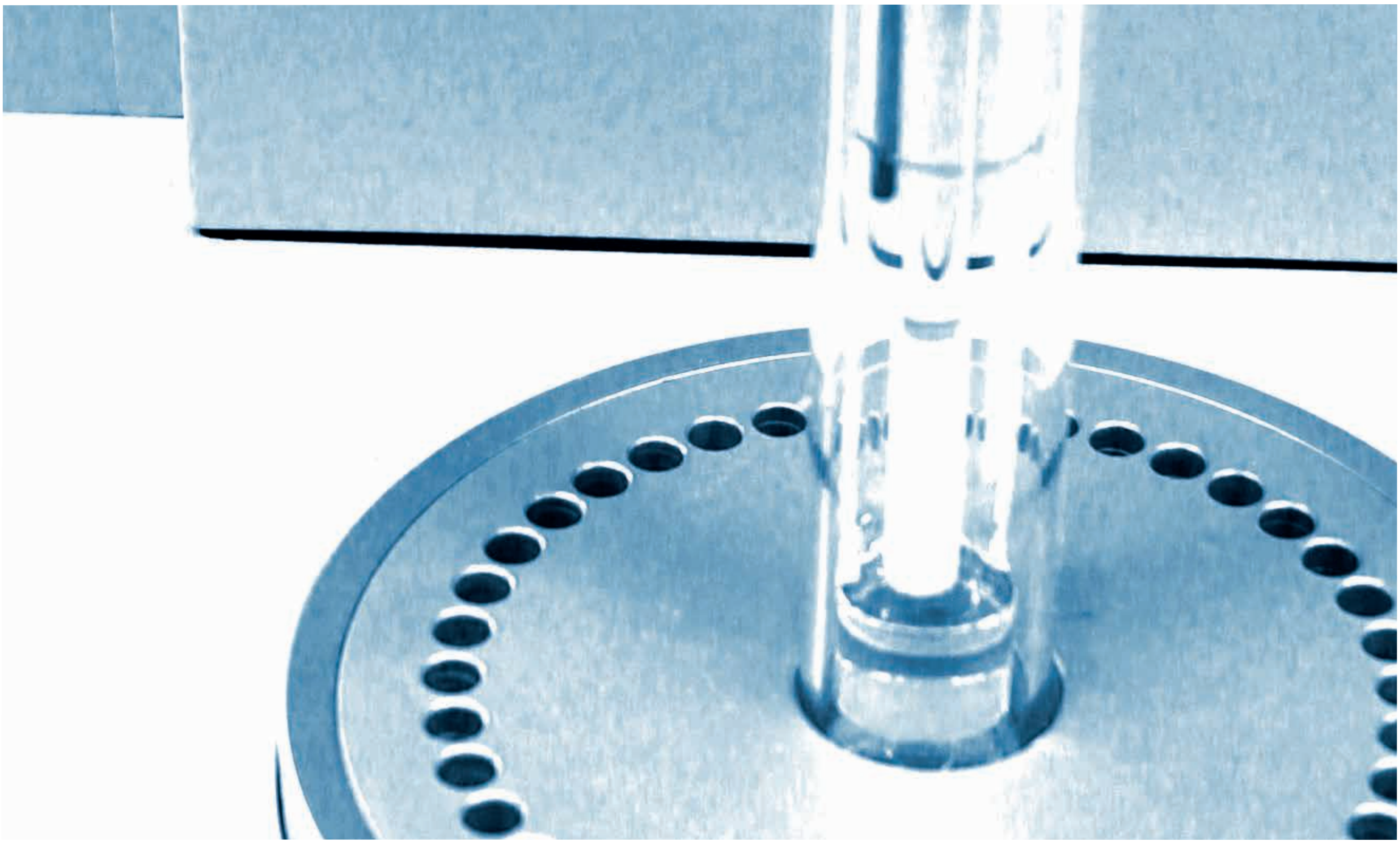
THERMAL ANALYSIS

THERMO-MECHANICAL ANALYSIS

TMA L71 *(former TMA PT 1000)*
TMA L72 *(former TMA PT 1600)*



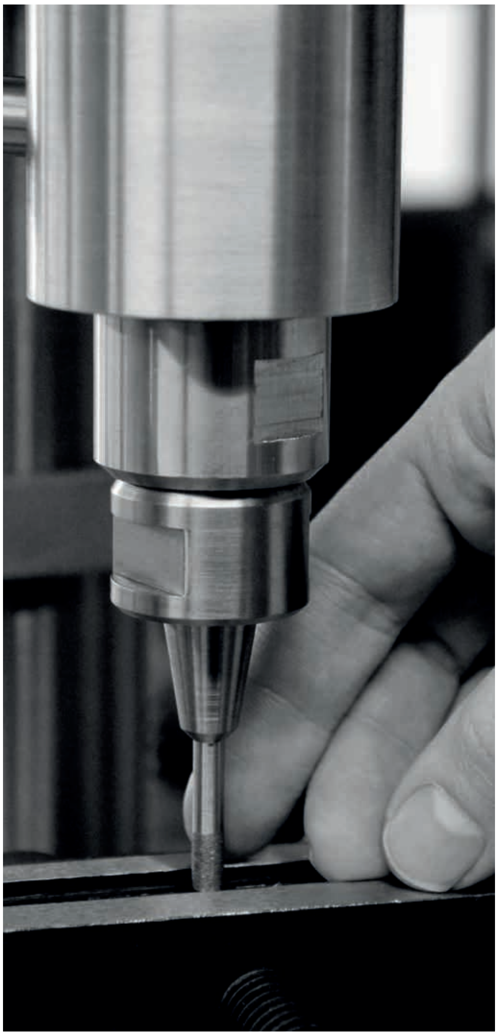
Since 1957 LINSEIS Corporation has been delivering outstanding service, know how and leading innovative products in the field of thermal analysis and thermo physical properties.

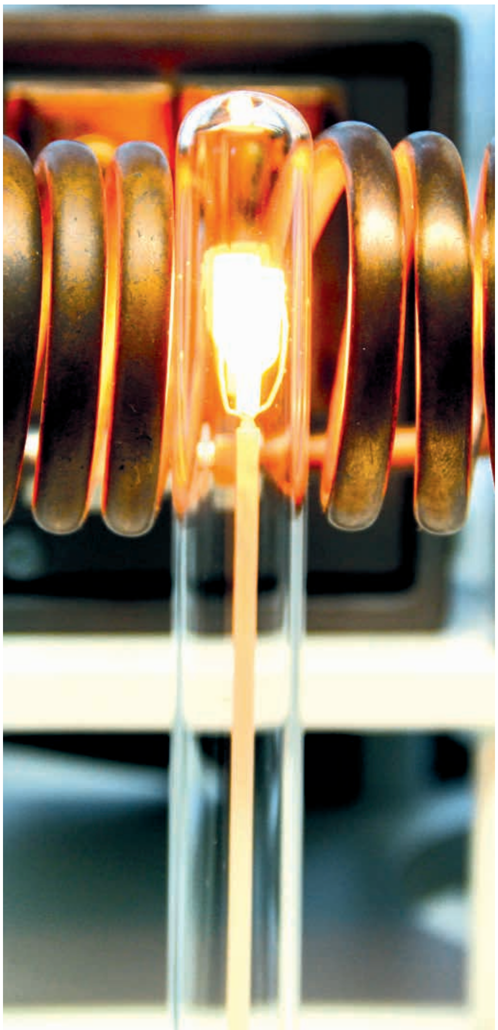
We are driven by innovation and customer satisfaction.

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.

Claus Linseis
Managing Director

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.





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

TMA / DTMA the method

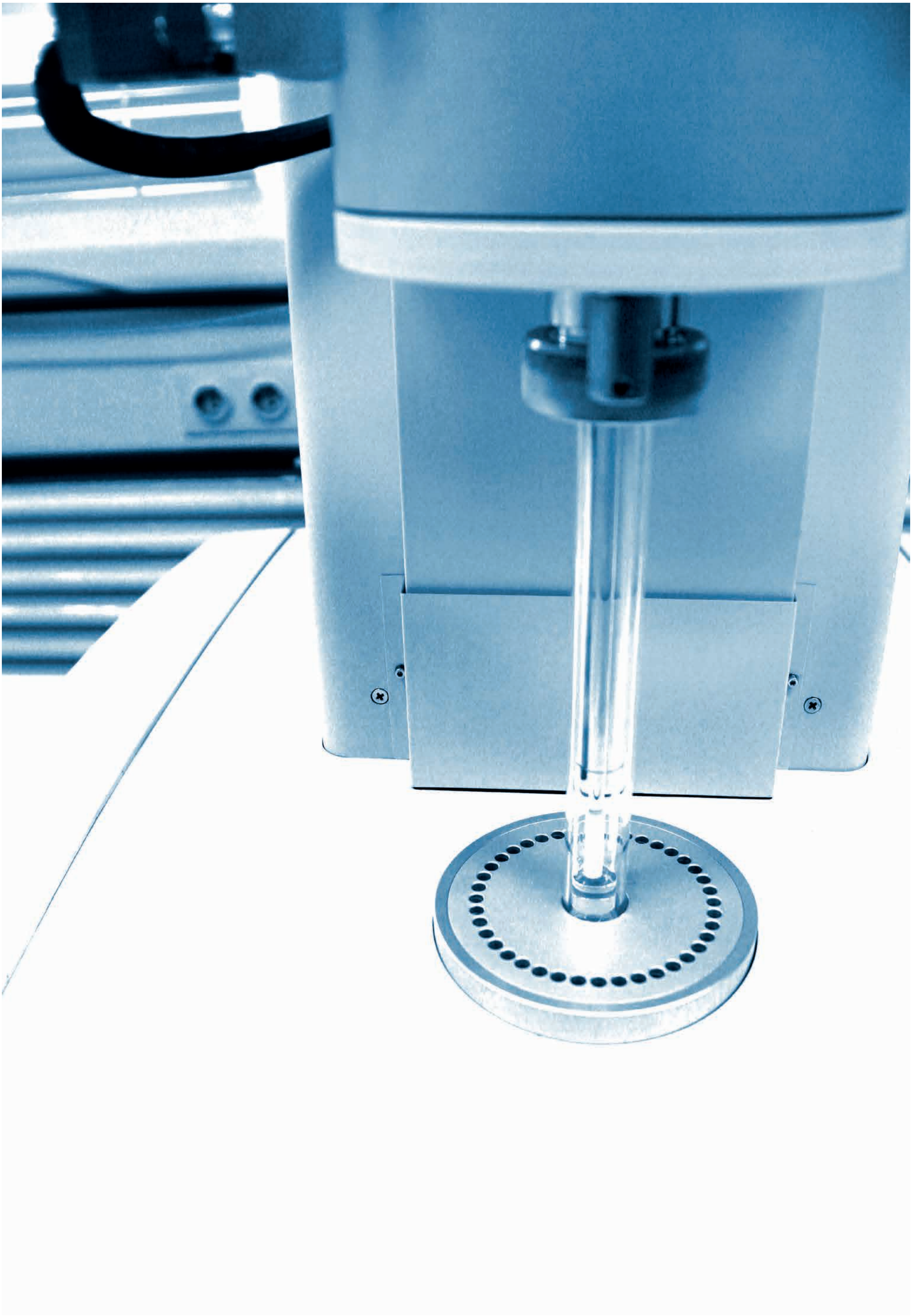
Thermo mechanical analysis (TMA) easily and rapidly measures sample displacement (growth, shrinkage, movement, etc.) as a function of temperature, time, and applied force. Traditionally, TMA is used to characterize linear expansion, glass transitions, and softening points of materials by applying a constant force to a specimen while varying temperature. For expansion measurements, a probe rests on a sample on a stage with minimal downward pressure. Other constant force experiments include measurement of penetration, bending, tension, shrinkage, swelling, and creep (sample motion measured as a function of time under an applied load).

Typical Applications

- Tension studies of the stress/strain properties of films and fibers
- Determination of softening behavior
- Glass transition temperatures and secondary transitions
- Phase change determination
- Determination of mechanical behavior under applied force
- Determination of expansion coefficient (dilatometry)
- Sintering behavior
- Volumetric expansion
- E modulus
- Slipping and friction resistance

- Highest Resolution
  allows to measure smallest nanometer changes
- Dynamic Load TMA
  measures weak transitions and elasticity
- Wide measuring range
  from - 150°C to 1600°C*
- Calculated DTA
  simultaneous measurements of thermal effects
- Modular design
  allows future expansion of instrument
- Gas tight cell
  controlled measurement environment
- Hyphenated techniques
  evolved gas analysis

* different furnaces (TMA L72)



THE CONCEPT

Sample chamber

The easily accessible chamber is located in the center of the furnace. Both temperature and atmosphere can be controlled. In addition an optional mass flow controller is available for purge gas regulation. The gas tight cell can be evacuated and allows you to measure under a defined atmosphere. Only such a system can provide definitive information concerning the samples sensitivity to oxidation.

Furnace

The TMA Platinum Series comes with a robust and reliable furnace. Its customized design enables rapid heat up and cool down times and an excellent heating rate control over the entire temperature range.

The expansion and temperature sensor

Every dimensional change of the sample is transmitted via the pushrod to the highly precise inductive transducer (LVDT sensor). Its precise and reliable response over the entire temperature range guarantees highest reproducibility of the TMA results. The temperature sensor is located right beside the sample leading to the high accuracy.

The Dynamic TMA mode

This feature allows you to study the visco-elastic behavior of materials. In D-TMA the force exerted on the probe alternates automatically by the given frequency.

Sample Holders

A broad range of sample holders is available for the TMA. Hence the best method for testing can be selected for every application. Furthermore LINSEIS can certainly provide aid for special customer requirements.

Automatic pressure control

The contact pressure can be continuously varied between 10mN and 20 N depending on the system. This feature continuously adjusts the contact pressure throughout expansion and/or shrinkage of the sample.

Cooling system

The liquid nitrogen cooling system has been completely automated; manual refilling is not necessary. This simplifies operation, improves reproducibility and allows measurements to be performed over a long period of time.

Integrated DTA signal

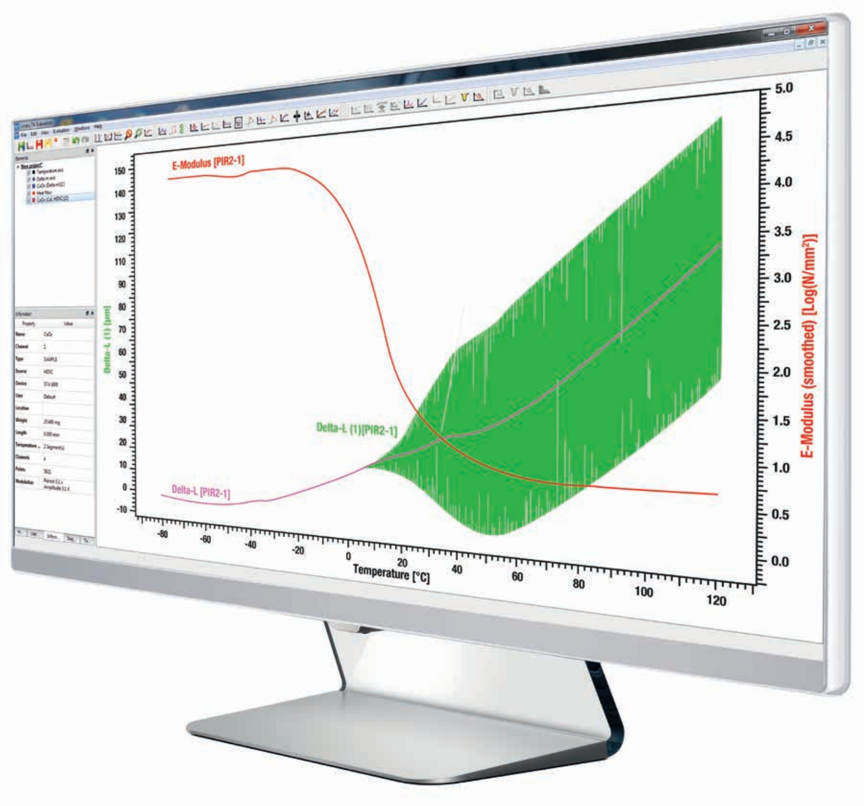
All LINSEIS TMA models are optionally equipped with the DTA evaluation feature. This provides the user with valuable additional endo- and exothermic sample information

SOFTWARE

All LINSEIS thermo analytical instruments are PC controlled. The individual software modules exclusively run under Microsoft® Windows® operating systems. The complete software consists of 3 modules: temperature control, data acquisition and data evaluation. The 32 bit software incorporates all essential features for measurement preparation, execution, and evaluation of a TMA/DTMA run. Thanks to our specialists and application experts, LINSEIS was able to develop comprehensive easy to understand user friendly application software.

Features -Software

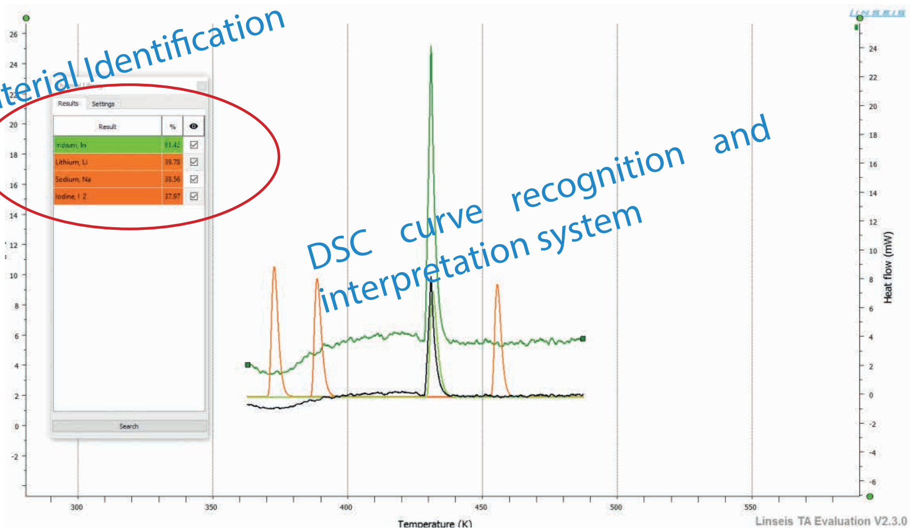
- Program capable of text editing
- Data security in case of powerfailure
- Thermocouple break protection
- Repetition measurements with minimum parameter input
- Evaluation of current measurement
- Curve comparison up to 32 curves
- Storage and export of evaluations
- Export and import of data ASCII
- Data export to MS Excel
- Multi-methods analysis (DSC TG, TMA, DIL, etc.)
- Zoom function
- 1st and 2nd derivation
- Programmable gas control
- Statistical evaluation package
- Automatic axis re-scaling
- E-Modulus
- Several system correction features
- Automatic zero point adjustment
- Auto-scheduler for up to 16 uninterrupted runs



Thermal Library

The LINSEIS Thermal Library software package comes as an option for the well-known, user friendly LINSEIS Platinum evaluation software that is integrated in almost all our instruments.

The Thermal Library allows you the comparison of the complete curves with a data base providing thousands of references and standard materials within only 1-2 seconds.



Measured and matching reference curves (green) and reference curves with poor fit (red).

Multi-Instrument

All LINSEIS instruments DSC, DIL, STA, HFM, LFA, etc. can be controlled from one software template.

Report Generator

Convenient template selection to generate customized measurement reports.

Data Base

State of the art data base design enables easy data handling.

Multi-Lingual

Our software is available in many different user exchangable languages, such as: English, Spanish, French, German, Chinese, Korean, Japanese, etc.

Multi-User

The administrator can generate different user levels providing different rights to operate the instrument. A optional Log file is available, too.

Kinetic software

Kinetic analysis of DSC, DTA, TGA, EGA (TG-MS, TG-FTIR) data for the study of the thermal behavior of raw materials and products.

SPECIFICATIONS

	TMA L71 EM	TMA L71	TMA L72
Temperature range	-150 up to 1000°C	-150 up to 1000°C	-180 up to 500°C RT up to 1400/1600/ 1750 /2000/2400°C
Force	1 or 5.7N	1, 5.7 or 20N	1 or 5.7N
Frequency	1 or 5Hz	—	0.05 to 1 or 50Hz
Resolution	0.125 nm/digit	0.125 nm/digit	0.125 nm/digit
Sample size	30/50mm	30/50mm	30/50mm
Atmosphere	inert, reduced react. gas	inert, reduced react. gas	inert, reduced react. gas
Temeperature precision		± 1°C	
Heating rate		0.1 to 100°C	
Isothermal temperature control		± 0.1°C	
Measurement precision for (CTE)		± 0.1% or better	
Sensitivity		1.25nm	
Dynamic baseline drift		≤ 1µm	
Force range		0.001 to 1 N	
Force resolution		0.001 N	
Operation of the system		system must be operated in... ...Standard operating mode i.e. temperature ramp, isostrain, force ramp ...Creep, stress relaxation, dynamic TMA	
Frequency range		0.01 to 1 Hz	
Purge gas control		digital mass flow controller must be provided for purge gas Option: L40/2051/MFC	
Sample size (solid)		min. length 20mm max. length up to 50mm diameter up to 10 mm	
Sample size (film/fibre)		up to 25mm(L), 0.4-0.6mm(T), up to 5 mm(W)	
Measurement mode		1. Standard expansion/shrinkage measurement: L77/150 2. Stress/Strain L77/130 3. Creep L77/130 or L77/150 4. Stress Relaxation L77/150 Dynamic TMA-storage modulul (E'), loss modulus (E") and tan (E"/E') are calculated as functions of temperature, time or stress	

TMA / DTMA FEATURES

With low constant load

- Linear thermal expansion evaluation
- Change of volume
- Phase transformation
- Sinter process evaluation
- Softening point determination
- Transformation points
- Swelling behavior
- Tension

With increased constant load

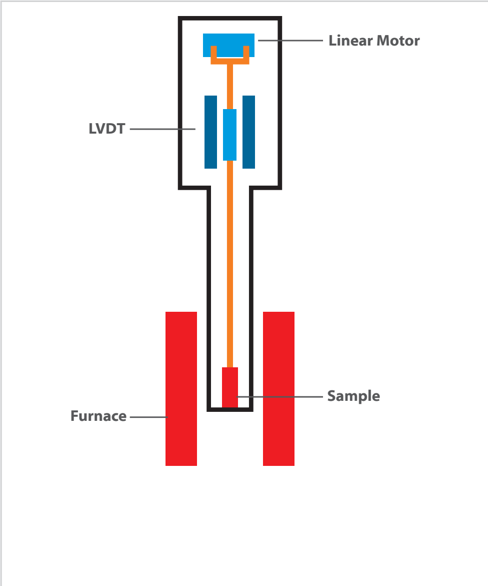
- Penetration
- Transition and comparison tests
- 3 point bending test

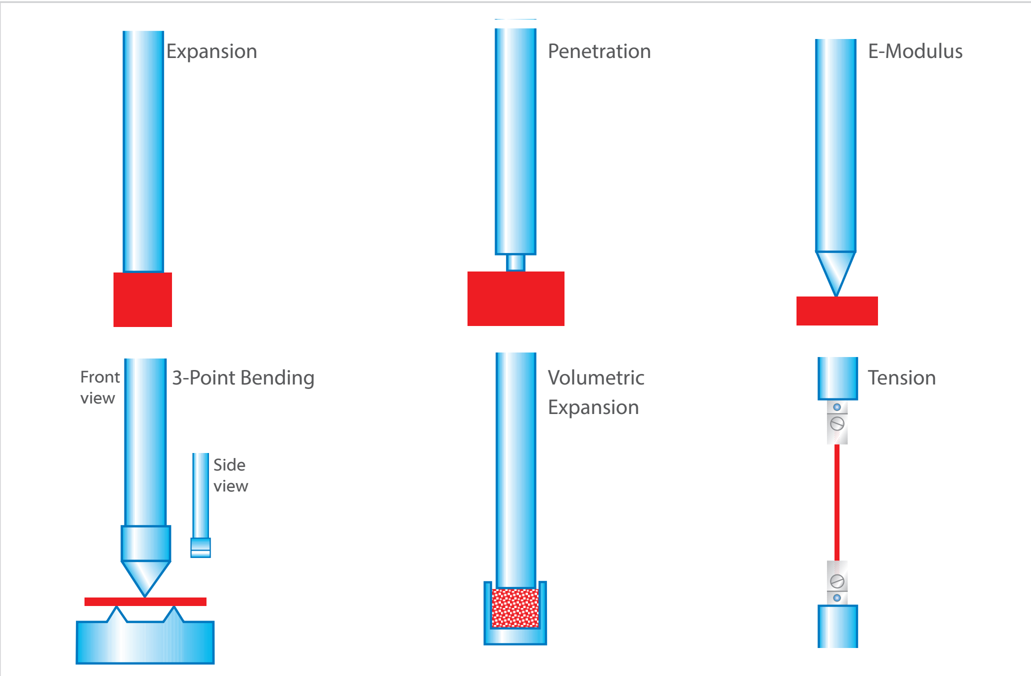
With dynamic load

- Visco-elastic behavior

Additional optional features

- DTA evaluation
- (RCS) Rate controlled sintering software





TMA with relative humidity

This innovative version of our proven TMA system allows you to perform thermomechanical analysis under precisely controlled humidity conditions. This is particularly advantageous for materials that are sensitive to the effects of moisture and therefore require more precise analysis.

FURNACES

TMA L71

Temperature	Type	Element	Atmosphere	Temperature sensor
-150°C* – 1000°C	L75/220	Kanthal	inert, oxid., red., vac.	Type K

*low temperature option

TMA L72

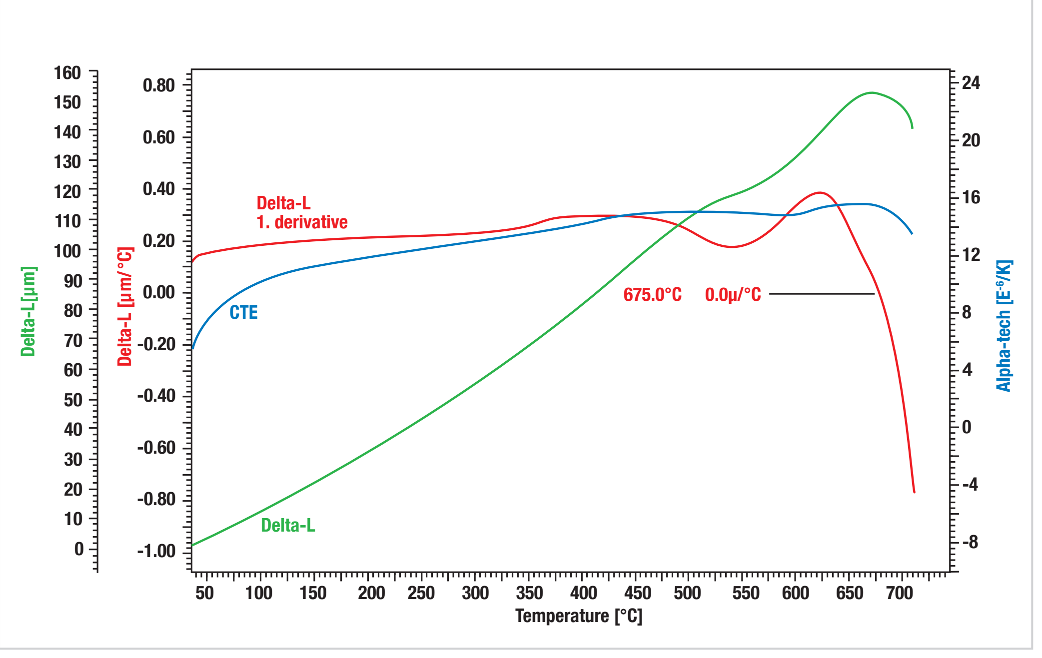
Temperature	Type	Element	Atmosphere	Temperature sensor
-180 – 500°C	L75/264	Thermo coax	inert, oxid., red., vac.	Type K
-180 – 700°C	L75/264 L75/220	Thermo coax	inert, oxid., red., vac.	Type K
RT – 1000°C	L75/220	Kanthal	inert, oxid., red., vac.	Type K
RT – 1400°C	L75/230	Kanthal	inert, oxid., red., vac.	Type S
RT – 1600°C	L75/240	SiC	inert, oxid., red., vac.	Type S
RT – 2000°C	L75/260	Graphite	N_2/Vac.	Type C and/or Pyrometer

SPECIFICATIONS

Temperature range	-150°C up to 2000°C
Temperature controller	Programmable
Temperature resolution	0.1 K
Heating rate/cooling rate	0.01 to 50°C/min programmable
Furnace construction	SiC furnace Model L75 is upgradable to any other furnace type
Environment compatibility	All gaseous environment possible
Maximum attainable vacuum	1x 10^{-3}mbar
Total displacement measurement range	+/- 25mm
Displacement resolution	0.1nm
Reproducibility of CTE measurement	1x10^{-6}/k CTE
Maximum allowable drift in CTE measurement	< 10nm/°C
Sample length	maximum 50 mm
Diameter/cross-section	6 mm
Sample holder	tube and rod types

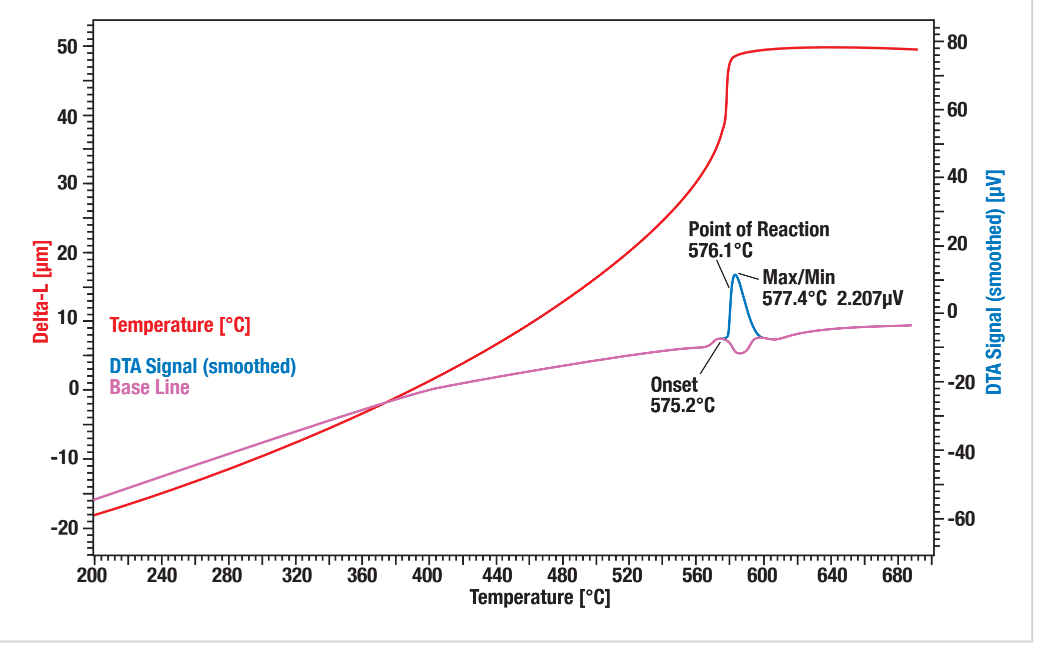
APPLICATIONS

Glass Ceramic



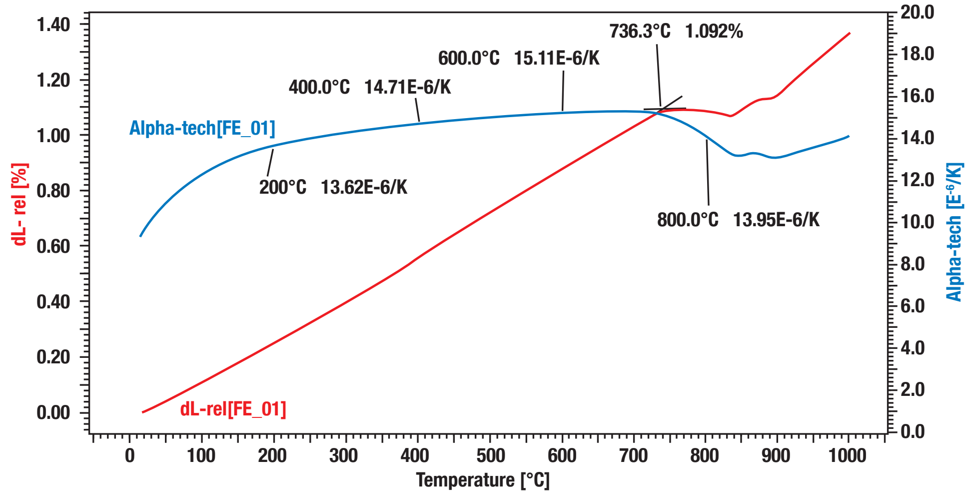
The dilatometric method is an excellent method to determine the thermal expansion (CTE) and the softening point of glass ceramic materials. Besides the absolute expansion and the expansion coefficient (CTE) you can find the first derivative of the absolute expansion. Where the first derivative goes through zero you can determine the max. of the thermal expansion and thus the softening point of the material.

DTA - Feature



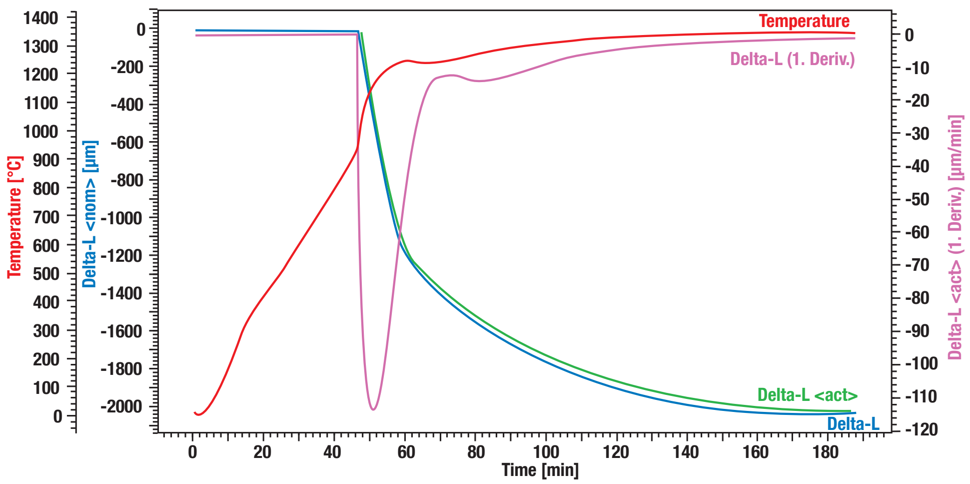
The thermal expansion of rock crystal (α-SiO_2) can be easily evaluated with the L75 Dilatometer. The additional DTA feature enables an in depth view of the thermal behavior of the material. The DTA measurement is a mathematical routine based on the sample temperature. Exo- and endothermic effects influence the change of the sample temperature during the dynamic heating or cooling cycle. At app. 575°C the phase transition from α- to β-SiO_2 takes place. The deviation of the measured temperature from the literature value (574°C) can be used for a temperature calibration.

Iron



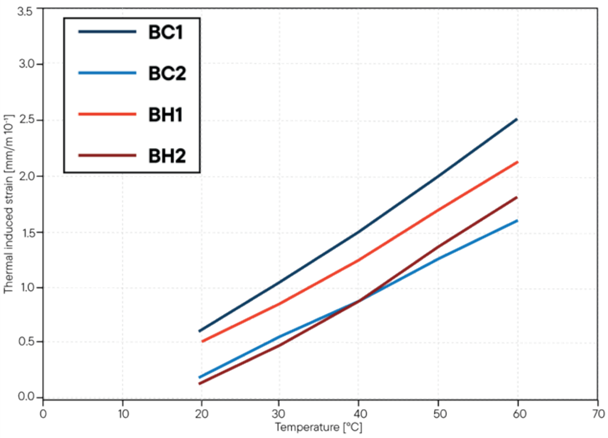
The linear thermal expansion (ΔL) and the CTE of the iron sample under argon atmosphere are evaluated. The heating rate was 5K/min. After 736.3°C (peak temperature of CTE) shrinkage was detected, which is due to a change in the atomic structure, known as the curie-point. The difference of measured and literature result can be attributed to contamination of the sample.

Ceramics / Powder metallurgy

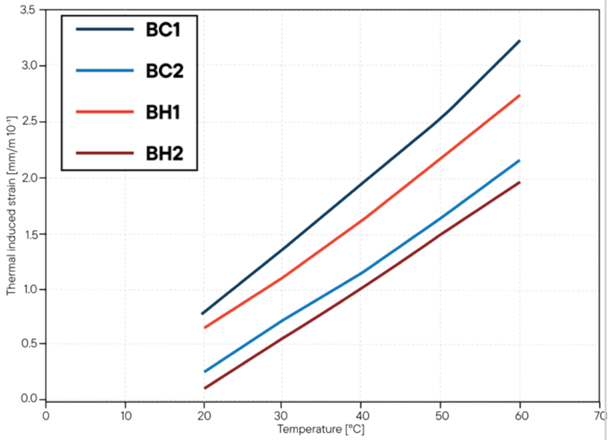


The glass sample was measured with the softening-detection feature. In this case, the heating ramp is terminated either if the target temperature set is reached, or if the shrinkage of the sample reaches a given threshold. In the run, the threshold was set to −100µm – when the change in length measured falls 100µm below the maximum value of the actual heating segment, the heating is terminated and the next (cooling) segment will be processed. So, the softening point can be detected in an easy and safe procedure.

Difference in thermal expansion behavior of bricks depending on relative humidity



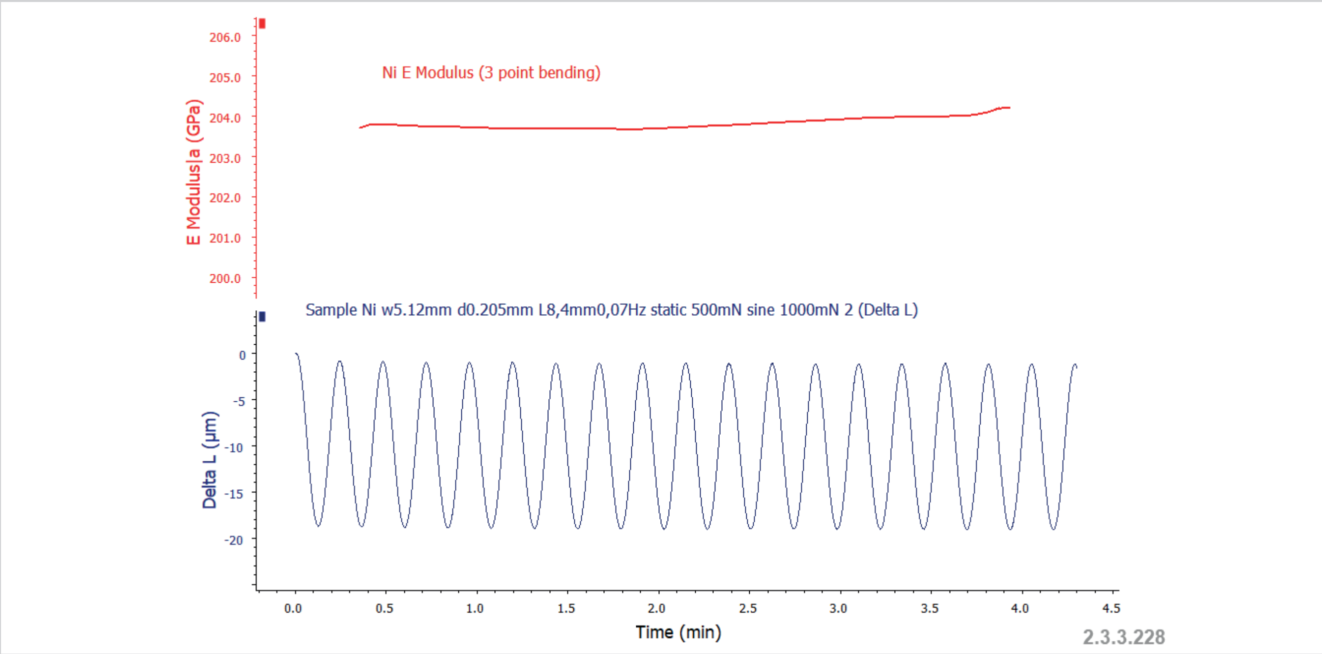
Thermal expansion of historical bricks BH1 and BH2 in comparison with contemporary bricks BC1 and BC2 at dry conditions



Thermal expansion of historical bricks BH1 and BH2 in comparison with contemporary bricks BC1 and BC2 at 35% relative humidity

Humidity can have a huge influence on many materials, especially materials that are used for packaging or construction such as polymers, wood, bricks, concrete and many more. In the given application, two historical brick variants have been investigated and compared to contemporary, similar bricks regarding their expansion at different humidity levels. Therefore, the materials were first measured at linear expansion mode using a Linseis TMA L72. The result was a straight forward linear expansion. Then the same experiment was repeated using a water vapor generator to simulate 35% relative humidity. The samples were kept at these conditions for 2 h and then the expansion coefficient was again determined vs temperature keeping the humidity level constant. The result shows a significant increase in linear expansion behavior due to swelling of the material. This effect will occur at any humidity level and should be considered carefully when calculating the expansion of such materials for any application.

Determination of Young’s Modulus using Linseis TMA PT1600



The Linseis TMA L72 can apply sinus shaped force programs to any sample. This enables the user to determine the bending and also Young’s Modulus of almost any material. Initially designed for mainly polymer applications, the Linseis TMA PT 1600 has recently been upgraded to be able to measure also harder materials such as metal alloys and pure metals, using force ranges up to 20 N by enlarging the possible sample size. In the application above a nickel sample of 8.4 mm length, 5.12 mm diameter and 0.2 mm thickness was measured using a static force of 500 mN and a fluctuating sinus shaped force of additional 1 N. The blue curve shows the movement of the sample as change in length that is caused by the force and the red curve shows the resulting E-modulus of this 3-point-bending experiment. The experiment was performed at 50 °C and is very close to literature data. One could also perform this at any temperature the instrument could achieve to be able to screen a larger range.

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Services: Service Lab, Calibration Service

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