



AF2903 Road Construction and Maintenance

Rheology

Royal Institute of Technology, Stockholm

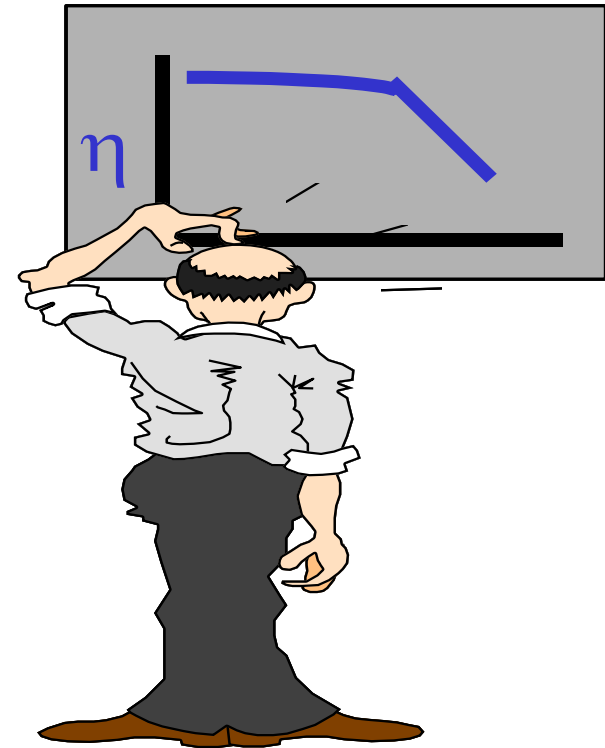
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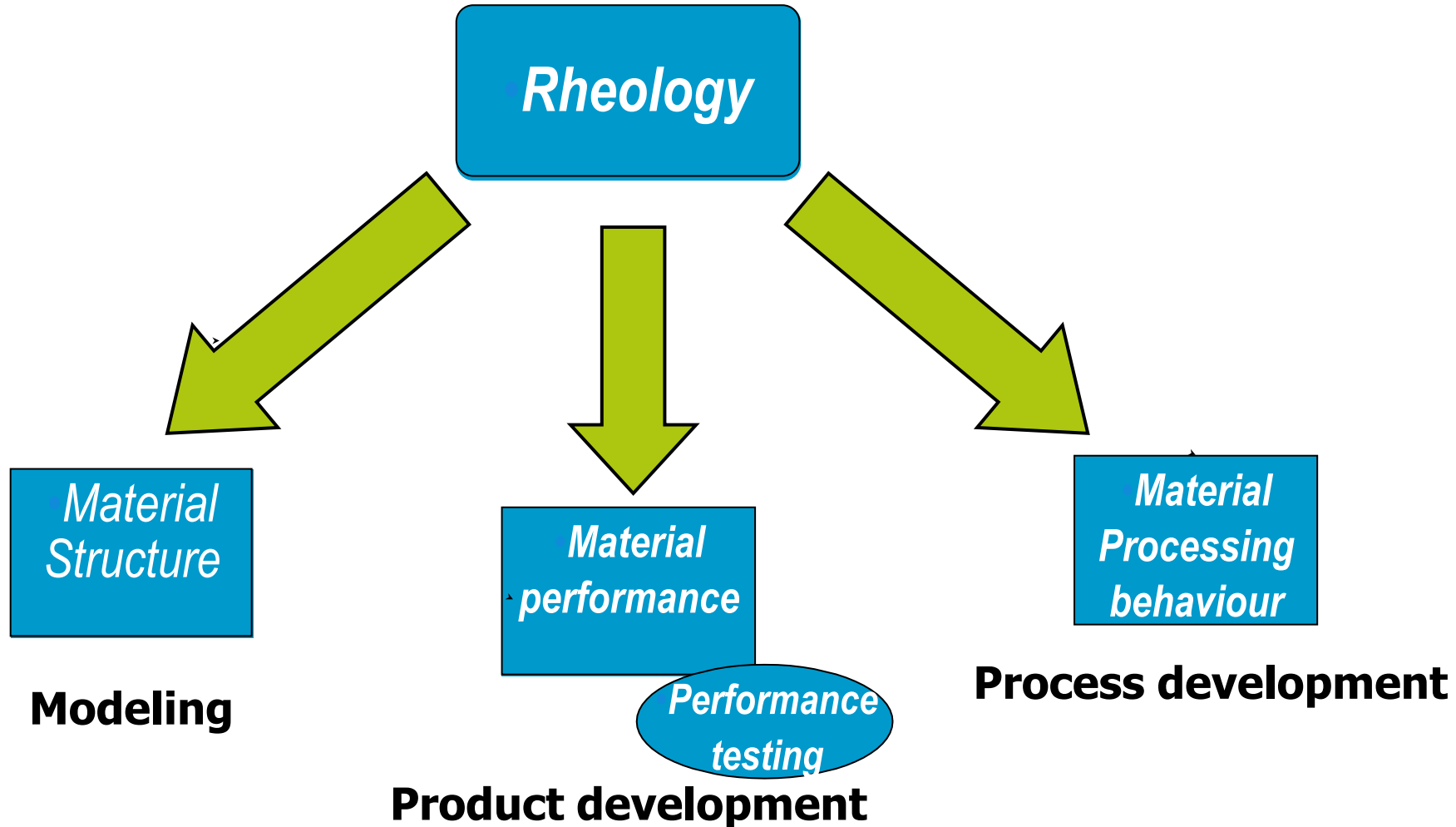


An Introduction to Rheology



Rheology

THE STUDY OF DEFORMATION AND FLOW OF MATTER



The Rheological Techniques

- Flow – pumping, dispensing, spraying etc
- Creep – what happens under gravity – stability
- Oscillation – structure and the effects of time and temperature

What is rheology looking at?

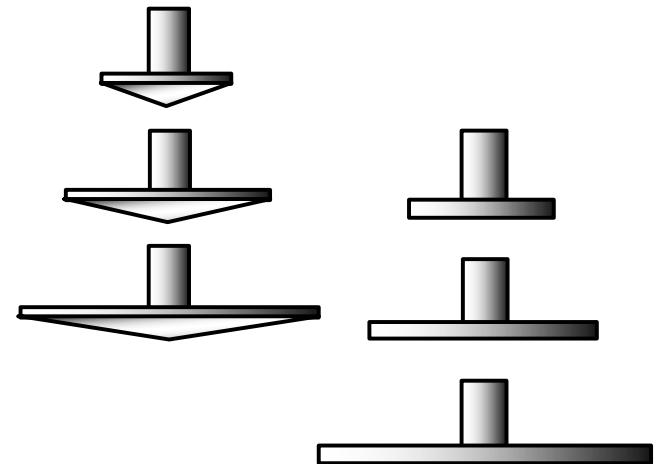
- A response to giving energy
 - Applying a shearing force – pushing
 - Changing temperature – excitation
- Is the energy used to do something or is it stored?

What is a Rheometer?

- Basically consists of five main components
 - Air bearing
 - near frictionless rotation
 - Motor
 - smooth application of torque
 - Optical encoder
 - measures rotation
 - Temperature control
 - accurate and source of energy
 - Geometry
 - what connects the rheometer to the sample



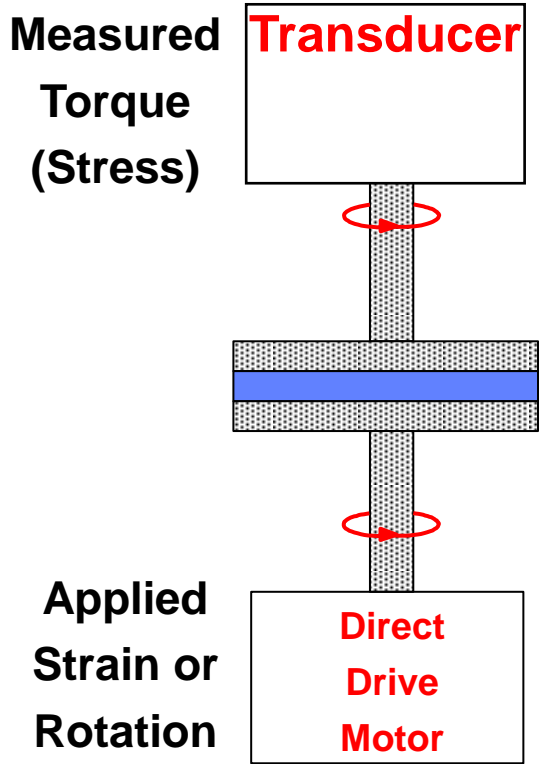
Dynamic Shear Rheometer (DSR)



Rotational Rheometers Designs

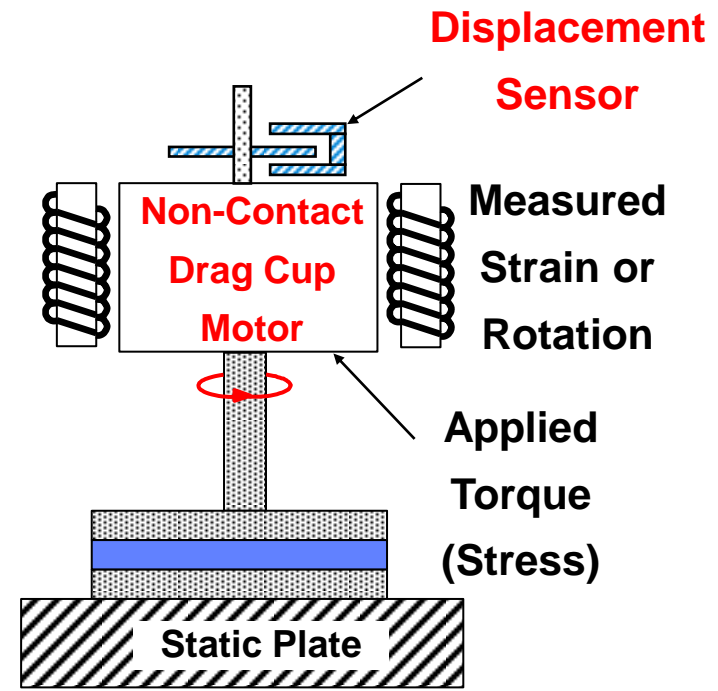
•Controlled Strain

•SMT – Separate motor & transducer



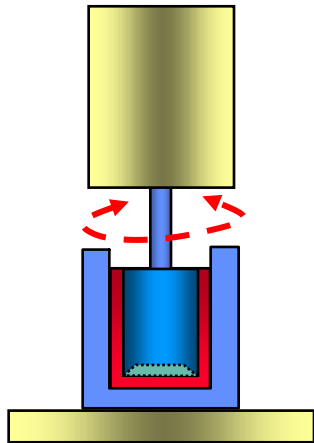
•Controlled Stress

•CMT – Combined motor & transducer



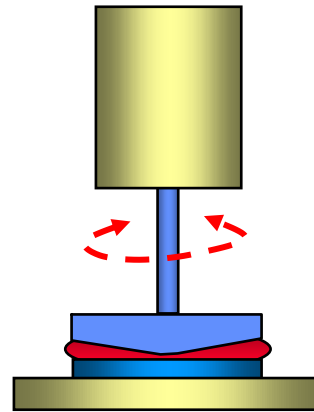
Geometries of DSR

Concentric
Cylinders



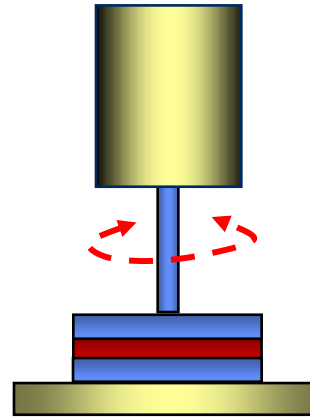
Very Low
to Medium
Viscosity

Cone and
Plate



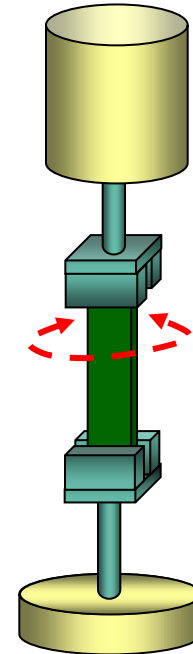
Very Low
to High
Viscosity

Parallel
Plate



Very Low
Viscosity
to Soft Solids

Torsion
Rectangular



Very Soft to Very
Rigid Solids

Water \longrightarrow to \longrightarrow Steel

How does a rheometer work?

- **Fundamentally a controlled stress rotational rheometer will**
 - **Apply a Torque (Force)**
 - **Measure an angular displacement**
 - **Calculate a rate of displacement (speed/velocity)**
- **If measured shear rate < requested shear rate, increase torque**
- **If measured shear rate > requested shear rate, decrease torque**
- **Feedback loop iterative process**
- **For controlled strain instruments, this process is the same just the variables are the other way round**
 - Apply velocity
 - Measure torque

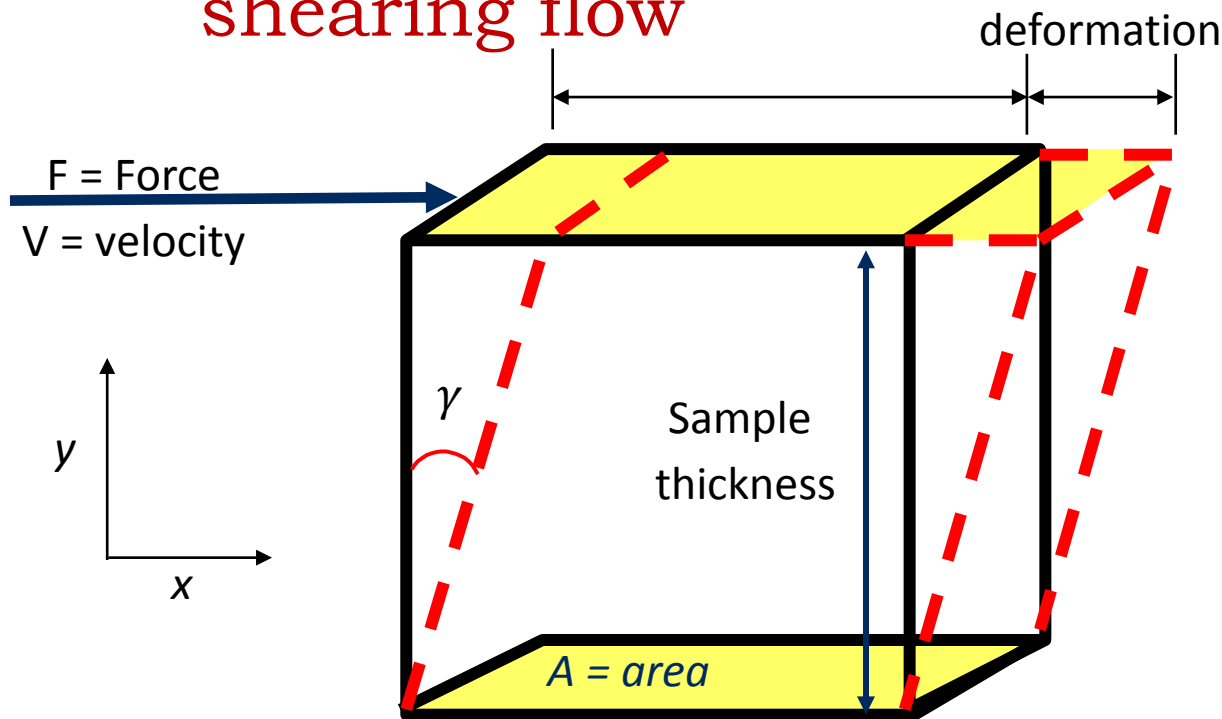
FLOW

- Process ability in handling and application

Viscosity

- Definition: resistance to flow
- The units of viscosity are
 - SI unit is the Pascal.Second (Pa.s)
 - cgs unit is the poise
 - 10 Poise = 1 Pa.s
 - 1 cP (centipoise) = 1 m Pa.s (milli-pascal-second)

Terminology - Simple shear and shearing flow



Shear Stress: $\sigma = F/A$

Strain: $\gamma = dx/y_0$

Shear Rate: $\dot{\gamma} = d\gamma/dt = V/y$

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

Modulus $G = \frac{\sigma}{\gamma}$

Cone Angles and Diameters

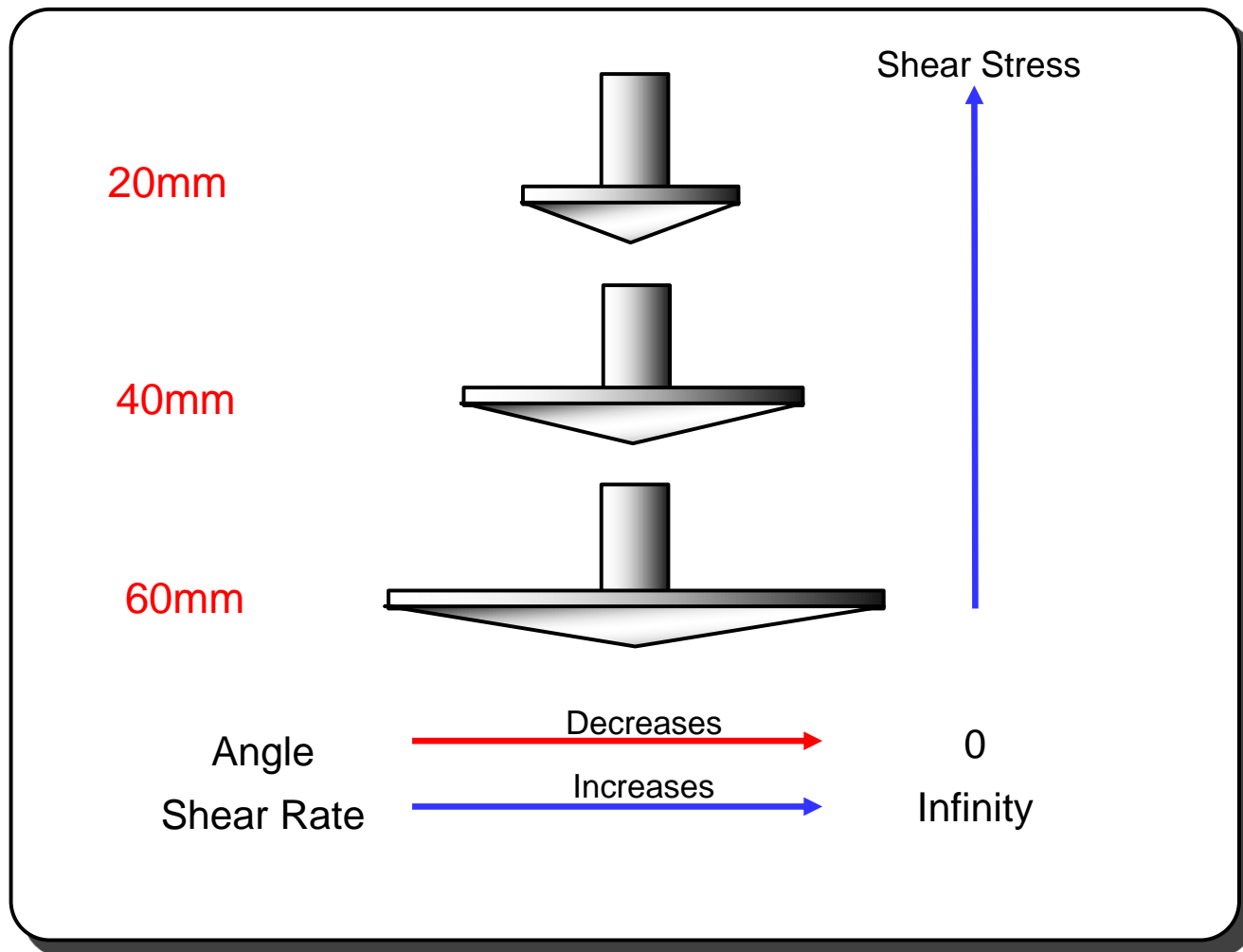
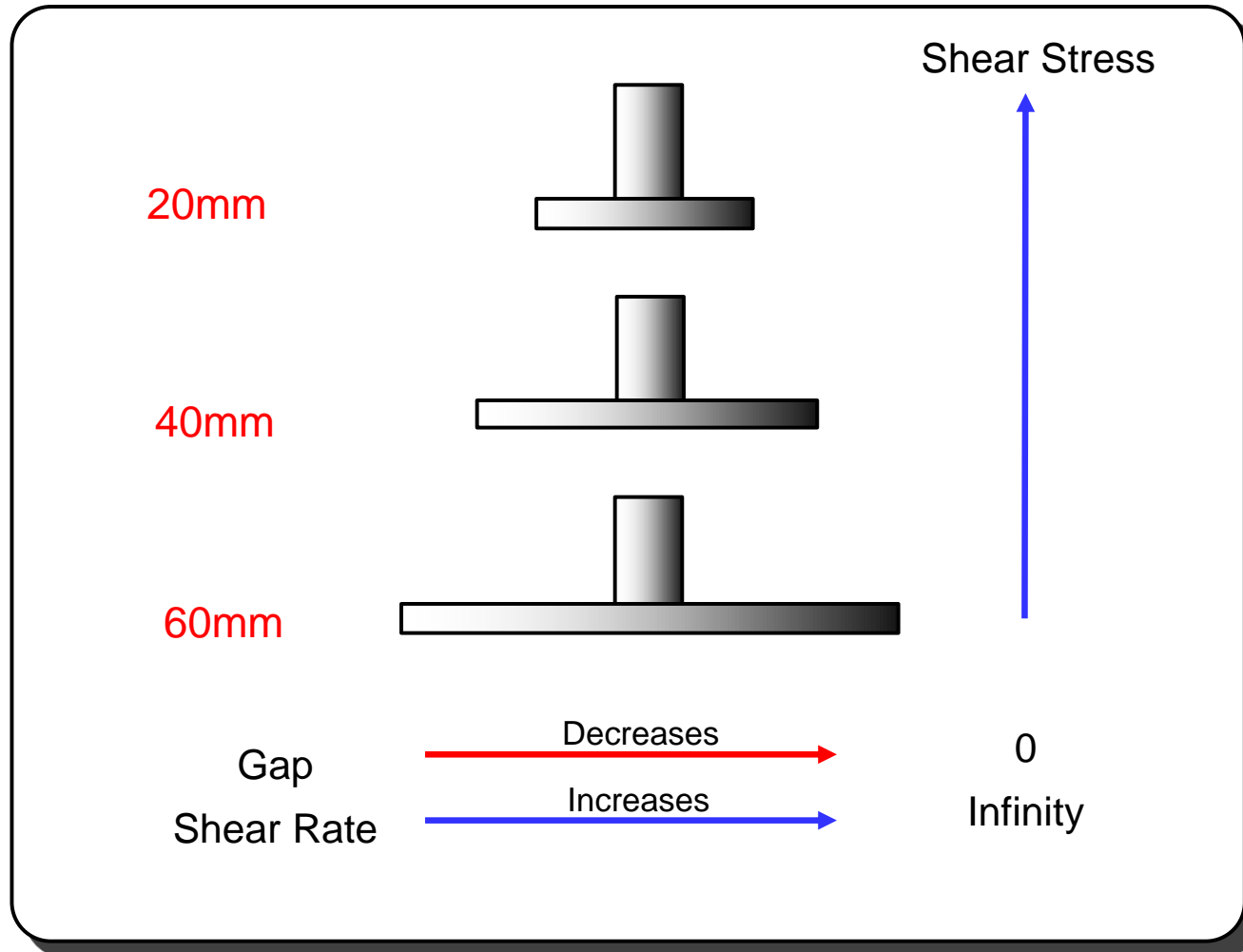


Plate Gaps and Diameters



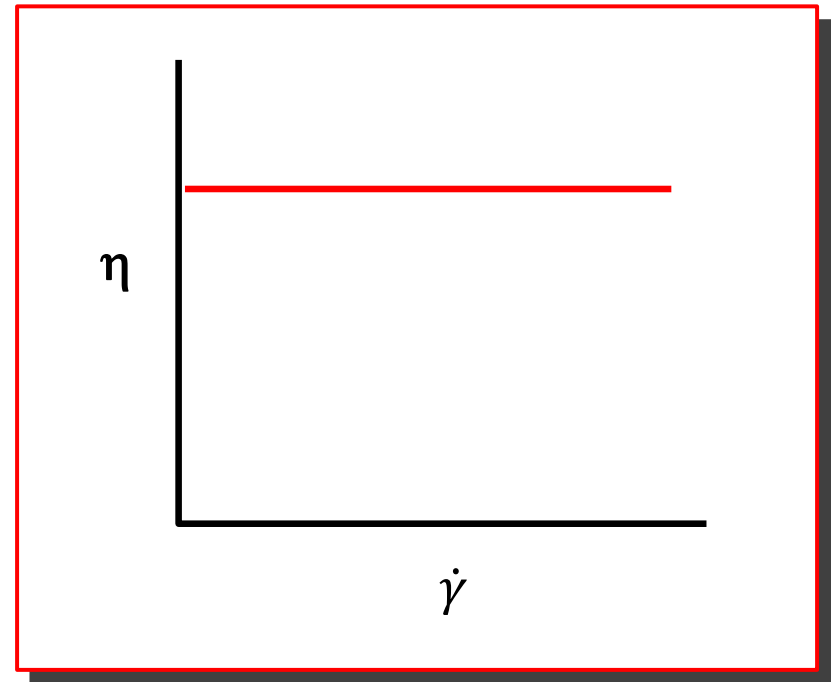
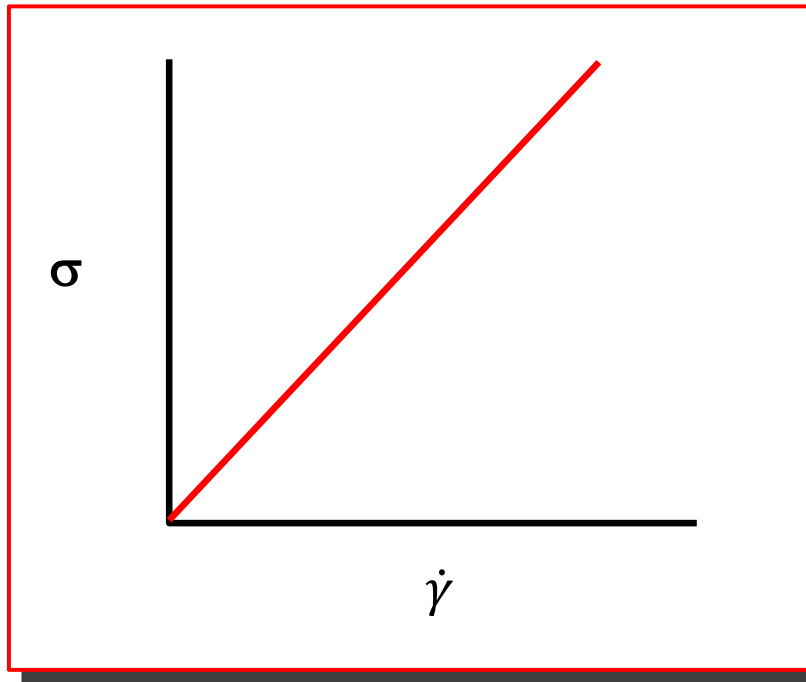
Typical Viscosities (Pa.s)

- Various materials and their viscosities

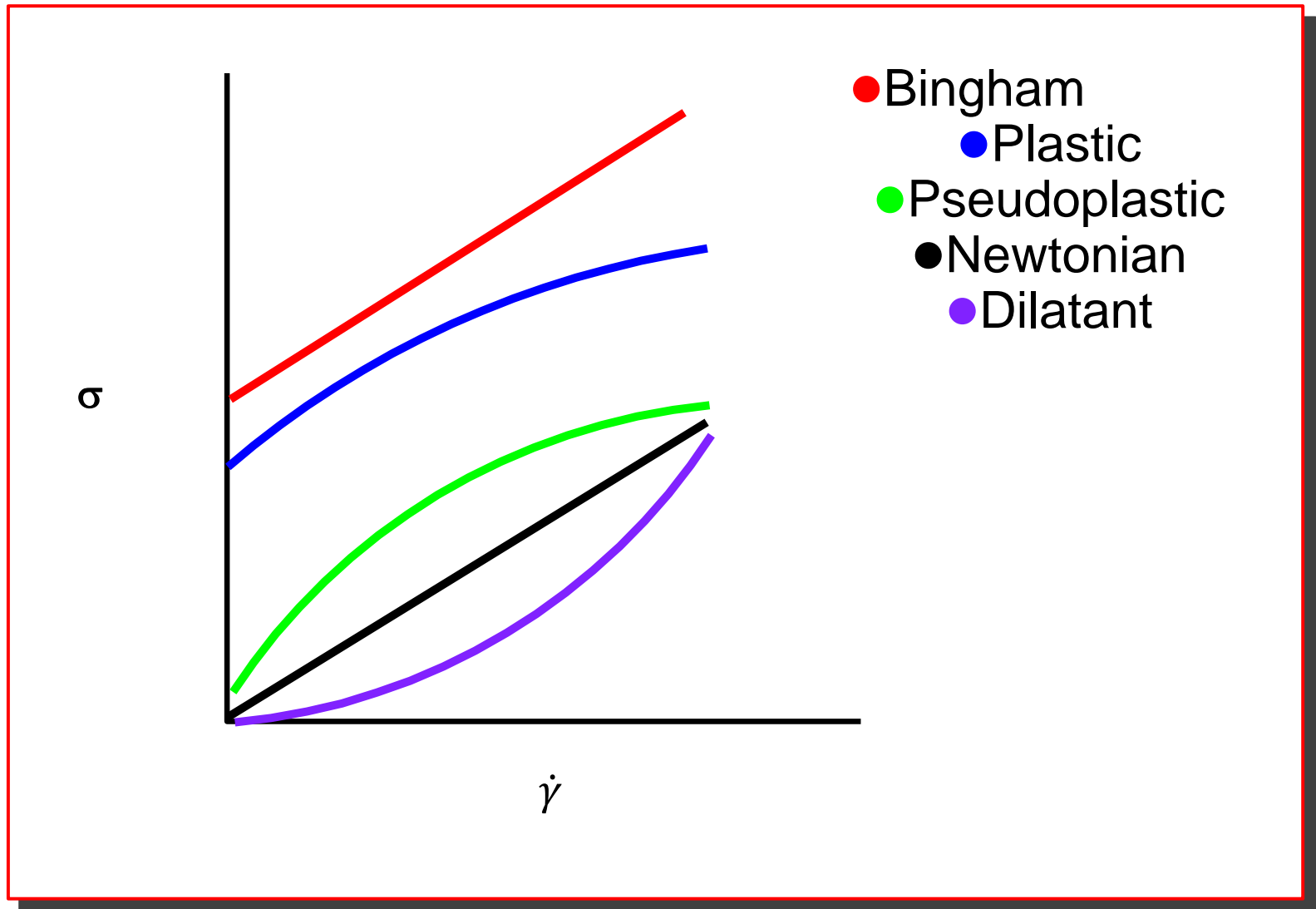
• BITUMEN	100 000 000
• POLYMER MELT	1 000
• GOLDEN SYRUP	100
• LIQUID HONEY	10
• GLYCEROL	1
• OLIVE OIL	0.01
• WATER	0.001
• AIR	0.00001

What Is A Flow Curve?

... a graph of shear stress versus shear rate

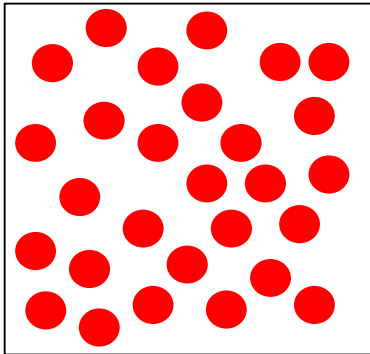


Newtonian - simplest case

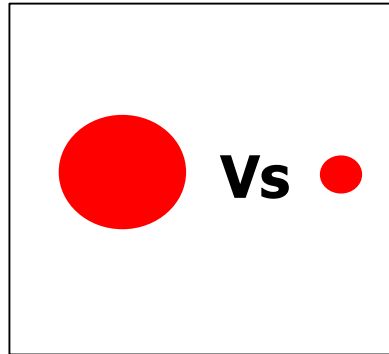


Factors Influencing Rheology

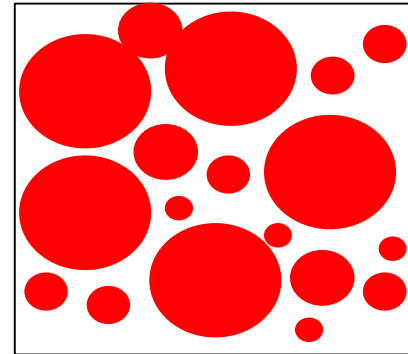
High volume fractions, ϕ



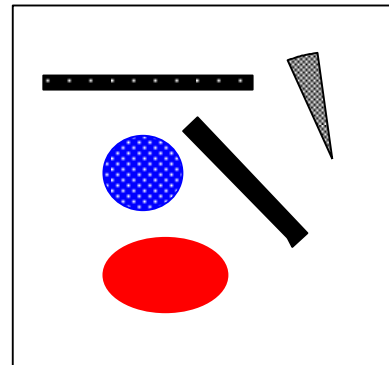
Particle size



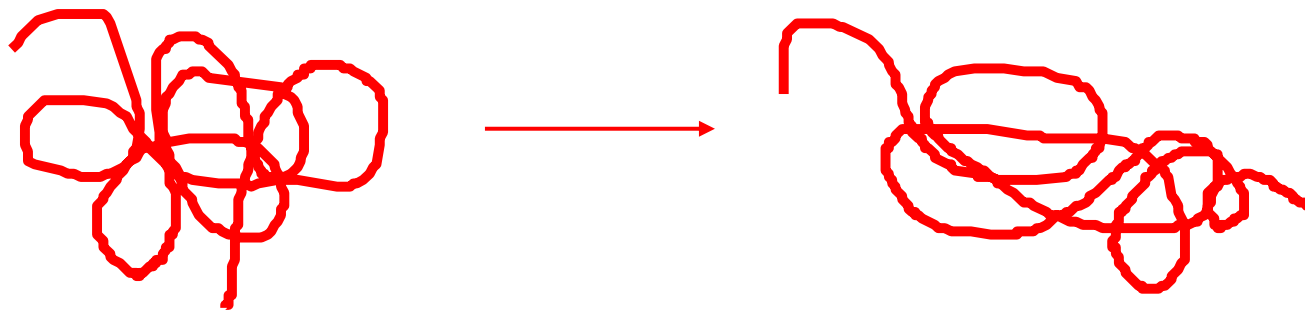
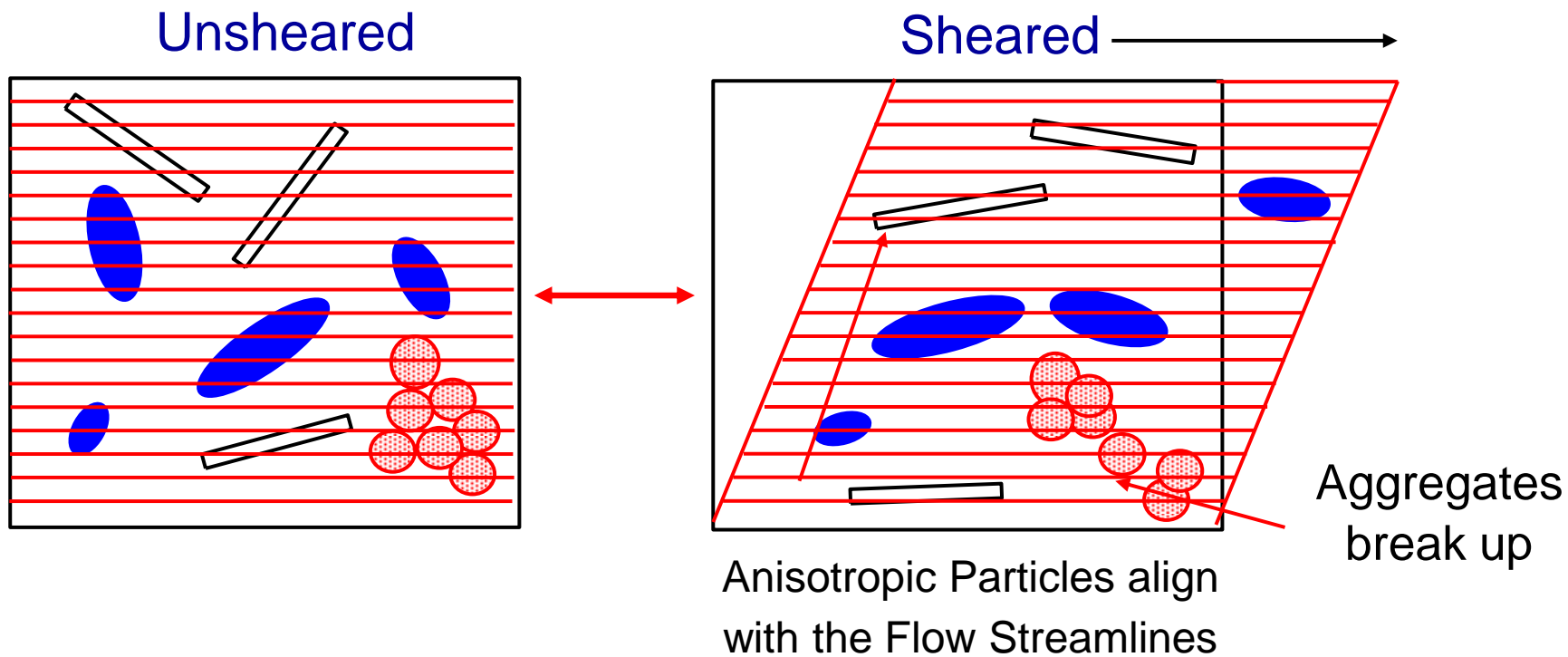
Particle size distribution



Particle shape

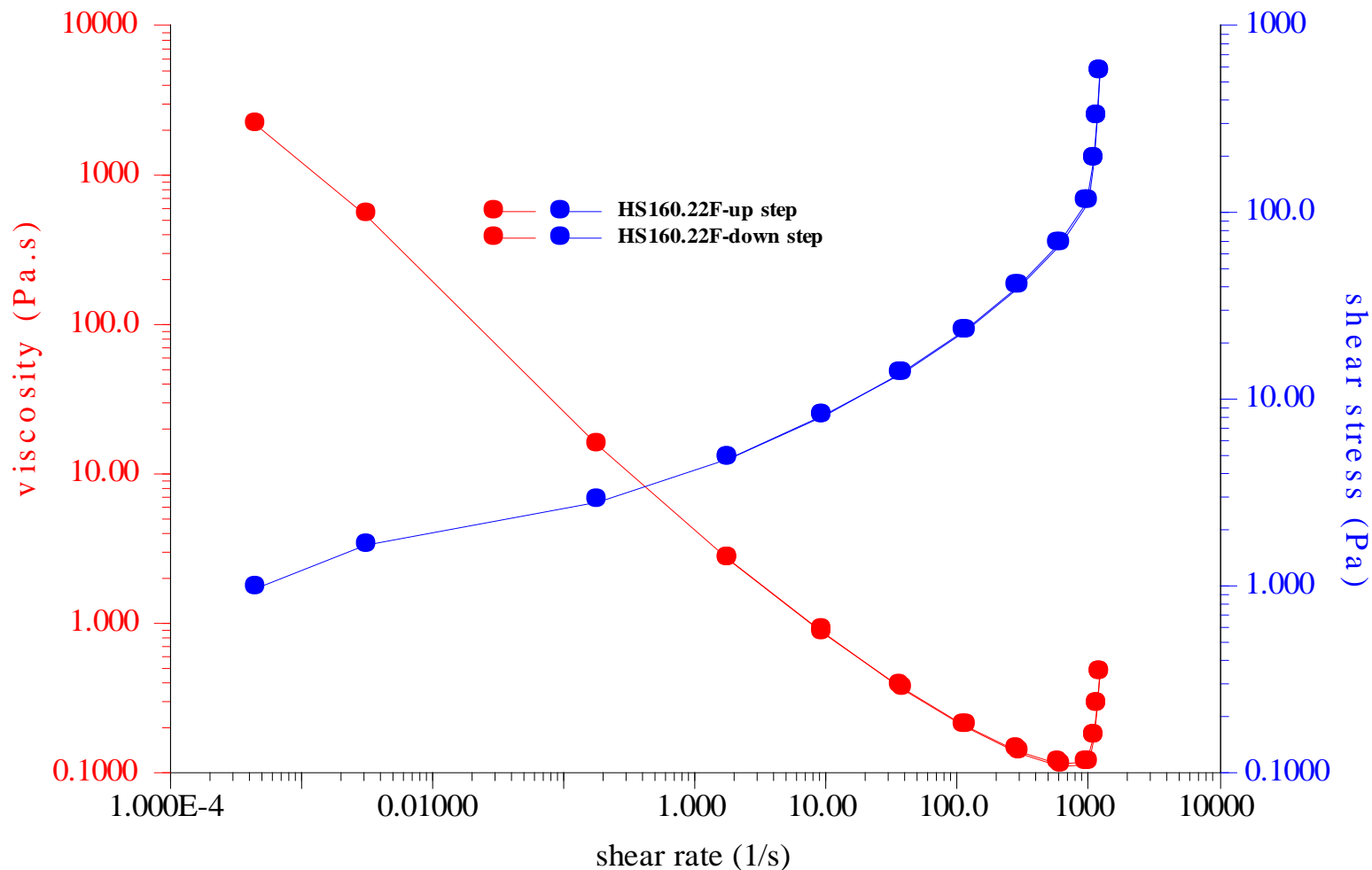


Shear thinning - why does it occur?



Shear-thickening of Silica Dispersion

Shear Thickening Particle Dispersion



Factors affecting viscosity

Shear Rate

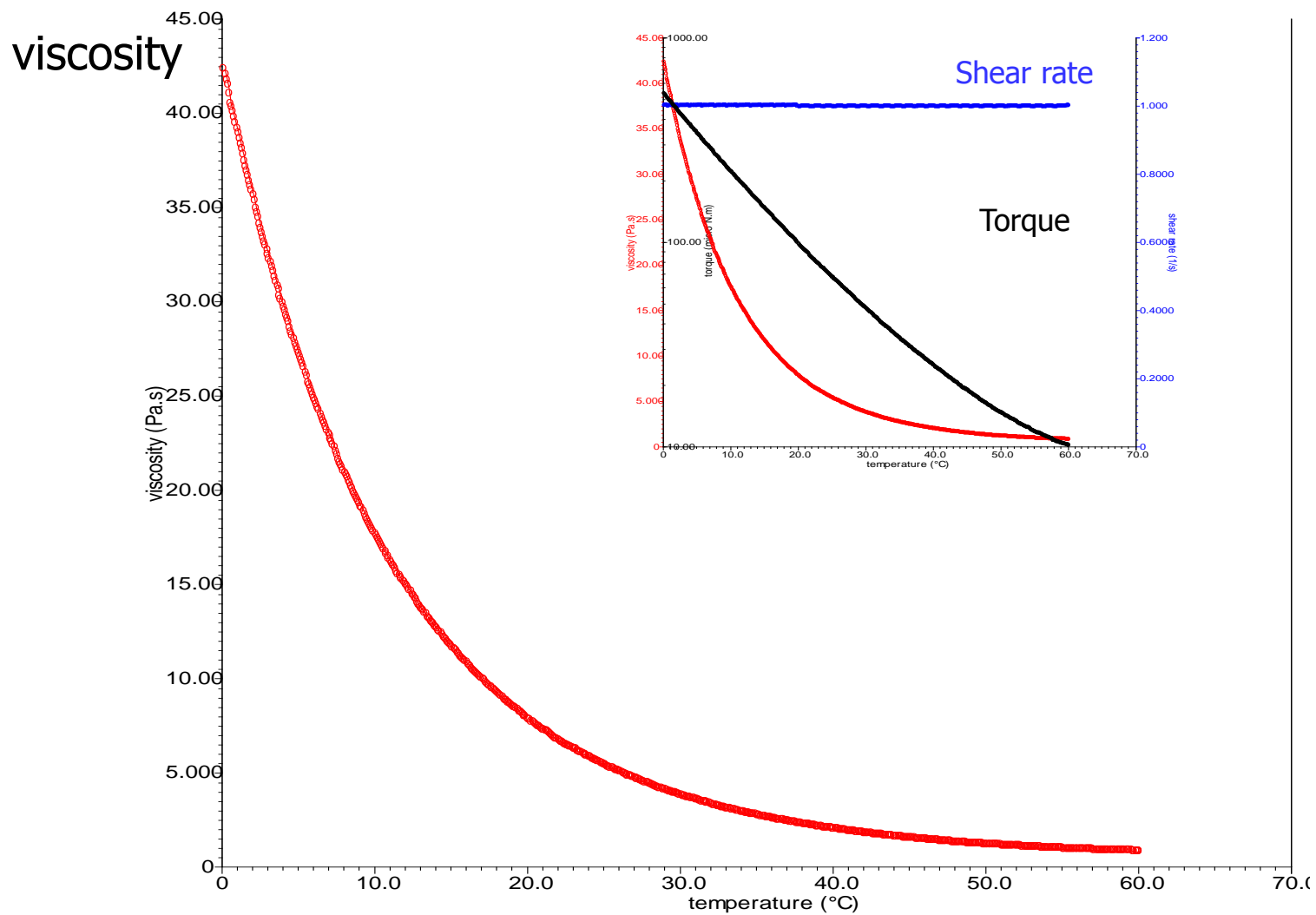
Time of Shearing

Temperature

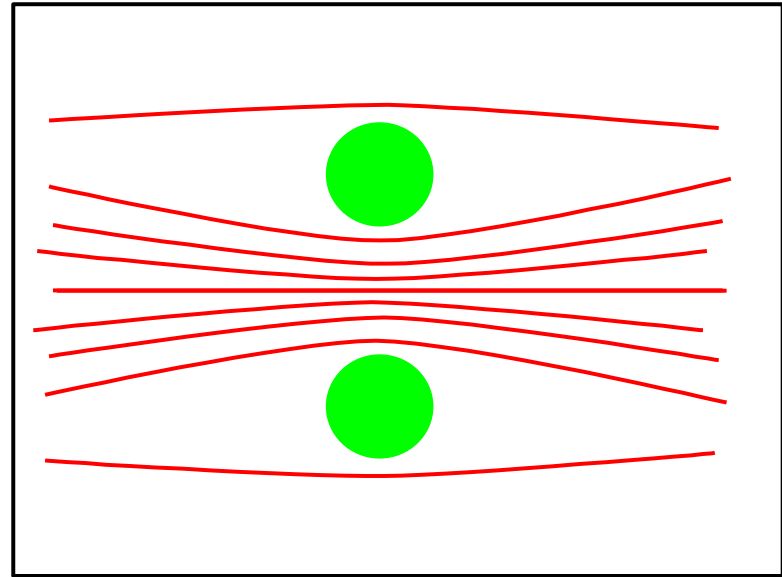
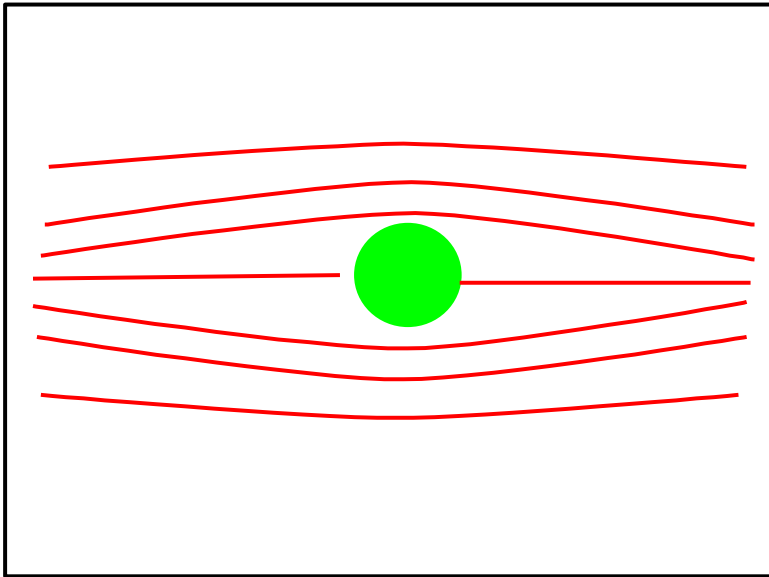
Concentration

Pressure

Effect of Temperature



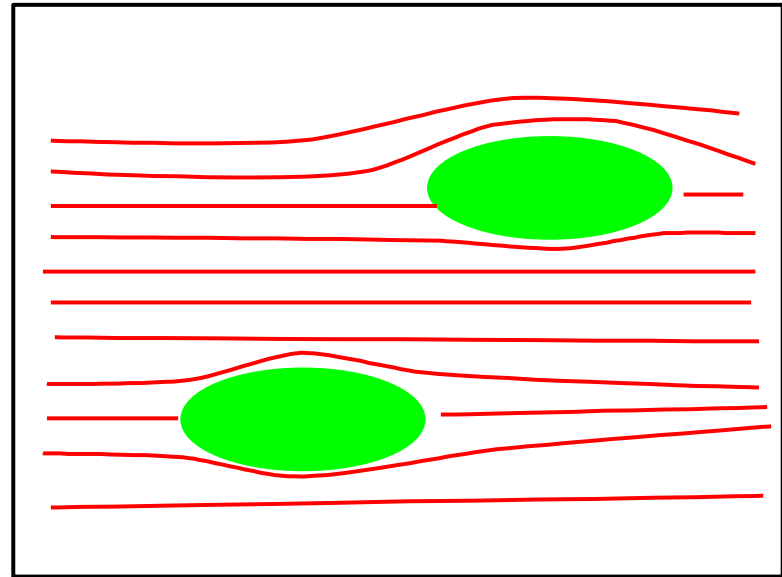
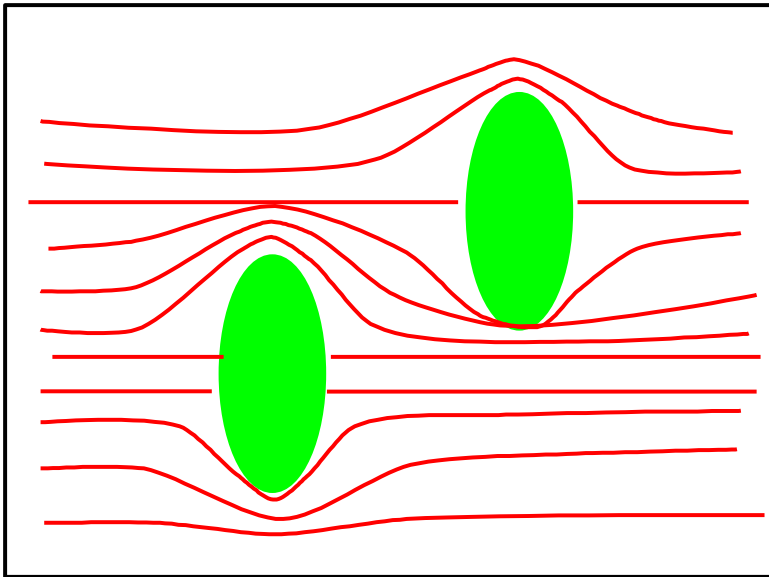
Effect of Concentration or Pressure



Flow restricted as more particles added, i.e. increase concentration

The limiting value of the reduced viscosity as the concentration approaches zero....

Effect of Shape and Alignment

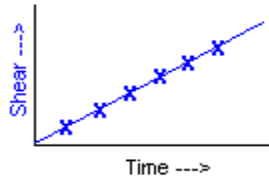


Aligned show less concentrated flow lines

Flow Measurements

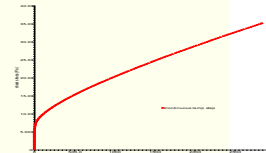
Test type

The following test types are available:

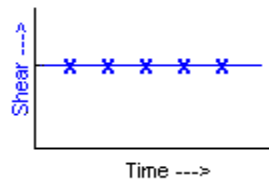


Continuous

Continually ramps the shear and samples at defined intervals.

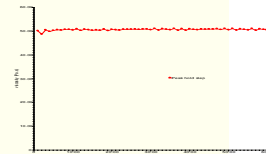


Typical plot shear stress vs. shear rate

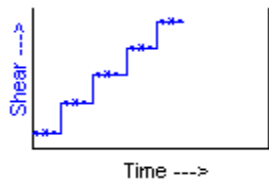


Peak hold

Holds the shear constant and samples at defined intervals.

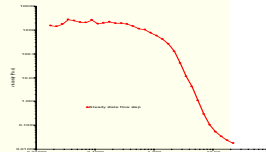


Typical plot viscosity vs. time

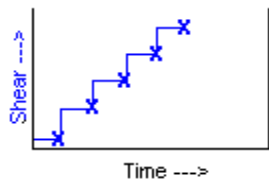


Steady state flow

Applies successive shear values. Data sampled under equilibrium conditions.

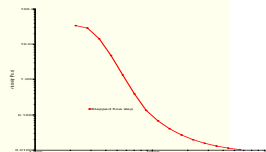


Typical plot viscosity vs. shear stress/rate

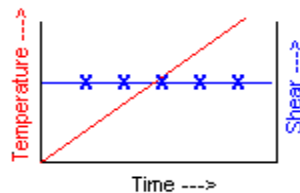


Stepped flow

Applies successive shear values. Data sampled over last x seconds.

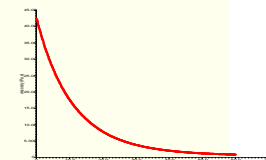


Typical plot viscosity vs. shear stress/rate



Temperature ramp

Holds the shear constant whilst ramping the temperature. Samples at defined intervals.



Typical plot shear stress vs. shear rate

Peak Hold – a “Brookfield” test

Name: Flow procedure

Steps:

- Conditioning Step
- Peak hold step
- Post-Experiment Step

Notes:

Test type: Peak hold

Test settings:

Hold: shear rate (1/s)

at: 10.00

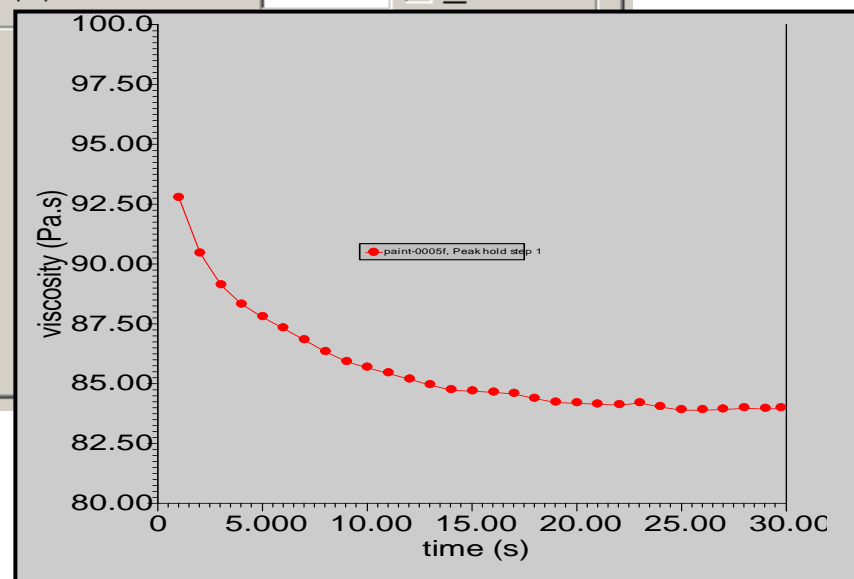
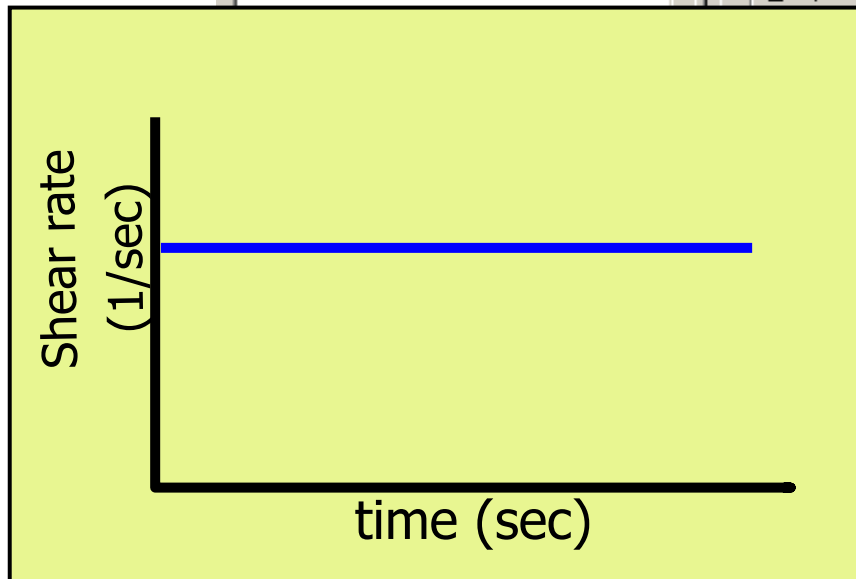
Duration (hh:mm:ss): 0:03:00

Sampling:

Delay time (hh:mm:ss): 0:00:01

Other settings:

Temperature (°C): 25.0 Wait



Continuous Flow Template

Name
Flow procedure

Steps

- Conditioning Step
- Continuous ramp step
- Post-Experiment Step

Notes

Test

Test type: Continuous ramp

Test settings

Ramp: torque (micro N.m)

From: 0 to 1000.00

Duration (hh:mm:ss): 0:03:00

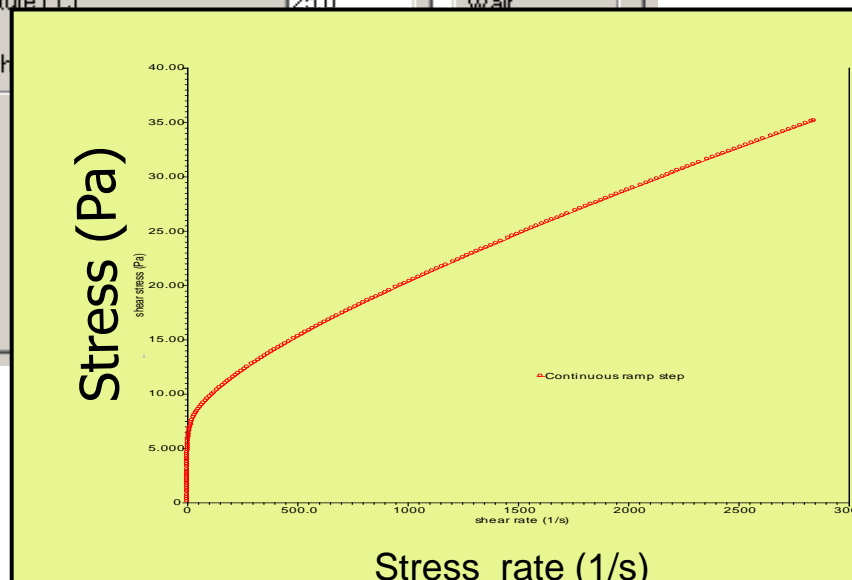
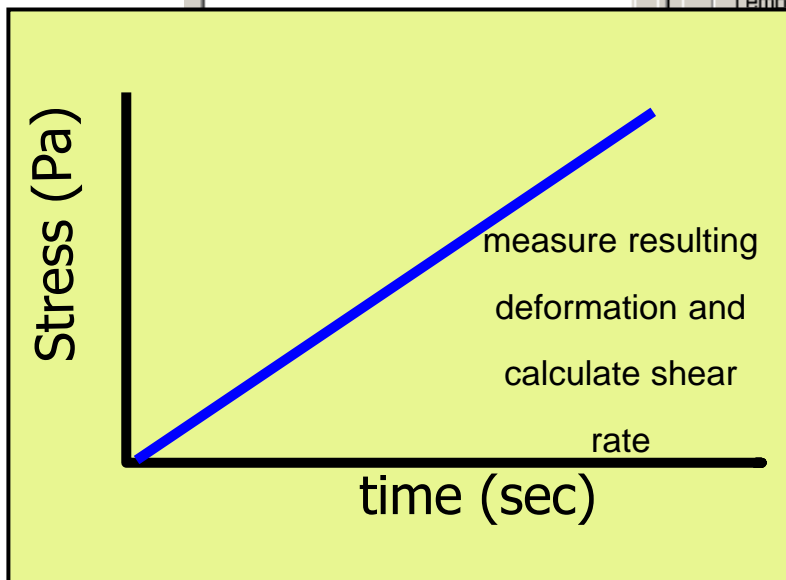
Mode: linear

Sampling

Delay time (hh:mm:ss): 0:00:01

Other settings

Temperature (°C): 25.0



Stepped Flow Template

Name: Flow procedure

Steps:

- Conditioning Step
- Stepped flow step
- Post-Experiment Step

Notes:

Test type: Stepped flow

Test settings:

Ramp: torque (micro N.m)

From: 0.10 to 1000.00

Mode: log

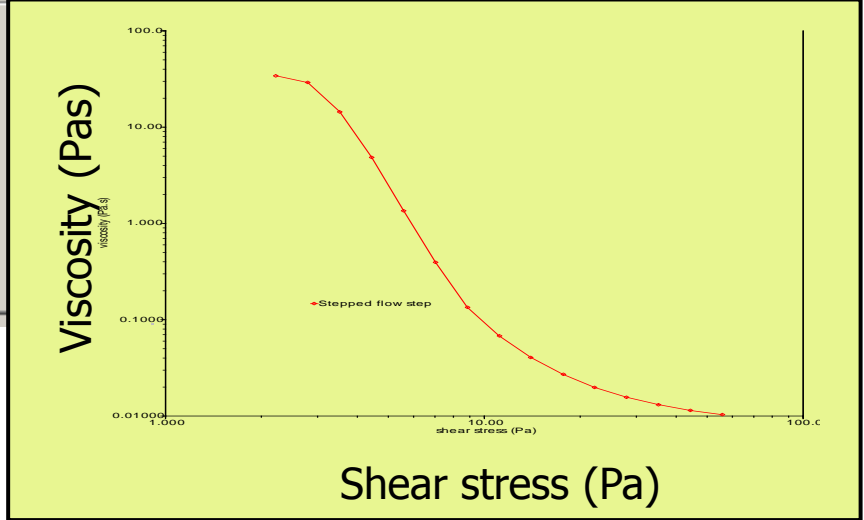
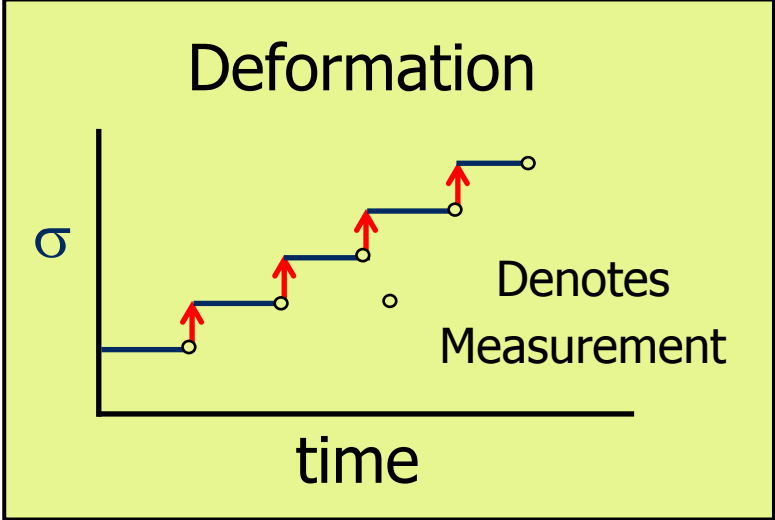
Points per decade: 10

Other settings:

Temperature (°C): 25.0 Wait

Constant time (hh:mm:ss): 0:00:10

Average last x seconds: 0:00:05



Steady State Flow Template

Name
Flow procedure

Steps

- Conditioning Step
- Steady state flow step**
- Post-Experiment Step

Notes

Test | Step termination | General

Test type: Steady state flow

Test settings

Ramp: torque (micro N.m)

From: 0.10 to 10000.00

Mode: Log

Points per decade: 10

Temperature (°C): 20.0 Wait

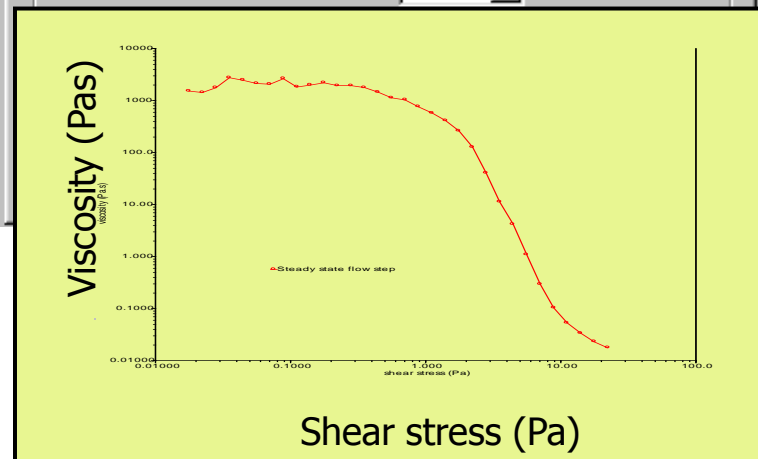
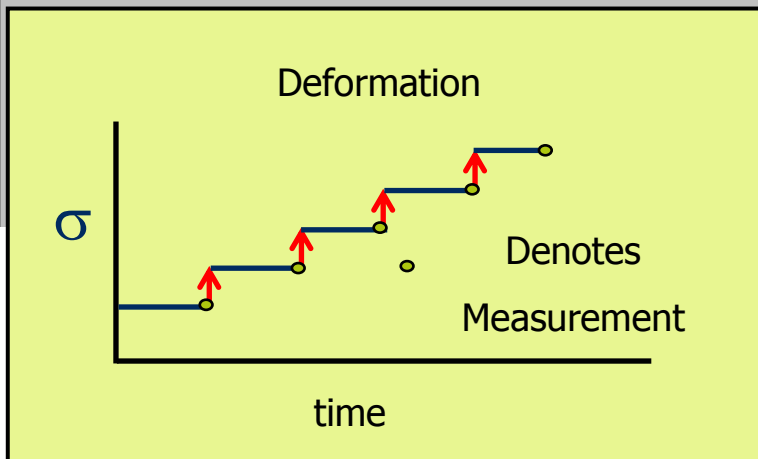
Sample period (hh:mm:ss): 0:00:10

Steady state

Percentage tolerance: 5.0

Consecutive within tolerance: 3

Maximum point time (hh:mm:ss): 0:01:00





Introduction to Viscoelasticity

Viscoelasticity Defined

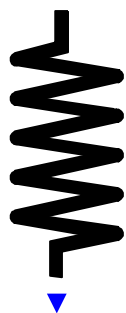
Range of Material Behaviour

Solid Like ----- Liquid Like
Ideal Solid ----- Most Materials ----- *Ideal Fluid*
Purely Elastic ----- *Viscoelastic* --- *Purely Viscous*

Viscoelasticity: Having both viscous and elastic properties

Response for Classical Extremes

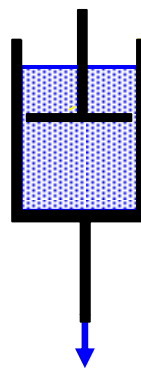
Spring



Purely Elastic
Response

Hookean Solid
 $\sigma = E\varepsilon$ or $\tau = G\gamma$

Dashpot



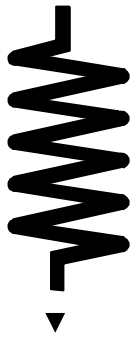
Purely Viscous
Response

Newtonian Liquid
 $= \eta\dot{\gamma}$

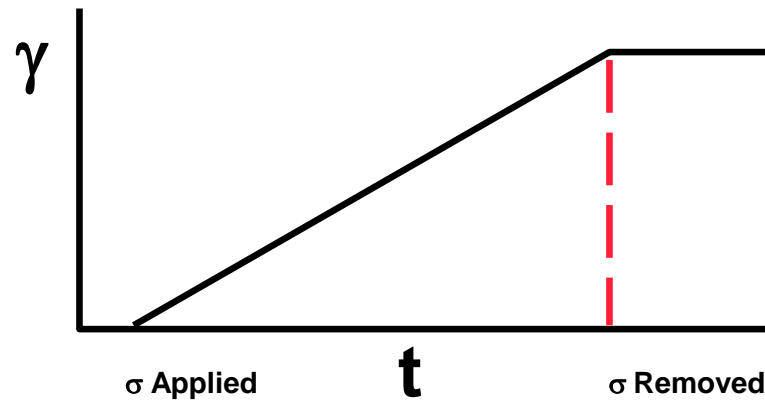
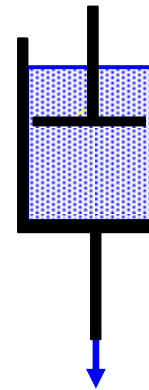
In the case of the classical extremes, all that matters is the values of stress, strain, strain rate. The response is independent of the loading.

Spring and Dashpot Models

Elastic Deformation



Viscous Flow



Viscoelasticity

Spring



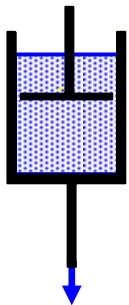
Purely Elastic
Response

Hookean Solid
 $\sigma = E\varepsilon$ or $\tau = G\gamma$

+



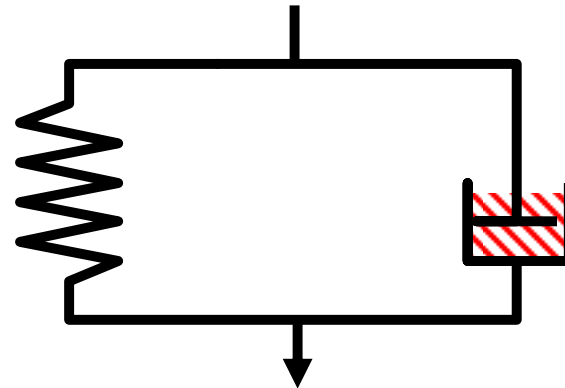
Dashpot



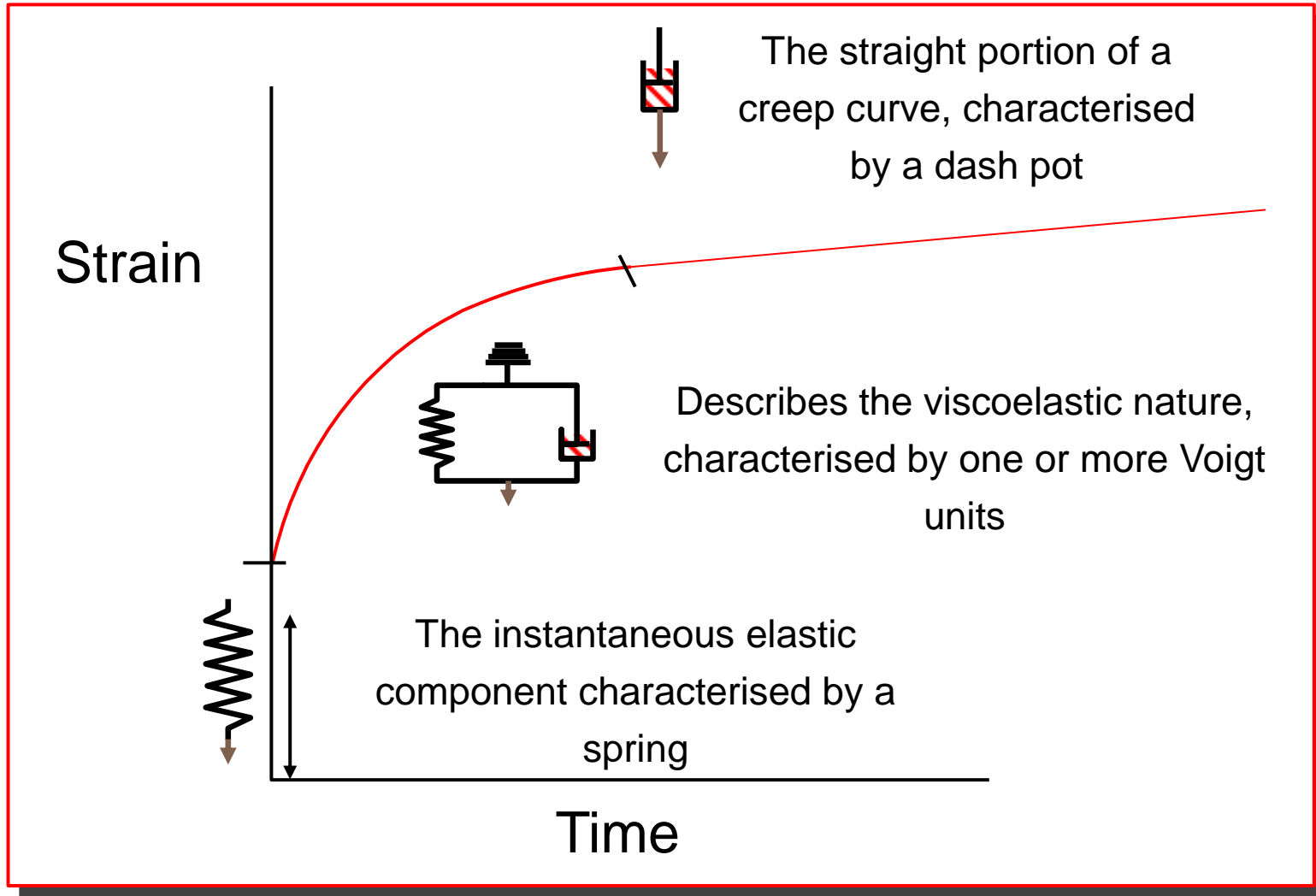
Purely Viscous
Response

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

ViscoElastic



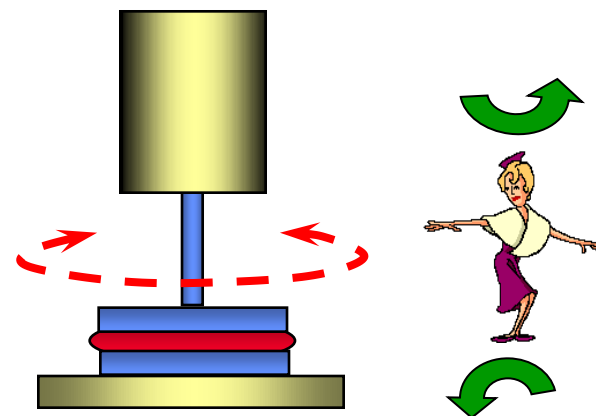
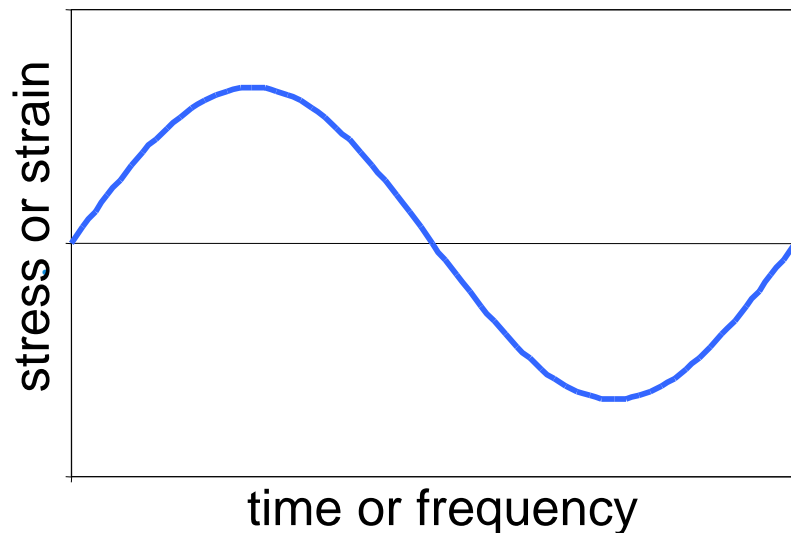
Parts of a creep curve





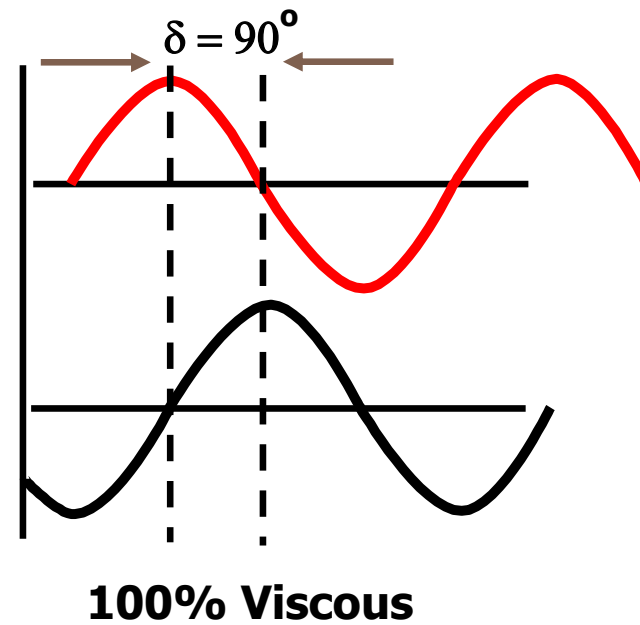
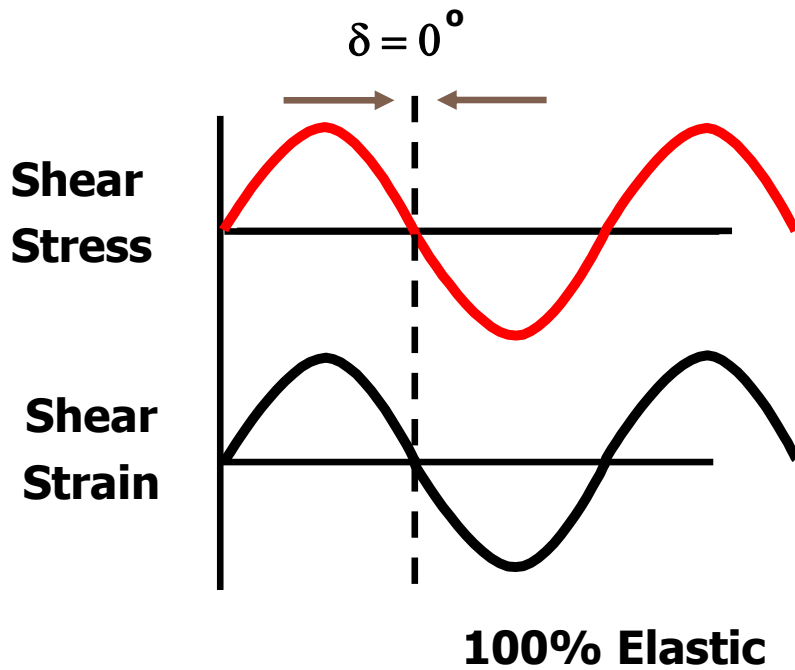
Introduction to Dynamic Measurements

What is Oscillation?



- Deformation applied Sinusoidally
- User defined Stress or Strain amplitude, frequency & temperature
- Control either
 - Amplitude – test how robust a material is
 - Frequency – responses to the rate at which energy is given
 - Temperature – cures and transitions
 - Nothing – watch structure form

Oscillation



Viscoelastic Parameters

G* - Complex Modulus: Measure of materials overall resistance to deformation.

$$G^* = \text{Stress}^*/\text{Strain}$$
$$G^* = G' + iG''$$

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

$$G' = (\text{stress}^*/\text{strain})\cos\delta$$

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

$$G'' = (\text{stress}^*/\text{strain})\sin\delta$$

Tan Delta:

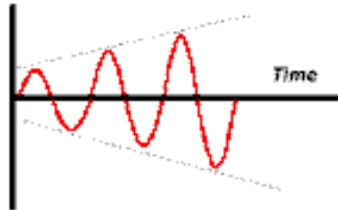
Measure of material damping - such as vibration or sound damping.

$$\text{Tan } \delta = G''/G'$$

Modes of Operation

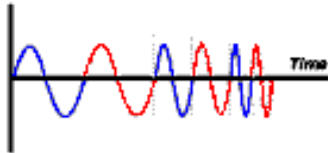
Oscillation procedure type

The following test types are available:



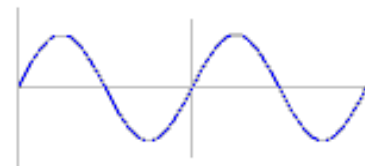
Strain / Stress Sweep

Applies a fixed frequency and increments the amplitude of the strain or stress.



Freq. Sweep

Applies a fixed amplitude and changes the frequency.



Temperature ramp

Applies a fixed frequency with a set amplitude. Monitors viscoelastic properties as a function of time / temperature.

Time / Temperature sweep

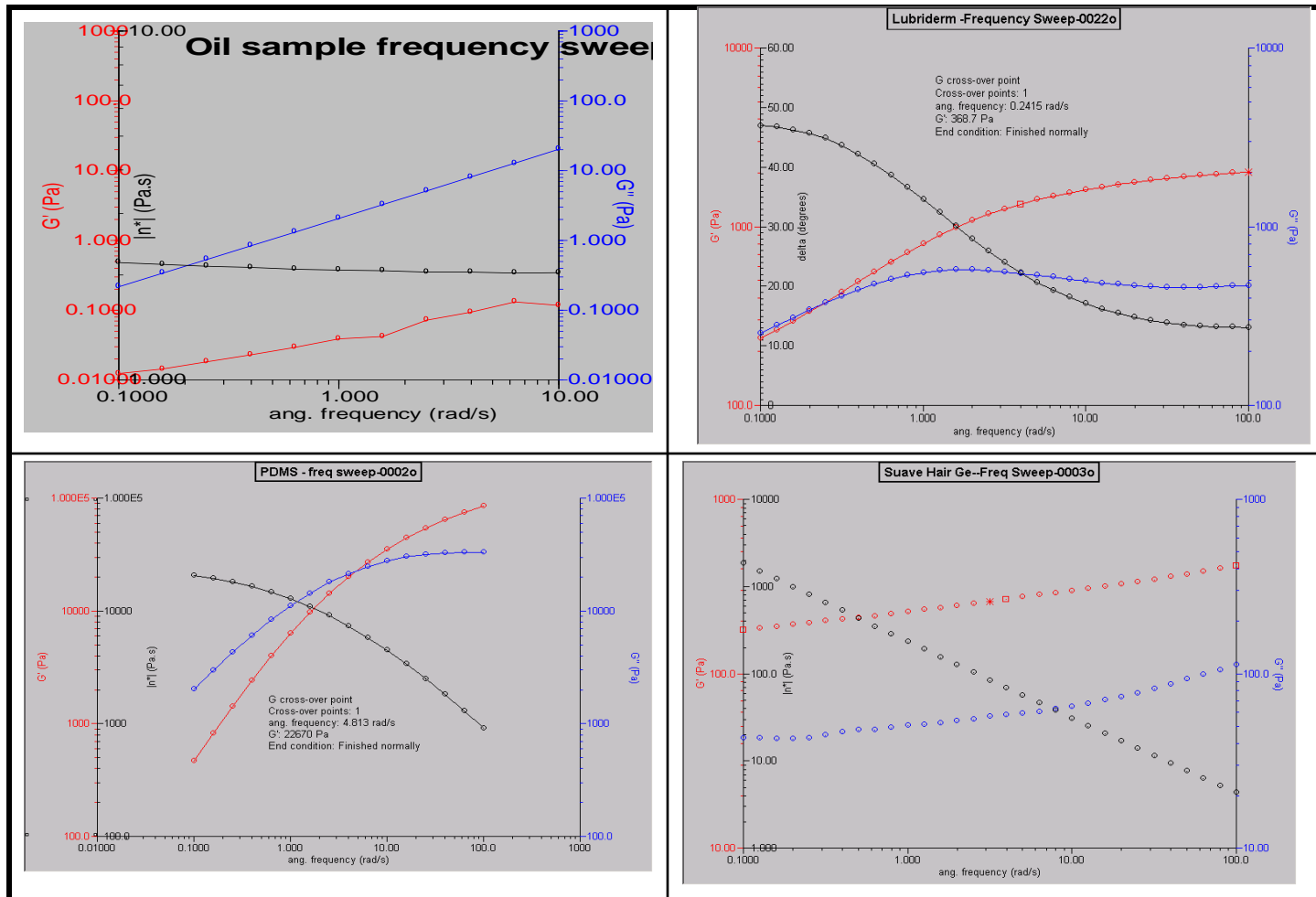
As per ramp , but applies a sequence of discrete temperature steps rather than a smooth ramp.

identification of the linear viscoelastic region (LVR)

“rheological fingerprint”

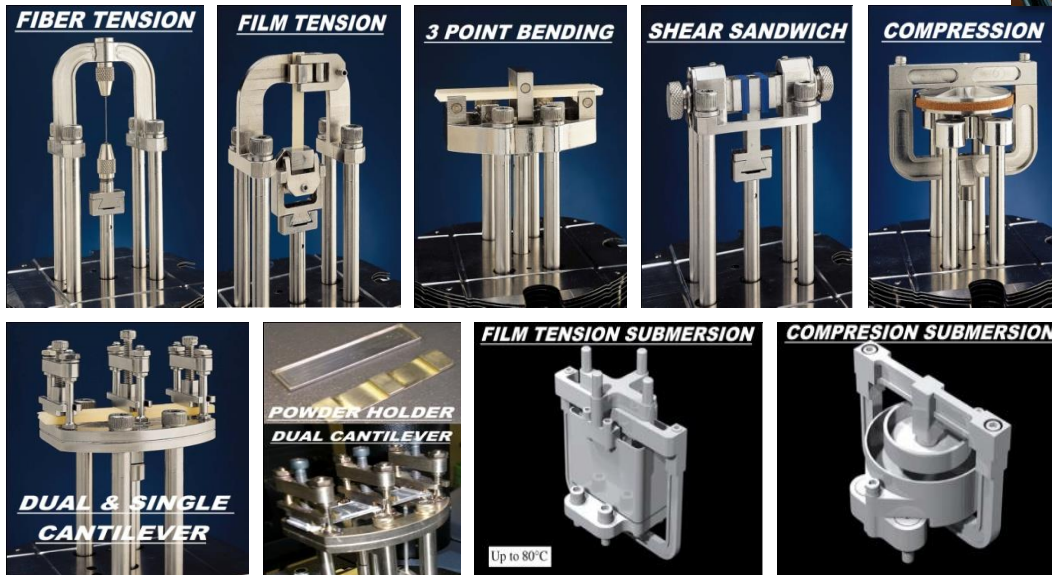
Process conditions, cure and physical transitions

Frequency Sweep Results



- It may look very different depend on samples

Dynamic Mechanical Analysis (DMA)



Modes of Operation:

1- MULTI-FREQUENCY

The multi-frequency mode can assess viscoelastic properties as a function of frequency, while oscillation amplitude is held constant. These tests can be run at single or multiple frequencies, in time sweep, temperature ramp, or temperature step/hold experiments.

2 - MULTI-STRESS/STRAIN

In this mode, frequency and temperature are held constant, and the viscoelastic properties are monitored as strain or stress is varied. This mode is primarily used to identify the Linear Viscoelastic Range (LVR).

3 - CREEP/STRESS RELAXATION

With creep, the stress is held constant and deformation is monitored as a function of time. In stress relaxation, the strain is held constant and the stress is monitored vs. time.

4 - CONTROLLED FORCE/STRAIN RATE

In this mode, the temperature is held constant while stress or strain is ramped at a constant rate. It is used to generate stress / strain plots to obtain Young's Modulus. Alternatively, stress can be held constant with a temperature ramp while strain is monitored.

5 - ISO STRAIN

In isostrain mode, available on the Q800, strain is held constant during a temperature ramp. Isostrain can be used to assess shrinkage force in films and fibers

