Section I: Rheology (& DMA) Theory, Instrumentation, and basic methods

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Rheology: An Introduction




Rheology is the science of flow and deformation of matter (primarily fluids)

Dynamic mechanical analysis is the science of flow and deformation of matter (primarily solids)

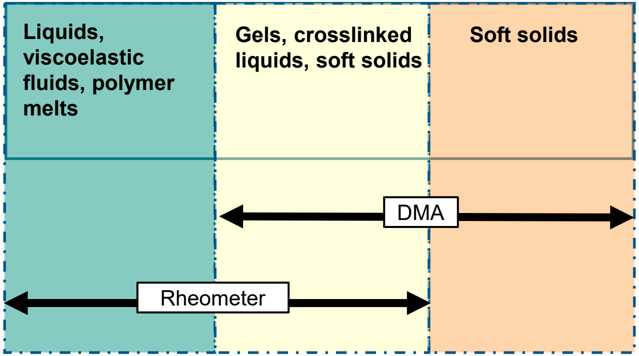


What does a Rheometer (or DMA) measure?

- A <u>rheometer</u> 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 a property of a material that exhibits both viscous and elastic character. Measurements of G', G", tan δ with respect to time, temperature, frequency and stress/strain are important for characterization
- Samples – liquids, semi-solids and soft solids

- A <u>DMA</u> can provide information about the material's:
  - **Viscoelasticity** - Measurements of G', G", tan δ with respect to time, temperature, frequency and stress/strain are important for characterization
- Samples – solids, composites, elastomers, soft solids

- Rheology and DMA are complementary
- <u>DHR Rheometers can do both</u>



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What Rheology (or DMA) Measures

- **Viscosity (Liquids)**

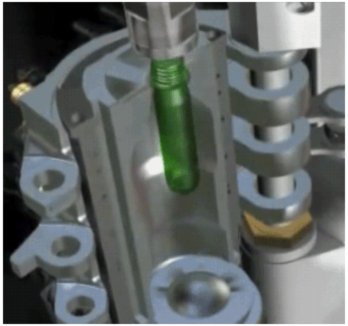
- **Elasticity (Solids)**

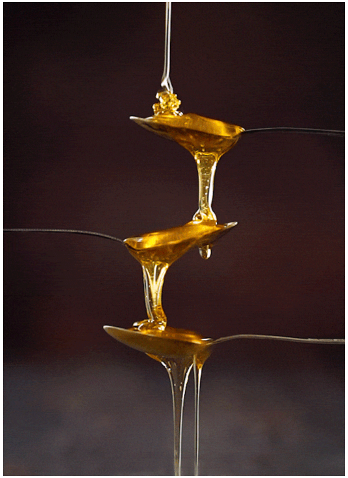
- **Viscoelasticity (Liquids to Solids)**

3

Why Rheology Matters

- What is Viscosity? – Resistance to flow
- Why do we care? – The various fluids we use day to day have to be processed (pumped, poured, mixed, etc.) whether it is water, cake batter, molten polymer, oil, etc.
- Viscosity can be used as a measure of shelf stability, molecular weight, and energy to process
- Rheology can give us viscosity data and much more

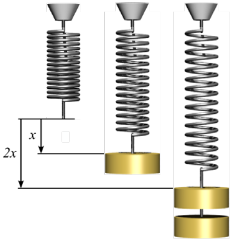


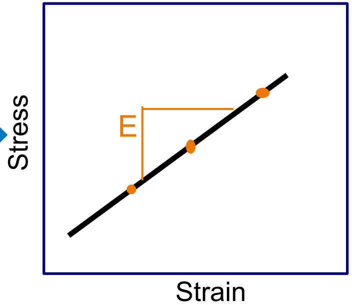


Basic Model of Elasticity

What is Elasticity?

- In 1660, Robert Hooke developed his “True Theory of Elasticity”
  - Model – spring
  - Observations – stress is linearly proportional to the deformation
  - Young’s Modulus is the slope of the stress and strain curve





$$\sigma = E\gamma$$

Hooke’s Law of Elasticity

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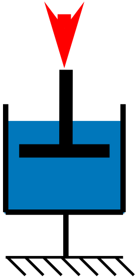
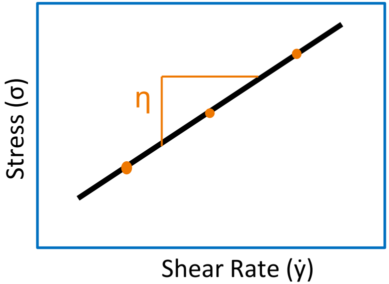
Basic Model of Viscosity

- In 1687, Isaac Newton studied the flow behavior of liquids
  - Model – dashpot
  - Observations – stress is linearly proportional to shear rate
  - Viscosity is – the ratio of the stress and rate curve





$$\sigma = \eta \dot{\gamma}$$

Newton's Law of Viscosity

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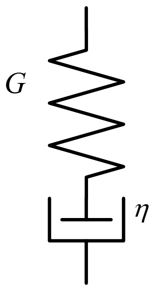
How to Describe Viscoelasticity?

- Viscoelastic Materials: Force depends on both deformation and rate of deformation and vice versa

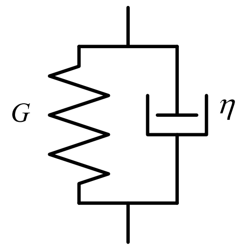
Maxwell Model (Stress Relaxation)



$$\gamma = \frac{\sigma}{G} + \frac{\sigma}{\eta} t$$

$$\frac{d\gamma}{dt} = \frac{1}{G}\frac{d\sigma}{dt} + \frac{\sigma}{\eta}$$

Kelvin-Voigt Model (Creep)



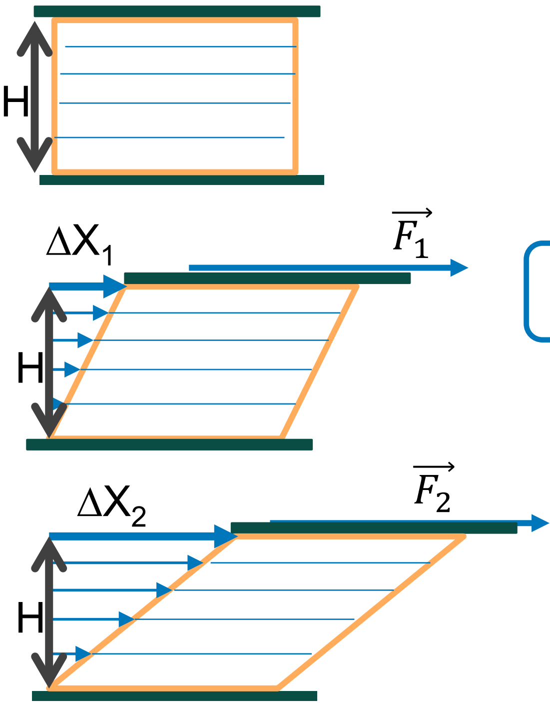
$$\sigma = G\gamma + \eta\dot{\gamma}$$

$$\sigma = G\gamma + \eta\frac{d\gamma}{dt}$$

How do Rheometers Work?

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

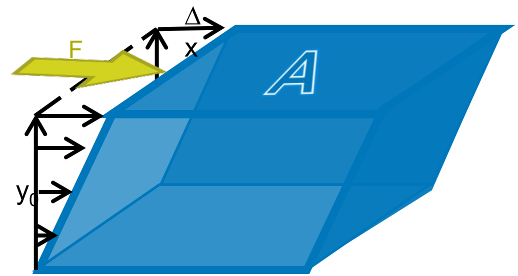
*Sliding Plates



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

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

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



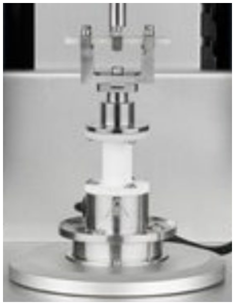
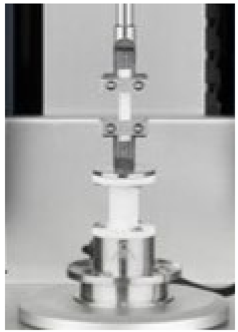
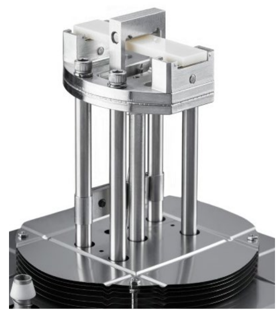
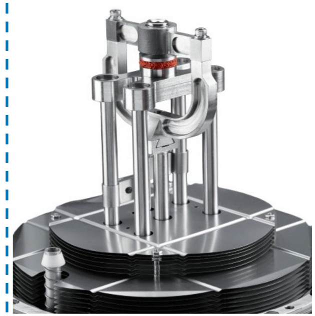
$$\frac{\text{Stress}}{\text{Strain}} = \text{Modulus}$$

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

- In practice rotational rheometers use angular displacement and angular strain
- This allows for high shear rates and infinite angular displacement

Variety of DMA clamps

- Cantilever, Tension, Three-point bend, compression

	S/D Cantilever	Film/Fiber Tension	3-Point Bending	Compression
DHR:				
DMA:				

- In DMA, we characterize the relationship between stress and strain with various geometries

How do DMAs Work?

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

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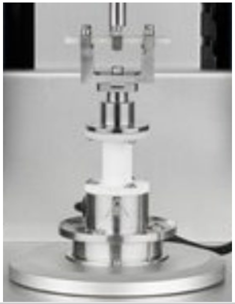
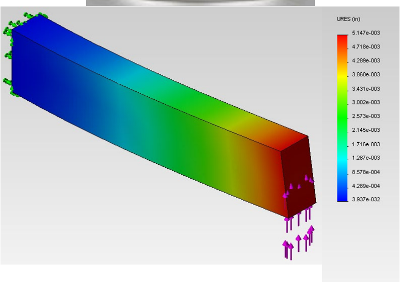
S/D Cantilever



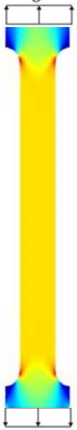


$$\sigma_x = \frac{3PL}{wt^2}$$

$$\varepsilon_x = \frac{3\delta t F_c}{L^2\left[1 + \frac{12}{5}(1+\nu)\left(\frac{t}{L}\right)^2\right]}$$

- Since we are testing solids here, we do not measure viscosity

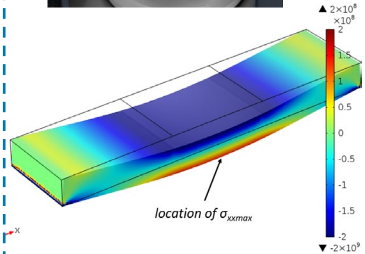
Film/Fiber Tension



$$\sigma_0 = \frac{P}{A_0}$$

$$\epsilon_0 = \frac{\Delta L}{L_0}$$

3-Point Bending



$$\sigma_x = \frac{3PL}{wt^2}$$

$$\varepsilon_x = \frac{6\delta t F_c}{L^2\left[1 + \frac{12}{5}(1+\nu)\left(\frac{t}{L}\right)^2\right]}$$

Compression

Torsion (Shear)

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

How do DMAs Work?

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

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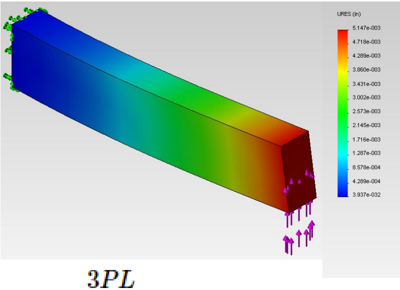
S/D Cantilever



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Film/Fiber Tension

$$\sigma_0 = \frac{P}{A_0}$$

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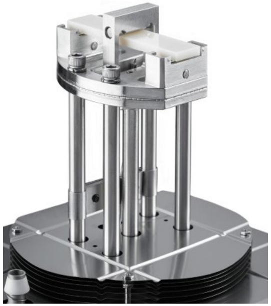
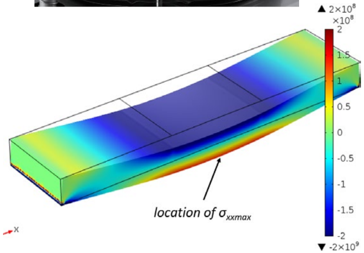
3-Point Bending





$$\sigma_x = \frac{3PL}{wt^2}$$

$$\varepsilon_x = \frac{6\delta t F_c}{L^2\left[1 + \frac{12}{5}\left(1+\nu\right)\left(\frac{t}{L}\right)^2\right]}$$

Compression

Shear Sandwich

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

Instrumentation

How do rotational Rheometers 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:

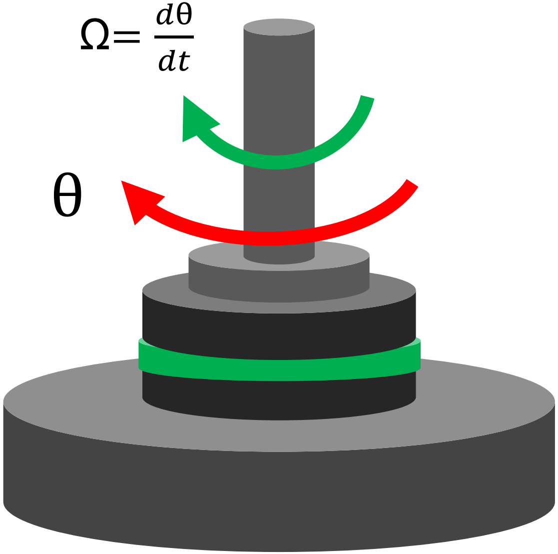
1. Torque (Force)
2. Angular Displacement
3. Angular Velocity

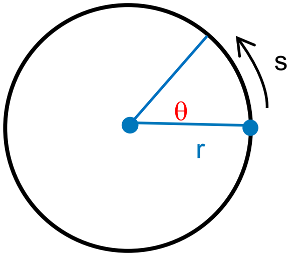
Measured parameter: angular displacement

- 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

$$\Omega = \frac{d\theta}{dt}$$



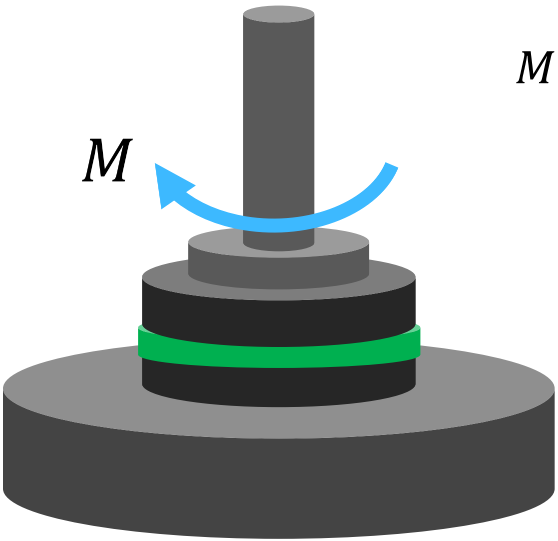


$$\theta = s/r$$

$$\Omega = \frac{d\theta}{dt} = \frac{1}{r}\frac{ds}{dt}$$

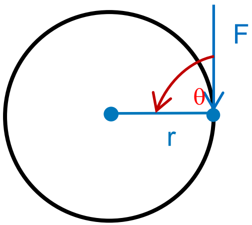
Measured parameter: torque

- 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



$$M = r \cdot F \cdot \sin\theta = \vec{r} \cdot \vec{F}$$

(for θ = 90° as shown)



Equation for modulus

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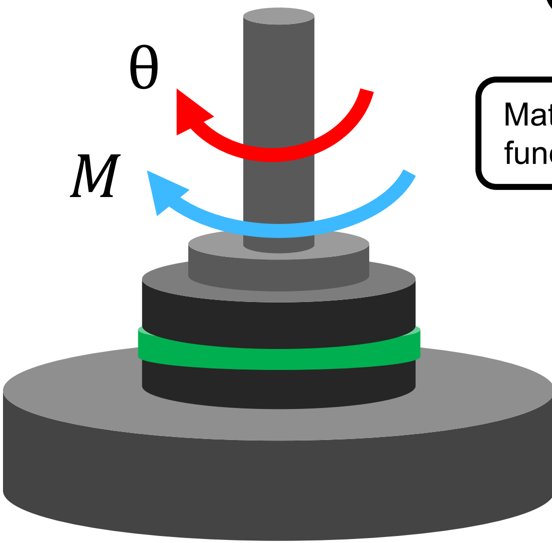


$$G = \frac{\sigma}{\gamma} = \frac{M \cdot K_\sigma}{\theta \cdot K_\gamma}$$

Material function

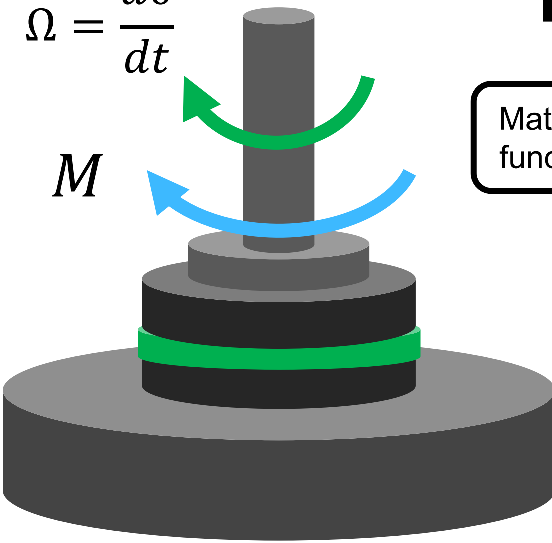
Constitutive equation

Measured signals

Geometry constants

Equation for viscosity

$$\Omega = \frac{d\theta}{dt}$$



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

Material function

Constitutive equation

Measured signals

Geometry constants

Geometry Options – Instrument measures Displacement and Torque

Strain and stress are calculated from geometry parameters

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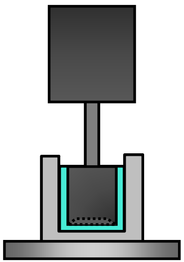
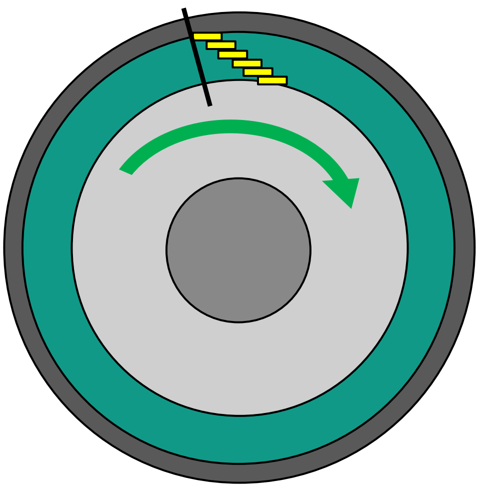
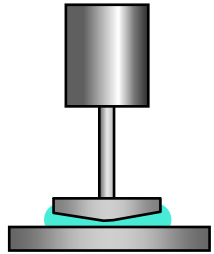
Concentric Cylinders

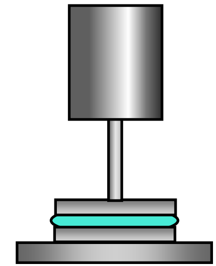


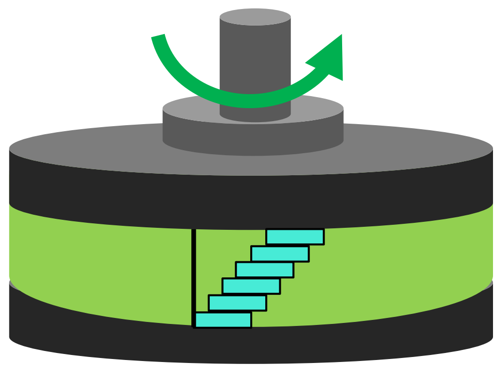


Cone and Plate



Parallel Plate





Geometry Options – Instrument measures Displacement and Torque

Strain and stress are calculated from geometry parameters

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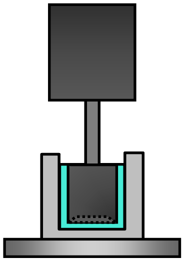
Concentric Cylinders



Strain Constant

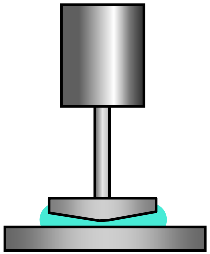
$$K_\gamma = \frac{(1+\delta^2)}{(\delta^2-1)}$$

Stress Constant

$$K_\sigma = \frac{(1+\delta^2)}{(4\pi L \times R_b^2 \times cL \times \delta^2)}$$

$$\delta = \frac{R_C}{R_B}$$

Cone and Plate



Strain Constant

$$K_\gamma = \frac{1}{\beta}$$

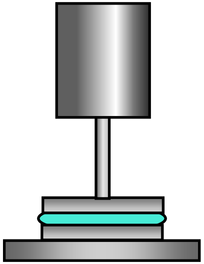
Stress Constant

$$K_\sigma = \frac{3}{2\pi R^3}$$

Normal Stress Constant

$$K_Z = \frac{2}{\pi R^2}$$

Parallel Plate



Strain Constant

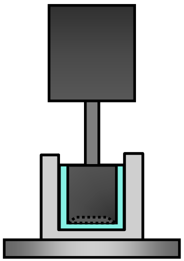
$$K_\gamma = \frac{R}{H}$$

Stress Constant

$$K_\sigma = \frac{3}{2\pi R^3}$$

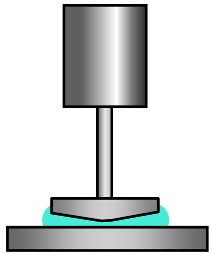
Geometry Options – Testing across a wide range of viscosity

Concentric Cylinders



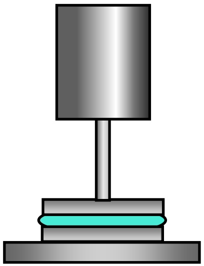
Very Low to Medium Viscosity

Cone and Plate



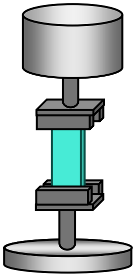
Very Low to High Viscosity

Parallel Plate



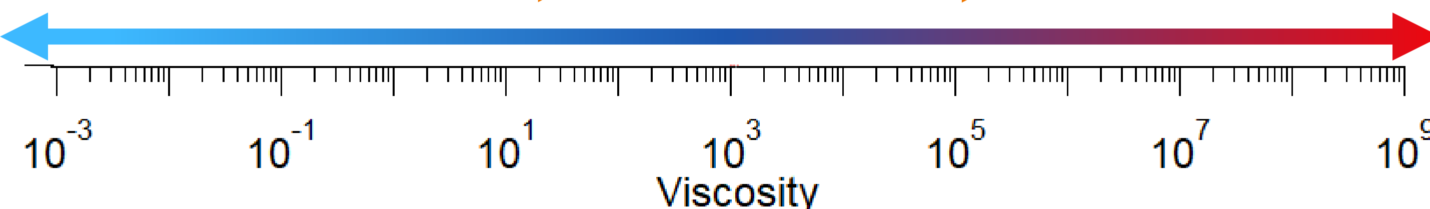
Very Low Viscosity to Soft Solids

Torsion Rectangular



Mid-modulus Solids

Water → to → Steel



Discovery Hybrid Rheometers by TA

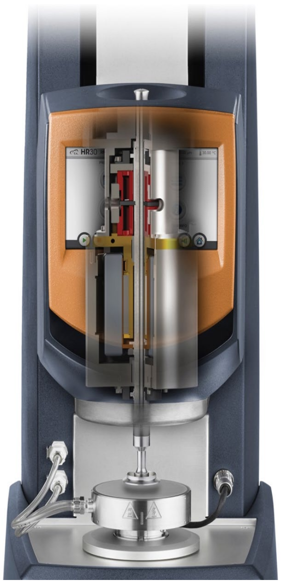
Discovery Hybrid Rheometer

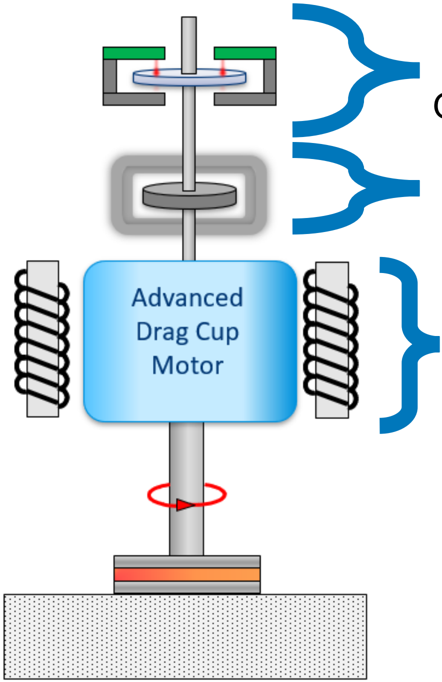
DHR-1,2,3

HR-10,20,30

Controlled Stress Design
Single Head

DHR - *Single head or CMT Combined motor & transducer*





Single or Dual Optical encoder - Measures Displacement (strain) and Velocity (shear rate)

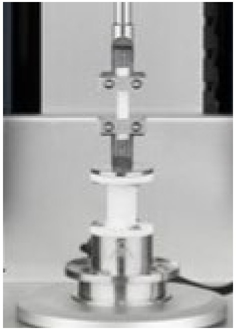
Magnetic Thrust Bearing - Provides both air and magnetic fields to minimize friction

Drag Cup Motor - Measures Torque (Stress)

- This design minimizes external disturbances (friction) to the measurement

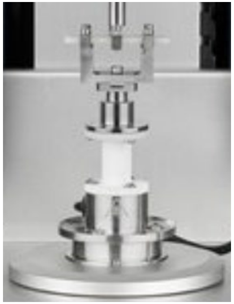
DMA Geometry Options – Testing across a wide range of viscosity

Film/Fiber Tension



- Thin films
- Elastomers
- Fibers

S/D Cantilever



- Supported thermosetting resins
- Elastomers
- Amorphous or lightly-filled thermoplastic materials

3-Point Bending

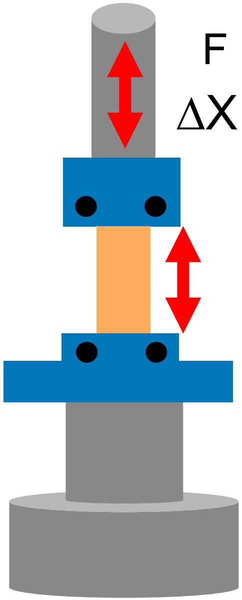
- Metals
- Ceramics
- Highly filled thermosetting polymers
- Highly filled and crystalline thermoplastic polymers

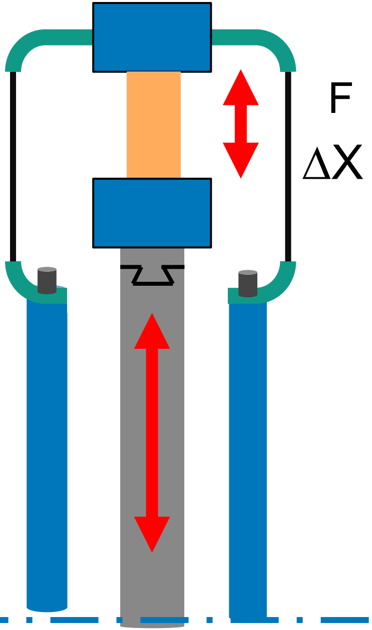
Compression

- Gels
- Weak elastomers

How do DMAs Work?

Film/Fiber Tension





F – force

Δx - displacement

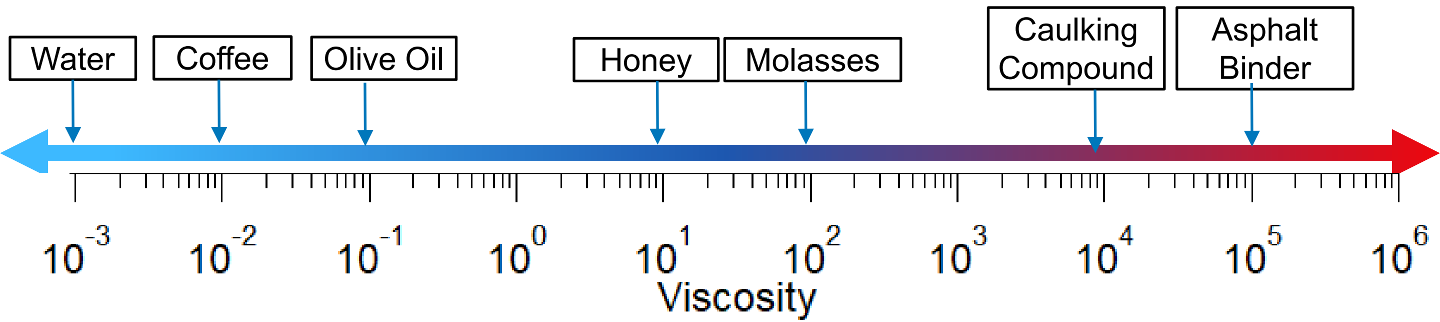
Shear strain $\gamma = \frac{\Delta x}{y_0}$

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

Rheology Theory and Experimental Designs

1. Flow (Steady Shear) Tests

Background on Viscosity Values and Flow Curves

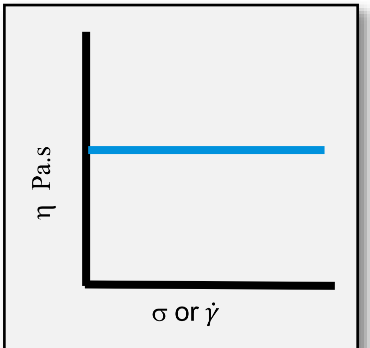


- Different liquid materials in the world can have significantly different viscosities, therefore , they also exhibit different flow behaviors.
- Unless a fluid is Newtonian, it does **not** have **a single viscosity value**!

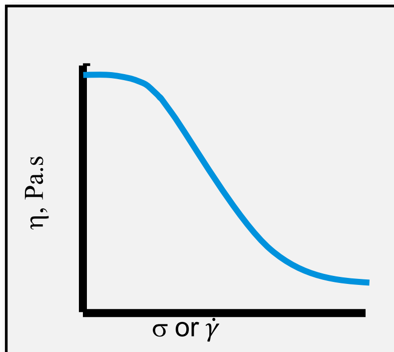
- Newtonian and non-Newtonian

Viscosity Behaviors

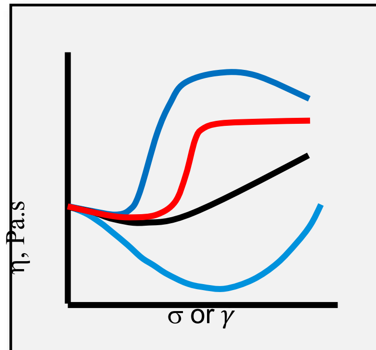
Newtonian



Shear Thinning



Shear Thickening



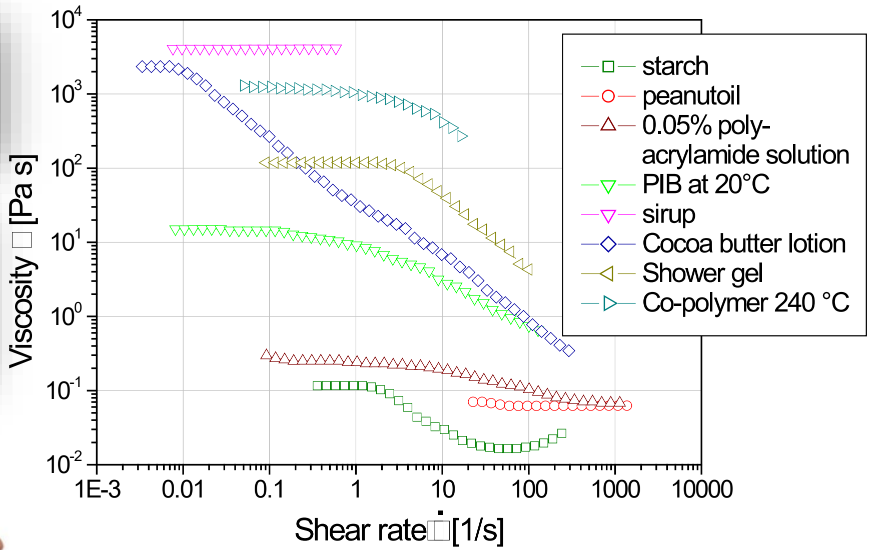







17

Viscosity Curves of Various Fluids



- For non-Newtonian fluids viscosity is a function of shear rate!

Basic Rheological Methods (Steady Shear)

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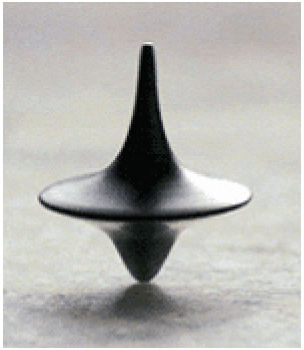
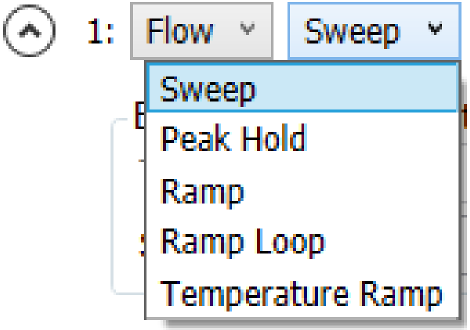
- Common rheological methods for measuring viscosity of liquids
  - Single rate/stress flow
  - Continuous rate/stress ramp
  - Stepped or steady state flow
  - Flow temperature ramp



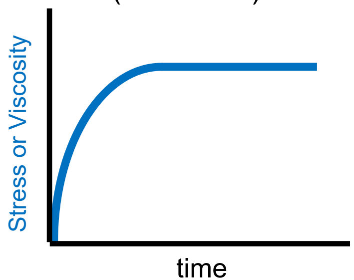


- ❑ Steady Shear –
  - ○ Continuous rotation of upper plate
  - ○ Destroys structure of fluid
  - ○ Provides information about sample when flowing

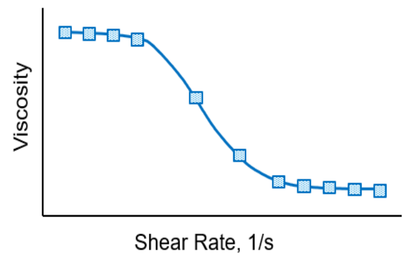
Basic Rheological Methods (Steady Shear)

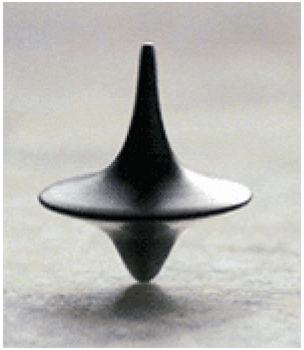
- Common rheological methods for measuring viscosity of liquids

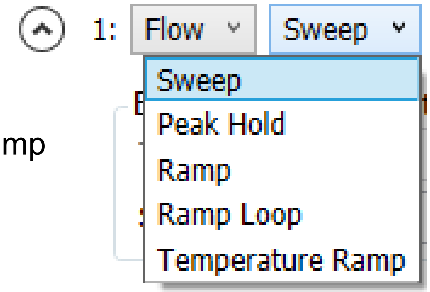
I. Single shear rate steady flow
(Peak hold)



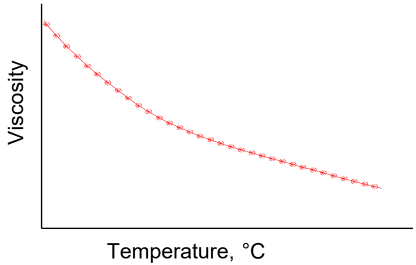
II. Stepped or steady state flow
(Sweep)



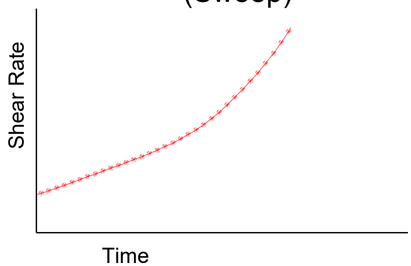




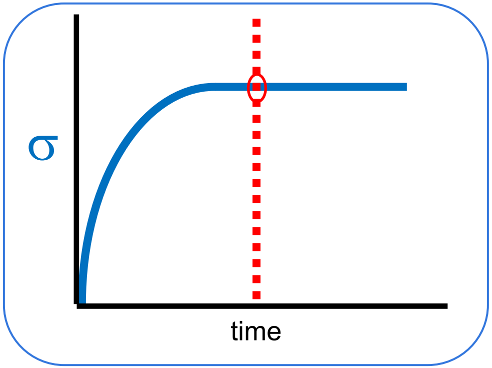
III. Flow temperature ramp



IV. Continuous rate/stress ramp
(Sweep)



Peak Hold– Steady State Flow

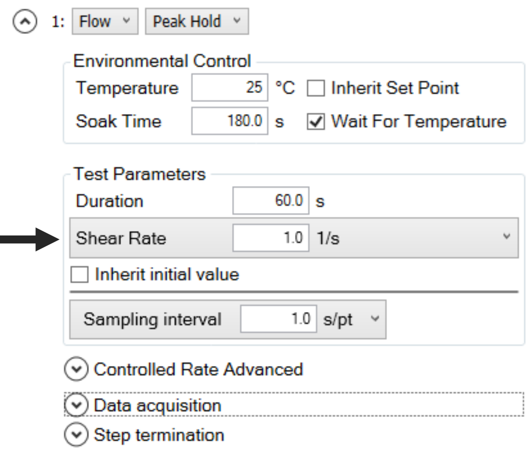


- In peak hold, a sample is held at one shear rate
- The viscosity here would be taken from the plateau

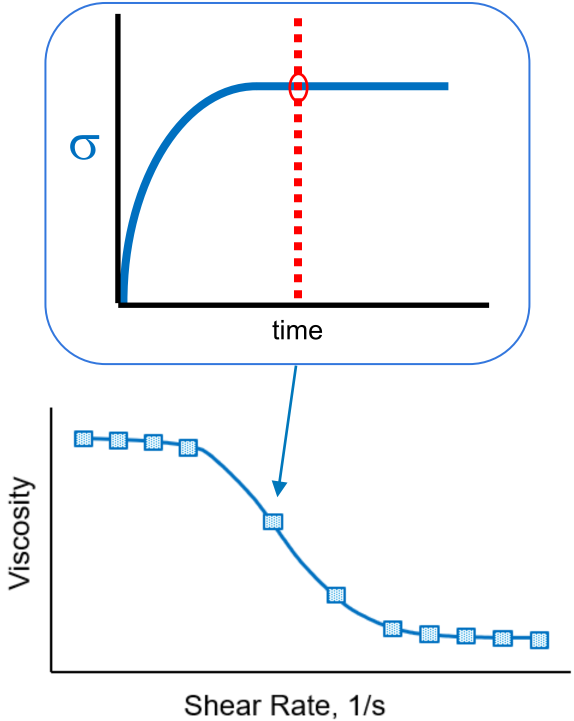
- viscosity is measured when steady state has been reached
- In TRIOS: Flow – Peak Hold

<u>USES</u>

- Viscosity at a shear rate
- Structure Recovery
- Preshear



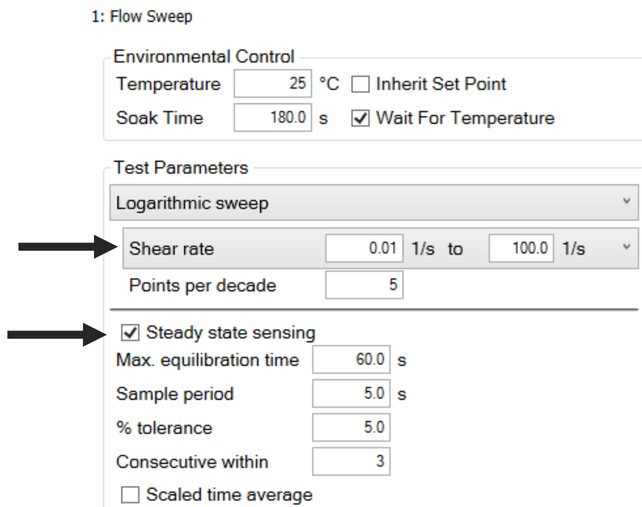
Flow Sweep – Steady State Flow



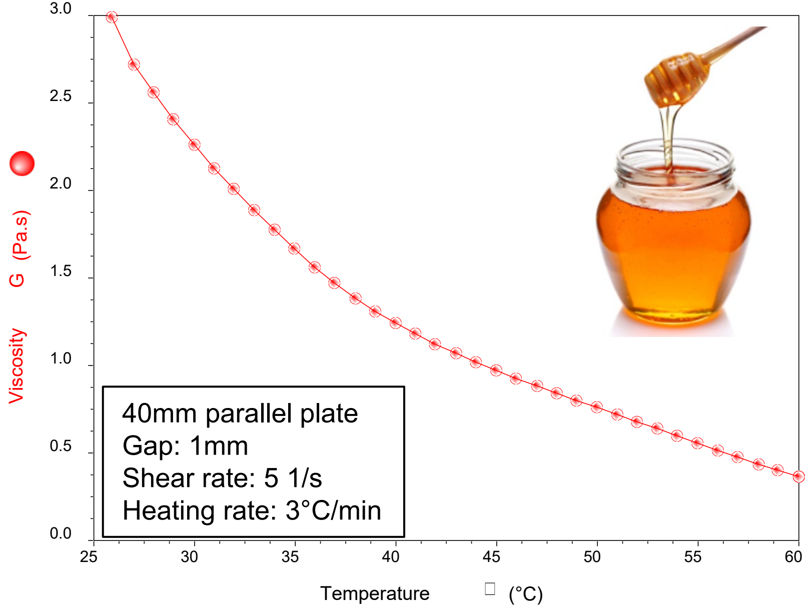
- Step stress or shear rate from low to high on a logarithmic scale
- At each step, viscosity is measured when steady state has been reached
- In TRIOS: Flow - Sweep

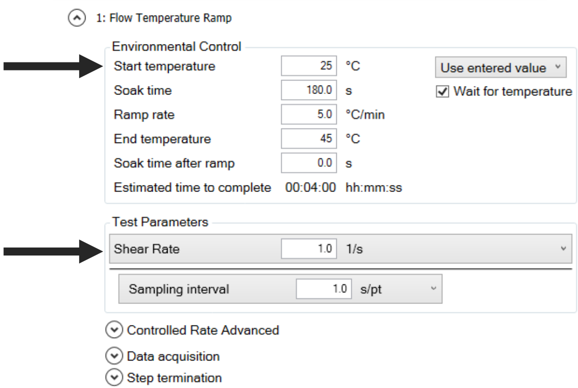
<u>USES</u>

- Viscosity Flow Curves
- Yield Stress Measurements



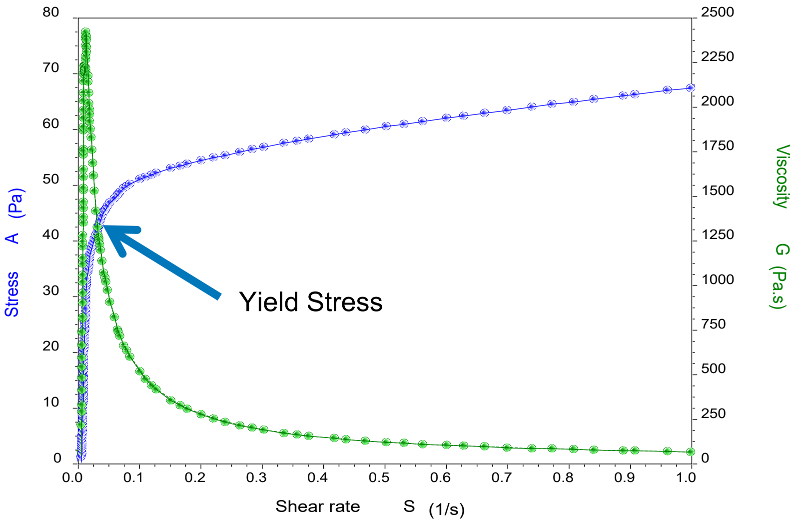
Viscosity of Honey: Temperature Dependence

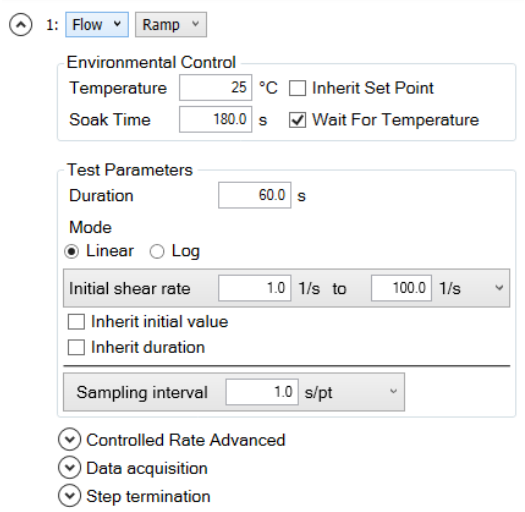




Measure Yield Stress of a Body Lotion

- Body lotion does not flow unless the applied stress exceeds a certain value – the yield point.

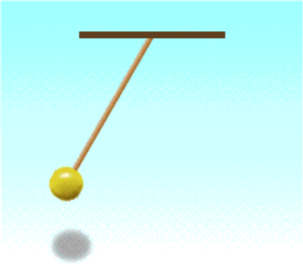




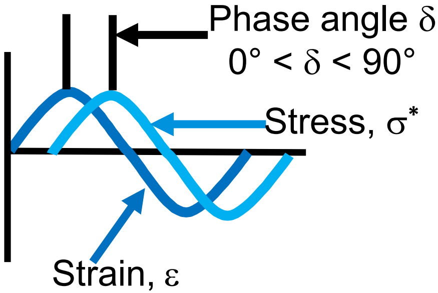
Rheology Theory and Experimental Designs

2. Oscillation Tests

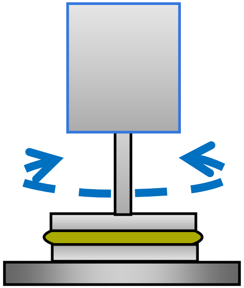
Dynamic Oscillatory Tests



- 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

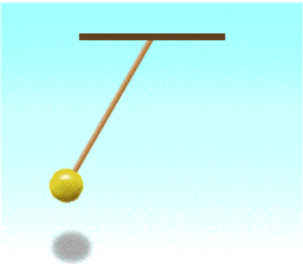


$$\gamma = \gamma_0 \cdot \sin(\omega t)$$
$$\sigma = \sigma_0 \cdot \sin(\omega t + \delta)$$



Dynamic Oscillatory Tests

- If deformation is small (within the Linear viscoelastic region) the structure of the sample is preserved
- Provides structural viscoelastic properties of sample when not forced to flow

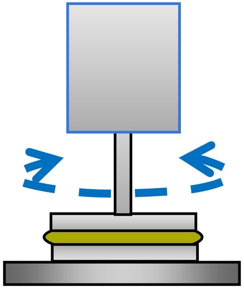


Small amplitude Oscillatory shear:

- Structure is preserved

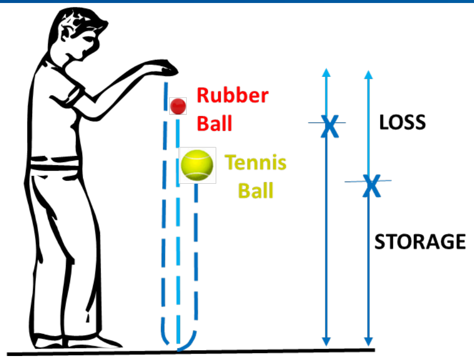
Steady Shear:

- Structure is destroyed

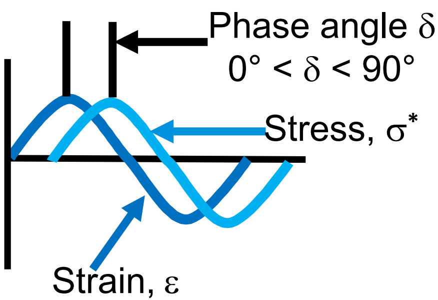


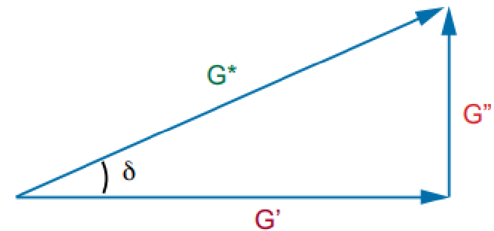
$$\gamma = \gamma_0 \cdot \sin(\omega t)$$
$$\sigma = \sigma_0 \cdot \sin(\omega t + \delta)$$

Storage and Loss of a Viscoelastic Material



Dynamic measurement represented as a vector





Rheological Parameters

- G* = Stress*/Strain
- G' = G*•cosδ
- G" = G*•sinδ
- tan δ = G"/G'

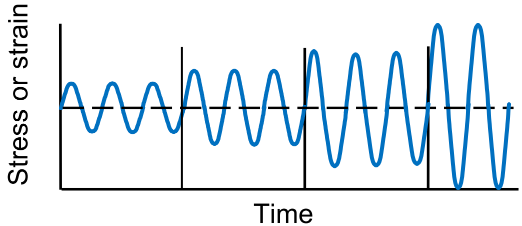
- We input either a stress or a strain waveform, and measure the phase angle between the resultant stress and strain

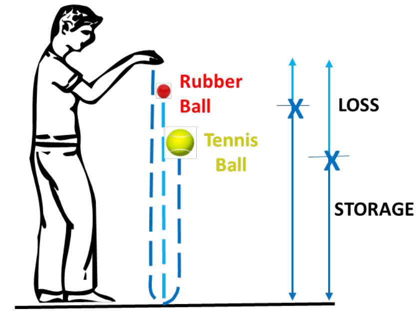
Storage and Loss of a Viscoelastic Material

- The three basic waveforms for modulating strain or stress:

Amplitude Sweep

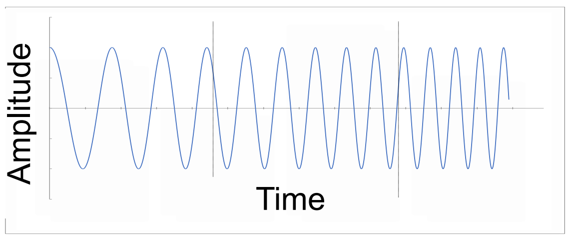
(constant frequency)





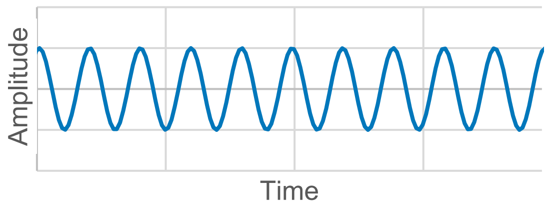
Frequency Sweep

(constant amplitude)



Time Sweep

(constant frequency and amplitude)



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

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

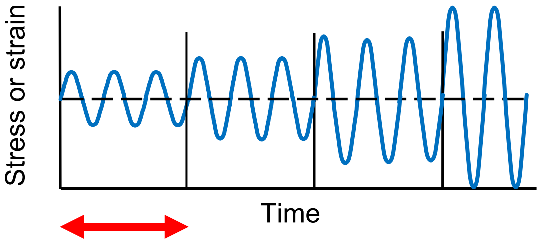
Tan Delta: Measure of material damping

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

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

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

Dynamic Strain or Stress Sweep (amplitude sweep)

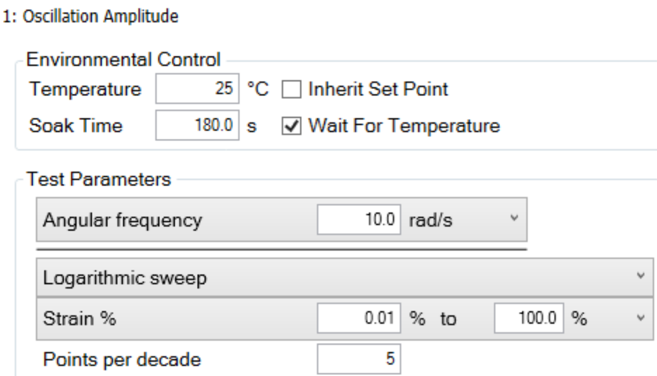


Instrument oscillates at a set amplitude for each data point, and then moves to the next amplitude

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

<u>USES</u>

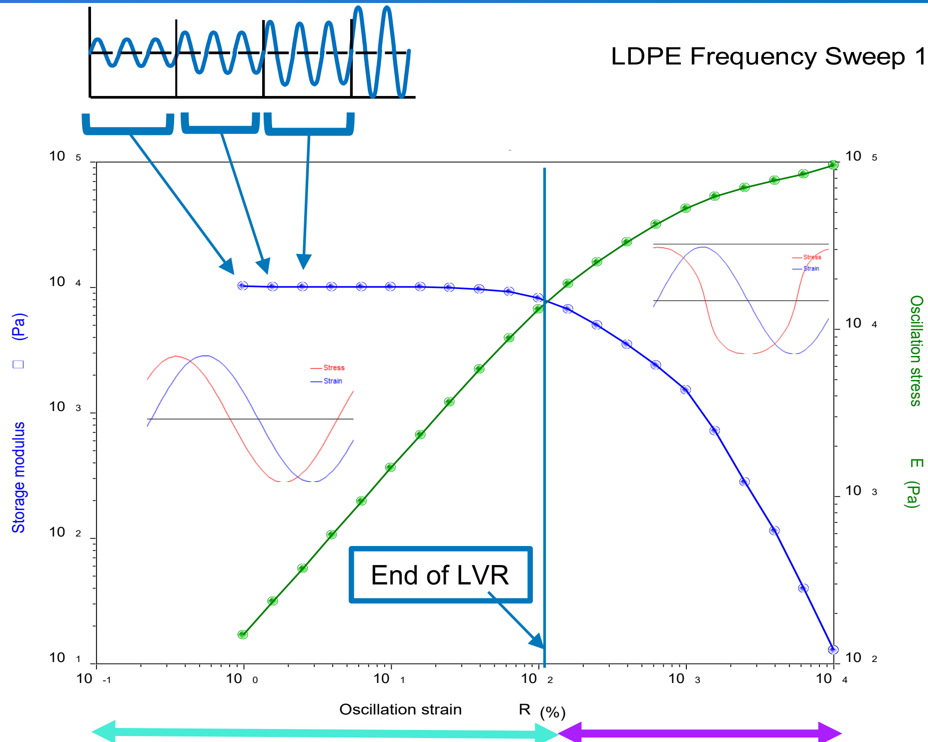
- Measure sample LVR
- Measure yield stress
- Measure non-linear viscoelastic properties □ LAOS)



Dynamic Strain or Stress Sweep (amplitude sweep)

Linear and Non-linear Viscoelasticity

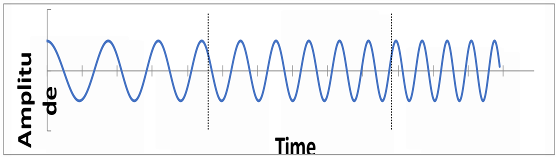
- At each data point, sample is oscillated for set number of time (or cycles)



SAOS
- Linear region: Sinusoidal excitation → Sinusoidal response
- Represented by fundamental in frequency domain

LAOS
- Nonlinear region: Sinusoidal excitation → Non-sinusoidal response
- Represented in frequency domain by fundamental and additional harmonics

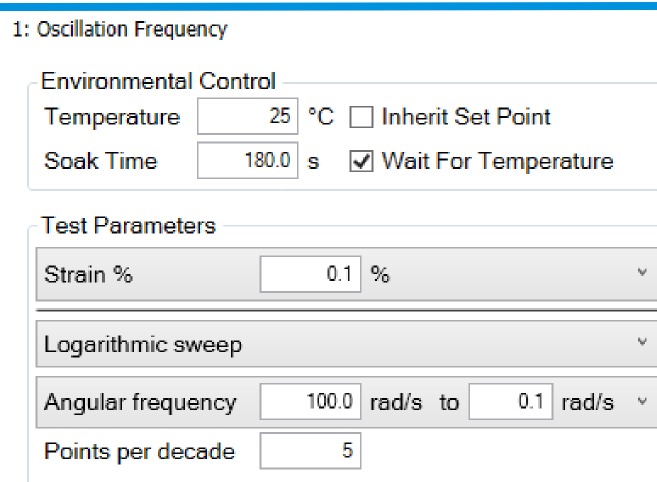
Frequency Sweep



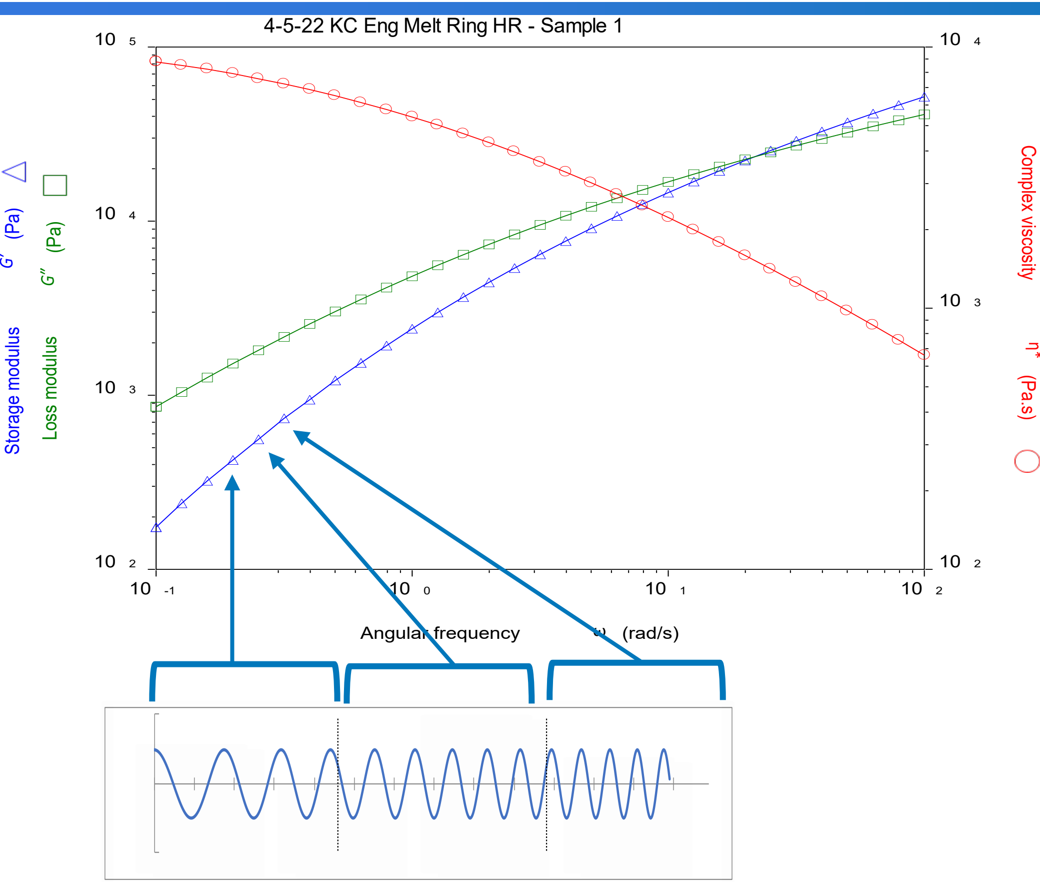
- The material response to increasing frequency (rate of deformation) is monitored at a constant amplitude (strain or stress) and temperature.
- In TRIOS: Frequency

USES

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

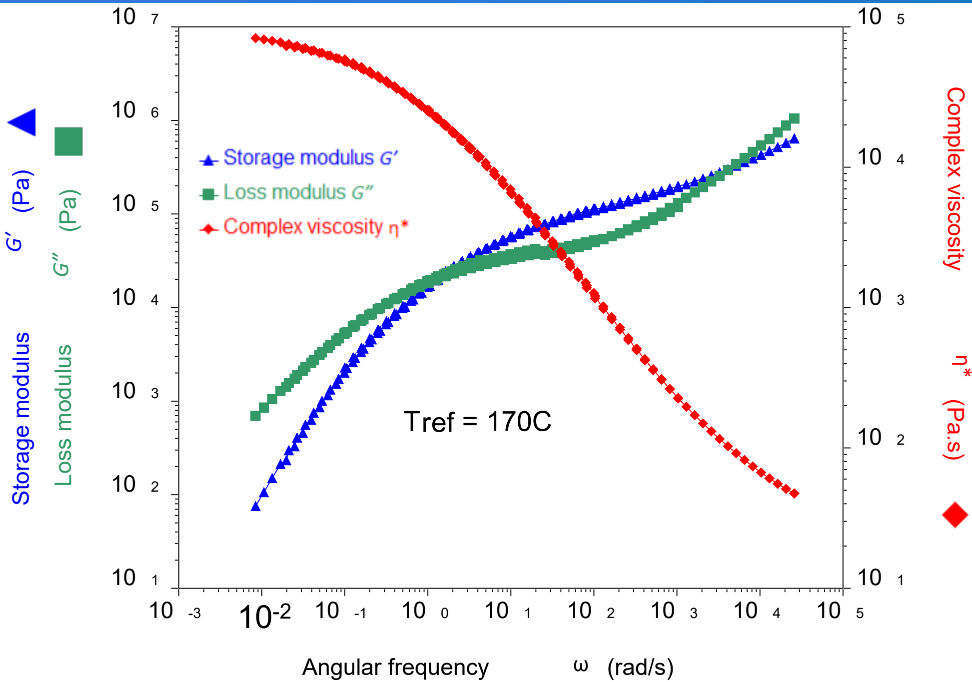


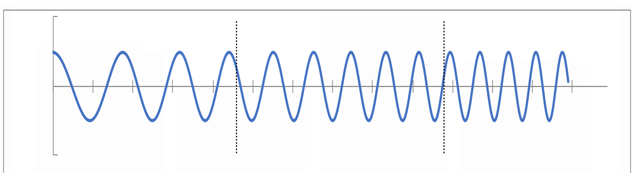
Frequency Sweep



- Complex Viscosity, storage modulus, loss modulus, and tan delta are obtained as a function of angular frequency
- This is an analogue to the flow sweep test, with the addition of viscoelasticity
- Here we consider angular frequency and complex viscosity, rather than shear rate and apparent viscosity

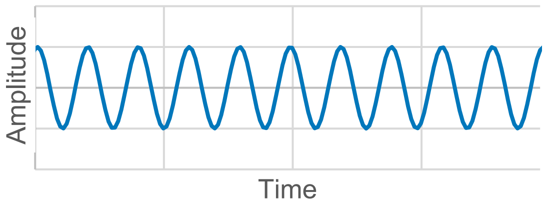
Frequency Sweep (Cont.)





- Frequency sweep data taken at multiple temperatures can be shifted using time-temperature-superposition (TTS) to generate a master curve (extended frequency range)

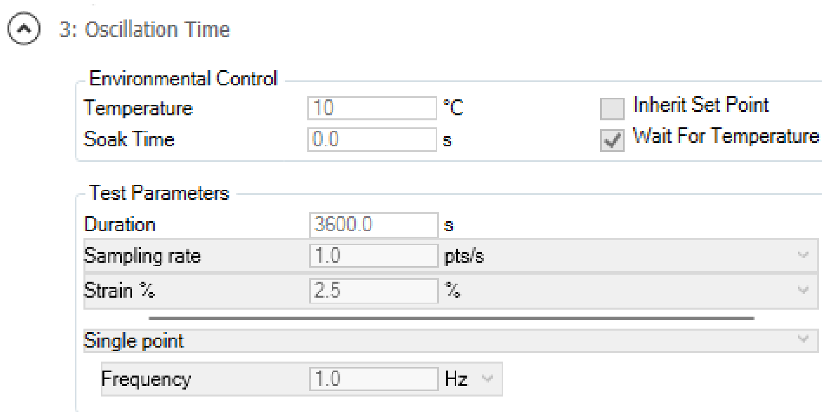
Oscillation Time

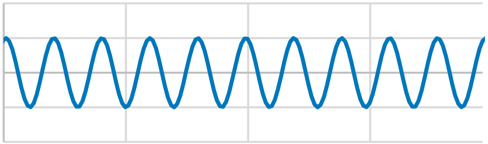


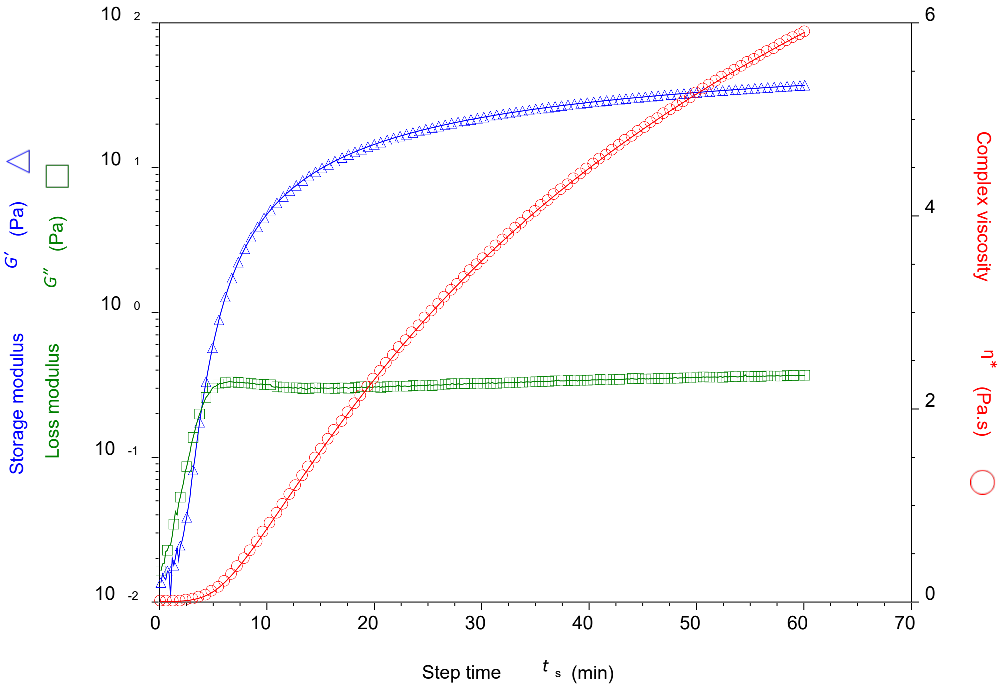
- The material response is monitored at a constant amplitude (strain or stress) and frequency.
- In TRIOS:Time

<u>USES</u>

- Sample Stability (Settling, thermal degradation)
- Sample recovery after shear (Thixotropy)
- Structural changes with time (viscoelasticity)
- Curing, Gelation

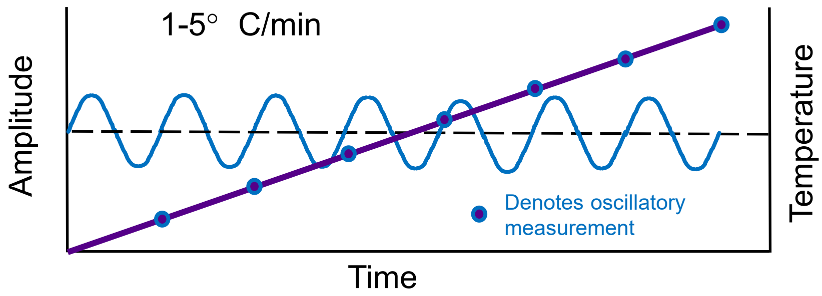






- Gelatin solution was quench cooled from 75°C to 10°C
- The oscillation time sequence was started once the sample reached 10°C
- The gelation of the solution is monitored as a function of time, at constant frequency and amplitude

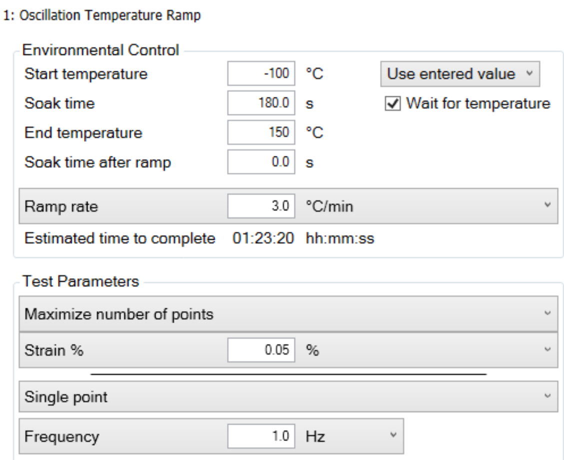
Oscillation Temperature Ramp



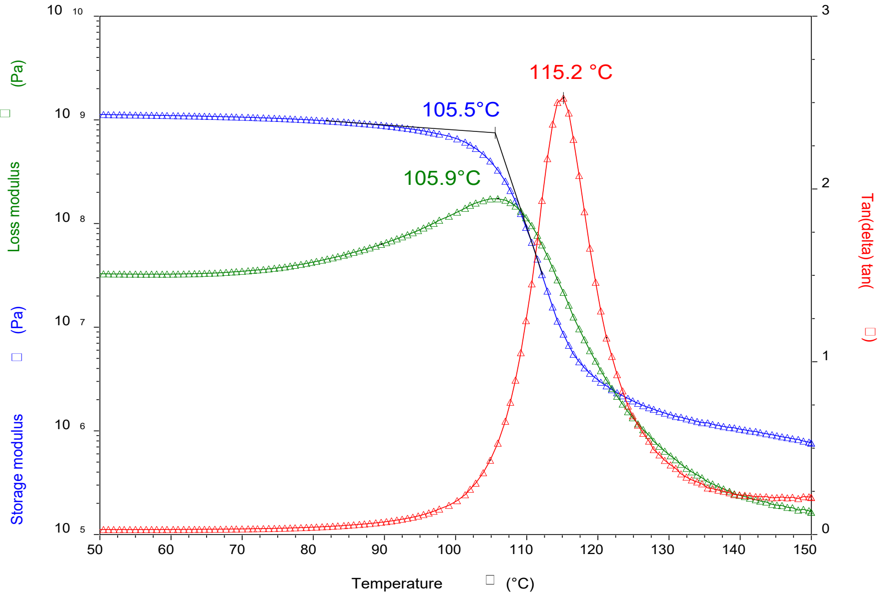
- Linear heating rate is applied, and the material response is monitored at a constant frequency and constant amplitude
- In TRIOS: Temp Ramp

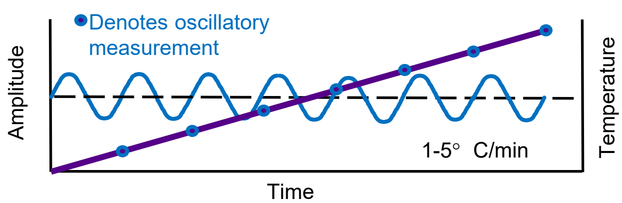
USES

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



DMA Cantilever data





- Measure moduli, tan δ and transitions

- ABS was heated from room temperature to 150°C
- The glass transition temperature was analyzed using three methods: onset of the storage modulus, the peak of the loss modulus, or the peak in tan delta

Time-Temperature Superposition (TTS)

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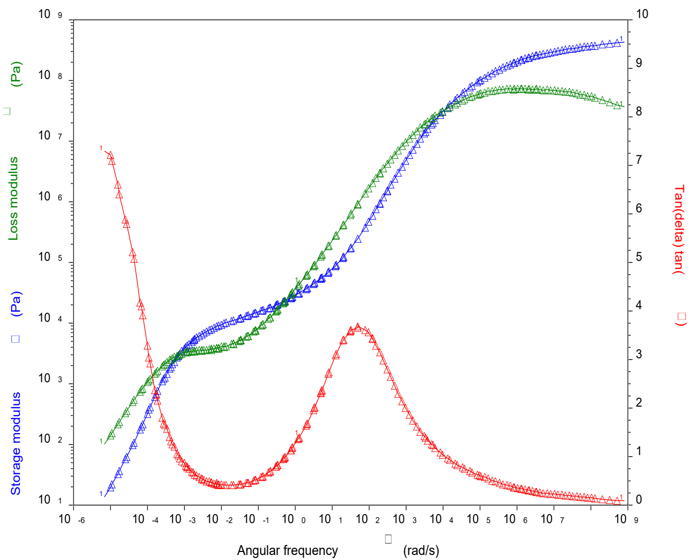
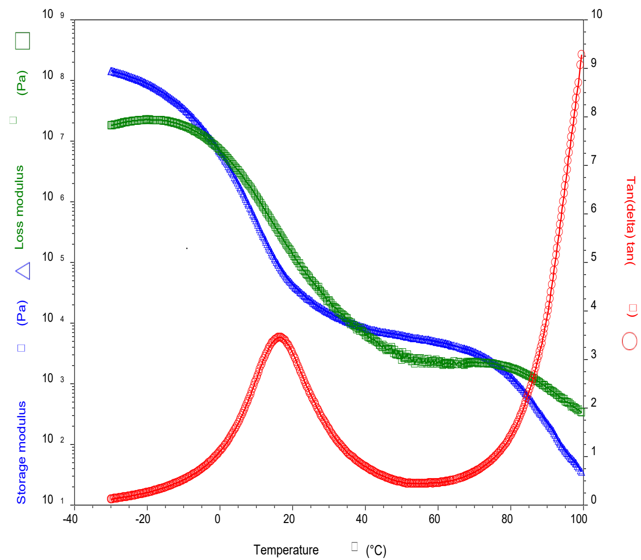
TTS master curve generated at 20°C



Time

Dynamic temperature ramp



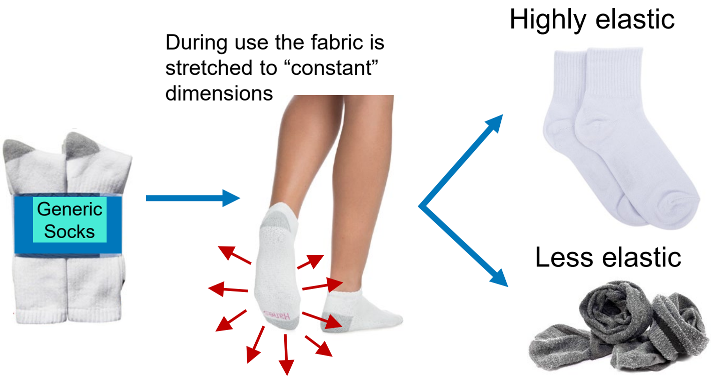
Temperature

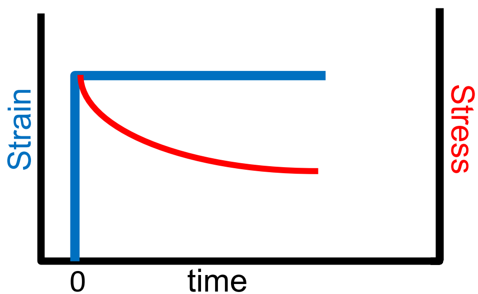
- We reviewed Oscillation Temperature Ramps and Oscillation Frequency Sweeps
- Time (frequency) and Temperature have an inverse effect on the complex properties for polymers

Rheology Theory and Experimental Designs

3. Transient Tests (Creep and Stress Relaxation)

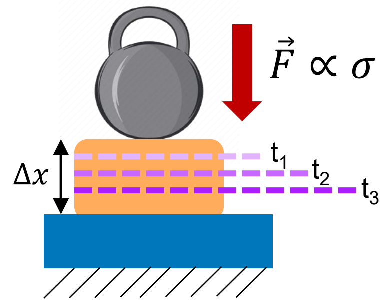
Stress Relaxation

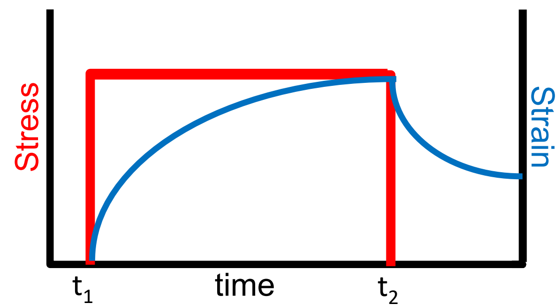




$$\text{Relaxation modulus} = \frac{\text{Stress}}{\text{Strain}}$$

Creep/Creep recovery

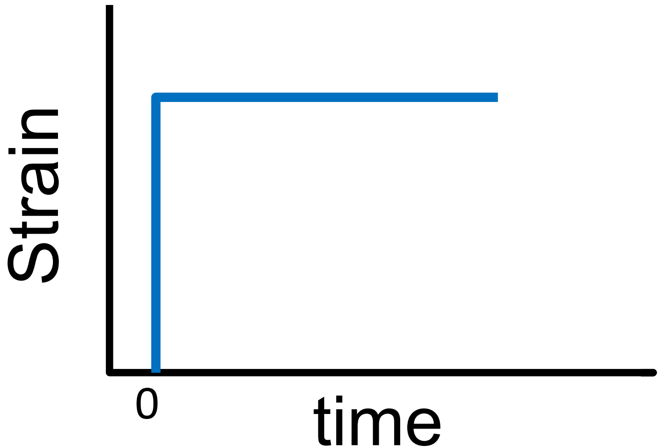




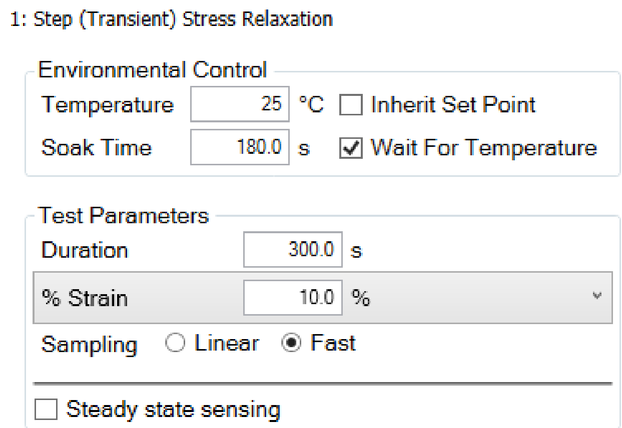
$$\text{Creep compliance J} = \frac{\text{Strain}}{\text{Stress}}$$

Stress Relaxation

- Strain is applied to sample instantaneously (in principle) and held constant with time.
- Stress is monitored as a function of time $\sigma(t)$.



$$\text{Relaxation modulus} = \frac{\text{Stress}}{\text{Strain}}$$



Stress Relaxation

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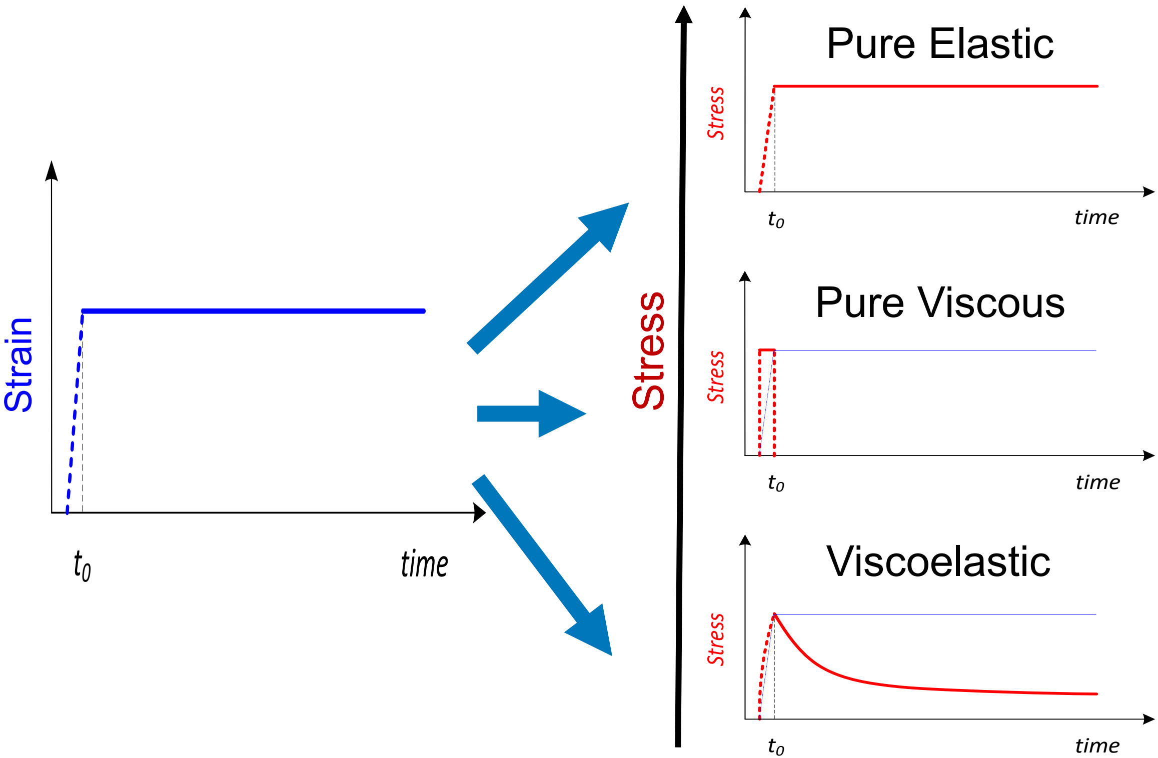
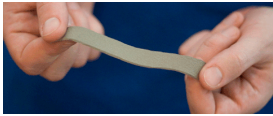
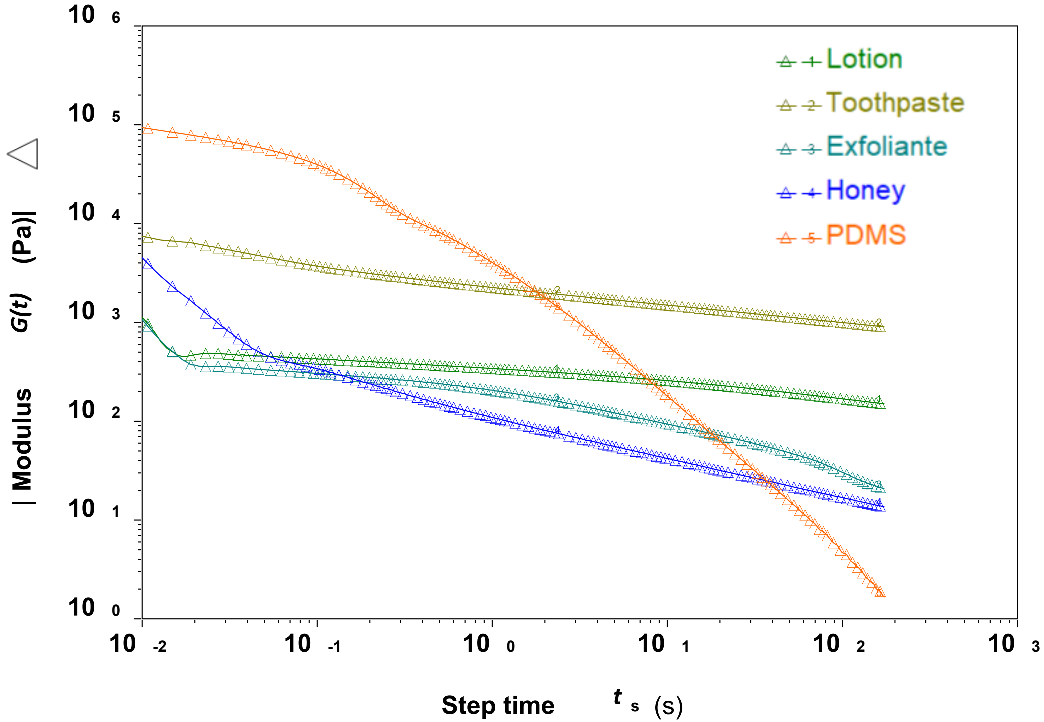




Stress Relaxation



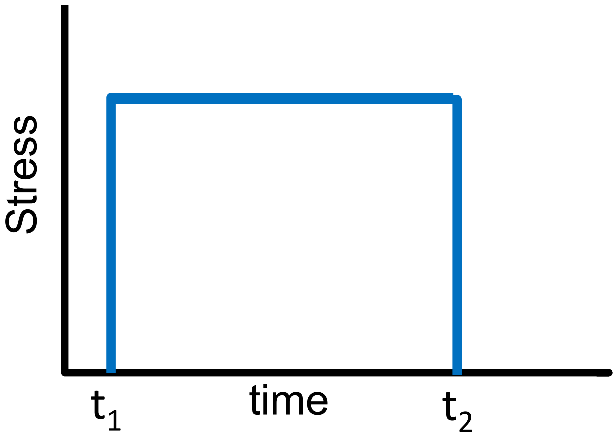
- Test using a 40mm sandblasted parallel plates at a strain of 5%

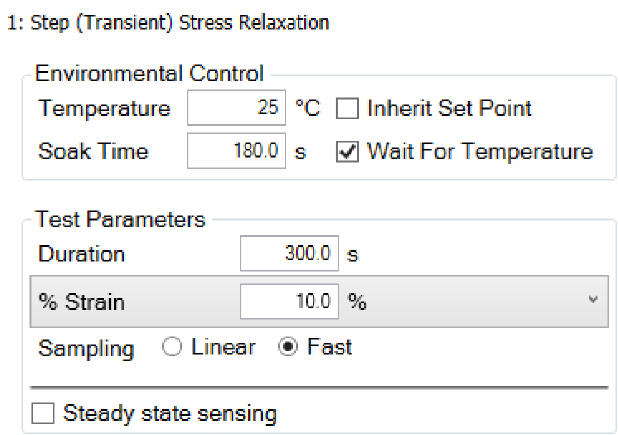
Creep Recovery

- Creep: Stress is applied to sample instantaneously at t_1, and held constant for a specific period of time. The strain is monitored as a function of time ($\gamma(t)$ or $\varepsilon(t)$)

- Recovery: Stress is reduced to zero at t_2, and the strain is monitored as a function of time ($\gamma(t)$ or $\varepsilon(t)$)

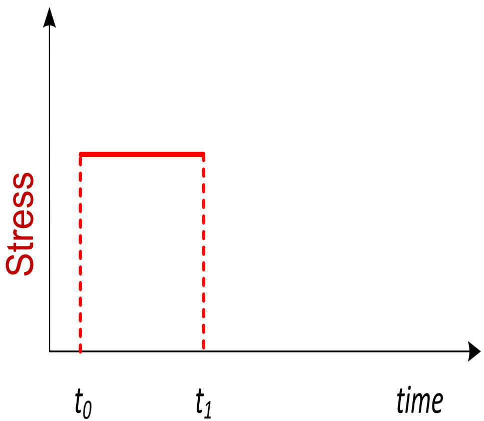
$$\text{Creep compliance } J = \frac{\text{Strain}}{\text{Stress}}$$

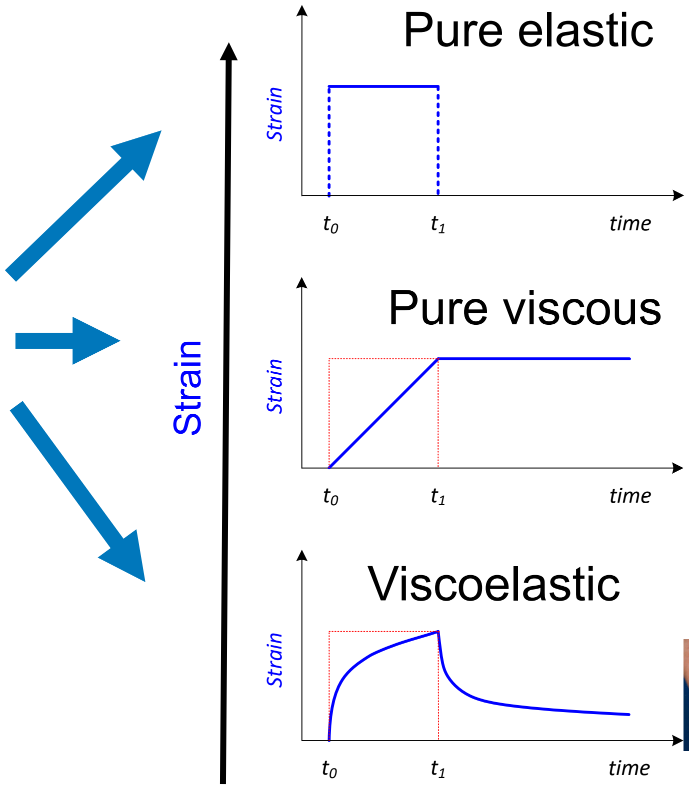




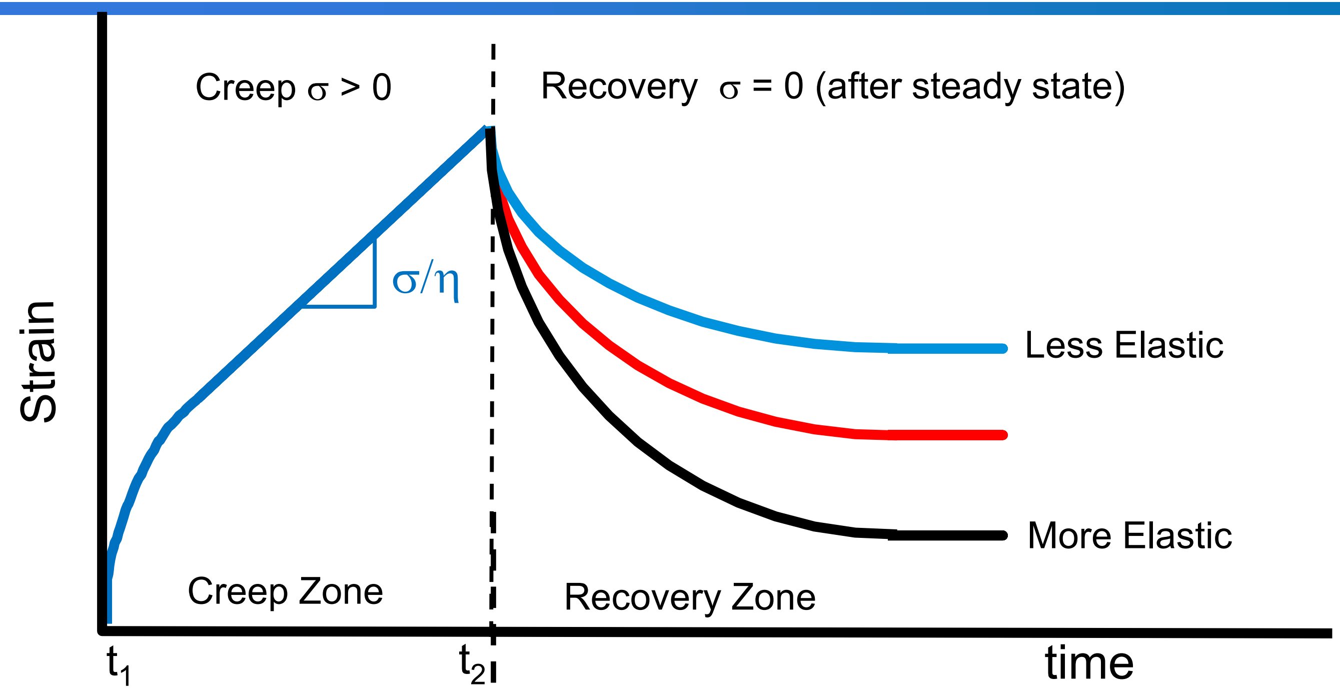
Creep Recovery

- Step Stress





Creep Recovery

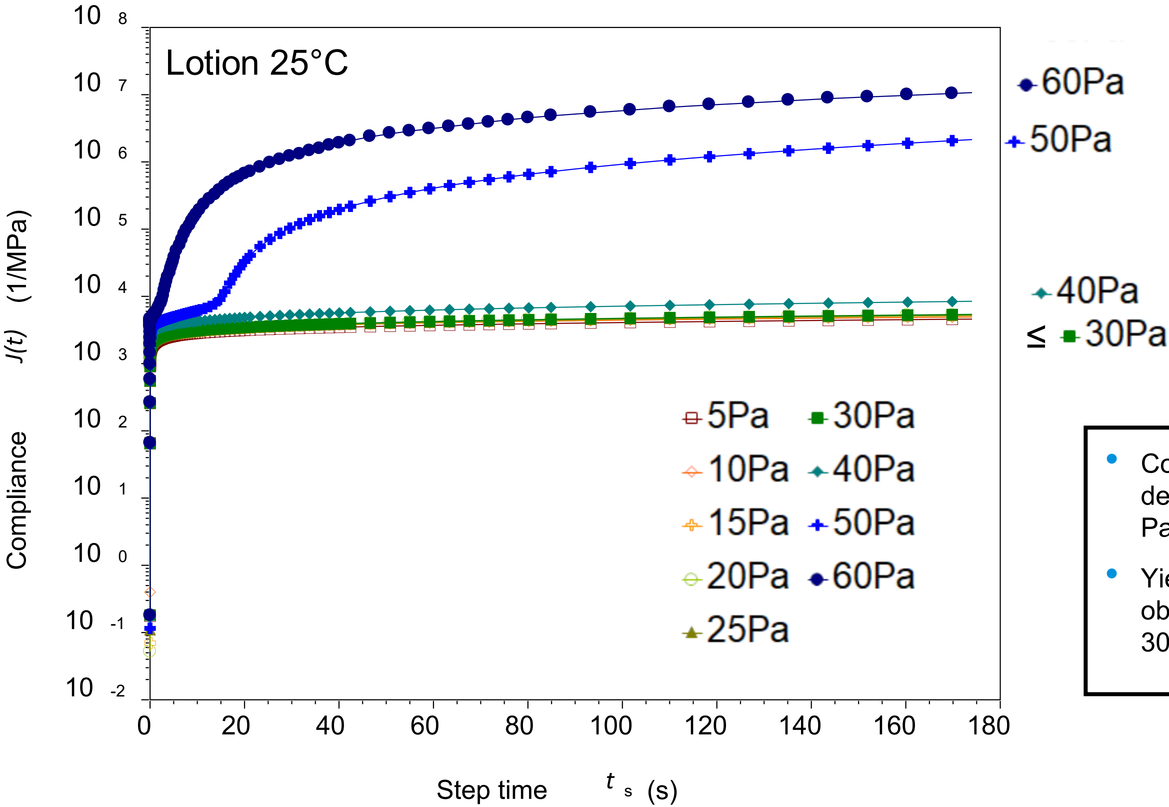


Strain rate decreases with time in the creep zone, until finally reaching a steady state.

In the recovery zone, the viscoelastic fluid recoils, eventually reaching a equilibrium at some small total strain relative to the strain at unloading.

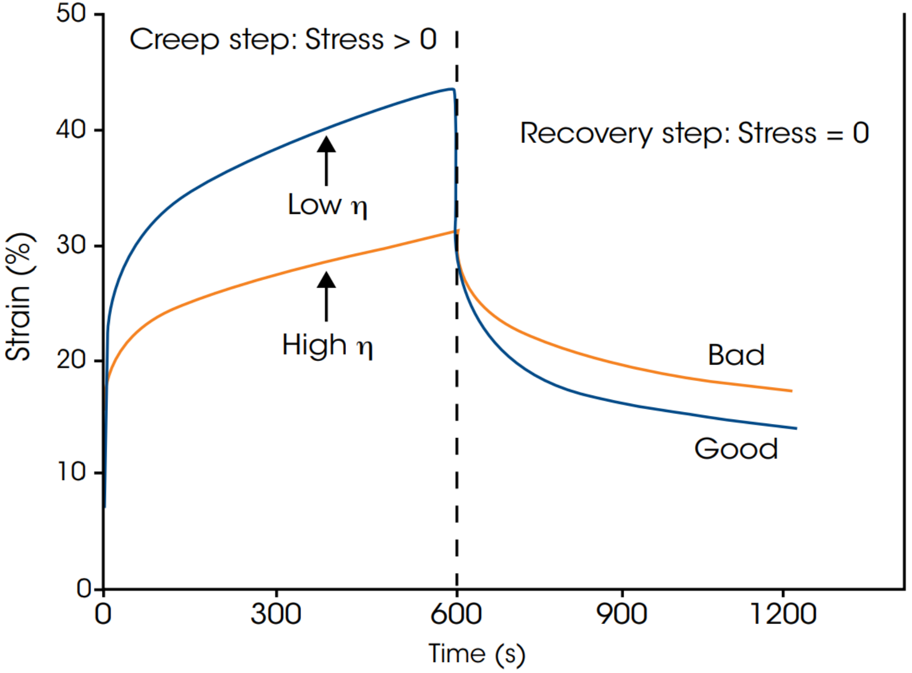
Mark, J., et. al., Physical Properties of Polymers, American Chemical Society, 1984, p. 102.

Creep (Yield Stress)



- Compliance deviates above 30 Pa of stress
- Yield stress can be observed between 30 and 40 Pa

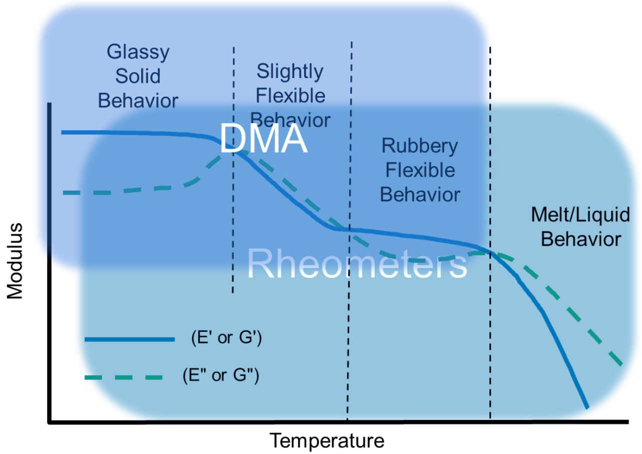
Creep Recovery (Elasticity)



DMA Testing modes

Overlapping tests with the Rheometer

- Creep and Stress Relaxation
- Oscillation tests



Unique to DMA (Both DHR and DMA850)

- Monotonic testing (pull to failure at a specified rate)

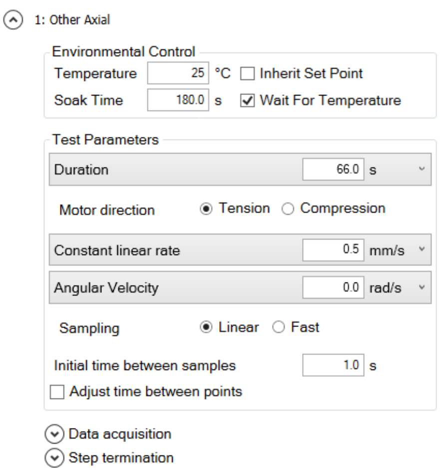
Unique to DMA850

- Fatigue Testing

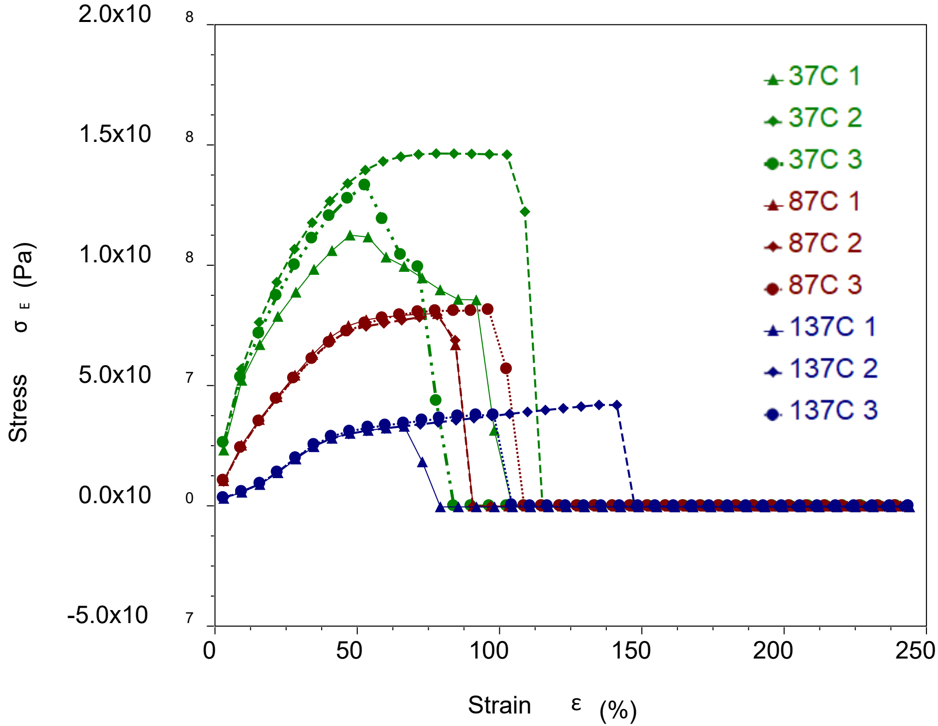
DMA Testing modes

Monotonic Testing – DMA Tension

- Pull sample at one rate
- Generate Stress vs Strain plot



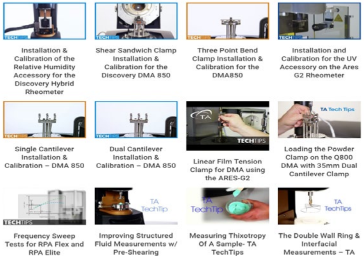
Battery Separator Films



TA Website – Other Resources

Waters™ | TA Instruments

Tech Tips



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

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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.

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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

Thank You!