Thermal Analysis Excellence

DMA/SDTA861^{e}

STAR^{e} System

Innovative Technology

Versatile Modularity

Swiss Quality

Dynamic Mechanical Analysis

Sets New Standards

METTLER TOLEDO

Precise Measurement Technology and Upgradeability

Dynamic mechanical analysis (DMA) is used to measure the mechanical and viscoelastic properties of a material as a function of temperature, time and frequency while it is subjected to an oscillating stress.

Features and benefits of the METTLER TOLEDO DMA/SDTA861e:

- **Measurement of both displacement and force** – results in a very accurate determination of moduli
- **Wide force range from 1 mN to 40 N** – allows very soft and very hard samples to be measured
- **Wide frequency range from 0.001 to 1000 Hz** – means that measurements can be performed under real conditions or more rapidly at higher frequencies
- **Innovative sample holders** – permit samples to be prepared and loaded externally
- **Sample temperature measurement** – allows accurate temperature adjustment as well as the measurement of thermal effects with SDTA
- **Extremely wide stiffness range** – a measurement can be performed over the entire temperature range of interest with one single sample holder

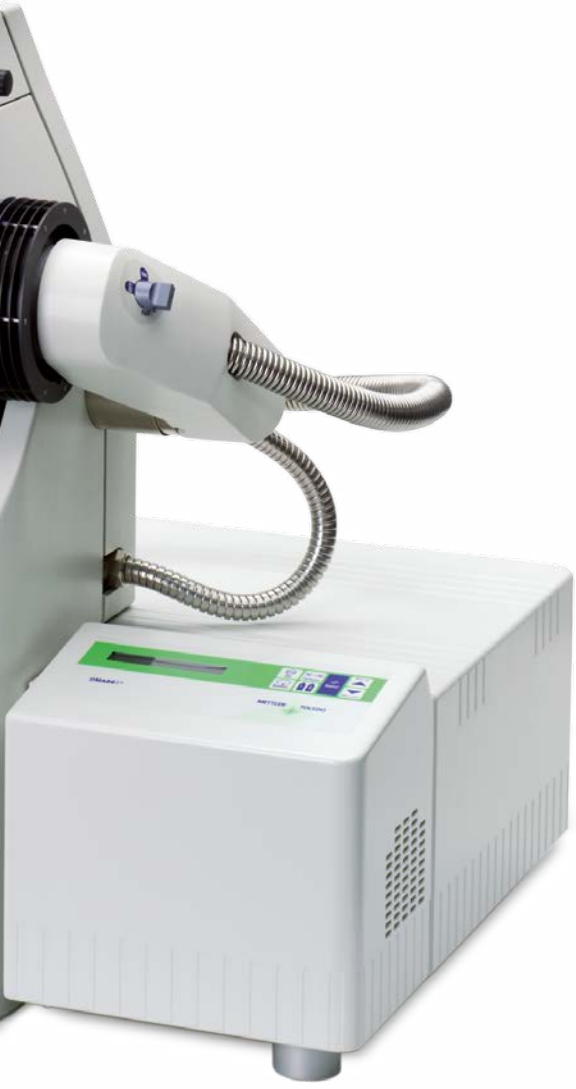
The modular design of the DMA/SDTA861e allows it to be expanded later on to meet new requirements.

Due to its revolutionary technology, the DMA/SDTA861e provides previously unattained performance and offers time-saving external sample clamping.

New Measurement Principle
Leads to Greater Accuracy

Wide frequency range from 0.001 to 1000 Hz
The frequency range has been extended to the kHz region for the first time ever in a DMA instrument. In shear mode, six decades are available. The region above 1 Hz is particularly interesting because it means that measuring times can be kept to a minimum.

Force measurement using a piezoelectric crystal
Force is measured directly by means of a piezoelectric crystal and is not set using a force-current graph as in conventional DMA instruments. The force measured is that which is actually applied to the sample. Compensation for frictional losses, membrane force and inertia is no longer necessary. Force measurement also allows the instrument to be operated in a way not possible with conventional DMAs:
The instrument can be operated under either force or displacement control and an intelligent automatic switched mode is also possible.



Simplified sample holder system
The METTLER TOLEDO sample holder system is a completely new design that helps save valuable instrument operating time. The samples are prepared and mounted externally in the sample holder. This can then be quickly installed in the instrument. The concept also allows you to change from one deformation mode to another without performing an adjustment. For example, you can prepare a bending experiment while a measurement in the tension mode is in progress.

Unmatched
Specifications

Greater stiffness range allows accurate measurements

The stiffness range is given by the displacement and force ranges. More than six decades are available with the DMA/SDTA861e. For the first time, samples can now be measured from the glassy state to the viscous range without having to change the sample geometry or the deformation mode.

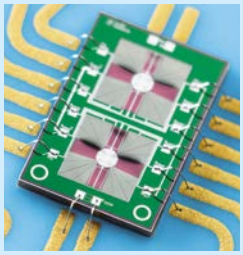
Thanks to the large stiffness range, soft samples can be measured equally well as very hard samples.

Complete thermal analysis system

A complete thermal analysis system consists of four basic measuring techniques. Each technique characterizes the sample in its own specific way. A complete picture that simplifies the interpretation is only obtained when all the results are combined. Besides the mechanical modulus (DMA), the heat flow (DSC, Flash DSC), the weight change curve (TGA), and the change in length (TMA) can be measured. All of these measurement variables change with temperature.

DMA

DSC



Flash DSC

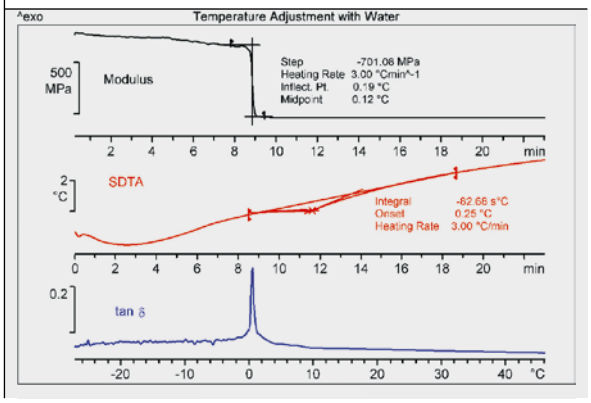
TGA

TMA

Sophisticated Solutions
for Maximum Accuracy

Sample temperature measurement using an additional sensor

Temperature adjustment is particularly important. An additional temperature sensor is located close to the sample. The sensor also allows thermal effects to be simultaneously measured by SDTA (Single DTA).



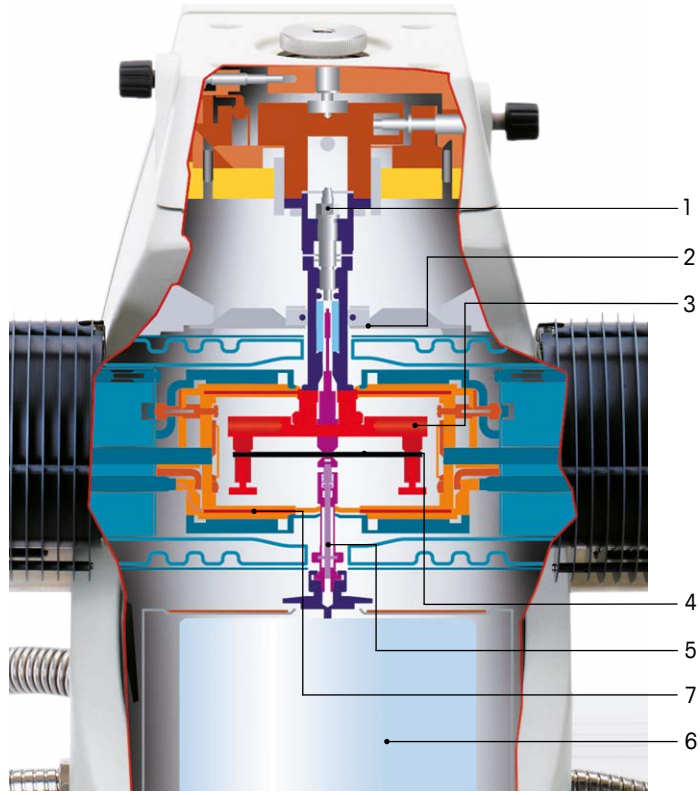
Adjustment traceable to reference standards

Temperature, force and displacement adjustments are based on defined reference standards. Temperature measurement close to the sample allows calibration with the melting points of pure substances. The displacement is adjusted using gauge blocks, and the force by means of a certified spring. An extremely precise spindle for the z-position capable of moving in defined 1-µm steps allows fully automatic length adjustment after the spindle has been adjusted with gauge blocks.

Unsurpassed Measurement Performance
Thanks to Innovative Solutions

New measuring system

The DMA/SDTA861e is very different to conventional DMA instruments. A massive stand results in a high natural resonance of the system of approximately 1500 Hz. The force sensor measures the force that acts on the sample. Modulus determination is very accurate because both force and displacement are measured. The measuring system can be adjusted using a three-dimensional alignment device. This ensures that the force applied to the sample is at an angle of exactly 90° and that no transverse forces result.



Key

1. Force sensor
2. LVDT (displacement sensor)
3. Clamp
4. Sample
5. Drive shaft
6. Linear motor
7. Furnace

Even more accurate displacement measurement

A special temperature-resistant LVDT allows measurements to be performed over a large measurement range with nanometer resolution. The LVDT is located close to the sample so that only the deformation of the sample is measured. This eliminates any effects due to deformation of the stand and also improves the accuracy of measurement of the phase shift. The reproducibility of the displacement measurement is improved by measuring the temperature of the LVDT sensor and correcting for the deviation.

Modular system

The DMA/SDTA861e has a modular design so that you can purchase the instrument configuration you need today and upgrade it at any time in the future to meet new requirements.
Stiffness range is ≥6 decades.

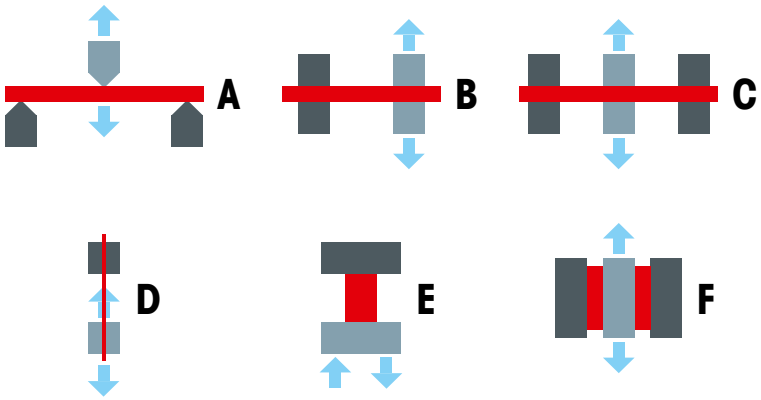
The following options are available:

• Maximum force	18 or 40 N
• Maximum frequency	200 or 1000 Hz

There is no interdependence between the two options.

Sample Holders

Simple, Ingenious and Timesaving



The various deformation modes

3-point bending (A): This bending mode is ideal for measuring extremely stiff samples, such as composite materials or thermosets, particularly below the glass transition temperature.
Single cantilever bending (B): This bending mode is suitable for samples that expand or contract in the longitudinal direction during the measurement. This applies primarily to thermoplastic samples.
Dual cantilever bending (C): Dual cantilever is the perfect mode for samples that would otherwise bend excessively under strong pretension. These are usually thermoplastics or thermosets.
Tension (D): Tension is the mode most suitable for films, fibers and thin bars and rods. The advantage is that sample clamping hardly affects the deformation.
Compression (E): This type of measurement is less suitable for the determination of absolute values of the modulus; valuable relative information can however be obtained when comparing soft materials like elastomers, pastes or foams.
Shear (F): The great advantage of the shear mode is that everything from viscous to very hard samples can be measured. This mode is therefore ideal for elastomers, thermoplastics, and thermosets.

Shear
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. A thermocouple mounted directly in the clamp measures the sample temperature so precisely that simultaneous heat flow effects can also be determined (SDTA).

Tension
In the tension mode, one end of the sample is fixed and the other is subjected to an oscillatory force. The sample must be prestressed to prevent it buckling during the oscillatory movement. The advantage of this mode is that sample clamping hardly affects the deformation.

Matching accessories
For mounting clamps, sample holders, and for performing calibrations, we supply an accessories box with all the materials needed for calibration (temperature and mechanical) and for installing samples (optional). This ensures that you quickly obtain precise measurement results.

Small clamping assembly

Deformation mode	Clamps
Tension	Small tension clamp: 9 mm
Shear	Small shear clamp
	Small shear clamp (for liquids)
	Small shear clamp (no surface structure)

3-point bending

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. A static preload force is applied to fix the sample in place. This mode interferes least with the actual sample measurement.

Single cantilever

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 to the moving part providing the oscillatory force.

Dual cantilever

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.

Compression

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. As a result, the geometry of the sample changes continuously and friction can occur at the contact surfaces. The sample can avoid the stress at its sides (uniaxial compression).

Dimensions
Length: 9 mm
Diameter: ≤14 mm Thickness: ≤6.5 mm

Large clamping assembly

Deformation mode	Clamps	Dimensions
Bending	Bending clamp for 3-point bending	Free length: 30 to 90 mm, max. length: 100 mm
	Single cantilever bending clamp	Free length: 10 to 40 mm, max. length: 100 mm
	Dual cantilever bending clamp	Free length: 20 to 80 mm, max. length: 100 mm
Tension	Tension clamp 5.5 mm	Length: 5.5 mm
	Tension clamp 10.5 mm	Length: 10.5 mm
	Tension clamp 19.5 mm	Length: 19.5 mm
Compression	Large compression clamp	Diameter: ≤14 mm, thickness: ≤6.5 mm

DMA Measurement
Force and Displacement Amplitudes

DMA theory

The different moduli can be calculated from the raw data namely the measured force and displacement amplitudes, F_a and L_a, and the phase shift δ:

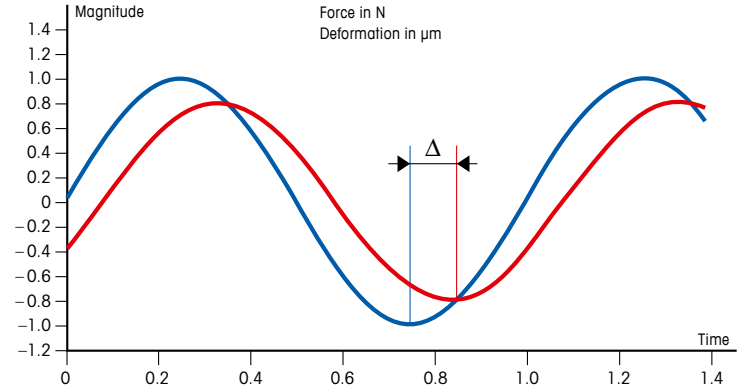
- Complex modulus M*, elastic modulus E* for bending, tension and compression or the shear modulus, G*, for shear deformation.
- Storage modulus M′ (proportional to the energy stored elastically and reversibly).
- Loss modulus M″ (proportional to the energy transformed into heat and irreversibly lost).
- Loss factor tan δ. Completely elastic materials exhibit no phase shift δ while purely viscous materials exhibit a 90° phase shift. The loss factor of viscoelastic materials is between 0 and infinity (δ = 90°).

The value of tan δ corresponds to the ratio of M″ to M′. The moduli are calculated from the measured stiffness according to the following equations:

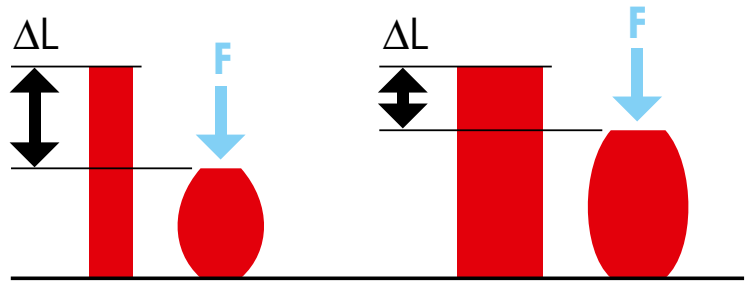
$$|M^*| = S^*g = \frac{F_a}{L_a} g \qquad S = \frac{F_a}{L_a}$$

where g is the geometry factor calculated from the sample dimensions. S is the stiffness of the sample (the actual measured quantity). The stiffness of the sample can thus be influenced by changing the sample geometry.

$$M' = |M^*| \cos \delta \qquad M'' = |M^*| \sin \delta \qquad \tan \delta = \frac{M''}{M'}$$

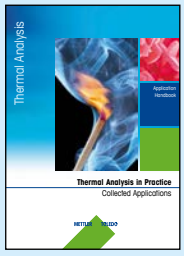


Force and displacement at a frequency f of 1 Hz. The time shift Δ results in the phase shift δ: $\delta = 2\pi f\Delta$.



F: Static force
ΔL: Deformation F

A thick sample is stiffer than a thin sample.



Important services

METTLER TOLEDO prides itself in supplying outstanding instruments and the support needed for you to be successful in your field of work. Our well-trained sales and service engineers are ready to help you in every possible way:

- Service and maintenance
- Calibration and adjustment
- Training and application advice
- Equipment qualification

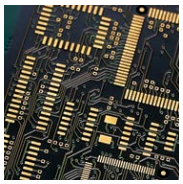
METTLER TOLEDO also provides comprehensive literature on thermal analysis applications.

Characterization of Materials
by Dynamic Mechanical Analysis

The DMA/SDTA861^{e} is the perfect solution when maximun accuracy is required and the material has to be characterized over a wide range of stiffness or frequency. Thanks to its wide dynamic force and displacement ranges and the wide variety of sample sizes and geometry, the DMA/SDTA861^{e} can be used to analyze practically all solid materials as well as medium and high viscosity fluids.

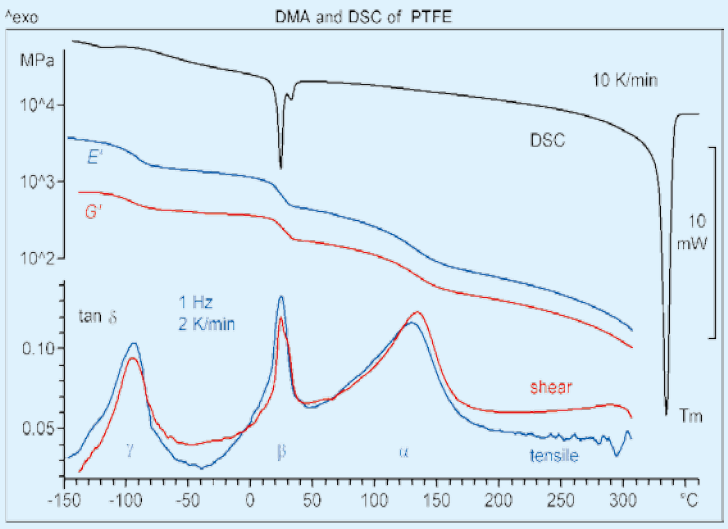
Materials are subjected to a wide range of different stresses in practical daily use. The most important factors are the frequency and intensity of the stress, the temperature, and the environment in which the load or stress is applied.

DMA analysis answers questions in application fields such as stability, practical use, processing steps, and damage and failure assessment. The materials tested can be both solid and highly viscous and include thermoplastics, thermosets, elastomers, adhesives, metals, composites, paints and varnishes, films and fibers, construction materials, pharmaceuticals, and foodstuffs.



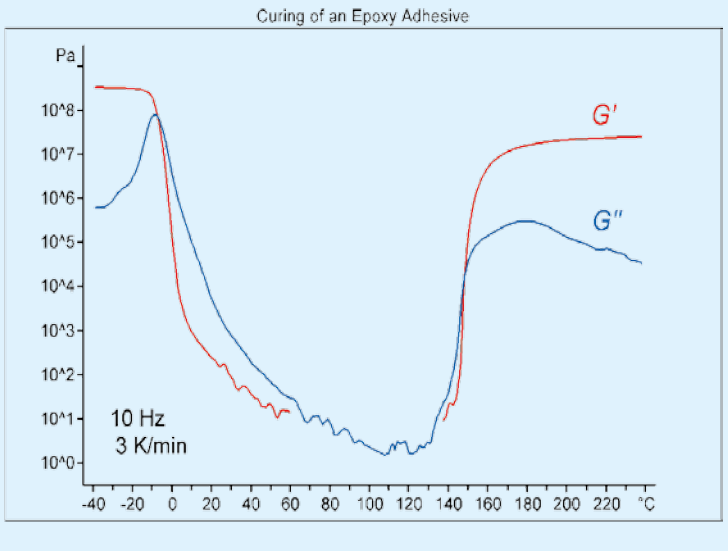
Effects and properties that can be characterized by the DMA/SDTA861^{e}:

- Viscoelastic behavior
- Relaxation behavior
- Glass transition
- Mechanical modulus
- Damping behavior
- Softening
- Viscous material flow
- Crystallization and melting
- Gelation
- Phase transitions
- Composition of blends
- Curing and polymerization reactions
- Material defects
- Effects caused by filler materials



Phase transitions of PTFE by DSC and DMA

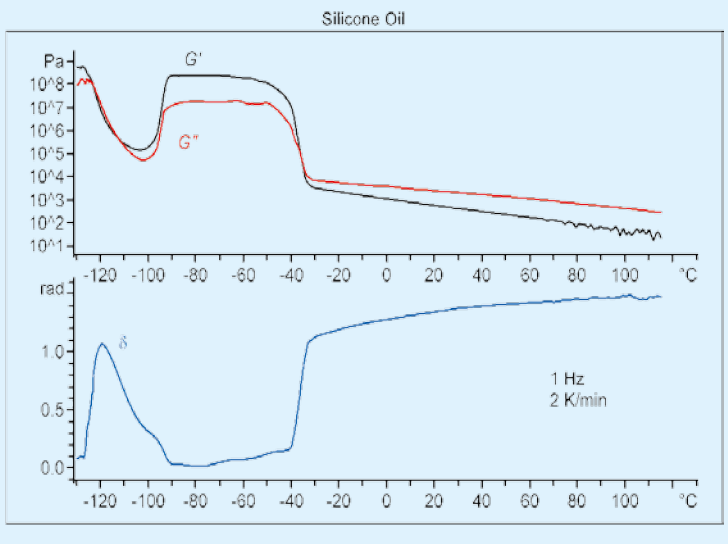
The DSC measurements of PTFE show the phase transitions at about –100 and 30 °C as well as melting at 327 °C. These transitions can also be measured by DMA. The effect at –100 °C is then much clearer. In addition, measurements in the shear and tension modes show the glass transition at 130 °C. The temperatures measured by the two methods agree very well. From Poisson's ratio, it follows that E′ is in principle always greater than G′.



Study of the curing of an epoxy amine system

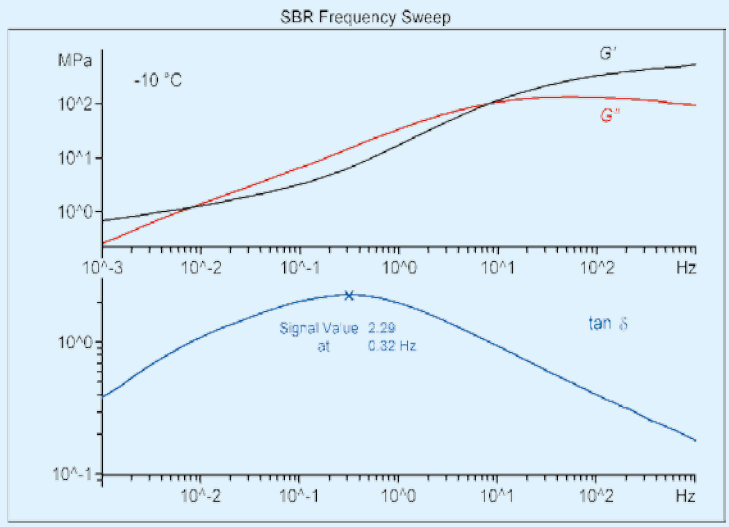
The curves show the curing of an epoxy amine system measured at 10 Hz using the shear sample holder for liquids. The sample was placed in the clamp, cooled to –50 °C, and then heated. At 0 °C, the resin changes from a hard glassy state to the liquid state. The storage modulus decreases by 7.5 decades.

From 130 °C onward, the modulus increases as a result of crosslinking reactions. The gel point is at 150 °C at the point of intersection of the G′ and G″ curves. The sample then becomes hard.



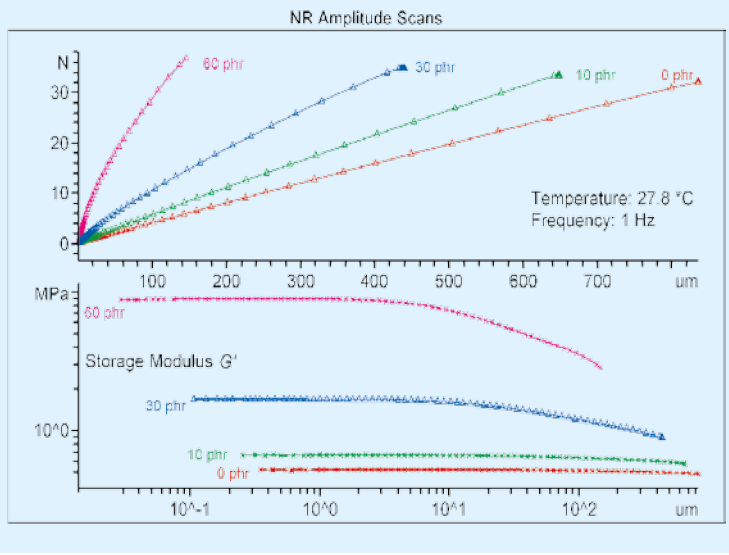
Characterization of silicone oil

A sample of silicone oil was prepared in the shear sample holder for liquids. This was then installed in the DMA, which had been cooled to –150 °C. On heating, the shock-cooled material exhibited a glass transition at –115 °C, crystallization at –100 °C, and melting at –40 °C, after which it was liquid (G″ > G′). At 120 °C, the phase angle almost reaches the limit of π/2 radians for a Newtonian fluid. The storage modulus changes by 7.5 decades.



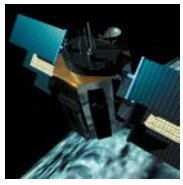
Frequency sweep of a styrene butadiene elastomer

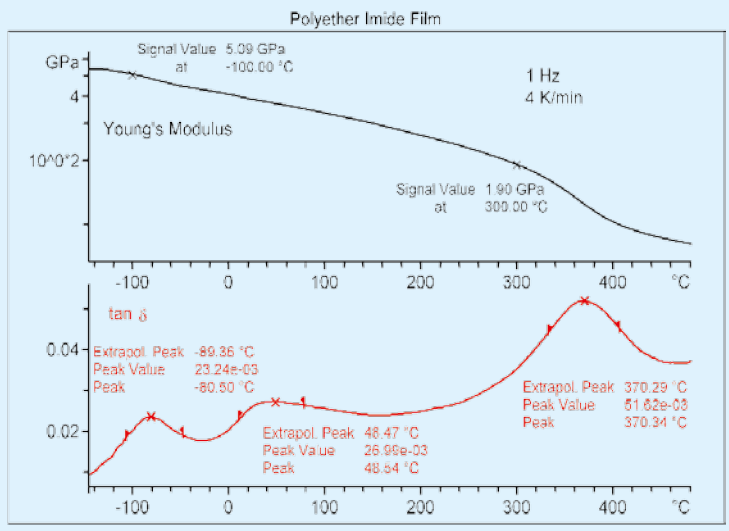
In practice, materials are stressed over a wide frequency range, the properties of the material change with frequency.
As shown in the measurement of SBR, the DMA/SDTA861^e can perform direct measurements over a very wide range of frequencies.
The diagram shows the main relaxation range at a temperature of –10 °C. The value of G′ changes by about 3 decades between 1 mHz and 1 kHz. The maximum value of the loss factor of 2.29 is at 0.32 Hz.



Displacement amplitude scans of natural rubber

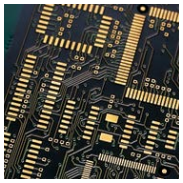
With filled polymers, the modulus increases with the filler content but decreases with increasing displacement amplitude. These effects are shown for four samples of filled NR with different carbon black contents. The measurements were performed using shear amplitudes of 30 nm to 1 mm. Information can be derived from the measurement curves on the linear range (Hooke's law) and the interaction between polymer and fillers.

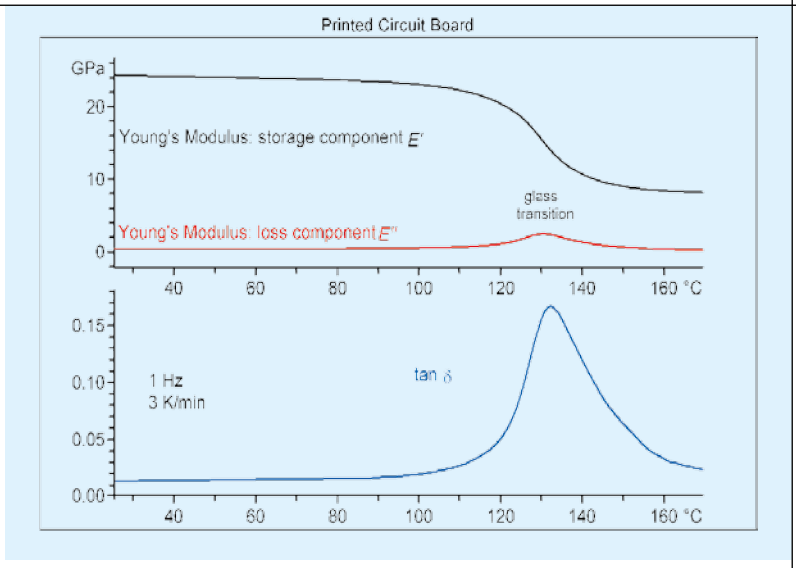




Characterization of a polyether imide film

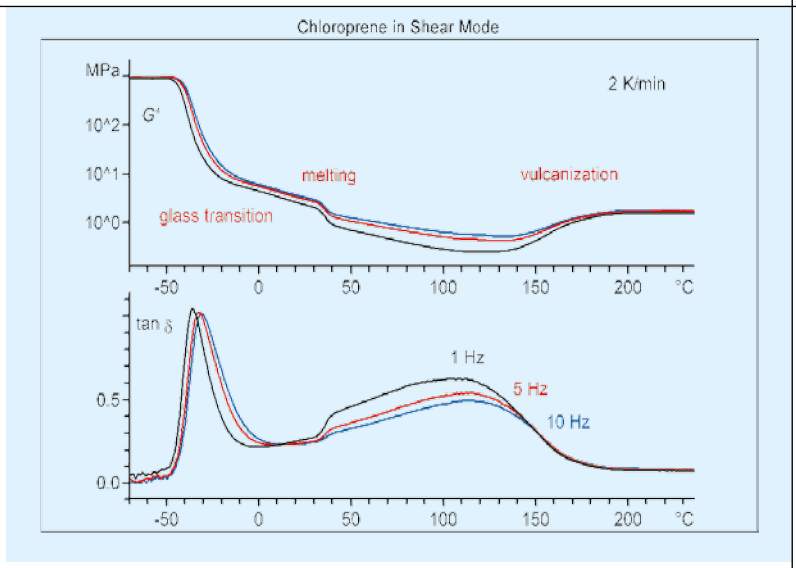
A 22-µm thick polyether imide film was measured over a wide temperature range between –150 and 480 °C in tension. At –100 °C the Young's modulus is about 5.1 GPa. Up until 300 °C, it decreases to 1.9 GPa. The glass transition is at a relatively high temperature (370 °C). In the loss factor curve, three relaxation regions with maxima at –82, 42 and 370 °C are observed. This information can be used to characterize the material.





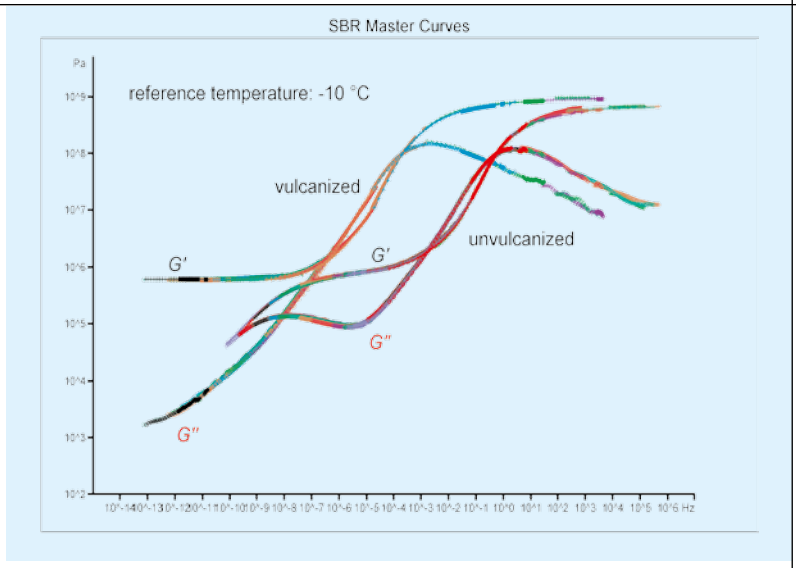
Determination of the modulus of a composite material

Composite materials made from filled, crosslinked polymers have a high storage modulus at their temperature of use. The modulus and transitions can be determined by 3-point bending measurements. The modulus determined at room temperature for the printed circuit board investigated was 24.2 GPa. Besides the absolute modulus, softening at the glass transition was also measured. The modulus decreases to 8.3 GPa. The step in the storage modulus is accompanied by peaks in the loss modulus and the loss factor.



Shear measurements of a chloroprene elastomer

Besides the measurement of mechanical properties, shear measurements of elastomers also enable thermal events to be analyzed. A chloroprene elastomer shows a glass transition, melting, and solidification due to vulcanization. The investigation of the vulcanization process is of great importance for quality assurance and damage and failure analysis because material failure can often be traced back to inadequate crosslinking.



Master curves of styrene butadiene elastomers

Mechanical spectroscopy over a wide frequency range provides detailed information on material properties. The DMA's excellent temperature stability and accuracy together with the possibility of measurements at high frequencies allow the precise and rapid construction of master curves. The figure displays master curves of unvulcanized and vulcanized SBR. Besides information on dynamic behavior, conclusions can also be drawn about the molecular structure and network.

DMA/SDTA861^{e} Specifications

Temperature	
Range	–150 to 500 °C
Technical resolution	0.003 K
Accuracy	0.5 K

Force	
Range	0.001 to 40 N, (18 N or 40 N)
Technical resolution	0.15 mN (0 to 5 N), 1.5 mN (0 to 50 N)
Sensitivity	1 mN

Displacement	
Range	±1.6 mm
Technical resolution	0.6 nm
Sensitivity	5 nm

Stiffness	
Range	10 to 10^{8} N/m
Precision	0.2%

Tan delta	
Range	0.0001 to 5000
Technical resolution	0.00001
Sensitivity	0.0001

Frequency	
Range	0.001 to 1000 Hz (*)
Technical resolution	0.00001
Frequency increments (Δf)	0.0001
Frequency modes	• Linear or logarithmic • Frequency series • Multi-frequency

Deformation modes	
3-point bending Dual cantilever Single cantilever	Length: 30 to 90 mm, length: 20 to 80 mm Width: <15 mm, thickness: <5 mm Max. Sample length: 100 mm
Bending stiffness range	1 to 10^{6} N/m
Shear	Diameter: ≤15 mm, thickness: ≤6.5 mm
Shear stiffness range	1 to 10^{8} N/m
Tension	Length: 19.5 mm, 10.5 mm, 9.0 mm, 5.5 mm Width: ≤7 mm, thickness: ≤3 mm
Tensile stiffness range	1 to 10^{7} N/m
Compression	Diameter: ≤20 mm, thickness: ≤9 mm
Compressive stiffness range	1 to 10^{7} N/m

(*) Depending on the deformation mode and the sample itself, the maximum frequency can be lower. The maximum frequencies vary (shear: 1000 Hz, bending: 300 Hz, tension: 300 Hz and compression: 300 Hz)

Approvals

IEC/EN61010-1:2001, IEC/EN61010-2-010:2003
CAN/CSA C22.2 no. 1010.1-92
UL Std. no. 3101-1

EN61326-1:2005 / EN61326-1:2006 (class B)
EN61326-1:2005 / EN61326-1:2006 (Industrial environments)
FCC, Part 15, class A (Declaration)
AS/NZS CISPR 11, AS/NZS 61000.4.3

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