Dynamic Mechanical Analysis

Basic Theory & Applications Training

Agenda

- Basic Theories of Dynamic Mechanical Analysis
- DMA instrumentation
- Calibrations
- Linear viscoelasticity
- Available test modes on DMAs
- Applications of Dynamic Mechanical Analysis

Is DMA Thermal Analysis or Rheology?

Definitions

- ***Thermal Analysis***
  - measurement as a *function of temperature or time*.
- ***Rheology***
  - the science of *stress* and *deformation* of matter.
- ***DMA*** is the general name given to an instrument that mechanically *deforms a sample* and measures the sample response. The response to the deformation can be monitored as a *function of temperature or time.*

What can be Studied with DMA?

- Composition
  - Degree of cross-linking
  - Comparison of crystallinity levels
  - Molecular Orientation
  - Effect of Filler
- Physical Properties
  - Prediction of impact resistance
  - Testing of creep or cold flow
  - Stress relaxation behavior
  - Cure behavior
- Viscoelastic Properties
  - Storage Modulus
  - Loss Modulus
  - Tan Delta
  - Glass Transition (T_g)
  - Sub-T_g molecular motions (beta and gamma relaxations)
- Lifetime predictions using Time Temperature Superpositioning

What does a DMA do?

Measures the mechanical properties of a sample as it is deformed over a range of **stress**, **strain**, **time and temperature**

- Can either apply **Stress** (Force) and measure **Strain** (Displacement), or apply **Strain** and measure **Stress**
- Determines the **Modulus** of the material (**Stress** / **Strain**)
- Controls the **Frequency** (Time) of the deformation to measure viscoelastic properties (**Storage Modulus, Loss Modulus, Tan Delta**)
- **Temperature** controlled in heating, cooling, or isothermal modes
- Modes of Deformation: Tension, Bending, Compression and Shear

How does a DMA work?

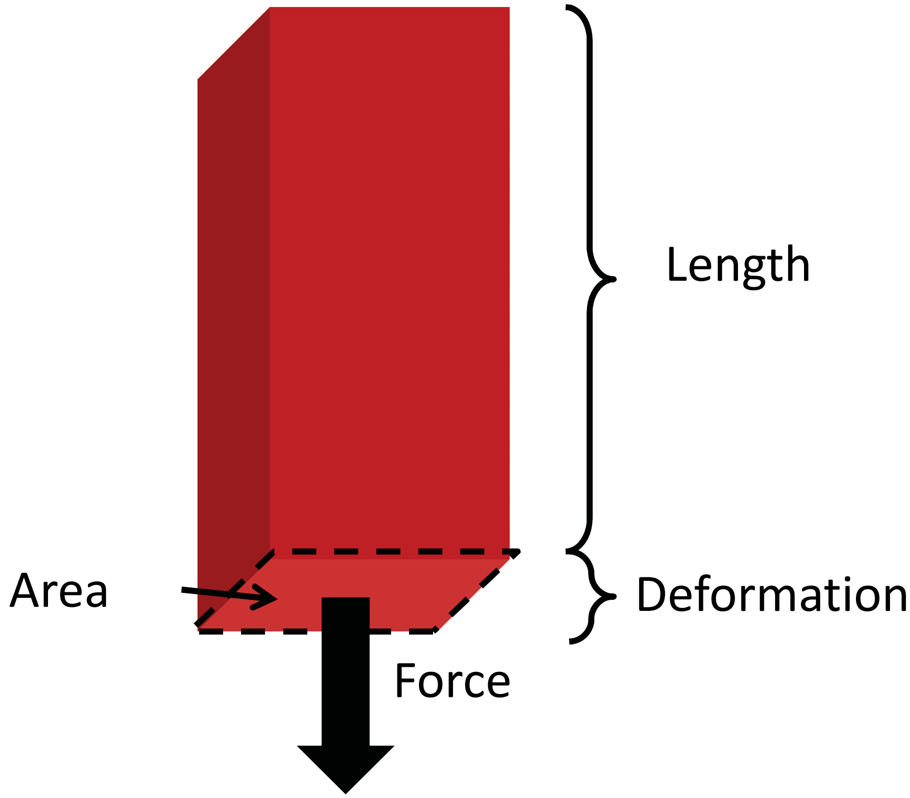
- The DMA measures raw instrument signals
  - Force, Displacement, Stiffness
- Dimensions of the sample are recorded
  - Length, Width, Thickness
- Software calculates mechanical parameters
  - Stress, Strain, Modulus

$$\text{Stress (Pa)} = \frac{\text{Force(N)}}{\text{Area(m}^2\text{)}}$$

$$\text{Strain} = \frac{\text{Deformation (m)}}{\text{Length (m)}}$$

$$\text{Modulus (Pa)} = \frac{\text{Stress (Pa)}}{\text{Strain}}$$

How does a DMA work?



$$\text{Stress (Pa)} = \frac{\text{Force(N)}}{\text{Area}(m^2)}$$

$$\text{Strain} = \frac{\text{Deformation (m)}}{\text{Length (m)}}$$

$$\text{Modulus (Pa)} = \frac{\text{Stress (Pa)}}{\text{Strain}}$$

How does a DMA work?

Instrument Signals		Sample Dimensions		Data
Force (N)	×	Stress Constant (Pa/N)	=	Stress (Pa)
Displacement (m)	×	Strain Constant (1/m)	=	Strain (unitless)
Stiffness (N/m)	×	Geometry Factor (1/m)	=	Modulus (Pa)

Q800

DMA Testing Modes

- Subjects the sample to either oscillatory deformation, oscillatory force, static deformation or static force

	Static	Oscillation
Stress Controlled	Iso-Force Force Ramp Creep	Multi Stress Multi Frequency-Stress
Strain Controlled	Iso Strain Strain Ramp Stress Relaxation	Multi Strain Multi Frequency-Strain

Basic Parameters and Units

Stress = Force /Area [Pa, or dyne/cm^2]

σ = tensile stress, τ = shear stress

Strain = Geometric Shape Change [unitless]

ε = tensile strain, γ = shear strain

Modulus = Stress / Strain [Pa or dyne/cm^2]

E = Young's or Tensile, **G** = Shear Modulus

Compliance = Strain / Stress [1/Pa or cm^2/dyne]

Typically denoted by **J**

Viscosity = Stress /Strain Rate [Pa·s or Poise]

Denoted by η

SI × 10 = CGS units

DMA Instrumentation

DMAs from TA Instruments

RSA G2

Controlled Strain
SMT

Q800

Controlled Stress
CMT

ARES G2
and DHR

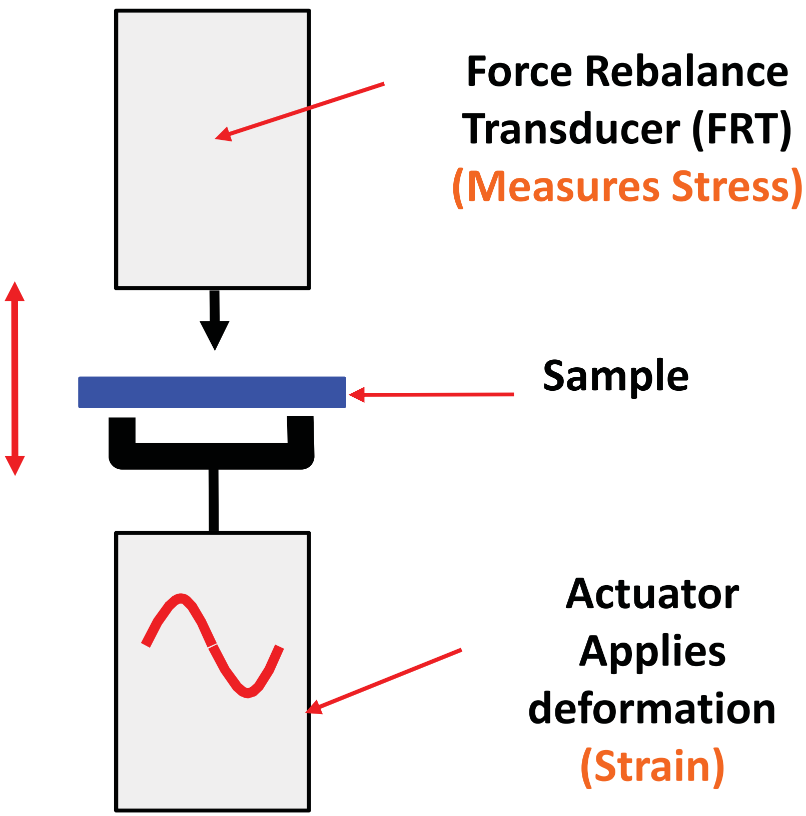
DMA mode
(oscillation)

DMAs from TA Instruments

RSA G2

Controlled Strain

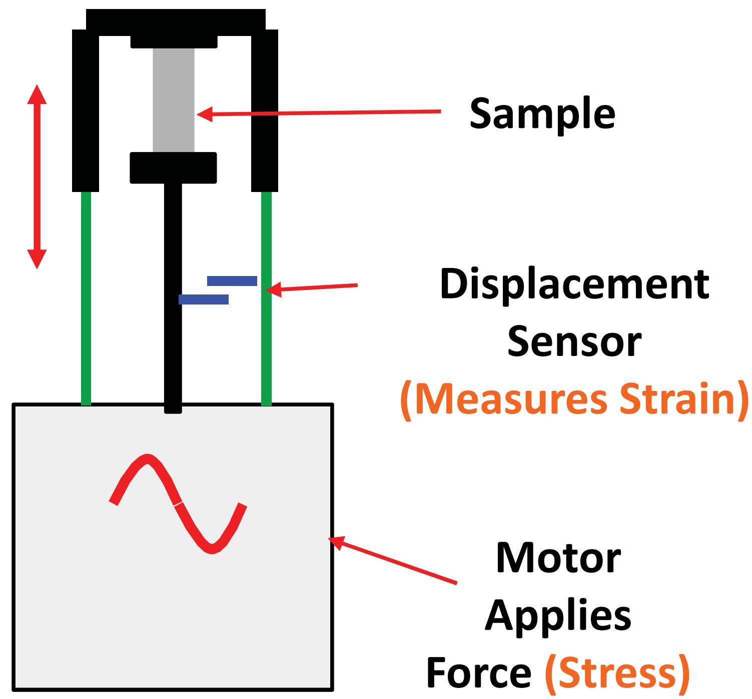
SMT – Separate Motor & Transducer



Q800

Controlled Stress

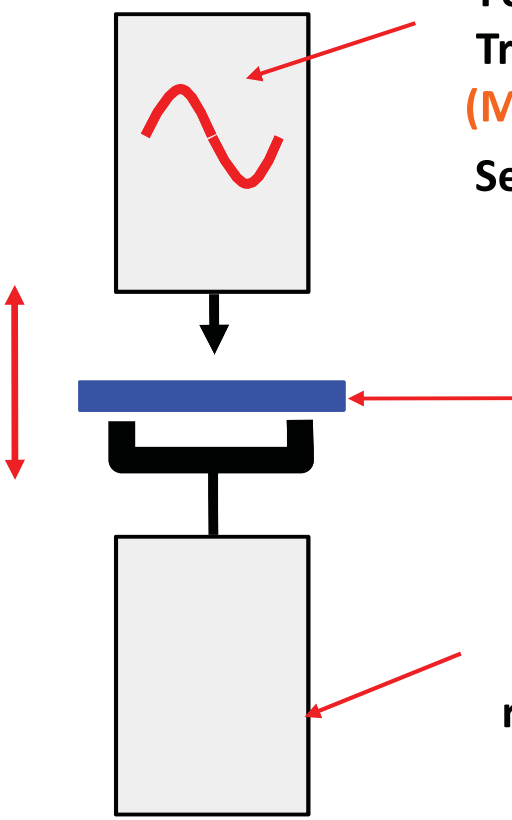
CMT – Combined Motor & Transducer



DMAs from TA Instruments

ARES G2 and DHR DMA

CMT – Combined Motor & Transducer



Force Rebalance Transducer (FRT) (Measures Stress)

Servo control on Null position (Strain)

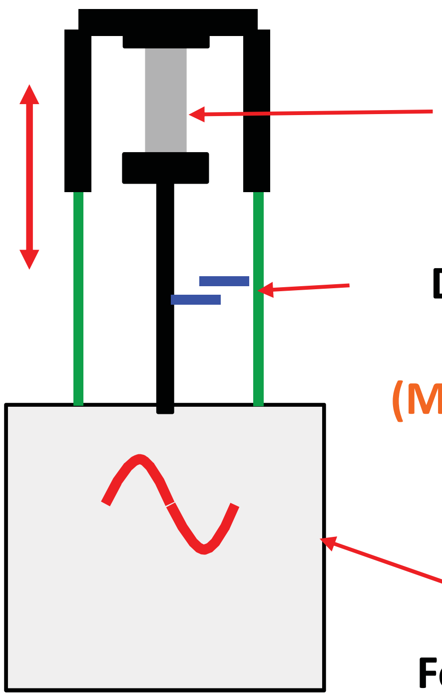
Sample

Bottom Actuator remains locked during DMA function

Q800

Controlled Stress

CMT – Combined Motor & Transducer

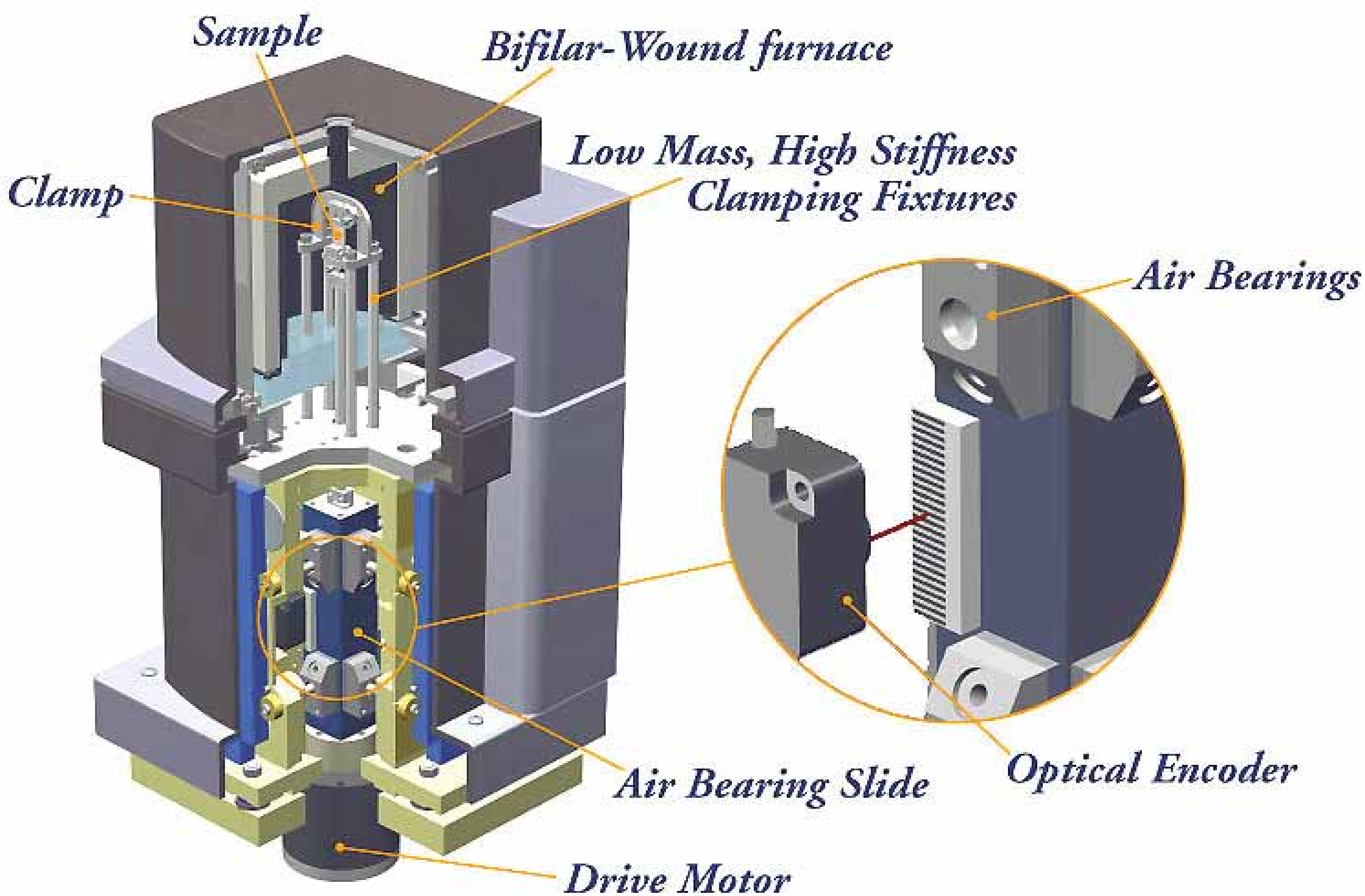


Sample

Displacement Sensor (Measures Strain)

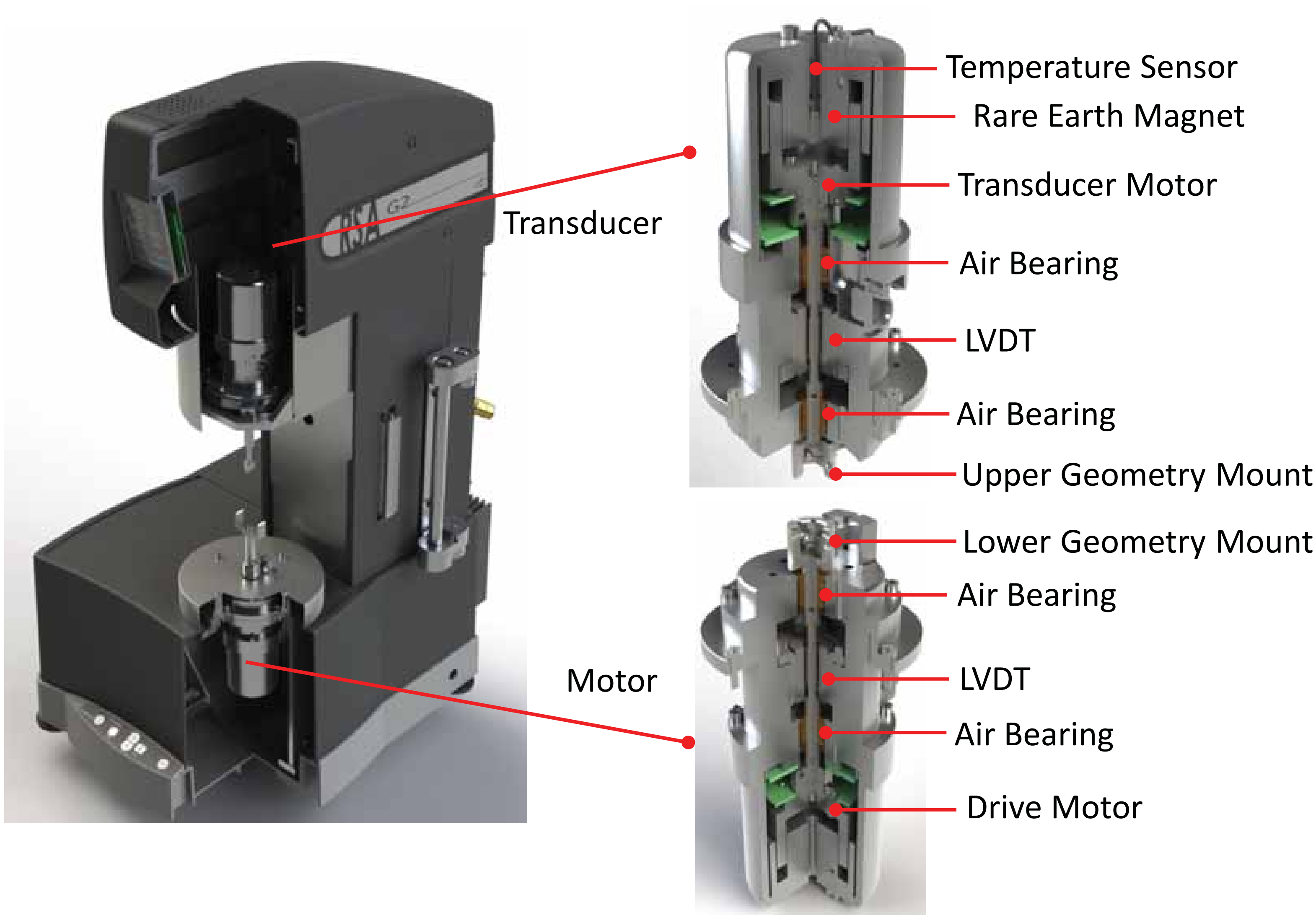
Motor Applies Force (Stress)

DMA Q800: Schematic



Q800: Humidity Option

RSA G2: Schematic Dual Head Design

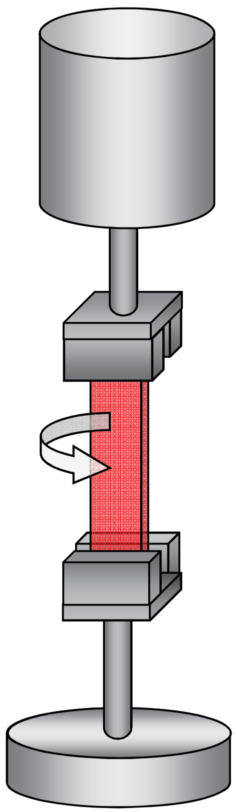


DMA Specifications

	RSA G2	Q800	ARES G2 DMA	DHR DMA (optional)
Max Force	35N	18N	20N	50N
Min Force	0.0005N	0.0001N	0.001N	0.1N
Frequency Range	1e-5 to 628 rad/s ($1.6e^{-6}$ to 100 Hz)	0.01 to 1250 rad/s ($1.6e^{-3}$ to 200 Hz)	$6.3e^{-5}$ to 100 rad/s ($1.0e^{-5}$ to 16 Hz)	$6.3e^{-5}$ to 100 rad/s ($1.0e^{-5}$ to 16 Hz)
Dynamic Deformation Range	+/- 0.05 to 1,500µm	+/- 0.5 to 10,0000µm	+/- 1 to 50 µm	+/- 1 to 100 µm
Control Stress/Strain	Control Strain (SMT)	Control Stress (CMT)	Control Strain (CMT)	Control Stress (CMT)
Heating Rate	0.1°C to 60°C/min	0.1°C to 20°C/min	0.1°C to 60°C/min	0.1°C to 60°C/min
Cooling Rate	0.1°C to 60°C/min	0.1°C to 10°C/min	0.1°C to 60°C/min	0.1°C to 60°C/min

TA

Torsion and DMA Measurements on Rheometers



- Torsion and DMA geometries allow solid samples to be characterized in a temperature controlled environment
  - DMA functionality is standard with ARES G2 and optional DHR

$$E = 2G(1 + \nu) \qquad \nu : \text{Poisson's ratio}$$

Modulus: G', G'', G^*

Rectangular and cylindrical torsion

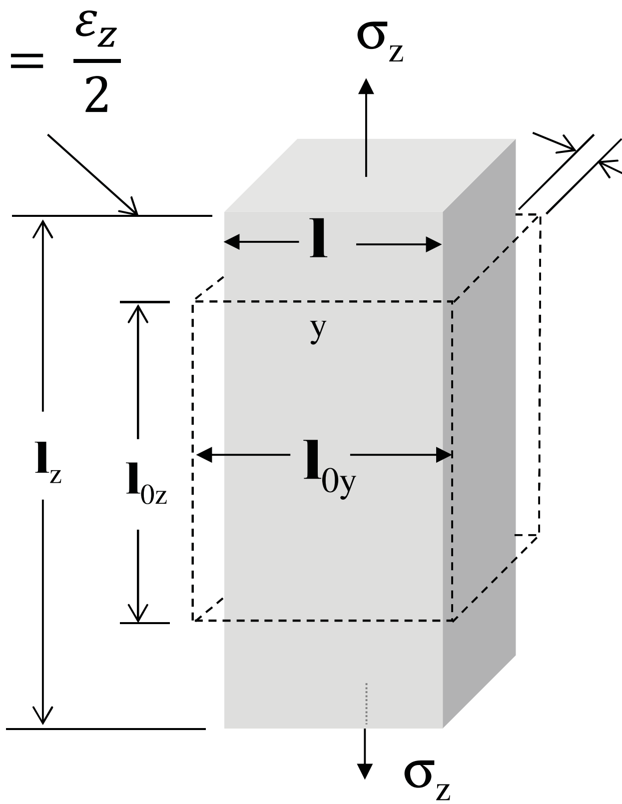
Modulus: E', E'', E^*

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

Poisson's Ratio

- Poisson's ratio, ν, is the ratio of transverse to axial strain
- Value in the range of 0.2 to 0.5

$$\frac{l_z - l_{0z}}{2} = \frac{\varepsilon_z}{2}$$



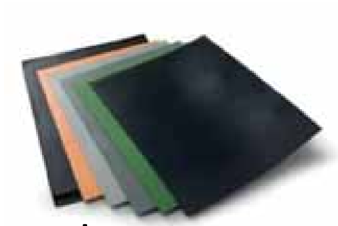
$$\frac{l_y - l_{0y}}{2} = \frac{-\varepsilon_y}{2}$$

Poisson's Ratio

$$\nu = \frac{-\varepsilon_y}{\varepsilon_z}$$

Clamps

- By changing the clamp, we can test a range of different materials: solid bars, elastomers, soft foams, thin films and fibers
- Automatic compliance calibration (and correction) **for each clamp**



Elastomers

Films

Fibers

Gels

Solid Polymers

Foams

Composites

Polymer Melts

Clamps for Q800

S/D Cantilever

Tension-Film

Shear-Sandwich

Submersible Compression

Submersible 3 Pt Bend

3-Point Bending

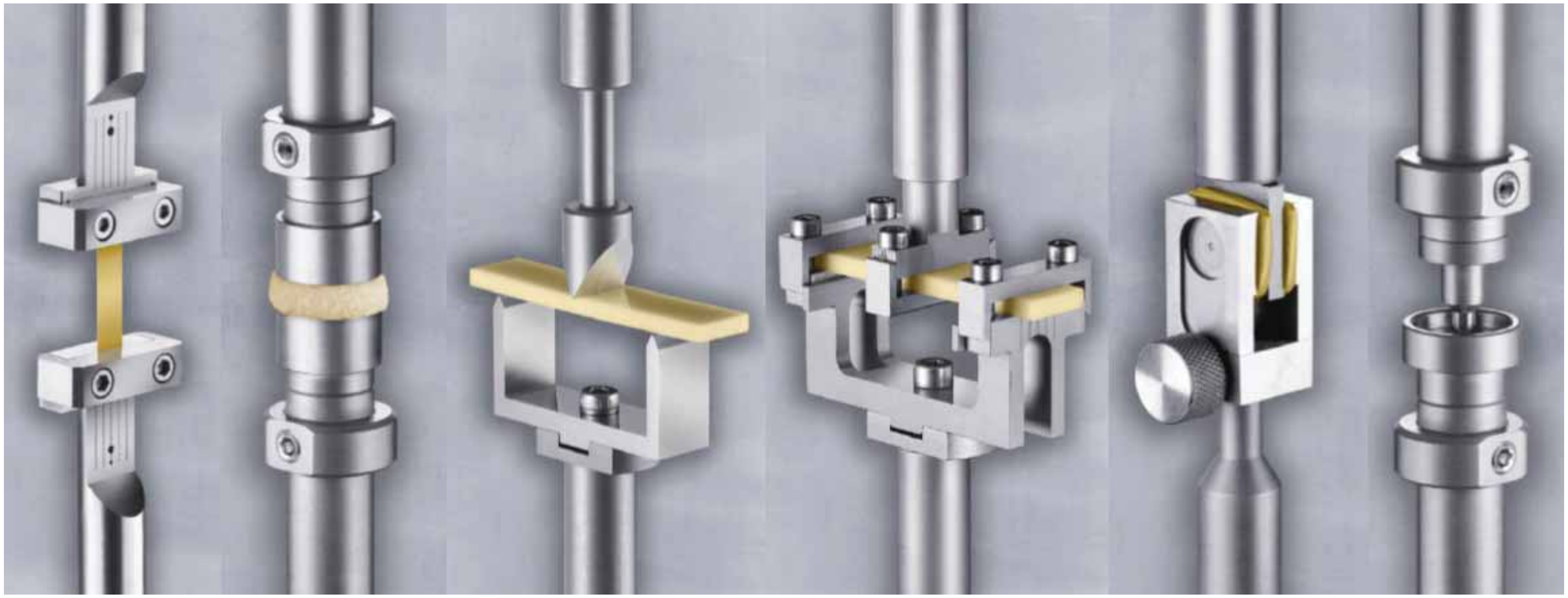
Tension-Fiber

Compression

Submersible Tension

Clamps for RSA G2

Film/Fiber 3-Pt Bending Shear Sandwich



Compression Cantilever Contact Lens

RSA G2 Immersion Clamps

Immersion clamp kit offers 3 geometries with temperature control from -10 to 200 °C in the FCO.

Tension

Compression

3 Point Bending

Tension: Up to 25 mm long, 12.5 mm wide and 1.5 mm thick.
Compression: 15 mm in diameter; maximum sample thickness is 10 mm.
Three Point Bending: includes interchangeable spans for lengths of 10, 15, and 20 mm. Maximum sample width is 12.5 mm and maximum thickness is 5 mm.

Choose the Correct Clamp for Testing

Sample Dimension

- Films and fibers: tension clamps
- Bars and cylinders: bending clamps
- O-rings and tablets: compression and/or shear

Deformation Mode:

- E [tension, compression and bending]
- G [shear]

Sample Stiffness:

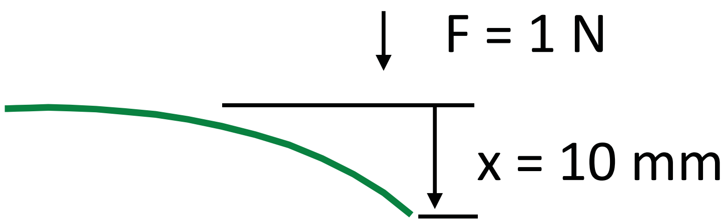
- Machine range fixed: **100 - 10, 000,000 N/m**. Stiffness of sample related to its dimensions [L, w, t]. Stiffness may limit sample size to below clamp maximum.

Sample Stiffness (K)

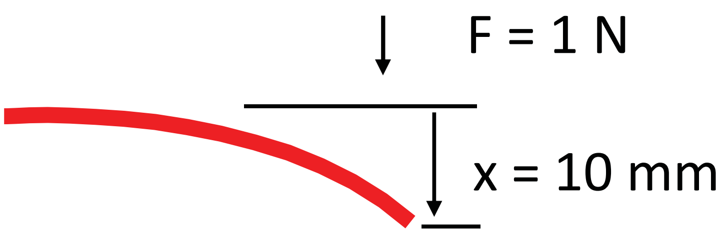
- The **fundamental measurement** of the DMA Q800 is **sample stiffness (K)**
  - **K = Force applied to sample/amplitude of deformation**
- Range of Stiffness on the DMA is **100 to 10,000,000 N/m**
- During an experiment, the raw signals measured are **FORCE** and **AMPLITUDE**.
  - In the RSA, Stress and Strain are calculated from Force and Amplitude
  - In Q800, Stiffness is calculated directly from force and amplitude and modulus is calculated by multiplying by the appropriate geometry factor (GF).
    - **Modulus = Stiffness × Geometry factor = K * GF**

Sample Stiffness and Material Modulus

Thick and Thin Samples Can Have The Same Stiffness

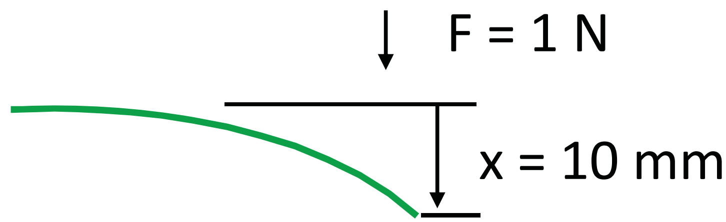


High Modulus Material

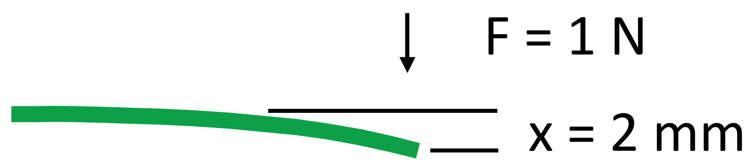


Low Modulus Material

Thick and Thin Samples That Have The Same Modulus

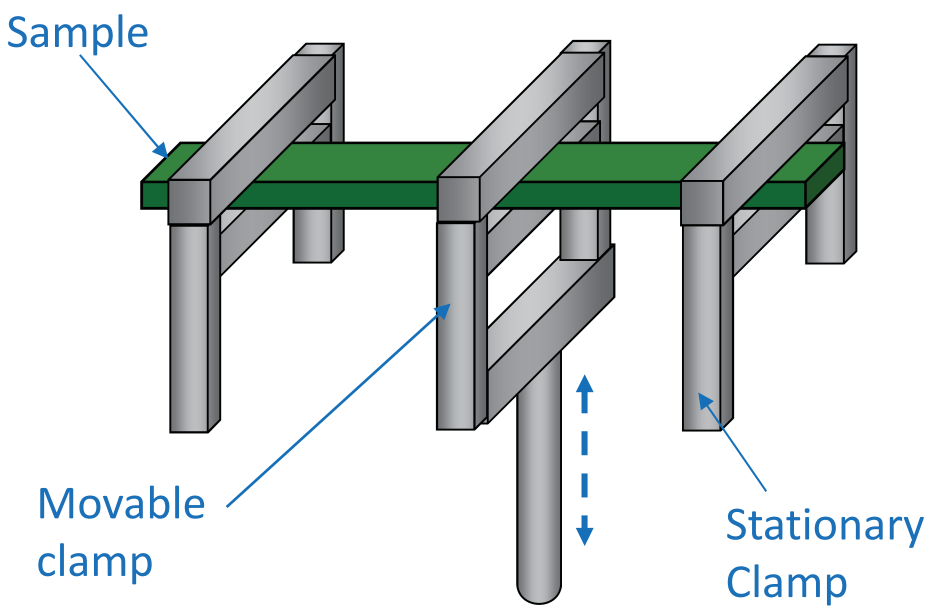


Low Stiffness Sample



High Stiffness Sample

DMA: Dual Cantilever Clamp



- Highly damped materials can be measured
- Best mode for evaluating the cure of supported materials

Geometry Factor - Dual Cantilever Clamp

Modulus = Stiffness × Geometry Factor

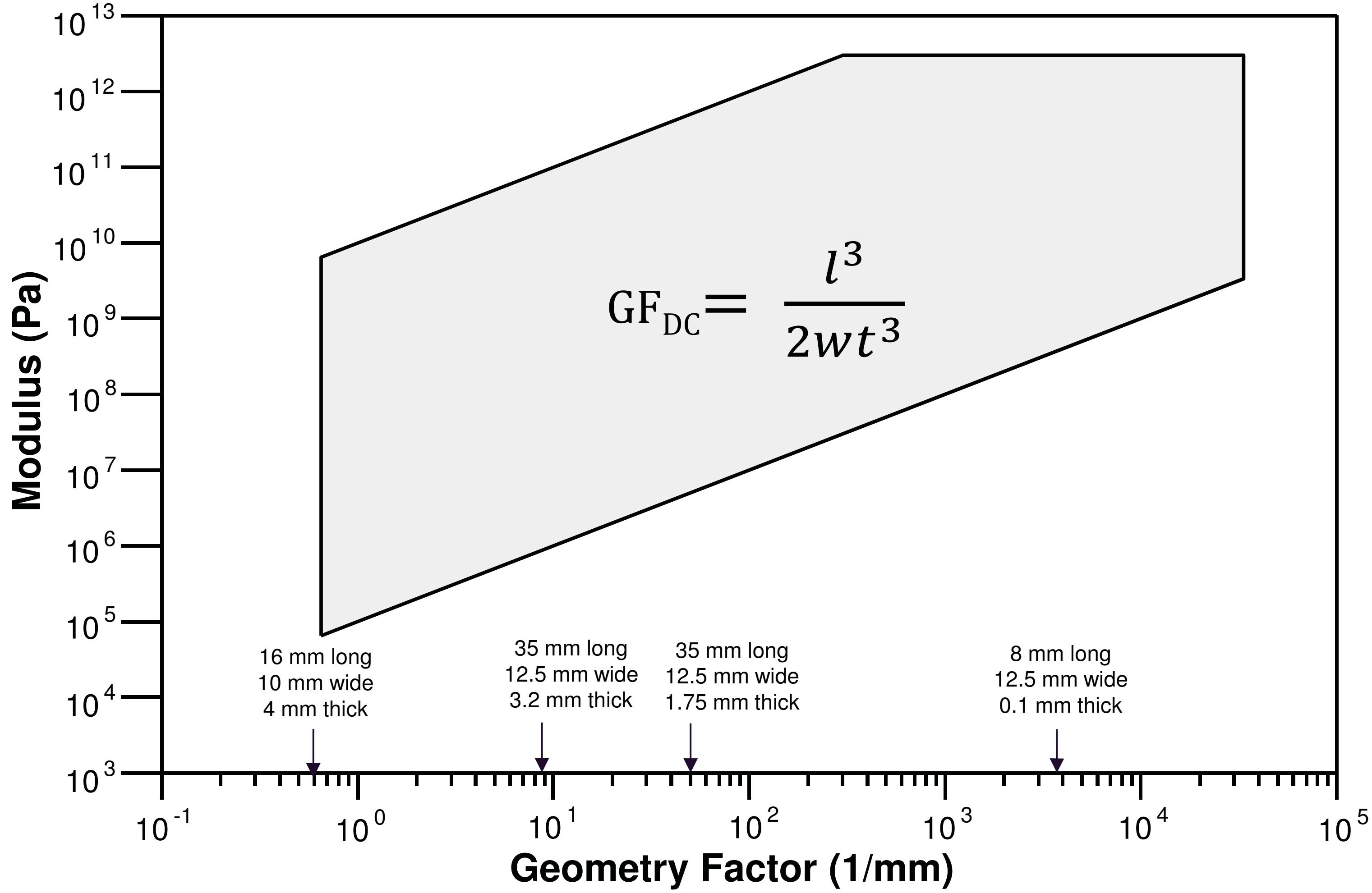
If length/thickness > 10,
Poisson's Ratio term is dropped

$$GF_{DC} = \frac{\cancel{12} \cdot l^3 \left[1 + \cancel{\frac{12}{5}(1+\nu)\left(\frac{t}{l}\right)^2}\right]}{2\ \cancel{24} w t^3}$$

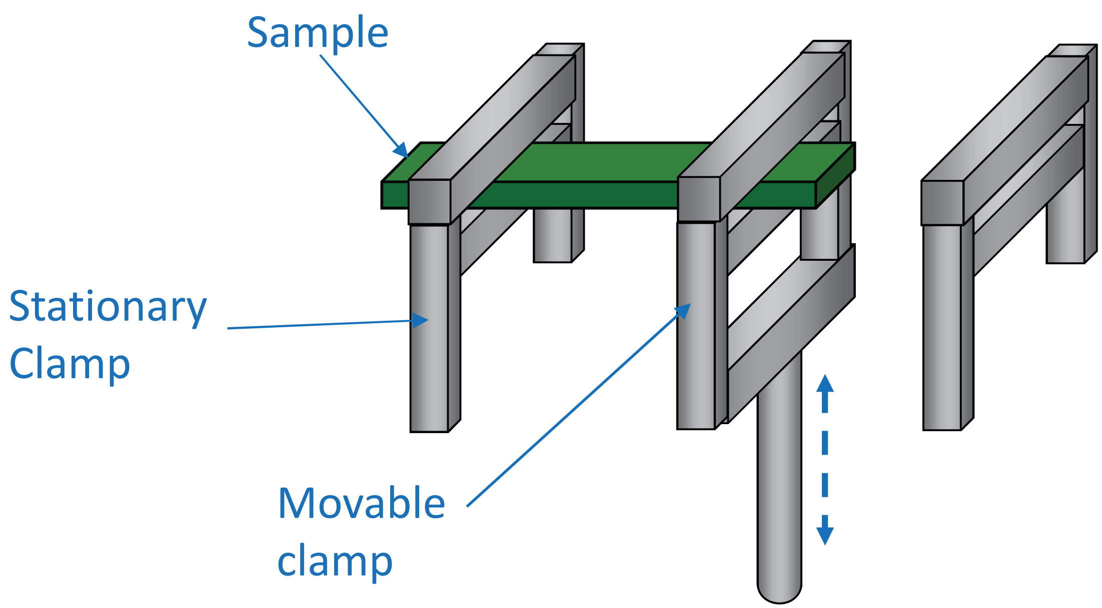
$$GF_{DC} = \frac{l^3}{2wt^3}$$

Operating Range of the Dual Cantilever Clamp

Modulus = Stiffness × Geometry Factor



DMA: Single Cantilever Clamp



- Best general purpose mode (thermoplastics)
- Preferred mode over dual cantilever for most neat thermoplastics (unreinforced), except elastomers
- Clamping torque is important

Geometry Factor - Single Cantilever Clamp

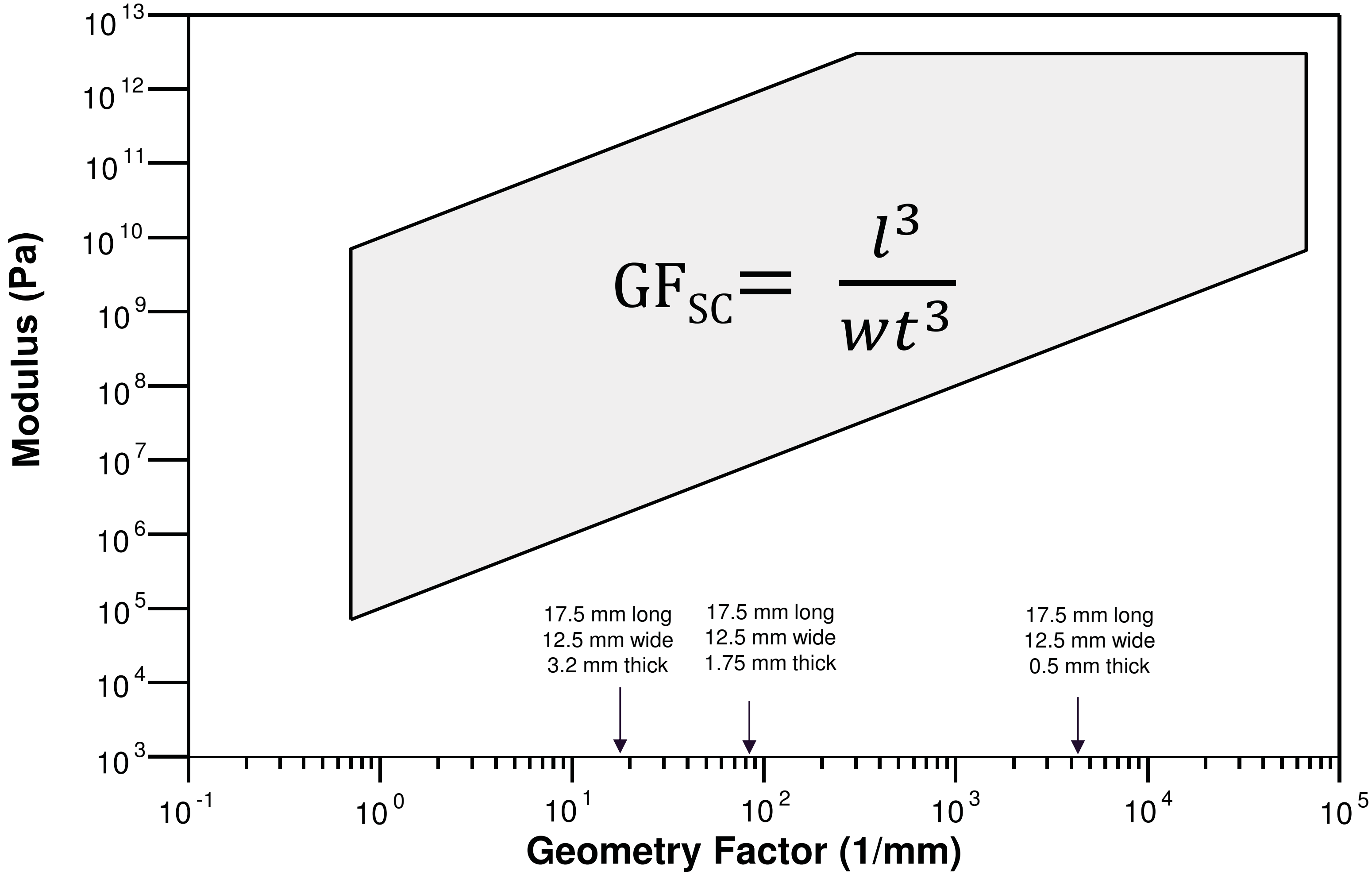
Modulus = Stiffness × Geometry Factor

If length/thickness > 10,
Poisson's Ratio term is dropped

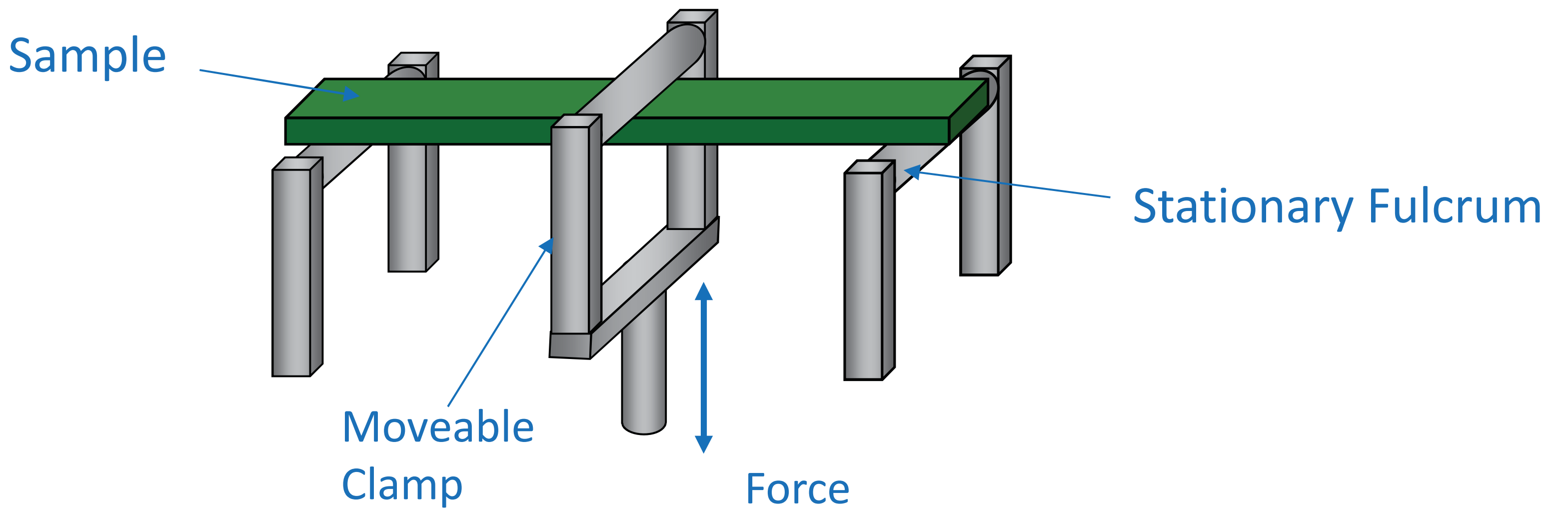
$$GF_{SC} = \frac{12 \cdot l^3 \left[1 + \frac{12}{5}(1+\nu)\left(\frac{t}{l}\right)^2\right]}{12wt^3}$$

$$GF_{SC} = \frac{l^3}{wt^3}$$

Operating Range of the Single Cantilever Clamp



DMA: 3 Point Bend Clamp



- Best mode for measuring medium to high modulus materials
- Conforms with ASTM standard test method for bending
- Purest deformation mode since clamping effects are eliminated

Geometry Factor - 3 Point Bending Clamp

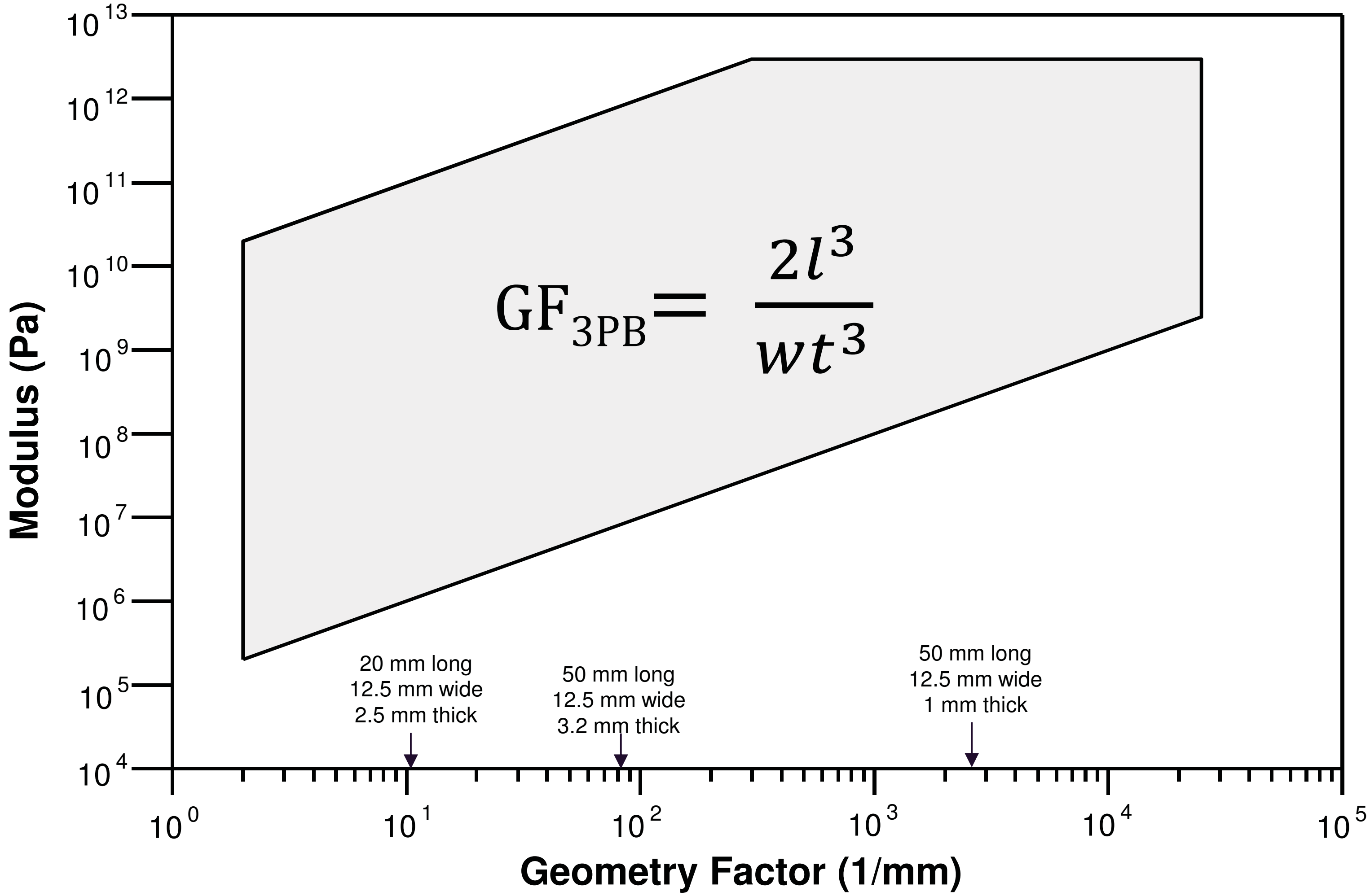
Modulus = Stiffness × Geometry Factor

If length/thickness > 10,
Poisson's Ratio term is dropped

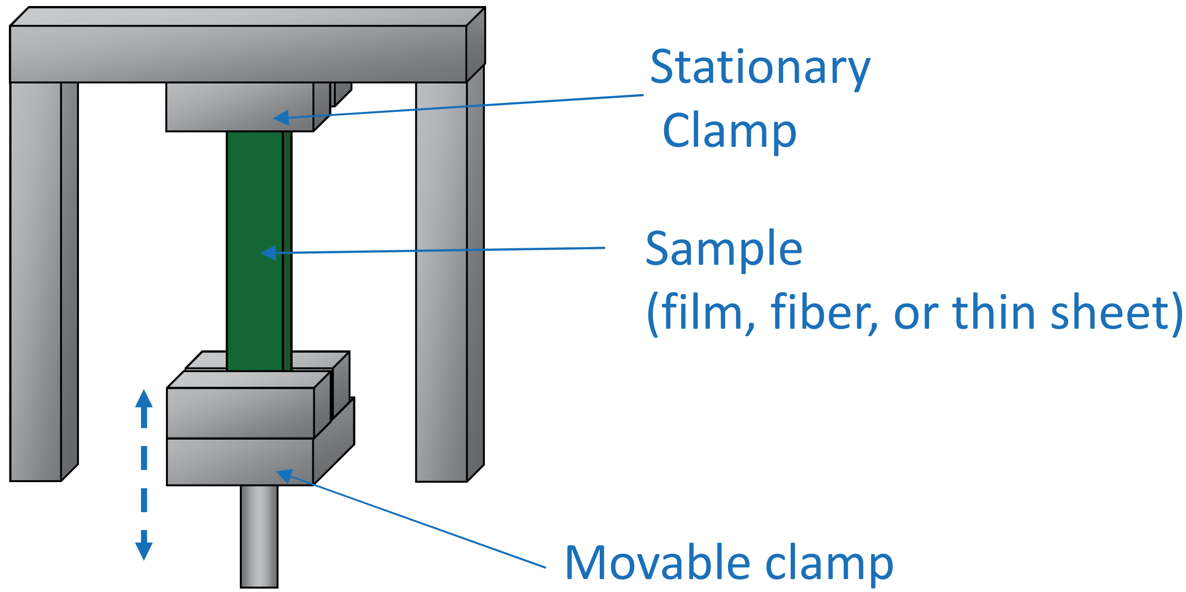
$$GF_{3PB} = \frac{\overset{2}{\cancel{12}} \cdot l^3 \left[1 + \cancel{\frac{6}{10}(1+\nu)\left(\frac{t}{l}\right)^2}\right]}{\cancel{6} w t^3}$$

$$GF_{3PB} = \frac{2l^3}{wt^3}$$

Operating Range of the 3 Point Bending Clamp



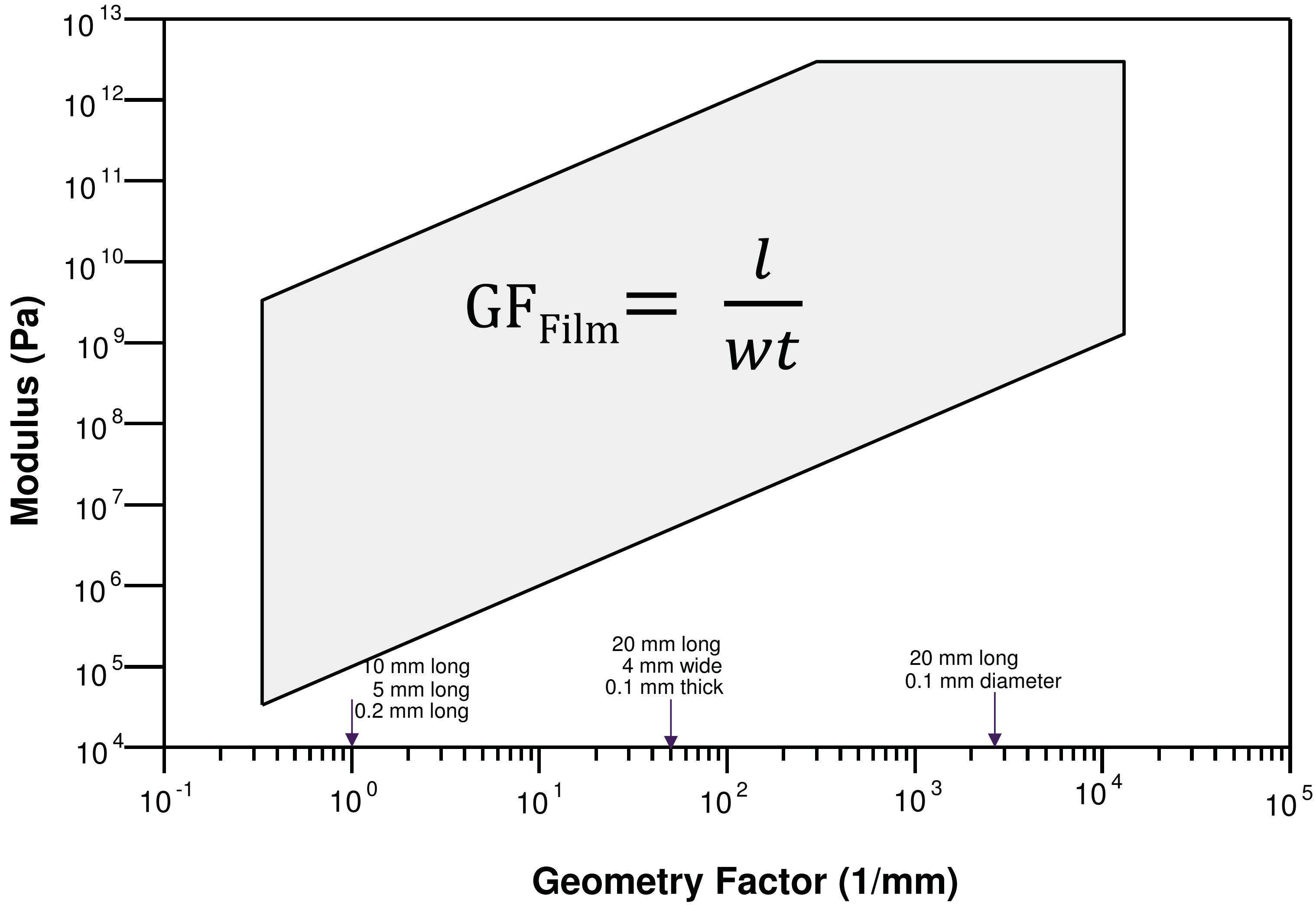
DMA: Film and Fiber Tension Clamp



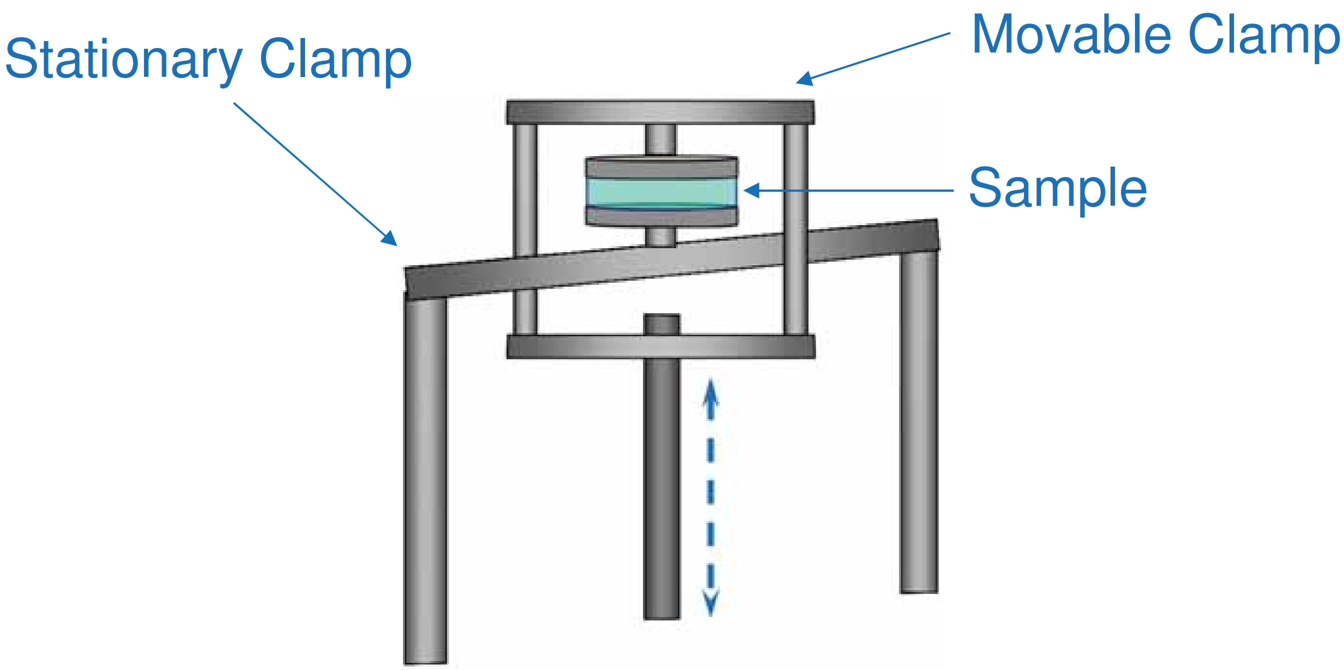
- Best mode for evaluation of thin films and fibers
  - bundle or single filaments
- Small samples of high modulus materials can be measured
- TMA-like constant force and force ramp measurements
  - aka. mini-tensile tester
- Force track and constant force control

Operating Range of the Film/Fiber Tension Clamp

Modulus = Stiffness × Geometry Factor



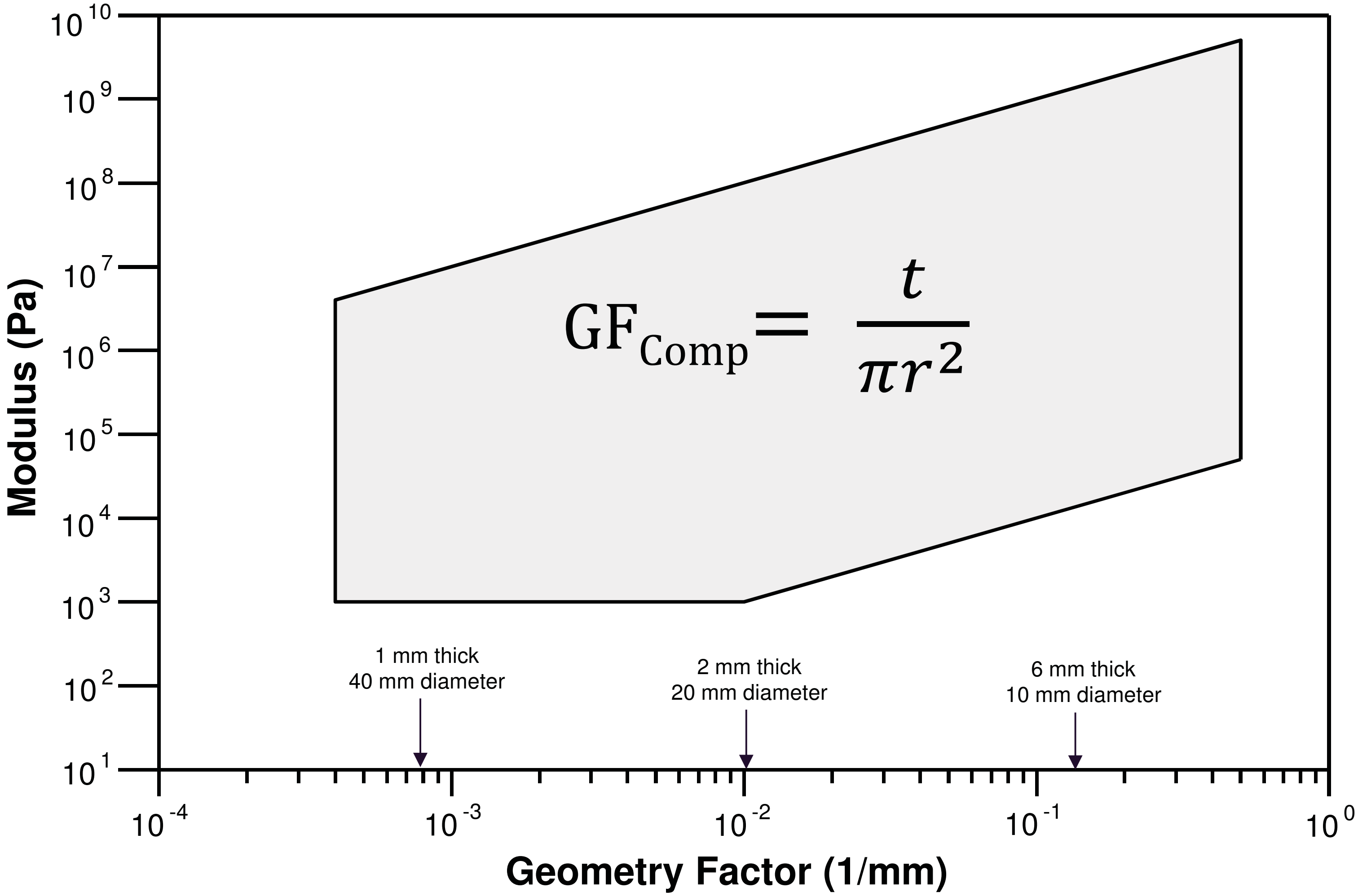
DMA: Compression Clamp



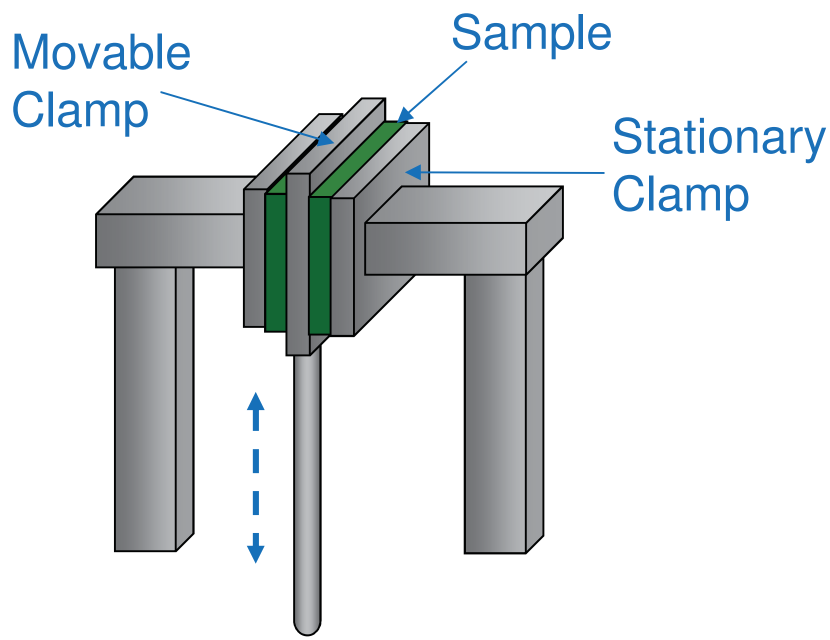
- Good mode for low to medium modulus materials (gels,elastomers)
- Materials must provide restoring force (support necessary static load)
- Options for expansion & penetration measurements

Operating Range of the Compression Clamp

Modulus = Stiffness × Geometry Factor



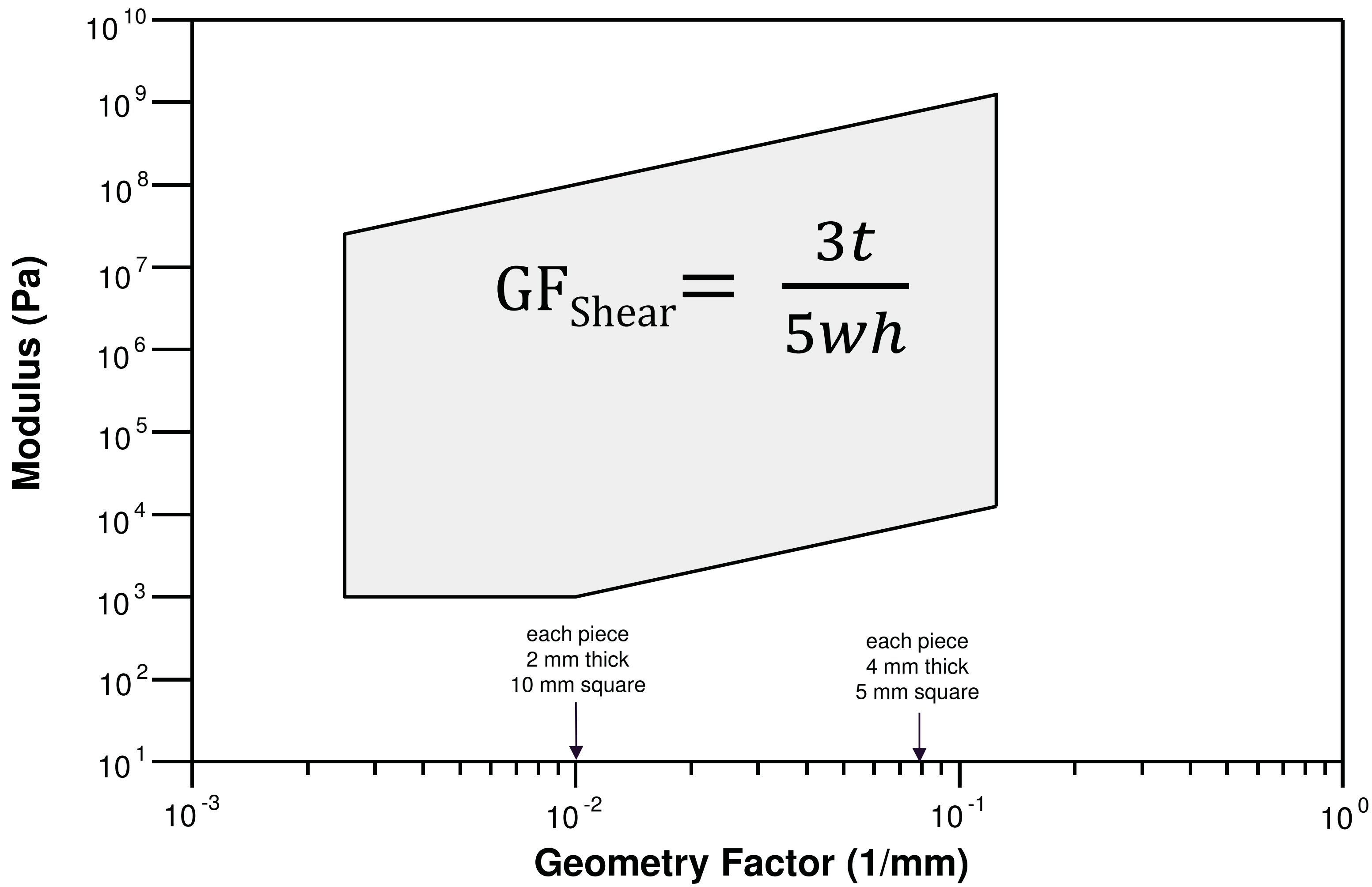
DMA: Shear Sandwich Clamp



- Square sample configuration provides pure shear deformation
- Provides *Shear Moduli: G*, G', G" & G(t)*
- Good for evaluating highly damped soft solids such as gels and adhesives & elastomers > Tg
- Can be used for high viscosity melts and resins

Operating Range of the Shear Sandwich Clamp

Modulus = Stiffness × Geometry Factor



Changing Sample Stiffness

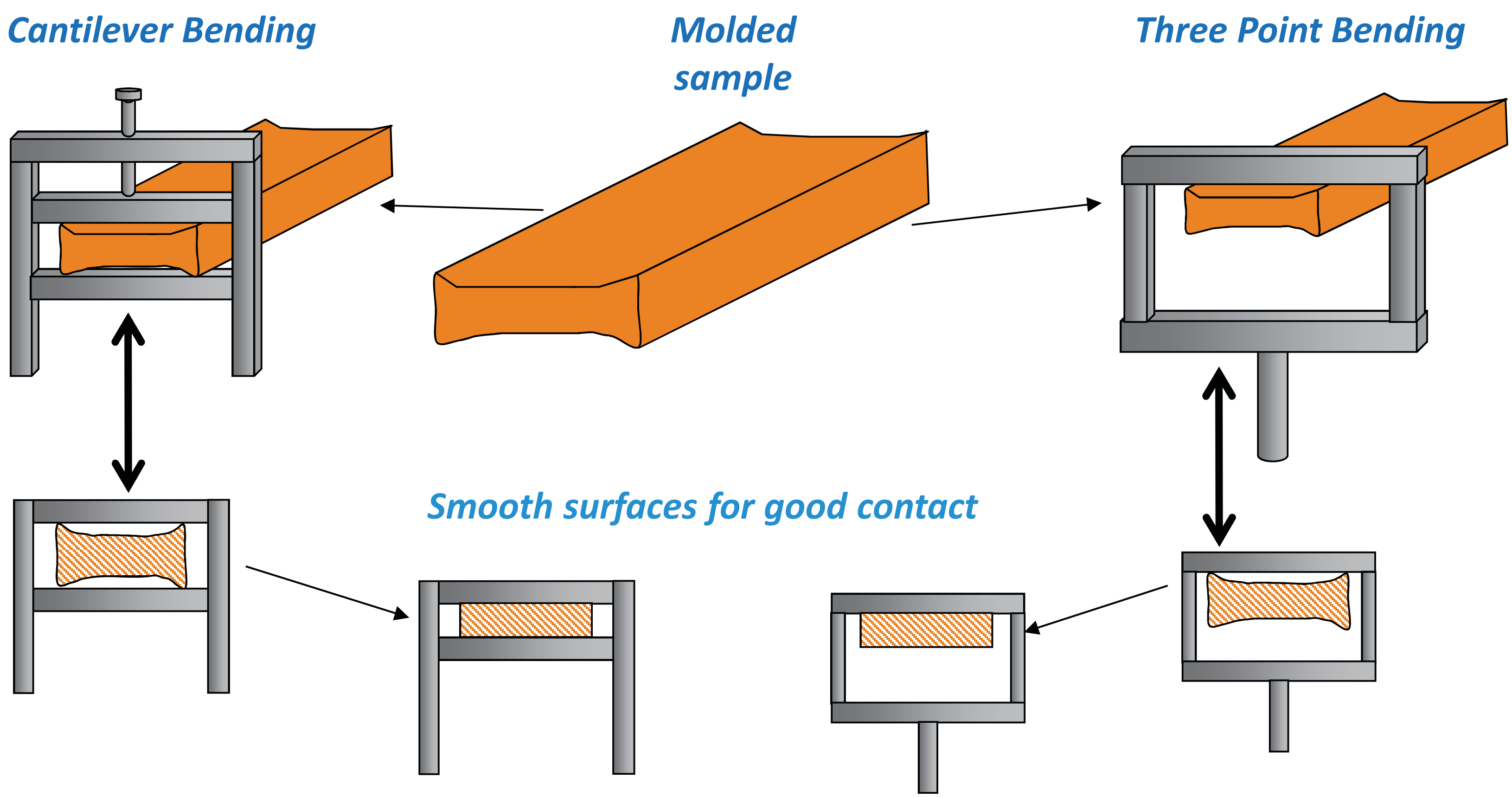
Clamp Type	To Increase Stiffness…	To Decrease Stiffness…
Tension Film	Decrease length or increase width. If possible increase thickness.	Increase length or decrease width. If possible decrease thickness.
Tension Fiber	Decrease length or increase diameter if possible.	Increase length or decrease diameter if possible.
Dual/Single Cantilever	Decrease length or increase width. If possible increase thickness. Note: $L/T \geq 10$	Increase length or decrease width,, If possible decrease thickness. Note: $L/T \geq 10$
Three Point Bending	Decrease length or increase width. If possible increase thickness.	Increase length or decrease width. If possible decrease thickness.
Compression – circular sample	Decrease thickness or Increase diameter.	Increase thickness or decrease diameter.
Shear Sandwich	Decrease thickness or Increase length and width.	Increase thickness or decrease length and width.

DMA Clamping Guide

Sample	Clamp	Sample Dimensions
High modulus metals or composites	3-point Bend Dual Cantilever Single Cantilever	L/T> 10 if possible
Unreinforced thermoplastics or thermosets	Single Cantilever	L/T >10 if possible
Brittle solid (ceramics)	3-point Bend Dual Cantilever	L/T>10 if possible
Elastomers	Dual Cantilever Single Cantilever Shear Sandwich Tension	L/T>20 for T<Tg L/T>10 for T<Tg (only for T> Tg) T<2 mm W<5 mm
Films/Fibers	Tension	L 10-20 mm T<2 mm
Supported Systems	8 mm Dual Cantilever	minimize sample, put foil on clamps

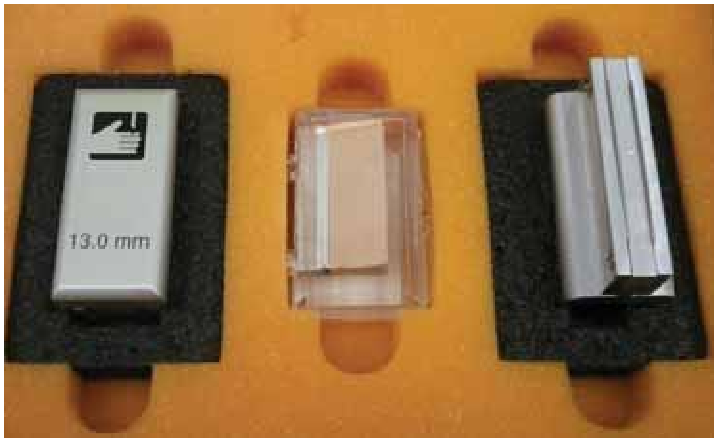
Preparing Samples – The Importance of Shape

Shape: Molded samples are often not flat. May lead to poor contact in Cantilever and Three Point Bend Clamps. Sand samples smooth.



Some Sample Prep Tools

Parallel Razor
Blade Cutter

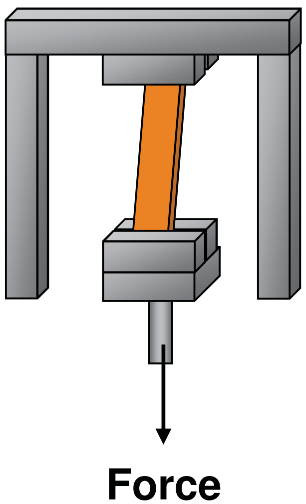


Good for Films and
Sheets of rubber.

Cork Borer

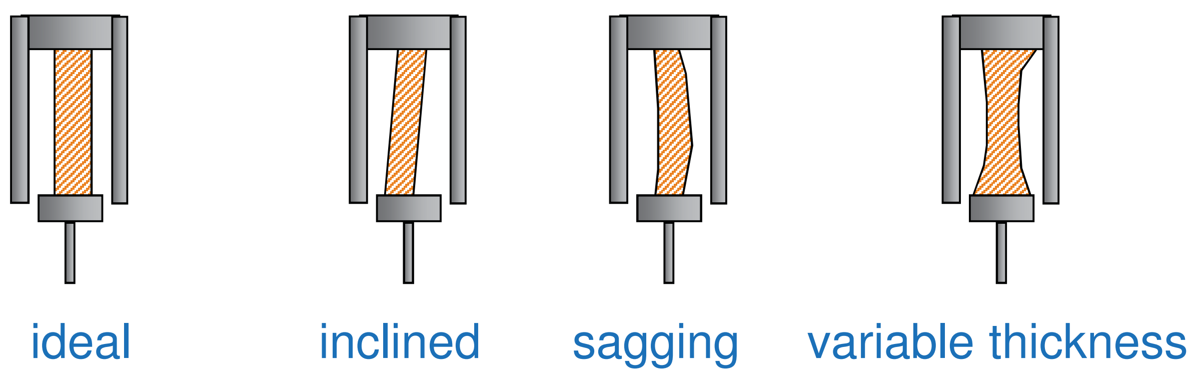
Good for stiff foams and
Sheets of rubber.

Alignment in Tension Mode



If sample buckles during Oscillation. Modulus will be artificially low.

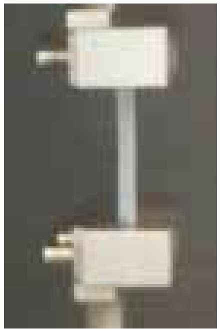
- Buckling during loading causes serious errors as buckled areas do not “feel” the force or deformation
- Buckling can be the result of non-uniform stretching, or crooked loading of a film.
- Observe film from edge while oscillating to verify goodness of load.
- If sample is buckling, reload a new sample.



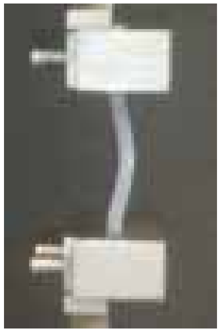
Clamping a Soft Sample in Tension

- Tightening film clamps create stresses in sample and may lead to variation in data.
- Increasing clamping torque may cause buckling and internal stresses.
  - Buckling may be relieved by applying preload force – monitor change in sample length once applied.
  - Internal stresses from the clamp pinch point may not be relieved and depending on sample length can contribute significantly to the modulus measured.

Lightly finger-tightened



+ ¼ turn

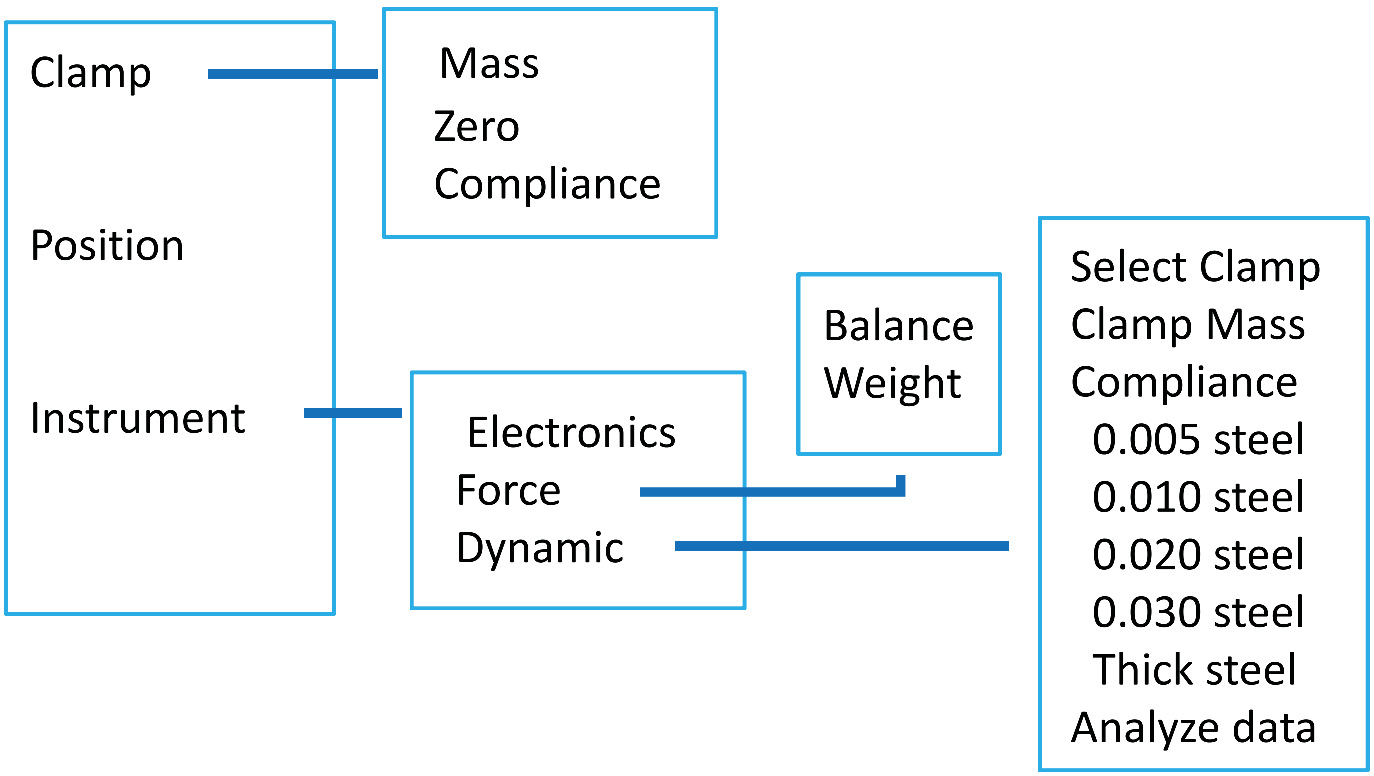


+ ½ turn

+ 1 turn

Instrument Calibrations and Confidence Check

Q800 Flow Chart of Calibration Procedures



Note: Details for calibrations are provided in the Appendix

RSA G2 Instrument Calibration

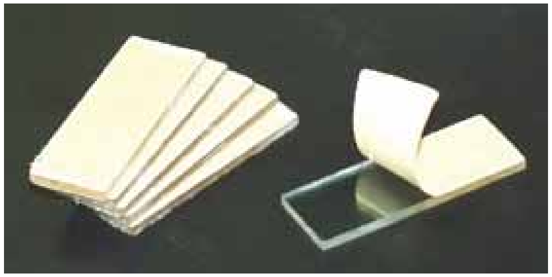
Calibration Tasks and Recommended Intervals

Calibration Task	Calibration Interval
Upper Fixture Mass Calibration	Mandatory: During geometry creation (is a part of geometry configuration)
Force Calibration	Suggested: Monthly. Mandatory: Following transducer replacement.
Phase Angle and Modulus Check	Suggested: Monthly Mandatory: Following actuator or transducer replacement.
Gap Temperature Compensation	Suggested: As required by the experiment.

Note: Details for calibrations are provided in the Appendix

DMA Confidence Check - Polycarbonate

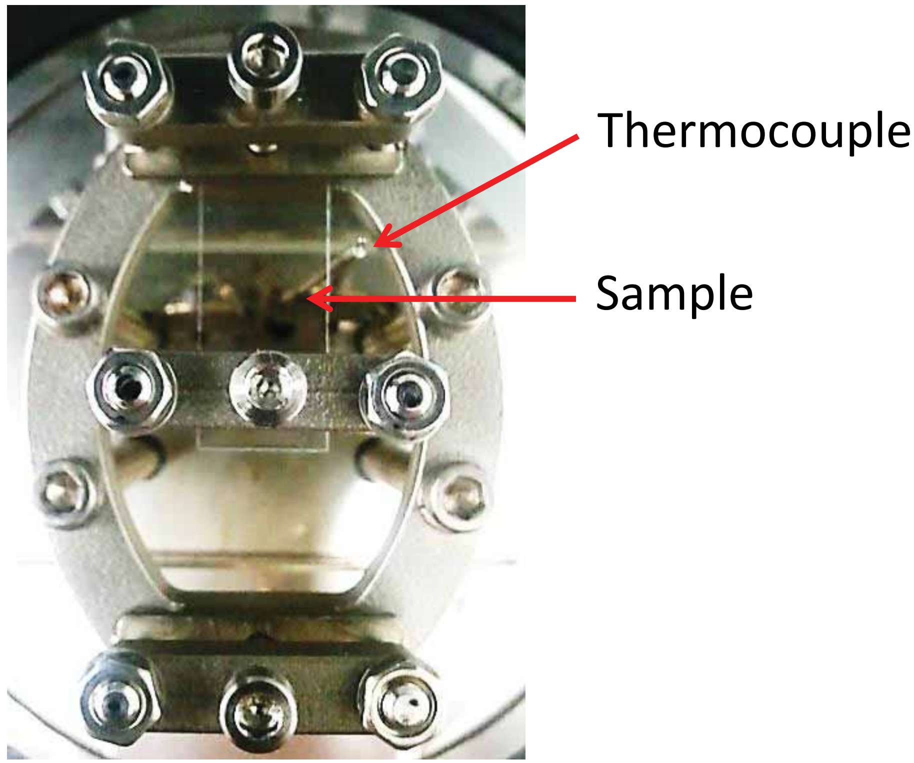
- Load Polycarbonate (L ≈ 17.5, w ≈ 12.85, t ≈ 0.8mm)
- Single Cantilever
  - 20-30 micrometer amplitude
  - 1 Hz frequency
- Storage Modulus at Room Temperature
  E' = 2.35 GPa (2350 MPa) +/- 5%
- Tan Delta at Room Temperature
  Tan δ < 0.01



- Transition Temperature
  Tan δ peak from 155-160℃ @ 1Hz, 3-5℃/min
  E'' peak will be about 5℃ lower

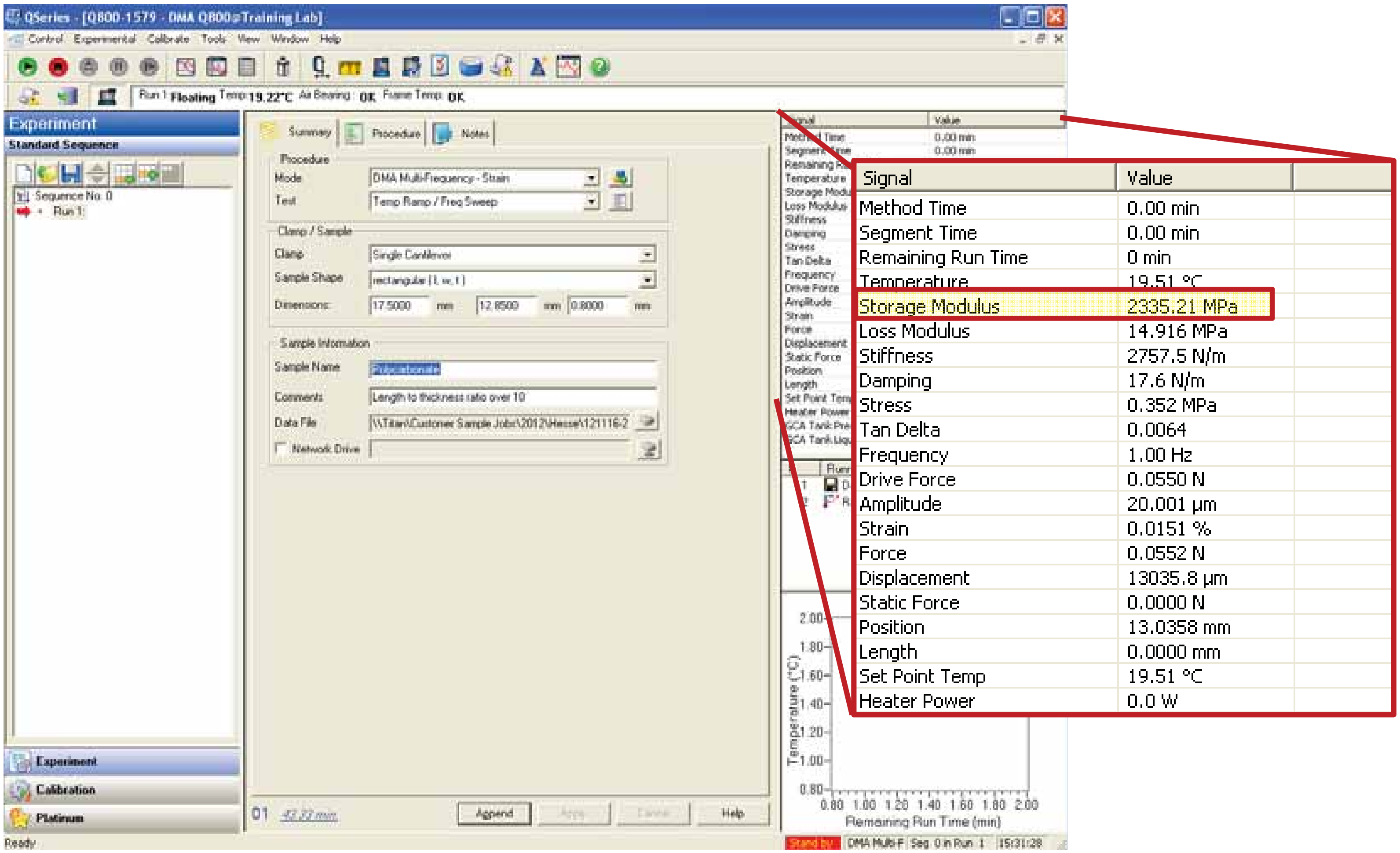
Mounting Sample

- Finger tighten the sample in position and then 'Lock' movable shaft to align clamp before tightening with torque wrench. Tighten to 8-10 in·lbs torque.



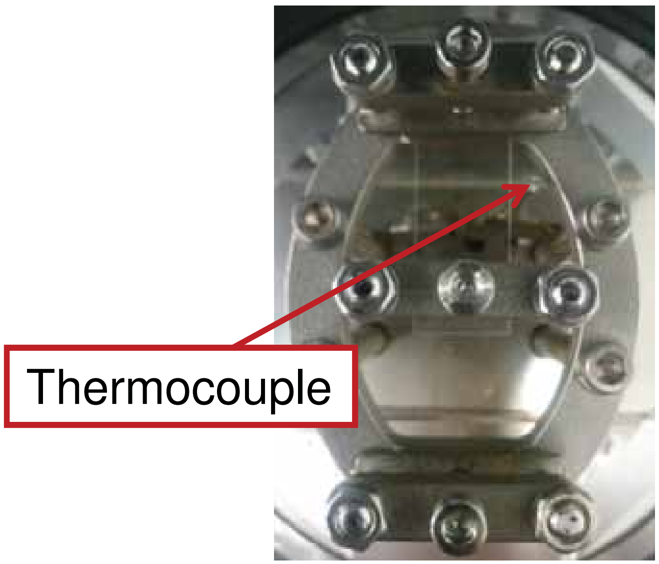
Polycarbonate at Ambient

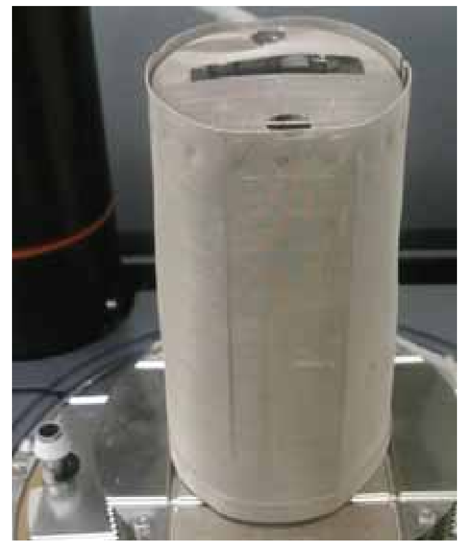
Confidence check: E' = 2350 MPa ±117.5 MPa



Temperature Ramp Results from Polycarbonate

- Transition Temperature:
  - Tan d peak from 155-160 °C @ 1Hz, 3-5 °C/min
  - E" peak will be about 5 °C lower

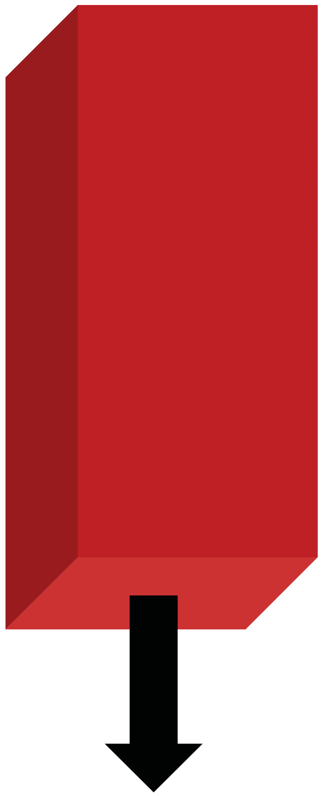




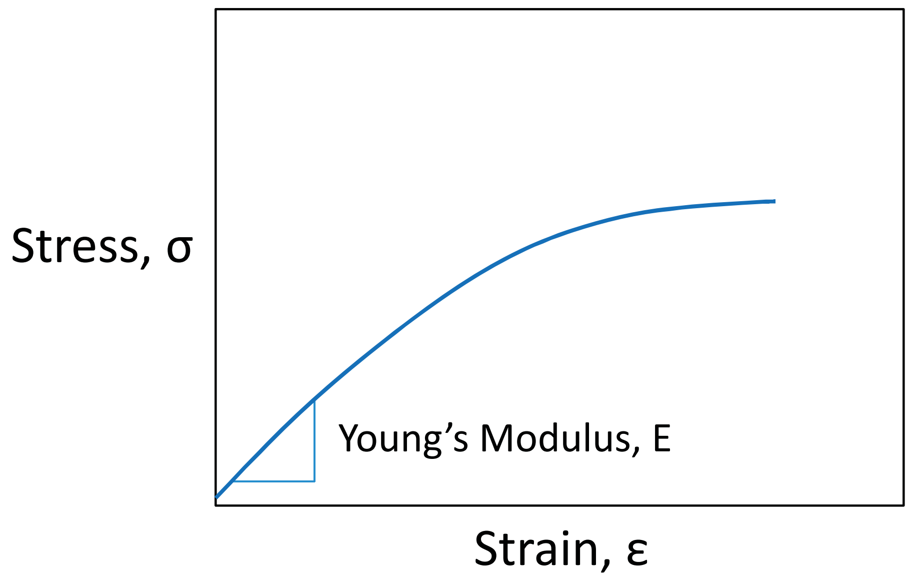
Note: Thermocouple position and sample or thermocouple shields can effect temperature results from a temperature ramp.

Linear Viscoelastic Theory

Mechanical Analysis- Tensile Modulus

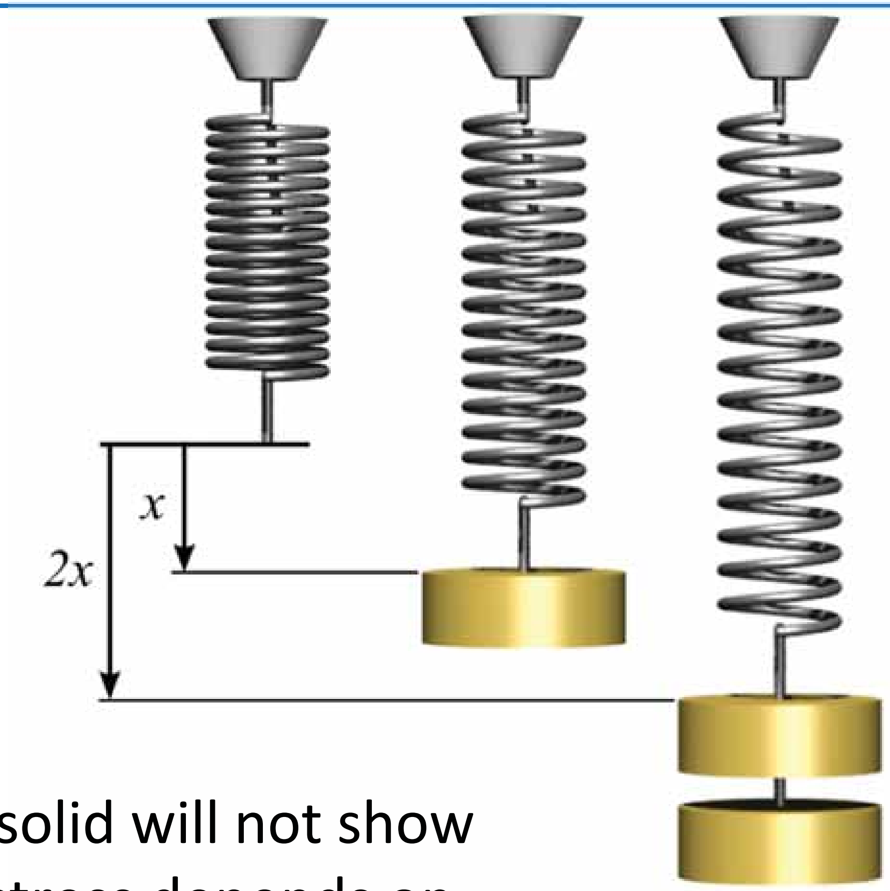


- In classical mechanics, stress is applied to a material to reach a strain, and a modulus is determined



Hooke's Law of Elasticity

- For an Elastic Solid, Stress and Strain have a constant proportionality $\sigma = E^{*}\varepsilon$
- If the material follows Hooke's Law, the deformation will be reversible when the stress is removed



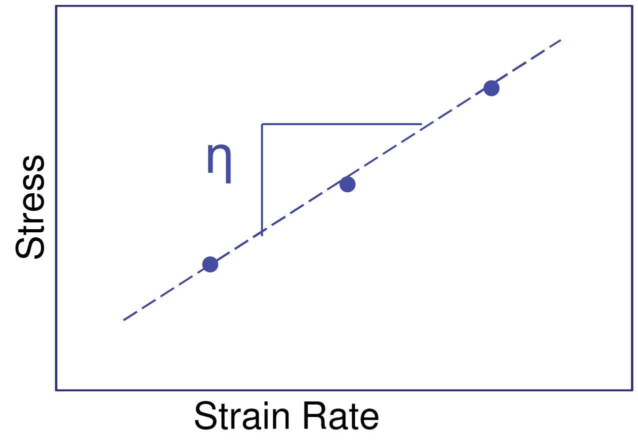
- The modulus of a Hookean solid will not show any time dependence- the stress depends on the strain, but not the *strain rate*

Newton's Law of Viscosity

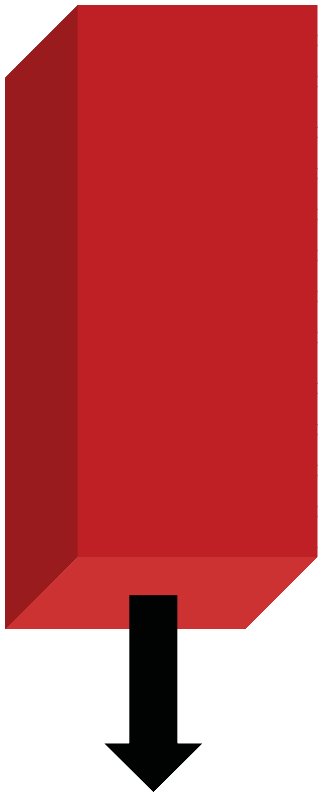
- For a Viscous Liquid, Stress is proportional to Strain Rate $d\varepsilon/dt$ by a coefficient of Viscosity η

$$\sigma = \eta^* \, d\varepsilon/dt$$

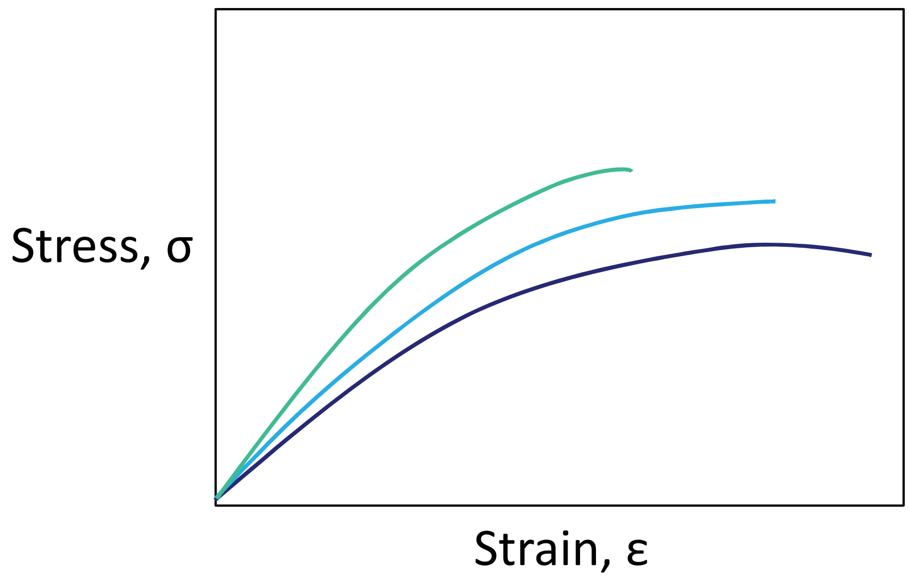
- The deformation of a liquid is non-reversible



Mechanical Analysis- Tensile Modulus



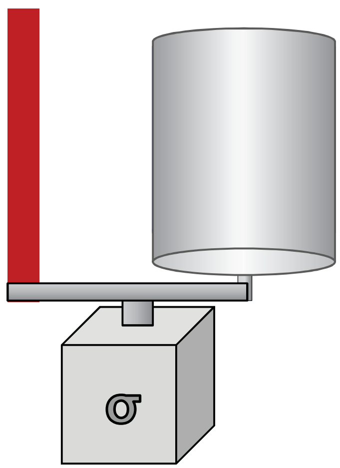
- In tensile testing of viscoelastic materials, the rate of extension will give different results
  - the stress depends on both the *strain*, and the *strain rate*
- $\sigma = E^{*}\varepsilon + (?)^{*}d\varepsilon/dt$



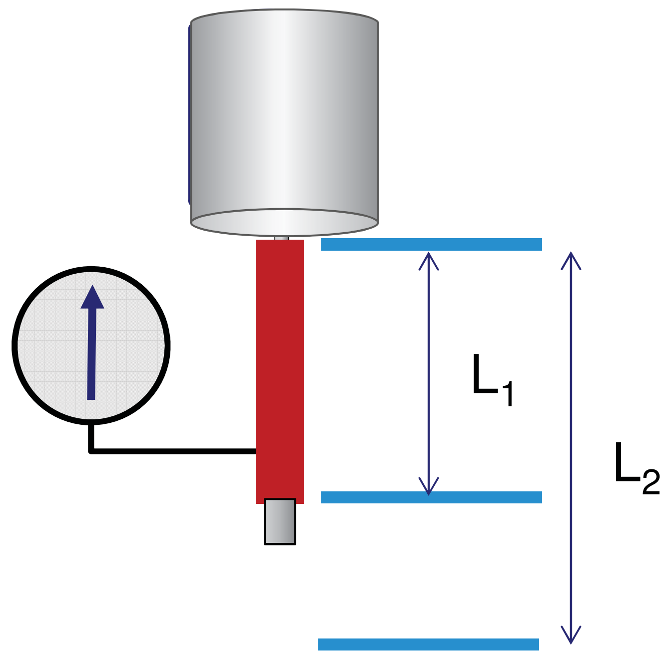
Viscoelastic Behavior

$$\sigma = E*\varepsilon + \eta*d\varepsilon/dt$$

Kelvin-Voigt Model (Creep)



Maxwell Model (Stress Relaxation)



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

Viscoelasticity Defined

Range of Material Behavior

Liquid Like ---------- Solid Like

Ideal Fluid ----- Most Materials ----- Ideal Solid

Purely Viscous ----- *Visco*elastic ----- Purely Elastic

Viscoelasticity: Having both viscous and elastic properties

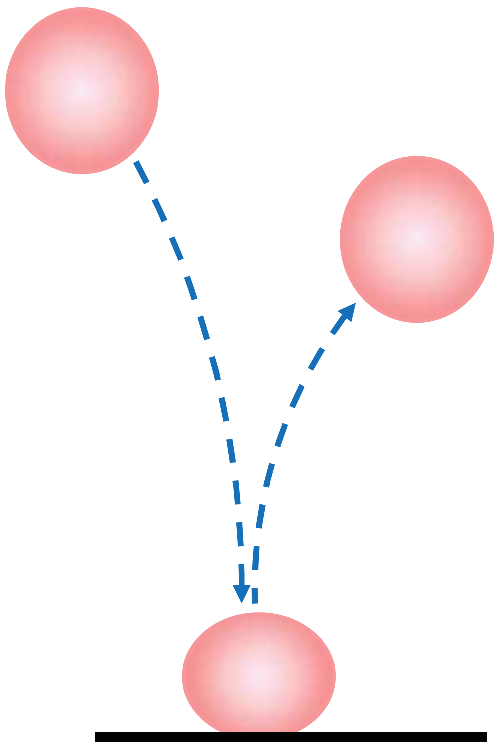
Pitch Drop Experiment

- Long deformation time: pitch behaves like a highly viscous liquid
  - 9th drop fell July 2013
- Short deformation time: pitch behaves like a solid

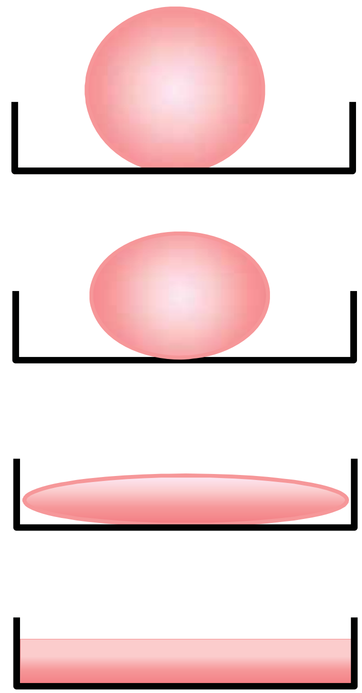
Started in 1927 by Thomas Parnell in Queensland, Australia

http://www.theatlantic.com/technology/archive/2013/07/the-3-most-exciting-words-in-science-right-now-the-pitch-dropped/277919/

Time-Dependent Viscoelastic Behavior



T is short [< 1s]



T is long [24 hours]

Deborah Number [De] = τ / T

Viscoelasticity, Deborah Number

- Old Testament Prophetess who said (Judges 5:5):
  "The Mountains ‘Flowed’ before the Lord"

- Everything Flows if you wait long enough!

- Deborah Number, De - The ratio of a characteristic relaxation time of a material (τ) to a characteristic time of the relevant deformation process (T).

$$De = \tau/T$$

Deborah Number

- Hookean elastic solid - τ is infinite
- Newtonian Viscous Liquid - τ is zero
- Polymer melts processing - τ may be a few seconds

High De	→	Solid-like behavior
Low De	→	Liquid-like behavior

IMPLICATION: Material can appear solid-like because
1) it has a very long characteristic relaxation time or
2) the relevant deformation process is very fast

Linear Viscoelasticity Region (LVR) Defined

"If the deformation is small, or applied sufficiently slowly, the molecular arrangements are never far from equilibrium.

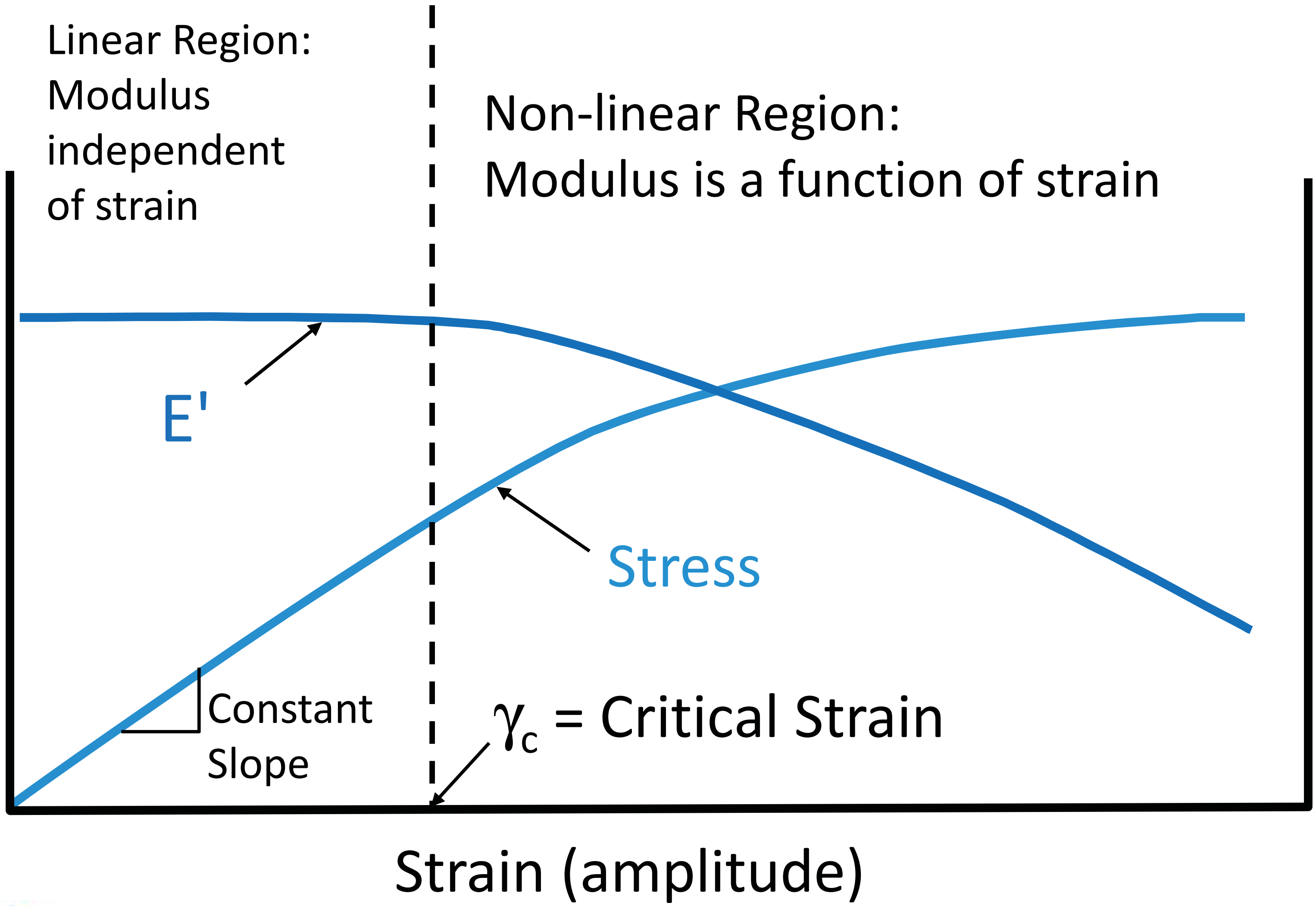
The mechanical response is then just a reflection of dynamic processes at the molecular level which go on constantly, even for a system at equilibrium.

This is the domain of LINEAR VISCOELASTICITY.

The magnitudes of stress and strain are related linearly, and the behavior for any liquid is completely described by a single function of time."

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

Linear Viscoelastic Region (LVR)



Importance of LVR

Linear Viscoelastic Properties

E' (or G'), E" (or G"), tan δ, η*

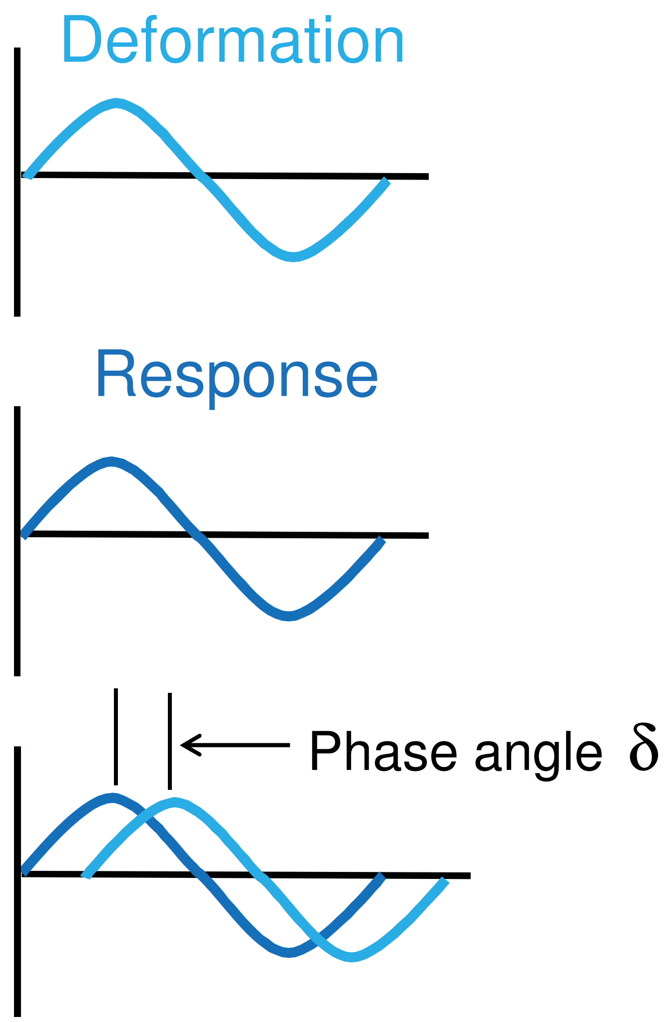
Measuring linear viscoelastic properties helps us bridge the gap between molecular structure and product performance

Available DMA Test Modes

Dynamic (Oscillatory) Tests

Dynamic Mechanical Testing

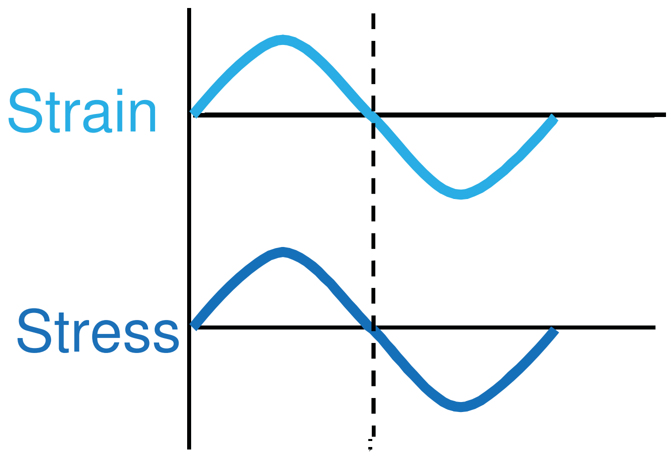
- An oscillatory (sinusoidal) deformation (stress or strain) is applied to a sample.
- The material response (strain or stress) is measured.
- The phase angle δ, or phase shift, between the deformation and response is measured.



Dynamic Testing: Response for Classical Extremes

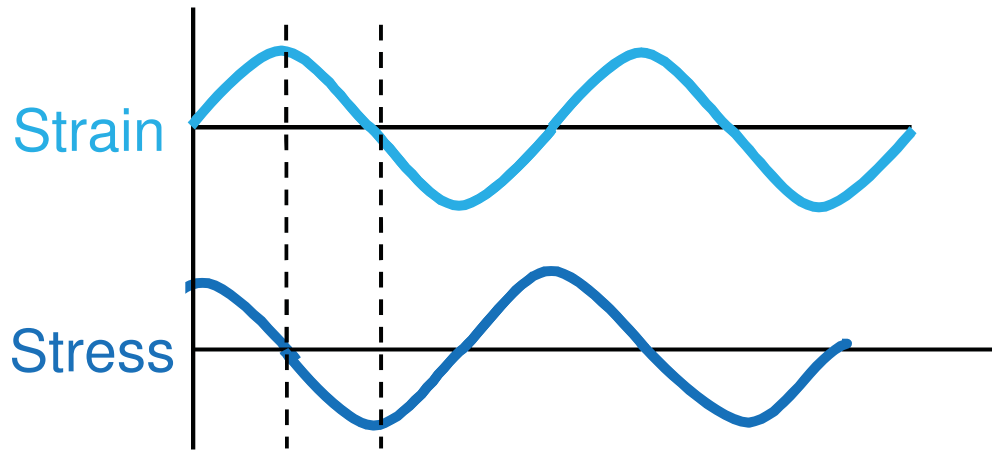
Purely Elastic Response
(Hookean Solid)

$\delta = 0°$

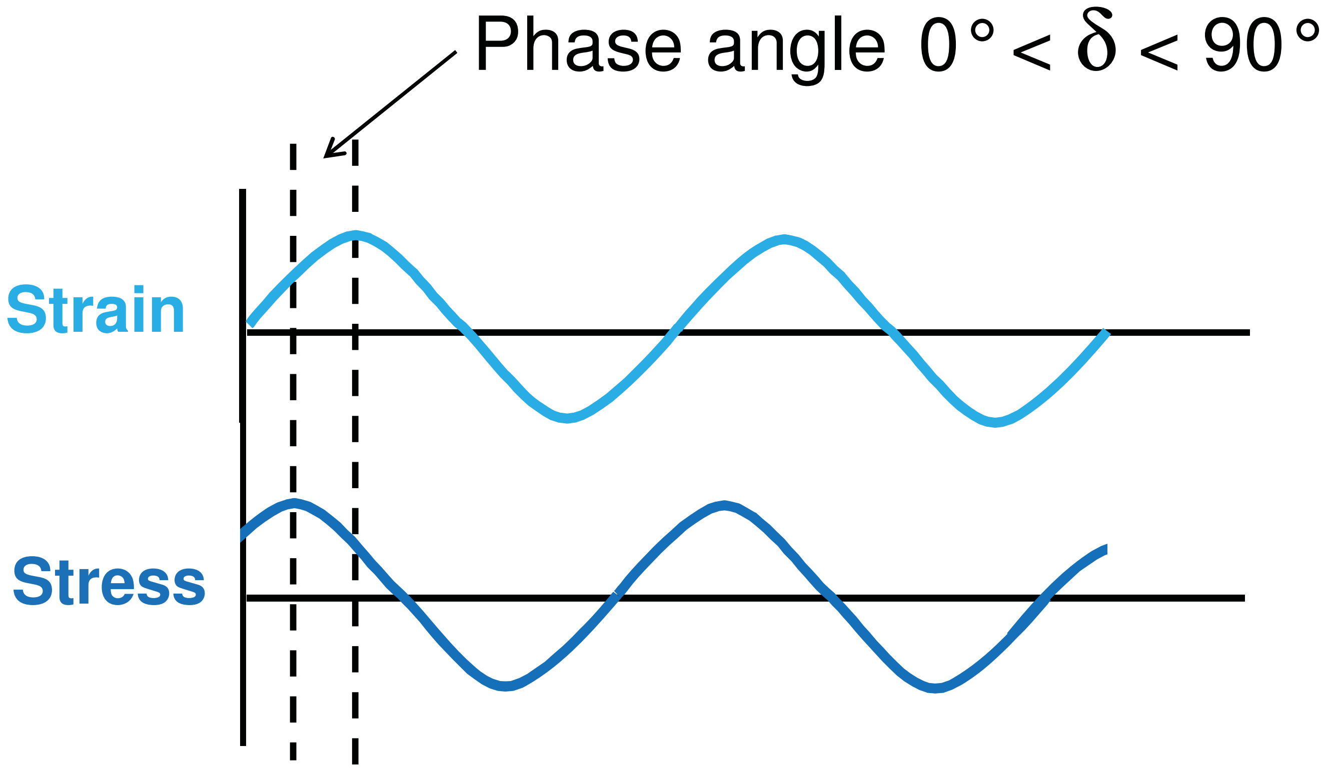


Purely Viscous Response
(Newtonian Liquid)

$\delta = 90°$



Dynamic Testing: Viscoelastic Material Response



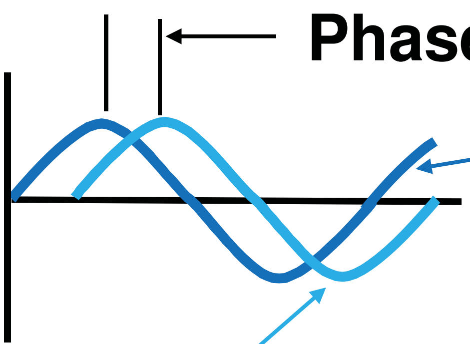
Viscoelastic Parameters: Complex, Elastic, & Viscous Stress

- The stress in a dynamic experiment is referred to as the complex stress σ^*
- The complex stress can be separated into two components:
  1) An elastic stress in phase with the strain. $\sigma' = \sigma^* \cos\delta$

     σ' is the degree to which material behaves like an elastic solid.
  2) A viscous stress in phase with the strain rate. $\sigma'' = \sigma^* \sin\delta$

     σ'' is the degree to which material behaves like an ideal liquid.

Phase angle δ



Complex Stress, σ* ➡ $\sigma^* = \sigma' + i\sigma''$

Strain, ε

Complex number: $|x + iy| = \sqrt{x^2 + y^2}$

The material functions can be described in terms of complex variables having both real and imaginary parts. Thus, using the relationship:

$$\text{Cos}\, x + j\text{Sin}\, x = e^{jx}$$

$$\text{where } j = \sqrt{-1}$$

DMA Viscoelastic Parameters

<u>The Modulus:</u> Measure of materials overall resistance to deformation.

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

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

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

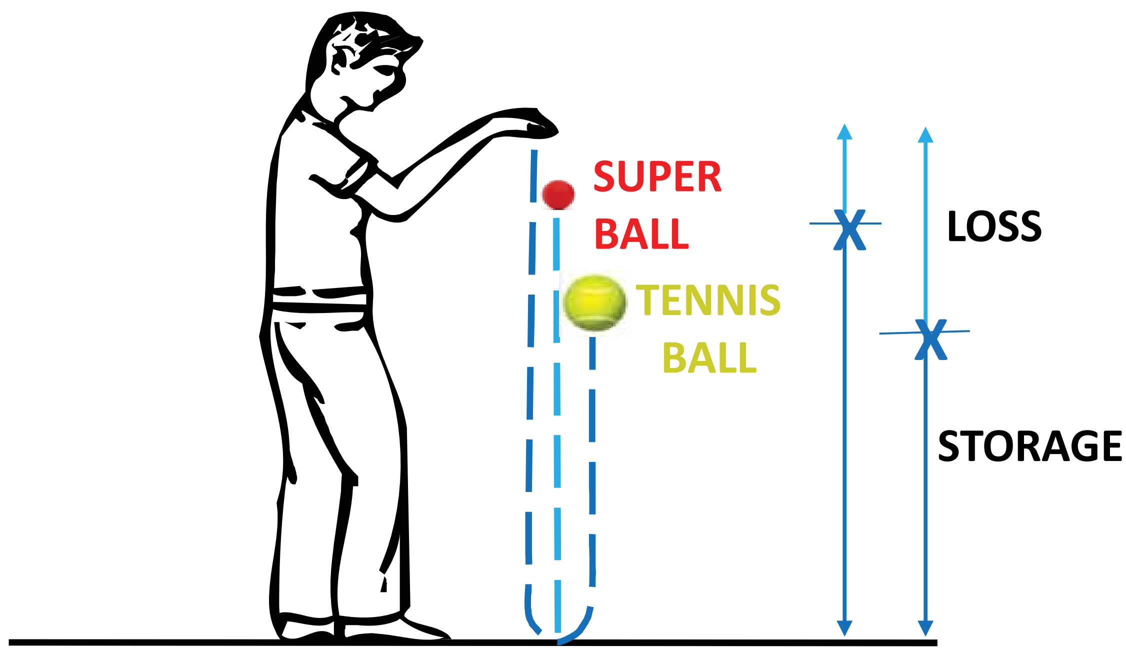
<u>The Viscous (loss) Modulus:</u>
The ability of the material to dissipate energy. Energy lost as heat.

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

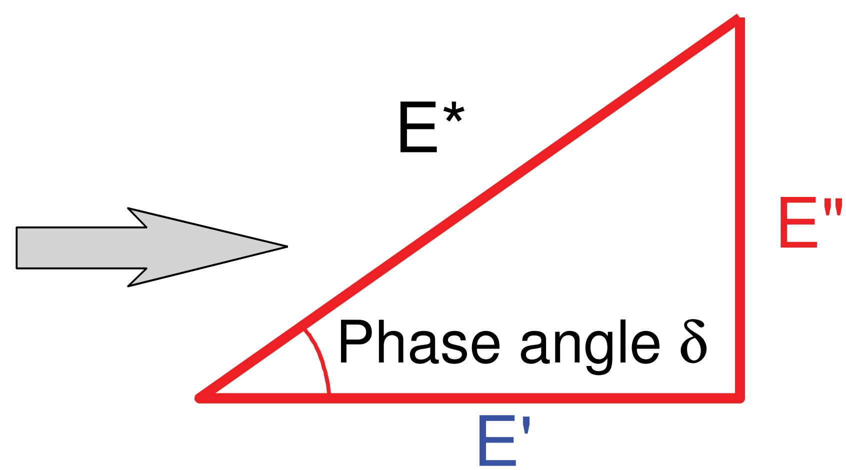
<u>Tan Delta:</u>
Measure of material damping - such as vibration or sound damping.

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

Storage and Loss of a Viscoelastic Material



Dynamic measurement represented as a vector

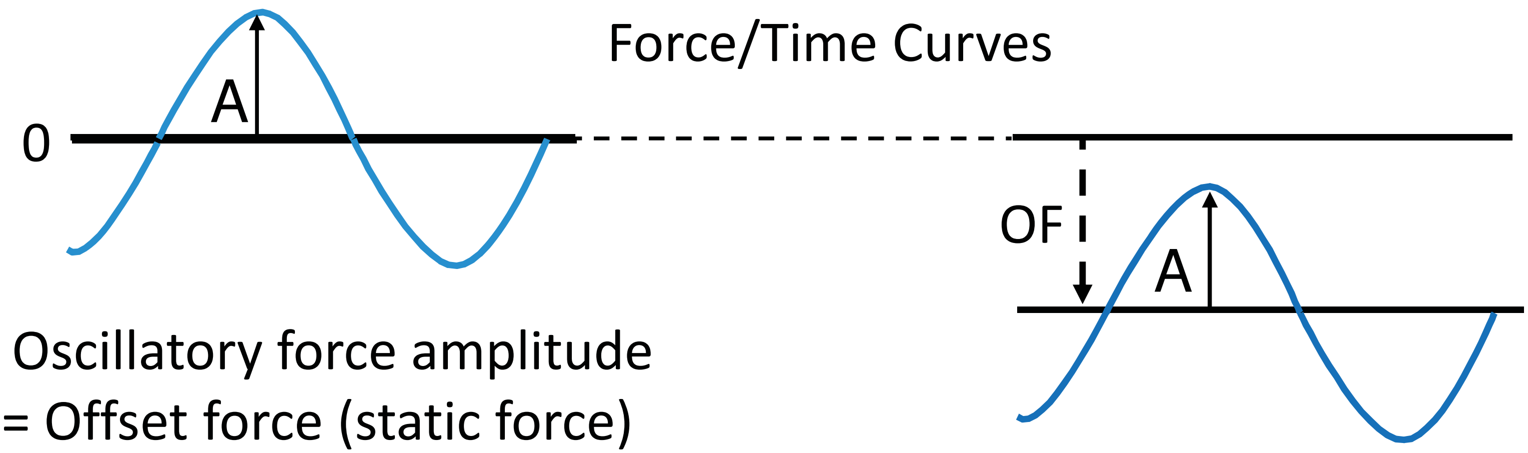


Dynamic (Oscillatory) Testing

Available oscillatory test modes

- Strain (stress) Sweep
- Time Sweep
- Frequency Sweep
- Temperature ramp
- Temperature Step (Sweep) (TTS)
- Others

Some Clamps Require Offset (static) Force!



A = Oscillatory force amplitude
OF = Offset force (static force)

Clamps without offset force:
- Single Cantilever
- Dual Cantilever
- Shear Sandwich

Clamps with offset force:
- Tension Film
- Tension: Fiber
- 3-Point Bend
- Compression
- Penetration

Offset (Static) Force

Static force [General]

- Used in tensioning clamps to prevent sample buckling [tension], or loss of contact with the probe [compression] during the test. Static force must exceed dynamic force at all times during experiment
- Applied before dynamic oscillation to automatically measure sample length

Constant Force

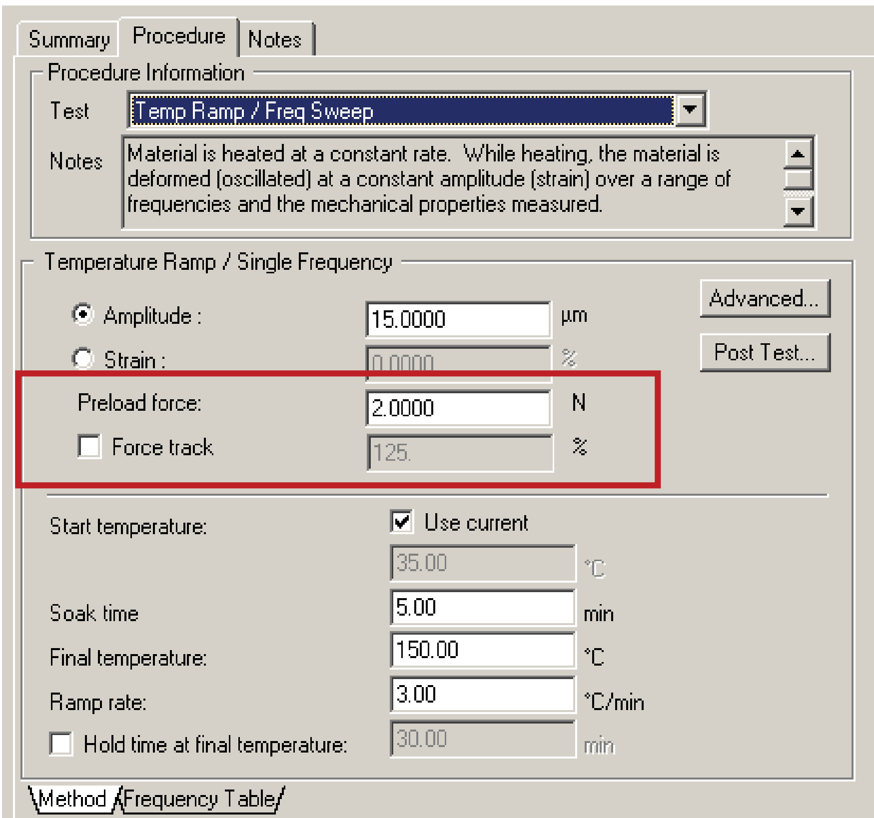
- Applies same static force throughout experiment
- Can be used with highly crystalline or cross-linked materials to measure displacement at constant force for quant. expansion

Force Track

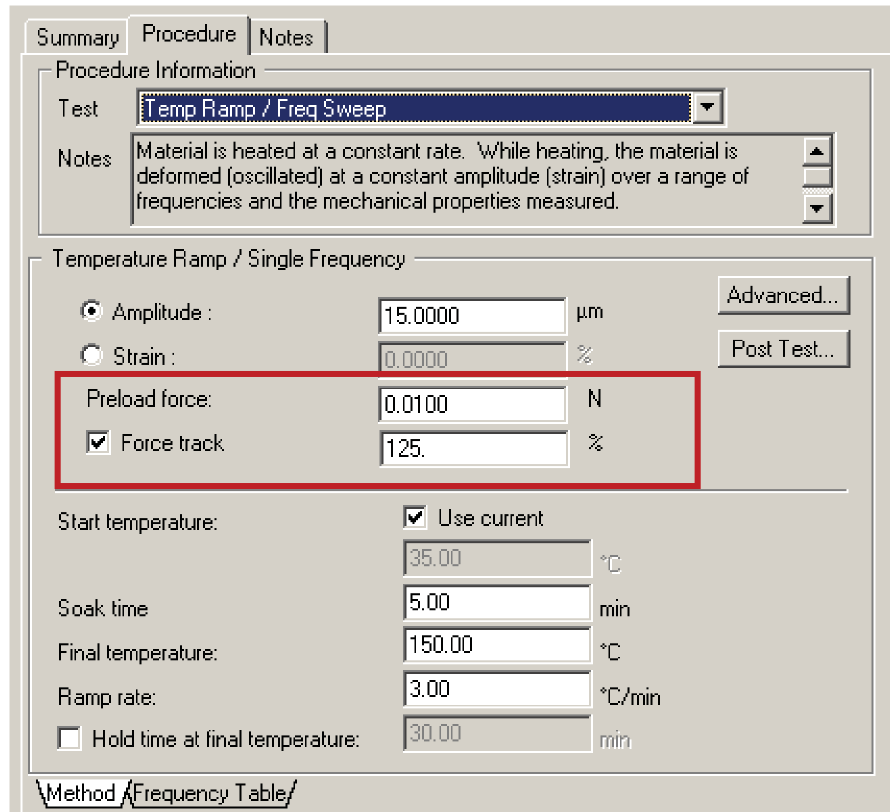
- Applies Static Force in Proportion to Sample Modulus. Used in "Tensioning" Clamps to reduce stretching as specimen weakens
- Ratio of static to dynamic force:
  - **Static Force = (Force Track %/100) x (Stiffness x Amplitude)**
  - where stiffness = Force applied to sample/amplitude of deformation
- Values from 125-150% work well for most samples

Offset Force on Q800

- Constant static force

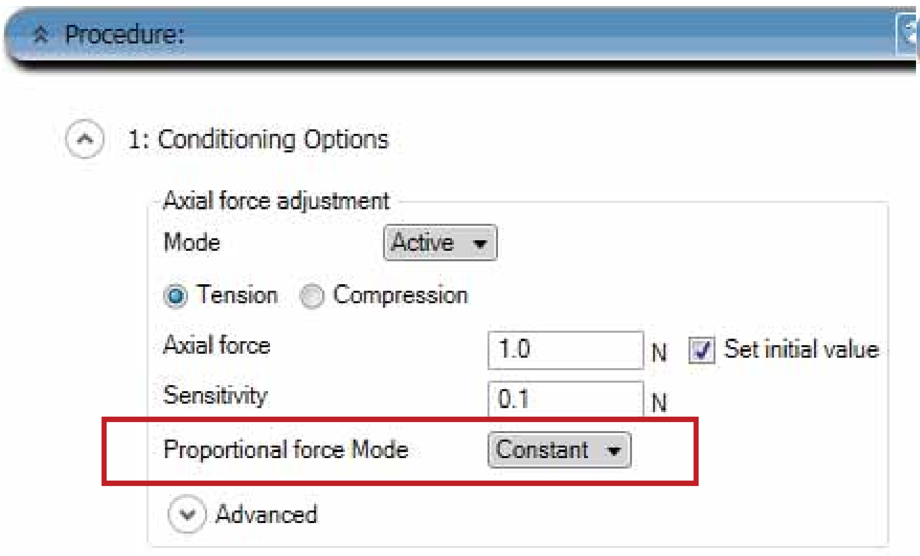


- Force track

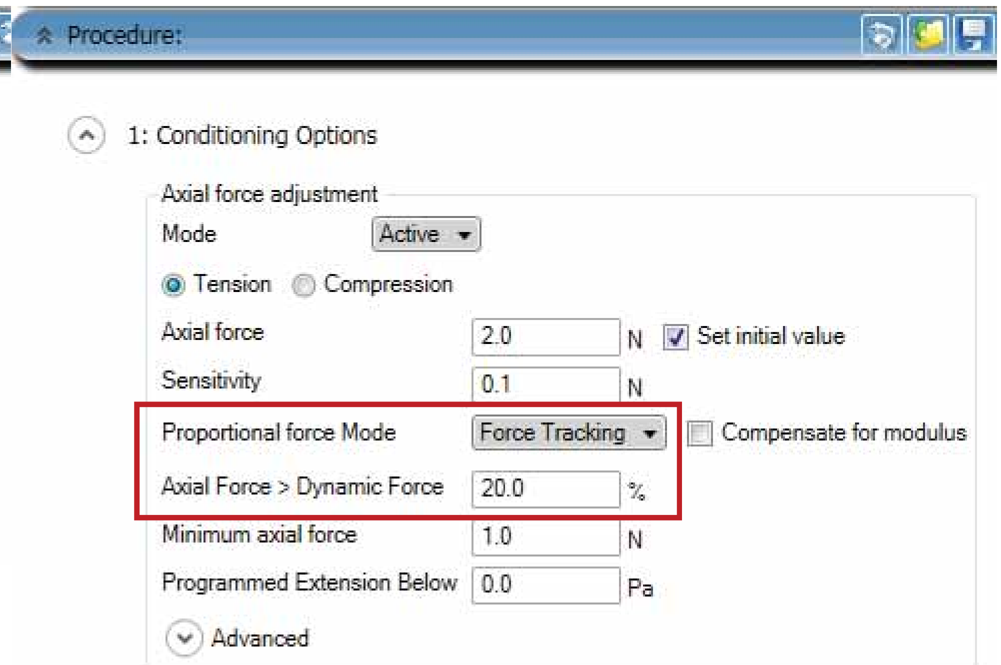


Offset Force on RSA G2

- Constant static force

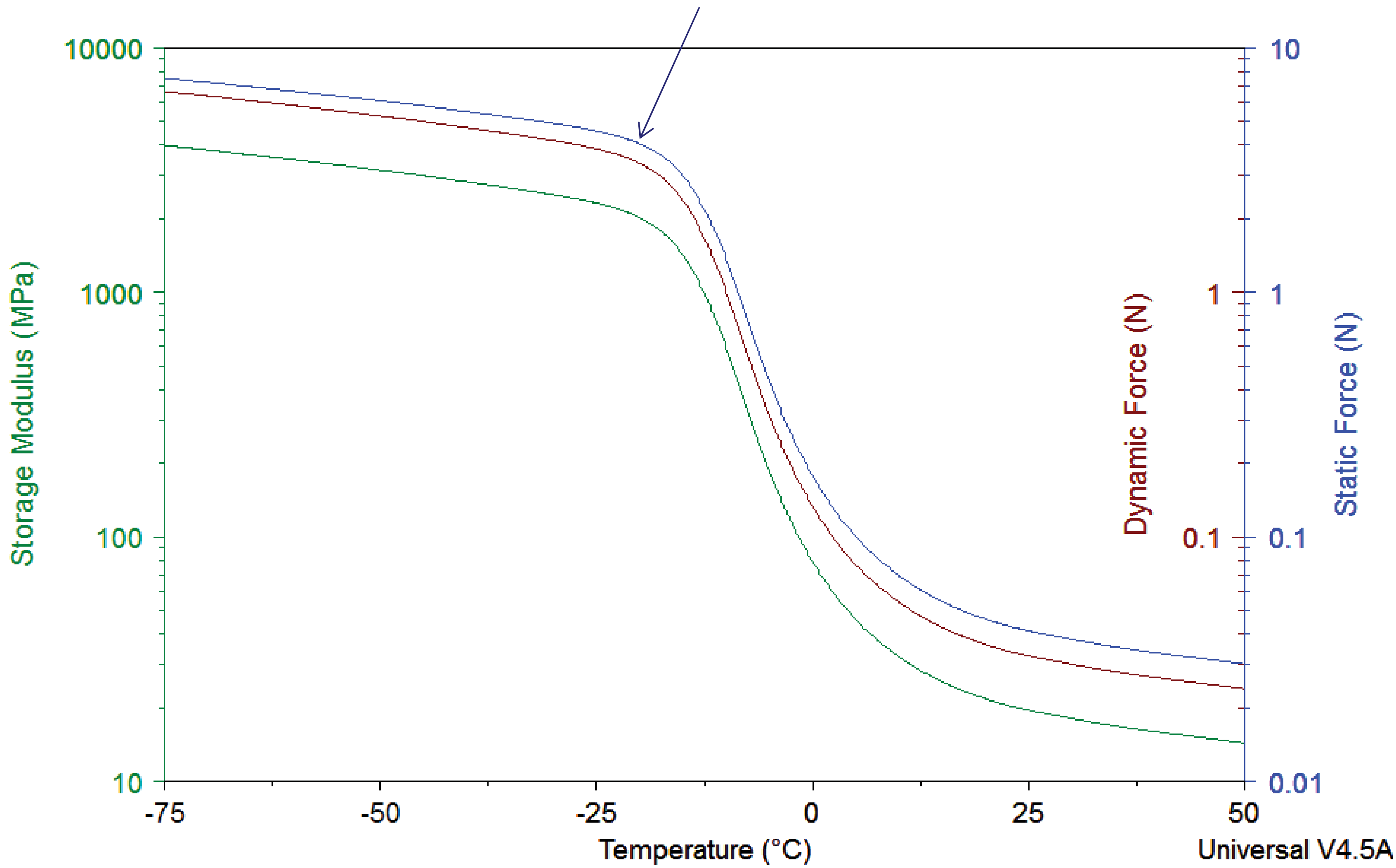


- Force track



Q800 Temperature Ramp with Force Track

- Static Force tracks Dynamic Force throughout Temperature Ramp



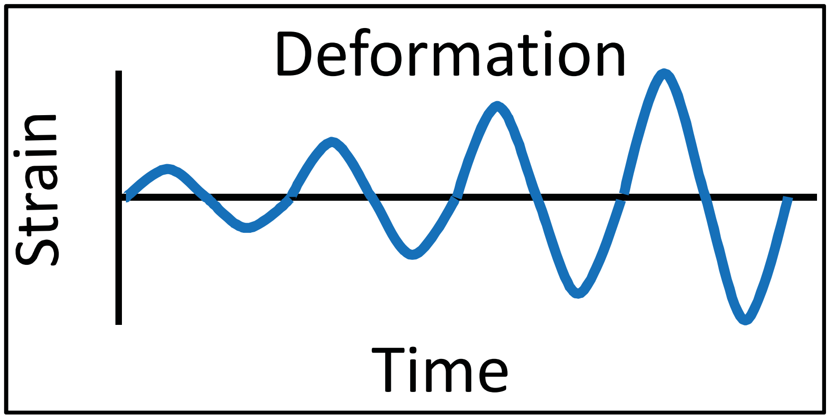
Choosing Force Track Parameters

Initial static force before testing

Clamp Type	Static Force	Force Track
Tension Film	0.01 N	120 to 150%
Tension Fiber	0.001 N	120%
Compression	0.001 to 0.01 N	125%
Three Point Bending Thermoplastic Sample	1 N	125 to 150%
Three Point Bending Stiff Thermoset Sample	1 N	150 to 200% Can use constant static force

Note: Constant (or static) force can be used as long as static force > dynamic force through out the entire experiment.

Dynamic Strain (Stress) Sweep

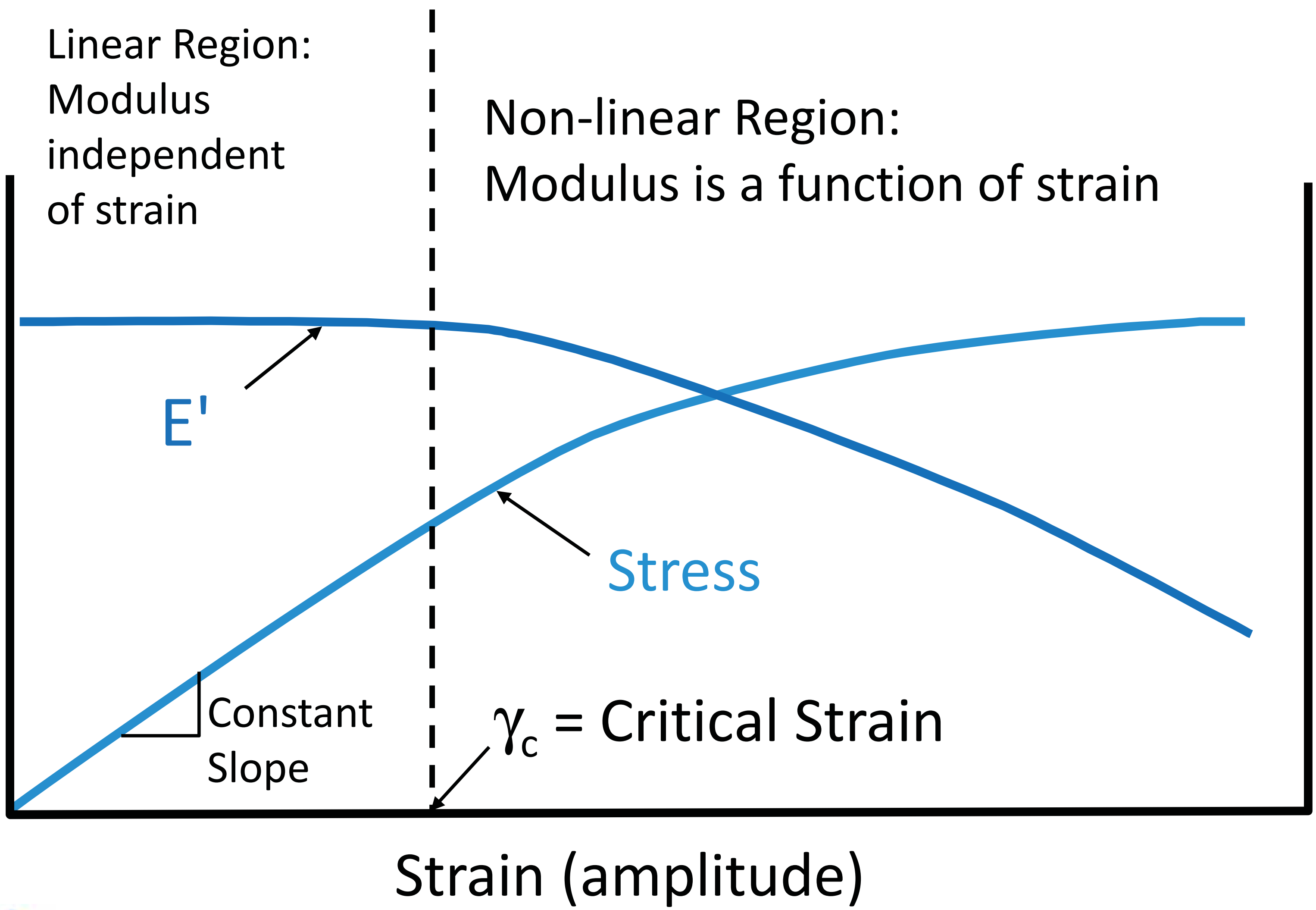


- The material response to increasing deformation amplitude is monitored at a constant frequency and temperature.

<u>USES</u>

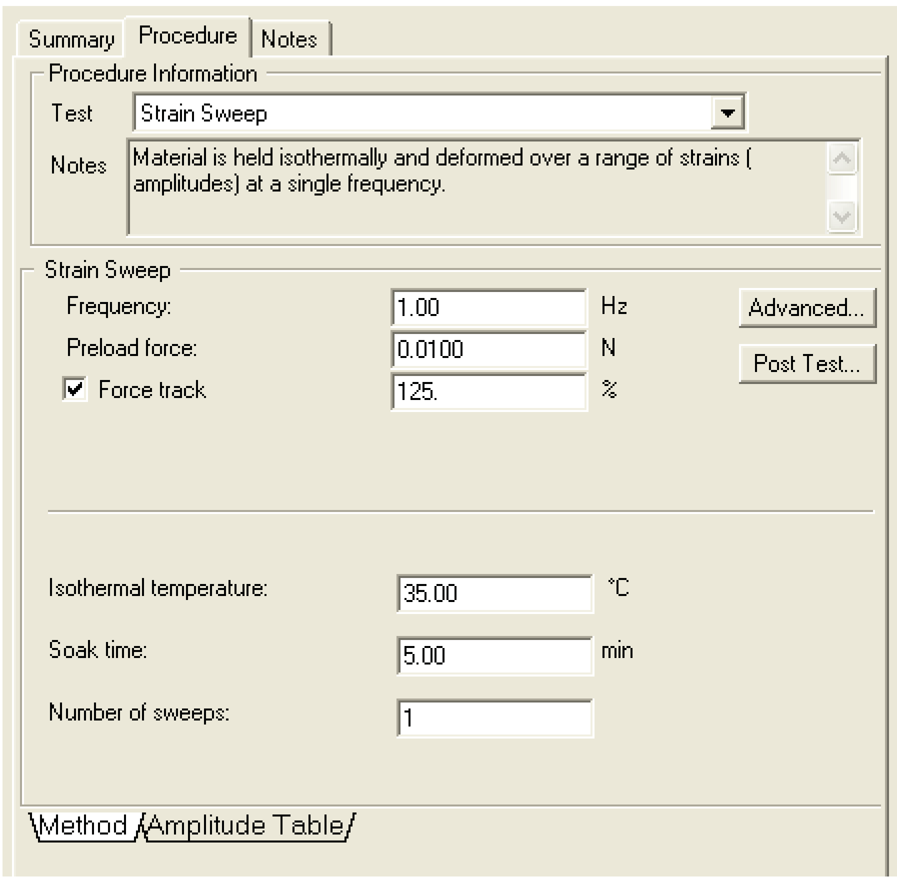
- Identify Linear Viscoelastic Region
- Resilience

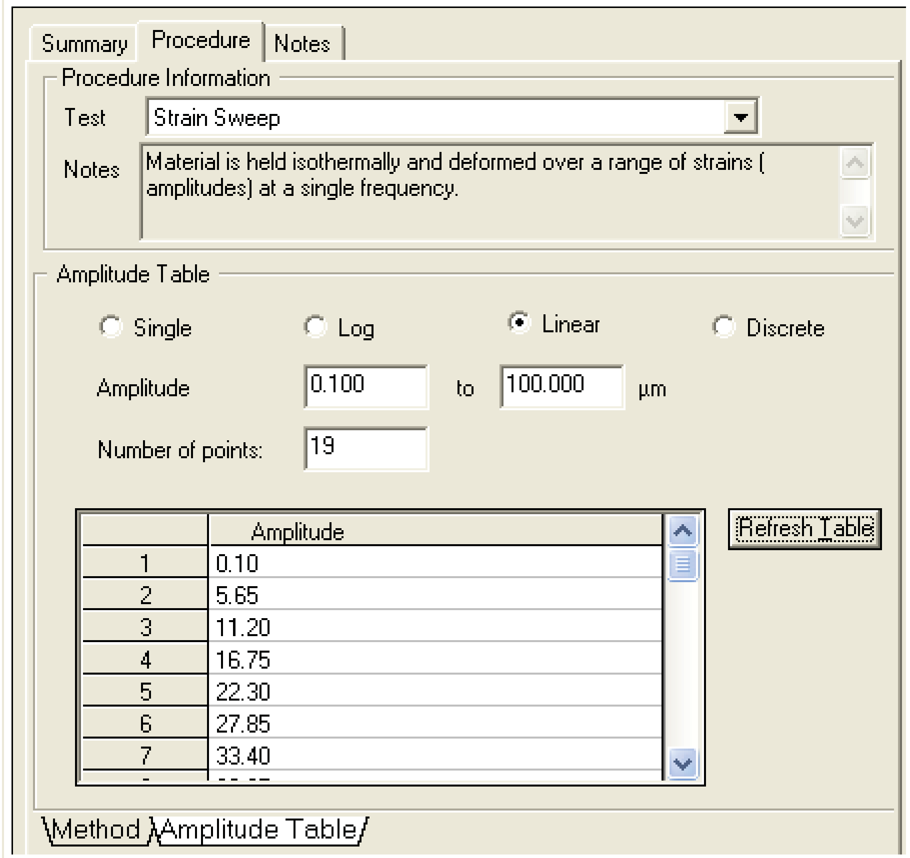
Dynamic Strain Sweep: Material Response



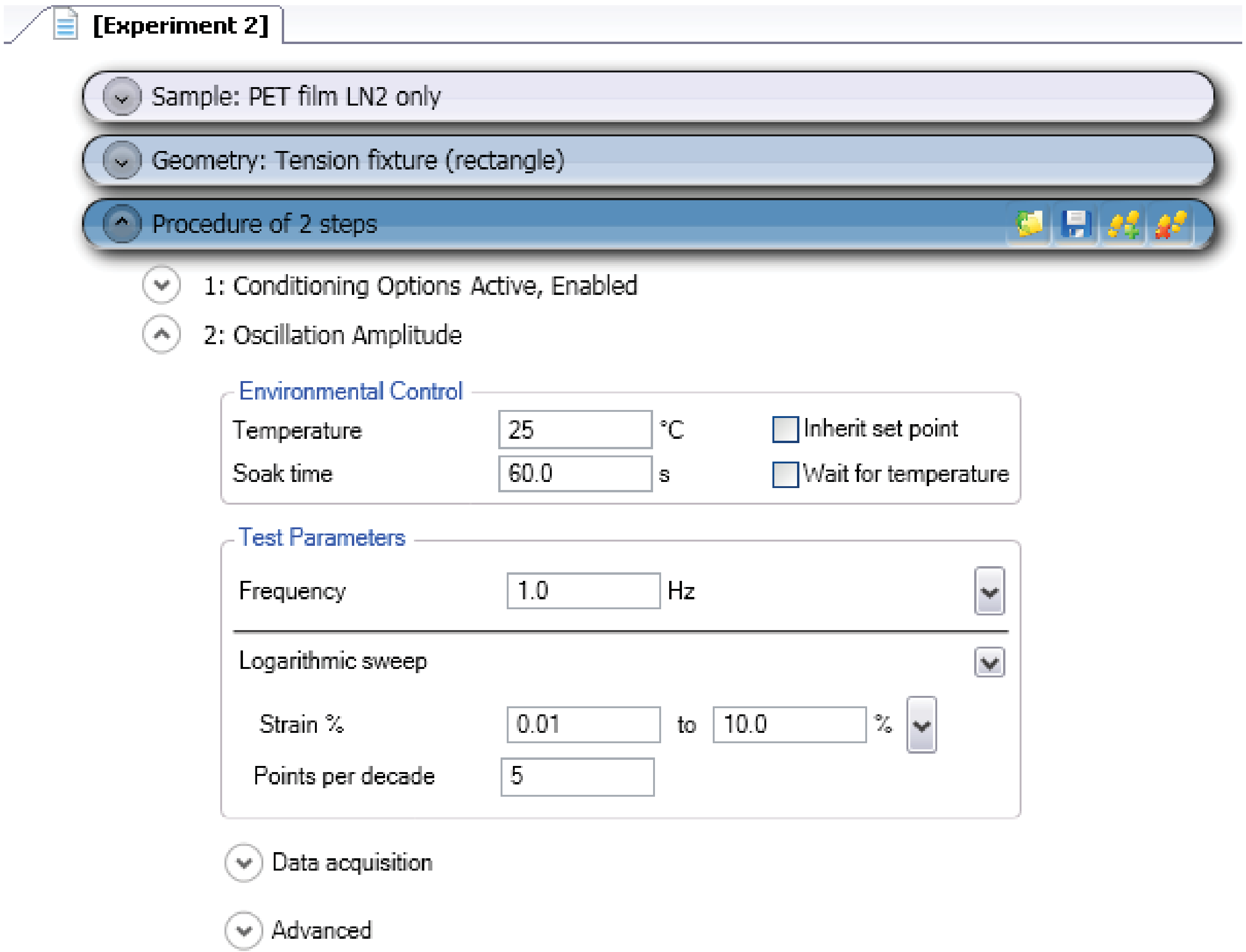
Programming Strain Sweep on Q800

Mode: DMA Multi-Strain

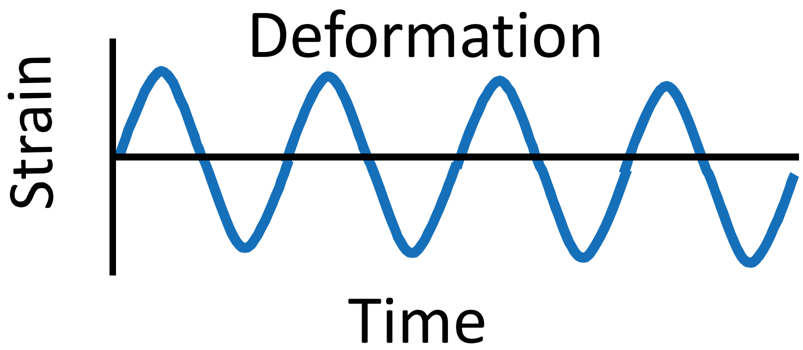




Programming Strain Sweep on RSA G2



Dynamic Time Sweep



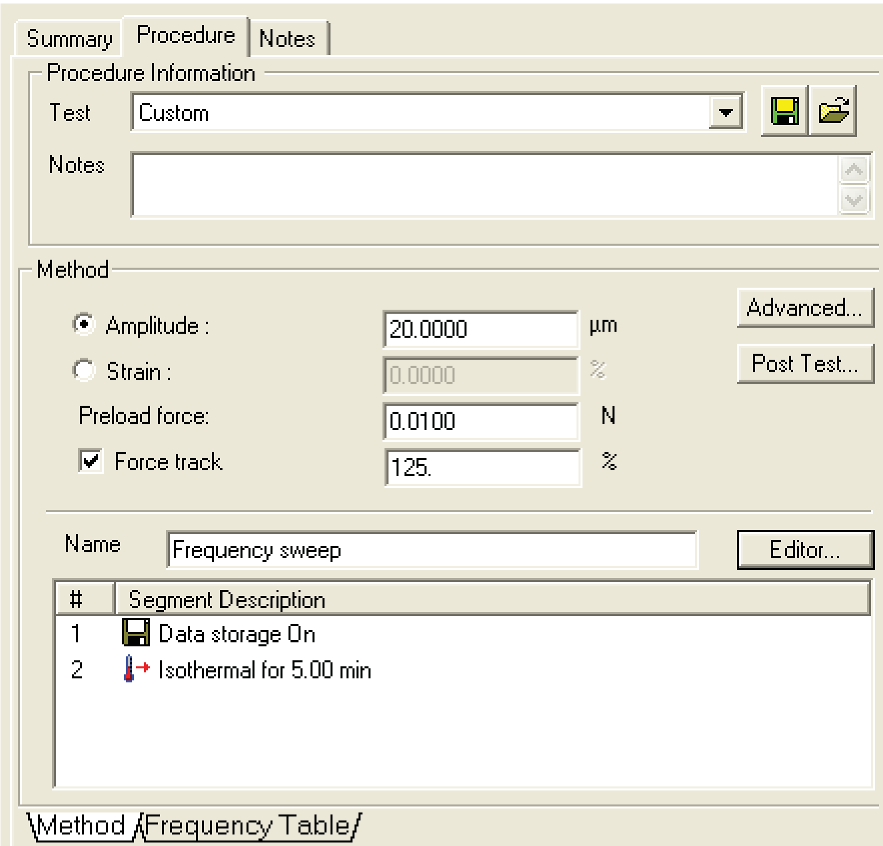
- The material response is monitored at a constant frequency, amplitude and temperature.

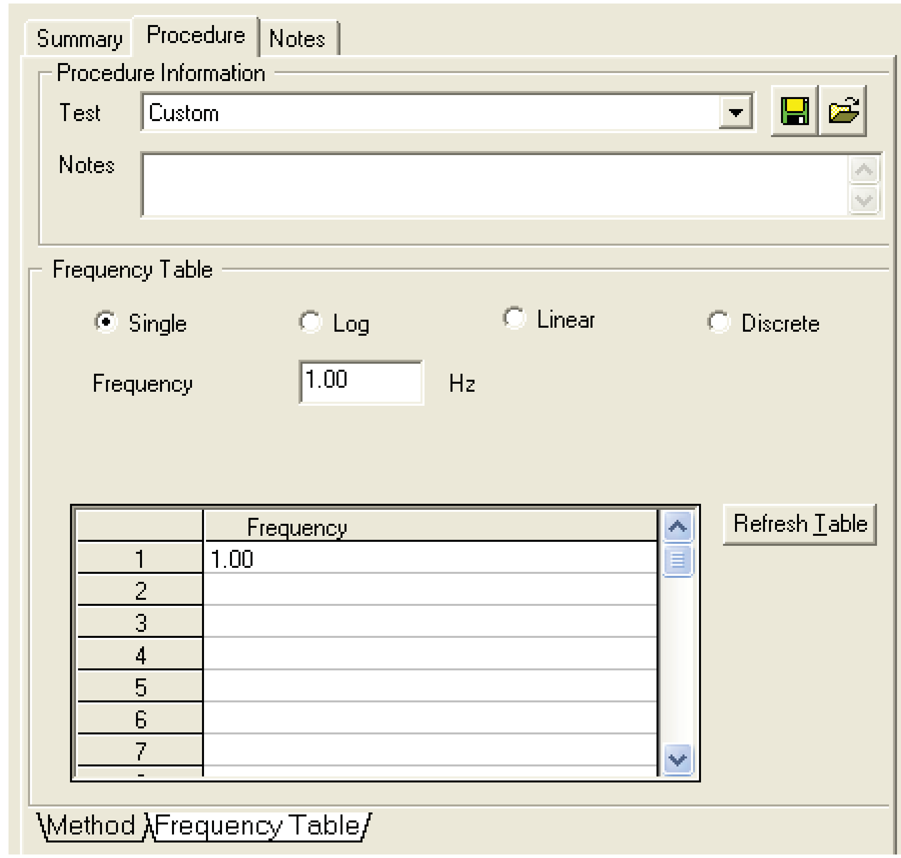
<u>USES</u>

- Cure Studies
- Stability against thermal degradation

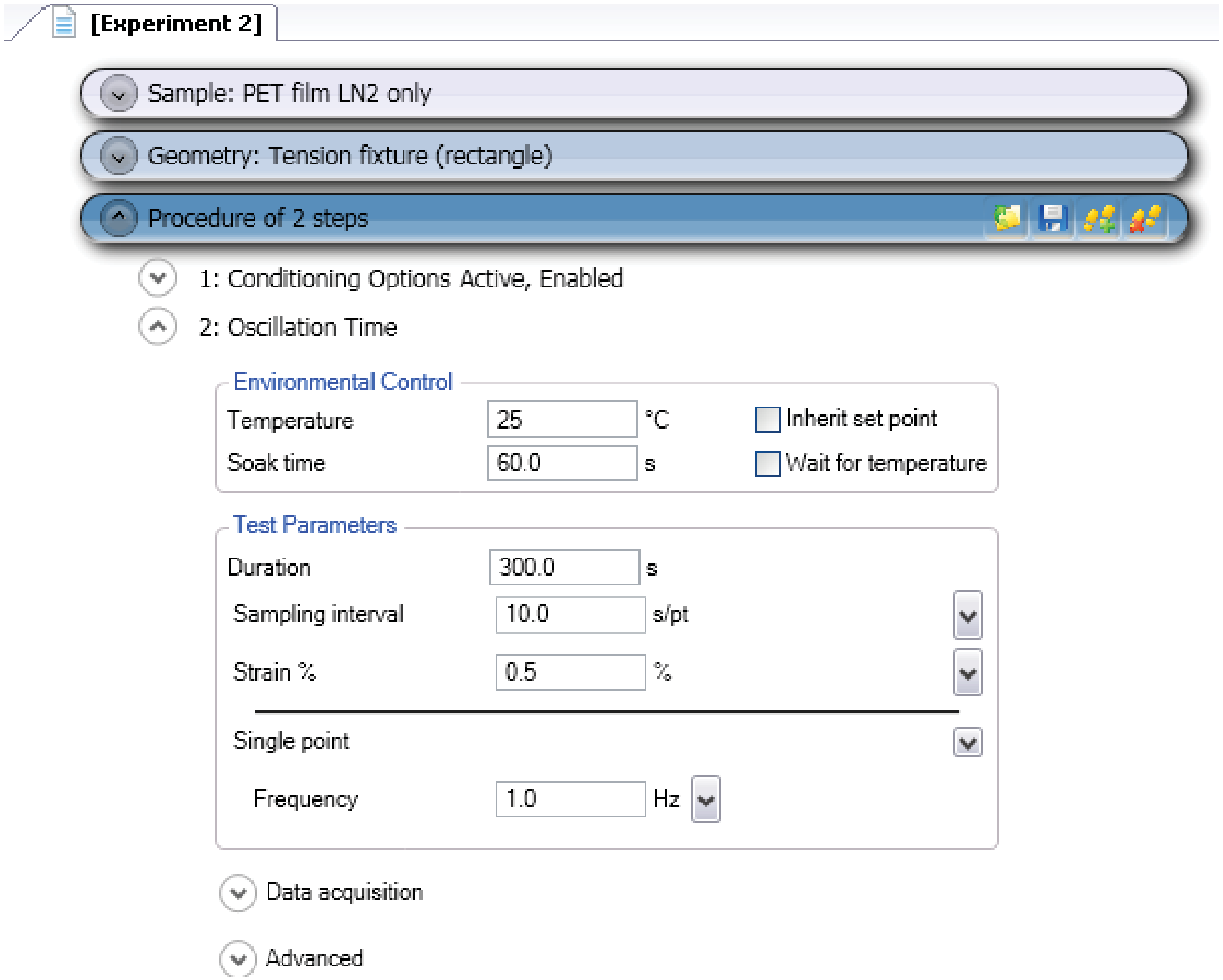
Programming Time Sweep on Q800

Mode: DMA Multi-Frequency-Strain

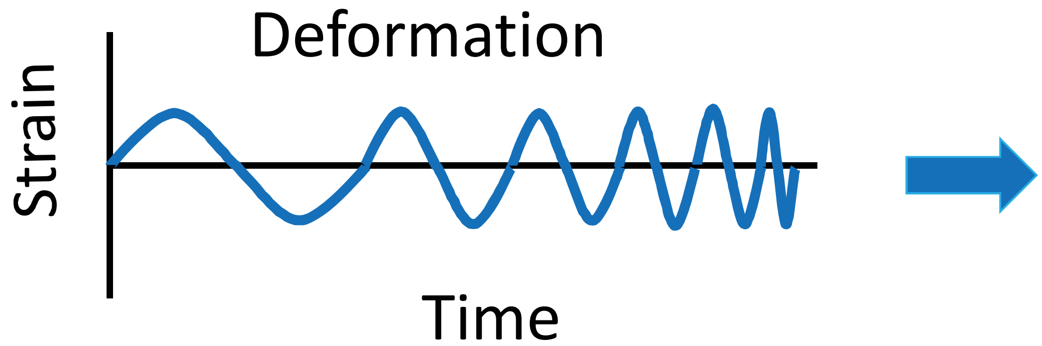




Programming Time Sweep on RSA G2



Frequency Sweep

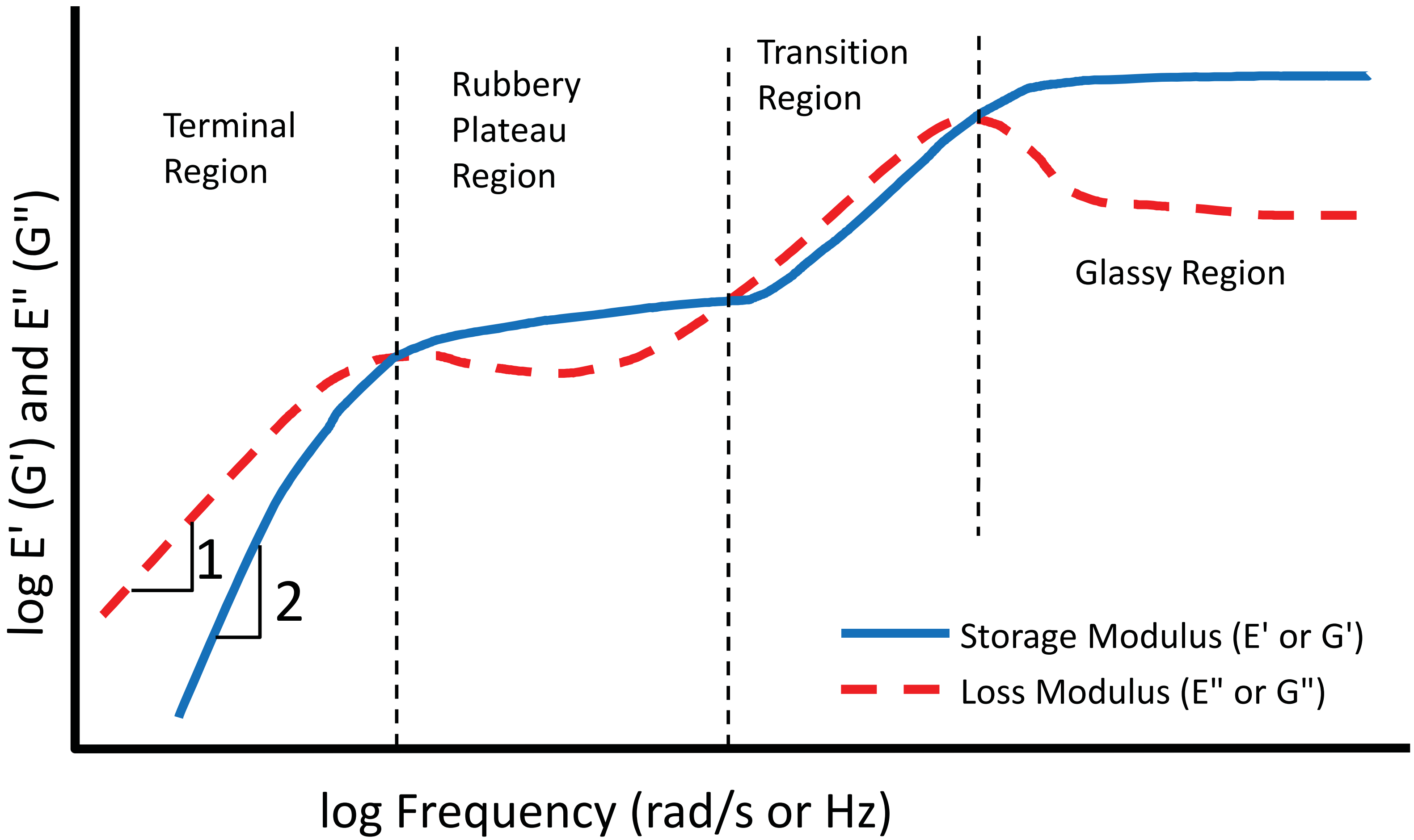


- The material response to increasing frequency (rate of deformation) is monitored at a constant amplitude and temperature.

<u>USES</u>

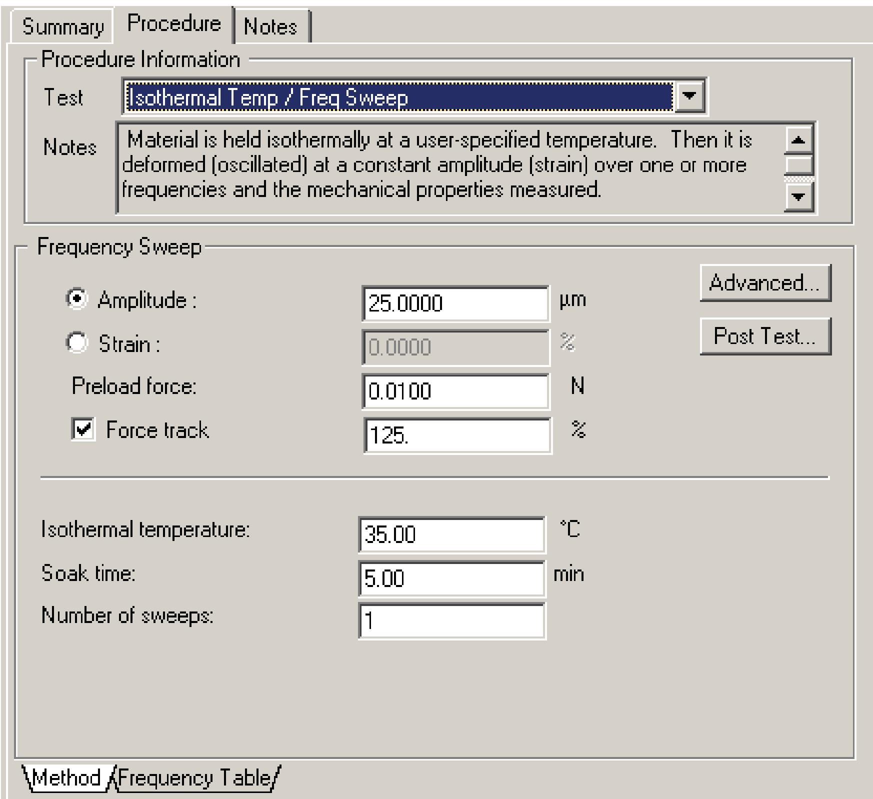
- High and Low Rate (short and long time) modulus properties.
- Polymer melt processing (shear sandwich).
- Extend range with TTS

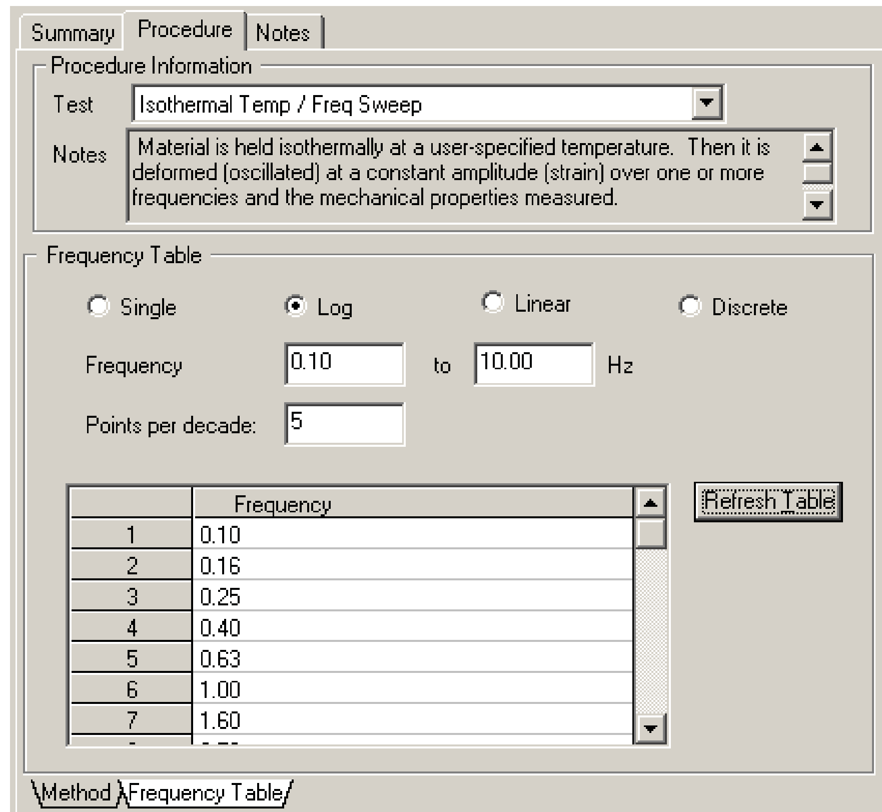
Frequency Sweep: Material Response



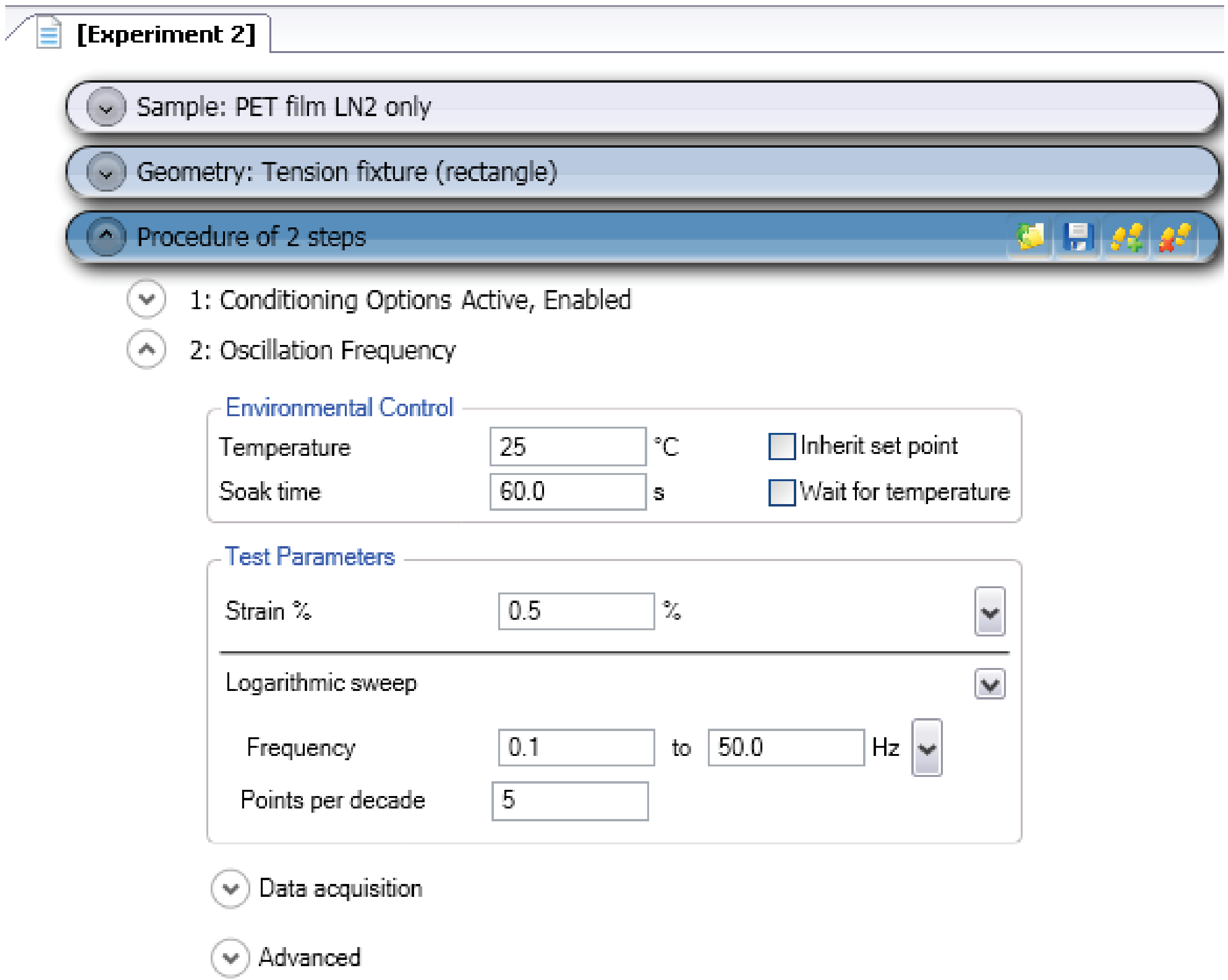
Programming Frequency Sweep on Q800

Mode: DMA Multi-Frequency-Strain

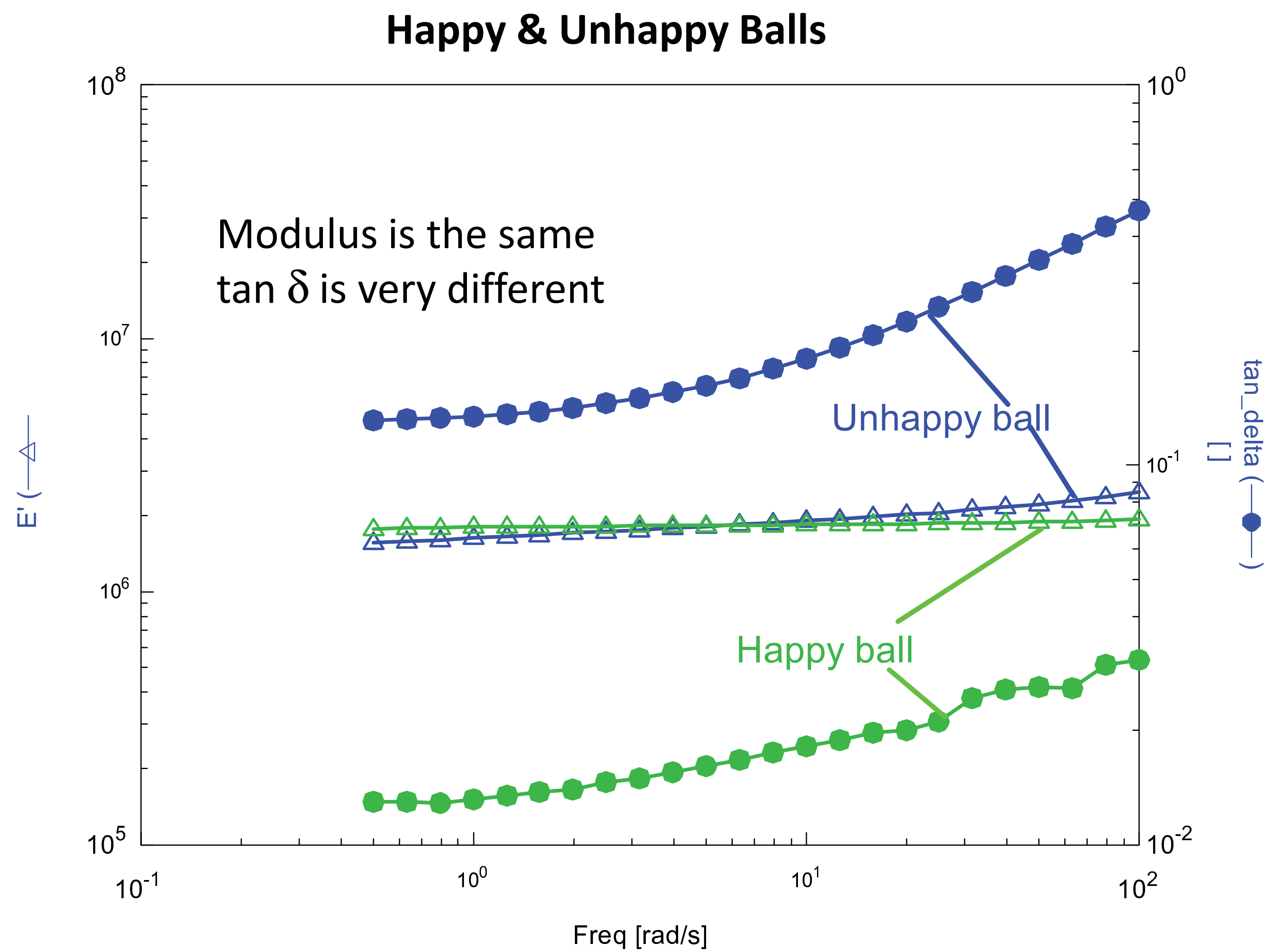




Programming Frequency Sweep on RSA G2

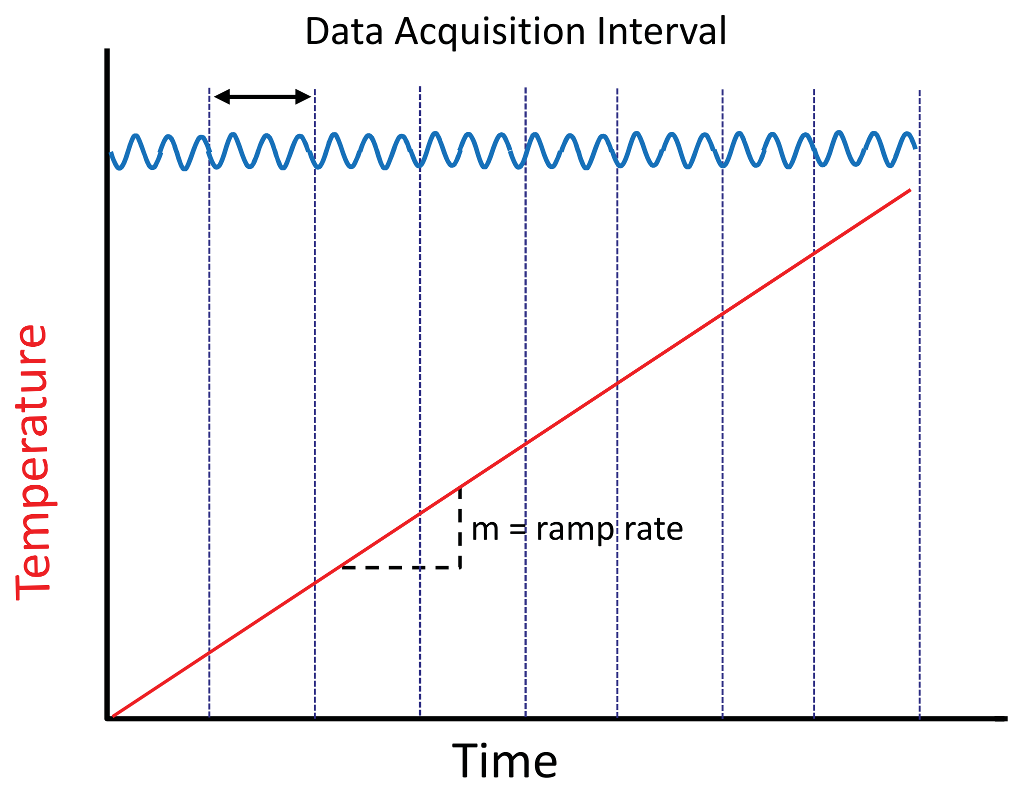


Frequency Sweeps on Solids



Dynamic Temperature Ramp

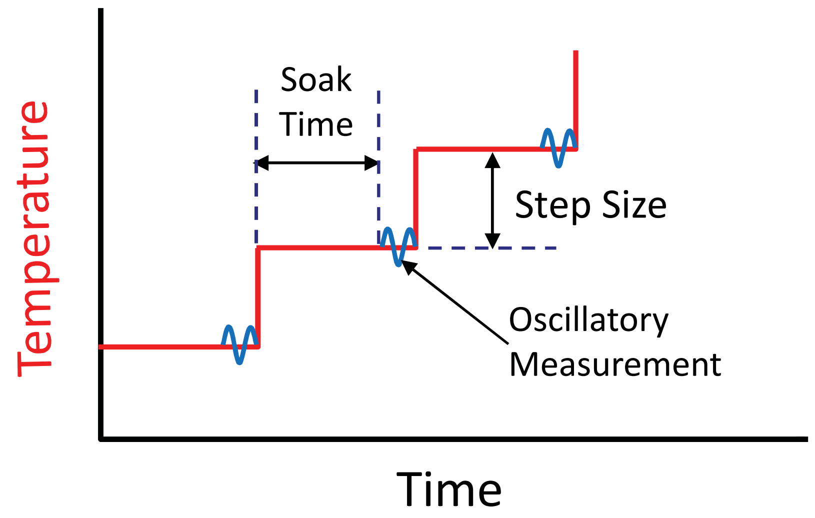
- A linear heating rate is applied. The material response is monitored at a constant frequency and constant amplitude of deformation. Data is taken at user defined time intervals.

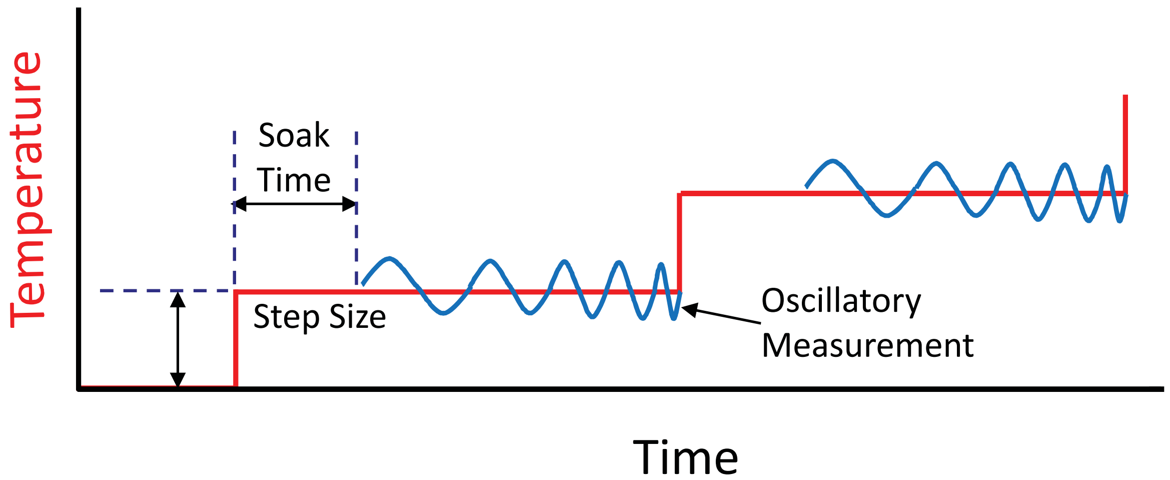


Recommend ramp rate for polymer testing: 1-5˚C/min.

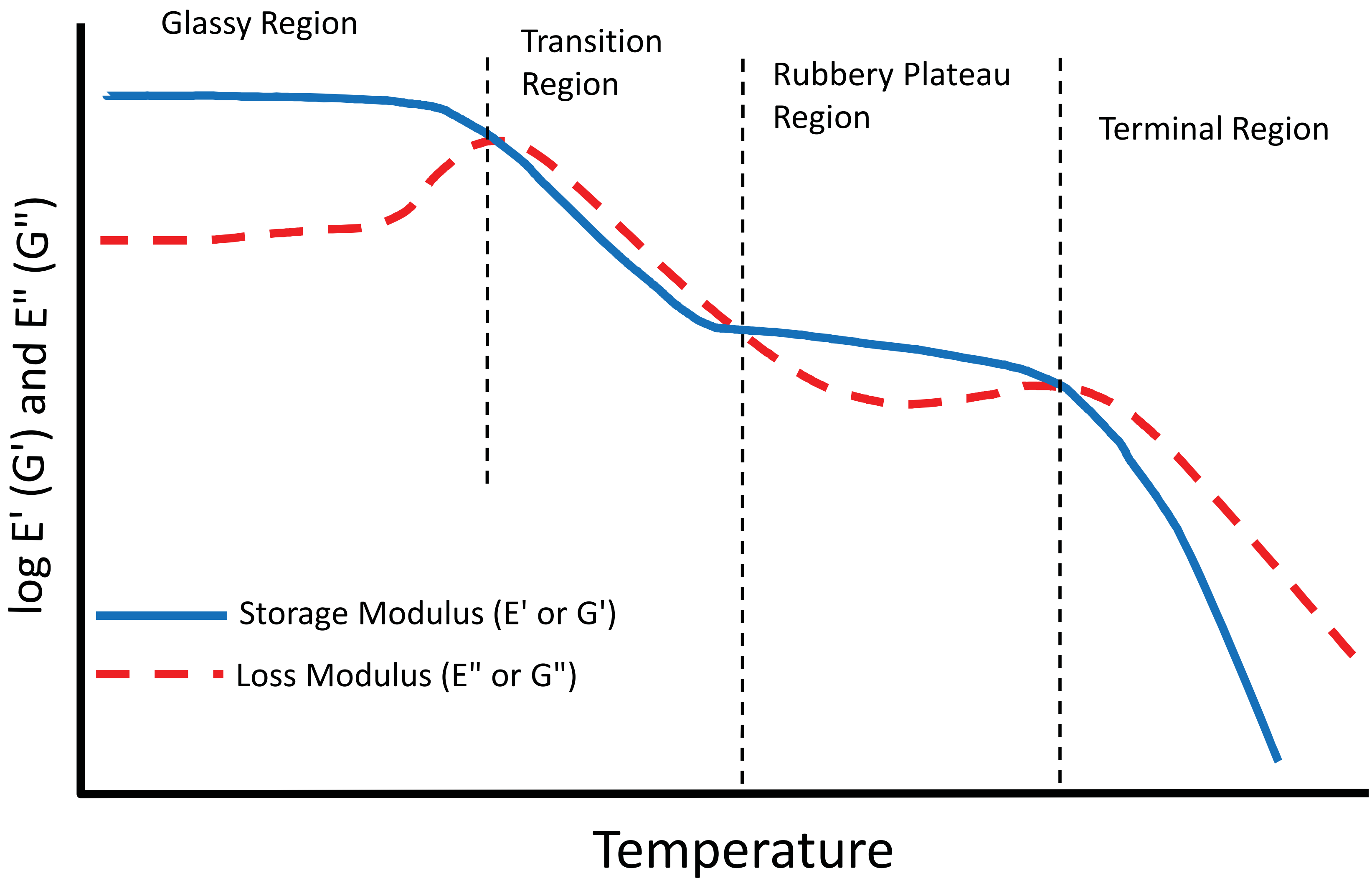
Temperature Step & Hold- Single /Multi-Frequency

- A step and hold temperature profile is applied. The material response is monitored at one, or over a range of frequencies, at constant amplitude of deformation.



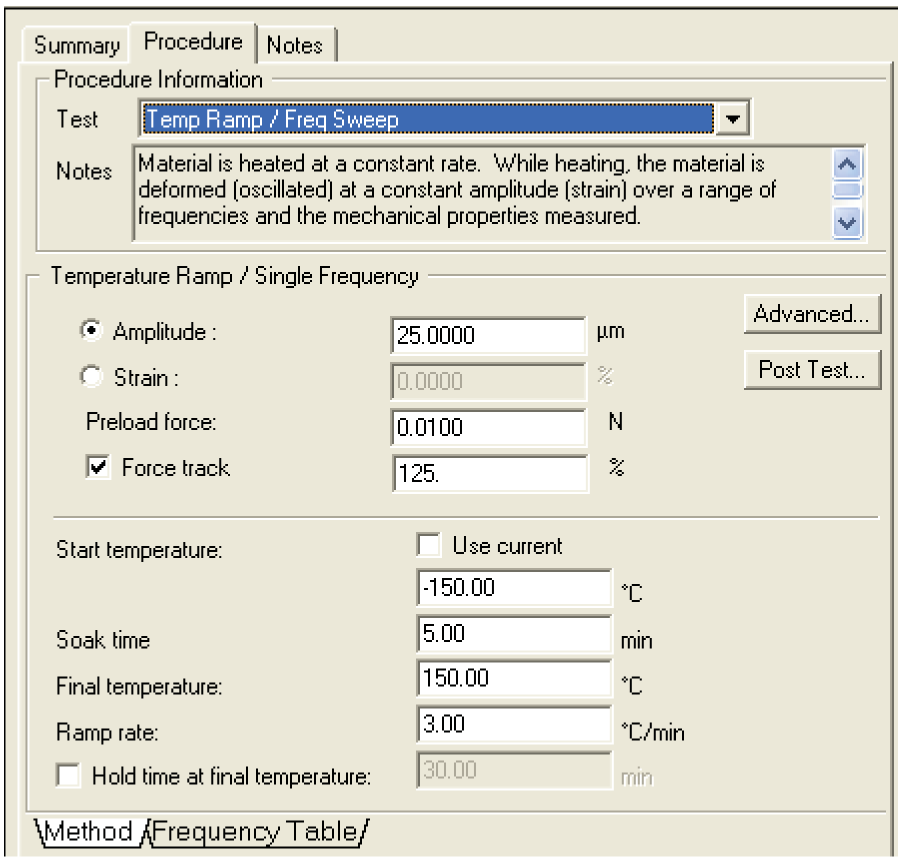


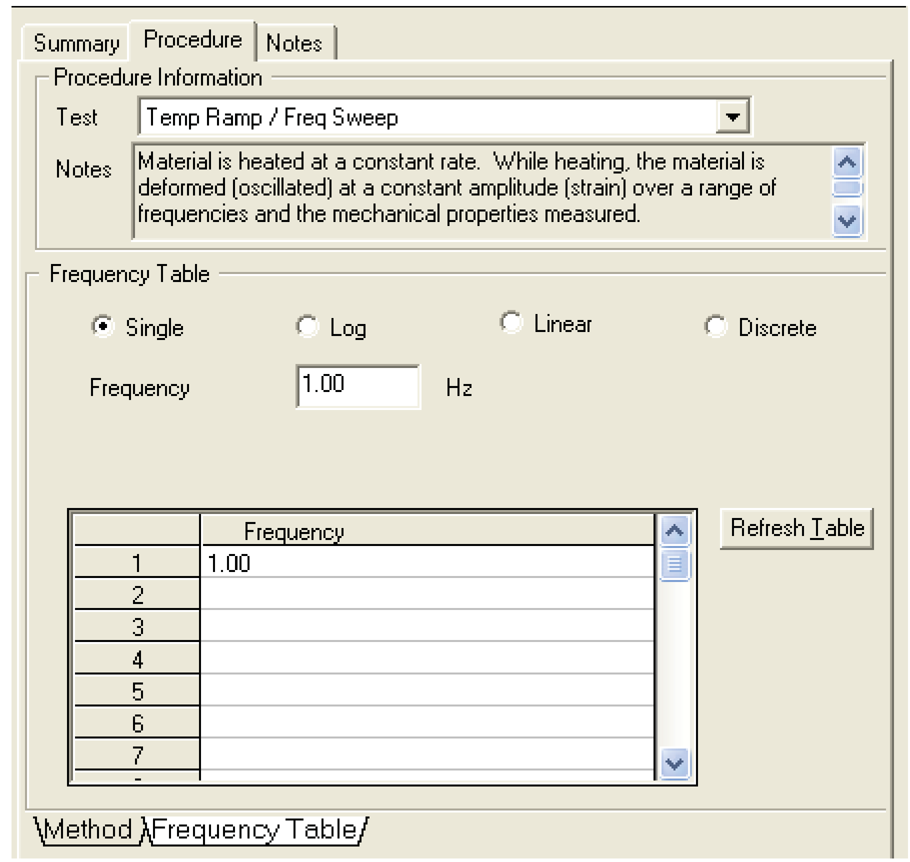
Dynamic Temperature Ramp or Step and Hold: Material Response



Programming Temperature Ramp on Q800

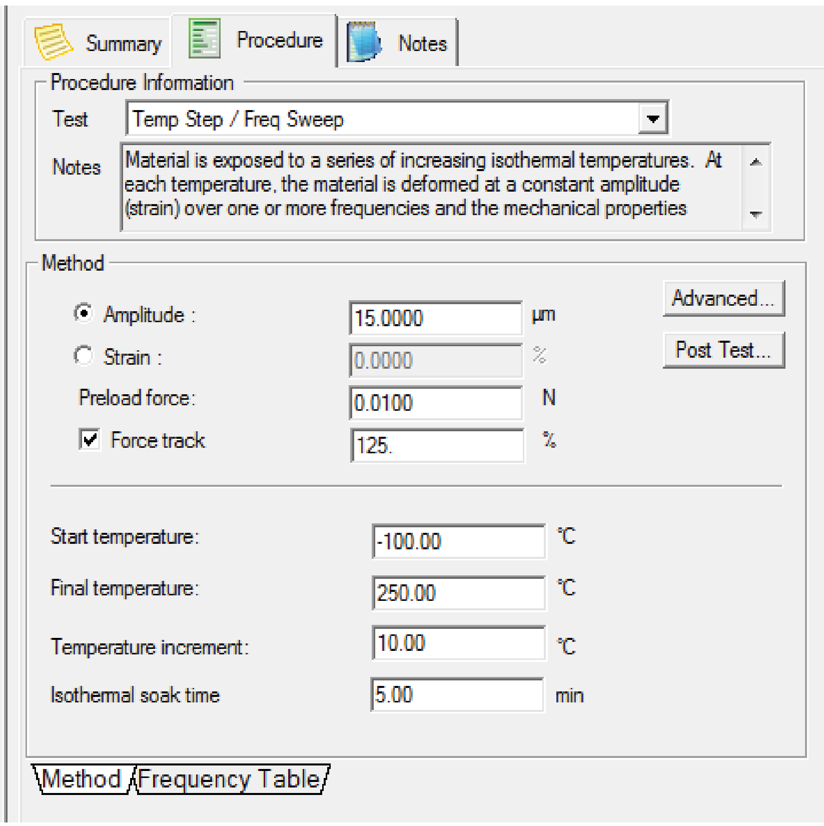
Mode: DMA Multi-Frequency-Strain

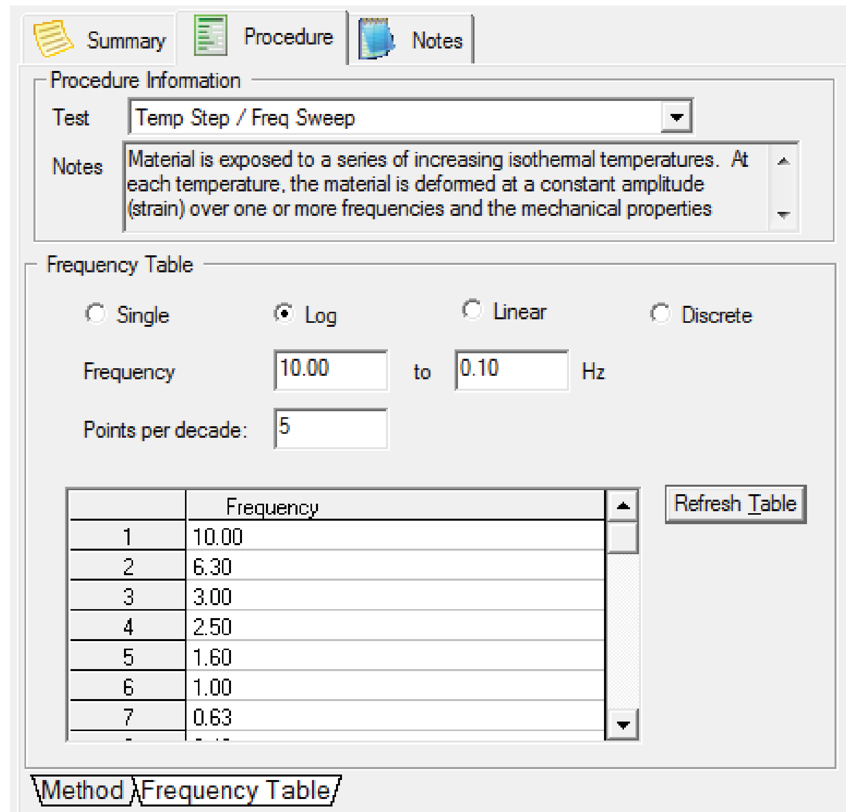




Programming Temperature Step on Q800

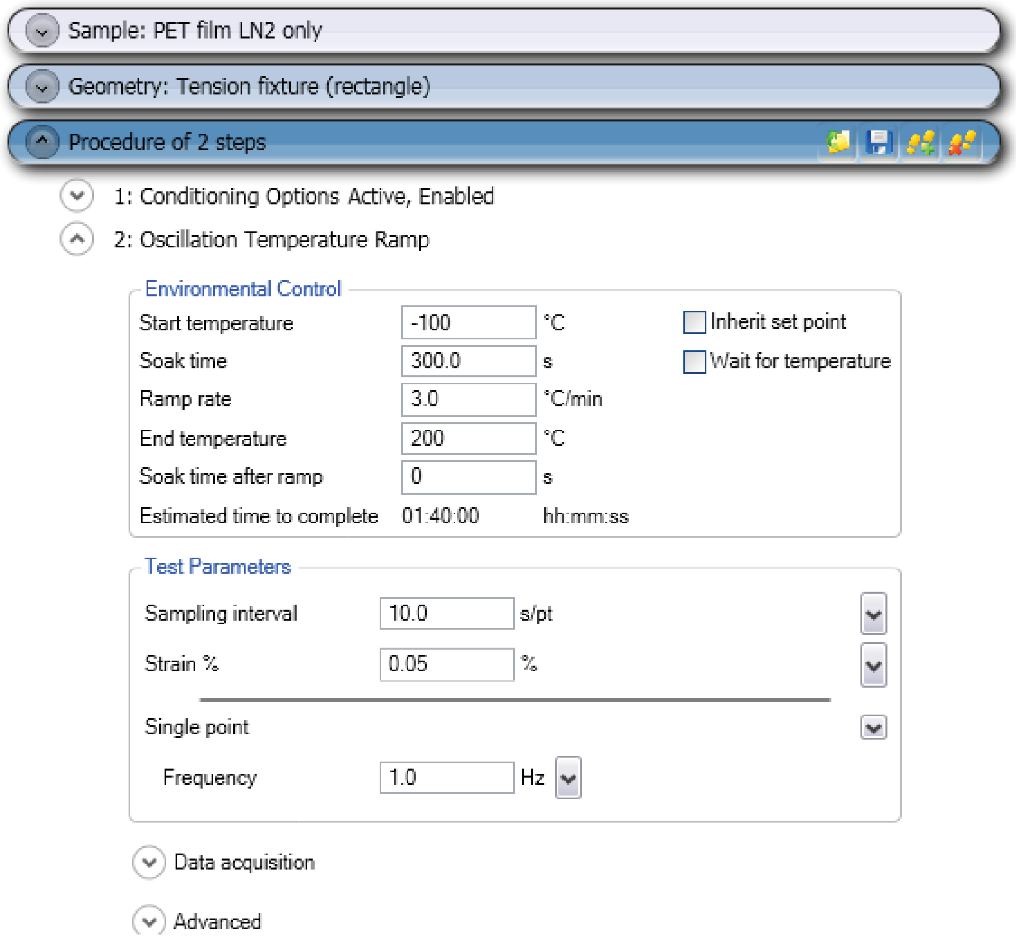
Mode: DMA Multi-Frequency-Strain





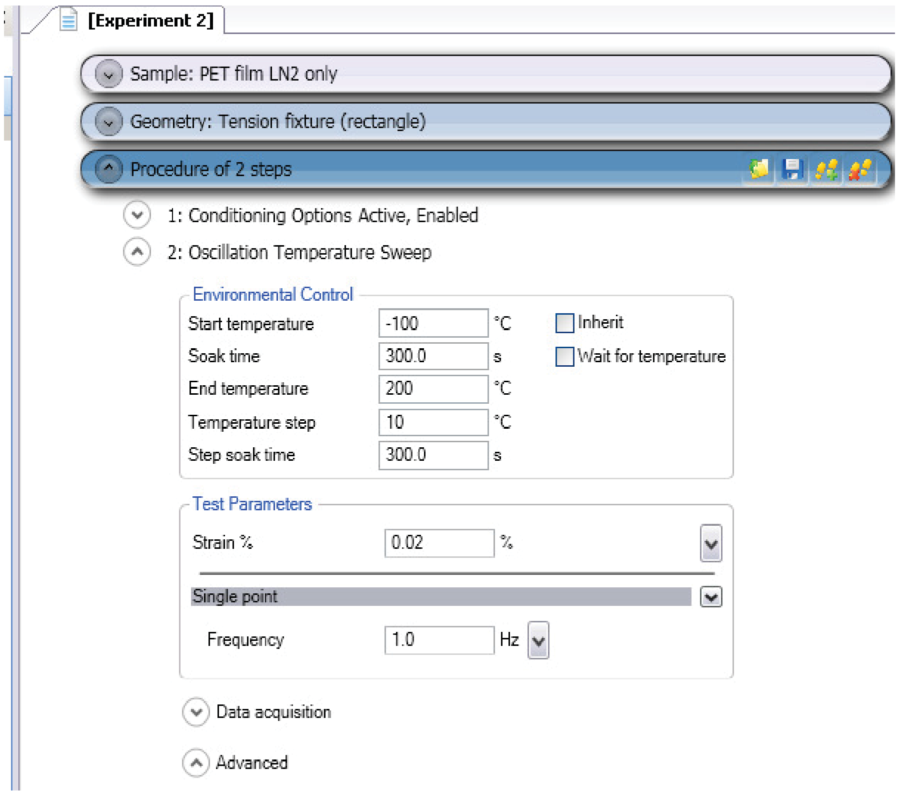
- One may choose to use a single frequency or multi frequencies.

Programming Temperature Ramp on RSA G2

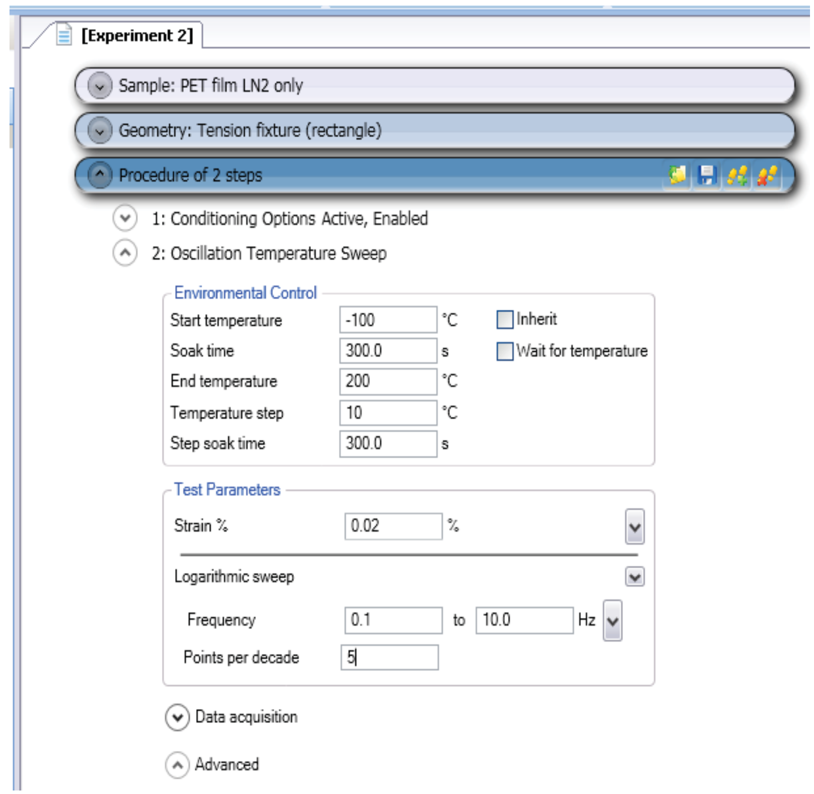


Programming Temperature Step on RSA G2

- Single frequency

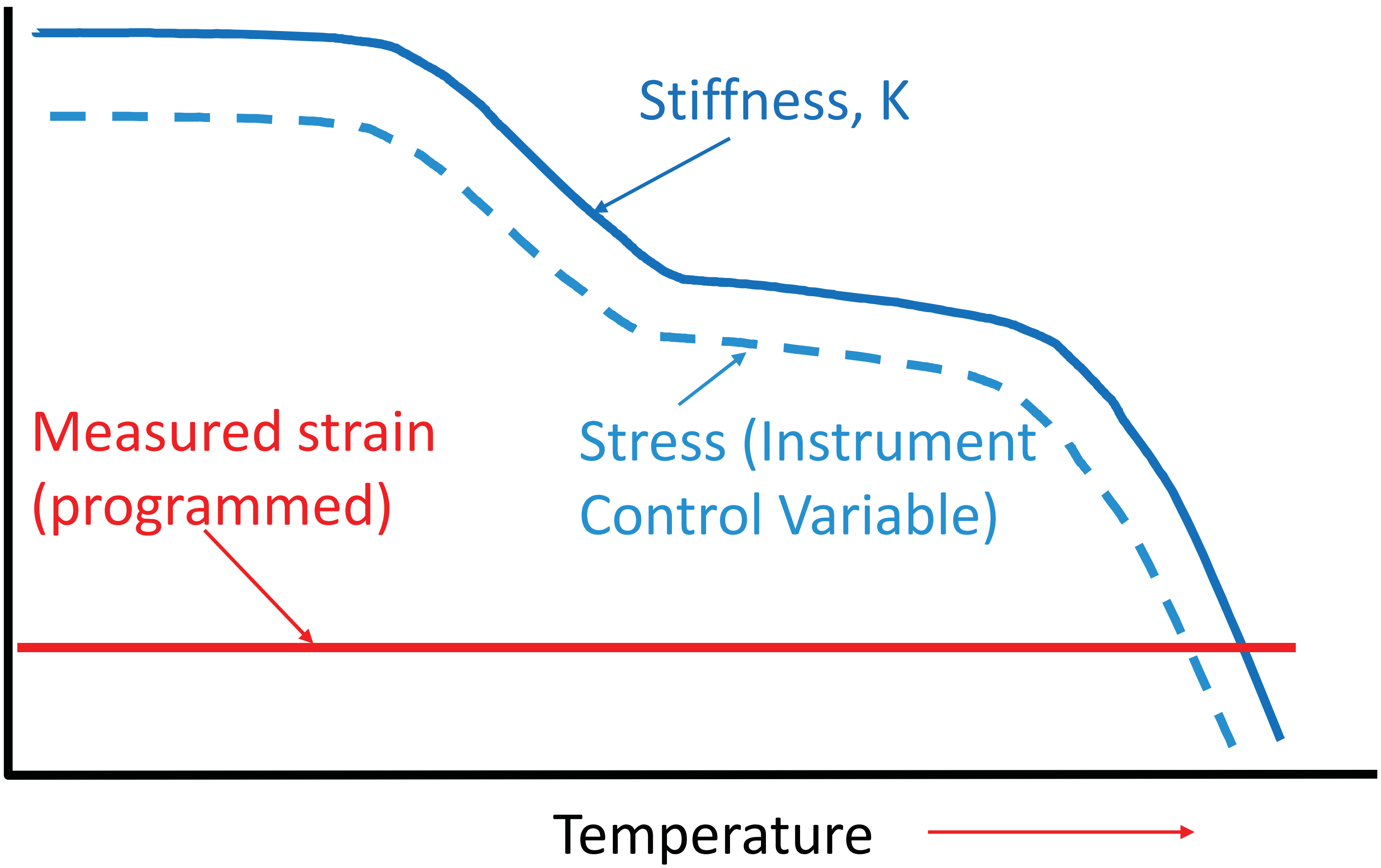


- Multi frequency



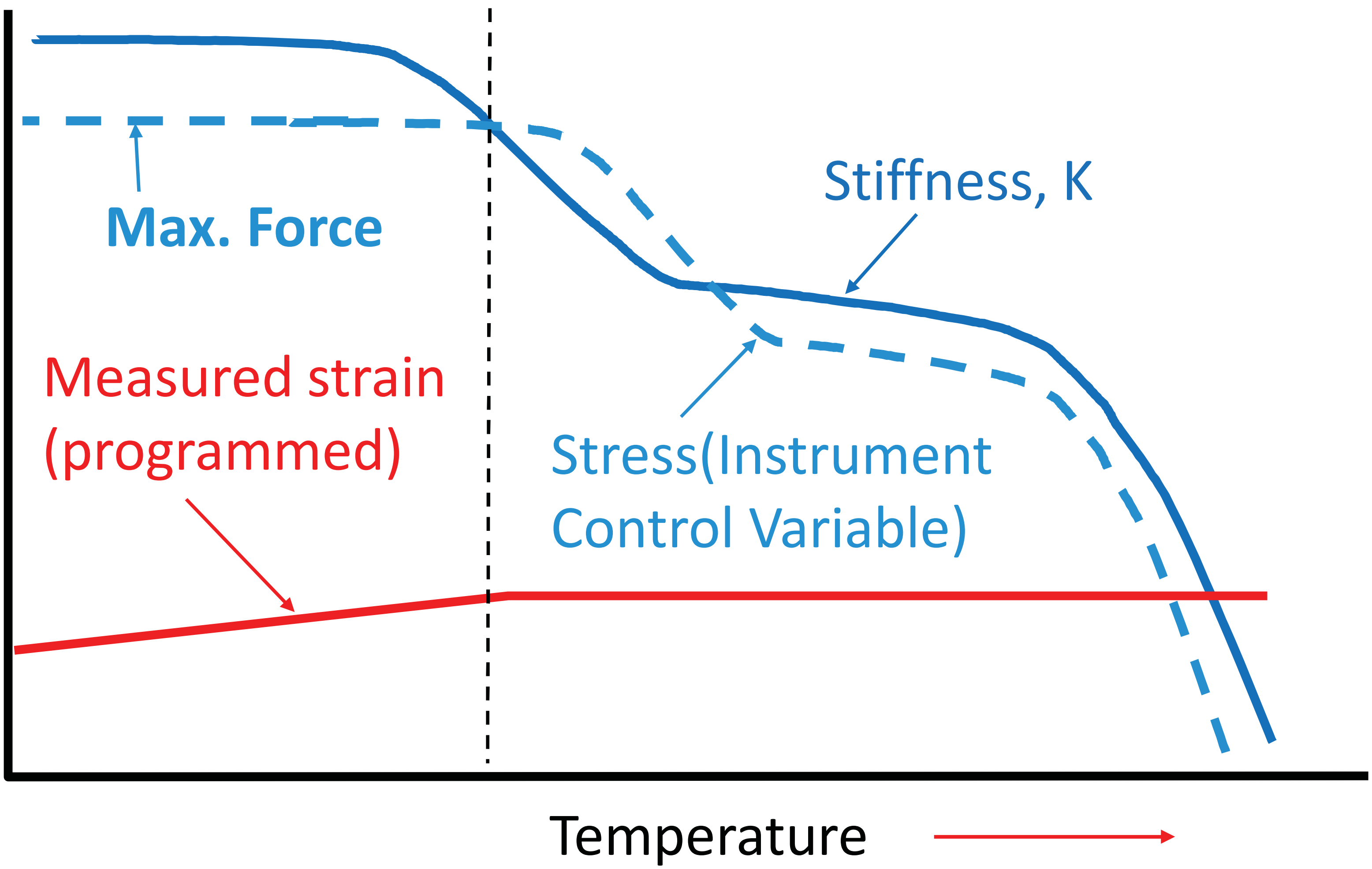
Stress Control During Temperature Ramp

Scenario 1: Maximum force is not required at maximum stiffness



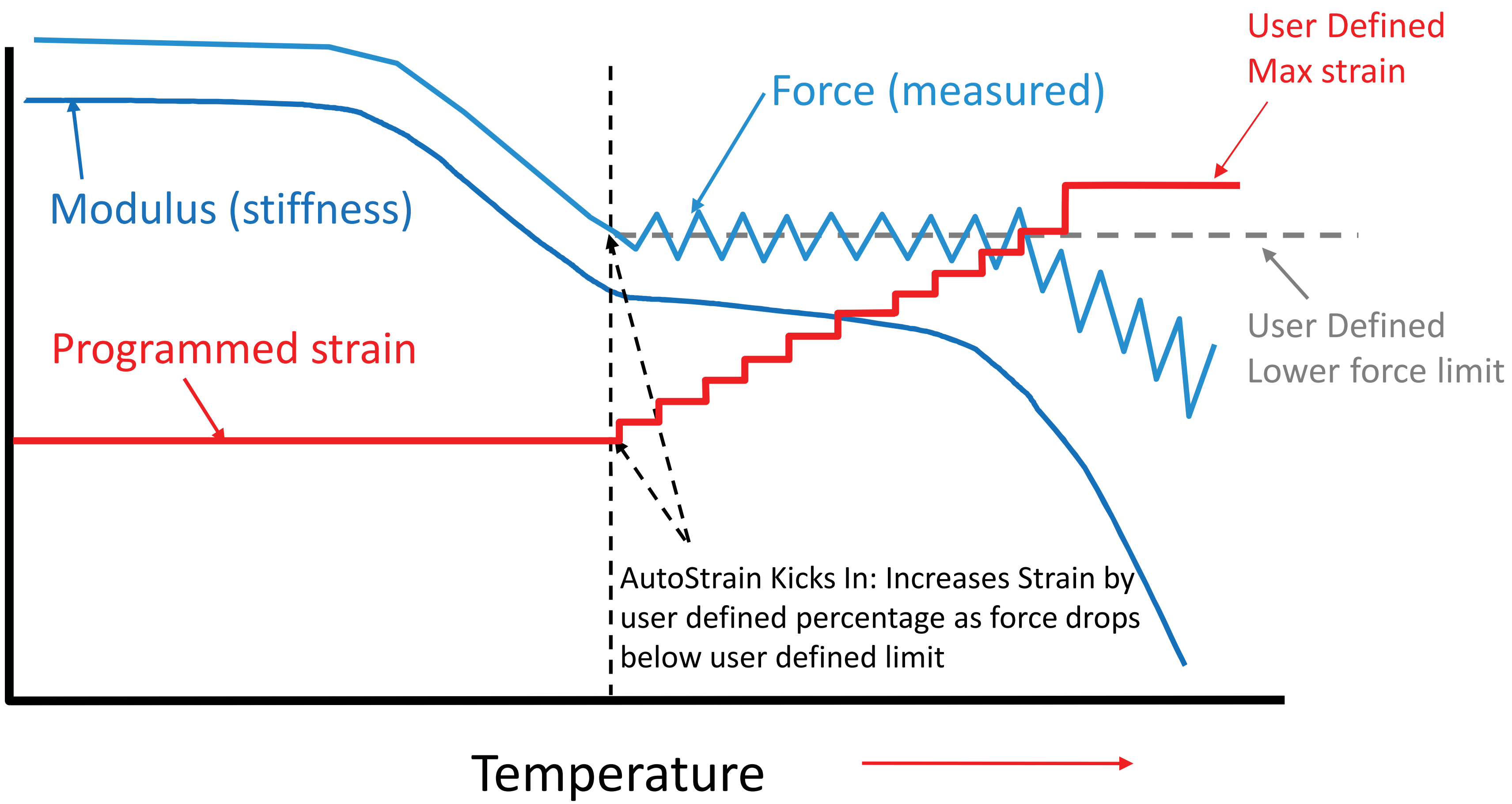
Stress Control During Temperature Ramp

Scenario 2: Maximum force is required at maximum stiffness

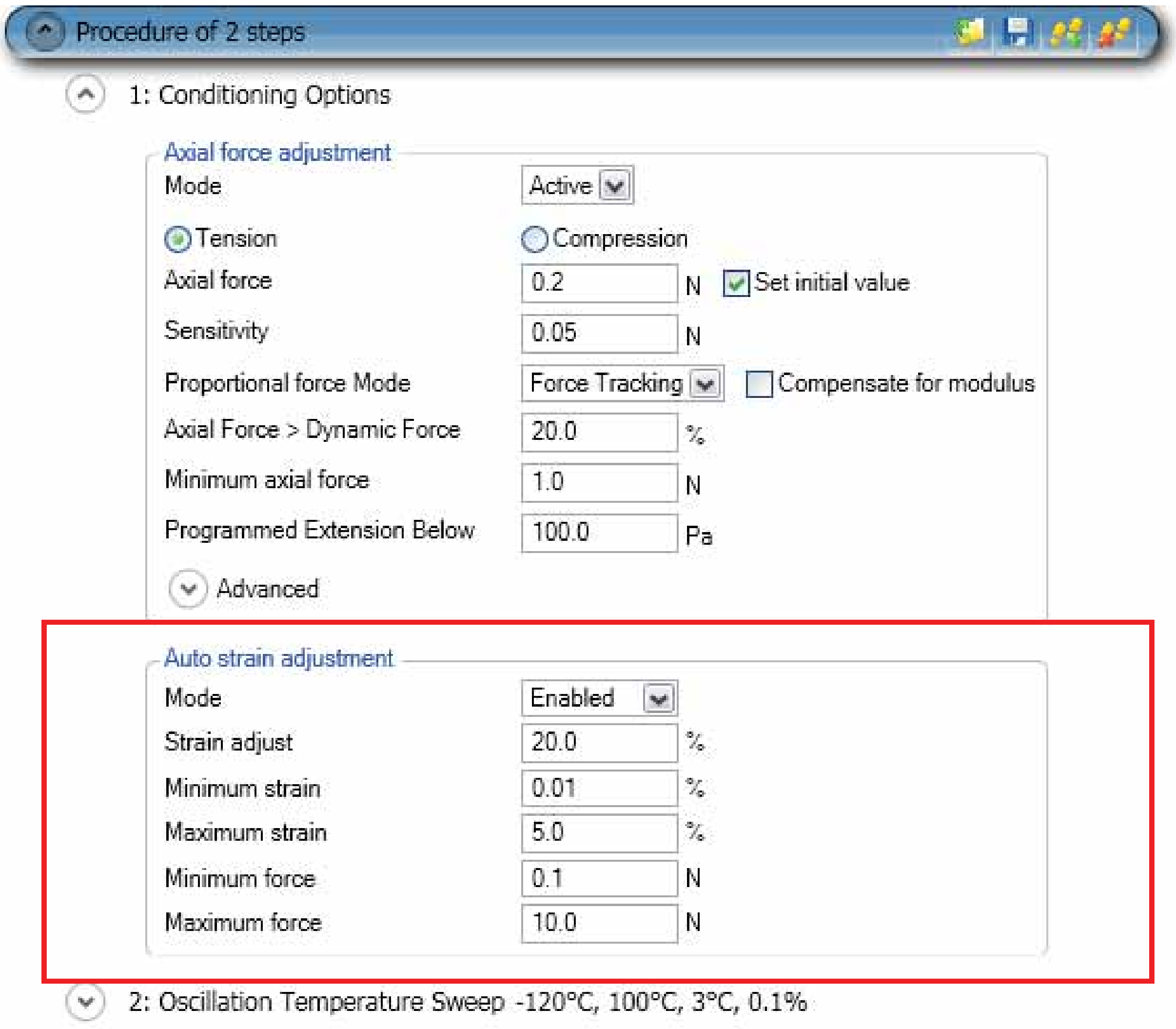


Auto-Strain on RSA G2

Over temperature range covered, force falls below user defined lower limit in strain controlled DMA.



Axial Force Control and Auto-Strain on RSA G2



Using Auto Strain in a Temperature Ramp- Up

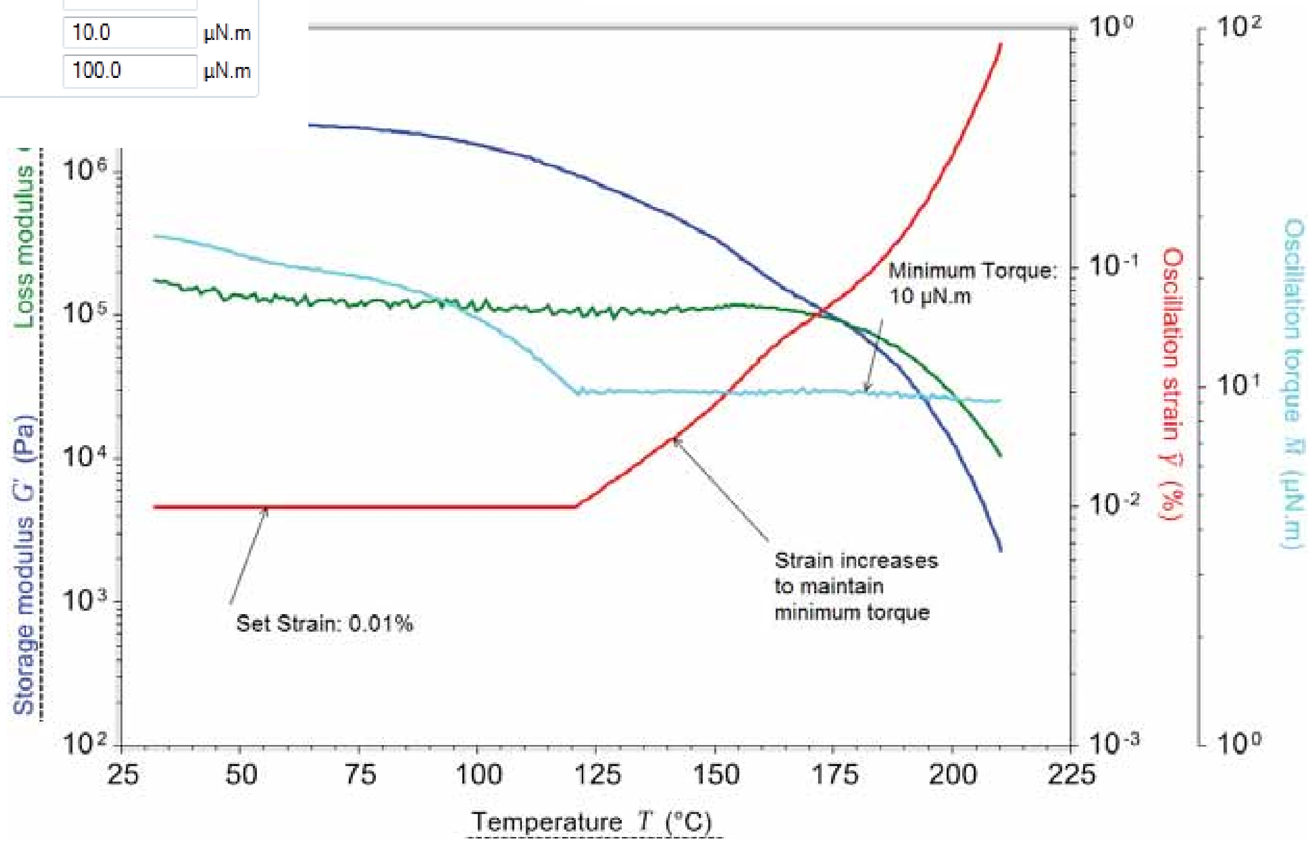
1: Conditioning Options

Auto strain adjustment

Mode	Enabled	
Strain adjust	20.0	%
Minimum strain	1.0e-3	%
Maximum strain	1.0	%
Minimum torque	10.0	µN.m
Maximum torque	100.0	µN.m

Test Parameters

Sampling rate	1.0	pts/s
Strain %	0.01	%
Single point		
Angular frequency	10.0	rad/s

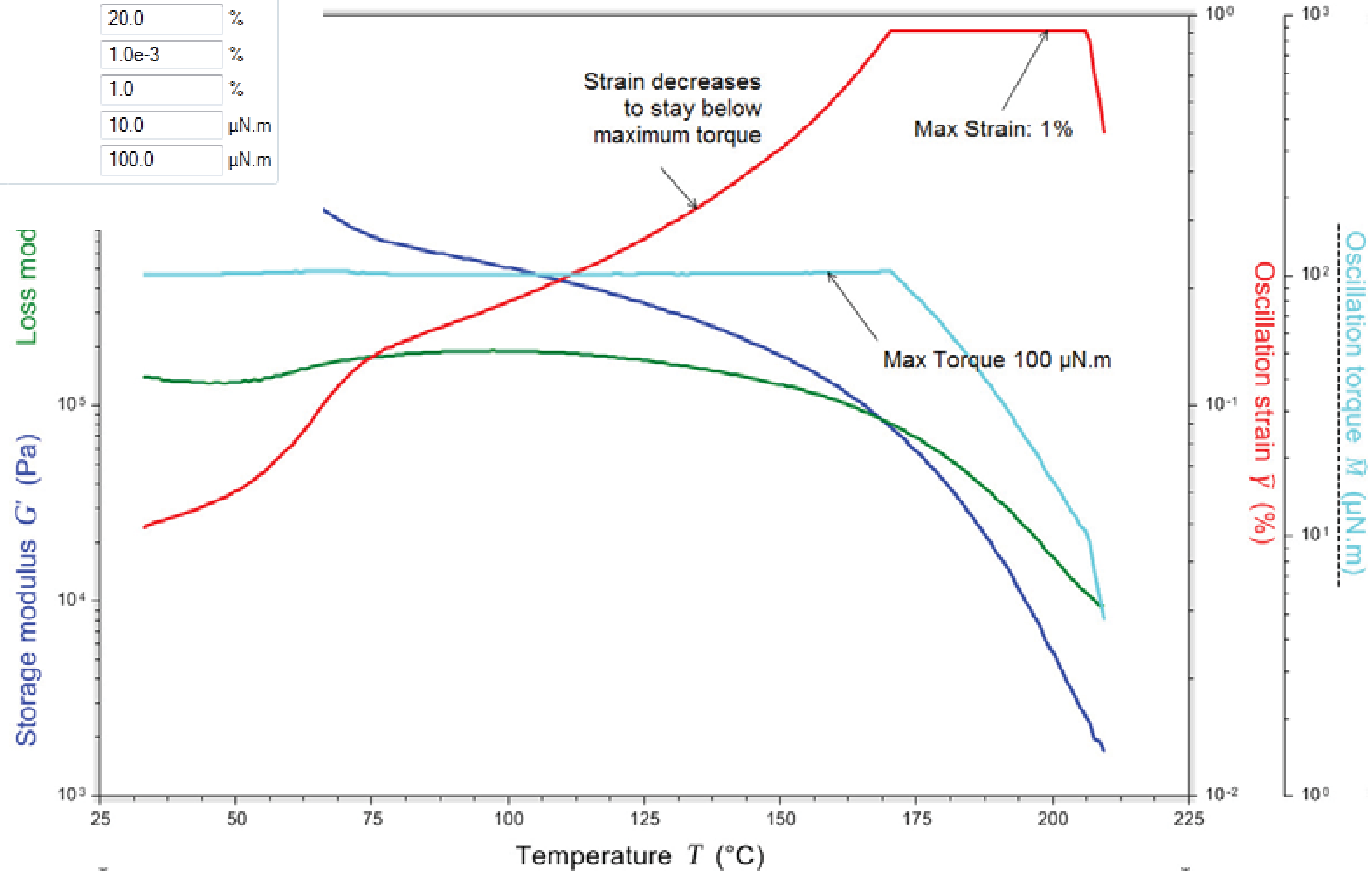


Using Auto Strain in a Temperature Ramp- Down

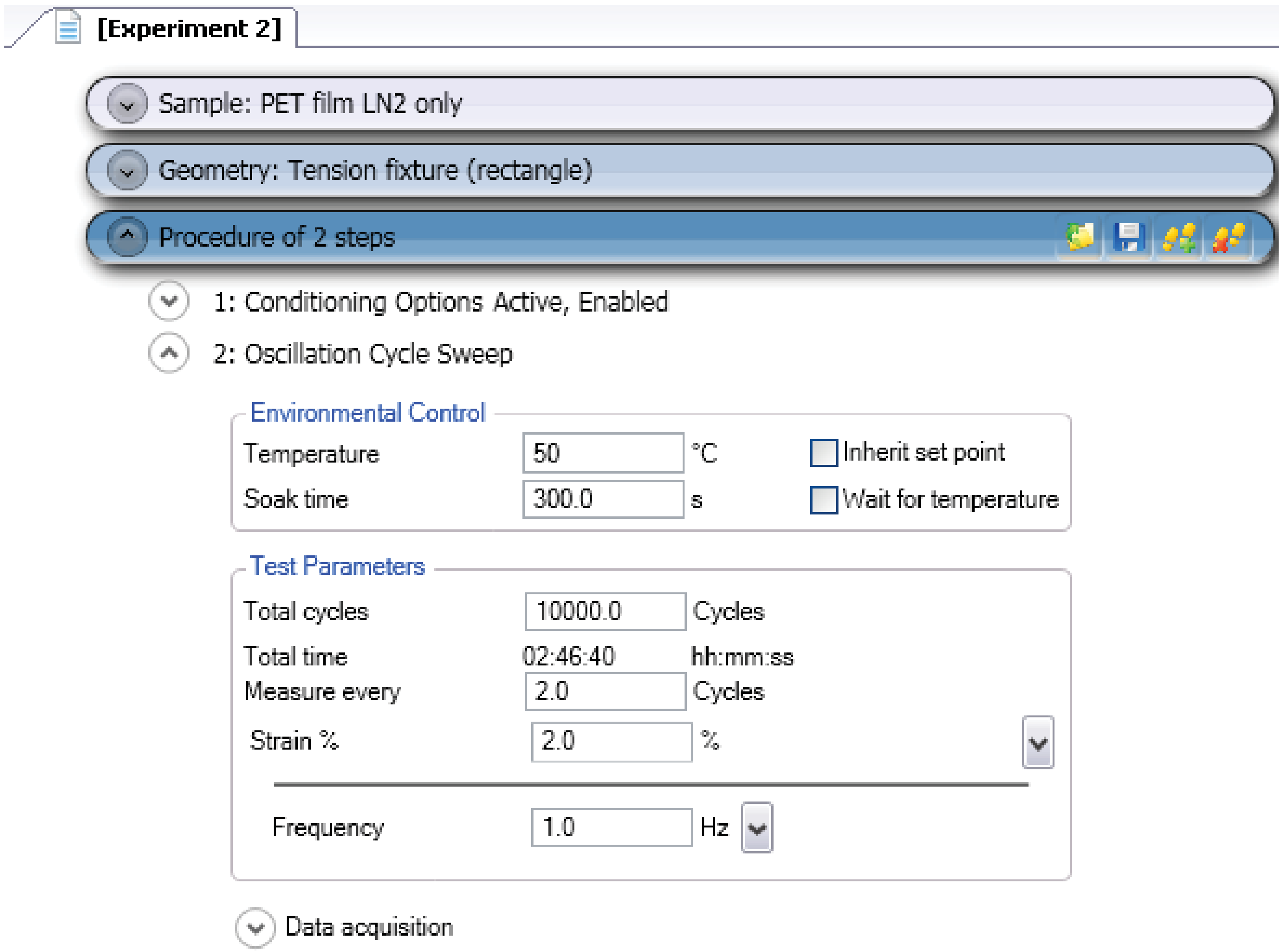
1: Conditioning Options

Auto strain adjustment

Mode	Enabled	
Strain adjust	20.0	%
Minimum strain	1.0e-3	%
Maximum strain	1.0	%
Minimum torque	10.0	μN.m
Maximum torque	100.0	μN.m



DMA Fatigue Testing on RSA-G2



Experimental Considerations

- The Sample
  - Deformation Mode
  - Stiffness (sample size and shape)
  - Clamp Type (sample size and shape)
- Static Force/Force Track
- Amplitude (single/multiple)
- Frequency (Single/multiple)
- Heating Rate/Temperature Program

Selecting an Amplitude/Strain

- Strain sweep/force ramp to determine linear viscoelastic region
  - Must go from low to high strain
- Factors to consider: Force, Yielding, Noise
- Force
  - Maximum - 18 N on Q800
  - Maximum - 35 N on RSA G2
- Yielding /Creep
  - If static/dynamic force too high specimen may deform irreversibly
  - Must consider behavior at all temperatures and frequencies
- Noise
  - Higher amplitude = lower noise (generally)
  - Trade off against yielding/creep behavior

Selecting an Amplitude - Suggested Starting Points

On Q800 and RSA G2:

Clamp	Amplitude (µm)
Tension Film or Fiber	15 to 25
Compression	10 to 20
3 Point Bend	25 to 40
Dual/Single Cantilever	20 to 30
Shear Sandwich	10 to 20
Specialty Fiber	15 to 25

Strain of 0.02% - 1% (needs to be in the LVR)

Frequencies

- **Single Frequency**
  - Temperature ramp most popular for rapid evaluations
  - 1 or 10 Hz (6.28 or 63 rad/sec) for most experiments
- **Multiple Frequencies**
  - Frequency sweeps at ambient for viscoelastic properties
  - Frequency sweeps at multiple temperatures for Time-Temperature Superpositioning (TTS)
  - Run from high to low frequencies for faster initial data acquisition
- **Data Collection Rate**
  - Lower frequencies take longer time - control experiment
  - More frequencies = longer experiment

Temperature Program

Ramps: Sample and Frequency Considerations

- Large samples = thermal gradients
- Minimum frequency/number of frequencies limit rate
    - 3°C/min is reasonable compromise

Isothermal Steps

- Can be used to ensure thermal and mechanical equilibrium
- Must be used for large frequency tables or low frequency
- Use 5°C or smaller steps (2 - 3°C recommended)

Available DMA Test Modes

Transient Tests

Transient Testing

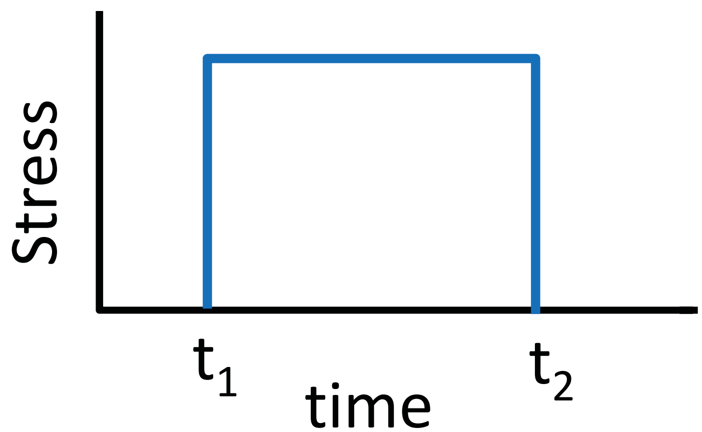
Available transient test modes

- Creep-Recovery
- Stress Relaxation
- Iso-strain Temperature Ramp
- Iso-force Temperature Ramp
- Stress-Strain Tests (Instron type of test)

Creep Recovery Experiment

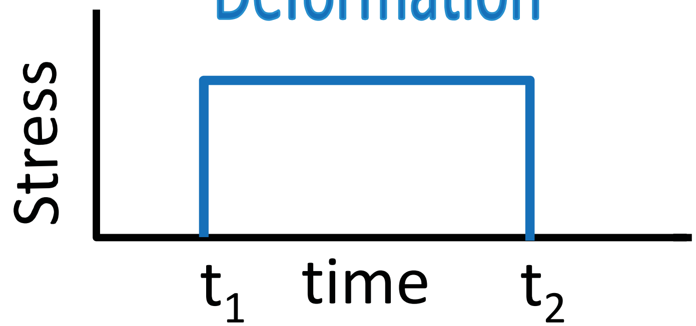
- Stress is applied to sample instantaneously, t_1, and held constant for a specific period of time. The strain is monitored as a function of time (γ(t) or ε(t)).

- The stress is reduced to zero, t_2, and the strain is monitored as a function of time (γ(t) or ε(t)).



Creep Recovery Experiment

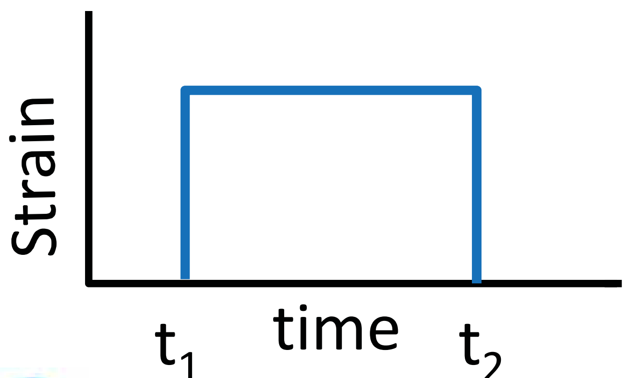
Deformation



Response of Classical Extremes

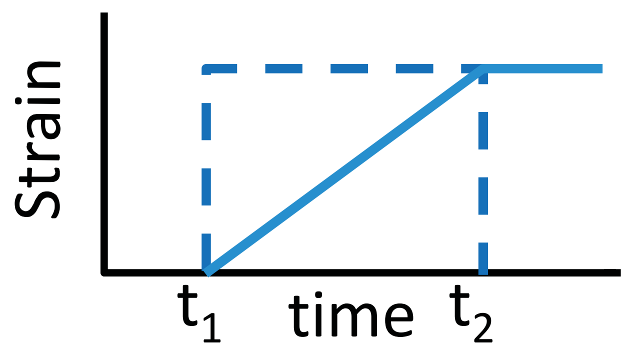
Elastic

- Stain for t>t1 is constant
- Strain for t >t2 is 0

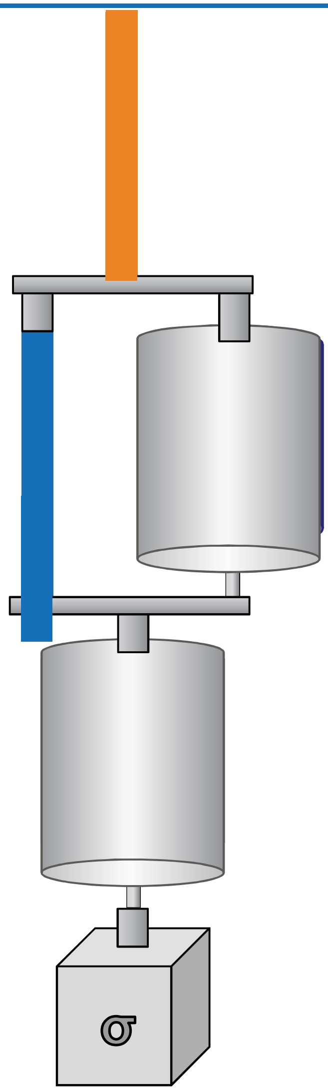


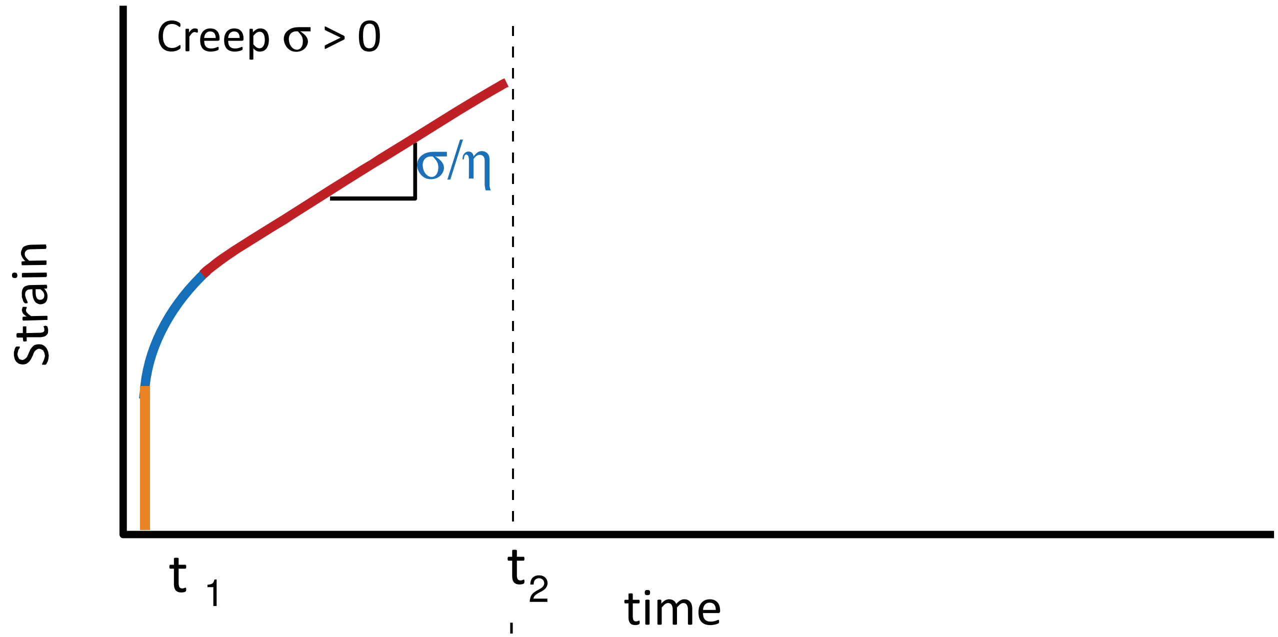
Viscous

- Stain rate for t>t1 is constant
- Strain for t>t1 increase with time
- Strain rate for t >t2 is 0



Creep Recovery: Response of Viscoelastic Material

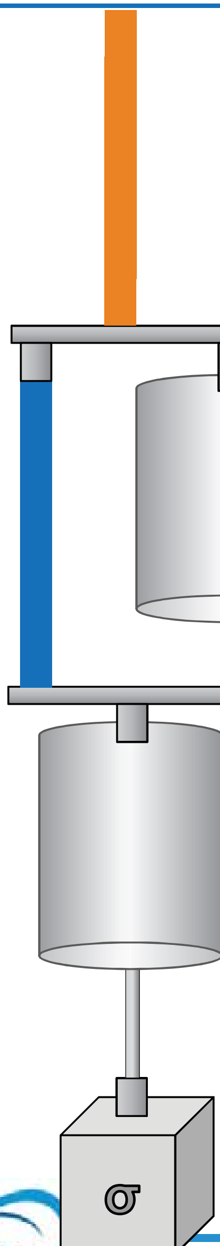


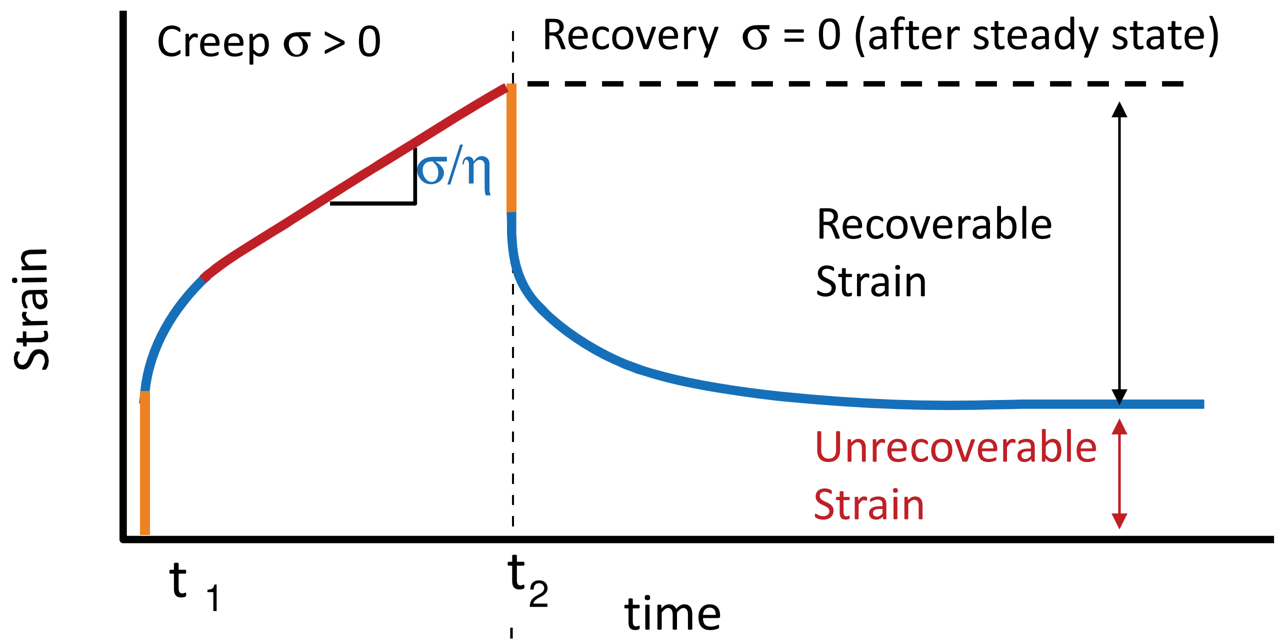


Strain rate decreases with time in the creep zone, until finally reaching a steady state.

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

Creep Recovery: Response of Viscoelastic Material



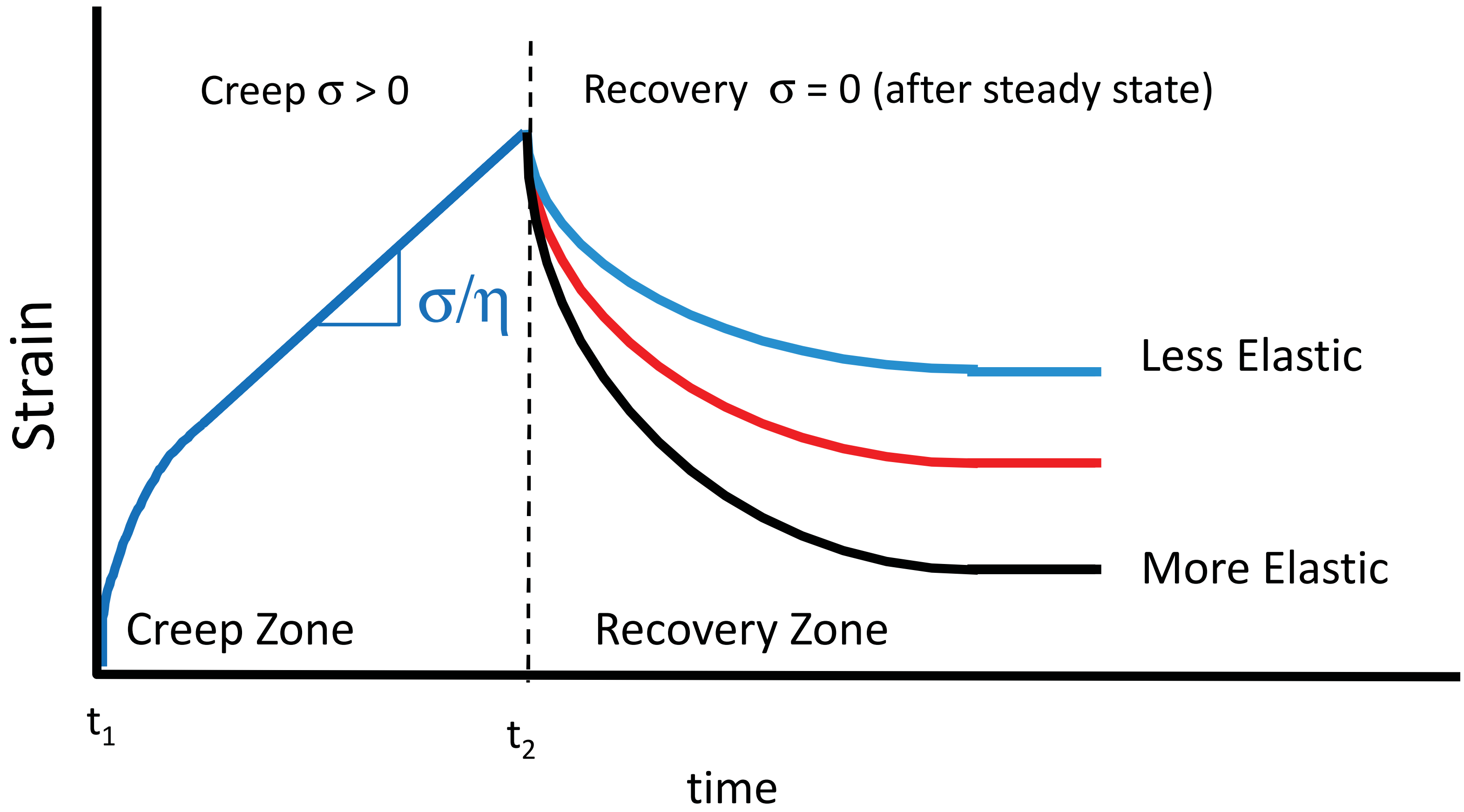


Strain rate decreases with time in the creep zone, until finally reaching a steady state.

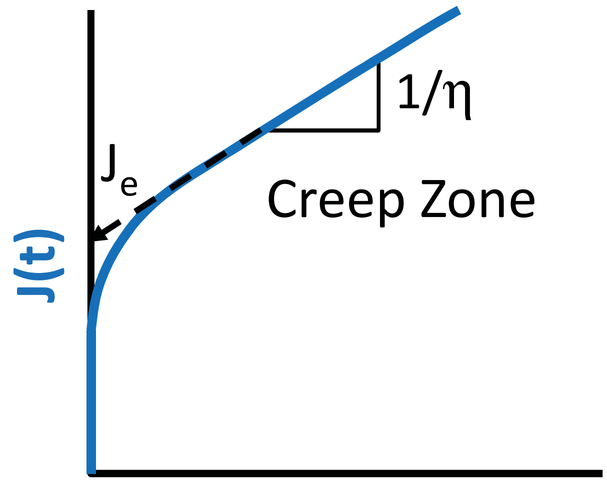
In the recovery zone, the viscoelastic fluid recoils, eventually reaching a equilibrium at some small total strain relative to the strain at unloading.

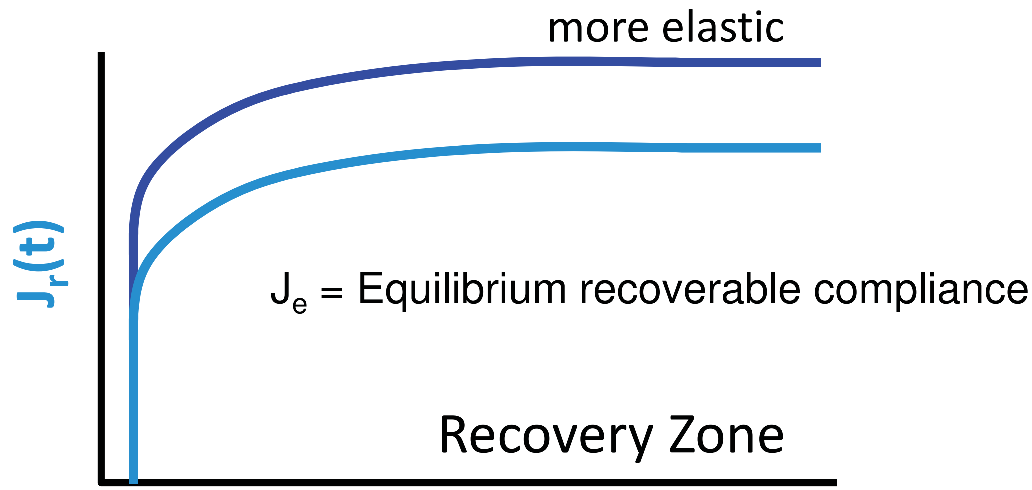
Mark, J., et. al., Physical Properties of Polymers, American Chemical Society, 1984, p. 102.

Creep Recovery Experiment



Creep Recovery : Creep and Recoverable Compliance





Creep Compliance

$$J(t) = \frac{\gamma(t)}{\sigma}$$

The material property obtained from Creep experiments:
Compliance = 1/Modulus (in a sense)

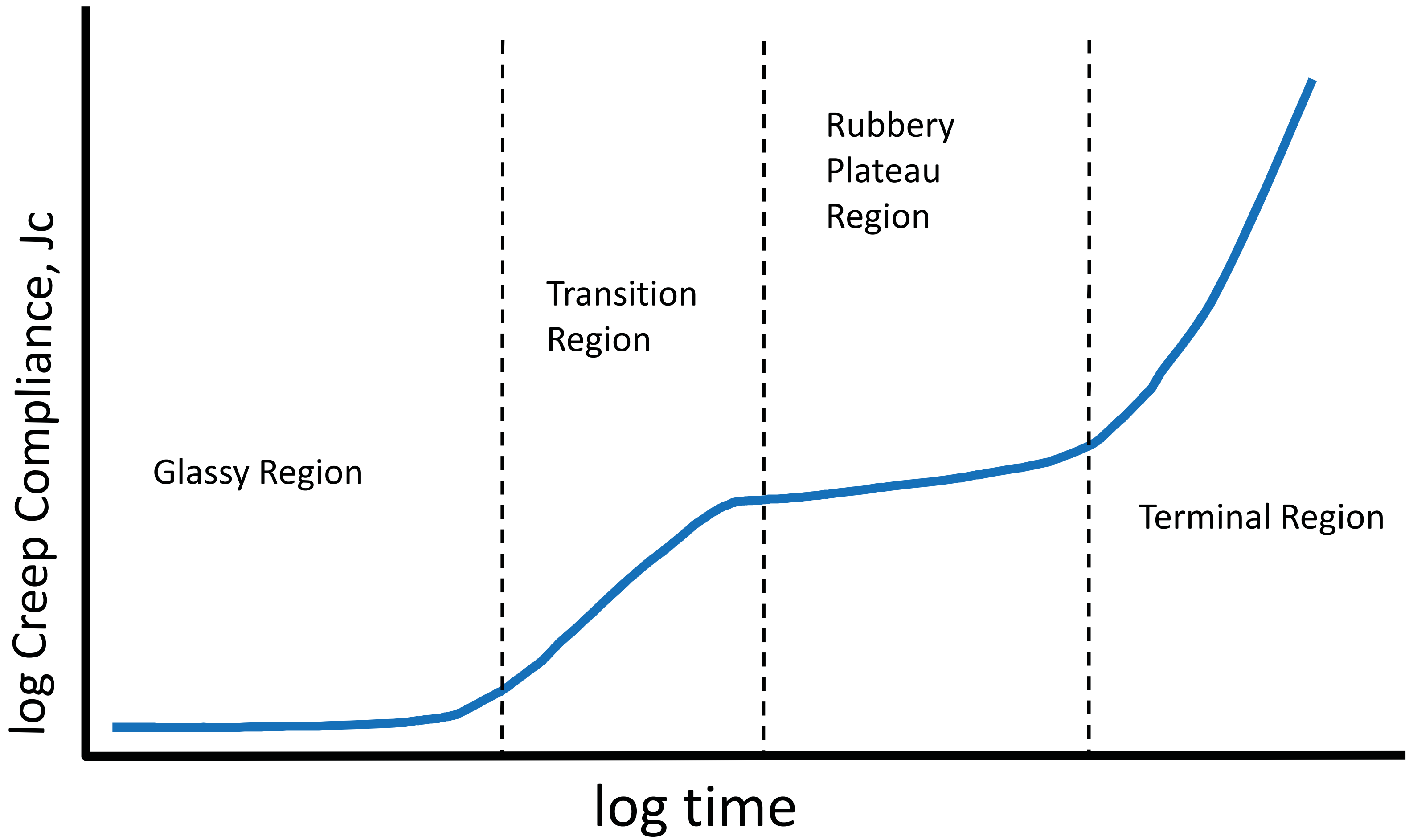
Recoverable Compliance

$$J_r(t) = \frac{[\gamma_u - \gamma(t)]}{\sigma}$$

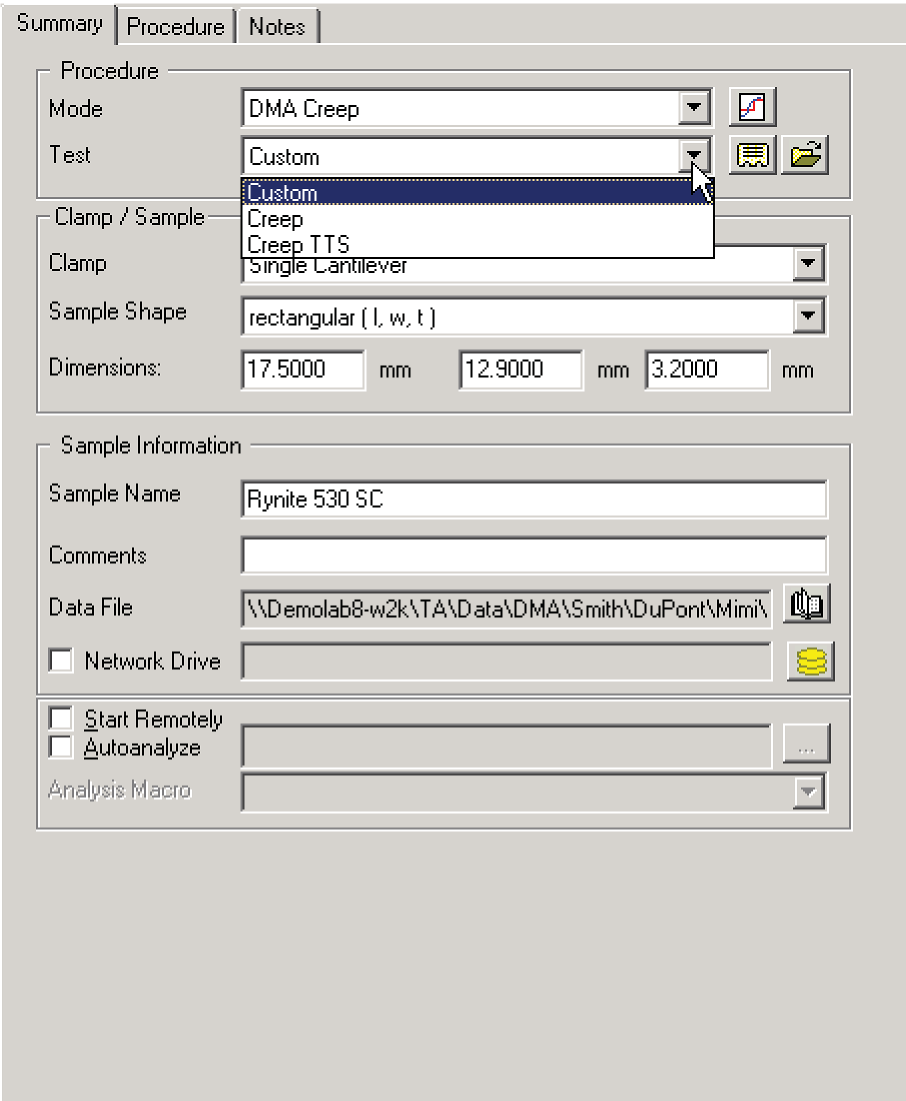
Where γ_u = Strain at unloading
$\gamma(t)$ = time dependent recoverable strain

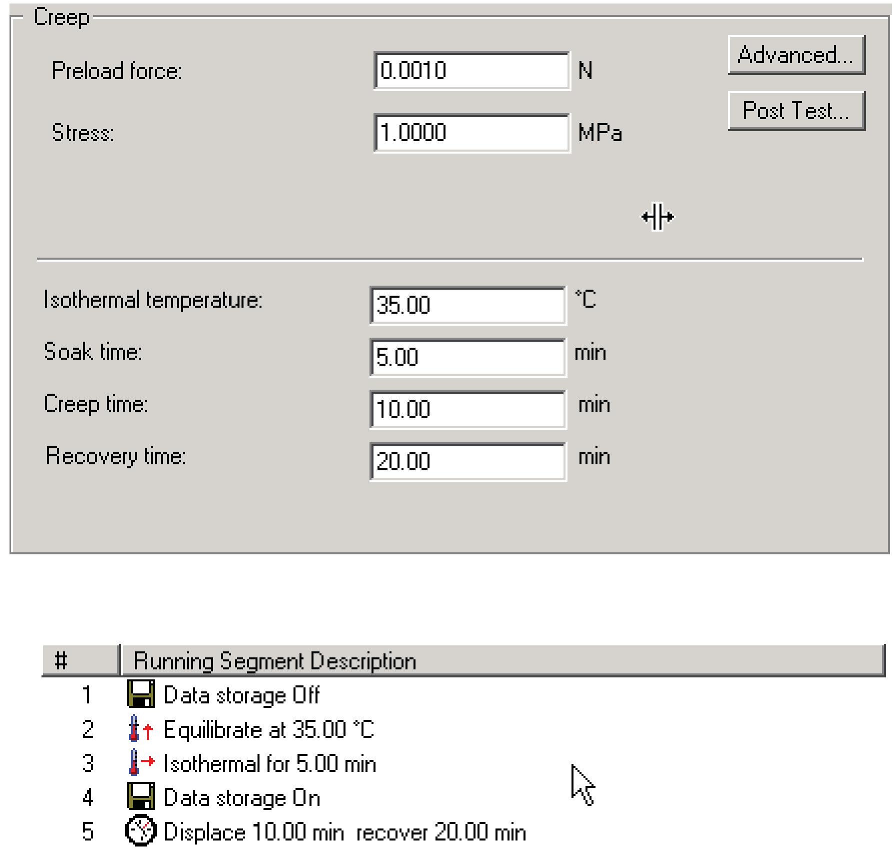
Mark, J., et. al., Physical Properties of Polymers, American Chemical Society, 1984, p. 102.

Creep: Material Response



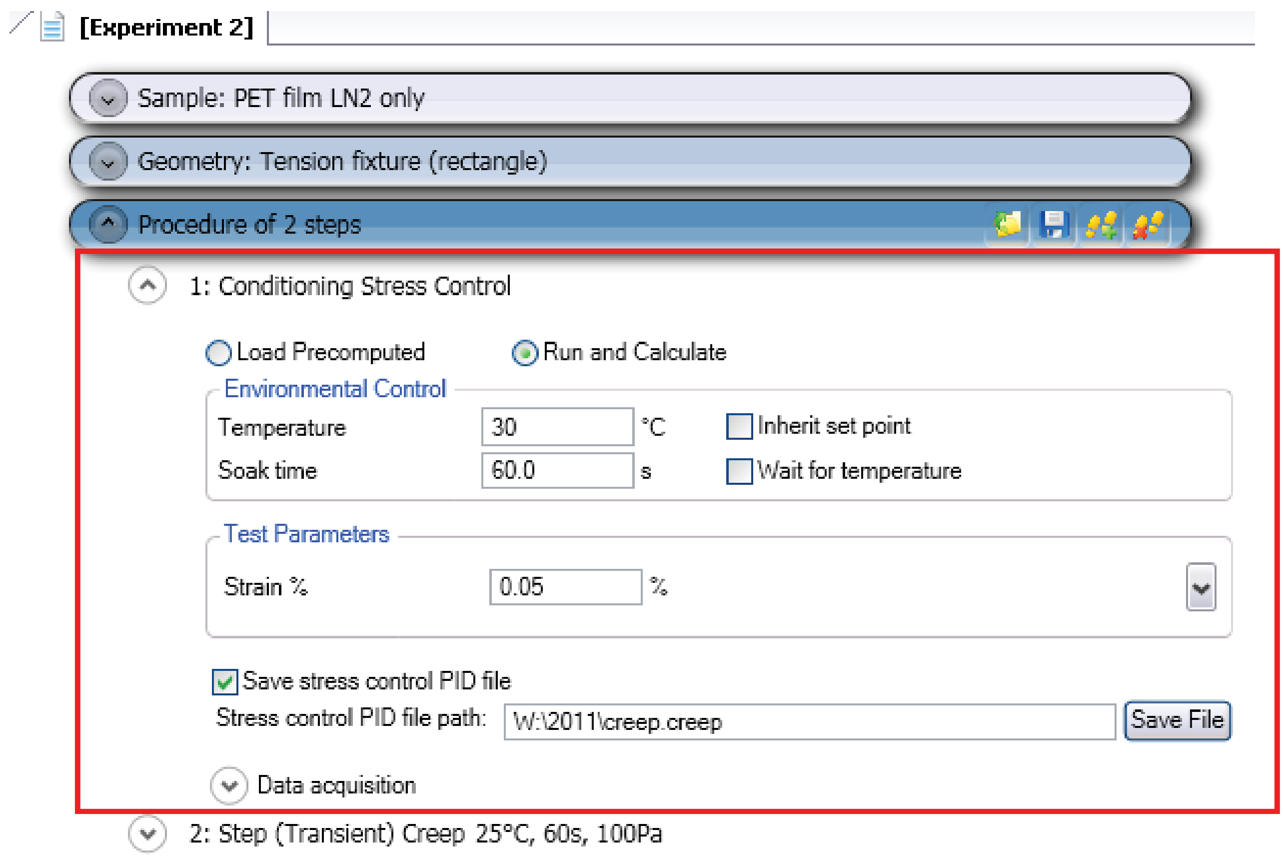
Programming Creep on a Q800





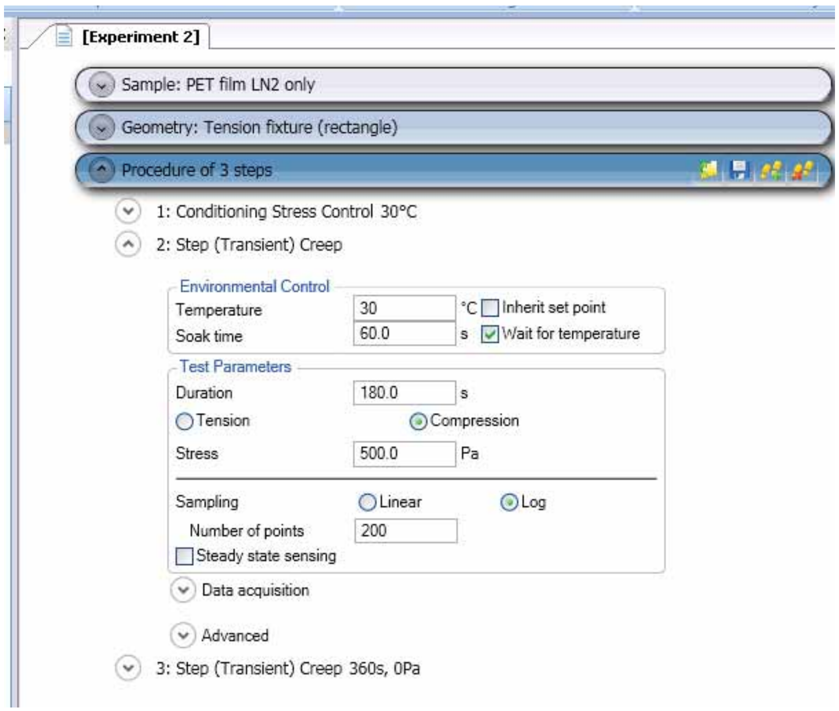
Programming Creep on a RSA-G2

- Set up a pre-test and get the sample information into the loop
- Stress Control Pre-test: frequency sweep within LVR

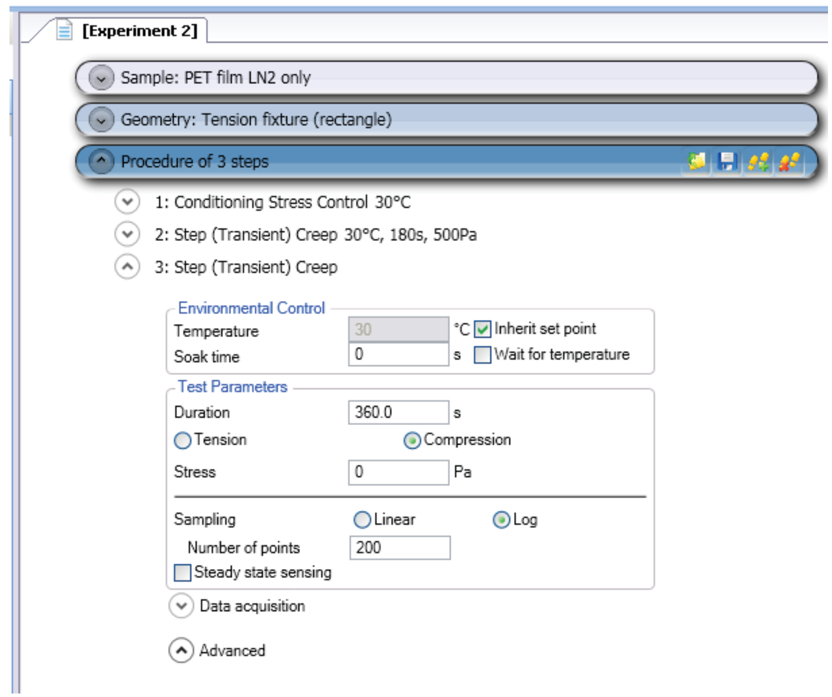


Programming Creep on a RSA-G2

Creep

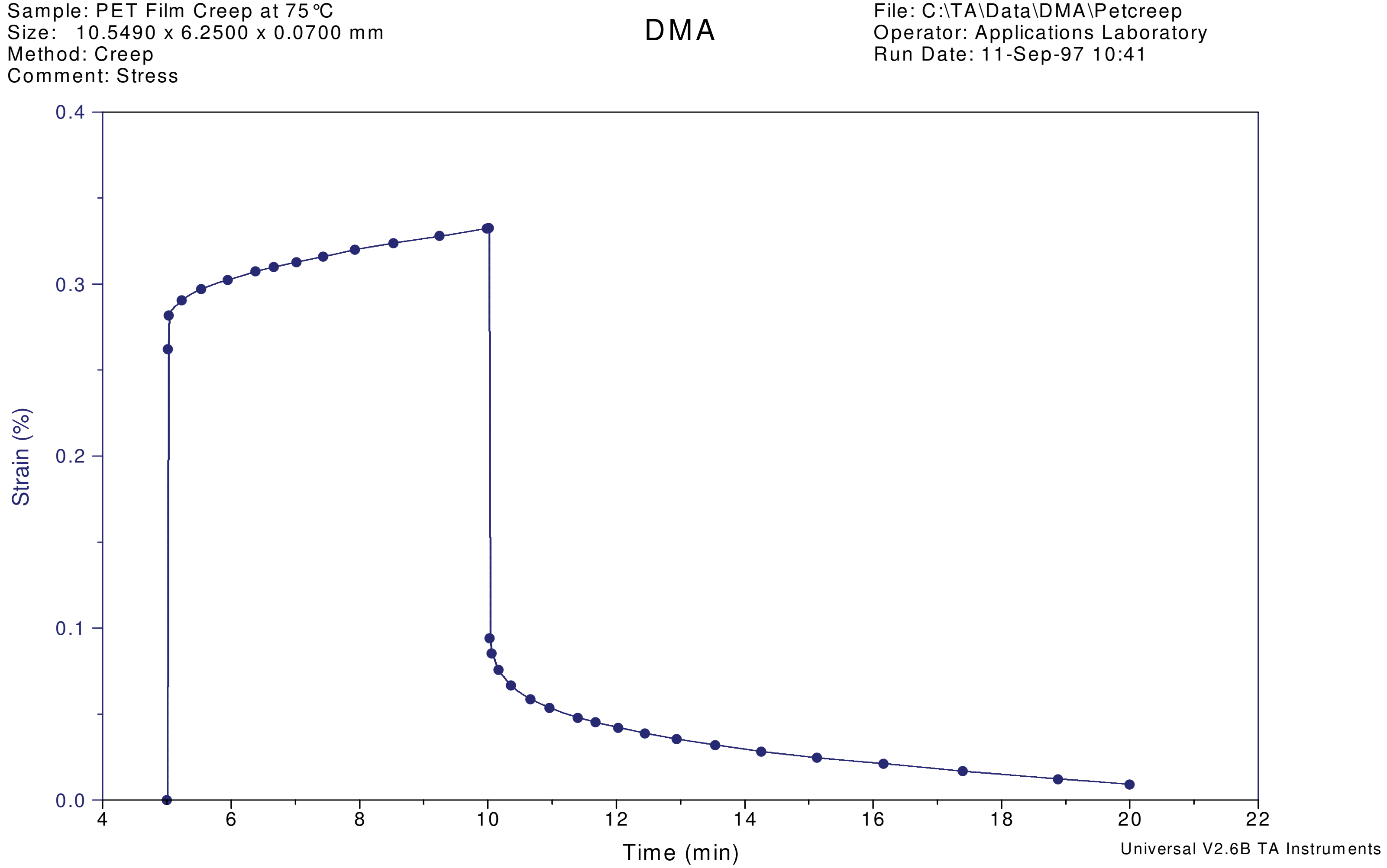


Recovery

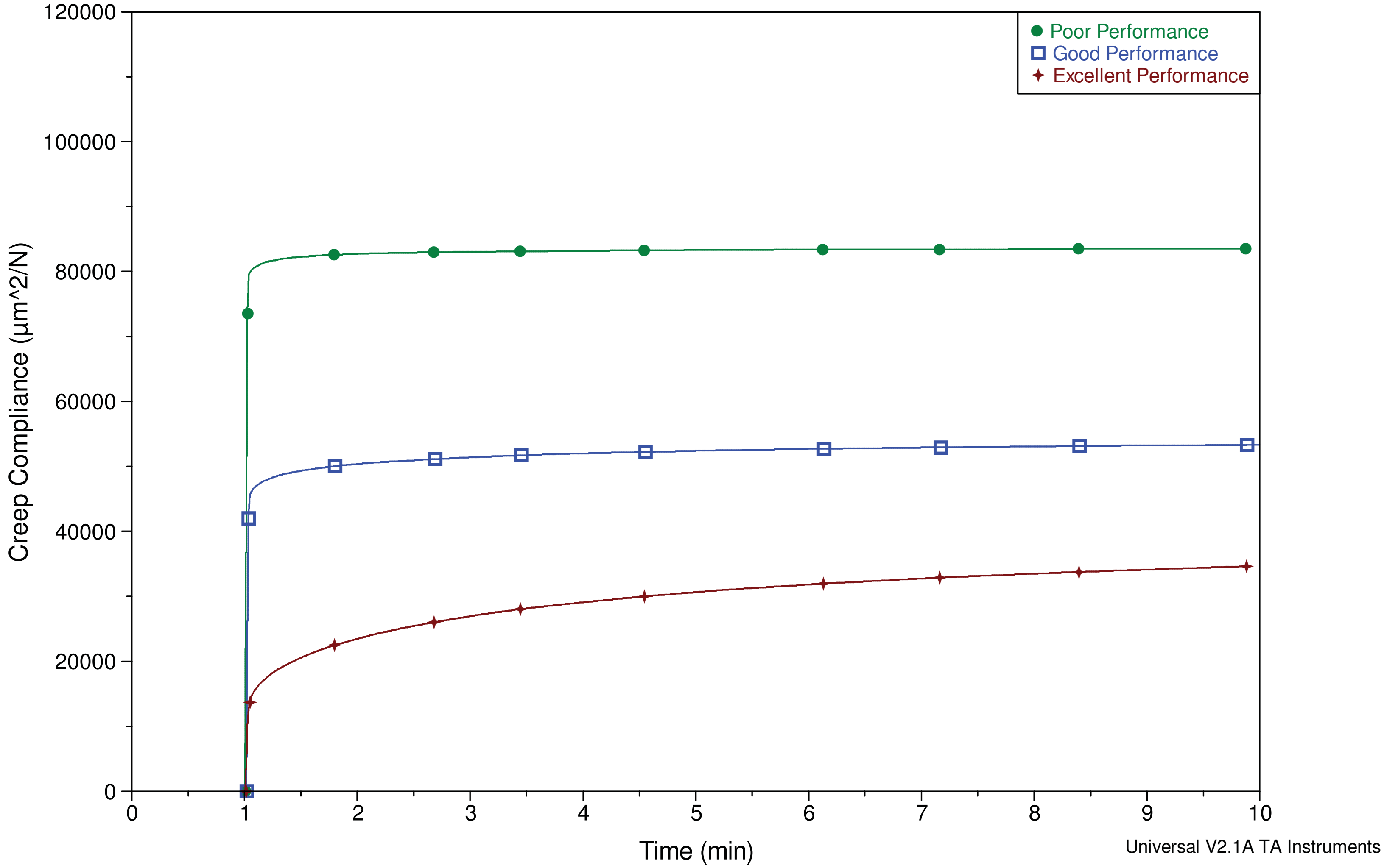


- Stress: needs to be in the linear region
- Creep time: until it reach steady state
- Recovery time: until the compliance and strain reach plateau

DMA Q800: Creep Test on PET Film

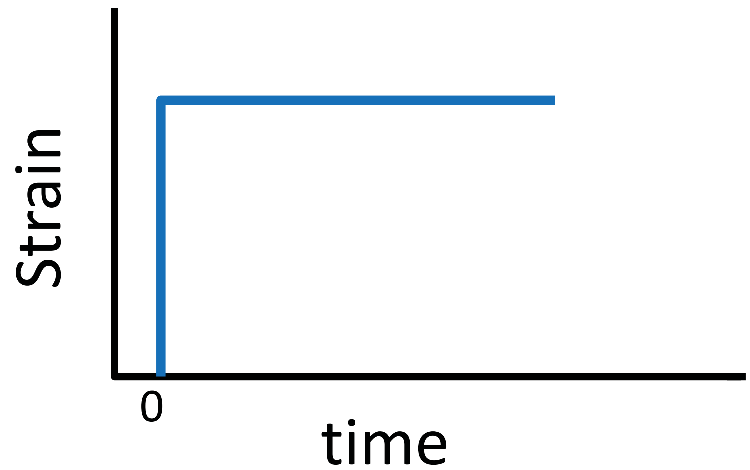


Creep on Packaging Films used in Thermoforming



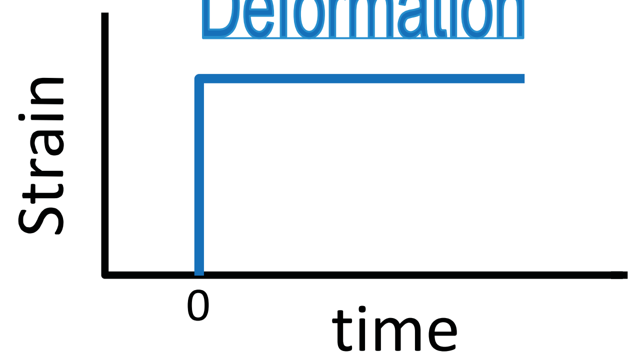
Stress Relaxation Experiment

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



Stress Relaxation Experiment

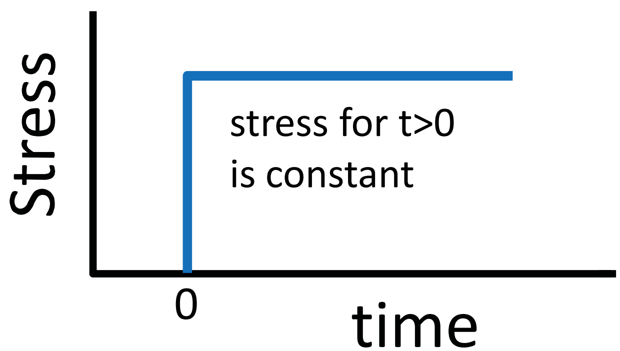
Deformation



Response of Classical Extremes

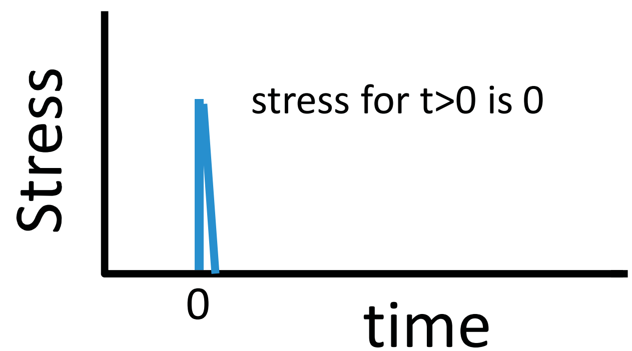
Elastic

Hookean Solid



Viscous

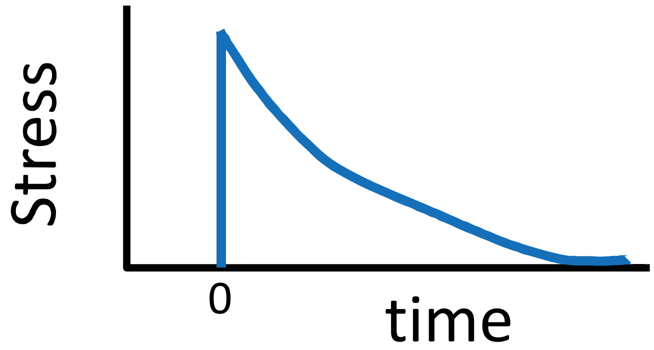
Newtonian Fluid



Stress Relaxation Experiment

Response of ViscoElastic Material

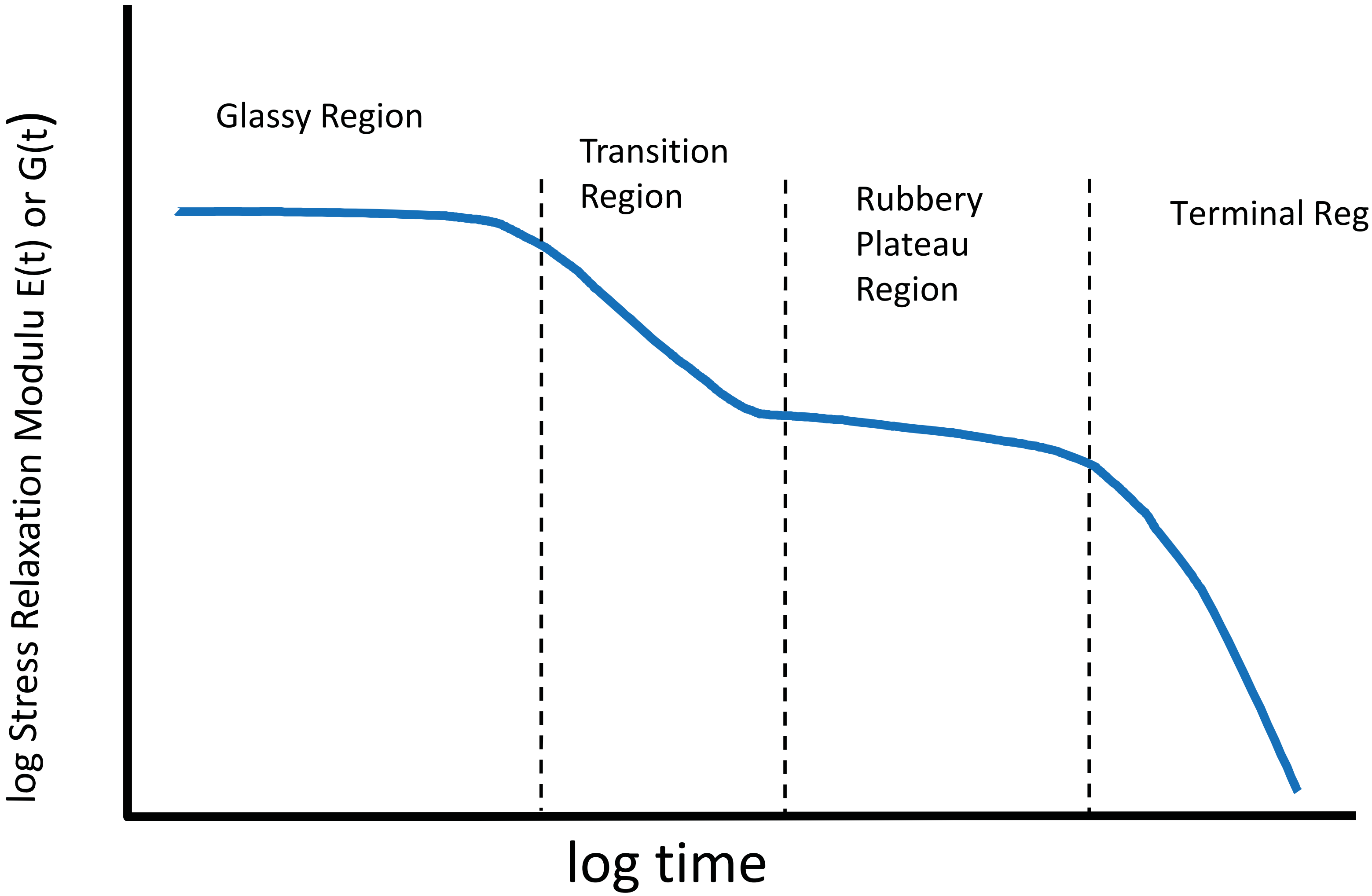
Stress decreases with time starting at some high value and decreasing to zero.



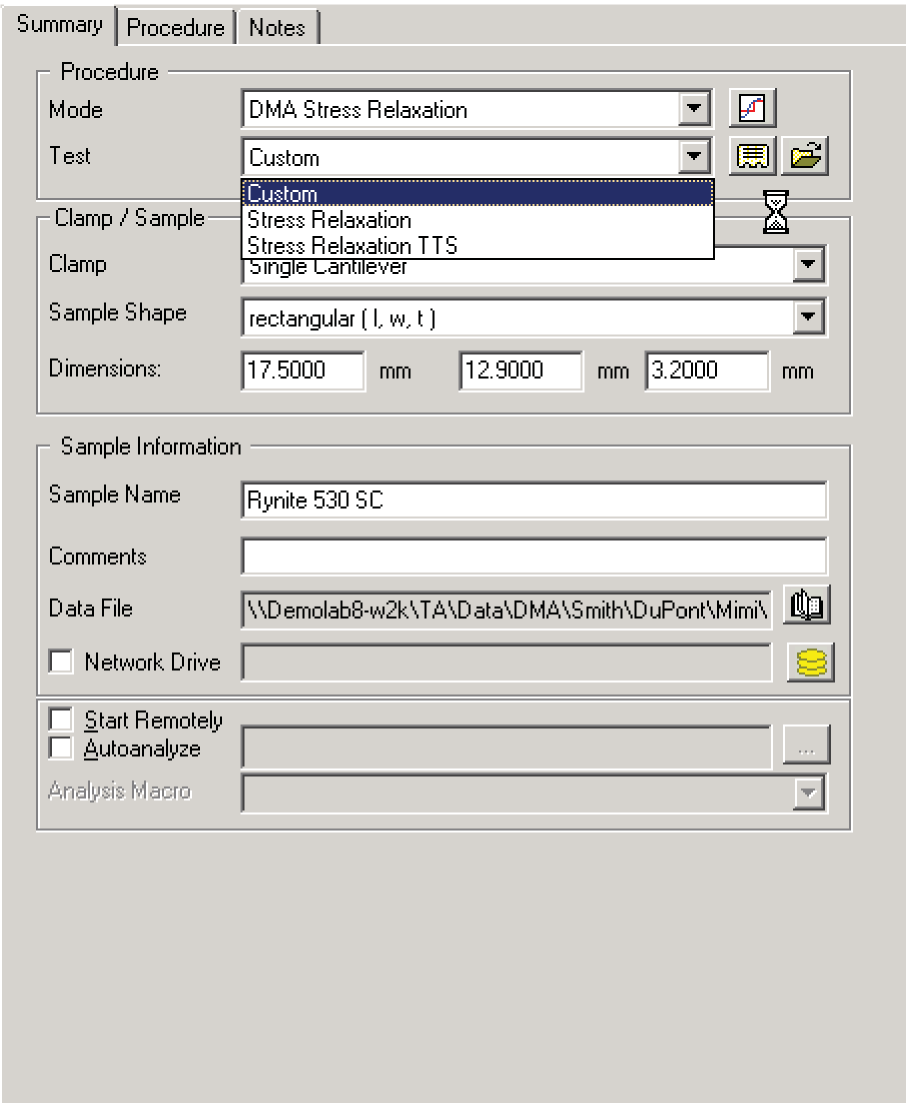
- For small deformations (strains within the linear region) the ratio of stress to strain is a function of time only.
- This function is a material property known as the STRESS RELAXATION MODULUS, G(t)

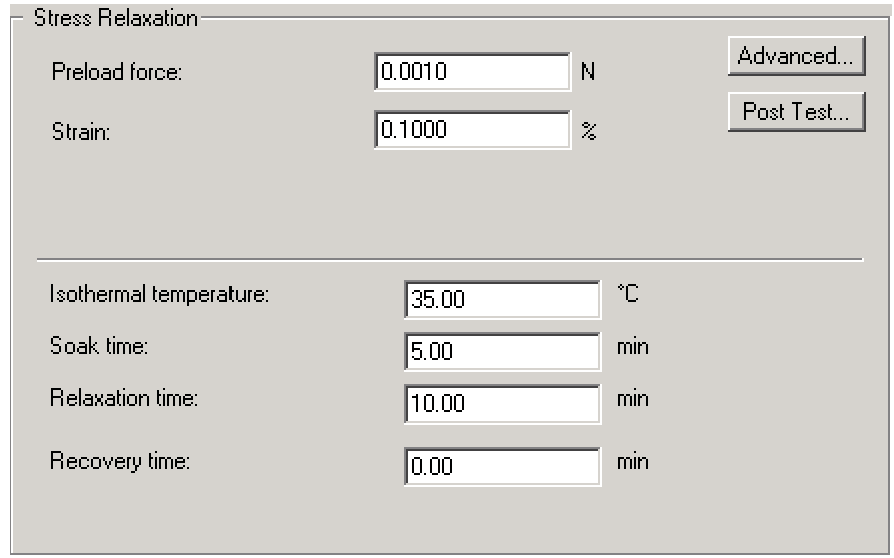
$$G(t) = \sigma(t)/\gamma$$

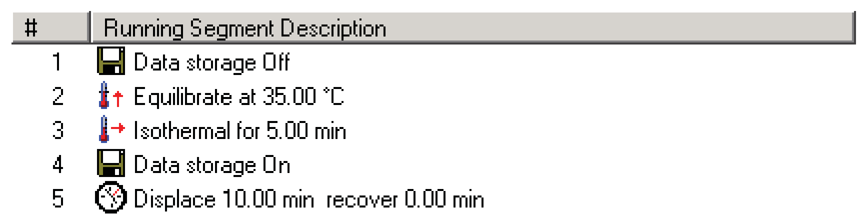
Stress Relaxation: Material Response



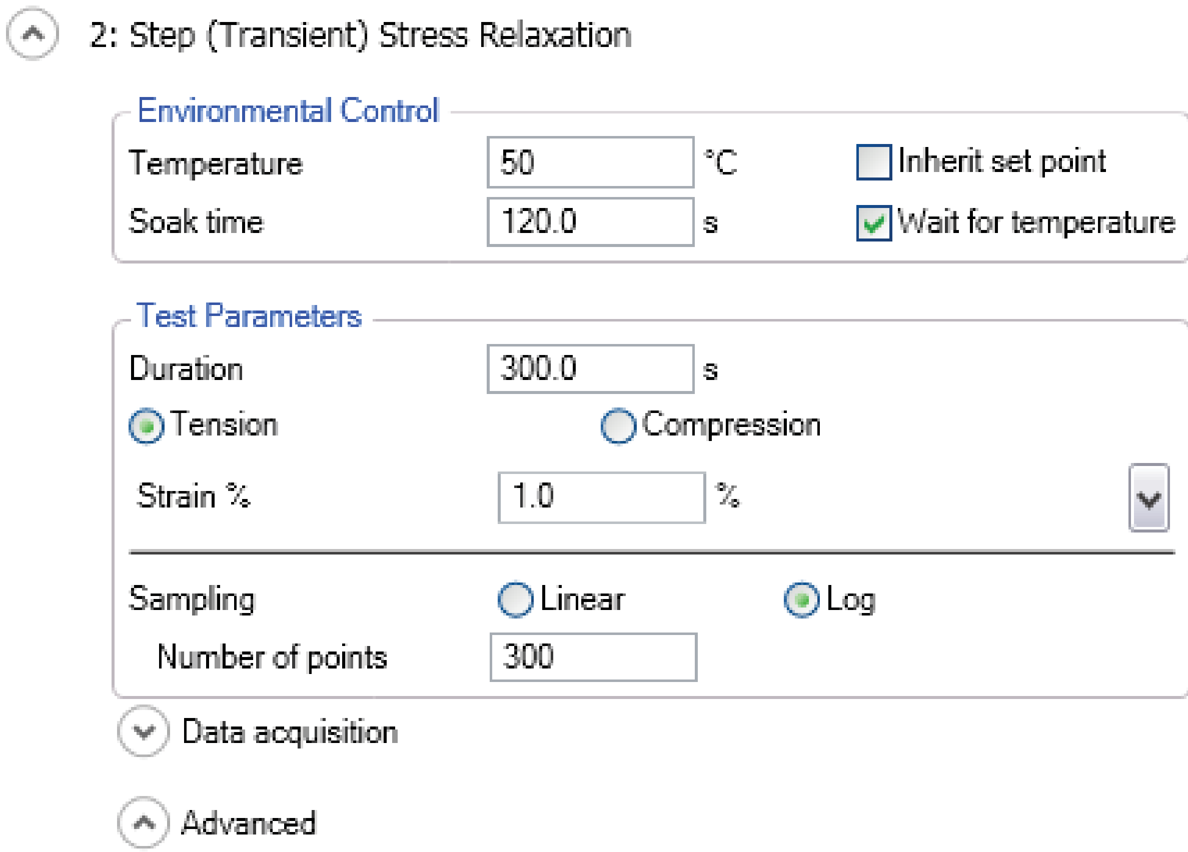
Programming Stress Relaxation on a Q800





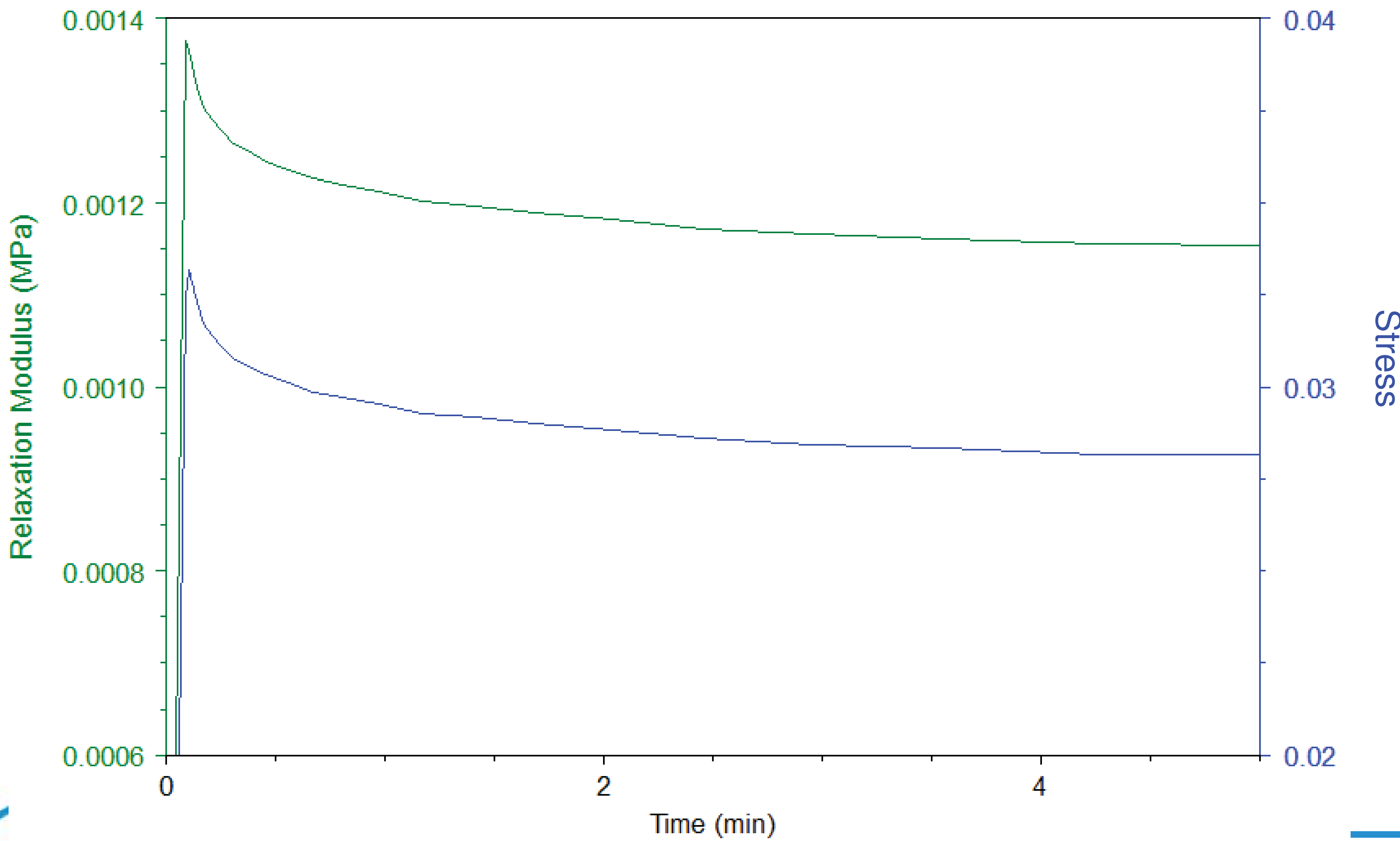


Programming Stress Relaxation on a RSA-G2

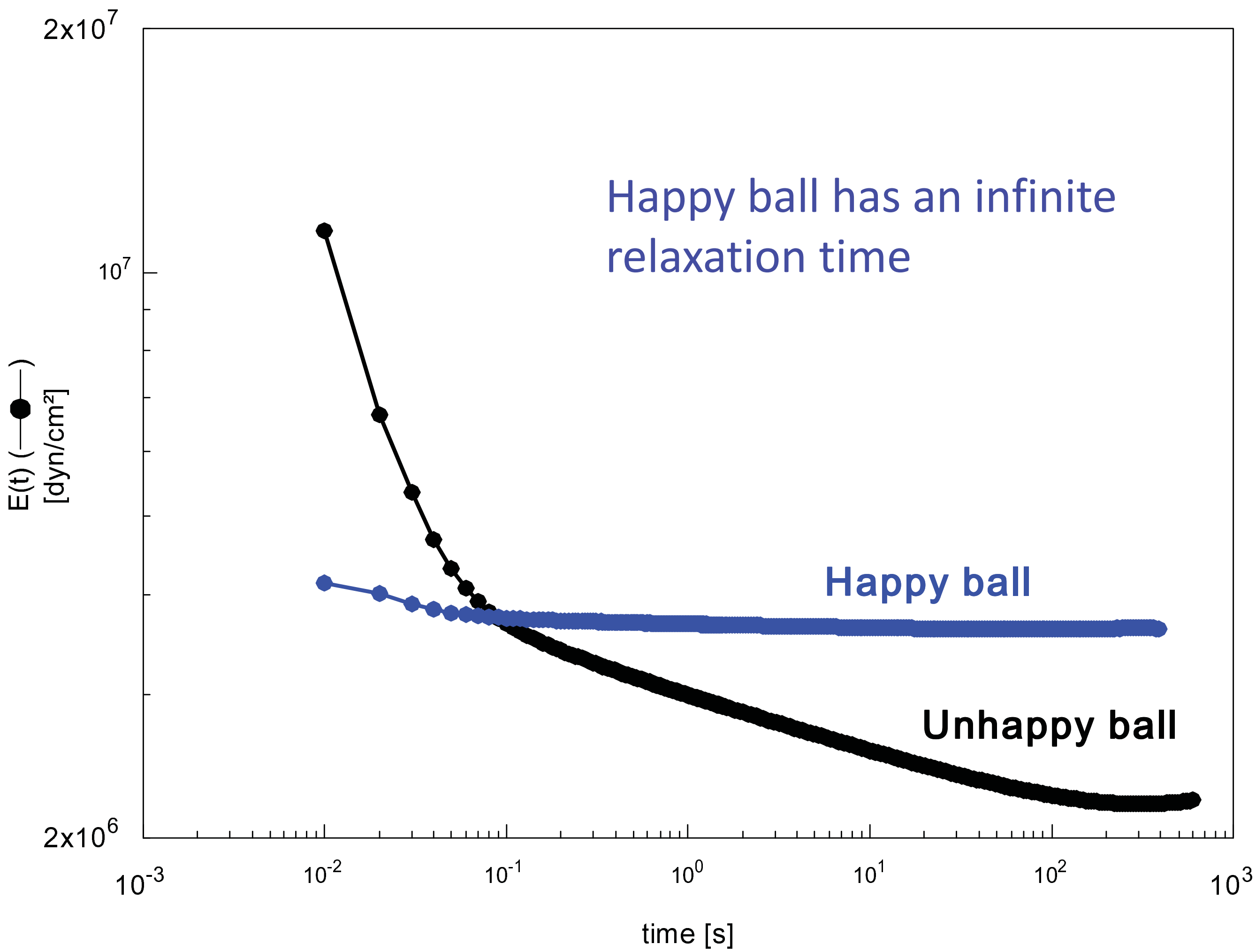


Stress Relaxation on Silicone Foam

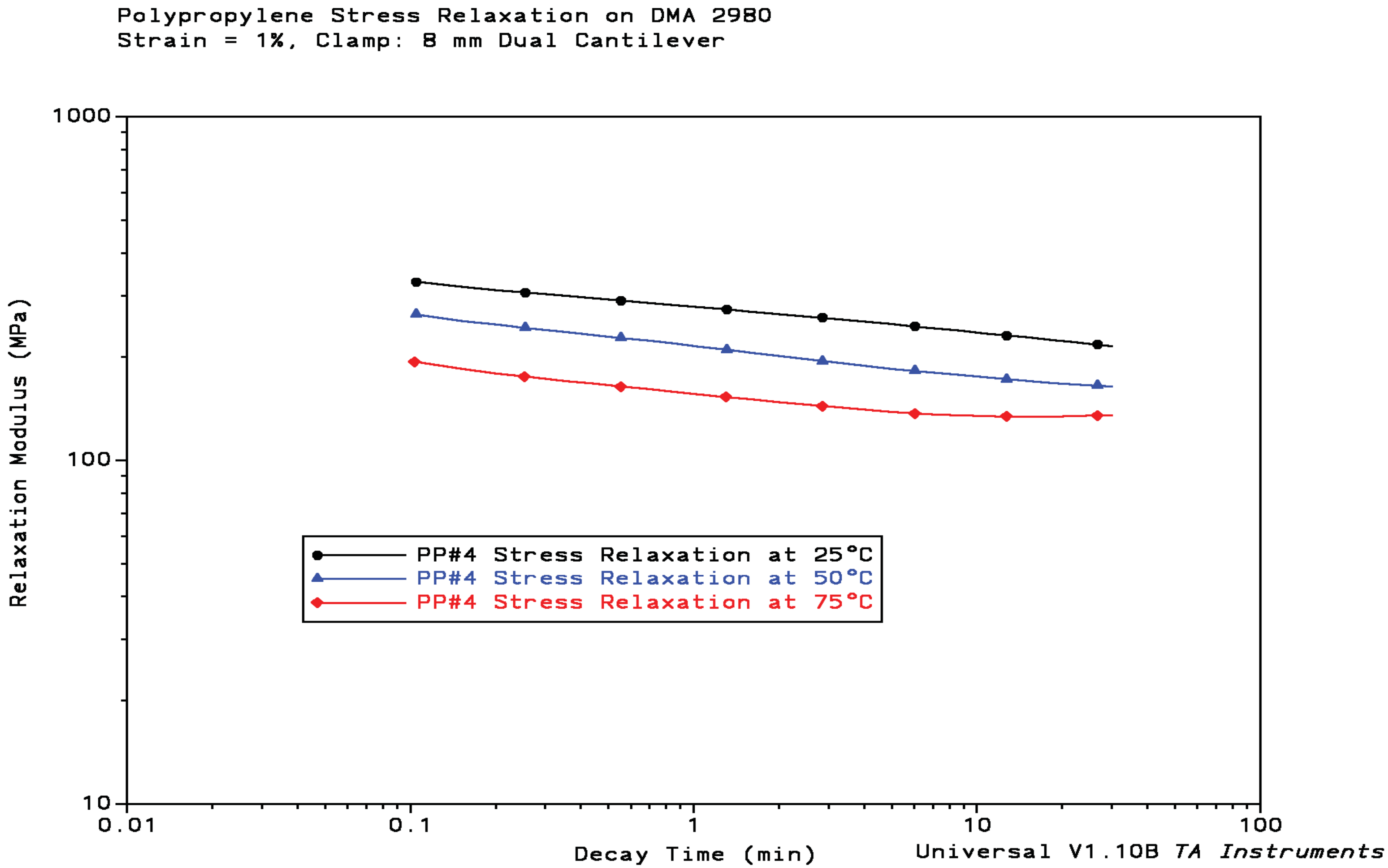
- Strain of 1% is maintained while stress is measured
  - Strain must be in LVR



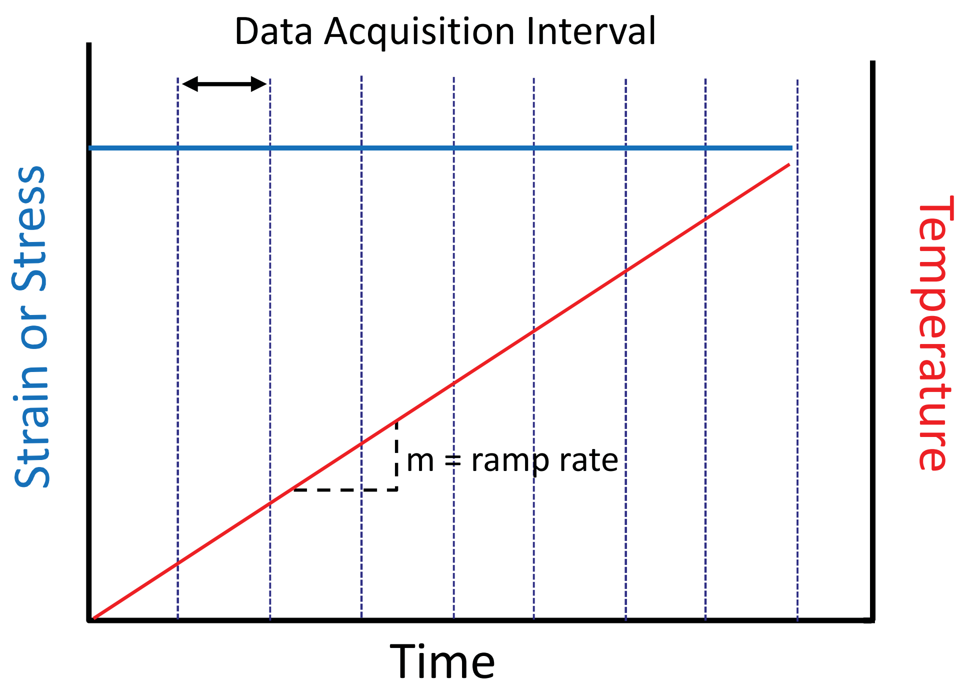
Stress Relaxation Happy and Unhappy Balls



DMA: Stress Relaxation on Polypropylene



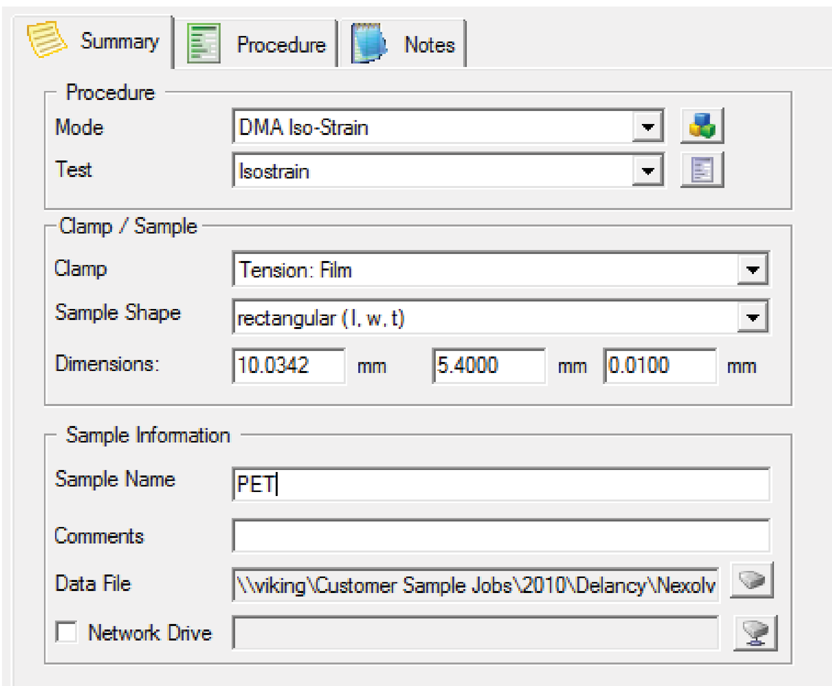
Iso-strain/Iso-stress Temperature Ramp

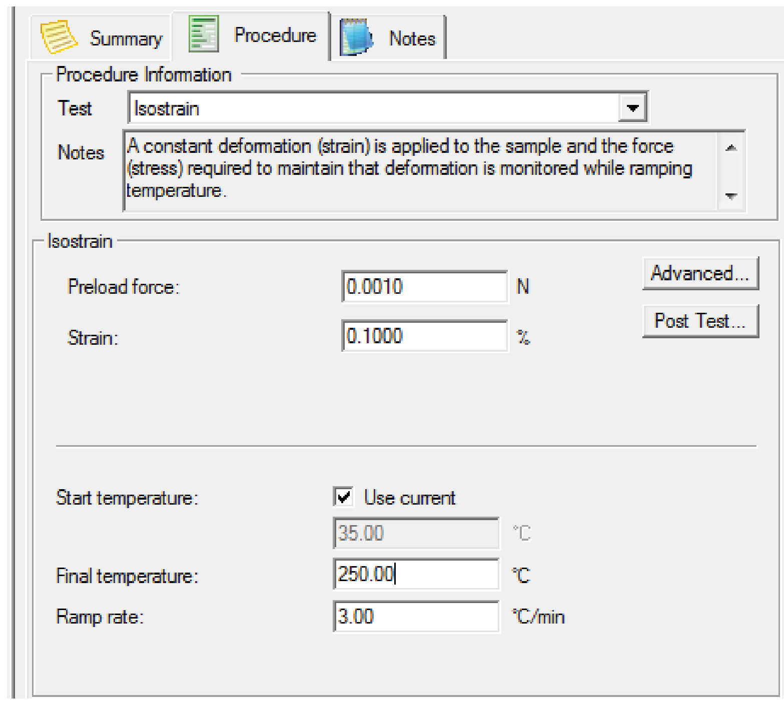


- The strain or stress is held at a constant value and a linear heating rate is applied.
- Valuable for assessing mechanical behavior under conditions of confined or fixed load (stress) or deformation (strain).
- Example: Measure sample shrinkage (length shrinkage or shrinking force)

Programming Iso-strain Testing on a Q800

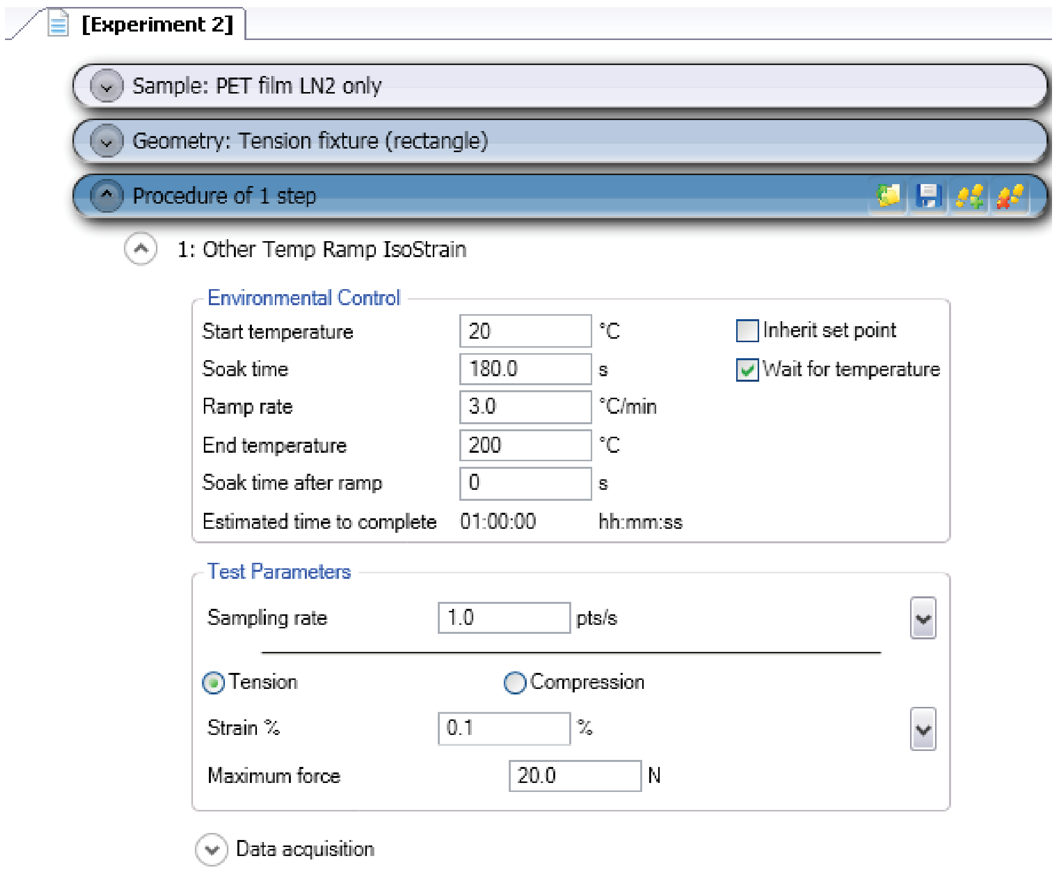
- Hold strain constant and measure sample shrinking force



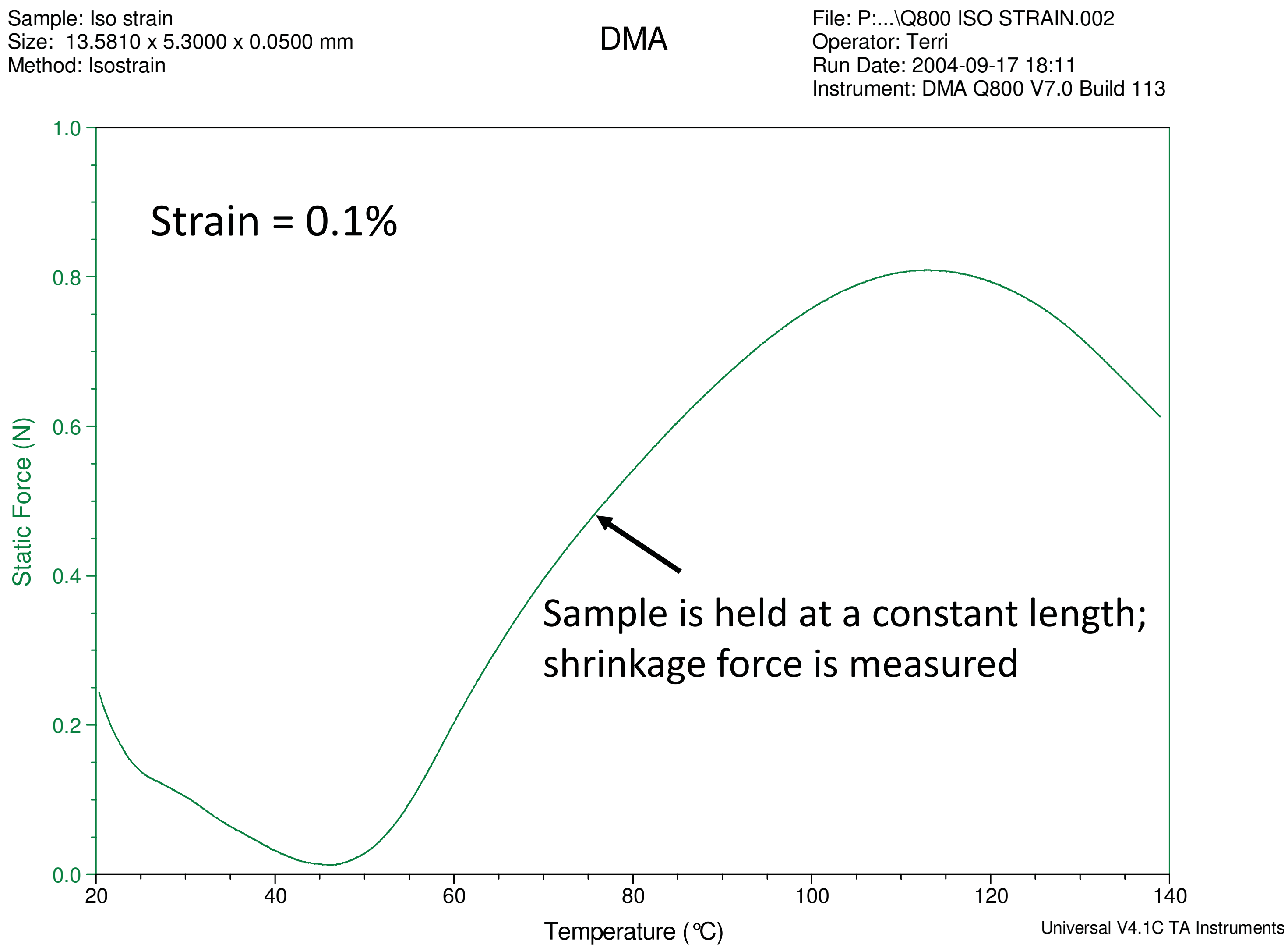


Programming Iso-strain Testing on a RSA-G2

- Hold strain constant and measure sample shrinking force

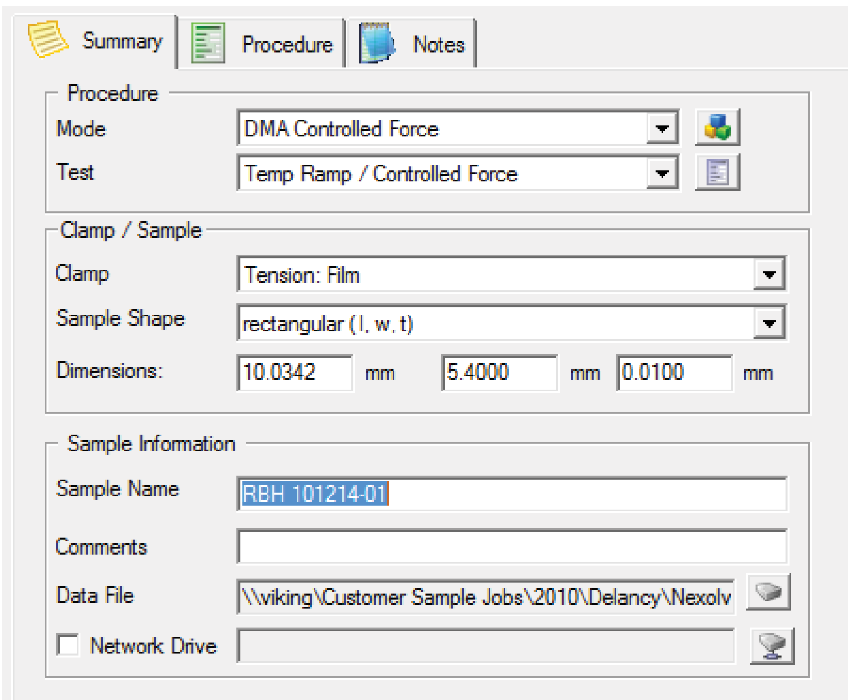


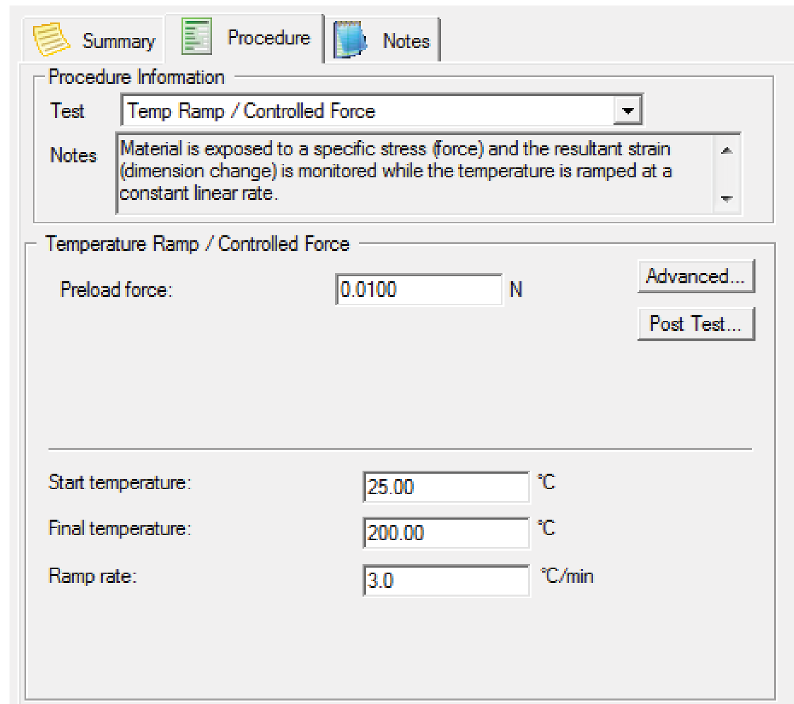
PVC Based Film - IsoStrain



Programming Iso-force Testing on a Q800

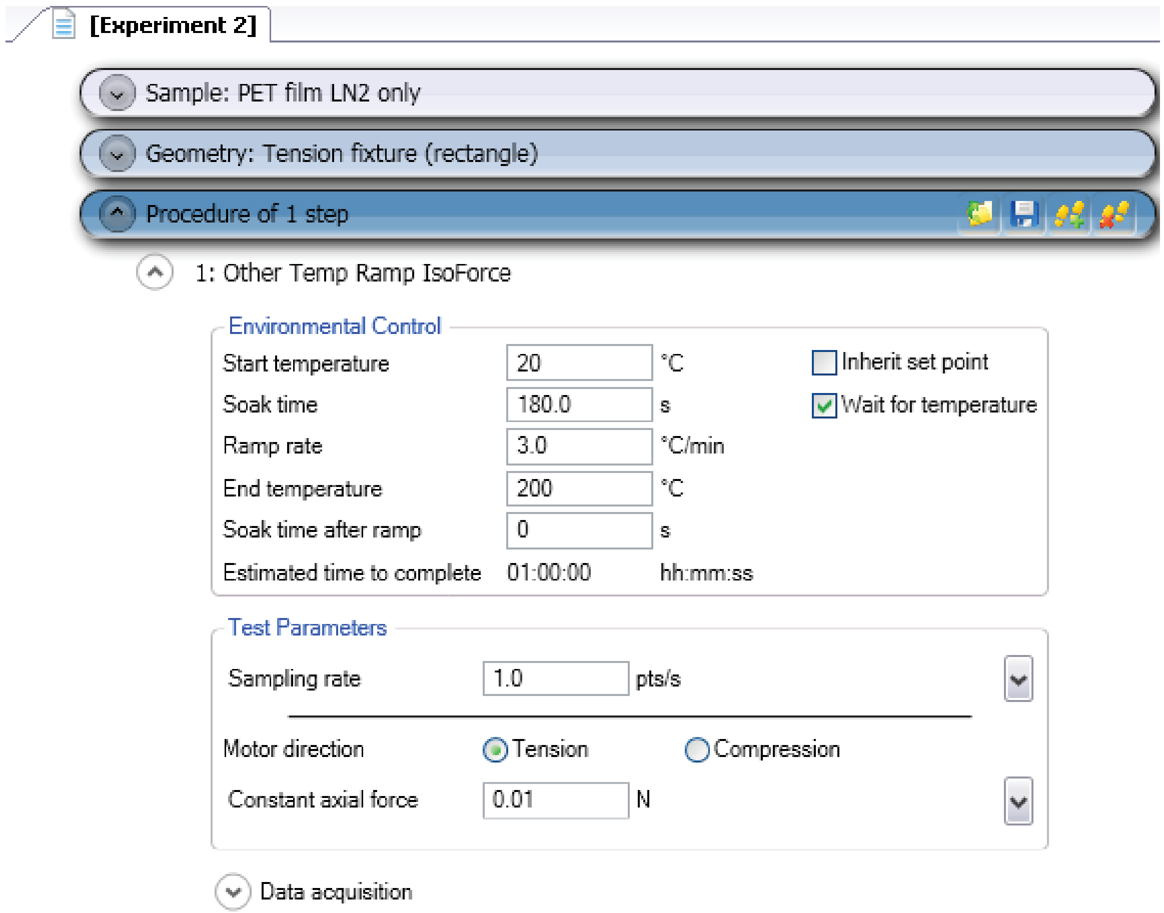
- Hold force constant and measure sample length shrinkage



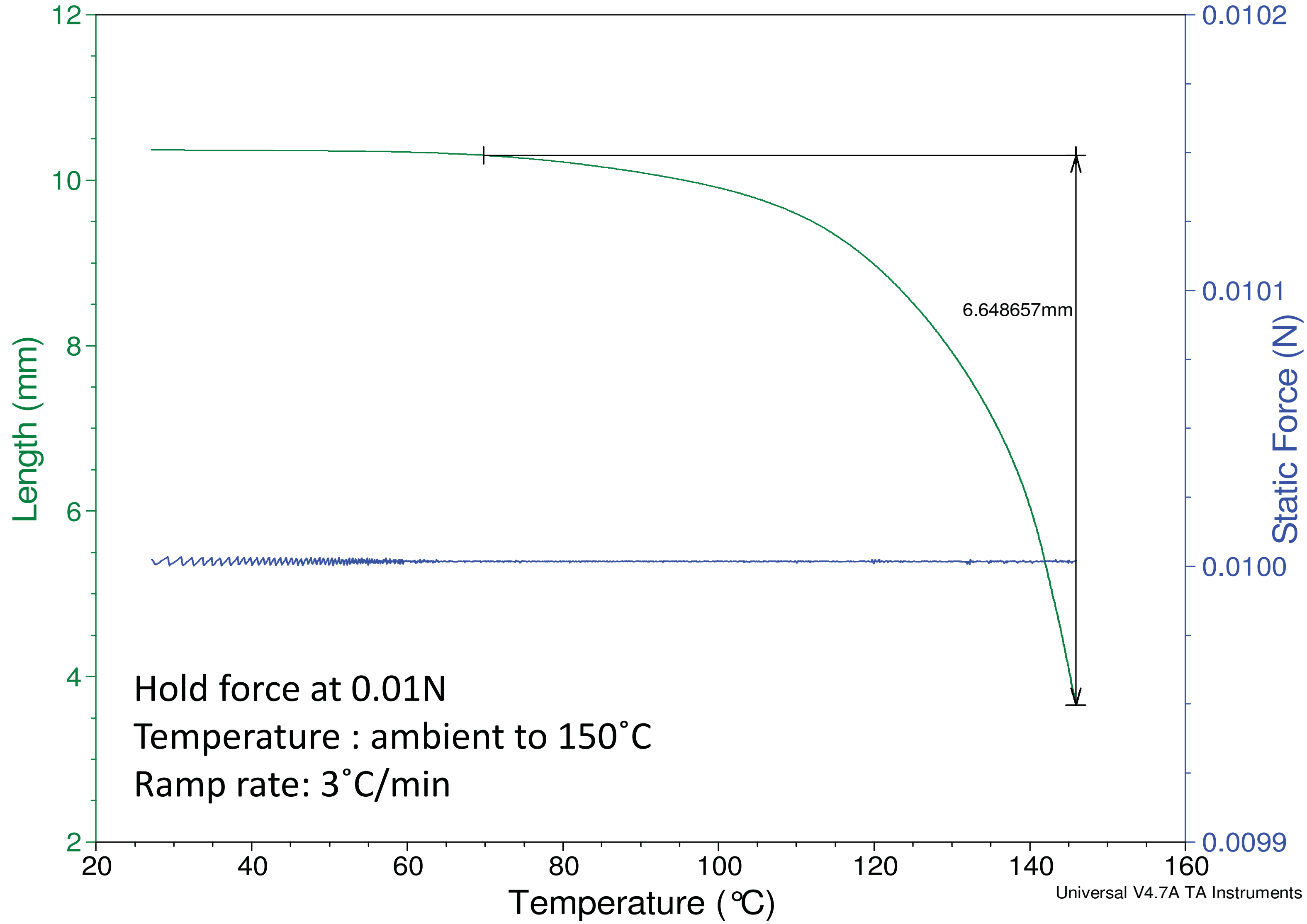


Programming Iso-force Testing on a RSA-G2

- Hold force constant and measure sample length shrinkage

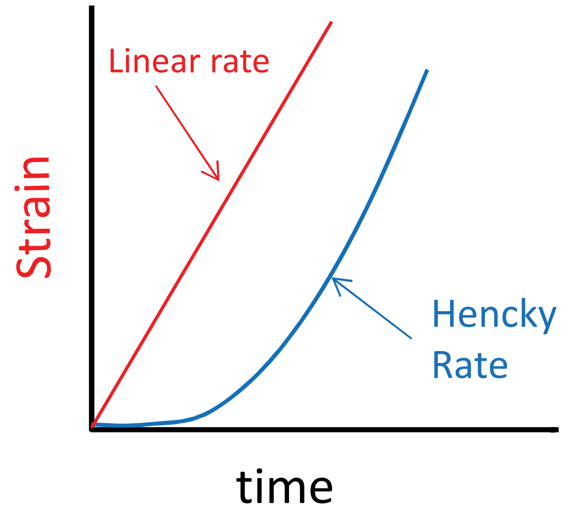


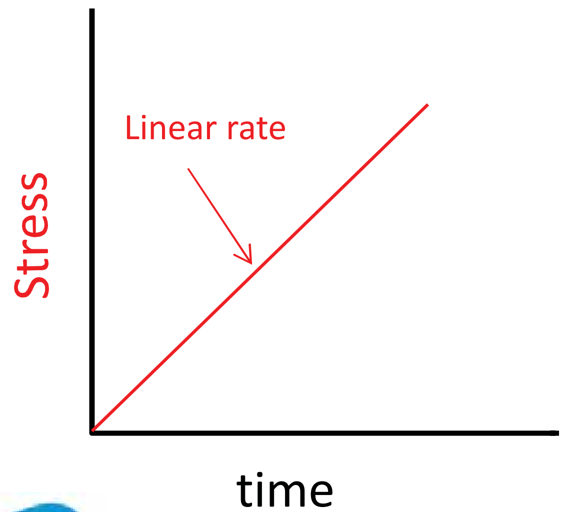
Iso-force Temperature Ramp: measure shrinkage



Stress-Strain Testing

Axial Test: Strain Rate Controlled

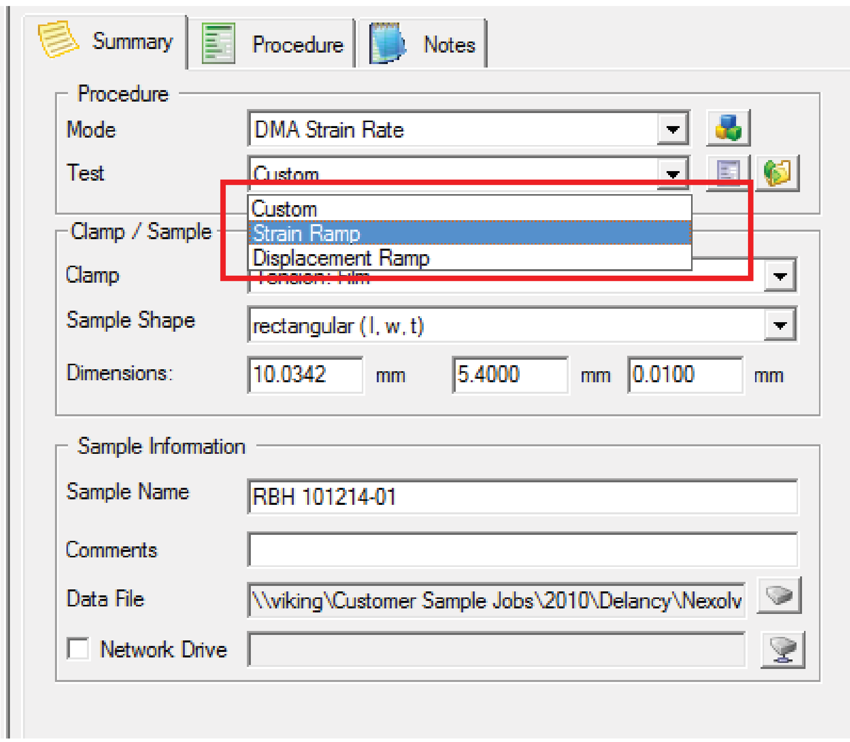


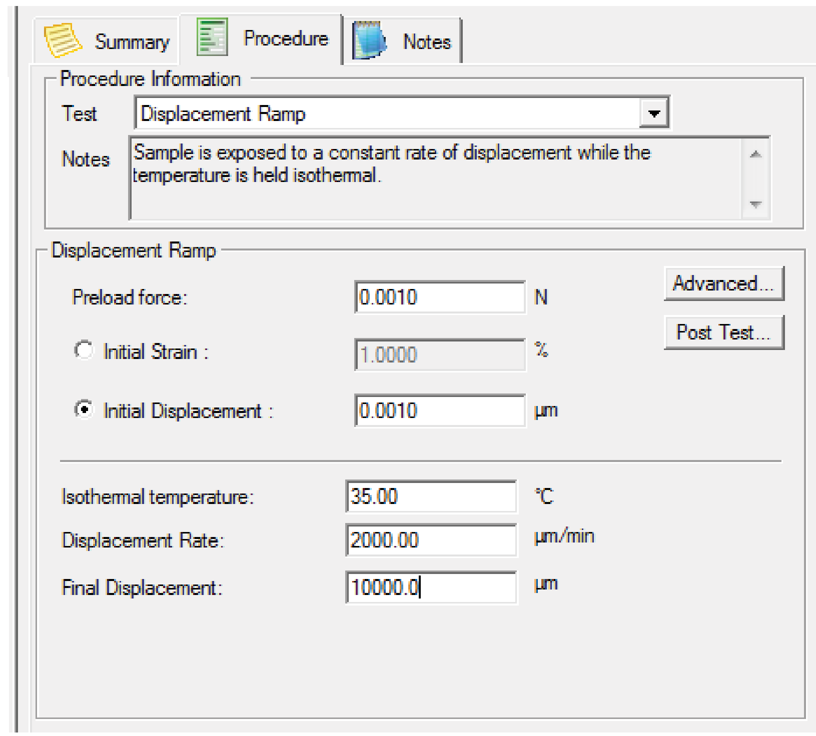


- Sample is deformed under a constant linear strain rate, Hencky strain rate, force, or stress for generating more traditional stress-strain curves.

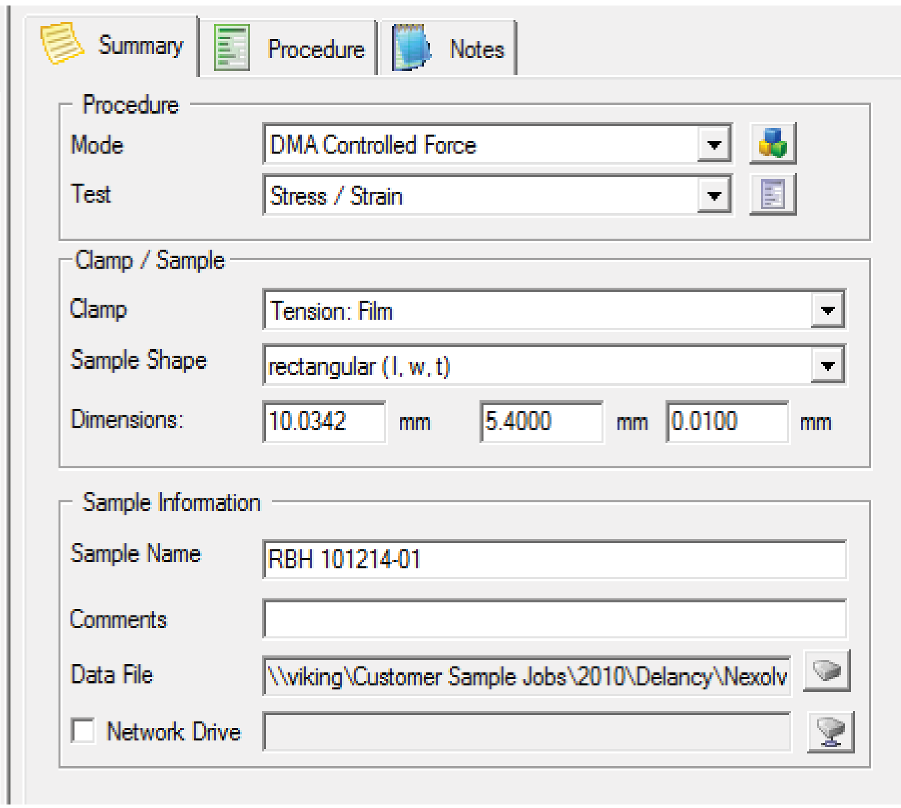
- Measure sample's Young's modulus, yield stress, strain hardening effect and sample fracture

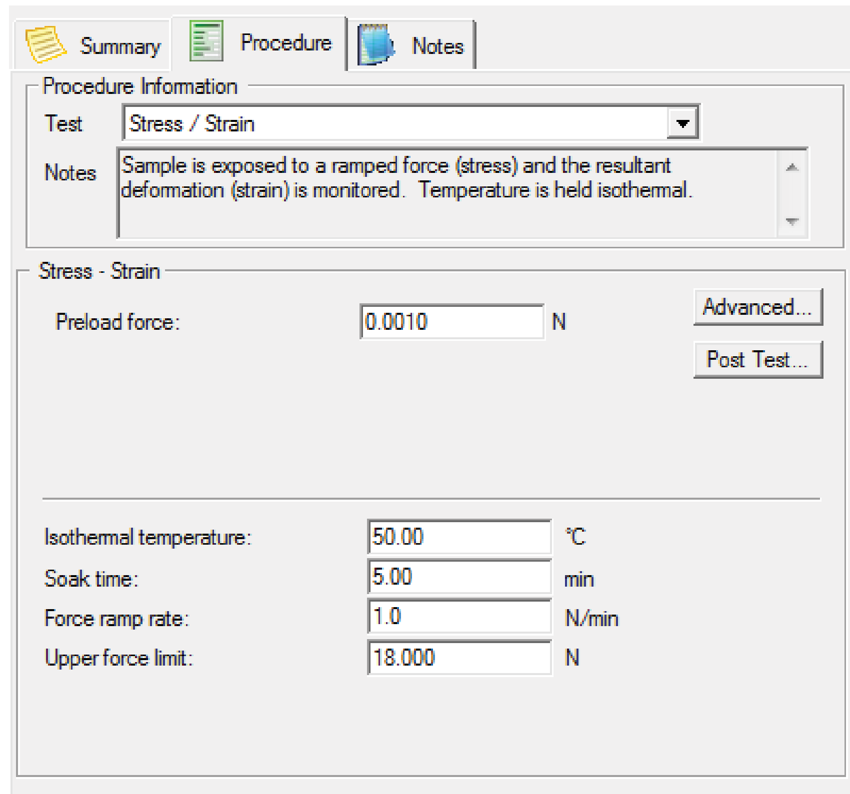
Programming Strain Ramp Testing on a Q800





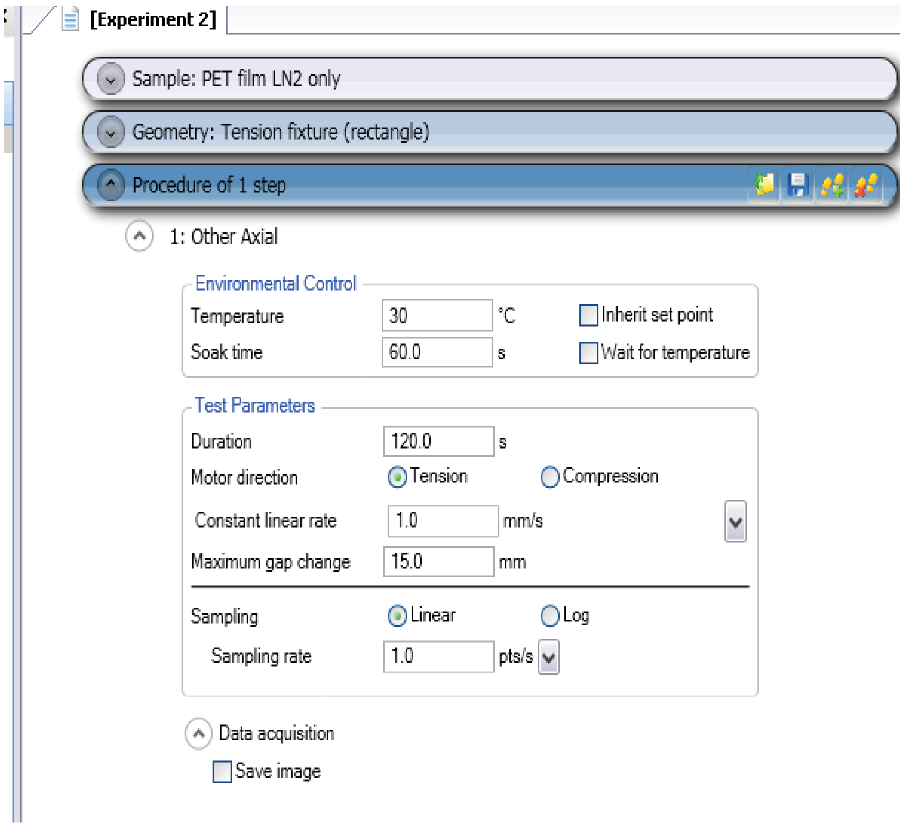
Programming Stress Ramp Testing on a Q800



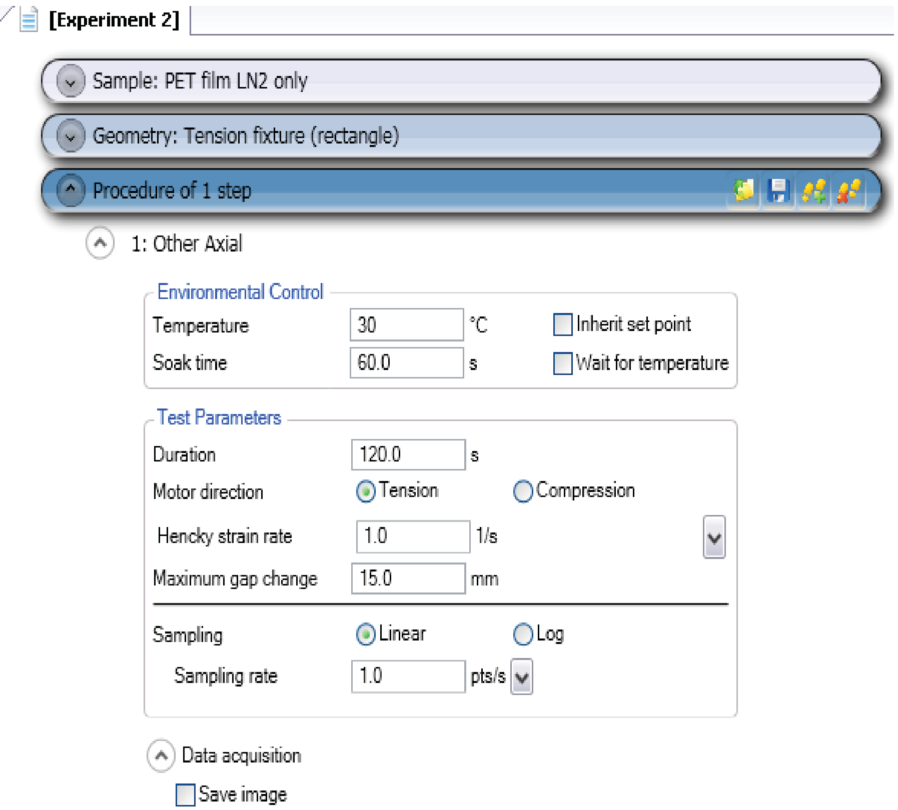


Programming Strain Ramp Testing on a RSA G2

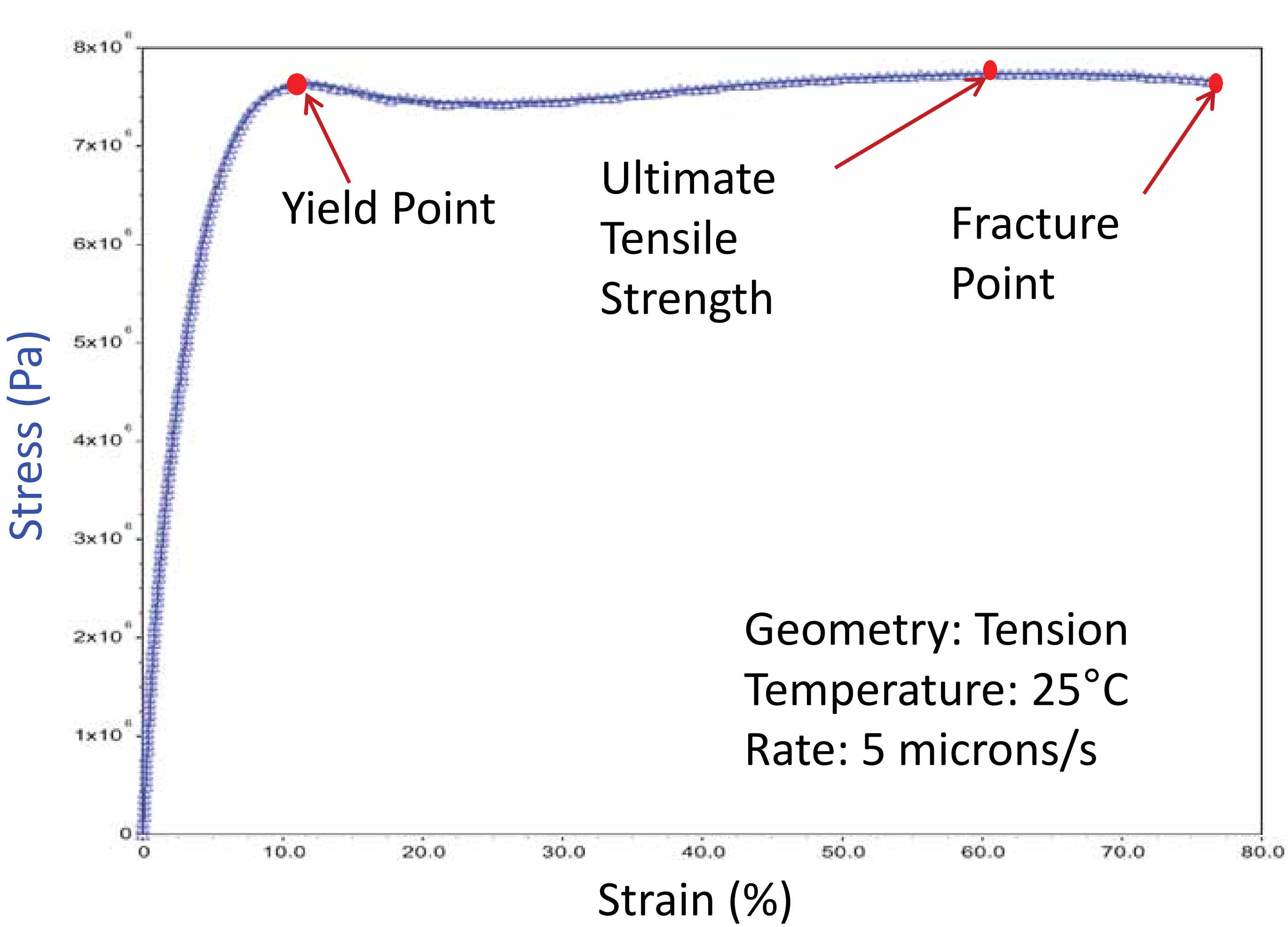
- Linear strain rate

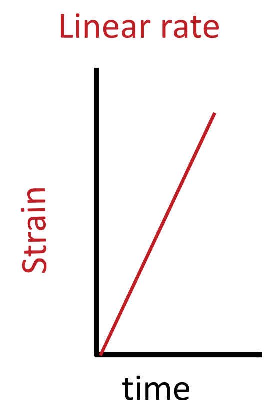


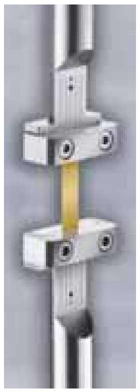
- Hencky strain rate



Polyethylene Bag Linear Rate Sweep







Dynamic Mechanical Analysis

Applications

Applications of DMA

- Understanding the Glass Transition
- Measuring Transitions in DMA
  - Viscoelastic properties and the Glass Transition
  - Testing considerations
  - Secondary Transitions
- Material properties and the Glass Transition
  - Molecular Structure, Composition, Environmental Effects
- Other Applications

The Glass Transition

- A transition over a ***range of temperature*** from a glassy state to a rubber state in an amorphous material
- Mechanical:
  - Below the Glass Transition, the material is in a brittle, glassy state, with a modulus of 10^9 Pa.
  - Above the Glass Transition, the material becomes soft and flexible, and the modulus decreases to 10^6 Pa.
- Molecular:
  - Below the Glass Transition, polymer chains are locked in place, without sufficient thermal energy to overcome the barrier for rotational or translational motion.
  - At temperatures above the Glass Transition, there is molecular mobility, and chains can slide past each other

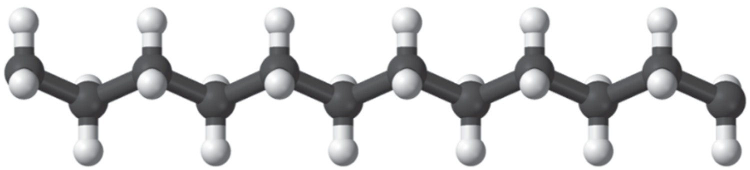
The Glass Transition

- "The glass transition is associated with the onset of long-range cooperative segmental mobility in the amorphous phase, in either an amorphous or semi-crystalline polymer."
- Any factor that affects segmental mobility will affect T_g, including…
  - the nature of the *moving segment*,
  - chain stiffness or steric hindrance
  - the free volume available for segmental motion

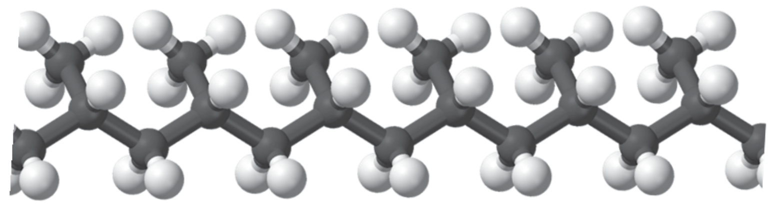
Turi, Edith, A, Thermal Characterization of Polymeric Materials, Second Edition, Volume I., Academic Press, Brooklyn, New York, P. 508.

Chemical Composition of Polymers

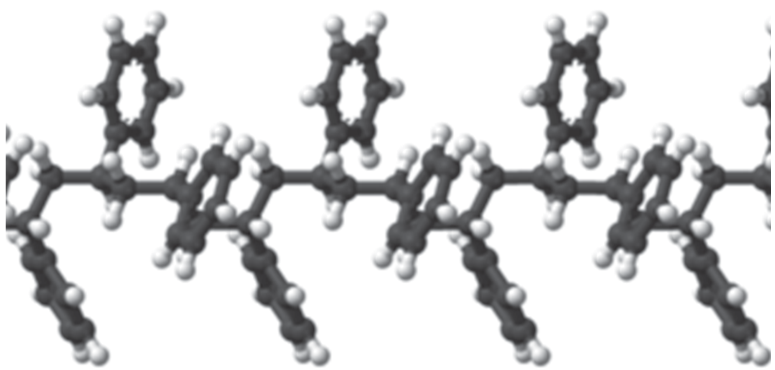
- Polymers are long chains of repeating units (monomers)
- The chemical composition determines mechanical properties, and the temperature where transitions occur



Polyethylene T_g = -128° C



Polypropylene T_g = -20° C



Polystyrene T_g = 100 ° C

"Introduction to Polymer Viscoelasticity," 2nd Edition. Aklonis, John J; MacKnight, William J. Wiley-Interscience

Dynamic Mechanical Analysis of Tg

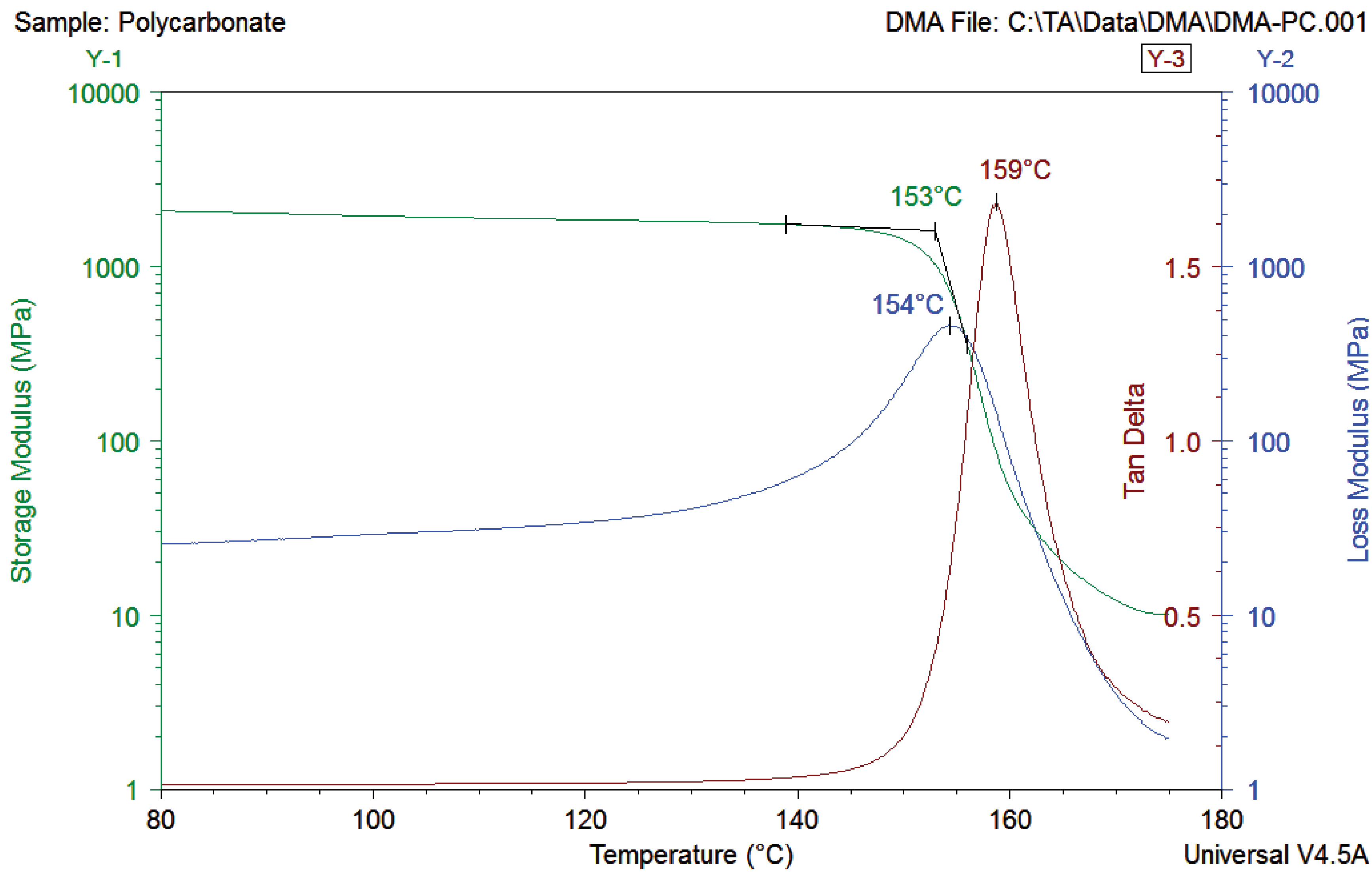
- DMA provides a higher sensitivity to Tg than DSC, because it directly measures changes in mechanical and viscoelastic properties as a function of temperature.
- Materials whose glass transitions cannot be resolved by DSC can often be measured easily in DMA
  - Semi-crystalline materials with low amorphous content
  - Composites in which the polymer weight fraction is small
  - Glass Transitions that occur over a wide range, or overlap with other thermal events
- Because DMA is based on mechanical properties, the Glass Transition measurement is particularly relevant to characterizing materials for their end-use properties

Glass Transition E' Onset, E" Peak, and Tan δ Peak

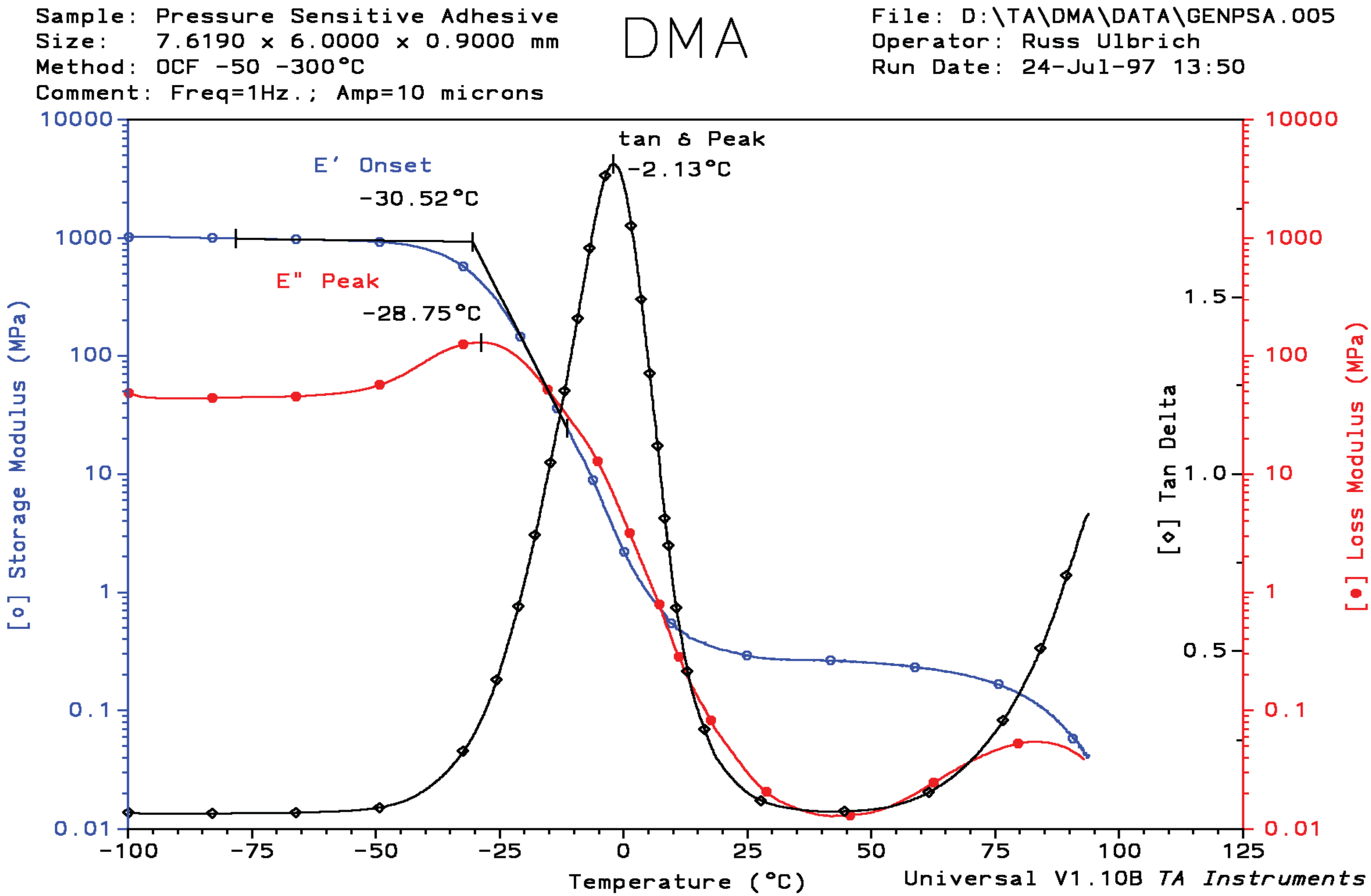
- **Storage Modulus E' Onset:**
  - Occurs at lowest temperature, relates to mechanical failure
- **Loss Modulus E" Peak:**
  - Occurs at middle temperature
  - Related to the physical property changes
  - Reflects molecular processes - the temperature at the onset of segmental motion
- **Tan δ Peak:**
  - 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: E', E", Tan δ

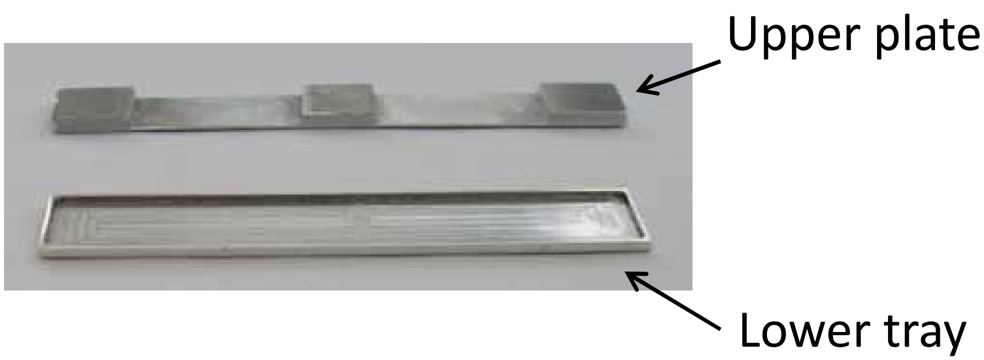


Glass Transition is a Range, not a Temperature

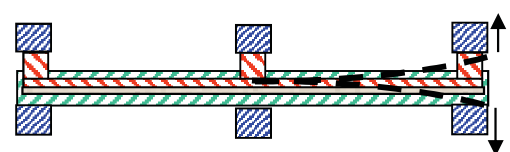


Powder Clamp

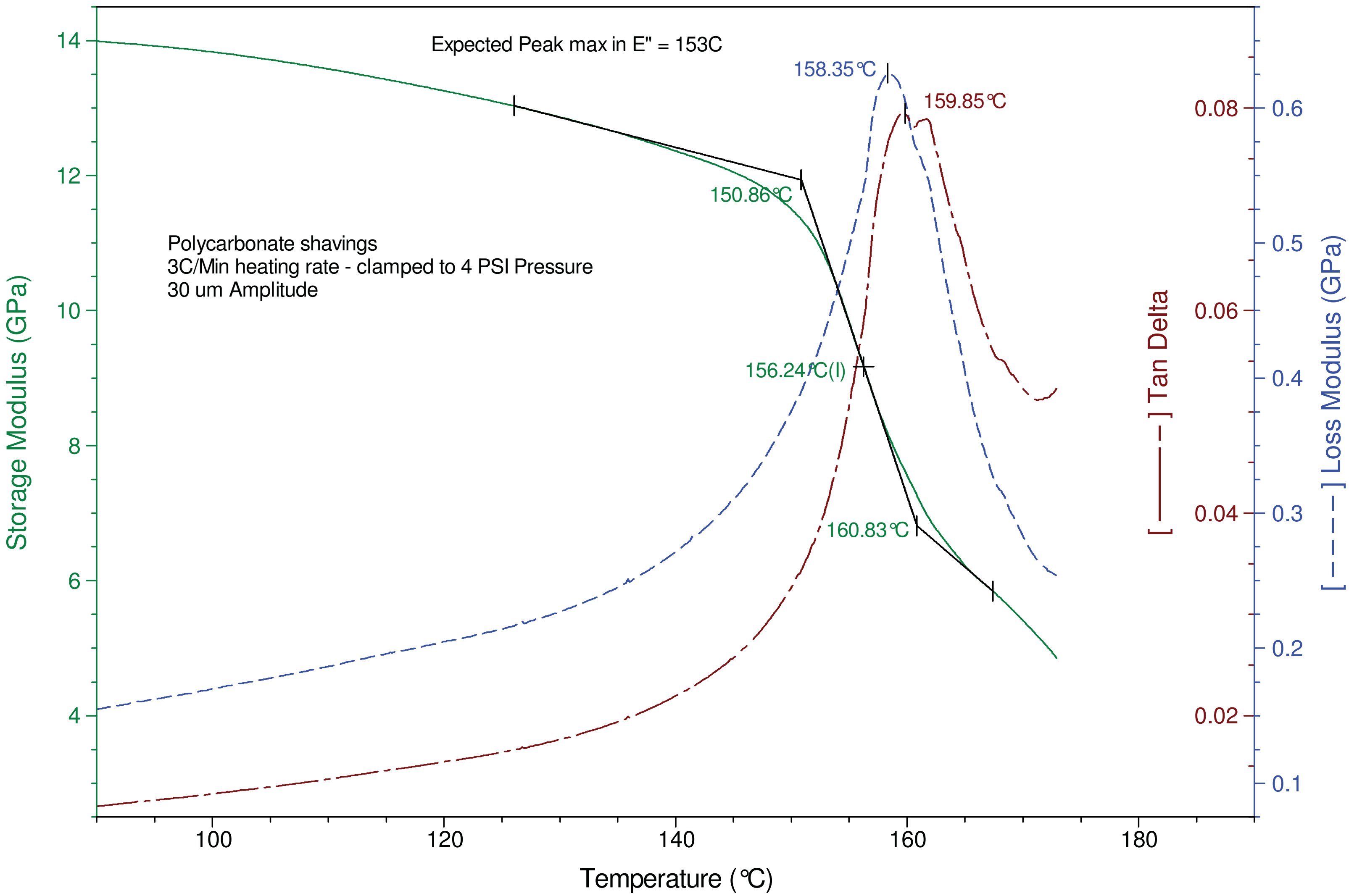
- Powder clamp measures thermal transitions in powders and is used with the standard 35 mm dual cantilever clamp



- Powder is placed in the lower tray
- Clamp will flex during measurements and contribution of the powder to the system is “magnified”
  - Use mesh or screen to aid in cleaning
  - Do not melt sample, cleaning will be difficult



Polycarbonate Shavings



Note: Disregard values of modulus

The Effect of Test Frequency on Tg

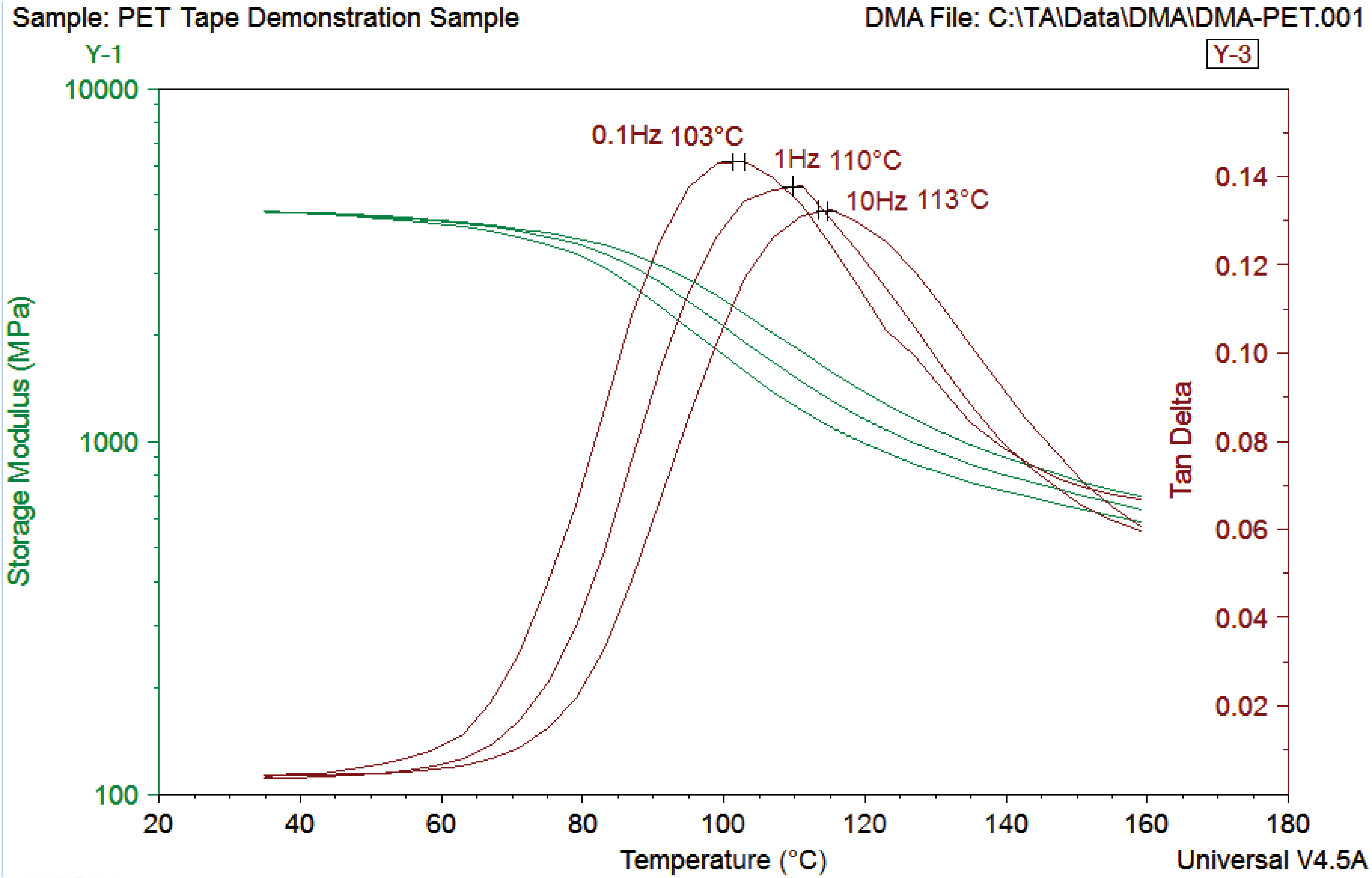
- The glass transition is a kinetic transition. It is therefore influenced strongly by the frequency of the test. The T_g is a molecular relaxation that involves cooperative segmental motion.
- Because the *RATE* of segmental motion depends on temperature, as the frequency increases, the relaxations associated with the T_g can only happen at higher temperatures.

- In general, increasing the frequency will
  - Increase the Tg
  - Decrease the intensity of tan δ or loss modulus
  - Broaden the peak
  - Decrease the slope of the storage modulus curve in the region of the transition.

Turi, Edith, A, Thermal Characterization of Polymeric Materials, Second Edition, Volume I., Academic Press, Brooklyn, New York, P. 529.

TA

PET Film: Effect of Frequency on Tg



RSA G2 Dielectric Accessory

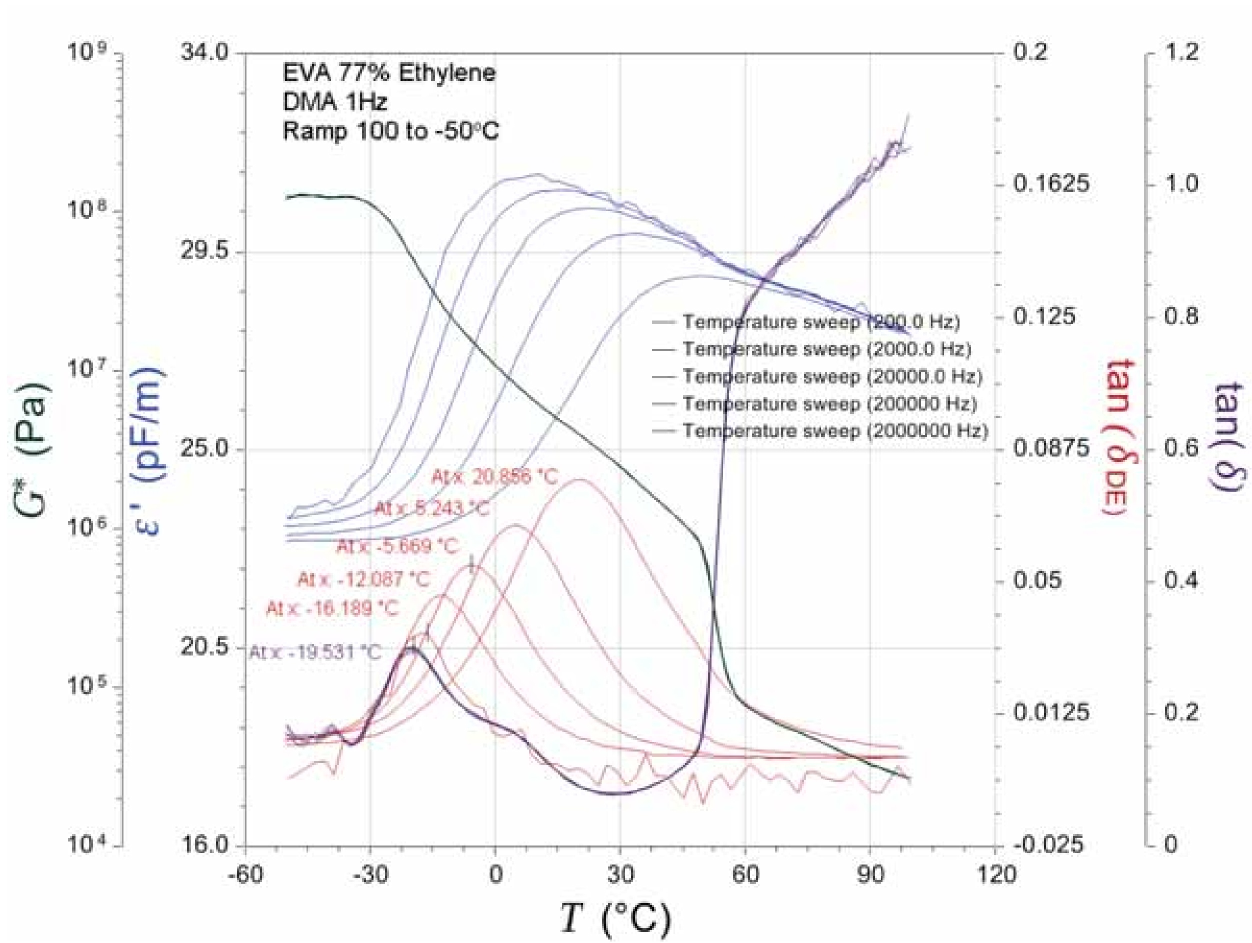
- RSA G2 can be positioned as two instruments in one.
  - DMA/Solids Analyzer
  - Dielectric Analyzer

Attribute	Specification
Geometry	25 mm PP
Temperature System Compatibility	FCO, Force Convection Oven
ARES/RSA to DE Bridge Interface	IEEE Internal to Instrument
Temperature Range	-160° to 300°C

(LCR Meter)	Frequency	AC Test Signal (potential)
Agilent 4285A	75 kHz to 30 MHz	0.005 to 10 Volts
Agilent E4980A	20 Hz to 2 MHz	0.005 to 20 Volts

Agilent LCR Meter Model 4980A

Relaxations in Ethylene Vinyl Acetate (EVA)



Temperature ramp from 100 to -50°C with dynamic mechanical response at 1Hz and simultaneous dielectric response at a frequency of:
200, 2000, 20000, 200000 and 2000000 Hz (Data collected on DHR rheometer)

The Glass & Secondary Transitions

Glass Transition

- Cooperative motion among a **large number** of chain segments, including those from neighboring polymer chains

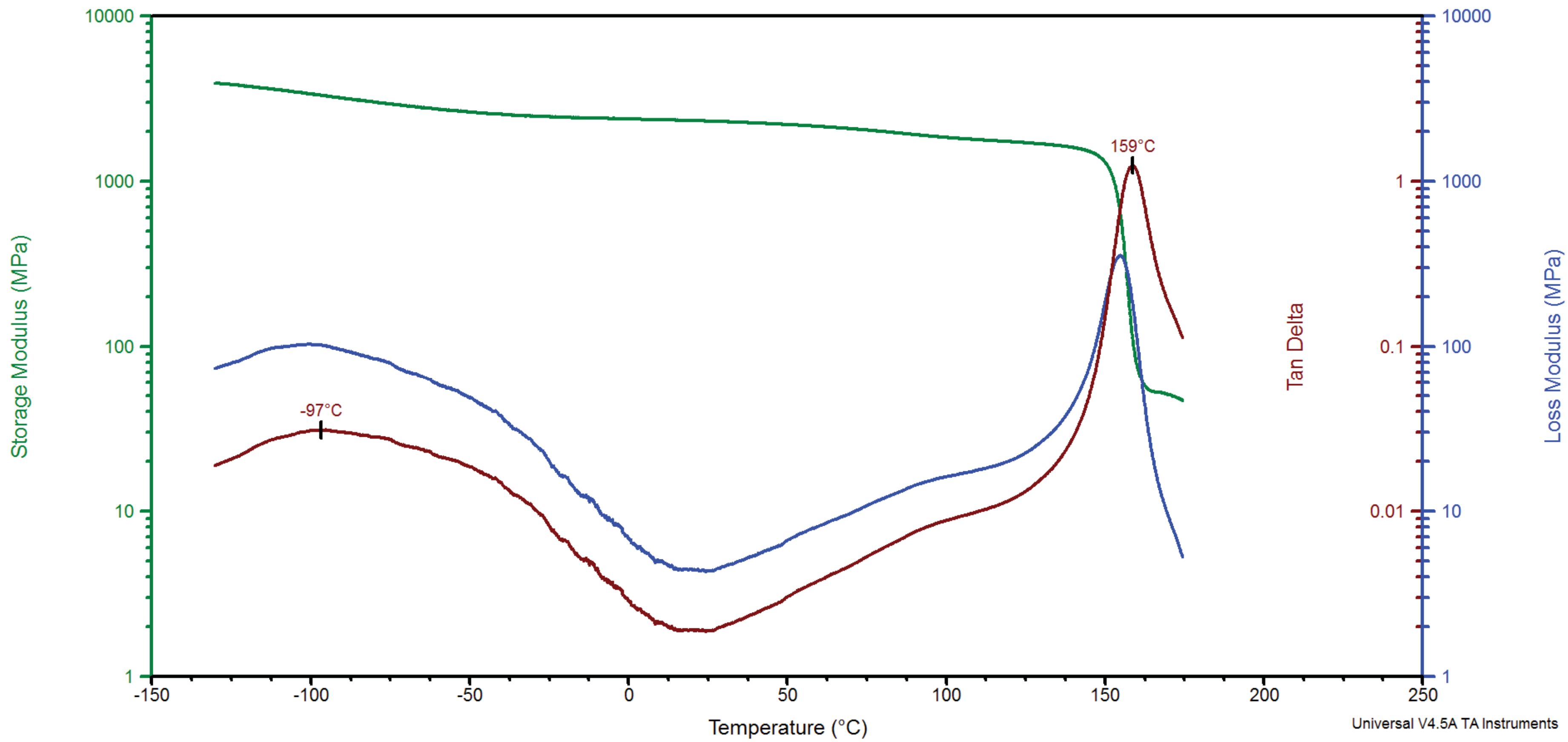
Secondary Transitions

- Local Main-Chain Motion – intra-molecular rotational motion of main chain segments four to six atoms in length
- Side group motion with some cooperative motion from the main chain
- Internal motion within a side group without interference from side group.
- Motion of, or within, a small molecule or diluent dissolved in the polymer (eg. plasticizer.)

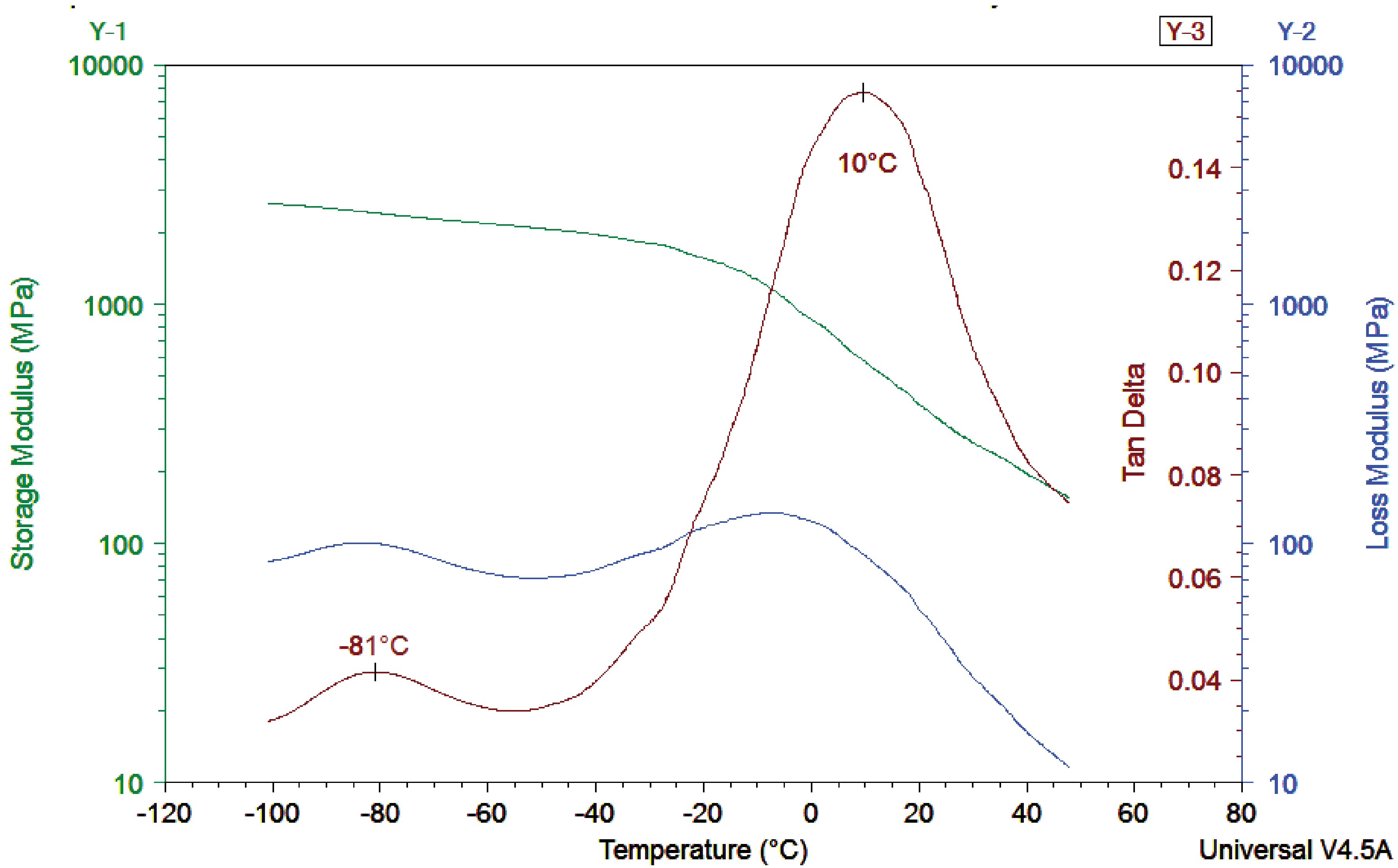
Turi,Edith, A, Thermal Characterization of Polymeric Materials, Second Edition, Volume I., Academic Press, Brooklyn, New York, P. 487.

Primary and Secondary Transitions in Polycarbonate

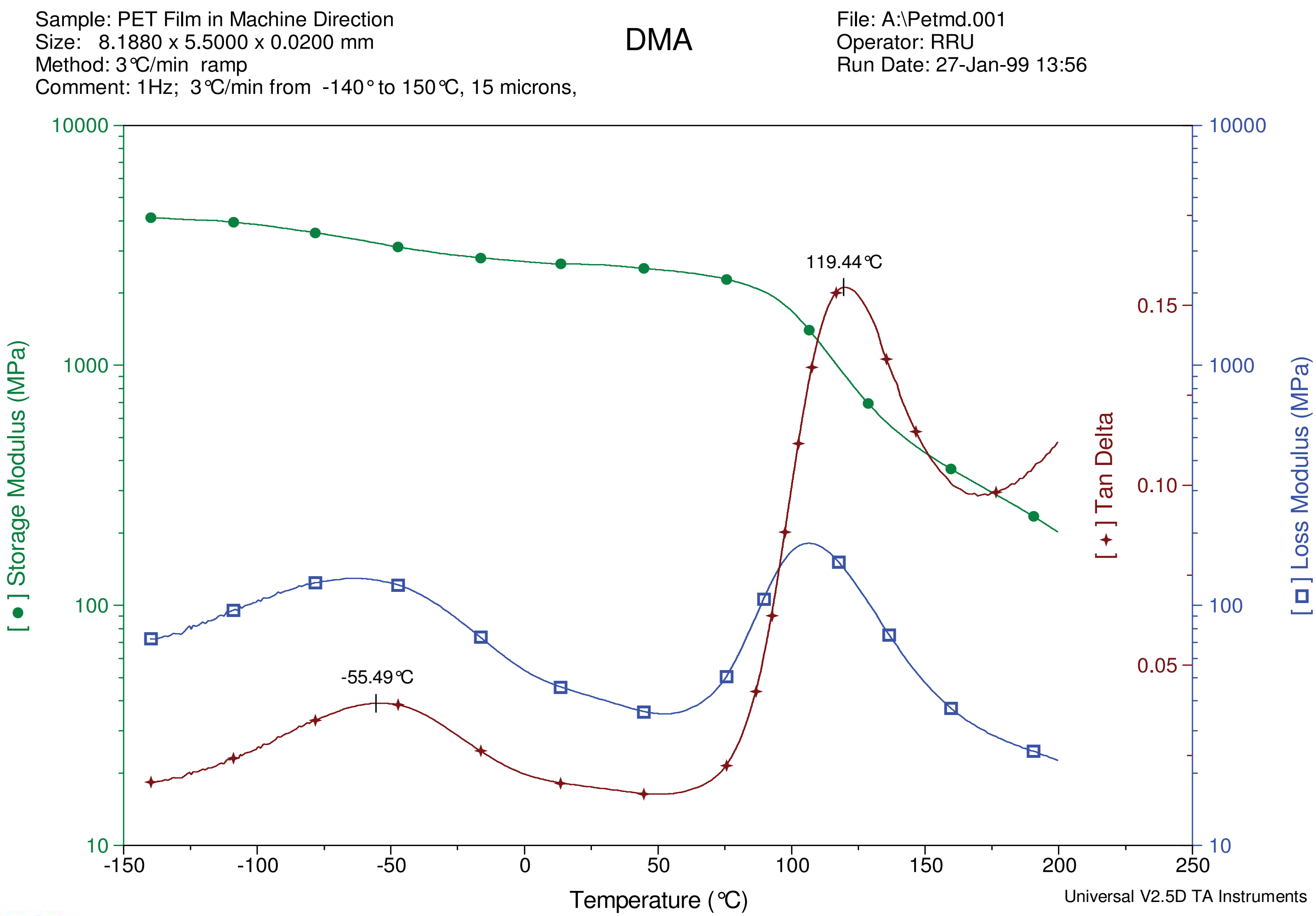
Single Cantilever, Multi Frequency- Strain
Temperature Ramp 2° C/ minute, 1 Hz, 15 µm



Primary and Secondary Transitions in Nylon

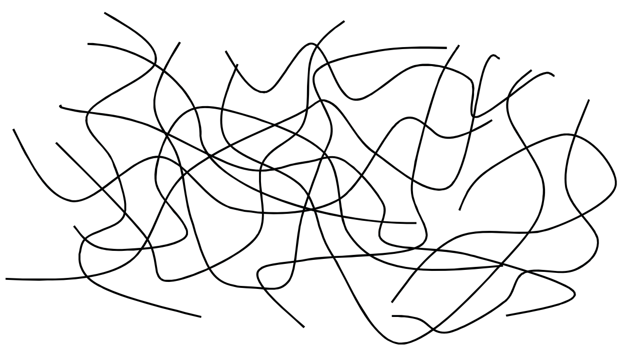


Primary and Secondary Transition in PET Film

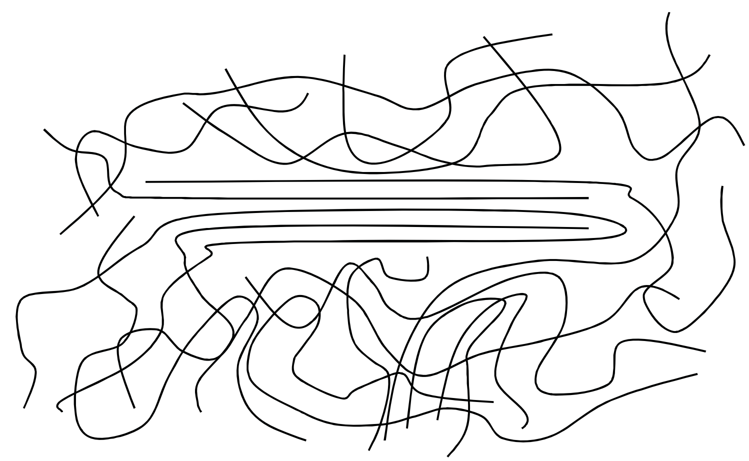


Effect of % Crystallinity on Modulus

"The major effect of the crystallite in a sample is to act as a crosslink in the polymer matrix. This makes the polymer behave as though it was a crosslinked network, but as the crystallite anchoring points are thermally labile, they disintegrate as the temperature approaches the melting temperature, and the material undergoes a progressive change in structure until beyond T_m, when it is molten"



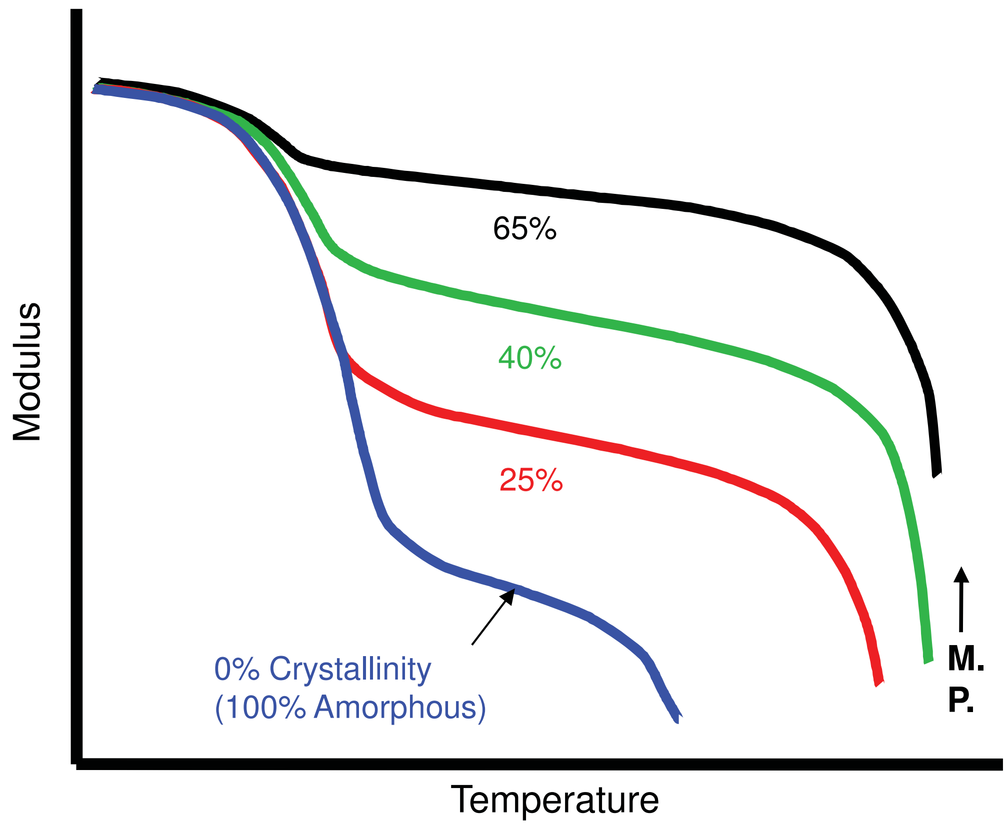
Random Chain
100% Amorphous



Fringed Micell
Crystalline

Cowie, J.M.G., Polymers: Chemistry & Physics of Modern Materials, 2nd Edition, Blackie academic & Professional, and imprint of Chapman & HallBishopbriggs, Glasgow, 1991p. 330-332. ISBN 0 7514 0134 X

Effect of % Crystallinity on Modulus



1. Crystallinity only affects the sample at $T_g < T < T_m$,
2. Below T_g the effect on the modulus is small
3. The Modulus at $T > T_g$ of a semi-crystalline polymer is directly proportional to the degree of crystallinity
4. Remains independent of temperature if the amount of crystalline order remains unchanged

Cowie, J.M.G., Polymers: Chemistry & Physics of Modern Materials, 2nd Edition, Blackie academic & Professional, and imprint of Chapman & HallBishopbriggs, Glasgow, 1991p. 330-332. ISBN 0 7514 0134 X

Effect of % Crystallinity on Glass Transition

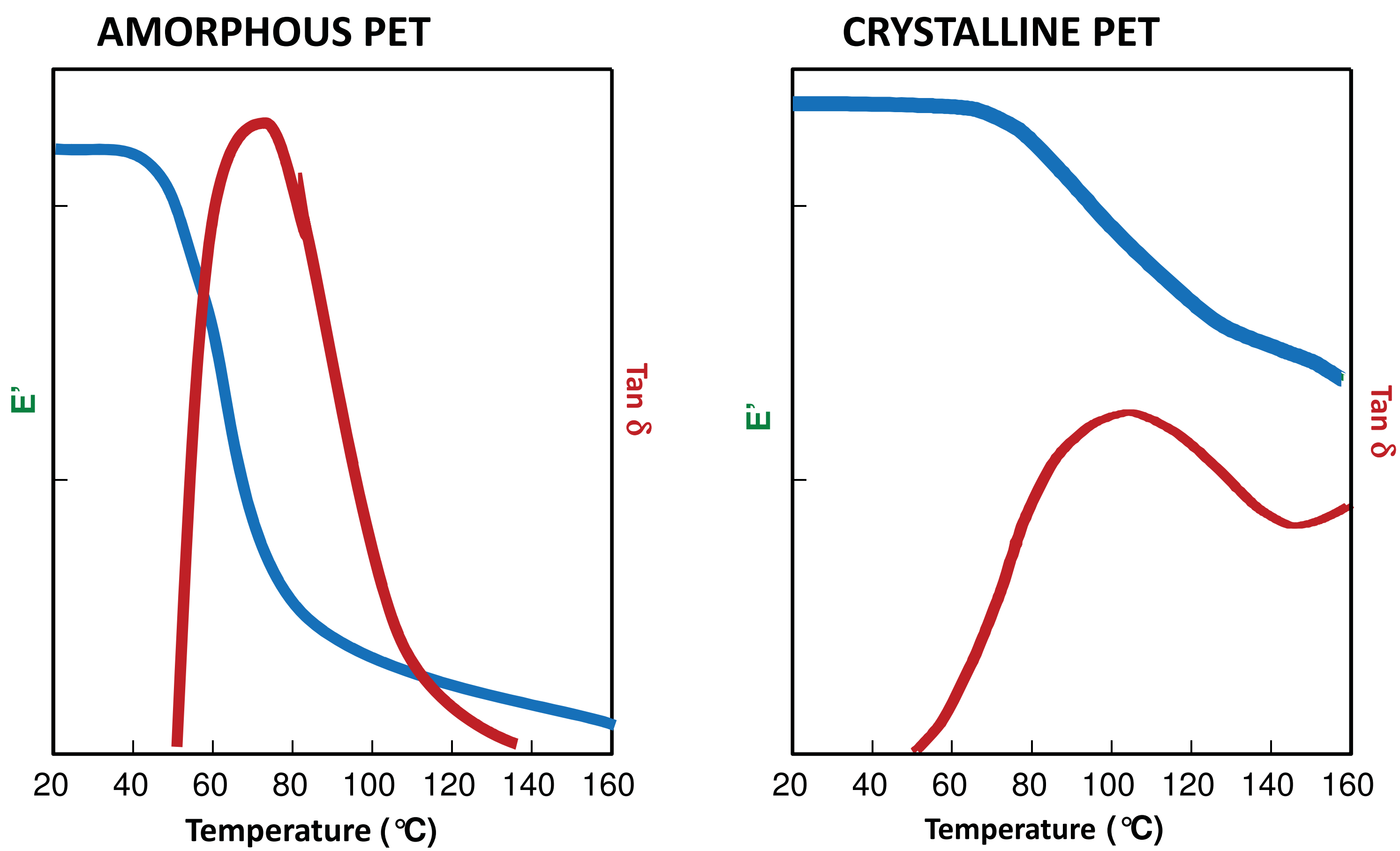
- Crystalline Polymers - multiphase systems - can be thought of as being made up of an amorphous phase containing dispersed crystalline units.

- Simple Picture of Two Phase System - crystalline phase acts to restrain mobility of neighboring amorphous regions

- General Case for Semicrystalline Polymers
  - Increasing Crystallinity will:
    - increase the glass transition temperature
    - decrease the intensity of the glass transition
    - broaden the transition temperature range

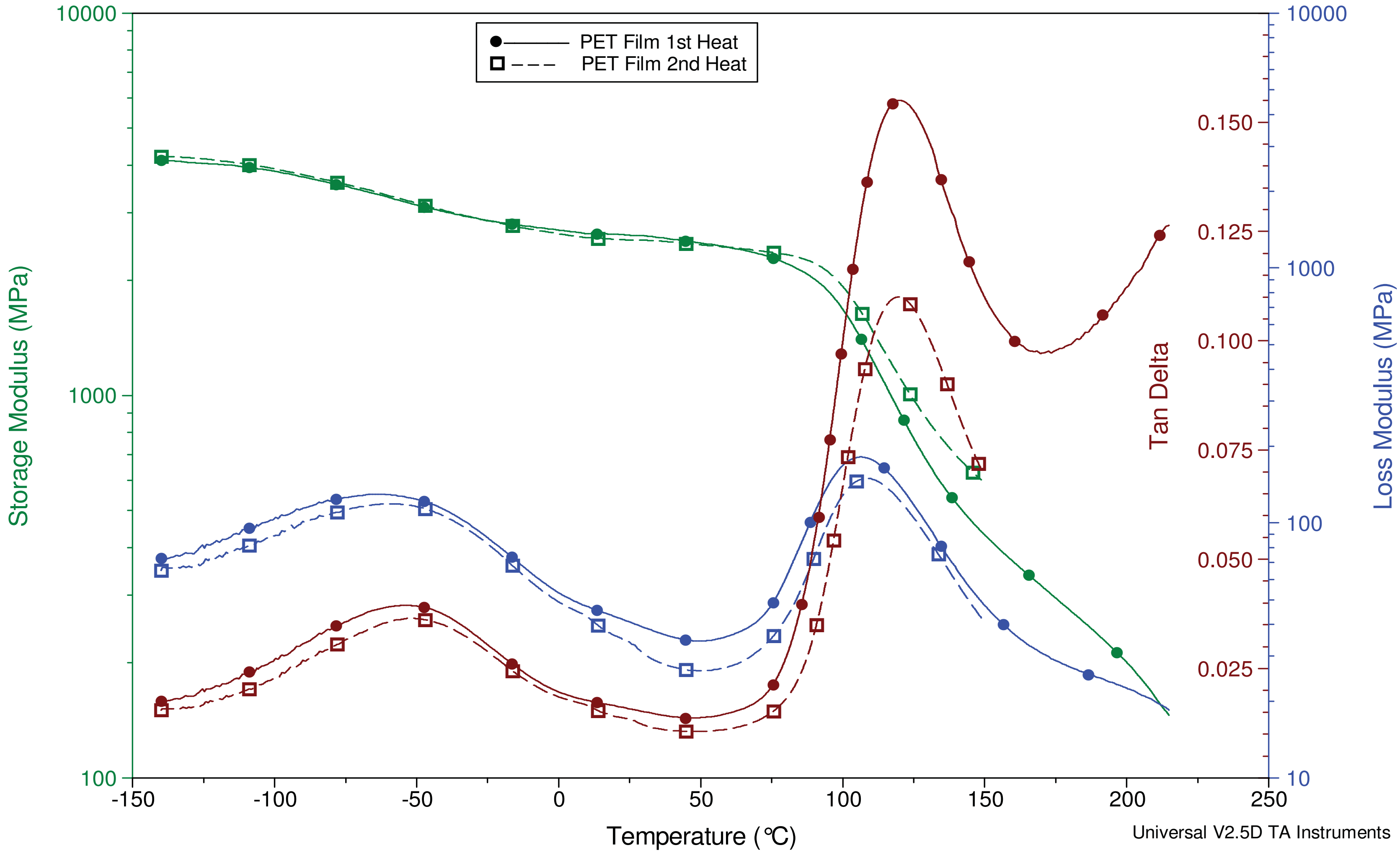
Turi, Edith, A, Thermal Characterization of Polymeric Materials, Second Edition, Volume I., Academic Press, Brooklyn, New York, P. 518.

Effect of % Crystallinity on Glass Transition

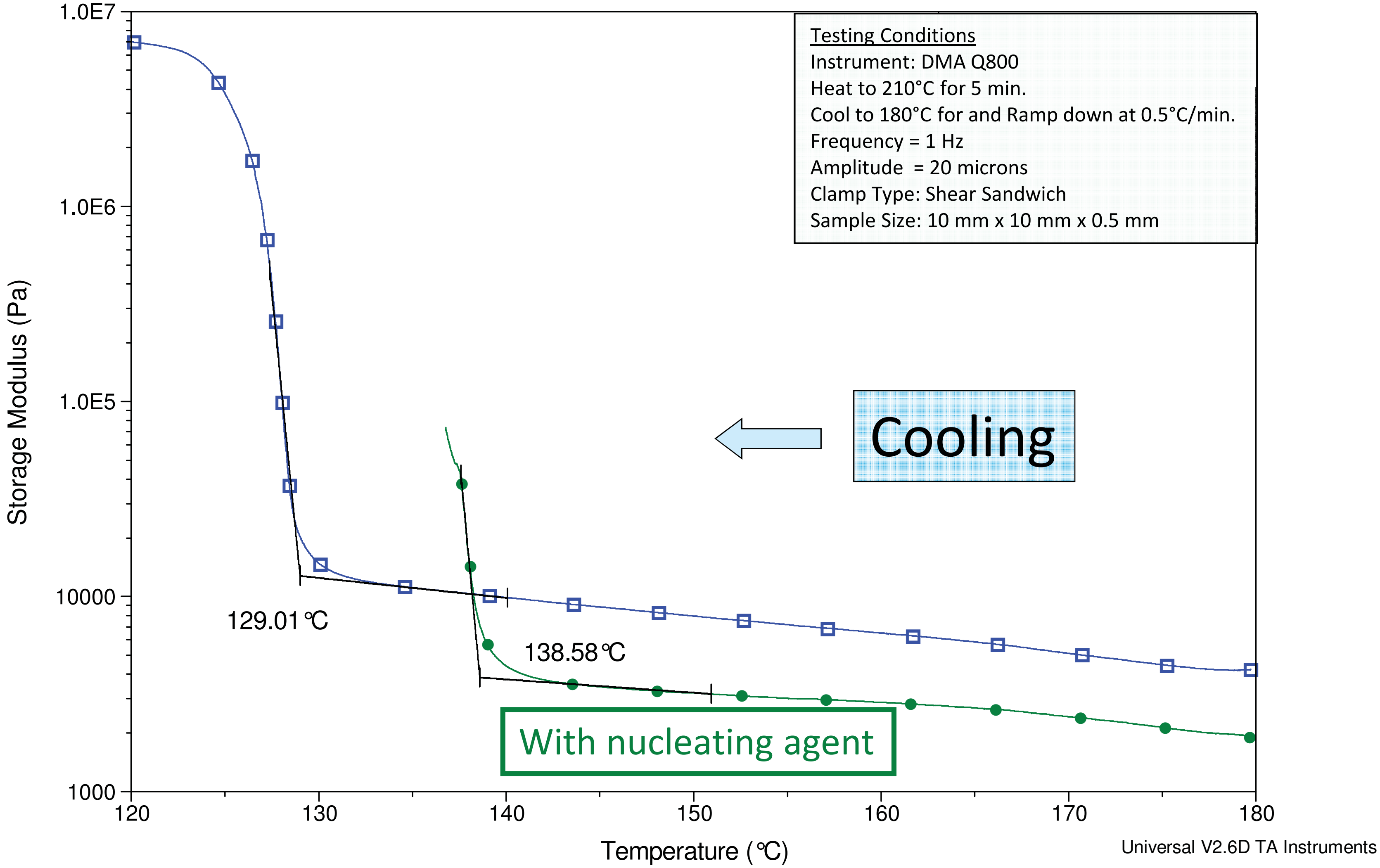


Redrawn with permission from Thompson and Woods, Trans. Faraday Soc., 52, 1383 (1956)

PET Film: Heat - Cool - Heat

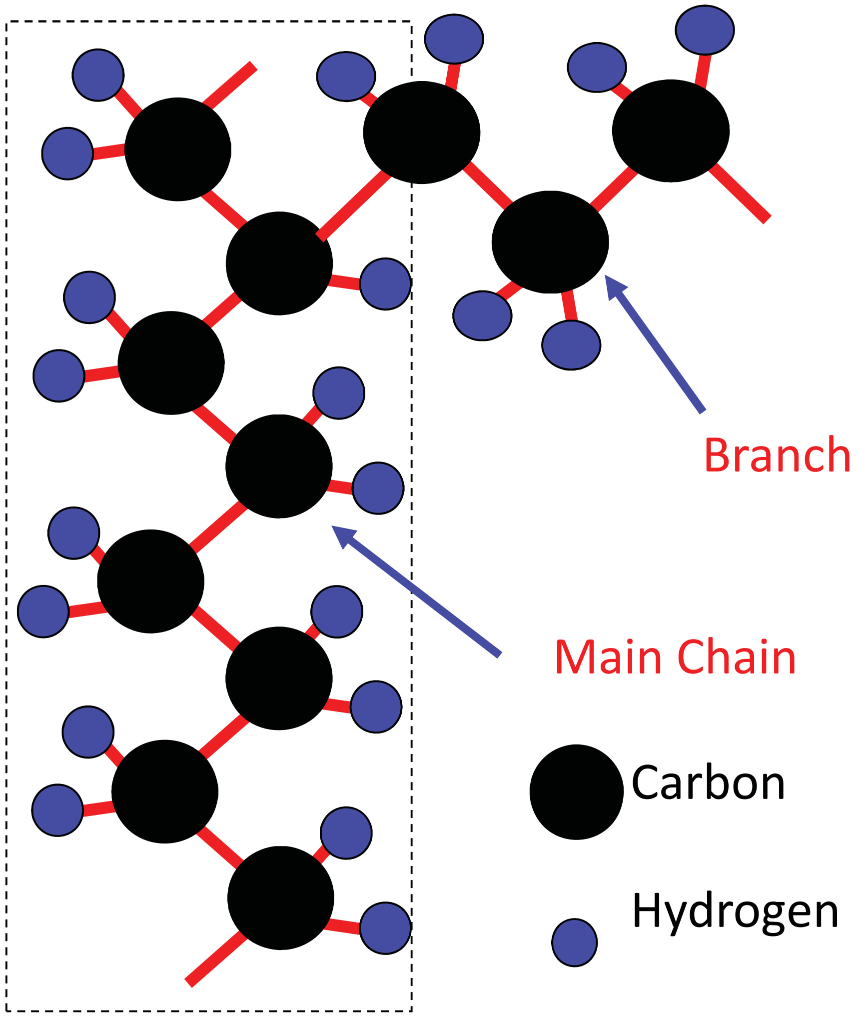


Polypropylene - Onset of Crystallization



Molecular Structure - Branching

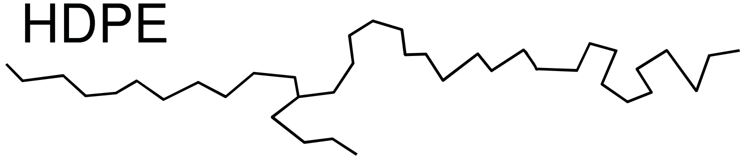
- A polymer chain can form a secondary chain initiating from a point on the main chain. This is called chain branching
- A schematic of a branched polyethylene chain is shown here. The presence of branches changes the mechanical properties considerably to linear polyethylene.



Ward, I.M., Hadley, D.W., An Introduction to the Mechanical Properties of Solid Polymers, John Wiley & Sons Ltd., New York, 1993, p.2.

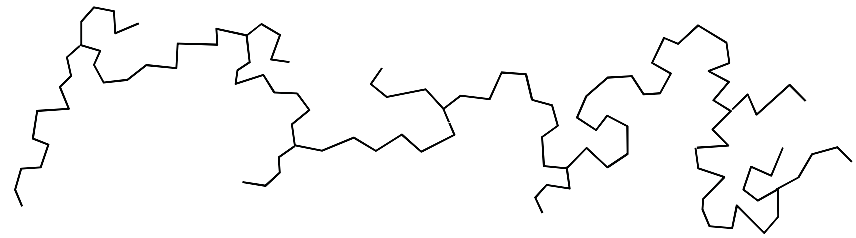
Effect of Branching on Polyethylene

HDPE



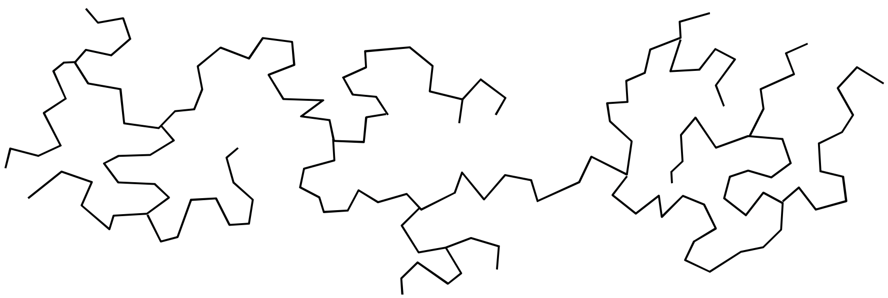
Structurally regular chain with few branch points. Polymer chains can pack efficiently resulting in **a highly crystalline material** with high density

LLDPE



Can be produced to have a chain with a controlled number of short chain branches yielding densities intermediate between HDPE and LDPE.

LDPE



Highly branched with **much lower crystalline content** and density compared to HDPE

Property	LDPE	LLDPE	HDPE
Melting Point (C)	110	120-130	>130
Density (g/cm^3)	0.92	0.93	0.96
Tensile strength (Mpa)	24	37	43

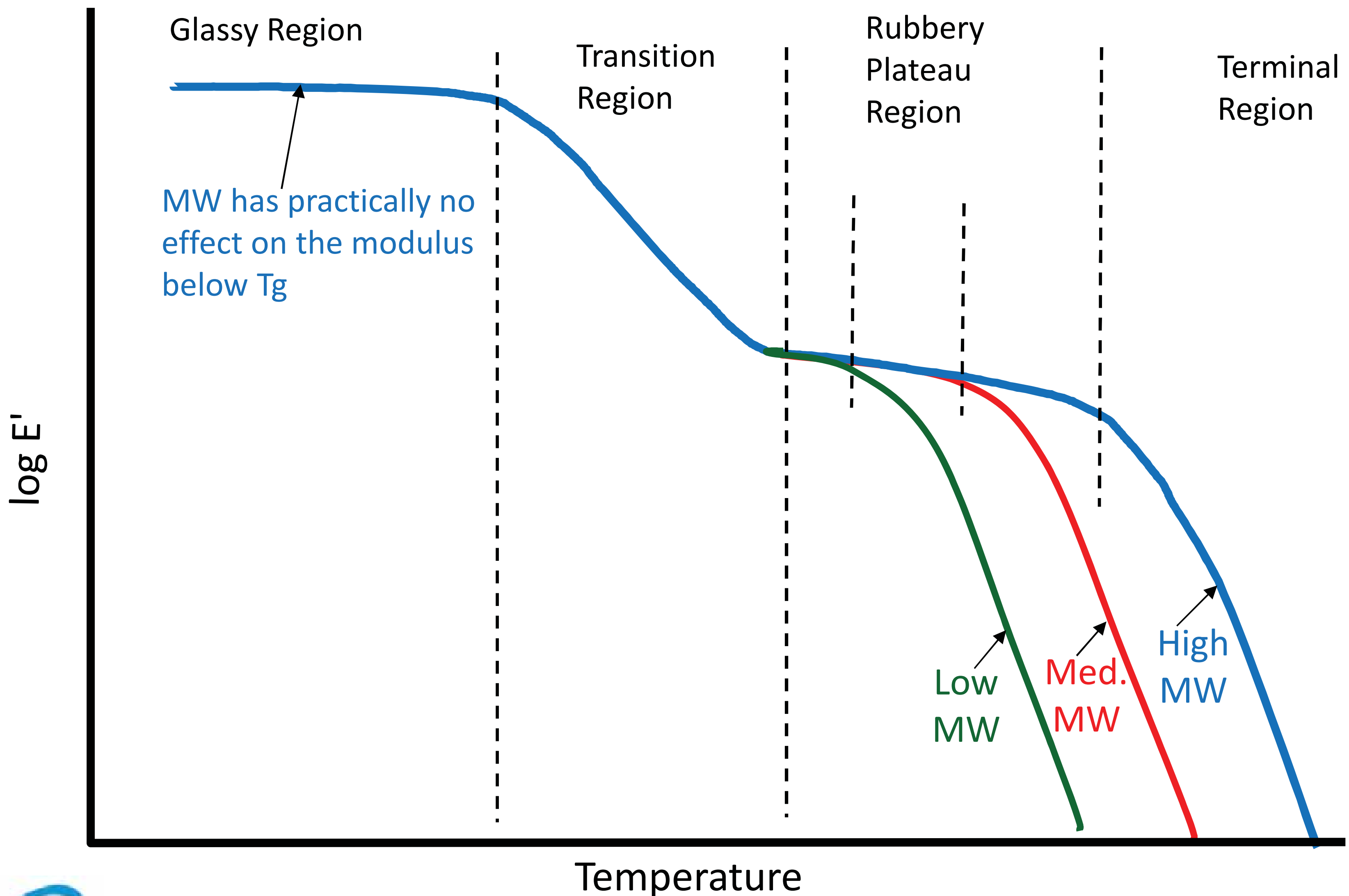
Cowie, J.M.G., Polymers: Chemistry & Physics of Modern Materials, 2nd Edition,
Blackie academic & Professional, and imprint of Chapman & Hall Bishopbriggs, Glasgow, 1991p. 342-345 ISBN 0 7514 0134 X

Effect of Molecular Weight

- Molecular Weight has practically no effect on the modulus below T_g.
- T_g and the drop in modulus are also nearly independent of MW if the MW is high enough to form entanglements
- The rubbery plateau region above T_g is strongly dependent on MW. In the absence of true crosslinks, the behavior is determined by *entanglements*.
- The length of the rubbery plateau ($T_g \leftrightarrow T_m$) is a function of the number of entanglements per molecule.

Nielsen, Lawrence E., Mechanical Properties of Polymers and Composites, Marcel Dekker, Inc., New York, 1974, p. 51-52.

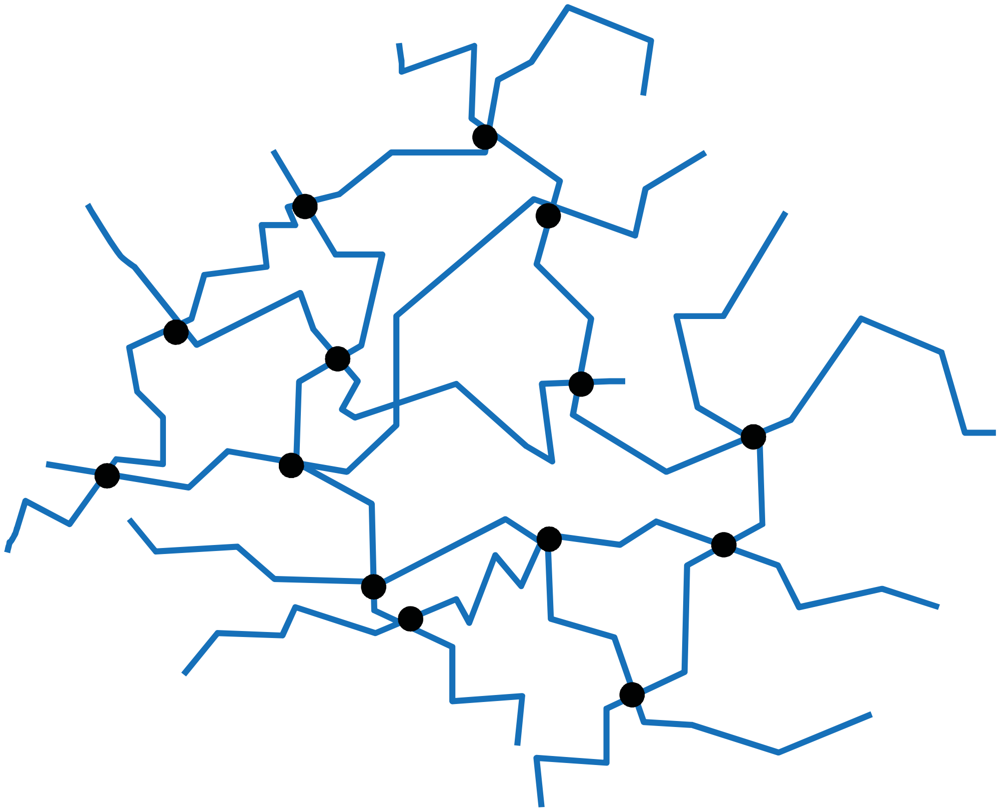
Molecular Structure - Effect of Molecular Weight



Molecular Structure - Crosslinking

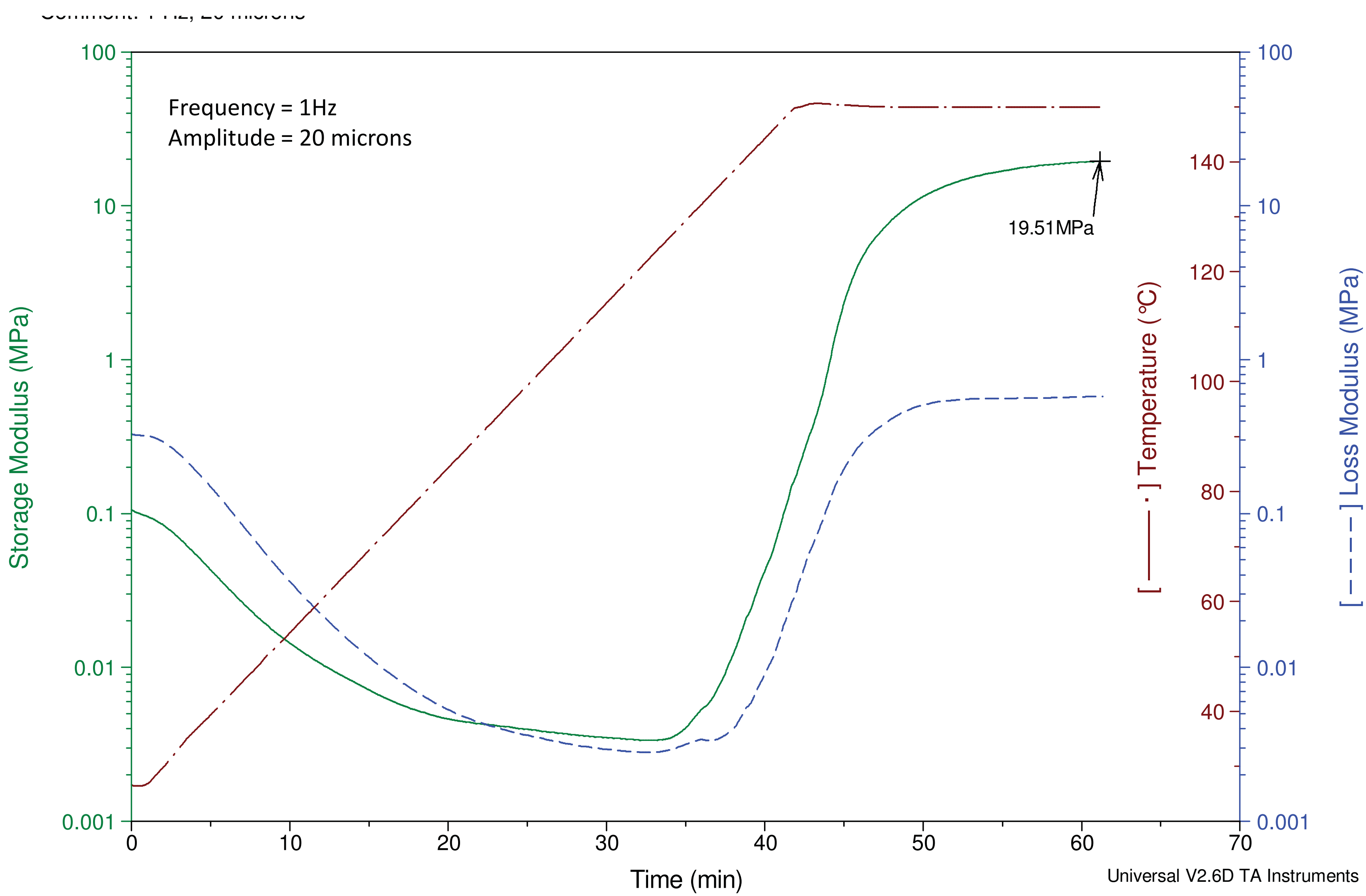
- Linear polymers can be chemically or physically joined at points to other chains along their length to create a crosslinked structure

- Chemically crosslinked systems are typically known as **thermosetting polymers** because the crosslinking agent is heat activated.

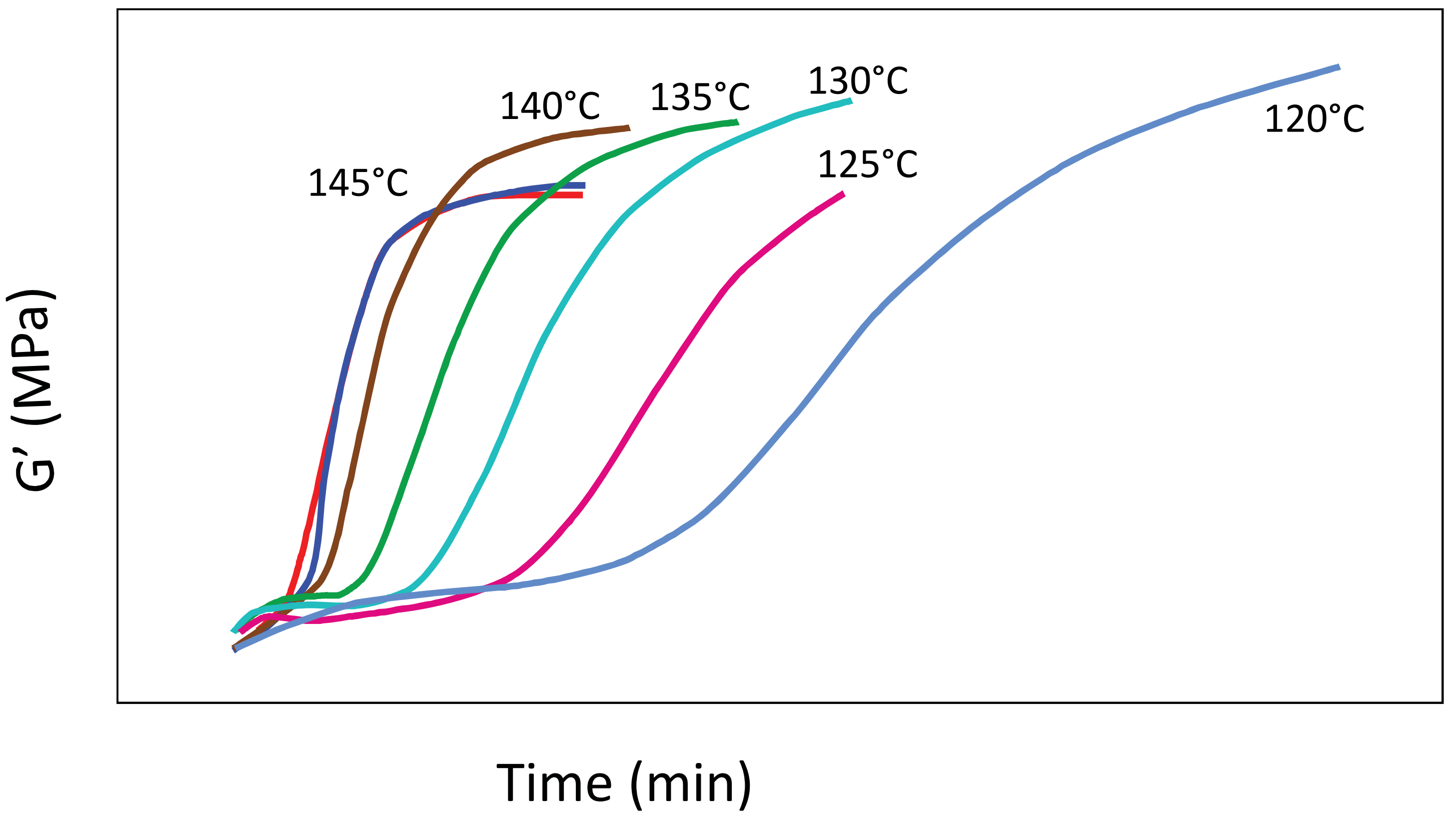


Ward, I.M., Hadley, D.W., An Introduction to the Mechanical Properties of Solid Polymers, John Wiley & Sons Ltd., New York, 1993

Sheet Molding Compound Cure in Shear Sandwich



Tire Compound: Effect of Curing Temperature



Effect of Crosslinking on T_g

Increasing Crosslinking

↓

Higher Density

↓

Less Free Volume

↓

Restricted molecular motion

↓

More Energy needed

↓

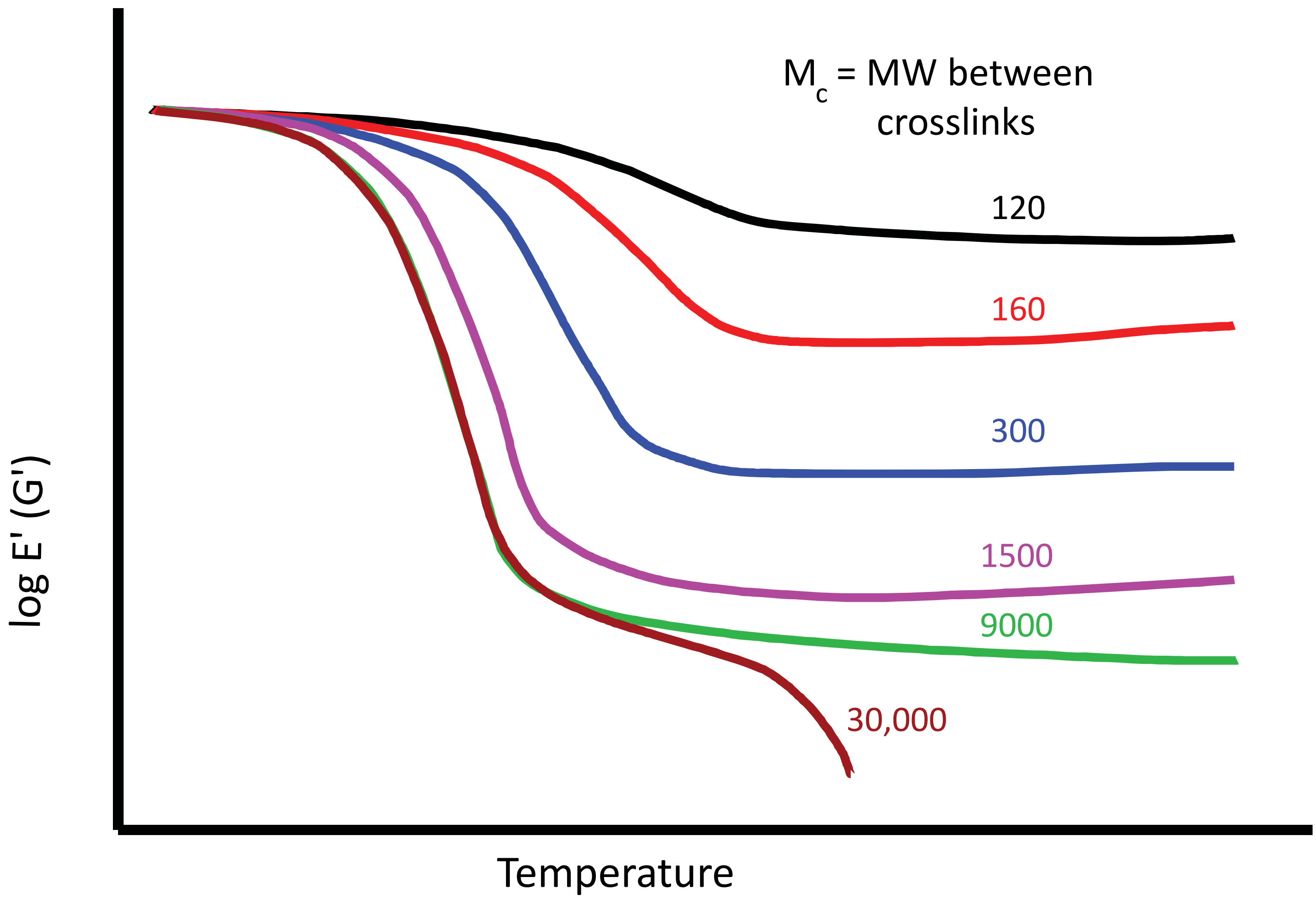
Higher Glass Transition Temperature

For low values of crosslink density, T_g can be found to increase linearly with the number of crosslinks.

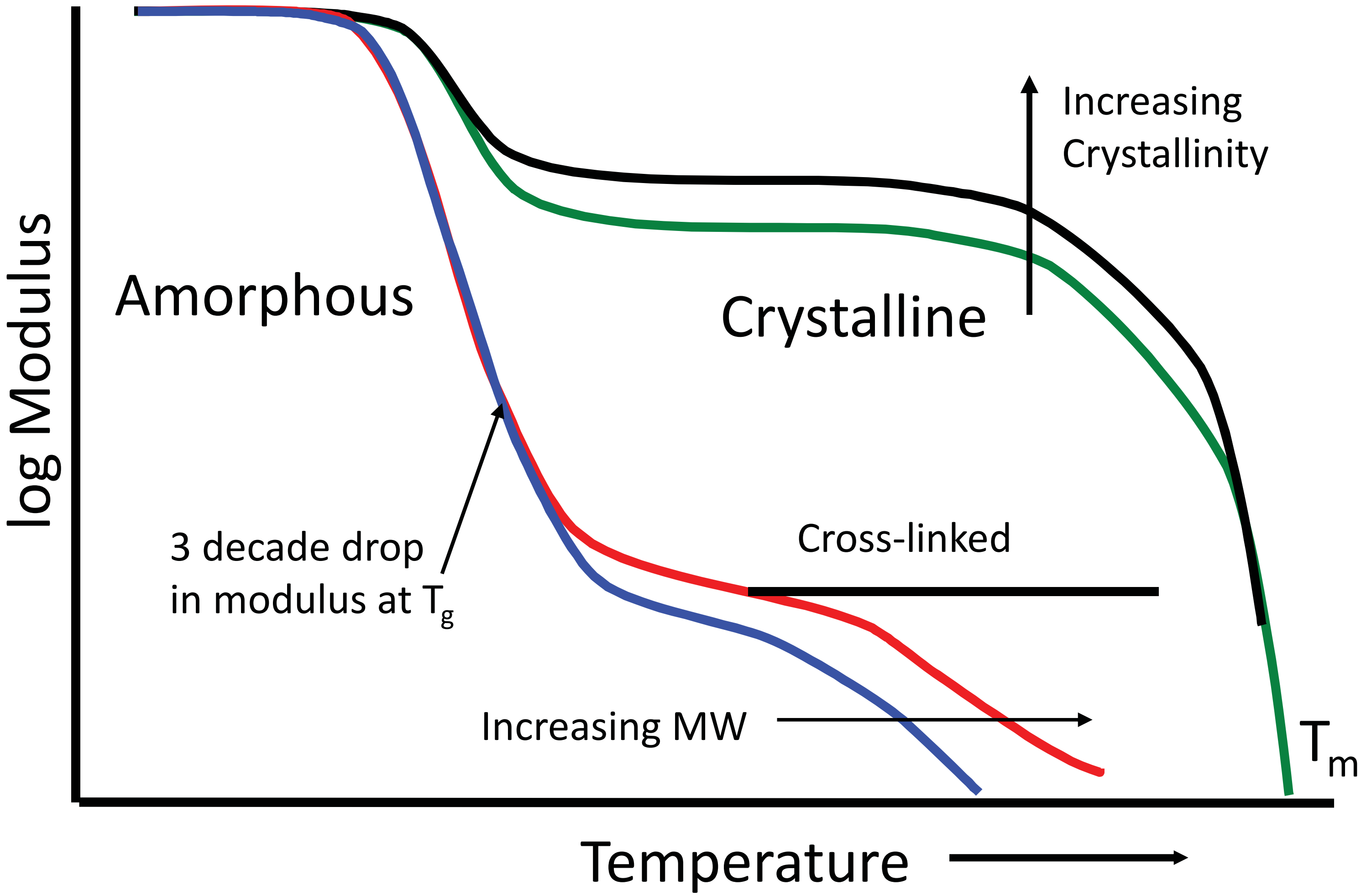
For high crosslink density, the T_g is broad and not well defined.

Cowie, J.M.G., Polymers: Chemistry & Physics of Modern Materials, 2nd Edition,
Blackie academic & Professional, and imprint of Chapman & HallBishopbriggs, Glasgow, 1991 p.262 ISBN 0 7514 0134 X

Effect of Crosslinking



Crystallinity, Molecular Weight, and Crosslinking



Effect of Orientation - Anisotropic Materials

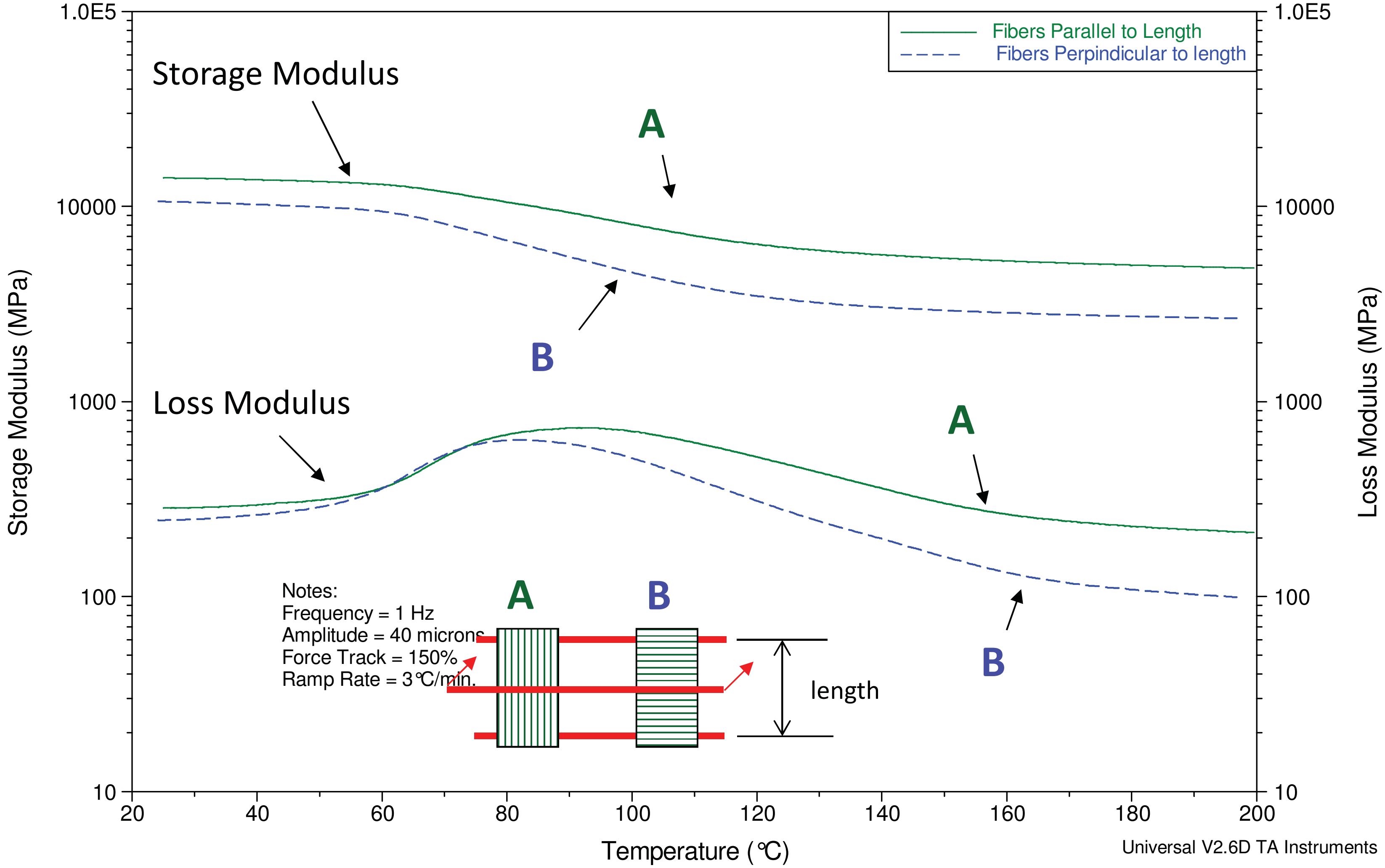
- Anisotropic materials have different properties in different directions.
  - fibers, wood, fiber-filled composites
  - oriented amorphous polymers, injection molded specimens
  - crystalline polymers in which the crystalline phase is not randomly oriented

- Have more than two independent moduli - generally a minimum of 5 or 6.

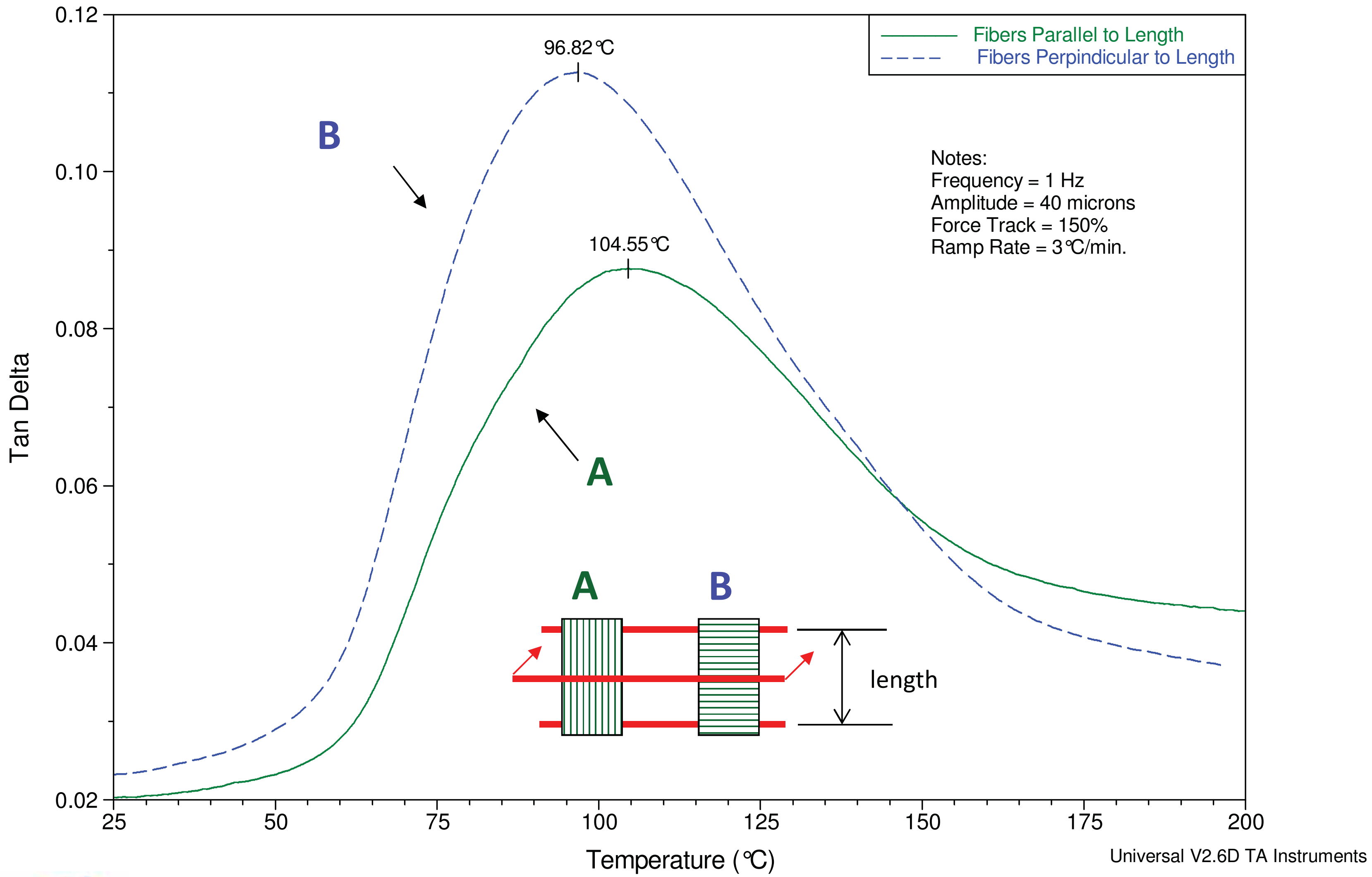
- The number of independent moduli depends on the symmetry in the system

Nielsen, Lawrence E., Mechanical Properties of Polymers and Composites, Marcel Dekker, Inc., New York, 1974, pp. 39-40.

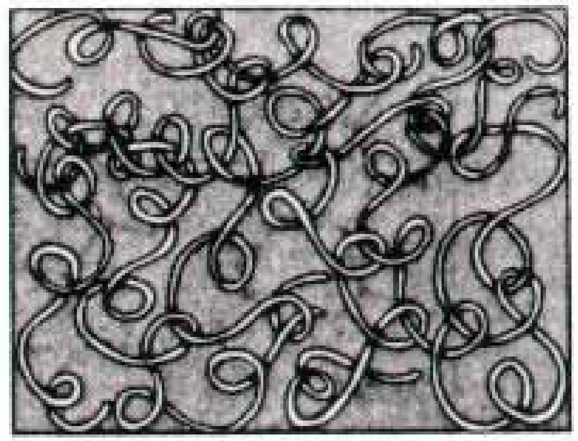
Anisotropic Material: Polyester/Glass Fiber Composite



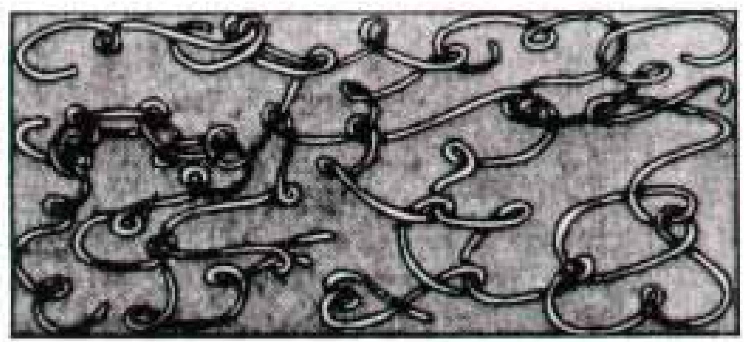
Anisotropic Material: Polyester/Glass Fiber Composite



Orientation in Amorphous Polymers



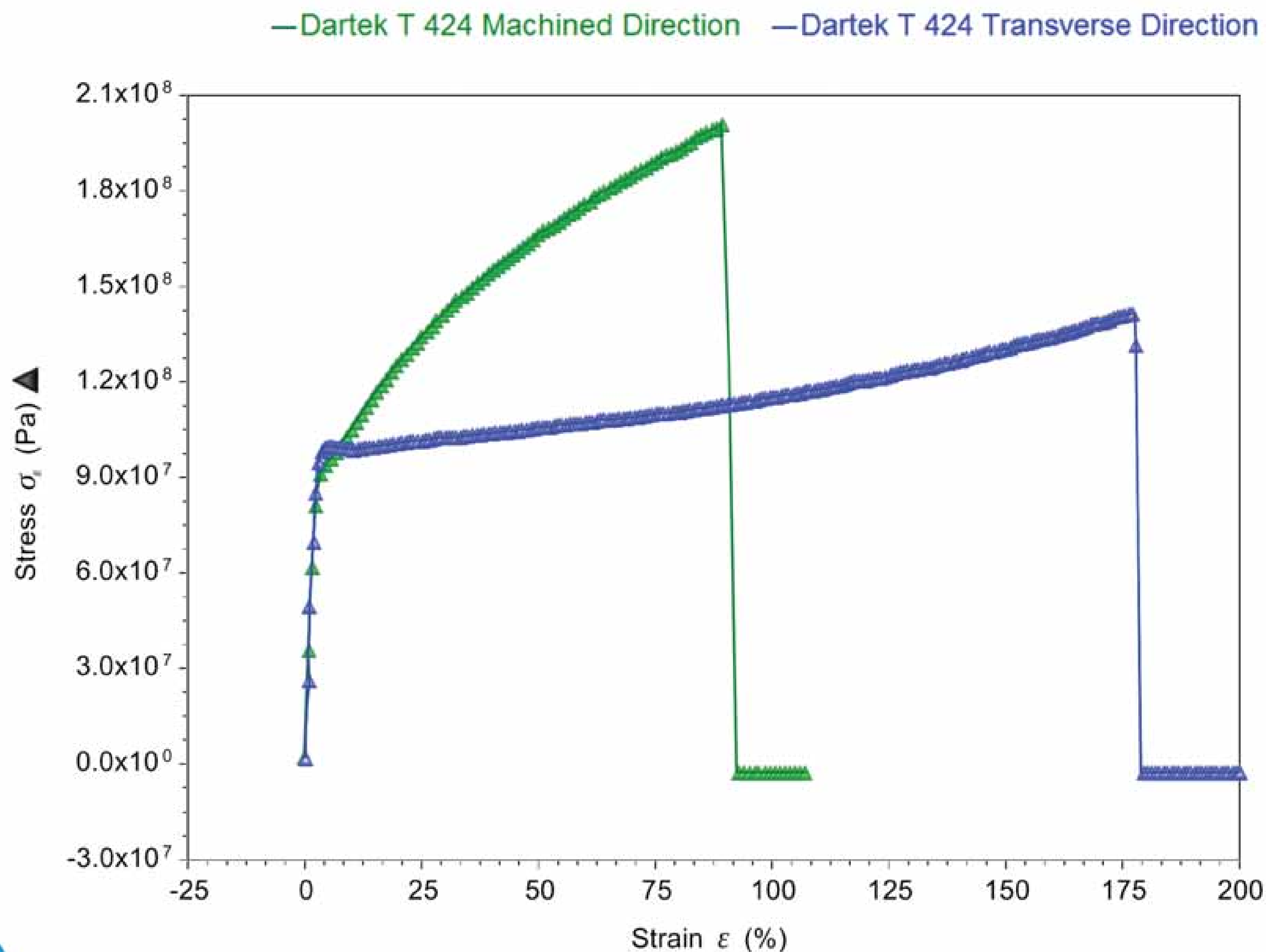
(a) Schematic of unoriented amorphous polymer



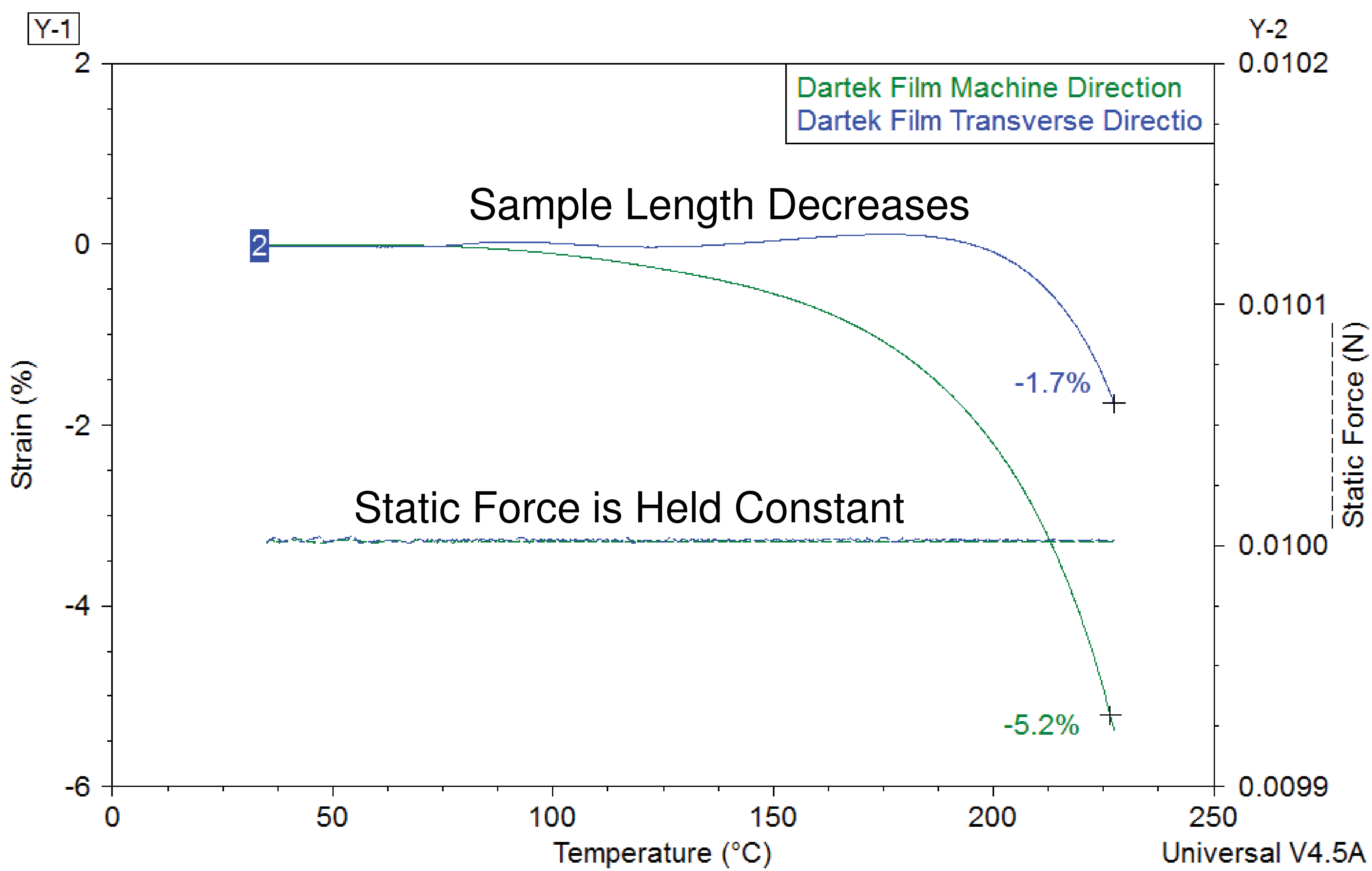
(b) Schematic of oriented amorphous polymer

Ward, I.M., Hadley, D.W., An Introduction to the Mechanical
Properties of Solid Polymers, John Wiley & Sons Ltd, New York, NY, 1993, p.11.

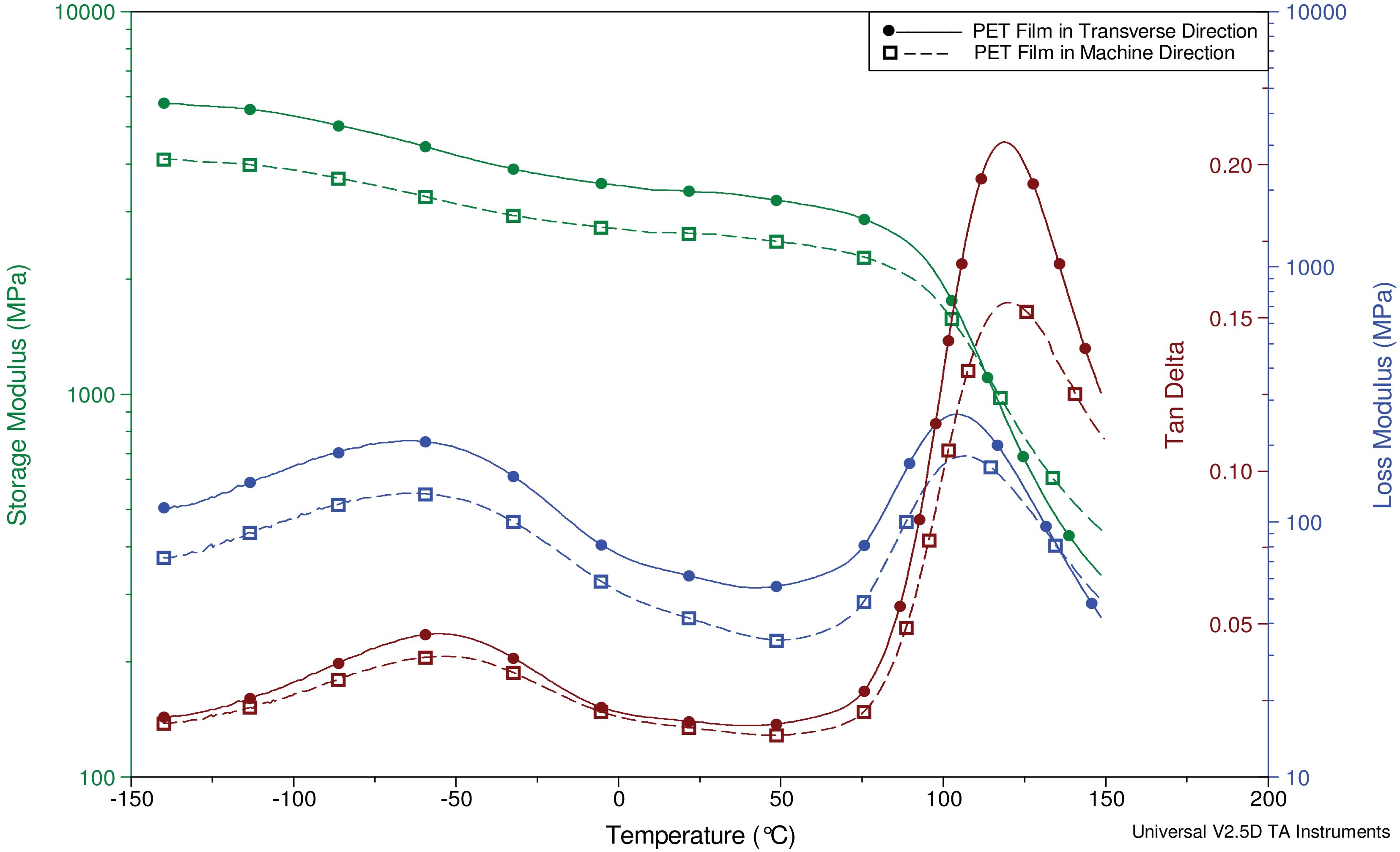
Stress-Strain of Oriented Film



Iso-Force Temp Ramp- Shrinkage of Oriented Film



PET Film in Machine and Transverse Direction

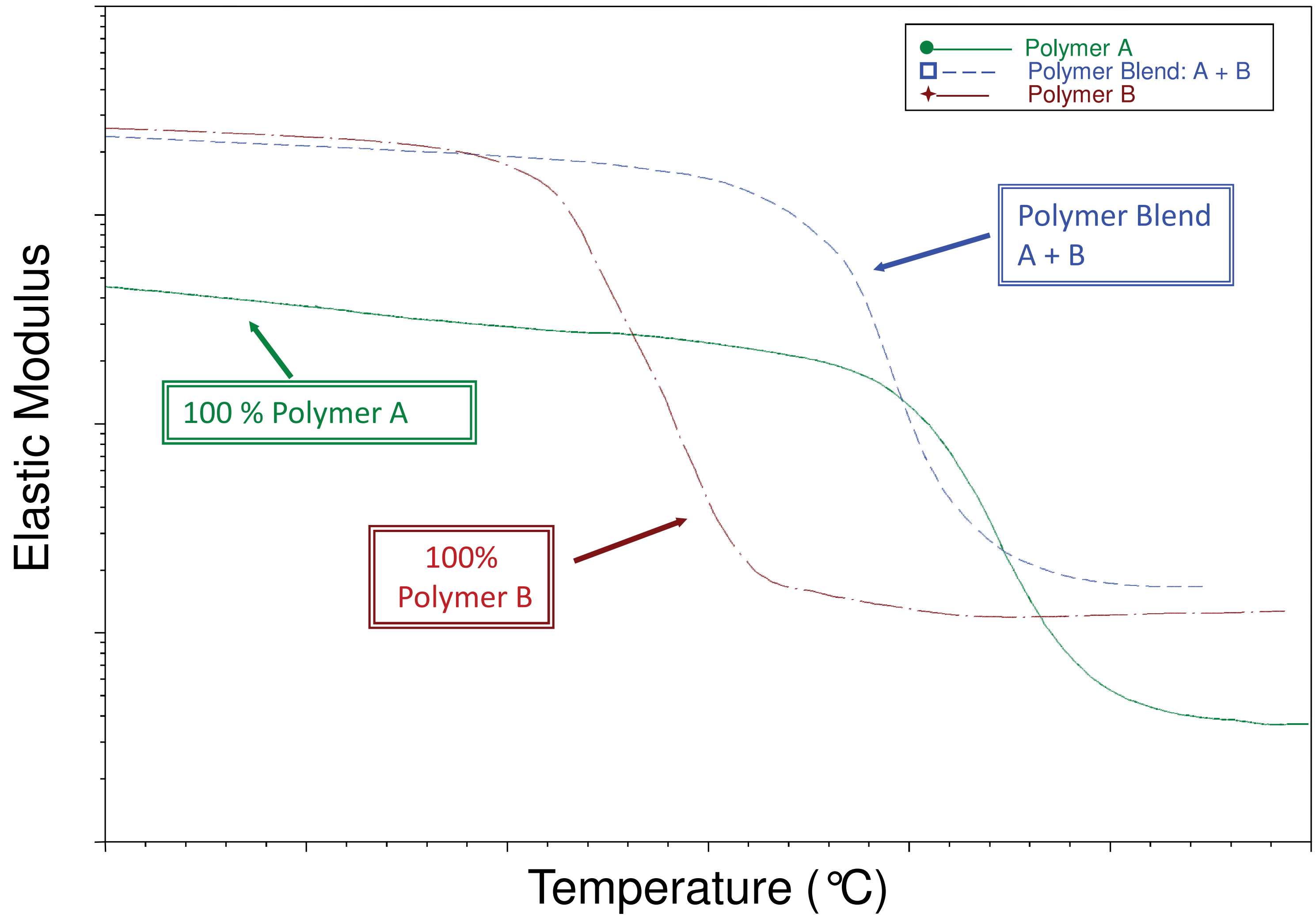


Blending of Amorphous Polymers

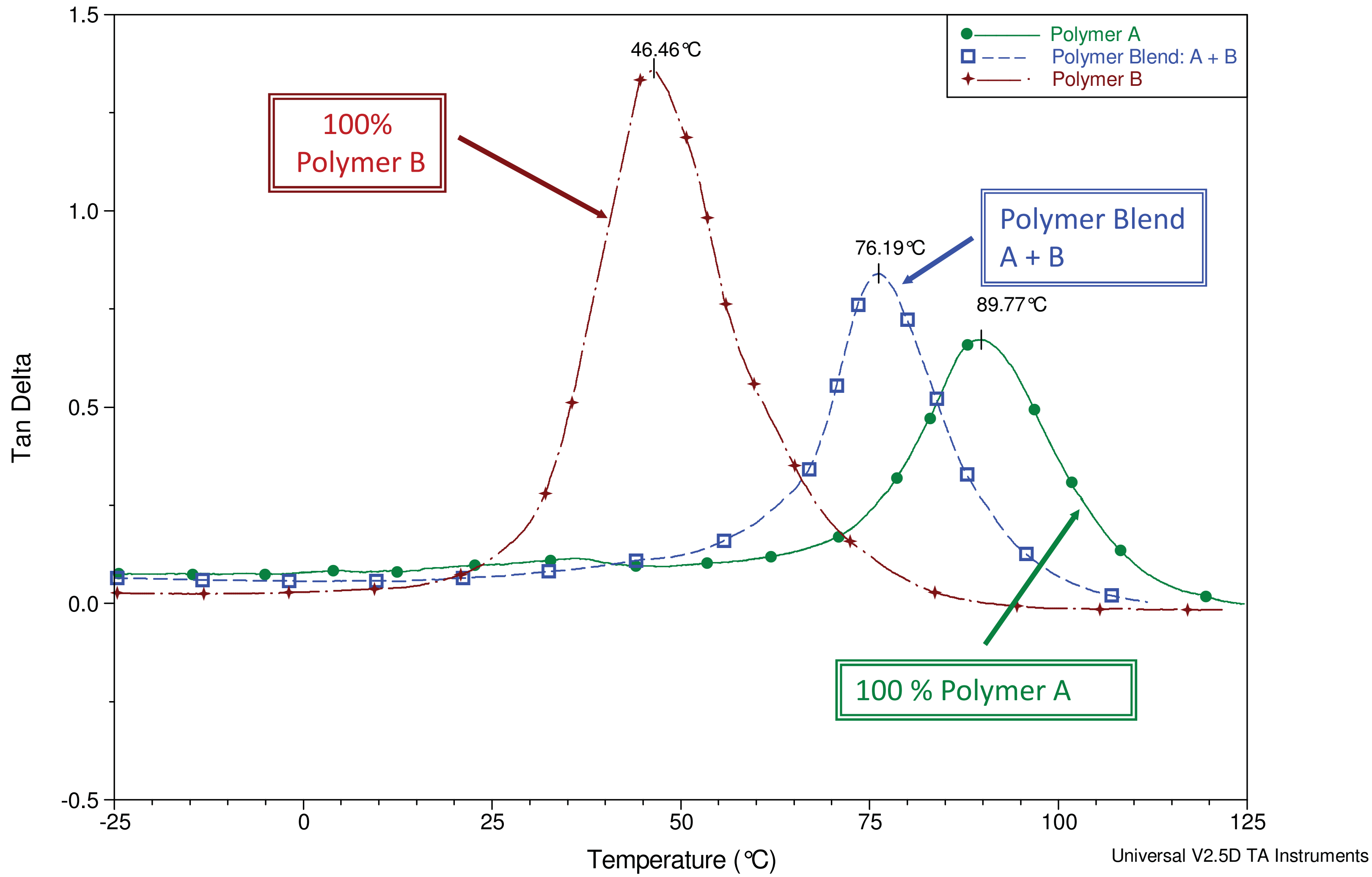
- Blending may produce a polymer whose modulus-temperature curve shows two transition regions
- If the polymers blended are completely compatible, then the blend behaves like an ordinary amorphous polymer with a single transition region and an intermediate glass transition temperature.
- Incompatible polymers will show separate glass transitions

Tobolsky, A.V., Properties and Structure of Polymers, John Wiley & Sons, Inc., New York, 1967, p.81.

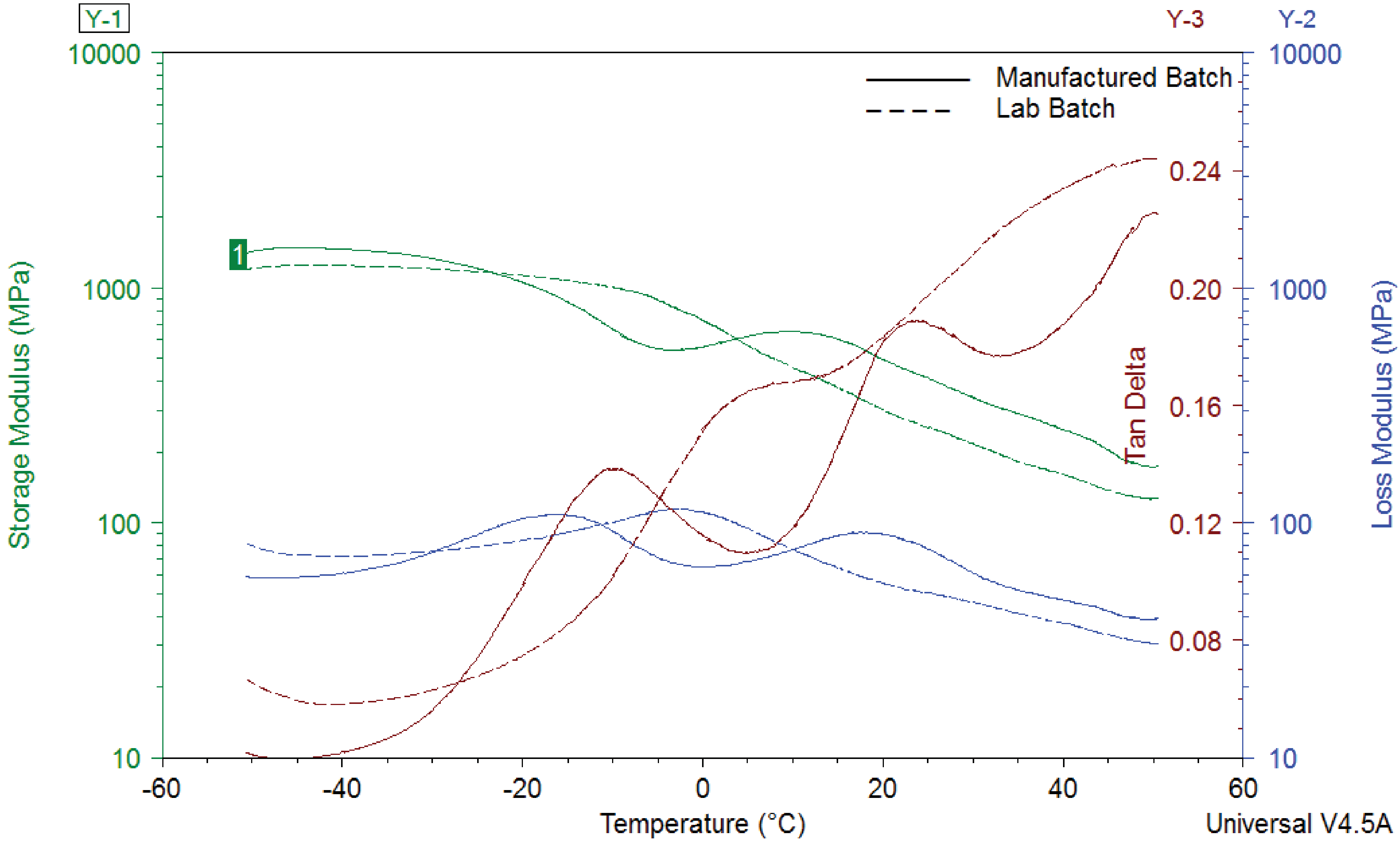
Polymer Blend - Aerospace Coating



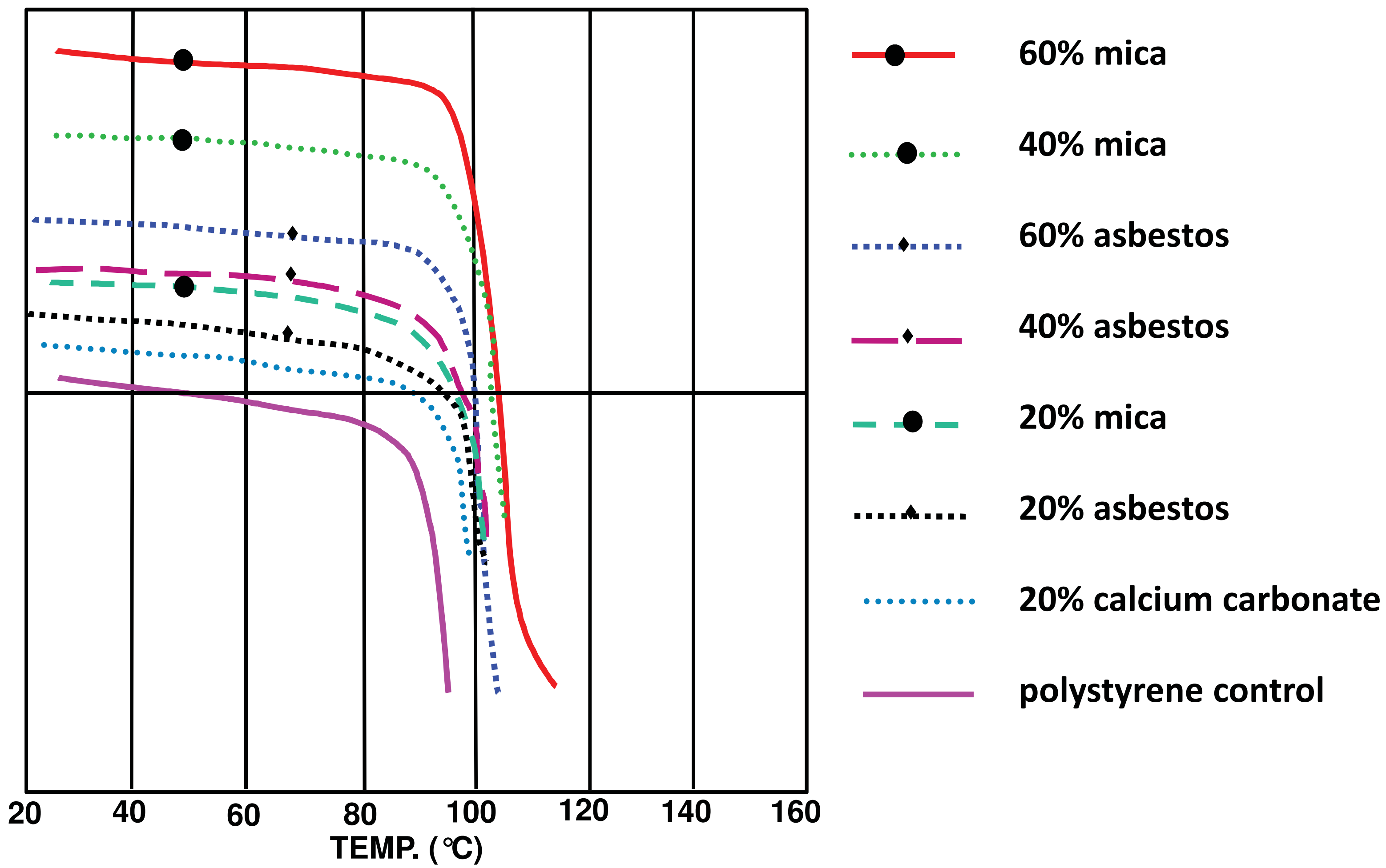
Polymer Blend - Aerospace Coating



Using Glass Transition to Evaluate Blending

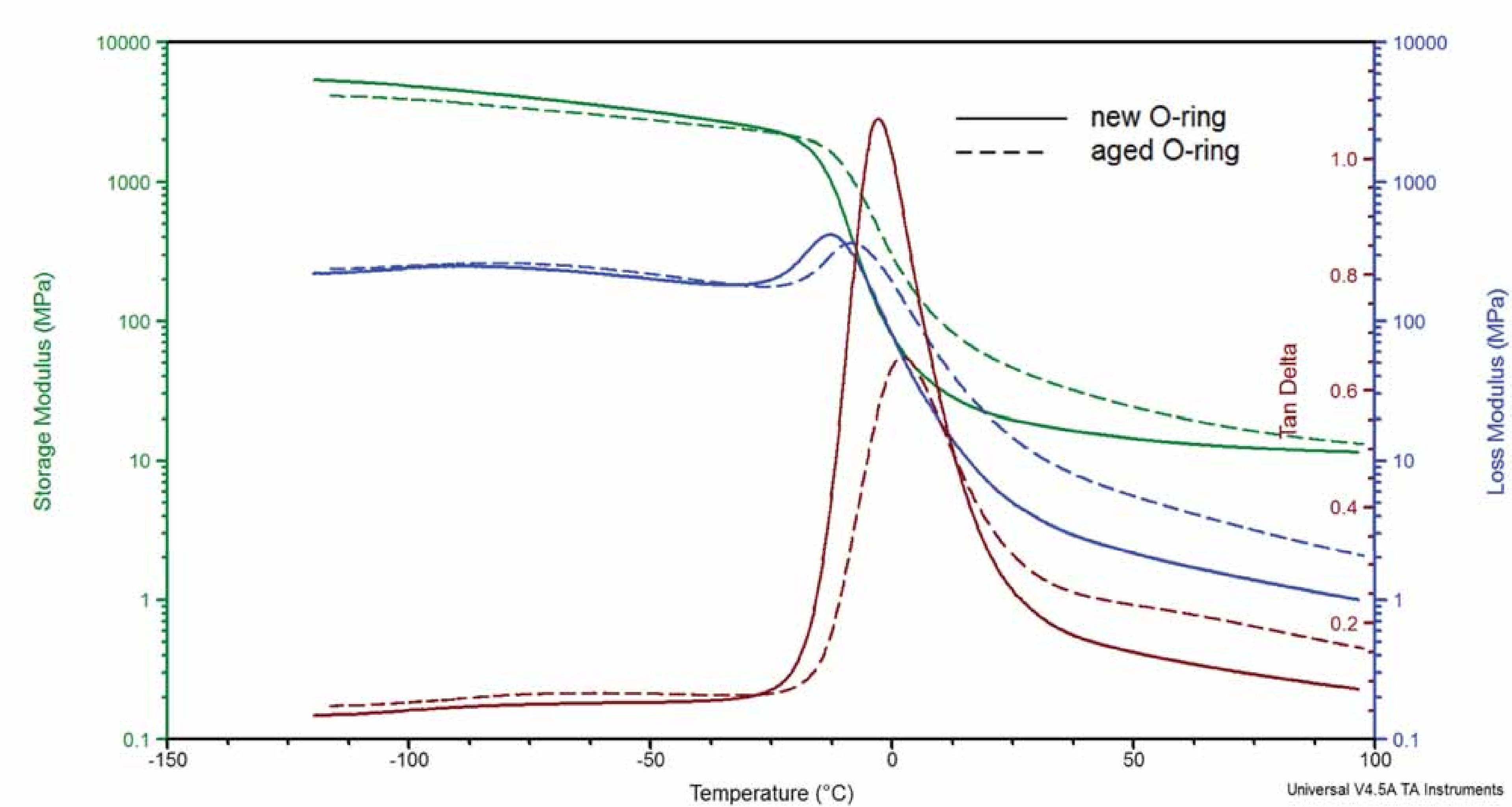


Effect of Filler on Modulus

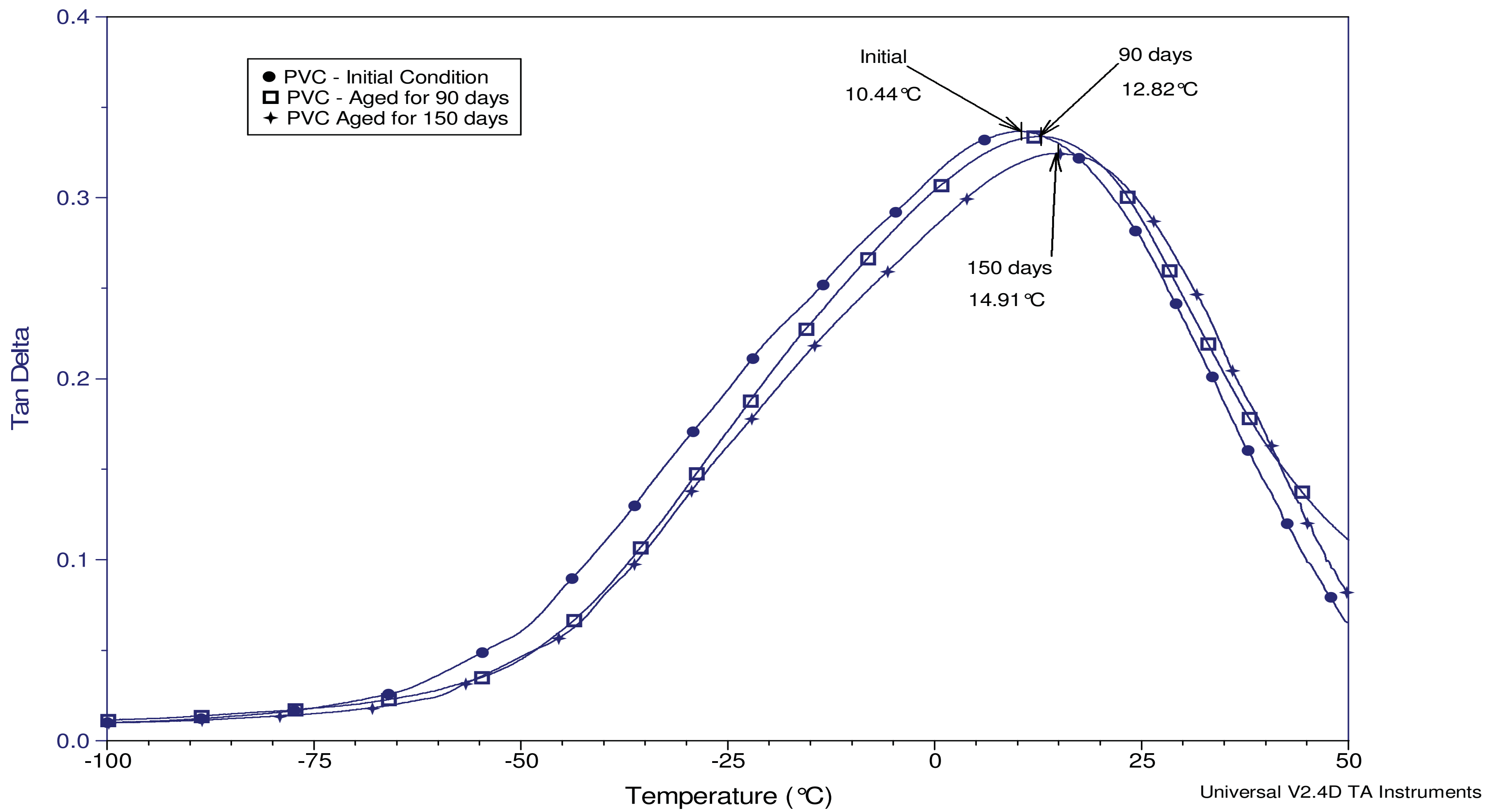


Nielson, L. E., Wall, R. A., and Richmond, P. G., **Soc. Plastics Eng. J.**, **11**, 22 (1966)

Effect of Aging on Elastomer O Rings



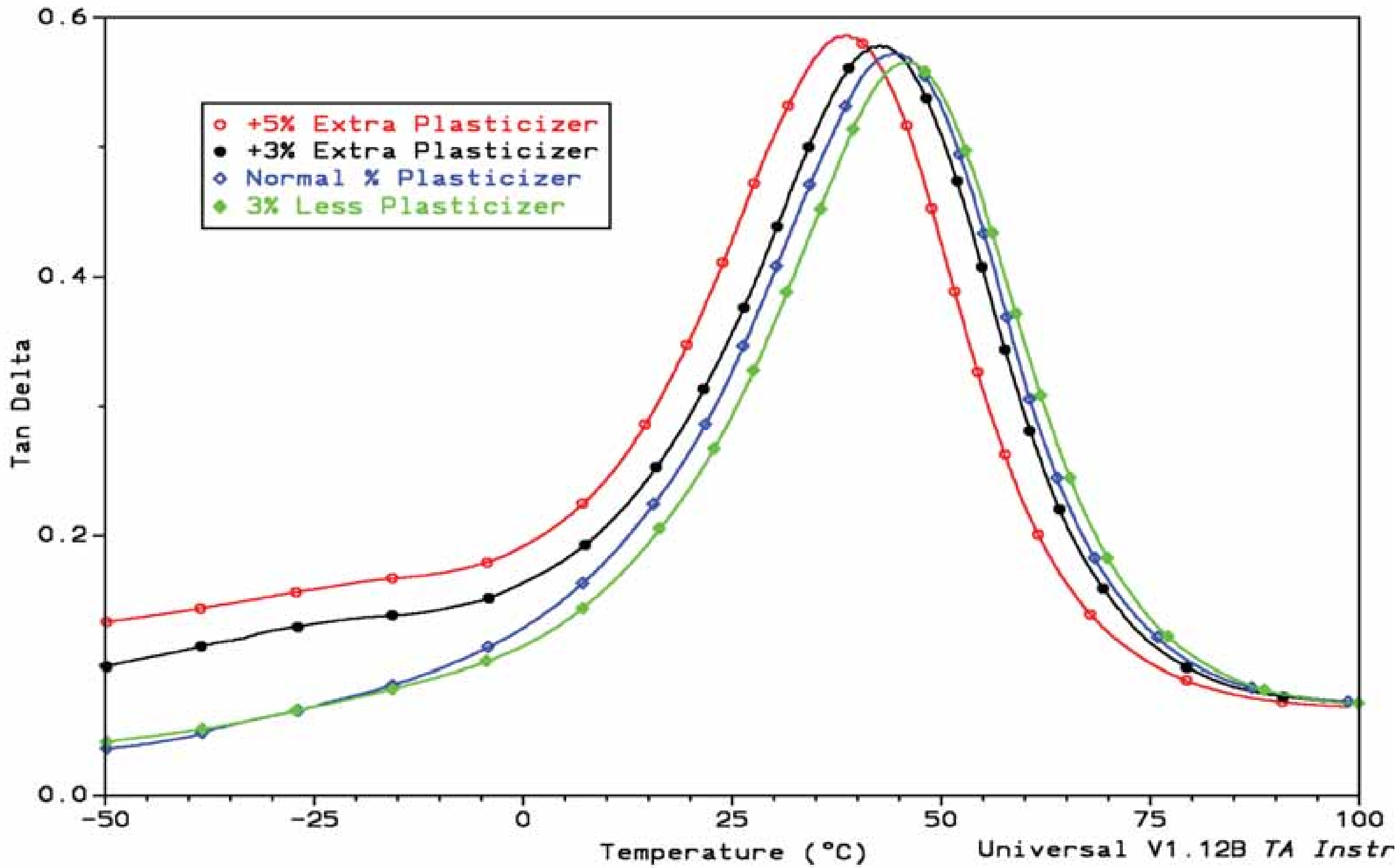
Effect of Aging on T_g of PVC Roofing Membrane



Effect of Plasticizer

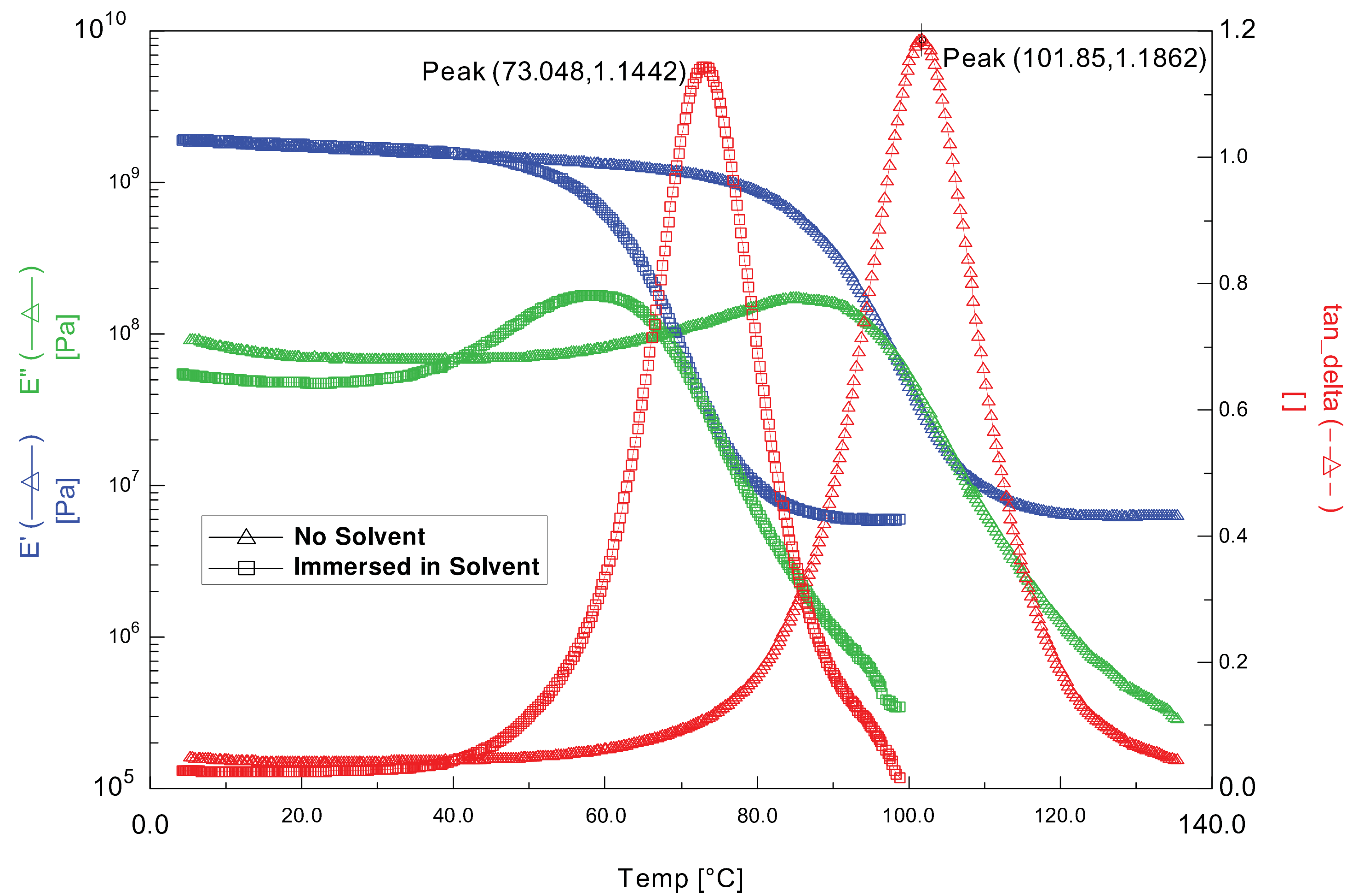
- *Plasticizers* are generally low molecular weight organic additives which are used to soften rigid polymers
- Typically added to a polymer for two reasons:
  1. To lower the T_g to make a rigid polymer become soft
  2. To make the polymer easier to process.
- *Plasticizers* make it easier for a polymer to change molecular conformation.
- Therefore plasticizers will have the effect of:
  1. Lowering the glass transition temperature and
  2. Broadening the tan δ peak

Effect of Plasticizer on Vinyl Flooring

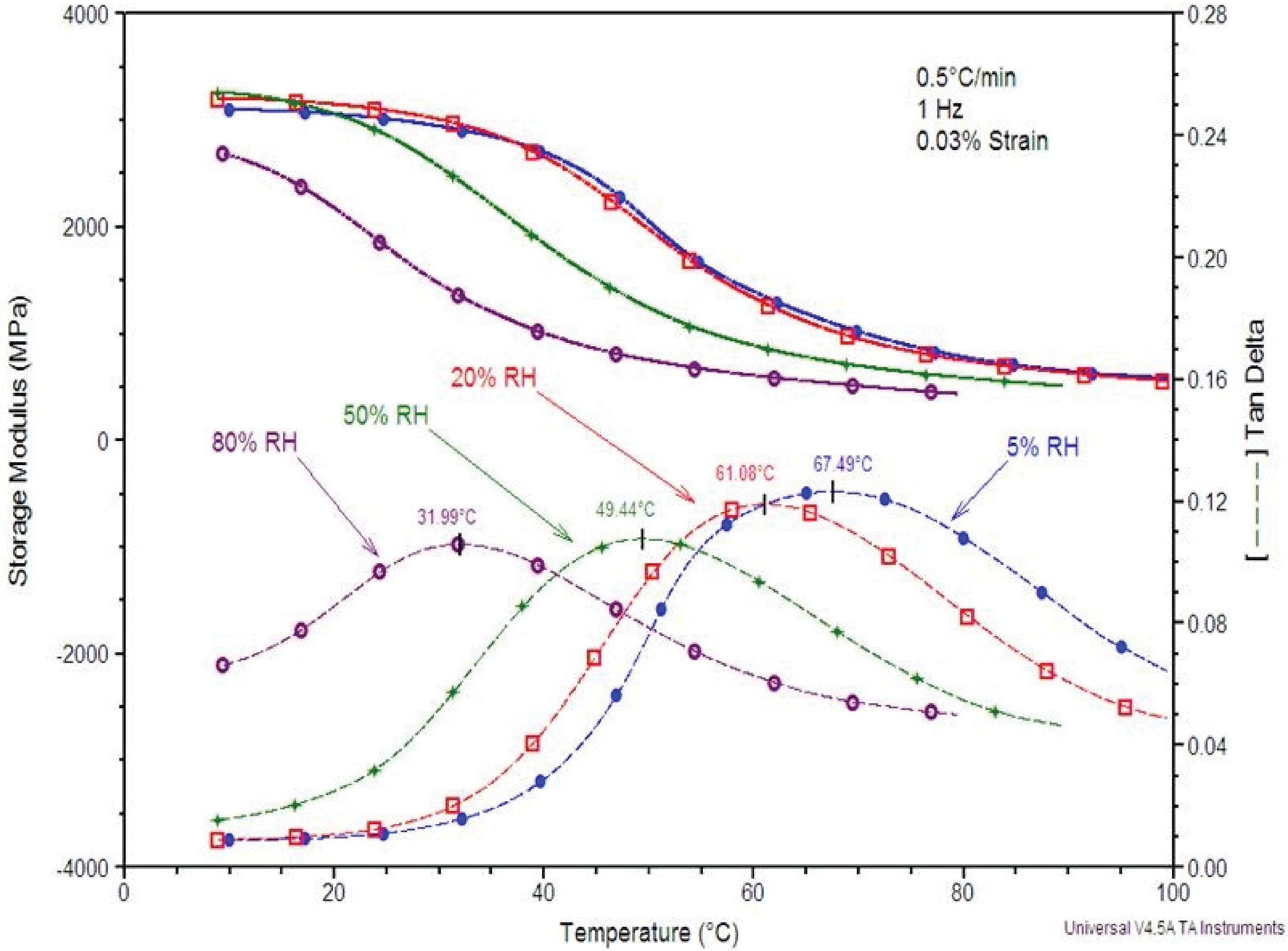


Effect of Moisture

- Automotive coating measured under dry vs. wet conditions

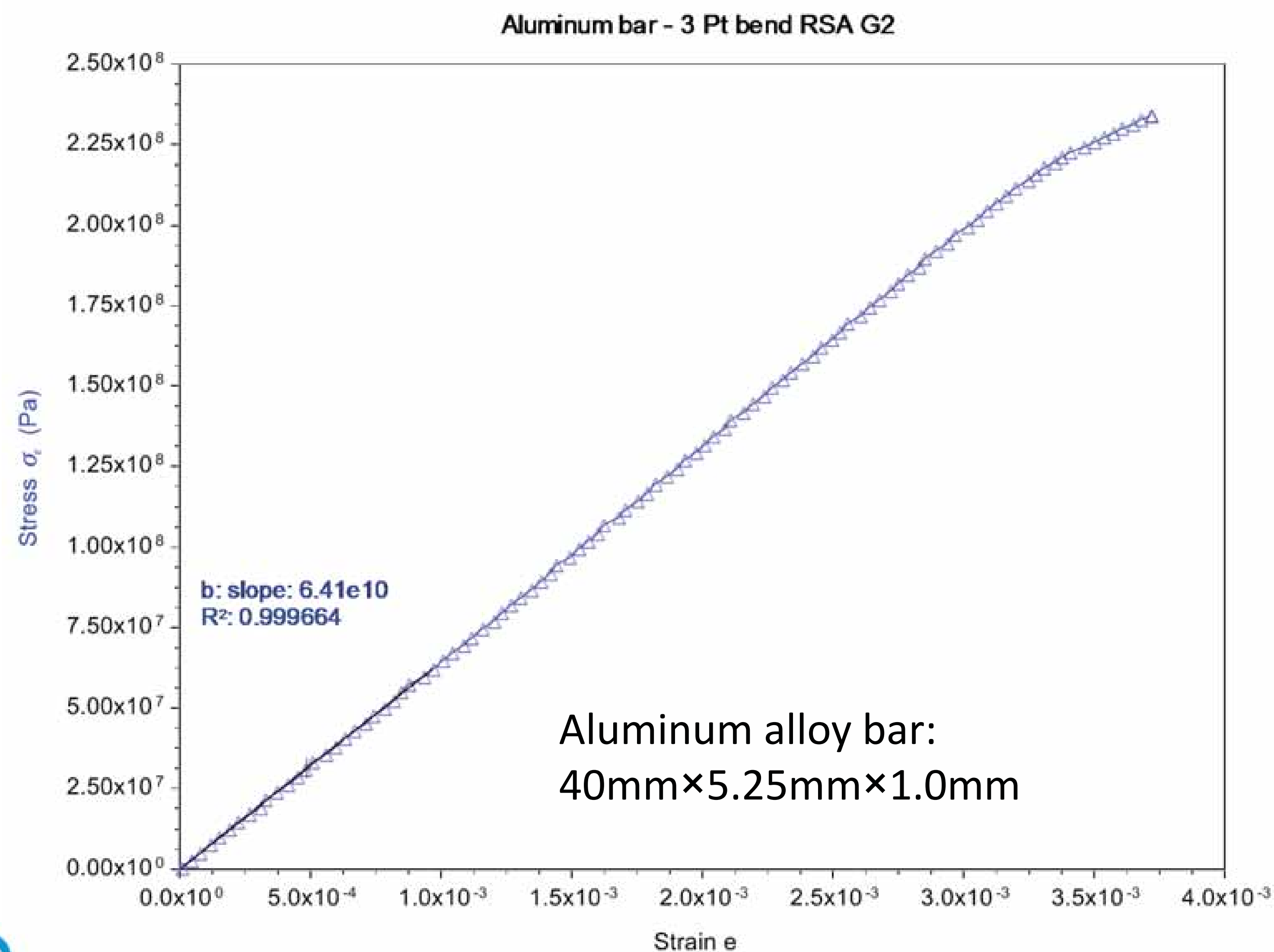


Nylon 6: Temperature Scans with Humidity Control

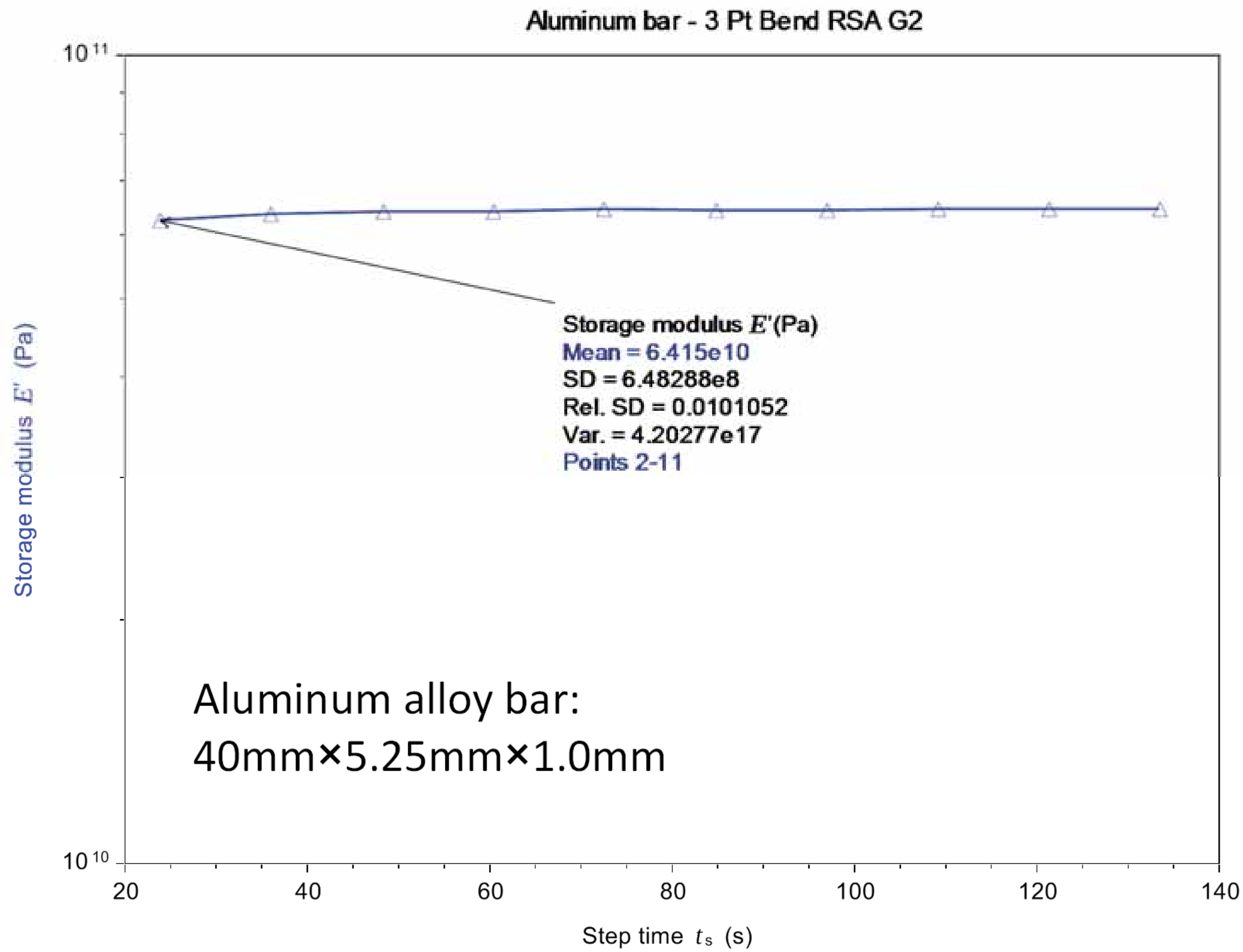


Other (Axial) – RSA G2

Constant Linear Rate: 0.1mm/min, 2 mm max gap change

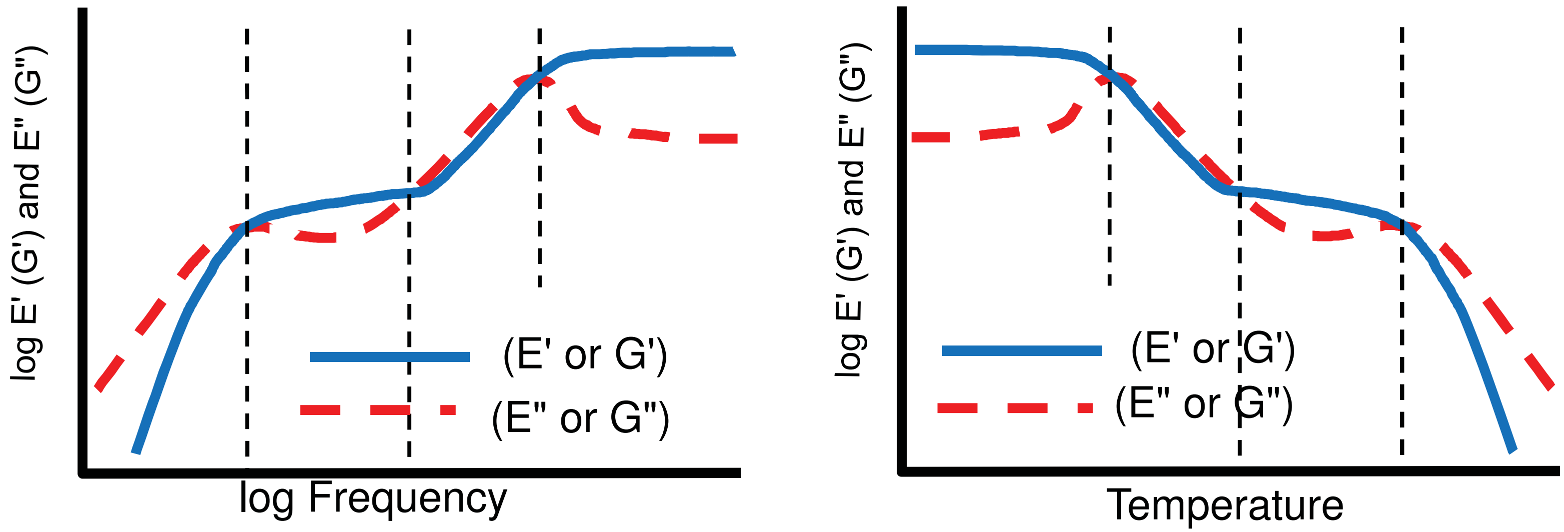


Time Sweep – RSA G2 (0.05% at 1 Hz)



Time-Temperature Superposition (TTS)

Time and Temperature Relationship



- Linear viscoelastic properties are both time-dependent and temperature-dependent
- Some materials show a time dependence that is proportional to the temperature dependence
  - Decreasing temperature has the same effect on viscoelastic properties as increasing the frequency
- For such materials, changes in temperature can be used to “re-scale” time, and predict behavior over time scales not easily measured

Time Temperature Superpositioning Benefits

- TTS can be used to extend the frequency beyond the instrument's range
- Creep TTS or Stress Relaxation TTS can predict behavior over longer times than can be practically measured
- Can be applied to amorphous, non modified polymers
- Material must be thermo-rheological simple
  - One in which all relaxations times shift with the same shift factor a_T

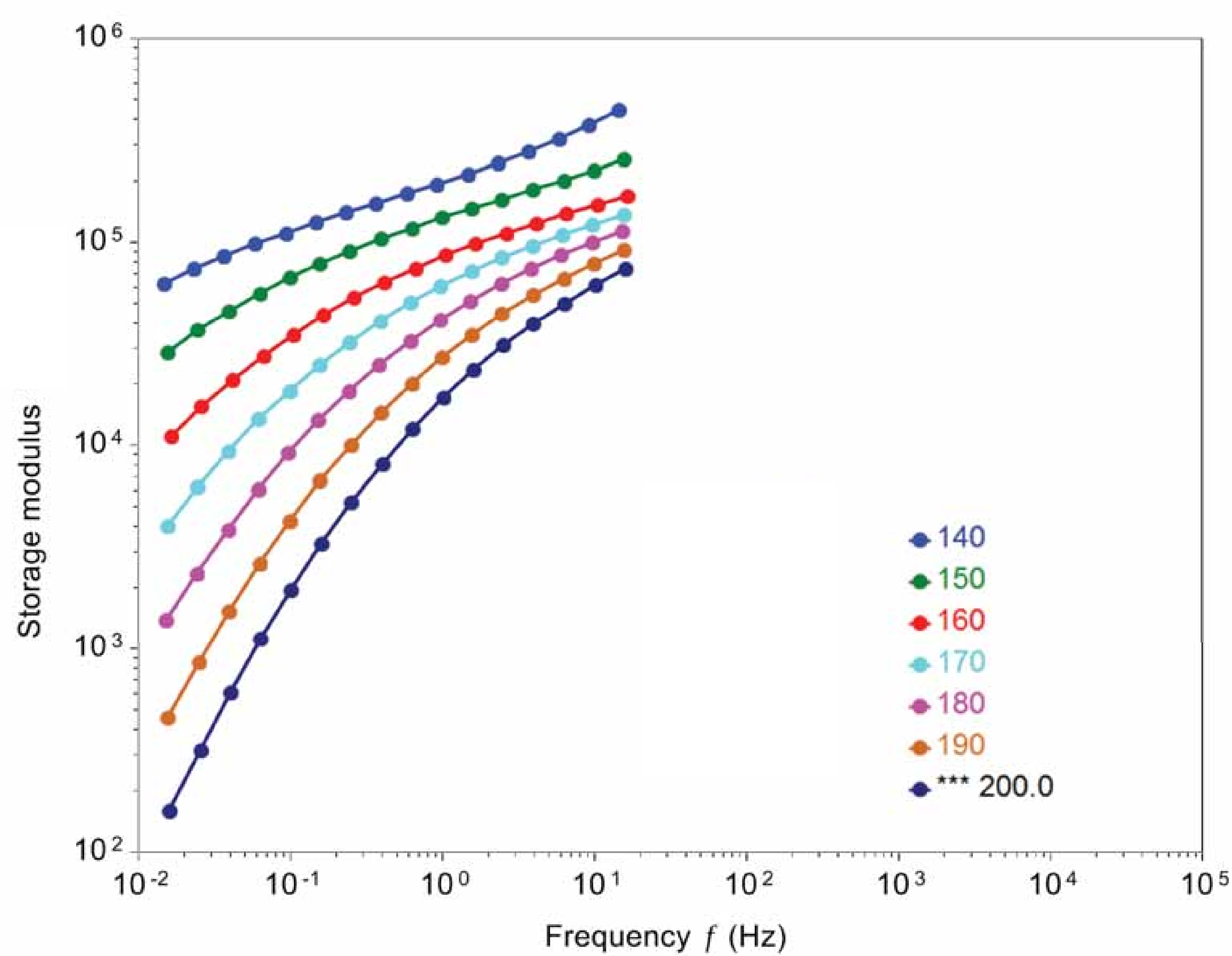
When Not to Use TTS

- If crystallinity is present, especially if any melting occurs in the temperature range of interest
- The structure changes with temperature
  - Cross linking, decomposition, etc.
  - Material is a block copolymer (TTS may work within a limited temperature range)
  - Material is a composite of different polymers
  - Viscoelastic mechanisms other than configuration changes of the polymer backbone
    - e.g. side-group motions, especially near the Tg
    - Dilute polymer solutions
    - Dispersions (wide frequency range)
    - Sol-gel transition

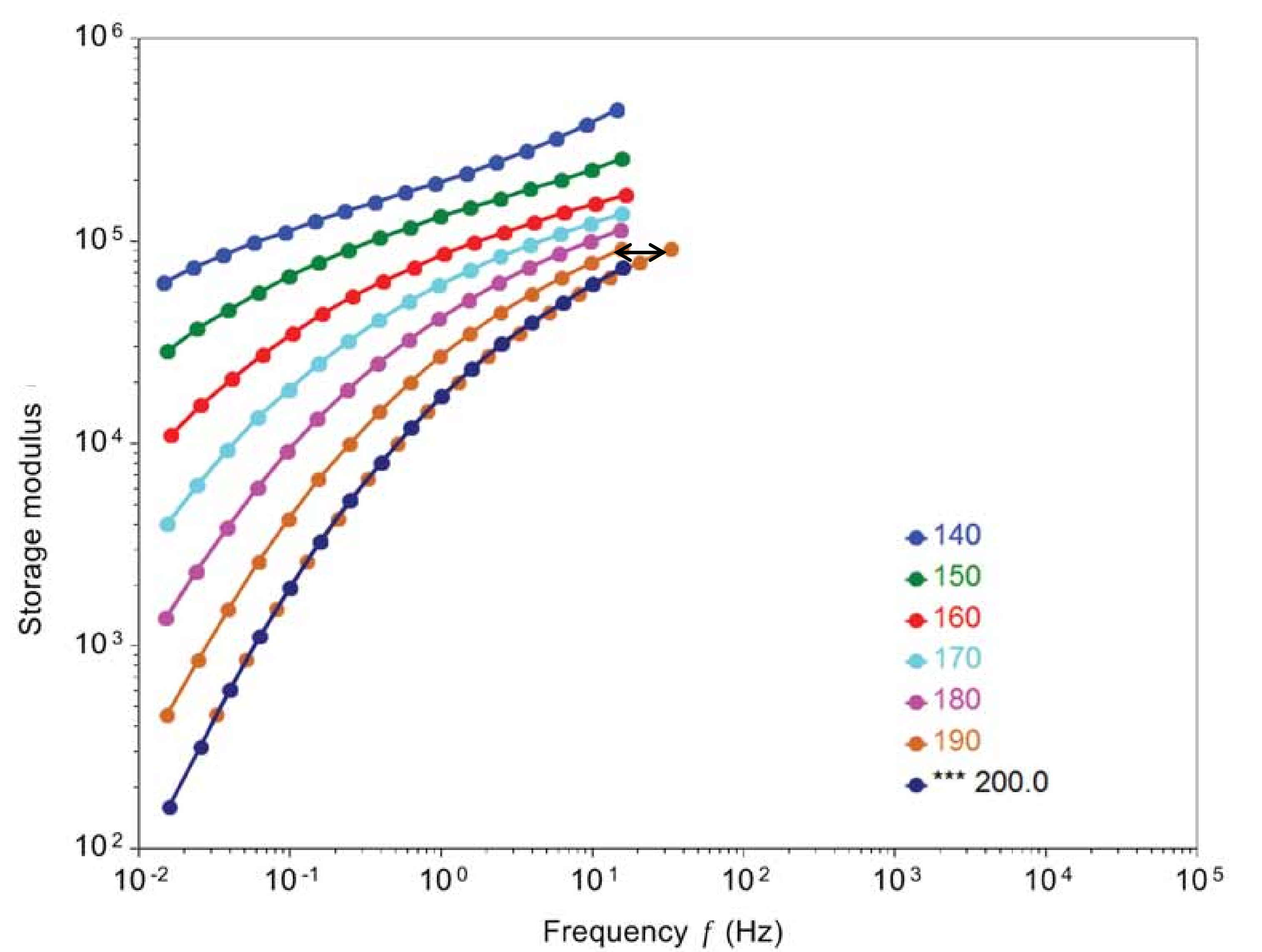
Guidelines for TTS

- Decide first on the Reference Temperature: T_0. What is the use temperature?
- If you want to obtain information at higher frequencies or shorter times, you will need to conduct frequency (stress relaxation or creep) scans at temperatures lower than T_0.
- If you want to obtain information at lower frequencies or longer times, you will need to test at temperatures higher than T_0.
- Good idea to scan material over temperature range at single frequency to get an idea of modulus-temperature and transition behavior.

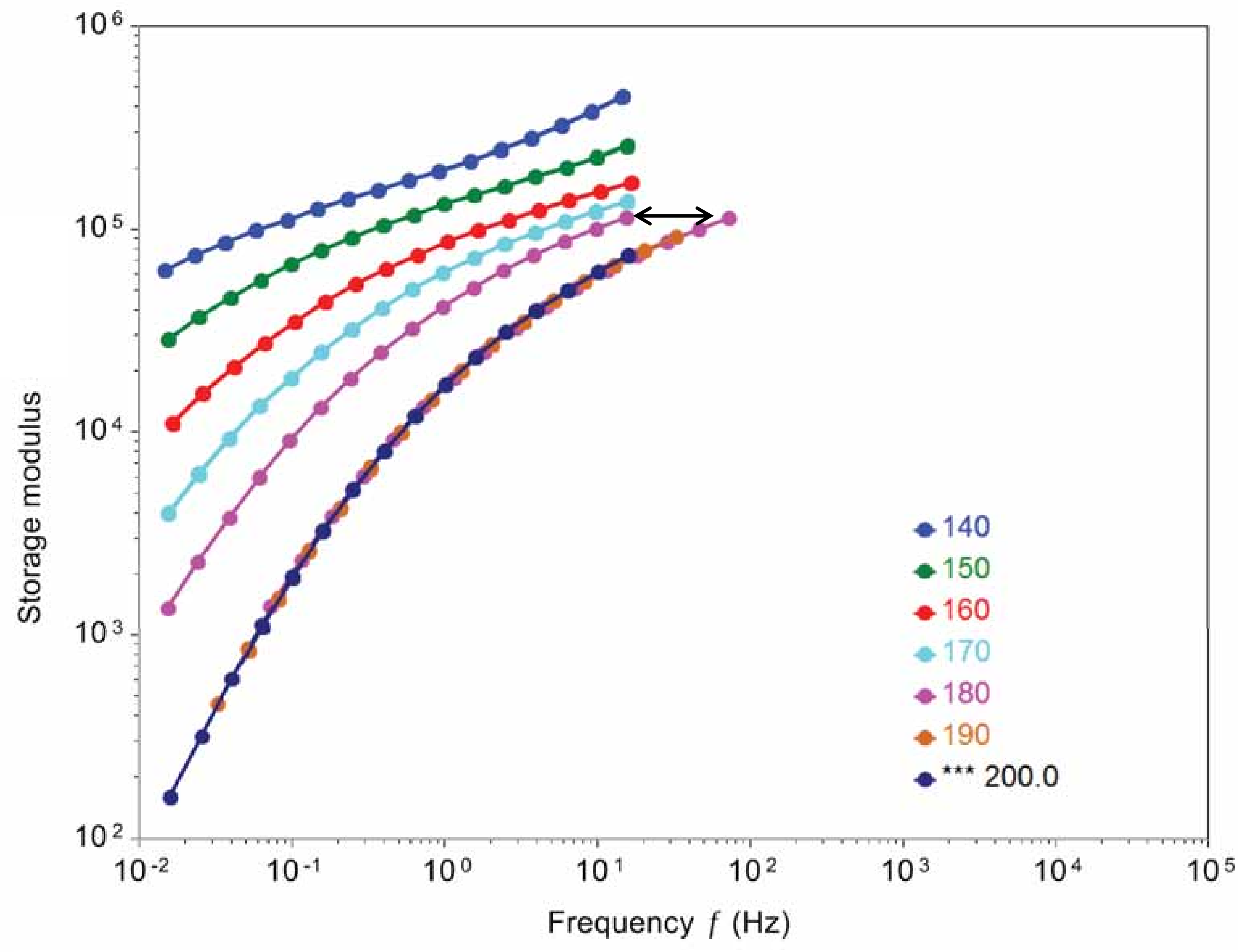
TTS Shifting



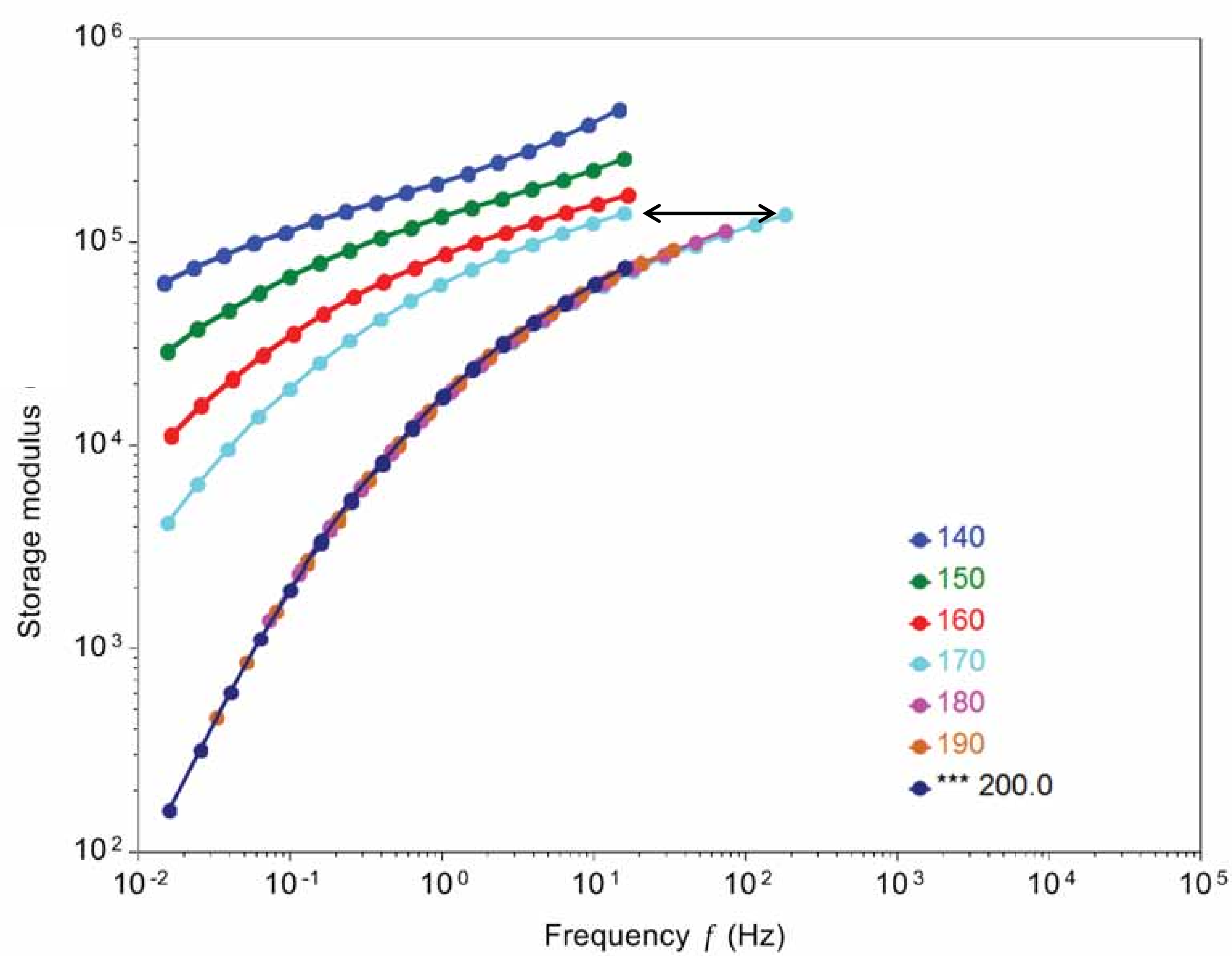
TTS Shifting



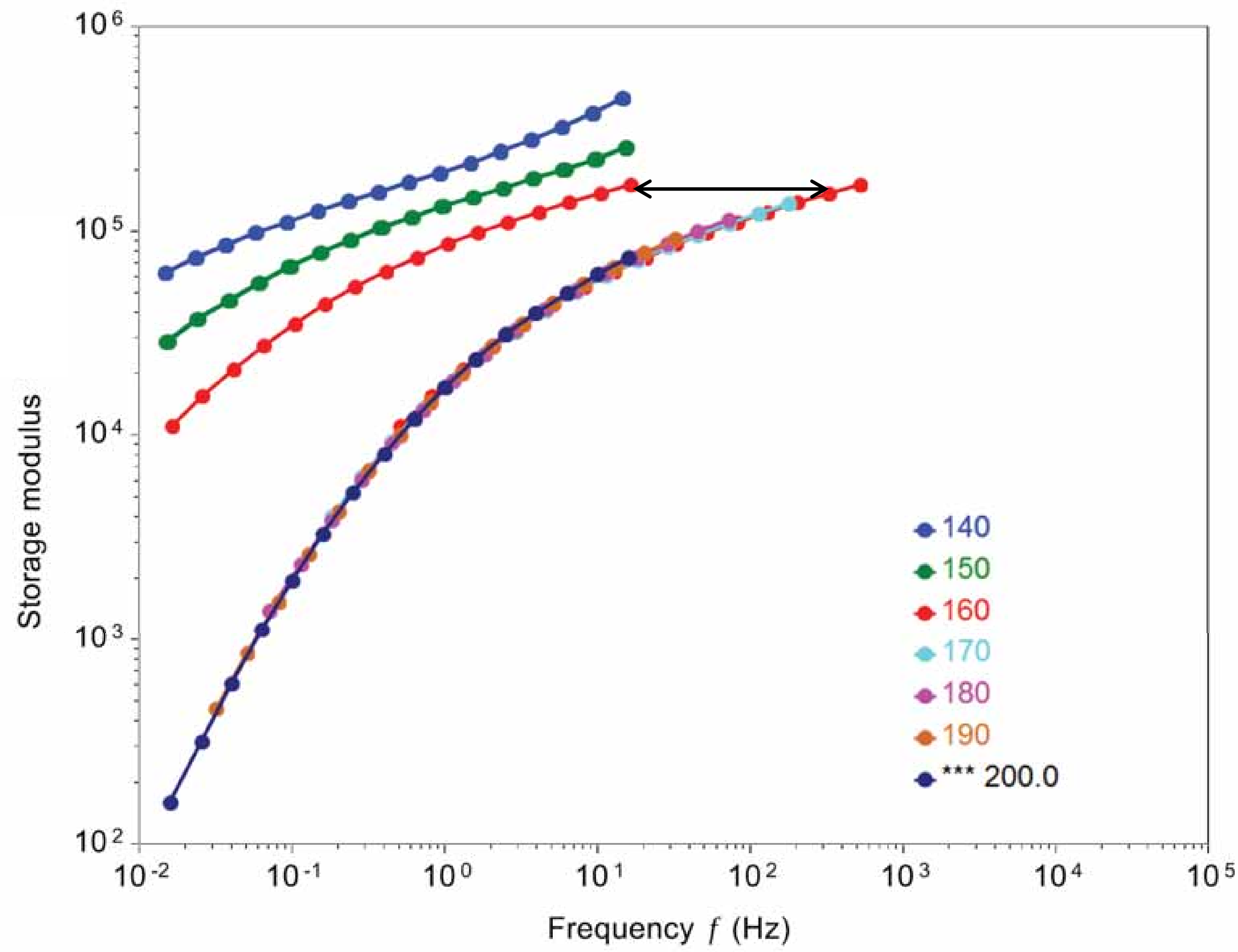
TTS Shifting



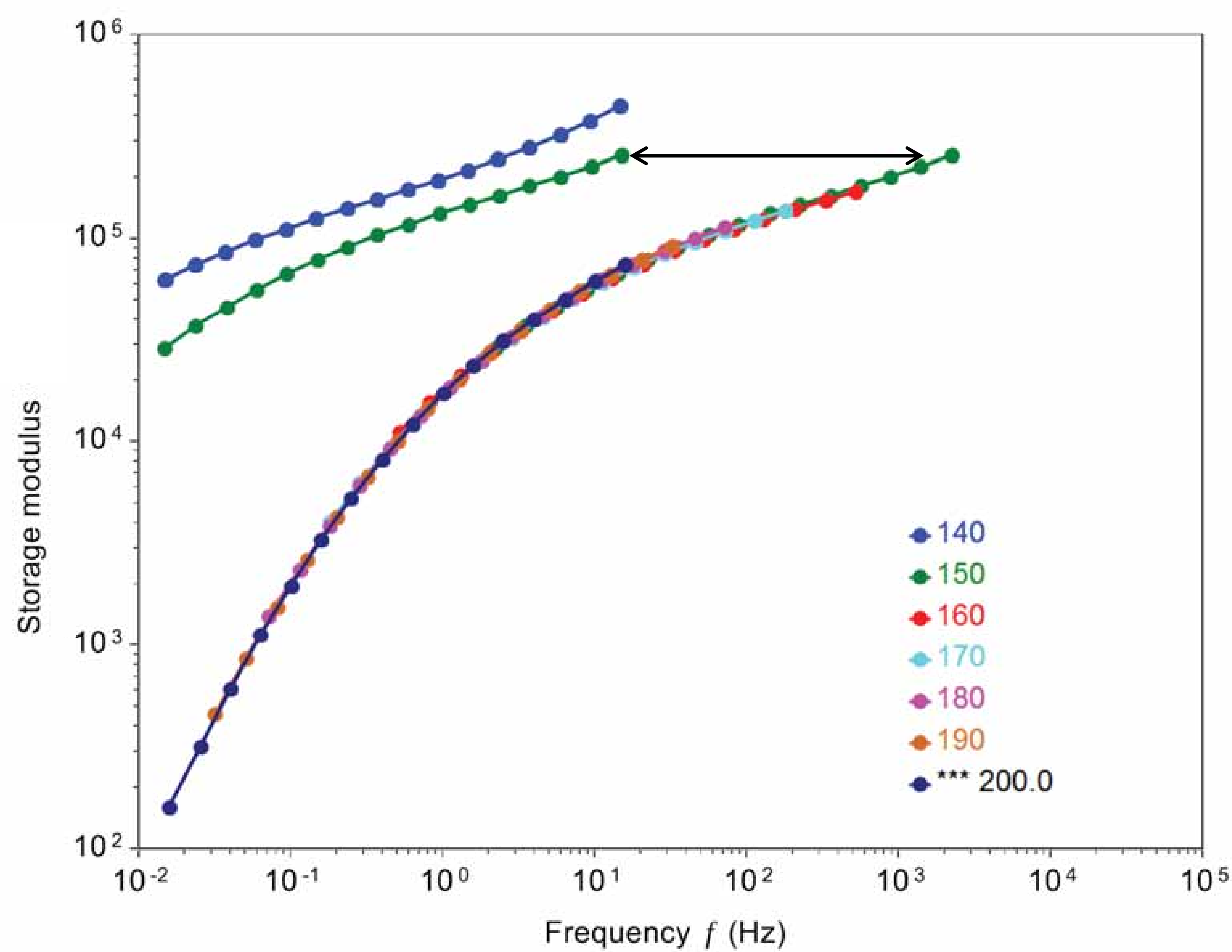
TTS Shifting



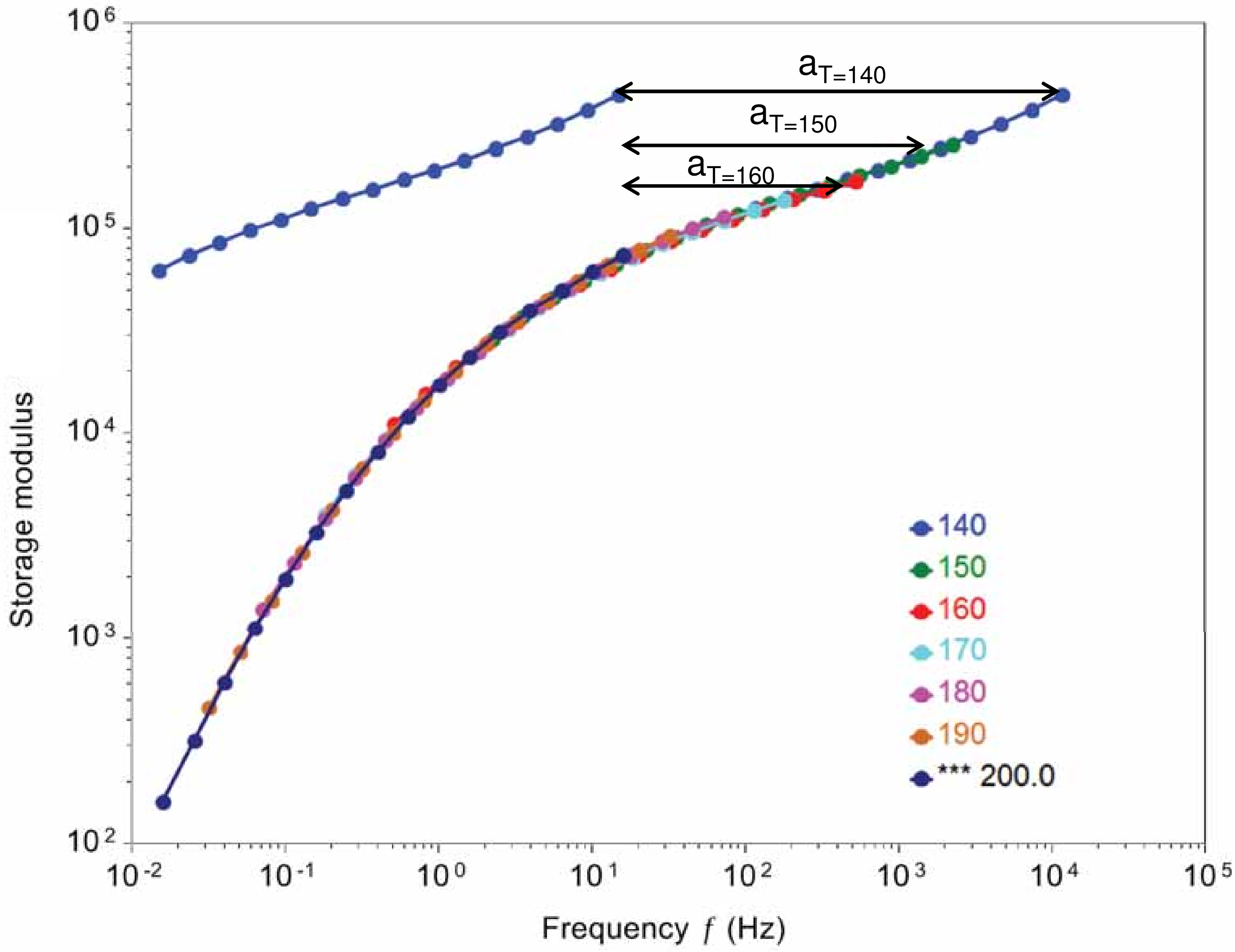
TTS Shifting



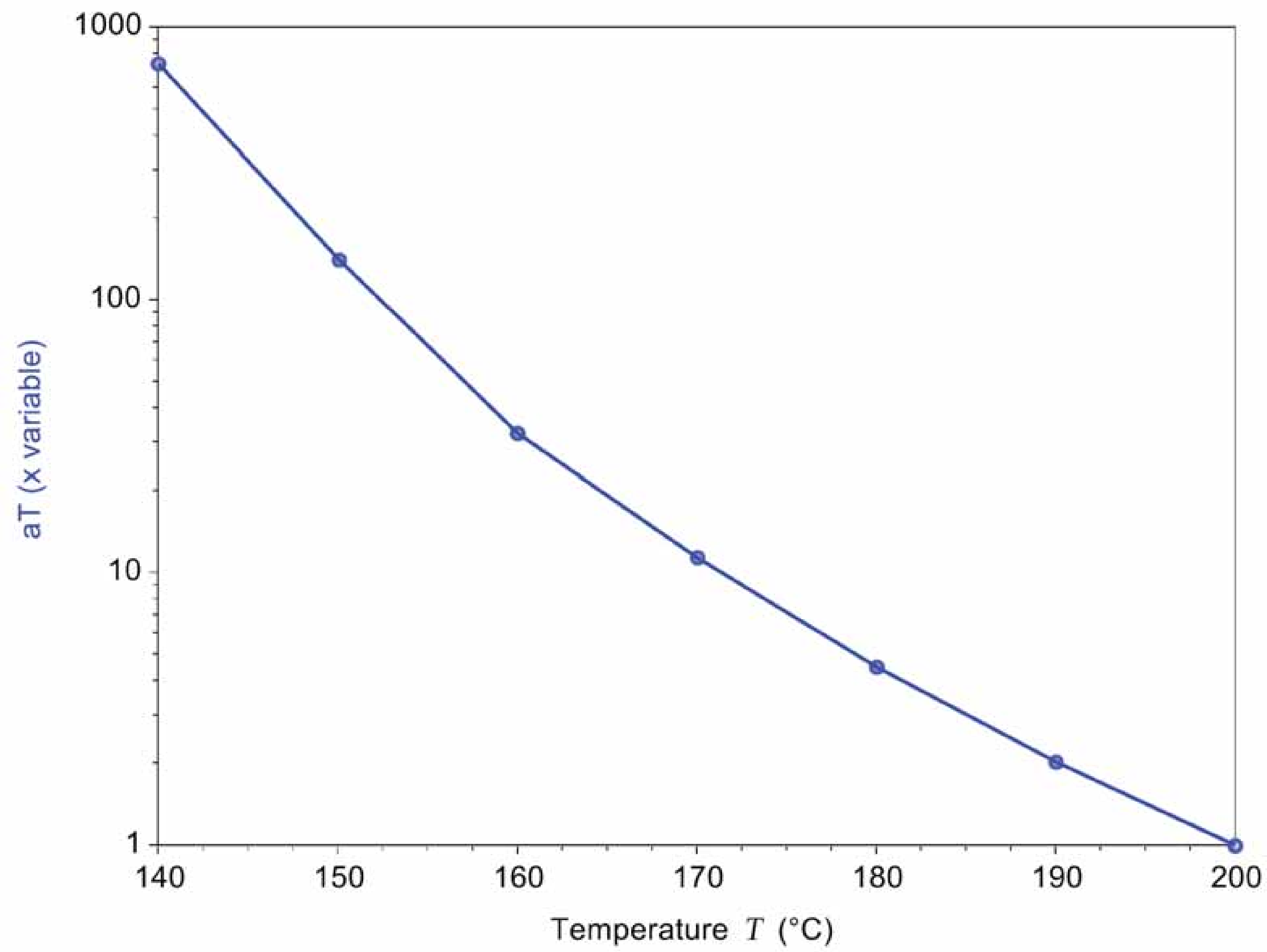
TTS Shifting



TTS Shifting



Shift Factors a_T vs Temperature



Shift Factors: WLF Equation

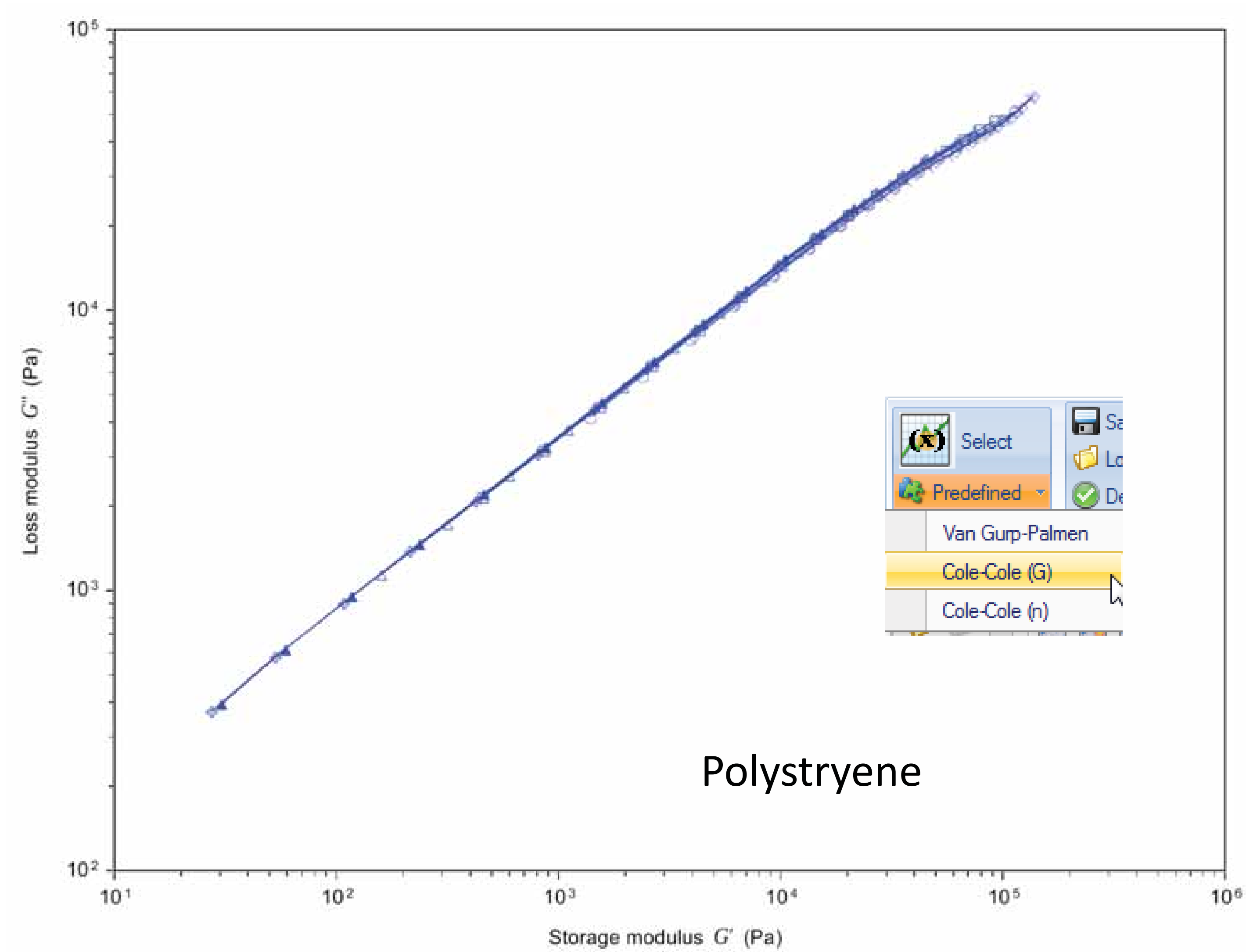
- Master Curves can be generated using shift factors derived from the Williams, Landel, Ferry (WLF) equation
    - $\log a_T = -c_1(T-T_0)/c_2+(T-T_0)$
  - a_T = temperature shift factor
  - T_0 = reference temperature
  - c_1 & c_2 = constants from curve fitting
    - Generally, c_1=17.44 & c_2=51.6 when $T_0 = T_g$

When not to use the WLF Equation

- Sometimes you shouldn't use the WLF equation (even if it appears to work)
- If $T > T_g + 100\,°C$
- If $T < T_g$ and polymer is not elastomeric
- If temperature range is small, then c_1 & c_2 cannot be calculated precisely
- In these cases, the Arrhenius form is usually better
  - $\ln a_T = (E_a/R)(1/T - 1/T_0)$

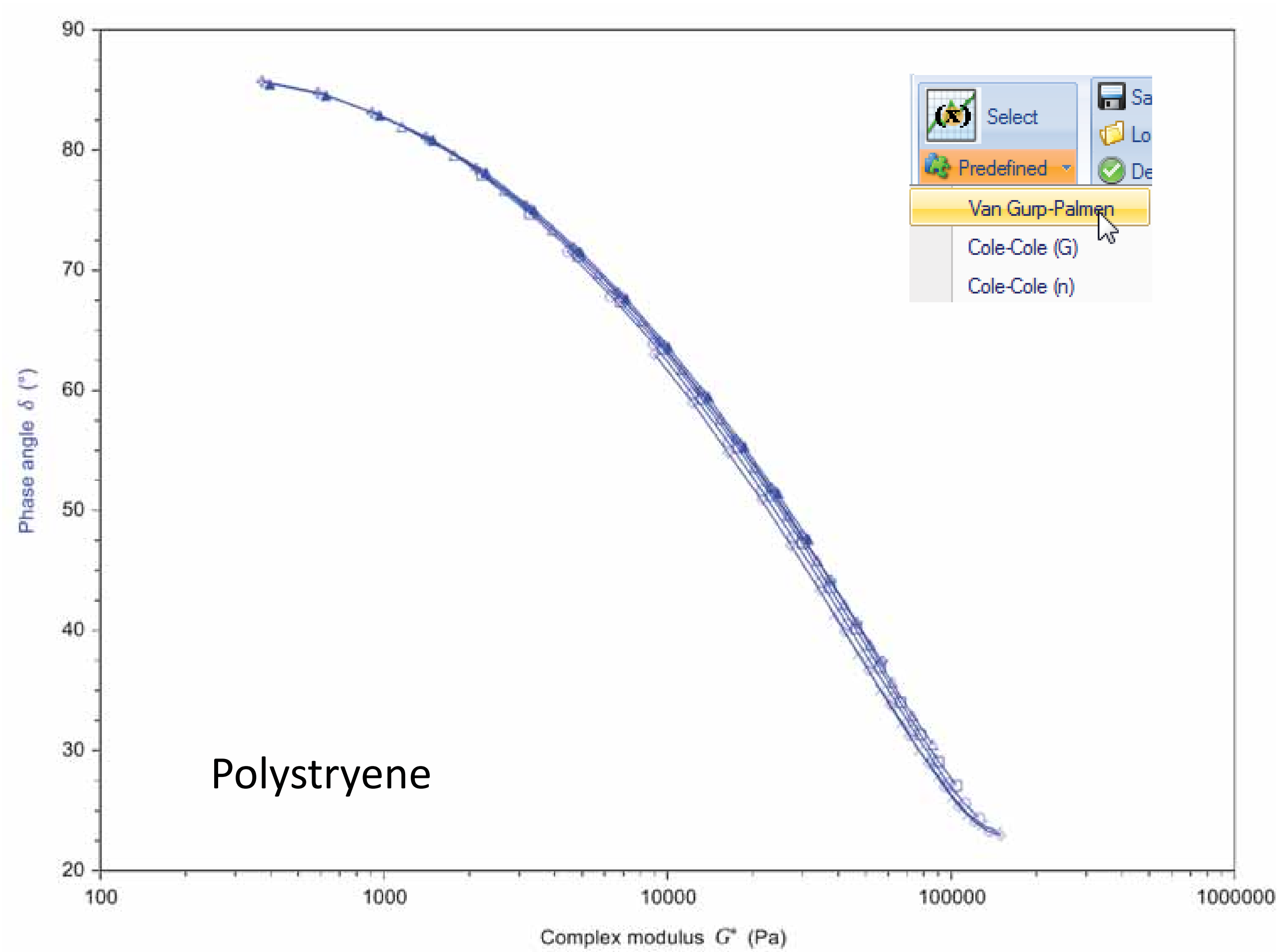
- a_T = temperature shift factor
- E_a = Apparent activation energy
- T_0 = reference temperature
- T = absolute temperature
- R = gas constant
- E_a = activation energy

Verify Data for TTS



Polystryene

Verify Data for TTS



References for TTS

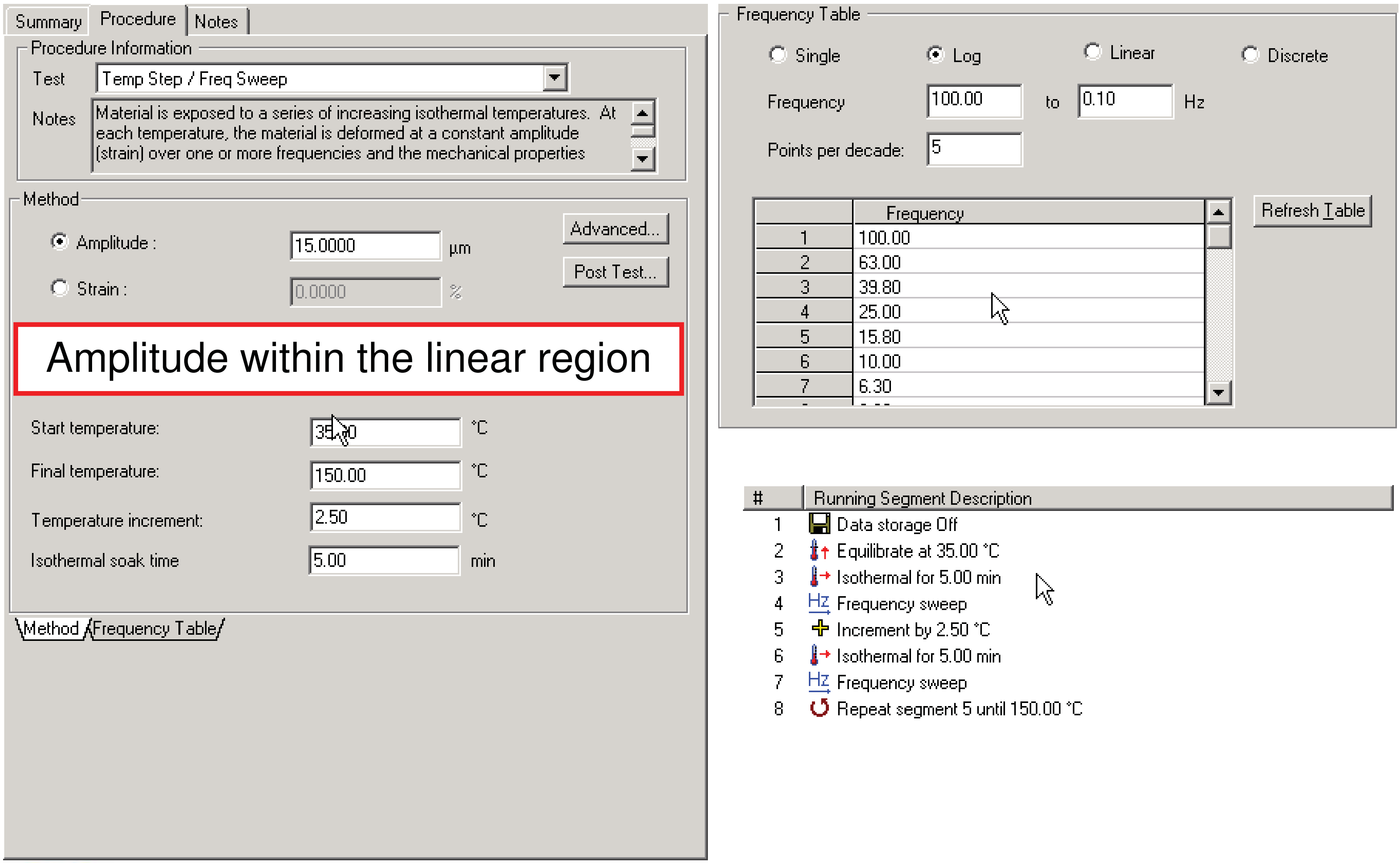
1) Ward, I.M. and Hadley, D.W., "*Mechanical Properties of Solid Polymers*", Wiley, 1993, Chapter 6.

2) Ferry, J.D., "*Viscoelastic Properties of Polymers*", Wiley, 1970, Chapter 11.

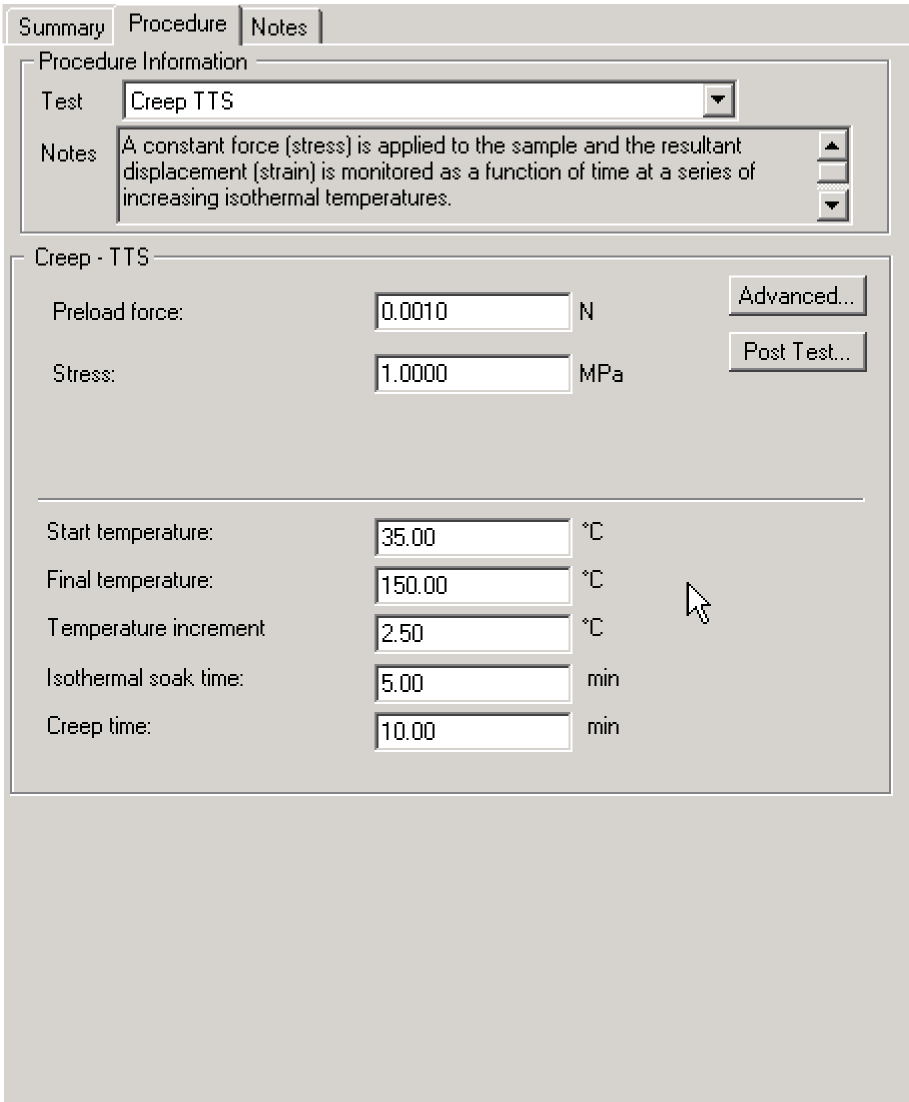
3) Plazek, D.J., "*Oh, Thermorheological Simplicity, wherefore art thou?*" Journal of Rheology, vol 40, 1996, p987.

4) Lesueur, D., Gerard, J-F., Claudy, P., Letoffe, J-M. and Planche, D., "*A structure related model to describe asphalt linear viscoelasticity*", Journal of Rheology, vol 40, 1996, p813.

Running Dynamic TTS on Q800

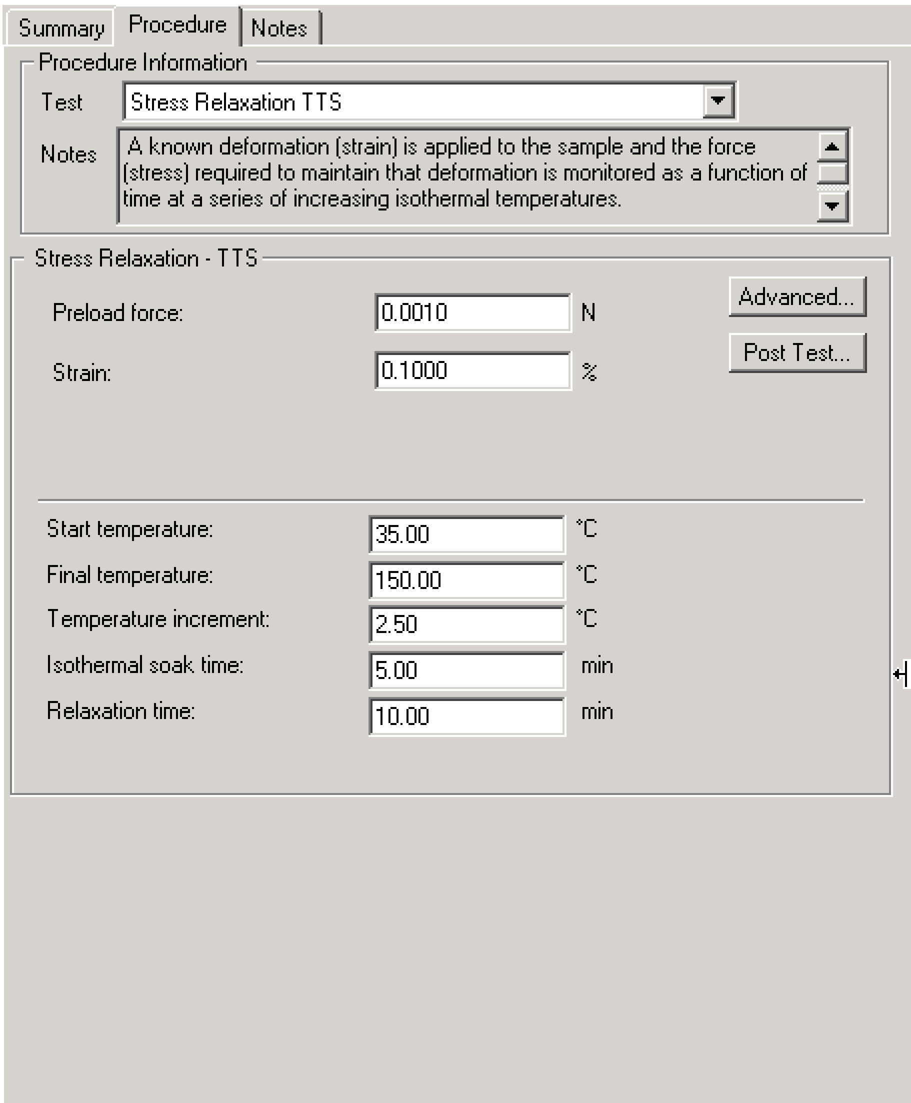


Running Creep TTS on Q800



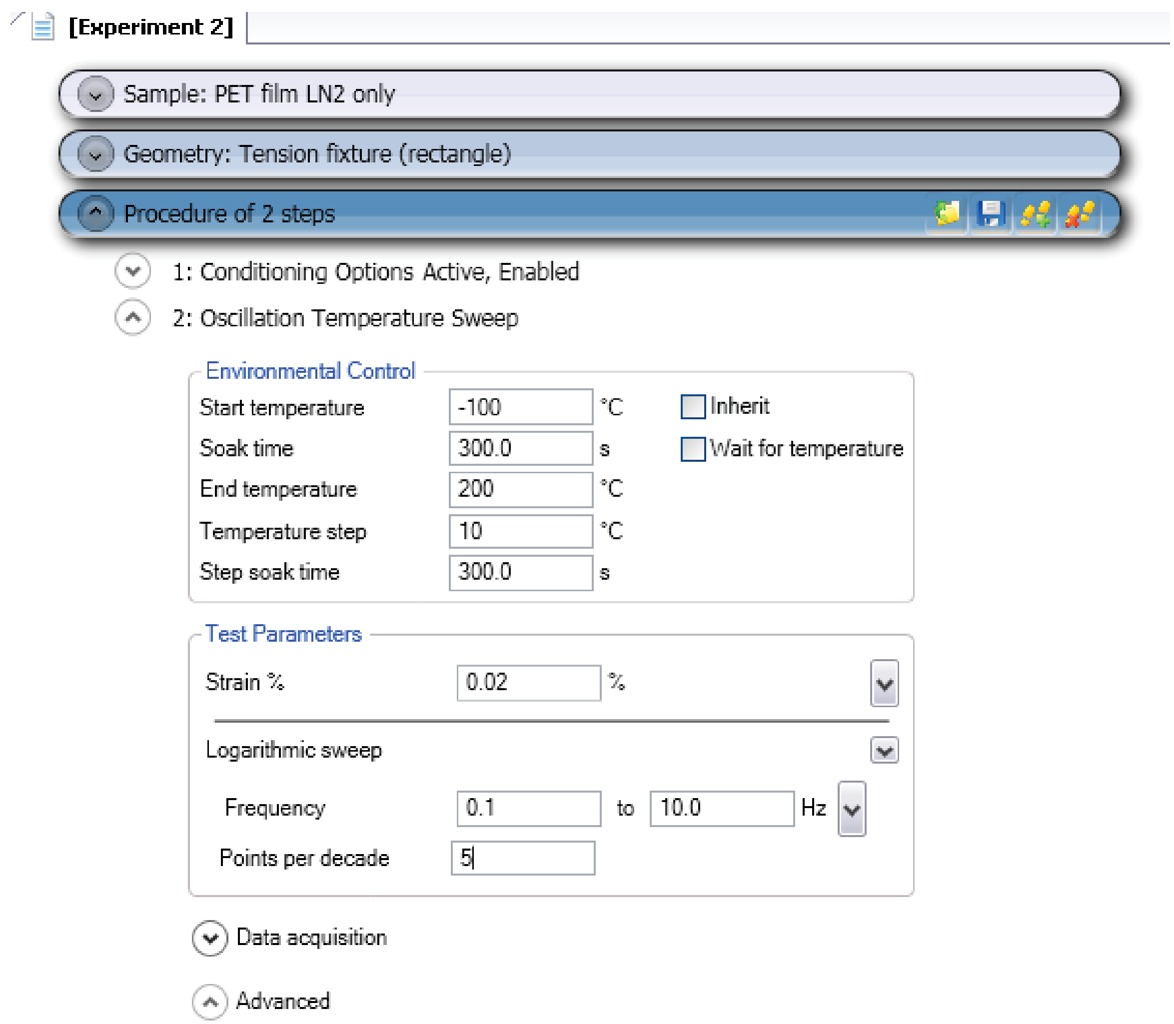
#	Running Segment Description
1	Data storage Off
2	Equilibrate at 35.00 °C
3	Isothermal for 5.00 min
4	Data storage On
5	Displace 10.00 min recover 0.00 min
6	Data storage Off
7	Increment by 2.50 °C
8	Repeat segment 3 until 150.00 °C

Running Stress Relaxation TTS on Q800



#	Running Segment Description
1	Data storage Off
2	Equilibrate at 35.00 °C
3	Isothermal for 5.00 min
4	Data storage On
5	Displace 10.00 min recover 0.00 min
6	Data storage Off
7	Increment by 2.50 °C
8	Repeat segment 3 until 150.00 °C

Running Dynamic TTS on the RSA G2



Getting Started Manuals

TA Instruments
Q Series™ Manuals

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

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

Instrument & Accessory Manuals

Tzero® PDSC Getting Started Guide

DSC Q Series™ Getting Started Guide
- RCS Getting Started Guide
- LNCS Getting Started Guide
- PCA Getting Started Guide
- DSC Pressure Cell Getting Started Guide
- DSC High Pressure Capsule Kit
- DSC High Volume Pan Kit
- DSC Circulator-Based Cooling System

TGA Q5000 IR Getting Started Guide

TGA Q Series™ Getting Started Guide
- TGA Hi-Res™ Option

DMA Q Series™ Getting Started Guide
- GCA Getting Started Guide
- DMA Humidity Accessory Getting Started

SDT Q Series™ Getting Started Guide

TMA Q Series™ Getting Started Guide
- MCA Getting Started Guide
- MCA70 Getting Started Guide

Q5000 SA Getting Started Guide

Software Manuals

Q Series™ Instrument Control Getting Started Guide

Universal Analysis Getting Started Guide

Advantage Integrity™ Getting Started Guide

Specialty Library Getting Started Guide

RMX File Utilities

Miscellaneous Documents

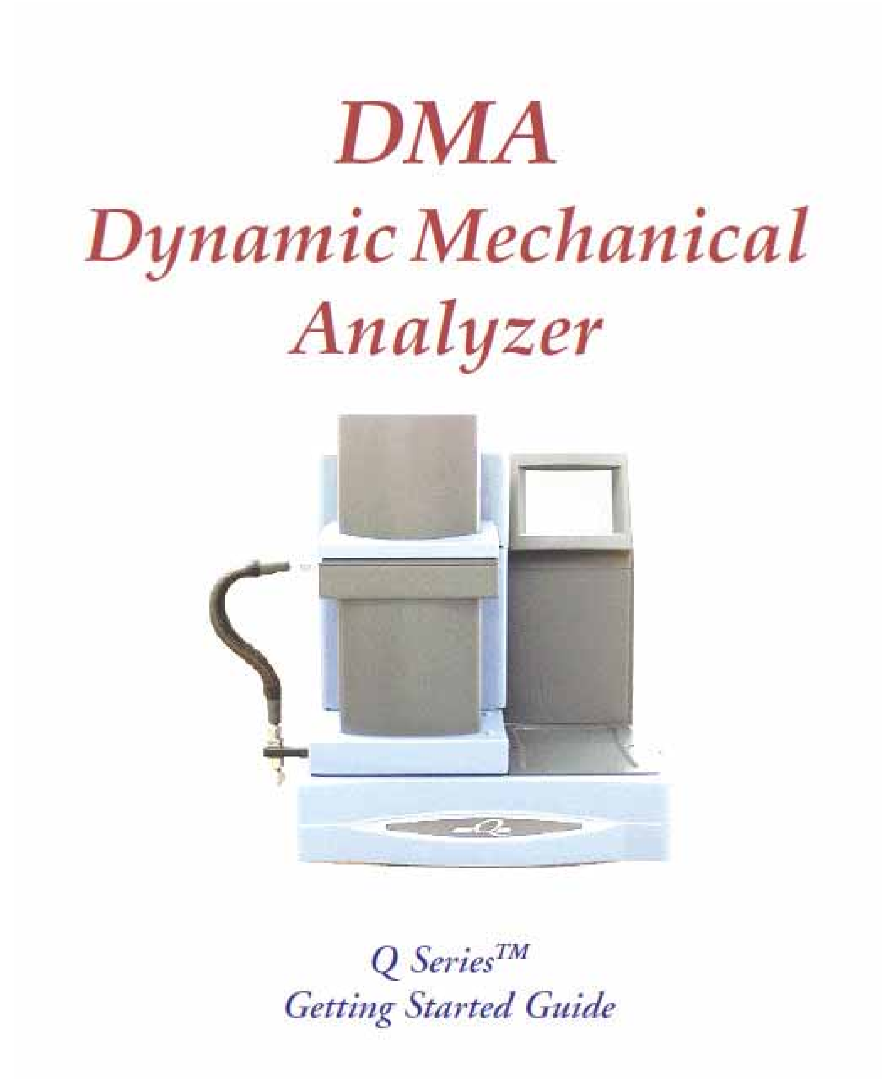
Installing/Updating Advantage™

Updating Q Series™ Instrument Software

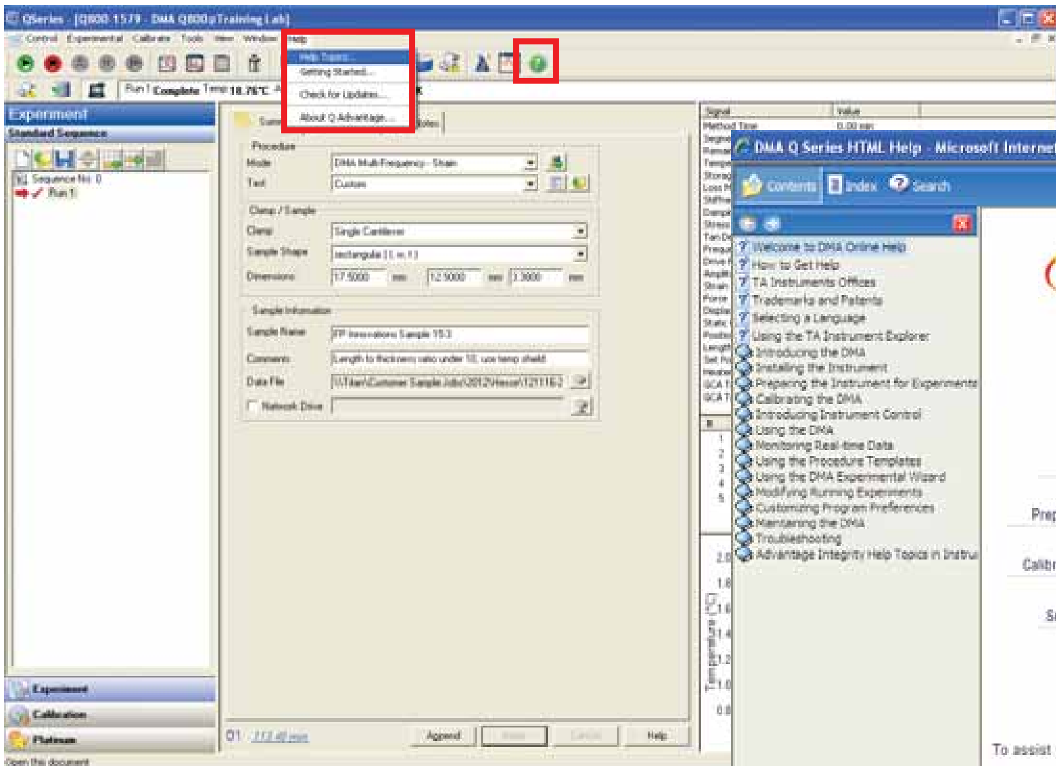
New Features in Advantage Q Series™

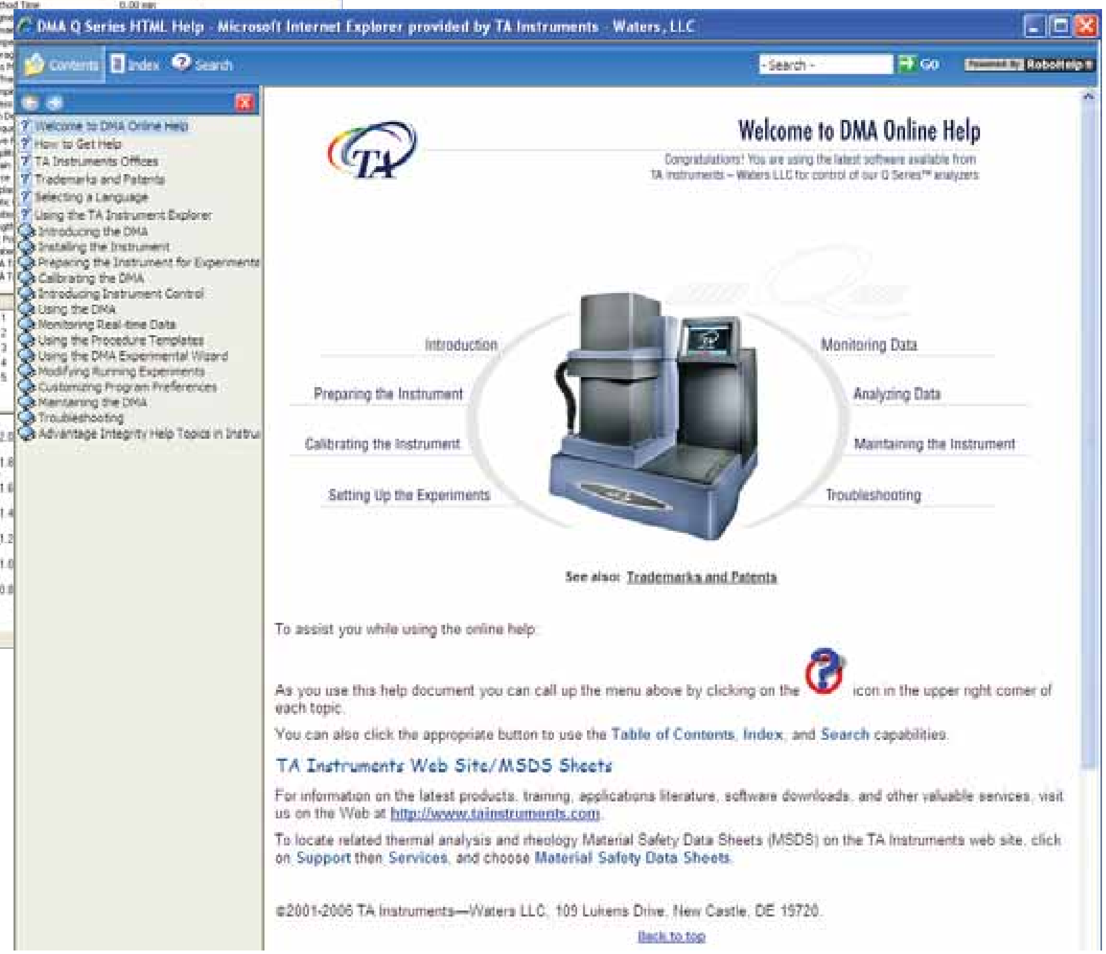
New Features in Advantage Integrity™

TA Update



Help Menu

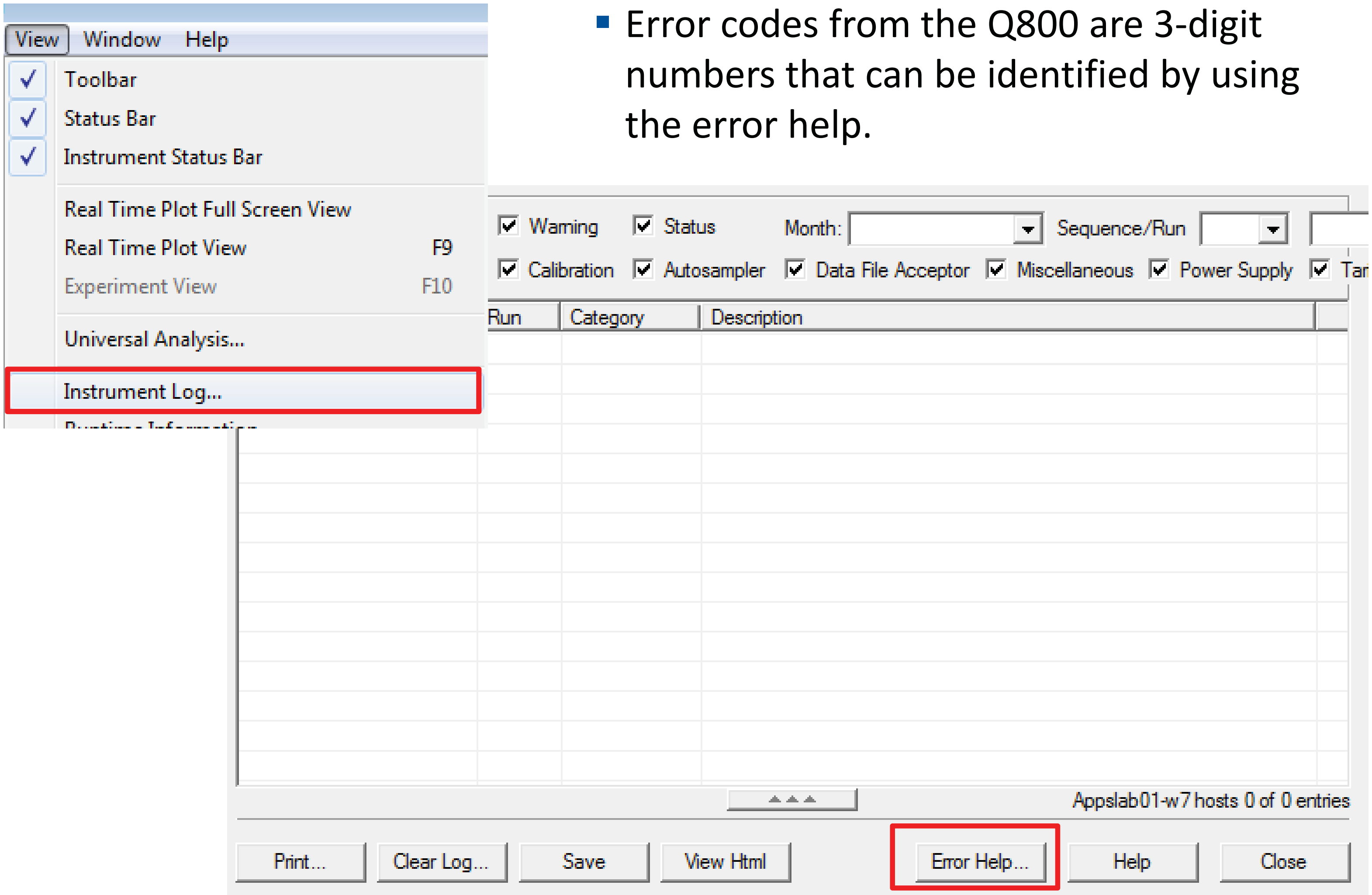




- Browse the Contents list or search using the Index tab.

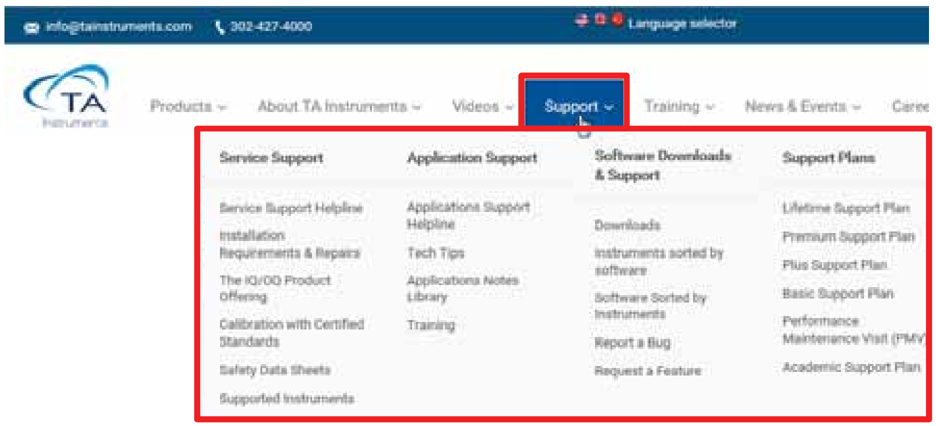
View Error Help (Q800)

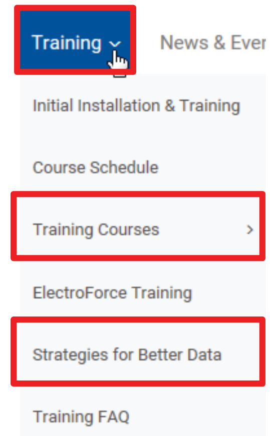
- Error codes from the Q800 are 3-digit numbers that can be identified by using the error help.



Instructional Videos

- From www.tainstruments.com click on Videos, Support or Training



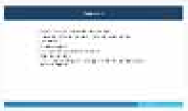


- Select Videos for TA Tech Tips, Webinars and Quick Start Courses

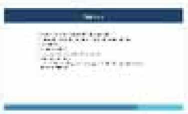
See also: https://www.youtube.com/user/TATechTips

Instructional Video Resources

Quickstart e-Training Courses



DMA Q800 Quickstart Course – Instrument and Experimental Setup



DMA Q800 – Analysis Quickstart

Universal Analysis QuickStart Course

Universal Analysis Advanced E-Training

Universal Analysis Custom Report

Web based e-Training Courses

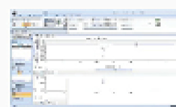
TA Instruments offers a variety of training opportunities via the Internet. e-Training opportunities include the following:

QUICKSTART e-TRAINING COURSES

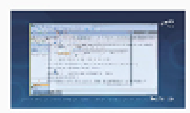
QuickStart e-Training courses are designed to teach a new user how to set up and run samples on their analyzers. These 60-90 minute courses are available whenever you are. These pre-recorded courses are available to anyone at no charge. Typically these courses should be attended shortly after installation.

Contact Us for Web based e-Training Courses

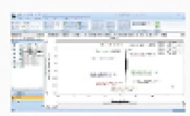
Search Results for "TRIOS"



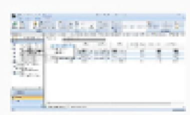
Discovery DSC – TRIOS Data Analysis



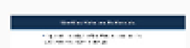
TRIOS Guardian — a tool to aid in 21 CFR 11 compliance



TRIOS – Analysis in Overlay



TRIOS – Analysis Reports



TRIOS – Cox Merz Transformation

Need DMA HELP?!?

- Check the manuals, help and error help.
- Contact the TA Instruments Rheology Hotline
  - **302-427-4070** M-F 8-4:30 ET
  - Email rheologysupport@tainstruments.com
- Call the TA Instruments Service Hotline
  - **302-427-4050** M-F 8-4:30 ET
- Call your local Technical or Service Representative
- Visit our Website www.tainstruments.com for training videos, TA Tech tips, application notes and much more!

Thank you!

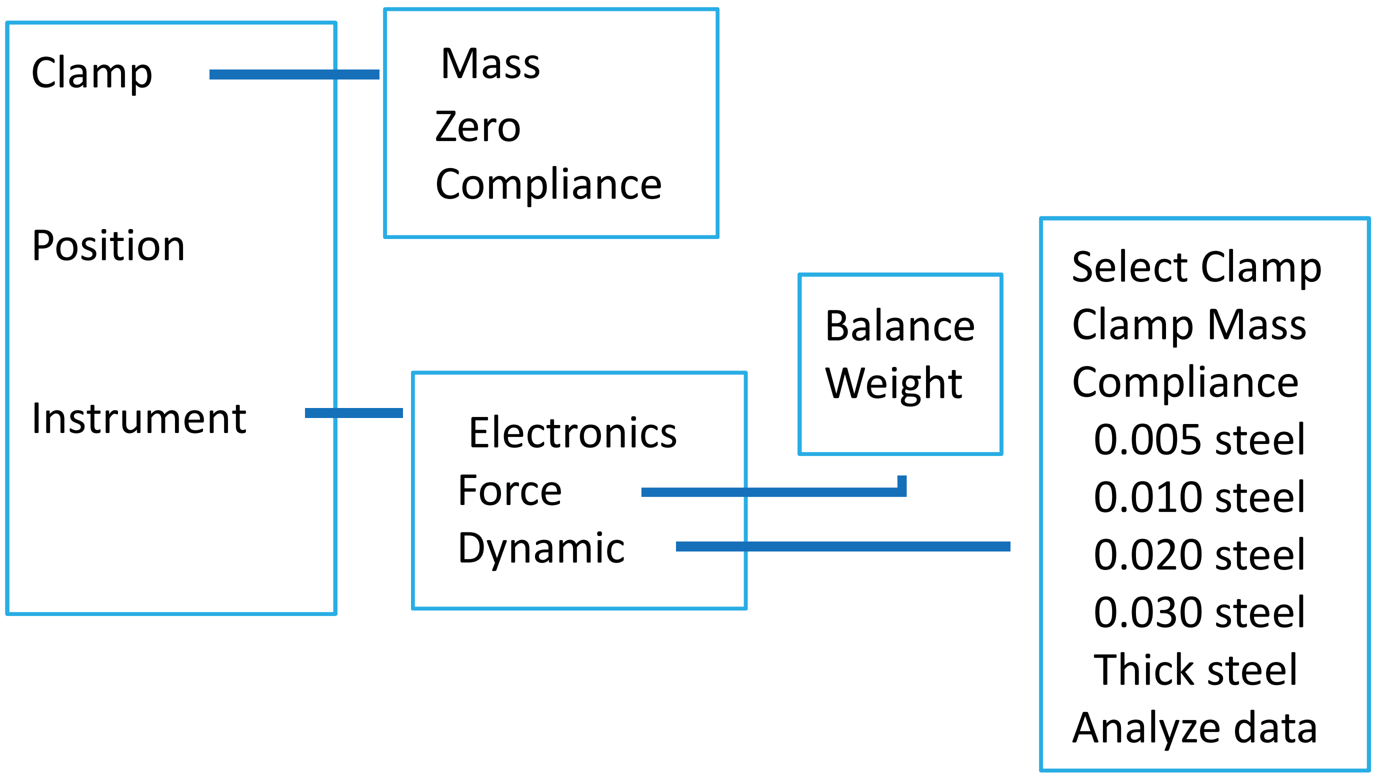
The World Leader in Thermal Analysis, Rheology, and Microcalorimetry

Appendix 1

Instrument Calibrations

Q800 DMA

Q800 Flow Chart of Calibration Procedures



TA Tech Tips – Clamp Calibrations

- Videos available at www.tainstruments.com under the Support\Videos tab or on the TA tech tip channel of YouTube™ (http://www.youtube.com/user/TATechTips)

DMA Clamp Calibration Details

- Clamp Calibration needs to be done ***initially for every clamp the first time you use that clamp.*** The calibration is stored in the software for that specific clamp.

- Experiment will not run without clamp calibration.

- There are three clamp calibrations: Mass, Zero, and Compliance.

- Not all calibrations need to be performed on all clamps. The following chart shows what calibrations to perform for the various clamps.

DMA Clamp MASS Calibration

- Weighs the clamp and compensates for its mass to ensure accuracy of force measurements.
- Mass calibration takes 5 minutes to run
- **MASS CALIBRATION CHECK:**
  - Release (float) the clamp by pressing the FLOAT/LOCK key on the module. The clamp should maintain its position or SLOWLY drift up or down.
  - If the clamp RAPIDLY sinks or rises, perform mass calibration.
  - If condition persists after mass calibration, perform position calibration.

DMA Clamp ZERO Calibration

Determines the point of zero sample length for accurate measurements of sample length (tension) or thickness (compression)

- COMPRESSION/PENETRATION CLAMPS: The Gauge Block Size window will appear. Leave all entries at **ZERO** and select **OK**.
- FILM/FIBER TENSION CLAMPS: Clamp Kit comes with an **OFFSET GAUGE.** Measure the length of the offset gauge and mount the gauge in the clamp. Enter the length of the offset gauge block and select **OK**.

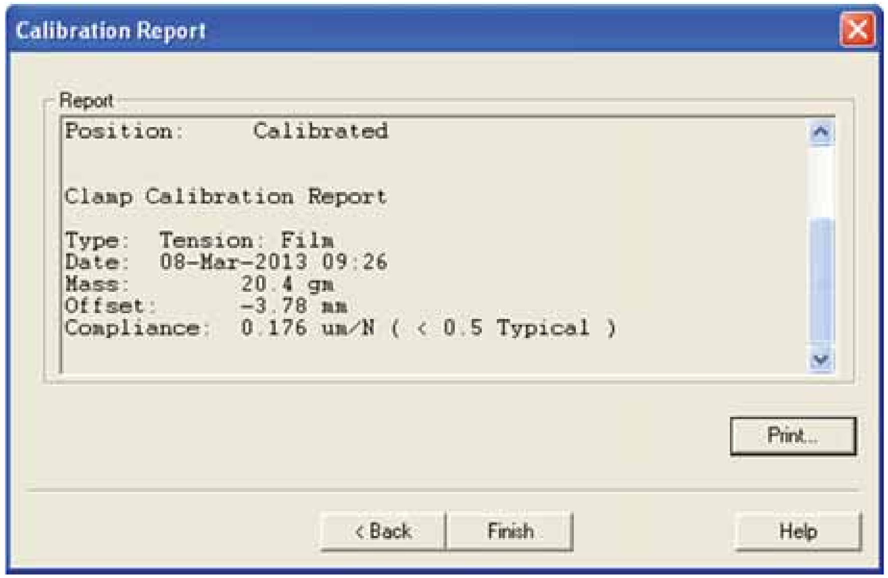
DMA Clamp COMPLIANCE Calibration

Measures the flexibility (compliance) of the clamp and calibrates the instrument to that flexibility ensure quantitative modulus and damping measurements on stiff samples.

- COMPRESSION/PENETRATION CLAMPS: Enter **ZERO** for all dimensions on the **Compliance Sample Calibration** window and select **OK**. Sample size must be zero for calibration.
- TPB & SINGLE/DUAL CANT.: Enter the width and thickness of the steel compliance sample. Enter the appropriate length
  - Single Cantilever- length between clamp faces
  - Dual Cantilever- length between back and middle clamp + length between middle and front clamp
  - 3 Point Bend- length between fixed clamps
  - Tension- length, as read by the instrument

DMA Clamp Calibration Report

REPORT returns the values for the currently installed clamp which can be printed out



Note:

- Mass calibration tolerance is ± 0.1 g
- Turn air bearings on for 20 min before mass calibration
- Mass can change with residue on clamps
- Clamp Compliance should be ≤ 1.0 µm/N

DMA Position Calibration

- Calibrates the absolute position of the drive shaft (and slide) as read by the Optical Encoder
- Recommended to perform routinely once a month.
- Perform when DMA is moved, reset, or powered down with float lock OFF
- To Perform Calibration
  - Remove both fixed and moveable clamps
  - Select **Position** from the **Calibrate** menu.
  - Calibration takes about 7 minutes

DMA Position Calibration Check

- ***Tension and Compression or Penetration Clamps:*** Manually position the clamp in the middle of its range of travel and release it. The clamp should maintain its position or slowly drift up or down. If the clamp rapidly sinks or rises, ***and the clamp mass has already been calibrated***, the clamp position requires calibration.

- ***For all other clamps:*** Press the **SCROLL** button on the DMA module until position is on the display. Manually move the clamp to the top of the travel. The position signal on the instrument display should read between **-0.5 to 1.0 mm**. Move the clamp so the bottom of the travel. The position signal should read between **24.5 to 25.5 mm**. If the upper or lower limit are not in the specified limits, calibrate position.

DMA INSTRUMENT Calibrations

There are Three INSTRUMENT CALIBRATIONS: ELECTRONICS, FORCE, AND DYNAMIC

- Instrument calibrations need to be performed at regular intervals or when the DMA is moved, feed hose for air cool or GCA is removed or installed.

ELECTRONICS Calibration

- Perform once a month to calibrate the electronics on the analog board.
- Takes approximately 10 minutes.
- Install shipping bracket to perform Electronics calibration

DMA INSTRUMENT Calibrations

FORCE Calibration

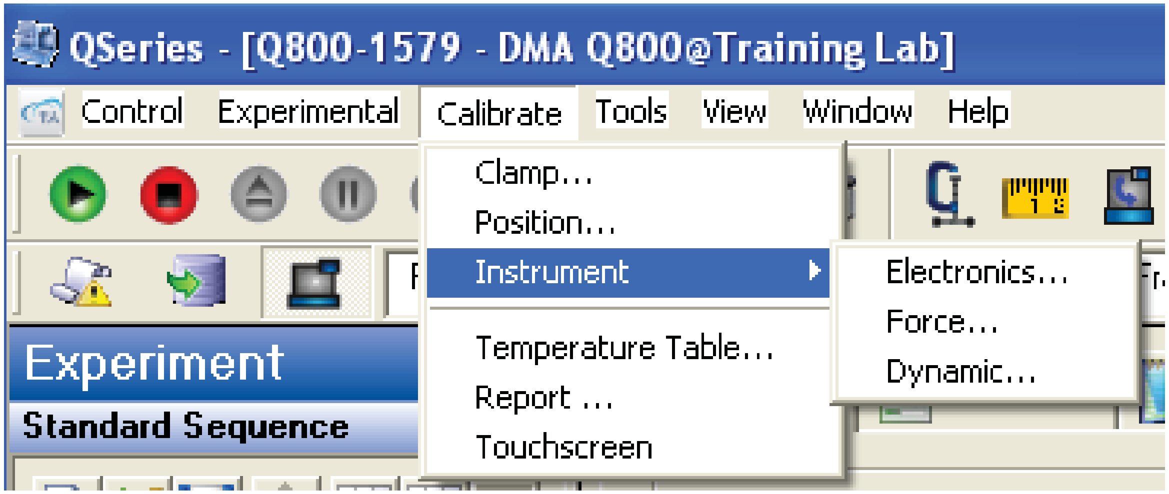
- Force Cal measures the moving mass of instrument and calibrates motor force response
- Force Calibration has two steps: ***BALANCE*** and ***WEIGHT***
- **Part 1: FORCE/BALANCE** calibration measures the amount of force needed to hold the position of the drive shaft.
- **Part 2: FORCE/WEIGHT** calibration measures the amount of force needed to hold the position of the drive shaft when a weight has been added.
- Each step take approximately 7 minutes

DMA INSTRUMENT Calibrations

DYNAMIC Calibration

- Multi-step process - measures samples of known stiffness and loss to determine the dynamic performance over wide range of force and frequency
- Perform calibration monthly or when instrument moved or when poor frequency performance is observed

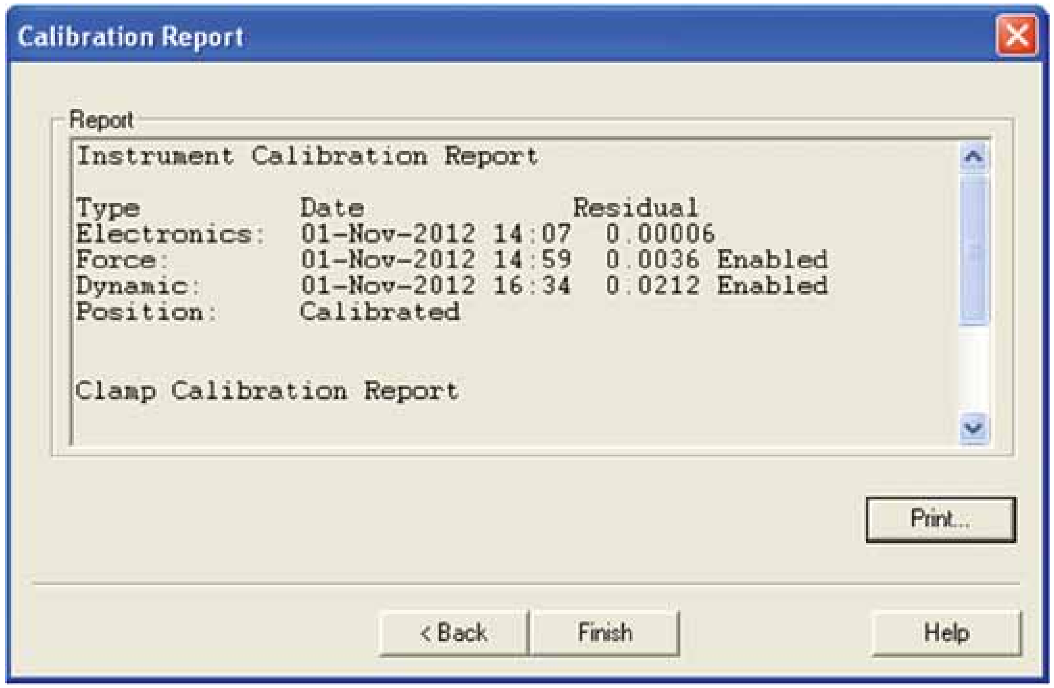
DMA Instrument Calibration



- To calibrate Instrument in ***Thermal Advantage***, launch the Instrument
- Calibration wizard from the "*Calibrate - Instrument*" menu.

- Follow steps outlined in each calibration wizard.

Instrument Calibration Report



Note:

- Dynamic fails above 0.1 The lower the residual value, the better the calibration. The residual values should be < 0.01.
- **If instrument does not report a calibration failure then all values are OK. It is typical for the values to change slightly with time.**

DMA Calibrations and Compliance Tables

Clamp	Mass	Zero	Compliance
Dual/Single Cantilever	X		X
3 Point Bend	X		X
Tension Film	X	X	X
Compression/Penetration	X	X	X
Shear Sandwich	X		
Specialty Fiber	X	X	

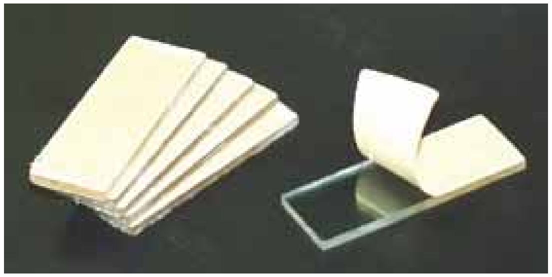
Clamp	Typical Compliance in µm/N
Single Cantilever	≤ 0.8
Dual Cantilever	≤ 0.2
3 Point Bend	≤ 0.6
Tension Film	≤ 0.5
Compression	≤ 0.6
Specialty Fiber	N/A
Shear Sandwich	N/A

DMA Confidence Check - Polycarbonate

- Load Polycarbonate (L ≈ 17.5, w ≈ 12.85, t ≈ 0.8mm)
- Single Cantilever
  - 20-30 micrometer amplitude
  - 1 Hz frequency
- Storage Modulus at Room Temperature

  E' = 2.35 GPa (2350 MPa) +/- 5%
- Tan Delta at Room Temperature

  Tan d < 0.01



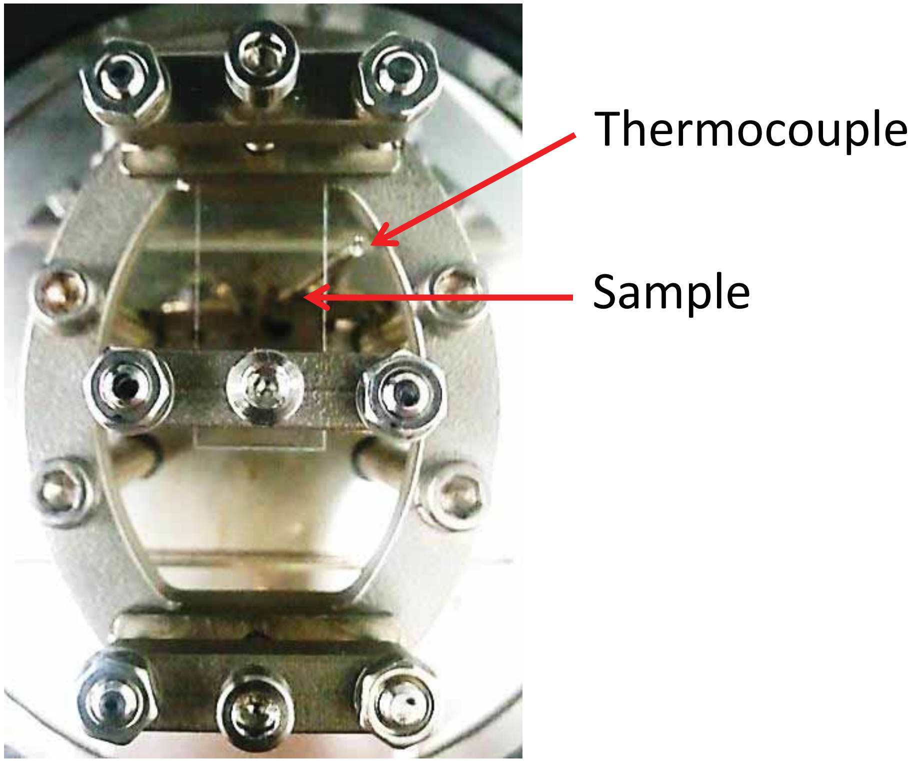
- Transition Temperature

  Tan d peak from 155-160℃ @ 1Hz, 3-5℃/min

  E'' peak will be about 5℃ lower

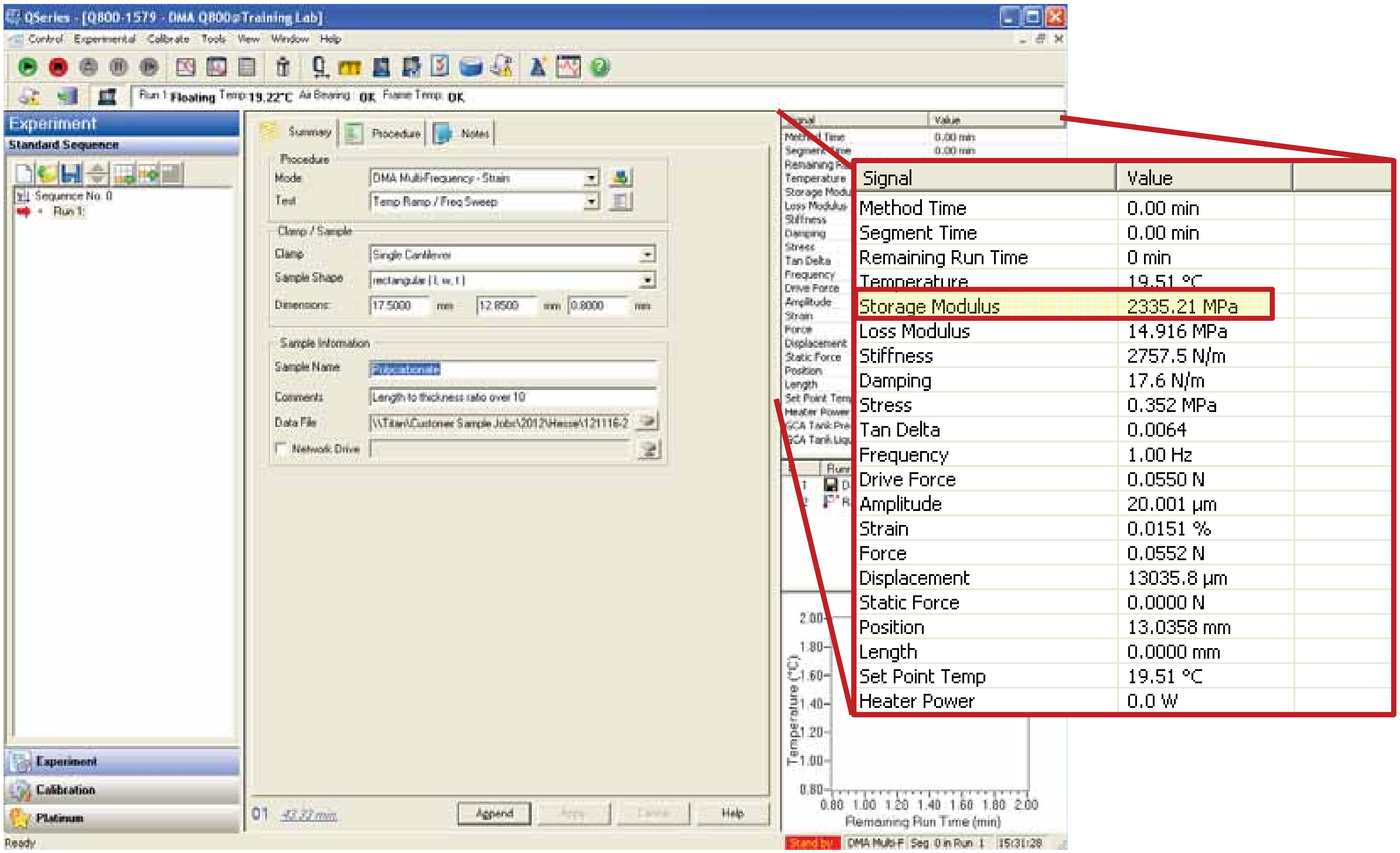
Mounting Sample

- Finger tighten the sample in position and then ‘Lock’ movable shaft to align clamp before tightening with torque wrench. Tighten to 8-10 in·lbs torque.



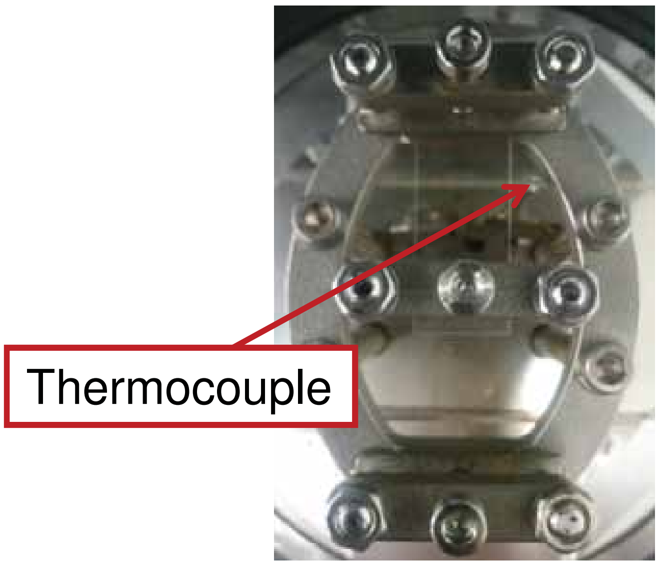
Polycarbonate at Ambient

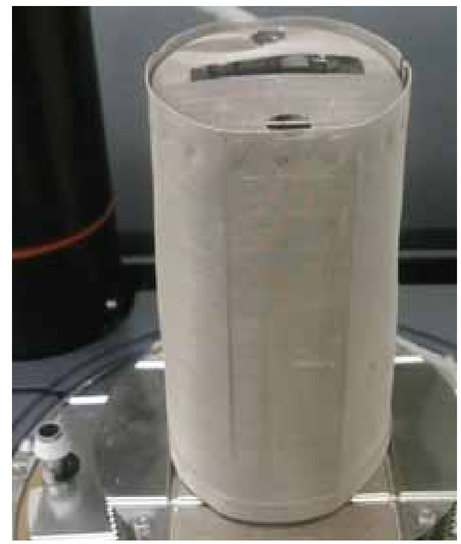
Confidence check: E' = 2350 MPa ±117.5 MPa



Temperature Ramp Results from Polycarbonate

- Transition Temperature:
  - Tan d peak from 155-160 °C @ 1Hz, 3-5 °C/min
  - E" peak will be about 5 °C lower





Note: Thermocouple position and sample or thermocouple shields can effect temperature results from a temperature ramp.

Instrument Calibrations

RSA G2 DMA

TA Instruments

RSA G2 Instrument Calibration

Calibration Tasks and Recommended Intervals

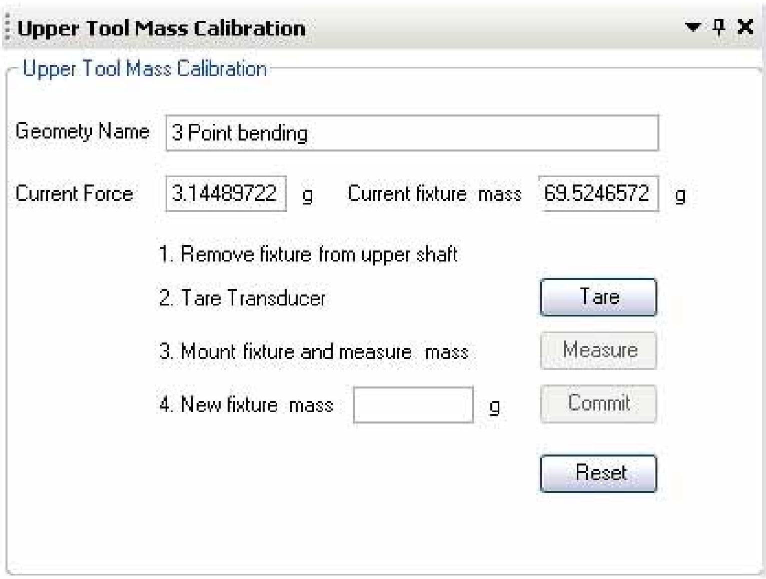
Calibration Task	Calibration Interval
Upper Fixture Mass Calibration	Mandatory: During geometry creation (is a part of geometry configuration)
Force Calibration	Suggested: Monthly. Mandatory: Following transducer replacement.
Phase Angle and Modulus Check	Suggested: Monthly Mandatory: Following actuator or transducer replacement.
Gap Temperature Compensation	Suggested: As required by the experiment.

Clamp Mass Calibration

Calibrating the Upper Geometry Mass

To calibrate the mass of the upper fixture:

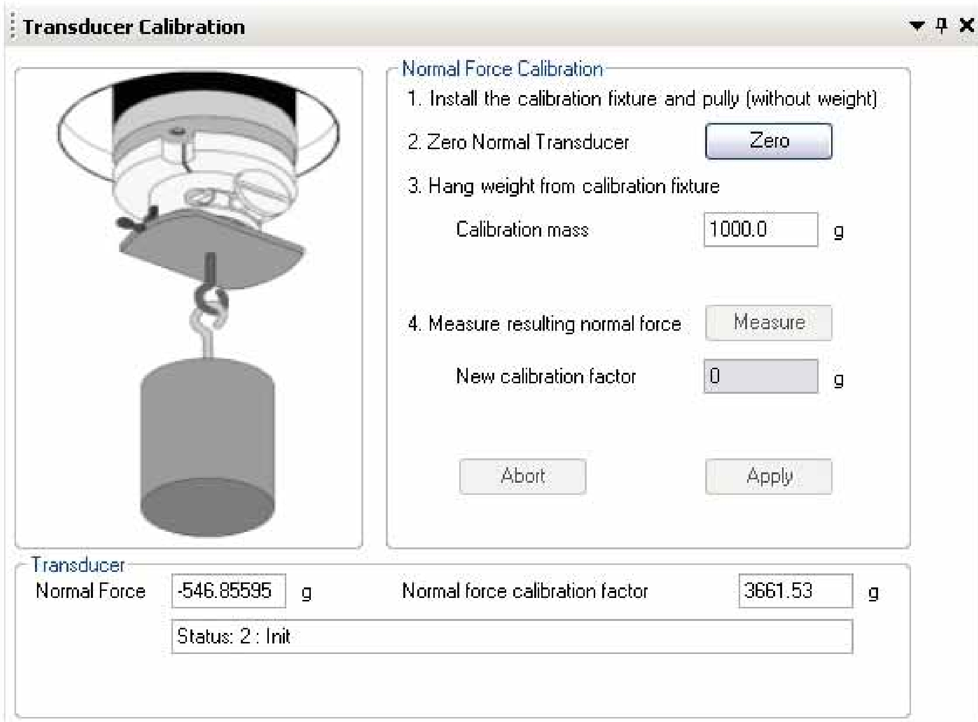
1. Click **Home** > **Geometry** ribbon and select the geometry to be calibrated from the drop-down list.
2. Click **Instrument** > **Calibrate** ribbon > **Fixture Mass** from the drop-down menu. The **Upper Fixture Mass Calibration** window (shown below) appears.



3. Follow the on-screen instructions to tare the transducer.
4. After you mount the fixture and click **Measure**, TRIOS enters the new mass (in grams) in the **New fixture mass** field.
   - Click **Commit** to have this new value applied to the **Upper geometry mass** field in the Geometry Wizard's **Geometry constants** screen.
   - Click **Reset** to restart the upper fixture mass calibration.
5. Close the **Upper Fixture Mass Calibration** window when finished.

Force Calibration

1. Within TRIOS software, select **Instrument** tab > **Calibrate** > **Force** to open the **Transducer Calibration** window (if not already open).



2. Follow the on-screen instructions to zero normal transducer.
3. After you hang the weight from the calibration fixture and click **Measure**, TRIOS enters the new value in the **New calibration factor** field.
   - Click **Apply** to send the new calibration factor to the instrument and save your settings.
   - Click **Abort** to restart the force calibration procedure.
4. When finished, remove the calibration fixture and pully from the instrument and close the **Transducer Calibration** window.

Phase Angle and Modulus Check

Procedure to Check the Phase Angle and Modulus

Follow these steps:

1. Set the transducer to **High** as follows:
   a. Access the **Transducer** Control panel by selecting the **Transducer Control** from the **Controls** menu on the **Home** tab.
   b. Select **High Range** from the transducer range down-down list.
2. Install the three-point bending geometry that came with the RSA-G2. Be sure to use the 40-mm span.
3. Load the calibration steel sample (part number 400-02589) that is supplied with the geometry.
4. Zero the force from the **Gap** Control panel.
5. Select or set up the three-point bending geometry using the Geometry Manager.
6. Conduct an Oscillation Frequency Sweep using the following parameters:
   - Conditioning block: Axial force control in compression, constant 700 g, 10 g sensitivity, 5 sec data sampling priority
   - Strain 0.02 %
   - Logarithmic Sweep 0.628 – 628 rad/s, 5 pts/decade
7. Ensure that the phase angle, δ, is ± 0.25° over the frequency range of 0.628 to 200 rad/s and ± 1° over the frequency range of 200 to 628 rad/s. If the values obtained from this test are outside this range, please contact Technical Services for further assistance.

To obtain good performance above 100 rad/s, ensure that the test station is on a stable work surface (such as a marble table).

Instrument Calibrations

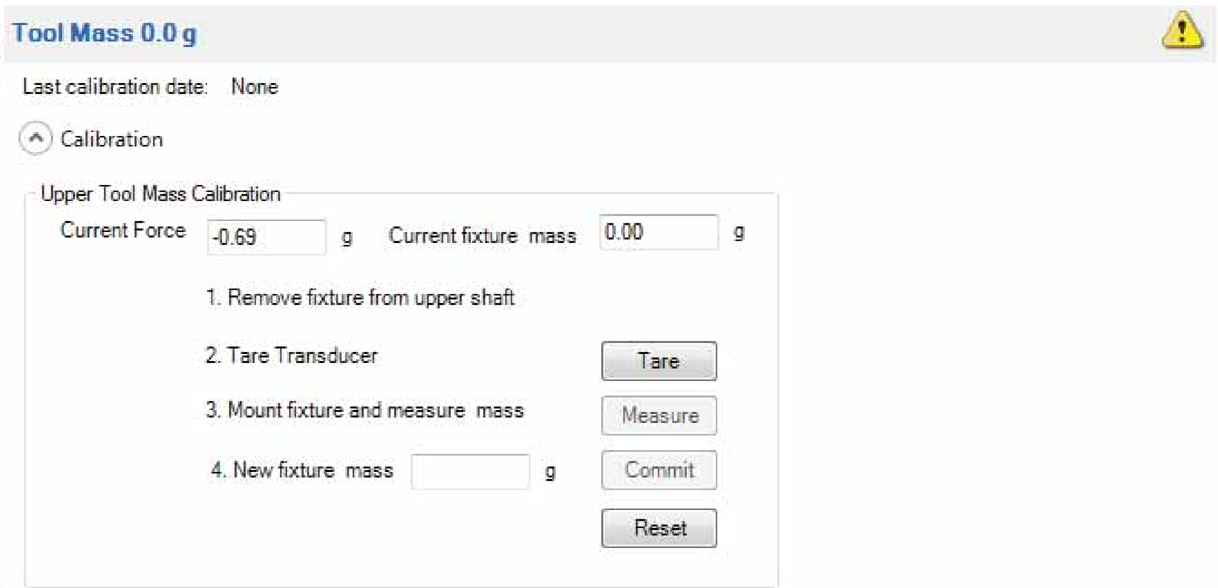
ARES-G2 and DHR DMA Mode

ARES-G2: Tool Mass Calibration

Calibrating the Upper Geometry Mass

To calibrate the mass of the upper fixture:

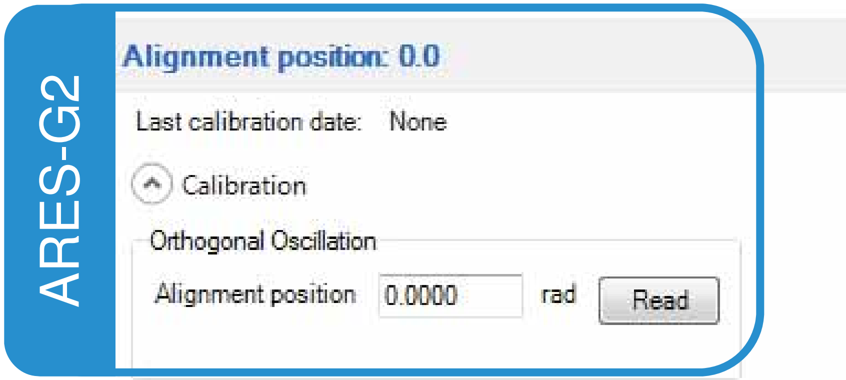
1. Click **Home** > **Geometry** ribbon and select the geometry to be calibrated from the drop-down list.
2. Click **Instrument** > **Calibrate** ribbon > **Fixture Mass** from the drop-down menu. The **Upper Fixture Mass Calibration** window (shown below) appears.

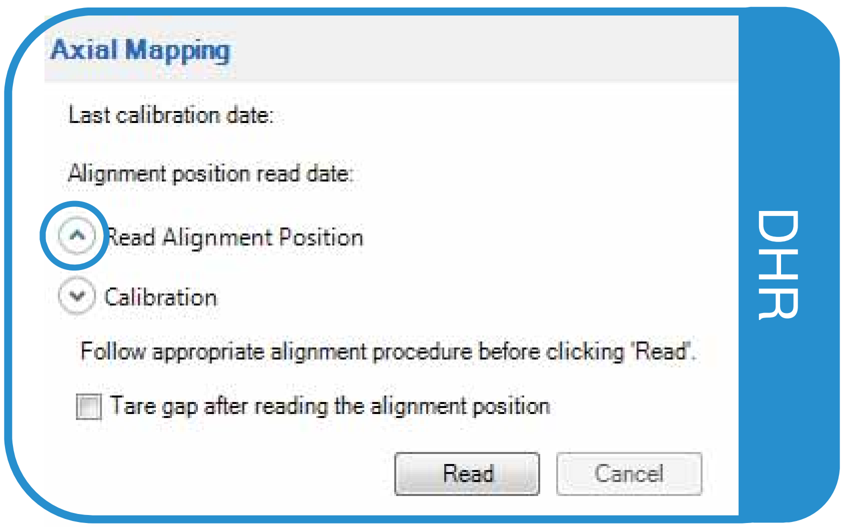


3. Follow the on-screen instructions to tare the transducer.
4. After you mount the fixture and click **Measure**, TRIOS enters the new mass (in grams) in the **New fixture mass** field.
   - Click **Commit** to have this new value applied to the **Upper geometry mass** field in the Geometry Wizard's **Geometry constants** screen.
   - Click **Reset** to restart the upper fixture mass calibration.
5. Close the **Upper Fixture Mass Calibration** window when finished.

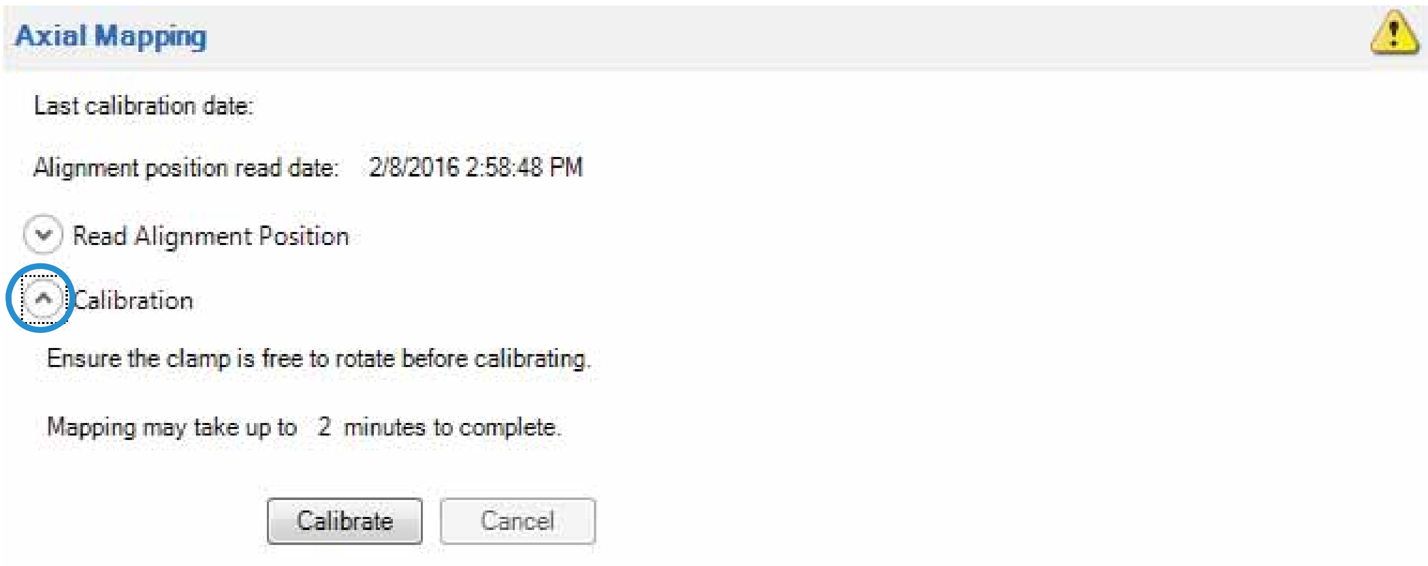
ARES-G2 and DHR: Angular Alignment Calibration

Allows for alignment of upper and lower geometries.
Requires alignment bar (3 pt bending, cantilever) or steel shim (tension)





DHR: Axial Mapping Calibration



Axial mapping is used to relate the desired displacement with the control of the magnetic bearing. Better understand inertial effects with the current geometry mass.