# Thermal Analysis Excellence



#### DMA/SDTA861°

STAR <sup>e</sup> System
Innovative Technology
Versatile Modularity
Swiss Quality



## Dynamic Mechanical Analysis Sets New Standards



# Precise Measurement Technology and Upgradability

Dynamic mechanical analysis (DMA) is used to measure the mechanical and viscoelastic properties of materials as a function of temperature, time and frequency when they are subjected to a periodic stress. The types of materials that can be analyzed with this technique include thermoplastics, thermosets, composites, elastomers, ceramics and metals.

Features and benefits of the DMA/SDTA861<sup>e</sup>:

- 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 Hz 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

And, thanks to its modular design, the DMA/SDTA861<sup>e</sup> can be upgraded later to meet future needs.

DMA provides quantitative and qualitative information that is of great value to process and application engineers, materials research scientists and chemists, such as:

- Young's modulus and shear modulus
- Damping characteristics and viscoelastic behavior
- Polymer structure and morphology
- Flow and relaxation behavior



#### 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.

## Wide stiffness range allows accurate measurement

The stiffness range is given by the force and displacement ranges. With the DMA/SDTA861<sup>e</sup>, more than six decades are available. This means that it is now possible to measure samples from the viscoelastic to the glassy state in one experiment without having to change the sample geometry or the deformation mode. There is no need to correlate data from two different experiments.

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

#### Highly accurate displacement measurement

A special temperature-resistant LVDT measures the displacement over an extremely wide 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 possible deformation of the stand and improves the accuracy with which the delay time (i.e. phase shift) between the force and displacement is determined. The reproducibility of the displacement measurement is further improved by measuring the temperature of the LVDT sensor. Deviations from the reference temperature are then compensated.

# **Unmatched Specifications**

- Direct measurement of force and displacement
- Force range up to 40 N
- Frequency range from 1 mHz to 1 kHz
- Innovative sample holders
- Direct measurement of sample temperature
- Wide stiffness range



## Force measurement using a piezoelectric crystal

Force is measured directly with a piezoelectric crystal and not set using a force-current graph as in conventional DMAs. This means that 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 mode not possible with conventional DMAs – namely under either force or displacement control.



An automatic intelligent softwareswitched mode is also possible.

#### Highest force range available

The force range together with the displacement range determines the stiffness range of a DMA instrument. Large, or small forces are required depending on the nature of the sample and the measurement temperature. The DMA/SDTA861<sup>e</sup> provides forces from a 1 mN to 40 N.

#### Innovative, easy-to-use 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 outside the instrument. The sample holder can then be quickly installed in the instrument. This concept also allows you to change from one deformation mode to another without adjustment.

You can, for example, prepare a bending experiment while a measurement in tensile mode is in progress.

## Signal evaluation with special algorithm

New technical solutions have also been realized in signal evaluation. A special Fourier algorithm developed at the University of UIm is used for this purpose. Its key features are that it is very fast and includes drift compensation. This allows the force and displacement amplitudes to be determined very accurately, which in turn results in very accurate modulus values.

#### Automatic offset control

A special Auto-Offset control has been developed for measurements in which pretension or predorfomation has to be applied to the sample. It is based on the measurement of force and displacement (application of a static force). The detection of a distorted sinus function in a measurement indicates that insufficient static force is being applied. The controller then automatically corrects this.

#### New measurement principle ensures high accuracy

The operating principles of the DMA/ SDTA861° are in many respects very different to those of the current generation of conventional DMA instruments. The massively built stand results in the system having an intrinsic resonance frequency of about 1500 Hz, well above the measurement frequencies used. The sample itself is fixed directly to the force sensor so the force actually applied to the sample is measured. This technique was developed at the Institute of Dynamic Material Testing, University of Ulm, Germany, where it has been in successful use for several years and has undergone continuous development. The modulus is calculated from the ratio of force to displacement multiplied by a geometry factor given by the sample dimensions. The modulus can be determined with areat accuracy because both force and displacement are measured. The fixed and moving parts can be adjusted via a three-dimensional

alignment device so that the force is applied at an angle of exactly 90° to the sample and no errors due to transverse forces occur.

## Furnace volume reduced to a minimum

The DMA/SDTA861<sup>e</sup> furnace consists of two symmetrical parts. This design allowed the volume of the furnace to be reduced to a minimum and eliminates undesired temperature gradients. The temperature of each half of the furnace is continuously measured and any deviations are immediately corrected. The lithographically deposited heater tracks are wrapped around a ceramic tube in meander form. This neutralizes any electromechanical forces that might otherwise affect the sample.

Surrounding the heater is the liquid nitrogen heat exchanger in which liquid nitrogen evaporates. During the actual measurement, the cooling effect is transferred to the sample via the furnace atmosphere so that the measurement results are not affected by the inflow of cold gas. For rapid cooling, cold liquid nitrogen vapor can be blown directly into the furnace chamber. The high cooling rates achieved allow you to start a new measurement more quickly.

The furnace atmosphere can also be switched (e.g. from an inert to an oxidative atmosphere) by means of a gas inlet valve. The furnace opens at the stroke of a key. The two furnace arms can be swung to the rear in order to gain full access to the DMA measuring system.

## Adjustment traceable to reference standards

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

#### Sample temperature measurement with additional sensor

Particular attention was paid to temperature adjustment. A special temperature sensor is located close to the sample. This sensor also allows the simultaneous measurement of enthalpy changes with DTA (single DTA).

#### Powerful software tools

The new DMA functions have been integrated into the already comprehensive and well-proven STAR<sup>e</sup> software. The software operates the instrument and evaluates the data. Important DMA-specific enhancements include:

- Pre-check of sample clamping at room temperature and the start temperature
- Evaluation with linear or logarithmic coordinate systems
- Master curve technique (timetemperature superposition)

In the method you can define what

should be done to the sample before the measurement.

For example, if the oscillatory force is applied to the sample before the measurement, you can check whether the sample has been properly clamped. This possibility is available both at room temperature and at the start temperature of the measurement.

In dynamic mechanical measurements, the modulus often changes by several orders of magnitude. The logarithmic display of the coordinate system is therefore extremely valuable. The curves can of course still

#### Programming different methods

The STAR<sup>e</sup> software enables you to program different types of methods:

- Isothermal and dynamic temperature segments
- Single, series and multi-frequency oscillations
- Frequency sweeps

You construct your temperature program from isothermal and dynamic segments. Independent of this, you can define the oscillatory force or displacement applied to the sample.

The best qualitative results are obtained with a single frequency. If you are interested in frequency



be evaluated with this presentation. With some samples, the time (frequency)-temperature superposition principle can be applied. This means that the behavior of a material at high frequencies is similar to that of the sample measured at lower temperatures with lower frequencies. This principle is the basis of the master curve technique.

It allows the frequency behavior of a sample to be extrapolated into regions where direct instrumental measurement is impossible. Information can therefore be obtained on the behavior of the material at very high frequencies or very low temperatures. behavior, you have two possibilities: you choose either a series of frequencies that is continually repeated during the measurement, or you use the multi-frequency function, which allows simultaneous measurement of four frequencies (4 frequencies within a decade in the ratio 1:2:5:10). In both cases, a curve is obtained for each frequency. This allows frequency-dependent effects (e.g. glass transitions) to be easily distinguished from effects that do not depend on frequency (e.g. melting).

The possibility of performing frequency sweeps isothermally is also very interesting. Measurements at



constant temperature eliminate possible temperature gradients within the sample. The sample is measured over a range of frequencies in one run. You are perfectly free in the choice of the frequency steps (linear steps or logarithmic steps). This procedure is normally used in the master curve technique.

Another interesting measurement mode is the force or displacement amplitude sweep. Here, the force or displacement amplitude is changed in predefined steps at constant temperature. This enables you to determine the linear range of a sample.



#### **DMA** theory

The desired modulus can be calculated from the measured force and displacement amplitudes, *Fa* and *La* and their phase shift,  $\delta$ :

- Complex modulus *M*<sup>\*</sup>, Young's modulus *E*<sup>\*</sup> for tension or the shear modulus *G*<sup>\*</sup>.
- Storage modulus M' proportional to the energy stored elastically and reversibly.
- Loss modulus M<sup>"</sup> proportional to the energy transformed into heat and irreversibly lost.
- Loss factor (tan  $\delta$ ). With completely elastic materials, no phase shift,  $\delta$ , occurs; completely viscous materials exhibit a phase shift of 90°. The loss factor of viscoelastic materials is between 0 and infinity ( $\delta = 90^{\circ}$ ).

The value of tan  $\delta$  corresponds to the ratio of M'' to M'. The moduli are calculated from the measured stiffness according to the following equations:

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

where g is the geometry factor calculated from the sample dimensions. S is the stiffness of the sample. The stiffness of the sample can be influenced by a change of sample geometry.

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



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



# **The STAR**<sup>e</sup> **System** – the Flexible Solution

- Sound investment
- Modular system
- 6 deformation modes



#### A sound investment

You begin with the instrument configuration that meets your current needs. Later on, you can upgrade the system with options or accessories to satisfy your new requirements. It's a sound investment, whatever you decide on.

Control of external accessories

You can connect a gas controller to the DMA/SDTA861° to perform measurements under different atmospheric conditions.





#### Straightforward computer control

The entire measurement method can be created with a few simple commands on the computer. Additional information and changes are easily entered. At the same time, the program checks whether the entries are correct. All the data generated are automatically stored in a relational database and can be accessed for documentation purposes, even years later. A large number of software options are available to simplify your daily work.

#### Direct module keypad control

The module can be operated from the PC or via the instrument keypad. With the DMA in particular, it is very convenient to be able to enter commands directly via the module keypad or read off certain measurement quantities directly on the module display.

#### Modular system

The modular design of the DMA/SDTA861<sup>e</sup> has many advantages. It means you can purchase the instrument configuration you need today and upgrade it according to your requirements at any time in the future. The options available are:

- Maximum force: 12, 18 or 40 N
- Maximum frequency: 200 or 1000 Hz
- Stiffness range: 4 or  $\geq$  6 decades

There is no interdependence between the three options.



(3)

(5)

#### Six different deformation modes The DMA/SDTA861° has six different

deformation modes:

- Shear (1)
- 3-point bending (2)
- Dual cantilever (3)
- Single cantilever (4)
- Tension (5)
- Compression (6)

(2)

(6)

# **Sample Holders** Simple, Ingenious and Timesaving



#### Shear

The shear mode has been somewhat neglected in the past because instruments were not properly designed to use it.

Two identical samples are clamped symmetrically between two outer fixed parts and the central moving part providing the oscillatory force. The great advantage of the shear mode is that everything from viscous to hard samples can be measured. It is suitable for elastomers, thermoplastics and thermosets. The shear clamps guarantee a homogeneous temperature environment. A thermocouple can be installed in the shear clamp. This measures the sample temperature so accurately that even simultaneous enthalpy changes can be measured (SDTA).

Diameter: $\leq 14 \text{ mm}$ Thickness: $\leq 6.5 \text{ mm}$ 

#### 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 by a moving knife-edge. A preload force is applied to fix the sample in place. This way of mounting samples interferes least with the actual sample measurement.

The 3-point bending mode is particularly suitable for hard samples such as reinforced thermosets, composites, metals and alloys. Max. sample length: 100 mm Free sample length: 30 – 90 mm in 5-mm steps, freely selectable

#### **Dual cantilever**

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

This mode is especially suitable for samples that would otherwise bend excessively under static stress. Examples of this are thermoplastics and thermosets.

Max. sample length: 100 mm Free sample length: 20 – 80 mm in 5-mm steps, freely selectable



#### Single cantilever

The single cantilever mode is very similar to the dual cantilever mode, except that only one side of the sample is fixed. The middle of the sample is clamped to the movable part of the system providing the oscillatory force.

This mode is suitable for samples that expand or shrink strongly along their length during the measurement. This is in particular the case with thermoplastic samples. Max. sample length: 100 mm Free sample length: 10 – 40 mm in 2.5-mm steps, freely selectable

#### Tension

In the tensile mode, one end of the sample is fixed and movable part of the system providing the oscillatory force is attached to the other end. Pretension is applied to prevent the sample from bending or buckling during the oscillation.

This mode is most suitable for films, fibers and thin bars. The advantage is that the clamping of the sample has practically no influence on the deformation of the sample. Free sample lengths: 5.5 mm, 9.0 mm, 10.5 mm and 19.5 mm

	Small clamping assembly	Large clamping assembly
Bending		3-point bending clamp
		Dual cantilever clamp
		Single cantilever clamp
Tension	Small tension clamp (9 mm)	5.5 mm tension clamp
		10.5 mm tension clamp
		19.5 mm tension clamp
Shear	Small shear clamp	
	Small shear clamp (liquid)	
	Small shear clamp (without texture)	
Compression		Large compression clamp

#### Compression

In the compressional mode, the sample is clamped between a fixed part and the moving part providing the oscillatory force. The sample is compressed statically and then subjected to an alternating load. The geometry of the sample changes continuously due to the applied stress. Friction occurs at the contact surfaces to the sample. At the sides the sample can escape the stress so that the volume continuously changes (uniaxial compression).

This type of measurement is therefore not suitable for absolute determinations of the modulus. Valuable comparative data for soft materials (pasty materials, elastomers and foams) can however be obtained. Diameter:  $\leq 20$  mm Thickness:  $\leq 9$  mm

# **Characterization of Materials** by Dynamic Mechanical Analysis

Materials are subjected to a varietv of different mechanical stresses in practical daily use. The most important factors are the frequency and the intensity of the stress, and the temperature. For example, in a powder coating process, the material is first uniformly distributed over the surface. On heating, a homogeneous liquid film is first produced and no droplets should be formed on the surface. The film finally cures. The finished coating must on the one hand be able to endure mechanical stress arising from the thermal expansion of the substrate without any cracks developing. In addition, it must also withstand sudden blows at low temperatures without flaking or damage.

A number of mechanical properties are of vital importance for the manufacture, storage, processing and application of materials. Knowledge of the material's viscoelastic behavior over a wide frequency and temperature range allows information to be gained about mechanical properties relevant to its application, and also about molecular rearrangement and structures. This opens up a large number of applications for dynamic mechanical analysis (DMA) such as:

- the determination of material properties
- material and process optimization as well as
- quality control and
- the analysis of material failure.

The wide dynamic range of various quantities that are directly measured and the large variation of sample size and sample geometry make the DMA/SDTA861e an excellent choice for the measurement of practically all solid materials and high to medium viscous liquids. Important application areas include:

- thermoplastics
- thermosets
- elastomers
- adhesives
- paints and lacquers
- films and fibers
- composites
- foodstuffs
- pharmaceutical
- fats and oils
- ceramic materials
- constructional materials and
- metals





The application examples described in the following pages demonstrate the outstanding performance of the DMA/SDTA861e. In fact, however, they represent just a few of the multitude of possibilities that the instrument offers.

The following list summarizes the effects and properties that can be investigated with DMA:

- viscoelastic behavior
- relaxation behavior
- glass transition
- mechanical modulus
- damping behavior
- softening
- viscous flow
- crystallization and melting
- phase separations
- gelation
- changes in morphology
- composition of blends
- filler activity
- material faults
- curing reactions
- cross-linking reactions
- vulcanization systems

The DSC measurements of PTFE show the phase transitions at about -100 °C and 30 °C as well as melting at 327 °C. These transitions can also be measured in the DMA.

The effect at -100 °C is then much clearer. Measurements in the shear and tensile modes in addition show the glass transition at 130 °C.

The temperatures measured by the two techniques agree very well. From Poisson's ratio, it follows that E' is in principle always greater than G'.





The curves show the curing of an epoxyamine system measured at 10 Hz using the shear sample holder for liquids. The sample was placed in the clamp assembly that had been 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 cross-linking reactions. The gel point is at 150 °C at the intersection of G' and G''. The sample then becomes hard.





With shear measurements, the entire mechanical behavior of thermoplastics can be determined in one single measurement. This is shown using shock-cooled PET as an example. Secondary relaxation ( $\beta$ -relaxation) occurs at -70 °C. The main relaxation (glass transition) is observed at 80 °C. The modulus increases due to cold crystallization at about 110 °C. On further heating, recrystallization and melting of the crystallites takes place. *G*' changes from 10<sup>9</sup> to  $5 \cdot 10^2$  Pa.





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





In practice, materials are stressed over a wide frequency range, whereby the properties of the material change with frequency. As shown in the measurement of SBR, the DMA/SDTA861° can perform direct measurements over a very wide range of frequencies. The diagrams show 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.





With filled polymers the modulus increases with the filler content, decreases however 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.





A 22-µm thick polyether imide film was measured over a wide temperature range between -150 °C 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 °C, 42 °C and 370 °C are observed. This can be used to characterize the material.









PVC was measured in Single Cantilever mode between -90 °C and 110 °C. Two frequency- dependent relaxation regions are observed. The secondary relaxation with a tan  $\delta$  peak at -30 °C is broad and shows a small step in the *E*<sup>2</sup>modulus. This effect determines the mechanical behavior at low temperatures. The main relaxation corresponds to the glass transition. The sample softens and the modulus decreases by 3 decades to about 10 MPa.

Composite materials made from filled cross-linked polymers have a high storage modulus at the temperature of use. The modulus determined for the printed circuit board in a 3-point bending experiment 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.







Besides the measurement of mechanical properties, shear measurements on elastomers also enable thermal events to be analyzed. The chloroprene sample measured 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 analysis because material failure can often be traced back to inadequate cross-linking.

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 on the molecular structure and network.





A videotape is based on stretched PET with a high value of Young's modulus at room temperature. The sample measured gave a value of 7.3 Gpa. In tension, two relaxation regions were detected. The larger at 100 °C corresponds to the glass transition of PET. The surface coating causes the smaller relaxation region at 50 °C. The high degree of crystallinity is the reason for the low change in E at the glass transition.





Films and fibers can be measured in tensile mode. In the example shown, a 35-µm thick epoxy-based film is analyzed. At the glass transition between 90 °C and 120 °C, *E'* decreases from 2 GPa to 30 MPa. The glass transition is accompanied by a peak in the loss modulus, *E''*, and in the loss factor, tan  $\delta$ .

### **DMA/SDTA861** Specifications

Temperature				
Range	–150 500 °C			
Technical Resolution	0.003 K			
Accuracy	0.5 K			
Force				
Range	0.001 40 N, (12, 18 or 40 N)			
Technical Resolution	0.15 mN (0 5 N), 1.5 mN (050 N)			
Sensitivity	1 mN			
Displacement				
Range	±1.6 mm			
Technical Resolution	0.6 nm			
Sensitivity	5 nm			
Stiffness				
Range	10 10 <sup>8</sup> N/m			
Precision	0.2 %			
Tan delta				
Range	0.0001 100			
Technical Resolution	0.00001			
Sensitivity	0.0001			
Frequency				
Range	0.001 1000 Hz (*)			
Technical Resolution	0.00001			
Accuracy ( $\Delta$ f)	0.0001			
	Logarithmic and linear scans			
Modes	<ul> <li>Multi-frequency (sequentially)</li> </ul>			
	<ul> <li>Multi-frequency (simultaneously)</li> </ul>			
Measurement modes				
Bending 3-point	Length: 30 90 mm, Length: 20 80 mm			
Dual cantilever	Width: < 15 mm, Thickness: < 5 mm			
	Max. sample length: 100 mm			
Stiffness range bending	10 10 <sup>6</sup> N/m			
Shear	Diameter: $\leq 15$ mm, Thickness: $\leq 6,5$ mm			
Stiffness range shear	10 10 <sup>8</sup> N/m			
Tension	Length: 19.5, 10.5, 9.0, 5.5 mm			
	Width: $\leq 7$ mm, Thickness: $\leq 3$ mm			
Stiffness range tension	10 10' N/m			
Compression	Diameter: $\leq$ 20 mm, Thickness: $\leq$ 9 mm			
Stiffness range compression   10 10 <sup>7</sup> N/m				
Approvals				
IEC/EN61010-1:2001, IEC/EN61010-2-010:2003				
UAN/USA UZZ.Z NO. 1010.1-92				
ENG1326-1:2005 / ENG1326-1:2006 (class B)				
FCC. Part 15. class & (Declaration)				
AS/NZS CISPR 11 AS/NZS 61000 & 3				
AS/NZS CISPR 11, AS/NZS 61000.4.3				

(\*) depending on the deformation mode and the sample itself, the maximum usable frequency may be lower. The maximum frequencies are different (shear 1000 Hz, bending 300 Hz, tension 300 Hz, compression 300 Hz).

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For more information

according to ISO 14001.

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