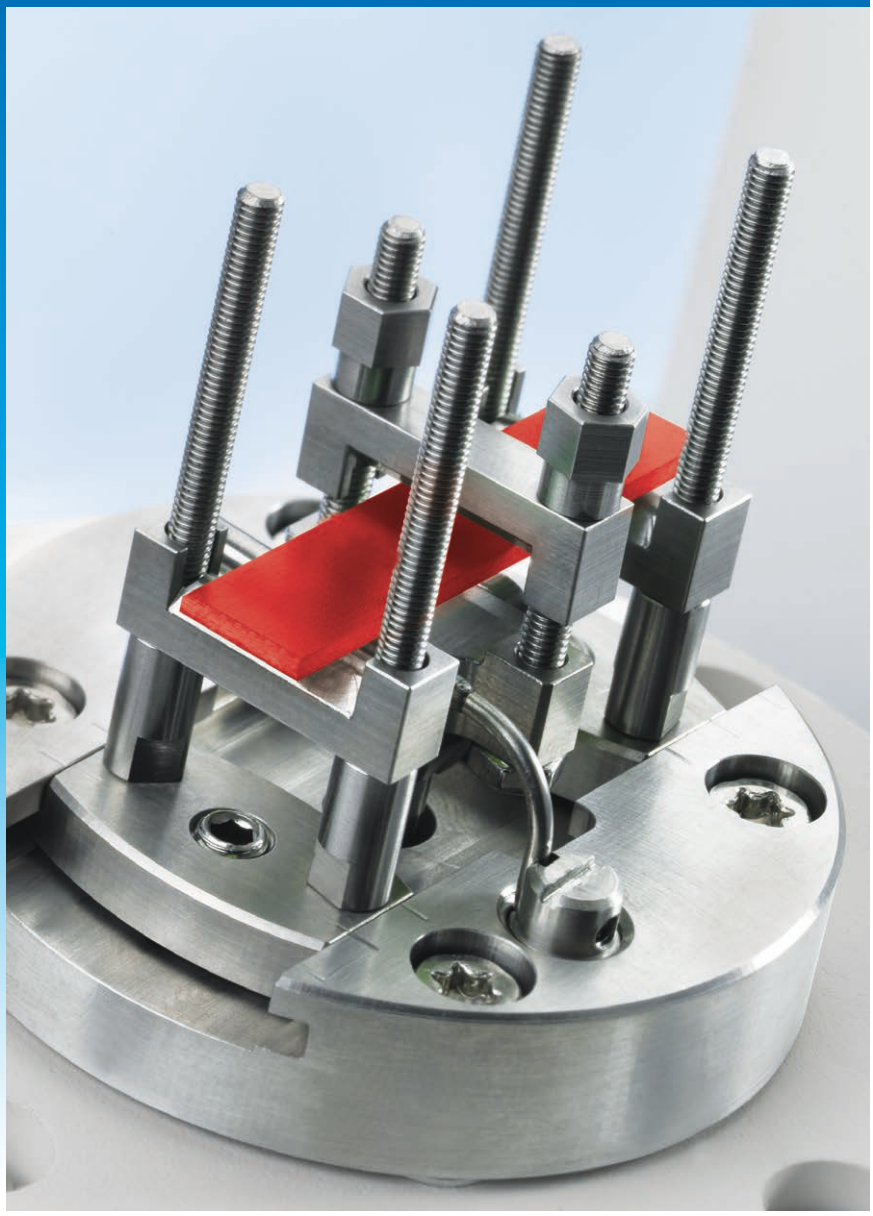
Thermal Analysis Excellence



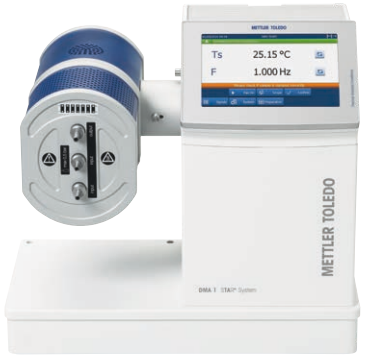
DMA 1

STAR^{e} System

Innovative Technology

Versatile Modularity

Swiss Quality



Dynamic Mechanical Analysis

Comprehensive Materials Characterization

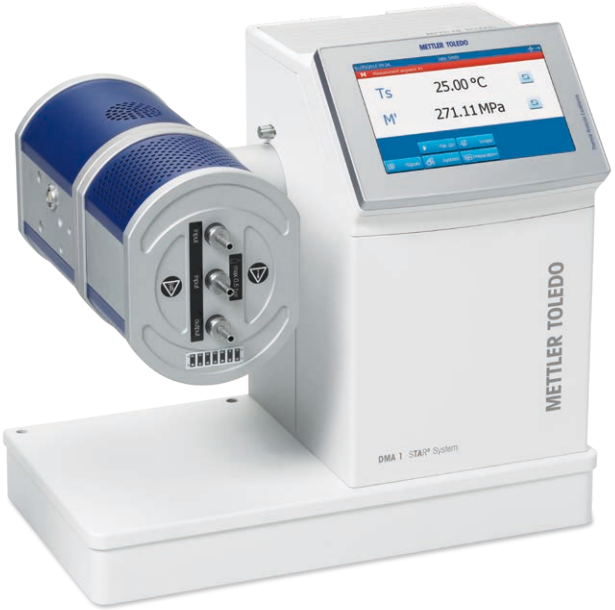
METTLER TOLEDO

Multipurpose DMA
The Perfect Solution for Materials Analysis

Dynamic mechanical analysis (DMA) is an important technique used to measure the mechanical and viscoelastic properties of materials such as thermoplastics, thermosets, elastomers, ceramics and metals. In DMA, the sample is subjected to a periodic stress in one of several different modes of deformation. The force and displacement amplitudes and phase shift are analyzed as a function of temperature, time and frequency.

Features and benefits of the METTLER TOLEDO DMA 1:

- **Flexible positioning of the measuring head –** measurements in all deformation modes, even in liquids or at different relative humidity levels
- **Easy operation –** allows fast change of deformation modes
- **TMA measurements –** for measuring expansion coefficients, effects due to creep, and relaxation times
- **Humidity option –** for sorption and desorption measurements
- **Ergonomic design with large touchscreen –** for convenient sample loading and monitoring of the measurement process
- **Wide temperature range –** from –190 °C to 600 °C
- **Extremely efficient and economical cooling –** saves valuable measurement time and reduces liquid nitrogen consumption



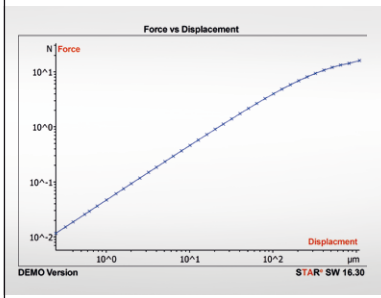
A unique aspect of the DMA 1 is its rotatable measuring head. Measurements can be carried out in all standard deformation modes, even in liquids or at defined relative humidity levels.

www.mt.com/ta-dma

Rapid Results
Thanks to Many Innovations

For easy sample preparation, the measuring head can be placed in the most convenient position for mounting sample holders and clamping samples. Afterward, it is set to the optimum position for measurement in the particular deformation mode.

Accurate displacement measurements



A key component of the DMA 1 is the LVDT (Linear Variable Differential Transformer). The LVDT measures changes in length over the entire measurement range of ± 1 mm with a mean resolution of 2 nm.

Titanium sample slamps

The DMA 1 clamps are made of a titanium alloy. They are extremely important for precise measurements and offer the following advantages:

- The resonant frequency won't interfere with the experiments.
- Titanium forms an inert oxide layer, so no reactions will occur with your samples.
- The thermal conductivity of titanium allows the clamps to have little influence on the furnace temperature.

Touch screen

The touchscreen allows visual contact with the instrument, and has two important functions:

- It displays the current spring displacement when mounting the sample holder and clamping the sample.
- It monitors the sinusoidal excitation function, which shows you whether the sample has been properly mounted in the sample holder.

Convenient sample clamping

The ergonomic design of the DMA 1 allows for easy sample clamping and quick changes of the sample clamps when different deformation modes are needed.

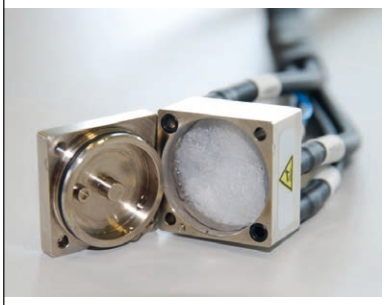
Perfectly Designed
Down to the Last Detail

The unparalleled versatility of the DMA 1 allows applications to be performed in the optimum measurement configuration. The DMA 1 is quick and easy to set up, whether for conventional DMA analyses, experiments using static forces, measurements in liquids, or under a defined relative humidity.

Fluid bath

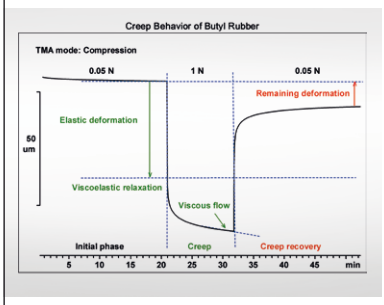
The fluid bath option consists of a special immersion bath and external temperature control using a circulating heating bath or chiller. The fluid bath can be operated between –20 and 200 °C.

DMA sorption



The humidity option consists of a special humidity chamber, a circulating heating bath and a humidity generator. It allows you to perform measurements under optimum conditions in every deformation mode. Special readjustment is not necessary after installing the humidity chamber.

TMA mode



The design of the DMA 1 allows it to be used for TMA measurements (using a static force). The sample holders and sample clamps are attached in the same way as when performing DMA measurements. Some special types of TMA measurement include:

- Creep/Recovery measurements
- Stress-Strain diagrams
- Deformation-Relaxation diagrams
- Coefficient of thermal expansion (CTE)

The fluid bath option allows you to perform DMA or TMA experiments in liquids using all the standard deformation modes. The entire sample holder and sample are immersed in the liquid.

www.mt.com/ta-moisture

Optimum Configuration
Saves Valuable Measurement Time

The cooling performance of the DMA 1 is very impressive. It cools the sample from room temperature to –190 °C in less than 10 minutes with an amazingly low consumption of liquid nitrogen (less than 1 liter for 3 cooling cycles to –100 °C). This saves both time and money because the container does not have to be refilled so often. The main advantage is increased sample throughput. If a measurement begins at room temperature, the DMA 1 can be operated without a cooling option.

1 liter dewar

A simple and convenient way to cool the DMA 1. Get up to three cooling cycles with the 1 liter liquid nitrogen dewar.

35 liter dewar

This large volume dewar allows multiple measurements at exteremely low temperature. It is also the best option for isothermal measurements below 0 °C.

Fan cooled

If a measurement begins at room temperature, the DMA can be operated without a cooling option, simplifying set up drastically.

Matching accessories

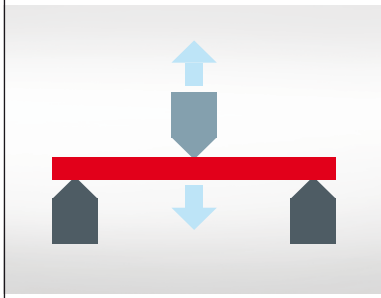
Contains all the titanium sample holders and clamps needed for mounting the sample holders and temperature sensor. The calibration box includes all the materials required for performing individual temperature adjustments. This is a key factor for achieving precise and reliable measurement results.

Deformation mode	Max. sample lenght (mm)	Max. sample width (mm)	Standard head position (without liquid)
Single cantilever bending	17.5	13	horizontal
Dual cantilever bending	35	13	horizontal
3-point bending	45	13	vertical (pointing up)
Tension	20	13	horizontal

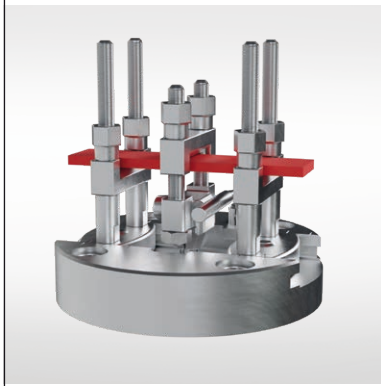
Deformation mode	Max. sample diameter (mm)	Max. sample tickness (mm)	Standard head position (without liquid)
Shear	10	12	horizontal
Compression	10	16	vertical (pointing up)

Sample Holders
Simple, Ingenious and Timesaving

3-point bending

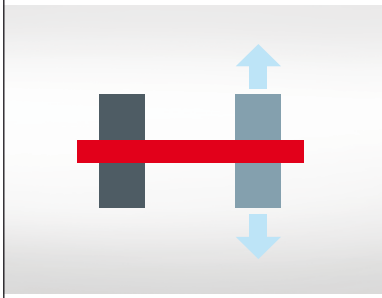


This bending mode is ideal for measuring extremely stiff samples, such as composite materials or thermosets, particularly below the glass transition temperature.

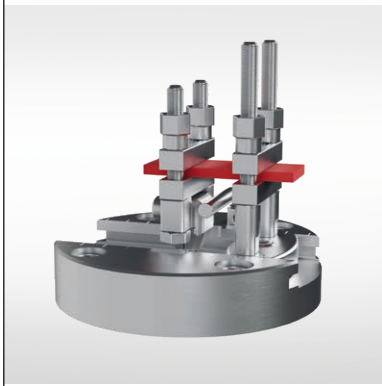


In the 3-point bending mode, the ends of the sample rest on two knife edges and an oscillatory force is applied to the middle of the sample by a moving knife edge.

Single cantilever bending

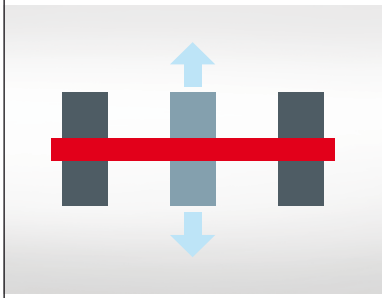


This mode is excellent for bar-shaped materials (metals, polymers) that display a high degree of stiffness. The single cantilever approach is ideal for measurements below the glass transition temperature and is the recommended mode for determining the loss factor (tan delta) of powdery materials.



The single cantilever mode is very similar to the dual cantilever mode except that only one end of the sample is fixed while the other end is connected.

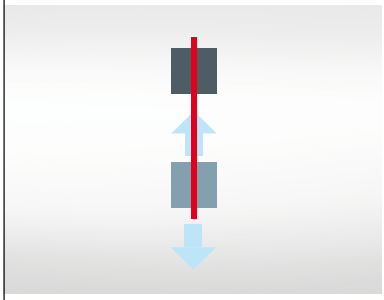
Dual cantilever bending



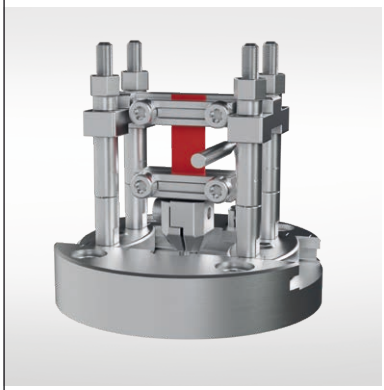
This mode is suitable for softer materials with a lower degree of stiffness, in particular thin samples such as films.

In the dual cantilever mode, the ends of the sample are fixed and the middle is clamped to the moving part providing the oscillatory force.

Tension

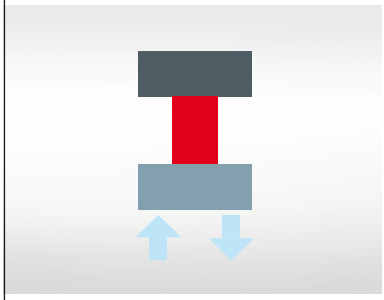


Tension is the mode most suitable for films, fibers and thin bars and rods. The advantage is that sample clamping hardly affects the deformation.



In the tension mode, one end of the sample is fixed and the other is subjected to an oscillatory force. The sample must be pre-stressed to prevent it from buckling during the oscillatory movement.

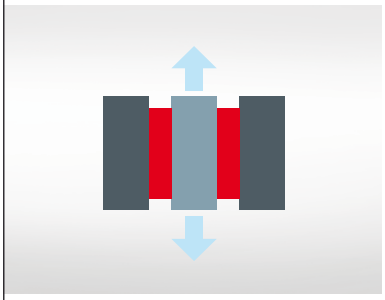
Compression



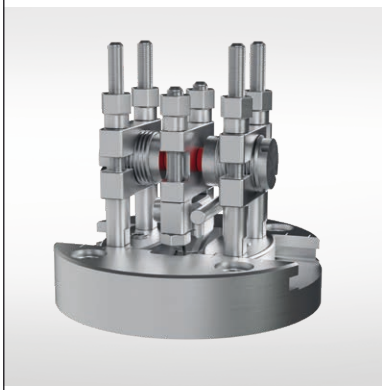
This type of measurement is most suitable for the determination the valuable material properties of soft materials like elastomers, pastes or foams.

In the compression mode, the sample is clamped between a fixed part and the moving part providing the oscillatory force. The sample is compressed statically and subjected to an alternating load.

Shear



The shear mode is ideal for soft samples, such as elastomers, pressure-sensitive adhesives and for studying curing reactions.



In the shear mode, two identical samples are clamped symmetrically between two fixed outer parts and a central moving part. The shear clamp guarantees a homogeneous temperature distribution.

DMA Theory
Force, Displacement and Phase Angle

The modulus is calculated from the applied force amplitude, F^a, the measured displacement amplitude, L^a, and the phase shift δ between the force and displacement signals.

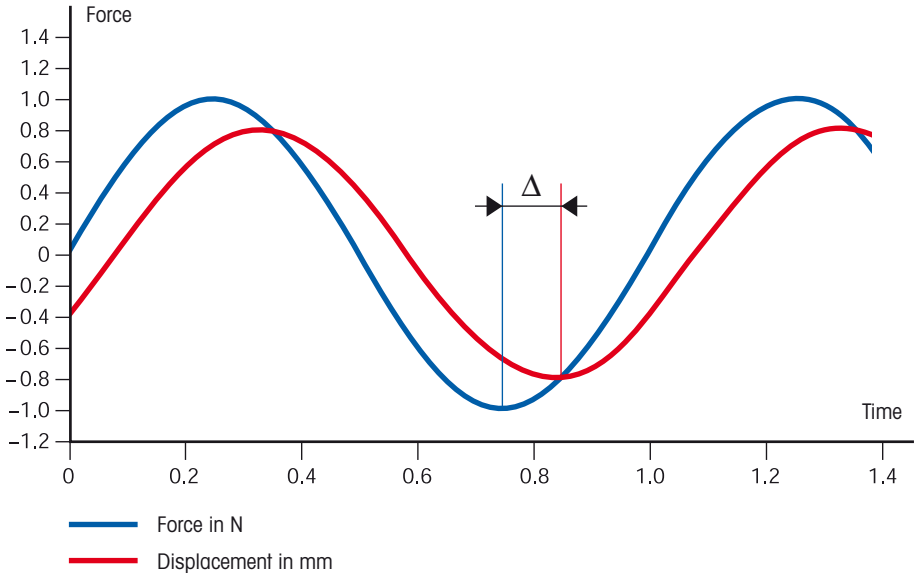
The types of modulus are:

- Complex modulus, M*, (elastic modulus, E*, for tension; G* for shear)
- Storage modulus, M′, (proportional to the energy stored elastically and reversibly)
- Loss modulus, M″, (proportional to the energy transformed into heat and irreversibly lost)

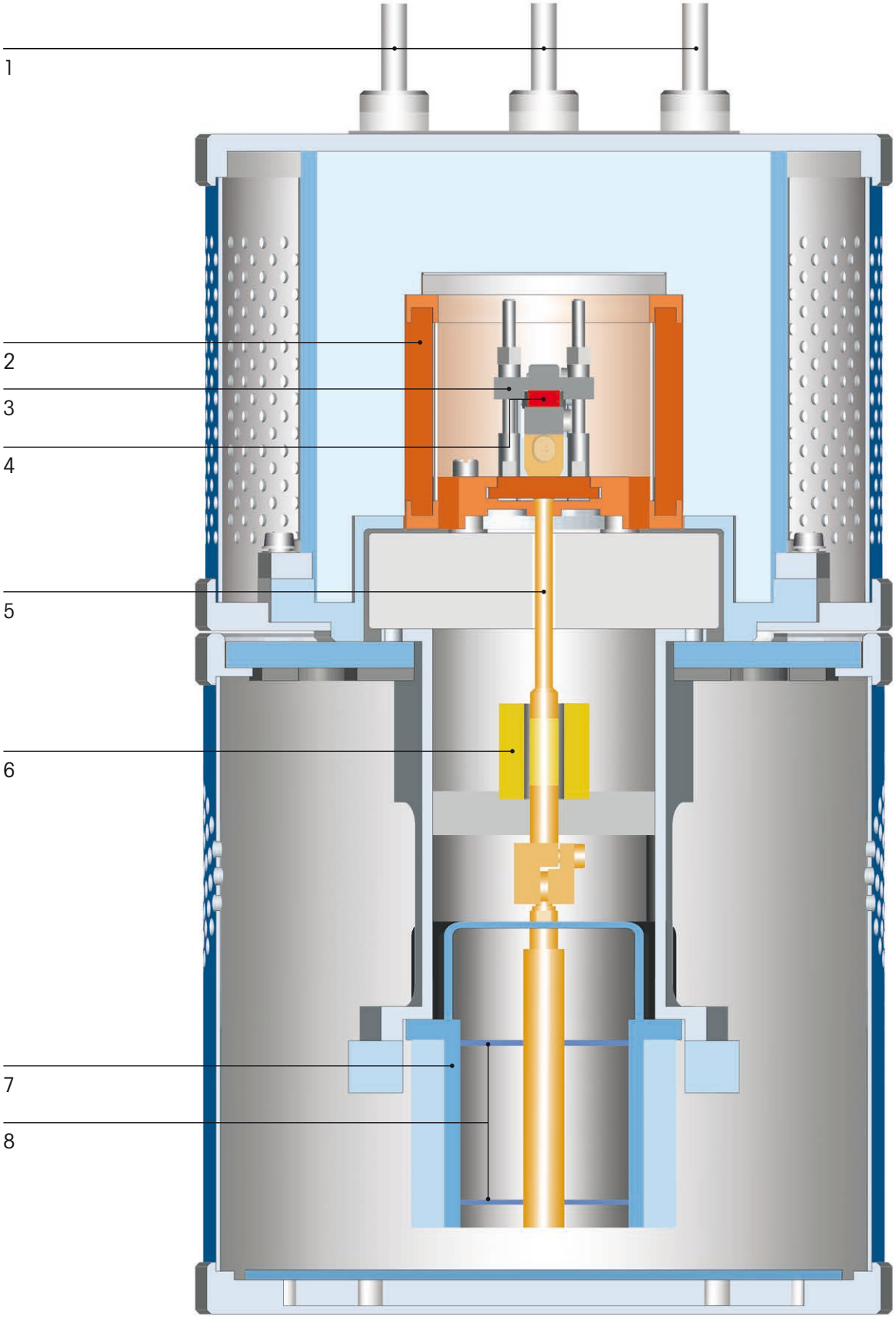
The modulus values can then be used to calculate the loss factor (tan δ), which corresponds to the ratio of M″ to M′. Completely elastic materials have a loss factor of 0, while purely viscous materials have an infinitely large loss factor (δ = 90°). The moduli are calculated from the measured stiffness S (N/m) and the geometry factor g. S is the quantity actually determined.

$M' = |M^*| \cos \delta \quad M'' = |M^*| \sin \delta \quad \tan \delta = M'' / M'$

$|M^*| = S^*g = F^a/L^a * g$; stiffness $S = F^a/L^a$



Force and displacement at a frequency, f, of 1Hz. The phase shift, δ, can be calculated from the time delay, Δ, using the equation $\delta = 2\pi f \Delta$.



Key

1. LN2 inlet/outlet
2. Heater element
3. Sample holder
4. Sample
5. Drive shaft
6. LVDT displacement sensor
7. Drive motor
8. Drive shaft guidance (spring)

Extremely Wide Application Range
For All Kinds of Materials

The DMA 1 is the ideal instrument to use for the dynamic mechanical analysis and characterization of materials. Measurements can be performed with conventional sample positions, submersed in liquids or at specific relative humidity levels. It facilitates a large number of applications and provides valuable information in quality control and in industrial/academic research.

Materials are subjected to a variety of different stresses in practical use. The most important factors are the time-dependent intensity of stresses, the temperature, and the environment in which the stress is applied. Dynamic mechanical analysis allows issues such as stability, practical application range, manufacturing processes, quality control, and material failure and defects to be addressed.

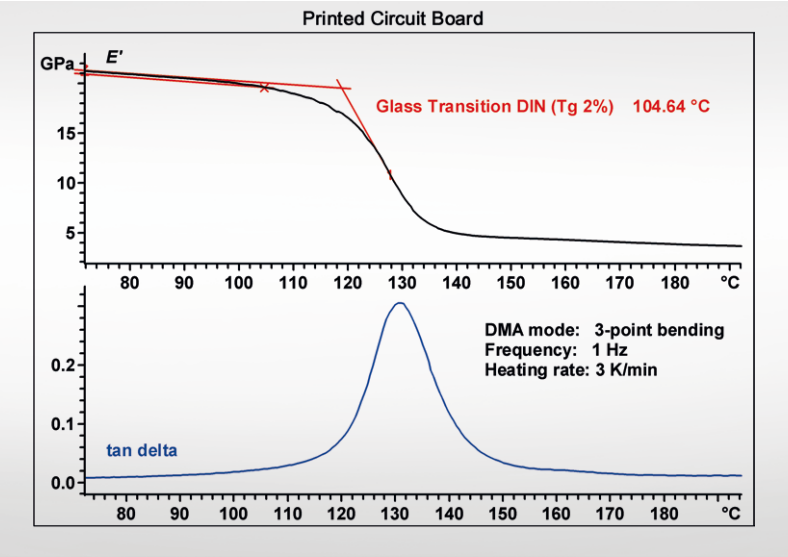
Effects and properties that can be characterized using the DMA 1 system

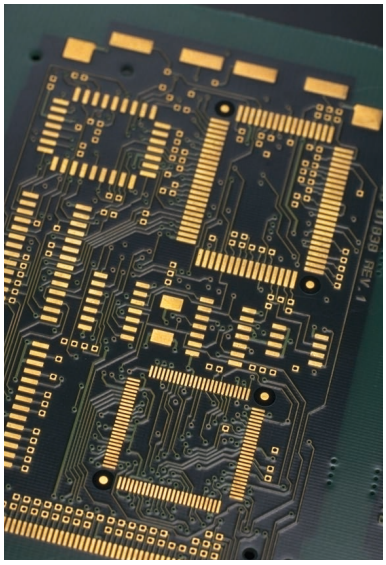
- Viscoelsatic behavior
- Relaxation behavior
- Glass behavior
- Mechanical moduli
- Damping behavior
- Softening
- Viscous flow
- Crystalization and melting
- Gelation
- Phase transformations
- Composition of blends
- Curing and polymerization reactions
- Material defects
- Effects due to filters

The materials most commonly analyzed are polymers such as thermoplastics, thermosets, elastomers and adhesives, metals, composites, paints and varnishes, foils and fibers, construction materials, pharmaceuticals, and foodstuffs.

www.mt.com/ta-applications

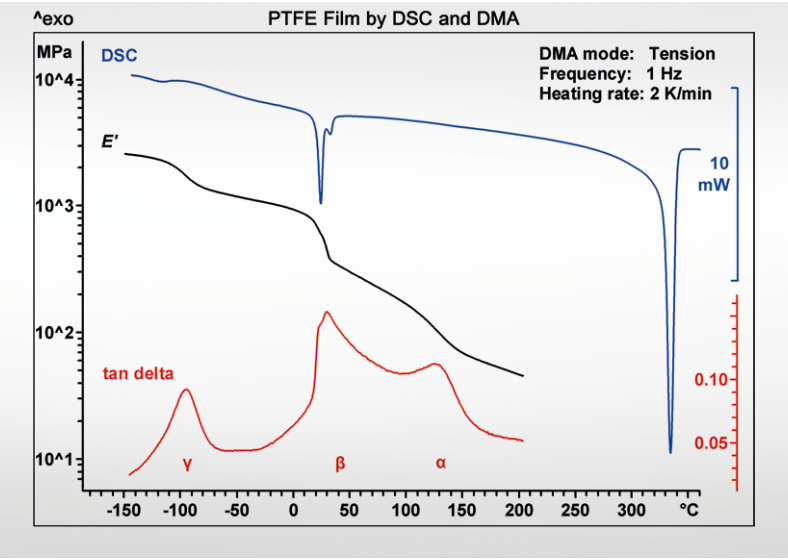
Composite materials

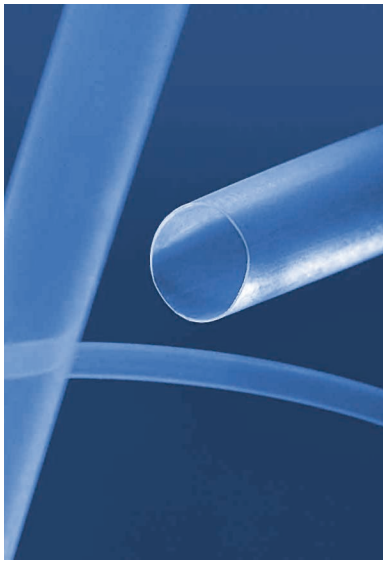




Composite materials made of filled cross-linked polymers have a high storage modulus at the temperature at which they are used. The modulus in this example is determined by 3-point bending. The upper curve shows the storage modulus of a printed circuit board. The value measured at 70 °C and a frequency of 1 Hz was 21.1 GPa. The curve also shows the softening process at the glass transition where the modulus falls to less than 5 GPa. The step in the storage modulus corresponds to the peak in the loss factor, tan delta.

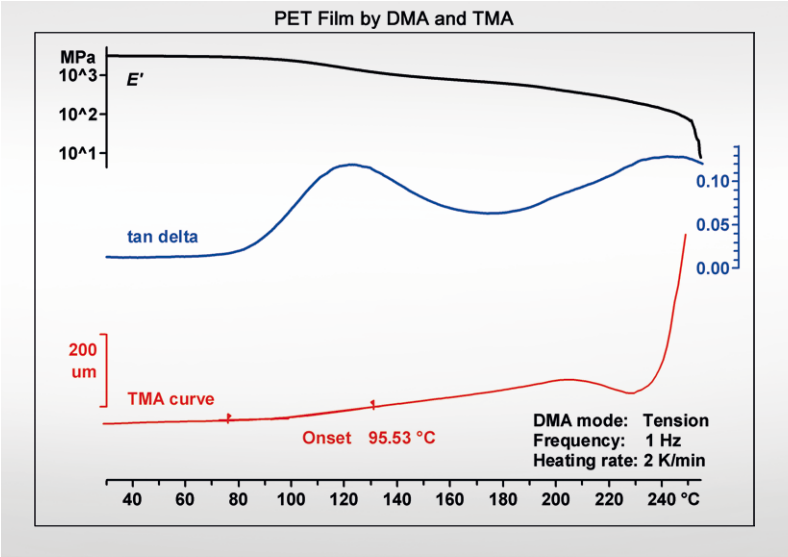
Phase transitions of PTFE

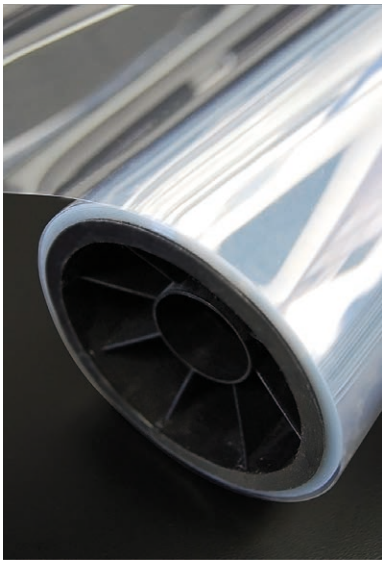




The DSC curve of PTFE shows phase transitions at about –100 °C and +30 °C as well as melting at approximately 330 °C. Theses phase transitions are also measurable by DMA in the tension mode. The transition temperatures measured by the two methods show excellent agreement. DMA is extremely sensitive when there are small changes in the modulus, therefore a glass transition is observed at +130 °C, which was not visible in the DSC curve.

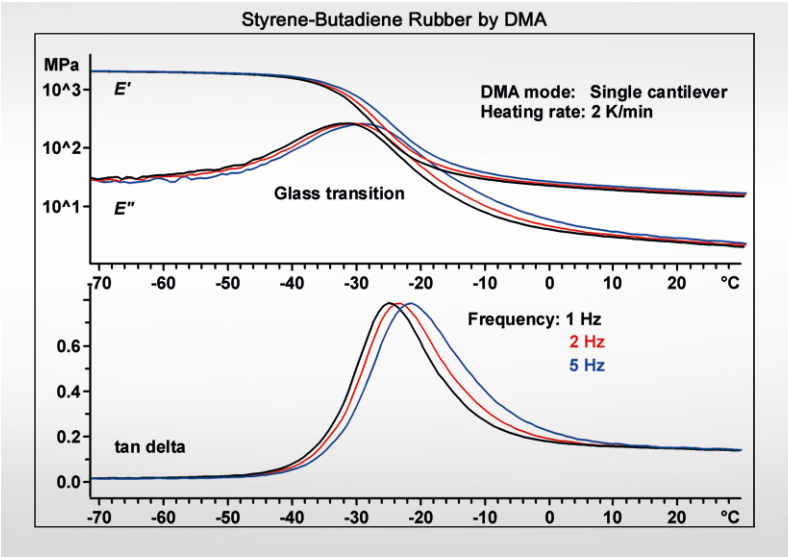
PET film by DMA and TMA





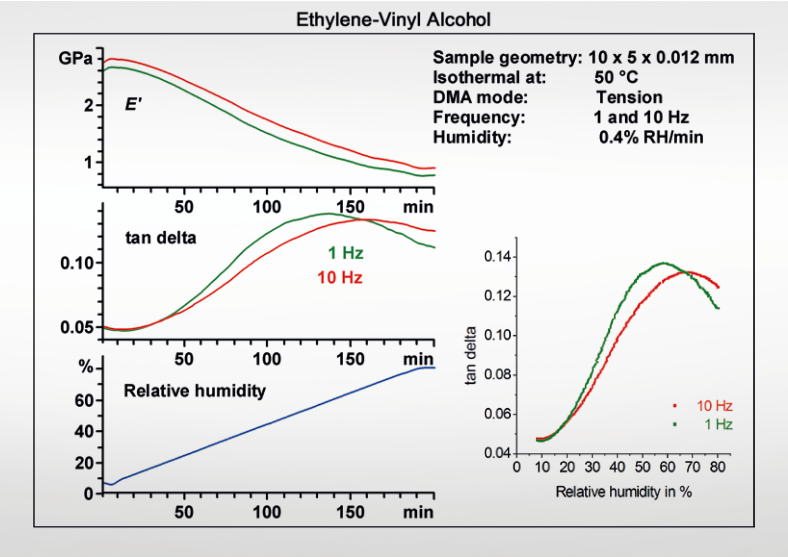
The diagram shows DMA curves of a PET film measured in the tension mode at 1 Hz. Curves like these are often used for quality control purposes. Due to crystallinity, the change in the modulus at the glass transition between 80 and 150 °C is only about one decade. The modulus shows a further decrease at the onset of melting at 230 °C. The tan delta curve exhibits a relaxation peak in the glass transition range. The bottom curve is measured in the TMA mode and shows the change in length of the film. The slope changes at the glass transition onset temperature of 95 °C. The film then shrinks between 210 and 230 °C.

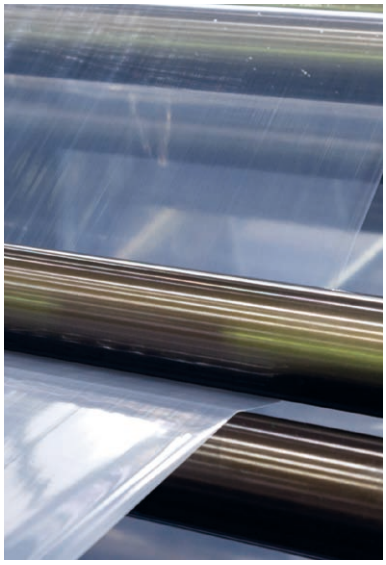
Styrene-butadiene rubber



Styrene-butadiene rubber (SBR) is used in car tires and for gaskets. A sample of SBR was measured at frequencies of 1, 2, and 5 Hz in the single cantilever mode. The glass transition occurs at about –20 °C and defines the lower temperature limit of use for this material. The tan delta curves also clearly show the frequency dependence of the glass transition. At higher frequencies, the glass transition shifts to higher temperatures. The storage modulus changes by about two decades during the glass transition.

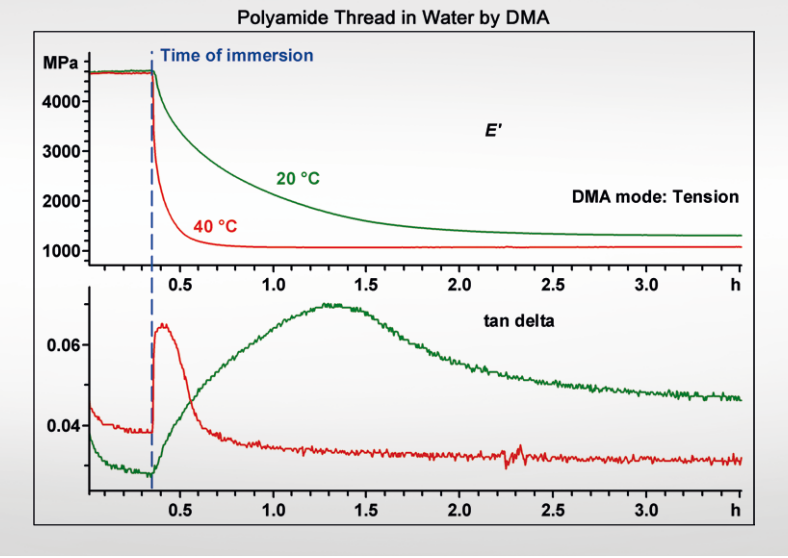
Effect of relative humidity





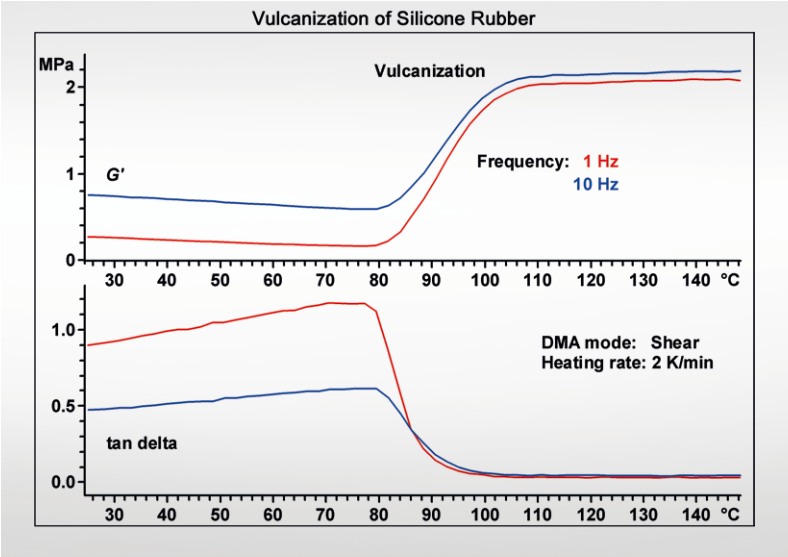
Ethylene-Vinyl Alcohol (EVOH) copolymer is often used in packaging films for foodstuffs because of its excellent barrier properties toward oxygen and water vapor. Since EVOH is hygroscopic and water acts as a plasticizer, the barrier properties of a film are influenced by its water content. Isothermal DMA measurements at 50 °C show that an increase in relative humidity leads to a decrease in the storage modulus. The peak in the tan delta curves is due to the decrease of the glass transition temperature with increasing relative humidity. Since the glass transition is frequency dependent, the peak measured at lower frequency appears at lower relative humidity.

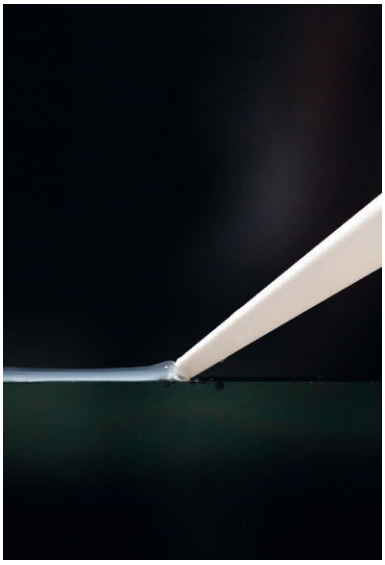
Polyamide thread in water



The mechanical properties of materials can change drastically in contact with liquids. Some polymers become hard and brittle in some liquids whereas other liquids act as plasticizers. The DMA 1 allows the mechanical behavior of a sample to be measured while it is fully immersed in a liquid. The example shows measurements of a polyamide thread in water at 20 °C and at 40 °C. The glass transition temperature decreases due to the absorption of water. The modulus curves show that the softening process occurs more quickly at 40 °C than at 20 °C.

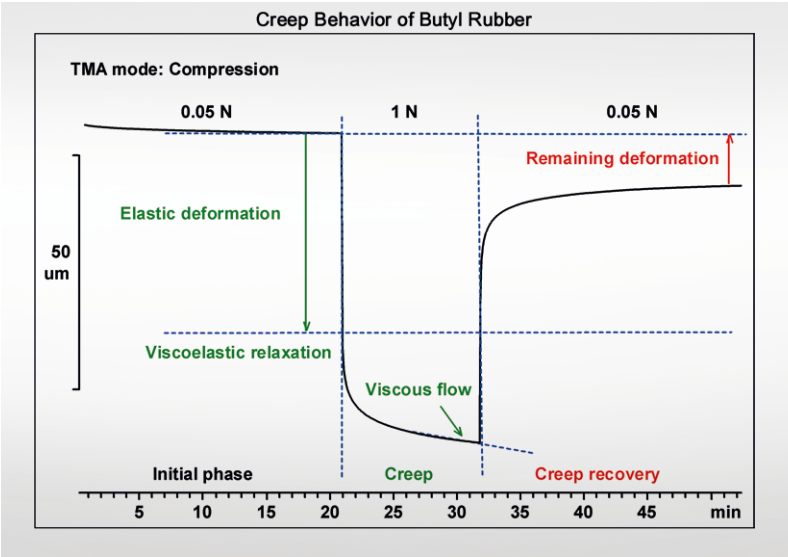
Silicone rubber

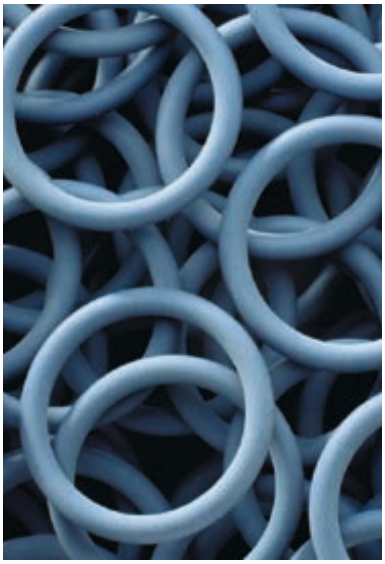




The vulcanization process converts a viscous liquid into a rubbery elastic solid with a low modulus. This change in material properties is clearly evident from the DMA curves. The figure displays the storage modulus and tan delta curves of silicone rubber measured in the shear mode at 1 Hz and 10 Hz. Vulcanization occurs between 80 °C and 90 °C. The curves show that the storage modulus increases during vulcanization whereas tan delta exhibits a marked decrease. The material is much more elastic after vulcanization than before.

Creep behavior with TMA mode



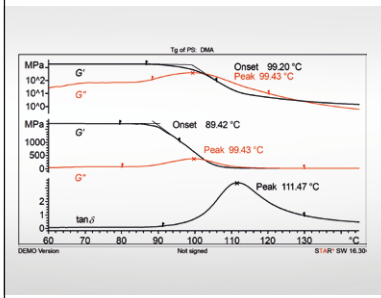


The recovery properties of an elastomer are crucial for its use as a seal. The sample measured was butyl rubber (IIR). Initially, a force of 0.05 N was applied. This was then suddenly increased to 1 N. The resulting deformation consists of three components: the immediate elastic deformation, the time-dependent viscoelastic relaxation, and viscous flow. The residual deformation that remains after the force has been removed is the permanent deformation due to viscous flow. An elastomer like this would only be of limited use for seals and gaskets.

Simple, Intuitive Operation
Straightforward, Efficient and Secure

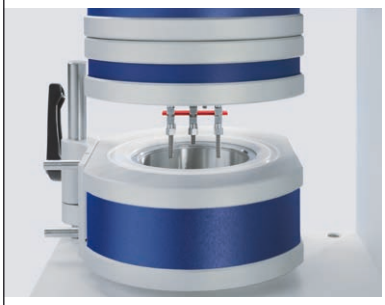
STARe software has been expanded to include new features that help you prepare your DMA 1 instrument for specific experiments, develop methods for advanced analyses and perform flexible result evaluations. Complex measurement programs are set up within minutes and the vast range of available tools permit curves to be evaluated both accurately and efficiently.

Glass transition (T_g)



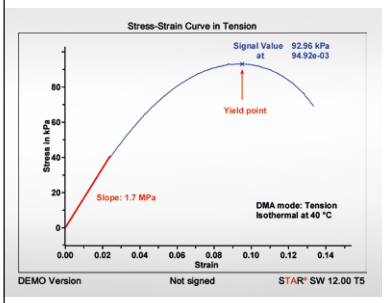
Glass transition (T_g) is a very important characteristic of polymeric materials. It can be determined and calculated with DSC, TMA and DMA. If the measurements are all the same, the T_g values of identical samples measured in different laboratories should not deviate by more than 2 K.

Submersion bath



The fluid bath option allows you to perform DMA or TMA experiments in liquids using all the standard deformation modes. The entire sample holder and sample is immersed in the liquid. The fluid bath option consists of a special immersion bath and external temperature control using a circulating heating bath or chiller. The fluid bath can be operated between –20 and 200 °C sample temperature.

Stress-Strain



A stress-strain diagram is obtained by subjecting a material to a force that slowly increases (i.e. quasi-static conditions). The applied force and the dimensional change are continuously measured. In this example, the tensile stress (force per area) is then plotted against the tensile strain (ΔL divided by the original length L_0).

A complete thermal analysis system consists of the basic six complementary measuring techniques, each of which bring fast and accurate results. Additional knowledge can be obtained by means of several hyphenated techniques.

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DMA 1 Specifications

Temperature data	
Temperature range	–190 to 600 °C
Technical resolution	0.1 K
Temperature accuracy	0.75 K
Heating rate	0.1 to 20 K/min
Cooling rate	0.1 to 30 K/min

Force data	
Force range	± 0.001 to ± 10 N
Technical resolution	0.25 mN
Sensitivity	1 mN

Displacement data	
Displacement range	± 1 mm
Technical resolution	2 nm
Sensitivity	30 nm

Stiffness	
Stiffness range	50 to 10^5 N/m
Precision	0.50%

Tan delta	
Tan delta range	0.0001 to 50
Technical resolution	0.00001
Sensitivity	0.0001

Frequency	
Frequency range	0.001 to 300 Hz
Technical resolution	0.0001 Hz
Accuracy	0.001 Hz
Frequency modes	• Logarithmic and linear scans • Multi-frequency (sequentially)

Maximum sample length	
Sample length	55 mm

Fluid Bath Option	
Temperature range	–20 to 200 °C

Humidity Option	
Temperature range	5 to 85 °C
Humidity range	5 to 95% RH

Approvals

IEC/EN61010-1:2001, IEC/EN61010-2-010:2003
CAN/CSA-C22.2 No. 61010-1-04 & -2-010
UL Std. No. 61010-1 (2nd Edition)
IEC61326-1:2005 / EN61326-1:2006 (class B)
IEC61326-1:2005 / EN61326-1:2006 (Industrial Environment)
FCC, Part 15, class A
AS/NZS CISPR 22, AS/NZS 61000.4.3
Conformity Mark: CE

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