# Materials Characterization by Thermal Analysis (DSC & TGA), Rheology, and Dynamic Mechanical Analysis (Part 2)

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Applications Scientists & Sales Representative

# What Does TA Instruments Make?

- Differential Scanning Calorimeters
- Thermogravimetric Analyzers
- Simultaneous Differential Thermal Analyzers
- Microcalorimeters of many types
- Dilatometers and Thermomechanical Analyzers
- Thermal Diffusivity
- Thermal Conductivity
- Mechanical Testers
- **Dynamic Mechanical Analyzers**
- Rotational Rheometers
- Rubber Rheometers

# Dynamic Mechanical Analyzers

# Is DMA Thermal Analysis or Rheology?

- ***Thermal Analysis***
  - measurement as a *function of temperature or time*.

- ***Rheology***
  - the science of *stress* and *deformation* of matter.

- ***DMA*** mechanically *deforms a sample* and measures the sample response. The response to the deformation can be monitored as a *function of temperature or time.*

# DMAs from TA Instruments

**RSA G2**

**Discovery DMA 850**

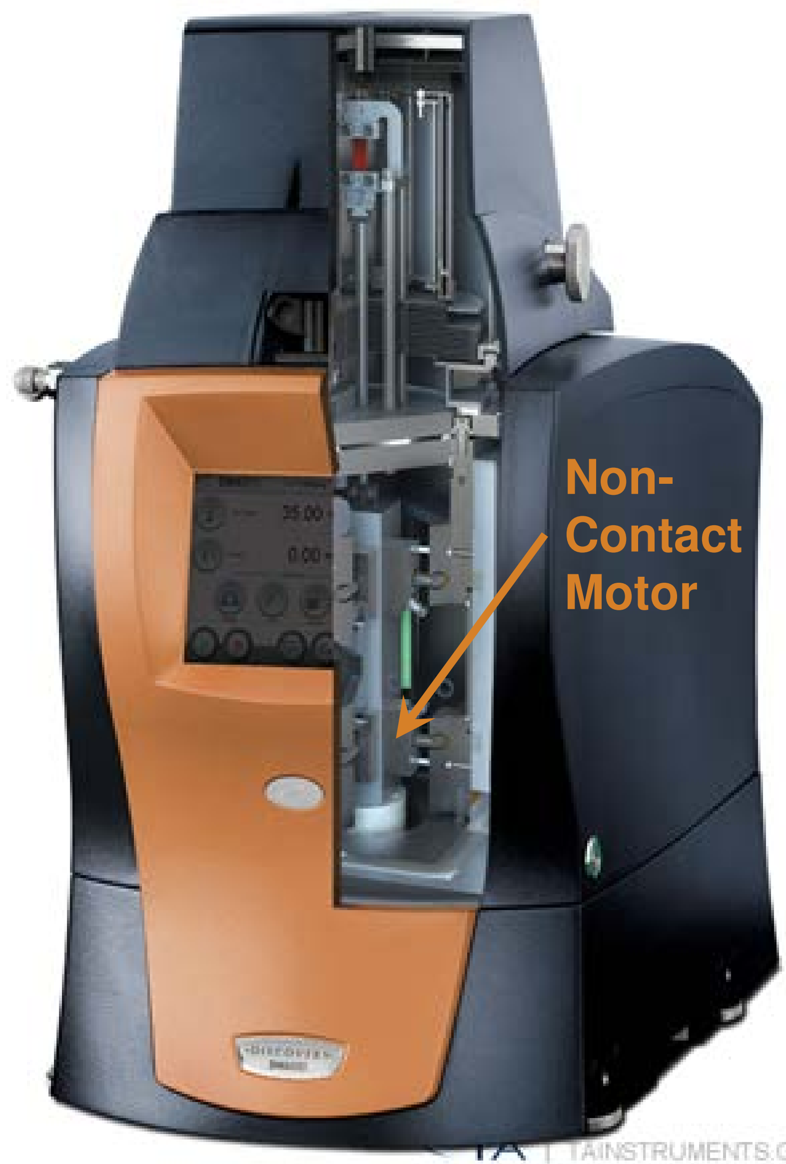
**Q800**

**ARES G2 and DHR**

DMA mode

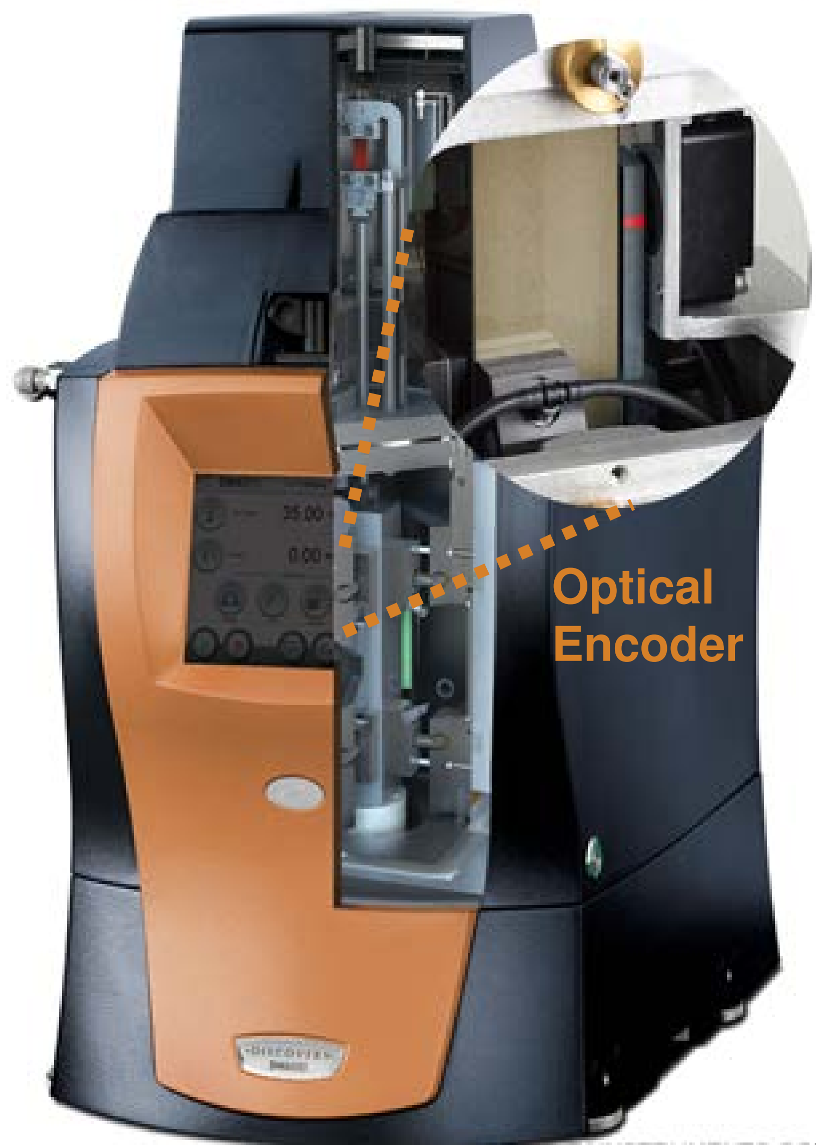
# Discovery DMA 850

- Direct Drive Motor
  - Non-contact motor for applying static and dynamic deformation
  - Constructed from high performance, lightweight composites for maximal stiffness and minimal inertia
  - Fastest motor control over widest continuous force range: 0.1 mN to 18 N
    - 50 ms step-displacement response
    - 100× improvement in stress accuracy
  - Apply any combination of static and dynamic forces up to 18 N

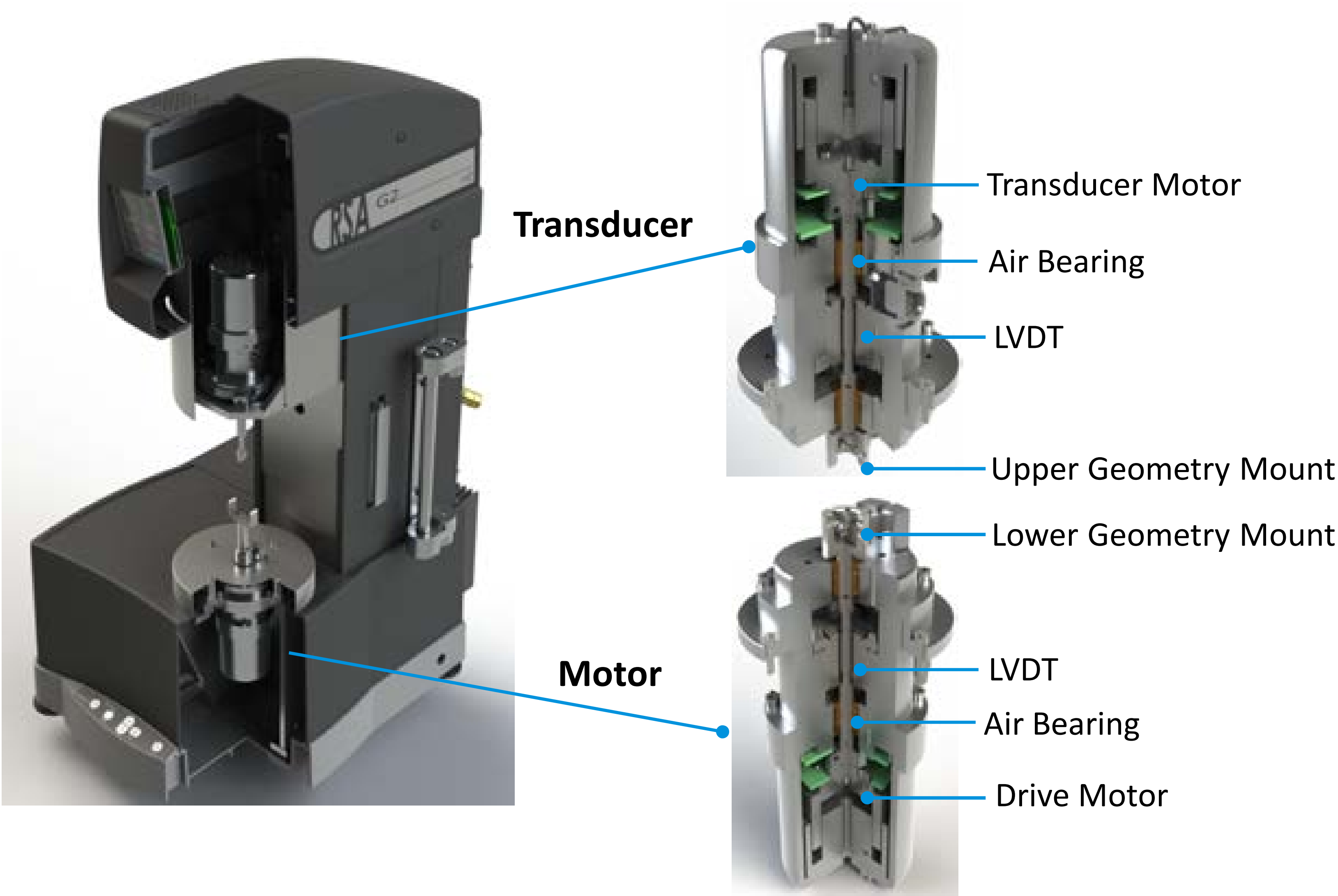


# Discovery DMA 850

- Air Bearing & Optical Encoder
  - Stiff, frictionless air bearing supports rectangular drive shaft
  - Set of 8 porous carbon air bearings for frictionless 'floating' of the drive shaft
  - Superior displacement control, sensitivity over unsupported and spring-supported designs
  - Optical encoder for displacement over 25 mm travel range with 0.1 nm resolution
    - 100× smaller displacements
    - 5 nm oscillation displacement control



# RSA G2 Schematic: Dual Head Design

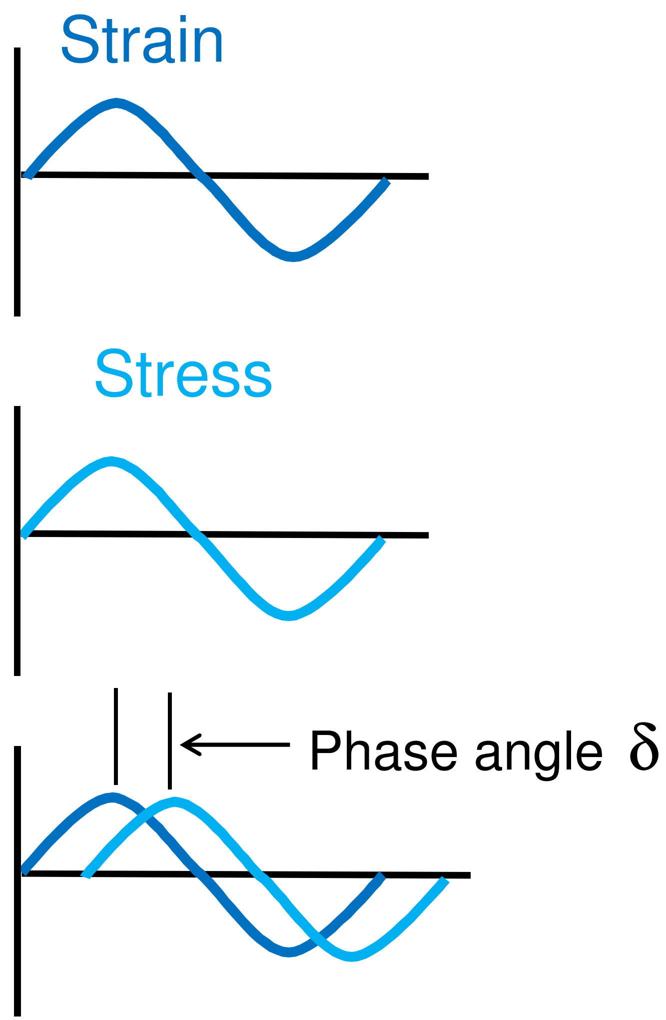


# DMA Oscillation Testing

- Viscoelastic materials (polymers, gels, composites)
- Oscillatory Deformation, controlled Frequency and Amplitude (Strain or Stress)
- Information provided:
  - Storage Modulus, Loss Modulus and Tan Delta
  - Glass Transition, Relaxation Time, Cure behavior
  - Polymer structure- Bulk property relationships

# Oscillation Testing

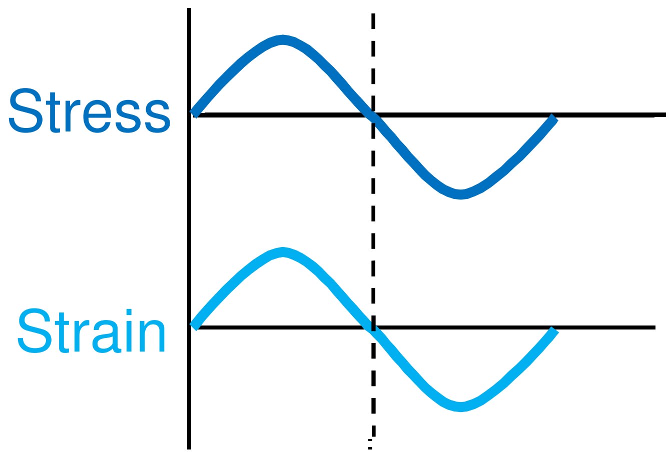
- An oscillatory (sinusoidal) deformation (strain) is applied to a sample.
- The material response (stress) is measured.
- The phase angle δ, or phase shift, between the deformation and response is measured.



# Oscillation Testing: Response for solids and liquids

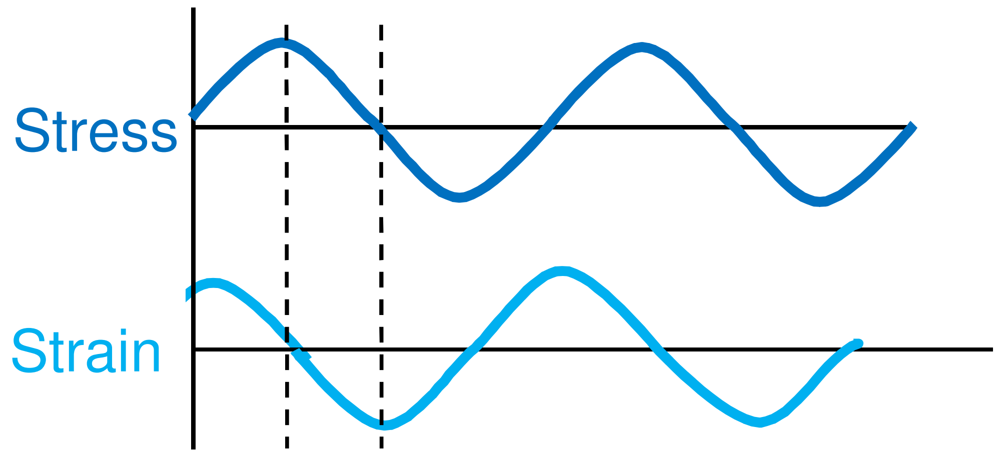
Purely Elastic Response
(Hookean Solid)

$\delta = 0°$

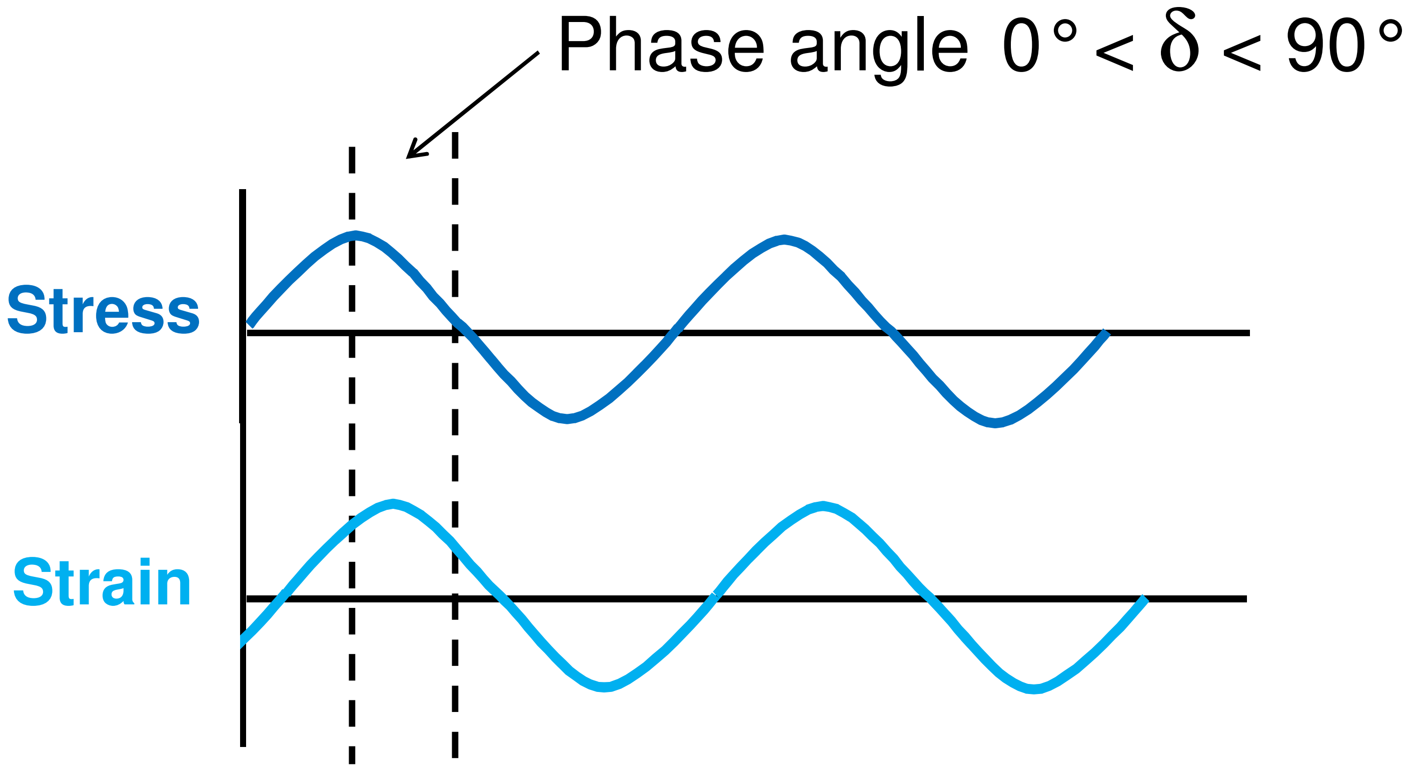



Purely Viscous Response
(Newtonian Liquid)

$\delta = 90°$



# Oscillation Testing: Viscoelastic Material



# DMA Viscoelastic Parameters

The Modulus: Measure of materials overall resistance to deformation.

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

The Elastic (Storage) Modulus:
Measure of elasticity of material. The ability of the material to store energy.

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

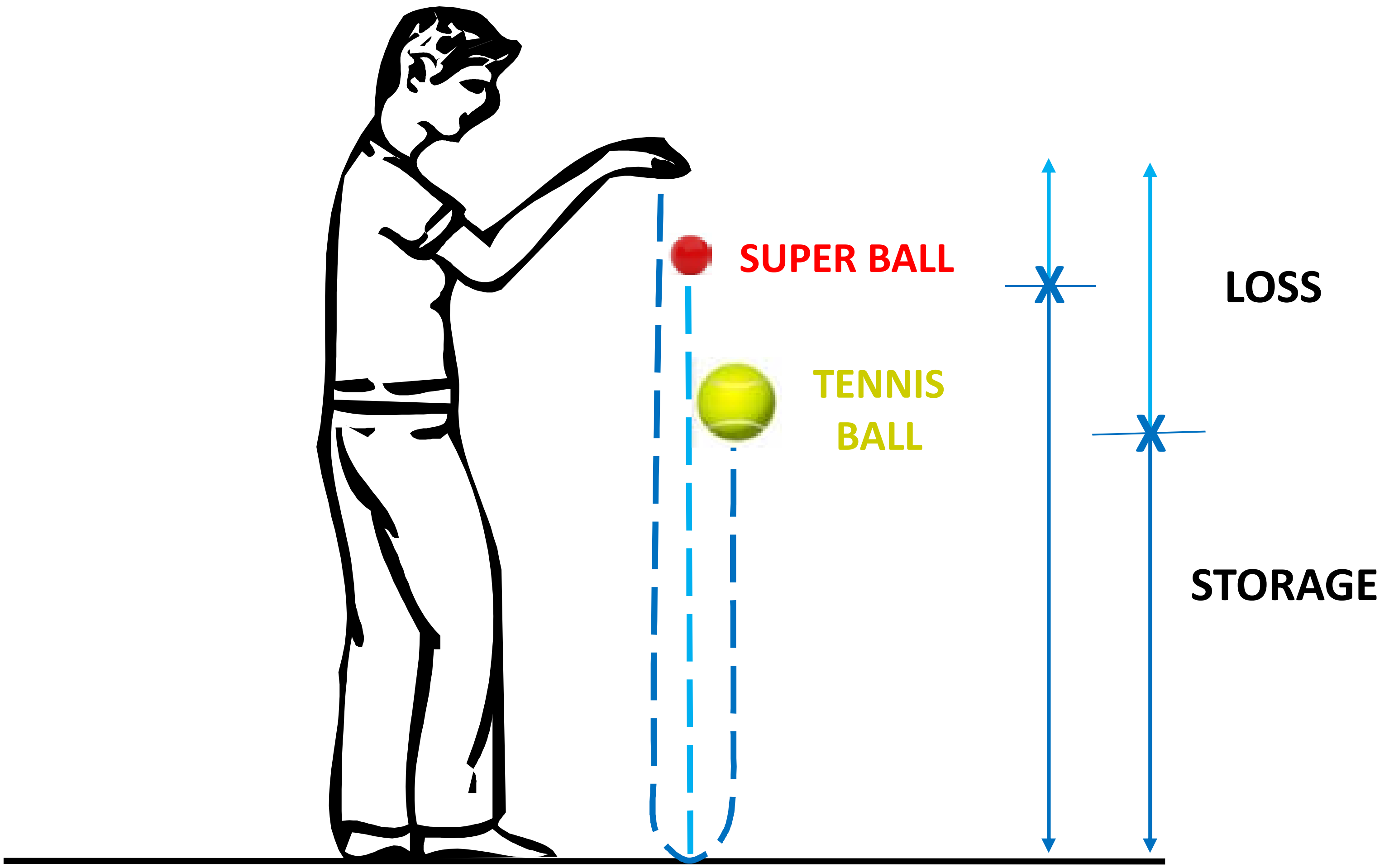
The Viscous (Loss) Modulus:
The ability of the material to dissipate energy. Energy lost as heat.

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

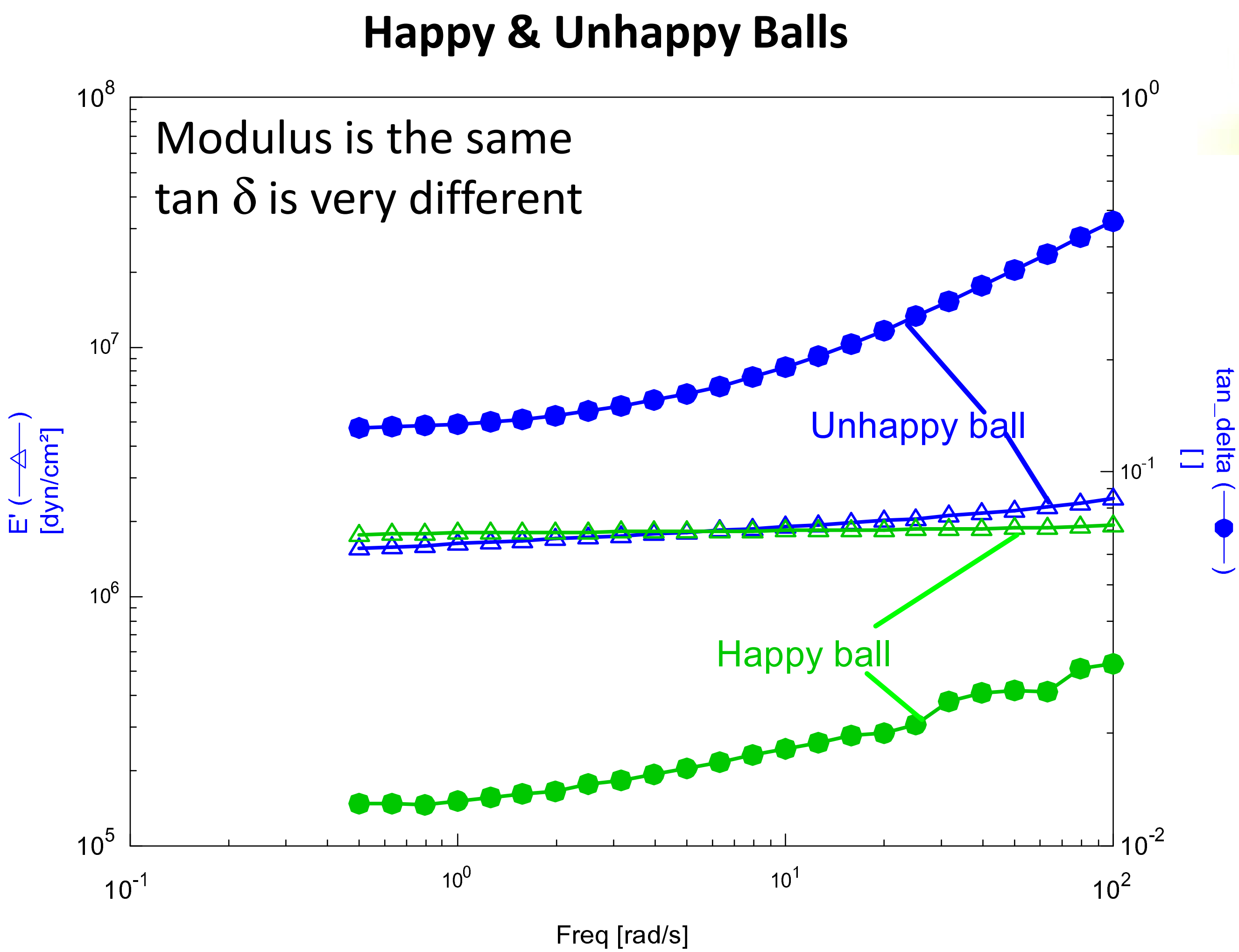
Tan Delta:
Measure of material damping - such as vibration or sound damping.

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

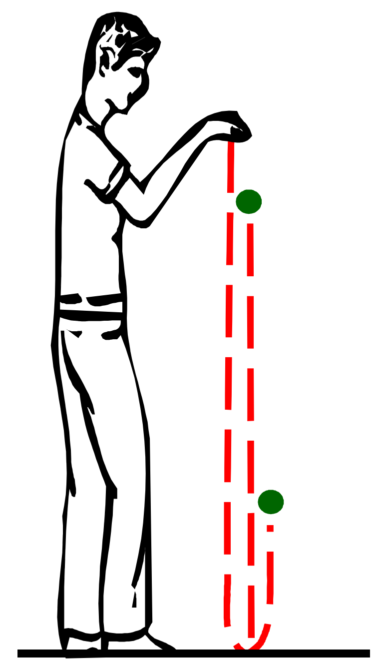
# Storage and Loss of a Viscoelastic Material



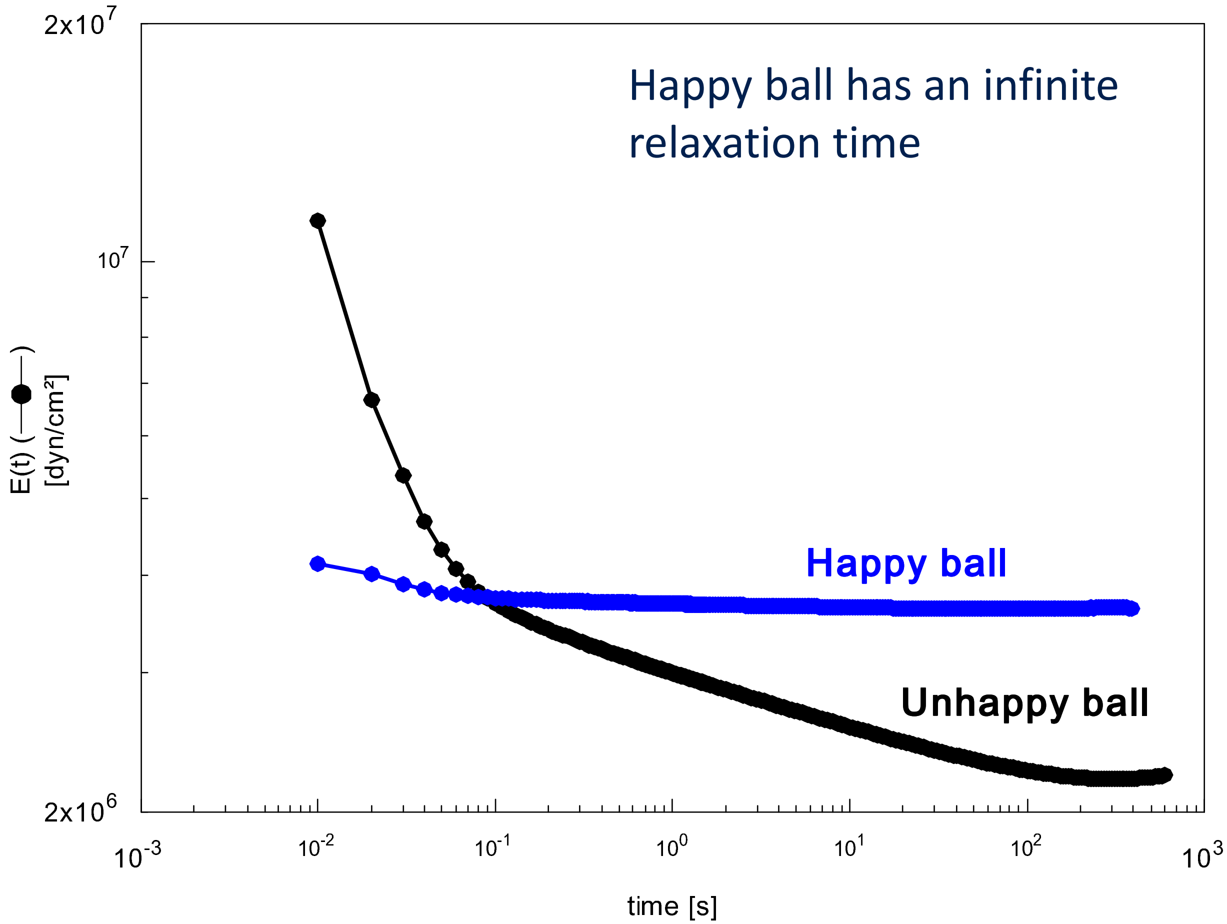
# Happy & Unhappy Balls



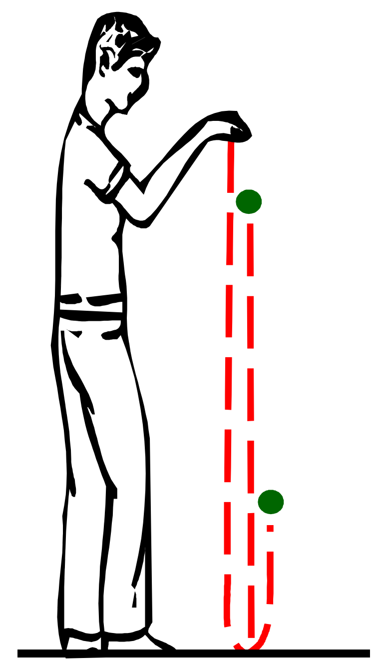




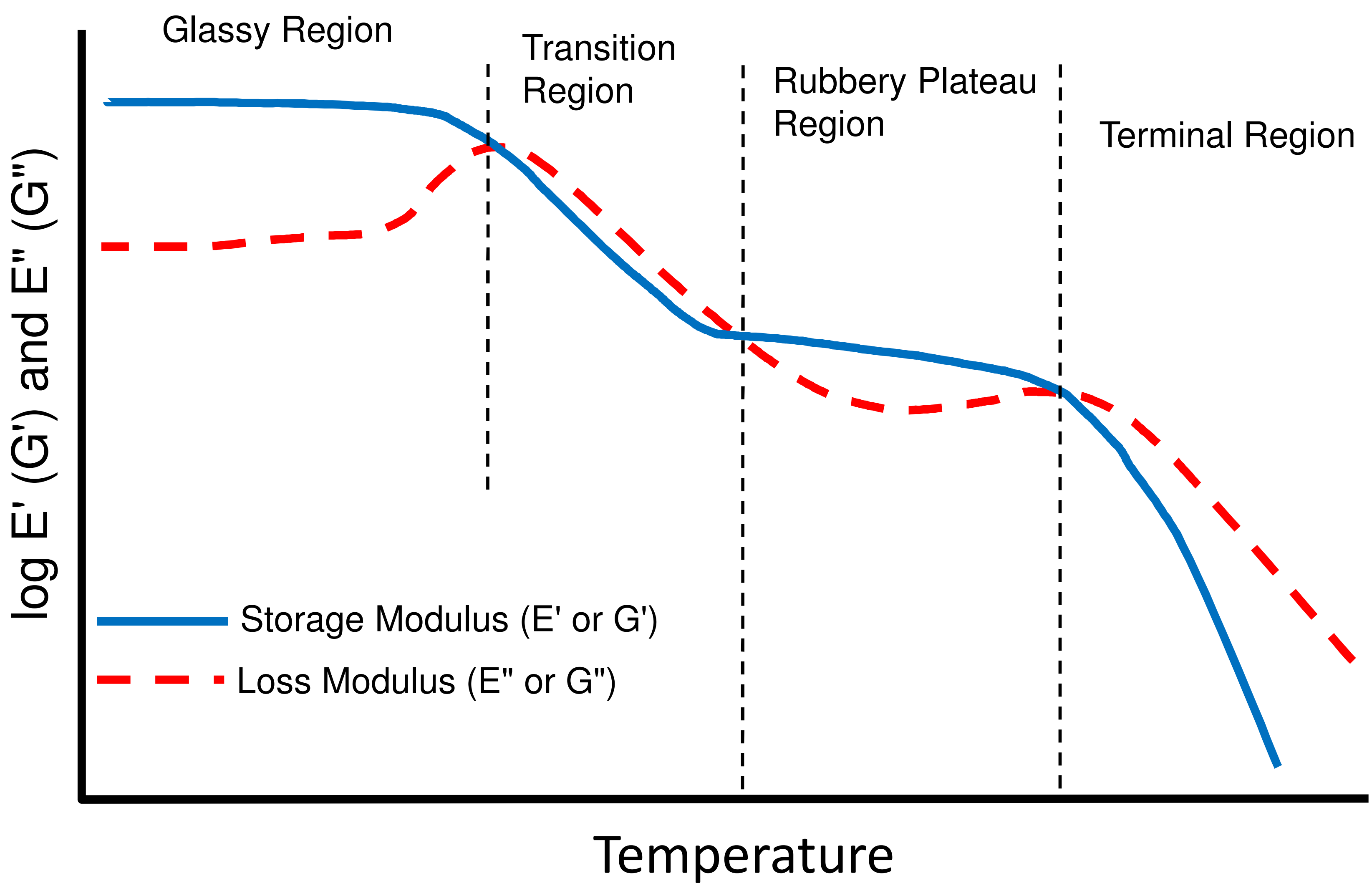
# Stress Relaxation Happy and Unhappy Balls







# Dynamic Temperature Ramp



# Glass Transition E' Onset, E'' Peak, and Tan $\delta$ Peak

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

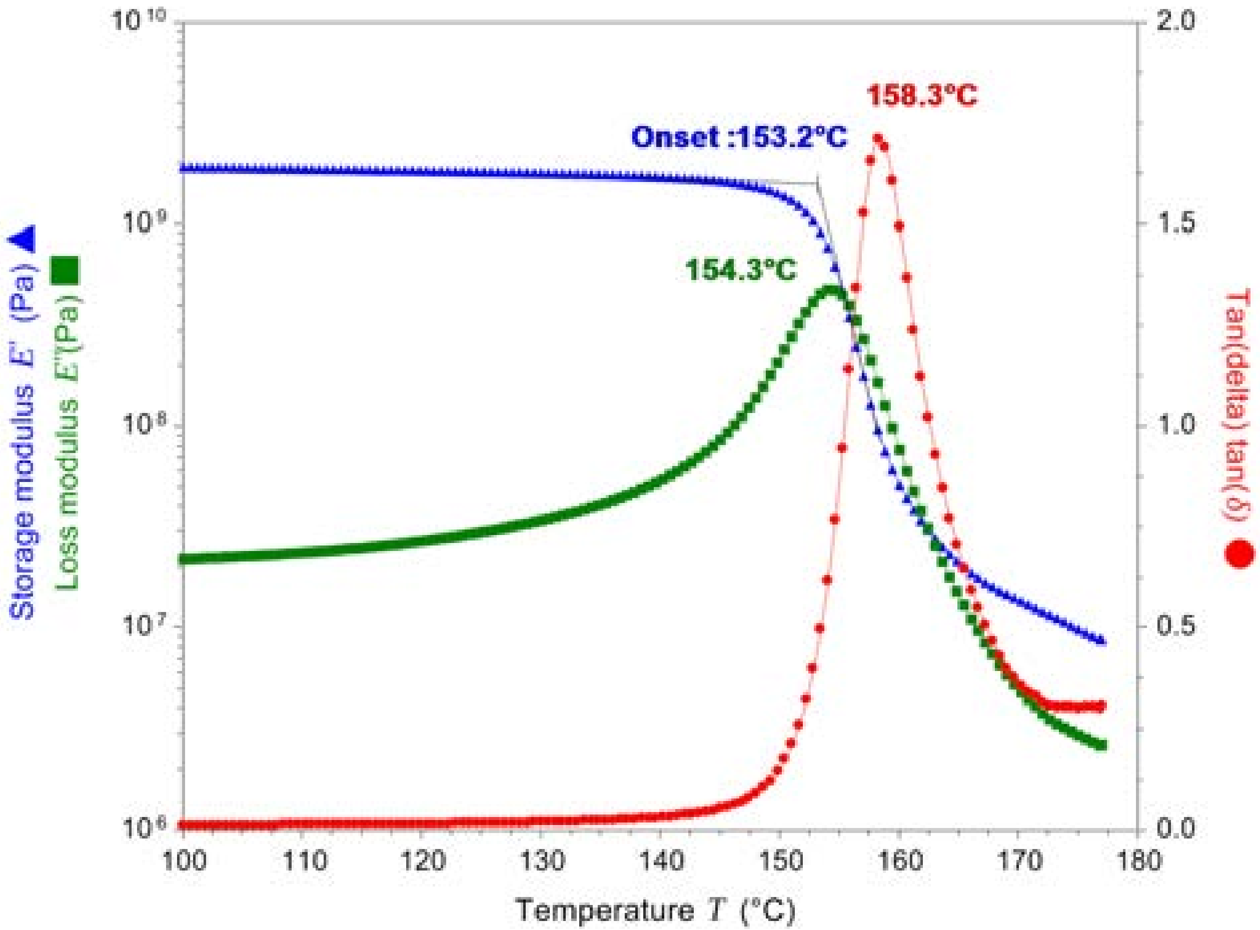
- **Loss Modulus E'' Peak**:
  - Occurs at middle temperature
  - Related to the physical property changes
  - Reflects molecular processes - the temperature at the onset of segmental motion

- **Tan Delta Peak:**
  - Occurs at highest temperature; Used historically in literature
  - Measure of the "leatherlike" midpoint between the glassy and rubbery states
  - Height and shape change systematically with amorphous content.

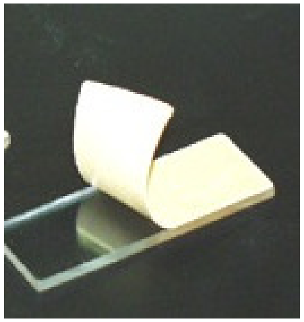
Turi, Edith, A, Thermal Characterization of Polymeric Materials, Second Edition, Volume I., Academic Press, Brooklyn, New York, P. 980.

# Glass Transition of Polycarbonate

- Available from TA for Instrument verification



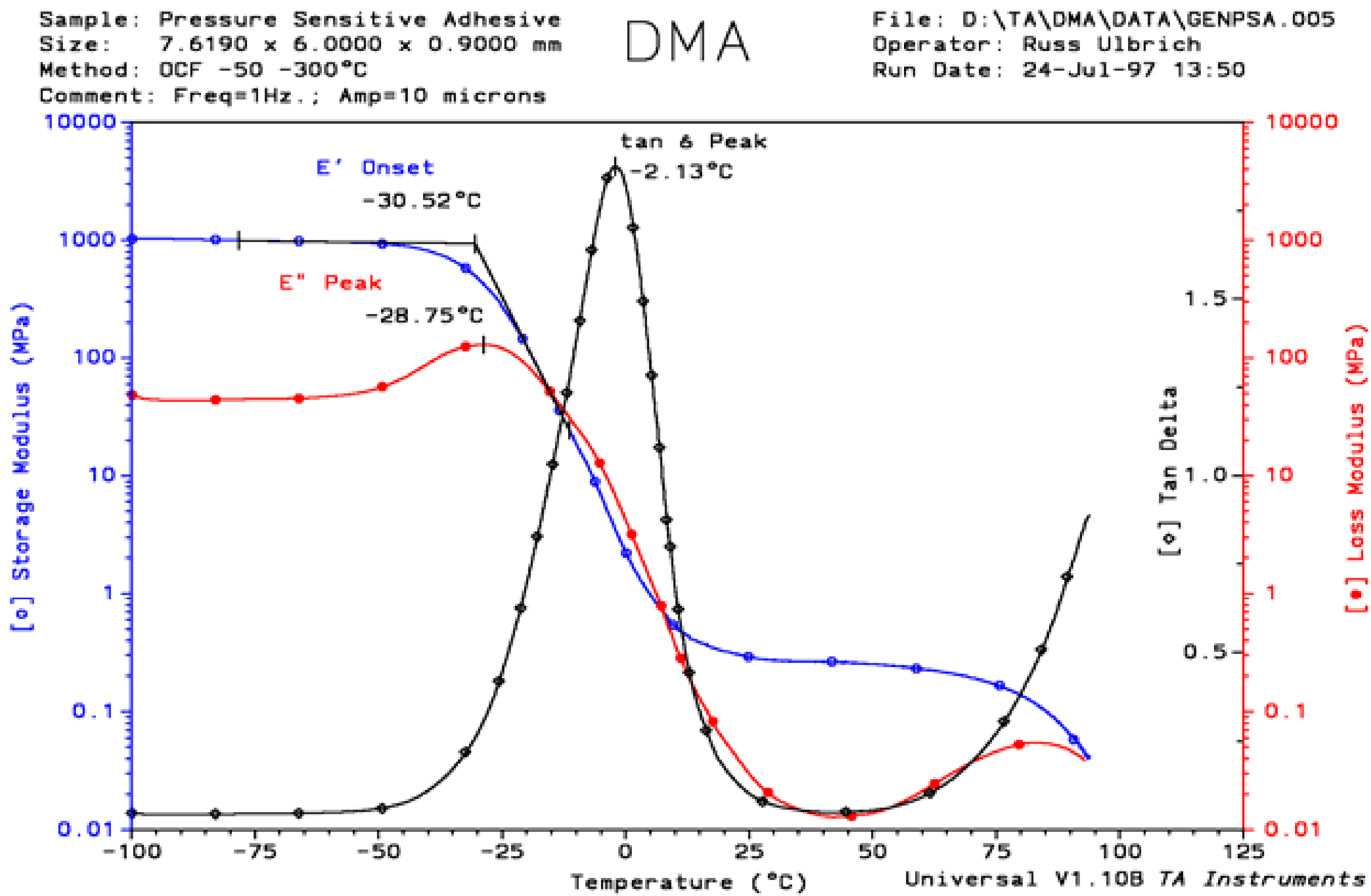

PC sample



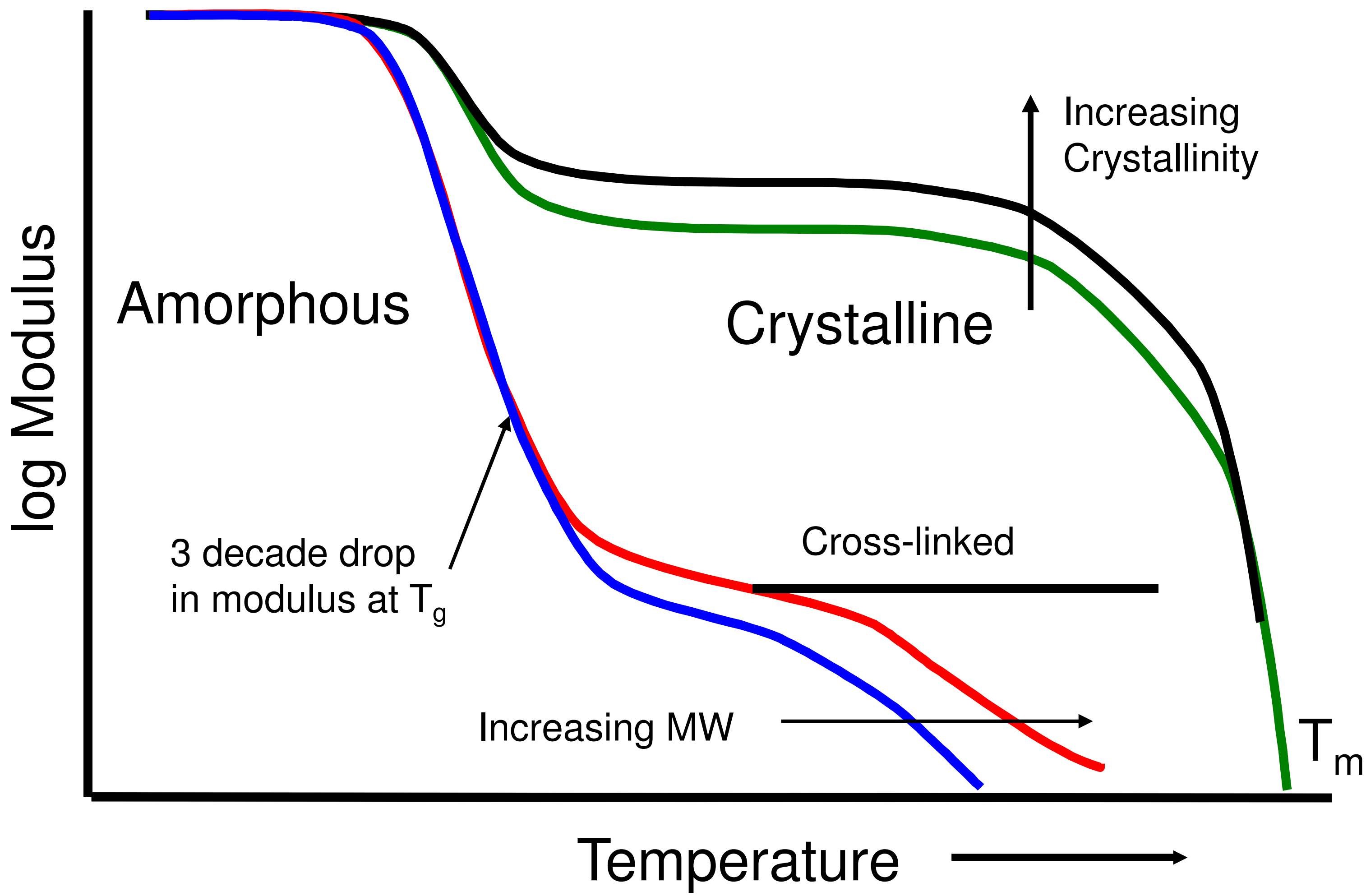
p/n: 982165.903

Clamp:
single cantilever
Temperature:
ambient to 180℃
Heating rate: 3℃/min
Frequency: 1 Hz
Amplitude: 20 µm

# Glass Transition is a Range, not a Temperature

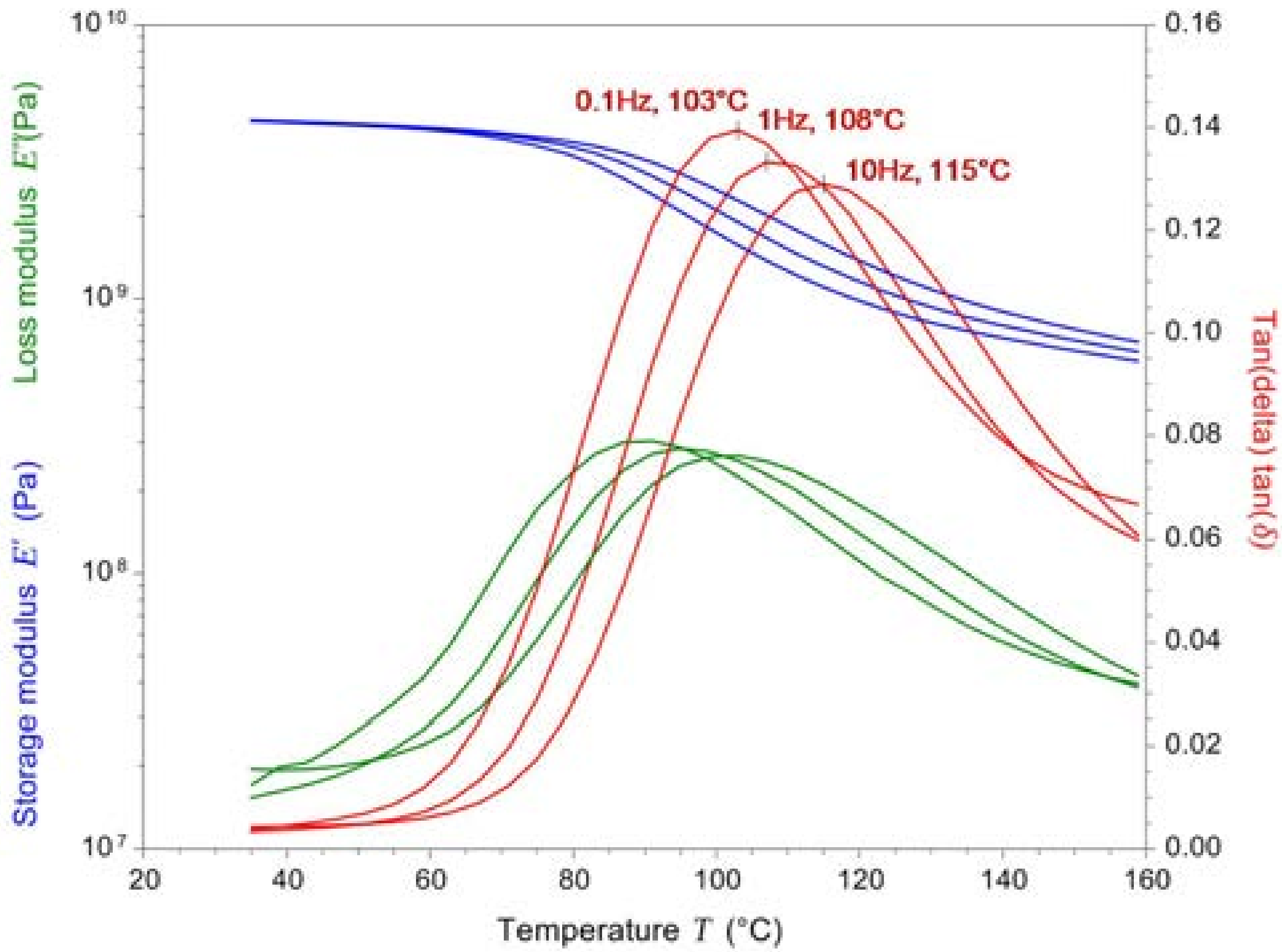


# Crystallinity, Molecular Weight, and Crosslinking



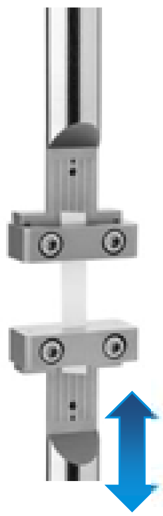
# PET Film: Effect of Frequency on Tg

- PET film tested at 0.1 Hz, 1Hz and 10 Hz



# DMA Deformation Modes

## Axial



### Tension

- Thin Films
- Fibers

### Compression

- Soft solids
- Foams
- Gels

### Bending

- Thermosets
- Composites

## Rotational

### Parallel Plate

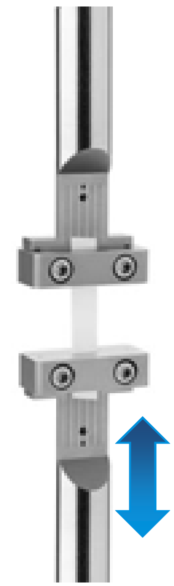
- Thermoplastic
- Thermosets
- Elastomers

### Torsion

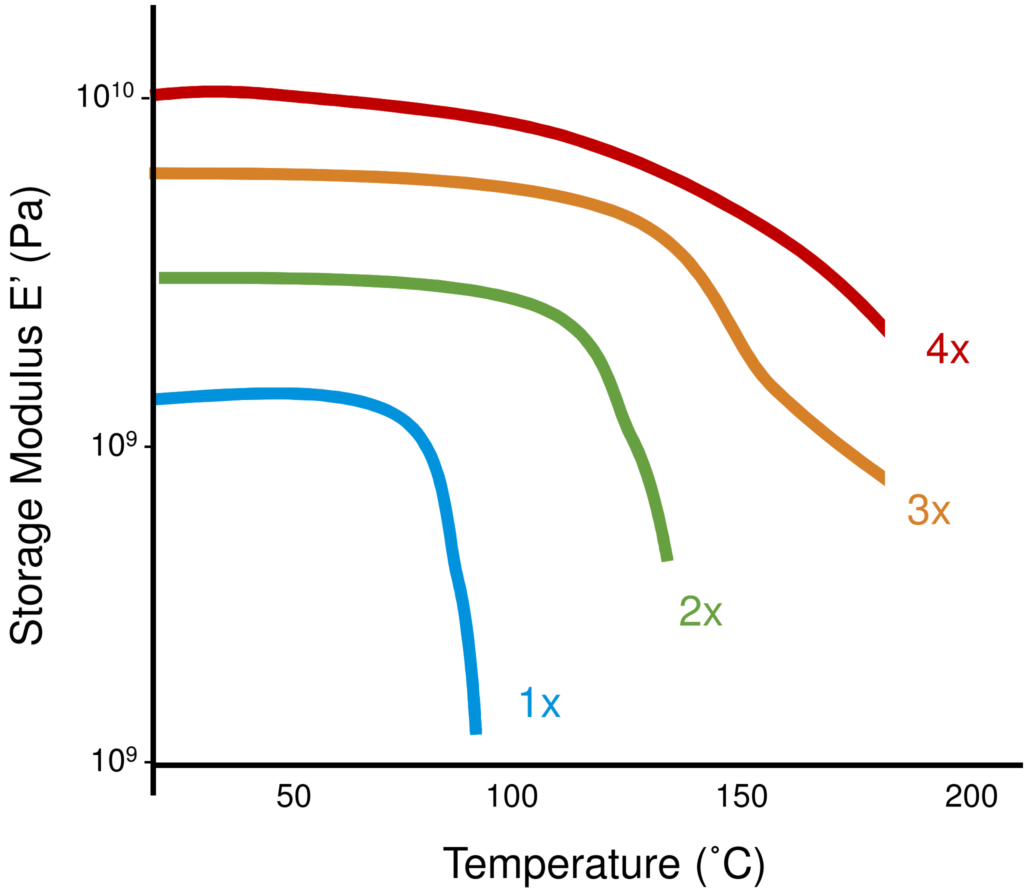
- Thermoplastic
- Thermosets
- Composites

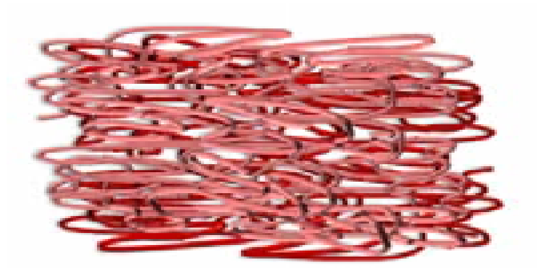
# Tension DMA

- **Young's Modulus (E)**
  - Easy to adjust clamps to accommodate different samples. Allows for thermal expansion or shrinkage.
- **Dimensions:**
  - Length can be adjusted directly, measured by instrument.
  - Thickness up to 2 mm
- **Materials:**
  - Polymer films (Mylar, Kapton)
  - Elastomers (o-rings, seals)
  - Free films of coatings (dried paint)
  - Fibers, bundled or single.

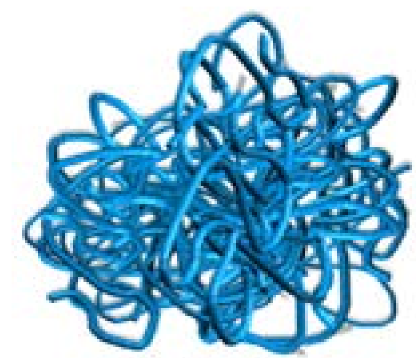


# Storage Modulus of PET Fiber- Draw Ratios



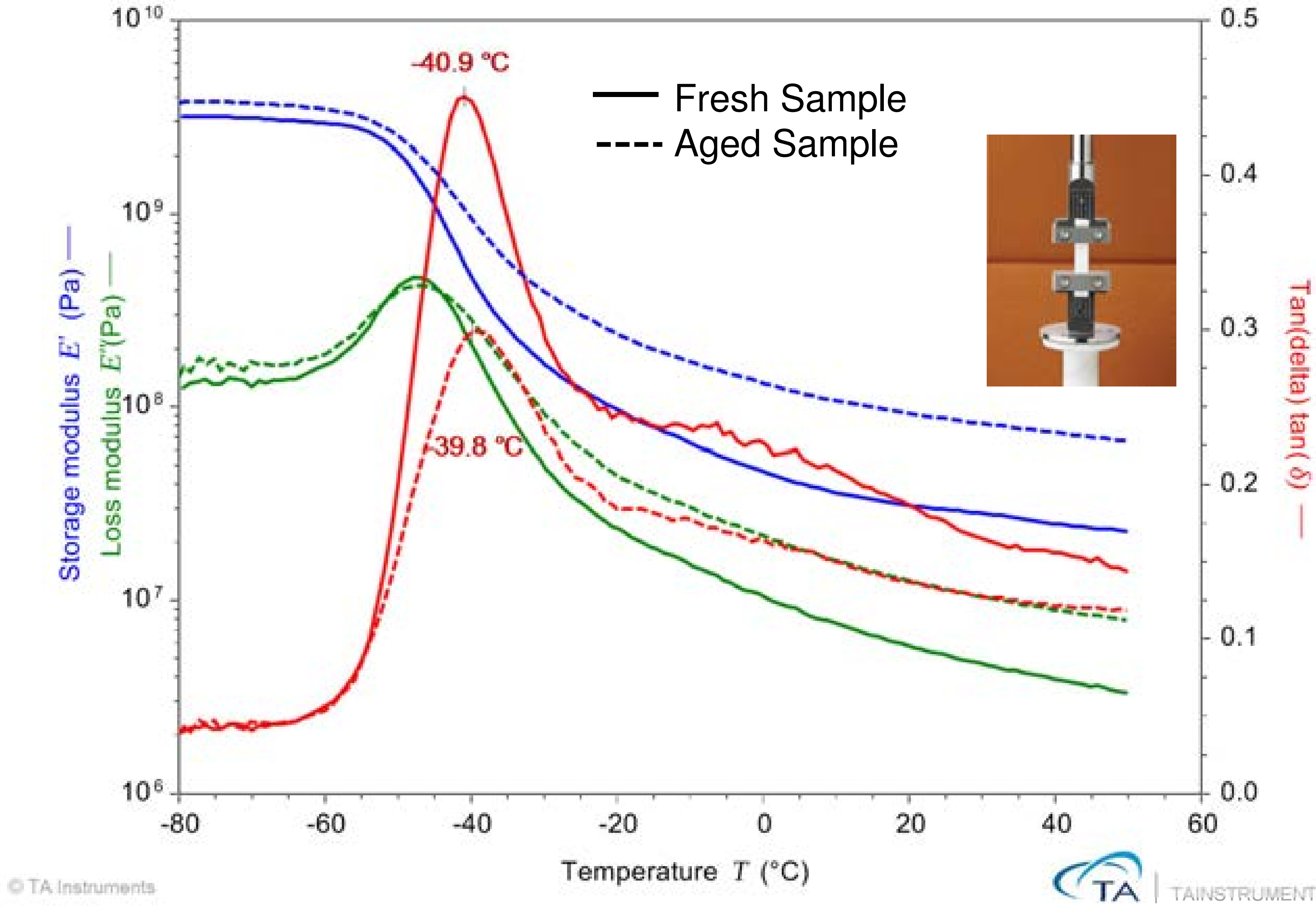
High uniaxial orientation



Random coil- no orientation

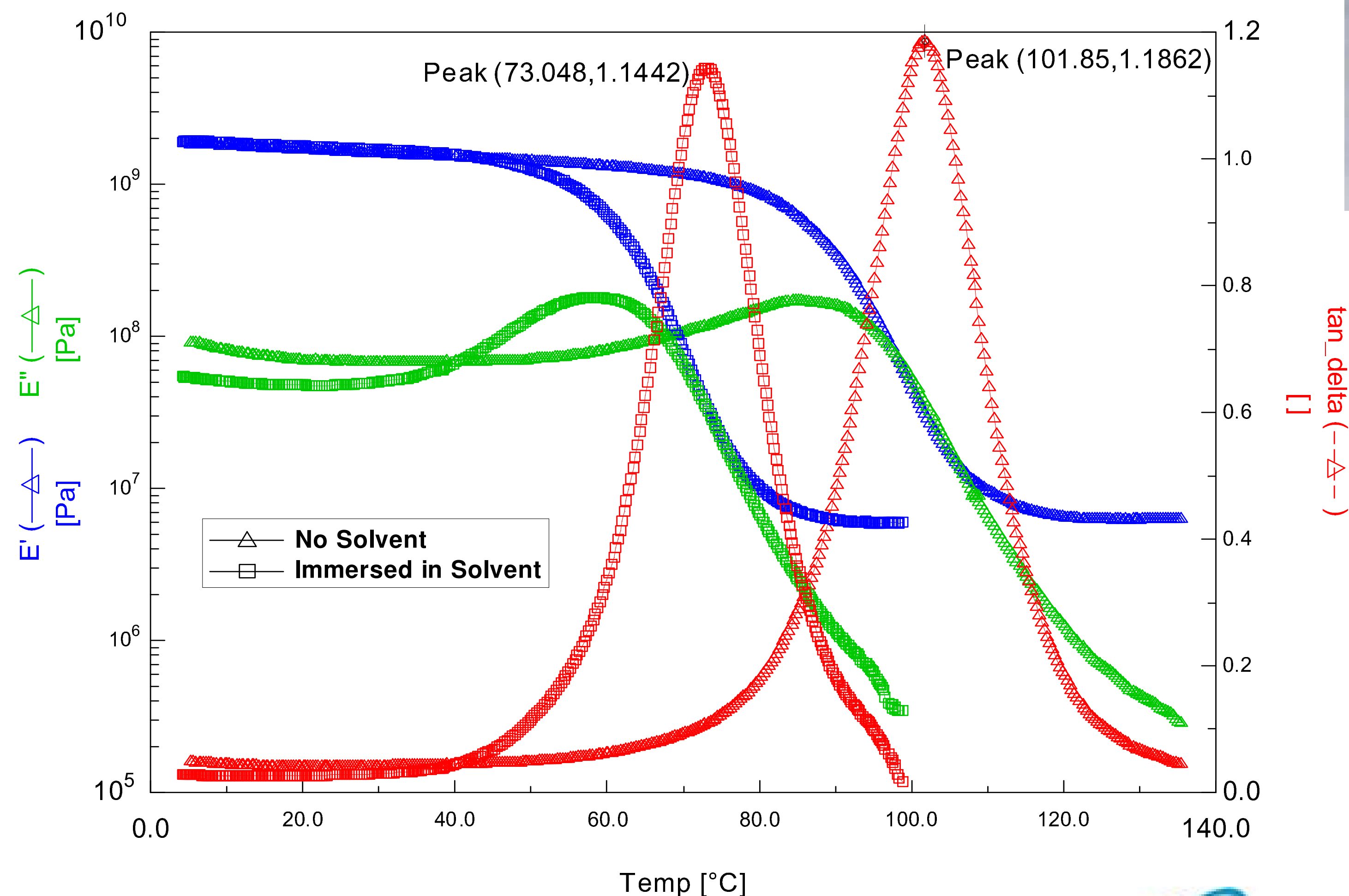

Murayama, Takayuki. "Dynamic Mechanical Analysis of Polymeric Material." Elsevier Scientific, 1978. pp. 80.

# Glass Transition of EPDM- DHR DMA Tension

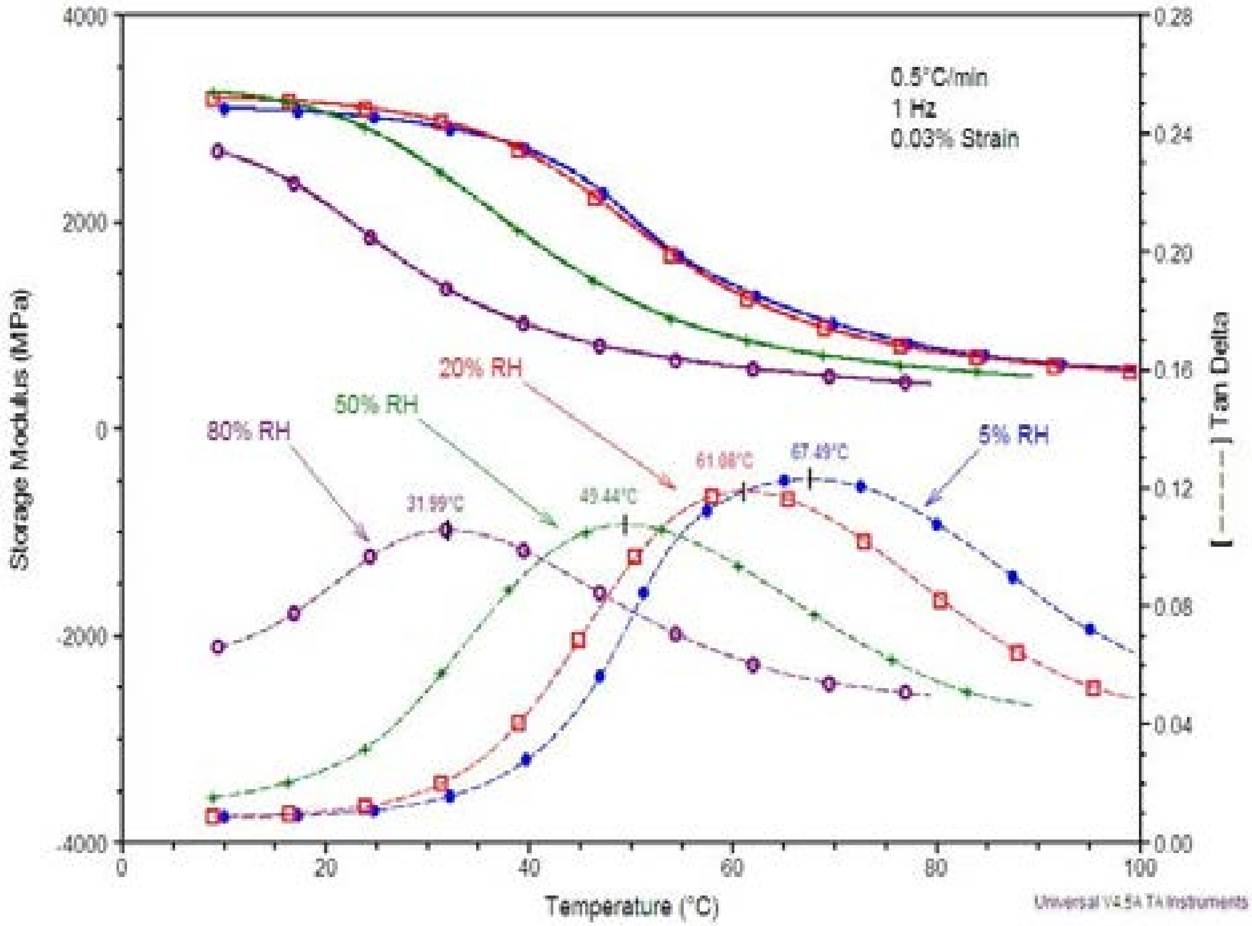


# Effect of Solvent

- Automotive coating measured with and without solvent



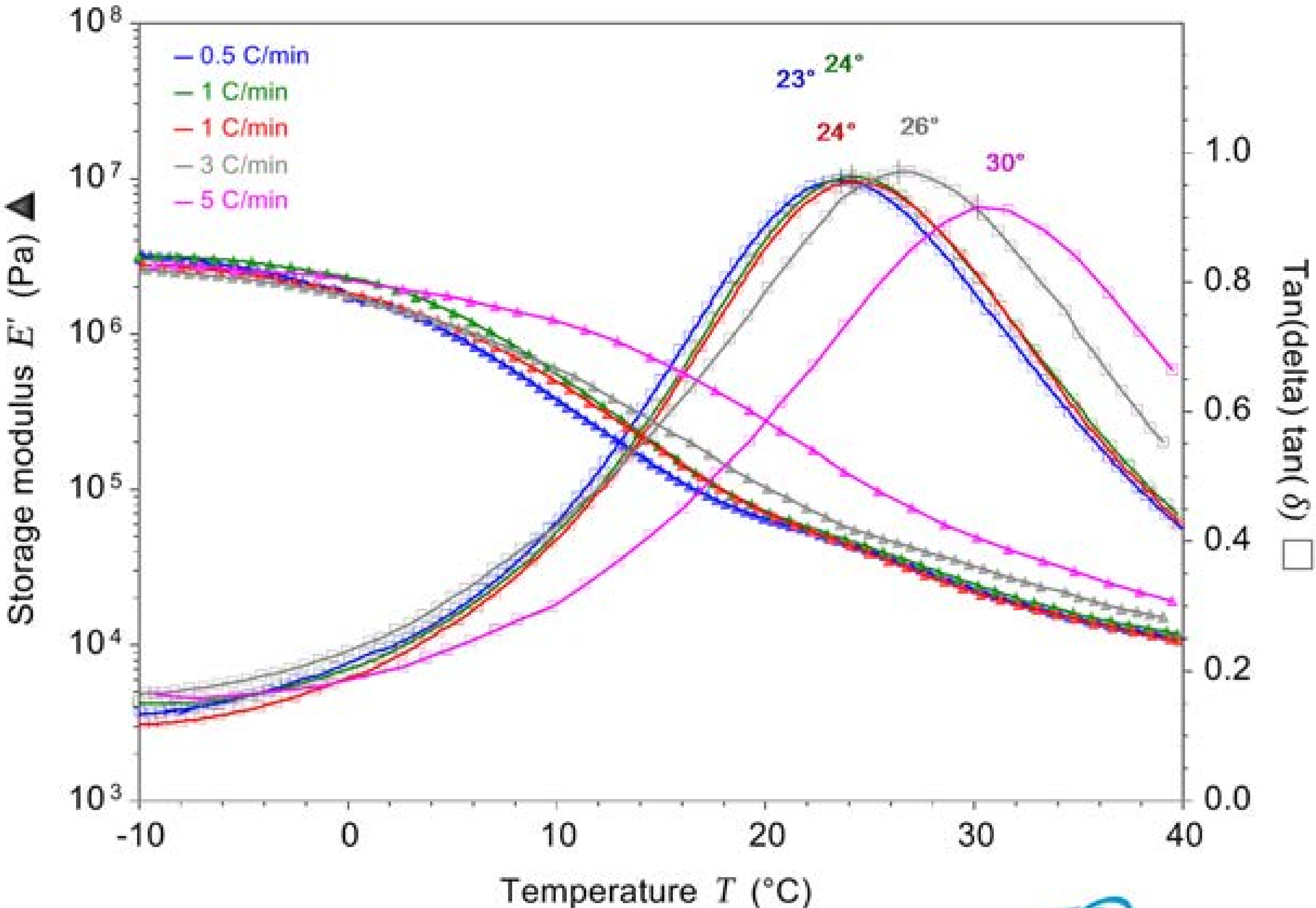

# Humidity Influence: Nylon Film



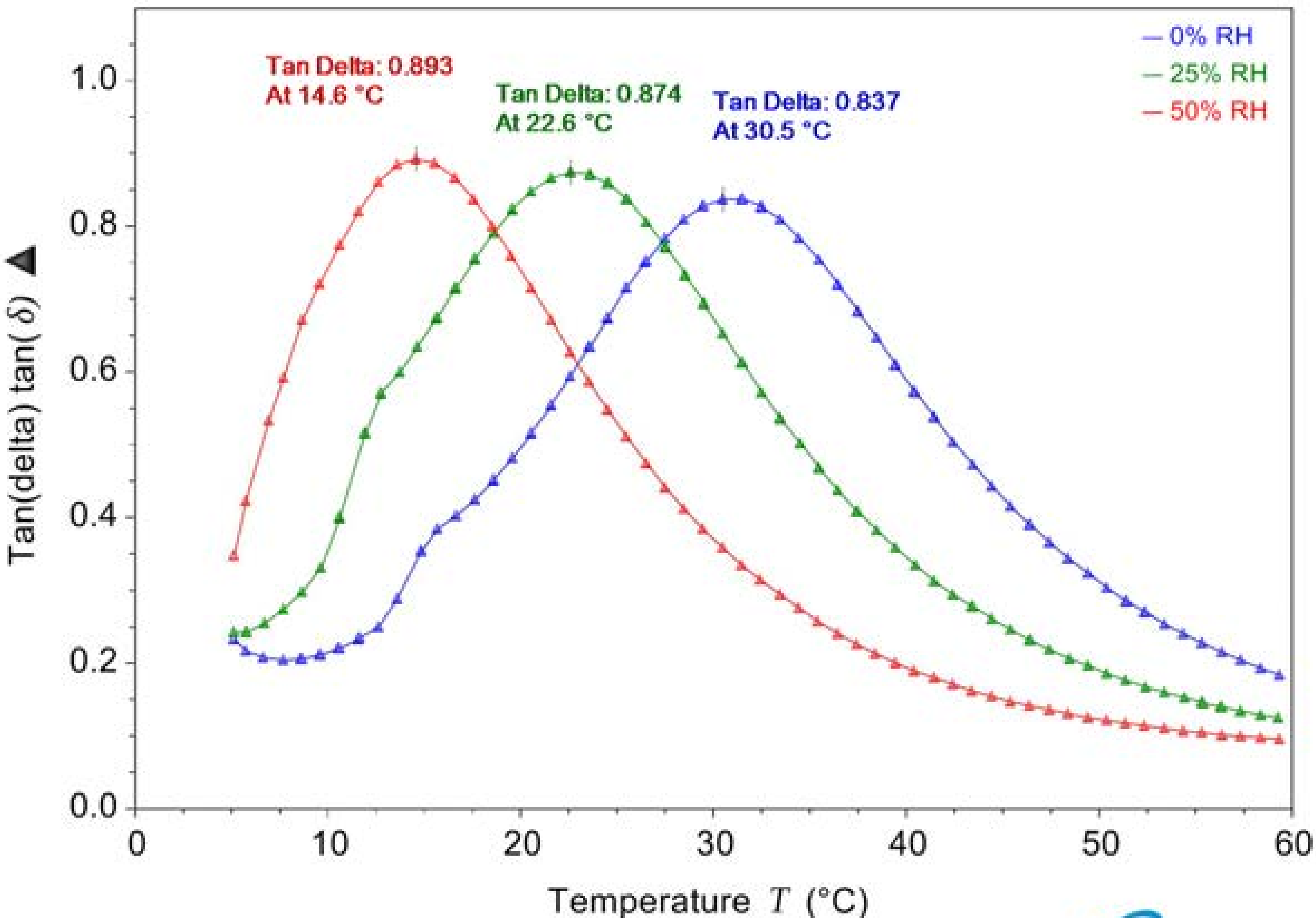

# Compression DMA

- Compression Modulus (E)
- Soft materials with high elasticity.
- Must be compressible, without yielding under deformation.
- Dimensions:
  - Ideally cut to diameter of the plates.  Can also accommodate smaller disks or rectangles.
  - 1-10 mm thick
- Materials:
  - Foams (mattress, packaging, anti-vibration)
  - Soft Elastomers (above Tg only!)
  - Stiff hydrogels, biological tissue

# **Foam Compression DMA:** Temperature Ramp Rate

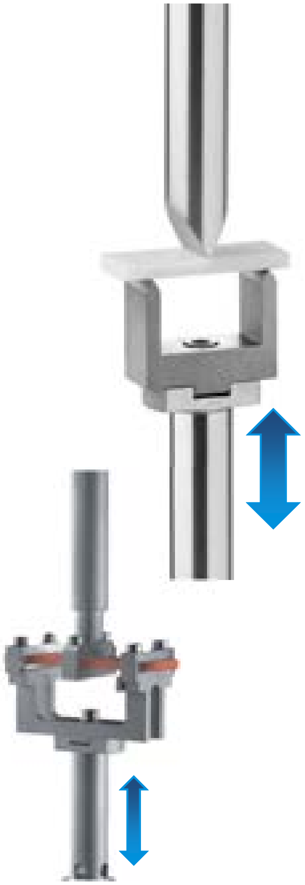


# Effects of Humidity on Glass Transition of Foam



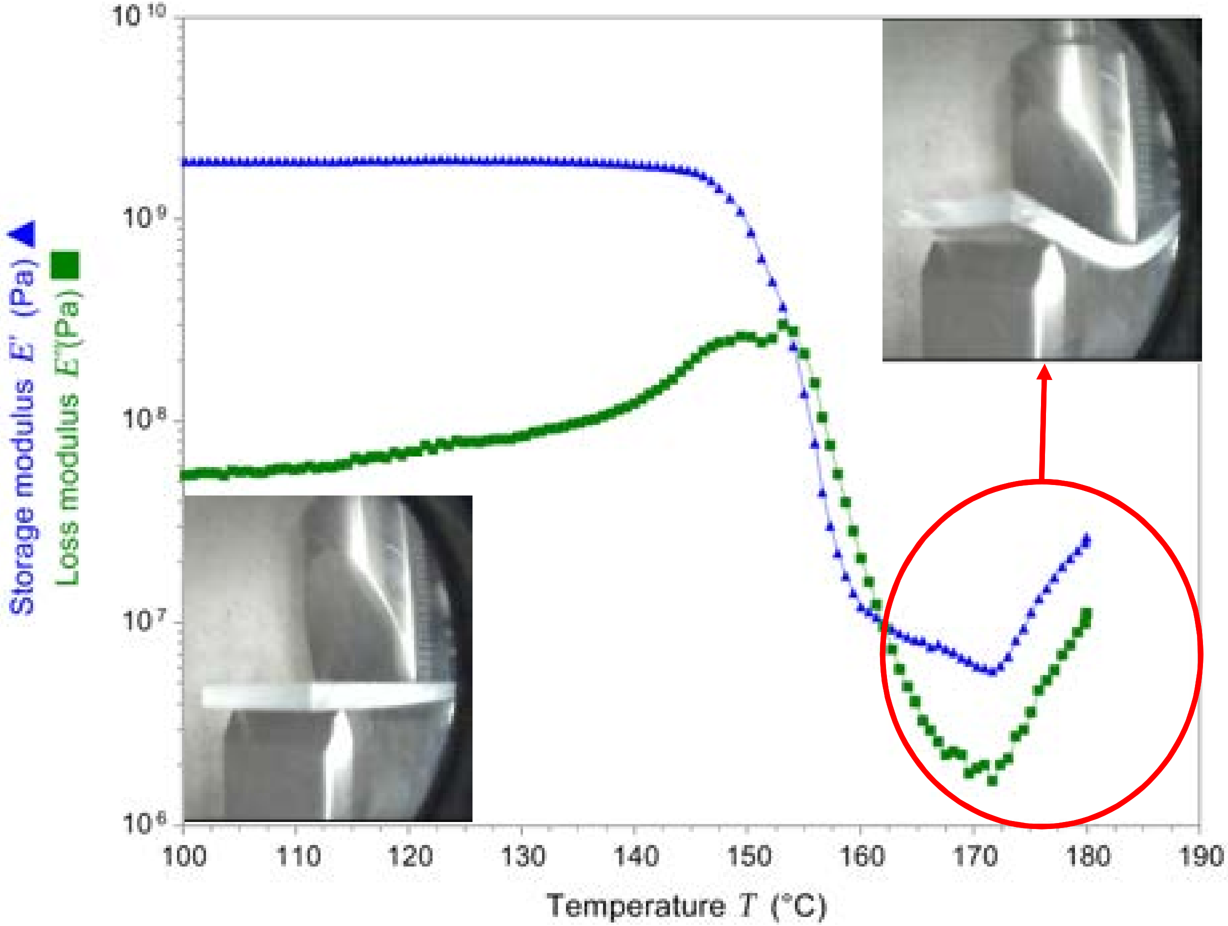
# Bending DMA

- **Flexural Modulus (E)**
- 3 Point Bend (unclamped) and Cantilever (clamped)
- **Dimensions**
  - Fixed lengths: (i.e. 40, 25 and 10 mm 3PB)
  - Width up to 12.5 mm
  - Thickness ideally less than 1/10 length.
- **Materials**
  - Unfilled thermoplastics (Cantilever only > Tg)
  - Elastomers (Cantilever)
  - Thermosets (3PB)
  - Composites (3PB)
  - Metals (3PB)



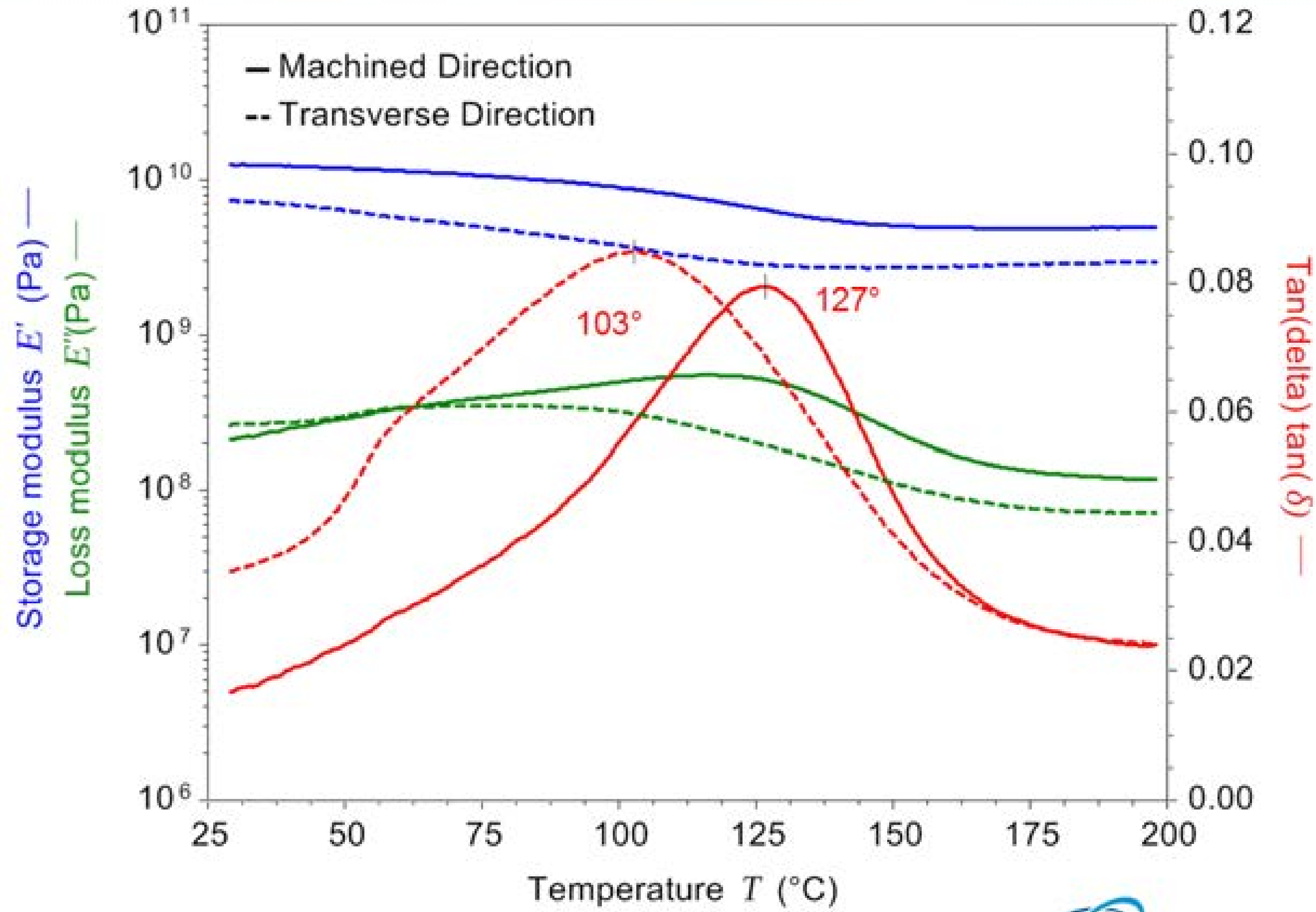
# What Causes E' Increase after Tg?

- Sample sagging after Tg
- Solution: use cantilever clamp instead of 3-p bending



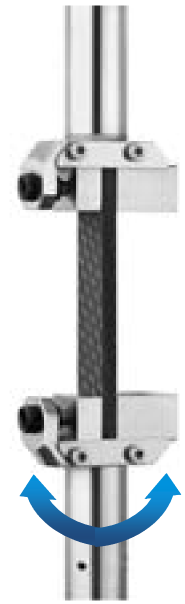

Instrument: RSA G2
Clamp: 3-p bending
Temperature:
50℃ to 180℃
Heating rate: 3℃/min
Frequency: 1Hz
Amplitude: 10 μm

# Fiber Reinforced Polymer- 3 Point Bending

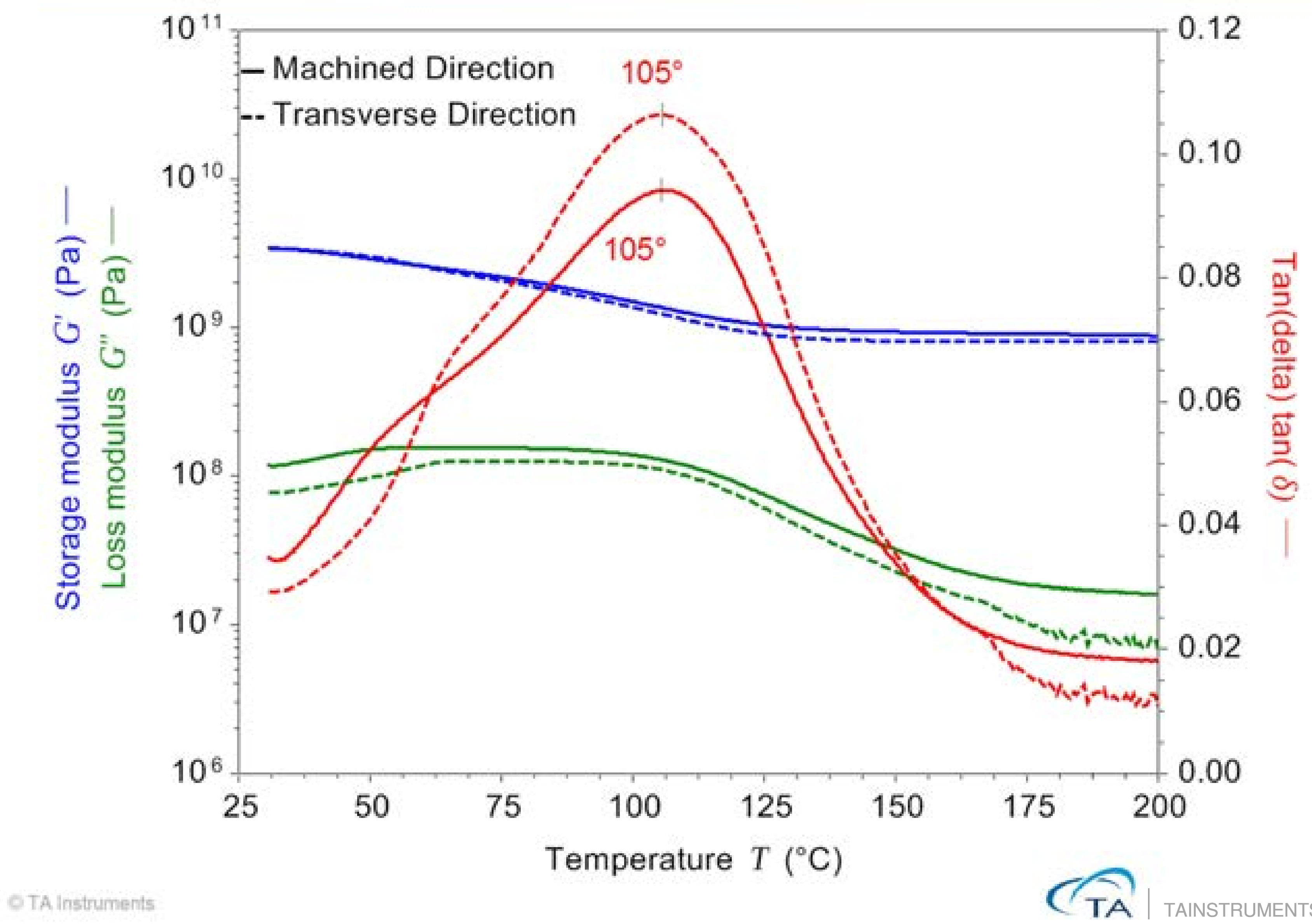


# Torsion DMA

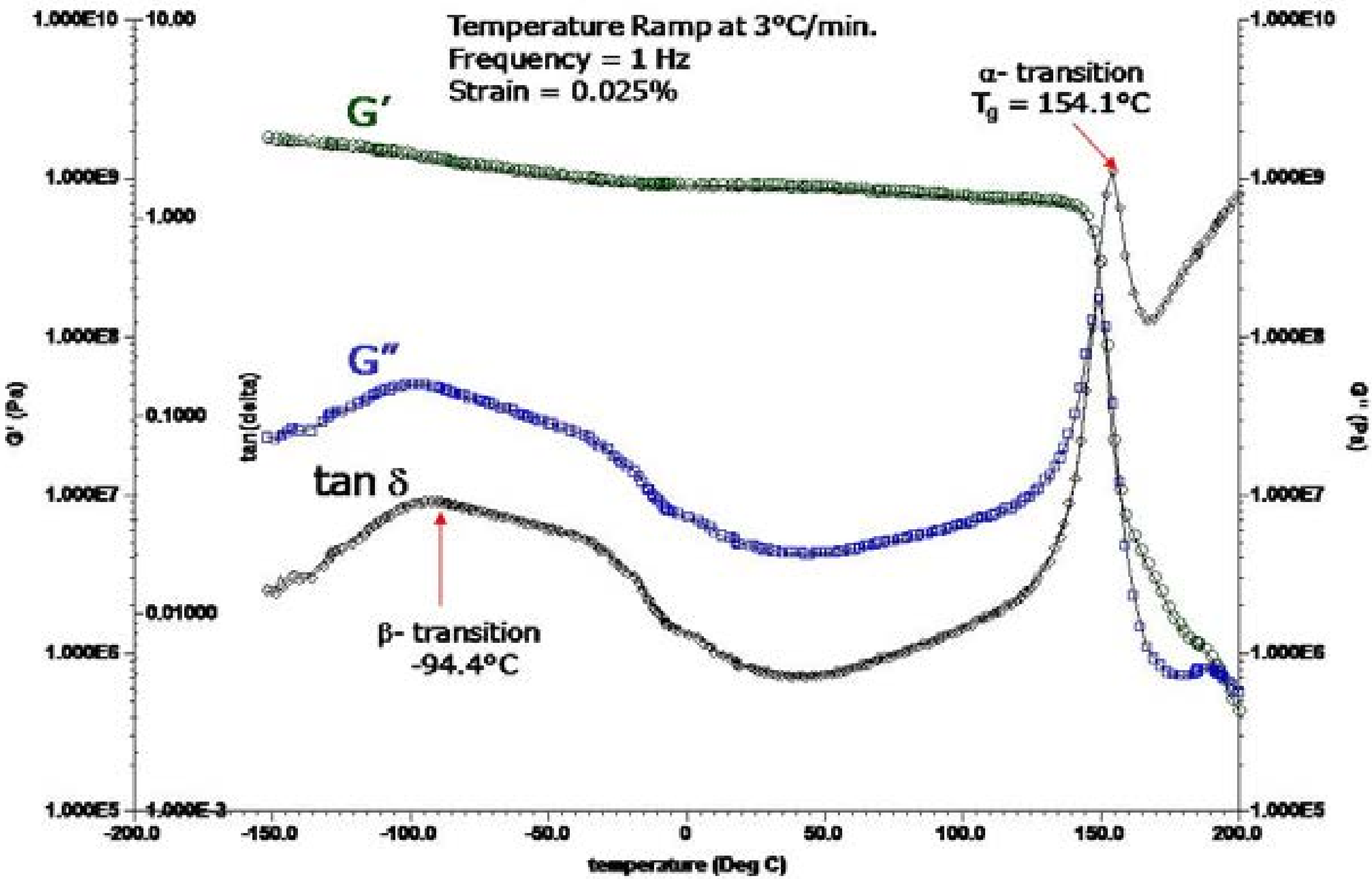
- **Shear Modulus (G)** “Modulus of Rigidity”
- Ideal for very high modulus materials; accommodates wide range of dimensions.
- **Dimensions:**
  - Small: 7 mm long, 3 mm wide, 0.5 mm thick
  - Large: 40 mm long, 12.5 mm wide, 4 mm thick
  - Cylinder: 1.5, 3 or 4.5 mm diameter
- **Materials:**
  - Thermoplastics and Thermosets
  - Elastomers
  - Composites
  - Metals



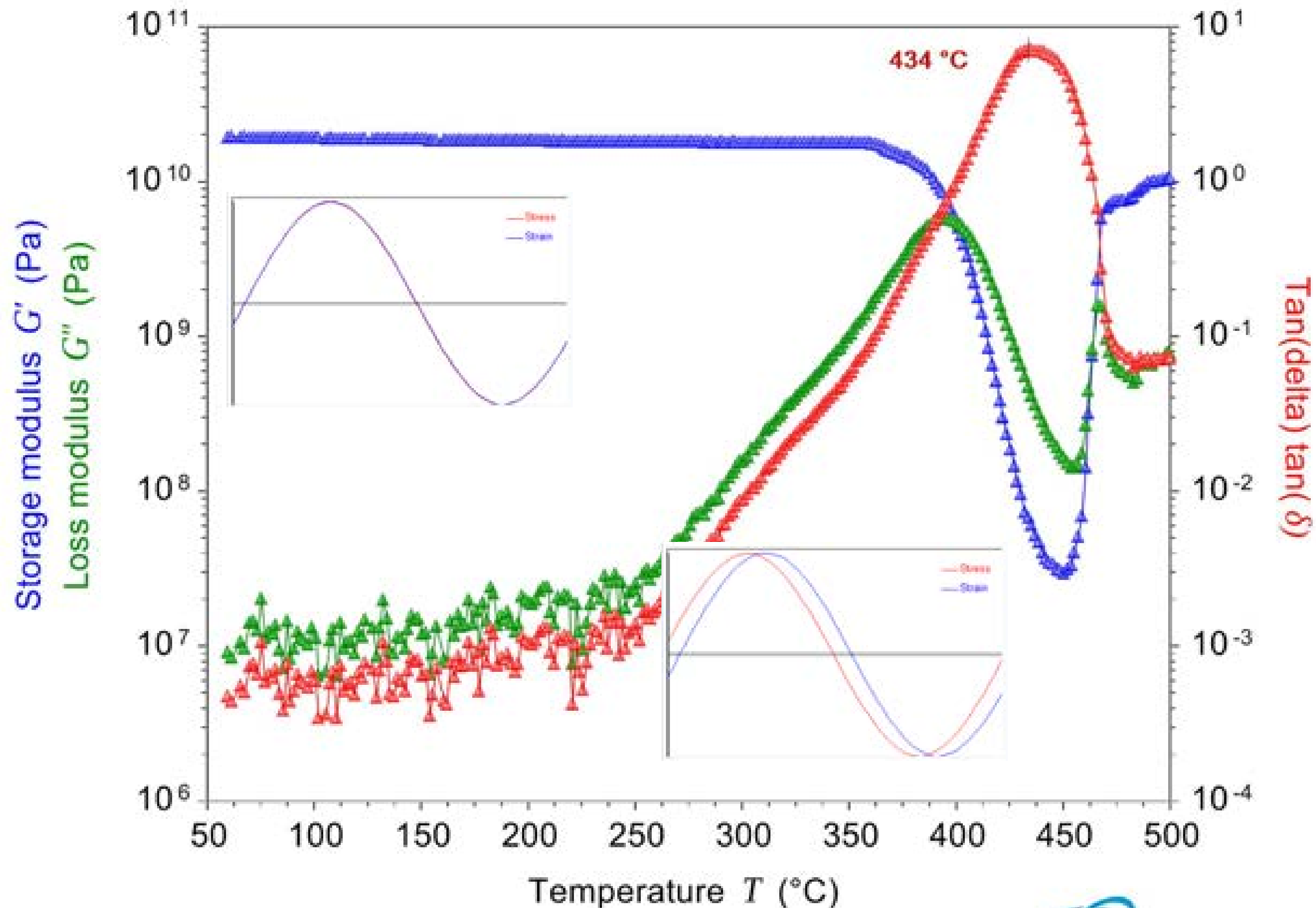
# Fiber Reinforced Polymer- Torsion



# Torsion DMA - Beta Transition

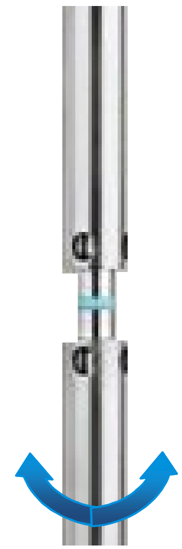


# Torsion: Metallic Glass (Amorphous Metal)

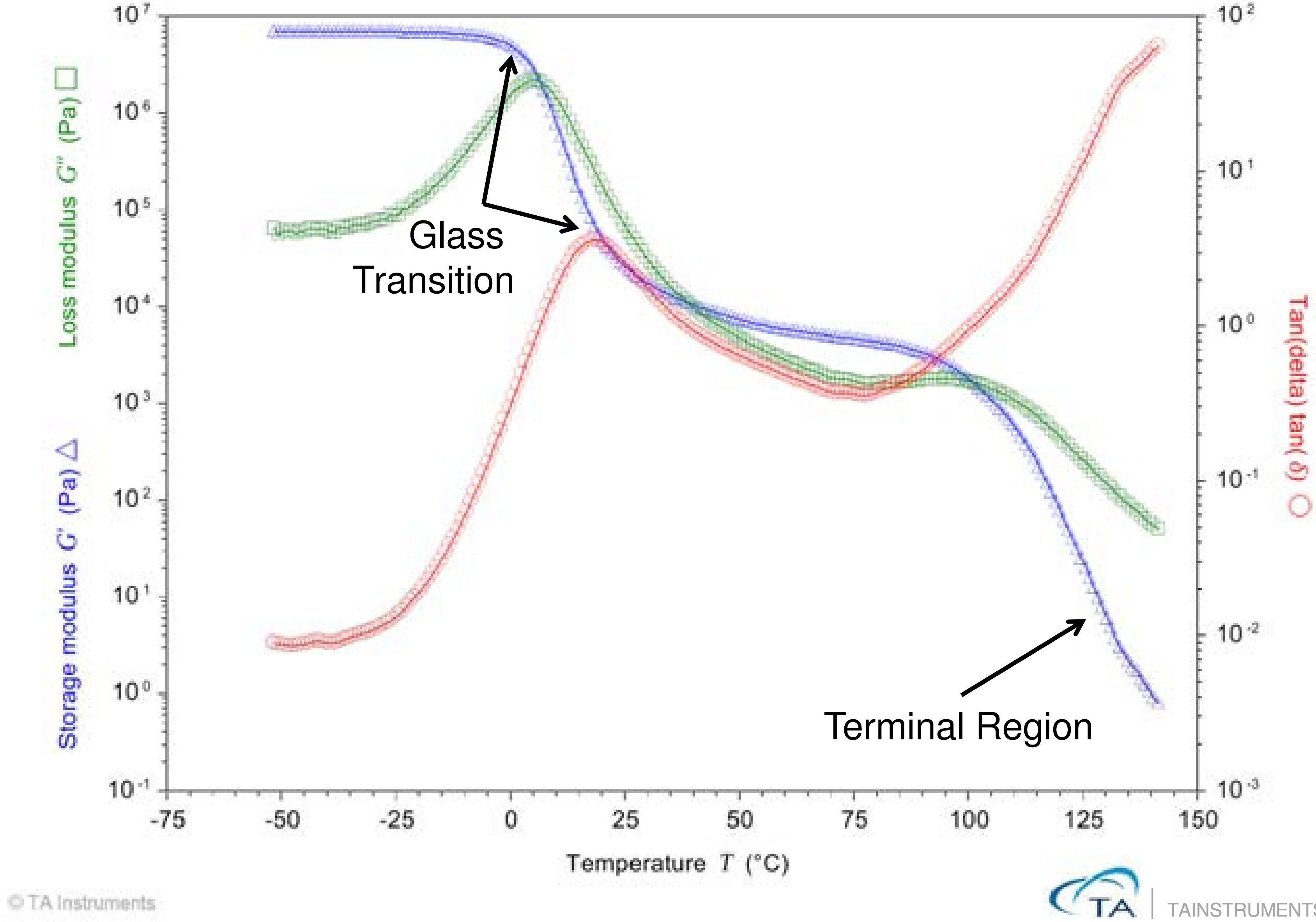


# Parallel Plate DMA

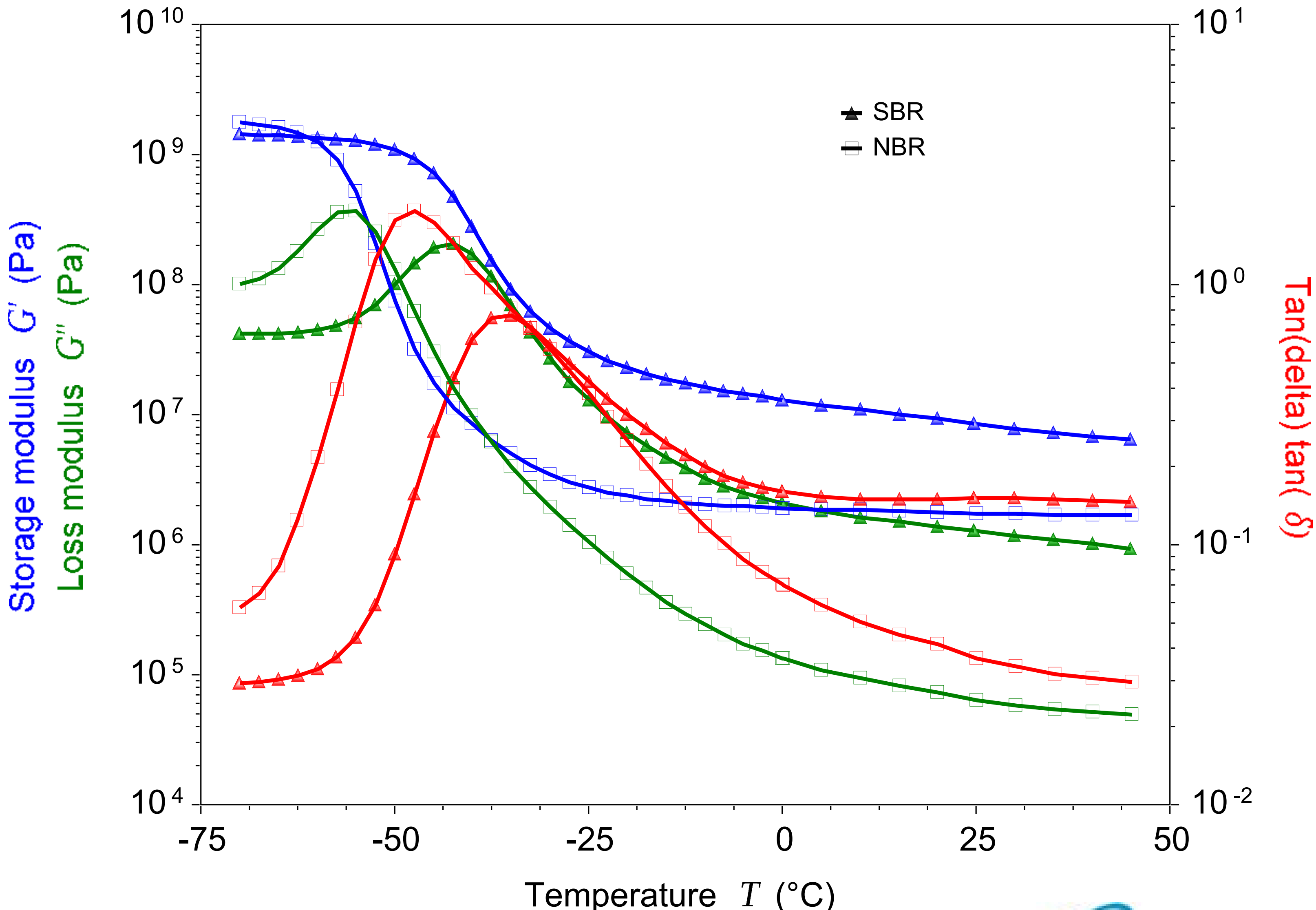
- **Shear Modulus (G)**
- Full range of viscoelastic behavior (glassy, rubbery and terminal region).
- **Dimensions:**
  - 25, 8 or 4 mm parallel plates
  - 0.5 – 3 mm gap thickness
- **Materials:**
  - Thermoplastics: load above softening point, ramp temperature down.
  - Thermosets: cure in place on disposable plates.
  - Elastomers: cut disk and glue to plates.
  - Adhesives: too soft to test with linear DMA



# Hot Melt Adhesive: Parallel Plates



# Rubber DMA: Parallel Plates



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- Thermal Conductivity
- Mechanical Testers
- Dynamic Mechanical Analyzers
- **Rotational Rheometers**
- Rubber Rheometers

# Rotational Rheometers

# What is Rheology?





Rheology: The study of
stress-deformation relationships





# Rheology: the study of flow and deformation

## Viscosity

- Non-Newtonian Viscosity
  - Shear thinning
  - Shear thickening
  - Thixotropy
  - Yield Stress
- Viscosity under processing conditions

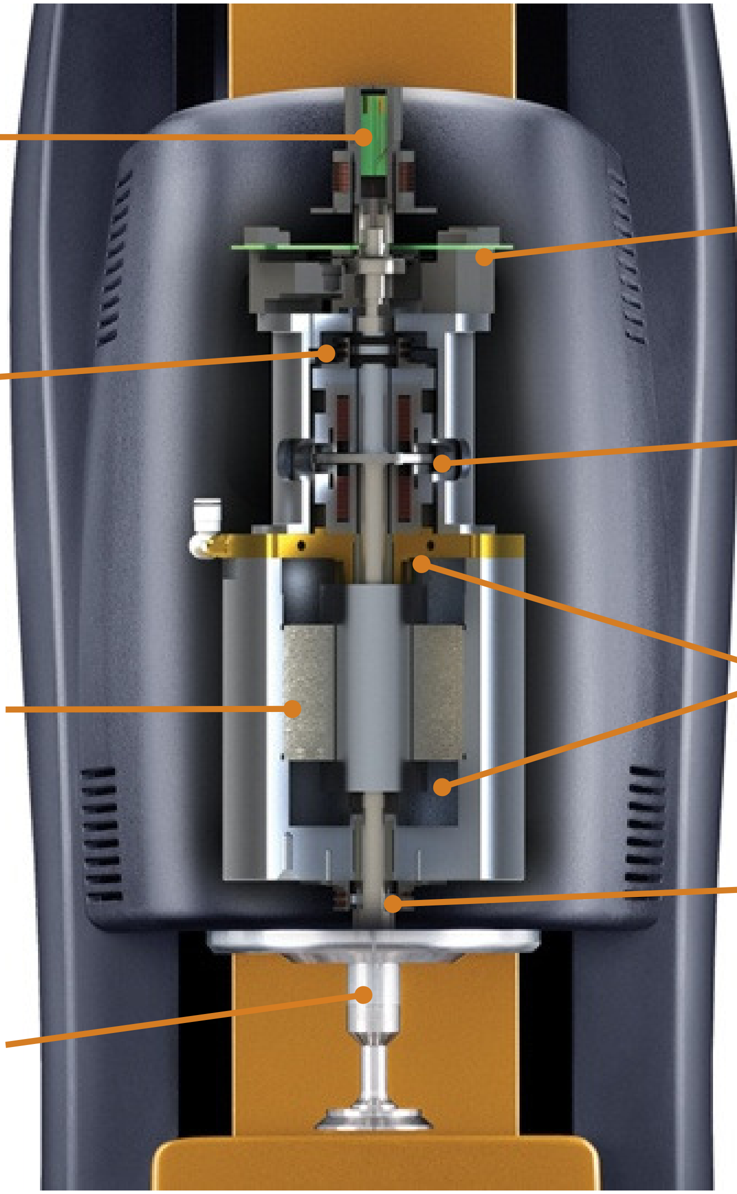
$$\frac{\text{Stress}}{\text{Shear rate}} = \text{Viscosity}$$

## Modulus

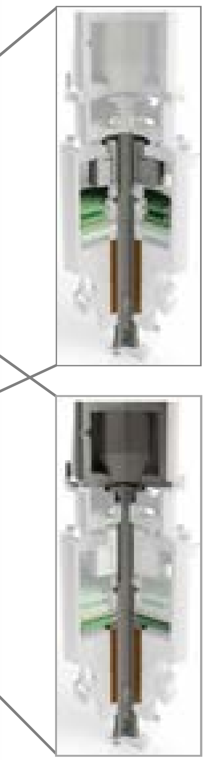
- Measure viscoelastic properties
  - Storage Modulus
  - Loss Modulus
  - Tan Delta
- Changes with time, temperature

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

# Discovery Hybrid Rheometer Technology



# ARES G2: Separate Motor and Transducer



Torque Transducer maintains the null position as the sample is deformed.

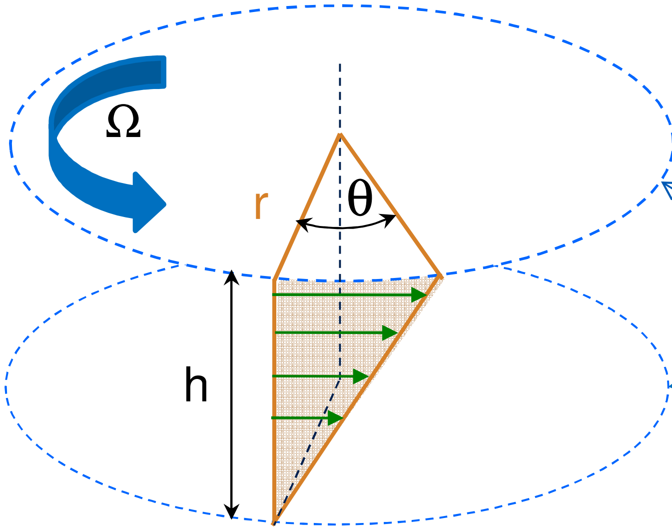
Sample torque to be measured directly, without contributions from motor friction or inertia.

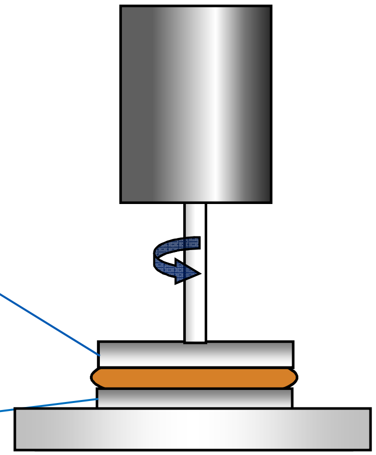
Normal Force Transducer provides highly accurate normal force measurements

High stiffness for precise gap control.

Direct Drive motor applies accurate and precise rotational deformation, without contributing to measured torque.

# Torsion Flow in Parallel Plates



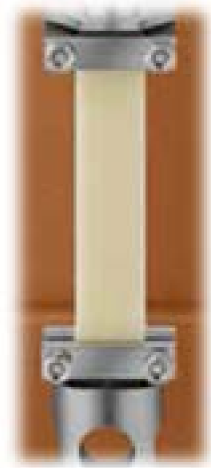
r = plate radius
h = distance between plates
M = torque (μN.m)
θ = Angular motor deflection (radians)
Ω= Motor angular velocity (rad/s)

**Stress (σ)** $\sigma = \frac{2}{\pi r^3} \times M$

**Strain (γ)** $\gamma = \frac{r}{h} \times \theta$

**Strain rate ($\dot{\gamma}$)** $\dot{\gamma} = \frac{r}{h} \times \Omega$

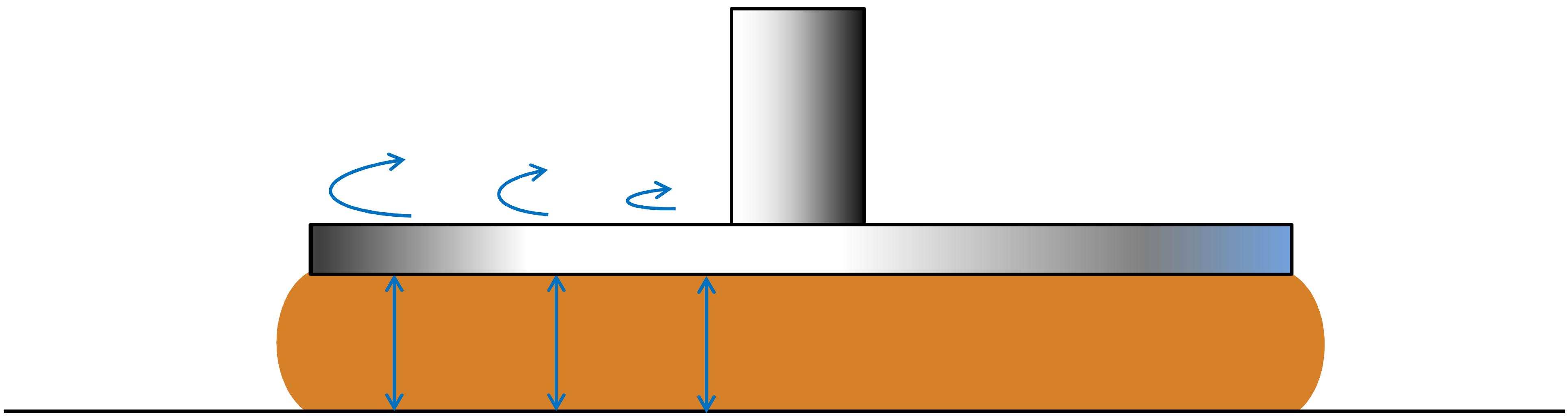
# Geometry Options

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

Water → to → Steel

# Shear Rate varies across a Parallel Plate

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



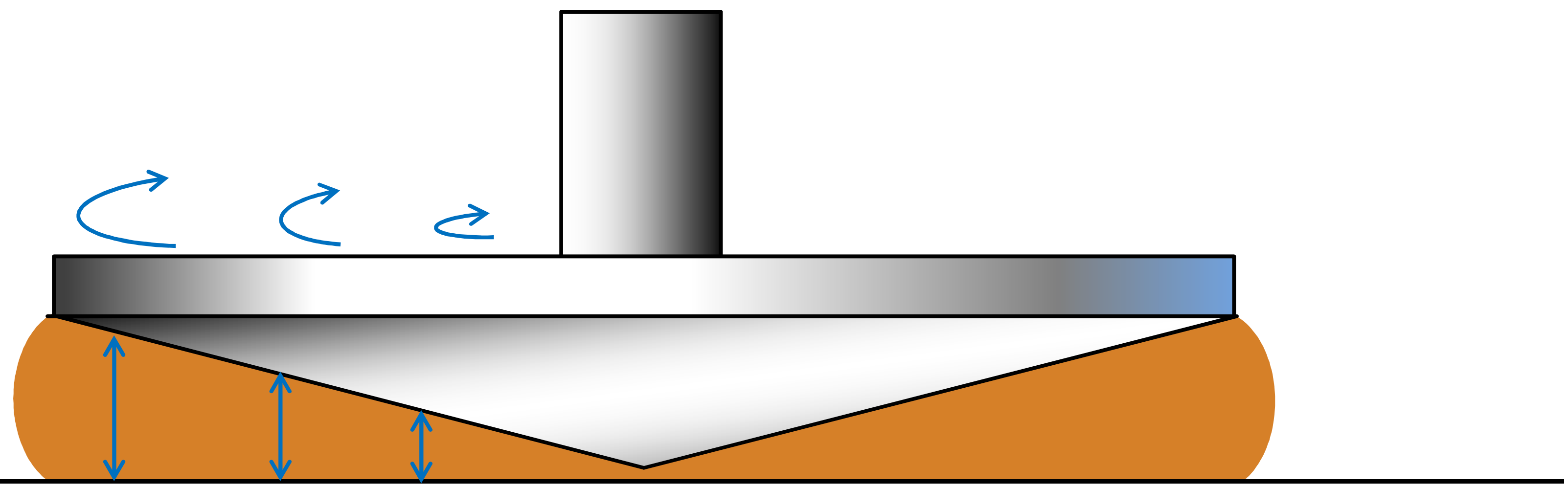
$$\gamma = \frac{dx}{h}$$

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

Single-point correction for the parallel plate geometry (0.76 radius)
[M.S. Carvalho, M. Padmanabhan and C.W. Macosko, *J. Rheol.* 38 (1994) 1925-1936]

# Shear Rate is Uniform across a Cone

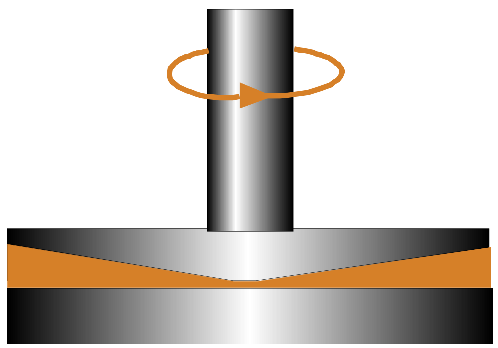
- The cone shape produces a smaller gap height closer to the center, so the shear on the sample is constant



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

*h* increases proportionally to *dx*, $\gamma$ is uniform

# Limitations of Cone and Plate



Cone & Plate

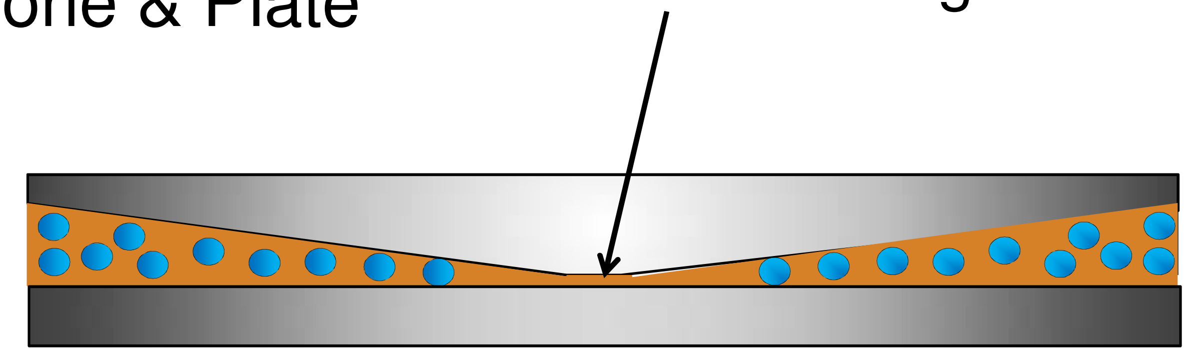
Typical Truncation Heights:

1° degree ~ 20 - 30 microns

2° degrees ~ 60 microns

4° degrees ~ 120 microns

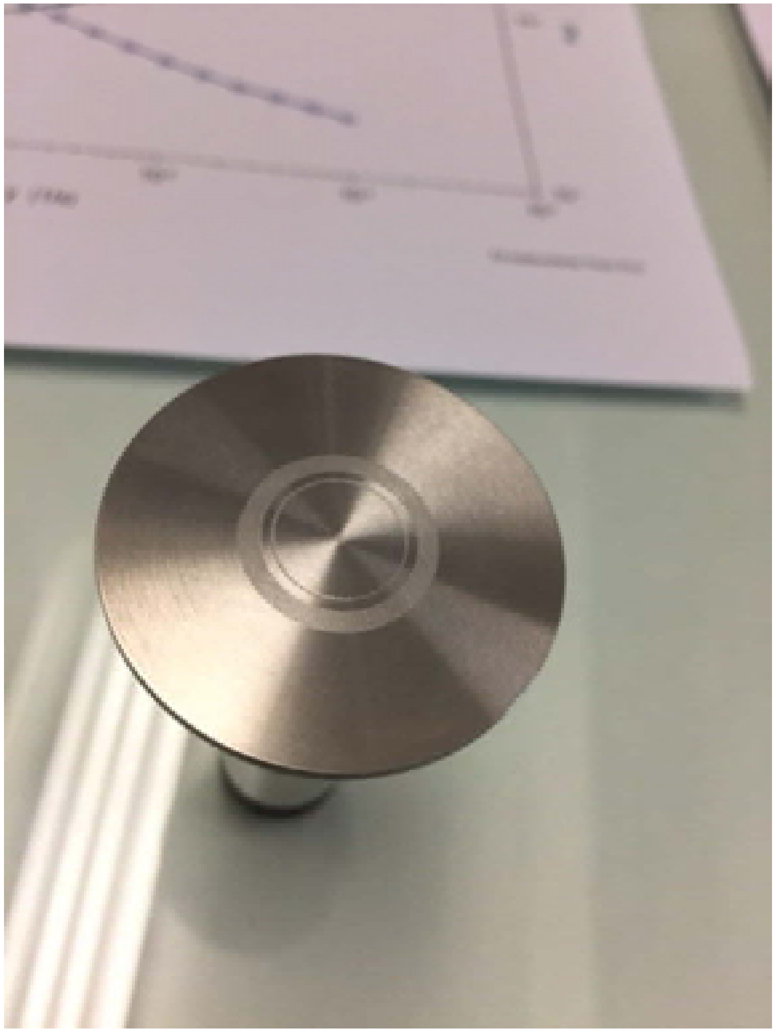
Truncation Height = Gap



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

# What Geometry should we use?

# What Geometry should we use?



# When to Use Concentric Cylinders

**Peltier Concentric Cylinder**

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

# Geometry Summary

| Parallel Plates | Cone and Plate | Concentric Cylinders |
|---|---|---|
| 1-2 mL | 50-500 µL | 7-25 mL |
| Liquids, gels, soft solids, dispersions, etc. | Unfilled liquids, isothermal tests | “Pourable” liquids, low viscosities, dispersions |
| Used for all samples. Roughened surfaces available to prevent slip. | Most accurate measurement of non-Newtonian Viscosity, small sample volume. | Least effected by sample loading technique or evaporation. |

# Polymer Rheology

# Polymer Rheology: Experimental Goals

**Characterization**

- Average Molecular Weight
- Molecular Weight Distribution
- Long-Chain Branching

**Processability**

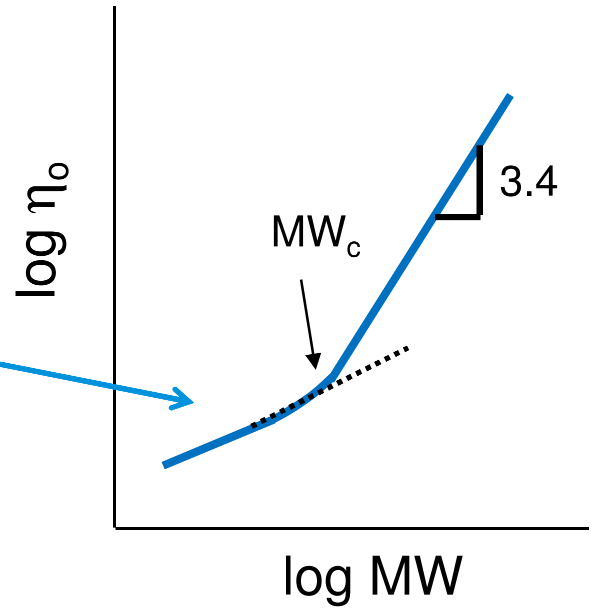
- Shear Thinning
- Die Swell
- Surface Roughness

**Final Product Performance**

- Tack and Peel
- Usable Temperature Range
- Mechanical Properties

# Melt Rheology: MW Effect on Zero Shear Viscosity

- Sensitive to Molecular Weight, MW
- For Low MW (no Entanglements) $\eta_0$ is proportional to MW
- For MW > Critical $MW_c$, $\eta_0$ is proportional to $MW^{3.4}$

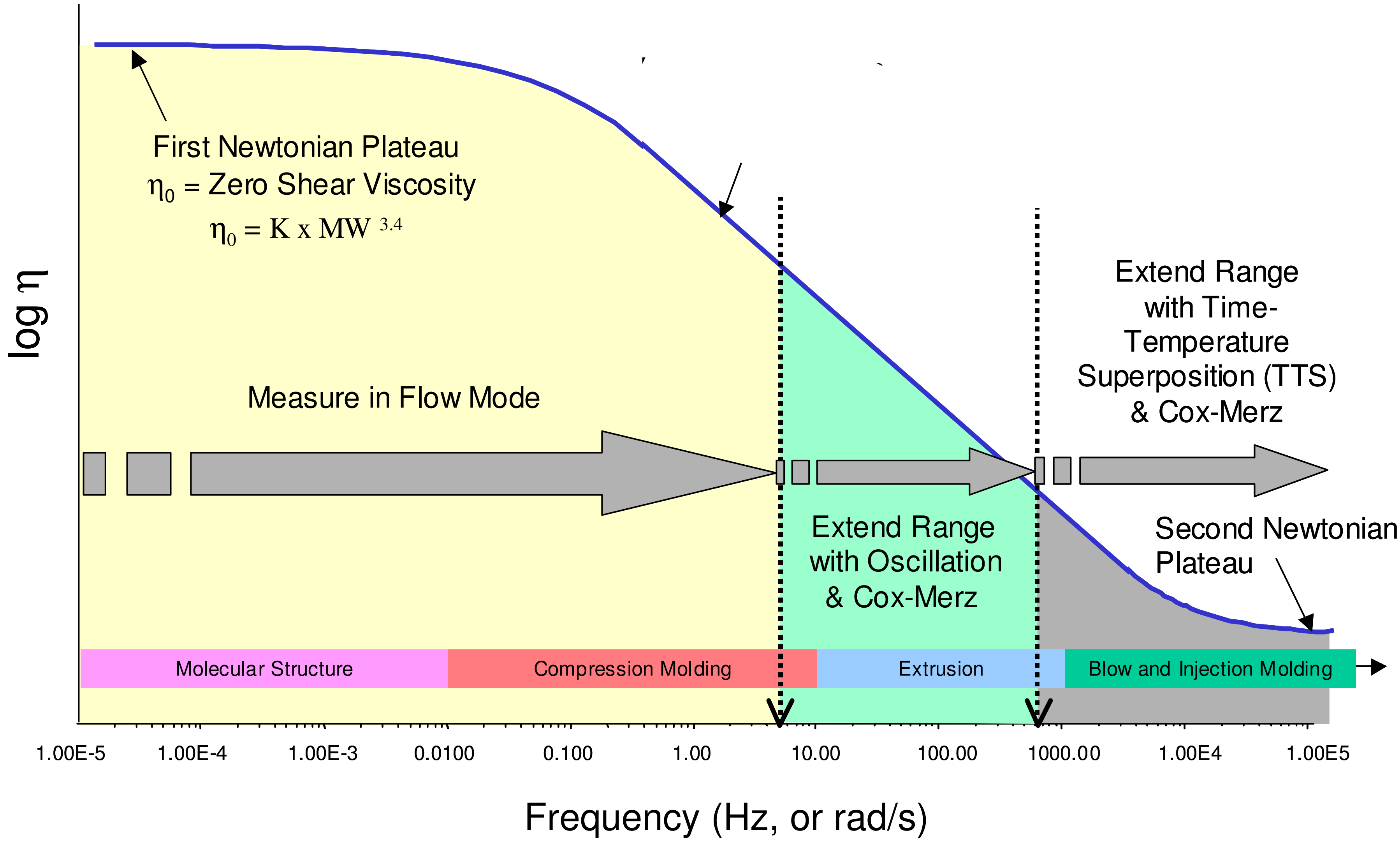



$\eta_0 = K \cdot Mw$

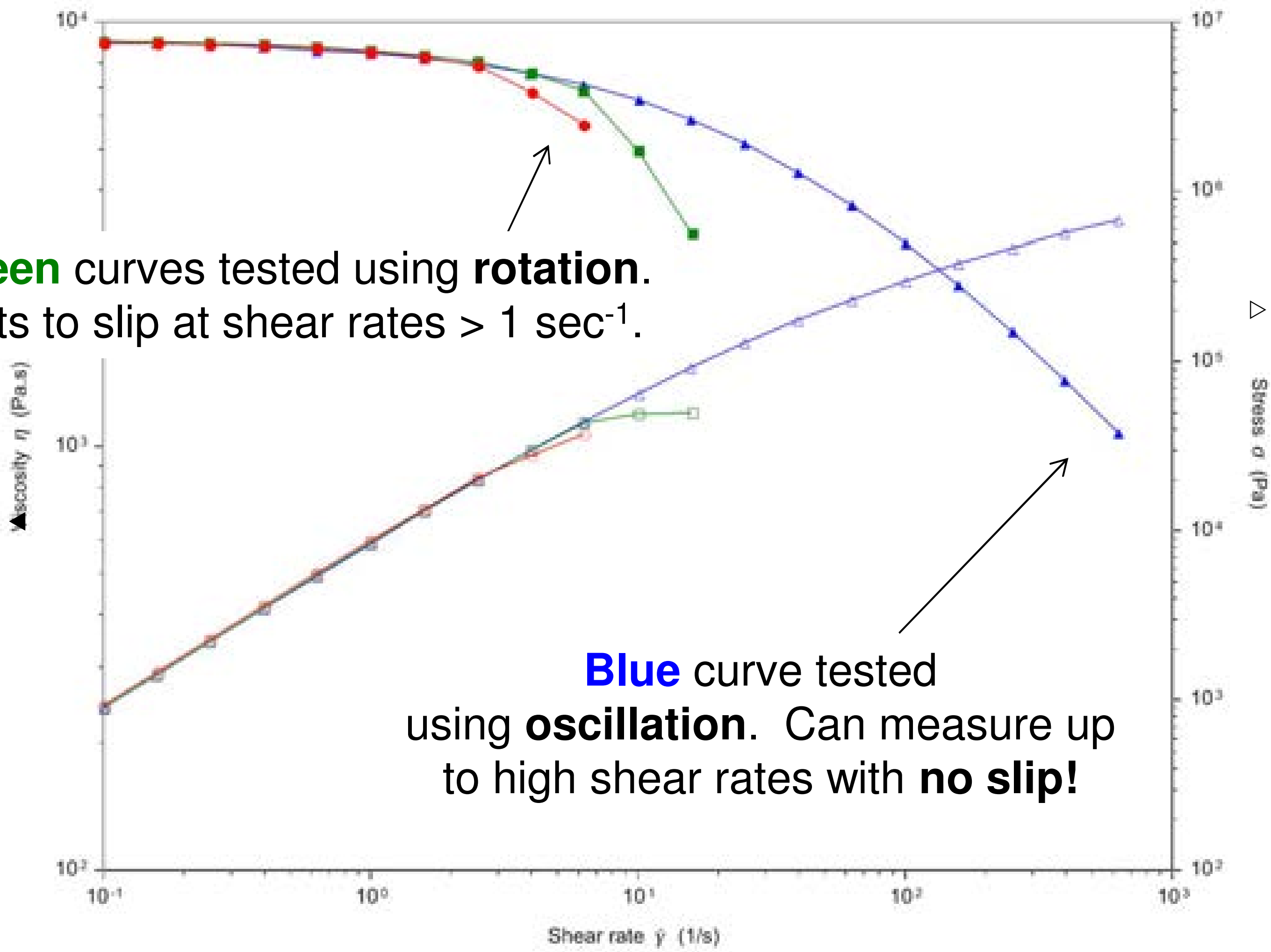
$\eta_0 = K \cdot Mw^{3.4}$

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

# Viscosity vs. Shear Rate – Polymer Melts



# Viscosity Measurements of LDPE at 190°C

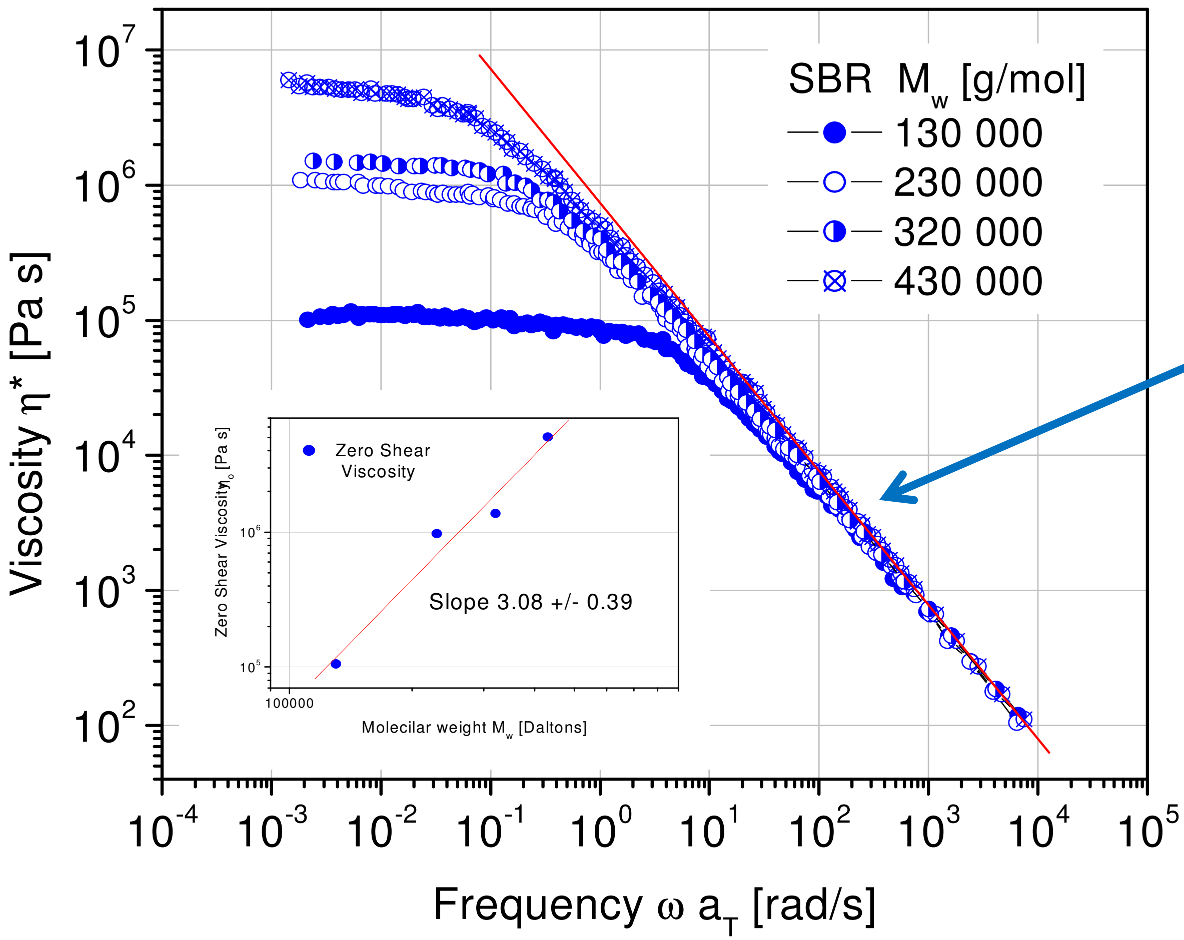



Red and Green curves tested using **rotation**. Sample starts to slip at shear rates > 1 $sec^{-1}$.

Blue curve tested using **oscillation**. Can measure up to high shear rates with **no slip!**

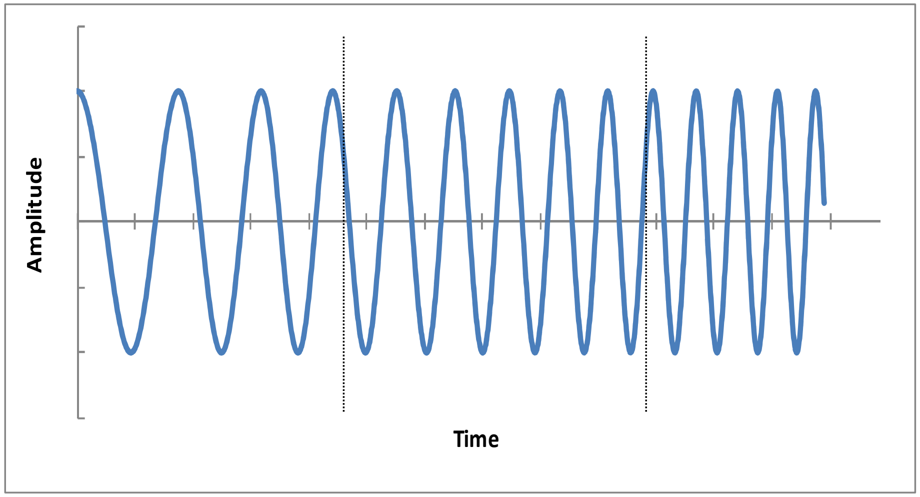
# Influence of Molecular Weight on Viscosity

The zero shear viscosity increases with increasing molecular weight.




The high shear rate viscosity is independent of the molecular weight

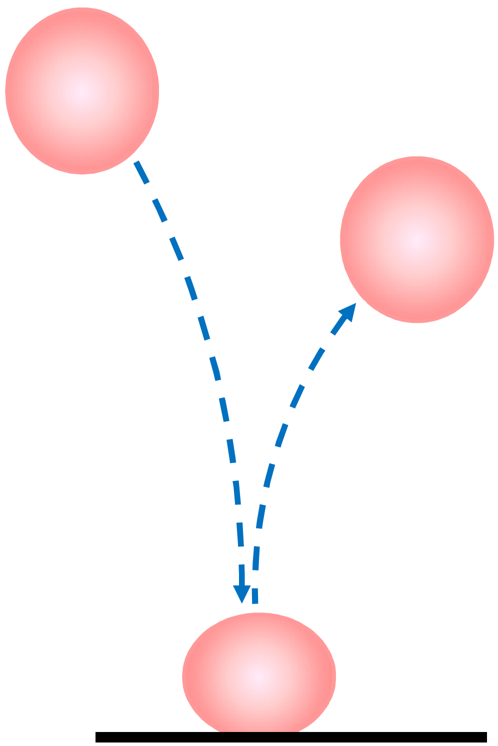
# Frequency Sweep



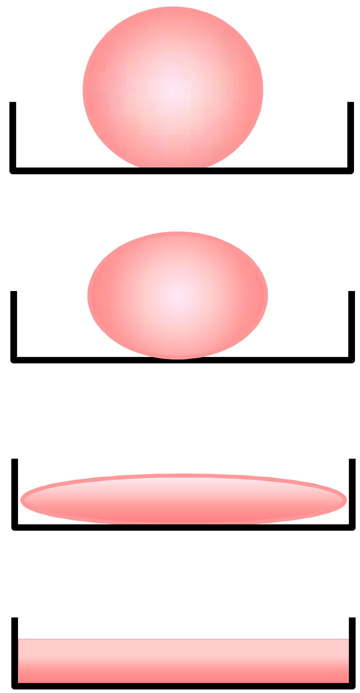

- The material response to increasing frequency (rate of deformation) is monitored at a constant amplitude (strain or stress) and temperature.

- Strain should be in LVR
- Sample should be stable
- Remember – Frequency is 1/time so low frequencies will take a long time to collect data – i.e. 0.001Hz is 1000 sec (over 16 min)

# Time-Dependent Viscoelastic Behavior



T is short [< 1s]

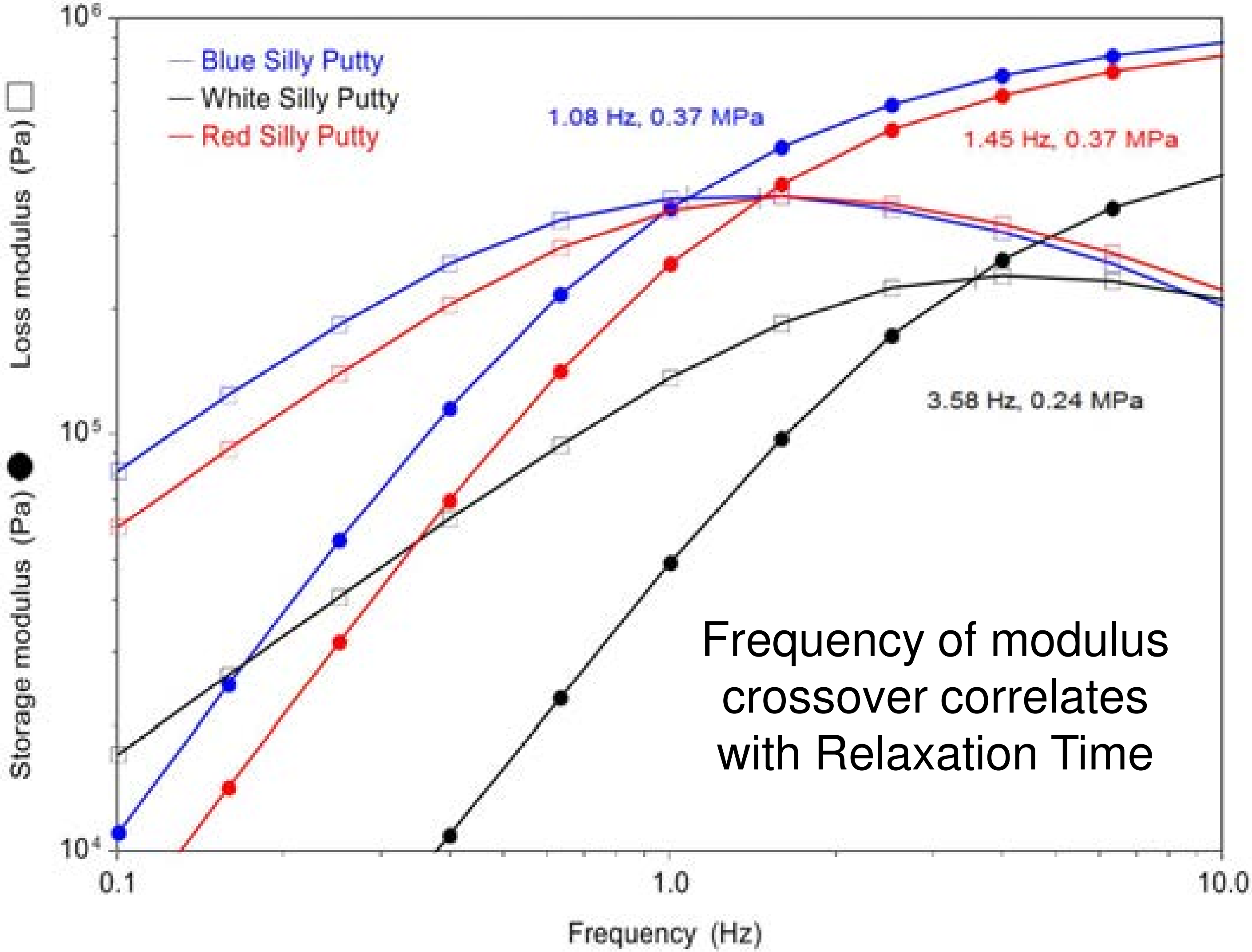


T is long [24 hours]

# Time-Dependent Viscoelastic Behavior

- Silly Putties have different characteristic relaxation times
- Dynamic (oscillatory) testing can measure time-dependent viscoelastic properties more efficiently by varying frequency (deformation time)

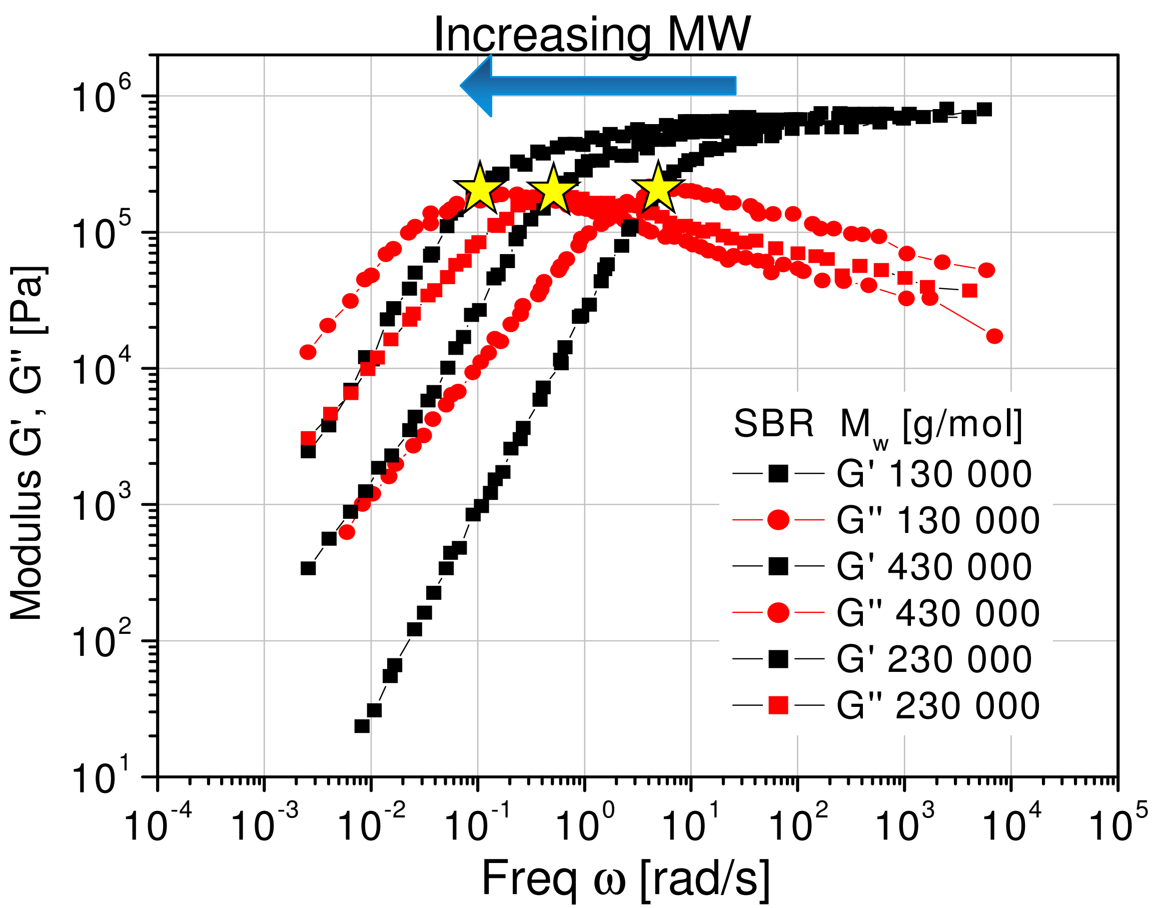
# Frequency Sweep- Time Dependent Viscoelastic Properties



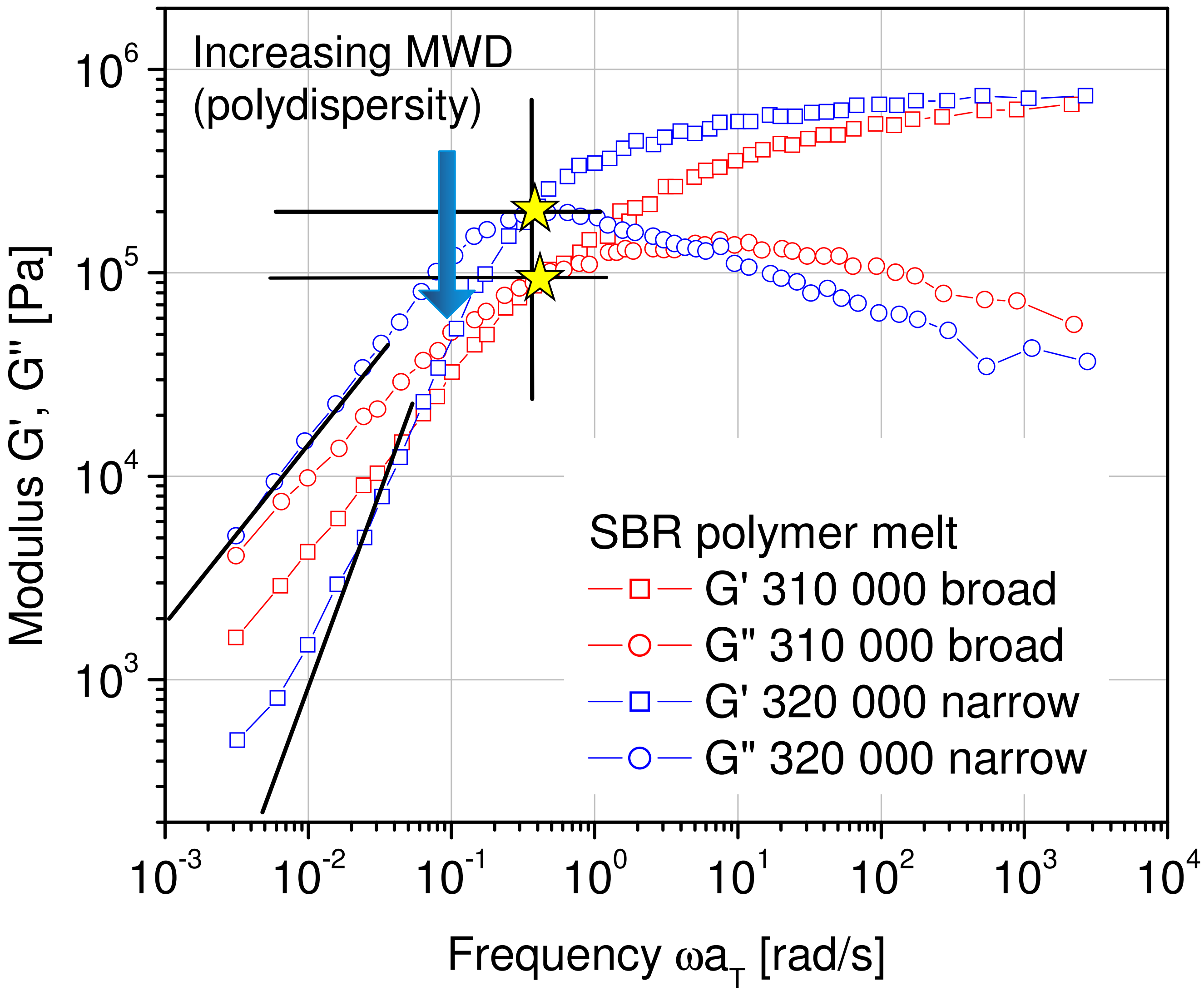

Frequency of modulus crossover correlates with Relaxation Time

# Influence of Molecular Weight on G’ and G”

The intersection of G’ and G” shifts to lower frequency as MW increases.



# Influence of MWD on G‘ and G“

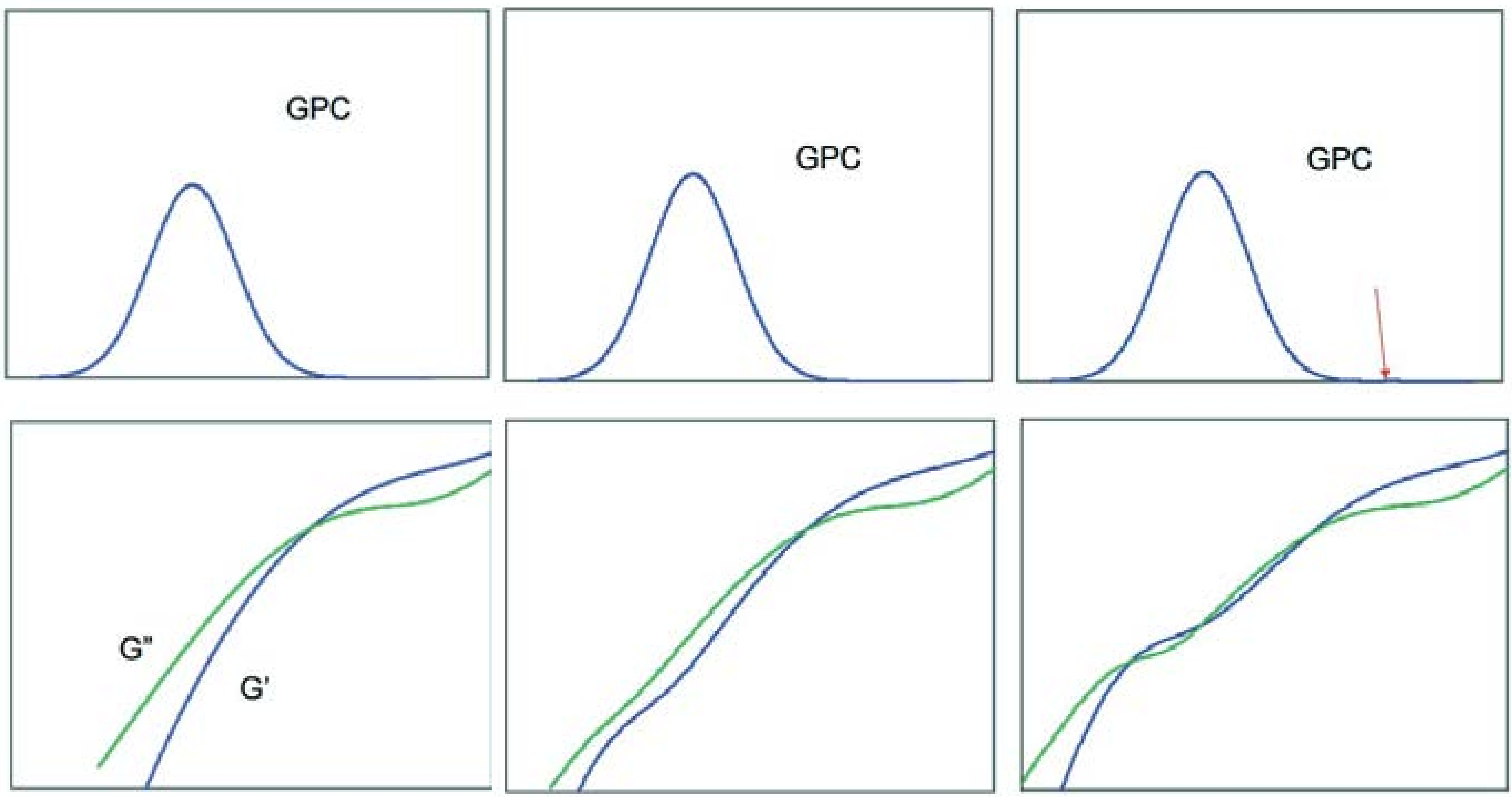



Higher crossover frequency: lower $M_w$

Higher crossover Modulus: narrower MWD

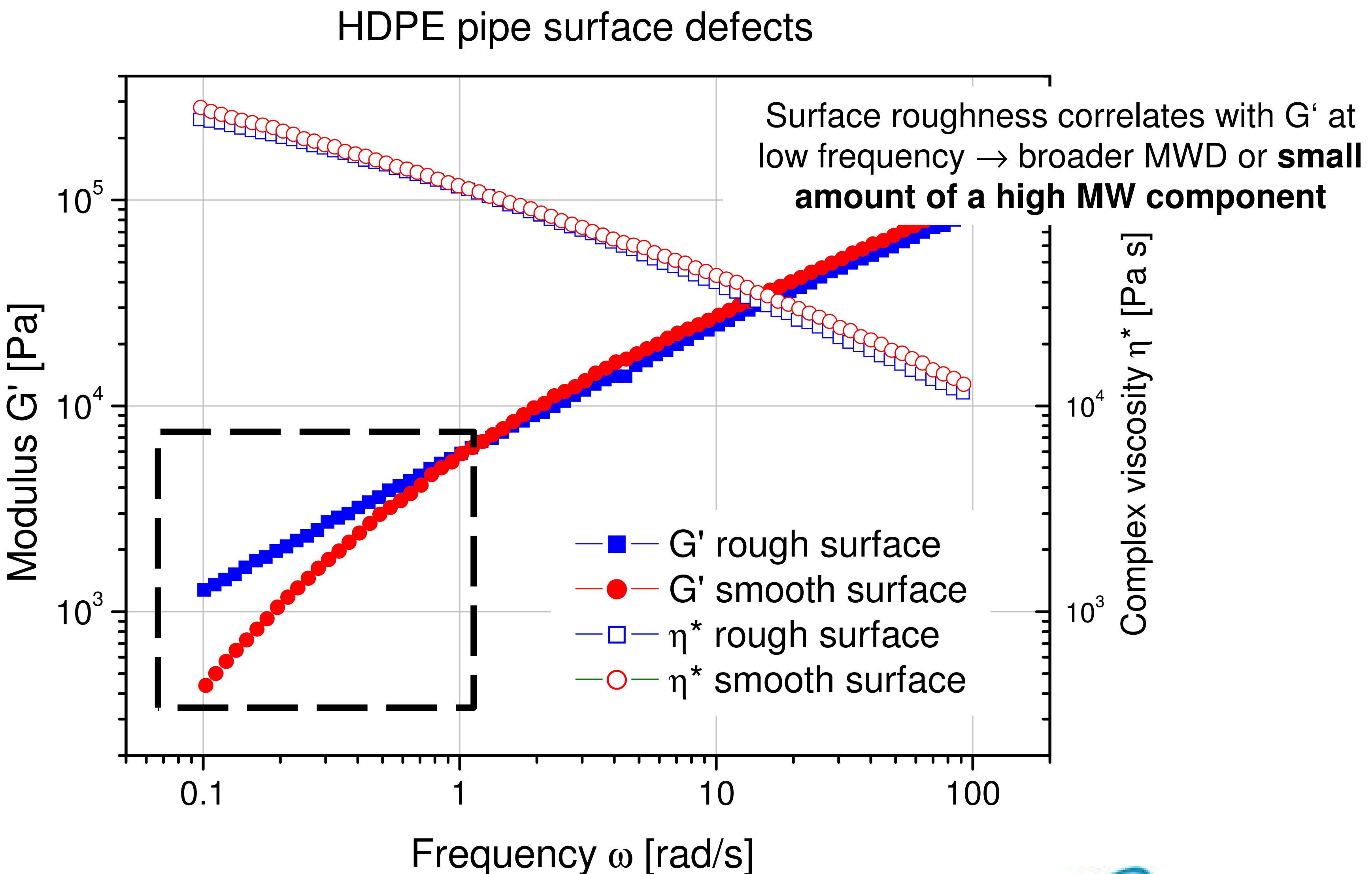
# High MW Contributions

| 400,000 g/mol PS | 400,000 g/mol PS + 1% 12,000,000 g/mol | 400,000 g/mol PS + 4% 12,000,000 g/mol |
|---|---|---|



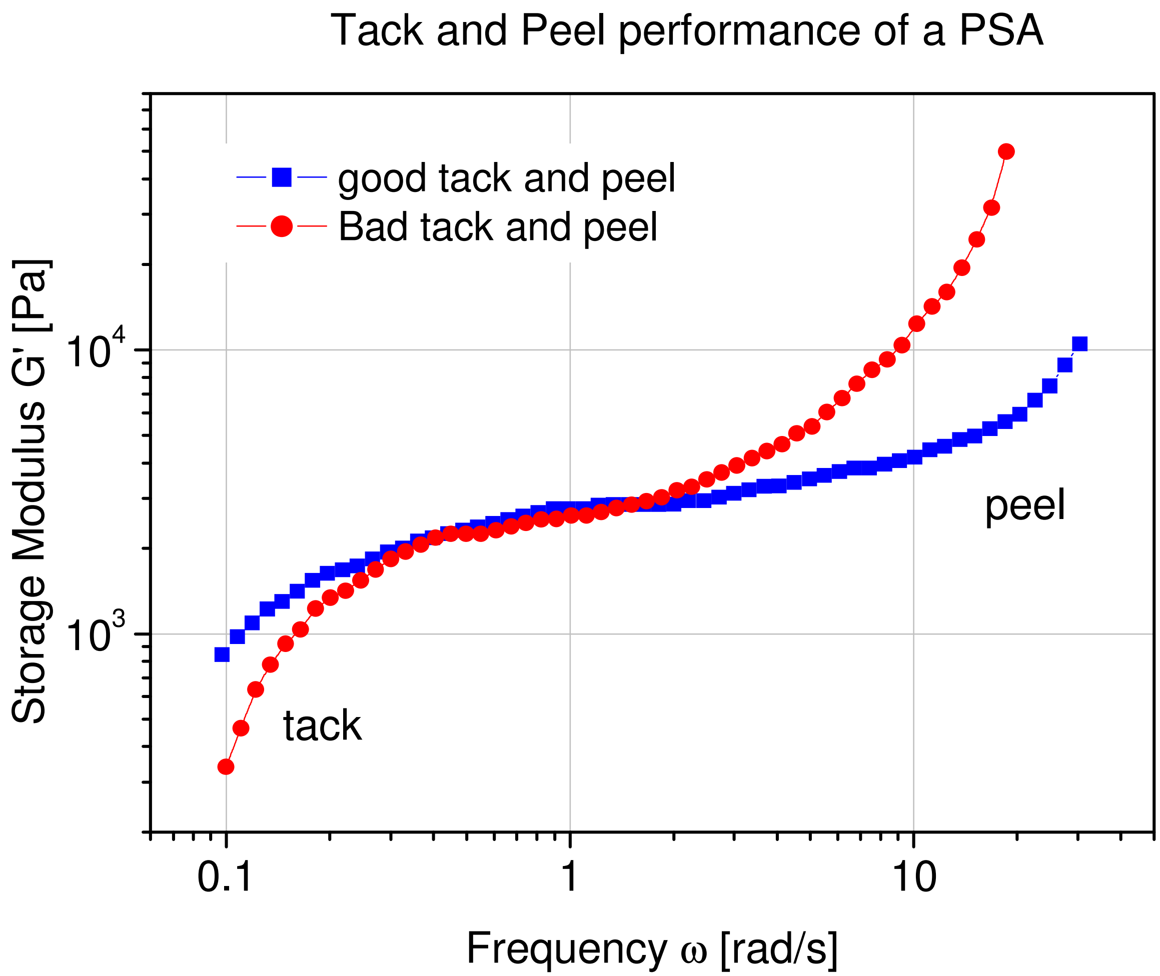

Macosko, TA Instruments Users' Meeting, 2015

# Example: Surface Defects during Pipe Extrusion



# Tack and Peel of Adhesives

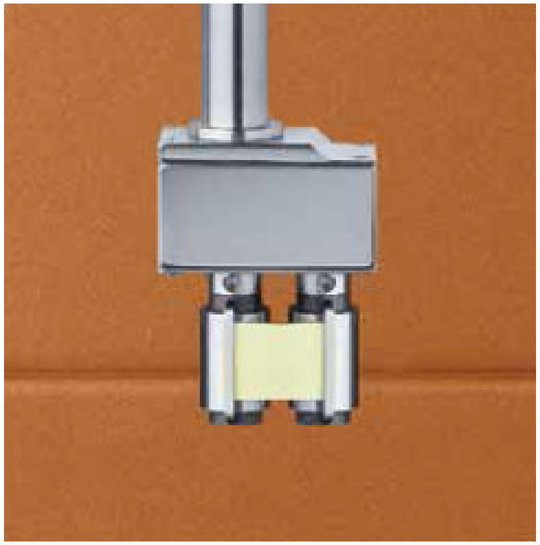
- Bond strength is obtained from peel (fast) and tack (slow) tests
- Tack and Peel are a function of viscoelastic properties at different frequencies



# Extensional Viscosity

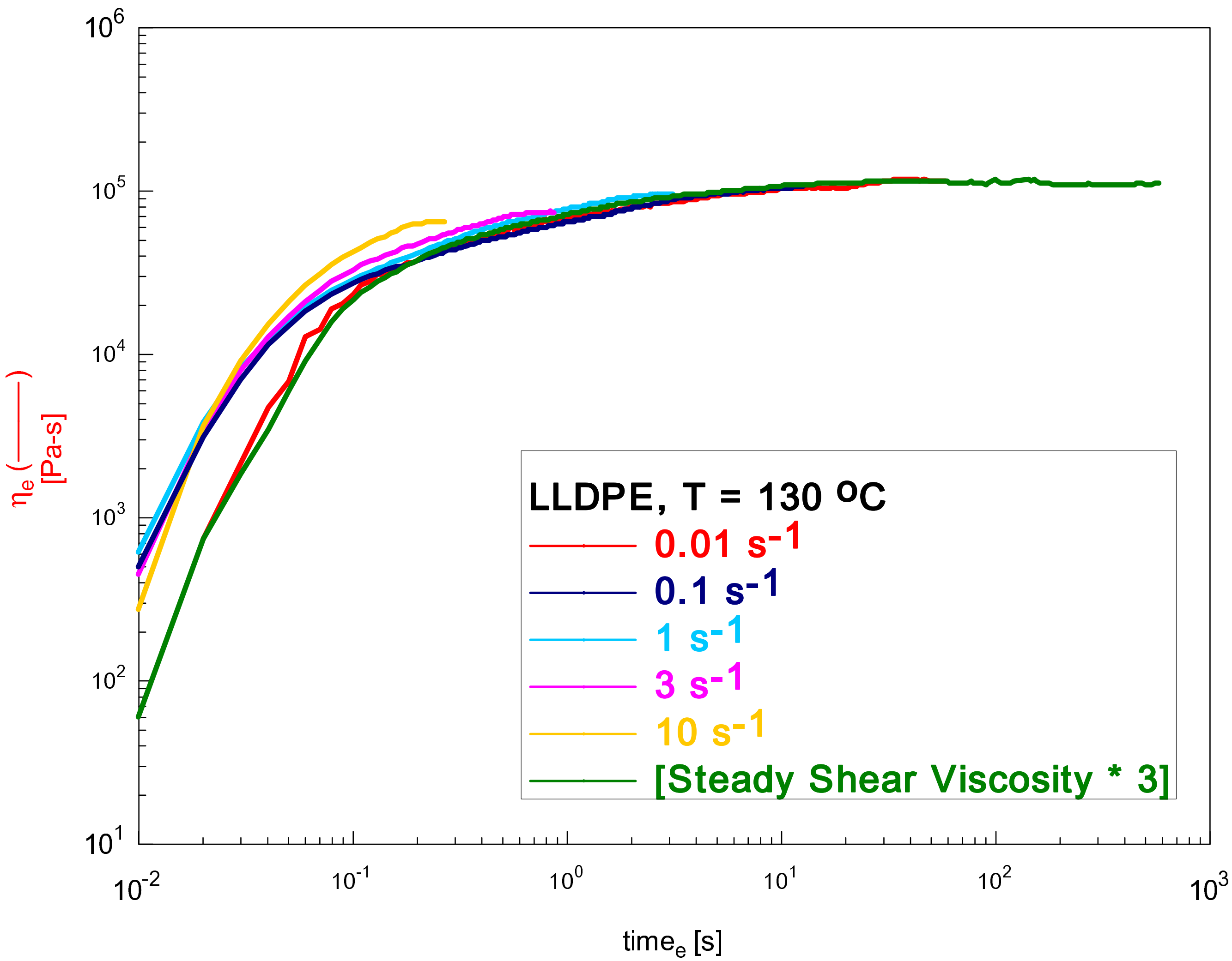
- Application to processing:
  - many processing conditions are elongation flows
  - testing as close as possible to processing conditions (spinning, blow-molding)

- Relation to material structure:
  - non linear elongation flow is more sensitive to polymer structure than shear flows (branching, polymer architecture)

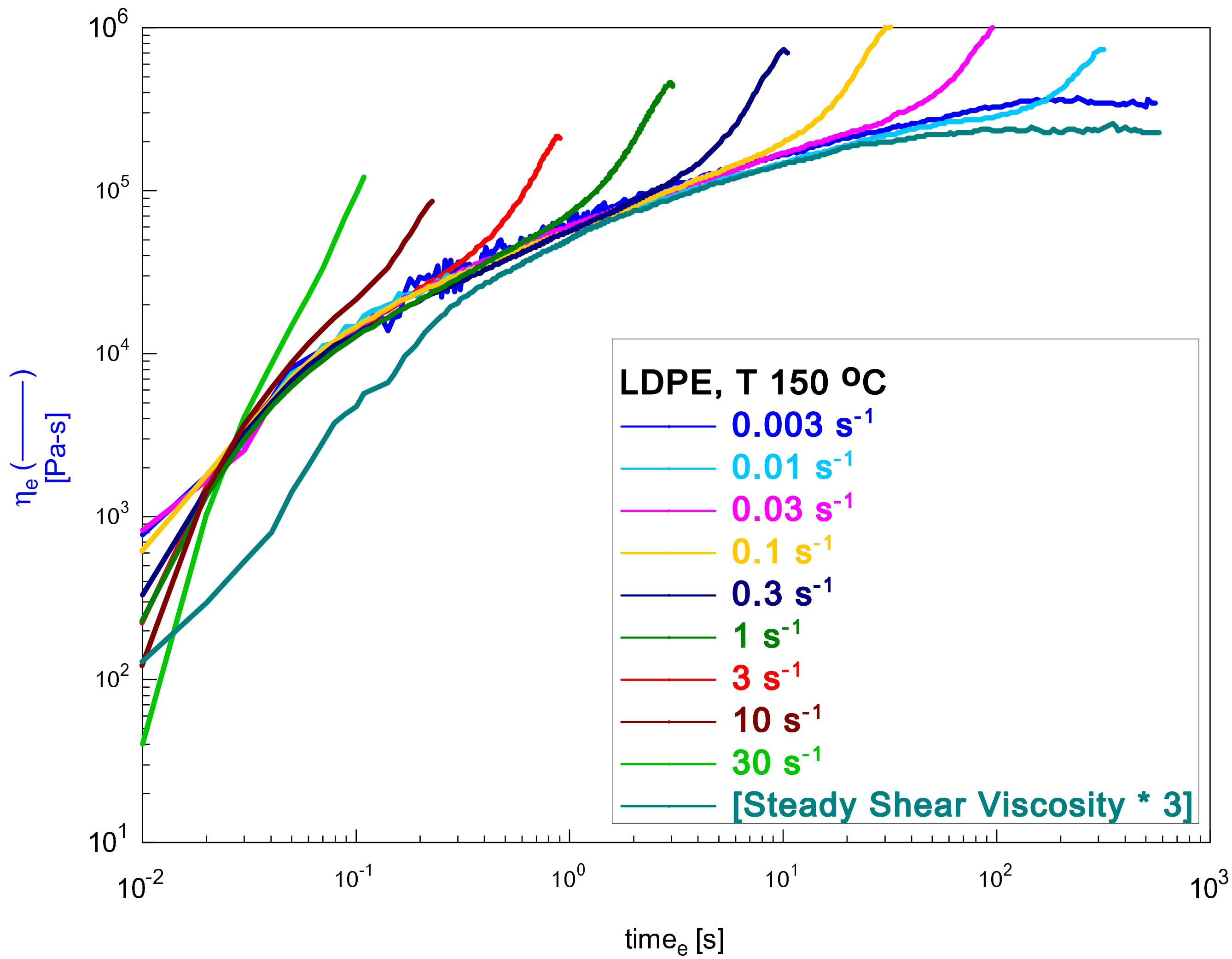


SER2 Extensional

# LLDPE (Low branching)



# LDPE (High branching)

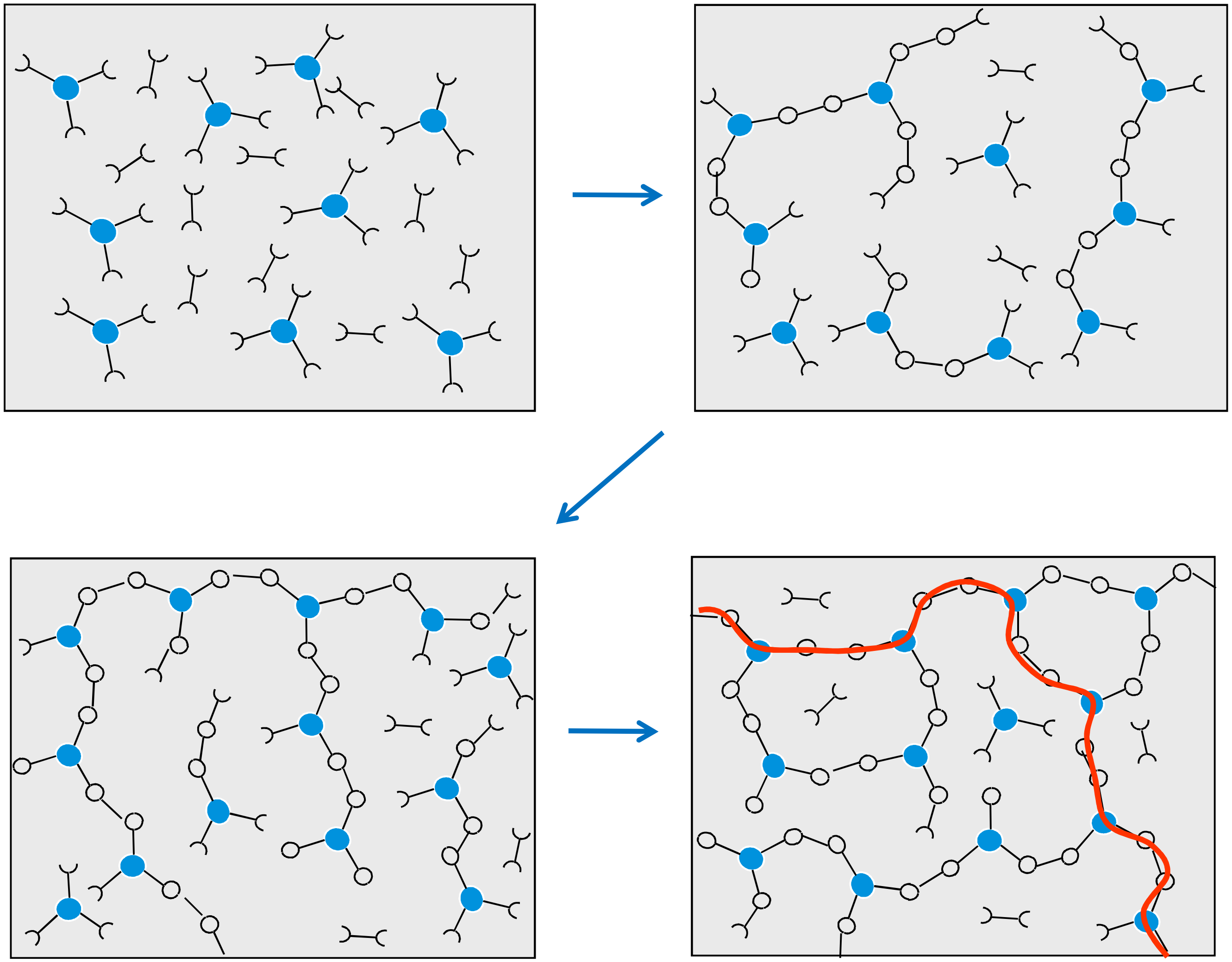


# Thermosets and Gels

# Thermosets and Gels

- What is a Gel?
  - A soft solid that contains a polymeric network and a substantial fraction of solvent
  - Latin: *gelatus* (frozen; immobile)
- "A substantially dilute crosslinked system that exhibits no flow in the steady state."
  - J.D. Ferry, Viscoelastic Properties of Polymers, 1980.
- Chemical Gel: Network of covalent interactions.
- Physical Gel: Network of non-covalent interactions.

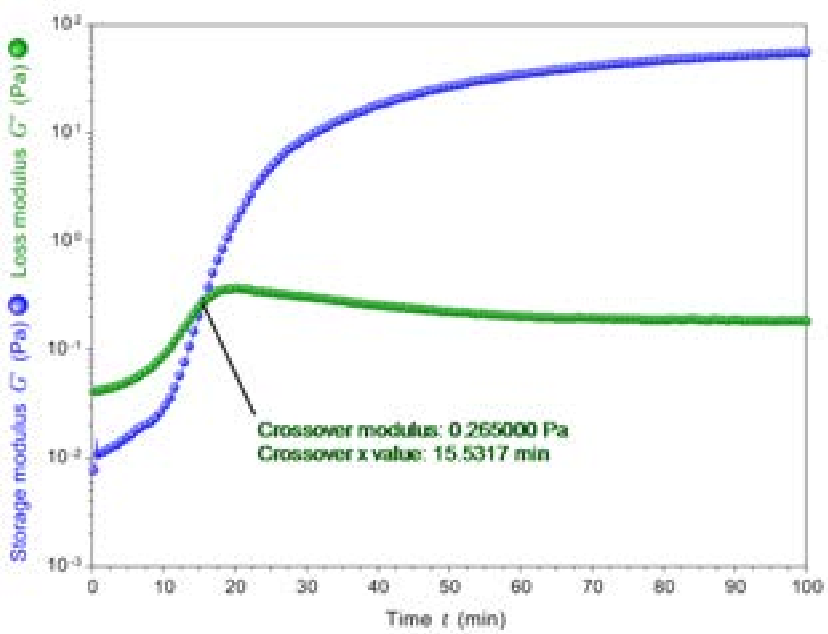
# Gelation

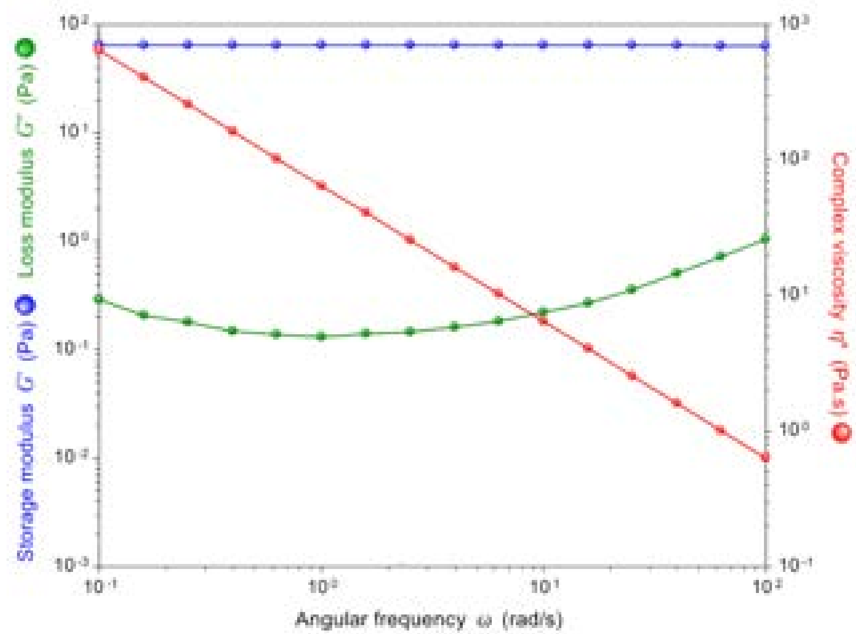


Gel point

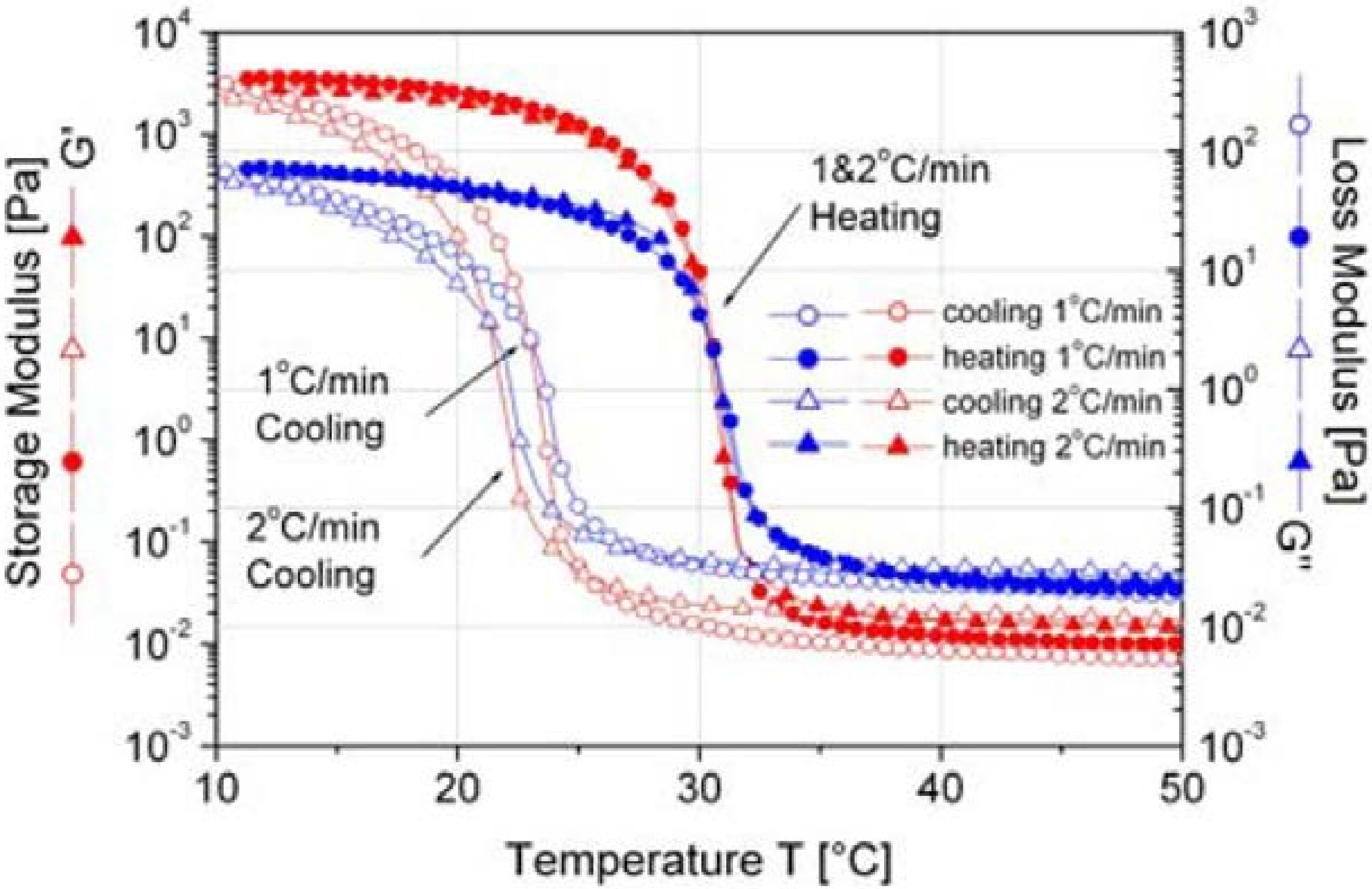
# Hyaluronic Acid Gels:

- Hyaluronic acid gels are used as lubricating agent during abdominal surgeries to prevent adhesion and also for join lubrication, wound healing etc.
- Rheology can monitor HA gelation and evaluate the gel strength

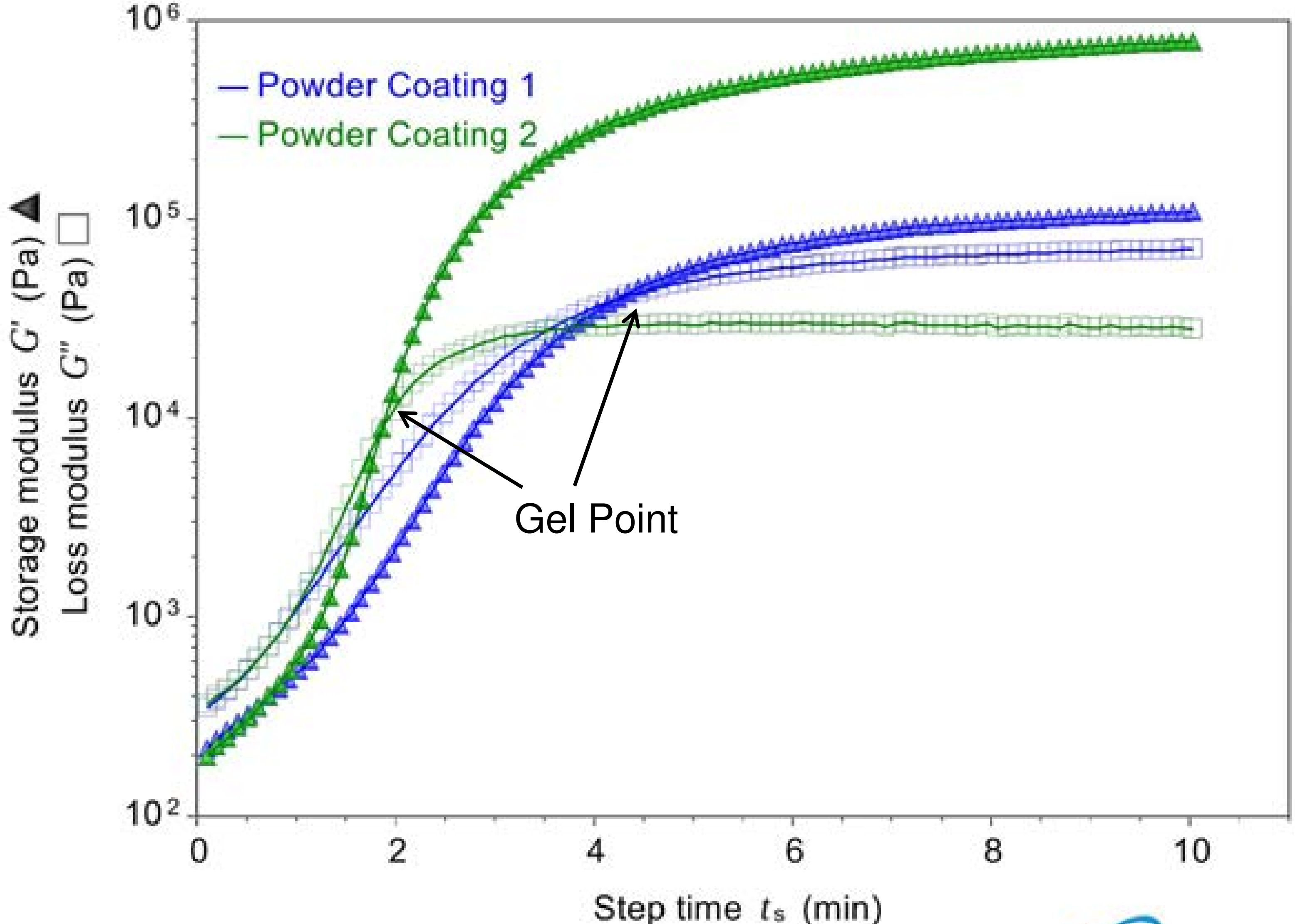


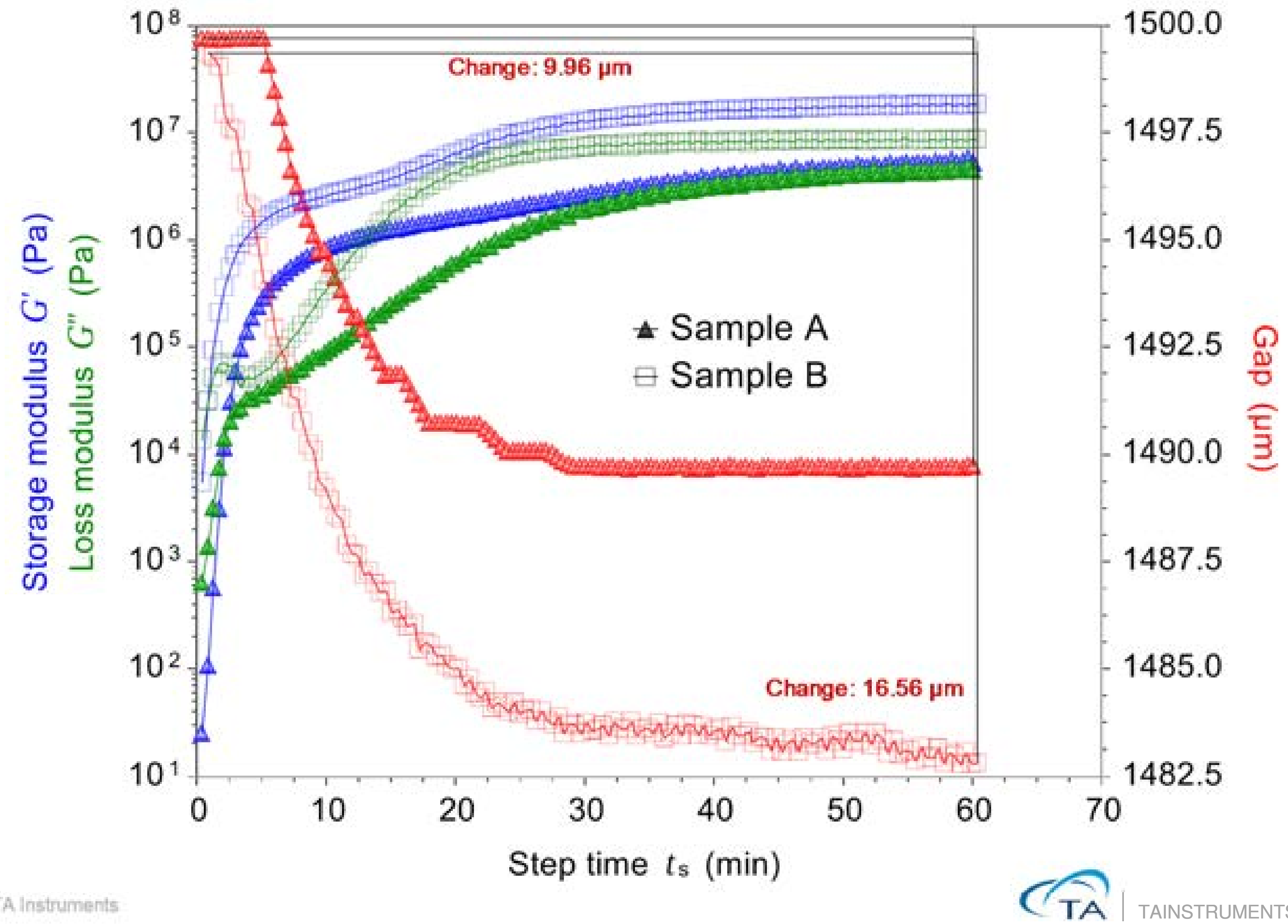
# Gelation During Cooling



# Powder Coating: Cure Test

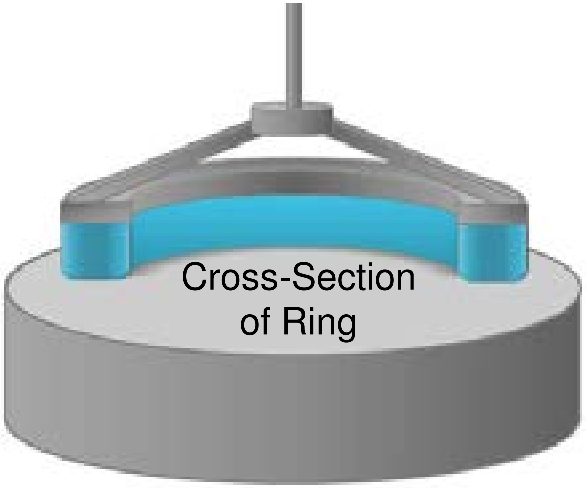


# Cure Testing- Dimensional Change

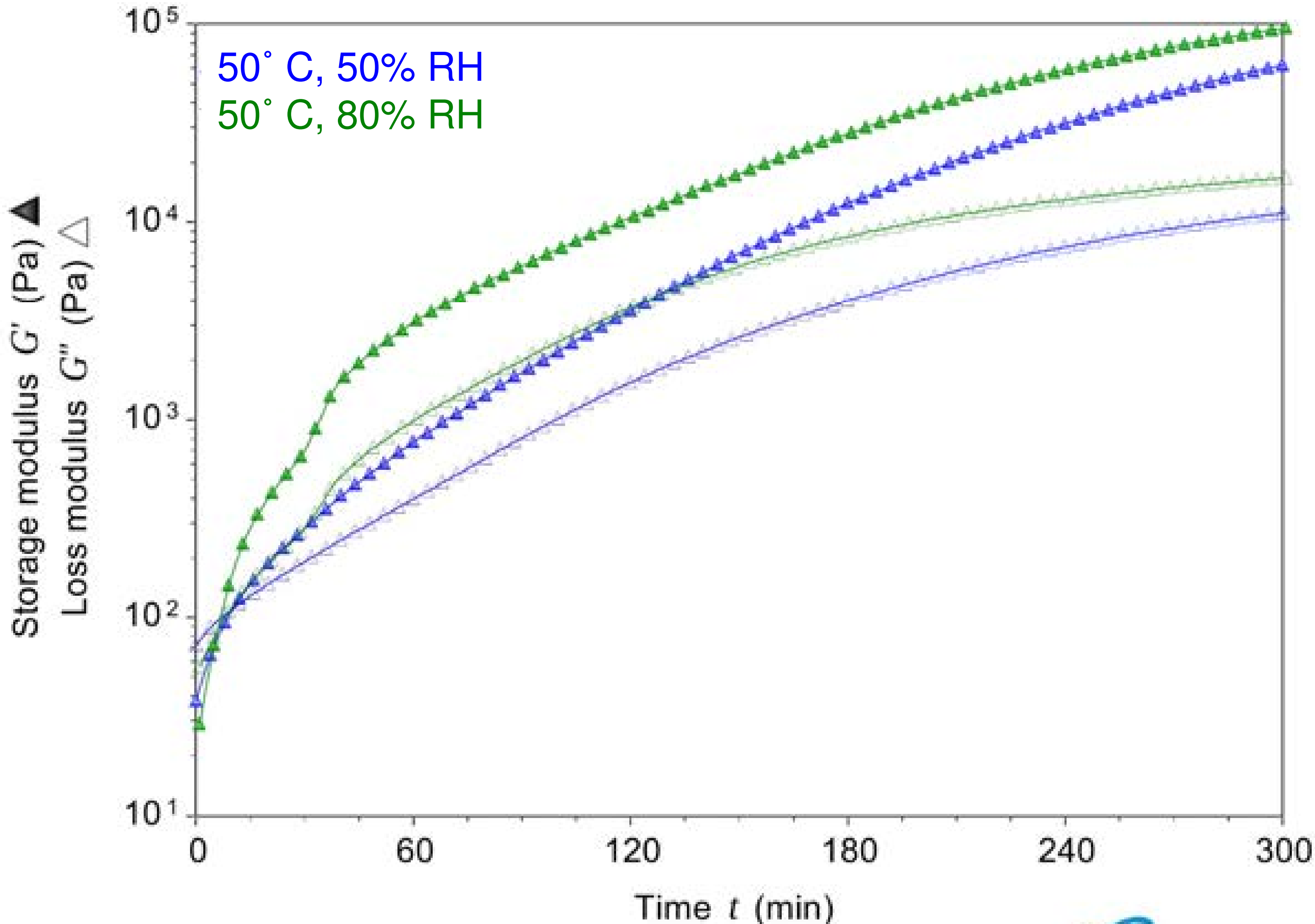


# Change in Mechanical Properties During Drying

- Relative Humidity and Temperature Controlled Chamber
- **Quantitative** measurement of modulus during drying of the bulk material.
- Characterize time of drying
  - Time needed to "set" (Crossover point)
- Determine conditions needed to achieve drying
- Test Method: constant temperature and humidity



# Moisture-Cured System- Humidity Control



# Flow Testing

# Newtonian and Non-Newtonian Fluids

- Newtonian Fluids - Viscosity does not change with changes in shear rate or time.

  *(examples: water, oil, honey)*

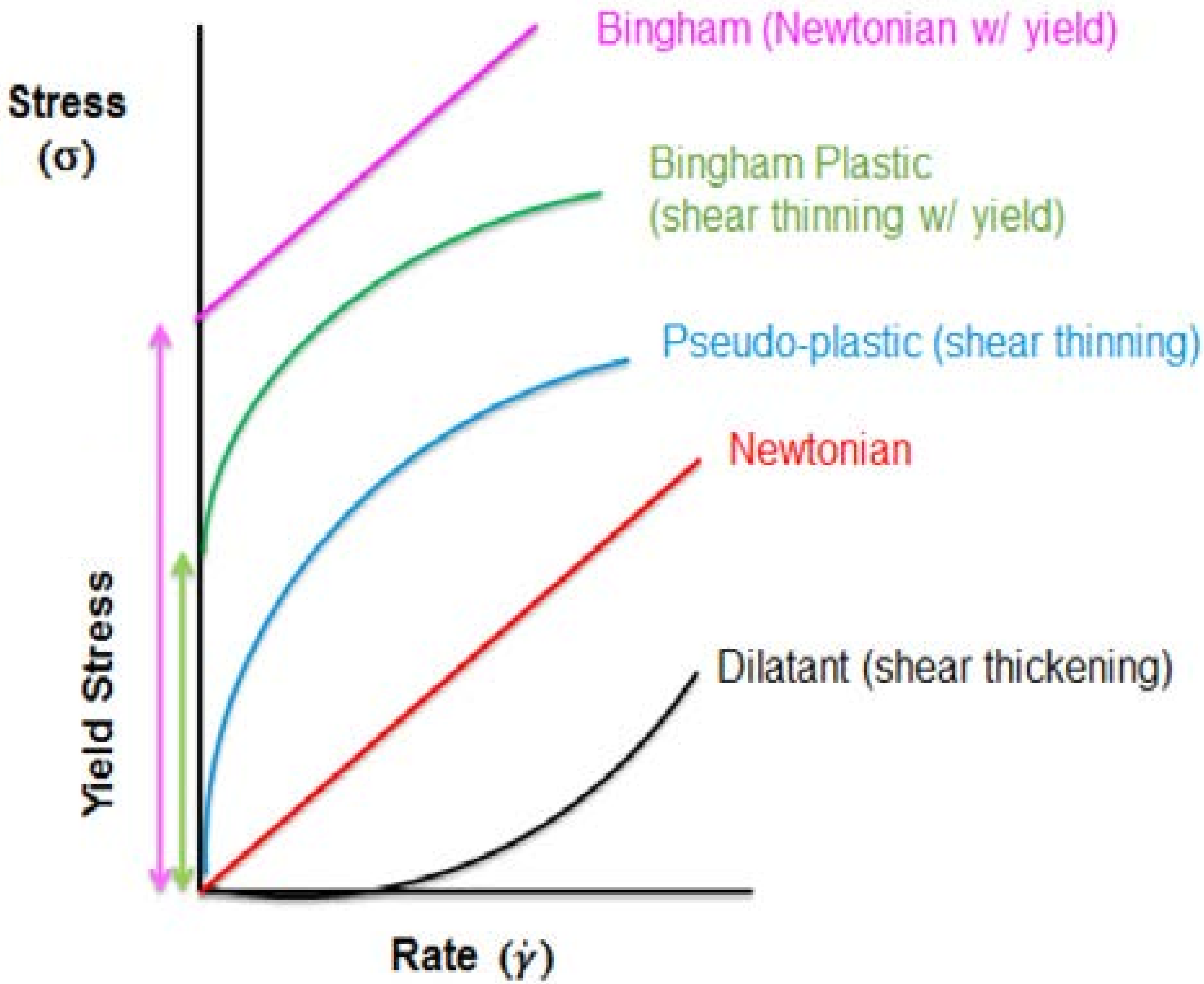
- Non-Newtonian Fluids - Viscosity is time or shear rate dependent

  *(examples: mayonnaise, paint, polymer, asphalt)*

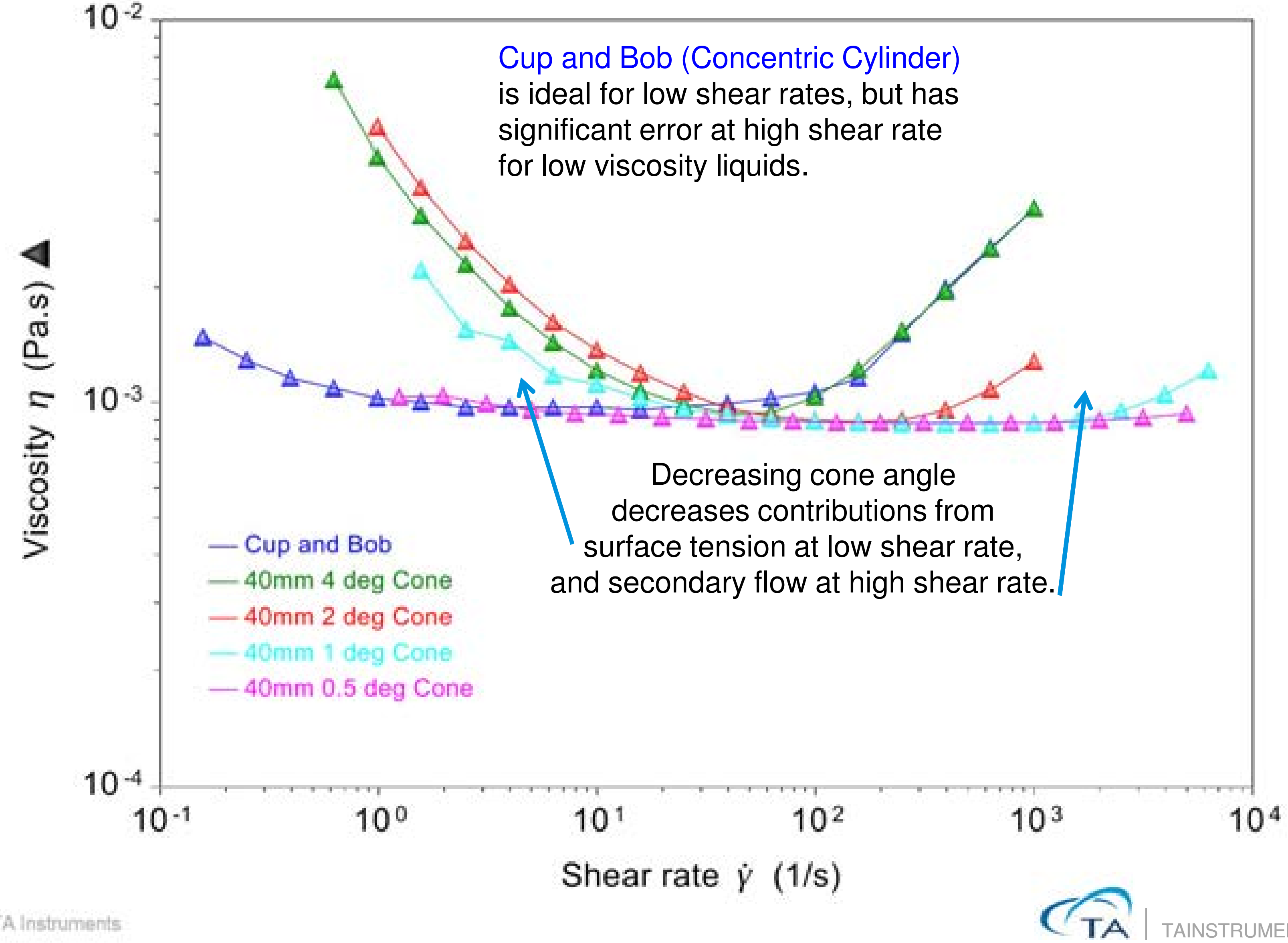
  - Shear – Thinning: viscosity **decreases** as shear rate increases
  - Shear – Thickening: viscosity **increases** as shear rate increases.

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

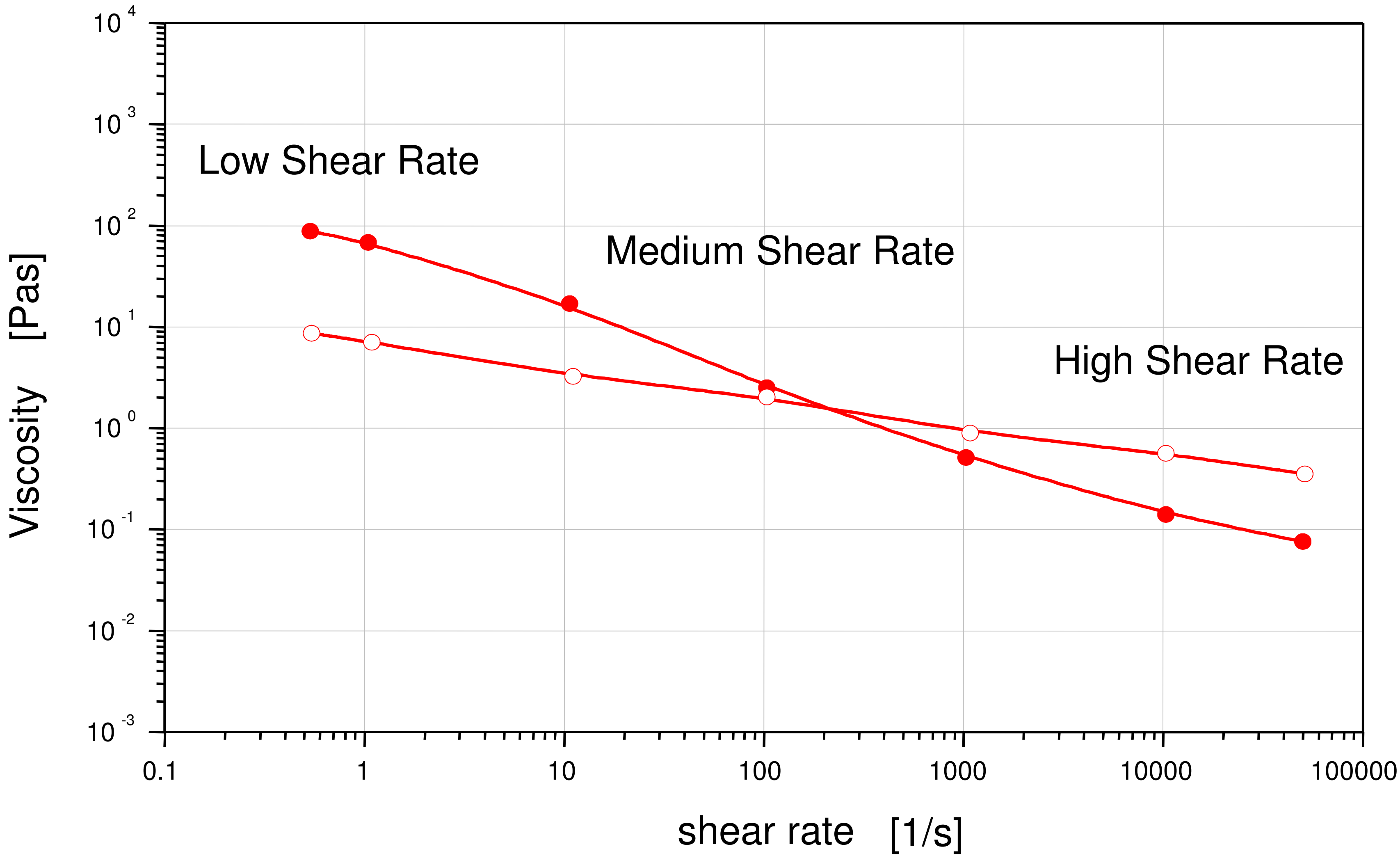
# Flow Behaviors



# Viscosity of Water

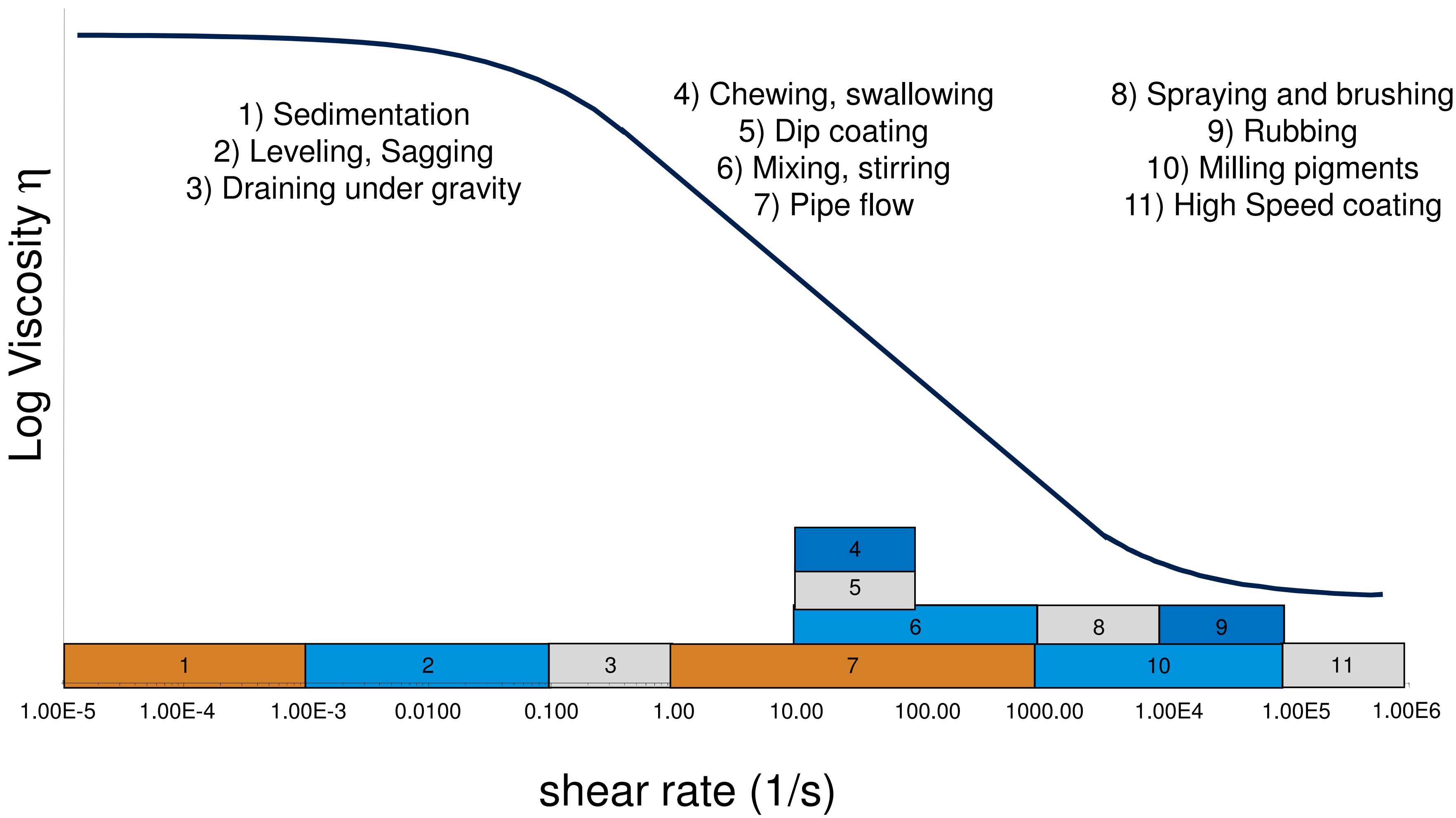


# Viscosity is curve, not a single value!

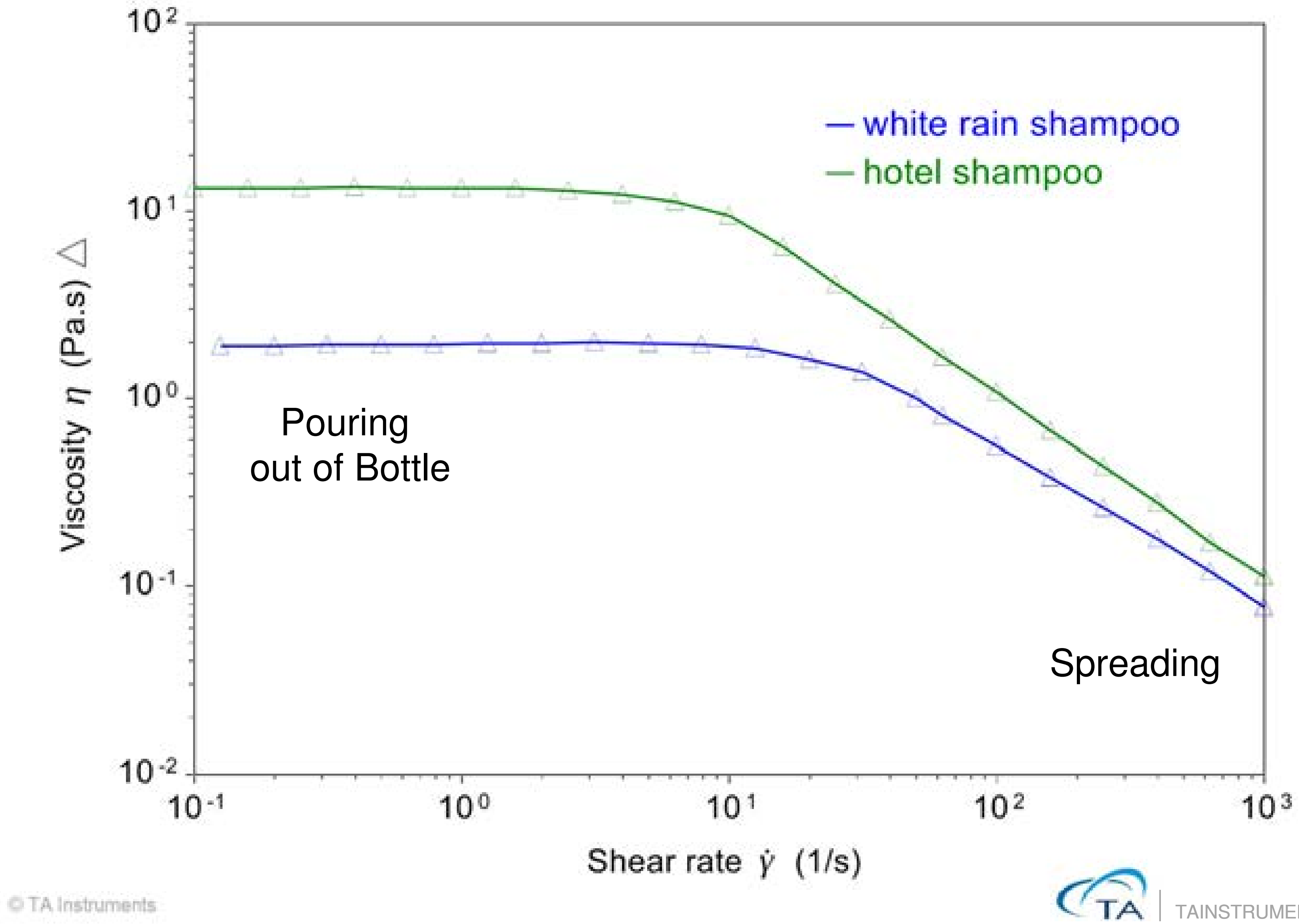


# Viscosity Flow Curve

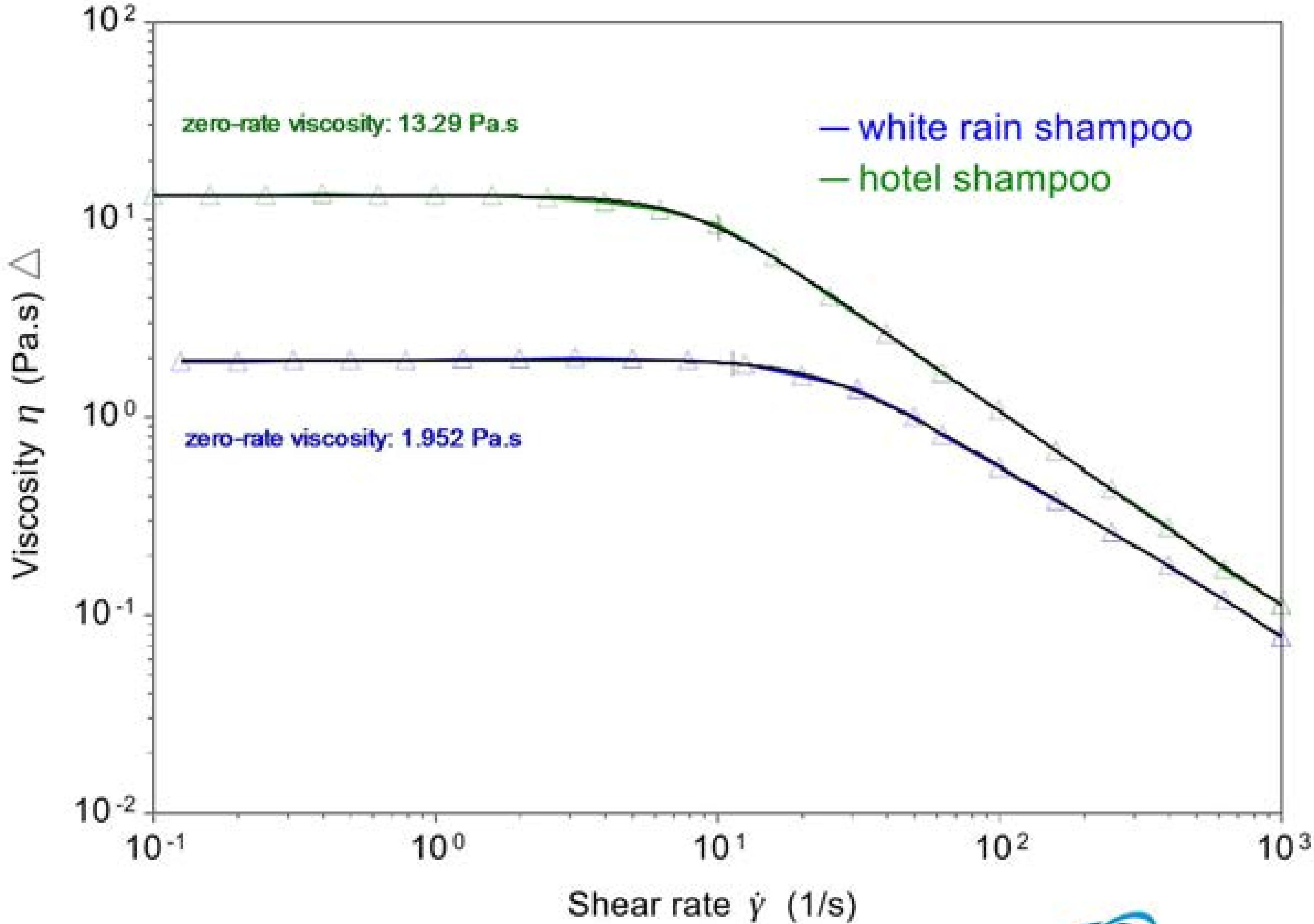
What shear rate?



# Viscosity vs. Shear Rate- Shampoo



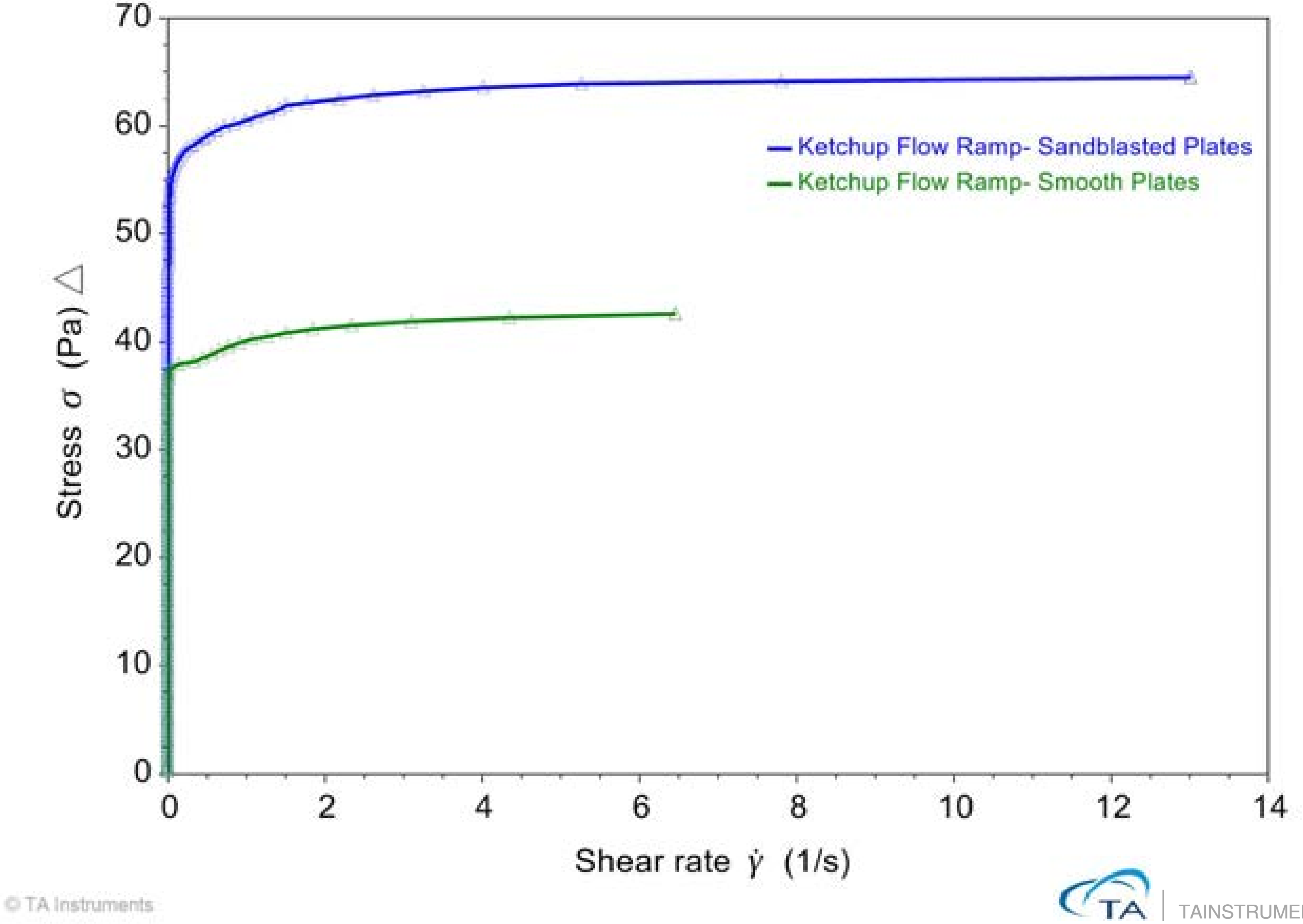
# Viscosity vs. Shear Rate- Shampoo



# What is Yield Stress?

- Some Structured Fluids behave like “solids” at rest.
- A critical stress must be applied for these materials to flow.
- What does Yield Stress do?
  - Stabilize against sedimentation of separation
  - Improve ease of use
  - Prevent dispensing of product

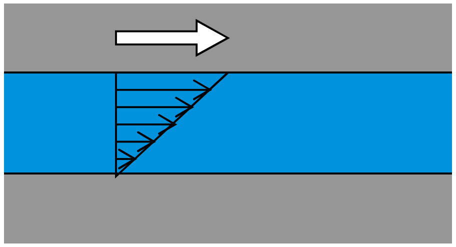
# Yield Stress- Ketchup



# Wall Slip

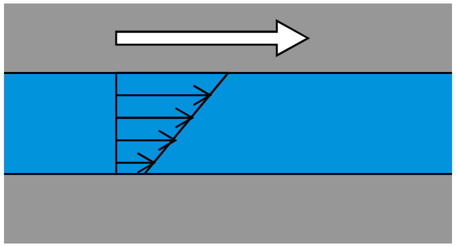
**No slip condition**

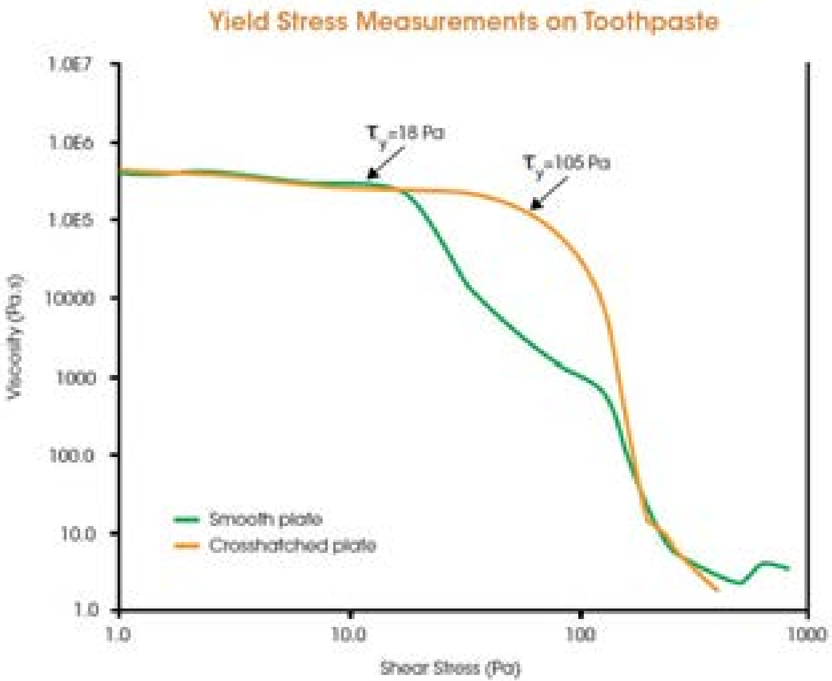
Ideal, Assumed Velocity Profile



**Wall Slip**

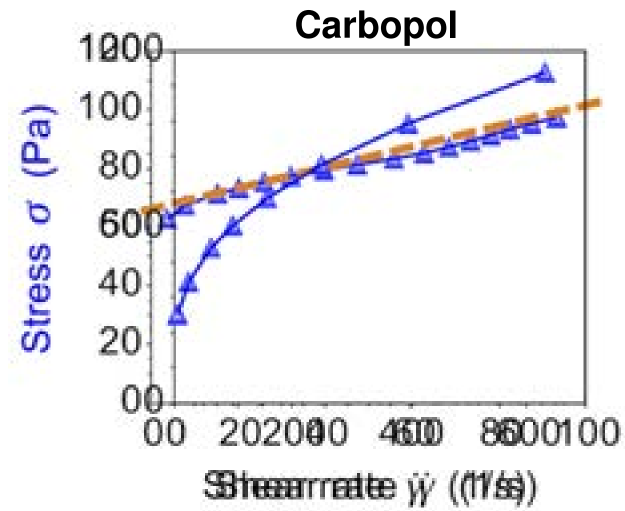
Incorrect Velocity Profile

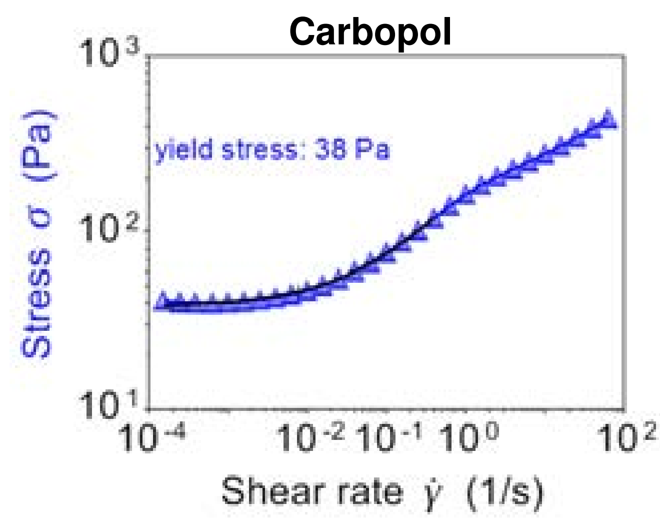




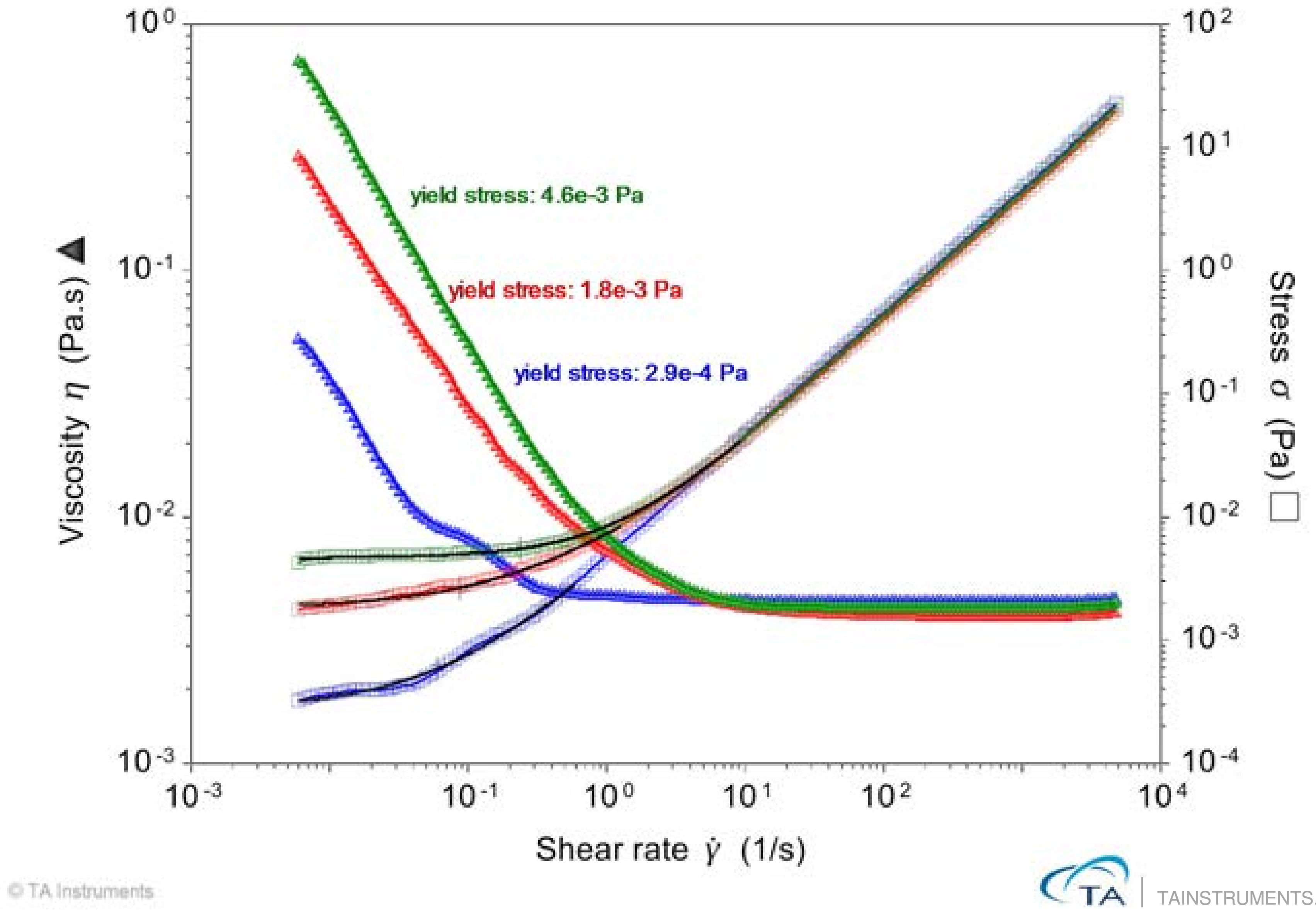
# Controlled-Rate Measurement of Yield Stress

- Indirect Yield Stress Measurement
  - Extrapolate Stress at Shear Rate = 0.0 $^{1}/_{sec}$
  - Assumes Newtonian viscosity after yield
  - Sample has already yielded.
- Steady-State Viscosity at low Shear Rates
  - Shear rate is decreased to very low rates.
  - Stress plateaus and approaches Yield Stress.
  - Herschel-Bulkley model

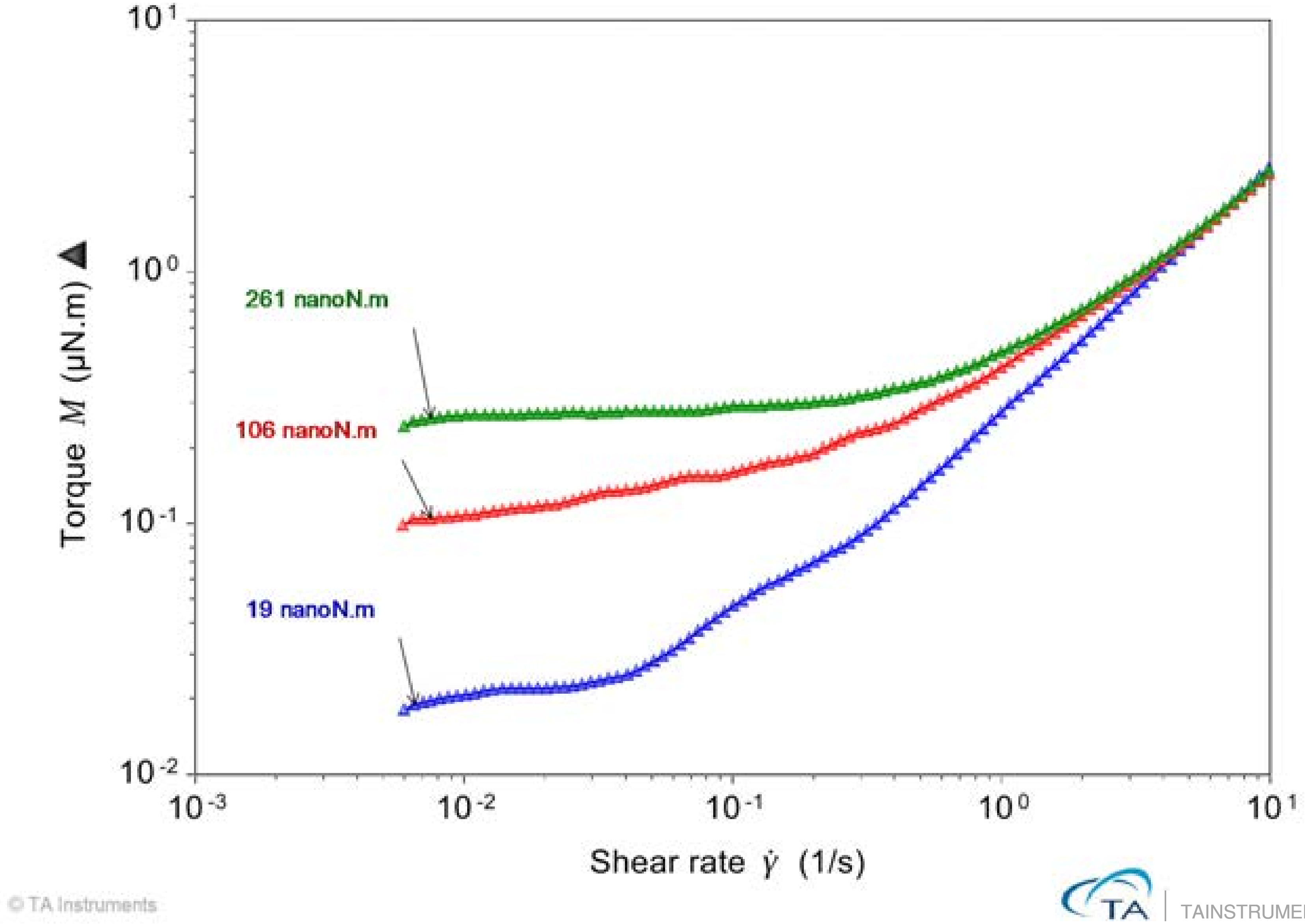


# Viscosity of Protein Solutions

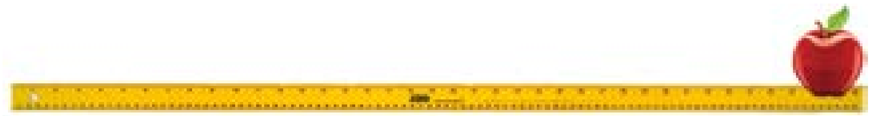


# Protein Solutions: Low Torque Sensitivity

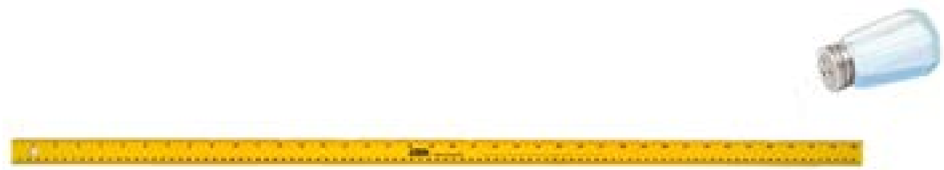


# Understanding Torque

- What is a **N.m**?
  - An apple (about 150 g) on the end of a meter stick
  - **1.5 N.m**



- What is a **µN.m**?
  - A grain of salt (about 0.5 mg) on the end of a meter stick)
  - **5 µN.m**



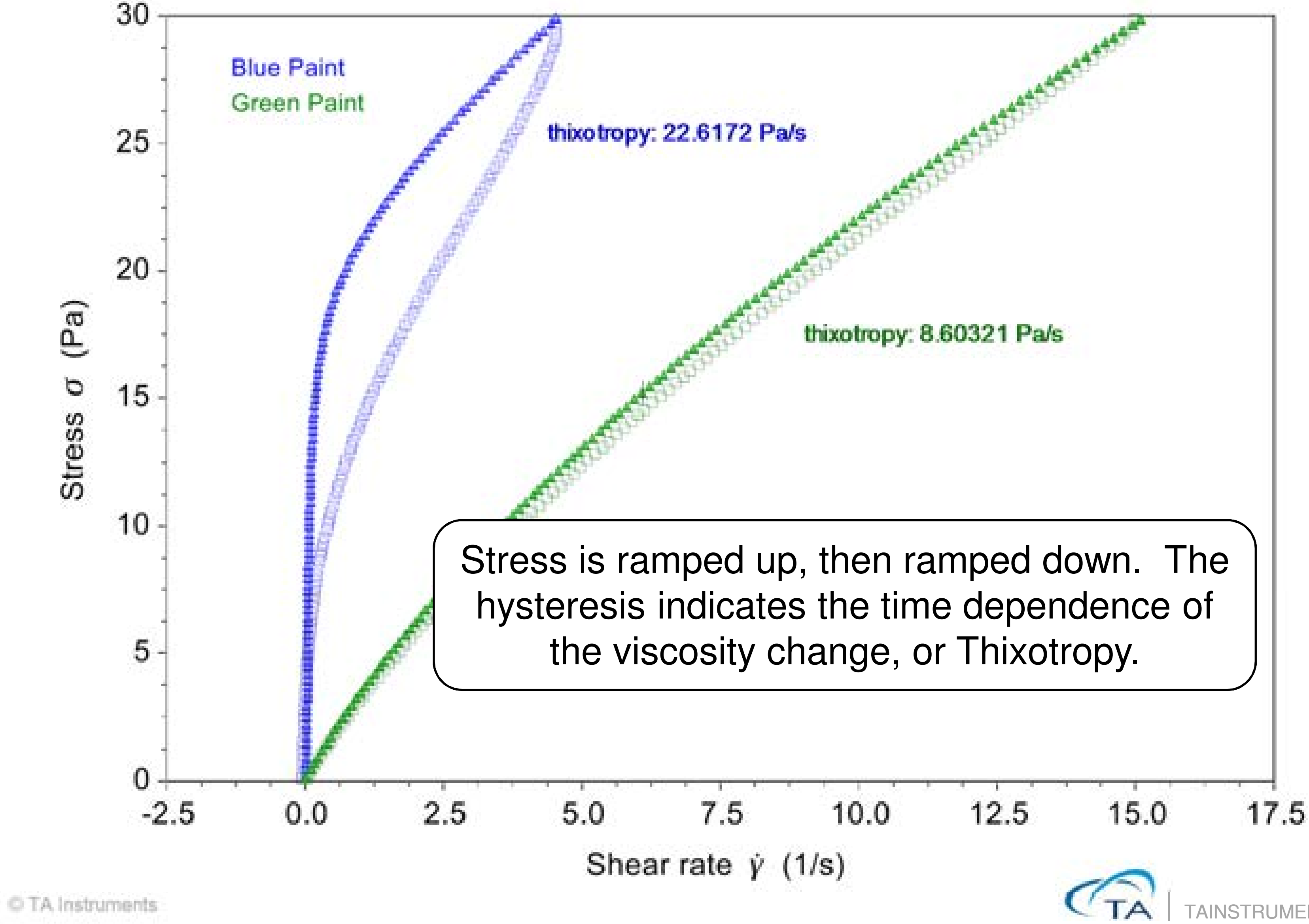
- And a **nanoN.m**?
  - A speck of dust (1 µg) on the end of a meter stick
  - **10 nanoN.m**

# Thixotropy

The thixotropy characterizes the time dependence of reversible structure changes in complex fluids. The control of thixotropy is important to control:

- process conditions; for example, to avoid structure build up in pipes during rest periods
- sagging and leveling; gloss of paints and coatings


# Thixotropy of Paint




Stress is ramped up, then ramped down. The hysteresis indicates the time dependence of the viscosity change, or Thixotropy.

# Specialized Accessories

- Wide range of accessories for different testing needs
- Environmental controls (Temperature, humidity, pressure, etc.)
- Rheo + other technique (optical, dielectric, etc.)
- Rheology response to stimulus (UV, electric, magneto, etc.)

# DHR Humidity Accessory

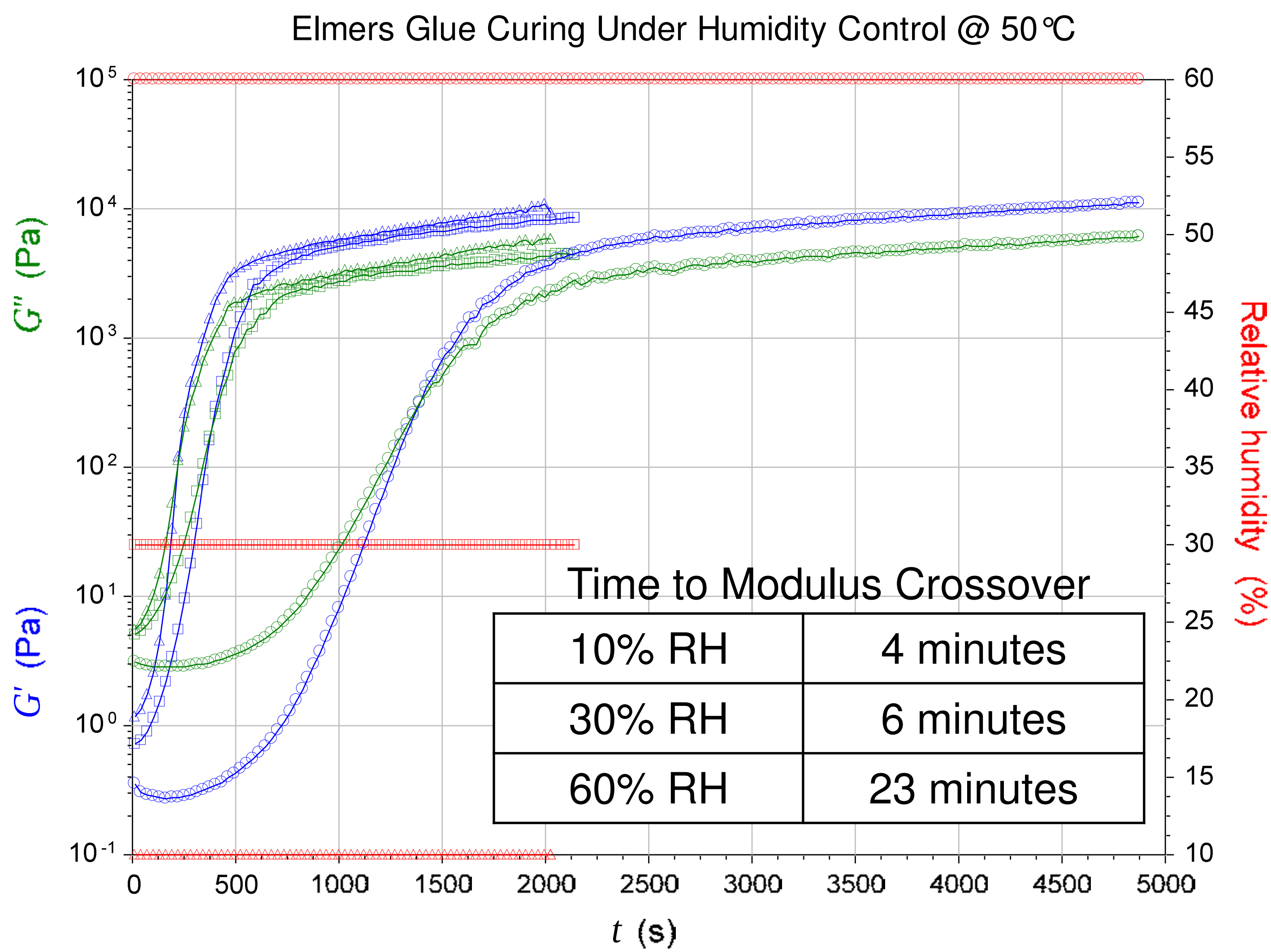
▪ Surface Diffusion

▪ Bulk Diffusion

▪ Film/fiber Tension

| |
|---|
| Temperature Range<br>5 ℃ – 120 ℃ |
| Temperature Accuracy<br>±0.5 ℃ |
| Heating/Cooling Rate<br>±1 ℃/min maximum |
| Humidity Range<br>5- 95% |
| Humidity Accuracy<br>5-90%RH: ±3% RH<br>>90%RH: ±5% RH |
| Humidity Ramp Rate<br>±2% RH/min<br>increasing or decreasing |

# Example: Glue Drying under Different Humidity

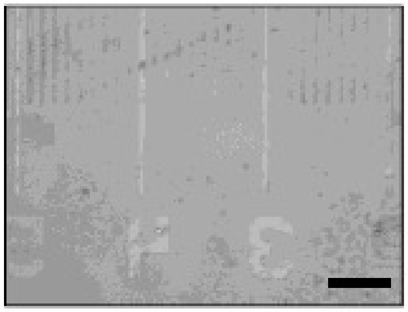



Time to Modulus Crossover

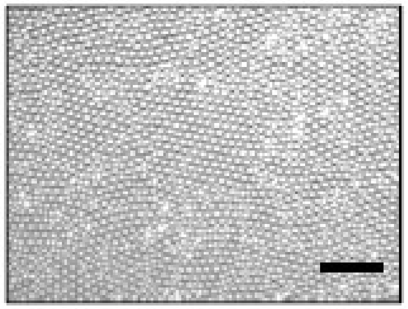
| | |
|---|---|
| 10% RH | 4 minutes |
| 30% RH | 6 minutes |
| 60% RH | 23 minutes |


# Modular Microscope Accessory (DHR)

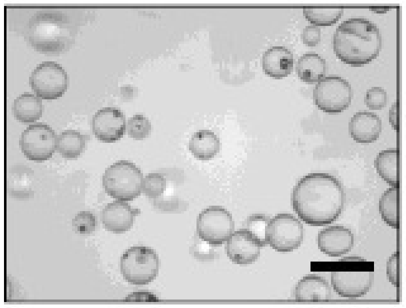
- Connecting Rheology with structure under flow conditions (counter rotation option also available). Modular video camera, light source, and interchangeable optical objectives.



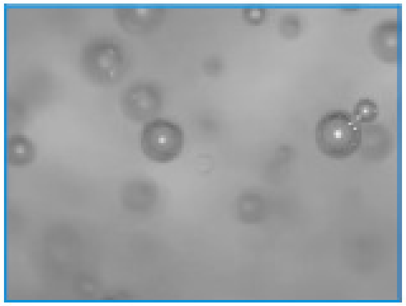
Calibration Grid, 20X



7 µm Fluorescent PS Spheres, 20X

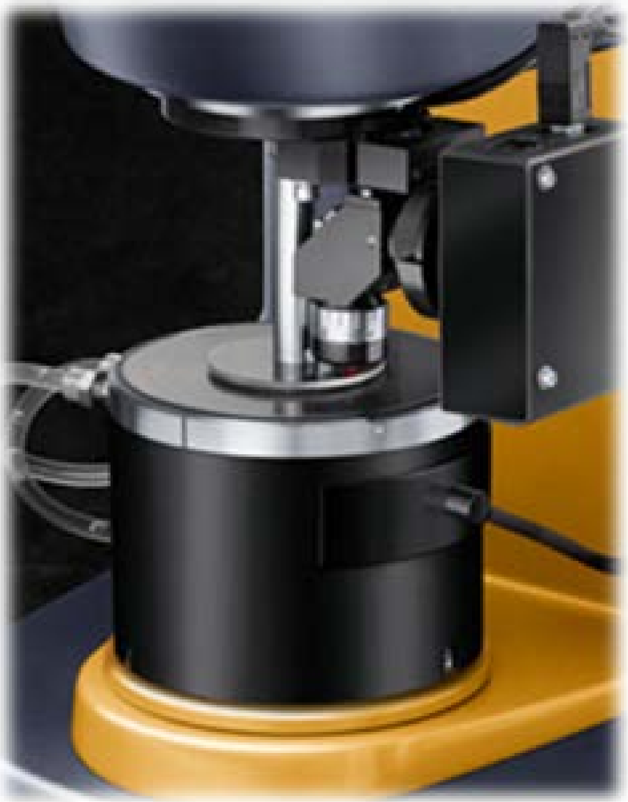


Glass Spheres in PDMS, 20X

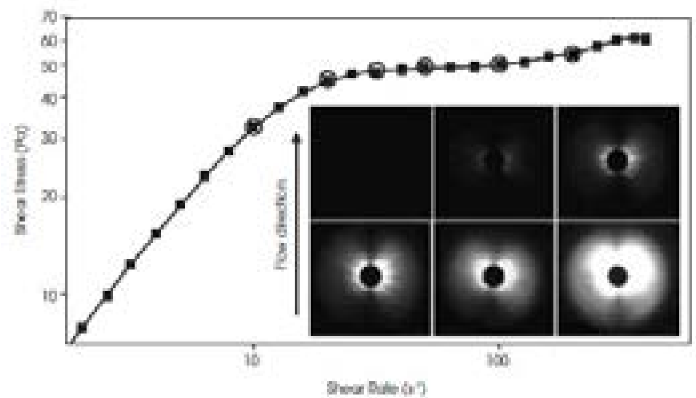


3D scan of hollow glass spheres, 20X

# Small Angle Light Scattering (DHR)



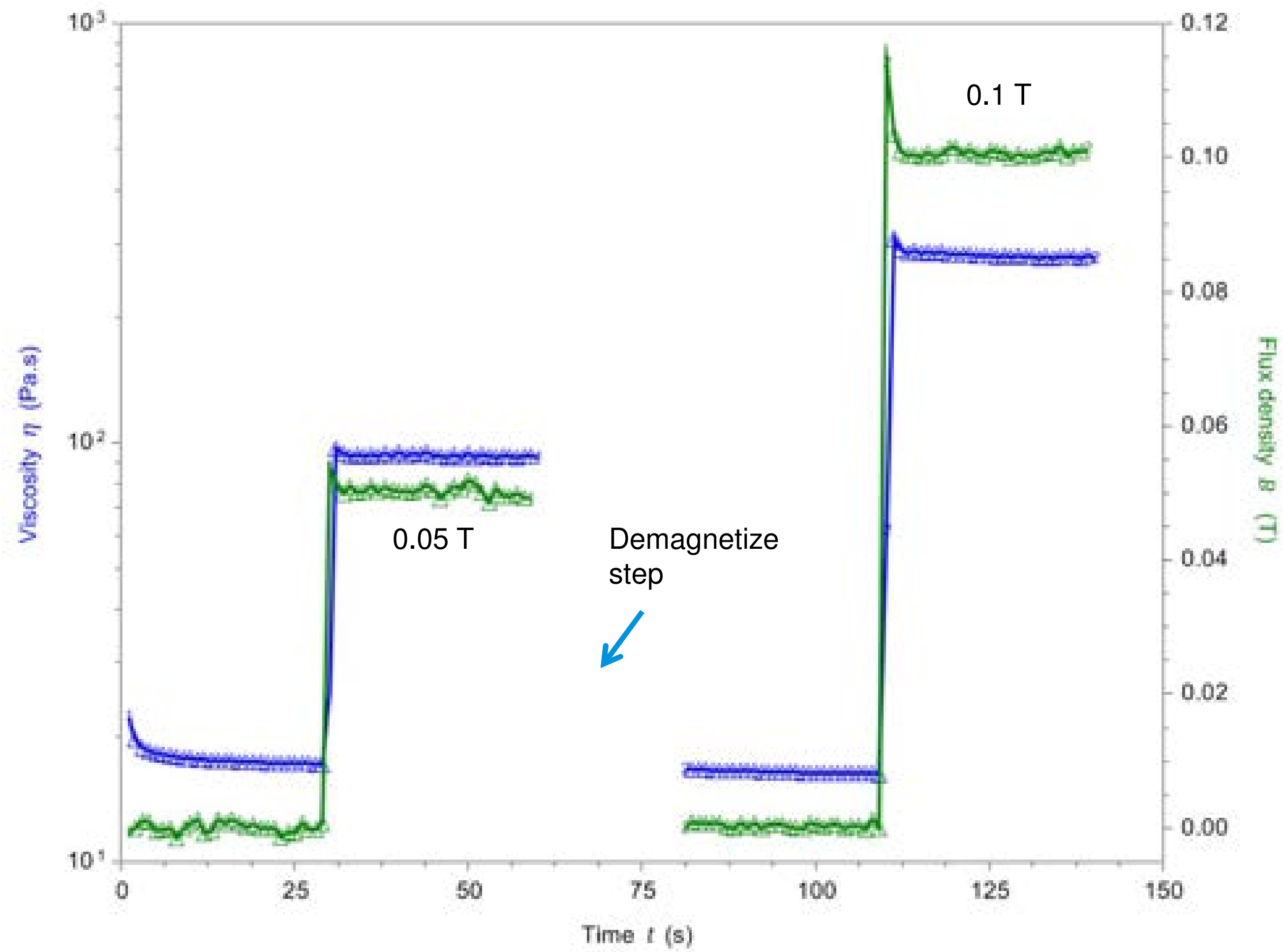
- Simultaneous rheology and structure information
- Laser light creates interference pattern
- Pattern reflects size, shape, orientation and arrangements of objects that scatter



# MagnetoRheology Accessory (DHR)

- The MR Accessory enables characterization of magneto-rheological fluids under the influence of a controlled field
- Applied fields up to 1 T with temperature range of -10 ℃ to 170 ℃ (standard and extended temperature options)
- The system accommodates an optional Hall probe for real-time measurement and closed-loop control of the sample field

# Magneto Rheology Accessory (DHR)




- Lord MR Fluid MRF-140CG (081610) – 300µm gap at 20°C

# Peltier Plate Tribo-rheometry Geometries

Ball on three Plates

Three Balls on Plate

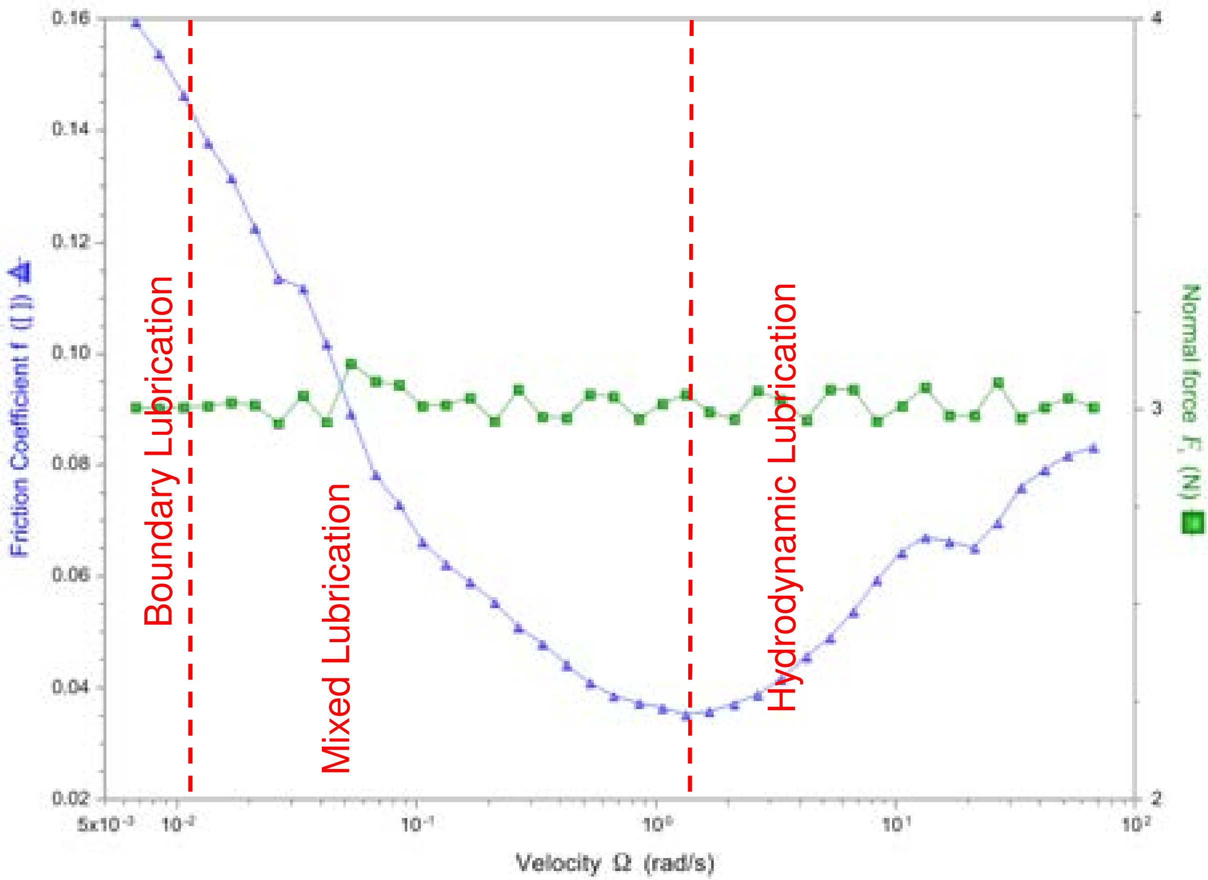
**Also available for ETC**

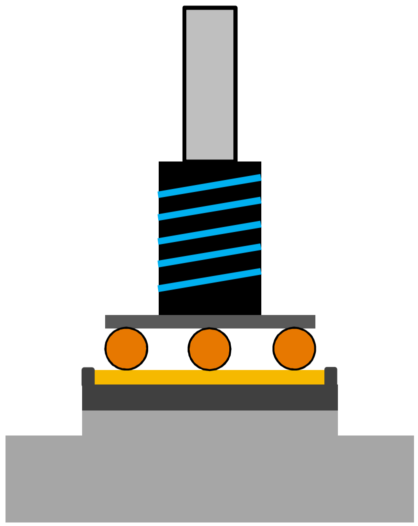
Ring on Plate

Ball on three Balls

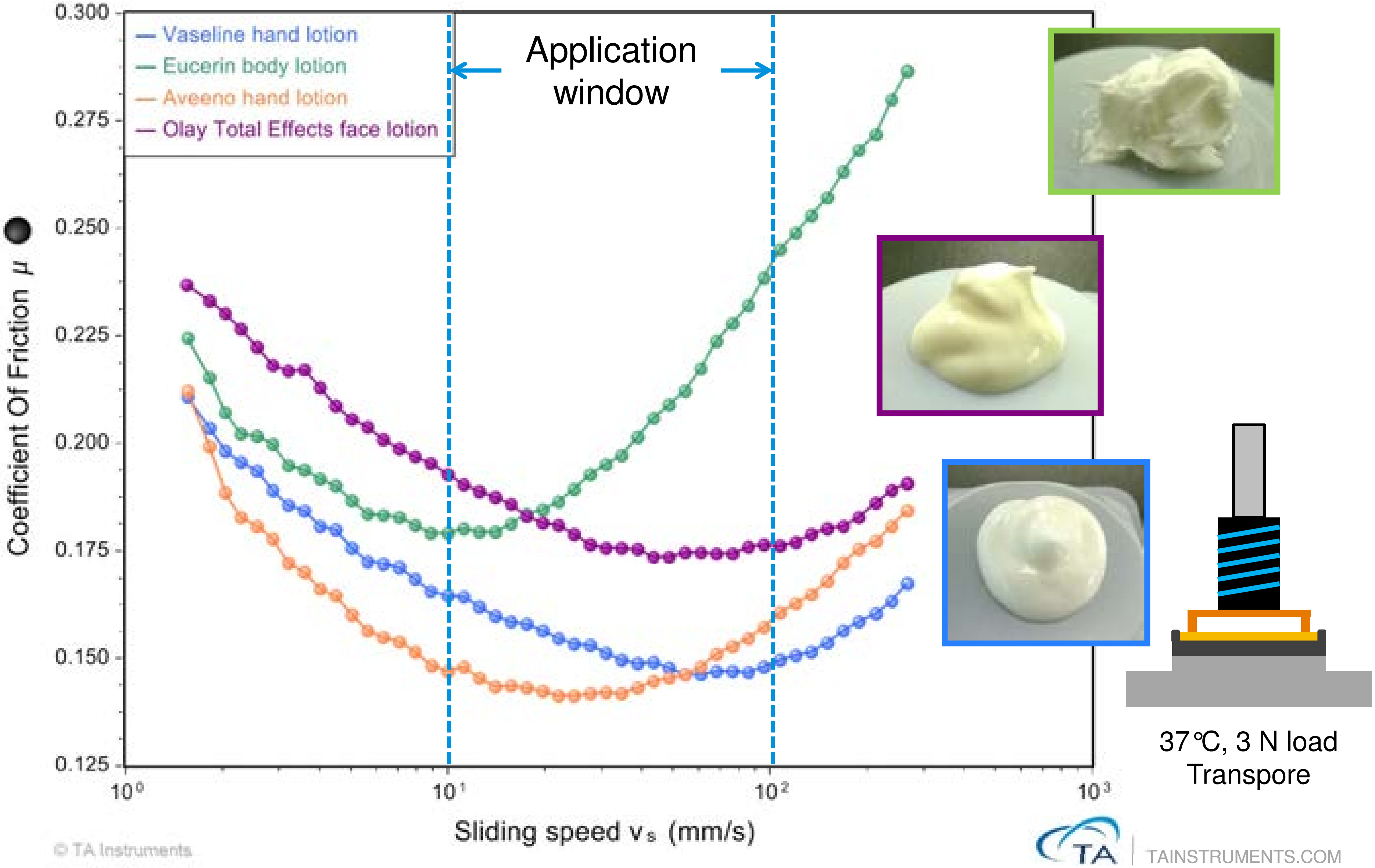
# Coefficient of Friction Measurement

PVC on Steel with 2.0 Pa.s oil as lubricant
Geometry: 3 Balls on Plate
Temperature: 25℃, Procedure: Flow ramp



# Ring on Plate: Lotions - CoF




37 ℃, 3 N load
Transpore

TAINSTRUMENTS.COM

# What Does TA Instruments Make?

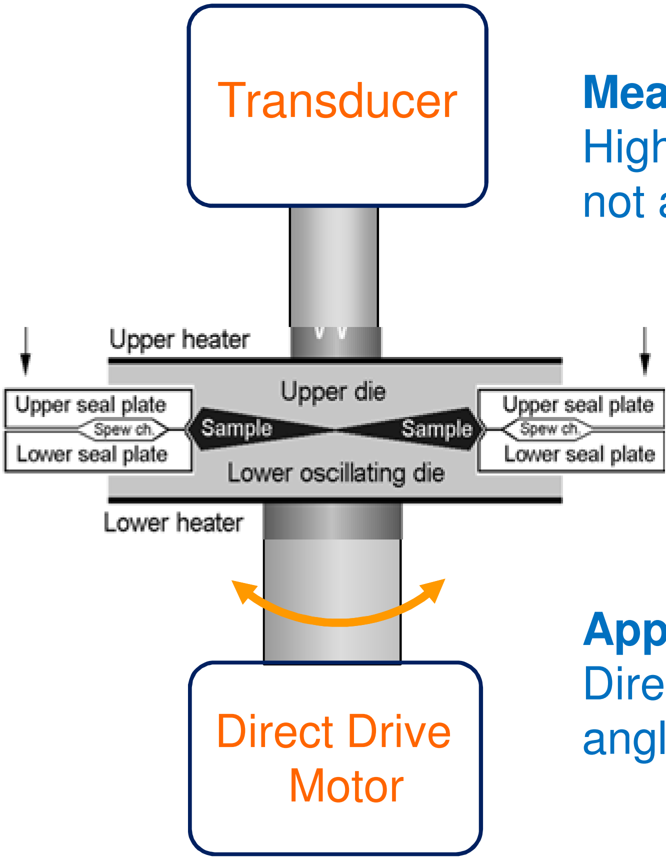
- Differential Scanning Calorimeters
- Thermogravimetric Analyzers
- Simultaneous Differential Thermal Analyzers
- Microcalorimeters of many types
- Dilatometers and Thermomechanical Analyzers
- Thermal Diffusivity
- Thermal Conductivity
- Mechanical Testers
- Dynamic Mechanical Analyzers
- Rotational Rheometers
- **Rubber Rheometers**

# Rubber Rheometers

# Rubber Rheology

- Mooney Viscometer
  - Widely used in the rubber industry.
- MDR: Moving Die Rheometer
  - Rotorless cure meter (improvement on ODR), used to measure vulcanization of rubber compounds.
- RPA: Rubber Process Analyzer
  - Fully capable rheometer for characterizing elastomers and rubber compounds.
  - Capabilities beyond MDR
    - Frequency Sweeps
    - Strain Sweeps
    - Temperature Ramps
    - Stress Relaxation
- Automated Hardness and Density testers, Autoloader for MDR and RPA

# RPA (Rubber Process Analyzer)



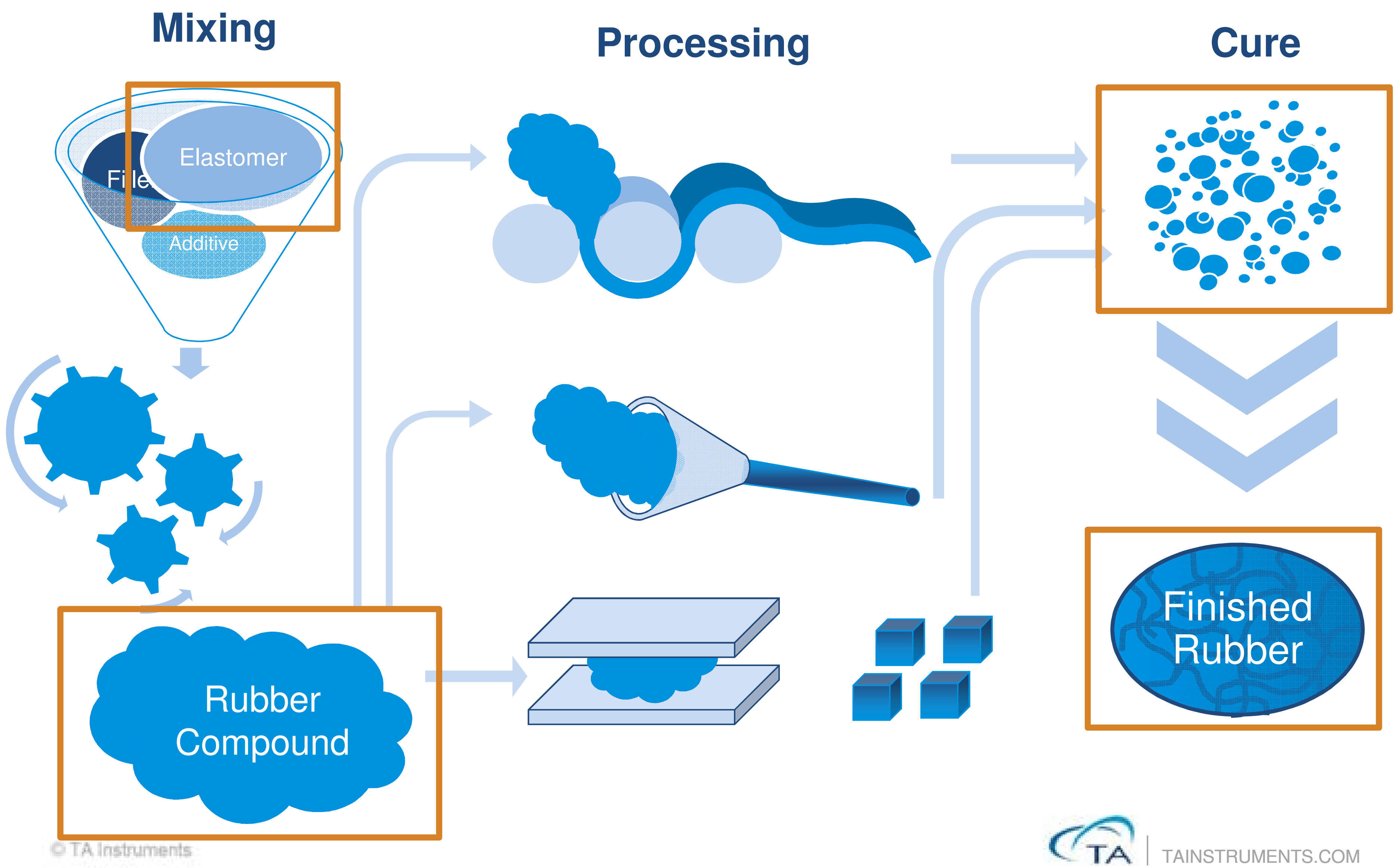

**Measured Torque**
High Stiffness transducer, not affected by compliance.

**Applied Deformation**
Direct, precise control of angle and frequency.

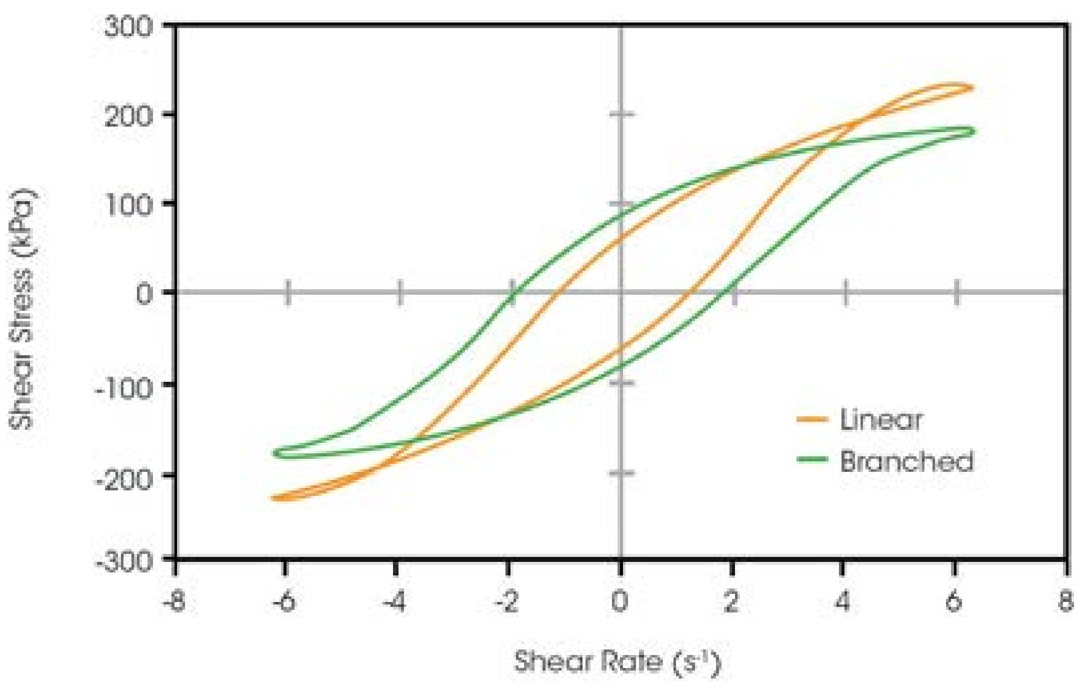
***RPA Elite***

**Information Provided:** Storage Modulus G', Loss Modulus G", Tan Delta and Relaxation Modulus G*(t)* as a function of strain, frequency, time and temperature.

# Rubber Production: Where does a Rheometer fit?

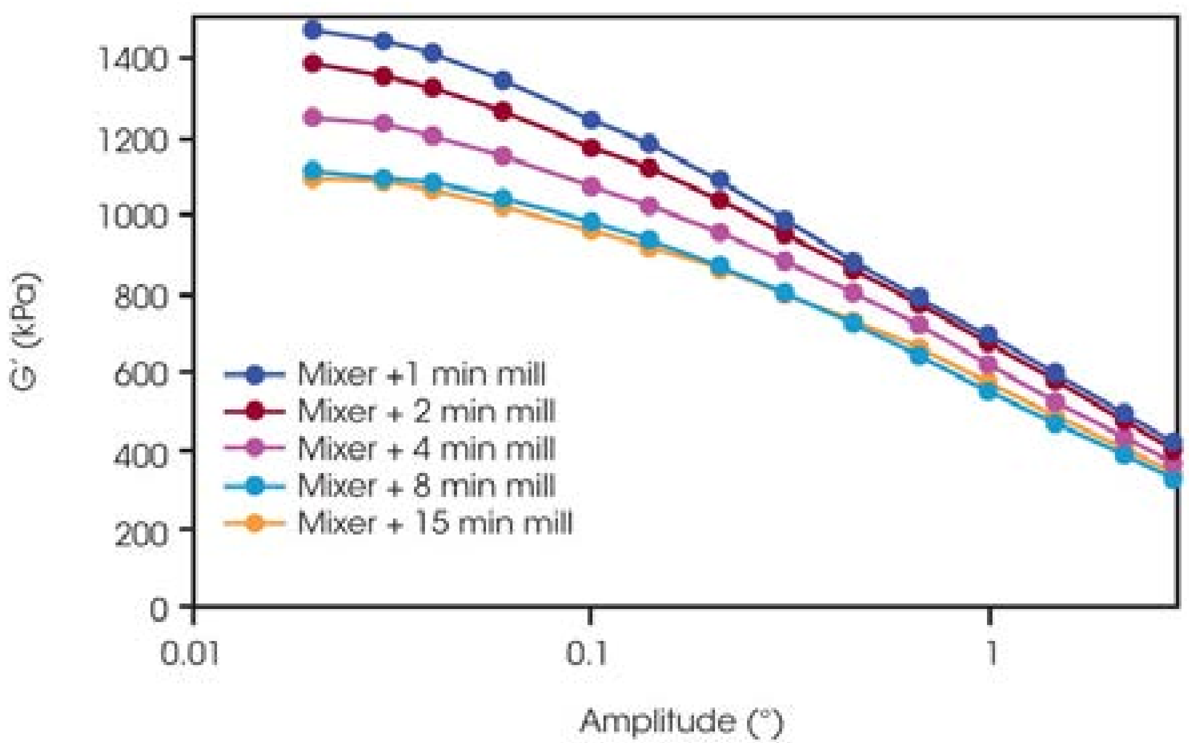


# RPA LAOS- Linear and Branched Polymer



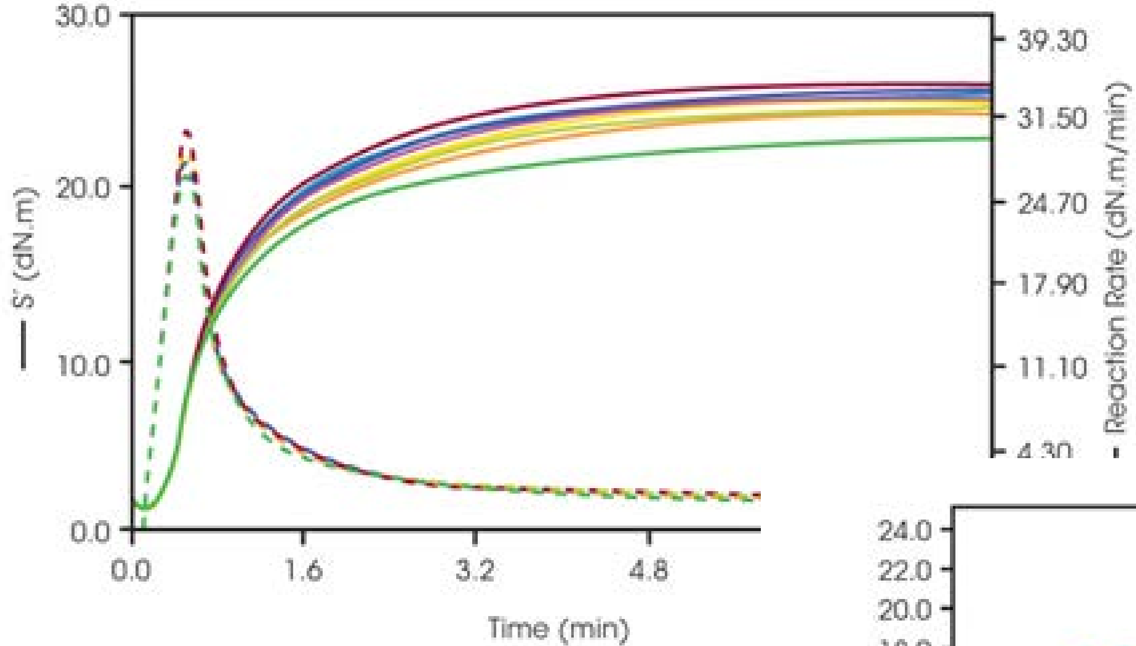

- Large Amplitude Oscillatory Shear (LAOS) measures outside of the Linear Viscoelastic Region
- LAOS testing is sensitive to differences in polymer architecture not detected in Linear Viscoelastic measurements

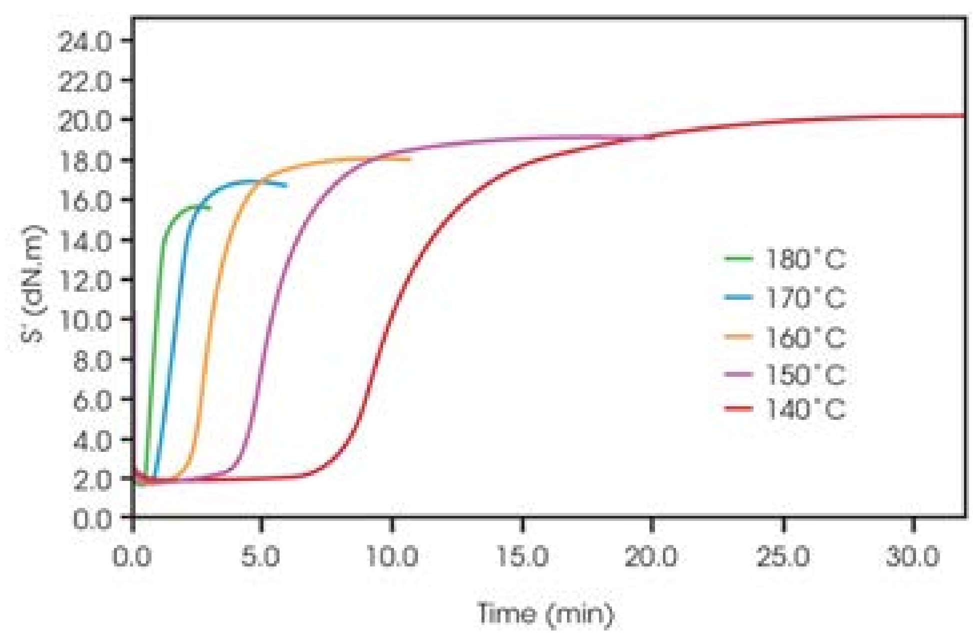
# RPA Strain Sweep- Dispersion of Filler




- The modulus of filled compounds depends on how the compound is processed.
- Longer milling time produces a better dispersion and breaks down agglomerates of carbon black.
- Testing mechanical properties helps to determine best milling time.

# Curing of Rubber



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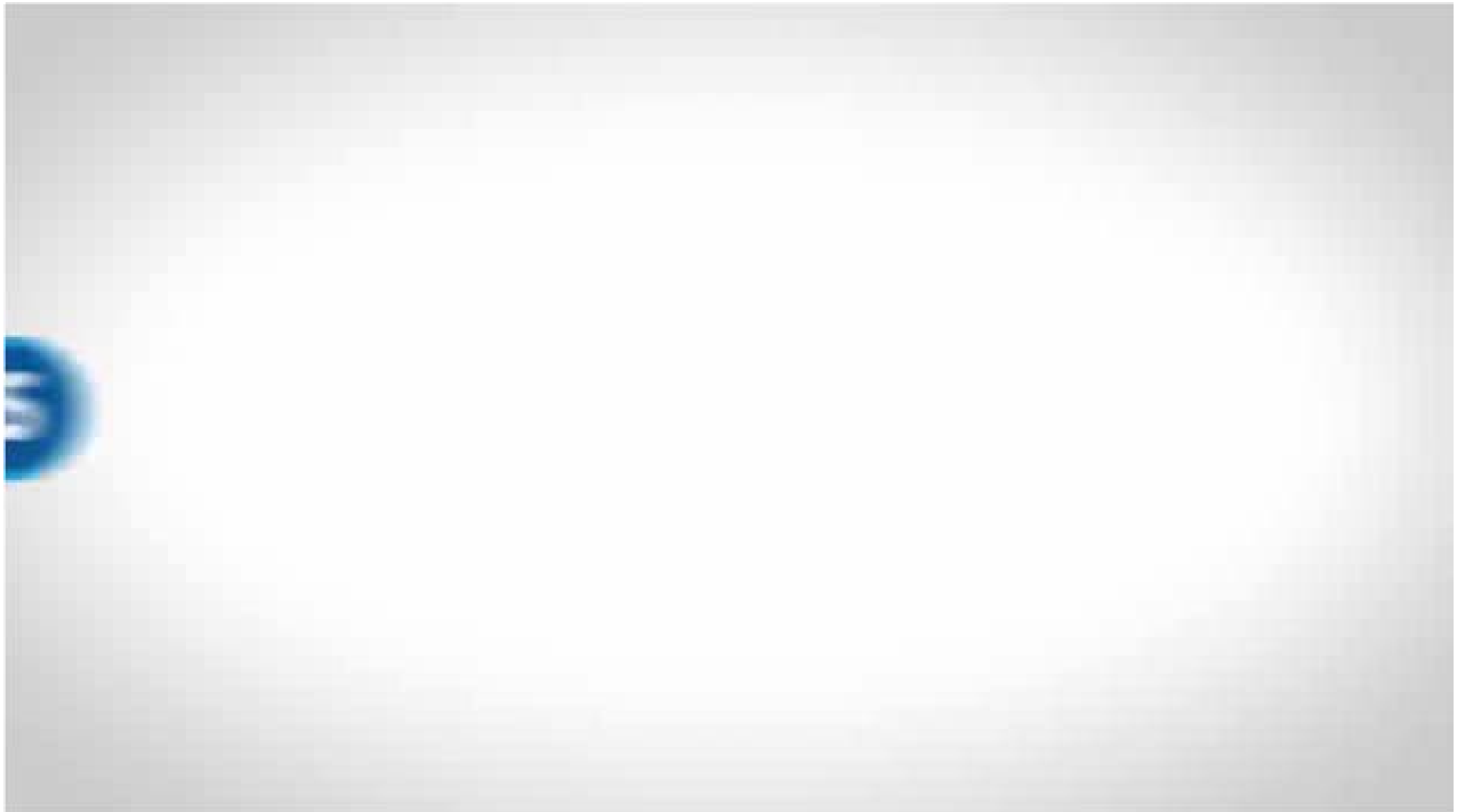
# ESG Mechanical Testers

# Load Frame Instruments

# Fatigue Testing



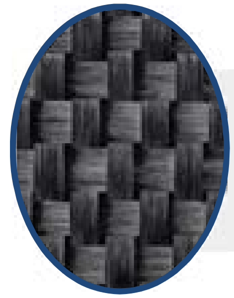
# TestBench Instruments

# Linear Motor Technology

- Direct Drive Electromagnetics
  - Highest Accelerations
  - Precise
  - Clean & Quiet
  - Unmatched Durability

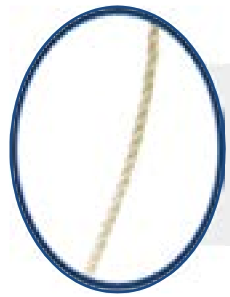

# Materials Application Examples

## Plastics
- TPE Fatigue
- TPU Fatigue



## Composites
- Carbon Composite Fatigue
- E-glass Composite Fatigue

## Elastomers-Rubber
- Tire Rubber DMA
- Tire Rubber Heat Build Up
- Tire Rubber Fatigue
- Shoe Foam Fatigue
- Molded Silicon Characterization



## Fibers-Textiles
- Smart Textile Characterization
- Spider Silk Pull-to-Failure

## Metals
- Hardened Steel Fatigue
- Stent Wire Fatigue
- Nuclear Fuel Rod Fatigue
- Solder Pull-to-Failure

# Thank You

The World Leader in Thermal Analysis, Rheology, and Microcalorimetry