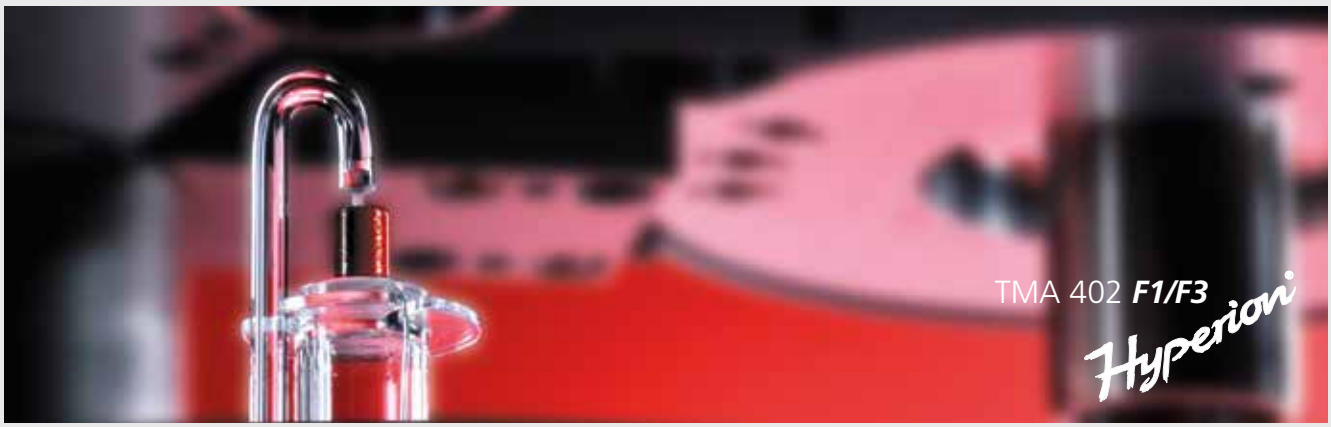
NETZSCH

Analyzing & Testing

Thermomechanical Analysis – TMA

Method, Technique, Applications

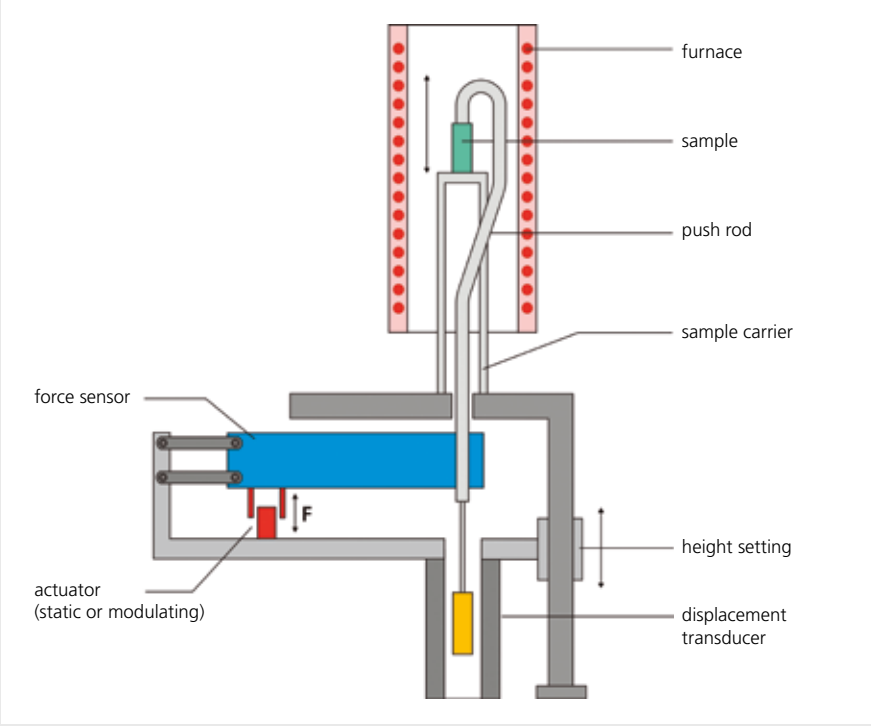


Leading Thermal Analysis

Thermomechanical Analysis – TMA

Thermomechanical analysis (TMA) determines dimensional changes of solids, liquids or pasty materials as a function of temperature and/or time under a defined mechanical force (DIN 51 005, ASTM E 831, ASTM D 696, ASTM D 3386, ISO 11359 – Parts 1 to 3). It is closely related to dilatometry, which determines the length change of samples under negligible load (DIN 51 045).

Many materials undergo changes of their thermomechanical properties during heating or cooling. For example, phase changes, sintering steps or softening can occur in addition to thermal expansion. TMA analyses can hereby provide valuable insight into the composition, structure, production conditions or application possibilities for various materials.
The application range of instruments for thermomechanical analysis extends from quality control to research and development. Typical domains include plastics and elastomers, paints and dyes, composite materials, adhesives, films and fibers, ceramics, glass and metals.



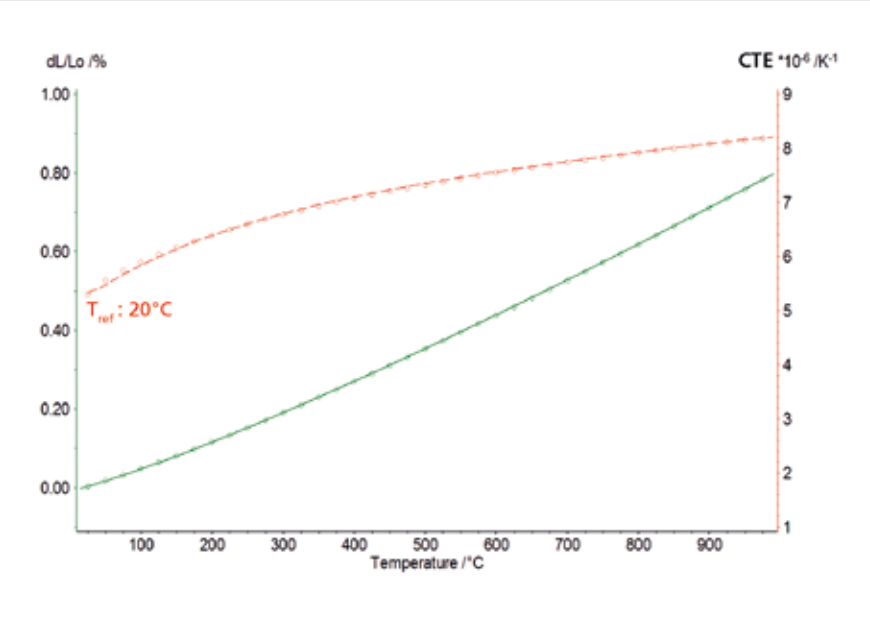
TMA: Operating principle

Operating Principle

Irrespective of the selected type of deformation (expansion, compression, penetration, tension or bending), every length change in the sample is communicated to a highly sensitive inductive displacement transducer (LVDT) via a push rod and transformed into a digital signal. The push rod and corresponding sample holders of fused silica or aluminum oxide can be quickly and easily interchanged to optimize the system to the respective application.

Results and Accuracy

The linear thermal expansion is an important variable for assessing the behavior of a material.
This chart shows the thermal expansion (dL/L_0 in %) and the resulting curve of the coefficient of thermal expansion (CTE in 1/K) of sapphire compared to literature values (circles). The results demonstrate the high accuracy which can be achieved using the TMA 402 *Hyperion*®.



TMA 402 ***F1*** *Hyperion*® with double furnace hoist

At a Glance

What sets the new TMA 402 *F1/F3 Hyperion*® apart:

- A modular concept of interchangeable furnaces covering the temperature range from -150°C to 1550°C, which are also compatible with other NETZSCH instruments
- The large number of sample holder types and adjustment possibilities
- The broad range of force, without added weight, digitally programmable from -3 N to 3 N

TMA 402 *Hyperion*® – Trend-Setting Technology

The Heart of the TMA 402 *Hyperion*® is a Highly Precise Inductive Displacement Transducer (LVDT)

This technology has stood the test of time; it is also used for dilatometers and allows measurement of even the smallest of length changes, into the nanometer range (digital resolution of 0.125 nm).

Vacuum-tight Thermostatic Measuring System

The entire TMA 402 *Hyperion*® measuring system is thermally stabilized via water-cooling. This ensures that the measurement will not be influenced by heat from the furnace or by temperature fluctuations in the local environment.

All joints have a vacuum-tight design to allow measurements in a highly pure atmosphere or under vacuum. Pressures of less than 10^{-4} mbar can be achieved with the use of a turbomolecular pump. In combination with the integrated mass flow controllers (MFC) for purge and protective gases (optional in the TMA 402 ***F3***), measurements in highly pure inert gas or in oxidizing atmospheres can be optimally controlled.

Further Information

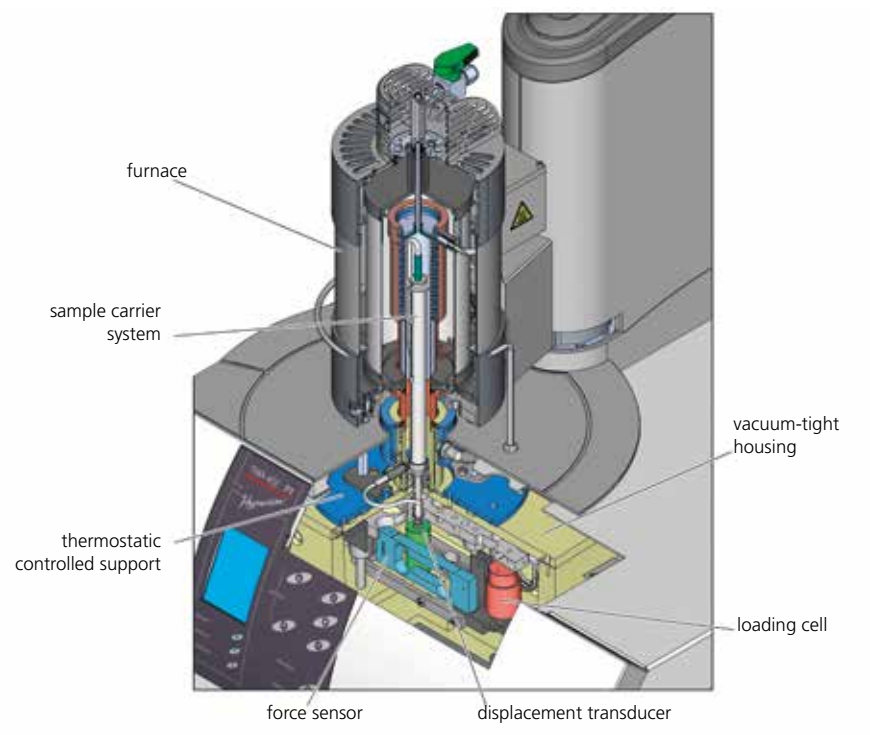
www.netzsch.com/n10830

Simultaneous Measurement of Force and Displacement Signal

The force operating on the sample is generated electromagnetically in the TMA 402 *Hyperion*®. This guarantees a quick response time for experiments with a changing load, e.g. tests on creep behavior. A highly sensitive force sensor (digital resolution < 0.01 mN) continuously measures the force exerted via the push rod and readjusts it automatically. This sets the TMA 402 *Hyperion*® apart from other instruments, which use only preset values.

Precise Force Control

The electronic control system for the TMA 402 *Hyperion*® allows forces to be set in the mN-range. This enables testing even on sensitive materials such as thin fibers or films. The force operating on the sample can be altered via the software in a stepwise or linear fashion. This makes it particularly simple to carry out such analyses as creep or stress sweep tests. The premium version of the *Hyperion*®, the TMA 402 ***F1***, provides even more capabilities. From single pulse in rectangular or ramp form to continuous modulation with a customizable frequency (up to 1 Hz), every possibility is covered. This model is particularly well-suited for determining visco-elastic material properties such as elasticity and creep modulus.



TMA 402 *Hyperion*®: Sectional Drawing

Technical Data	TMA 402 ***F1/F3***
Max. sample length	30 mm
Measuring Range	5000 µm (total) / 500 µm
Dig. resolution (length)	1.25 nm / 0.125 nm
Force range	0.001 N to 3 N in steps of 0.2 mN (tension or pressure)
Dig. resolution (force)	< 0.01 mN
Modulated force	Up to 1 Hz (only ***F1***)
Final vacuum pressure	< 10^{-4} mbar
Gas connections	Protective gas, 2 purge gases
MFC	Standard (***F3*** optional)

Highest Precision – Maximum Flexibility

The Modular Design of the TMA 402 *Hyperion*® Makes it Unique among the Competition

To adjust the instrument for the respective temperature ranges, all that needs to be done is to exchange the furnace. This can be done by the operator. Due to the double furnace hoist, switching to a second furnace takes only moments.

Further Information

www.netzsch.com/n10830

Interchangeable Furnaces

The furnaces can be easily interchanged among various high-temperature series thermal analysis instruments (STA 449 *Jupiter*®, DSC 404 *Pegasus*®). The selection of available furnace models is continuously being expanded. The TMA 402 *Hyperion*® can thus cover the entire temperature range from -150°C to 1550°C and is prepared for temperature extensions.

Sample Holder Systems Tailored to the Task

Depending upon the question at hand and the geometry of the sample, the operator has a variety of sample holders to choose from. Holding devices for expansion, penetration, and tension measurements are available, as well as push rods and supports for analyses in 3-point bending. Accessories for the temperature range up to 1000°C are made of fused silica. For the high-temperature range, aluminum oxide varieties are available.

In the TMA 402 *Hyperion*®, the thermocouple can be changed quickly and easily. Besides type K, the S and E varieties are also available. The system electronics recognizes the installed sensor automatically.

Special Sample Holders

With the help of special sample containers, the expansion behavior of powders, pastes and liquids can be analyzed, as can metals all the way to the melting point. Accessories for experiments on swelling behavior upon immersion are also available.

Coupling with MS / FT-IR

To analyze gases evolving upon heating (EGA), the TMA 402 *Hyperion*® can be coupled to a mass spectrometer and/or FT-IR spectrometer.

Common System Configurations by Temperature Range

-150°C to 1000°C	Steel furnace with LN_2 cooling, sample holder system of fused silica, Type K thermocouple
RT to 1550°C	SiC furnace, sample holder system of Al_2O_3, Type S thermocouple

Other configurations can easily be realized due to the modular concept.

Feature	Advantage	Benefit
Modular concept	Furnaces are interchangeable and compatible with other NETZSCH instruments	Synergies in procuring and operating, easy and cost-effective expansion and retrofitting
Gas flows controllable via software	Programmable atmosphere change	Analysis of, for example, oxidation behavior without manual valve operation
Digitally programmable force control up to 3 N	Force alteration (linear/stepwise), continuous force modulation (TMA 402 ***F1***)	Determination of visco-elastic properties
A variety of accessories, e.g. special sample holders, immersion baths	Extension of the application range	Measurement of powders, liquids, pastes or metals (until melting), immersion measurements

Sample holder and push rods
(fused silica, aluminum oxide)

Proteus® Software for the TMA 402 ***F1/F3*** *Hyperion*®

The software includes everything necessary for carrying out measurements and evaluating the results. Simple menu navigation and automated routines make the software easy to operate while allowing for complex analyses.

General Software Characteristics:

- For Windows® XP and Windows® 7 operating systems
- Multi-tasking: Simultaneous measurement and evaluation
- Multi-moduling: Operation of several pieces of equipment via a single PC
- Combined analysis: Comparison and/or evaluation of TMA, DMA, DEA, DIL, DSC, TGA and STA measurements in a single presentation
- Labeling: Text elements can be entered and positioned freely
- Calculation of the 1st and 2nd derivative
- Export of graphics and data
- Selectable colors and line types
- Saving and recovery of the analysis status
- Macro-recorder (optional)
- Context-based help system
- Temperature calibration
- Software developed by ISO-certified enterprise of the NETZSCH Group

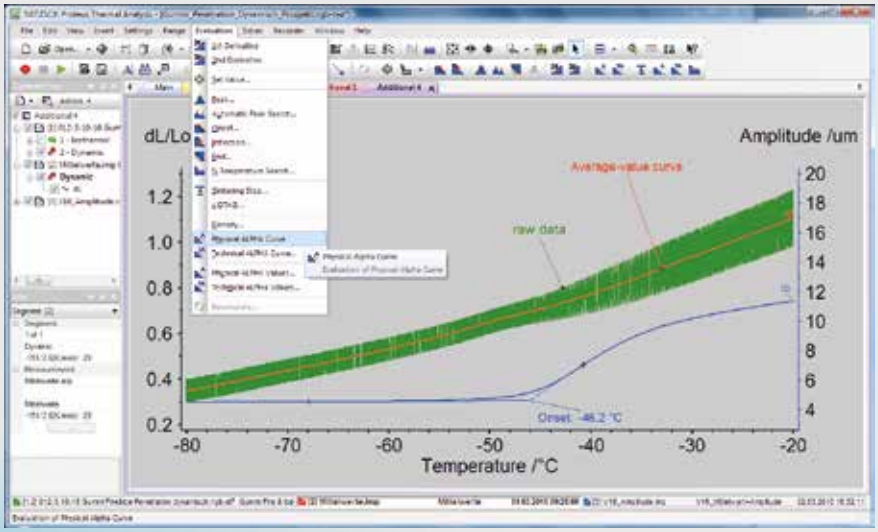
NETZSCH

Features Specific to TMA:

- Various correction possibilities for taking the expansion behavior of the system itself into account, as well as offset and sample length correction
- Automatic adjustment of sign for force and displacement signal to the measurement configuration being used
- Routines for the determination of characteristic temperatures such as onset, peak and final temperatures
- Evaluation of glass transitions and softening points in accordance with DIN
- Automatic shut-off at softening point
- Expansion coefficients: Calculation and presentation of the technical and physical expansion coefficients
- Automatic sintering step evaluation
- Measurements with variable force: Determination of visco-elastic properties

Extensions

- c-*DTA*® function: Evaluation of the temperature signal for endothermic or exothermic effects
- *Thermokinetics*: Extensive characterization and optimization of sintering processes
- Density Software: Determination of changes in volume and density until the liquid state
- *Peak Separation*: The separation of overlapping effects
- Rate-Controlled Sintering (RCS) Software: Sintering with a constant shrinkage rate (optional)

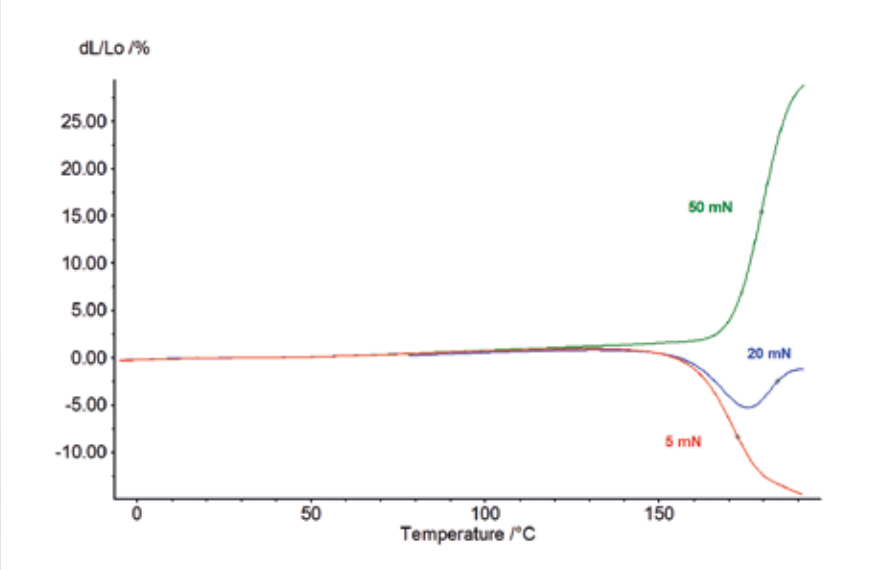


Meaningful Material Characterization in the Low-Temperature Fields

Application Examples

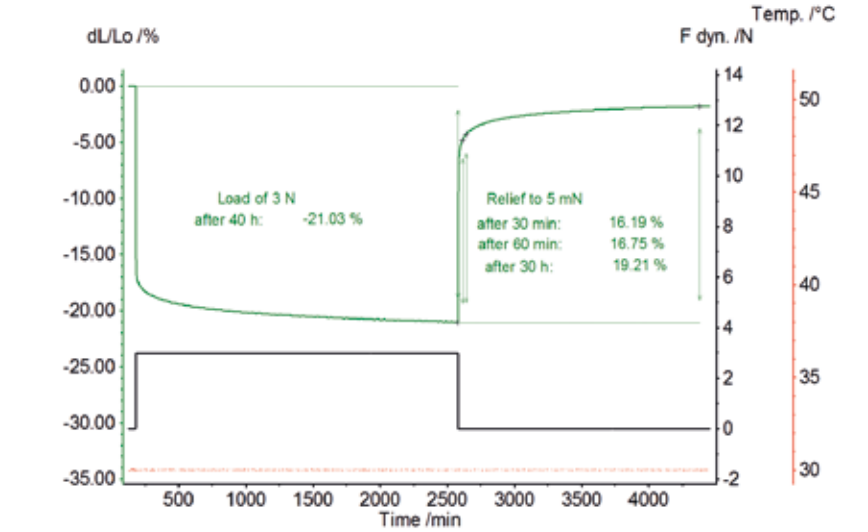
Tension Test on a Polymer Film

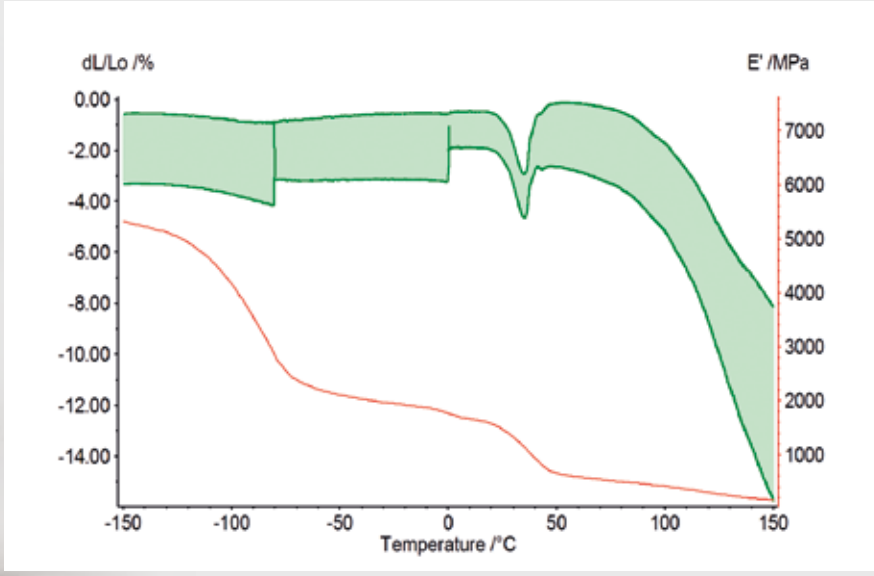
Orientation effects, stretch conditions and shrinkage are measured under load for films.
In this example, the expansion and contraction behavior of a 40-µm thick polycarbonate film was tested under tensile load. The results varied considerably depending on the load. Under low amounts of force (5 mN), the film contracted at higher temperatures; however, it expanded if greater force (50 mN) was applied.



Behavior of Polymers under Pressure

The extent to which the elastic properties of a seal remain intact after being subjected to a constant load of longer duration is very important.
To test this, an elastomer seal was loaded with a force of 3 N and then relieved to 5 mN. Following a 40-hour load time, 21% compression was observed. After a 30-minute relief period, the compression had reversed by 16.2%; after 60 min, by 16.8%. The visco-elastic properties of the elastomer were such that the sample did not return to its original length even after 30 hours.





Visco-Elastic Properties of Polytetrafluoroethylene

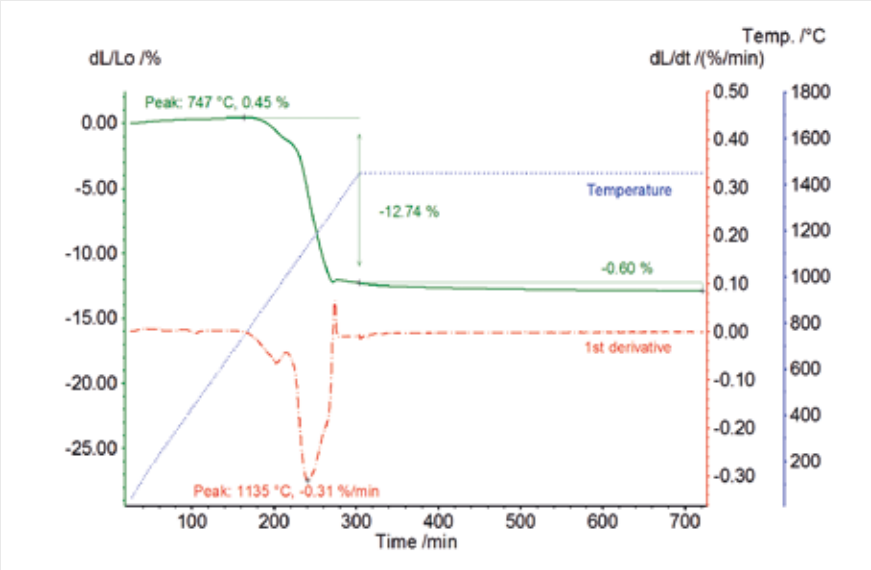
The combination of a 3-point bending sample holder and a modulated sample force allows the visco-elastic properties of a material to be determined.
A PTFE bar was measured between -150°C and 150°C under (rectangular) modulated force (fixed static force of 0.2 N and three different dynamic forces). Based on the expansion data and the sample geometry, the storage modulus E´ can be calculated as a function of temperature.
The three typical PTFE transformations are clearly visible at around -100°C (ß transition), between 0°C and 50°C (crystal to condis-crystal transformation) and above 100°C (glass transition).

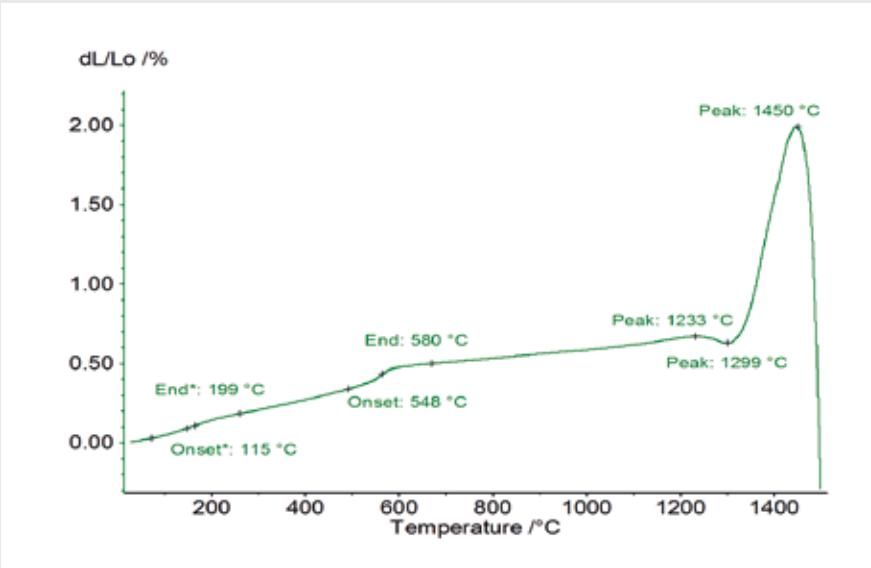
Meaningful Material Characterization in the High-Temperature Fields

Application Examples

Sintering of Aluminum Titanate

One use of aluminum titanate is as a carrier material for catalytic converters in the automobile industry.
Shown here is the measurement of an aluminum titanate green body in the temperature range from RT to 1450°C with a subsequent isothermal line at 1450°C of 7 hours. During heating, a shrinkage of 12.7% is observed. Sintering takes place in two steps with a maximum sintering rate of 0.31%/min. In the subsequent isothermal phase, further shrinkage of 0.6% occurs.
To optimize the sintering process, an RCS measurement (rate-controlled sintering; optionally available) can additionally be carried out.



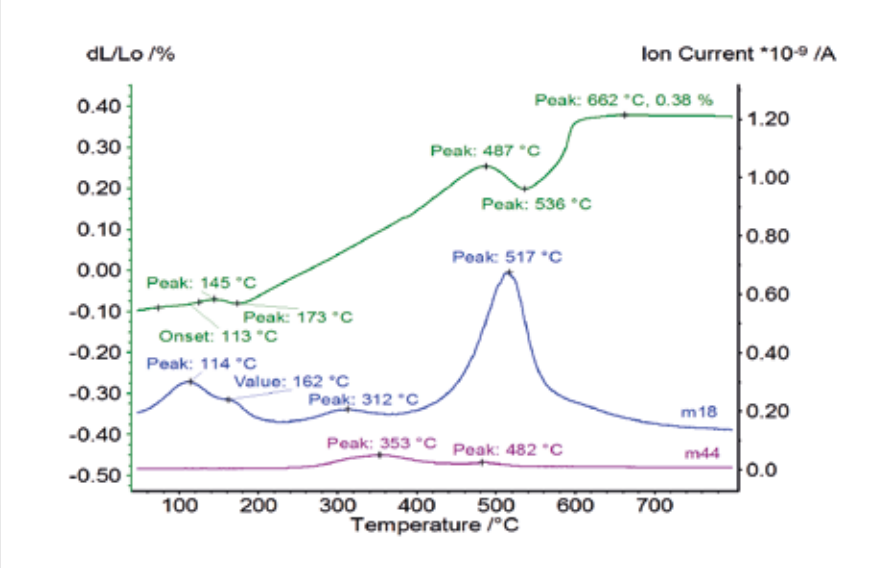


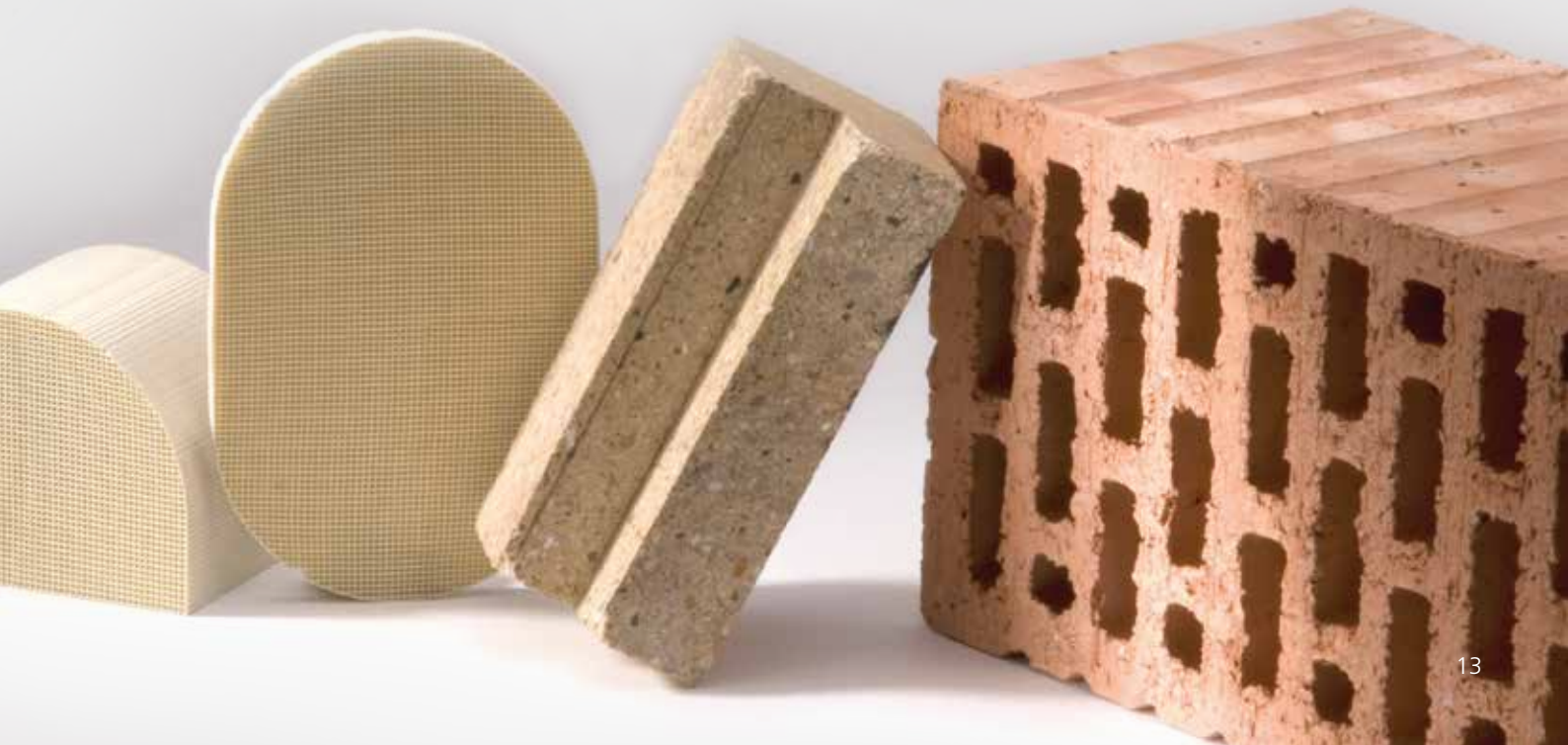
Refractory Materials

The life span and efficiency of any technical furnace can be greatly improved with an appropriate configuration, along with a high-quality interior lining made of refractory materials. An important criterion in assessing such materials is thermal expansion.
Shown in the example is the thermal behavior of a typical coarse-grained refractory material. At the beginning of the measurement, the α-ß transformation of the tridymite is first observed, followed by the α-ß transformation of the free quartz between 548°C and 580°C. After another transformation between 1233°C and 1299°C, the material begins to soften at 1450°C.

Measurement with EGA (Evolved Gas Analysis)

A clay sample (powder) was tested in air using a TMA-MS (*Aëolos*®) coupling in the temperature range from RT to 800°C.
At the beginning of the measurement, the adsorptively bound water and the interlayer water are released (shrinkage of 0.01%). Above 300°C, the sample's organic constituents burn up (release of water, m18, and CO_2, m44). There is no visible influence on the expansion curve here, due to the very low proportion. Between 487°C and 536°C, dehydroxylation of the sample's clay mineral content takes place.
Associated with that is a sample shrinkage of 0.05%.





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
- Calibration service
- IQ / OQ / PQ
- 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 (solids, powders and liquids).

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.

For production problems, we can work with you to analyze concerns and develop solutions. The minimal investment in our testing and services will reward you with reduced down time and reject rates, helping you optimize your processes across the board.

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The NETZSCH Group is a mid-sized, family-owned German company engaging in the manufacture of machinery and instrumentation with worldwide production, sales, and service branches.

The three Business Units – Analyzing & Testing, Grinding & Dispersing and Pumps & Systems – provide tailored solutions for highest-level needs. Over 3,000 employees at 163 sales and production centers in 28 countries across the globe guarantee that expert service is never far from our customers.

When it comes to Thermal Analysis, Adiabatic Reaction Calorimetry and the determination of Thermophysical Properties, 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.

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