terms in the normal stresses. Thus, in concentrated polymer solutions substantially decreased stability may often be expected relative to a Newtonian liquid, and such behavior has been observed experimentally (6).

ACKNOWLEDGMENT

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NOTATION

a = wave number
$\mathbf{A}_n$ = *nth* Rivlin-Ericksen tensor
D_n = grouping defined following Equation (16)
$\mathbf{F}$ = stress function in simple fluid theory
$\mathbf{F}_n$ = functions of invariants of A_1 and A_2
$\mathbf{I}$ = identity
J_n = grouping defined following Equation (16)
M = $1 - \Omega_2/\Omega_1$
p = hydrostatic pressure
P_1,P_2 = reduced viscometric functions
Q_n = grouping defined following Equation (16)
r = radial variable
R = inner cylinder radius
R_n = grouping following Equation (16)
S_n = $n^2\pi^2 + a^2$
t = time
T = Taylor number
u = radial velocity perturbation
$\overline{u}$ = transform of u
U_{pm} = grouping defined following Equation (18)
v = angular velocity perturbation
$\overline{v}$ = transform of v
$\mathbf{v}$ = velocity vector
V = undisturbed angular velocity
w = axial velocity perturbation
W_{pm} = grouping defined following Equation (18)
x = reduced radial distance
z = axial distance

Greek Letters

$\alpha_1,\alpha_2,\alpha_3$ = viscometric functions
Γ = shear rate
δ = gap width
θ = angular coordinate
ρ = density
σ = exponential growth factor
$\boldsymbol{\tau}$ = stress tensor
Ω_1,Ω_2 = angular velocities of cylinders

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Rotational Stability and Measurement of Normal Stress Functions in Dilute Polymer Solutions

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The onset of rotational flow instability was determined experimentally for dilute solutions of six polymers. In general, the onset of secondary motions occurred later than for a Newtonian liquid of equal apparent viscosity, although, depending upon geometric ratios, some instabilities occurred earlier than in the Newtonian liquid. The stability theory of Ginn and Denn was used to calculate viscometric normal stress functions for ten solutions, with excellent agreement obtained with rheogoniometric measurements of the primary normal stress measurements in all but three cases. Torque measurements of the laminar secondary motion following instability showed some frictional drags significantly less than those observed in Newtonian liquids, a phenomenon analogous to the turbulent drag reduction observed previously in dilute polymer solutions.

The onset of instabilities in rotational Couette flow of dilute polymer solutions must depend not only on inertial stresses, as in a Newtonian liquid, but upon the total rheological description of the material. In the theory developed by Ginn and Denn (*11*) the critical Taylor number, defining the rotational speed at which secondary motions begin, is related to both of the viscometric normal stress functions. Depending upon the relative magnitudes of the two normal stress functions and the algebraic sign of the secondary normal stress difference instabilities might occur

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either earlier or later than in a Newtonian liquid of the same apparent viscosity.

The stability theory is very sensitive to small changes in the value of the secondary normal stress difference. Several investigators have reported measurements of this quantity in a variety of liquids (*3, 10, 12 to 14, 21*) but the experimental difficulties are severe and there is disagreement over even the proper algebraic sign of the secondary normal stress difference in similar materials. This precludes a direct experimental test of the accuracy of the stability theory, but does suggest an inverse procedure in which reproduceable stability phenomena might be employed as a means of measuring the physical properties of dilute polymer solutions and, incidentally, as a check on the theory. This paper reports on an experimental study of the rotational flow stability of several dilute polymer solutions and the usefulness of such measurements and stability theory in predicting both primary and secondary normal stress differences in dilute solutions.

BACKGROUND

When a liquid is sheared between long concentric cyclinders the flow pattern which first exists is one of circular streamlines in a plane normal to the axis of rotation. This is the flow pattern which is assumed to occur in Couette viscometers in relating torque measurements to viscosity (*3, 7*). At some rotational speed this flow becomes unstable and a small axisymmetric cellular secondary motion becomes superposed on the primary flow. Unlike the transition to turbulence in a pipe, the secondary motion in rotational Couette flow is a stable laminar flow with streamlines which are independent of time.

The onset of the secondary motion may be observed either visually, using dye or bubble tracer techniques, or through torque measurements, for the slope of the torque-shear rate curve will be discontinuous as a result of the increased frictional drag resulting from the secondary flow. For any liquid, considerations of dimensional analysis require that the point of instability occur at a critical value of the dimensionless group known as the "Taylor number,"

$$T = \frac{2\rho^2\Omega^2\delta^3 R}{\alpha_1{}^2} \tag{1}$$

Here, R is the outer radius of the inner cylinder, $R + \delta$ the inner radius of the outer cylinder, which is assumed stationary, Ω the angular velocity of the inner cylinder, ρ the density, and α_1 the shear-dependent viscosity of the liquid. This is the only dimensionless group which appears in the flow equations for a Newtonian liquid, and the critical value is found to be 3,390. Excellent agreement is obtained between theory and experiment, with the pertinent results summarized in the monograph of Chandrasekhar (*1*).

Two additional dimensionless groups, known as "Weissenberg numbers," arise in considering the flow stability of dilute polymer solutions. These groups reflect the ratio of each of the viscometric normal stress differences to the inertial stresses as follows:

$$P_1 = \frac{\tau_{11} - \tau_{22}}{\Gamma^2} \frac{1}{2\rho\delta^2} = -\alpha_3/\rho\delta^2 \tag{2}$$

$$P_2 = \frac{\tau_{22} - \tau_{33}}{\Gamma^2} \frac{1}{2\rho\delta^2} = (\alpha_2 + 2\alpha_3)/2\rho\delta^2 \tag{3}$$

Here, the viscometric flow is in the 1 (angular) direction with velocity variations in the 2 (radial) direction. The shear rate, Γ, is very nearly equal to the constant

$$\Gamma = R\Omega/\delta \tag{4}$$

and the stress tensor is related to the deformation through the constitutive equation

$$\boldsymbol{\tau} + p\mathbf{I} = \alpha_1\mathbf{A}_1 + \alpha_2\mathbf{A}_1{}^2 + \alpha_3\mathbf{A}_2 \tag{5}$$

p is a hydrostatic pressure; α_1, α_2, and α_3 are functions of the shear rate; and $\mathbf{A}_1$ and $\mathbf{A}_2$ are the first two Rivlin-Ericksen acceleration tensors (*3*). The stability theory developed by Ginn and Denn (*11*) for dilute polymer solutions relates the critical Taylor number to the values of P_1 and P_2.

The first experimental study of the rotational stability of dilute polymer solutions was carried out by Merrill, et al. (*15*), who generally found little deviation from Newtonian behavior, although they reported significant destabilization for some solutions of polyisobutylene. Their fluids were not characterized in terms of normal stress behavior, so no comparison with theory is possible. Denn and Dugan (*5*) reported significant elastic effects, but these appear to have been a spurious consequence of the data reduction techniques required by their experimental design. Rubin and Elata (*18*) studied dilute aqueous solutions of three polymers and found substantial stabilization, with Taylor numbers as high as 7,000. They could measure no change with Taylor number in the wave number, which determines the axial length of a cell, while the theory of Ginn and Denn requires the wave number to be a slowly decreasing quantity with increasing Taylor number. Rubin and Elata also reported no normal stress information. Finally, Giesekus (*8*) found substantial destabilization for two concentrated solutions to which the theory might not apply, although qualitatively the concentrated solutions could be expected from the theory to have small Taylor numbers. He also reported no normal stress information.

The functions α_1 and α_3 may be easily measured for moderately dilute polymer solutions by conventional viscometric techniques (*3, 7*), but accurate measurements of α_2 (in practice, of $\alpha_2 + 2\alpha_3$) are quite difficult. The strong dependence of the critical Taylor number on P_1 and P_2 suggests a procedure for using the well-defined onset of secondary motions as a means of measuring the functions α_2 and α_3 in dilute solutions, particularly those in which the magnitude of even the primary normal stress difference $\tau_{11} - \tau_{22}$ is too small for determination by viscometric methods. Simply, stability measurements should be made on a liquid for several values of R/δ, representing a range of shear rates. Regression methods can then be used to determine the functions α_2 and α_3 which produce the measured critical Taylor numbers according to the stability theory. Agreement with viscometric measurements of the same quantities could substantiate the theory and justify its use for determining rheological properties of dilute polymer solutions.

EXPERIMENTAL STUDY

An experimental study of the rotational stability of dilute solutions of six polymers was carried out between concentric cylinders. The outer cylinder inner radius $(R + \delta)$ was fixed at 5.905 cm., while three inner cylinders of outer radii (R) 5.631, 5.700 and 5.777 were used, referred to in the remainder of the paper as $R/\delta = 20.5$, 28, and 45, respectively. All cylinders were 4.1 cm. high. In order to minimize end effects each inner cylinder was machined at the base to a cone of base angle $\delta/(R + \delta)$ so that the shear rate in the conical gap at the bottom was identical to the shear rate in the annular gap. Constant physical properties for the test solutions were thus assured throughout the instrument, and con-

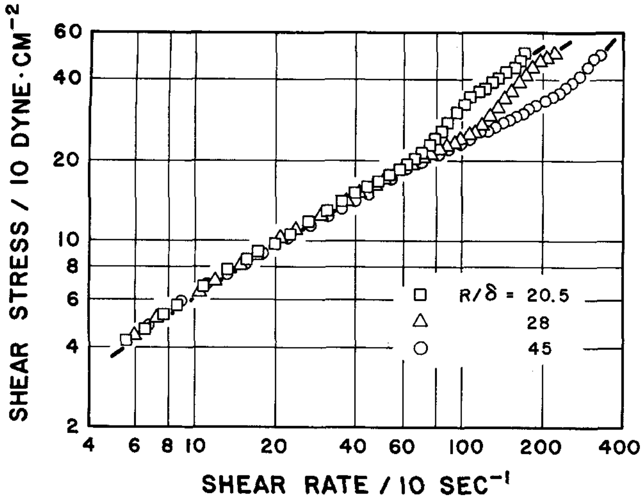

Fig. 1. Shear stress-shear rate relations showing onset of instabilities, 0.50% Natrosol.

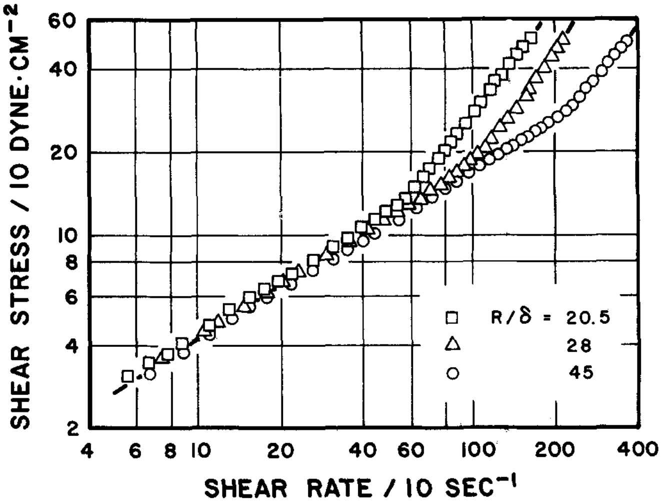

Fig. 3. Shear stress-shear rate relations showing onset of instabilities, 0.70% Polyox.

ventional Couette viscometer relations (7) could be used to relate shear stress to shear rate.

The cylinders were mounted on a commercial Ferranti-Shirley viscometer, the inner cylinder replacing the cone on the drive shaft and the outer cylinder centered stationary over the plate. In this way, perfect centering was assured and the constant speed drive and torque measuring capabilities of the commercial instrument could be used. A calibration of the torque scale was carried out for each inner cylinder using Newtonian glycerine-water solutions and National Bureau of Standards oils of known viscosity. The details of calibration and experimental procedure for obtaining shear stress-shear rate curves from the modified viscometer are discussed elsewhere (*17*).

Limitations of the modified viscometer restricted the experimental investigation to liquids demonstrating instabilities at shear rates below 4,000 sec.$^{-1}$ and shear stresses below 600 dynes/sq.cm. Meaningful data in all three sets of cylinders were obtained for a total of ten solutions of six polymers, aqueous solutions of Polyox (Union Carbide polyethylene oxide WSR301), 0.70% by weight; ET597 (Dow partially hydrolyzed polyacrylamide), 0.05, 0.10, 0.25, 0.35, 0.45, and 0.60%; Natrosol (Hercules hydroxyethylcellulose 250H), 0.50%; CMC (Hercules sodium carboxymethylcellulose type 7H), 0.25%; and one organic solution, polyisobutylene (Enjay Vistanex L-100) in decalin (decahydronapthalene), 0.50%. Details of sample preparation are contained elsewhere (*17*).

Figures 1 through 4 show typical shear stress-shear rate data for four of the solutions, 0.50% Natrosol, 0.45% ET597, 0.70% Polyox, and 0.50% polyisobutylene in decalin. At low rates of shear the data for all three values of R/δ lie upon a single line, from which the non-Newtonian viscosity function α_1 can be calculated. At a critical value of the shear rate for each value of R/δ the shear stress curve shows an abrupt increase in slope, indicating the onset of a secondary motion and increased shear stress. In this way, both the rotational speed and viscosity are determined at the onset of instability and the critical value of the Taylor number is computed from Equation (1). The uncertainty in establishing the precise value of the shear rate at which instability occurs and an analysis of the experimental errors associated with viscosity measurements indicate that the uncertainty in establishing the critical Taylor number is of the order of 10% (*17*).

The data are summarized in Table 1. The values of the Taylor number shown for Newtonian liquids represent in each case the average of five or six measurements, with standard deviations ranging between 5.3 and 5.8%. The average values of 3,450 and 3,400 for the two smaller gaps agree quite well with the theoretical value of 3,390, while the critical Taylor number in the widest gap is nearly 10% too low. This is a consequence either of unaccounted for end effects or the breakdown of the small gap ($R/\delta \rightarrow \infty$) approximation. The

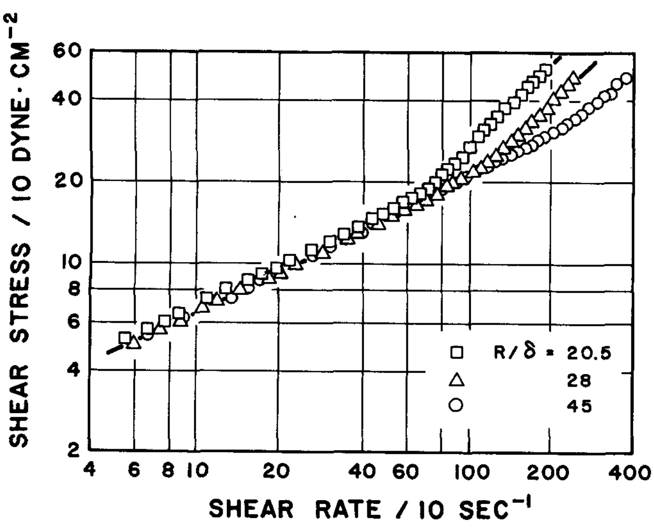

Fig. 2. Shear stress-shear rate relations showing onset of instabilities, 0.45% ET597.

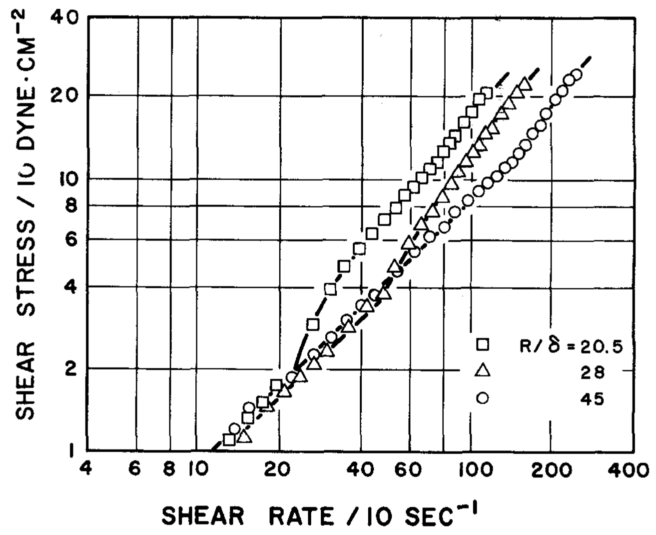

Fig. 4. Shear stress-shear rate relations showing onset of instabilities, 0.50% Polyisobutylene in decalin.

TABLE 1. CRITICAL TAYLOR NUMBERS

Solution		Critical Taylor Number R/δ = 20.5	 R/δ = 28	 R/δ = 45
Newtonian (avg.)		3,130	3,450	3,400
Polyox	0.10%			3,510
	0.15%			3,160
	0.70%	3,540	4,870	5,270
ET597	0.05%	4,000	3,270	6,550
	0.10%	3,510	4,250	6,590
	0.25%	3,230	3,280	4,790
	0.35%	3,180	4,130	4,330
	0.45%	3,720	4,260	5,030
	0.60%	3,300	2,900	3,450
Natrosol	0.50%	2,940	3,250	3,900
CMC	0.25%	3,020	3,940	3,680
Polyisobutylene	0.50%	2,770	3,510	4,940

general trend among the polymer solutions is to flow stabilization, particularly in the smallest gap. In many cases, however, little difference can be noted from Newtonian values, and there is some evidence of destabilization, particularly for polyisobutylene in $R/\delta = 20.5$.

According to the Ginn and Denn theory, the stability behavior found here is possible only when $\alpha_2 + 2\alpha_3$ and α_3 have the same algebraic sign ($\tau_{22} - \tau_{33} < 0$). This point is discussed further. The data for 0.10 and 0.15% Polyox in $R/\delta = 45$ are included for comparison with the study of Rubin and Elata (*18*). These authors reported visual observation of the onset of instability for the same material at Taylor numbers exceeding 6000 in an instrument of dimensions comparable to those in this study for $R/\delta = 20.5$. The discrepancy may partially reflect different methods of sample preparation.

VISCOMETRIC FUNCTIONS

The primary normal stress function $\tau_{11} - \tau_{22}$ was measured for all solutions for which three critical Taylor numbers were available except the polyisobutylene in decalin, where even the primary normal stress difference is too small to be determined by conventional methods. The measurements were made using the total normal force technique on a Weissenberg Rheogoniometer in cone and plate configuration. The experimental technique used for the University of Delaware rheogoniometer has been recorded previously (*9*, *20*). For all liquids the value of P_1 at the onset of instability was less than 0.04, in which case the critical Taylor numbers recorded in Table 1 and the Ginn and Denn stability theory (*11*) would require that P_2R/δ lie in the range $0 \geqq P_2R/\delta \geqq -0.02$. Measurement of values of P_2 many times larger in magnitude than those required here is exceedingly difficult, so that, as anticipated, the stability theory cannot be verified in this direct way.

The inverse procedure described previously was adopted, in which the stability measurements alone are used to predict the viscometric normal stress functions. Since only three data points were available for each solution, a maximum of three parameters could be specified. Thus, in accordance with most rheogoniometric determinations, the primary normal stress function was assumed to have a power-law dependence on shear rate, or

$$-\alpha_3 = a\,\Gamma^b \tag{6}$$

With only a single remaining free parameter, it was necessary to assume the same power dependence for the secondary normal stress difference, so that

$$\alpha_2 + 2\alpha_3 = 2\lambda a\Gamma^b \tag{7}$$

Here, λ, the ratio of secondary to primary normal stress differences,

$$\lambda = \frac{\tau_{22} - \tau_{33}}{\tau_{11} - \tau_{22}} \tag{8}$$

is assumed to be a constant over the shear rate range of interest. Though these are severely restrictive assumptions, behavior of the type described by Equations (7) and (8) has been reported for some liquids (*10*, *12*, *13*). The three parameters a, b, and λ were then chosen such that the Taylor numbers predicted by the stability theory corresponded to the experimental Taylor numbers.

Based on the deviation between theory and experiment in the largest gap, all Taylor numbers for $R/\delta = 20.5$ were increased by 10% prior to computation. The parameters were chosen by trial and error to simultaneously minimize the maximum deviation between theory and experiment and keep the average deviation for a given solution as close to zero as possible. The rheogoniometer data were used to obtain first estimates of the parameters and may introduce a limited amount of bias into the results, for ranges of parameter values were not sought. For all solutions but 0.05, 0.25, and 0.60% ET597 parameters were found which gave a satisfactory fit to the experimental Taylor numbers in that the average difference between the twenty-one experimental Taylor numbers and those calculated from the chosen values of a, b, and λ was 1.1% and the standard deviation 5.5%. As this is approximately the standard deviation found for the measurement of the Taylor number from the Newtonian data, there is no statistical difference between the experimentally observed and computed Taylor numbers. The inability to carry out such a calculation for the three remaining solutions indicates either at least one very bad data point or an inappropriate assumption in Equation (7).

Figures 5 through 7 show typical values of the func-

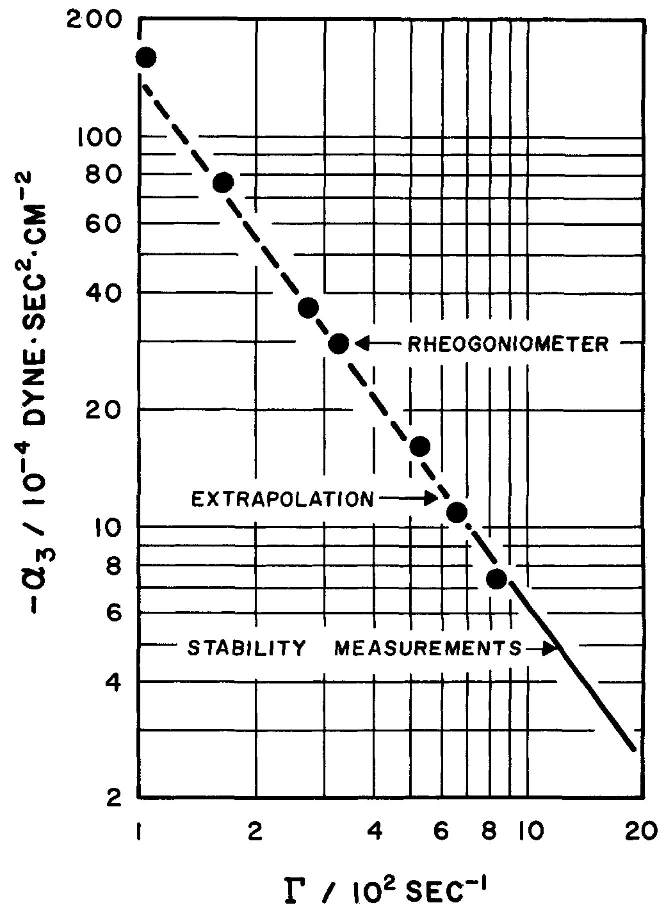

Fig. 5. α_3 vs. shear rate from stability measurements and rheogoniometer, 0.50% Natrosol.

tion α_3 computed from the stability data for solutions of 0.50% Natrosol, 0.45% ET597, and 0.70% Polyox, respectively. The solid line represents Equation (6) over the range of the experimental stability measurements and the dashed line the extrapolation. The data shown are the independent rheogoniometer measurements of α_3, which are the average of results from clockwise and counterclockwise shearing, and the agreement is excellent. The 0.50% polyisobutylene in decalin has a value of α_1 which is nearly independent of shear rate, in which case it is consistent with the simple fluid theory (*3*) that b be equal to two, which is the result obtained from the stability calculations. Thus, with only three exceptions, the limited data from the stability experiment are sufficient to predict the function α_3 with considerable accuracy, a result which is suggestive of the validity of the stability theory of Ginn and Denn (*11*) and holds promise as a potential measuring technique of the rheological properties of dilute polymer solutions.

The values of λ computed for the seven solutions from the stability data are shown in Table 2. All are negative and none exceeds 0.05 in magnitude. The stability theory provides the only technique by which both normal stress functions may be obtained simultaneously, by measurements of comparable magnitude, and on a single instrument. The excellent results obtained for α_3, then, provide strong supporting evidence for the validity of these values of λ, particularly in view of the sensitivity of the critical Taylor number to the viscometric function P_2. However, for the Polyox and Natrosol solutions studied here, Huppler (*12*) has measured values of λ which are a factor of ten or more greater in magnitude and of positive algebraic sign by determining the radial pressure drop in axial flow between concentric cylinders. The experiment is a difficult one, but errors of the required magnitude would be surprising, though Broadbent, et al. (*1*) have recently suggested a source of large error in pressure tap measurements of normal stresses. The two results must stand as contradictory and in need of resolution, but we do note again that for positive values of λ the stability theory admits only destabilization.

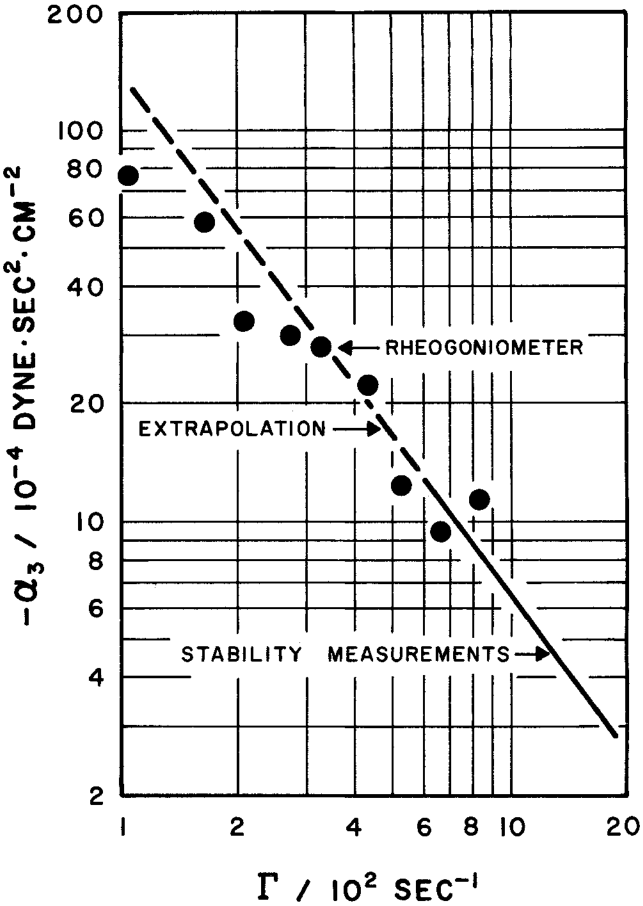

Fig. 7. α_3 **vs. shear rate from stability measurements and rheogoniometer, 0.70% Polyox.**

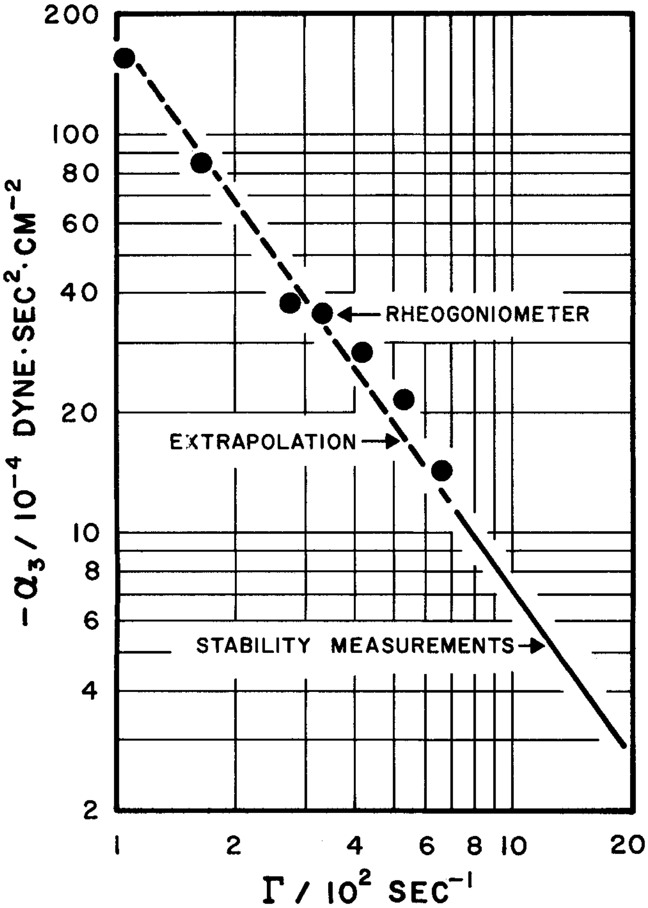

Fig. 6. α_3 **vs. shear rate from stability measurements and rheogoniometer, 0.45% ET597.**

TABLE 2. RATIO OF NORMAL STRESS FUNCTIONS FROM STABILITY MEASUREMENTS

Solution		$\lambda = \frac{\tau_{22} - \tau_{33}}{\tau_{11} - \tau_{22}}$
Polyox	0.70%	—0.028
ET597	0.10%	—0.048
	0.35%	—0.018
	0.45%	—0.025
Natrosol	0.50%	—0.014
CMC	0.25%	—0.023
Polyisobutylene	0.50%	—0.010

While there is further evidence of positive λ in the data of Markovitz and Brown (*14*) for solutions of polyisobutylene in cetane, which might be expected to have properties similar to polyisobutylene in decalin, Ginn (*10*) has recently obtained rheogoniometer data in cone-and-plate and parallel plate configurations of both normal stress functions for three concentrated solutions of polyisobutylene in decalin in which he has found λ to be negative. Furthermore, the magnitude decreases with concentration in such a way that the value obtained here for a dilute 0.50% solution lies on a continuous curve with his data for concentrated solutions. This is strong supporting evidence for the validity of the results reported here and their use as a measuring technique.

Negative λ has also been reported (*1, 13*) for a solution of high molecular weight polyisobutene in low molecular weight polyisobutene.

FRICTIONAL DRAG FROM SECONDARY FLOWS

The secondary flow remains a stable cellular motion for a range of shear rates beyond the critical Taylor number. For Newtonian liquids the strength of the secondary flow and the additional frictional drag which it generates have been analyzed (*4, 6, 22*), with the results shown in Figure 8. Following Stuart (*22*), the total torque is denoted by G with the theory presented as a friction factor-Reynolds number plot. Scale factors are used to normalize all data with respect to conditions at the onset of the secondary flow. The straight line is the undisturbed viscometric flow and the bow-shaped line the values corresponding to the superposition of circular and cellular flows. The data shown for a Newtonian 60% Glycerine-water solution are typical of the agreement between theory and experiment in both this and previous studies. Significant deviations begin to occur only after a rotational speed more than twice that of the onset of instability, following which the theory underestimates the actual frictional drag.

All of the dilute polymer solutions showed substantial deviations from the curve for Newtonian liquids, but in general, a meaningful interpretation is difficult because of ambiguity in computing the effective viscosity, which is affected by the secondary motion. Such is not the case, however, with the 0.50% polyisobutylene in decalin, which has nearly a constant viscosity, and the three sets of data for this solution are plotted in Figure 8. For $R/\delta = 20.5$, where the solution was less stable than a Newtonian liquid, the frictional drag associated with the secondary motion exceeds the Newtonian value. The usefulness of the data for this gap size, however, is doubtful. On the other hand, for $R/\delta = 28$, where the stability characteristics did not differ significantly from the Newtonian, the secondary flow data follow the general trend of the Newtonian data, while for $R/\delta = 45$, where the stability was greatly enhanced, the subsequent secondary motion generates an additional frictional drag which is only one-third that for a Newtonian liquid.

The drag reduction observed in this time-independent laminar secondary flow is highly reminiscent of the reduced frictional drag in the turbulent pipeline flow of dilute polymer solutions (*16, 19*) and might be expected to be associated with the same damping mechanism. In Couette flow the stability theory indicates that the significant factor is the secondary normal stress difference which, though extremely small, enters with a geometric factor of R/δ, which can become large. Similar large geometric ratios exist between pipe diameter and characteristic eddy sizes in turbulent flow.

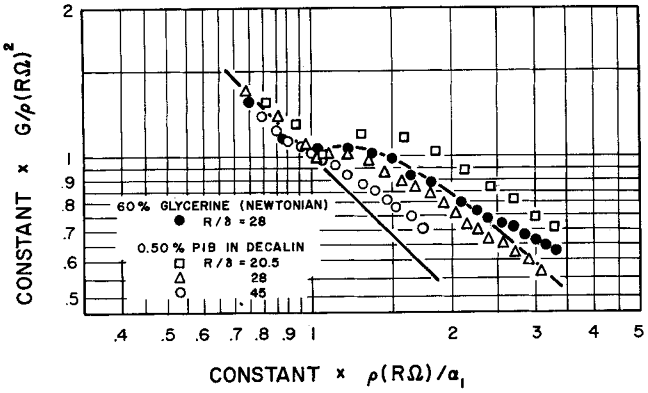

Fig. 8. Torque generated by secondary flow, 60% Glycerine (Newtonian) and 0.50% Polyisobutylene in decalin.

ACKNOWLEDGMENT

This study was part of a program of investigating unsteady Lagrangian flows of viscoelastic liquids supported by the National Science Foundation under grant no. GK 896.

NOTATION

a = parameter in power relation for α_3
$\mathbf{A}_n$ = nth Rivlin-Ericksen tensor
b = exponent in power relation for α_3
G = total torque
$\mathbf{I}$ = identity tensor
p = hydrostatic pressure
P_1, P_2 = reduced viscometric functions
R = radius of inner cylinder
T = Taylor number

Greek Letters

$\alpha_1, \alpha_2, \alpha_3$ = viscometric functions
Γ = shear rate
δ = gap width
λ = ratio of normal stress differences
ρ = density
$\boldsymbol{\tau}$ = stress
Ω = angular velocity of inner cylinder

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