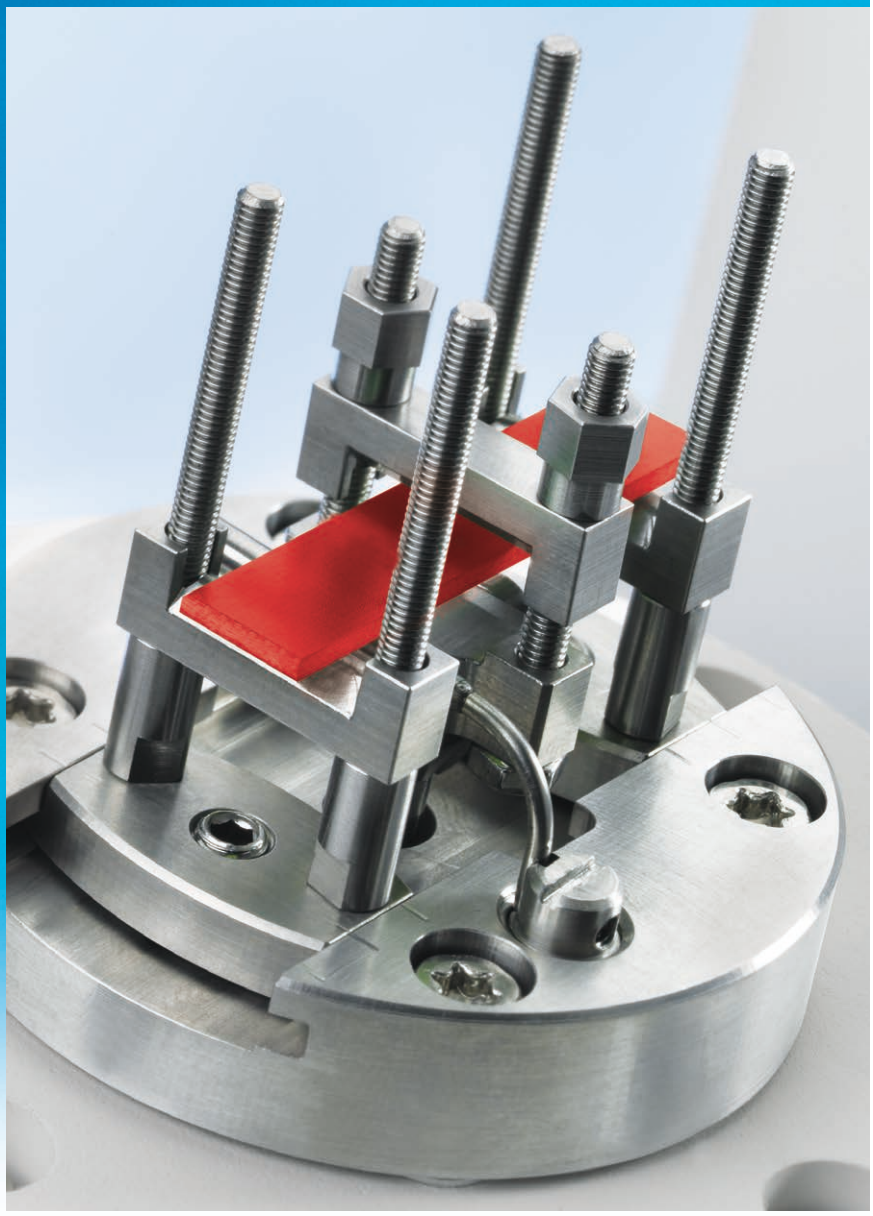
Thermal Analysis Excellence



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

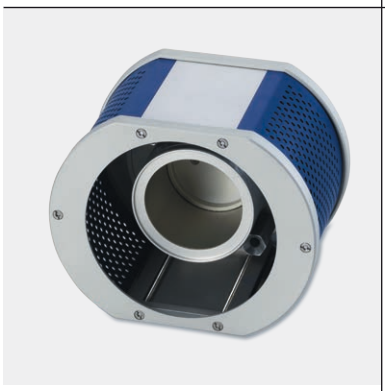
Dynamic mechanical analysis (DMA) is an important technique used to measure the mechanical and viscoelastic properties of materials such as thermoplastics, thermosets, elastomers, ceramics and metals. In DMA, the sample is subjected to a periodic stress in one of several different modes of deformation. 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 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.

Unmatched Versatility
the Optimum Configuration for All Applications



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 or measurements in liquids.

Measurements at controlled relative humidity

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.

Measurements with static forces

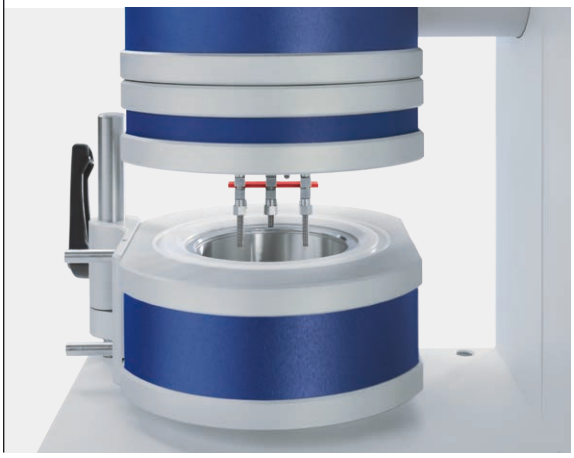
Besides the dynamic mode, the DMA 1 permits measurements to be performed using static forces (TMA mode). All the deformation modes available for DMA can be used.

Typical TMA applications include:

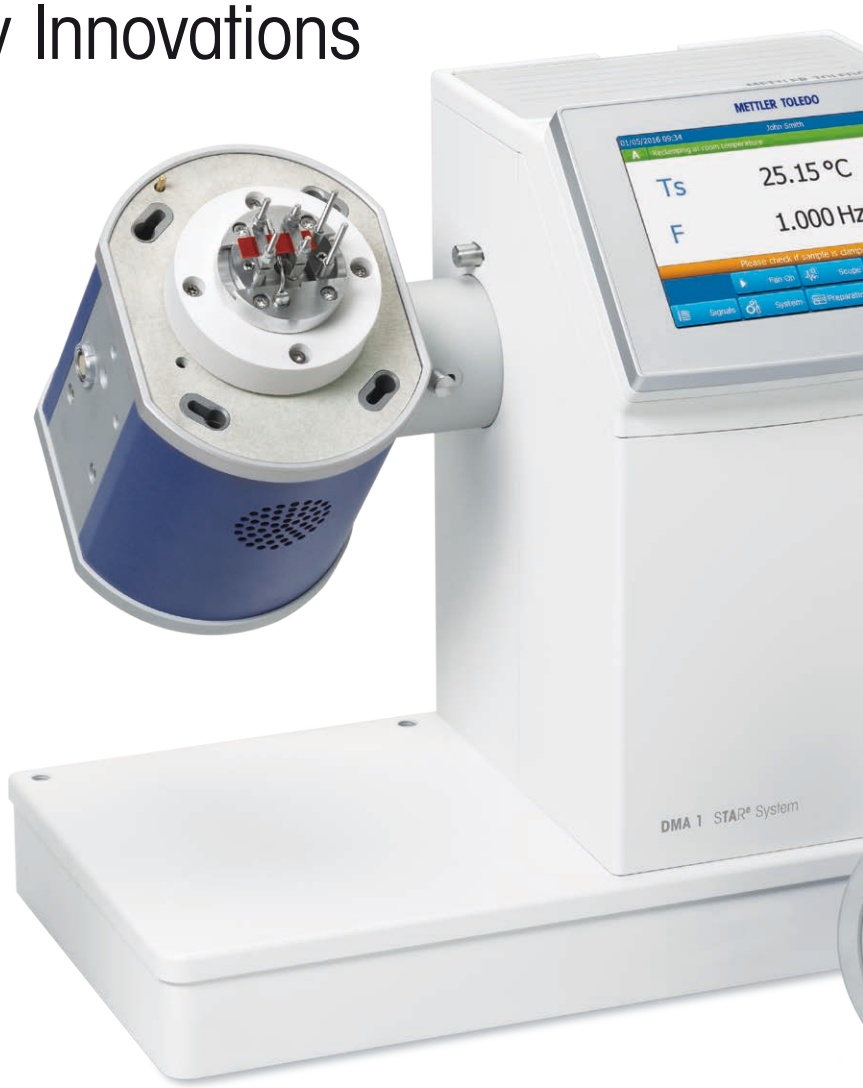
- Determination of coefficients of thermal expansion
- Creep and recovery experiments
- Stress-strain diagrams
- Deformation-relaxation diagrams
- Softening temperature of materials

Measurements in liquids

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.



Rapid Results
Thanks to Many Innovations

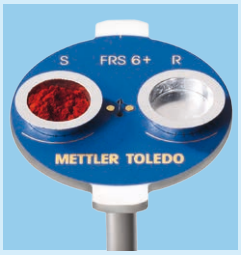


Convenient sample clamping
The Measuring Head can be placed in the most convenient position for mounting sample holders and clamping samples. Afterward, it is set to the optimum position for measurement in the particular deformation mode. The orientation of the Measuring Head is automatically detected.

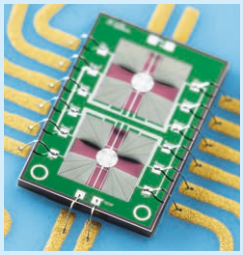
Although the positions are different, the system does not require calibration or readjustment.

Complete thermal analysis system
A complete thermal analysis system consists of four different measurement techniques. Each characterizes the sample in its own specific way. The combination of all the results gives the complete picture and simplifies interpretation. DMA measures the mechanical modulus, DSC and Flash DSC the heat flow, TGA the weight curve, and TMA the length change. All these quantities change as a function of temperature.


DMA



DSC


Flash DSC


TGA

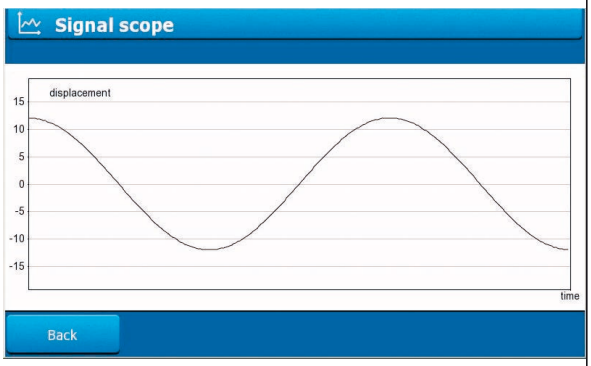

TMA

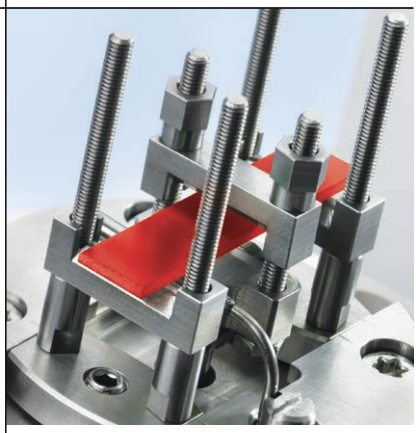
Perfectly Designed
Down to the Last Detail

Touchscreen of the DMA 1

The touchscreen allows visual contact with the instrument, even from a distance and has two important functions:

- It displays the current spring displacement when mounting the sample holder and clamping the sample. This protects the measurement system and ensures that nothing gets damaged.
- It monitors the sinusoidal excitation function. This is extremely important, particularly at the start of a measurement. It shows you whether the sample has been properly mounted in the sample holder.





Titanium sample clamps

Sample clamps are extremely important for precise measurements. The DMA 1 clamps are made of a titanium alloy. This offers the following advantages:

- The natural resonant frequency of the system is shifted to higher frequencies due to the low weight of the clamps.
- The clamps are very resistant to corrosion because titanium forms an inert oxide layer in contact with air. This is especially advantageous for measurements in liquids.
- The clamps can be heated or cooled more quickly because the thermal conductivity of titanium is better than that of most other potentially suitable materials.

Matching accessories

The Accessory Box contains all the sample holders and sample clamps needed for mounting sample holders and the 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.

Swiss Quality

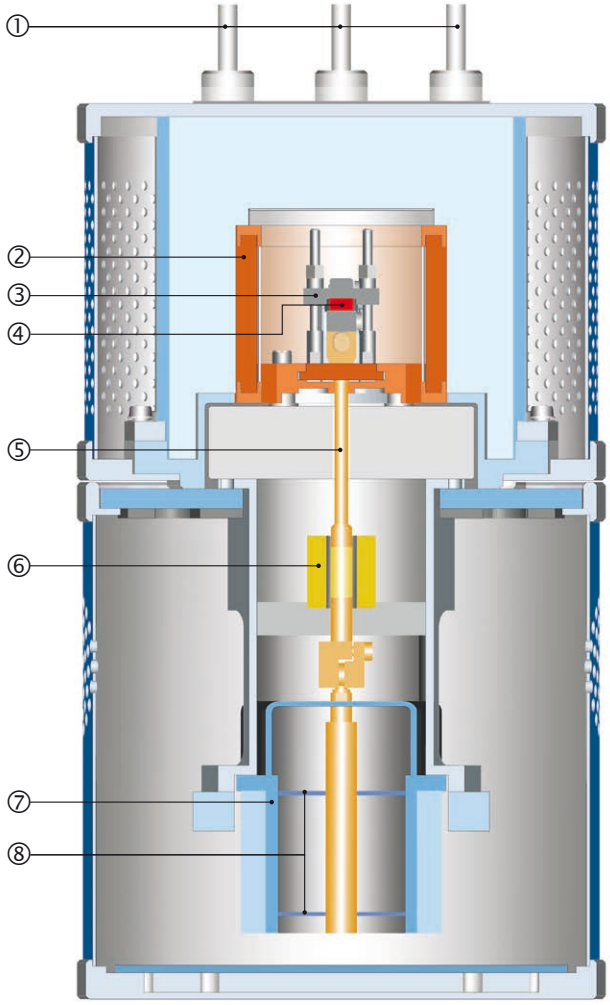
Reliable, First Class Performance
over the Whole Temperature Range

Measuring principle

In dynamic mechanical analysis (DMA), the sample is subjected to an oscillating force and the resulting displacement amplitude is measured. The phase shift between the force and displacement signal is derived from the time lag between the two curves.

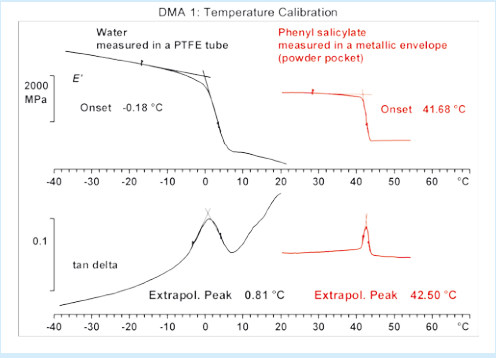
Accurate displacement measurements

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. It is fitted near the sample to minimize any influences caused by deformation of the measuring system. This improves the accuracy of the measurement of the time lag (the phase shift) between the force and displacement.



Key

1 LN_2 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)



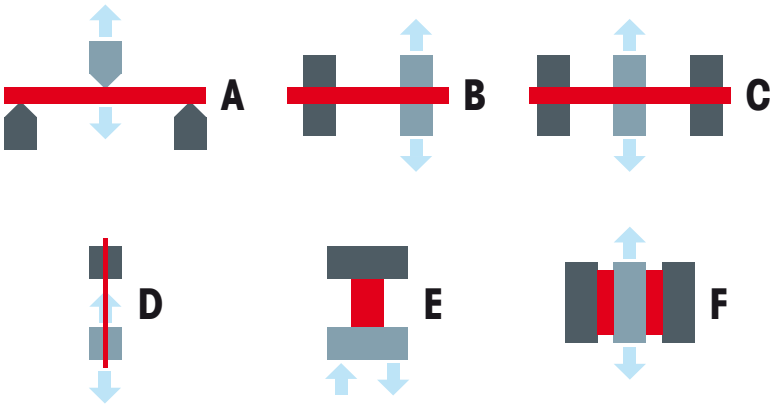
Sample with Pt100

The Pt100 temperature sensor is positioned as close as possible to the sample. Temperature adjustment in the desired deformation mode ensures that temperatures are correctly measured.

Optimized Sample Holder
Convenient and Easy Handling

The DMA 1 offers a choice of six different deformation modes. The most suitable mode for a particular application depends on the information required and the nature and geometry of the sample. The stiffness of the sample must be chosen so that it is appreciably lower than that of the measuring system. All six deformation modes can be used for dynamic and static measurements.

An important aspect of the sample holder system is the ease with which the sample length can be adjusted in the sample holder. The relevant length can be set in steps of 2.5 mm from the minimum length defined for the particular mode up to the maximum length. Sample lengths can also be continuously set using special screws.



The different deformation modes

3-point bending (A): This mode is used for accurate measurements of very stiff samples, such as composite materials or thermosets, particularly below the glass transition temperature. It is also very important for TMA measurements.
Single cantilever bending (B): 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.
Dual cantilever bending (C): This mode is suitable for softer materials with a lower degree of stiffness, in particular thin samples such as films.
Tension (D): This is the usual deformation mode for films or fibers. It is also very important for TMA measurements.
Compression (E): The compression mode is used to measure foams, gels, and foodstuffs and for static force (TMA) measurements.
Shear (F): The shear mode is ideal for soft samples, such as elastomers, pressure-sensitive adhesives and for studying curing reactions.

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

Unsurpassed Cooling Capability
Saves Valuable Measurement Time

Temperature range and cooling options

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 (RT), the DMA 1 can be operated without a cooling option.

LN_2 cooling	**1 liter-Dewar**	**35 liter-Dewar**
Temperature range	–190 to 600 °C	–190 to 600 °C
LN_2 consumption for cooling once from RT to –190 °C	<1 liter LN_2	~1.8 liter LN_2
LN_2 consumption for cooling from RT to –100 °C	<0.3 liter LN_2	<0.4 liter LN_2
Time taken to cool from RT to –190 °C	<10 min	<15 min

Option	**DMA measurements**	**TMA measurements**	**Relative humidity**	**Liquids**
DMA 1 basic instrument	All modes	All modes	All modes	All modes
Cooling option 1-liter Dewar	•	•		
Cooling option 35-liter Dewar	•	•		
Humidity chamber			•	
Humidity generator			•	
Fluid bath				•
Circulating heating bath or chiller			•	•

Flexible DMA
for DMA and TMA Measurements

DMA theory

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:

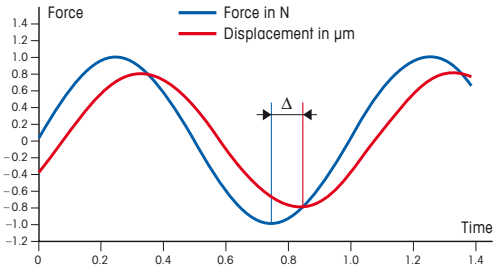
- Complex modulus, M*, (elastic modulus, E*, for tension; G* for shear)
- Storage modulus, M′, (proportional to the energy stored elastically and reversibly)
- Loss modulus, M″, (proportional to the energy transformed into heat and irreversibly lost)

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°).

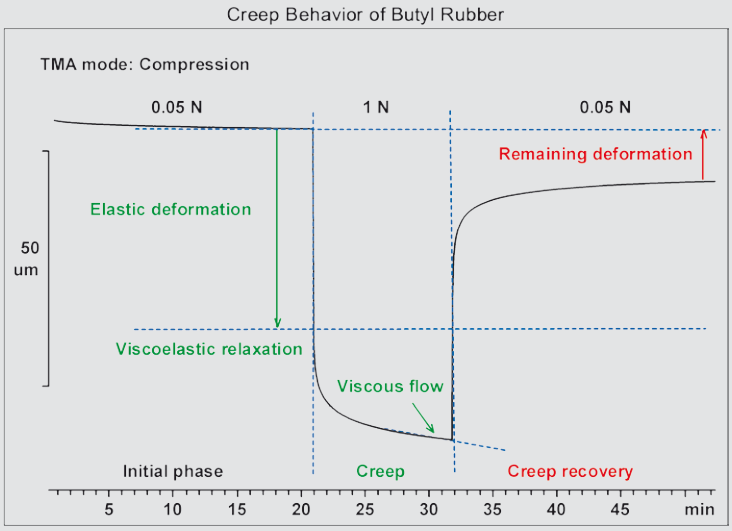
The moduli are calculated from the measured stiffness S (N/m) and the geometry factor g. S is the quantity actually determined.

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

$|M^*| = S^*g = F_a/L_a{}^*g$; stiffness $S = F_a/L_a$



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



Thermomechanical measurements

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**
  The sample is suddenly subjected to a high static force. After a certain time, the force is removed and the recovery of the sample is measured as a function of time.
- **Stress-Strain diagrams**
  The tension (force per unit area acting on the sample) and resulting strain are measured and plotted in a diagram. Typically, the initial portion of the curve is linear for low levels of deformation. The behavior is more complex for larger deformation levels and is no longer linear up to the point at which the sample finally breaks.
- **Deformation-Relaxation diagrams**
  These isothermal measurements show how quickly a material deforms under a static force before a state of equilibrium is reached. This yields the resulting relaxation time.

Support and repair

Support and diagnosis in case of technical issues. Carrying out repairs at a customer's site or at one of our service centers.

Quality assurance and certification

Qualification, documentation, calibration with certificate.

Performance services and preventive maintenance

Professional installation (IQ, OQ) and ensuring optimum performance during the life-time of the instrument (PQ and preventive maintenance).

Training and applications support

Professional applications support, basic and customized training courses, comprehensive applications literature.

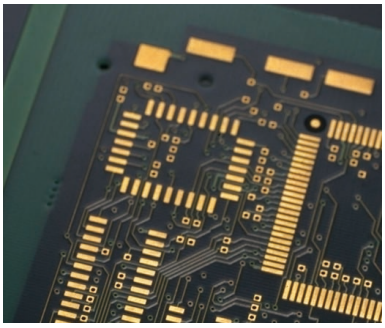
DMA Provides Answers in Many Application Fields

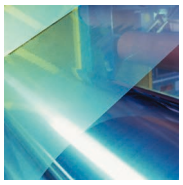
The DMA 1 is the ideal instrument to use for the dynamic mechanical analysis and characterization of materials, even in liquids or at specific relative humidity levels. It facilitates a large number of applications and provides valuable information in quality control and in industrial/academic research.

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.

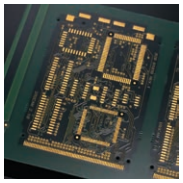
The materials most commonly analyzed are polymers such as thermoplastics, thermosets, elastomers and adhesives, metals, composites, paints and varnishes, foils and fibers, construction materials, pharmaceuticals, and foodstuffs. They can be solid or highly viscous.

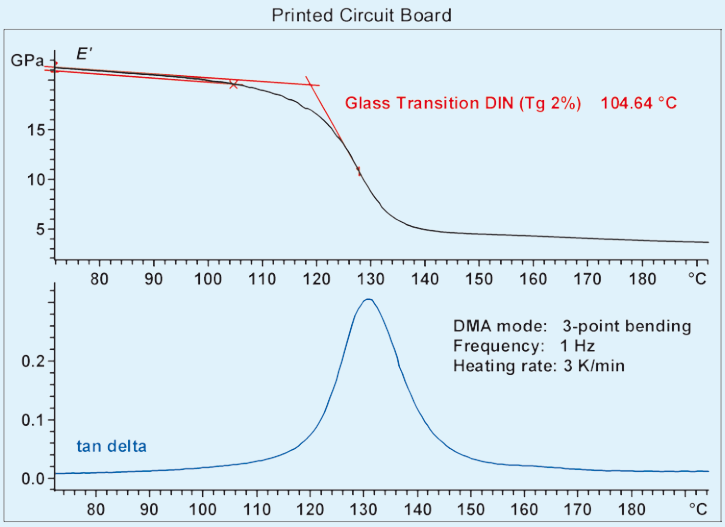




Effects and properties that can be characterized using the DMA 1 system:

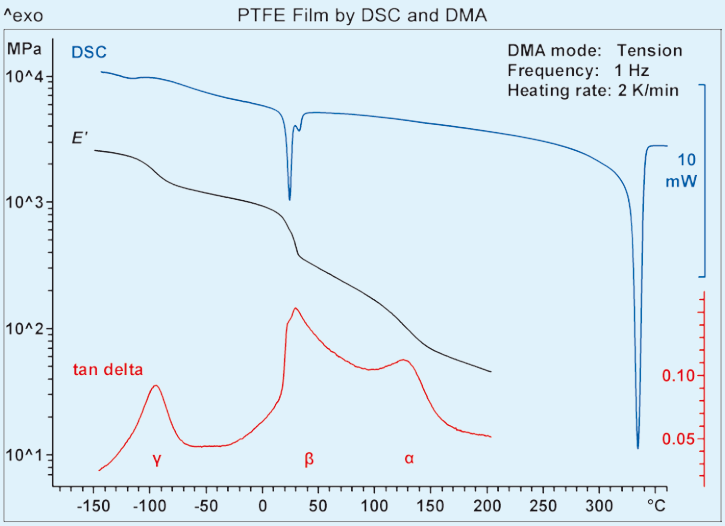
- Viscoelastic behavior
- Relaxation behavior
- Glass transition
- Mechanical moduli
- Damping behavior
- Softening
- Viscous flow
- Crystallization and melting
- Gelation
- Phase transformations
- Composition of blends
- Curing and polymerization reactions
- Material defects
- Effects due to fillers





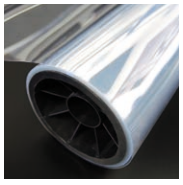
Composite materials

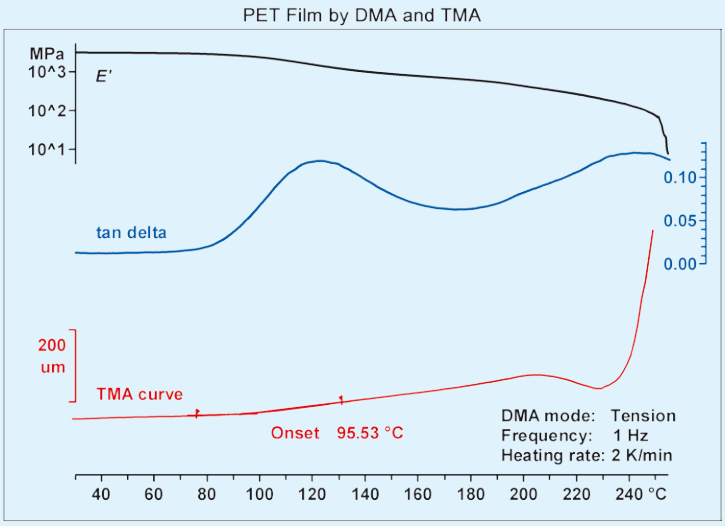
Composite materials made of filled cross-linked polymers have a high storage modulus at the temperature at which they are used. The modulus is usually determined by 3-point bending. The upper curve shows the storage modulus of a printed circuit board. The value measured at 70 °C and a frequency of 1 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

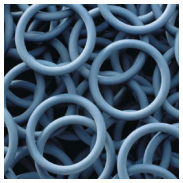
The DSC curve of PTFE shows phase transitions at about –100 °C and +30 °C as well as melting at 327 °C. The phase transitions can also be measured by DMA in the tension mode. The glass transition is then observed in addition at +130 °C. The transition temperatures measured by the two methods show excellent agreement.

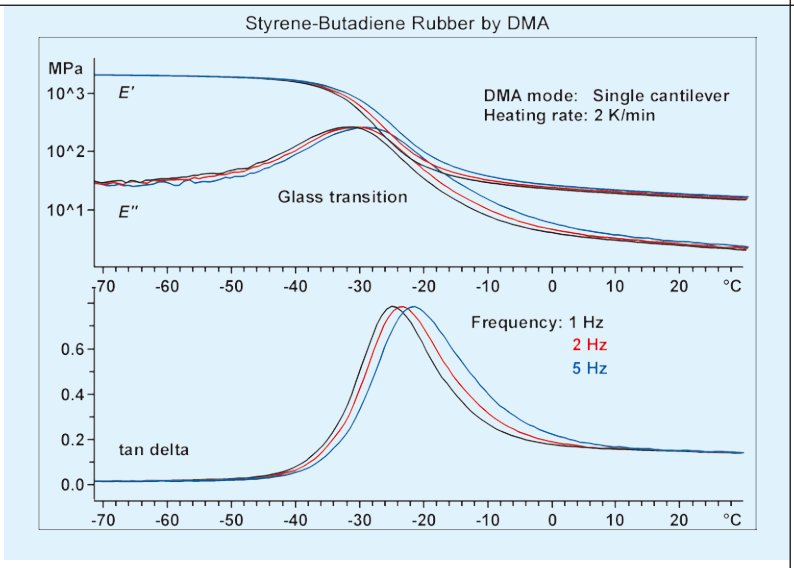




PET film

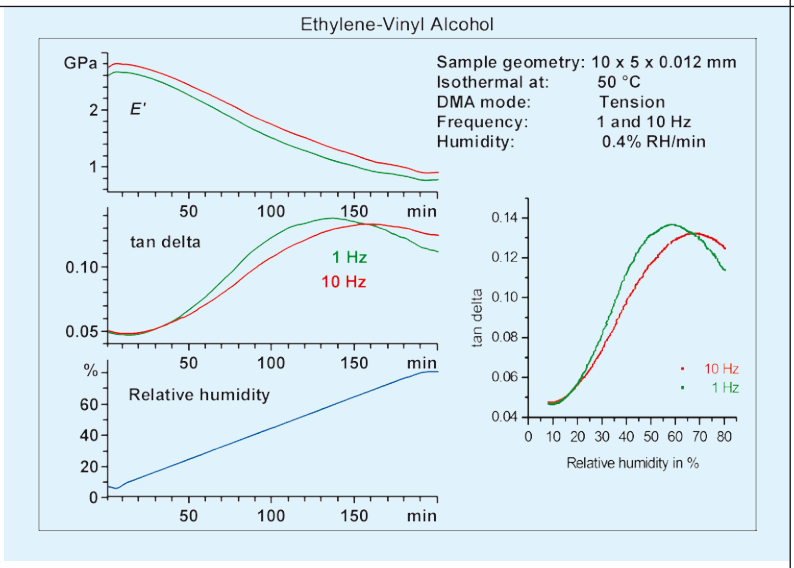
The diagram shows DMA curves of a PET film measured in the tension mode at 1 Hz. Curves like these are often used for quality control purposes. Due to crystallinity, the change in the modulus at the glass transition between 80 and 150 °C is only about one decade. The modulus shows a further decrease when melting begins 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 shrinks between 210 and 230 °C.





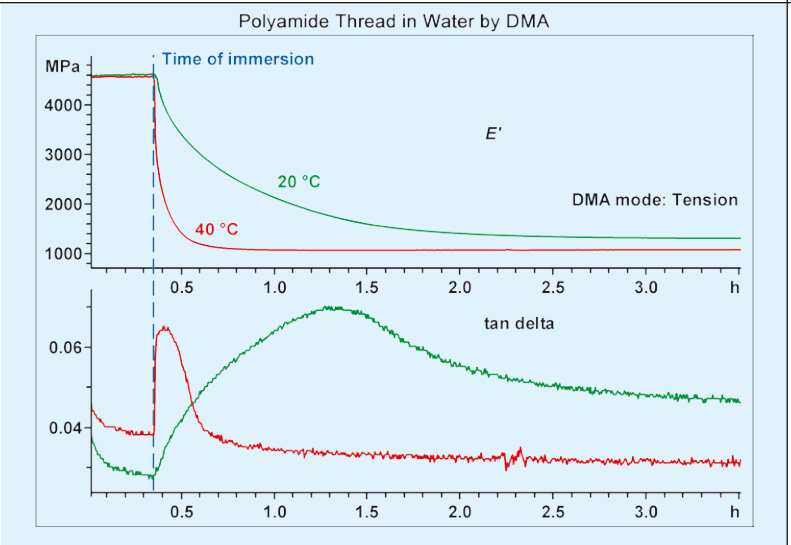
Styrene-butadiene rubber

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.



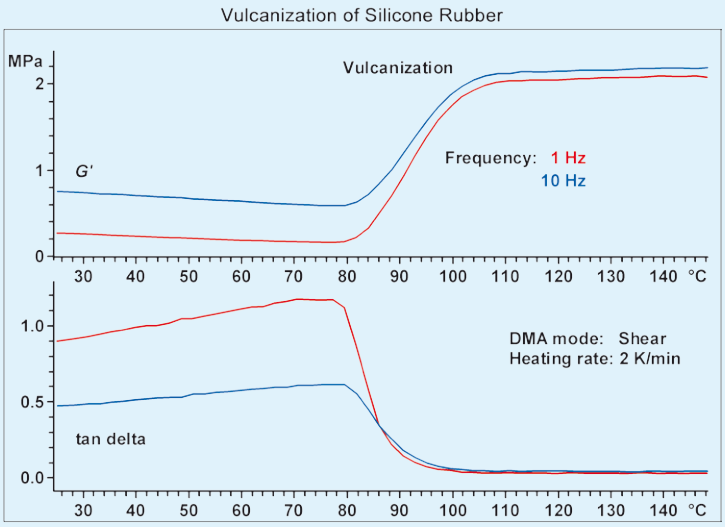
Effect of Relative Humidity

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.



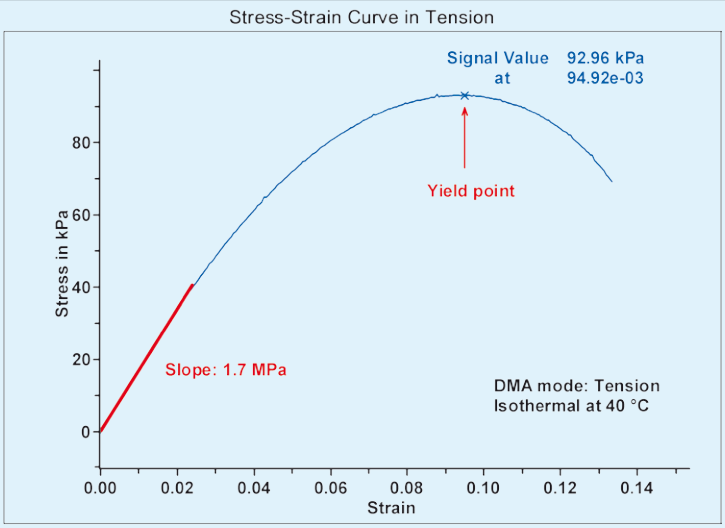
Polyamide thread in water

The mechanical properties of materials can change drastically in contact with liquids. 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 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.



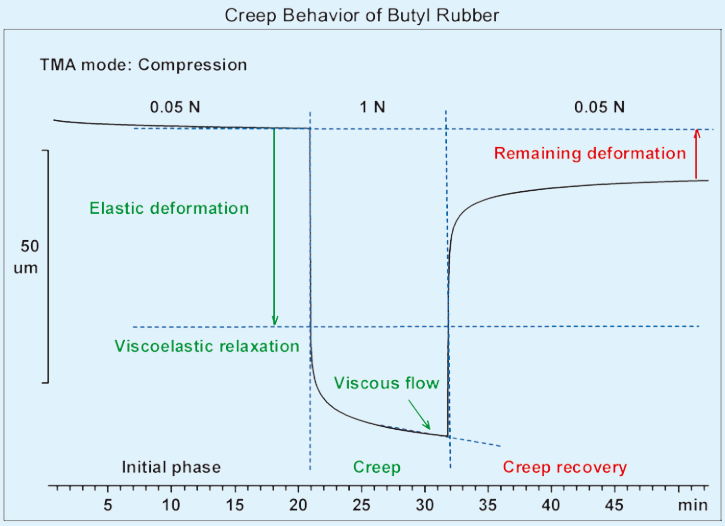
Silicone rubber

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 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.



Stress-Strain curves

The quasi-static mechanical properties over a large deformation range are often determined by measuring a stress-strain curve in the tension mode. The diagram shows the measurement curve of a sample of aged styrene-butadiene rubber (SBR) at 40 °C. At low levels of deformation, stress and strain exhibit a linear relationship. The slope of the curve in the linear range up to about 2% deformation is the elastic modulus, 1.7 MPa. In the non-linear range, the curve flattens off and the modulus decreases. The maximum is known as the yield point.



Creep behavior

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.

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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Quality certificate. Development, production and testing according to ISO 9001.

Environmental management system according to ISO 14001.

"European conformity". The CE conformity mark provides you with the assurance that our products comply with the EU directives.