Dynamic Mechanical Analysis
Basic Theory & Applications Training
Part 1

Course outline

Part 1

- Basic Theories of Dynamic Mechanical Analysis
- DMA Instrumentation and Clamps
- Introduction to DMA Experiments
  - Dynamic tests
  - Transient tests
- Appendix: Screenshots from the instrument control software

Part 2

- Recap of Part 1
- DMA Applications and data interpretation
- Troubleshooting experimental Issues
- Time-temperature superposition (TTS)

Basic Theories of
Dynamic Mechanical Analysis

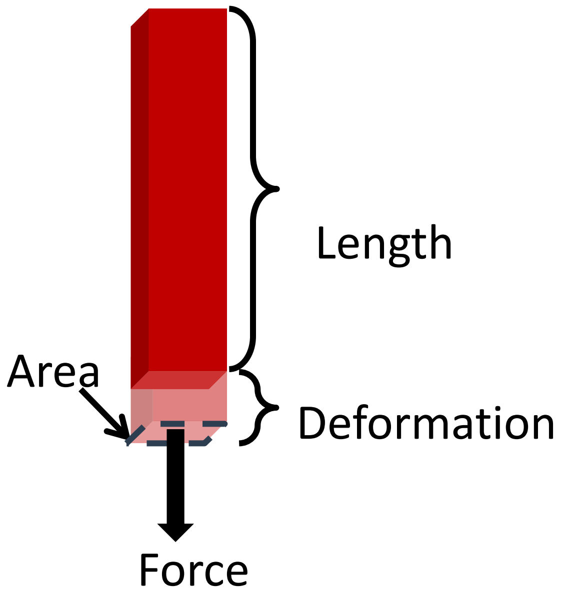
DMA definitions

- A Dynamic Mechanical Analyzer (DMA) measures the mechanical/rheological properties of a material as a function of time, frequency, temperature, stress and strain

- Typical materials tested on a DMA
  - Thermal plastic and thermosets
  - Elastomers/ rubbers
  - Gels
  - Foams
  - More….

Working principle of a DMA

- Apply a force or a deformation to a sample, then measure sample's response, which will be a deformation or a force.
- All mechanical parameters (stress, strain, modulus, stiffness et al) are calculated from these 2 raw signals



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

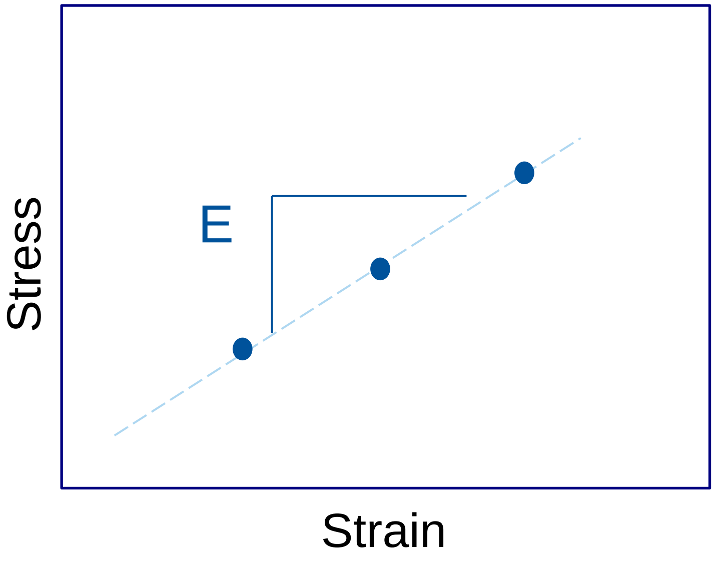
$$\text{Strain} = \frac{\text{Deformation (m)}}{\text{Length (m)}}$$

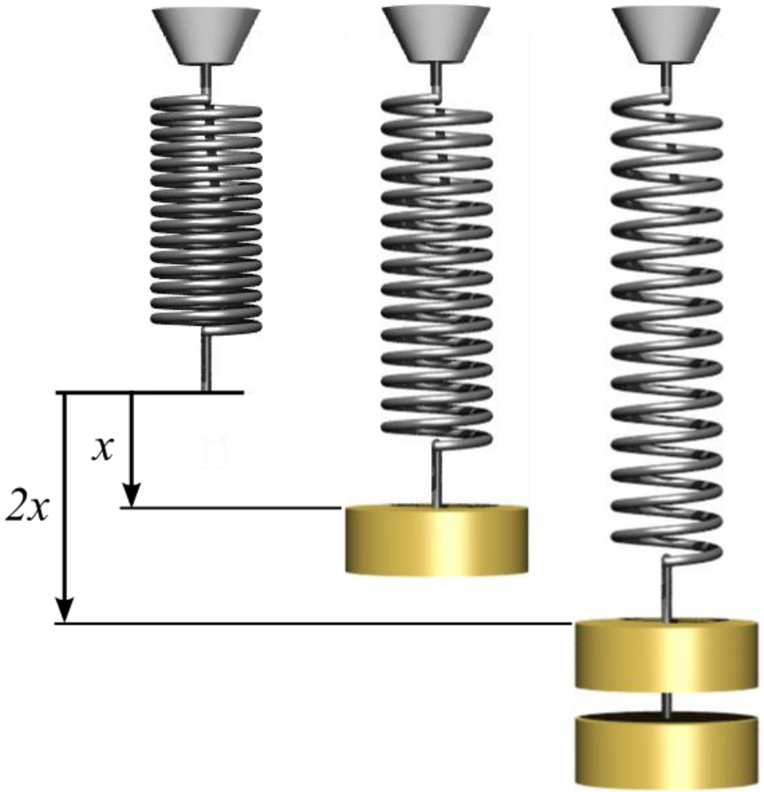
$$\text{Modulus (Pa)} = \frac{\text{Stress (Pa)}}{\text{Strain}}$$

Hooke’s Law of Elasticity

- For a purely Elastic Solid, Stress and Strain have a constant proportionality
- The slope of stress over strain is the Young’s modulus of the material

$$\sigma = E*\varepsilon$$

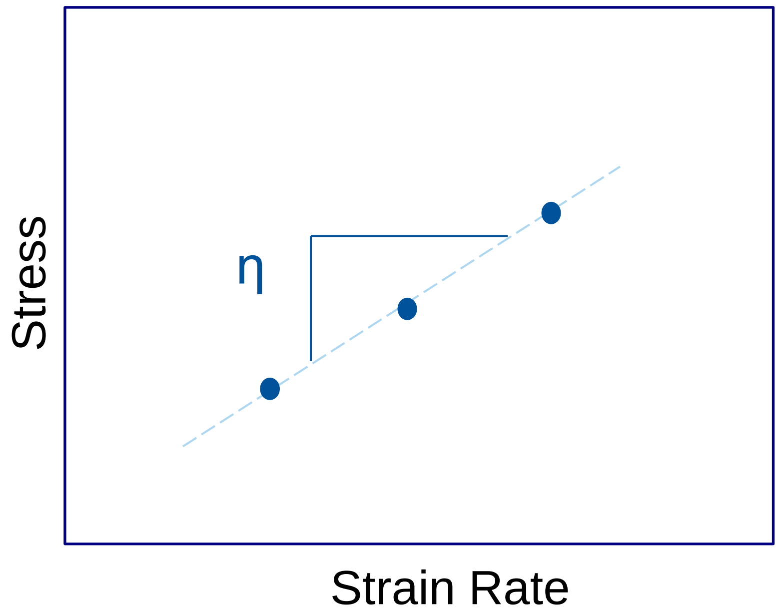




Newton's Law of Viscosity

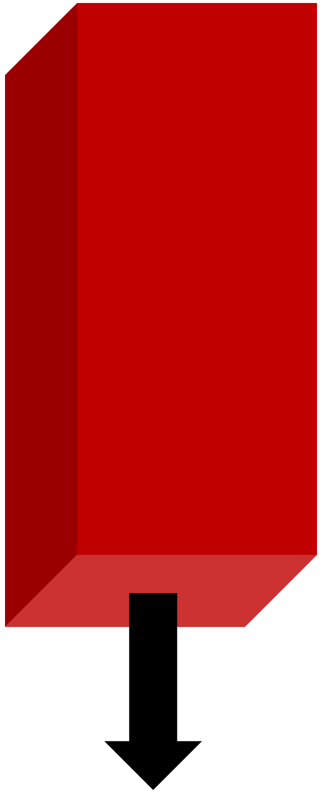
- For a purely Viscous Liquid, Stress is proportional to Strain Rate $d\varepsilon/dt$
- The slope of stress over strain rate is the viscosity of the material

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



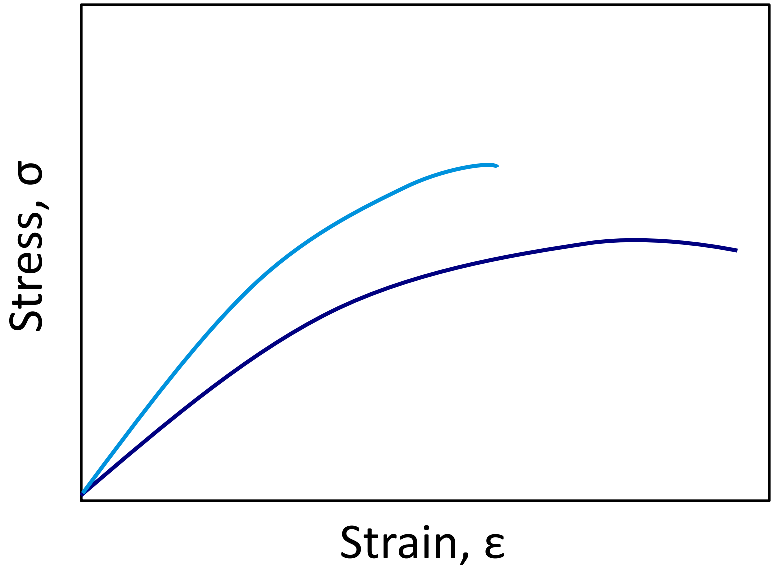
t
T
Viscous
Viscoelastic
Elastic

Time dependency of mechanical properties in viscoelastic materials



- 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*

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



Time-dependent viscoelastic behavior

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

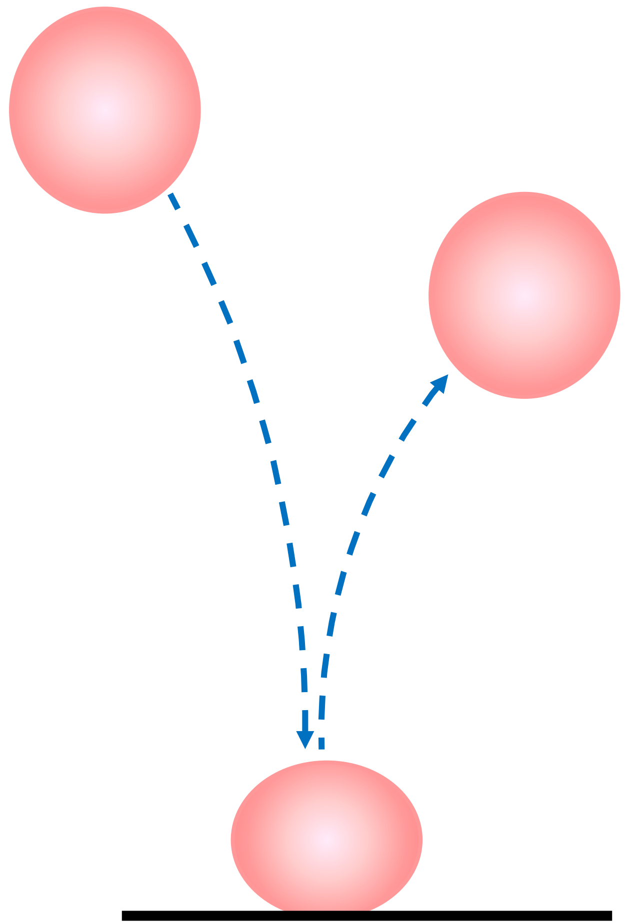
Started in 1927 by Thomas Parnell in Queensland, Australia

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

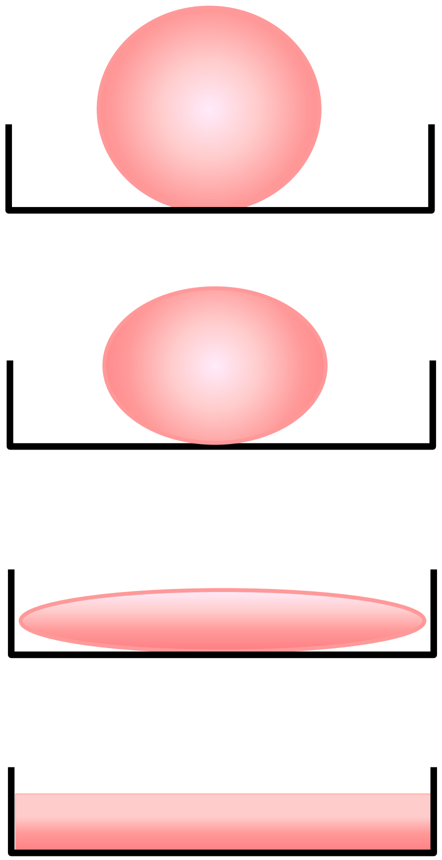
Silly putty video

- Let’s take another common example – silly putty
- When we pull on silly putty slowly, it is flowy and stretchy like a liquid.
- However, if we pull on it quickly, it snaps and breaks like a solid.

Time-dependent viscoelastic behavior (silly putty)



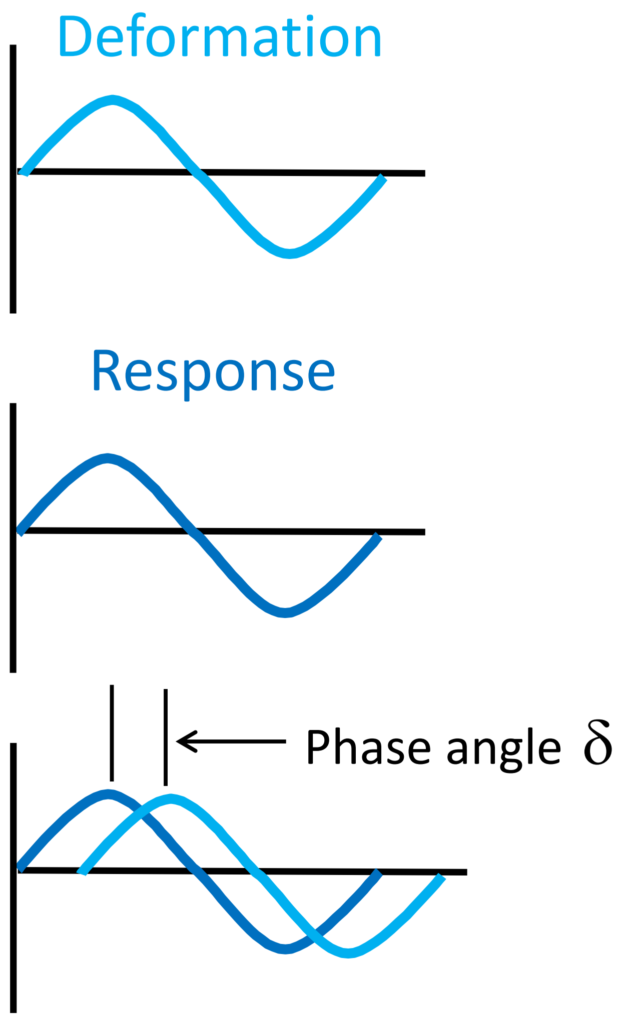
Time-period of deformation is short (high frequency)



Time-period of deformation is long (low frequency)

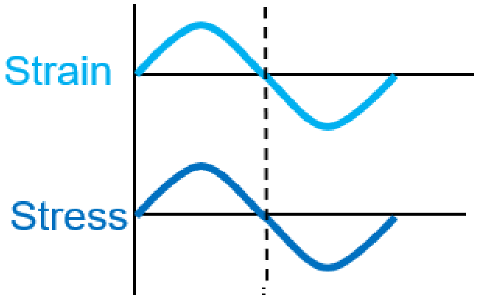
Dynamic Mechanical Testing

- 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 also measured.

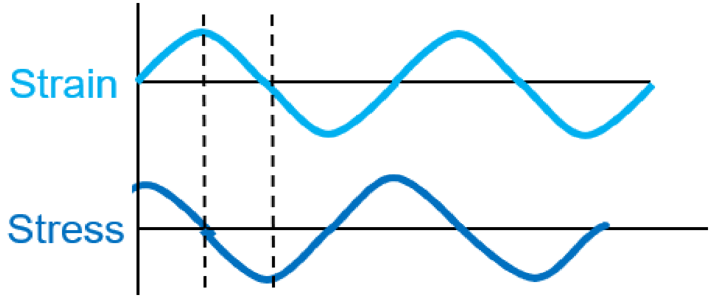


Phase angle response in dynamic mechanical tests

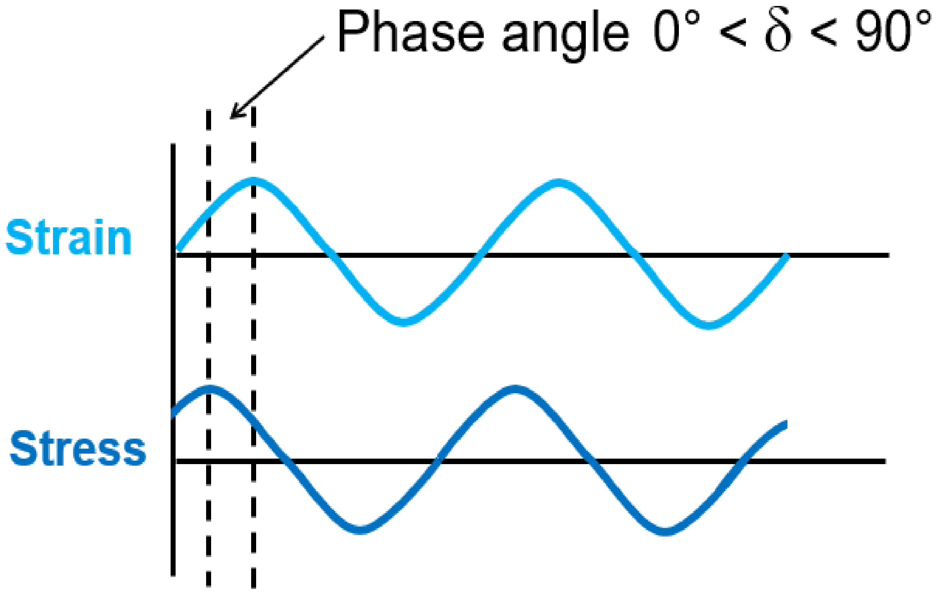
Purely Elastic Response
(Hookean Solid)
$\delta = 0°$



Purely Viscous Response
(Newtonian Liquid)
$\delta = 90°$



Viscoelastic Response
(Most materials)



Viscoelastic parameters obtained from DMA tests

Complex Modulus: Measure of materials overall resistance to deformation.

$$E^* = \frac{stress\ amplitude\ (\sigma)}{strain\ amplitude\ (\gamma)}$$

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

$$E' = \left(\frac{\sigma}{\gamma}\right)\cos\delta$$

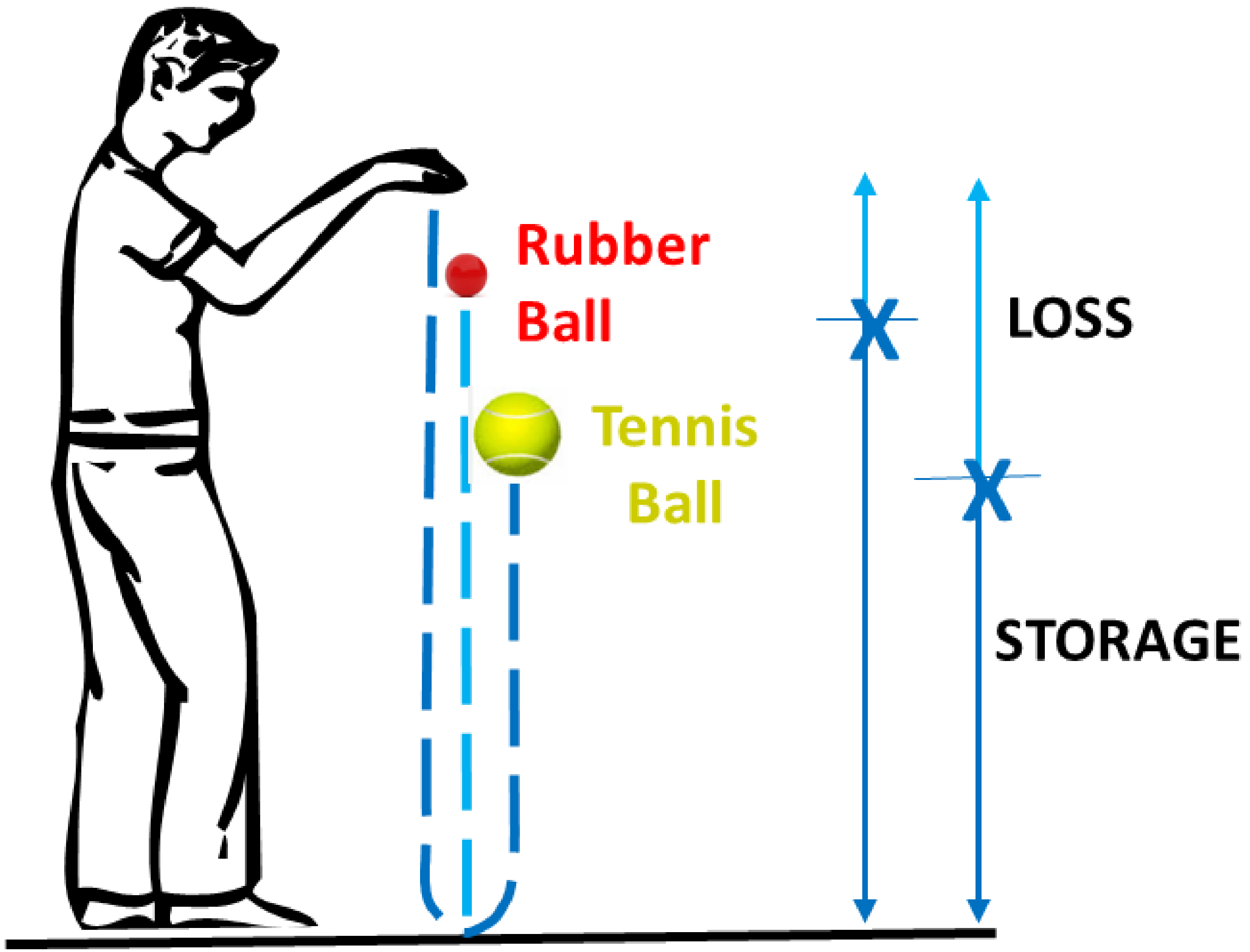
The Viscous (loss) Modulus:
The ability of the material to dissipate energy. Energy lost as heat.

$$E'' = \left(\frac{\sigma}{\gamma}\right)\sin\delta$$

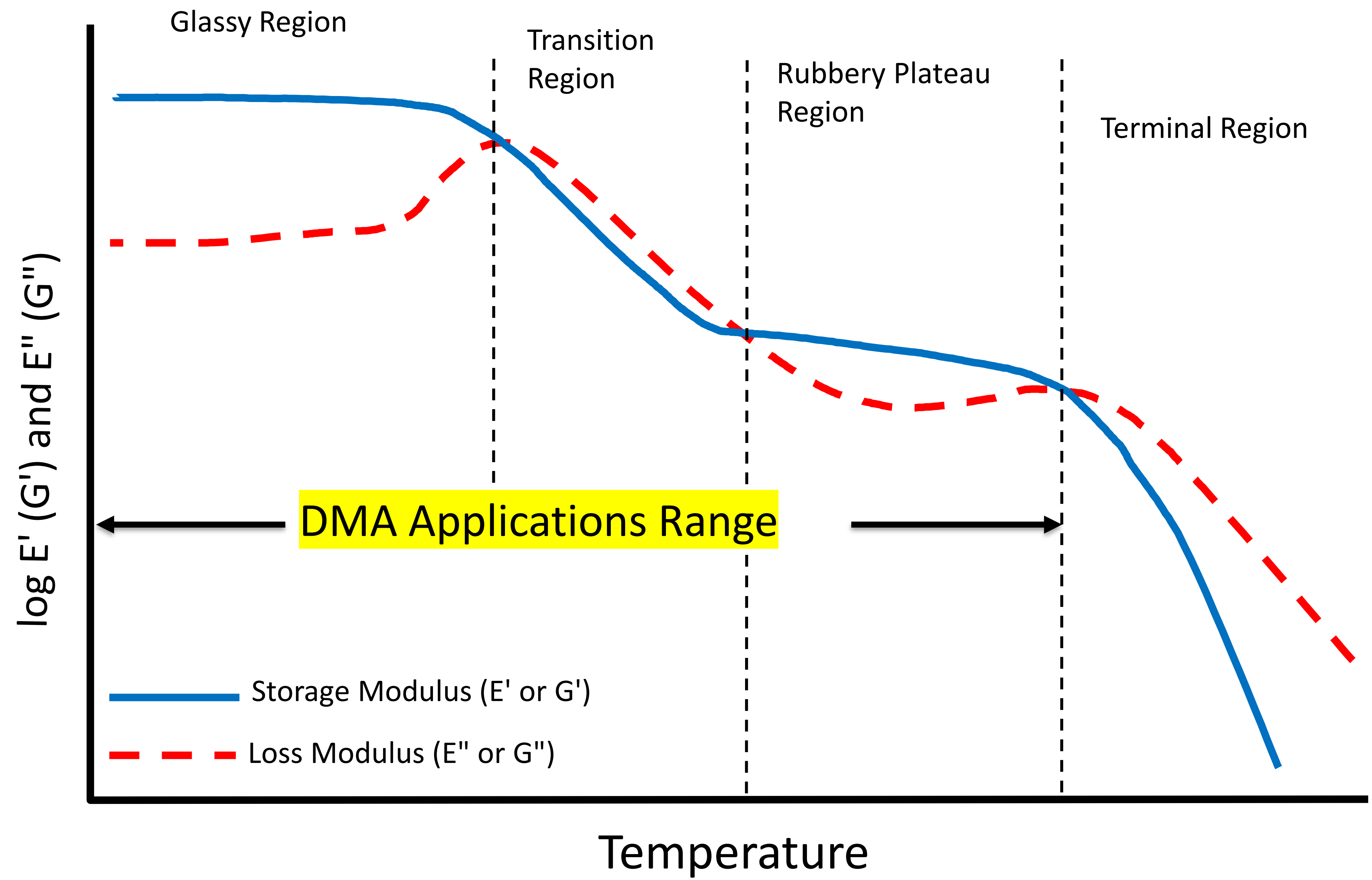
Tan Delta:
Measure of material damping. Increasing tan δ implies a greater potential for energy dissipation and lower elasticity, and vice-versa. Measure of viscous property while having the appropriate level of stiffness.

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

Storage and Loss of a Viscoelastic Material



Viscoelastic spectrum for a typical amorphous polymer



DMA results can correlate to…..

Molecular Structures

Product Properties

Stress

Strain

Stiffness

Damping factor

Transition temperatures

Modulus (E, G) / Compliance (J)

Processing Conditions

DMA Instrumentation and Clamps

DMA instrumentation

RSA G2

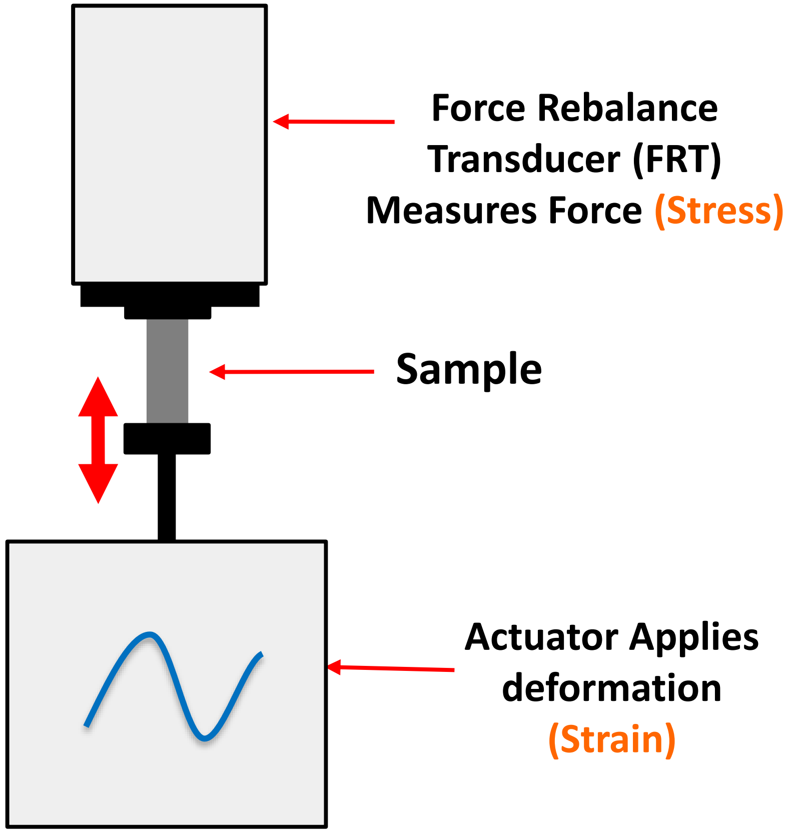
Discovery DMA850

Electroforce series (high load frame, fatigue)

DMA instrumentation

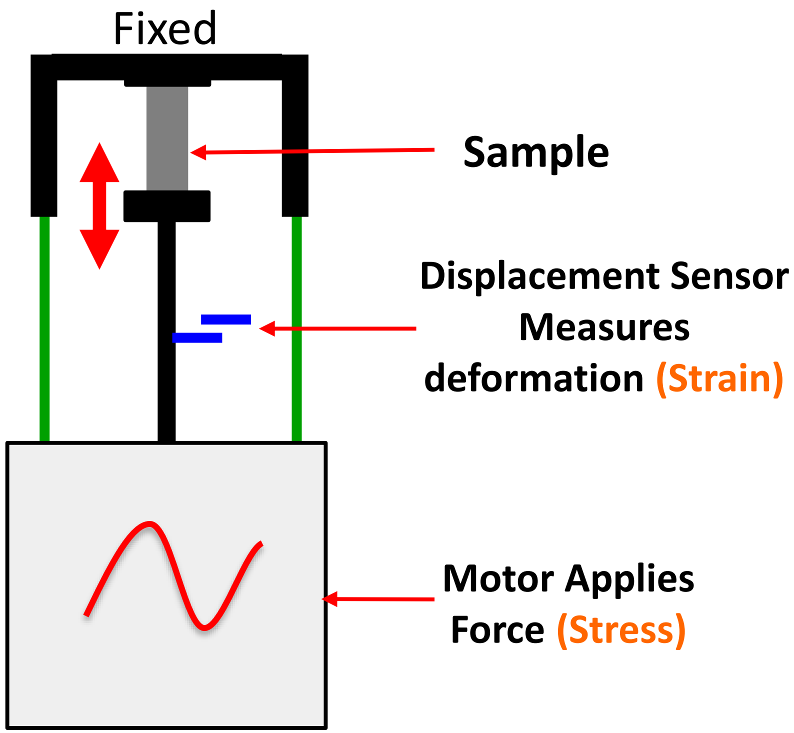
RSA G2

Separate Motor & Transducer

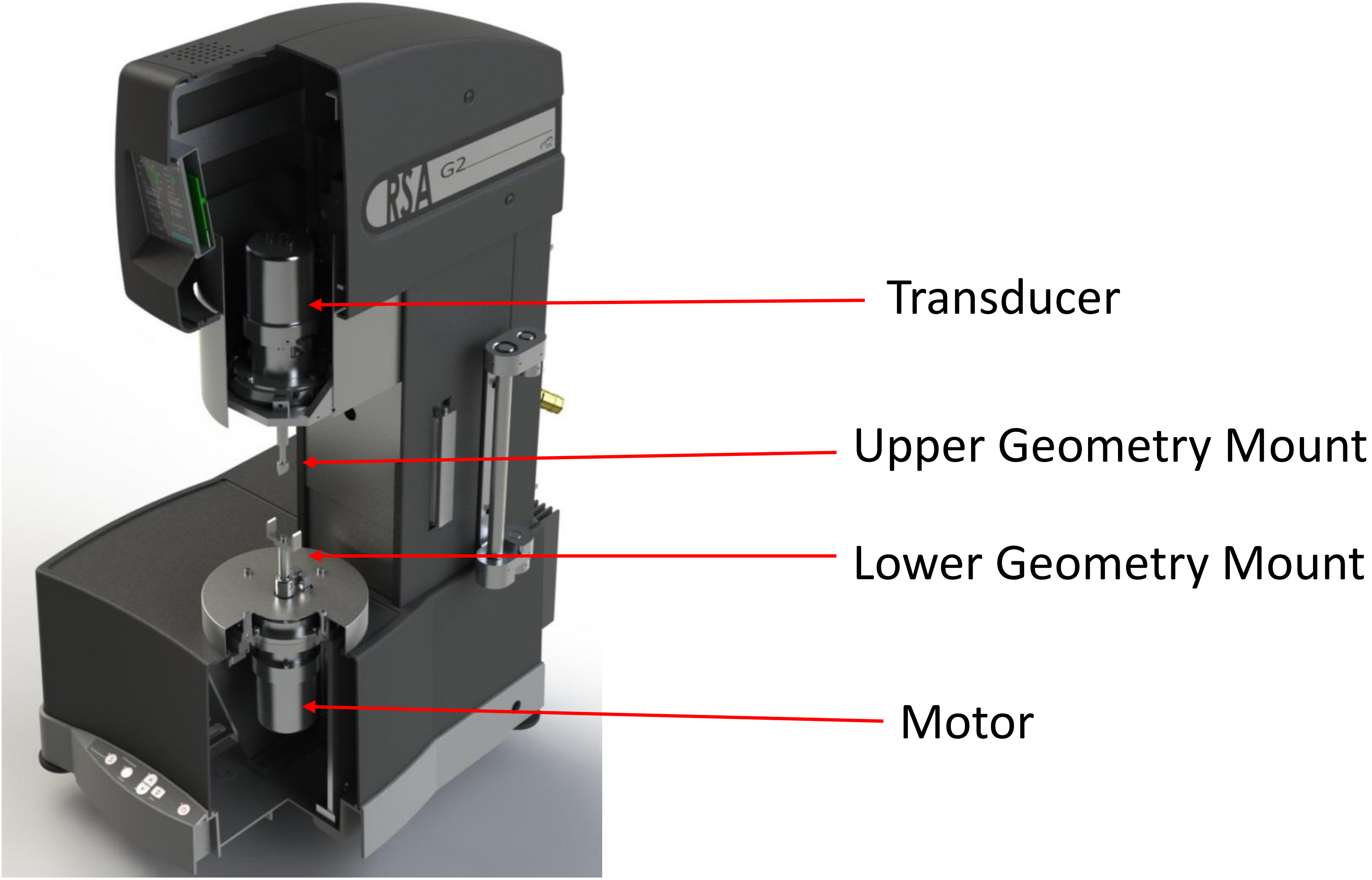


DMA850 and Q800

Combined Motor & Transducer



RSA G2: Schematic Dual Head Design



DMA850: Schematic

DMA850 and Q800: Humidity Option

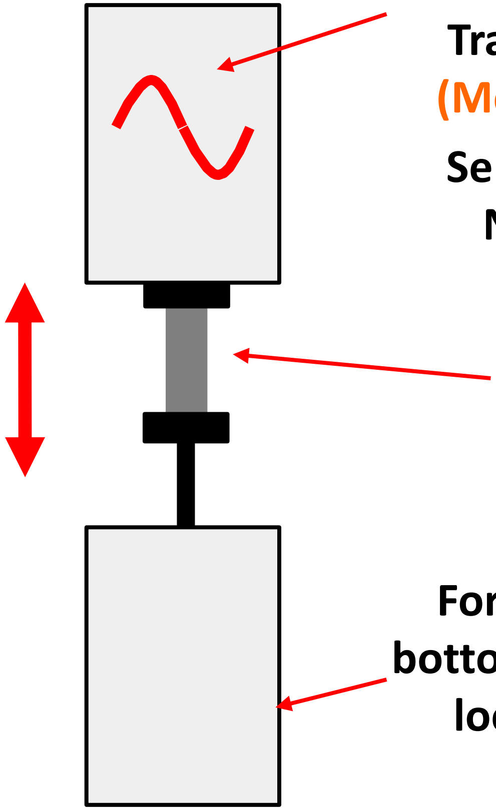
DMA Specifications

	RSA G2	DMA850	Q800
Max Force	35 N	18 N	18 N
Min Force	0.0005 N	0.0001 N	0.0001 N
Displacement Resolution	1 nm	0.1 nm	1 nm
Frequency Range	2×10^{-6} to 100 Hz	1×10^{-4} to 200 Hz	1×10^{-2} to 200 Hz
Dynamic Deformation Range	$\pm 5 \times 10^{-5}$ to 1.5 mm	$\pm 5 \times 10^{-6}$ to 10 mm	$\pm 5 \times 10^{-4}$ to 10 mm
Temperature range	-150 to 600°C	-150 to 600°C	-150 to 600°C
Isothermal Stability	± 0.1	± 0.1	± 0.1
Heating Rate	0.1°C to 60°C/min	0.1°C to 20°C/min	0.1°C to 20°C/min
Cooling Rate	0.1°C to 60°C/min	0.1°C to 10°C/min	0.1°C to 10°C/min

DMA Mode on DHR and ARES-G2

ARES G2 and DHR DMA Mode

Strain control & dynamic test only



Force Rebalance Transducer (FRT)
(Measures Stress)

Servo control on Null position
(Strain)

Sample

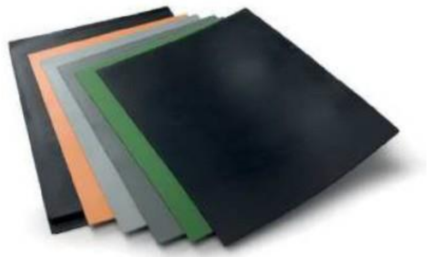
For the ARES-G2, the bottom Actuator remains locked during DMA function

Specifications of the DHR-DMA and the ARES-G2 DMA

Dynamic test only	DHR – DMA mode	ARES-G2 DMA mode
Motor Control	FRT	FRT
Minimum Force (N) Oscillation	0.003	0.001
Maximum Axial Force (N)	50	20
Minimum Displacement (μm) Oscillation	0.01	0.5
Maximum Displacement (μm) Oscillation	100	50
Axial Frequency Range (Hz)	1×10^{-5} to 16	1×10^{-5} to 16

What samples can be measured on a DMA?

- By changing the clamp, we can test a range of different materials



Elastomers

Films

Fibers

Gels

Plastics

Foams

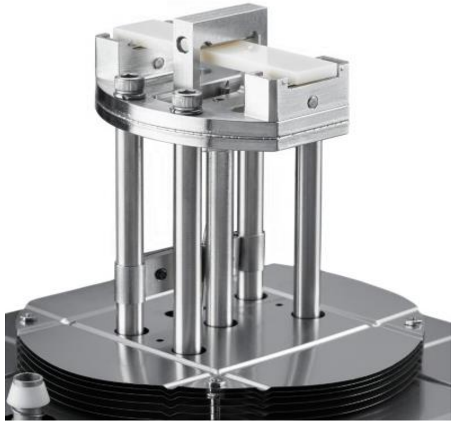
Composites

Clamps for DMA850 and Q800

S/D Cantilever

Film/Fiber Tension

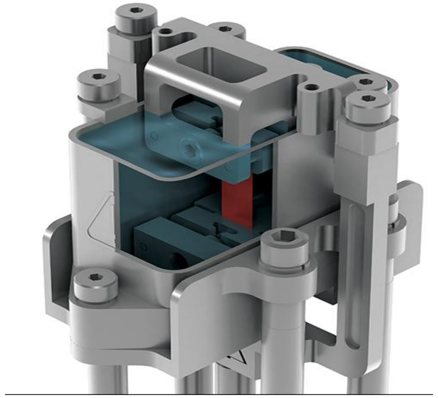
3-Point Bending



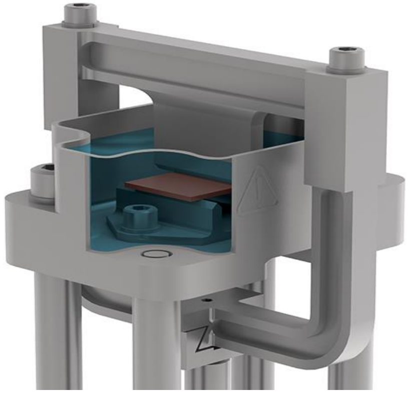
Compression

Shear Sandwich

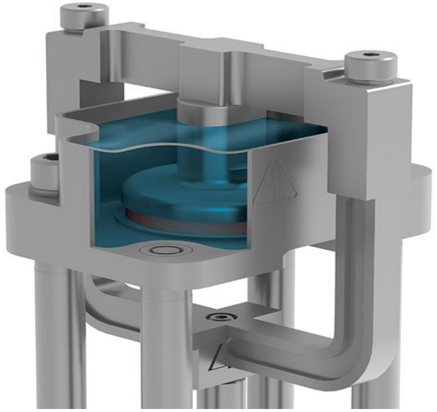
Submersible Tension



Submersible Bending



Submersible Compression

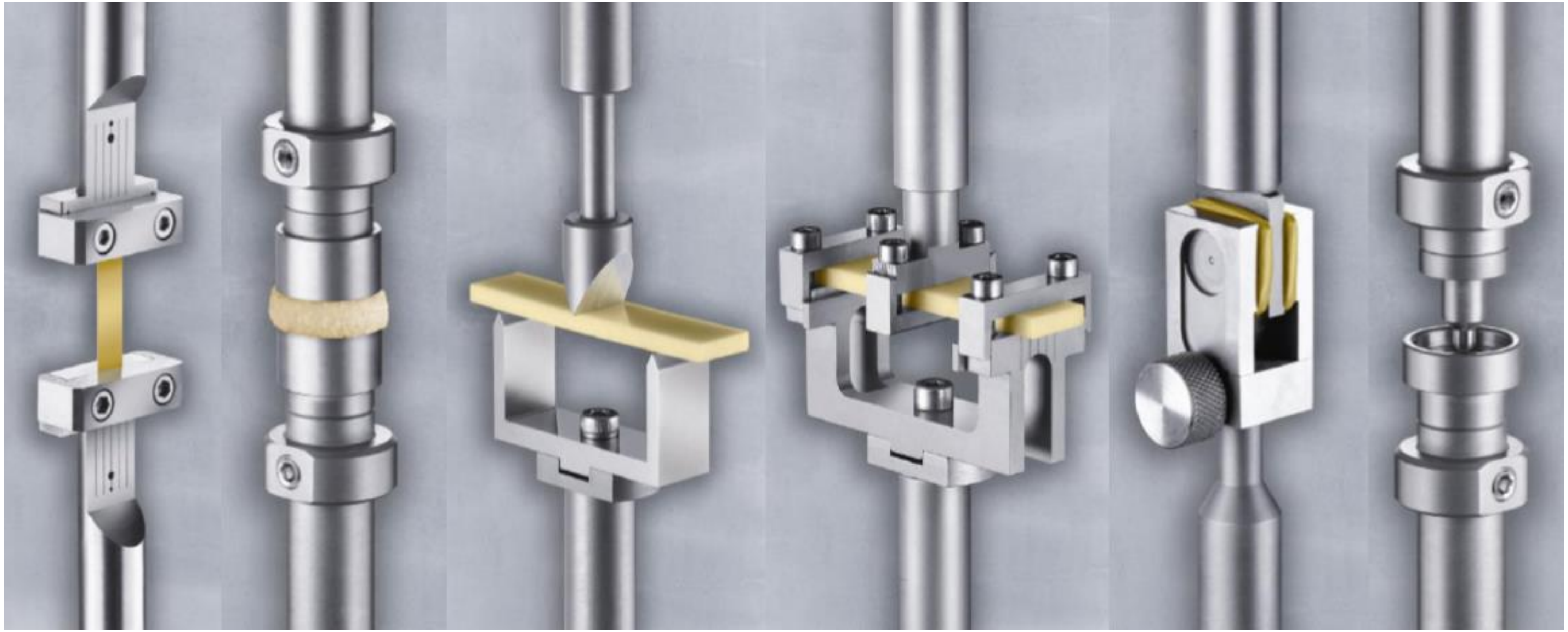


Clamps for RSA G2

Film/Fiber Tension

3-Point Bending

Shear Sandwich



Compression

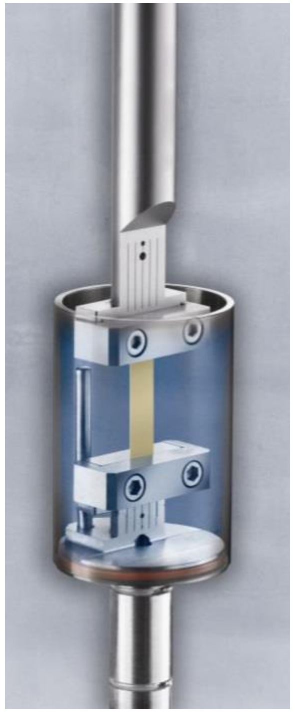
S/D Cantilever

Contact Lens

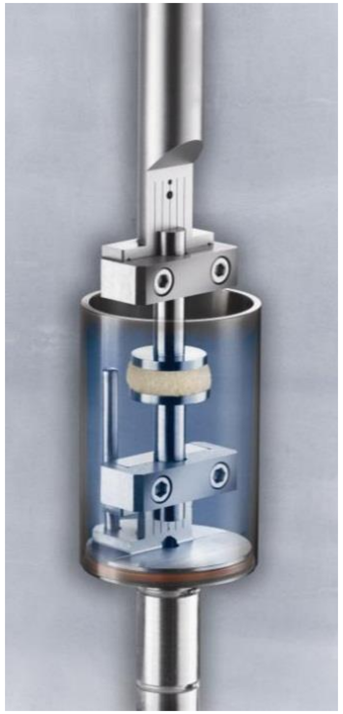
RSA G2 Immersion Clamps

- 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.

Testing Solids on a Rheometer

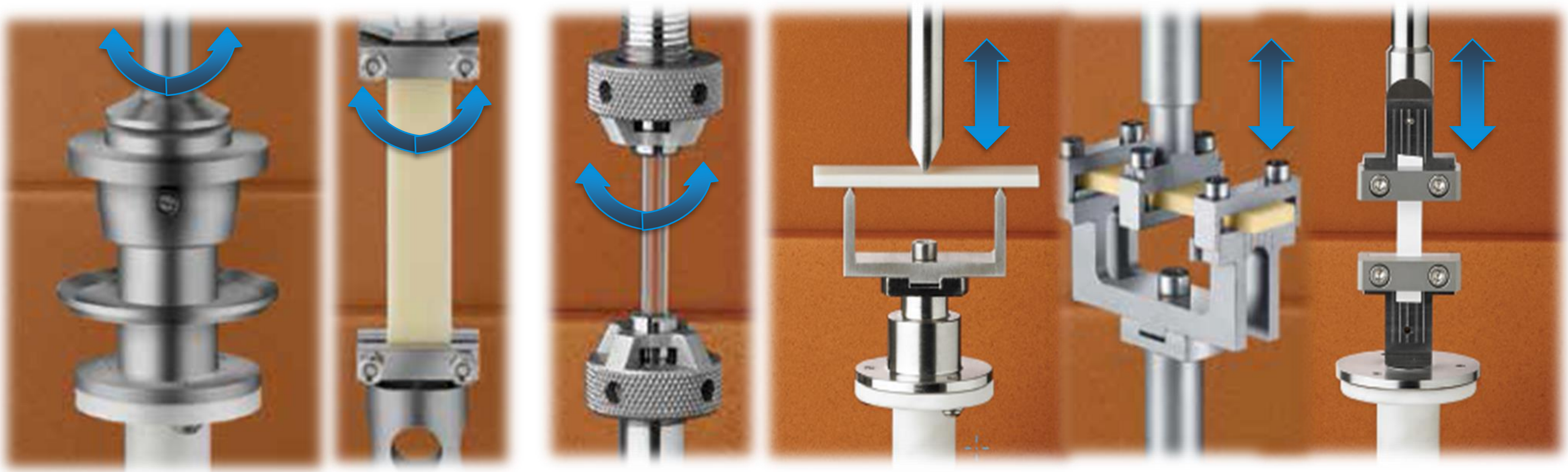
Torsion (rotational) and DMA (axial) geometries allow solid samples to be characterized in a temperature controlled environment.

$$E = 2G(1 + \nu)$$

ν : Poisson's ratio

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

Young's Modulus: E', E'', E^*



Parallel plate

Rectangular and cylindrical torsion

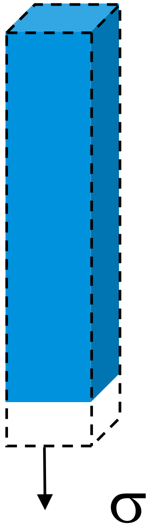
3-point bending

Cantilever

Tension

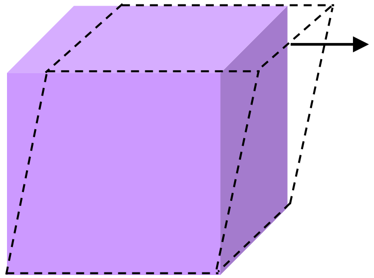
Three fundamental modes of deformation

Young's Modulus



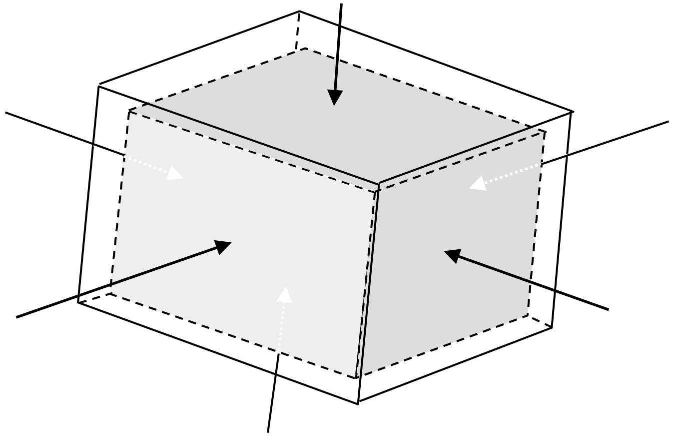
$$E = \frac{\sigma}{\varepsilon}$$

Shear Modulus



$$G = \frac{\tau}{\gamma}$$

Bulk Modulus



$$B = \frac{\sigma_{hyd}}{\Delta V/V_o}$$

Where Dashed lines indicate initial stressed state

σ = uniaxial tensile or compressive stress

τ = shear stress

σ_{hyd} = hydrostatic tensile or compressive stress

ε = normal strain

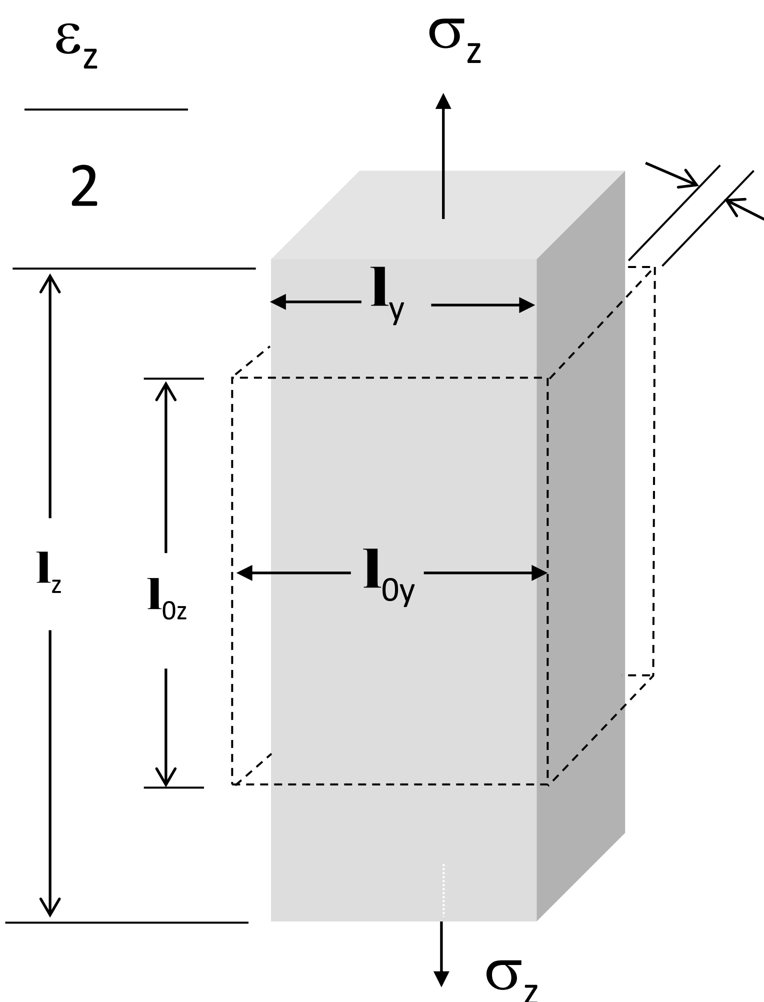
γ = shear strain

$\Delta V/V_o$ = fractional volume expansion or contraction

Poisson's Ratio

- Poisson's ratio, ν, is the ratio of transverse to axial strain

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



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

Poisson's Ratio

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

Relationship between moduli and Poisson's ratio for elastic isotropic materials

- Elastic Isotropic materials are materials in which properties at a point are the same in all directions. Some examples of isotropic materials are unoriented amorphous polymers and annealed glasses [1].

- If any of the two elastic constants of a homogenous (in which properties do not vary from point to point) isotropic material, the other two may be calculated [2].

$$E = 2G(1 + \nu) = 3B(1 + 2\nu)$$

1. Nielsen, Lawrence E., Mechanical Properties of Polymers and Composites, Marcel Dekker, Inc., New York, 1974, p. 1.

2. Hayden, H. W., Moffatt, W.G., and Wulff, J., The structure and Properties of Materials, Volume III, Mechanical Behavior, John Wiley & Sons, Inc, New York, 1965, p.26.

Comparison of Moduli and Poisson's Ratio

Material	E (GPa)	ν	G (GPa)
Steel	220	0.28	85.9
Copper	120	0.35	44.4
Glass	60	0.23	24.4
Granite	30	0.30	15.5
Polystyrene	34	0.33	12.8
Polyethylene	24	0.38	8.7
Natural Rubber	0.02	0.49	0.0067

Cowie, J.M.G., Polymers: Chemistry & Physics of Modern Materials, 2nd Edition, 1991, p. 275, ISBN 0 7514 0134 X

Modulus calculations in DMA

DMA850 and Q800	RSA G2
Stiffness (K) = Force / Displacement Modulus (E) = K x GF GF: Geometry factor. Clamp dependent Can be found in online help manual	Stress (σ) = Force x K_σ Strain (γ)= Displacement x K_γ Modulus (E) = σ / γ K_σ : Stress constant K_γ : Strain constant Clamp dependent Can be found in online help manual

$$GF = K_\sigma / K_\gamma$$

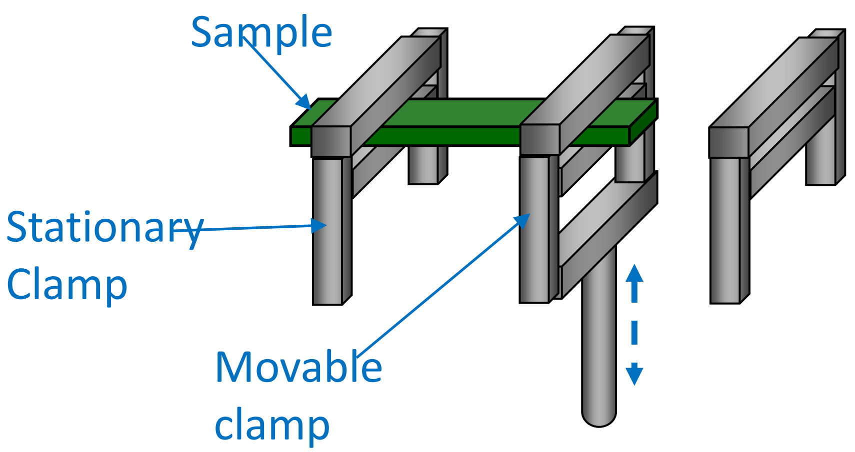
Choose the correct clamp for testing

- **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: **10^2 - 10^7 N/m**. Stiffness of sample related to its dimensions [L, w, t]. Stiffness may limit sample size to below clamp maximum.

DMA: Single Cantilever Clamp



- Sample which are stiff with a well-defined geometry allowing for sample dimensions can be measured accurately.
  - Precautions:
    - Soft samples (Tg < RT) with well-defined geometry such as elastomers may get pinched during clamping and cause errors in measurement.
    - Samples with high CTE can expand between the clamp faces and buckle, causing significant errors in measurement
- Mechanical properties, secondary transitions, T_g of polymers (thermoplastics/thermosets)
  - Measurement of modulus and tan δ
- Typical sample length is 17.5 mm. Smaller sizes available.
- Use a consistent clamping torque (typically 10 in-lbs)

Geometry Factor - Single Cantilever Clamp

Modulus = Stiffness × Geometry Factor

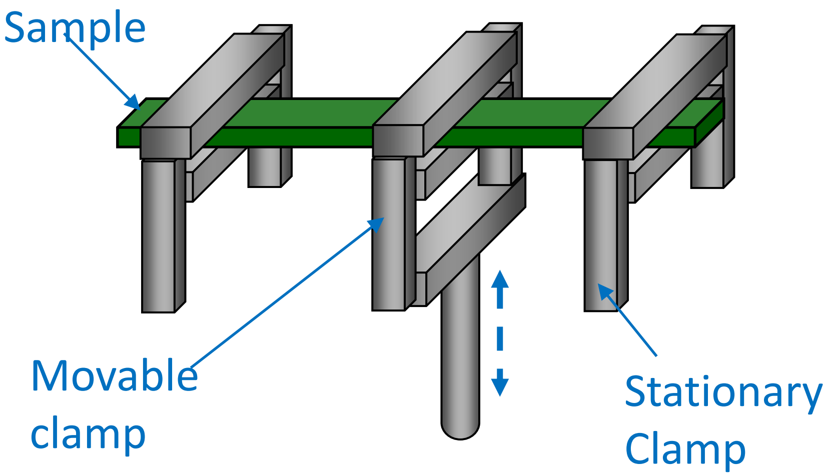
If length/thickness > 10, the contribution of the term containing the Poisson’s Ratio can be approximated to be negligible

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

$$GF_{SC} = \frac{l^3}{w t^3}$$

w = sample width
l = sample length
t = sample thickness

DMA: Dual Cantilever Clamp



- Samples which are stiff with a well-defined geometry allowing for sample dimensions can be measured accurately.
  - Precautions:
    - Soft samples (with Tg < RT) such as elastomers may get pinched during clamping and cause errors in measurement.
    - Samples with high CTE can expand between the clamp faces and buckle, causing significant errors in measurement
- Tracking cure of thermosets/composites, mechanical properties, secondary transitions and T_g of polymers (thermoplastics/thermosets)
  - Measurement of modulus and tan δ
- Typical sample length is 35 mm. Smaller sizes available. Good for materials that require a larger sample size for homogeneity
- Use a consistent clamping torque (typically 10 in-lbs)

Geometry Factor - Dual Cantilever Clamp

Modulus = Stiffness × Geometry Factor (GF)

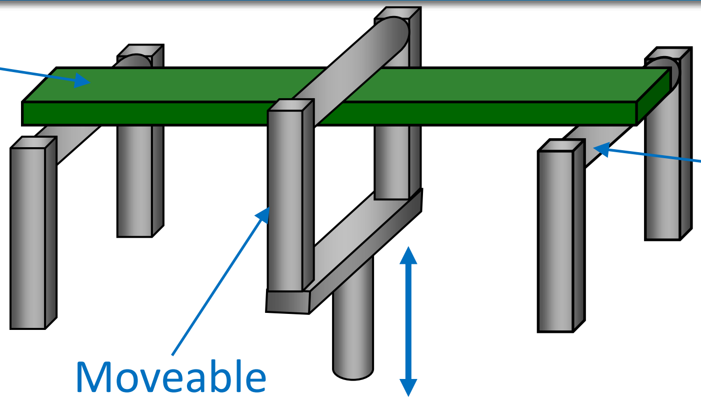
If length/thickness > 10, the contribution of the term containing the Poisson's Ratio can be approximated to be negligible

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

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

w = sample width
l = sample length
t = sample thickness

DMA: 3 Point Bend Clamp



- Conforms with ASTM standard test method for bending
- Purest deformation mode since clamping effects are eliminated
- Samples which are stiff with a well-defined geometry allowing for sample dimensions can be measured accurately.
  - Precautions:
    - Samples that get soft around T_g (typically unfilled thermoplastics) can sag and introduce errors in modulus measurements.
- Tracking cure of thermosets/composites, mechanical properties and T_g of polymers that are stiff past the glass transition (filled thermoplastics/thermosets/elastomers)
  - Measurement of modulus and tan δ
- Typical sample lengths 50 mm and 20 mm. Smaller sizes available.
- Sample alignment along the stationary fulcrum is important.

Geometry Factor - 3 Point Bending Clamp

Modulus = Stiffness × Geometry Factor

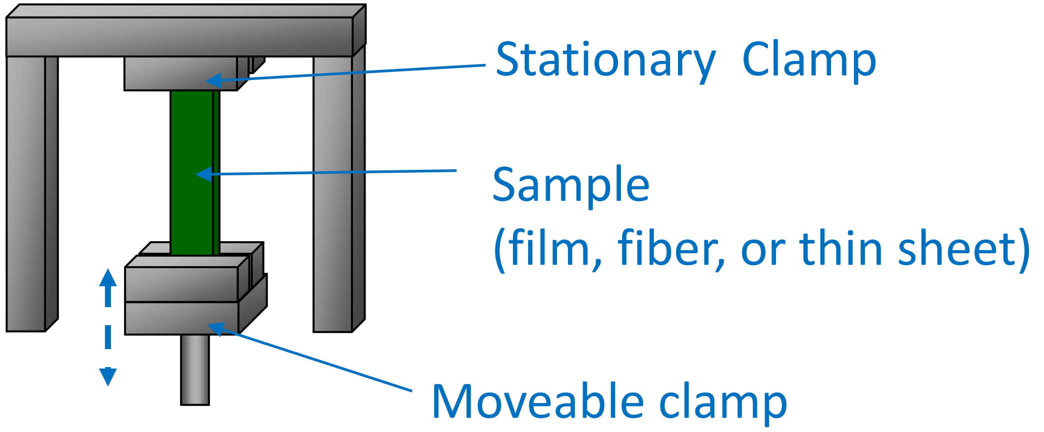
If length/thickness > 10, the contribution of the term containing the Poisson's Ratio can be approximated to be negligible

$$GF_{3PB} = \frac{\cancel{3}l^3\left[1+\cancel{\frac{6}{10}(1+\nu)\left(\frac{2t}{l}\right)^2}\right]}{\underset{4}{\cancel{12}}wt^3}$$

$$GF_{3PB} = \frac{l^3}{4wt^3}$$

w = sample width
l = sample length
t = sample thickness

DMA: Film/Fiber/Tension Clamp



- Films and fibers need to have a well-defined geometry allowing for sample dimensions can be measured accurately. Sample length is calculated automatically by the instrument.
- Applications
  - Mechanical properties, Tg, secondary transitions (modulus and tan δ)
  - Creep and stress relaxation
  - Temperature controlled constant force or displacement tests to understand processing effects and shrinkage
  - Generation of stress-strain curves
- Sample alignment between the clamps is important.
- Use a consistent clamping torque (typically 3-5 in-lbs)

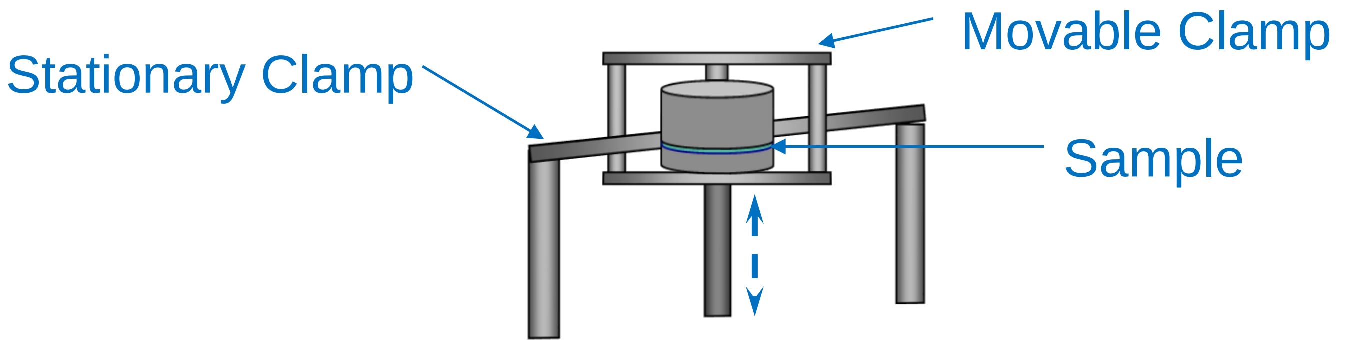
Geometry Factor – Film/fiber/tension clamp

Modulus = Stiffness × Geometry Factor

$$\mathrm{GF}_{\mathrm{Film}} = \frac{l}{wt}$$

w = sample width
l = sample length
t = sample thickness

DMA: Compression Clamp



- Good mode for low to medium modulus materials (gels,elastomers) which are compressible throughout the test temperature range
  - Precautions:
    - Samples that are incompressible (typically below the Tg) are difficult to test under compression
    - Samples that are too soft and cannot support the load of the clamp need alterations in sample dimensions to get meaningful measurements
- Option for penetration measurements (no modulus information in penetration, only transitions and tan δ)
- Applications:
  - Mechanical properties, Tg, secondary transitions (modulus and tan δ)
  - Creep and stress relaxation
  - Temperature controlled constant force or displacement tests to understand processing effects
- Alignment of plates attached to the moveable and stationary clamps is important.
- Sample diameter <= plate diameter (15 mm and 40 mm options). Use exact sample diameter

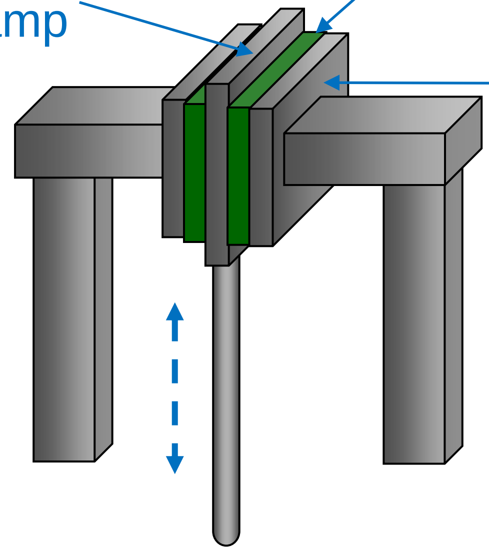
Geometry Factor – Film/fiber/tension clamp

Modulus = Stiffness × Geometry Factor

$$\mathrm{GF}_{\mathrm{Comp}} = \frac{thickness}{sample\ surface\ area} = \frac{t}{\pi r^2}$$

r = sample radius
t = sample thickness, between clamp faces

DMA: Shear Sandwich Clamp



- Good for evaluating highly damped soft solids such as gels and adhesives & elastomers > T_g
  - Precautions:
    - Samples should be able to support their own weight under gravity (no flow through the test temperature)
    - Clamping between the plates need to be consistent
- Applications:
  - Mechanical properties, Tg, secondary transitions (modulus and tan δ)
- Sample size <= plate size

Operating Range of the Shear Sandwich Clamp

Modulus = Stiffness × Geometry Factor

$$\mathrm{GF}_{\mathrm{Shear}} = \frac{3t}{5wh}$$

w = sample width, i.e. horizontal dimension
h = sample height, i.e. vertical dimension
t = sample thickness, between clamp faces

Changing Sample Stiffness

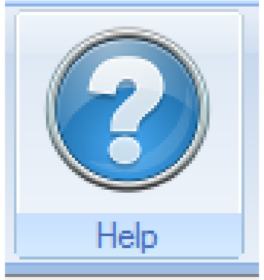
Clamp Type	To Increase Stiffness…	To Decrease Stiffness…
Tension Film	Decrease length or increase width. If possible increase thickness.	Increase length or decrease width. If possible decrease thickness.
Tension Fiber	Decrease length or increase diameter if possible.	Increase length or decrease diameter 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

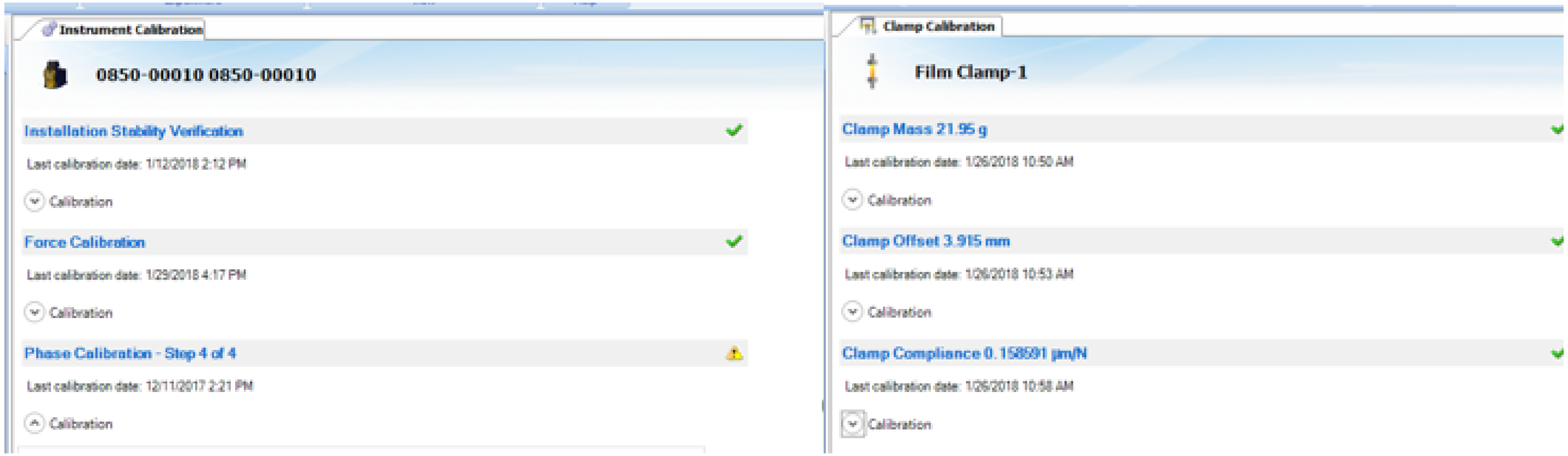
Sample	Clamp	Sample Dimensions
High modulus materials and composites	3-point Bend Dual Cantilever Single Cantilever	L/T> 10 if possible
Unreinforced thermoplastics or thermosets	Single Cantilever Dual cantilever	L/T >10 if possible
Brittle solid (ceramics)	3-point Bend	L/T>10 if possible
Elastomers	3-point bend Tension	L/T>10 if possible T<1 mm
Films/Fibers	Tension	L 10-20 mm T<2 mm
Supported Systems	8 mm Dual Cantilever	minimize sample, put foil on clamps

Instrument calibration

DMA850 Flow Chart of Calibration Procedures



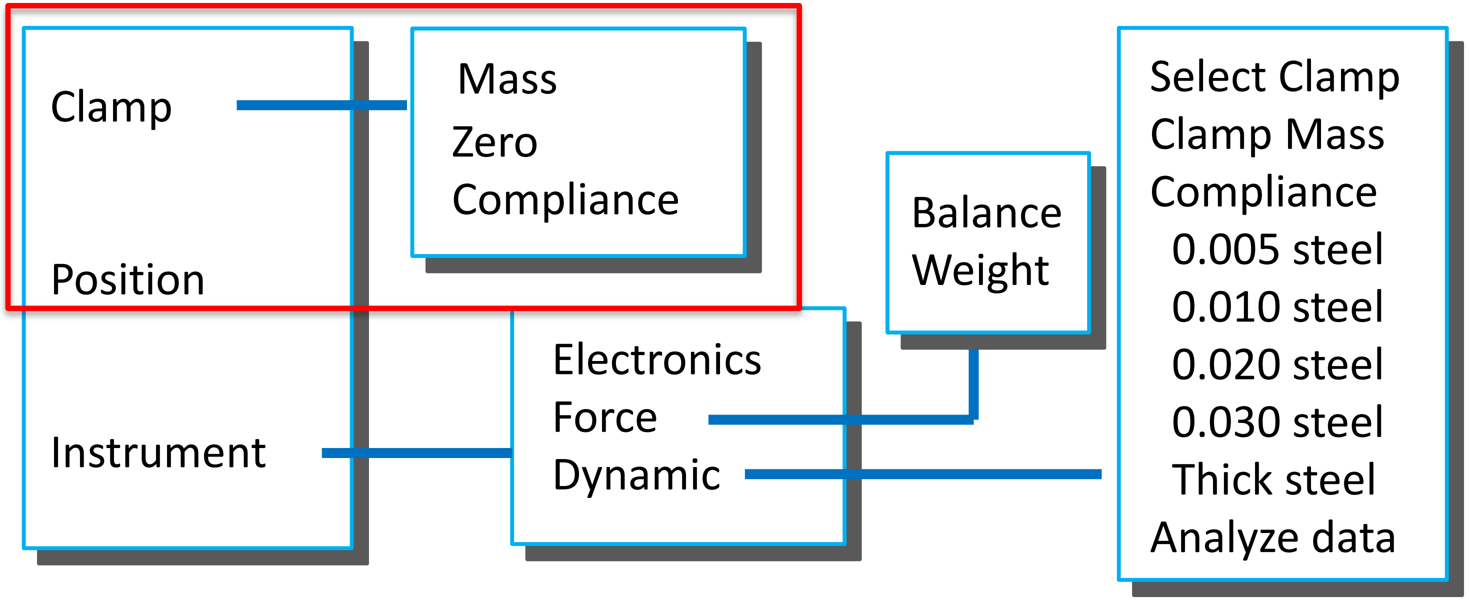
- Follow the online help manual
- The stability verification is performed at installation
- Instrument calibration includes 2 steps: force and phase
- Clamp calibration: perform when newly attached.
- Position calibration from touchscreen



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

Q800 Flow Chart of Calibration Procedures

- Instrument calibration includes 3 steps: Electronics, Force, and Dynamic
- Position calibration: calibrate the absolute position of the drive shaft. Perform this calibration when DMA is moved, reset, or powered down.
- Clamp calibration: perform when newly attached.



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

RSA G2 Flow Chart of Calibration Procedures

- Follow the online help manual
- Instrument calibration: force and phase angle check
- Clamp calibration: mass (perform when newly attached)

Help

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
Temperature Offset Table Calibration	Suggested: As needed

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

Available DMA Experiments

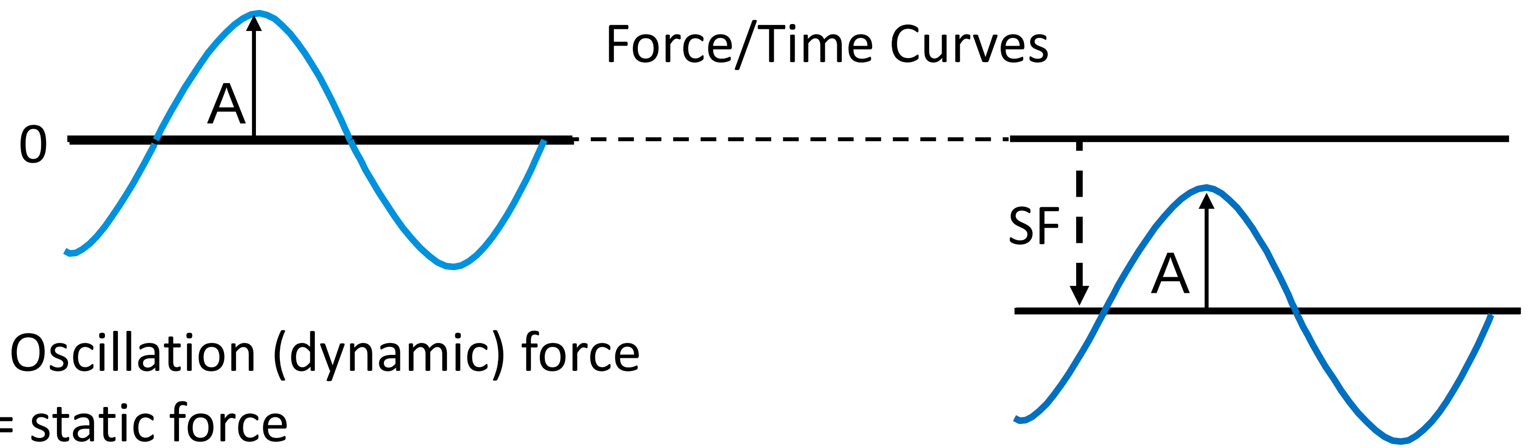
(1) Dynamic Tests

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!

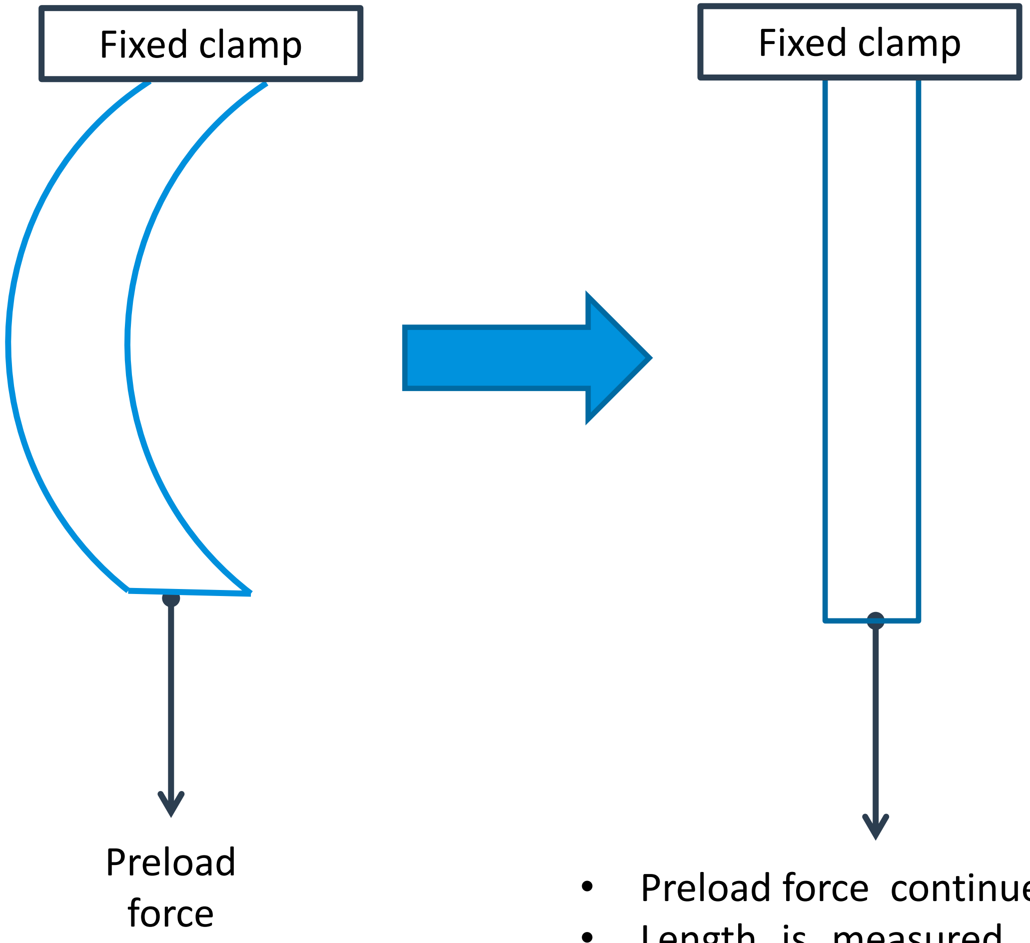


A = Oscillation (dynamic) force
SF = static force

<u>Clamps **without** static force:</u>
- Single Cantilever
- Dual Cantilever
- Shear Sandwich

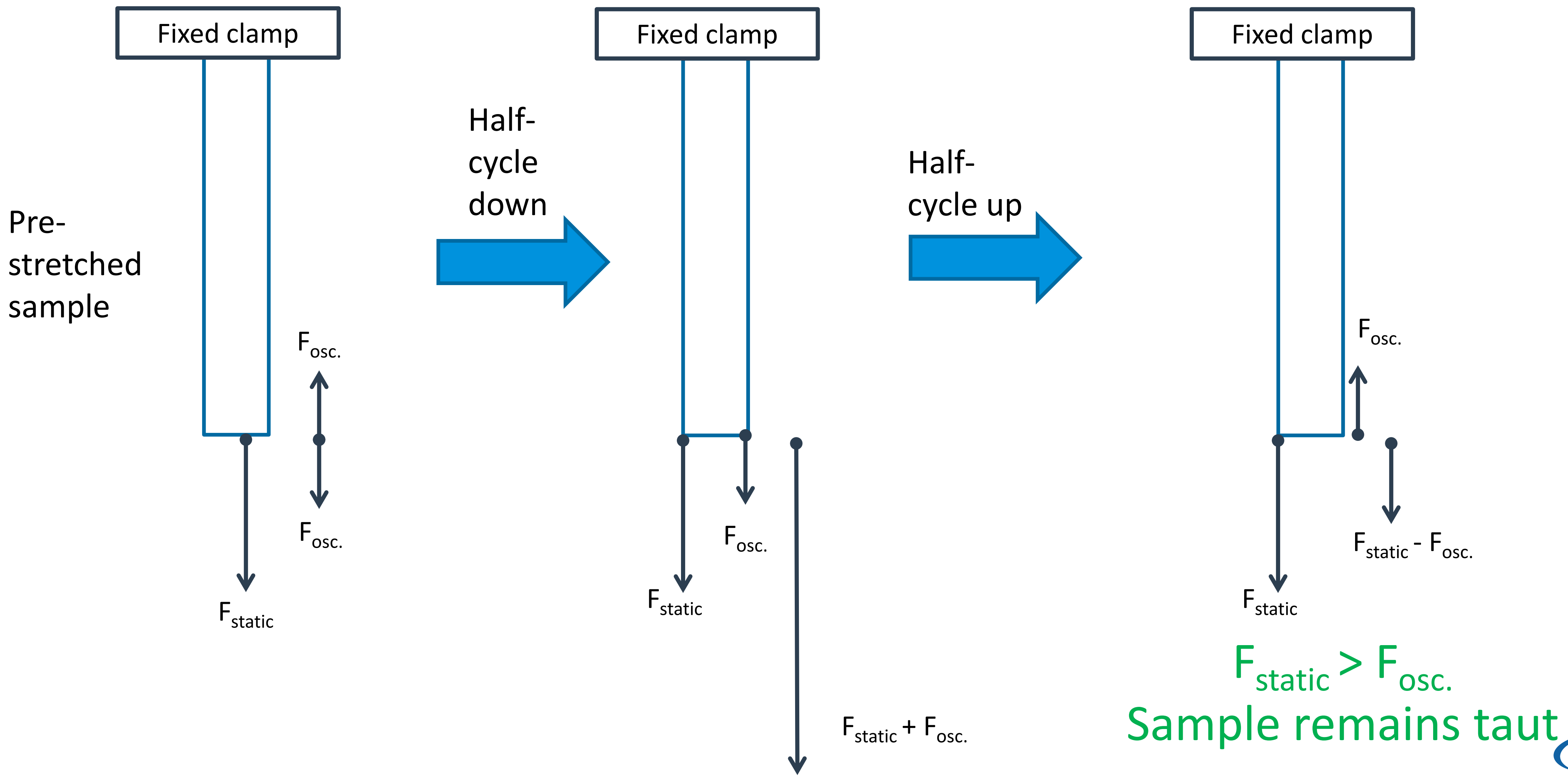
<u>Clamps **with** static force:</u>
- Tension Film
- Tension: Fiber
- 3-Point Bend
- Compression
- Penetration

Preload force in tension

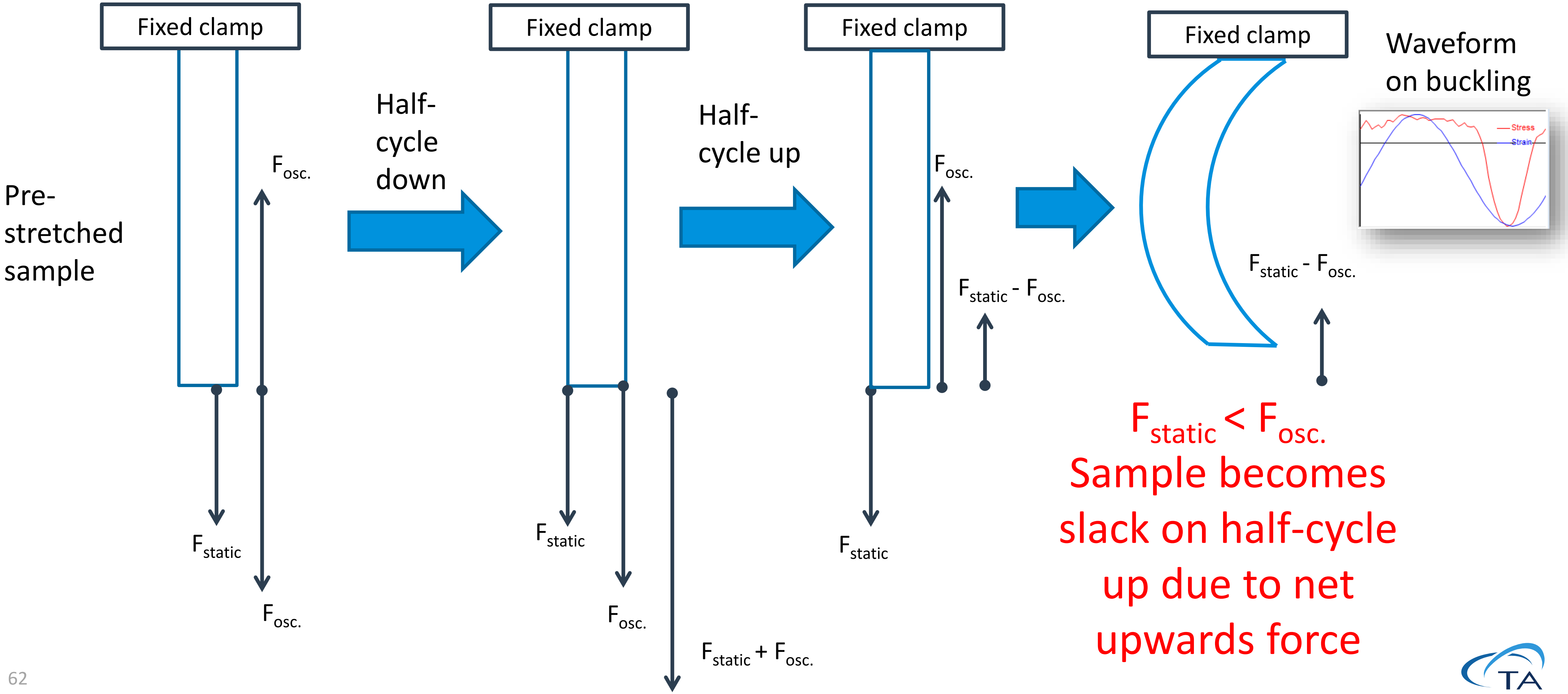


- Preload force continues to act.
- Length is measured and updated automatically.

Net forces acting during oscillation (tension)
static force > osc. force



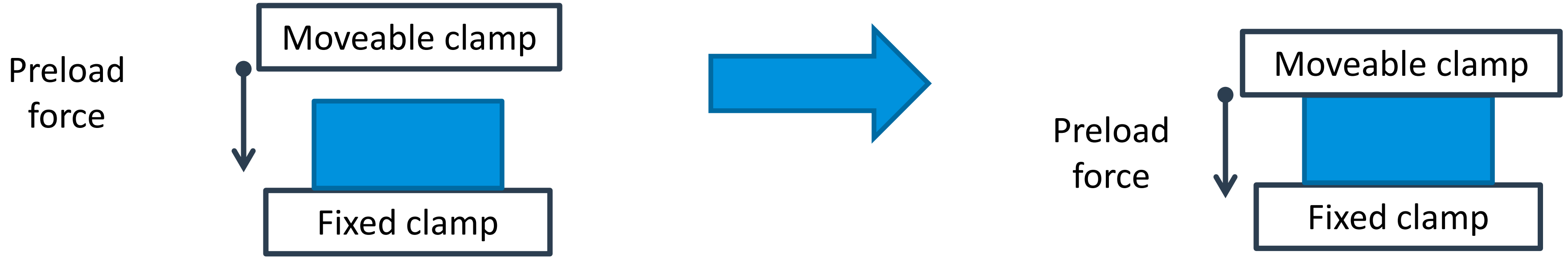
Net forces acting during oscillation (tension) static force < osc. force



$F_{static} < F_{osc.}$
Sample becomes slack on half-cycle up due to net upwards force

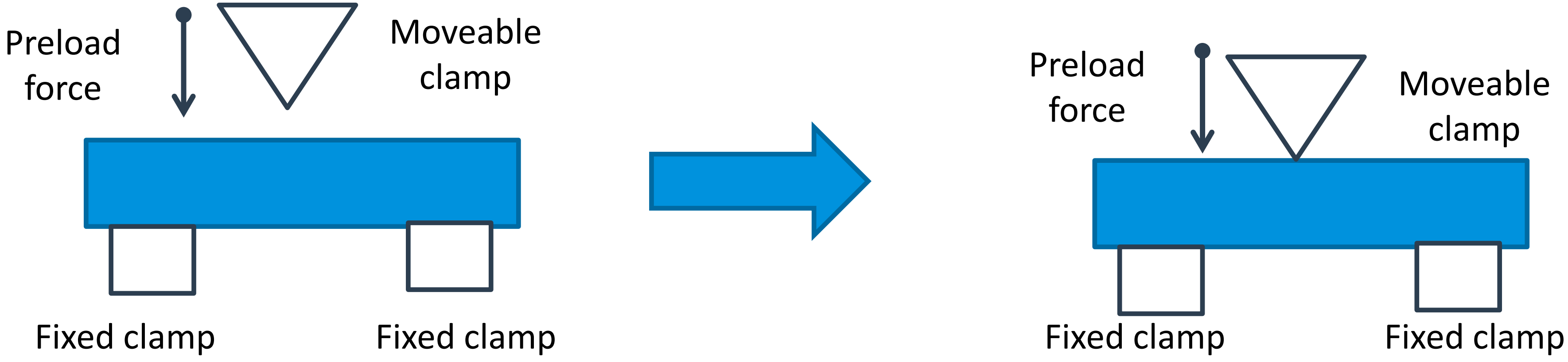
Preload force in compression, 3-point bending

COMPRESSION



- **Preload force continues to act.**
- **Thickness is measured and sample information is updated automatically.**

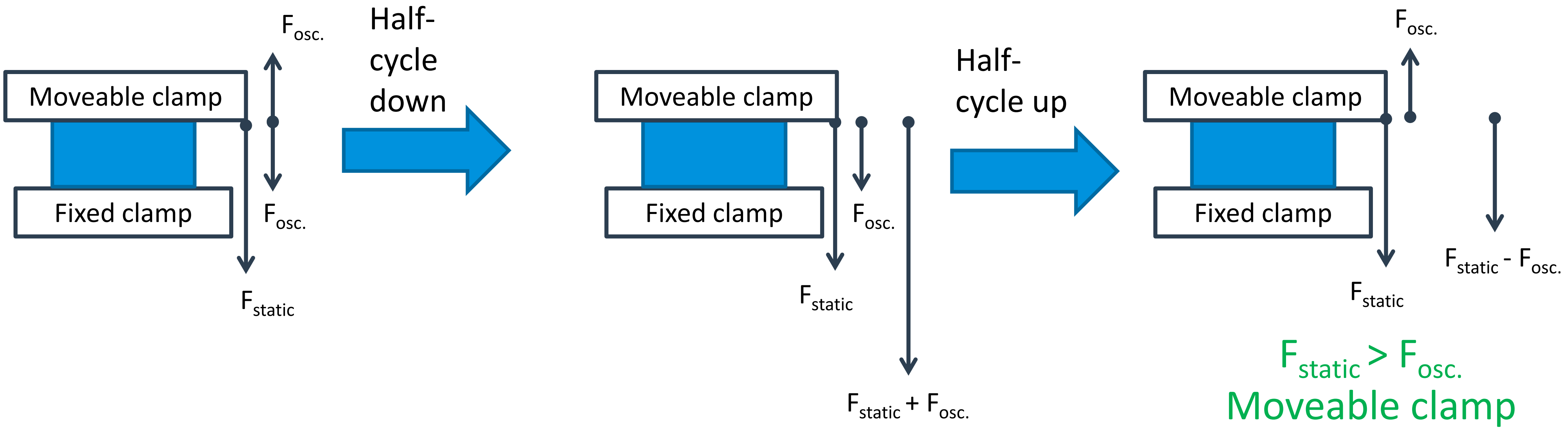
3-POINT BENDING



- **Sample dimensions are entered manually prior to start of experiment (no automatic update of thickness)**

- **Preload force continues to act.**

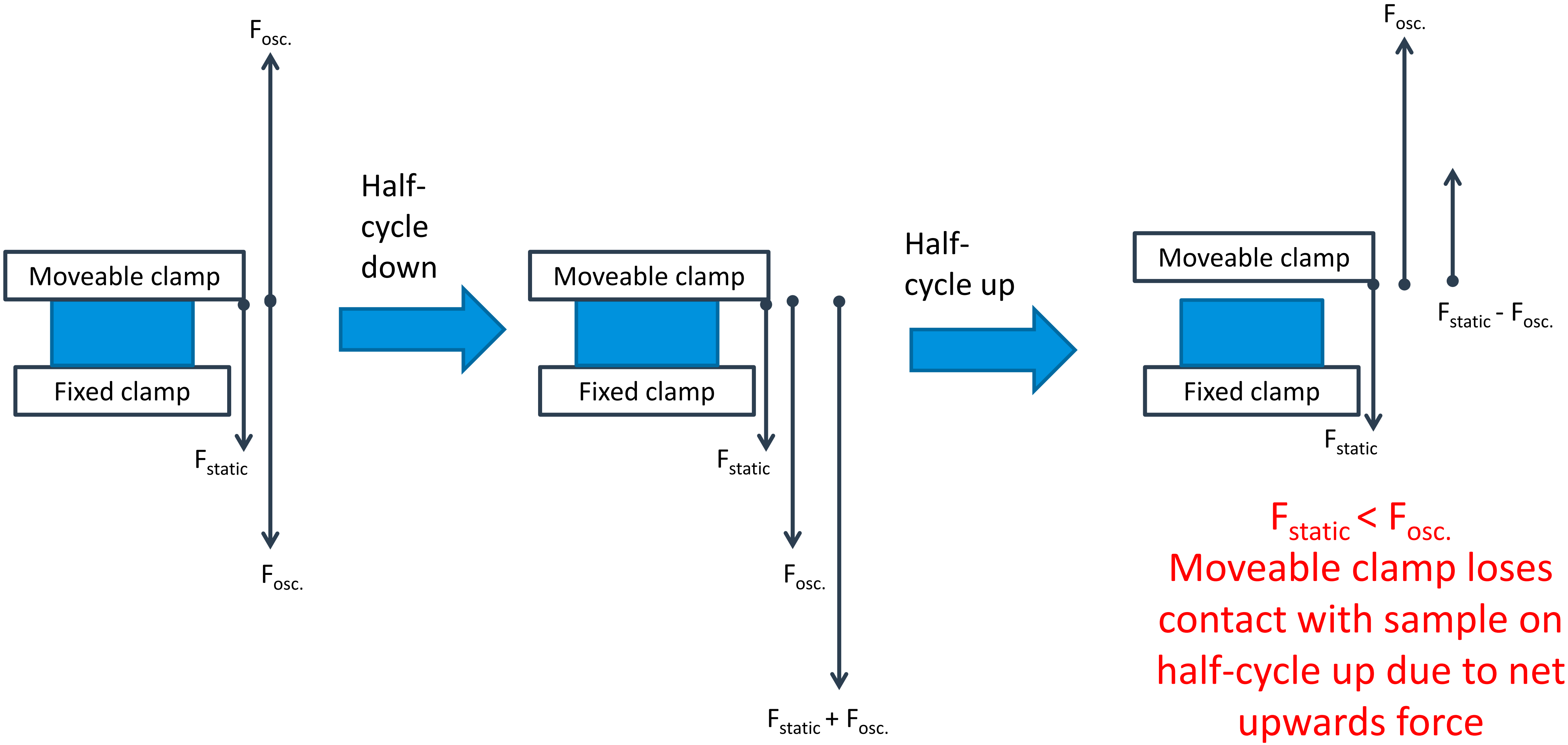
Net forces acting during oscillation (compression and 3PB) static force > osc. force



$F_{static} > F_{osc.}$

Moveable clamp maintains contact with sample at all times due to net downward force

Net forces acting during oscillation (compression) static force < osc. force



$F_{static} < F_{osc.}$

Moveable clamp loses contact with sample on half-cycle up due to net upwards force

Force track

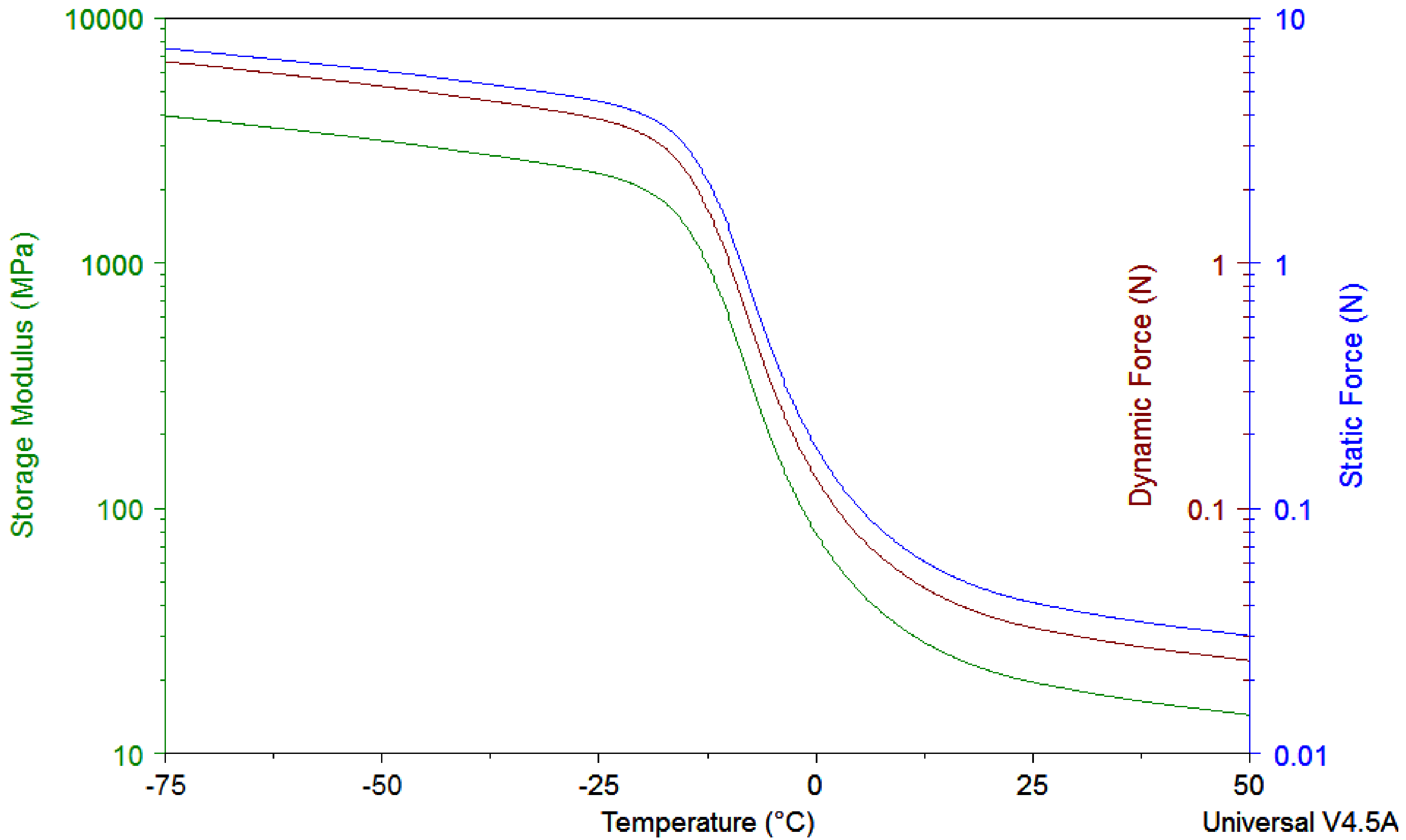
- Recap: Desired situation for all clamps is that $F_{static} > F_{osc}$
- Force track = $\frac{F_{static}}{F_{osc}}$
- If $\frac{F_{static}}{F_{osc}} > 1$, then **$F_{static} > F_{osc}$**
- Force track ratio is expressed as a percentage
  - On 850 and 800, Force track = $\frac{F_{static}}{F_{osc}}$ x 100%
  - On RSA-G2, Force track = $\left(\frac{F_{static}}{F_{osc}} - 1\right)$ x 100%

Benefits of using force track

- Force track ensures that static force exceeds oscillation force throughout the experiment
- Values from 125-150% (850/Q800) or 25-50% (RSA-G2) is a good starting point for most samples
- **Decreases static force in proportion to sample modulus in "Tension clamps" to reduce stretching as specimen weakens on increasing temperature.**
- Constant (or static) force can be used as long as static force > oscillation force through out the entire experiment.
  - Stiff samples in 3-point bending (thermosets)

Temperature Ramp with Force Track

- Q800 uses the term “Dynamic Force” to denote oscillation force (F_{osc})
- Static Force tracks Dynamic Force throughout Temperature Ramp to prevent over-stretching

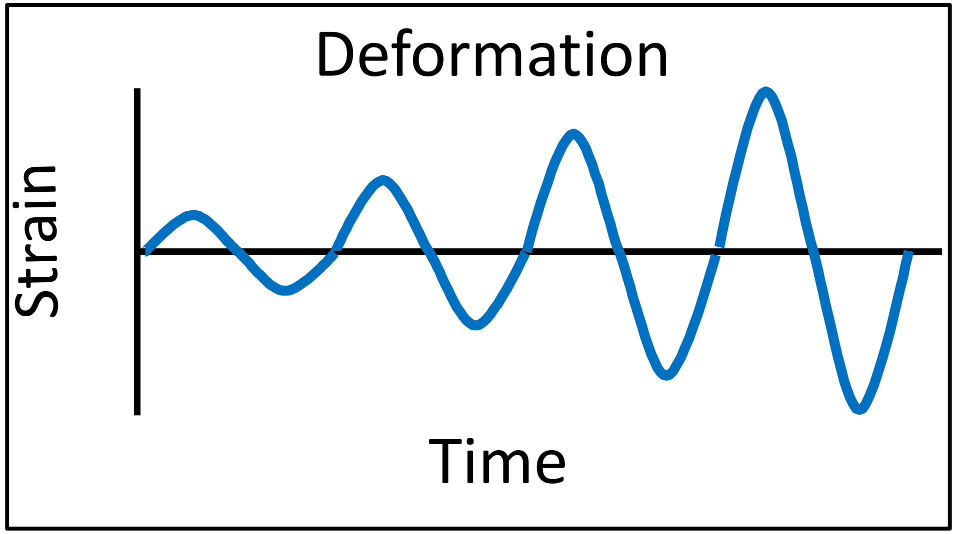


Choosing Force Track Parameters

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.
- **Refer to appendix for screenshots of setting force track parameters for your respective instrument**

Dynamic Strain (Stress) Sweep

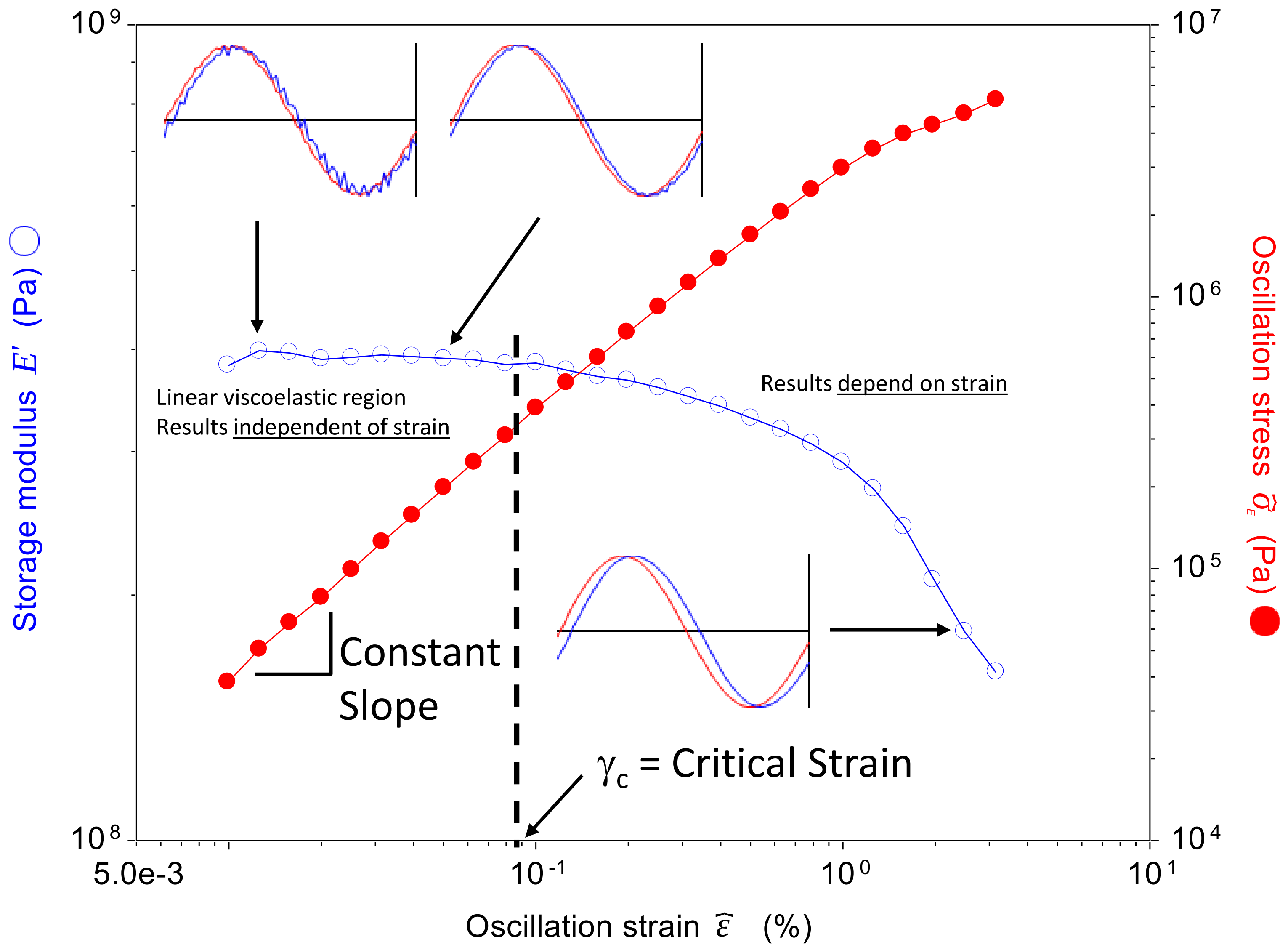


The material response to increasing deformation amplitude is monitored at a constant frequency and temperature.

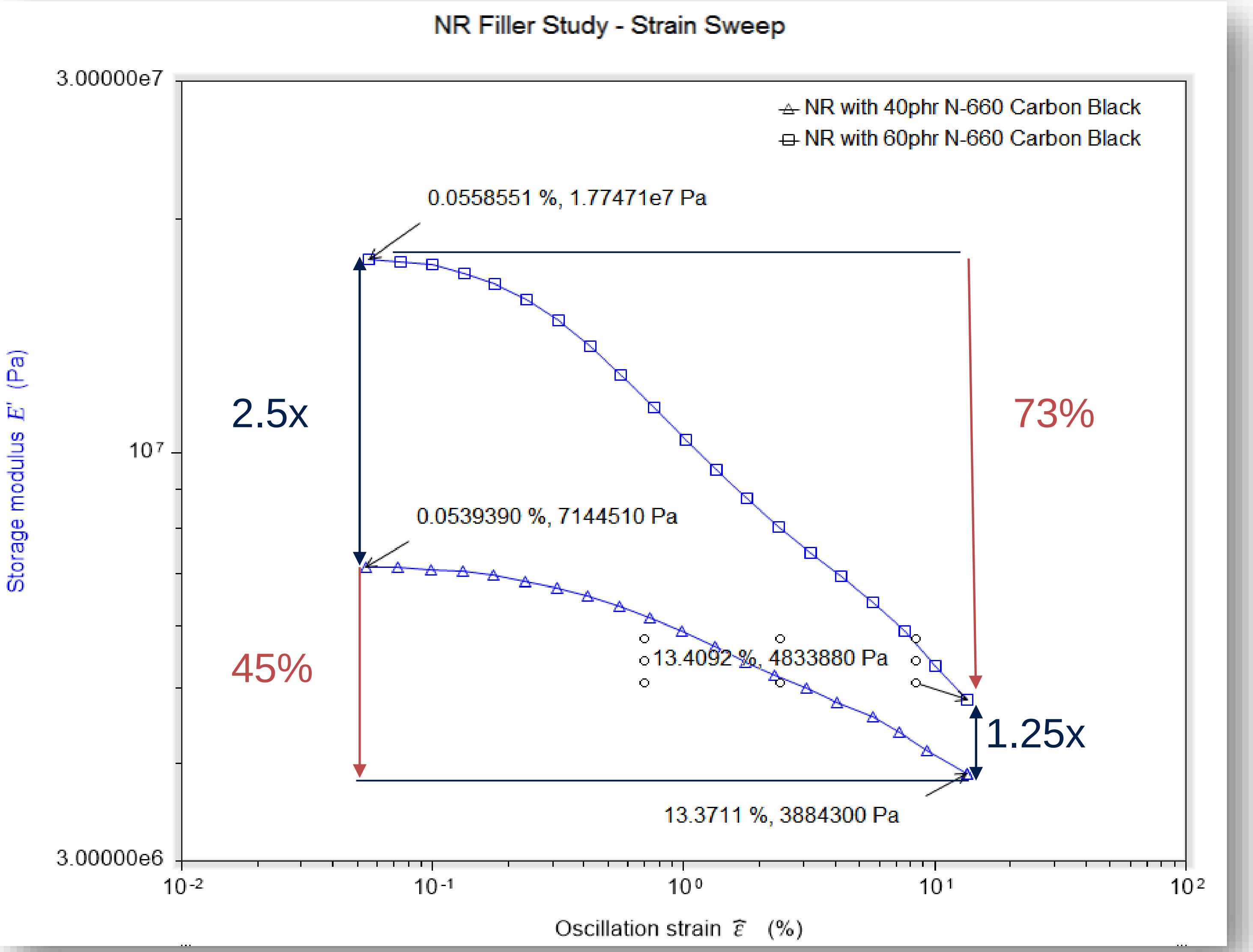
USES

- Identify Linear Viscoelastic Region
- Resilience/elasticity

Dynamic Strain Sweep: Material Response

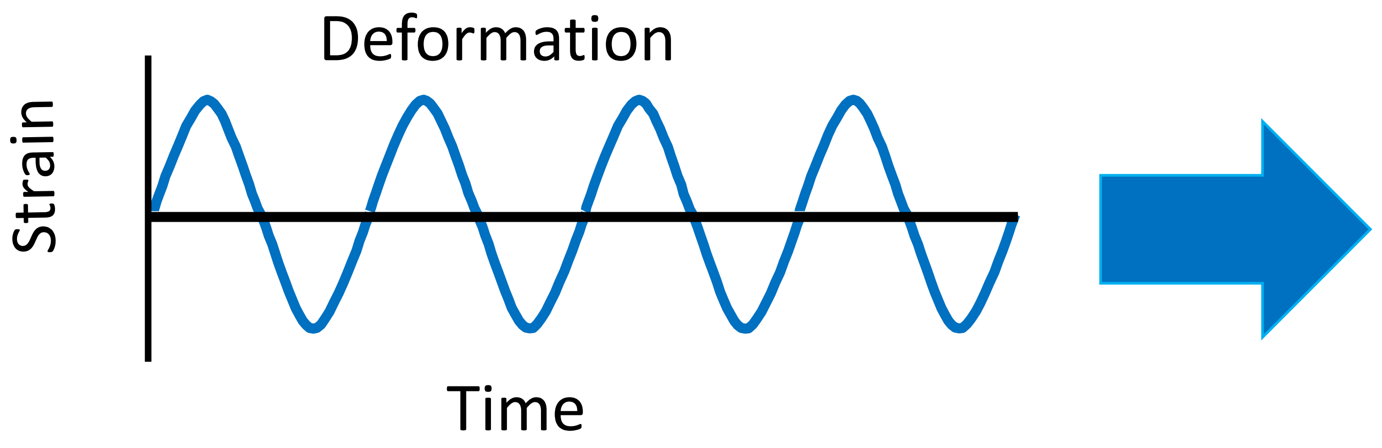


Rubber: Effect of Filler Content in Tire Rubber (NR)



- Instrument: TA Instruments ElectroForce DMA 3200 with FCO and ACS-3
- Samples: Cured Natural Rubber Compound with Two Filler Levels (40 & 60 PHR)
- Clamp: Tension
- Method: Strain Sweep at 10Hz

Dynamic Time Sweep

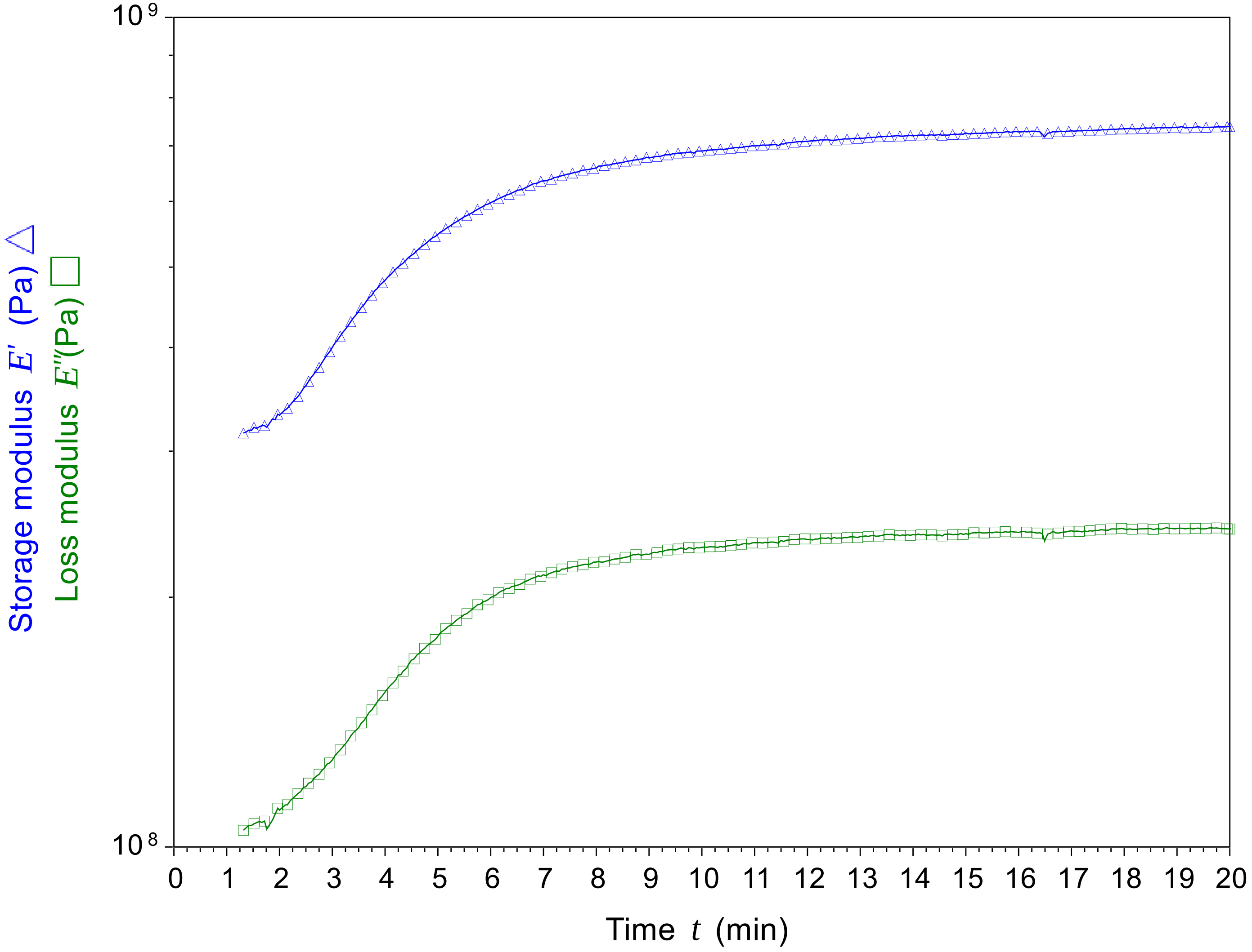


The material response is monitored at a constant frequency, amplitude and temperature.

USES

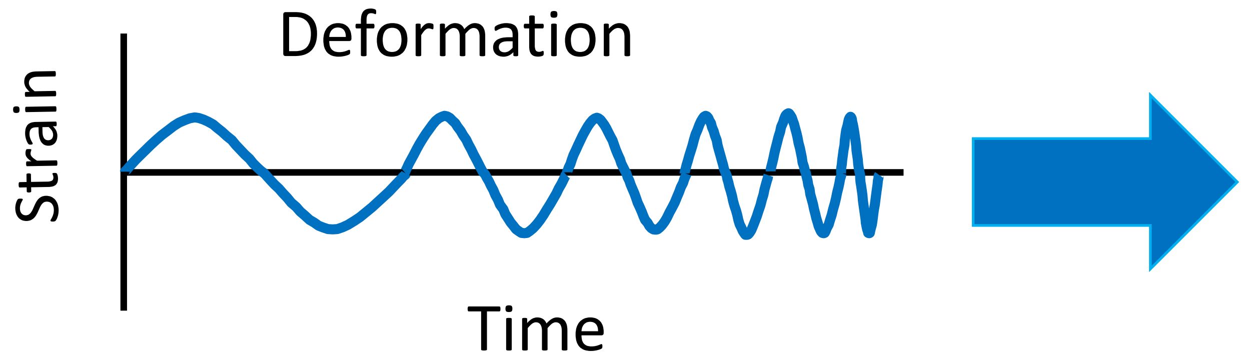
- Curing studies
- Fatigue tests
- Stability against thermal degradation

Epoxy Curing on Glass Braid



Instrument: DMA850
Clamp: Dual cantilever
Sample: Epoxy coated on glass braid
Dynamic time sweep
Temperature: 35°C
Frequency: 1 Hz
Amplitude: 10 μm

Frequency Sweep



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

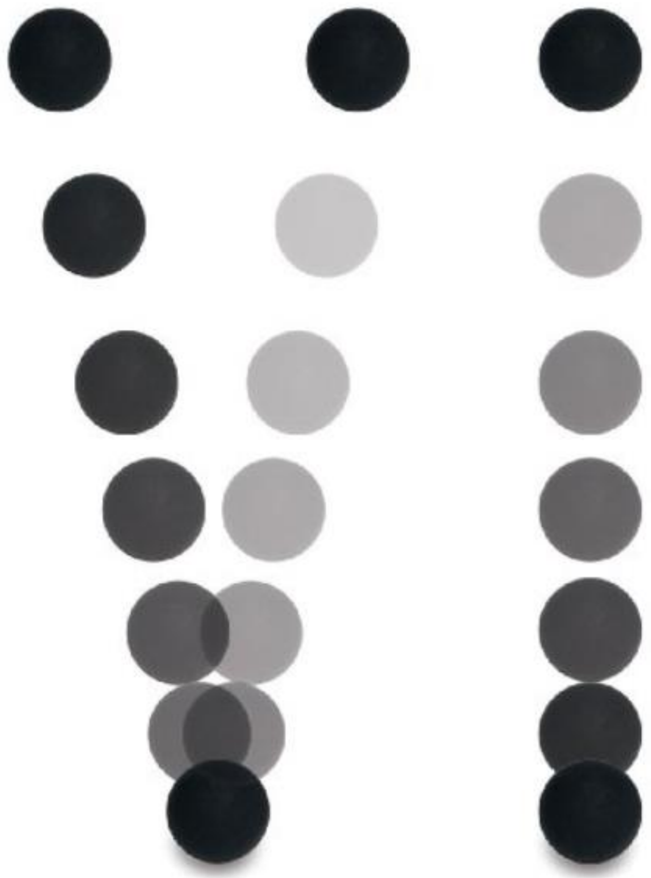
USES

- Quick comparison on modulus and elasticity on solids
- Study polymer melt processing (shear sandwich).
- Estimate long term properties with extended frequency (time) range using TTS

Happy and sad balls vide

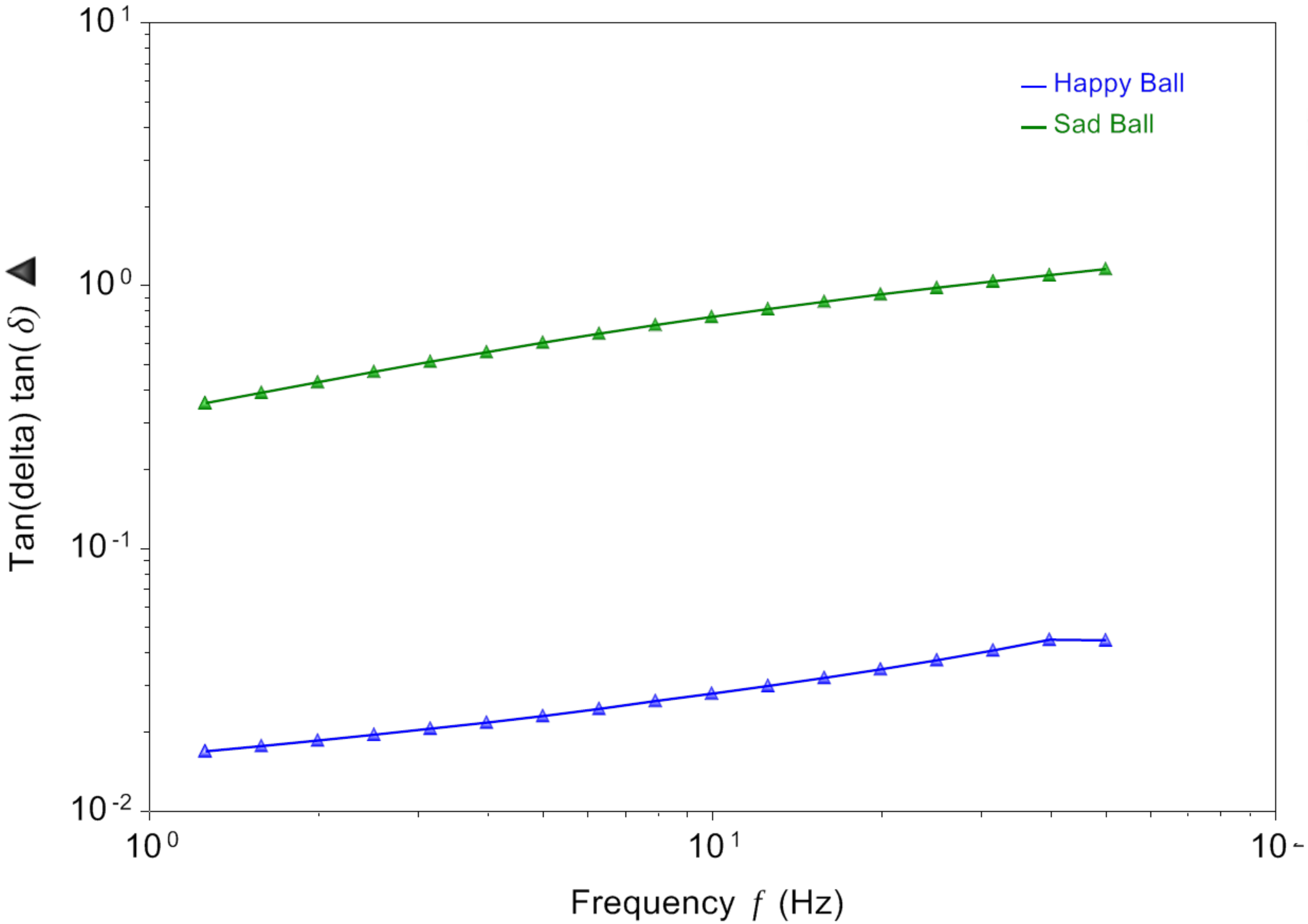
- Set of 2 black rubber balls used as demonstration of viscoelastic behavior.
  - When dropped, the Happy Ball bounces and the Sad Ball does not.

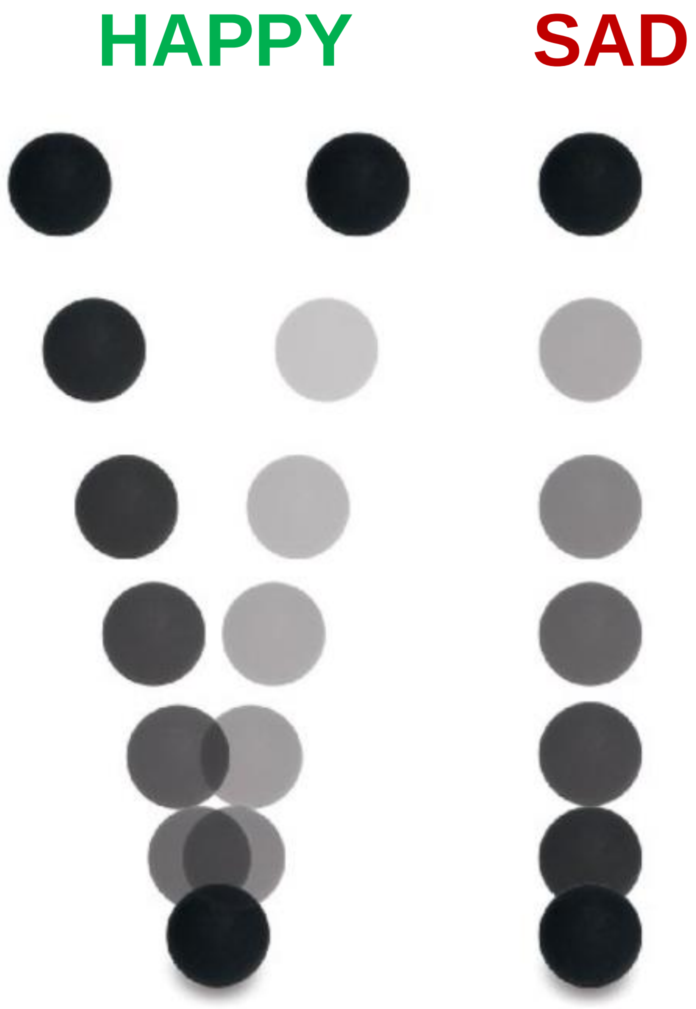
HAPPY SAD



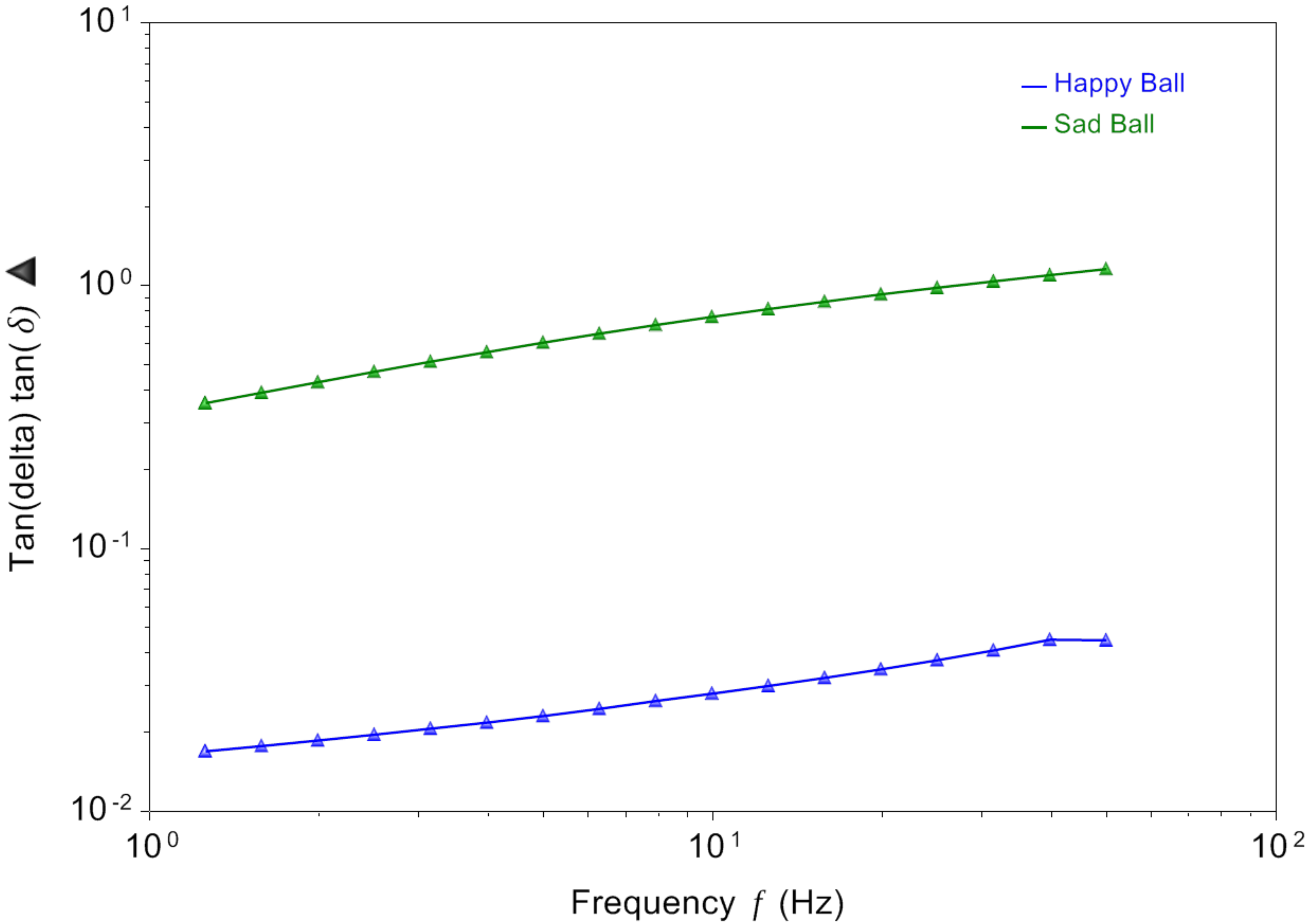
https://www.youtube.com/watch?v=lubaukB6B34

Dynamic Mechanical Properties: Tan Delta





Dynamic Mechanical Properties: Tan Delta



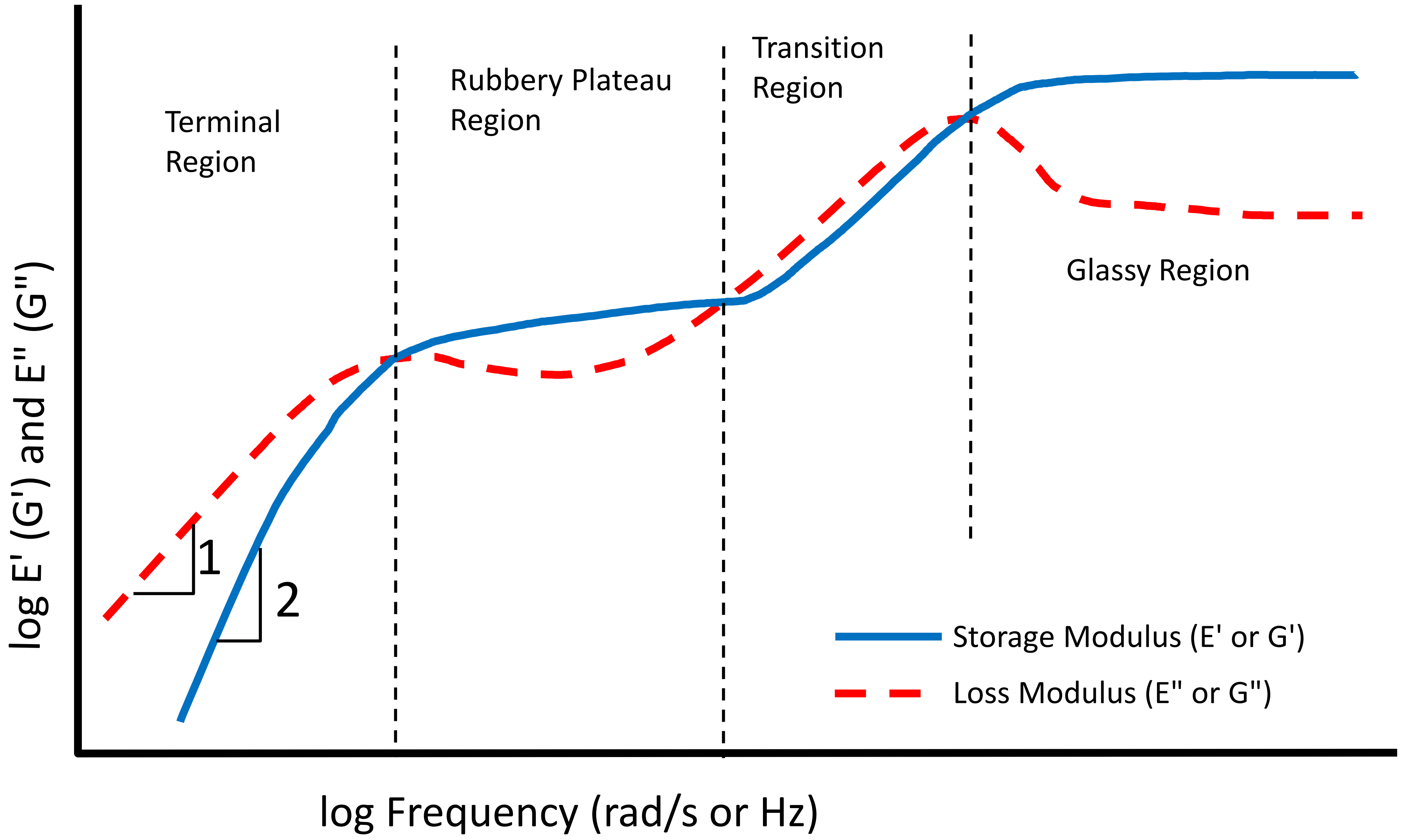
- tan δ: Measure of material damping. Balancing viscous property while having the appropriate level of stiffness.

- Increasing tan δ implies a greater potential for energy dissipation and lower elasticity, and vice-versa.

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

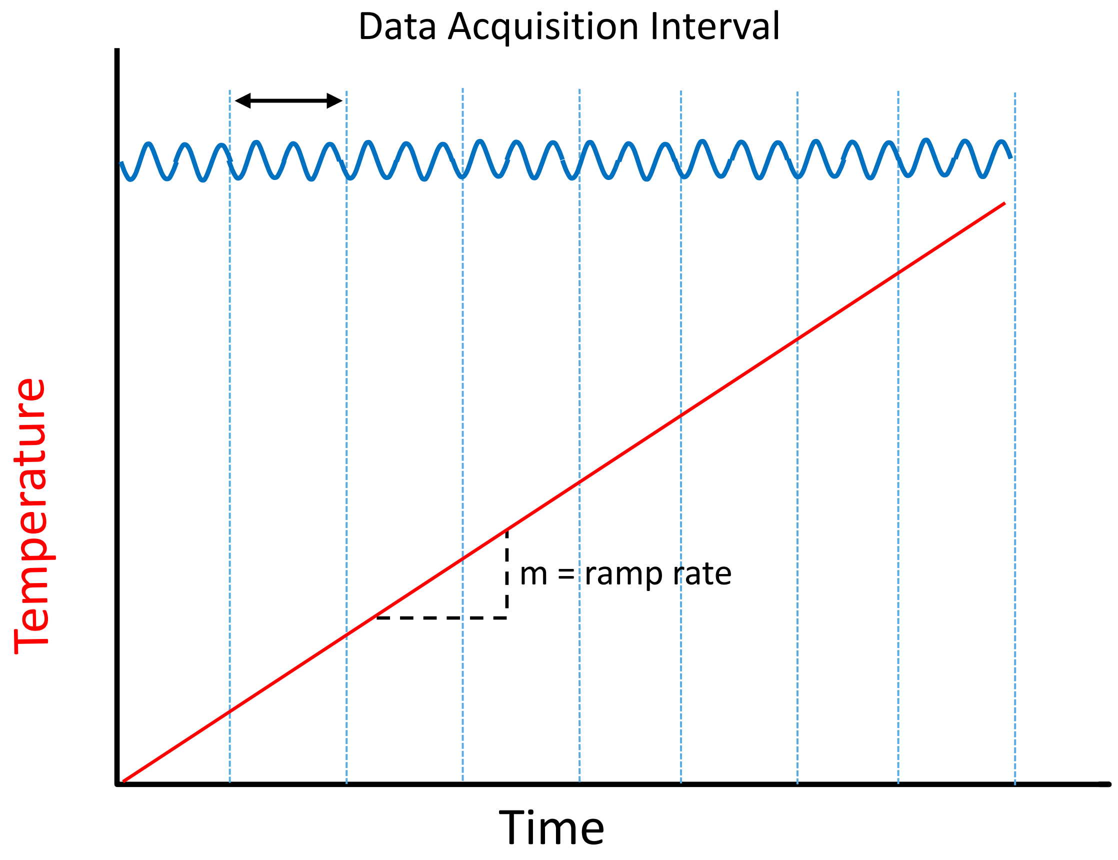
- Sad ball has a much higher tan δ compared to the happy ball

Frequency Sweep: Material Response



Dynamic Temperature Ramp

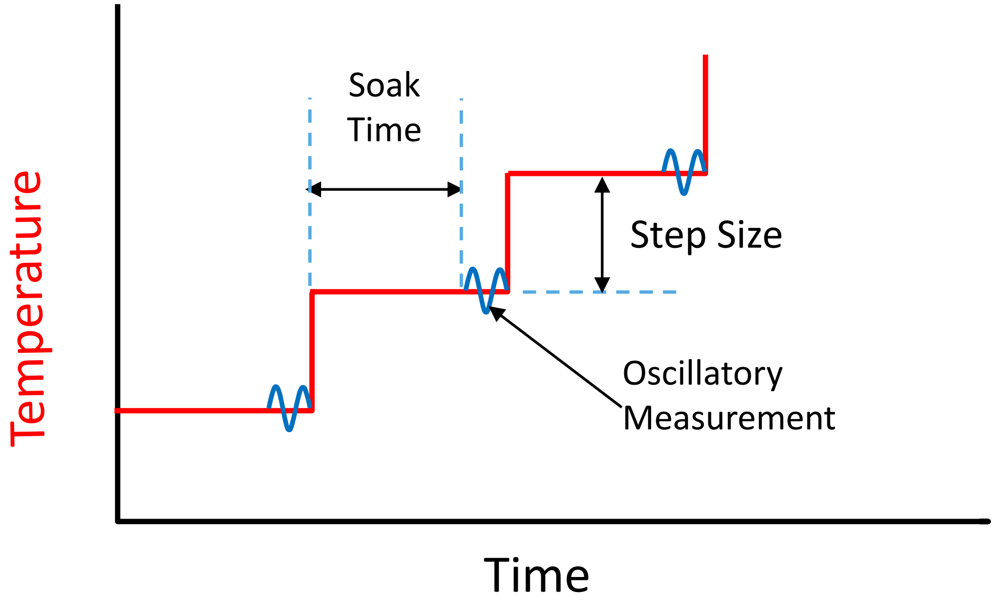
- A linear heating rate is applied. The material response is monitored at a constant frequency and constant amplitude of deformation. Data is taken at user defined time intervals.

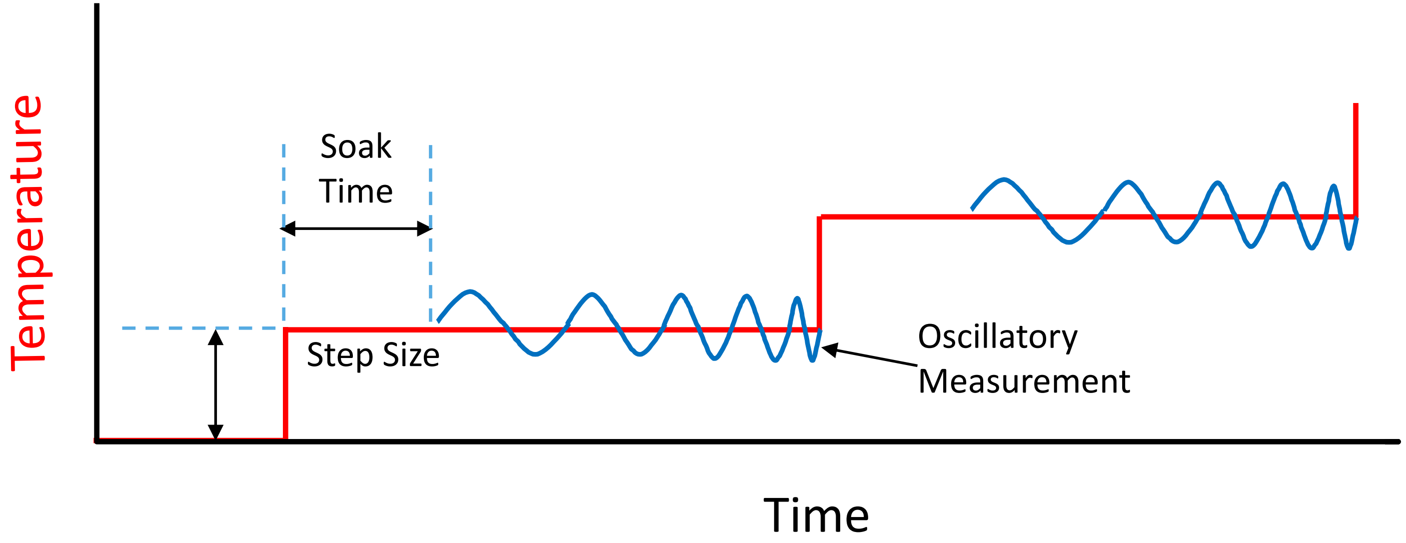


Recommend ramp rate for polymer testing: 1-5˚C/min.

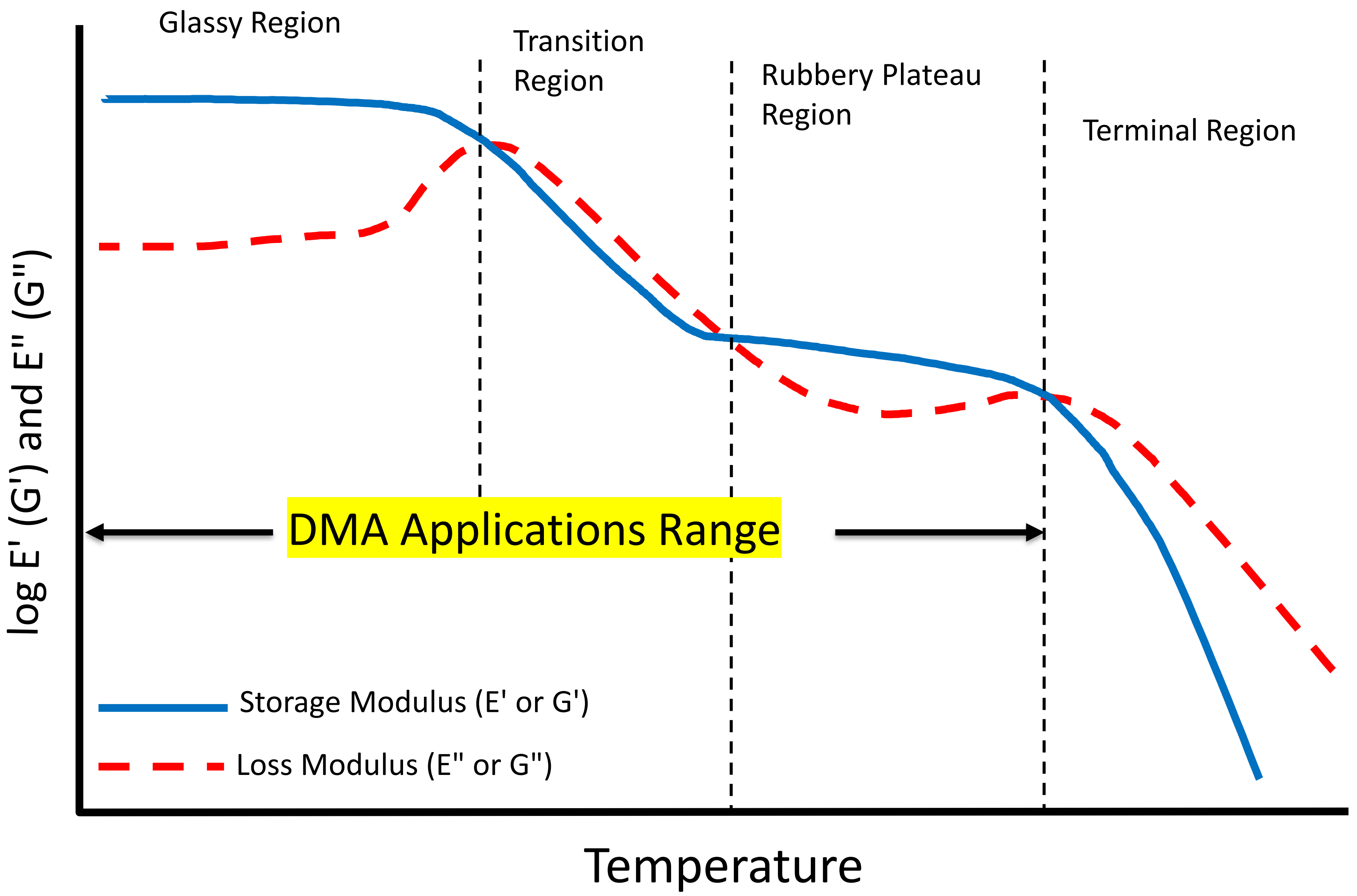
Temperature Step & Hold- Single /Multi-Frequency

- 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

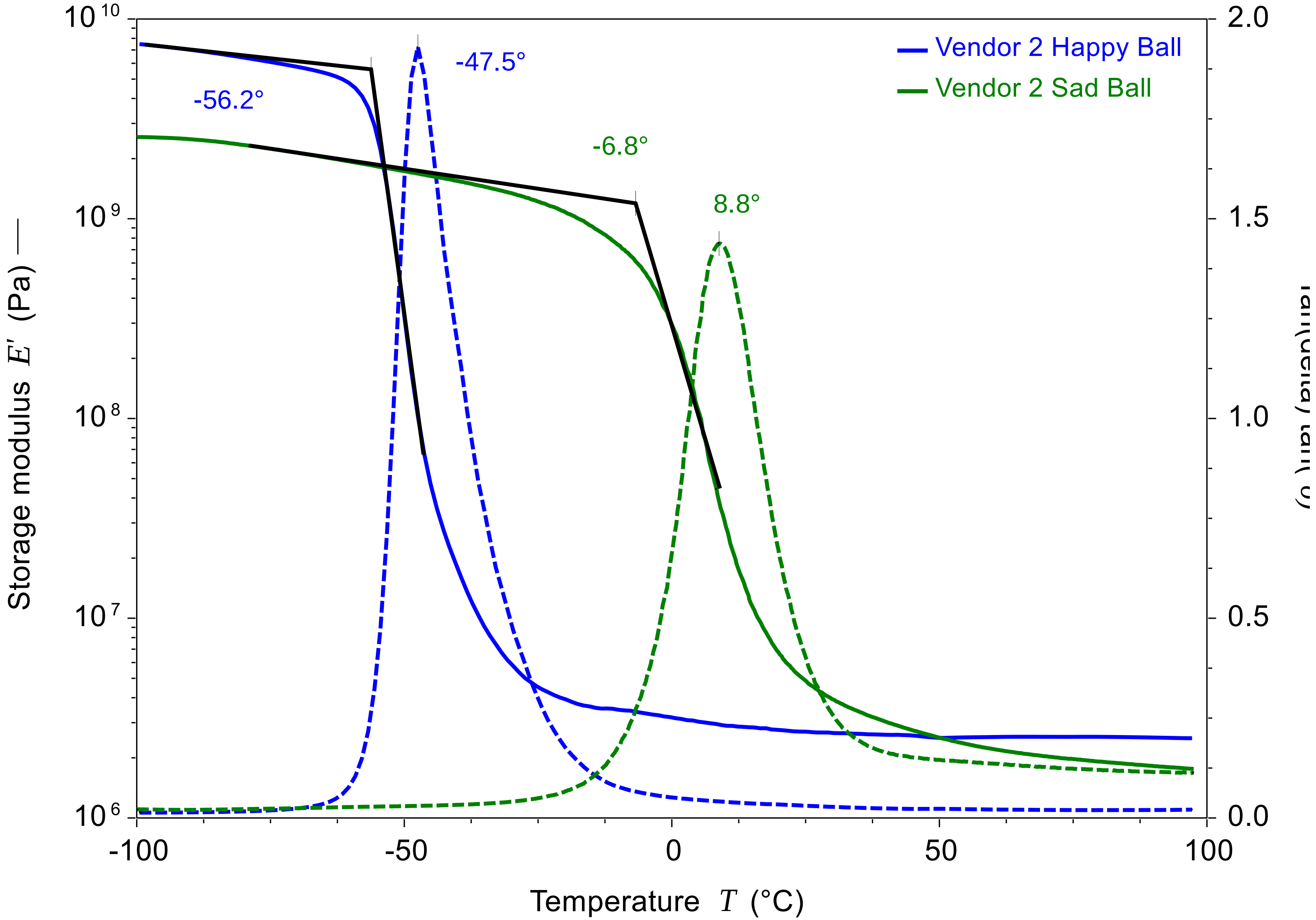


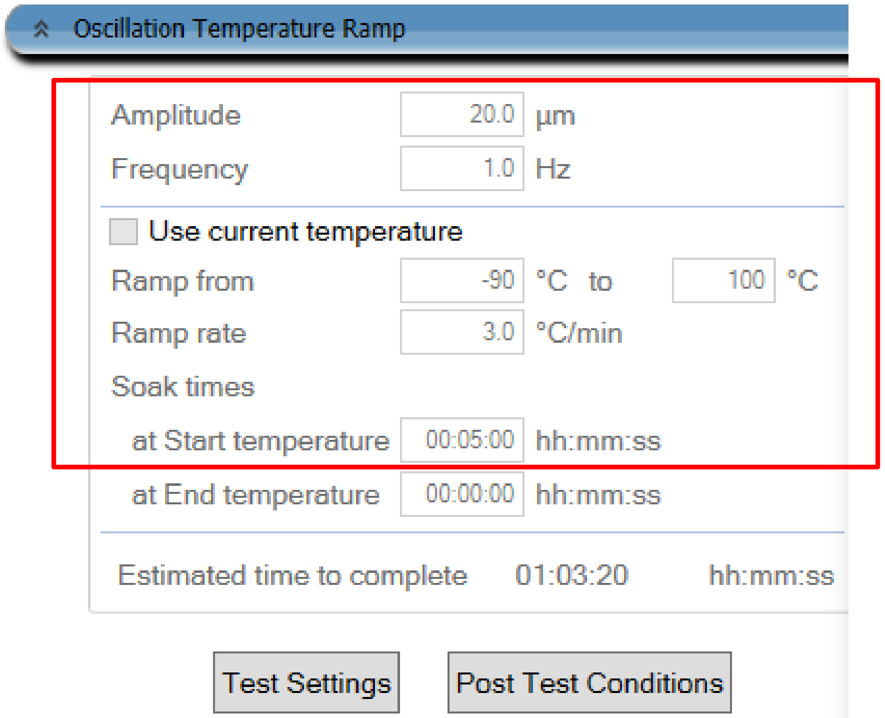


Temperature Profile on Amorphous Polymers



DMA temperature ramp: Happy and sad balls





Experimental Considerations

- 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 Appropriate Amplitude and Force

- Strain consideration
  - Must be within the linear region
- Force consideration
  - Maximum - 18 N on Q800, DMA850
  - Maximum - 35 N on RSA G2
- Yielding /Creep
  - If the force is too high the specimen may deform irreversibly
  - Must consider behavior at all temperatures and frequencies
- Noise
  - Higher amplitude = lower noise (generally)
  - Trade off against yielding/creep behavior

Frequencies

- Single Frequency
  - In a temperature ramp the most commonly used frequency is between 1 to 10 Hz (6.28 or 63 rad/sec)
- Multiple Frequencies
  - For an ambient test the commonly used frequency range is from 0.1 – 10Hz.
  - Frequency sweeps at multiple temperatures for Time-Temperature Superpositioning (TTS)
  - Run from high to low frequencies for faster initial data acquisition (for DMA850 and Q800 users)
- Data Collection Rate
  - Lower frequencies take longer time - control experiment
  - More frequencies = longer experiment

Temperature Program

- Temp ramp
  - Commonly used heating rate: 1-5°C/min
  - Larger samples have more thermal lag
  - Use slower ramp rates for lower frequencies and frequency sweeps because these take more time
- Temp sweep
  - No thermal lag but time consuming
  - Commonly used for TTS testing, typical temp step: 5-10°C
- Multiple temp steps
  - Commonly used to mimic certain application temperature profile

Available DMA Experiments

(2) Transient Tests

Transient Testing

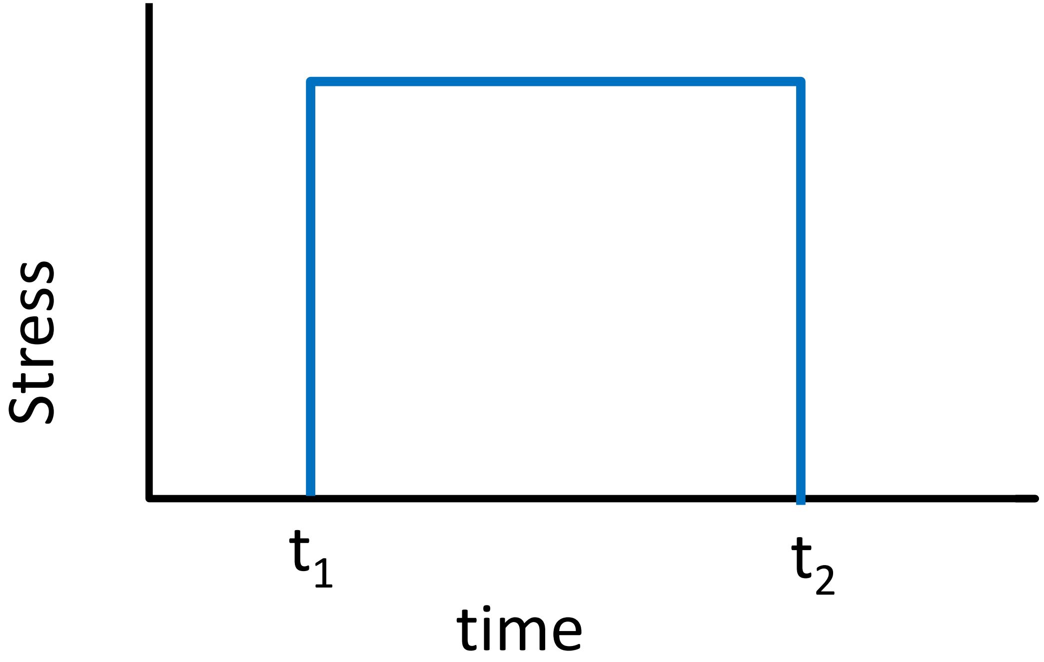
Available transient test modes

- Creep-Recovery
- Stress Relaxation
- Iso-strain Temperature Ramp
- Iso-force Temperature Ramp
- Stress-Strain Rate Tests

Creep recovery

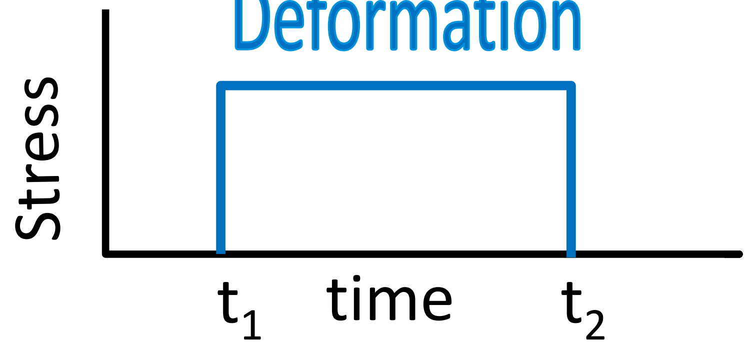
- A stress is applied to sample instantaneously at t_1 and held constant for a specific period of time. The strain is monitored as a function of time ($\gamma(t)$ or $\varepsilon(t)$).

- The stress is reduced to zero at t_2 and the strain is monitored as a function of time ($\gamma(t)$ or $\varepsilon(t)$).



Creep recovery

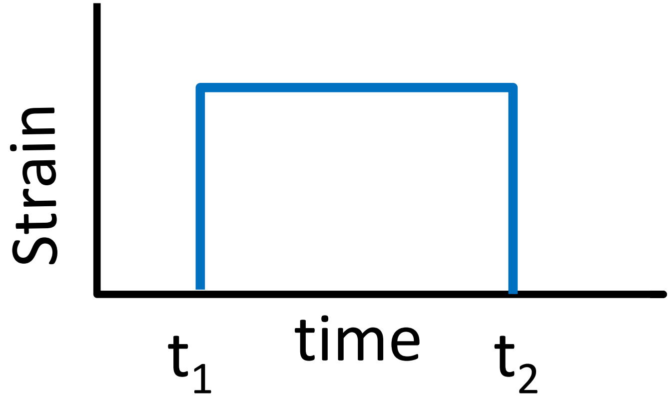
Deformation



Response of Classical Extremes

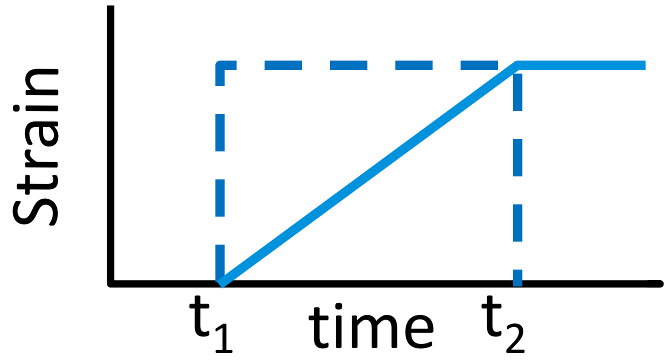
Elastic

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

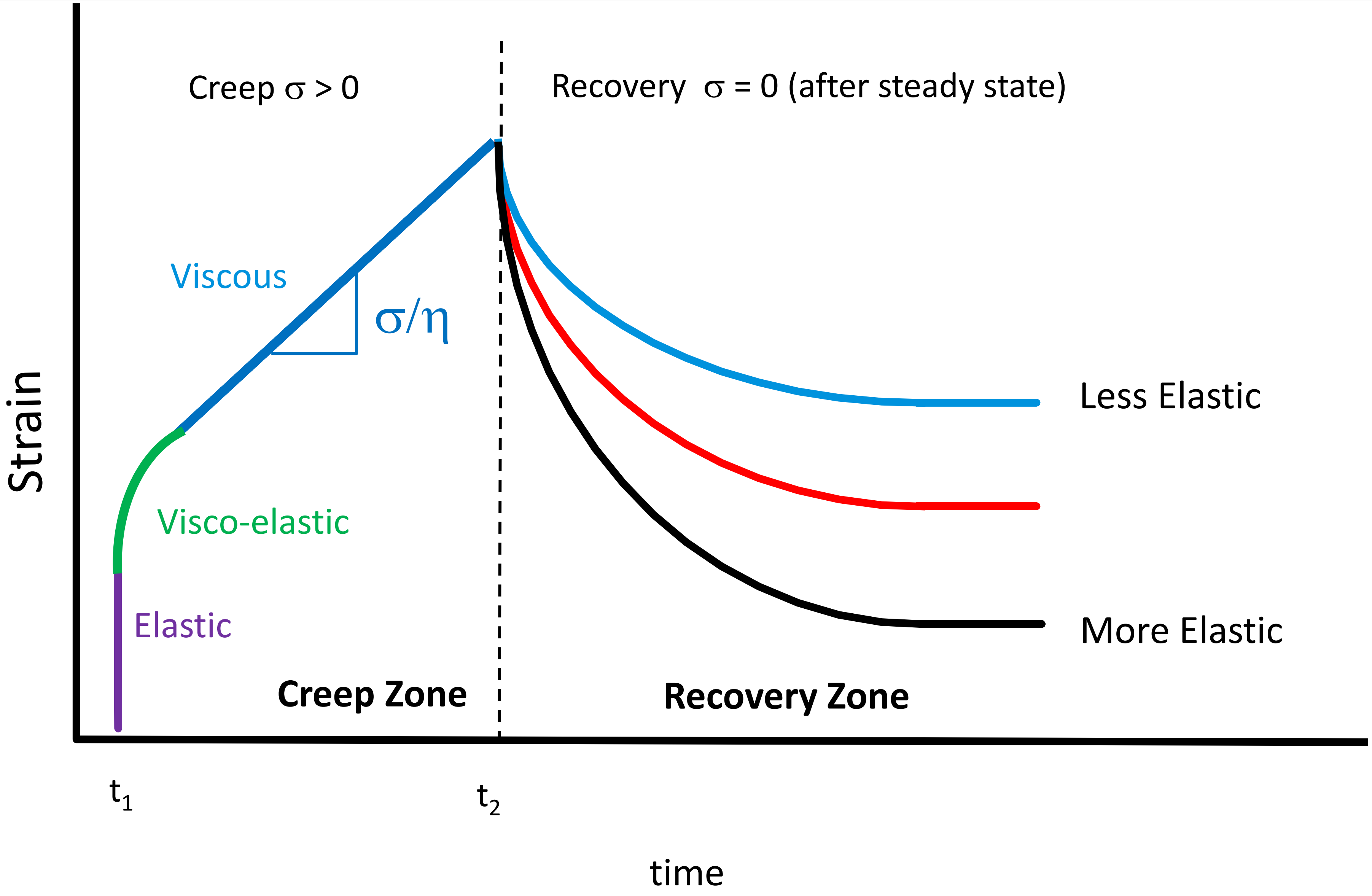


Viscous

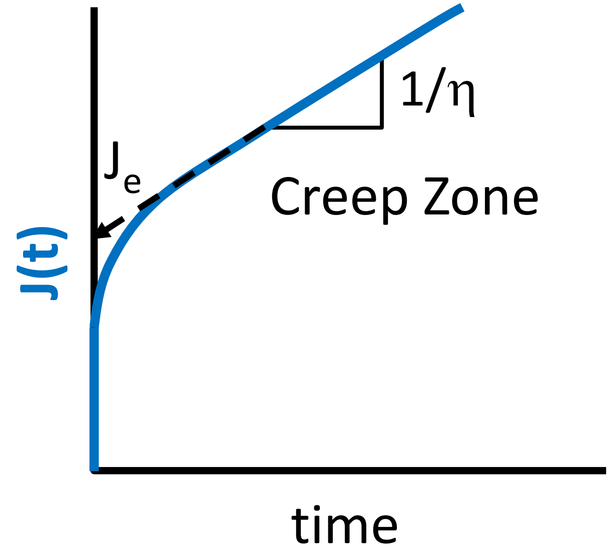
- Stain rate for t>t1 is constant
- Strain for t>t1 increase with time
- Strain rate for t >t2 is 0

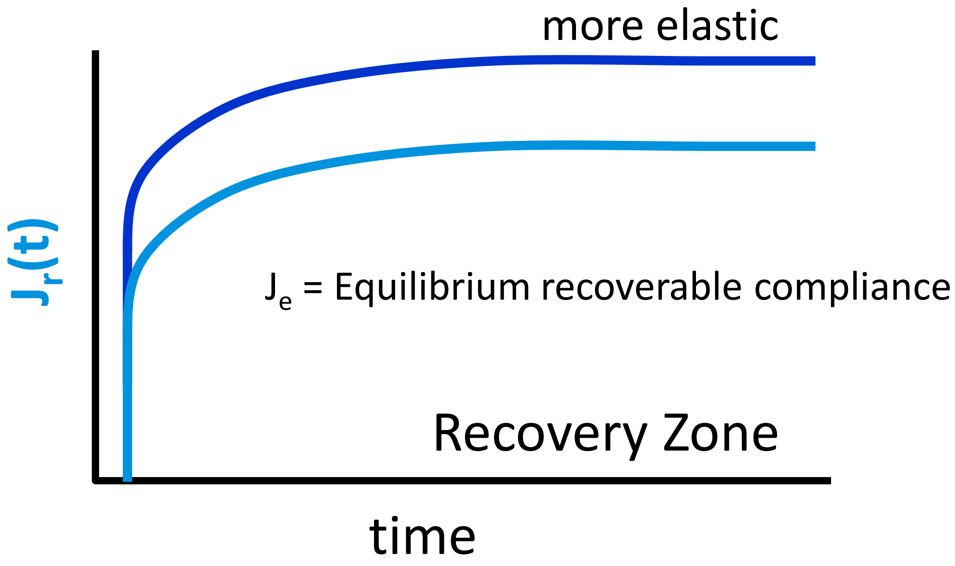


Creep recovery



Creep recovery : Creep and Recoverable Compliance





<u>Creep Compliance</u>

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

Creep experiments report the material property *Compliance* which is in a sense the inverse of Modulus

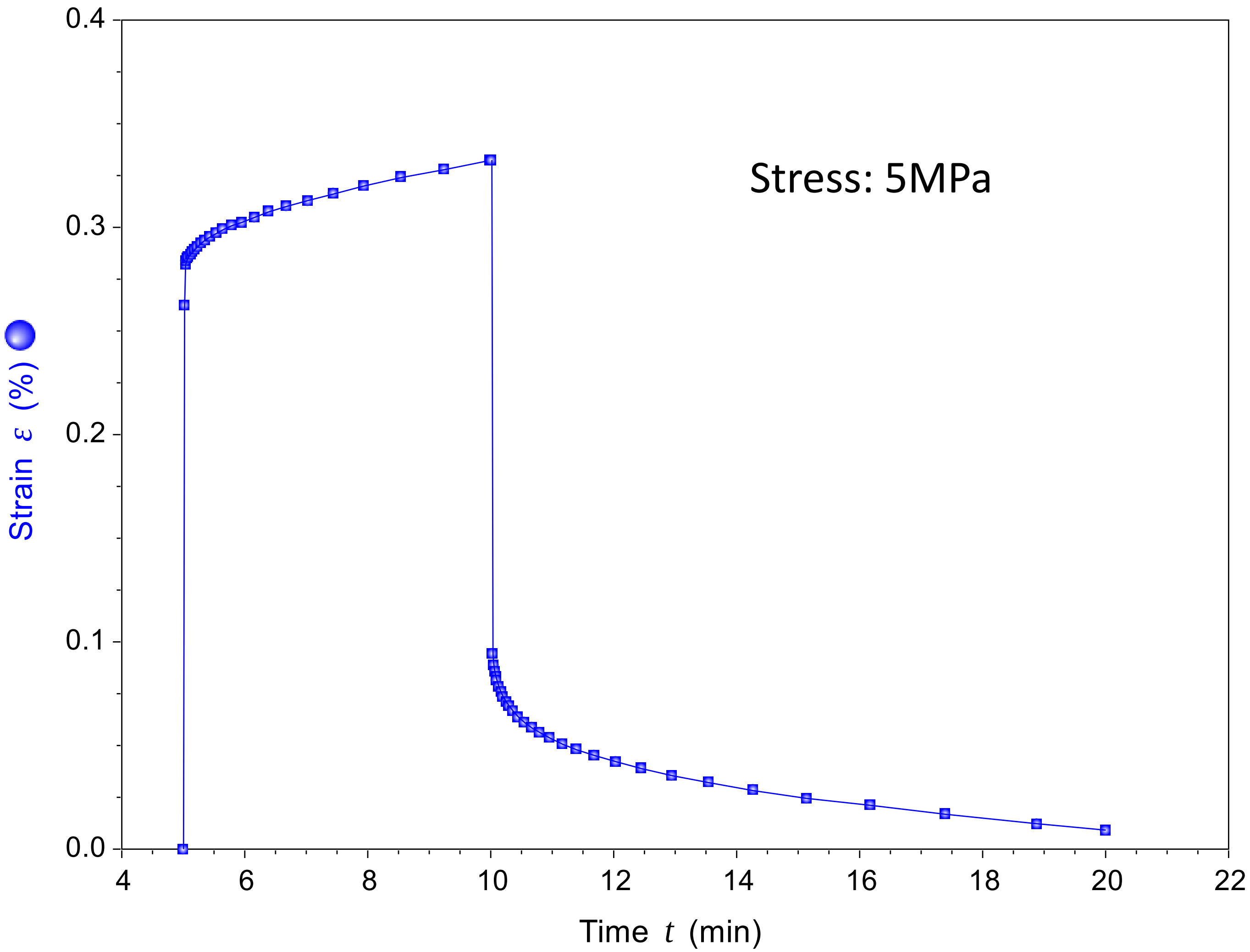
<u>Recoverable Compliance</u>

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

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

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

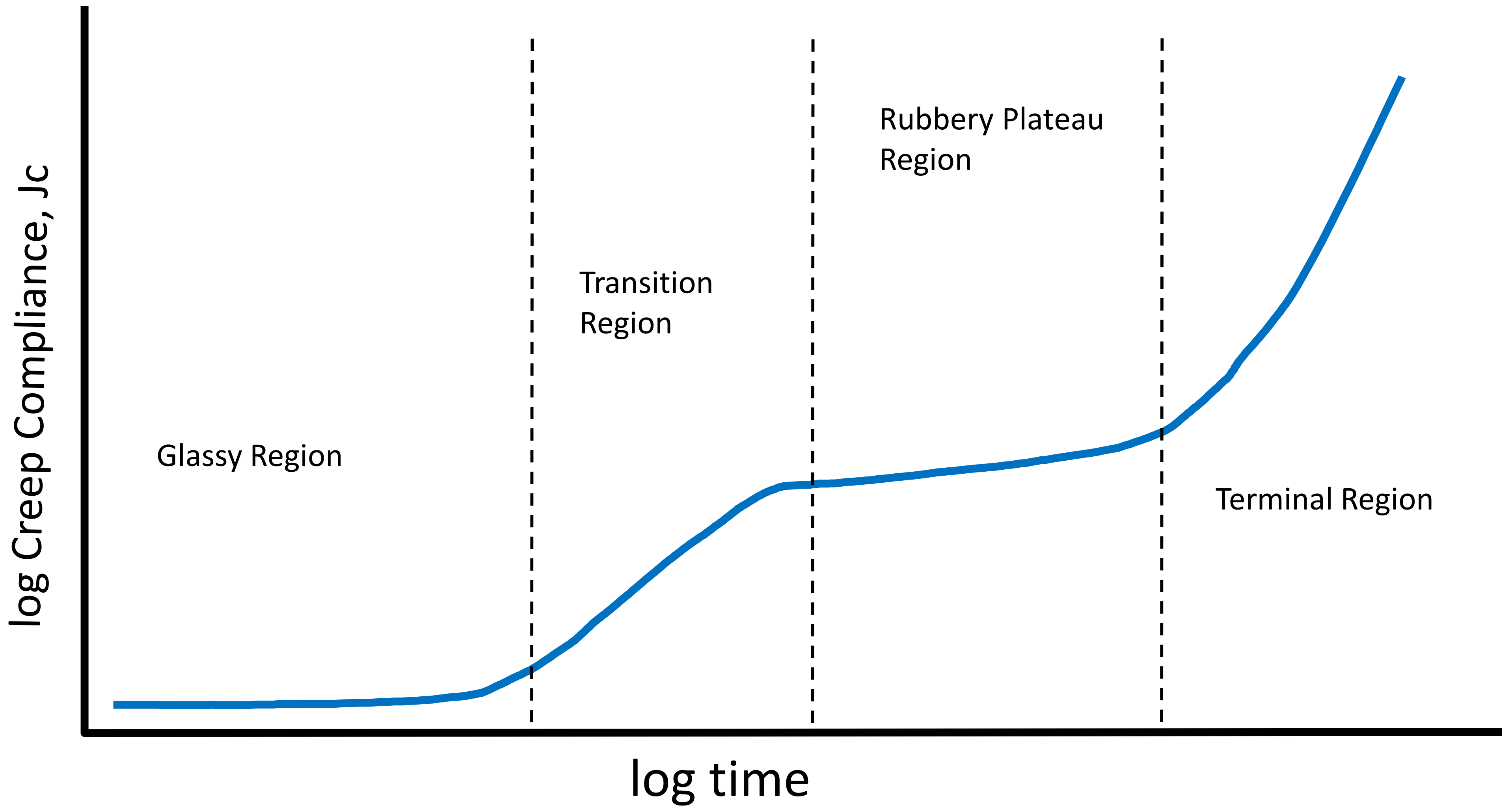
Creep-recovery test on PET Film



p/n: 984309.901

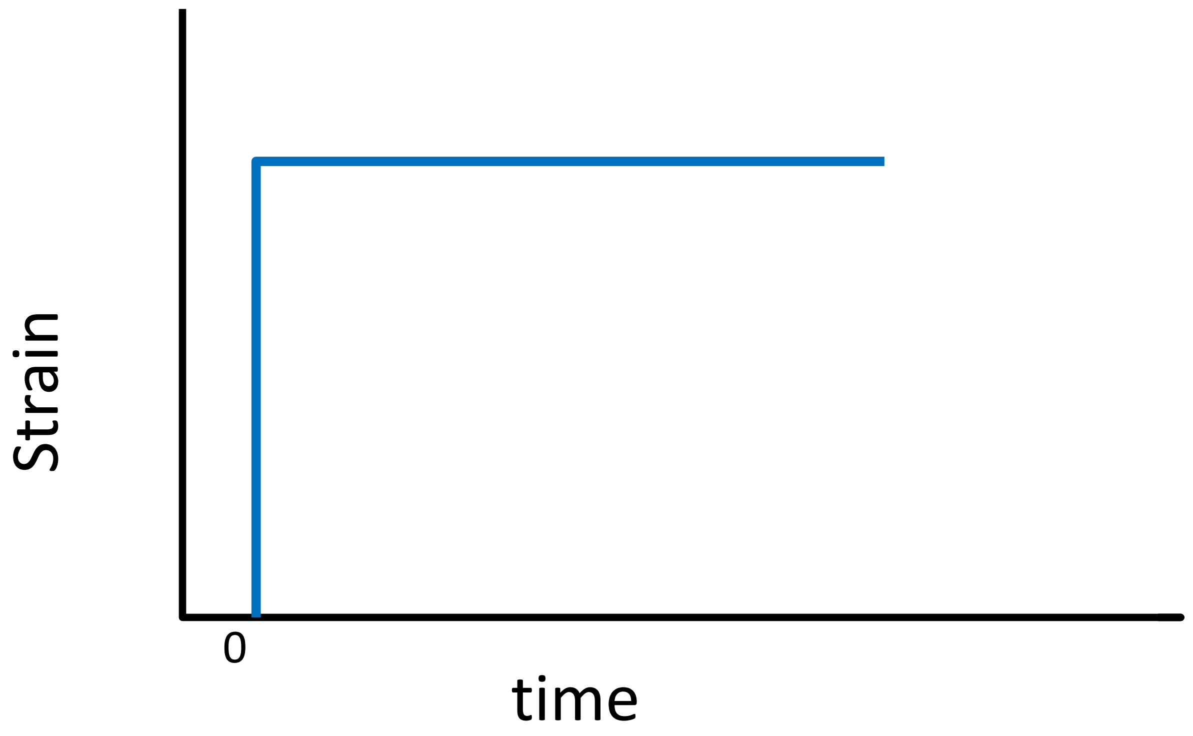
Instrument: Q800
Clamp: Tension
Temperature: 75°C
Stress: 5MPa

Creep: Material response

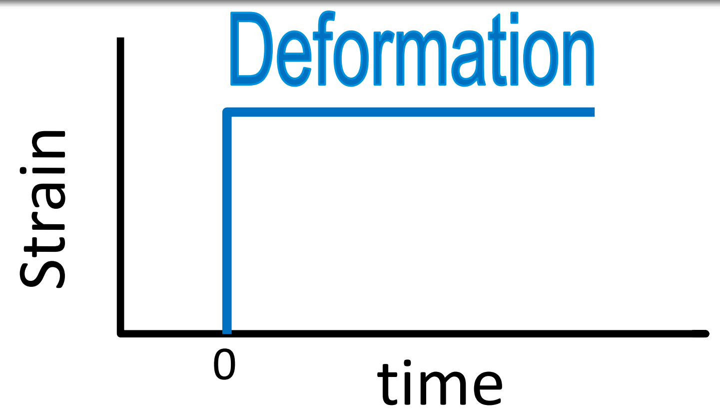


Stress relaxation experiment

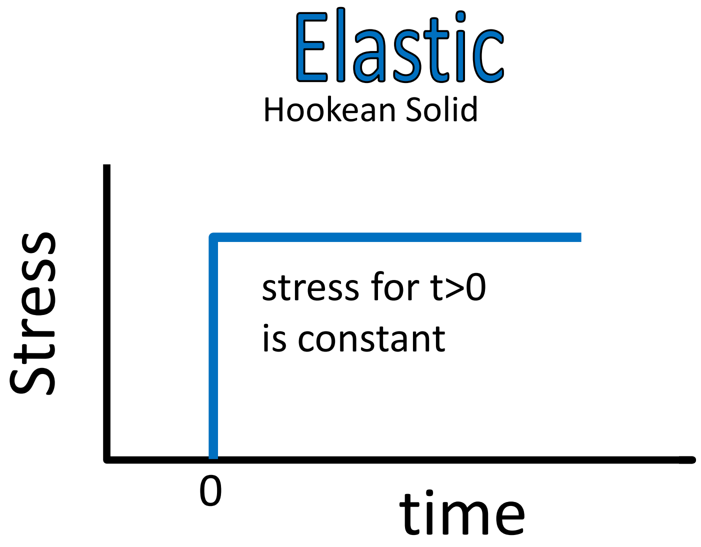
- Strain is applied to sample instantaneously (in principle) and held constant with time.
- Stress is monitored as a function of time $\sigma(t)$.

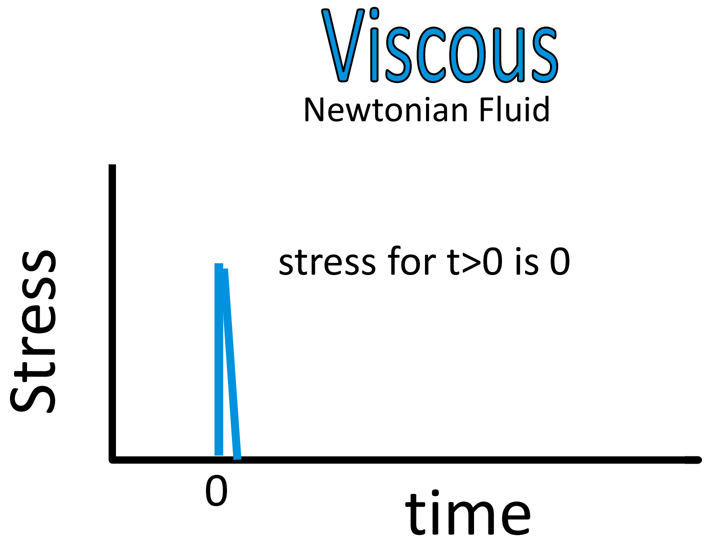


Stress relaxation experiment



Response of Classical Extremes

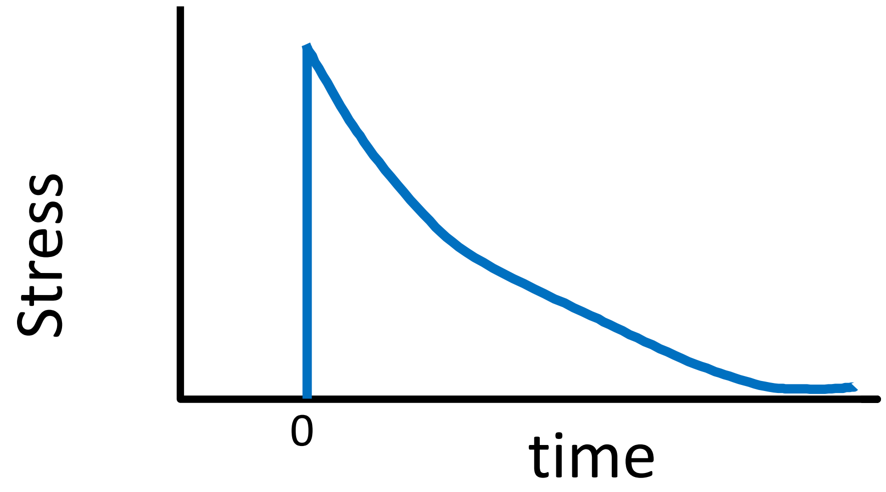




Stress relaxation experiment

Response of **ViscoElastic** Material

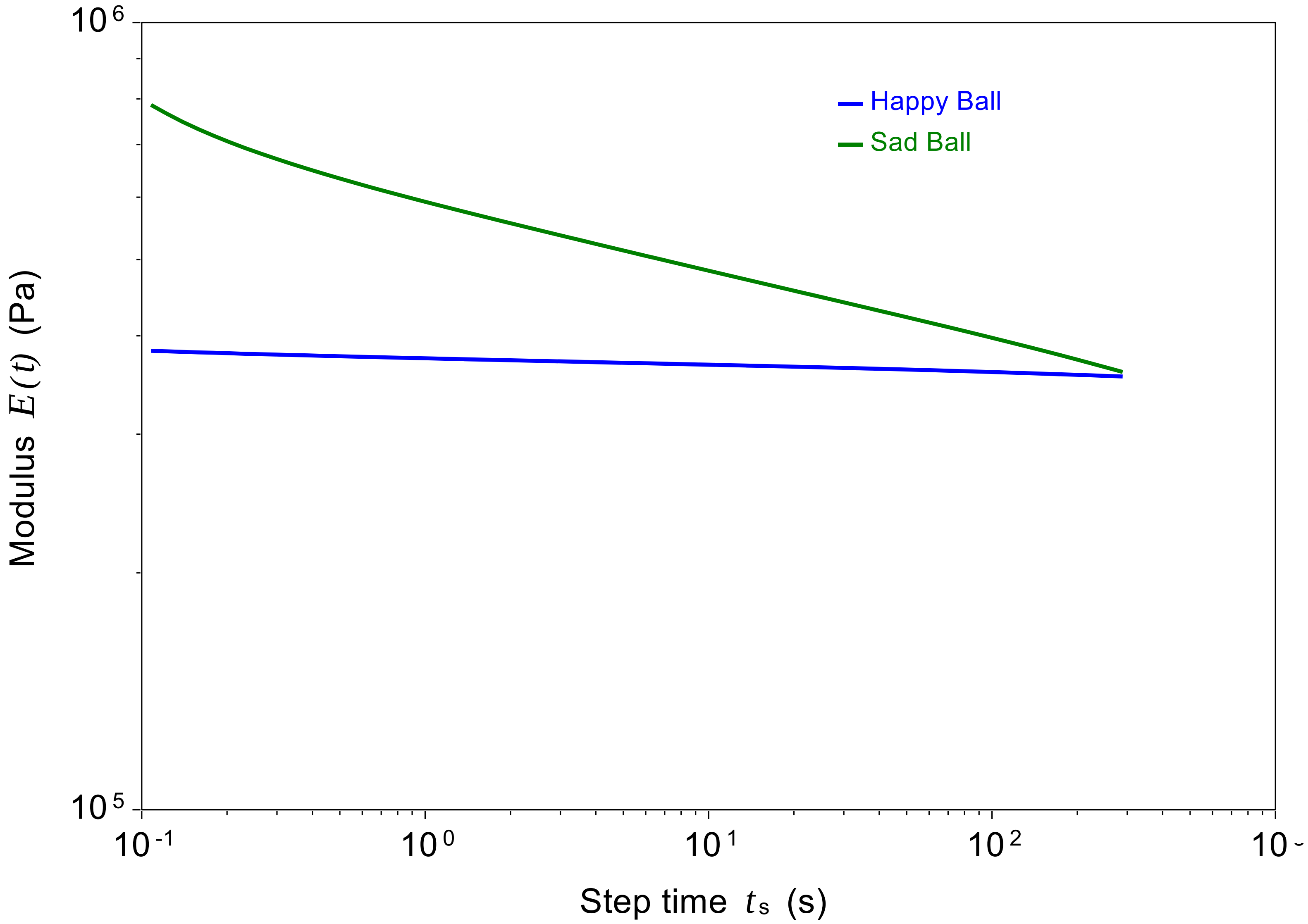
Stress decreases with time starting at some high value and decreasing to zero.



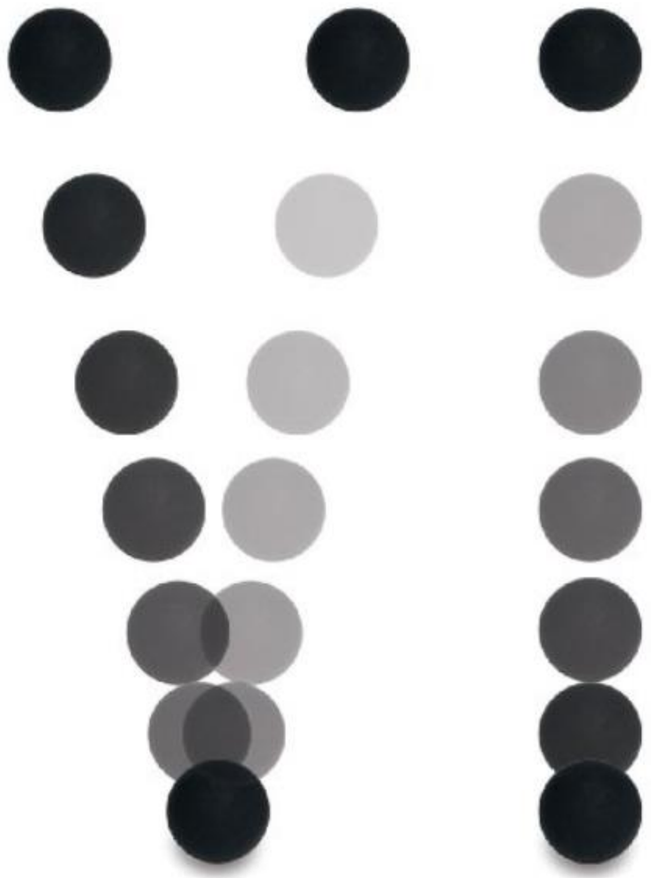
- For small deformations (strains within the linear region) the ratio of stress to strain is a function of time only.
- This function is a material property known as the STRESS RELAXATION MODULUS, E(t)

$$\mathbf{E(t) = \sigma(t)/\gamma}$$

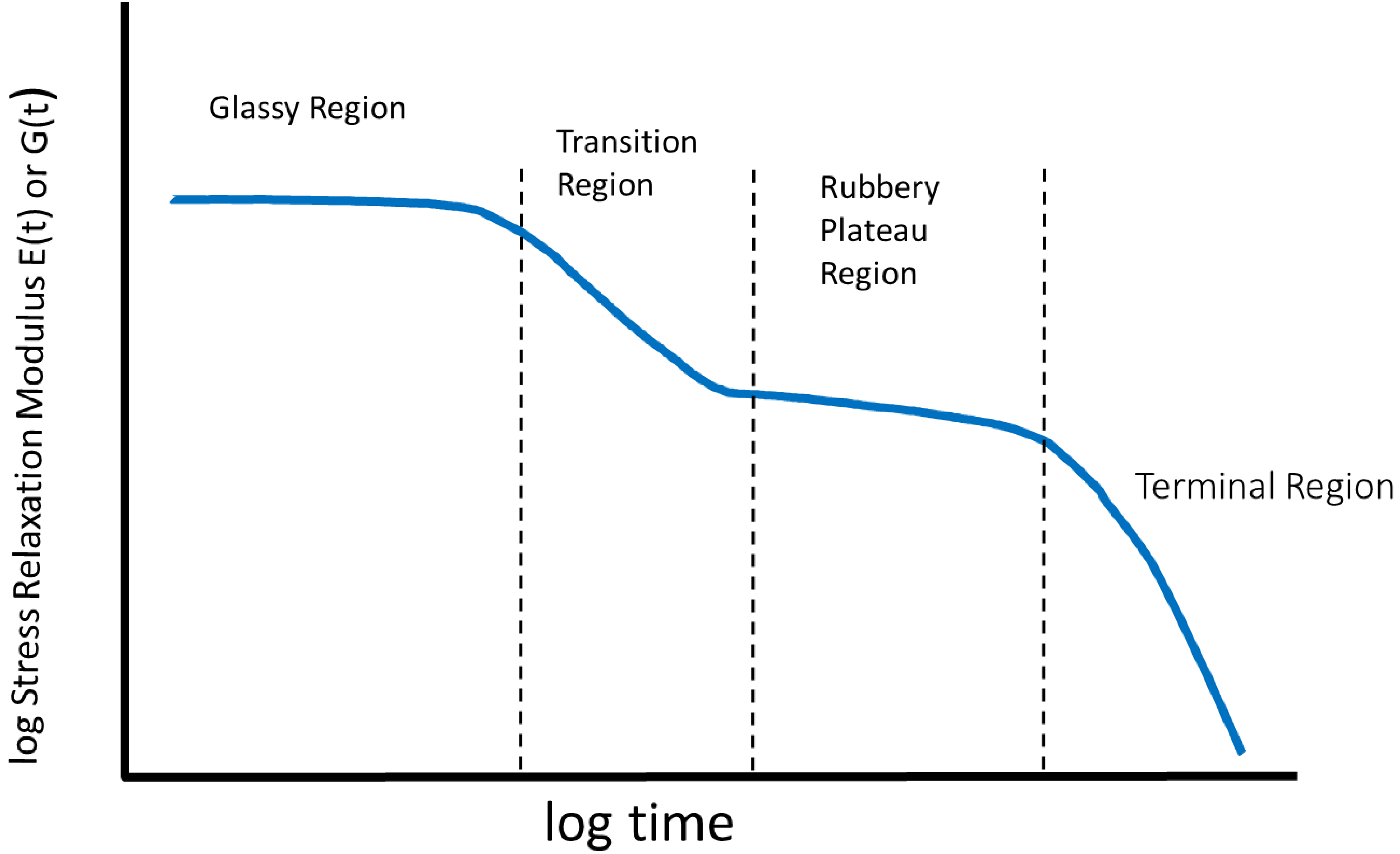
Stress Relaxation: Compression



HAPPY SAD

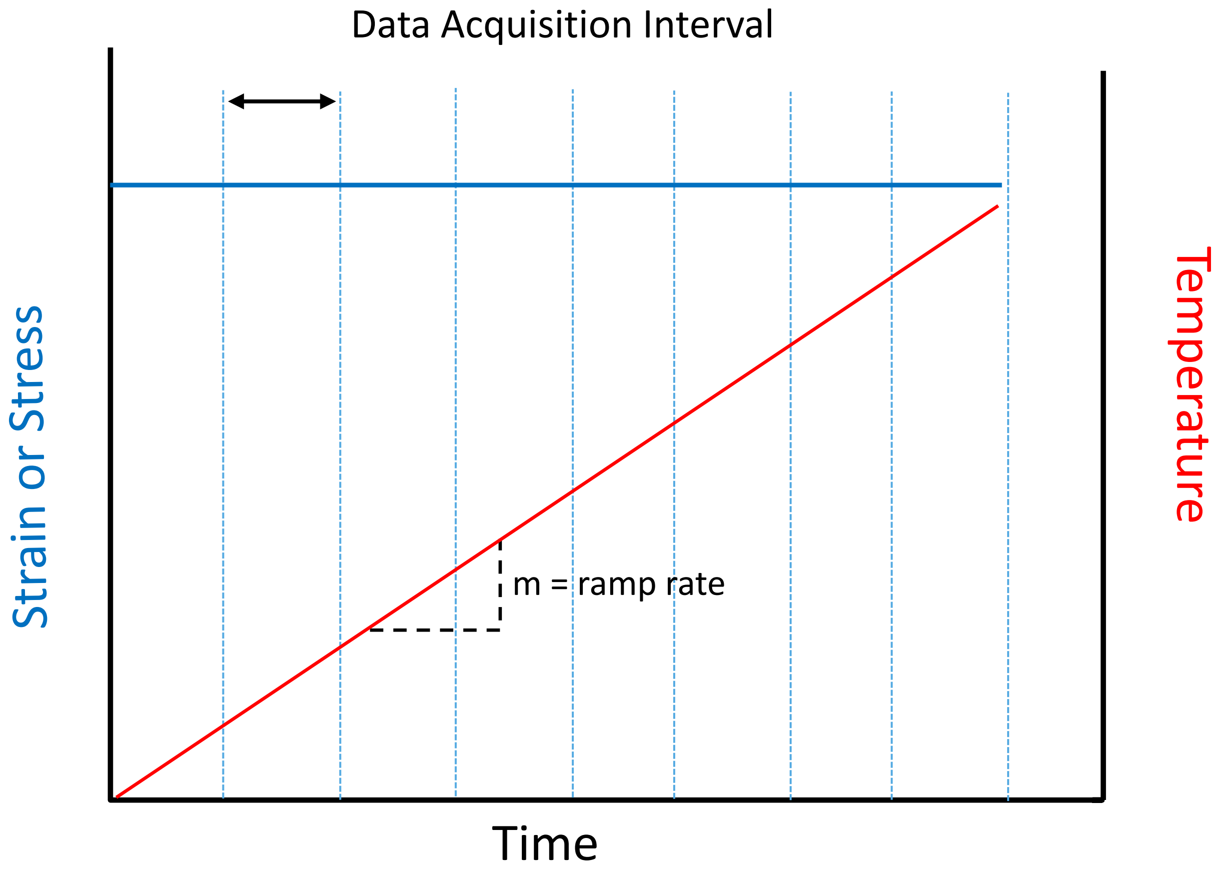


Stress Relaxation: Material response

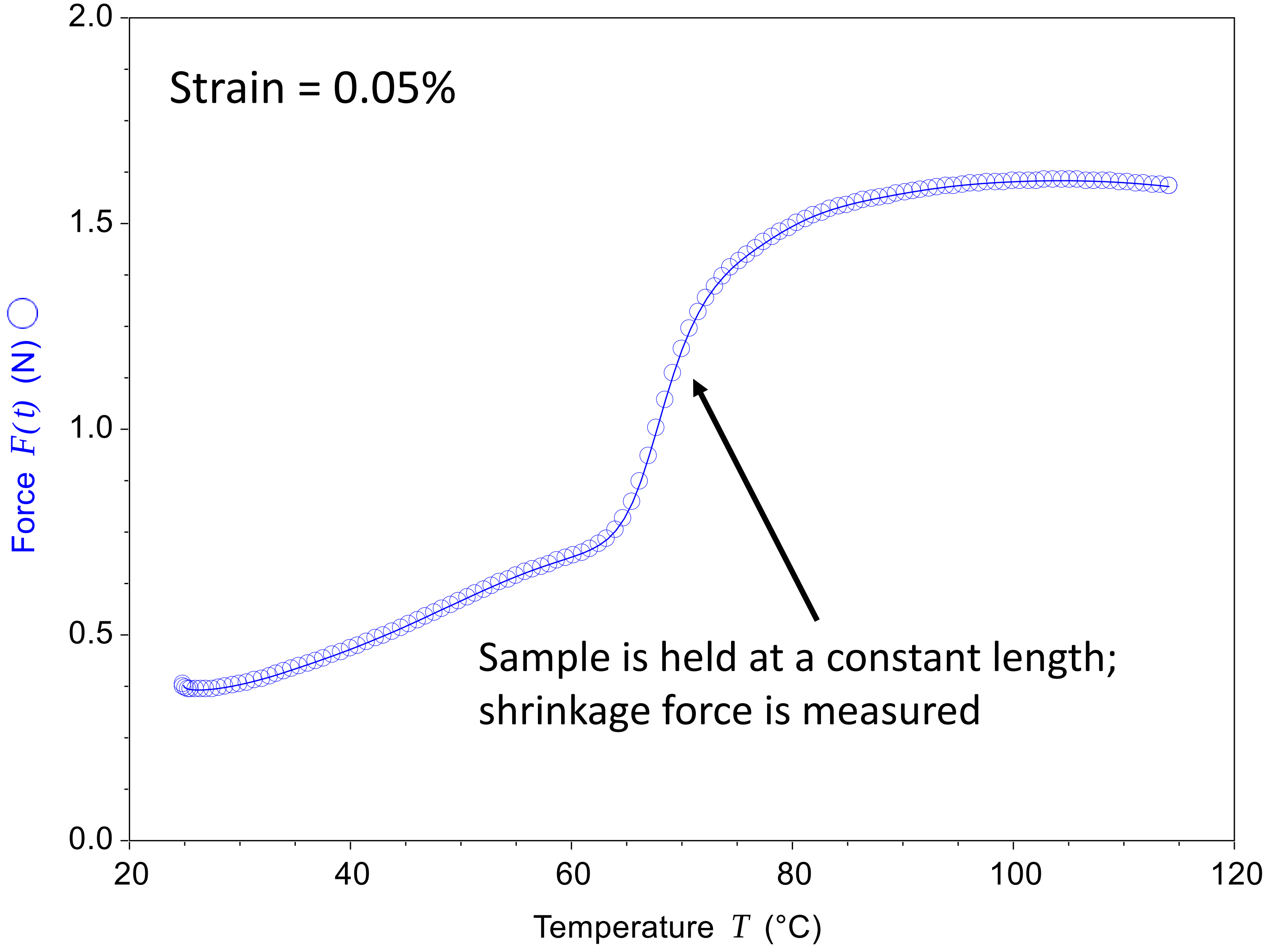


Iso-strain/Iso-stress Temperature Ramp

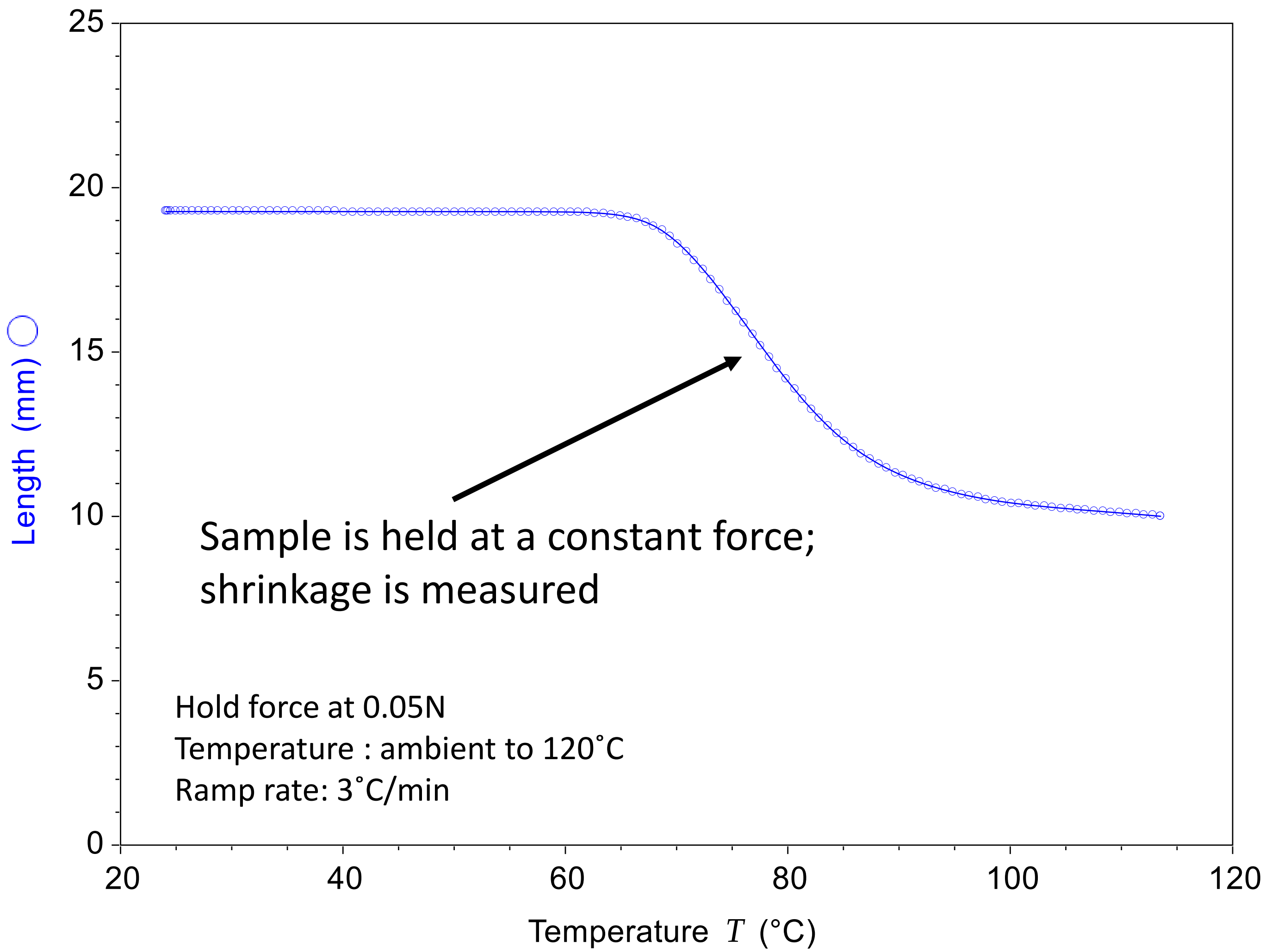
- The strain or stress is held at a constant value and a linear heating rate is applied.
- Valuable for assessing mechanical behavior under conditions of confined or fixed load (stress) or deformation (strain).
- Example: Measure sample shrinkage (length shrinkage or shrinking force)



Iso-Strain Temp Ramp: Measure Shrinking Force

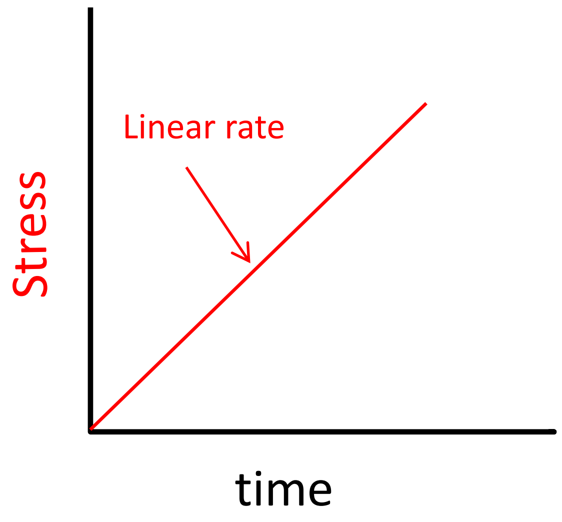


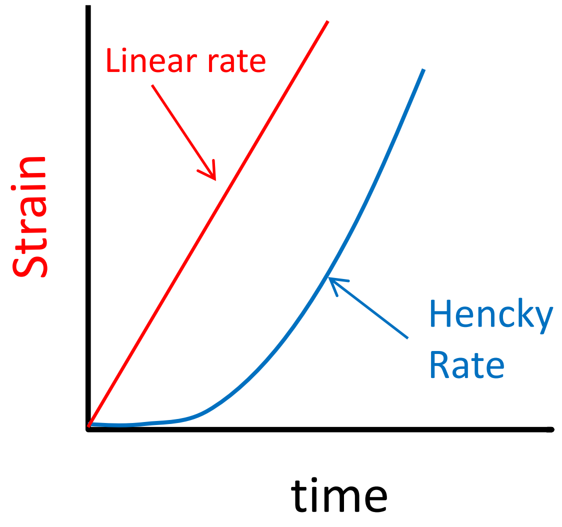
Iso-Force Temp Ramp: Measure Length Shrinkage



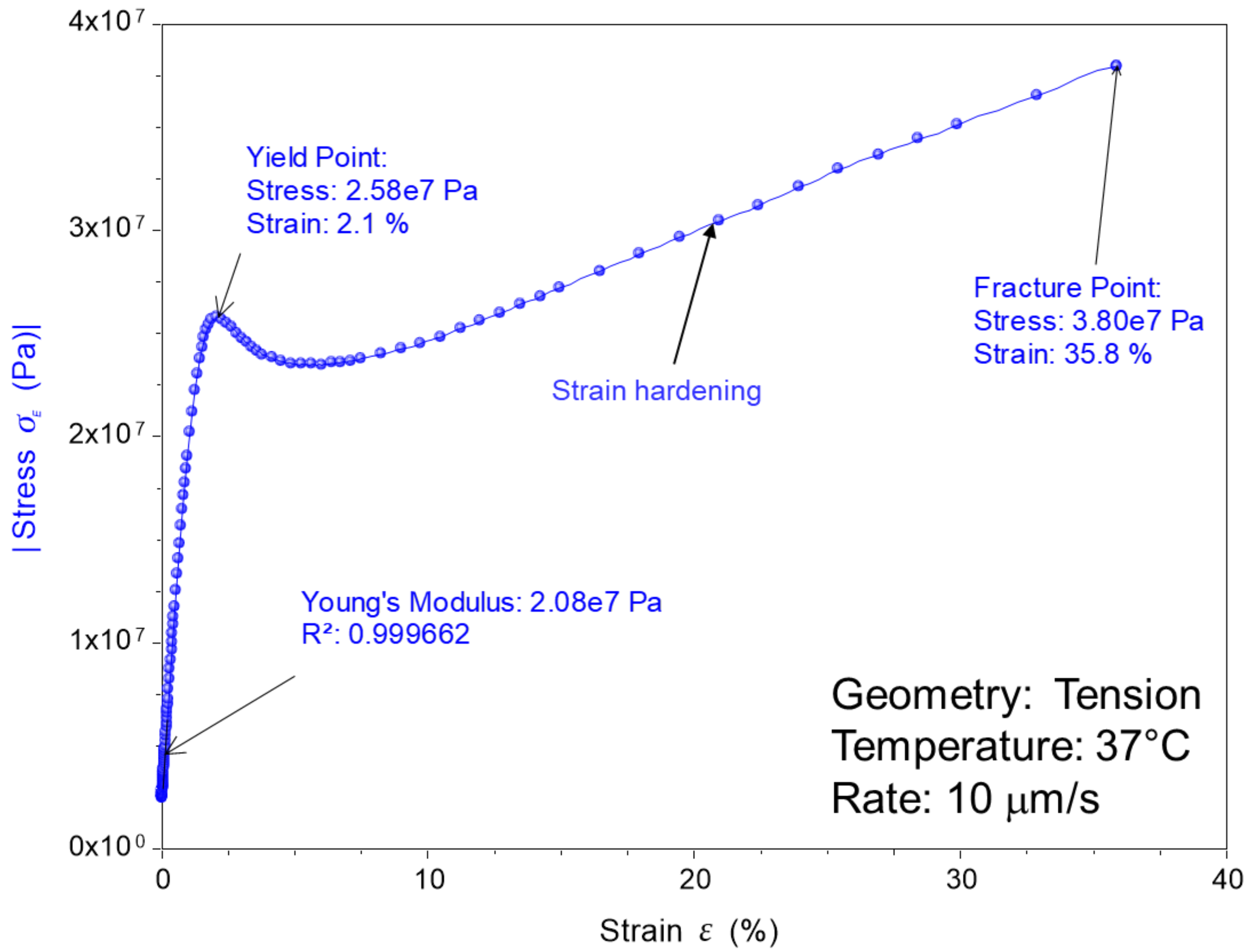
Stress-Strain Testing

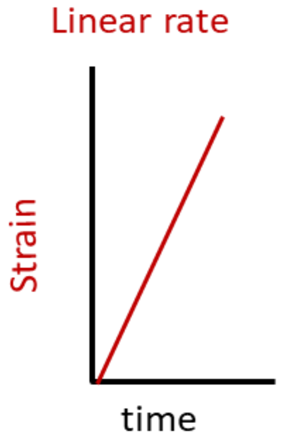
- Sample is deformed under a constant linear strain rate, Hencky strain rate, force, or stress for generating more traditional stress-strain curves.
- Measure sample's Young's modulus, yield stress, strain hardening effect and sample fracture





Polysaccharide Film Stress-Strain Test





Summary of Part 1

Summary

- Introduction to Dynamic Mechanical Analysis Importance of mechanical analysis
  - Conventional (non-oscillatory) vs dynamic (oscillatory) mechanical analysis
  - Viscoelasticity
    - Definition and physical significance of viscoelastic parameters
- DMA Instrumentation and Clamps
  - DMAs offered by TA Instruments
    - Discovery DMA 850
    - RSA-G2
    - TA Electroforce series
  - Common clamp configurations
    - Use of stiffness as a guide to choose the appropriate clamp

Summary: DMA experiments

- Significance of pre-load force and force track
- Dynamic tests
    - Strain/amplitude sweep
    - Time sweep
    - Frequency sweep
    - Temperature ramp
    - Temperature sweep
- Transient tests
    - Creep-recovery
    - Stress-relaxation
    - Iso-strain temperature ramp
    - Iso-stress temperature ramp
    - Stress-strain tests

End of Part 1

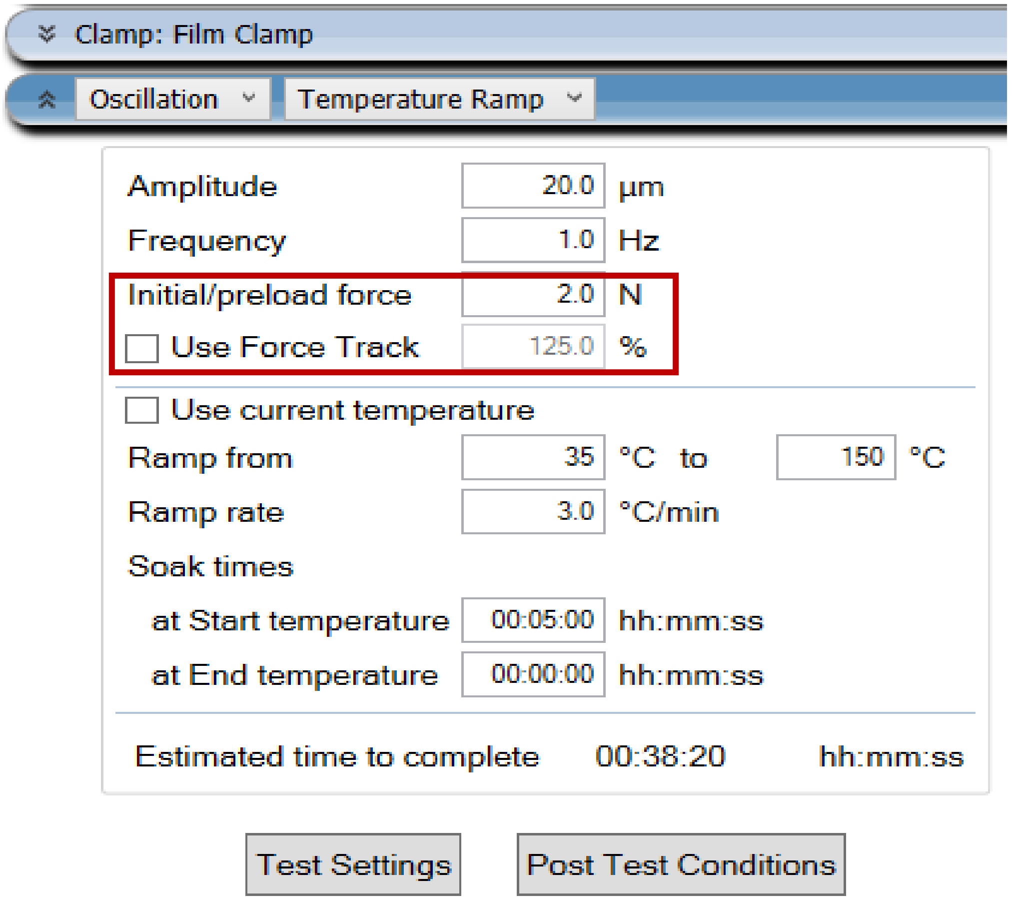
See appendix in the subsequent slides for additional experimental setup details

Appendix
Instrument control software screenshots

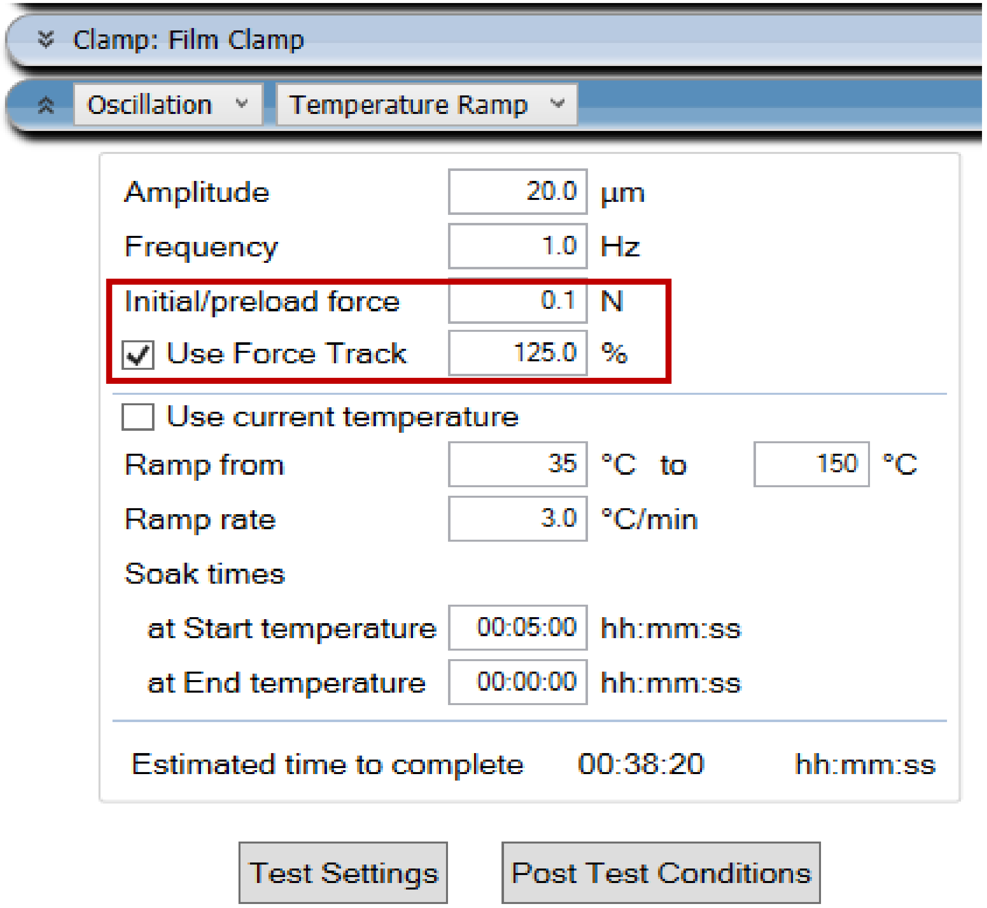
Dynamic/oscillation experiments

Offset force and force track on DMA850

- Constant static force

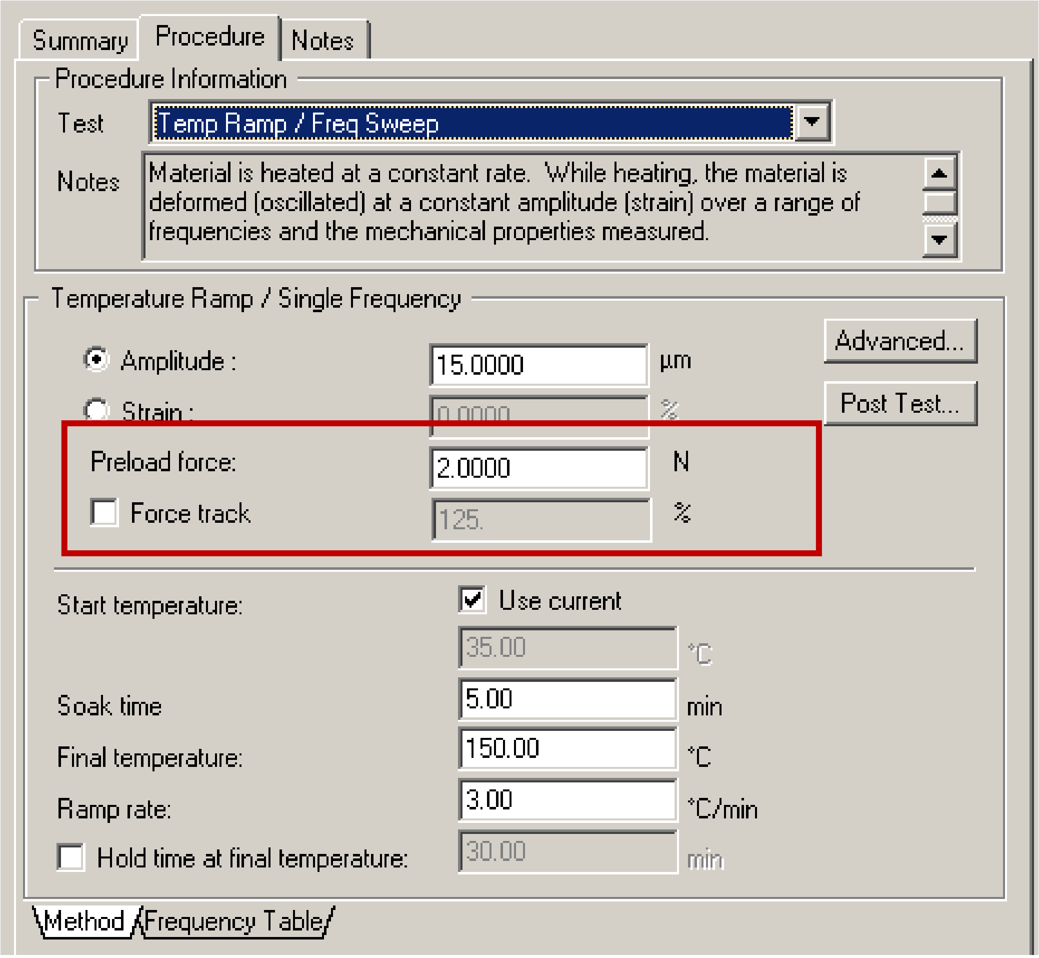


- Force track

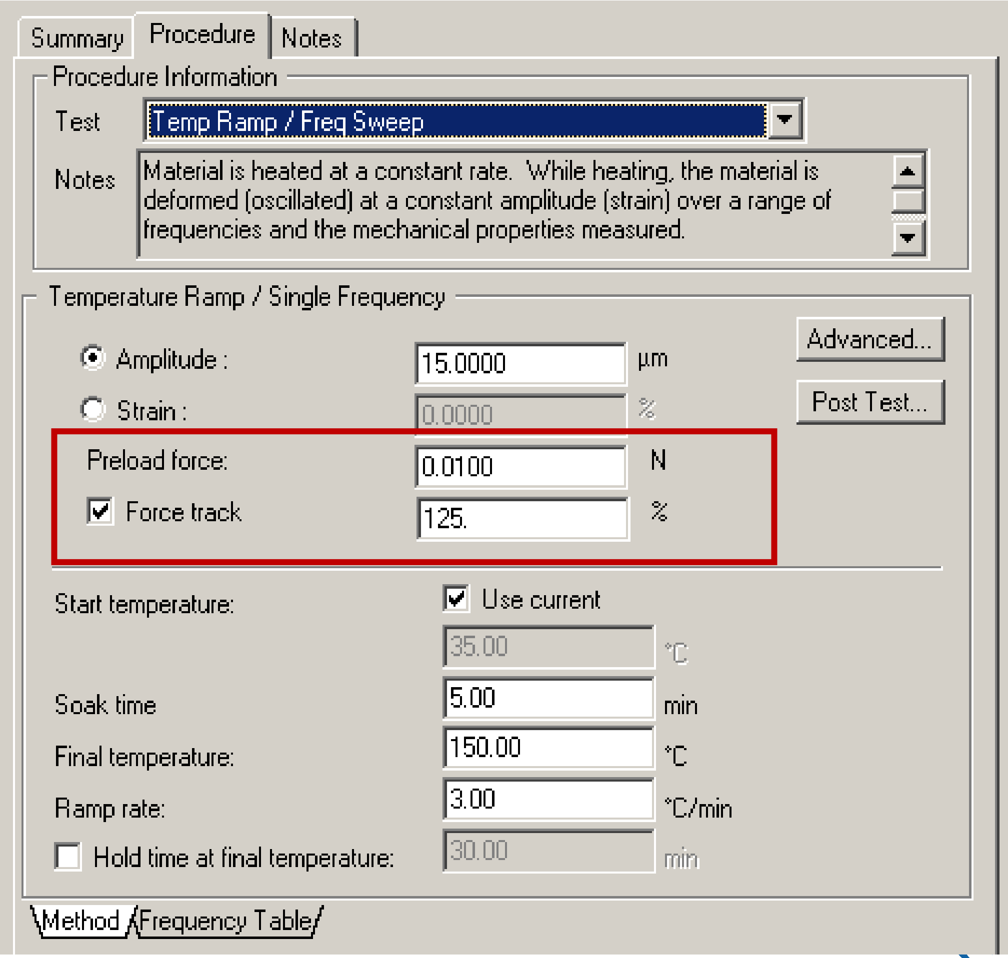


Offset force and force track on Q800

- Constant static force

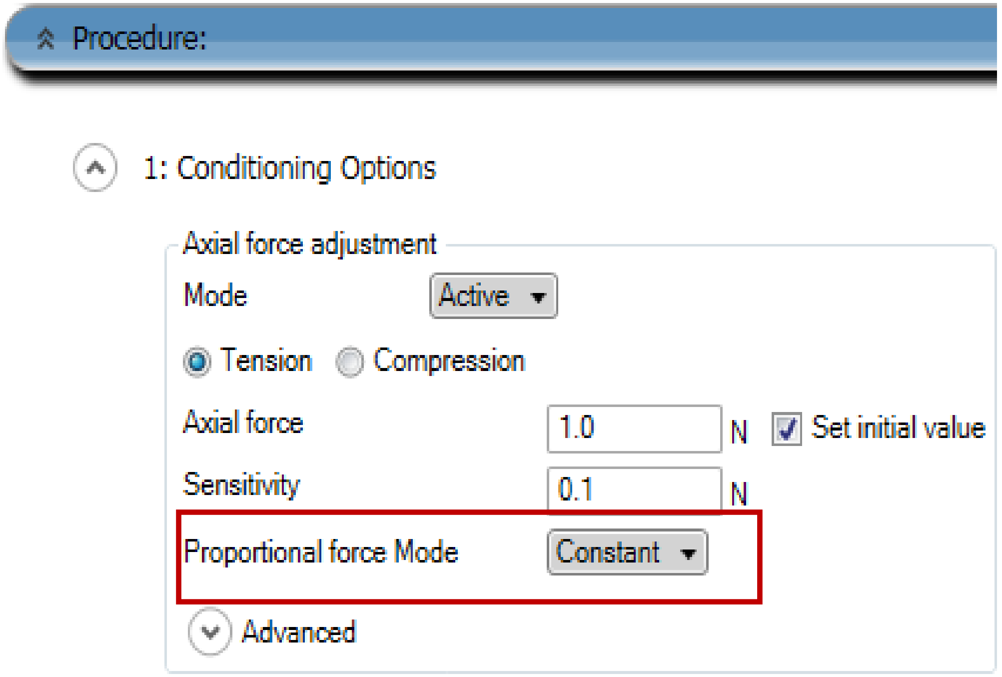


- Force track

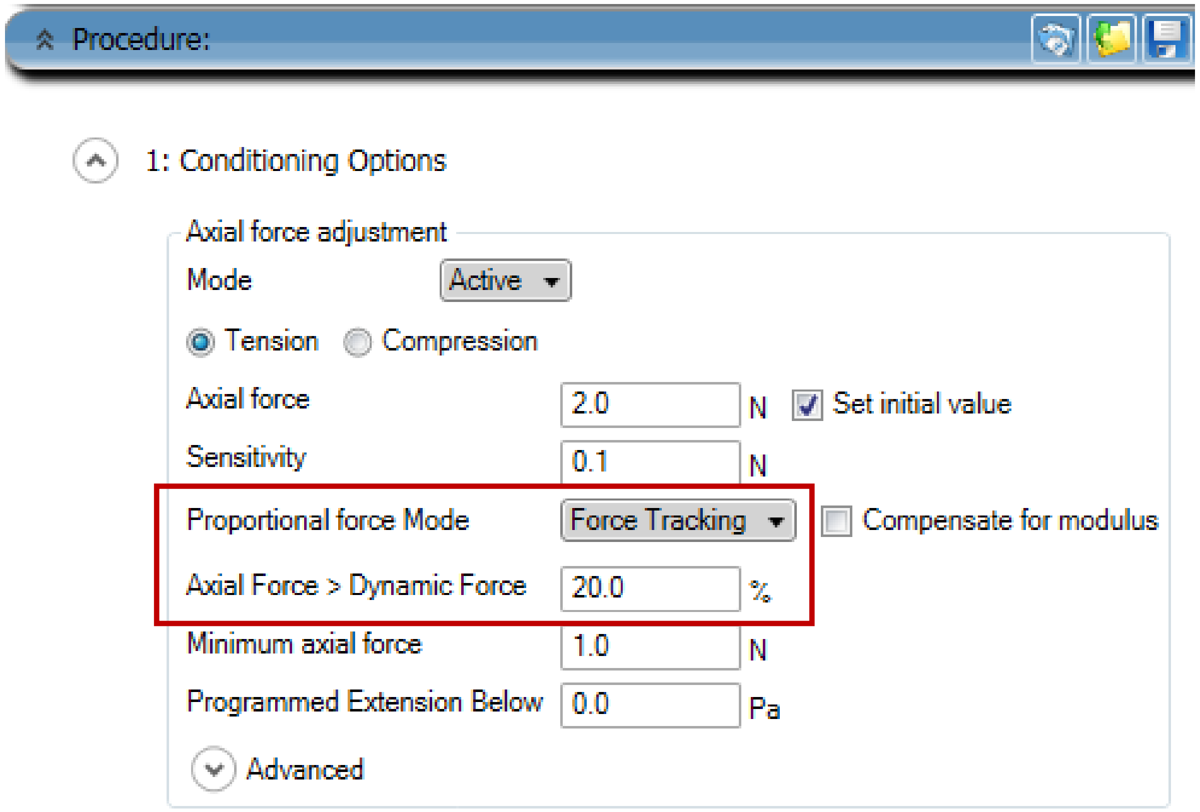


Offset force and force track on RSA G2

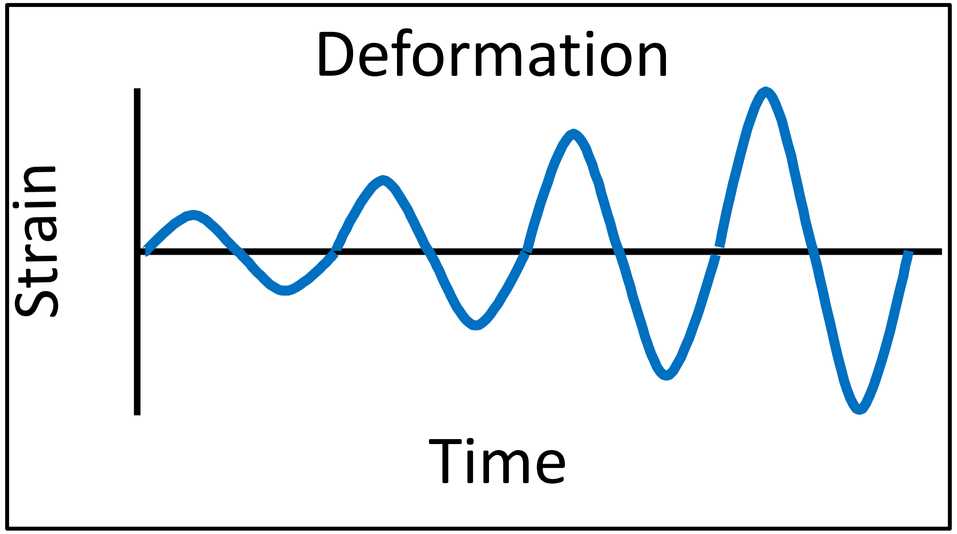
- Constant static force



- Force track



Dynamic Strain (Stress) Sweep

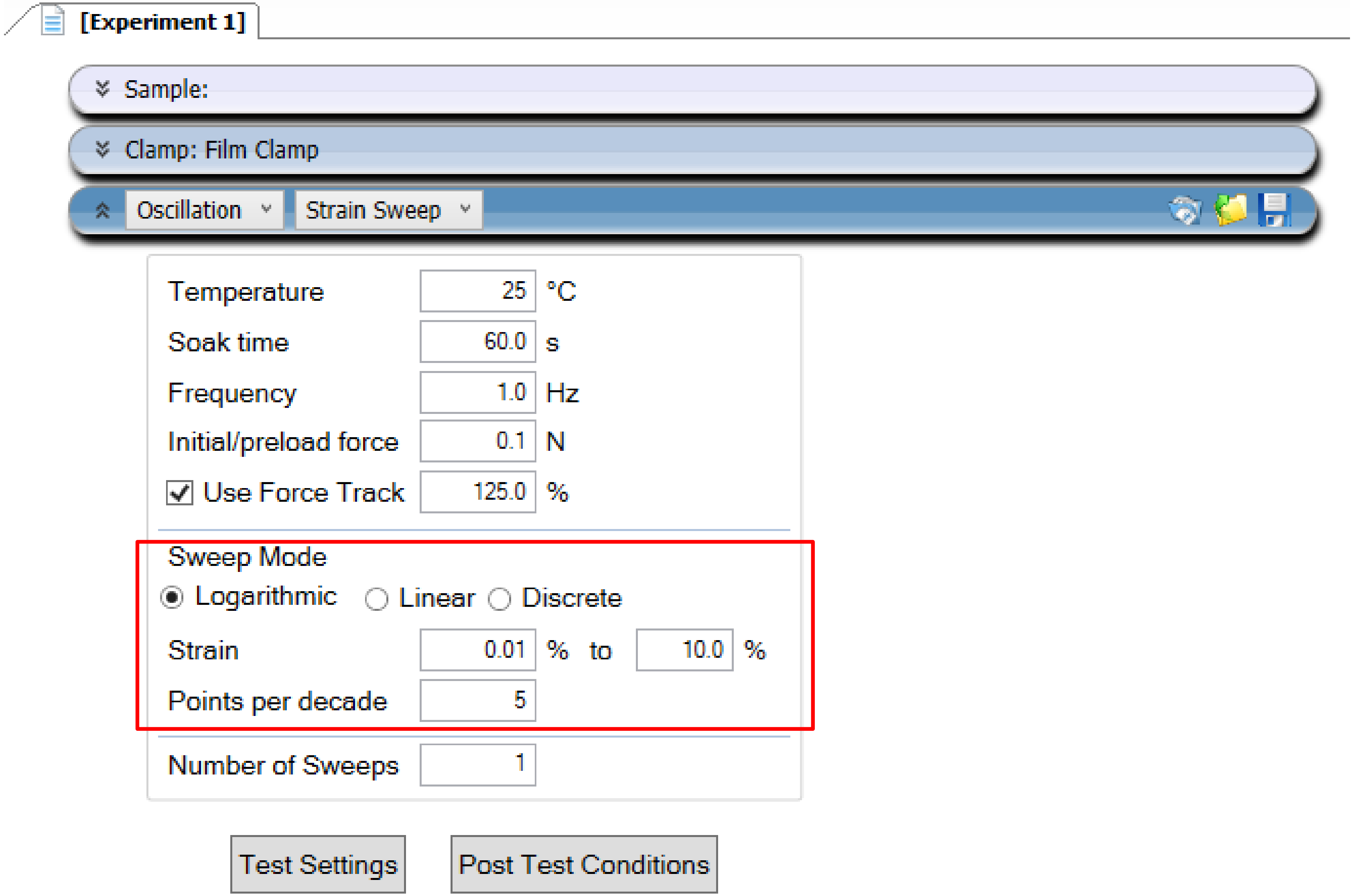


The material response to increasing deformation amplitude is monitored at a constant frequency and temperature.

USES

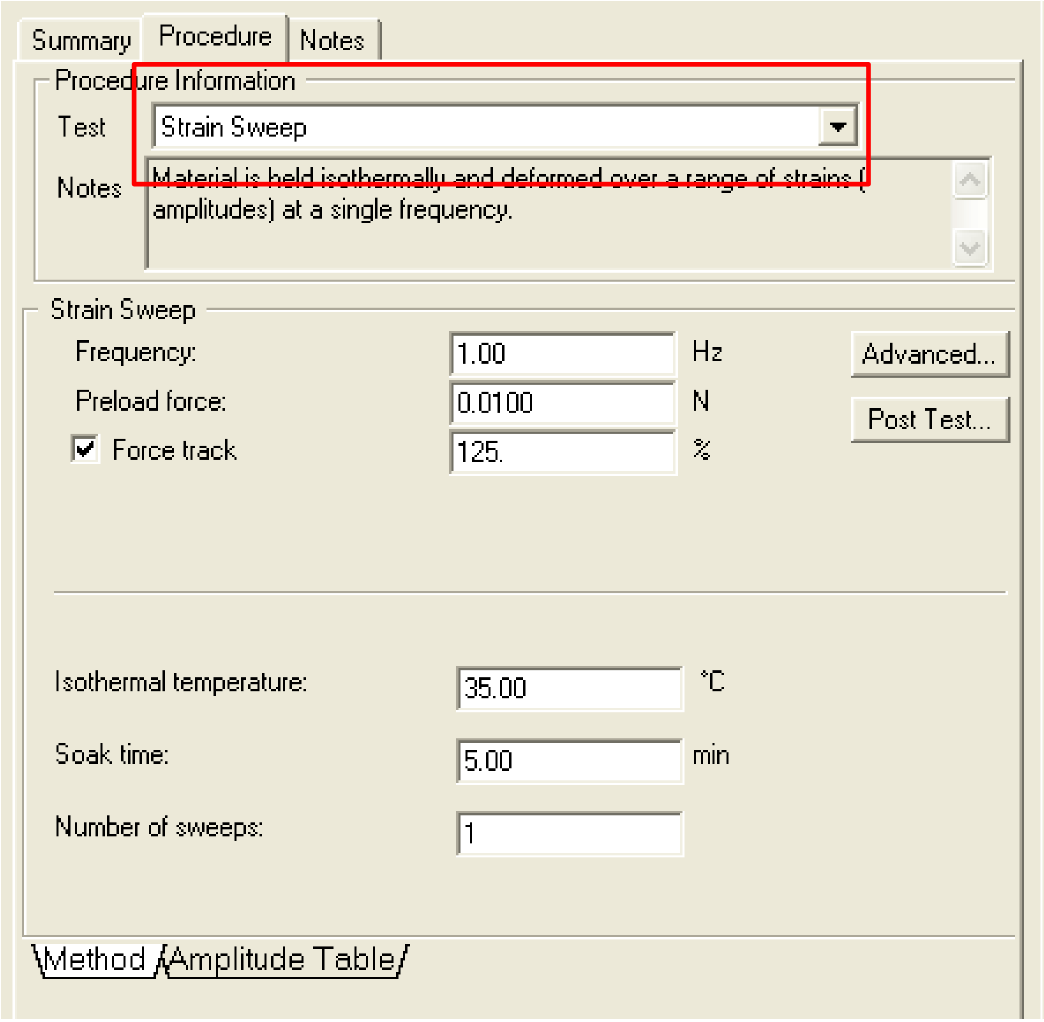
- Identify Linear Viscoelastic Region
- Resilience/elasticity

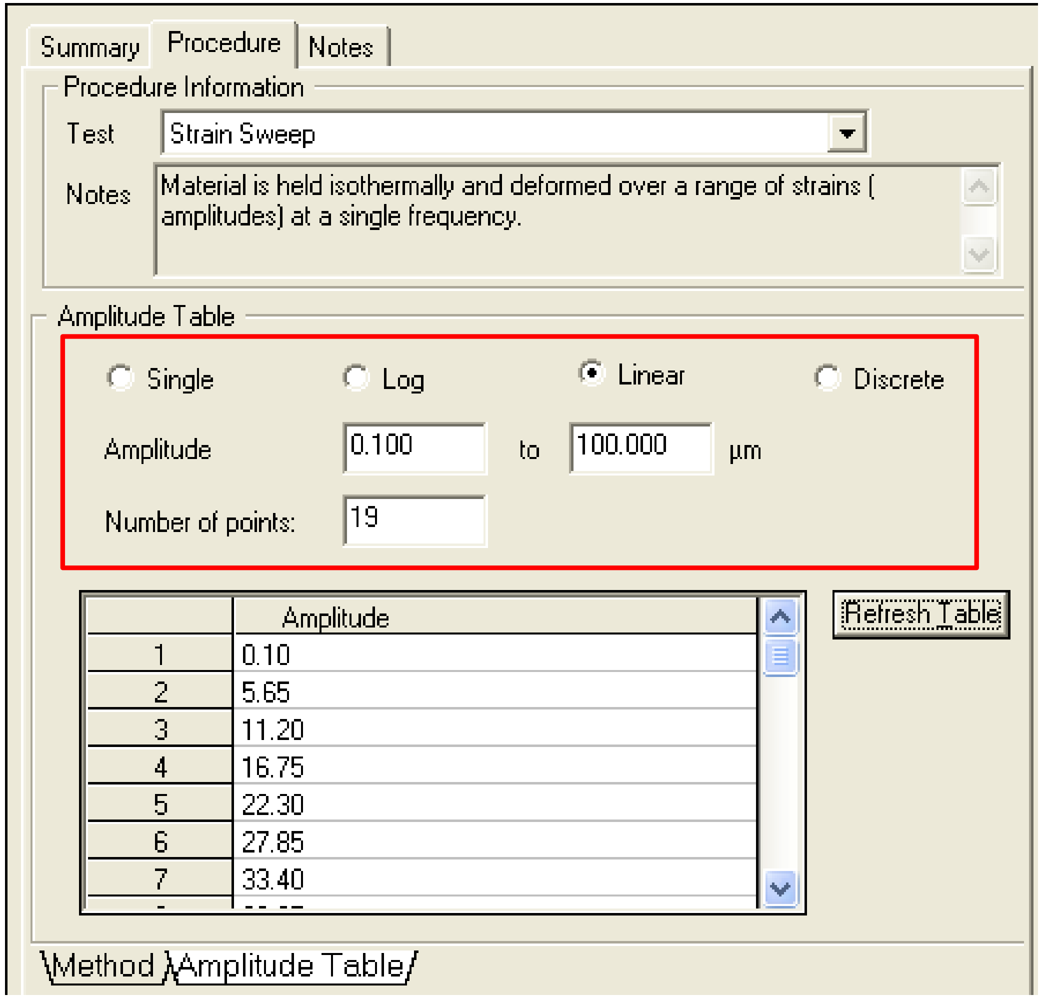
Programming Strain Sweep on DMA850



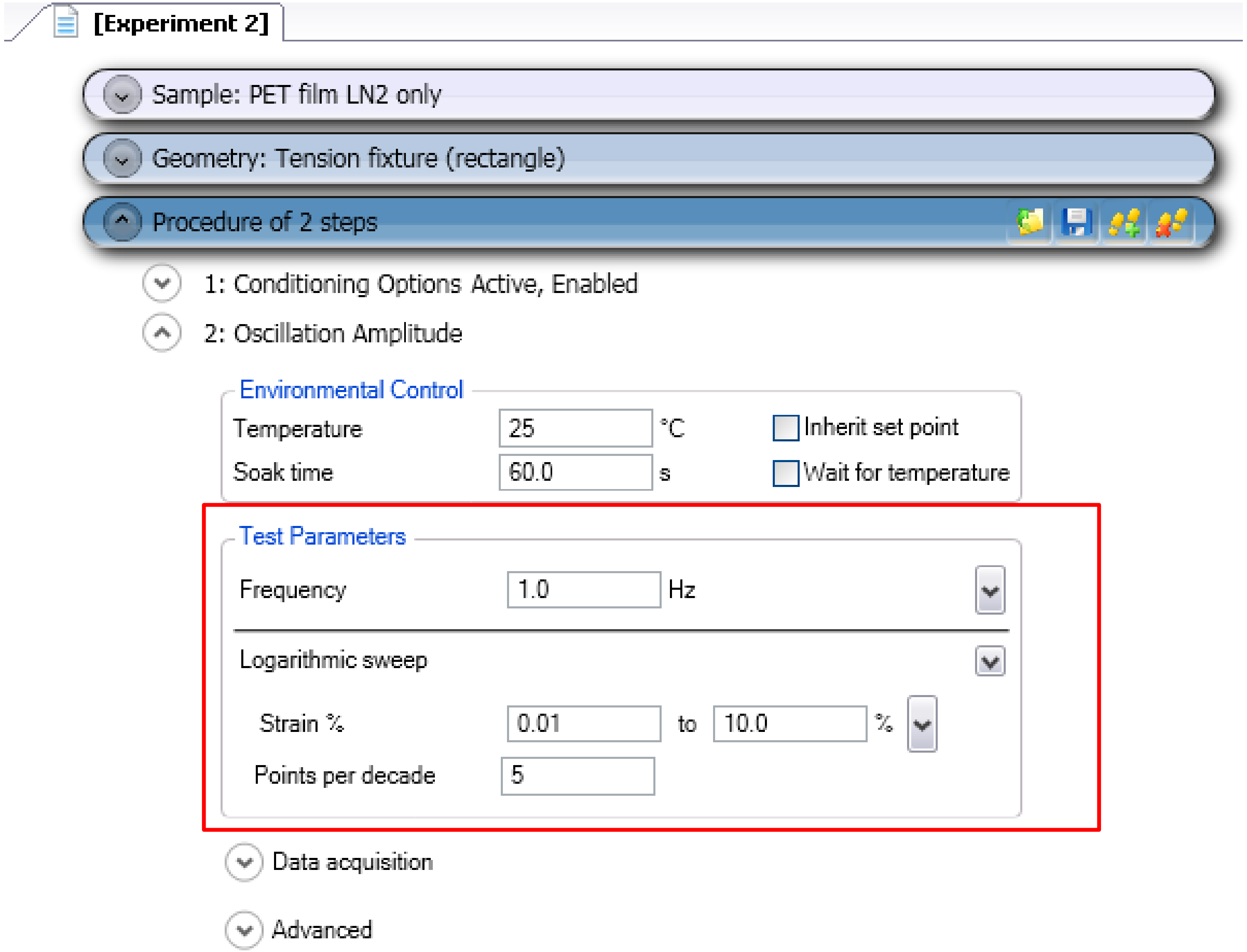
Programming Strain Sweep on Q800

Mode: DMA Multi-Strain

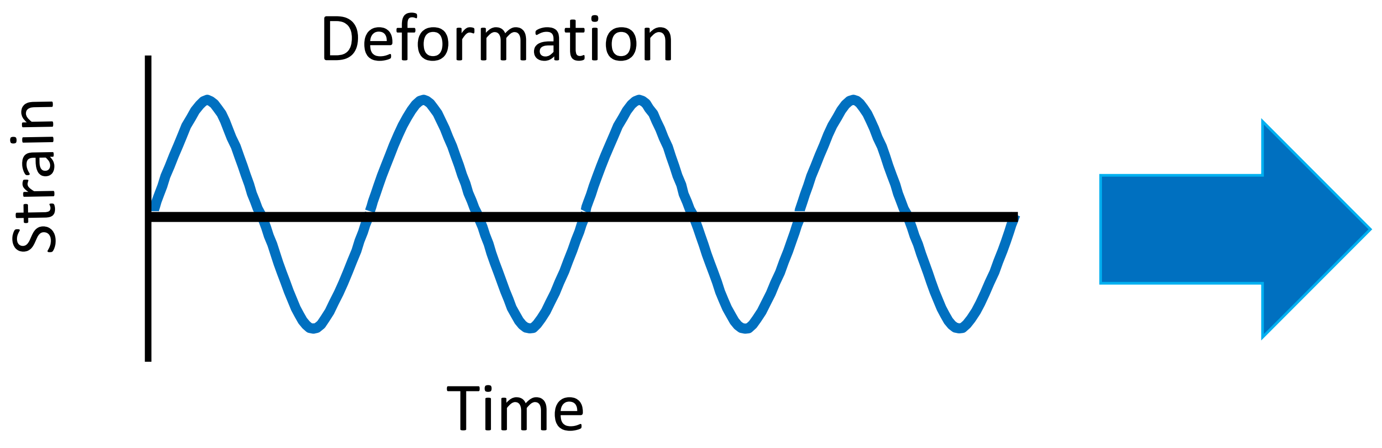




Programming Strain Sweep on RSA G2



Dynamic Time Sweep



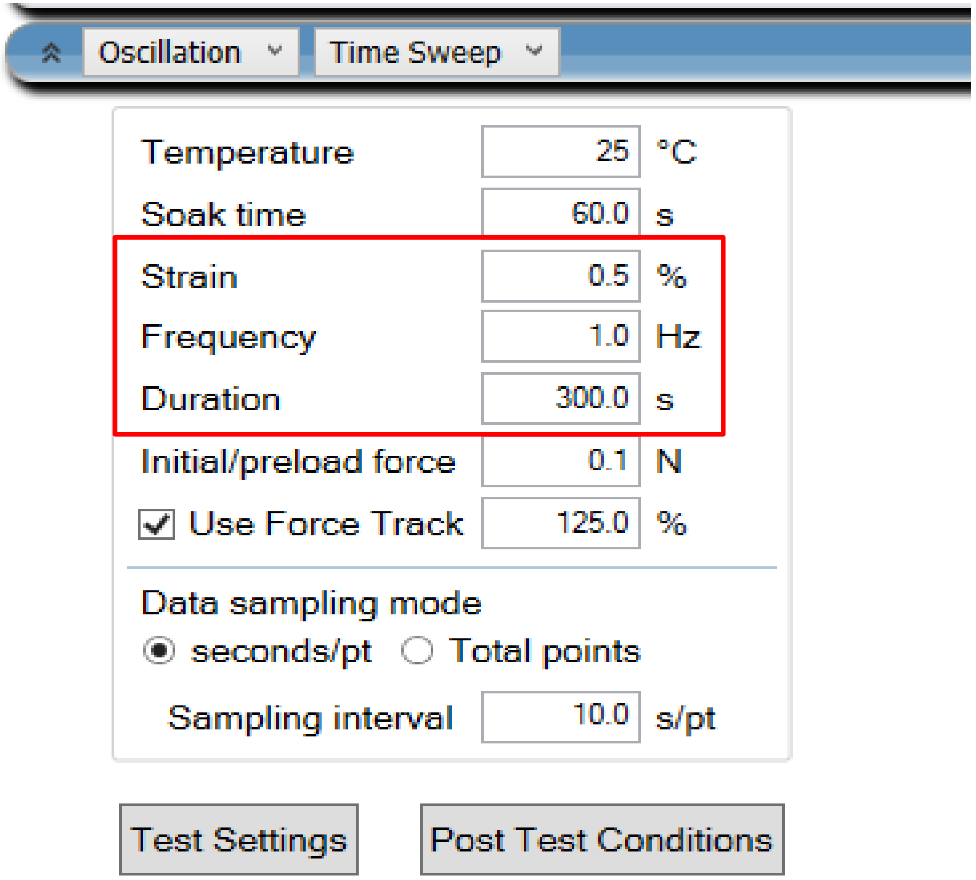
The material response is monitored at a constant frequency, amplitude and temperature.

USES

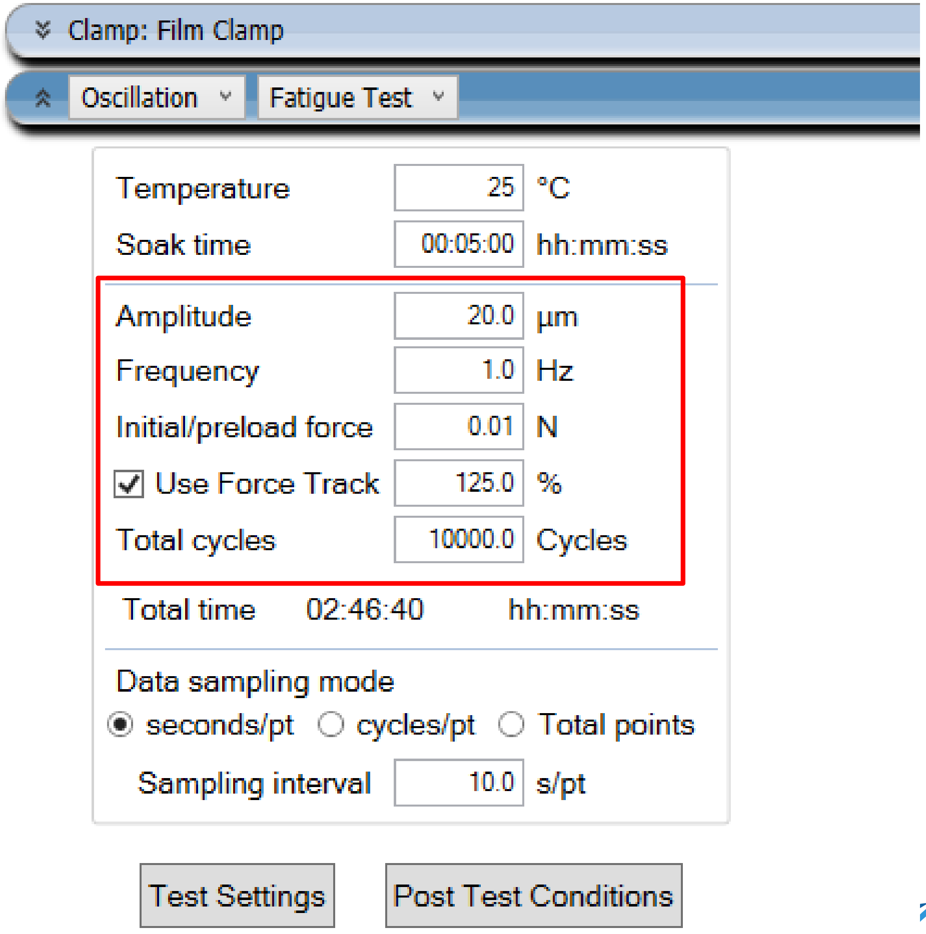
- Curing studies
- Fatigue tests
- Stability against thermal degradation

Programming Time Sweep/fatigue on DMA850

- Time sweep example

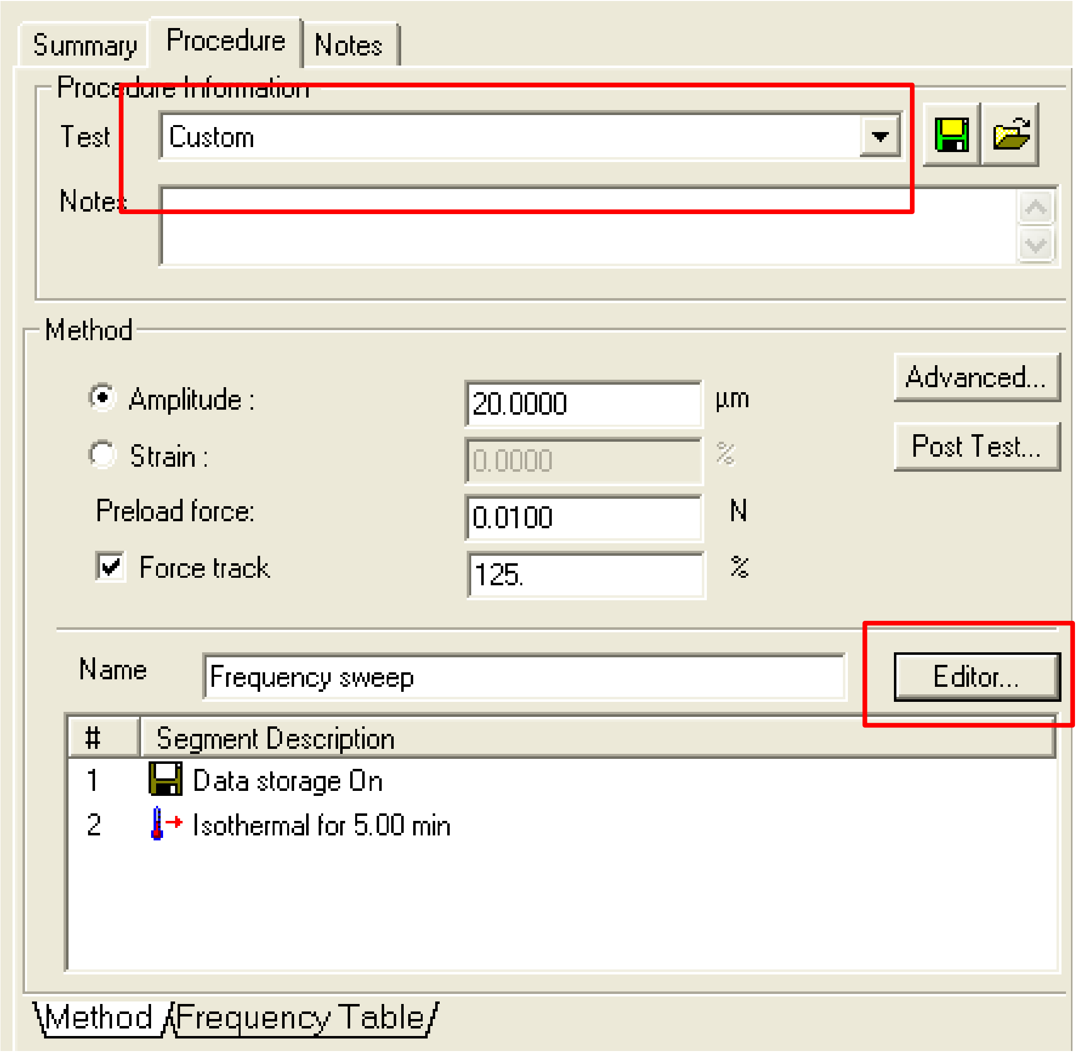


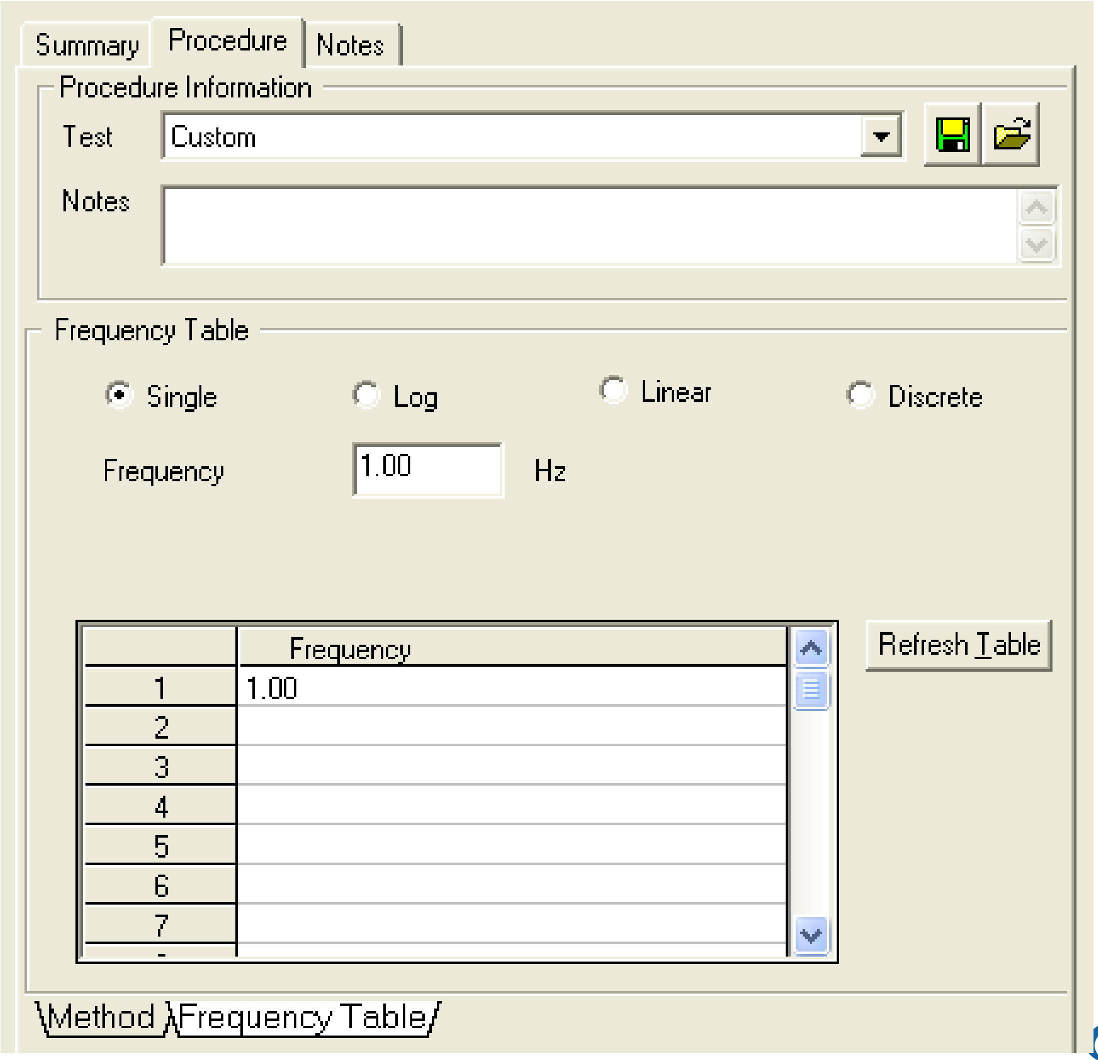
- Fatigue example



Programming Time Sweep on Q800

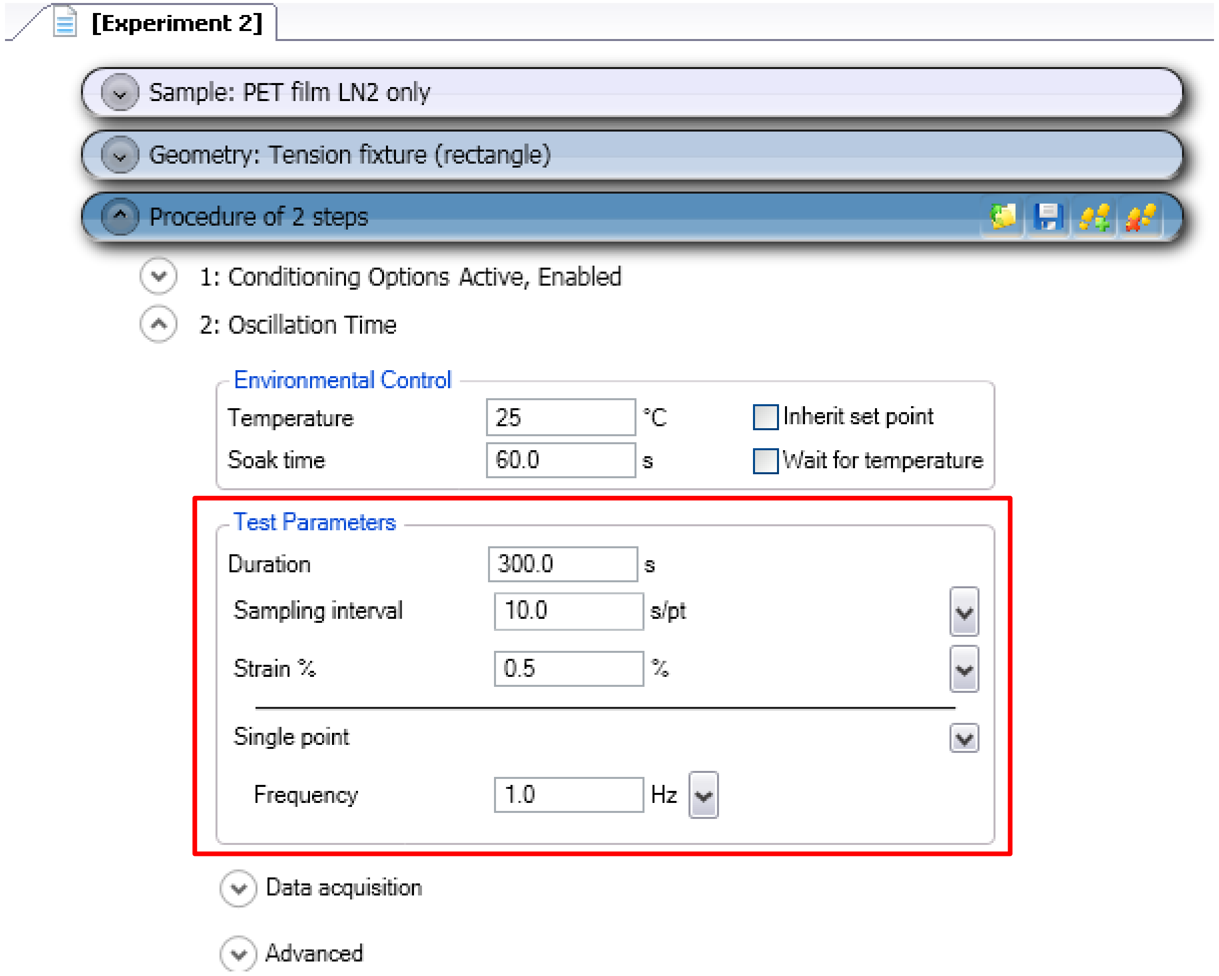
Mode: DMA Multi-Frequency-Strain



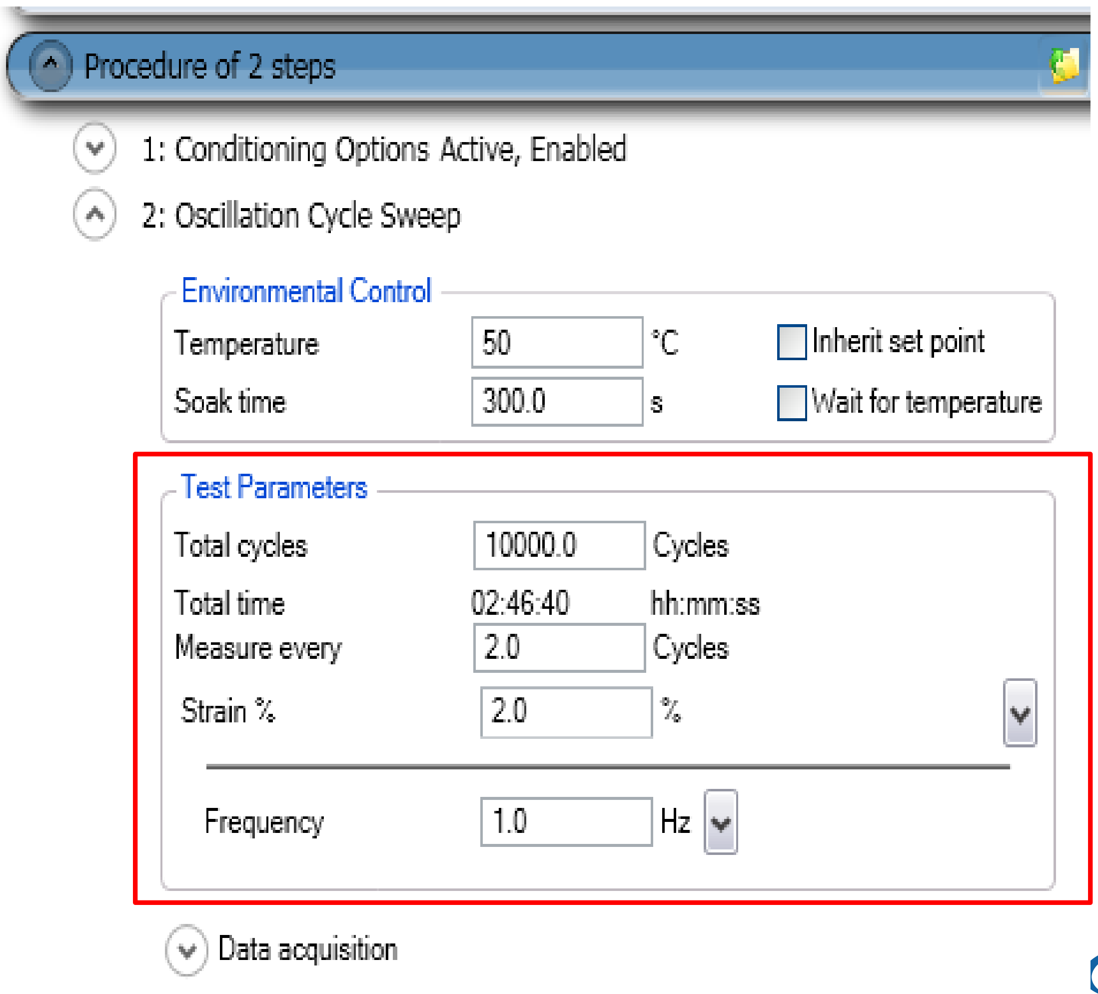


Programming Time Sweep on RSA G2

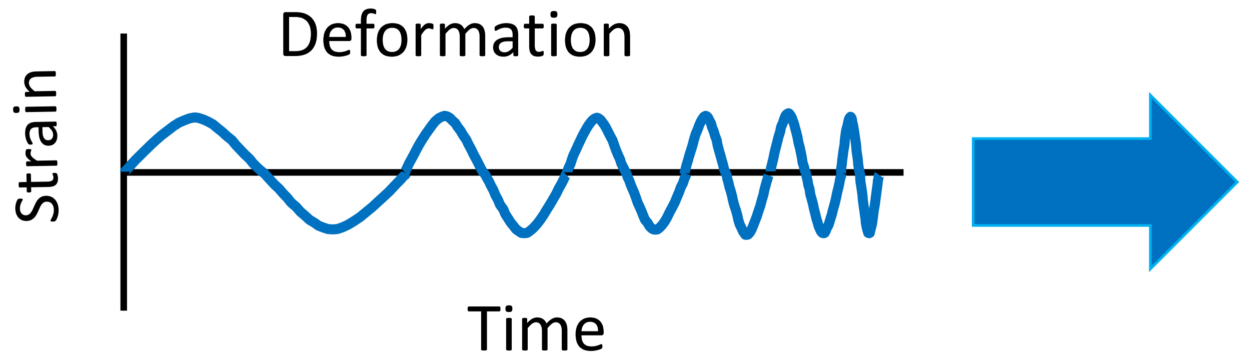
- Time sweep example



- Fatigue example



Frequency Sweep

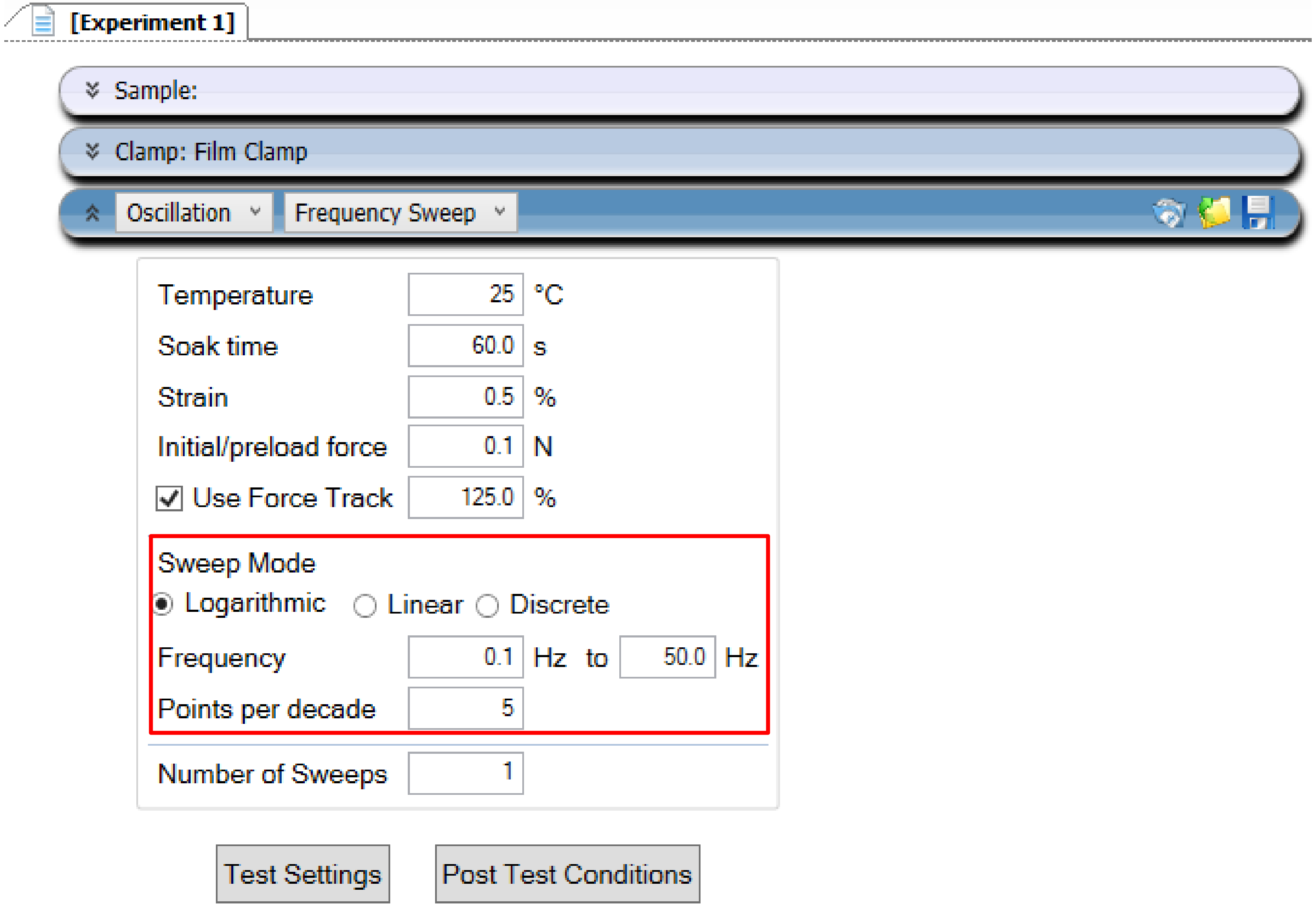


- The material response to increasing frequency (rate of deformation) is monitored at a constant amplitude and temperature.

<u>USES</u>

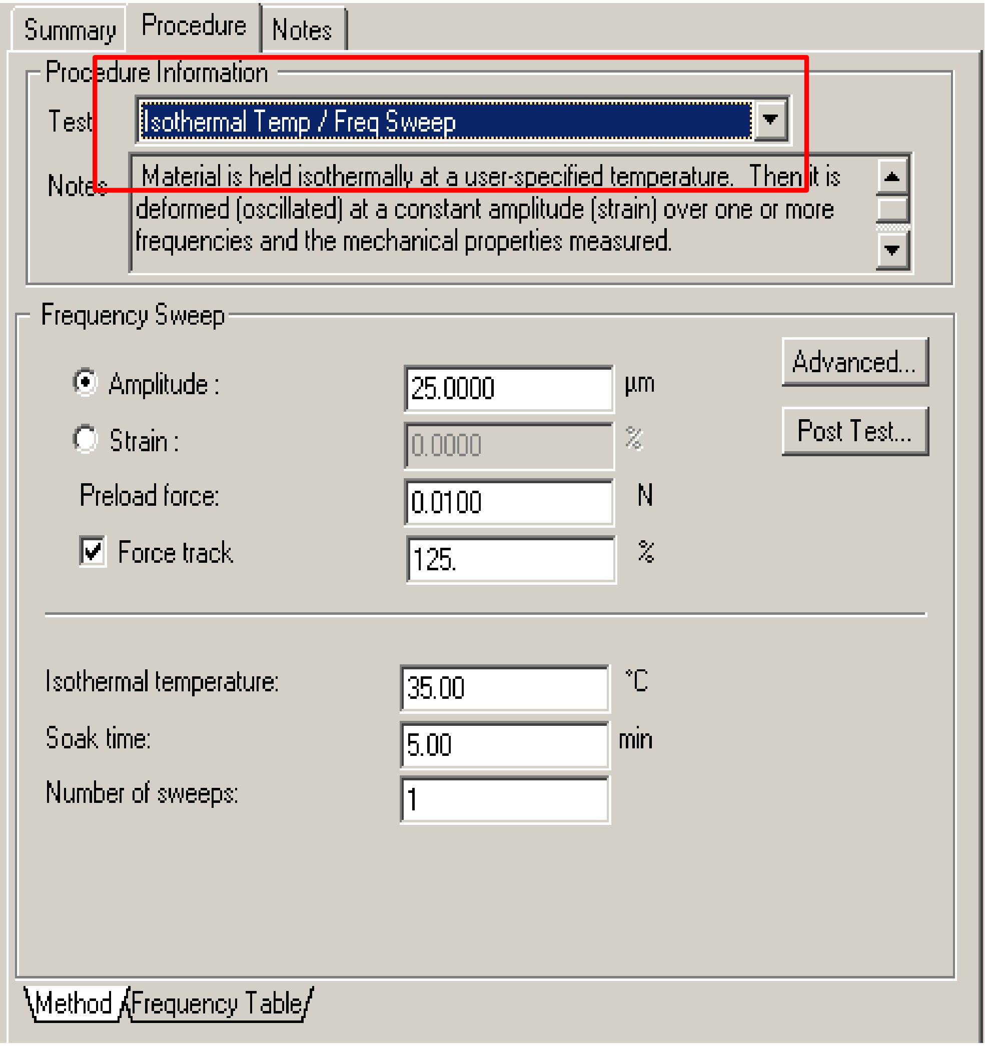
- Quick comparison on modulus and elasticity on solids
- Study polymer melt processing (shear sandwich).
- Estimate long term properties with extended frequency (time) range using TTS

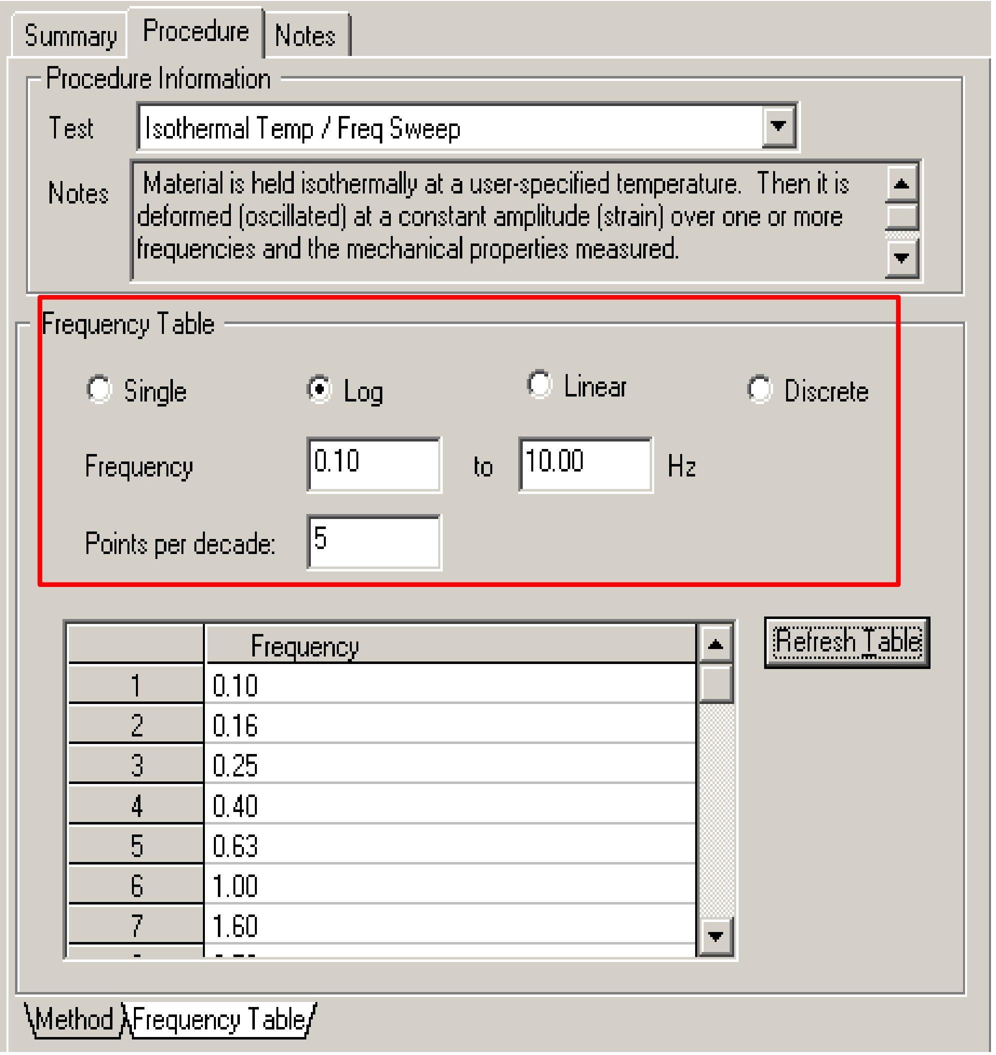
Programming Frequency Sweep on DMA850



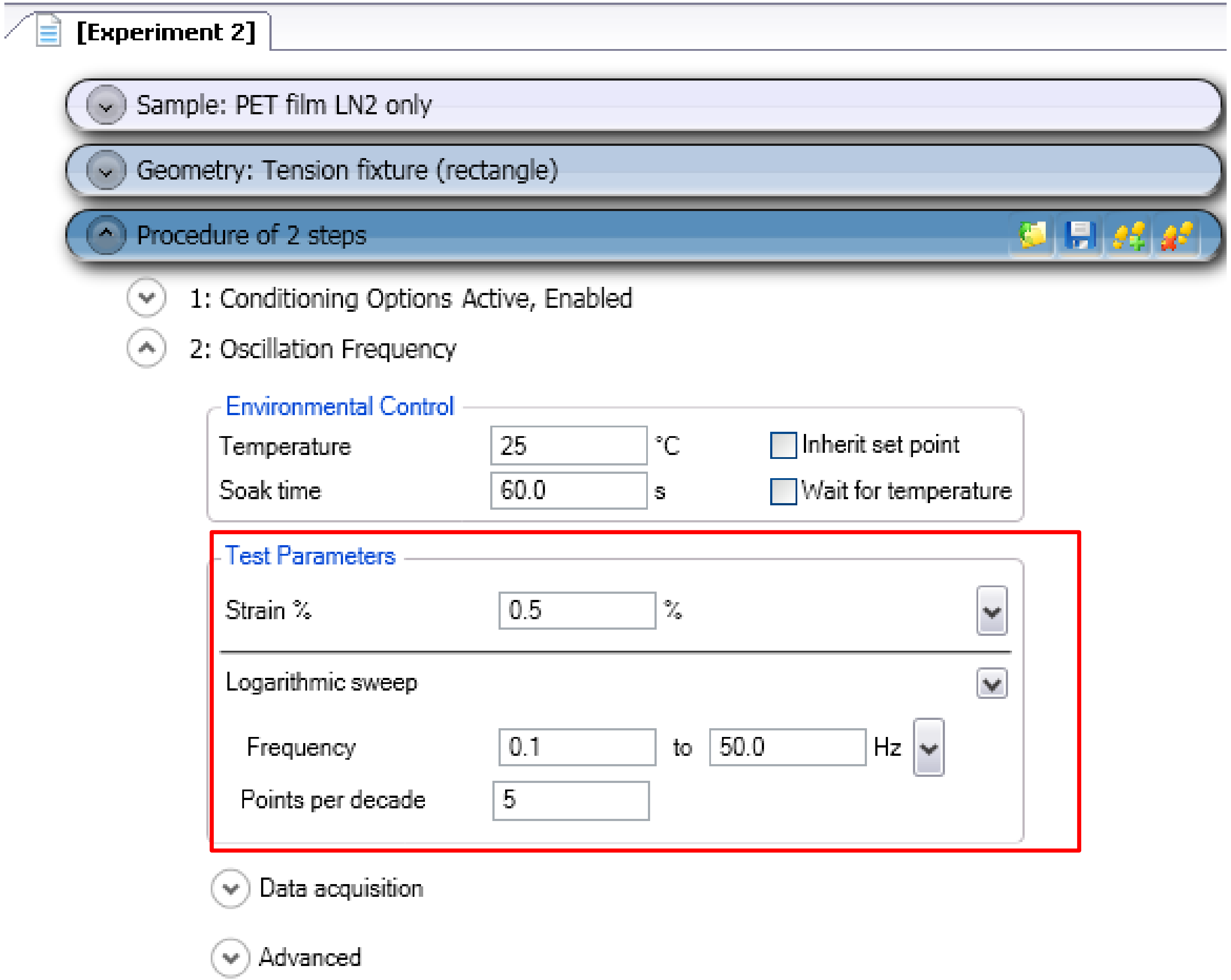
Programming Frequency Sweep on Q800

Mode: DMA Multi-Frequency-Strain



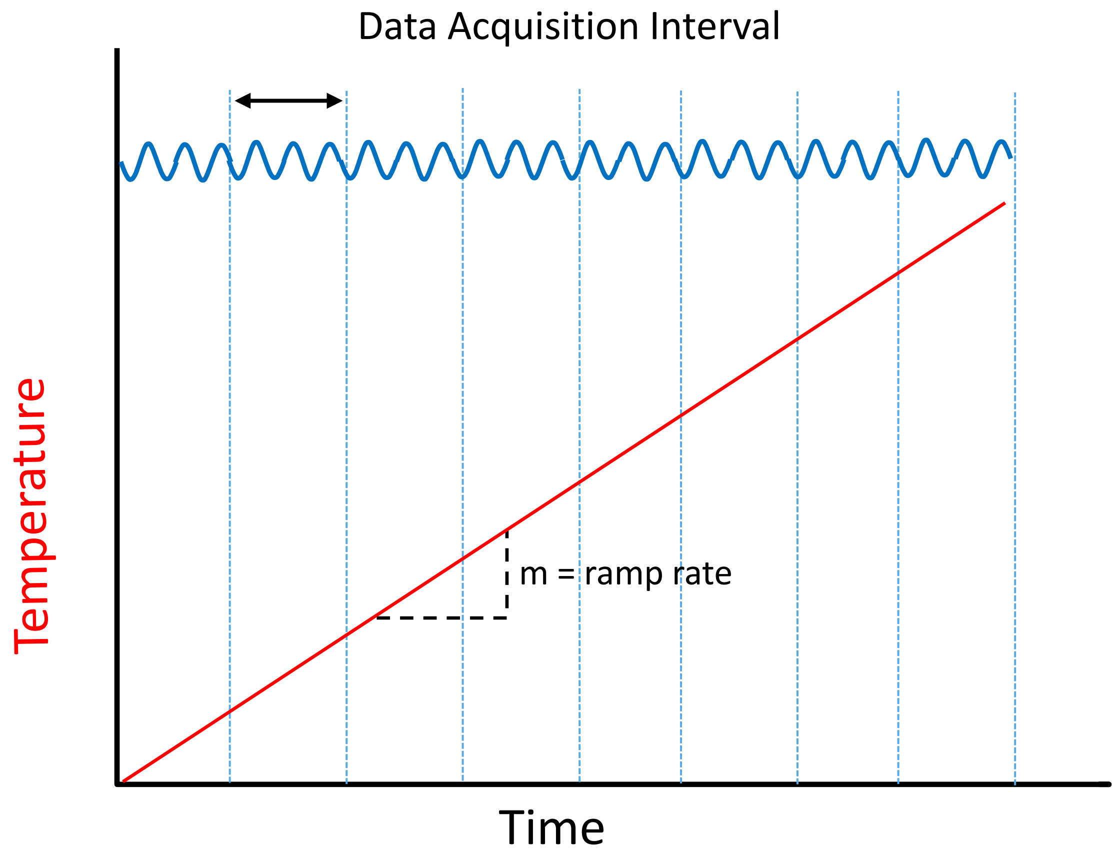


Programming Frequency Sweep on RSA G2



Dynamic Temperature Ramp

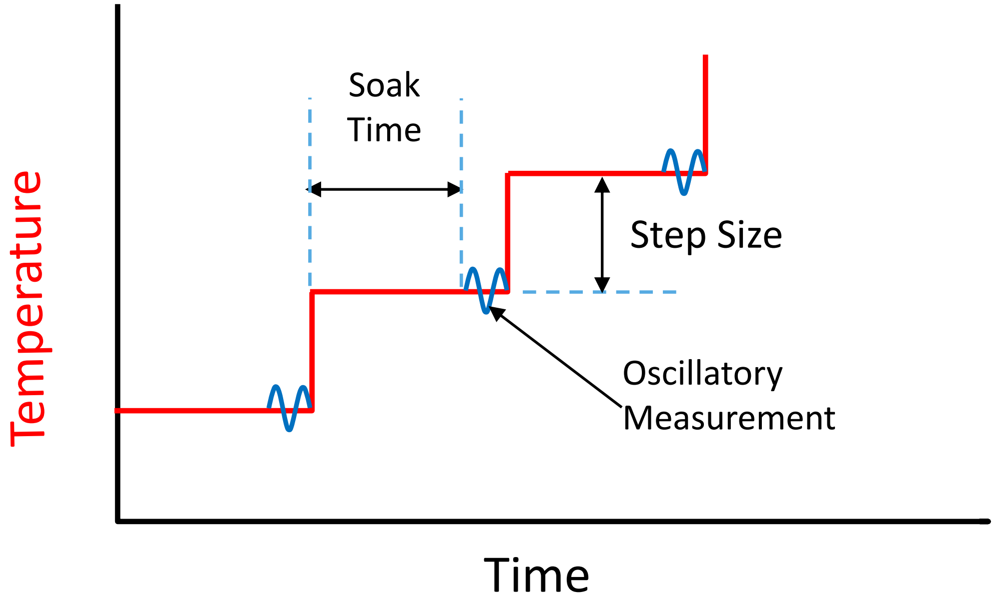
- A linear heating rate is applied. The material response is monitored at a constant frequency and constant amplitude of deformation. Data is taken at user defined time intervals.

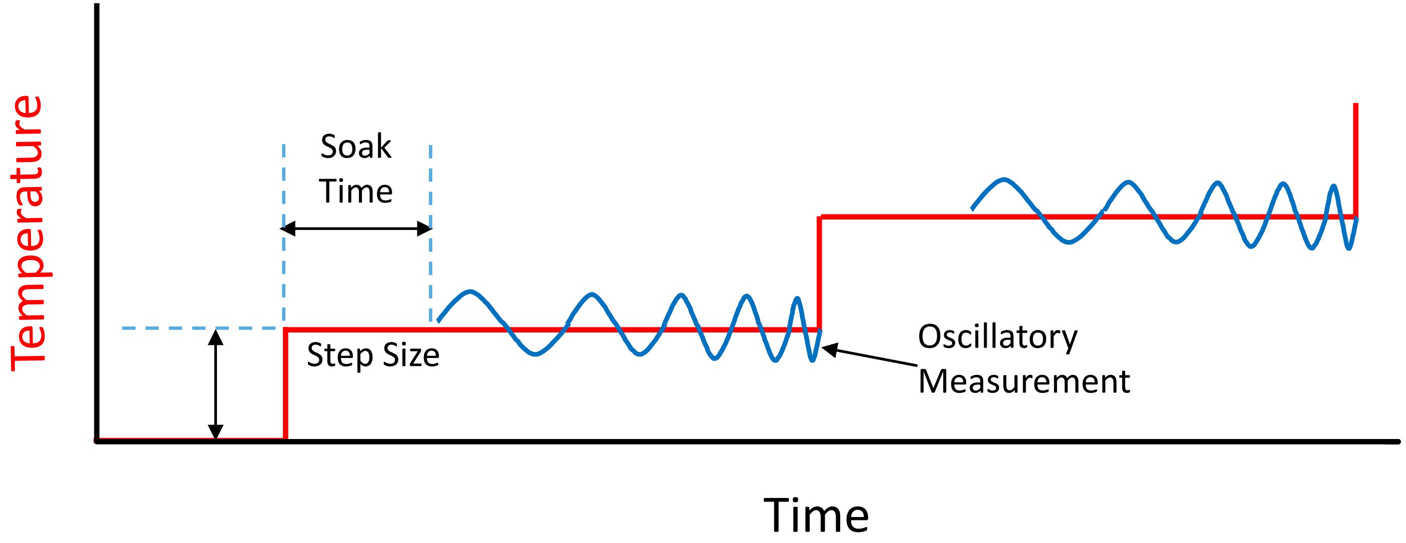


Recommend ramp rate for polymer testing: 1-5˚C/min.

Temperature Step & Hold- Single /Multi-Frequency

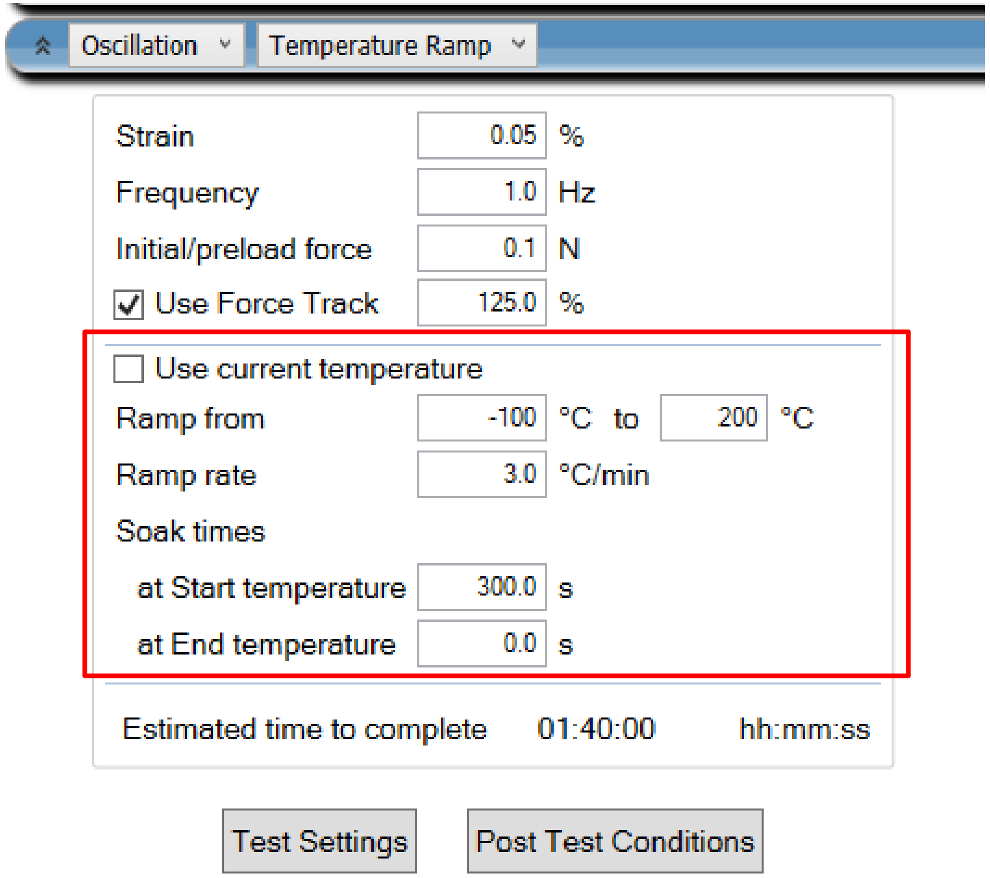
- A step and hold temperature profile is applied. The material response is monitored at one, or over a range of frequencies, at constant amplitude of deformation



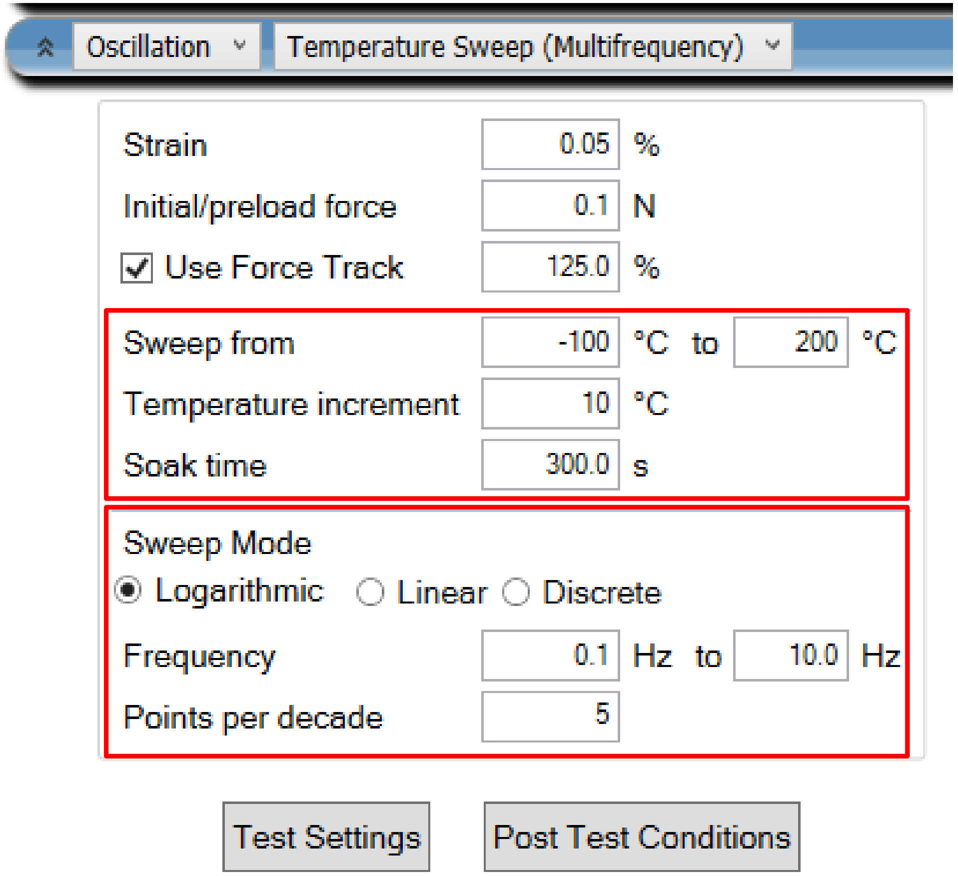


Programming Temp Ramp/Sweep on DMA850

- Temp Ramp Example



- Temp Sweep Example

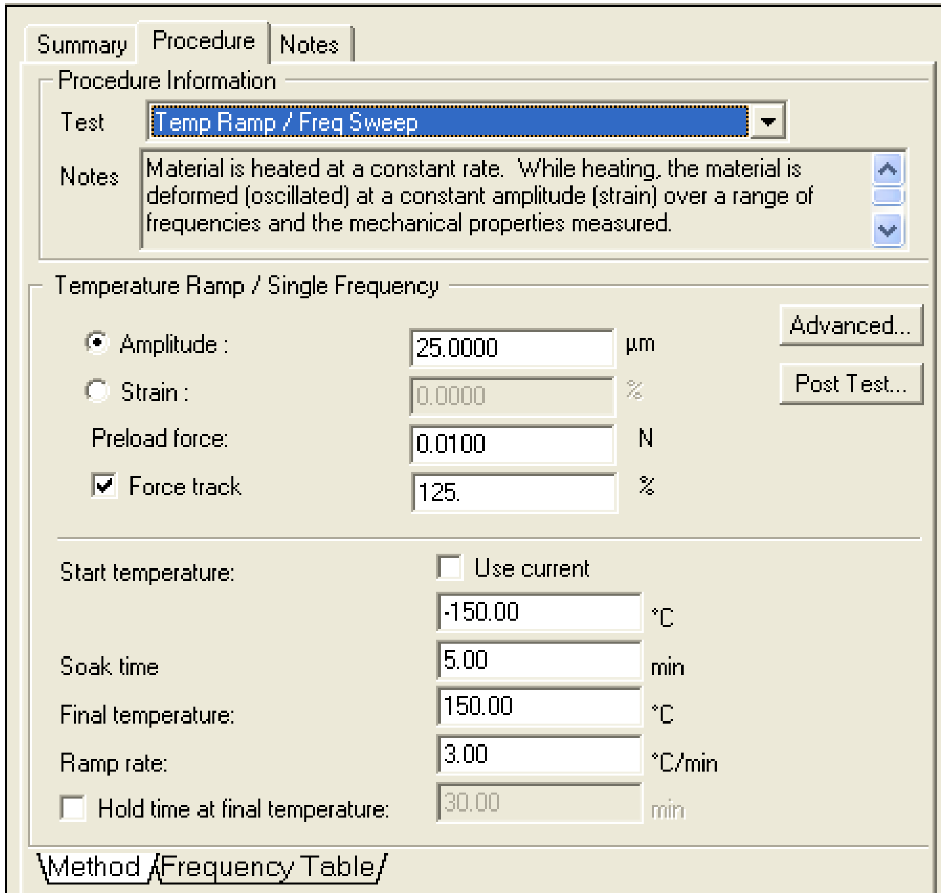


Note: Measurement can be done with single or multiple frequencies

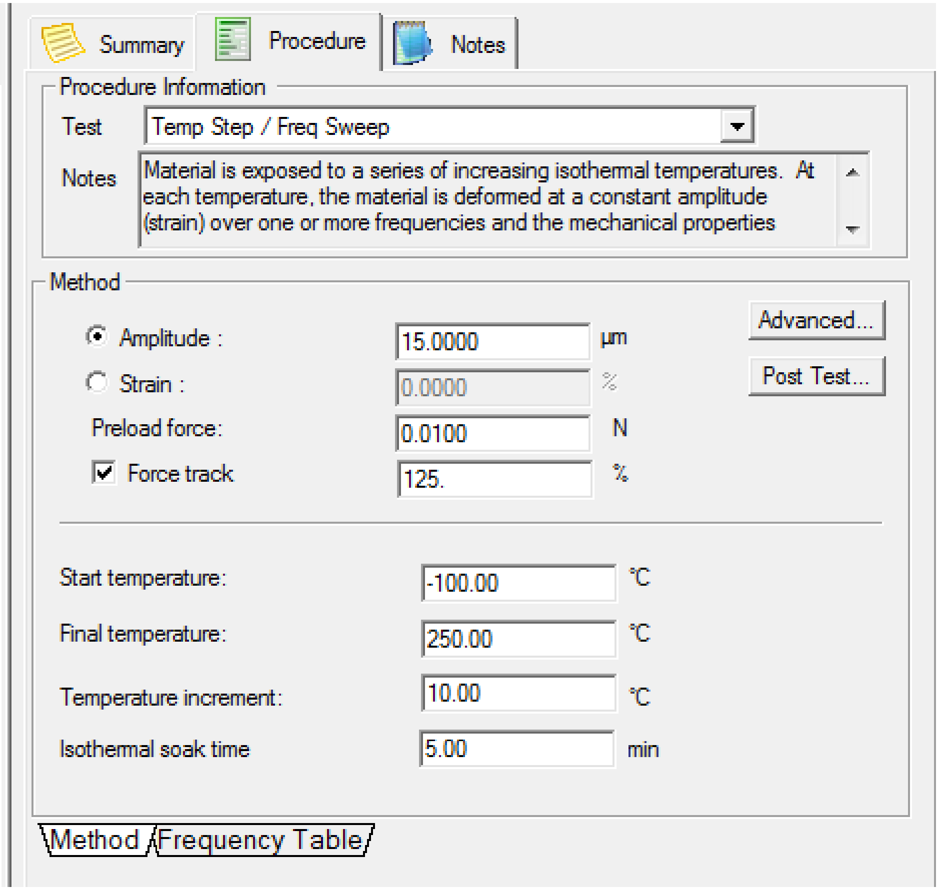
Programming Temp Ramp/Step on Q800

Mode: DMA Multi-Frequency-Strain

- Temp Ramp Example



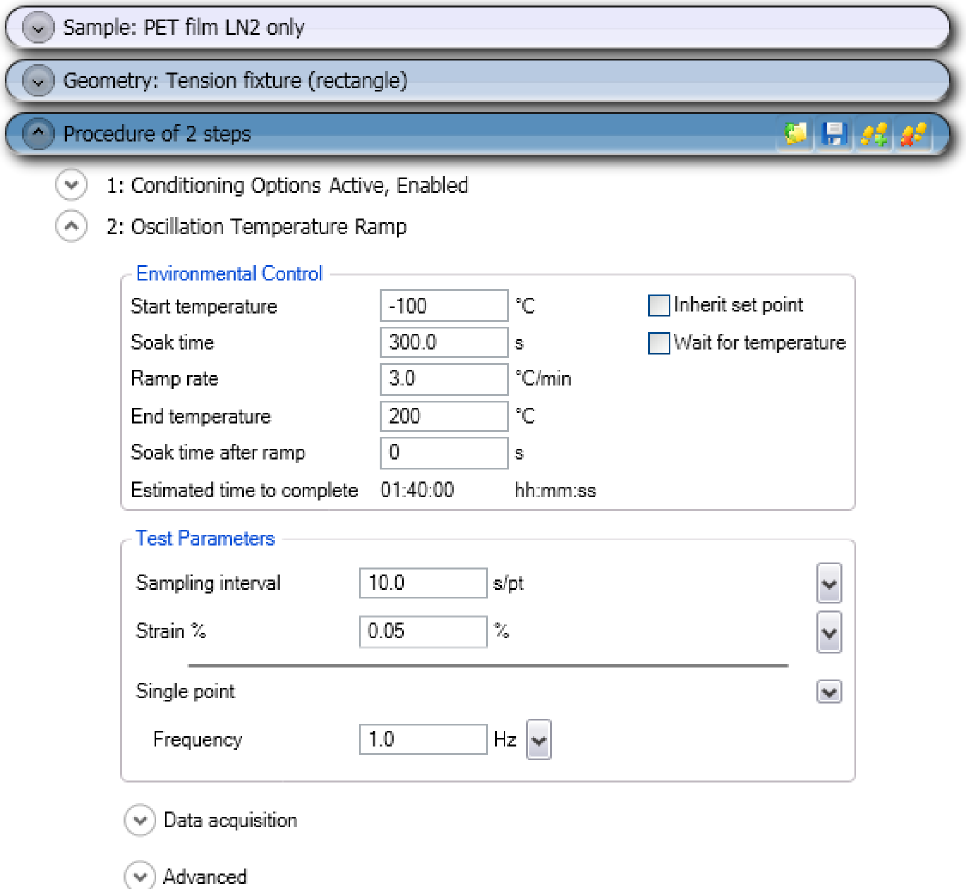
- Temp Sweep Example



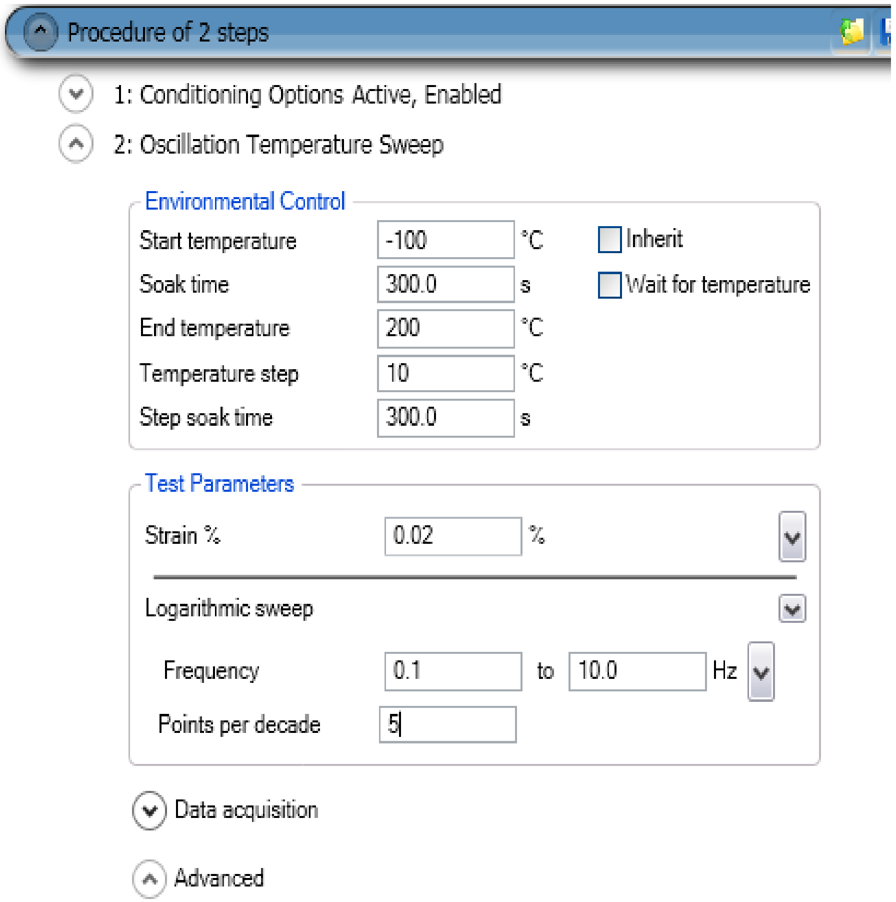
Note: Measurement can be done with single or multiple frequencies

Programming Temp Ramp/Sweep on RSA G2

- Temp Ramp Example



- Temp Sweep Example



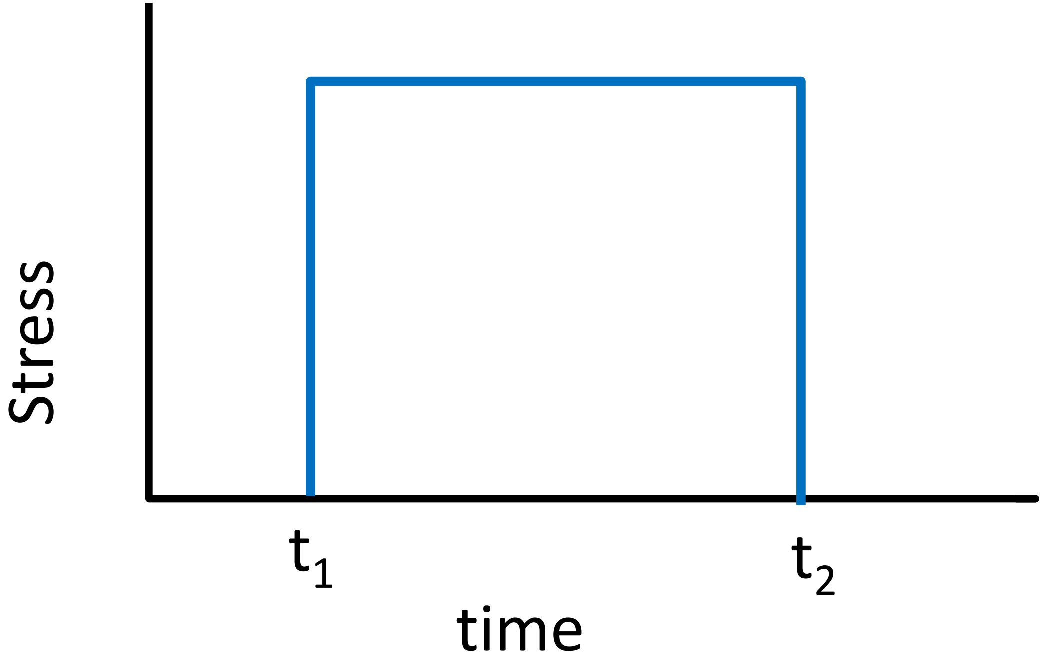
Note: Measurement can be done with single or multiple frequencies

Transient experiments

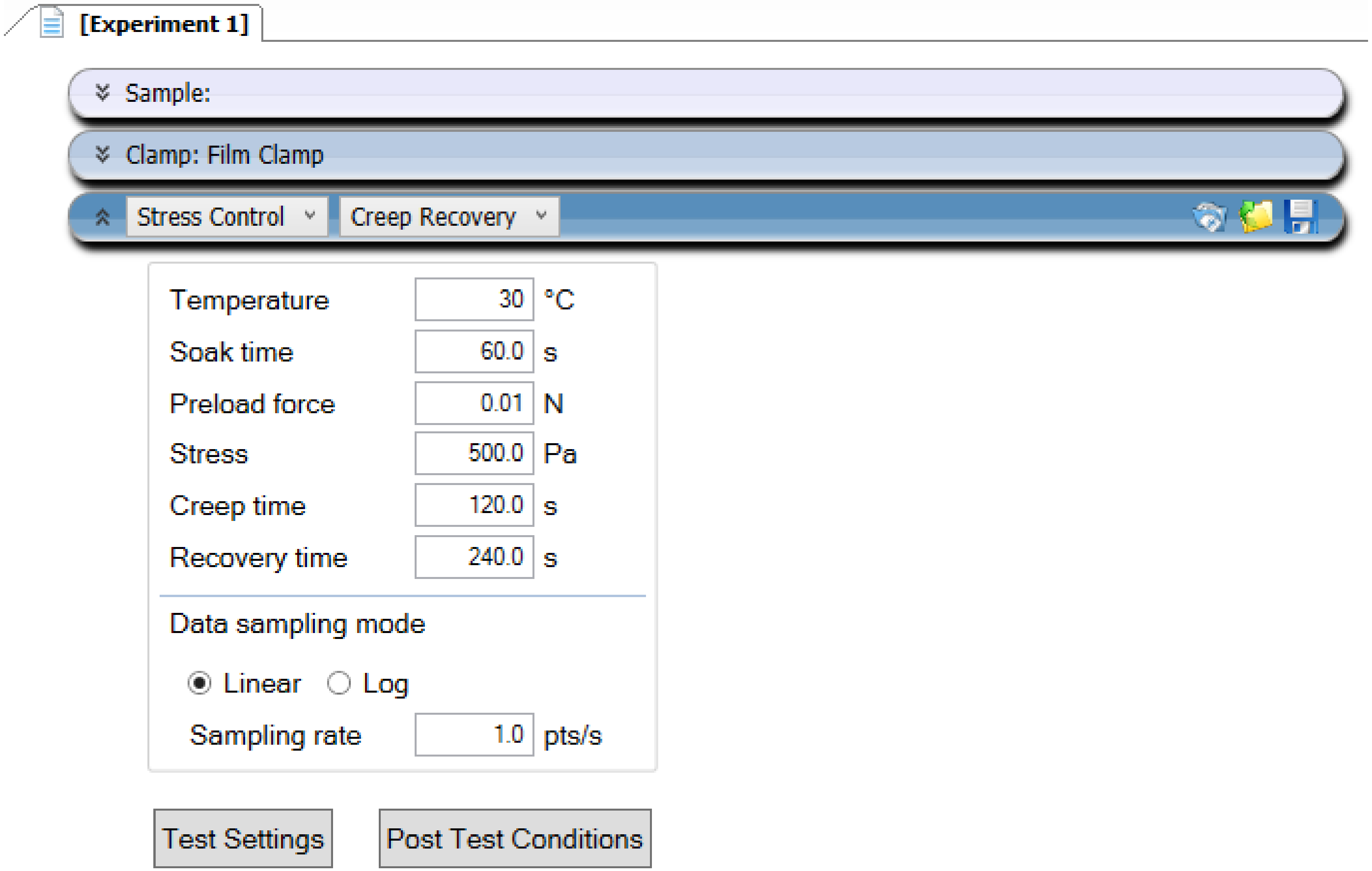
Creep Recovery Experiment

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

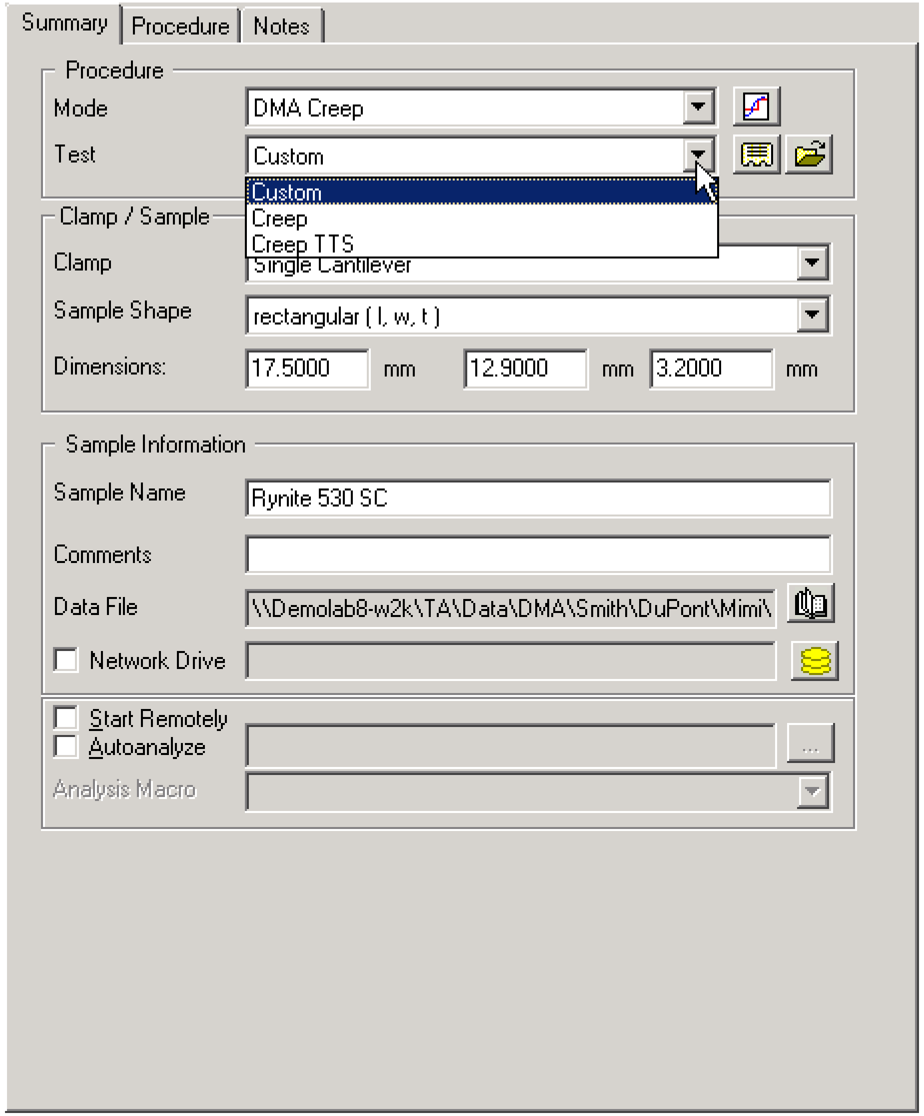
- The stress is reduced to zero at t_2 and the strain is monitored as a function of time ($\gamma(t)$ or $\varepsilon(t)$).

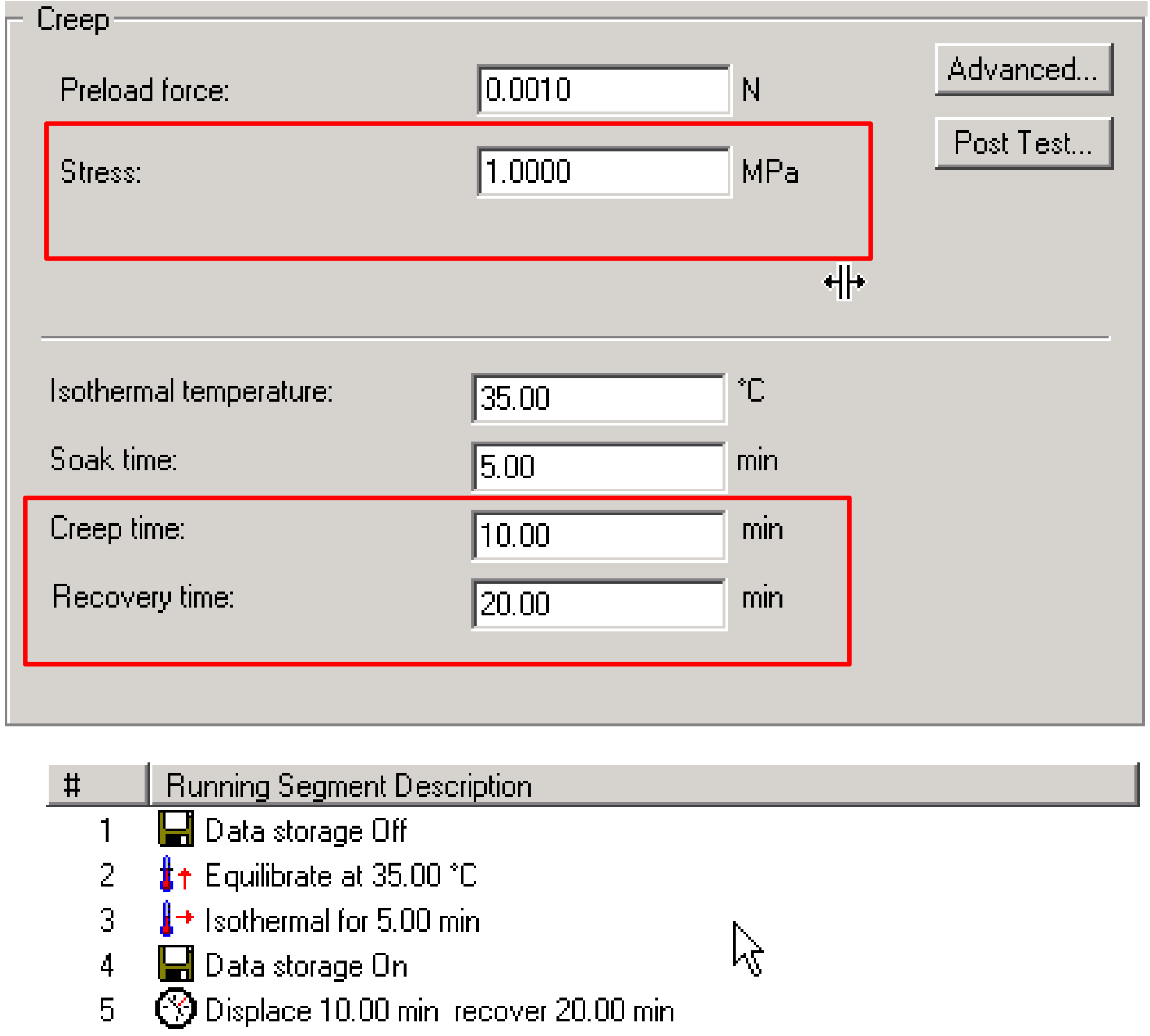


Programming Creep Recovery on a DMA850



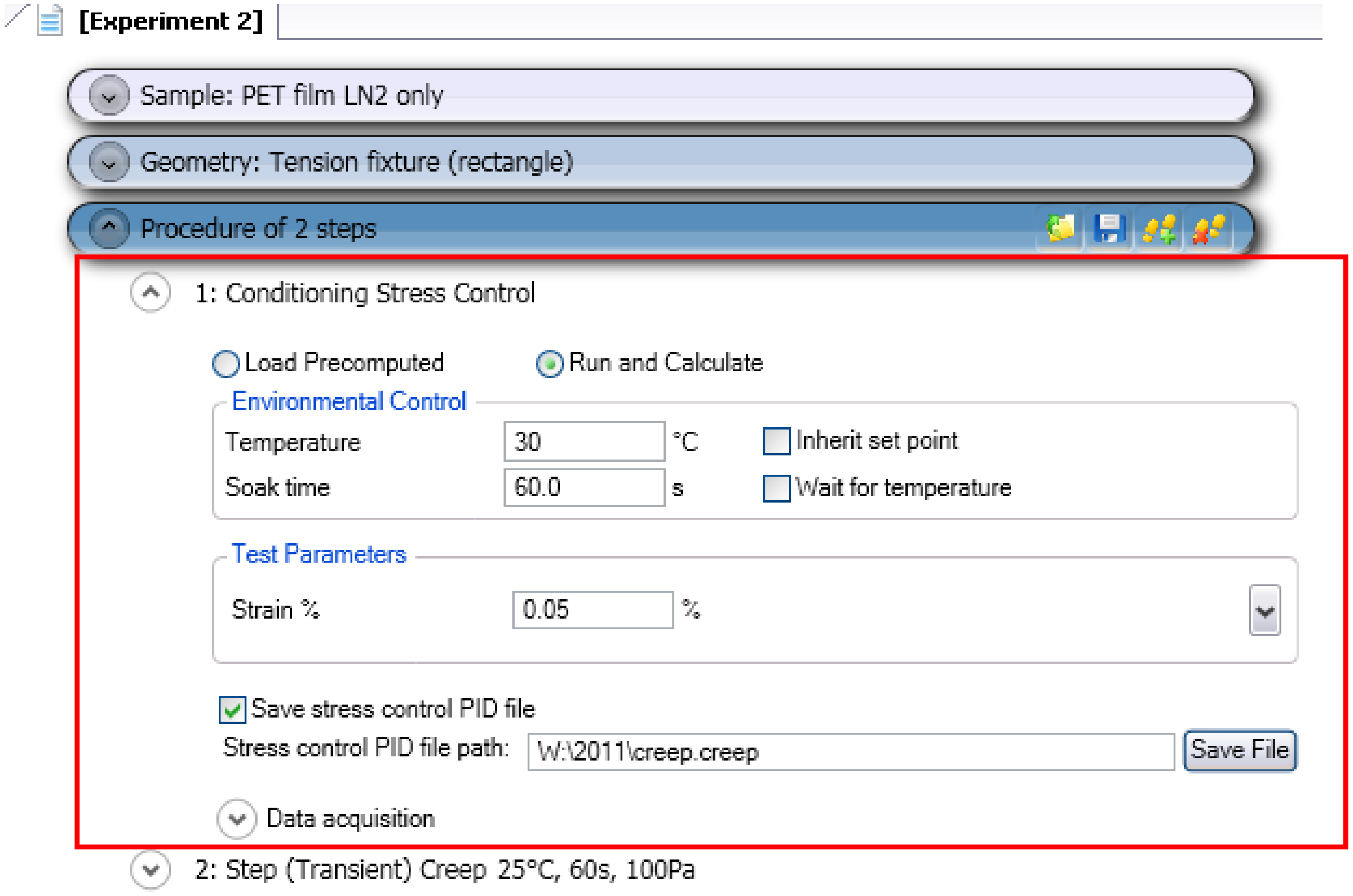
Programming Creep Recovery on a Q800





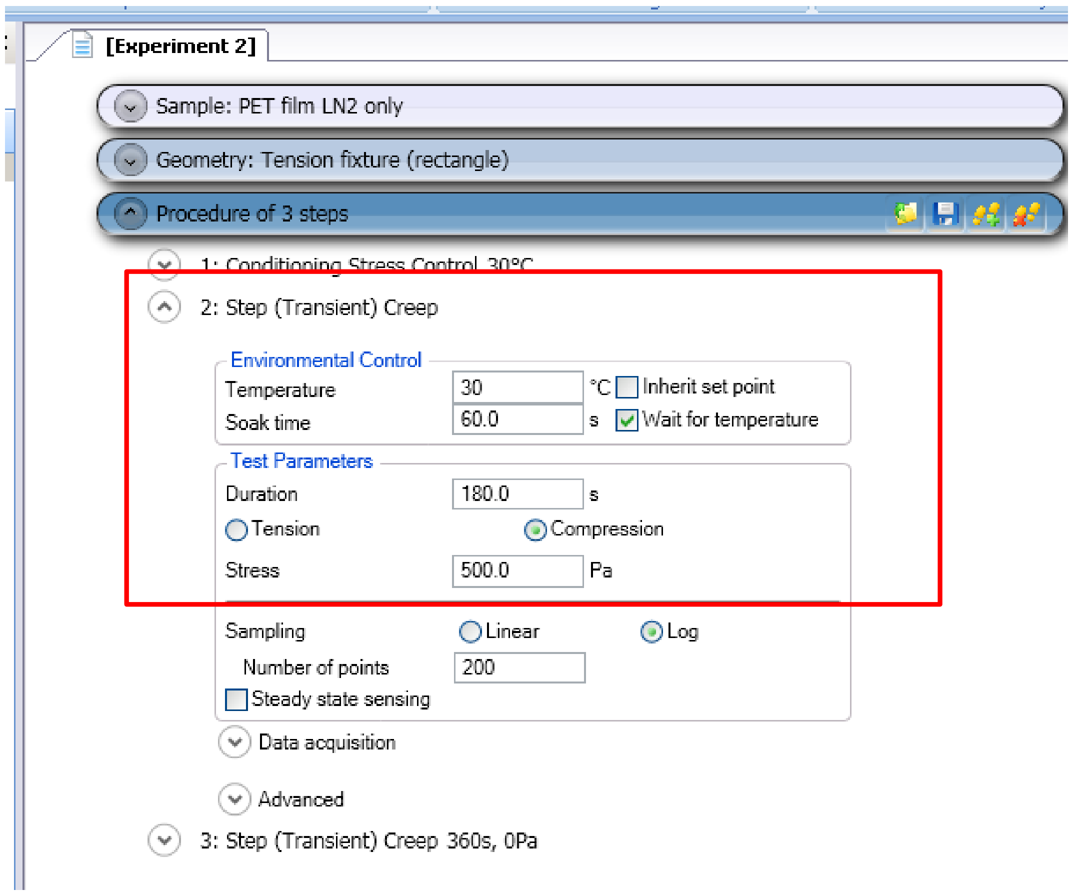
Programming Creep Recovery on a RSA-G2

- A pre-test is required to obtain sample information for the feedback loop
- Stress Control Pre-test: frequency sweep within LVR

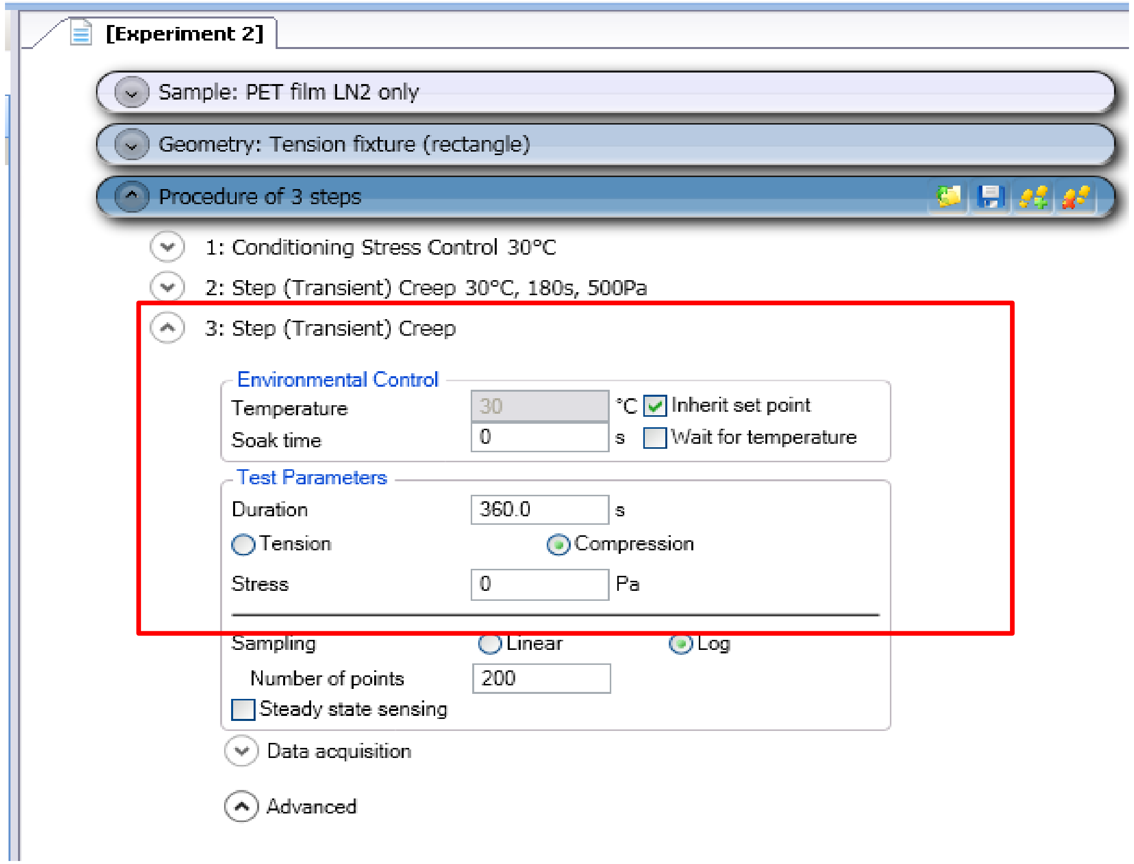


Programming Creep Recovery on a RSA-G2

Creep



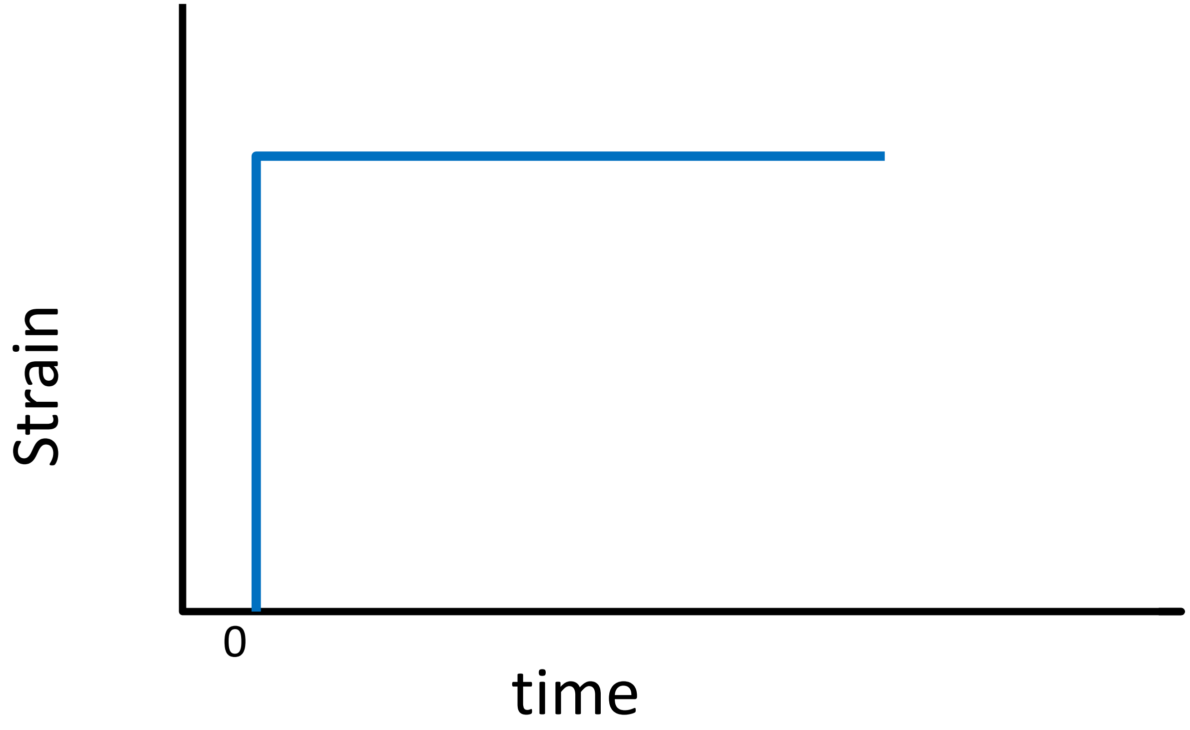
Recovery



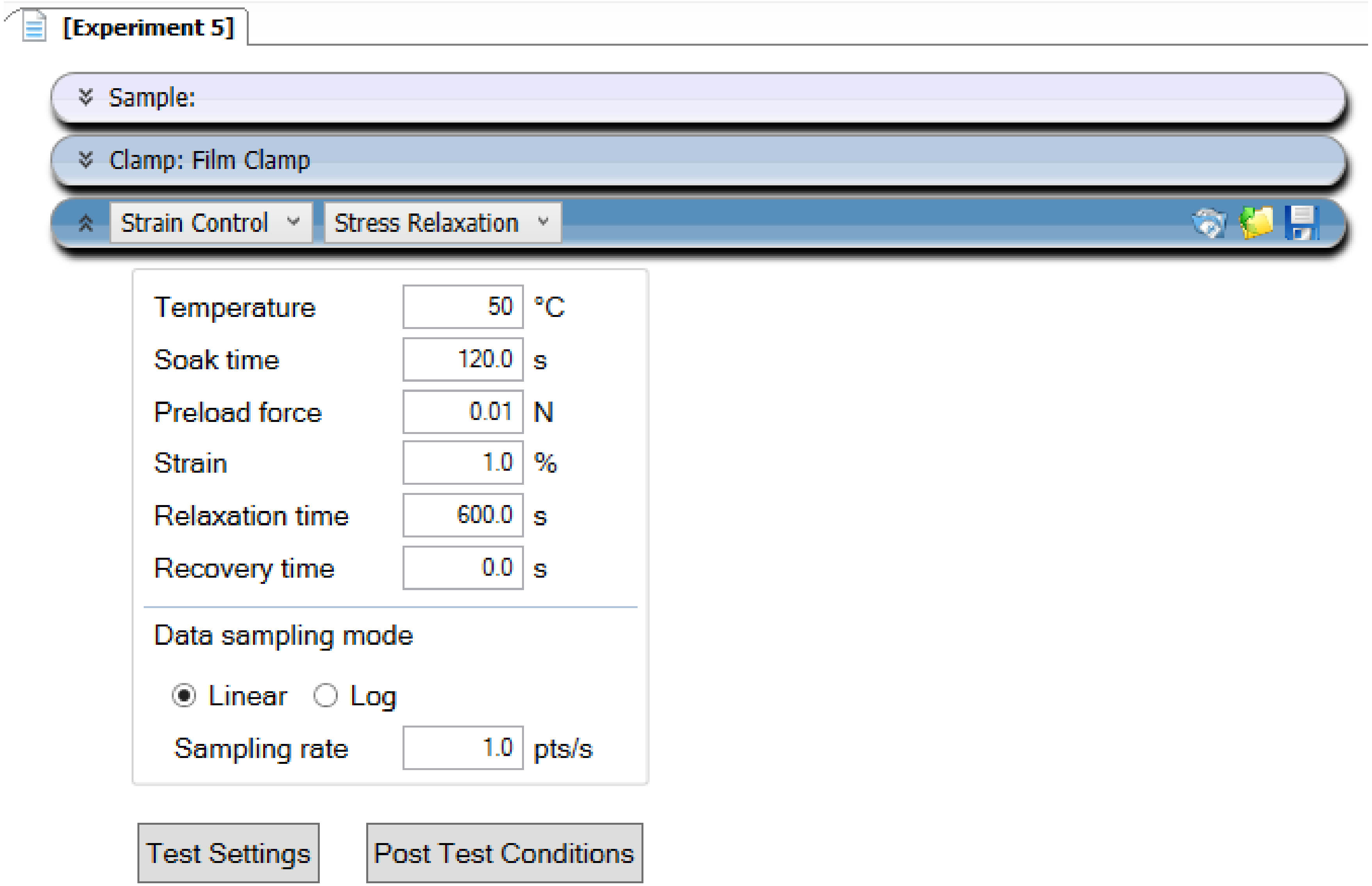
- Stress: needs to be in the linear region
- Creep time: until it reaches steady state
- Recovery time: until the compliance and strain reach plateau

Stress relaxation experiment

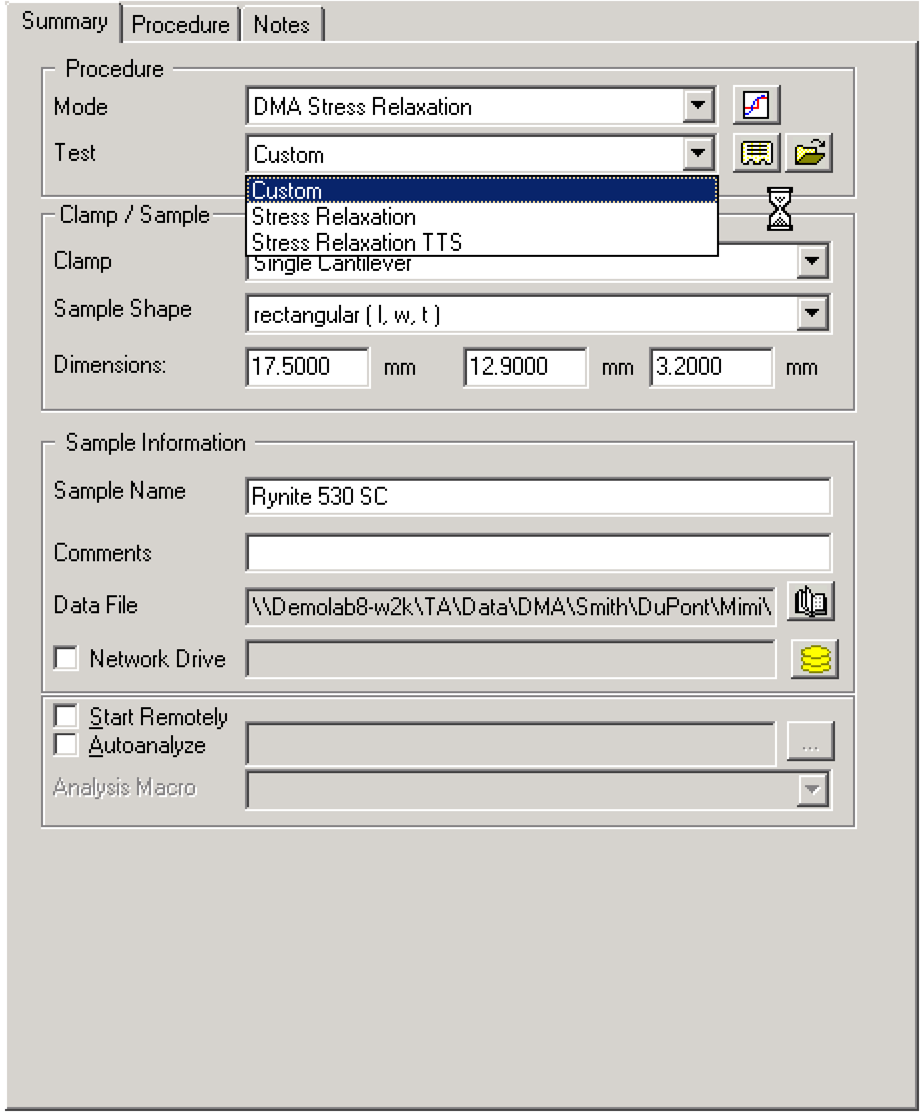
- Strain is applied to sample instantaneously (in principle) and held constant with time.
- Stress is monitored as a function of time $\sigma(t)$.

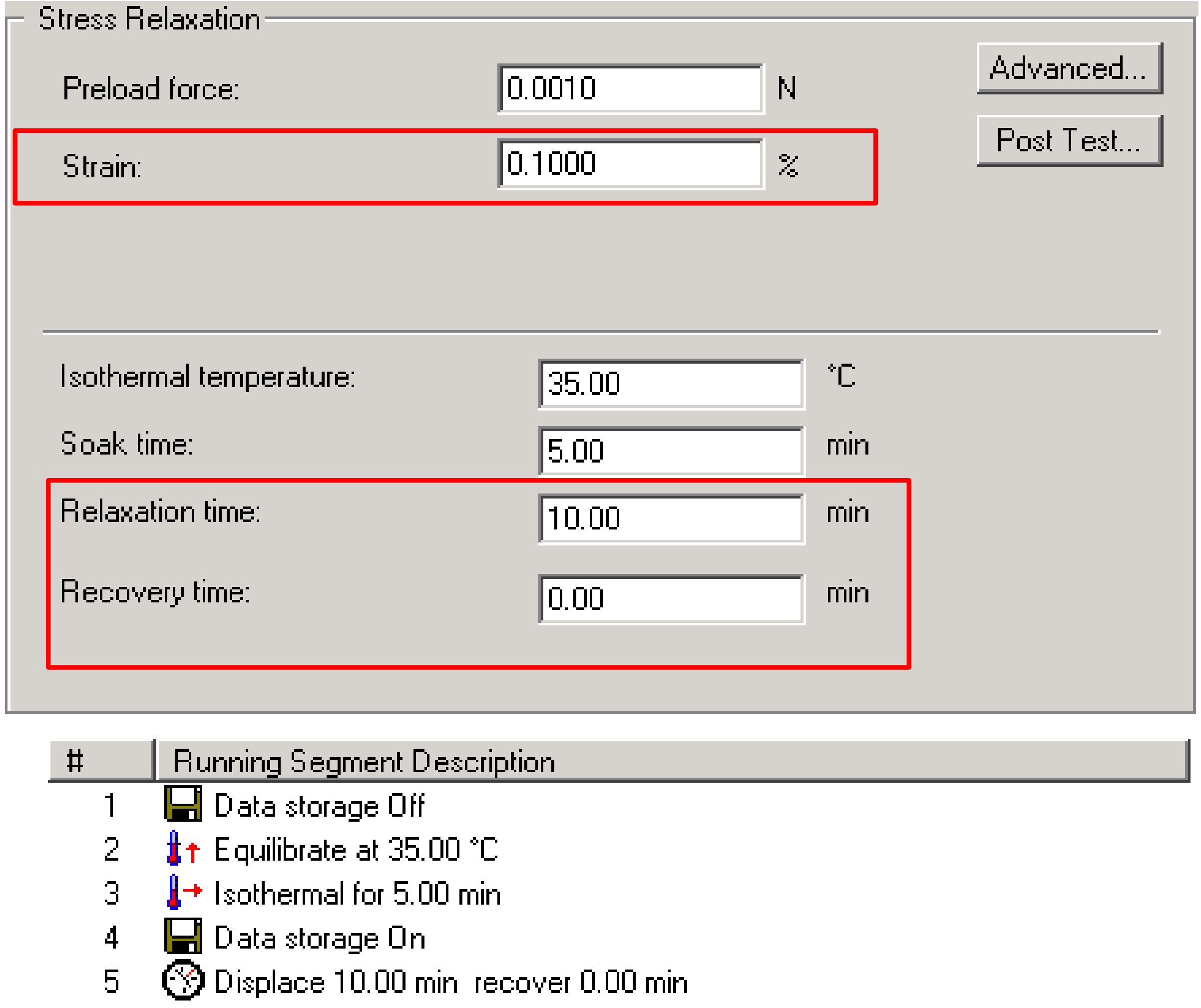


Programming Stress Relaxation on a DMA850

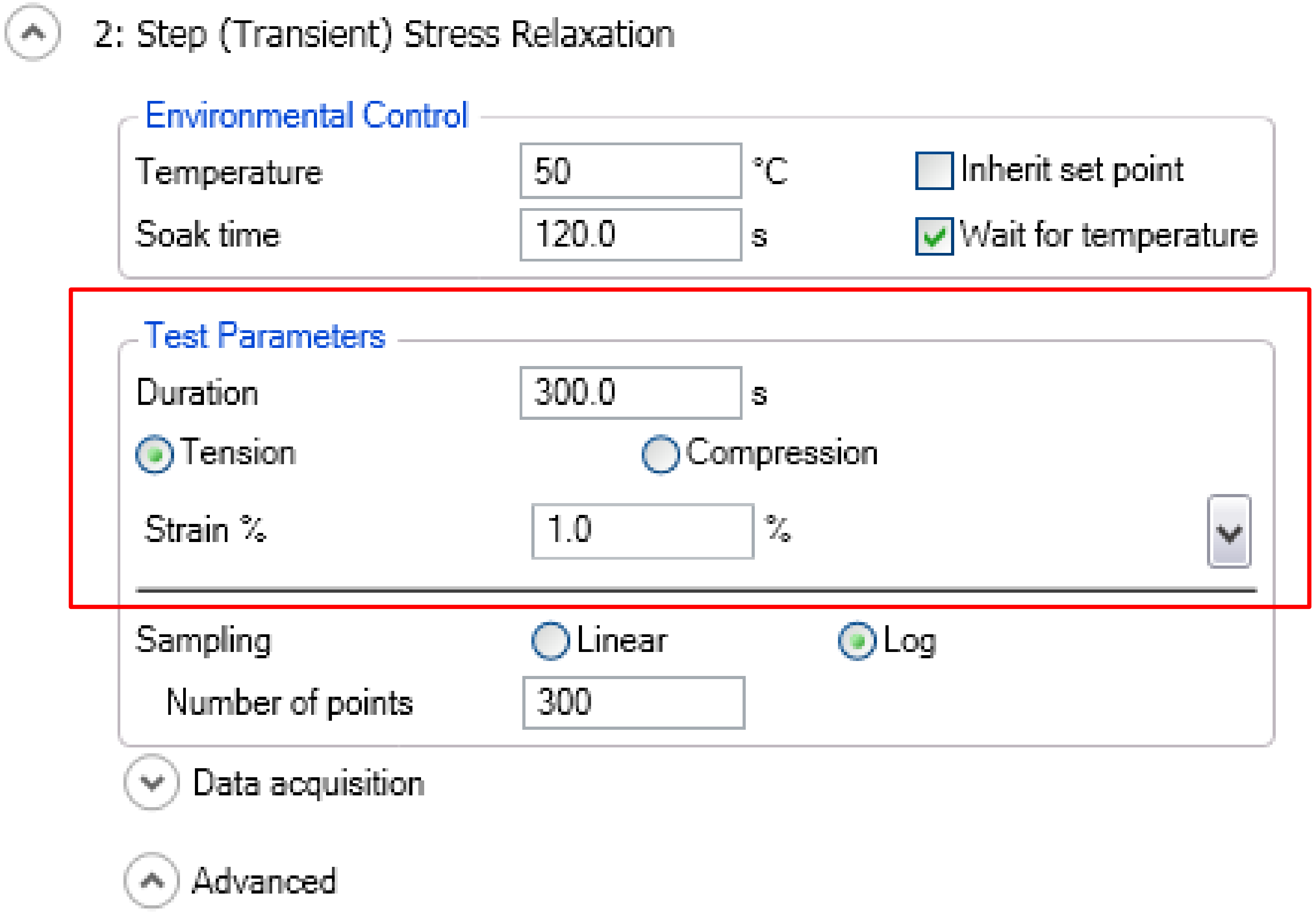


Programming Stress Relaxation on a Q800



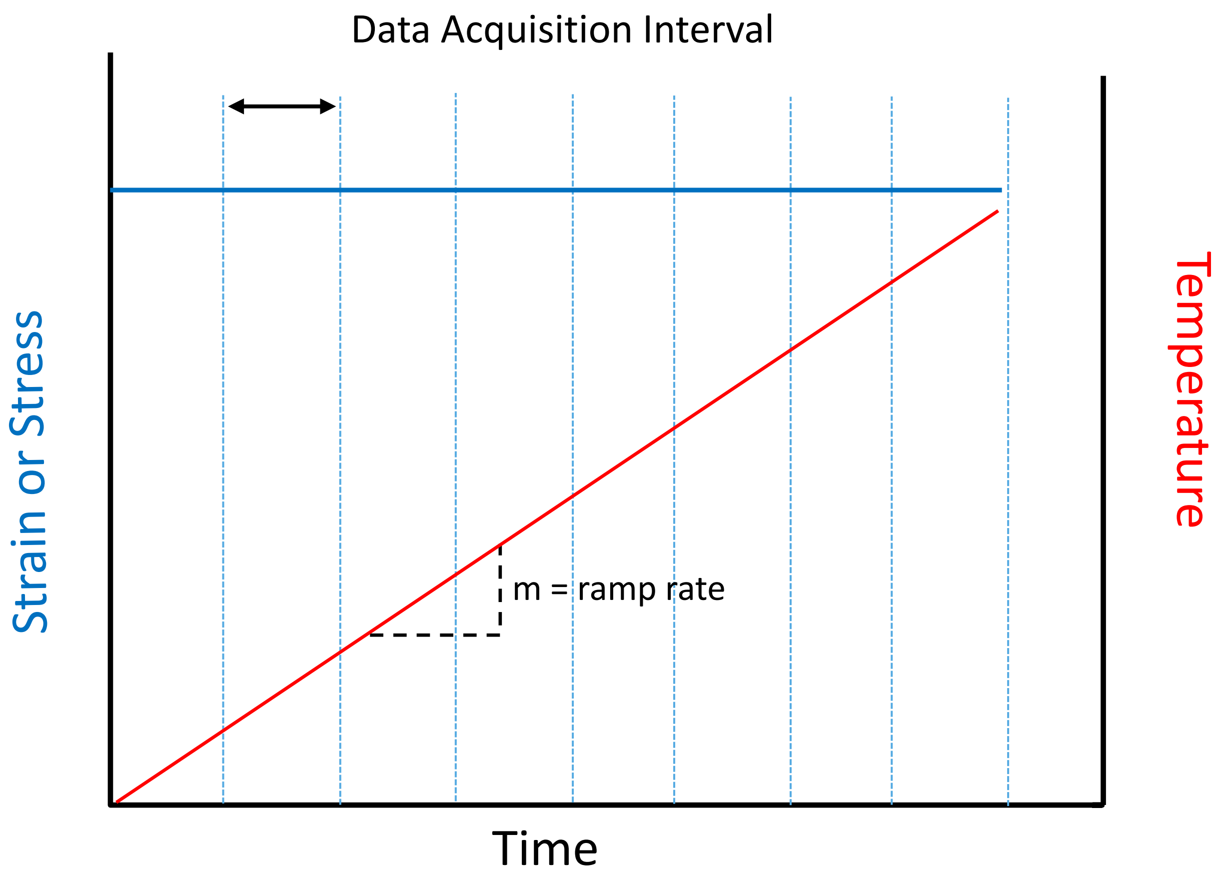


Programming Stress Relaxation on a RSA-G2



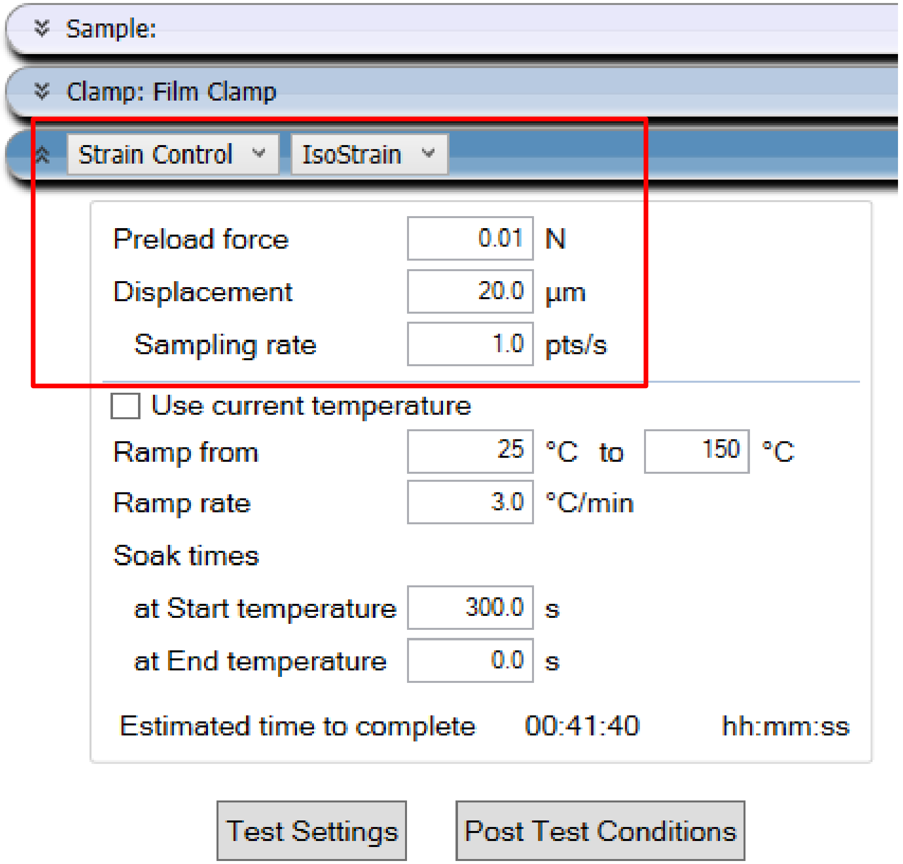
Iso-strain/Iso-stress Temperature Ramp

- 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)

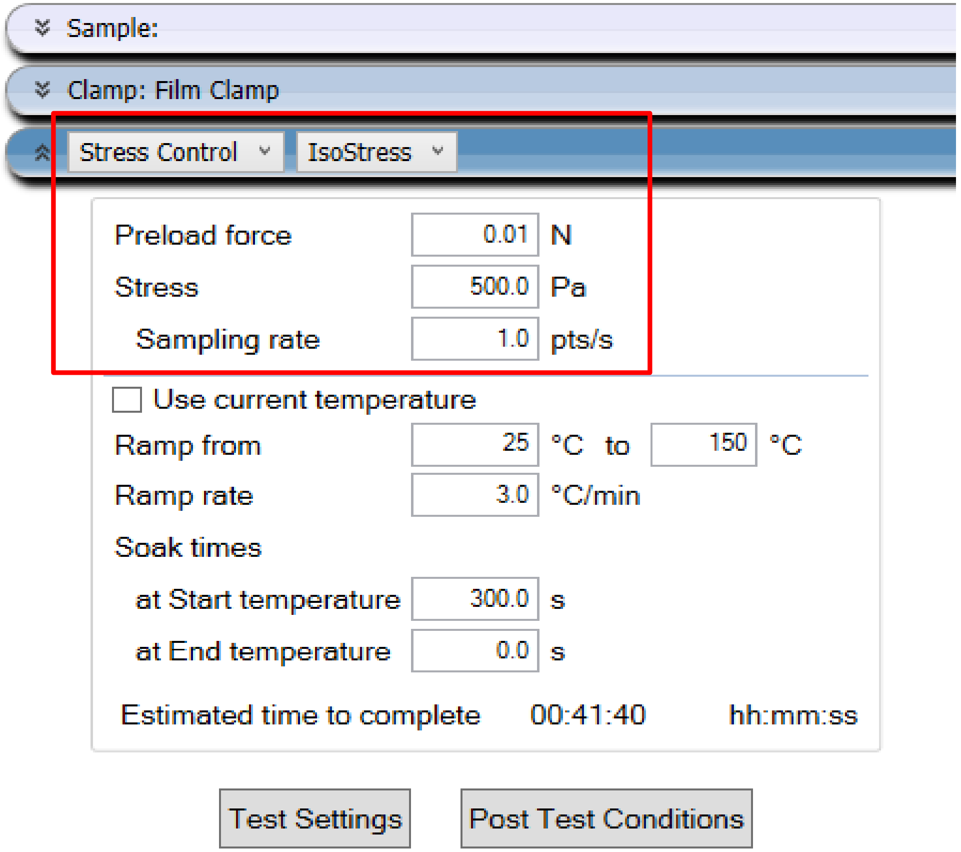


Iso-Strain/Iso-Stress on a DMA850

- DMA Iso-strain
- Hold strain constant and measure sample shrinking force

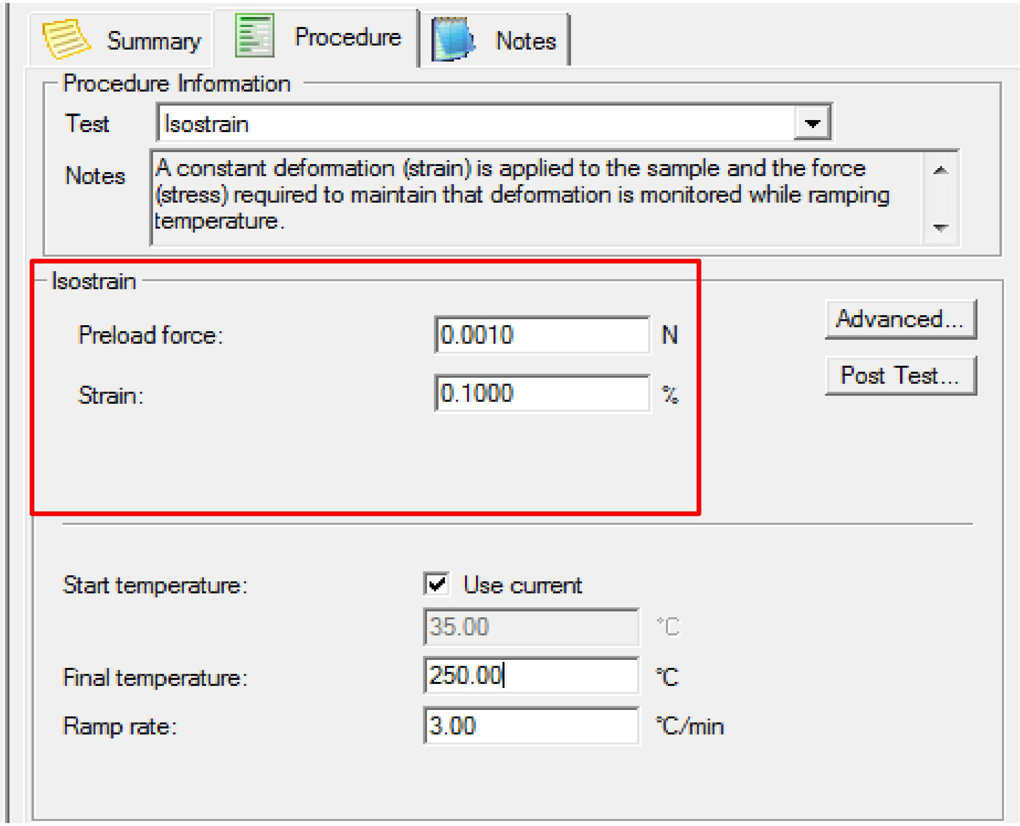


- DMA Iso-stress
- Hold stress constant and measure sample dimension change

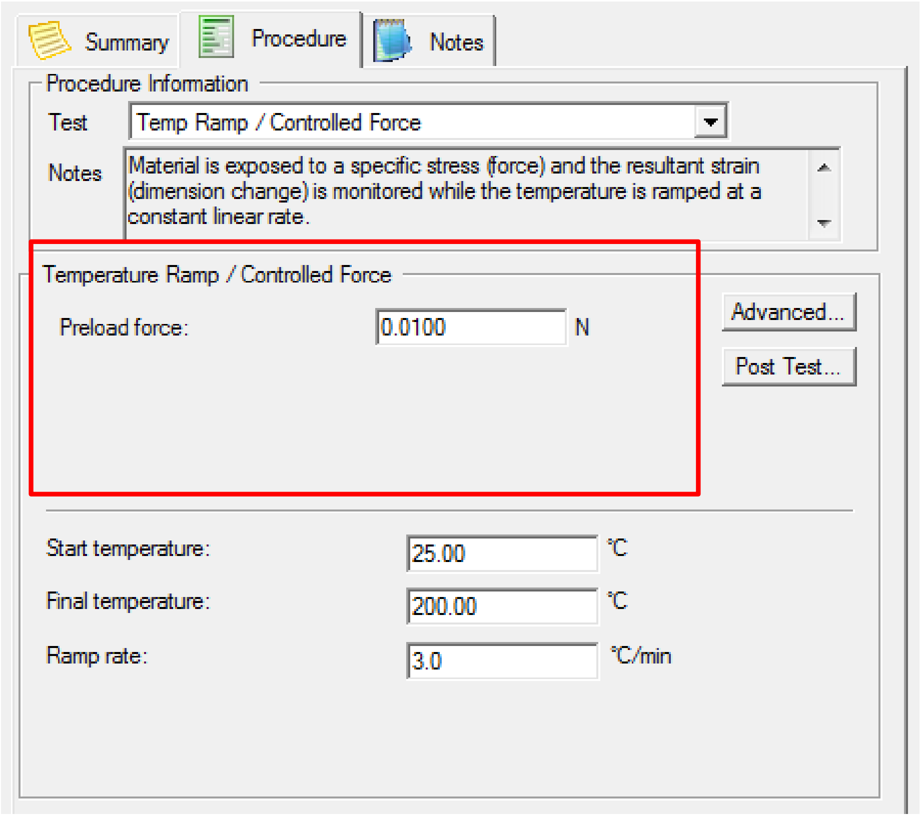


Iso-Strain/Iso-Stress on a Q800

- DMA Iso-strain
- Hold strain constant and measure sample shrinking force
- Only works with film-tension clamp on the Q800

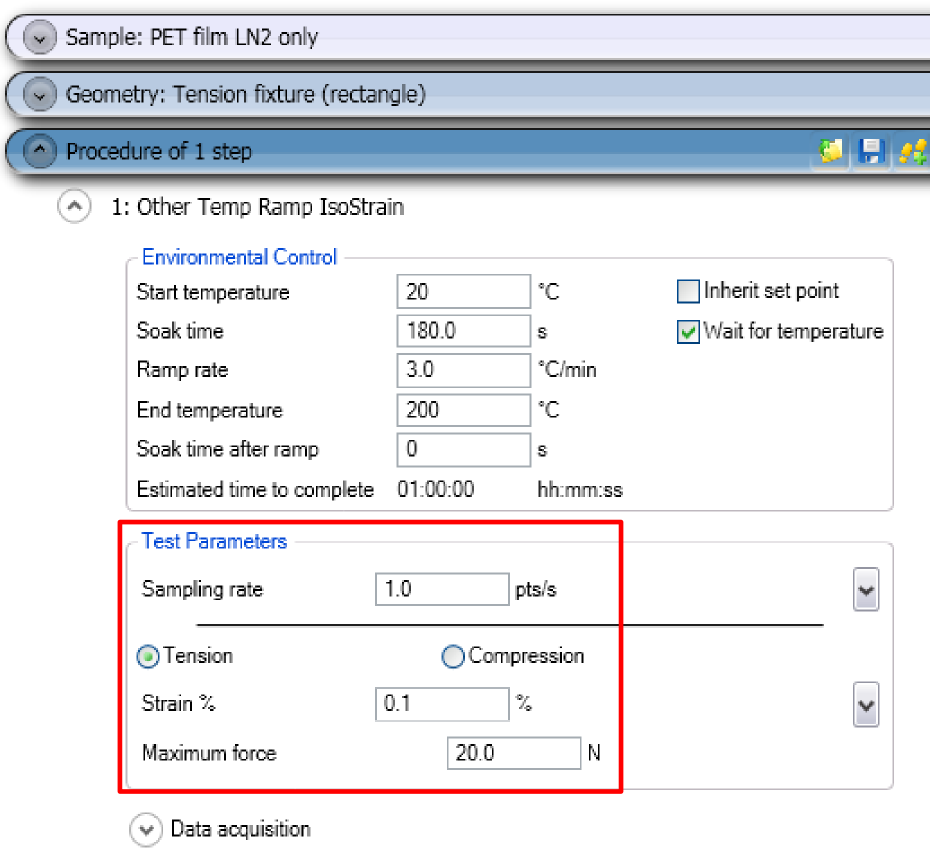


- DMA Control force
- Hold force constant and measure sample dimension change

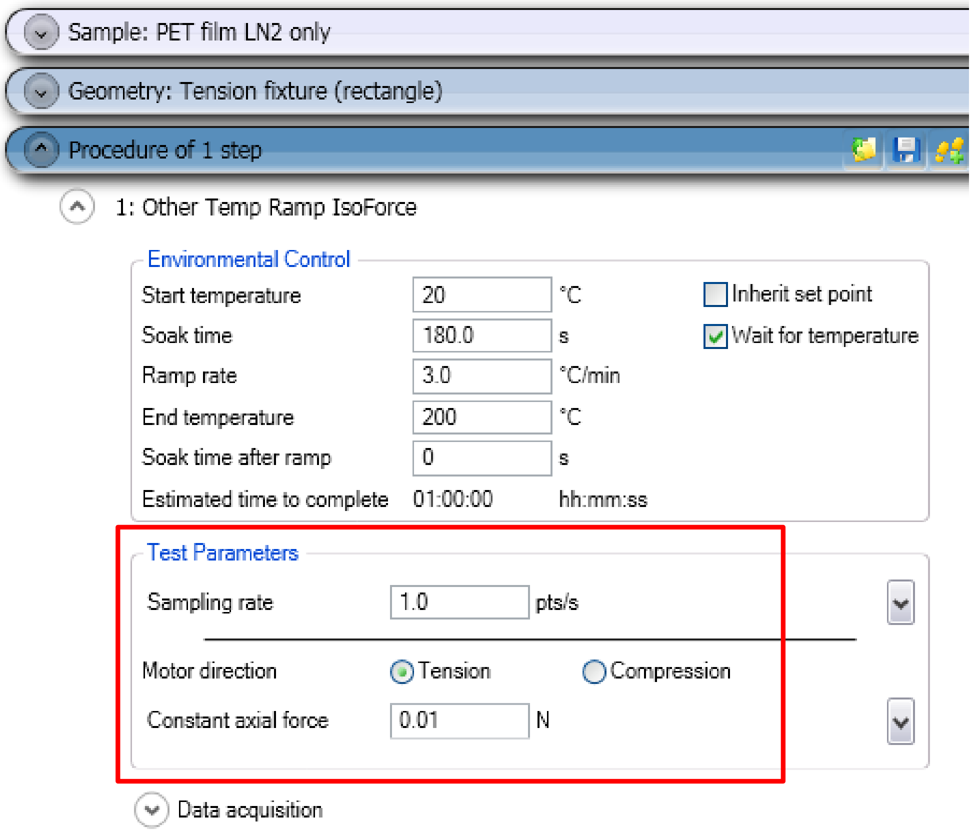


Iso-Strain/Iso-Stress on a RSA G2

- DMA Iso-strain
- Hold strain constant and measure sample shrinking force

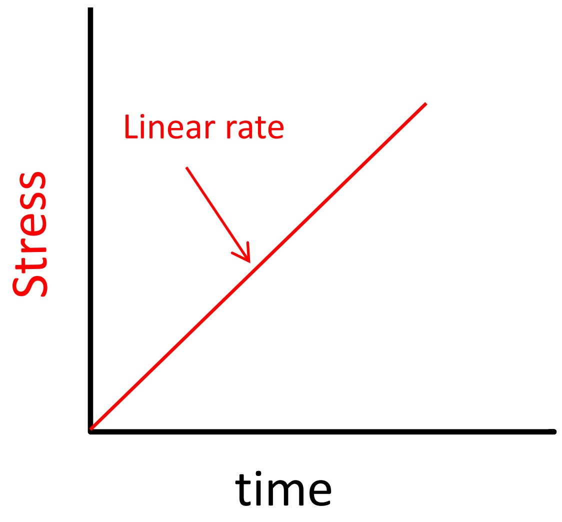


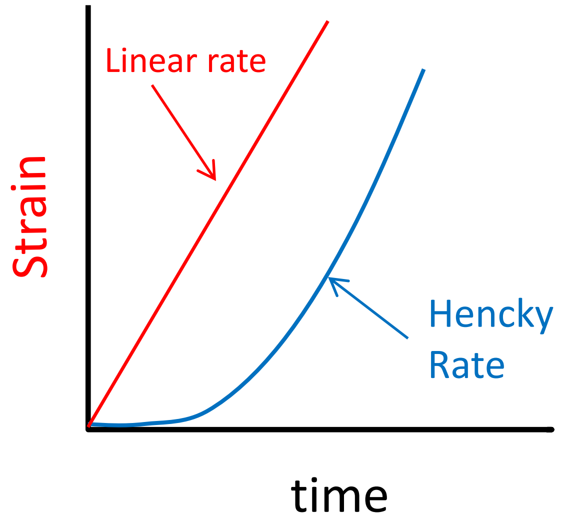
- DMA Iso-force
- Hold stress constant and measure sample dimension change



Stress-Strain Testing

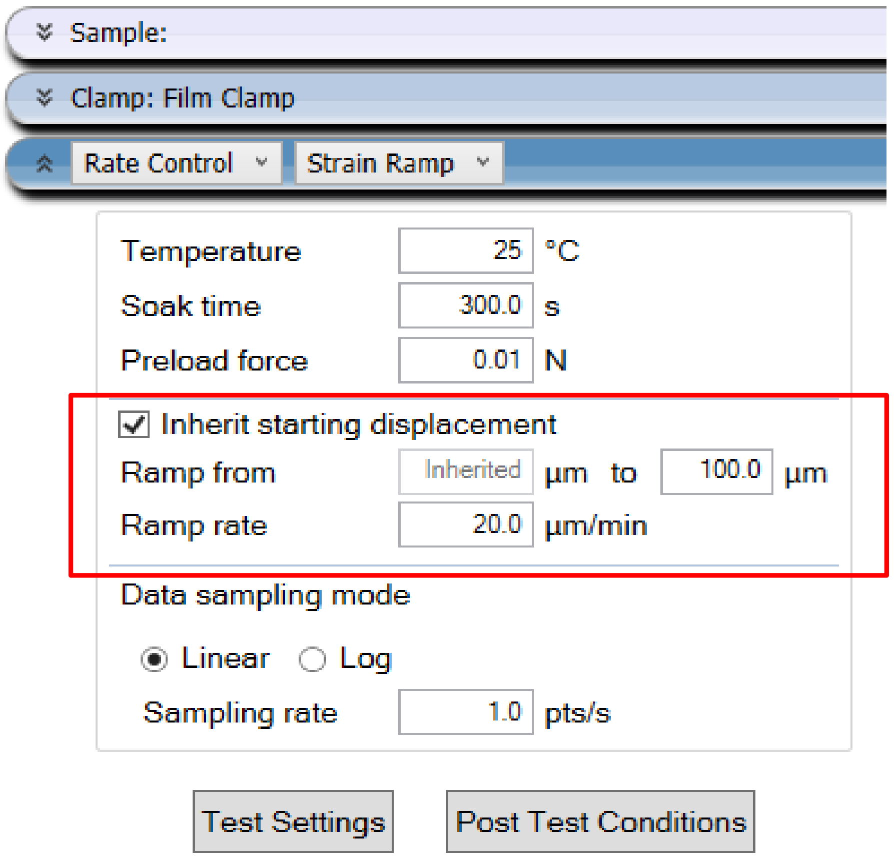
- Sample is deformed under a constant linear strain rate, Hencky strain rate, force, or stress for generating more traditional stress-strain curves.
- Measure sample's Young's modulus, yield stress, strain hardening effect and sample fracture



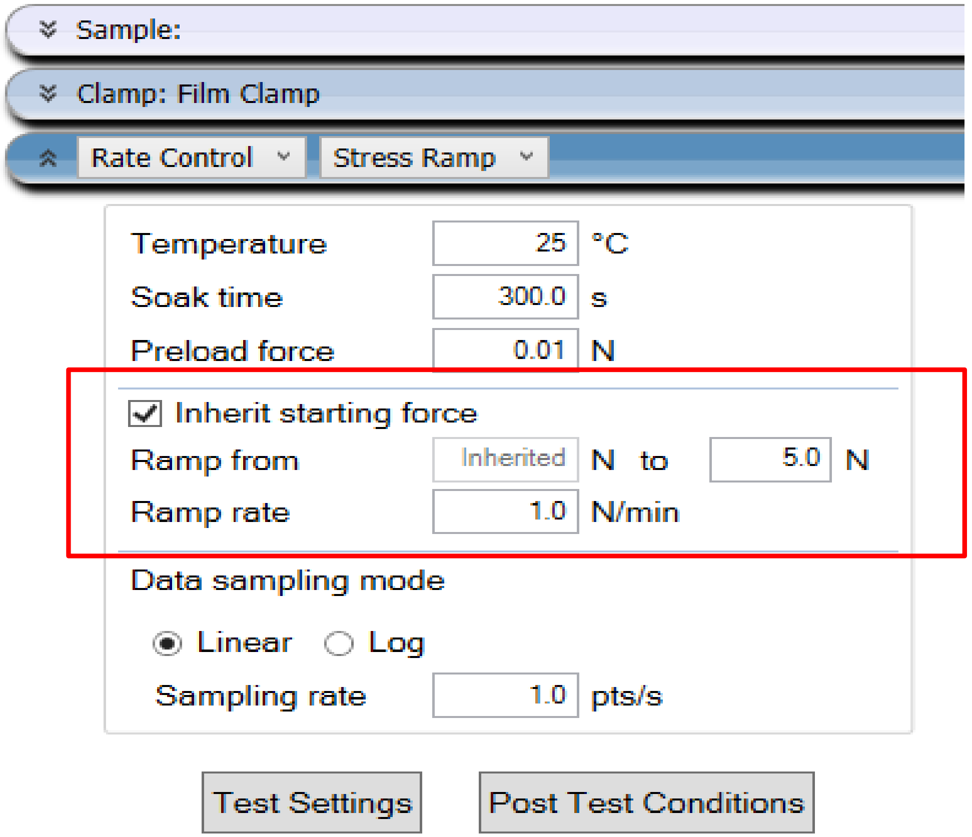


Strain/Stress Ramp on a DMA850

Strain Ramp

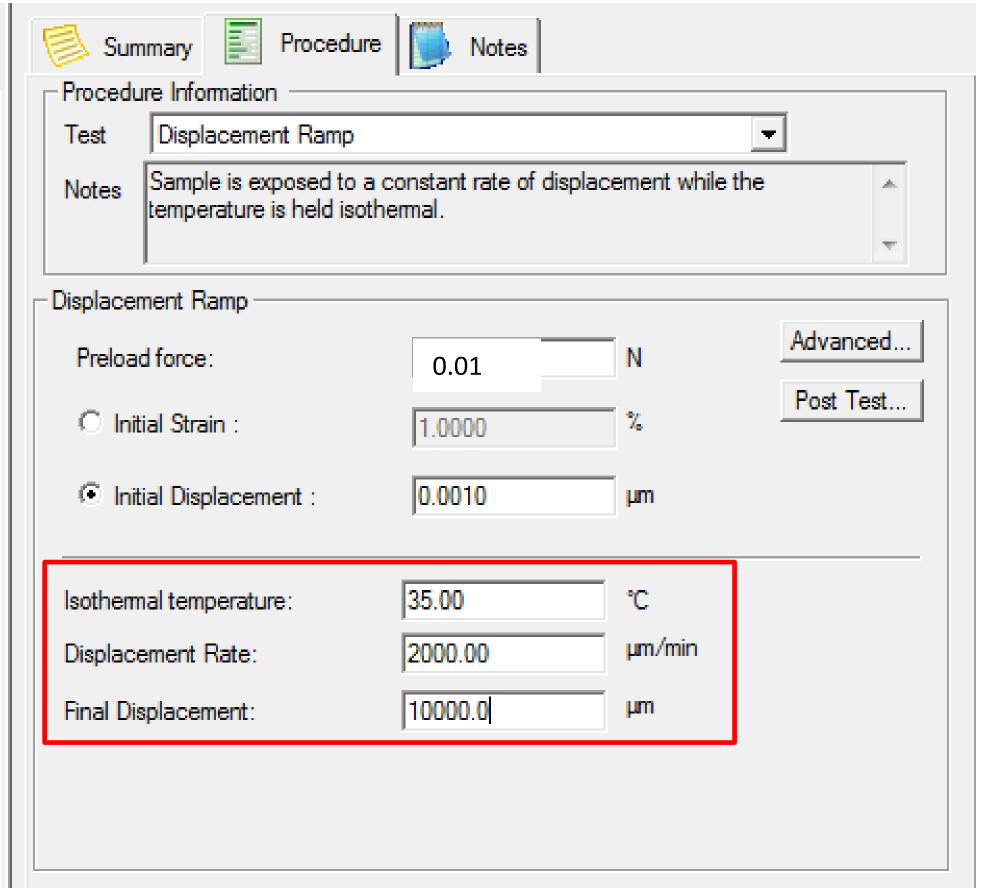


Stress Ramp

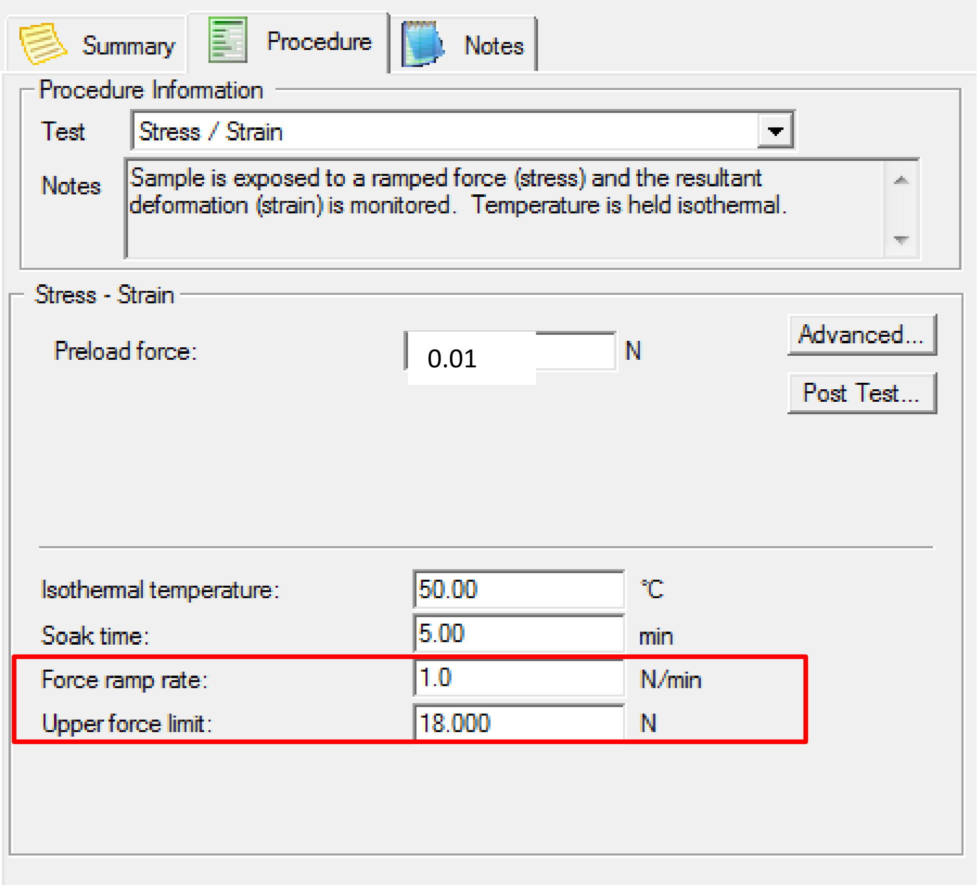


Strain/Stress Ramp on a Q800

- DMA strain rate mode
- Strain ramp
- Displacement ramp

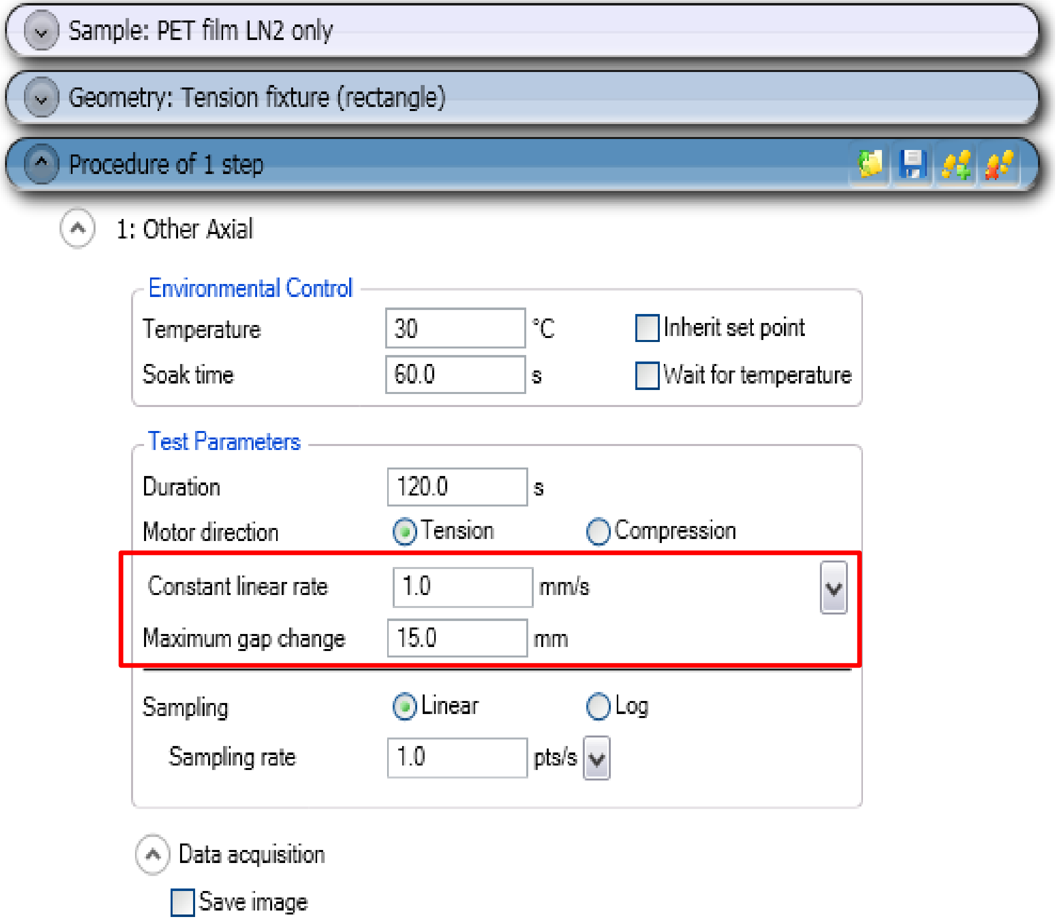


- DMA control force mode
- Force ramp

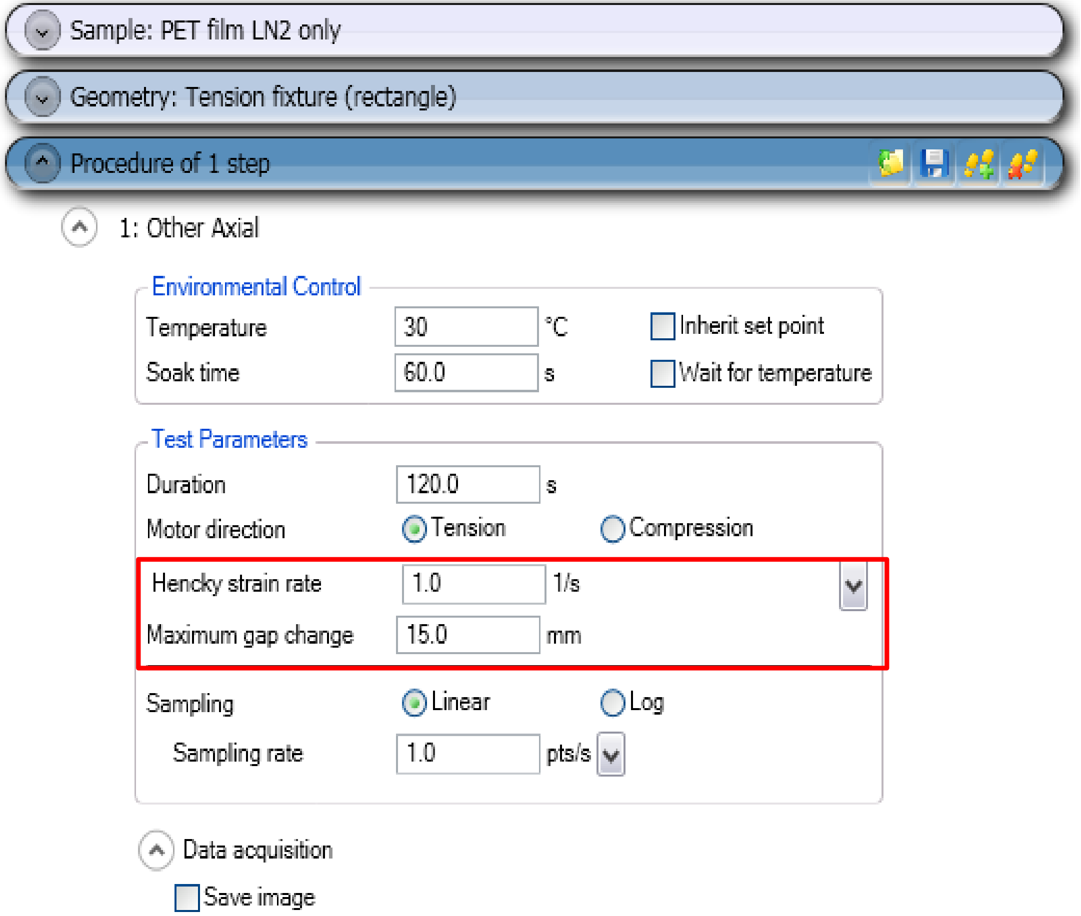


Strain Ramp on a RSA G2

- Linear strain rate

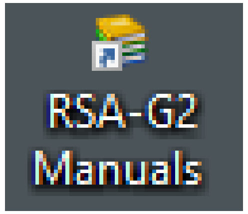


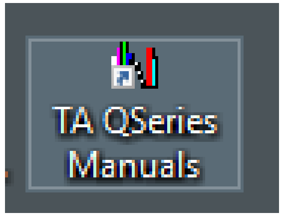
- Hencky strain rate



Additional resources for experimental setup

Getting Started Manuals on your desktop





TA Instruments

Discovery DMA 850 Manuals

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

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

Instrument Documentation

Discovery DMA 850 Getting Started Guide

Accessory Documentation

Air Chiller System (ACS) Getting Started Guide
DMA-RH Accessory Getting Started Guide
Gas Cooling Accessory (GCS) Getting Started Guide
Nitrogen Purge Cooler (NPC) Getting Started Guide

Software Documentation

What's New in TRIOS Software
Installing TRIOS Software

Site Preparation Guides and Installation Requirements

Discovery DMA 850 Site Preparation Guide

Additional Information

DMA Clamping Factors for Compression Clamps

Issued February 2020

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RSA-G2 Manuals

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

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

Instrument & Accessory Documentation

RSA-G2 Getting Started Guide
RSA-G2 FCO Camera Kit Installation Guide
RSA-G2 LN2 Kit Installation Guide
RSA-G2 Chiller Panel Kit Installation Instructions
RSA-G2 Dielectric Accessory Getting Started Guide
ACS Getting Started Guide - UPDATED!

Software Documentation

What's New in TRIOS Software
Installing TRIOS Software
Configuring a New Geometry in TRIOS Software

Miscellaneous Documentation

RSA-G2 Site Preparation Guide

Issued December 2018

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™

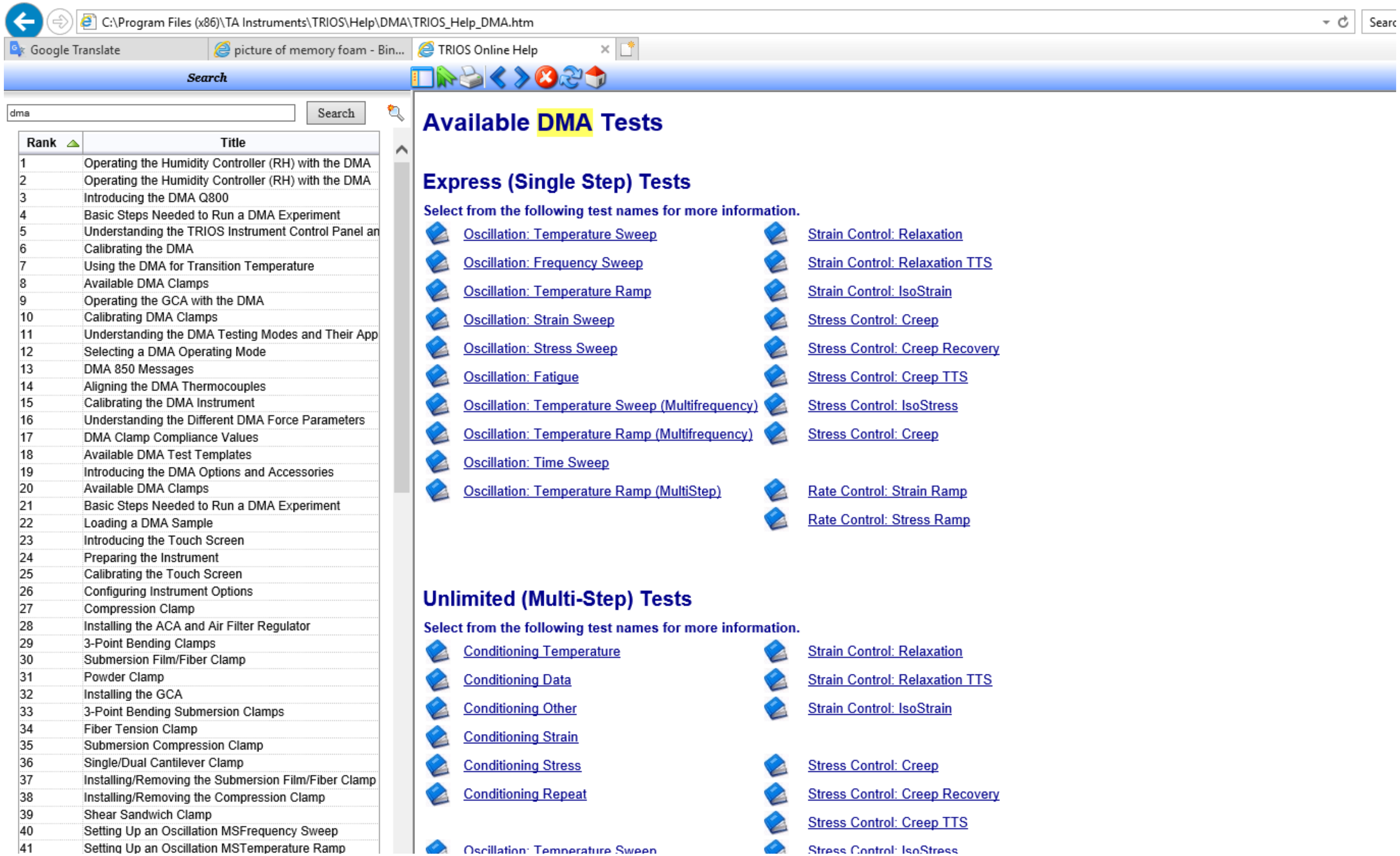
Updating Q Series™ Instrument Software

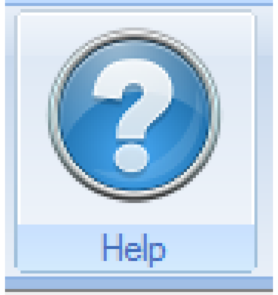
New Features in Advantage Q Series™

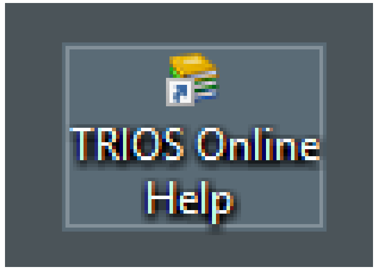
New Features in Advantage Integrity™

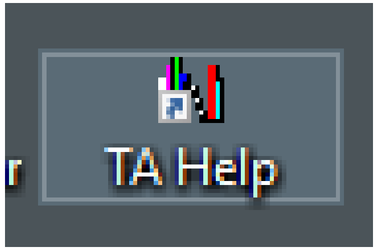
TA Update

Trios and Advantage Help



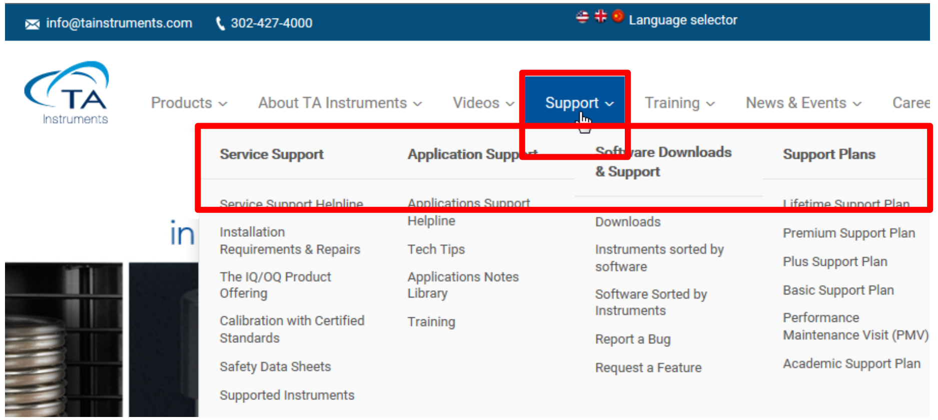


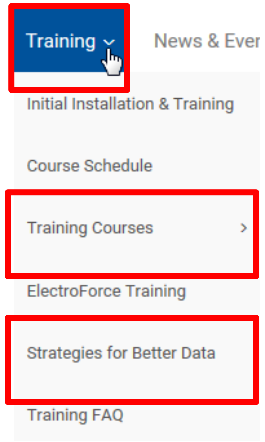




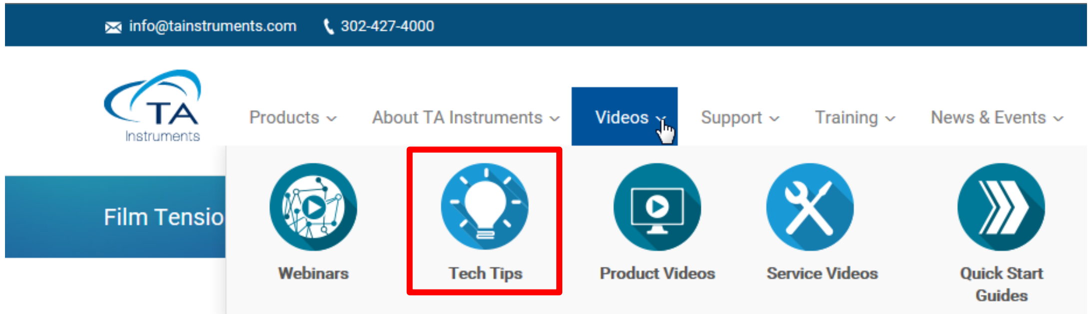
Instructional Videos

- 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

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

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Instructional Video Resources

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- For additional questions:
- Email: rheologysupport@tainstruments.com
- Please put Online Training Questions in the subject line
- Link to download the presentation: https://www.tainstruments.com/online-training-course-downloads/