Practical Rheology: Application Tips and Tricks

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Agenda

- Fundamental rheology
- Differences between a viscometer and a rheometer
- Flow analysis, application tips and tricks
- Oscillation analysis, application tips and tricks
- Introduction to rheology accessories and hyphenated techniques

TA Instruments website: Free Online thermal and rheology training courses

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Rheology: An Introduction



Rheology is the science of *flow* and *deformation* of matter



What Rheology measures?

- Rheology can provide information about the material's:

 - **Viscosity** - defined as a material's resistance to flow and is a function of shear rate or stress, with time and temperature dependence

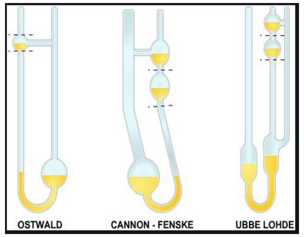
- **Viscoelasticity** - is the property of a material that exhibits both viscous and elastic character. Measurements of storage modulus (G'), Loss modulus (G"), and damping factor (tan d= G"/G') with respect to time, temperature, frequency and stress/strain are important for characterization

$$\frac{\text{Stress}}{\text{Shear rate}} = \text{Viscosity}$$

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

Rheology Instrumentation

Viscometers



Rotational rheometers

Capillary viscometers

DMAs and mechanical testers

What is the difference between a **Viscometer** and a **Rheometer**?

Viscometers

Capillary viscometers

- Only measure viscosity
- Only handle liquid samples
- Limited measurement range
- Report apparent viscosity vs. rpm and/or temperature
- May not be able to measure true shear viscosity vs. shear rate

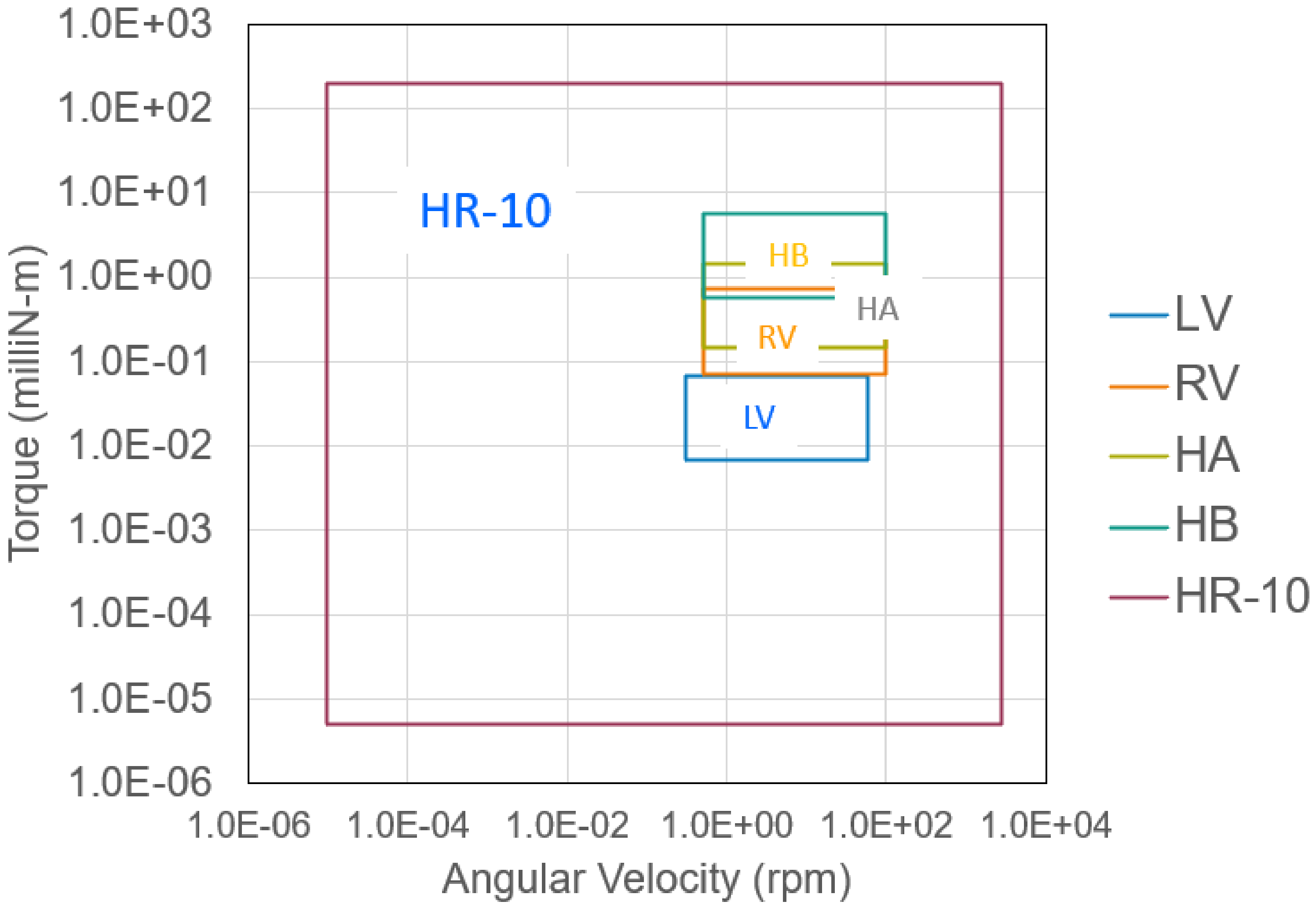
What is the difference between a **Viscometer** and a **Rheometer**?

Rotational rheometers

DMAs and mechanical testers

- Capable of handling any samples from low viscosity liquid to high modulus solid
- Capable of performing flow measurement to directly measure viscosity vs. shear rate, shear stress and temperature
- Capable of performing oscillation measurement to measure viscoelastic properties of a sample
- Capable of perform transient measurement such as creep, stress relaxation, or stress/strain

Comparison of Angular Velocity and Torque Ranges of HR-10 vs. Brookfield Viscometer

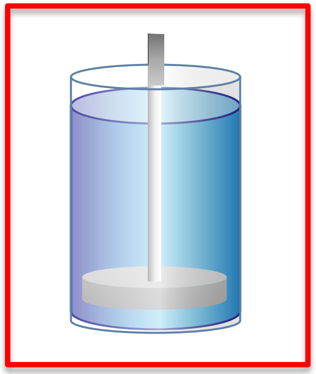


- With flow testing, the Discovery HR-X0 rheometers provide a much wider range of angular velocities and torques than do common viscometers.

- LV, RV, HA, HB are different type of viscometer spindles

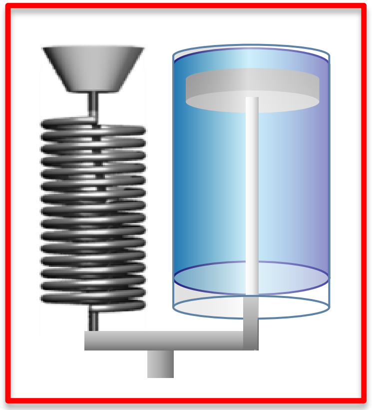
What Type of Samples a Rheometer Can Measure?

- **Viscosity (liquids)**

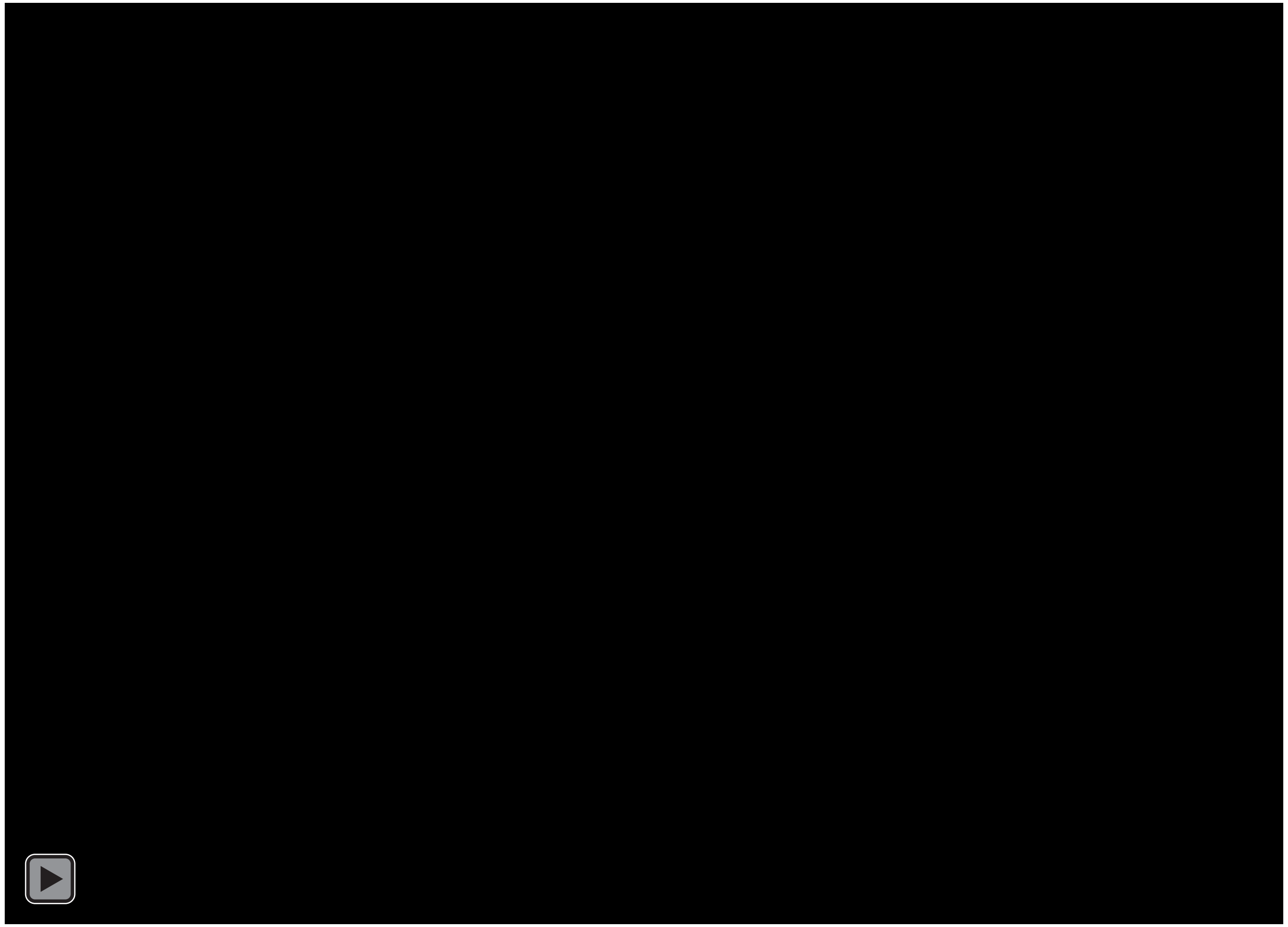


- **Elasticity (solids)**

- **Viscoelasticity (semi-solids, pastes and gels)**

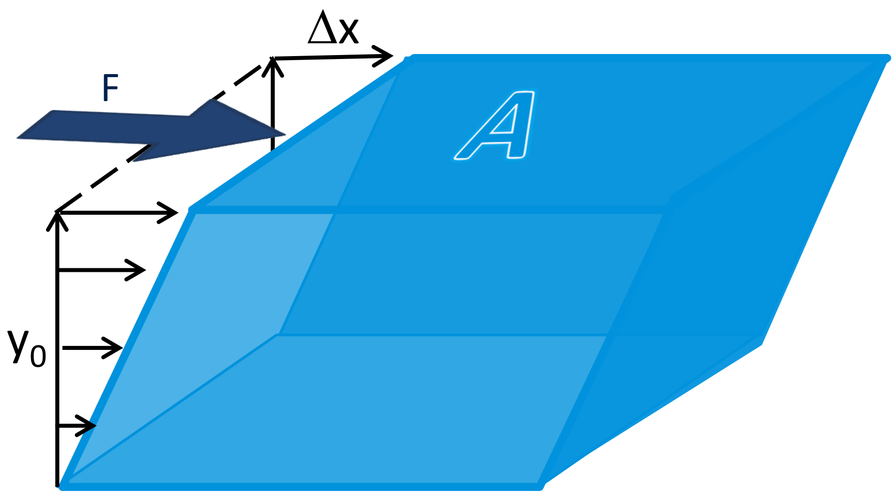


A Rotational Rheometer- How does it work?



How does a Rotational Rheometer Work?

- The study of *stress* and *deformation* relationship



Shear stress $\sigma = \frac{F}{A}$

Shear strain $\gamma = \frac{\Delta x}{y_0}$

Shear rate $= \dot{\gamma} = \frac{1}{y_0} \cdot \frac{dx(t)}{dt}$

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

$$\frac{\text{Stress}}{\text{Shear rate}} = \text{Viscosity}$$

How does a Rotational Rheometer Work?

- In a rheological measurement, stress; strain and strain rate (shear rate) are all calculated signals
- The raw signals behind the scene are torque; angular displacement and angular velocity

Fundamentally, a rotational rheometer will apply or measure:

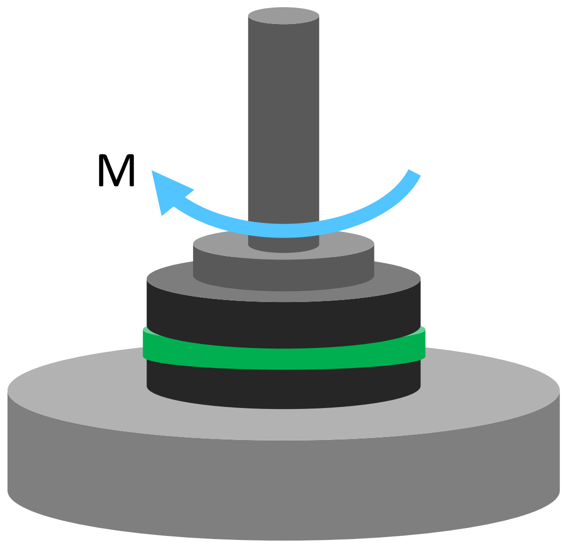
1. Torque (Force)
2. Angular Displacement
3. Angular Velocity

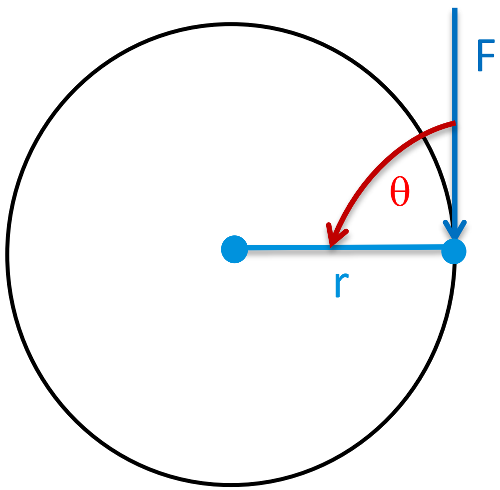
Measured parameter: torque

- Torque (M) is a measure of how much a force (F) acting on an object causes that object to rotate.
  - The object rotates about an axis, called the pivot point
  - The distance (r) from the pivot point to the point where the force acts is called the moment arm
  - The angle (θ) at which the force acts at the moment arm

$$M = r \cdot F \cdot \sin\theta = r \cdot F$$

(for θ = 90° as shown)





Calculated parameter: stress

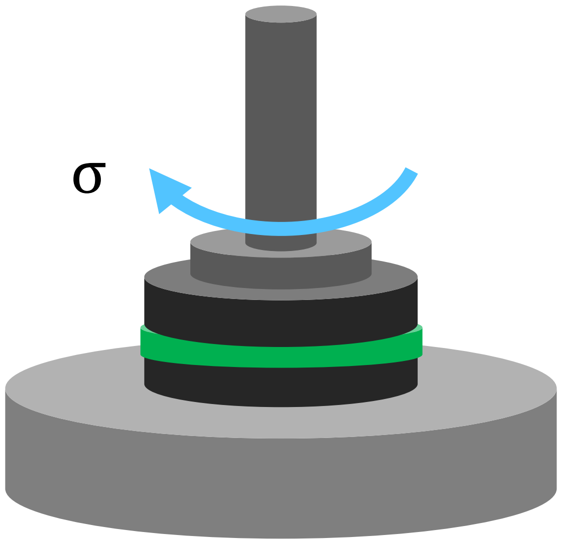
- Shear stress is calculated from the torque and geometry stress constant

$$\sigma = M \cdot K_\sigma$$

σ = shear stress (Pa or Dyne/cm^2)

M = torque (N·m or gm·cm)

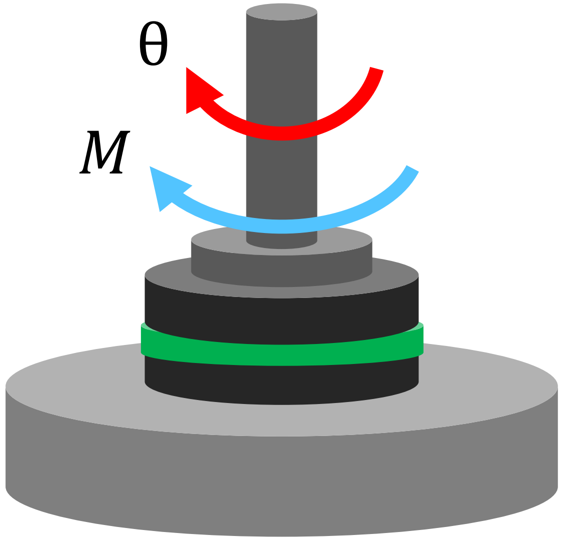
K_σ = stress constant



- The stress constant, K_σ, is dependent on measurement geometry and/or initial sample dimensions

Measured parameter: angular displacement

- Angular displacement (θ) is the angle, in radians, through which an object moves on a circular path

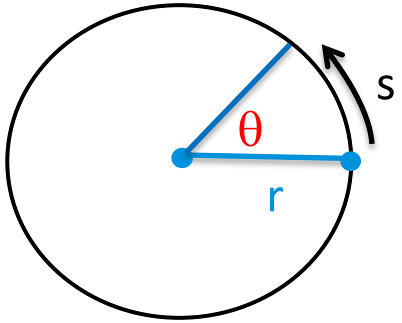


s = arc length (or linear displacement)

r = radius of a circle

Conversion: degrees = radians·180/π

$$\theta = s/r$$



Calculated parameter: strain

- Strain is a measure of deformation representing the angular displacement relative to a reference length

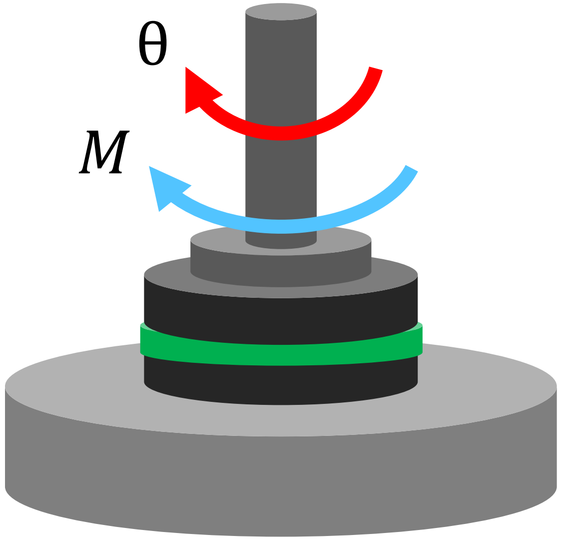
$$\gamma = \theta \cdot K_\gamma$$

γ = shear strain (no units)

θ = angular displacement (radians)

K_γ = strain constant

- The strain constant, K_γ, is dependent on measurement geometry and/or initial sample dimensions



Equation for modulus

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

Material function

Constitutive equation

Measured signals

Geometry constants

Measured parameter: angular velocity

- Angular velocity (Ω) is the change in angular displacement (θ) per unit time of measurement

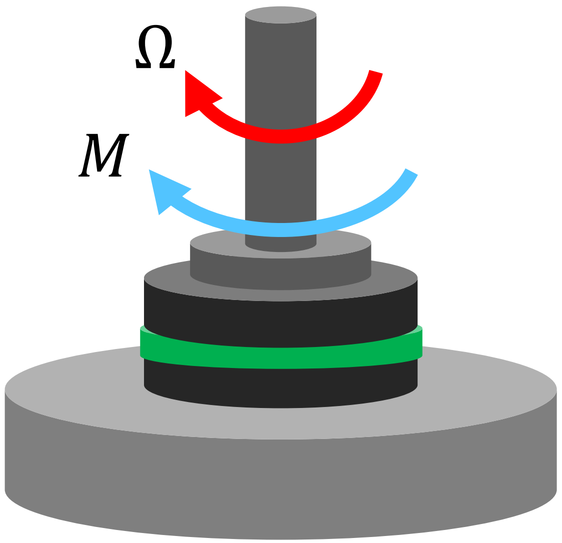
  Note: linear velocity V = $\Delta s/\Delta t$

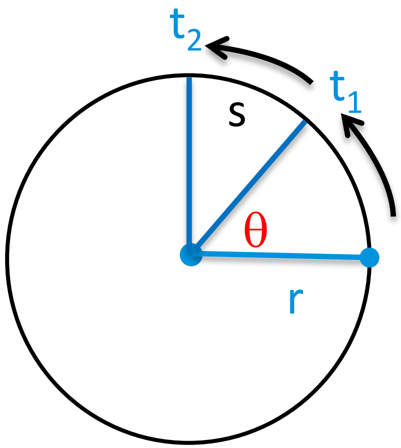
$$\Omega = \Delta\theta/\Delta t$$

Ω = angular velocity (radians/s)

θ = angular displacement (radians)

t = time (s)





Calculated parameter: shear rate

- Shear rate is calculated from the angular velocity and geometry strain constant

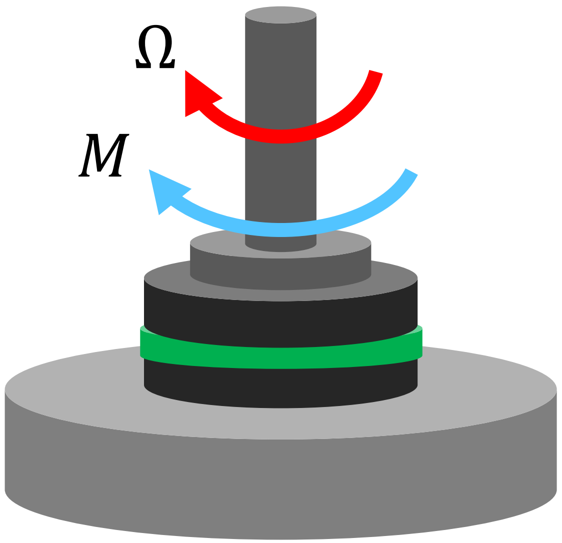
$$\dot{\gamma} = \Omega \cdot K_\gamma$$

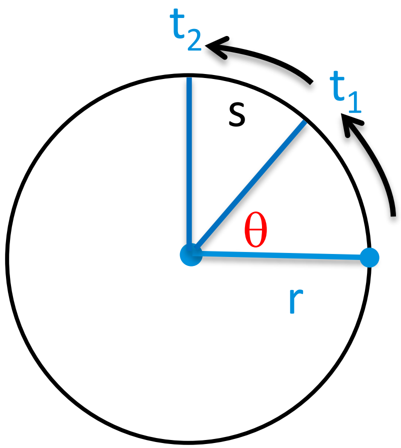
$\dot{\gamma}$ = shear rate (s^{-1})

Ω = angular velocity (radians/s)

K_γ = strain constant

- The strain constant, K_γ, is dependent on measurement geometry and/or initial sample dimensions





Equation for viscosity

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

Material function

Constitutive equation

Measured signals

Geometry constants

Discovery Hybrid Rheometer Specifications

Technical Specifications

Specification	HR 30	HR 20	HR 10
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	0.3	1	5
Minimum Torque (nN.m) Steady Shear	1	3	5
Maximum Torque (mN.m)	200	200	200
Torque Resolution (nN.m)	0.05	0.1	0.1
Minimum Frequency (Hz)	1.0E-7	1.0E-7	1.0E-7
Maximum Frequency (Hz)	100	100	100
Minimum Angular Velocity[1] (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	Standard	N/A
Displacement Resolution (nrad)	2	2	10
Step Time, Strain [2] (ms)	15	15	15
Step Time, Rate [2] (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

DMA Mode Specifications	
Motor Control	Force Rebalance Transducer
Minimum Force in Oscillation	3 mN
Maximum Axial Force	50 N
Minimum Displacement in Oscillation	0.01 µm
Maximum Displacement in Oscillation	100 µm
Axial Frequency Range	6×10^{-5} rad/s to 100 rad/s (10^{-5} Hz to 16 Hz)

ARES-G2 Rheometer Specifications

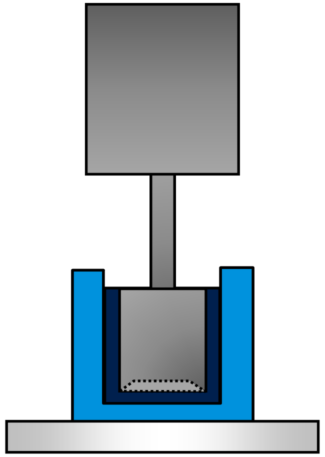
Force/Torque Rebalance Transducer (Sample Stress)	
Transducer Type	Force/Torque Rebalance
Transducer Torque Motor	Brushless DC
Transducer Normal/Axial Motor	Brushless DC
Minimum Torque (μN.m) Oscillation	0.05
Minimum Torque (μN.m) Steady Shear	0.1
Maximum Torque (mN.m)	200
Torque Resolution (nN.m)	1
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 x 10^{-5} to 16

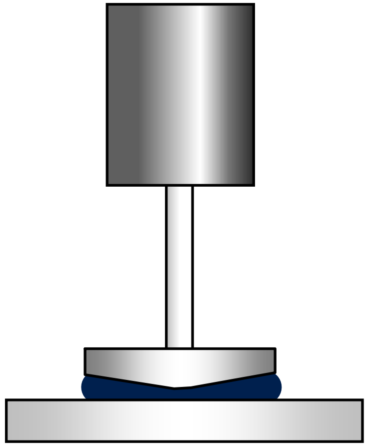
Geometry Options

Concentric Cylinders



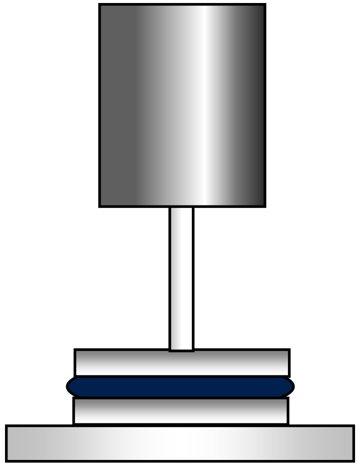
Very Low to Medium Viscosity

Cone and Plate



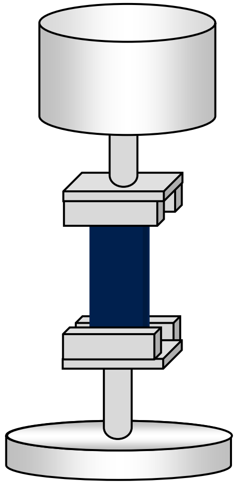
Very Low to High Viscosity

Parallel Plate



Very Low Viscosity to Soft Solids

Torsion Rectangular



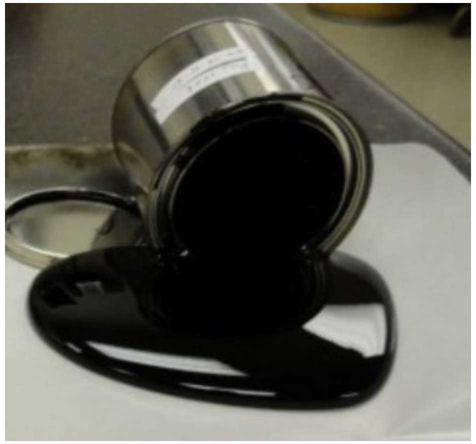
Mid-modulus Solids

Water to Steel

Rheology Applications
Flow Analysis - Tips and Tricks

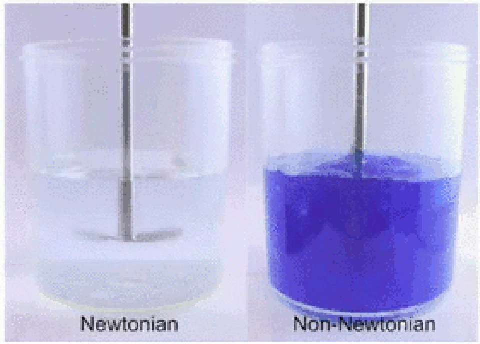
Viscosity Ranges

Materials	Viscosity η (Pa.s)
Air /Gas	0.00001
Water	0.001
Milk/ Coffee	0.01
Olive oil	0.1
Glycerol	1
Liquid Honey	10
Molasses	100
Polymer Melt	1000
Asphalt Binder	100,000



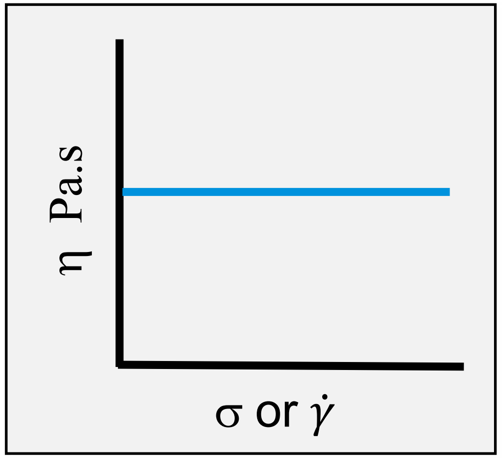
Viscosity Behaviors

Rod Climbing Effect

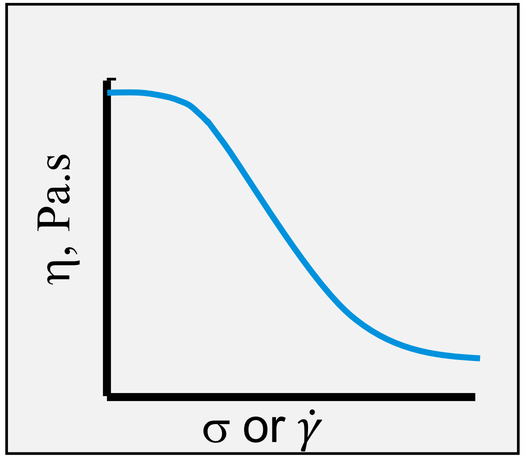


- Newtonian and non-Newtonian

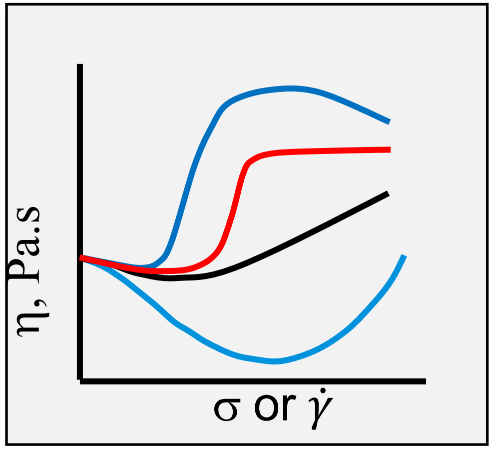
Newtonian



Shear Thinning



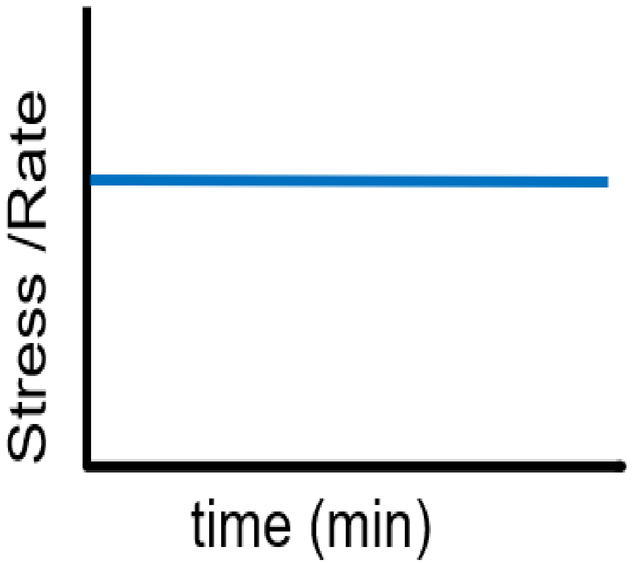
Shear Thickening



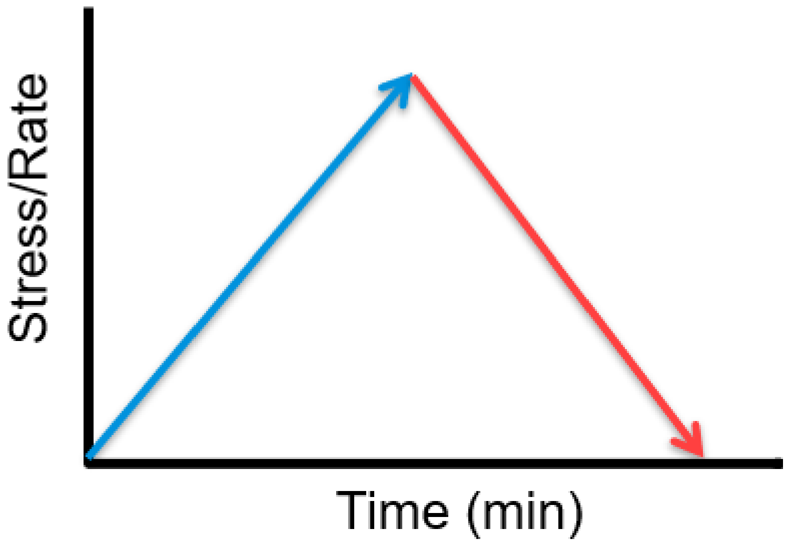
Viscosity Measurement Methods

- Common rheological methods for measuring viscosity of liquids

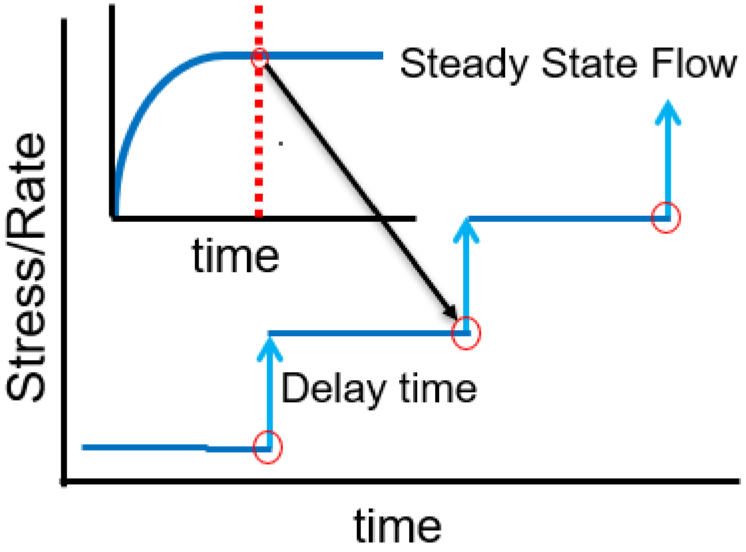
Single rate/stress



Flow ramp



Stepped or steady state flow



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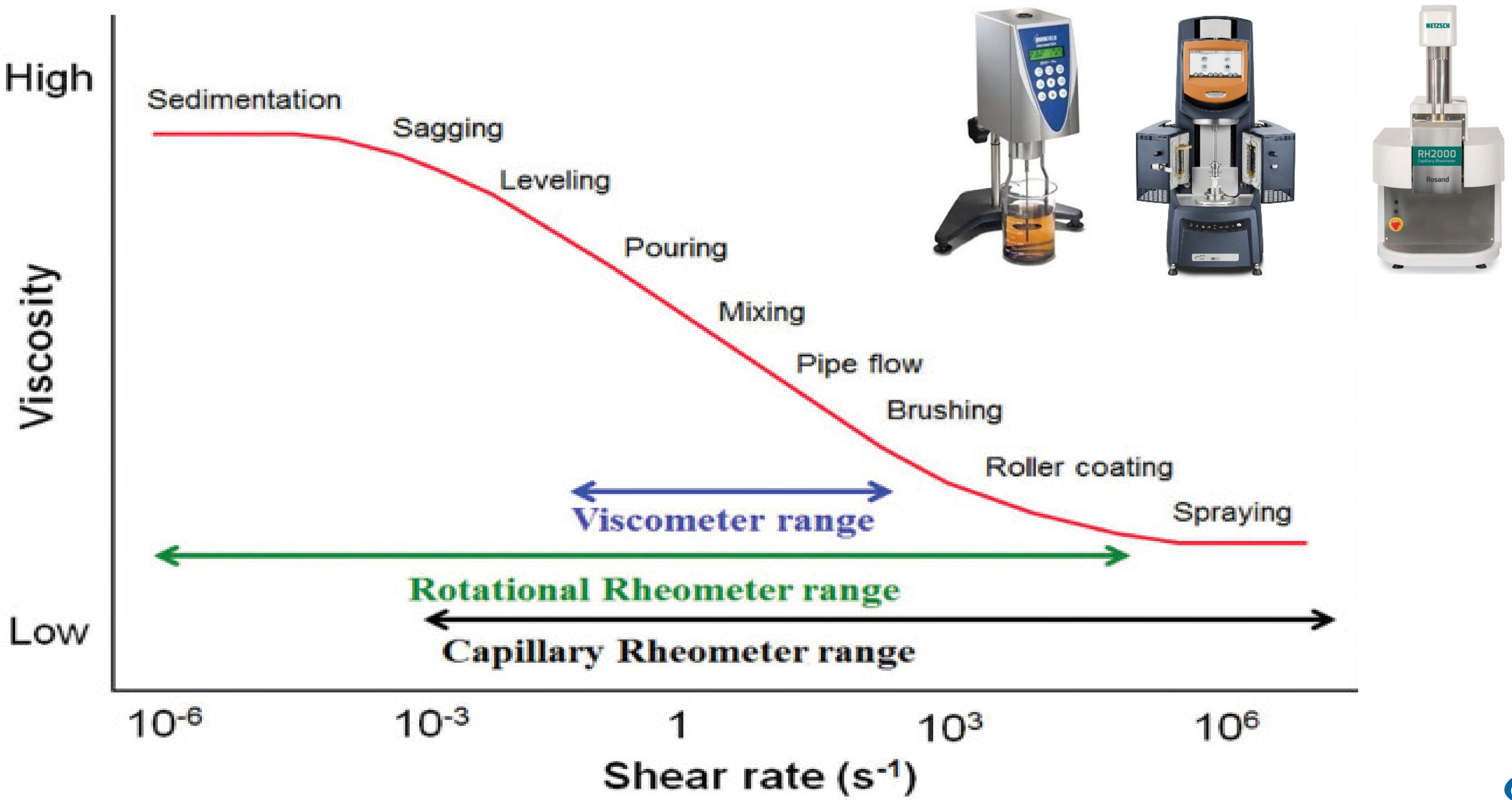
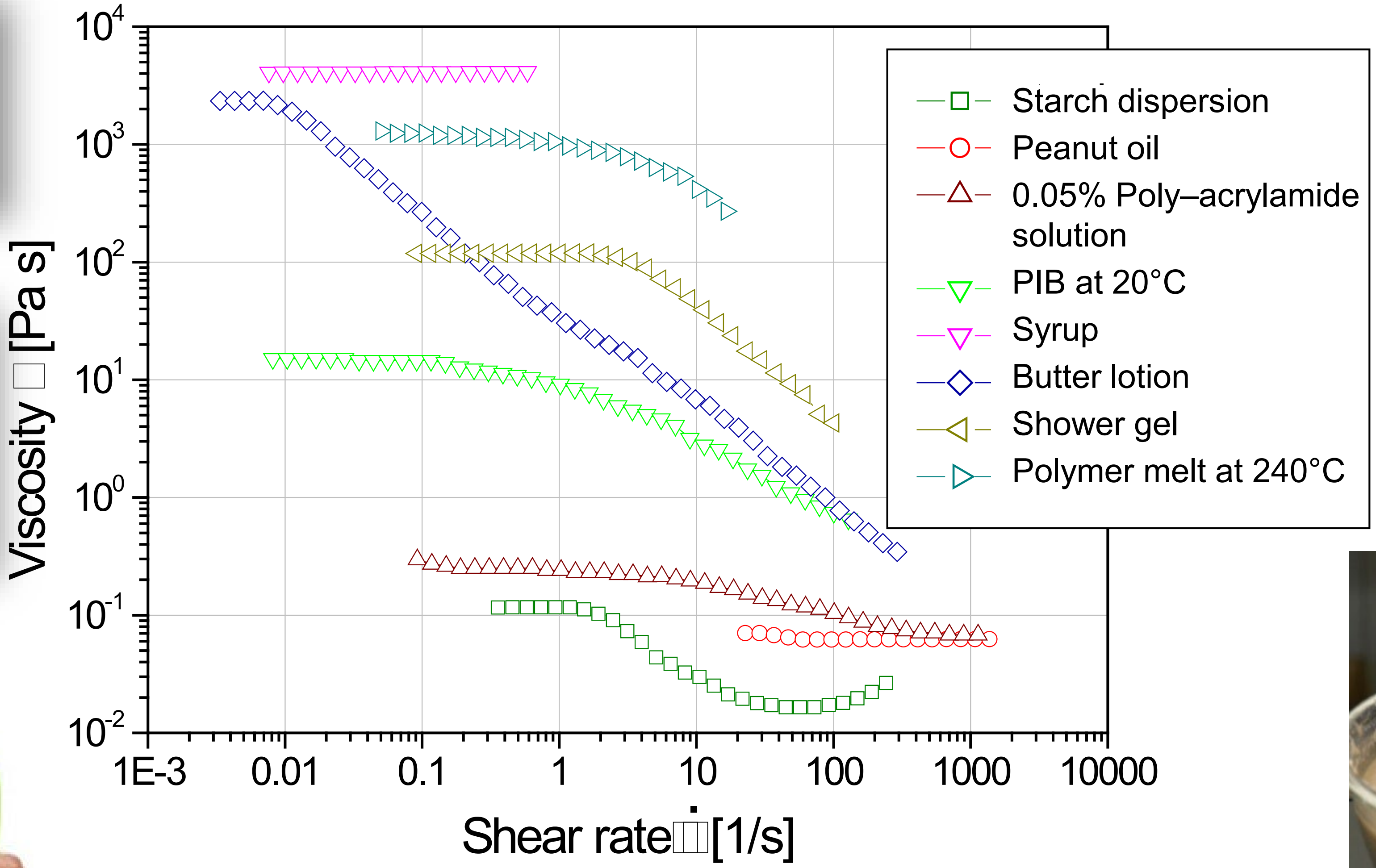
Information from a Flow Curve

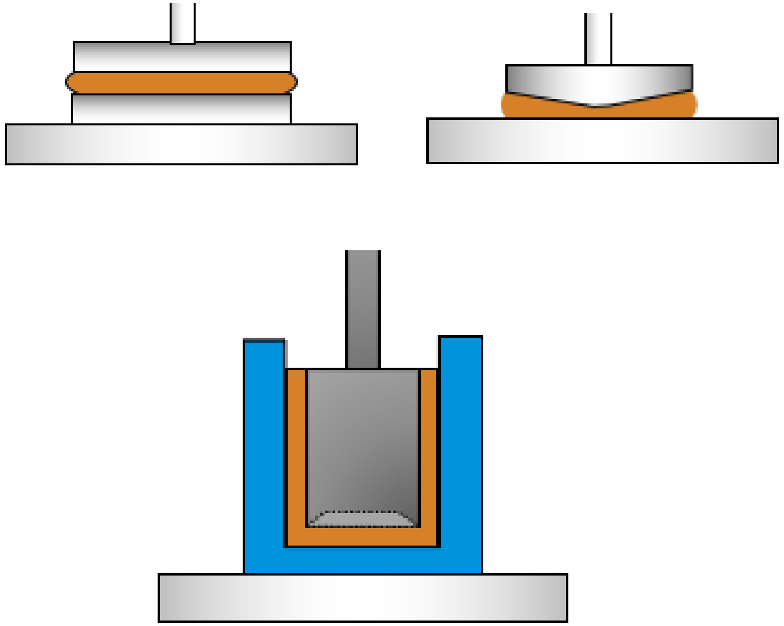


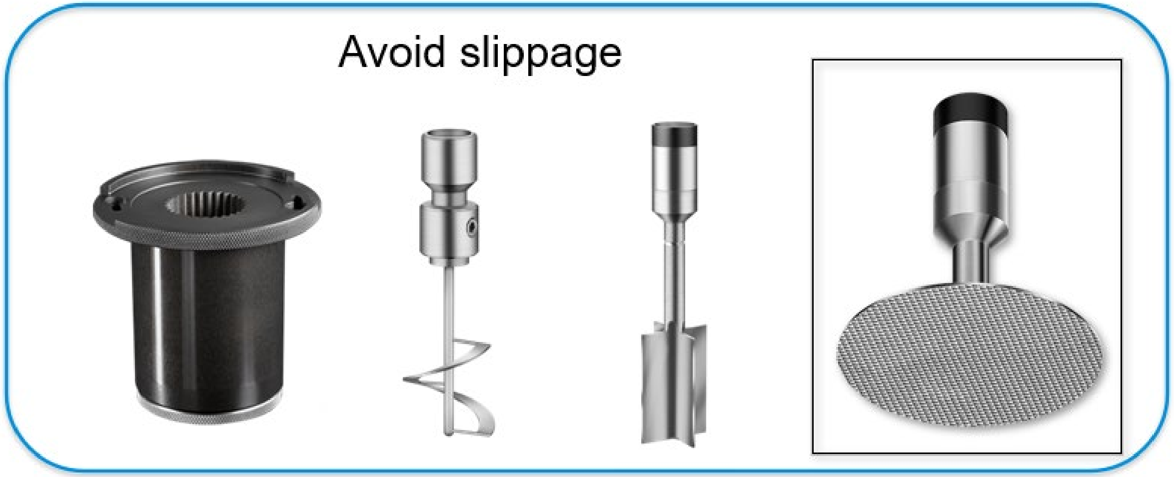
Viscosity Curves of Various Fluids



What Geometry to Use?

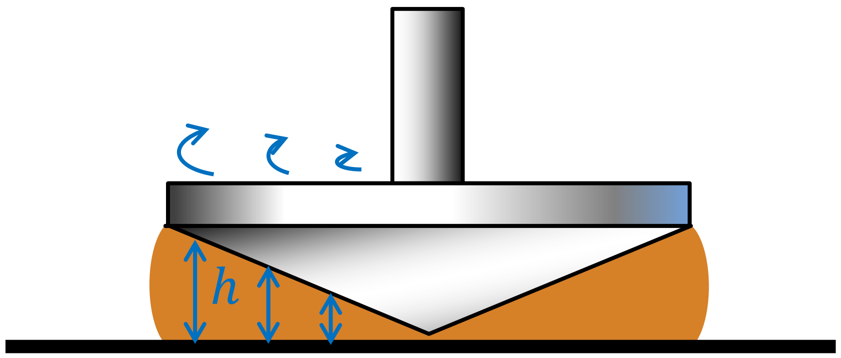
- Cone or parallel plate (e.g. 60mm, 40mm, 25mm, 20mm, 8mm)
- Roughen surface parallel plate to avoid slippage (e.g. crosshatched, sandblasted)
- Concentric cylinder cup with DIN, recess-end, vane or helical rotor





What is the difference between a Cone-plate vs. a parallel plate?

Provide uniform shear across the sample

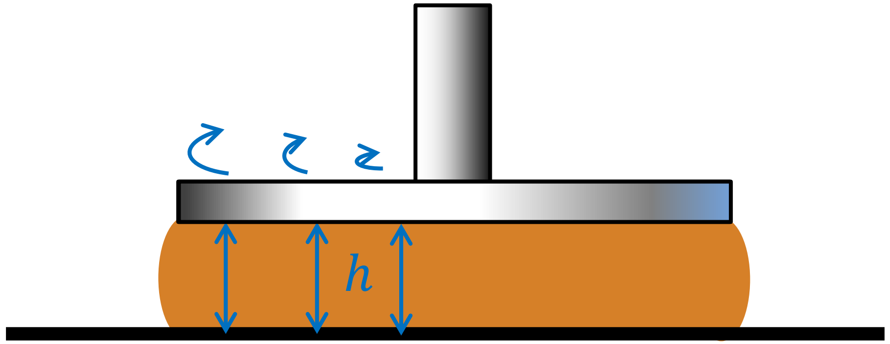


- The cone shape produces a smaller gap height closer to inside, so the shear on the sample is constant

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

h increases proportionally to *dx*, γ is uniform

Non-uniform shear but can be corrected



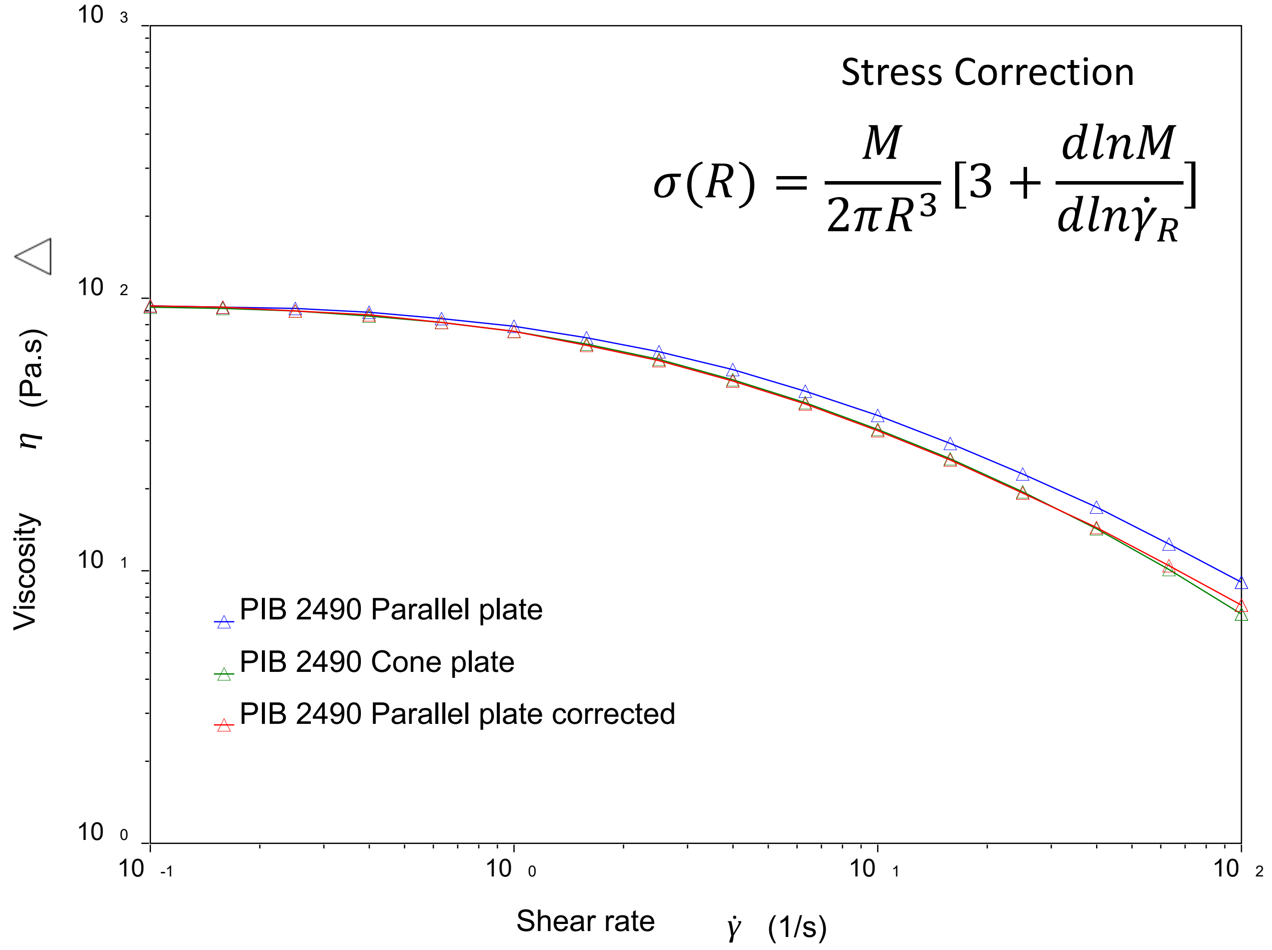
- For a given angle of deformation, there is a greater arc of deformation at the edge of the plate than at the center

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

dx increases further from the center, *h* stays constant

Parallel Plate Stress Correction

- The parallel plate viscosity can be corrected through the Weissenberg-Rabinowitsch correction so that parallel plate data can be compared with cone and plate data.



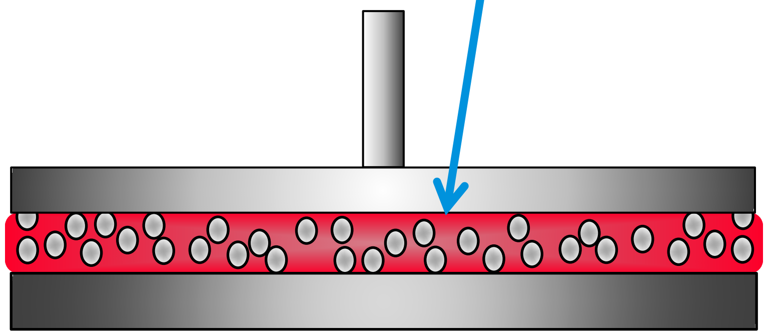
Be Careful When Using a Cone and Plate

- Most structured fluids contain particles with size in the micrometer range
- Depending on the cone angle, the truncation gap of a cone geometry will be between 10 and 120 mm
- Parallel plate geometry is recommended

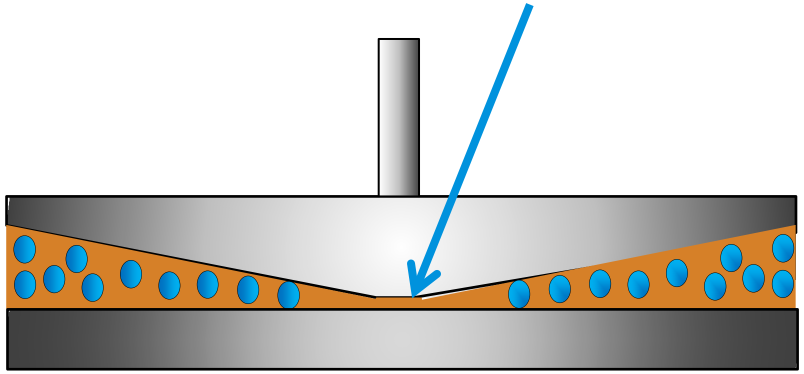
Typical Truncation Heights:

1° degree ~ 20 - 30 microns
2° degrees ~ 60 microns
4° degrees ~ 120 microns

Gap can be adjusted
Common gap at 0.5-2mm



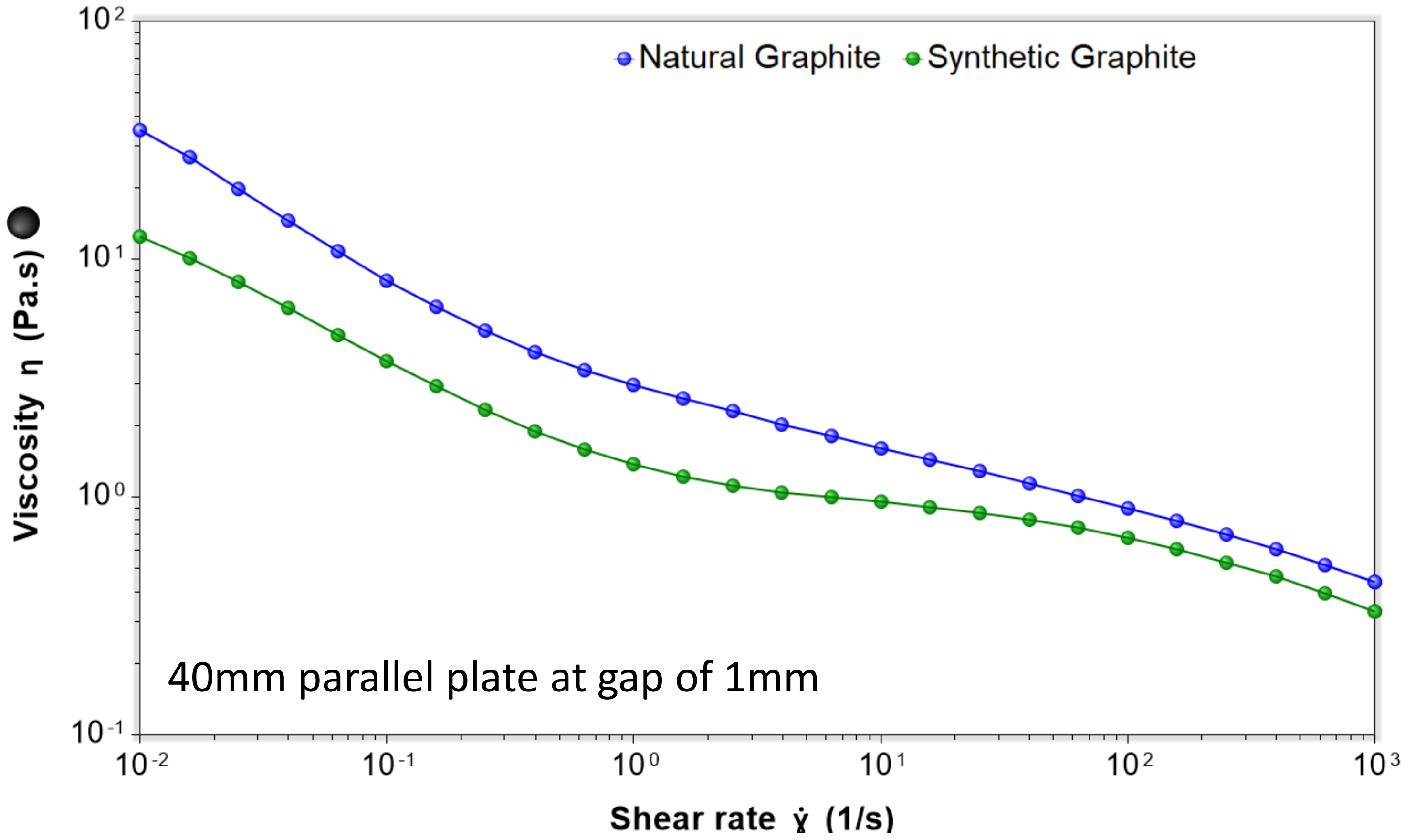
Truncation Height = Gap

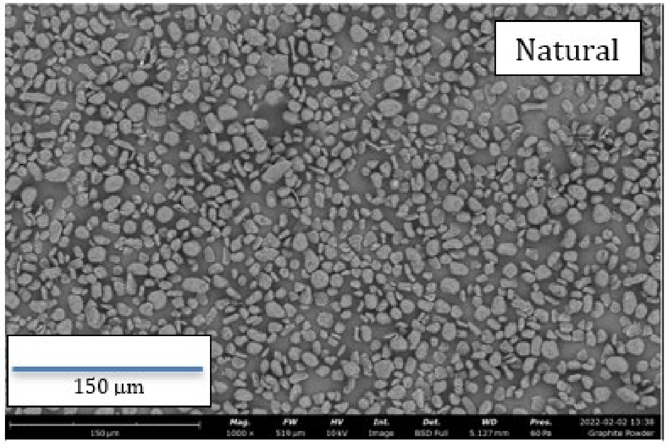


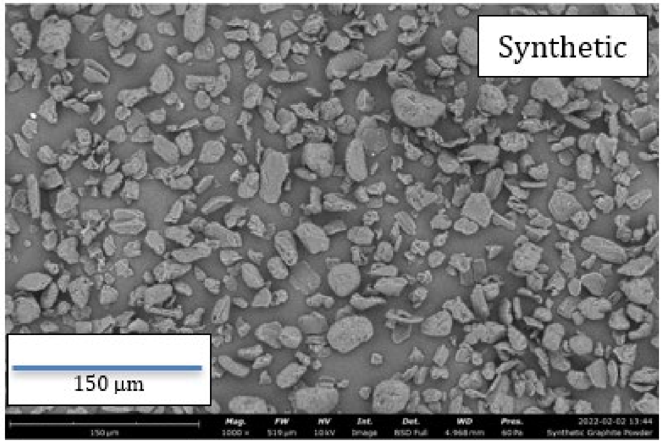
Gap must be > or = 10 x particle size

Example Viscosity Test on Battery Slurries

- Slurry viscosity scan over a wide range of shear rate
- Particle size and shape affect the flow behavior of the slurry



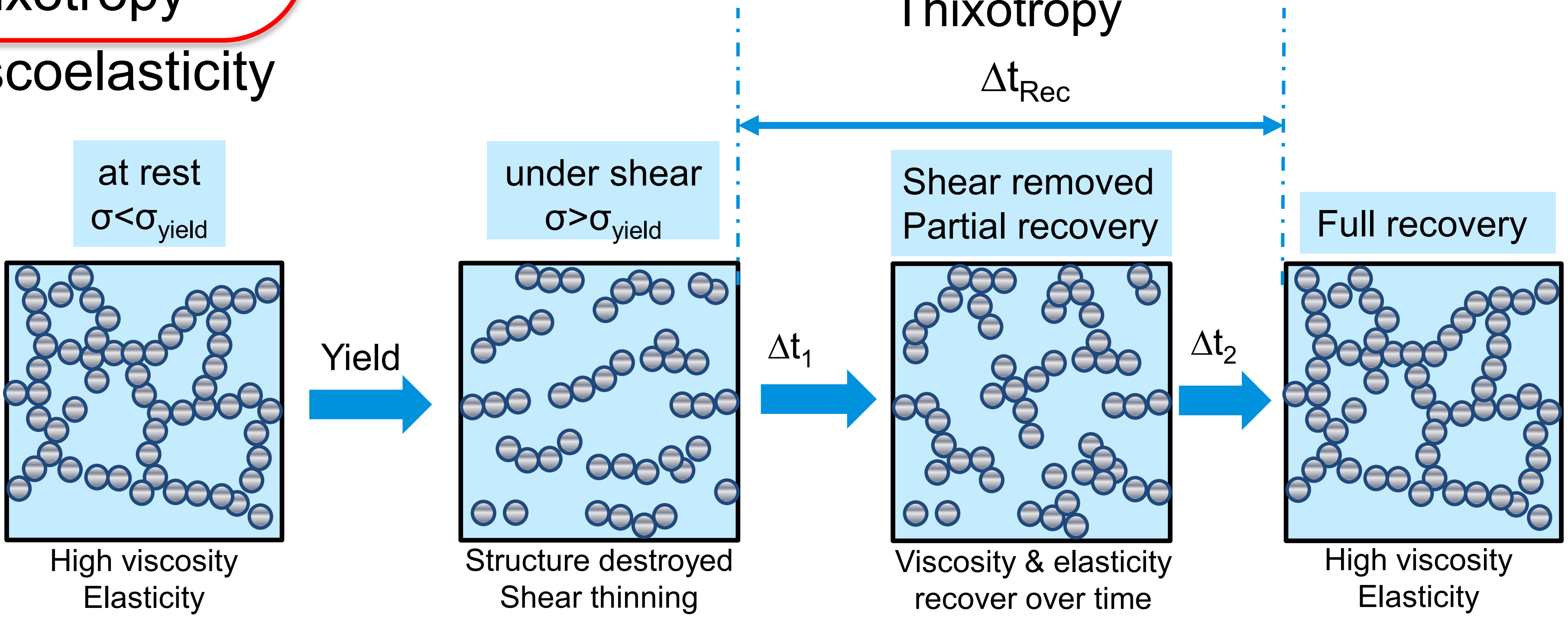




T Chen, Apps note RH119

What else a flow test can measure? -Yield and Thixotropy

- Structured fluid properties
  - Non-Newtonian
  - Yield stress
  - Thixotropy
  - Viscoelasticity



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What is Yield?

- Yield stress is a time dependent characteristic that is associated with many structured fluids such as Mayonnaise, Ketchup, hand lotion, hair gels, paints etc.
- A material that has yield does not flow unless the applied stress exceeds a certain value – yield point
- Yield stress is created in formulation by adding additional thickeners
- Yield helps stabilize complex fluids
    - Avoid sedimentation and increase shelf life
    - Reduce flow under gravity
    - Stabilize a fluid against vibration

How to Measure Yield

- Yield can be quantitatively measured on a rotational rheometer
- Common methods
  - Stress ramp
  - Stress sweep
  - Shear rate ramp
  - Dynamic stress/strain sweep

No settling?!

Note:
Yield behavior is a time dependent characteristic. Measured yield stress values will vary depending on experimental parameters

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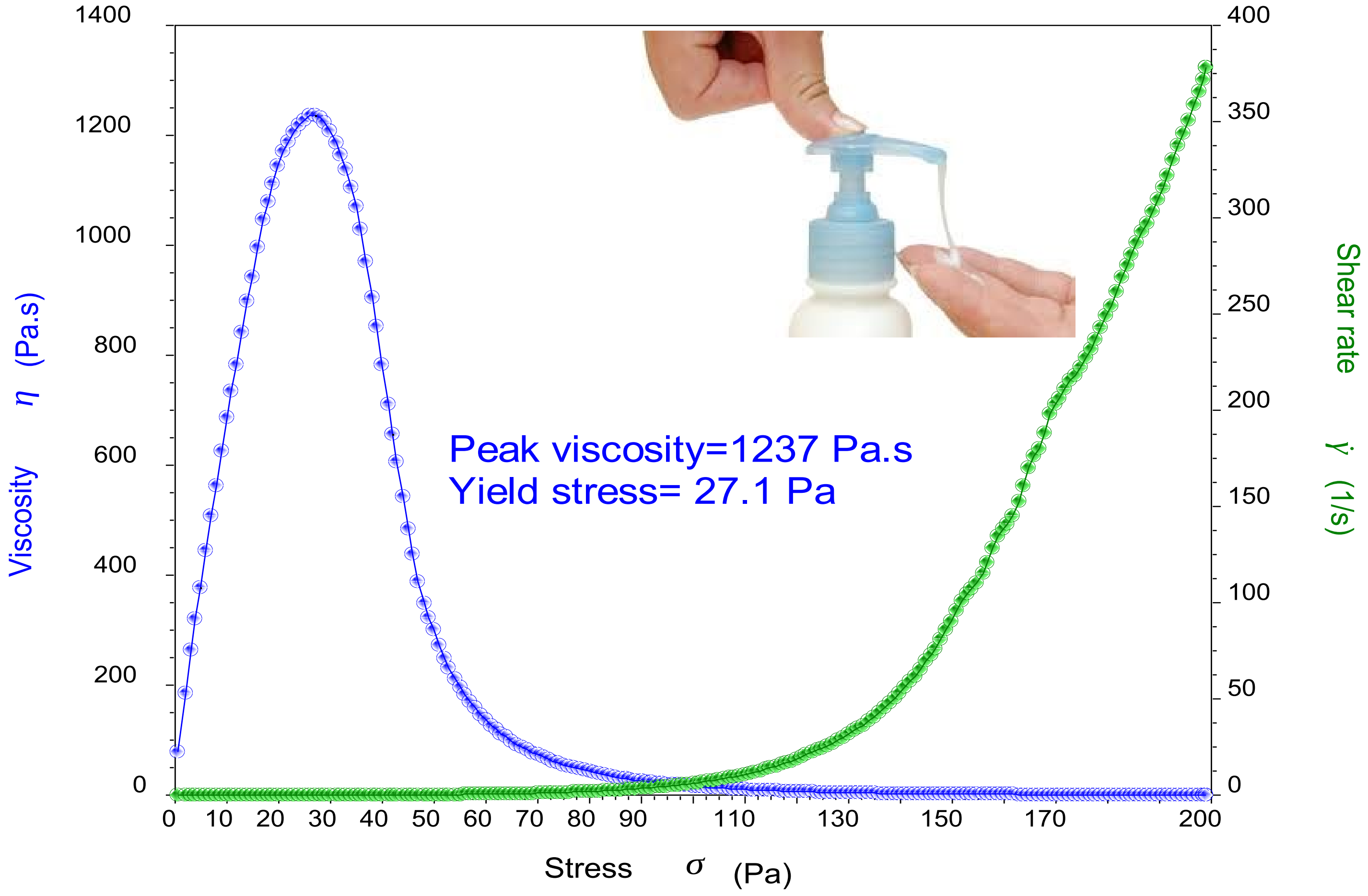
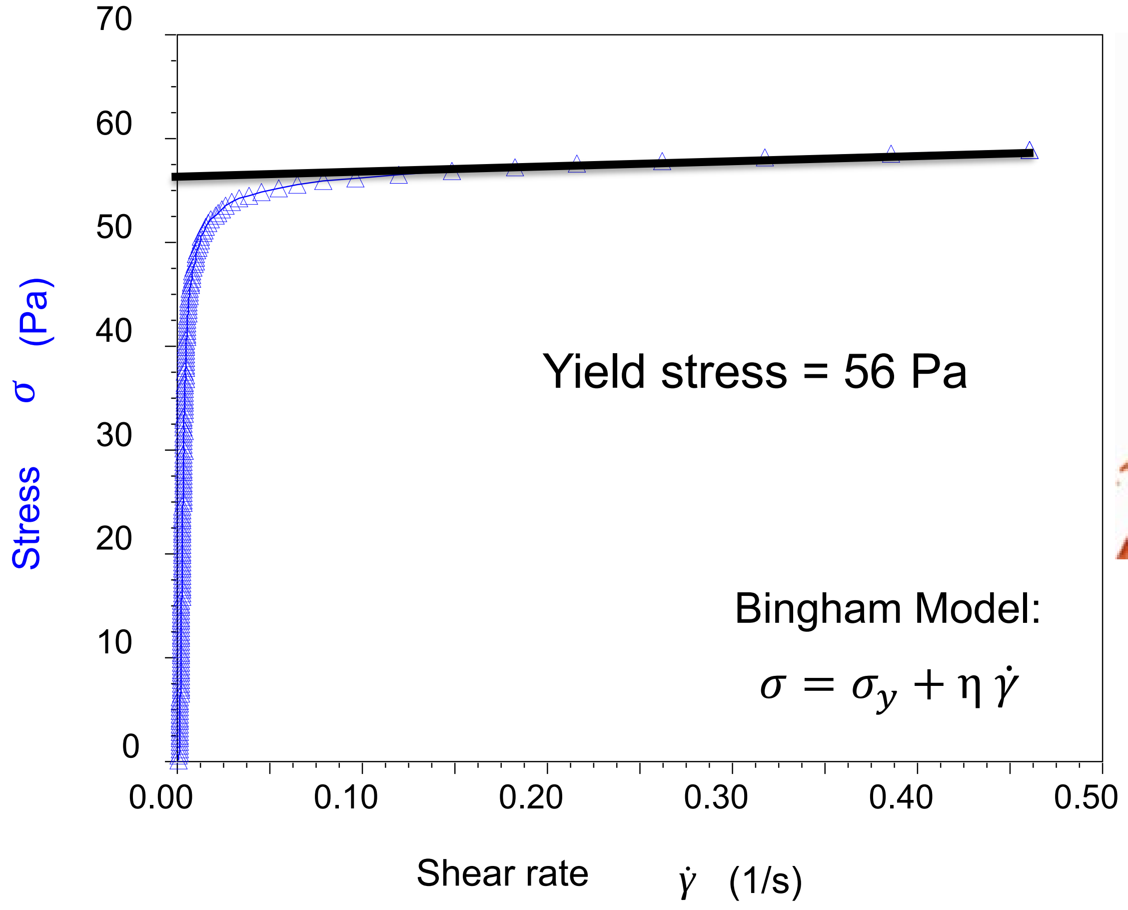
Yield Stress of a Body Lotion

- **Stress ramp** from 0 to 200 Pa in 60 seconds
- Yield is determined at the point where viscosity shows a peak



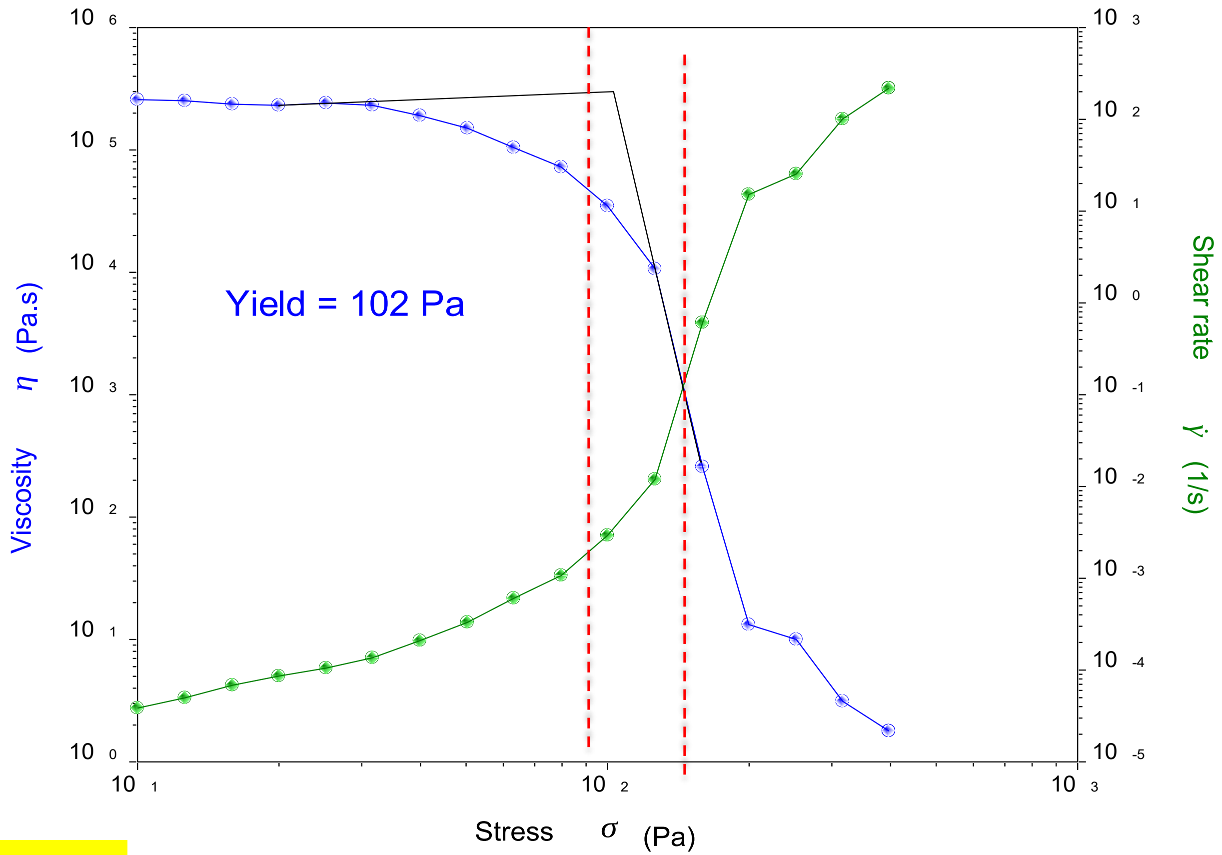
Yield Stress of Ketchup

- **Stress ramp** test on Katchup
- Yield is computed by fitting the flow curve with a mathematical model



Yield Stress of a Toothpaste

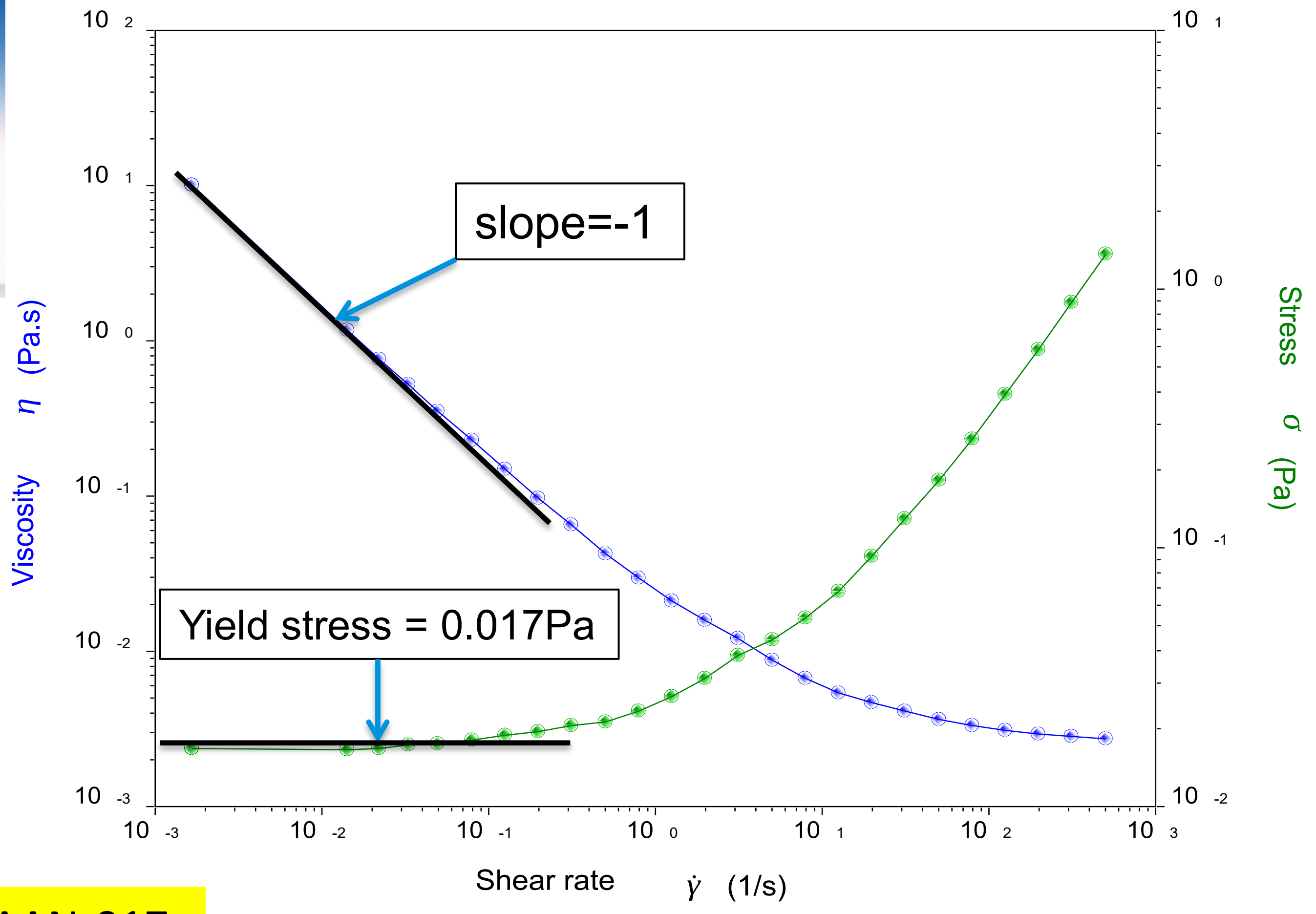
- **Steady state stress sweep** from 10 Pa to 500 Pa
- Yield stress is determined by a sharp decrease in viscosity over a narrow range of applied shear stress
- Take the onset of viscosity vs. stress curve



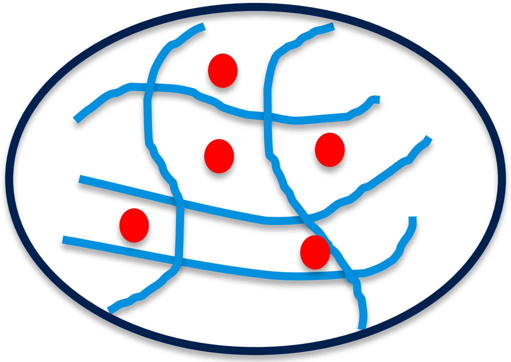
Chen apps note: RH094

Yield Stress of Orange Juice

- **Shear rate ramp down** from 500 to 0.001 1/s
- Yield is identified by the stress plateau



No settling?!



Chen apps note: AAN-017

What is Thixotropy?

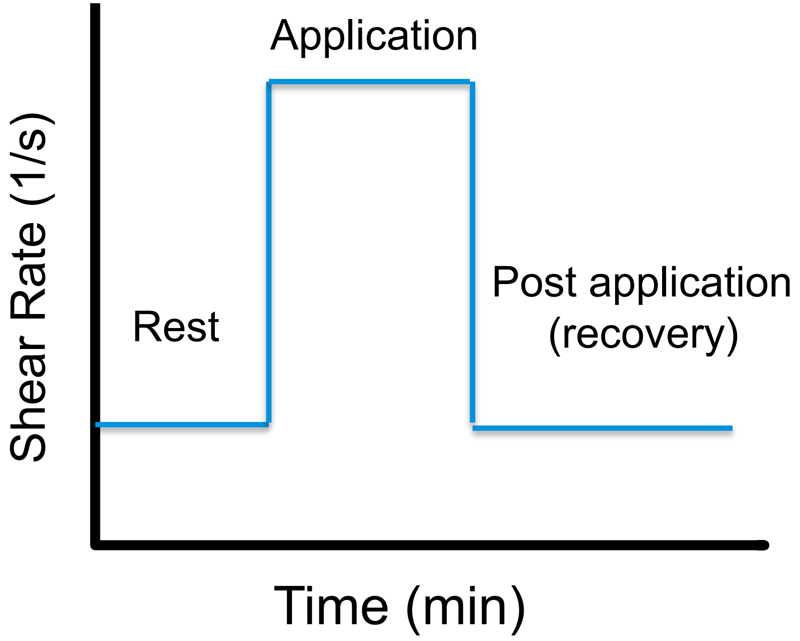
- Thixotropy is a time-dependent shear thinning property, which is used to characterize structure change reversibility
- A thixotropic fluid takes a finite time to attain equilibrium viscosity when introduced to a step change in shear rate
- Thixotropy is a desired property for many applications such as:
  - Control sagging and levelling of paints
  - Start up of pipleline flow after rest

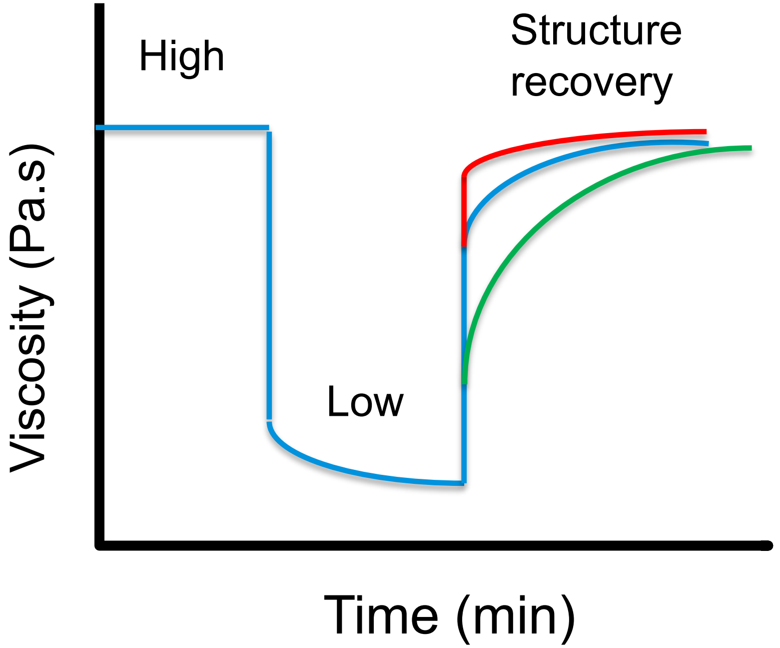
How to Measure Thixotropy

- Thixotropy can be quantitatively measured on a rotational rheometer
- Common methods
  - Stepped flow method
  - Stepped dynamic method
  - Stress ramp up and down method (Thixotropic loop)
  - Dynamic time sweep after pre-shear method

Note:
Thixotropic behavior is a time dependent characteristic. Measured thixotropy will vary depending on experimental parameters.

Stepped Flow Method





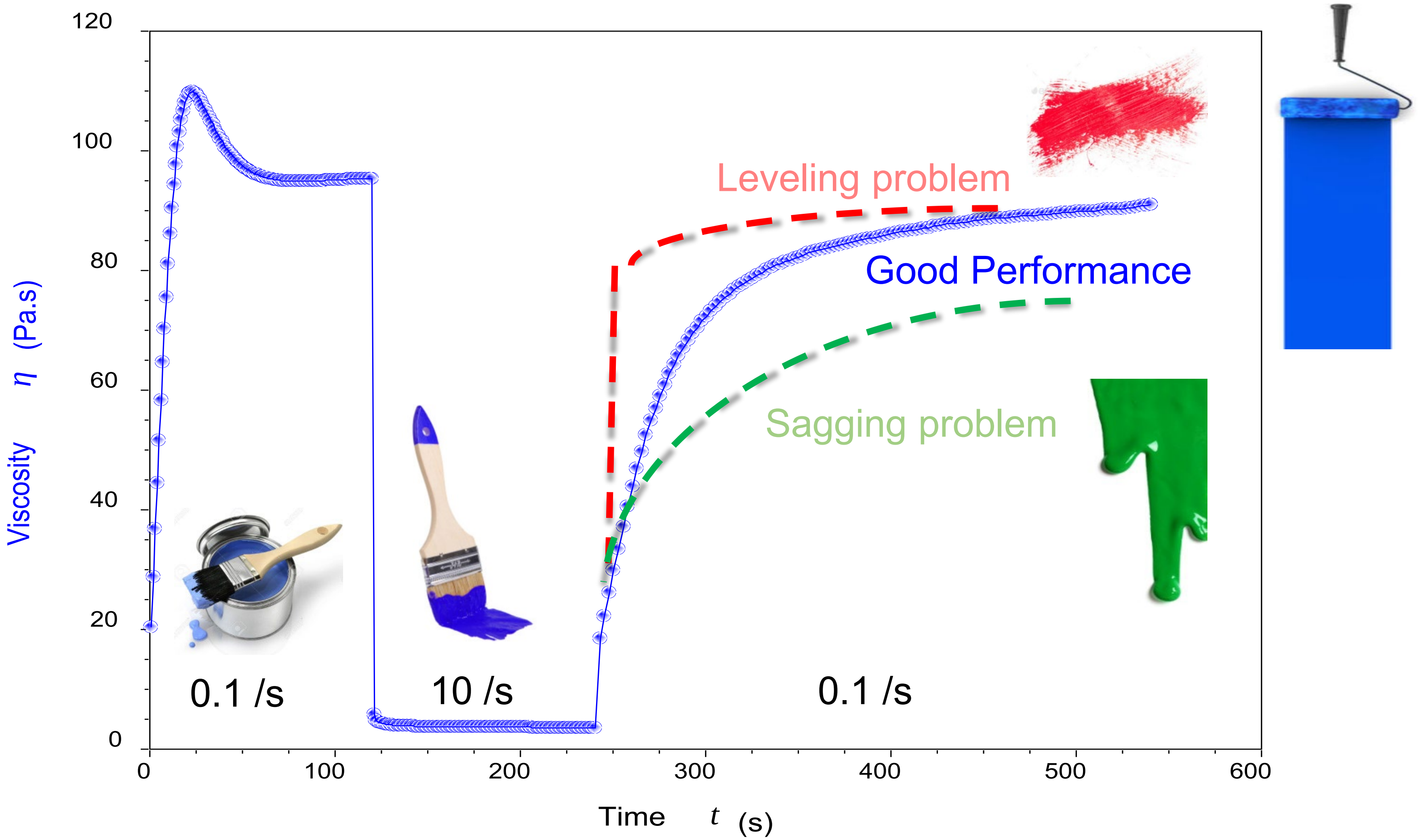
Experimental:

Step 1: Low Shear (e.g. 0.1 1/s), state of rest

Step 2: High Shear (e.g. 10 1/s), structural destruction

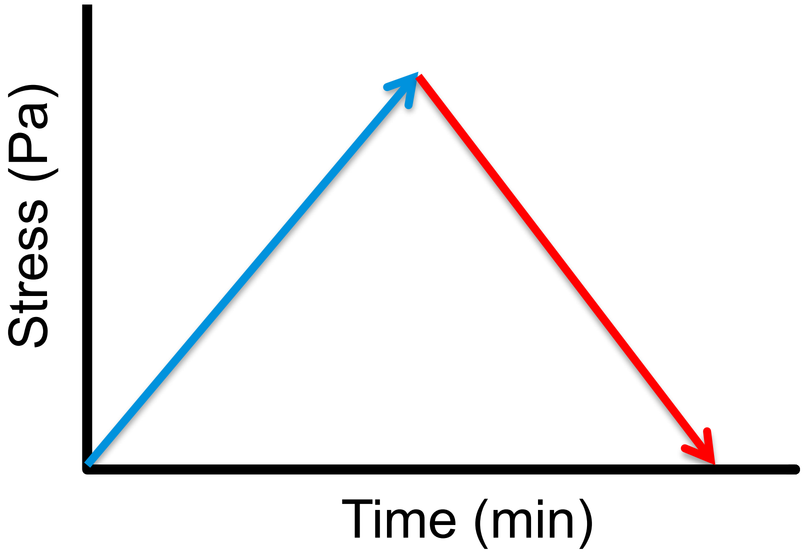
Step 3: Low Shear (e.g. 0.1 1/s), structural regeneration

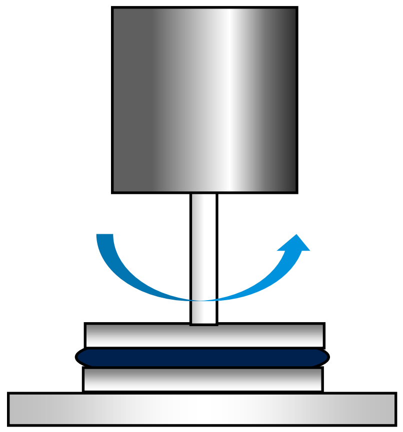
Thixotropic Analysis of a Blue Paint



Chen apps note: RH106

Stress/Rate Ramp Up and Down Method

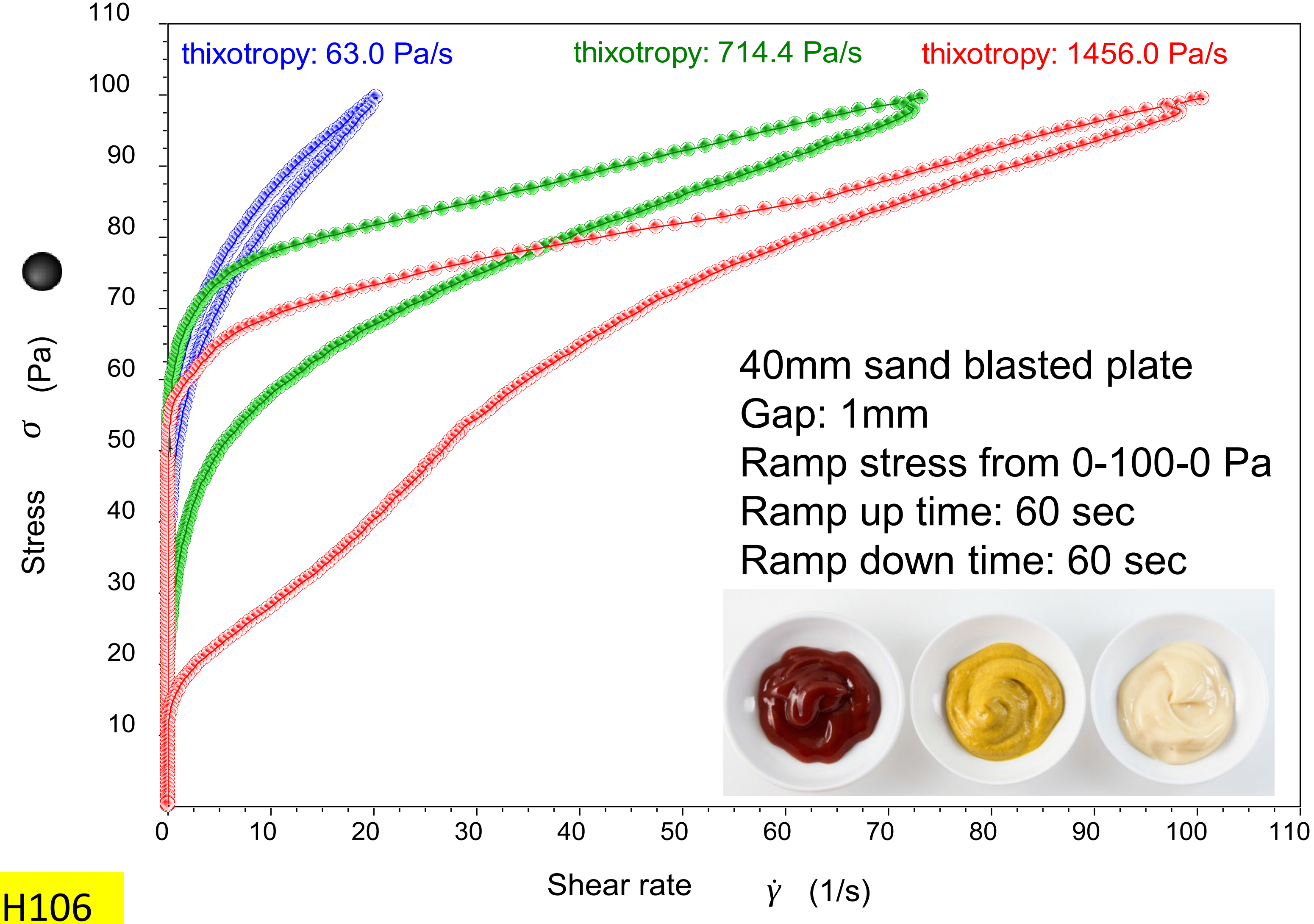




- Ramp shear stress linearly from zero up until sample flows, then ramp stress back down to zero
- Thixotropic index is measured by taking the area between the up and down stress curves
- TA Tech Tip: https://www.youtube.com/watch?v=8lZangOp1SY

Thixotropic Loop Testing on Foods

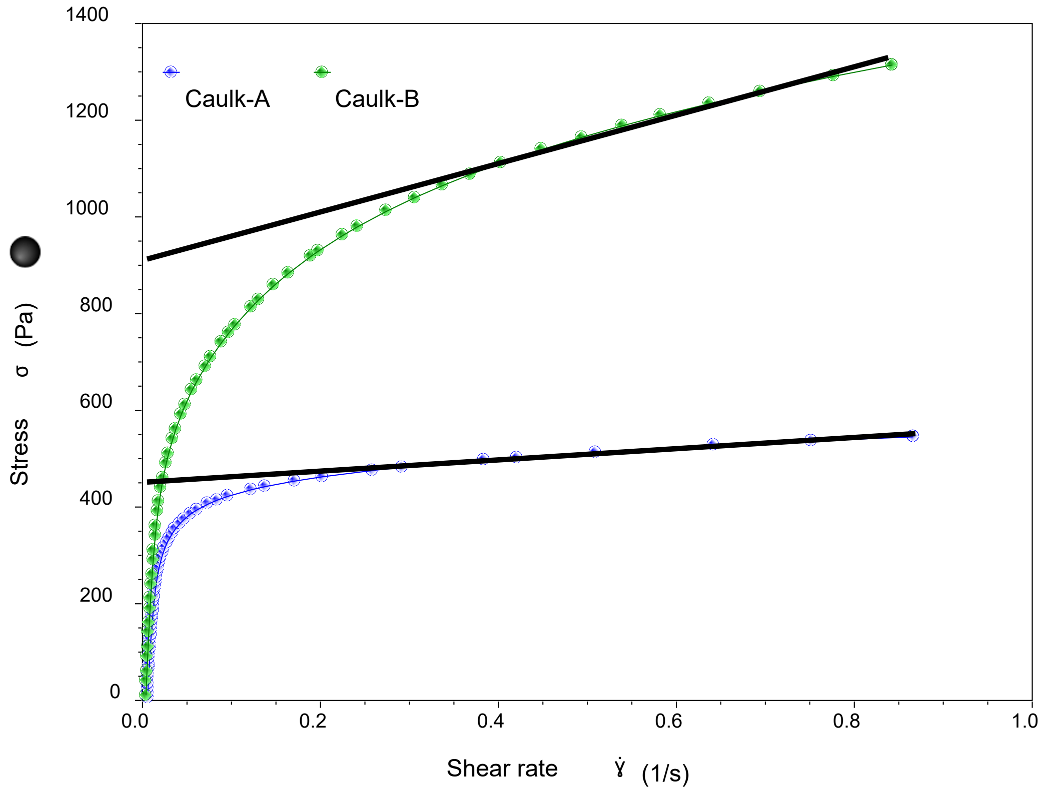
Mayonnaise, Yellow Mustard, and Ketchup

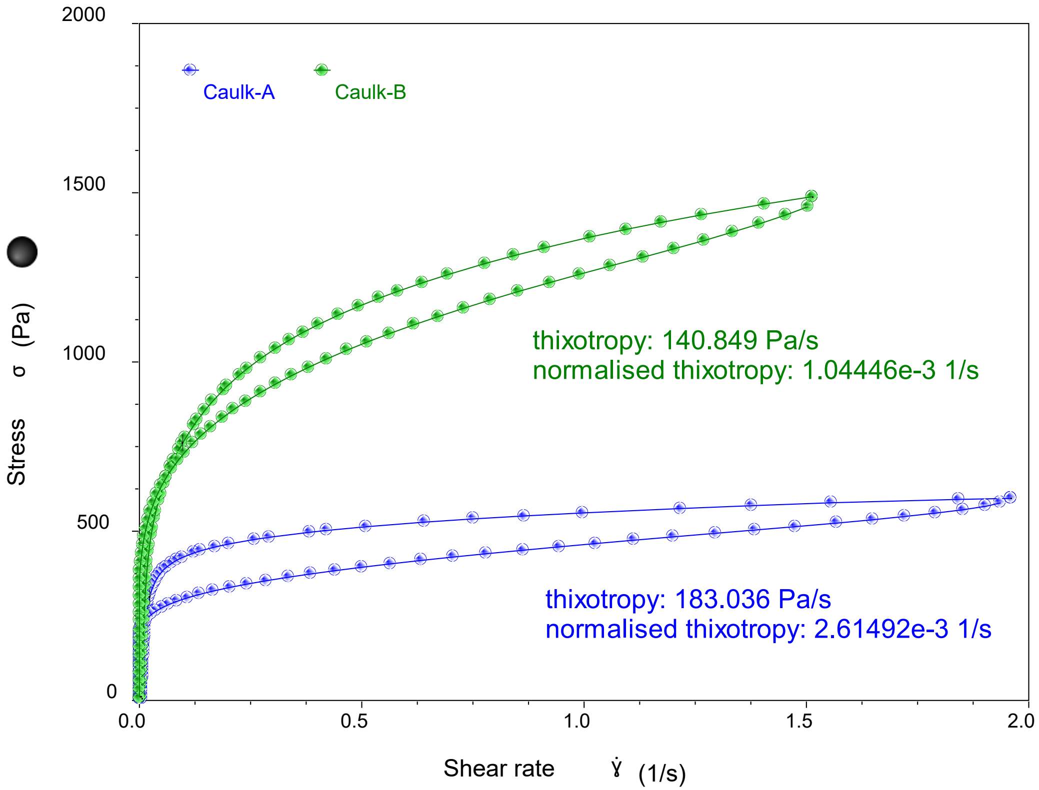


Chen apps note: RH106

Caulk - Yield and Thixotropy

- Caulk exhibits a yield behavior. Below the yield stress, caulk does not flow
- Caulk shows thixotropic property, which can be measured using a flow loop test





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TA Practical Series Training Course

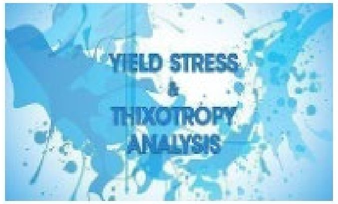
https://www.tainstruments.com/a-practical-approach-to-rheology/

- How to measure Yield stress
- How to measure Thixotropy
- How to avoid wall slip and edge fracture
- How to fit flow curves with models

Viscosity Measurements on Liquids

The second chapter this four part series on rheology will cover viscous behaviors of liquids.

View Archive

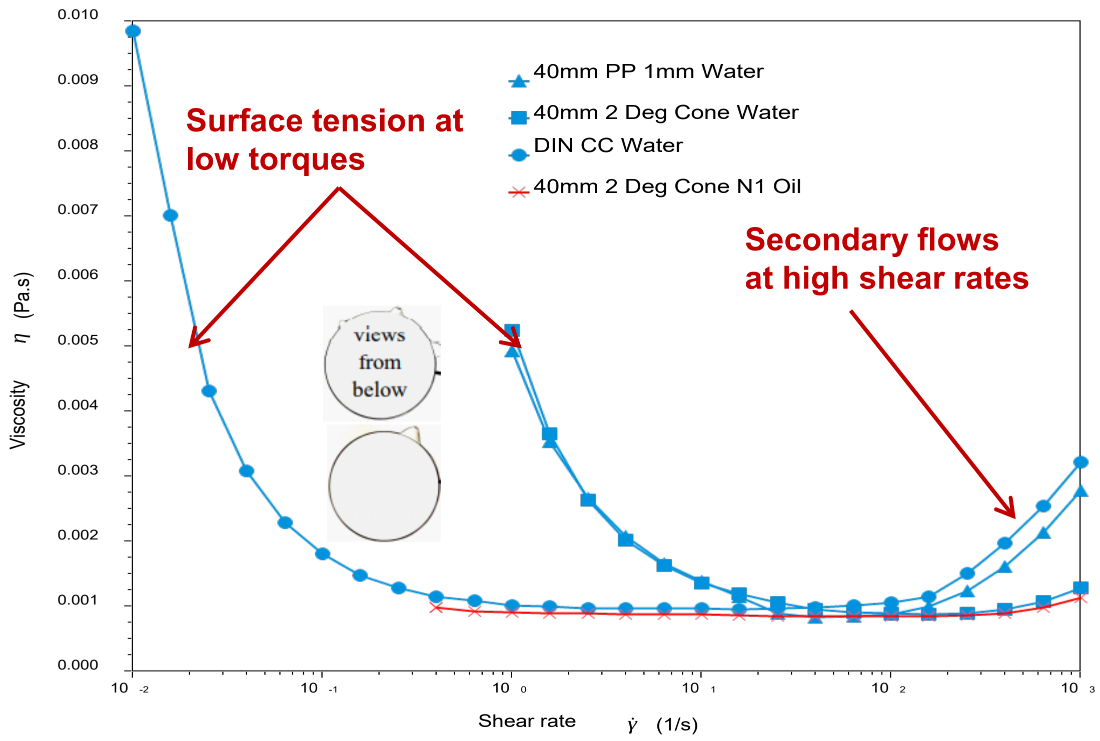


Yield and Thixotropy Measurements

Beyond viscous behavior, complex fluids can exhibit other important flow characteristics, most notably yield stresses and thixotropy. In this third part, we will explore effective ways to quantify these behaviors.

Water is not a Good Viscosity Standard

- Surface tension causes artifact shear thinning under low torque
- Secondary flow shows artifact shear thickening under high shear
- Use a large diameter geometry with a smaller gap

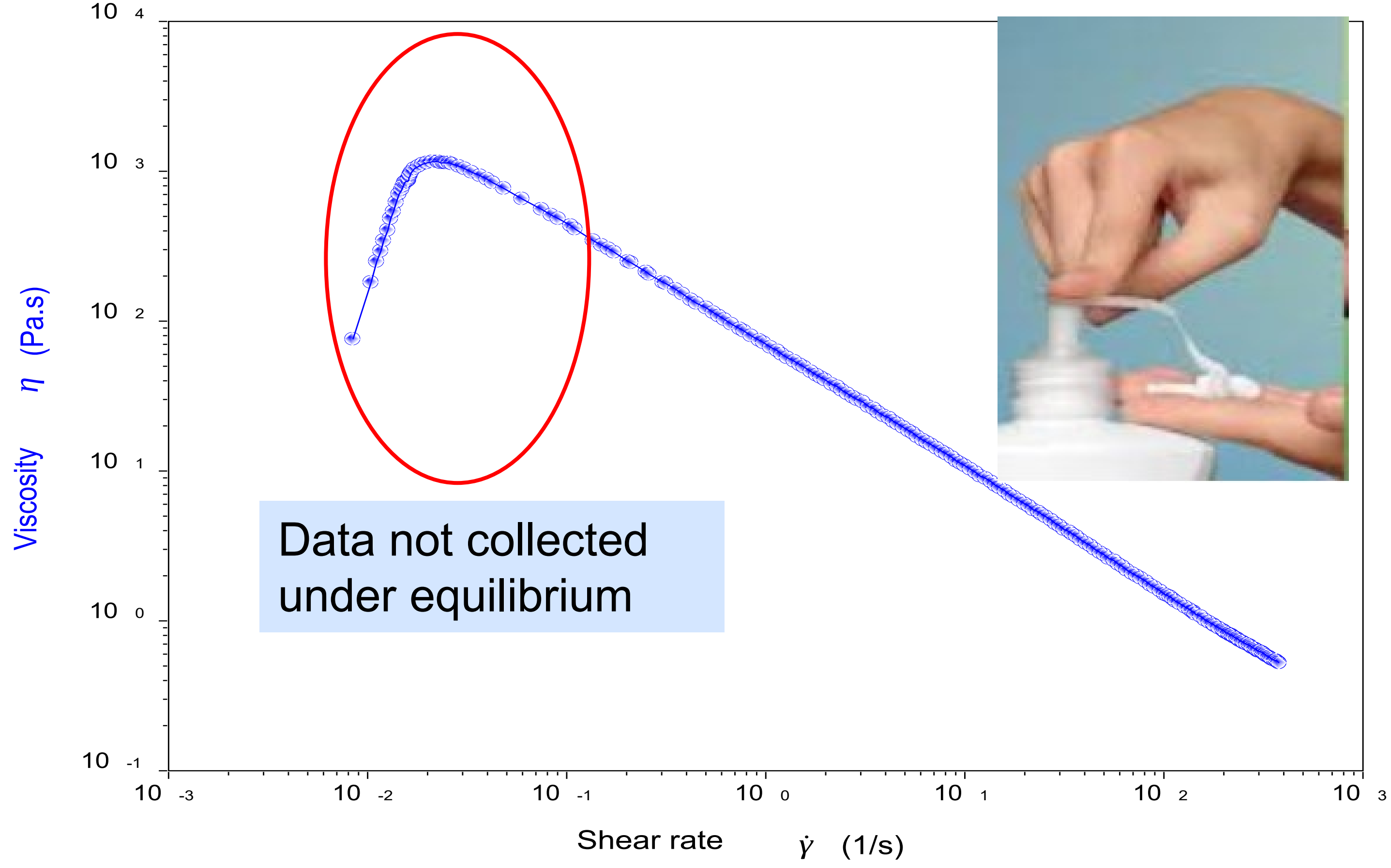


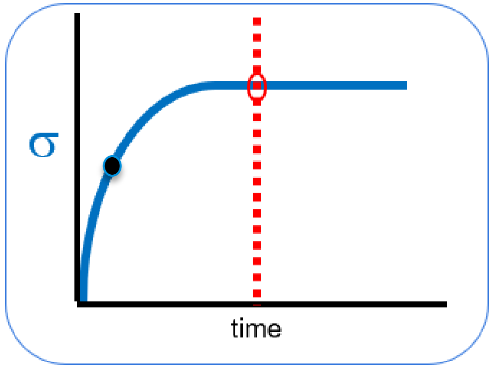
- TA Webinar - Professor Randy H. Ewoldt
  http://www.tainstruments.com/randy-h-ewoldt-experimental-challenges-of-shear-rheology-how-to-avoid-bad-data-2/

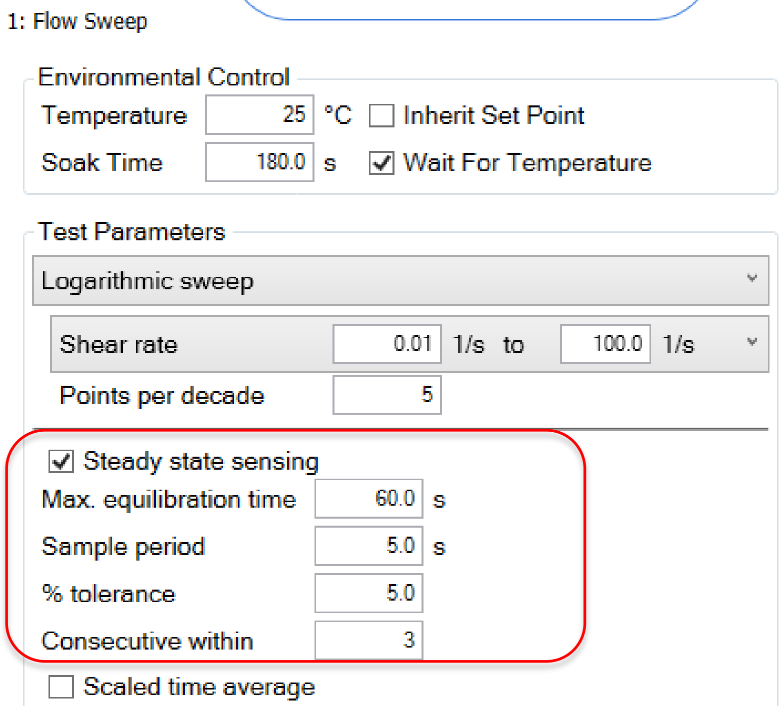
Ewoldt R.H., Johnston M. T., Caretta L.M., "Experimental challenges of shear rheology: how to avoid bad data", in: S. Spagnolie (Editor), *Complex Fluids in Biological Systems*, Springer (2015) 1-36

What is wrong with viscosity under low shear rates?

- Stress ramp from 0 to 200 Pa within 60 seconds.

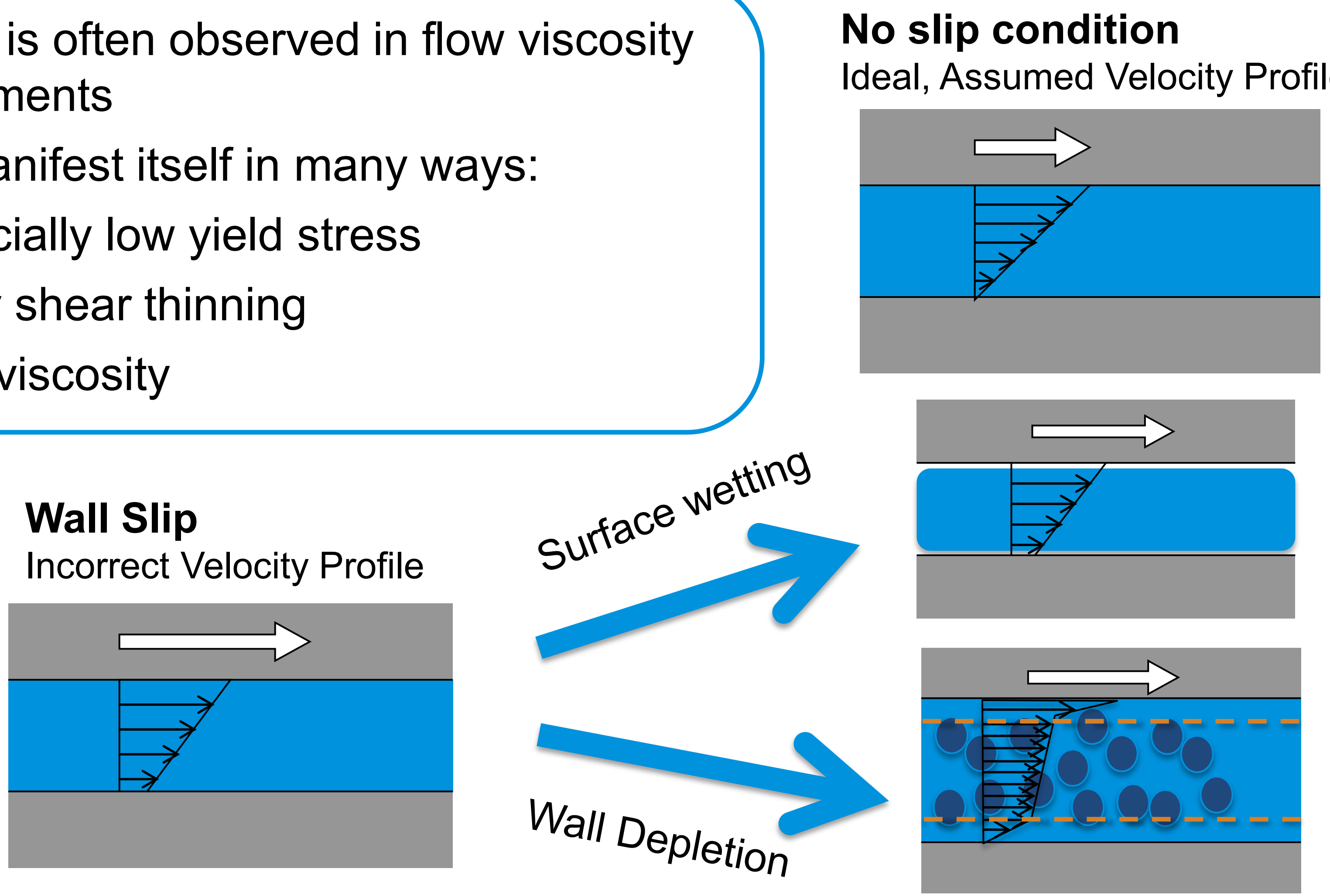






Wall Slip Phenomena

- Wall slip is often observed in flow viscosity measurements
- It can manifest itself in many ways:
  - Artificially low yield stress
  - Early shear thinning
  - Low viscosity



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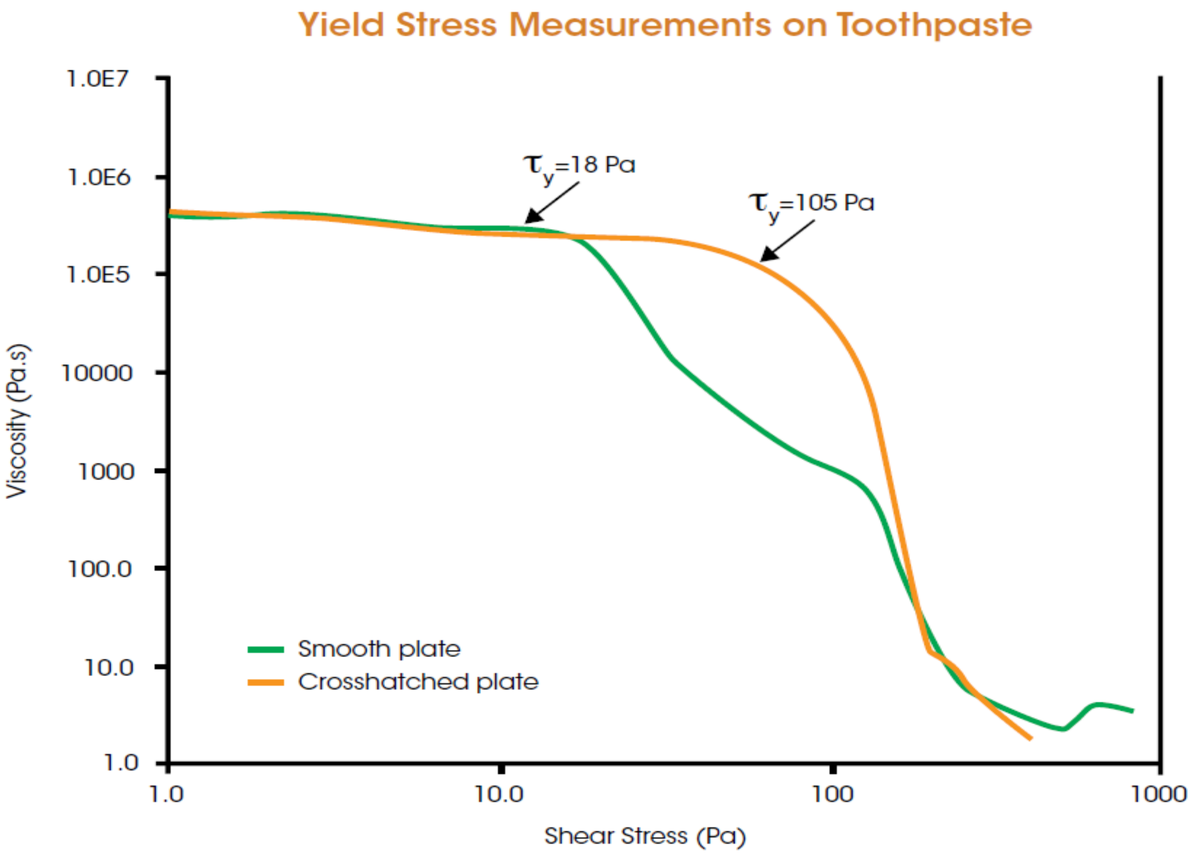
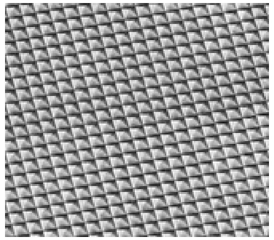
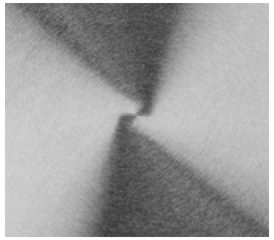
Wall Slip – Artifact Yield

- Incidence of wall slip is often observed when testing structured fluids
- Wall slip shows artifact yield





crosshatched plate



smooth plate

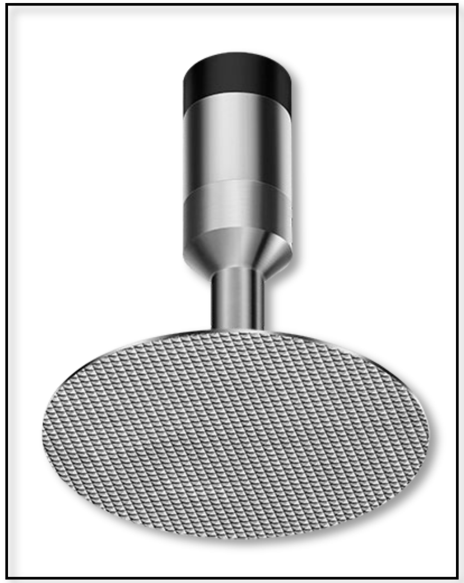
T Chen, Apps note RH094

Solutions To Minimize Wall Slip

- Diagnosis method
  - Running the same experiment at different gaps. For samples that don't slip, the results will be independent of the gap

- Solutions
  - Use a grooved cup with vane or helical shape rotor geometry
  - Use a roughened surface geometry

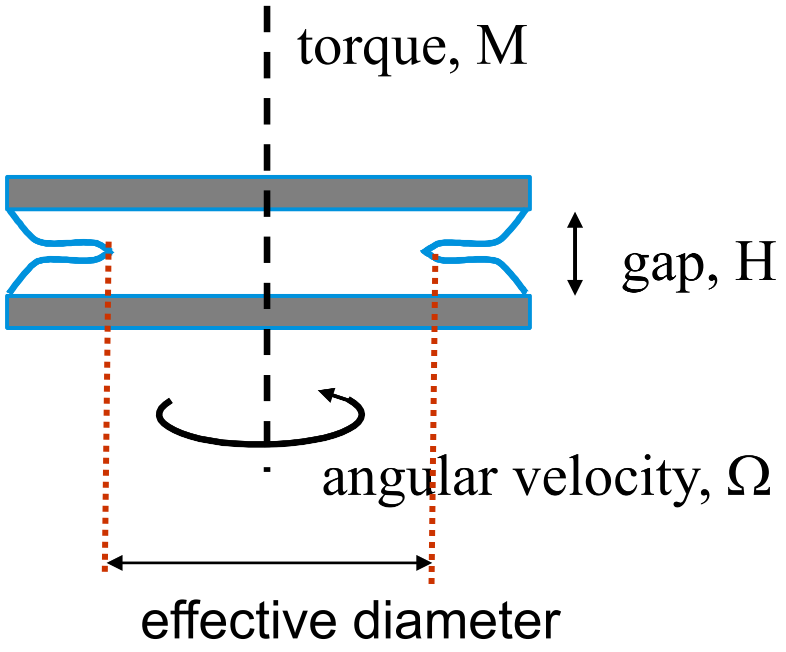


Edge Fracture

- Edge fracture is caused by the elasticity of the fluids
- When shearing a viscoelastic material, a large normal stress difference (created from its elasticity) can lead to a crack formation at the geometry edge. This is called edge fracture.
- Results: decrease in viscosity

- To minimize edge fracture
  - Decrease measurement gap
  - Use partitioned plate



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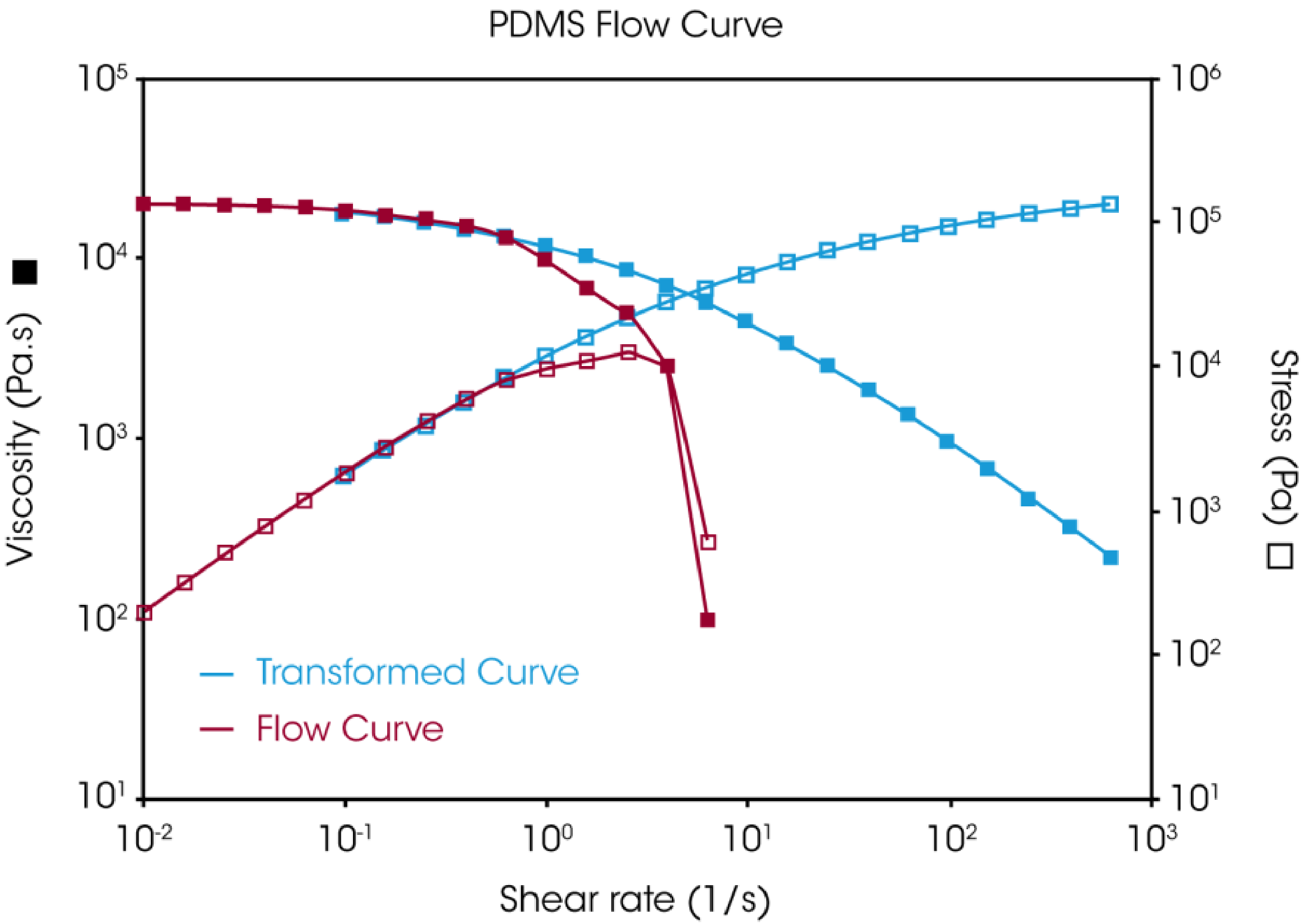
Flow Viscosity Testing Limited due to Edge Fracture

- Edge fracture occurs when testing the shear viscosity of molten polymers due to its viscoelastic nature
- Cox-Merz transformation: convert dynamic complex viscosity vs. frequency to transient shear viscosity vs. shear rate

$$\eta = \sigma/\dot{\gamma} \qquad \eta^* = G^*/\omega$$

$$\eta(\dot{\gamma}) \equiv \eta^*(\omega)$$

- The Cox-Merz transformation works primarily with polymer melts and polymer solutions



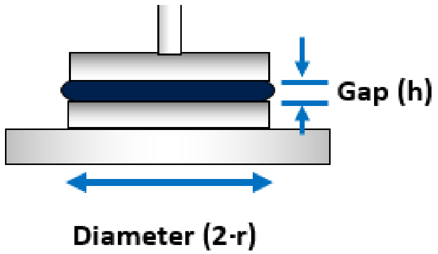
Challenges for High Shear Rate Testing

- Max velocity on rheometer (Ω) : 300 rad/s
- What is the max shear rate the instrument/geometry can reach?
- Can a sample be tested up to these shear rate?

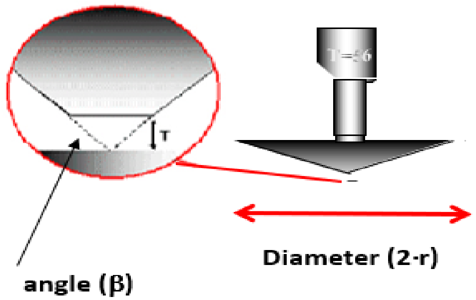
Geometry	Diameter (mm)	Cone angle (°)	Gap (um)	Sample volume (mL)	Max shear rate (approx.) 1/s
Cone and parallel plate	8	0	1000	0.05	**1,200**
	20	0	500	0.16	**6,000**
	40	0	1000	1.26	**6,000**
		0	100	0.13	**60,000**
		0	10	0.013	**600,000**
		1	25	0.29	**17,200**
		2	52	0.59	**8,600**
Concentric cylinder	DIN rotor			22.0	**3,656**
	Recessed end rotor			7.5	**3,656**

$$\dot{\gamma} = \Omega \cdot K_{\gamma}$$

Parallel plate: $K_{\gamma} = \frac{r}{h}$



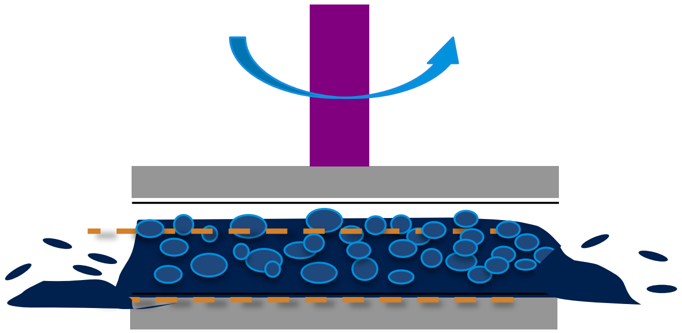
Cone plate: $K_{\gamma} = \frac{1}{\beta}$



Challenges for High Shear Rate Testing

- The maximum measurable shear rate is often depending on the sample and not the instrument
  - Turbulence affects the viscosity reading
  - Sample spin out of the plate or splash out of the cup
  - Shear heating
- Solutions
  - Reduce test gap
  - Use a capillary rheometer
  - Use flow model fitting to predict high-shear viscosity

Sample spin out of the plate at high shear

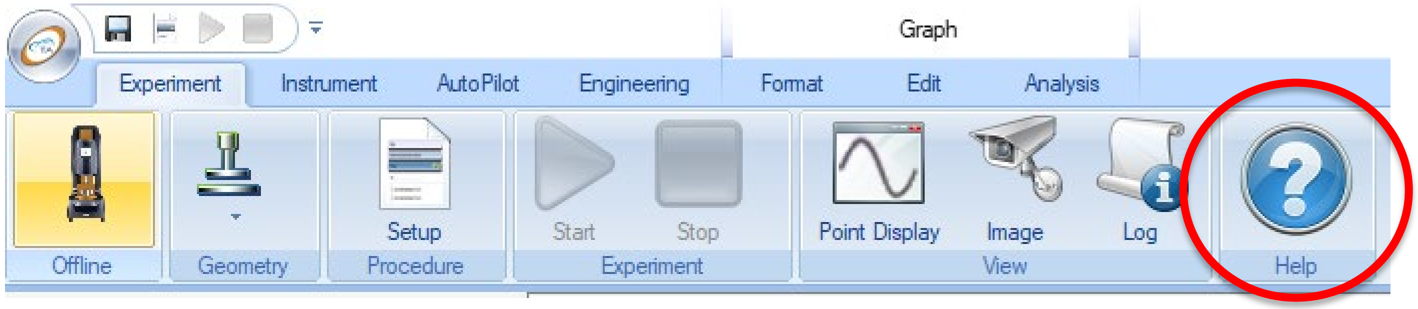


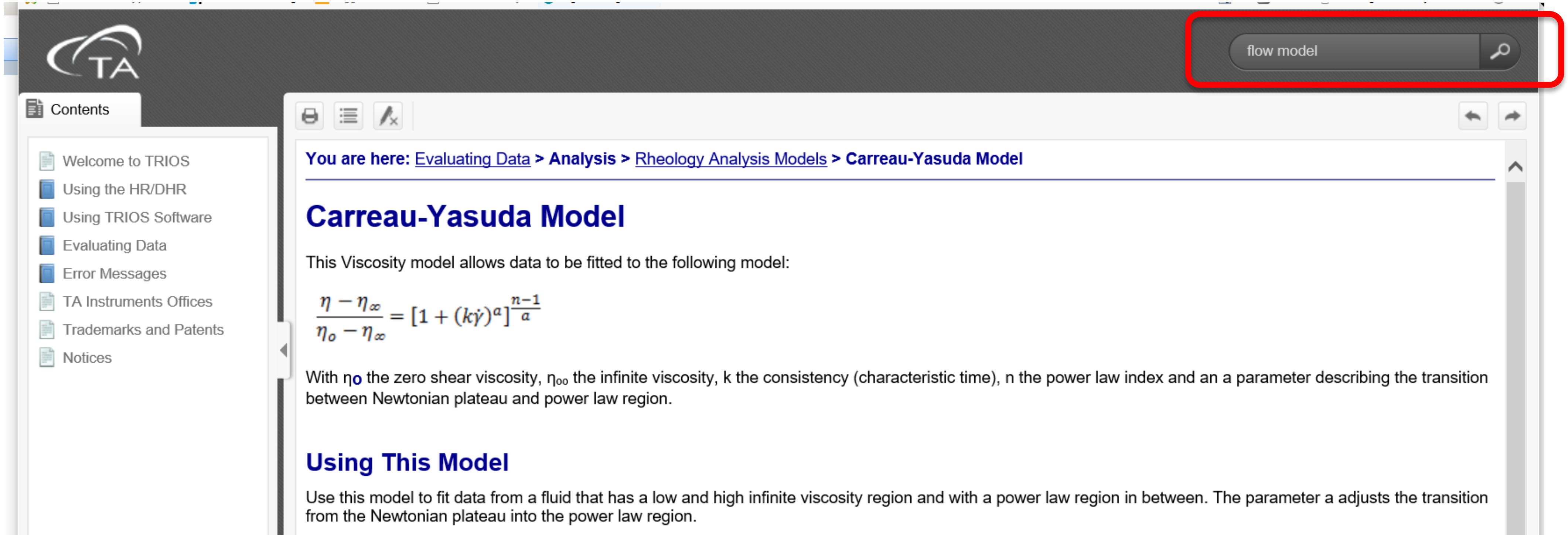
Use a capillary rheometer

Fit the Flow Curve to a Mathematical Model

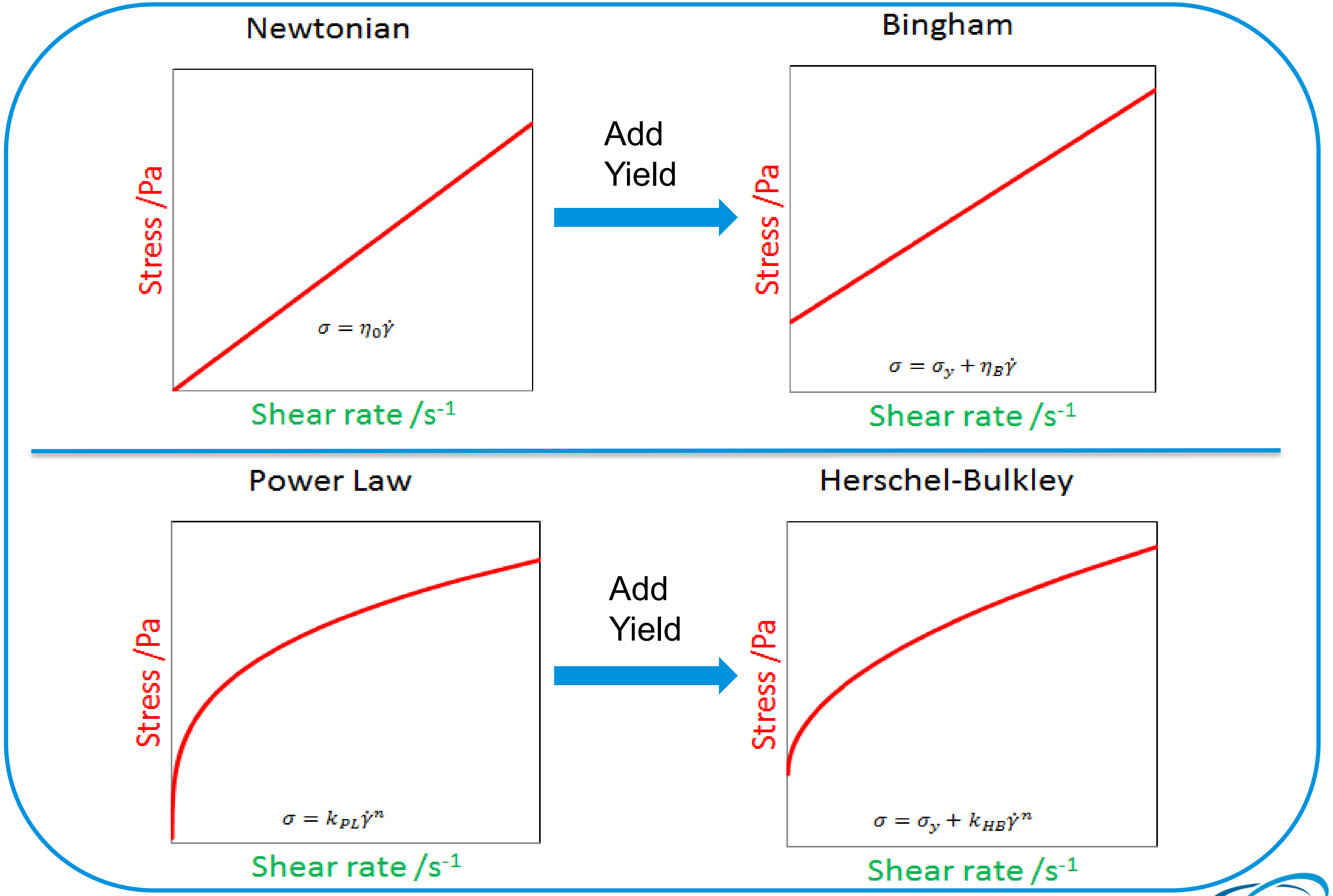
- Fit viscosity curves with mathematical models
  - Newtonian model
  - Power law model
  - Bingham model
  - Williamson model
  - Sisko model
  - Herschel-Bulkley model
  - Cross model
  - Carreau model
  - Carreau-Yasuda model
  - Casson model
  - Ellis model
- Extrapolate to zero shear and infinite shear viscosity
- Calculate yield stress
- Calculate viscosity at a specific point

Where to Find Model Equations



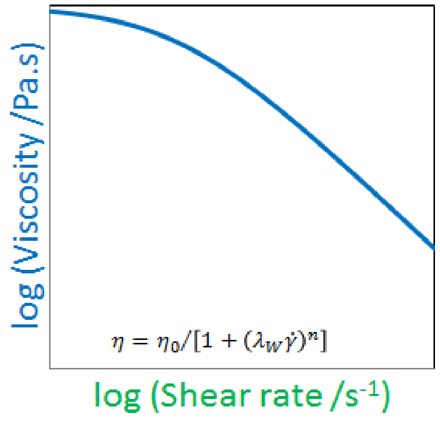


Example Flow Models



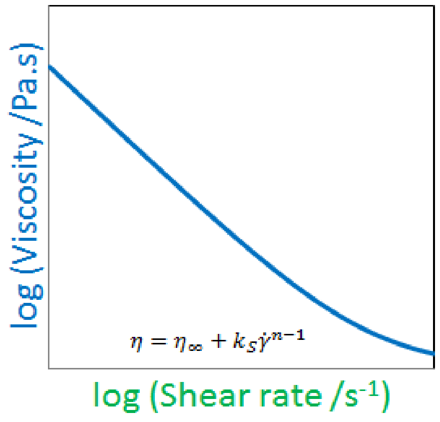
Example Flow Models– Continued

Williamson



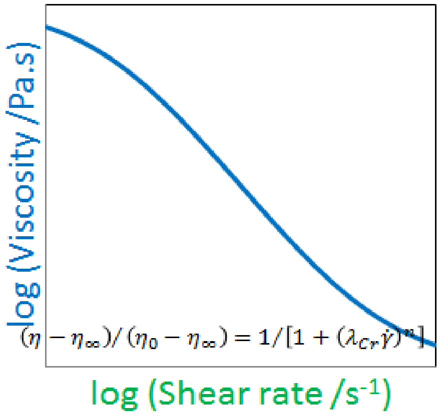
- **Zero Shear Viscosity**

Sisko

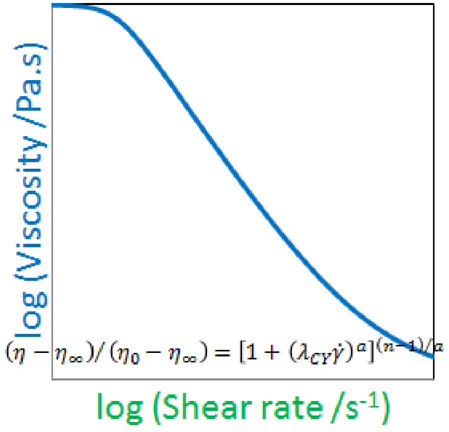


- **Infinite Shear Viscosity**

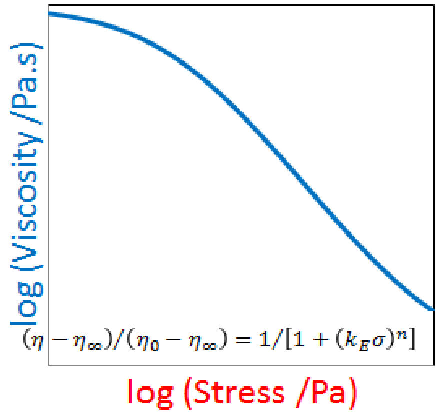
Cross



Carreau-Yasuda



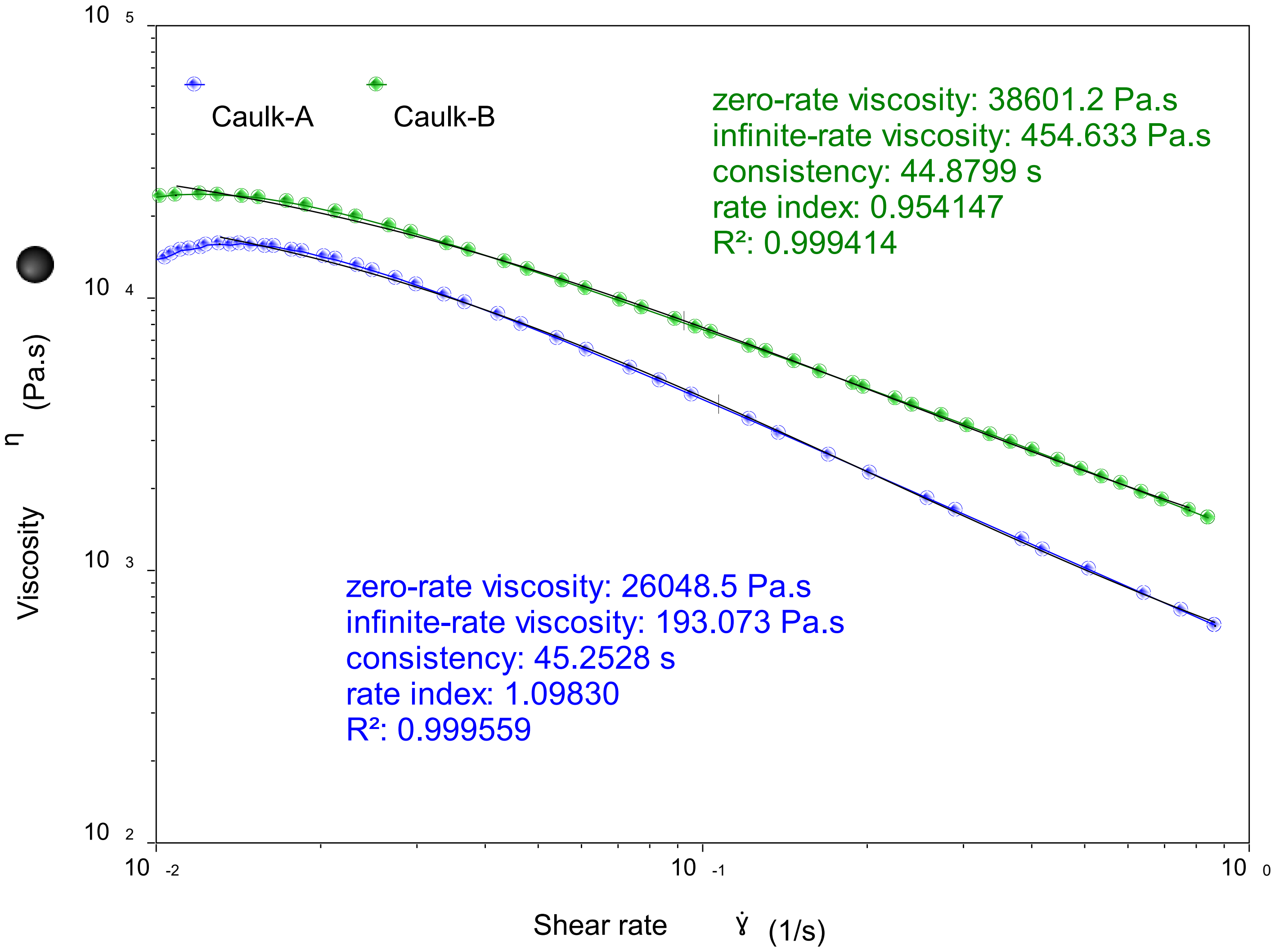
Ellis

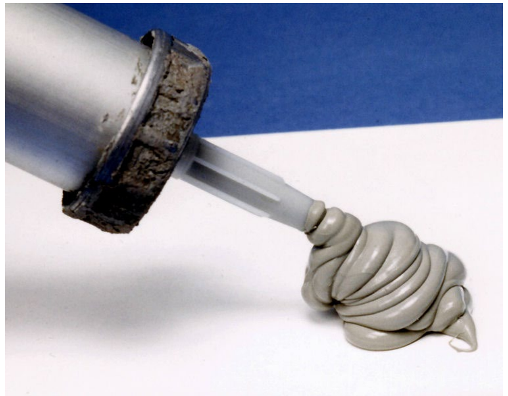


- **Both Zero Shear and Infinite Shear Viscosity**

Caulk – Viscosity Measurements

- Slippage and edge fracture are observed before the shear rate reached 1 1/sec.

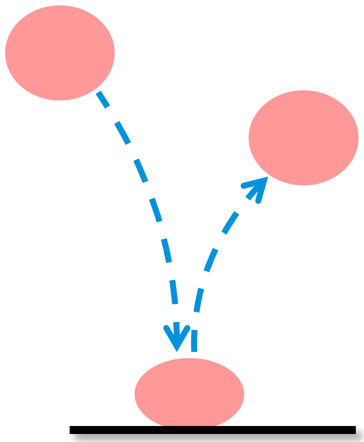


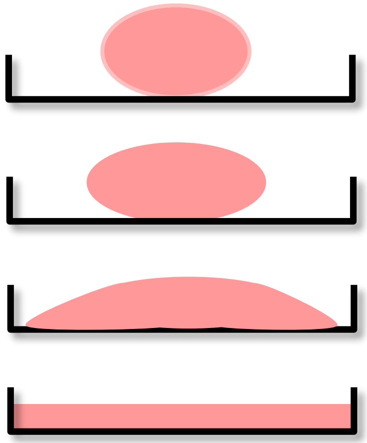


Rheology Applications
Oscillation Analysis - Tips and Tricks

Not Everything Flows

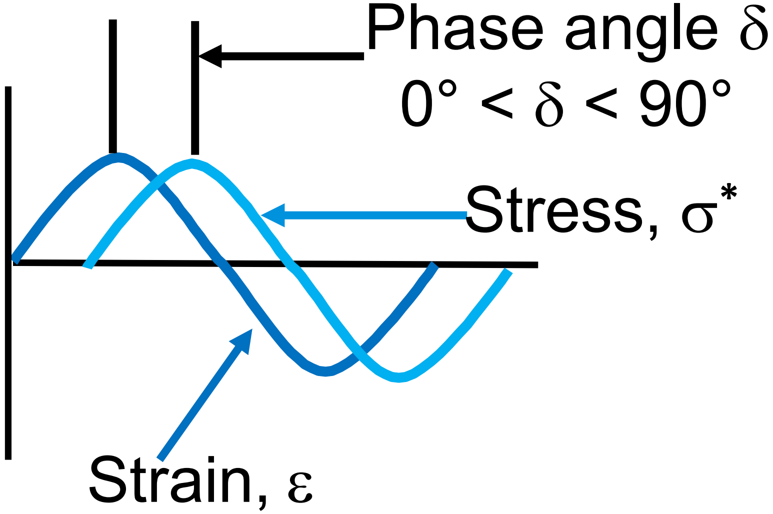
- **Liquid** - flows freely, remains at constant volume and takes the shape of its container
- **Solid** - has a fixed shape and volume, no flow
- **Semi-solid** – shows both viscous and elastic behavior. May flow under certain temperature or time scale



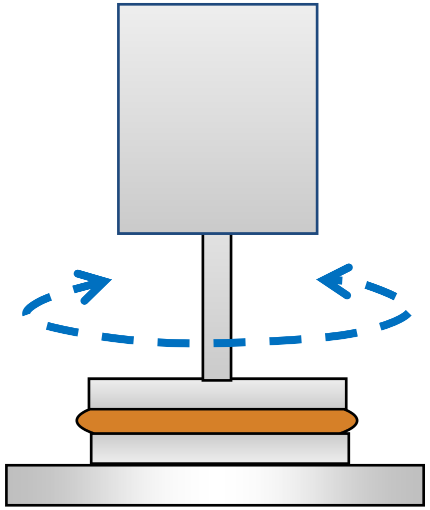


Dynamic Oscillatory Tests

- Apply a sinusoidal strain to the sample at a certain frequency
- Monitor sample response in stress
- The shift between the input strain and output stress is the phase angle



$$\gamma = \gamma_0 \cdot \sin(\omega t)$$
$$\sigma = \sigma_0 \cdot \sin(\omega t + \delta)$$



Viscoelastic Parameters

Complex Modulus: Measure of materials overall resistance to deformation

$$G^* = \left(\frac{\text{Stress}^*}{\text{Strain}}\right)$$

Elastic (Storage) Modulus: Measure of elasticity of material and ability to store energy

$$G' = \left(\frac{\text{Stress}^*}{\text{Strain}}\right)\cos\delta$$

Viscous (loss) Modulus: The ability of the material to dissipate energy

$$G'' = \left(\frac{\text{Stress}^*}{\text{Strain}}\right)\sin\delta$$

Tan Delta: Measure of material damping

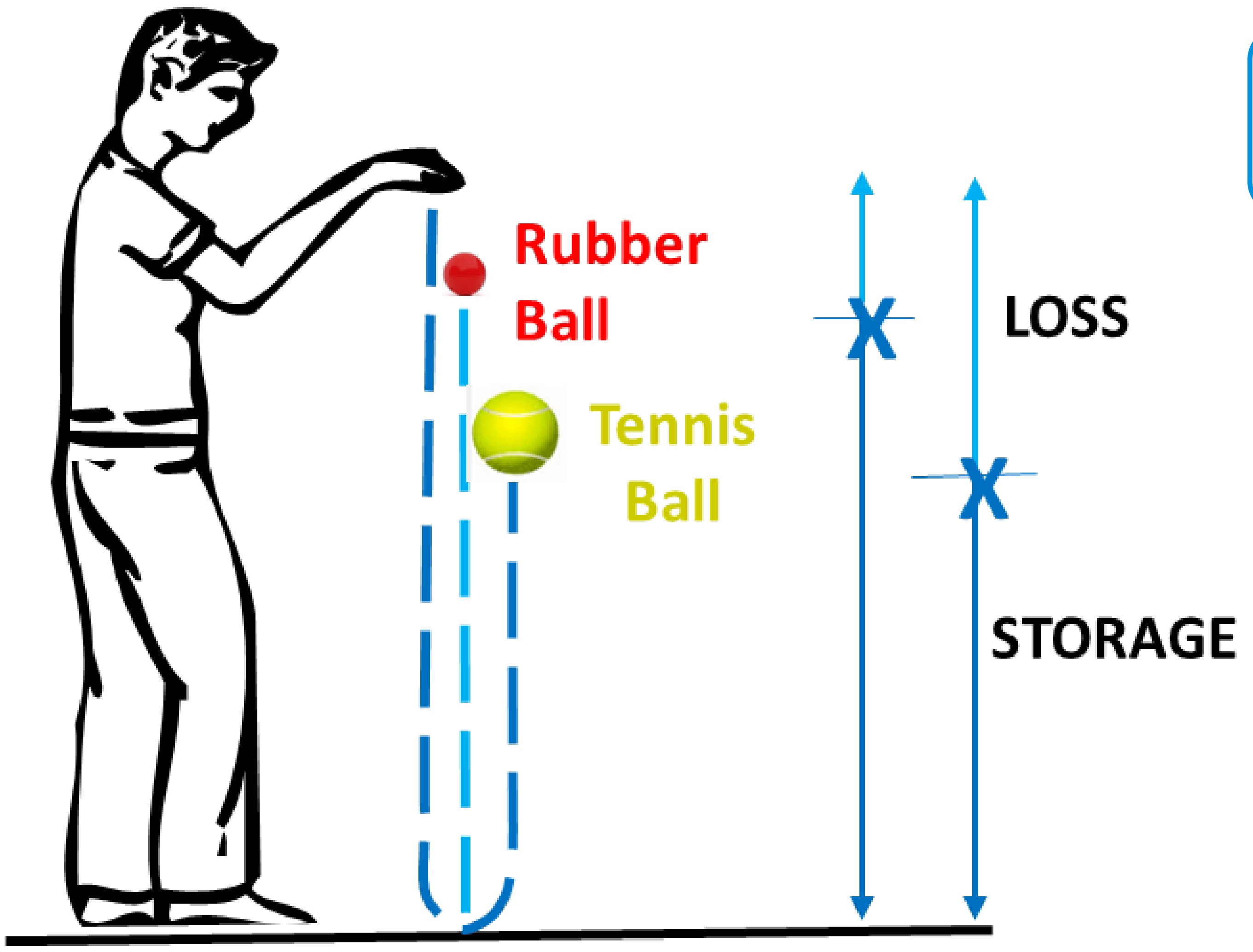
$$\tan\delta = \left(\frac{G''}{G'}\right)$$

Complex Viscosity: Viscosity measured in an oscillatory experiment (ω in rad/s)

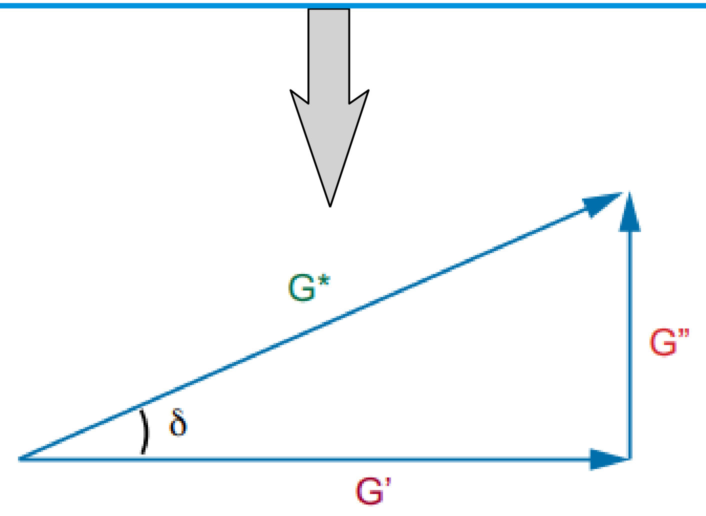
$$\eta^* = \left(\frac{G^*}{\omega}\right)$$

$$\eta^* = \eta' - i\eta''$$

Storage and Loss of a Viscoelastic Material



Dynamic measurement represented as a vector

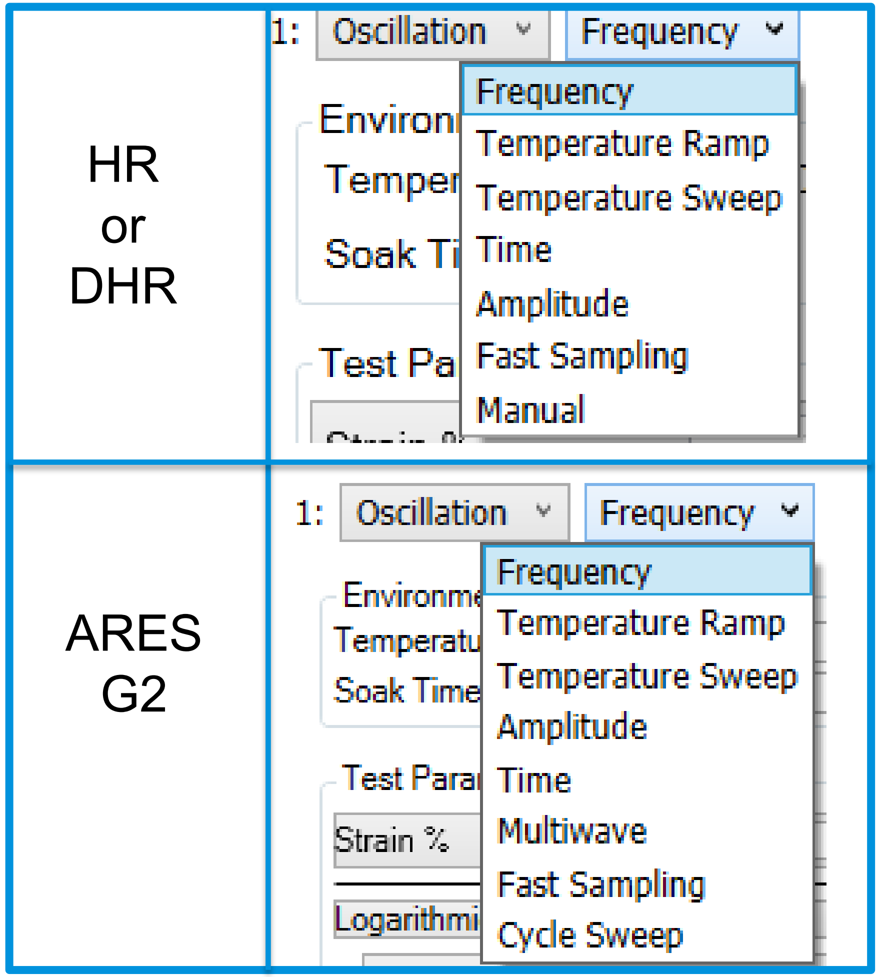


Rheological Parameters

- $G^* = \text{Stress}^*/\text{Strain}$
- $G' = G^* \cdot \cos\delta$
- $G'' = G^* \cdot \sin\delta$
- $\tan\delta = G''/G'$

Dynamic Oscillation Methods

- Stress, strain, or amplitude sweep
- Time sweep
- Frequency sweep
- Temperature ramp
- Temperature sweep (or step)
  - Time temperature superposition (TTS)



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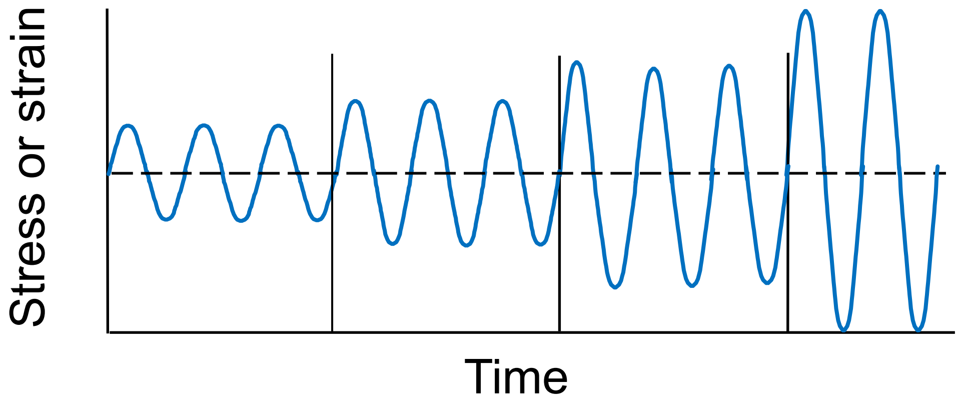
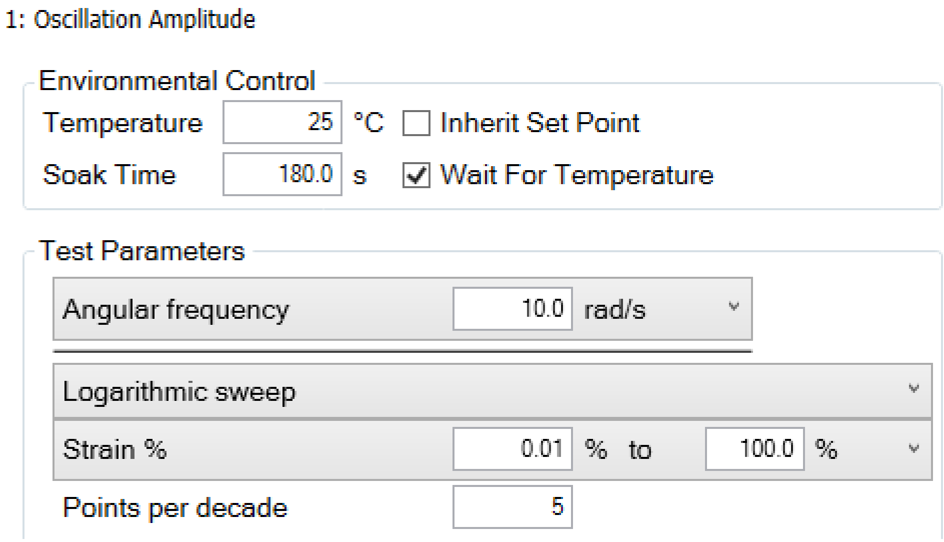
Dynamic Strain or Stress Sweep



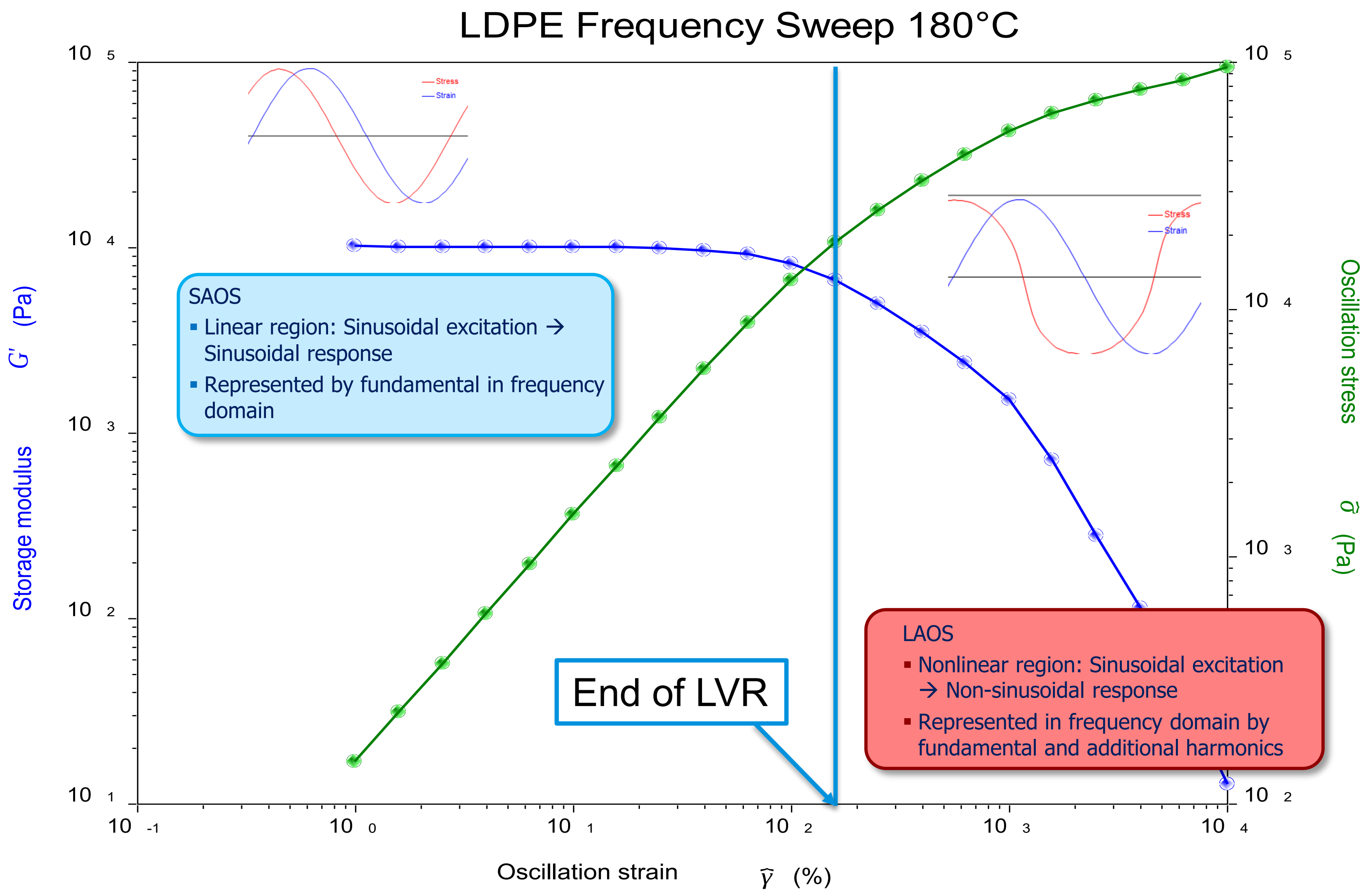
- The material response to increasing deformation amplitude (strain or stress) is monitored at a constant frequency and temperature
- In TRIOS: Amplitude



USES

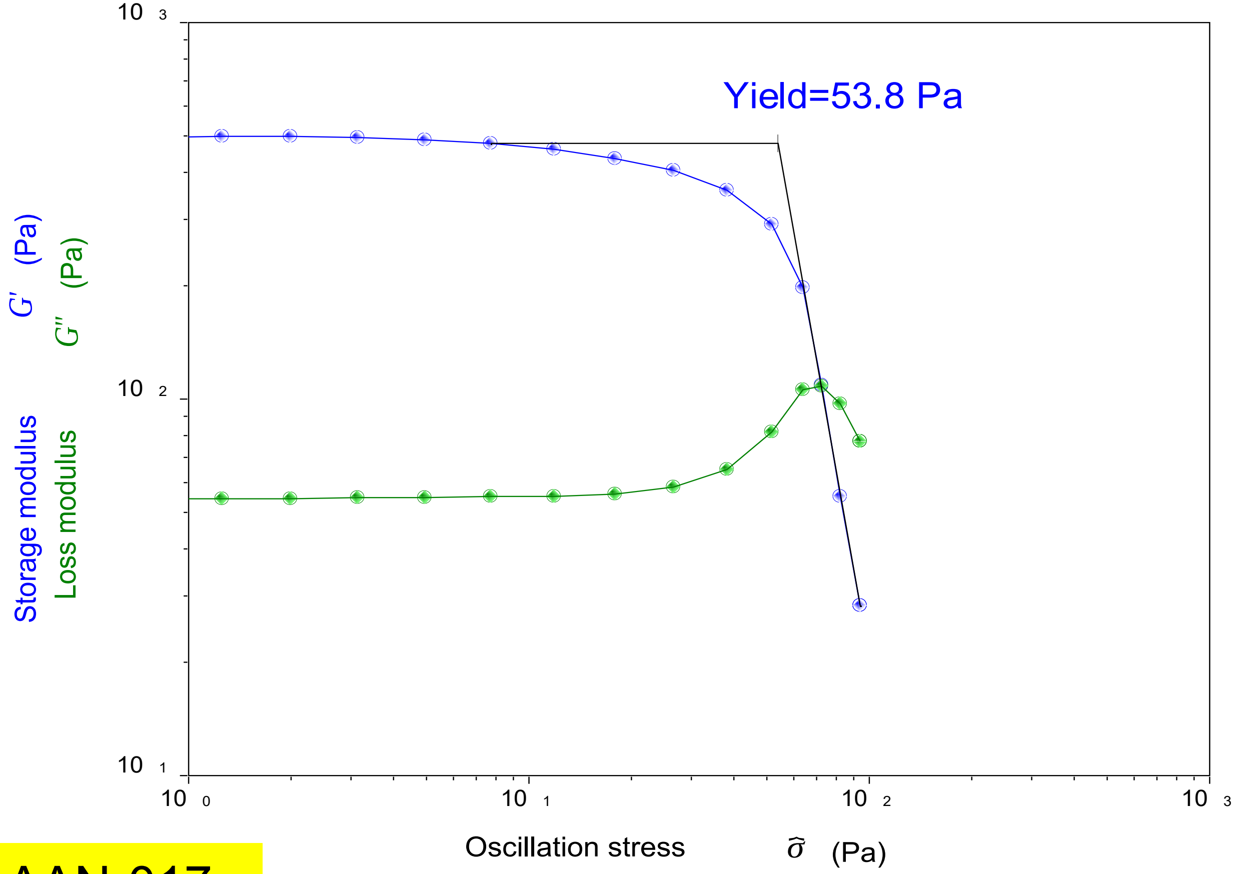
- Measure sample LVR
- Measure yield stress
- Measure non-linear viscoelastic properties (LAOS)

Linear and Non-linear Viscoelasticity



Yield Stress of Mayonnaise

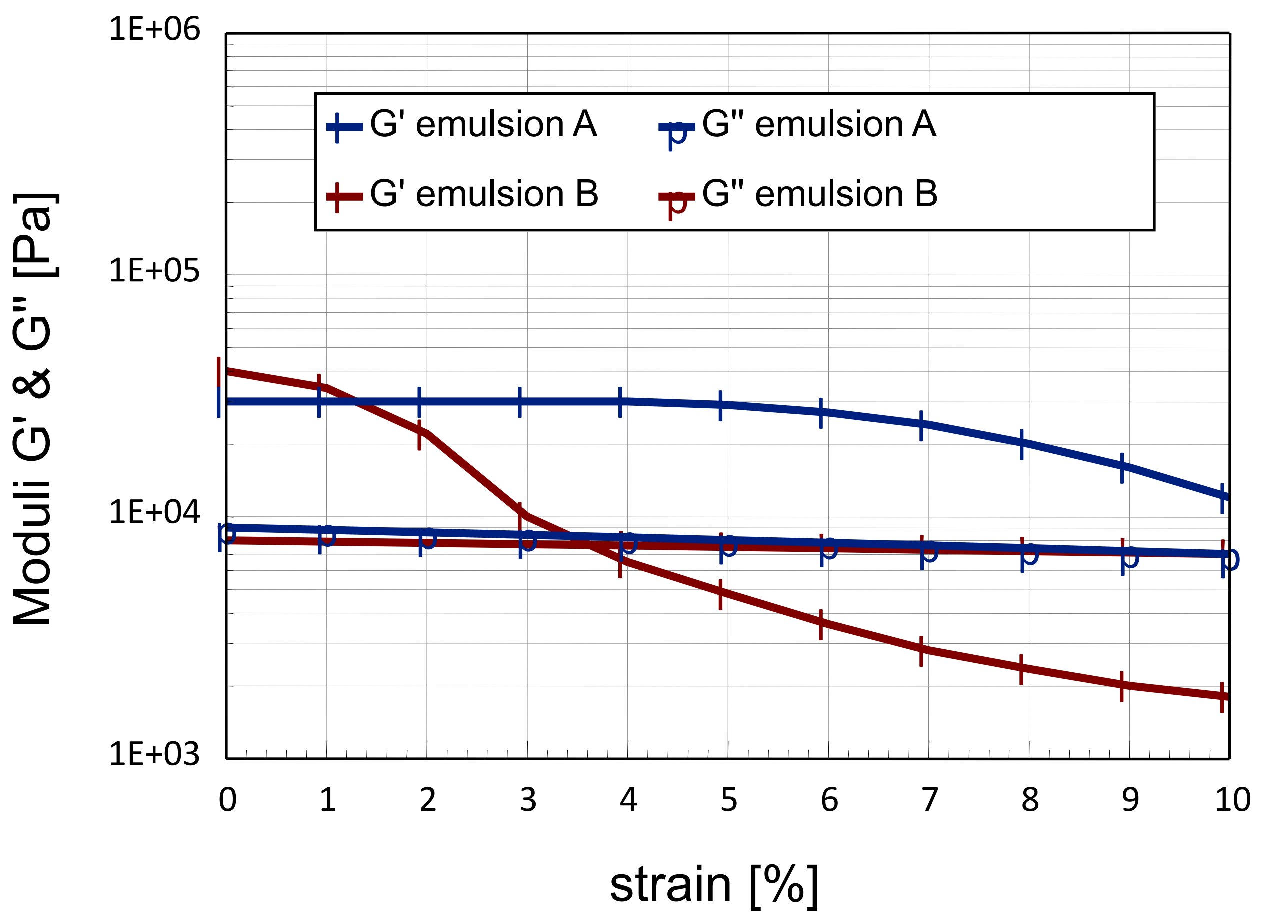
- Dynamic stress/strain sweep test on Mayonnaise
- Yield stress is signified at the onset of G' vs. stress curve
- Yield determined by this method indicates the critical stress at which irreversible plastic deformation occurs



Chen apps note: AAN-017

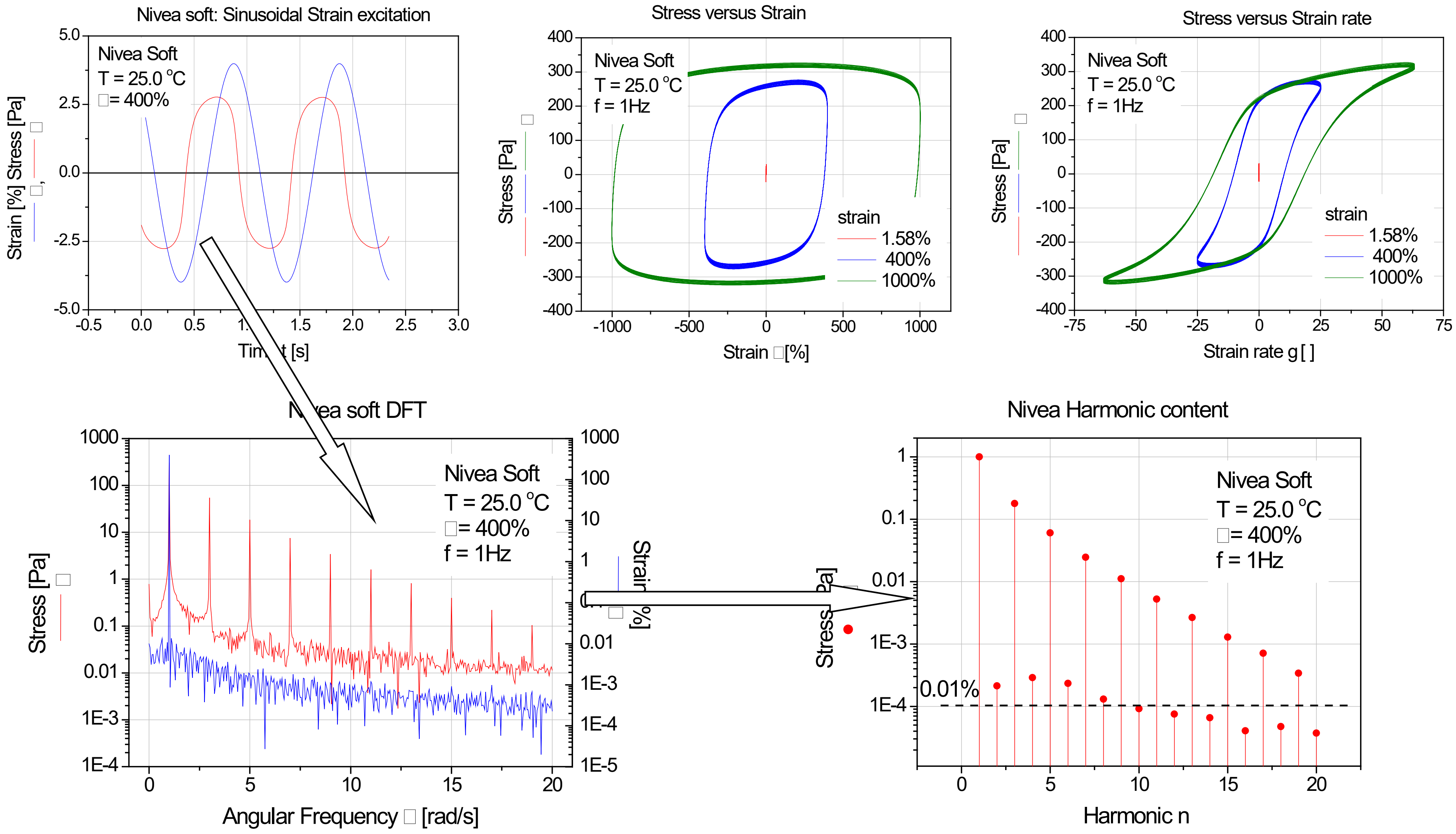
Creams/Lotions: Predict Stability

Stability, phase separation of a cosmetic cream



LAOS Analysis of a soft cosmetic cream

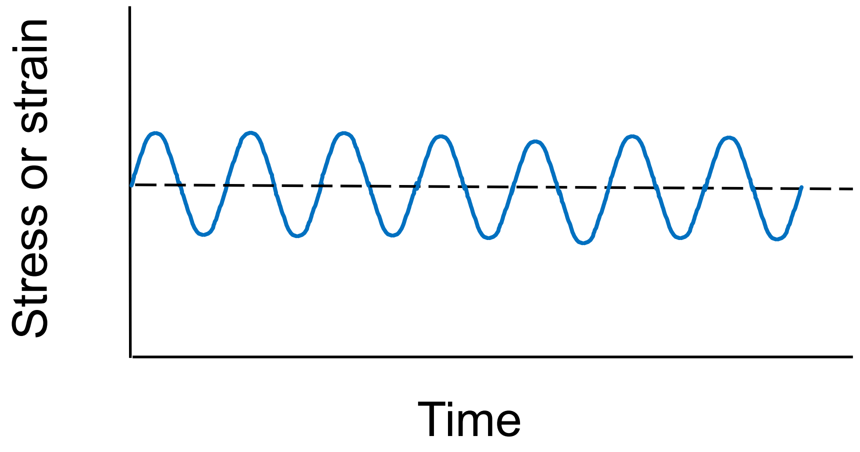
- Geometry: Sandblasted parallel plates



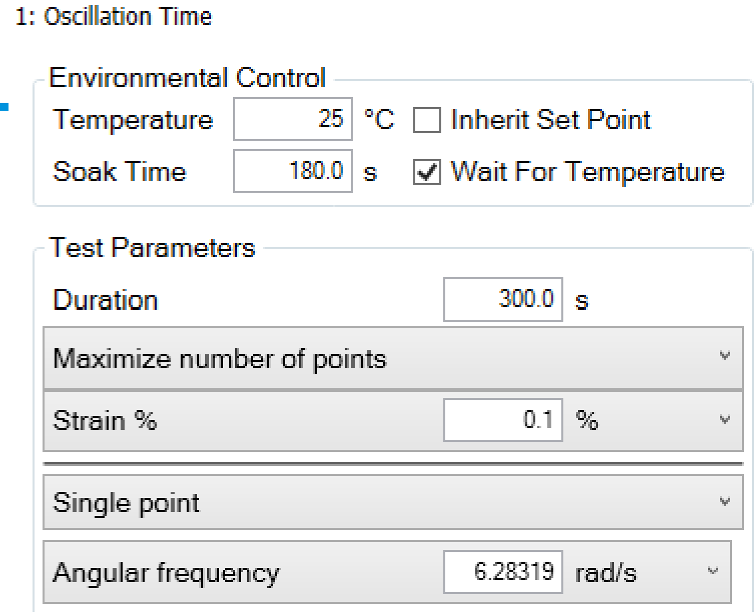
- TA Webinar - Professor Gareth H. Mckinley

https://www.tainstruments.com/rheological-fingerprinting-of-complex-fluids-ta-instruments-webinar/

Dynamic Time Sweep



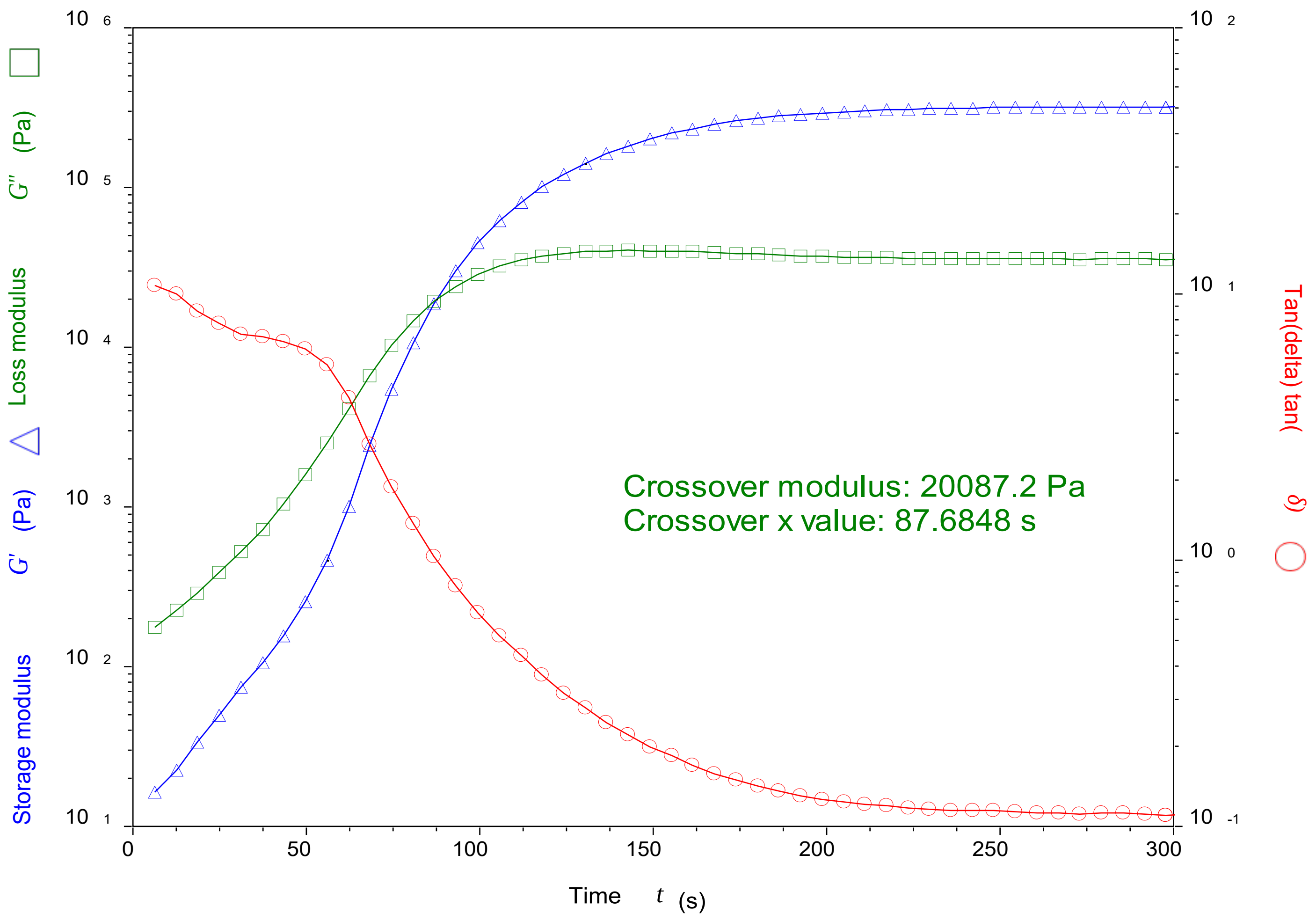
- The material response is monitored at a constant frequency, amplitude and temperature.
- In TRIOS: Time



<u>USES</u>

- Cure Studies
- Stability against thermal degradation
- Time dependent Thixotropy

Epoxy Curing

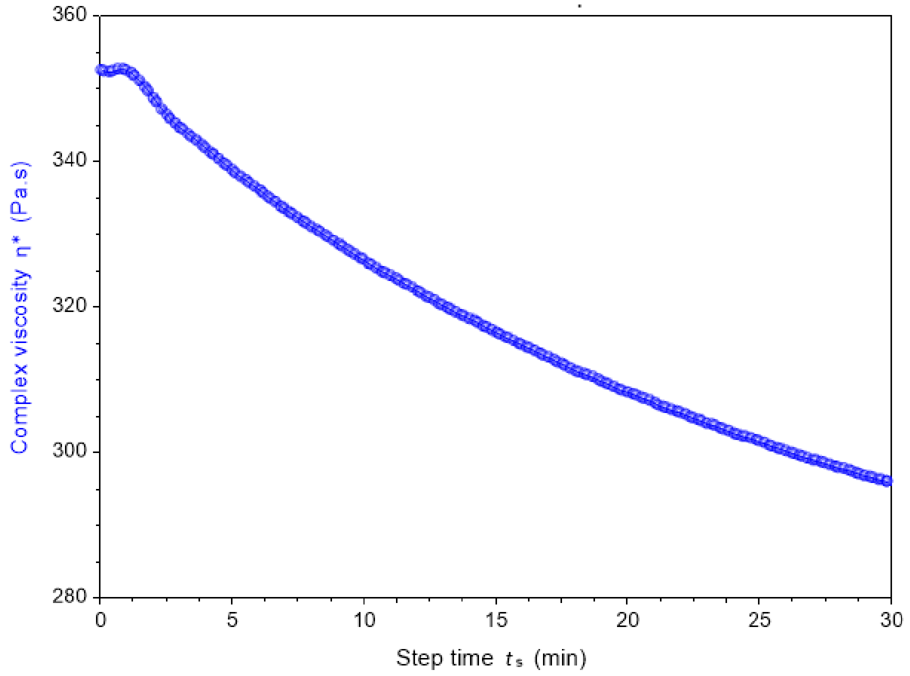


https://www.tainstruments.com/thermal-rheological-and-mechanical-characterizations-of-thermosets/

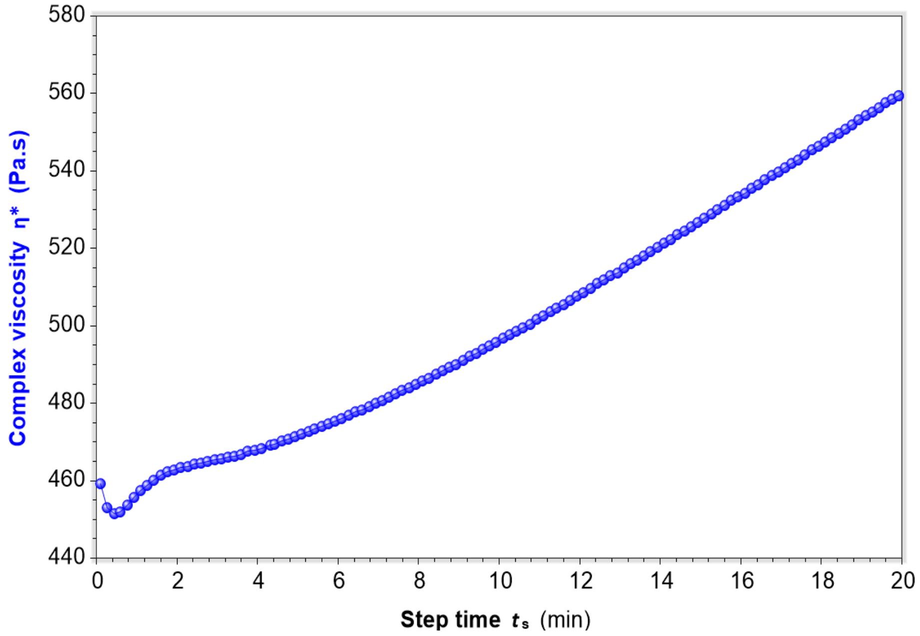
Using Time Sweep to Evaluate Polymer Melt Stability

Dynamic time sweep: Determine if melt properties are changing over time
- Decrease in viscosity → Degradation (chain scission, MW decreases)
- Increase in viscosity → Crosslinking (MW increases)

PP at 230°C (degradation)



PET at 280°C (crosslinking)



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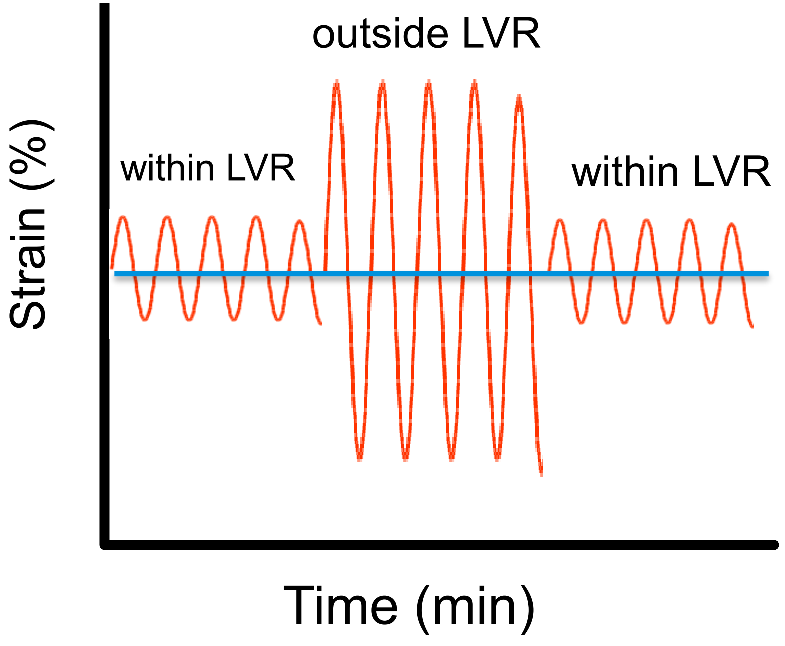
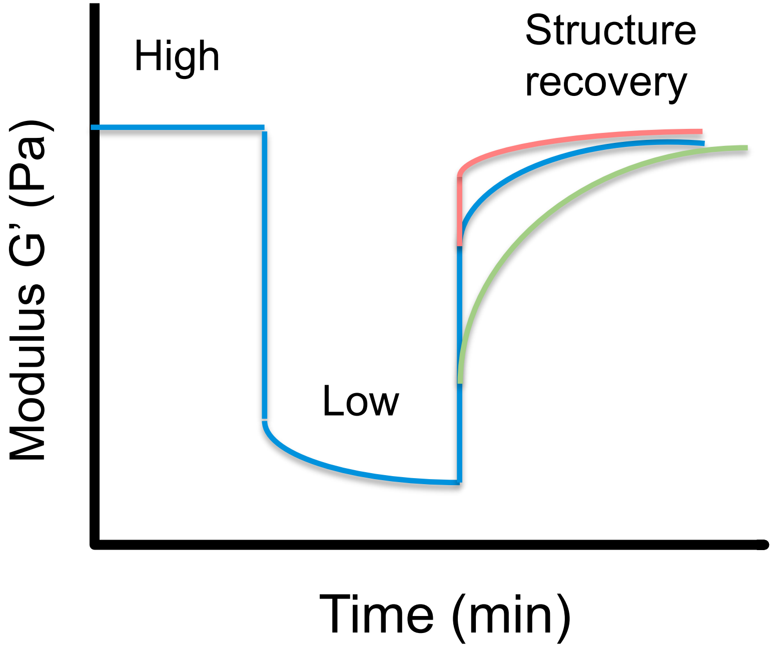
Stepped Time Sweep Method for Thixotropy Measurement





Experimental:

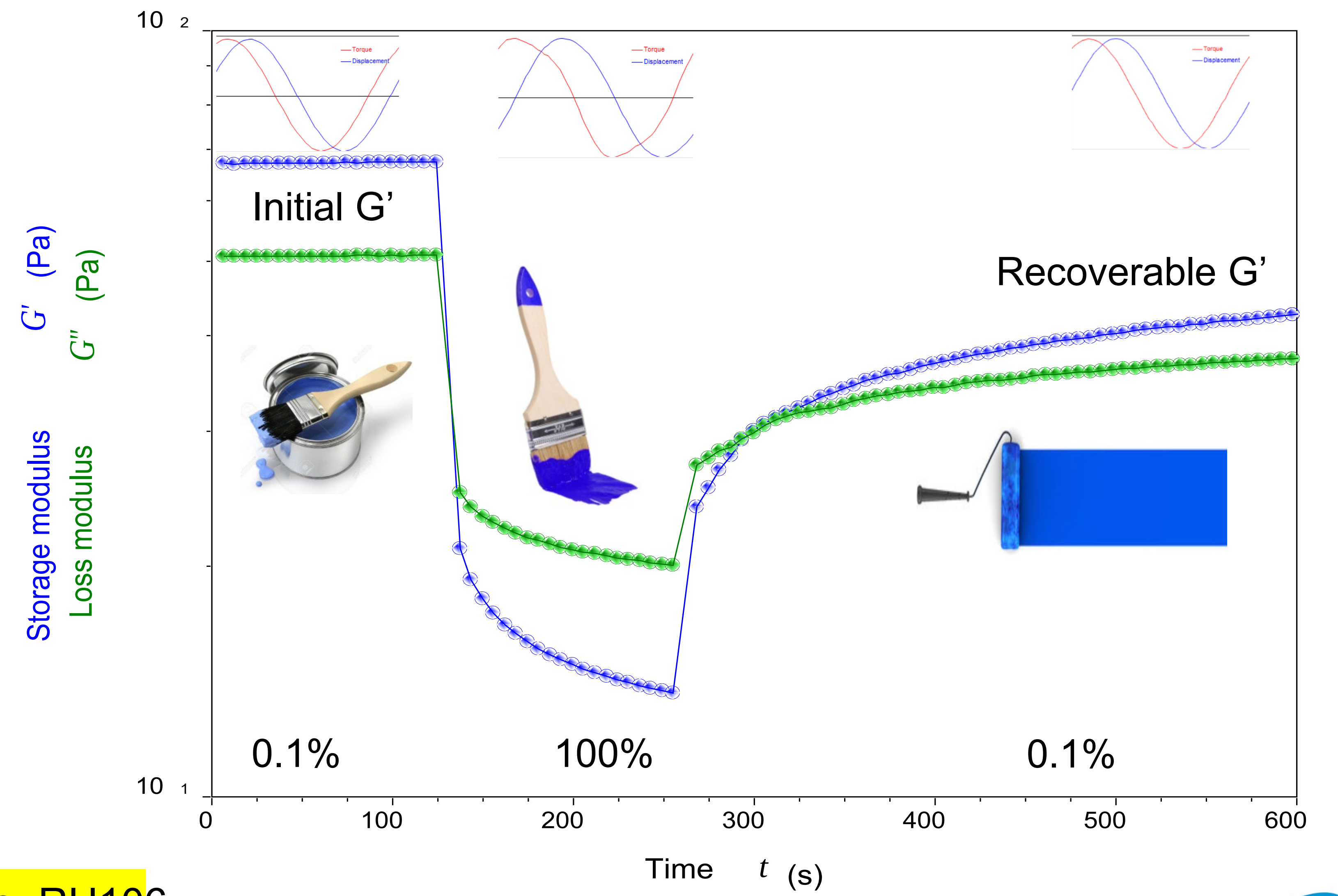
Step 1: Dynamic time sweep within LVR, structure at rest

Step 2: Dynamic time sweep outside LVR, structural destruction

Step 3: Dynamic time sweep within LVR, structural regeneration

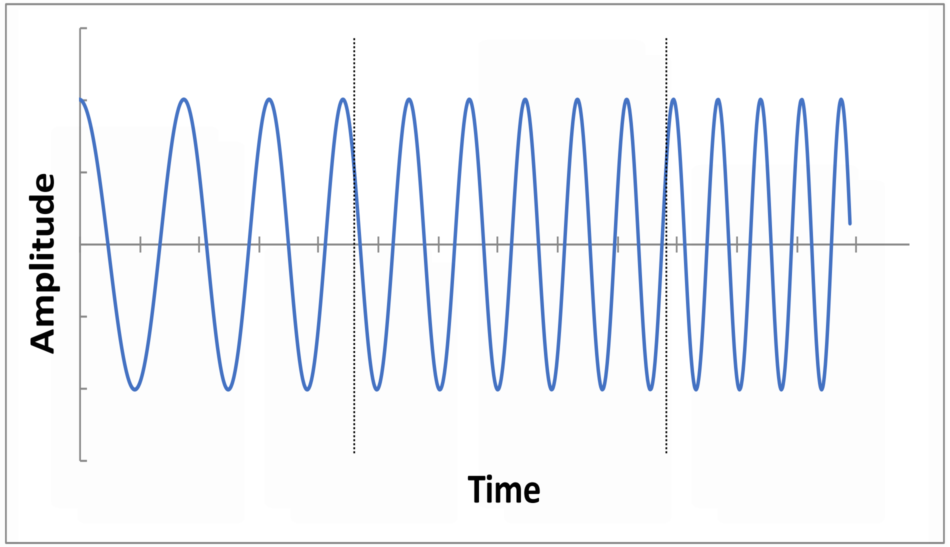
Good for measuring high viscosity samples

Blue Paint: Stepped Time Sweep

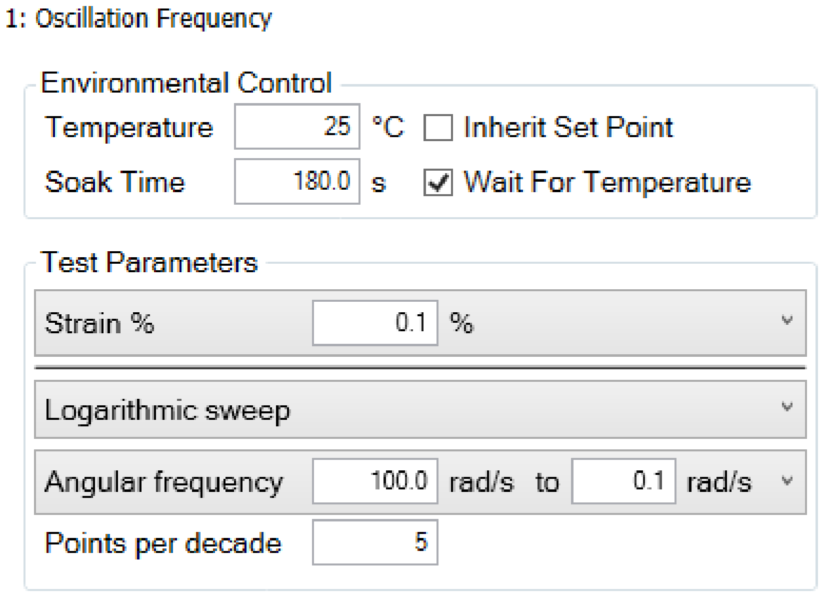


Chen apps note: RH106

Frequency Sweep



- The material response to increasing frequency (rate of deformation) is monitored at a constant amplitude (strain or stress) and temperature.
- In TRIOS: Frequency

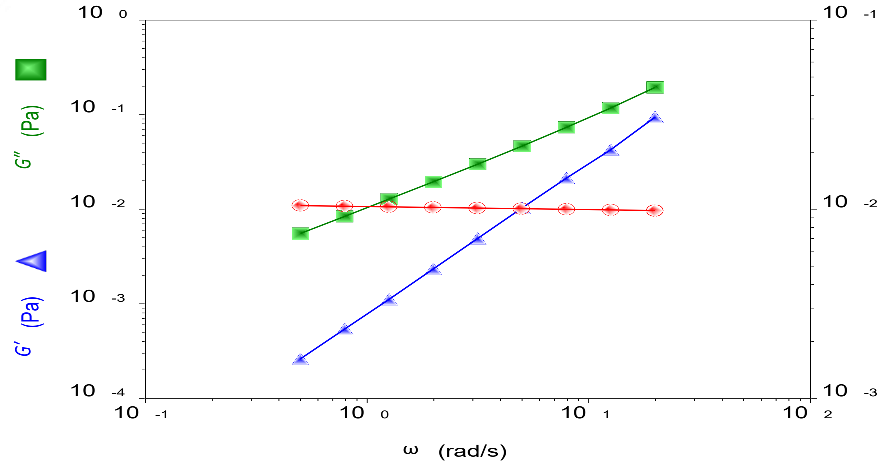


<u>USES</u>

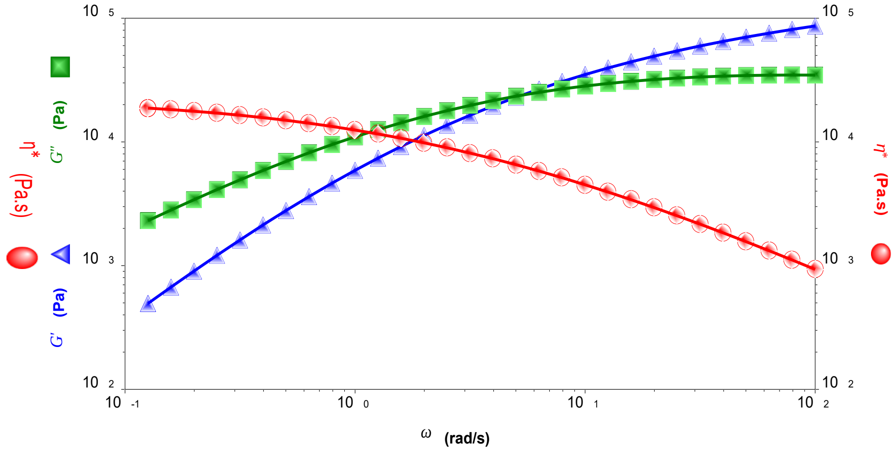
- Measure polymer relaxation
- Measure polymer Mw/ MWD
- Scouting differences of viscoelastic properties between formulations

Differences in Viscoelasticity using Frequency Sweep

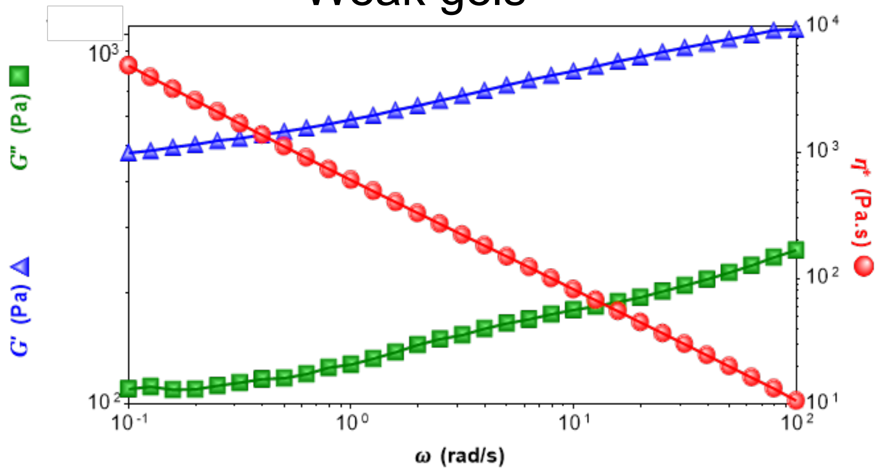
Low viscosity liquids



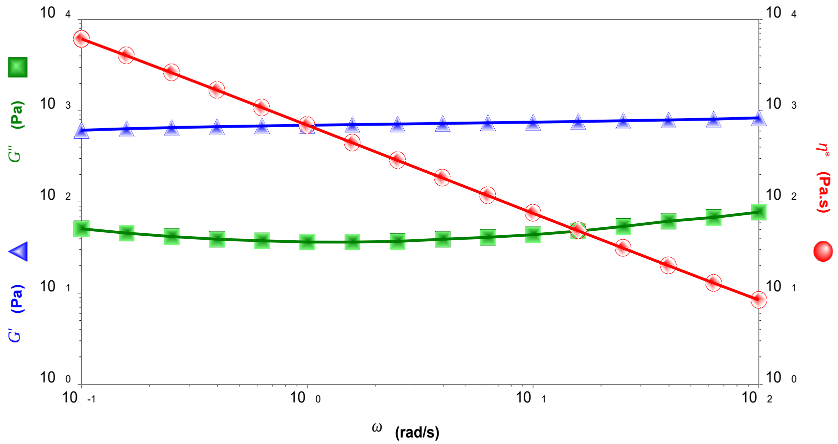
Viscoelastic materials

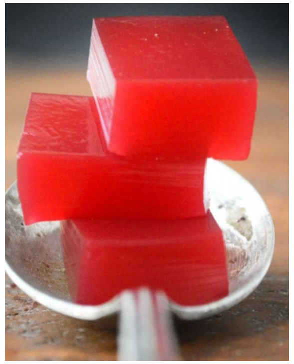


Weak gels



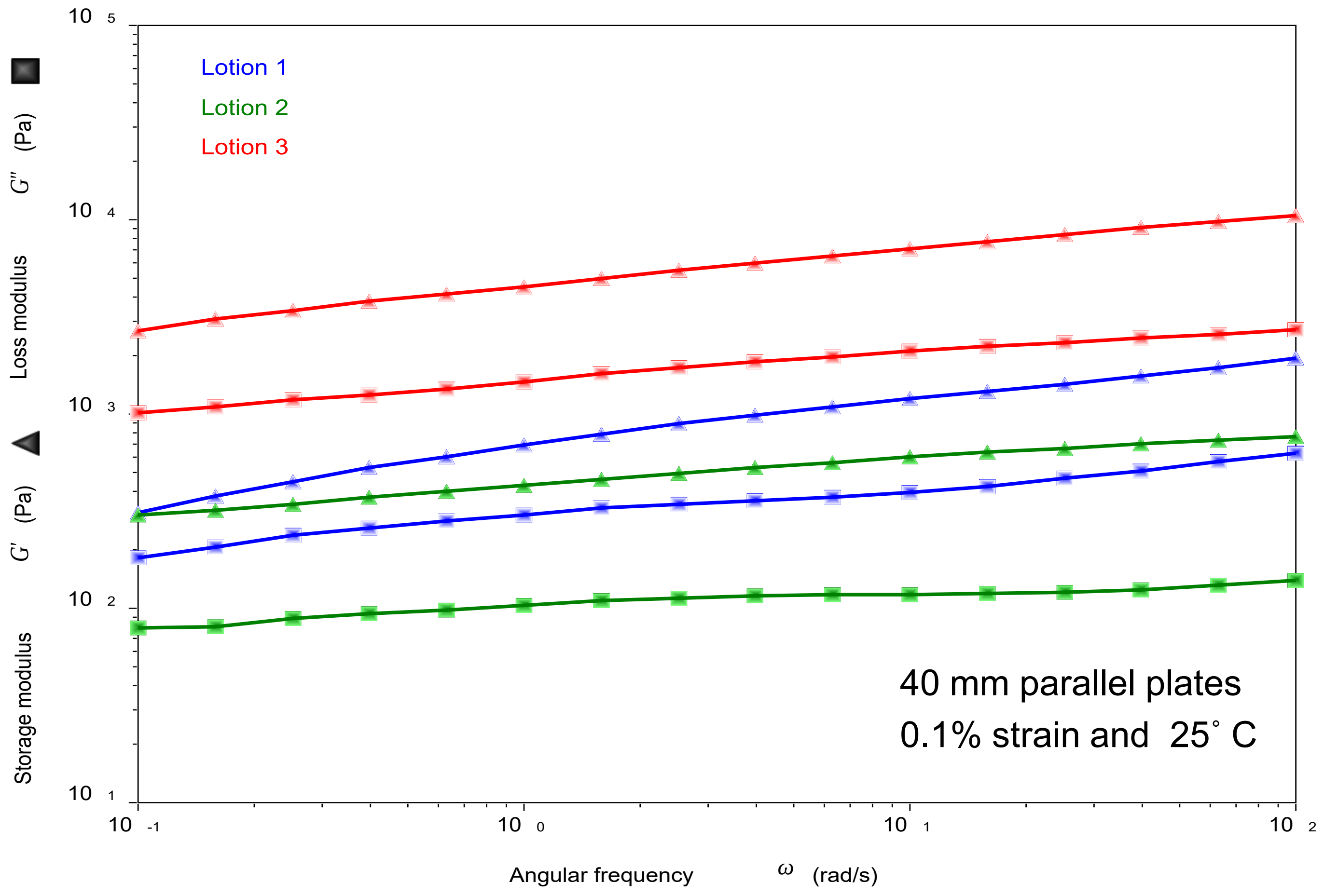
Elastic solids





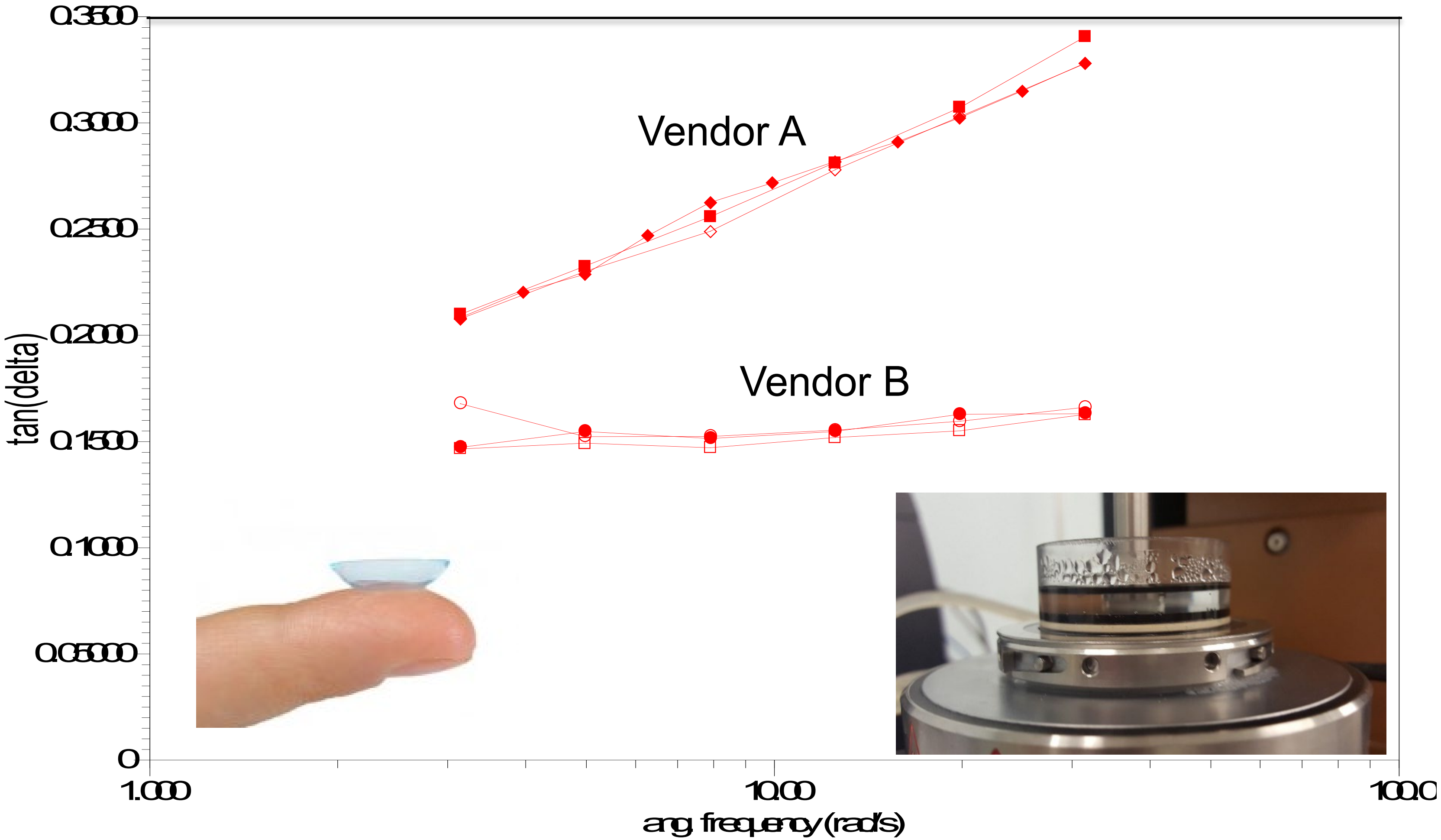
Frequency Sweep – Lotions

- Quantitative comparison on different formulations



Contact Lens Visco-elasticity

- Compare frequency dependency of contact lens' elasticity

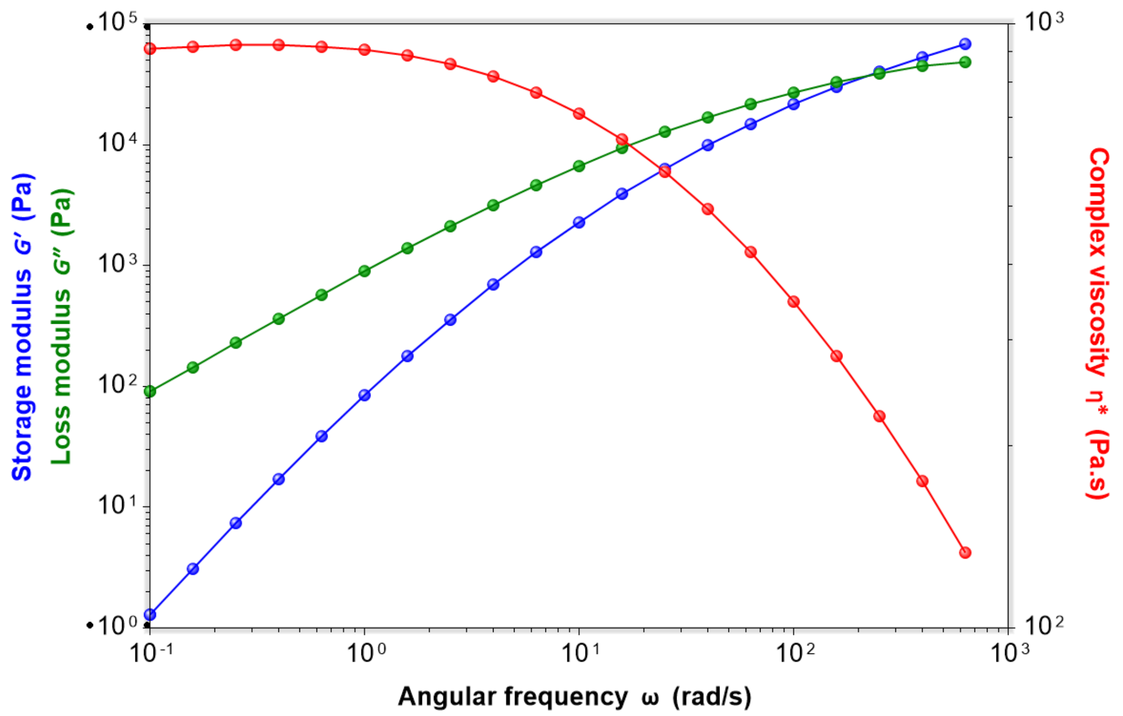


T Chen, TA apps note RH124

Melt Rheology Testing – ASTM D4440

- ASTM 4440 – “Dynamic Mechanical Properties, Melt Rheology”
- Dynamic frequency sweep at the molten temperature
- Sample moduli (G’, G” and G*) and complex viscosity is measured isothermally as a function of frequency (shear rate)
- The measured complex viscosity vs. frequency can be converted to shear viscosity vs. shear rates using the Cox-Merz Rule

PP frequency sweep at 200°C



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Influence of MW/MWD on G'/G" Crossover Point

The shape of G',G" curves in frequency sweep tests are sensitive to molecular chain entanglement

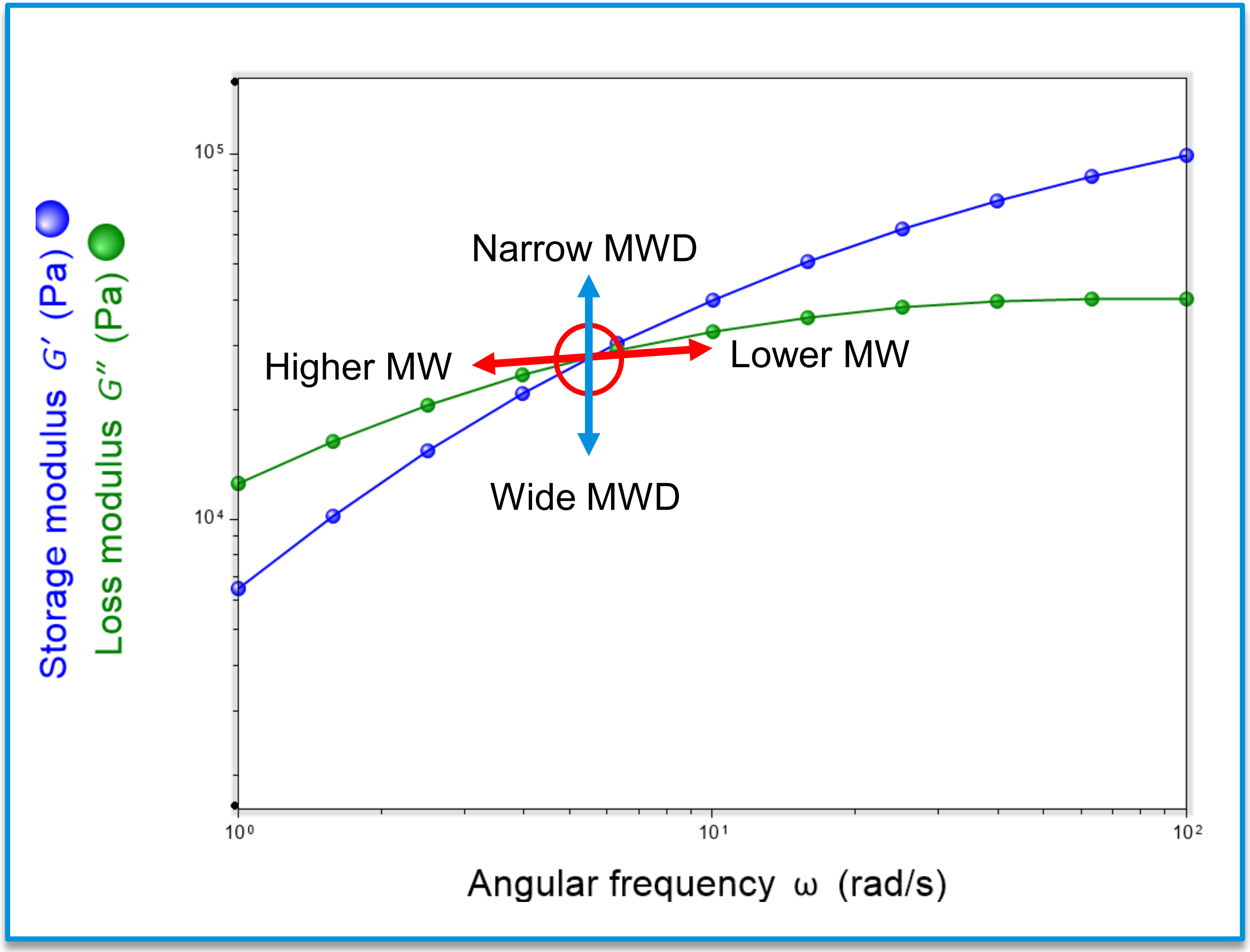
The G crossover point → sensitive indicator for probing molecular architecture (MW, MWD)

– Higher G crossover frequency
→ **lower MW**

– Lower G crossover Modulus
→ **Wider MWD**

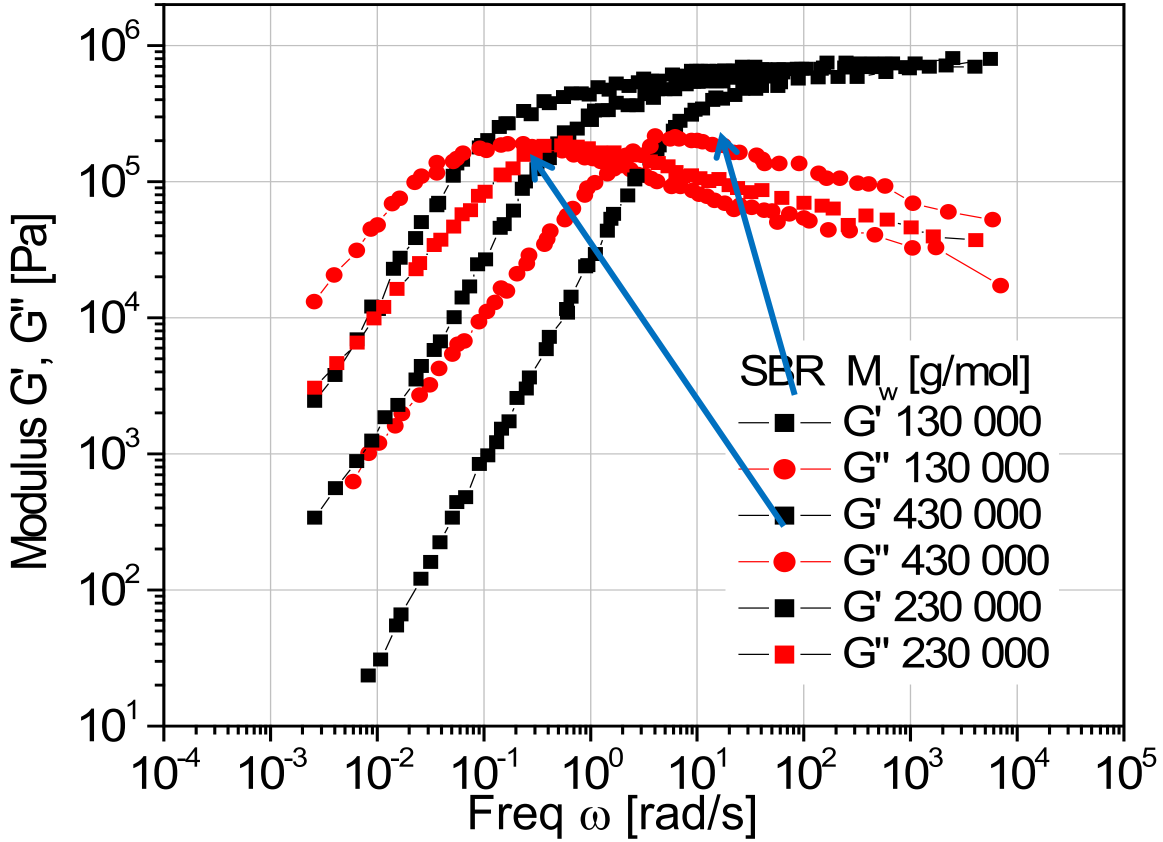
– Quickly screen and compare differences between polymer batches

TA apps note: AAN013



Influence of MW on G‘ and G“

- The G‘ and G“ curves are shifted to lower frequency with increasing molecular weight.



TA Instruments Webinar

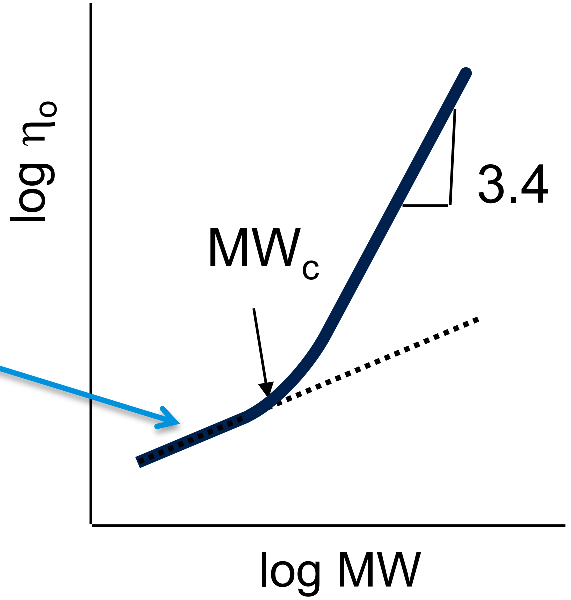
https://www.tainstruments.com/analyzing-molecular-weight-distribution-w-rheology/

Professor Chris Macosko – Analyzing Molecular Weight Distribution w/ Rheology

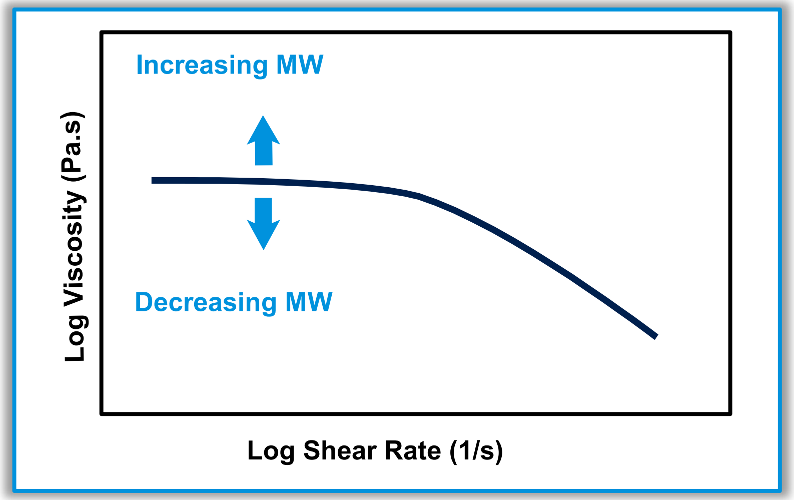
Zero Shear Viscosity (η_0)

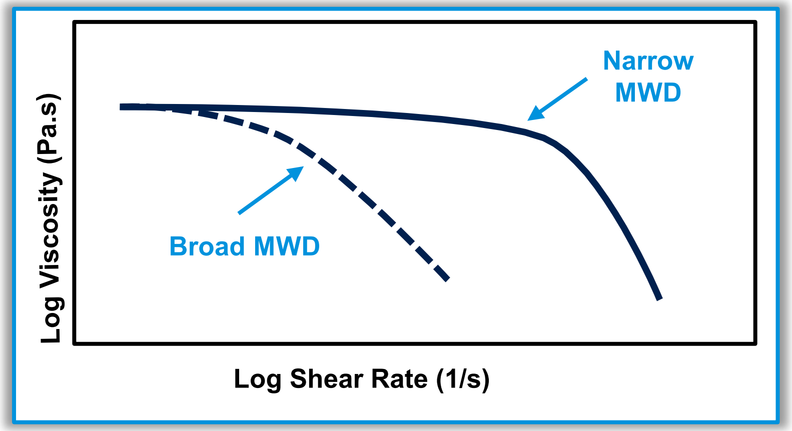
- **Low MW →** η_o is proportional to MW
- **MW > Critical MWc →** η_o is proportional to MW^ 3.4 (i.e. $\eta_0 \propto M_w^{3.4}$)
  - Critical MWc: polymer chain is long enough to interact with itself → entanglement starts to happen
- **Higher Mw →** Higher η_o

$\eta_0 = K \cdot Mw$



$\eta_0 = K \cdot Mw^{3.4}$





Ref. Graessley, Physical Properties of Polymers, ACS, c 1984.

Fingerprinting Polymer Architecture

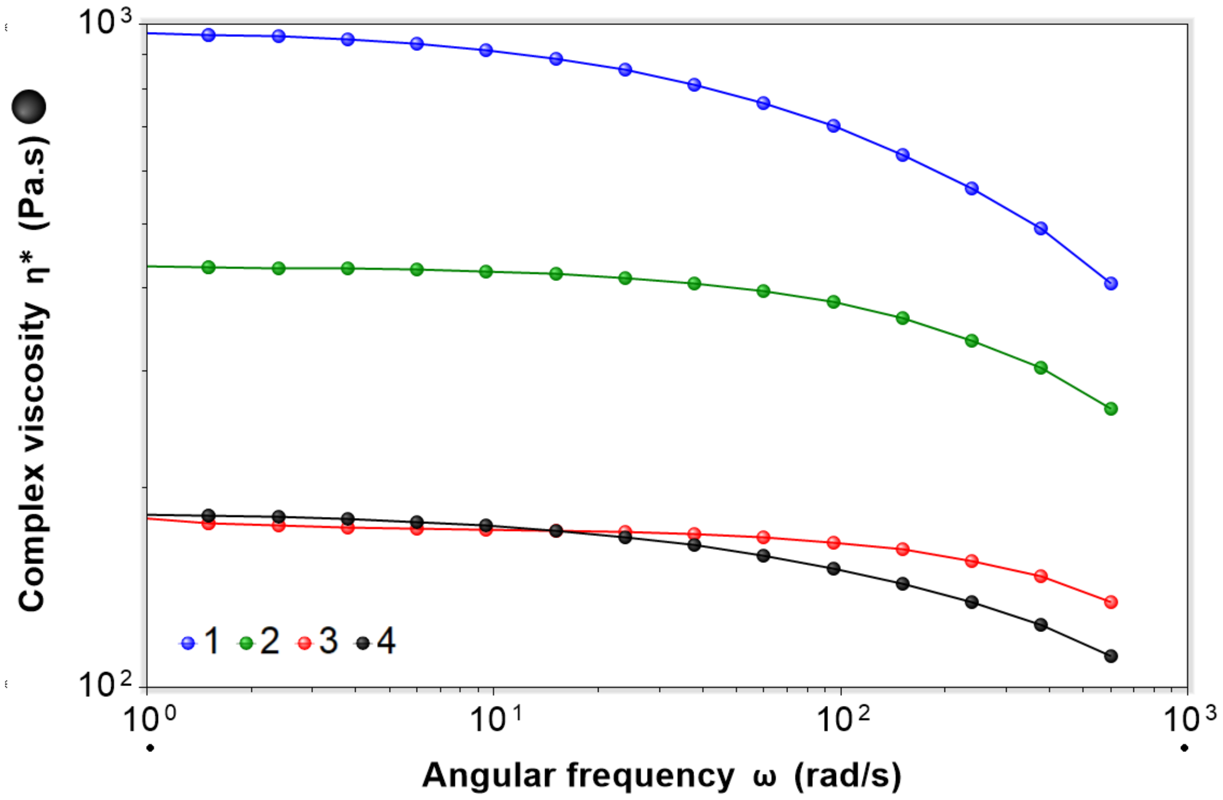
Recycled PET from different sources can largely differ with respect to:

- Molecular weight (MW)
- Molecular weight distribution(MWD)

- **Problem:** Feedstock processing could cause significant MW reduction
- **Solution:** Viscosity measurement provides comparison to the MW and MWD information

	Zero Shear Viscosity (Pa.s)	Molecular Weight	MWD
Sample 1	983		
Sample 2	430		
Sample 3	176		
Sample 4	186		

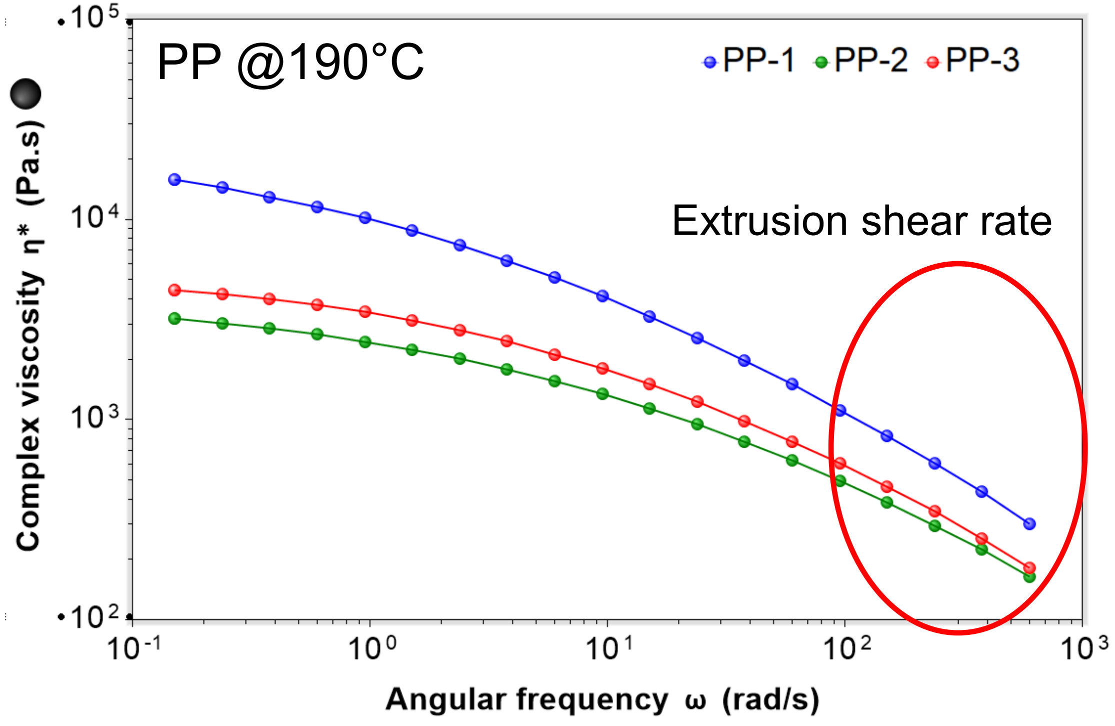
PET @ 280°C



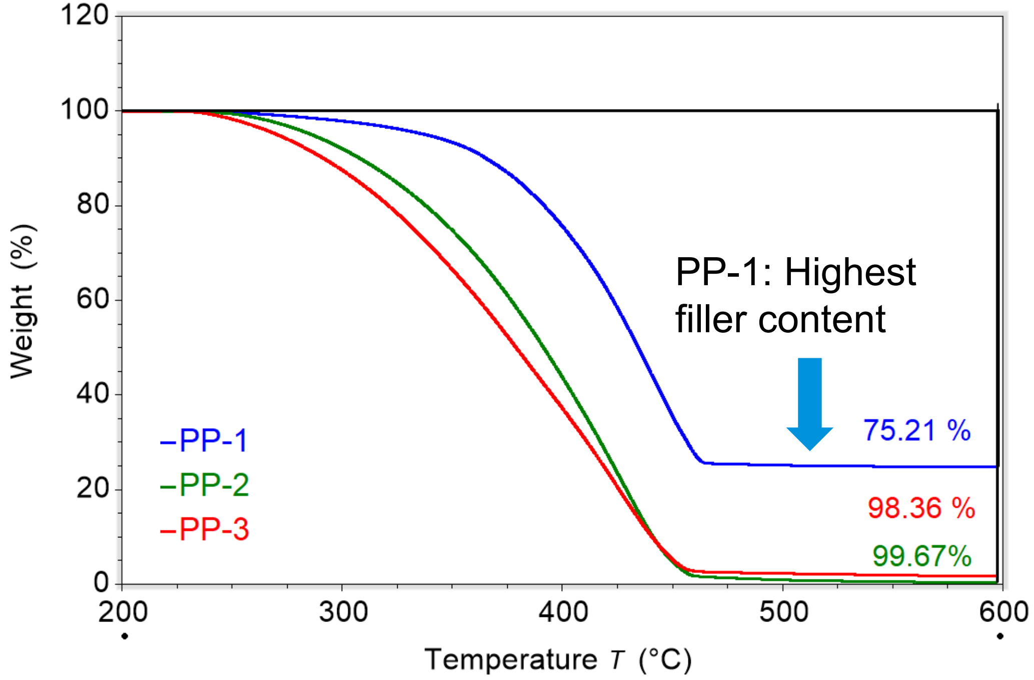
T Chen, TA apps note RH122

Guide Processing of Recycled PP from Different Sources

- Rheology: Directly measure melt viscosity at processing temperature
- TGA: Supporting information (impurity/ filler content)

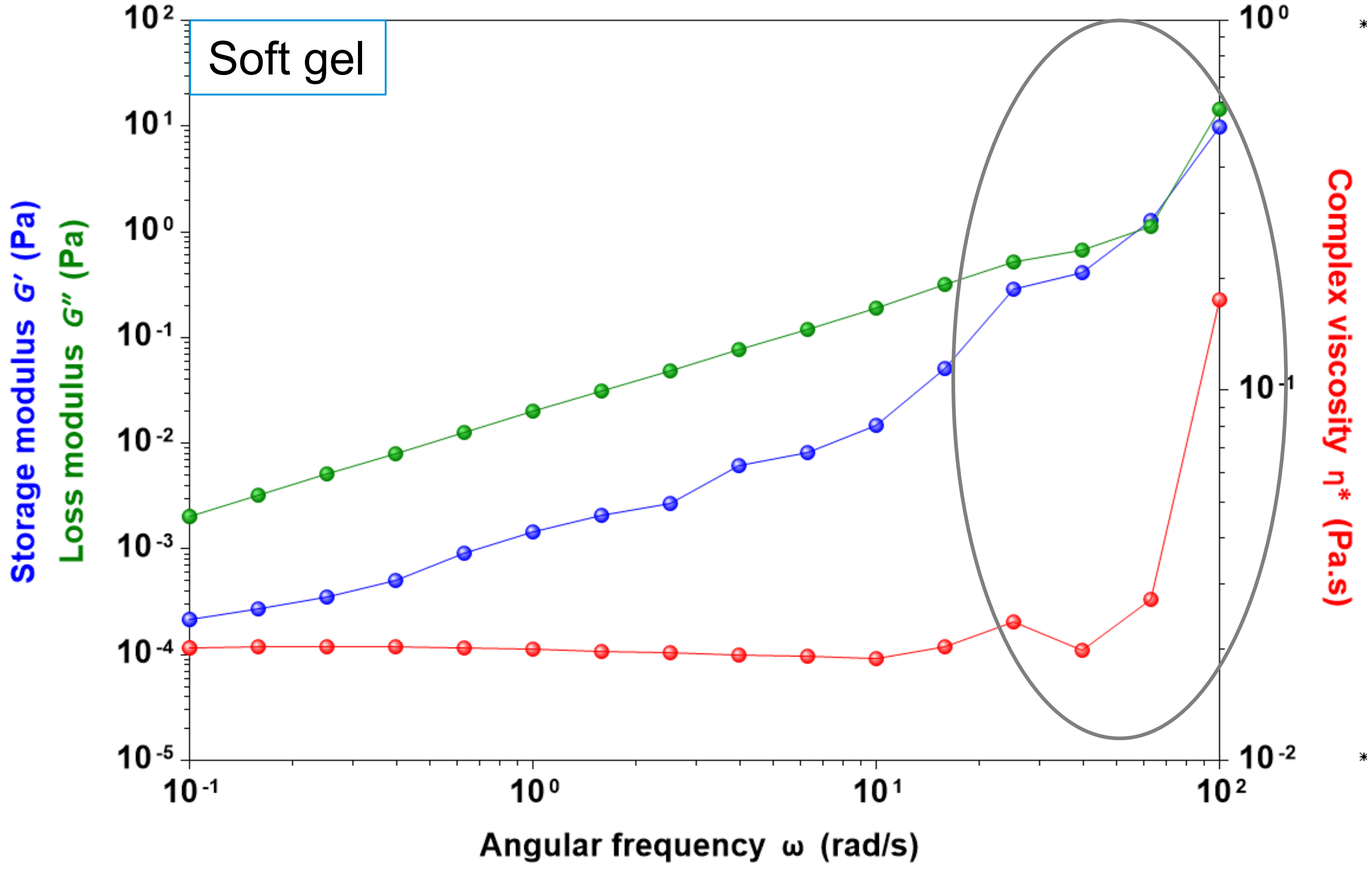


TGA analysis at 10°C/min



Frequency Sweep Test Challenges

- Why data look so bad at high frequencies?

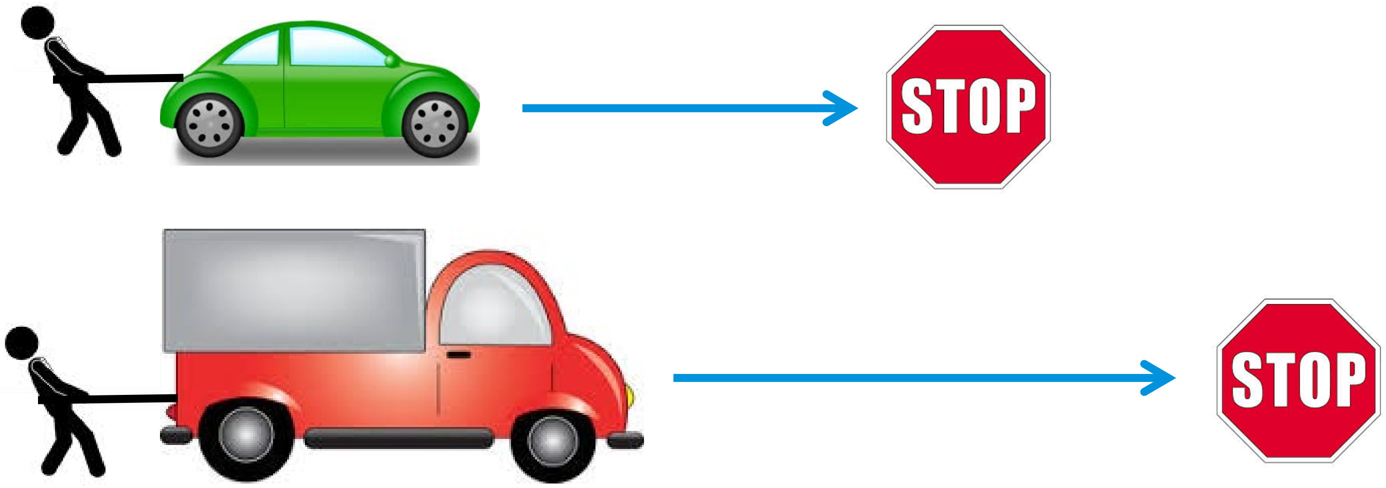


System Inertia

- <u>What is Inertia?</u>:

Inertia is a property of matter, which manifests itself as a resistance to any change in momentum of a body

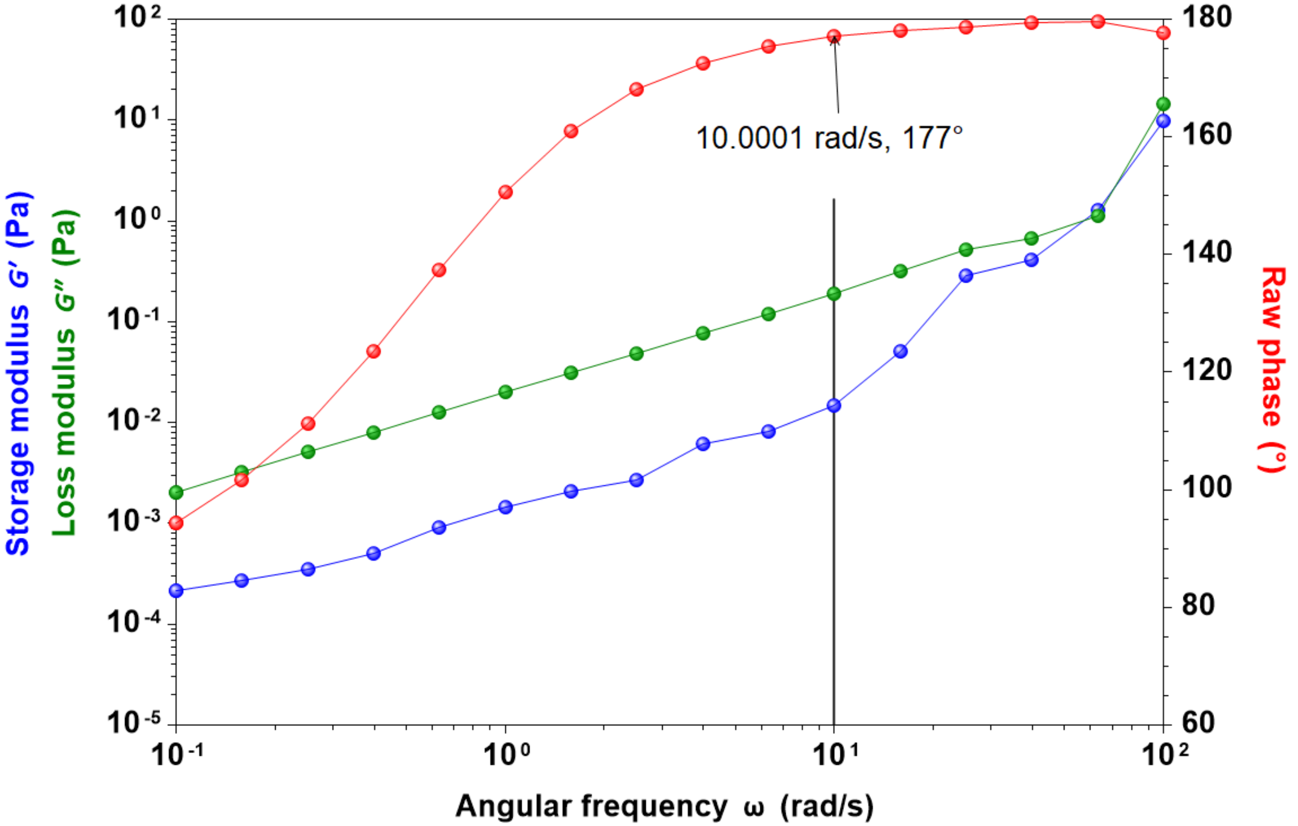
  - Instrument has inertia (motor + geometry)
  - Sample has inertia



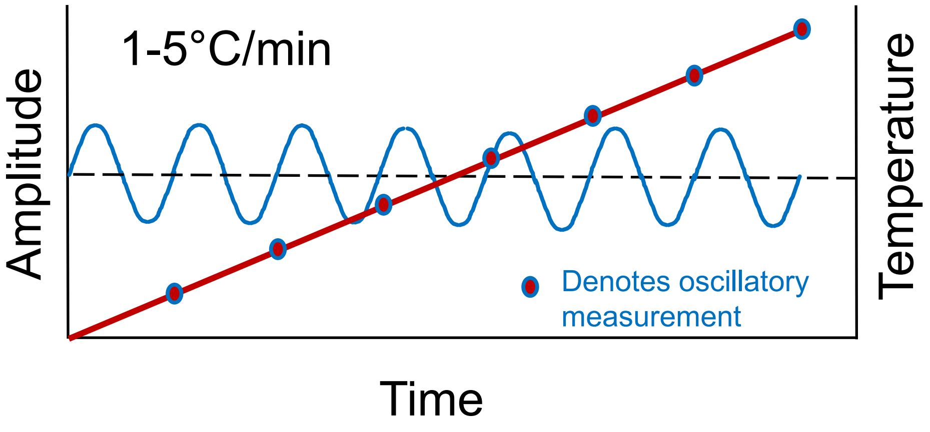
Note: ARES G2 has separator motor and transducer design, so motor inertia has no impact to the measurement results!

Understand and Avoid Inertia Influence

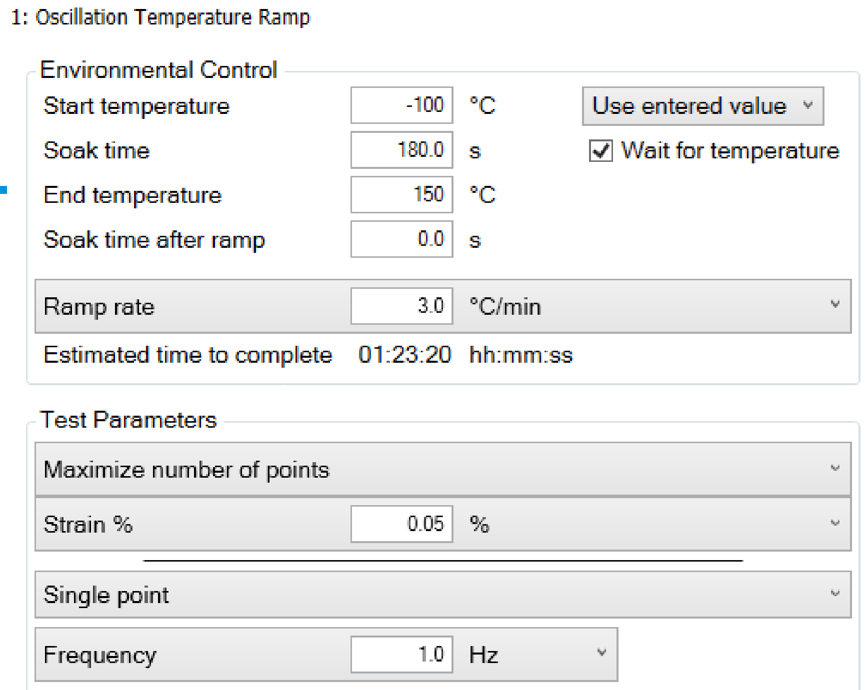
- How do we know when inertia is dominating my results?
  - In general, consider delete data when “Raw Phase” is greater than 170°
- How to reduce inertia influence?
  - Use a lighter geometry (e.g., Al)
  - Increase sample stiffness (e.g., reduce gap)



Dynamic Temperature Ramp



- Linear heating rate is applied, and the material response is monitored at a constant frequency and constant amplitude
- In TRIOS: Temp Ramp

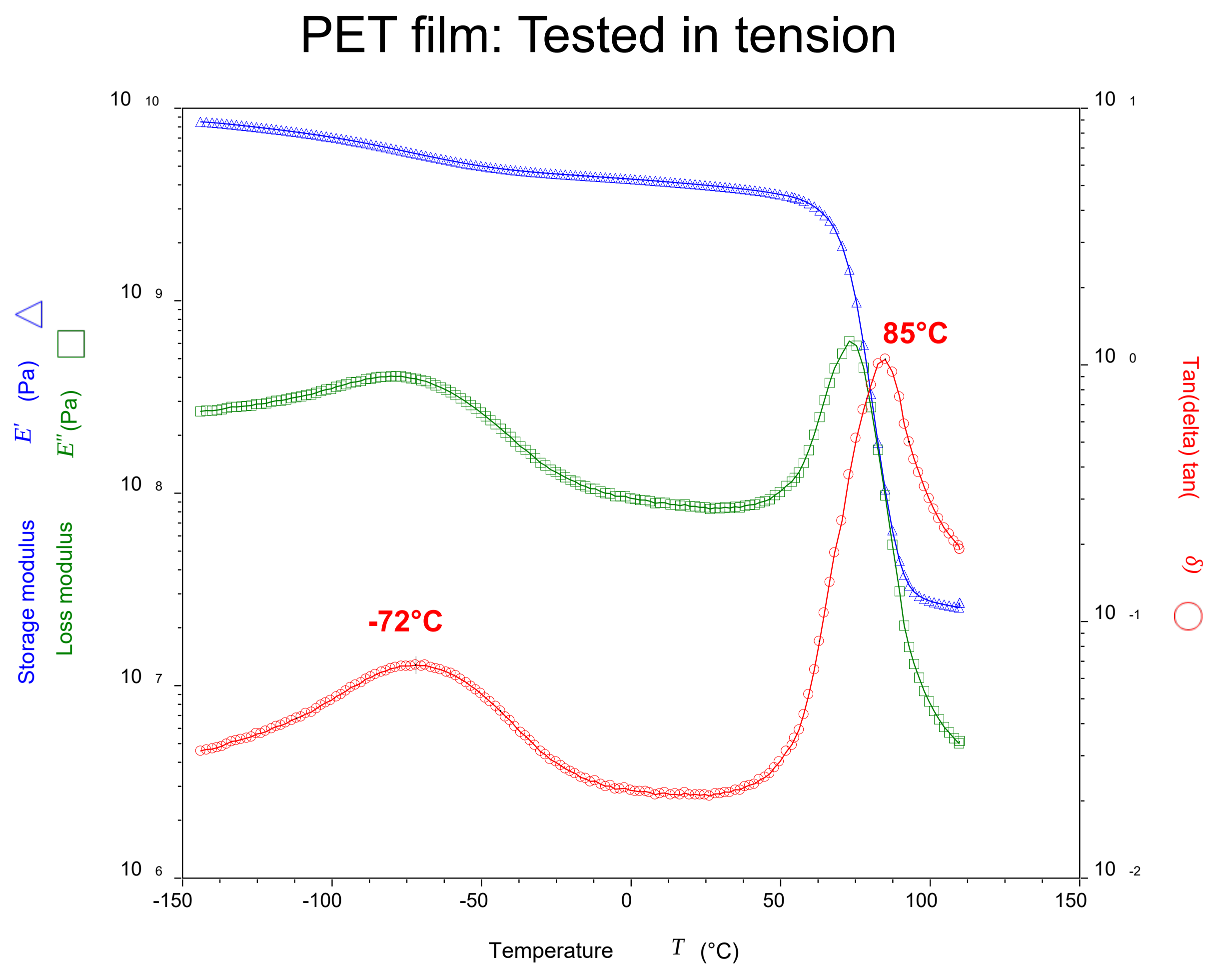


USES

- Measure material's viscoelastic properties vs. temperature
- Measure glass transition and sub-ambient transition temperatures

Rheometers and DMAs are more sensitive to weak amorphous transitions than DSC

PET film: Tested in tension



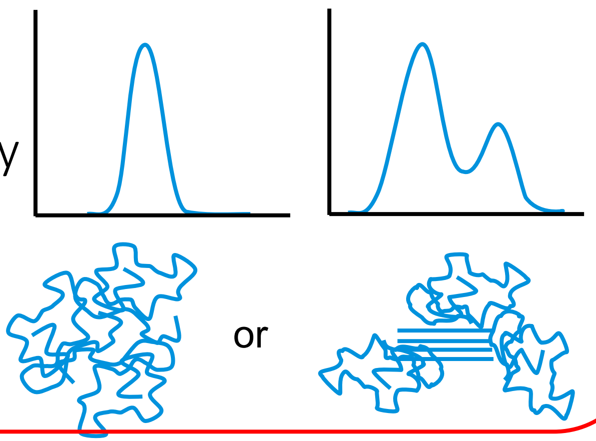
Glass Transition (T_g): Cooperative motion among a **large number** of chain segments

Secondary Transitions (T_β, T_γ): Local or side group motion

Rheology/DMA is 100-1000× more sensitive than DSC for identifying weak amorphous transitions

Monitor Tg using DMA to study:

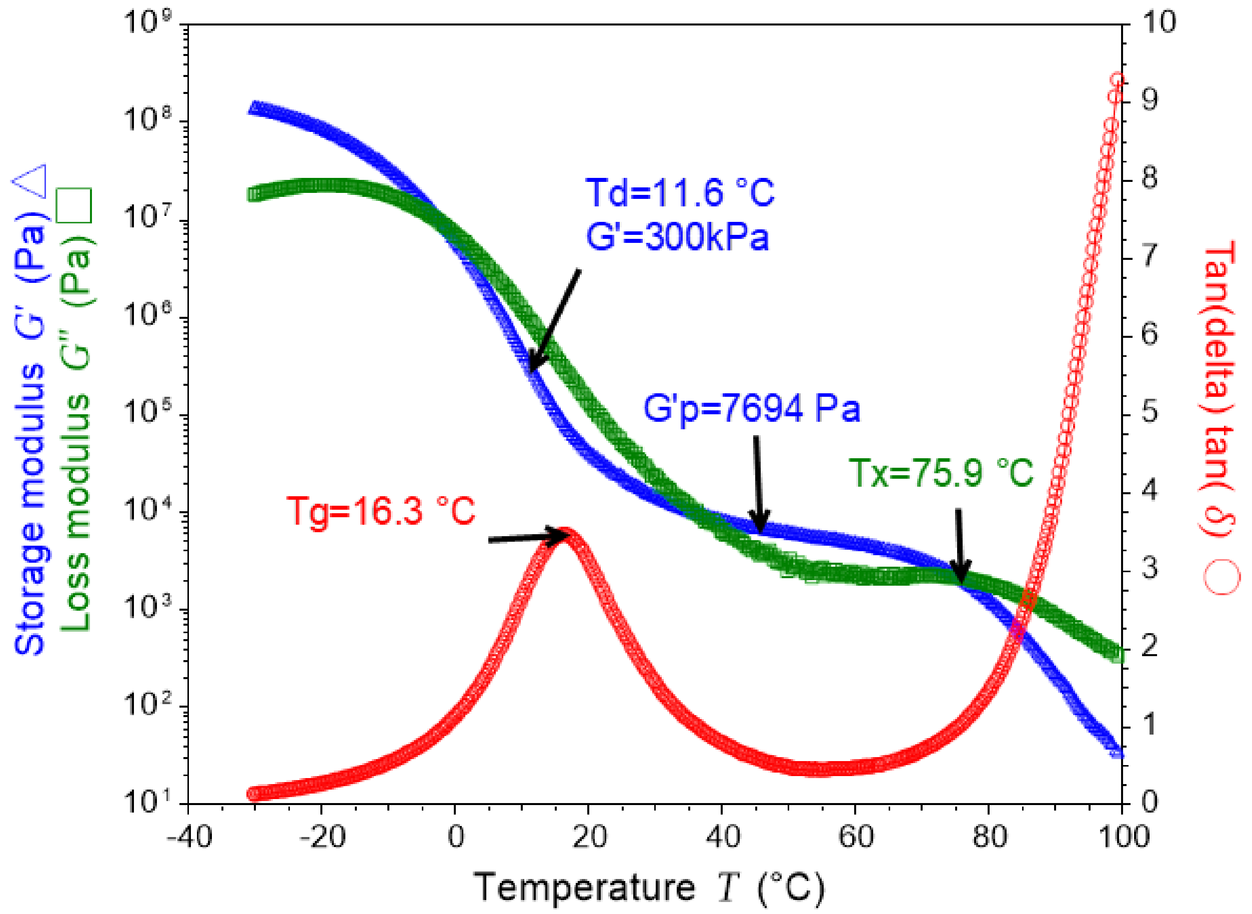
- Blend Miscibility
- Crystallinity
- Crosslinking density



https://www.tainstruments.com/recorded-theory-applications-training/

What do We Learn from a Temp Ramp Test?

- Most popular test for PSA evaluations
- Results correlate to the PSA performance with temperature



Applications temperature window – Between Tg (or Td) and Tx

Tg: Glass transition
Td: Dahlquist temperature
Tx: G crossover temperature

Cohesion strength:
Gp: Plateau modulus

https://www.tainstruments.com/strategies-for-rheological-evaluation-of-adhesives/

How to Design a Good Temp Ramp Test?

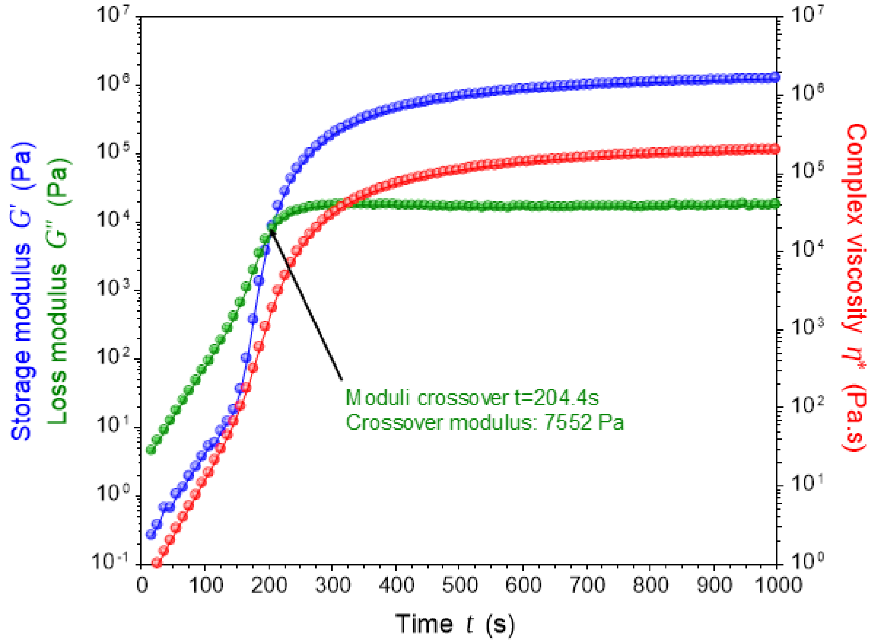
- Sample loading and trimming
  - High temp (easy to trim)
- Geometry
  - 8mm or 25mm parallel plate
- Temperature range
  - From below Tg into the molten state
- Heating or cooling rate
  - 2-5 degrees/min
- Frequency
  - Commonly at 1Hz or 10 rad/s
- Oscillation strain %
  - Within the linear viscoelastic region (LVR) of the sample
- Axial force control
  - Ensure good contact and no slipping
  - Compensate for geometry and sample thermal expansion or shrinkage

https://www.tainstruments.com/strategies-for-rheological-evaluation-of-adhesives/

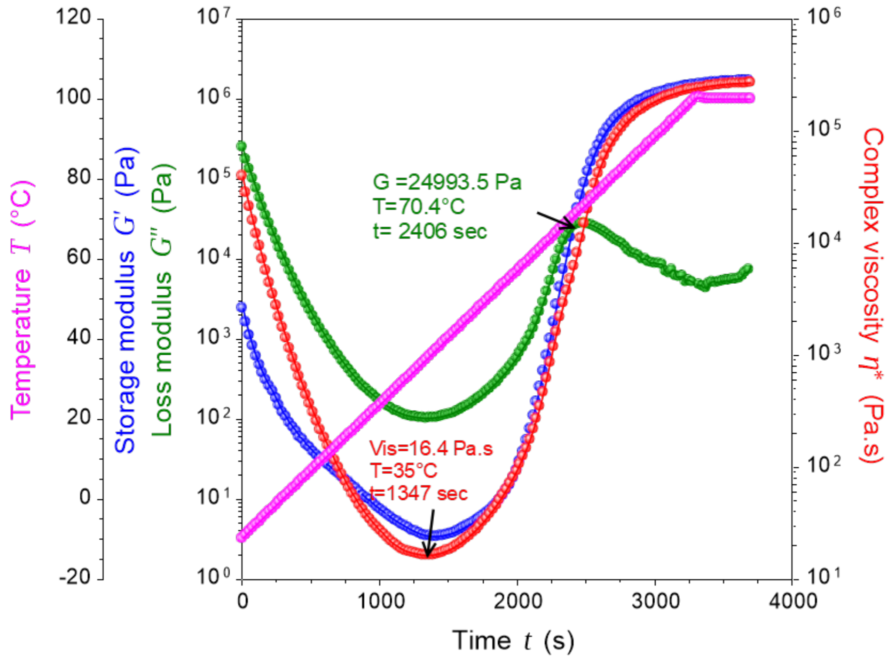
Rheology for Thermoset Characterization

- Measure viscosity change before crosslinking
- Monitor gelation and measure the gel point
- Monitor sample viscoelastic property change (G’ and G”) during curing
- Evaluate the mechanical properties of the end-use product

Isothermal Curing



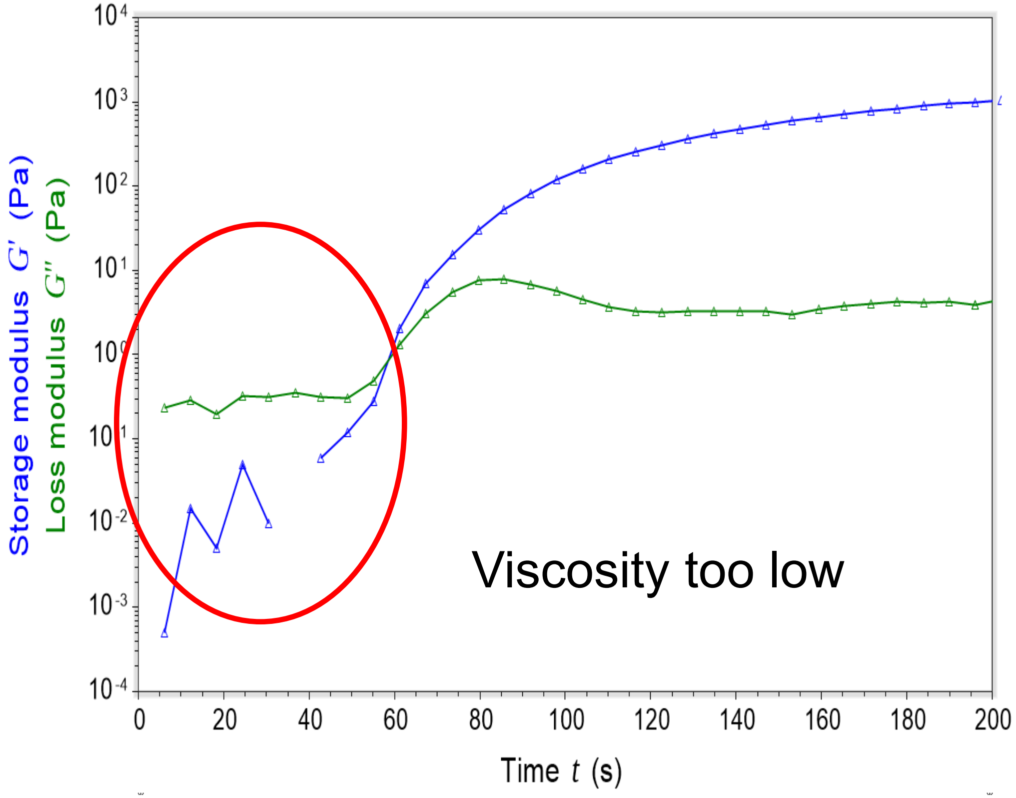
Temperature Ramp Curing

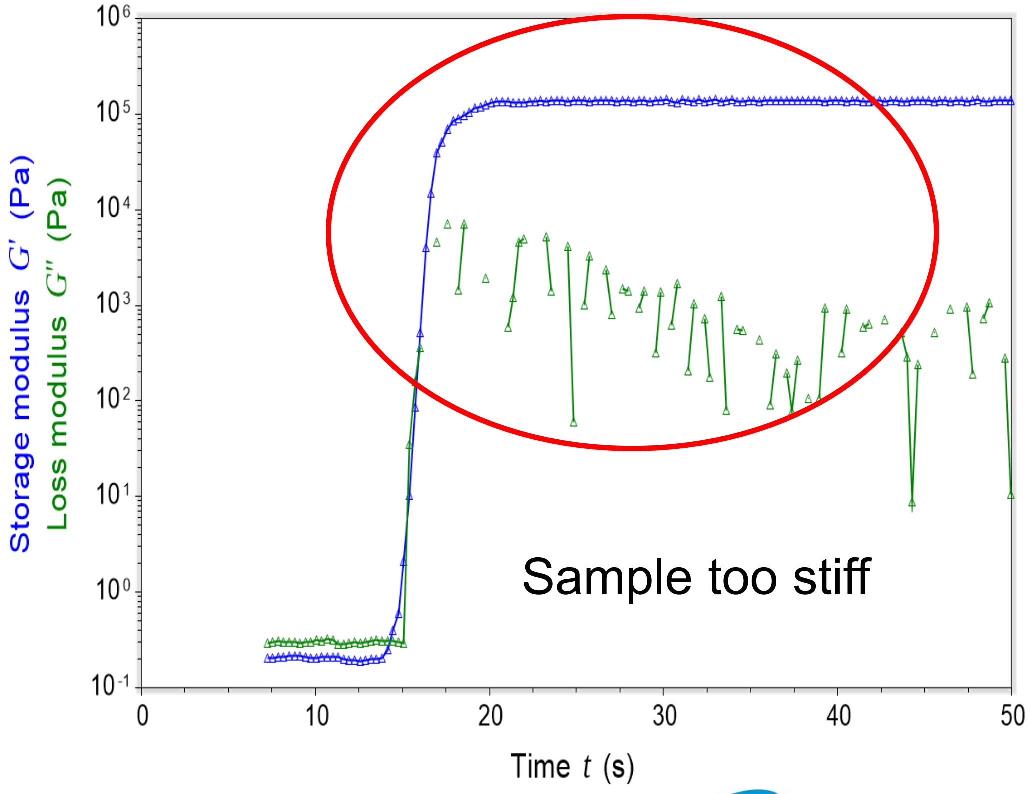


https://www.tainstruments.com/thermal-rheological-and-mechanical-characterizations-of-thermosets/

Challenge in Experimental Designs

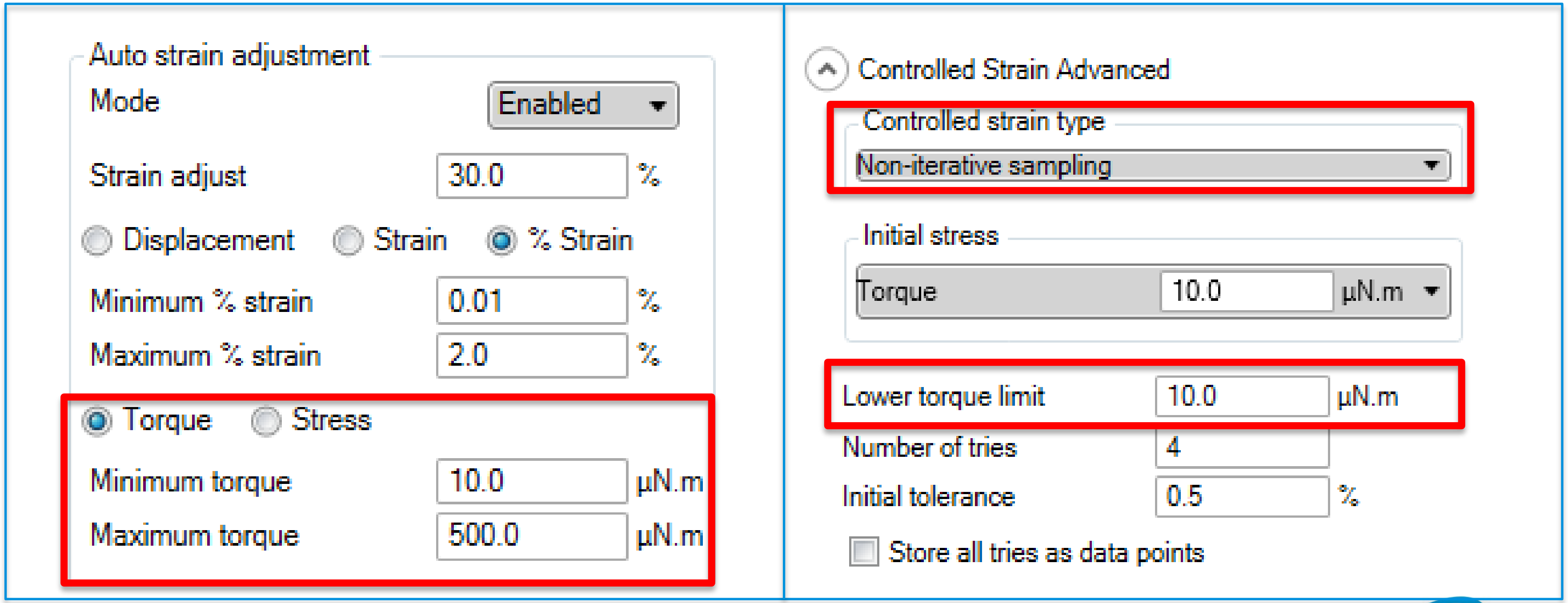
- Significant change in viscoelastic properties (6-8 decades) through curing
- Appropriate geometry size (8mm, 25mm) and test gap (1-2mm)
- Challenge in setup appropriate test parameters





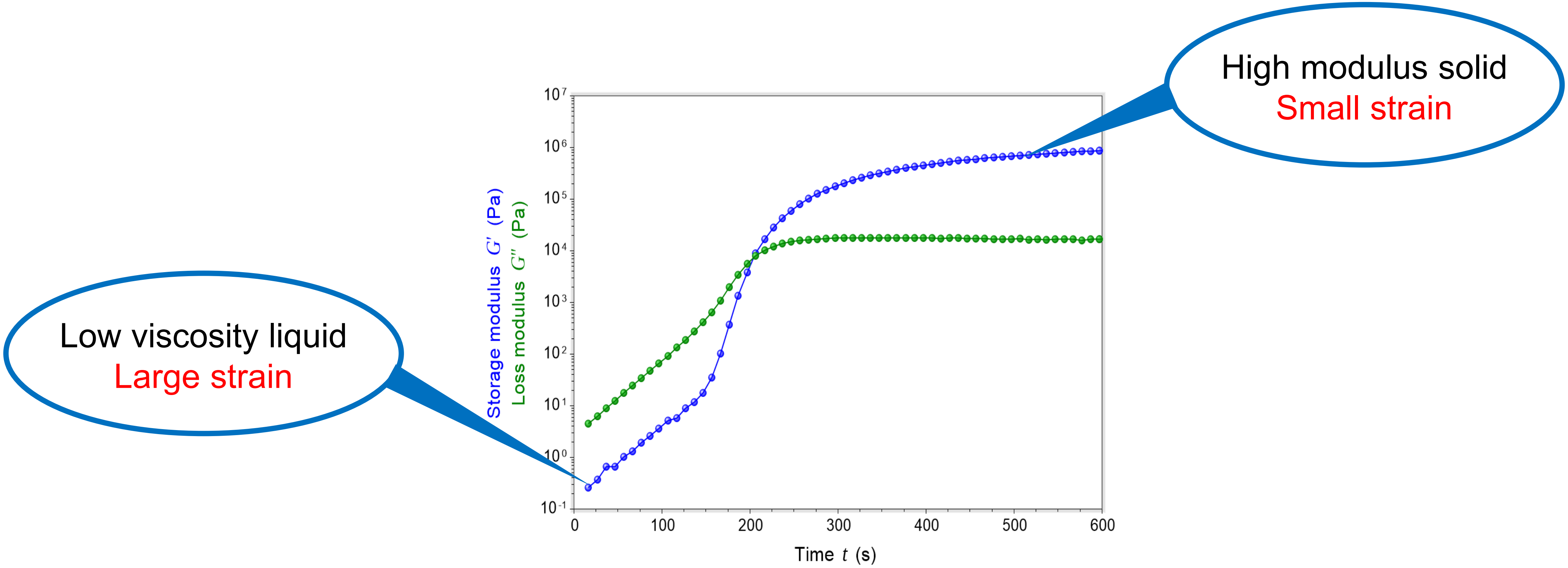
How to Adjust Strain During a Test?

- Setup a torque range in a procedure. During the test, when applied torque is outside of the torque window, strain will be adjusted. (Example: ARES G2 or DHR)
- Setup a minimum operating torque in a procedure. Use a reasonably small strain to start the test. When torque is below the minimum set value, strain will be increased. (Example: DHR or AR)



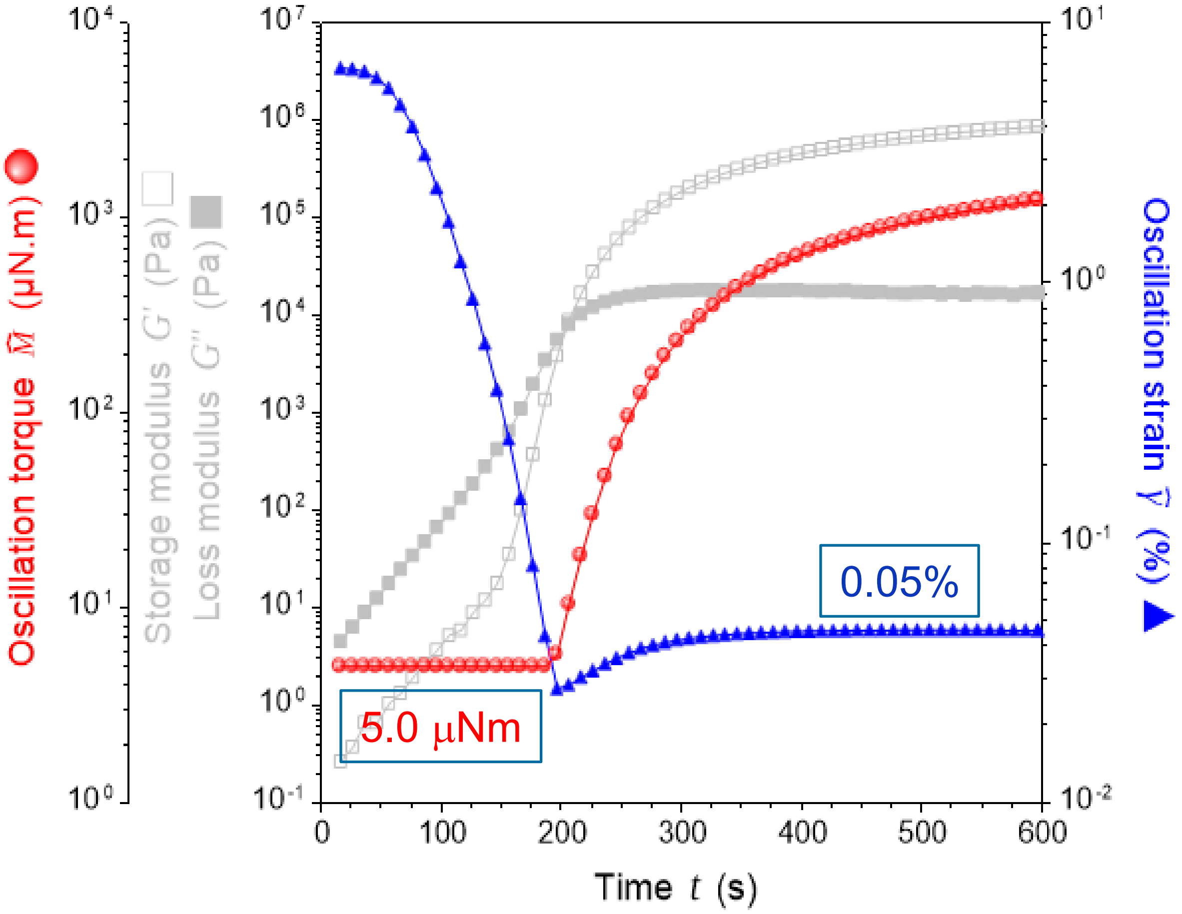
Approaches for Optimum Test Parameters

- The key to ensure a good measurement result
  - Change the measurement strain

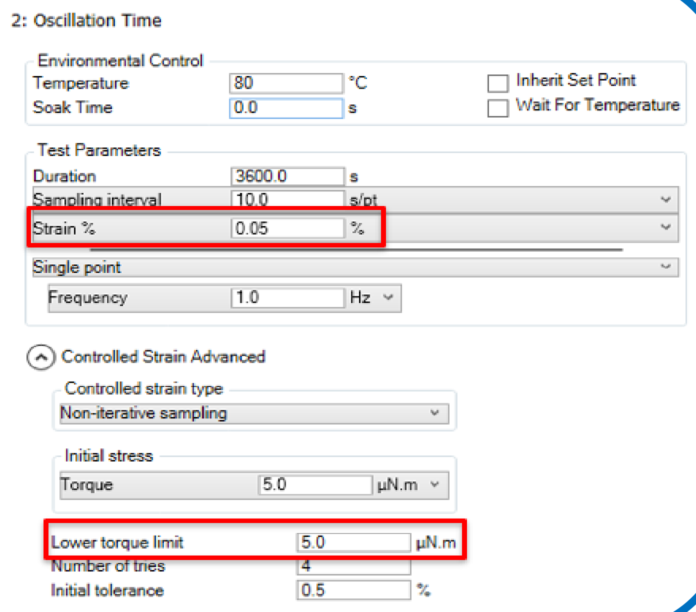


Strain Adjustment Method-1

- An Isothermal curing example



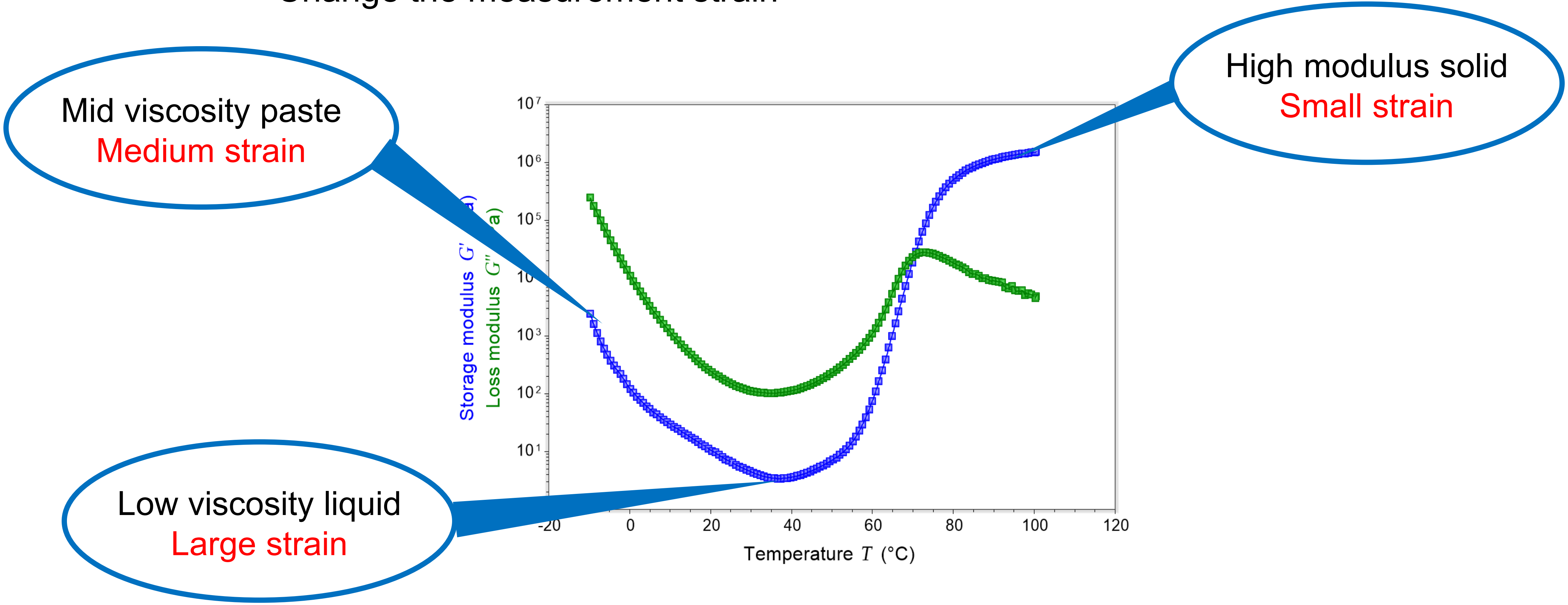
- Good for all controlled-stress rheometer (AR, DHR, HR)
- **Setup an instrument lower torque limit**
- When sample has low viscosity, auto-switch to use stress/torque control for the test.



T Chen, 2019 SAMPE TP19-1595

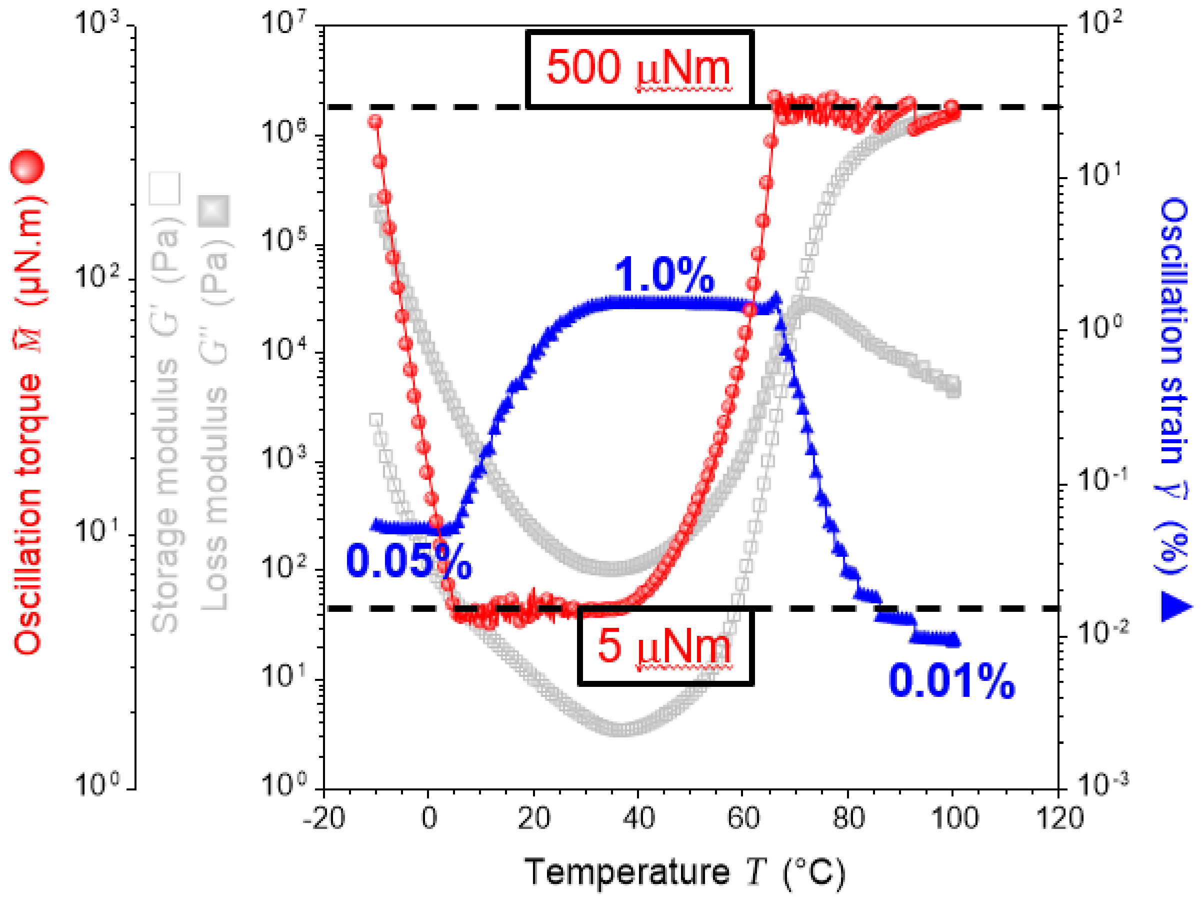
Approaches for Optimum Test Parameters

- The key to ensure a good measurement result
  - Change the measurement strain

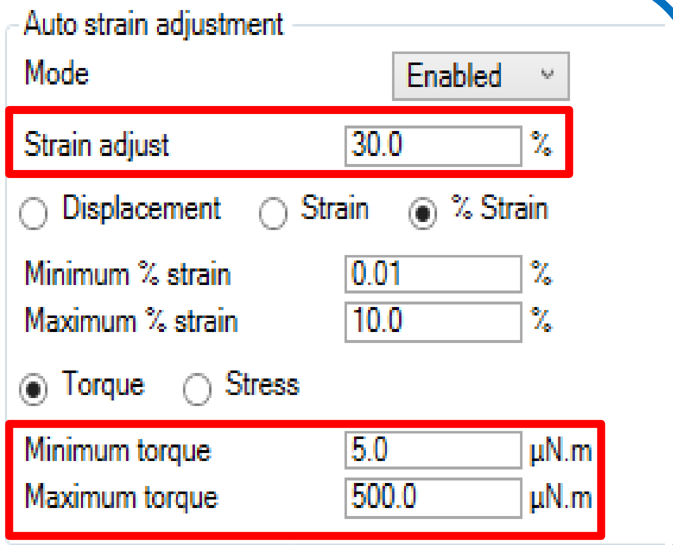


Strain Adjustment Method-2

- A temp ramp curing example



- Good for both controlled-stress and controlled-strain rheometers
- **Setup a smart torque window**
- When measurement torque goes outside of the window, strain will be adjusted



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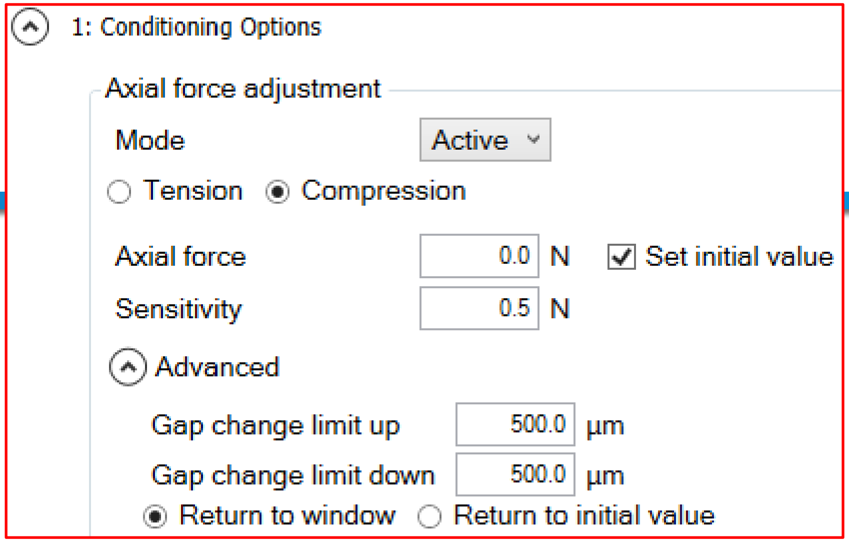
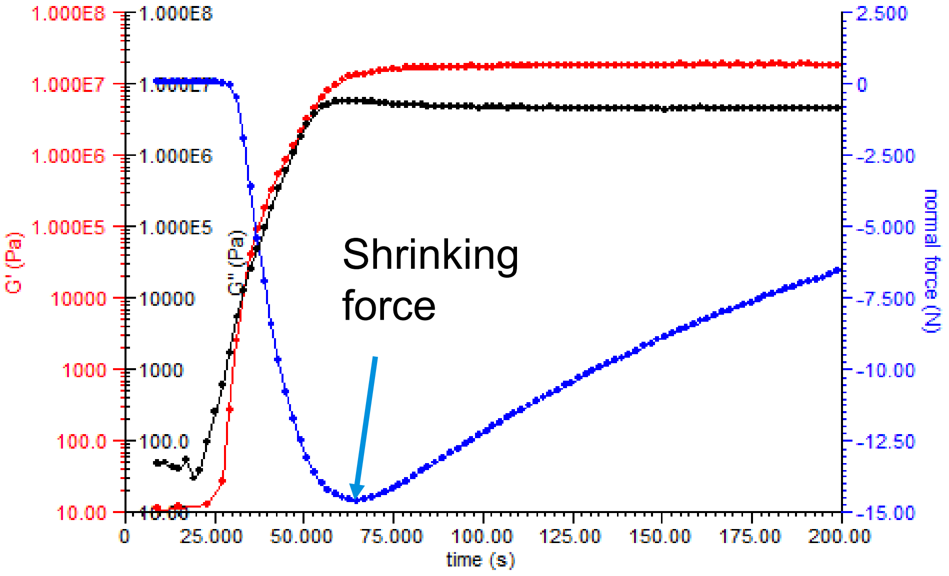
Monitoring Shrinkage

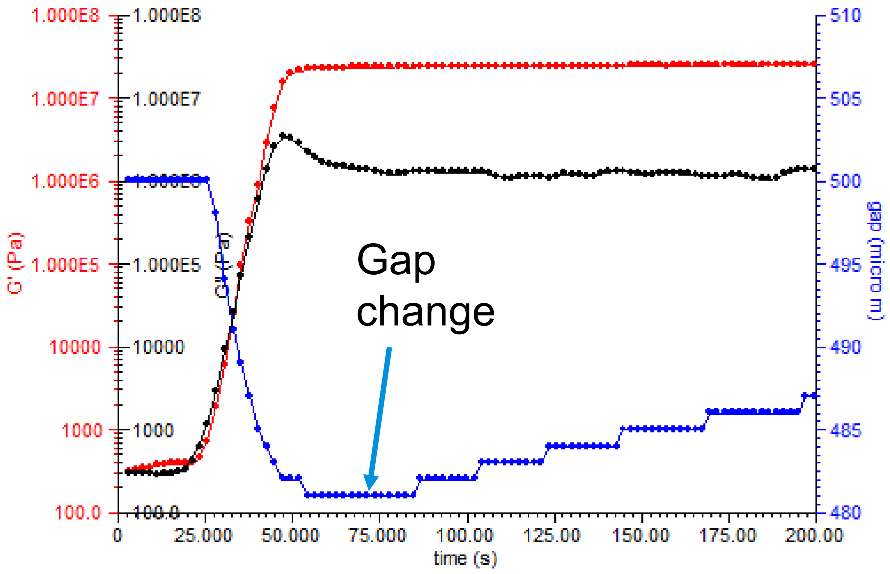


- Thermoset material shrinks during curing
- The amount of shrinkage could cause cracking or failure to the end products

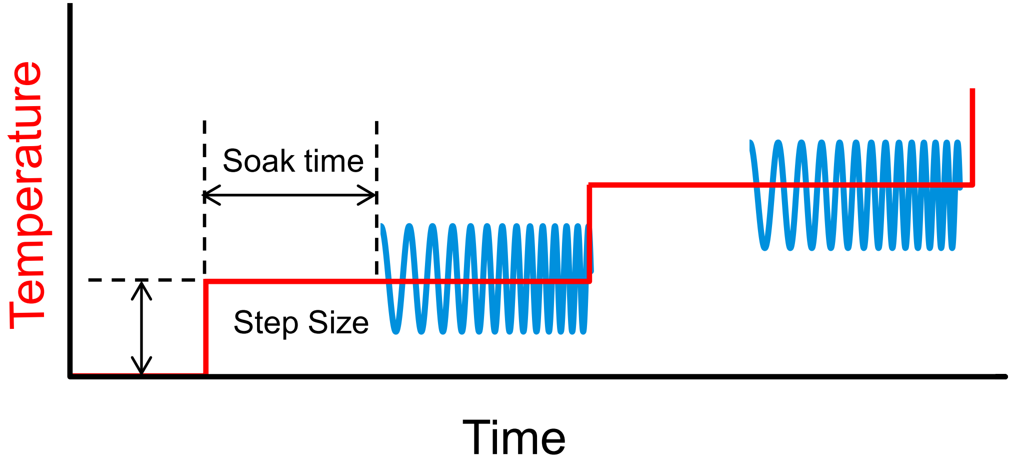
(1) Set Gap Constant
monitor shrinking force



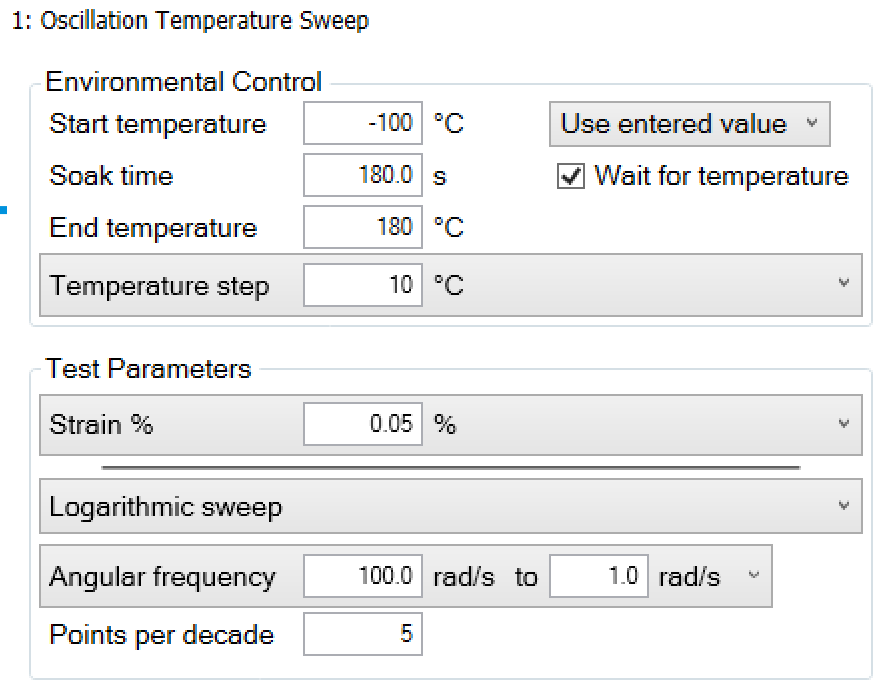
(2) Set Axial Force = 0
monitor dimension(gap) change



Temperature Sweep (or Step)



- Step and hold temperature then monitor material response. No thermal lag
- In TRIOS: Temp Sweep


1: Oscillation Temperature Sweep

Environmental Control

Start temperature	-100	°C	Use entered value
Soak time	180.0	s	☑ Wait for temperature
End temperature	180	°C	
Temperature step	10	°C	

Test Parameters

Strain %	0.05	%		
Logarithmic sweep				
Angular frequency	100.0	rad/s to	1.0	rad/s
Points per decade	5			

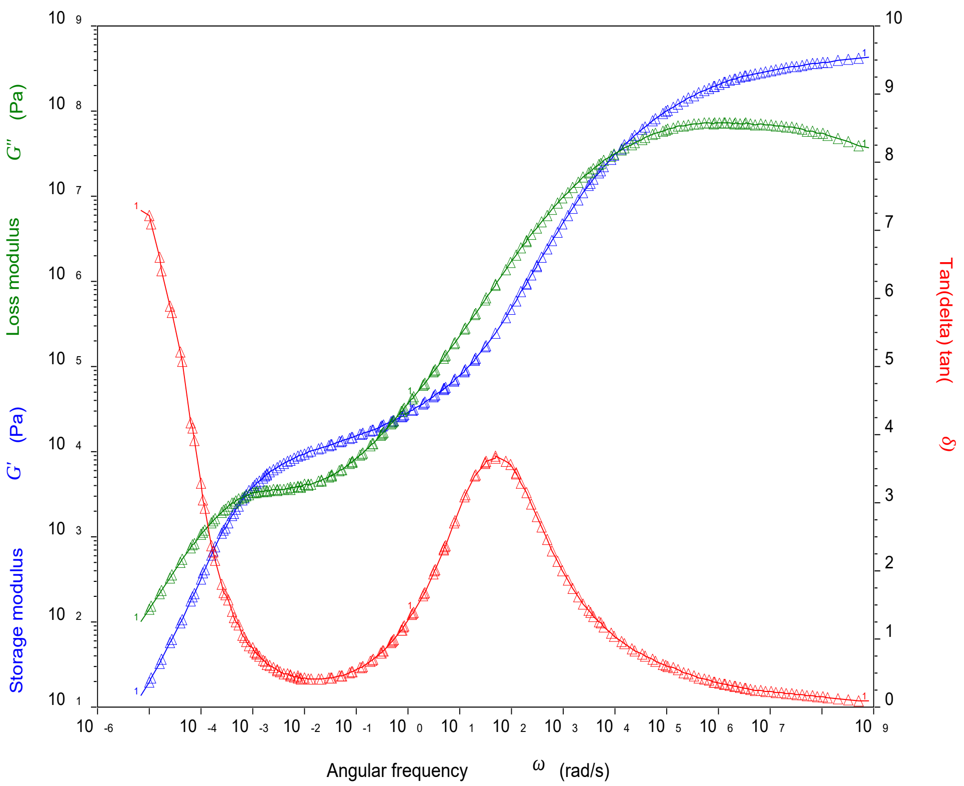
USES

- Measure material's viscoelastic properties vs. temperature
- Time-Temperature Superposition test (TTS)

Time-Temperature Superposition (TTS)

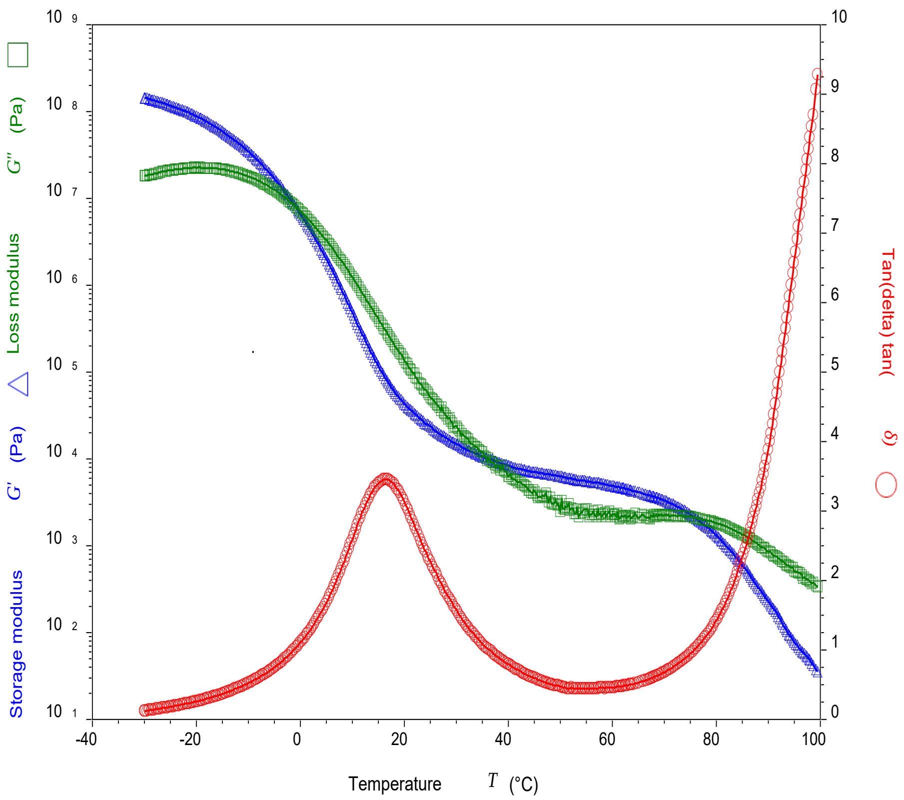
- The viscoelastic properties of an amorphous polymer as a function of temperature is mirror imaged as its properties as a function of time (frequency)

TTS master curve generated at 20°C



Time

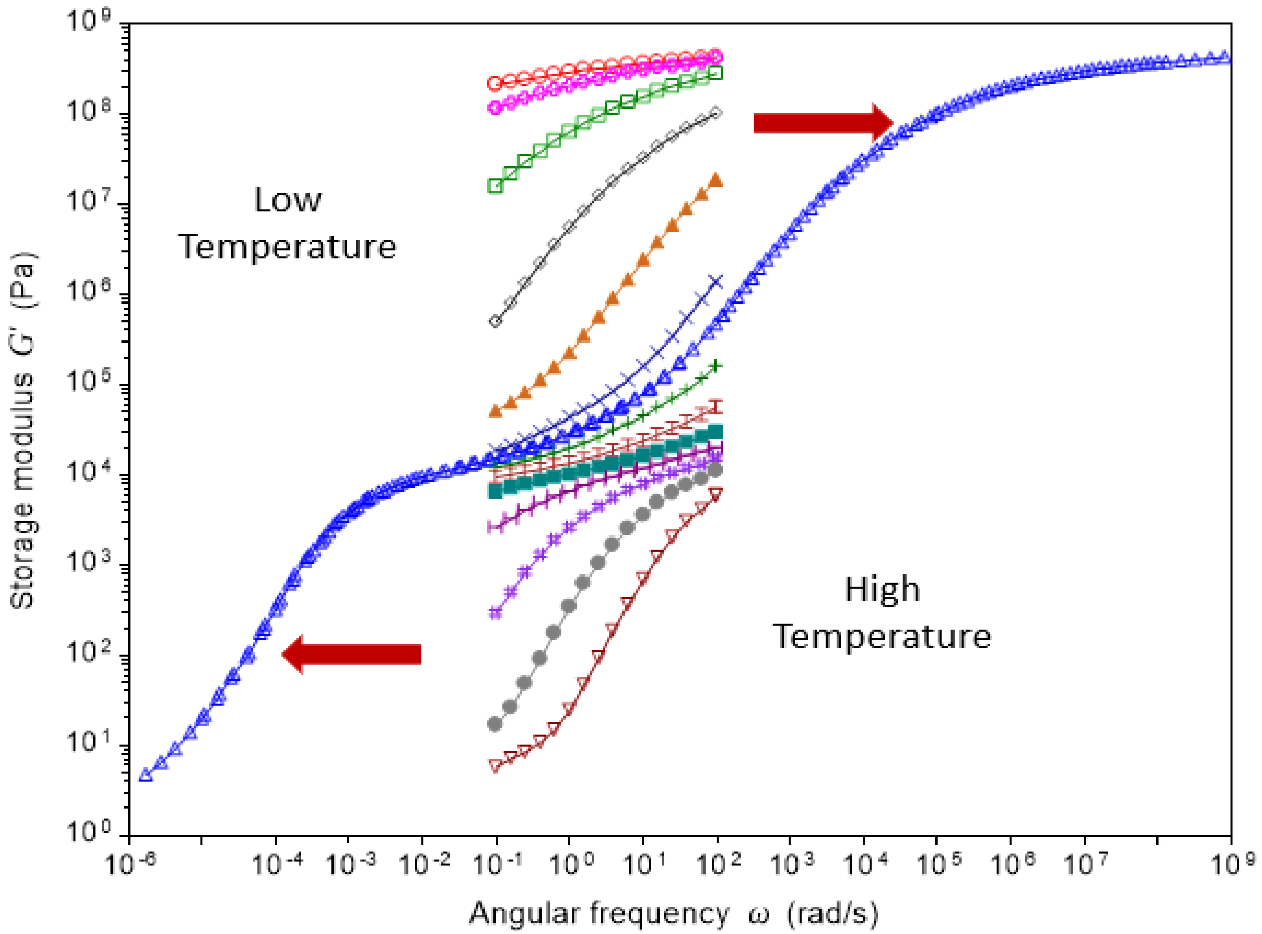
Dynamic temperature ramp



Temperature

Benefit of Time-Temperature Superposition

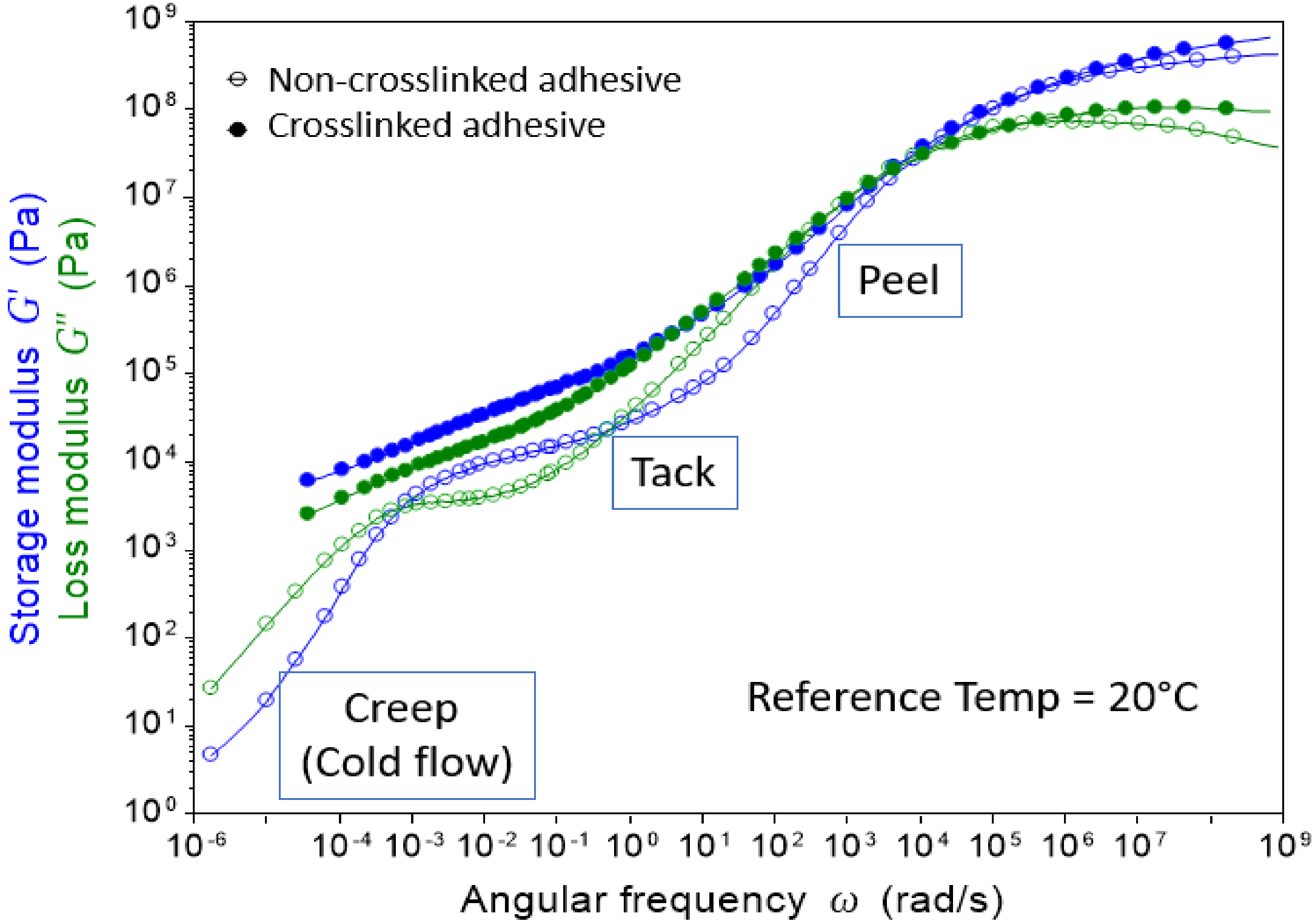
- Perform frequency sweeps at different temperature
- Extrapolates properties to time scales outside the instrumental range



- Frequency sweep data collected under different temperature steps can be shifted along the x-axis.
- Access to a wide range of frequency or time scales
- Predict properties outside of instrument capability
- TTS can be applied to both dynamic and transient measurements

TTS Benefit on Adhesive Analysis

- Dynamic TTS results for a non-crosslinked and a crosslinked pressure sensitive adhesive materials



Introduction to rheology accessories and hyphenated techniques

Popular Accessories on Rheometers

Powder accessory

Humidity chamber

Extensional Viscosity

UV accessory

OSP accessory

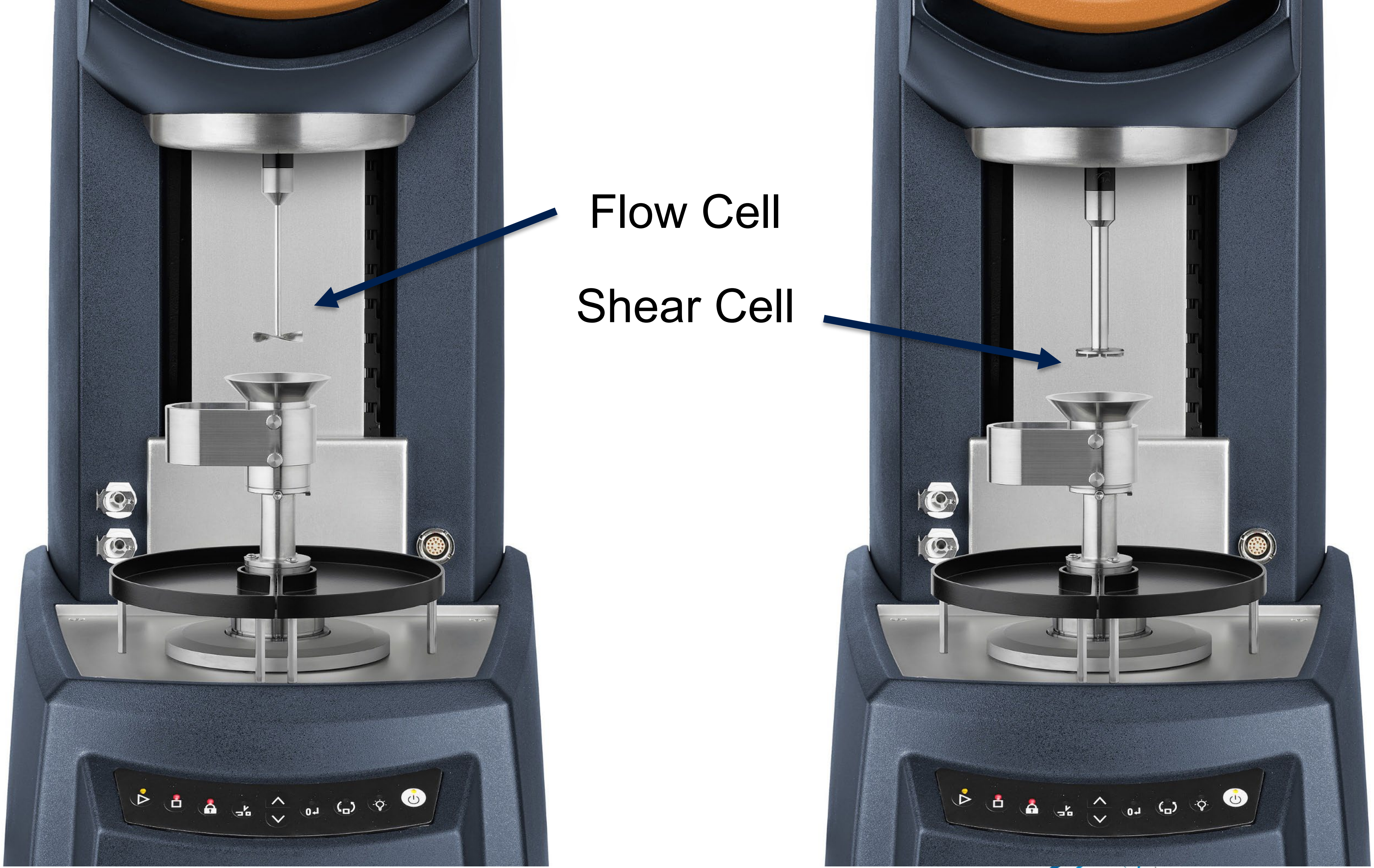
Rheo-Microscopy

Rheo-Raman

Rheo-Dielectric

HR x0 Powder Rheology Accessory

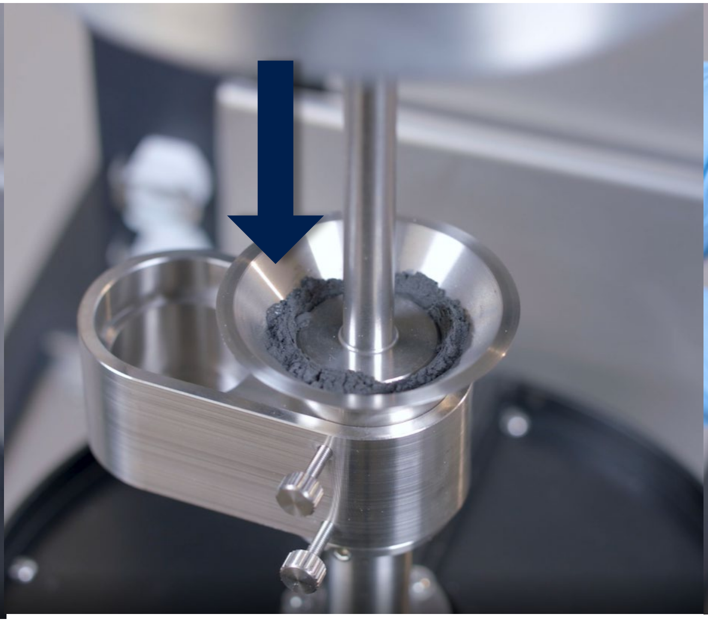
- Available on HR 10, HR20 and HR30 rheometer. Powder solution offers comprehensive powder flow analysis
  - Flowability
  - Shear
  - Wall friction (2023)
  - Compressibility (2023)
- Excellent repeatability (within 5%)
- Easy sample preparation: loading and trimming
- Integrated Smart Swap design. Accessory change in less than 10 seconds
- One click data analysis package in Trios


Flow Cell
Shear Cell

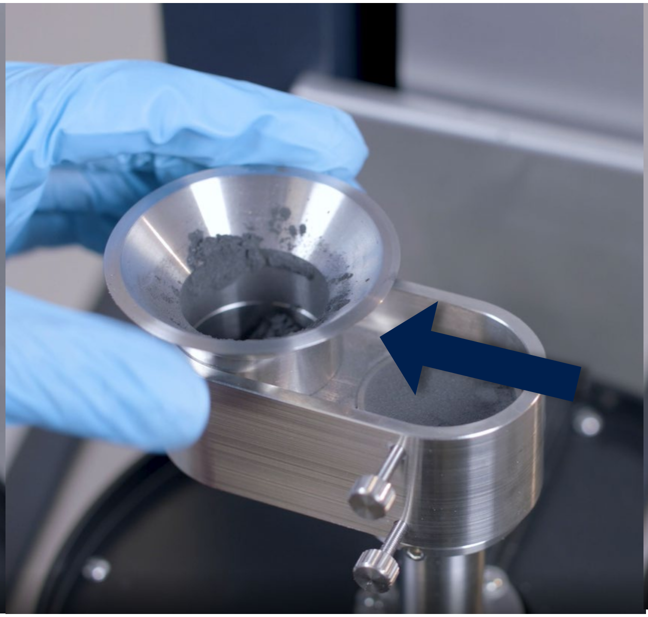
Powder Shear Cell sample preparation

Load Sample

>15 mL

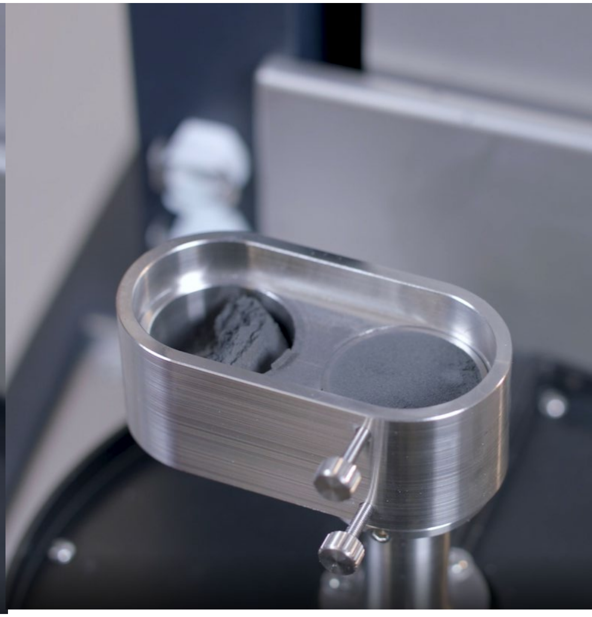


Sample Consolidation



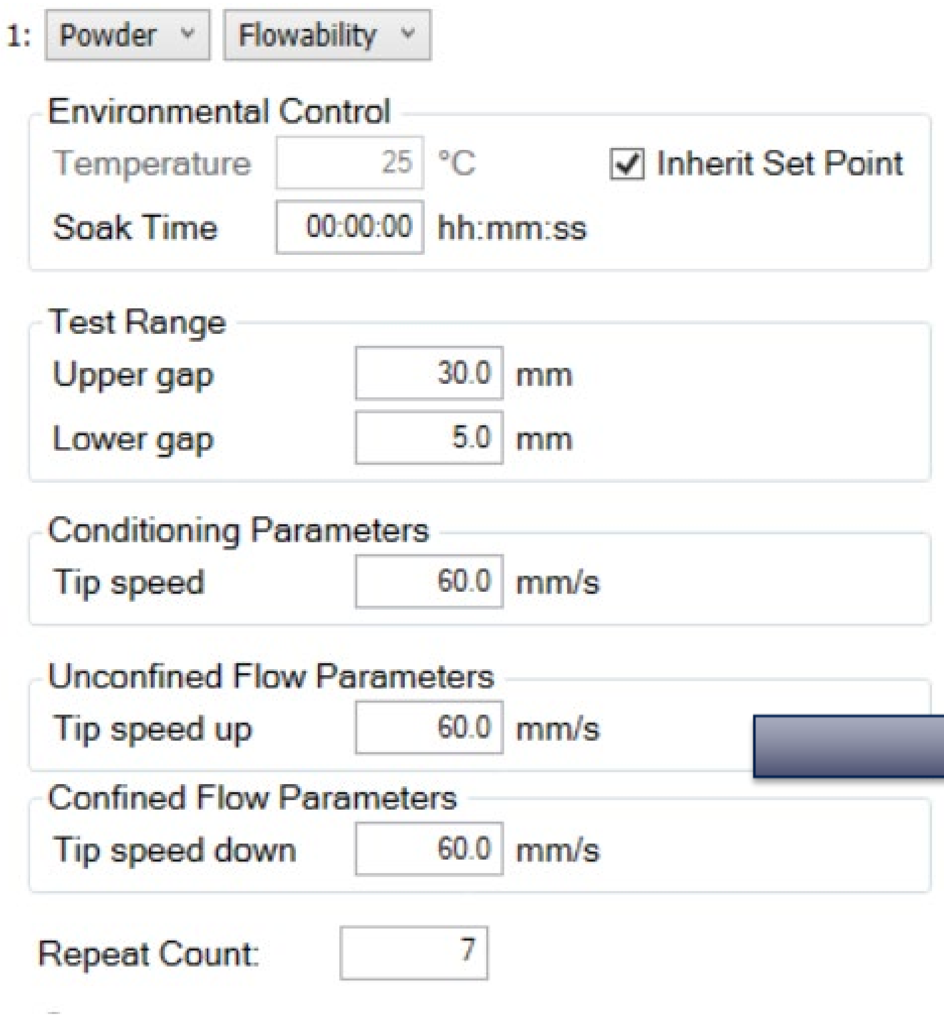
Trim Sample

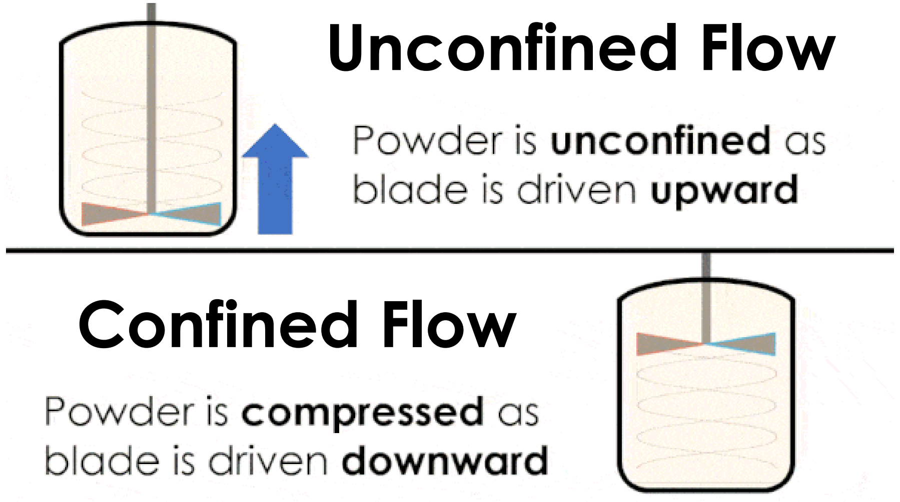
User prompt from the touchscreen

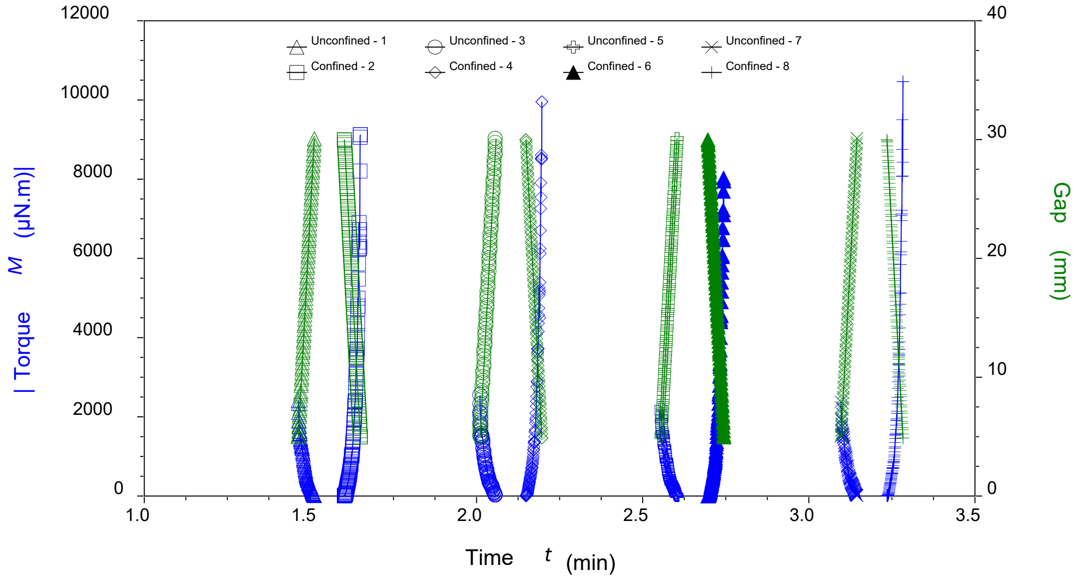


Start the Test

TRIOS simplifies Powder Flowability test

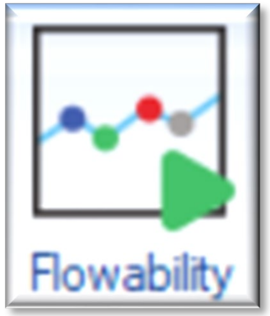






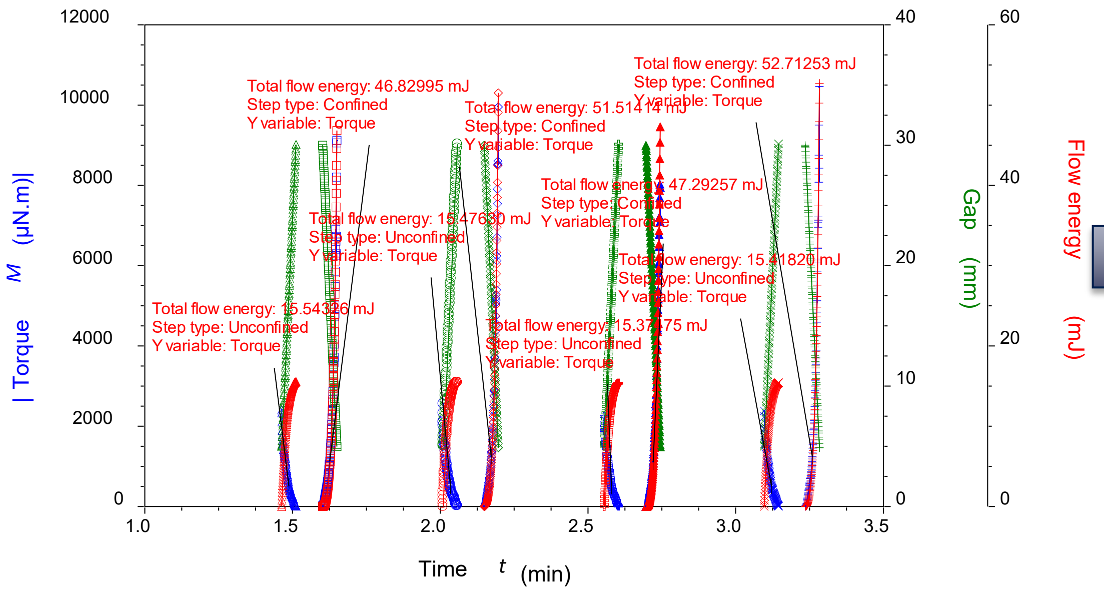
- Repeat steps – Stability Index
- Vary Tip Speed – Flow Rate Index

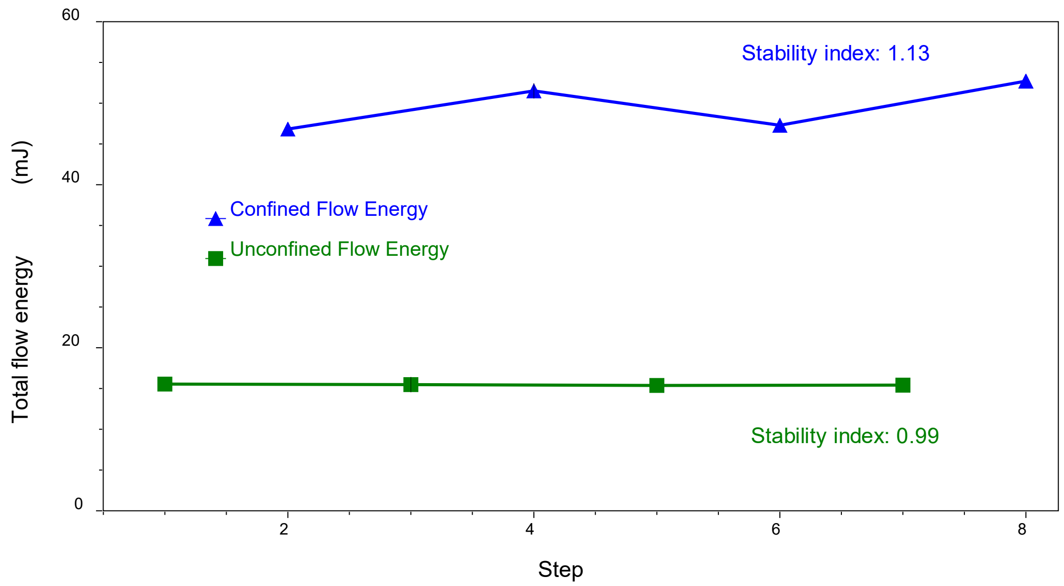
Powder Analysis Key automates Flowability calculation



TRIOS Powder Analysis Software key (optional)

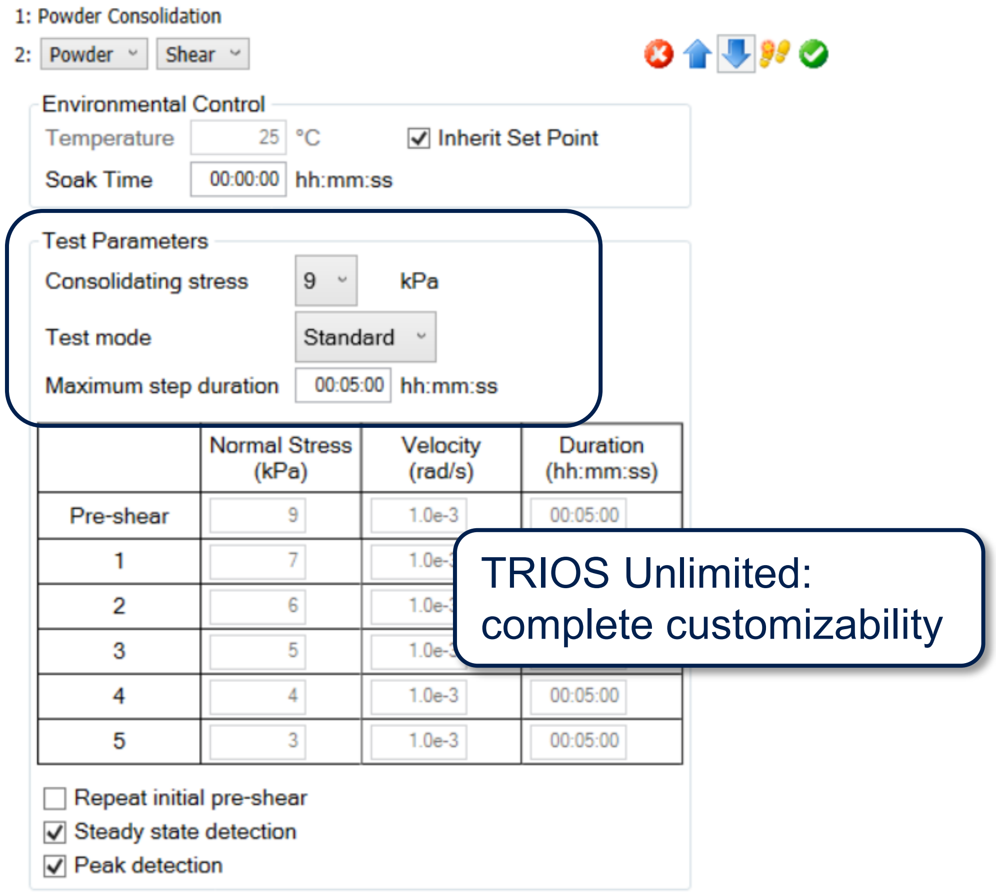
- Powder Key Performance Indicators are ***One Click away***
- Automated process saves user from complicated mathematical analysis
- Quantitative, relevant, repeatable results





$$\text{Flow Energy} = \underbrace{\int T * d\theta}_{\text{Torque energy}} + \underbrace{\int N * dh}_{\text{Force energy}}$$

TRIOS simplifies Powder Shear Test



1: Powder Consolidation
2: Powder | Shear

Environmental Control
Temperature 25 °C ☑ Inherit Set Point
Soak Time 00:00:00 hh:mm:ss

Test Parameters
Consolidating stress 9 kPa
Test mode Standard
Maximum step duration 00:05:00 hh:mm:ss

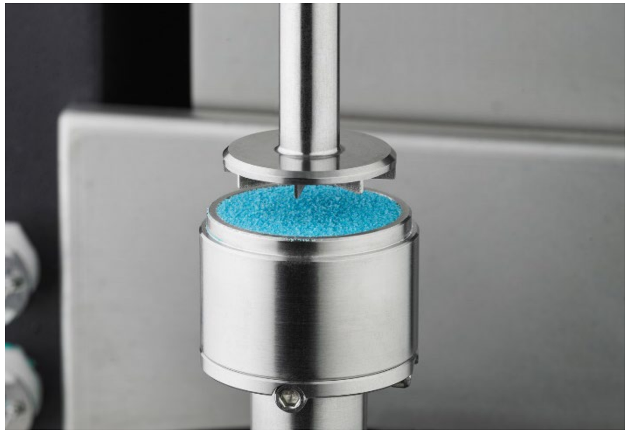
	Normal Stress (kPa)	Velocity (rad/s)	Duration (hh:mm:ss)
Pre-shear	9	1.0e-3	00:05:00
1	7	1.0e-	[illegible]
2	6	1.0e-	[illegible]
3	5	1.0e-	[illegible]
4	4	1.0e-3	00:05:00
5	3	1.0e-3	00:05:00

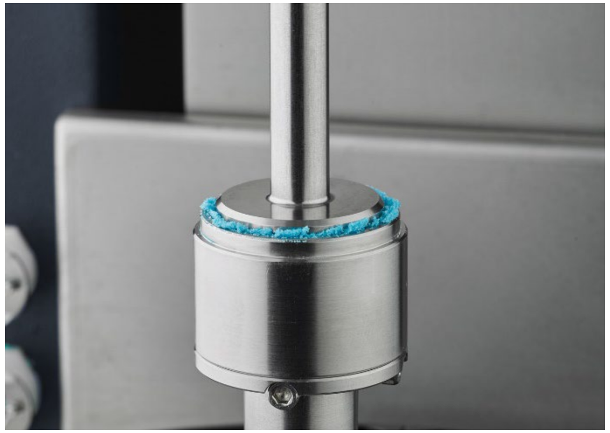
☐ Repeat initial pre-shear
☑ Steady state detection
☑ Peak detection

TRIOS Unlimited: complete customizability

TRIOS Powder-Shear test form performs ASTM D7891 measurement

- "Pre-Shear": Apply consolidating normal stress (9 kPa), pre-shear until steady state
- "Shear" Reduce normal stress, shear until yield
- Repeat consolidating pre-shear
- Repeat shear with decreasing normal stress





TRIOS simplifies Powder Shear Test

1: Powder Consolidation

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Environmental Control

Temperature 25 °C ☑ Inherit Set Point

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4	4	1.0e-3	00:05:00
5	3	1.0e-3	00:05:00

☐ Repeat initial pre-shear

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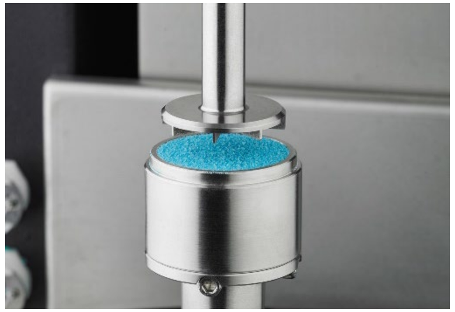
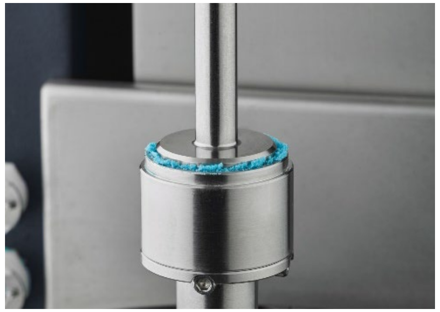
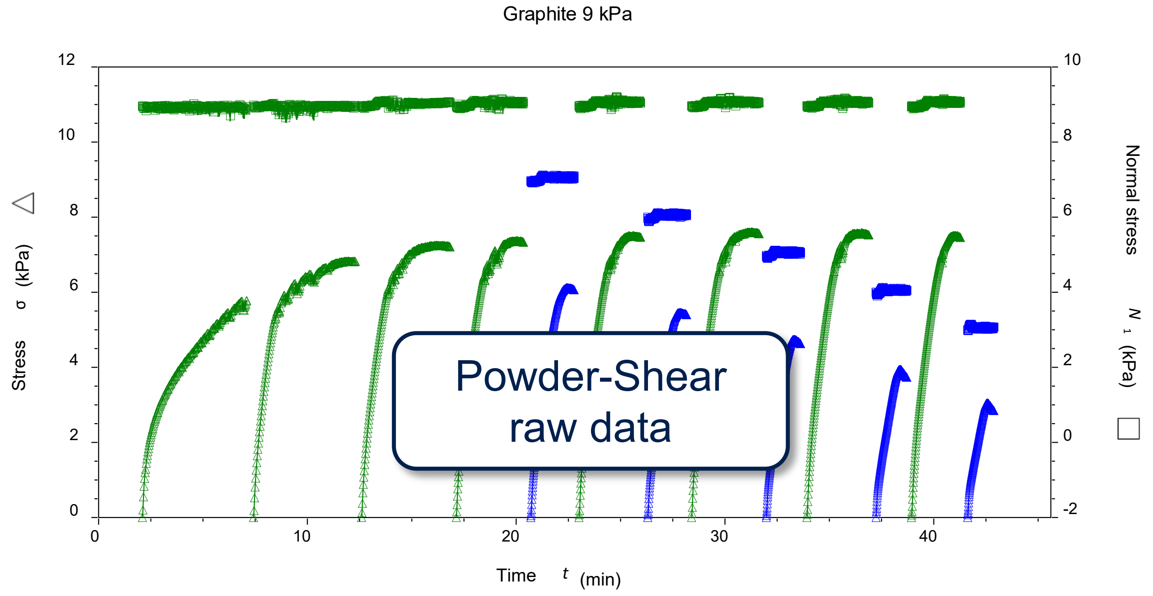
☑ Peak detection



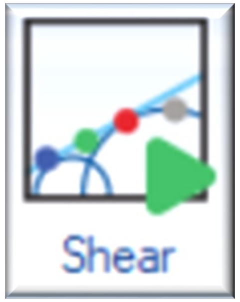


TRIOS Powder – Shear test form performs measurement per **ASTM D7891**

- "Pre-Shear": Apply consolidating normal stress (9 kPa), pre-shear until steady state
- "Shear" Reduce normal stress, shear until yield
- Repeat consolidating pre-shear
- Repeat shear with decreasing normal stress



Powder Analysis Key automates Shear calculations



TRIOS Powder Analysis Software key (optional)

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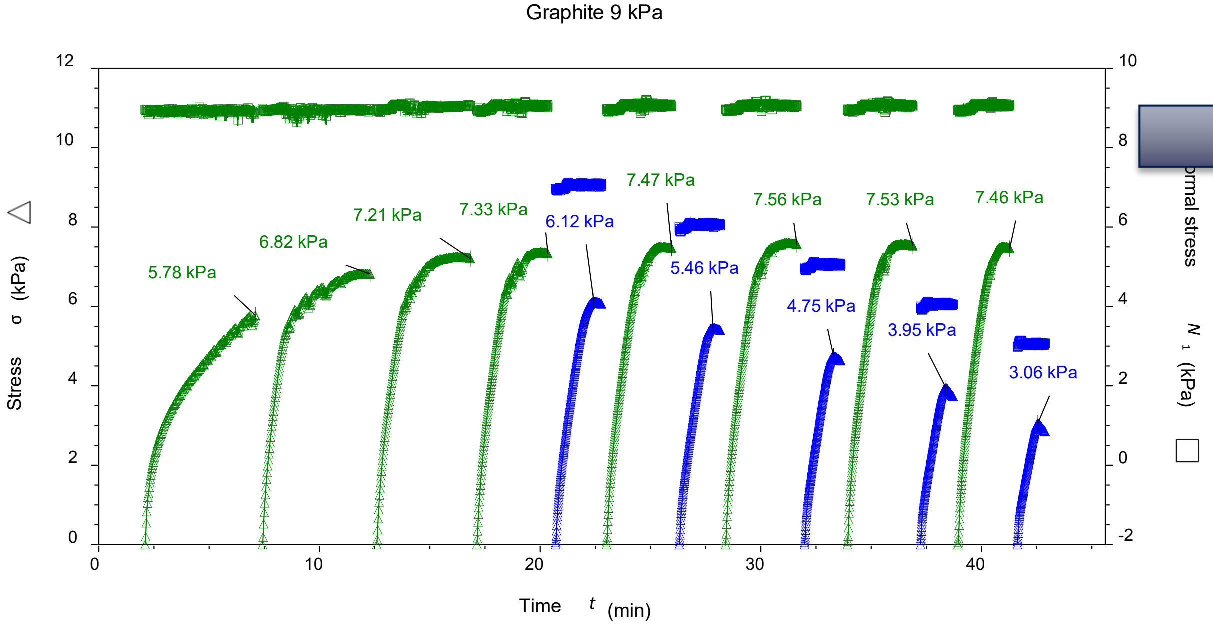
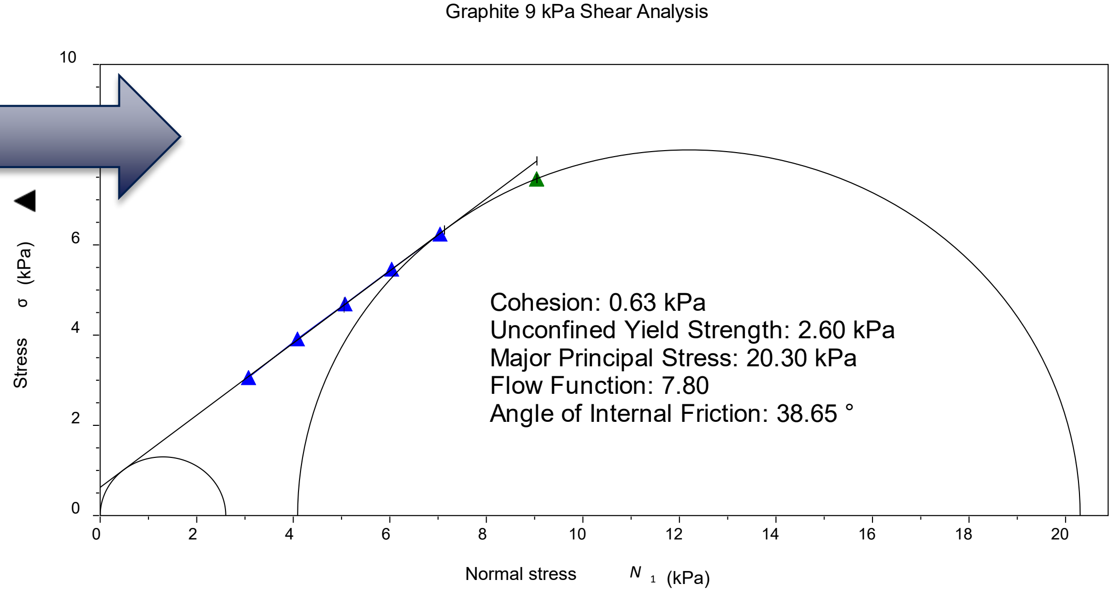




DHR-RH Accessory

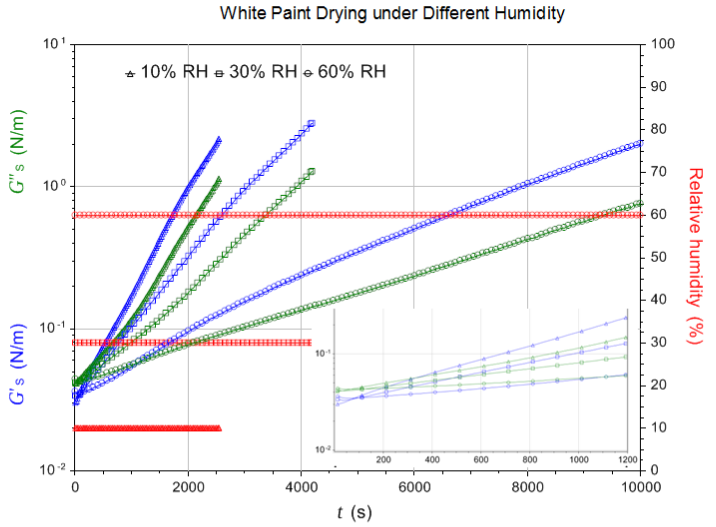
Features and Benefits

- Uniform, reliable control of temperature and relative humidity over full operating range
- Optimized gas flow eliminates interference from laboratory conditions
- Completely integrated system including native control and coordination of temperature, humidity, and rheology through powerful TRIOS software
- Wide variety of test geometries:
  - Standard parallel plate
  - Disposable parallel plate
  - Film Tension
  - Annular Ring
  - Surface Diffusion
  - Torsion Rectangular
  - Three-Point Bending
- Innovative geometries for RH: true humidity-dependent rheology, not dominated by diffusion
- True Dynamic Axial Film Tension
- Tribo-rheometry – Ring on plate, 3 Balls on Plate
- DMA geometries for true dynamic deformation in the axial direction: film tension, 3-point bending, single/dual cantilever, compression

Specifications	
Temperature Range	5 ˚C to 120 ˚C
Humidity Range	5% to 95%
Humidity Accuracy	5-90% RH: ± 3% RH > 90% RH: ± 5% RH

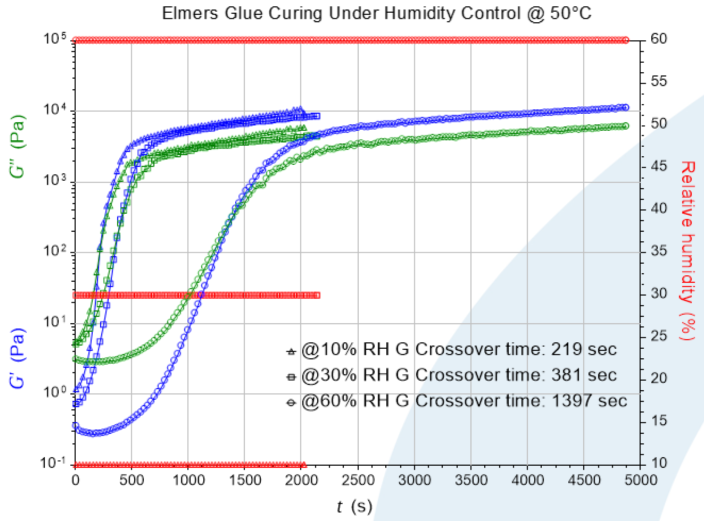
Example of Humidity Controlled Measurements

- Paint drying



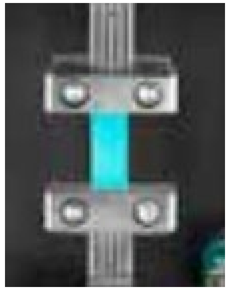
T Chen, TA apps note RH024

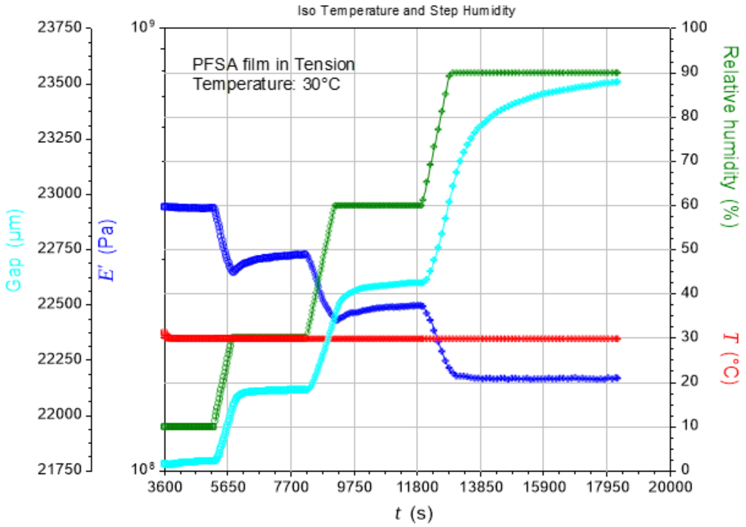
- Adhesive curing



T Chen, TA apps note RH101

- Polymer CHE





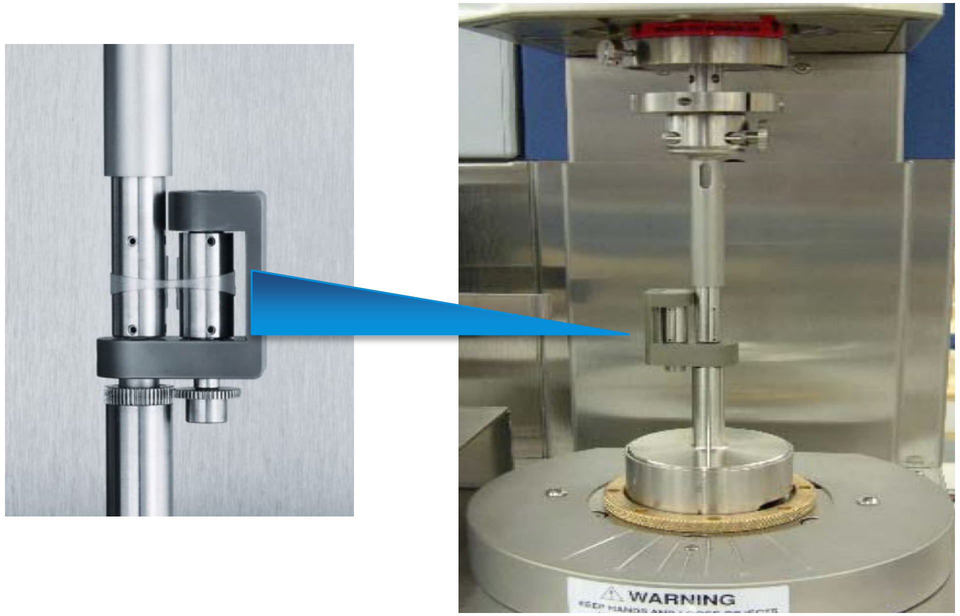
T Chen, TA apps note RH112

Extensional Viscosity Measurements on Polymer Melts

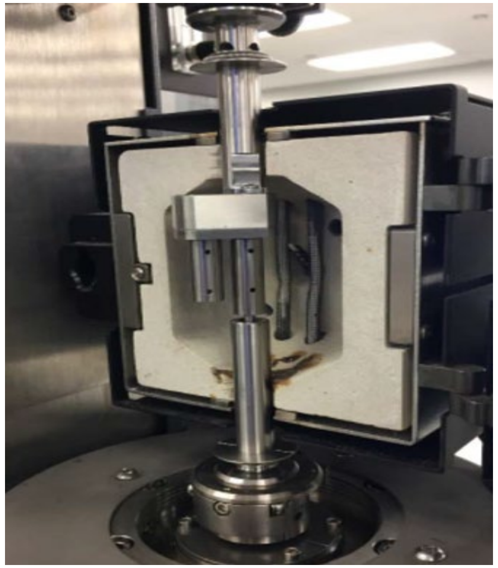
- Nonlinear elongation flow is more sensitive for some structure elements (e.g. branching) than shear flows

- Many processing flows are elongation flows. Extensional viscosity measurements can be used to help predict processability

ARES G2 - EVF



ARES G2 - EVA

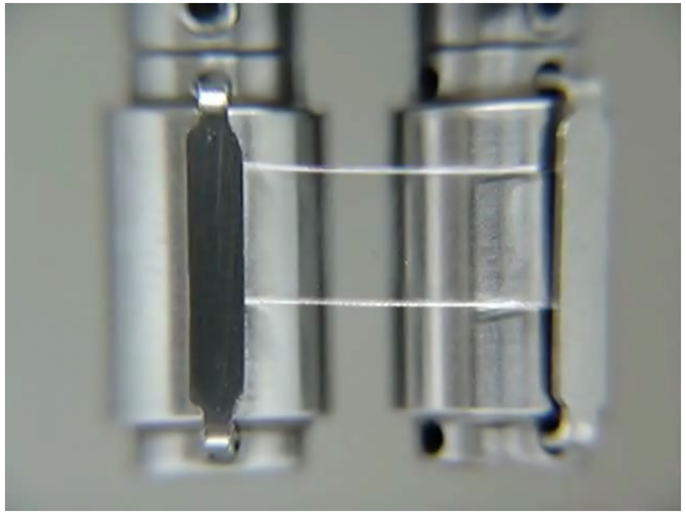


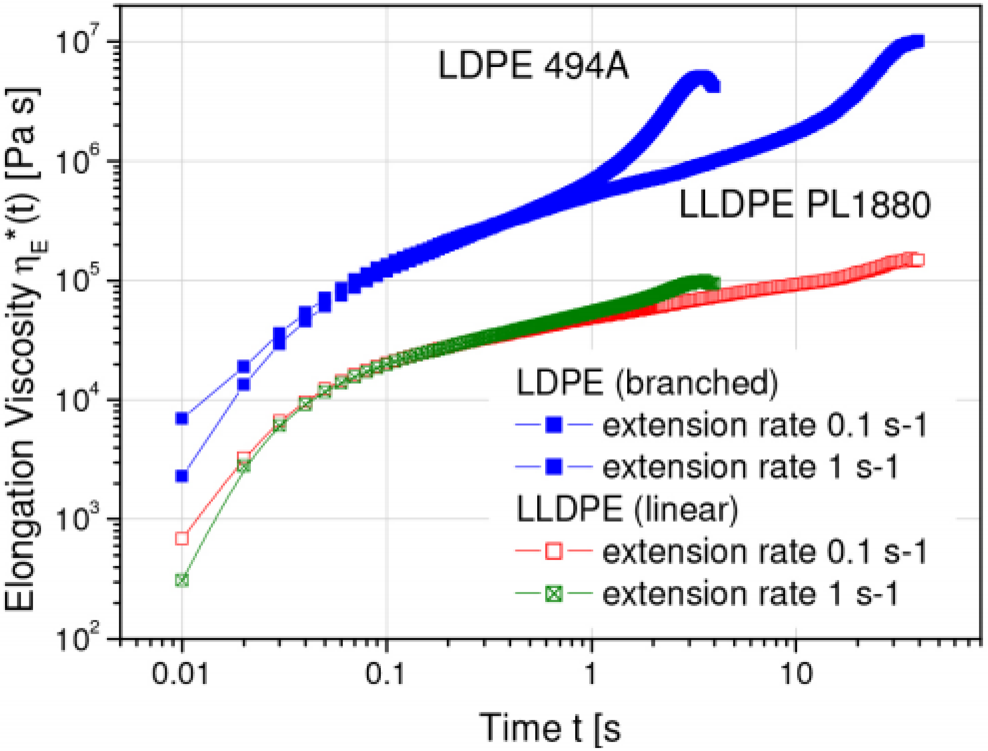
DHR/HR - EVA

Example of Extensional Rheology

- Many processing flows are elongational flows
- Nonlinear elongation flow is more sensitive for some structure elements (e.g. branching) than shear flows

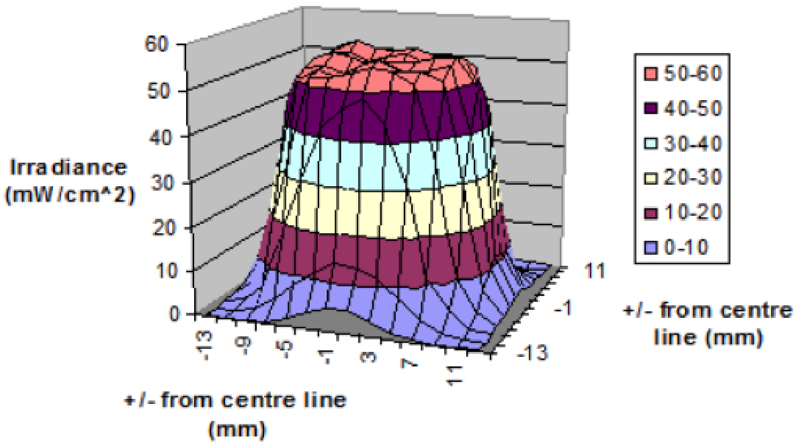
- Polymer melt extension – SER or EVF





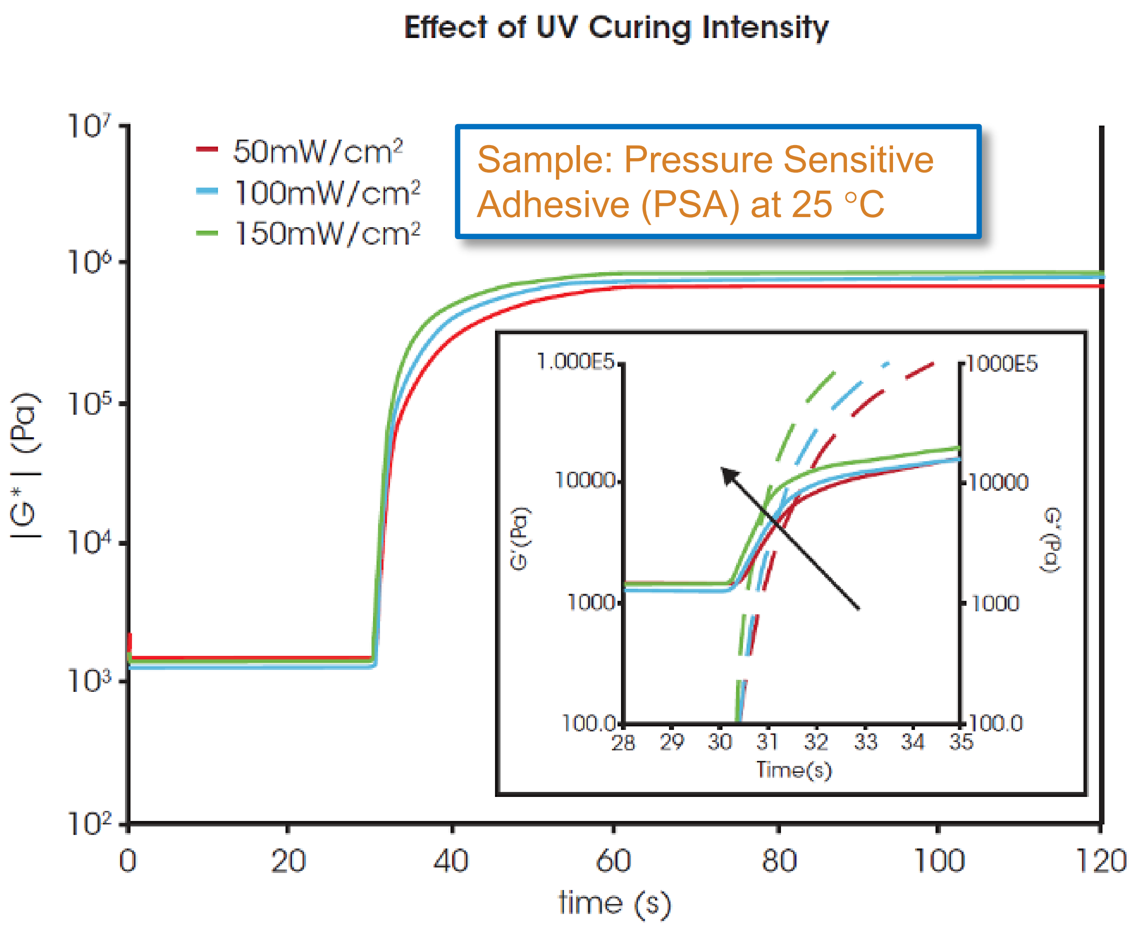
TA apps note APN002

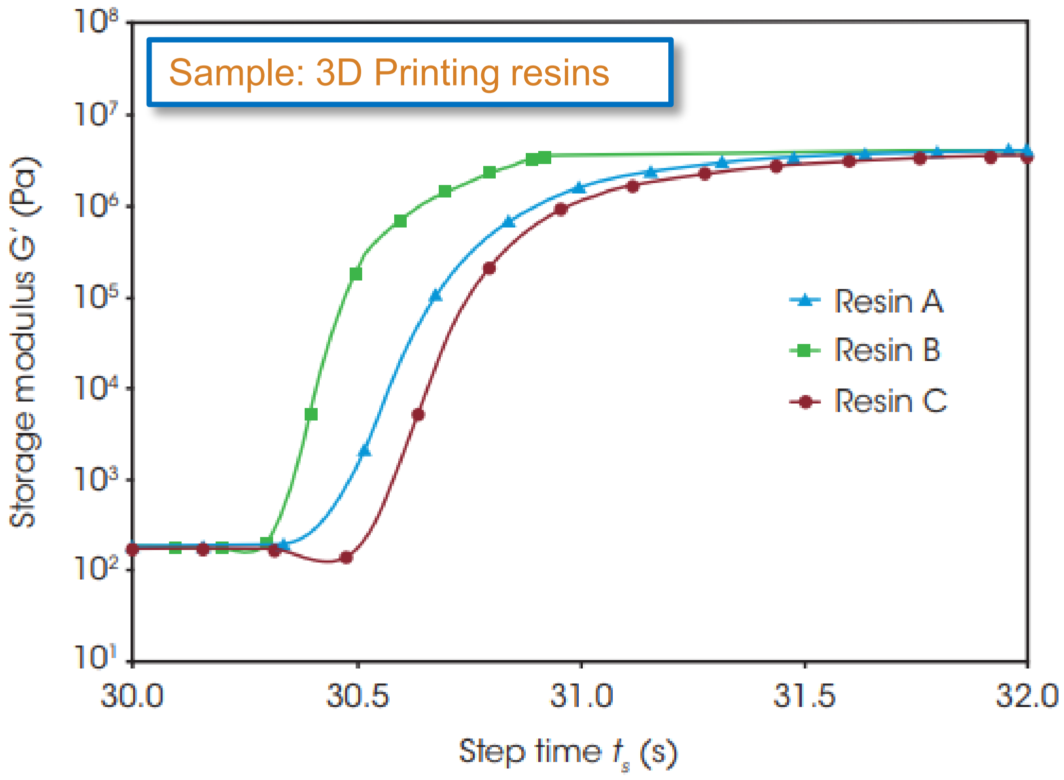
UV Light Guide Curing Accessory



- Collimated light and mirror assembly insure uniform irradiance across plate diameter
- Maximum UV 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 available
- LED UV for HR rheometer: choice of two standard wavelengths at 365nm and 455nm.

UV Curing Applications

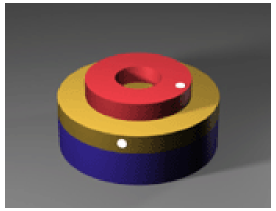




T Chen, TA apps notes AAN021, RH032

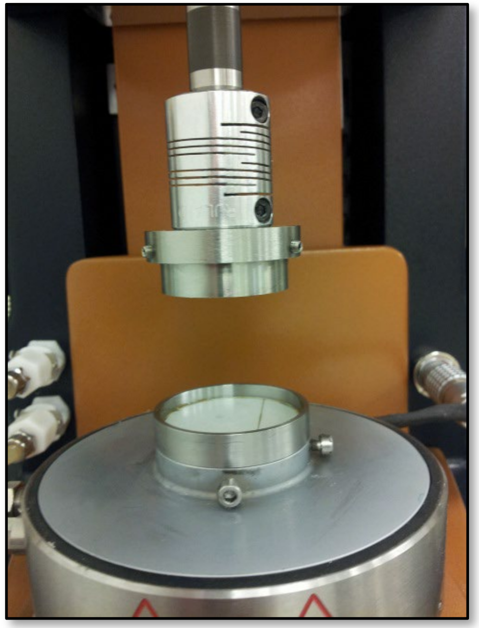
K Coasey, TA apps note RH118

Tribo-rheometry Accessory



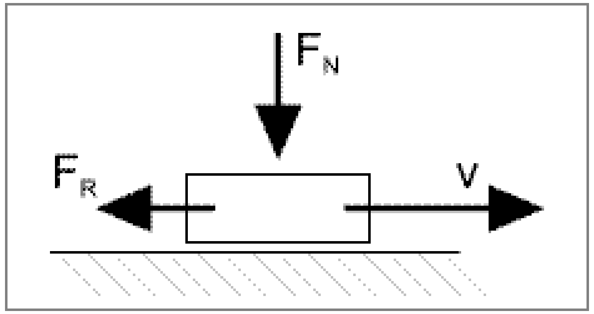
Tribology is the study of interacting surfaces in relative motion, it includes:

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



Requires:

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

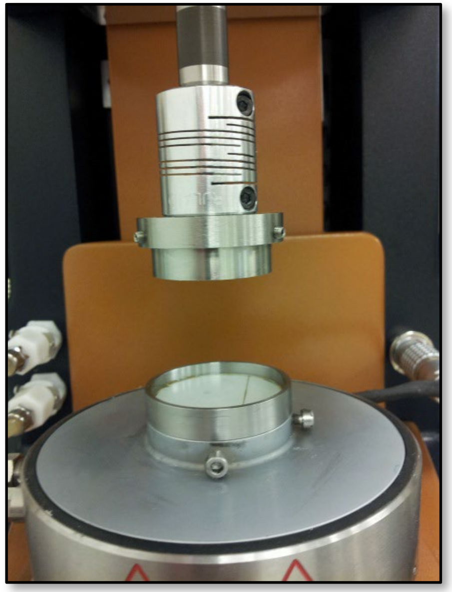


$$\mu = \frac{F_R}{F_N} = \frac{\text{shear stress}}{\text{normal stress}}$$

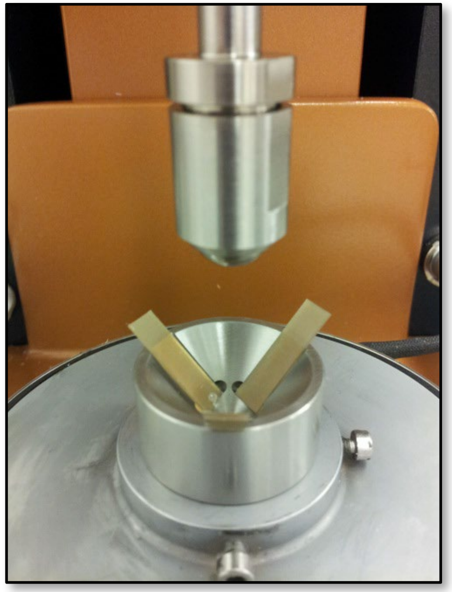
Peltier Tribo-rheometry Accessory

- Available on both Discovery HR rheometers and the ARES G2

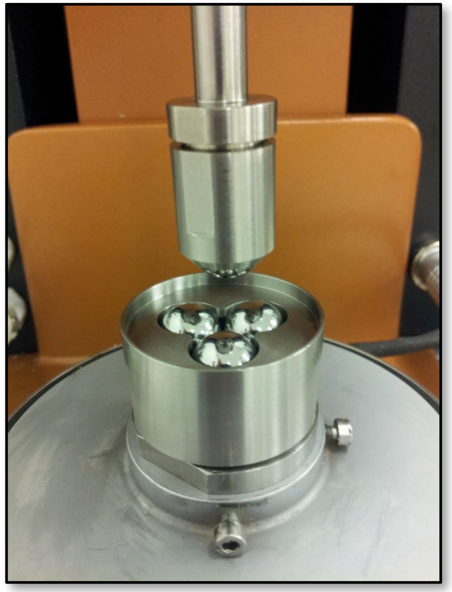
Ring on Plate



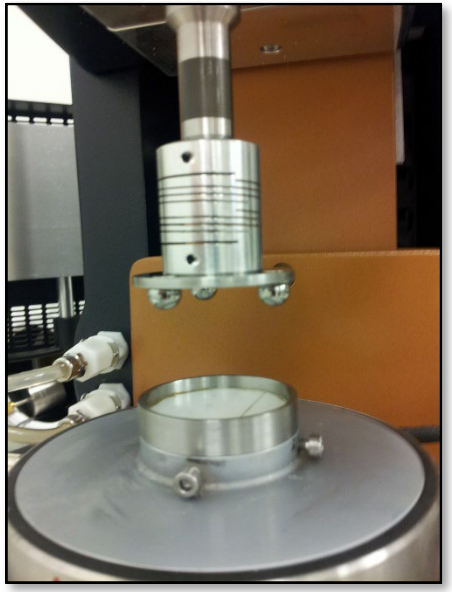
Ball on 3 Plates



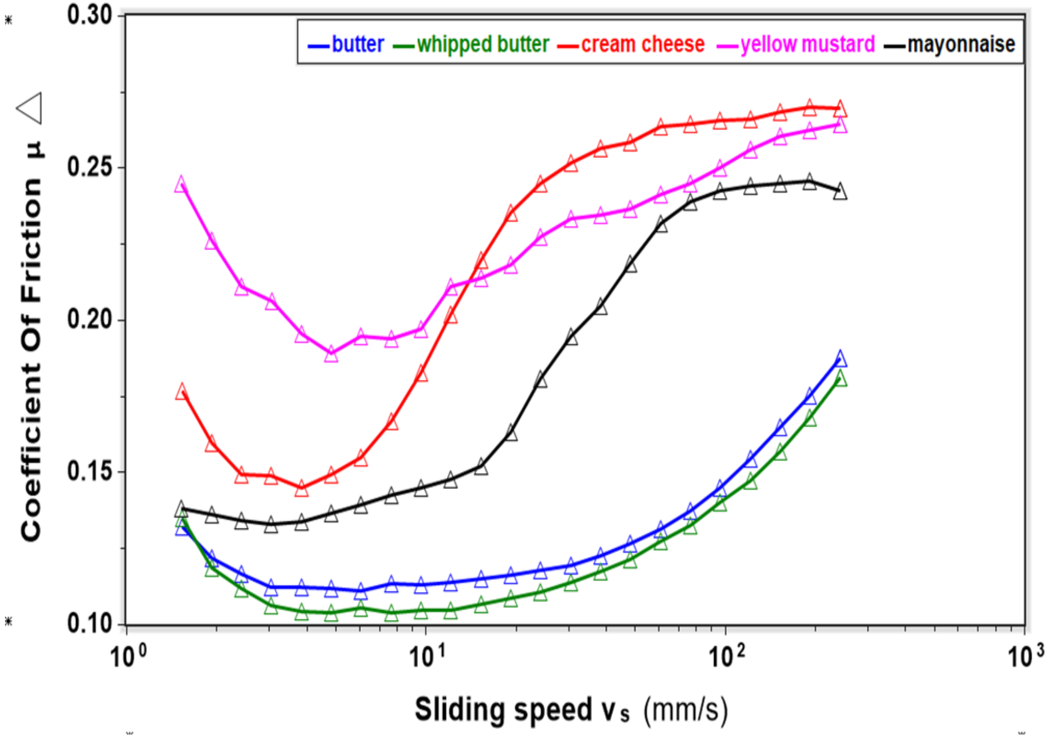
Ball on 3 Balls

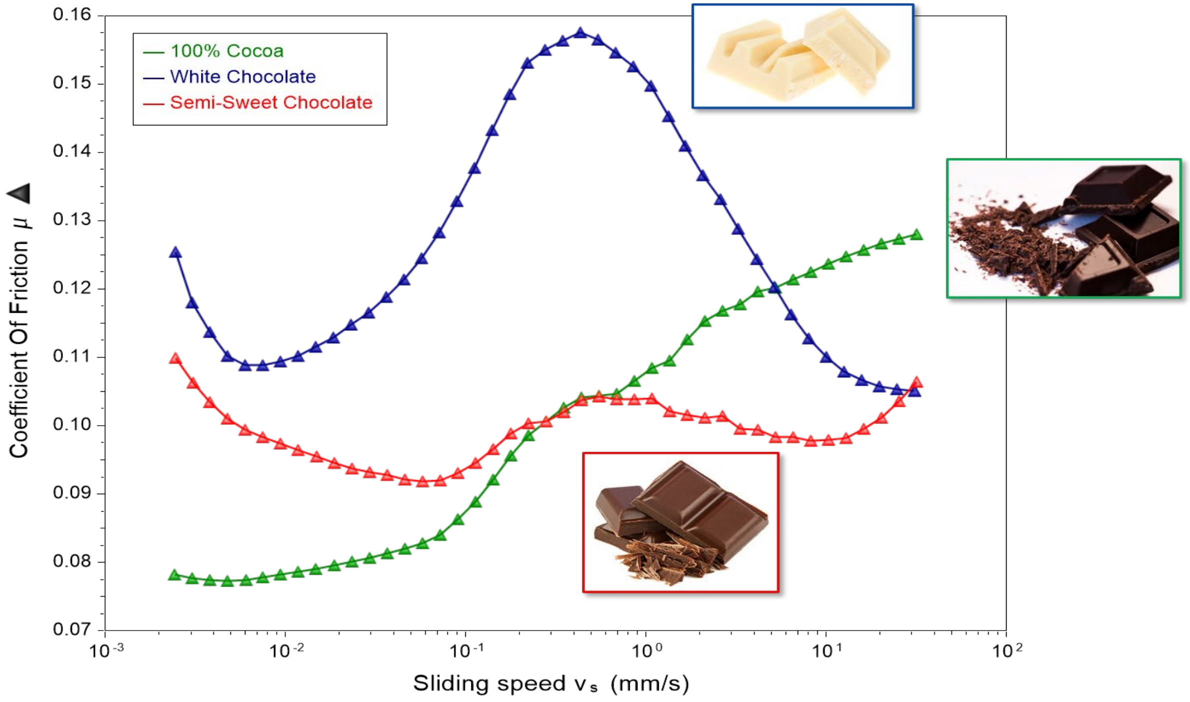


3 Balls on Plate



Examples of Food tribology





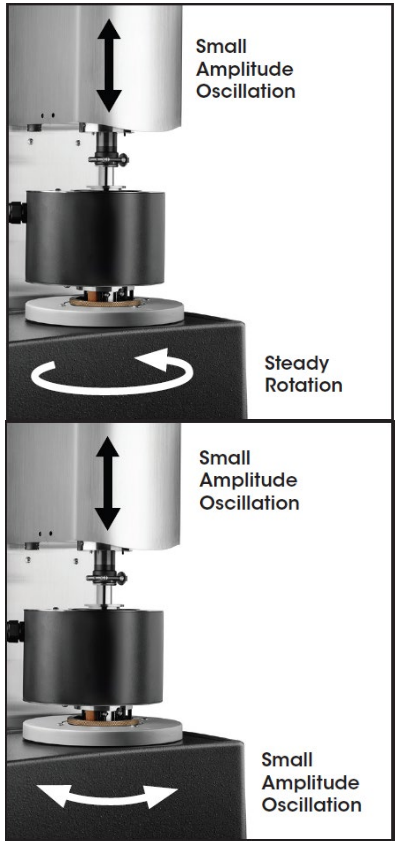
TA Webinars:
https://www.tainstruments.com/an-introduction-to-tribo-rheometry-quantifying-friction/
https://www.tainstruments.com/applications-of-tribo-rheometry-in-material-characterization/

Orthogonal Superposition Accessory

OSP Features and Benefits

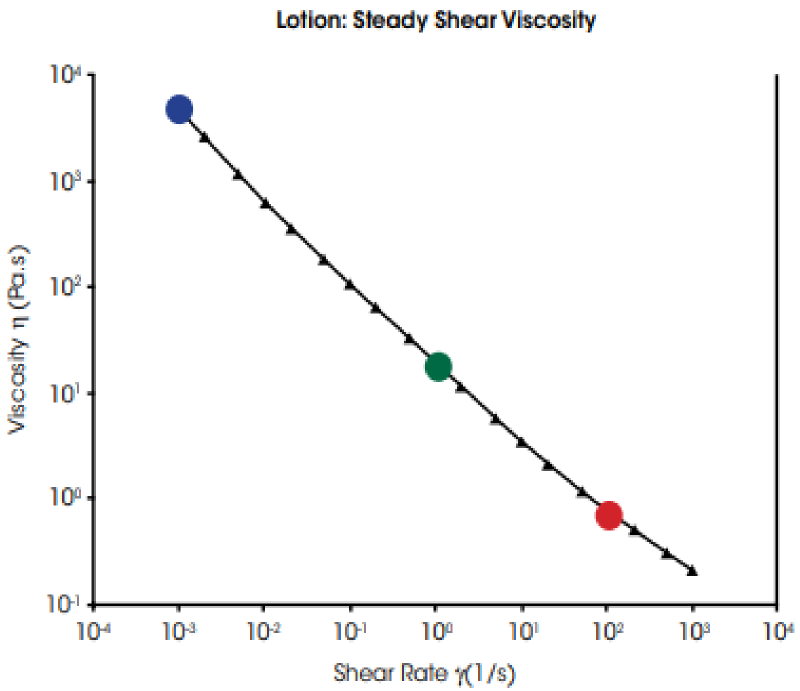
- Available on both Discovery HR rheometer and ARES G2
- Ensure accuracy in both rotational and axial measurements, avoiding artifacts from pumping effects and surface tension with the specially designed OSP double-gap cup and rotor
- OSP and 2D-SAOS experiments fully programmable from TRIOS Software. Simultaneous measurements in two directions
- Monitor temperature-dependent changes and replicate real-world processing conditions with optional temperature control through the Environmental Test Chamber (-10 °C to 150 °C)

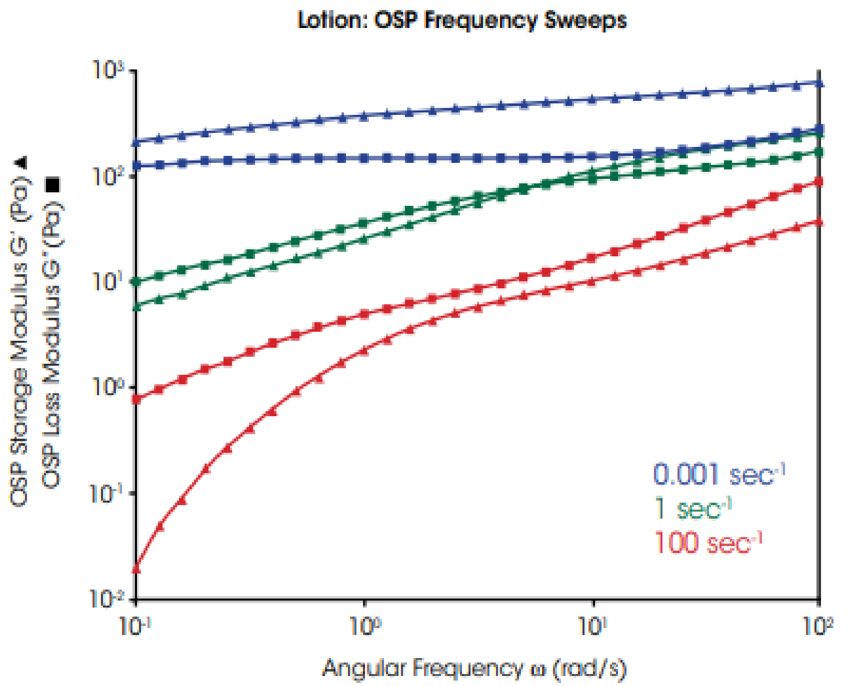
ARES G2 Rheometer



HRx0 Rheometer

Example of Orthogonal Superposition analysis on Lotion



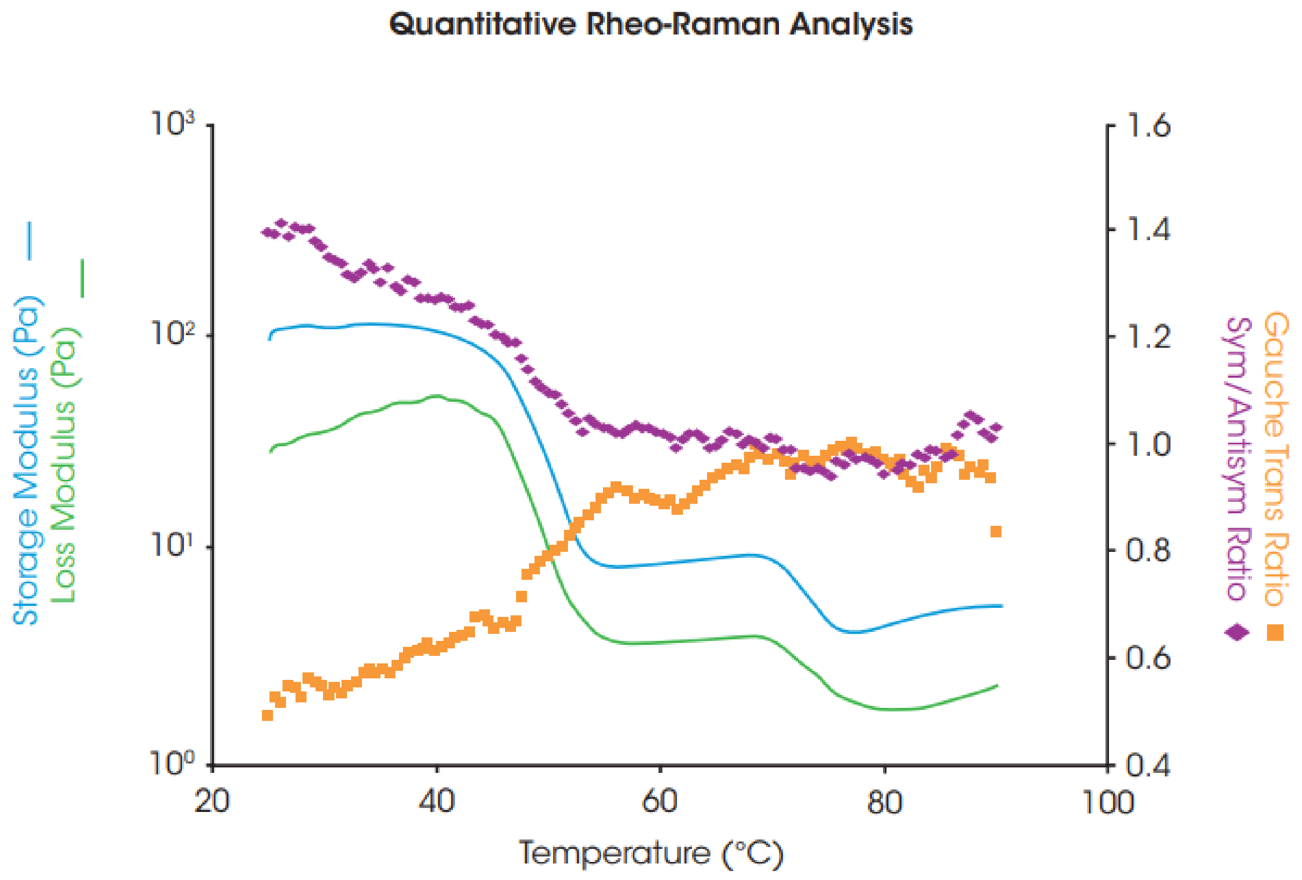


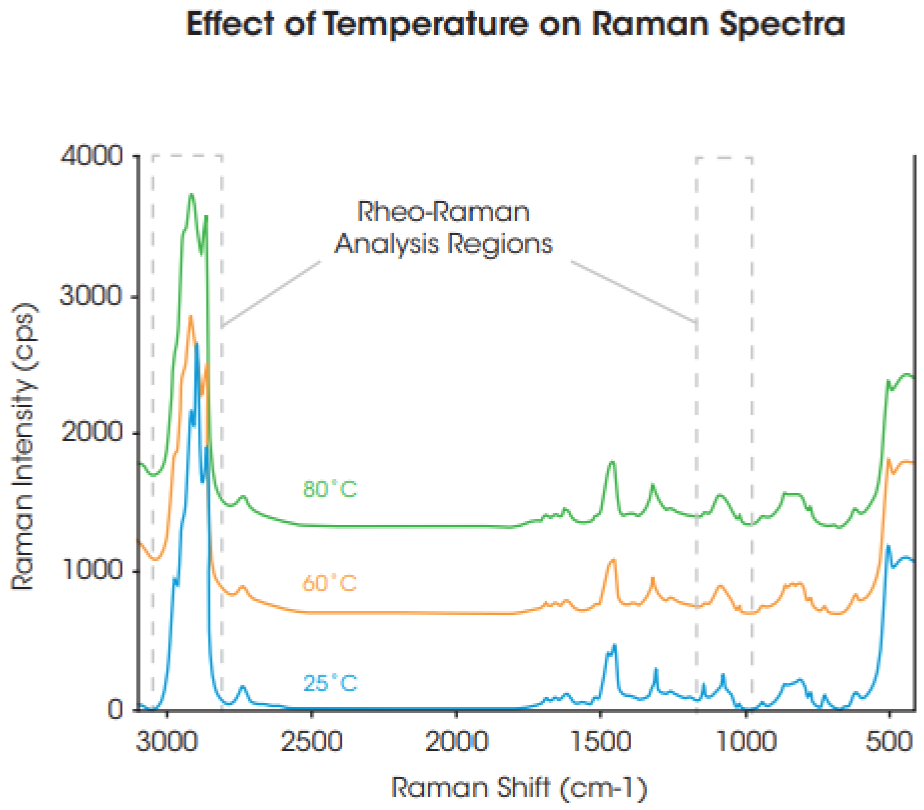
Rheo-Raman

Features and Benefits

- Integrated operation with Thermo Fisher™ iXR Raman spectrometer
- Choice of different lasers for maximum versatility
  - 432 nm, 532 nm and 785 nm (high brightness, high power options)
  - User installable laser sets — quick and easy to switch lasers
- Multiple interlocks for class 1 laser classification
- Safe under all conditions of normal use
- Free space coupling maximizes laser intensity at sample
- Software integration provides seamless Rheo-Raman experiments
  - Synchronized data collection between TRIOS and Thermo Scientific™ OMNIC™ software
  - Rheo-Raman data analysis performed in OMNIC software
- Temperature control up to 100 °C using Upper Heated Plate (UHP)
- Optical table provides excellent stability, alignment, and vibration free measurements
- Micrometer driven access to any radial positions
- Fine axial adjustment for focusing inside the sample
- Quartz plates minimize fluorescence artefacts

Simultaneous Rheology-Ramen Analysis

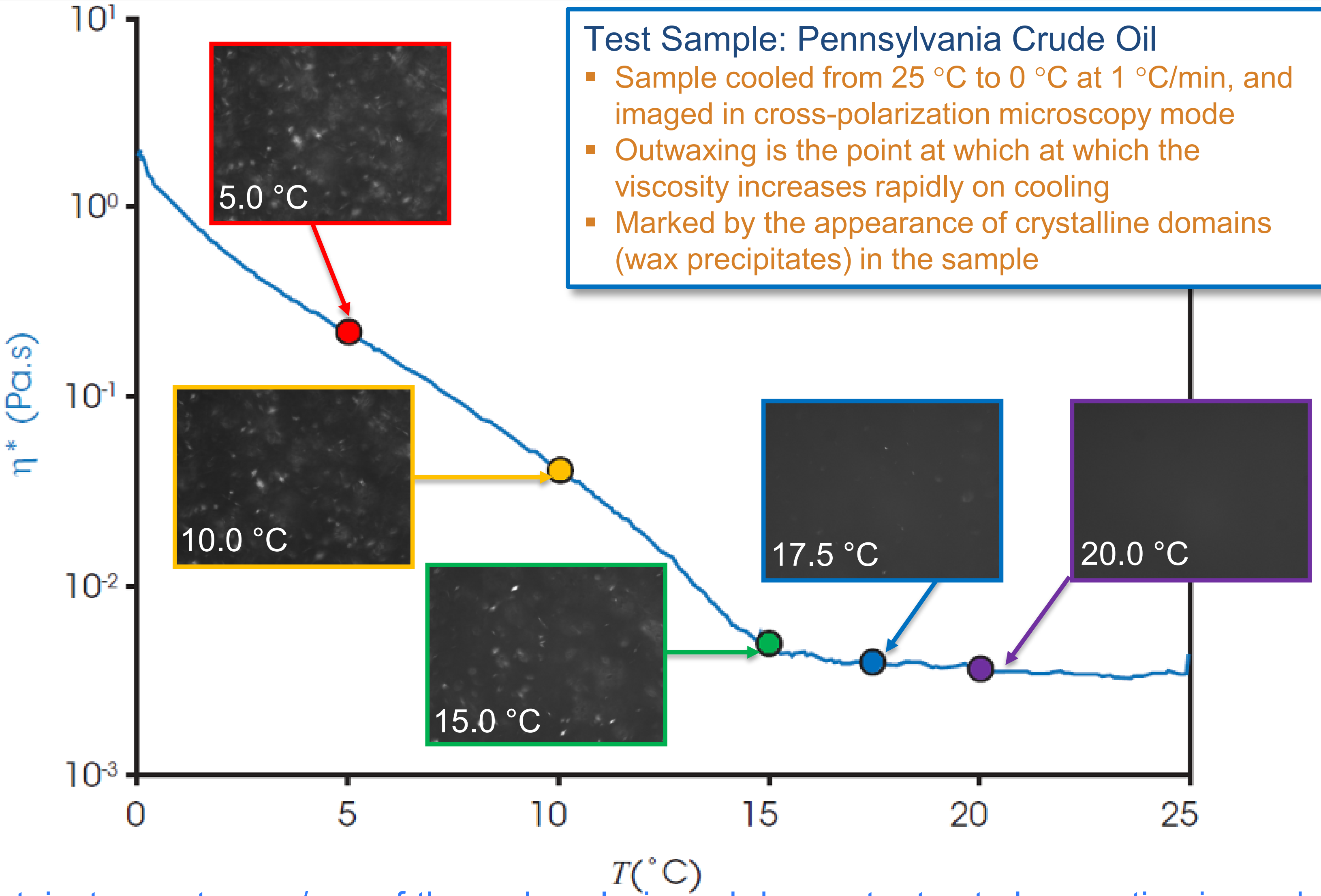




Modular Microscope Accessory (MMA)

- Available on Discovery HR rheoemters.
- Compact, modular design, that mounts on instrument frame for easy alignment and minimum vibration
- Uses standard Nikon microscopy objectives. Tested with 20× to 100×.
- Fast speed camera for image capture up to 90 fps
- X-Y-Z micrometer stage controls to select observation position
- Brightfield microscopy with cross-polarization
- Modular Options:
  - Counter-rotating plate for zero-velocity stagnation plane
  - Piezo-scanning stage for 100 µm depth profiling
  - Dichroic splitter for fluorescence microscopy
  - UHP for temperature control up to 100°C

MMA Application: Crude Oil Outwaxing



Test Sample: Pennsylvania Crude Oil

- Sample cooled from 25 °C to 0 °C at 1 °C/min, and imaged in cross-polarization microscopy mode
- Outwaxing is the point at which at which the viscosity increases rapidly on cooling
- Marked by the appearance of crystalline domains (wax precipitates) in the sample

https://www.tainstruments.com/use-of-thermal-analysis-and-rheometry-to-study-waxation-in-crude-oil/

Dielectric Accessory

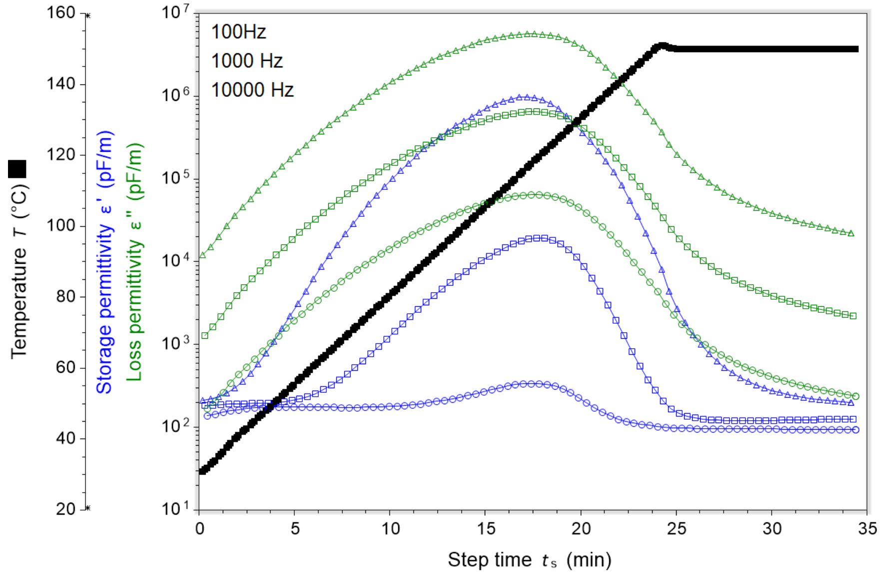
Agilent E4980A LCR meter

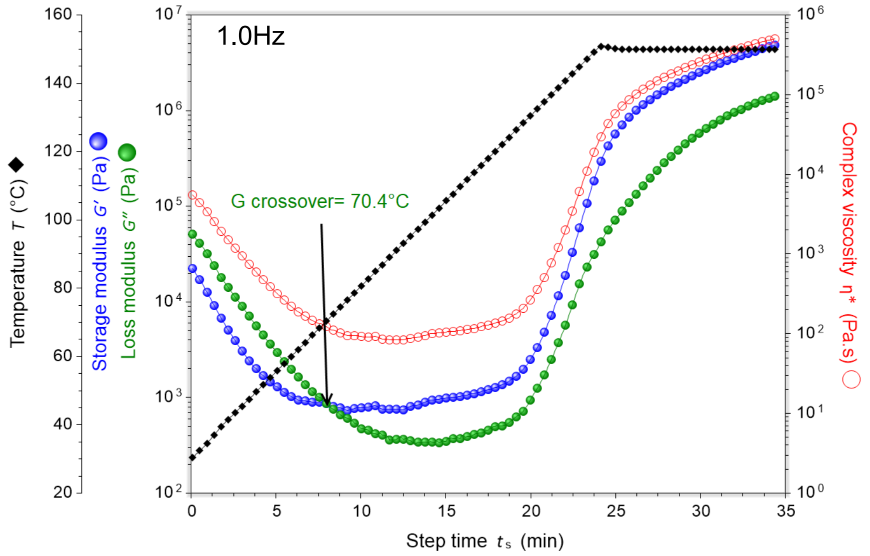
Grounded Geometries with Ceramic Insulator (standard or disposable)

Simultaneous Rheology-Dielectric Analysis on Thermoset

- Dielectric:
  - Permittivity (ε'; ε"; ε*)
  - Loss tangent
  - Capacitance
  - Conductance

- Rheology:
  - Modulus (G'; G"; G*)
  - Tan delta
  - Complex viscosity
  - Stress and strain





T Chen, TA apps note RH104

Other Accessories

Small Angle Light Scattering

Interfacial Accessories

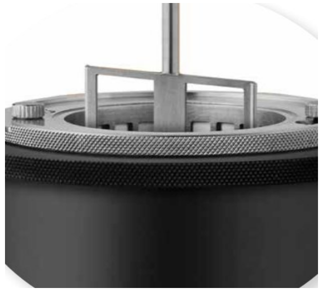
Magneto-Rheology

Electro-Rheology

Pressure Cell

Starch Pasting Cell

Immobilization Cell



Building material cell

Thank You!

Where to Find Help

On your desktop

HR-DHR Manuals

TA Instruments
HR/DHR Manuals

To view the desired manual using Acrobat Reader, click the name in the list below:

TA Manual Supplement
(Contains important information applicable to all manuals.)

Instrument Documentation

HR/DHR Series Getting Started Guide - UPDATED
AR-G2/AR2000ex/AR1500ex Rheometer Getting Started Guide

Accessory Documentation

Air Chiller System (ACS) Getting Started Guide
Asphalt Submersion Cell Getting Started Guide
Dielectric Accessory Getting Started Guide
Electrically Heated Cylinder (EHC) Getting Started Guide
Electrically Heated Plate (EHP) Getting Started Guide
Electrorheological Accessory Getting Started Guide
Environmental Testing Chamber (ETC) Getting Started Guide
Gas Cooling Accessory Getting Started Guide
High Sensitivity Pressure Cell Getting Started Guide
Immobilization Cell Getting Started Guide
Interfacial Subphase Exchange Cell Getting Started Guide
MagnetoRheology Getting Started Guide
Modular Microscopy Accessory Getting Started Guide
Optics Plate Accessory Getting Started Guide
Peltier Plate Concentric Cylinder Getting Started Guide
Peltier Plate Getting Started Guide

ARES-G2 Manuals

TA Instruments
ARES-G2 Manuals

To view the desired manual using Acrobat Reader, click the name in the list below:

TA Manual Supplement
(Contains important information applicable to all manuals.)

Instrument Documentation

ARES-G2 Getting Started Guide

Accessory Documentation

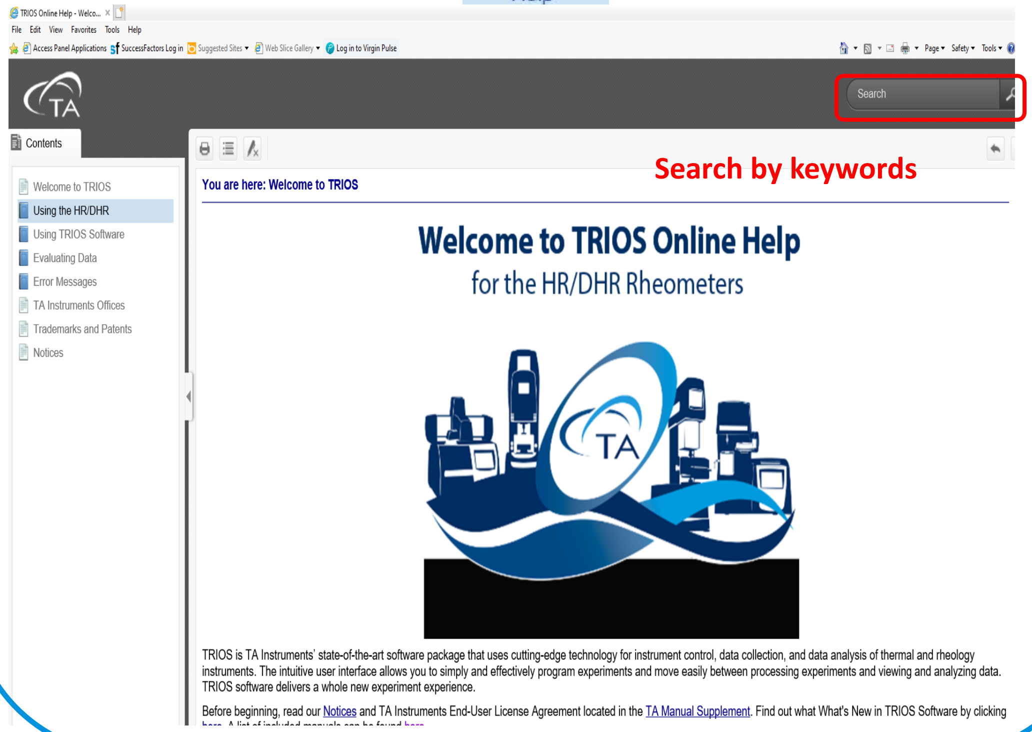
Air Chiller System (ACS) Getting Started Guide - UPDATED!
Advanced Peltier System (APS) Getting Started Guide
Chiller Panel Kit Installation Instructions
Dielectric Accessory Getting Started Guide
Electrorheological (ER) Accessory Getting Started Guide
FCO Camera Kit Installation Guide
High Sensitivity Pressure Cell Getting Started Guide - NEW!
Interfacial Double Wall Ring (DWR) Getting Started Guide
LN2 Kit Installation Guide
Partitioned Plate Getting Started Guide
Peltier Plate Kit Installation Instructions
Sealed Fluid Bath Kit Installation Guide
Sealed Fluid Bath Kit Upgrade Kit
UV Curing Accessory Getting Started Guide

Software Documentation

What's New in TRIOS Software

In TRIOS

Help



Search by keywords

Web Based e-Training Courses

Web based e-Training Courses

TA Instruments offers a variety of training opportunities via the Internet. e-Training opportunities include the following:

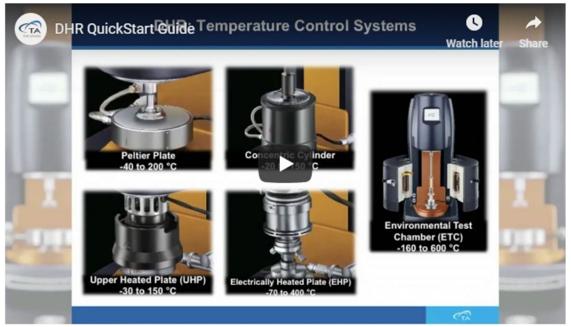
QUICKSTART e-TRAINING COURSES

QuickStart e-Training courses are designed to teach a new user how to set up and run samples on their analyzers. These 60-90 minute courses are available whenever you are. These pre-recorded courses are available to anyone at no charge. Typically these courses should be attended shortly after installation.

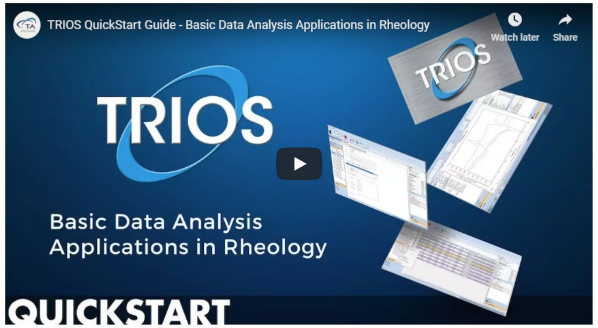
https://www.tainstruments.com/training/e-training-courses/

ARES G2

Discovery HR

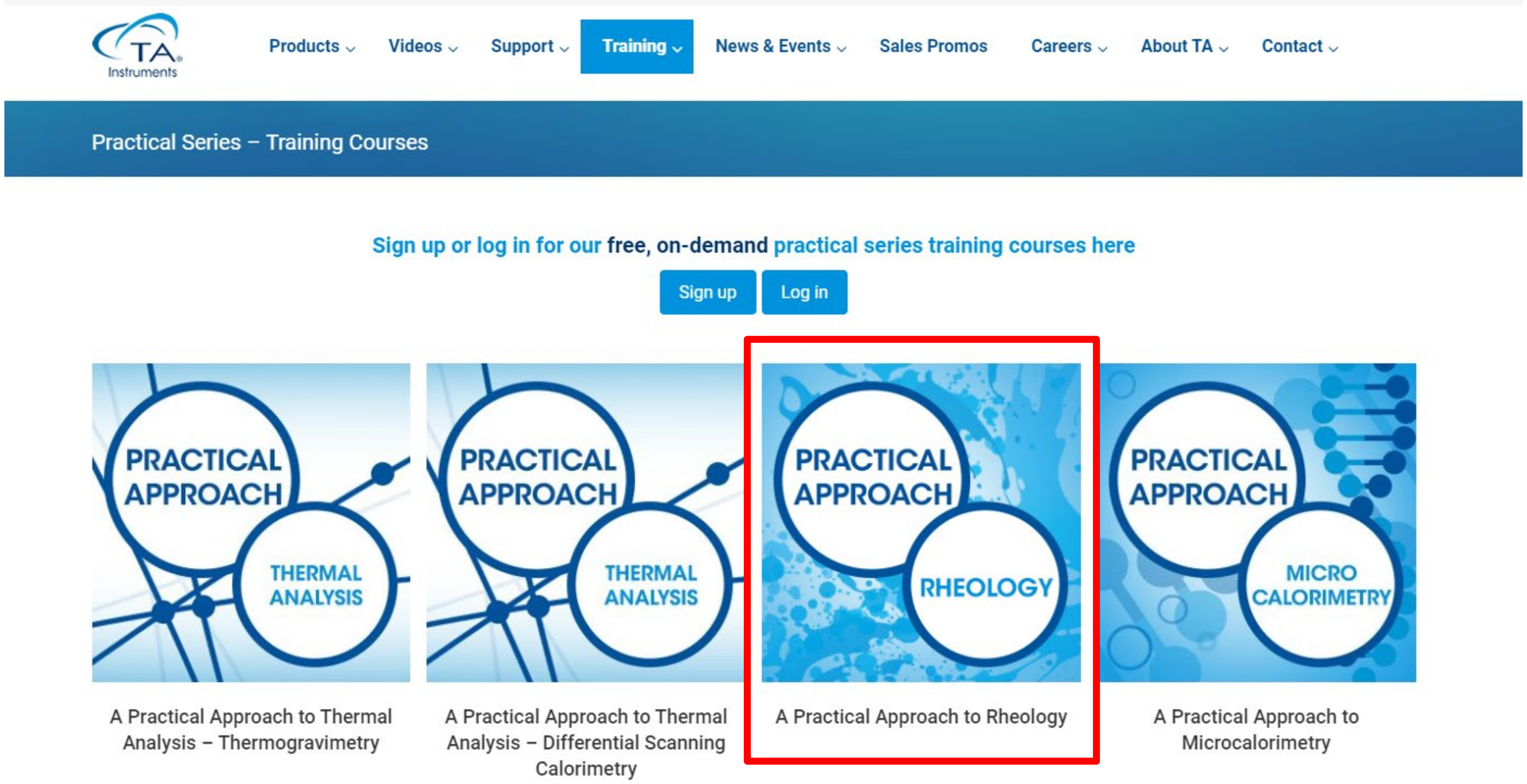


TRIOS Software



Practical Series Training Course

https://www.tainstruments.com/practical-series-training-courses/



TA Webinars - Rheology

https://www.tainstruments.com/support/webinars/

View all | Electroforce | Dilatometry | Microcalorimetry | Rheology | Rubber | Thermal Analysis

Interfacial Rheology: Fundamental Overview and Applications

Designing New Materials for Additive Manufacturing: Vat Photopolymerization

Strategies for Rheological Evaluation of Adhesives

An Introduction To High Pressure Rheology

Randy H. Ewoldt: Experimental Challenges of Shear Rheology, How to Avoid Bad Data

Norman J. Wagner: An Introduction to Colloidal Suspension Rheology

Professor João Maia: The Role of Interfacial Elasticity on the Rheological Behavior of Polymer Blends

Neil Cunningham: Essential tools for the new Rheologist

Extensional Rheology in Polymer Processing

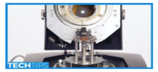
An Introduction to Tribo-Rheometry: Quantifying Friction

Rheo-Microscopy: Bridging Rheology, Microstructure & Dynamics

Extensional Rheology & Analytics of Material Characterization

Tech Tips

Installation & Calibration of the Relative Humidity Accessory for the Discovery Hybrid Rheometer



Shear Sandwich Clamp Installation & Calibration for the Discovery DMA 850

Three Point Bend Clamp Installation & Calibration for the DMA850

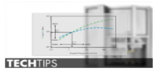
Installation and Calibration for the UV Accessory on the Ares G2 Rheometer

Single Cantilever Installation & Calibration – DMA 850

Dual Cantilever Installation & Calibration – DMA 850

Linear Film Tension Clamp for DMA using the ARES-G2

Loading the Powder Clamp on the Q800 DMA with 35mm Dual Cantilever Clamp



Frequency Sweep Tests for RPA Flex and RPA Elite

Improving Structured Fluid Measurements w/ Pre-Shearing

Measuring Thixotropy Of A Sample- TA TechTips

The Double Wall Ring & Interfacial Measurements – TA

site

Applications Notes Library

Applications Notes Library

Our instruments are used in a variety of products, in multiple industries. The application notes below provide more detail on specific potential applications. You can search for specific app notes with the search field.

rheology

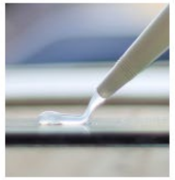
261 item

Title	Product Category	Ref#	Link
Hot Melt Adhesives	Rheology	AAN001	Download Note
Generating Mastercurves	Rheology	AAN005e	Download Note
Analytical Rheology	Rheology	AAN006e	Download Note
Normal Stresses in Shear Flow	Rheology	AAN007e	Download Note
Mischungsregein Komplexer Polyersysteme	Rheology	AAN008d	Download Note
Mixing Rules for Complex Polymer Systems	Rheology	AAN008e	Download Note
Application of Rheology of Polymers	Rheology	AAN009	Download Note
Synergy of the Combined Application of Thermal Analysis and Rheology Monitoring and Characterizing Changing Processes in Materials	Rheology	AAN010e	Download Note

Seminar Series: Instant Insights

Seminars:

- Thermal Analysis and Rheology
- Medical Device and Biomaterials Testing
- Elastomers and Rubber Compounds
- TRIOS AutoPilot & TRIOS Guardian



Thermal, Rheological and Mechanical Characterizations of Thermoset

Tianhong (Terri) Chen, Ph.D.

Thermosetting materials, such as epoxy, have been widely applied in many areas including automotive, aerospace and electronics industries in the form of surface coating, structural adhesives, advanced composites and packaging materials.

View Archive

Advancements in the Characterization of Pharmaceuticals by DSC

Jason Saienga, Ph.D.

Differential Scanning Calorimetry is a simple, yet powerful technique to gain a broad understanding of the characteristics of pharmaceutical materials, from the crystalline structure that exists to the compatibility of a specific formulation.

View Archive

Steady State & Flash Methods for Thermal Diffusivity and Thermal Conductivity Determination

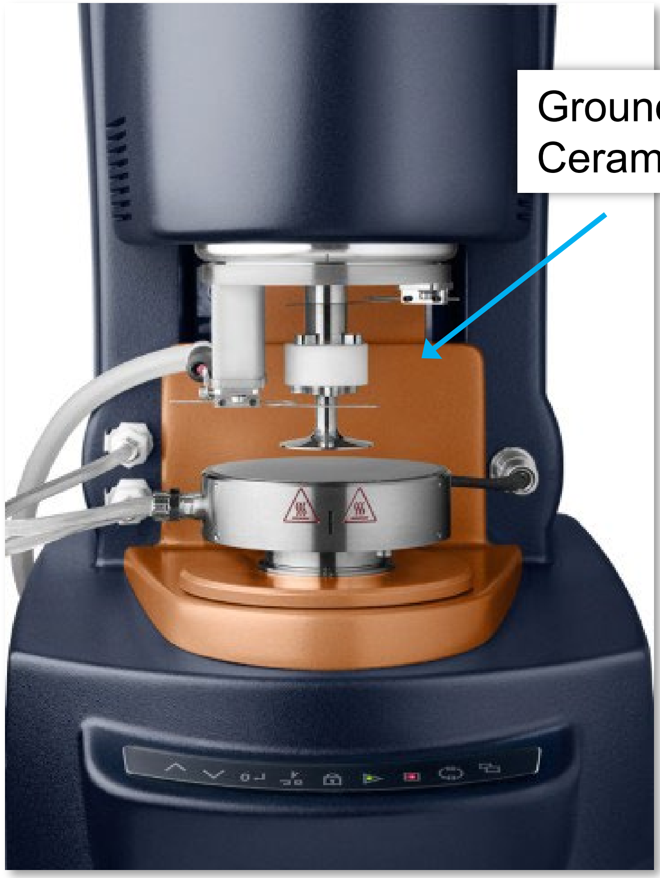
Justin Wynn

In this presentation we will demonstrate accurate and high-throughput methods to measure the critical heat transfer properties of thermal diffusivity and thermal conductivity.

View Archive

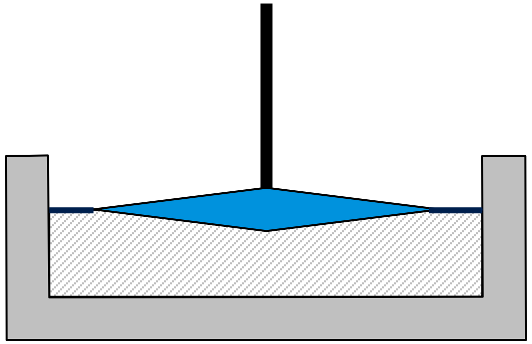
TA

Electro-Rheology (ER) Accessory



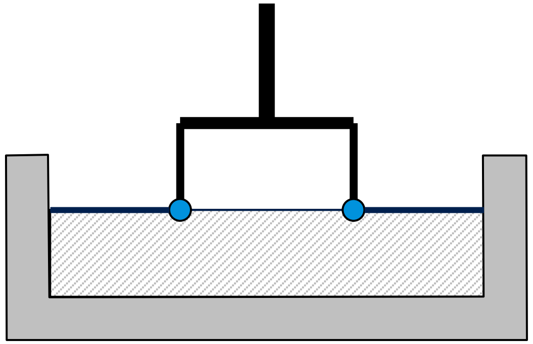
Grounded Geometries with Ceramic Insulator

DHR Interfacial Accessories



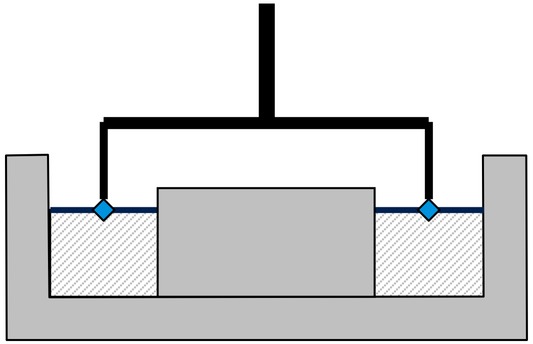
Bicone

Steady Shear Viscosity at air/liquid and liquid/liquid interface.



DuNouy Ring

Qualitative Viscoelastic measurements at air/liquid and liquid/liquid interface.

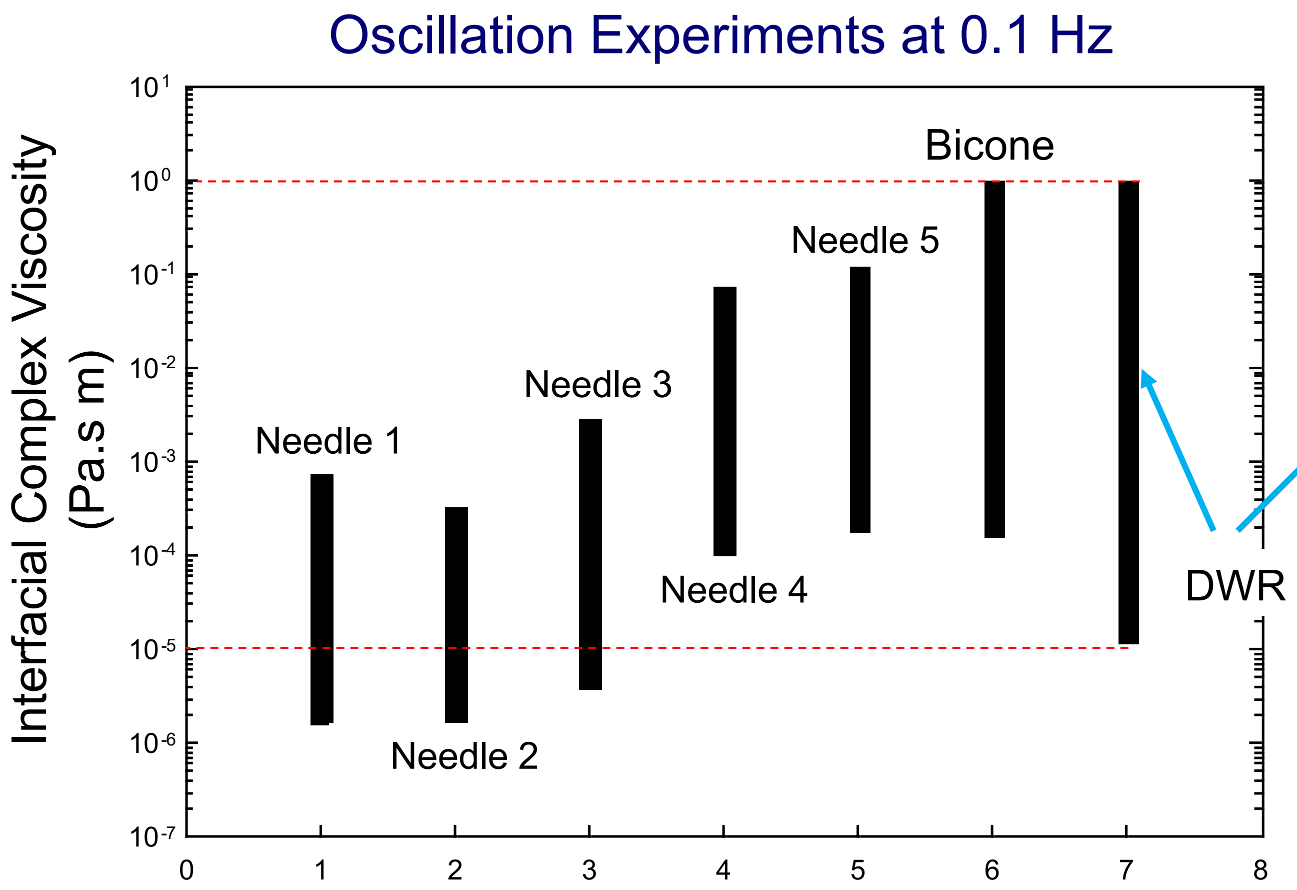


Double Wall Ring

Quantitative Viscoelastic measurements at air/liquid and liquid/liquid interface.

- Interfacial shear rheology of thin layers at liquid-liquid or liquid-gas interfaces
- Effect of particles, surfactants or proteins at the interface
- Applications: food, biomedical, enhanced oil recovery

Patented DWR Interfacial System



Interfacial Exchange Cell

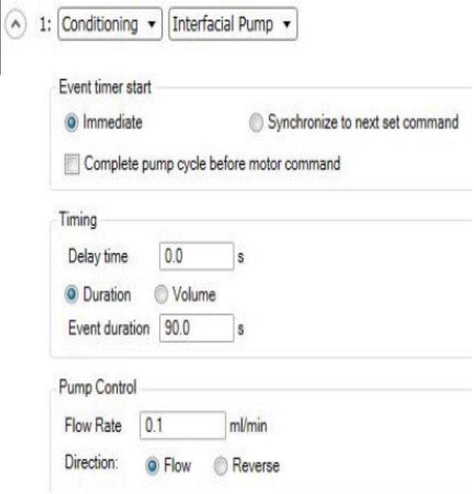
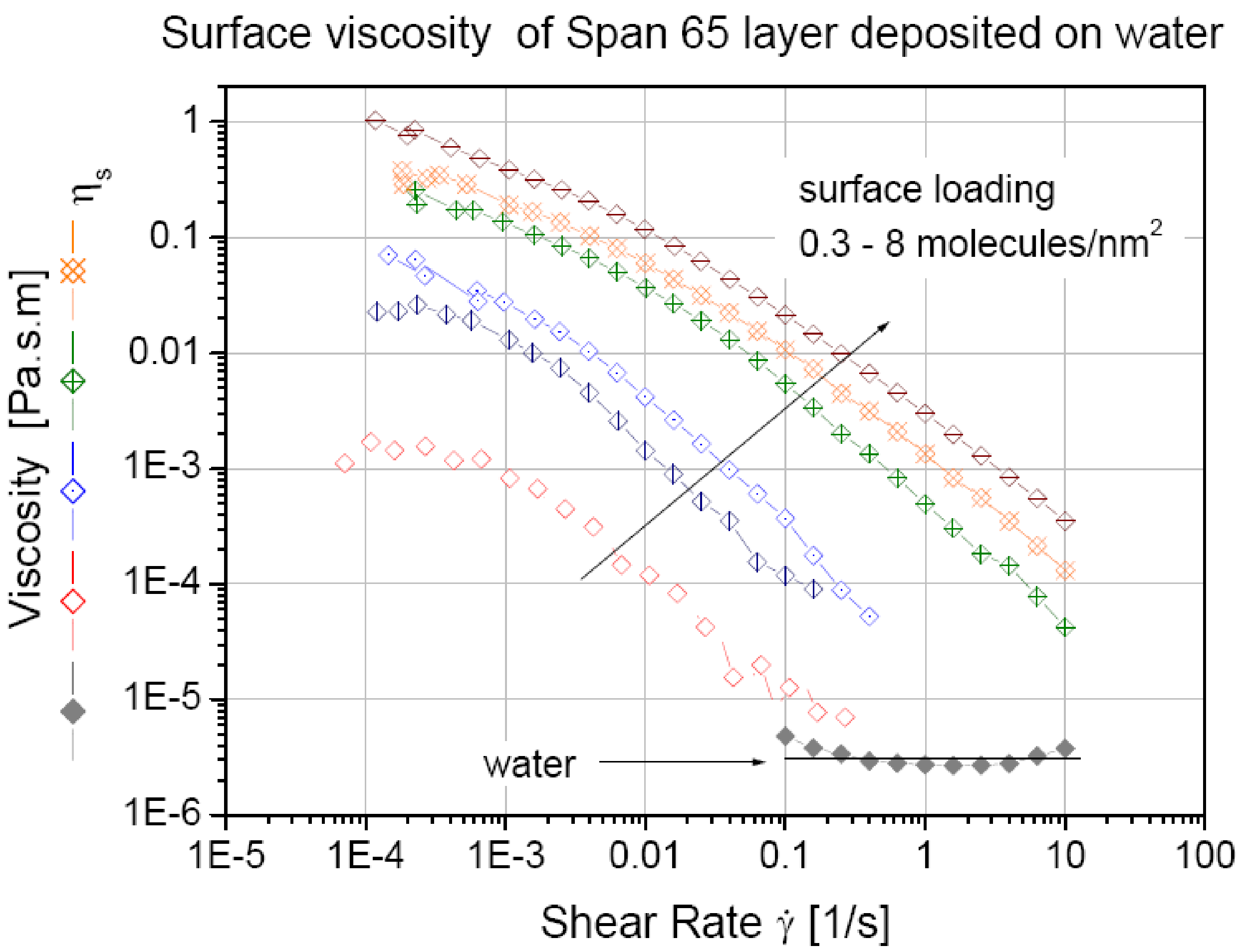


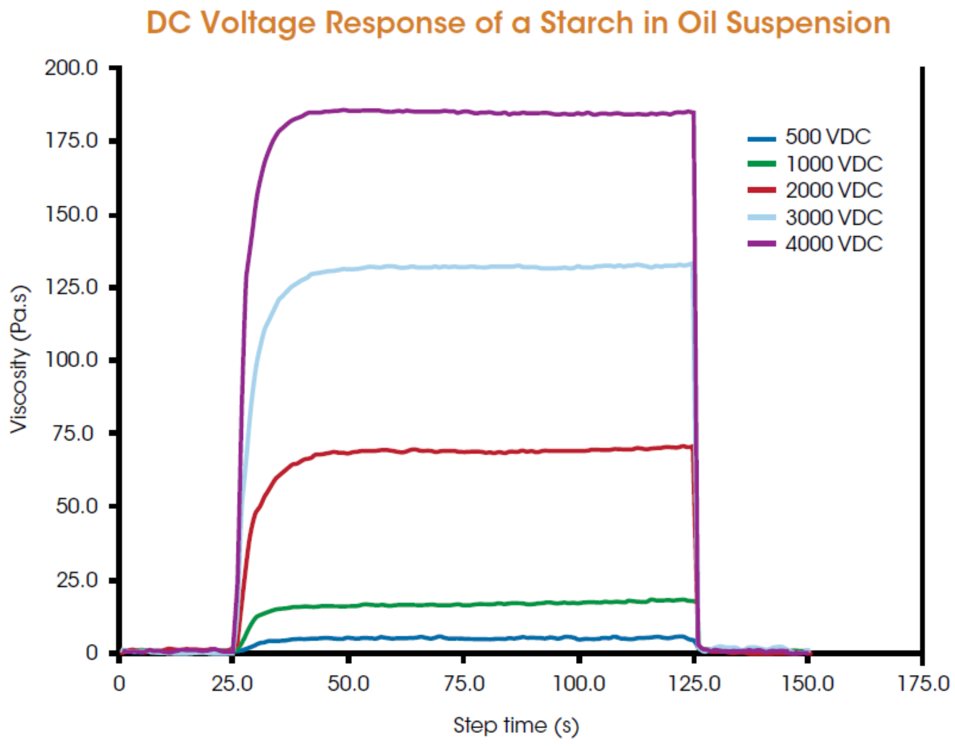
Table 1: Interfacial Exchange Cell Specifications

Subphase Volume	37.7 mL
Recommended Infusion Rate	6 mL/min
Trough to Tee Fitting Tube Length	280 mm
Maximum Operating Temperature	100°C

Surface Concentration Effects on Interfacial Viscosity



Electro-Rheology (ER) Accessory



- Plates and DIN concentric cylinders
- Fully programmable from TRIOS
- Wide range of voltage profiles
  - Constant voltage
  - Step voltage, ramp voltage
  - Sine wave voltage function
  - Triangle wave voltage function
  - Wave functions with DC offsets

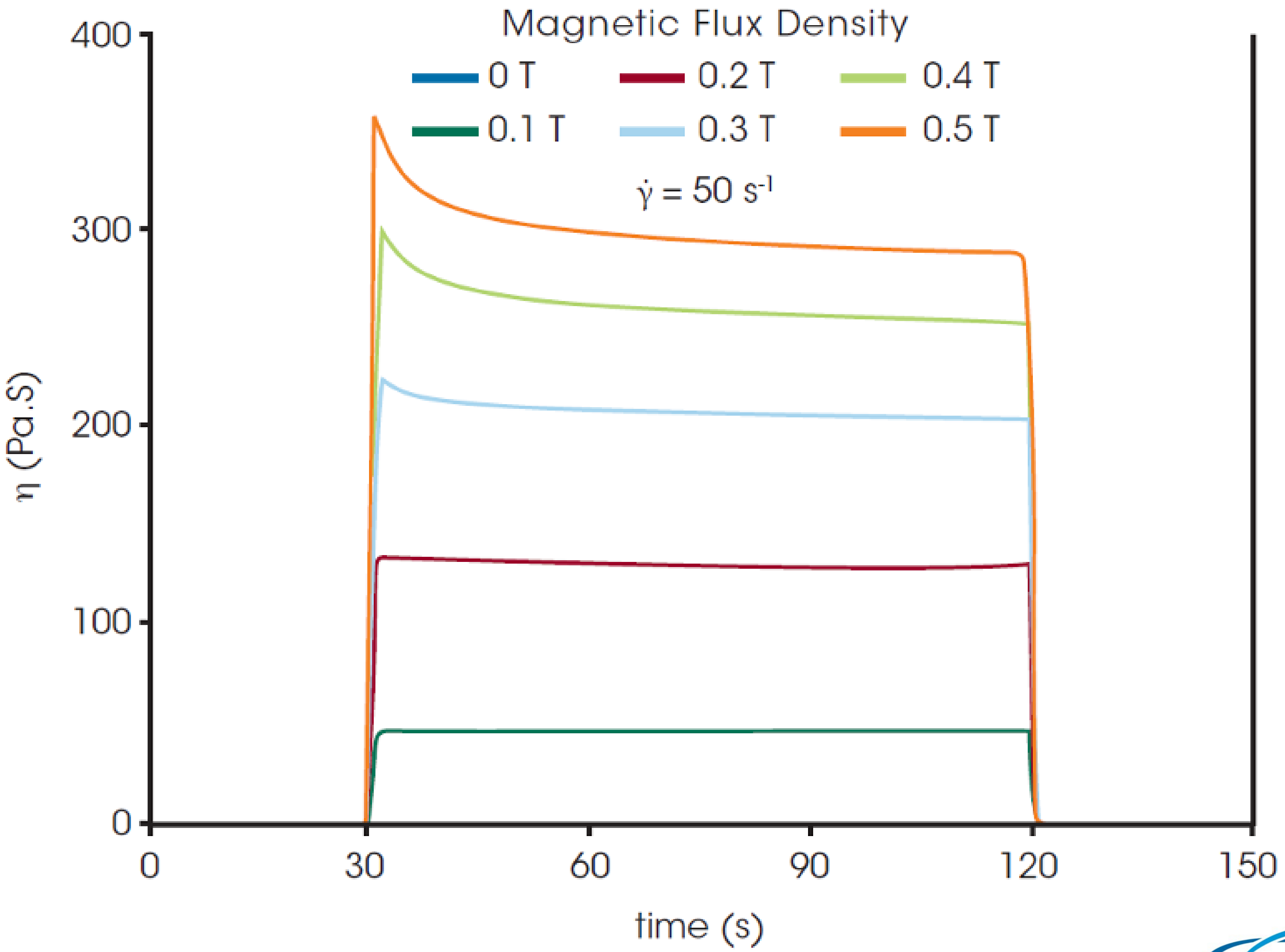
Applications:

- Hydraulic valves and clutches
- Shock absorbers
- Bulletproof vests
- Polishing slurries
- Flexible electronics

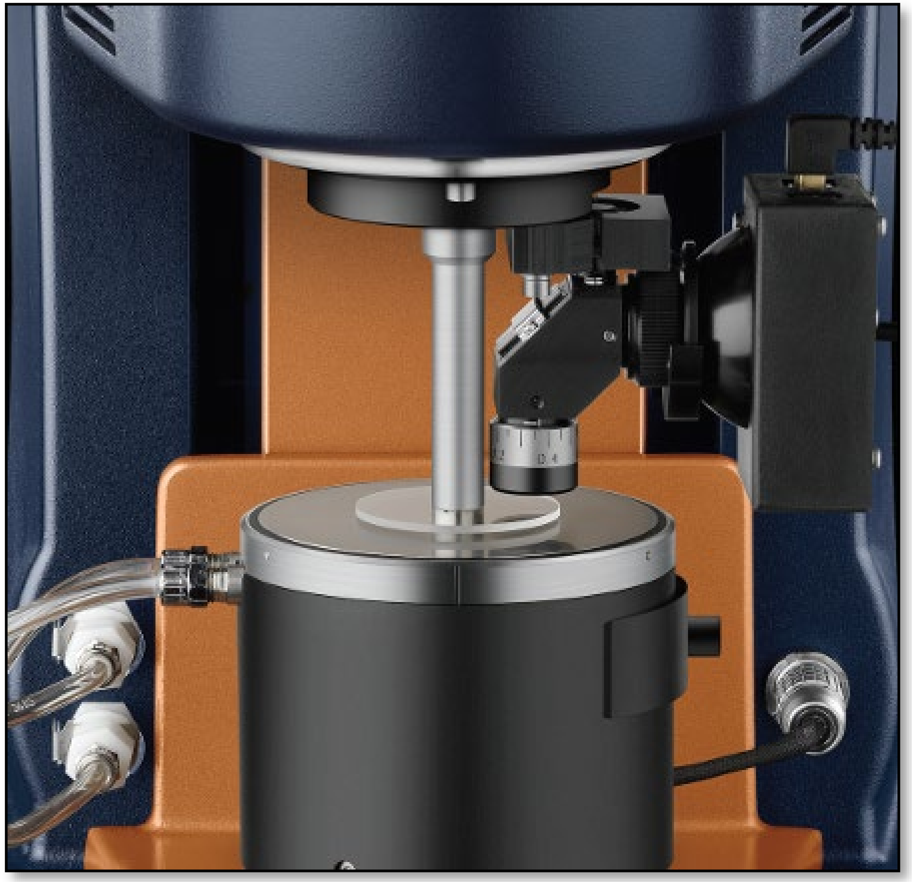
Magneto-Rheology (MR) Accessory

- MR fluids are “smart materials” that respond to an applied magnetic field
- Rheological properties of these materials can be easily tuned by controlling the strength of the field
- Can apply magnetic field up to 1T, over temperature range of -10 to 170 °C
- Exclusive closed loop control of magnetic field strength for increased accuracy
- Complete control of magnetic field profile in all standard rheological test modes:
  - Constant field
  - Step, ramp field
  - Sine wave
  - Triangle wave
  - Wave functions with field offsets

Magneto-Rheology (MR) Accessory

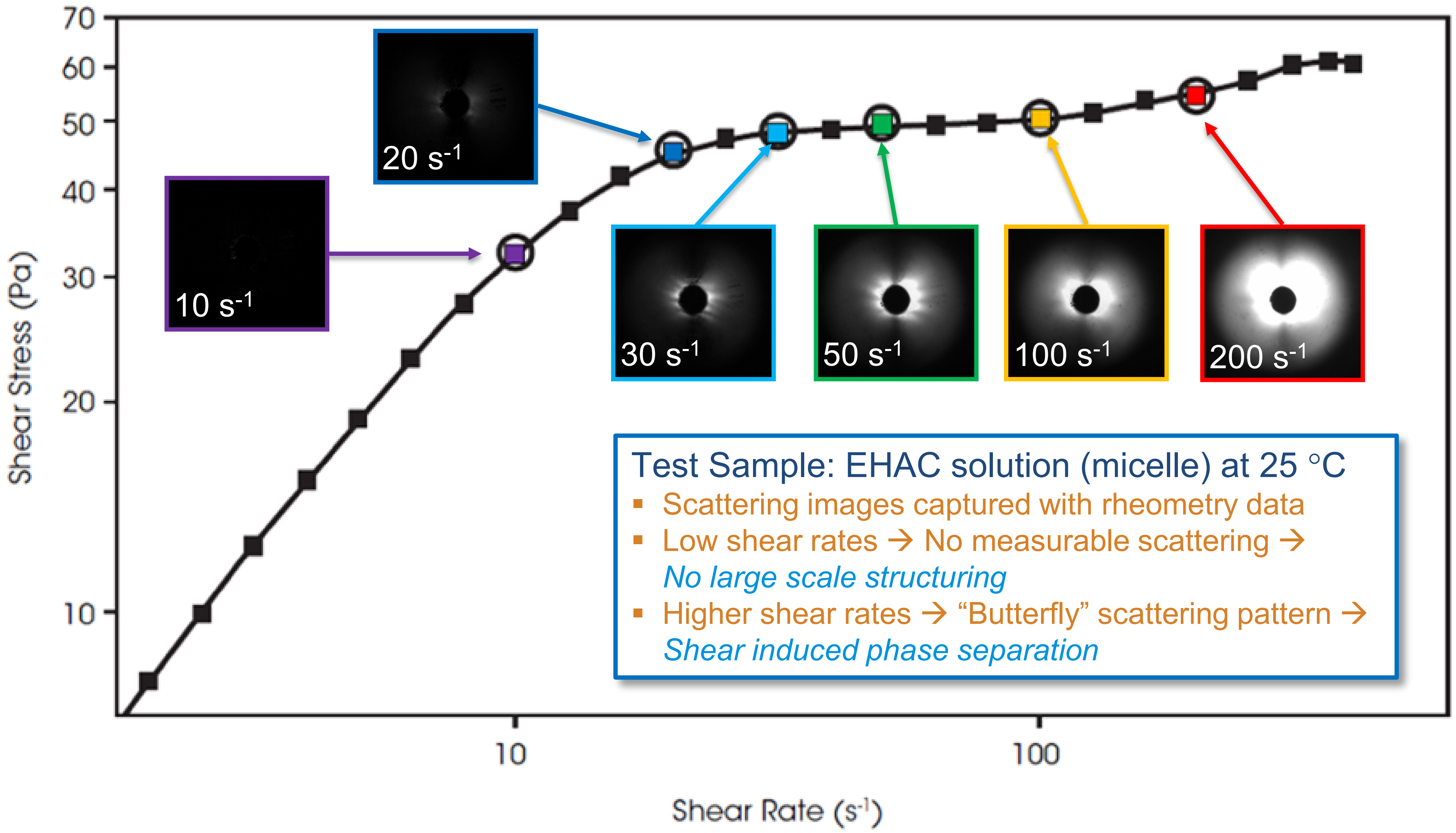


Small Angle Light Scattering (SALS)



- Provides the capability to simultaneously collect rheological and structural information
- SALS can reveal information about particle size, shape, orientation, and spatial distribution
- Patented Peltier plate temperature control from 5 to 95 °C
- Laser light (635 nm wavelength) passes through a 50 mm quartz geometry
- CCD Camera collects images that are stored with rheology data
- q vector range: 1.38 μm^{-1} to 6.11 μm^{-1} (length scale range: 1.0 μm to ~ 4.6 μm)

SALS Application: Shear Induced Phase Separation



Amorphous, Crystalline and Crosslinked Polymers