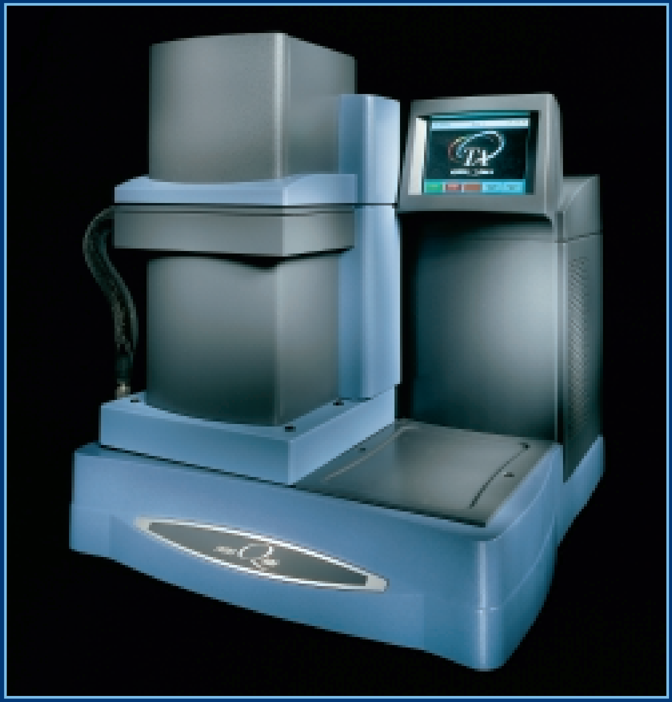
TA Instruments Dynamic Mechanical Analyzer

The TA Instruments Dynamic Mechanical Analyzer

The TA Instruments Q800 is the culmination of years of engineering expertise in Dynamic Mechanical Analyzers. TA Instruments pioneered DMA instrumentation with the 980 DMA introduced in 1976 and continues the tradition today. The Q800 is the sixth generation DMA and incorporates unique technology. Backed by the best customer support available, the TA Instruments Q800 DMA sets the standard for Dynamic Mechanical Analyzers.

DMA Q800



The most important features of any DMA are how the stress is applied to the sample, and how the resultant strain is measured. The ***Q800*** *DMA utilizes state-of-the-art non-contact, linear drive motor technology to provide precise control of the stress. Strain is measured using optical encoder technology that provides unmatched sensitivity and resolution. Along with low friction air bearing technology, the Q800 provides superior performance compared to traditional designs that employ LVDT technology, stepper motors, and mechanical springs.*

Technical Specifications

Maximum Force	18 N
Minimum Force	0.001 N
Force Resolution	0.0001 N
Strain Resolution	1 nanometer
Modulus Range	$10E^{3}$ to $3x10E^{12}$ Pa
Modulus Precision	+/- 1%
Tan δ (delta) Range	0.0001 to 10
Tan δ (delta) Sensitivity	0.0001
Tan δ (delta) 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
Automated Furnace Movement	Yes
Time/Temperature Superposition Software	Yes

Output Values

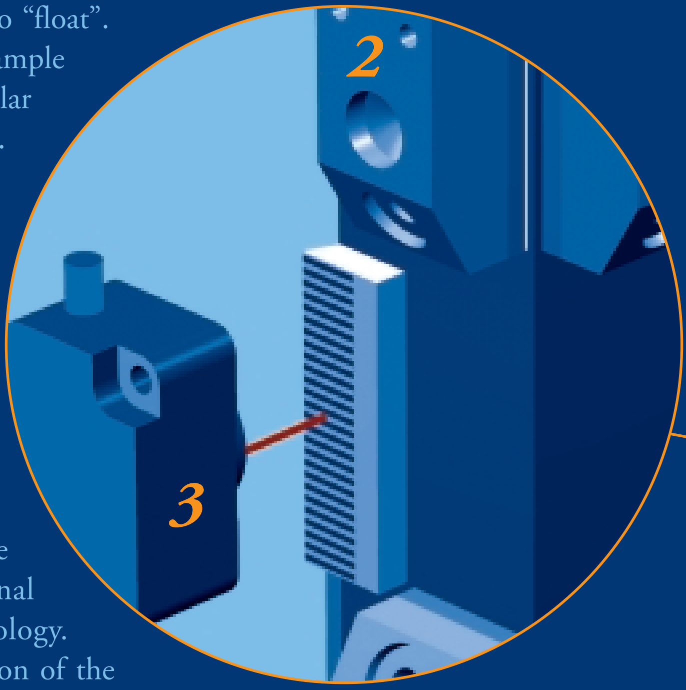
Storage Modulus	Complex/Dynamic Viscosity	Time
Loss Modulus	Creep Compliance	Stress/Strain
Storage/Loss Compliance	Relaxation Modulus	Frequency
Tan Delta (δ)	Static/Dynamic Force	Sample Stiffness
Complex Modulus	Temperature	Displacement

Design Features and Benefits

The performance of a Dynamic Mechanical Analyzer is a function of its ability to apply a force and measure displacement. The Q800 DMA incorporates the latest technology for this purpose as described below.

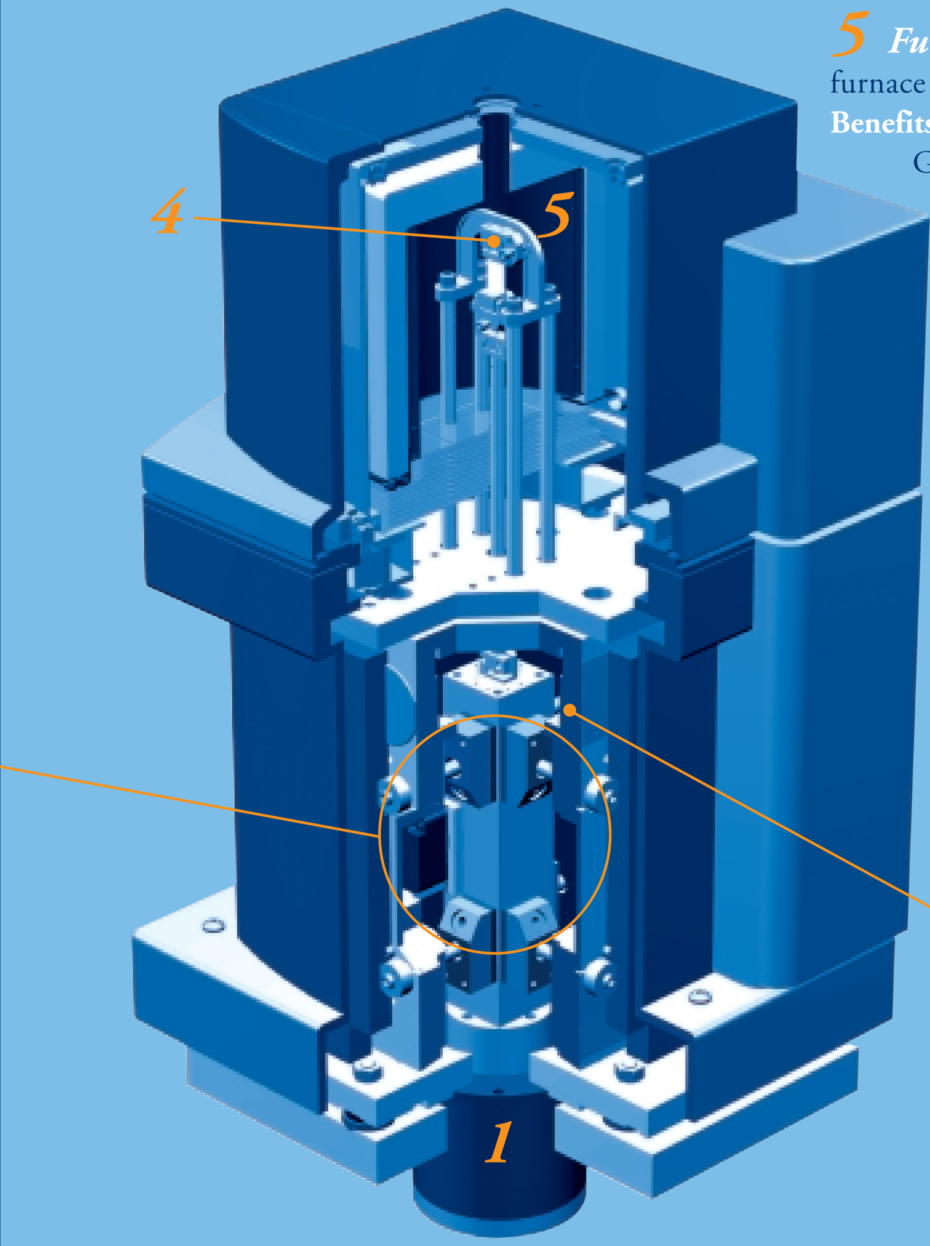
1 Drive Motor The Q800 uses a non-contact, direct drive motor to provide the oscillatory or static force required. The motor is constructed of high performance composites that ensure low compliance and is thermostated to eliminate heat build-up even when using large oscillation amplitudes and high deformation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

2 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. **Benefits:** Very weak materials like films and fibers can be characterized with ease since the drive shaft can move with virtually no friction and the unique rectangular slide prevents twist in the sample.



3 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. **Benefits:** 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.

4 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. **Benefits:** 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.

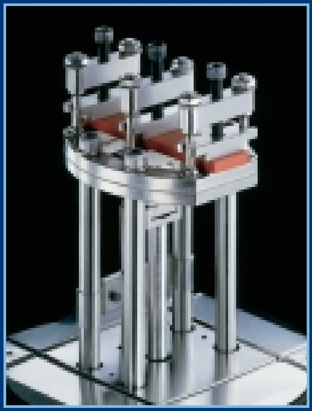


5 Furnace The Q800 features a bifilar wound furnace that automatically opens and closes. **Benefits:** The furnace design combined with the Gas Cooling Accessory provides for efficient and precise temperature control over the entire temperature range, both in heating, cooling, and isothermal operation.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

6 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. **Benefits:** The rigid aluminum housing minimizes system compliance and the temperature-controlled housing ensures precise data.

Modes of Deformation

The Q800 offers all the major deformation modes required to characterize solid bars, elastomers, soft foams, thin films and fibers. The deformation modes include bending (single cantilever, dual cantilever, and 3-point bend), shear, compression, and tension. In addition, submersible compression and film tension clamps are available.



Dual Cantilever

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.

Three clamp sizes are available:

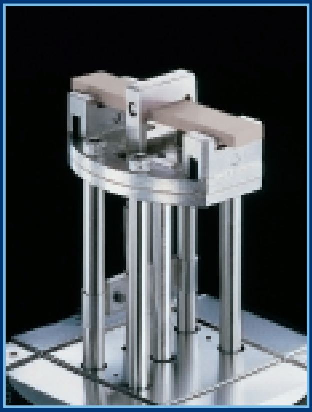
Dual/Single Cantilever	8/4mm (L), Up to 15mm (W) and 5mm (T)
Dual/Single Cantilever	20/10mm (L), Up to 15mm (W) and 5mm (T)
Dual/Single Cantilever	35/17.5mm (L), Up to 15mm (W) and 5mm (T)

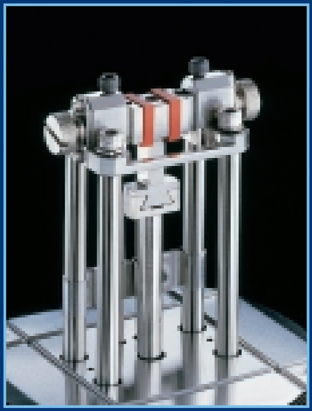
3 Point Bend

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 mm and 20 mm clamps feature a unique low-friction support using roller bearings that improves accuracy.

Three clamp sizes are available:

3-pt bend, Small	5, 10, or 15mm (L). Up to 15mm (W) and 7mm (T)
3-pt bend, 50 mm	50mm (L), Up to 15mm (W) and 7mm (T)
3-pt bend, 20 mm	20mm (L), Up to 15mm (W) and 7mm (T)





Shear Sandwich

In this mode, two equal sizes of the same material are sheared between a fixed outer plate and the moving center plate. This mode is ideal for gels, adhesives, high viscosity resins and other highly damped materials.

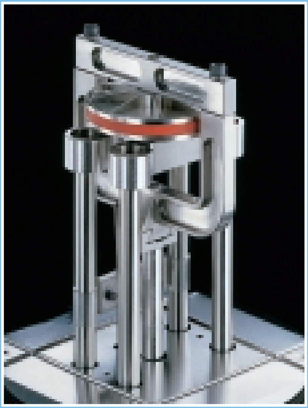
Shear Sandwich	10mm square. Up to 4mm (T)

Compression

In this mode, the sample is placed on a fixed flat surface and force is applied by an oscillating upper plate. Compression is suitable for low to moderate modulus materials (e.g., foams, elastomers), provided the material has some elasticity. With minor changes this mode can be used to make measurements of expansion or penetration using a static force.

Two clamp sizes are available:

Compression (small)	15mm diameter up to 10mm thick
Compression (large)	40mm diameter up to 10mm thick



Films

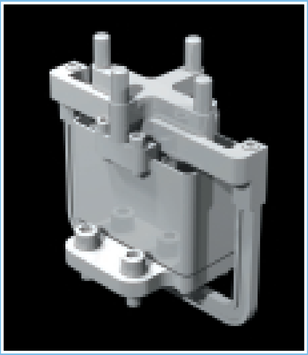
Tension

In this mode, the sample is placed in tension between a fixed and moveable clamp. In oscillation experiments, a small static force is applied in order to prevent buckling. The clamps feature very simple mounting and because the drive shaft does not rotate, no twist is induced in the sample that can lead to inaccurate data.

Two clamps are available:

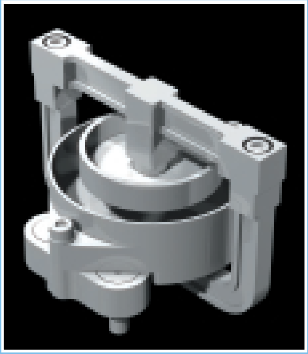
Fibers

Tension (film)	5 to 30mm (L); up to 8mm (W) and 2mm (T)
Tension (fiber)	5 to 30mm (L); 5 denier (0.57 tex) to 0.8mm diam. single fibers or fiber bundles



Submersible Clamps

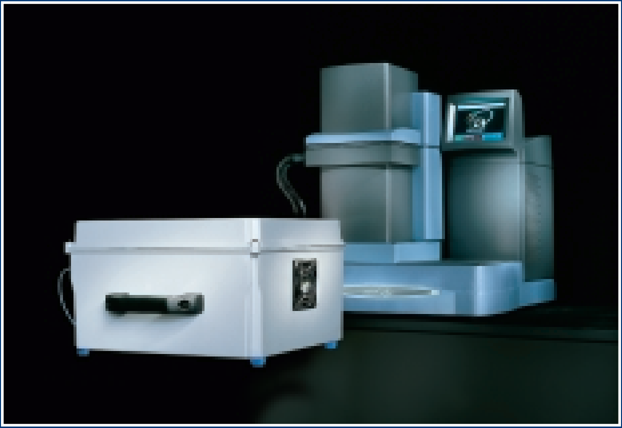
Both film tension and compression clamps are available in submersible configurations. These clamps allow samples to be analyzed in a fluid environment up to 80°C.



Accessories

Gas Cooling Accessory

The Gas Cooling Accessory (GCA) extends the operating range to subambient temperatures. 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. This 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).



Air Compressor Accessory

The Q800 uses air bearings for support of the drive shaft. The air bearings use a clean, dry compressed air or nitrogen supply that typically comes from a centralized supply. In cases where this is not possible, the Air Compressor Accessory (ACA) is available. This self-contained air compressor provides the clean, dry air supply required for the air bearings.

Accessory Kit

The standard accessory kit supplied with the Q800 provides all the necessary tools and calibration materials needed for operation. Included with the kit is the 35 mm dual/single cantilever clamp that is used for many common applications. A set of precision digital calipers is included for accurate sample dimension measurements. A torque wrench is supplied since when mounting samples, an accurate, reproducible torque must be applied to mounting screws on the sample clamp. Finally, a variety of calibration samples are provided and used for instrument calibration.

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.

Range of Material Behavior

Solid Like ←--------→ Liquid Like

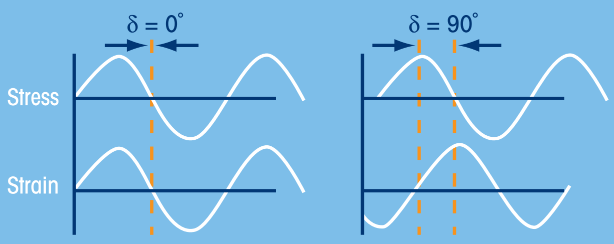
Ideal Solid ←-----→ Most Materials ←-----→ Ideal Fluid

Purely Elastic ←----→ Viscoelastic ←----→ Purely Viscous

Viscoelasticity: Having both viscous and elastic properties

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 vs. time is measured. Both tests are used to assess the viscoelastic nature of materials.

FIGURE 1

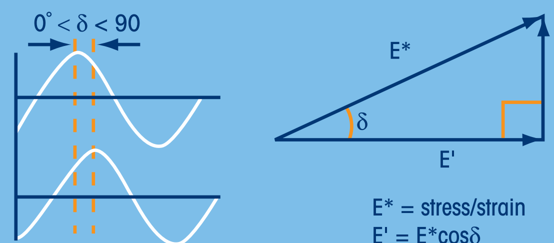


100% Elastic Behavior   100% Viscous Behavior

The most common test is the dynamic oscillatory test, where a sinusoidal stress is applied to the material and a sinusoidal strain is measured ***(Figure 1)***. Also measured is the phase difference, δ, between the two sine waves. The phase lag will be zero degrees for purely elastic materials and 90 degrees for purely viscous materials. 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 δ, 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 samples stiffness. E'', the loss modulus, is the viscous component and is related to the samples 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 component. These parameters can be calculated as a function of time, temperature, frequency, or amplitude (stress or strain) depending on the application.

FIGURE 2



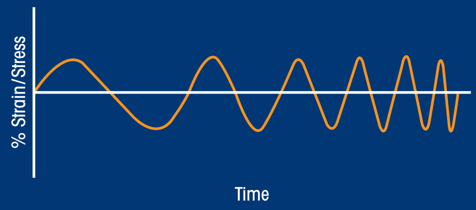
Viscoelastic Behavior

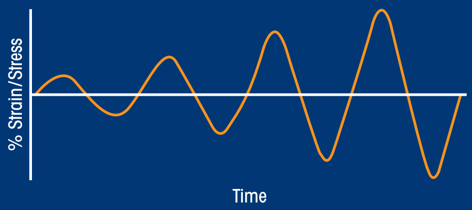
$E^* = \text{stress/strain}$
$E' = E^*\cos\delta$
$E'' = E^*\sin\delta$
$\tan\delta = E''/E'$

Modes of Operation

The Q800 DMA provides multiple operating modes that facilitate a wide variety of common experiments. The modes include:

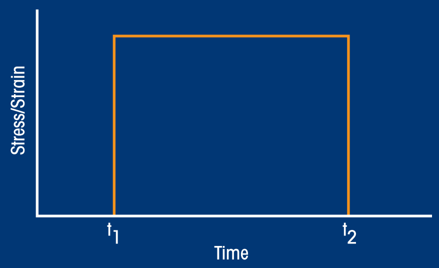
Multi-Frequency Mode: The multi-frequency mode can assess viscoelastic properties as a function of frequency, while oscillation amplitude is held constant. These tests can be run at single or multiple frequencies, in time sweep, temperature ramp, or temperature step/hold experiments.

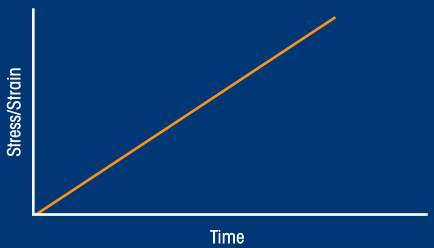




Multi-Stress/Strain Mode: In this mode, frequency and temperature are held constant, and the viscoelastic properties are monitored as % strain or the stress is varied. This mode is primarily used to identify the Linear Viscoelastic Range (LVR).

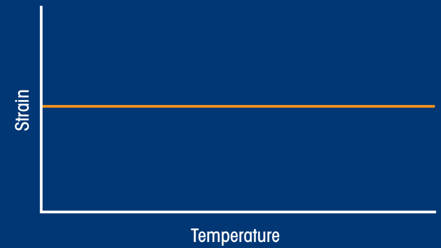
Creep/Stress Relaxation Mode: With creep, the stress is held constant and deformation is monitored as a function of time. In stress relaxation, the deformation is held constant and the stress is monitored vs. time.





Controlled Force/Strain Rate Mode: 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 Mode: With this mode, strain is held constant during a temperature ramp. Isostrain can be used to assess shrinkage force in films and fibers.

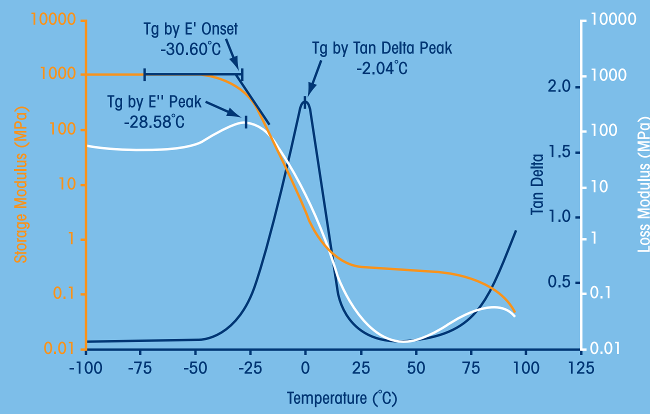


Applications

Measurement of T_g of a Pressure Sensitive Adhesive (PSA)

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. ***Figure 3*** 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.

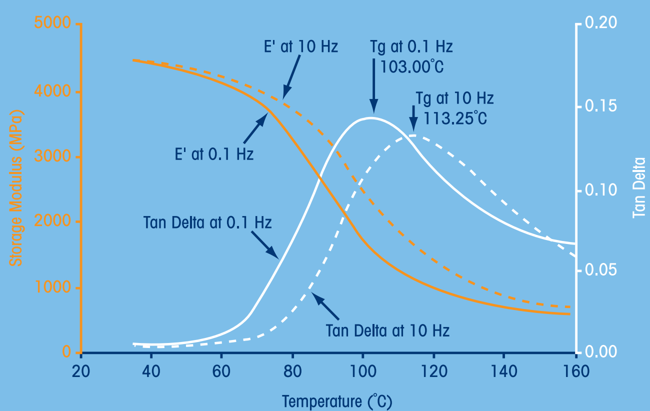
FIGURE 3



Frequency Effect on Modulus and Glass Transition of Polyethylene Terephalate (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 in ***Figure 4***. 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.

FIGURE 4



The Measurement of Secondary Transitions in Vinyl Ester

DMA is one of the only techniques that can measure β and γ secondary transitions. Secondary transitions arise from side group motion with some cooperative motion from the main chain as well as internal motion within a side group. The transitions, as shown in ***Figure 5***, 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.

FIGURE 5

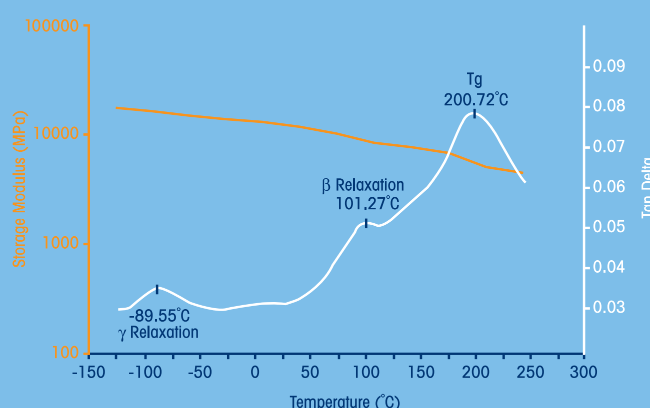
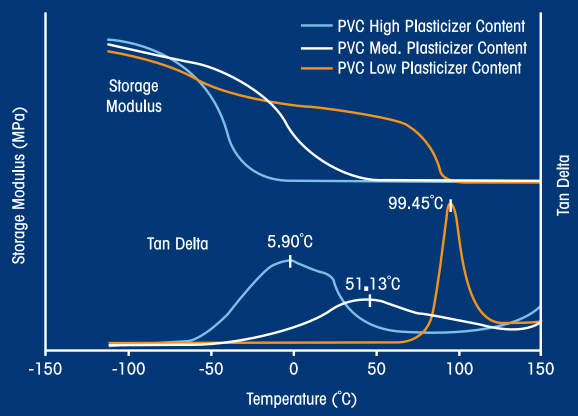


FIGURE 6



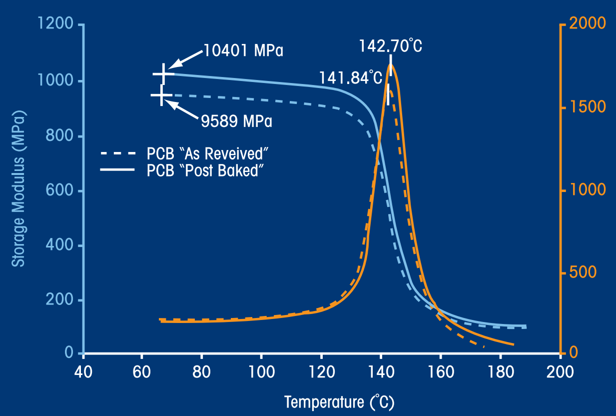
Effect of Plasticizer Content on PVC

Figure 6 illustrates another very common application for DMA. This data on Polyvinyl Chloride (PVC) was generated using the single cantilever bending mode. The three samples have varying amounts of plasticizer which dramatically effects both the absolute value of modulus as well as the Tg. Assessing the effects of plasticizer and other additives such as fillers, modifiers, and colorants on modulus and transition temperatures is a common use for DMA.

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. ***Figure 7*** shows a typical PCB run in single cantilever bending. The Tg is clearly discernible and the difference between the "as received" and "post baked" sample clearly shows the effect that further crosslinking has on both the Tg and the absolute value of modulus.

FIGURE 7



Subambient Characterization of Soft Foams

FIGURE 8

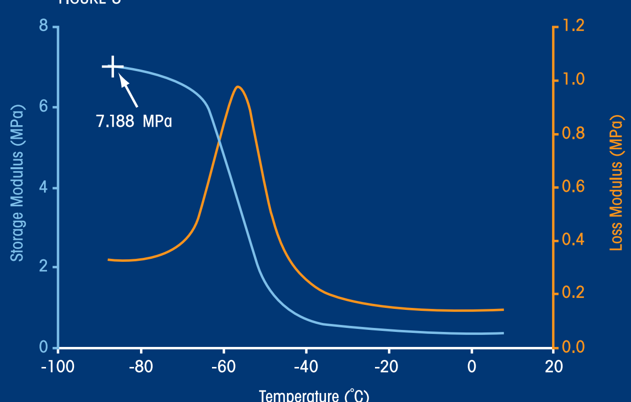


Figure 8 shows a single cantilever bending experiment on a soft foam material. These materials can also be characterized using shear and compression modes, but the single cantilever mode provides information well below the Tg. This data also illustrates the effectiveness of the Q800 for characterizing materials that exhibit very low stiffness from the glassy plateau, through the transition region, and well into the rubbery plateau. This is only possible on instruments that have superior stress and strain control.

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. ***Figure 9*** 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.

FIGURE 9

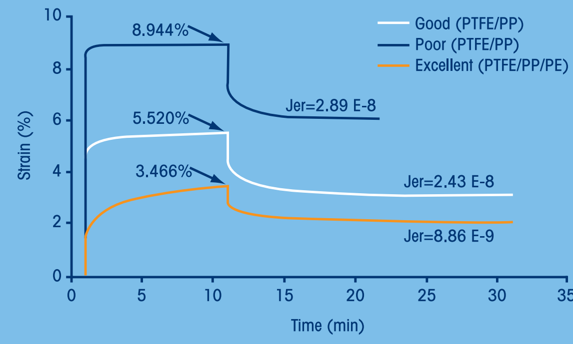
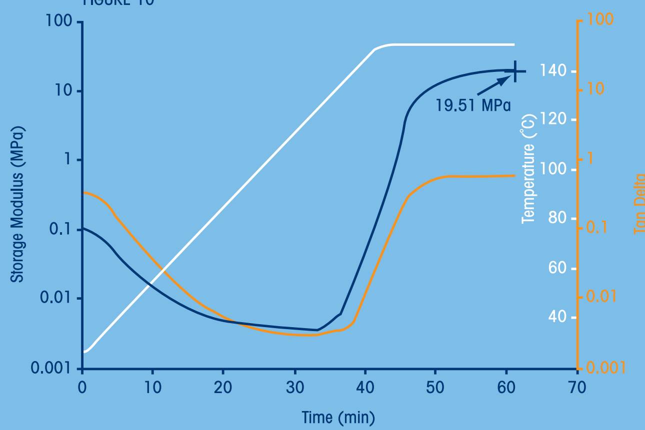


FIGURE 10



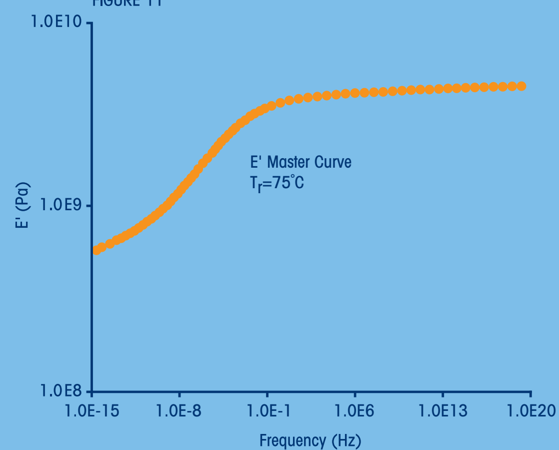
Sheet Molding Compound Cure

Figure 10 shows the cure profile of a sheet molding compound using the shear mode. In this experiment, the sample was heated to 150°C and then held isothermally. Data was collected vs. time. The sample first softens then begins to cure, reaching a final maximum modulus. This profile will be influenced greatly by the heating profile. An assessment can be made on the trade-off in physical properties for various cure profiles by conducting this experiment under various conditions.

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 an isothermal step-hold experiment 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 in ***Figure 11*** 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.

FIGURE 11



Thermal Advantage Software

A quality Thermal Analyzer requires flexible, intelligent software to power it. No one believes this more than our software engineers, who have pioneered most of the features commonly seen in today's modern thermal analyzers. Thermal Advantage™ for Q Series™ software is Microsoft Windows® based, and expandable to meet growing user needs.

Thermal Advantage - Instrument Control

- ***Multitasking –*** conducts experiments and simultaneously analyzes data
- ***Multimode –*** can operate up to 8 modules simultaneously
- ***Wizards –*** guides and prompts in setting up of experiments
- ***Real Time Plot –*** provides a real-time display of the progress of the experiment
- ***Autoqueuing –*** permits pre-programmed set-up of future experiments
- ***"How To" Feature –*** provides extensive, context sensitive assistance

Universal Analysis 2000 - Data Analysis

- ***Single Package –*** analyze data from all TA Instruments modules
- ***Picture-in-a-Picture –*** provides easy one plot analysis of large and small events
- ***Real Time Data Analysis –*** ability to analyze data "as it arrives"
- ***Ability to create up to 20 simultaneous curve overlay plots***
- ***Custom Report Generation –*** within UA 2000 using Microsoft Word® & Excel® templates
- ***Saved Analysis –*** for quick retrieval of previously analyzed data files

Time/Temperature Superpositioning (TTS)

- ***Flexible Mutifrequency Data Input –*** accepts data from ramp or step-hold experiments
- ***Accepts Creep and Stress Relaxation Data***
- ***Reference Temperature –*** user selectable reference temperature
- ***Automatic/Manual Shifting –*** provides for automatic shifting or manual shifting of data
- ***Shift Factor Curves –*** includes real-time interaction between curve shift and shift factor graphs
- ***Shift Factor Models–*** includes both WLF and Arrhenius models
- ***Master Curve–*** can be stored as separate file and generated at any explicit temperature

Why TA Instruments

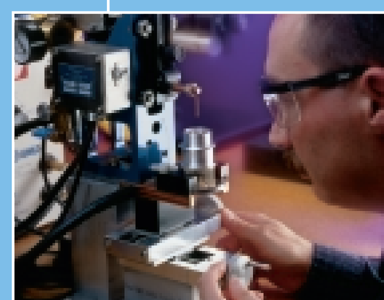
More worldwide customers choose TA Instruments than any competitor as their preferred thermal analysis and rheology supplier. We earn this distinction by best meeting customer needs and expectations for high technology products, quality manufacturing, timely deliveries, excellent training, and superior after sales-support.

Sales and Service

We pride ourselves in the technical competence and professionalism of our sales force, whose only business is thermal analysis and rheology. TA Instruments is recognized worldwide for its prompt, courteous and knowledgeable service staff. Their specialized knowledge and experience are major reasons why current customers increasingly endorse our company and products to their worldwide colleagues.

Innovative Engineering

Our company is the recognized leader for innovation and cutting-edge technology. Representative examples include the Q Series™ Modules, Modulated DSC®, the AR 2000 Rheometer with Mobius Drive™ and Smart Swap™, and the Q800 Dynamic Mechanical Analyzer. We invest twice the industry average in research and development and consistently outpace our competitors in introducing new products.

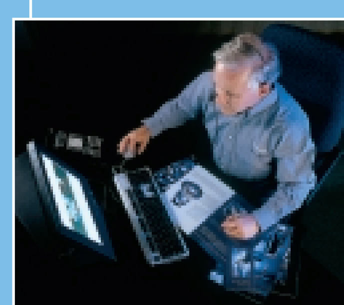


Quality Products

All thermal analyzers are manufactured to ISO 9002 procedures in our expanded plant in New Castle, DE, USA. State-of-the-art flow manufacturing procedures and a highly motivated work force ensure high quality products with industry-leading delivery times.

Technical Support

Customers prefer TA Instruments because they buy analyzers to solve problems and often need operation and applications assistance. Our worldwide technical support staff is the largest and most experienced in the industry, and are accessible daily by telephone, e-mail, or via our website. We also provide multiple training opportunities in our worldwide facilities.



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