Determination of Yield Stress of Complex Fluids by Multiple Creep Tests on a Rotational Rheometer – Moisturizing Lotion

Introduction

Many complex fluids, such as network forming polymers, surfactant mesophases and concentrated emulsions do not flow until the applied stress exceeds a certain critical value, known as the yield stress. Materials exhibiting this behavior are said to be exhibiting yield flow behavior. The yield stress is therefore defined as the stress that must be applied to the sample before it starts to flow. Below the yield stress the sample will deform elastically (like stretching a spring), above the yield stress the sample will flow like a liquid.

Most fluids with yield stress can be considered as a structural skeleton that extends over the entire volume of the system. The strength of the skeleton is governed by the structure of the dispersed phase and its interactions. Normally, the continuous phase is low in viscosity, however high volume fractions of a dispersed phase can increase the viscosity by a thousand times and induce solid-like behavior at rest.

When a complex fluid that exhibits yield behavior is sheared at low shear rates, in the range between 0.01 -0.1 s^{-1} and below its critical strain, the system is subjected to work hardening. This is characteristic of solid-like behavior and results from elastic elements being stretched in the shear field. When such elastic elements approach their critical strain, the structure begins to break down causing shear thinning (strain softening) and consequent flow. The stress at which this catastrophic breakdown of the structural skeleton occurs is the yield stress.

There are a number of experimental tests for determining yield stress. A shear stress ramp is often employed as it is an easy and quick means of determining yield stress, however, a more accurate method is to perform series of creep tests and look for changes in the gradient of the compliance versus time curve [1].

Depending upon the nature of the material being tested, the creep response can be quite different as is illustrated in Figure 1.
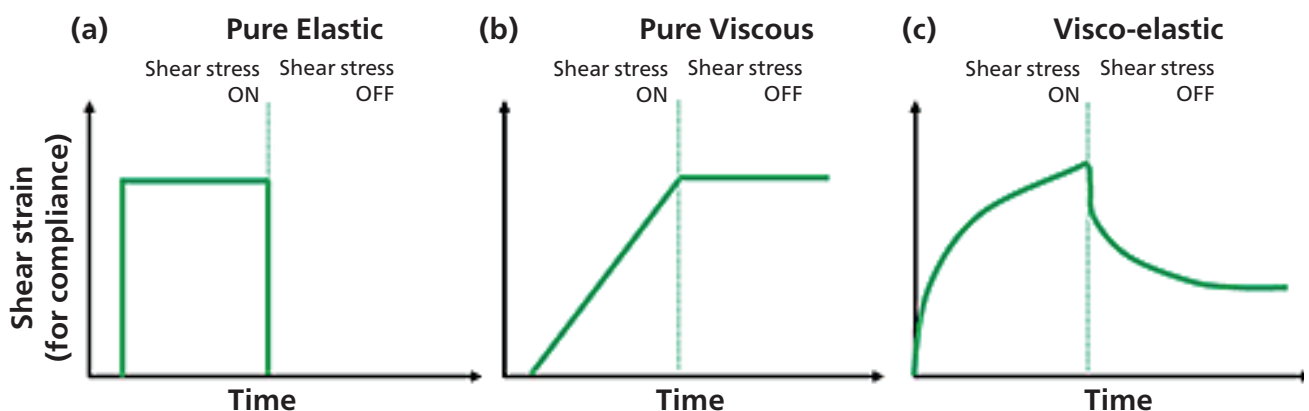
1 Schematic plots of strain versus time showing material responses to application of a shear stress (creep) and removal of shear stress (recovery) for different material types - (a) Elastic; (b) Viscous and (c) Viscoelastic

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Since the actual change of strain will be dependent upon the applied stress, it is usual to talk about the compliance rather then the strain. The creep shear compliance (J) can be determined from the preset shear stress (σ) and the resulting deformation (γ) through:

$$J(t) = \gamma(t) / \sigma_0 \qquad \text{Equation 1}$$

Using this notion, creep curves generated using different stresses can be directly compared. All J(t) curves overlap with each other independent of the applied stress as long as the stress is within the linear viscoelastic region. When this criterion is no longer met, the material is considered to have yielded. This is illustrated in Figure 2 from which it can be deduced for the sample under test that the yield stress is between 3 and 4 Pa since at 4 Pa, the curve no longer follows the same profile.

This Application Note shows methology and data from multiple creep testing for a moisturizing lotion.

Experimental

- A commercial moisturizing lotion was used as the sample under evaluation.

- Rotational rheometer measurements were made using a Kinexus rheometer with a Peltier plate cartridge and a cone and plate measuring system[2], and utilizing standard pre-configured sequences in rSpace software.

- A standard loading sequence was used to ensure that the sample was subject to a consistent and controllable loading protocol.

- A series of creep tests were run at seven different applied stresses between 30 Pa and 66 Pa.

- Each creep test was stopped after a set time (120 s), and a recovery test of equal time was subsequently performed between the creep tests.

- All rheological measurements were performed at 25°C unless specifically stated.
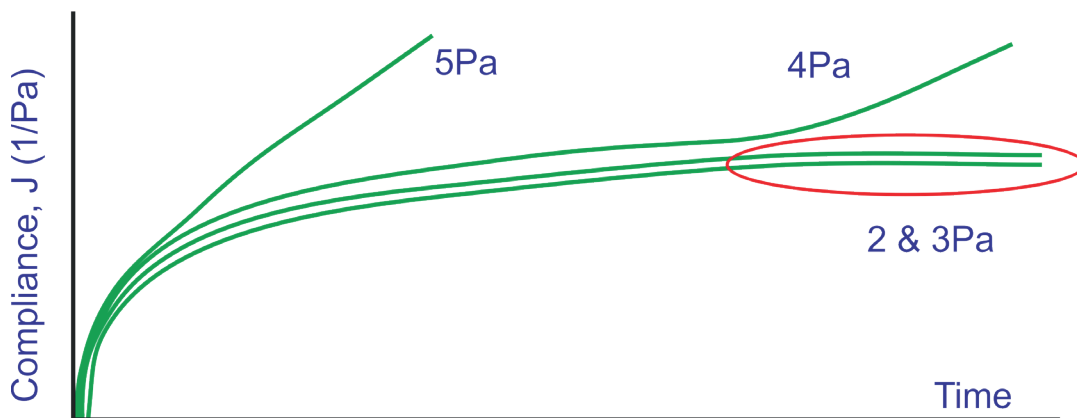
2 Illustration of multiple creep tests for a material with yielding at 4 Pa

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Results and Dicussion

Figure 3 compares creep compliance (*J*) with time at all seven stresses. Below 42 Pa, the compliance curves are superimposed and there does not appear to be an increase in compliance with time, suggesting that no flow is occurring below this stress i.e. the material is behaving as a viscoelastic solid.

At 48 Pa, there is a noticeable change in gradient indicating time dependent behaviour and hence viscous flow.

This is perhaps more clearly demonstrated in Figure 4, which shows the final compliance at each stress following the 120 second creep test. It can be inferred from the latter chart that the emulsion product has a yield stress between 42 and 48 Pa.

To achieve a more precise estimate of the yield stress, it would be necessary to repeat the test with small incremental increases in stress between these two values and evaluate in a similar manner.
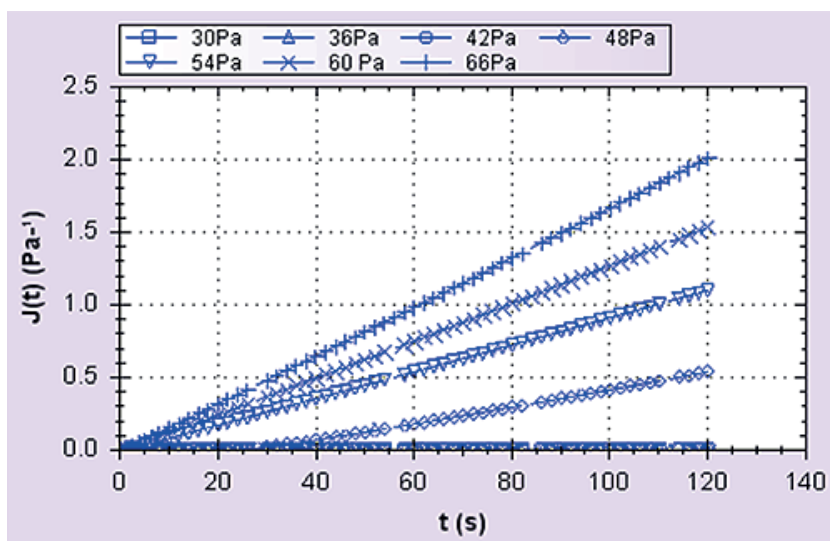
3 Creep measurements carried out on a moisturizing lotion at seven different stresses
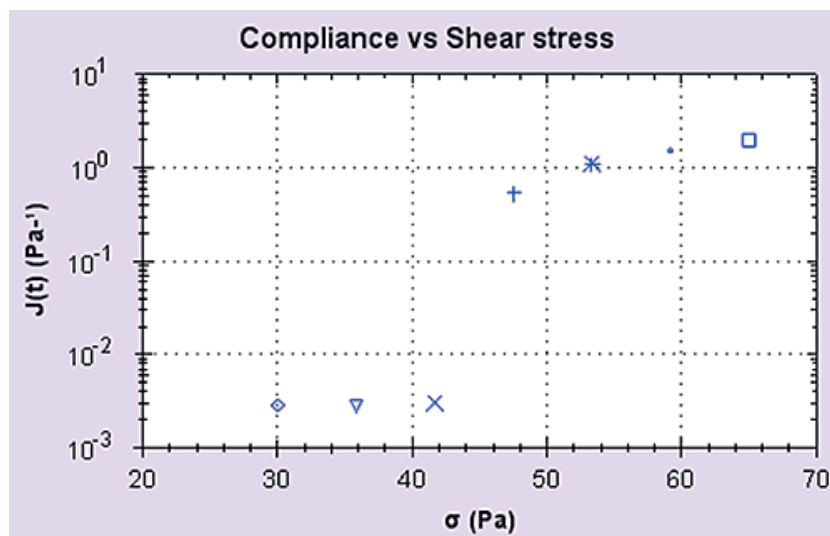
4 Compliance (after 120 s) as a function of shear stress for a moisturizing lotion

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Conclusion

For the moisturizing lotion tested, the maximum stress where the compliance is within the linear viscoelastic region is 42 Pa, while at 48 Pa the yield stress is exceeded. The yield stress therefore has a value between 42 Pa and 48 Pa.

For a more precise value of yield stress for this material, further test iterations within this narrow stress band are required.

Multiple creep testing to derive yield stress is an accurate method, but can require multiple iterations and correct user interpretation.

References

[1] White Paper – Understanding Yield Stress Measurements, NETZSCH Instrument

[2]Please note …

that a parallel plate geometry can also be used – with this geometry being preferred for dispersions and emulsions with large particle sizes. Such material types may also require the use of serrated or roughened geometries to avoid artifacts relating to slippage at the geometry surface.

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