



Normal Force Measurement on the *Rheolyst* series AR 1000-N Rheometer - PART I

Introduction:

When certain viscoelastic samples are sheared, a force normal to the plane of the applied shear is produced, this is called the normal force (or normal stress if the correct geometry factors are considered). The most obvious display of normal force is the rod-climbing phenomenon more commonly referred to as the *Weissenberg effect*. When a rotating rod is dipped into a shallow vessel containing an elastic liquid the liquid actually climbs the rod whereas a Newtonian liquid would be forced outwards by inertia. (See figure 1.)

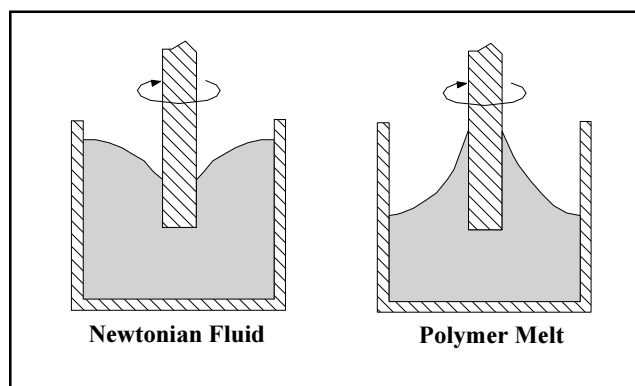


Figure 1
The Weissenberg Effect

The presence of normal forces has several implications in practical situations particularly in the manufacture of rods, tubes and sheets of polymeric material. Such items are produced by the extrusion of molten polymer which, being an elastic liquid, can exhibit post-extrusion swelling, (commonly known as die swell,) a direct consequence of normal force. (See figure 2.) This can lead to uncontrollable variations in the thickness of any articles produced in this manner. By determining the magnitude of the normal force generated under shear this die-swell phenomenon can be allowed for during processing.

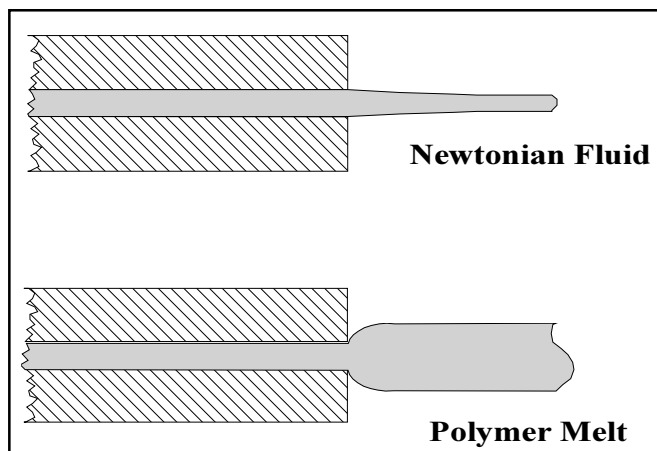


Figure 2
The Die Swell Phenomena

Another consequence of normal force, of importance commercially, is in laminar mixing processes. In inelastic liquids the resulting flow patterns arise from the contribution made by both the viscous and inertial forces. The liquid is forced out radially from the impeller. (See figure 3 - a.) For highly elastic liquids the direction of flow can be completely reversed and the Weissenberg effect predominates. (See figure 3 - b.) There are some liquids in which both types of flow pattern occur. (See figure 3 - c.)

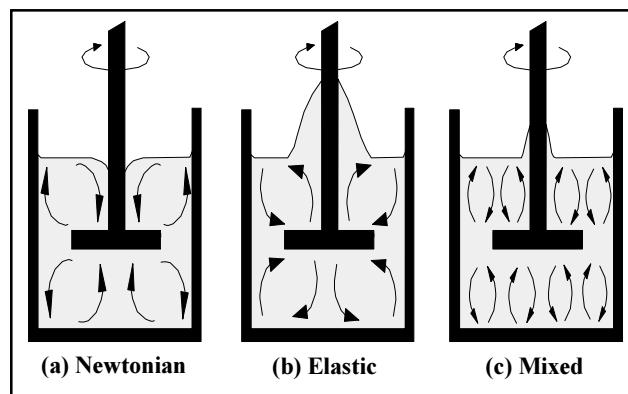


Figure 3
Flow Patterns

Unfortunately in the practical situation, if you are trying to mix two different liquids it is unlikely that each would show the same flow pattern and therefore achieving good miscibility can sometimes be difficult. The type of flow pattern obtained in a given situation will depend upon the dominance of either the elastic or inertial forces.

The Normal Force Transducer:

The normal force transducer on the TA Instruments AR 1000-N rheometer uses the latest high sensitivity load cell technology. This provides a very robust, virtually deflectionless measurement system capable of detecting normal forces in the range 0.01N to 50N. The transducer measures the raw force but this can be expressed via the software as a normal stress once the appropriate geometry factors are used.

There are several ways in which this normal force transducer can be utilized, namely:

- measurement of normal forces generated during shear
- monitoring of normal forces generated during loading
- loading under normal force control

Normal Force & Elasticity:

Apart from being able to determine whether a material will exhibit die swell during extrusion or whether two liquids will mix well, measuring normal force under steady shear conditions can also be related to the amount of elasticity. Highly elastic samples will correspondingly exhibit high normal forces.

Figure 4 shows a comparison of two samples of low viscosity motor oil whose elasticity, under high stresses was required at sub-ambient temperatures. It would not be possible to determine the elasticity in the conventional dynamic mode because high stresses on low viscosity samples means the displacements would be too high. Using a continuous flow experiment at the high stresses required and measuring the resultant normal forces dramatically and effectively shows the difference in the elasticity. Sample A has a more elastic structure than Sample B.

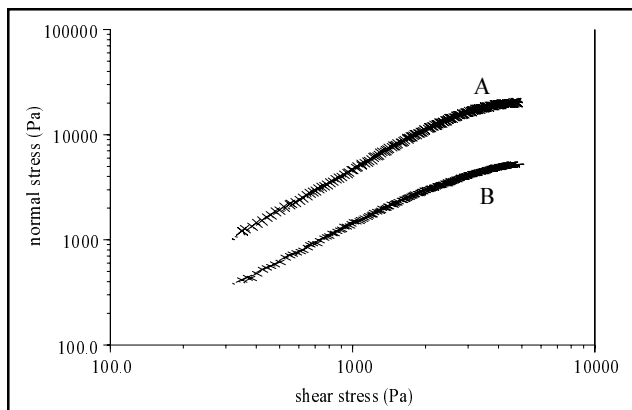


Figure 4

Low Viscosity Motor Oils at low temperatures

Normal Force During Loading:

When a sample is compressed between the two plates of a rheometer there can be appreciable upthrust force generated which will decay once the compression stops or slows down. (See figure 5)

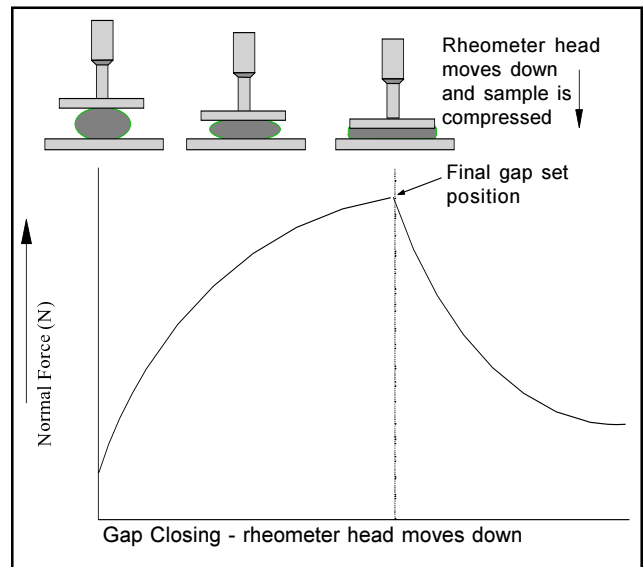


Figure 5

Normal force during loading

The magnitude of these forces will be entirely sample dependent and if not monitored or at least accounted for may lead to reproducibility problems in any subsequent data produced. This effect is particularly noticeable in stiff samples such as polymer melts, which have the additional problem of uncontrolled internal stresses occurring during the melting process. By monitoring the normal force during loading and using it to control the loading process these problems are significantly reduced for most samples.

However even fairly weakly structured materials can exhibit normal force during loading. By recording how the normal force changes during loading it can be ensured that successive samples are loaded under the same conditions and hence validity of the subsequent data can be confirmed. By optimising the initial closure velocity and the peak normal force value preservation of sample structure during loading is also assured. Typical data showing the reproducibility of loading by monitoring normal forces generated during loading a weakly structured gel is shown for two successive samples in figure 6.

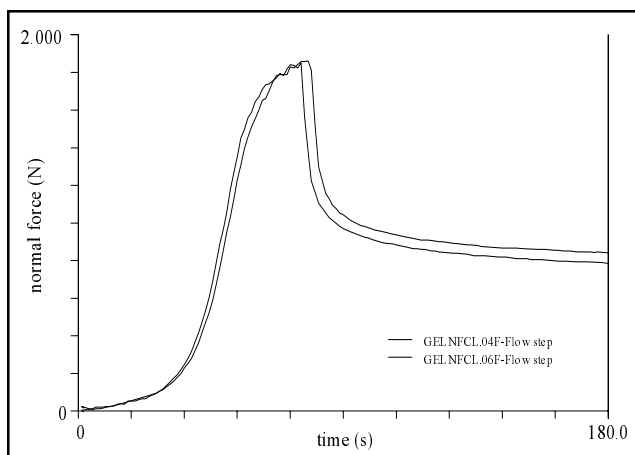


Figure 6
Normal force monitoring during loading

Loading under normal force control:

The rheometer head will initially move down towards the sample at a pre-programmed user defined velocity. As soon as the sample contacts the upper geometry and a normal force is registered the rate of descent will decrease proportionally to the rate at which the normal force is increasing until a user entered peak normal force value is reached. At this point the velocity of descent will drop to zero until the sample has relaxed so that the normal force is again below the entered peak value. The head will then continue downwards in the same manner as previously described until the geometry gap is set. At this point the normal force will decay. The length of time for the sample to

relax and the normal force to decrease to zero will vary from minutes to hours depending upon the sample under test. The user then has the option to either wait until the decaying normal force reaches equilibrium or start the experiment once the normal force has dropped below a user defined value.

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