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TA Instruments

THERMAL ANALYSIS

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DYNAMIC MECHANICAL **ANALYSIS**

Accurate, Precise, Versatile DMA Measurements

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DMA Q800
SPECIFICATIONS



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The Q800 is the world's best-selling DMA, for very good reason. 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.

Maximum Force	18 N
Minimum Force	0.0001 N
Force Resolution	0.00001 N
Strain Resolution	1 nanometer
Modulus Range	10^3 to $3x10^{12}$ Pa
Modulus Precision	± 1%
Tan δ Sensitivity	0.0001
Tan δ Resolution	0.00001
Frequency Range	0.01 to 200 Hz
Dynamic Sample Deformation Range	± 0.5 to 10 000 µm
Temperature Range	-150 to 600°C
Heating Rate	0.1 to 20°C/min
Cooling Rate	0.1 to 10°C/min
Isothermal Stability	± 0.1°C
Time/Temperature Superposition	Yes
RH Control	Optional

Output Values

Storage Modulus	Complex/Dynamic Viscosity	Time
Loss Modulus	Creep Compliance	Stress/Strain
Storage/Loss Compliance	Relaxation Modulus	Frequency
Tan Delta (δ)	Static/Dynamic Force	Sample Stiffness
Complex Modulus	Temperature	Displacement
Relative Humidity (Optional)		

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DMA **DEFORMATION MODES & SAMPLE SIZE**

Dual/Single Cantilever	
	8/4* mm (L), Up to 15 mm (W) and 5 mm (T)
	20/10* mm (L), Up to 15 mm (W) and 5 mm (T)
	35/17.5* mm (L), Up to 15 mm (W) and 5 mm (T)
3-Point Bend	
	5, 10, or 15 mm (L), Up to 15 mm (W) and 7 mm (T)
	20 mm (L), Up to 15 mm (W) and 7 mm (T)
	50 mm (L), Up to 15 mm (W) and 7 mm (T)
Tension	
Film/Fiber	5 to 30 mm (L), Up to 8 mm (W) and 2 mm (T)
Fiber	5 to 30 mm (L), 5 denier (0.57 tex) to 0.8 mm diameter
Shear	10 mm square, Up to 4 mm (T)
Compression	15 and 40 mm diameter, Up to 10 mm (T)
Submersion	
Tension	Fixed at 15 mm (L), Up to 8 mm (W) and 2 mm (T)
Compression	25 mm diameter, Up to 10 mm (T)
3-Point Bend	5, 10, or 15 mm (L), Up to 15 mm (W) and 7 mm (T)

*Lengths are for dual/single cantilever



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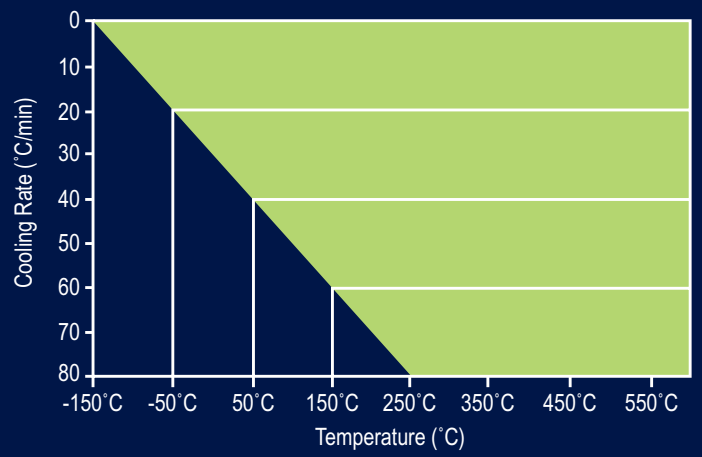


SUBAMBIENT **OPERATION**

Gas Cooling Accessory

The Gas Cooling Accessory (GCA) extends the operating range of the Q800 to -150°C. The GCA uses cold nitrogen gas generated from controlled evaporation of liquid nitrogen. Automated filling of the GCA tank can be programmed to occur after the scan is complete.

The GCA will provide ballistic or controlled cooling rates over the entire operating range of the Q800 DMA (-150 to 600°C). In general, the maximum cooling rate is a function of the installed clamp and the thermal characteristics of the sample. The figure below shows the typical range* of controlled cooling rates available as a function of temperature.



*Actual performance may vary slightly depending on laboratory conditions and the clamping system installed.

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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.

Furnace

The Q800 features a bifilar wound furnace with automated movement. 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.

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 δ sensitivity allowing the Q800 DMA to characterize a broad range of materials.

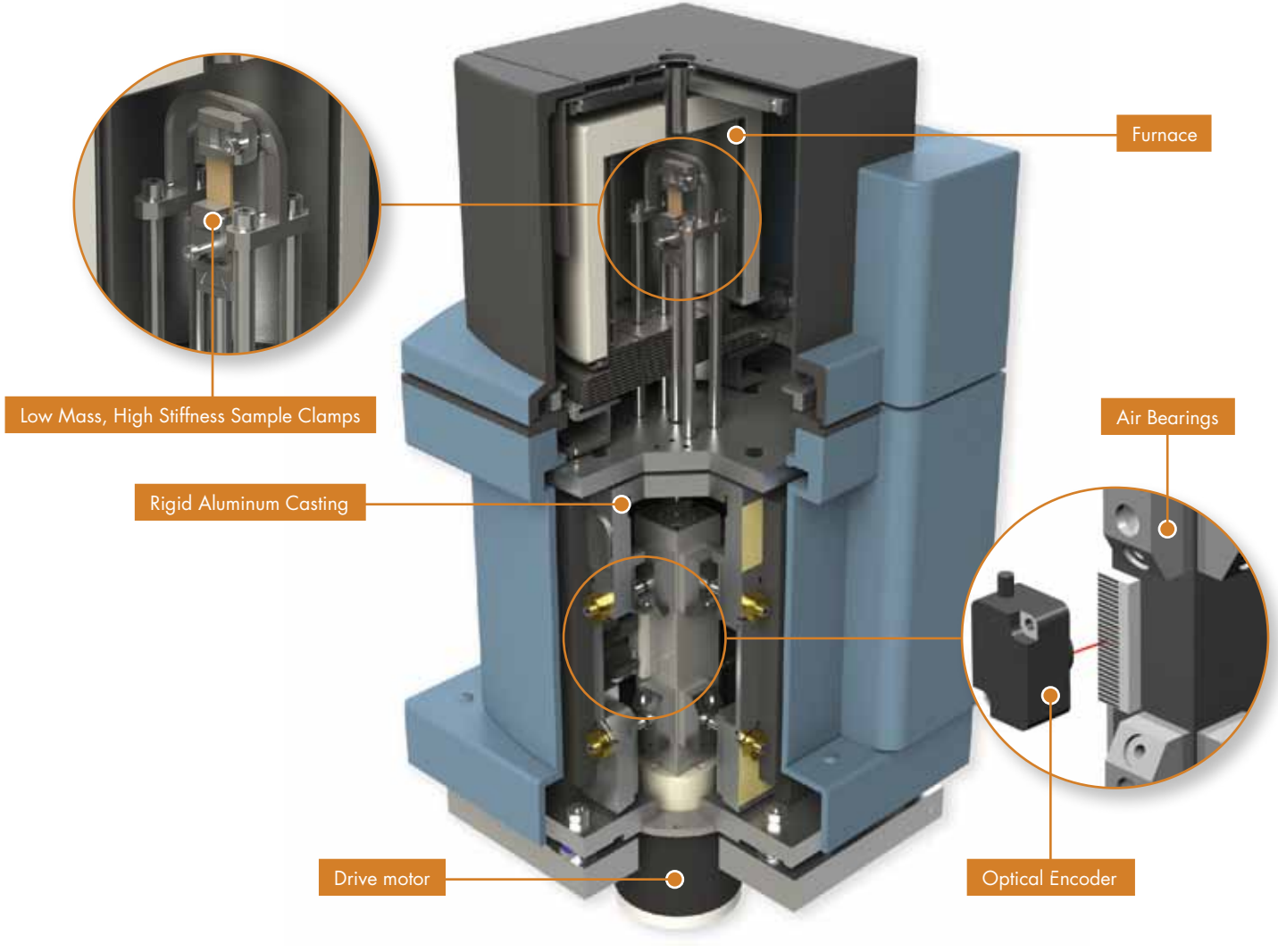
Low Mass, High Stiffness Sample Clamps

The Q800 features a variety of sample clamps that provide for multiple modes of deformation. The clamps are optimized using finite element analysis to provide high stiffness, with low mass, and attach to the drive shaft with a simple dovetail connection. The clamps are easy to use and adjust, and each is individually calibrated to ensure data accuracy. A broad range of samples can be analyzed. The high stiffness minimizes clamp compliance, and the low mass ensures rapid temperature equilibration. These 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.

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Furnace
Low Mass, High Stiffness Sample Clamps
Air Bearings
Rigid Aluminum Casting
Drive motor
Optical Encoder

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. A powder clamp is also available for characterizing transitions in powder materials.

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.

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.



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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.

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.

Submersible Clamps

Film tension, compression, and 3-point bend clamps are available in submersible configurations for the Q800. These clamps allow samples to be analyzed in a fluid environment up to 80°C.



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DMA ACCESSORIES

DMA-RH

The new DMA-RH Accessory allows mechanical properties of a sample to be analyzed under controlled and/or varying conditions of both relative humidity and temperature. It is designed for use with the Q800 Dynamic Mechanical Analyzer.

The DMA-RH Accessory is a fully integrated unit and includes the following hardware components:

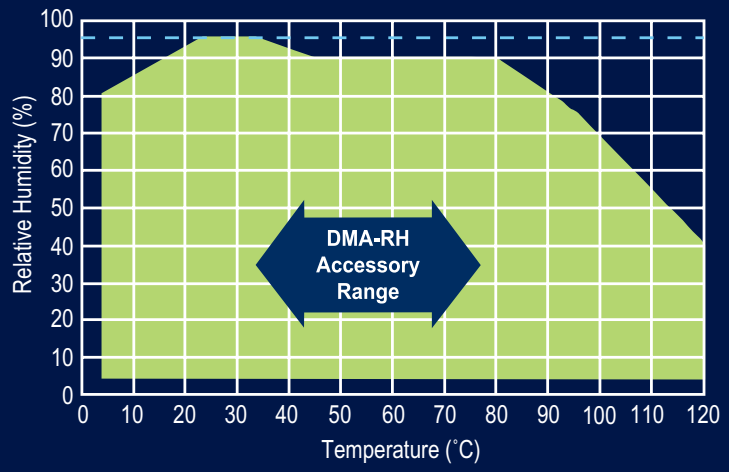
1. The sample chamber mounts to the DMA in place of the standard furnace. Peltier elements in the chamber precisely control the temperature to +/-0.1°C. The sample chamber accommodates standard DMA clamps (tension, cantilever, and 3-point bending). It is quickly removed for conversion back to the standard DMA furnace.

2. A heated vapor-transfer line is maintained at a temperature above the dew point temperature of the humidified gas in order to avoid condensation and provide accurate results.

3. The DMA-RH Accessory contains the humidifier and electronics which continuously monitor and control temperature and humidity of the sample chamber.

The DMA-RH accessory offers the widest range of temperature and relative humidity.

Temperature Range	5 to 120°C
Temperature Accuracy	±0.5°C
Heating/Cooling Rate	Maximum ±1°C/min
Humidity Range	See humidity range chart.
Humidity Accuracy	5-90% RH: ±3% RH >90% RH: ±5% RH
Humidity Ramp Rate (both increasing and decreasing)	2% RH/min (fixed)



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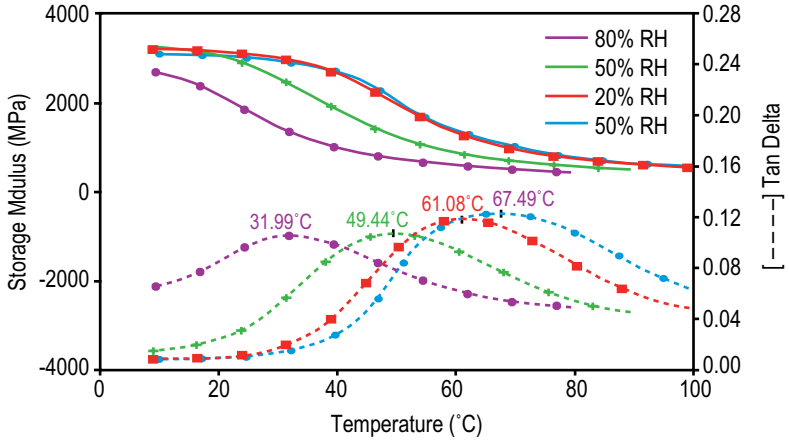


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DMA-RH **APPLICATIONS**

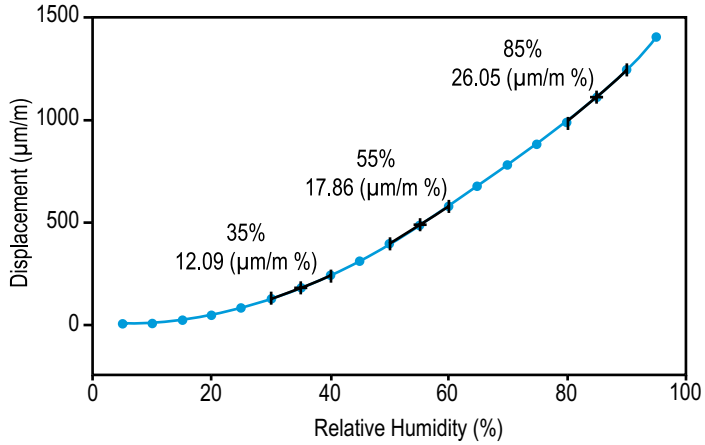
Effect of Relative Humidity on the Glass Transition of Nylon 6

Nylon 6 is strongly plasticized by water; as such the mechanical properties will be dependent on the surrounding relative humidity. The data in this figure demonstrate the effect of relative humidity on the glass transition of Nylon 6 as measured on the Q800 DMA equipped with the DMA-RH accessory. The sample was analyzed in single cantilever mode at a frequency of 1 Hz at a variety of constant RH conditions. Note how the mechanical properties and glass transition are significantly influenced by the imposed relative humidity.



Measurement of the Coefficient of Hygroscopic Expansion (CHE)

Hygroscopy is defined as the ability of a substance to attract water molecules from the surrounding environment through either absorption or adsorption. The effect of moisture sorption on the mechanical characteristics of a material can be quantified by the Coefficient of Hygroscopic Expansion (CHE), the constant which relates the dimensional change of a material to a change in the surrounding relative humidity. The data in this figure show the effect of imposed relative humidity on the Nylon 6 sample as measured by the Q800 DMA with the DMA-RH Accessory. As the relative humidity is increased the sample expands. The resulting slope of the line is equivalent to the CHE for the material.

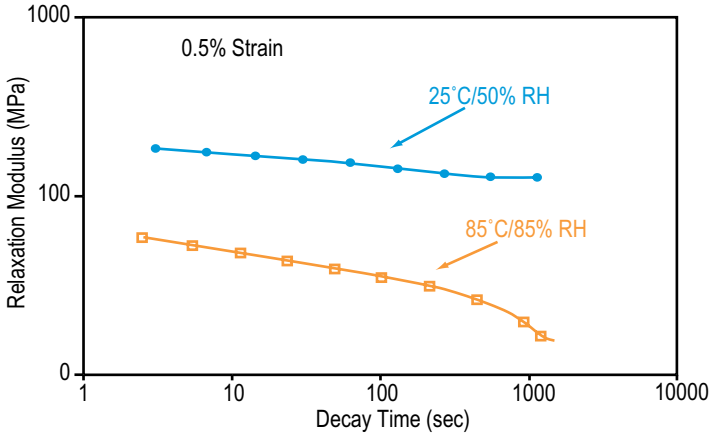


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Stress Relaxation of Nafion® 112 Under Varying Temperature/RH Conditions

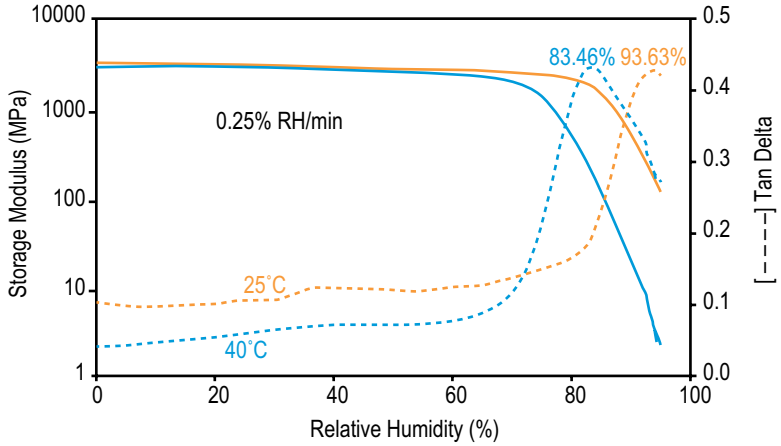
Recent research has focused on alternative fuel technologies including Proton Exchange Membrane Fuel Cells (PEMFC) which contain polymeric membranes such as Nafion* 112. PEM properties can significantly change as functions of time and exposure to elevated temperatures and humidity, as water is the primary by-product of the electrochemical reaction of the fuel cell. The Q800 DMA equipped with the DMA-RH accessory is the ideal platform to study the effect of temperature and humidity on the time-dependent processes of the PEM. In this example, the stress relaxation behavior of a Nafion 112 membrane is analyzed in tension mode under two discrete conditions; 25°C/50% RH (controlled ambient) and under elevated temperature and RH conditions of 85°C/85% RH.

*Nafion is a registered trademark of DuPont Co.



Analysis of a Pharmaceutical Gelatin Capsule

Gelatin capsules are widely used in the pharmaceutical and dietary supplement market. When stored in an ambient, low-humidity environment gelatin is remarkably stable. However, when combined with water, gelatin forms a semi-solid colloid gel which can profoundly affect its mechanical properties. The data in this example illustrate the effect of increasing relative humidity on a gelatin sample cut from the side wall of a two-piece capsule at 25°C and 40°C. As the relative humidity is increased, the material undergoes a multi-step transition resulting in a significant decrease in modulus near 80% RH. The transition is resolved in both the storage modulus and tan δ signals.



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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. 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. Also measured is the phase difference, δ, between the two sine waves. The phase lag will be 0° for purely elastic materials and 90° for purely viscous materials (Figure 1). However, viscoelastic materials (e.g. polymers) will exhibit an intermediate phase difference (Figure 2a).

Since modulus equals stress/strain, the complex modulus, E*, can be calculated. From E* and the measurement of δ, the storage modulus, E′, and loss modulus, E′′, can be calculated as illustrated in Figure 2b. The storage modulus (E′) is the elastic component and related to the sample's stiffness. The loss modulus (E′′) 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 δ, is another common parameter that provides information on the relationship between the elastic and inelastic components.

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.

Range of Material Behavior

Solid Like ⟷ Liquid Like

Ideal Solid ⟷ Most Materials ⟷ Ideal Fluid

Purely Elastic ⟷ Viscoelastic ⟷ Purely Viscous

Viscoelasticity: Having both viscous and elastic properties

Figure 1

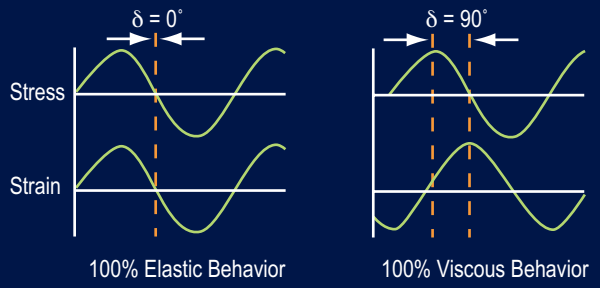


Figure 2a

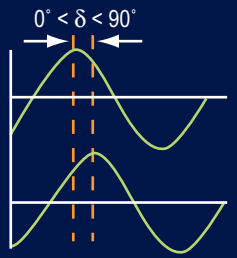
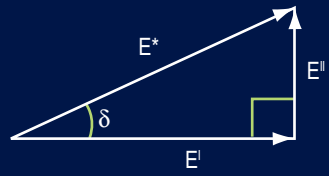


Figure 2b



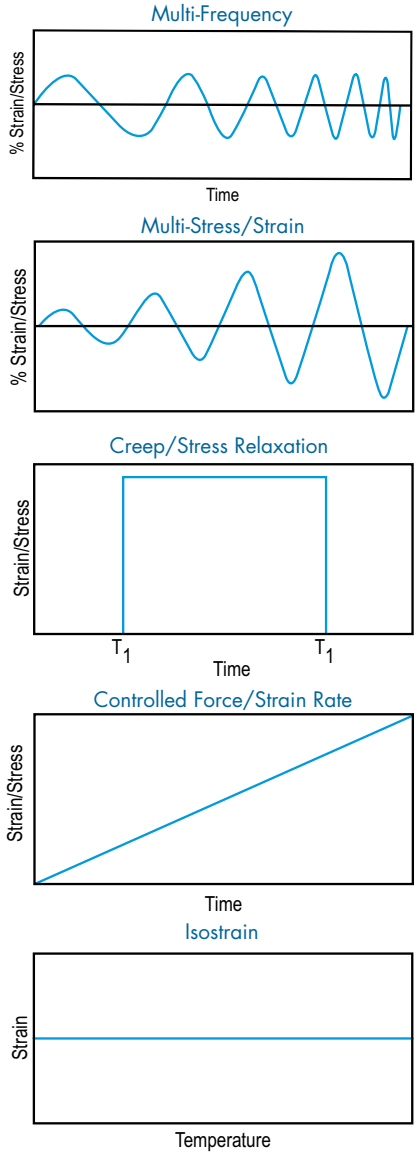
E* = stress/strain

$E' = E^*\cos\delta$

$E'' = E^*\sin\delta$

$\tan\delta = E''/E'$

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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.

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.

Isostrain

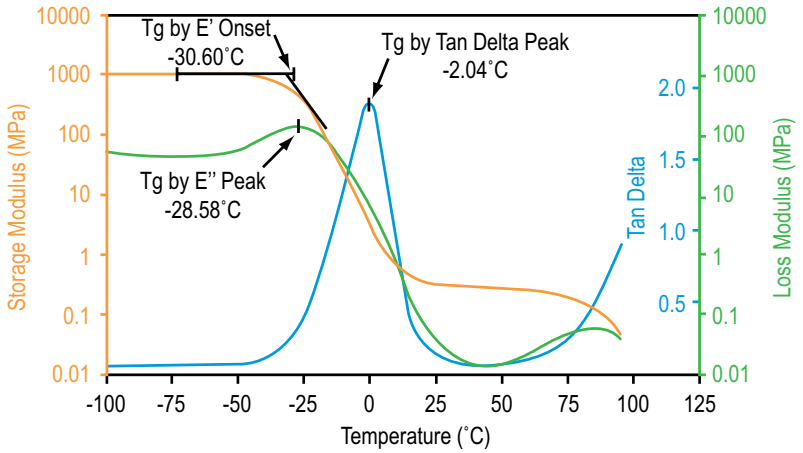
In isostrain mode, available on the Q800, strain is held constant during a temperature ramp. Isostrain can be used to assess shrinkage force in films and fibers.

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APPLICATIONS

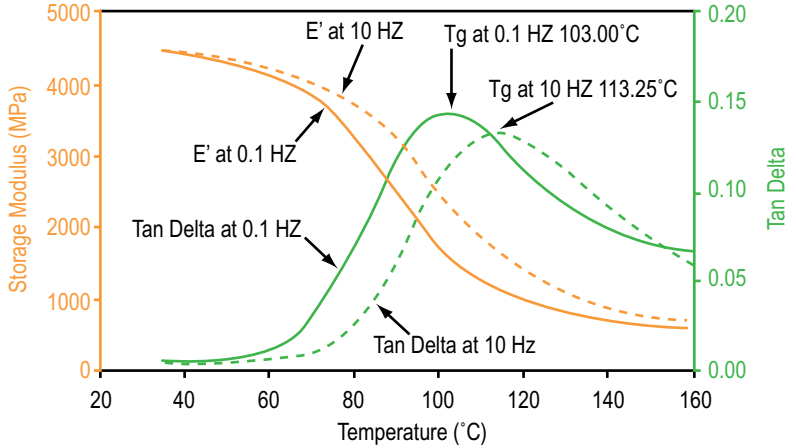
Measurement of Tg of Polymeric Materials

A common measurement on polymers is the glass transition temperature, Tg. It can be measured with various techniques, but DMA is by far the most sensitive. The figure to the right shows a scan of a pressure sensitive adhesive run in the tension clamps at a frequency of 1 Hz. Tg can be measured by the E′ onset point, by the E′′ peak, or the peak of Tan δ. In addition to the Tg, the absolute value of the various viscoelastic parameters is also useful.



Frequency Effect on Modulus and Glass Transition of Polyethylene Terephthalate (PET)

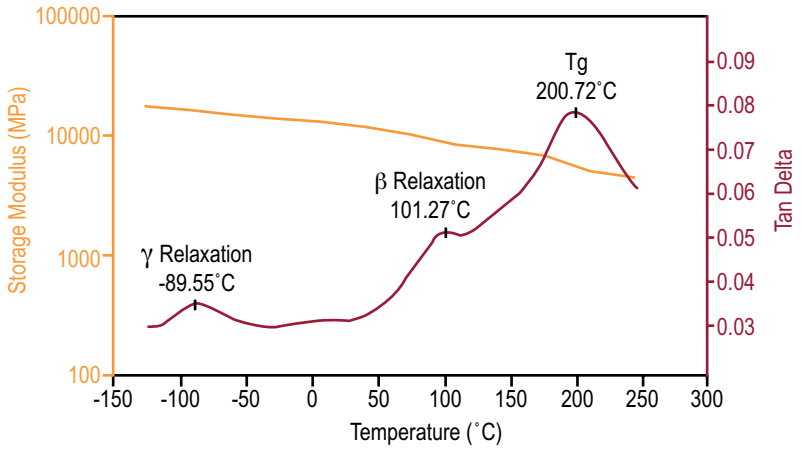
Because the Tg has a kinetic component, it is strongly influenced by the frequency (rate) of deformation. As the frequency of the test increases, the molecular relaxations can only occur at higher temperatures and, as a consequence, the Tg will increase with increasing frequency as illustrated to the right. 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.



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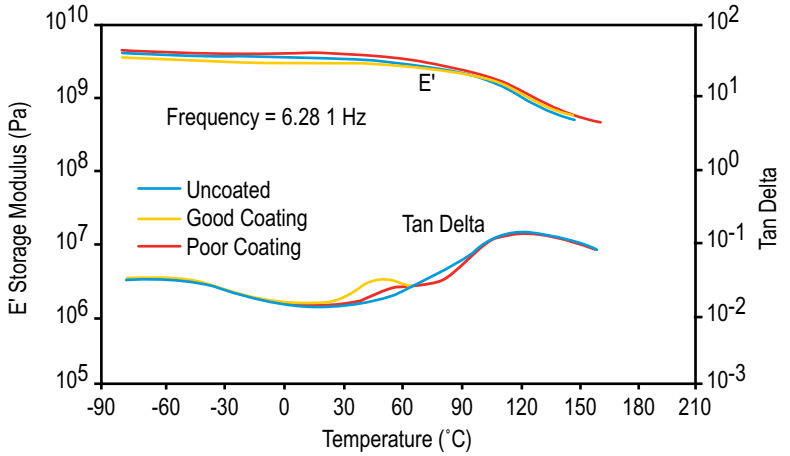
The Measurement of Secondary Transitions in Vinyl Ester

DMA is one of the few techniques sensitive to 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 Tg 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

This figure shows a comparison among three PET samples in tension on the DMA; 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.

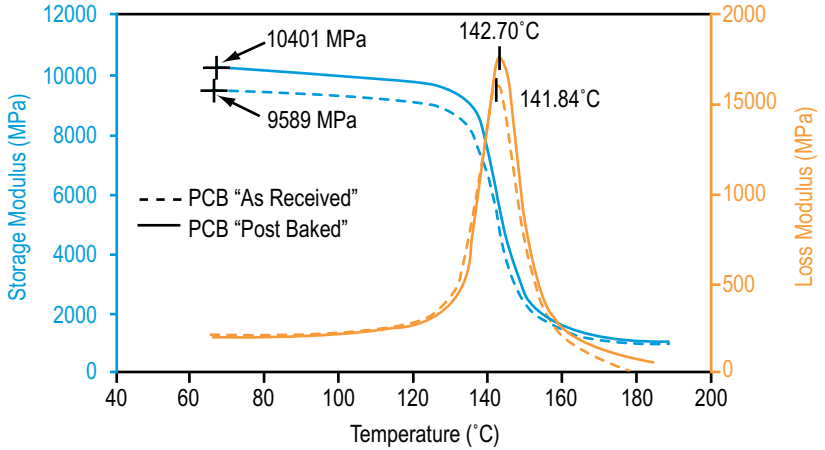


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APPLICATIONS

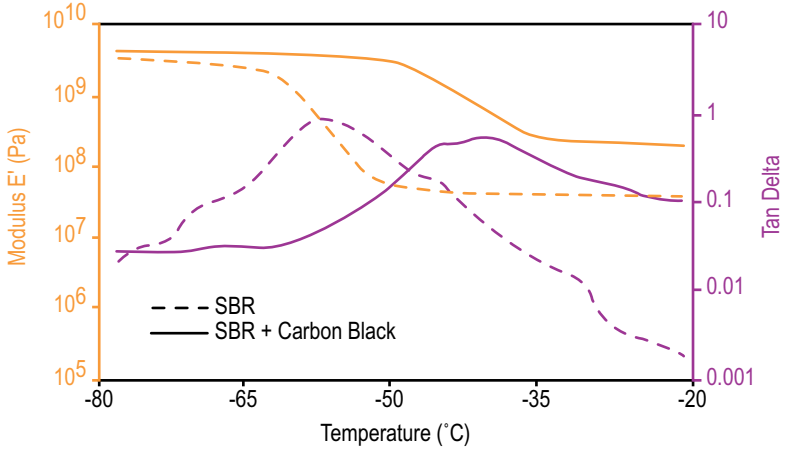
Characterizing Printed Circuit Boards

Printed Circuit Boards (PCB) are typically comprised of fiberglass braid impregnated with a thermosetting resin. Characterizing the Tg of PCB's is often difficult due to the very low amount of resin used. This figure shows a typical PCB run in single cantilever bending. The Tg 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 Tg and the absolute value of modulus.



Effect of Carbon Black in Elastomers

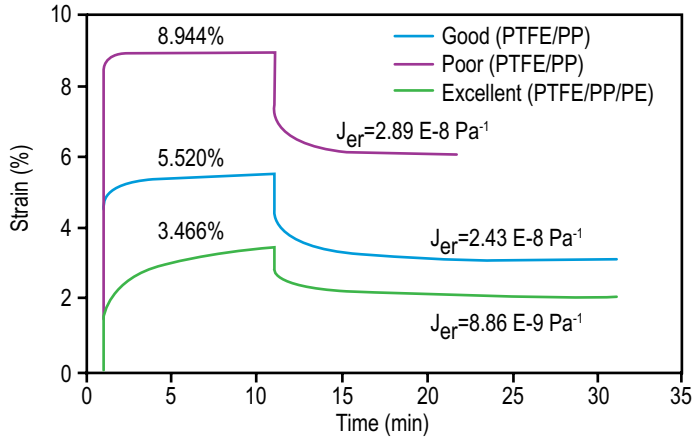
Another very common application is the effect of fillers and additives on viscoelastic properties. The figure to the right illustrates the effect on storage modulus (E') and Tan δ when adding carbon black to an SBR rubber. This test, performed in dual cantilever on the DMA, shows that adding carbon black increases the absolute value of the storage modulus and significantly increases the Tg temperature. Understanding how fillers and additives affect material properties is crucial in many industrial applications.



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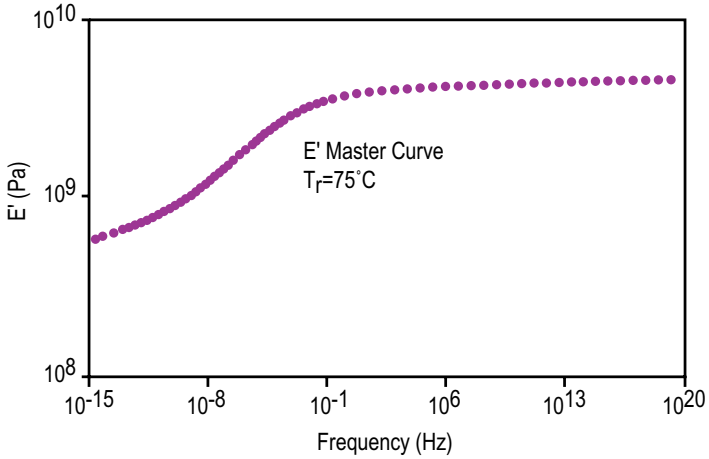
Characterizing Packaging Films Using Creep

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. This figure illustrates data on a packaging film using the tension mode. In the recovery phase, the equilibrium recoverable compliance, (J_{er}) can be calculated. If the sample compliance is too high, as observed by a high J_{er}, 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 Williams-Landel-Ferry (WLF) or Arrhenius model. Finally, a master curve at a specific temperature is generated as illustrated to the right 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.



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