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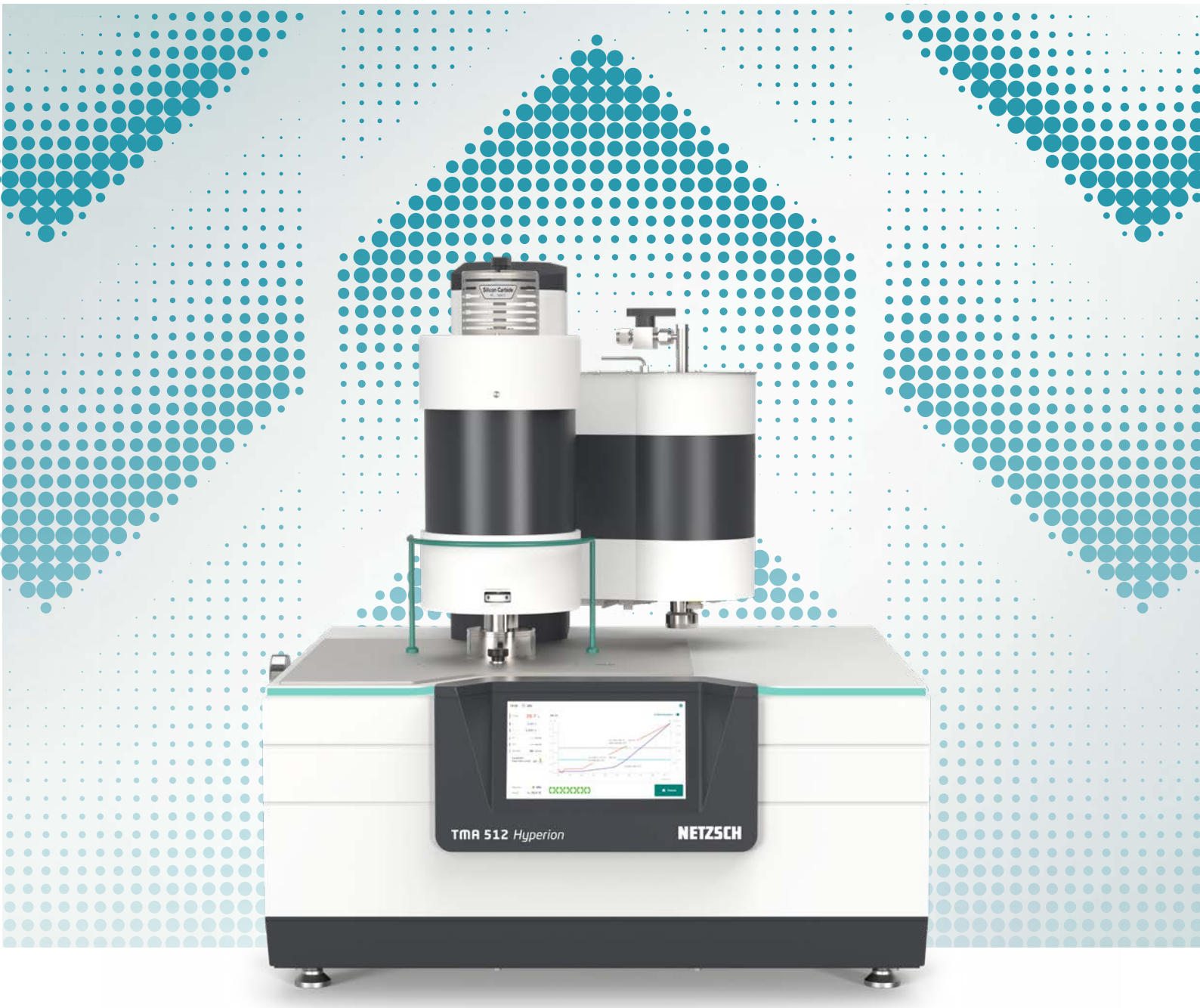


Thermomechanical Analysis – TMA 512 *Hyperion*® Series

Method, Technique and Applications

Analyzing & Testing

Thermomechanical Analysis (TMA)

For Research and Development, Simulation Input and Quality Control

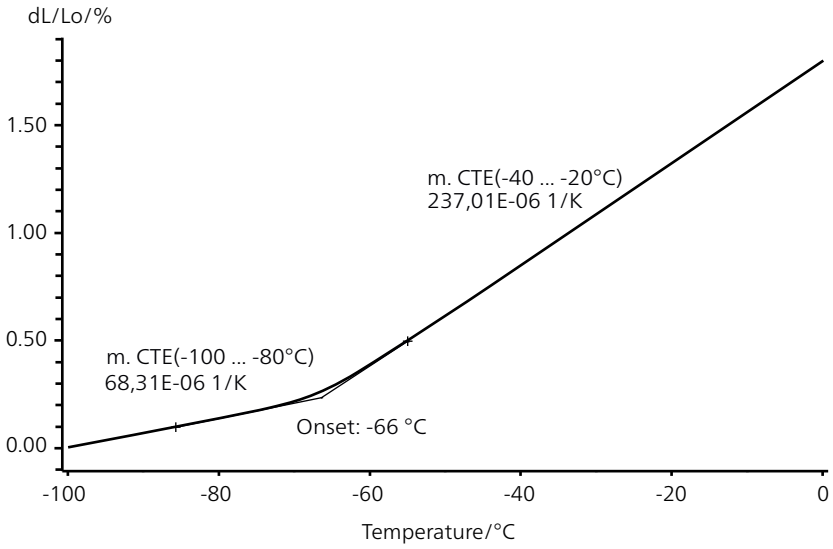
TMA Analysis Results

- Linear thermal expansion
- Coefficient of thermal expansion
- Glass transition temperatures
- Delamination
- Isostrain
- Creep
- Relaxation
- Stress/strain curve
- Phase transition temperatures
- Sintering temperatures
- Shrinkage steps
- Sintering kinetics
- Softening points
- Density changes
- Volumetric expansion

Thermomechanical analysis (TMA) is a technique for determining the dimensional changes in solids, liquids or pasty materials as a function of temperature and/or time under a defined mechanical force (DIN 51005, ASTM E 831, ASTM D696, ASTM D3386, ISO 11359 – Parts 1 to 3). It is closely related to dilatometry, which determines the length change of specimens under negligible load (DIN 51045).

Many materials undergo changes in their thermomechanical properties when heated or cooled. Phase changes, sintering steps or softening, for example, can occur in addition to thermal expansion. TMA measurements can be performed in different modes, e.g., deformation, compression, penetration, tension or bending.

The application range of TMA is extensive, encompassing all aspects of research and development as well as quality control. The materials analyzed are typically in the fields of plastics and elastomers, thermosets, composite materials, adhesives, films, and fibers. However, ceramics, glass, and metals may also be investigated.



TMA measurement in expansion mode on an elastomer sample (NR50): Quartz glass sample holder; 2-mm sample thickness; heating rate of 5 K/min; helium atmosphere.

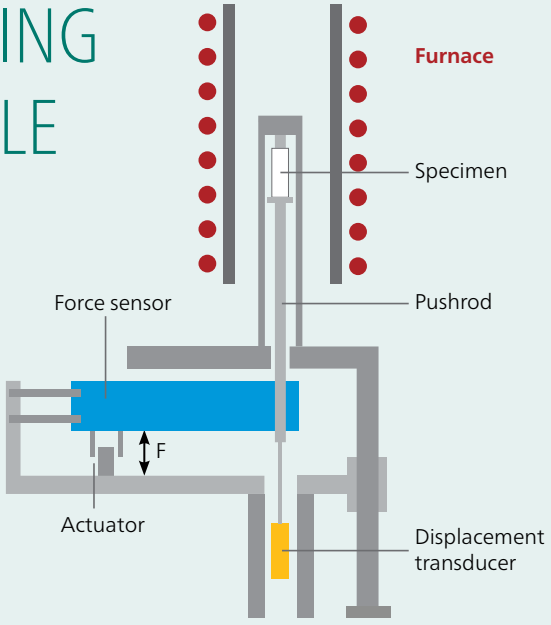
Thermal Expansion

Linear thermal expansion shows how much a material will shrink or expand during processing, whether dissimilar materials can be joined, where the phase change occurs, and where the CTE changes.

This figure shows the thermal expansion of an NR50 elastomer specimen, between -100°C and 0°C. The glass transition temperature (T_g) was determined to be -66°C. This marks the reversible transition from a hard, relatively brittle state to a softer, rubber-like state.

TMA – THE METHOD PRECISELY DETERMINES DIMENSIONAL CHANGES

OPERATING PRINCIPLE



Irrespective of the type of deformation selected (expansion, compression, penetration, tension or bending), every length change in the specimen is communicated to a highly sensitive inductive displacement transducer (LVDT) via a pushrod and transformed into a digital signal.

The pushrod and corresponding fused silica sample holders can be quickly and easily exchanged in order to optimize the system for the respective application.

TMA 512 *Hyperion*® *Select* and *Supreme*

Gaining Key Insights into Product Performance and Processing Behavior

Select

- -150°C to 1500°C, optional 1600°C
- Furnaces: Intracooler, SiC, Steel
- Force Range: 0.001 N to 3 N
- Software: *AutoEvaluation*, StrainControl

Detect Even the Slightest Dimensional Changes

The LVDT constitutes the centerpiece of the NETZSCH TMA 512 *Hyperion*®. The technology behind it is tried-and-true: Even the slightest of length changes, into the nanometer range (digital resolution of 0.125 nm), can be detected and measured.

Innovative Display and LED Strip for Easy Monitoring

The TMA 512 *Hyperion*® series offers an LED light bar that visually indicates the instrument status from a distance. The touch display provides a convenient way to monitor and start a measurement prepared in the *Proteus*® software.

Compatibility with Other NETZSCH Instruments

Furnaces for the TMA 512 *Hyperion*® are compatible with other NETZSCH high-temperature thermal analysis instruments and vice versa (e.g., STA 509 *Jupiter*®, DSC 500 *Pegasus*®).

Furnace Type	Temperature Range
Steel	-150°C to 1000°C
Intracooler compatible	-70°C to 450°C
Silicon carbide (SiC)	RT to 1500°C/1600°C
Copper*	-150°C to 500°C (under humid atmosphere: 0° to 100°C)
Water-vapor*	RT to 1250°C

* *Supreme* only

Supreme

- -150°C to 1600°C
- Furnaces: Intracooler, Steel, Copper, SiC, Water-Vapor
- Force Range: 0.001 N to 4 N
- Software: c-*DTA*, *Identify*, *AutoEvaluation*, Temperature- and Force Modulation, StrainControl

Cooling System	Atmospheres
Air, passive (fan)/ compressed air, LN_2	Inert, oxidizing, reducing, vacuum
Intracooler	Inert, oxidizing, reducing, vacuum
Air, passive (fan)	Inert, oxidizing, reducing, vacuum, hydrogen
Air, passive (fan)/ compressed air, LN_2	Inert, oxidizing, reducing, vacuum, humidity
Air, passive (fan)	Inert, oxidizing, reducing, vacuum, water vapor

Flexible Temperature Range and Atmospheres

Easily interchangeable furnaces adapt the instrument for various temperature ranges and enable measurements in different atmospheres. In the dual furnace version, switching to the second furnace is quick; it takes only a few minutes. The TMA 512 *Hyperion*® *Supreme* covers the entire temperature range from -150°C to 1600°C and can be used for measurements under various gases, humidity and even water vapor.

Controlled Atmospheres in a Vacuum-Tight TMA System

The TMA 512 *Hyperion*® versions are equipped with vacuum-tight connections, enabling the execution of measurements in high-purity atmospheres or under vacuum conditions. The TMA 512 *Hyperion*® *Supreme* includes one mass flow controller (MFC), which is upgradable to up to four gases. This provides optimum flexibility in gas control and the ability to easily change purge gases, atmospheres and gas flow rates.

H_2Secure Concept by NETZSCH

The TMA 512 *Hyperion*® in connection with the SiC furnace and the *H_2Secure* box offers a comprehensive solution for testing in environments with varying hydrogen concentrations up to 100%, ensuring maximum safety through a robust safety protocol (see page 9).

Measurement Update in Passing – LED Status Bar

The TMA 512 *Hyperion*® series provides an LED light bar that allows you to check the status of your instrument as you walk by, with different colors representing different statuses. It is reassuring to see from afar, without having to log into your PC, that your measurement is running smoothly and to be able to read instrument status notifications such as:

- Instrument is ready
- Measurement is running
- Measurement progress
- Heating/Cooling to setpoint
- User interaction needed
- A problem occurred

Improving Your Productivity and Workflow with Innovative User Interface

The integrated color display allows you to start a measurement that was previously prepared in the NETZSCH *Proteus*® software. Just touch the prepared measurement button on the display and you will be informed about the setup of the measurement. This moves the final check before you start a new measurement directly onto the instrument. The color touch display offers the following conveniences:

- Start measurements with the touch of a finger
- Check recently finished measurements
- See the progress of your measurement and time remaining
- Check and change gases, idle state
- Get an immediate overview of the evaluated measurement
- Start *AutoVac*
- Select measurement mode and sample holder
- Tare instrument directly on the display
- Sample length detection
- Change static force

Accessories

Wide Selection of Sample Holders Make the TMA 512 *Hyperion*® Stand Out

Prepared for Future Applications – Large Variety of Easily Exchangeable Sample Holders

Depending upon the question at hand and the geometry of the specimen, the operator has a variety of sample holders to choose from. Holding devices for expansion, penetration, and tension measurements are available, as well as pushrods and supports for analyses in 3-point bending. Accessories for the temperature range up to 1100°C are made of fused silica. For the high-temperature range, alumina varieties are available.

Testing Liquids, Molten Salts or Metals

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 immersion swelling experiments are also available, as are containers for molten salt experiments.

Sample containers of alumina, sapphire, fused silica and graphite for measurements on powders, pastes and liquids

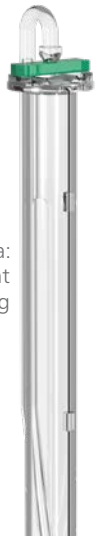
Special sample container for molten salts

Type	Specimen Dimension (max.)
Expansion/Compression, Penetration	Ø: 8 mm, length: 30 mm, Ø: 10 mm, length: 30 mm Ø: 12 mm, length: 30 mm
3-Point Bending	Length: 24 mm, width: 5 mm, free bending length: 5, 10, 20 mm
Tension	Length: 30 mm, width: 6/8 mm, thickness: 1 mm

Fused silica: expansion/compression – pushrod with flat tip, Ø 4 mm

Fused silica: penetration – pushrod with flat tip, Ø 1 mm

Fused silica: tension



Fused silica: 3-point bending

Fused silica: expansion/compression for Intracooler furnace

Alumina: expansion

Measurements in Humid Atmosphere

Simulation of Environmental Influences

For TMA measurements in humid atmospheres, there are two furnaces available.

The **water-vapor** furnace covers a temperature range from room temperature to 1250°C. The furnace can be connected to a humidity generator or to a water-vapor generator, which produces steam by evaporating water.

The **copper** furnace can be used for conventional TMA measurements from -150°C to 500°C. It can be conveniently connected to a humidity generator, allowing for in-situ drying up to 500°C and a controlled humidity environment between 0°C and 100°C. For your convenience, a humidity calculator is integrated into the TMA *Proteus*® software.

Humidity Generator

Copper or Water-Vapor Furnace

- Defined relative humidity by mixing wet and dry gas flow
- Maximum dew point of 80°C, corresponding to 47% molar concentration
- Minimum 5% relative humidity at 20°C, corresponding to 0.1% molar concentration (or dry)
- Programmable humidity ramps/steps
- Easy refill, also while operating
- Integrated software for humidity measurements

Water-Vapor Generator

Water-Vapor Furnace

- Steam by evaporating liquid water
- Maximum 100% molar concentration
- Possible dilution by inert gas
- Minimum 5% molar concentration (or dry)
- Gas-tight water tank

Water-vapor generator

Humidity generator connected to the TMA 512 *Hyperion*®

Hydrogen Research
Using H_2Secure

The *H_2Secure* concept developed by NETZSCH features a complete solution for conducting tests in environments with varying hydrogen concentrations while providing a maximum in safety. This flexibility is achieved through a comprehensive safety protocol embedded in the system, enabling seamless performance of complex oxidation-reduction cycles and precise analysis of reaction kinetics and material behavior under different conditions.

Setup

1 Hydrogen Gas Supply
Hydrogen can be supplied from an H_2 generator or H_2 cylinder and is connected to the special H_2 gas inlet on the rear of the TMA with integrated safety valves.

2 Optimized Gas Path
This provides a precise concentration of gas, e.g., up to 100% hydrogen, while maintaining a protective gas atmosphere.

3 Continuous Monitoring of Gas Concentrations
TMA exhaust gas flow is monitored for H_2 and O_2 concentrations.

4 *H_2Secure* Box
Central communication box to control signals and allow or restrict gas flows depending on the H_2 or O_2 limits defined.

Proteus®

Force Modulation to Gain Advanced Material Insights

Simultaneous Measurement of Force and Displacement Signal

The force acting on the specimen is generated electromagnetically. This ensures a quick response time for experiments with a changing load. A highly sensitive force sensor (digital resolution < 0.01 mN, max. force ±4 N*) continuously measures the force exerted via the pushrod and automatically readjusts it. This sets the NETZSCH TMA 512 *Hyperion*® apart from other instruments, which only use preset values.

From Sensitive to Stiff Materials

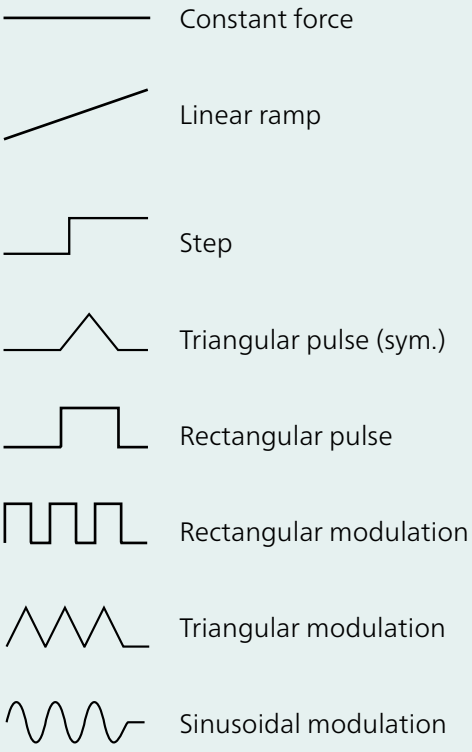
The electronic control system allows users to set the force value in the mN range. This enables testing even on sensitive materials such as thin fibers or films. For bigger geometries, a force load up to 4 N* can be applied using the TMA 512 *Hyperion*® *Supreme*. The force being exerted upon the specimen can be altered via the software. This facilitates the execution of tests, such as creep, with minimal complexity.

Determination of Visco-Elastic Properties like Relaxation, Creep and Stress/Strain

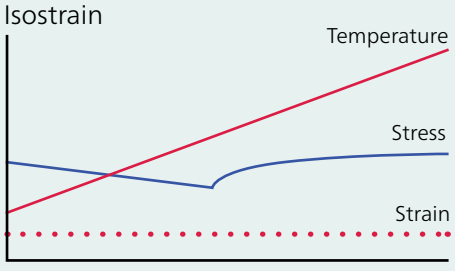
The TMA 512 *Hyperion*® offers the ability to change the dL displacement and measure the corresponding force. This can be used, for example, in a stress-relaxation test where a sample is stretched by a specific amount at a defined temperature. During the test, the deformation is kept constant and the progression of the force is recorded. The force continuously decreases as a result of material relaxation. The stress relaxation is ultimately defined by the residual stress measured after a defined exposure period. In the stress-time diagram, it is then possible to read off both the stress-relaxation behavior and the values for the relaxation rate and time.

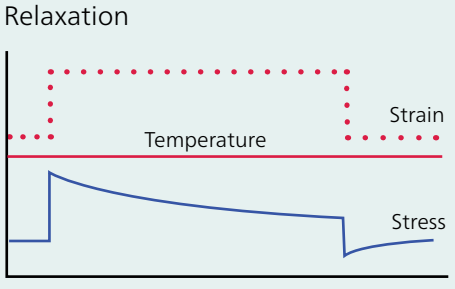
* *Supreme* only

Force Control



Displacement Control





Software Features to Enhance Your Results

Density Determination

This software option allows for determination of the density and volumetric change of specimens based on the measured thermal expansion. It can be applied for solids, liquids or transitions from solid to liquid, among others.

Rate-Controlled Sintering (RCS)

RCS allows for control of the sintering rate during a TMA measurement by delivering precise, non-linear heating profiles that optimize your materials' microstructure and densification. The temperature program of the furnace is controlled so as to achieve the predefined sintering rate for the specimen. Depending on the selected RCS mode, the furnace may no longer be heated at a constant rate: The heating process may be stopped/started or continuously adapted depending on the sintering behavior of the specimen. The measured temperature profile can then be used for optimizing the production process.

Temperature-Modulated TMA

Temperature modulation in TMA separates reversible effects, such as glass transitions, from non-reversible processes, such as relaxation or evaporation. For sintering studies, it is possible to separate shrinkage from expansion. For temperature-modulated TMA measurements, a sample is subjected to a superposition of a linear and a periodic temperature program, where a constant heating rate is overlapped by further modulated temperature control. The evaluation software allows for the determination of the:

- Total dL
- Reversing and non-reversing dL
- Total CTE
- Reversing and non-reversing CTE
- Amplitude and phase

Proteus® also offers graphic display of the results curve in multi-window technique along with the ability to export graphs and print out or export data as ASCII files.

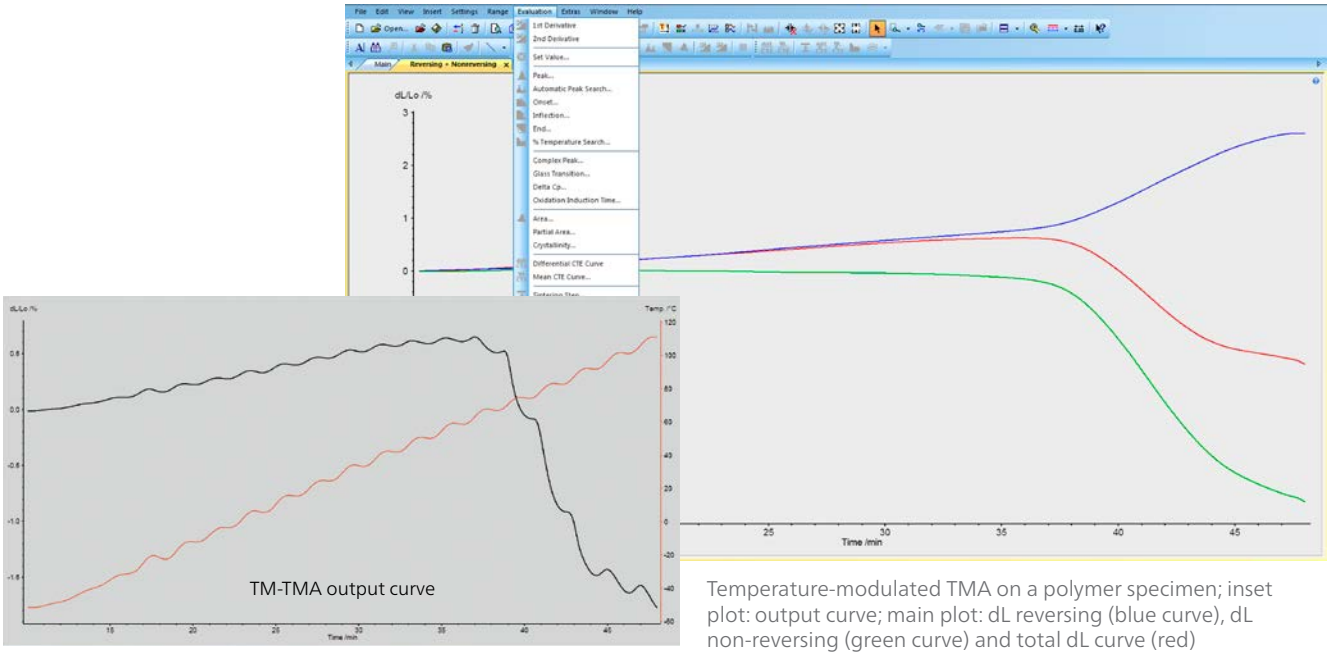
Patented* c-*DTA*®

The c-*DTA*® signal yields the opportunity for simultaneous analysis of length changes and endothermal/exothermal effects. It can also be used for temperature calibration.

* DE102013100686

Kinetics Neo – Process Optimization by Prediction

In the face of rising energy costs and increasing demands for high-quality ceramic products, optimizing production processes has become more critical than ever. The Kinetics Neo software offers a cutting-edge solution that combines precision and efficiency to revolutionize ceramic manufacturing, saving both time and resources without compromising quality.



Temperature-modulated TMA on a polymer specimen; inset plot: output curve; main plot: dL reversing (blue curve), dL non-reversing (green curve) and total dL curve (red)

Proteus®

It's a Breeze to Get Reliable Results – Just One Click!

Input Assistant for a Fast Measurement Start and Method-Based Automatic Evaluation

The *Proteus*® software facilitates the application of methods derived from previously executed measurement files to new measurements with a single mouse click, thereby streamlining the process. For instance, the evaluation steps for a reference test run can be stored in a method and automatically applied to a sample measurement even after its termination.

Furthermore, the software has the capacity to identify any results that deviate from the user-defined quality criteria.

AutoEvaluation – The One-Click Evaluation

AutoEvaluation is an intelligent software function made by NETZSCH. Its evaluation of thermoanalytical measurement curves is an autonomous process that functions independently of predefined macros. This is an immense support and time saver.

AutoEvaluation offers special functions for the evaluation of various materials. When measuring polymers, *AutoEvaluation* will automatically find the onset of the glass transition as well as the peaks of softening points and evaluate these with just one click.

Identify – Identification and Classification of TMA Curves

The *Identify* database offers a state-of-the-art means of verifying materials; it allows for comparison of a given curve with other individual curves (e.g., groups of curves in quality control) or with literature data from selected libraries. Any libraries or classes created by the user can be edited or expanded within *Identify*.

The standard libraries – comprising over 1100 entries – include measurements and literature data for DSC, c_p, TGA, and DIL/TMA from the application fields of polymers, organics, foods, pharmaceuticals, metals/alloys, ceramics and inorganics as well as the chemical elements.

Database entries can be filtered by a variety of criteria, and measurement curves – even those of different types – can be overlapped for comparison purposes.

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Proteus® Search Engine

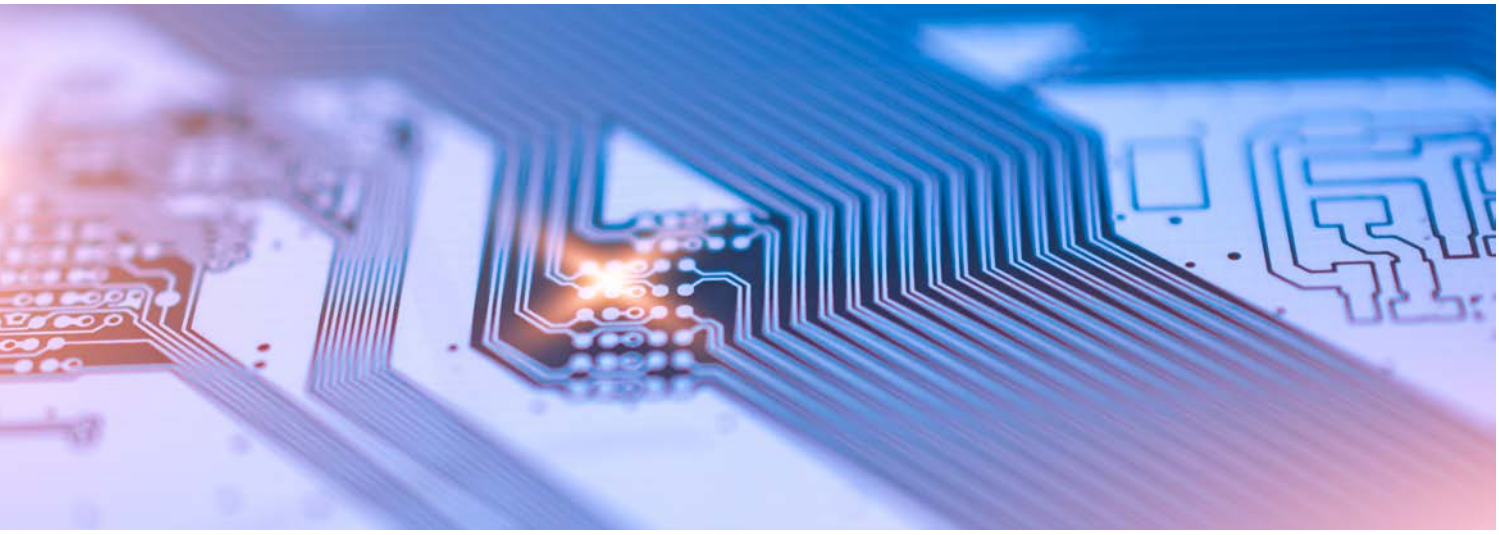
- Efficiently manage data
- Directly access and sort data by criteria
- Quickly view measurement and analysis previews without opening files
- Retrieve data quickly and easily
- Search by such criteria as instrument name, method, operator, file and signal type, date, measurement conditions or evaluated effects

At a Glance – Highlights of the TMA *Proteus*® Software

	Select	*Supreme*
Report Generator	■	■
Softening point detection	■	■
Automatic sample length detection	■	■
Displacement control	■	■
Force adjustment/segment	■	■
Eco Mode	■	■
AutoEvaluation	■	■
Identify	□	■
c-*DTA*® (caloric effects or calibration)	□	■
Force modulation	□	■
Temperature modulation	□	■
Density determination	□	□
Rate-Controlled Sintering (RCS)	□	□
Kinetics Neo	□	□
Proteus® *Search Engine*	□	□

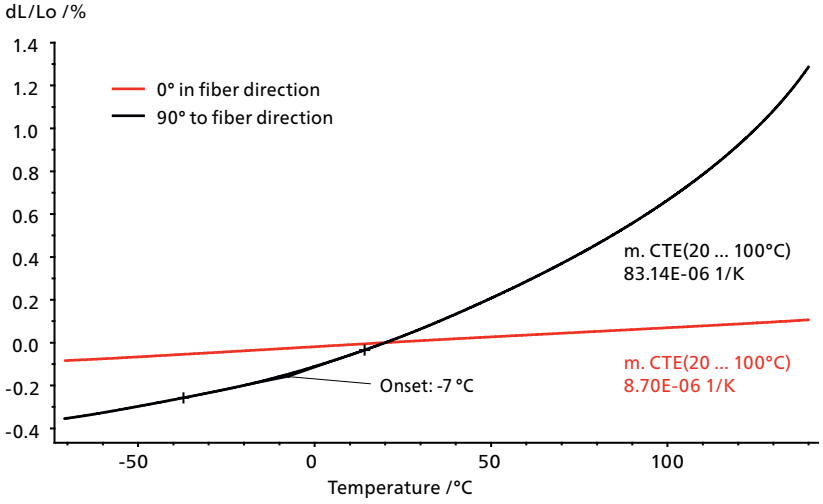
■ Included in standard configuration
□ Optional

APPLICATIONS



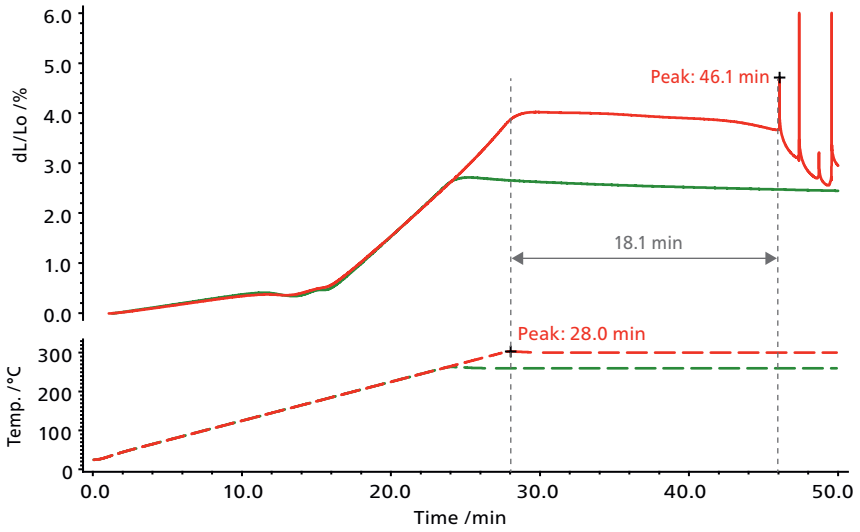
Anisotropic Behavior in Composite Systems

The glass transition temperature (T_g) in a thermoset and thermoplastic matrix composite can be determined by means of TMA. Detection of the onset of the glass transition is a reliable method that determines the upper application limit of a thermoset material. For thermoplastic matrix composites such as PP-GF, T_g indicates the temperature area where the material starts to soften. The degree of anisotropy of the filler and the filler orientation both have great impact on the linear coefficient of thermal expansion (CTE). For example, uni-directional composites exhibit the CTE of the fibers in the fiber direction. They also exhibit a mixture of the matrix and fiber CTE as a function of the fiber volume content perpendicular to the fiber direction. The red curve depicts the measurement in the fiber direction; the CTE is dominated by the low thermal expansion of glass. The specimen that is measured 90° to the fiber direction is dominated by the polypropylene matrix and shows a much higher CTE. Therefore, it is only possible to observe the T_g of PP measured in this direction.



Composite material, measured in two directions; specimen length: 25 mm, heating rate: 5 K/min from -70°C to 140°C, N_2 atmosphere, sample holder made of fused silica for expansion mode.

Specifying the Right Material for Functional Electronics



Determination of time to delamination on an FR4 composite circuit board. Specimen size 6.35 mm^2 as defined by IPC, dried for 2 hours pre-measurement at 105°C, heating rate 10 K/min, N_2 atmosphere, sample holder made of fused silica; isothermal segments at 260°C and 300°C.

Industry standards require the measurement of thermal expansion, glass transition and softening point under IPC standards [see IPC-TM-650 2.4.24.1 Time to Delamination (TMA Method)]. After the changeover to lead-free soldering processes in circuit board production, the melting temperature of soldering materials increased. This led to the delamination of printed circuit boards and assemblies due to the higher thermal load. Manufacturers had to react by changing to FR4 substrates with a higher T_g.

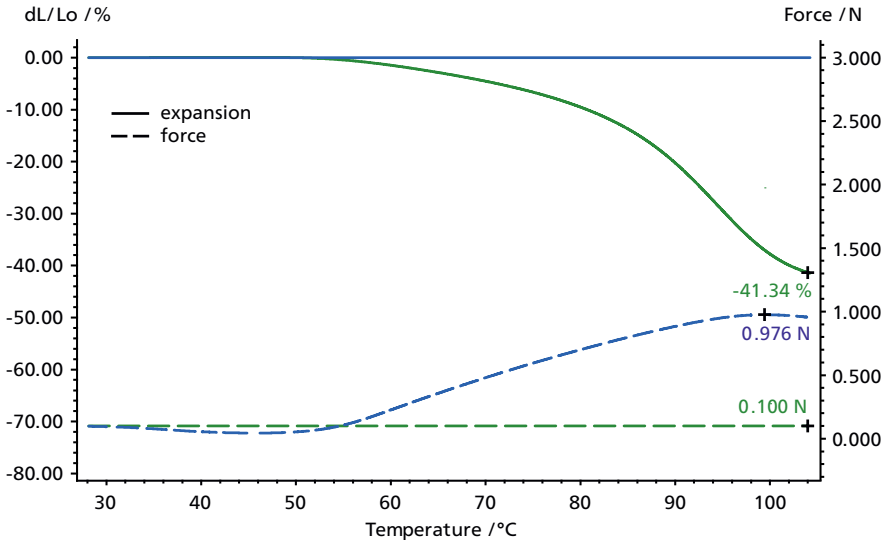
Nevertheless, even today most FR4 substrates are ordered with general material specifications and can vary in their material properties. At the glass transition event, the rate of expansion for the epoxy matrix increases, which can lead to delamination between the fibers and matrix and consequently to product failure.

The figure shows a measurement on an FR4 composite in which the time to delamination was recorded. Two measurements were conducted; one with an isothermal temperature of 260°C (green curve) and a second one with an isothermal temperature of 300°C (red curve). In the first measurement, no delamination effect was visible. In the second measurement, the time to delamination was recorded at 18.1 min after being held at an isothermal temperature of 300°C.

Thermal expansion is a major factor in the failure of electronic products.

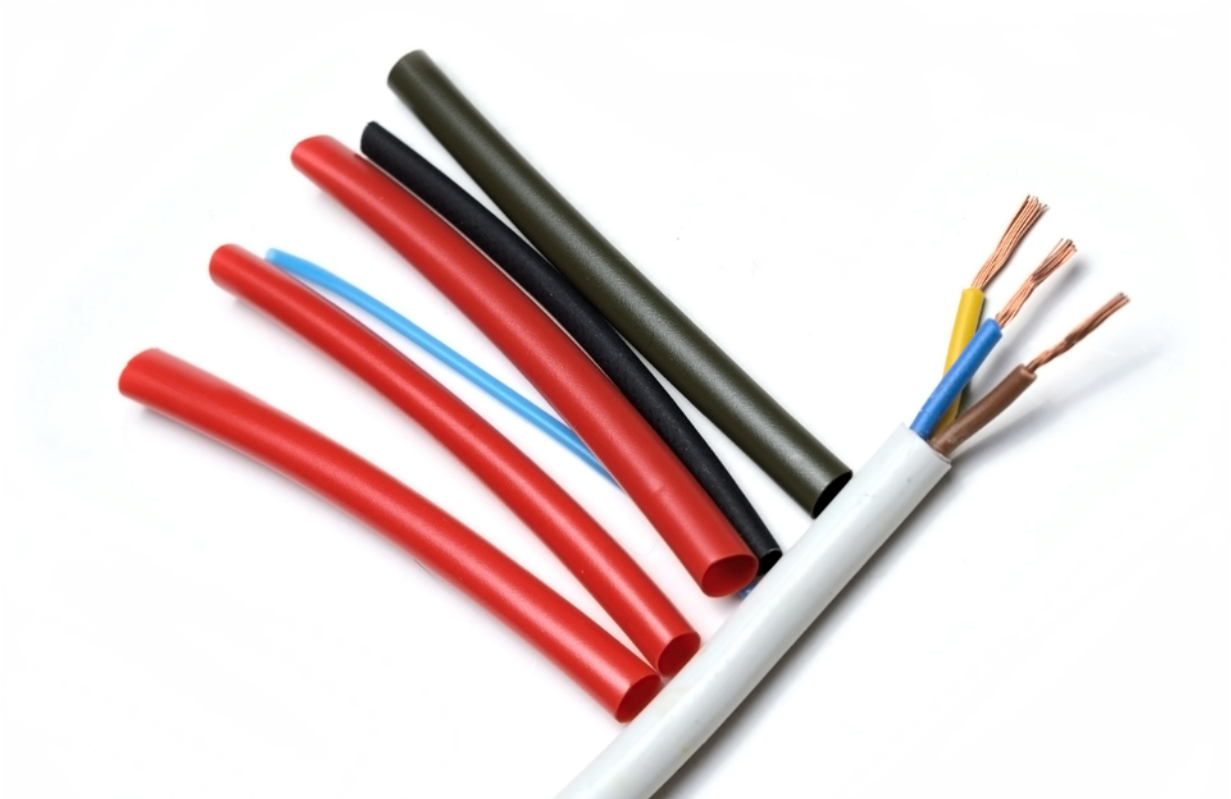
Typical de-coloration as a sign of beginning degradation: left: before measurement, middle: after being measured at 260°C, right: after being measured at 300°C. Neither of the measured specimens shows any visible delamination, although the TMA method is sensitive enough to detect it at 300°C.

Test on Heat-Shrink Tubing



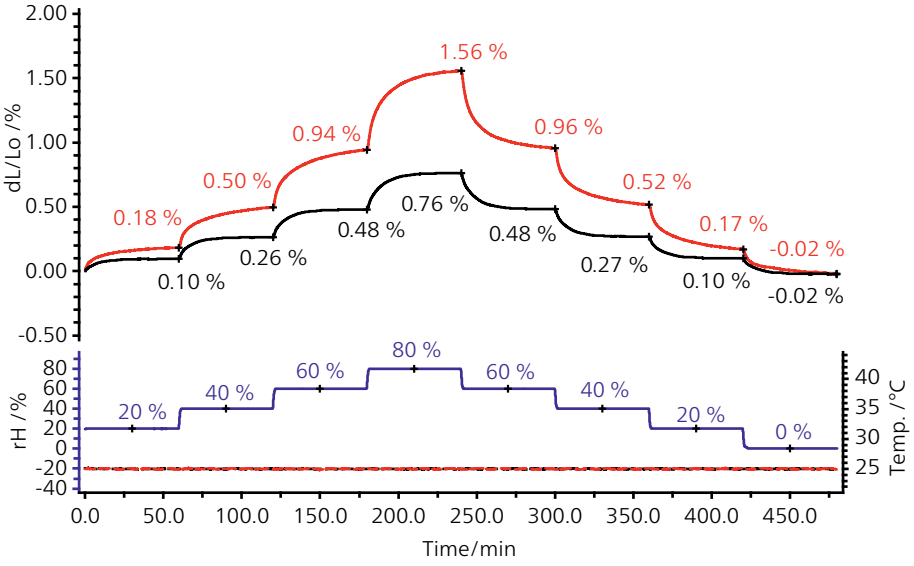
Measurement on a heat-shrink tubing under constant force (green curves) and under constant displacement (blue curves). Heating from RT to 120°C, atmosphere: N_2, sample length: 25 mm (blue) and 10 mm (green).

Heat-shrink tubing, also known as shrink sleeve, is used to repair and insulate wires and cables. After sliding the tubing onto the cable, a heat source is used to make it shrink and create a tight seal. Heat-shrink tubing, by its very nature, is stretchy and changes its shape. A TMA can help gather information about the temperature at which the material starts to shrink, how much it shrinks and with how much force. The plot shows the two samples measured. The first sample (green curves) starts to shrink at around 60°C with a shrinkage of 40% by the end of the measurement. In the second sample, the displacement was kept constant and the corresponding force measured. A maximum force of 0.976 N (blue curves) was recorded.



Defining Ideal Production and Application Conditions for Polymer Materials

Moisture Absorption in Biopolymers – Made Visible with TMA



TMA measurements on two biopolymers under defined humidity. Biopolymer 1 without starch (black curve) and biopolymer 2 with 20% thermoplastic starch, TPS (red curve).

Starch is a widely used, renewable raw material in biopolymer blends. Not only does it reduce costs, but it also specifically influences mechanical and ecological properties. However, due to their hydrophilic nature, starch-containing materials are sensitive to ambient humidity. Therefore, it is important to better understand how moisture absorption influences the length and volume changes of such materials.

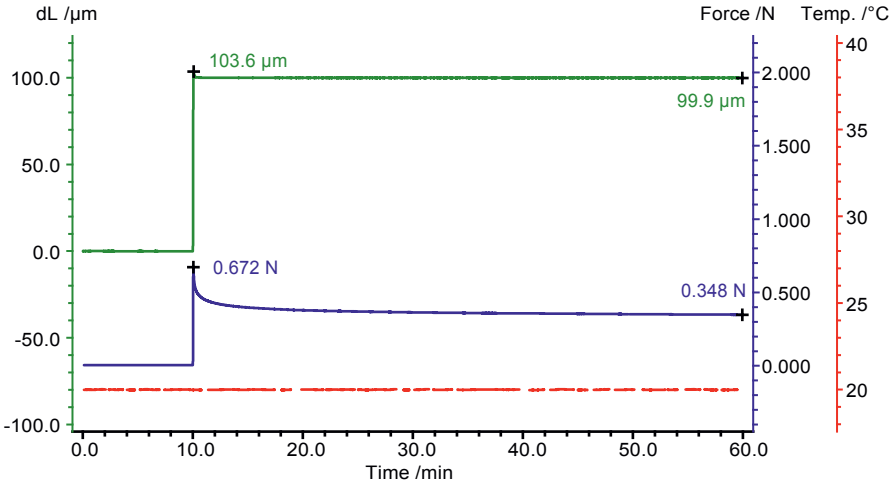
The TMA measurement shown analyzed the length change of two biopolymers – biopolymer 1 (without starch) and biopolymer 2 (with 20% thermoplastic starch, TPS) – over time under defined humidity conditions. Clearly, the addition of TPS leads to increased moisture absorption, resulting in higher swelling and length increase.

Specimen preparation is particularly challenging in such analyses. Biopolymers are usually brittle and difficult to handle when completely dry. Ideally, the measurement would start in a dry state. Therefore, a defined initial moisture content is crucial for reliable analysis, both for handling and for reproducible results.

Relaxation – An Important Material Property for Polymer Films

Plastic packaging must be flexible, lightweight, and strong. It should also be impermeable and easy to sterilize when necessary. The resulting property profile is determined by the material and process conditions. It is also important to understand the material's relaxation properties. This allows for predictions regarding its fatigue and wear resistance during use.

Shrink films apply a film's tendency to relax at elevated temperatures after having been pre-stretched during processing. Over time, shrink wrap tends to loosen due to creep and stress relaxation. In the test shown here, the sample was kept under constant strain and the progression of the tensile force was recorded.

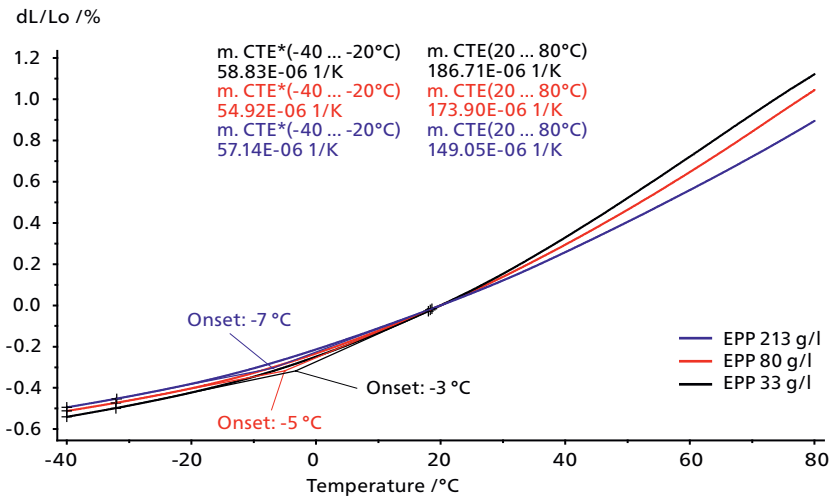


Relaxation measurement on an LDPE film, fused silica sample holder. Measured at room temperature under a nitrogen atmosphere; sample length: 10 mm; sample width: 5 mm.

Thermal Expansion of Insulation Material

Polymer foams are used where light weight is required along with an excellent strength-to-weight ratio, superior thermal and acoustical insulation properties, and good energy absorption behavior.

The use of expanded foams like EPS and EPP is on the rise in the building and automotive industries. Therefore, thermal expansion is an important quality criterion when choosing insulation material, since expansion behavior differs significantly among materials with different densities. The foam with the lowest density (black curve) shows the highest CTE while still having a similar T_g.

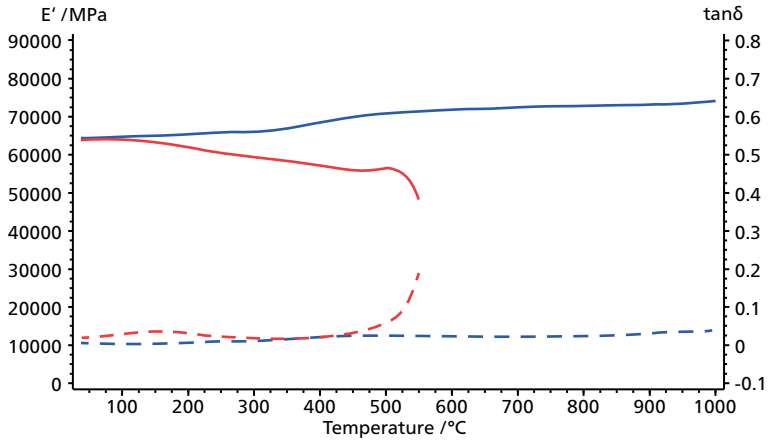


Expansion measurements on three different EPP foams with different densities. Specimen length: 20 mm, heating rate: 5 K/min from -40°C to 80°C under an N_2 atmosphere, expansion sample holder made of fused silica and 50 mN sample load.

Comparison of the Visco-Elastic Properties of Fused Silica and Flat Glass

These TMA measurements on fused silica and flat glass were carried out in 3-point bending at a heating rate of 5 K/min between room temperature and 1000°C and 550°C, respectively.

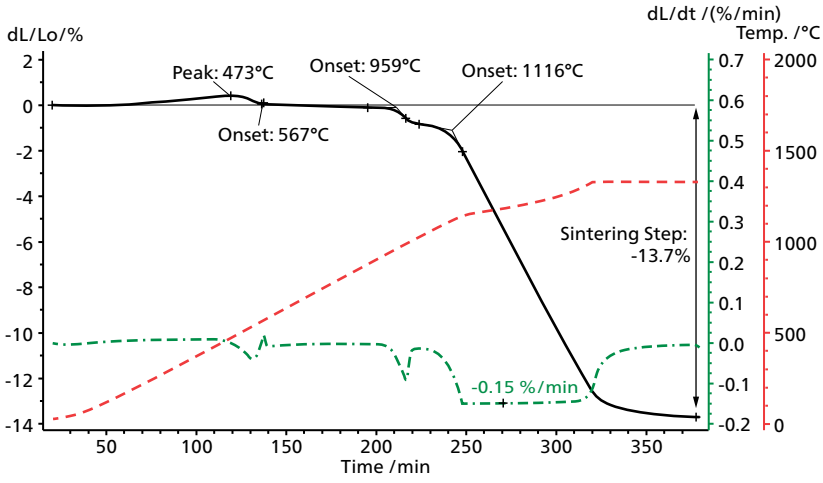
As expected for most materials, the modulus for flat glass (red curve) decreases with rising temperature until the material's softening point is reached at approx. 520°C, resulting in a sharp drop in stiffness accompanied by a rising tan δ (dotted red line). In contrast, fused silica (blue curve) exhibits increasing stiffness as temperature rises.



Visco-elastic behavior of fused silica (blue) and flat glass (red). Force modulation: 0.5 Hz, static force: 1.5 N, amplitude: 1.45 N, bending distance: 20 mm; specimen thickness: approx. 1 mm, specimen width: approx. 4.8 mm. The solid lines represent the E' modulus and dotted lines tan δ.

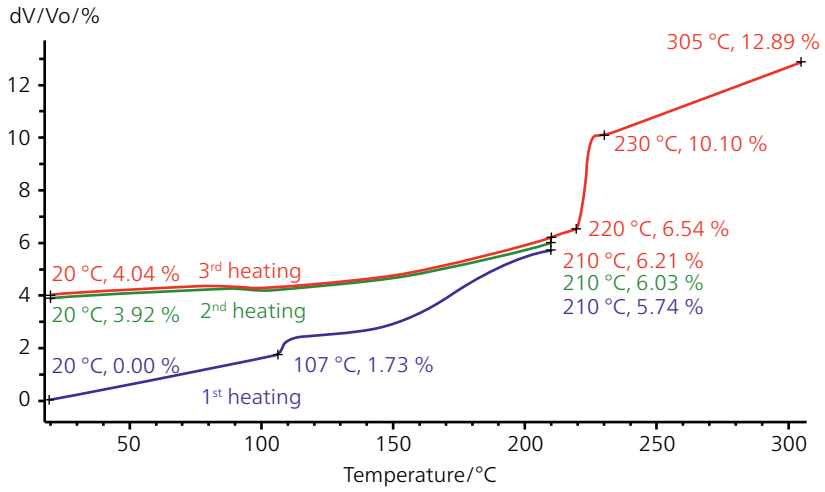
Densification of a Ceramic Green Body by Rate-Controlled Sintering

The sintering of a ceramic green body was here studied using rate-controlled sintering (RCS). The furnace temperature program is controlled to achieve the prede-fined sintering rate for the specimen. In this case, the RCS starts at 1040°C using the start/stop mode with a predefined sintering rate of 0.15%. In the length change curve (black), the dehydroxylation of the kaolinite at 473°C is overlapped by the quartz transition at 567°C (onset). At the onset temperature of 959°C, an additional phase transition takes place, which is confirmed by the peak at 215 min in the 1st derivative of the length change curve (green). At 250 min, sintering starts, with a constant expansion rate of 0.15%/min. The sintering step amounts to 13.7% (black curve).



The RCS measurement in expansion mode (Al_2O_3 sample carrier), SiC furnace between room temperature and 1350°C; sample length approx. 5.5 mm, Ø 6-7 mm, RT -1350°C at a heating rate of 5 K/min followed by an isothermal segment of 60 min; RCS start at 1040°C, start/stop mode, threshold 0.15%.

Molten Salts – Analysis of the Thermophysical Properties of an $NaNO_3$-KNO_3 Mixture with Metastable Phases



Temperature dependence of the volume change of a salt mixture of 50 mol% $NaNO_3$ - 50 mol% KNO_3 within three heating cycles [*]

Sample container for molten salts

Salt systems are primarily of interest for their use as heat transfer fluids or chemical reactants in key industrial sectors such as metallurgy, nuclear energy and solar energy. The $NaNO_3$-KNO_3 system has been the focus of extensive research due to its potential applications as a heat transfer fluid and a thermal energy storage (TES) material for concentrated solar power (CSP) plants. The SolarSalt mixture (consisting of 60% $NaNO_3$ and 40% KNO_3) is employed in the majority of CSP plants, due to its advantageous thermophysical characteristics.

In this study, the volume change of 50 mol% $NaNO_3$ - 50 mol% KNO_3 mixture (pressed at 20 kN for 24 h) was measured in a graphite container, specifically designed for testing molten salts. Measurements of molten salts are challenging due to their high corrosivity, creeping effect and possible vaporization.

Significant differences in phase-transition temperatures and volume changes were observed between the 1st and 2nd heating cycles (see figure above). In the 3rd heating cycle, melting of the mixture was observed at 220°C.

These findings demonstrate that TMA can be used to accurately determine the volume change of both the solid and liquid phases. In addition, TMA can be used for in-situ observations of the sintering behavior of pressed salt pellets [*].

* More details of this study and a comparison of this material measured with other analytical methods (LFA, DSC, XRD) can be found in the open access article published under the terms of the Creative Commons CC-BY license in the Journal of Materials Research and Technology, Volume 36, May-June 2025 (https://doi.org/10.1016/j.jmrt.2025.03.128): "Comprehensive analysis of thermophysical properties of the $NaNO_3$-KNO_3 mixture with metastable phases", D. Sergeev, G. Nénert, D. Rapp, F. Beckstein, M. Schöneich, M. Müller, J. Gertenbach.

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TMA 512 *Hyperion*®		
	Select	*Supreme*
Design	Top-loading	Top-loading
Instrument interface	Touch display	Touch display
Temperature range	-150 °C to 1500°C/1600°C*	-150°C to 1600°C
Measuring ranges/ Δl resolution	▪ 500 µm (± 250 µm)/0.125 nm ▪ 5000 µm (± 2500 µm)/1.25 nm	▪ 500 µm (± 250 µm)/0.125 nm ▪ 5000 µm (± 2500 µm)/1.25 nm
Furnace hoist	Single, motorized (double optional)	Double, motorized
Furnace	▪ IC furnace for use with intracooler ▪ SiC furnace ▪ Steel furnace	▪ SiC furnace ▪ Steel furnace ▪ IC furnace for use with intracooler ▪ Copper furnace ▪ Water-vapor furnace
Heating/cooling rate	0.001 K/min to 50 K/min	0.001 K/min to 50 K/min
Cooling systems	▪ Intracooler for IC furnace ▪ Fan for SiC furnace	▪ Intracooler for IC furnace ▪ Fan for SiC furnace For steel and copper furnace: ▪ Liquid nitrogen cooling ▪ Vortex tube
Atmospheres	Inert, oxidizing, static, dynamic, vacuum, reducing	Inert, oxidizing, static, dynamic, vacuum, reducing
Humid atmospheres*	No	Humidity, water vapor
Hydrogen atmospheres*	Yes	Yes
Gas flow control	3-way switch or 1-/3-/4-way MFC*	1-way MFC or 3-/4-way MFC*
Measurement modes	Expansion, penetration, 3-point bending, tension	
Force and displacement	Simultaneous measurement of force and displacement signal	
Force range (load at the sample)	0.001 N to 3 N without using additional weights	0.001 N to 4 N without using additional weights
Force resolution	< 0.01 mN	< 0.01 mN
Interchangeable sample holder systems	▪ Fused silica: up to 1100°C ▪ Alumina: up to 1600°C	▪ Fused silica: up to 1100°C ▪ Alumina: up to 1600°C
Special sample containers	For tests on pastes, powders, liquids, molten metals, molten salts, waxes, immersion	
Coupling*	Yes	Yes

* optional

Technical Specifications

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