



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



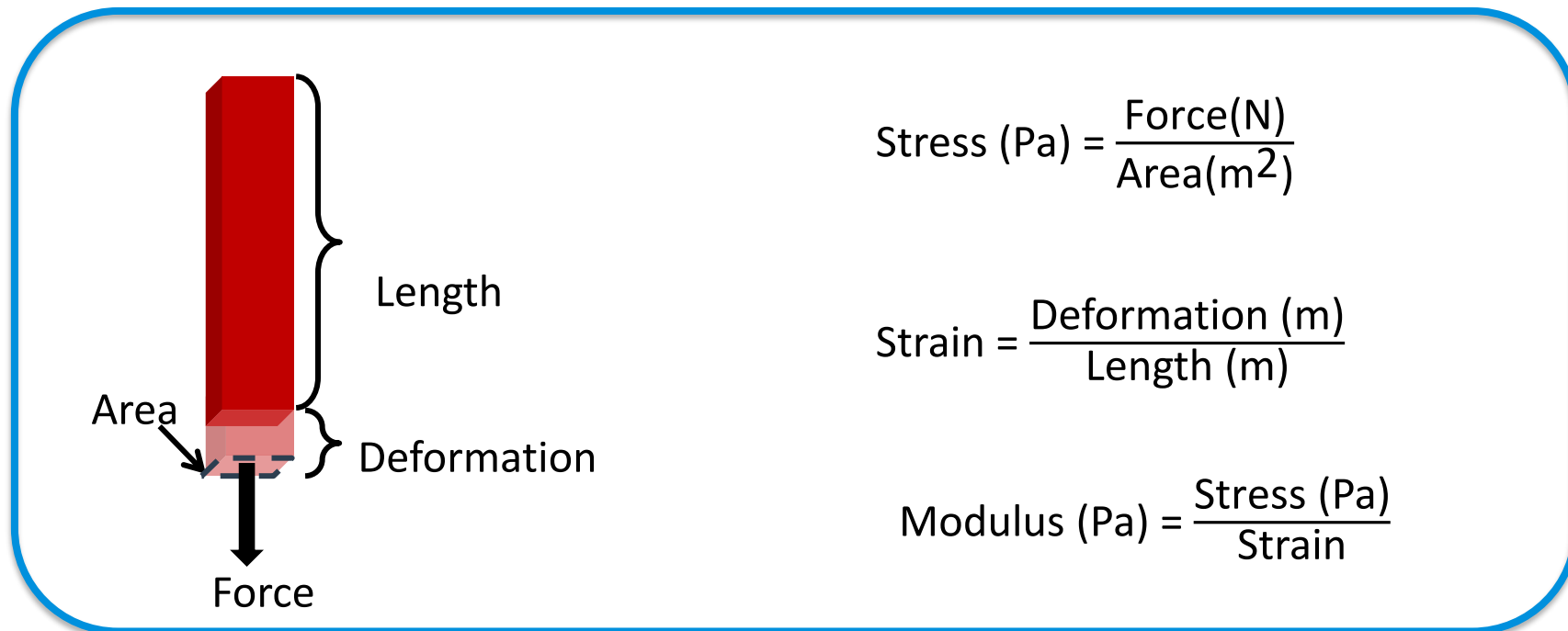
DMA definitions

- 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

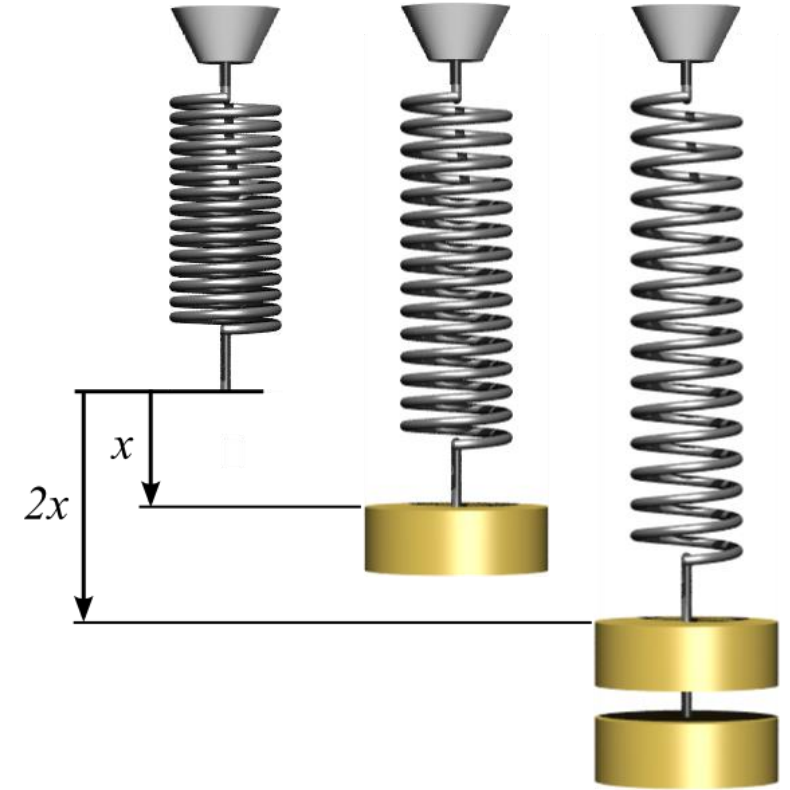
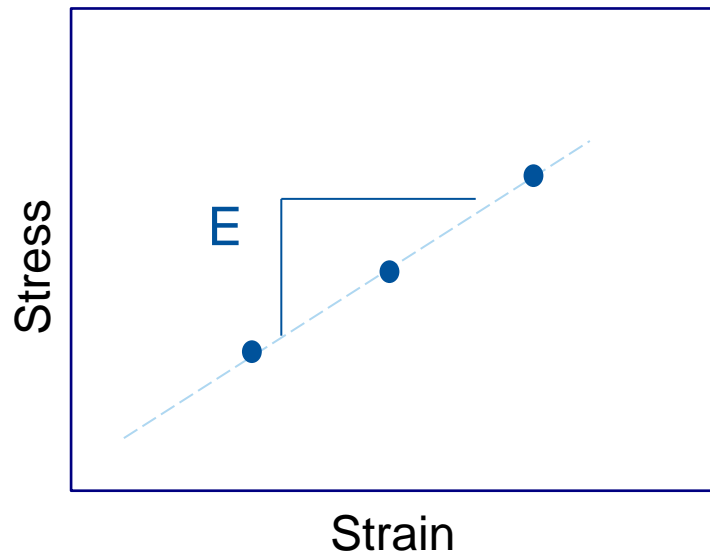
- Apply a **force** or a **deformation** to a sample, then measure sample's response, which will be a **deformation** or a **force**.
- All mechanical parameters (stress, strain, modulus, stiffness et al) are calculated from these 2 raw signals



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

$$\sigma = E \cdot \epsilon$$

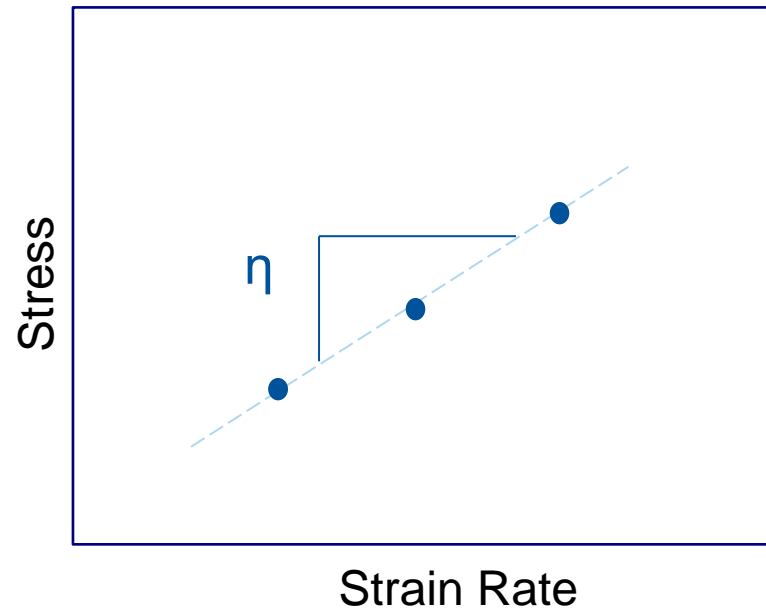


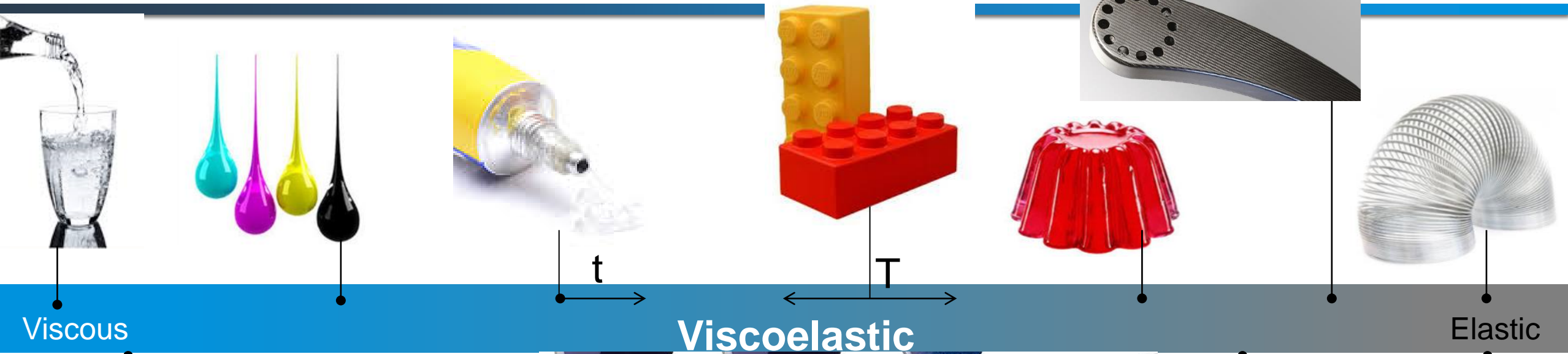
Newton's Law of Viscosity



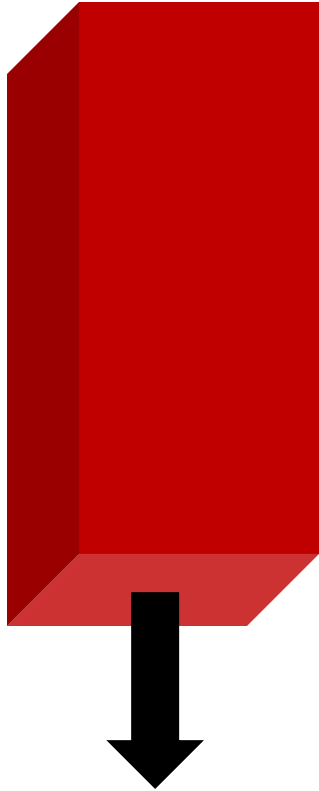
- For a purely Viscous Liquid, Stress is proportional to Strain Rate $d\varepsilon/dt$
- The slope of stress over strain rate is the viscosity of the material

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



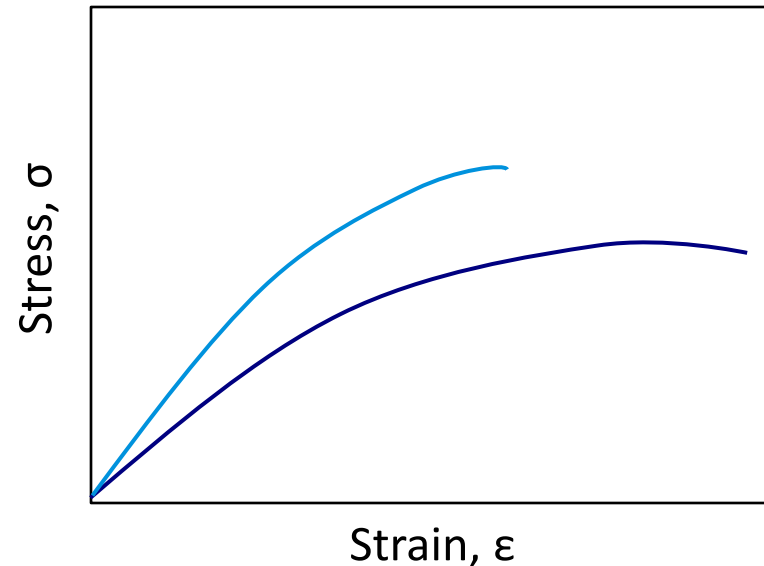


Time dependency of mechanical properties in viscoelastic materials



- 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*

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



Time-dependent viscoelastic behavior



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

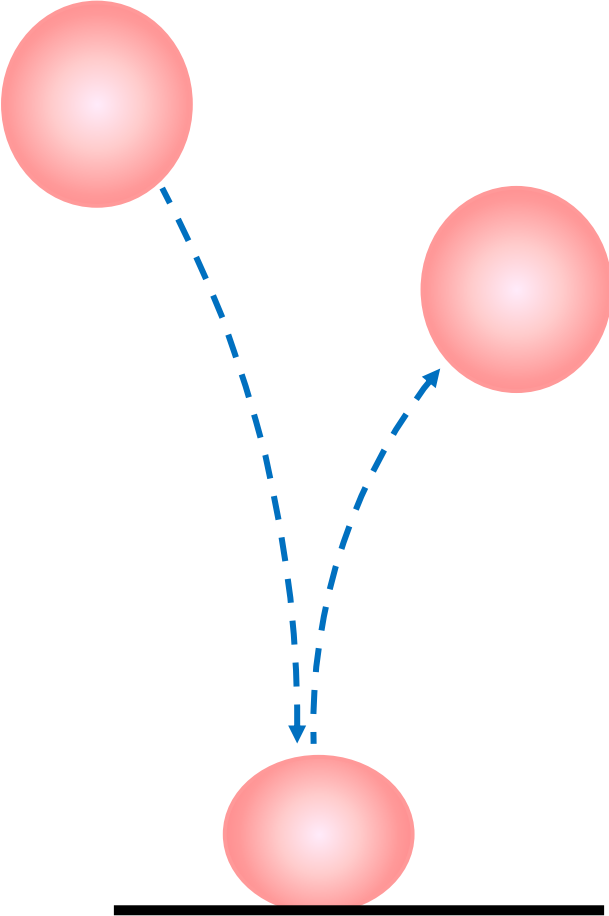


Started in 1927 by Thomas Parnell in Queensland, Australia

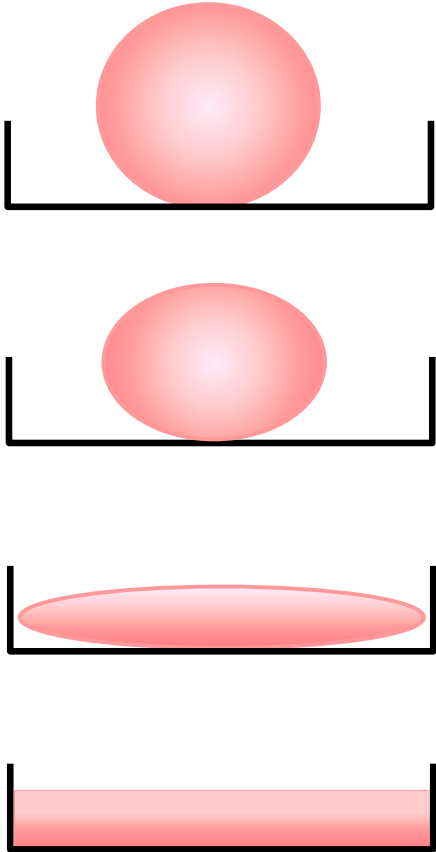
Silly putty video

- 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)



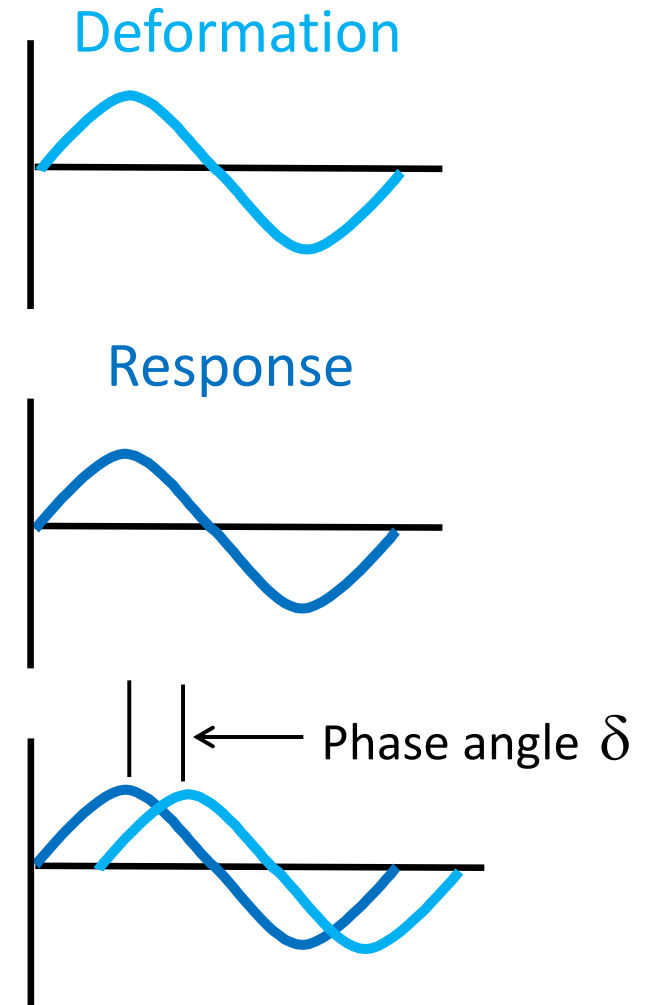
Time-period of deformation is short (high frequency)



Time-period of deformation is long (low frequency)

Dynamic Mechanical Testing

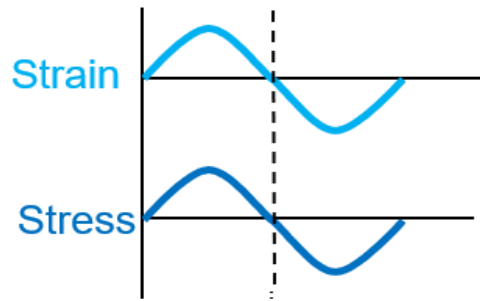
- An oscillatory (sinusoidal) deformation (stress or strain) is applied to a sample.
- The material response (strain or stress) is measured.
- The phase angle δ , or phase shift, between the deformation and response is also measured.



Phase angle response in dynamic mechanical tests

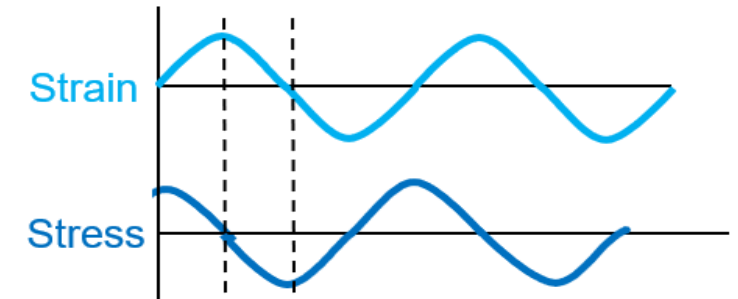
Purely Elastic Response
(Hookean Solid)

$$\delta = 0^\circ$$



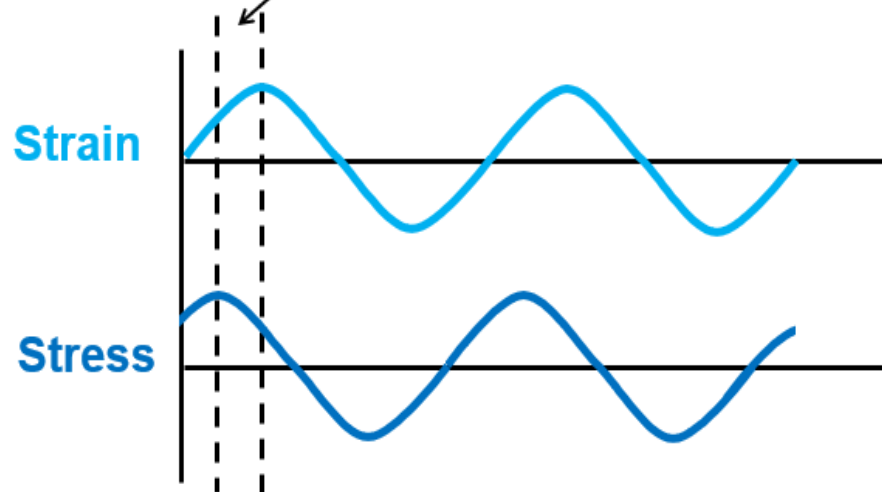
Purely Viscous Response
(Newtonian Liquid)

$$\delta = 90^\circ$$



Viscoelastic Response
(Most materials)

$$\text{Phase angle } 0^\circ < \delta < 90^\circ$$



Viscoelastic parameters obtained from DMA tests

Complex Modulus: Measure of materials overall resistance to deformation.

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

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.

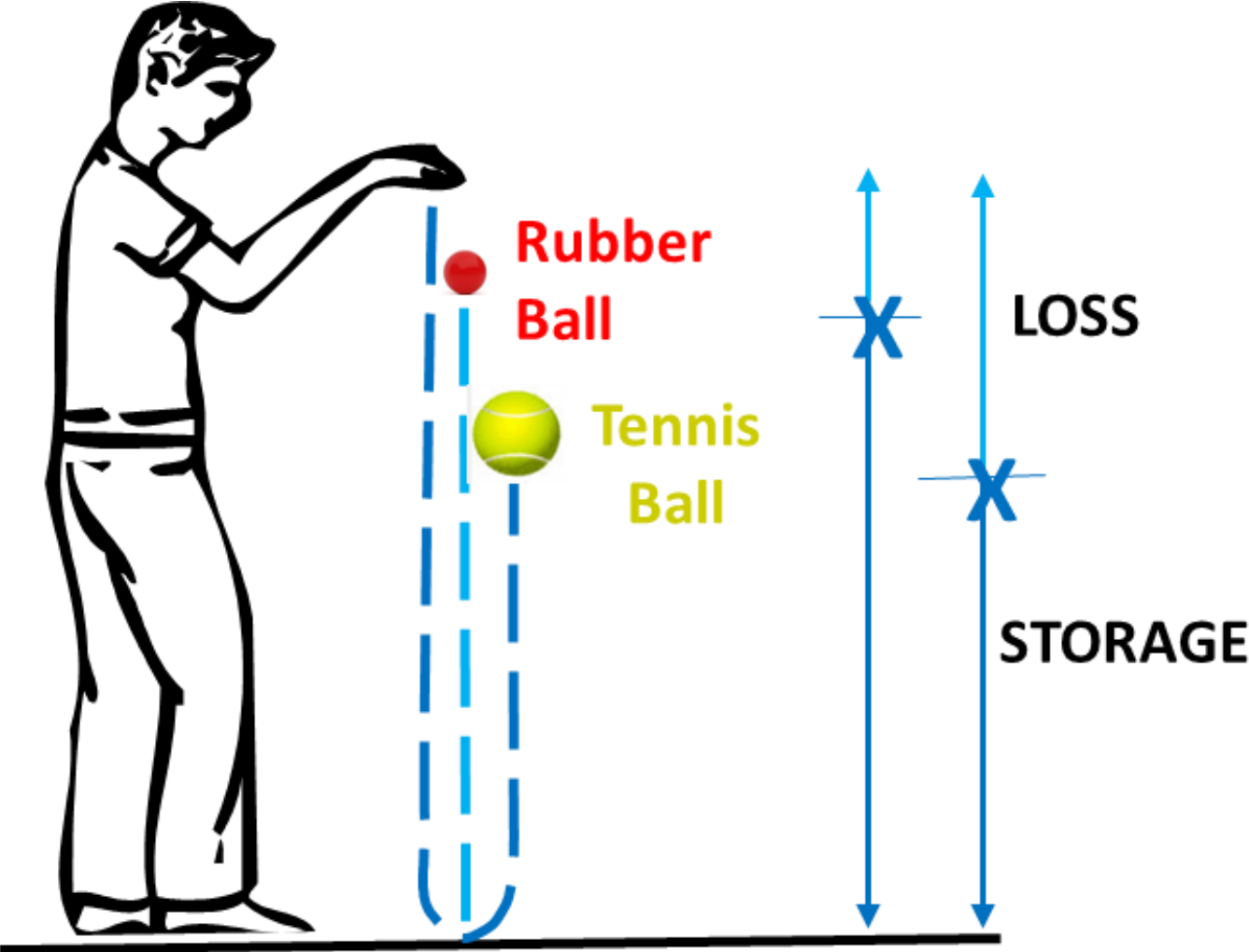
$$E'' = \left(\frac{\sigma}{\gamma} \right) \sin \delta$$

Tan Delta:

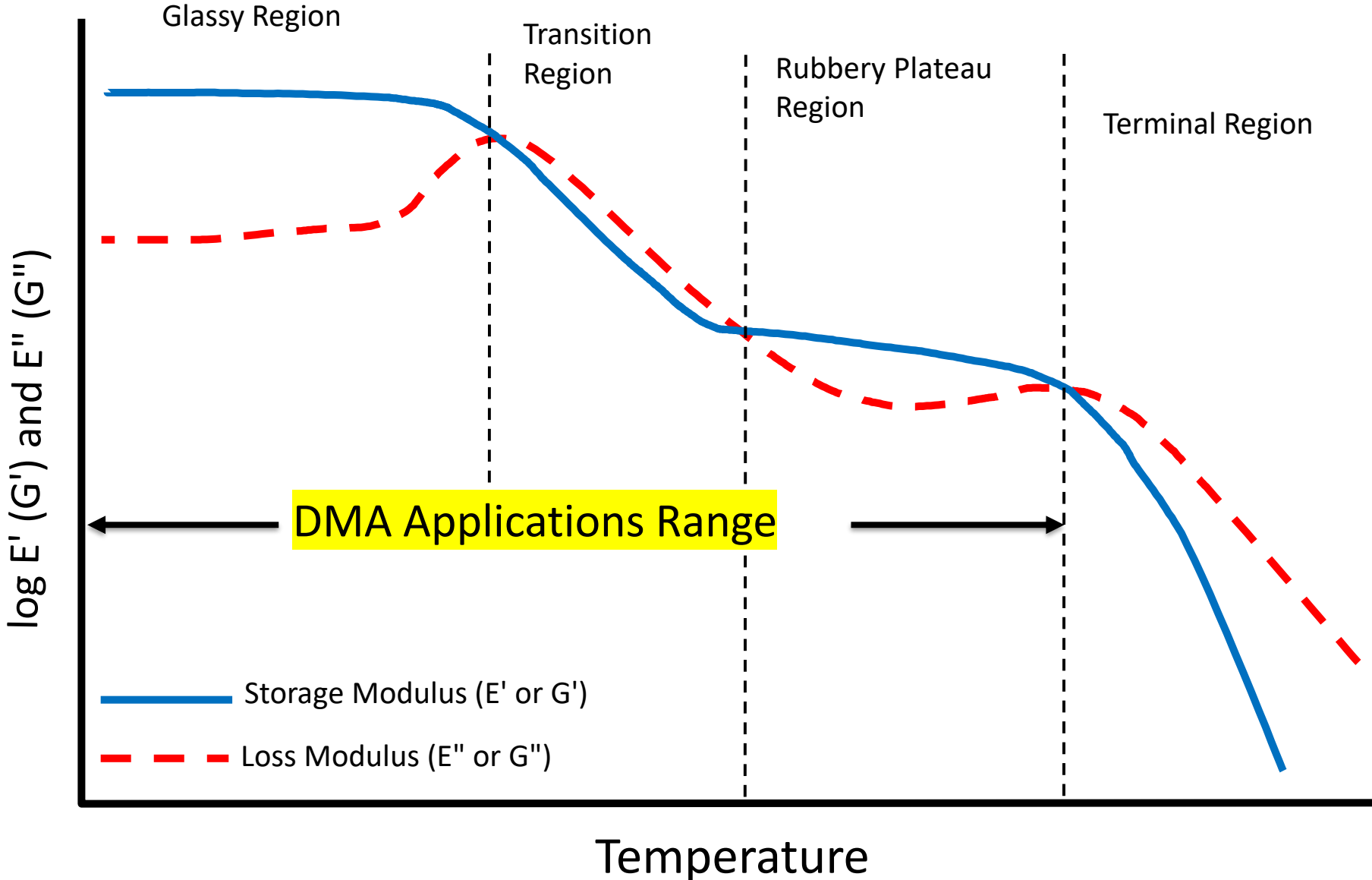
Measure of material damping. Increasing $\tan \delta$ implies a greater potential for energy dissipation and lower elasticity, and vice-versa. Measure of viscous property while having the appropriate level of stiffness.

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

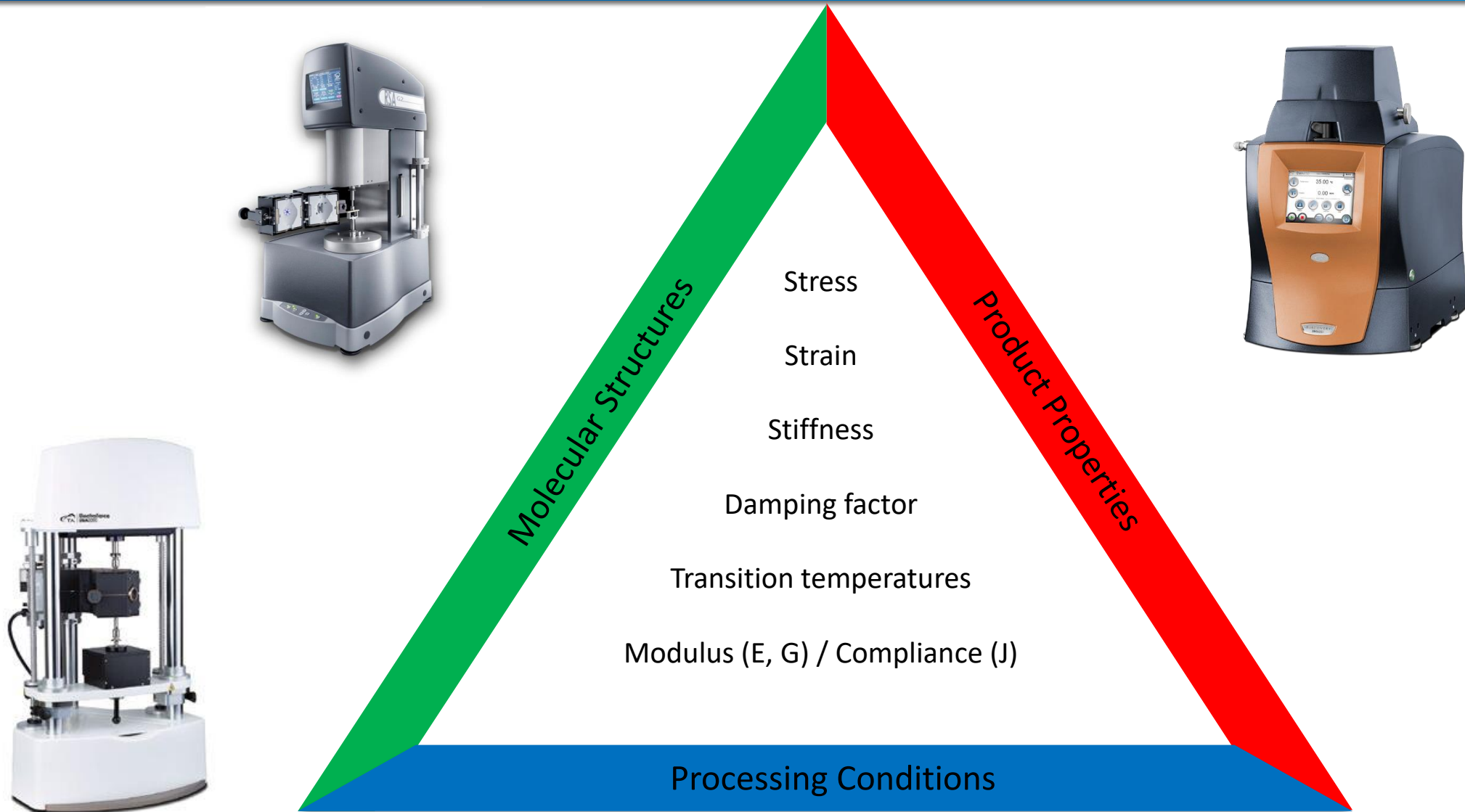
Storage and Loss of a Viscoelastic Material



Viscoelastic spectrum for a typical amorphous polymer



DMA results can correlate to.....



DMA Instrumentation and Clamps



DMA instrumentation

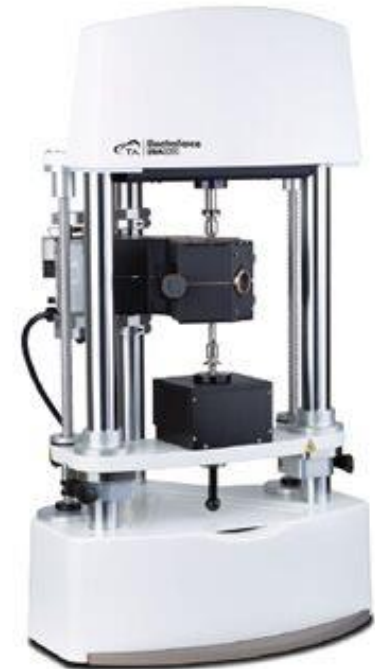
RSA G2



Discovery DMA850



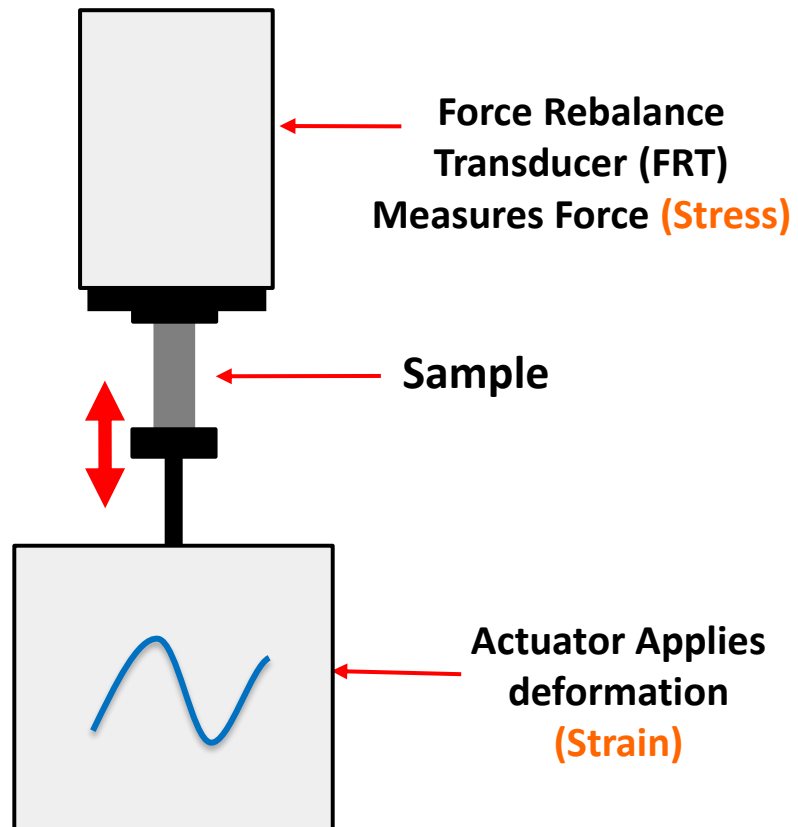
Electroforce series (high load frame, fatigue)



DMA instrumentation

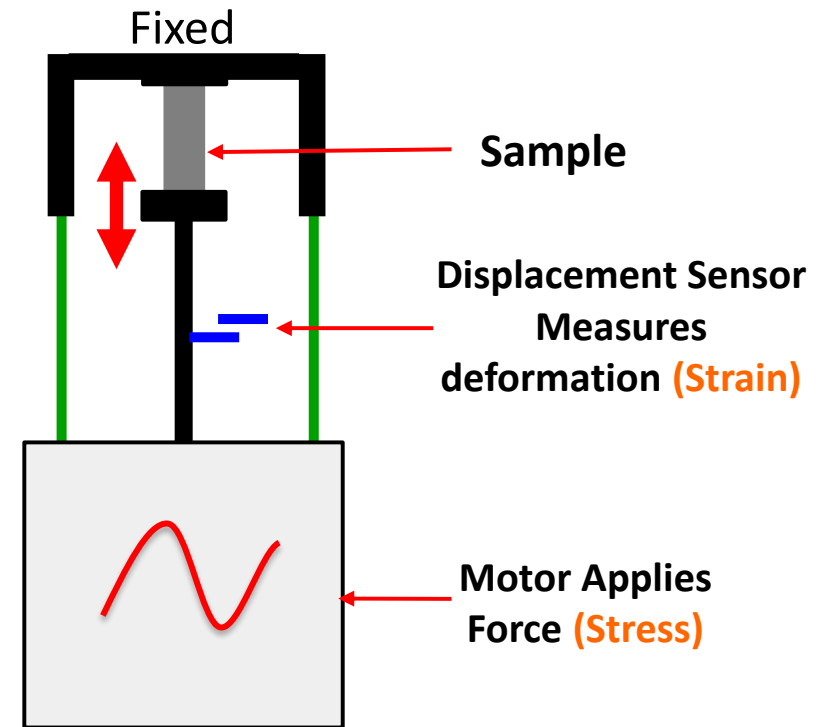
RSA G2

Separate Motor & Transducer

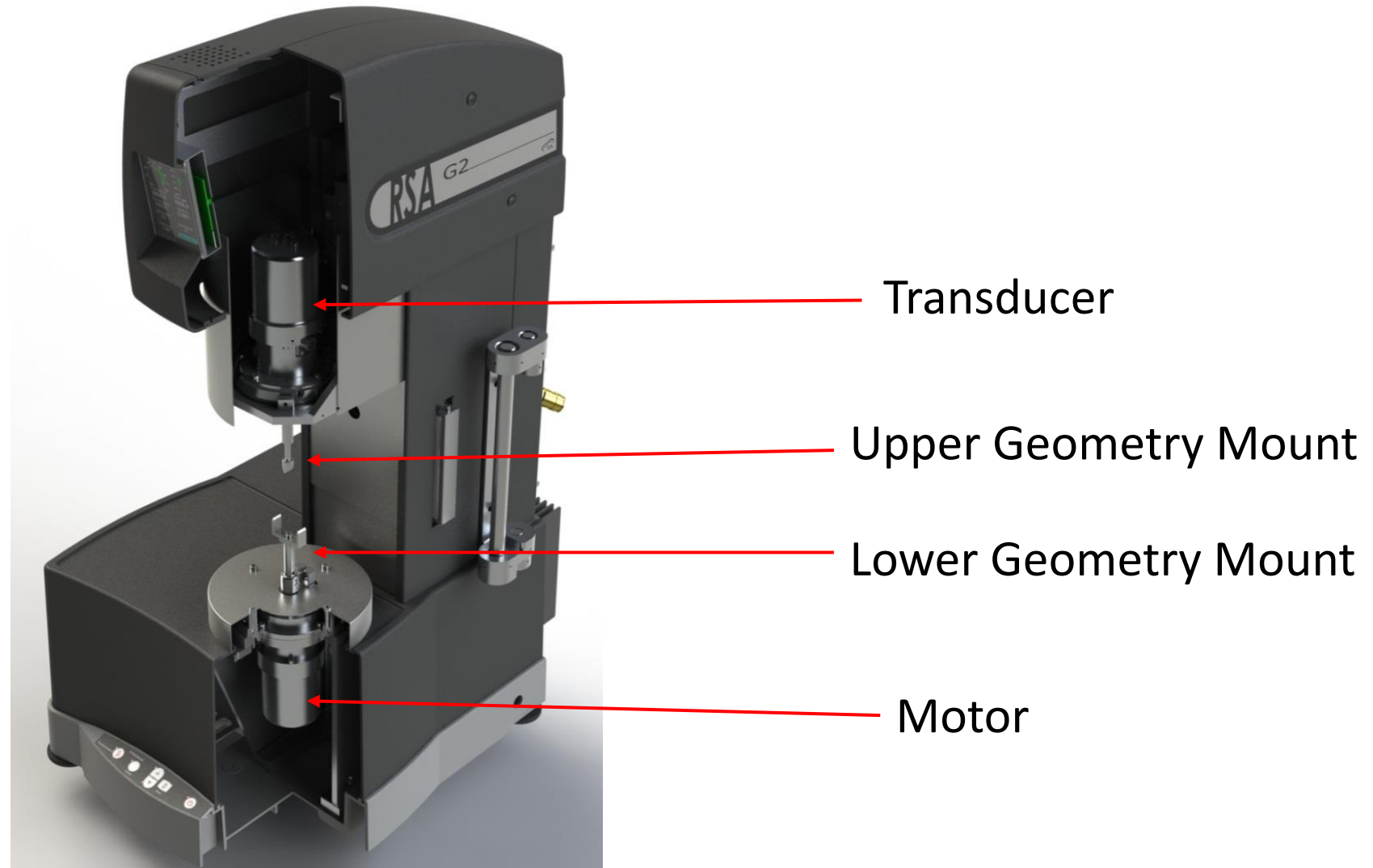


DMA850 and Q800

Combined Motor & Transducer



RSA G2: Schematic Dual Head Design



DMA850: Schematic



DMA850 and Q800: Humidity Option



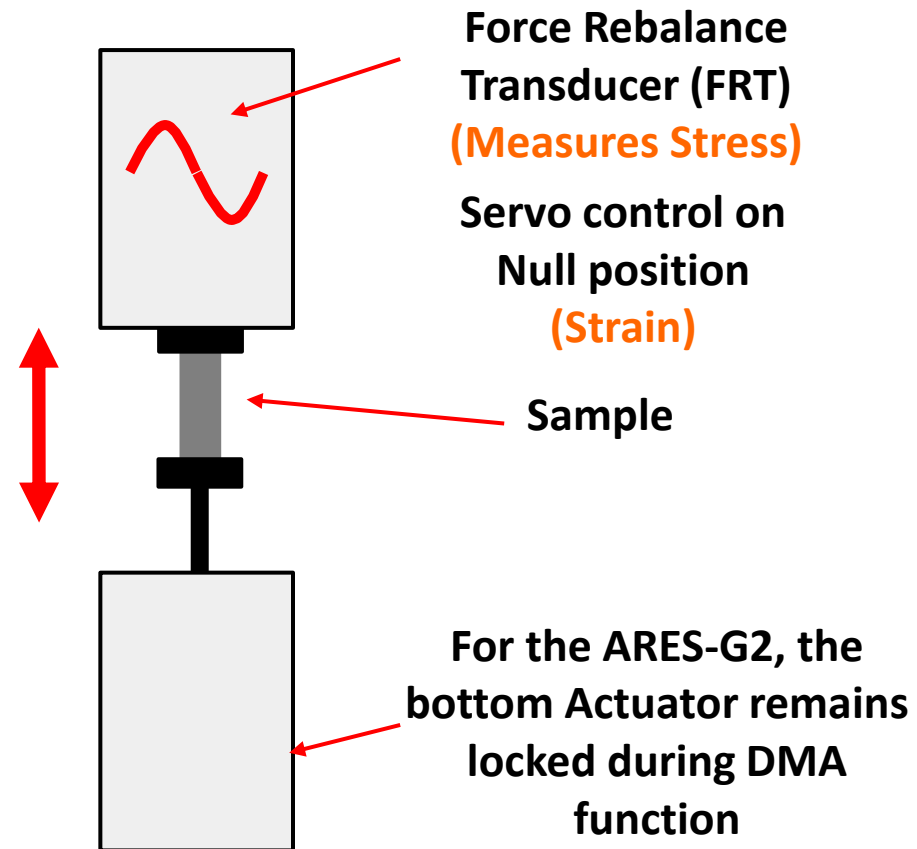
DMA Specifications

	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*

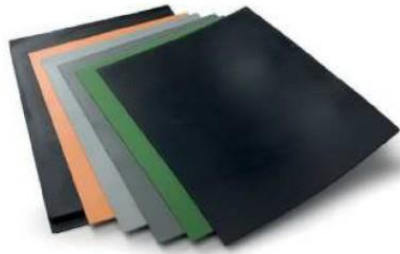


Specifications of the DHR-DMA and the ARES-G2 DMA

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 x 10 ⁻⁵ to 16	1 x 10 ⁻⁵ to 16

What samples can be measured on a DMA?

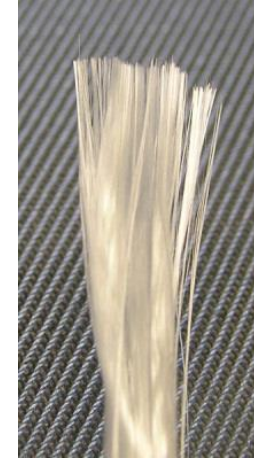
- By changing the clamp, we can test a range of different materials



Elastomers



Films



Fibers



Gels



Plastics



Foams



Composites

Clamps for DMA850 and Q800

S/D Cantilever



Film/Fiber Tension



3-Point Bending



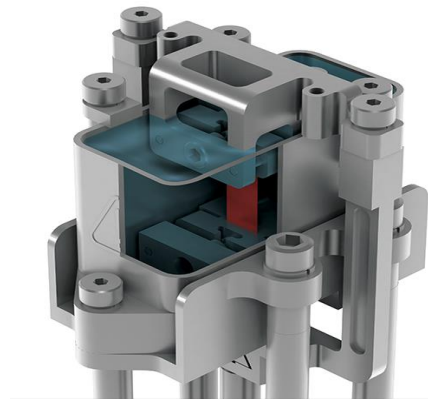
Compression



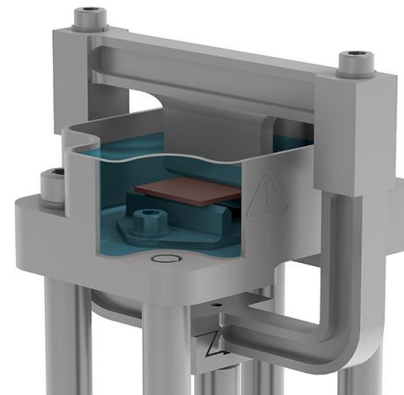
Shear Sandwich



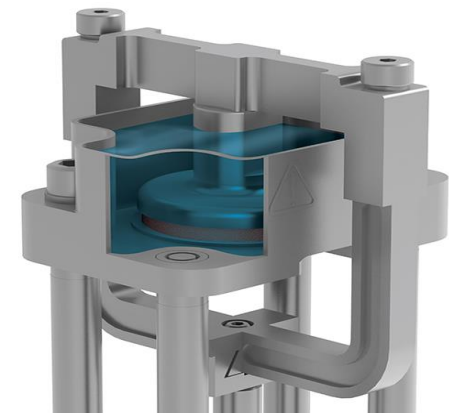
Submersible Tension



Submersible Bending



Submersible Compression

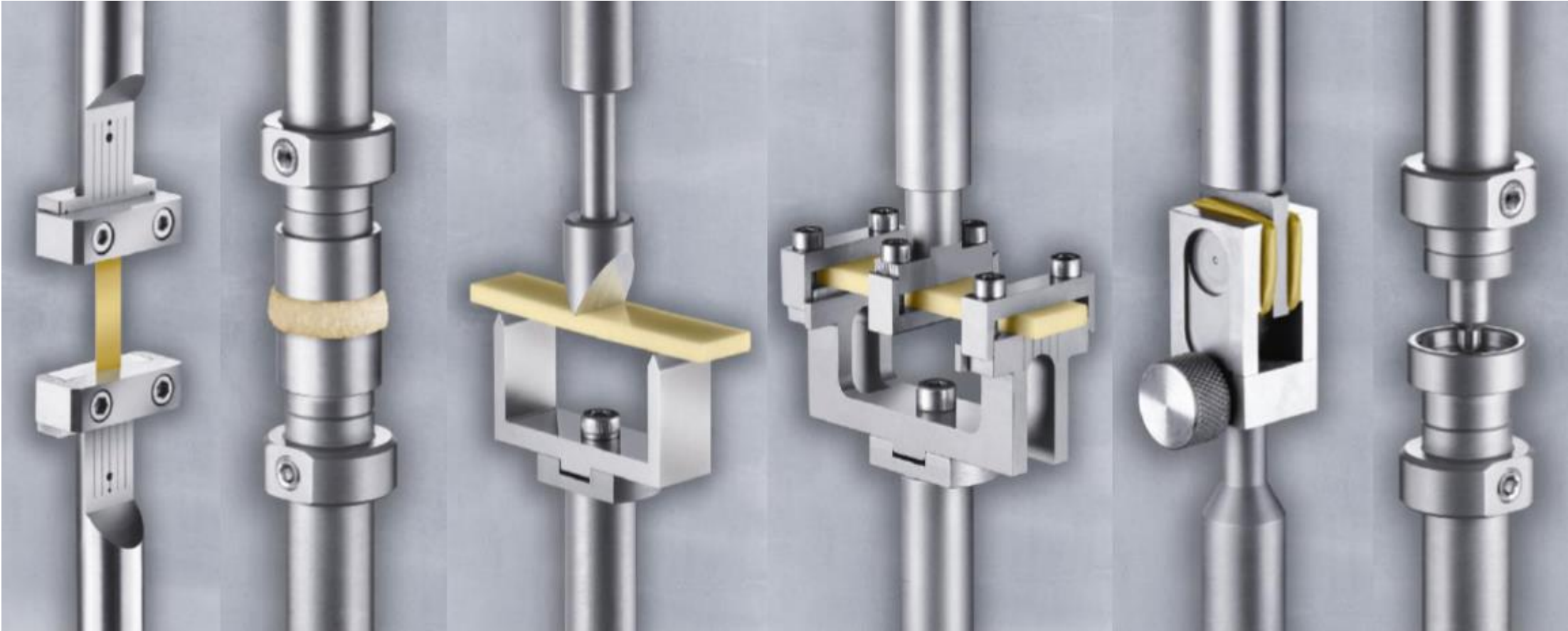


Clamps for RSA G2

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

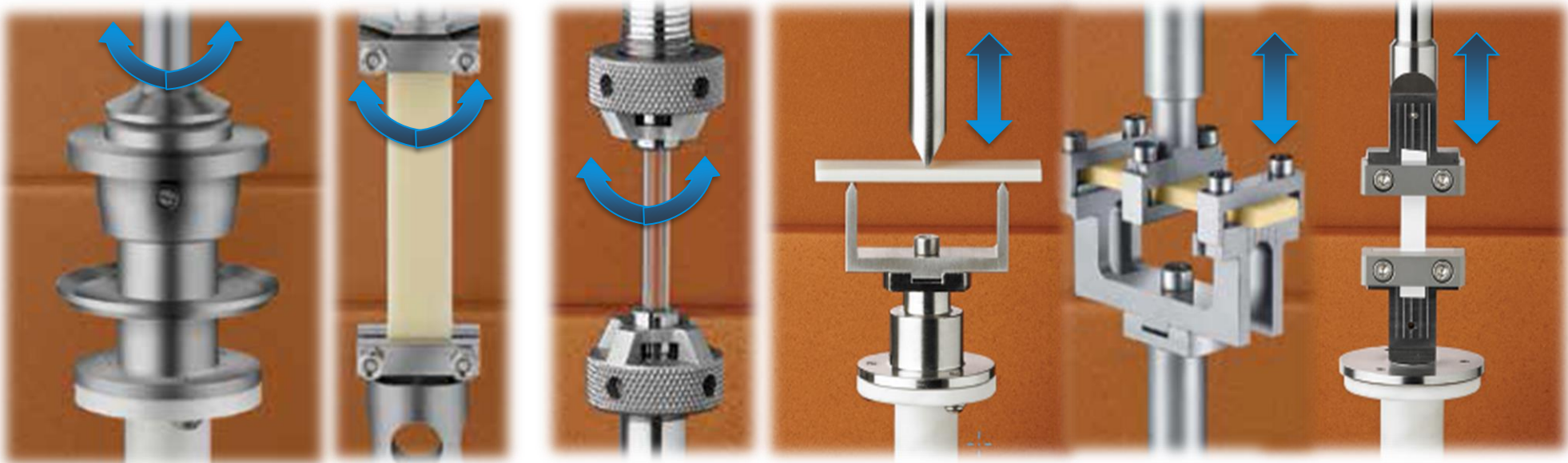
Torsion (rotational) and DMA (axial) geometries allow solid samples to be characterized in a temperature controlled environment.

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

ν : Poisson's ratio

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

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



Parallel plate

Rectangular and cylindrical torsion

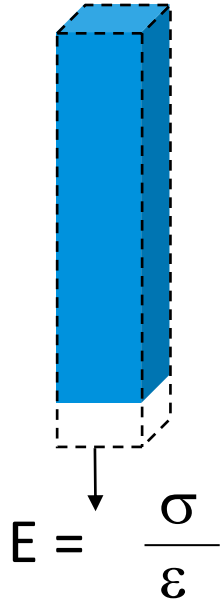
3-point bending

Cantilever

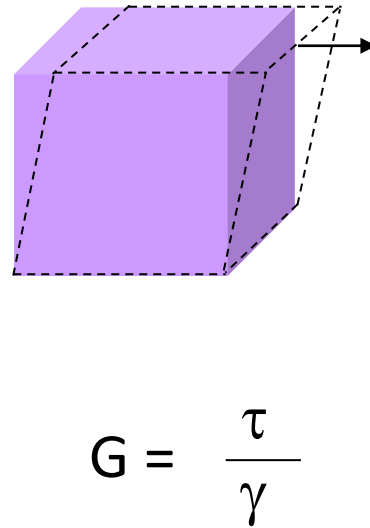
Tension

Three fundamental modes of deformation

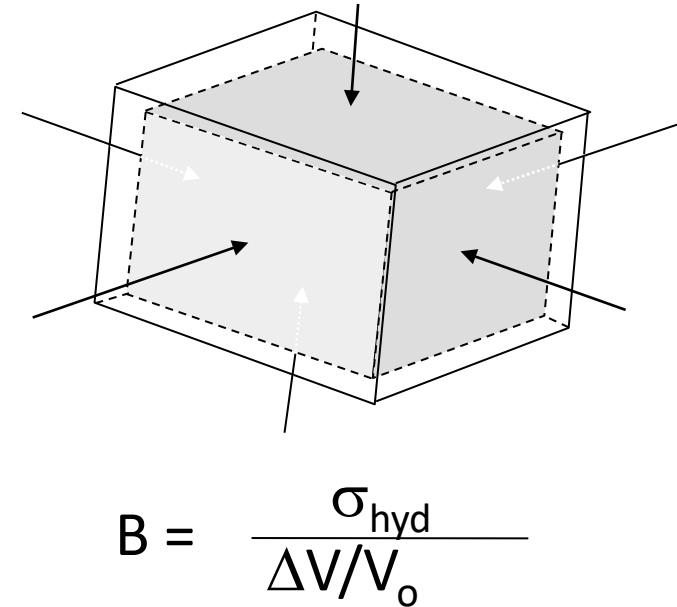
Young's Modulus



Shear Modulus



Bulk Modulus



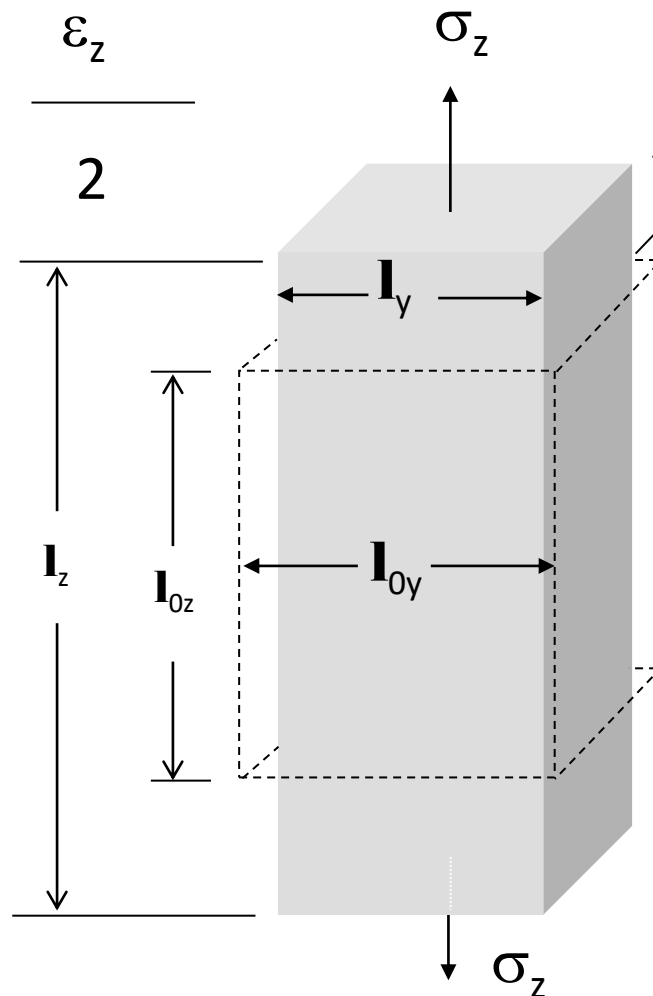
Where

- 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
- $\Delta V/V_0$ = fractional volume expansion or contraction

Poisson's Ratio

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

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



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

Poisson's Ratio

$$\nu = \frac{-\epsilon_y}{\epsilon_z}$$

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

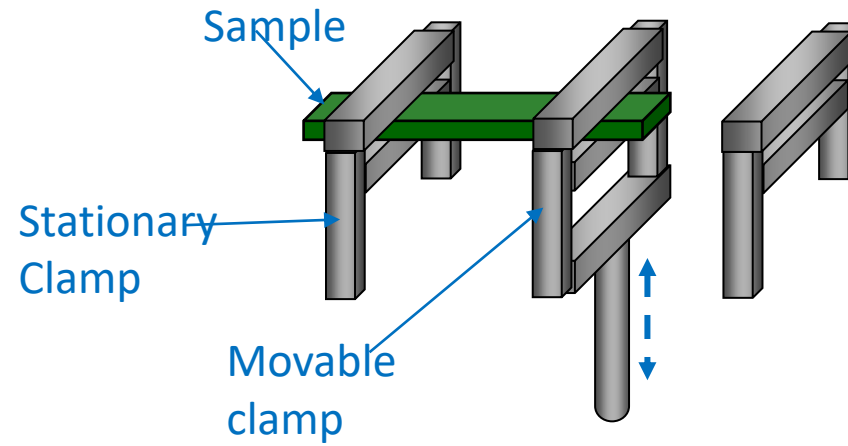
DMA850 and Q800	RSA G2
<p>Stiffness (K) = Force / Displacement</p> <p>Modulus (E) = K x GF</p> <p>GF: Geometry factor. Clamp dependent Can be found in online help manual</p>	<p>Stress (σ) = Force x K_σ</p> <p>Strain (γ) = Displacement x K_γ</p> <p>Modulus (E) = σ / γ</p> <p>K_σ : Stress constant K_γ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

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

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 ($T_g < RT$) 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 $\tan \delta$
- 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 \times Geometry Factor

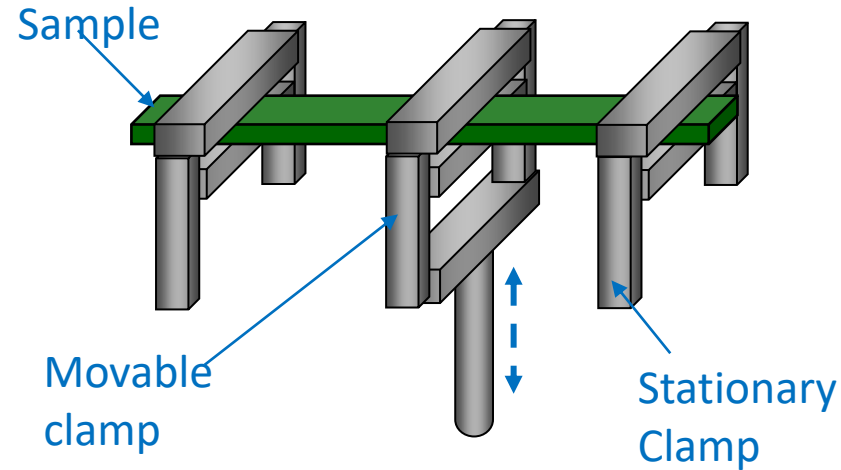
$$GF_{SC} = \frac{\cancel{12} \cdot l^3 \left[1 + \frac{12}{5} (1 + \nu) \left(\frac{t}{l} \right)^2 \right]}{\cancel{12} w t^3}$$

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

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

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 $T_g < RT$) 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 $\tan \delta$
- 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 \times Geometry Factor (GF)

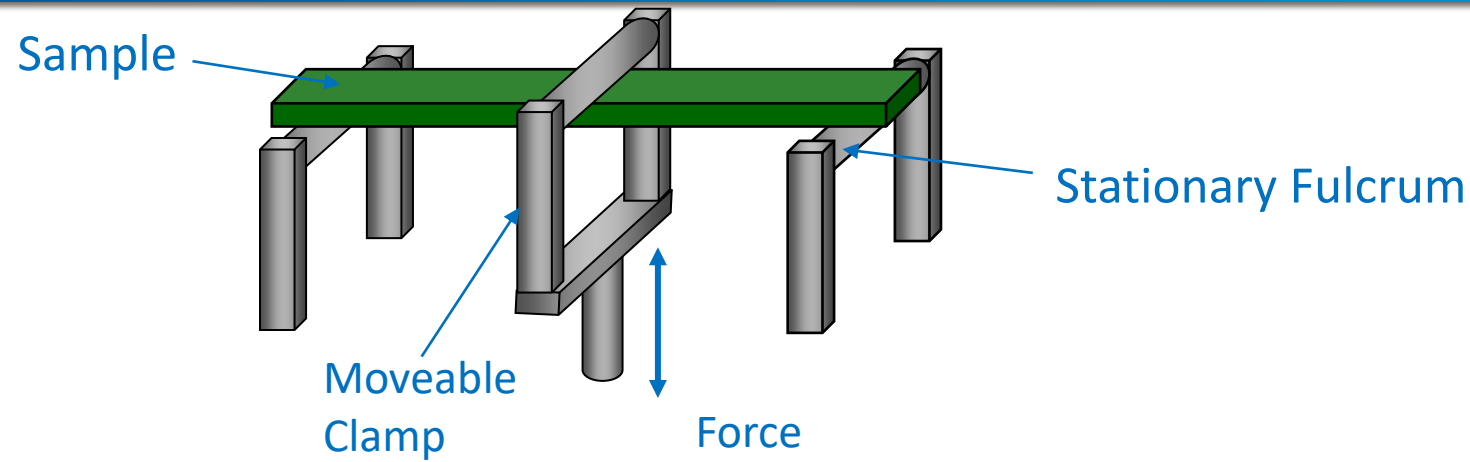
$$GF_{DC} = \frac{\cancel{12} \cdot l^3 \left[1 + \frac{\cancel{12}}{\cancel{5}} (1 + \nu) \left(\frac{\cancel{t}}{\cancel{l}} \right)^2 \right]}{\cancel{24} w t^3 \cancel{2}}$$

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

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

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 $\tan \delta$
- 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

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

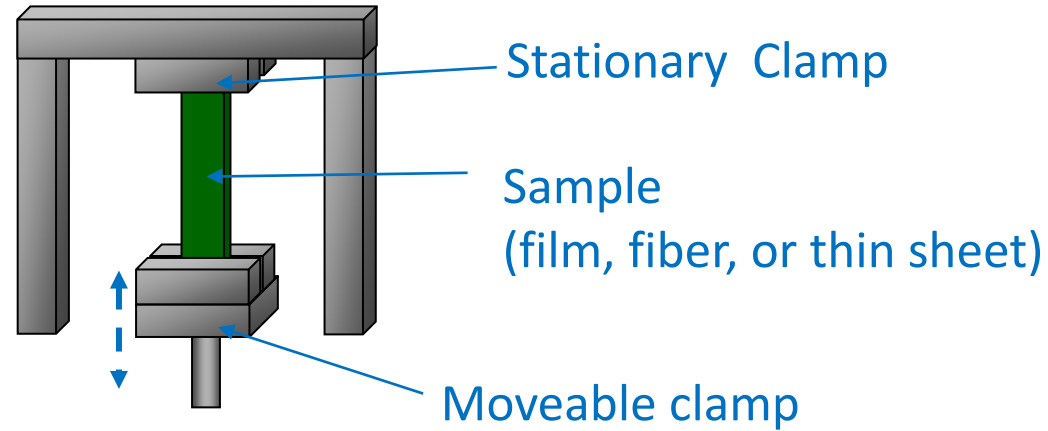
4

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

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

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, T_g , secondary transitions (modulus and $\tan \delta$)
 - 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)

Modulus = Stiffness × Geometry Factor

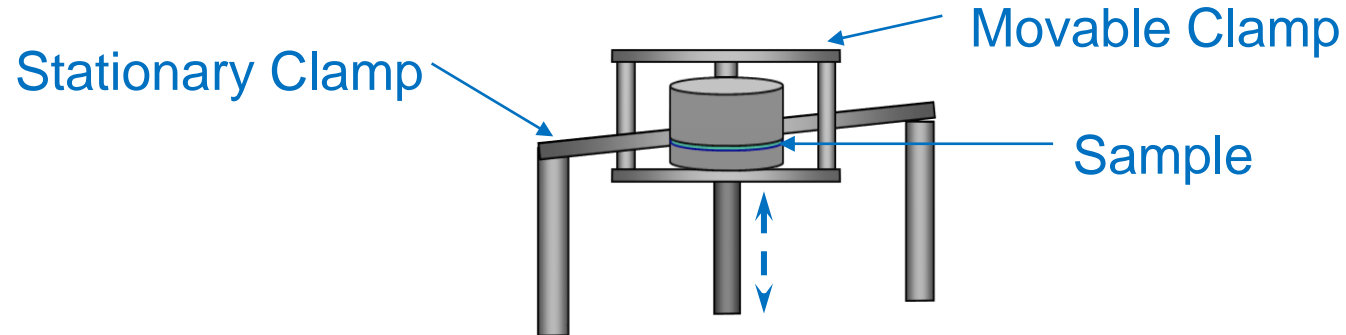
$$GF_{\text{Film}} = \frac{l}{wt}$$

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 T_g) 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 $\tan \delta$)
- Applications:
 - Mechanical properties, T_g , secondary transitions (modulus and $\tan \delta$)
 - 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 \leq plate diameter (15 mm and 40 mm options). Use exact sample diameter

Geometry Factor – Film/fiber/tension clamp

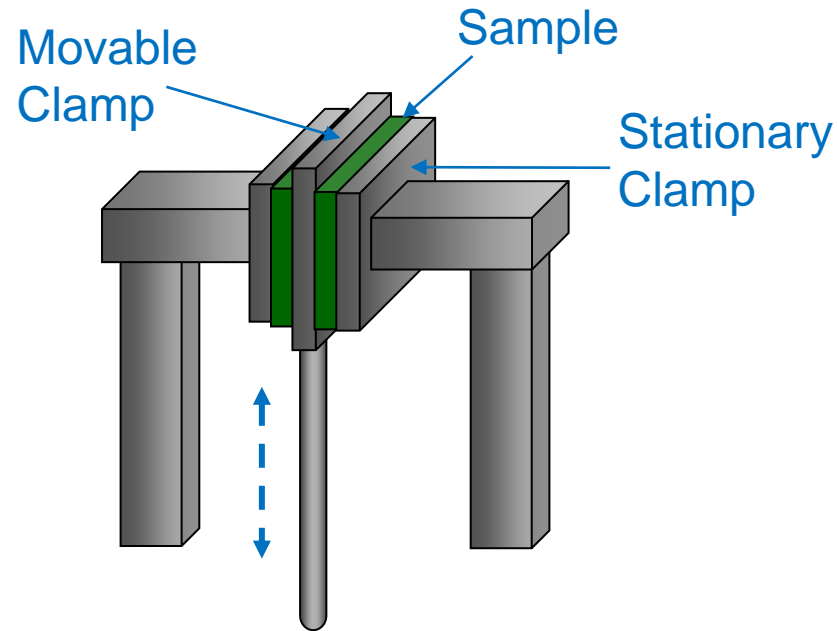
Modulus = Stiffness × Geometry Factor

$$GF_{\text{Comp}} = \frac{\textit{thickness}}{\textit{sample surface area}} = \frac{t}{\pi r^2}$$

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, T_g , secondary transitions (modulus and $\tan \delta$)
- 49 Sample size \leq plate size

Operating Range of the Shear Sandwich Clamp

Modulus = Stiffness × Geometry Factor

$$GF_{\text{Shear}} = \frac{3t}{5wh}$$

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

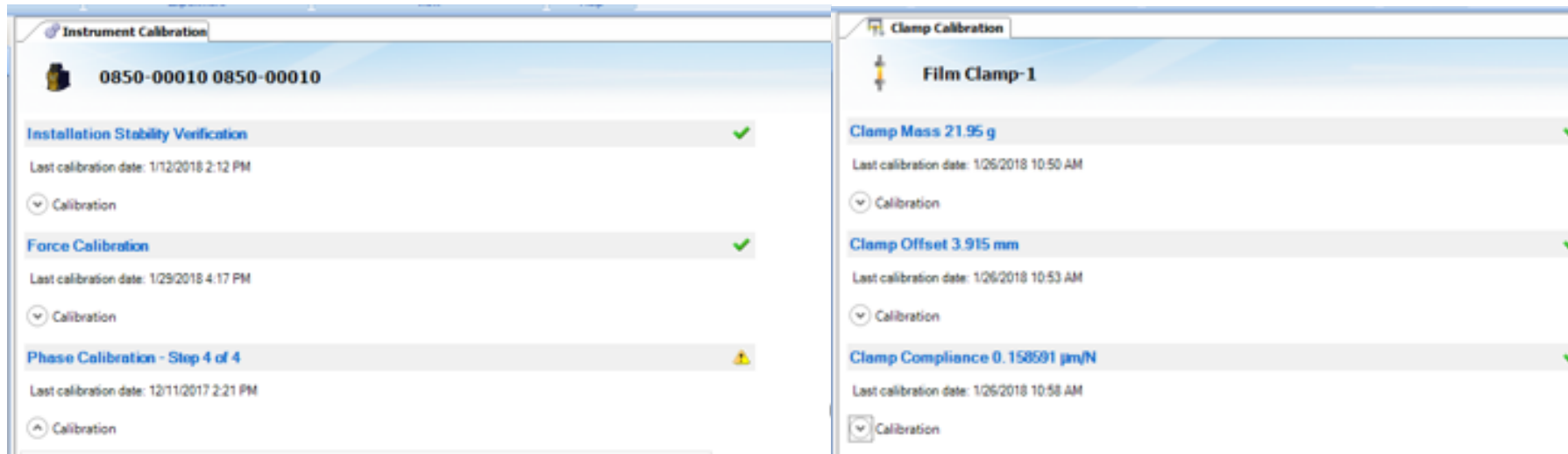
Sample	Clamp	Sample Dimensions
High modulus materials and composites	3-point Bend Dual Cantilever Single Cantilever	$L/T > 10$ if possible
Unreinforced thermoplastics or thermosets	Single Cantilever Dual cantilever	$L/T > 10$ if possible
Brittle solid (ceramics)	3-point Bend	$L/T > 10$ if possible
Elastomers	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

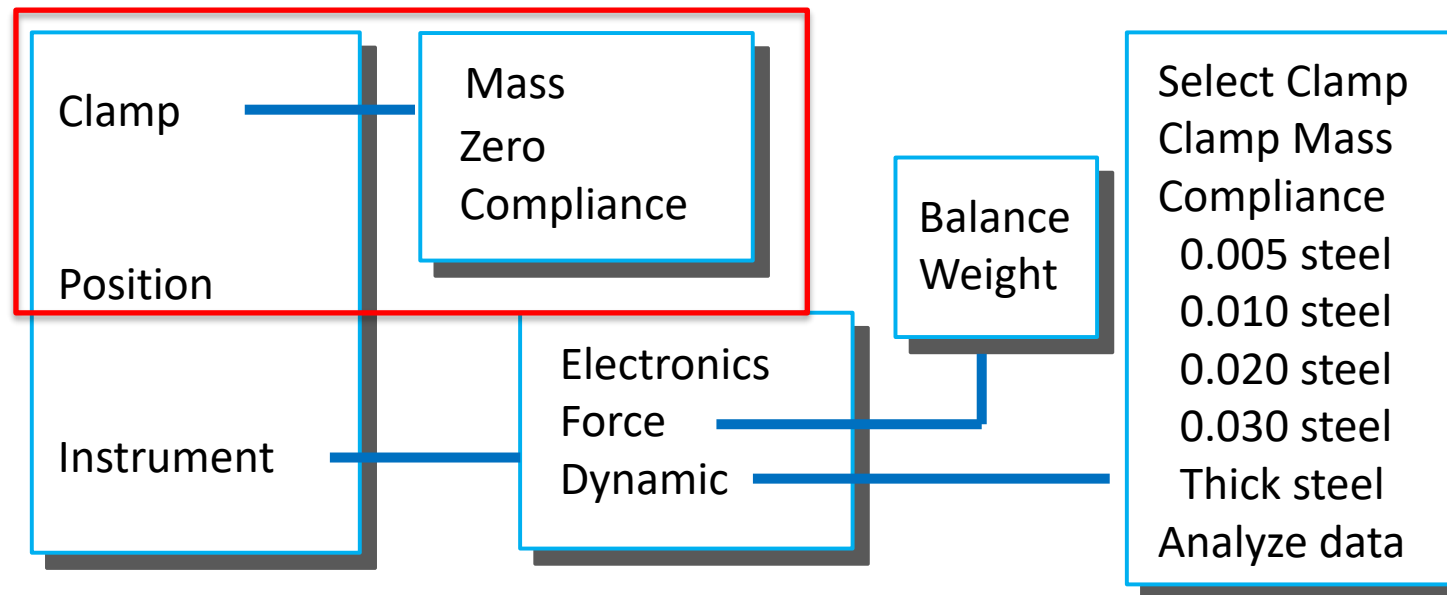
- Follow the online help manual
- The stability verification is performed at installation
- Instrument calibration includes 2 steps: force and phase
- Clamp calibration: perform when newly attached.
- Position calibration from touchscreen



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)



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

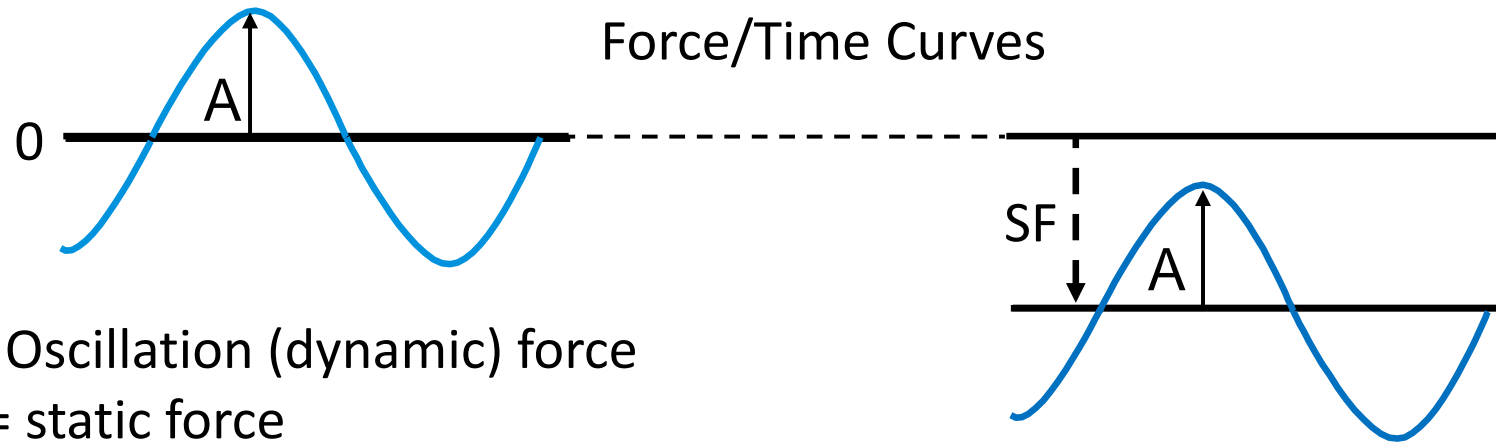


Dynamic (Oscillatory) Testing

Available oscillatory test modes

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

Some Clamps Require Offset (static) Force!



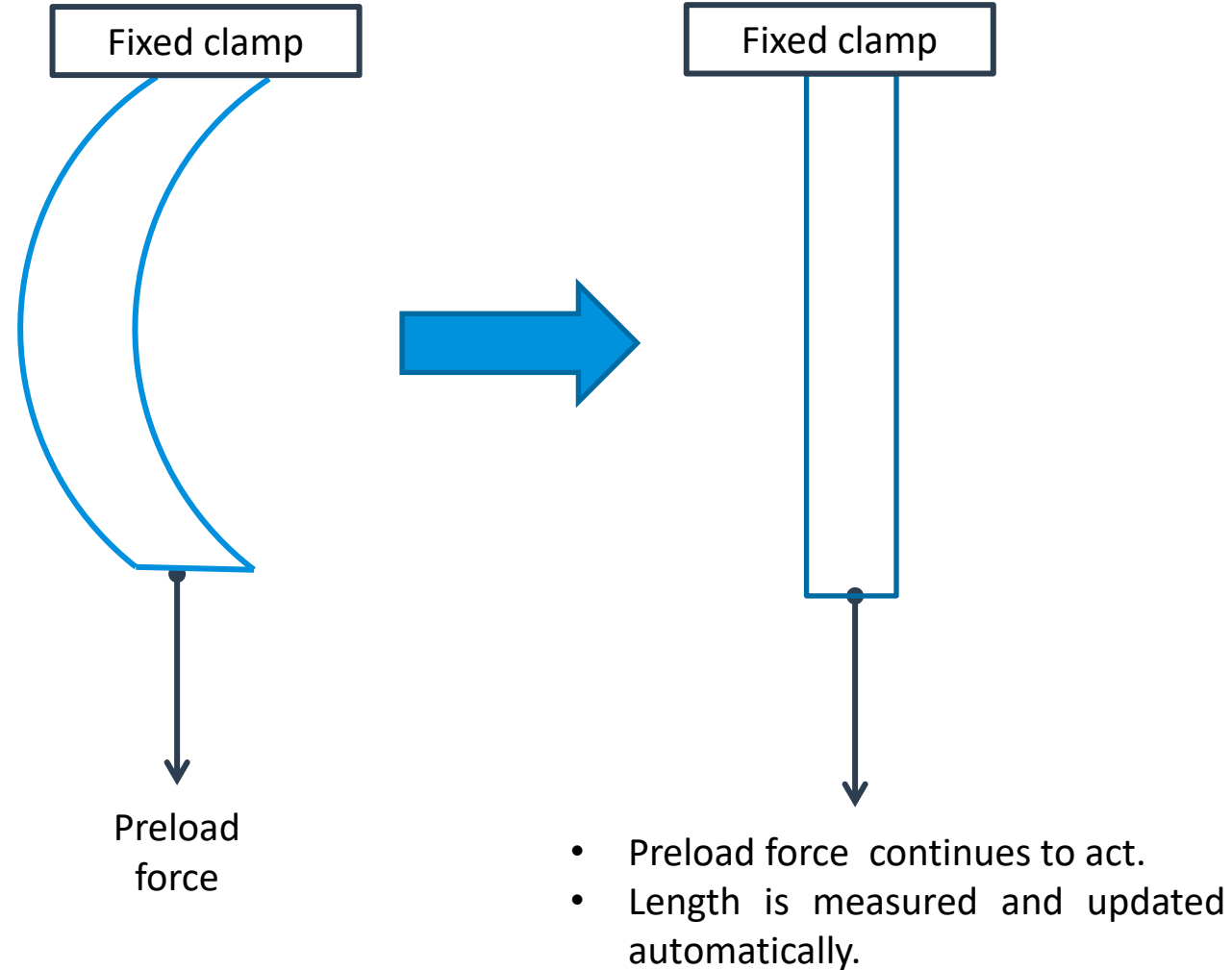
Clamps **without** static force:

- Single Cantilever
- Dual Cantilever
- Shear Sandwich

Clamps **with** static force:

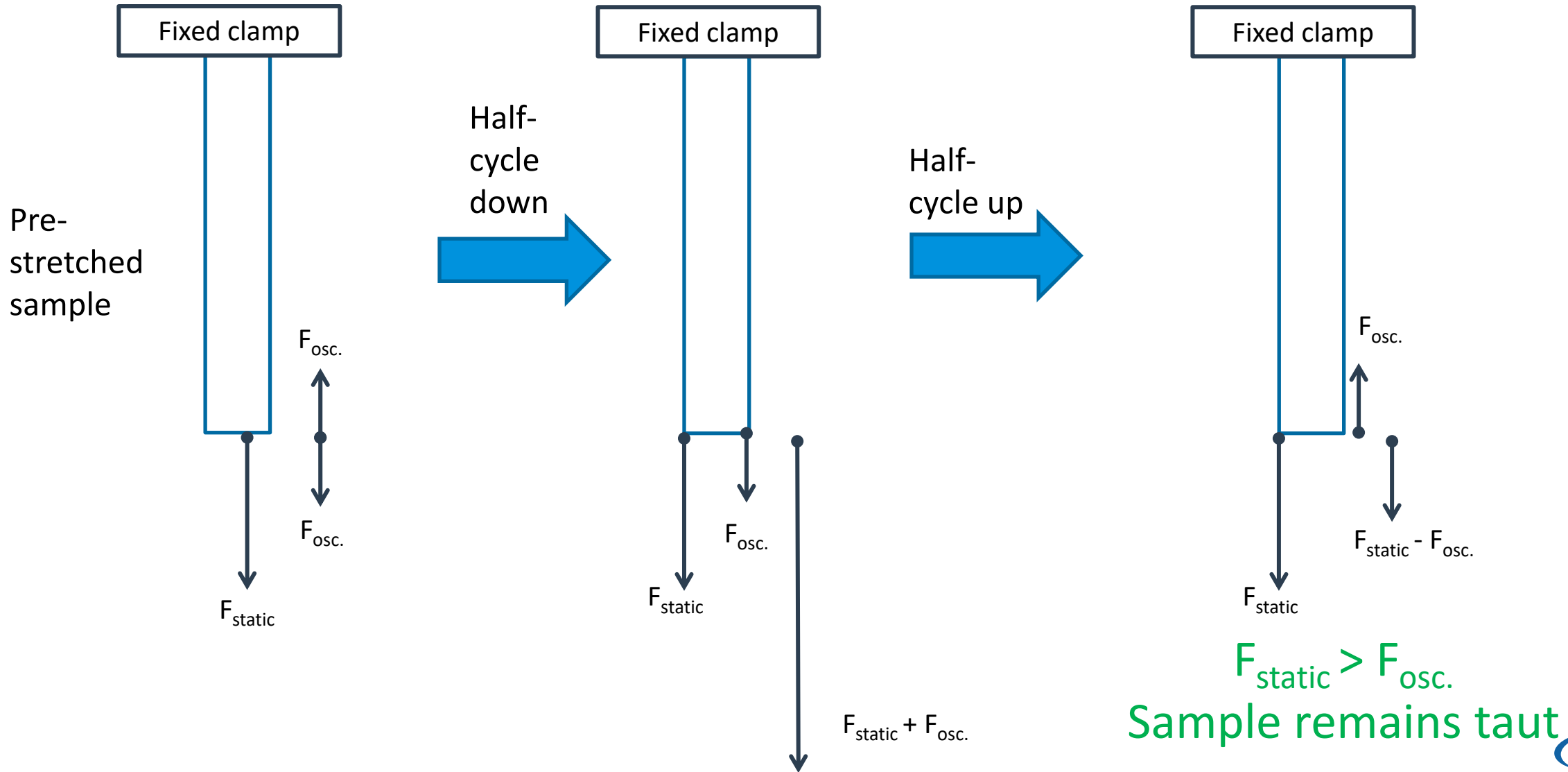
- Tension Film
- Tension: Fiber
- 3-Point Bend
- Compression
- Penetration

Preload force in tension



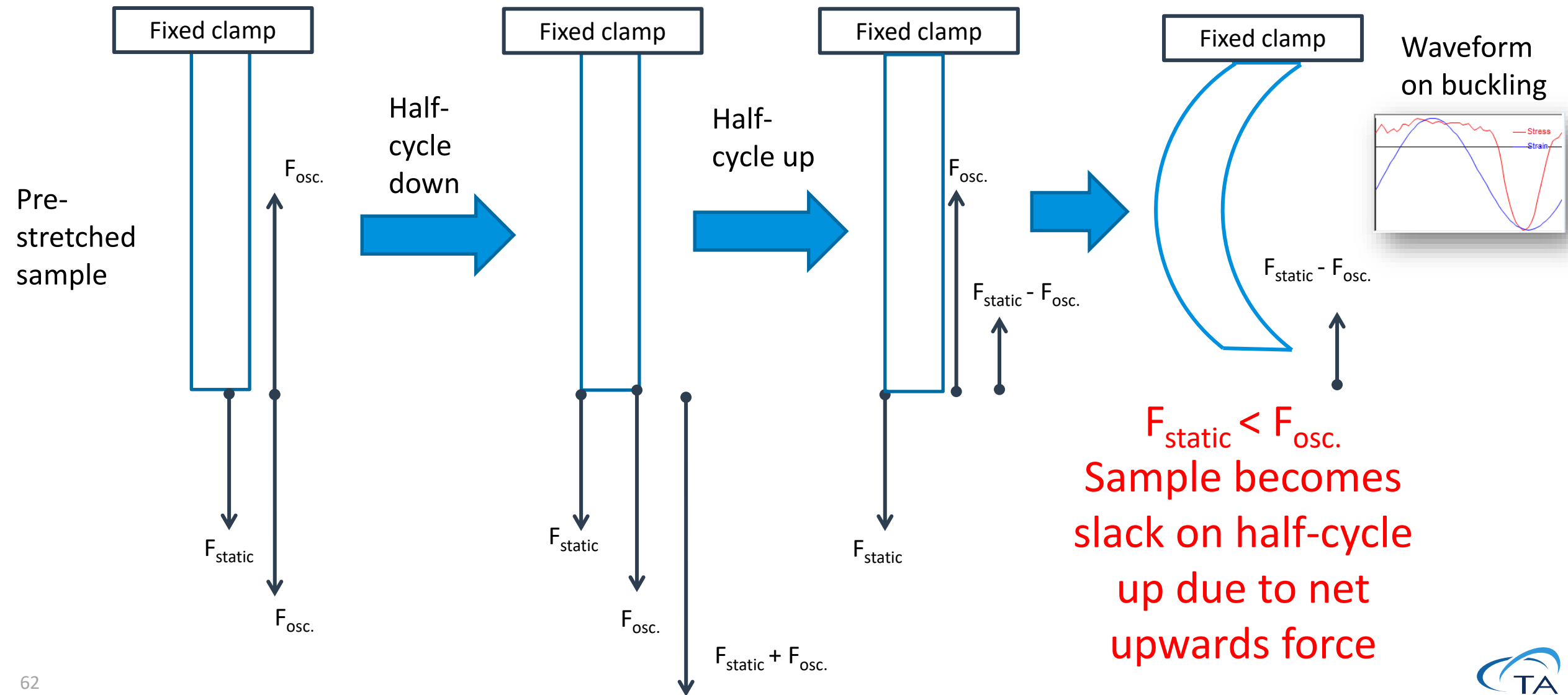
Net forces acting during oscillation (tension)

static force > osc. force



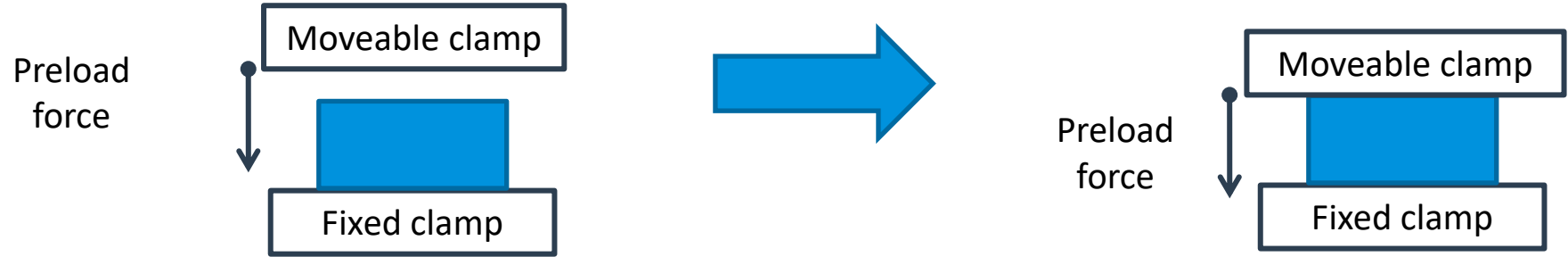
Net forces acting during oscillation (tension)

static force < osc. force



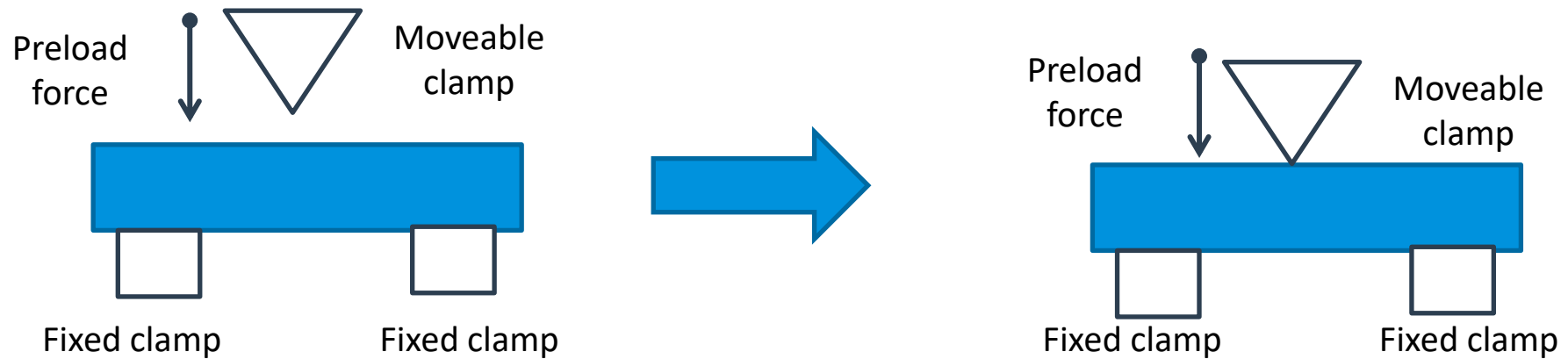
Preload force in compression, 3-point bending

COMPRESSION



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

3-POINT BENDING

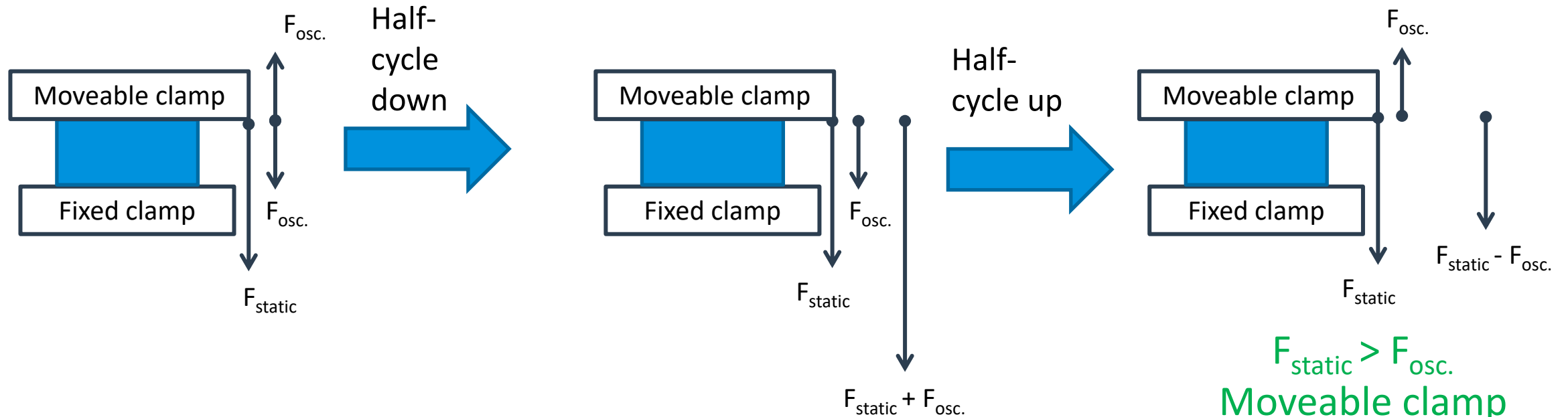


- Sample dimensions are entered manually prior to start of experiment (no automatic update of thickness)

- Preload force continues to act.

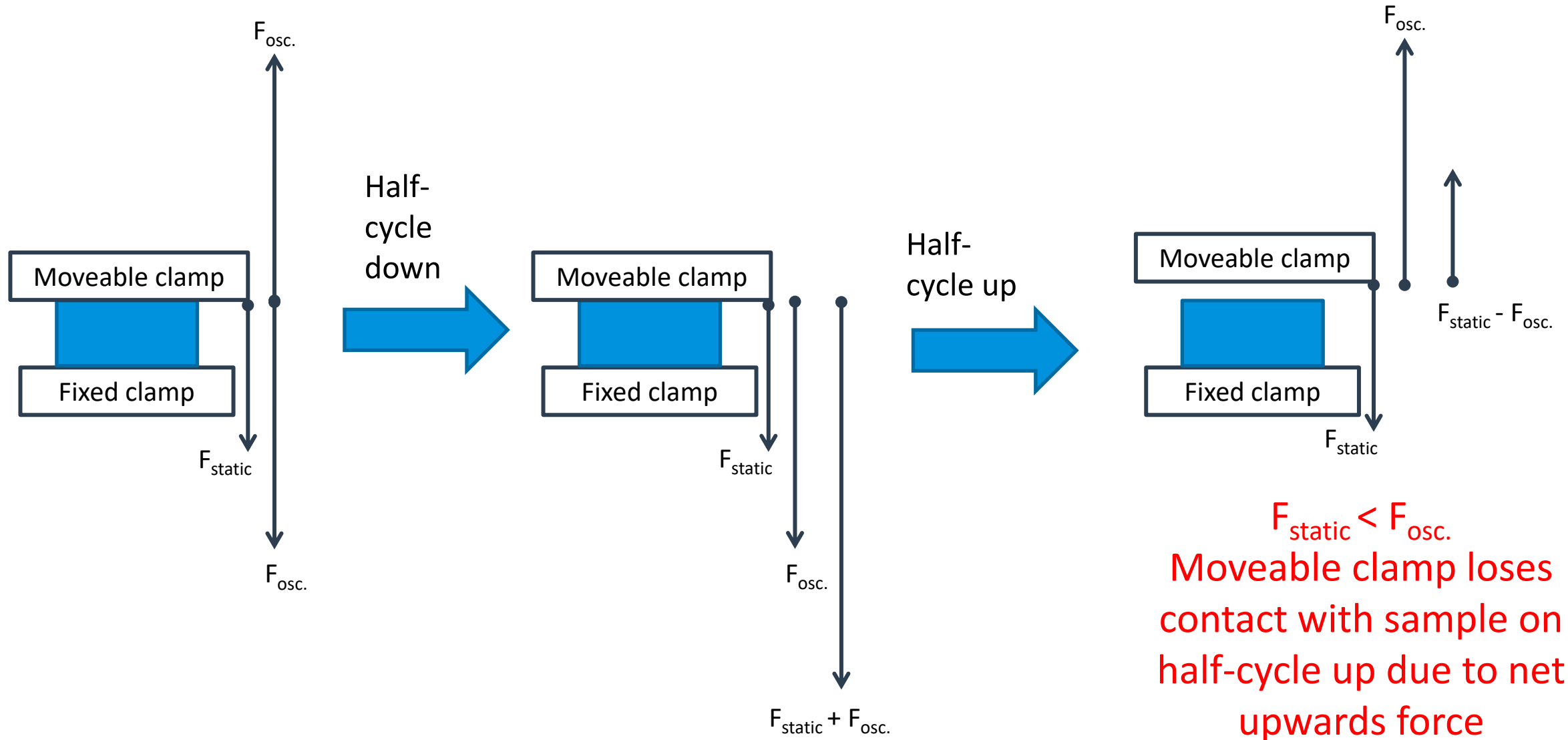
Net forces acting during oscillation (compression and 3PB)

static force > osc. force



Net forces acting during oscillation (compression)

static force < osc. force



Force track

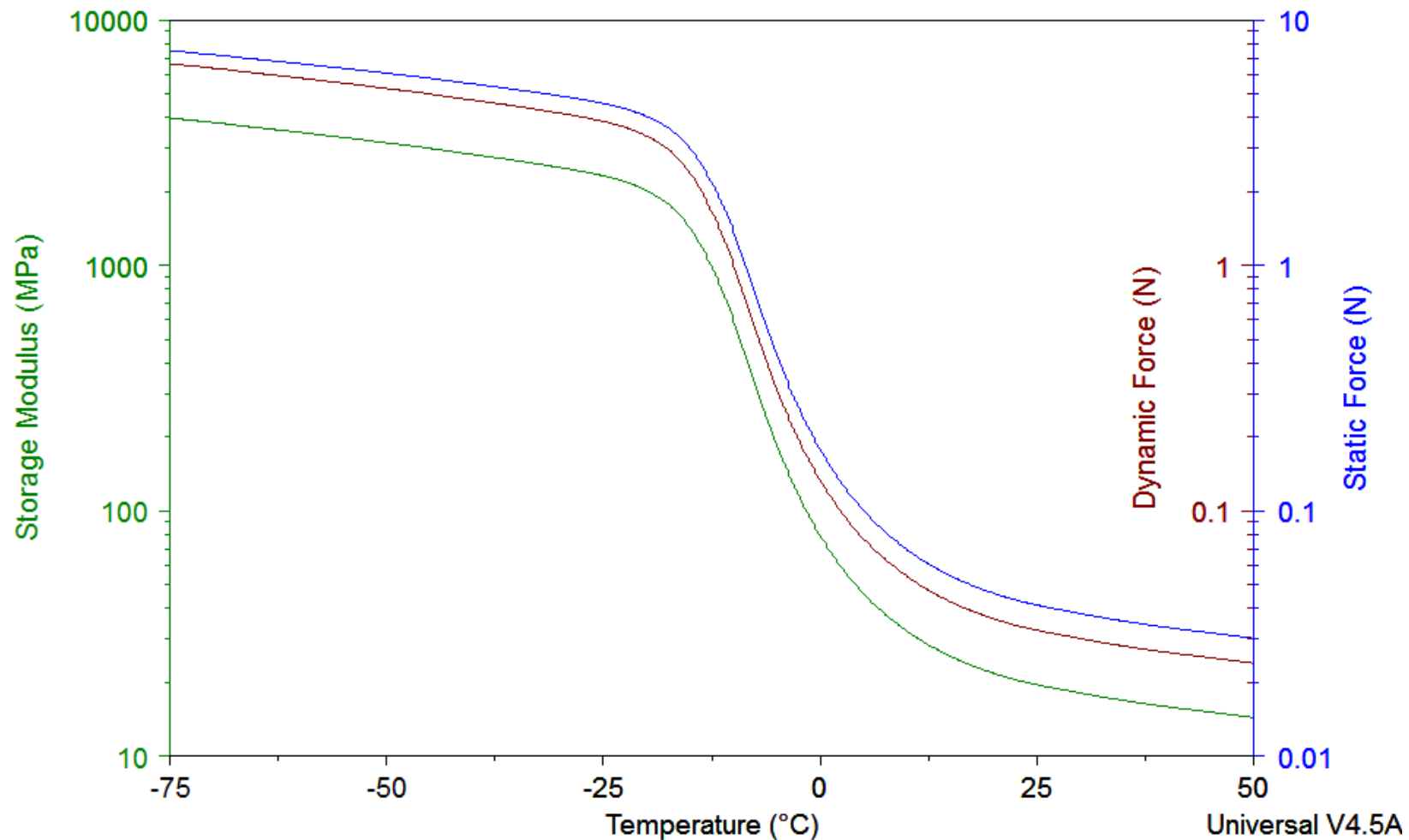
- **Recap: Desired situation for all clamps is that $F_{static} > F_{osc}$**
- Force track = $\frac{F_{static}}{F_{osc}}$
- If $\frac{F_{static}}{F_{osc}} > 1$, then $F_{static} > F_{osc}$
- Force track ratio is expressed as a percentage
 - On 850 and 800, Force track = $\frac{F_{static}}{F_{osc}} \times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

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

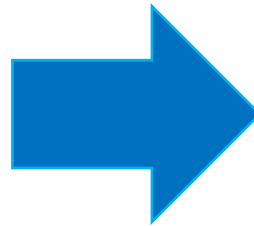
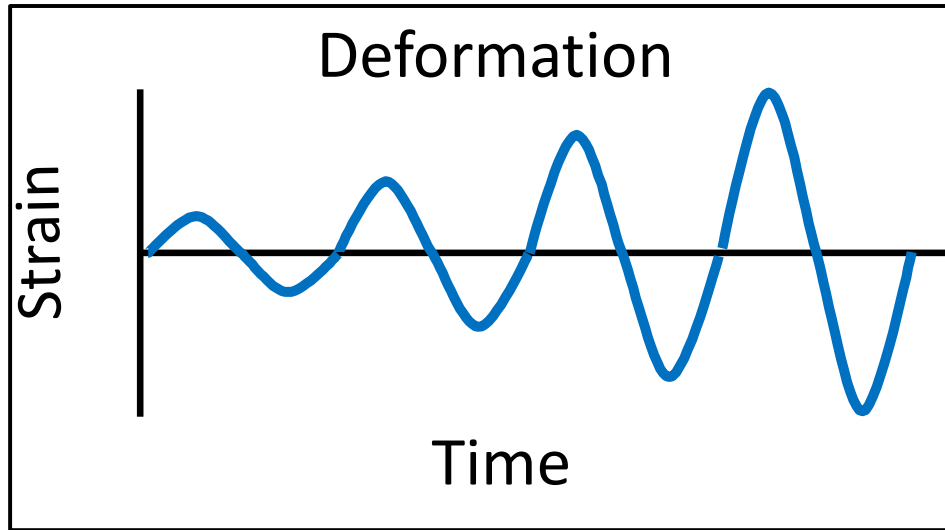


Choosing Force Track Parameters

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

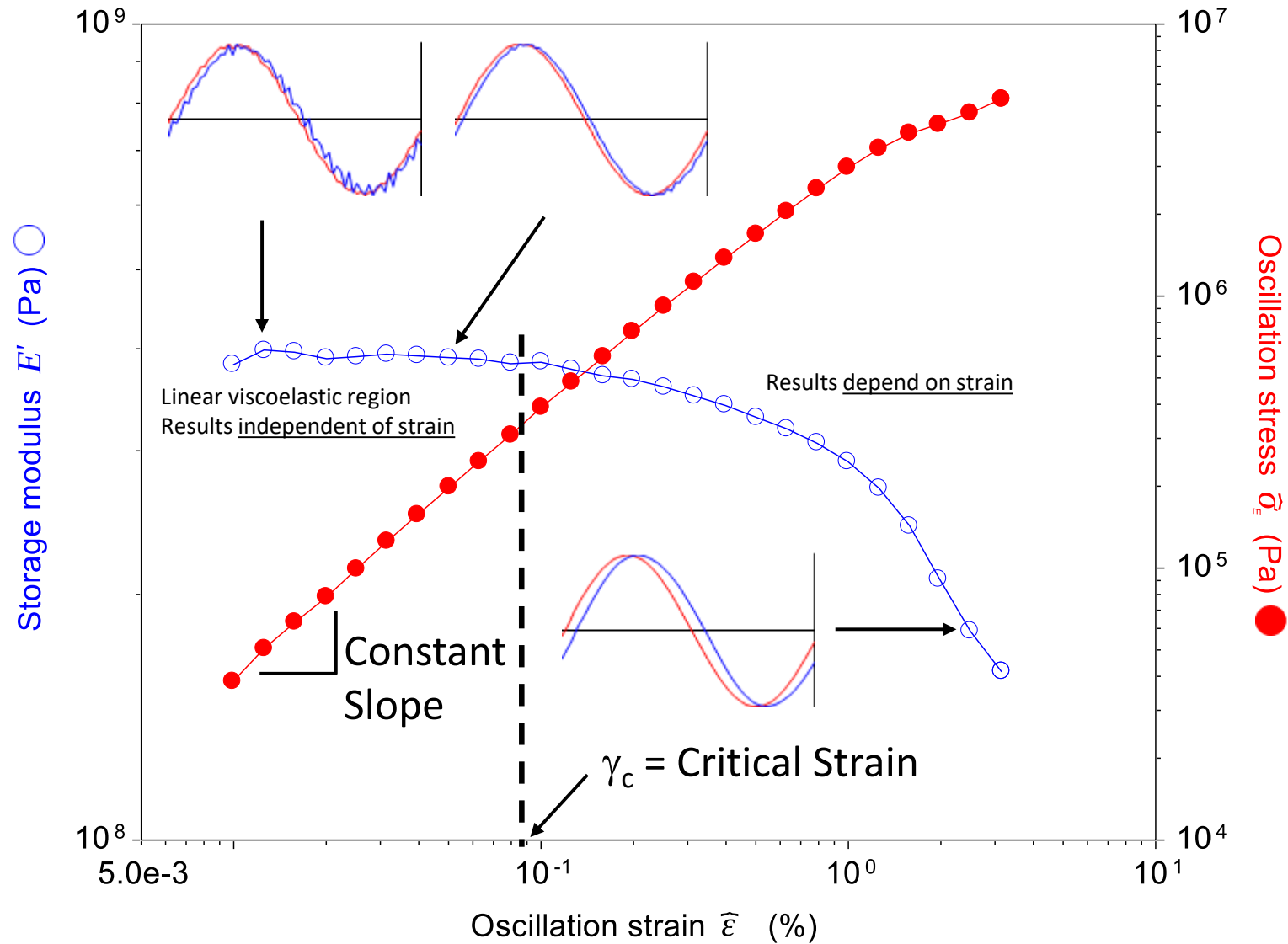


The material response to increasing deformation amplitude is monitored at a constant frequency and temperature.

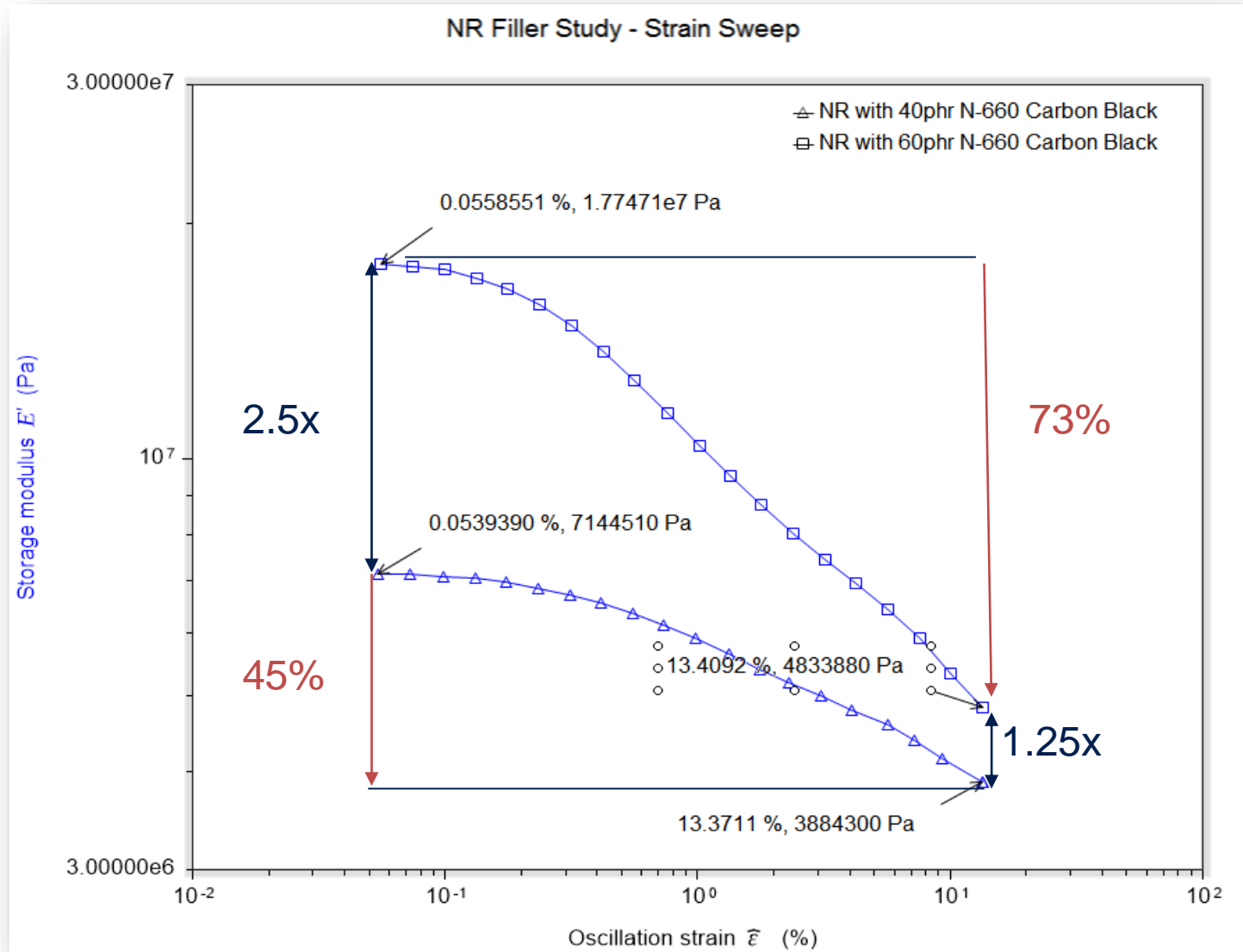
USES

- Identify Linear Viscoelastic Region
- Resilience/elasticity

Dynamic Strain Sweep: Material Response

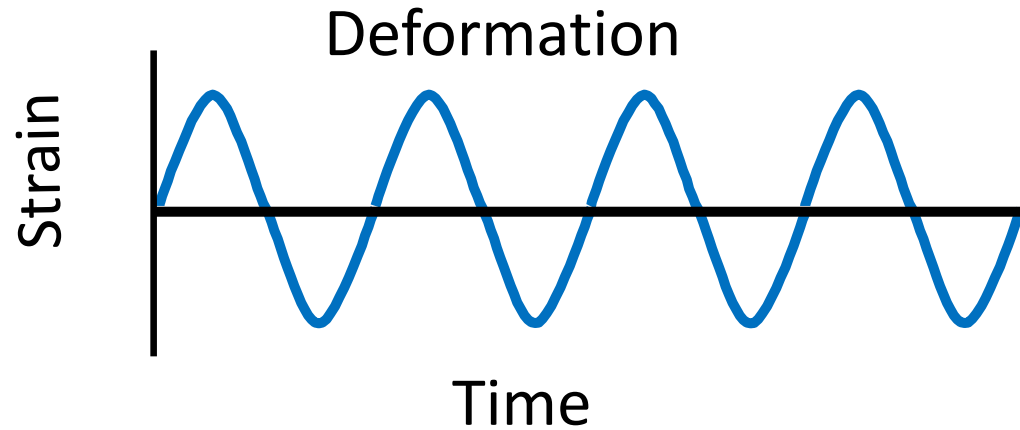


Rubber: Effect of Filler Content in Tire Rubber (NR)



- 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

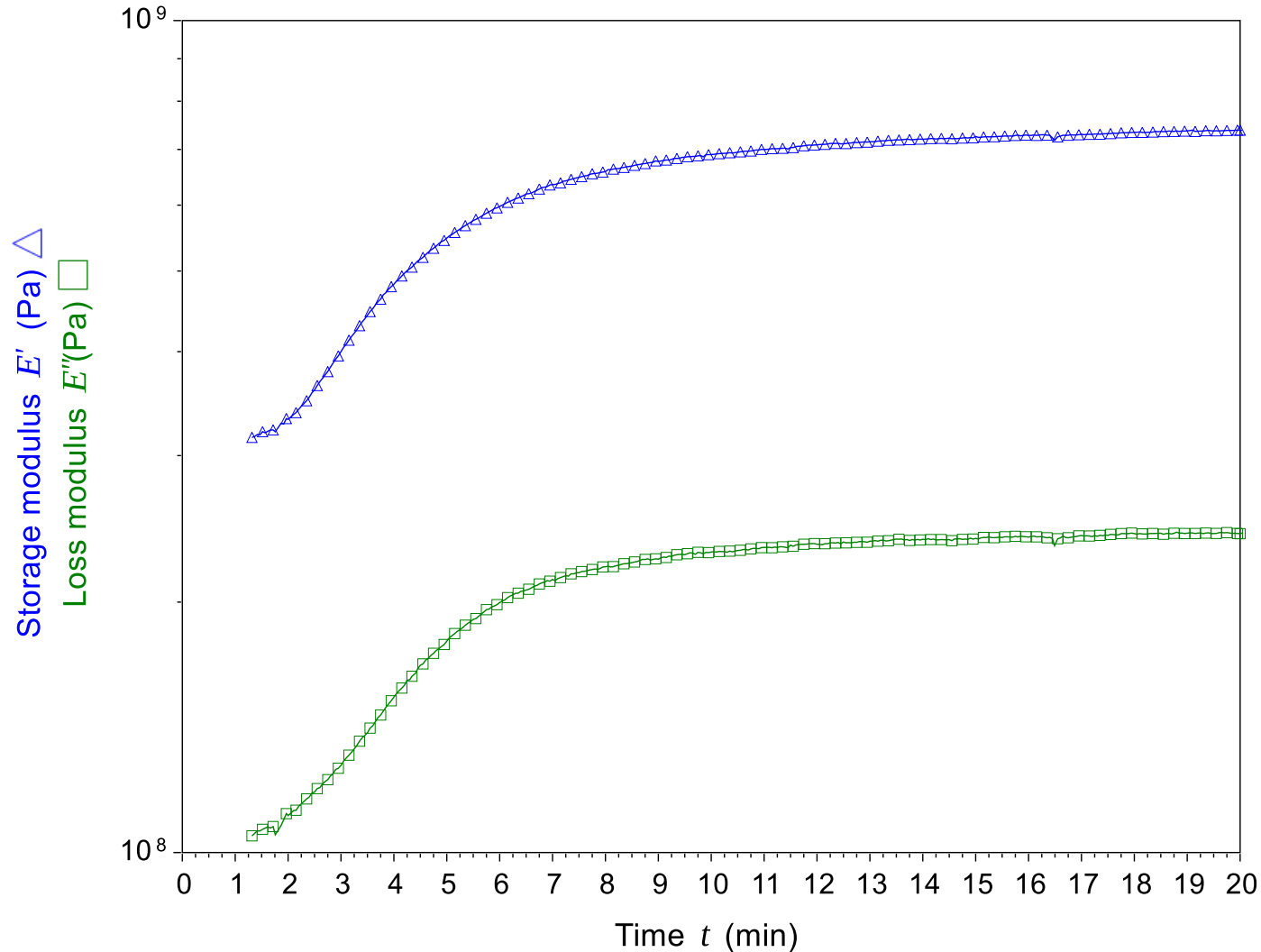


The material response is monitored at a constant frequency, amplitude and temperature.

USES

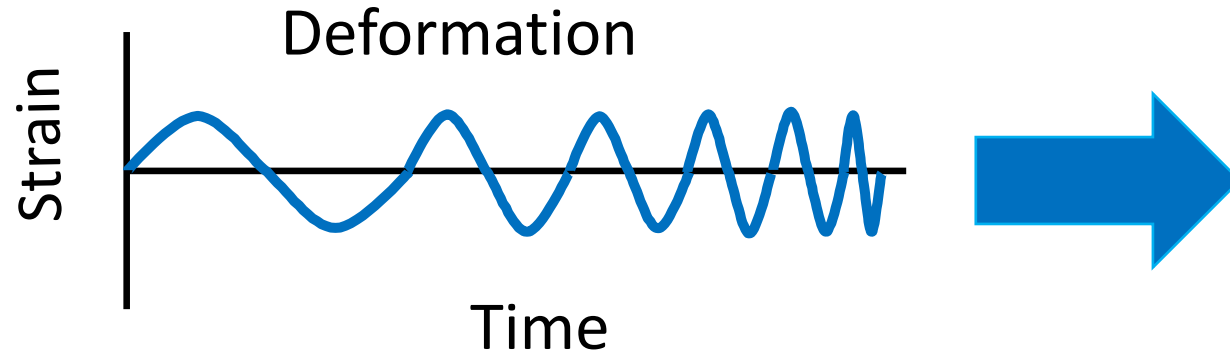
- Curing studies
- Fatigue tests
- Stability against thermal degradation

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



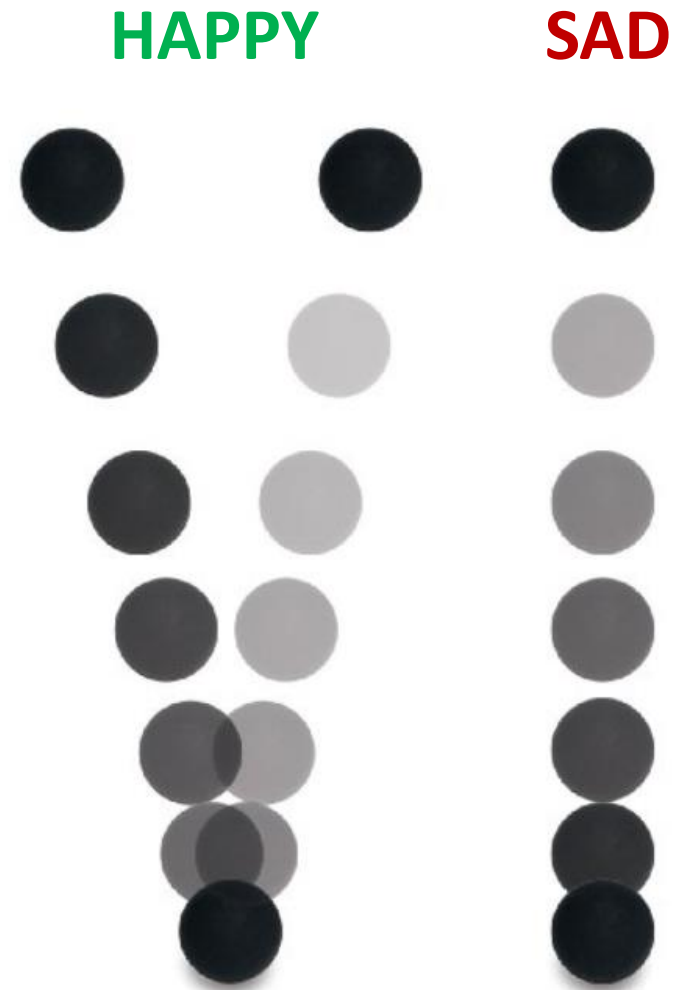
- The material response to increasing frequency (rate of deformation) is monitored at a constant amplitude and temperature.

USES

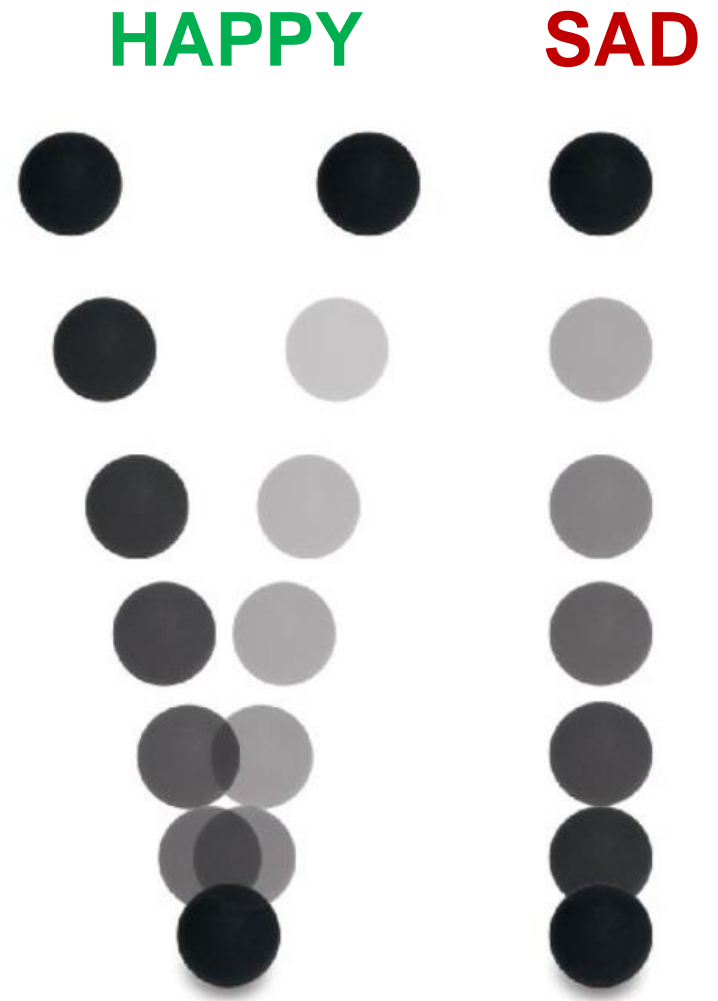
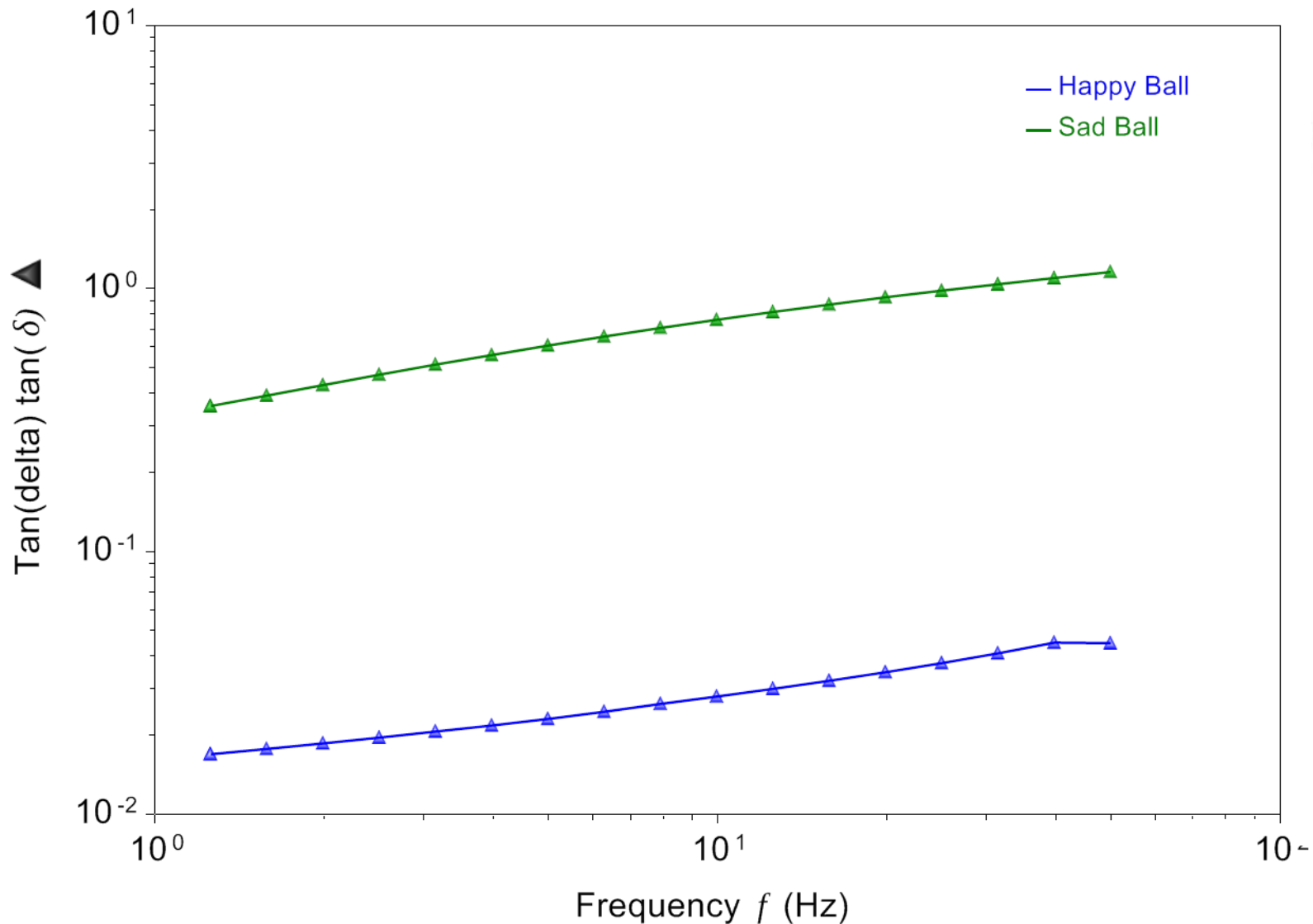
- Quick comparison on modulus and elasticity on solids
- Study polymer melt processing (shear sandwich).
- Estimate long term properties with extended frequency (time) range using TTS

Happy and sad balls vide

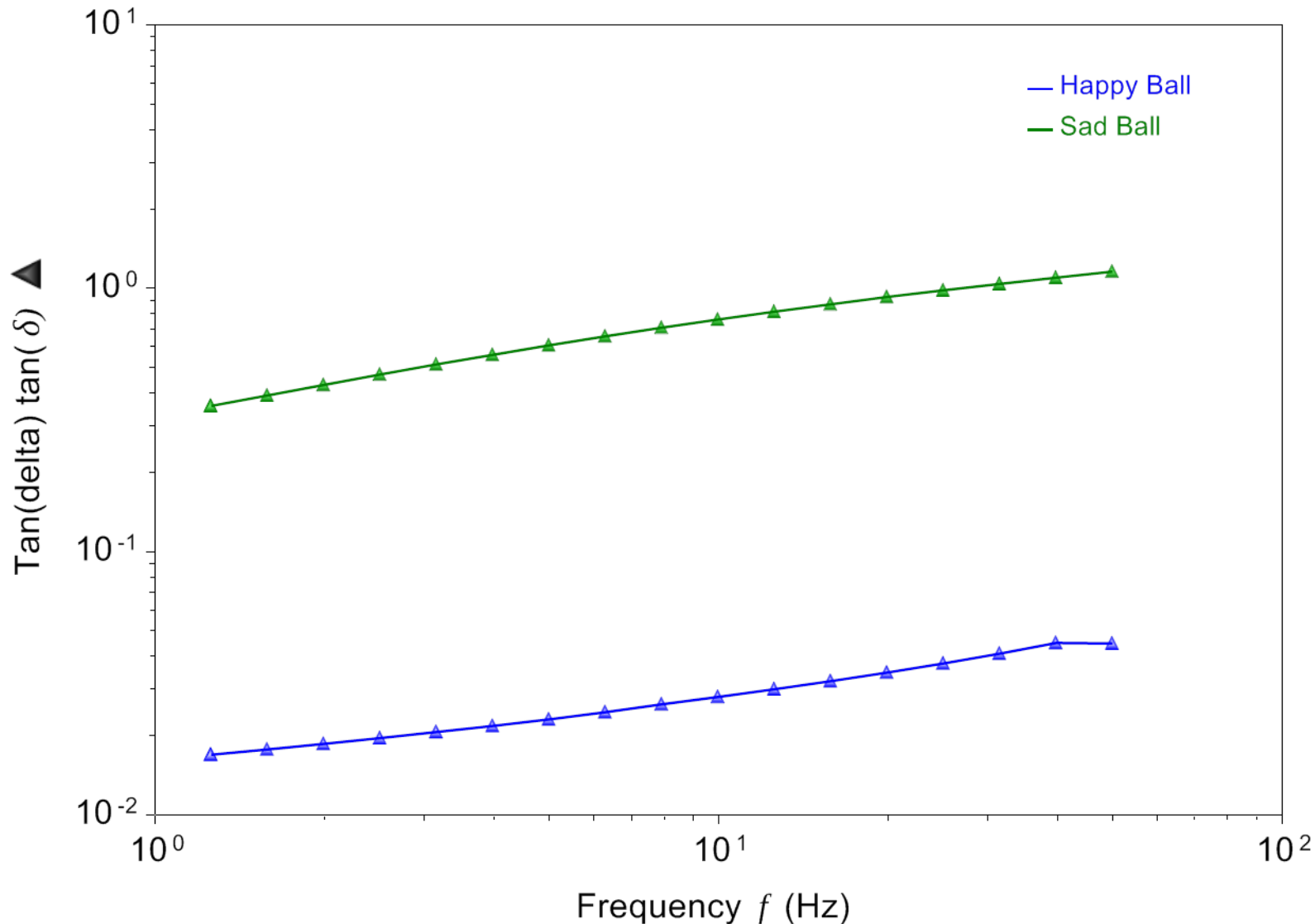
- Set of 2 black rubber balls used as demonstration of viscoelastic behavior.
 - When dropped, the Happy Ball bounces and the Sad Ball does not.



Dynamic Mechanical Properties: Tan Delta



Dynamic Mechanical Properties: Tan Delta

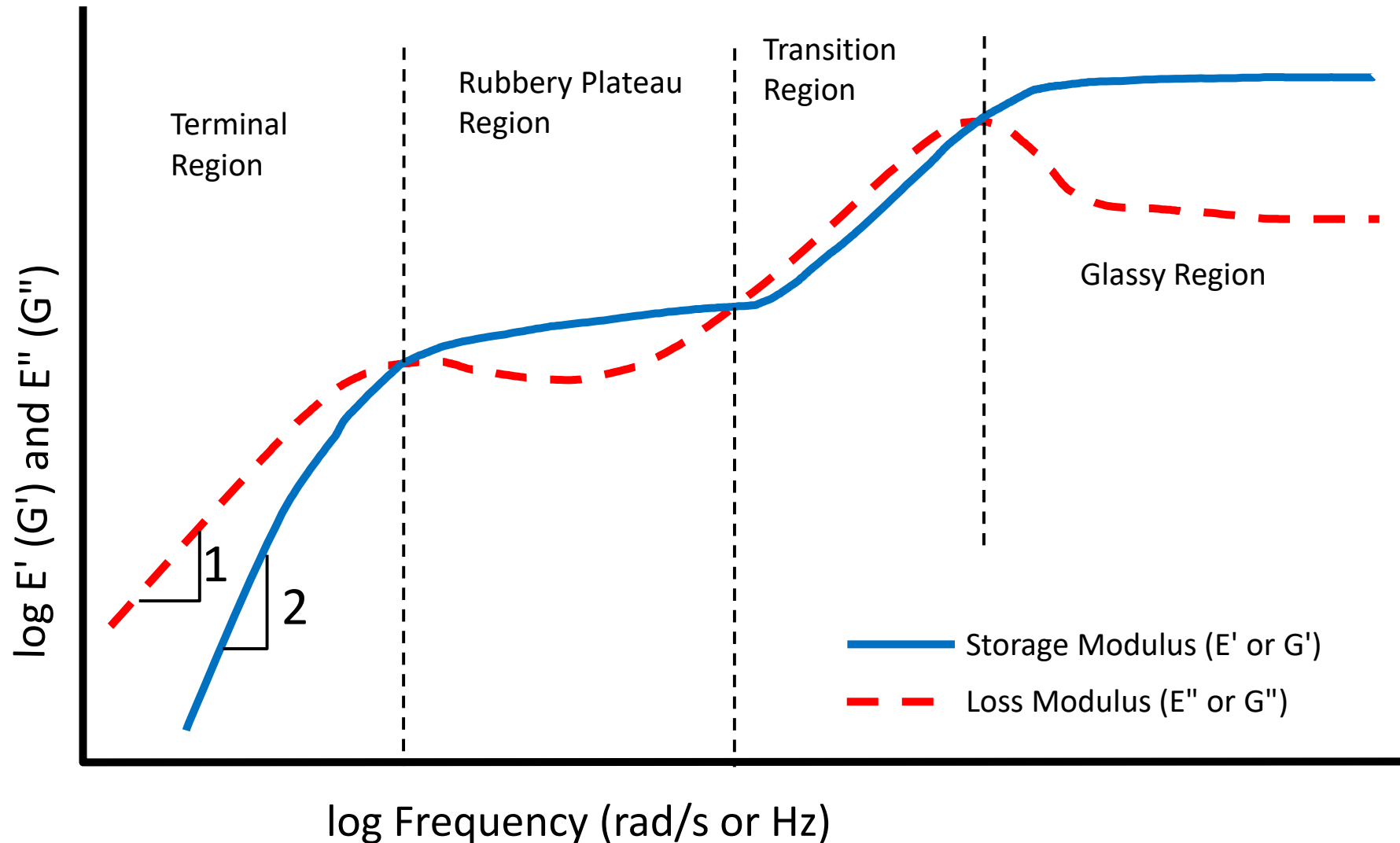


- $\tan \delta$: Measure of material damping. Balancing viscous property while having the appropriate level of stiffness.
- Increasing $\tan \delta$ implies a greater potential for energy dissipation and lower elasticity, and vice-versa.

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

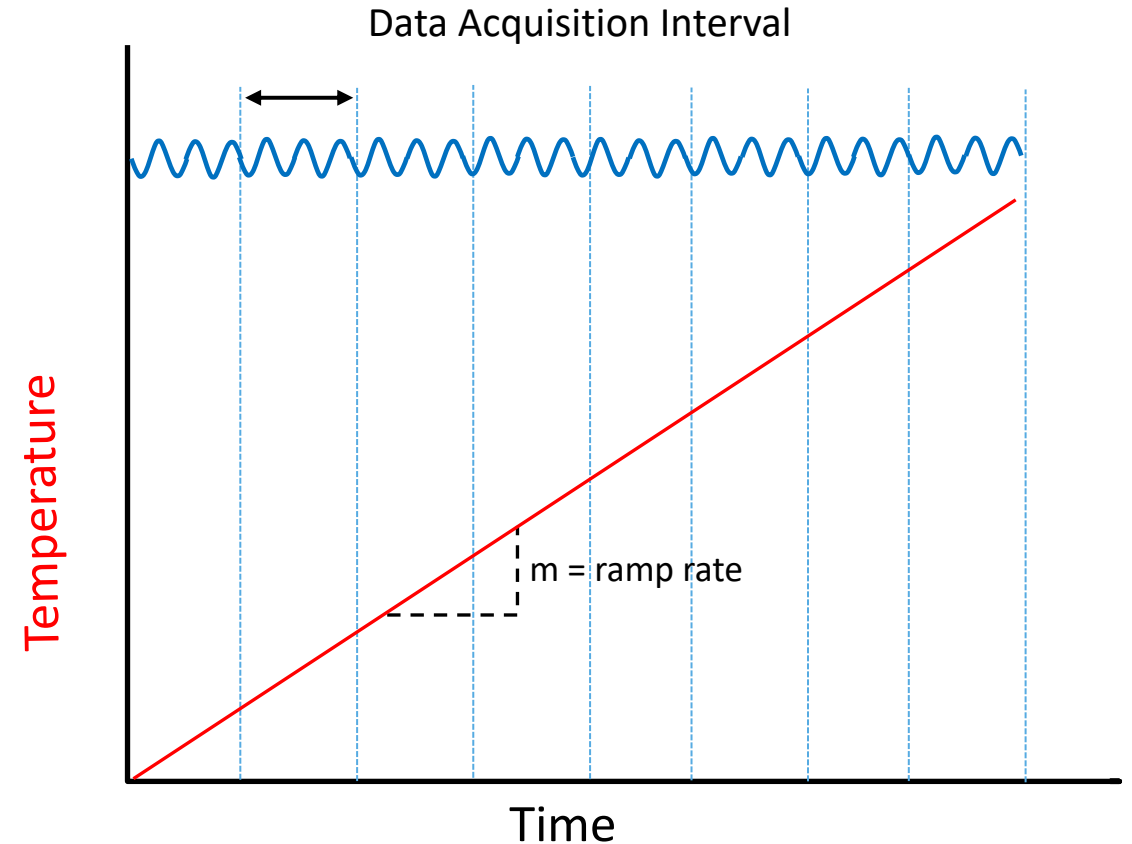
- Sad ball has a much higher $\tan \delta$ compared to the happy ball

Frequency Sweep: Material Response



Dynamic Temperature Ramp

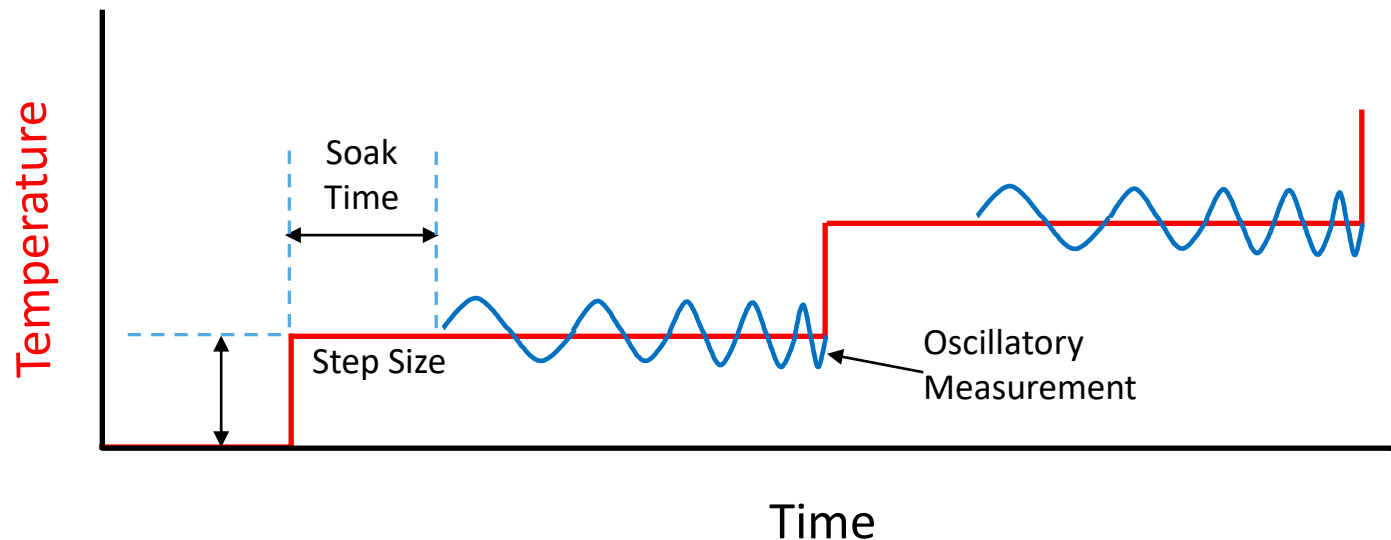
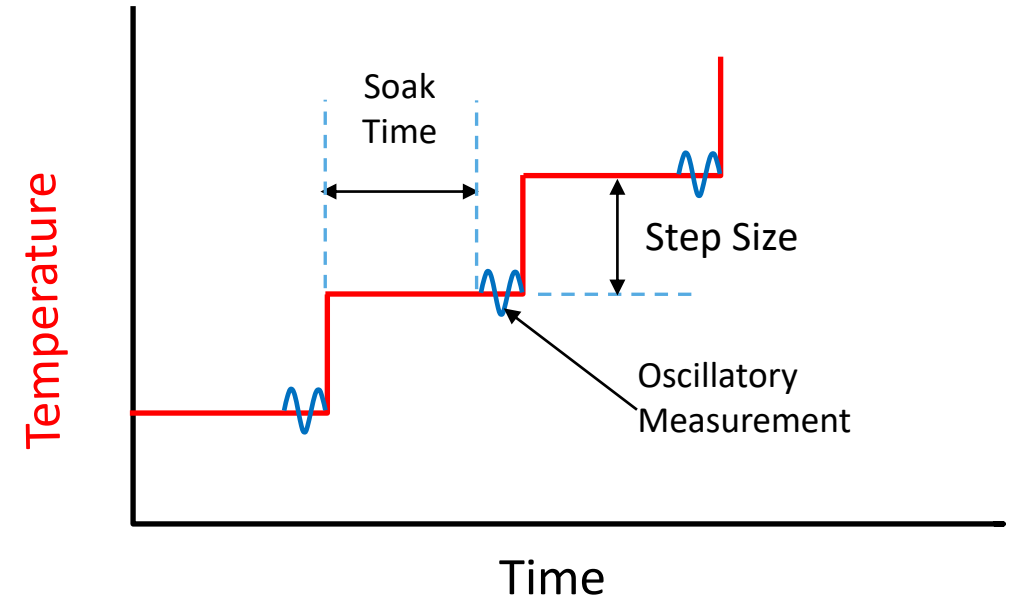
- A linear heating rate is applied. The material response is monitored at a constant frequency and constant amplitude of deformation. Data is taken at user defined time intervals.



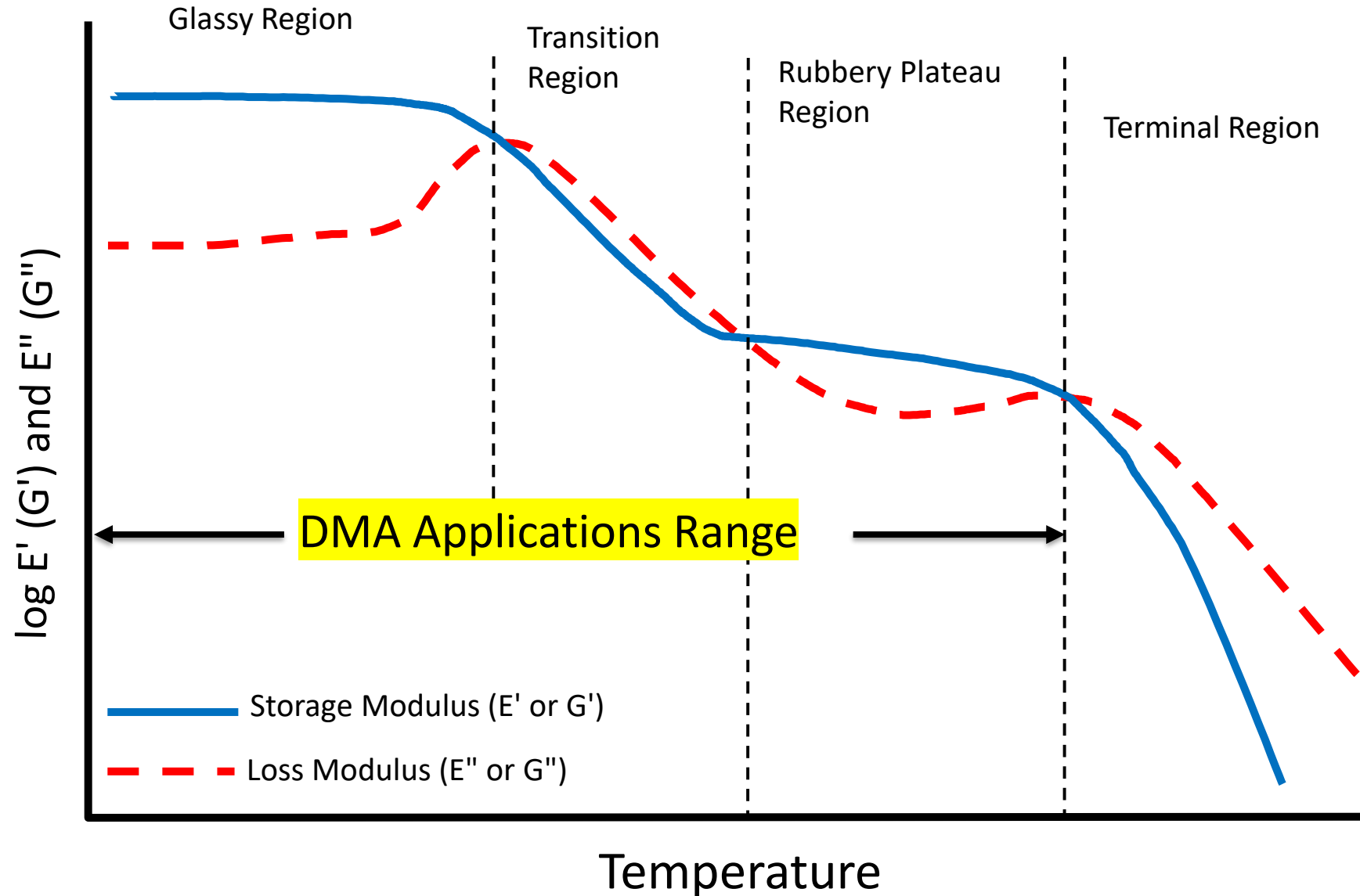
Recommend ramp rate for polymer testing: 1-5°C/min.

Temperature Step & Hold- Single /Multi-Frequency

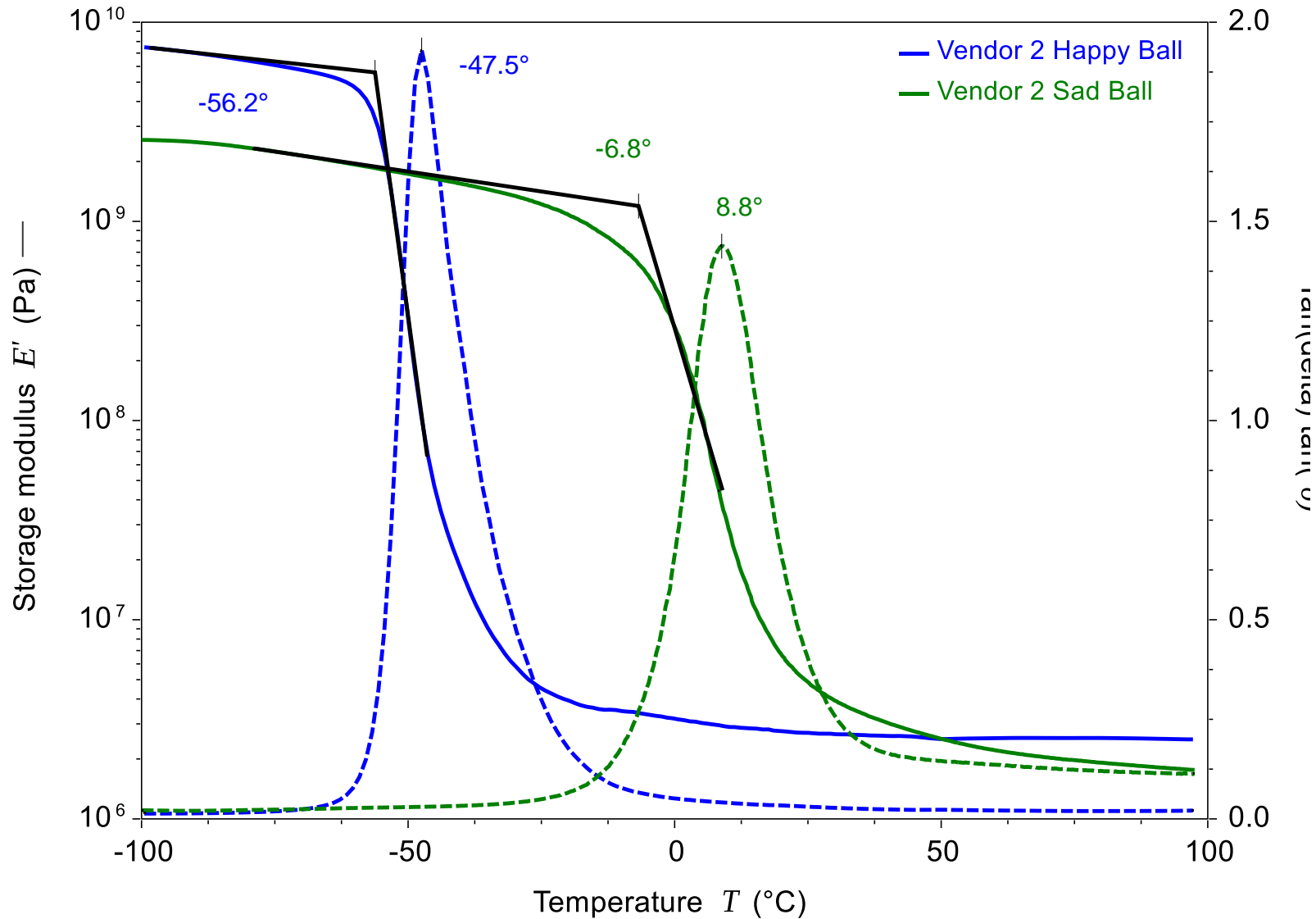
- A step and hold temperature profile is applied. The material response is monitored at one, or over a range of frequencies, at constant amplitude of deformation



Temperature Profile on Amorphous Polymers



DMA temperature ramp: Happy and sad balls



⌵ Oscillation Temperature Ramp

Amplitude	<input type="text" value="20.0"/>	μm
Frequency	<input type="text" value="1.0"/>	Hz
<input type="checkbox"/> Use current temperature		
Ramp from	<input type="text" value="-90"/> °C	to <input type="text" value="100"/> °C
Ramp rate	<input type="text" value="3.0"/>	°C/min
Soak times		
at Start temperature	<input type="text" value="00:05:00"/>	hh:mm:ss
at End temperature	<input type="text" value="00:00:00"/>	hh:mm:ss
Estimated time to complete	<input type="text" value="01:03:20"/>	hh:mm:ss

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



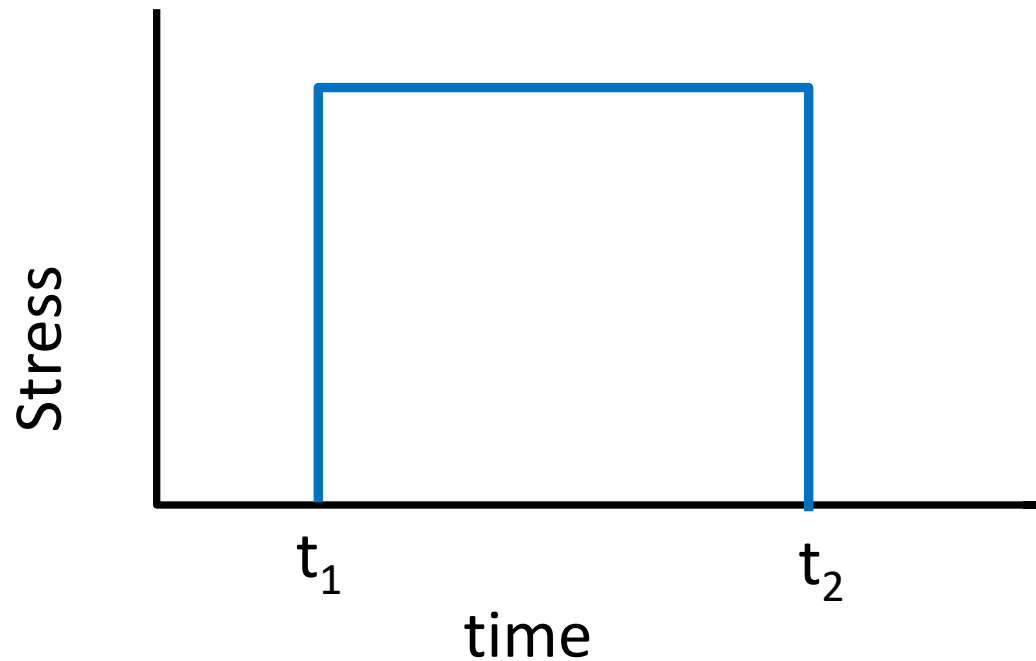
Transient Testing

Available transient test modes

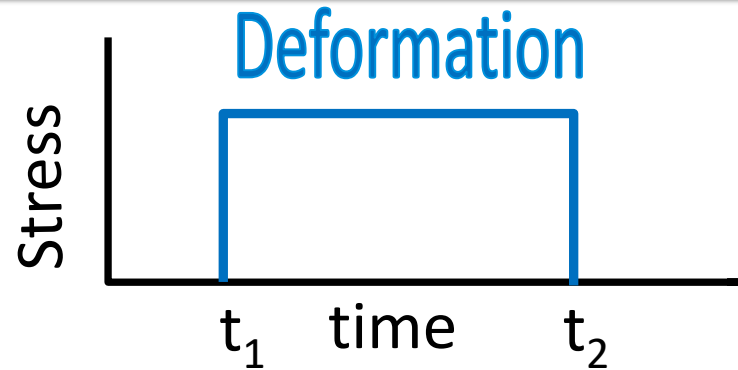
- Creep-Recovery
- Stress Relaxation
- Iso-strain Temperature Ramp
- Iso-force Temperature Ramp
- Stress-Strain Rate Tests

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 $\varepsilon(t)$).



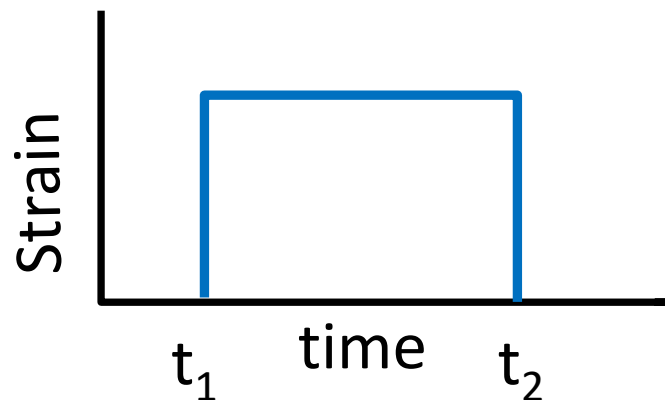
Creep recovery



Response of Classical Extremes

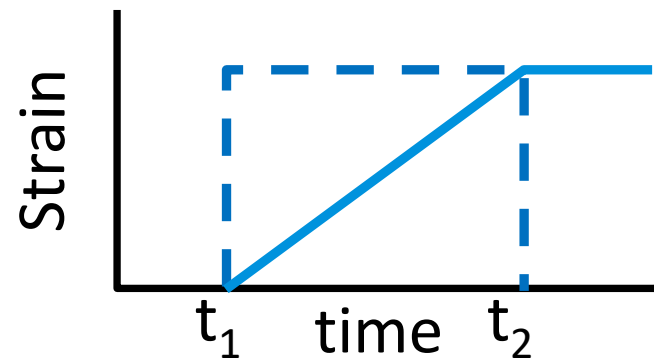
Elastic

- Strain for $t > t_1$ is constant
- Strain for $t > t_2$ is 0

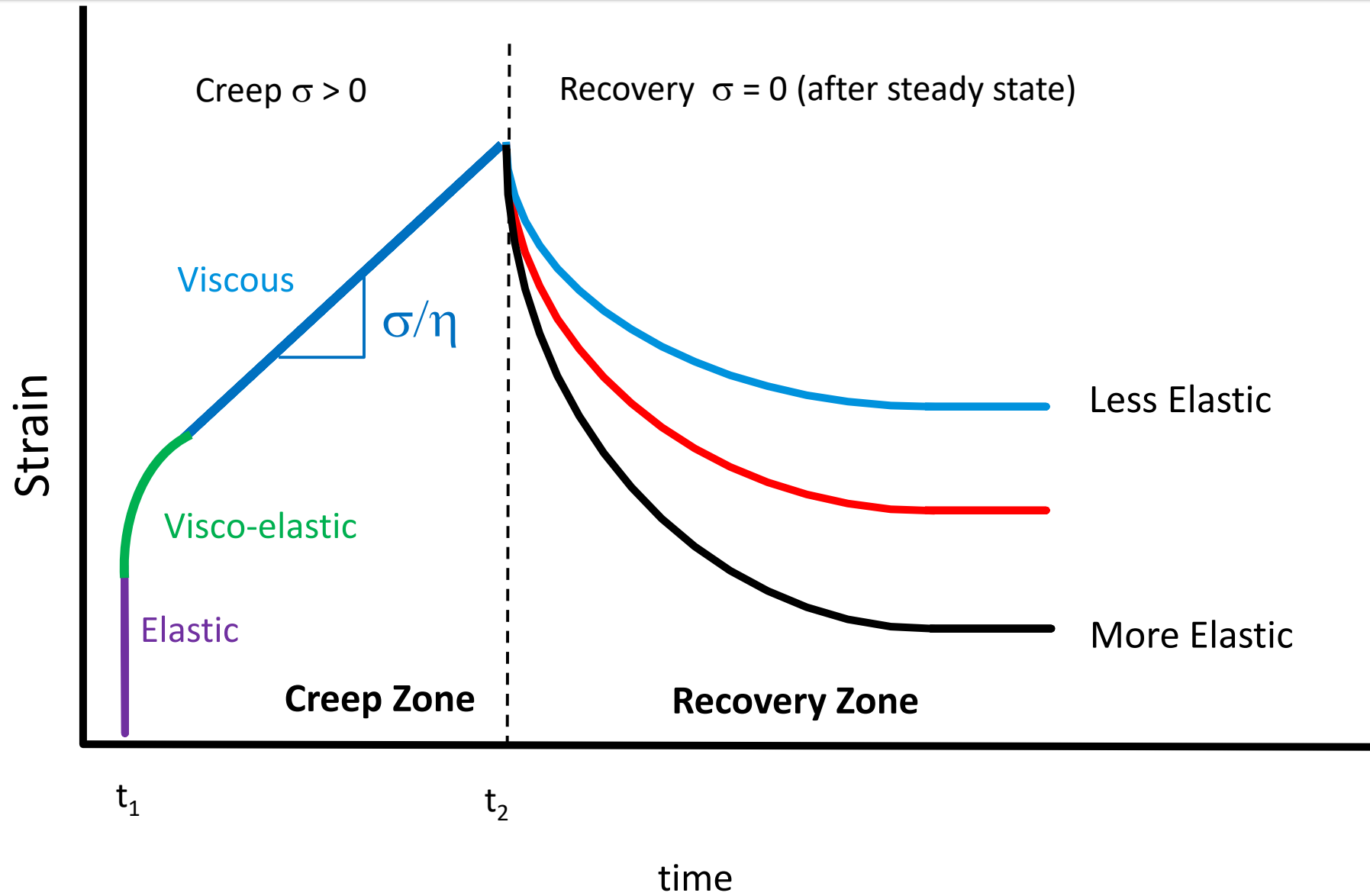


Viscous

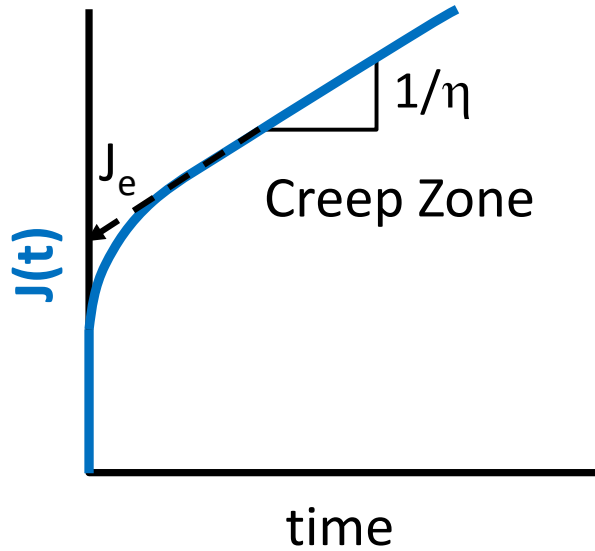
- Strain rate for $t > t_1$ is constant
- Strain for $t > t_1$ increase with time
- Strain rate for $t > t_2$ is 0



Creep recovery



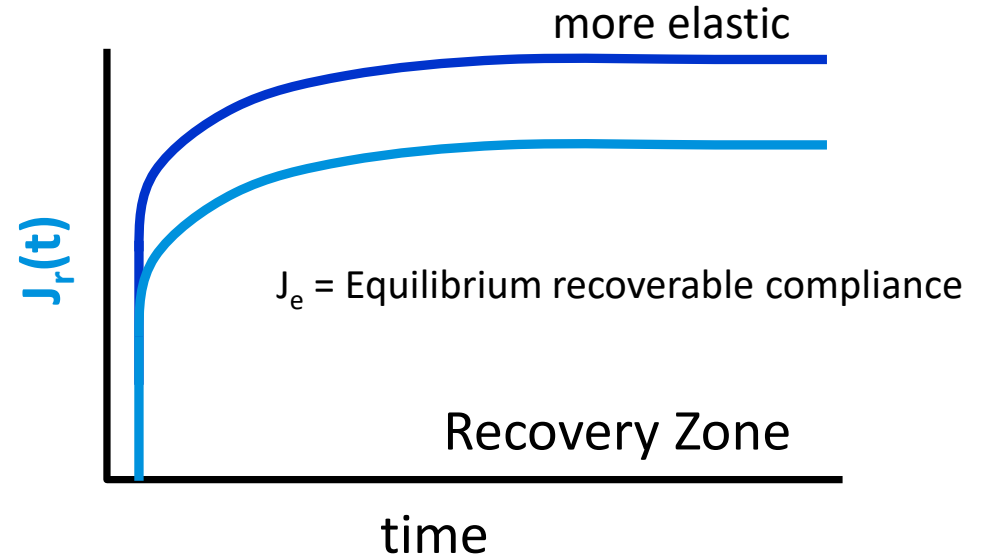
Creep recovery : Creep and Recoverable Compliance



Creep Compliance

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

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

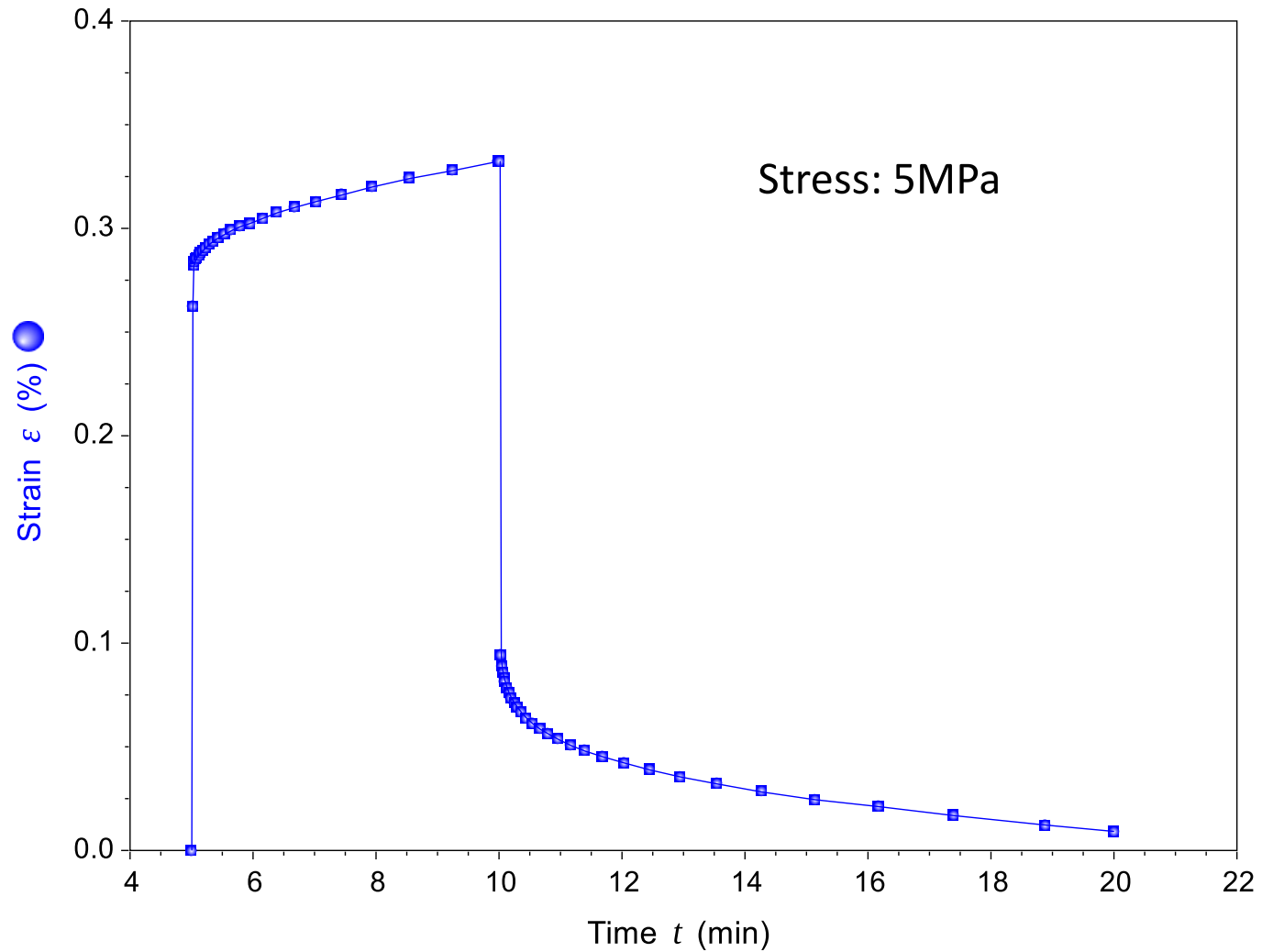


Recoverable Compliance

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

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

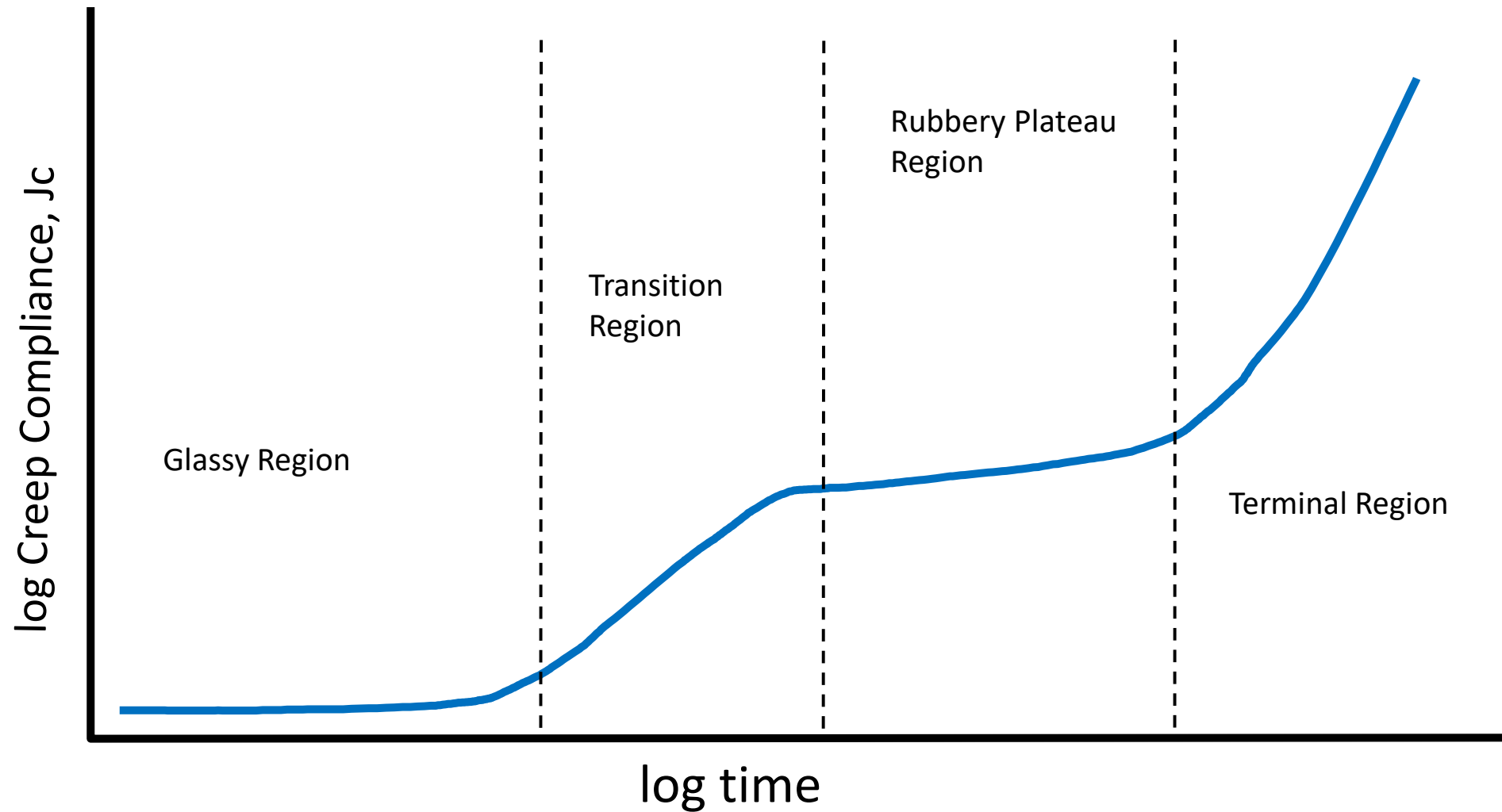
Creep-recovery test on PET Film



p/n: 984309.901

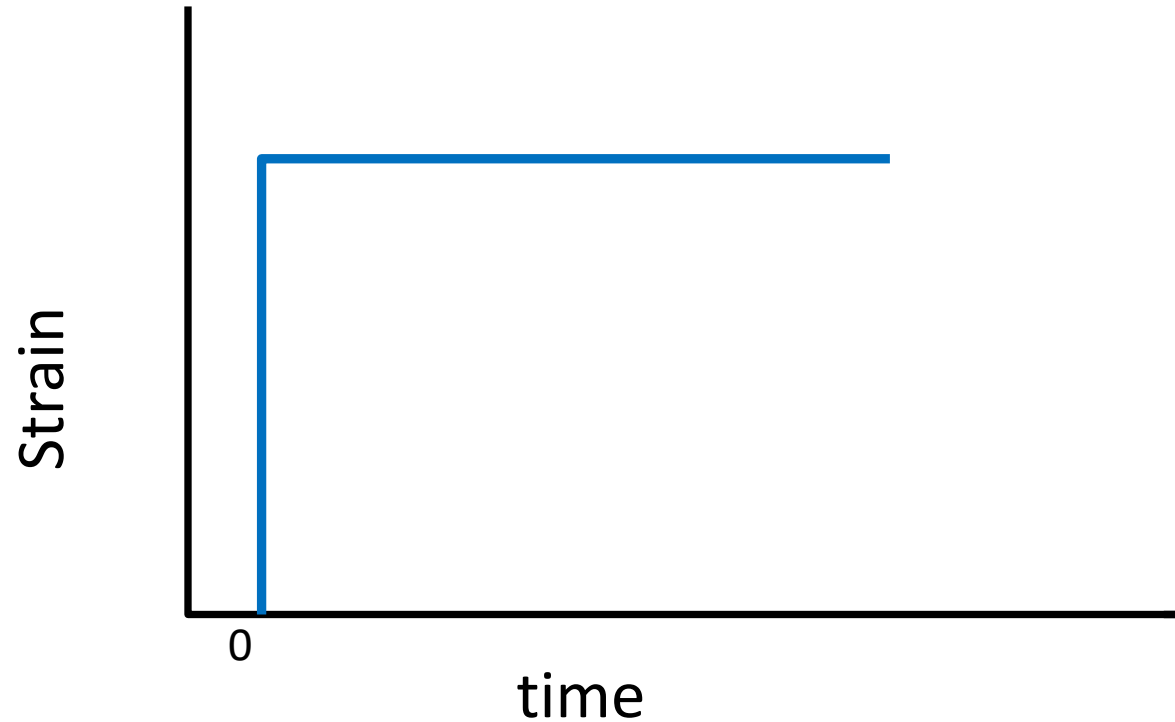
Instrument: Q800
Clamp: Tension
Temperature: 75°C
Stress: 5MPa

Creep: Material response

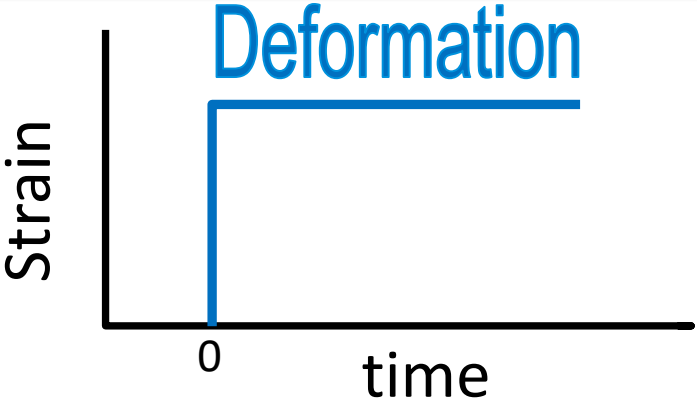


Stress relaxation experiment

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



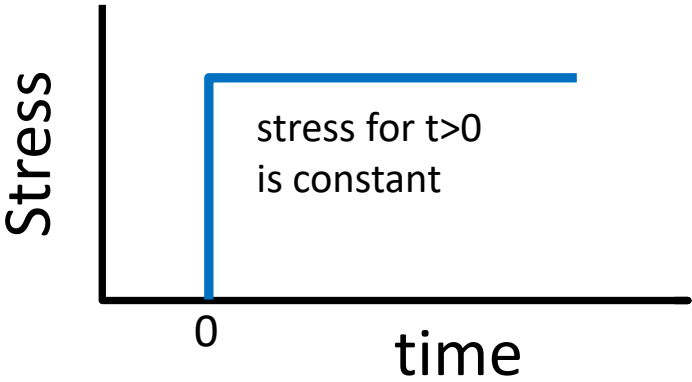
Stress relaxation experiment



Response of Classical Extremes

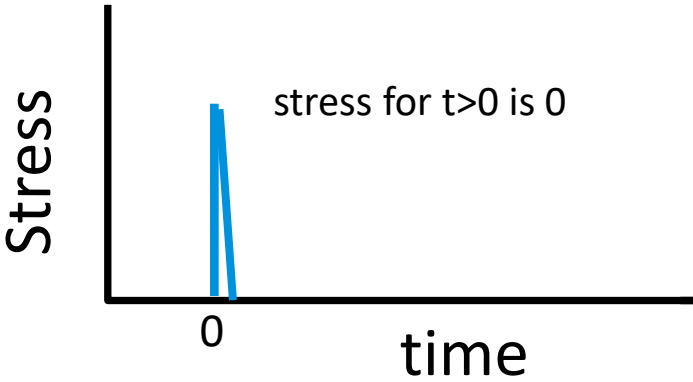
Elastic

Hookean Solid



Viscous

Newtonian Fluid



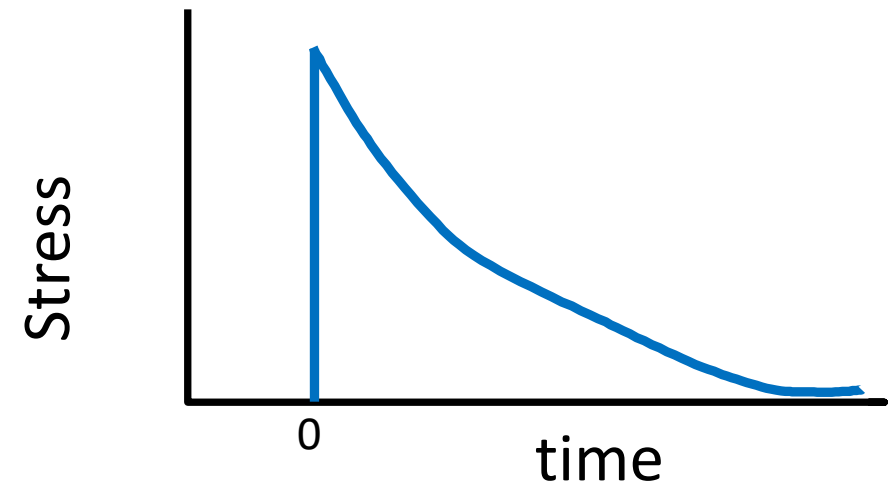
Stress relaxation experiment

Response of **ViscoElastic** Material

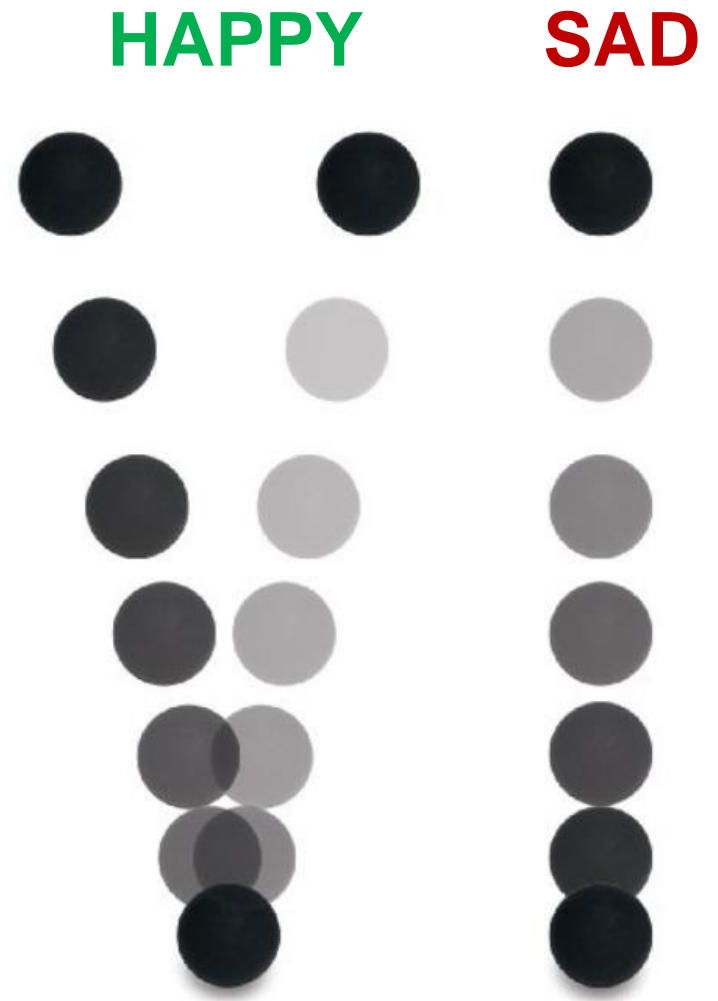
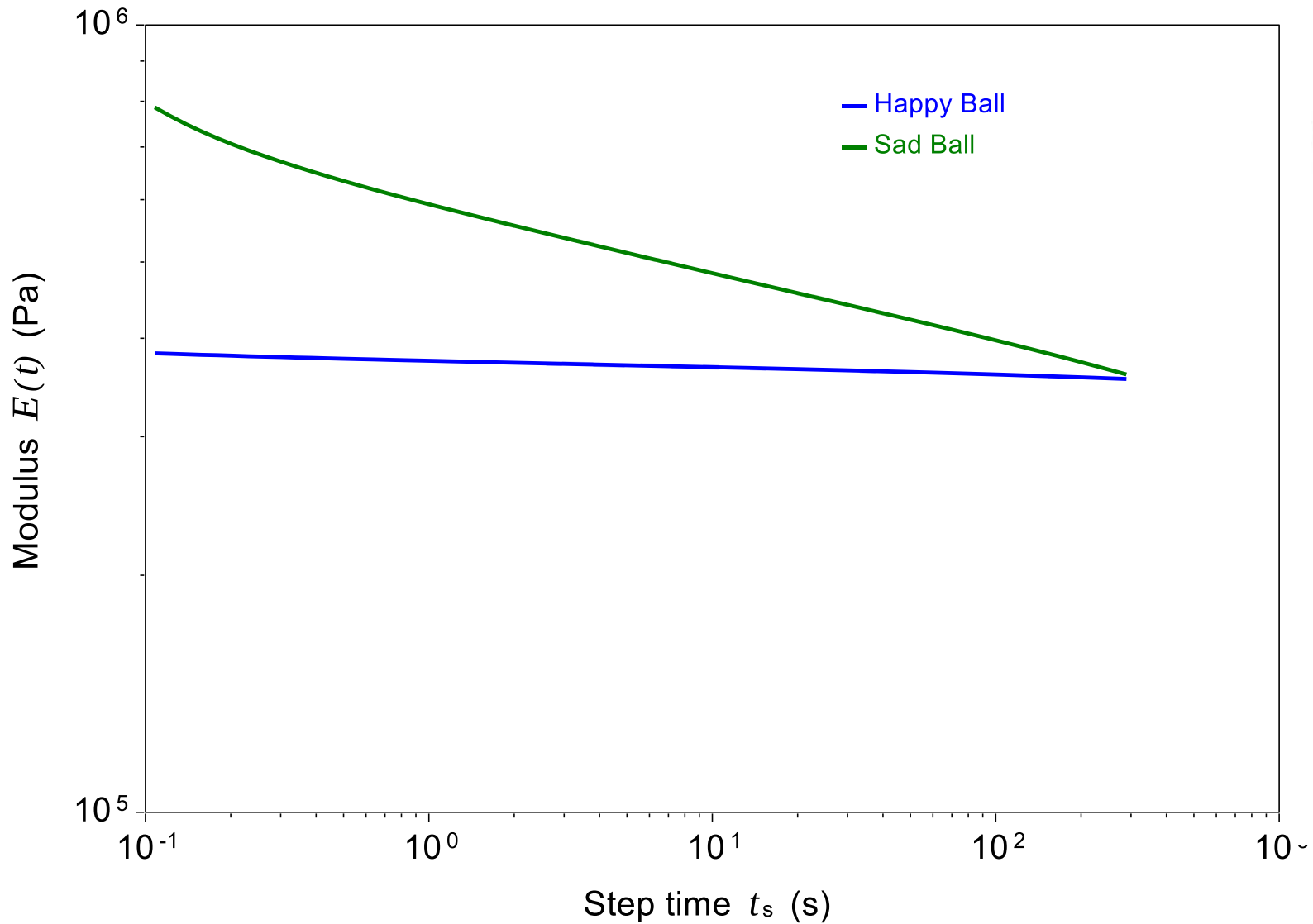
Stress decreases **with time** starting at some high value and decreasing to zero.

- 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, $E(t)$**

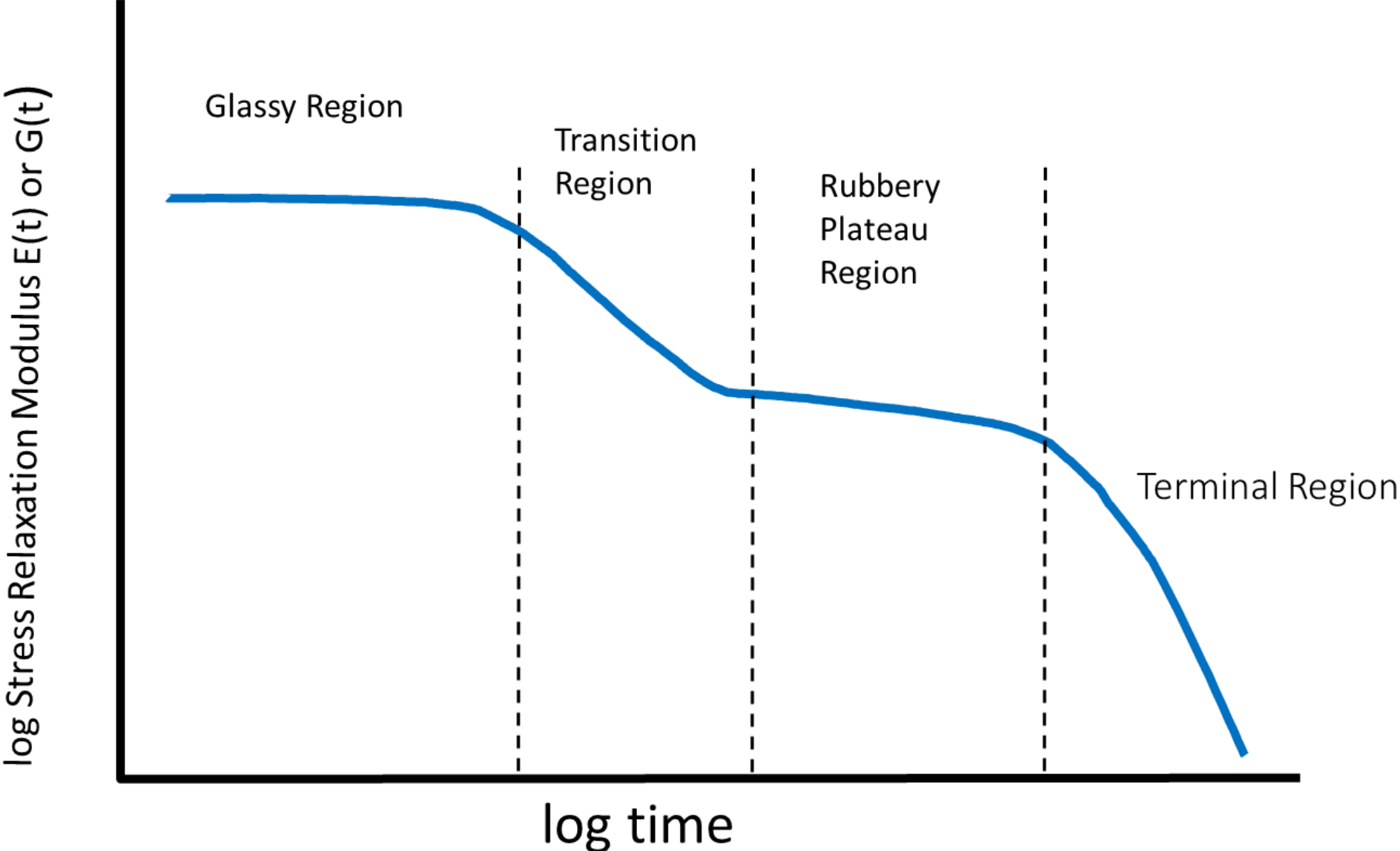
$$E(t) = \sigma(t)/\gamma$$



Stress Relaxation: Compression

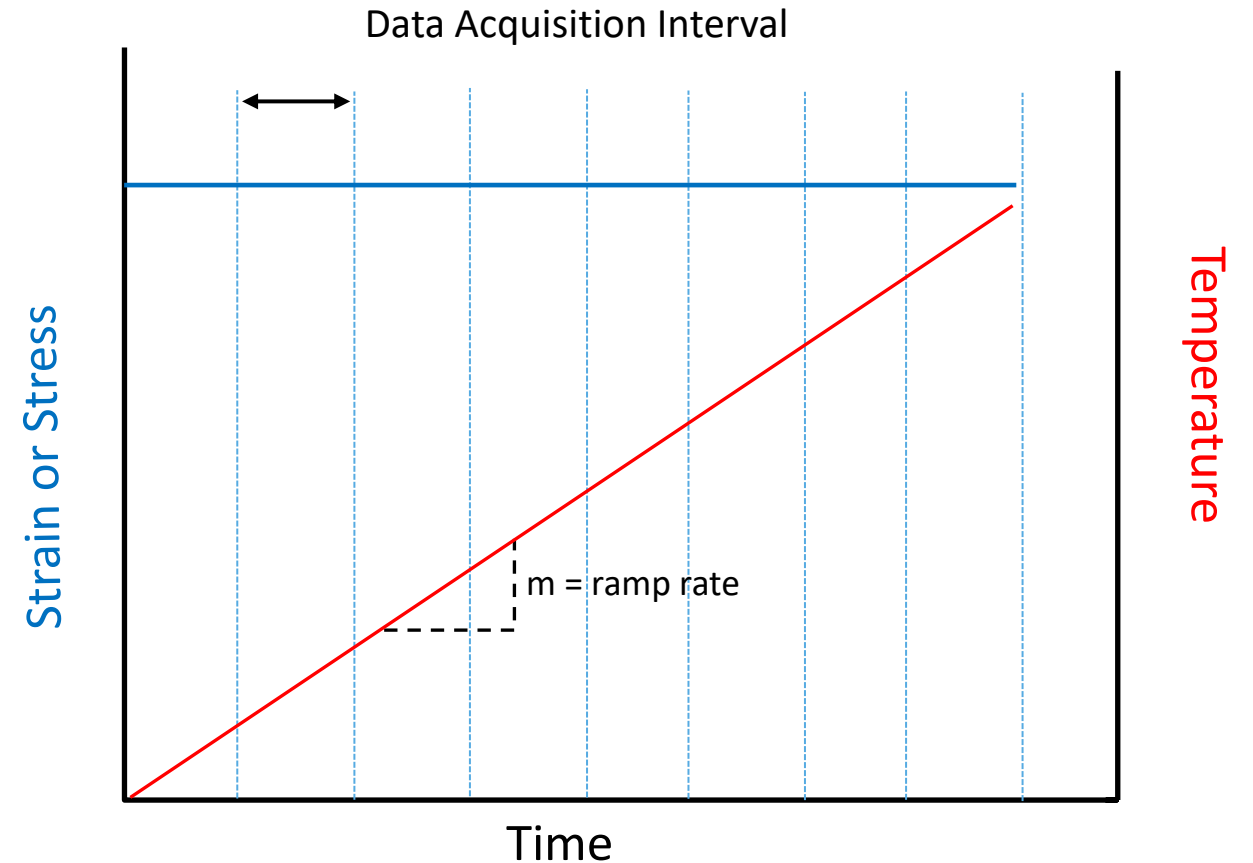


Stress Relaxation: Material response

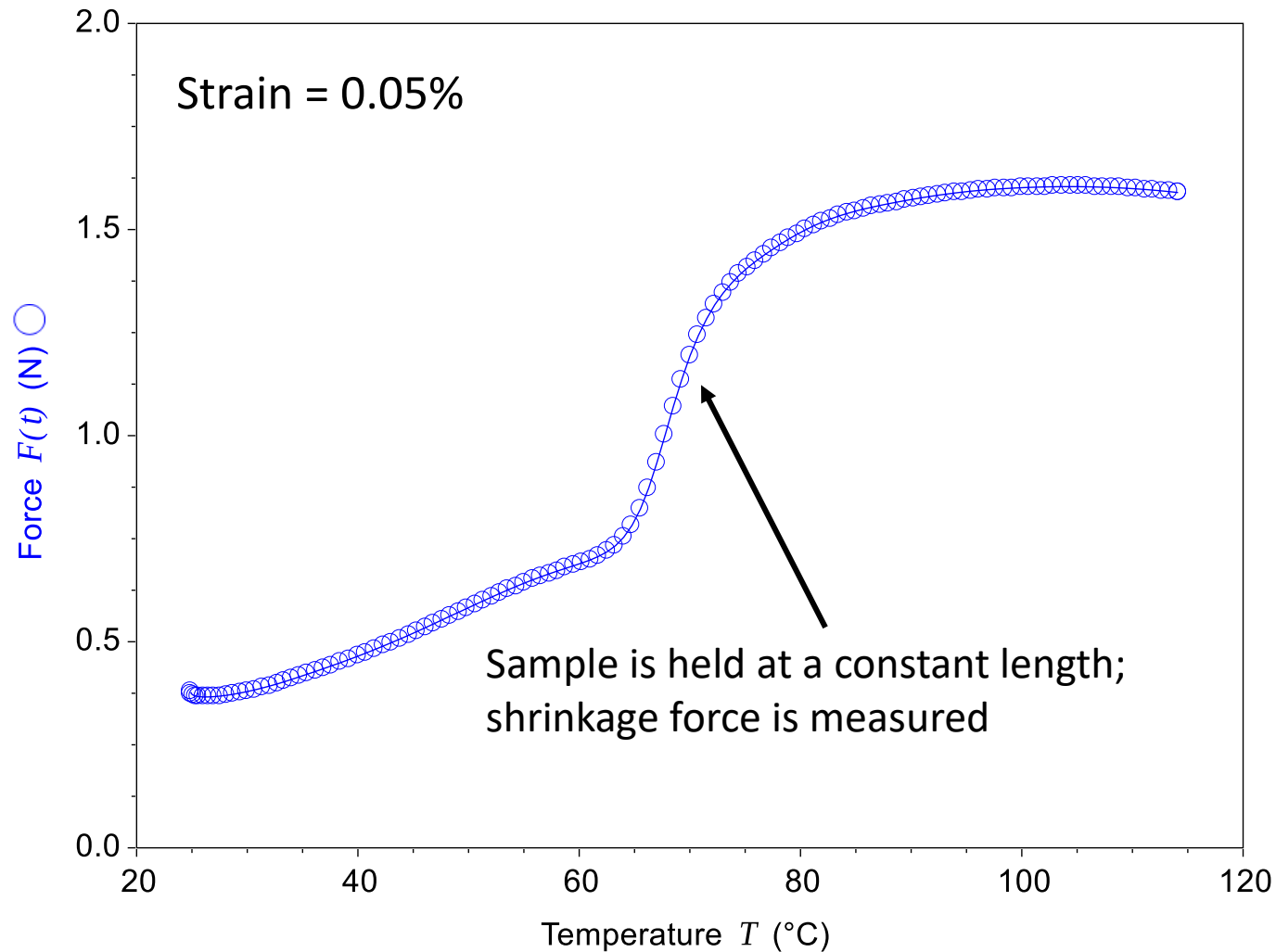


Iso-strain/Iso-stress Temperature Ramp

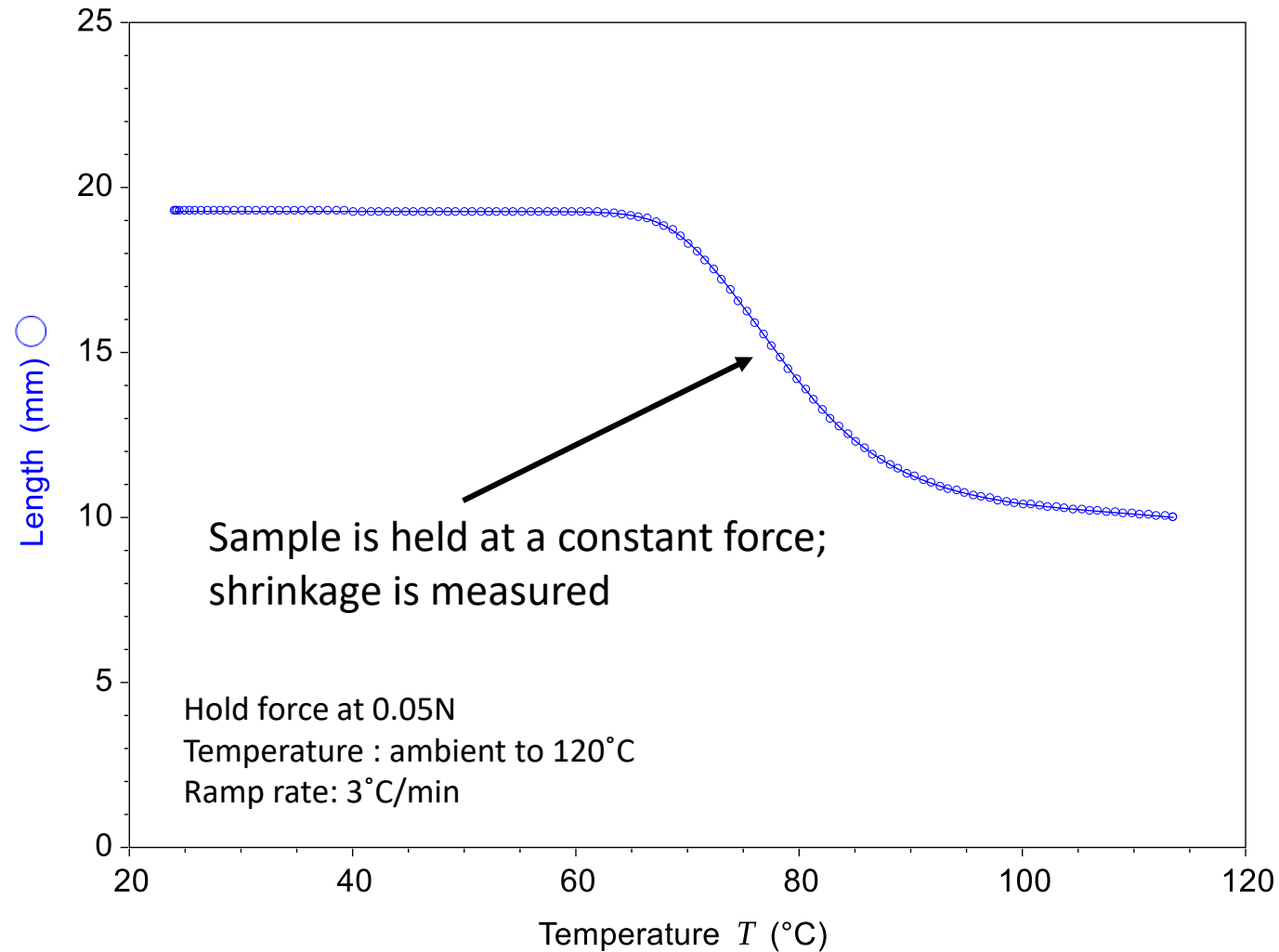
- The strain or stress is held at a constant value and a linear heating rate is applied.
- Valuable for assessing mechanical behavior under conditions of confined or fixed load (stress) or deformation (strain).
- Example: Measure sample shrinkage (length shrinkage or shrinking force)



Iso-Strain Temp Ramp: Measure Shrinking Force

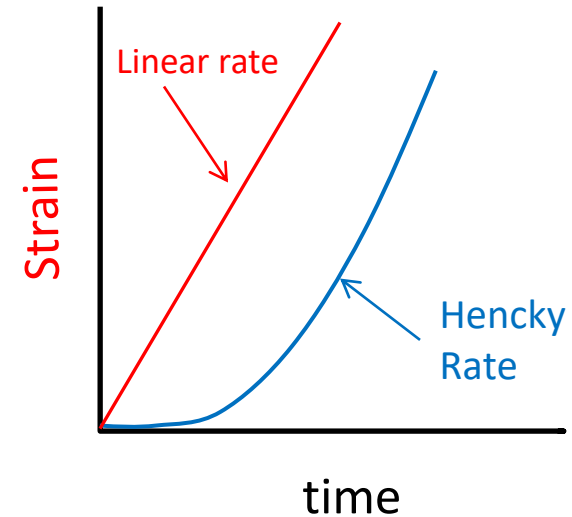
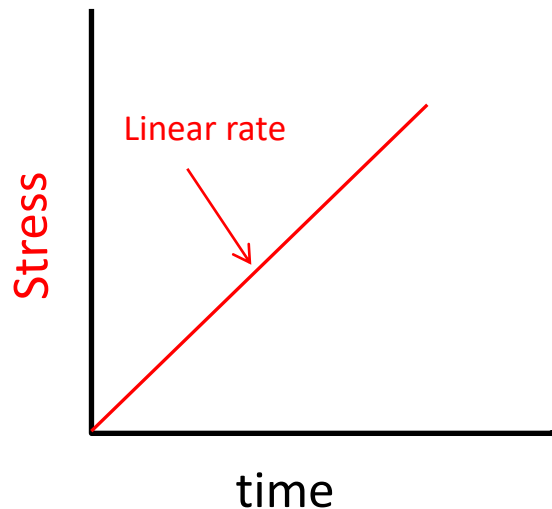


Iso-Force Temp Ramp: Measure Length Shrinkage

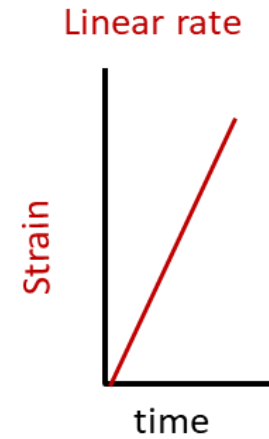
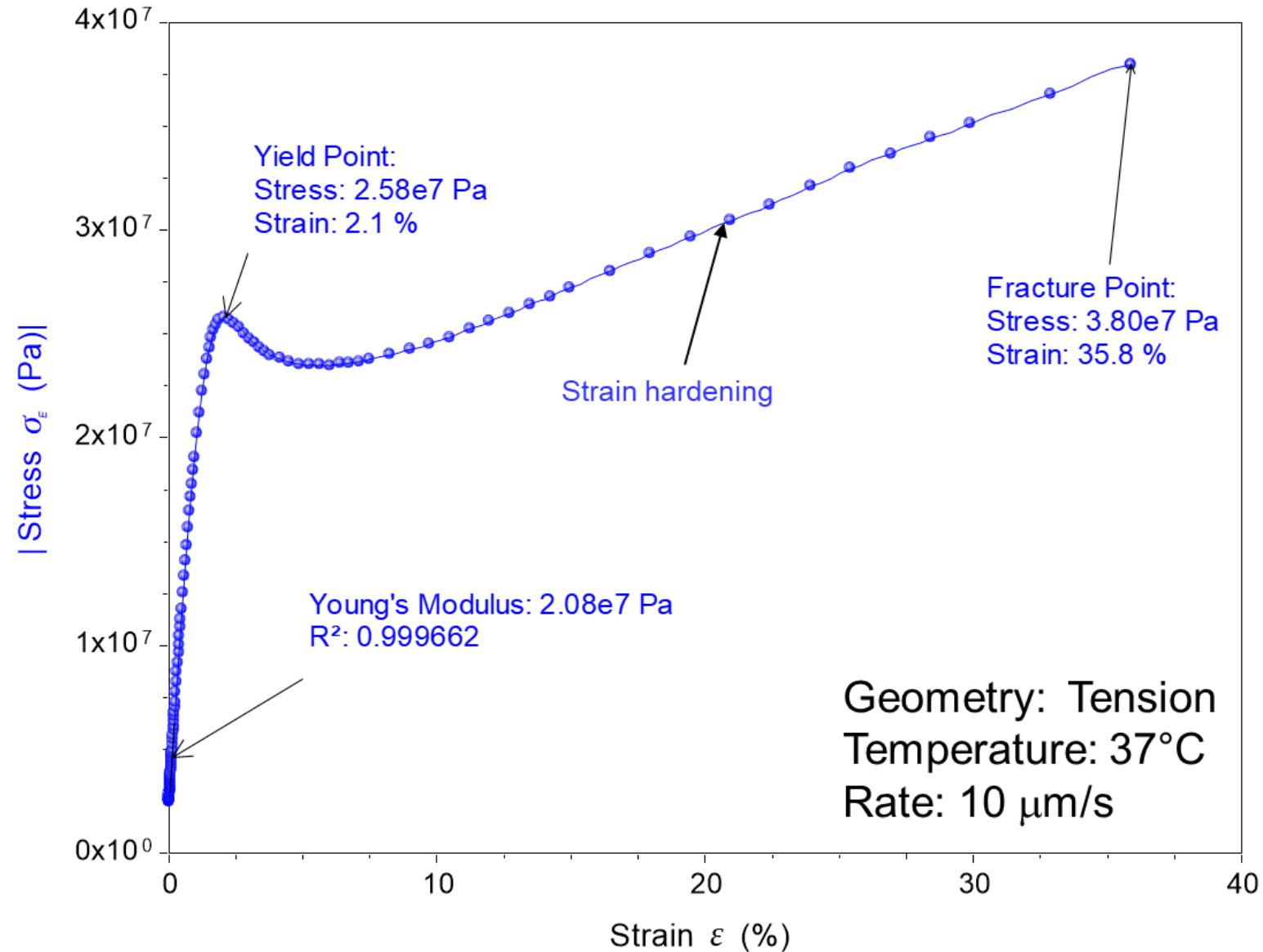


Stress-Strain Testing

- 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

Clamp: Film Clamp

Oscillation | Temperature Ramp

Amplitude	20.0	μm
Frequency	1.0	Hz
Initial/preload force	2.0	N
<input type="checkbox"/> Use Force Track	125.0	%

Use current temperature

Ramp from 35 °C to 150 °C

Ramp rate 3.0 °C/min

Soak times

at Start temperature 00:05:00 hh:mm:ss

at End temperature 00:00:00 hh:mm:ss

Estimated time to complete 00:38:20 hh:mm:ss

Test Settings

Post Test Conditions

- Force track

Clamp: Film Clamp

Oscillation | Temperature Ramp

Amplitude	20.0	μm
Frequency	1.0	Hz
Initial/preload force	0.1	N
<input checked="" type="checkbox"/> Use Force Track	125.0	%

Use current temperature

Ramp from 35 °C to 150 °C

Ramp rate 3.0 °C/min

Soak times

at Start temperature 00:05:00 hh:mm:ss

at End temperature 00:00:00 hh:mm:ss

Estimated time to complete 00:38:20 hh:mm:ss

Test Settings

Post Test Conditions

Offset force and force track on Q800

- Constant static force

Summary Procedure Notes

Procedure Information

Test: Temp Ramp / Freq Sweep

Notes: Material is heated at a constant rate. While heating, the material is deformed (oscillated) at a constant amplitude (strain) over a range of frequencies and the mechanical properties measured.

Temperature Ramp / Single Frequency

Amplitude: 15.0000 μm Strain: 0.0000 %

Preload force: 2.0000 N

Force track: 125 %

Start temperature: Use current: 35.00 $^{\circ}\text{C}$

Soak time: 5.00 min

Final temperature: 150.00 $^{\circ}\text{C}$

Ramp rate: 3.00 $^{\circ}\text{C}/\text{min}$

Hold time at final temperature: 30.00 min

Method / Frequency Table /

- Force track

Summary Procedure Notes

Procedure Information

Test: Temp Ramp / Freq Sweep

Notes: Material is heated at a constant rate. While heating, the material is deformed (oscillated) at a constant amplitude (strain) over a range of frequencies and the mechanical properties measured.

Temperature Ramp / Single Frequency

Amplitude: 15.0000 μm Strain: 0.0000 %

Preload force: 0.0100 N

Force track: 125 %

Start temperature: Use current: 35.00 $^{\circ}\text{C}$

Soak time: 5.00 min

Final temperature: 150.00 $^{\circ}\text{C}$


Ramp rate: 3.00 $^{\circ}\text{C}/\text{min}$

Hold time at final temperature: 30.00 min

Method / Frequency Table /

Offset force and force track on RSA G2

- Constant static force

Procedure: 

1: Conditioning Options

Axial force adjustment

Mode


Tension Compression

Axial force N Set initial value

Sensitivity N

Proportional force Mode

- Force track

Procedure: 

1: Conditioning Options

Axial force adjustment

Mode

Tension Compression

Axial force N Set initial value

Sensitivity N

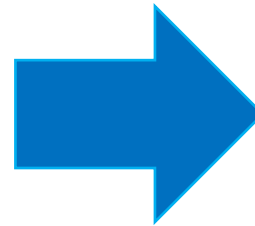
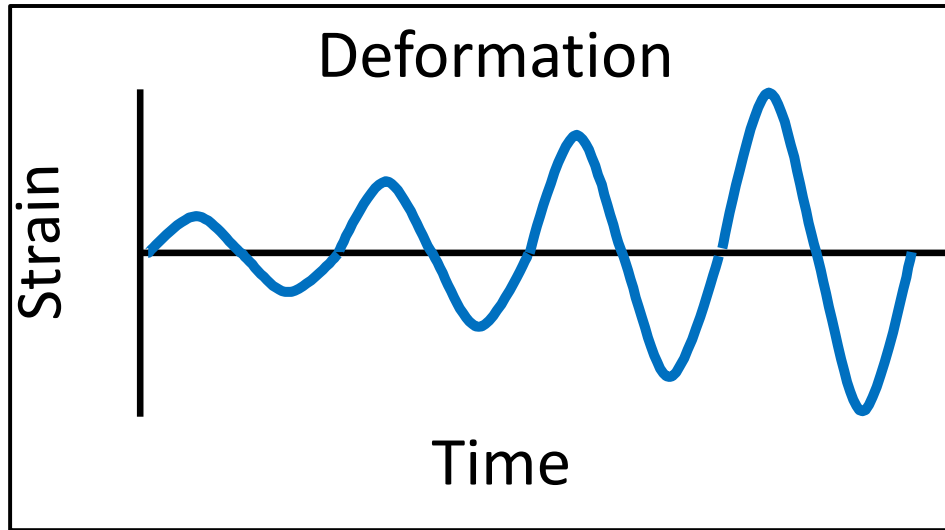
Proportional force Mode Compensate for modulus

Axial Force > Dynamic Force %

Minimum axial force N

Programmed Extension Below Pa

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

[Experiment 1]

Sample:

Clamp: Film Clamp

Oscillation ▾ Strain Sweep ▾

Temperature	<input type="text" value="25"/>	°C
Soak time	<input type="text" value="60.0"/>	s
Frequency	<input type="text" value="1.0"/>	Hz
Initial/preload force	<input type="text" value="0.1"/>	N
<input checked="" type="checkbox"/> Use Force Track	<input type="text" value="125.0"/>	%

Sweep Mode

Logarithmic Linear Discrete

Strain % to %

Points per decade

Number of Sweeps

Test Settings Post Test Conditions

Programming Strain Sweep on Q800

Mode: DMA Multi-Strain

Summary Procedure Notes

Procedure Information

Test: Strain Sweep

Notes: Material is held isothermally and deformed over a range of strains (amplitudes) at a single frequency.

Strain Sweep

Frequency: 1.00 Hz

Preload force: 0.0100 N

Force track 125. %

Advanced... Post Test...

Isothermal temperature: 35.00 °C

Soak time: 5.00 min

Number of sweeps: 1

Method Amplitude Table

Summary Procedure Notes

Procedure Information

Test: Strain Sweep

Notes: Material is held isothermally and deformed over a range of strains (amplitudes) at a single frequency.

Amplitude Table

Single Log Linear Discrete

Amplitude: 0.100 to 100.000 μm

Number of points: 19

	Amplitude
1	0.10
2	5.65
3	11.20
4	16.75
5	22.30
6	27.85
7	33.40
-	---

Refresh Table

Method Amplitude Table

Programming Strain Sweep on RSA G2

[Experiment 2]

- Sample: PET film LN2 only
- Geometry: Tension fixture (rectangle)
- Procedure of 2 steps
 - 1: Conditioning Options Active, Enabled
 - 2: Oscillation Amplitude

Environmental Control

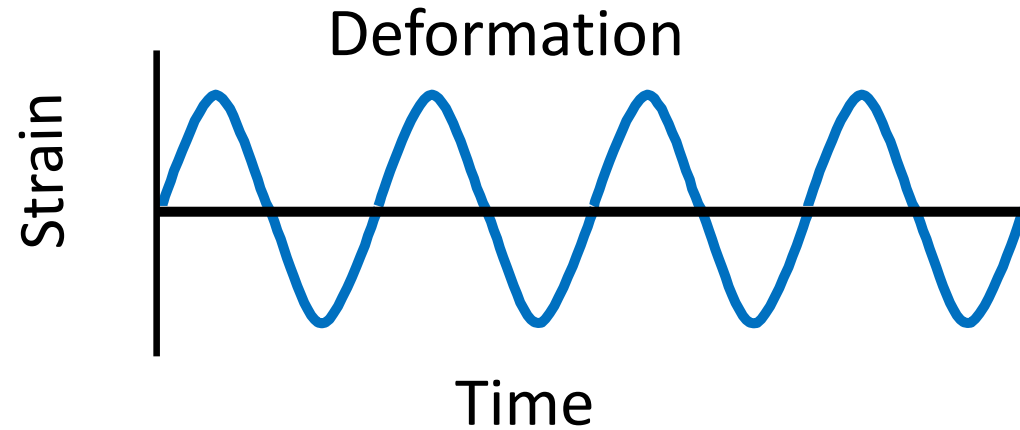
Temperature °C Inherit set point
Soak time s Wait for temperature

Test Parameters

Frequency Hz
Logarithmic sweep
Strain % to %
Points per decade

- Data acquisition
- Advanced

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

^ Oscillation v Time Sweep v

Temperature	<input type="text" value="25"/>	°C
Soak time	<input type="text" value="60.0"/>	s
Strain	<input type="text" value="0.5"/>	%
Frequency	<input type="text" value="1.0"/>	Hz
Duration	<input type="text" value="300.0"/>	s
Initial/preload force	<input type="text" value="0.1"/>	N
<input checked="" type="checkbox"/> Use Force Track	<input type="text" value="125.0"/>	%

Data sampling mode
 seconds/pt Total points

Sampling interval s/pt

Test Settings Post Test Conditions

- Fatigue example

^ Clamp: Film Clamp

^ Oscillation v Fatigue Test v

Temperature	<input type="text" value="25"/>	°C
Soak time	<input type="text" value="00:05:00"/>	hh:mm:ss
Amplitude	<input type="text" value="20.0"/>	µm
Frequency	<input type="text" value="1.0"/>	Hz
Initial/preload force	<input type="text" value="0.01"/>	N
<input checked="" type="checkbox"/> Use Force Track	<input type="text" value="125.0"/>	%
Total cycles	<input type="text" value="10000.0"/>	Cycles

Total time hh:mm:ss

Data sampling mode
 seconds/pt cycles/pt Total points

Sampling interval s/pt

Test Settings Post Test Conditions

Programming Time Sweep on Q800

Mode: DMA Multi-Frequency-Strain

Summary Procedure Notes

Procedure Information

Test: Custom

Notes:

Method

Amplitude : 20.0000 μm

Strain : 0.0000 %

Preload force: 0.0100 N

Force track 125. %

Advanced...
Post Test...

Name: Frequency sweep

Editor...

#	Segment Description
1	Data storage On
2	Isothermal for 5.00 min

\Method /Frequency Table/

Summary Procedure Notes

Procedure Information

Test: Custom

Notes:

Frequency Table

Single Log Linear Discrete

Frequency: 1.00 Hz

	Frequency
1	1.00
2	
3	
4	
5	
6	
7	
-	

Refresh Table

\Method /Frequency Table/

Programming Time Sweep on RSA G2

- Time sweep example

[Experiment 2]

Sample: PET film LN2 only

Geometry: Tension fixture (rectangle)

Procedure of 2 steps

- 1: Conditioning Options Active, Enabled
- 2: Oscillation Time

Environmental Control

Temperature 25 °C Inherit set point

Soak time 60.0 s Wait for temperature

Test Parameters

Duration 300.0 s

Sampling interval 10.0 s/pt

Strain % 0.5 %

Single point

Frequency 1.0 Hz

- Data acquisition
- Advanced

- Fatigue example

Procedure of 2 steps

- 1: Conditioning Options Active, Enabled
- 2: Oscillation Cycle Sweep

Environmental Control

Temperature 50 °C Inherit set point

Soak time 300.0 s Wait for temperature

Test Parameters

Total cycles 10000.0 Cycles

Total time 02:46:40 hh:mm:ss

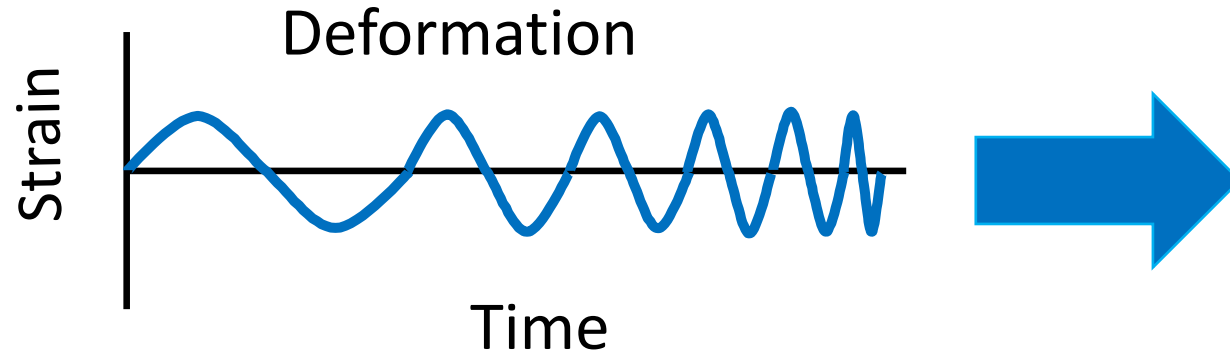
Measure every 2.0 Cycles

Strain % 2.0 %

Frequency 1.0 Hz

- Data acquisition

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

Programming Frequency Sweep on DMA850

[Experiment 1]

Sample:

Clamp: Film Clamp

Oscillation Frequency Sweep

Temperature	<input type="text" value="25"/>	°C	
Soak time	<input type="text" value="60.0"/>	s	
Strain	<input type="text" value="0.5"/>	%	
Initial/preload force	<input type="text" value="0.1"/>	N	
<input checked="" type="checkbox"/> Use Force Track	<input type="text" value="125.0"/>	%	
Sweep Mode			
<input checked="" type="radio"/> Logarithmic <input type="radio"/> Linear <input type="radio"/> Discrete			
Frequency	<input type="text" value="0.1"/>	Hz to <input type="text" value="50.0"/>	Hz
Points per decade	<input type="text" value="5"/>		
Number of Sweeps	<input type="text" value="1"/>		

Test Settings Post Test Conditions

Programming Frequency Sweep on Q800

Mode: DMA Multi-Frequency-Strain

Summary Procedure Notes

Procedure Information

Test: Isothermal Temp / Freq Sweep

Notes: Material is held isothermally at a user-specified temperature. Then it is deformed (oscillated) at a constant amplitude (strain) over one or more frequencies and the mechanical properties measured.

Frequency Sweep

Amplitude : 25.0000 μm Strain : 0.0000 %

Preload force: 0.0100 N

Force track 125. %

Advanced...
Post Test...

Isothermal temperature: 35.00 $^{\circ}\text{C}$

Soak time: 5.00 min

Number of sweeps: 1

Method / Frequency Table /

Summary Procedure Notes

Procedure Information

Test: Isothermal Temp / Freq Sweep

Notes: Material is held isothermally at a user-specified temperature. Then it is deformed (oscillated) at a constant amplitude (strain) over one or more frequencies and the mechanical properties measured.

Frequency Table

Single Log Linear Discrete

Frequency: 0.10 to 10.00 Hz

Points per decade: 5

	Frequency
1	0.10
2	0.16
3	0.25
4	0.40
5	0.63
6	1.00
7	1.60
...	...

Refresh Table

Method / Frequency Table /

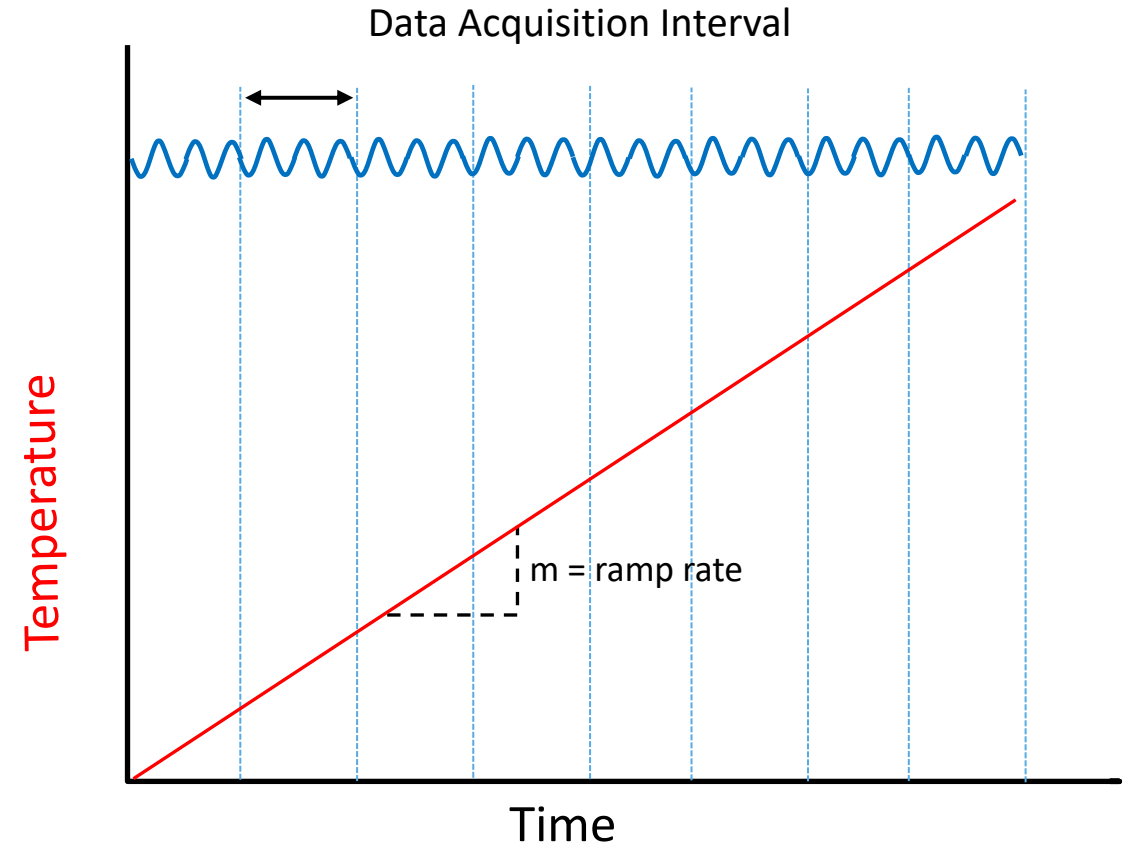
Programming Frequency Sweep on RSA G2

[Experiment 2]

- Sample: PET film LN2 only
- Geometry: Tension fixture (rectangle)
- Procedure of 2 steps
 - 1: Conditioning Options Active, Enabled
 - 2: Oscillation Frequency
 - Environmental Control
 - Temperature: 25 °C Inherit set point
 - Soak time: 60.0 s Wait for temperature
 - Test Parameters
 - Strain %: 0.5 %
 - Logarithmic sweep
 - Frequency: 0.1 to 50.0 Hz
 - Points per decade: 5
- Data acquisition
- Advanced

Dynamic Temperature Ramp

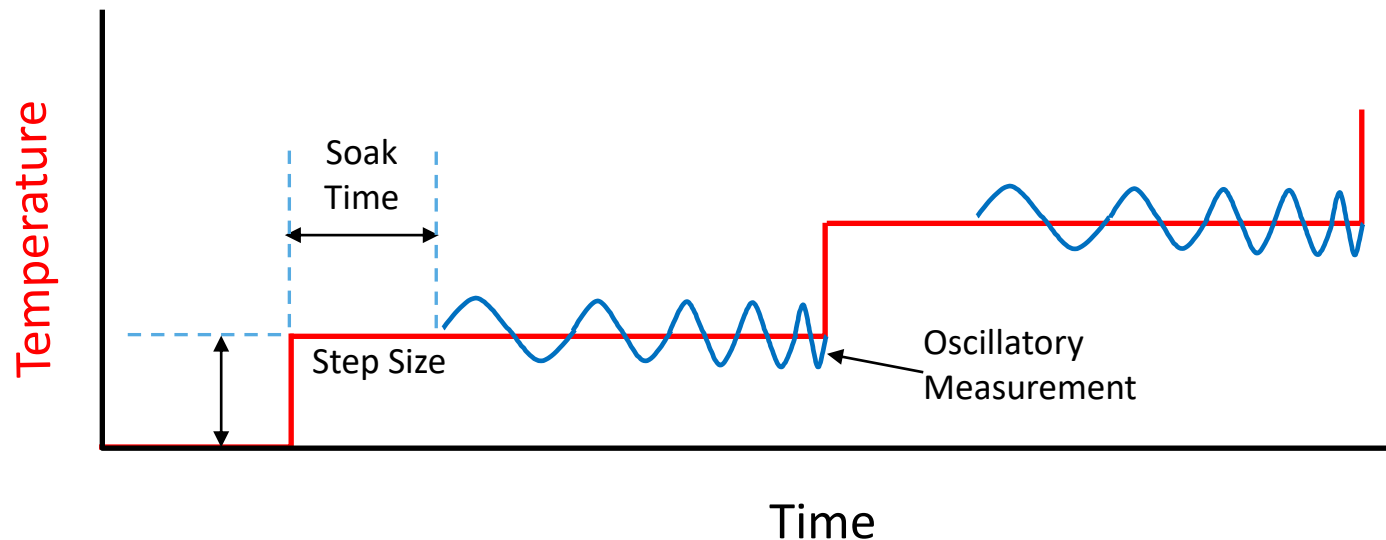
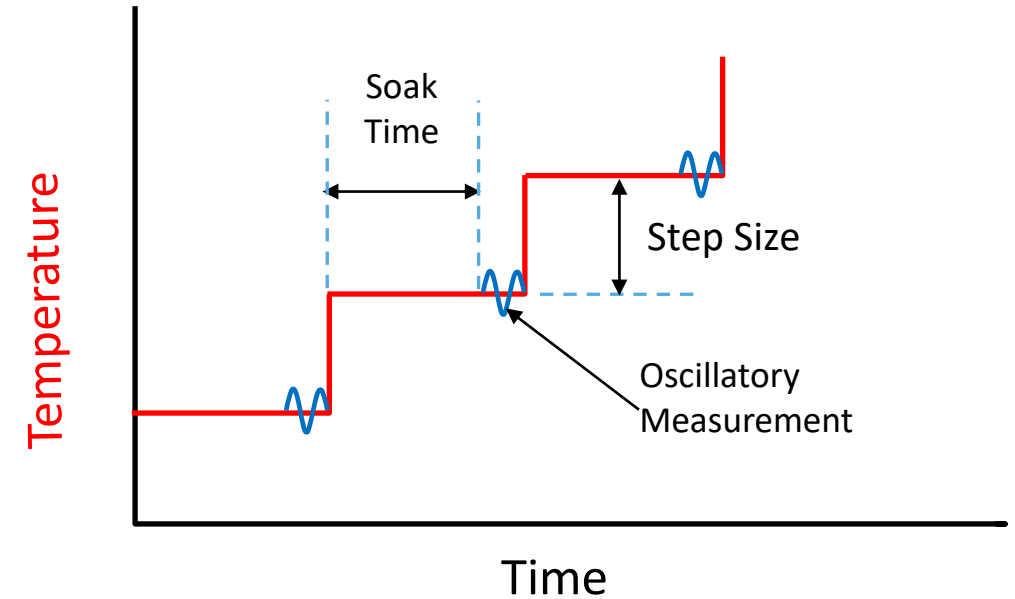
- A linear heating rate is applied. The material response is monitored at a constant frequency and constant amplitude of deformation. Data is taken at user defined time intervals.



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

⤴ Oscillation ▾ Temperature Ramp ▾

Strain	<input type="text" value="0.05"/>	%	
Frequency	<input type="text" value="1.0"/>	Hz	
Initial/preload force	<input type="text" value="0.1"/>	N	
<input checked="" type="checkbox"/> Use Force Track	<input type="text" value="125.0"/>	%	
<input type="checkbox"/> Use current temperature			
Ramp from	<input type="text" value="-100"/>	°C to <input type="text" value="200"/>	°C
Ramp rate	<input type="text" value="3.0"/>	°C/min	
Soak times			
at Start temperature	<input type="text" value="300.0"/>	s	
at End temperature	<input type="text" value="0.0"/>	s	

Estimated time to complete 01:40:00 hh:mm:ss

- Temp Sweep Example

⤴ Oscillation ▾ Temperature Sweep (Multifrequency) ▾

Strain	<input type="text" value="0.05"/>	%	
Initial/preload force	<input type="text" value="0.1"/>	N	
<input checked="" type="checkbox"/> Use Force Track	<input type="text" value="125.0"/>	%	
Sweep from	<input type="text" value="-100"/>	°C to <input type="text" value="200"/>	°C
Temperature increment	<input type="text" value="10"/>	°C	
Soak time	<input type="text" value="300.0"/>	s	
Sweep Mode			
<input checked="" type="radio"/> Logarithmic	<input type="radio"/> Linear	<input type="radio"/> Discrete	
Frequency	<input type="text" value="0.1"/>	Hz to <input type="text" value="10.0"/>	Hz
Points per decade	<input type="text" value="5"/>		

Note: Measurement can be done with single or multiple frequencies

Programming Temp Ramp/Step on Q800

Mode: DMA Multi-Frequency-Strain

- Temp Ramp Example

The screenshot shows the software interface for programming a Temp Ramp example. The 'Test' dropdown is set to 'Temp Ramp / Freq Sweep'. The 'Notes' field contains the text: 'Material is heated at a constant rate. While heating, the material is deformed (oscillated) at a constant amplitude (strain) over a range of frequencies and the mechanical properties measured.' The 'Temperature Ramp / Single Frequency' section includes the following settings: Amplitude (radio button selected) is 25.0000 μm ; Strain (radio button unselected) is 0.0000 %; Preload force is 0.0100 N; Force track (checkbox checked) is 125 %; Start temperature is -150.00 $^{\circ}\text{C}$ (with 'Use current' checkbox unselected); Soak time is 5.00 min; Final temperature is 150.00 $^{\circ}\text{C}$; Ramp rate is 3.00 $^{\circ}\text{C}/\text{min}$; and Hold time at final temperature is 30.00 min. There are 'Advanced...' and 'Post Test...' buttons. At the bottom, there is a link for 'Method / Frequency Table'.

- Temp Sweep Example

The screenshot shows the software interface for programming a Temp Sweep example. The 'Test' dropdown is set to 'Temp Step / Freq Sweep'. The 'Notes' field contains the text: 'Material is exposed to a series of increasing isothermal temperatures. At each temperature, the material is deformed at a constant amplitude (strain) over one or more frequencies and the mechanical properties'. The 'Method' section includes the following settings: Amplitude (radio button selected) is 15.0000 μm ; Strain (radio button unselected) is 0.0000 %; Preload force is 0.0100 N; Force track (checkbox checked) is 125 %; Start temperature is -100.00 $^{\circ}\text{C}$; Final temperature is 250.00 $^{\circ}\text{C}$; Temperature increment is 10.00 $^{\circ}\text{C}$; and Isothermal soak time is 5.00 min. There are 'Advanced...' and 'Post Test...' buttons. At the bottom, there is a link for 'Method / Frequency Table'.

Note: Measurement can be done with single or multiple frequencies

Programming Temp Ramp/Sweep on RSA G2

- Temp Ramp Example

Sample: PET film LN2 only

Geometry: Tension fixture (rectangle)

Procedure of 2 steps

- 1: Conditioning Options Active, Enabled
- 2: Oscillation Temperature Ramp

Environmental Control

Start temperature	-100 °C	<input type="checkbox"/> Inherit set point
Soak time	300.0 s	<input type="checkbox"/> Wait for temperature
Ramp rate	3.0 °C/min	
End temperature	200 °C	
Soak time after ramp	0 s	
Estimated time to complete	01:40:00	hh:mm:ss

Test Parameters

Sampling interval	10.0 s/pt	▼
Strain %	0.05 %	▼
Single point		▼
Frequency	1.0 Hz	▼

- Data acquisition
- Advanced

- Temp Sweep Example

Procedure of 2 steps

- 1: Conditioning Options Active, Enabled
- 2: Oscillation Temperature Sweep

Environmental Control

Start temperature	-100 °C	<input type="checkbox"/> Inherit
Soak time	300.0 s	<input type="checkbox"/> Wait for temperature
End temperature	200 °C	
Temperature step	10 °C	
Step soak time	300.0 s	

Test Parameters

Strain %	0.02 %	▼
Logarithmic sweep		▼
Frequency	0.1 to 10.0 Hz	▼
Points per decade	5	

- Data acquisition
- Advanced

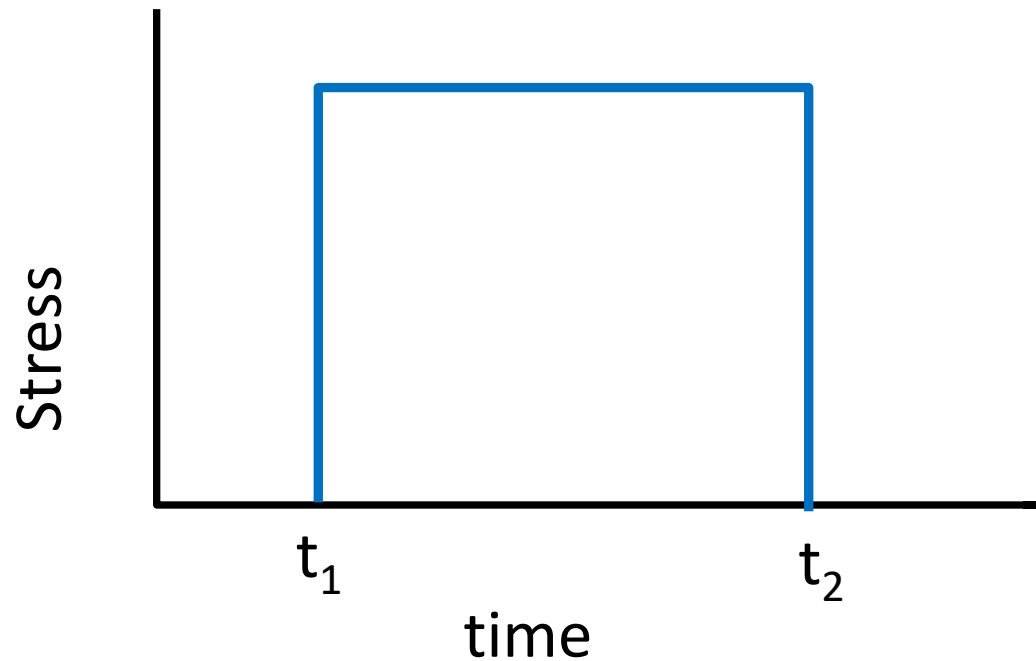
Note: Measurement can be done with single or multiple frequencies

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

[Experiment 1]

Sample:

Clamp: Film Clamp

Stress Control Creep Recovery

Temperature	<input type="text" value="30"/>	°C
Soak time	<input type="text" value="60.0"/>	s
Preload force	<input type="text" value="0.01"/>	N
Stress	<input type="text" value="500.0"/>	Pa
Creep time	<input type="text" value="120.0"/>	s
Recovery time	<input type="text" value="240.0"/>	s

Data sampling mode

Linear Log

Sampling rate pts/s

Test Settings Post Test Conditions

Programming Creep Recovery on a Q800

Summary | Procedure | Notes

Procedure

Mode: DMA Creep

Test: Custom

Clamp / Sample

Clamp: Creep

Sample Shape: rectangular (l, w, t)

Dimensions: 17.5000 mm 12.9000 mm 3.2000 mm

Sample Information

Sample Name: Rynite 530 SC

Comments:

Data File: \\Demolab8-w2k\TA\Data\DMA\Smith\DuPont\Mimi\

Network Drive

Start Remotely

Autoanalyze

Analysis Macro:

Creep

Preload force: 0.0010 N

Stress: 1.0000 MPa

Advanced...

Post Test...

Isothermal temperature: 35.00 °C

Soak time: 5.00 min

Creep time: 10.00 min

Recovery time: 20.00 min

#	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 20.00 min


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

✍ [Experiment 2] _____

▼ Sample: PET film LN2 only

▼ Geometry: Tension fixture (rectangle)

▲ Procedure of 2 steps 

▲ 1: Conditioning Stress Control


Load Precomputed Run and Calculate

Environmental Control

Temperature °C Inherit set point

Soak time s Wait for temperature

Test Parameters

Strain % % 

Save stress control PID file

Stress control PID file path:

▼ Data acquisition

▼ 2: Step (Transient) Creep 25°C, 60s, 100Pa

Programming Creep Recovery on a RSA-G2

Creep

[Experiment 2]

Sample: PET film LN2 only

Geometry: Tension fixture (rectangle)

Procedure of 3 steps

1: Conditioning Stress Control 30°C

2: Step (Transient) Creep

Environmental Control

Temperature: 30 °C Inherit set point

Soak time: 60.0 s Wait for temperature

Test Parameters

Duration: 180.0 s

Tension Compression

Stress: 500.0 Pa

Sampling Linear Log

Number of points: 200

Steady state sensing

Data acquisition

Advanced

3: Step (Transient) Creep 360s, 0Pa

Recovery

[Experiment 2]

Sample: PET film LN2 only

Geometry: Tension fixture (rectangle)

Procedure of 3 steps

1: Conditioning Stress Control 30°C

2: Step (Transient) Creep 30°C, 180s, 500Pa

3: Step (Transient) Creep

Environmental Control

Temperature: 30 °C Inherit set point

Soak time: 0 s Wait for temperature

Test Parameters

Duration: 360.0 s

Tension Compression

Stress: 0 Pa

Sampling Linear Log

Number of points: 200

Steady state sensing

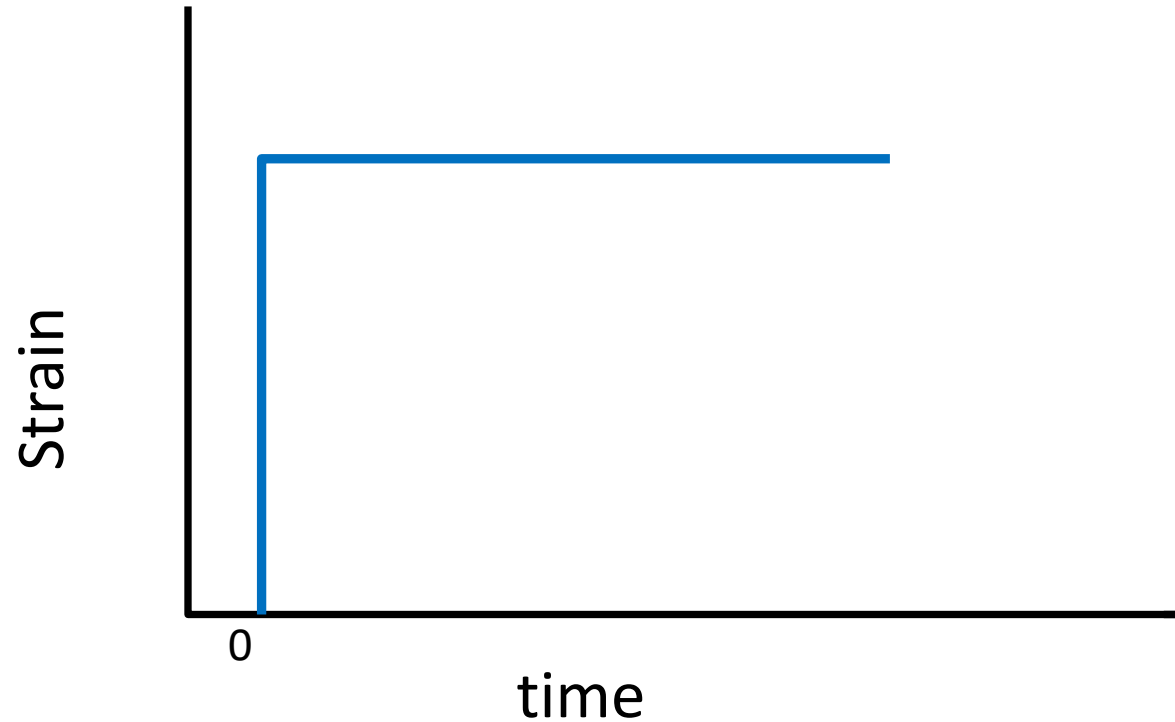
Data acquisition

Advanced

- 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

[Experiment 5]

Sample:

Clamp: Film Clamp

Strain Control Stress Relaxation

Temperature	50	°C
Soak time	120.0	s
Preload force	0.01	N
Strain	1.0	%
Relaxation time	600.0	s
Recovery time	0.0	s

Data sampling mode

Linear Log

Sampling rate 1.0 pts/s

Test Settings Post Test Conditions

Programming Stress Relaxation on a Q800

Summary | Procedure | Notes

Procedure

Mode: DMA Stress Relaxation

Test: Custom

Clamp / Sample: Custom

Clamp: Stress Relaxation
Stress Relaxation TTS
Single Cantilever

Sample Shape: rectangular (l, w, t)

Dimensions: 17.5000 mm 12.9000 mm 3.2000 mm

Sample Information

Sample Name: Rynite 530 SC

Comments:

Data File: \\Demolab8-w2k\TA\Data\DMA\Smith\DuPont\Mimi\

Network Drive

Start Remotely

Autoanalyze

Analysis Macro:

Stress Relaxation

Preload force: 0.0010 N

Strain: 0.1000 %

Advanced...

Post Test...

Isothermal temperature: 35.00 °C

Soak time: 5.00 min

Relaxation time: 10.00 min

Recovery time: 0.00 min

#	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

Programming Stress Relaxation on a RSA-G2

2: Step (Transient) Stress Relaxation

Environmental Control

Temperature °C Inherit set point
Soak time s Wait for temperature

Test Parameters

Duration s
 Tension Compression
Strain % %

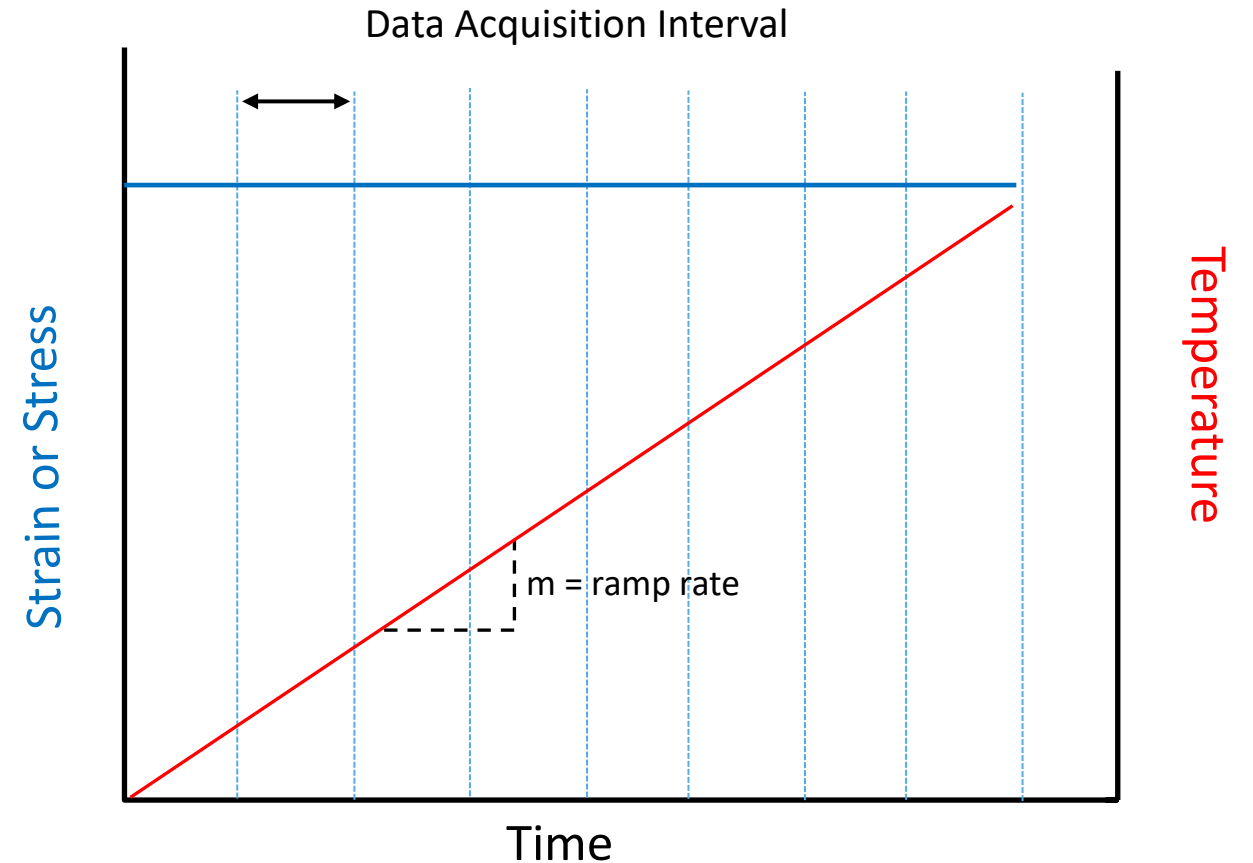
Sampling Linear Log
Number of points

Data acquisition

Advanced

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

Sample: [dropdown]
Clamp: Film Clamp

Strain Control ▾ IsoStrain ▾

Preload force	<input type="text" value="0.01"/>	N
Displacement	<input type="text" value="20.0"/>	μm
Sampling rate	<input type="text" value="1.0"/>	pts/s

Use current temperature

Ramp from °C to °C

Ramp rate °C/min

Soak times

at Start temperature s

at End temperature s

Estimated time to complete 00:41:40 hh:mm:ss

Test Settings Post Test Conditions

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

Sample: [dropdown]
Clamp: Film Clamp

Stress Control ▾ IsoStress ▾

Preload force	<input type="text" value="0.01"/>	N
Stress	<input type="text" value="500.0"/>	Pa
Sampling rate	<input type="text" value="1.0"/>	pts/s

Use current temperature

Ramp from °C to °C

Ramp rate °C/min

Soak times

at Start temperature s

at End temperature s

Estimated time to complete 00:41:40 hh:mm:ss

Test Settings Post Test Conditions

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

The screenshot shows the software interface for configuring a DMA test. The 'Procedure Information' section has 'Isostrain' selected in the 'Test' dropdown. The 'Notes' field contains the text: 'A constant deformation (strain) is applied to the sample and the force (stress) required to maintain that deformation is monitored while ramping temperature.' Below this, the 'Isostrain' section is highlighted with a red box and contains the following fields: 'Preload force:' with a value of 0.0010 N, and 'Strain:' with a value of 0.1000 %. To the right of these fields are buttons for 'Advanced...' and 'Post Test...'. At the bottom, the 'Start temperature:' is 35.00 °C (with 'Use current' checked), 'Final temperature:' is 250.00 °C, and 'Ramp rate:' is 3.00 °C/min.

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

The screenshot shows the software interface for configuring a DMA test. The 'Procedure Information' section has 'Temp Ramp / Controlled Force' selected in the 'Test' dropdown. The 'Notes' field contains the text: 'Material is exposed to a specific stress (force) and the resultant strain (dimension change) is monitored while the temperature is ramped at a constant linear rate.' Below this, the 'Temperature Ramp / Controlled Force' section is highlighted with a red box and contains the following fields: 'Preload force:' with a value of 0.0100 N. To the right of these fields are buttons for 'Advanced...' and 'Post Test...'. At the bottom, the 'Start temperature:' is 25.00 °C, 'Final temperature:' is 200.00 °C, and 'Ramp rate:' is 3.0 °C/min.

Iso-Strain/Iso-Stress on a RSA G2

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

Sample: PET film LN2 only

Geometry: Tension fixture (rectangle)

Procedure of 1 step

1: Other Temp Ramp IsoStrain

Environmental Control

Start temperature	20 °C	<input type="checkbox"/> Inherit set point
Soak time	180.0 s	<input checked="" type="checkbox"/> Wait for temperature
Ramp rate	3.0 °C/min	
End temperature	200 °C	
Soak time after ramp	0 s	
Estimated time to complete	01:00:00	hh:mm:ss

Test Parameters

Sampling rate	1.0 pts/s
Motor direction	<input checked="" type="radio"/> Tension <input type="radio"/> Compression
Strain %	0.1 %
Maximum force	20.0 N

Data acquisition

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

Sample: PET film LN2 only

Geometry: Tension fixture (rectangle)

Procedure of 1 step

1: Other Temp Ramp IsoForce

Environmental Control

Start temperature	20 °C	<input type="checkbox"/> Inherit set point
Soak time	180.0 s	<input checked="" type="checkbox"/> Wait for temperature
Ramp rate	3.0 °C/min	
End temperature	200 °C	
Soak time after ramp	0 s	
Estimated time to complete	01:00:00	hh:mm:ss

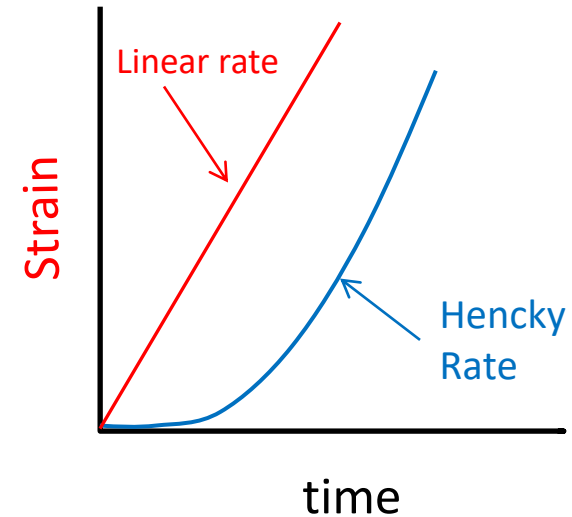
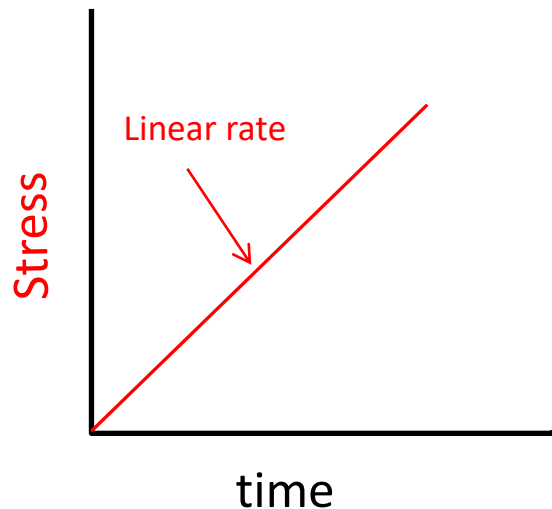
Test Parameters

Sampling rate	1.0 pts/s
Motor direction	<input checked="" type="radio"/> Tension <input type="radio"/> Compression
Constant axial force	0.01 N

Data acquisition

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

Sample: []

Clamp: Film Clamp

Rate Control ▾ Strain Ramp ▾

Temperature °C

Soak time s

Preload force N

Inherit starting displacement

Ramp from μm to μm

Ramp rate μm/min

Data sampling mode

Linear Log

Sampling rate pts/s

Test Settings Post Test Conditions

Stress Ramp

Sample: []

Clamp: Film Clamp

Rate Control ▾ Stress Ramp ▾

Temperature °C

Soak time s

Preload force N

Inherit starting force

Ramp from N to N

Ramp rate N/min

Data sampling mode

Linear Log

Sampling rate pts/s

Test Settings Post Test Conditions

Strain/Stress Ramp on a Q800

- DMA strain rate mode
- Strain ramp
- Displacement ramp

Summary Procedure Notes

Procedure Information

Test: Displacement Ramp

Notes: Sample is exposed to a constant rate of displacement while the temperature is held isothermal.

Displacement Ramp

Preload force: 0.01 N

Initial Strain: 1.0000 %

Initial Displacement: 0.0010 μm

Isothermal temperature: 35.00 °C

Displacement Rate: 2000.00 μm/min

Final Displacement: 10000.0 μm

- DMA control force mode
- Force ramp

Summary Procedure Notes

Procedure Information

Test: Stress / Strain

Notes: Sample is exposed to a ramped force (stress) and the resultant deformation (strain) is monitored. Temperature is held isothermal.

Stress - Strain

Preload force: 0.01 N

Isothermal temperature: 50.00 °C

Soak time: 5.00 min

Force ramp rate: 1.0 N/min

Upper force limit: 18.000 N

Strain Ramp on a RSA G2

- Linear strain rate

Sample: PET film LN2 only

Geometry: Tension fixture (rectangle)

Procedure of 1 step

1: Other Axial

Environmental Control

Temperature °C Inherit set point

Soak time s Wait for temperature

Test Parameters

Duration s

Motor direction Tension Compression

Constant linear rate mm/s

Maximum gap change mm

Sampling Linear Log

Sampling rate pts/s

Data acquisition Save image

- Hencky strain rate

Sample: PET film LN2 only

Geometry: Tension fixture (rectangle)

Procedure of 1 step

1: Other Axial

Environmental Control

Temperature °C Inherit set point

Soak time s Wait for temperature

Test Parameters

Duration s

Motor direction Tension Compression

Hencky strain rate 1/s

Maximum gap change mm

Sampling Linear Log

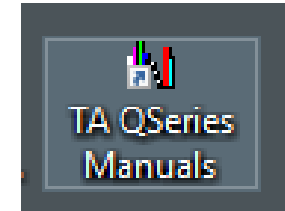
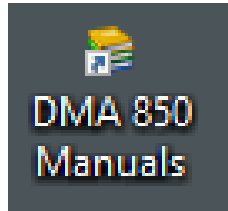
Sampling rate pts/s

Data acquisition Save image

Additional resources for experimental setup



Getting Started Manuals on your desktop



TA Instruments

Discovery DMA 850 Manuals

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*TA Manual Supplement
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Trios and Advantage Help

Browser address bar: C:\Program Files (x86)\TA Instruments\TRIOS\Help\DMA\TRIOS_Help_DMA.htm

Search bar: dma

Rank	Title
1	Operating the Humidity Controller (RH) with the DMA
2	Operating the Humidity Controller (RH) with the DMA
3	Introducing the DMA Q800
4	Basic Steps Needed to Run a DMA Experiment
5	Understanding the TRIOS Instrument Control Panel
6	Calibrating the DMA
7	Using the DMA for Transition Temperature
8	Available DMA Clamps
9	Operating the GCA with the DMA
10	Calibrating DMA Clamps
11	Understanding the DMA Testing Modes and Their App
12	Selecting a DMA Operating Mode
13	DMA 850 Messages
14	Aligning the DMA Thermocouples
15	Calibrating the DMA Instrument
16	Understanding the Different DMA Force Parameters
17	DMA Clamp Compliance Values
18	Available DMA Test Templates
19	Introducing the DMA Options and Accessories
20	Available DMA Clamps
21	Basic Steps Needed to Run a DMA Experiment
22	Loading a DMA Sample
23	Introducing the Touch Screen
24	Preparing the Instrument
25	Calibrating the Touch Screen
26	Configuring Instrument Options
27	Compression Clamp
28	Installing the ACA and Air Filter Regulator
29	3-Point Bending Clamps
30	Submersion Film/Fiber Clamp
31	Powder Clamp
32	Installing the GCA
33	3-Point Bending Submersion Clamps
34	Fiber Tension Clamp
35	Submersion Compression Clamp
36	Single/Dual Cantilever Clamp
37	Installing/Removing the Submersion Film/Fiber Clamp
38	Installing/Removing the Compression Clamp
39	Shear Sandwich Clamp
40	Setting Up an Oscillation MSFrequency Sweep
41	Setting Up an Oscillation MSTemperature Ramp

Available DMA Tests

Express (Single Step) Tests

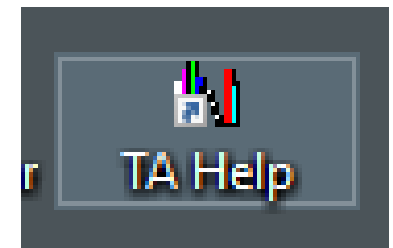
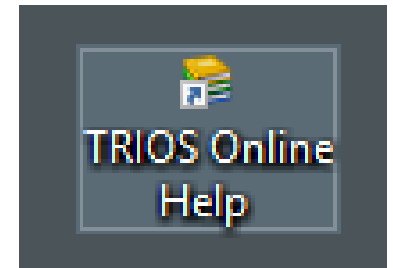
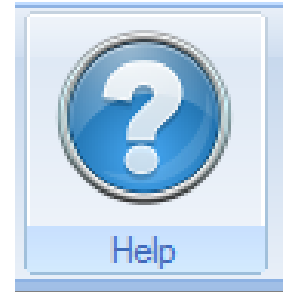
Select from the following test names for more information.

- Oscillation: Temperature Sweep
- Oscillation: Frequency Sweep
- Oscillation: Temperature Ramp
- Oscillation: Strain Sweep
- Oscillation: Stress Sweep
- Oscillation: Fatigue
- Oscillation: Temperature Sweep (Multifrequency)
- Oscillation: Temperature Ramp (Multifrequency)
- Oscillation: Time Sweep
- Oscillation: Temperature Ramp (MultiStep)
- Strain Control: Relaxation
- Strain Control: Relaxation TTS
- Strain Control: IsoStrain
- Stress Control: Creep
- Stress Control: Creep Recovery
- Stress Control: Creep TTS
- Stress Control: IsoStress
- Stress Control: Creep
- Rate Control: Strain Ramp
- Rate Control: Stress Ramp

Unlimited (Multi-Step) Tests

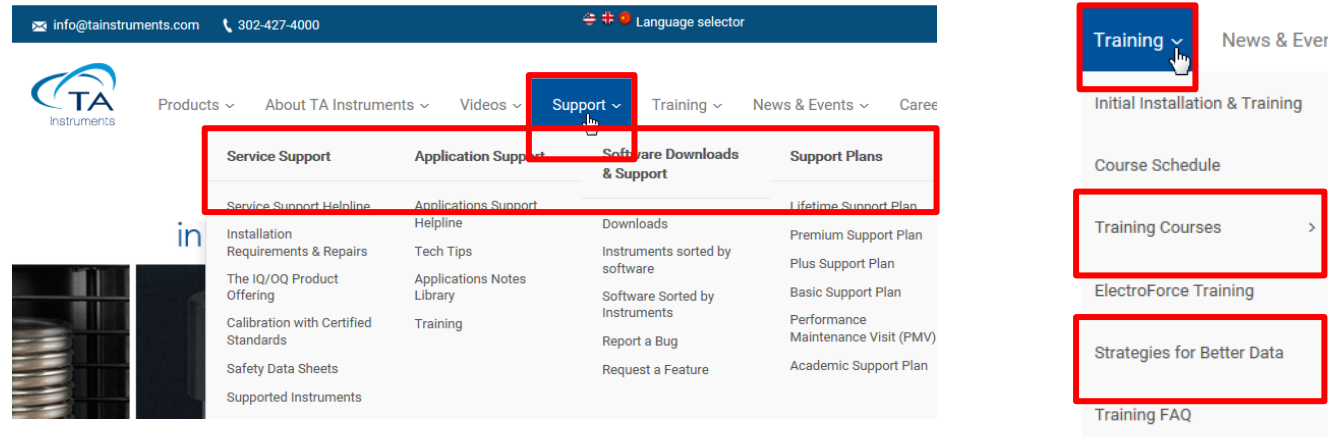
Select from the following test names for more information.

- Conditioning Temperature
- Conditioning Data
- Conditioning Other
- Conditioning Strain
- Conditioning Stress
- Conditioning Repeat
- Oscillation: Temperature Sweep
- Strain Control: Relaxation
- Strain Control: Relaxation TTS
- Strain Control: IsoStrain
- Stress Control: Creep
- Stress Control: Creep Recovery
- Stress Control: Creep TTS
- Stress Control: IsoStress

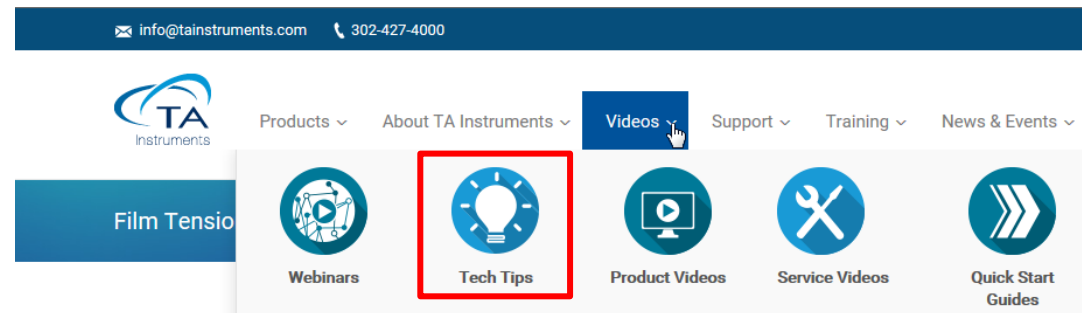


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