

## DMA – RHEOLOGY OF SOLID-LIKE MATERIALS



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## Topics in the Waters – TA Characterization Workshop

- Optimizing DSC and TGA Testing
- GPC Techniques and Applications
- Rheology from Liquids to Solids
- DMA – Rheology of Solid-Like Materials

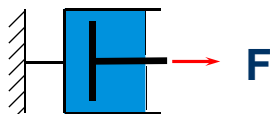
We're approaching the finish line.  
We're going to make it. 😊



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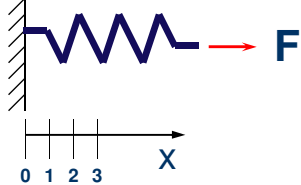
## QUICK REVIEW OF RHEOLOGY

### Rheology: The study of the flow and deformation of matter



**Flow: Fluid Behavior; Viscous Nature**

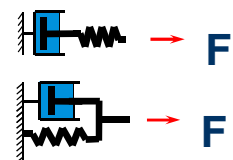
$$F = F(v); F \neq F(x)$$



**Deformation: Solid Behavior  
Elastic Nature**

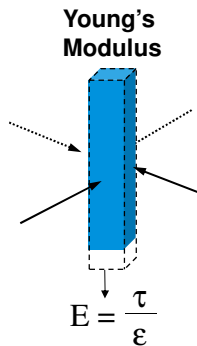
$$F = F(x); F \neq F(v)$$

We discussed these concepts in the Rheology section of the workshop.



**Viscoelastic Materials: Force depends on both Deformation and Rate of Deformation and vice versa.**

## Tensile Deformation



### Deformation Parameters

$L_0$  = Initial Length (m)

$L$  = Stretched Length (m)

$\epsilon$  = Elongational Strain,  $(L/L_0) - 1$  (unitless) (Engineering Strain)

▪ Strain is the amount of deformation normalized for the type of deformation and the dimensions of the specimen.

### Force Parameters

$T$  = Tensile force (Newtons)

$w_0$  = Initial Width (m)

$t_0$  = Initial Thickness (m)

$\tau$  = Tensile Stress,  $T/(w_0 \cdot t_0)$  (Pa)

▪ Stress is the amount of force normalized for the type of deformation and the dimensions of the specimen.

### Conversions:

Machine → Rheological

Displacement → Strain

Force → Stress

### Elongational Properties

$E = \tau/\epsilon$  (Pa) Modulus

$D = \epsilon/\tau$  (1/Pa) Compliance



## Rheological Parameters

### FLUIDS TESTING

Parameter	Shear	Elongation	Units
Rate	$\dot{\gamma}$	$\dot{\epsilon}$	Seconds <sup>-1</sup>
Stress	$\sigma$	$\tau$	Pascals
Viscosity	$\eta = \sigma/\dot{\gamma}$	$\eta_E = \tau/\dot{\epsilon}$	Pascal-seconds

### SOLIDS TESTING

Parameter	Shear	Elongation	Units
Strain	$\gamma$	$\epsilon$	Unitless
Stress	$\sigma$	$\tau$	Pascals
Modulus	$G = \sigma/\gamma$	$E = \tau/\epsilon$	Pascals

We saw these parameters in the Rheology section.



## Rheological Parameters

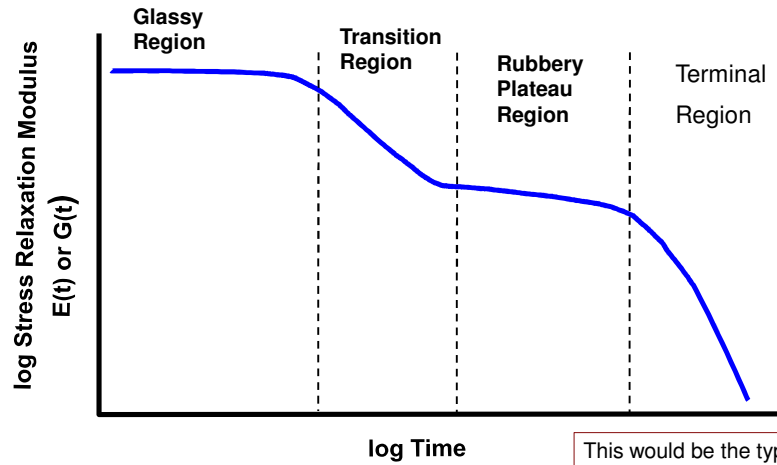
### CREEP TESTING

Parameter	Shear	Elongation	Units
Stress	$\sigma$	$\tau$	Pascals
Strain	$\gamma$	$\epsilon$	Unitless
Compliance	$J = \gamma/\sigma$	$D = \epsilon/\tau$	1/Pascals

## UNIDIRECTIONAL TYPES OF TESTS ON THE DMA

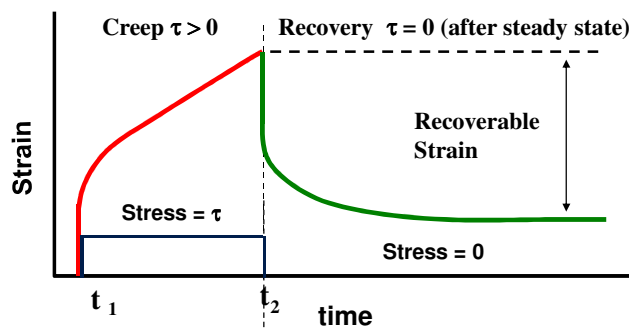
- **TRANSIENT**
  - Stress Relaxation
    - Deformation applied instantaneously  $\Rightarrow$  Force measured as a function of time
    - Deformation (mm) converted to Strain ( $\epsilon$ ), Force (N) to Stress ( $\tau$ )
    - Stress ( $\tau$ )/Strain( $\epsilon$ ) = Modulus (E)
  - Creep
    - Force applied instantaneously  $\Rightarrow$  Deformation measured as a function of time
    - Force to Stress ( $\tau$ ), Deformation converted to Strain ( $\epsilon$ )
    - Strain ( $\epsilon$ )/Stress ( $\tau$ ) = Compliance (D)
- **PRACTICAL**
  - Strain Ramp
    - Strain increased linearly with time or, optionally, exponential with the RSA-G2
  - Iso-Strain
    - Strain held constant as temperature is varied
  - Stress Ramp
    - Stress increased linearly or exponentially with time
  - Controlled Stress
    - Stress held constant as temperature is varied

## Stress Relaxation: Material Response



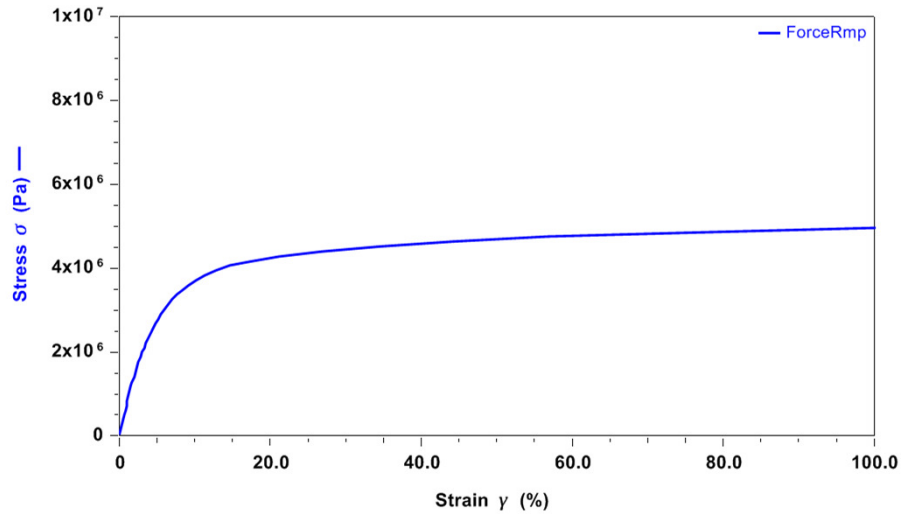
This would be the type of curve expected for an uncrosslinked polymer if given sufficient time.

## Creep Testing



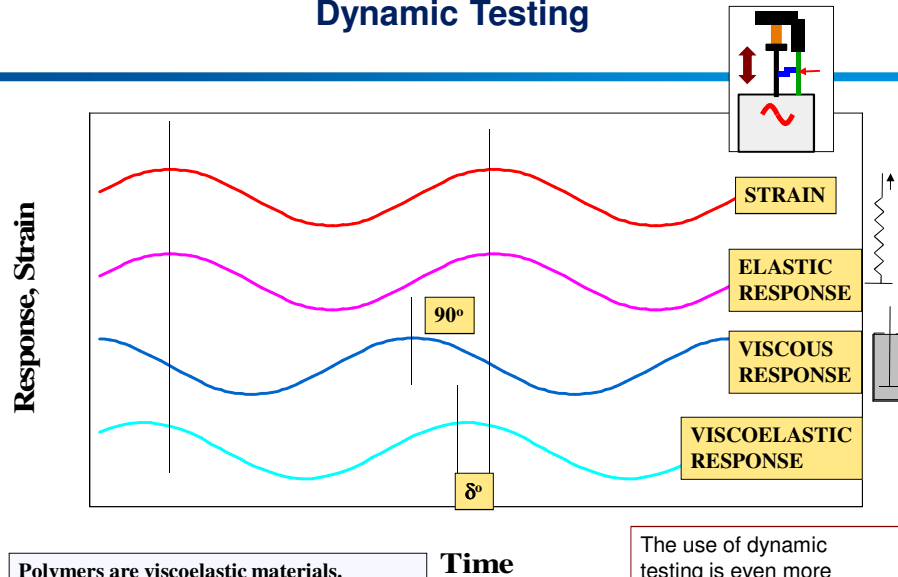
The greater the elasticity, the greater the recovery.

## Polyethylene Stress Ramp



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## Dynamic Testing



Polymers are viscoelastic materials.  
Both components – viscosity and elasticity  
– are important.

The use of dynamic testing is even more prevalent with DMA than it is with rheometers.

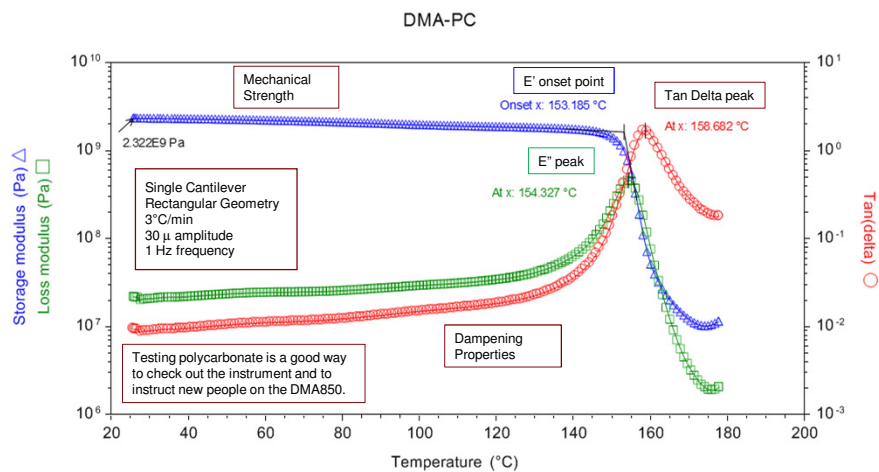
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## Dynamic Rheological Parameters

Parameter	Shear	Elongation	Units
Strain	$\gamma = \gamma_0 \sin(\omega t)$	$\varepsilon = \varepsilon_0 \sin(\omega t)$	---
Stress	$\sigma = \sigma_0 \sin(\omega t + \delta)$	$\tau = \tau_0 \sin(\omega t + \delta)$	Pa
Storage Modulus (Elasticity)	$G' = (\sigma_0/\gamma_0) \cos \delta$	$E' = (\tau_0/\varepsilon_0) \cos \delta$	Pa
Loss Modulus (Viscous Nature)	$G'' = (\sigma_0/\gamma_0) \sin \delta$	$E'' = (\tau_0/\varepsilon_0) \sin \delta$	Pa
Tan $\delta$	$G''/G'$	$E''/E'$	---
Complex Modulus	$G^* = (G'^2 + G''^2)^{0.5}$	$E^* = (E'^2 + E''^2)^{0.5}$	Pa
Complex Viscosity	$\eta^* = G^*/\omega$	$\eta_E^* = E^*/\omega$	Pa-sec

We will be mainly concerned with the Elongation column in this table.

## Polycarbonate Testing on the DMA 850



This is the main test performed on DMA instruments.

## E' Onset, E'' Peak, and $\tan \delta$ Peak

- **E' Onset:** Occurs at the lowest temperature - Relates to mechanical Failure
- **E'' Peak:** Occurs at the middle temperature - more closely related to the physical property changes attributed to the glass transition in plastics. It reflects molecular processes - agrees with the idea of  $T_g$  as the temperature of the onset of segmental motion.
- **$\tan \delta$  Peak:** Occurs at the highest temperature - used historically in literature - a good measure of the "leatherlike" midpoint between the glassy and rubbery states - height and shape change systematically with amorphous content.

Turi, Edith, A, Thermal Characterization of Polymeric Materials, Second Edition, Volume I., Academic Press, Brooklyn, New York, P. 980.



## DMAs from TA Instruments

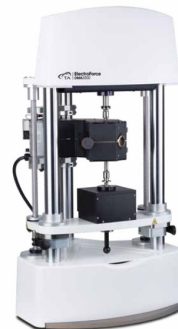
**RSA G2**



**Discovery DMA850**



**ESG 3200**

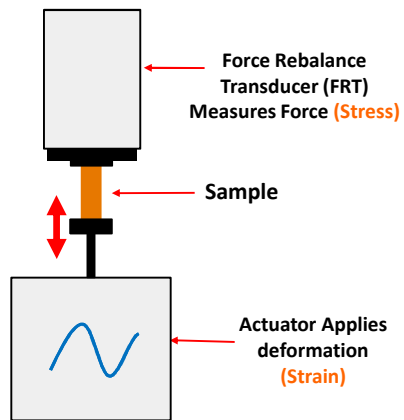




## DMAs from TA Instruments

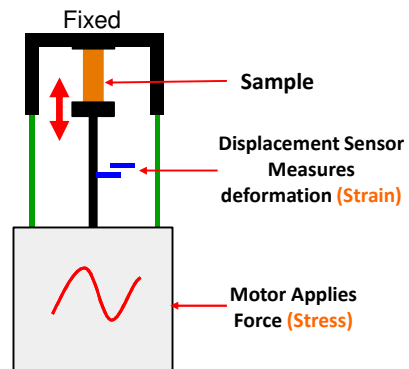
### RSA G2

*Separate Motor & Transducer*

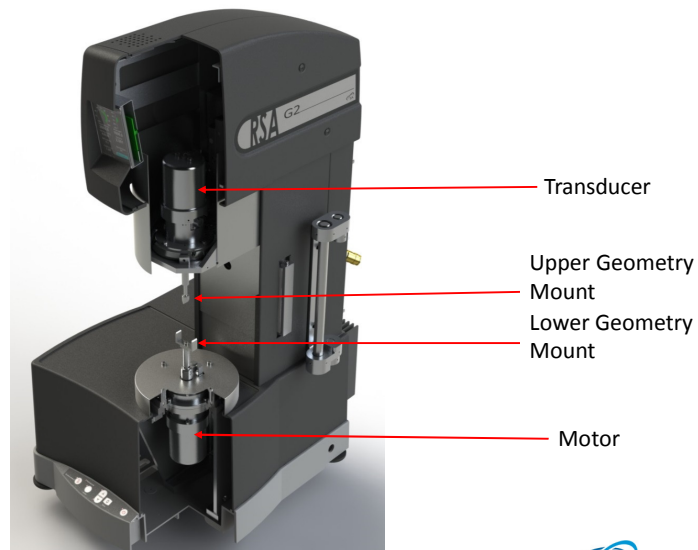


### DMA850

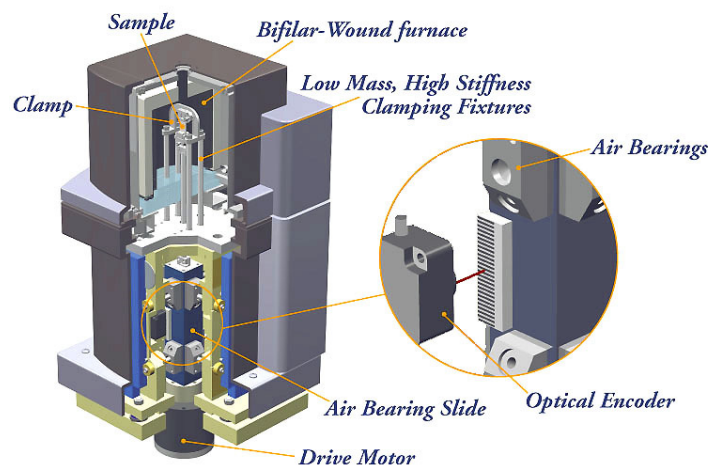
*Combined Motor & Transducer*



## RSA G2: Schematic Dual Head Design



## DMA850: Schematic



## DMA Specifications

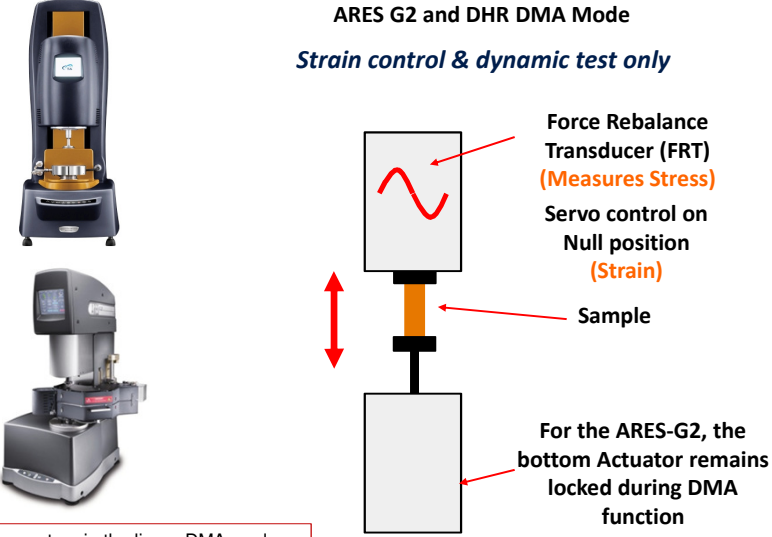
	RSA G2	DMA850	Q800
Max Force	35 N	18 N	18 N
Min Force	0.0005 N	0.0001 N	0.0001 N
Displacement Resolution	1 nm	* 0.1 nm	1 nm
Frequency Range	$2 \times 10^{-6}$ to 100 Hz	* $1 \times 10^{-4}$ to 200 Hz	$1 \times 10^{-2}$ to 200 Hz
Dynamic Deformation Range	$\pm 5 \times 10^{-5}$ to 1.5 mm	* $\pm 5 \times 10^{-6}$ to 10 mm	$\pm 5 \times 10^{-4}$ to 10 mm
Temperature range	-150 to 600 °C	-150 to 600 °C	-150 to 600 °C
Isothermal Stability	$\pm 0.1$	$\pm 0.1$	$\pm 0.1$
Heating Rate	0.1 °C to 60 °C/min	0.1 °C to 20 °C/min	0.1 °C to 20 °C/min
Cooling Rate	0.1 °C to 60 °C/min	0.1 °C to 10 °C/min	0.1 °C to 10 °C/min

\* Denotes improvement by DMA 850 compared with Q800



## DMA Mode on DHR and ARES-G2

**ARES G2 and DHR DMA Mode**  
*Strain control & dynamic test only*



Force Rebalance Transducer (FRT)  
 (Measures Stress)

Servo control on Null position  
 (Strain)

Sample

For the ARES-G2, the bottom Actuator remains locked during DMA function

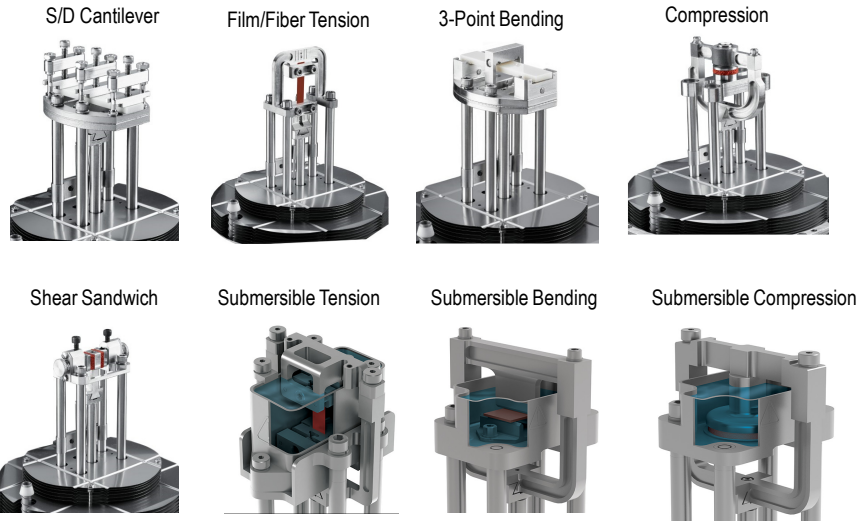
With the rheometers in the linear DMA mode, both the DHR and ARES-G2 are CMT design.

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## Specifications of the DHR-DMA and the ARES-G2 DMA

	DHR – DMA mode	ARES-G2 DMA mode
Motor Control	FRT	FRT
Minimum Force (N) Oscillation	0.1	0.001
Maximum Axial Force (N)	50	20
Minimum Displacement (μm) Oscillation	1.0	0.5
Maximum Displacement (μm) Oscillation	100	50
Displacement Resolution (nm)	10	10
Axial Frequency Range (Hz)	1 x 10 <sup>-5</sup> to 16	1 x 10 <sup>-5</sup> to 16

## Clamps for DMA850



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## DMA 850 Sample Sizes

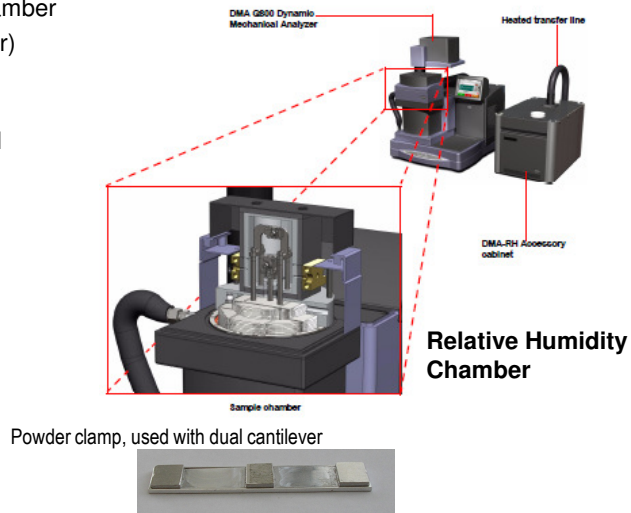
<b>Dual/Single Cantilever</b>	<b><math>L = 8/4 \text{ mm}; W \leq 15 \text{ mm}; T \leq 5 \text{ mm}</math></b> <b><math>L = 20/10 \text{ mm}; W \leq 15 \text{ mm}; T \leq 5 \text{ mm}</math></b> <b><math>L = 35/17.5 \text{ mm}; W \leq 15 \text{ mm}; T \leq 5 \text{ mm}</math></b>
<b>3-Point Bend</b>	<b><math>L = 5, 10 \text{ or } 15 \text{ mm}; W \leq 15 \text{ mm}; T \leq 7 \text{ mm}</math></b> <b><math>L = 20 \text{ mm}; W \leq 15 \text{ mm}; T \leq 7 \text{ mm}</math></b> <b><math>L = 50 \text{ mm}; W \leq 15 \text{ mm}; T \leq 7 \text{ mm}</math></b>
<b>Tension</b>	<b><math>L = 5 \text{ to } 30 \text{ mm}; W \leq 8 \text{ mm}; T \leq 2 \text{ mm}</math></b> <b><math>L = 5 \text{ to } 30 \text{ mm}; 5 \text{ denier}; D \leq 0.8 \text{ mm}</math></b>
<b>Shear</b>	<b>10 mm square; <math>T \leq 4 \text{ mm}</math></b>
<b>Compression</b>	<b>15 and 40 mm diameter; <math>T \leq 10 \text{ mm}</math></b>

For cantilever, 3-pt bend, and tension, these dimensions refer to length between clamps.  
Allow for an additional ~20 mm for the clamps.

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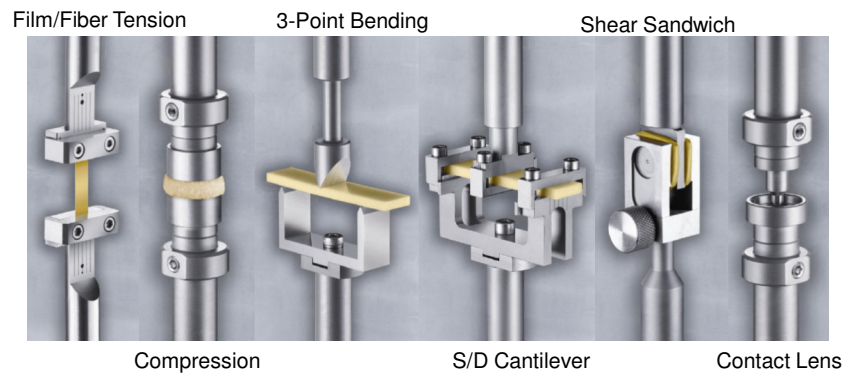
## DMA 850 – Other Accessories

- Relative Humidity Chamber (described later)
- Powder Kit for 35-mm Cantilever Clamp
- Low Friction 3-pt bend
- Penetration Clamp
- Glass Support Cloth



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## Clamps for RSA G2



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## RSA G2 Immersion Clamps

- Immersion clamp kit offers 3 geometries with temperature control from -10 to 200 °C in the FCO.

Tension



Compression



3 Point Bending



**Tension:** Up to 25 mm long, 12.5 mm wide and 1.5 mm thick.

**Compression:** 15 mm in diameter; maximum sample thickness is 10 mm.

**Three Point Bending:** includes interchangeable spans for lengths of 10, 15, and 20 mm. Maximum sample width is 12.5 mm and maximum thickness is 5 mm.



## DMA Clamping Guide

Sample	Clamp	Sample Dimensions
High modulus metals or composites	3-point Bend Dual Cantilever Single Cantilever	$L/T > 10$ if possible
Unreinforced thermoplastics or thermosets	Single Cantilever	$L/T > 10$ if possible
Brittle solid (ceramics)	3-point Bend Dual Cantilever	$L/T > 10$ if possible
Elastomers	Dual Cantilever Single Cantilever Shear Sandwich Tension	$L/T > 20$ for $T < T_g$ $L/T > 10$ for $T < T_g$ (only for $T > T_g$ ) $T < 2$ mm $W < 5$ mm
Films/Fibers	Tension	$L$ 10-20 mm $T < 2$ mm
Supported Systems	8 mm Dual Cantilever	minimize sample, put foil on clamps



## RSA-G2 DETA



- RSA-G2 can be positioned as two instruments in one.
  - DMA/Solids Analyzer
  - Dielectric Analyzer

Attribute	Specification
Geometry	25 mm PP
Temperature System	FCO, Force Convection
Compatibility	Oven
ARES/RSA to DE Bridge Interface	IEEE Internal to Instrument
Temperature Range	-160° to 300°C



Keysight LCR Meter  
Model 4980A

(LCR Meter)	Frequency	AC Test Signal (potential)
Keysight E4980A	20 Hz to 2 MHz	0.005 to 20 Volts
Keysight E4980AL/120	20 Hz to 1 MHz	0.001 to 2 Volts

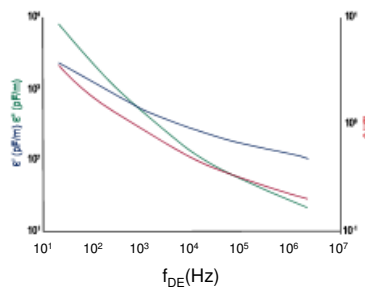


## DETA Testing

### Frequency Sweep

Figure 15 shows an example of a dielectric frequency sweep run on a rubber sample under ambient conditions. Shown are the storage and loss permittivity and loss tangent over a frequency range of 20 Hz to 2 MHz.

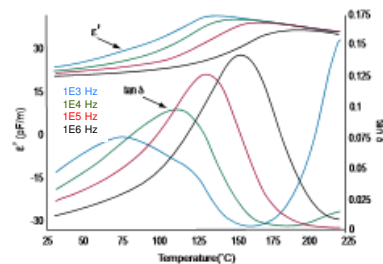
Figure 15: Dielectric Frequency Sweep on Rubber



### Temperature Ramp

Figure 16 shows an example of a ramp on a PMMA sample at four different Dielectric Frequencies ranging from 1kHz to 1MHz. It can be seen here that the magnitude of  $\epsilon'$  decreases with increasing frequency through the transition region and the peak of the transition in  $\tan \delta$  moves to higher temperatures with increasing frequency.

Figure 16: DETA Temperature Ramp on PMMA



## Testing Solids on a Rheometer

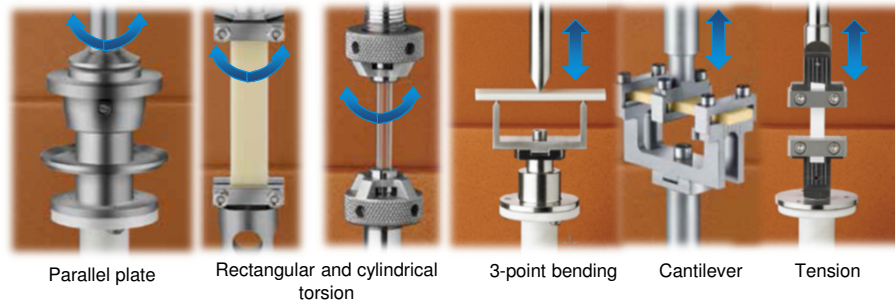
- Torsion (rotational) and DMA (axial) geometries allow solid samples to be characterized in a temperature controlled environment.

$$E = 2G(1 + \nu)$$

$\nu$  : Poisson's ratio

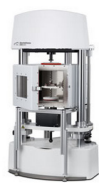
Shear Modulus:  $G'$ ,  $G''$ ,  $G^*$

Young's Modulus:  $E'$ ,  $E''$ ,  $E^*$



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## ESG DMA Instruments



ElectroForce DMA 3200 High Force DMA



ElectroForce 3100



ElectroForce 3200



ElectroForce 3300



ElectroForce 3500

The ESG division of TA Instruments also provides DMA measurements. Many of these can impose very high forces. See the spec sheet on the following page for the force capabilities.

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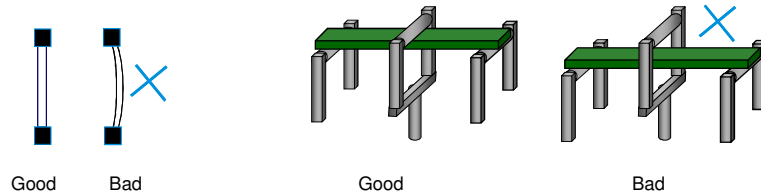


### ESG Specs – much higher force capacity than DMA 850 and RSA-G2

	3100	3200	3300	3510
<b>Linear Motor</b>				
Standard				
Peak/Max Sine	± 22 N	± 225 N	± 1000 N	± 7500 N
Static or RMS (continuous)	± 22 N	± 160 N	± 700 N	± 5300 N
<b>High Force Option</b>				
Peak/Max Sine	—	± 450 N	± 3000 N	—
Static or RMS (continuous)	—	± 320 N	± 2100 N	—
Displacement	5 mm	13 mm	25 mm	50 mm
Extended Stroke Option	—	150 mm	150 mm	—
<b>Linear Velocity</b>				
	0.0025 µm/s – 1.0 m/s	0.0065 µm/s – 3.2 m/s	0.013 µm/s – 1.5 m/s <sup>(1)</sup> 0.013 µm/s – 2.0 m/s <sup>(2)</sup>	0.025 µm/s – 1.5 m/s
<b>Frequency</b>				
	0.00001 Hz – 100 Hz	0.00001 Hz – 300 Hz	0.00001 Hz – 100 Hz	0.00001 Hz – 100 Hz
<b>Torsional Motor Option</b>				
Standard				
Peak/Max	—	± 5.6 N-m	± 14 N-m <sup>(3)</sup> / ± 24 N-m <sup>(4)</sup>	± 49 N-m
Static or RMS (continuous)	—	± 5.6 N-m	± 14 N-m <sup>(3)</sup> / ± 24 N-m <sup>(4)</sup>	± 42 N-m
<b>High Torque Option</b>				
Peak/Max	—	—	± 49 N-m <sup>(3)</sup>	± 70 N-m
Static or RMS (continuous)	—	—	± 42 N-m <sup>(3)</sup>	± 50 N-m
Rotation	—	Multi-Turn (± 20 revolutions Standard)	Multi-Turn (± 20 revolutions Standard)	Multi-Turn (± 20 revolutions Standard)
<b>Thermal Chamber Option</b>				
	—	-150 to 315°C	-150 to 350°C	-150 to 350°C
<b>Fluid/Saline Bath Option</b>				
	Ambient to 40°C	Ambient to 40°C	Ambient to 40°C	Ambient to 40°C



### Some Clamps Require an Offset (Static) Force



1. With the tension clamp, one wants to avoid the buckling of the specimen that is shown with the tension specimen on the right.
2. With the 3-pt bend clamp, one wants to avoid the loss of contact of the center clamp with the specimen that is shown with the 3-pt bend specimen on the right. The same principle applies to the compression clamp.

#### Clamps without offset force:

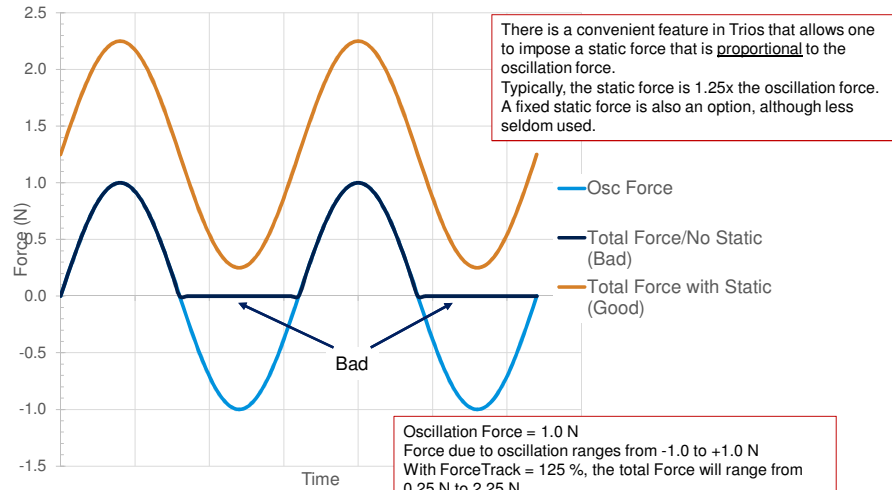
Single Cantilever  
Dual Cantilever  
Shear Sandwich

#### Clamps with offset force:

Tension Film  
Tension: Fiber  
3-Point Bend  
Compression  
Penetration

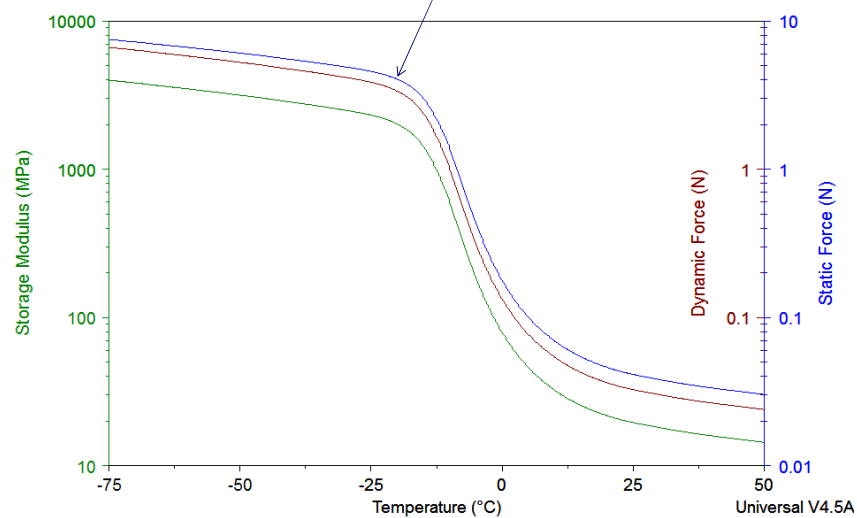


## Waveforms with and without Static Force



## Temperature Ramp with Force Track

- Static Force tracks Dynamic Force throughout Temperature Ramp



## Force Track in Trios with the DMA 850

✕ Clamp: Film Clamp

✕ Procedure: Sample Temperature Ramp

Initial/preload force  N

☒ Use Force Track  %

^ 1: Oscillation Temperature Ramp

Amplitude   $\mu\text{m}$

Frequency  Hz

☐ Use current temperature

Ramp from   $^{\circ}\text{C}$  to   $^{\circ}\text{C}$

Ramp rate   $^{\circ}\text{C}/\text{min}$

Soak times

at Start temperature  hh:mm:ss

at End temperature  hh:mm:ss

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## Force Track in Trios with the RSA-G2

^ 1: Conditioning Options

Axial force adjustment

Mode

☒ Tension ☐ Compression

Axial force  N ☒ Set initial value

Sensitivity  N

Proportional force Mode  ☐ Compensate for modulus

Axial Force > Dynamic Force  %

Minimum axial force  N

Programmed Extension Below  Pa

^ Advanced

Max gap change up   $\mu\text{m}$

Max gap change down   $\mu\text{m}$

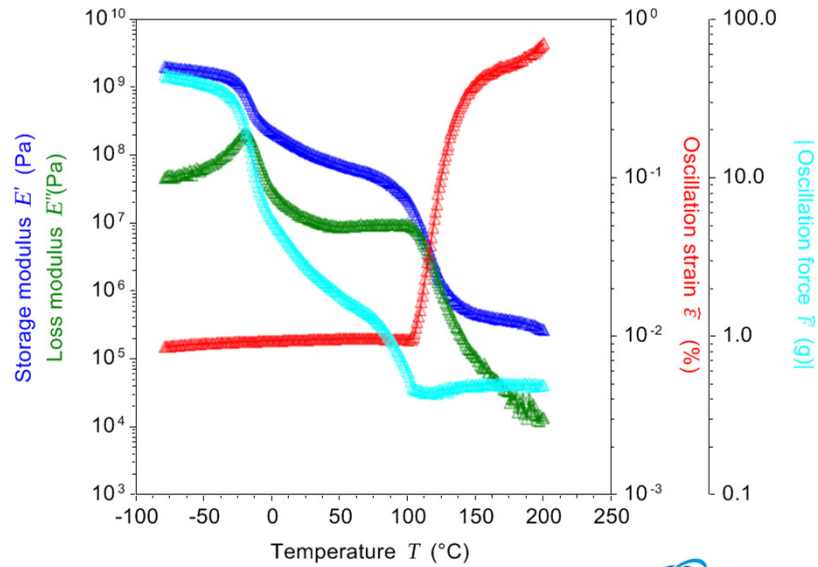
☒ Return to window ☐ Return to initial value

Priority ☒ Data sampling ☐ Force control

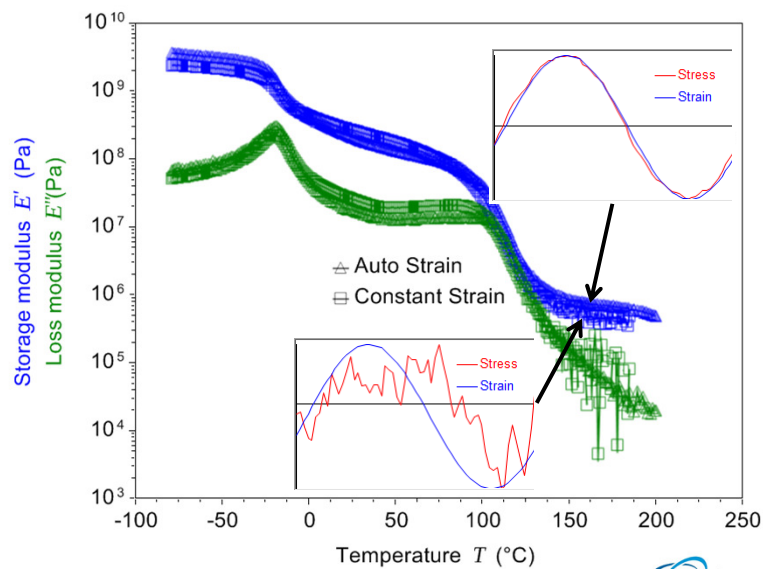
Adjustment time out  hh:mm:ss

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## Auto Strain- Temperature Ramp



## Temperature Ramp with and without Auto Strain



## Strain Adjustment during the Temperature Ramp

### RSA-G2: Auto-Strain

#### 1: Conditioning Options

Axial force adjustment

Mode

☒ Tension ☐ Compression

Auto strain adjustment

Mode

Strain adjust  %

Minimum strain  %

Maximum strain  %

Minimum force  N

Maximum force  N

### DMA 850: Auto-Ranging

#### Test Settings

Controlled Test Parameter

☐ Amplitude ☒ Strain ☐ Stress ☐ Force

☒ Enable Direct Strain

Data Sampling Mode

Sampling interval  s/pt

Data acquisition

☒ Standard ☐ Fast ☐ Enhanced ☐ User defined

☐ Zero displacement at start

☐ Measure again after method equilibration

☒ Save waveform

Auto Range Mode

☐ Standard ☒ Enhanced

Minimum force  N

Maximum force  N

Minimum oscillation displacement   $\mu\text{m}$

Maximum oscillation displacement   $\mu\text{m}$

Limit checking

☐ Enabled



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## Trios Software

- Non-restricted, available for free on our website
- Versatile
- User Friendly



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## Test Modes for the DMA 850 in Trios

Conditioning  
Oscillation  
Strain Control  
Stress Control  
Rate Control

Temperature  
Data  
Other  
Repeat  
Stress  
Strain

Conditioning  
Oscillation  
Strain Control  
Stress Control  
Rate Control

Creep  
Creep Recovery  
Creep TTS  
IsoStress

Conditioning  
Oscillation  
Strain Control  
Stress Control  
Rate Control

Frequency Sweep  
Strain Sweep  
Stress Sweep  
Temperature Sweep  
Temperature Ramp  
Time Sweep  
Temperature Sweep (Multifrequency)  
Fatigue Test  
Temperature Ramp (Multifrequency)

Conditioning  
Oscillation  
Strain Control  
Stress Control  
Rate Control

Strain Ramp  
Stress Ramp

Conditioning  
Oscillation  
Strain Control  
Stress Control  
Rate Control

Stress Relaxation  
Stress Relaxation TTS  
IsoStrain

We discussed the features of Trios in the Rheology presentation. Most of those features are also available with the DMA's. You can devise any kind of test that you want, including mixing test modes.



## Test Modes for the RSA-G2 in Trios

Oscillation  
Step (Transient)  
Other  
Conditioning  
Script

Frequency  
Temperature Ramp  
Temperature Sweep  
Amplitude  
Time  
Multiwave  
Fast Sampling  
Cycle Sweep

Oscillation  
Step (Transient)  
Other  
Conditioning  
Script

Transducer  
Sample  
Stress Control  
Dielectric  
UV Curing  
ElectroRheology  
Sample Loading  
End Of Test  
Advanced Options  
Options

Oscillation  
Step (Transient)  
Other  
Conditioning  
Script

Creep  
Stress Relaxation  
Sine Strain

Oscillation  
Step (Transient)  
Other  
Conditioning  
Script

Source

Oscillation  
Step (Transient)  
Other  
Conditioning  
Script

Axial  
Arbitrary Wave  
Temperature Ramp IsoForce  
Temperature Ramp IsoStrain

There are some minor differences between the choices for the RSA-G2 and the DMA 850.



## Common Dynamic (Oscillatory) Testing

- Available oscillatory test modes
  - Strain (stress) Sweep
  - Time Sweep
  - Frequency Sweep
  - Temperature ramp
  - Temperature Step (Sweep) (TTS)

**Typical Sequence:**  
Strain Sweep → Temperature Ramp



## Trios Oscillation Strain Sweep for the DMA 850

1: Oscillation Strain Sweep

Frequency	1.0	Hz
Sweep Mode		
<input type="radio"/> Logarithmic <input checked="" type="radio"/> Linear <input type="radio"/> Discrete		
Amplitude	5.0	um to 50.0 um
Increment	5.0	um
Number of sweeps		
1		

Test Settings

Controlled Test Parameter

☒ Amplitude ☐ Strain

☐ Enable Direct Strain

Data acquisition

☒ Standard ☐ Fast ☐ Enhanced ☐ User defined

☐ Zero displacement at start

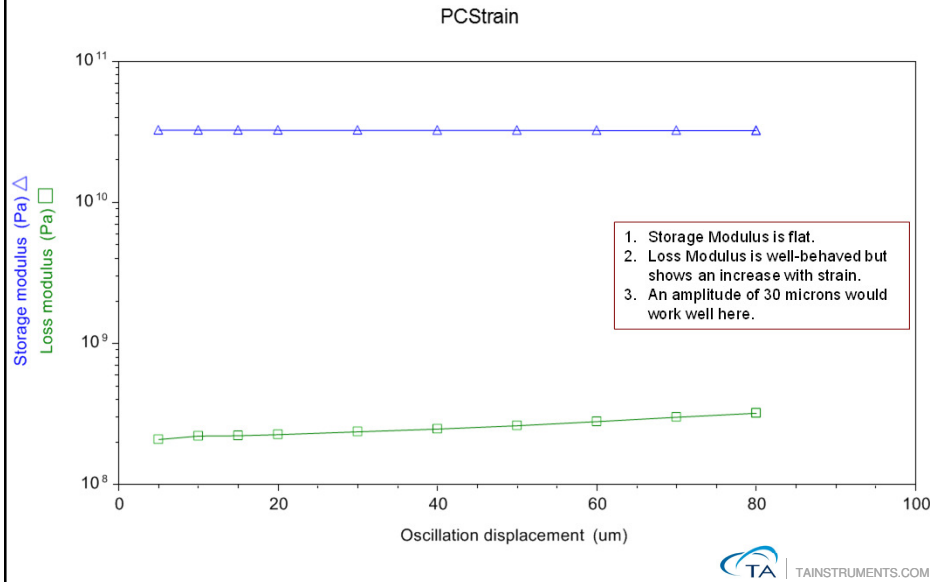
☐ Measure again after method equilibration

☒ Save waveform

There are some differences for the RSA-G2, but the DMA principles are the same as those for the DMA 850.



## Dynamic Strain Sweep – Trios Plot



## Trios Oscillation Temperature Ramp for the DMA 850

1: Oscillation Temperature Ramp

Amplitude: 20.0 um  
Frequency: 1.0 Hz

☒ Use current temperature  
Ramp from: Current °C to 180 °C  
Ramp rate: 3.0 °C/min  
Soak times:  
at Start temperature: 0  
at End temperature: 00:00:00 hh:mm:ss  
Estimated time to complete: hh:mm:ss

Test Settings

Controlled Test Parameter:  
☒ Amplitude ☐ Strain ☐ Stress ☐ Force  
☐ Enable Direct Strain

Data Sampling Mode:  
Sampling interval: 3.0 s/pt

Data acquisition:  
☒ Standard ☐ Fast ☐ Enhanced ☐ User defined  
☐ Zero displacement at start  
☐ Measure again after method equilibration  
☒ Save waveform

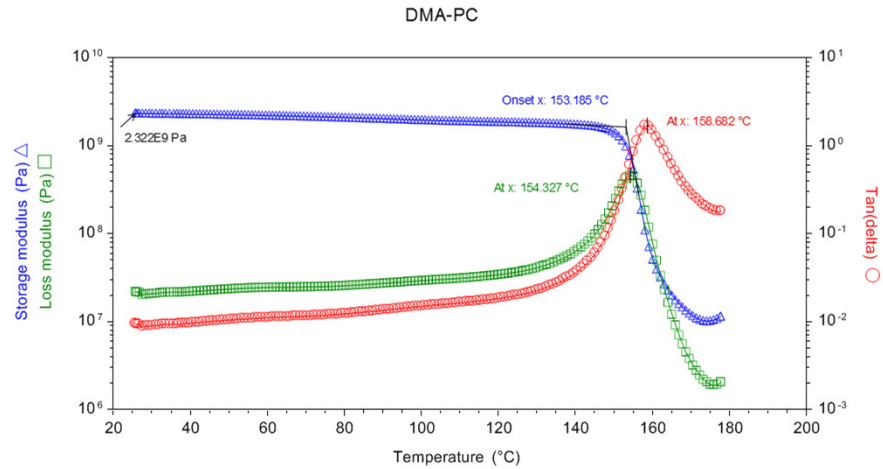
Auto Range Mode:  
☒ Standard ☐ Enhanced  
Minimum force: 0.1 N  
Maximum oscillation displacement: 1000.0 um

Again, the method for the RSA-G2 would be essentially the same.

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## Polycarbonate Testing on the DMA 850



This is the same graph that we saw at the beginning of the dynamic oscillation section.  
This is a very common test.

More Examples

## Polymer Structure-Property Characterization

- Glass transition
- Secondary transitions
- Crystallinity
- Molecular weight/cross-linking
- Phase separation (polymer blends, copolymers,...)
- Composites
- Aging (physical and chemical)
- Curing of networks
- Orientation
- Effect of additives

Turi, Edith, A, Thermal Characterization of Polymeric Materials, Second Edition, Volume I., Academic Press, Brooklyn, New York, P. 489.



## The Glass & Secondary Transitions

Glass Transition - Cooperative motion among a large number of chain segments, including those from neighboring polymer chains

### Secondary Transitions

Local Main-Chain Motion - intramolecular rotational motion of main chain segments four to six atoms in length

- Side group motion with some cooperative motion from the main chain
- Internal motion within a side group without interference from side group.
- Motion of or within a small molecule or diluent dissolved in the polymer (eg. plasticizer.)

Turi, Edith, A, Thermal Characterization of Polymeric Materials, Second Edition, Volume I., Academic Press, Brooklyn, New York, P. 487.

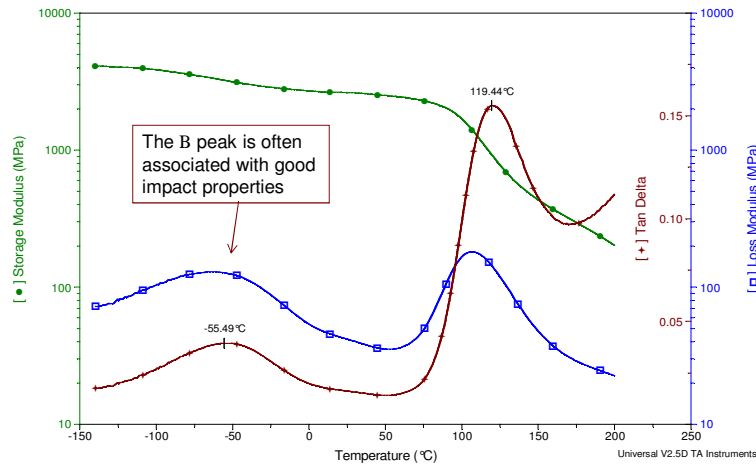


## Primary and Secondary Transition in PET Film

Sample: PET Film in Machine Direction  
Size: 8.1880 x 5.5000 x 0.0200 mm  
Method: 3 °C/min ramp  
Comment: 1Hz; 3 °C/min from -140 ° to 150 °C, 15 microns,

DMA

File: A:\Petmd.001  
Operator: RRU  
Run Date: 27-Jan-99 13:56



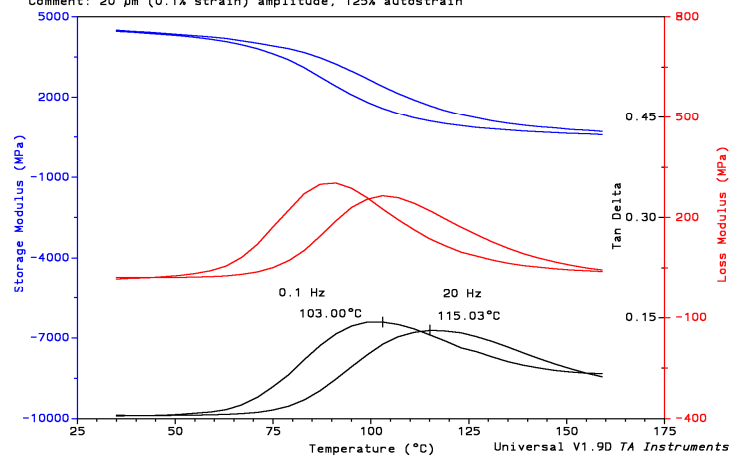
Universal V2.5D TA Instruments

## PET Film: Effect of Frequency

Sample: PET Tape Demonstration Sample  
Size: 19.8650 x 6.4200 x 0.0750 mm  
Method: step-iso frequency sweeps  
Comment: 20  $\mu$ m (0.1% strain) amplitude, 125% autostrain

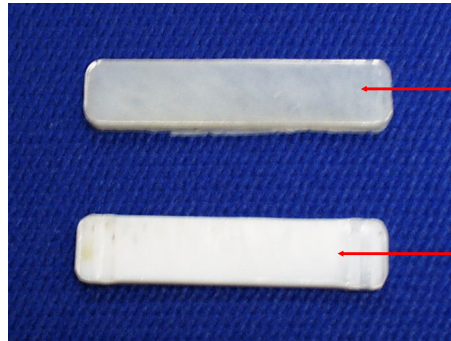
DMA

File: D:\TA\DMA\DATA\DMA-PET.001  
Operator: Apps. Lab  
Run Date: 25-Feb-97 14:01



Universal V1.9D TA Instruments

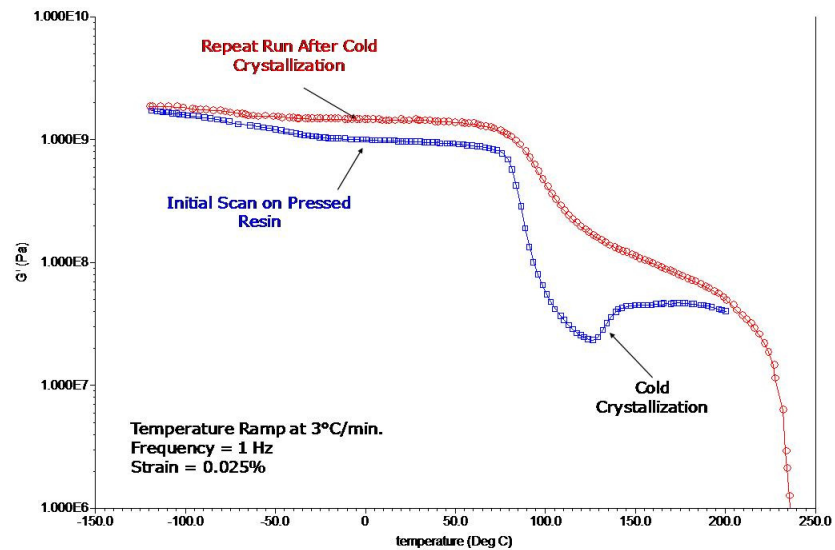
## PET Bottle Resin - Polyethylene terephthalate



Pressed PET  
Bottle Resin

PET After Temperature  
Ramp Scan  
(Cold Crystallization)

## PET Bottle Resin – Before & After Cold Crystallization



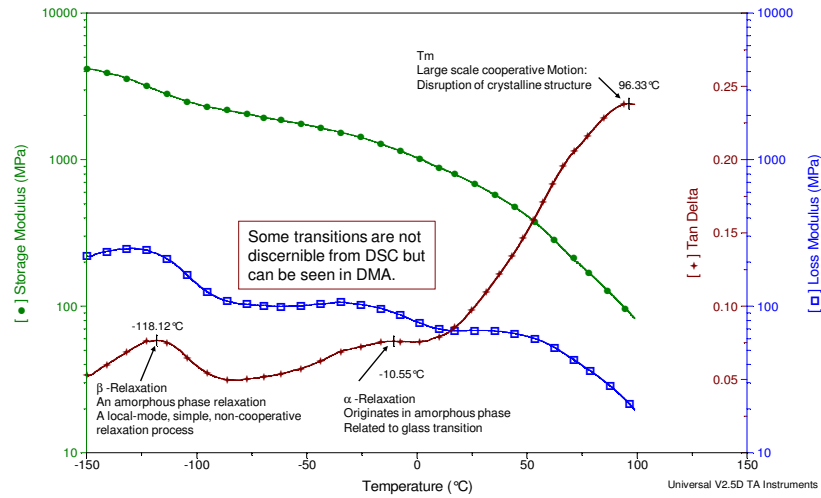
## LDPE: Primary and Secondary Transitions

Sample: Polyethylene in Tension  
Size: 8.4740 x 5.7500 x 1.0000 mm

DMA

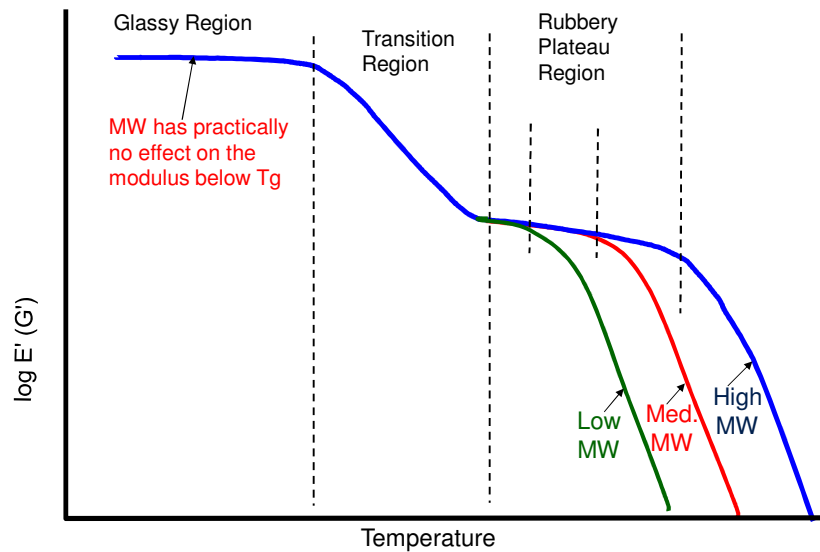
File: F:\DMADATA\Peten.tr1  
Operator: RRU  
Run Date: 18-Jan-99 16:10

Comment: 15 microns, 120% Autostrain, -150 °C to 100 °C



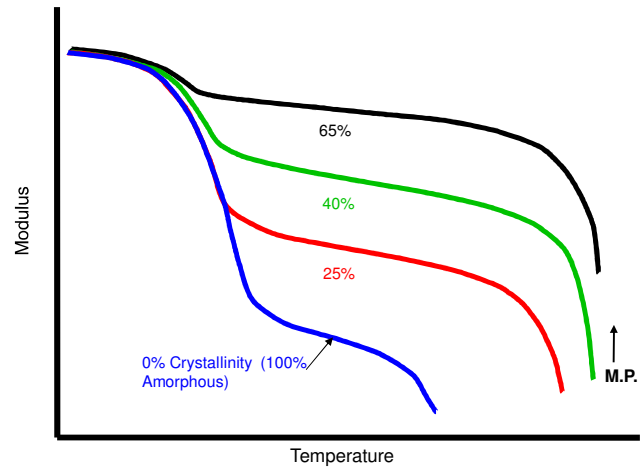
Universal V2.5D TA Instruments  
TA TAINSTRUMENTS.COM

## Molecular Structure - Effect of Molecular Weight

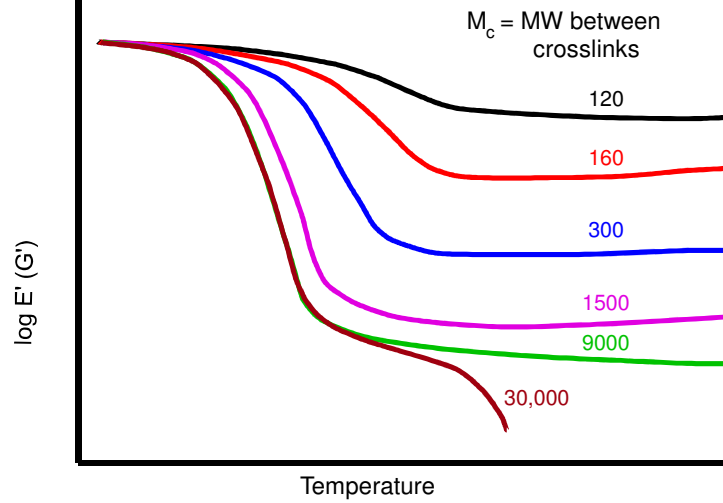


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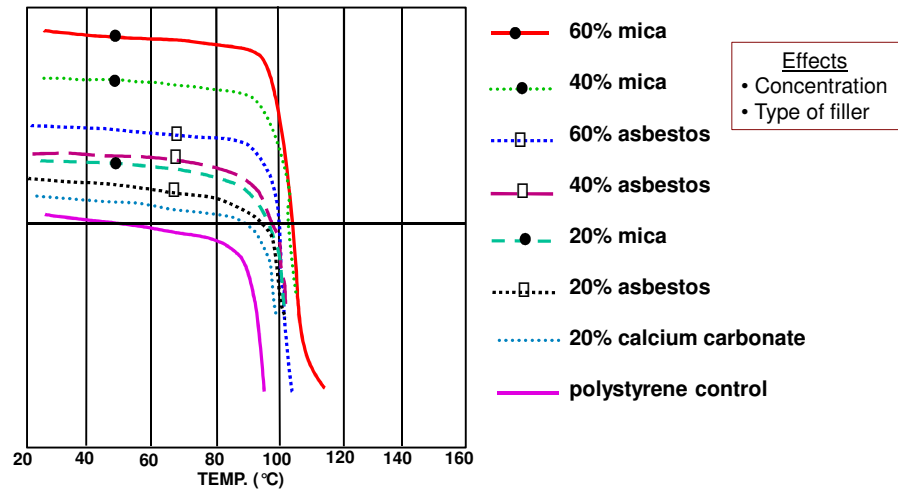
## Effect of Crystallinity



## Effect of Crosslinking



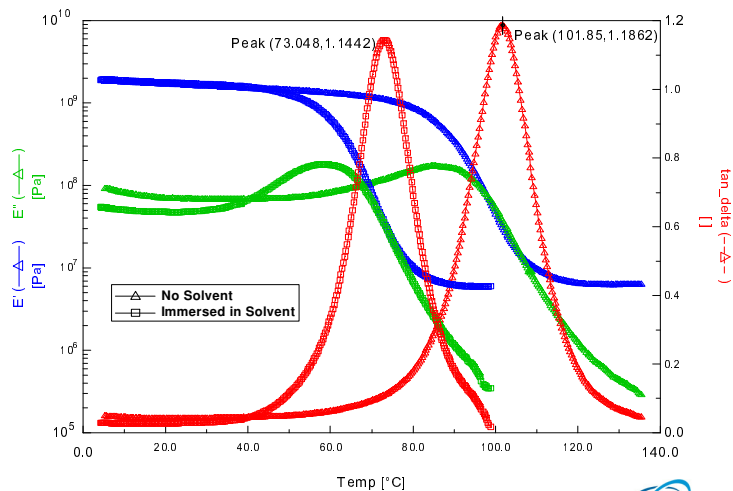
## Effect of Filler on Modulus



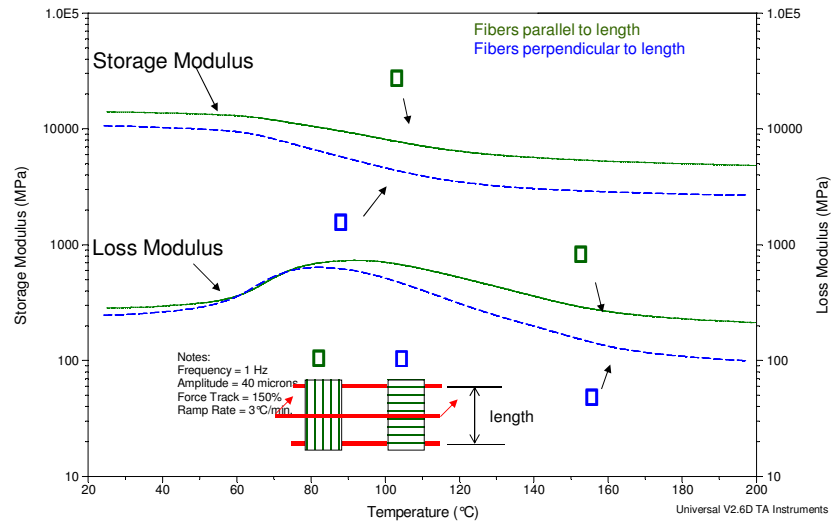
Nielson, L. E., Wall, R. A., and Richmond, P. G., *Soc. Plastics Eng. J.*, **11**, 22 (1966)

## Effect of Moisture

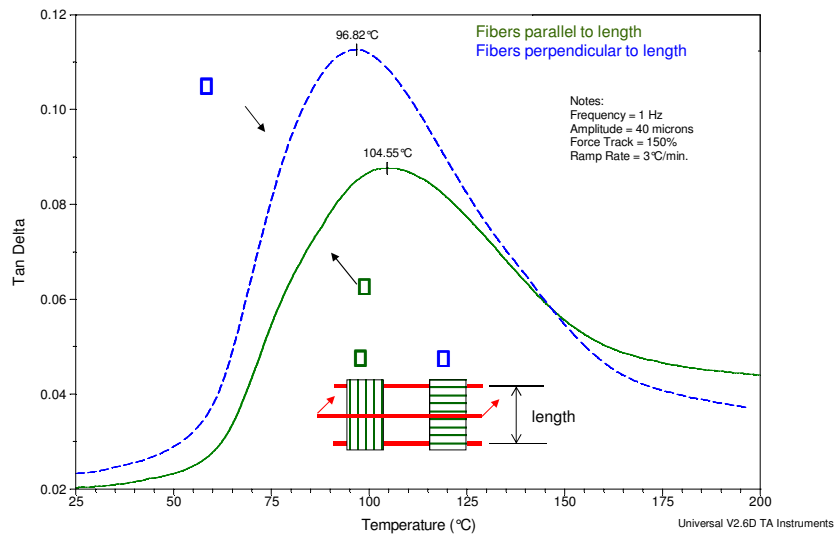
- Automotive coating measured under dry vs. wet conditions



## Anisotropic Material Thermoset Polyester/ Glass Fiber Composite

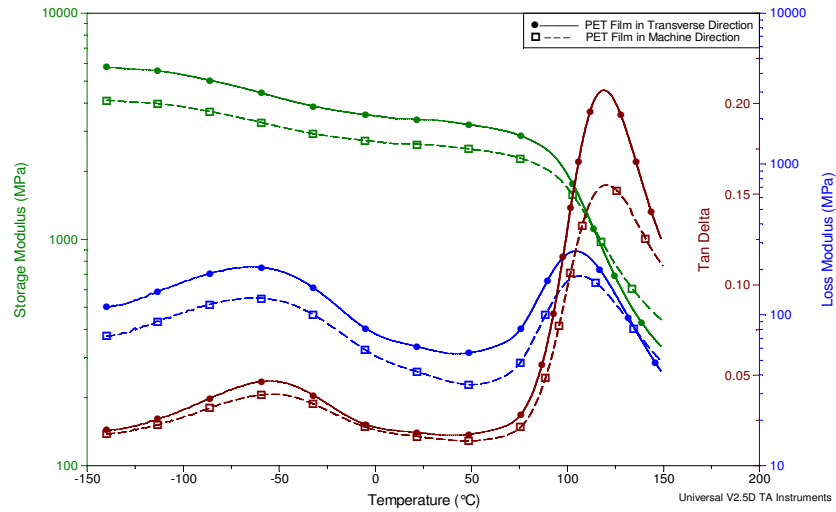


## Anisotropic Material Thermoset Polyester/ Glass Fiber Composite



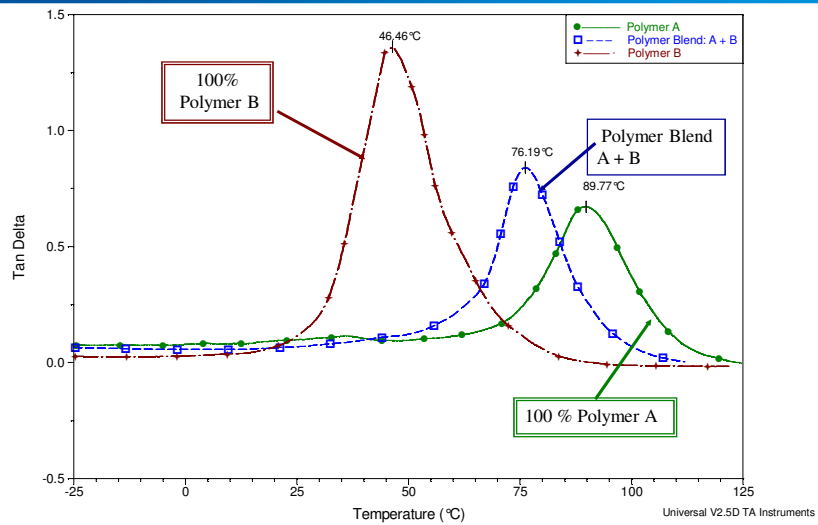


## PET Film in Machine and Transverse Direction



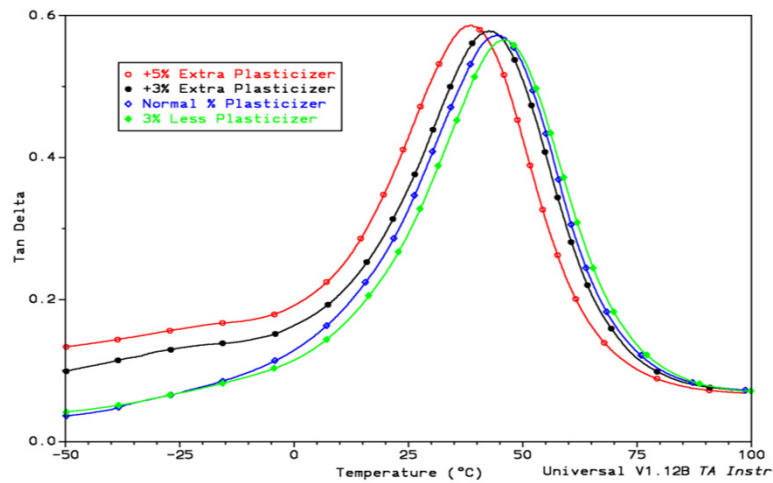
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## Polymer Blend - Aerospace Coating



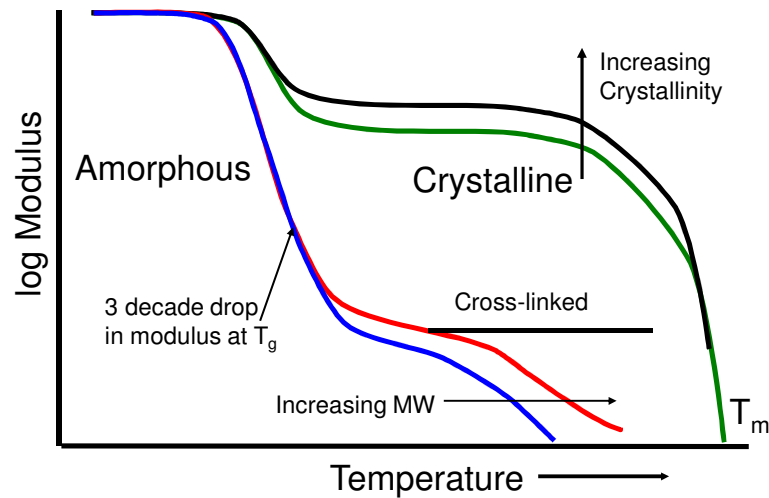
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## Effect of Plasticizer on Vinyl Flooring



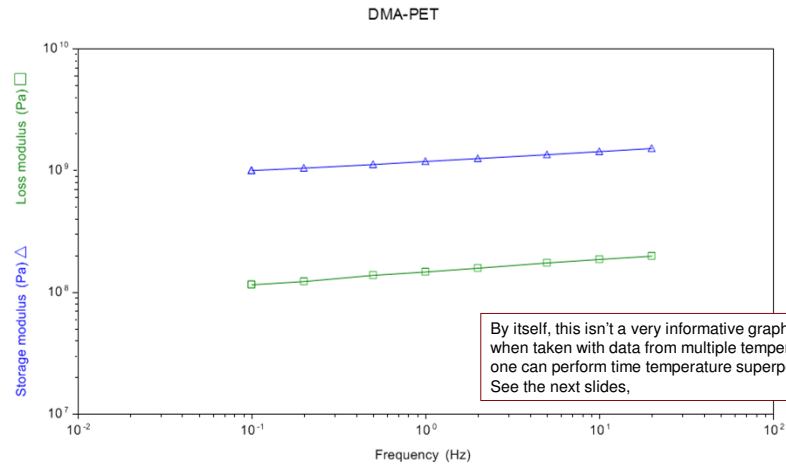
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## Summary of Effects of Crystallinity, Molecular Weight, and Crosslinking

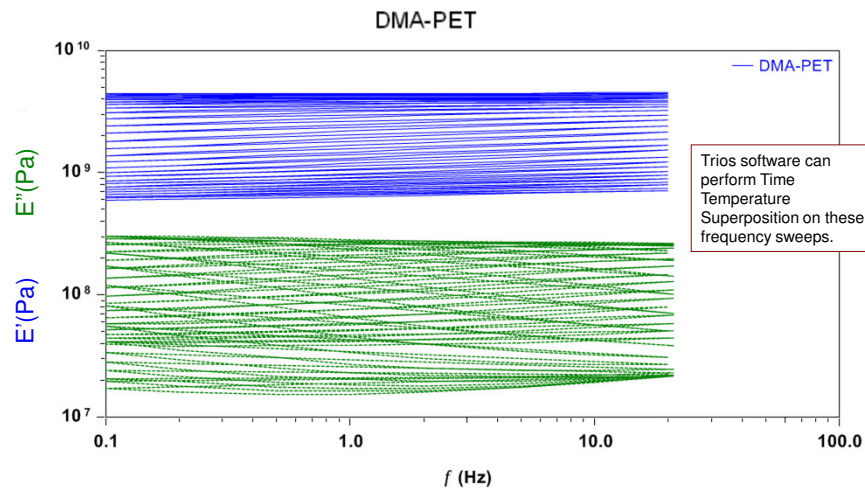


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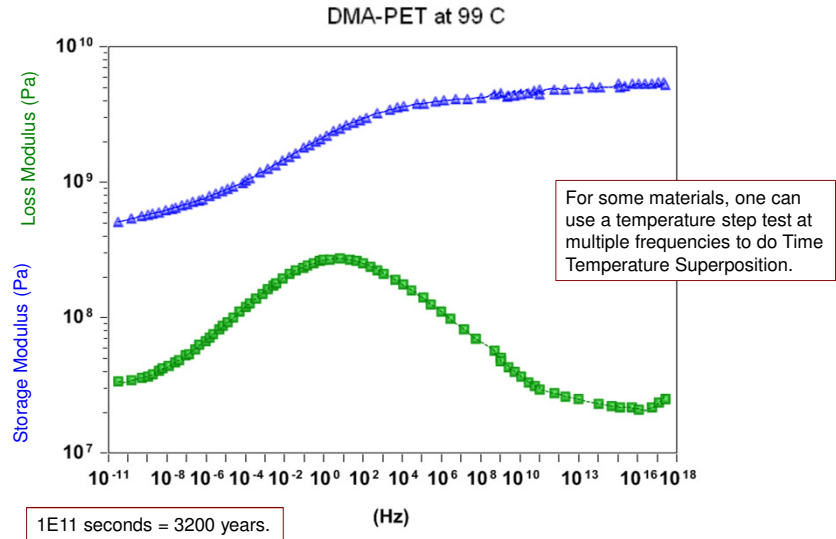
## Dynamic Frequency Sweep



## Temperature/Frequency Sweep

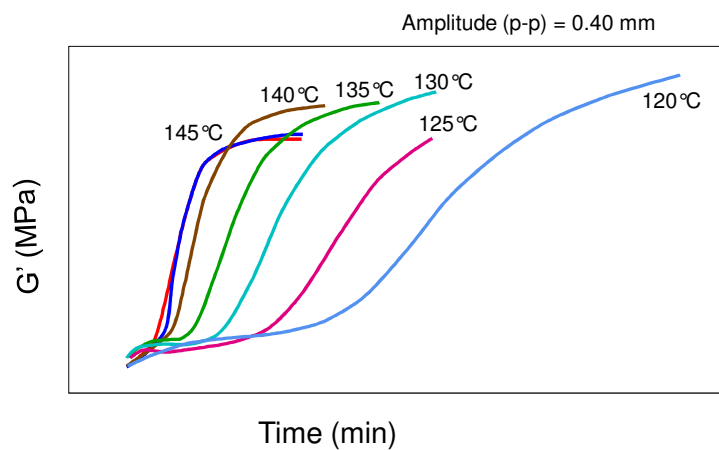


### Frequency/Temperature Sweep – Time Temperature Superposition



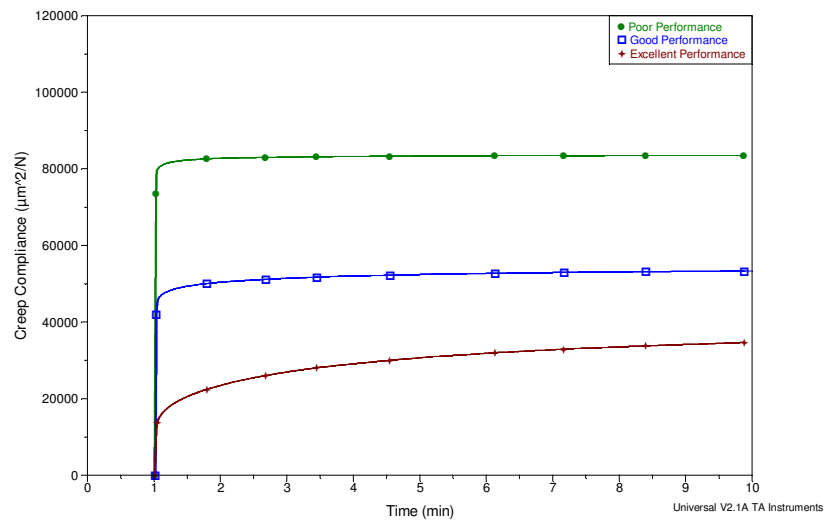
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### Isothermal Cure of Tire Compound: Effect of Curing Temperature



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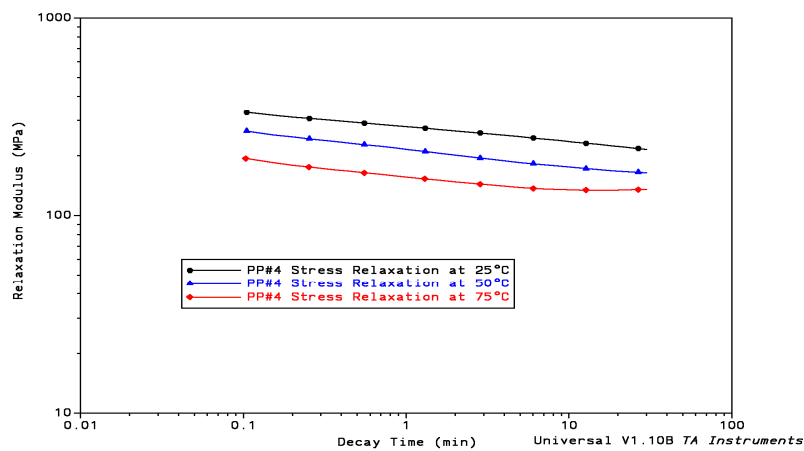
## Creep on Packaging Films used in Thermoforming



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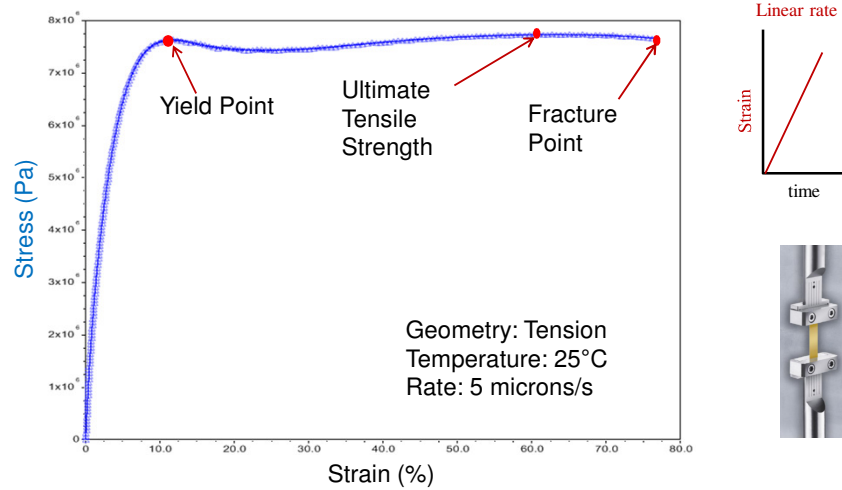
## DMA: Stress Relaxation on Polypropylene

Polypropylene Stress Relaxation on DMA 2980  
Strain = 1%, Clamp: 8 mm Dual Cantilever

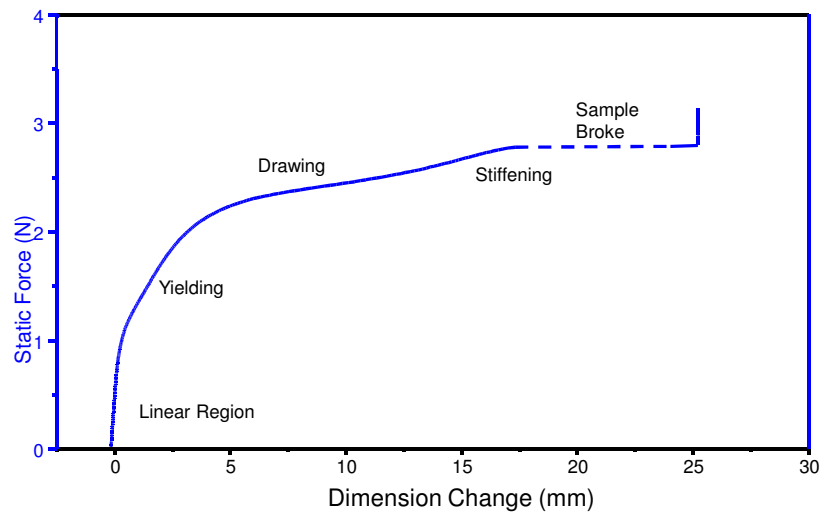


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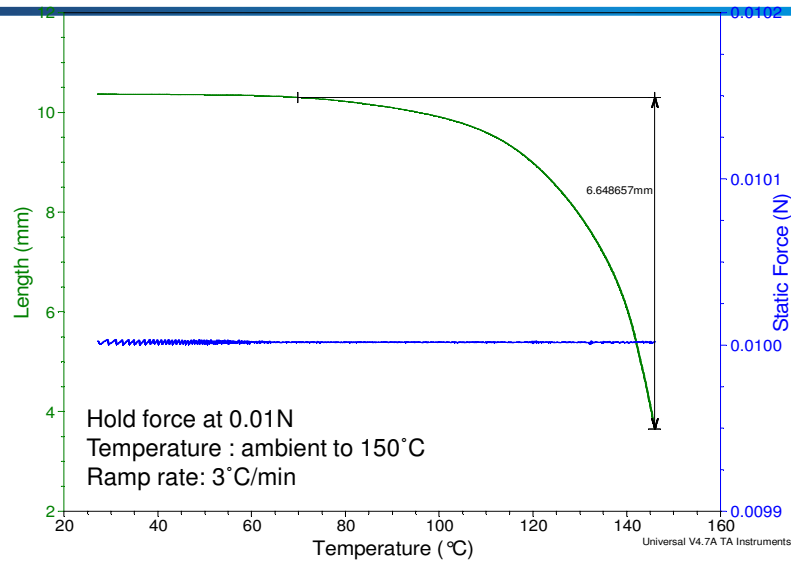
## Polyethylene Bag Linear Strain Ramp



## Stress-Strain Experiment (Ramped Force) Polyethylene Film 4 mm



### Iso-force Temperature Ramp: measure sample shrinkage



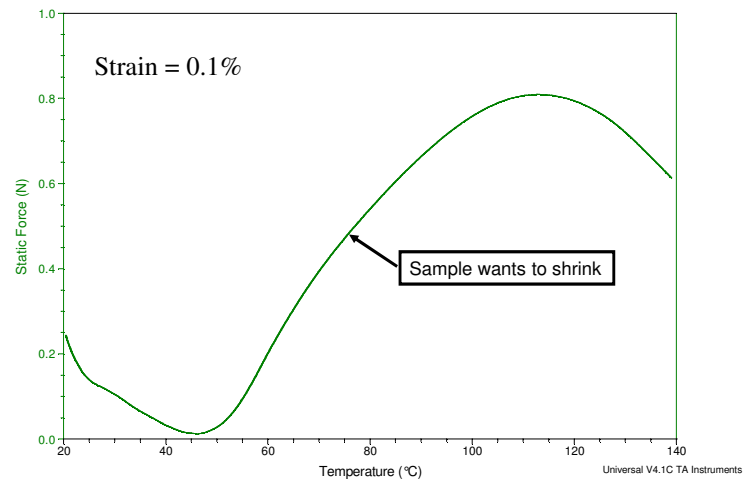
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### PVC Based Film - IsoStrain

Sample: Iso strain  
Size: 13.5810 x 5.3000 x 0.0500 mm  
Method: Isostrain

DMA

File: P:\Q800 ISO STRAIN.002  
Operator: Terri  
Run Date: 2004-09-17 18:11  
Instrument: DMA Q800 V7.0 Build 113



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## DMA-Humidity

- Dynamic Mechanical Analysis performed under controlled conditions of both temperature and relative humidity
- Potential Applications
  - Fuel Cell Components
  - Packaging Films
  - Pharmaceutical Components
  - Food Materials
  - Electronics

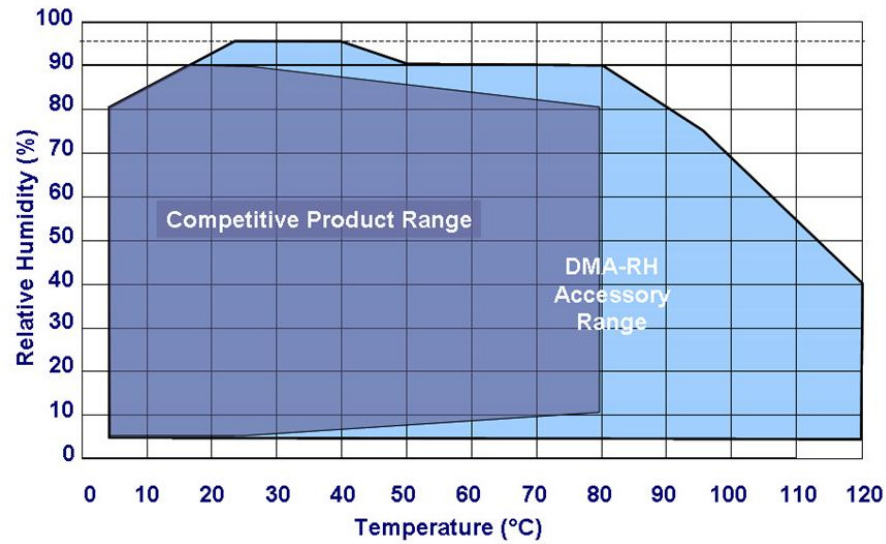


## DMA850 and Q800: Humidity Option





## Q800: DMA-RH Operating Range



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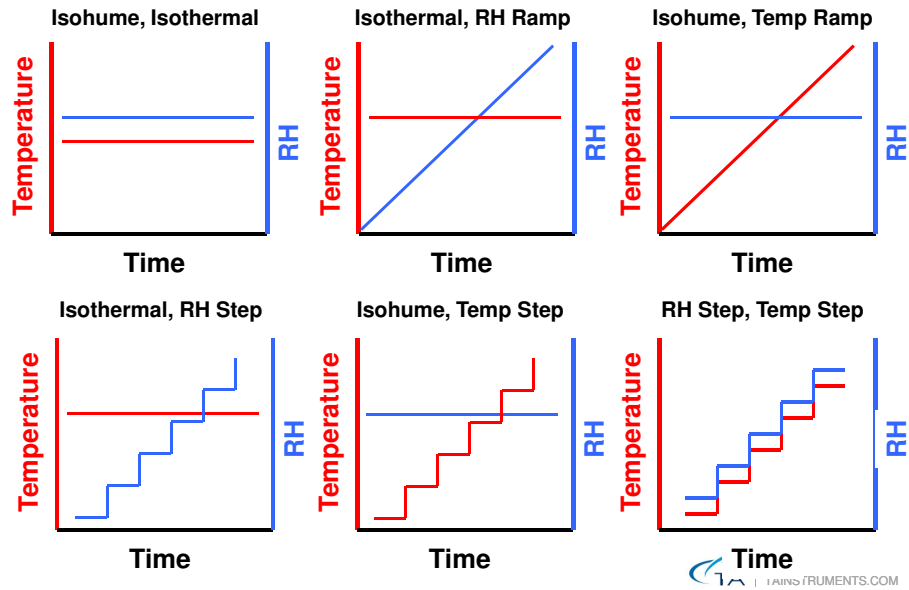
## DMA-RH Performance Specifications

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

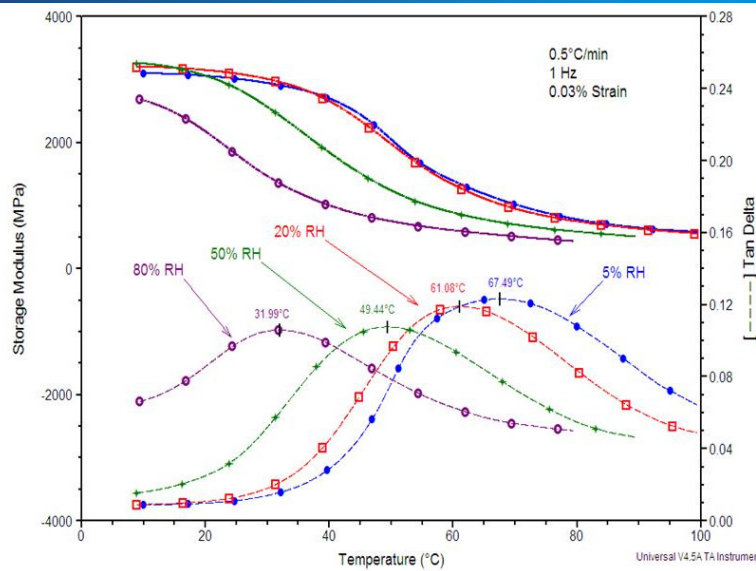
\*Alternative pseudo-linear RH ramp rates can be achieved through Step-Iso control

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## DMA-RH Experimental Options



## Analysis of Nylon 6: Isohume-Temperature Scans

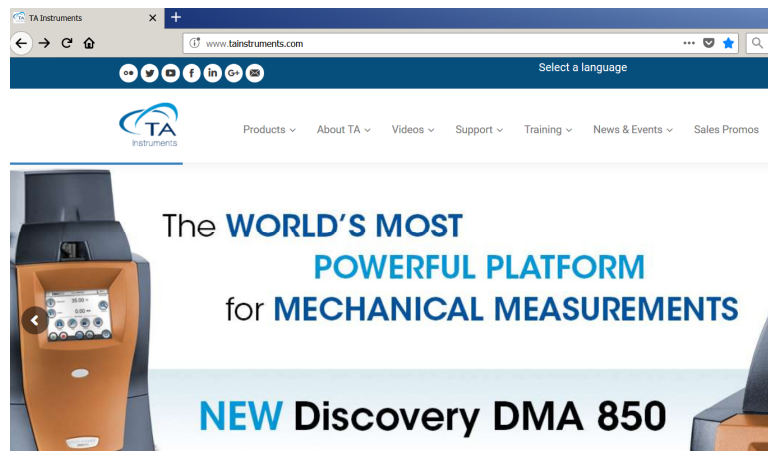


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- Instrument Manuals
- Help Feature in Trios
- TA Instruments website
- TA Instruments Rheology Helpline
  - [rsupport@tainstruments.com](mailto:rsupport@tainstruments.com)



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We made it!

Remember – Rheology is FUN.