RHEOLOGY AND DYNAMIC MECHANICAL ANALYSIS – What They Are and Why They're Important

Presented for

University of Wisconsin - Madison

by

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TA Instruments

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Rheology: An Introduction



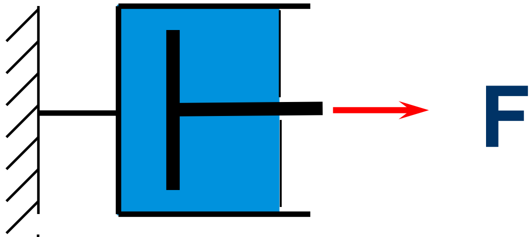


Rheology: The study of the flow and deformation of matter.
Rheological behavior affects every aspect of our lives.
Dynamic Mechanical Analysis is a subset of Rheology



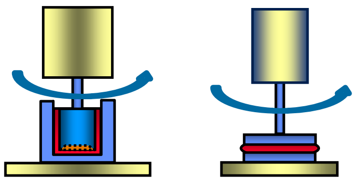


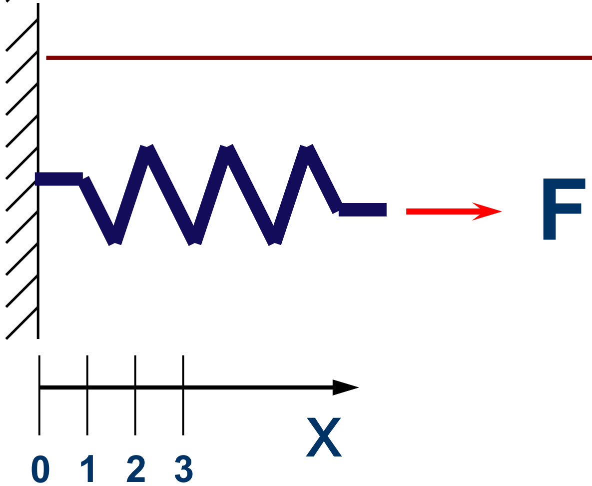
Rheology: The study of the flow and deformation of matter



Flow: Fluid Behavior; Viscous Nature

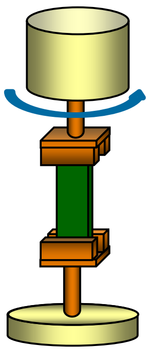
$F = F(v); F \neq F(x)$

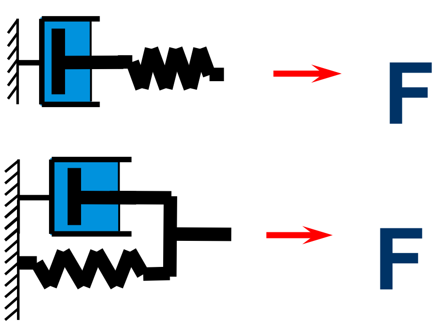




Deformation: Solid Behavior
Elastic Nature

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





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

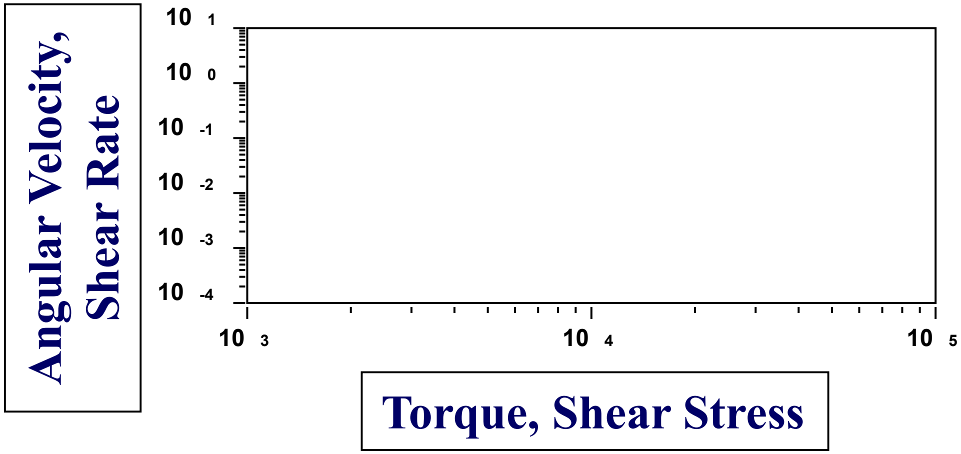
1. ROTATIONAL RHEOLOGY

2. DYNAMIC MECHANICAL ANALYSIS (LINEAR TESTING)

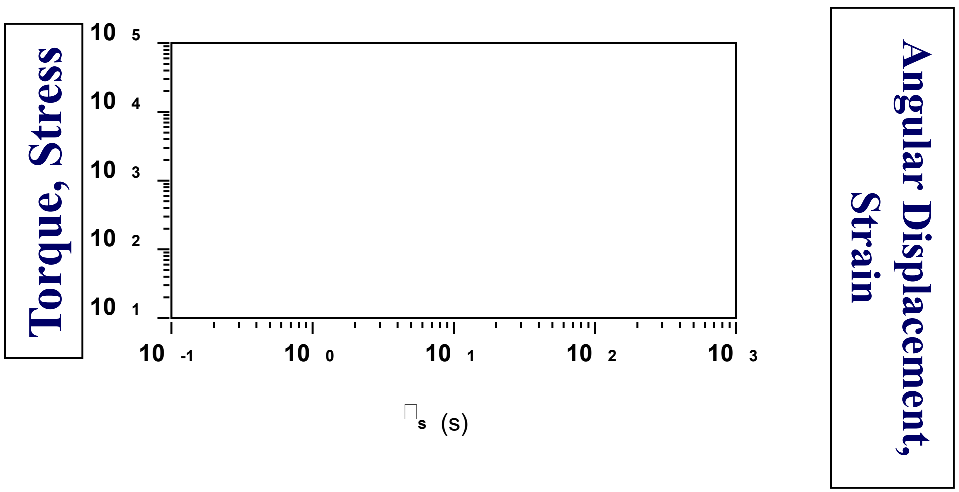
Rheological Testing – Rotational - Unidirectional

- **2 Basic Rheological Methods**

1. **Apply Force (Torque)and measure Deformation and/or Deformation Rate (Angular Displacement, Angular Velocity) - Controlled Force, Controlled Stress**

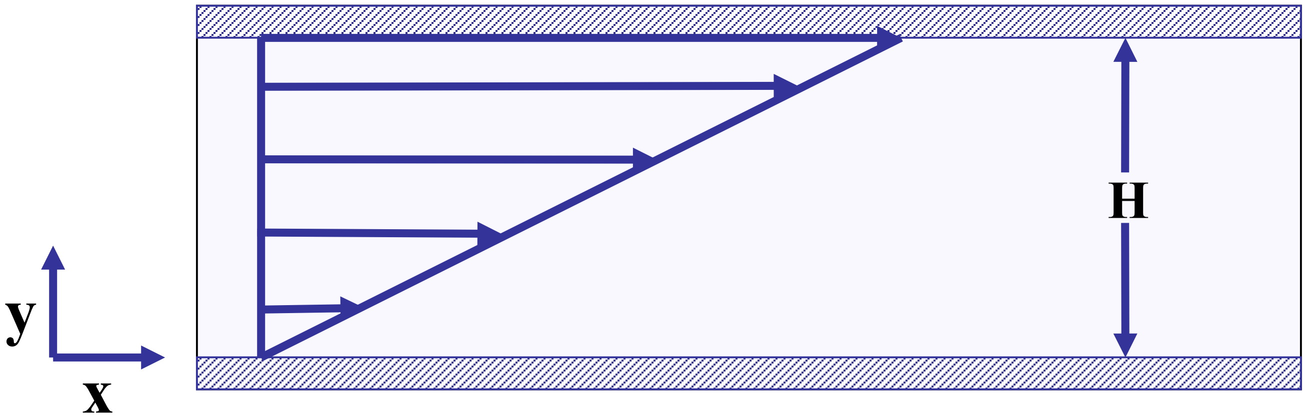


2. **Control Deformation and/or Deformation Rate and measure Force needed (Controlled Displacement or Rotation, Controlled Strain or Shear Rate)**



Steady Simple Shear Flow

Top Plate Velocity = V_0; Area = A; Force = F



Bottom Plate Velocity = 0

$v_x = (y/H)*V_0$

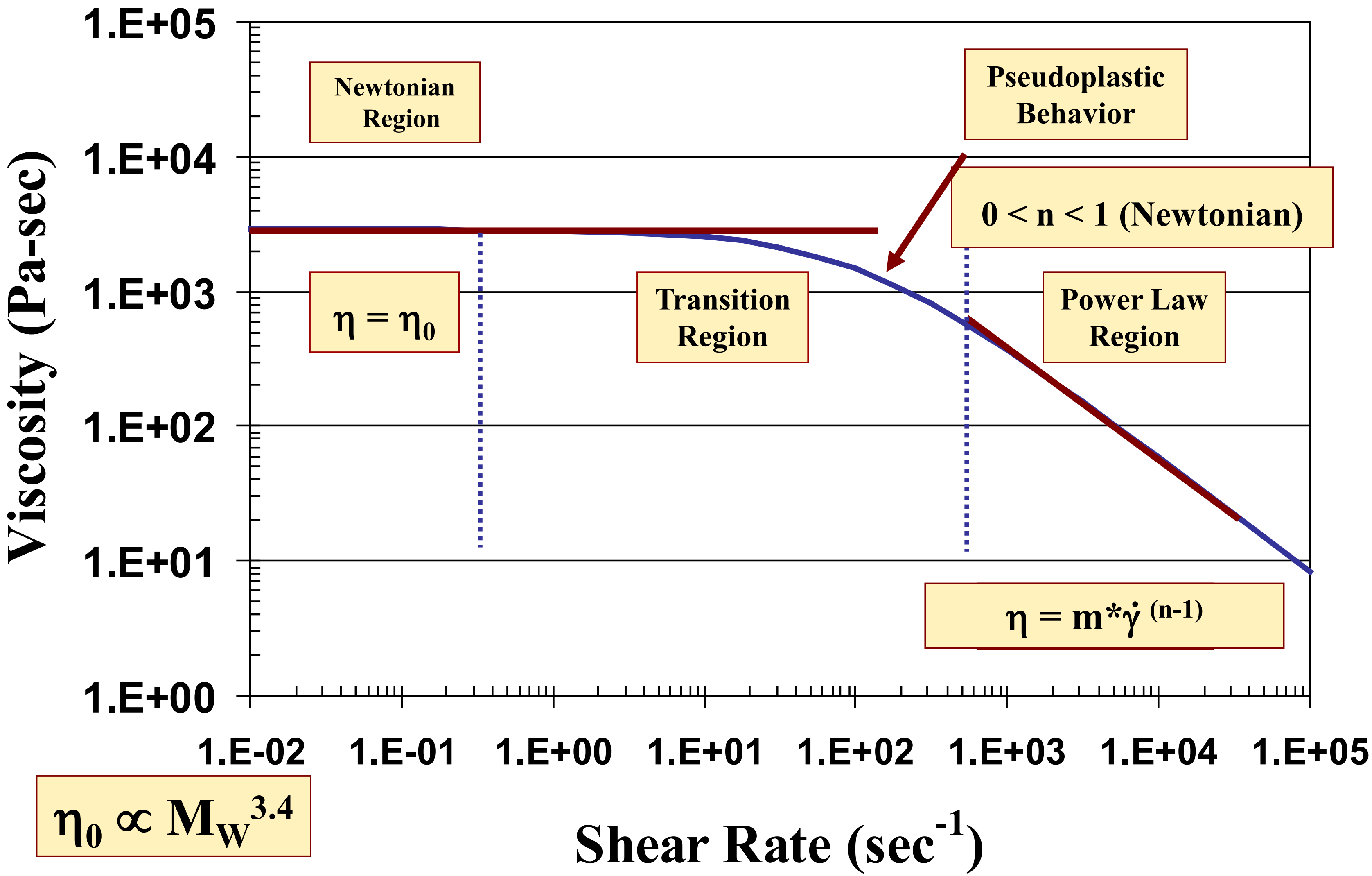
$\dot{\gamma} = dv_x/dy = V_0/H$ — Shear Rate, sec^{-1}

$\sigma = F/A$ — Shear Stress, Pascals

$\eta = \sigma/\dot{\gamma}$ — Viscosity, Pa-sec

- These are the fundamental flow parameters. Shear rate is always a change in velocity with respect to distance.

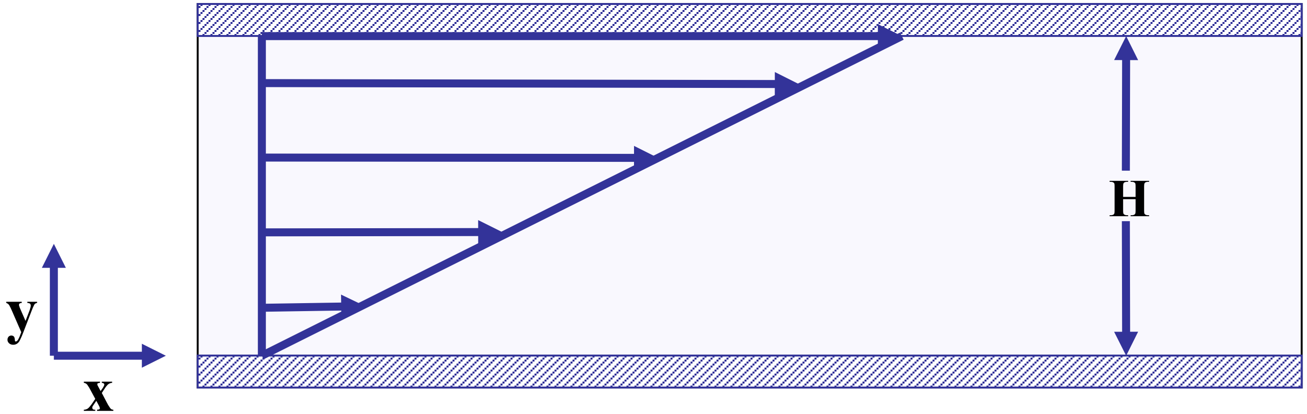
Representative Flow Curve



$\eta_0 \propto M_W^{3.4}$

Simple Shear Deformation

Top Plate Displacement = X_0; Area = A; Force = F



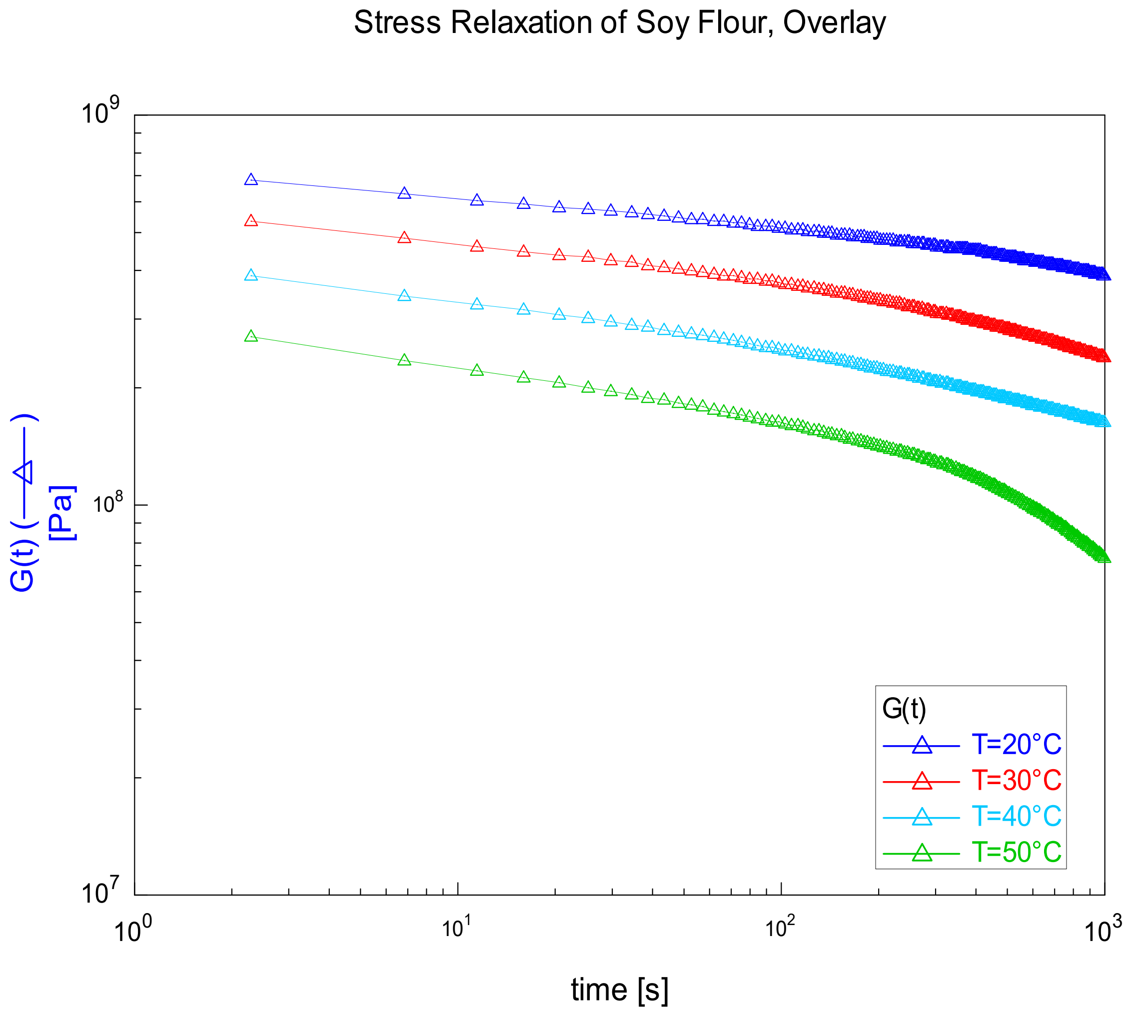
Bottom Plate Displacement = 0

$$x = (y/H)*X_0$$

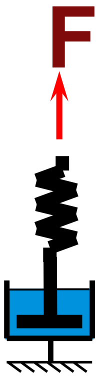
$\gamma = dx_x/dy = X_0/H$	**Shear Strain, unitless**
$\sigma = F/A$	**Shear Stress, Pascals**
$G = \sigma/\gamma$	**Modulus, Pa**

➢ **These are the fundamental deformation parameters. Shear strain is always a change in displacement with respect to distance.**

Stress Relaxation

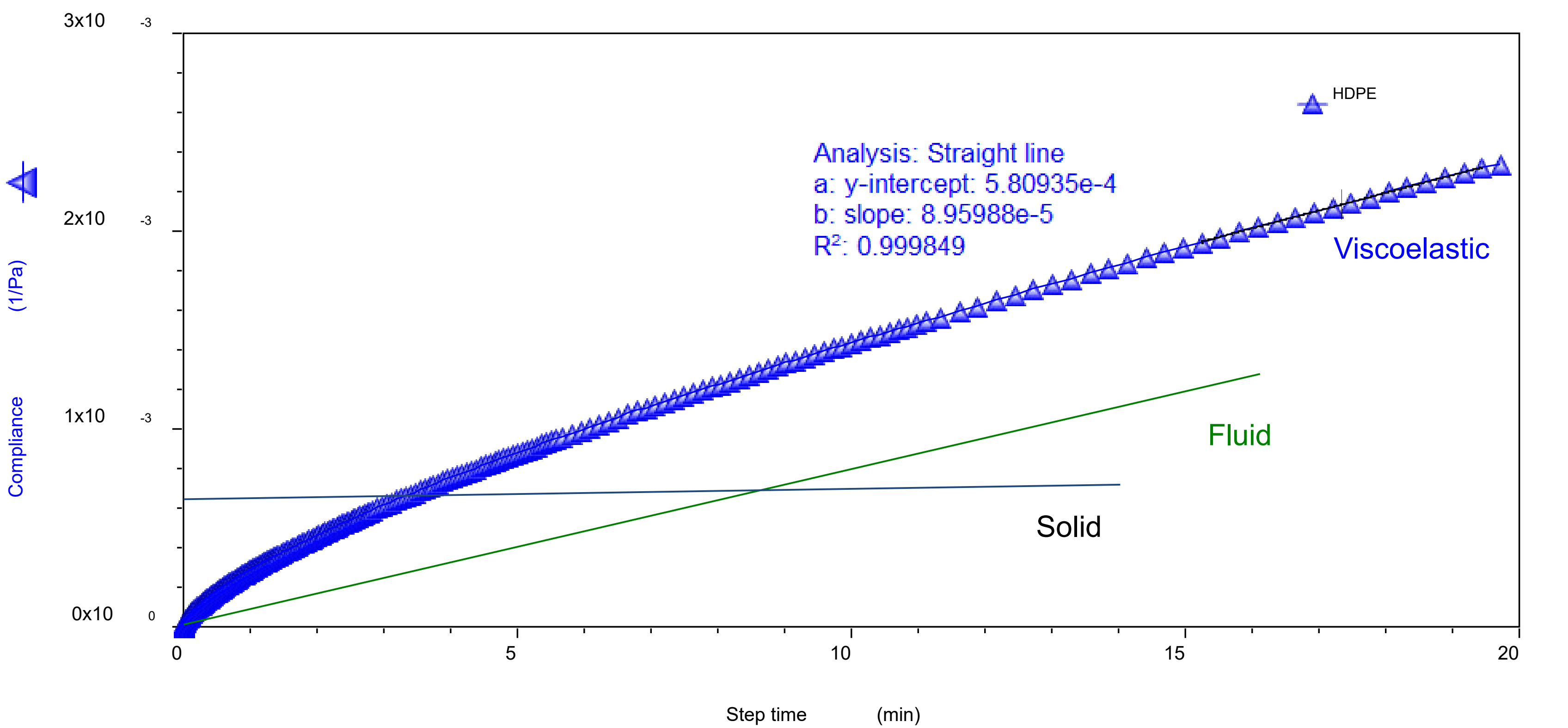


- **Instantaneous Strain**
- **Note the decrease in the modulus as a function of time.**



Creep Testing

Stress is held constant.
Strain is the measured variable.



Geometry Options

Concentric Cylinders

Very Low
to Medium
Viscosity

Cone and Plate

Very Low
to High
Viscosity

Parallel Plate

Very Low
Viscosity
to Soft Solids

Torsion Rectangular

Solids

Water → to → Steel

Examples for Common Configurations

Geometry	Examples
Concentric Cylinder	Coatings Beverages Slurries (vane rotor option) Starch pasting
Cone and Plate	Low viscosity fluids Viscosity standards Sparse materials Polymer melts in steady shear
Parallel Plate	Widest range of materials Adhesives Polymer melts Hydrogels Asphalt Curing of thermosetting materials Foods Cosmetics
Torsion Rectangular	Thermoplastic solids Thermoset solids

Rheology – Speak

Machine Parameter	Rheological Parameter
Angular Velocity (rad/sec)	Shear Rate (1/sec)
Angular Displacement (rad)	Shear Strain (-)
Torque (μN-m)	Shear Stress (N/m^2, Pa)

Converting Machine to Rheological Parameters in Rotational Rheometry

$$\frac{M \, x \, K_\sigma}{\Omega \, x \, K_\gamma} = \frac{\sigma}{\dot{\gamma}} = \eta$$

$$\frac{M x K_\sigma}{\theta x K_\gamma} = \frac{\sigma}{\gamma} = G$$

Machine Parameters

M: Torque

Ω : Angular Velocity

θ : Angular Displacement

Conversion Factors

K_σ : Stress Conversion Factor

K_γ : Strain (Rate) Conversion Factor

Rheological Parameters

σ : Shear Stress (Pa)

$\dot{\gamma}$: Shear Rate (sec^{-1})

η : Viscosity (Pa-sec)

γ : Shear Strain

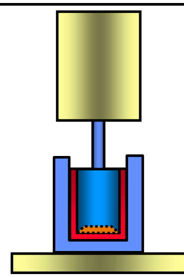
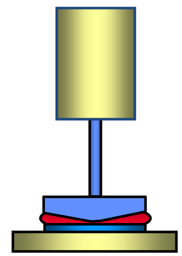
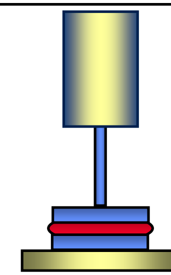
G : Shear Modulus (Pa)

The conversion factors, K_σ and K_γ, will depend on the following:

Geometry of the system – concentric cylinder, cone and plate, parallel plate, and torsion rectangular

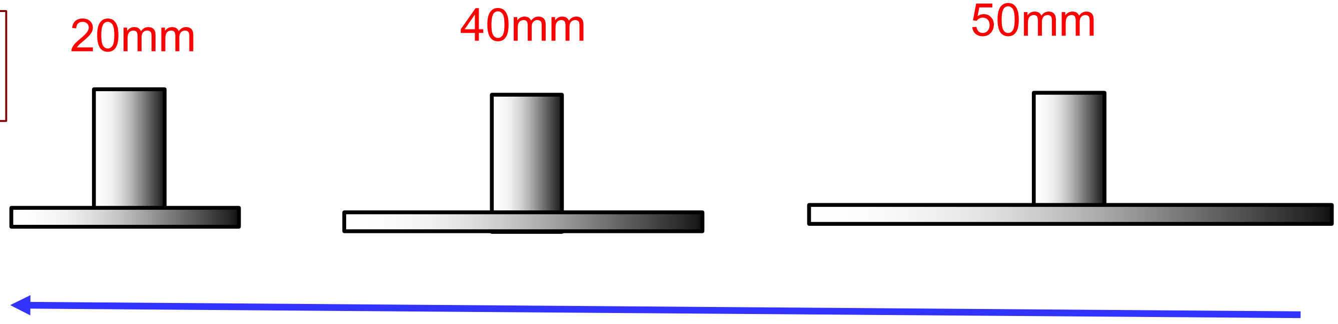
Dimensions – gap, cone angle, diameter, thickness, etc.

Shear Rate and Shear Stress Calculations

Conversion Factor	Geometry		
	Couette	Cone & Plate	Parallel Plates
			
K_γ	$R_{avg}/(R_o-R_i)$	$1/\beta$	R/h
K_σ	$1/(2*\pi R_i^2 L)$	$3/(2\pi R^3)$	$2/(\pi R^3)$

Cone & Plate and Parallel Plate Geometric Considerations

Standard DHR Peltier plate geometry diameters



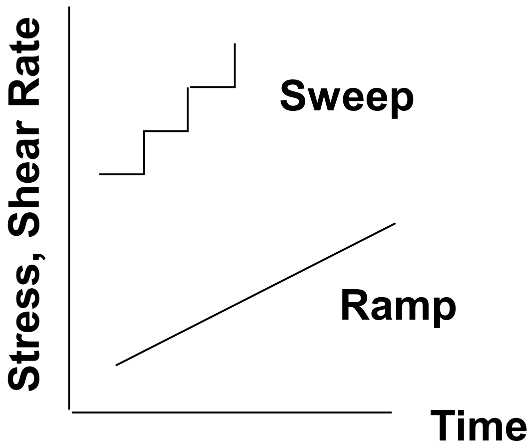
Shear Stress
At given torque,
Increase in diameter → Decrease in shear stress

Shear Rate
At given angular velocity
Increase in cone angle → Decrease in shear rate
Increase in gap (parallel plate) → Decrease in shear rate

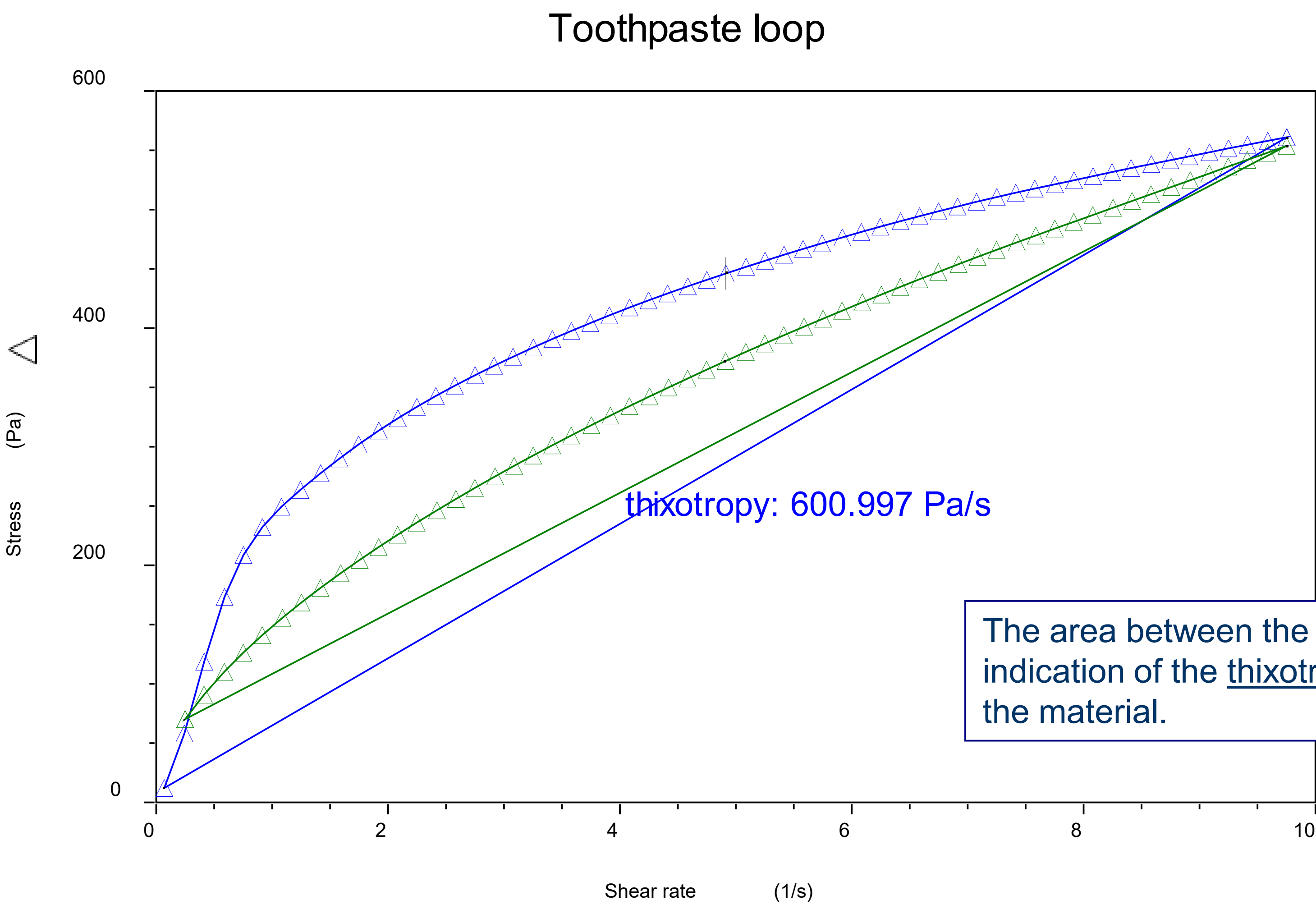
So, for low viscosity fluids, use the largest diameter cone or plate.
For high viscosity fluids, use the smallest diameter cone or plate

Rheological Methods – Unidirectional Testing

- Flow
  - Stress/Rate Ramp
  - Stress/Rate Sweep
  - Time sweep/Peak Hold
  - Temperature Ramp
- Creep (constant stress)
- Stress Relaxation (constant strain)
- Axial Testing

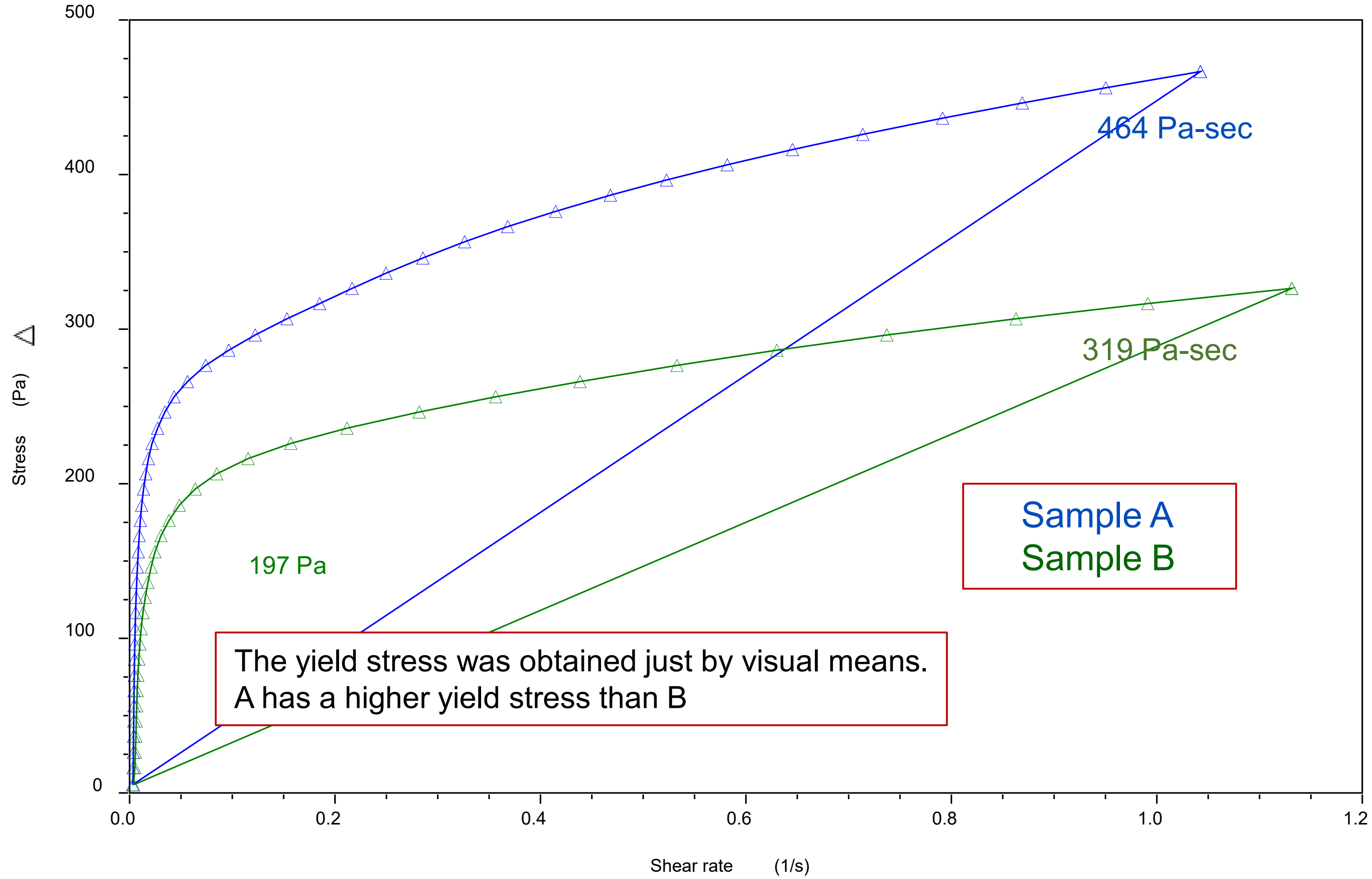


Thixotropy: Up & Down Flow Curves

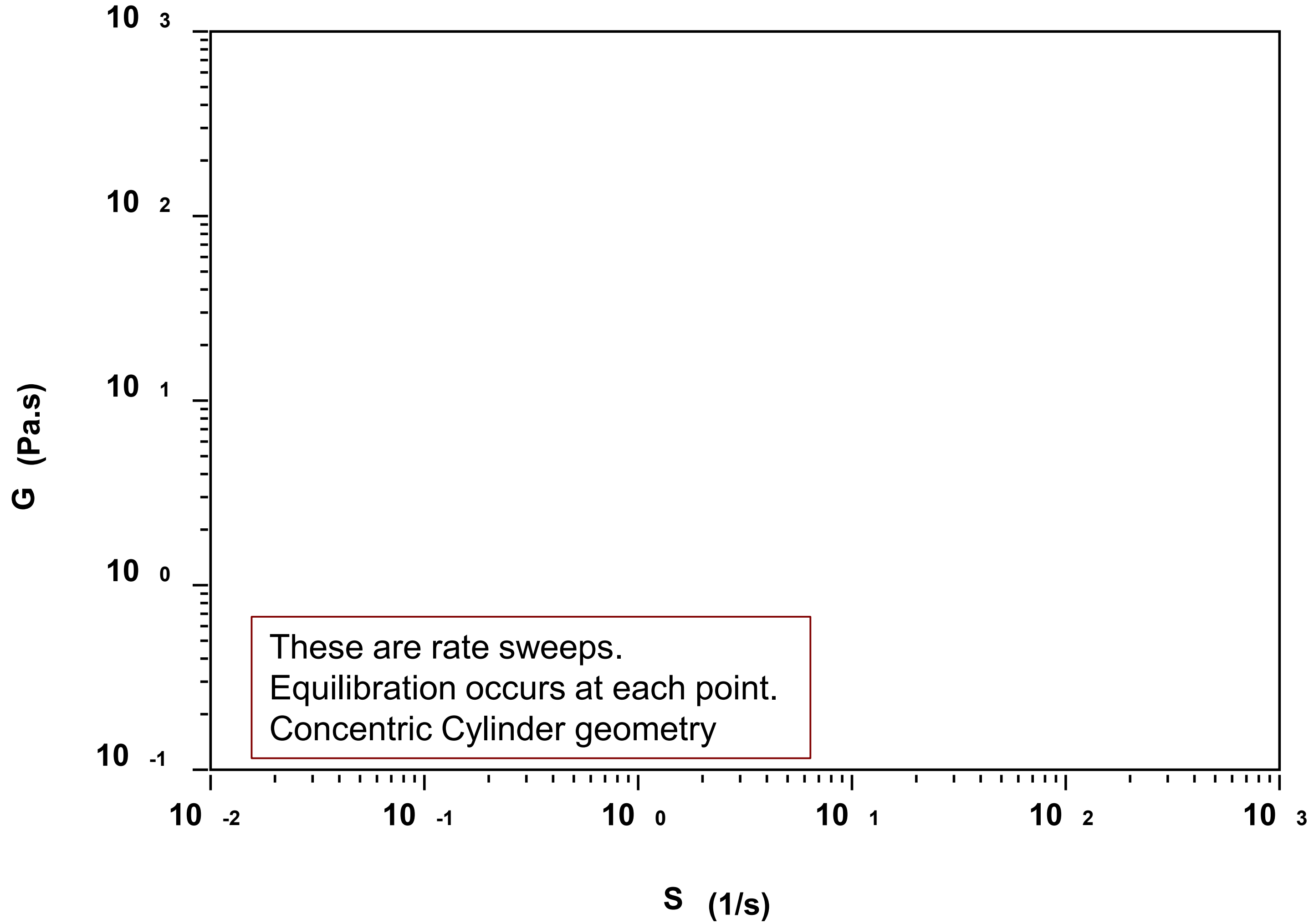


The area between the curves is an indication of the thixotropic nature of the material.

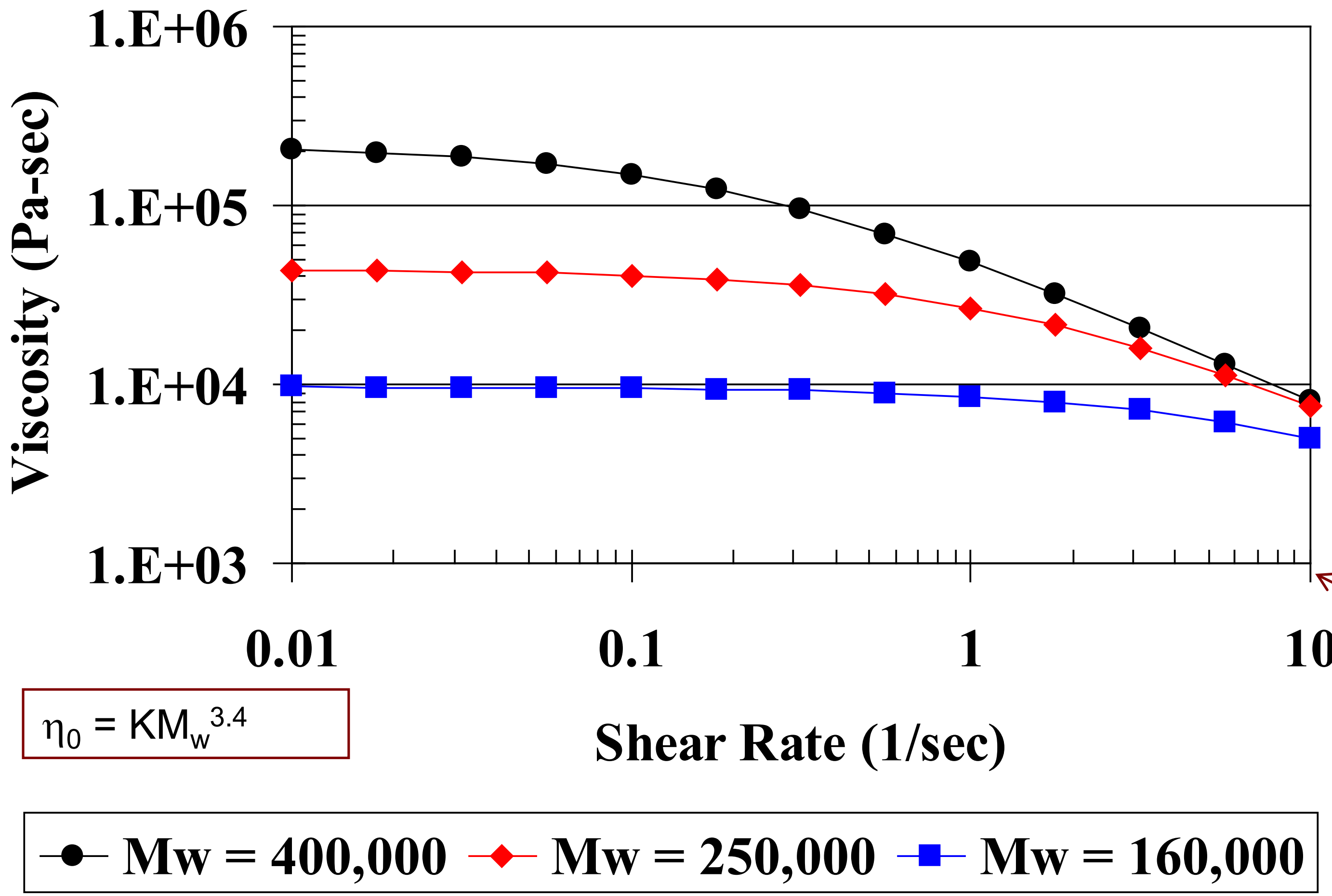
Flow Stress Ramp on a Yielding Material



Steady State Flow at 25 C - Coatings



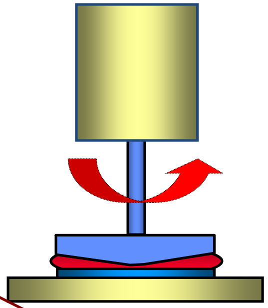
Polymer Melt Flow Curve



Williamson Model
$\eta = \eta_0/(1+(\sigma\dot{\gamma})^c)$

$\eta_0 = KM_w^{3.4}$

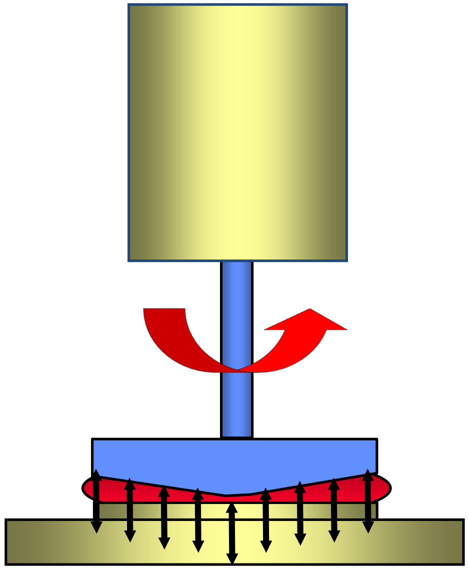
Cone and Plate



Steady testing with cone and plate and parallel plate geometries is often limited to low shear rates.

The shear rate and shear stress are constant throughout the gap with the cone-and-plate geometry.
Parallel plate data can be corrected with the Rabinowitsch correction.

Normal Force Measurements with Cone & Plate

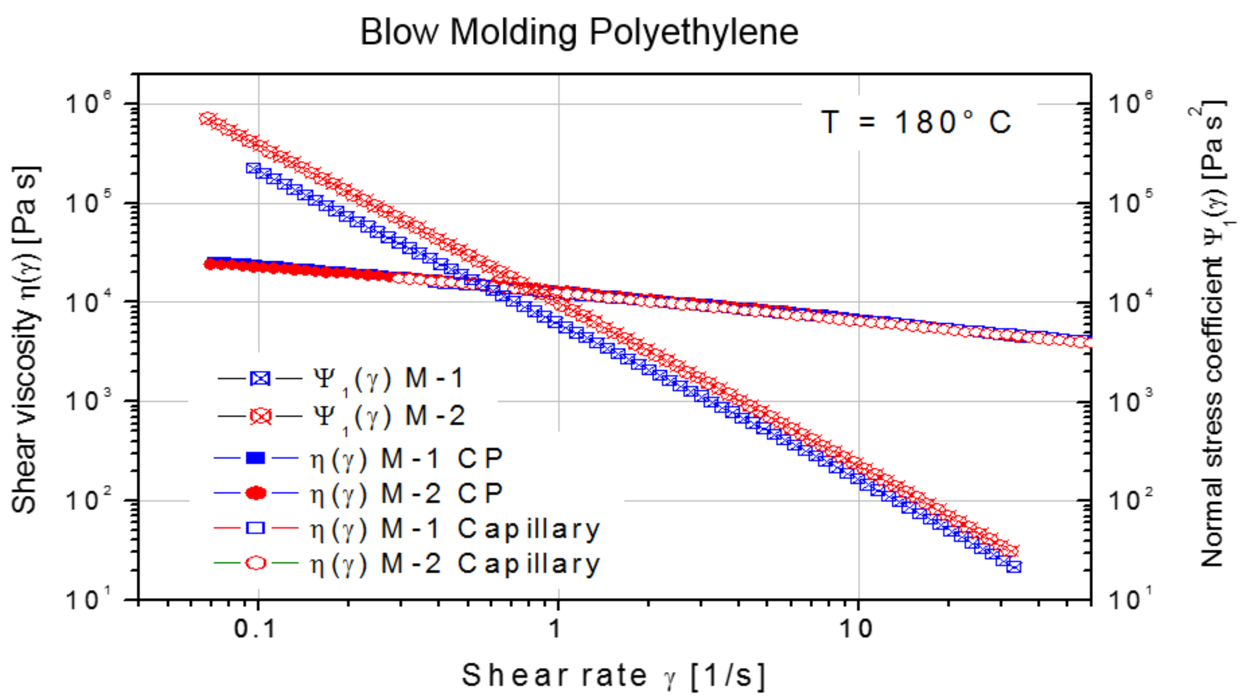


Normal Stress Difference:

- In steady flow, polymeric materials can exert a force that tries to separate the cone and the plate.
- A parameter to measure this is the Normal Stress Difference, N1, which equals

  $\sigma_{xx} - \sigma_{yy}$ from the Stress Tensor.
- $N1 = 2F/(\pi R^2)$, where F is the measured force.
- $\Psi_1 = N1/$?^{2} This is the primary normal stress coefficient.

Effect of HDPE Variations in Blow Molding

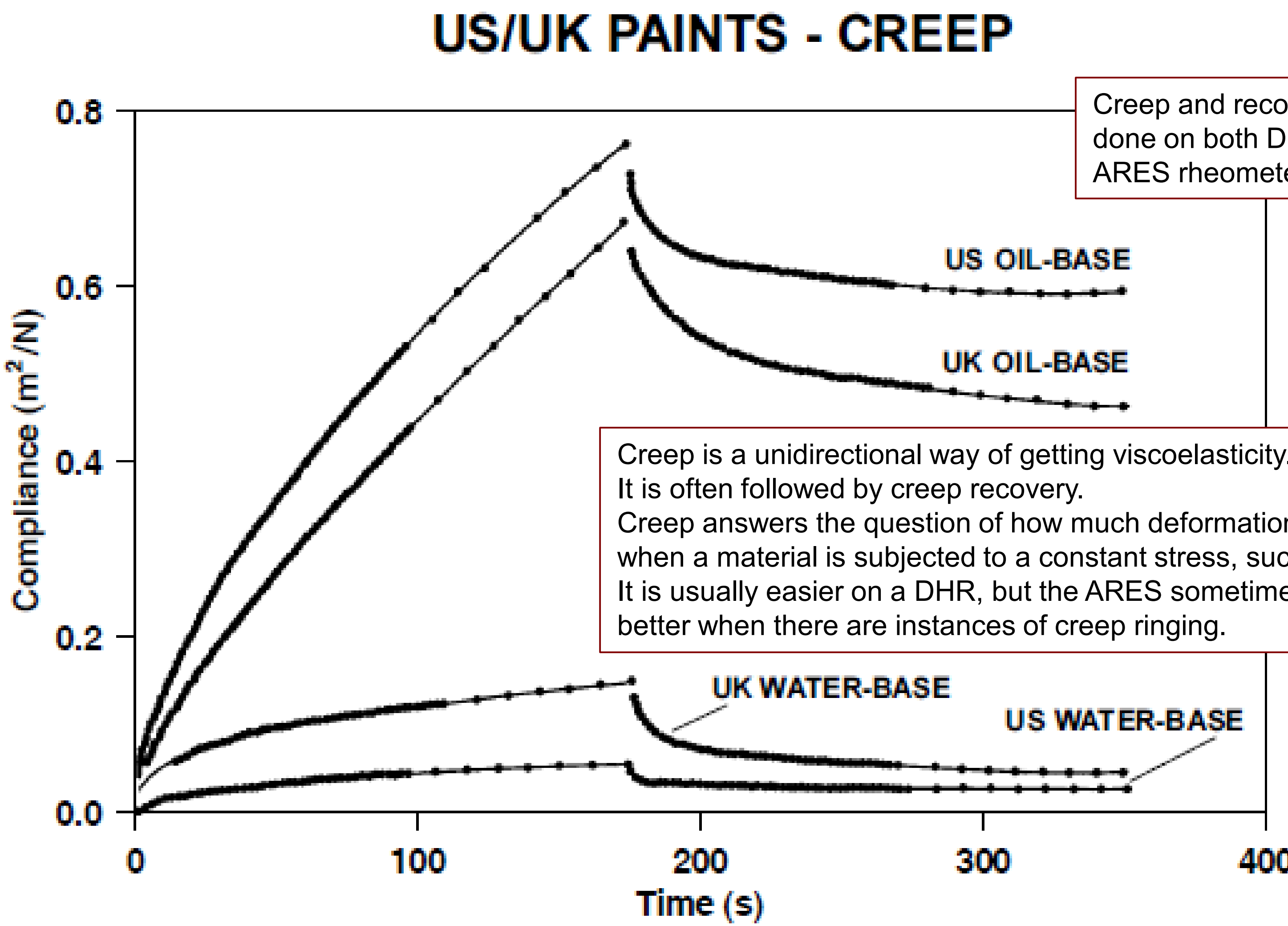


Parameter	Sample	
	M-1	M-2
MFI	0.6	0.5
GPC-M_W	131K	133K
Viscosity at 1 sec^{-1}	8.4K	8.3K
Die Swell	28	42

No differences in MFI, Viscosity, or GPC!

M-2 produces heavier bottles in blow molding due to increased parison swell

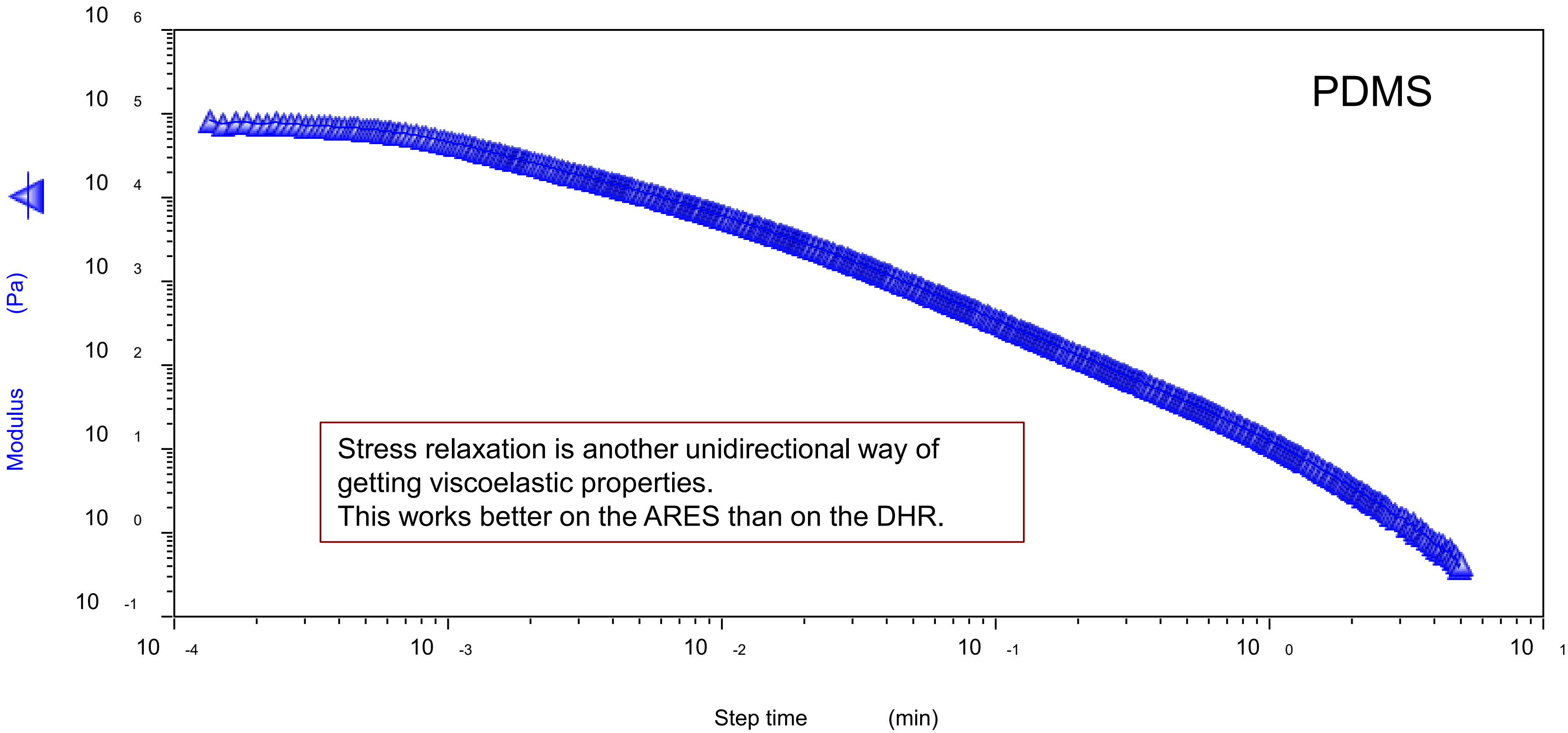
Creep Testing on US and UK Paints



Creep and recovery can be done on both DHR and ARES rheometers

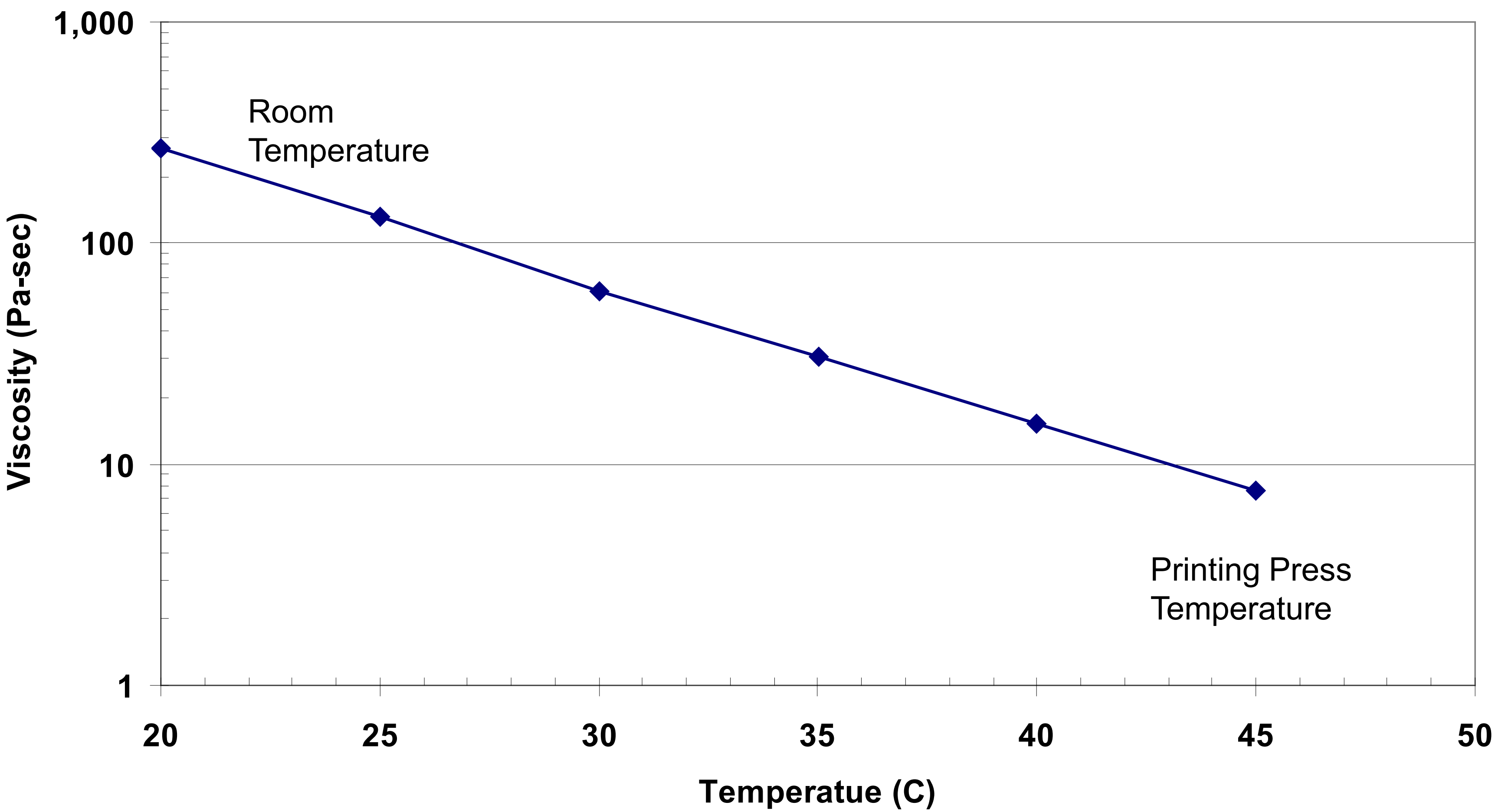
Creep is a unidirectional way of getting viscoelasticity.
It is often followed by creep recovery.
Creep answers the question of how much deformation to expect when a material is subjected to a constant stress, such as gravity
It is usually easier on a DHR, but the ARES sometimes works better when there are instances of creep ringing.

Stress Relaxation

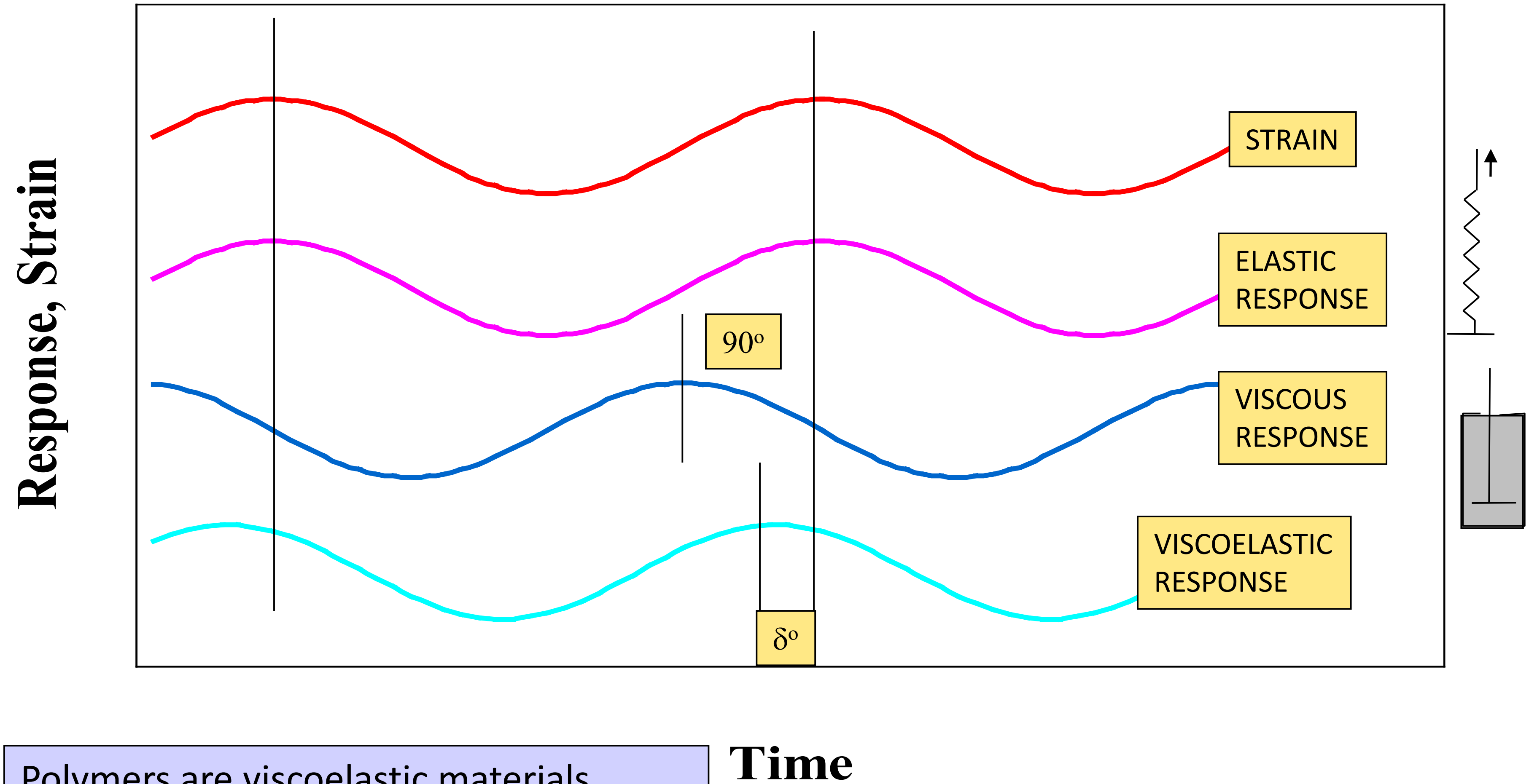


Stress relaxation is another unidirectional way of getting viscoelastic properties.
This works better on the ARES than on the DHR.

Flow Temperature Ramp – Printing Inks



Dynamic Testing



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

Dynamic Rheological Parameters

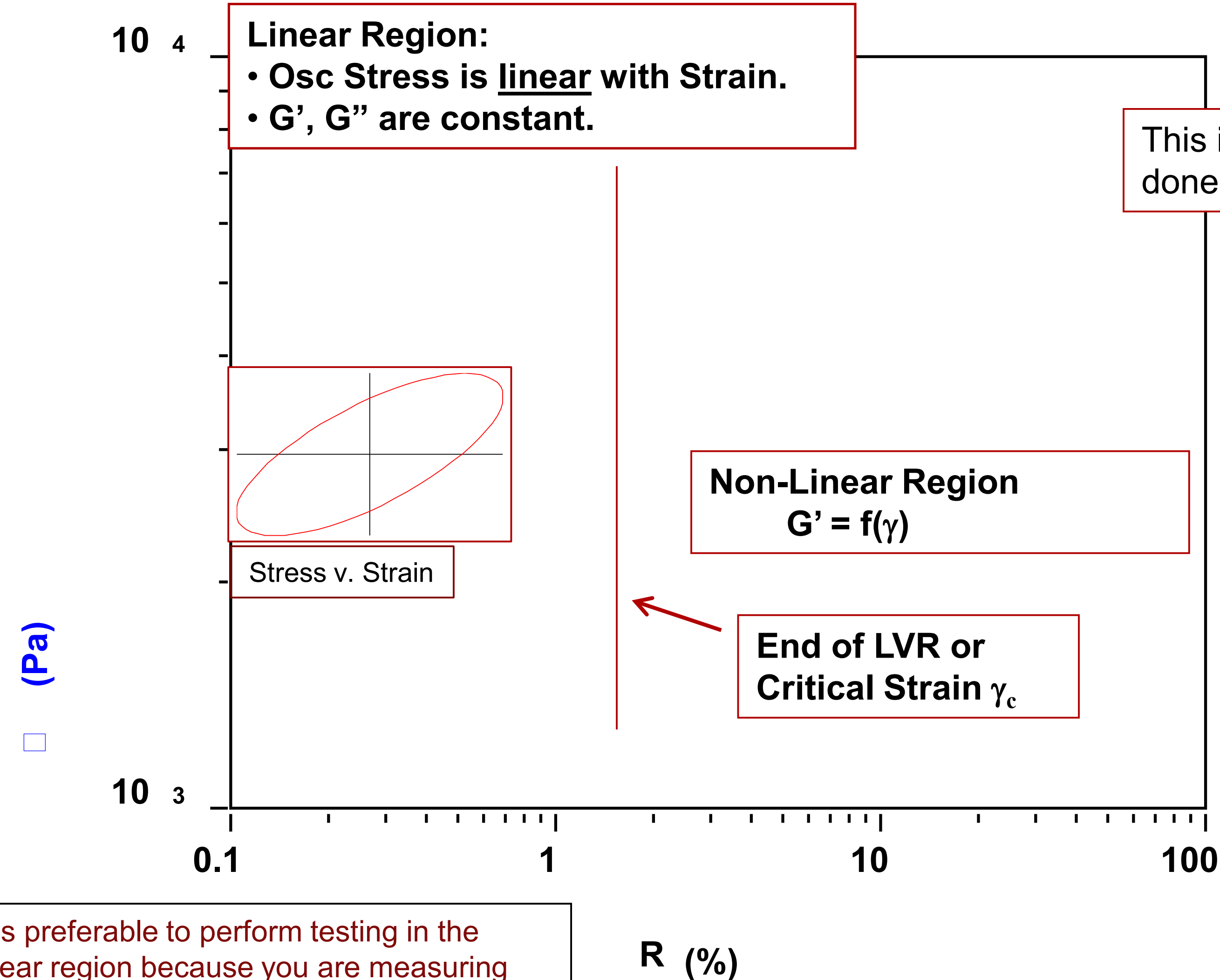
Parameter	Shear	Elongation	Units
Strain	$\gamma = \gamma_0 \sin(\omega t)$	$\varepsilon = \varepsilon_0 \sin(\omega t)$	---
Stress	$\sigma = \sigma_0 \sin(\omega t + \delta)$	$\tau = \tau_0 \sin(\omega t + \delta)$	Pa
Storage Modulus (Elasticity)	$G' = (\sigma_0/\gamma_0)\cos\delta$	$E' = (\tau_0/\varepsilon_0)\cos\delta$	Pa
Loss Modulus (Viscous Nature)	$G'' = (\sigma_0/\gamma_0)\sin\delta$	$E'' = (\tau_0/\varepsilon_0)\sin\delta$	Pa
Tan δ	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

Cox-Merz Rule for Linear Polymers: $\eta^*(\omega) = \eta(\dot{\gamma})$ @ $\dot{\gamma} = \omega$

Dynamic Testing

- Dynamic Stress or Strain Sweep
- Dynamic Time Sweep
- Dynamic Frequency Sweep
- Dynamic Temperature Ramp
- Dynamic Temperature Sweep

Linear Viscoelasticity



This is typically the first test done on unknown materials.

It is preferable to perform testing in the linear region because you are measuring intrinsic properties of the material, not material that has been altered.

Dynamic time sweep following shear

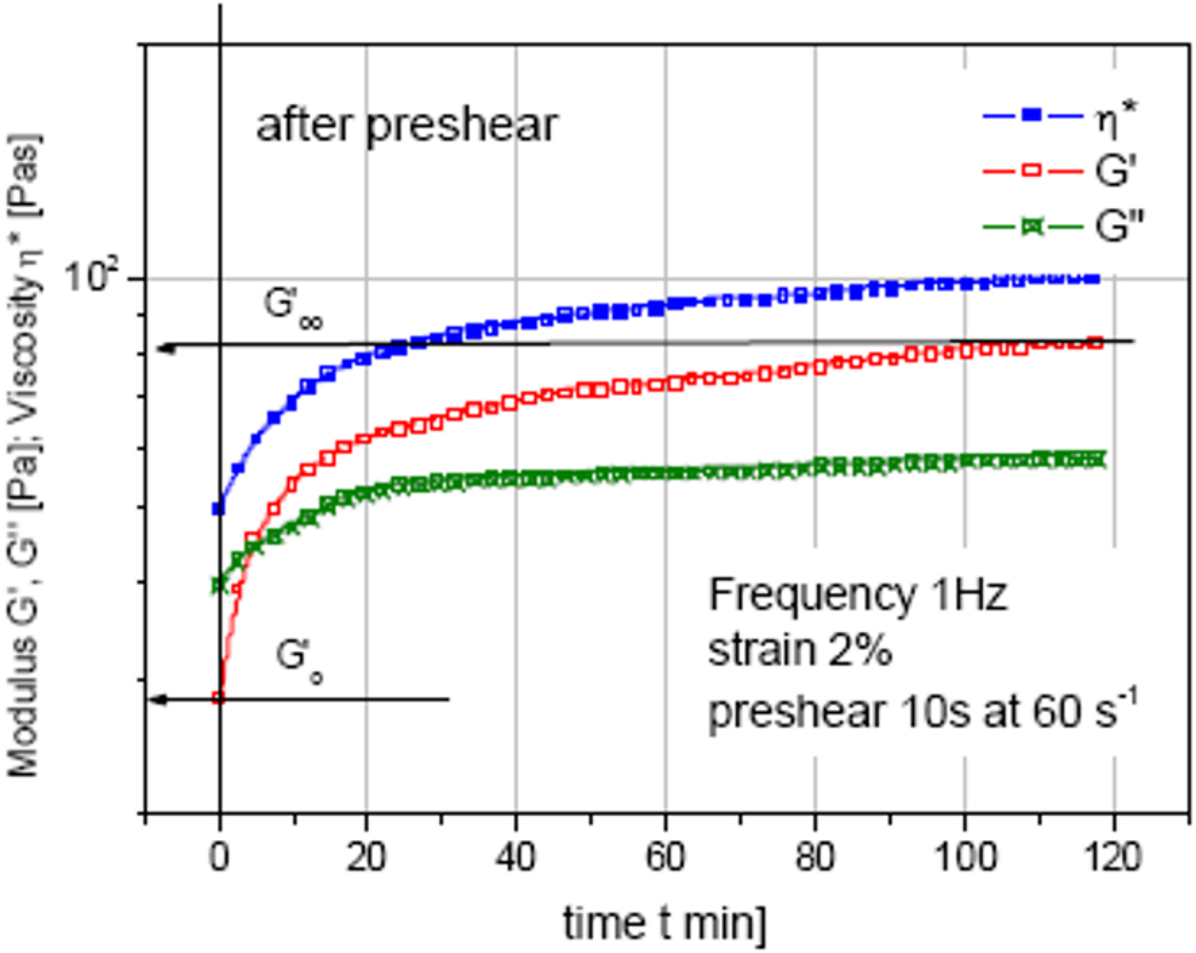
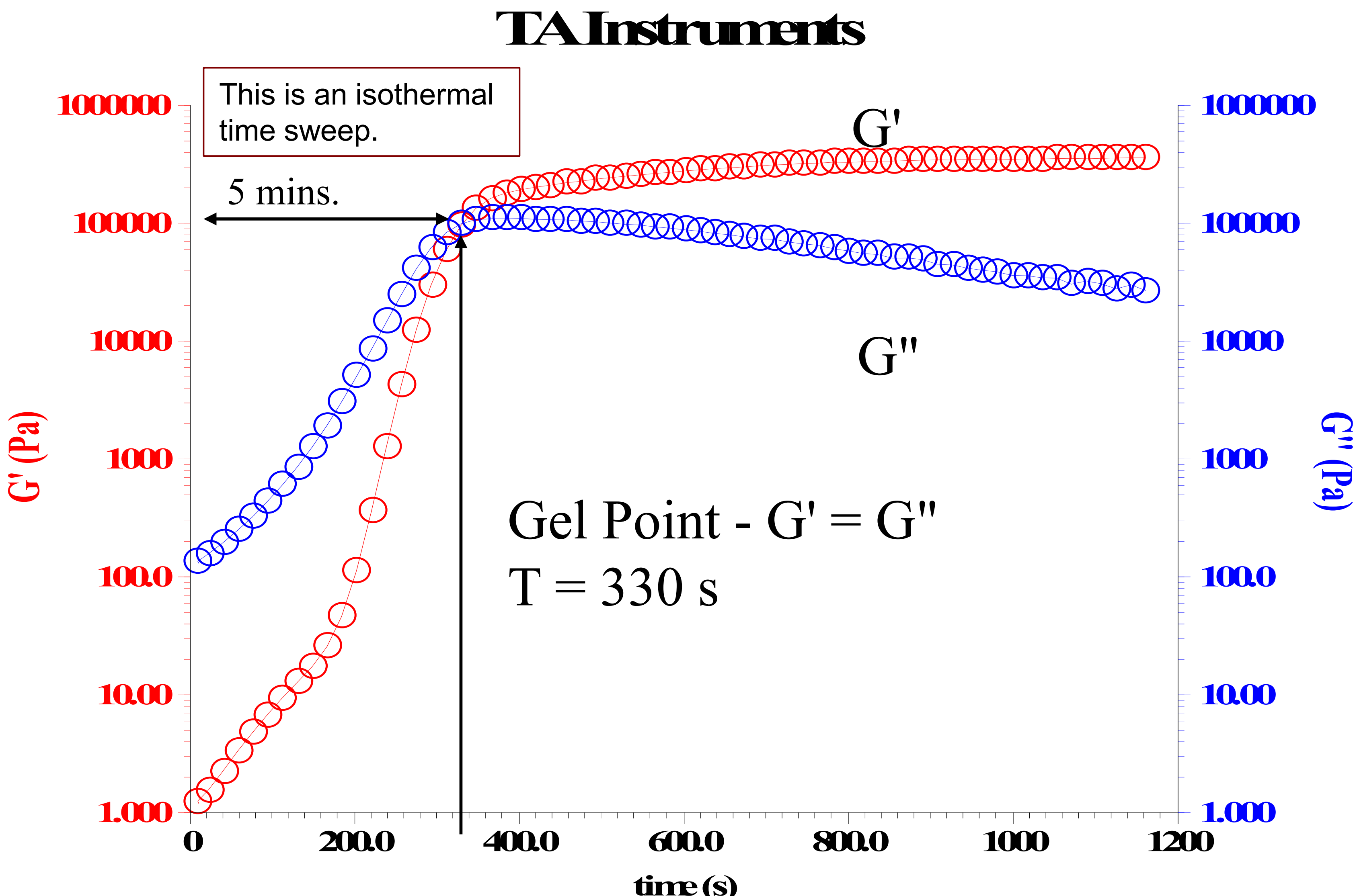


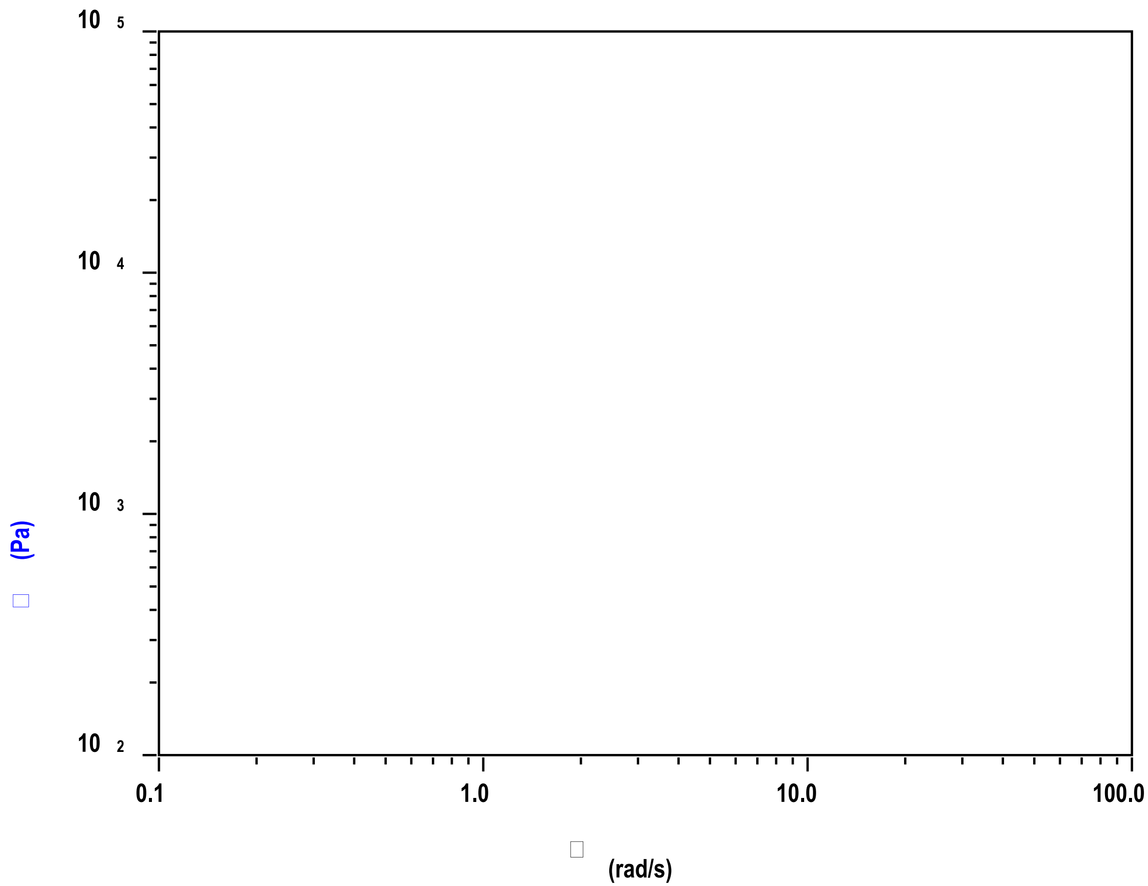
Figure 7: Recovery of structure

This shows how rapidly the structure is rebuilding.

Cure of a "5 minute" Epoxy



Frequency Sweep on PDMS



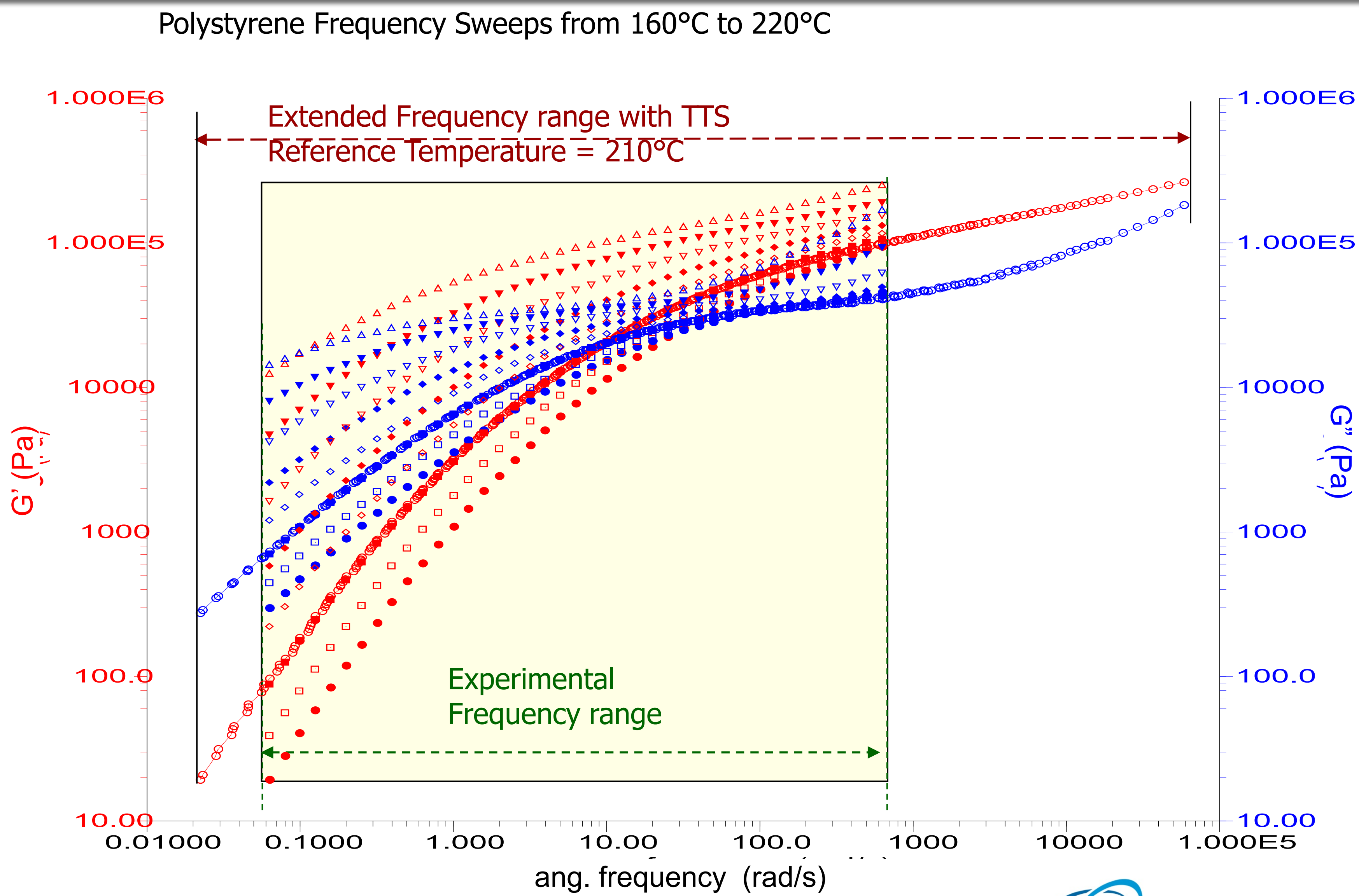
High frequency – elasticity predominates.
Low frequency – viscosity predominates.

Most frequent test for polymer melts:
ASTM D4440 – Standard Test Method for Plastics: Dynamic Mechanical Properties: Melt Rheology

100 to 0.1 rad/sec
5 pts. per decade
3 minutes of equilibration
Frequency sweep takes about 6.5 min.

Cox-Merz Rule for Linear Polymers: $\eta^*(\omega) = \eta(\square)$ @ $\square = \omega$

ETC Application: TTS on Polymer Melt



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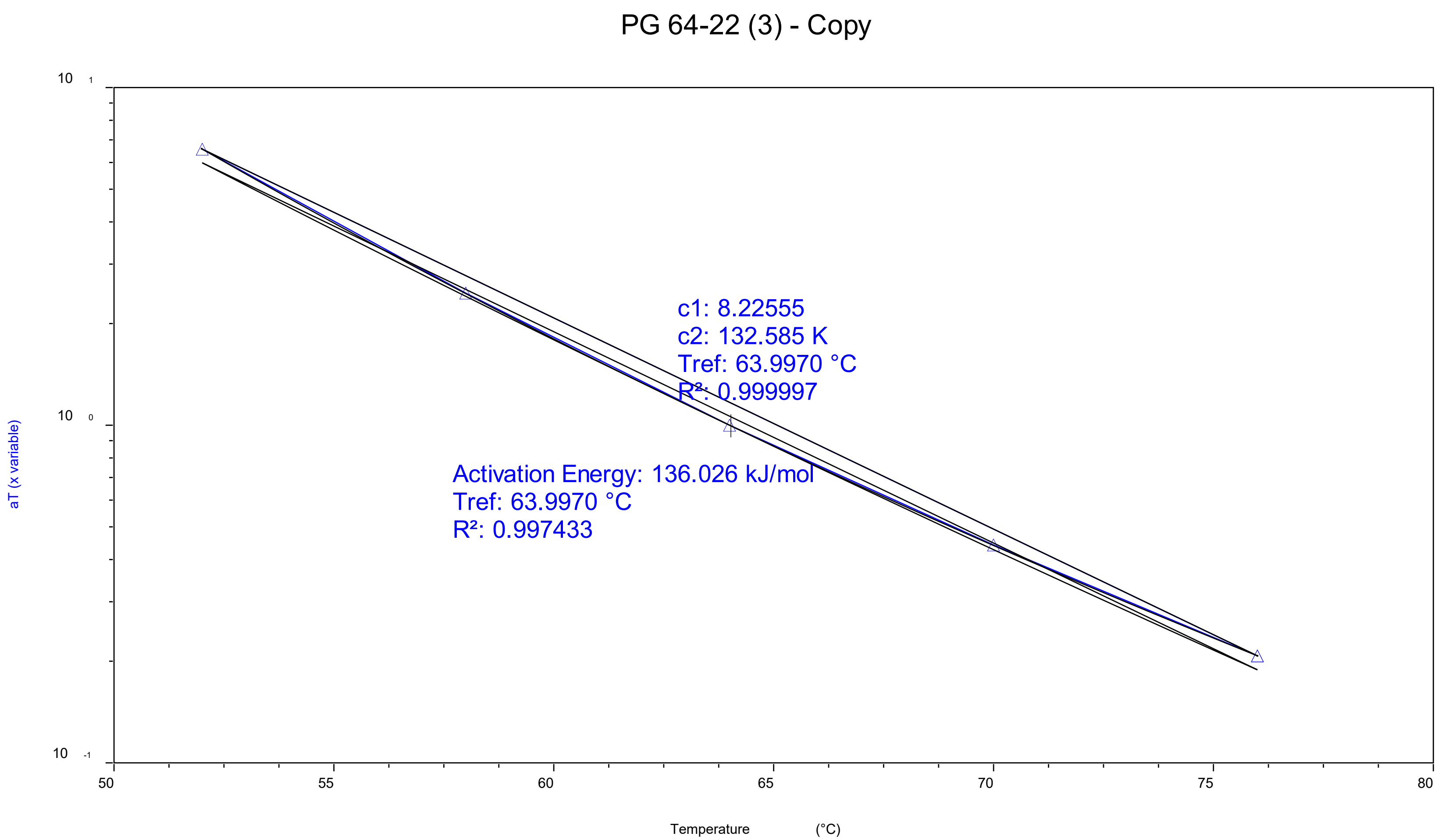
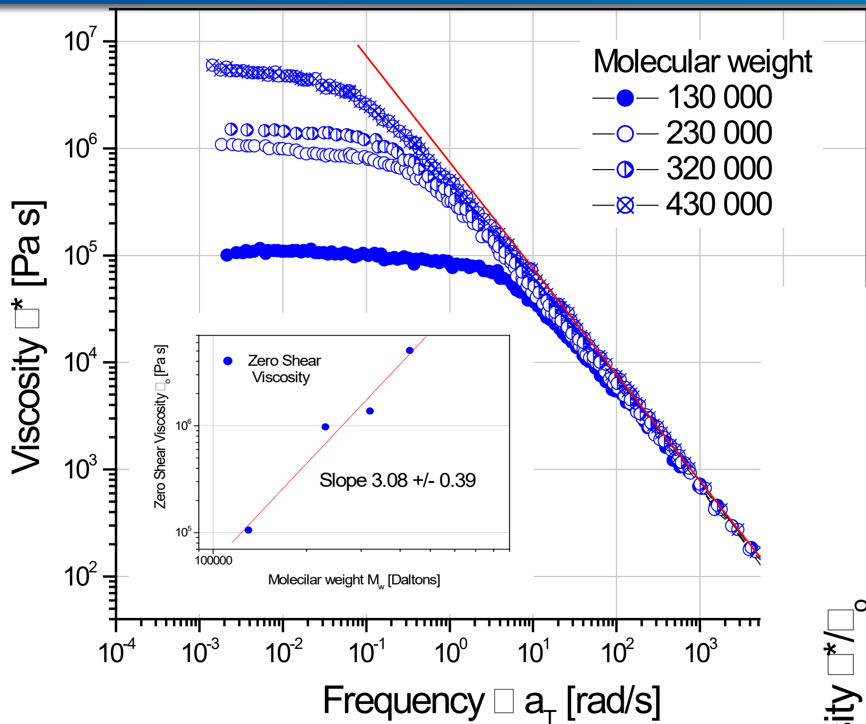
Shift Factors for TTS

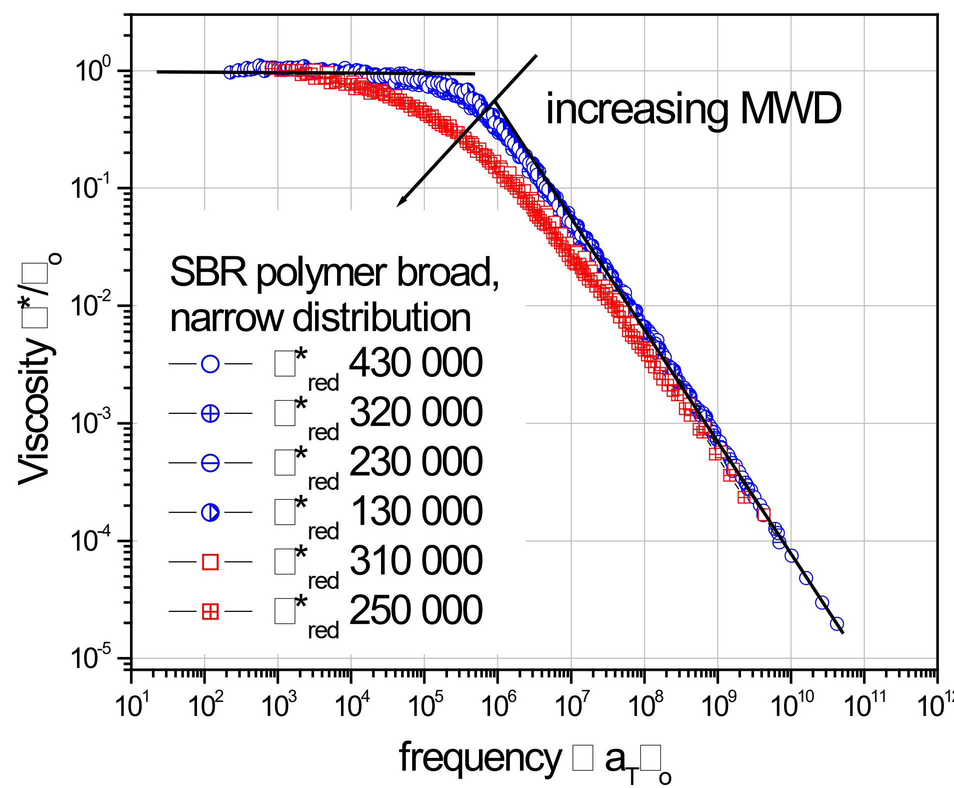


Dynamic testing: Dependence on M_w and MWD

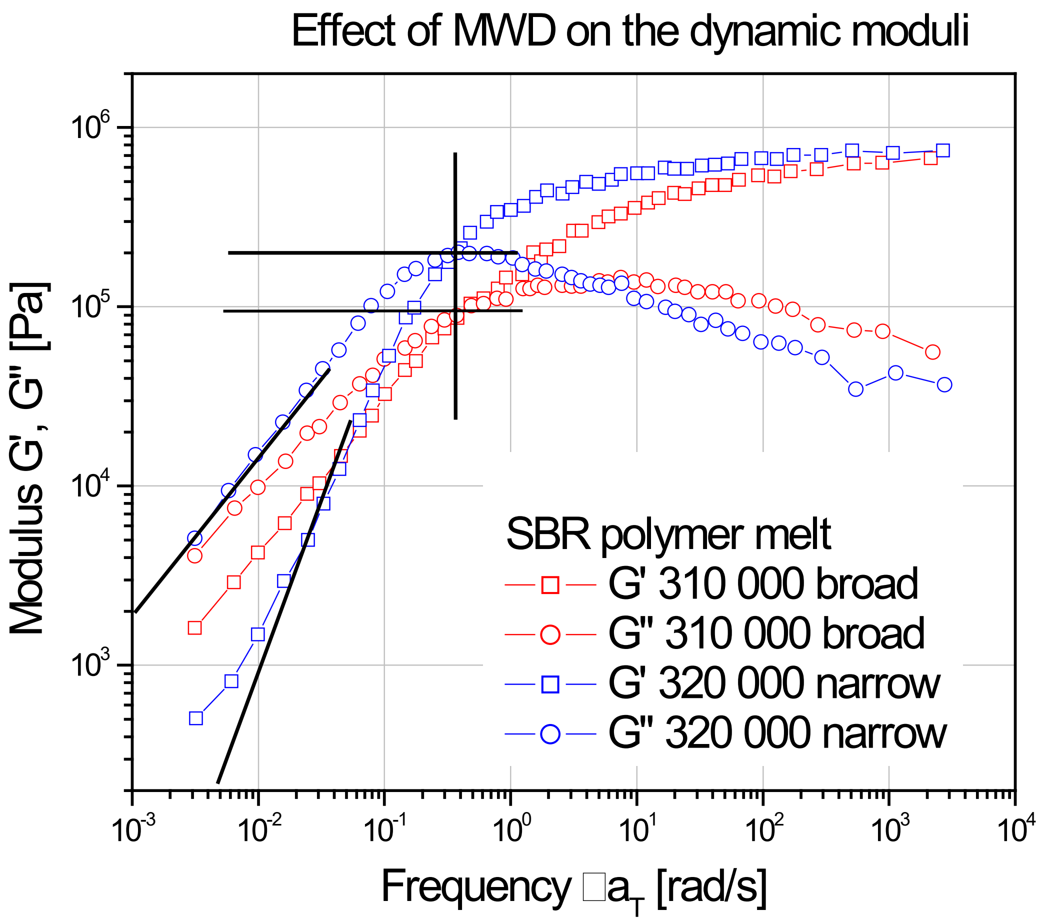


Zero shear viscosity increases with increasing MW

When shifting along an axis of -1, all the curves can be superposed, unless the width of the MWD is not the same.



MWD and Dynamic Moduli

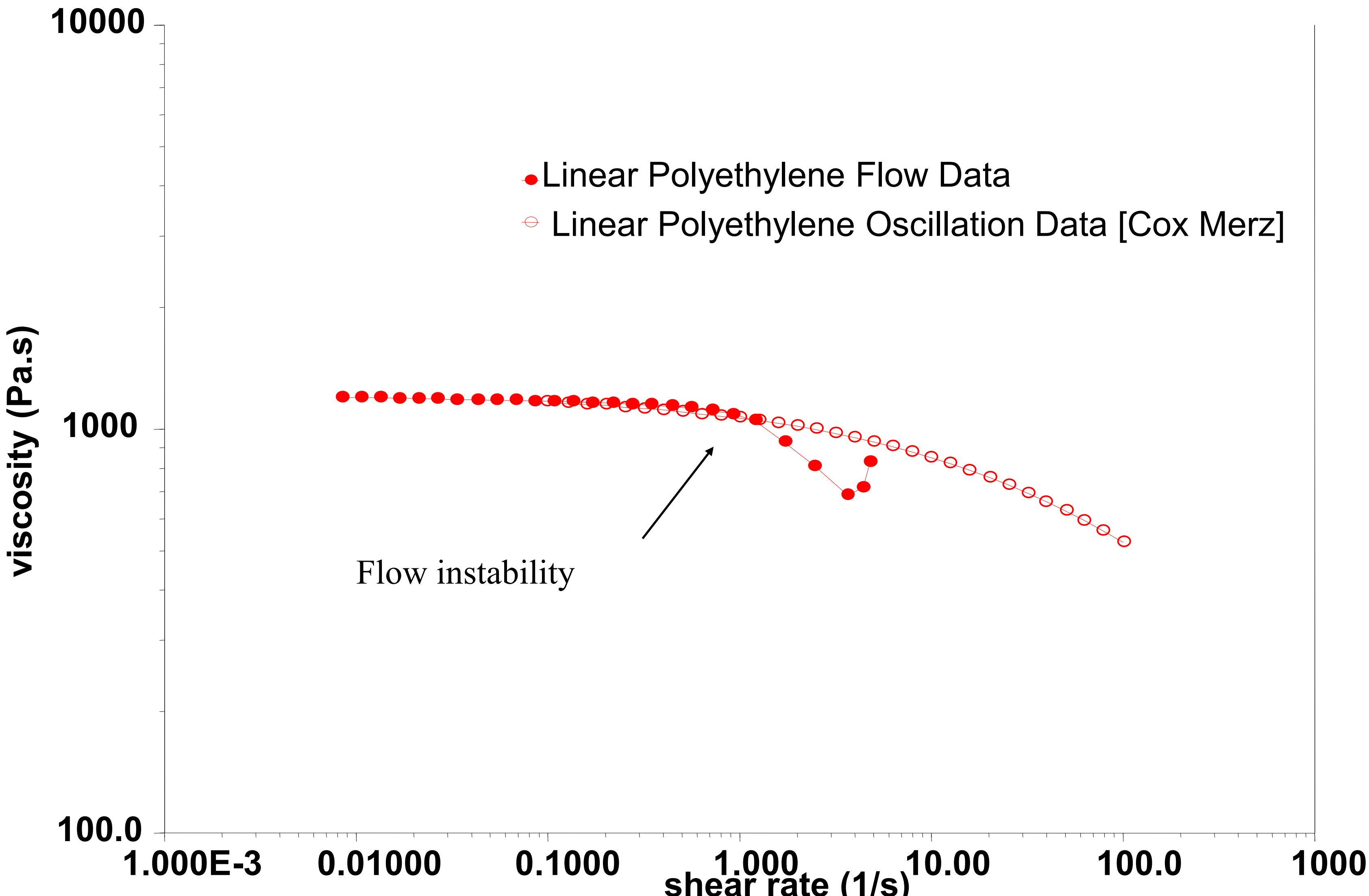


- **The storage and loss modulus of a typical polymer melt cross over between 1 and 100 rad/s.**
- **$\omega_c \propto 1/M_w$.**
- **$G_c \propto 1/MWD$.**

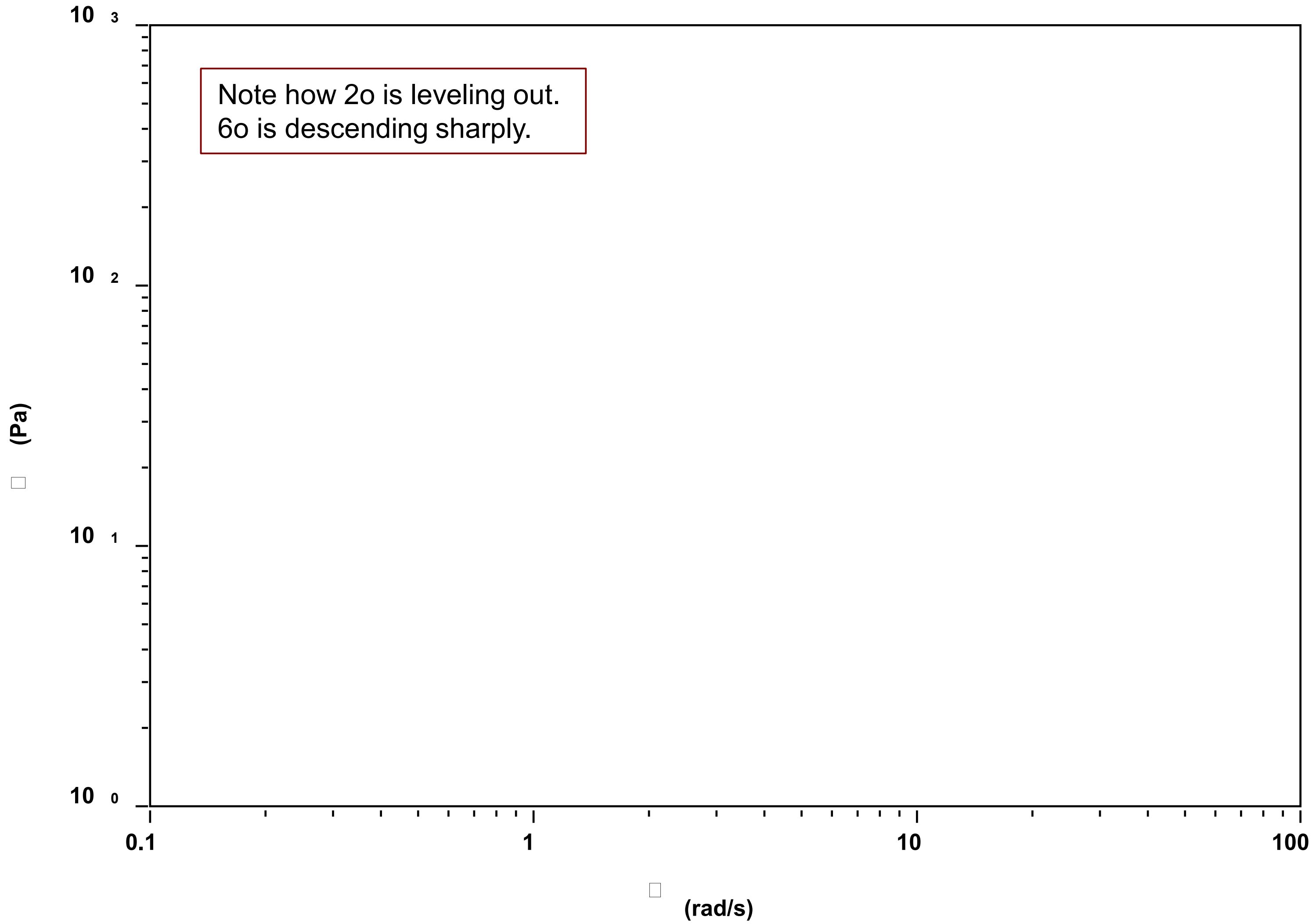
Higher crossover frequency = lower M_w

Higher crossover Modulus = narrower MWD

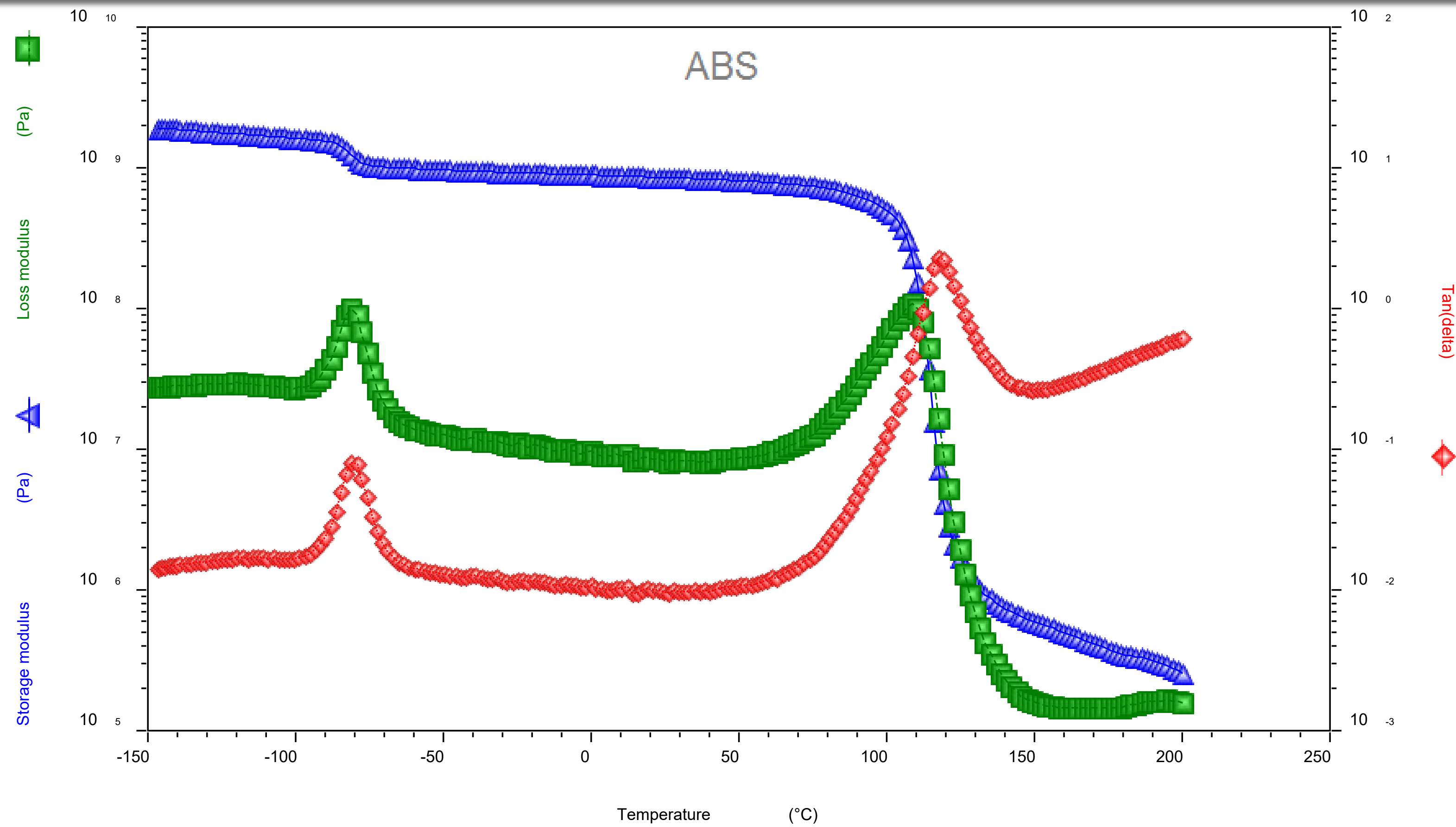
Example of Cox-Merz Rule



Coatings Frequency Sweep



Dynamic Temperature Ramp - Torsion



The storage modulus curve indicates the temperature range over which the material can contribute mechanically. Glass transition temperatures are observed by onset in the storage modulus curve and peaks in the loss modulus and tan delta curves.

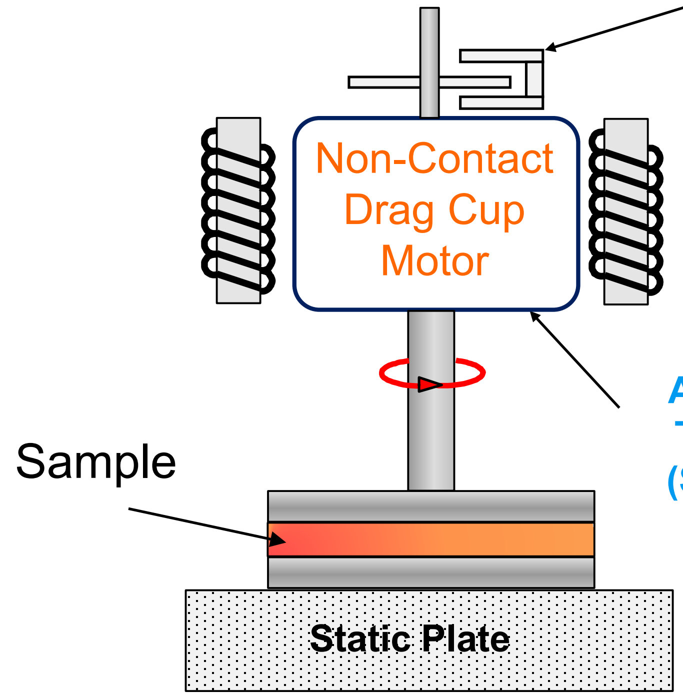
THE RHEOMETERS

DHR Rotational Rheometer

DHR

Controlled Stress
Single Head
CMT

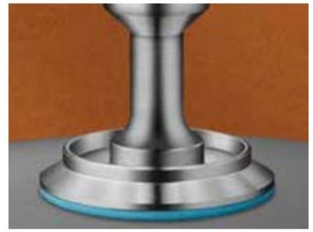
Single head or CMT
Combined motor & transducer



DHR Accessories – Visual Display

Peltier Plate Temperature Systems

Advanced Peltier Plate



Dual Stage Peltier Plate

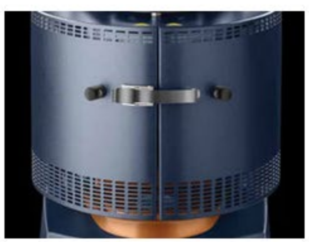
Upper Heated Plate for Peltier Plate

Peltier Concentric Cylinders

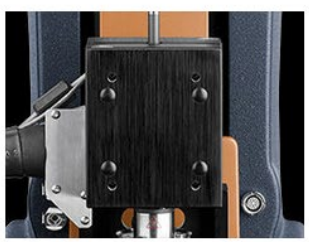
Electrically Heated Cylinder (EHC)

Pressure Cell

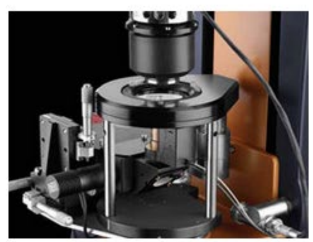
Electrically Heated Plates



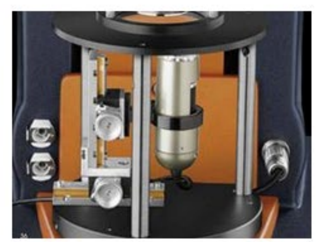
Environmental Test Chamber



Relative Humidity Accessory



Modular Microscope (MMA)



Optical Plate

You can see the updated list of accessories on our website, www.tainstrument.com.

DHR Accessories – Visual Display

Small Angle Light Scattering

Interfacial Accessories

Tribo-Rheometry Accessory

Magneto-Rheology

Electro-Rheology

UV Curing Accessories

Dielectric Measurement

Immobilization Cell

Starch Pasting Cell

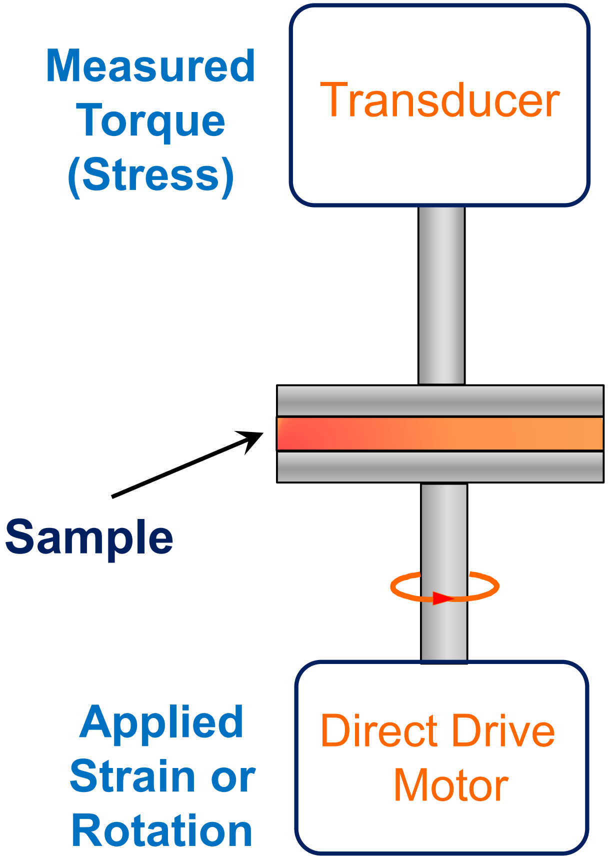
Dynamic Mechanical Analysis

For the current listing of accessories, go to https://www.tainstruments.com.

Open Boundary Rotational Rheometer - SMT

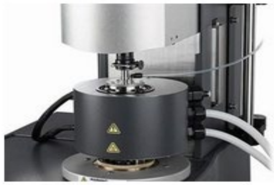
ARES G2

Controlled
Strain
SMT or DH



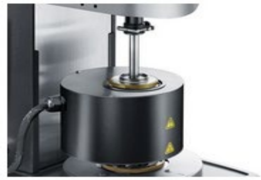
SMT: Separate Motor & Transducer
DH: Dual Head

ARES-G2 Accessories

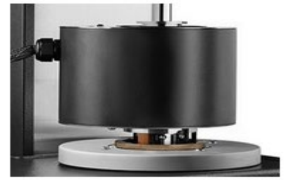


High Sensitivity Pressure Cell (HSPC)

Forced Convection Oven (FCO)

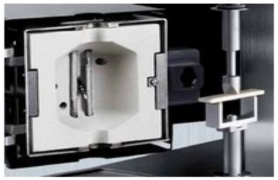


Advanced Peltier System (APS)



Orthogonal Superposition & 2D-SAOS

Electrorheology (ER) Accessory



Dynamic Mechanical Analysis (DMA)

UV Curing Accessory

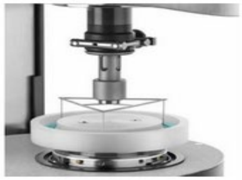
Dielectric Thermal Analysis Accessory (DETA)

Extensional Viscosity Fixture (EVF)

Tribo-Rheometry Accessory

Air Chiller System

Cone and Partitioned Plate Accessory



Interfacial Rheology

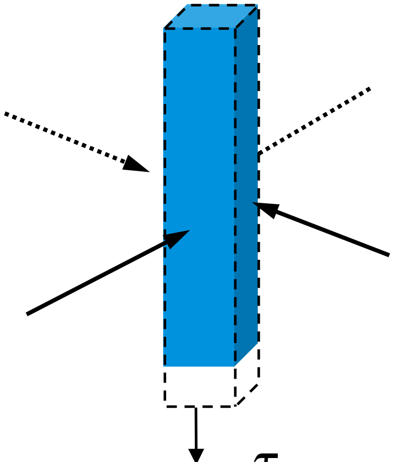
For the current listing of accessories, go to https://www.tainstruments.com.

1. ROTATIONAL RHEOLOGY

2. DYNAMIC MECHANICAL ANALYSIS (LINEAR TESTING)

Tensile Deformation

Young's Modulus



$$E = \frac{\tau}{\varepsilon}$$

<u>Deformation Parameters</u>

L_0 = Initial Length (m)

L = Stretched Length (m)

ε = 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.**

<u>Force Parameters</u>

T = Tensile force (Newtons)

w_0 = Initial Width (m)

t_0 = Initial Thickness (m)

τ = Tensile Stress, $T/(w_0 * t_0)$ (Pa)

Conversions:
Machine → Rheological
Displacement → Strain
Force → Stress

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

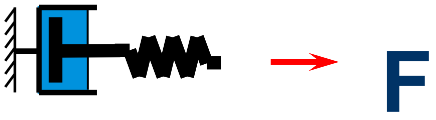
<u>Elongational Properties</u>

E = τ/ε (Pa) Modulus

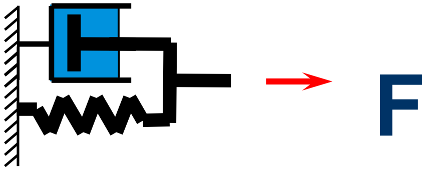
D = ε/τ (1/Pa) Compliance

UNIDIRECTIONAL TYPES OF TESTS ON THE DMA

- **TRANSIENT**
  - Stress Relaxation
    - Deformation applied instantaneously ⇒ Force measured as a function of time
    - Deformation (mm) converted to Strain (ε), Force (N) to Stress (τ)
    - Stress (τ)/Strain(ε) = Modulus (E)

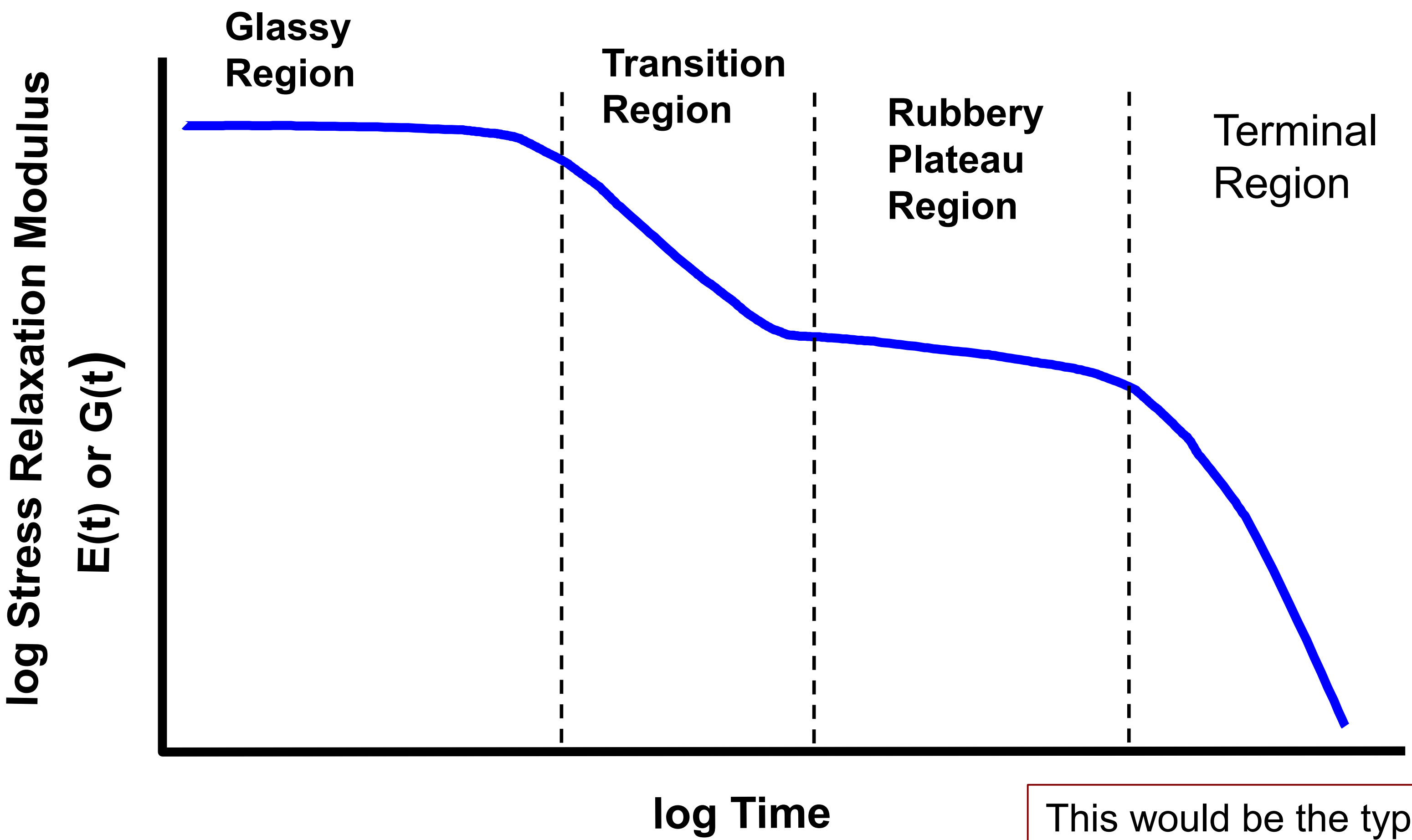


  - Creep
    - Force applied instantaneously ⇒ Deformation measured as a function of time
    - Force to Stress (τ), Deformation converted to Strain (ε)
    - Strain (ε)/Stress (τ) = Compliance (D)



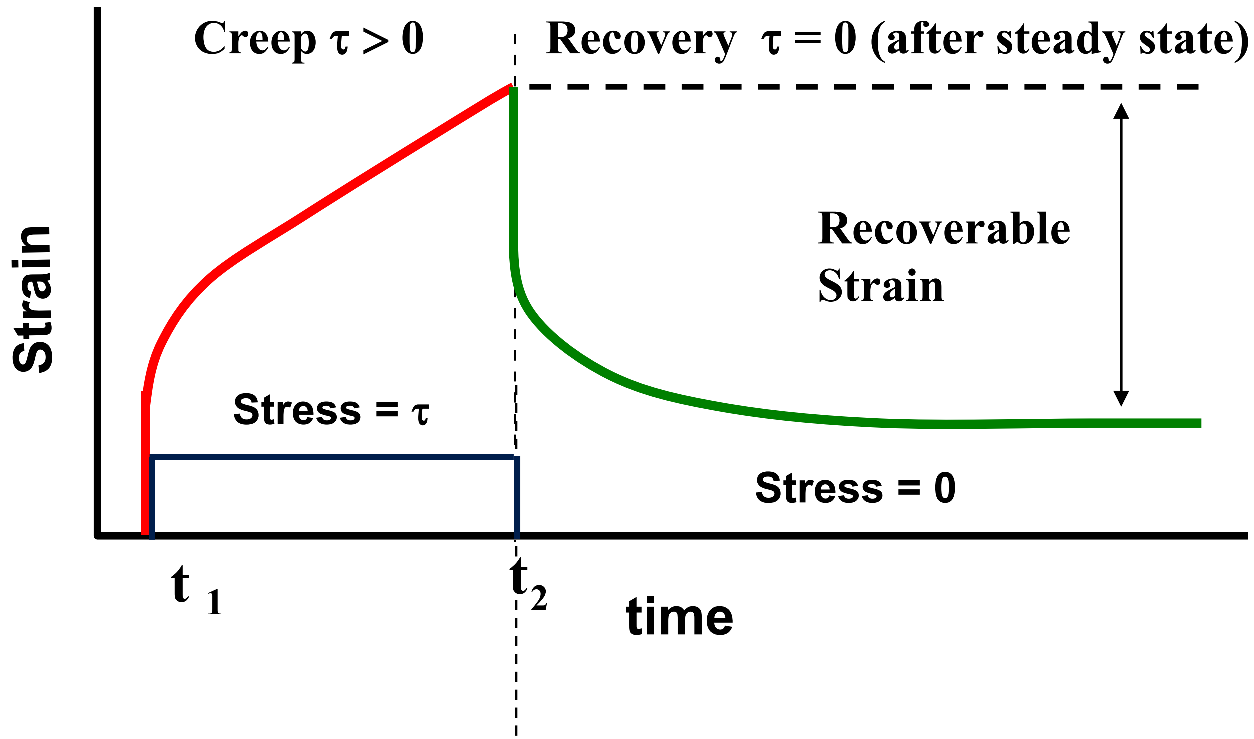
- **PRACTICAL**
  - Strain Ramp
    - Strain increased linearly with time or, optionally, exponential with the RSA-G2
  - Iso-Strain
    - Strain held constant as temperature is varied
  - Stress Ramp
    - Stress increased linearly or exponentially with time
  - Controlled Stress
    - Stress held constant 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

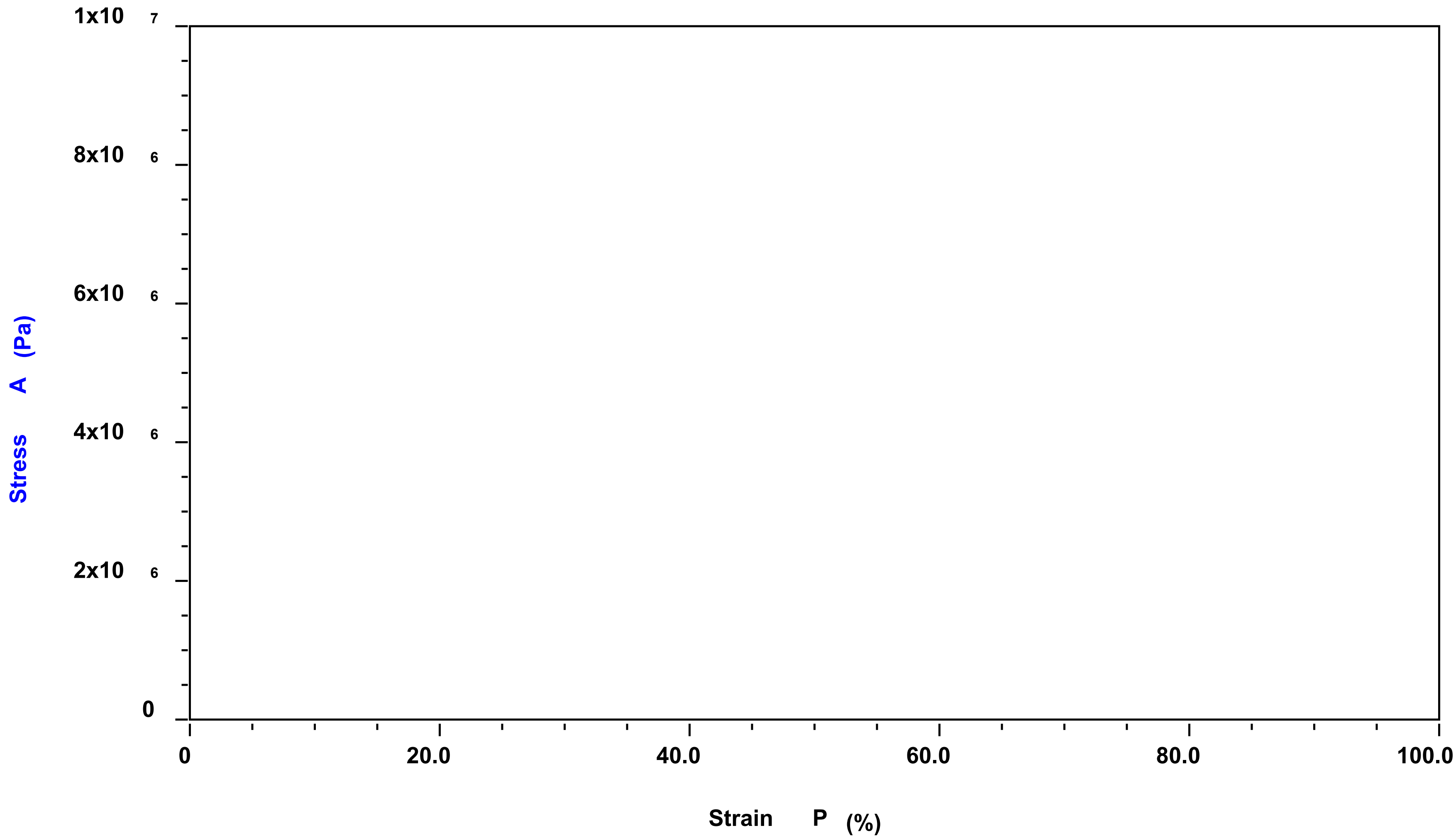


The greater the elasticity, the greater the recovery.

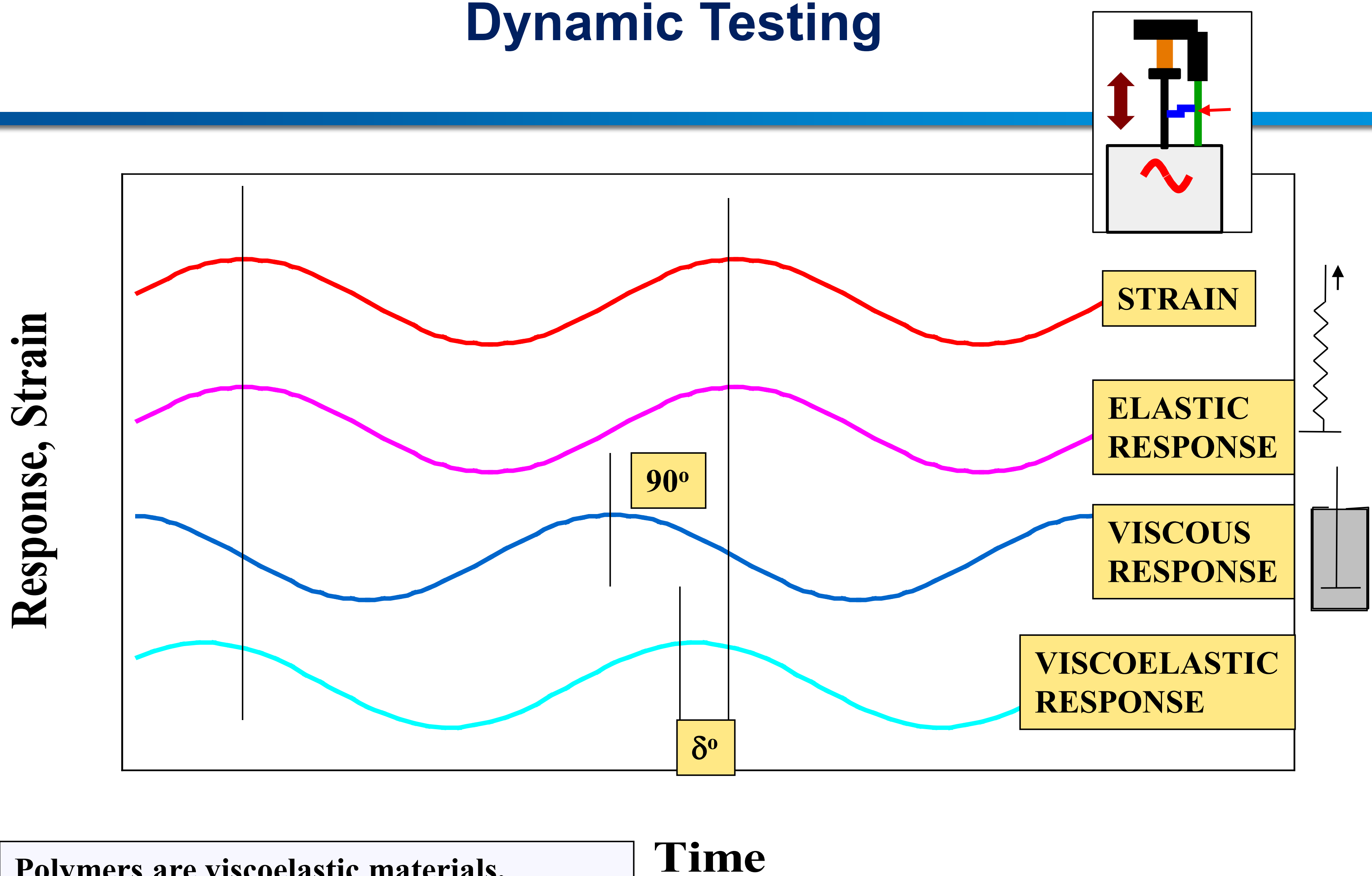
With uncross-linked systems, strain increases indefinitely.
If you have reached steady state flow, you can calculate the viscosity and the recoverable compliance.
With cross-linked systems, there is a limiting strain.

Reference: Mark, J., et.al., Physical Properties of Polymers ,American Chemical Society, 1984, p. 102.

Polyethylene Stress Ramp (or Strain Ramp)



Dynamic Testing



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

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 δ	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.

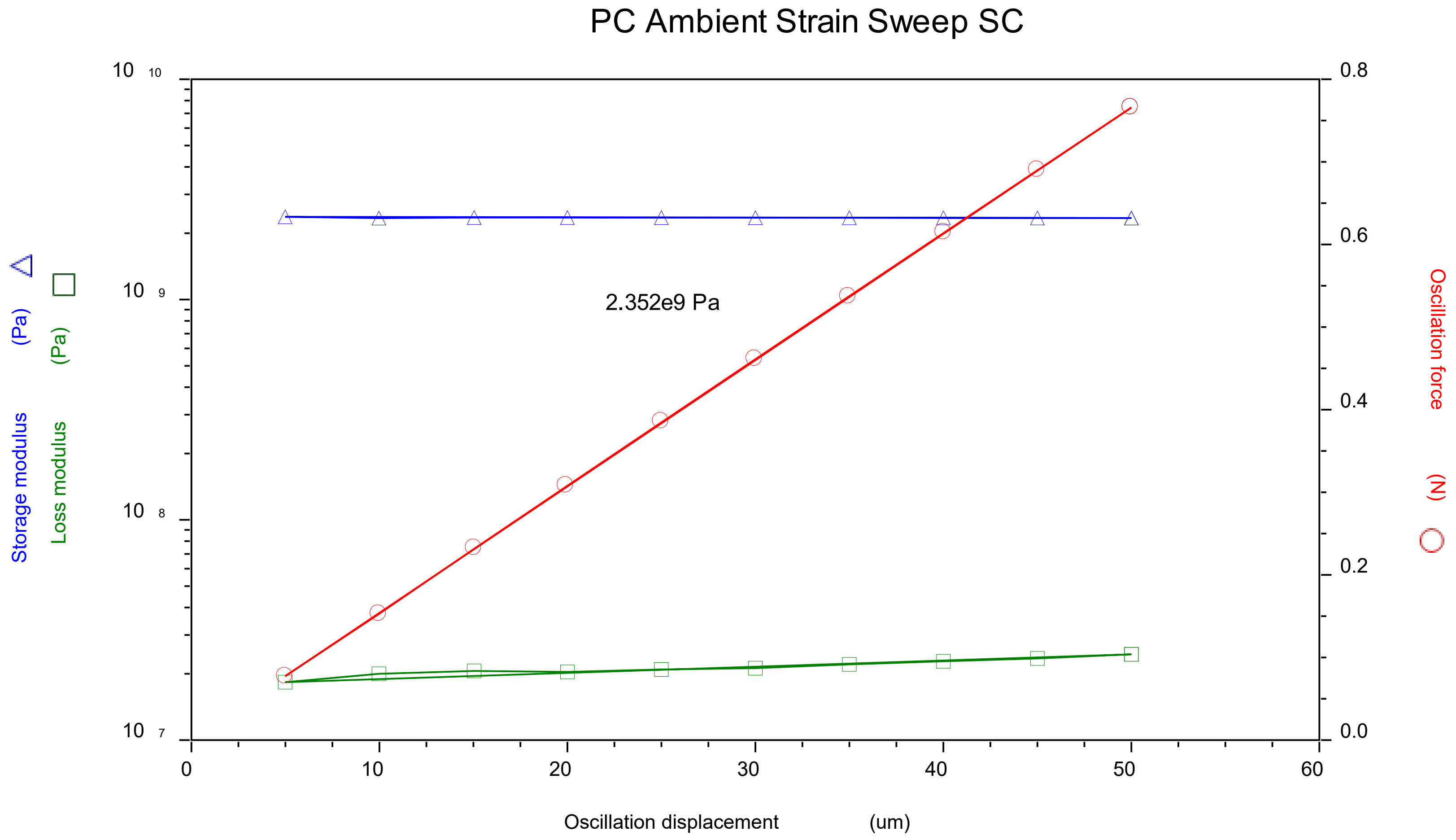
Dynamic Oscillatory Testing Methods

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

Most common sequence

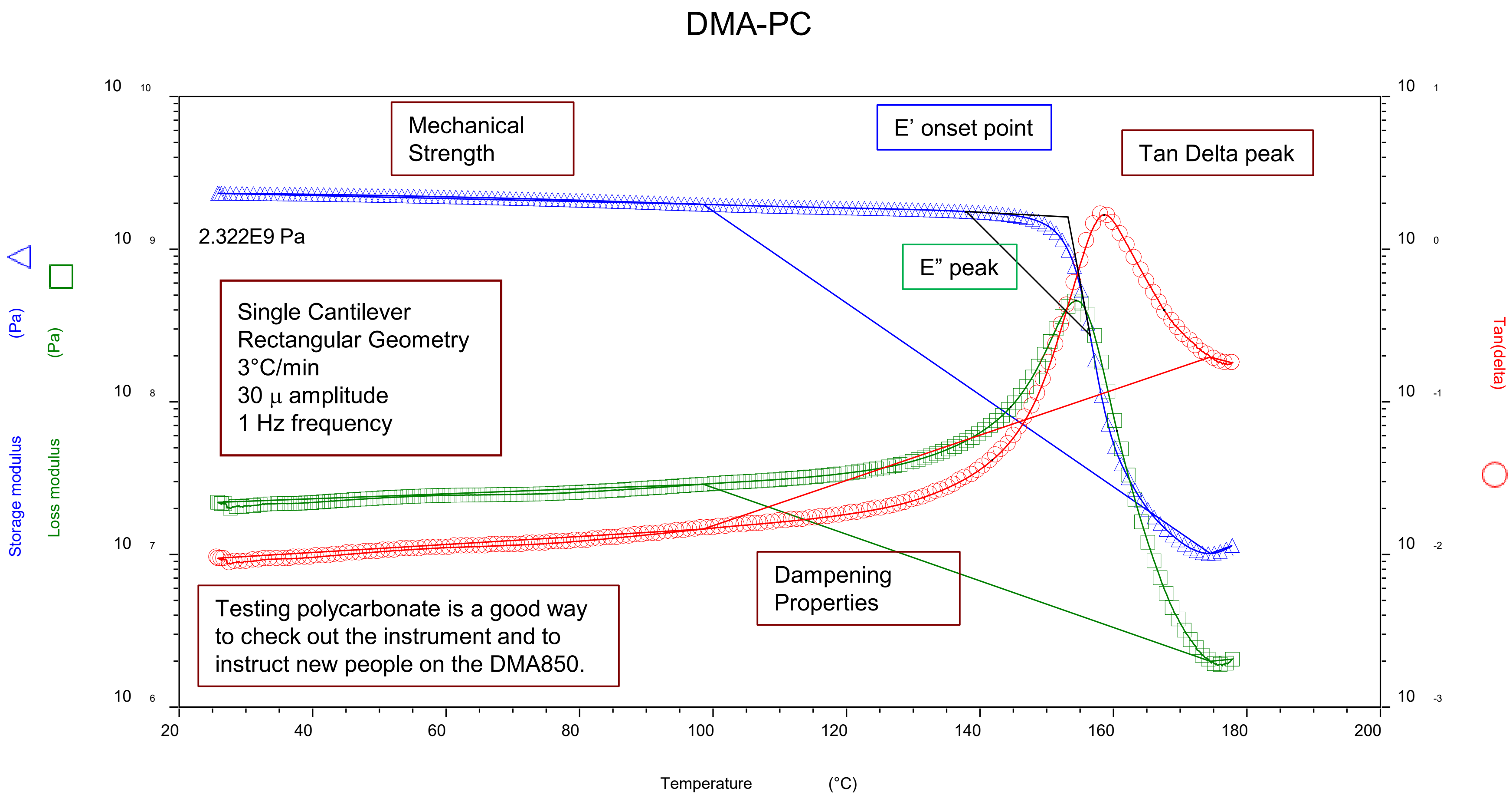
- Strain sweep at 1 Hz to find the “sweet spot” for testing and the Linear Viscoelastic Region (LVR)
- Temperature ramp at 1 Hz and 3 C/min using amplitude from strain sweep testing.

Dynamic Strain Sweep



The material shows a nice Linear Viscoelastic Region (LVR).
The oscillation force at 30 microns is about 0.4 N, which is definitely in the sweet spot for our DMA's.
We aim for a storage modulus of 2350 MPa at room temperature with polycarbonate.

Polycarbonate Testing on the DMA 850



This is the main test performed on DMA instruments.

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 Transition

- "The glass transition is associated with the onset of long-range cooperative segmental mobility in the amorphous phase, in either an amorphous or semi-crystalline polymer."
- Any factor that affects segmental mobility will affect T_g, including…
  - the nature of the *moving segment*,
  - chain stiffness or steric hindrance
  - the free volume available for segmental motion

Turi, Edith, A, Thermal Characterization of Polymeric Materials, Second Edition, Volume I., Academic Press, Brooklyn, New York, P. 508.

Glass Transition E' Onset, E" Peak, and Tan δ Peak

- **Storage Modulus E' Onset**:
  - Occurs at lowest temperature, relates to mechanical failure

- **Loss Modulus E" Peak**:
  - Occurs at middle temperature
  - Related to the physical property changes
  - Reflects molecular processes - the temperature at the onset of segmental motion

- **Tan δ Peak:**
  - Occurs at highest temperature; Used historically in literature
  - 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.

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 (e.g. plasticizer.)

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

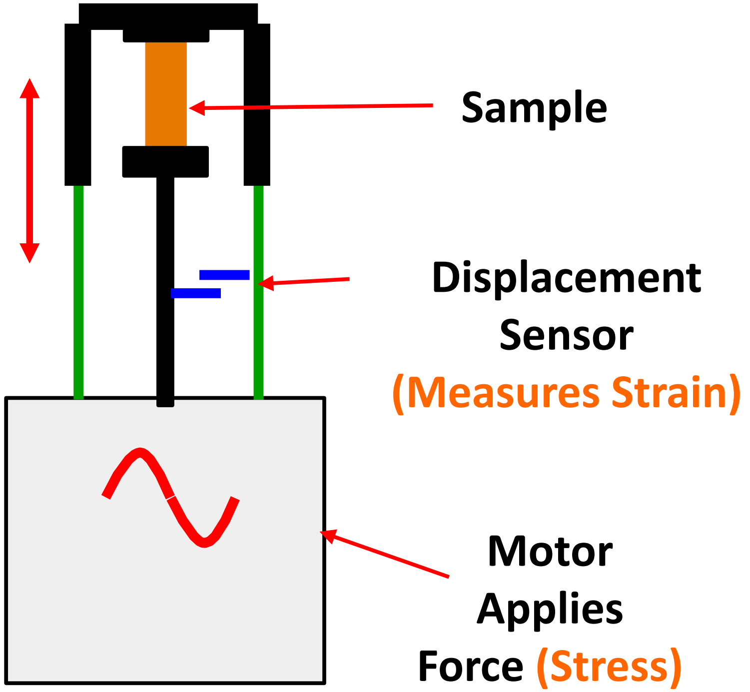
THE DMA'S

DMA 850

DMA 850

Controlled Stress

CMT – Combined Motor & Transducer

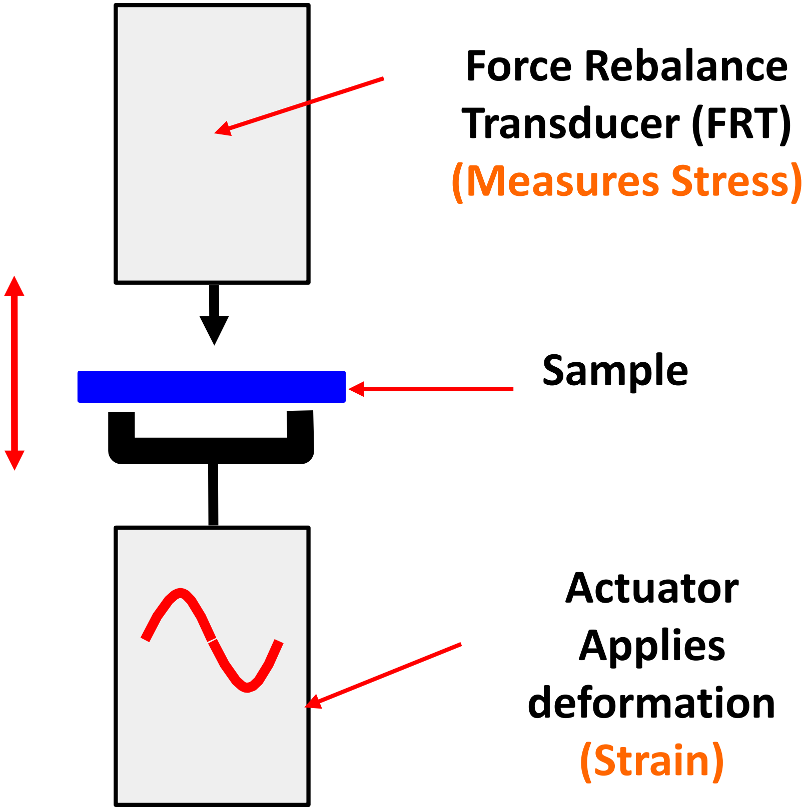


RSA-G2

RSA G2

Controlled Strain

SMT – Separate Motor & Transducer



DMA Specifications

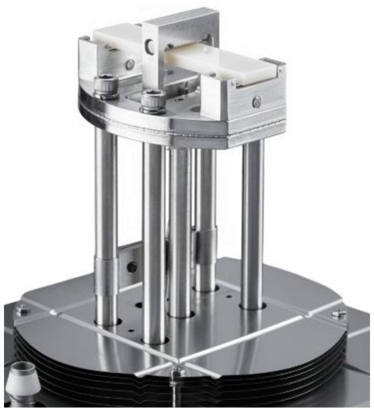
	DMA 850	RSA G2	ARES G2 DMA	DHR DMA (optional)
Max Force	18N	35N	20N	50N
Min Force	0.0001N	0.0005N	0.001N	0.1N
Frequency Range	0.01 to 1250 rad/s (1.6e-3 to 200 Hz)	1e-5 to 628 rad/s (1.6e-6 to 100 Hz)	6.3e-5 to 100 rad/s (1.0e-5 to 16 Hz)	6.3e-5 to 100 rad/s (1.0e-5 to 16 Hz)
Dynamic Deformation Range	+/- 0.5 to 10,0000mm	+/- 0.05 to 1,500mm	+/- 1 to 50 mm	+/- 1 to 100 mm
Control Stress/Strain	Control Stress (CMT)	Control Strain (SMT)	Control Strain (CMT)	Control Stress (CMT)
Heating Rate	0.1°C to 20°C/min	0.1°C to 60°C/min	0.1°C to 60°C/min	0.1°C to 60°C/min
Cooling Rate	0.1°C to 10°C/min	0.1°C to 60°C/min	0.1°C to 60°C/min	0.1°C to 60°C/min

Clamps for DMA 850

S/D Cantilever

Film/Fiber Tension

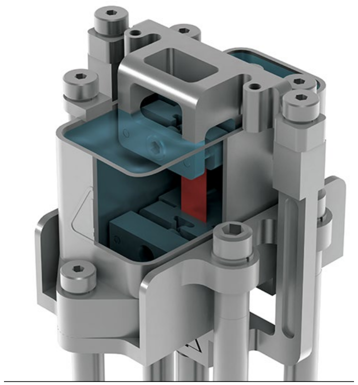
3-Point Bending



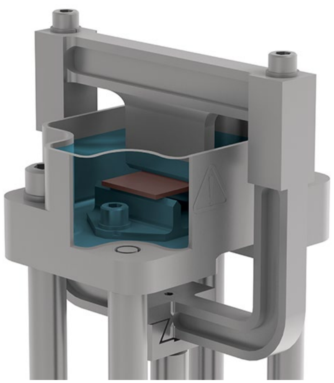
Compression

Shear Sandwich

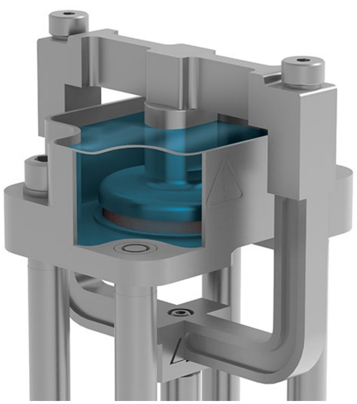
Submersible Tension



Submersible Bending



Submersible Compression



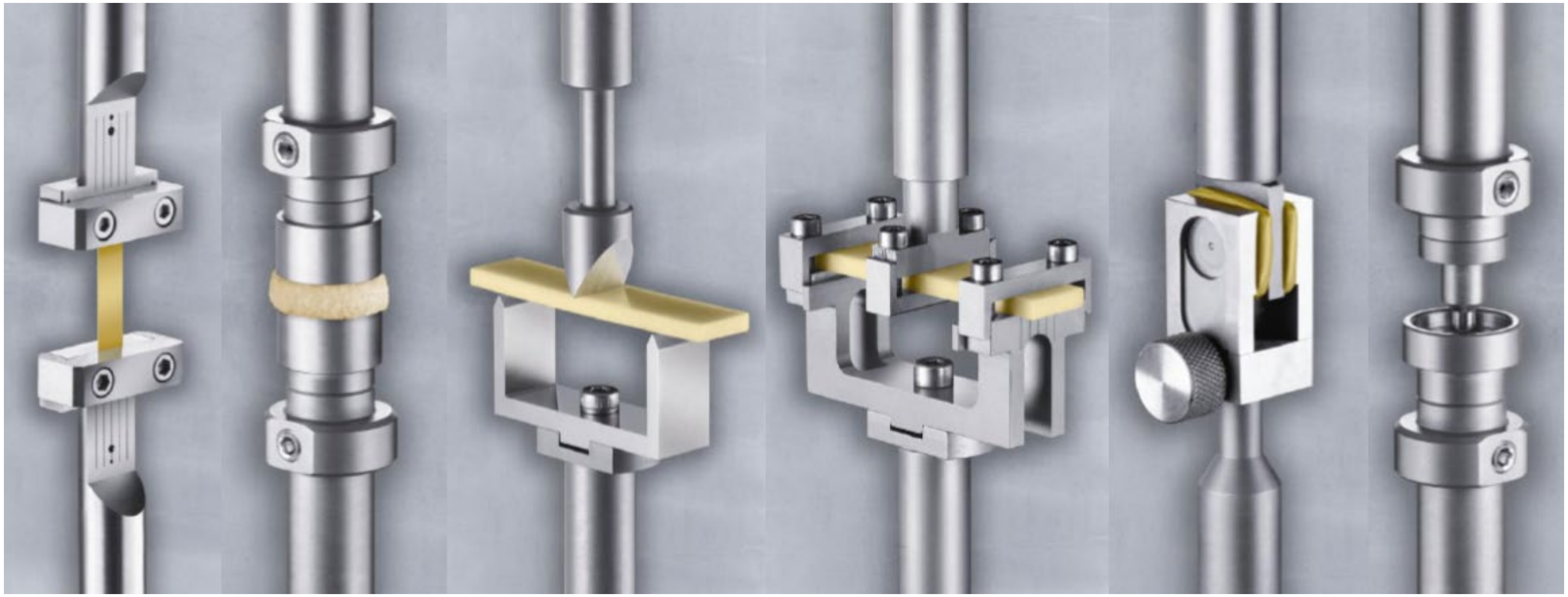
The standard size S/D cantilever clamp is included with the purchase of the DMA 850.

Clamps for RSA G2

Film/Fiber

3-Pt Bending

Shear Sandwich



Compression

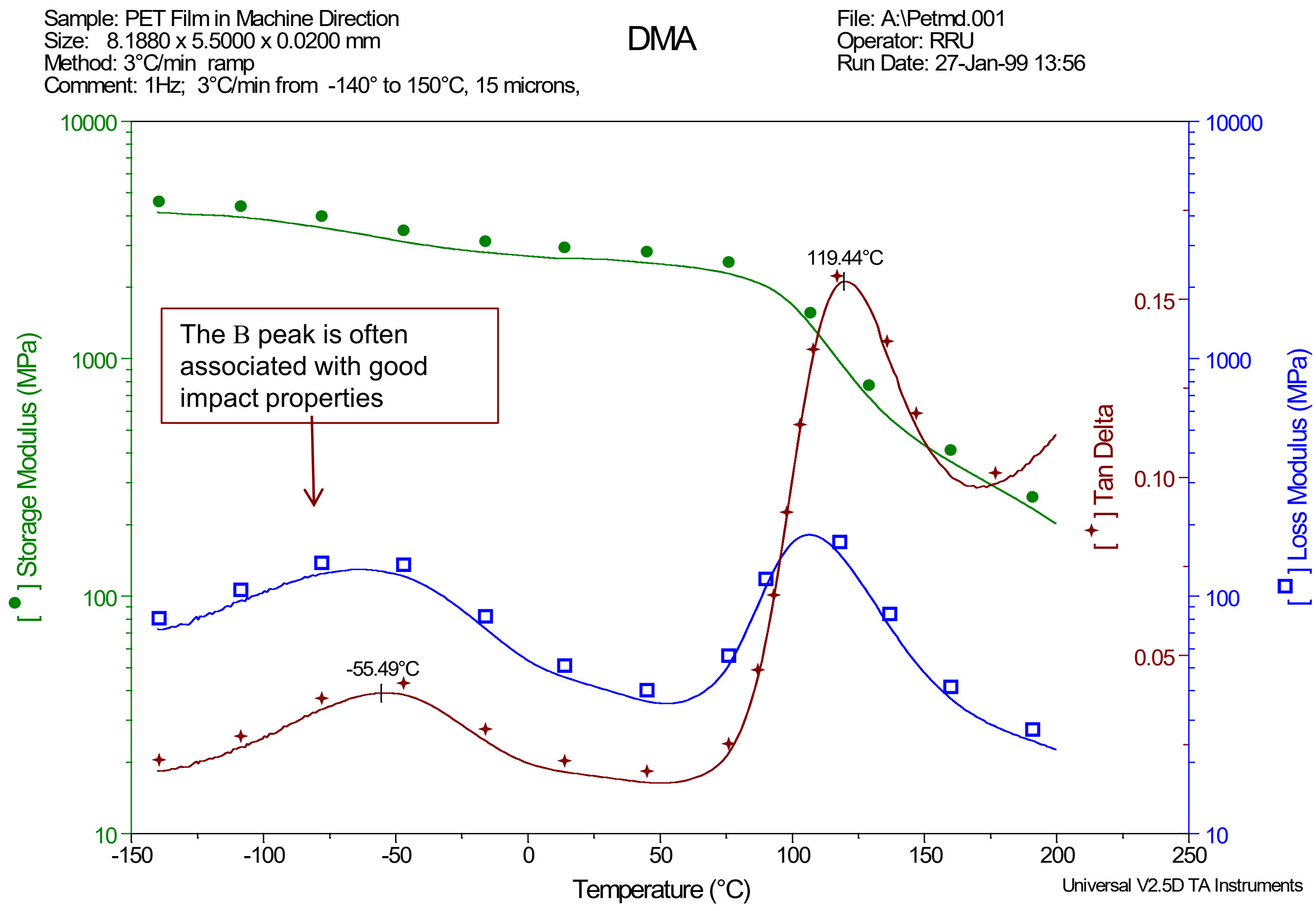
Cantilever

Contact Lens

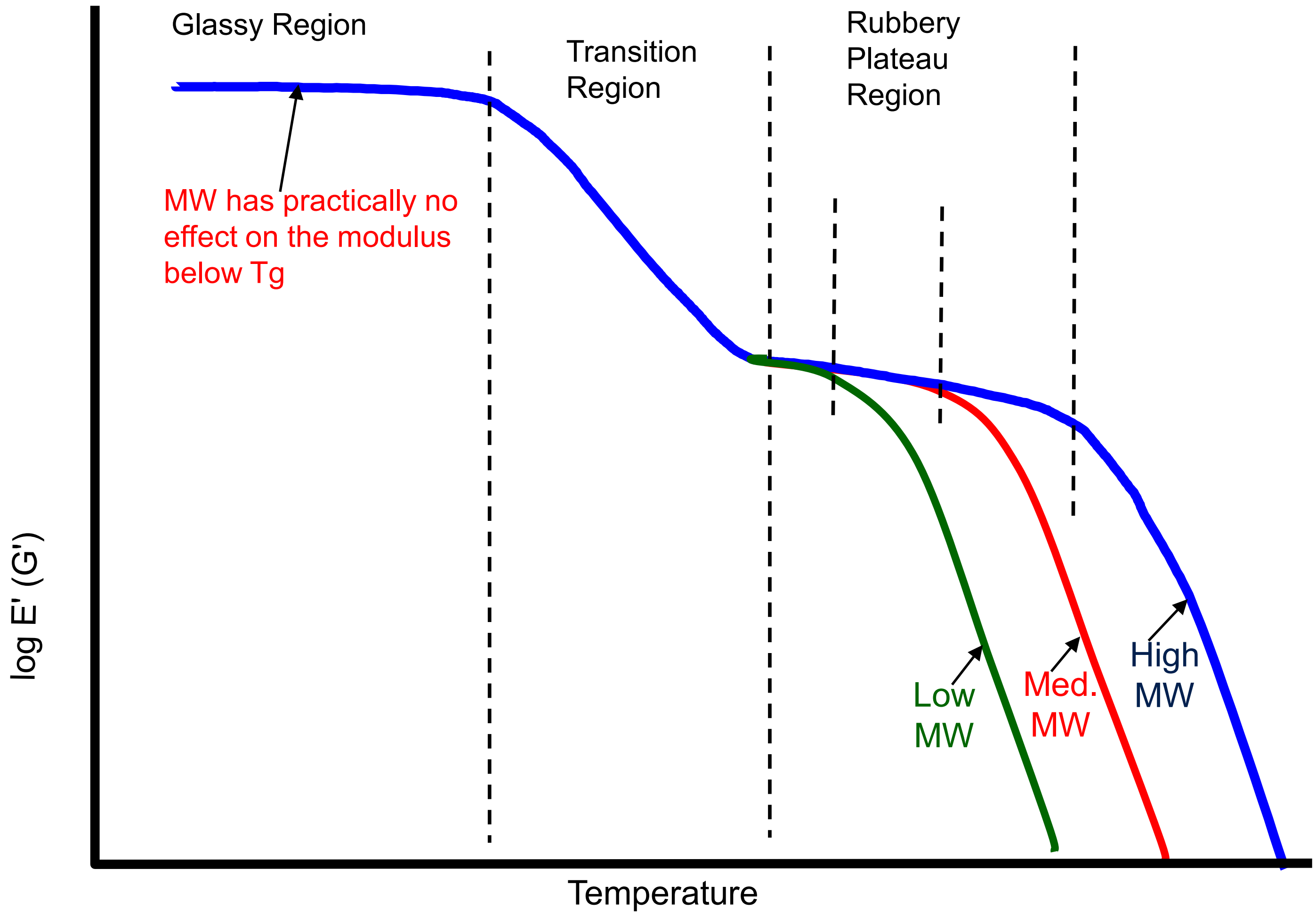
DMA Clamping Guide

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<Tg L/T>10 for T<Tg (only for T> Tg) 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

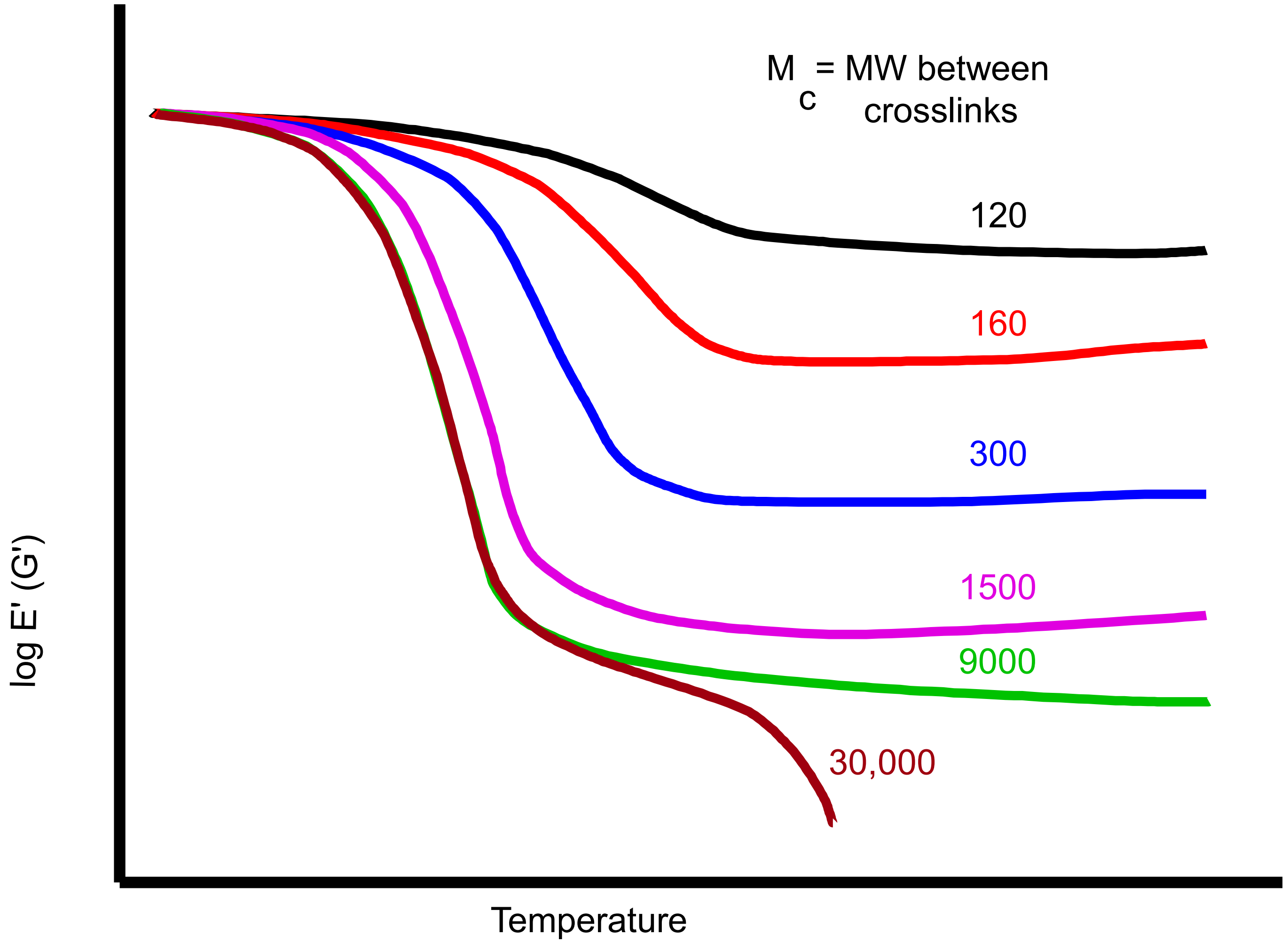
Primary and Secondary Transition in PET Film



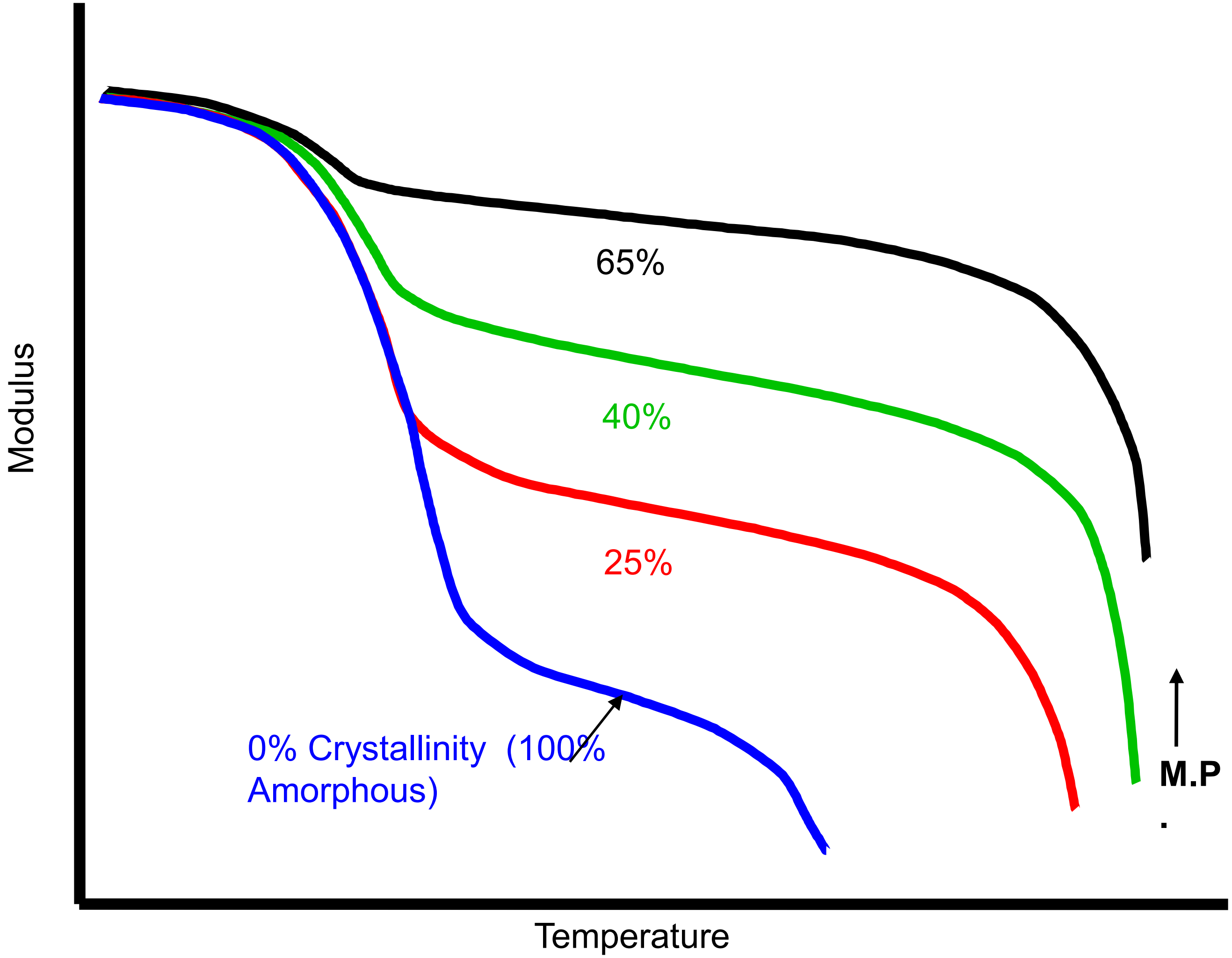
Molecular Structure - Effect of Molecular Weight



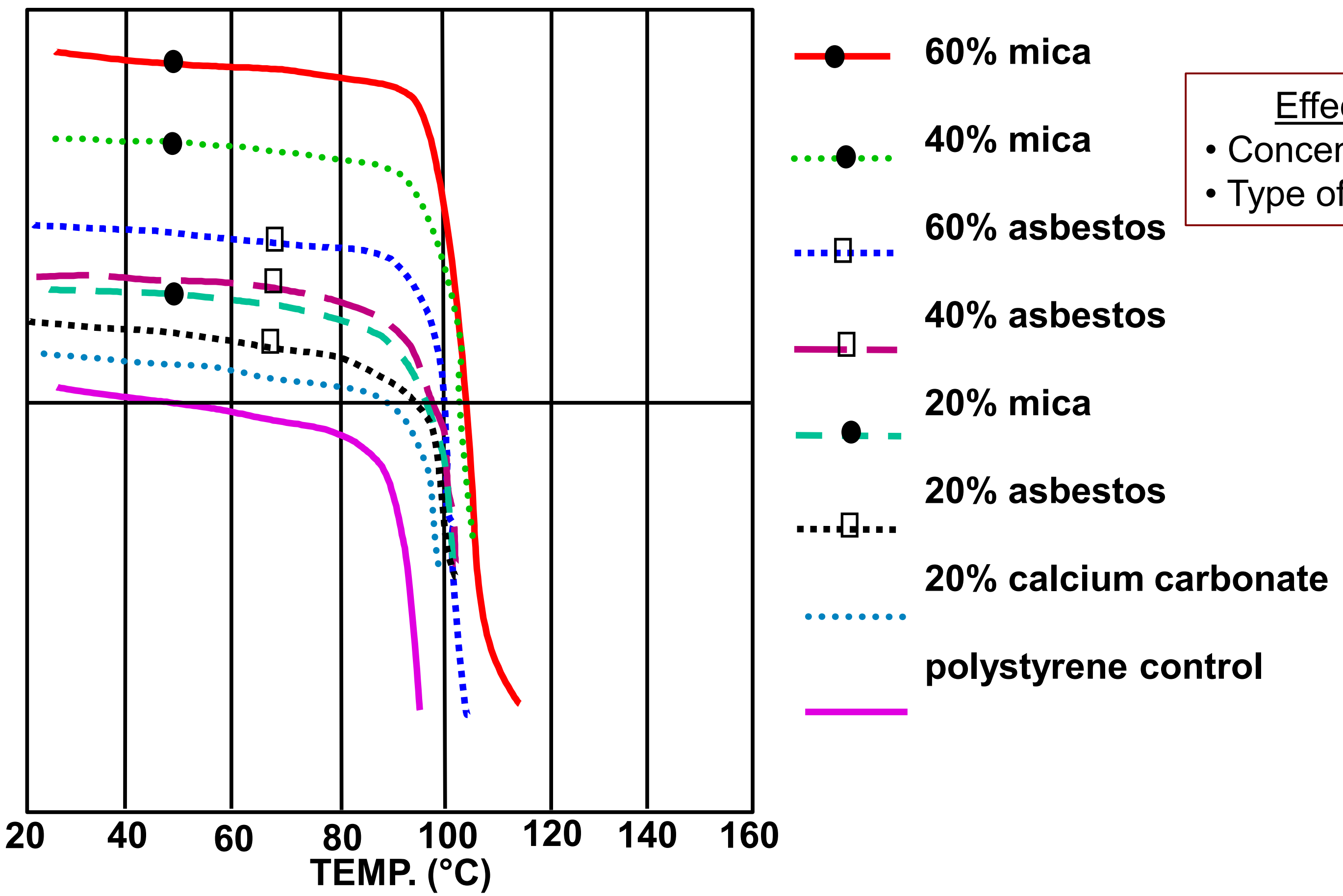
Effect of Crosslinking



Effect of Crystallinity



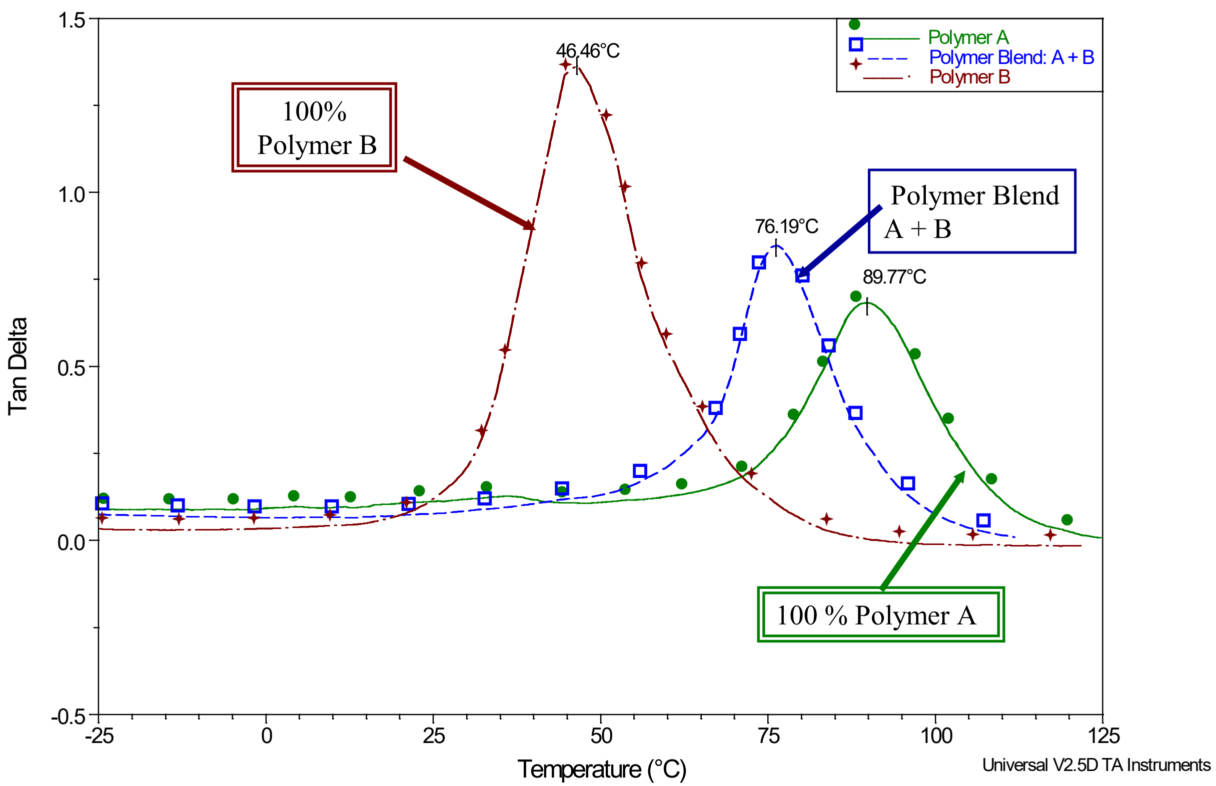
Effect of Filler on Modulus



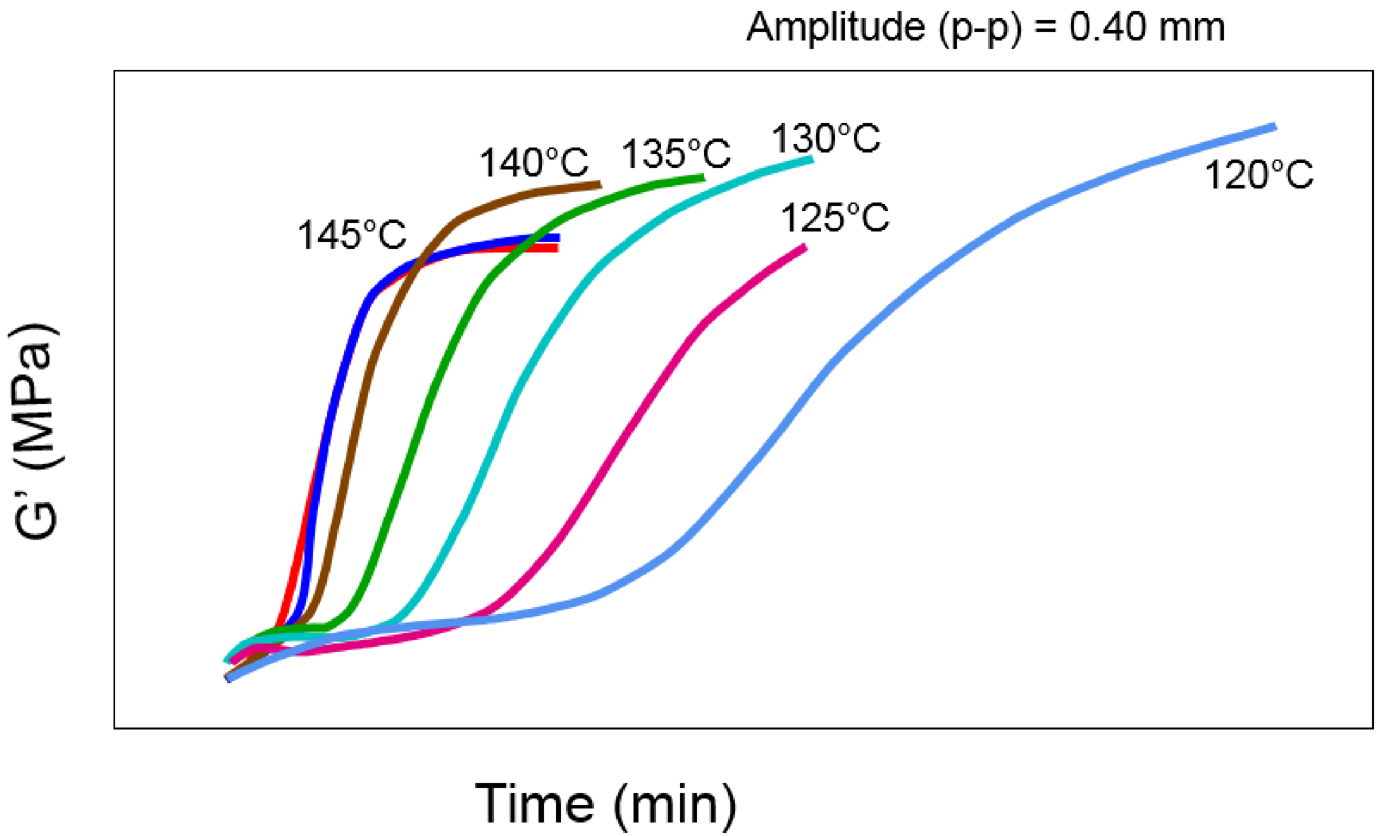
Effects
- Concentration
- Type of filler

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

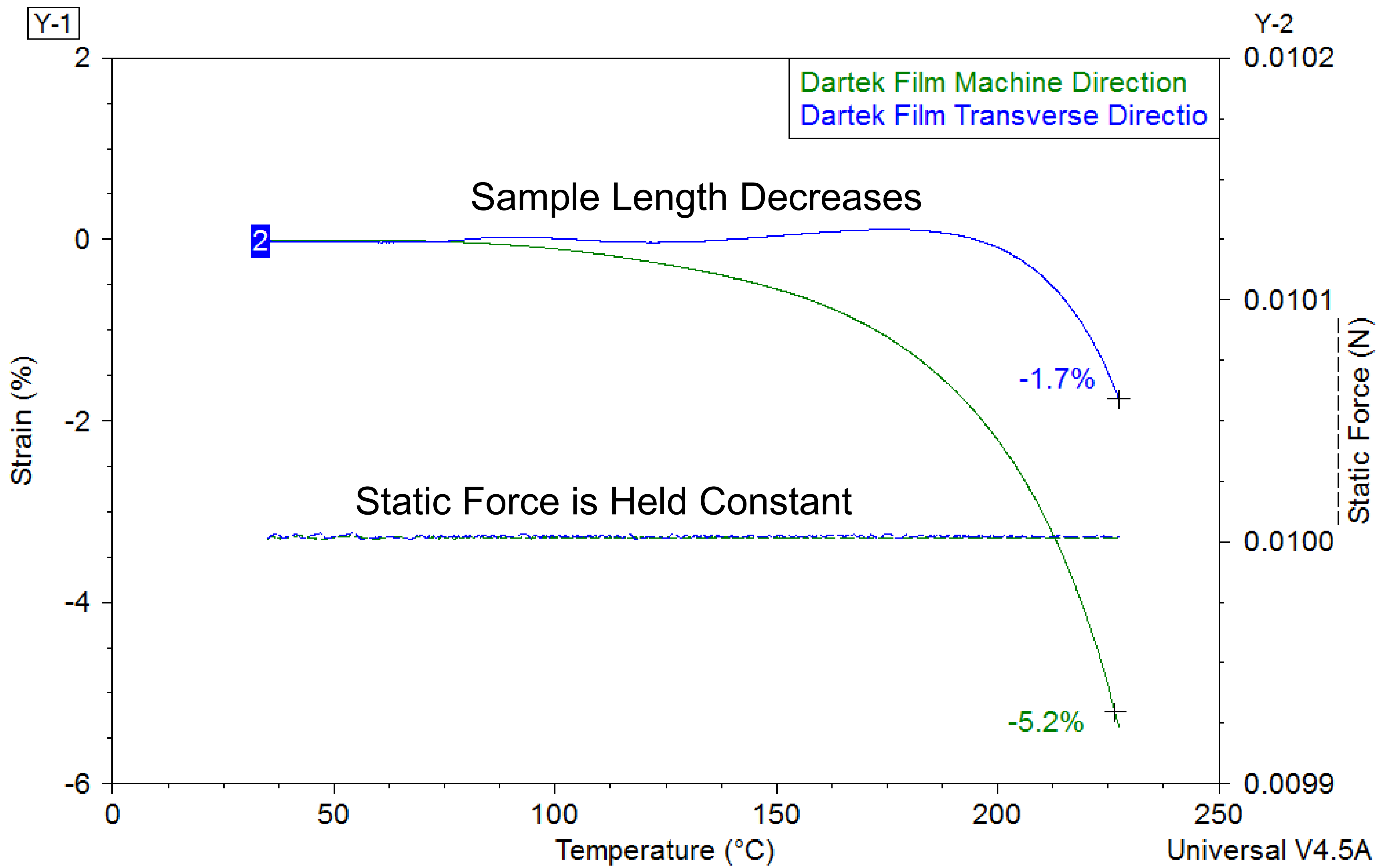
Polymer Blend - Aerospace Coating



Isothermal Cure of Tire Compound: Effect of Curing Temperature



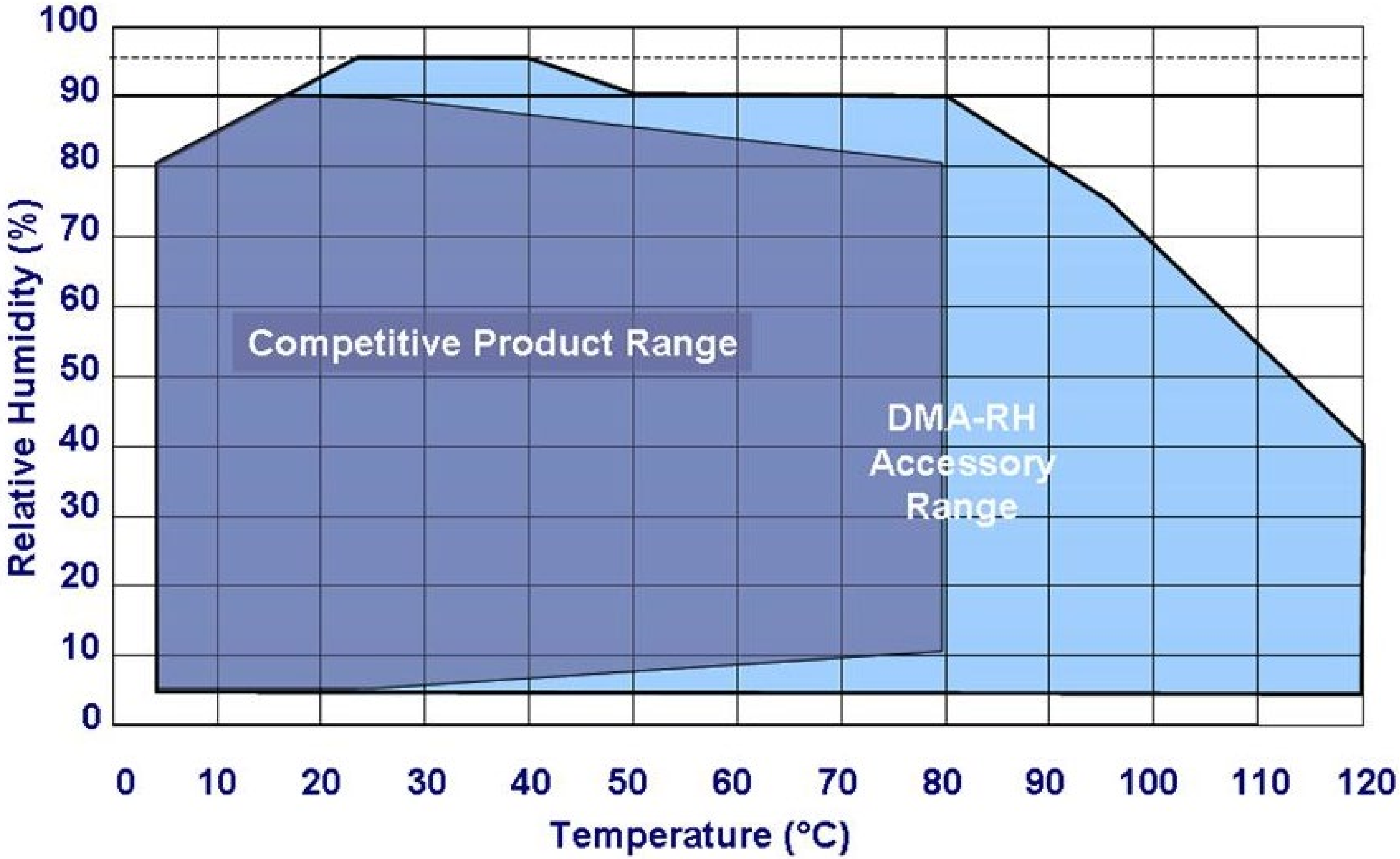
Iso-Force Temp Ramp- Shrinkage of Oriented Film



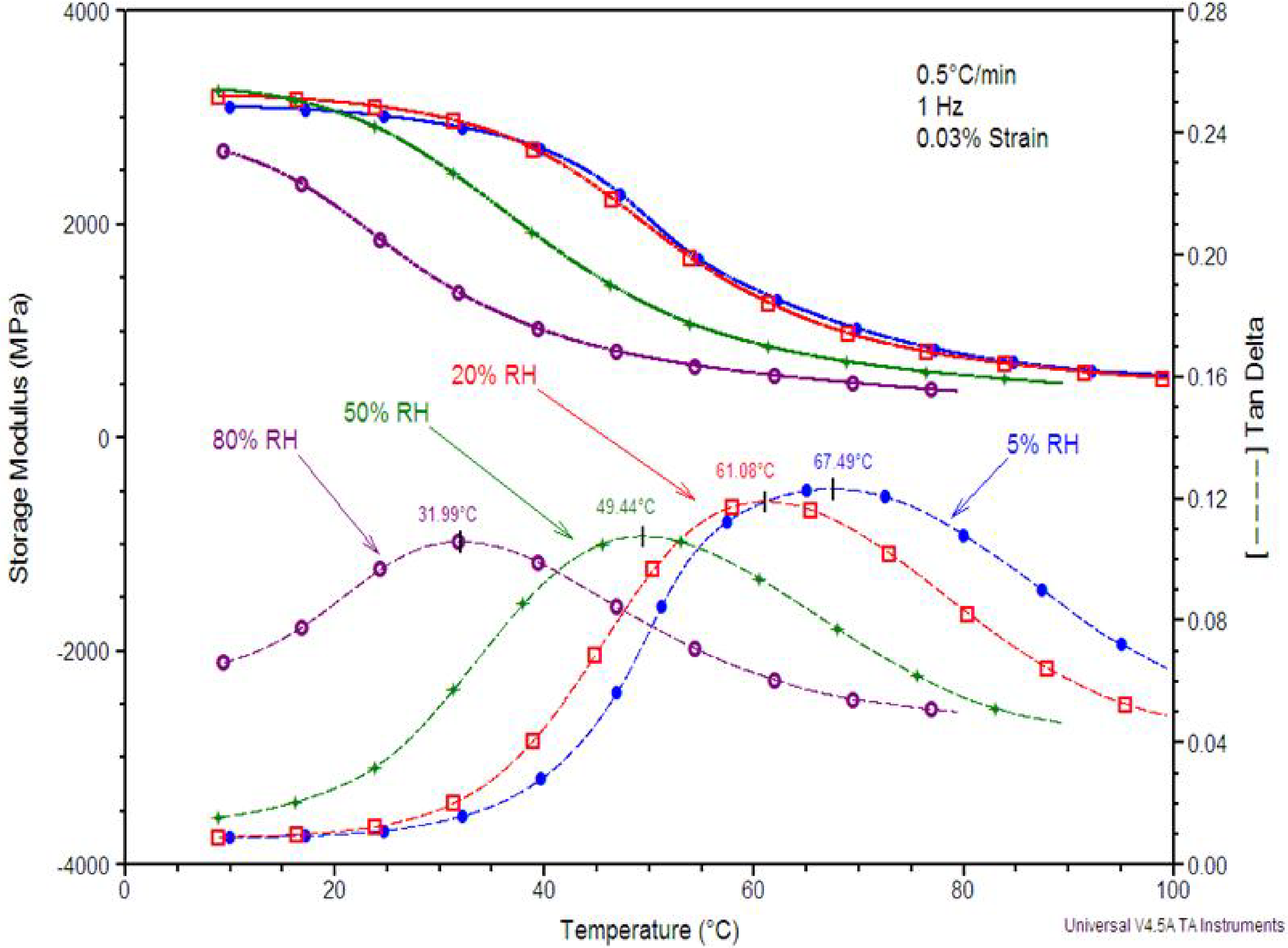
Humidity Option

This is the temperature/humidity chamber that was used to control the environment for this testing.

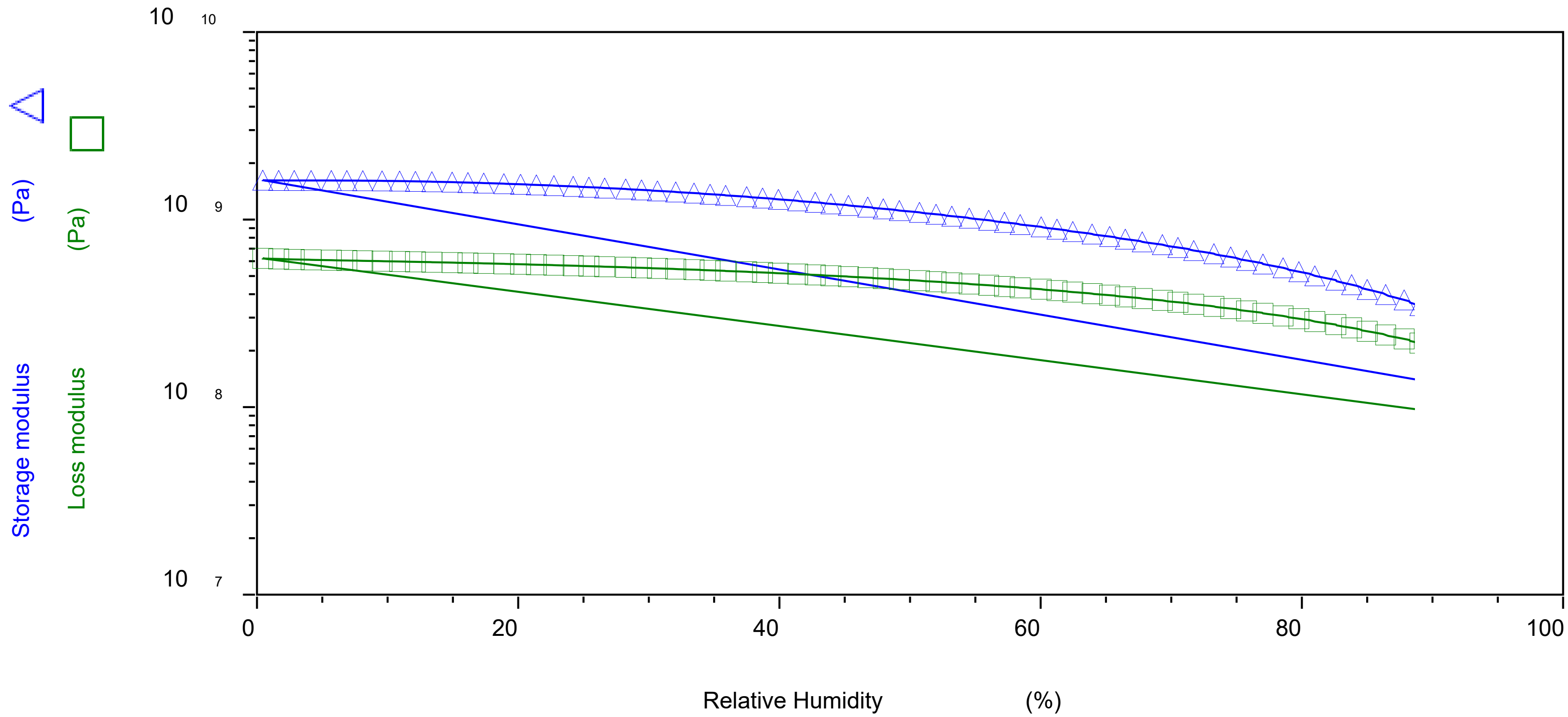
Q800: DMA-RH Operating Range



Analysis of Nylon 6: Isohume-Temperature Scans

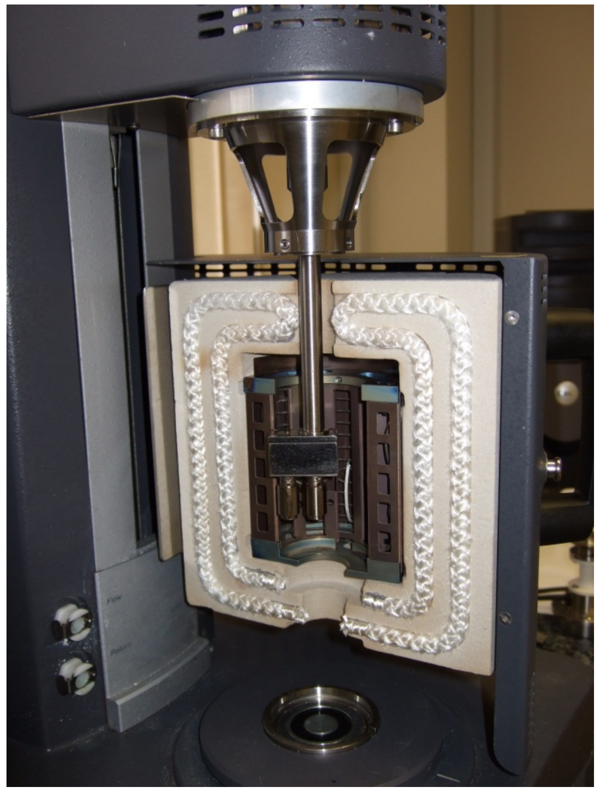


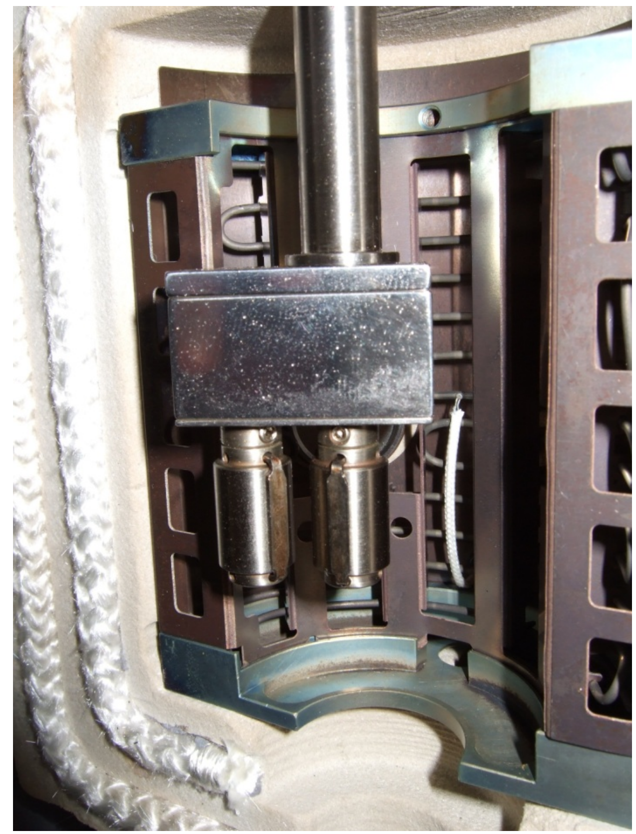
Dynamic Humidity Ramp



NEW AND MORE SPECIALIZED TESTING

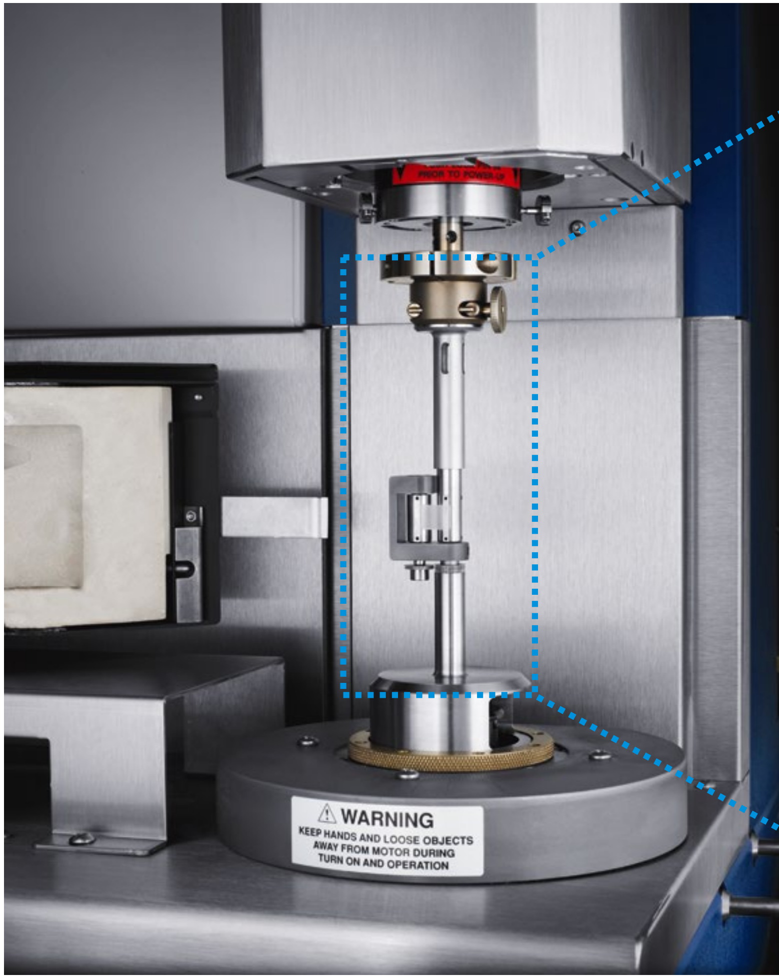
SER2 for DHR Rheometers



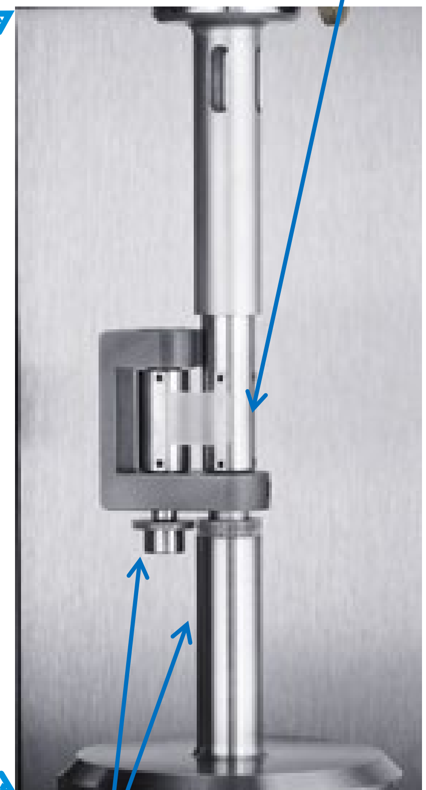


This is an interesting application of using the rotational rheometer to determine elongational viscosity

Extensional Viscosity Measurements



Fix drum connected to transducer



Rotating drum connected to the motor:

- rotates around its axis
- rotates around axis of fixed drum

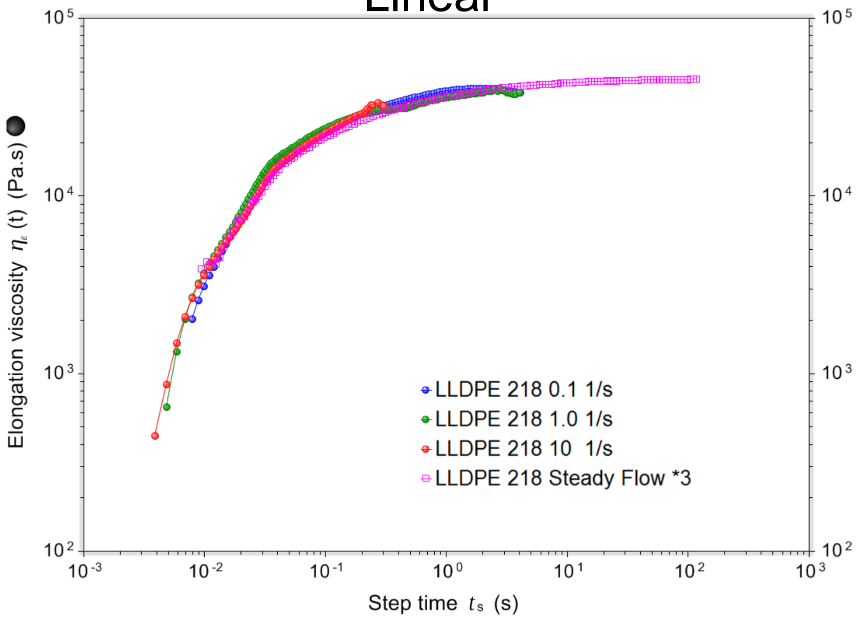
Extensional Viscosity

- Extensional rheology is very sensitive to polymer chain entanglement. Therefore it is sensitive to LCB
- The measured extensional viscosity is 3 times of steady shear viscosity

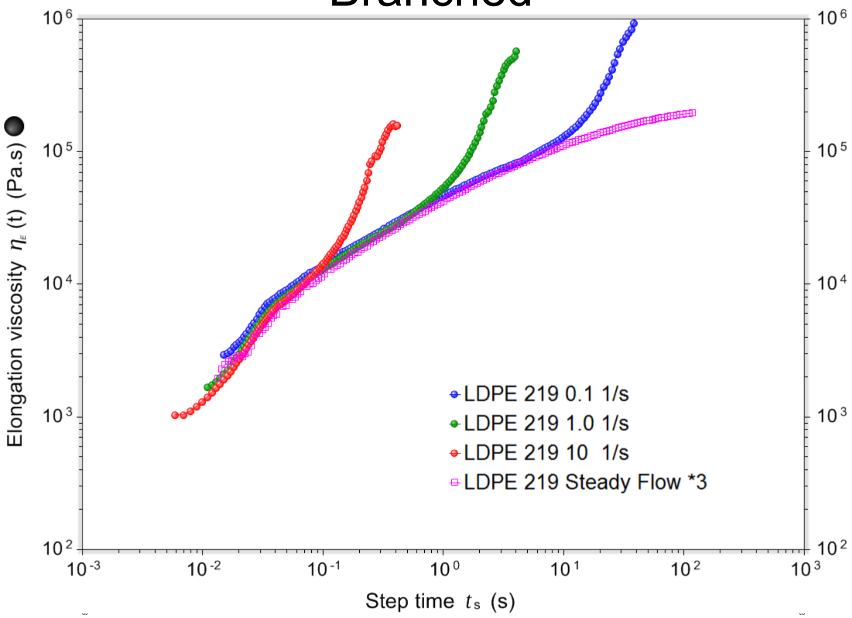
$$\eta_E = 3 \times \eta_0$$

- LCB polymer shows strain hardening effect

Linear



Branched

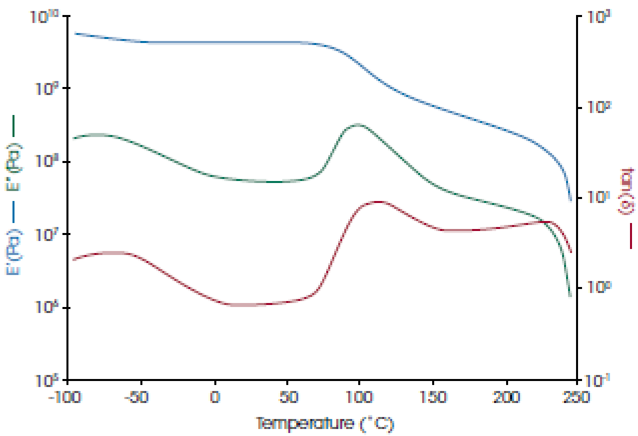


Film Tension

Compression

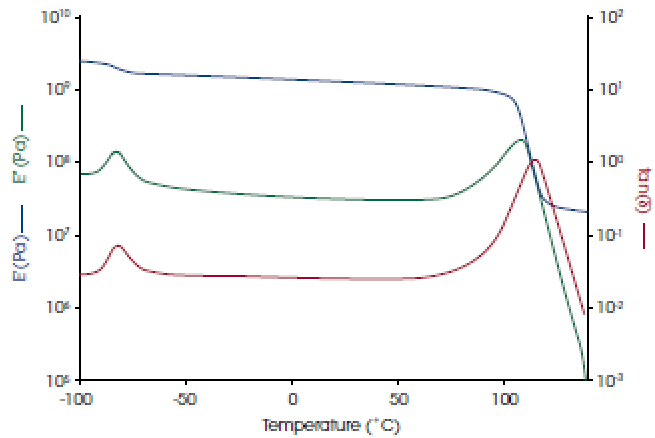
Dual Cantilever

PET Film - Tension 50 µm thick



- Polyethylene terephthalate (PET)
- Three major transitions are observed
- ß-transition: -80 °C
  α-transition (Tg): 111 °C
- Melting: 236 °C
- Reveals semi-crystalline structure with two amorphous relaxations

ABS bar - Cantilever 3 mm x 12.75 mm x 25 mm



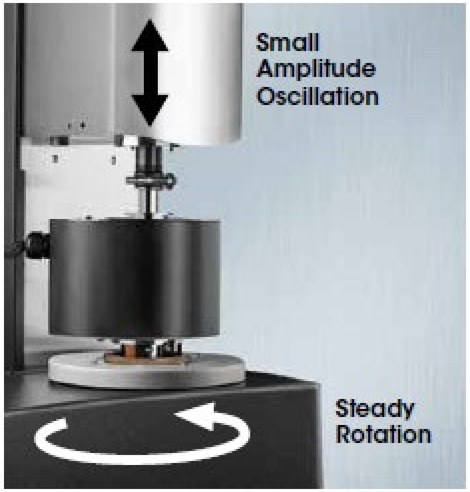
- Acrylonitrile butadiene styrene (ABS)
- Two major transitions
- Tg (butadiene): -82 °C
  Tg (styrene) 115 °C
- Indicates incompatibility of the two monomers

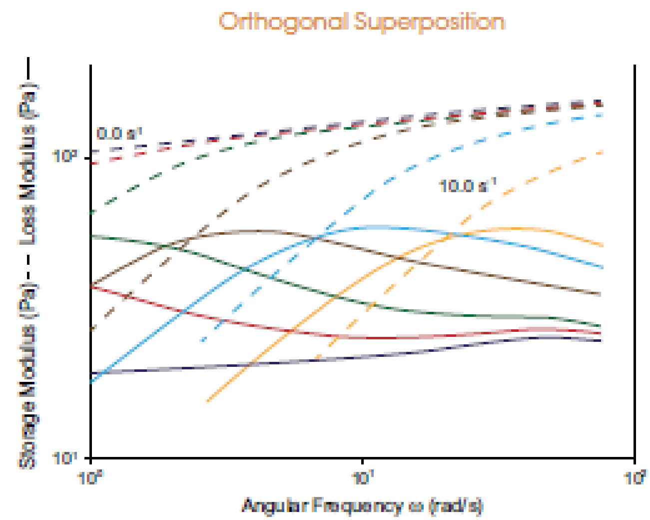
ARES-G2 DMA Mode

Features and Benefits

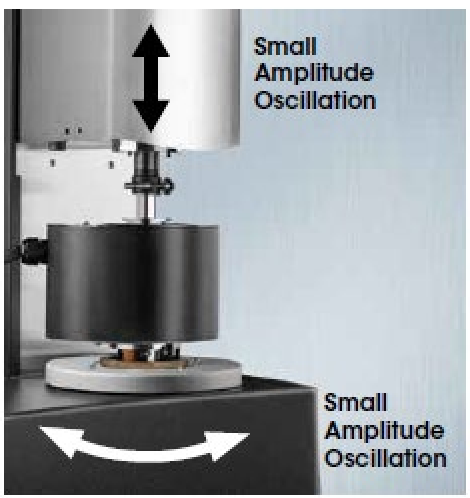
- Exclusive to the ARES-G2 rheometer
- Wide range of geometries:
  - 3-Point Bending
  - Film/Fiber Tension
  - Single and Dual Cantilever (Clamped Bending)
  - Parallel Plates Compression
- Axial Force Control tracks material stiffness and automatically adapts static load
- AutoStrain adjusts applied strain to changing sample stiffness
- Responsive FCO temperature control: -150 °C to 600 °C
- Sample visualization with FCO camera

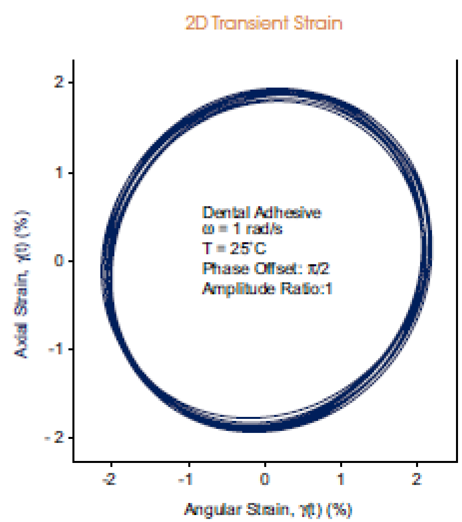
Orthogonal Superposition – 2 Modes

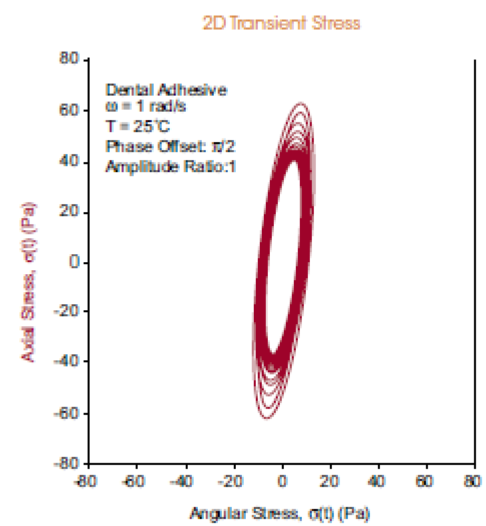




Only on ARES-G2



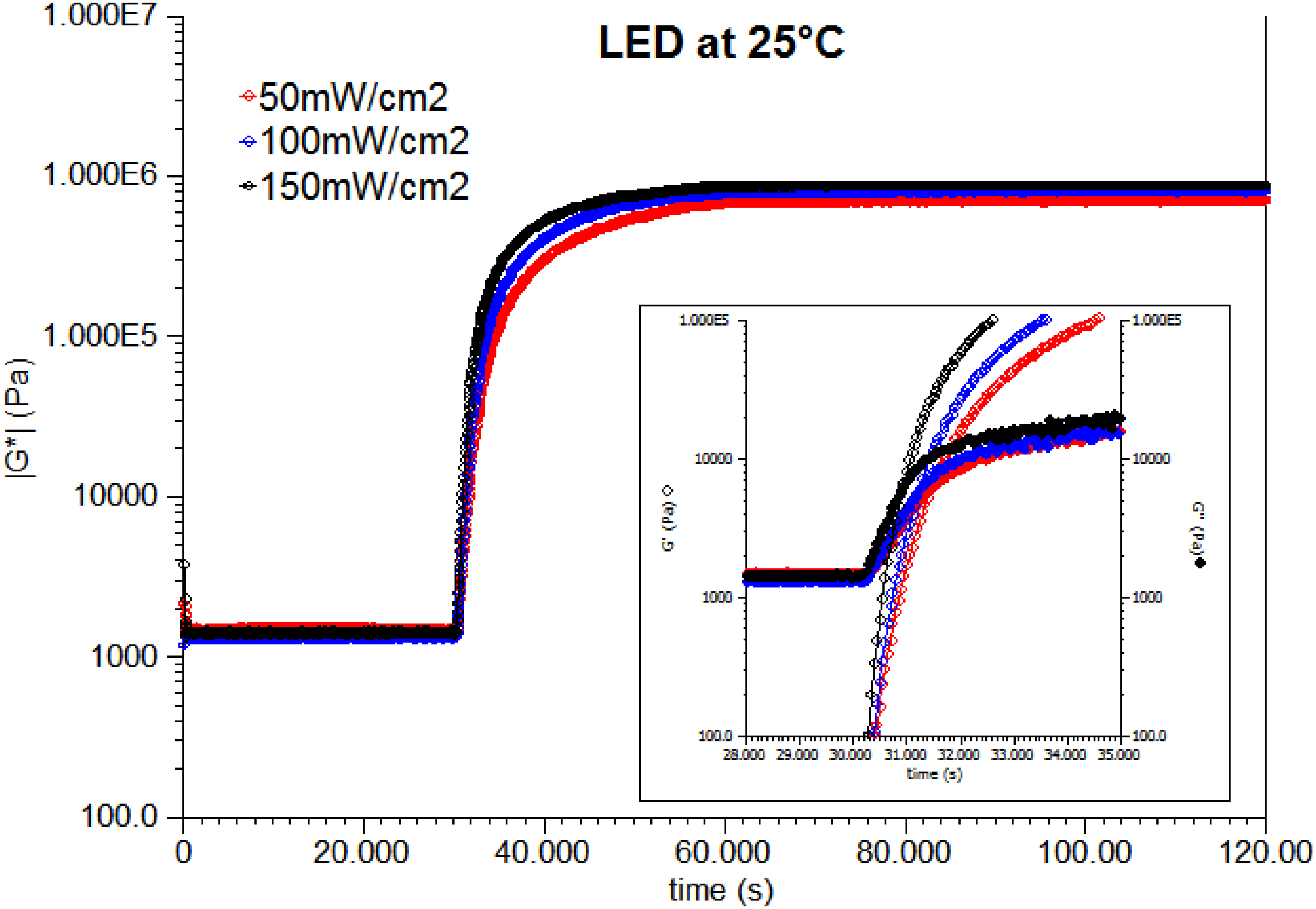




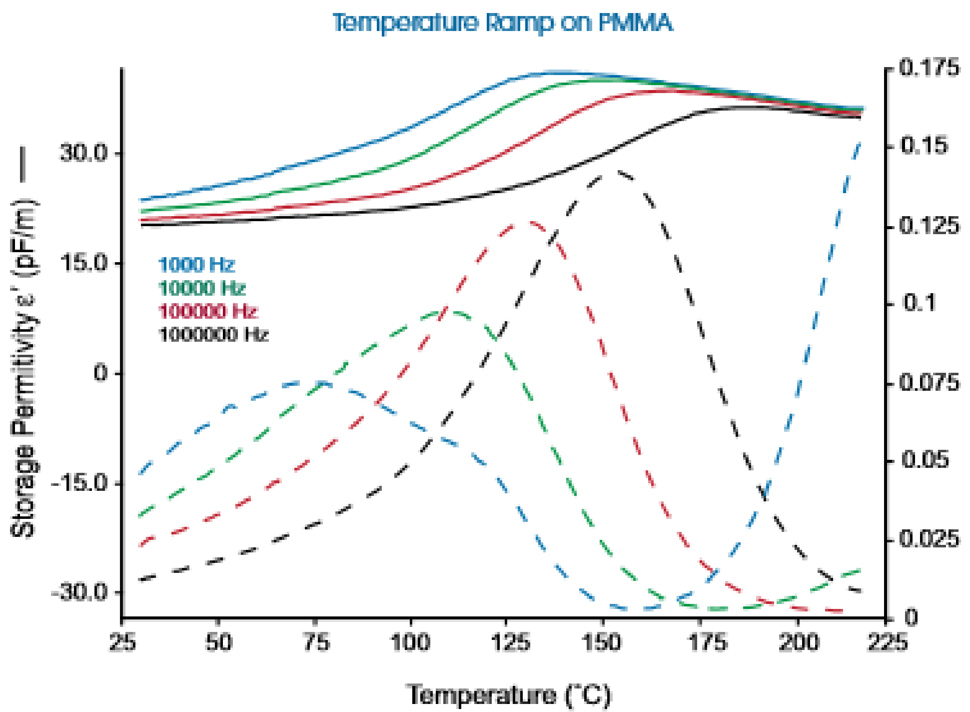
UV Light Guide Curing Accessory

- Collimated light and mirror assembly insure uniform irradiance across plate diameter
- Maximum intensity at plate 300 mW/cm^2
- Broad range spectrum with main peak at 365 nm with wavelength filtering options
- Cover with nitrogen purge ports
- Optional disposable acrylic plates

UV Cure Profile Changes with Intensity

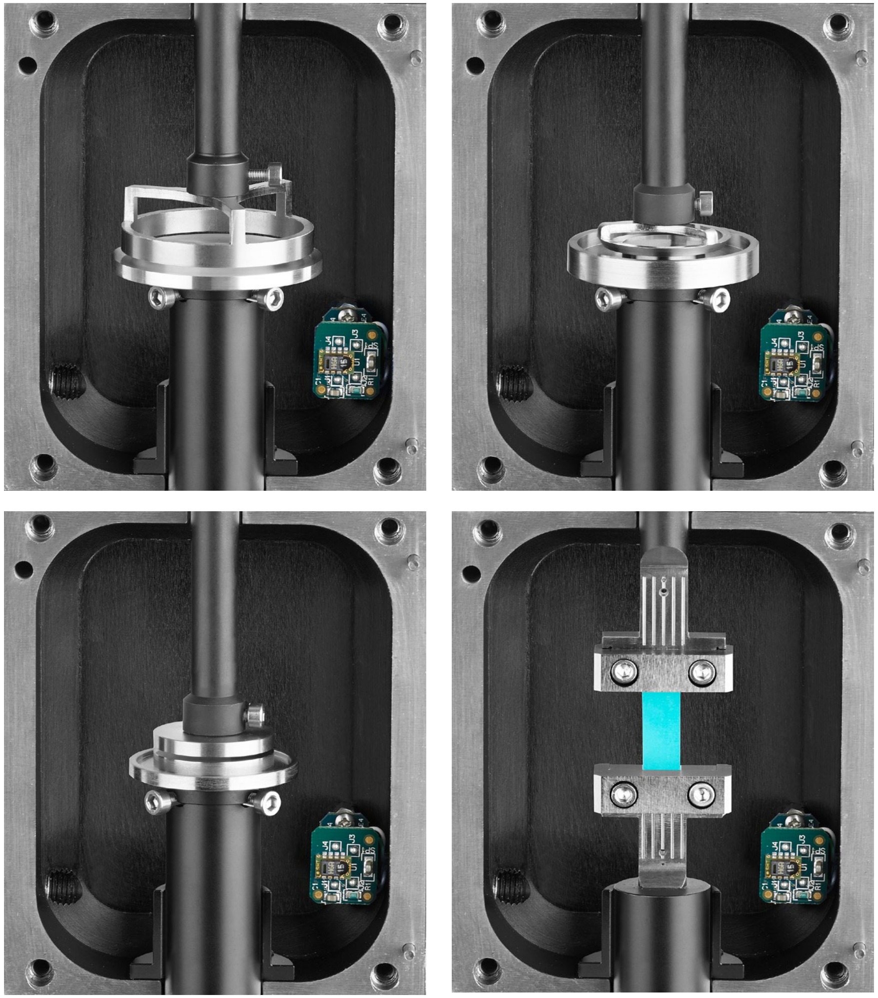


DETA on DHR and ARES-G2

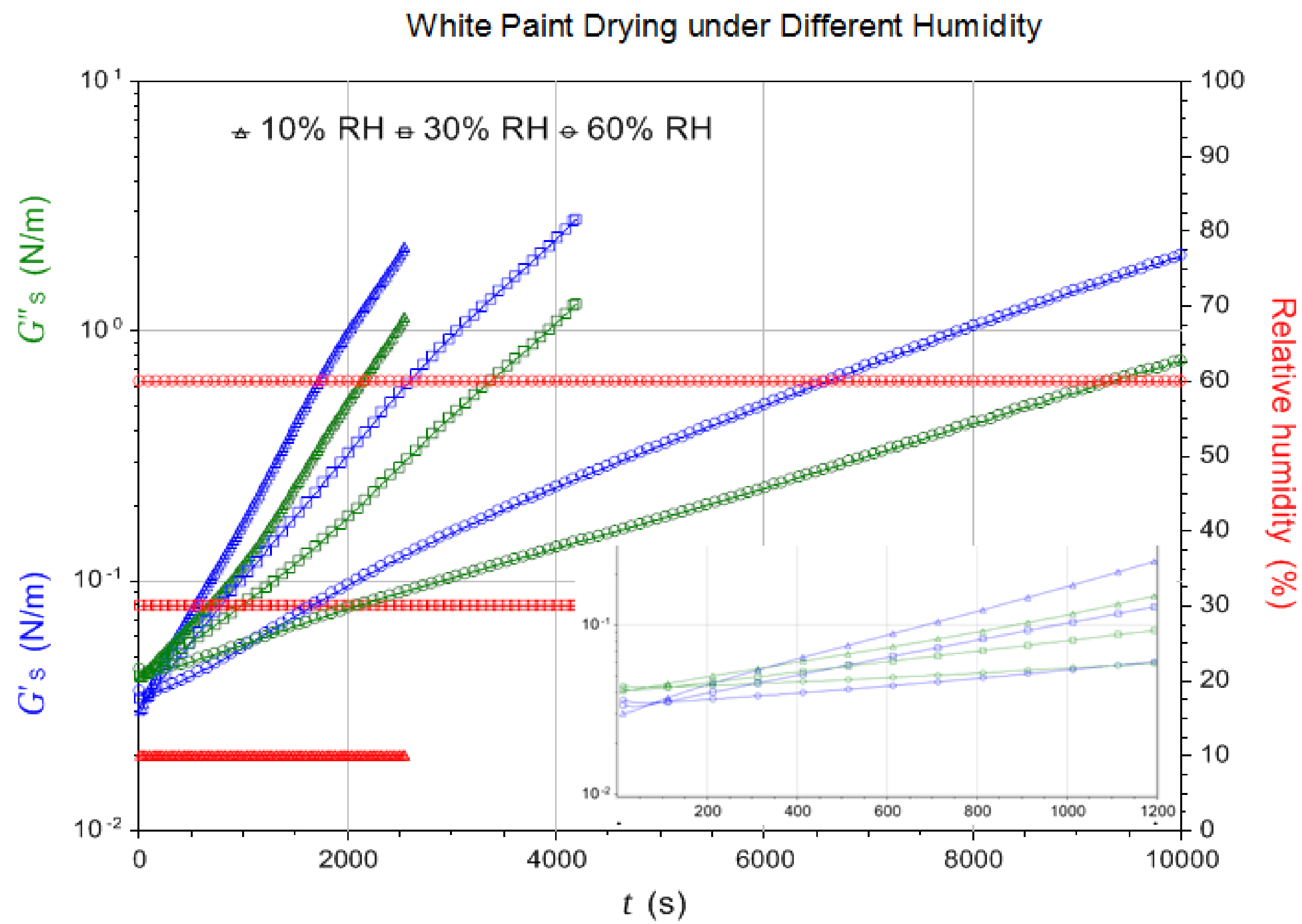


Test Geometries

- Wide variety of test geometries:
    - Standard parallel plate
    - Disposable parallel plate
    - Annular Ring
    - Surface Diffusion
    - Rectangular Torsion
- Innovative geometries for RH: true humidity-dependent rheology, not dominated by diffusion
- True Axial DMA:
    - Film Tension
    - Three-point Bending

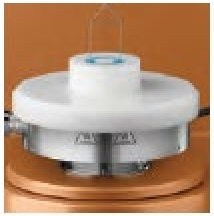


Paint Drying at Different Humidity Levels

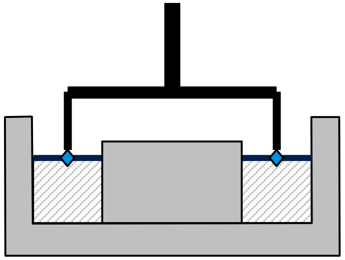


Interfacial Accessories

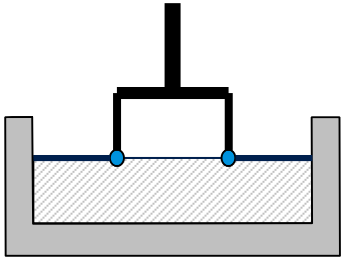
Bicone



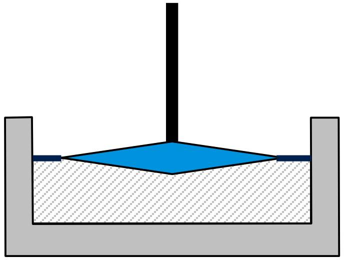
Double Wall Du Noüy Ring (DDR)



Double Wall Ring

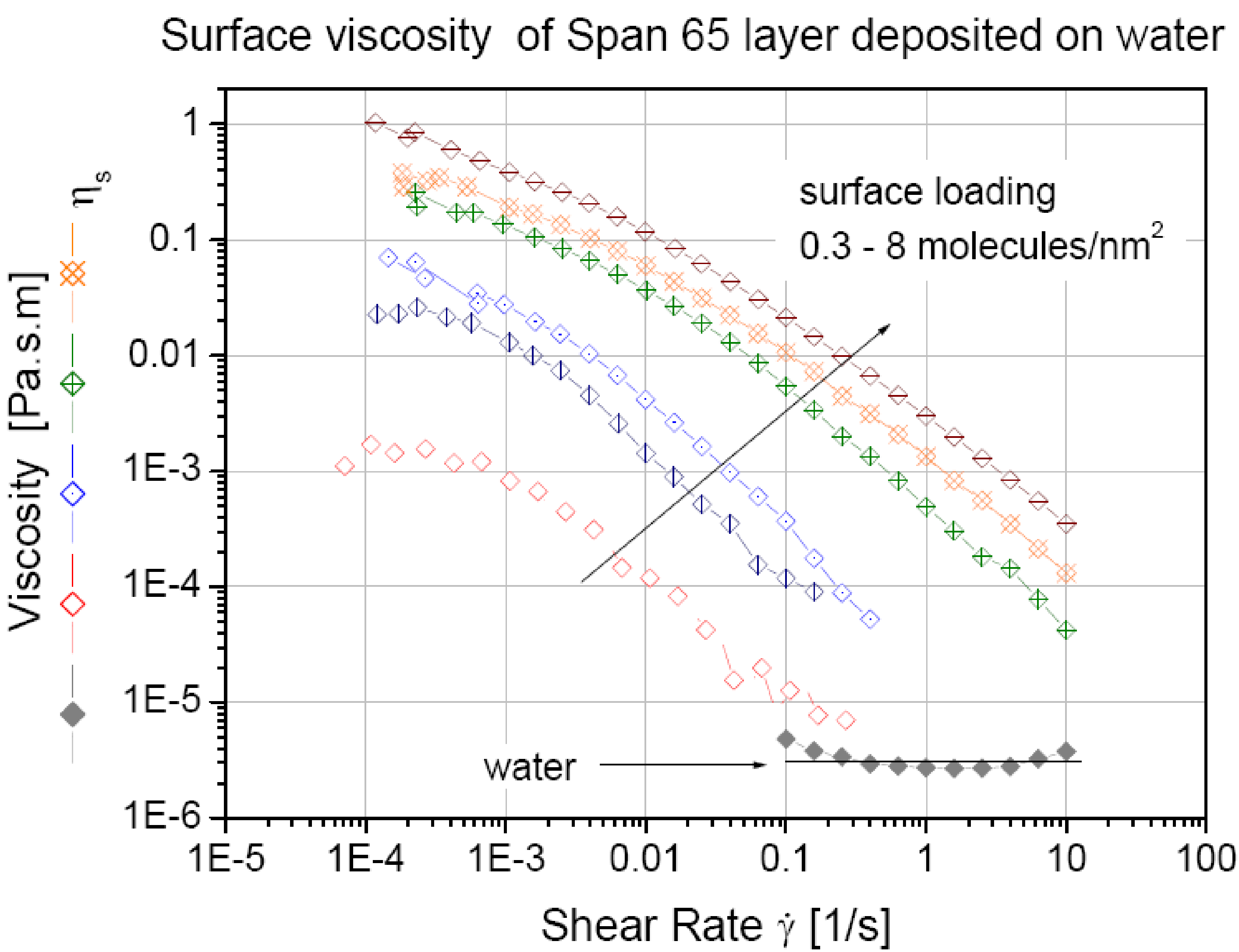


DuNouy Ring

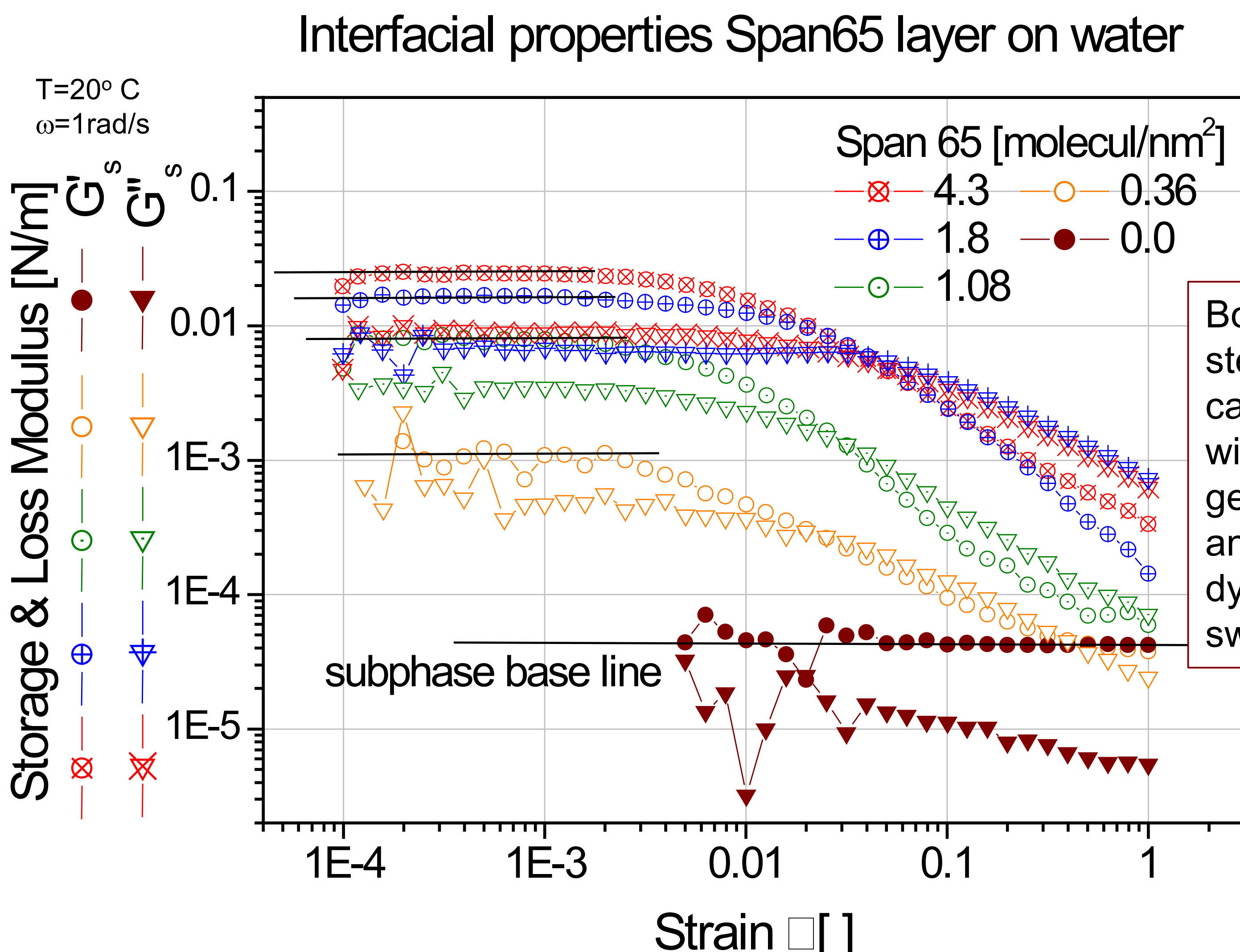


Bicone

Surface Concentration Effects on Interfacial Viscosity

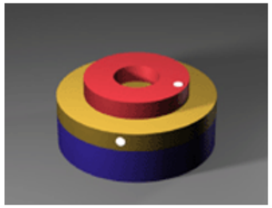


SPAN65® Layer Spread on Water



Both dynamic and steady flow tests can be performed with the interfacial geometry. This is an example of a dynamic strain sweep.

Tribo-rheometry Accessory

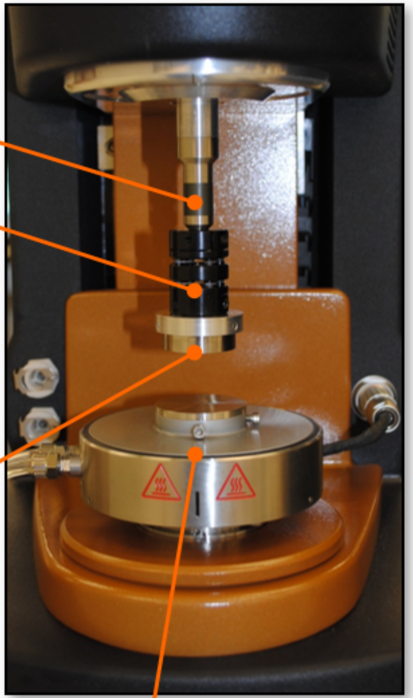


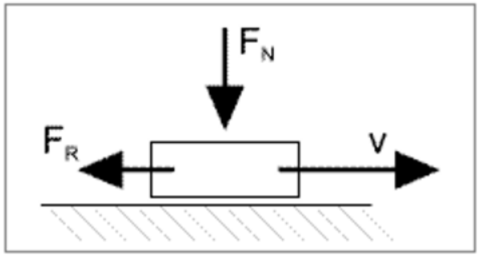
Tribology is the study of interacting surfaces in relative motion, it includes:

- Solid and liquid lubrication, lubricating oils and greases
- Friction, wear, surface damage
- Surface modifications and coatings

Requires:

- Small gaps → Alignment → Beam/Disc Coupling
- Normal Force Control → Compliance → Beam/Disc Coupling

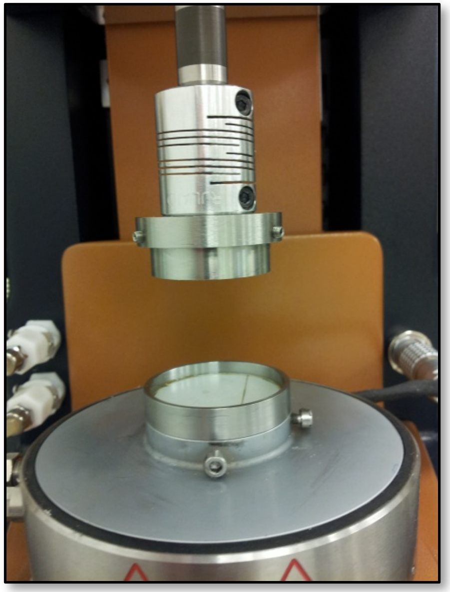




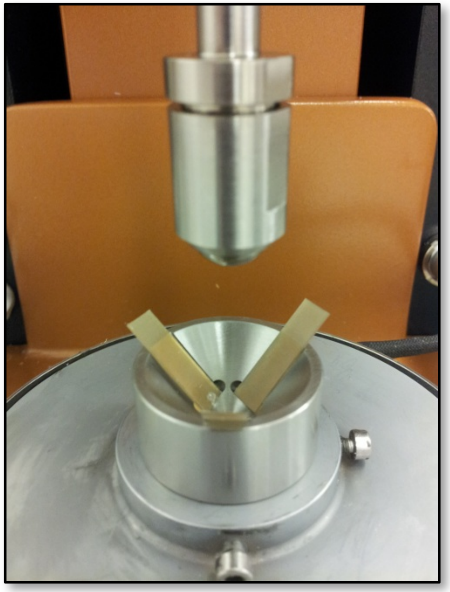
$$\mu = \frac{F_R}{F_N} = \frac{\text{shear stress}}{\text{normal stress}}$$

DHR Peltier Tribo-rheometry Accessory

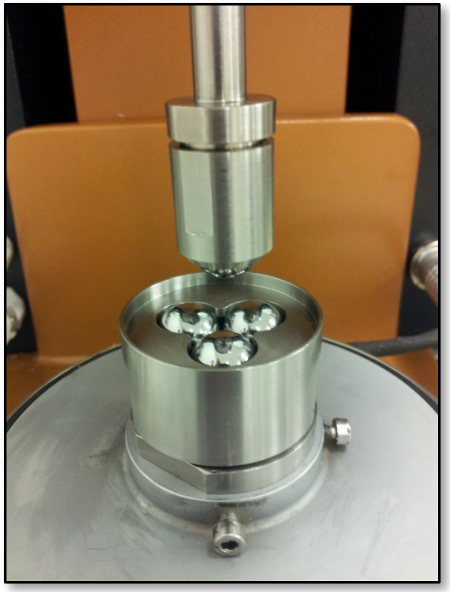
Ring on Play



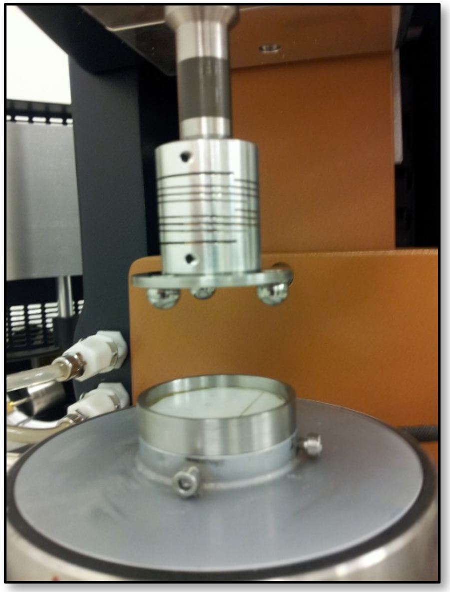
Ball on 3 Plates



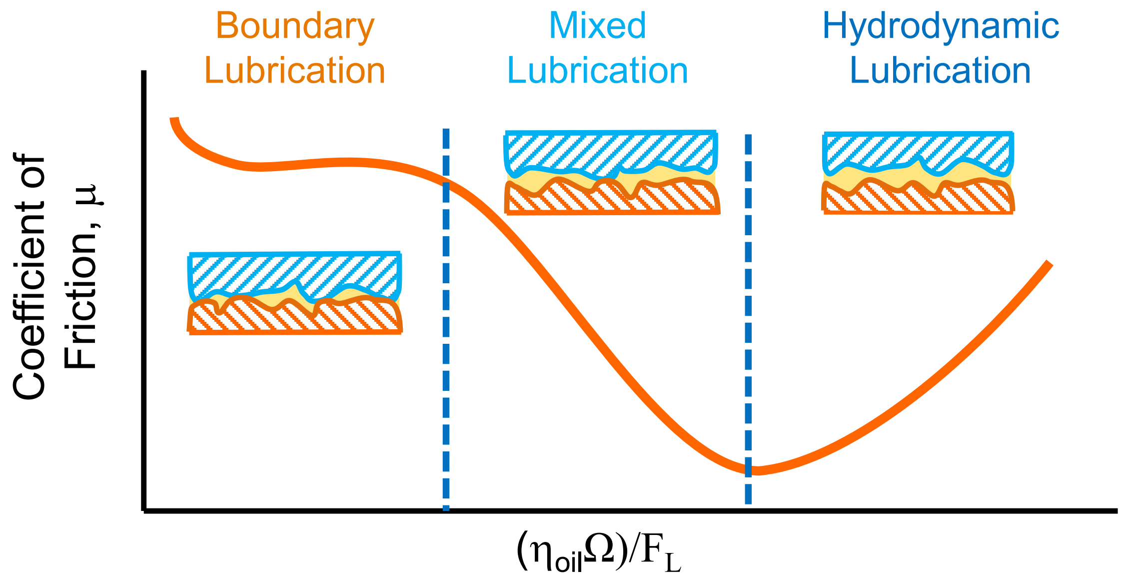
Ball on 3 Balls



3 Balls on Plate

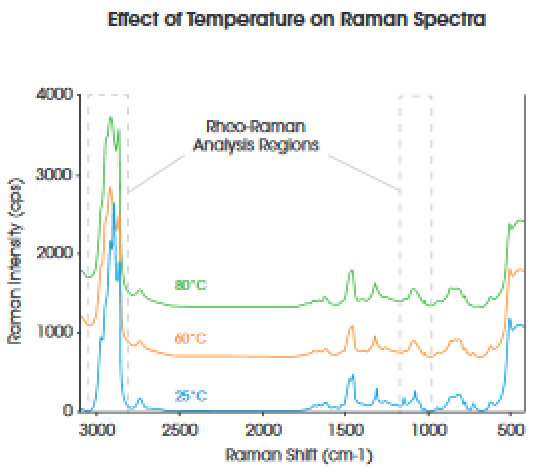


Tribology of Lubricated Systems

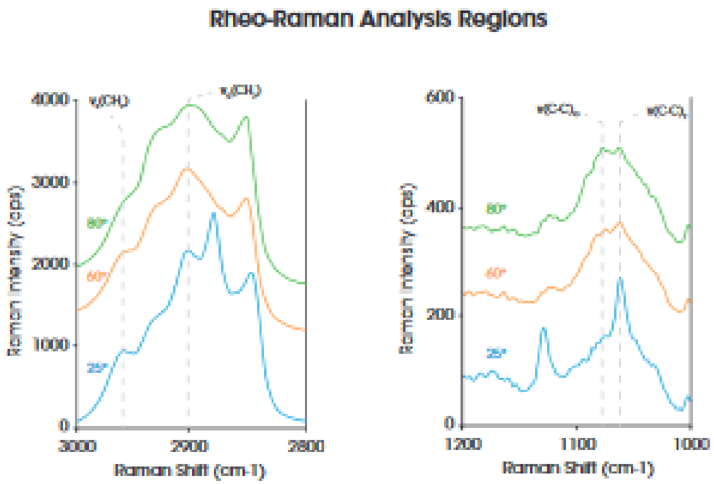


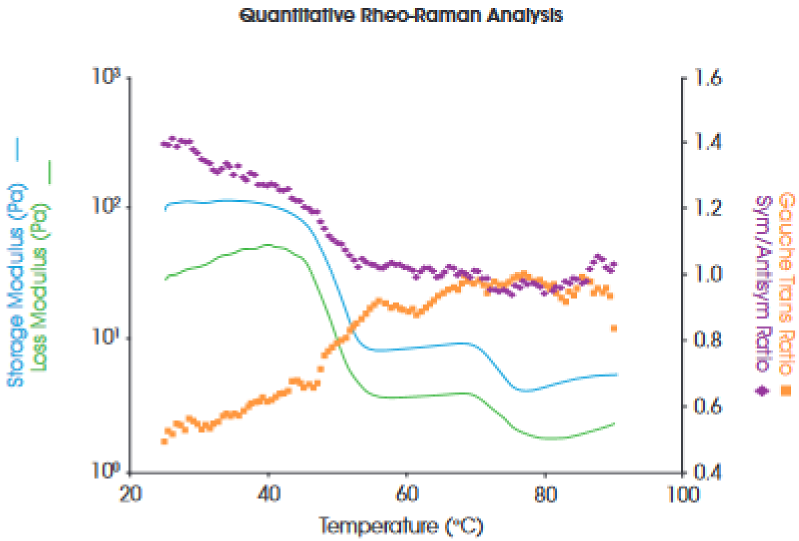
- In lubricated systems, the 'Stribeck curve' captures influence of lubricant viscosity(η_{oil}), rotational velocity (Ω) and contact load (F_L) on μ
- At low loads, the two surfaces are separated by a thin fluid film (gap, d) with frictional effects arising from fluid drag (Hydrodynamic Lubrication)
- At higher loads, the gap becomes smaller and causes friction to go up (Mixed Lubrication)
- At extremely high loads, there is direct solid-solid contact between the surface asperities leading to very high friction (Boundary Lubrication)

Rheo-Raman Accessory

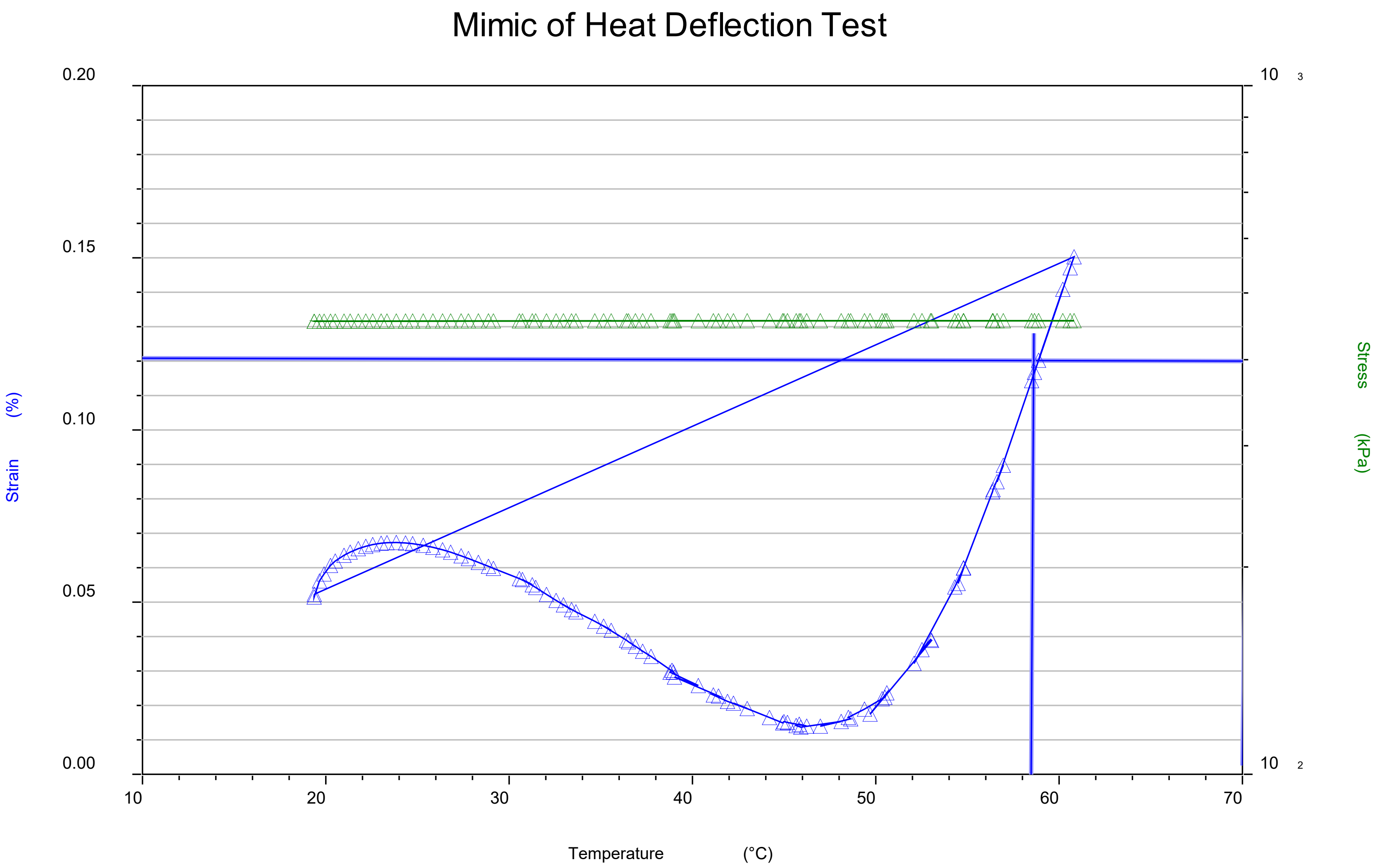


Rheo-Raman on a hand lotion





Mimic of Heat Deflection Test on the DMA



Thank You

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I hope you have enjoyed this presentation on rheology and DMA.