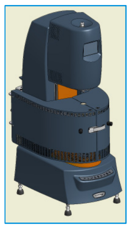
TA Instruments – Waters LLC



Materials Characterization by Rheological Methods

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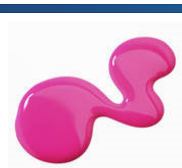
Rheology: An Introduction





Rheology: The study of the flow and deformation of matter.
Rheological behavior affects every aspect of our lives.





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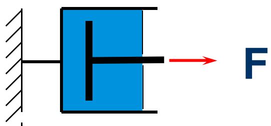
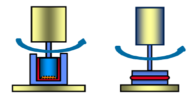
2

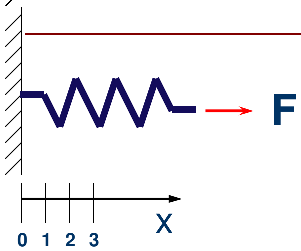
Rheology: The study of the flow and deformation of matter



Flow: Fluid Behavior; Viscous Nature

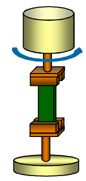
F = F(v); F ≠ F(x)

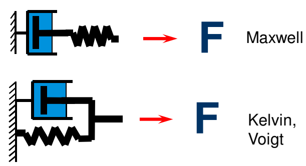




Deformation: Solid Behavior
Elastic Nature

F = F(x); F ≠ F(v)





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

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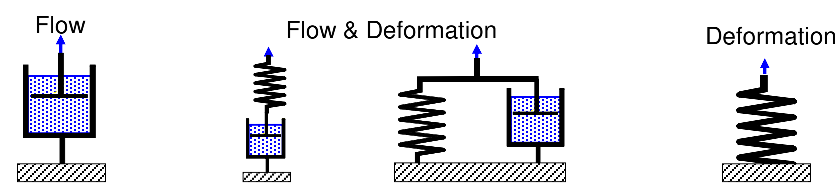
Basic Material Behaviors

Water	Oil	Soap	Egg white	Polymer Melt	Ceramic ?	Metal



Viscous Liquids | Viscoelastic | Elastic Solids

$$\text{Viscosity} = \frac{\text{Stress}}{\text{Strain Rate}}$$

$$\text{Modulus} = \frac{\text{Stress}}{\text{Strain}}$$

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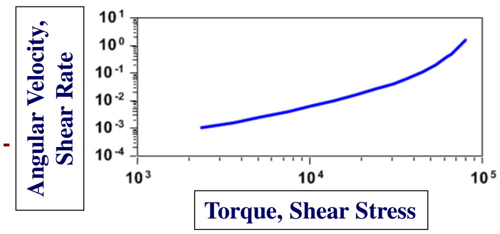
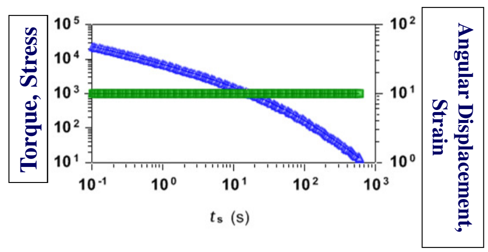
Rheological Testing - Rotational

- 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)



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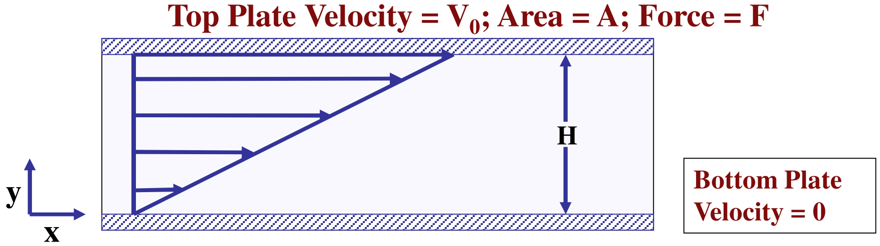
Steady Simple Shear Flow

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



$$v_x = (y/H)*V_0$$

$\dot{\gamma} = dv_x/dy = V_0/H$ 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.

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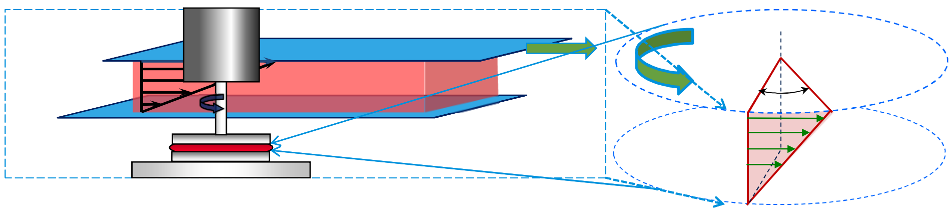
Parallel Plate Shear Flow



Stress	σ	Force or torque
Strain	γ	Linear or angular displacement
Strain Rate	$\dot{\gamma}$	Linear or angular velocity

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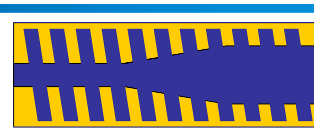
SHEAR RATE CALCULATIONS IN EXTRUSION

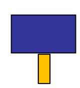
Extruder: $\dot{\gamma} = \pi DN/(60*h)$

D = Diameter; N = rpm; h = gap



Circular, Rod: $\dot{\gamma} = 32Q/(\pi D^3)$

compounding, cable



Q = Output rate;
D = orifice diameter

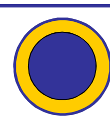
Rectangular, Slit: $\dot{\gamma} = 6Q/(wh^2)$

cast film, sheet

Q = Output rate;
w = width
h = gap

Annulus: $\dot{\gamma} = 12Q/(\pi(D1+D2)h^2)$

blown film, blow molding



Q = Output rate;
D1 = Inner diameter
D2 = Outer diameter
h = gap

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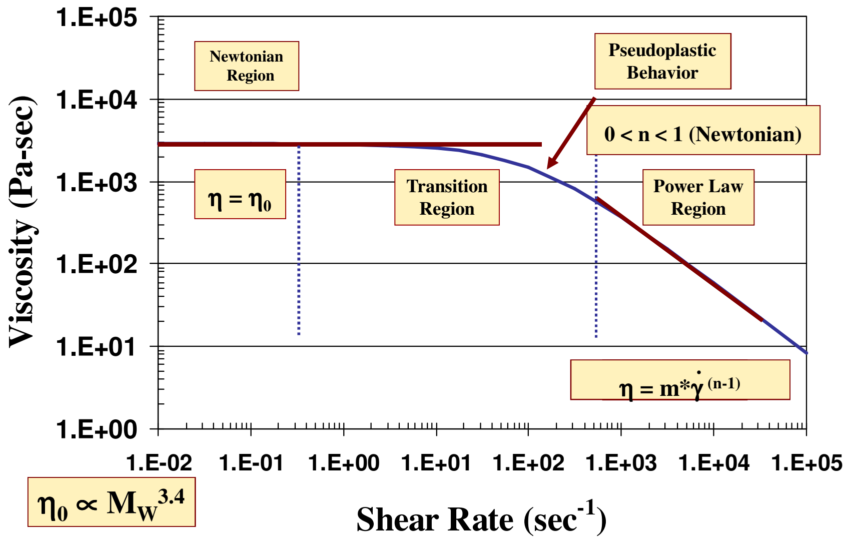
Representative Flow Curve



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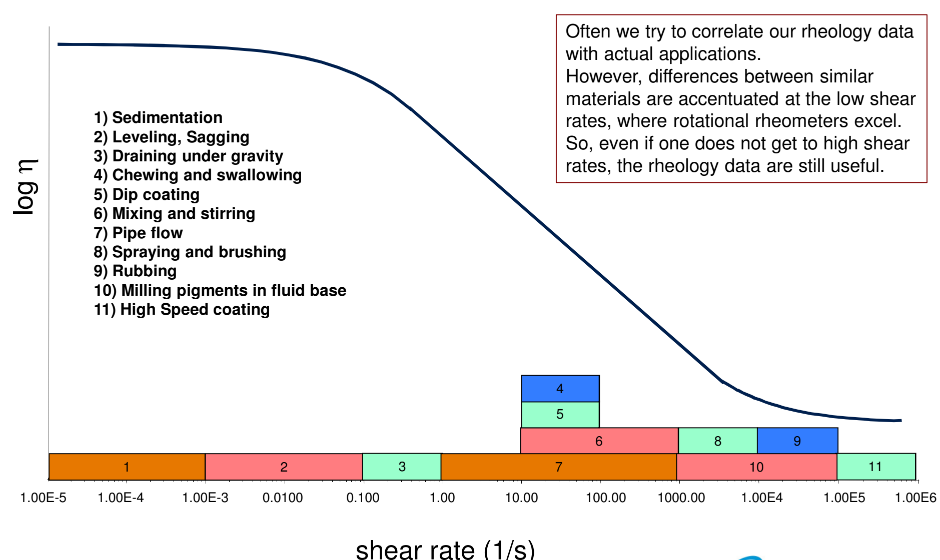
Idealized Flow Curve



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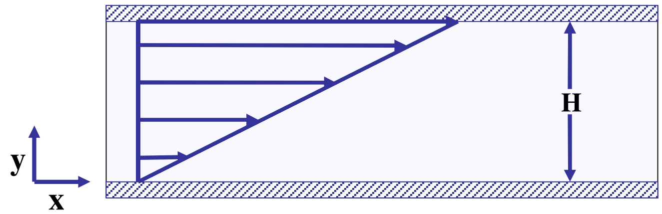
Simple Shear Deformation

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.

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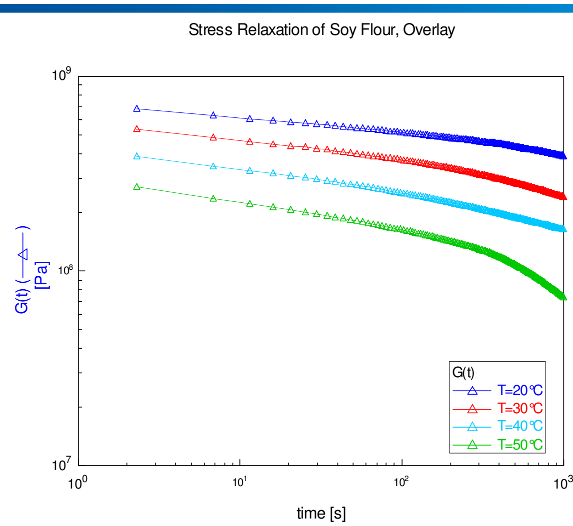
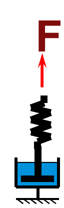
Stress Relaxation



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



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Rheological Parameters

FLUIDS TESTING

Parameter	Shear	Elongation	Units
Rate	$\dot{\gamma}$	$\dot{\varepsilon}$	$Seconds^{-1}$
Stress	σ	τ	Pascals
Viscosity	$\eta = \sigma/\dot{\gamma}$	$\eta_E = \tau/\dot{\varepsilon}$	Pascal-seconds

SOLIDS TESTING

Parameter	Shear	Elongation	Units
Strain	γ	ε	Unitless
Stress	σ	τ	Pascals
Modulus	$G(t) = \sigma/\gamma$	$E = \tau/\varepsilon$	Pascals

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Rheological Parameters

CREEP TESTING

Parameter	Shear	Elongation	Units
Stress	σ	τ	Pascals
Strain	γ	ε	Unitless
Compliance	$J = \gamma/\sigma$	$D = \varepsilon/\tau$	1/Pascals

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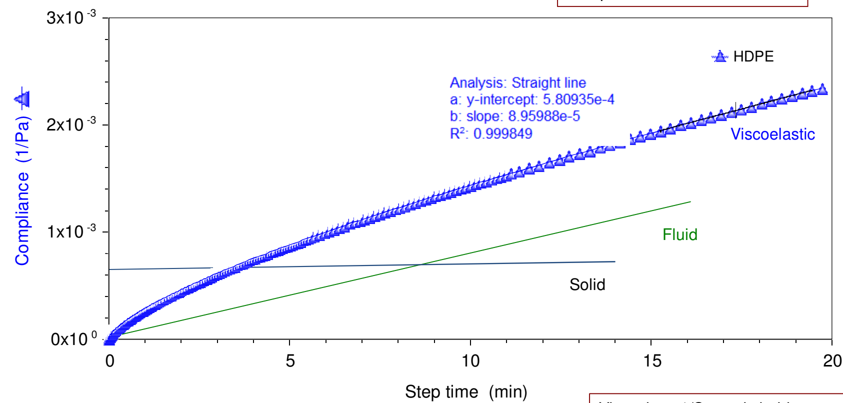
Creep Testing

Stress is held constant.
Strain is the measured variable.
Compliance = Strain/Stress



Viscosity = 1/Stress is held constant.
Strain is Slope in steady flow regime.
Intercept = non-recoverable Compliance

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GEOMETRIES

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Geometry Options

Concentric Cylinders	Cone and Plate	Parallel Plate	Torsion Rectangular
			
Very Low to Medium Viscosity	Very Low to High Viscosity	Very Low Viscosity to Soft Solids	Solids

Water → to → Steel

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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.

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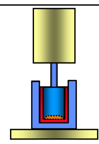
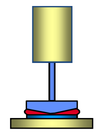
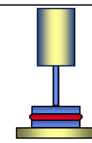
Shear Rate and Shear Stress Calculations

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

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Choosing a Geometry Size

- Assess the 'viscosity' of your sample
- When a variety of cones and plates are available, select diameter appropriate for viscosity of sample
  - Low viscosity (milk) - 60mm geometry
  - Medium viscosity (honey) - 40mm geometry
  - High viscosity (caramel) – 20 or 25mm geometry
- Examine data in terms of absolute instrument variables torque/displacement/speed and modify geometry choice to move into optimum working range
- You may need to reconsider your selection after the first run!

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Geometry Options

- With the variety of cones, plates, cups and rotors available, select a geometry based on desired experimental parameters and the material properties

Cones and Plates

No Solvent Trap (NST) | With Solvent Trap | With Solvent Trap and Heat Break (HB)

Smooth, Sandblasted, and Cross hatched

Concentric Cylinders (or Cups) and Rotors (or Bobs)

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When to use Cone and Plate

- Very Low to High Viscosity Liquids
- High Shear Rate measurements
- Normal Stress Growth
- Unfilled Samples
- Isothermal Tests
- Small Sample Volume

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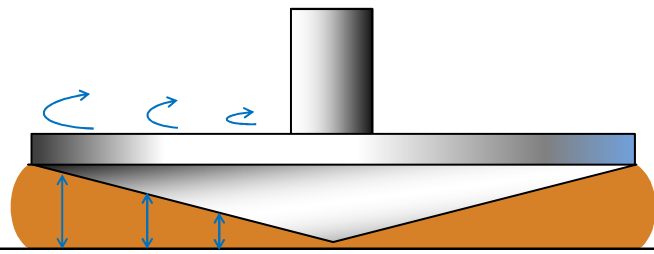
Shear Rate is Normalized across a Cone

- The cone shape produces a smaller gap height closer to the inside, so the shear on the sample is constant



$$\gamma = \frac{dx}{h}$$

h increases proportionally to *dx*, γ is uniform

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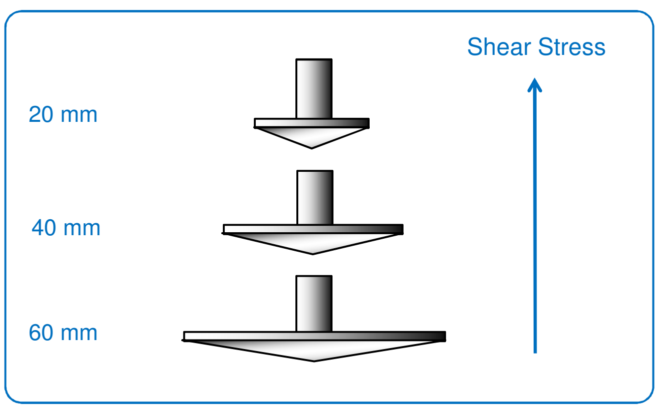
Cone Diameters



As diameter decreases, shear stress increases $\sigma = M\frac{3}{2\pi r^3}$

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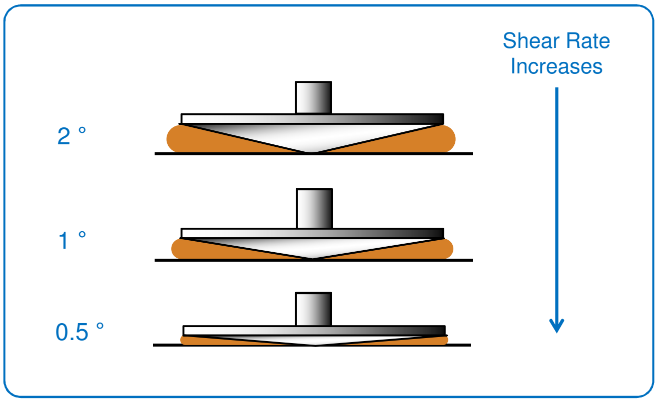
Cone Angles



As cone angle decreases, shear rate increases $\dot{\gamma} = \Omega \frac{1}{\beta}$

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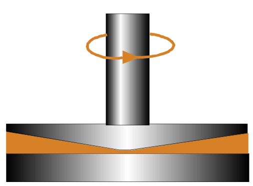
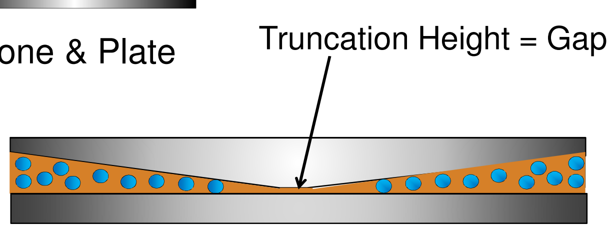
Limitations of Cone and Plate



Cone & Plate

Typical Truncation Heights:
- 1° degree ~ 20 - 30 microns
- 2° degrees ~ 60 microns
- 4° degrees ~ 120 microns



Gap must be > or = 10 [particle size]!!

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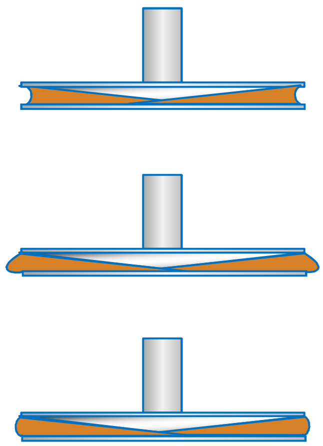
Correct Sample Loading



× Under Filled sample:
Lower torque contribution

× Over Filled sample:
Additional stress from drag along the edges

✓ **Correct Filling**

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When to use Parallel Plates

- Low/Medium/High Viscosity Liquids
- Soft Solids/Gels
- Thermosetting materials
- Samples with large particles
- Samples with long relaxation time
- Temperature Ramps/ Sweeps
- Materials that may slip
  - Crosshatched or Sandblasted plates
- Small sample volume

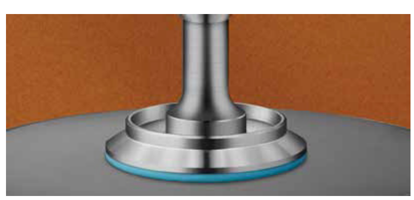

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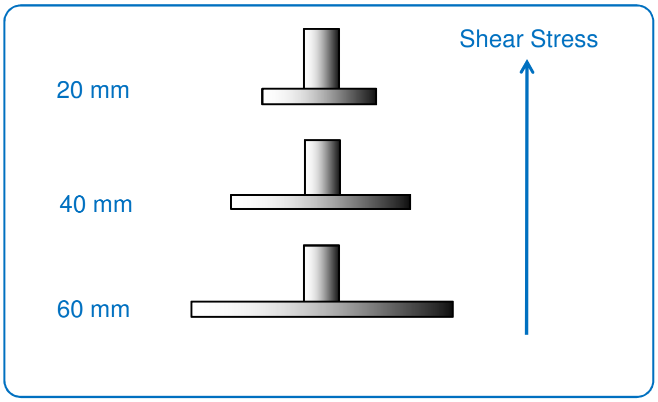
Plate Diameters



As diameter decreases, shear stress increases $\sigma = M\frac{2}{\pi r^2}$

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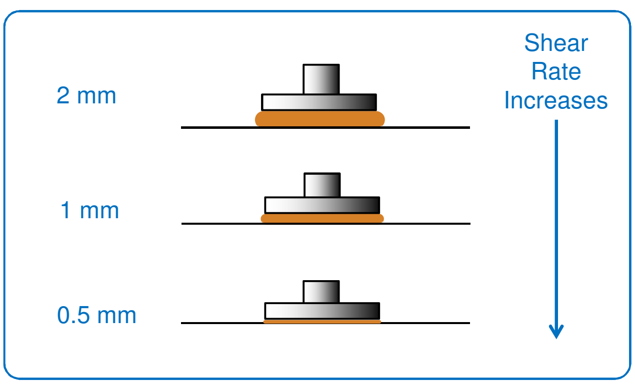
Plate Gaps



As gap height decreases, shear rate increases $\dot{\gamma} = \Omega\frac{r}{h}$

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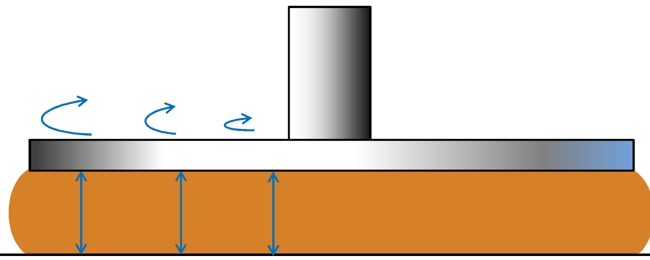
Effective Shear Rate varies across a Parallel Plate

- For a given angle of deformation, there is a greater arc of deformation at the edge of the plate than at the center



$$\gamma = \frac{dx}{h}$$

dx increases further from the center,
h stays constant

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When to Use Concentric Cylinders

- Low to Medium Viscosity Liquids
- Unstable Dispersions and Slurries
- Minimize Effects of Evaporation
- Weakly Structured Samples (Vane)
- High Shear Rates

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Peltier Concentric Cylinders

Concentric Cylinder Cup and Rotor Compatibility Chart

Cup/Rotor	DIN	Recessed End	Starch Impeller	Vane	Wide Gap Vane	Double Gap	Helical Rotor
Standard (rad= 15 mm)	●	●		●	●		
Large Diameter (rad= 22 mm)	●	●	●	●	●		●
Starch (rad= 18.5 mm)	●	●	●	●	●		●
Grooved				●	●		
Double Gap						●	
Helical (rad= 17 mm)							●

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Geometry Information – Estimated Min and Max Shear Rates

Geometry	Diameter (mm)	Degree	Gap (micron)	Sample Volume (mL)	**Max Shear Rate (approx) 1/s**	Min Shear Rate (approx) 1/s
Parallel Plate and Cone and Plate	8	0	1000	0.05	**1.20E+03**	4.00E-07
		0	500	0.03		
		0.5	18	1.17E-03		
		1	28	2.34E-03		
		2	52	4.68E-03		
		4	104	9.37E-03		
	20	0	1000	0.31	**3.00E+03**	1.00E-06
		0.5	18	0.02	**3.44E+04**	1.15E-05
		1	28	0.04	**1.72E+04**	5.73E-06
		2	52	0.07	**8.60E+03**	2.87E-06
		4	104	0.15	**4.30E+03**	1.43E-06
	25	0	1000	0.49	**3.75E+03**	1.25E-06
		0.5	18	0.04		
		1	28	0.07		
		2	52	0.14		
		4	104	0.29		
	40	0	1000	1.26	**6.00E+03**	2.00E-06
		0.5	18	0.15	**3.44E+04**	1.15E-05
		1	28	0.29	**1.72E+04**	5.73E-06
		2	52	0.59	**8.60E+03**	2.87E-06
		4	104	1.17	**4.30E+03**	1.43E-06
	60	0	1000	2.83	**9.00E+03**	3.00E-06
		0	500	1.41		
		0.5	18	0.49	**3.44E+04**	1.15E-05
		1	28	0.99	**1.72E+04**	5.73E-06
		2	52	1.97	**8.60E+03**	2.87E-06
		4	104	3.95	**4.30E+03**	1.43E-06
Concentric Cylinder	Conical Din Rotor			19.6	**4.36E+03**	1.45E-06
	Recessed End			6.65	**4.36E+03**	1.45E-06
	Double Wall			11.65	**1.59E+04**	5.31E-06
	Pressure Cell			9.5		
	Standard Vane			28.72		

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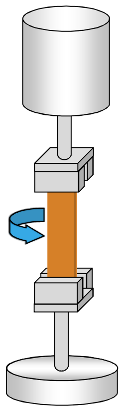
Torsion Rectangular



$$K_\gamma = \frac{t}{l\left[1 - 0.378\left(\frac{t}{w}\right)^2\right]}$$

$$K_\tau = \frac{\left(3 + \frac{1.8}{w}\right)}{(w \cdot t^2)}$$

w = Width
l = Length
t = Thickness

Advantages:

- High modulus samples
- Small temperature gradient
- Simple to prepare

Disadvantages:

- No pure Torsion mode for high strains

Torsion cylindrical also available

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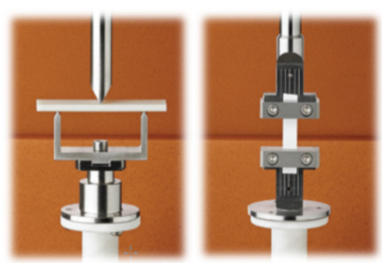
Torsion and DMA Measurements

- Torsion and DMA geometries allow solid samples to be characterized in a temperature controlled environment
  - Torsion measures G', G", and Tan δ
  - DMA measures E', E", and Tan δ
    - ARES G2 DMA is standard function (50 µm amplitude)
    - DMA is an optional DHR function (100 µm amplitude)

Rectangular and cylindrical torsion



DMA 3-point bending and tension (cantilever not shown)

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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

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Geometry Overview

Geometry	Application	Advantage	Disadvantage
Cone/plate	fluids, melts viscosity > 10mPas	true viscosities	temperature ramp difficult
Parallel Plate	fluids, melts viscosity > 10mPas	easy handling, temperature ramp	shear gradient across sample
Couette	low viscosity samples < 10 mPas	high shear rate	large sample volume
Double Wall Couette	very low viscosity samples < 1mPas	high shear rate	cleaning difficult
Torsion Rectangular	solid polymers, composites	glassy to rubbery state	Limited by sample stiffness
DMA	Solid polymers, films, Composites	Glassy to rubbery state	Limited by sample stiffness (Oscillation and stress/strain)

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Geometry Size Selection

- DHR
  - Most common is 40-mm parallel plate; 1000 micron gap
  - Use 60-mm cone and plate and parallel plate for low viscosity materials, say, up to 100 mPa-sec, but 40-mm geometries can often handle these materials too.
  - 20-mm plates are often used at higher viscosities.
  - 25-mm parallel plates are the preferred choice for polymer melts.
  - 40-mm 2-degree is the most common cone geometry. This is often used to verify an instrument with a viscosity standard.
  - 8-mm plates are often used for pressure sensitive adhesives and for asphalt around room temperature.
- ARES-G2
  - The most common geometry on the ARES-G2 is the 25-mm parallel plate. Examples would be polymer melts and thermosetting materials.
  - Low viscosity fluids are run with 50-mm plates or cone-and-plate.
  - Again, 8-mm plates are used for adhesives and asphalt at room temperature.

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TEST METHODS UNIDIRECTIONAL

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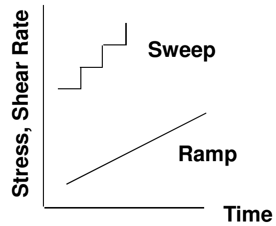
Rheological Methods – Unidirectional Testing

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



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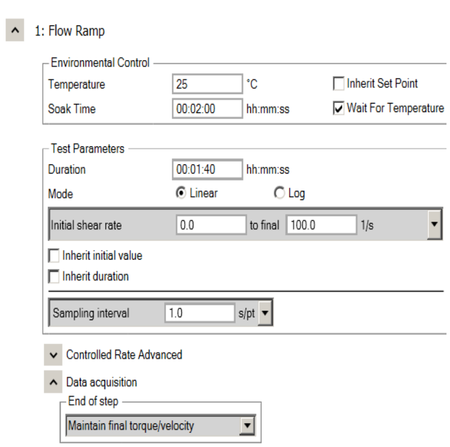
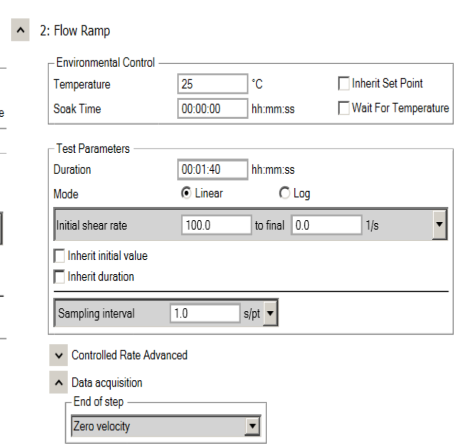
Procedure for Rate Ramp Up/Ramp Down





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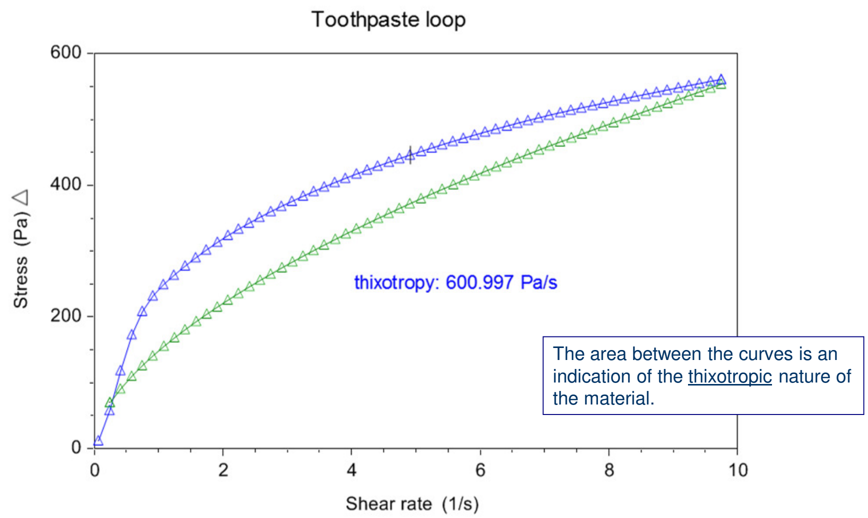
Thixotropy: Up & Down Flow Curves



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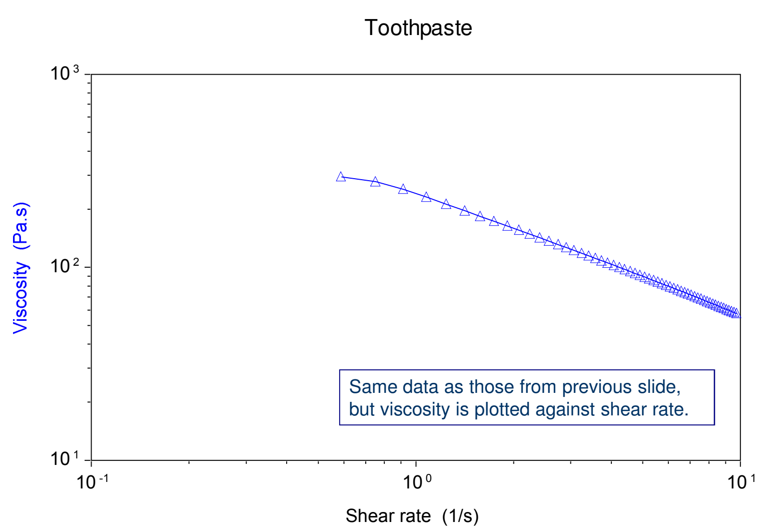
Rate Ramp



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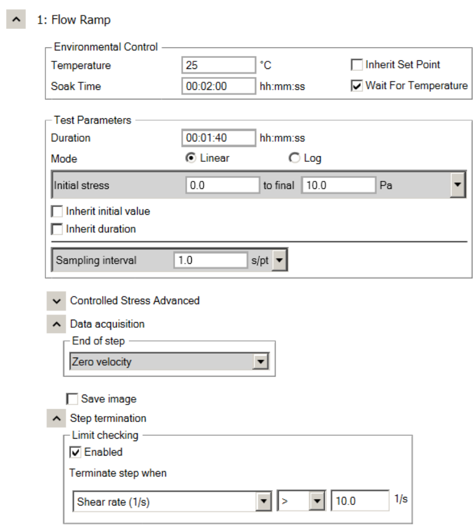
Stress Ramp for Yield Stress Determination



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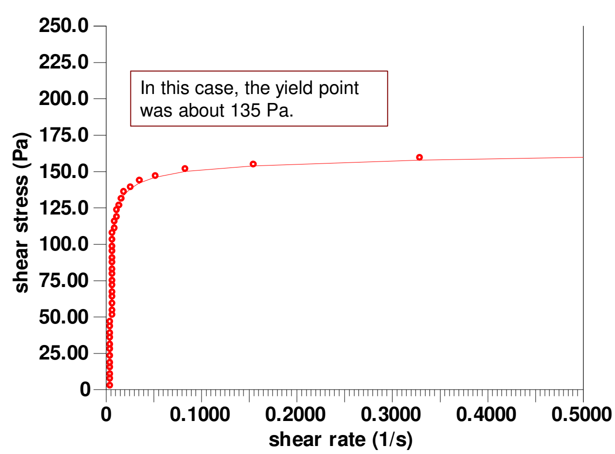
Stress Ramp with Yield



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Ramp Selection

- Do a ramp as a preliminary scouting test, prior to the more fundamentally sound rate or stress sweep.
- For rate ramps, a common acceleration rate is 1 sec^{-1} per second. For example, 0 to 100 sec^{-1} in 100 seconds. This is a starting point. The operator can select a rate or a range that is more appropriate for the sample in question.
- Ramp up/Ramp down tests are common for determining thixotropy. The area between the up and down curves is often reported as a thixotropy parameter.
- There have been times when the reproducibility is better with the down curve than it is with the up curve.
- Ramps are good for characterizing materials that may slip or exude from the gap as the shear rate is increased. Often one can get to higher shear rates with ramps than with sweeps because one doesn't dwell at the high rates as long.
- Stress ramps are often used to get the yield point of a material. Sometimes these are not always clear-cut. Also, one has to be cautious when working with models. There have been instances where negative (!?) yield stresses are determined by software for the selected model.
- For stress ramps, use the Step Termination feature to prevent over-speed.

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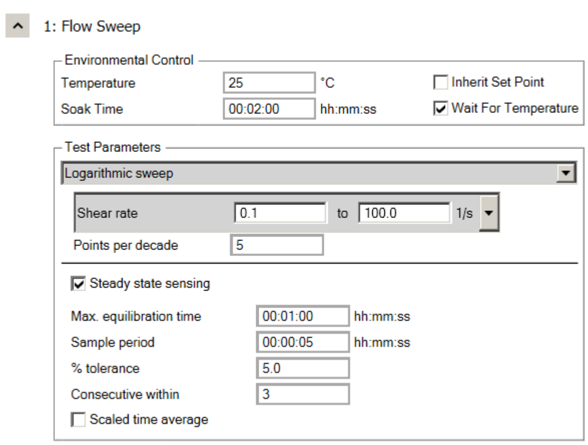
Steady State Flow Test



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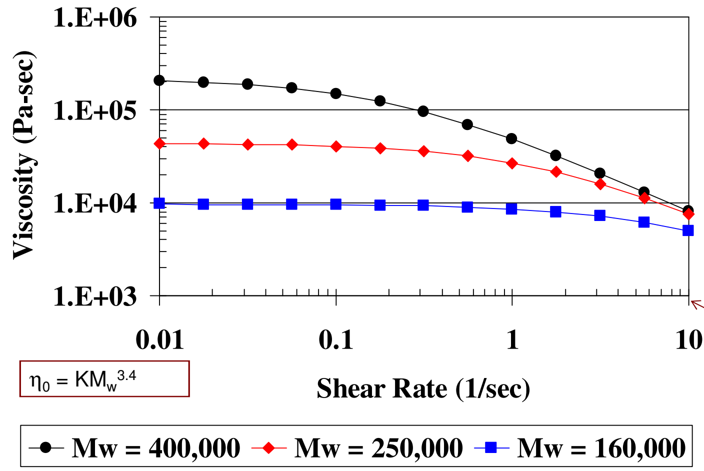
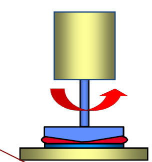
Rate Sweeps - Polymer Melt Flow Curves



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

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.

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Structured fluids

Structured fluids are mostly colloidal dispersions and can be classified into three categories.

- **Suspension** Solid particles in a Newtonian fluid
- **Emulsion** Fluid droplets in a Newtonian fluid
- **Foam** Gas in a fluid (or solid)

- Examples:
  - Paints
  - Coatings
  - Inks
  - Personal Care Products
  - Cosmetics
  - Foods
- Properties:
  - Yield Stress
  - Non-Newtonian Viscous Behavior
  - Thixotropy
  - Elasticity

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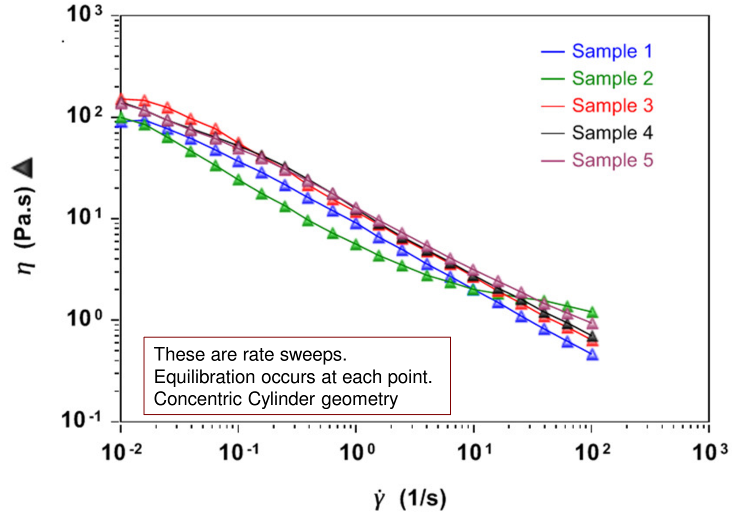
Steady State Flow at 25 C - Coatings



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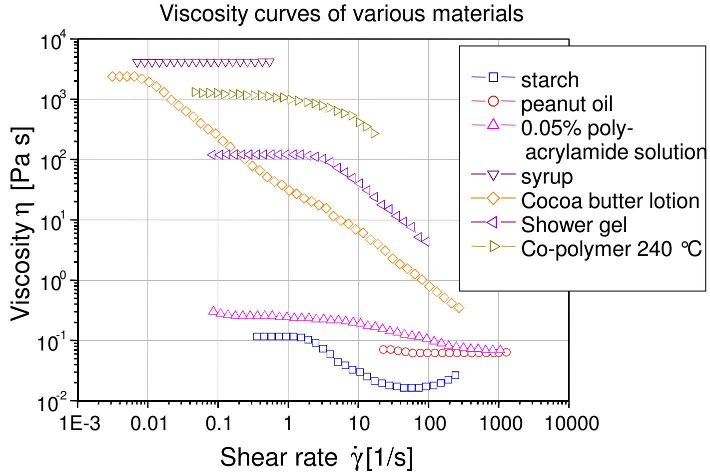
Viscosity curve of various structured fluids

Viscosity curves of various materials



Viscosity function of various structured fluids

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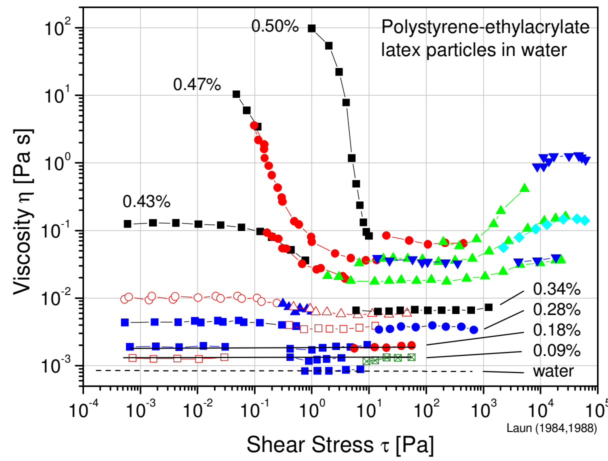
Viscous response of suspensions



Rheological parameters of interest:Yield stress, viscosity, time dependence, linear viscoelasticity

H.M. Laun *Angew. Makromol. Chem.* 335, 124 (1984)

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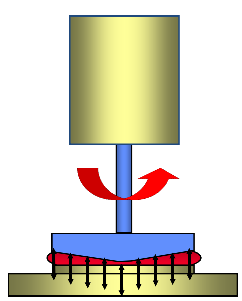
Normal Force Measurements with Cone & Plate



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

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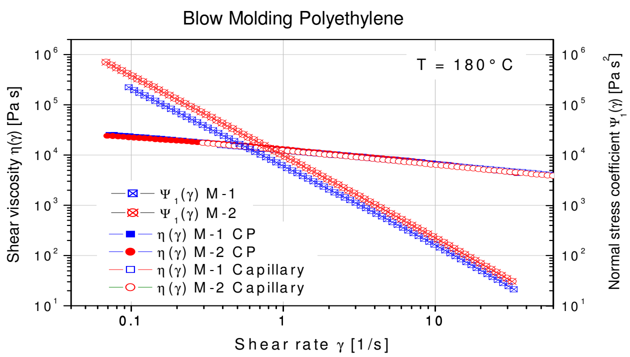
Effect of HDPE Variations in Blow Molding



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

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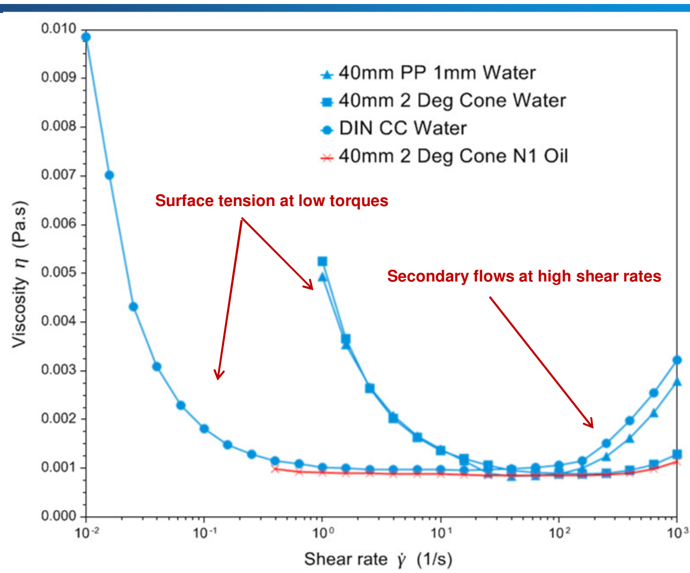
Water at 25°C – Secondary Flow



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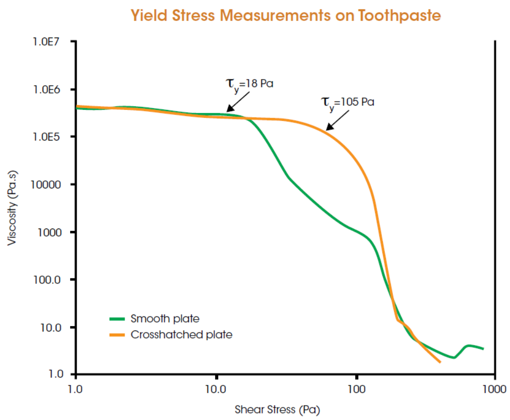
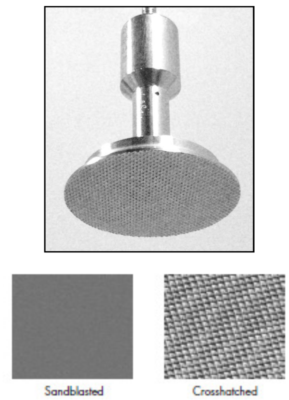
Wall Slip





- Wall slip can manifest as “apparent double yielding”
- Can be tested by running the same test at different gaps
- For samples that don’t slip, the results will be independent of the gap

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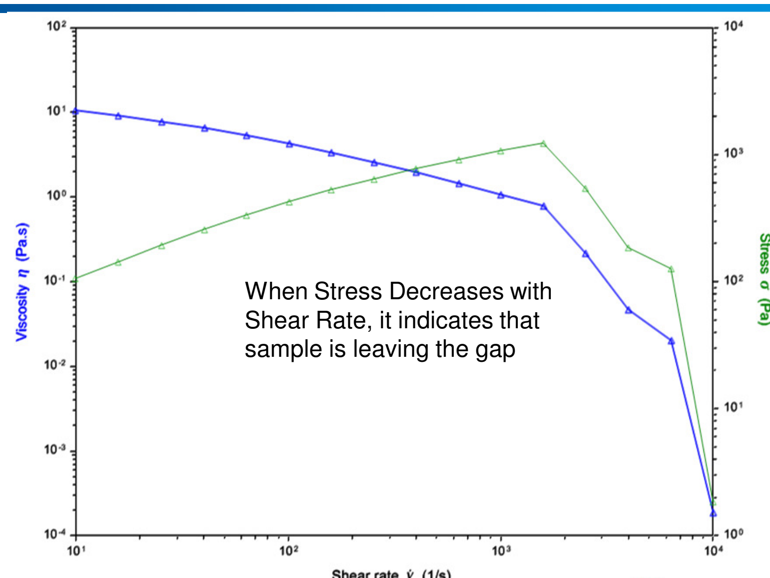
Shear Thinning or Sample Instability?



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Rate and Stress Sweeps

- Sweeps are preferable to ramps because the material is given a chance to equilibrate at a particular shear rate or stress.
- These are useful tests to see how materials will perform in flow, such as transporting fluid through pipes and tubing, after structure has been broken.
- The most common rate range is 0.1 to 100 1/sec, 5 points per logarithmic decade.
- The Steady State sensing feature is a useful tool to perform valid rate or stress sweeps in the shortest amount of time.
- For materials that exhibit flow instability, the ramp may be preferable. With sweeps, the material is exposed to high shear rates for prolonged periods, whereas, with ramps, the dwell time at a particular rate is shorter.

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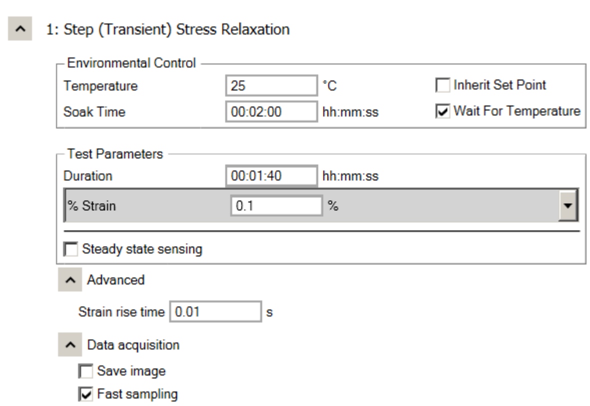
Stress Relaxation



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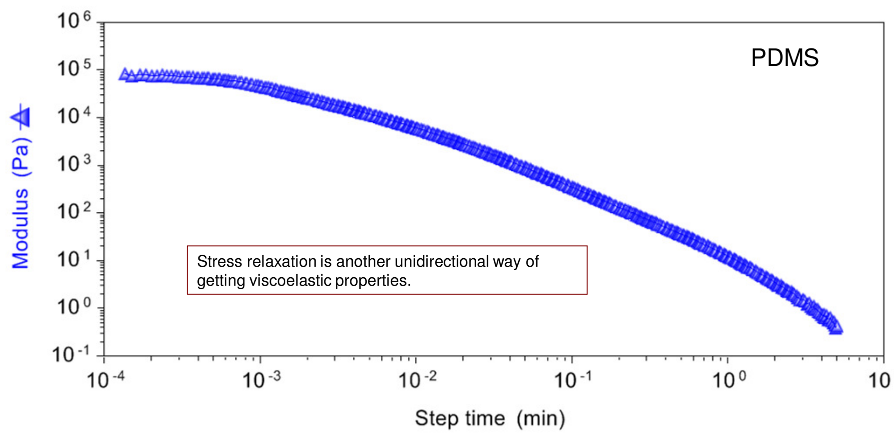
Stress Relaxation



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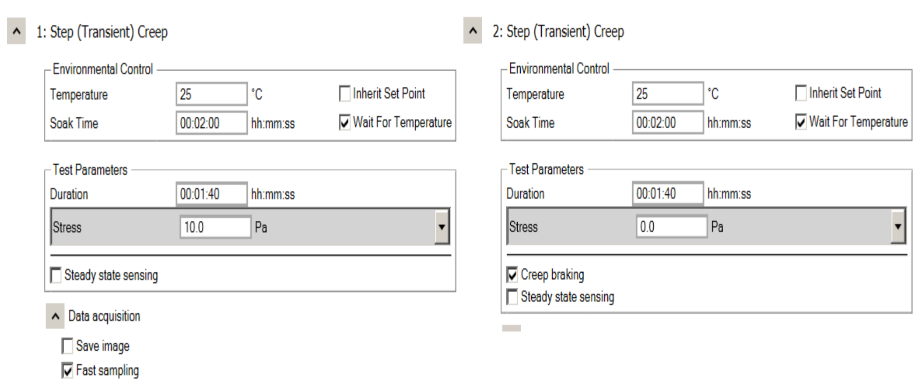
Creep and Recovery



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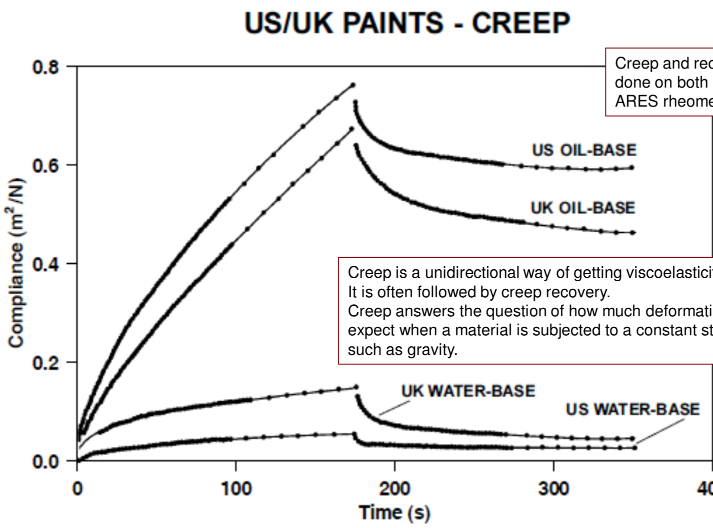
Creep Testing on US and UK Paints



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.

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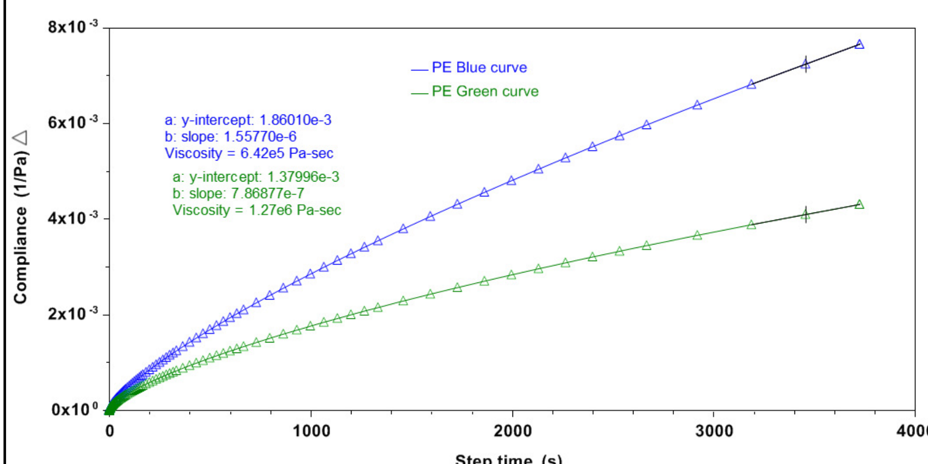
Creep Testing for Zero Shear Viscosity



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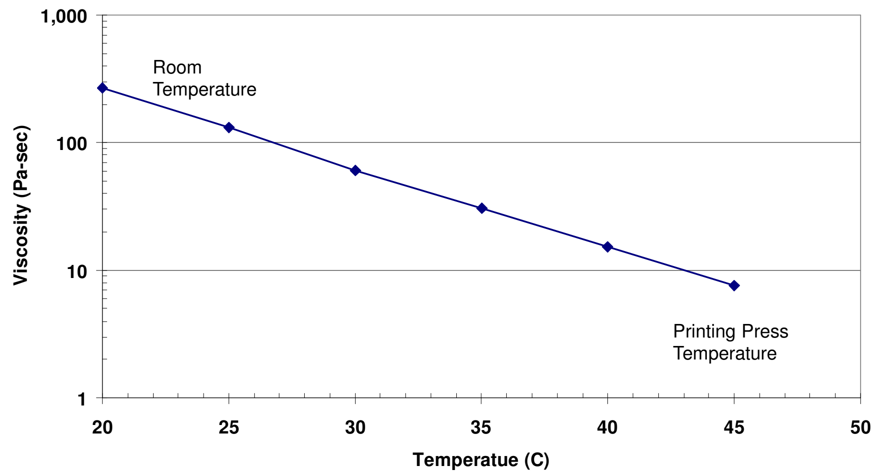
Flow Temperature Ramp – Printing Inks



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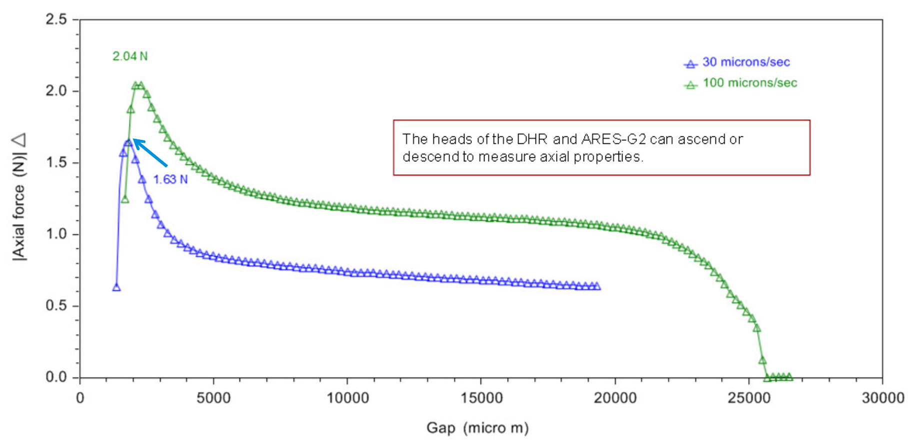
Tack Testing



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TEST METHODS
DYNAMIC TESTING

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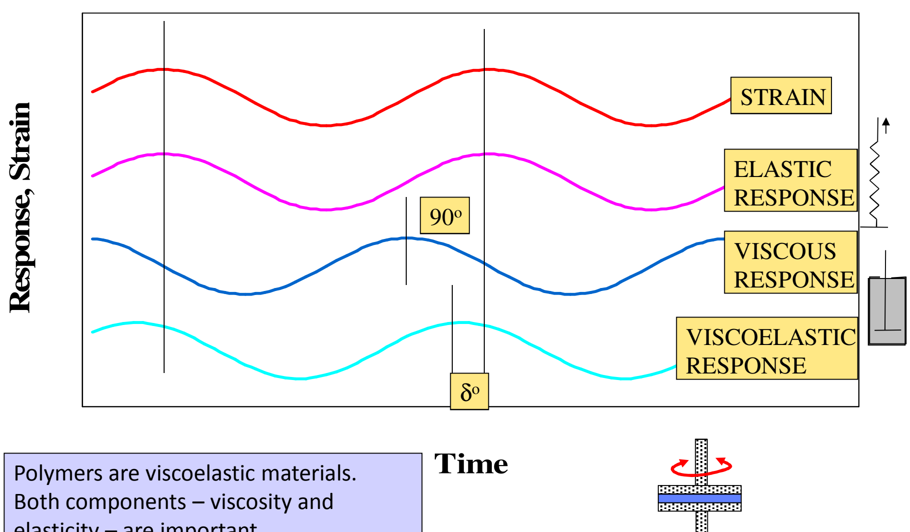
Dynamic Testing



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

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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 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: $\eta^*(\omega) = \eta(\dot{\gamma})$ @ $\dot{\gamma} = \omega$

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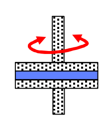
Rheological Methods – Dynamic Oscillatory Testing

- Dynamic Strain Sweep/Dynamic Stress Sweep
- Isothermal Dynamic Time Sweep
- Isothermal Dynamic Frequency Sweep
- Dynamic Temperature Ramp
- Dynamic Temperature Sweep at 1 or Multiple Frequencies.



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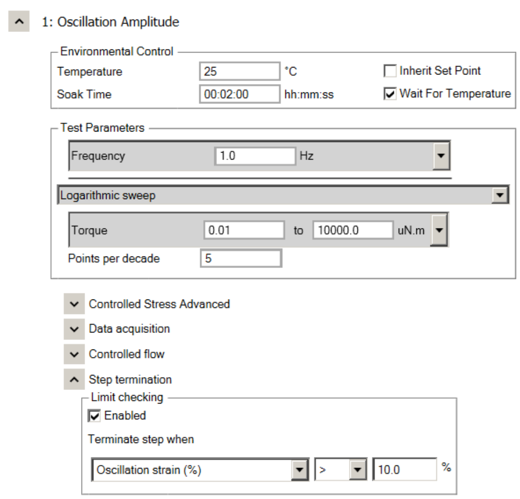
Dynamic Stress Sweep



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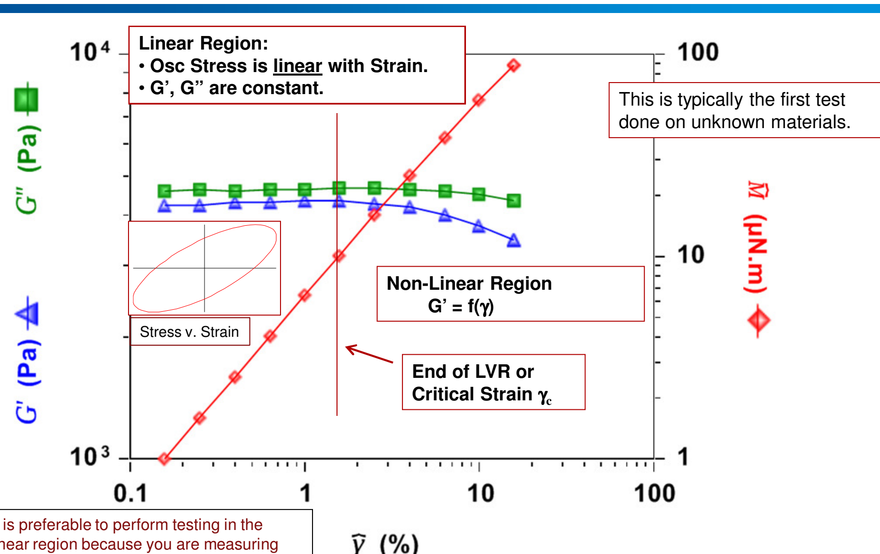
Linear Viscoelasticity



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.

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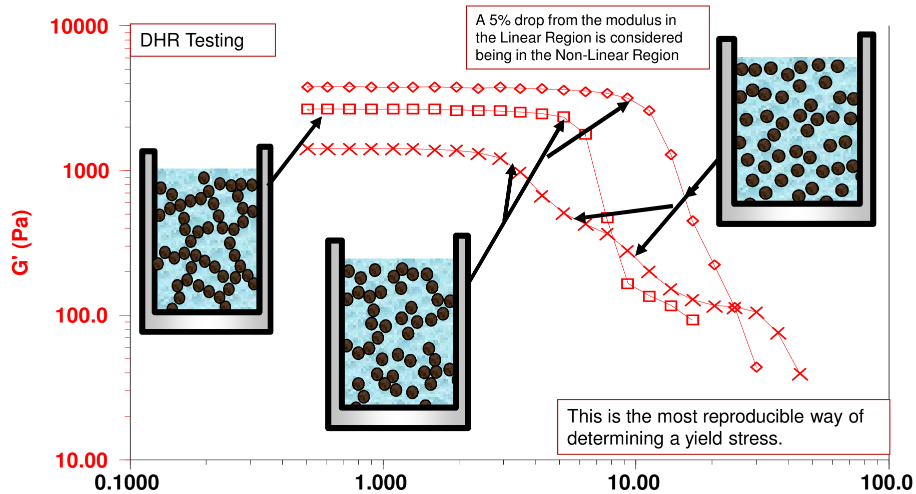
Stability is Related to Structure in Inks

Ink Samples: Oscillation Stress Sweeps @ 6.28 rad/s



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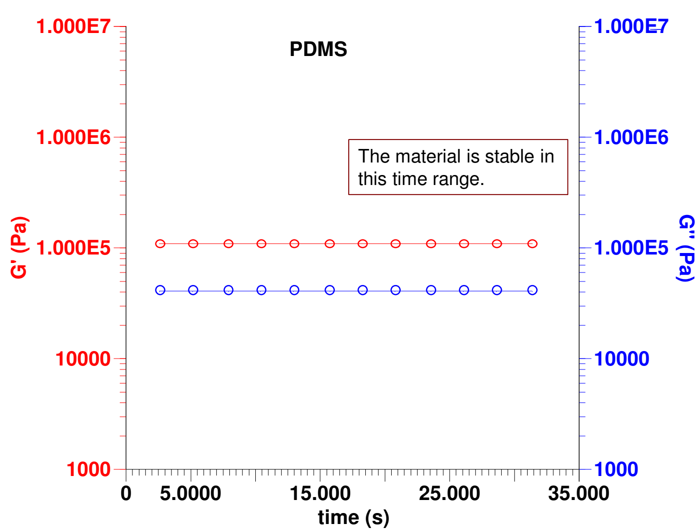
Dynamic Time Sweep: Material Response



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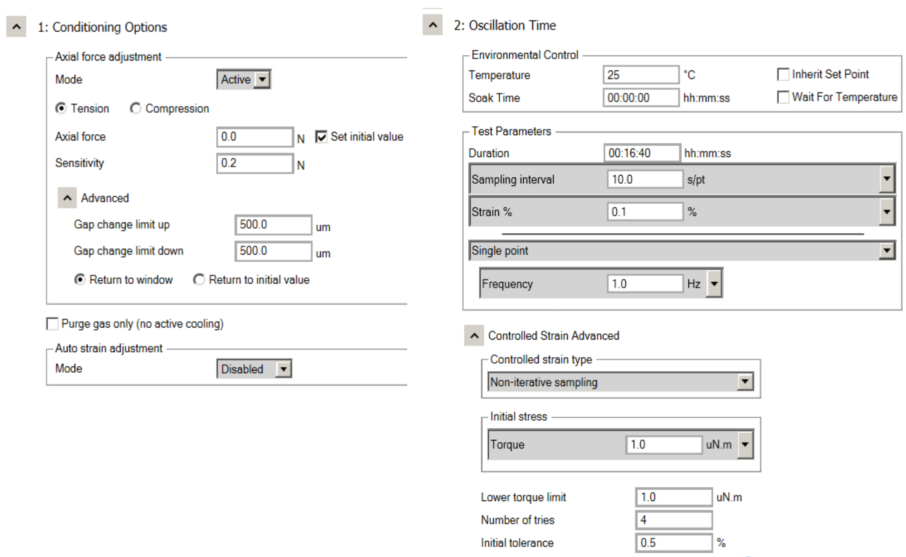
Dynamic Time Sweep for Curing



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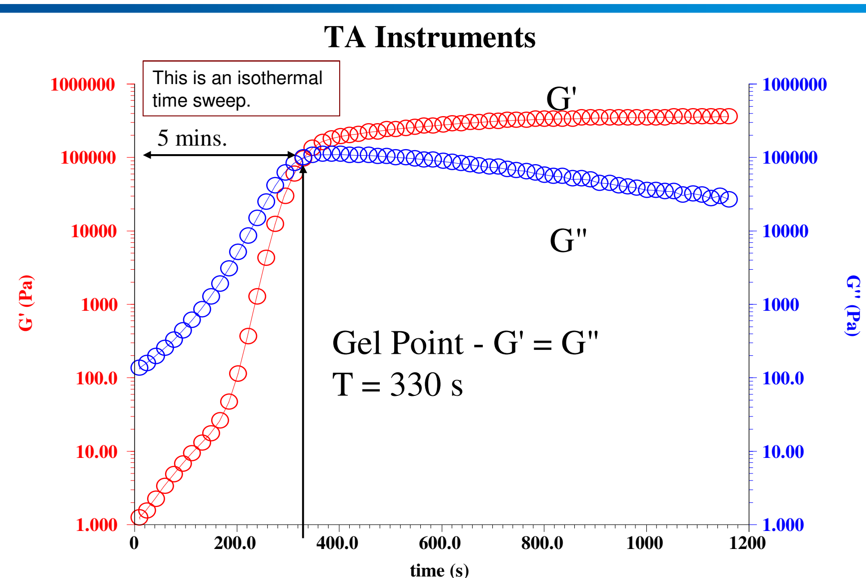
Cure of a "5 minute" Epoxy



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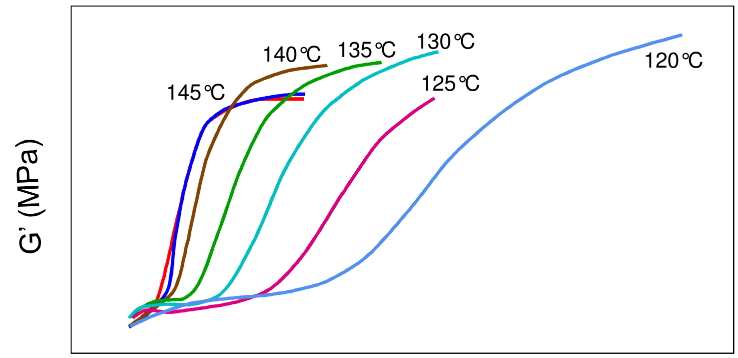
Isothermal Cure of Tire Compound Effect of Curing Temperature



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Dynamic time sweep following shear

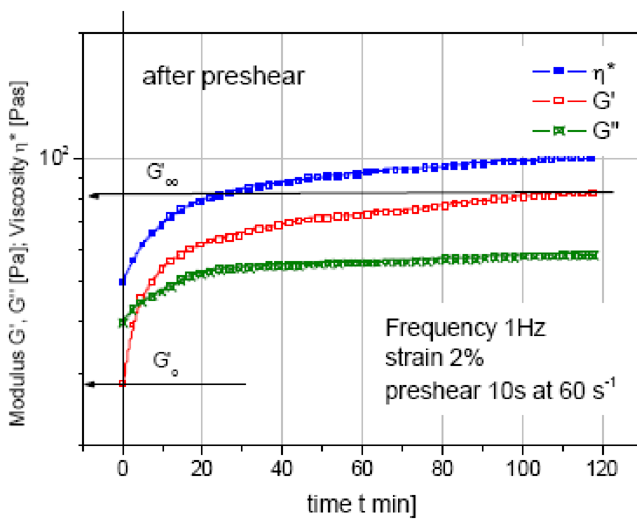

Figure 7: Recovery of structure

This shows how rapidly the structure is rebuilding.

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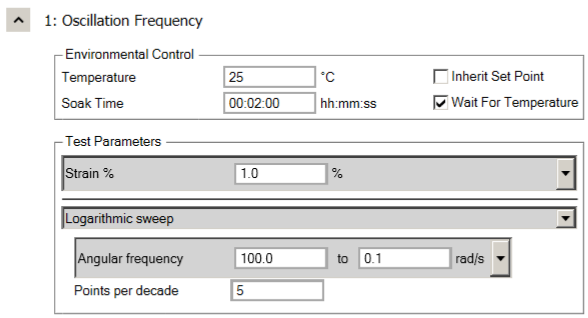
Frequency Sweep



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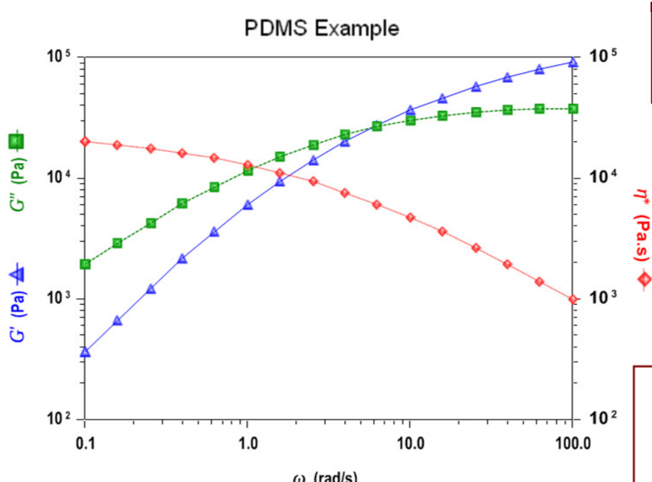
Frequency Sweep on PDMS



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.

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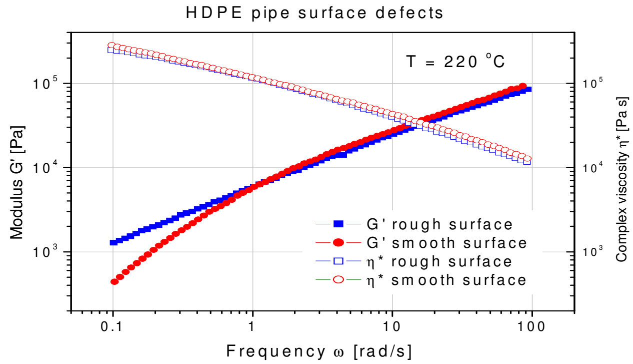
Surface Defects during Pipe Extrusion

Indicated by Elasticity at low frequency

Caused by Broader MWD



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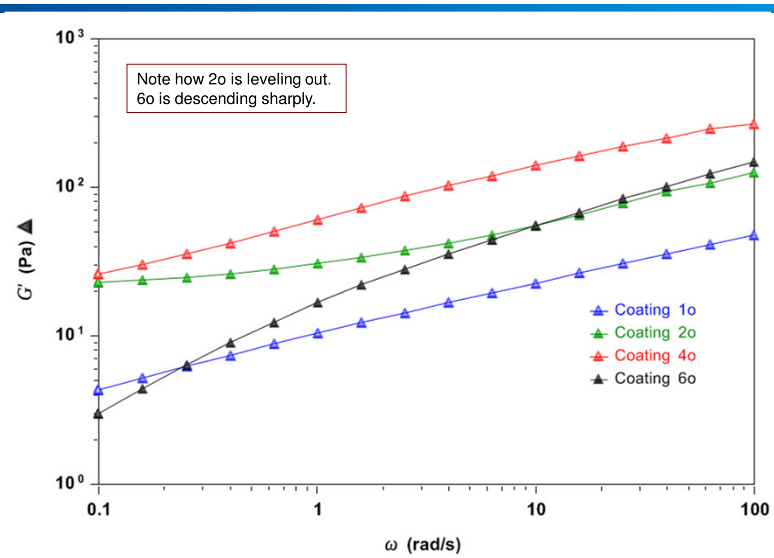
Coatings Frequency Sweep



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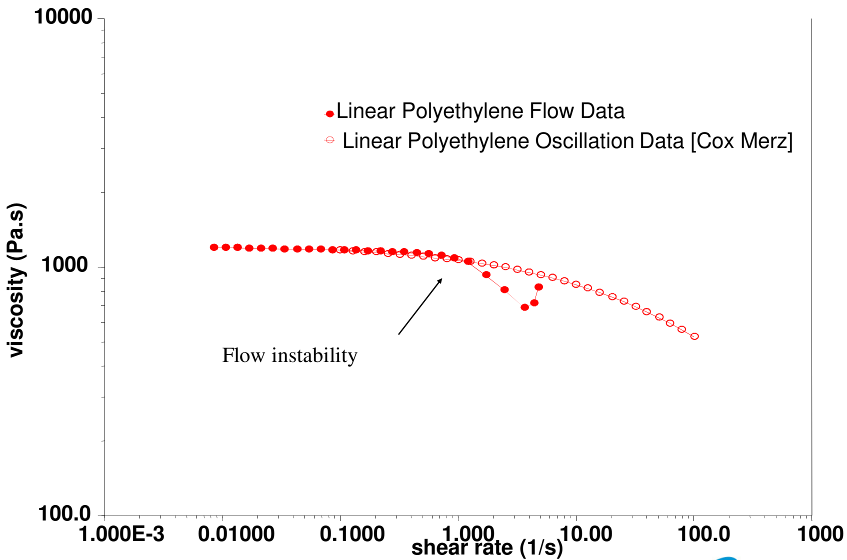
Example of Cox-Merz Rule



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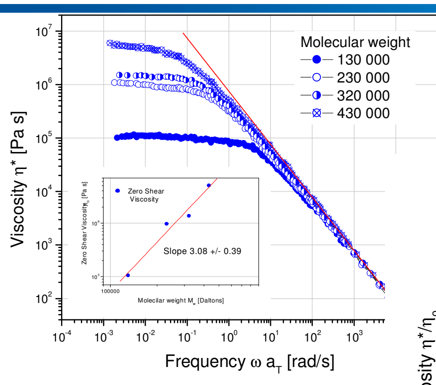
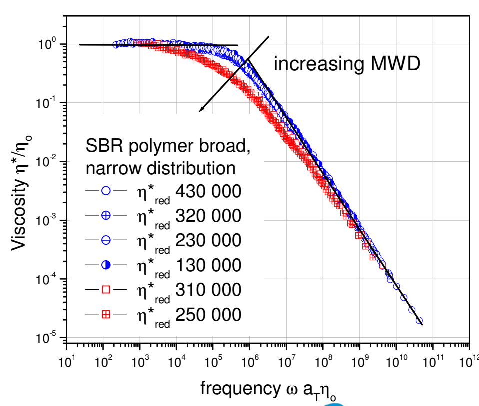
Dynamic testing: Dependence on M_w and MWD



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.



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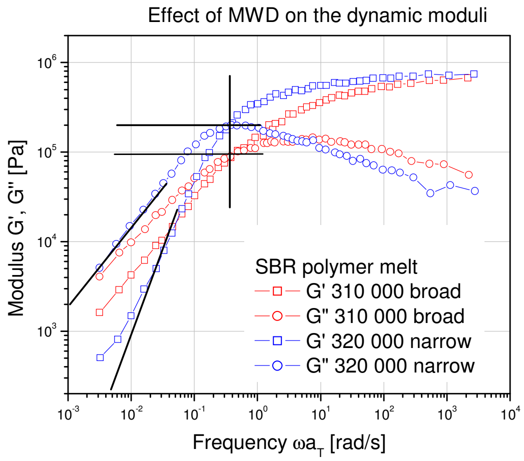
MWD and Dynamic Moduli



- **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

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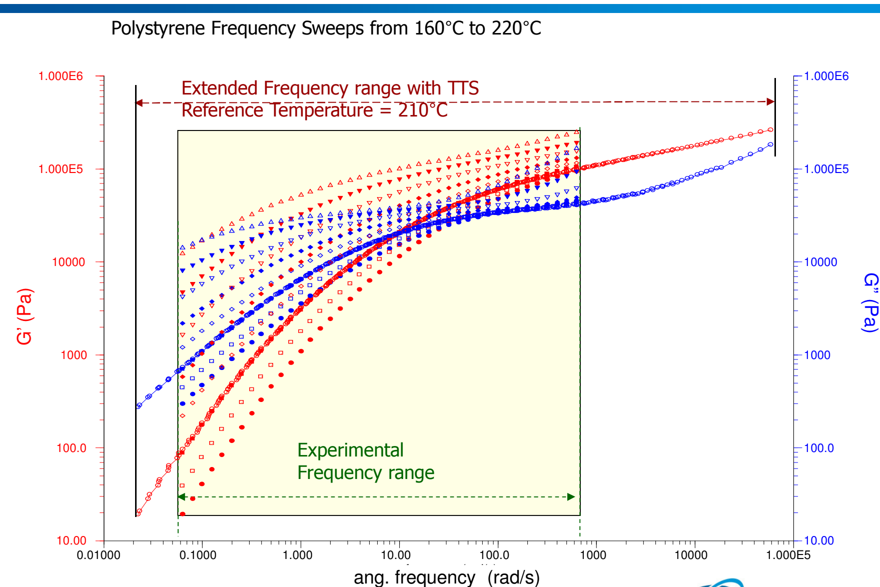
ETC Application: TTS on Polymer Melt



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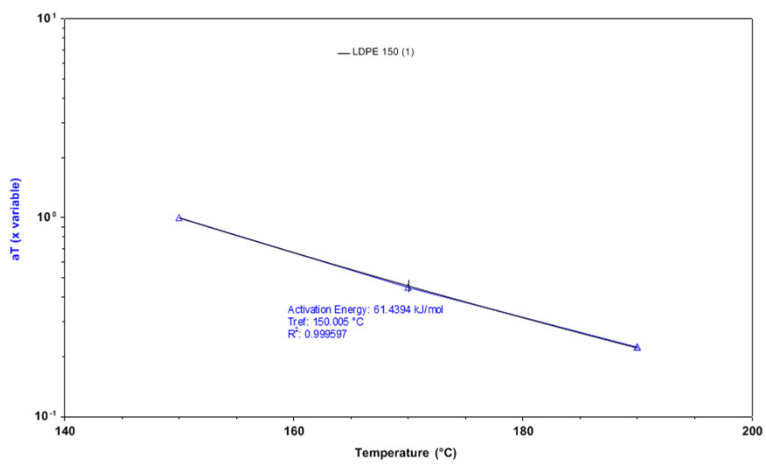
Shift Factors from TTS for LDPE



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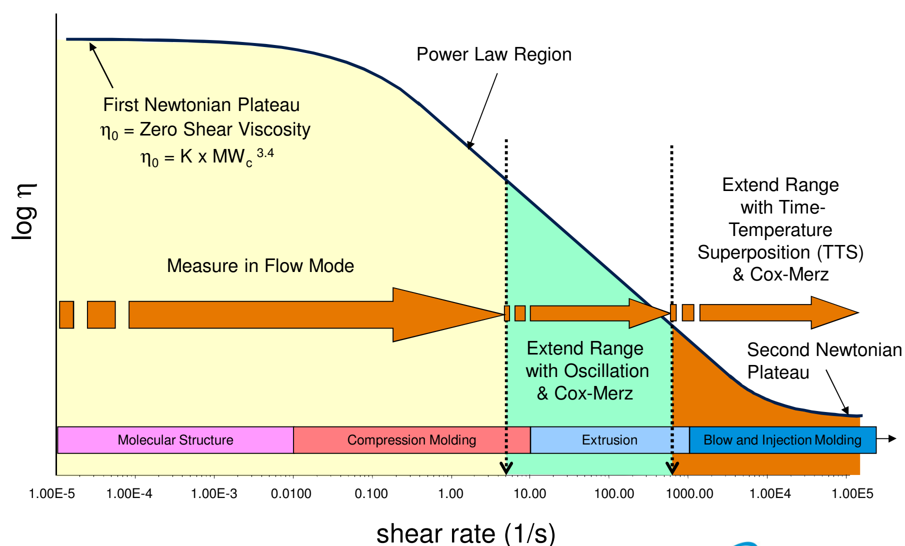
Idealized Flow Curve - Polymers



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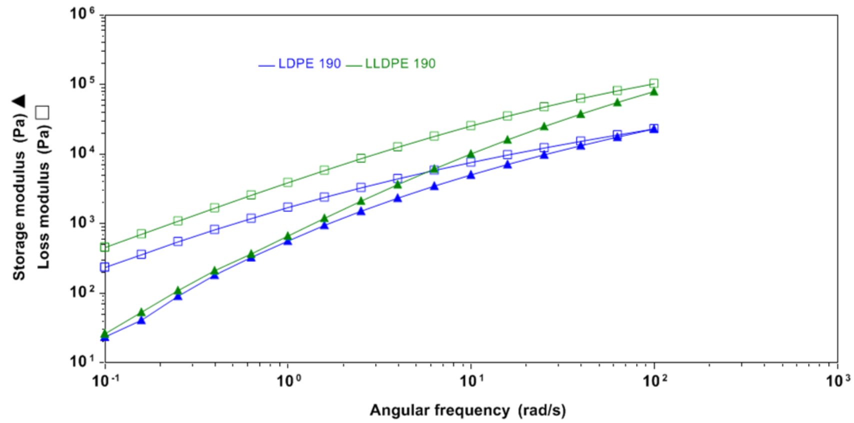
Example: Dynamic Frequency Sweeps



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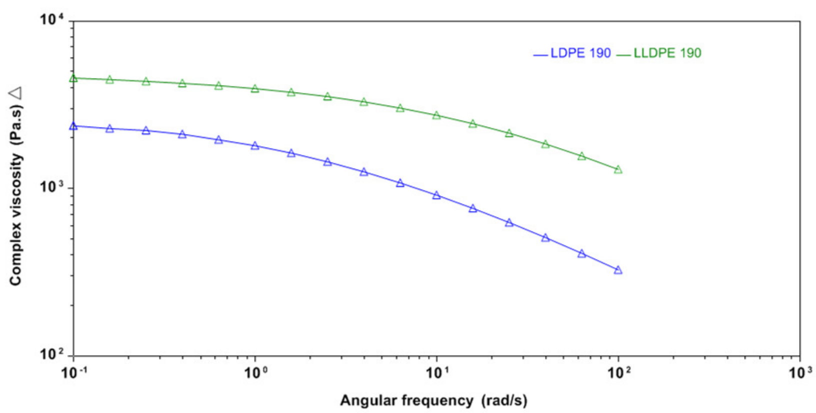
Dynamic Frequency Sweeps



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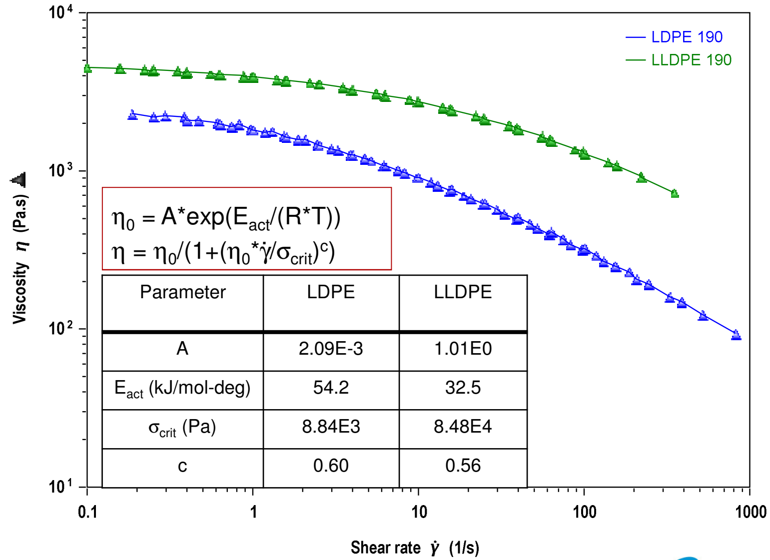
Fitting Data to Williamson/Arrhenius Models



$$\eta_0 = A^*\exp(E_{act}/(R^*T))$$
$$\eta = \eta_0/(1+(\eta_0^*\dot{\gamma}/\sigma_{crit})^c)$$

Parameter	LDPE	LLDPE
A	2.09E-3	1.01E0
E_{act} (kJ/mol-deg)	54.2	32.5
σ_{crit} (Pa)	8.84E3	8.48E4
c	0.60	0.56

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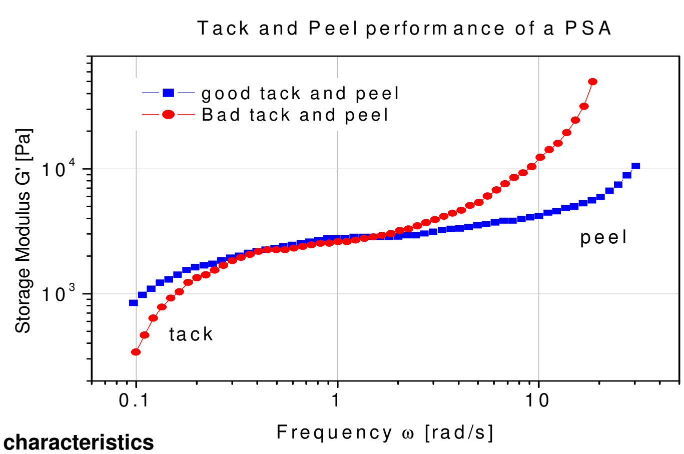
Tack and Peel of Adhesives



Desirable PSA characteristics
Tack: high G‘ at low frequency
Peel: low G‘ at high frequency

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Correlation of Rheological Parameters to Adhesive Properties

Property	Rheological Properties	Practical Adhesive Property
Tack	• Low tan δ and Low G' • Low Cross-links (G" > G' @ ~1 Hz)	High Tack
Shear Resistance	• High G' @ < 0.1 Hz • High Viscosity @ Low Shear Rates	High Shear Resistance
Peel Strength	• High G" @ ~> 100 Hz	High Peel Strength
Cohesive Strength	• High G', low tan δ	High Cohesive Strength
Adhesive Strength	• High G", high tan δ	High Adhesion Strength with Surface

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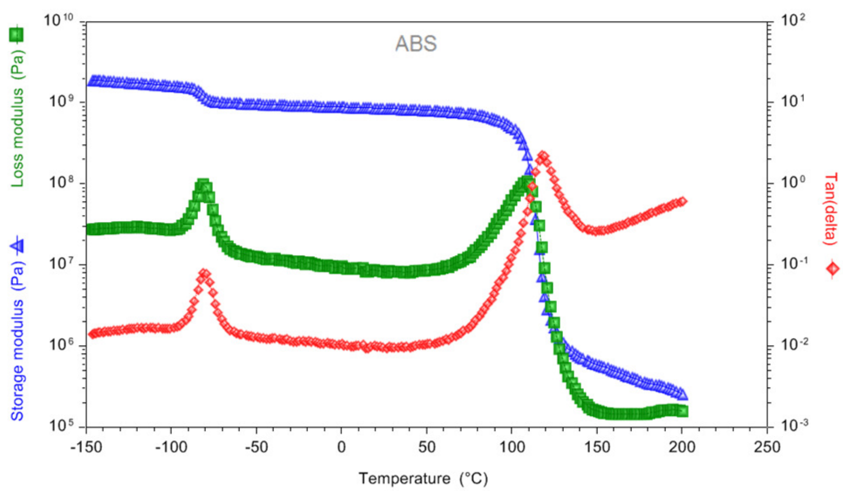
Dynamic Temperature Ramp - Torsion



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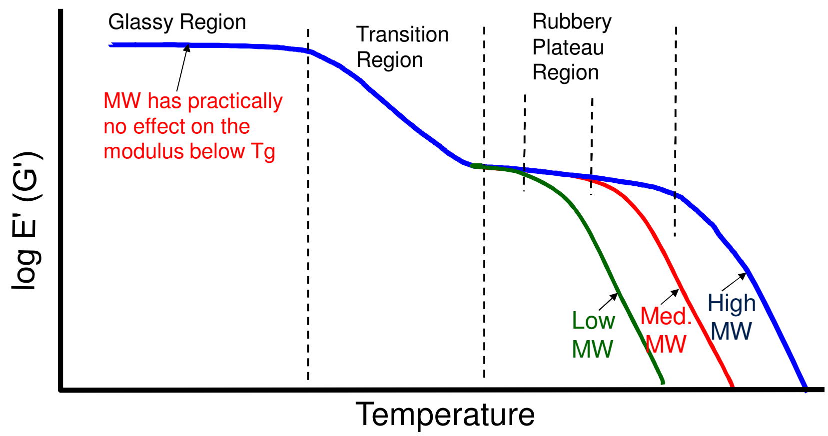
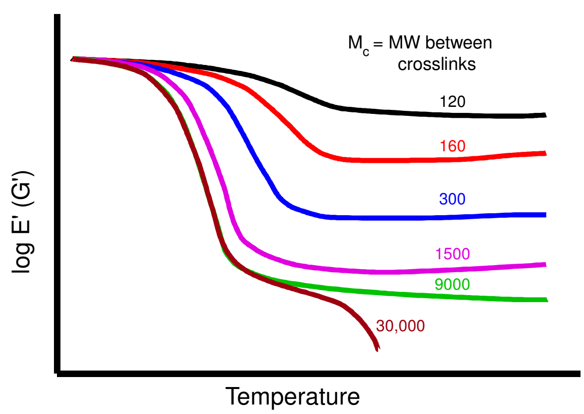
Effect of Molecular Weight



95

Effect of Crosslinking



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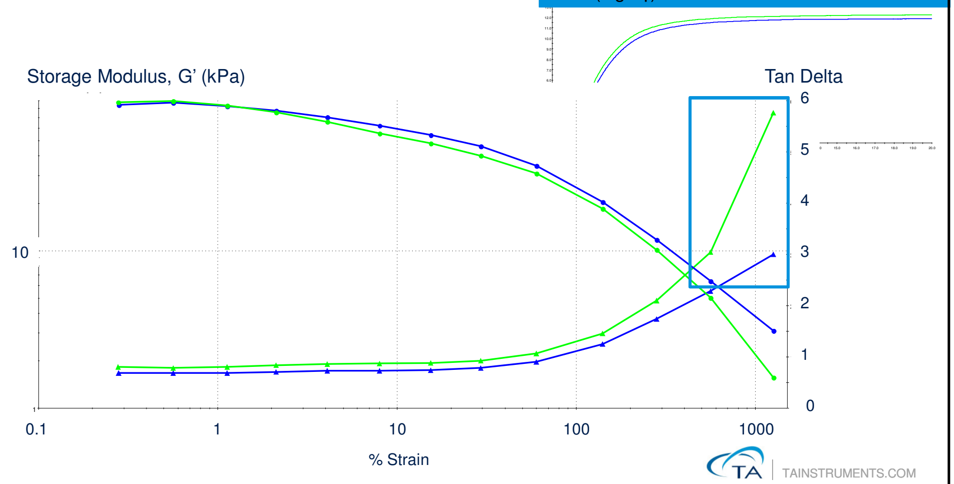
Rubber Compound: Similar cure curve, different processing

High Strain tan δ

	Good	Bad
Viscosity, η*	Similar	
tan δ (low γ)	1.0	0.8
tan δ (high γ)	5.7	3.0



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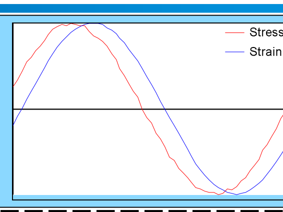
Level 2: Examination of Higher Harmonics

SAOS

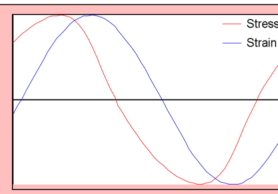
$\gamma(t) = \gamma_0 \sin(\omega t)$

$\sigma(t) = \sigma_0 \sin(\omega t+\delta)$



LAOS

$\gamma(t) = \gamma_0 \sin(\omega t)$



Fourier Series expansion:

$$\sigma(t) = \sigma_1 \sin(\omega_1 t+\varphi_1) + \sigma_3 \sin(3\omega_1 t+\varphi_3) + \sigma_5 \sin(5\omega_1 t+\varphi_5) + \ldots$$

$$= \sum_{\substack{n=1 \\ \text{odd}}}^{\infty} \sigma_n \sin(n\omega_1+\varphi_n)$$

LAOS is often called Fourier Transform Rheology, or FT Rheology. A useful way of quantifying non-linear behavior is to compare intensity ratios like σ_3/σ_1, σ_5/σ_1.

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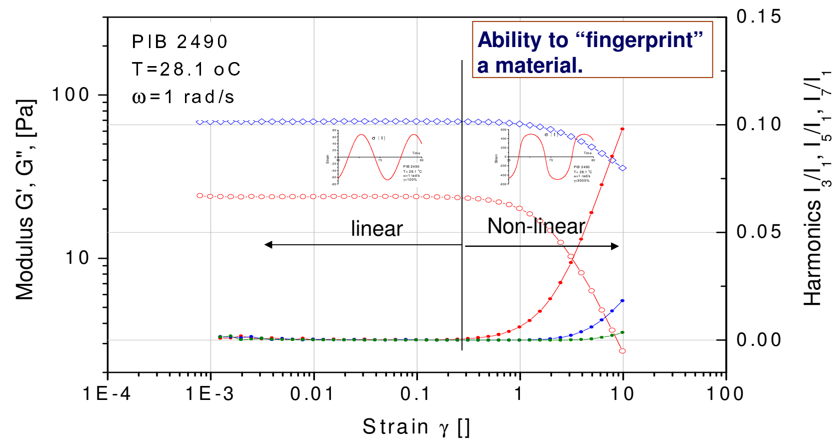
Large Angle Oscillatory Shear: Higher Harmonics



The transition from linear to nonlinear regime for viscoelastic materials shows in a decrease of G' & G" and an increase of the magnitude of the harmonics.

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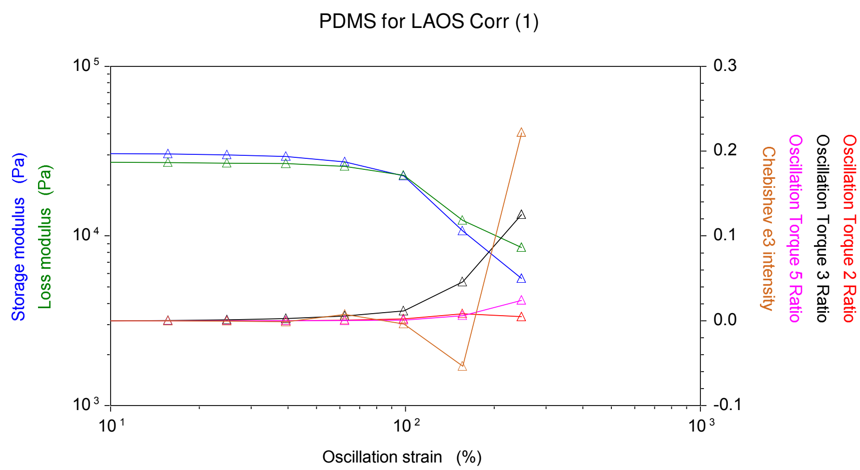
LAOS on PDMS



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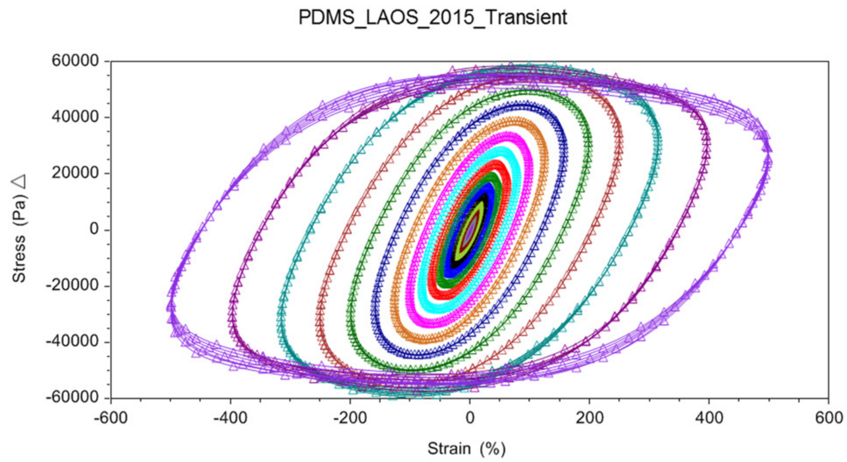
LAOS with Transient Data Acquisition



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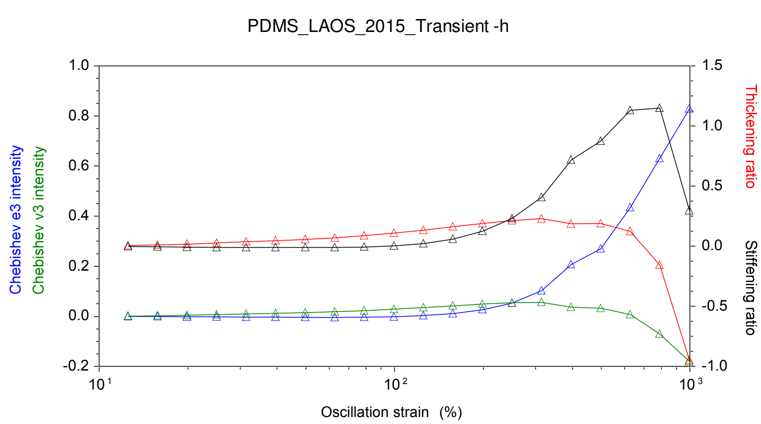
LAOS with Transient Data Acquisition



Cone and Partitioned Plate Accessory

Transient data acquisition with ARES-G2 or DHR-2, DHR-3

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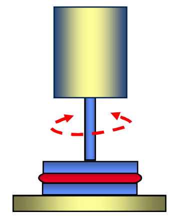
Dynamic Testing

- Of all the tests performed on a rheometer, dynamic oscillatory testing is the most common. Typically, this is the most convenient way of getting a material's viscous and elastic nature.
- Dynamic Stress or Strain sweeps are useful for determining a dynamic yield point of a material and can suggest strains or stresses to use in subsequent tests, such as frequency sweeps or temperature ramps.
- Dynamic Frequency sweeps are the most useful tests for characterizing polymer melts and adhesives. They provide information on the molecular weight and molecular weight distribution of a material.
- Time temperature superposition can often be used to provide knowledge beyond the usual limits of 0.1 to 100 rad/sec.
- The rheometer can be used as a DMA to provide glass transition temperatures and thermal mechanical integrity.



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THE RHEOMETERS

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Five Important Rheometer Specifications

- Torque range
- Angular Resolution
- Angular Velocity Range
- Frequency Range
- Normal Force

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TA Instruments Rotational Rheometers

Discovery HR Rheometer

Combined Motor and Transducer
"Native Mode" = Force (Stress)

ARES-G2

Separate Motor and Transducer
"Native Mode" = Deformation/Deformation Rate
(Strain/Shear Rate)

With computer feedback, the instruments can do both, deformation/deformation rate control and force control.

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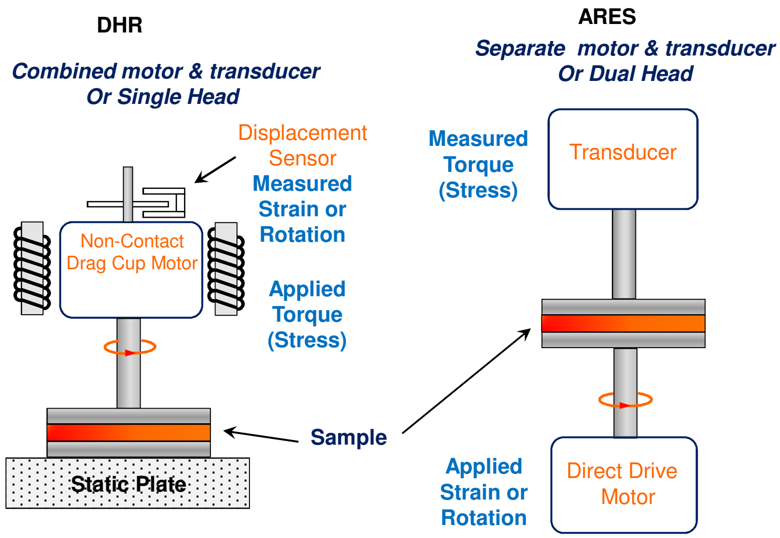
Rotational Rheometer Designs



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Discovery Hybrid Rheometer Specifications

Specification	HR-3	HR-2	HR-1
Bearing Type, Thrust	Magnetic	Magnetic	Magnetic
Bearing Type, Radial	Porous Carbon	Porous Carbon	Porous Carbon
Motor Design	Drag Cup	Drag Cup	Drag Cup
Minimum Torque (nN.m) Oscillation	0.5	2	10
Minimum Torque (nN.m) Steady Shear	5	10	20
Maximum Torque (mN.m)	200	200	150
Torque Resolution (nN.m)	0.05	0.1	0.1
Minimum Frequency (Hz)	1.0E-07	1.0E-07	1.0E-07
Maximum Frequency (Hz)	100	100	100
Minimum Angular Velocity (rad/s)	0	0	0
Maximum Angular Velocity (rad/s)	300	300	300
Displacement Transducer	Optical encoder	Optical encoder	Optical encoder
Optical Encoder Dual Reader	Standard	N/A	N/A
Displacement Resolution (nrad)	2	10	10
Step Time, Strain (ms)	15	15	15
Step Time, Rate (ms)	5	5	5
Normal/Axial Force Transducer	FRT	FRT	FRT
Maximum Normal Force (N)	50	50	50
Normal Force Sensitivity (N)	0.005	0.005	0.01
Normal Force Resolution (mN)	0.5	0.5	1

DHR - DMA mode (optional)	
Motor Control	FRT
Minimum Force (N) Oscillation	0.1
Maximum Axial Force (N)	50
Minimum Displacement (µm) Oscillation	1.0
Maximum Displacement (µm) Oscillation	100
Displacement Resolution (nm)	10
Axial Frequency Range (Hz)	1 x 10^{-5} to 16

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DHR Instrument Model Features

All Discovery Hybrid Rheometers Feature:	**Moving from *HR-1* to HR-2 Adds:**	**Moving from HR-2 to HR-3 Adds:**
- Patented Ultra-low Inertia Drag-Cup Motor	- 5X better low torque in Oscillation	- 4X better low torque in Oscillation
- Single-Thrust & Dual-Radial Bearing Design	- 2X better low torque in steady Shear	- 2X better low torque in steady Shear
- Patented Second Generation Magnetic Bearing	- 25% Higher torque	- Optical Encoder Dual Reader (pat. Pend.)
- Nano-Torque Motor Control	- 2X better NF Sensitivity	- 5X better angular resolution
- High-Resolution Optical Encoder	- Direct Strain Oscillation	- 3X better phase angle resolution
- Superior Stress and Strain Control	- Fast data sampling	- No encoder drift
- Force Rebalance Normal Force (FRT)	- Transient Data Acquisition/LAOS	
- Patented Smart Swap Geometries	- Stress Growth (Transient NF)	
- True Position Sensor (Patent Pending)	- Access to UV Curing Options	
- Ultra-low Compliance Single-Piece Frame	- Access to SALS Option	
- Heat and vibration Isolated Electronics Design	- Access to Interfacial Options	
- Smart Swap™ Temperature Systems		
- Superior Peltier Technology		
- Patented Heat Spreader Technology		
- Patented Active Temperature Control		
- Color Display		
- Capacitive Touch Keypad		
- TRIOS Software		
- Navigator Software		
- Electronic Bearing Lock		
- NIST Traceable Torque Calibration		

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ARES-G2 Rheometer Specifications

Force/Torque Rebalance Transducer (Sample Stress)	
Transducer Type	Force/Torque Rebalance
Transducer Torque Motor	Brushless DC
Transducer Normal/Axial Motor	Brushless DC
Minimum Torque (µN.m) Oscillation	0.05
Minimum Torque (µN.m) Steady Shear	0.1
Maximum Torque (mN.m)	200
Torque Resolution (nN.m)	1
Transducer Normal/Axial Force Range (N)	0.001 to 20
Transducer Bearing	Groove Compensated Air

Driver Motor (Sample Deformation)	
Maximum Motor Torque (mN.m)	800
Motor Design	Brushless DC
Motor Bearing	Jeweled Air, Sapphire
Displacement Control/ Sensing	Optical Encoder
Strain Resolution (µrad)	0.04
Minimum Angular Displacement (µrad) Oscillation	1
Maximum Angular Displacement (µrad) Steady Shear	Unlimited
Angular Velocity Range (rad/s)	$1x\ 10^{-6}$ to 300
Angular Frequency Range (rad/s)	$1x\ 10^{-7}$ to 628
Step Change, Velocity (ms)	5
Step Change, Strain (ms)	10

Orthogonal Superposition (OSP) and DMA modes	
Motor Control	FRT
Minimum Transducer Force (N) Oscillation	0.001
Maximum Transducer Force (N)	20
Minimum Displacement (µm) Oscillation	0.5
Maximum Displacement (µm) Oscillation	50
Displacement Resolution (nm)	10
Axial Frequency Range (Hz)	1×10^{-5} to 16

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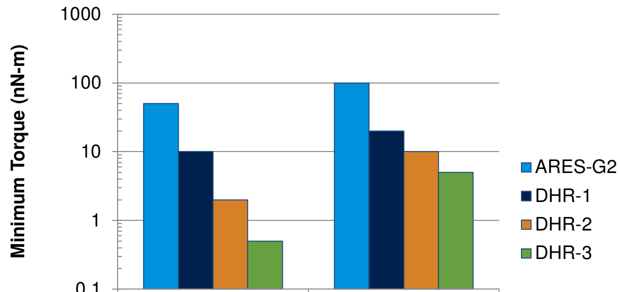
Minimum Torque Specs



	Oscillation	Steady
ARES-G2	50	100
DHR-1	10	20
DHR-2	2	10
DHR-3	0.5	5

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SELECTED ACCESSORIES

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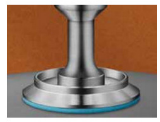
DHR Accessories – Visual Display


Peltier Plate Temperature Systems


Advanced Peltier Plate


Dual Stage Peltier Plate


Upper Heated Plate for Peltier Plate

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

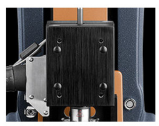

Peltier Concentric Cylinders

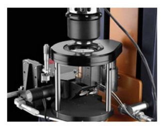

Electrically Heated Cylinder (EHC)

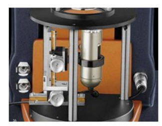

Pressure Cell


Electrically Heated Plates


Environmental Test Chamber


Relative Humidity Accessory


Modular Microscope (MMA)


Optical Plate

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DHR Accessories – Visual Display


Small Angle Light Scattering

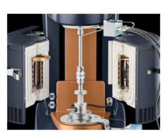

Interfacial Accessories


Tribo-Rheometry Accessory


Magneto-Rheology


Electro-Rheology


UV Curing Accessories


Dielectric Measurement


Immobilization Cell


Starch Pasting Cell


Dynamic Mechanical Analysis

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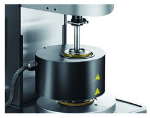
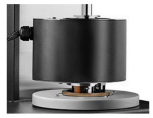
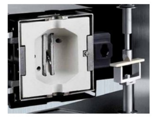
ARES-G2 Accessories


Forced Convection Oven (FCO)


Advanced Peltier System (APS)


Orthogonal Superposition & 2D-SAOS


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


Electrorheology (ER) Accessory

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DMA Capabilities

Motor Control	Force Rebalance Transducer
Minimum Force in Oscillation	0.1 N
Maximum Axial Force	50 N
Minimum Displacement in Oscillation	1 μm
Maximum Displacement in Oscillation	100 μm
Displacement Resolution	10 nm
Axial Frequency Range	1×10^{-5} to 16 Hz

- DHR Film/Fiber Tension Clamp Accessory kit
- DHR 3-Point Bending Clamp Accessory kit
- DHR Cantilever Bending Clamp Accessory kit

The DMA capabilities of the DHR and ARES-G2 are unique for commercial rheometers.

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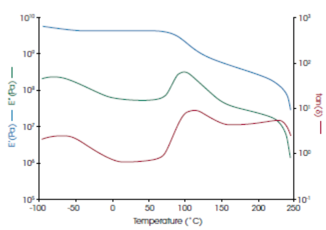
Film Tension

Compression

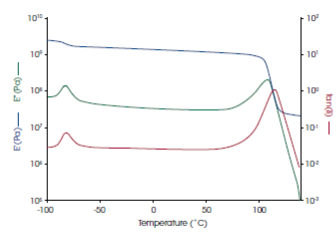
Dual Cantilever

PET Film - Tension 50 µm thick



- Polyethylene terephthalate (PET)
- Three major transitions are observed
- β-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

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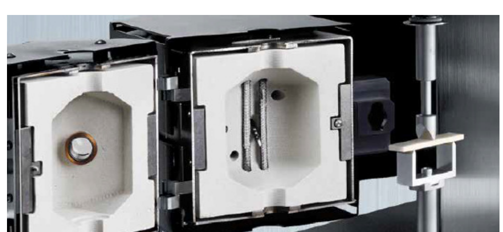
ARES-G2 DMA Mode

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

Tension

Dual and Single Cantilever



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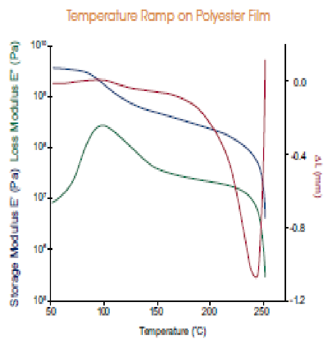
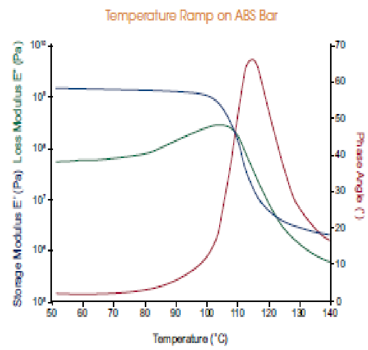
ARES-G2 DMA Testing





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DMA Specifications

	RSA G2	DMA 850	ARES G2 DMA	DHR DMA (optional)
Max Force	35N	18N	20N	50N
Min Force	0.0005N	0.0001N	0.001N	0.1N
Frequency Range	1e-5 to 628 rad/s (1.6e-6 to 100 Hz)	6.28e-3 to 1250 rad/s (0.001 to 200 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	+/- 0.05 to 1,500µm	+/- 0.005 to 1e4 µm	+/- 1 to 50 µm	+/- 1 to 100 µm
Control Stress/Strain	Control Strain (SMT)	Control Stress (CMT)	Control Strain (CMT)	Control Stress (CMT)
Heating Rate	0.1°C to 60°C/min	0.1°C to 20°C/min	0.1°C to 60°C/min	0.1°C to 60°C/min
Cooling Rate	0.1°C to 60°C/min	0.1°C to 20°C/min	0.1°C to 60°C/min	0.1°C to 60°C/min

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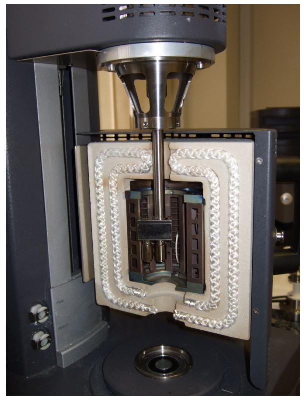
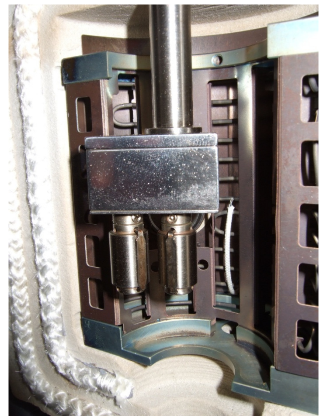
SER2 for DHR Rheometers





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

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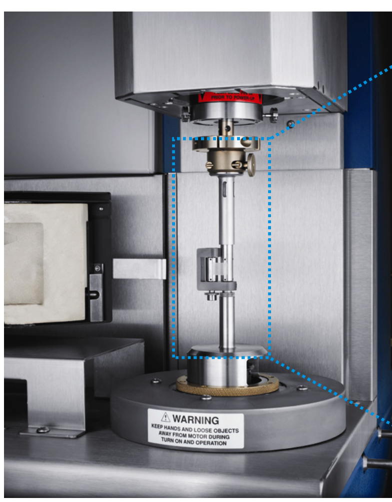
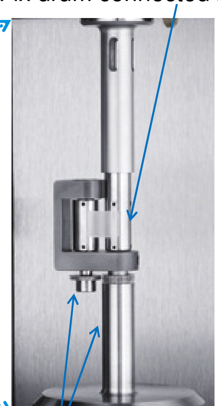
Extensional Viscosity Measurements



Fix drum connected to transducer



Rotating drum connected to the motor:

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

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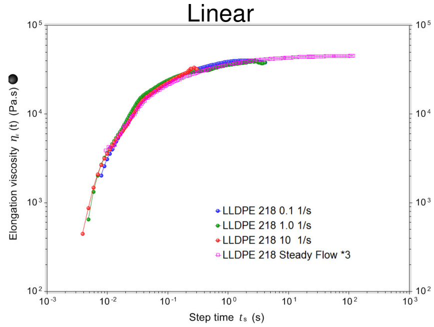
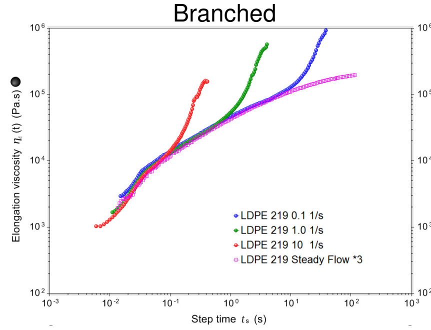
Extensional Viscosity

- 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





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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

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UV LED Curing Accessory

- Mercury bulb alternative technology
- 365 nm wavelength with peak intensity of 150 mW/cm^2
- 455 nm wavelength with peak intensity of 350 mW/cm^2
- No intensity degradation over time
- Even intensity across plate diameter
- Compact and fully integrated design including power, intensity settings and trigger
- Cover with nitrogen purge ports
- Optional disposable Acrylic plates

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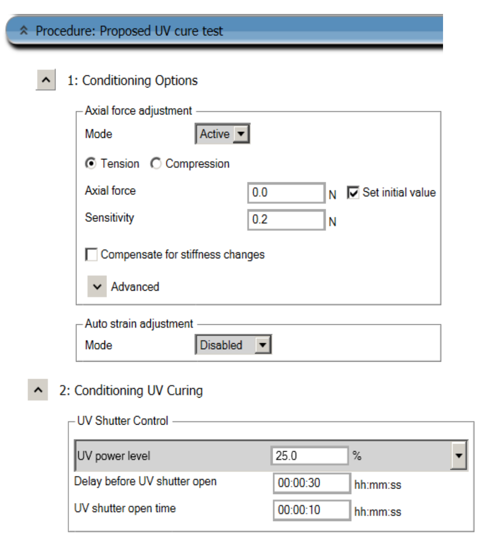
UV Curing Procedure



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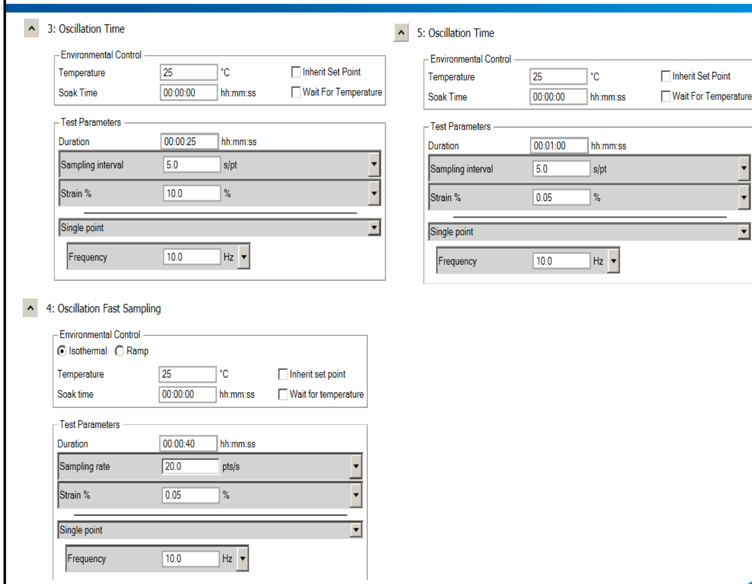
UV Curing Procedure



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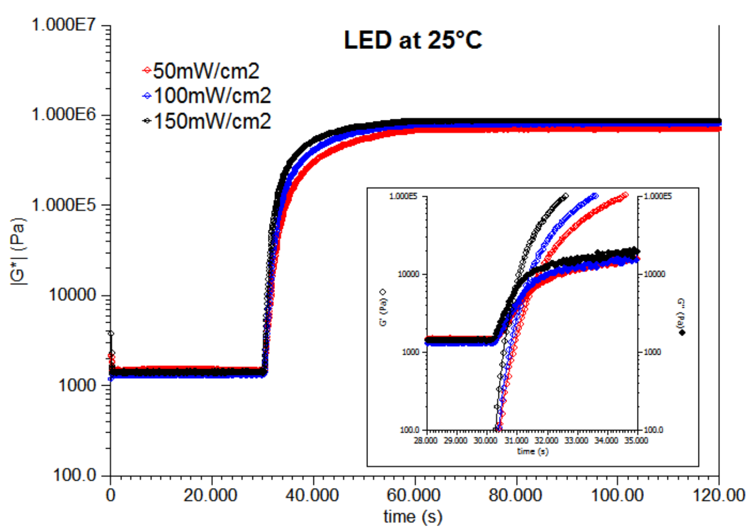
UV Cure Profile Changes with Intensity



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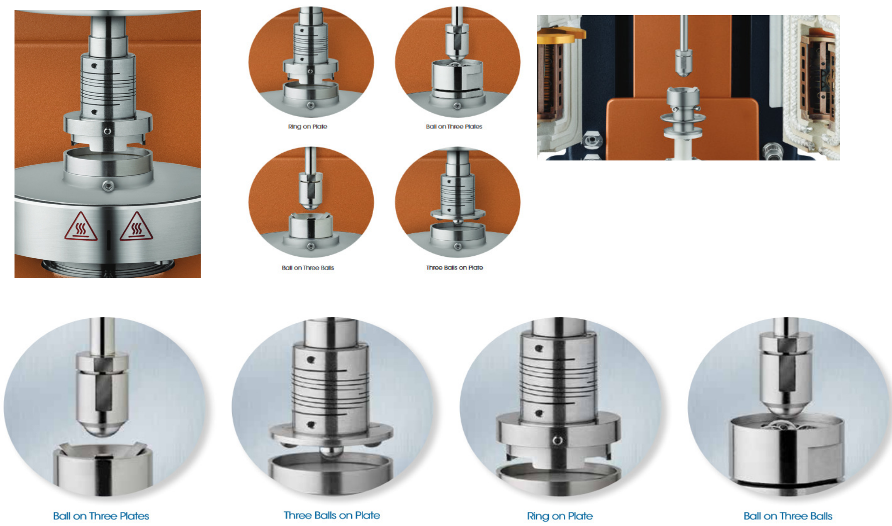
Tribology



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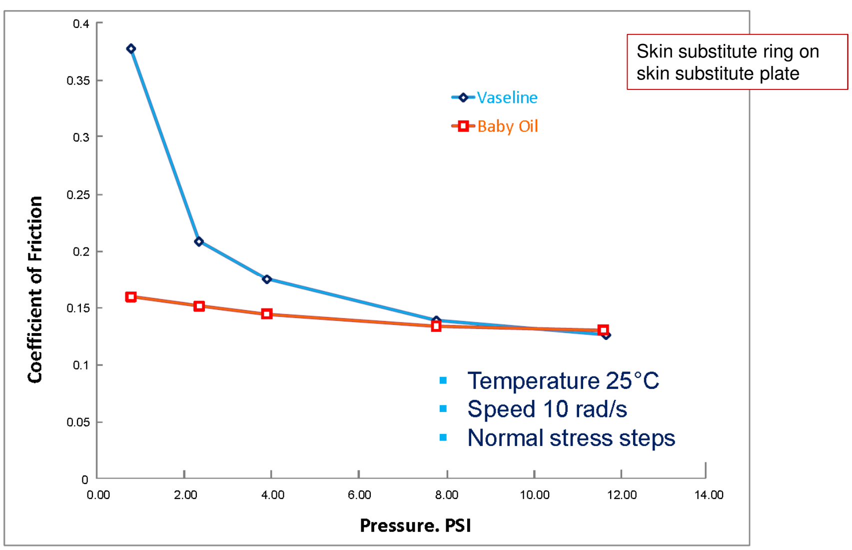
COF – Single Speed Tests



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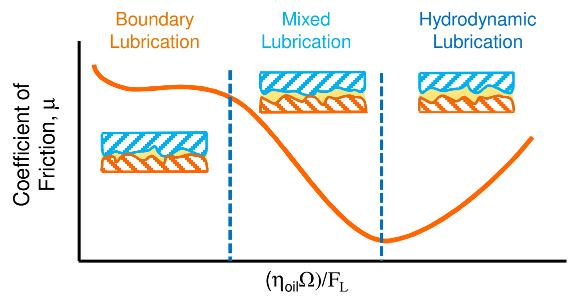
Tribology of Lubricated Systems



- 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)

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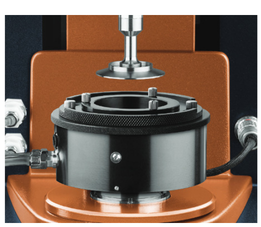
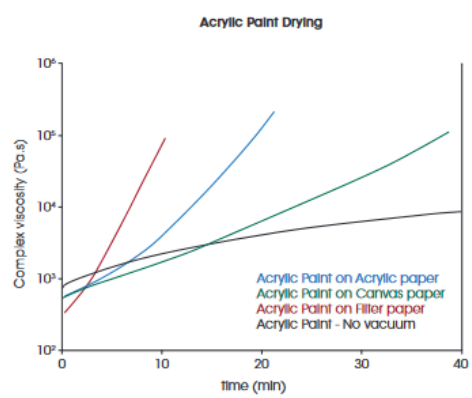
DHR Immobilization Cell





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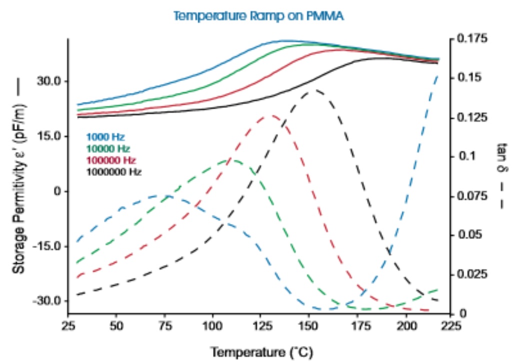
DETA on DHR and ARES-G2



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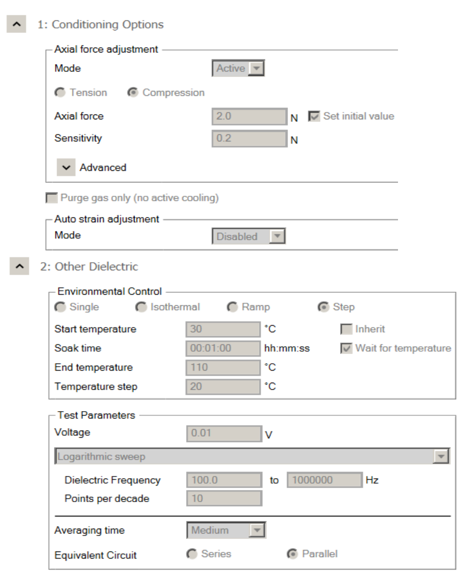
Dielectric Example in Trios



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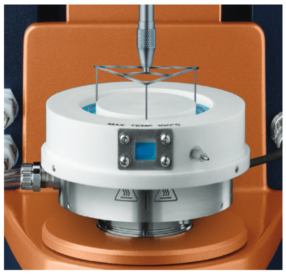
Interfacial Rheology



Bicone

Double Wall Du Noüy Ring (DDR)

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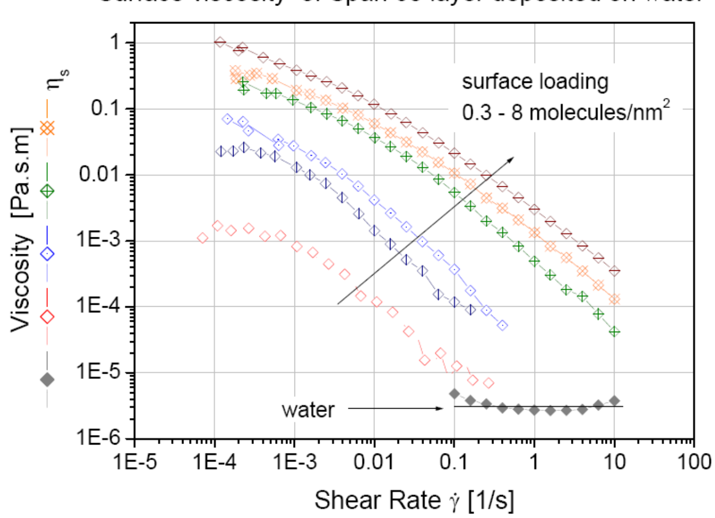
Surface Concentration Effects on Interfacial Viscosity

Surface viscosity of Span 65 layer deposited on water



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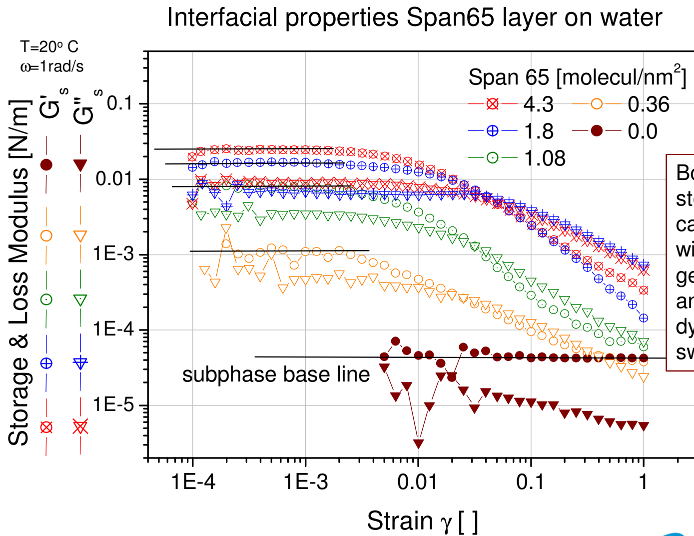
SPAN65® Layer Spread on Water



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

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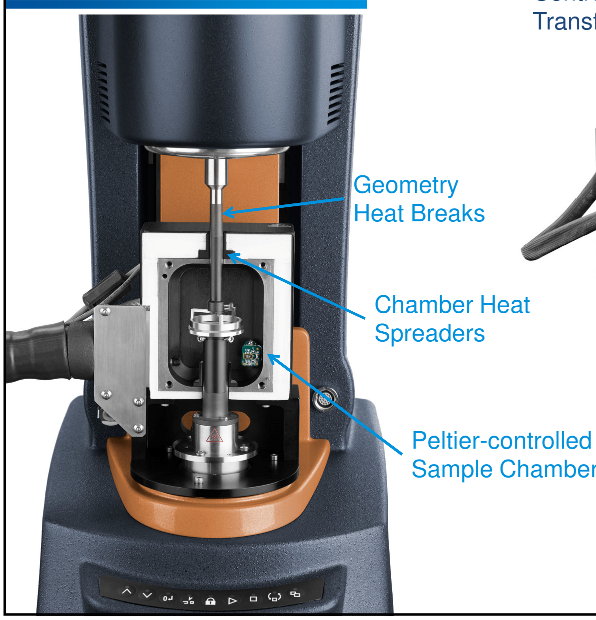
DHR-RH Accessory Technology



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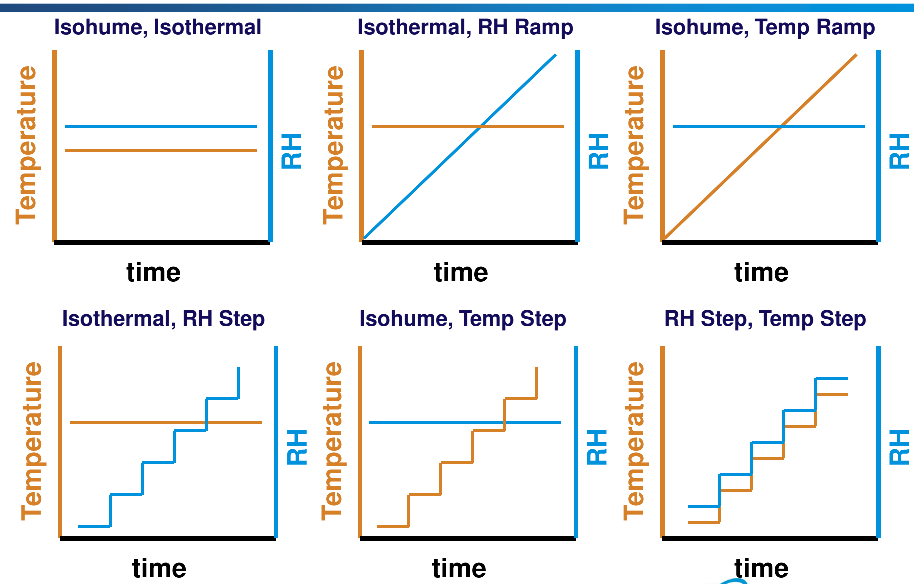
DMA-RH Experimental Options



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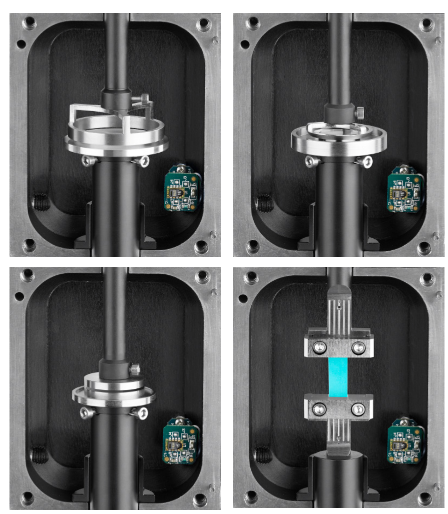
Test Geometries

- 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



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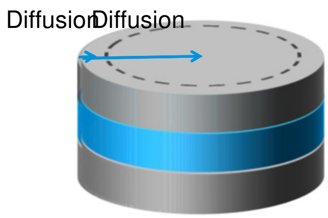
Annular Ring Geometry



- Parallel plate: long diffusion length
- Remove center section
- Annular ring: short diffusion length
- Provides quantitative bulk rheology with less delay and gradients associated with diffusion

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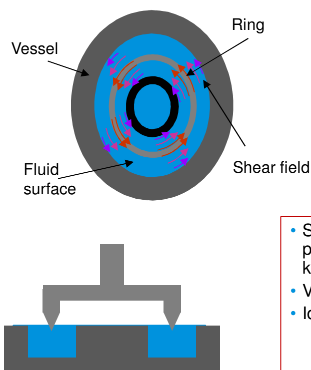
Surface Diffusion Geometry



- Surface rheological properties and process kinetics
- Very simple sample loading
- Ideal for:
  - Fast evolving systems
  - Samples with shallow interaction depth
  - Drying, curing…

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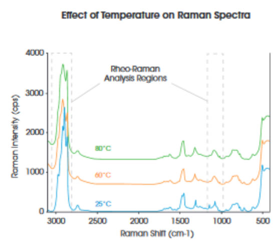
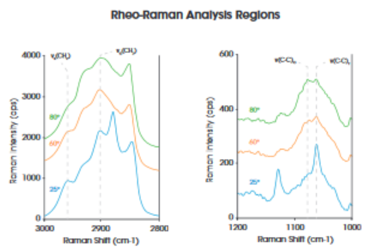
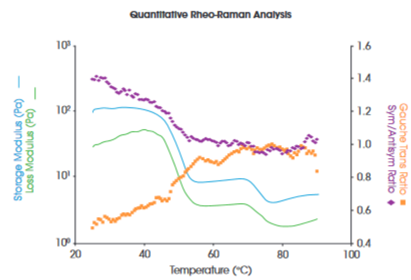
Rheo-Raman Accessory



Rheo-Raman on a hand lotion





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Need Some Assistance?

- Instrument Manuals
- Help Feature in Trios
- TA Instruments website
- TA Instruments Rheology Helpline
  - rsupport@tainstruments.com

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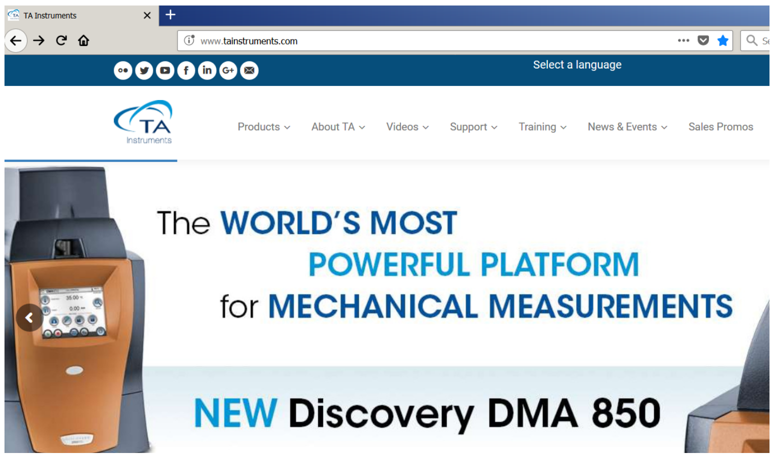
Website: www.tainstruments.com



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Site Preparation Guides	Tech Tips	Instruments sorted by software	Premium Support Plan
The IQ/OQ Product Offering	Applications Notes Library	Software Sorted by Instruments	Plus Support Plan
Calibration with Certified Standards	Training	Report a Bug	Basic Support Plan
Safety Data Sheets		Request a Feature	Performance Maintenance Visit (PMV)
Supported Instruments			Academic Support Plan
Service Shop			ElectroForce Support Plans

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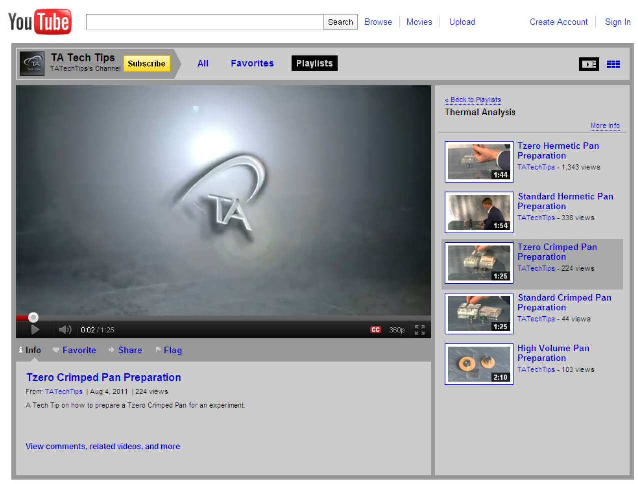
TA Tech Tips



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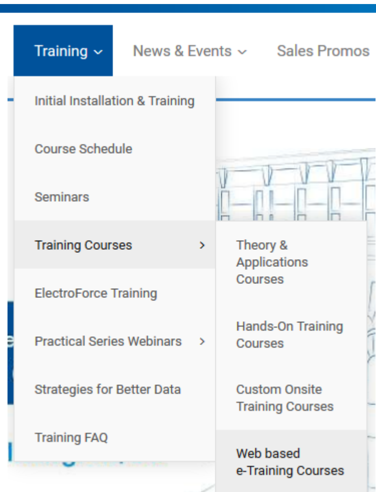
On Line Training



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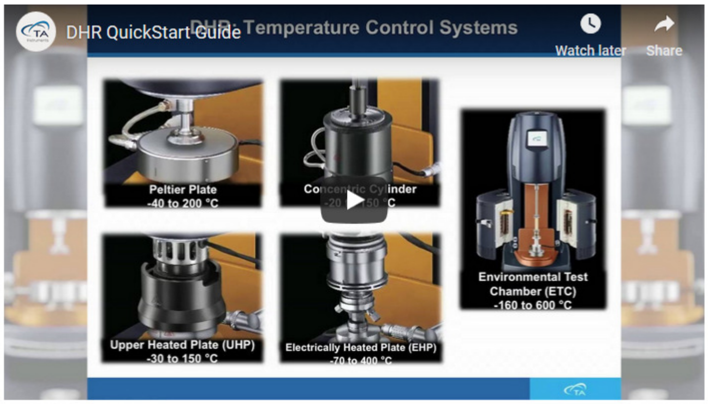
DHR Quick Start Guide



TRIOS QuickStart Guide – Basic Data Analysis Applications in Rheology

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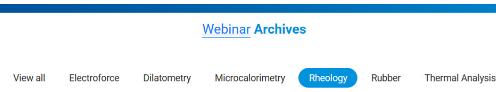
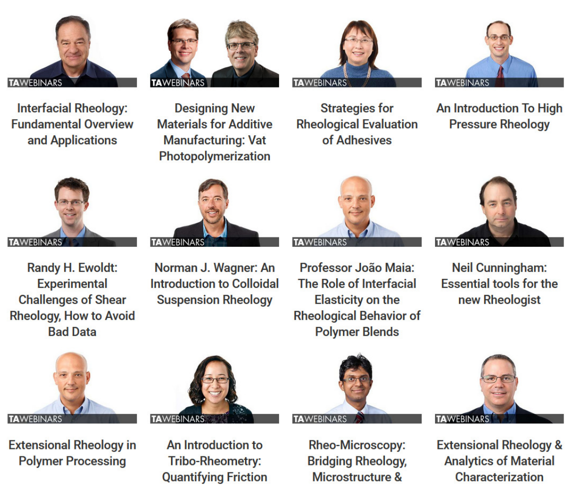
Sampling of Available Webinars





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Thank You

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