06673-7-P

THE UNIVERSITY OF MICHIGAN

COLLEGE OF ENGINEERING
DEPARTMENT OF CHEMICAL AND METALLURGICAL ENGINEERING

Semi-Annual Status Report

for the period 1 December 1966 to 31 May 1967

Submitted to:

National Aeronautics and Space Administration
Washington, D. C.
Fluid Physics Program in NonNewtonian Fluid Mechanics

A Rheological Study of Glass Fibers in a Newtonian Oil

N67-30073
(ACCESSION NUMBER)
44
(PAGES)
CR-85257
(NASA CR OR TMX OR AD NUMBER)
(THRU)
1
(CODE)
12
(CATEGORY)
FACILITY FORM 602

Submitted by:
J. D. GODDARD, Project Director
LEO F. CARTER

The work described in this report was performed under NASA Grant No. NsG-659 during the period 1 June 1965 to 31 May 1967

Administered through: *June 1967*

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Abstract

Results are presented of some rheological tests on suspensions of 3.5 micron-diameter glass micro fibers in a 180 poise Newtonian oil.

Thirteen monodisperse suspensions, with three distinct ratios of length to diameter, 57, 114, and 228, with fiber volume fractions in the range .00511 to 0.02200, were studied.

Shear-stress and total-normal-thrust measurements were made, over a range of steady shear rates from 0.01 to 350 sec^{-1}, with a cone-and-plate rheometer.

With the exception of the longest fibers, only slight shear thinning was observed. From the total normal thrust studies it is found that the first normal stress difference is approximately linear in the shear rate and is on the order of one-fourth or more the magnitude of the shear stress. Unsteady shear tests failed to reveal any evidence of elastic behavior in the suspensions.

Anomalous behavior, with separation during testing was observed with the highest concentrations of the longer particles.

Certain parameters, obtained from the theory of dilute suspensions, are proposed tentatively for correlating rheological properties with fiber length and concentration. Owing to the large ratio of particle-to-instrument dimensions in these studies, there is some doubt as to the validity of a continuum interpretation of the suspension behavior, although some independent tests tend to support the viscosity data.

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I. Introduction

The following is a report on part of the research on non-Newtonian fluid mechanics which is being carried out at the University of Michigan, under Grant NsG659 from the National Aeronautics and Space Administration. Broadly speaking, the research program is concerned with the rheology of disperse, or microscopically heterogeneous, fluid systems. More specifically, the study reported here represents an attempt to relate the macroscopic or bulk rheological properties of certain particle suspensions to their microstructure.

It is a well-established fact that most fluid-like suspensions of particulate solids in ordinary (Newtonian) liquids exhibit the interesting and complex flow behavior referred to generally as non-Newtonian. Because of their natural and technological importance, materials of this type have been the subject of many experimental studies. They have also received a good deal of theoretical study, since analyses of certain idealized particle suspensions provide a conceptual basis for understanding non-Newtonian effects in more general fluids.

Recent refinements in both experimental and theoretical methods have greatly facilitated the investigation and classification of complex fluid behavior at the continuum level. As with other types of materials, one of the primary problems at present is to correlate the continuum or macroscopic behavior of fluids with the details of their microstructure. The objective of the present study* was to investigate the rheological behavior of well-defined, monodisperse fibers in a Newtonian liquid. Most of the past studies have dealt only with gross flow characteristics or with the viscosity of fiber suspensions (I). In view of their possible applications in areas such as lubrication and turbulent-drag reduction there is some practical interest in a more detailed understanding of thier rheology.

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* The report is based mainly on results from the Ph.D. dissertation of Leo F. Carter, "A Study of the Rheology of Rod-Shaped Particles in a Navier-Stokes Liquid," University of Michigan, 1967, hereafter referred to by the Roman numeral I. The other reference numbers appearing as superscripts refer to the Bibliography here, which is reproduced from the above work. Figure numbers correspond to those of this work.

II. Description of Suspensions and Experimental Apparatus.

A. Description of the Samples and Their Preparations

The materials tested in this study consist of microscopic glass rods suspended in a viscous low molecular weight (MW$\sim$700) polybutene oil (Amoco "INDOPOL H-100"). At the test temperature of 25°C the measured viscosity of the pure liquid is 186 poises. The glass rods have been cut from continuous strands of type E glass fibers provided by The Owens Corning Fiberglas Company. Average fiber diameter was 3.5 microns (μ).

In order to prepare samples it is found most convenient to cut large quantities of fibers into prescribed lengths (200, 400, 800 μ) using a sliding microtome.* This operation is facilitated by imbedding large numbers of parallel strands of glass thread in wax. This is accomplished by constructing a frame on which fibers can be wound, and which can then be employed as part of a mold to receive the wax.

The wax-fiber plug made in this way is then placed in the microtome, and rodlets of the desired lengths are produced. These rodlets, however, are still surrounded by paraffin, and a preliminary separation is accomplished by heating the wax-glass mixture in distilled water. The glass settles readily to the bottom of the container while the wax remains on the surface of the water. The wax solidifies, is removed, and the

* The method of sample preparation used here is similar to that described by Nawab and Mason.[35]

collected fibers are then dewaxed in a Soxhlet extractor using a xylene solvent. After rinsing with acetone and distilled water, the samples are dried and viewed under a microscope. Essentially uncontaminated fibers were produced by this method, and a very narrow distribution of lengths results. (More than 90% of the fibers are within 5% of the desired length.)

After sufficient fibers of a given length have been collected, various quantities of fibers are placed in test tubes of known weight, and the weight of fibers in each test tube determined. The viscous liquid is then added to the fibers and the total weight determined. The densities of both materials are known so that the volume fraction of fibers is readily calculated. Independent measurements of the densities of the glass fibers and the liquid agree with the manufacturers values to within $\pm$ .4%. The specific density of the fibers is 2.54 while that of the liquid is 0.881. Gentle stirring disperses the particles readily, producing apparent homogeneity of the suspensions. It is found to be most efficient to stir samples thoroughly about one day in advance of use and then gently before using. Trapping of air bubbles can be minimized with care. Visible trapped bubbles can be removed after the sample has been loaded onto the cone and plate. It has been observed that visible settling requires more than two days. Twelve fiber suspensions have been prepared and tested and their composition and axis ratios are listed in the following table.

SAMPLE NO.	AXIS RATIO, r	VOLUME FRACTION FIBERS, c
1	228	0.01081
2	228	0.02178
3	228	0.00580
4	228	0.01650
5	114	0.01870
6	114	0.01581
7	114	0.01073
8	114	0.00564
9	57	0.02200
10	57	0.01303
11	57	0.00931
12	57	0.00511
00(Pure suspending fluid)	--	0

Table 1 Suspension Properties

All samples have been used five times or less with about four grams of material used each time. Samples have been reused since only small quantities of sample ($\sim$15g.)have been made. Microscopic study, prior to and after testing, has revealed no evidence of significant particle destruction or contamination during testing.

B. Summary of Experimental Apparatus and Measurement

The experimental apparatus for this study consisted mainly of the Weissenberg rheogoniometer. Two different instruments were actually employed, one located at the University of Michigan and the other at the Dow Chemical Company in Midland, Michigan.* Both these were of the commerical variety (manufactured by Farol Research Engineers, Ltd., and sold in the United States by the Martin-Sweets Company), the "R-16 Weissenber Rheogoniometer." A more detailed discussion of the instrumentation and the ancillary equipment for recording of data and temperature control is to be found in Reference I.

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*Whose contribution is gratefully acknowledged.

In the initial phase of each measurement sample temperatures were controlled to 25°C $\pm$ 0.2°C (In the total normal-force measurements the room temperature was maintained at 24.4°C $\pm$ .56°C.).

The following is a brief summary of the tests performed.

1. Shear-stress tests

These tests were all done in a 7.5 cm diameter cone-and-plate attachment to the rheogoniometer. The angle of the cone is 0.02822 radians and its tip truncation is 0.00250 inches. The calibration of all torsion bars employed was re-established.

Steady-state shear tests were run on all samples in the shear-rate range 0.167 to 167 sec^{-1}. Also, a large number of combined steady-shear and periodic oscillatory-shear tests were performed. Finally, some stress "build-up" experiments, with an imposed step-function shear-rate, were done. An ultraviolet strip-chart recorder was used for all the unsteady shear tests to record stress and deformation.

A more detailed description of experimental technique is to be found in Reference I, together with a tabulation of raw data.

2. Steady normal stress measurements

Preliminary observations of normal stresses were made by means of a standard plexiglas manometer-head attachement for the rheogoniometer.

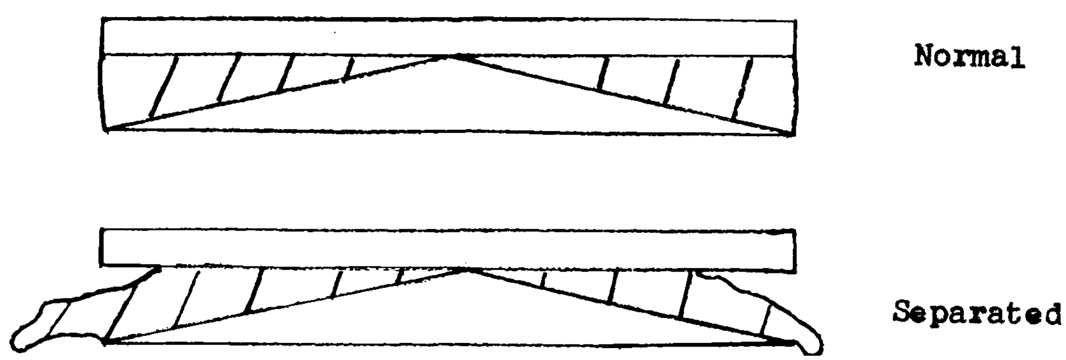
Quantative measurements of total axial thrust in the cone-and-plate were later made (on the Dow instrument). In these tests a 7.5 cm diameter cone, with an angle of 0.0268 radians and a tip truncation of 0.00259 inches, was employed. Normal-force data were taken over the range of shear rates 11.12 to 352.2 sec^{-1}.

III. Results and Conclusions

A. Viscometric Data

1. Steady shear viscosity:

The steady shear viscosity data obtained in this work are presented in Figure IIID-1 through Figure IIID-3 as a function of shear rate γ with each plot presented for fixed axis ratio r. The concentrations (in volume fractions) are listed with the sample numbers and plotting symbols, and can be seen to range from pure material c = 0 to the most highly concentrated material c = 0.0220. It is apparent from these figures that shear thinning occurs, and is more pronounced for increased concentration and increased axis ratio. For the two larger axis ratios the higher concentration suspensions show extremely large viscosities for small shear rates as shown in Figures IIID-2 and IIID-3. This occurs simultaneously with the appearance of separation of the material from the upper plate. Effectively, the outer radius is decreased to some lesser value. The following sketch serves to illustrate this.



Sketch of separation effect.

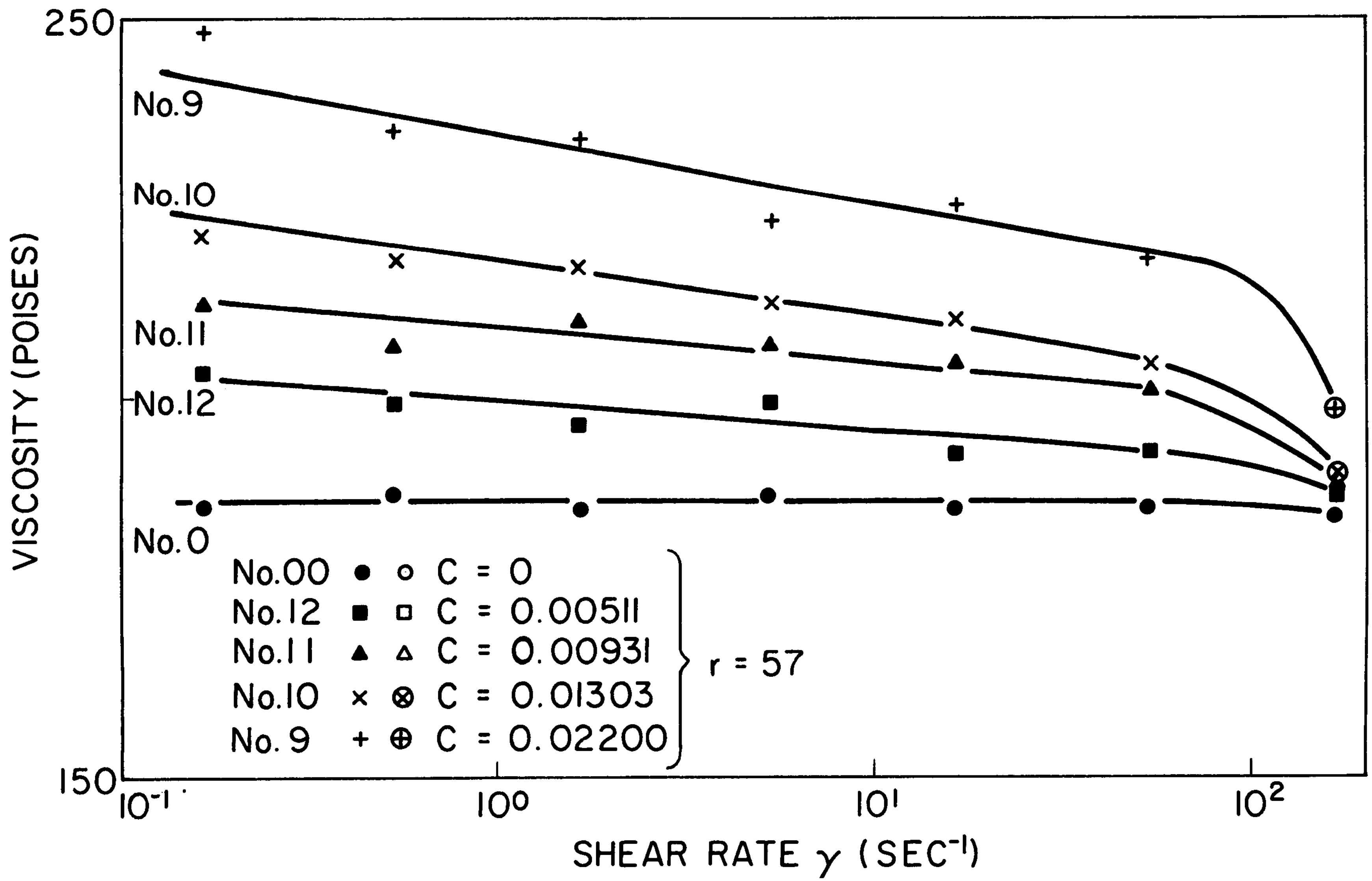


Figure IIID-1. Viscosity as a function of shear rate for various concentrations with r = 57.

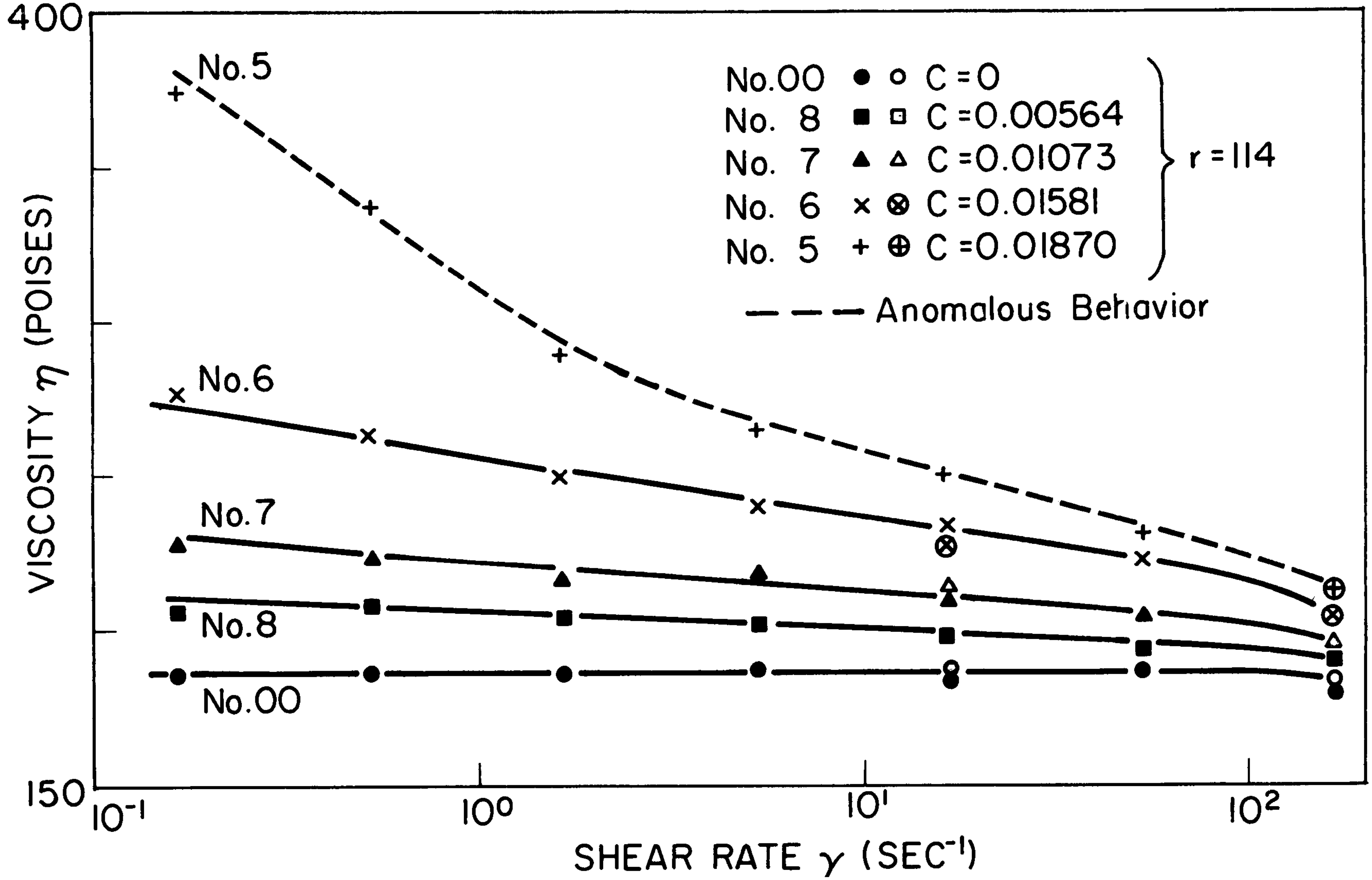


Figure IIID-2. Viscosity as a function of shear rate for various concentrations with r = 114.

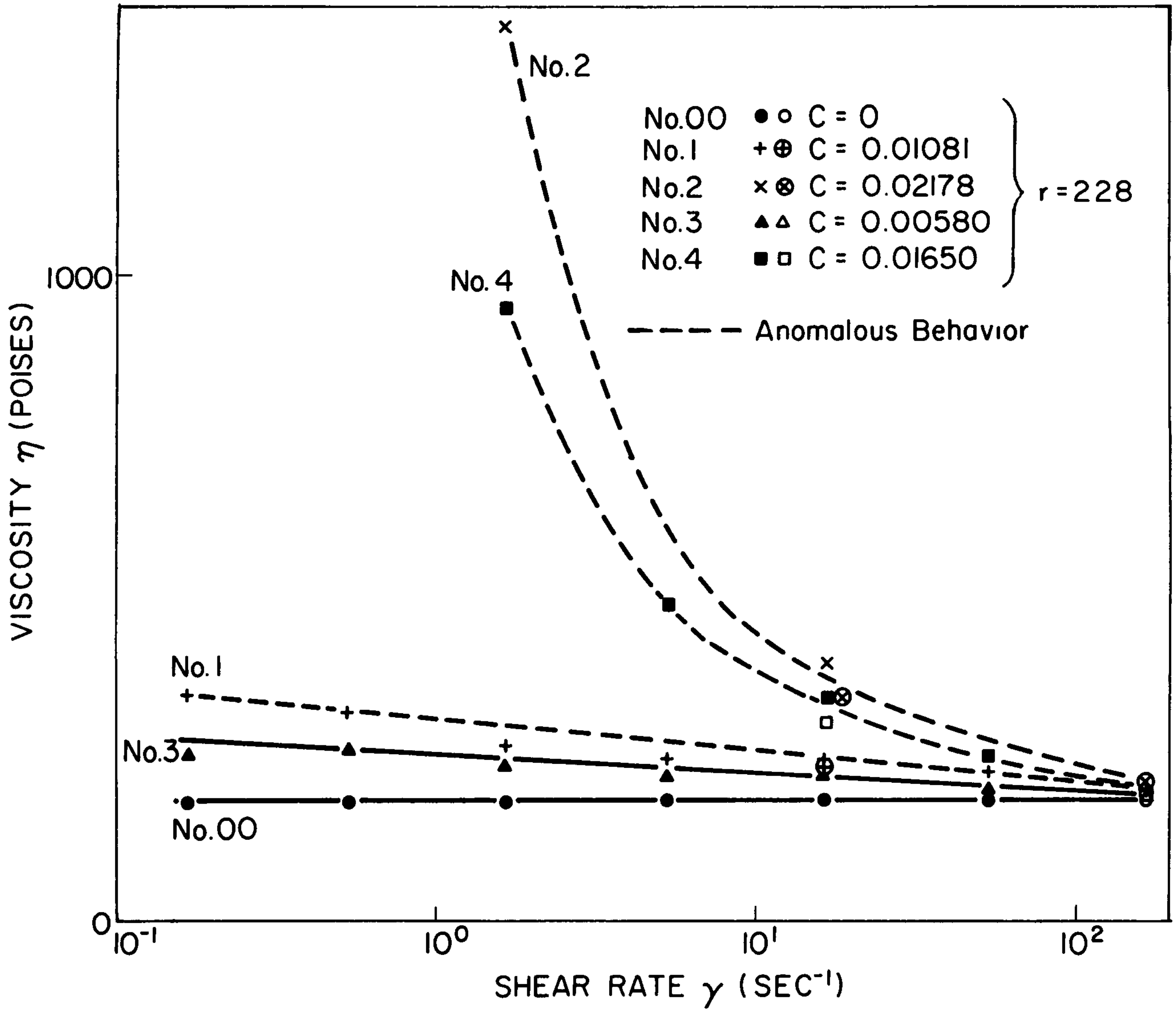


Figure IIID-3. Viscosity as a function of shear rate for various concentrations with r = 228.

The data that were taken when this occurred do not, of course, give correct viscosities; they are indicated with broken lines on the plots* merely to provide a minimum bound for the viscosity. It is believed that separation occurs because of the natural tendency of a group of fibers in these high concentration, high axis ratio cases (suspensions #5, 4, 2, 1 and possibly #6) to expand to a volume larger than that of the containing liquid. In so doing the expanding mat of fibers takes the liquid with it as it leaves the gap. Some further information supporting the idea that a fiber mat may tend to expand has been obtained while making the suspensions. It is observed that fibers dropped loosely into a test tube cannot be covered completely by the volume of liquid required to produce a desired concentration, and have to be compressed in order to reach the desired suspension concentrations for the above mentioned samples. Two other factors have been observed which shed light on this matter. The first is Morrision and Harper's[34] observation of yield stresses in concentrated suspensions of fibers and their comment that "clumping" or agglomeration was involved. In connection with this, the settling of fibers and rising of air bubbles in the present suspensions have shown that some of the more concentrated higher axis ratios suspensions (#'s 1, 2, 4) retain air bubbles indefinitely, and exhibit little or no visible tendency toward fiber settling. Evidently a stable net-

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* Broken lines or circled data points indicate regions or points where separation effects or viscous heating was apparent or suspected.

work (or entanglement) of fibers exists for these materials which can support the bouyant force exerted by an air bubble. This is essentially in agreement with the results of Morrison and Harper[34]. Moreover, phase separation and wall-slip have been mentioned by Nawab and Mason[35]. There is a possibility that separation effects may be dependent on the pure liquid viscosity. This effect has not been examined here. We are primarily interested in the data obtained for suspensions dilute enough such that separation and yield effects do not occur, and we find for the present system that we can define limits of concentration and axis ratio below which these effects are not observed.

The values of the viscosity decrease as the shear rate increases, and tend to drop off sharply near the highest shear rate (167 $sec.^{-1}$). The latter effect is attributable to two factors. The first is viscous heating, which has been found to cause temperature rises observable with the thermocouple attached to the upper platen for shear rates greater than 167 sec^{-1}. These increases in temperature lower the viscosity of the suspending liquid. A second factor is the possible occurrence of centrifugal effects which cause material to be thrown out of the gap at a shear rate of about 500 sec^{-1}. Most of the present data has been taken at shear rates well below this value, so that the net centrifugal force acting at the rim is negligible. Independent experimental determination of centrifugal effects in cone and plate flow[2] indicates that measureable normal stress effects begin to be observed for a shear rate of 350 $sec.^{-1}$. It is probable that the sharp decline of the viscosities at high shear rates is mainly a heating effect.

Observation of shear thinning for these materials does not correspond with the results of Nawab and Mason[35], but it must be noted that they worked in a narrow range of shear rates ($\sim$9 to 90 $sec.^{-1}$) which would make it difficult to observe shear thinning effects. By way of contrast the present data includes a four decade range of shear rate (0.167 to 167 $sec.^{-1}$). It is worth noting that within the range of shear rates reported by Nawab and Mason our results for the zero concentration intrinsic viscosity, $(\eta\ sp/c)_{c\ =\ 0}$, are in good agreement with theirs for our smallest axis ratio. They find experimental values of this variable of 8.2 and 15.2 for axis ratios of 43.3 and 75 respectively. Our experimental value is 9.3 for an axis ratio of 57. The liquid used in the above mentioned study was Caster Oil ($\eta \sim 25$ poise), and the fibers were rayon. This gives some assurance that the results of the present study are not dependent on the specific liquid or fibers chosen, but instead can be thought of as applying to any fiber suspension as long as axis ratio, concentration, and other properties are taken for the wetted material where swelling or surface reactions might be involved. Perhaps more importantly, this agreement of our results with those for the concentric cylinders experiment tends to rule out serious wall effects in our tests for an axis ratio of 57.

Figures IIID-5 and IIID-6 are parametric plots of the specific viscosity

$$\eta_{sp} = \frac{\eta - \eta_o}{\eta_o}$$

versus fiber volume fraction c and fiber length-to-diameter ratio r, for two shear rates of 1.67 and 16.7 $sec.^{-1}$ respectively. In Figures IIID-9 and IIID-10 the same data are replotted versus a single variable which is suggested by the theory of dilute suspensions of rods as developed by Jeffrey, Burgers, and others (I)[6,30]. One might use the slope of these plots to evaluate certain averages associated with the angular distribution of rods in dilute suspensions. However, most of the present suspensions are beyond the concentration range where the theory of dilute suspensions of non-interacting particles could be expected to apply. Thus, the main value of the theory here would seem to be its suggestion of a correlating parameter.

2. Normal stresses:

The first observation of a normal stress pattern made in this study has been accomplished using a cone and plate geometry with manometer tubes in the upper platen. The pure material is found not to rise in the tubes for any of the shear rates employed, although there is about a 0.8 cm rise in the center tube tapering off to about 0.4 cm at the outer tubes which remains for all shear rates (including zero shear rate). These values are rough estimates since the plastic platen is about

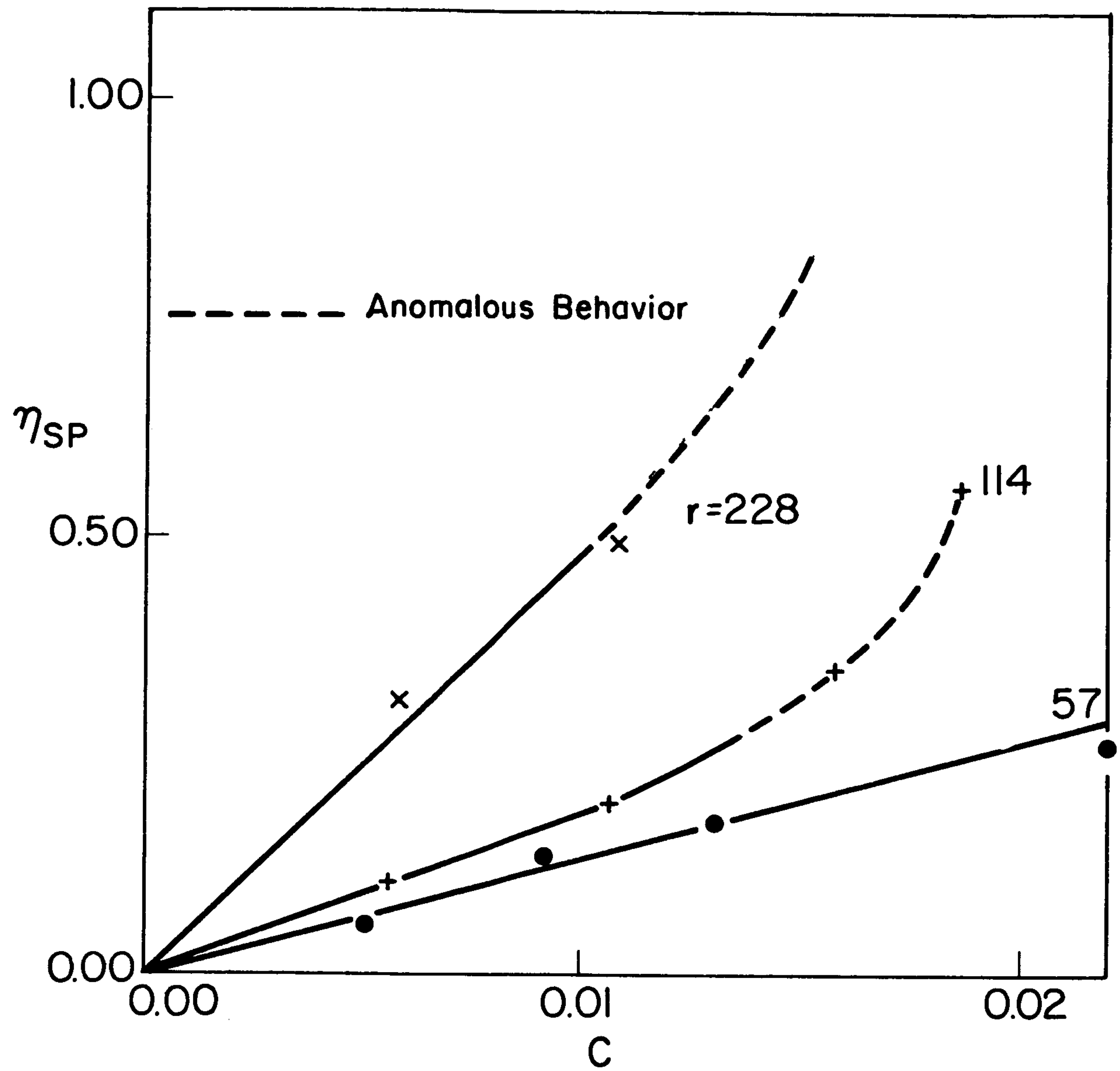


Figure IIID-5. Specific Viscosity as a function of concentration for three axis ratios for a shear rate of 1.67 sec^{-1}.

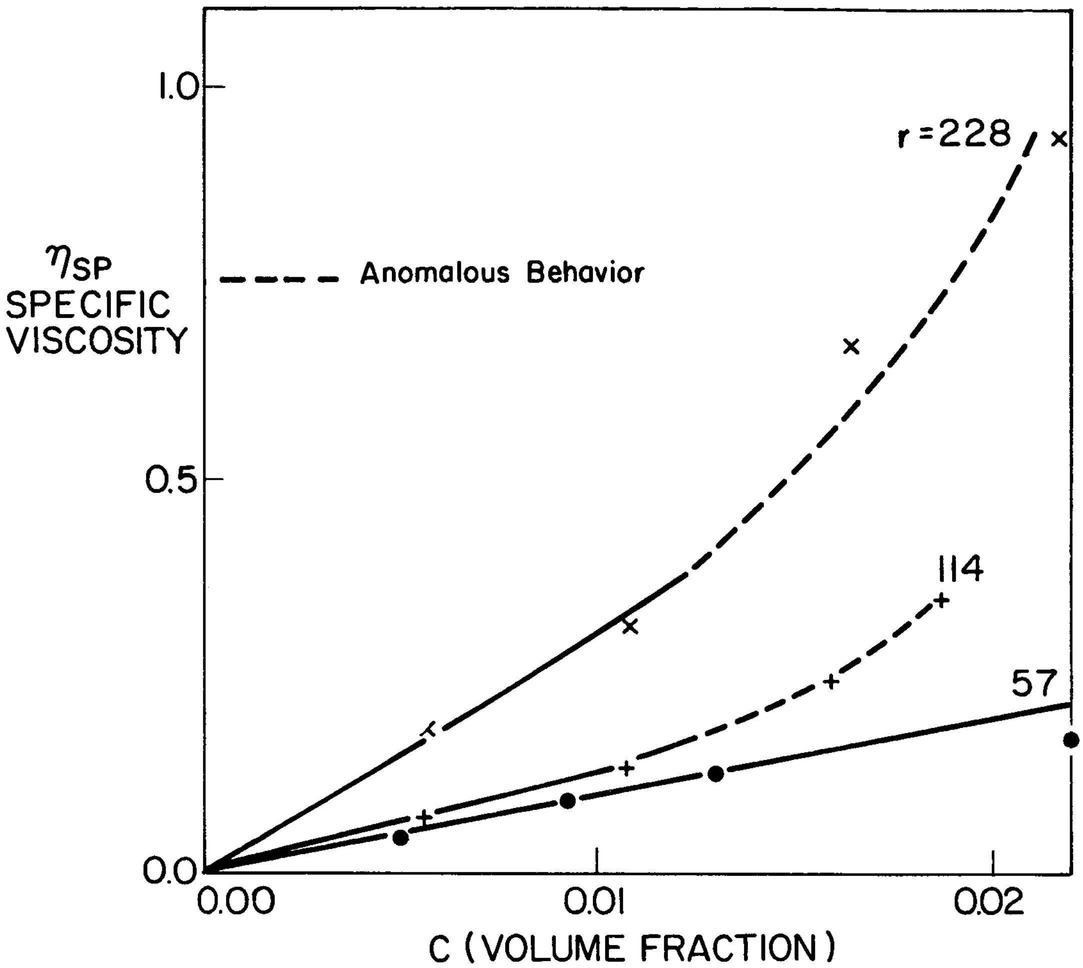


Figure IIID-6. Specific viscosity as a function of concentration for three axis ratios for a shear rate of 16.7 sec^{-1}.

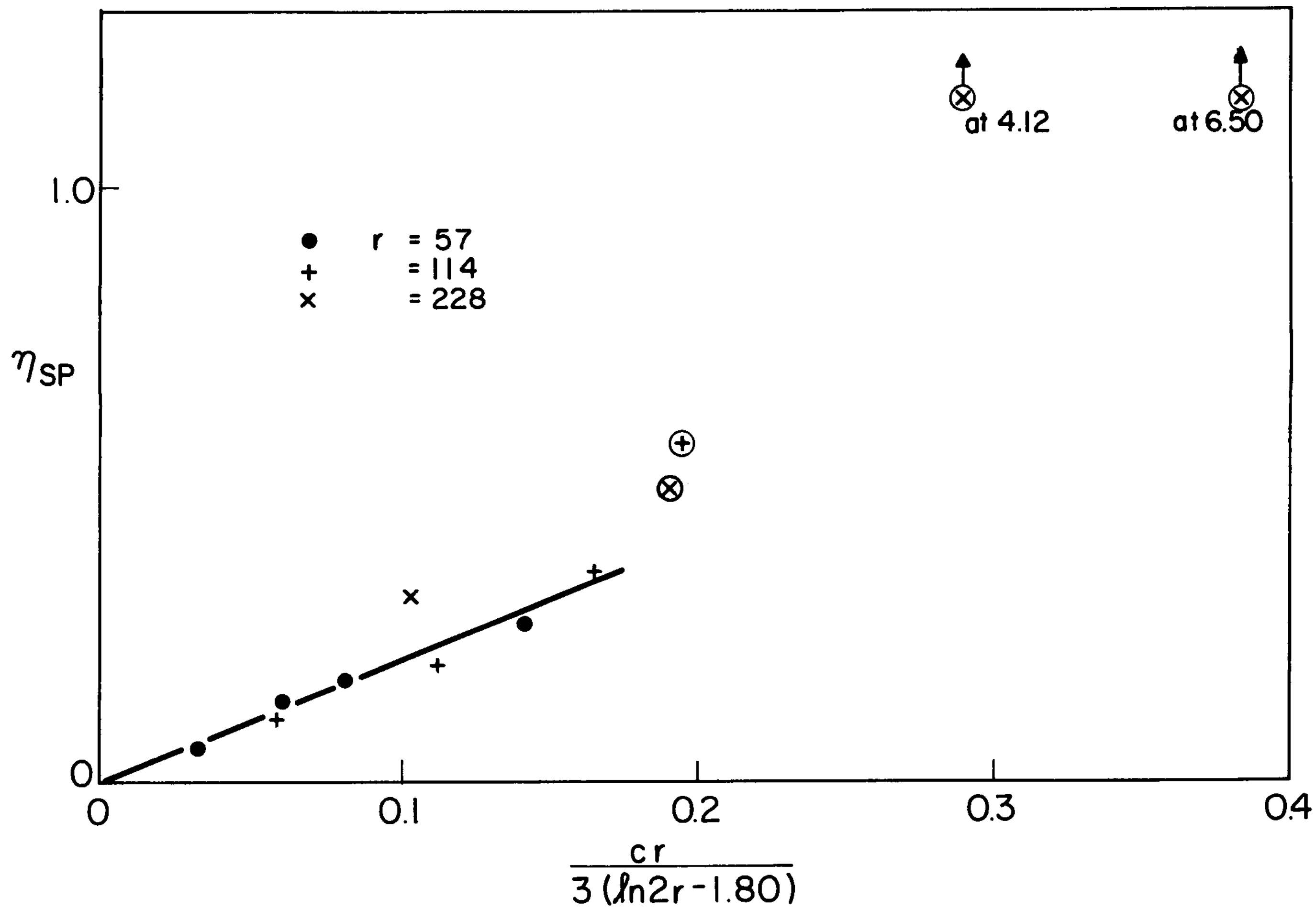


Figure IIID-9. Specific viscosity as a function of generalized concentration for a shear rate of 1.67 sec^{-1}.

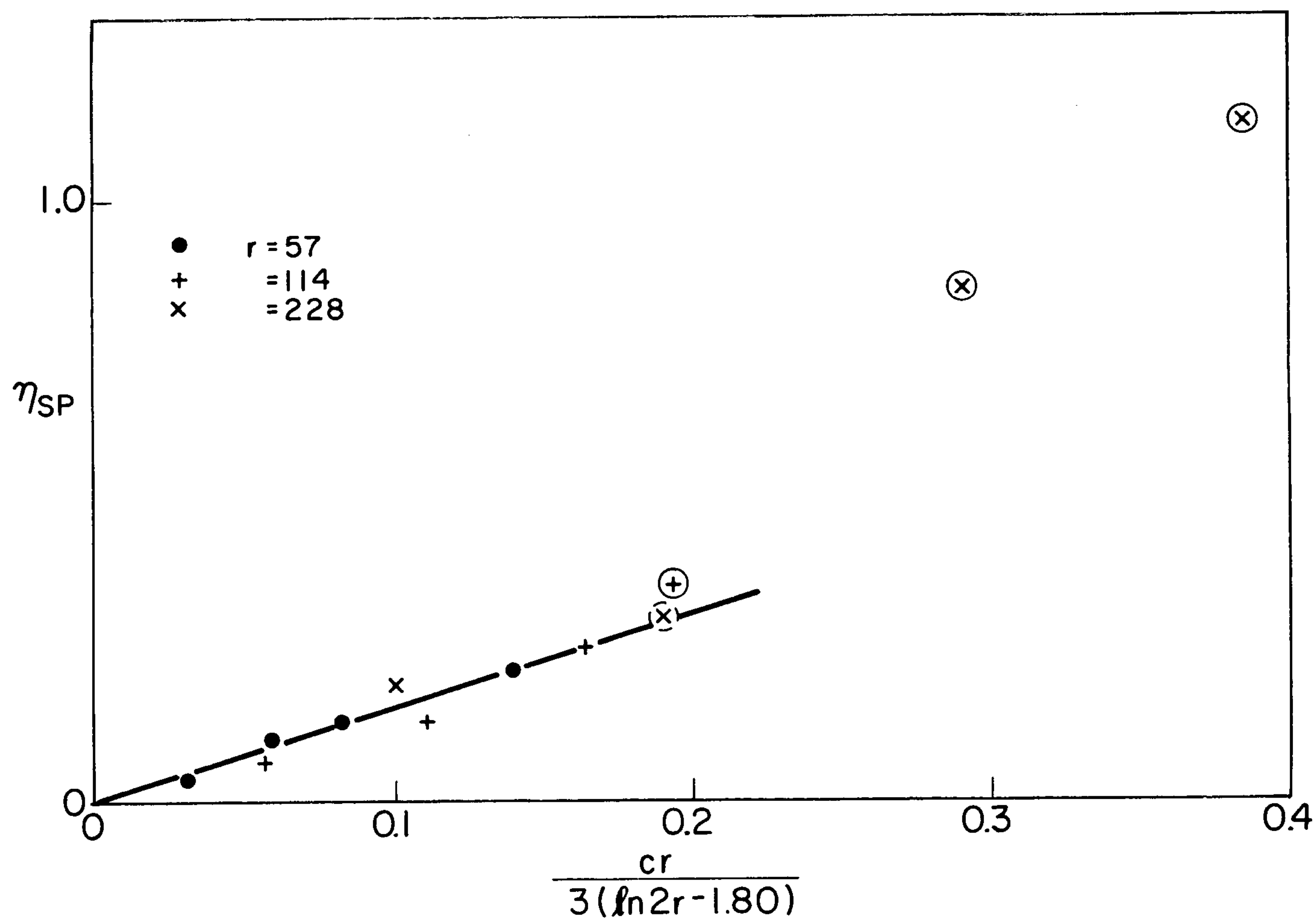


Figure IIID-10. Specific viscosity as a function of generalized concentration for a shear rate of 16.7 sec^{-1}.

about one centimeter thick. Data have been obtained by averaging the heights of the columns of liquid which are symmetrically placed about the central manometer tube. The values averaged differed by as much as 40% because of imperfect alignment. It has been found that larger intentional misalignment can give small averaged manometer readings for the pure material also. It was not possible to get positive values on both tubes to be averaged however with pure material. The results obtained here are therefore considered to be primarily of qualitative interest. In addition to demonstrating rather conclusively the presence of normal stresses in the suspensions studied, these data for which a typical sample is represented graphically in Figure IIID-11 indicate that a small non-zero normal stress may exist at the outer radius; or that the Roberts-Weissenberg stress pattern (see reference 19) does not hold. This agrees with the results of the stress optical study made by Philippoff[37] for suspensions of rigid anisotropic particles. It has been stated, however, by Markovitz[8] and Giesekus[38] that values obtained near the edge are not likely to be meaningful since the flow is no longer ideal. (Deviation from the Roberts-Weissenberg stress pattern would also be expected from our theoretical work as well as that of Hand[28] and Ericksen[15].)

A second point of interest which arises from this data is the extremely low value of the indicated stress at the center tube. To check these low central values a similar experiment has been run in which the gap has been widened by a

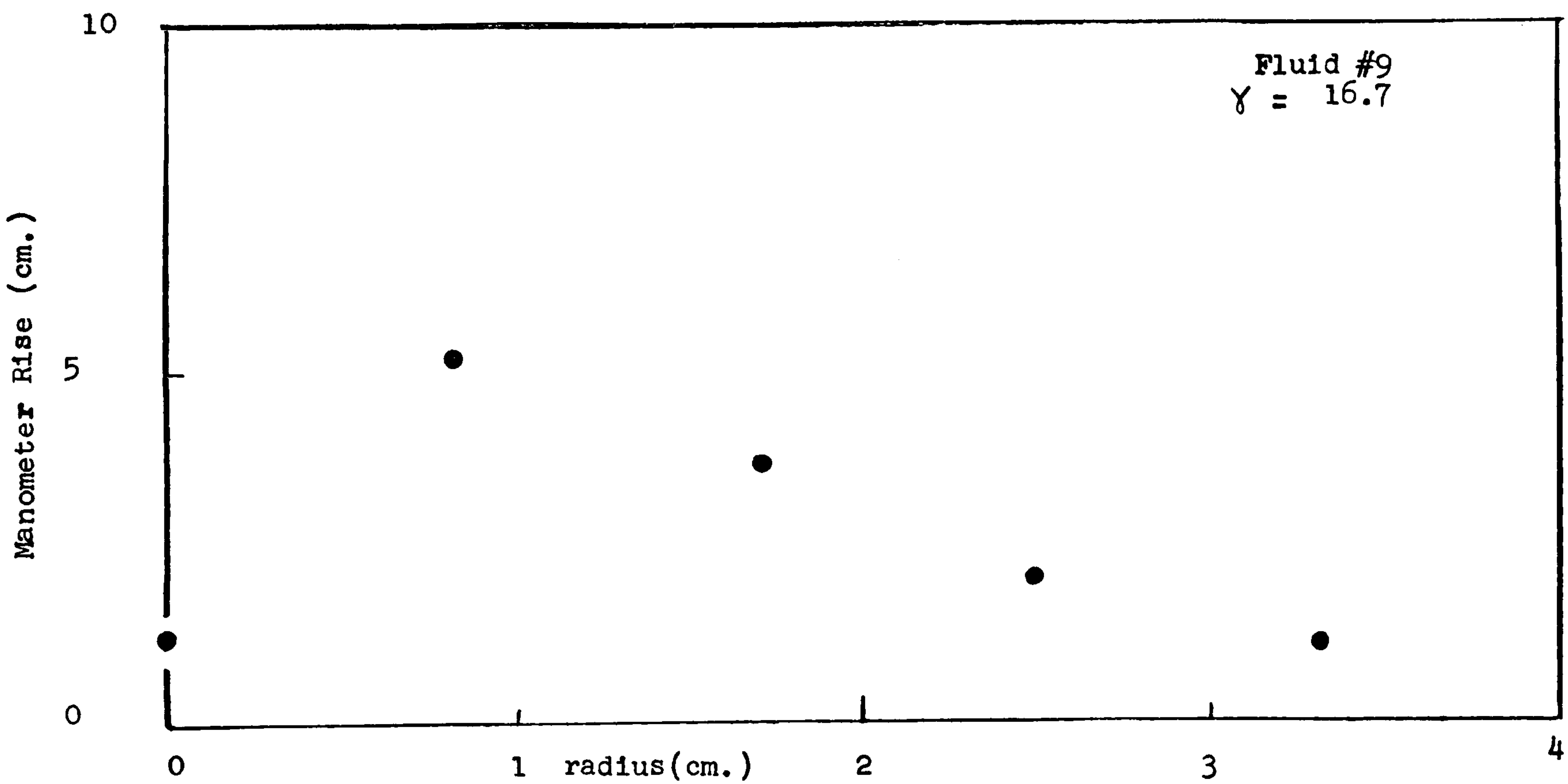


Figure IIID-11. Manometric rise as a function of radial position for sample number 9.

constant amount, and the shear adjusted such that the average value matches the corresponding cone and plate shear rate. The result is a significant rise in the center tube. The cone and plate experiment is then repeated, and the centerline manometer rise is found to remain effectively constant at the height attained during the flow with the widened gap. Thus, it appears that the response of the height of material in the central tube to the flow pattern and consequent stresses is too slow to obtain significant data for materials of this type. A logarithmic plot of the radial dependence of the normal stresses is presented in Figure IIID-12 for the same data plotted in Figure IIID-11. It appears that the logarithmic relationship predicted for simple shear flow is approximated. The value of the manometer rise h at the outer radius is estimated from Figure IIID-11. The line chosen in Figure IIID-12 has been required to pass through zero at the outer radius of the platen.

Manometer-head tests also reveal an increase in normal stress with concentration and axis ratio. This is not surprising in view of the already observed viscosity dependence of these materials on concentration and axis ratio. The shear rate dependence of the normal stresses will be discussed in the following paragraphs where total-normal-force data are presented and discussed for a range of shear rates.

Logarithmic plots of total normal-force as a function of shear rate appear in Figures IIID-13 through IIID-15 in which

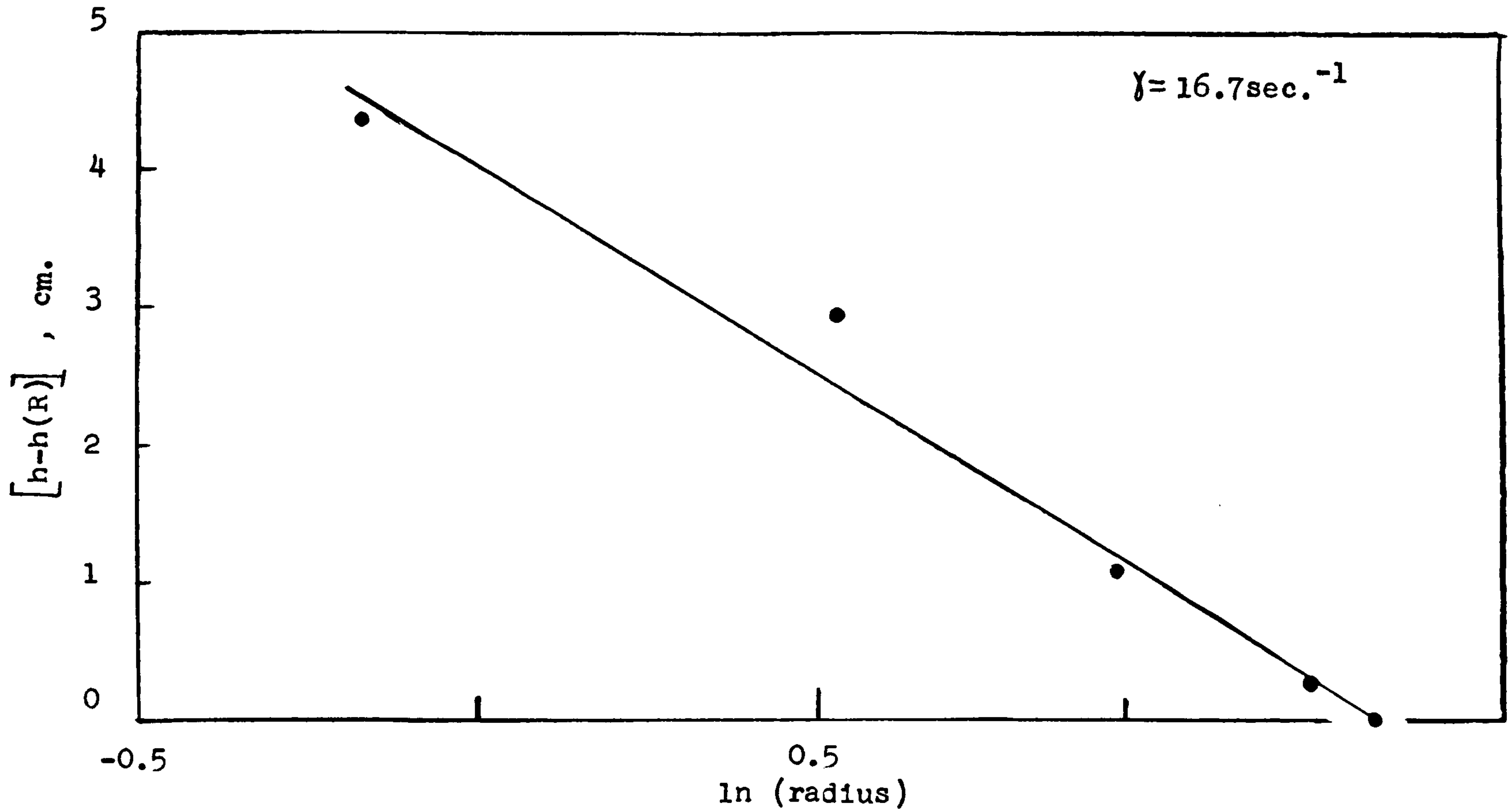


Figure IIID-12. Logarithmic plot of manometric results for fluid number 9.

data is shown for fixed axis ratio on each graph. For the data obtained with $r = 57$ the dependence of normal-force on shear rate is seen to be first order. Power dependences on shear rate of 0.86 and 0.90 for $r = 114$ and $r = 228$ respectively indicate a small deviation from linearity. It is possible that these deviations may result from combinations of wall-effects, particle interactions, and particle induced hydrodynamic disturbances. The approximately linear dependence on shear rate observed here is in qualitative agreement with dilute suspension theory (I). Also, Ericksen's continuum model[15] predicts a linear dependence of the normal stresses on shear rate for the flow considered here.

It will be recalled that the total normal force provides in principle a measure of the first normal stress difference $(p_{11} - p_{22})$ for a fluid[8]. For the suspensions studied here (and the 7.5 cm cone and plate used) one finds this quantity is, roughly speaking, one-fourth the magnitude of the shear stress.

Figures IIID-16 and IIID-17 show the dependence of total normal force on concentration c and axis ratio r. (The straight lines drawn there are somewhat arbitrary).

A theoretical analysis (I) of dilute suspensions of non-interacting, rod-like particles indicates that the viscometric normal stresses should be zero, since an isolated particle tends to spend equal amounts of time in positions of tension and compression. The rather strong normal stresses found in the present study must be attributed, then, to some of the factors

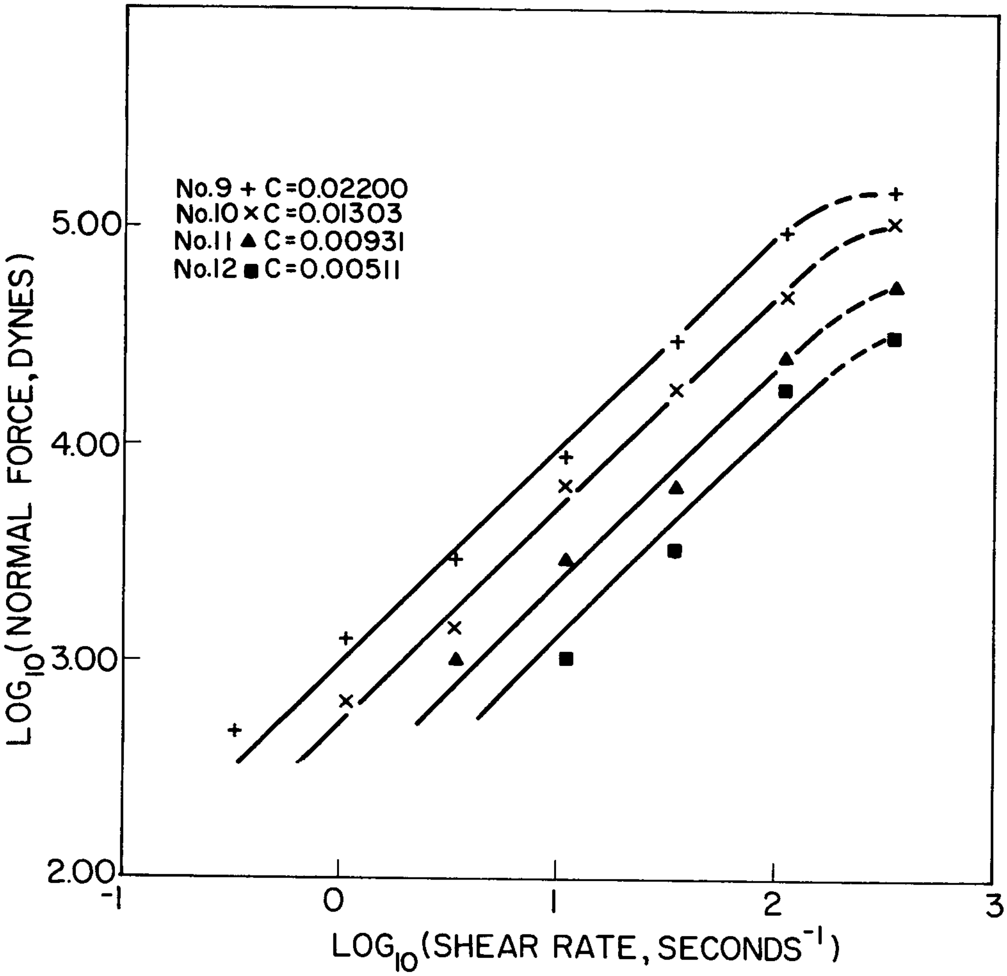


Figure IIID-13. Logarithmic plot of normal force as a function of shear rate for various concentrations with r = 57.

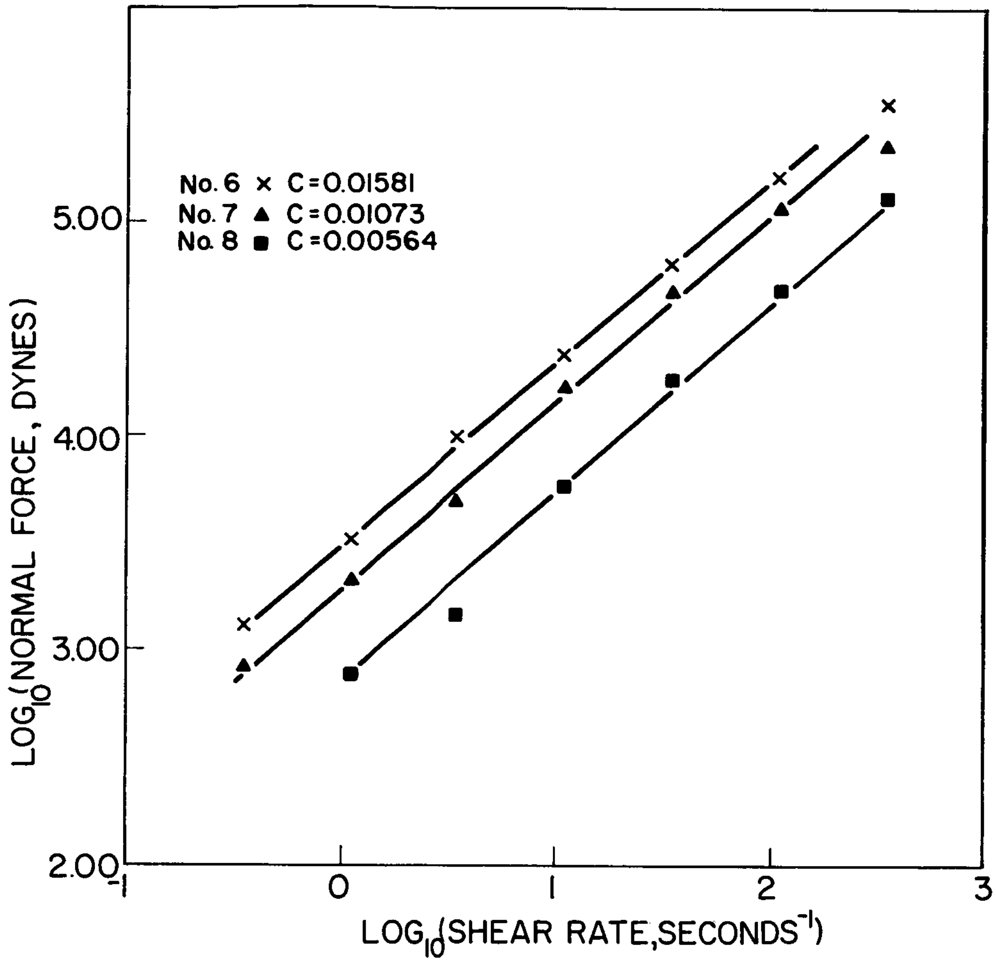


Figure IIID-14. Logarithmic plot of normal force as a function of shear rate for various concentrations with r = 114.

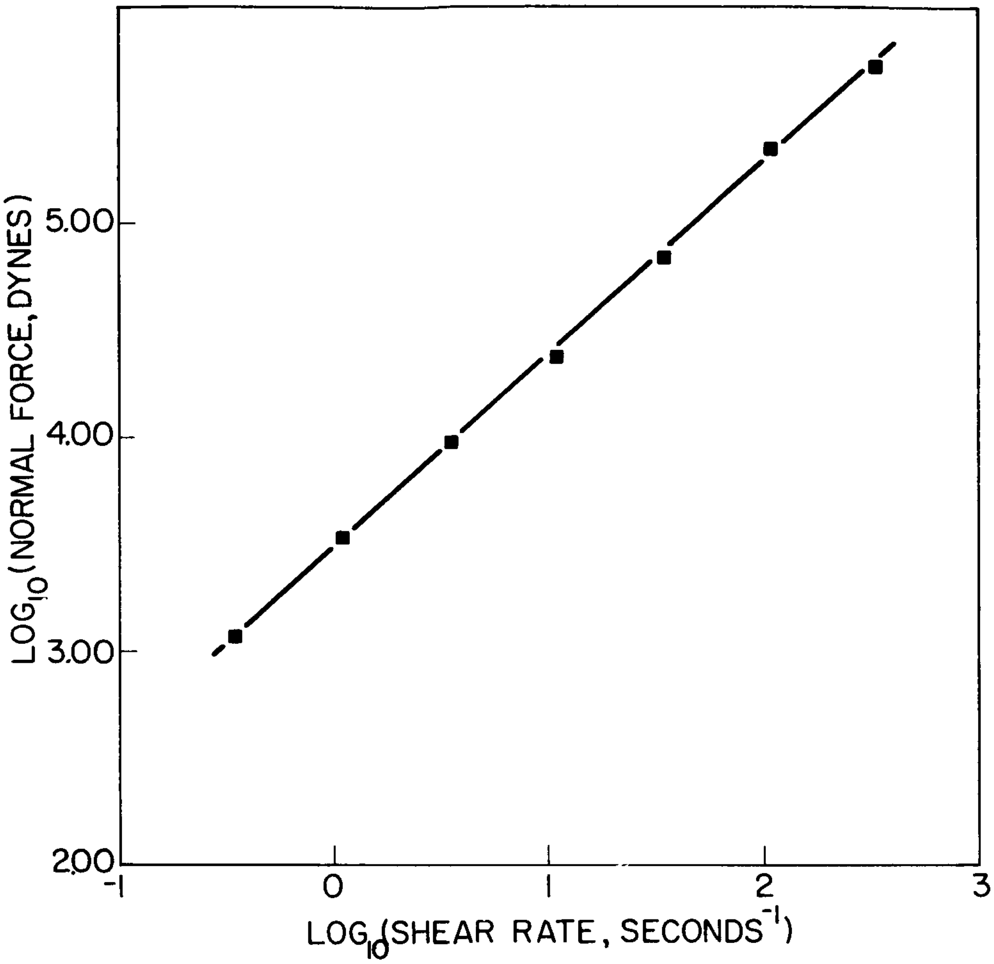


Figure IIID-15. Logarithmic plot of normal force as a function of shear rate for $c = 0.00580$ with $r = 228$.

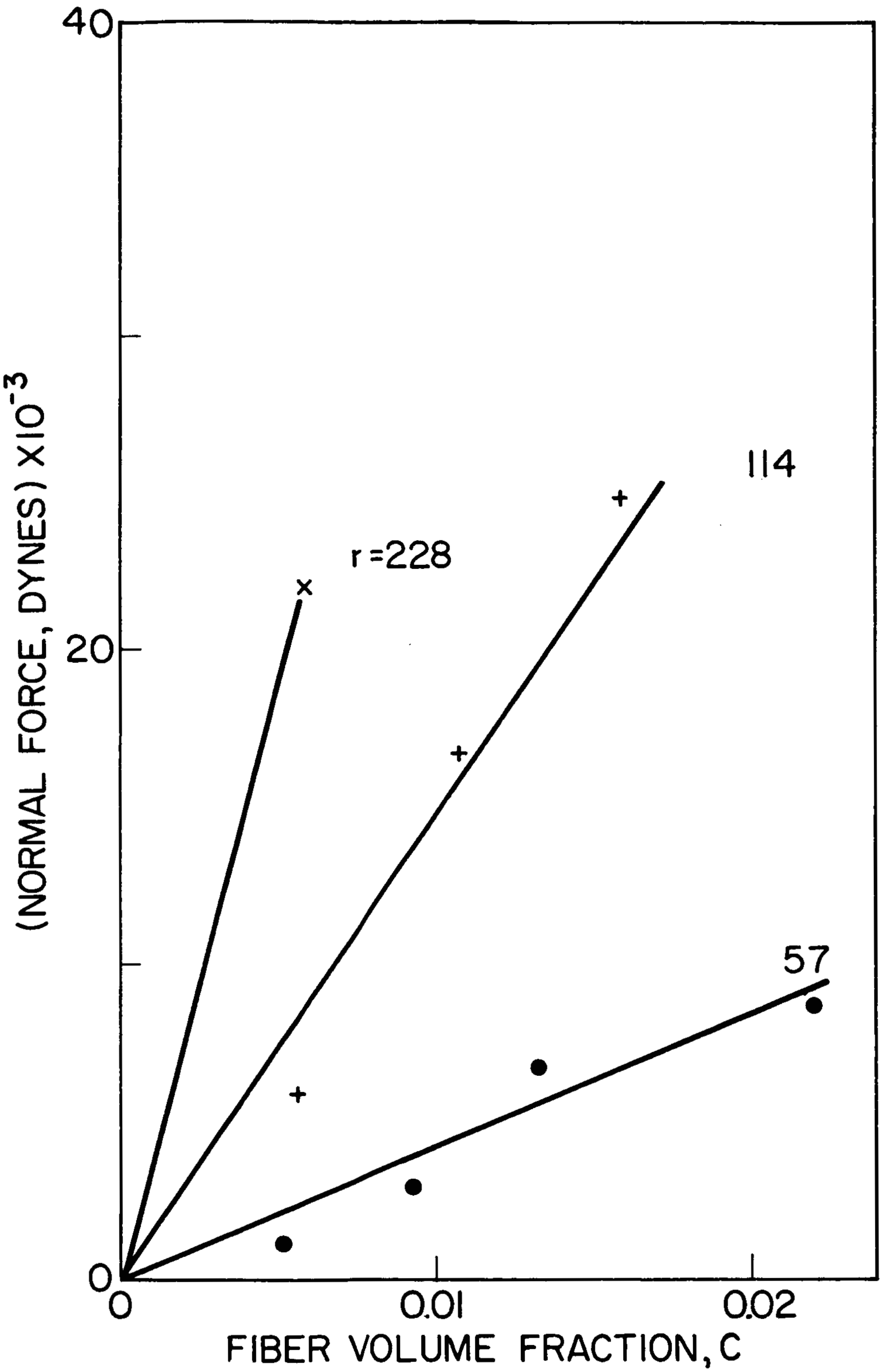


Figure IIID-16. Concentration dependence of normal force for various r's with γ = 11.12 sec^{-1}.

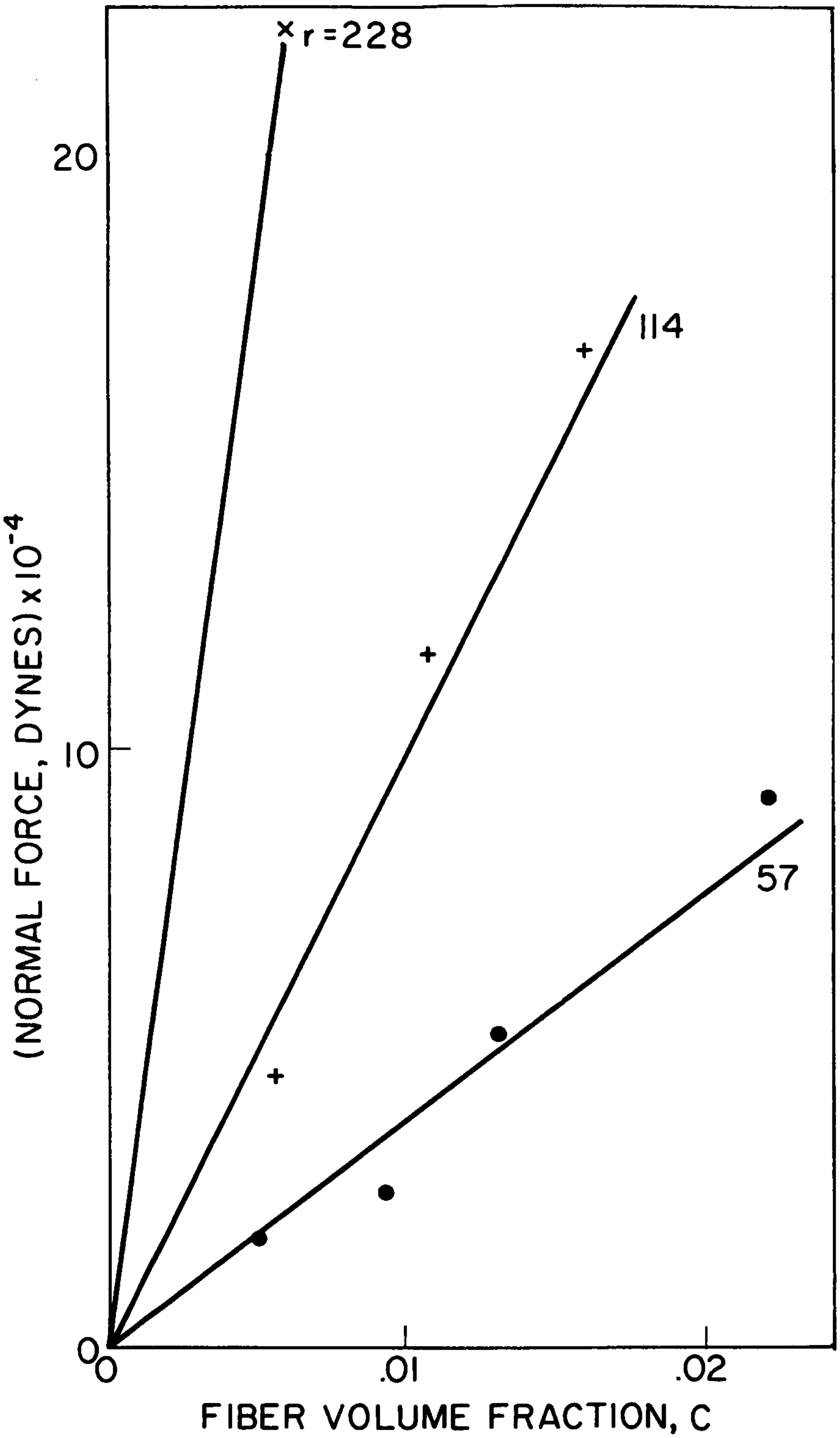


Figure IIID-17. Concentration dependence of normal force for various r's with γ = 111.2 sec^{-1}.

cited above. Of these, wall effects cannot be ruled out, especially for the long partilces, in view of the large ratio of particle length to gap width in the cone and plate used in this study. The maximum gap width, at the outer periphery of the cone and plate is 0.9 mm., while the lengths of the glass rods were respectively 0.2,0.4, and 0.8 mm for the three types of sample studied. This raises serious questions as to the validity of interpreting the suspension behavior from a continuum standpoint, especially, for the longest rods. However, the agreement of viscosity data with previous work[35], done in a wide-gap cylinder, tends to support a continuum interpretation of the data for the shortest rods $r = 57$.

Also it should be recalled that, even on widening the gap appreciably during tests with the manometer head attachment (to give effectively a torsional flow geometry) normal stresses were found to persist.

Thus, while wall effects may be important, it is thought that these effects do not account entirely for the observed normal-stress behavior.

On the basis of a rather crude theoretical treatment, with some ad hoc assumptions as to the angular distribution of rods in steady shear (I), the variable shown on the abcissa of Figures IIID-18 and IIID-19 suggests itself as a parameter for correlating the normal-force data.

With the limited number of data and their scatter, the straight lines drawn on these plots are somewhat arbitrary.

Indeed, it would be difficult to justify a linear dependence of normal stress on concentration, if particle interactions are the primary cause of this phenomenon.

B. Unsteady Shear Tests

In addition to the steady tests discussed above some rather extensive periodic, oscillatory tests were carried out in the cone-and-plate instrument.

In the case of purely oscillatory tests some 300 data points, in the frequency range 0.0377 to 377 cycles per second failed to reveal a significant phase lag between stress and deformation rate. From these tests it was concluded that the suspensions studied were not elastic in their behavior. Similar conclusions resulted from several combined, steady-plus-oscillatory shear tests.

Some studies of "stress-building" following a suddenly imposed shear-rate were also done. The type of behavior obtained here is illustrated by Figure IIID-23.

Following a steady shear in one direction the flow is stopped and then after a time is restarted abruptly. If the new shear direction is that of the previous, then the stress builds up a monotonically and rapidly to its former steady level. By contrast, if the flow direction is reversed there is a distinct "over shoot" (about 10% of the steady value) of the stress, in the approach to steady state. This latter phenomenon is doubtless due to the rearrangement of particle configurations accompanying a change of shear direction and could depend on wall effects.

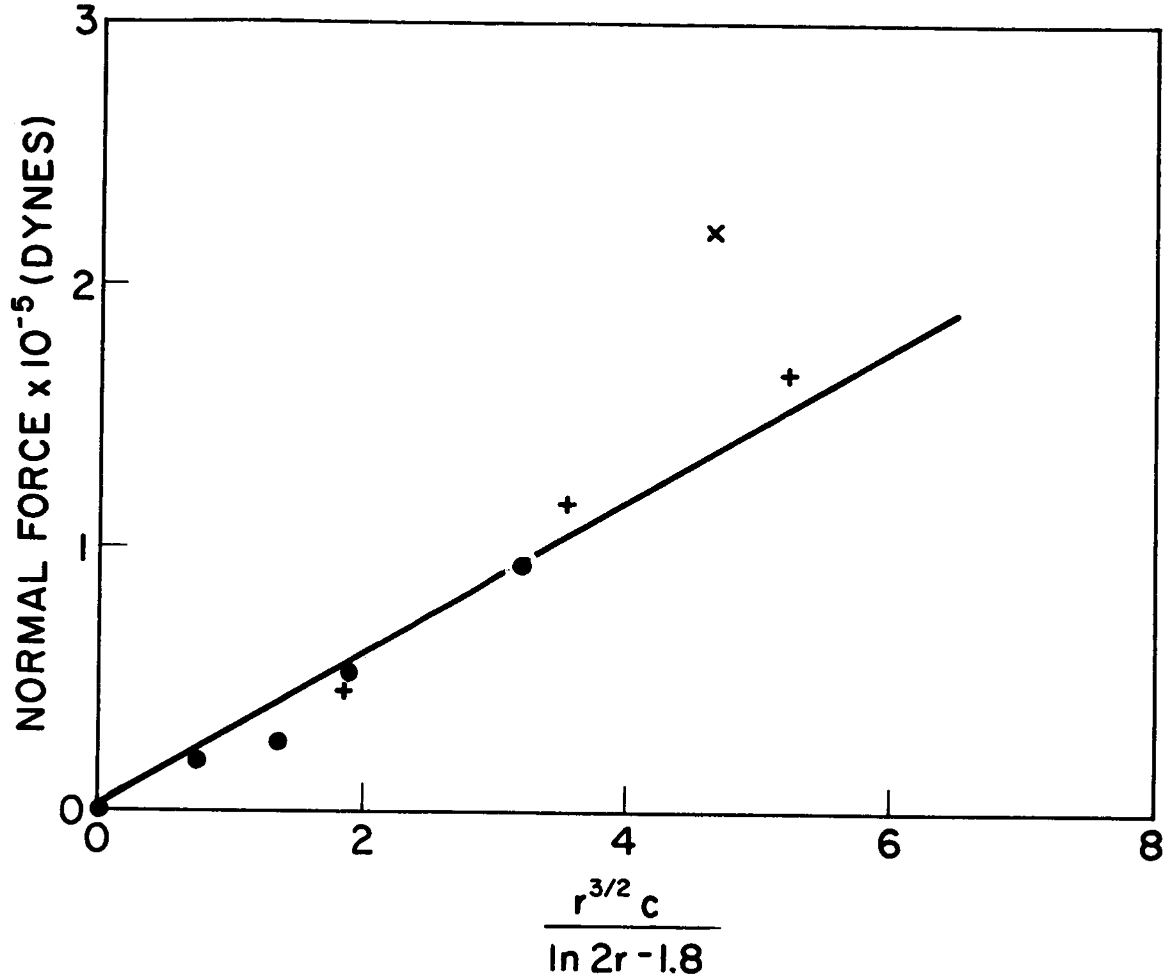


Figure IIID-18. Plot of normal force with dilute suspension parameters for γ = 11.12 sec^{-1}.

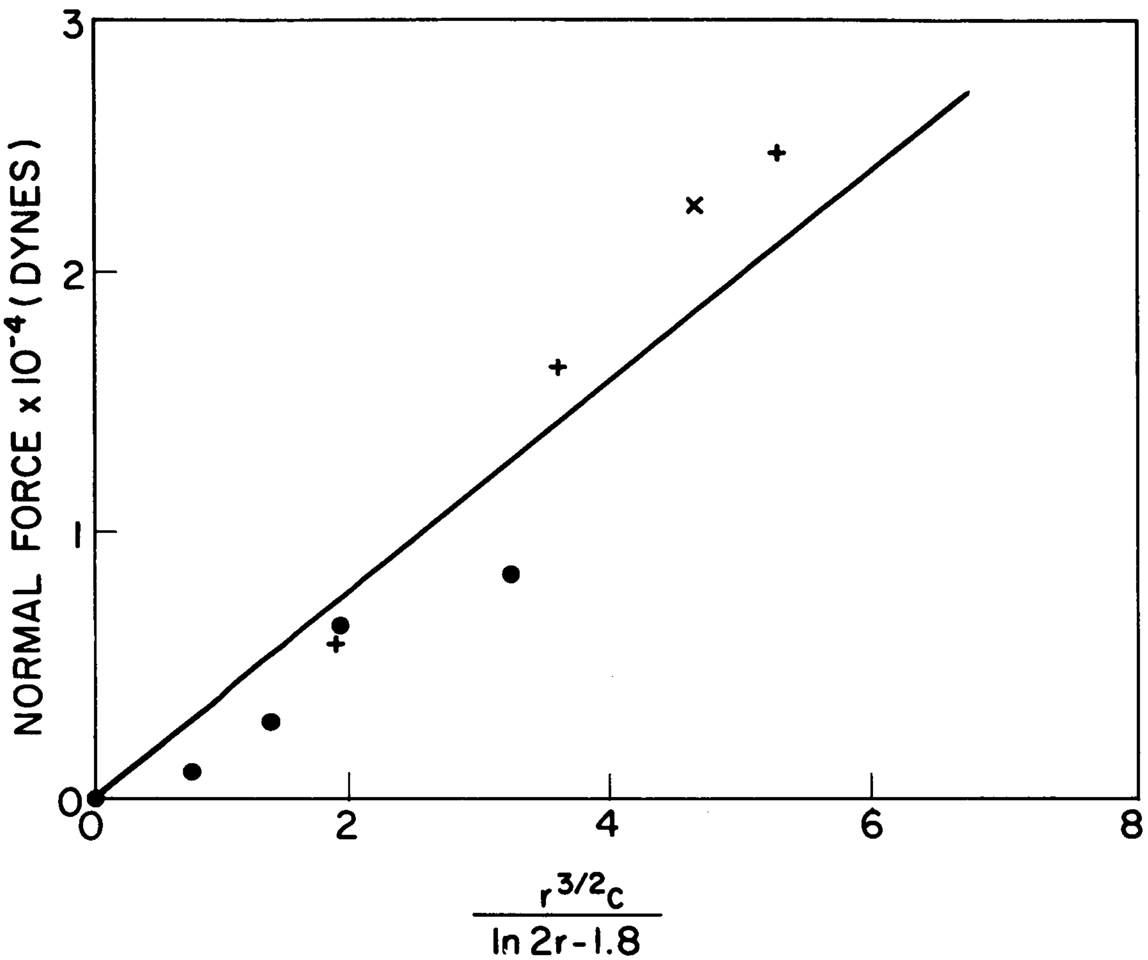
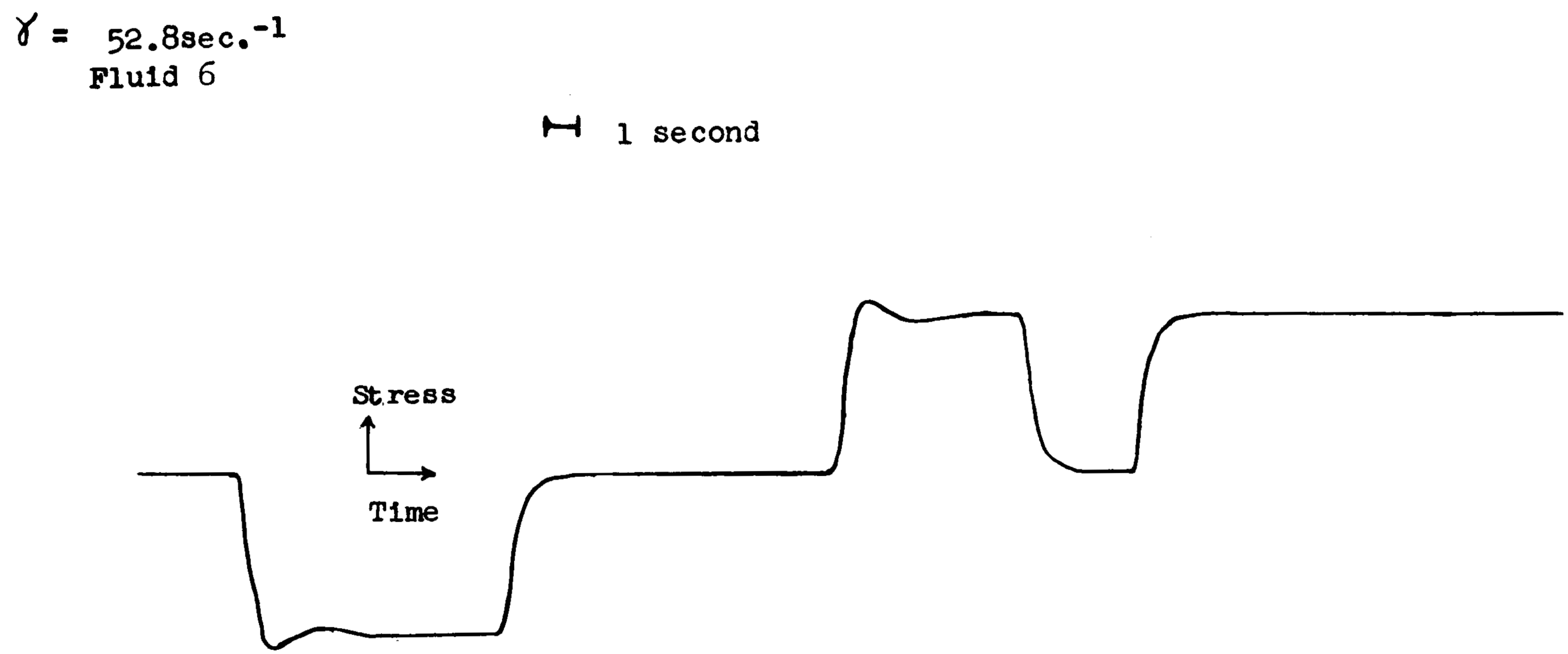


Figure IIID-19. Plot of normal force with dilute suspension parameters for γ = 111.2 sec^{-1}.



Clutch-brake Always Used With Motor At Full Speed

Figure IIID-23. Direction dependent time effect.

C. Conclusions

From the results of this study we conclude, with past workers, that general non-Newtonian effects can be expected to occur in suspensions of microscopic particles. In particular, for the glass-fiber suspensions studied here the phenomena of shear thinning and normal stress effects are found to occur in steady shear tests. However, these effects are relatively weak compared to those often found in other non-Newtonian systems such as, most notably, concentrated solutions of macromolecules.

In the present systems the decrease in the apparent viscosity was on the order of 5-10% over a four-decade range of shear rate(1.67 to 167) except for the most concentrated suspension of the longest particles, and the first normal stress difference was roughly one fourth the shear stress. Moreover, stress-relaxation or elastic effects were found to be negligible in unsteady shear tests on the present systems.

On the basis of dilute-suspension theory it is concluded that no significant viscometric normal stresses nor shear thinning should occur in suspensions of non-interacting rod-shaped particles. Hence, the non-Newtonian behavior found in this study is attributed to the combined effects of particle anisotropy and particle interactions.

Also, wall effects (particle-wall interactions) may account partially for the observed behavior, since the ratio of particle-length to maximum-gap-width (in the cone and plate) ranged from about one-fourth to unity for the longest particles. While the viscosity data obtained here for the shortest rods agrees favorably with that of a previous study, wall effects cannot be conclusively ruled out of the present results.

With this reservation as to a continuum interpretation of the present experimental data, it appears that parameters obtained from dilute suspension theory are useful for correlating rheological properties with particle shape and concentration. Also, the near linear-dependence of normal-stresses on shear rate and the unsteady shear behavior of the suspensions suggest that an "Anisotropic fluid" model,[15,28] might provide an adequate rheological description of such suspensions.

List of Symbols

c	Fiber concentration, volume fraction
p_{ij}	Deviatoric stresses $(i, j = 1,2,3)$
r	Axial or length-to-diameter ratio for rods or fibers
η	Apparent viscosity of suspension (poise)
η_o	Viscosity of suspending fluid (poise)
η_{sp}	Specific viscosity $(\eta - \eta_o)/\eta_o$
γ	Shear rate (sec^{-1})

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