PN009

Measuring Normal Force

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Shear normal stresses can be most easily measured from the total thrust in cone and plate or parallel plate geometries in rotational rheometers. The ARES-LS uses a patented rebalance transducer with quasi infinite stiffness (Product brief: FRT technology) to measure total thrust and an air bearing supported motor to reduce system compliance to a minimum and to eliminate axial run-out so typical for ball bearings. The ARES-LS – thus is a rotational rheometer specifically designed and optimized to make accurate normal stress measurements.

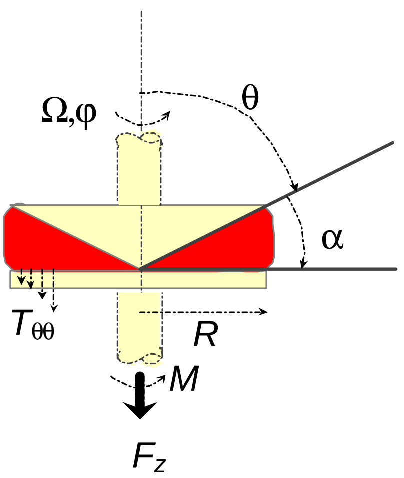

Figure 1. Schematic of a cone and plate rheometer.

THE CONE PLATE FLOW CELL

In a cone and plate flow cell, the shear strain is given as

$$g = j / \sin a \qquad (1)$$

where j is the angle of rotation and a is the angle between the cone and the plate (see Figure 1). For a small cone angle $\sin a = a$, thus .

$$g \cong j / a \qquad (2)$$

Similarly the shear rate can be approximated by

$$\dot{g} = \Omega / a \qquad (3)$$

where Ω is the angular totation rate. Even for α = 0.2 rad, errors resulting from these approximations are less than 3%.

The total thrust, F_z, between the plate and cone gives the first normal stress difference

$$T_{11} - T_{22} = N_1 = \frac{2F_z}{pR^2} \qquad (4)$$

Note: For flow between parallel plates, the shear strain and strain rate are not constant but depend on the radial position r:

$$g_r = \frac{jr}{h} \qquad (5)$$

and

$$\dot{g}_r = \frac{\Omega r}{h} \qquad (6)$$

where h is the gap between the plates. The trust between the plates depends on both the first and the second normal stress differences

$$T_{11} + T_{22} = (N_1 - N_2)N_1 - N_2 = \frac{F_z}{pR^2}\left[2 + \frac{d\ln F_z}{d\ln \dot{g}_R}\right] \qquad (7)$$

Thus in order to accurately evaluate $N_1 - N_2$ with a parallel plate rheometer, not only does one need to measure the thrust, F_z, but also how it changes with shear rate or rotation rate W. For 1 <$d\ln F_z/d\ln g_R$ <2.5 /1/ equation above reduces to:

$$(N_1 - N_2) = \frac{4F_z}{pR^2} \qquad (8)$$

where $(N_1 - N_2)$ is evaluated at $\gamma_r = 0.76g_R$. With accurate N_1 *data* from cone and plate and $(N_1 - N_2)$ from parallel plates N_2 can be determined by the difference.

MEASUREMENT OF N_1

Although the equations given above are fairly straight forward, care must be taken to obtain accurate normal stress measurements. Most problems come with highly viscous and elastic samples at elevated temperatures, i.e., typically polymer melts /2/. It is difficult to reach high shear rates since the polymer is unstable at the free surface. By using smaller cone angles (or gaps, *h*, in parallel plates) somewhat higher shear rates can be obtained. However, small cone angles can cause severe errors in transient (time dependent) tests. The normal thrust will cause the instrument to deflect slightly. This pushes open the gap by a small amount and the sample flows toward the center of the cone. This cross flow retards the true normal force reading. The smaller the cone angle, the longer the material takes to flow. The consequences on the normal stress build up in a start up experiment are shown in Figure 2. After 15 s the data from tests with all five cone angles agree; but the true normal force overshoot is recorded at short time only for cone angles of 0.1 rad or larger /3/. According to Hansen & Nazem /4/ the instrument response time can be estimated from:

$$l_{inst} \approx \frac{6pRh_o}{K_Z a^3} \qquad (9)$$

For good transient normal stress response l_{inst} must be small. The ARES LS uses a unique rebalance transducer, which provides high stiffness, $K_z = 1kgf/mm$, yet high sensitivity. From equation 8, the strong effect of increasing cone angle, as shown in Figure 2 becomes evident. Decreasing radius also helps reduce l_{inst} .For polymer melts a cone with a > 0.1 rad and R < 12.5 mm is an optimized geometry for the ARES system, with a system response time of < 1 s for a melt with a viscosity of 10^4 Pas.

Higher system stiffness, will only marginally

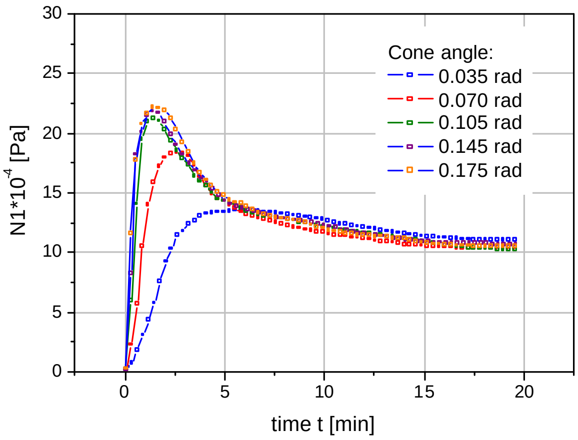

Figure 2. Normal stress vs. time upon start-up of steady shear, ~ =10 s-1; low density polyethylene with ?o =5 i0⁴ Pa S. Cone diameter 24 mm and five different angles. (replotted from Meissner, 1972).

affect the system response time, but would make the normal force measurement virtually impossible, due to the noise arising from temperature, i.e. density fluctuations. For a typical polymer melt expansion coefficient (10^{-4} 1/°C), temperature fluctuations of 0.1 °C, cause dilatation fluctuations of 0.2μm (gap 2mm in parallel plate geometry). These fluctuations in a stiff system superimpose on the real normal force measurement – thus can reduce the system normal force sensitivity significantly

Another problem with small cone angles or small gaps and viscous samples is the great N_1 sensitivity to instrument alignment. Small gaps also make it difficult to load samples. It can take hours for stresses to relax, especially if the material has a yield stress. Shearing very slowly during and after loading can spread relaxation. The AutoGap function, standard on ARES allows automatic loading of high viscous samples. However, the best solution to reduce the loading time is to pre-mold a specimen to the right size and insert between the cone and plate.

Measuring normal stresses for lower viscosity systems like polymer solutions is easier but still has pitfalls. Inertia of the fluid itself causes a normal force opposite the elastic one at high rotation rates /5/. This effect can be approximately corrected by:

$$F_{Z,inertia} = -0.075 p r \Omega^2 R^4 \qquad (10)$$

The preferred boundary condition is to use a cone and a plate of the same diameter and to fill the sample to the edge as illustrated in Figure 1. If a larger plate is used, a “flooded” condition will result and lead to increased N_1 readings.

EXPERIMENTAL HINTS

For high viscous materials, it is best to trim the sample, at a position, 20 to 50mm above the final gap setting. During the final gap setting holes and fissures created during the trimming operation are filled again. This is important, as the outer radius contributes very strongly to the total force i.e. torque. For low viscous fluids, an overflow condition is much easier to control – thus improves the reproducibility of the test results. The down side is a systematic error in the result.

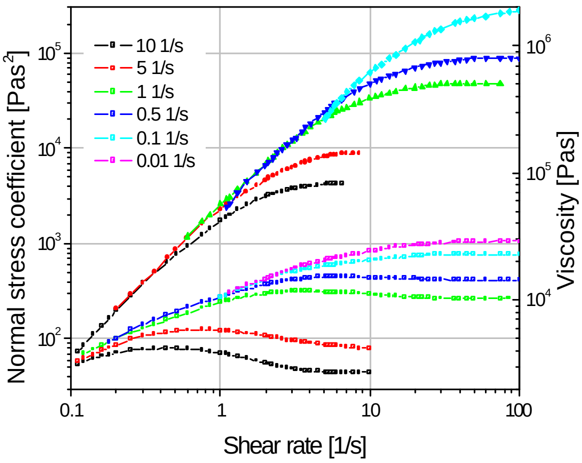

Figure 3: First normal and viscosity data determined in start up experiments at different shear rates for a LDPE

TYPICAL RESULTS FOR LDPE

Figure 3 shows the viscosity and the first normal stress traces for a standard LDPE at different shear rates. Note that steady state could not be reached in normal force. The viscosity as well as the normal force trace exhibits an overshoot, when high shear rates are being applied. At lower shear rates, all the data fall onto the linear viscoelastic curve.

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