

## Rheology -

1. Dynamic Rheology – Oscillation
2. Thixotropy

### 1 Dynamic Rheology – Oscillation

The viscoelastic properties of a material are measured in an oscillation test. Oscillation is the technique whereby we apply a sinusoidal stress or strain; the induced response must also follow a sine wave. In this manner, we continuously excite the sample, but never exceed a strain large enough to destroy structure. If we overstrain the sample, the elastic structure will be destroyed so care must be taken to keep the strain low; to work within the *linear viscoelastic region (LVR)*.

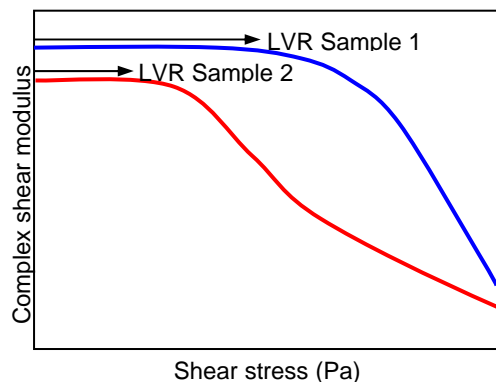


Figure 1: Amplitude sweep

To determine the LVR, we perform an amplitude sweep over a stress or strain range. The resulting graph can often give an indication of stability (figure 1). While sample structure is maintained, the complex modulus is constant; when the applied stress becomes too high, breakdown occurs and the modulus decreases. Sample 2 has a much shorter LVR than sample 1; therefore it breaks down much more easily. The length of the LVR is a measure of stability. ALL further oscillation analysis must be performed in the LVR!

Once the LVR is determined, a frequency sweep at a stress in this area can be used to determine the nature of the material. In the case of figure 2, there is a clear difference between samples 1 and 2. The former shows some frequency independence and also the

storage (or elastic) modulus,  $G'$  is dominant over the entire frequency range. The system is gelled, showing little change in viscoelastic characteristics. Sample 2, on the other hand, is frequency dependent and in this case, is dominated by the loss (or viscous) modulus,  $G''$ . The system has little internal network and is easily disturbed.

#### Measurement conditions:

**Samples:** Gels, toothpaste etc.

**Geometry:** 4°/40 mm cone and plate

**Amplitude sweep:** 0.1 – 100 Pa, UP, Logarithmic

**Frequency sweep:** 10 to 0.1 Hz, DOWN

**Strain:** As found from LVR

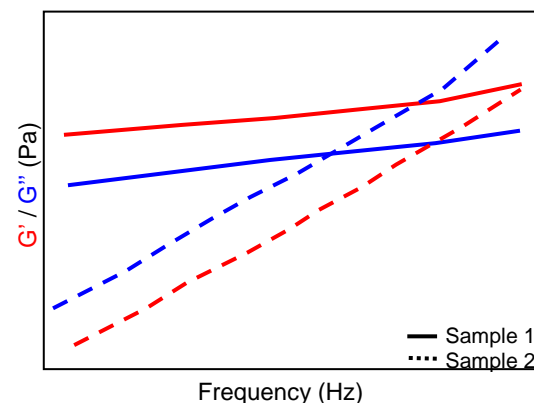


Figure 2: Frequency sweep

#### Conclusion

Oscillation may be used to determine the strength and stability of a material. It gives a clear indication of the behaviour of the sample, whether viscous or elastically dominated, over a given frequency range.

## 2 Thixotropy

Thixotropy is a measure of the time dependency of the sample. Under shear, there is a reduction in apparent viscosity; if the sample takes a while to return to its initial value, then it is time dependant. The material is gel-like at rest, but exhibits fluid like behaviour when agitated.

### Experimental

A hysteresis loop is produced whereby the sample is subjected to ascending and descending shear stresses. The loop indicated by the blue line in figure 3 illustrates a high degree of thixotropy, the sample has changed significantly before and after shearing. For the red graph, on the other hand, the up and down stresses overlay each other, indicating that the sample is shear thinning. This gives a measure of thixotropy at its most basic, the magnitude of the loop indicating the degree of time dependency.

### Measurement conditions

**Samples:** Paints, sauces, toothpaste *etc.*

**Geometry:** 4°/40 mm cone and plate

**Rheology option:** Viscometry

**Shear rate:** Sweep up & down 1 – 900 s<sup>-1</sup>

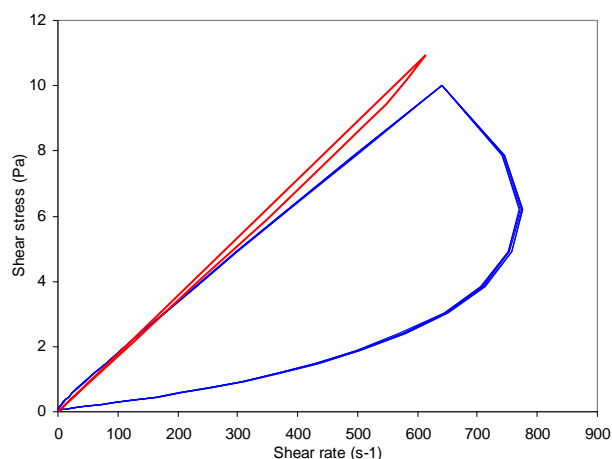


Figure 3: Flow hysteresis

### Conclusion

Sample history must be taken into account when analysing a thixotropic material. Either sufficient time must be taken between measurements to allow the sample structure to rebuild to its initial state or an identical level of pre-shear must be applied prior to each test to ensure the same shear history.

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