Determination of normal stress differences in steady shear flow

I. Stability of a polyisobutene liquid

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With 3 figures

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Introduction

Since the original work of Weissenberg (1) Russell (2) and Garner and Nissan (3), many measurements of the two differences of normal stress components in shear flow of high polymer solutions and melts have been reported in the literature, but comparatively little attention has been devoted to the problem of validating the methods used. Such results as have been published yield conclusions which vary according to the test used: Adams and Lodge (4) found inconsistency between values of rim pressure and pressure gradients in cone-and-plate and plate-and-plate systems, while Markovitz (5) found consistency between values of pressure gradient in cone-and-plate and plate-and-plate systems and pressure difference in a concentric cylinder system.

A similar situation exists in relation to the additional test which is furnished by flow birefringence data if one makes the assumption that stress and refractive index ellipsoids are coaxial – an assumption which is very reasonable for solutions in which solute and solvent mean refractive indices are so nearly equal that effects of "form birefringence" can be neglected.

Philippoff (6) found consistency between values of extinction angle, shear stress, and total thrust in a cone-and-plate system; Lodge (4) found inconsistency between values of extinction angle, shear stress, and pressure gradients in cone-and-plate and plate-and-plate systems.

In these circumstances, it does not seem possible to place much confidence in any normal stress measurement. In an attempt to clarify the situation, we have embarked on a more extensive series of measurements involving apparatus of each of the kinds referred to above, used under carefully controlled conditions. The results will be presented in this and in subsequent papers. A brief notification of some tentative conclusions based on our first such set of data has been published elsewhere [Broadbent et al. (8)].

In the present paper (1), we show that pressure gradients in a cone-and-plate apparatus can be measured very accurately and we then use such data to establish the homogeneity and the stability, over a period of days, of a certain polyisobutene liquid which exhibits large normal stress effects. A liquid of this type is used in the subsequent extended series of measurements (paper II).

Pressure gradient accuracy

In the apparatus of Adams and Lodge (4), a polymer solution is sheared between a cone or plate rotating about a vertical axis and a horizontal stationary plate. In the latter, small holes filled with static liquid are connected to diaphragm-capacitance transducers which enable measurements to be made of the pressure \( p(r) \) which the sheared liquid exerts on the plate at a distance \( r \) from the axis of rotation. Although the scatter in the values of \( p(r) \) measured on this apparatus was never great, it was particularly noticeable in the values assigned (by extrapolation) to the "rim pressure" \( p(R) \), where \( R \) denotes the radius of the rotating member.

In the hope of reducing the scatter, the following minor changes were made before the present series of measurements were begun: (a) in place of three Negretti and Zambra inclined-tube manometers, a single Casella micromanometer (of variable-height flexible U-tube type) was used to measure air pressure on the three diaphragm-capacitance gauges; (b) the end movement in the rotating member was reduced from about 30 to about 2 microinches; (c) a stationary plate having pressure-measuring holes of diameter 1.3 mm (instead of 0.6 mm) was used; (d) the fluctuation of temperature in the water bath (from which water is circulated to keep plate and pressure transducers at...
constant temperature) was reduced so that the rate-of-change of temperature did not exceed 0.007 C deg/min.

We believe that (a) gave a more uniform accuracy over an increased range; although it is not possible to be certain about this (because we have been unable to find any instrument, of guaranteed accuracy, for the measurement of differential pressures in the region of 1 cm water gauge about atmospheric pressure, against which our manometers might be calibrated), we did find disagreements of up to 4.4% between readings taken with the three inclined-tube manometers when these were calibrated using the Casella micromanometer as a standard. We believe that (b) reduced the superimposed periodic pressure attributed to end movement of the rotating member; the arithmetic mean of the maximum and minimum values of this periodic pressure, which was used as a measure of the required pressure \( p(r) \), should therefore be less liable to error; this should reduce the scatter more at the smaller values of \( r \), since the amplitude of the periodic pressure increases as \( r \) decreases. We did, in fact, use a larger range of \( r \)-values than before. Finally, we believe that both (c) and (d) resulted in greater stability of the zero of the pressure transducer.

Fig. 1 presents some of the first results taken with the apparatus of Adams and Lodge after these modifications were made. The solution contained 6 gm polyisobutene ‘Oppanol B 100’ (\( M_w = 1.1 \times 10^6 \)) in 100 ml ‘Oppanol B 1’ (viscosity 0.24 poise at 25 °C; \( M_w = 400 \)). All measurements in this paper were taken at 25.0 °C. Each point \( \bar{p} \) represents the arithmetic mean of two pressure values recorded using two senses of rotation of the cone; the difference between these two values was always small (less than 50 dyn/cm²).

Contributions to the pressure distribution arising from inertial forces, estimated by means of the expression \( \rho \omega^2 r^2/20 \) (9), where \( \rho \), \( \omega \) denote density and angular velocity, do not exceed 1 dyn/cm², and are therefore negligible. The relation between \( p(r) \) and \( \log r \) should, therefore, be linear; this expectation is very well borne out by the data of fig. 1.

A straight line, fitted to the six data points of fig. 1 by means of the method of least squares, has a slope whose standard deviation is only 0.65% of the slope. Moreover, much of this standard deviation arises from the two points which have a common value of \( r \); since these points represent pressures recorded by two transducers connected to holes which were simultaneously on opposite sides of (and equidistant from) the axis of rotation, it is likely that the difference in the values of pressure was the result of a systematic error arising from a slight tilt in the stationary plate. Such an error can be substantially eliminated by taking the arithmetic mean of the two pressure readings (4). When this is done, the resulting five points in fig. 1 give a straight line whose slope has a standard deviation equal to 0.27% of the slope. The 95% confidence limits for this slope are ± 0.86% of the slope. In this sense, we can say that the pressure gradient, \( r \) \( \partial p/\partial r \), can be measured to an accuracy of 1% for this solution.

The fact that \( \bar{p} \) is found to vary linearly with \( \log r \) does not preclude the possibility that the values of \( \bar{p} \) were subject to systematic errors arising from (i) secondary flow, and (ii) perturbation of the velocity field by the holes used for pressure measurement.

It is known that the ‘primary flow’ (in which each infinitesimally thin liquid cone coaxial with the rigid cone rotates rigidly about the common axis) cannot satisfy the stress equations of motion (even when inertial terms are omitted) and that in consequence a ‘secondary flow’ (in which liquid elements have non-zero velocity components