RHEOLOGY – FROM LIQUIDS TO SOLIDS

Topics in the Waters – TA Characterization Workshops

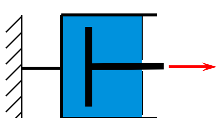
- Optimizing DSC and TGA Testing
- GPC Techniques and Applications
- Rheology from Liquids to Solids
- DMA: Rheology of Solid-Like Materials

Rheology: An Introduction

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

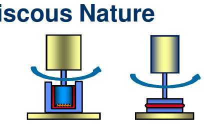
TAINSTRUMENTS.COM

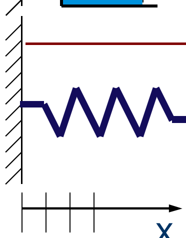
Rheology: The study of the flow and deformation of matter



Flow: Fluid Behavior; Viscous Nature

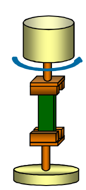
F = F(v); F ≠ F(x)

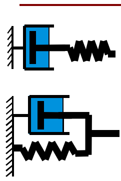




Deformation: Solid Behavior
Elastic Nature

F = F(x); F ≠ F(v)



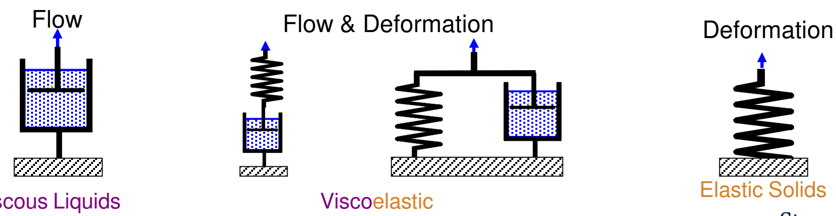


Viscoelastic Materials: Force depends on both Deformation and Rate of Deformation and vice versa.

TAINSTRUMENTS.COM

Basic Material Behaviors

Water | Oil | Soap | Egg white | Polymer Melt | Ceramic ? | Metal



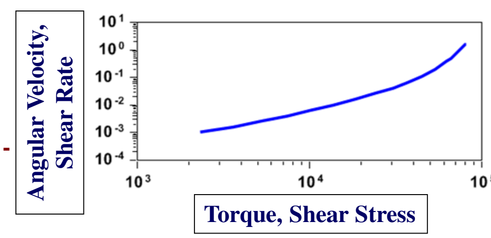
$$\text{Viscosity} = \frac{\text{Stress}}{\text{Strain Rate}}$$

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

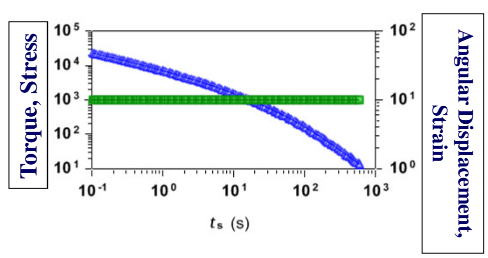
TA | TAINSTRUMENTS.COM

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)**



TA | TAINSTRUMENTS.COM

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.

TAINSTRUMENTS.COM

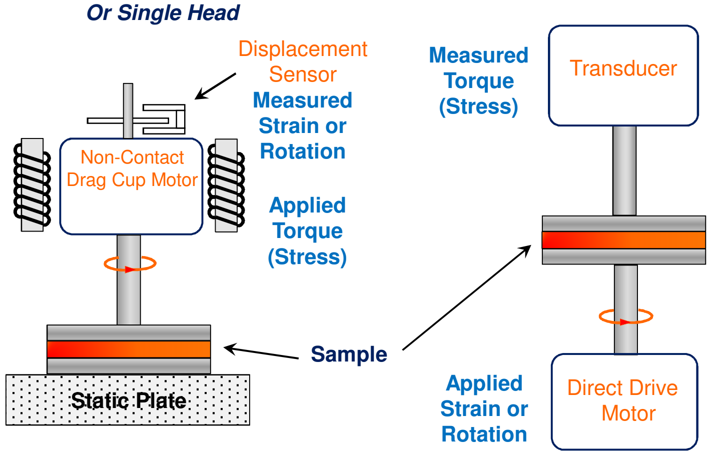
Rotational Rheometer Designs

DHR

Combined motor & transducer Or Single Head

ARES

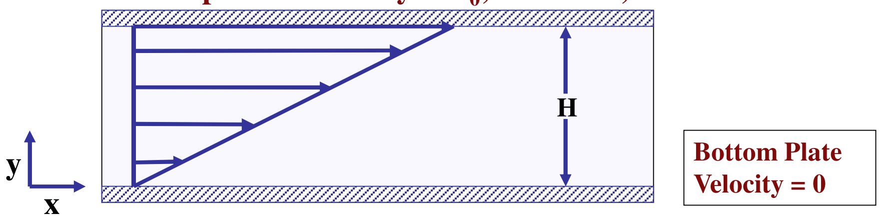
Separate motor & transducer Or Dual Head



TAINSTRUMENTS.COM

Steady Simple Shear Flow

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

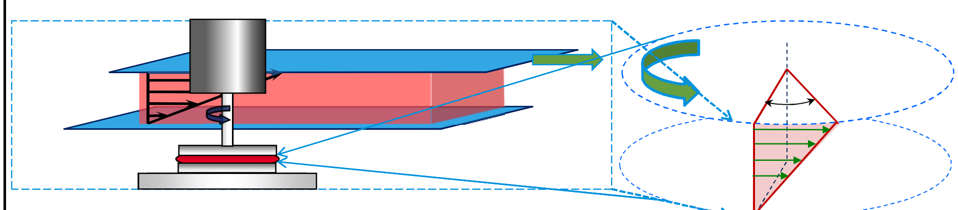


$v_x = (y/H)*V_0$

$\dot{\gamma} = dv_x/dy = V_0/H$	Shear Rate, sec^{-1}
$\sigma = F/A$	Shear Stress, Pascals
$\eta = \sigma/\dot{\gamma}$	Viscosity, Pa-sec

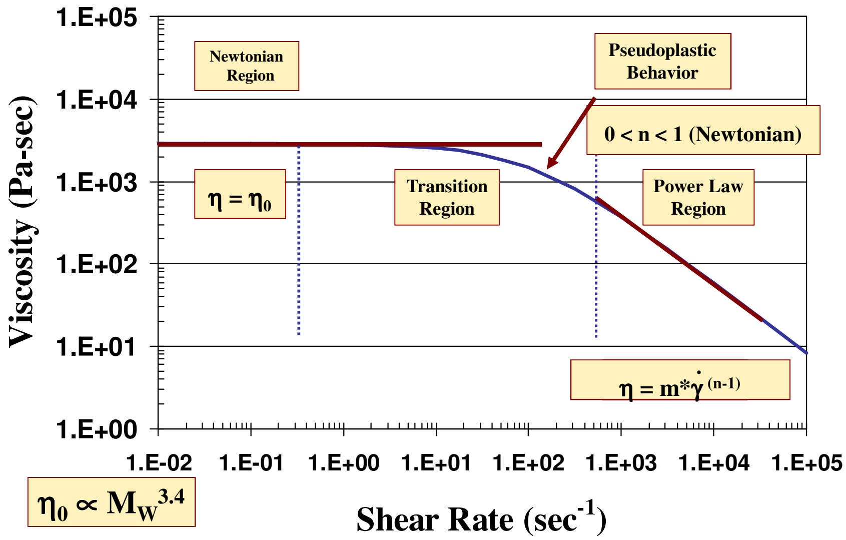
➢These are the fundamental flow parameters. Shear rate is always a change in velocity with respect to distance.

Parallel Plate Shear Flow



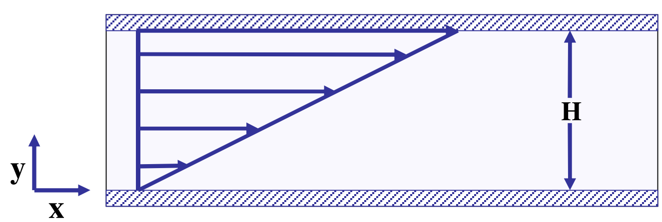
Stress	σ	Force or torque
Strain	γ	Linear or angular displacement
Strain Rate	$\dot{\gamma}$	Linear or angular velocity

Representative Flow Curve



Simple Shear Deformation

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



Bottom Plate Displacement = 0

$x = (y/H)*X_0$

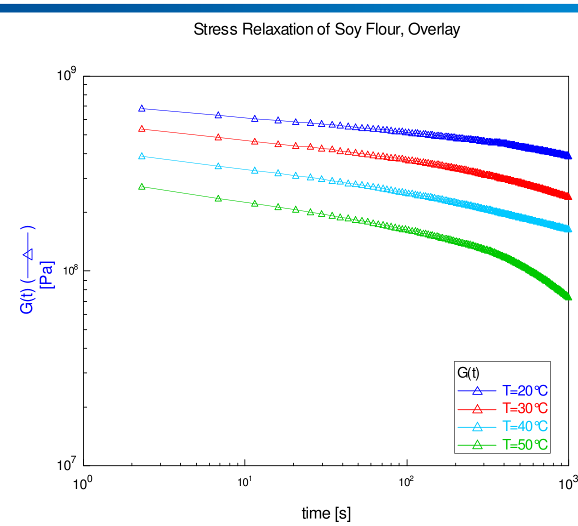
$\gamma = dx_x/dy = X_0/H$ Shear Strain, unitless

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

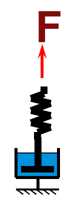
$G = \sigma/\gamma$ Modulus, Pa

➢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.



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

Rheological Parameters

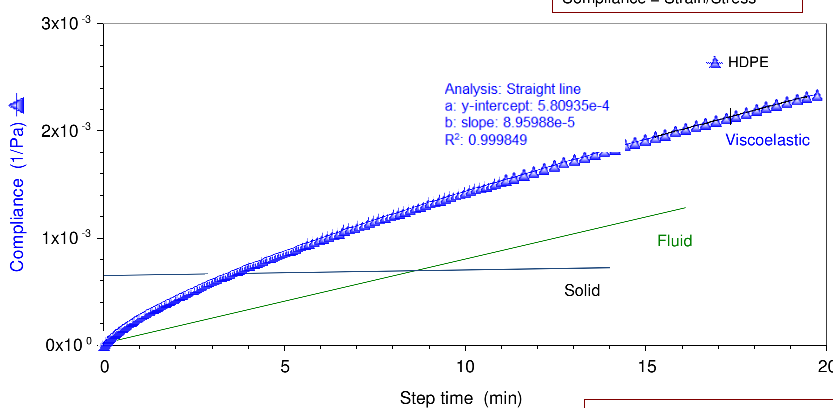
CREEP TESTING

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

TAINSTRUMENTS.COM

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

TAINSTRUMENTS.COM

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

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

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

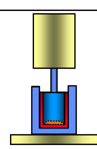
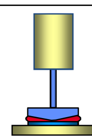
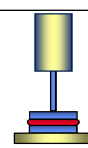
Conversion Factors
K_σ : Stress Conversion Factor
K_γ: Strain (Rate) Conversion Factor

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.

TAINSTRUMENTS.COM

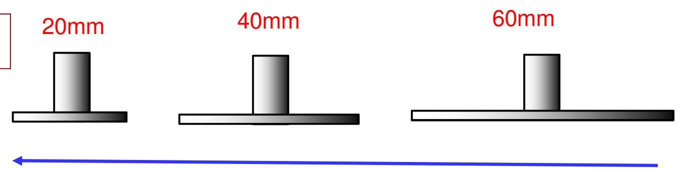
Shear Rate and Shear Stress Calculations

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

TAINSTRUMENTS.COM

Cone & Plate and Parallel Plate Geometric Considerations

Standard DHR Peltier plate geometry diameters



Shear Stress

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

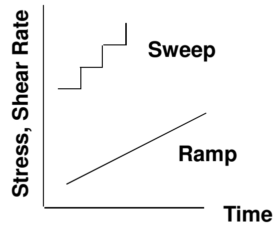
So, for low viscosity fluids, use the largest diameter cone or plate.
For high viscosity fluids, use the smallest diameter cone or plate

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.

Rheological Methods – Unidirectional Testing

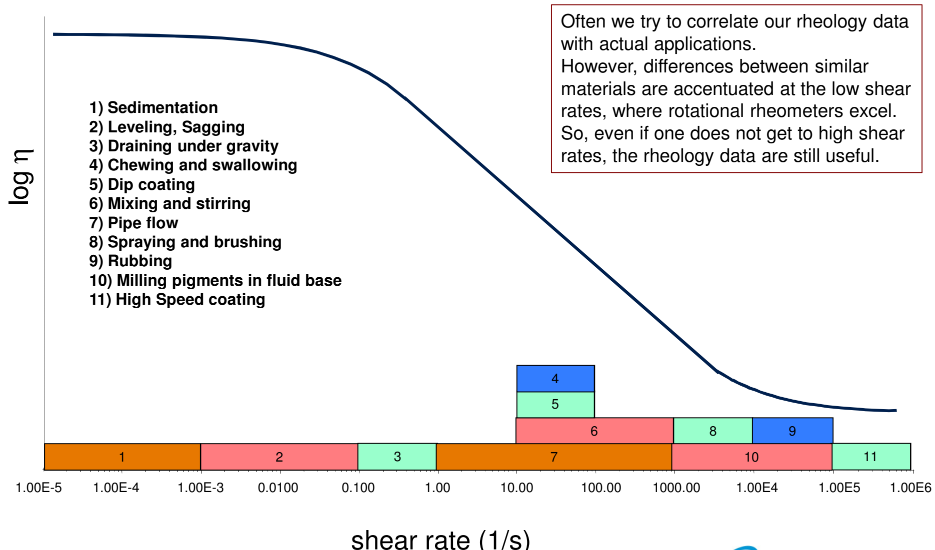
- Flow
  - Stress/Rate Ramp
  - Stress/Rate Sweep
  - Time sweep/Peak Hold/Stress Growth
  - Temperature Ramp
- Creep (constant stress)
- Stress Relaxation (constant strain)



TA | TAINSTRUMENTS.COM

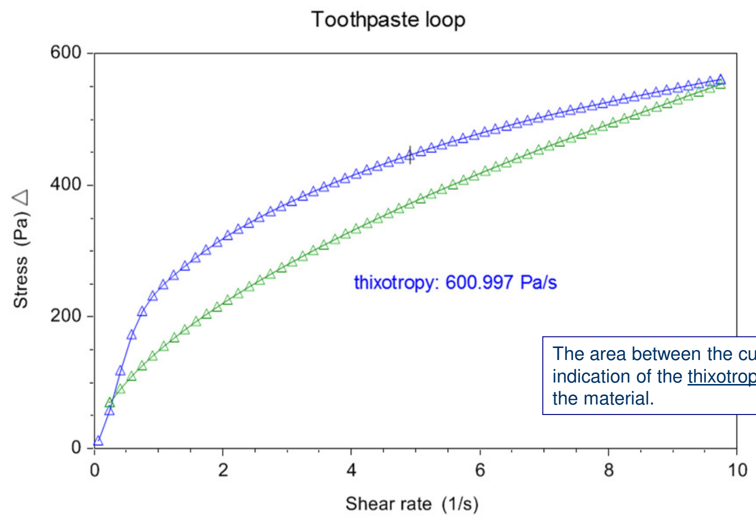
Idealized Flow Curve

Often we try to correlate our rheology data with actual applications.
However, differences between similar materials are accentuated at the low shear rates, where rotational rheometers excel.
So, even if one does not get to high shear rates, the rheology data are still useful.



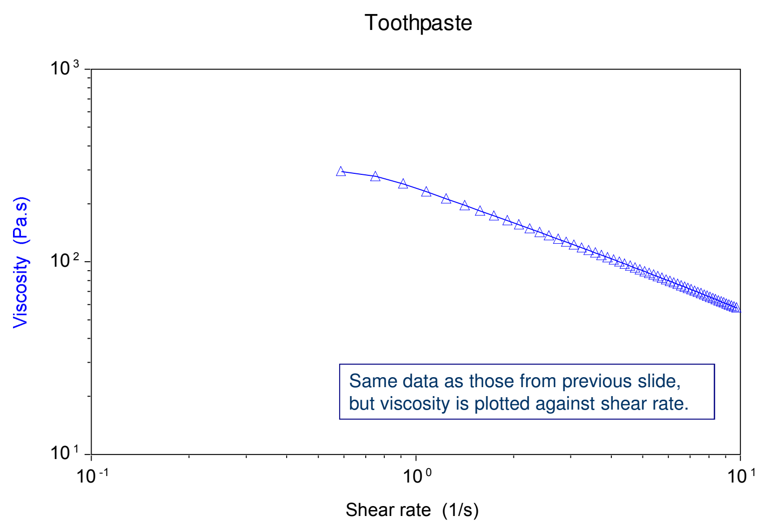
TA | TAINSTRUMENTS.COM

Thixotropy: Up & Down Flow Curves



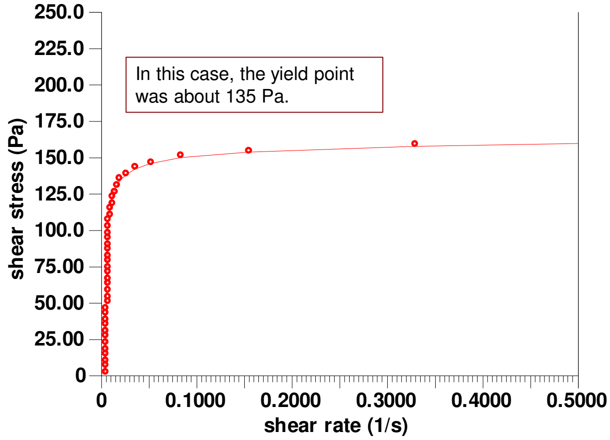
The area between the curves is an indication of the thixotropic nature of the material.

Rate Ramp



Same data as those from previous slide, but viscosity is plotted against shear rate.

Stress Ramp with Yield



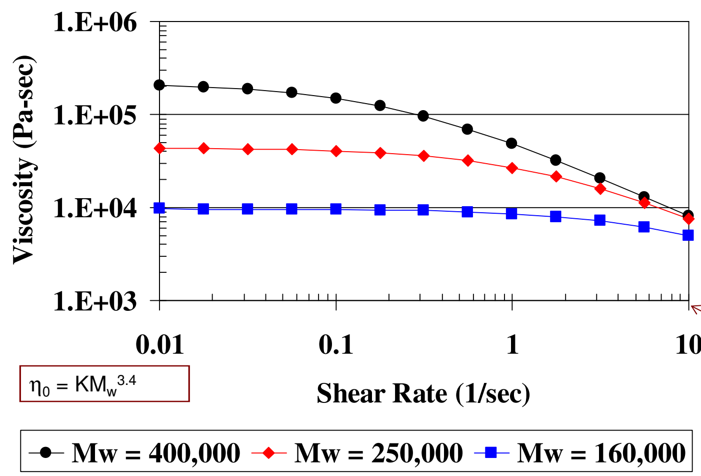
TAINSTRUMENTS.COM

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.

TAINSTRUMENTS.COM

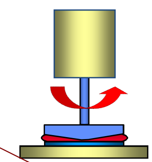
Rate Sweeps - Polymer Melt Flow Curves



$\eta_0 = KM_w^{3.4}$

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.

TAINSTRUMENTS.COM

Structured fluids

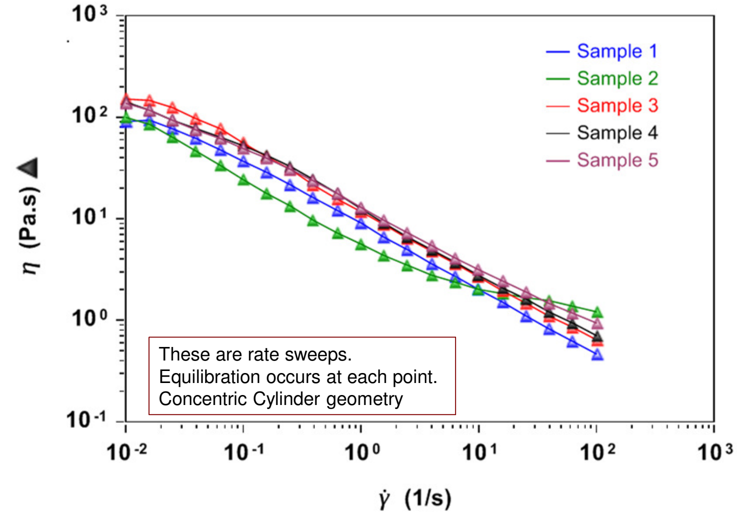
Structured fluids are mostly colloidal dispersions and can be classified into three categories.

- **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

TAINSTRUMENTS.COM

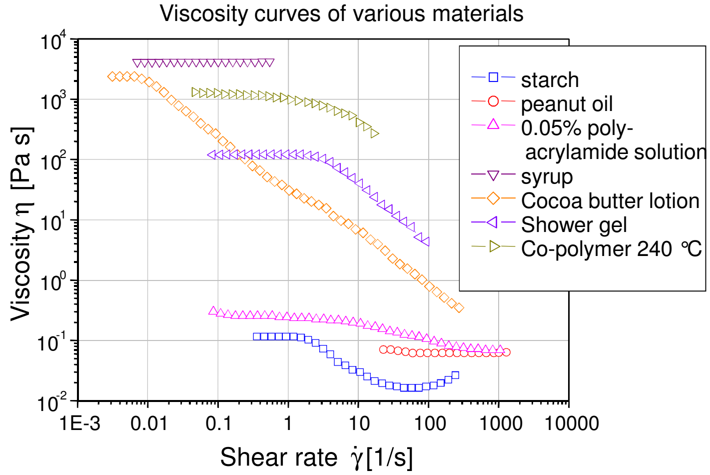
Steady State Flow at 25 C - Coatings



TA | TAINSTRUMENTS.COM

Viscosity curve of various structured fluids

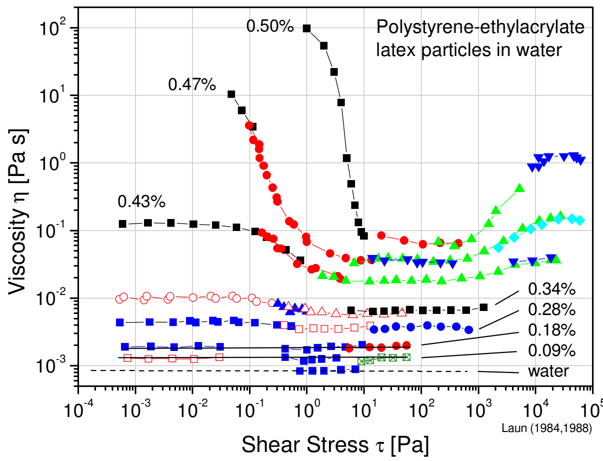
Viscosity curves of various materials



Viscosity function of various structured fluids

TA | TAINSTRUMENTS.COM

Viscous response of suspensions

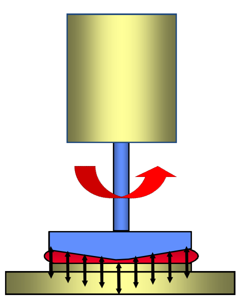


Rheological parameters of interest:Yield stress, viscosity, time dependence, linear viscoelasticity

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

TAINSTRUMENTS.COM

Normal Force Measurements with Cone & Plate

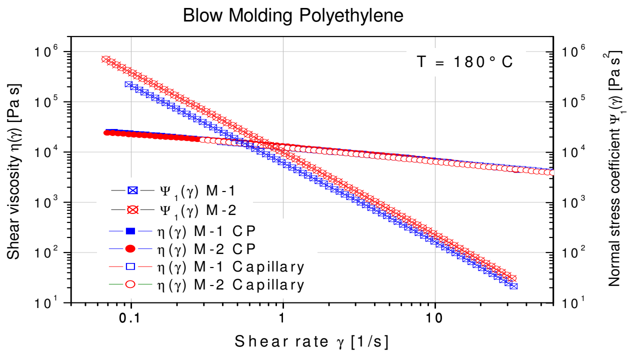


Normal Stress Difference:

- **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.**

TAINSTRUMENTS.COM

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

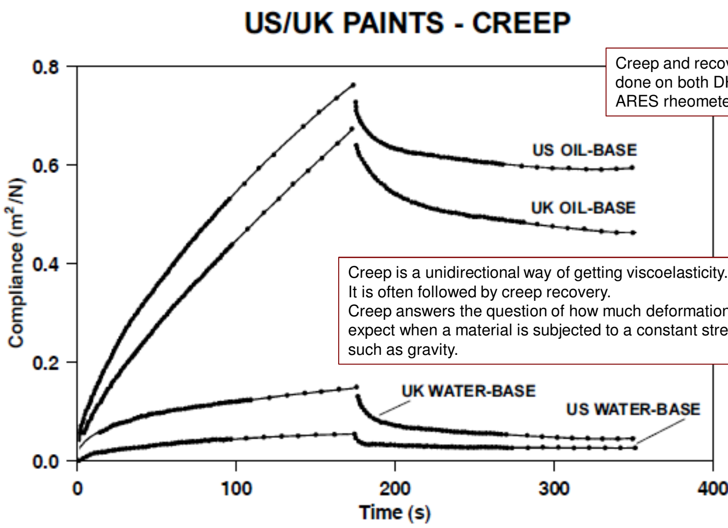
No differences in MFI, Viscosity, or GPC!

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

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.

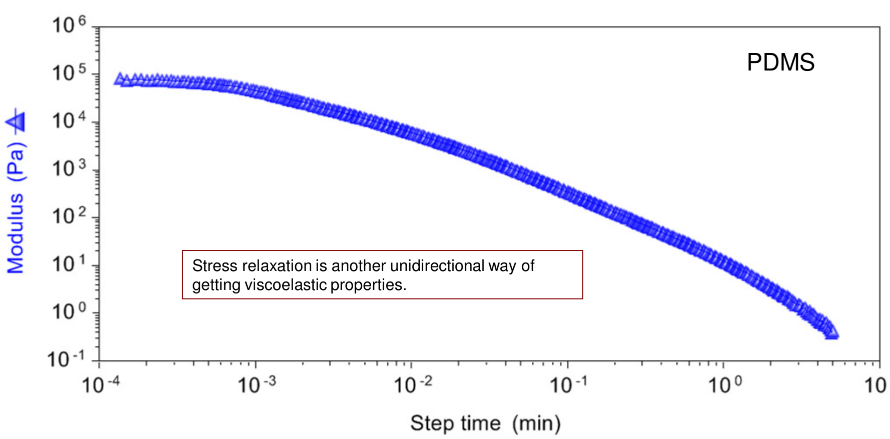
Creep Testing on US and UK Paints



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

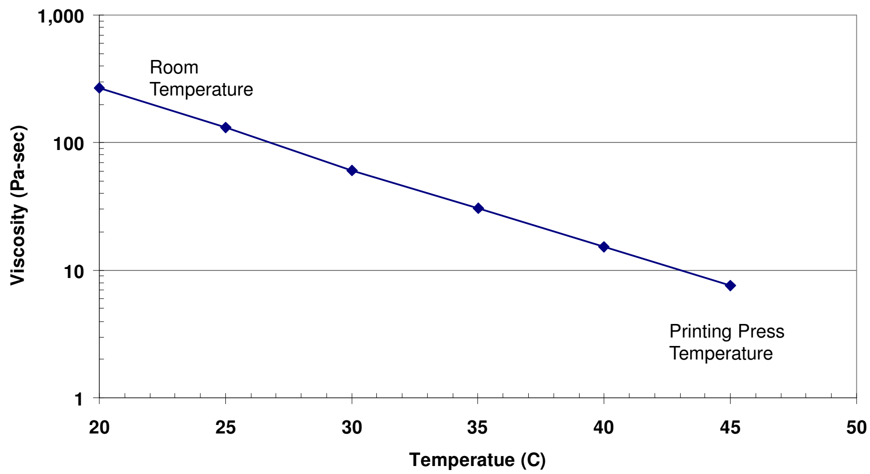
Creep is a unidirectional way of getting viscoelasticity.
It is often followed by creep recovery.
Creep answers the question of how much deformation to expect when a material is subjected to a constant stress, such as gravity.

Stress Relaxation

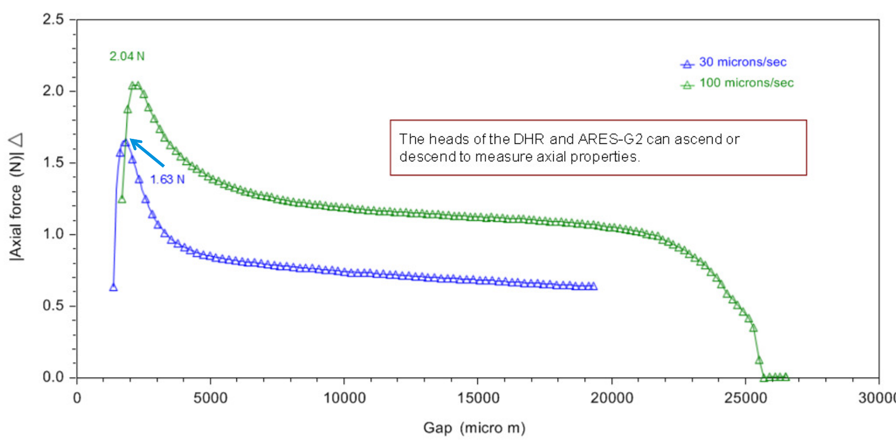


Stress relaxation is another unidirectional way of getting viscoelastic properties.

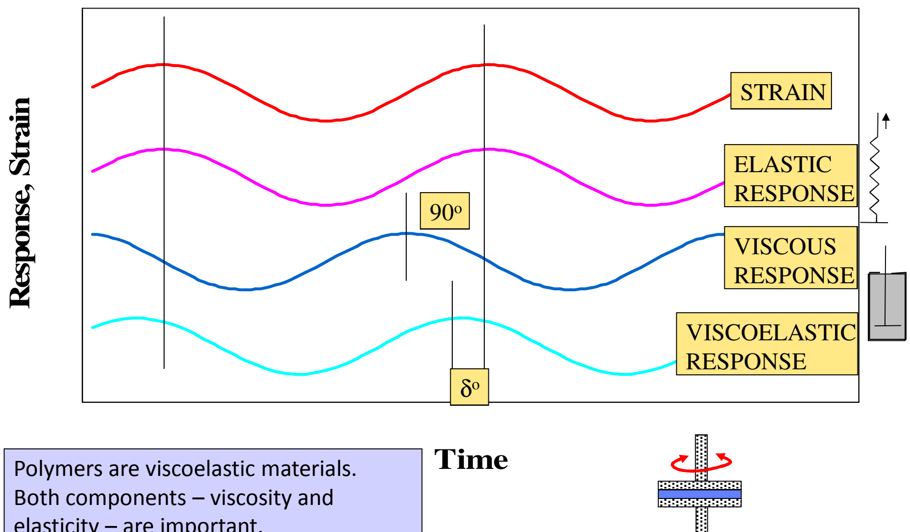
Flow Temperature Ramp – Printing Inks



Tack Testing



Dynamic Testing



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

TAINSTRUMENTS.COM

Dynamic Rheological Parameters

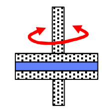
Parameter	Shear	Elongation	Units
Strain	$\gamma = \gamma_0 \sin(\omega t)$	$\varepsilon = \varepsilon_0 \sin(\omega t)$	---
Stress	$\sigma = \sigma_0 \sin(\omega t + \delta)$	$\tau = \tau_0 \sin(\omega t + \delta)$	Pa
Storage Modulus (Elasticity)	$G' = (\sigma_0/\gamma_0)\cos\delta$	$E' = (\tau_0/\varepsilon_0)\cos\delta$	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: $\eta^*(\omega) = \eta(\dot{\gamma})$ @ $\dot{\gamma} = \omega$

TAINSTRUMENTS.COM

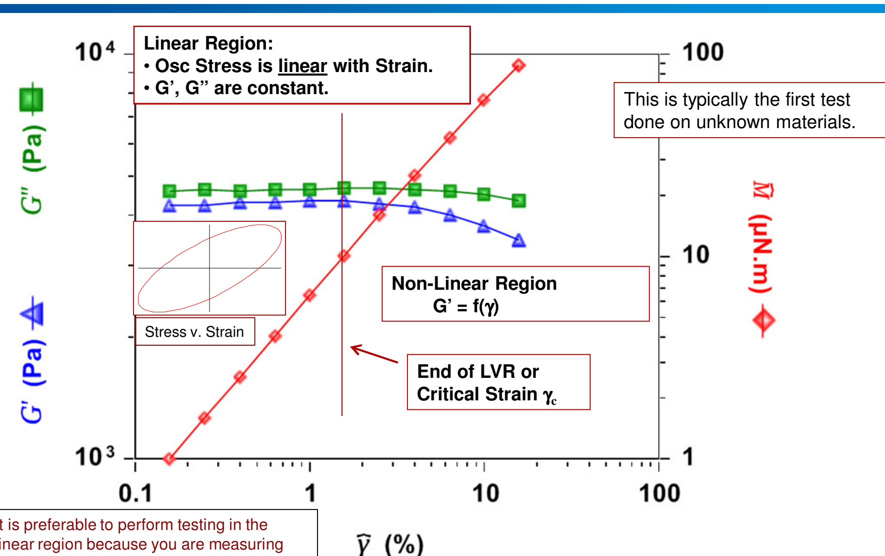
Rheological Methods – Dynamic Oscillatory Testing

- 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.



TA | TAINSTRUMENTS.COM

Linear Viscoelasticity

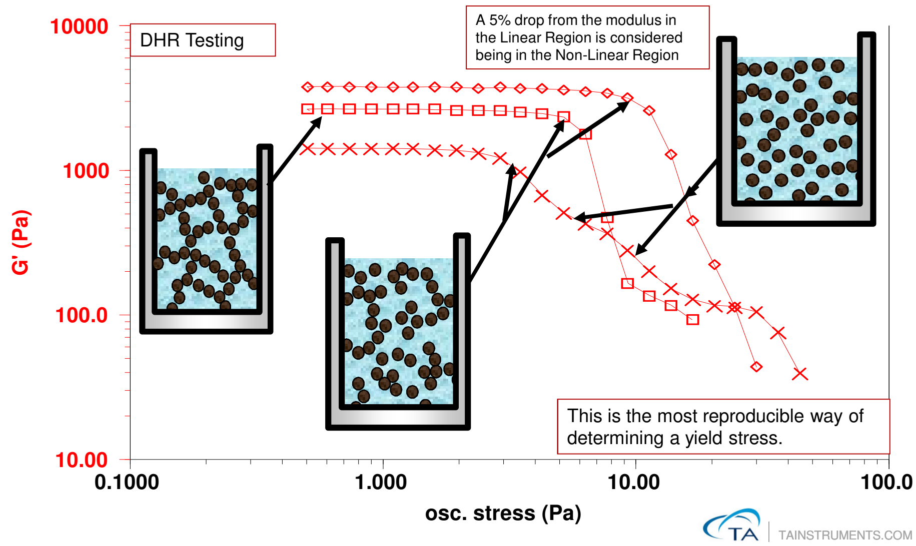


It is preferable to perform testing in the linear region because you are measuring intrinsic properties of the material, not material that has been altered.

TA | TAINSTRUMENTS.COM

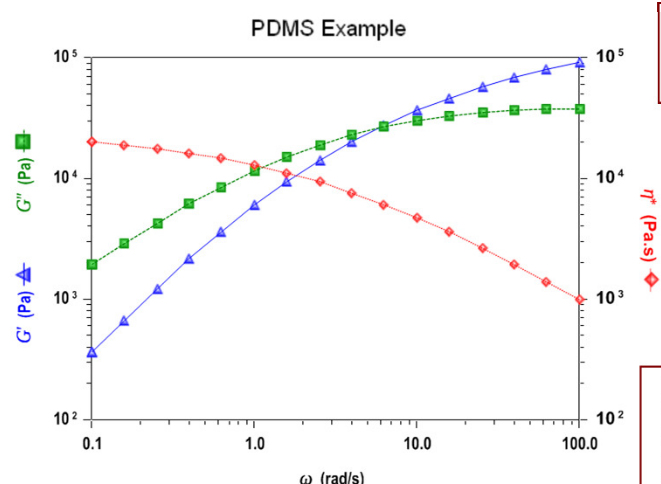
Stability is Related to Structure in Inks

Ink Samples: Oscillation Stress Sweeps @ 6.28 rad/s



TA | TAINSTRUMENTS.COM

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

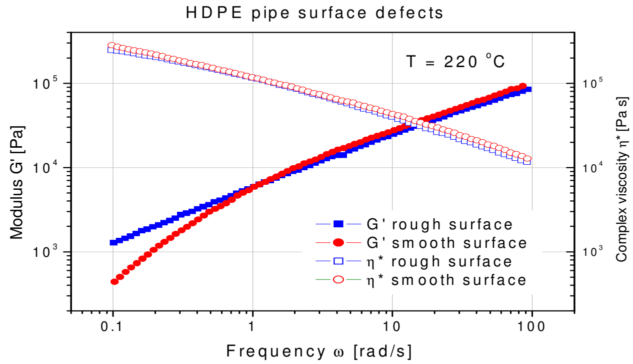
100 to 0.1 rad/sec
5 pts. per decade
3 minutes of equilibration
Frequency sweep takes about 6.5 min.

TA | TAINSTRUMENTS.COM

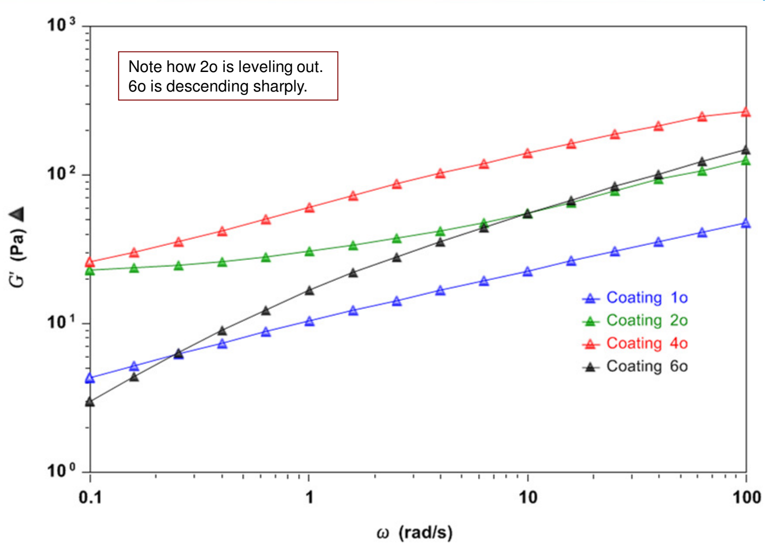
Surface Defects during Pipe Extrusion

Indicated by Elasticity at low frequency

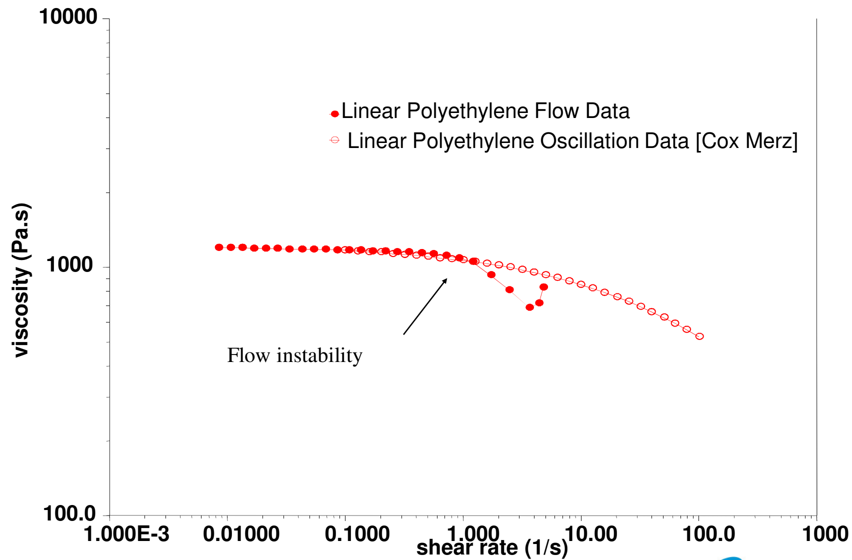
Caused by Broader MWD



Coatings Frequency Sweep

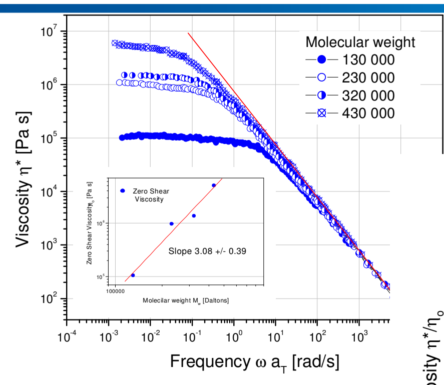


Example of Cox-Merz Rule



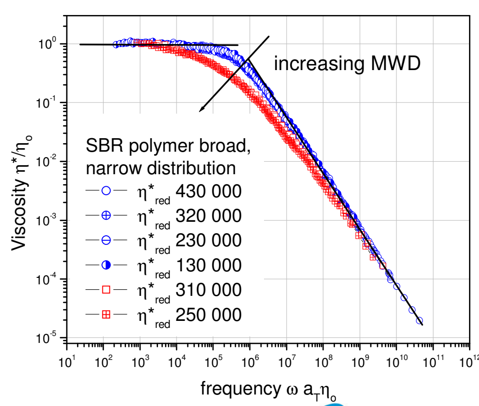
TA | TAINSTRUMENTS.COM

Dynamic testing: Dependence on M_w and MWD



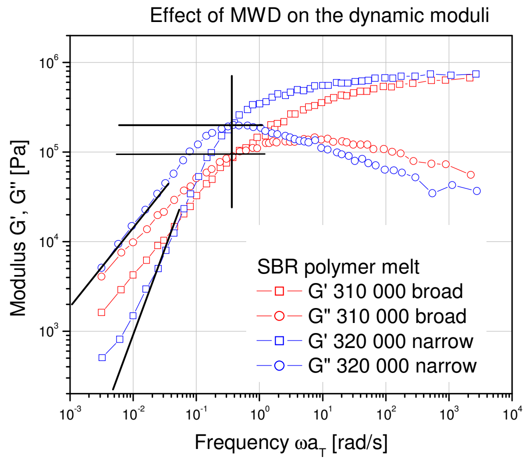
Zero shear viscosity increases with increasing MW

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



TA | TAINSTRUMENTS.COM

MWD and Dynamic Moduli



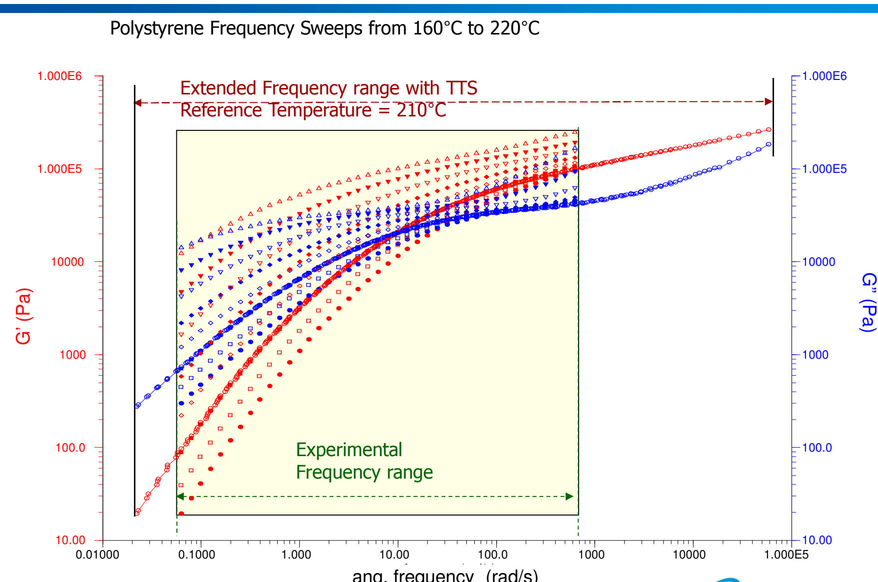
- **The storage and loss modulus of a typical polymer melt cross over between 1 and 100 rad/s.**
- **$\omega_c \propto 1/M_w$.**
- **$G_c \propto 1/MWD$.**

Higher crossover frequency = lower M_w

Higher crossover Modulus = narrower MWD

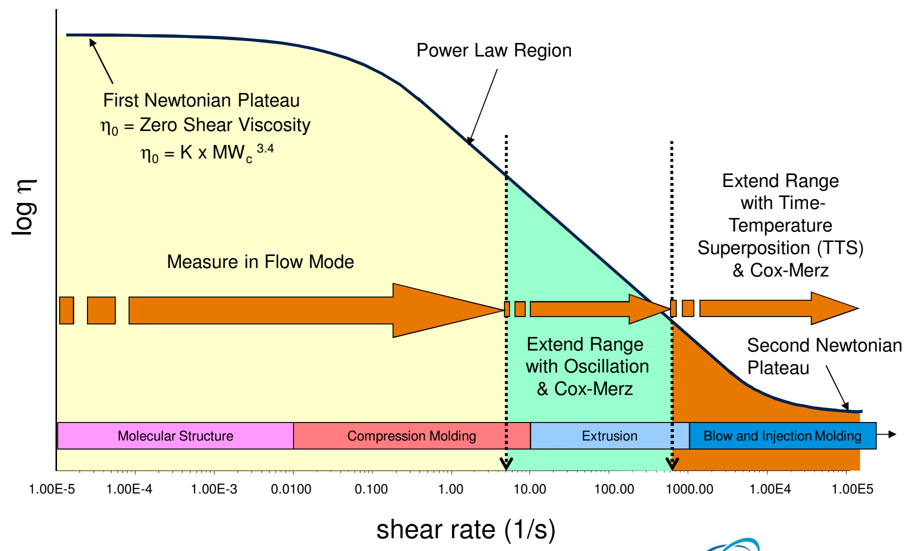
TAINSTRUMENTS.COM

ETC Application: TTS on Polymer Melt



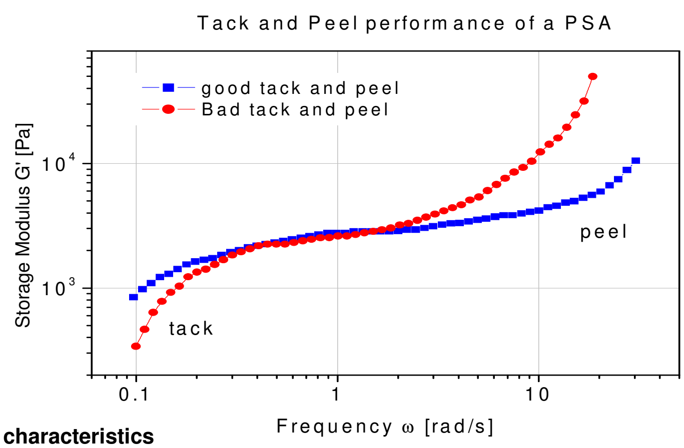
TAINSTRUMENTS.COM

Idealized Flow Curve - Polymers



TA | TAINSTRUMENTS.COM

Tack and Peel of Adhesives



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

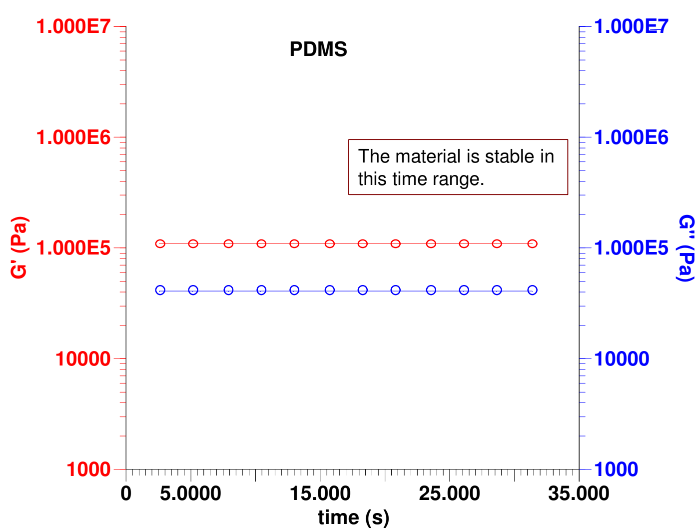
TA | TAINSTRUMENTS.COM

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

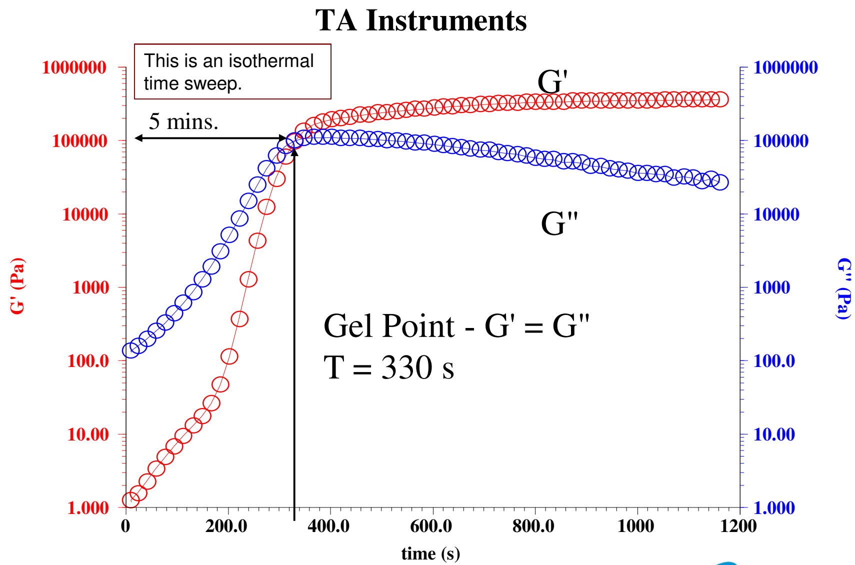
TA | TAINSTRUMENTS.COM

Dynamic Time Sweep: Material Response



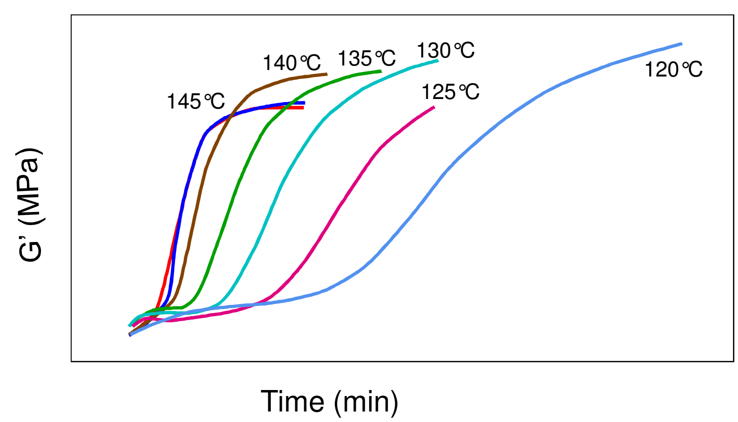
TA | TAINSTRUMENTS.COM

Cure of a "5 minute" Epoxy



TAINSTRUMENTS.COM

Isothermal Cure of Tire Compound Effect of Curing Temperature



TAINSTRUMENTS.COM

Dynamic time sweep following shear

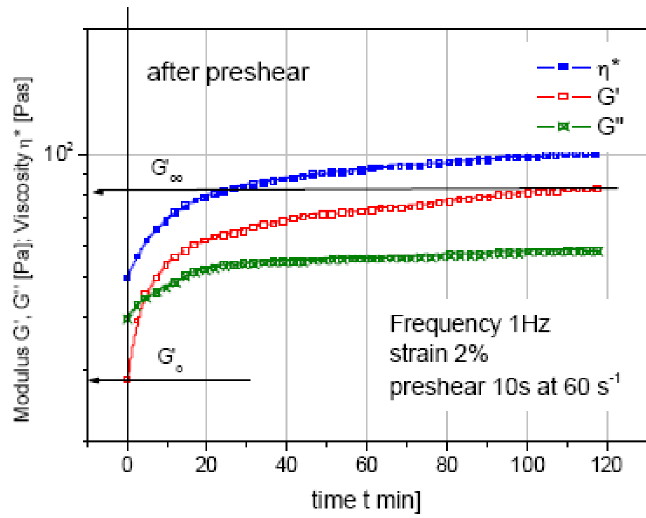
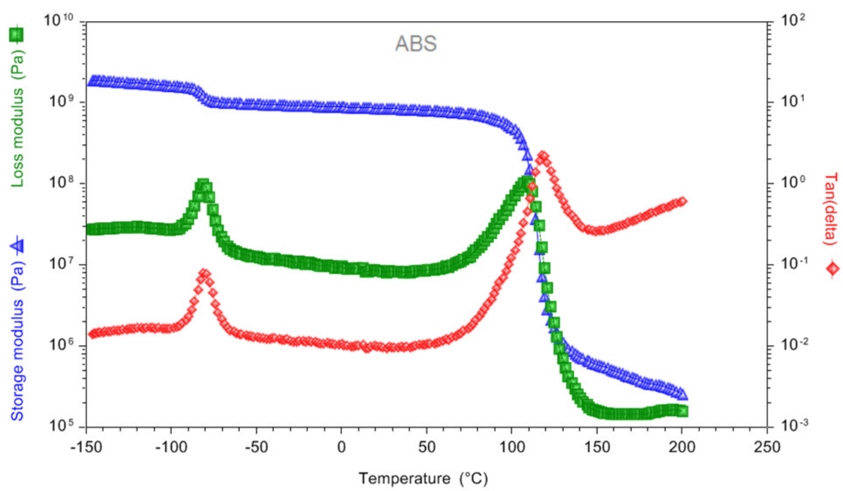


Figure 7: Recovery of structure

This shows how rapidly the structure is rebuilding.

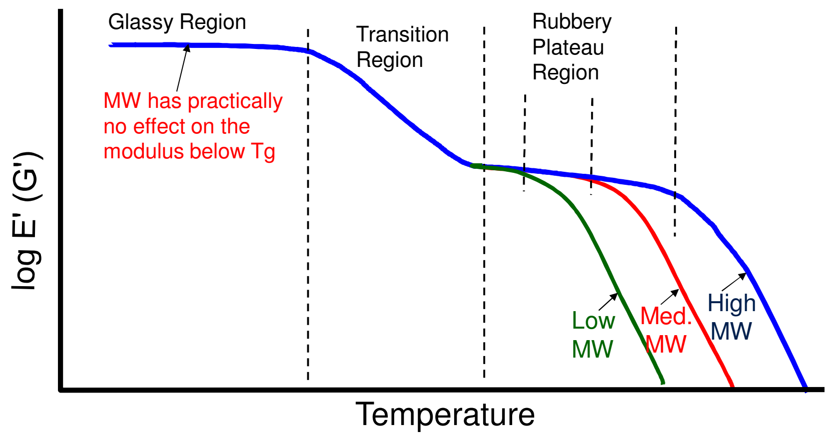
TAINSTRUMENTS.COM

Dynamic Temperature Ramp - Torsion



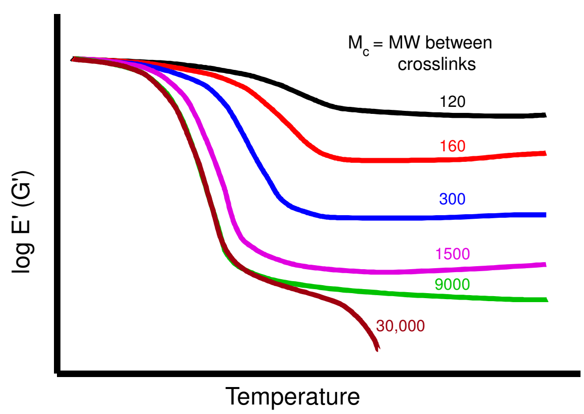
TAINSTRUMENTS.COM

Effect of Molecular Weight



TAINSTRUMENTS.COM

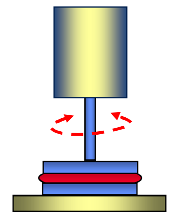
Effect of Crosslinking



TAINSTRUMENTS.COM

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.



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.

DHR Instrument 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 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		

ARES-G2 Specifications

Force/Torque Rebalance Transducer (Sample Stress)

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

Drive Motor (Sample Deformation)

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

Orthogonal Superposition and DMA modes

Motor Control	Force Rebalance Transducer
Minimum Transducer Force in Oscillation	0.001 N
Maximum Transducer Force	20 N
Minimum Displacement in Oscillation	0.5 μm
Maximum Displacement in Oscillation	50 μm
Displacement Resolution	10 nm
Axial Frequency range	1 x 10^{-5} Hz to 16 Hz

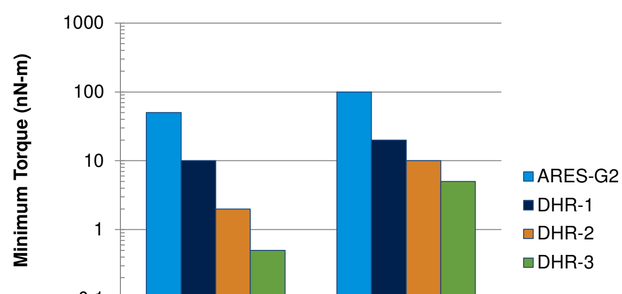
Stepper Motor

Movement/Positioning	Micro-stepping Motor/ Precision Lead Screw
Position Measurement	Linear Optical Encoder
Positioning Accuracy	0.1 micron

Temperature Systems

Smart Swap	Standard
Forced Convection Oven, FCO	-150 °C to 600 °C
FCO Camera Viewer	Optional
Advanced Peltier System, APS	-10 °C to 150 °C
Peltier Plate	-40 °C to 180 °C
Sealed Bath	-10 °C to 150 °C

Minimum Torque Specs

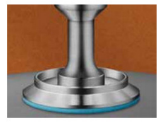


	Oscillation	Steady
ARES-G2	50	100
DHR-1	10	20
DHR-2	2	10
DHR-3	0.5	5

DHR Accessories – Visual Display


Peltier Plate Temperature Systems


Advanced Peltier Plate


Dual Stage Peltier Plate


Upper Heated Plate for Peltier Plate

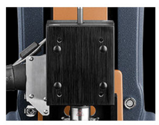

Peltier Concentric Cylinders

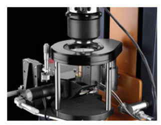

Electrically Heated Cylinder (EHC)

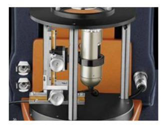

Pressure Cell


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.

TA | TAINSTRUMENTS.COM

DHR Accessories – Visual Display


Small Angle Light Scattering

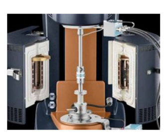

Interfacial Accessories


Tribo-Rheometry Accessory


Magneto-Rheology


Electro-Rheology


UV Curing Accessories


Dielectric Measurement


Immobilization Cell

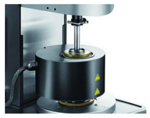

Starch Pasting Cell


Dynamic Mechanical Analysis

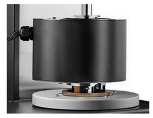
TA | TAINSTRUMENTS.COM

ARES-G2 Accessories

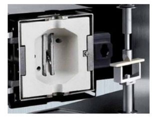
Forced Convection Oven (FCO)



Advanced Peltier System (APS)



Orthogonal Superposition & 2D-SAOS



Dynamic Mechanical Analysis (DMA)

UV Curing Accessory

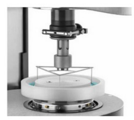
Dielectric Thermal Analysis Accessory (DETA)

Extensional Viscosity Fixture (EVF)

Tribo-Rheometry Accessory

Air Chiller System

Cone and Partitioned Plate Accessory



Interfacial Rheology

Electrorheology (ER) Accessory

TA | TAINSTRUMENTS.COM

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.

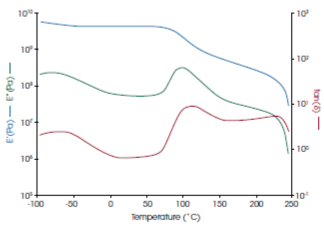
TA | TAINSTRUMENTS.COM


Film Tension


Compression

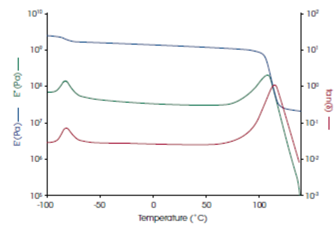

Dual Cantilever

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



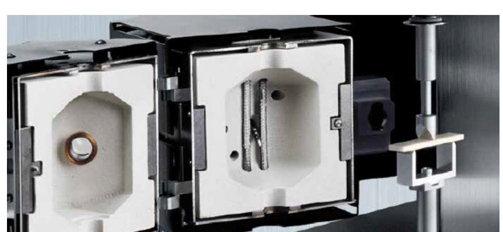
- Acrylonitrile butadiene styrene (ABS)
- Two major transitions
- Tg (butadiene): -82 °C
  Tg (styrene) 115 °C
- Indicates incompatibility of the two monomers

TA | TAINSTRUMENTS.COM

ARES-G2 DMA Mode

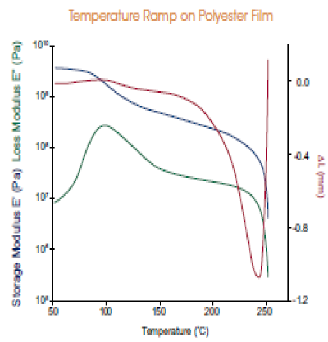
Features and Benefits

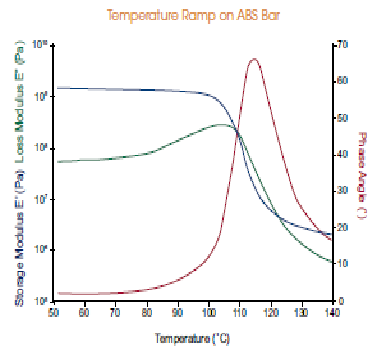
- Exclusive to the ARES-G2 rheometer
- Wide range of geometries:
  - 3-Point Bending
  - Film/Fiber Tension
  - Single and Dual Cantilever (Clamped Bending)
  - Parallel Plates Compression
- Axial Force Control tracks material stiffness and automatically adapts static load
- AutoStrain adjusts applied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



TA | TAINSTRUMENTS.COM

ARES-G2 DMA Testing





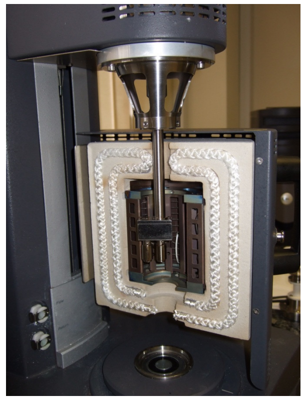
TAINSTRUMENTS.COM

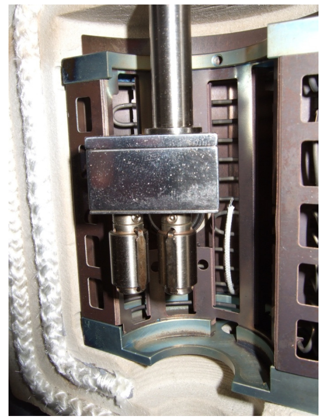
DMA Specifications

	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

TAINSTRUMENTS.COM

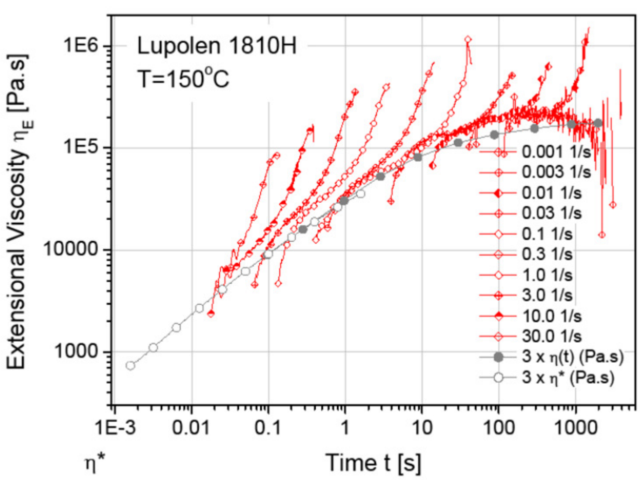
SER2 for DHR Rheometers



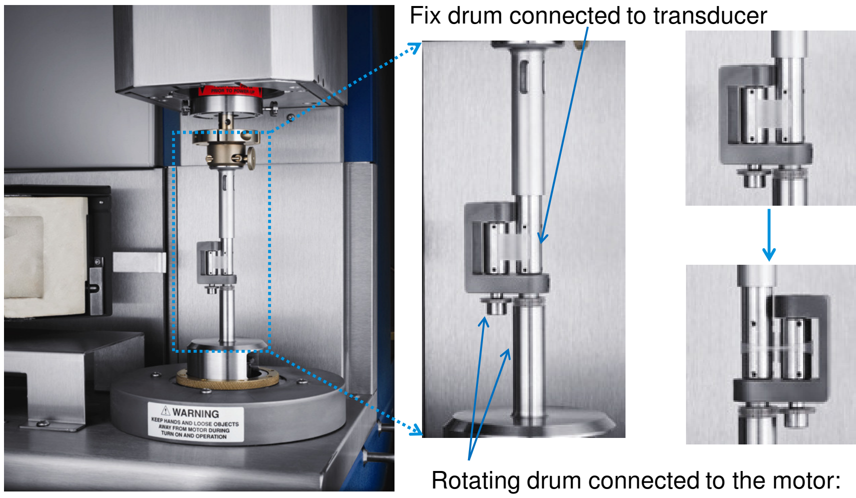


This is an interesting application of using the rotational rheometer to determine elongational viscosity

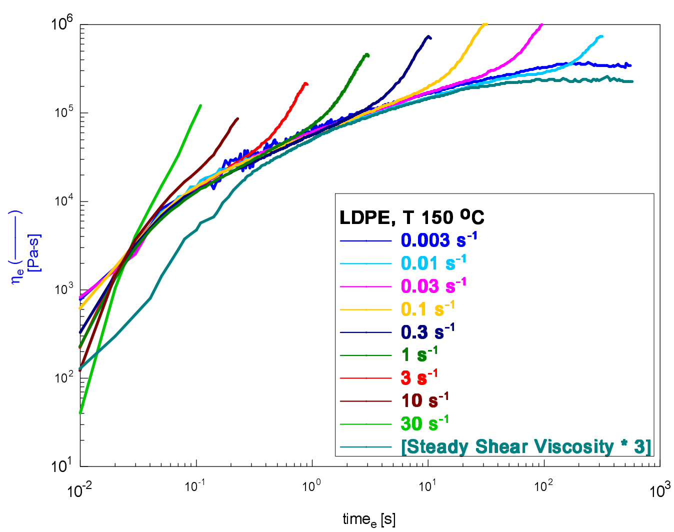
SER2 DHR Data



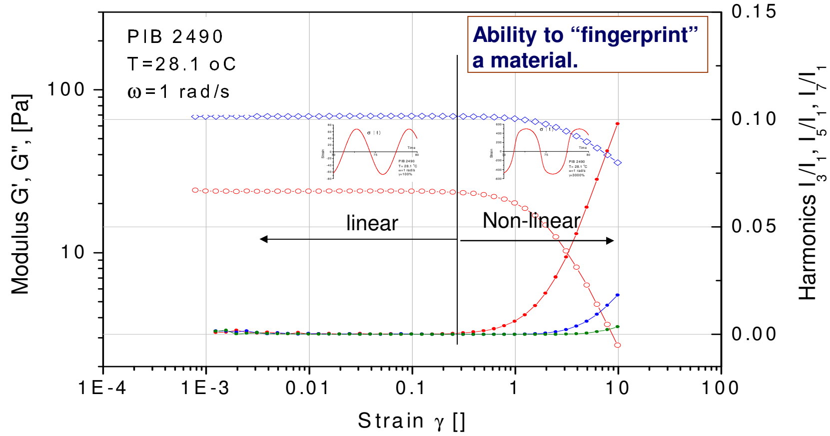
Extensional Viscosity Measurements



LDPE (High branching)



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.

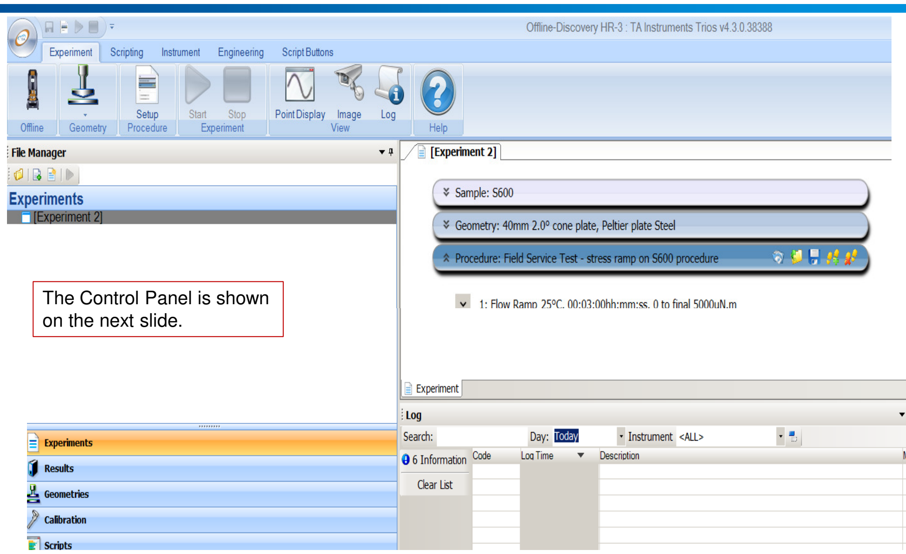
Trios Software

Trios Software

- Trios is non-restricted. Anyone can download Trios from our website at no cost and install it on any computer.
- Trios is versatile and friendly. It combines the best of Rheology Advantage (AR Rheometers) and Orchestrator (ARES) and has additional features too.
- Trios has all the necessary procedures for conventional as well as specialized testing.
- In Trios, with the DHR rheometer, you can add steps and modify steps that haven't started while the test is in progress.
- There is the Auto-tension feature, which is used in torsion rectangular testing and linear DMA testing.
- There is the Auto-strain feature, which allows the strain to change during an experiment based on the torque. This is useful for temperature ramps and curing tests.
- The graphics quality is excellent. Formatting graphs is easy with the Automatic options and very versatile with the Customized graphing.
- Data can be read in spreadsheet form. If you copy and paste into Excel, you will get the data and the headings.
- There are numerous variables to select from, but you can also construct user-defined variables.
- Trios can read data from Rheology Advantage and Orchestrator data files.

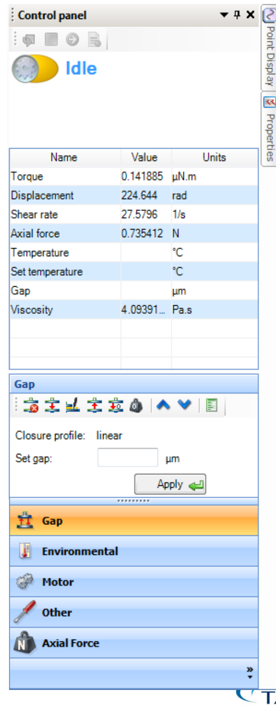
TAINSTRUMENTS.COM

Trios Screen

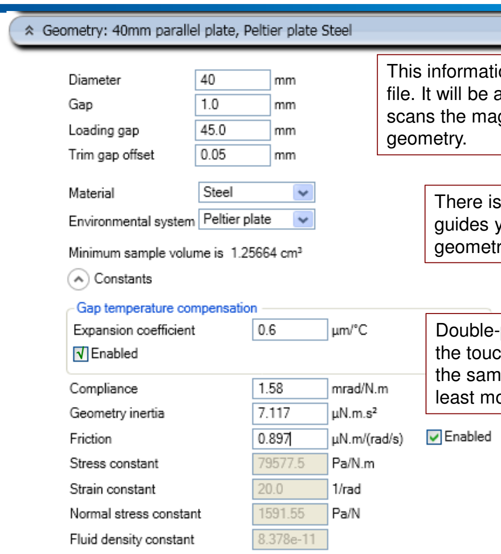


TAINSTRUMENTS.COM

You can control many things from the Control Panel section:
1. Gap
2. Temperature
3. Spindle speed
4. The laser for SALS
5. Axial force.



Geometry Information

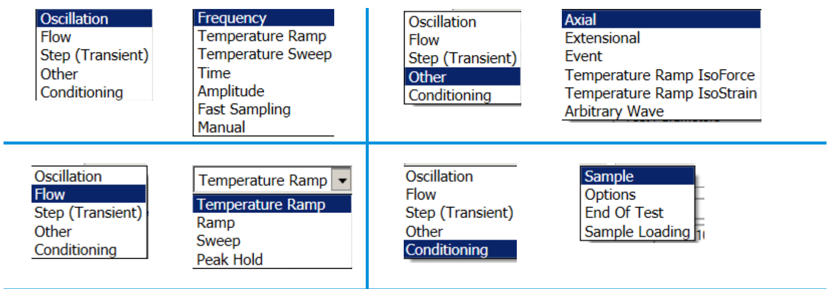


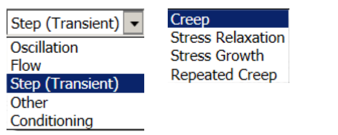
This information will be stored with the geometry file. It will be accessed when the magnetic reader scans the magnetic strip at the top of the geometry.

There is a friendly Geometry wizard that guides you through preparing a new geometry.

Double-pressing the Bearing Lock icon on the touch-pad and loading the geometry in the same location. enables you to retain at least most of the mapping

Trios Software Procedures

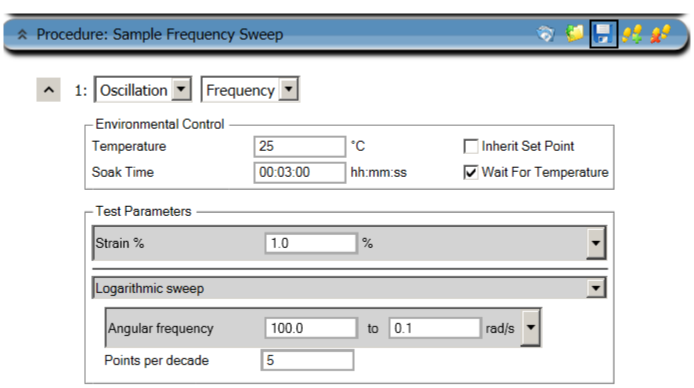




These are the choices in Trios for the DHR. There are minor differences with the choices for the ARES-G2.

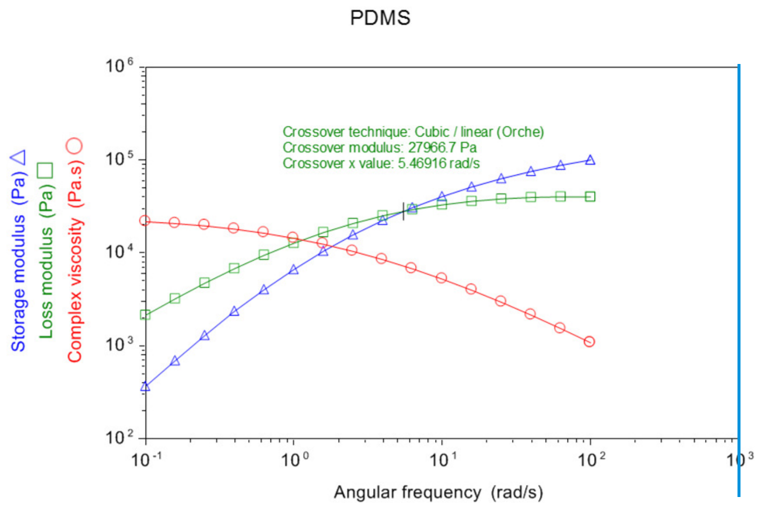
TAINSTRUMENTS.COM

Oscillation Procedure

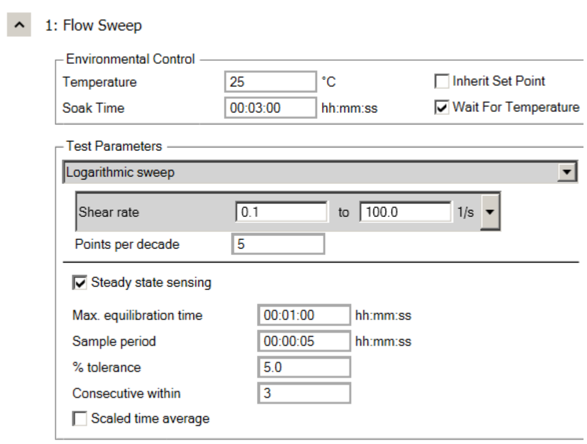


TAINSTRUMENTS.COM

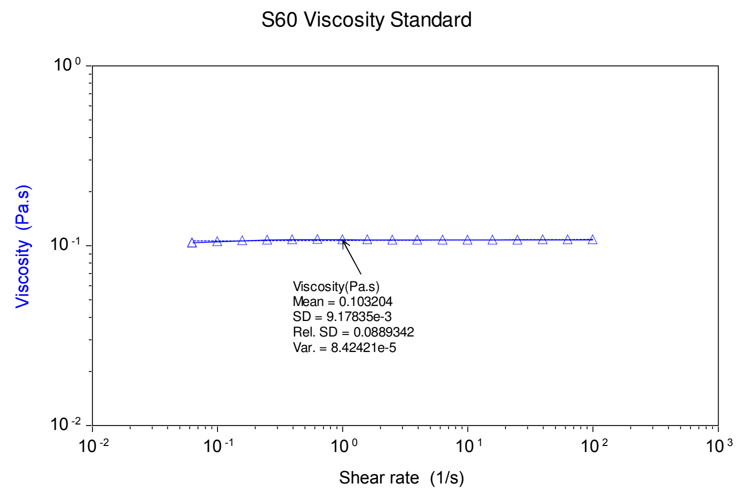
Display of Frequency Sweep Data



Flow Procedure



Display of Flow Data



TA | TAINSTRUMENTS.COM

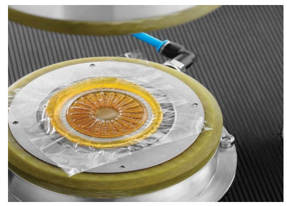
TA Instruments Rubber Testing Products

- Rheometer
- Viscometer
- Density Meter
- Hardness Tester

TA | TAINSTRUMENTS.COM

RPA Elite

RPA *elite*
Rubber & Polymer Analyzer

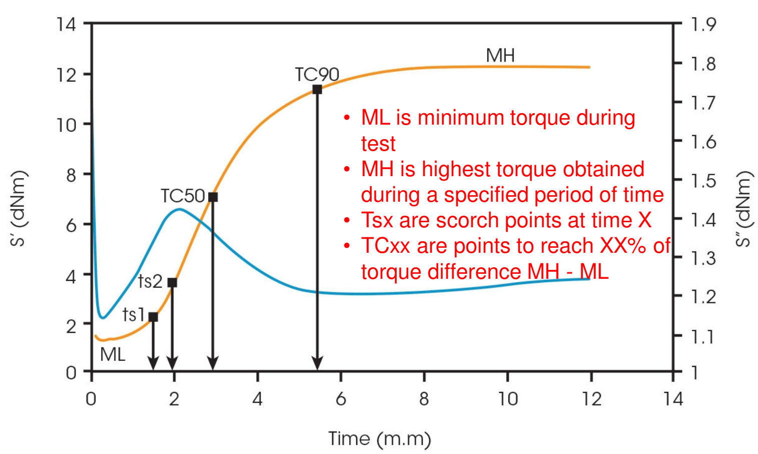


Densitometer

We obtained the RPA and others for rubber testing when we acquired Scarabaeus recently.

Shore A Hardness

ASTM D5289: MDR/RPA Isothermal Curing



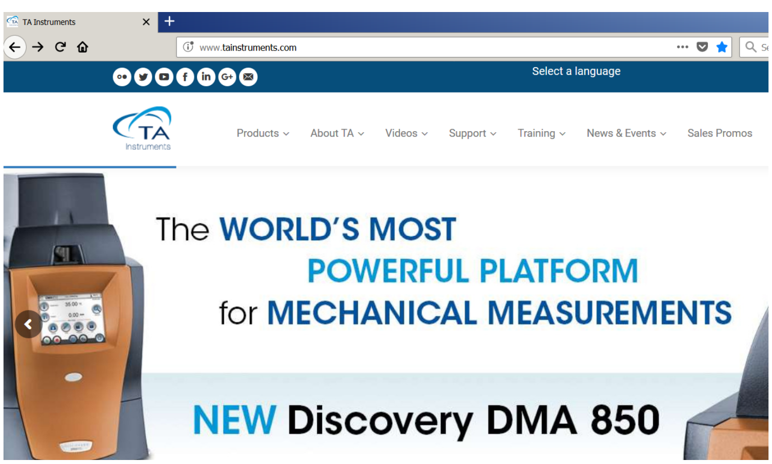
- ML is minimum torque during test
- MH is highest torque obtained during a specified period of time
- Tsx are scorch points at time X
- TCxx are points to reach XX% of torque difference MH - ML

Need Some Assistance?

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

TAINSTRUMENTS.COM

Website: www.tainstruments.com



TAINSTRUMENTS.COM

Website: www.tainstruments.com

Products | About TA | Videos | Support | Training | News & Events | Sales Promos

Service Support
- Service Support Helpline
- Site Preparation Guides
- The IQ/OQ Product Offering
- Calibration with Certified Standards
- Safety Data Sheets
- Supported Instruments
- Service Shop

Application Support
- Applications Support Helpline
- Tech Tips
- Applications Notes Library
- Training

Software Downloads & Support
- Software Downloads
- Instruments sorted by software
- Software Sorted by Instruments
- Report a Bug
- Request a Feature

Support Plans
- Lifetime Support Plan
- Premium Support Plan
- Plus Support Plan
- Basic Support Plan
- Performance Maintenance Visit (PMV)
- Academic Support Plan
- ElectroForce Support Plans

TAINSTRUMENTS.COM

Thank you for attending

Don't forget http://www.tainstruments.com/

If you have questions or need help
Contact the TA Instruments Technical Helpline
http://www.tainstruments.com/support/applications/applications-hotline/