



# TA INSTRUMENTS

Thermal Analysis



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**DMA**  
Q800



Dynamic Mechanical Analysis

# Q800



The Q800 is the world's best-selling DMA, for very good reasons. It utilizes state-of-the-art, non-contact, linear drive technology to provide precise control of stress, and air bearings for low friction support. Strain is measured using optical encoder technology that provides unmatched sensitivity and resolution. With its unique design, the Q800 easily outperforms competitive instruments, and is ideal for high-stiffness applications including composites.

## TECHNICAL SPECIFICATIONS

Maximum Force	18 N
Minimum Force	0.0001 N
Force Resolution	0.00001 N
Strain Resolution	1 nanometer
Modulus Range	$10^3$ to $3 \times 10^{12}$ Pa
Modulus Precision	$\pm 1\%$
Tan $\delta$ Sensitivity	0.0001
Tan $\delta$ Resolution	0.00001
Frequency Range	0.01 to 200 Hz
Dynamic Sample Deformation Range	$\pm 0.5$ to 10,000 $\mu\text{m}$
Temperature Range	-150 to 600 °C
Heating Rate	0.1 to 20 °C/min
Cooling Rate	0.1 to 10 °C/min
Isothermal Stability	$\pm 0.1$ °C
Time/Temperature Superposition	Yes

### OUTPUT VALUES

Storage Modulus	Complex/Dynamic Viscosity	Time
Loss Modulus	Creep Compliance	Stress/Strain
Storage/Loss Compliance	Relaxation Modulus	Frequency
Tan Delta ( $\delta$ )	Static/Dynamic Force	Sample Stiffness
Complex Modulus	Temperature	Displacement

# RSA III



The RSA III provides a powerful platform for high-performance DMA measurements. The RSA III uses an advanced direct-drive linear motor to apply the strain and the patented Force Rebalance Transducer™ to measure force. Low friction air bearings ensure optimal sensitivity. The RSA III is particularly well-suited for compression testing of soft materials, such as gels and elastomers, and for low stiffness, high frequency measurements on films and fibers.

## TECHNICAL SPECIFICATIONS

Maximum Force	35 N
Minimum Force	0.001 N
Force Resolution	0.0001 N
Strain Resolution	1 nanometer
Modulus Range	$10^3$ to $3 \times 10^{12}$ Pa
Modulus Precision	± 1%
Tan $\delta$ Sensitivity	0.0001
Tan $\delta$ Resolution	0.00001
Frequency Range	$2 \times 10^{-5}$ to 80 Hz
Dynamic Sample Deformation Range	± 0.5 to 1,500 $\mu$ m
Temperature Range	-150 to 600 °C
Heating Rate	0.1 to 60 °C/min
Cooling Rate	0.1 to 60 °C/min
Isothermal Stability	± 0.1 °C
Time/Temperature Superposition	Yes

### OUTPUT VALUES

Storage Modulus	Complex/Dynamic Viscosity	Time
Loss Modulus	Creep Compliance	Stress/Strain
Storage/Loss Compliance	Relaxation Modulus	Frequency
Tan Delta ( $\delta$ )	Static/Dynamic Force	Sample Stiffness
Complex Modulus	Temperature	Displacement

## DEFORMATION MODES & SAMPLE SIZE

INSTRUMENT/CLAMP	SAMPLE SIZE
<b>DUAL/SINGLE CANTILEVER</b>	
RSA III (Dual Only)	30 or 48* mm (L), Up to 12.5 mm (W) and 6 mm (T)
Q800	8/4** mm (L), Up to 15 mm (W) and 5 mm (T)
Q800	20/10** mm (L), Up to 15 mm (W) and 5 mm (T)
Q800	35/17.5** mm (L), Up to 15 mm (W) and 5 mm (T)
<b>3-POINT BEND</b>	
RSA III	30, 40 or 50 mm (L), Up to 12.5 mm (W) and 5 mm (T)
Q800	5, 10, or 15 mm (L), Up to 15 mm (W) and 7 mm (T)
Q800	20 mm (L), Up to 15 mm (W) and 7 mm (T)
Q800	50 mm (L), Up to 15 mm (W) and 7 mm (T)
<b>TENSION</b>	
RSA III (Film/Fiber)	Up to 35 mm (L), Up to 12.5 mm (W), and 1.5 mm (T)
Q800 (Film/Fiber)	5 to 30 mm (L), Up to 8 mm (W) and 2 mm (T)
Q800 (Fiber)	5 to 30 mm (L), 5 denier (0.57 tex) to 0.8 mm diameter
<b>SHEAR</b>	
RSA III	15 mm square, 0.5, 1.0 and 1.5 mm (T)
Q800	10 mm square, Up to 4 mm (T)
<b>COMPRESSION</b>	
RSA III	8, 15, and 25 mm diameter
Q800	15 and 40 mm diameter, Up to 10 mm (T)
<b>SUBMERSION</b>	
RSA III Tension	2 to 25 mm (L), up to 12 mm (W) and 2 mm (T)
RSA III Compression	15 mm diameter, up to 5 mm (T)
Q800 Tension	5 to 30 mm (L), Up to 8 mm (W) and 2 mm (T)
Q800 Compression	15 and 40 mm diameter, Up to 10 mm (T)

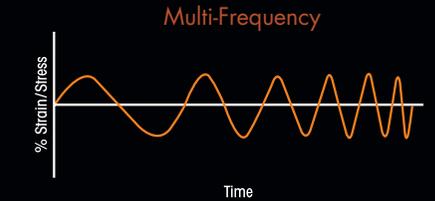
\*Dual cantilever only

\*\*Lengths are for dual/single cantilever

## MODES OF OPERATION

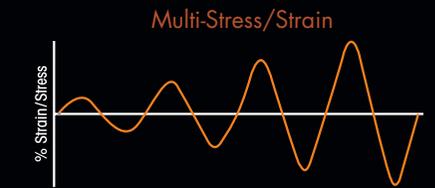
### MULTI-FREQUENCY

The multi-frequency mode can assess viscoelastic properties as a function of frequency, while oscillation amplitude is held constant. These tests can be run at single or multiple frequencies, in time sweep, temperature ramp, or temperature step/hold experiments. The RSA III has the additional capability of multiwave analysis during an isothermal or temperature ramp.



### MULTI-STRESS/STRAIN

In this mode, frequency and temperature are held constant, and the viscoelastic properties are monitored as strain or stress is varied. This mode is primarily used to identify the Linear Viscoelastic Range (LVR).



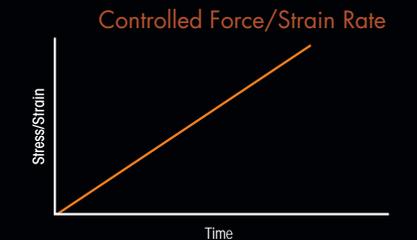
### CREEP/STRESS RELAXATION

With creep, the stress is held constant and deformation is monitored as a function of time. In stress relaxation, the strain is held constant and the stress is monitored vs. time.



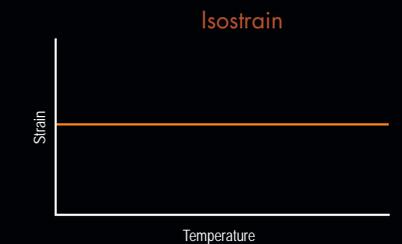
### CONTROLLED FORCE/STRAIN RATE

In this mode, the temperature is held constant while stress or strain is ramped at a constant rate. This mode is used to generate stress / strain plots to obtain Young's Modulus. Alternatively, stress can be held constant with a temperature ramp while strain is monitored.



### ISO STRAIN

In isostrain mode, available on the Q800, strain is held constant during a temperature ramp. Isostrain can be used to assess shrinkage force in films and fibers.



## Q800 TECHNOLOGY

### DRIVE MOTOR

The Q800 uses a non-contact, direct drive motor to provide the oscillatory or static force required. The motor is constructed of high performance composites that ensure low compliance and is thermostated to eliminate heat build-up even when using large oscillation amplitudes and high deformation forces. Sophisticated electronics enable the motor current to be rapidly adjusted in small increments. The motor can deliver reproducible forces over a wide range and the force can be changed rapidly, enabling a broad spectrum of material properties to be measured.

### AIR BEARINGS

The non-contact drive motor transmits force directly to a rectangular air bearing slide. The slide is guided by eight porous carbon air bearings grouped into two sets of four near the top and bottom of the slide. Pressurized air or nitrogen flows to the bearings forming a frictionless surface that permits the slide to "float". The slide, which connects to the drive shaft and sample clamp, can move vertically 25 mm and its rectangular shape eliminates any twisting of the sample. Very weak materials like films and fibers can be characterized with ease.

### OPTICAL ENCODER

A high-resolution linear optical encoder is used to measure displacement on the Q800 DMA. Based on diffraction patterns of light through gratings (one moveable and one stationary), optical encoders provide exceptional resolution compared to typical LVDT technology. Due to the excellent 1 nanometer resolution of the optical encoder, very small amplitudes can be measured precisely. This combined with the non-contact drive motor and air bearing technology provides excellent modulus precision and high  $\tan \delta$  sensitivity, allowing the Q800 DMA to characterize a broad range of materials.

### FURNACE

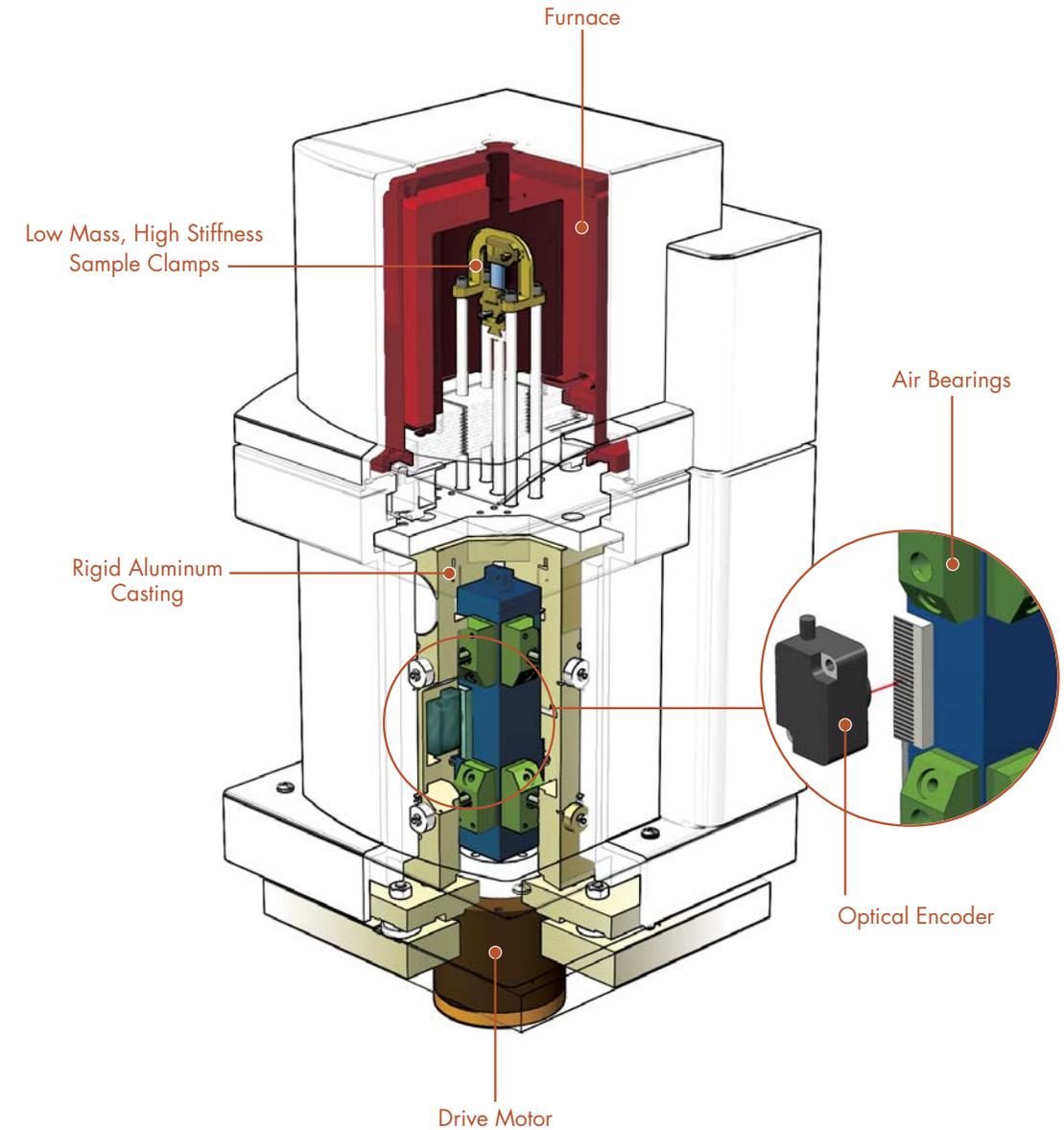
The Q800 features a bifilar wound furnace that automatically opens and closes. The furnace design combined with the Gas Cooling Accessory provides for efficient and precise temperature control over the entire temperature range, both in heating, cooling, and isothermal operation. The automatic furnace movement simplifies experimental setup.

### LOW MASS, HIGH STIFFNESS SAMPLE CLAMPS

The Q800 features a variety of sample clamps that provide for multiple modes of deformation. The clamps were designed using finite element analysis to provide high stiffness, with low mass, and attach to the drive shaft with a dovetail connection. The clamps are simple to use and adjust, and each is individually calibrated to insure data accuracy. A broad range of samples can be analyzed. The high stiffness minimizes clamp compliance, and the low mass ensures rapid temperature equilibration. The simple, yet elegant designs reduce the time necessary to change clamps and load samples.

### RIGID ALUMINUM CASTING

The Q800 drive motor, air bearing slide assembly with optical encoder and air bearings are all mounted within a rigid aluminum casting that is temperature controlled. The rigid aluminum housing minimizes system compliance and the temperature-controlled housing ensures precise data.



# RSA III TECHNOLOGY

## DRIVE MOTOR

Strain is applied to the sample via a high-performance linear motor. The motor is a direct-drive, DC servo actuator that controls strain, strain rate, and frequency. Temperature compensated, rare-earth magnets provide high force. Accurate measurements of viscoelastic properties are possible, and the fast response ensures exceptional performance during transient tests such as stress relaxation. High force expands the range of applications.

## FRAME ASSEMBLY AND LINEAR SLIDE

All the components of the RSA III DMA are housed in a rigid, cast steel frame with low compliance. The transducer (upper head) is attached to the frame via a linear slide. A motor drives the precision slide via a preloaded spindle to prevent backlash. Low compliance, stiff housing and linear slide ensures that all measurements made are accurate and precise.

## AIR BEARINGS

The high performance motor and the Force Rebalance Transducer™ incorporate air bearings that provide a very stiff, yet low friction means of supporting linear motion. Air bearings reduce friction and enhance the sensitivity of measurements, especially for weak samples like films and fibers.

## FORCE TRANSDUCER

The RSA III patented\* Force Rebalance Transducer™ (FRT) measures the force generated by the sample when the motor applies the deformation. In an FRT, a position sensor (3A) detects movement and a linear motor (3B) measures the reaction force required to drive the clamp back to the original position. The FRT provides a wide force range with high force sensitivity and negligible inertia. Since the motor inertia is decoupled from the force measurement, the FRT can make accurate and precise measurements over the complete frequency range, independent of sample stiffness.

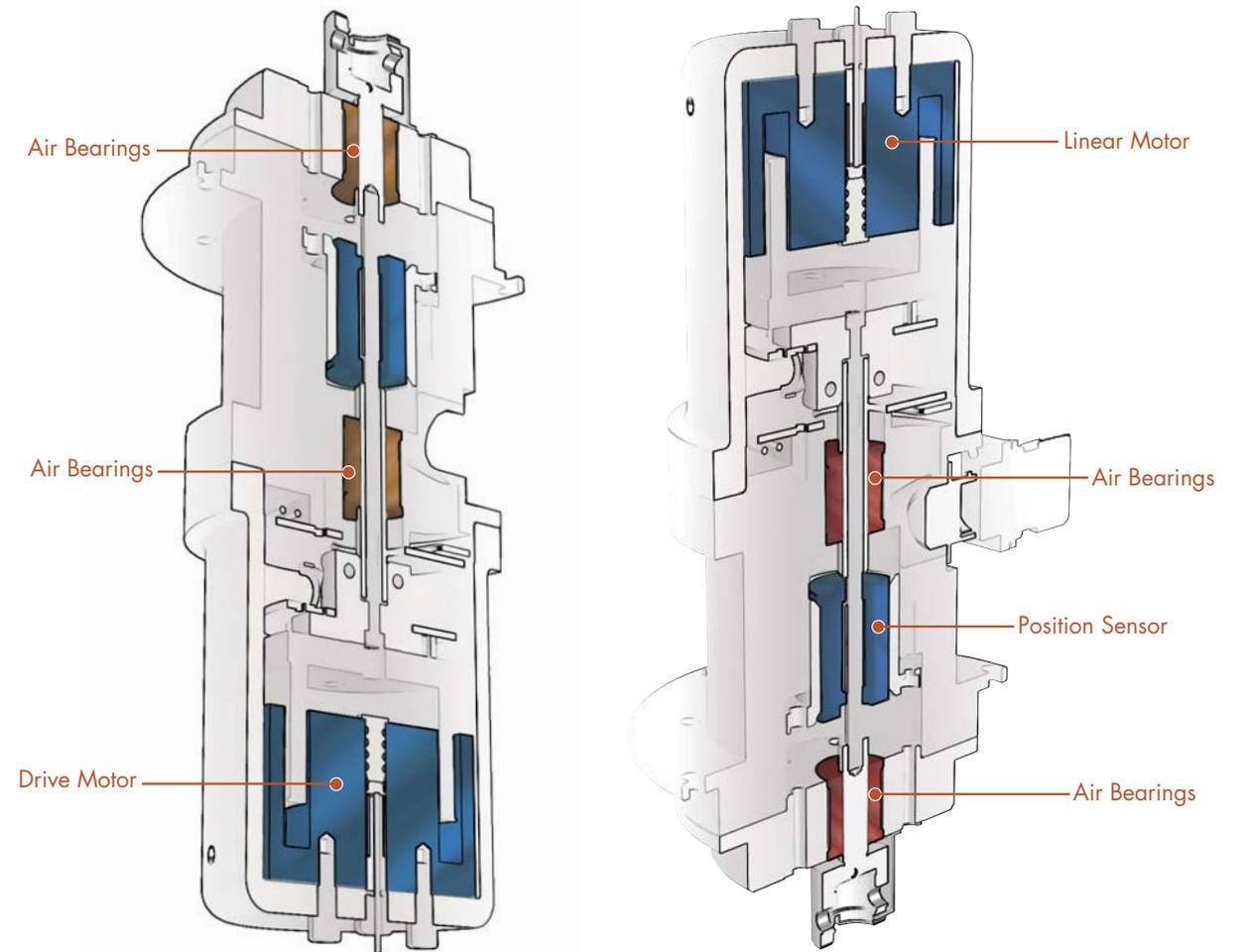
\*U.S. Patent No. 4601195

## FURNACE

The Forced Convection Oven (FCO) is an air convection oven with dual-element heaters and counter-rotating airflow for unmatched temperature stability. Optional cooling devices include a liquid nitrogen cooling device for subambient operation to -150 °C. The FCO includes a sample sight glass. Combined with either of the two cooling devices, the FCO oven provides accurate and precise temperature control, and fast heating rates, for a broad range of applications. The sight glass allows samples to be viewed during the experiment.

## SIMULTANEOUS MEASUREMENTS

The RSA III is available with optional simultaneous measuring techniques. These include dielectric measurements and exposing a sample to a UV light source. Simultaneous measurements extend the range of applications.



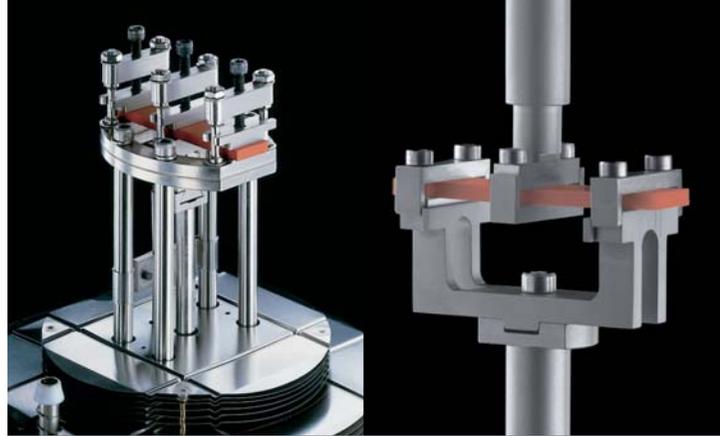
LOWER ASSEMBLY - MOTOR

UPPER ASSEMBLY - TRANSDUCER

## MODES OF DEFORMATION

### DUAL/SINGLE CANTILEVER

In this mode, the sample is clamped at both ends and either flexed in the middle (dual cantilever) or at one end (single cantilever). Cantilever bending is a good general-purpose mode for evaluating thermoplastics and highly damped materials (e.g., elastomers). Dual cantilever mode is ideal for studying the cure of supported thermosets.



Q800

RSA III

### 3-POINT BEND

In this mode, the sample is supported at both ends and force is applied in the middle. 3-point bend is considered a "pure" mode of deformation since clamping effects are eliminated. The 50 and 20 mm clamps on the Q800 utilize unique low-friction, roller bearing supports that improve accuracy.



Q800

RSA III

### SHEAR SANDWICH

In this mode, two equal-size pieces of the same material are sheared between a fixed and moveable plate. This mode is ideal for gels, adhesives, high viscosity resins, and other highly damped materials. The RSA III also offers a quartz shear clamp for exposing a sample to a UV light source.



Q800

RSA III

### COMPRESSION

In this mode, the sample is placed on a fixed flat surface and an oscillating plate applies force. Compression is suitable for low to moderate modulus materials (e.g., foams and elastomers). This mode can also be used to make measurements of expansion or contraction, and tack testing for adhesives.



Q800

RSA III

### TENSION

In this mode, the sample is placed in tension between a fixed and moveable clamp. In oscillation experiments, the instruments use a variety of methods for applying a static load to prevent buckling and unnecessary creep. The clamps are suitable for both films and fibers.

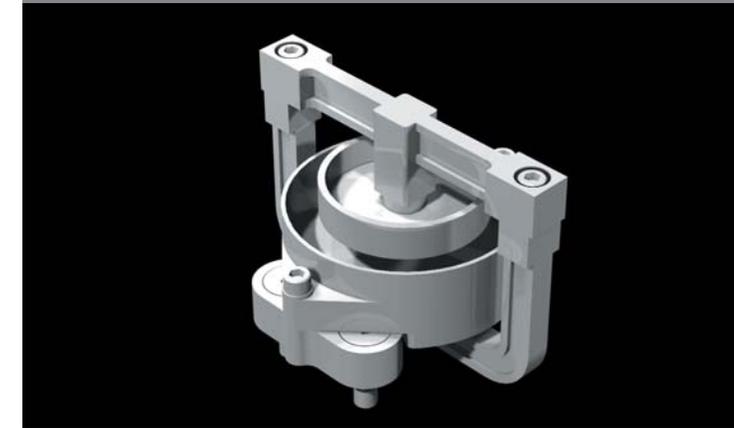


Q800

Q800

### SUBMERSIBLE CLAMPS

Both film tension and compression clamps are available in submersible configurations for the Q800. A tension submersion clamp is available for the RSAIII. These clamps allow samples to be analyzed in a fluid environment up to 80 °C.



Q800

# ACCESSORIES

## Q800 SUBAMBIENT OPERATION

The Gas Cooling Accessory (GCA) extends the operating range of the Q800 to -150 °C. The GCA uses cold nitrogen gas generated from controlled heating of liquid nitrogen. Automated filling of the GCA tank can be programmed to occur either after the scan is complete or during a run. The ability to automatically refill during the middle of a run is particularly useful during long DMA experiments typically encountered when generating data for Time/Temperature Superposition (TTS).

## RSA III SUBAMBIENT OPTIONS

The RSA III is available with a liquid nitrogen cooling system for operation from -150 to 600 °C. This accessory connects directly to a bulk liquid nitrogen dewar and provides rapid cooling capability.



# DMA THEORY



Dynamic Mechanical Analysis (DMA) is a technique used to measure the mechanical properties of a wide range of materials. Many materials, including polymers, behave both like an elastic solid and a viscous fluid, thus the term viscoelastic. DMA differs from other mechanical testing devices in two important ways. First, typical tensile test devices focus only on the elastic component. In many applications, the inelastic, or viscous component, is critical. It is the viscous component that determines properties such as impact resistance. Second, tensile test devices work primarily outside the linear viscoelastic range. DMA works primarily in the linear viscoelastic range and is therefore more sensitive to structure.

DMA measures the viscoelastic properties using either transient or dynamic oscillatory tests. Transient tests include creep and stress relaxation. In creep, a stress is applied to the sample and held constant while deformation is measured vs. time. After some time, the stress is removed and the recovery is measured. In stress relaxation, a deformation is applied to the sample and held constant, and the degradation of the stress required to maintain the deformation is measured versus time.

The most common test is the dynamic oscillatory test, where a sinusoidal stress (or strain) is applied to the material and a resultant sinusoidal strain (or stress) is measured (Figure 1). Also measured is the phase difference,  $\delta$ , between the two sine waves. The phase lag will be zero degrees for purely elastic materials and 90 degrees for purely viscous materials. Viscoelastic materials (eg. polymers) will exhibit an intermediate phase difference.

Since modulus is stress/strain, the complex modulus,  $E^*$ , can be calculated. From  $E^*$  and the measurement of  $\delta$ , the storage modulus,  $E'$ , and loss modulus,  $E''$ , can be calculated as illustrated in Figure 2.  $E'$ , the storage modulus is the elastic component and related to the sample's stiffness.  $E''$ , the loss modulus, is the viscous component and is related to the sample's ability to dissipate mechanical energy through molecular motion. The tangent of phase difference, or  $\tan \delta$ , is another common parameter that provides information on the relationship between the elastic and inelastic components. All of these parameters can be calculated as a function of time, temperature, frequency, or amplitude (stress or strain) depending on the application.

Figure 1

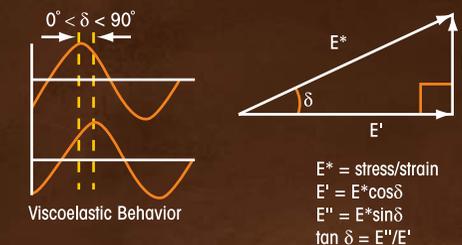
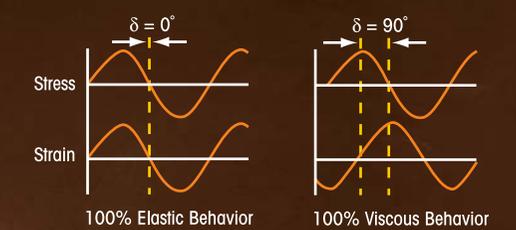


Figure 2



# APPLICATIONS

## MEASUREMENT OF T<sub>g</sub> OF POLYMERIC MATERIALS

A common measurement on polymers is the glass transition temperature, T<sub>g</sub>. It can be measured with various techniques, but DMA is by far the most sensitive. The figure below shows a scan of a pressure sensitive adhesive run in the tension clamps at a frequency of 1 Hz. T<sub>g</sub> can be measured by the E' onset point, by the E'' peak, or the peak of Tan δ. In addition to the T<sub>g</sub>, the absolute value of the various viscoelastic parameters is also useful.

## FREQUENCY EFFECT ON MODULUS AND GLASS TRANSITION OF POLYETHYLENE TEREPHTHALATE (PET)

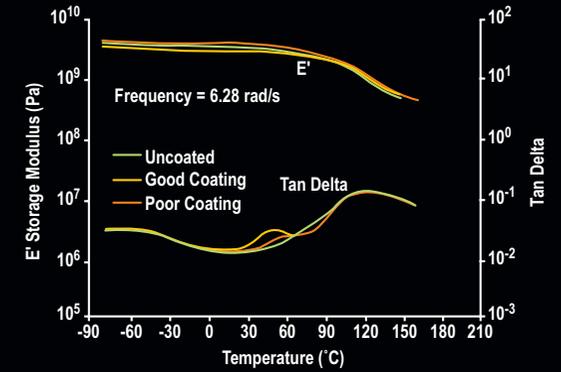
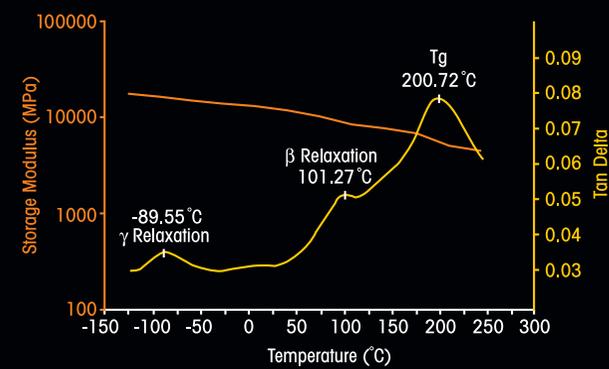
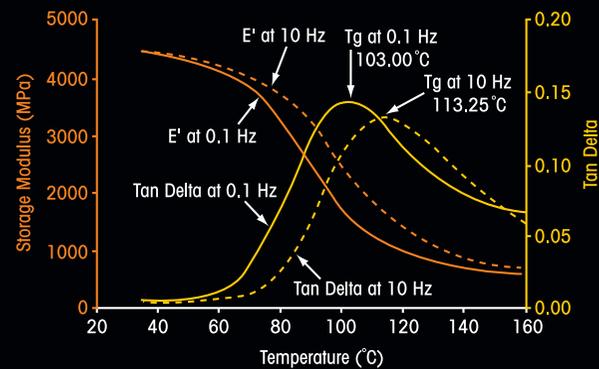
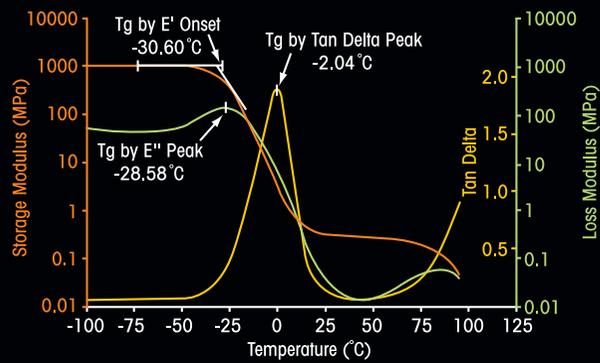
Because the T<sub>g</sub> has a kinetic component, it is strongly influenced by the frequency (rate) of deformation. As the frequency of the test increases, the molecular relaxations can only occur at higher temperatures and, as a consequence, the T<sub>g</sub> will increase with increasing frequency as illustrated below. In addition, the shape and intensity of the Tan δ peak as well as the slope of the storage modulus in the transition region will be affected. Based on end-use conditions, it is important to understand the temperature and frequency dependence of transitions.

## THE MEASUREMENT OF SECONDARY TRANSITIONS IN VINYL ESTER

DMA is one of the few techniques that can measure β and γ secondary transitions. Secondary transitions arise from side group motion with some cooperative vibrations from the main chain as well as internal rotation within a side group. The transitions are below the T<sub>g</sub> and typically subambient. They are very important as they affect impact resistance and other end-use properties. This data was generated using 3-point bending and also illustrates the ability to run stiff composites.

## MEASURING EFFECT OF ADHESIVE COATINGS ON FILMS

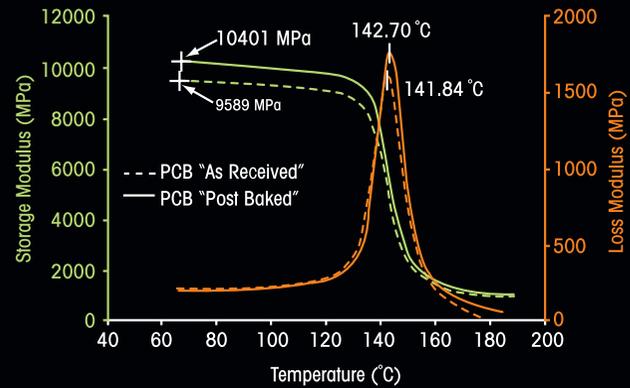
Below shows a comparison among three PET samples in tension on the RSA III: one with a uniform adhesive layer that performs well; one with a non-uniform layer that performs poorly; and one that is uncoated. A transition peak due to the adhesive is seen in Tan δ around 40 °C in the "good" sample, whereas the "poor" sample shows a much smaller peak. Knowing the characteristics of good and poor samples enables quality control of the coating process and the finished product.



# APPLICATIONS

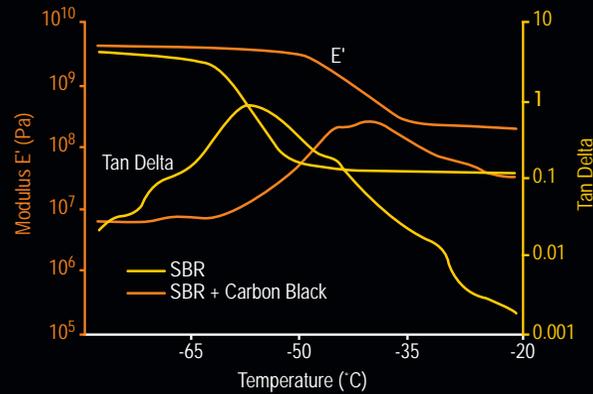
## CHARACTERIZING PRINTED CIRCUIT BOARDS

Printed Circuit Boards (PCB) are typically comprised of fiberglass braid impregnated with a thermosetting resin. Characterizing the T<sub>g</sub> of PCB's is often difficult due to the very low amount of resin used. The figure below shows a typical PCB run in single cantilever bending. The T<sub>g</sub> is clearly discernible and the difference between the sample "as received" and "post baked" clearly shows the effect that further crosslinking has on both the T<sub>g</sub> and the absolute value of modulus.



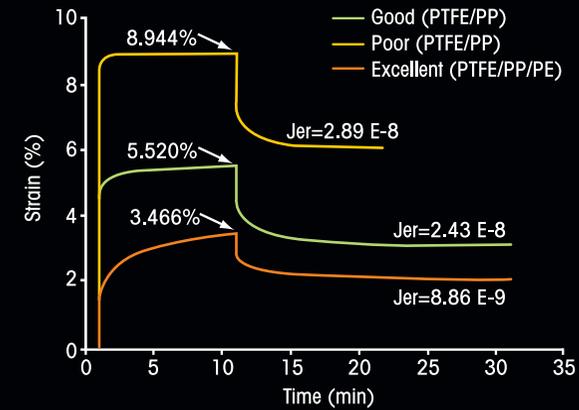
## EFFECT OF CARBON BLACK IN ELASTOMERS

Another very common application is the effect of fillers and additives on viscoelastic properties. Below illustrates the effect on Storage Modulus (E') and Tan δ when adding carbon black to an SBR rubber. This test, performed in dual cantilever on the RSA III, shows that adding carbon black increases the absolute value of the Storage Modulus and significantly increases the T<sub>g</sub> temperature. Understanding how fillers and additives affect material properties is crucial in many industrial applications.



## CHARACTERIZING PACKAGING FILMS USING CREEP

In a thermoforming process, a film is pulled down into a heated mold to form a desired shape. The ability to produce a stable product can be predicted by using a creep-recovery experiment. Below illustrates data on a packaging film using the tension mode. In the recovery phase, the equilibrium recoverable compliance, (J<sub>er</sub>) can be calculated. If the sample compliance is too high, as observed by a high J<sub>er</sub>, then the elasticity may be too low at the forming temperature to maintain the desired shape.



## PREDICTING MATERIAL PERFORMANCE USING TIME / TEMPERATURE SUPERPOSITIONING (TTS)

The TTS technique, well grounded in theory, is used to predict material performance at frequencies or time scales outside the range of the instrument. Data is usually generated by scanning multiple frequencies during a series of isothermal step-hold experiments over a temperature range. A reference temperature is selected and the data shifted. A shift factor plot is generated and fit to either a William-Landel-Ferry (WLF) or Arrhenius model. Finally, a master curve at a specific temperature is generated as illustrated below for a PET film sample. Using this technique, properties at very high frequencies (short time scales) or very low frequencies (long time scales) can be assessed.

