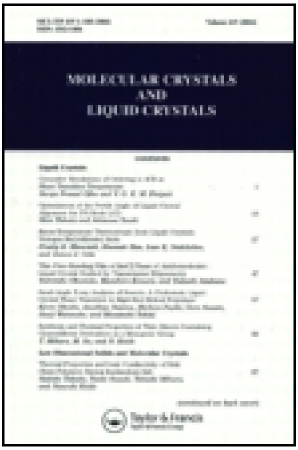

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RHEOLOGICAL PROPERTIES OF ARAMID SOLUTIONS: TRANSIENT FLOW AND RHEO-OPTICAL MEASUREMENTS.

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Abstract The rheological properties of a 20% (w/w) solution of poly-para-phenylene-therephthalamide (PpPTA) in concentrated sulfuric acid have been investigated using a Contraves Rheomat 135S and a device that allows microscopic investigation of the solution during transient shear flow using polarized light.

INTRODUCTION

Lyotropic solutions of poly-para-phenyleneterephthala-mide (PpPTA) in sulfuric acid are used to produce high-modulus and -strength fibres. Rheological properties of these solutions were studied by measuring the transient shear stress and by a rheo-optical method.

A 20% (w/w) solution of PpPTA in 99.8% H_2SO_4 was used. The average molecular weight Mw was about 30.000. A nematic lytropic phase is found from 70 - 120°C. Higher temperatures cause rapid degradation.

TRANSIENT SHEAR-STRESS MEASUREMENTS

We used a Contraves Rheomat 135S for the transient shear-stress measurements. This has a Couette type geometry. In Figure 1 an interrupted shear-stress measurement is shown.

The initial oscillations in the shear-stress are

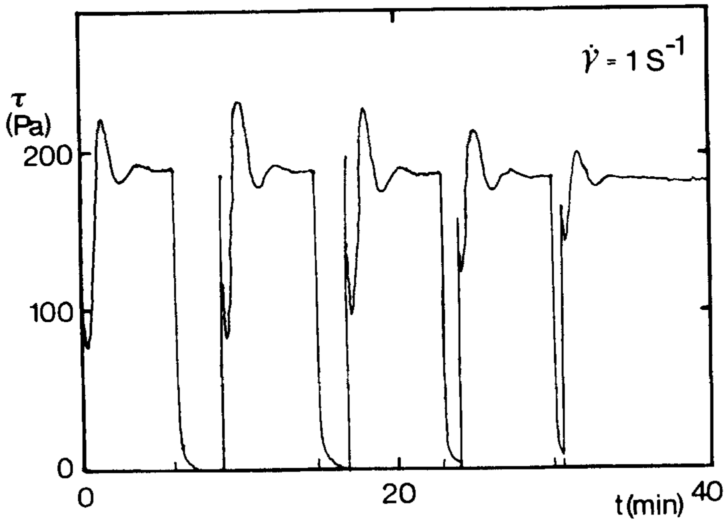

FIGURE 1. Interrupted shear stress measurement.

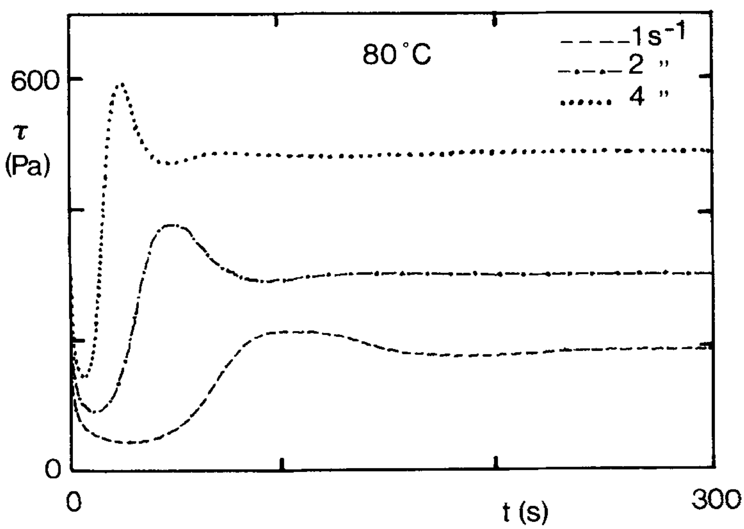

FIGURE 2. Shear-stress versus the elapsed time after the start-up of the shear flow.

reproduced reasonably well after waiting for 180 seconds or more. Complete stress relaxation takes about 60 seconds.

The oscillations are shown in Figure 2 for several shear rates. The steady-state shear stress is shown versus the shear-rate in Figure 3.

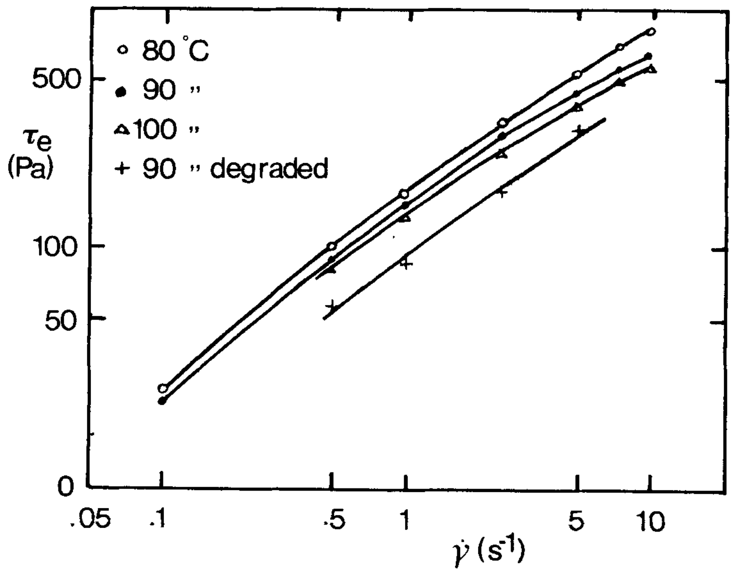

FIGURE 3. Steady-state shear stress versus the shear-rate.

TABLE I Power-law parameters of PpPTA solutions.

Temperature (°C)	K (Pa)	n
80	166	0.70
90	146	0.66
100	133	0.65
90*	82	0.80

* degraded sample

A power-law type of behaviour is observed:

$$\tau_e = K\,(\dot{\gamma})^n \tag{1}$$

some values of K and n are given in Table I.

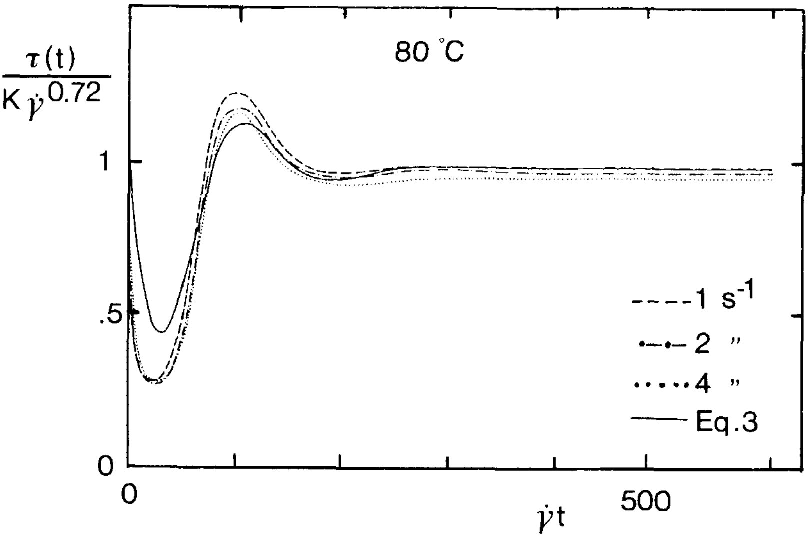

FIGURE 4. Transient shear-stress measurements using the dimensionless variables of Eq. 2.

As the period of the oscillations decreases with increasing shear-rate and their amplitude is proportional to the steady-state shear-stress, we find the following dimensionless variables:

$$\dot{\gamma}t \text{ and } \frac{\tau(t)}{K(\dot{\gamma})n} \tag{2}$$

that give rise to Figure 4. An approximate equation for the transient shear-stress is

$$\tau(t) = K(\gamma)^n \left(1 - e^{-\frac{\dot{\gamma}t}{k_1}} \sin \frac{\dot{\gamma}t}{k_2}\right) \qquad (3)$$

This is also shown in Figure 4.

The parameters k_1 and k_2 are about 58 and 25[1] respectively. The oscillations in the transient stress are governed by the total shear $\dot{\gamma}$ t. The shear-rate only determines the absolute value of the measured shear-stress level.

RHEO-OPTICAL MEASUREMENTS

For these measurements a device was built that provides a controlled shear flow between microscope slides. A similar instrument has been described by Alderman and Mackley[2]. The distance between the glass slides is determined by tungsten spacers (25, 50, and 100 µm).

Shear-rates from 0.25 s^{-1} to 1000 s^{-1} are possible. A Eurotherm 422 temperature controller and a Delta SM 6020 power supply coupled to a Leitz 350 hot-stage regulate the temperature (20 - 450°C). The temperature stability is better than 0.3°C. The shear-flow cell and hot-stage are mounted on a Leitz Orthoplan-Pol polarising microscope with a Leitz Vario-Orthomat II camera. Magnifications of 50 to 500 times are possible. The photo-multiplier of the camera is used to measure the transmitted light intensity.

The texture during flow shows a rather grainy structure giving the impression of small "regions" tumbling over each other. The apparant size of the "regions" decreases with increasing shear-rate. The "regions" are not elongated during the shear flow. After cessation of the flow a banded texture is formed[3]. The distance between the bands increases in about 60 seconds from 2 μm to about 15 μm.

The light intensity transmitted by the sample (crossed polarisers) is shown as a function of the total shear (γt) in Figure 5. The flow was parallel to the polariser. An oscillaton like with the shear-stress measurements is observed. In Figure 6 the oscillation time is shown versus the shear-rate. Again the oscillations are related to the total shear.
There is a reasonable agreement between the rheo-optical and the shear-stress measurements. Depending on the layer thickness one find k_2 values ($k_2 = \dot{\gamma}$ Tosc./(2π)) of about 35 - 50.

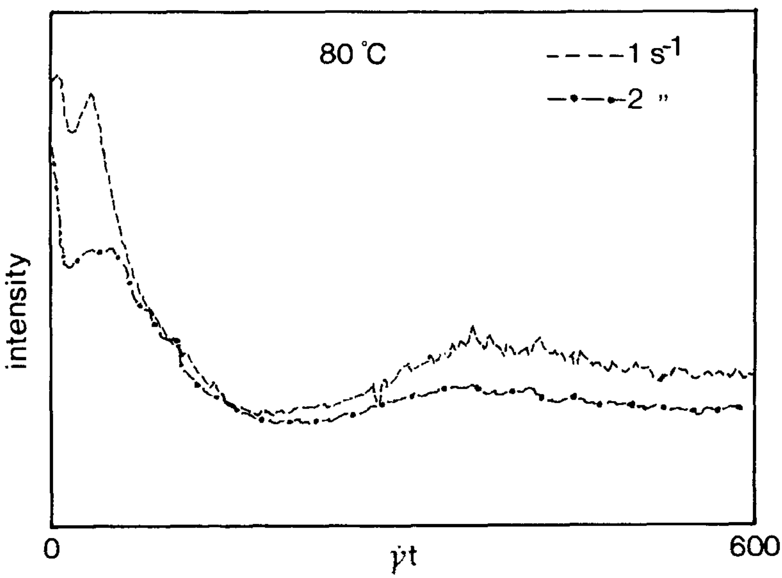

FIGURE 5. Transmitted light intensity (crossed polarisers) versus the total shear after start-up of the flow. The graphs have been displaced vertically for easy comparison.

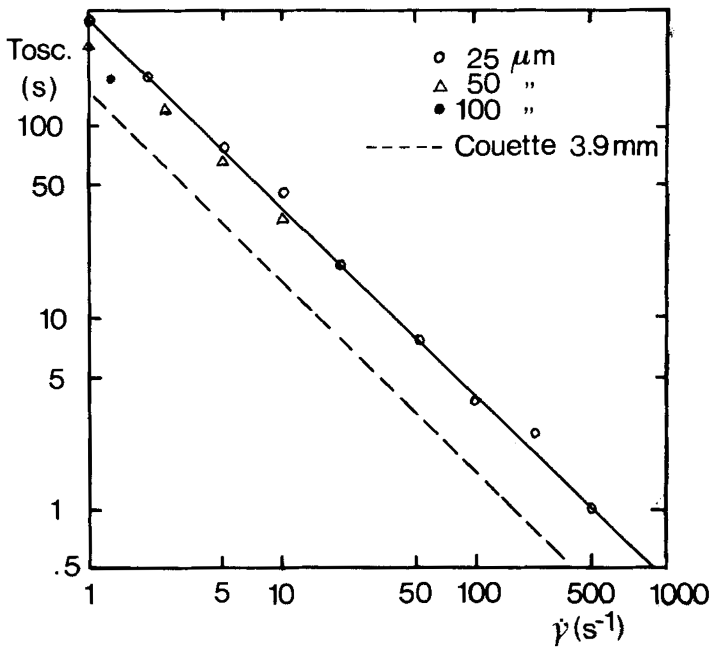

FIGURE 6. Oscillation time in the transmitted light-intensity versus the shear rate. The results from the shear-stress measurements are indicated by the dotted line.

An impression of the degree of alignment of the director can be obtained from the depolarisation ratio $I_{\perp}/I_{\parallel}$ (perpendicular and parallel analyser) as shown in Figure 7. The alignment of the director seems to improve with increasing shear-rate despite the fact that even at high shear-rates the "grainy texture" remains visible.

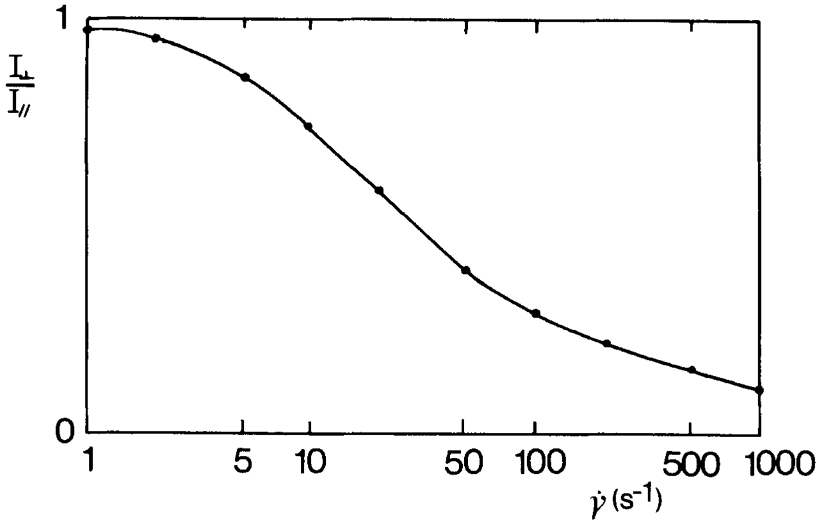

FIGURE 7. Depolarisation ratio $I_\perp/I_{//}$ versus the shear-rate $\dot{\gamma}$.

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