INFLUENCE OF VISCOSITY ON GRINDING IN BALL-MILLS.

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by

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ABSTRACT.

An investigation has been made on the rheology of suspensions of mineral powders and the influence of rheological properties of ball-mill pulps on the efficiency of grinding.

A viscometer has been developed which permits the determination of the viscosity of coarse, settling, mineral suspensions. This instrument has been used to measure the viscosity of a series of pulps with various percentage solids, particle sizes, shapes and densities over a range of shear-rates. A brief study has been made of the influence of a detergent on the viscosity of such suspensions through its effects on the friction between the particles. (Some experiments on friction are reported in an Appendix to the thesis).

An experimental instrumented ball-mill has been developed which, with the viscometer, has permitted a study of the operation and efficiency of ball-mills. Various series of tests have been conducted at constant or variable speeds and over ranges of viscosities with synthetic or practical pulps. Many of the tests have been

carried out with equivalent energy inputs and equivalent ball paths. The main result from these experiments has been the determination of a relationship between the viscosity of the pulp and the efficiency of grinding. Reasons are given for the various features of this relationship.

Slight increases in the maximum efficiency of grinding have been obtained by the use of certain chemical additives. Friction between the grinding surfaces is considered to be an important function of the pulp.

CONTENTS.

List of Symbols.

F = force (p.21)
A = area
v = velocity
x = width of sheared material
η = viscosity
Re = Reynolds number (p.28)
V = mean velocity
κ = instrument length parameter
ν = kinematic viscosity
η_s = viscosity of suspension (p.31)
η_L = viscosity of liquid medium
θ = vol. conc. of solids
k = interaction factor (p.32)
K = constant
v = av. void diameter
$a_0 a_1 a_2 a_3$ k, β = constants (p.33)
η_{sp} = specific viscosity (p.34)
f = axial ratio
η_o = viscosity of suspending fluid
F = fluidity of suspension (p.35)
η = viscosity
F_o = fluidity of liquid
$\theta_{eff}, \theta_\infty$ = concentrations
R = ratio of solid to liquid density (p.36)
D = average particle size (p.36)
D_m = mean particle size (p.37)
η_{rel} = relative viscosity (p.37)
τ_w = shear-stress at wall (p.38)
D = dia. of tube,
Δp = pressure drop
L = length of tube
k = friction factor
u = vel. of flow
E = shear rate
n = non-Newtonian index
ρ = density (p.51)
η = viscosity of oil
η_o = viscosity at initial temp. (p.52)
E = activation energy
$\mathcal{R}$ = gas constant
T = abs. temp.
η_o = viscosity of solvent (p. 53)
η = viscosity of suspension
λ = specific conductivity
a = particle radius

ζ = zeta potential
ϵ = dielectric constant
η_a = apparent viscosity (p.55)
T = temperature
n = no. of particles/cc.
c_i = ionic concentration
k = Boltzmann's const.
K = $\frac{1}{\text{double layer thickness}}$
R_o, R_1 = radii of cyliners (p.77)
h = depth of shear
U = max. surface velocity
D = shear rate (p.84)
τ = shear stress
K = constant
n = degree of shear-rate dependence
R_c, R_b = radii of cylinders ($R_c/R_b = \epsilon$)
= speed of rotation
$f(sb)$ = shear rate
S = speed of mill (p.124)
D = Diameter
M = surface of balls factor
C = consistency
F = diameter factor
E = relative efficiency (p.126)
W, W_1 = wts. of charges
S_1, S_o = surface factors
N_1, N_o = grinding factors
K = size modulus (p.132)
y = cumulative fraction of material finer than size x
x = mesh size
α = distribution modulus
θ = shaft deflection (p.150)
T = torque
J = polar moment of inertia
l = length of shaft
q = shear stress
r = radius of shaft
μ = coef. of friction (p.225)
W = trolley wt.
w = balance wt.
x = distance of traverse
t = time of traverse

Chapter 1.

INTRODUCTION.

Ball-milling is a convenient, mechanical, operation used widely for the comminution of minerals, and for the preparation of colloidal dispersions.

Patents were taken out in this country for the use of the ball-mill, as early as 1863 by R. Brooman. The early mills were used mainly for the grinding of metalliferous ores and ceramic materials. The application of ball-mills for pigment dispersion in the paint and allied industries is, however, comparatively recent.

The work in this thesis has been carried out from the standpoint of the mineral technologist. His interest is in grinding an ore in order to liberate particles of valuable mineral from worthless gangue. It is, also, to reduce the ore to a form in which it presents a large surface area to leaching reagents. Much of the work, however, will apply to the other industries already mentioned.

The main part of a ball-mill is a hollow, steel, cylindrical chamber partially filled with steel balls or pebbles. The mill is rotated about its major axis, which is mounted horizontally. A certain amount of the material to be ground is placed in the chamber, and rotation of the mill causes the balls and material to be lifted up the inner ascending wall, until they tumble over each other. This action crushes the particles of material which get caught between the balls themselves, and against the surfaces of the mill. Grinding is thought (1)(2) to be brought about by a combination of failure under compression from impact, chipping of corners by shear, and abrasion of the material by adjacent particles and balls.

Mills are made from about 5" to 12'0" in diameter, depending on the quantity of material to be ground, and, generally, have a diameter-to-length ratio of about 1 : 1.5.

A charge of rods is sometimes used instead of balls. This produces a more closely sized product, and is used particularly in gravity concentration processes where the presence of over-ground material is disadvantageous. Rods, being heavier than balls, are capable of greater impacts required for coarse feeds, but are less efficient than balls for the production of finely ground material.

In mineral processing the feed is usually introduced into the mill in the form of an aqueous slurry, when it is termed "wet-milling". This is done to prevent aggregation of the fine particles, to produce a pulp which will coat the mill surfaces (improving grinding efficiency), and to assist in handling the ground product. The latter reason is of particular importance in continuous flow grinding. In this case the mill forms part of an ore processing circuit; the material is fed into one end of the mill, and after being ground it is discharged by means of a scoop, or through a grate or overflow trunnion. The product is classified and oversize material is re-introduced back into the mill feed. Fully enclosed mills are termed "batch mills". These are more suitable if a fine product is required and the long residence time in a continuous mill would be impracticable. Batch mills are naturally used when only small quantities of material are to be ground.

The inner surfaces of ball-mills are usually furnished with liners which can be replaced after excessive wear. The liners often take the form of projections which lift the grinding bodies to produce a higher trajectory; such projections are termed "lifters".

The work described in this thesis is mainly concerned with wet, batch mills possessing smooth liners. Closer control can be exercised over many of the variables in batch rather than in continuous operations. The results, however, are largely applicable to either.

Ball-milling is an energetically inefficient process, accounting for an important part of the costs of a mineral processing plant. For example, grinding represented 40% of the plant operating costs for the processing of some Swedish, low-grade iron ores and 12% of the total mining company operating costs (3).

Only a fraction of the total power going into milling is used to produce new surface. The actual size reduction of the material is almost incidental to the operation of the mill, for there would be little reduction in the power consumed if there were no pulp present.

Despite the simplicity of operation of ball-mills, the actual grinding process and dynamics of the grinding bodies is complex and not yet fully understood. In an attempt to improve the efficiency, many workers have sought to discover, by means of extensive tests, the optimum grinding conditions in terms of mill dimensions, percentage charge, number and size of balls, mill speed etc.. Plant operators are normally obliged to optimize empirically the working conditions of a given mill.

Some work has been directed to the material being ground, with a view to changing its characteristics by means of additives of various kinds. Rehbinder and co-workers(4) in 1944 claimed that it was possible to change the mechanical properties of materials by the adsorption of electrolytes and surface-active agents from solution. He suggested that the liquids were adsorbed into micro-cracks and fissures in minerals. Thus, the freshly-formed surfaces were prevented from healing up as soon as the stress was released. The work of Rehbinder is cited by many workers (5 to 11), (some of whom are quoted below), in support of their results on additive grinding. Although the adsorption theory is quite generally accepted, other reasons are also suggested for increased efficiency of grinding when additives are used.

Von Szantho (6) in 1949 conducted a series of tests using oleic acid, sodium oleate and "Flotigam P" (C_{12}- C_{14} amine from coconut oil). He claimed up to 120% increase in surface area, when grinding quartzite in a wet, batch mill. Von Szantho concluded that the coefficient of friction is of major importance in the grinding rate. He stated that at high concentrations, the decrease in the coefficient of friction outweighs the advantages gained from the reduction in surface energy arising from the surface adsorption of the additive.

Frangiskos and Smith (9) in 1957 obtained increases in efficiency of grinding by using sodium hydroxide, sodium carbonate and other surface active agents such as silicones in a laboratory drop-weight stamp mill. Some of their work was repeated in 1960 by Ghosh, Harris and Jowett (10) who obtained similar grinding efficiencies. Gilbert and Hughes (12) in 1961 attempted to repeat this work on the effect of additives on grinding, and to investigate further the Rehbinder micro-crack theory. Increases in the efficiency of grinding were obtained using a stamp-mill, but some of their tests with a ball-mill were inconclusive. A series of experiments using "Armac T" were irreproducible, inspiring further exploratory tests. "Armac T" is a well-known collector for quartz: it is a mixture of saturated and unsaturated C_{16} and C_{18} amine acetates derived from tall oil. The possibility of the additive affecting the viscosity of the medium was investigated by carrying out tests with a number of suspending fluids, the viscosities of which ranged from 0.469 to 1.056 cP. This was brought about by deliberately altering the temperature of the mill. However, no significant changes in grinding efficiency were observed. They suggested, from further tests using a rod-mill, that the flocculated pulp might have had a "cushioning" effect on the rods. The degree of flocculation was noted to be proportional to the concentration of "Armac T".

The viscosity of the suspending fluid was considered to be important by Shepherd (7), as affecting the velocity of penetration of liquid into microcracks. Increases in the efficiency of breakage were obtained using a drop-weight apparatus with additions of sodium chloride and aluminium chloride. He concluded that these increases were due to the Rehbinder effect, but no conclusive results were obtained, however, for the influence of viscosity.

Rehbinder (4), in conjunction with the microcrack hypothesis, proposed that a possible effect of additives on fine grinding was to inhibit agglomeration of fines by chemical dispersion, and hence prevent "cushioning" of the impacts.

The results obtained using "Armac T" and the work of Von Szantho, Shepherd, Rehbinder etc. indicate the likely dependence of the efficiency of grinding on the viscosity of the pulp rather than the suspending fluid alone. (The term "viscosity" is used here and throughout the thesis to describe a resistance to shear, of coarse slurries as well as pure liquids. It is realised that the prefix "apparent", "effective" or "pseudo" might be appropriate but the term "viscosity" alone is used for convenience. Similarly, the term "viscometer" is used for its measurement rather than "viscosimeter", "consistometer", etc.). Furthermore, until a knowledge of viscous effects has been obtained, other laboratory experiments on the Rehbinder effect on ball-milling are likely to be misleading, because the additives would be likely to change the rheological properties of the pulp.

If the viscosity of the pulp within a ball-mill were increased to a very high value, it is evident that the grinding bodies could adhere to the wall of the mill and, hence, reduce the efficiency to zero. On the other hand, a thin fluid pulp would be associated with a very different set of grinding conditions. Thus, it is possible for the viscosity of the pulp to exert a considerable influence on the rate of grinding.

The present project was planned, therefore, to investigate the degree of dependence of the efficiency of grinding upon the rheological nature of the pulp. Then, to discover if, and how, additives may be used to influence the rate of grinding in another way from the adsorption of surface active agents into micro-cracks.

A consideration of the limiting values of the viscosity of the pulp reveals that consequent changes in power consumption of the mill are bound to occur. Furthermore, experiments on milling efficiency, as a function of viscosity or additives, are liable to misinterpretation unless corresponding changes in the power consumed are determined. This is a variable which has been largely neglected in previous investigations.

Few studies have been made of the influence of viscosity of the pulp on grinding. This is mainly because of the lack of knowledge of the rheological nature of coarse suspensions, such as those present in ball-milling. Without this knowledge, pulps may only be characterised in descriptive terms, or with reference to some other parameter such as particle size or pulp density. The main problem, in the measurement of the viscosity of coarse, settling suspensions, is maintaining homogeneity throughout the sample while it is being subjected to a shearing stress. Most of the investigations of coarse suspensions have been made using particles of equal density to the suspending fluid, or using slow-settling particles. Other studies have been made of settling suspensions using efflux-type viscometers. Their main disadvantage is the inability to provide a range of shear-rates to permit the study of anomalous behaviour under Newtonian conditions of shear. Rising or falling sphere viscometers are also subject to this problem of indeterminate shear-rates. Although a rotary, concentric cylinder type of viscometer is the most acceptable, the problem of settling particles still remains. Therefore, the first part of the project was to develop an instrument which would permit the examination of the rheological properties of suspensions, such as those found in ball-milling. Having developed such an instrument, the rheological characterisation of a number of types of settling suspensions was considered to be a worthwhile undertaking. In view of the large number of different types of particles encountered in ball-milling, it was considered necessary to study the relative influences on suspensions of particle shape, size, size distribution, density etc. Not only is such knowledge valuable in comminution but also in the hydraulic transport of solids (13)(14) or the mixing of ceramic materials etc.

A literature survey has been carried out on the rheology of suspensions. The present thesis contains a summary of generally accepted principles governing the flow of suspensions, and a résumé of results of investigations associated with the problem.

Ball-mills may contain mineral particles ranging from sub-micron size (1 micron (μ) = 10^{-4} cm) to lumps of several centimetres across. The work described in this thesis included viscosity measurements on suspensions of mineral particles of up to 210μ. It is realised, however, that much larger particles may contribute to the measurement of the viscosity of a suspension. This would depend upon the viscosity of the suspending fluid, the concentration of solids, the average particle size and, to some extent, the measuring instrument (15).

Several theoretical studies of the rheology of coarse suspensions and the mechanics of the operation of ball-mills have been published. This work is reviewed later; but the primary object of this investigation was to obtain experimental evidence as to the influence of rheological factors on the operation of a ball-mill. Not only were such data lacking, but the situation was judged to be too complex for theoretical treatment, at least before the qualitative phenomena have been clarified.

A preliminary consideration of the problem revealed that there was no simple relationship between viscosity of the pulp and grinding efficiency. Many variables were likely to be involved, for example, size of mill, speed of rotation, loading, size of balls, nature of mineral being ground, solid/liquid ratio, particle size distribution, particle shape, time of grinding, degree of flocculation etc. The character of the pulp must be taken to include a wide range of rheological phenomena such as thixotropy, dilatancy, plasticity etc.. Each of these must be expected to produce different effects on grinding. Furthermore, each set of conditions within the mill is likely to have a different effect on impact from attrition grinding.

The conventional approach to a complex system of this type, is to design a statistical "block" of experiments and to fix those variables which are believed to be independent. Such an approach is practicable when only a small number of variables is involved. A comprehensive investigation involving many variables would, however, lead to a vast number of experiments, the majority of which would reveal nothing.

The rational approach, which was likely to yield a maximum of information in the limited time available (2 to 3 years), was to restrict the enquiry to a few clear-cut viscosity effects, which seemed likely to influence the rate of grinding. It was important to try to anticipate which were the most significant factors, on the basis of existing information. An appraisal of the problem was made by Dr. J.A. Kitchener who proposed a number of tentative hypotheses as to how the viscosity might affect grinding. The validity of the hypotheses could then be tested by means of practical experiments. It was suggested, for example, that changes of viscosity, if great enough, are bound to influence the movement of the grinding bodies and, hence, the power consumption, and the number and momentum of the impacts in the mill. Several methods of testing were put forward, including determination of the rate of breakage of closely controlled feed charges, cinematography using a glass-ended mill, analysis of the sound level and the study of the size and frequency of indentations on a few soft balls introduced into the mill. The influence of the viscosity of the pulp on the cataracting speed of the mill was suggested as a likely source of interest, together with consequent changes in the power consumed. The proposed method of testing was by grinding a small charge of coarse, brittle solid suspended in acid-soluble, synthetic pulps of various viscosities. The size distribution of the hard material was to be determined by sieve analysis after the synthetic pulp had been removed by acid. A number of grinds of short duration were proposed, above and below the cataracting speed of the balls. This permitted the study of any correlation between the onset of cataracting and the effective fracture of large particles. A glass ended mill would be required to permit observation of the onset of cataracting.

It was suggested that much of the power in wet milling is dissipated in viscous flow of the medium. A study was proposed of the influence of high viscosity on the rate of attrition grinding. The method of testing was to be similar to that for the cataracting tests but at lower speeds.

Finally, it was postulated that the apparent viscosity of a practical pulp is influenced by the state of dispersion of the sub-sieve fractions, and that this affects the subsequent progress of grinding. In order to test this, a large batch of clean, ground quartz was to be prepared, and different samples treated by various dispersing or coagulating agents. The influence of each type of media was then to be observed on the rate of grinding by sedimentation analysis, the power consumption, and the mechanics of the grinding operation.

The proposals were entirely tentative and subject to amendment as the project progressed. These, however, together with a knowledge of other investigations in this field, provided a basis for further experimental work.

A summary is given in this thesis of previous studies which have been made on the influence of viscosity on the different variables connected with grinding. This includes such factors as the diameter of the mill, speed of rotation and power consumption. Those parameters connected with the ground product, such as the size distribution, average particle size, etc. have also been studied.

The influence of friction between the grinding bodies appeared to be of particular importance at one stage in the work. Some measurements were made of the coefficient of friction between mild steel surfaces, in the presence of several of the pulps used in the milling experiments. The results are recorded separately in an appendix to the thesis.

A second appendix is also included in the thesis, containing tables of results, both measured and calculated, of the viscosity and grinding experiments.

Chapter 2.

VISCOSITY OF SUSPENSIONS.

The purpose of this chapter is to show the extent of present knowledge of the rheological properties of suspensions. Many practical and theoretical investigations have been made on this subject, but few relate directly to suspensions similar to those found in ball-milling. A review is given starting at the basic concept of viscous flow and describing the well-known types of anomalous behaviour which exist. The results of others, which are relevant to the present project, are analysed: discrepancies and gaps in the knowledge of suspensions are discussed in order to show which aspects are worthy of further investigation.

Fluids are considered to flow according to the basic law of Newton, which is visualized by the flow of a liquid between two flat plates, to obey the formula:

$$F = \eta A(dv/_{dx}) \tag{2.1}$$

where F is the force to maintain velocity, A is the area of plates, $dv/_{dx}$ is the velocity gradient and η is the coefficient of viscosity. The c.g.s. unit of flow is the poise and its hundredth part the centipoise (cP). A substance has a viscosity of one poise when a shearing stress of one dyne per sq.cm.(F/A) produces a velocity gradient of one cm. per sec. per cm. (or $sec^{-1} = dv/_{dx}$) (16)(17)(18). Water has a viscosity of approximately 1cP at 20°C.

Graphs of shear-stress against shear-rate are used to describe the viscosity of a fluid. In the case of Newtonian flow, the shear-stress is proportional to the shear-rate, i.e. the coefficient of viscosity is constant. A great number of suspensions, however, behave differently, and such graphs are commonly used to illustrate the various types of viscous flow and associated anomalies.

Newtonian flow is the simplest type of viscous behaviour, occurring in all simple and pure liquids, i.e. homogeneous liquids, excluding macromolecular fluids. Newtonian fluids are considered to have a loose molecular structure in which the cohesive forces are not great enough, in competition with disordering effects of thermal energy, to impart to the fluid any measurable rigidity (Fig.2.1).

Highly diluted, deflocculated suspensions may also behave as Newtonian fluids if the structure is loose and discontinuous, and shear can take place in the vehicle which fills the spaces between the particles (19).

Plastic materials do not flow until the applied shearing stress exceeds a certain minimum value known as the yield value (f), which is indicated as an intercept on the stress axis (Fig.2.2). Below this value the material can hold its shape (slightly distorted) indefinitely (20). Beyond the yield stress the material behaves in a Newtonian manner, the slope of the graph giving the "pseudo-viscosity".

These materials are also known as Bingham-bodies. Plastic behaviour was explained by Bingham (21) on the assumption that the particles of a plastic suspension form a network or structure. This formation is responsible for the yield stress and must be broken before flow can be initiated.

Dilatancy is a type of flow found with suspensions of high solid content. It is characterized by an increase in viscosity as the rate of shear increases (Fig.2.3).

The particles of an undisturbed, dilatant suspension do not interact and if the material is colloidal it is deflocculated, and possesses a low yield value compared with a flocculated structure. It was suggested by Reynolds (22) that the particles settle to a state of minimum voids

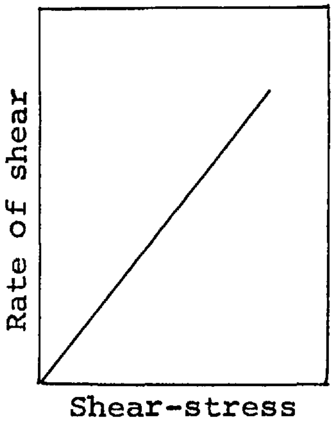


Fig.2.1
Newtonian flow

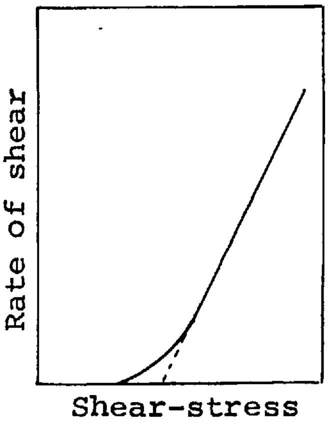


Fig.2.2
Plastic flow

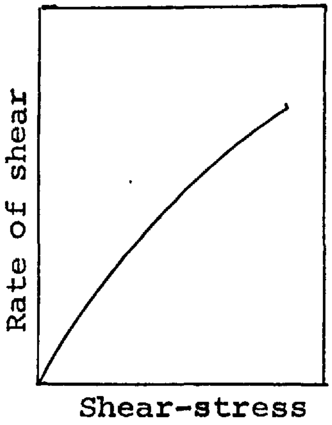


Fig.2.3
Dilatant flow

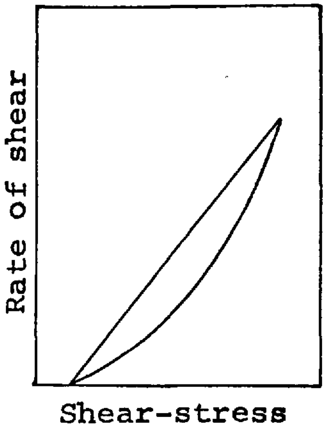


Fig.2.4
Thixotropic flow

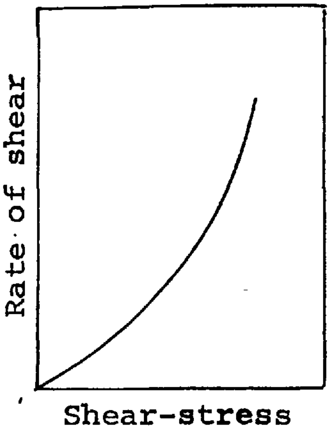


Fig.2.5
Pseudo-plastic flow

Types of rheological flow.

and that any disturbance causes rearrangement of the particles with greater void volume. The total volume of the suspension consequently appears to increase or dilate. A classic example of this type of behaviour is the dry, hard appearance of wet sand under stress. This behaviour has been explained as a loss of lubricating effect between the particles under stress, causing them to pile up and act as a solid mass. The possibility has been suggested (23), of an electrokinetic effect resulting from the high charge density of the closely packed particles. A field is set up, which is resistant to flow at high shear-stresses, owing to the proximity of strong forces of repulsion. However, in coarse suspensions dilatancy is clearly caused by a mechanical "jamming" of particles.

Another type of rheological flow found in suspensions is "thixotropy". It is defined (24) as "that property of a body by virtue of which the ratio of shear-stress to rate of deformation is temporarily reduced by previous deformation". The time factor is essential, distinguishing thixotropy from pseudoplasticity; both types are illustrated in Figs.2.4 and 2.5 respectively.

The degree of thixotropy is measured from the loop on the shear-rate - shear-stress diagram (Fig.2.4). Thus, the time dependence of the reduction in viscosity is estimated (25).

Thixotropy is mainly found in colloidal, flocculated suspensions, and the concentration at which it occurs is much lower for elongated or plate-shaped particles. An explanation of this phenomenon is that flow is restricted by weak inter-particle bonds, which have to be broken before flow may start; these bonds reform again when the material is left undisturbed. The re-formation of these links is accelerated in certain suspensions by gentle agitation or stirring; this property has been termed "rheopexy" by Freundlich and Juliusberger (26).

Pryce-Jones (27) distinguished two types of what is commonly, loosely called "thixotropy", one being the normal type and the other being "false-body" in which the time of restoration of higher consistency after agitation is very short. Pseudo-plastic materials also possess this latter quality although they do not possess a yield stress as do most thixotropic suspensions. Pseudo-plastic suspensions exhibit a decrease in shear-stress with increasing rate of shear with no time-dependent, thixotropic loop or yield stress (Fig.2.5). Pseudo-plasticity, or "structural viscosity" as it is sometimes called, has been explained by a temporary reduction in particle size, by the shearing of the solvated shells in lyophilic suspensions, when subjected to a shear-stress (28). It could also be a reversible association-dissociation of macromolecules. Another cause of pseudo-plasticity is the reorientation of long molecules in the direction of flow (which is opposed by rotational Brownian motion (29).

In solutions of macromolecular polymers of flexible chain structure, flow may produce an unravelling and extension of the chains. When the shearing stress is removed the molecules relax to their normal length. This phenomenon produces "visco-elasticity". (It does not occur, however, with simple, particulate suspensions).

The types of flow defined so far do not cover the whole range, but serve only as broad categories for labelling the majority of suspensions. Certain foodstuffs, paints, printing inks, etc., exhibit more than one of the characteristics described. Fig.2.6 shows examples of these combined types of flow where (a) is a thixotropic-dilatant suspension such as vinyl-plastisol paint or certain clay-water suspensions: (b) is a thixotropic-pseudo-plastic material exemplified by emulsion paints: (c) is a thixotropic-plastic suspension such as certain silicone fluids, honey and mustard.

The foregoing types of flow apply to laminar or streamline behaviour in which distinct layers of fluid pass over each other. In turbulent flow no distinct layers are observed; layers mix one with another, creating eddy currents. The criterion for the onset of turbulence for Newtonian fluids was expressed by Reynolds (30) as a ("Reynolds") dimensionless number:

$$R_e = \frac{Vx}{\nu} \tag{2.2}$$

where x is the instrument length parameter, V the mean velocity and ν the kinematic viscosity. When this number exceeds a certain value the flow becomes turbulent, and the previous types of flow become, more or less, indistinguishable and a flow curve, as shown in Fig.2.7, is obtained (31). The apparent viscosity increases with increasing rate of shear, although what in fact happens is that the turbulence increases with shear-rate. Thus, the increased shear-stress is absorbed in the creation of further vortices rather than

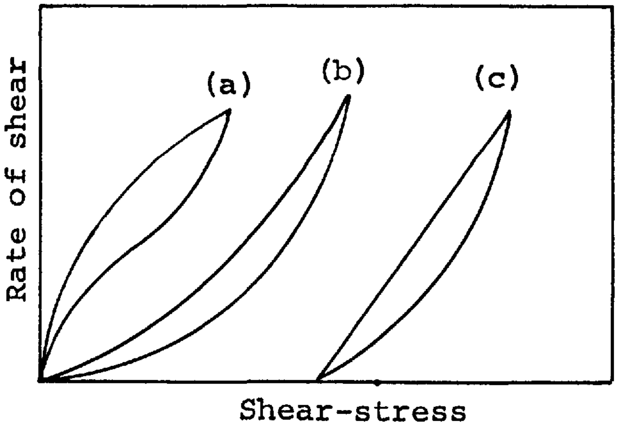


Fig.2.6

a) Thixotropic-dilatant flow
b) Thixotropic-pseudo-plastic flow
c) Thixotropic-plastic flow

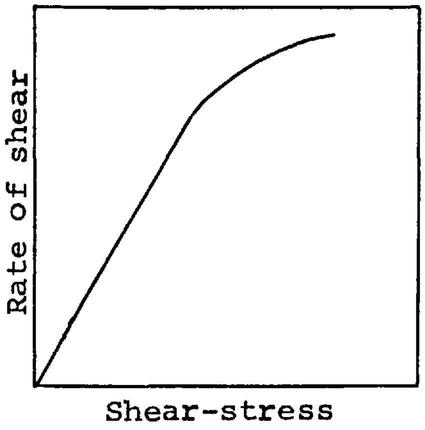


Fig.2.7
Turbulent flow.

in the flow of the bulk of the material.

In the analysis of flow curves, and the estimation of viscosity in unit operations, careful attention has, therefore, to be given to deciding whether anomalous behaviour is due to turbulence or not.

The Effect of Some of the Variables in Particulate Suspensions.

The flow of particulate suspensions is complicated, involving many variables. Considering the solid, there is volume concentration, density, particle size, size distribution, shape, extent of flocculation and lyophilic tendency. The fluid's intrinsic viscosity may vary according to temperature, pressure or even its previous history.

Many investigators have studied the effects of some of these variables and many formulae, mainly empirical, have been evolved to predict the rheological properties of suspensions. Some of these formulae and their validity are discussed below and the relative effects of several of the variables are examined.

The theoretical starting point was due to Einstein (32) who arrived at:

$$\eta_s = \eta_L(1 + 2.5\theta) \tag{2.3}$$

where η_s is the viscosity of the suspension, η_L is the viscosity of the liquid medium and θ is the volume concentration of solids.

The conditions for the validity of this equation are that the particles be rigid spheres, the suspension sufficiently dilute that no particle causes disturbance to the hydrodynamic flow of solvent past another particle, and the effects of gravitation, inertia and turbulence of the solvent negligible. This formula has been confirmed by many workers (33)(34). Although it covers only a narrow field of application, it nevertheless provides a basis for many further theoretical and empirical equations suggested for other conditions. Equations have been proposed for higher concentrations by Vand, Manley, Mooney and others; several are given below. The problem of the viscosity of concentrated suspensions even of spherical particles has not, however, been resolved theoretically, except under very limiting conditions.

$$\eta_s = \eta_L\frac{(1 + 0.5\theta)}{(1 - \theta)^2} \text{ - Einstein (35)} \tag{2.4}$$

$$\eta_s = \eta_L \frac{2.5\theta}{(1 - k\theta)} \quad \text{- Mooney}^{(36)} \qquad (2.5)$$

where k is a factor dependent on hydrodynamic interaction and crowding.

$$\eta_s = \eta_L (1 + 2.5\theta + 7.17\theta^2 + 16.2\theta^3) \text{ - Vand}^{(37)} \qquad (2.6)$$

$$\eta_s = \eta_L (1 + 2.5\theta + 10.05\theta^2) \text{ - Manley}^{(38)} \qquad (2.7)$$

$$\eta_s = \eta_L \left[(1 + \frac{2.5\theta}{2(1 - 1.35\theta)} \right]^2 \text{ - Eilers}^{(39)} \qquad (2.8)$$

$$\eta_s = \frac{K}{v} \quad \text{- Traxler}^{(40)} \qquad (2.9)$$

where K is a constant for the suspension and v is the average void diameter depending on the particle size, shape and uniformity of shape.

$$\eta_s = \eta_L \left[1 + \frac{k\theta}{(1 - S\theta)} \right] \quad \text{- Williams}^{(41)} \qquad (2.10)$$

Recent theoretical approaches have been made by Kynch $^{(42)}$ and Maude $^{(43)}$ who claim agreement with experimental values up to 25 vol.% and 28 vol.% concentration respectively.

$$\eta_s = \eta_L \left[1+2.5\theta . \frac{k^2}{4\pi\eta a} \frac{(a_2 + \frac{1}{25} a_3 k^2 a^2)(1+\beta\theta) - \frac{3}{5} . \beta\theta a_1}{a_o - k^2 a^2 a_1} \right]$$

Kynch$^{(42)}$ (2.11)

where $\underline{a_o}$, $\underline{a_1}$, $\underline{a_2}$, $\underline{a_3}$, $\underline{k}$ and $\underline{\beta}$ are constants for the suspension.

$$\eta_s = \eta_L \cdot \frac{1}{(1 - 2.5\theta)} \quad \text{- Maude}^{(43)} \qquad (2.12)$$

At low concentrations both of these formulae agree with the Einstein equation (Eqn. 2.3)

Rutgers (44) has made a comprehensive survey of equations for suspensions, empirical and theoretical, comparing their validity and range of application.

The diversity of the formulae suggests that not all the relevant factors have been considered, or that they apply only over a particular restricted range. Many are over-simplified, depending purely on solid concentration and the intrinsic viscosity of the medium. Factors such as roughness of particles, density of particles, degree of dispersion and shear-rate are all potential sources of error in viscosity measurements. Many of the formulae are empirical, applying only to spherical glass beads suspended in a liquid of equal density, under conditions similar to those specified for the initial Einstein equation.

Some workers have evolved formulae to take the shape of the particle into account. Jeffrey (45) investigated ellipsoids; his work was extended by Eisenschitz (45) and later by Guth(45). The basic assumption is that a particle traverses layers of liquid of different velocities and these tend to make the particle rotate. The result is that the particle precesses about an axis at right angles to the plane in which the maximum velocity gradient exists. The Eisenschitz (45) formula takes Brownian motion into account and is applicable to solutions of high polymers and long molecules:

$$\eta_{sp} = \frac{1.15}{\pi} \cdot \frac{f\theta}{\text{Log } 2f} \quad \text{for no Brownian motion} \qquad (2.13)$$

and $$\eta_{sp} = \frac{f^2\theta}{15(\text{Log } 2f - 3/2)} \quad \text{for strong Brownian motion}$$

where $\eta_{sp} = \frac{\eta_s - \eta_o}{\eta_o}$ and η_{sp} is the specific viscosity; $f = \frac{\text{major axis}}{\text{minor axis}}$; θ is the volume concentration.

The main application of these formulae is probably in the paint, paste and printing ink industries; the formulae might be useful for systems where all but one of the conditions are held constant, and it is required only to find the correct constants for the given conditions.

A method of representing the results obtained for both settling and non-settling suspensions used by some workers (46 to 50) is the fluidity equation for which constants are empirically evaluated.

$$F = \frac{1}{\eta} = (1 - \theta)F_o \qquad (2.14)$$

where F is the fluidity of the suspension, F_o is the fluidity of the liquid medium and θ is the volume concentration of solids.

$$\text{or} \quad F = (1 - \theta_{eff.})F_o$$

where $\theta_{eff.}$ is the effective volume concentration. At the maximum concentration θ is called θ_∞, then the above equation is expressed as

$$F = (1 - \theta/_{\theta_\infty})F_o ,$$

$$\text{where} \quad \theta_{eff.} = \theta/_{\theta_\infty}$$

$$\text{or} \quad \eta_{Sp} = \frac{\eta_s - \eta_L}{\eta_L} = \frac{\theta}{\theta_\infty - \theta} ,$$

$$\text{or} \quad \frac{\theta}{\eta_{Sp}} = \theta_\infty - \theta .$$

Thus, by plotting θ/η_{Sp} against θ, linear graphs have been obtained and the values of θ_∞ **have** been evaluated for the particular system.

Ting and Luebbers (46) conducted experiments with suspensions of spherical particles, and developed an empirical formula from the fluidity equation, taking into account the relative proportion of cubical to tetrahedral packing for a monodisperse system.

$$\eta_s = \frac{\eta_o}{1 - \dfrac{\theta}{0.46 - 1.58 \times 10^{-3} (\eta_o/_R)^{0.469}}} \qquad (2.15)$$

where R is the density ratio of solid to liquid.

Purohit and Roy (47) included a factor for particle size effects when they arrived at an empirical formula for a system of quartz grains in water.

$$\frac{\eta_s}{\eta_s - \eta_L} = 0.08 + \frac{0.8D^{-0.23}}{\theta}, \qquad (2.16)$$

where D is the average particle size. A further semi-empirical equation was developed by Sherman (51) (Equn.2.17). This made allowance for the effect exerted by particle size on pseudo-plastic suspensions at high rates of shear, and on Newtonian flow in moderately concentrated monodisperse systems. His work is applied to emulsions as well as to suspensions of solid particles because the surfaces are immobilized by adsorbed films.

$$\text{Log}\ \eta_{rel} = \frac{0.036\ D_m}{\sqrt[3]{(\theta_{max}/\theta) - 1}} - 0.15 \qquad (2.17)$$

where $\underline{\theta}$ is the concentration vol.%, $\underline{\theta_{max.}}$ is the optimum volume of disperse phase which can be incorporated into the system, $\underline{D_m}$ is the mean particle diameter and $\underline{\eta_{rel}}$, the relative viscosity (η_s/η_L).

With mineral suspensions where such a wide range of particle shapes, size distributions, density ratios, etc. is possible, in conjunction with varying liquid properties and indeterminate shear-rates, the application of such formulae is very limited.

The viscosity of finely subdivided suspensions may be influenced by the sub-sieve fractions. The term "colloidal" is strictly applicable to particles below 0.1μ in diameter; but even up to 10μ the particles may share some of the characteristics of colloids, being significantly influenced by surface forces. Colloids are conventionally divided into lyophobic and lyophilic. The latter usually consist of very small particles, often macromolecules which are wetted by the liquid and may join up in a network and form a gel or floc. The viscosity of lyophilic suspensions is usually appreciably raised by even small concentrations, and at high concentrations these solutions often often exhibit structural viscosity.

The flow of such suspensions may be represented by the empirical Ostwald (52) relation:-

$$\tau_w \propto E^n \text{ when } \tau_w > \tau_y \qquad (2.18)$$

where τ_w is the shear-stress at the wall, E is the shear-rate and n is the measure of deviation from Newtonian flow. For flow in a tube (52), for example, this relation takes the form:

$$\frac{D\Delta p}{4L} = k\left(\frac{8u}{D}\right)^n \qquad (2.19)$$

where u is the average velocity of flow through the tube (total volume/cross-section), k is a factor dependent on the frictional properties of the surfaces of the tube, D is the diameter of the tube, L is the length of the tube, Δp is the pressure drop and the exponent n is a measure of the deviation from Newtonian flow.

Lyophobic suspensions, on the other hand, contain particles which are solvent-hating, they are not dissolved by the liquid and it is possible to flocculate them by means of certain chemical reagents. The contribution to the viscosity made by dispersed particles is, as a consequence, by hydrodynamic and physical forces; and small concentrations of solid scarcely increase the viscosity of the suspension above that of the liquid. The deciding factor between the two types of colloidal suspensions is the nature of the liquid-solid interface. Any formula which predicts the flow of a suspension, therefore, should take into account the degree of attraction between disperse phase and medium: depending on the particle size, the Brownian motion may need to be considered in theoretical treatments (53)(54).

As the present project is concerned mainly with the grinding of mineral ores, relatively coarse suspensions will generally be considered, where surface forces are not important. It is clear, however, that this will not apply to materials such as clay slurries and suspensions contaminated with "fines", for surface forces have a significant influence on the mechanics of particles of diameter below 10μ.(55).

The most important variable in the flow of particulate suspensions is probably the concentration of solids. The extent of its effects, however, is dependent on several other factors. Some typical results, obtained by Schack, Dean and Molloy (56), are illustrated in Fig.2.8. Certain workers quote a "critical concentration" below which the viscosity is affected relatively little and above which it rises rather sharply. The critical concentration for uniform, roughly spherical particles is about 40% by weight for minerals of s.g. about 2.5, if the minerals have an approximate size range of 10μ to 60μ.

A type of flow that is greatly affected by concentration is dilatancy, as it depends on the mutual interference of the particles. Below a certain percentage of solids the particles are free and do not greatly interfere, even when the suspension is subjected to a shear-stress; but when the particles begin to touch each other, flow is immediately restricted when a shear-stress is applied (57). The "critical concentration" can be estimated from geometric consideration of the relative volumes of voids and particles in close packing (58).

DeVaney and Shelton (59) showed experimentally that the critical concentration with various minerals was at

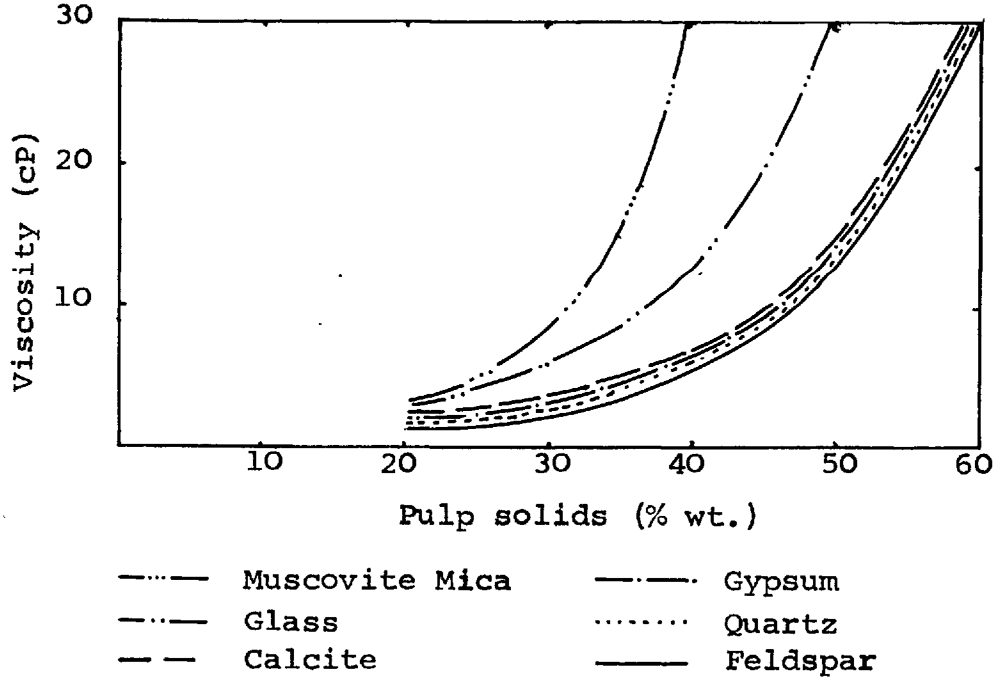


Fig.2.8 - Schack, Dean and Molloy

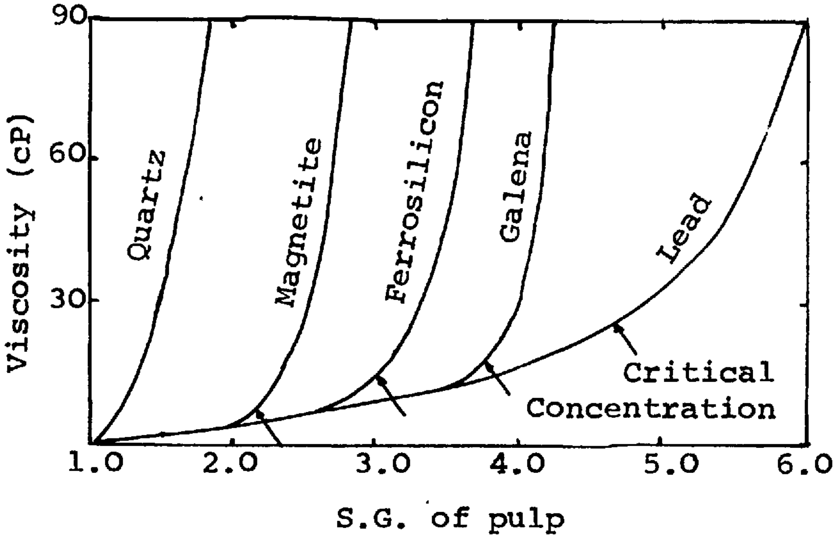


Fig.2.9 - DeVaney and Shelton

Increase in viscosity with
concentration of solids.

about 25% solids by volume and zero mobility was at about 44%, using suspensions of particles at - 200 + 325 mesh as shown in Fig.2.9. No density effect on the critical concentration can be detected from these results: i.e. the viscosity appears independent of the density of the particles. Several workers (46)(49)(50) have obtained similar graphs to that of DeVaney and Shelton showing this critical concentration with linear regions above and below it.

In general, all workers agree that with increasing concentration the viscosity increases: the extent, however, is dependent on many other factors such as particle shape and size, degree of dispersion, etc.. The effect of these various other factors will, therefore, be discussed separately.

Theoretical considerations of particulate suspensions largely ignore the effect of particle size. Under closely defined conditions this assumption can be justified (Fig.2.10).

Investigations were made by Schack, Dean and Molloy (56) using an efflux viscometer with ground quartz in distilled water. They showed that the effect of particle size on viscosity can be quite considerable, depending on the concentration of solids (Fig.2.10). However, the effect

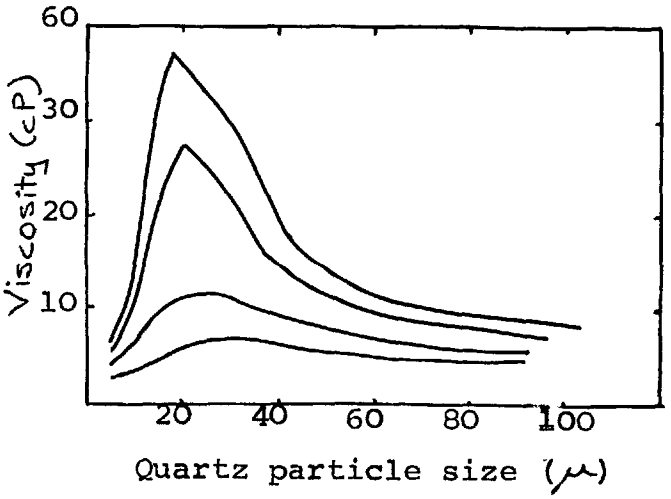
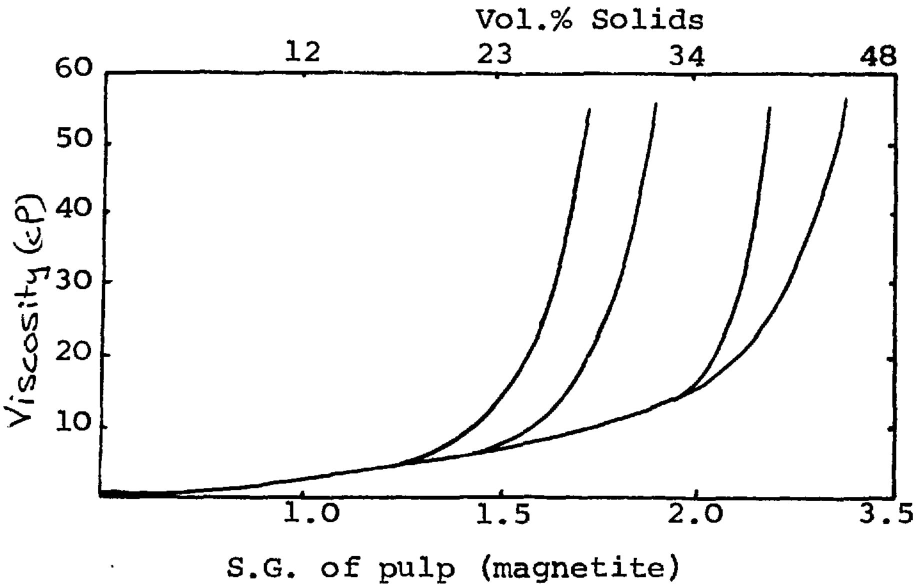


Fig.2.10 - Schack, Dean and Molloy



Average particle size

A=15.8 μ B=25.7 μ

C=37.6 μ D=51.7 μ

Fig.2.11 - DeVaney and Shelton.

of particle size below concentrations of about 45 wt.% for quartz, is small. The viscosity at higher concentrations was found to rise sharply with particle size up to 20μ, and thereafter the viscosity declined to a nearly constant value for particles of around 100μ. The peak viscosity at 20μ for 75 wt.% solids was 200 cP, and yet at 85 wt.% solids the suspension with particles of 4μ was still highly fluid. The results of DeVaney and Shelton (59) illustrated in Fig.2.11 show a more consistent increase in viscosity with decreasing particle size. Their experiments were conducted with magnetite (from 51.7 to 15.8μ) in water.

In an attempt to repeat the above-mentioned work by Schack and co-workers, Pakianathan (60) obtained a graph as shown in Fig.2.12. There is agreement over the larger particle size range; but he obtained a further increase in viscosity as the particles became smaller for 50 wt.% solids.

It is likely that results obtained with efflux viscometers are unreliable at high concentrations of large particles. The tendency for plug flow to occur increases with particle size, because of the enhanced forces of mechanical interaction which prevent shear from taking place in the main body of the fluid. Experiments on the transport

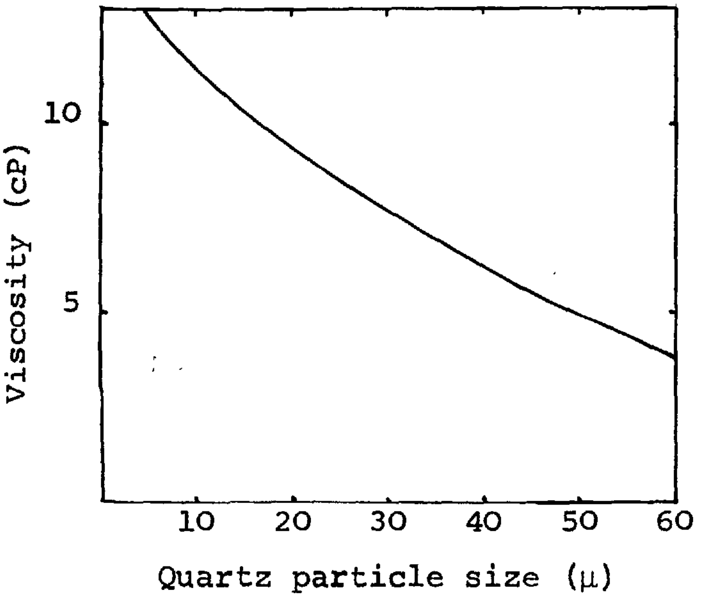


Fig.2.12 - Pakianathan

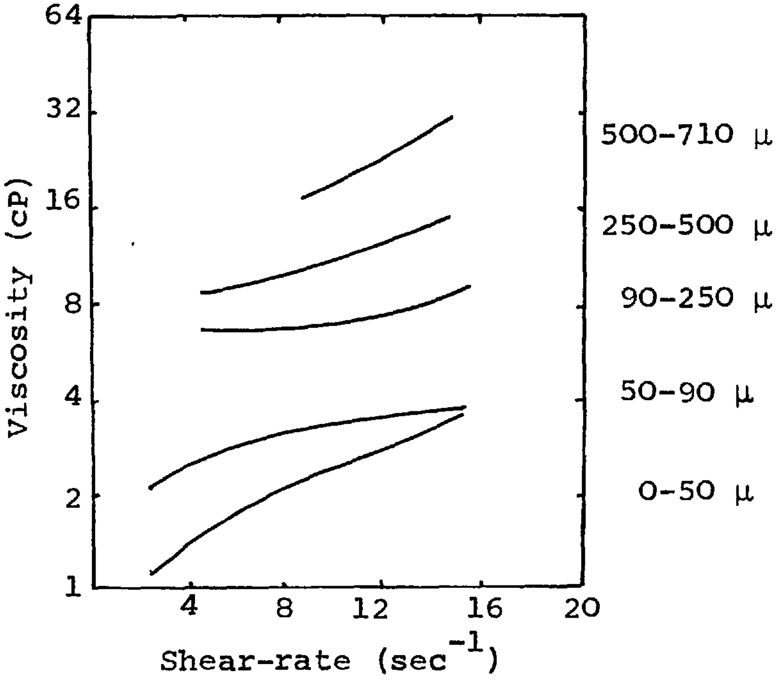


Fig.2.13 - De Bruijn and Meerman

Effect of particle size on viscosity.

of solids (61) in a liquid flowing through a pipe, have shown the tendency for the solids to travel along the centre of the tube. If this phenomenon takes place in efflux viscometers then homogeneity of the sample is lost and shear takes place mainly in the outer fluid layers. Williams(62) investigated a lower size range of silica, at 10 vol.%, in a linseed stand-oil and paraffin mixture. He found that, for particle sizes of about 1μ and smaller, as the size decreased the viscosity increased and became non-Newtonian. With particles of 0.025μ the suspension was thixotropic and possessed a yield value even at low concentrations. Suspensions of glass spheres in organic liquids, tested by Broughton and Windebank (63), behaved as Newtonian liquids when the particle size exceeded about 30μ. Below this value some anomalous behaviour was observed which they attributed to flocculation of the spheres in the suspending fluid.

Some experiments were conducted by Eveson, Ward and Whitmore (64) with different relative percentages of coarse and fine particles. The suspensions were made up of methylmethacrylate polymer spheres (-72 +100 mesh and -300 mesh) in solutions of aqueous lead nitrate and glycerol. They found that, by increasing the proportion of coarse particles, the relative viscosity decreased to about 50% of the original value. Further increases in the coarse fraction, however, caused a slight increase in the viscosity.

Eveson (65), carrying out further tests, found that, with small spheres at low rates of shear, the viscosity was independent of particle size above 15μ, while below 15μ the viscosity increased with decreasing size. The same decrease in viscosity with particle size was obtained by Sweeny and Geckler (66), but for a greater size range (viz. 10 - 400μ), at various shear-rates and concentrations. They obtained different results, however, with non-aqueous solutions. This was ascribed to the differences in adsorption properties. Ward and Whitmore (67) carried out some tests on the influence of particle size on viscosity using a rising sphere viscometer. They also found a decrease in viscosity with increasing particle size over the range 38 - 279μ with rough methyl-methacrylate spheres, suspended in aqueous liquid of equal density. However, when smooth glass spheres were used under similar conditions, the viscosity of the suspension was found to be independent of the particle size (from 38 to 165μ) (68). The reason put forward was that an envelope of immobile liquid, the thickness of which was independent of particle size, was occluded by the rough spheres, with the result that the effective volume increased as the particle size diminished. The results quoted so far show that either the viscosity is independent of particle size, or that there is an increase in viscosity with decreasing particle size (51)(69)(70). In contradiction to these results, De Bruijn and Meerman (71) found an increase in viscosity with increasing particle size (Fig.2.13). They used settling suspensions of glass spheres (50 - 700μ at 13.8 vol.%) in a rotational viscometer with a medium of ammonia solution (viscosity 1 cP). It can be seen that the suspensions that they used were also slightly dilatant in nature. Similar increases in viscosity with particle size were obtained by Purohit and Roy (47). They also used settling suspensions in a rotational viscometer. The suspensions were made up of quartz and haematite (23 - 137μ) in water. They obtained, however, a decrease in viscosity with increasing particle size when using coal (from 23 - 314μ) under similar conditions. The reasons suggested for this phenomenon were the differences in wetting characteristics and the greater angularity of quartz and haematite of large particle size. Suspensions of coal in mineral oil were studied by Moreland (49), who obtained similar reductions in viscosity with particle size (from 80 to 170 mesh).

In conclusion, there seems to be little agreement between the results of the workers quoted, particularly with particle sizes above 15μ. Below 15μ the viscosity generally seems to increase with decreasing particle size, and particularly at the higher concentrations. This is presumably because of surface chemical forces. Possible sources of discrepancy above 15μ are differences in the types of viscometer used, the ratio of density of solid to liquid, and the size ratio of the largest to the smallest particles. There is clearly a need for further work on this aspect of viscosity of suspensions.

The effect of shape and surface of the particles, on the flow properties of suspensions, has been considered theoretically for ideally shaped particles of rods and plates: but the formulae, even for these ideal shapes, do not cover the higher concentration range typical of mineral treatment operations.

Practical investigations have been made by DeVaney and Shelton (59) with smooth or angular particles of the same material suspended in an aqueous medium (Fig.2.14). The greater viscosity with rough particles is clearly

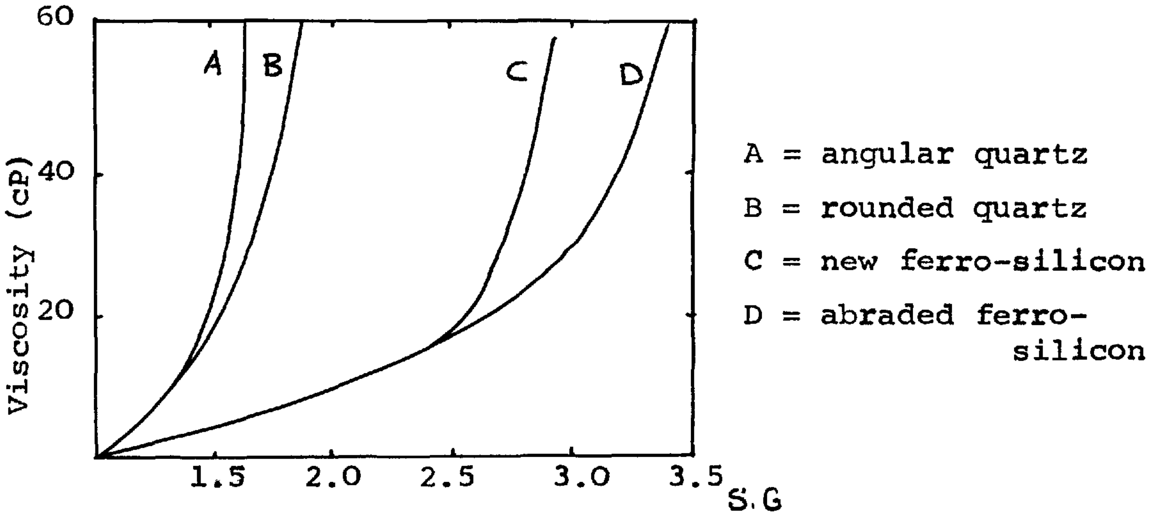


Fig.2.14 - DeVaney and Shelton

Effect of particle shape on viscosity

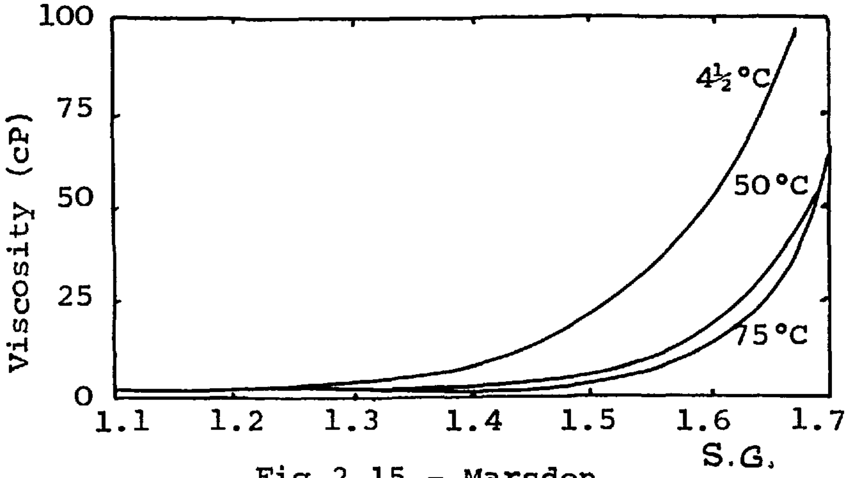


Fig.2.15 - Marsden

Effect of temperature on viscosity of pulp illustrated. The reason suggested is that the rough particles get interlocked and wedged. Below a certain concentration the particles do not interfere and there is no difference in the viscosity. Moreland (49) obtained up to 50% reduction in the viscosity of suspensions of rounded rather than freshly ground grains of coal in mineral oil ($\rho = 0.847$, η = 18 cP) at high concentrations (>50 wt.%).

Schack, Dean and Molloy (56) studied the effect of particle shape with six different minerals, two of which (mica and gypsum) were more plate-like and angular than the glass, quartz, calcite and feldspar. Referring to Fig.2.8, it can be seen that the critical concentration is reached at lower values for the two angular minerals rather than with rounded grains. Mica, being the more angular, showed the effect to a greater extent than gypsum. Schack and co-workers also concluded that the crystallinity, hardness and solubility of the minerals that they used had no observable effect on the viscosity.

Relating surface characteristics to particle size, Whitmore (72) stated that the viscosity of a suspension of hard spherical particles increases as the particle size decreases, but only when the surfaces are rough or the particles are twinned or distorted when the effective hydrodynamic volume of a given quantity of solids tends to increase with diminishing particle size.

The influence of temperature on the viscosity of suspensions appears to depend mainly on the medium alone: the viscosity of a pure liquid is well known (73) to be reduced by rise in temperature generally according to the equation:

$$\eta = \eta_o e^{E/RT} \quad \begin{pmatrix} R = \text{Gas constant} \\ T = \text{Abs. temp.} \end{pmatrix} \quad (2.20)$$

where E is the so-called energy of activation for viscous flow. Tests by DeVaney and Shelton confirmed that the effect of temperature on an aqueous slurry with -200 +325 mesh ferrosilicon was only slight, the viscosity decreased with increasing temperature, although the effect was observed to be greater for pulps containing more than 36 vol.% solids. The results of some experiments by Marsden (74) on the effect of temperature on the viscosity of some South African gold mine slurries are shown in Fig. 2.15. The influence of a change of 25°C in the temperature is seen to cause about 15% drop in viscosity, while a 70°C increase caused drops from about 75 cP to 25 cP. Marsden's results suggest that there may be other factors in operation besides the influence of the temperature on the intrinsic viscosity of the medium. A suggested reason for these results is that the change in temperature produced a change in the volume of the medium. this altered the concentration of solids, the effect of which has been discussed.

In suspensions containing particles smaller than 10μ, the viscosity is influenced by electrical double-layer effects (the electro-viscous effect). When a suspension is subjected to shear, the ionic atmosphere is deformed, giving rise to a dissipation in energy within the fluid which increases the viscosity. Von Smoluchowski (75) extended the Einstein formula to allow for this electro-viscous effect, the resulting expression being:-

$$\eta = \eta_o \left\{ 1 + 2.5\theta \left[1 + \frac{1}{\lambda \eta_o a^2} \left(\frac{\zeta \epsilon}{2\pi} \right)^2 \right] \right\} \qquad (2.21)$$

where η_o is the viscosity of the solvent, ϵ is the dialectric constant, λ is the specific conductivity of the suspension, a is the radius of the particles, ζ is the zeta potential and θ is the vol.% concentration of solid particles. This equation, like that of Einstein, assumes no interaction between the particles and is, therefore, applicable only to dilute suspensions. The conditions are that the particles must be spherical, insulating, insoluble, unable to swell, stable in the presence of electrolytes and over a period of time, with a radius of less than 5 x 10^{-6} cm.; the thickness of the electrical double layer must be very much less than the radius of the particle, and ions or charges in the surface must be immobile. These and other conditions render practical checking of the theory difficult. Experiments by Bull (76) using ovalbumin, and Briggs (77) using gum arabic, sodium caseinate and β-lactoglobin, showed that the electroviscous effects predicted by the theory were much greater than those found experimentally; but these macro-molecular substances are unsuitable models, as their molecular configuration can change. Further work by Dobry (78), however, which approximated more closely to the given conditions with silicon carbide particles, still showed the electro-viscous effect to be less than that predicted by Equation (2.21).

It has been found (79) that the electro-viscous effect is more prominent when the particles are very small, such that the thickness of the double layer is of the same order as the radius of the particles, and when the concentration is such that the double-layer causes interference of the particles. In theory, the effect increases with decrease in ionic strength, since under these conditions the effective thickness of the double layer increases. Elton (80) considered the electro-viscous effect under conditions of higher concentrations of microscopic particles. He calculated the apparent viscosity (η_a) of a suspension of spherical particles of radius a (Equation 2.22):

$$\eta_a = \eta + \frac{16 c_1 \epsilon k T a^3 n}{3 \pi \lambda} \cdot \sinh^2 \frac{e \zeta}{2kT} \tag{2.22}$$

which at small values of ζ reduces to:

$$\eta_a = \eta + \frac{\epsilon^2 \zeta^2 a^3 n}{6 \pi \lambda K^2} \tag{2.23}$$

where T is the temperature, c_1 is the ionic concentration, n is the number of particles per c.c., k is Boltzmann's constant, K is the reciprocal of the thickness of the double layer, other symbols having their previous meaning. Use of these equations to obtain rates of sedimentation of a colloidal carborundum in solutions of various ionic strengths gave moderate agreement with experiment.

In the field of mineral dressing, electro-viscous effects are of limited interest because the average particle size is much greater than that concerned in the electro-viscous effect. The small percentage of fines produced in mineral treatment operations may fall in the electro-viscous range, however, and hence contribute significantly to the overall viscosity of the pulp.

The influence of pH on the viscosity of the pulp was the subject of some experiments conducted by Marsden (74) on gold mine slurries (Figs. 2.16 and 2.17): the influence was negligible with slurries of densities less than 1.4. Above this, the critical concentration of solids occurred at a higher specific gravity for increasing acidity, such that at a density of 1.65, an increase from pH2 to pH6 caused an increase of about 100% in the viscosity. The pH-viscosity relationship is shown in Fig. 2.17. The pH value made relatively little difference up to pH7, whereas further increases caused quite sharp increases in viscosity. An explanation of this phenomenon is that the viscosity depends on the degree of dispersion of the particles; as the pulp became more acid so the finer particles ($< 1\mu$) became flocculated. The new flocs formed could have behaved as fewer, larger particles, causing a decrease in viscosity due to particle size effects.

Dispersing agents are often used to maintain

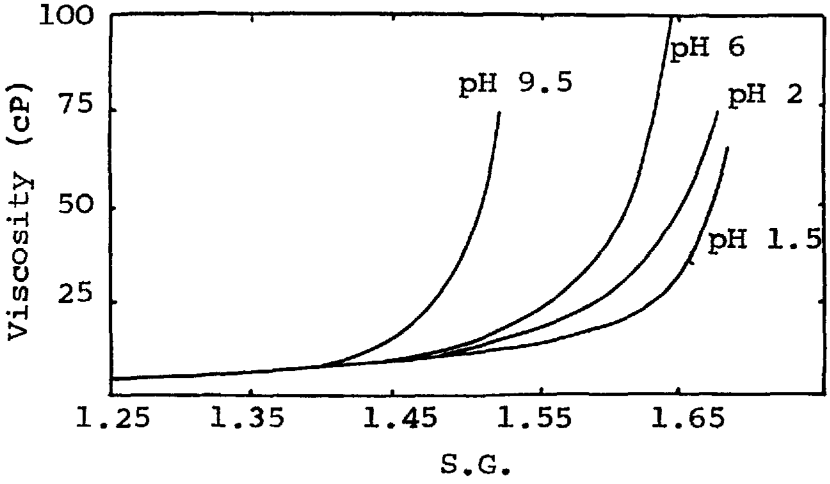


Fig.2.16 - Marsden

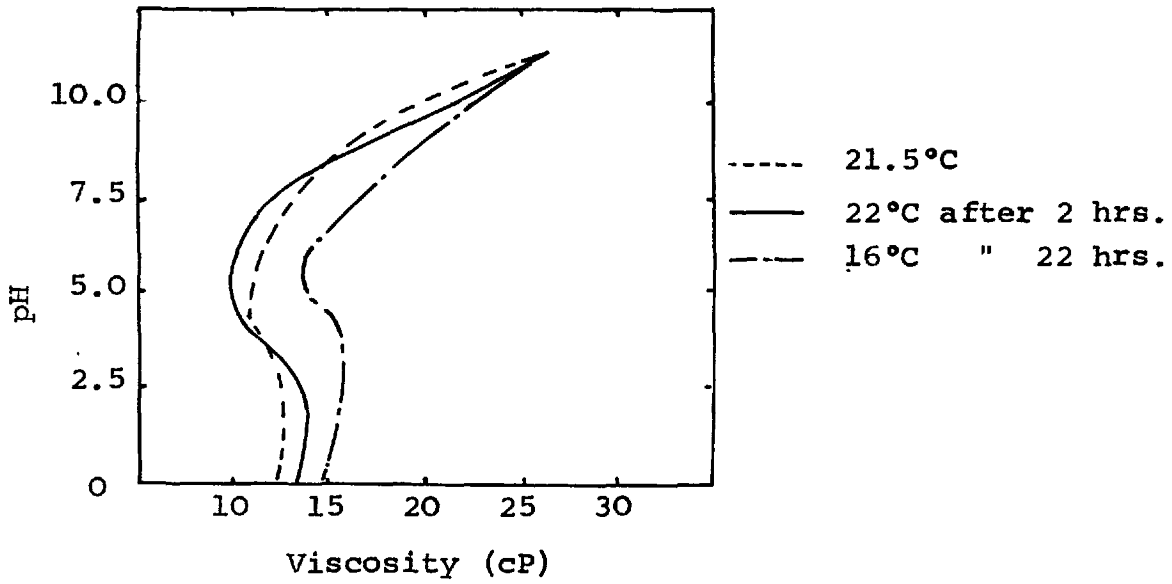


Fig.2.17 - Marsden

Effect of pH on viscosity of pulp.

separation between particles, by offsetting the forces causing them to form a connected structure or agglomerate. The agents adsorb onto the surface of the particles, causing the formation of an electrical double-layer with ionic agents or a solvation layer with non-ionic agents. When a double-layer has been established the particles repel each other, repulsion being greater the more strongly the particles are charged and the thicker the diffuse double-layer (81)(82). Some investigators, therefore, when wishing to study the purely hydrodynamic or mechanical interaction effects, add a certain amount of dispersant in order to avoid the formation of flocs.

The effect of dispersants and flocculants is, of course, greater the smaller the particles: for large particles gravitational and inertial forces are predominant.

The addition of a flocculant encourages the formation of aggregates or a connected structure. Under certain circumstances, a flocculant may induce thixotropy in a previously Newtonian or dilatant material. It is generally considered that flocculation is necessary for thixotropy, although the reverse is not necessarily true (83). A flocculant may cause particle size effects by the production of aggregates, the effective particle size being the size of the group.

If a weakly connected structure is formed which is broken down on flow, the continuous breaking down and reforming contributes to the shear force, resulting in plasticity, pseudo-plasticity or thixotropy. In plastic flow, the yield value is sometimes taken to be a measure of the force of flocculation per unit area which exists between the suspended particles (84).

Ageing can cause changes in the viscosity of a suspension; the rate of flocculation arising from surface sorption changes is one of the reasons suggested for this effect(85).

Most operations involving pulps, slurries or suspended media are concerned with a wide range of particle sizes from the sub-sieve fractions up to the initial feed size. Schack, Dean and Molloy (56) investigated a range of size distributions of mineral grains in water. They found that the viscosity was merely intermediate between the maximum and the minimum values for the individual fractions composing the mixture. On the other hand, using varying percentages of 60μ and smaller fractions of quartz, Pakianathan (60) found that the viscosity was greater the higher the proportion of fine to coarse particles and the smaller the fine fraction (i.e. the wider the size range) (Fig.2.18). The viscosity was also found to be more dependent on the size distribution at concentrations of over 20% by volume. The increase in viscosity when equal proportions of the mixture were used is, perhaps, surprising.

By increasing the percentage of fine particles, Eveson, Ward and Whitmore (64) found that they could induce a pseudo-plastic effect. They found that, with increasing coarse fraction, the viscosity decreased to a minimum value at equal percentages and then increased somewhat at all concentrations over 7.5 vol.% (Fig.2.19).

Significant reductions in viscosity were obtained by Moreland (61) by taking 60 - 70% of the coarse fraction and 30 - 40% of the finer with a size ratio of 10:1. He obtained these results using settling suspensions of coal grains in mineral oil with a rotational viscometer.

Ward and Whitmore (68), using non-settling suspensions in a rising sphere viscometer, found that the viscosity decreased with increasing size-range to a constant value. The tests were carried out using size-

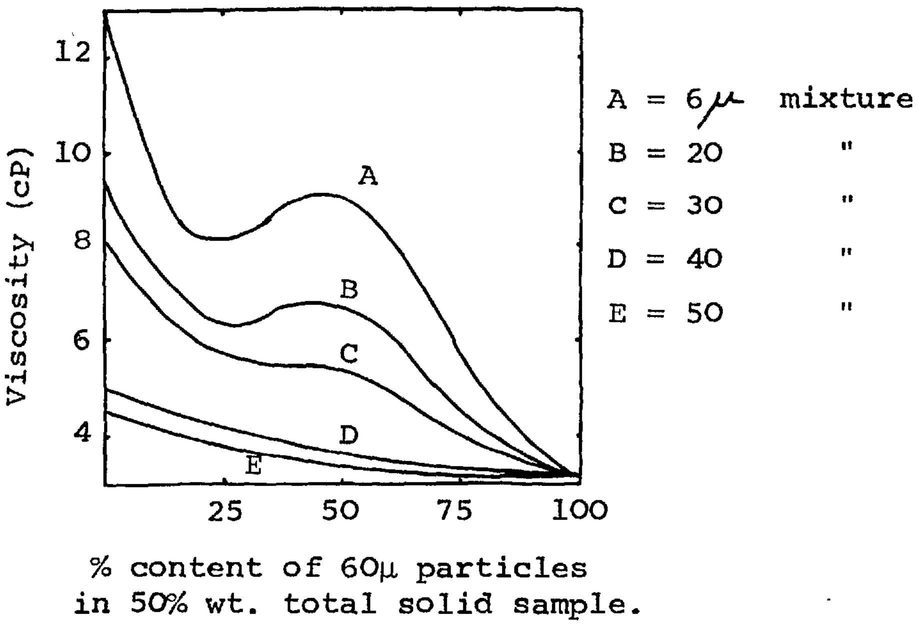


Fig.2.18 - Pakianathan.

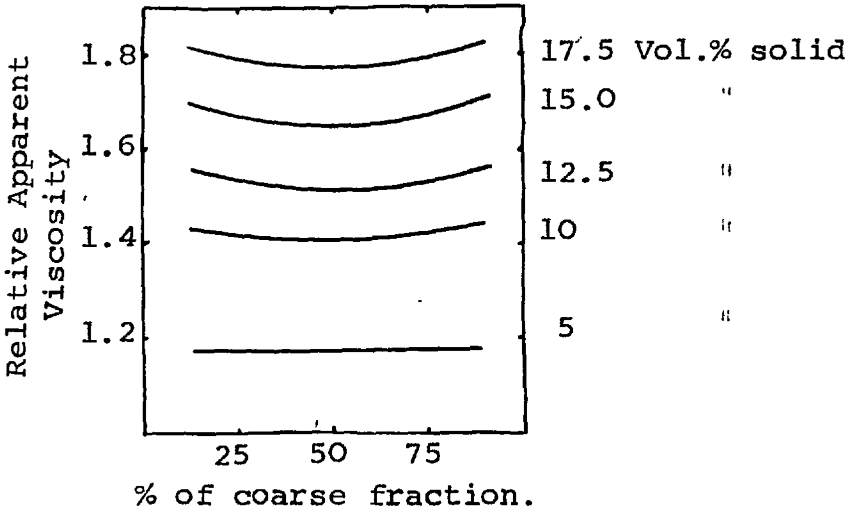


Fig.2.19 - Eveson, Ward and Whitmore.

distributions (flat-topped) of methyl-methacrylate spheres ranging from 152 - 177μ to 76 - 208μ (Fig.2.20).

In general, it appears that when the smaller fraction can circulate freely in the voids between the larger, then significant reductions in viscosity will result; the extent, of course, depends on the relative percentage of each fraction and also the difference in the average particle size.

If a very wide size range is used, then the very fine particles will be subject to colloidal effects, and small percentages of material can cause anomalies or high viscosity, e.g. with bentonite. The coarse material would then behave as though suspended in a viscous fluid, and packing and interaction between large and small particles would be negligible.

This aspect of the viscosity is closely allied to the particle size and surface area effect. For a constant volume, smaller particles have a greater surface area. The presence of fines in a suspension would, therefore, increase the effective surface area as well as the size - distribution - interaction effect. The increase in surface area was one of the variables studied by Salmon and Sirois (86) when

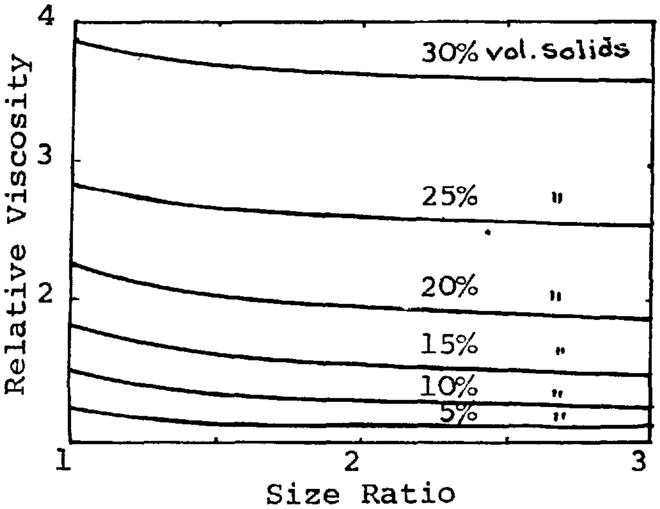


Fig.2.20 - Ward and Whitmore

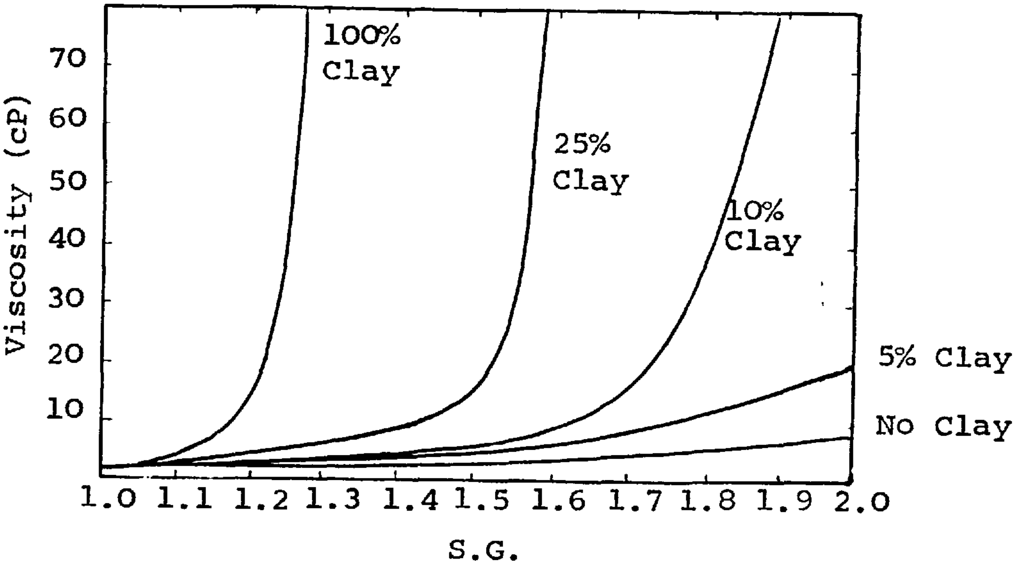


Fig.2.21 - Geer, Sokaski, West and Yancey.

grinding quartz in a ball-mill. They found that for pulp densities over 45 wt.%, small increases in the surface area caused large increases in the viscosity. For example, an increase in surface area per 100 gm from about 7,000 to 37,000 sq.cm. caused an increase in viscosity from 2.5 to 205 cP at 70 wt.% solids.

Some experiments were conducted by Geer, Sokaski, West and Yancey (87) in order to discover the effect of clay contamination on the viscosity of mineral ore suspensions, using a DeVaney-Shelton viscometer. Their results, showing the effect of differing percentages of clay and magnetite are shown in Fig.2.21. It can be seen that a 5% fraction of the fine clay made an appreciable difference to the viscosity, while 10% clay made a great difference, beyond specific gravities of 1.7. The effect of a bentonitic clay, however, must not be too closely approximated to that of fine particles of the material itself, because of variations in colloidal and electroviscous properties.

The effect of shear-rate is probably the most neglected variable in the study of suspensions. The reason for this is mainly because of the use of capillary and efflux viscometers which rarely make provision for this measurement.

The results of different workers vary. Moreland (49) found settling suspensions of coal or haematite in mineral oil (of 18 cP) to be pseudo-plastic above 25 vol.%, with a rotational viscometer. On the other hand, De Bruijn and Meerman (71) obtained results showing slight dilatancy, using a rotational viscometer with settling glass spheres in an ammonia solution (of 1 cP) (Fig.2.13). With non-settling suspensions of hard, smooth, non-interacting spheres, Ward and Whitmore (68) and others (88)(89) claimed that the viscosity is practically independent of the rate of shear up to 30 vol.% concentration. The latter result was confirmed by Metzner and Whitlock (90). They observed no shear-rate dependence with glass beads (up to 100μ) suspended in sucrose solutions (from 17 cP to 42 cP), up to 50 vol.% concentration. They did, however, observe "drying" of the surface of the suspensions under shear. This suggests the possibility of dilatancy which was obscured by slip at the surfaces of the cylinders of the rotational viscometer. These investigations did reveal marked dilatancy, however, with titanium dioxide particles (size 0.2 - 1μ) in water and sucrose solutions.

Salmon and Sirois (86) measured some ball-mill pulps of fine particles of quartz and water at varying concentrations with a rotary viscometer. All the pulps were found to be dilatant by nature. The dilatancy of sand and water at high concentrations is well known, although there is very little published work on the lower limit of this effect. Freundlich and Röder (57) observed dilatancy of quartz (1.5 - 5μ) and water only between 42 and 45 vol.% solids, which is surprising. In view of the lack of agreement over the effect of shear-rate on fine suspensions, and the almost total absence of publications on its influence on coarse, fast-settling suspensions, this is clearly a topic worthy of further investigation.

The effect of turbulence on the viscosity of mineral suspensions was studied by Van de Walt and Fourie.(91) Tests were conducted using 22 mesh shale contaminated with fine coal from a coal bath separator (curve A), Baum jig refuse ground to -200 mesh (curve B), and -200 mesh magnetite (curve C), each suspended in water. As the rate of shear increased, the degree of turbulence increased with a result similar to dilatant flow (Fig. 2.22). The need for care when interpreting results at high shear-rates is clearly indicated. The dotted lines show where the effect of

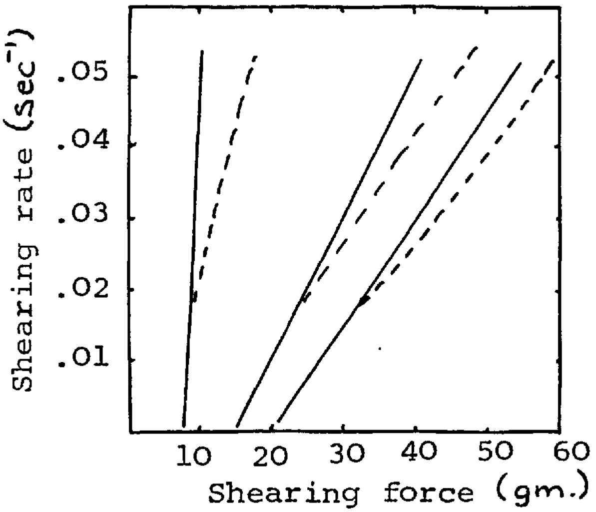


Fig.2.22 - Van der Walt and Fourie

Effect of turbulence on viscosity.

turbulence increased the necessary shear-force from that which would be normally required if the flow was laminar.

In general, the results obtained by the different workers on the viscosity of mineral suspensions show a high degree of discrepancy. In fact the influence of none of the variables is completely clear-cut and well established. The need of a further investigation into the relative importance of the different variables is clear, and particularly with coarse, settling suspensions. The following chapters describe the details of such an investigation and associated apparatus.

Chapter 3.

DESIGN AND DEVELOPMENT OF A VISCOMETER FOR MINERAL SUSPENSIONS.

Choice of Instrument.

There are two main types of viscometers which have been used for rapidly-settling suspensions, namely, the efflux type and the concentric cylinder type. The efflux viscometer gives a measure of the viscosity proportional to the time of efflux of a specific volume of the suspension. A stirred reservoir is often used to maintain homogeneity of the sample before it flows out through a narrow tube or orifice. This type of instrument was used by DeVaney and Shelton (59), Schack and co-workers (56) and Pakianathan (60). The main disadvantages of the efflux viscometer are, firstly, that, although the shear-rate may be varied by pressure, it is indeterminate and, secondly, at high concentrations wall corrections are probably unreliable and plug flow predominates.

The concentric cylinder viscometer follows more closely the theoretical ideal of laminar flow between two parallel plates. The cylinders are immersed in a vessel containing the sample, and one of the cylinders is rotated while the other remains stationary. The torque is usually transmitted to the stationary cylinder which acts against a calibrated spring or torsion wire, the deflection giving a measure of the shear-stress of the sample. The main problem with this type of viscometer is to keep the sample homogeneous if the particles are fast-settling. Eveson (92) designed a concentric cylinder viscometer which possessed an external diaphragm pump. The sample, including settled particles, were circulated from the bottom of the container and reintroduced at the top of the cylinders. Thus, the particles could settle freely, while undergoing shear. This type would not, however, handle coarse, granular, high-concentration solids suspensions typical of ball-mill pulps. Van de Walt and Fourie (91) designed a rotational viscometer for coarse suspensions, but shear took place between the inner cylinder wall and baffles. This is a departure from the ideal concept of flow between parallel plates. They also used a conical bob which might lead to non-homogeneity, if the solids settled at a faster rate than the circulation of the fluid.

Another type of rotational viscometer was developed by Bhattacharya and Roy (93), which was later used by

Purohit and Roy (47). The stirring mechanism consisted of two paddles attached to the inner cylinder, which stirred up the solids when rotated at high speeds (up to 1000 r.p.m.). Disadvantages of this type are the dangers of turbulence at high speed; the contribution to shear-stress of the rotating paddle and the problem of the rotating <u>inner</u> cylinder which can cause instability (see below).

It is claimed (94) that, even with pure liquids, instability sets in at lower Reynolds numbers, if the outer cylinder is stationary, owing to centrifugal forces. Taylor (95) was the first to observe the appearance of circular flow patterns (now known as "Taylor vortices") in sections of the annulus. Schlichting (96) found that the rotating outer cylinder had a stabilizing effect on settling suspensions. Movement of particles outwards is restricted because centrifugal forces acting on particles in inner layers are smaller than for those further away from the axis. Critical Reynolds numbers of 12,500, 19,300 and 50,000 are quoted by Taylor (95), Schlichting (96) and Ball and Colwell (quoted by Van Wazer, Lyons, Kim and Colwell (94)) respectively. Further, Squires (quoted by Schlichting (97)) showed that if the flow is three-dimensional, then the system is further stabilized.

A source of error which many designers overlook is slip at the walls of the cylinders if these are smooth. The fault can be prevented by suitable grooving (98).

Constructional Details of the Viscometer.

A survey of the commercially available viscometers revealed a lack of any that would deal with the sort of suspensions encountered in ball-milling. As a potential solution of the problem a Ferranti portable viscometer (model VL) with louvres was obtained (Fig.3.1). It was designed for use with fine suspensions and was acquired with the intention of modifying it as necessary.

The viscometer was of the concentric cylinder type, with a rotating outer cylinder and eight sets of interchangeable cylinders. Five speeds provided a wide range of shear-rates from 43 to 950 sec^{-1} approximately. The speeds could be changed instantly for the determination of flow curves. The viscometer carried a three-bladed impeller inside the top of the outer cylinder, and three holes in the top of the sides of the outer cylinder. Hence, the sample was circulated upwards through the annulus and out at the top, as the outer cylinder rotated. However, this circulation mechanism was not powerful enough to deal with

	KEY
No.	Item
1	Outer cylinder
2	Inner cylinder
3	Grooved surfaces
4	Cylinder support strut
5	Inner cylinder spindle
6	Outer cylinder quick release attachment
7	Inner cylinder release aperture
8/9	Inner cylinder quick release system
10	Steady bearing
11	Turret gear box
12	Inner cylinder locking mechanism
13	Spring loaded secondary bearing
14	Synchronous motor
15	Phasing condenser
16	Transformer
17	Main jewelled bearing
18	Torque spring
19	Viscosity indicating pointer

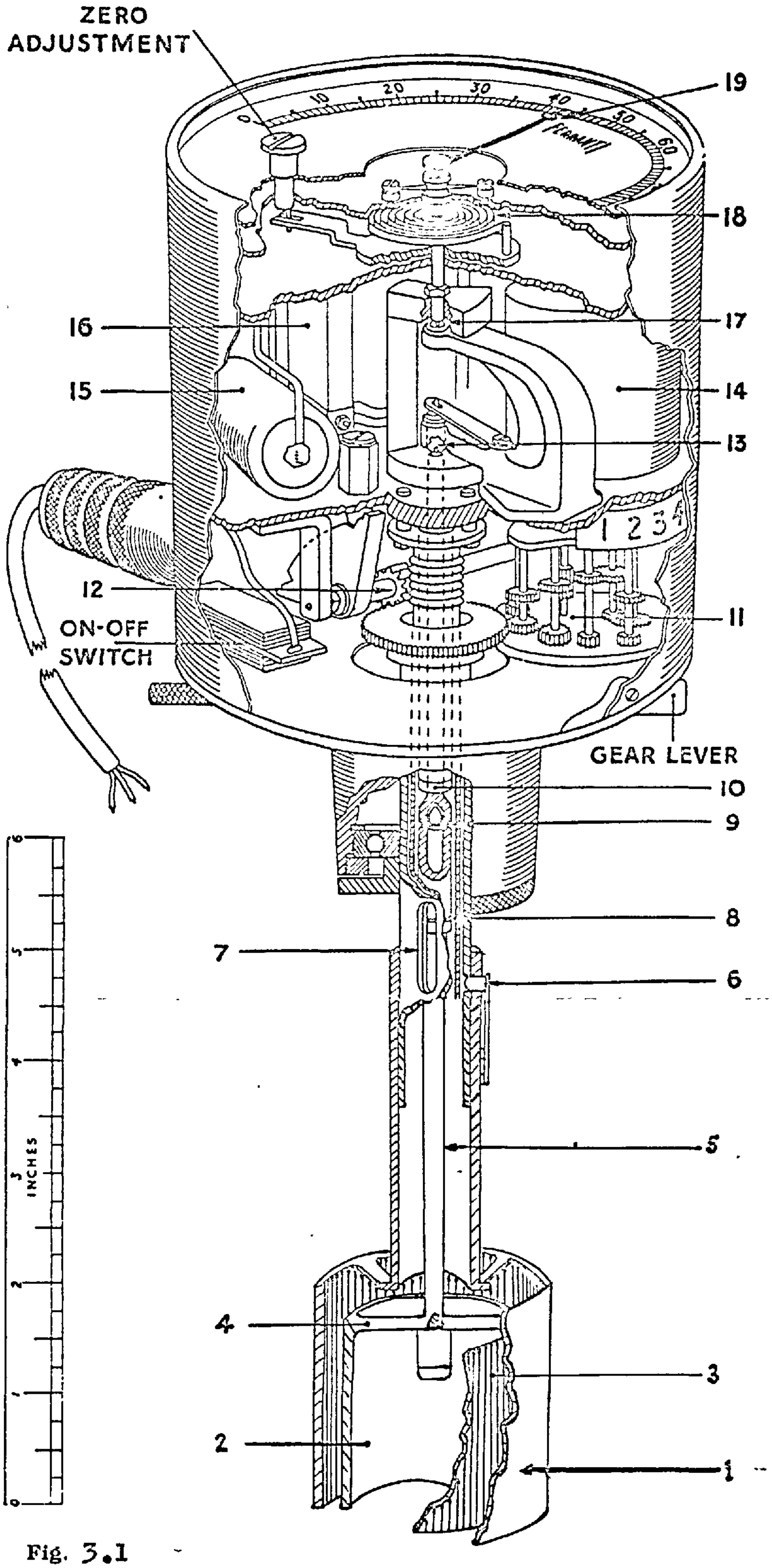


Fig. 3.1

Modified Ferranti Viscometer

suspensions containing coarse, mineral grains. The first stage of modification was to provide a special, glass-sided sample cup. A four-bladed, variable-speed impeller was introduced through a hole in the base of the cup via a water-tight gland. The impeller rotated at the bottom of the cup beneath four horizontal baffles; these, and four vertical baffles at the sides of the cup, were fitted to minimise rotation of the pulp, and to encourage vertical circulation of the sample throughout the measuring compartment. Experiments were carried out to find the best shape and number of baffles. The final design of sample cup is shown in Fig.3.2. A circular, flat plate was fitted to the top of the inner section of the blades of the impeller, also shown in Fig.3.2. This was done in order to prevent the stirrer from causing excessive deflection of the inner cylinder of the viscometer. The plate was fitted to the impeller rather than the baffles to prevent material from settling upon it. The resulting flow pattern was outwards at the bottom of the cup, upwards along the vertical baffles and down over the top of the cylinders. The original direction of flow between the cylinders was, therefore, reversed. The three blades within the top of the outer cylinder were removed, and holes were cut into the <u>tops</u> of both inner and outer cylinders,

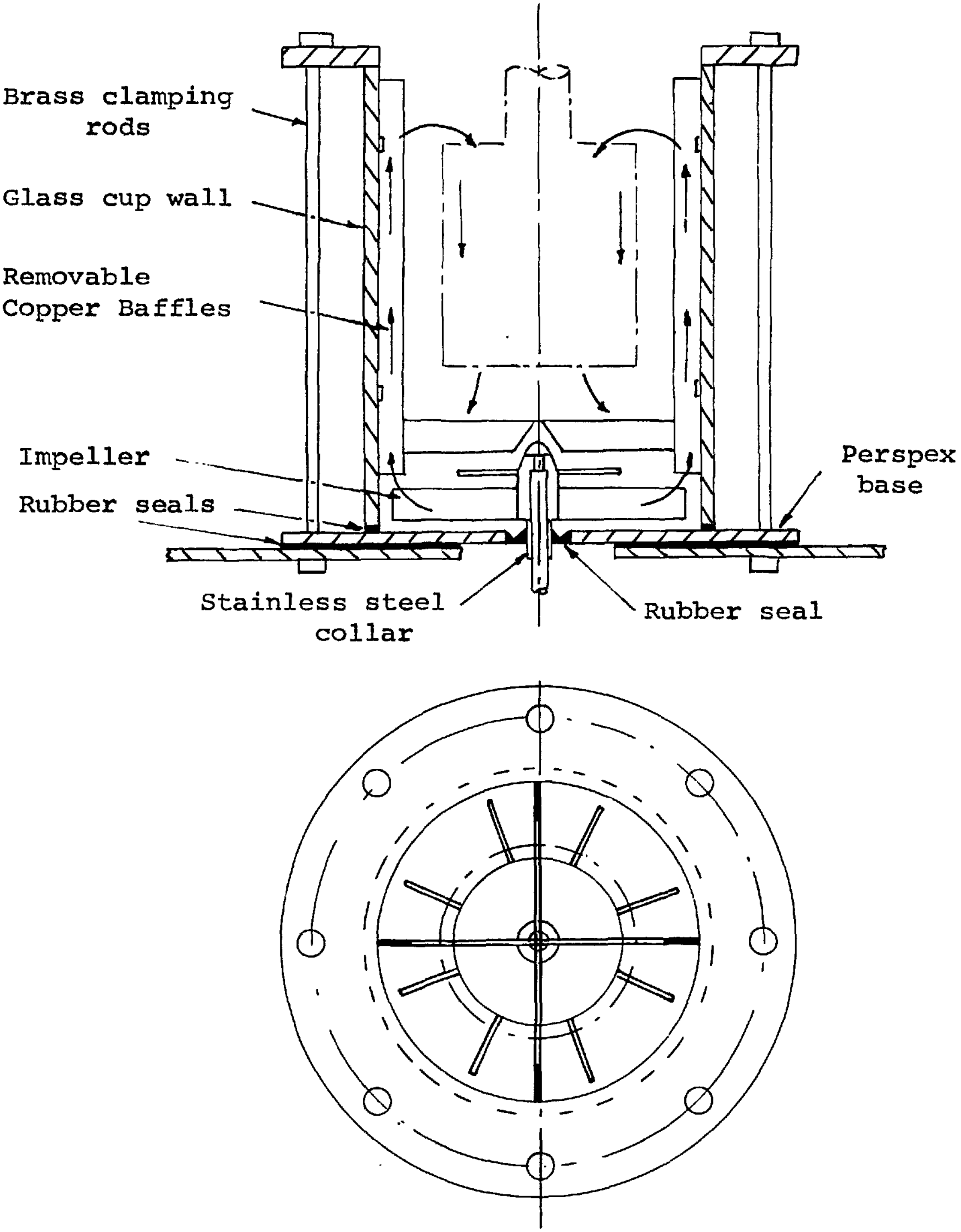


Fig.3.2 Details of Sample Cup and Impeller

leaving them supported only by slender struts. Thus unhindered settling was permitted between the cylinders. In the manufacturers' design there was a non-uniform rate of shear around the three blades which was now removed. In trials of this arrangement, a pipette was used to extract samples of the suspension at different points within the system to check for homogeneity.

Preliminary tests with this modified viscometer revealed that there was slip occurring at the cylinder surfaces. It appeared as an apparent decrease or minimal increase in viscosity with increasing concentration of solids. Further tests were, therefore, conducted with sandpaper and wire-mesh linings bonded to the cylinder surfaces; the results supported the conclusion that slip had previously occurred. From these tests, and by consideration of the work of Van Wazer and Carver (98), it was decided to cut vertical grooves 0.0625" wide and 0.04" deep and 0.1" apart into the cylinder walls. Design considerations which determined the size of these grooves were, firstly, that they should be small compared to the cylinder diameter (less than 1:25) in order to maintain the original, cylindrical shapes of the surfaces; secondly, that at least two or three particles could be contained side by side in the grooves so that they could easily flow in and out and, thirdly, that their dimensions should be within machining limits. Details of the modified cylinders can be seen in Fig.3.1.

The cutting of the grooves meant that the annulus gap was effectively wider; therefore, the instrument needed rechecking for stability.

$R_e = \frac{Uh}{\nu}$ (96) for concentric cylinders where U is the maximum surface velocity, h is the depth of shear and ν is the kinematic viscosity.

$$R_e = \frac{\omega R_o (R_o - R_1)}{\nu}$$

$$R_e \bumpeq 3{,}850 \quad (3{,}850 < 12{,}500)$$

The system is well within the laminar range limit of the lowest value quoted by Taylor of 12,500 (95). The figure calculated is for the worst case of η = 0.01 poise, maximum gap width and the highest speed. Density values of 2 were encountered in the experiments but these were accompanied by a corresponding increase in viscosity.

For all of the tests, the VL outer cylinder was used having an inside diameter of $1\frac{7}{8}''$. The VLB inner cylinder was used for most of the tests with an outside diameter of

$1^{11}/_{16}$", but certain higher viscosity experiments were carried out with the <u>VLC</u> <u>inner</u> cylinder of outside diameter $1^{9}/_{16}$". The length of both inner cylinders was 1½".

Details of speeds of rotation, shear-rates, etc. are as follows:

Cylinders	Inner Cylinder	Gear No.	Speed r.p.m. at 50 c/s	Shear rate (sec.$^{-1}$)	Approx. range (poise) 20 gm.cm. Spring.
with VL Cylinders	A	1	297.3	950	0.168
		2	248.2	791	0.2
		3	196.4	628	0.25
		4	148.6	475	0.33
		5	99.5	318	0.5
	B	1	Ditto	328	0.58
		2		274	0.68
		3		217	0.85
		4		164	1.13
		5		110	1.68
	C	1	Ditto	204	1.08
		2		170	1.3
		3		135	1.6
		4		102	2.1
		5		68	3.2

cont:

Cylinders	Inner Cylinder	Gear No.	Speed r.p.m. at 50 c/s	Shear rate (sec.$^{-1}$)	Approx. range (poise) 20 gm.cm. Spring.
with VM Cylinders	A	1	297.3	483	0.5
		2	248.2	403	0.6
		3	196.4	319	0.8
		4	148.6	242	1.0
		5	99.5	162	1.6
	B	1	Ditto	250	1.2
		2		209	1.4
		3		165	1.8
		4		125	2.3
		5		84	3.5
	C	1	Ditto	173	2.0
		2		144	2.5
		3		114	3.2
		4		85	4.2
		5		58	6.0
with VH Cylinders	A	1	Ditto	235	11.6
		2		196	14
		3		155	17.5
		4		117	23
		5		79	35
	B	1	Ditto	127	32
		2		106	39
		3		84	49
		4		64	65
		5		43	97

A sample of 450 ml was used throughout the tests. The sample cup and cylinders were immersed in a special thermostatted water bath set at 25°C ± 0.1. The bath was designed to allow the centrifugal-stirrer shaft to act through the base via a water-tight rubber seal. The stirrer motor was bolted to a platform, which was supported by three pillars, so that a tachometer could be fitted to the lower end of the drive shaft. The general arrangement of the instrument is shown in Fig.3.3 and Plate 3.1.

The viscometer was calibrated with a set of six aqueous glycerol solutions at 2.09, 7.45, 19.6, 42.7, 86.3 and 152.4 cP ± 1%. These solutions, in turn, were originally calibrated using standard viscosity-density tables (99) and checked by an Ostwald viscometer. A little formulin was added to the solutions to prevent the growth of fungi. The instrument was calibrated completely empirically, using each solution, at each speed of rotation for each set of cylinders used and at stirrer speeds of 400, 500 and 600 r.p.m. This procedure eliminated uncertainties arising from the rotational effect of the stirrer on the inner cylinder. In other words, it was not assumed that the torque was simply proportional to the speed of rotation of the viscometer. The solutions were preserved

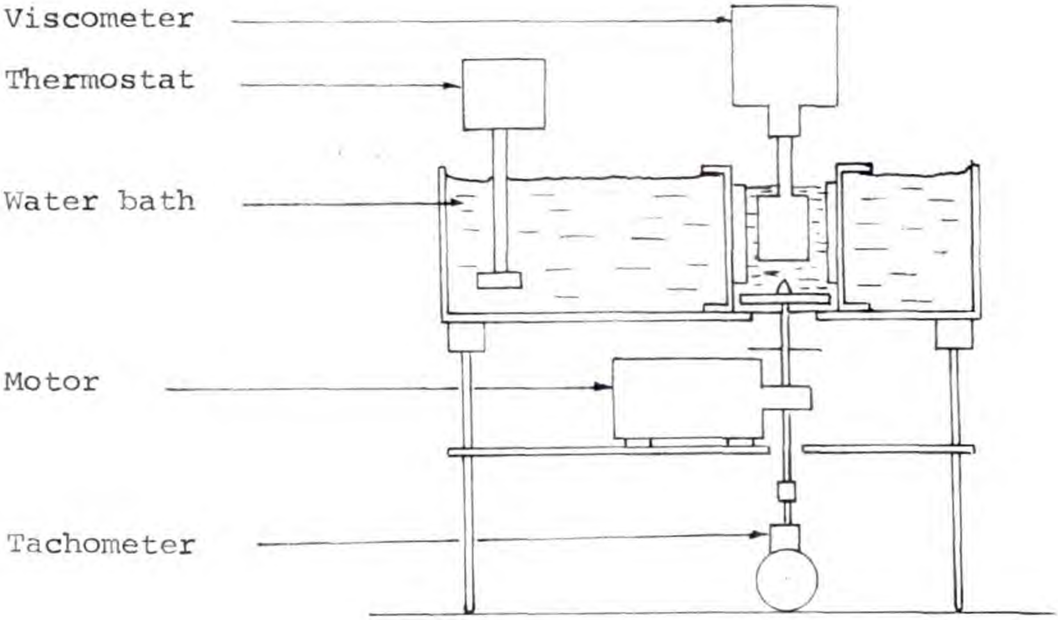


Fig.3.3

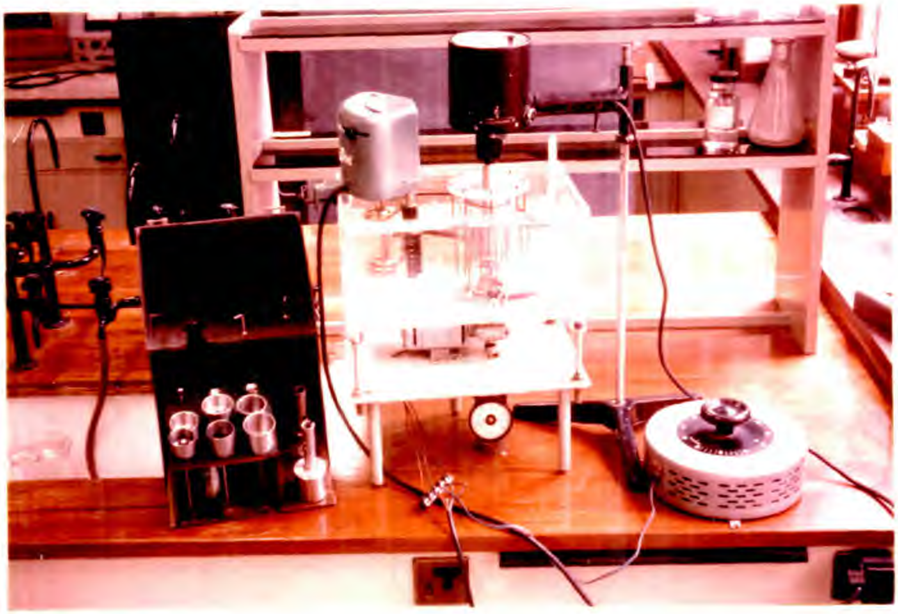


Plate 3.1

for regular checking of the instrument (See pages 245 & 246).

The size of particles that could be handled in the viscometer was determined by the width of the annulus. Van Wazer et al (100) quote a ratio of 10:1 for annulus width to particle size, as a permissible lower limit (to avoid "bridging"). Consequently, the minimum gap width used, namely $^3/_{32}$" (2.38 mm), permitted a maximum particle size of 238μ (< 72 mesh) to be employed. Although, at high concentrations of coarse particles "bridging" did tend to occur at high rates of shear, this was considered to be due to centrifugal forces overcoming the stabilizing effect (96) of the rotating outer cylinder. Thus, a layer of particles was built up on the outer wall which eventually filled the gap. This phenomenon was observed by stopping the viscometer and examining the outer cylinder to see if there was a layer of particles deposited there. This build-up could be avoided for these suspensions, to a large extent, by taking instantaneous readings of the instrument after starting from rest. The results, therefore, for highly concentrated, coarse suspensions, at high shear-rates, do not possess the same degree of accuracy as the other suspensions, but it was considered worthwhile including them in the thesis.

The design of the viscometer inevitably permitted several minor sources of error. The original calibration curves prepared from the aqueous glycerol solutions were estimated to be accurate to within ± 2%, considering the calibration of the original solutions, the accuracy of the stirrer speed, the viscometer dial reading and temperature variations. This was considered adequate, as the main object of the project was the observation of the effect of viscosity on grinding, rather than absolute measurements of viscosity - which, in any case, are subject to limitations of interpretation. It was estimated that reproducibility was about ± 2% giving an overall accuracy to about ± 3% or 1 cP, whichever was the greater. The scatter of points on the graphs at high concentrations of solids reveals that there was a reduction in accuracy in this range. However, such results are still worth including in the thesis. In order to minimise such errors many tests were rechecked several times.

The values of shear-rate quoted are those at the inner cylinder surface (as provided by the manufacturer). It is known that the rate of shear for non-Newtonian materials varies across the annulus in a rotational viscometer. It follows that the results quoted for shear-rate effects are nominal rather than absolute. In order to obtain absolute values of "viscosity", empirical power functions have been proposed to fit flow curves of anomalous materials of the form:

$$^{(101)}\text{Shear-rate, } D = \frac{\tau^{n}}{K} \qquad (3.1)$$

where τ is the shear-stress, K is a constant and n indicates the degree of shear-rate dependence. With new materials, neither n nor K is known and, therefore, a process of numerical approximation must be employed and an adequate number of points is needed to fit the data. Furthermore, all suspensions do not conform to a law of this kind and the treatment is not applicable.

A general expression was evolved by Kreiger and Elrod $^{(102)}$ to give the shear-rate for all fluids at the surface of the bob:

$$f_{(Sb)} = \frac{\Omega}{\ln\epsilon}\left[1 + \left(\ln\epsilon \cdot \frac{d\ln\Omega}{d\ln_{Sb}}\right) + \left(\frac{(\ln\epsilon)^{2}}{3\,\Omega} \cdot \frac{d^{2}\Omega}{d(\ln_{Sb})^{2}}\right) - \left(\frac{(\ln\epsilon)^{4}}{45\,\Omega} \cdot \frac{d^{4}\Omega}{d(\ln_{Sb})^{4}}\right) + \ldots\ldots\right] \qquad (3.2)$$

where ϵ is R_c/R_b (the ratio of the radii of the cup and the bob), $f_{(Sb)}$ is the shear-rate, and Ω is the speed of rotation.

Application of this formula is intended for the determination of absolute values of viscosity of high accuracy and would require much computation of results. It was, therefore, decided to treat the measurements as "nominal" for the purposes of the present research, and the shear-rates quoted by the manufacturer for the inner cylinder surface have been used.

Chapter 4.

EXPERIMENTAL - RHEOLOGY

Details of samples

Five types of solid were used, namely:- ground quartz, glass platelets, glass spheres, glass rods and polymethylmethacrylate spheres.

The quartz was high purity Norwegian quartz received in 1" to 3" lumps. It was jaw-crushed and disc-milled to the sizes required. The fractions were sieved through a set of B.S. sieves, washed and dried. The plates were purchased as a commercial product, washed and dried. They were approximately 100μ thick and 400μ diameter; they were irregular in shape, having been crushed down to this size. The glass rods were made by crushing glass wool under water in a pestle and mortar; they were then manually abraded through an 18 mesh sieve while still wet. The approximate dimensions of the rods were 750μ long and 30μ diameter. It is realised that the permissible gap-to-particle ratio of 10:1, defined by Van Wazer et al, is exceeded by the plates and particularly the rods, with respect to the width of the viscometer gap. There may be some doubt concerning the significance of the results obtained with these particles; but the results appeared to be satisfactorily reproducible and have, therefore, been included in the results section.

The poly-methyl-methacrylate spheres and glass beads of two densities were purchased in the form used, sieved to -200 +300 mesh, washed and dried.

All the samples were weighed out to ± 0.1 gm. The densities of the materials were determined by means of an s.g. bottle, so that the concentrations of all the suspensions could be expressed as percentage solids <u>by volume</u>.

Method of measurement.

The minimum percentage solids to be used (usually 5 vol.%) was placed, with the appropriate volume of distilled water, in the sample cup. The inner cylinder of the viscometer was selected which would give the maximum deflection of the needle; the same outer cylinder (VL) was used for all the tests. A stirrer speed was selected which was the minimum required to keep the suspension homogeneous (usually 400 r.p.m.). The contents of the sample cup were allowed several minutes until they had reached the same temperature as the thermostatted water bath (25° ± 0.1°C). The stirrer and viscometer were set in motion and the suspensions were allowed to circulate for several seconds in order to allow conditions to become steady. The viscometer dial reading was then noted at the five rates of shear.

The stirrer and viscometer were switched off, so that the particles settled, and a certain percentage (usually 5 vol.%) was drawn off the supernatent liquid with a pipette. The equivalent volume of particles was added (by weight) and the measuring procedure repeated.

Variables studied.

The effects of the following variables on the viscosity of suspensions were investigated: concentration, shear-rate, particle size, particle shape, particle density, particle size mixtures and inter-particle friction.

Results.

Effect of concentration, with quartz grains. Every test showed an increase in viscosity with increasing volume fraction of the solid content (Figs.4.1 to 4.3). The extent of the increase was dependent on particle size and on shear-rate. It can be seen from these graphs that the viscosity increases gradually at first - though at a far greater rate than corresponds to that quoted by Vand (89), for example (Fig. 4.3), for non-settling spheres of high concentration. In fact the results would not fit any formulae which did not take into account shear-rate and size of the particles. At about 27.5 vol.% solids there is a rapid increase in viscosity and small further additions of solid content cause large increases in viscosity. The transition is not sharp, however; it sets in at lower concentrations, and is less sharp the higher the rate of shear.

The maximum concentration, measurable within the range of the instrument, was 34 vol.% with particles of -350 mesh, and 27.5 vol.% with those of -72 +100 mesh. Further increases in the concentration were restricted as the suspensions were becoming a cohesive mass.

Effect of shear-rate with quartz grains: The suspensions were either dilatant or Newtonian by nature, depending on the concentration of solids and the particle size (Figs. 4.4 to 4.6). Greater dilatancy was found with the coarse grained suspensions rather than the fine, and also with increasing concentration of solids. Hence, an increase in viscosity of up to 100% was obtained with a suspension of particles of

-72 +100 mesh at 27.5 vol.% solids, over the range of shear-rates 110 to 327 sec^{-1}. The suspensions became, more or less, Newtonian below 10 vol.% solids with the largest particles (-72 +100 mesh) and below 20 vol.% for the smallest (-350 mesh +10μ), other sizes having intermediate values.

Effect of particle size, with quartz grains: The viscosity of the suspensions increased in direct proportion to the particle size, for a given concentration and a given rate of shear (Figs.4.7 and 4.8). Thus, an increase in viscosity of approximately 280% was obtained over a mean particle size range of 30μ to 180μ at all concentrations and shear-rates.

Effect of particle shape, with quartz grains and various glass particles: It was found that the viscosity increased with the degree of anisometry of form of the particles at equivalent concentrations and shear-rates (Figs.4.9 and 4.10). The suspension of rod-shaped particles was a connected structure of 26 cP at 20 vol.%, while that of spherical particles was still free flowing at 40 vol.%, with a viscosity of 18 cP. Intermediate values were obtained with the plate-shaped and granular particles.

The same general shape of curve was obtained of viscosity-concentration for all particle shapes, i.e. small increases in viscosity with concentration were found up to a point where the particles began to interact, when there were rapid increases up to the maximum measurable concentration. Above this maximum concentration the particles appeared to "jam". The relative concentrations beyond which this occurred were rods 20 vol.%, plates 25 vol.%, rough grains 30 vol.% and spheres 50 vol.%.

The shear-rate tests showed that the character of the suspensions depended on the degree of anisometry of the particles (Figs.4.11 to 4.14). The suspensions of rod-shaped particles were the most dilatant, giving increases of viscosity from 11 to 20 cP at 20 vol.% solids over a range of shear-rates from 102 to 204 sec^{-1}. The suspensions of spheres, on the other hand, were barely dilatant at 20 vol.%, but gave increases of 10 to 13.5 cP at 40 vol.% solids over a similar range of shear-rates. The degrees of dilatancy for the suspensions of plate-shaped and granular particles, were intermediate to those of the spheres and the rods.

Effect of density of particles, with glass and poly-methyl-methacrylate spheres: Particles of greater density were found to produce suspensions of higher viscosity, for a given concentration and a given shear-rate (Fig.4.15). An increase in viscosity of 100% was obtained over a range of density of 1.168 to 2.36 at a rate of shear of 327.7 sec^{-1}, dropping to about 40% at a rate of shear 110 sec^{-1} (Fig.4.16).

The lighter spheres exhibited no dependence on shear-rate at all, even at 50 vol.% solids. The suspensions of both types of glass bead, on the other hand, were dilatant by nature, the heavier beads at concentrations over 15 vol.% and the lighter over 25 vol.%.

Particle size mixtures, with quartz grains: The series was conducted with mixtures of particle sizes (-100 +150 mesh and -350 mesh + 10μ) at 20 vol.% total, solid content. The tests with all large grains naturally gave the same increase of viscosity over those with all small grains, as was found in the particle size tests. A proportion of large to small grains of 16% to 4%, however, caused a marked drop in the viscosity (Fig.4.17). As the proportions of the two size fractions became equal so further decreases in the viscosity were observed, until the viscosity was barely above that for the small size alone. Further increases in the proportion of small grains produced little difference in the viscosity of the suspension. Hence, a ratio of large to small grains of 4% to 16% only produced an increase in viscosity of 2% over that with 20 vol.% small grains.

Shear-rate tests showed that the suspensions containing the higher proportion of large particles were more dilatant than those with more small particles (Fig.4.18). The anomalous reduction in viscosity at the middle speeds of the viscometer is within the error margin of the instrument.

Effect of addition of detergent: A conventional, concentrated, fluid, anionic detergent containing 23% solid ("Stergene") was added to suspensions of glass spheres, poly-methyl-methacrylate spheres and quartz grains (all -200 +300 mesh). In each case the detergent reduced the viscosity of the suspension until a minimum was reached.

A decrease in viscosity of 50% was obtained for quartz at 30 vol.% solids (Fig.4.19). This was greater than the decrease with glass or plastic sphere suspensions, which showed decreases of approximately 8% and 34% at 40 and 50 vol.% solids respectively, for 2% fluid detergent addition.

The addition of detergent made the suspensions of plastic spheres slightly dilatant from their previous Newtonian character. The shear-rate dependence of the glass and quartz suspensions was unaffected (Figs.4.20 and 4.21).

The tests conducted using detergent suggested the likelihood of friction playing an important part in the viscosity of suspensions. A further series of tests was carried out, therefore, to examine the influence of detergent upon the static friction between two glass surfaces. A measure of the coefficient of friction was obtained by noting the maximum angle at which a watch glass could be supported on a flat plate without slip. The tests were carried out under water and the angle of the plate was varied by a screw mechanism. The results, however, were inconclusive, but visual observations showed some reduction in the dynamic friction between the surfaces when detergent was added to the water.

Conclusions.

(1) Flow properties: Almost all the suspensions studied were appreciably non-Newtonian, the viscosity increasing with increasing rate of shear. As pointed out earlier, the numerical values obtained for the coefficient of viscosity

are nominal rather than absolute.

No entirely adequate instrument has yet been devised for measuring the rheological characteristics of highly dilatant materials and fast-settling suspensions. The modified Ferranti viscometer proved usable, however, with particles up to 211μ diameter and volume concentrations up to 20 to 50%, depending on the shape and size of the particles.

Rotational viscometers fail at high rates of rotation with highly concentrated suspensions containing coarse particles,because of centrifugal segregation of solids on the wall of the outer cylinder.

(2) Effect of concentration: The viscosity of a suspension increases in all cases with increasing concentration of solids. The extent of the increase, however, is dependent on the degree of roughness, density, anisometry of form of particles and the rate of shear. In general, the increase at the lower concentrations (< 20 vol.%) is small compared to that at higher concentrations (> 20 vol.%) when the rate increases to a point where the viscosity approaches that of a solid mass (at about 35 vol.% for quartz). In contradiction to the results in the literature, no distinct

"critical concentration" was observed, particularly at high shear-rates, although an approximate value of 27.5 vol.% is estimated for quartz at low shear-rates.

(3) <u>Effect of particle size</u>: The viscosity increases linearly with particle size for a given concentration and shear-rate. This result is contrary to many of those in the literature, who have found a decrease; although, strictly, no direct comparison can be made with those results obtained in different types of viscometers.

(4) <u>Interaction between particles</u>: It is clear that the results obtained depend largely on the varying degrees of interaction between the particles. There may be several factors involved.

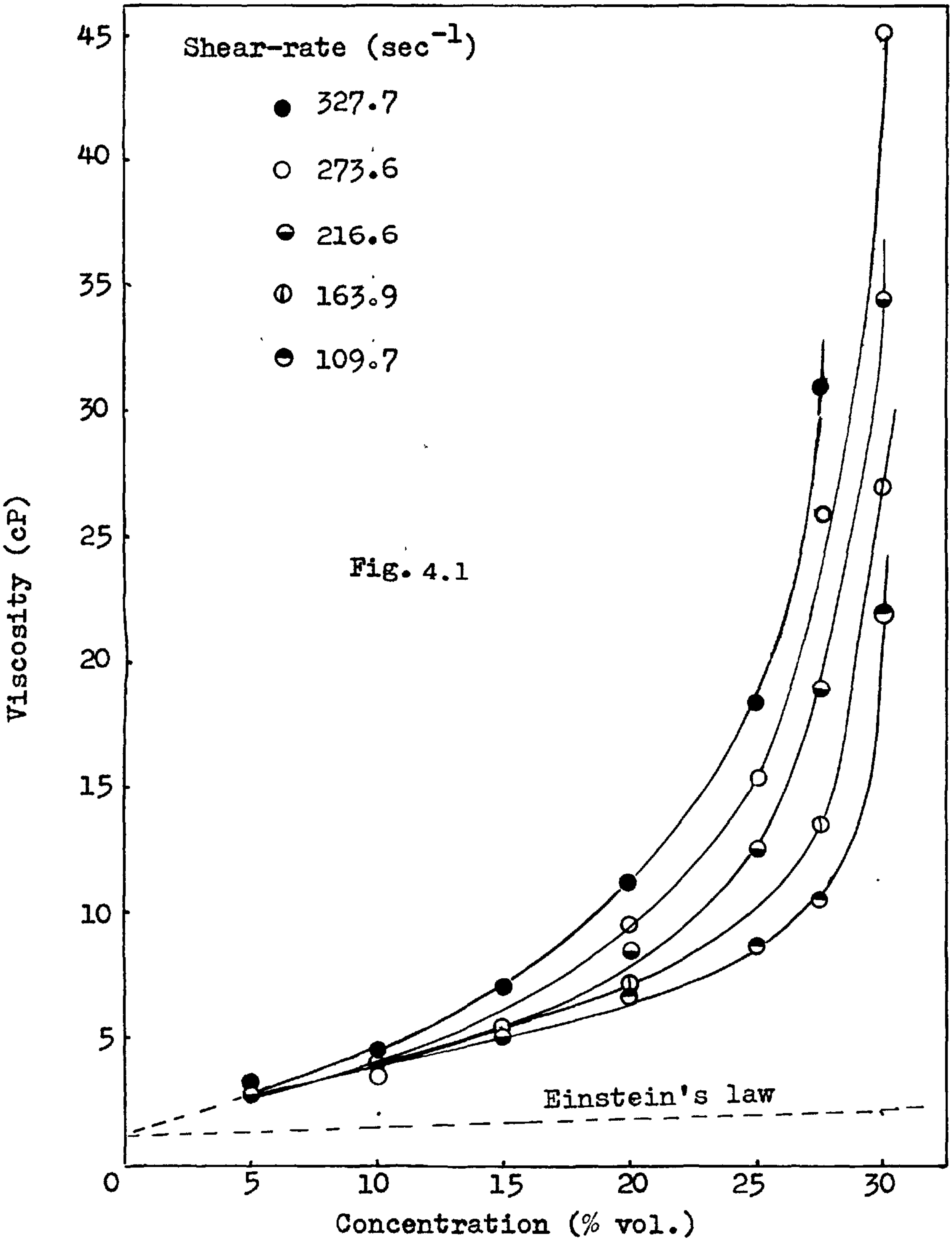
One type of interaction present in even moderately concentrated suspensions is hydrodynamic interference of the flow lines of adjacent particles (37).

Secondly, anisometric particles undergo rotation or precession with the result that they sweep out larger volumes than do spheroidal particles. (It has not been established whether such motion causes local turbulence of the liquid at the rates of shear used in the present work).

Thirdly, the fact that detergents reduce the viscosity of many of these concentrated suspensions suggests that friction between particles is an important factor. (Presumably surface-chemical forces, such as affect the state of dispersion of microscopic particles, can scarcely influence the motion of coarse particles except via friction on contact).

Fourthly, the degree of interaction depends on the size distribution of the solids, such that a small proportion of fine particles may circulate in the voids between the coarse, to reduce the degree of interference for a given concentration, and hence cause a drop in viscosity.

Finally, the flow of any concentrated suspension involves displacement of particles at right angles to the stream lines. Consequently, even with smooth spherical particles, inertia plays a part: particles have to be accelerated and retarded and in both stages their inertia, (if their density differs from that of the liquid), will affect the amount of energy required to absorb these contacts. This dissipation of energy, of course, appears as extra "viscosity". Hence, the viscosity of suspensions of glass beads is greater than that of plastic spheres even when frictional effects of both are reduced as far as possible by addition of a detergent.



Effect of concentration on viscosity
for -72# +100# quartz at five shear-rates.

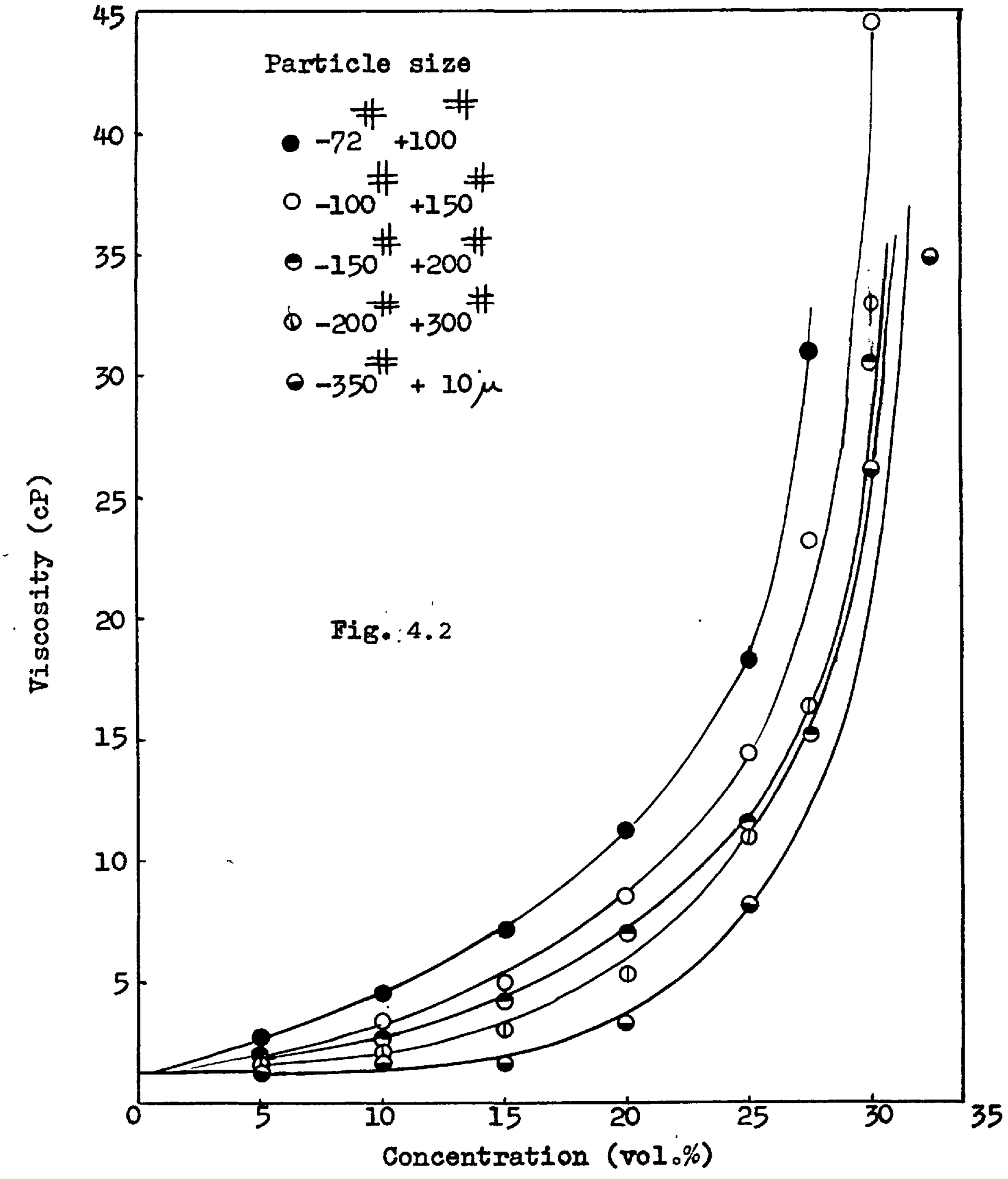


Fig. 4.2

Effect of concentration on viscosity of quartz suspensions of different particle size (at a shear-rate of 327.7 sec^{-1}).

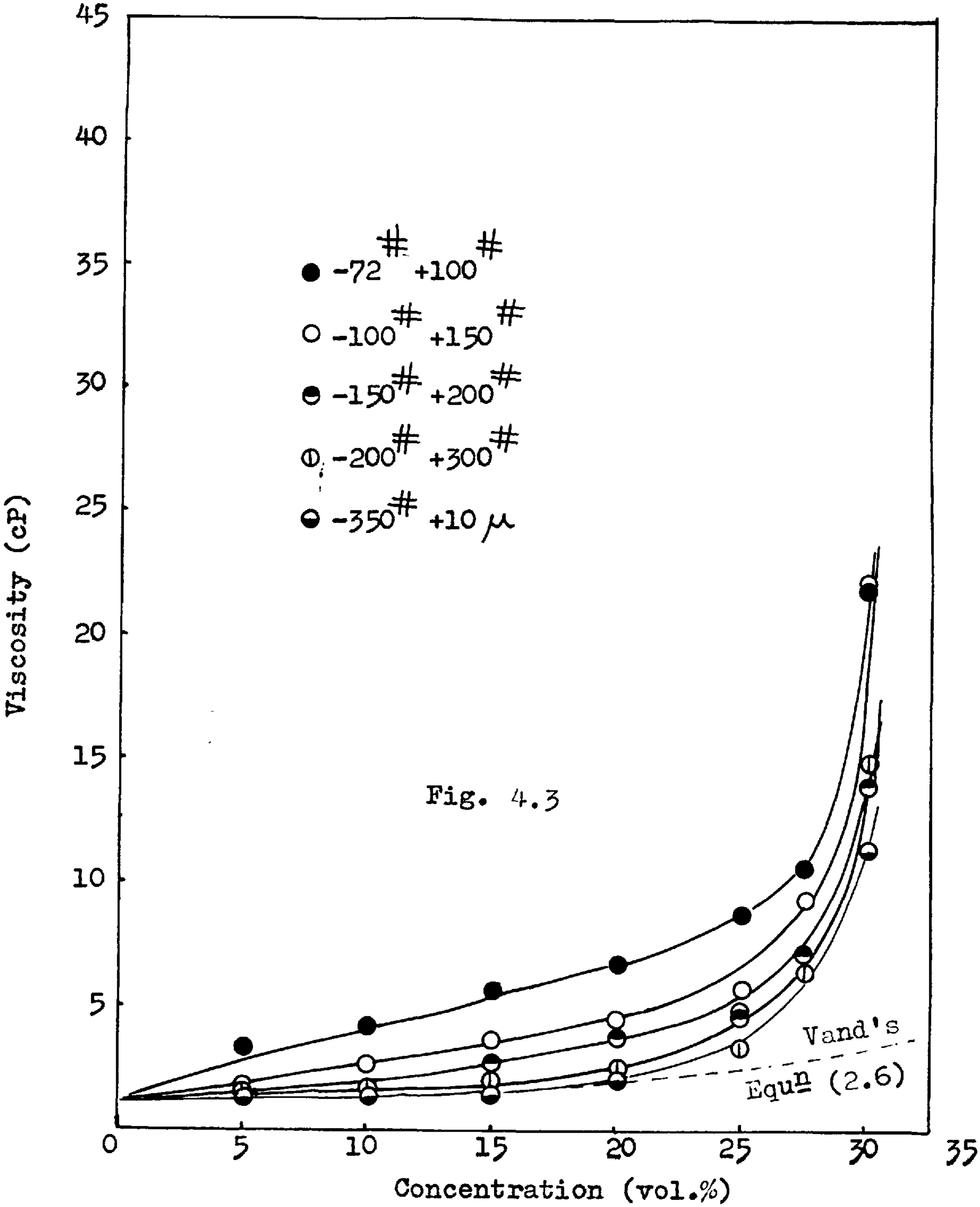
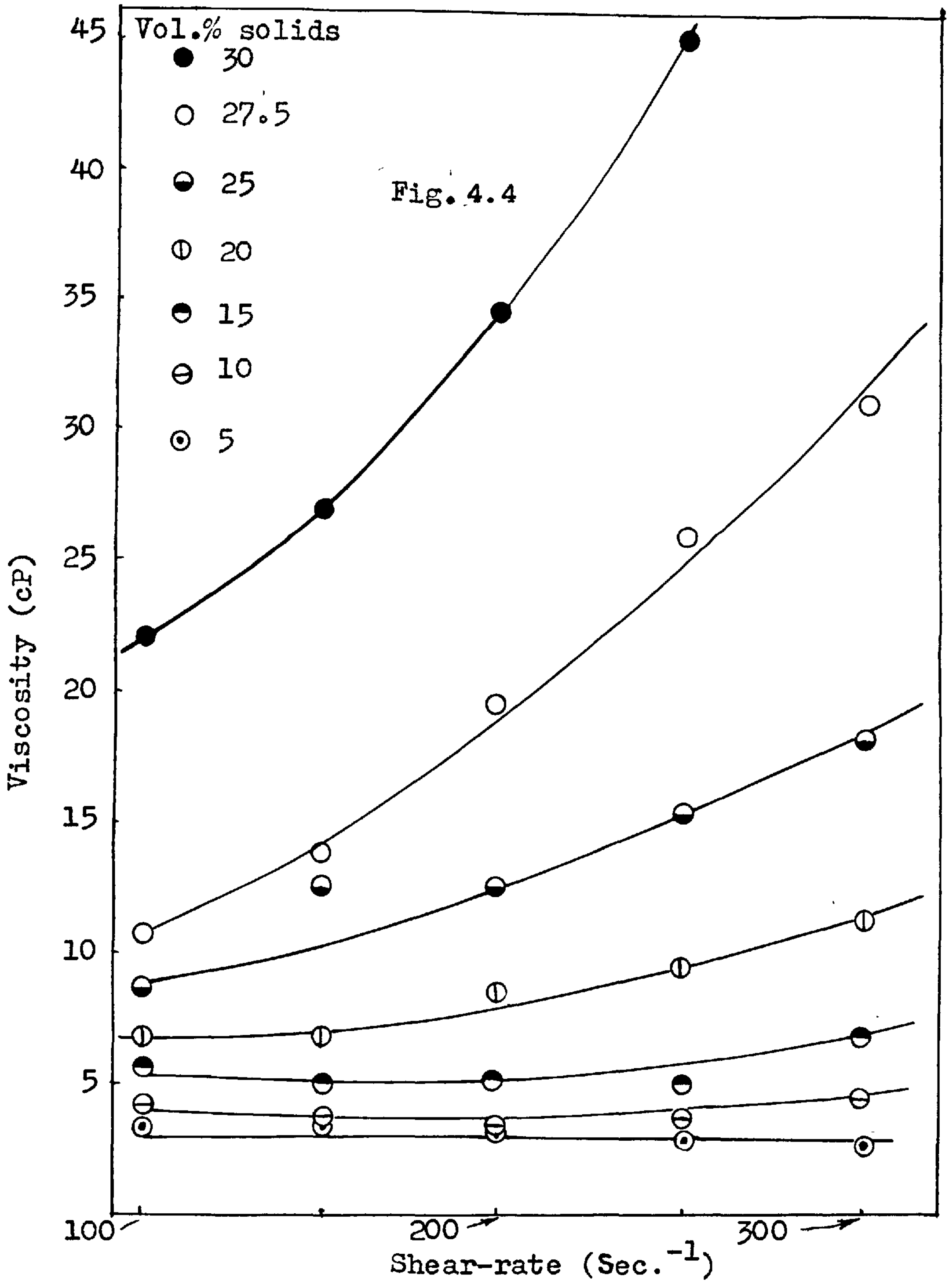
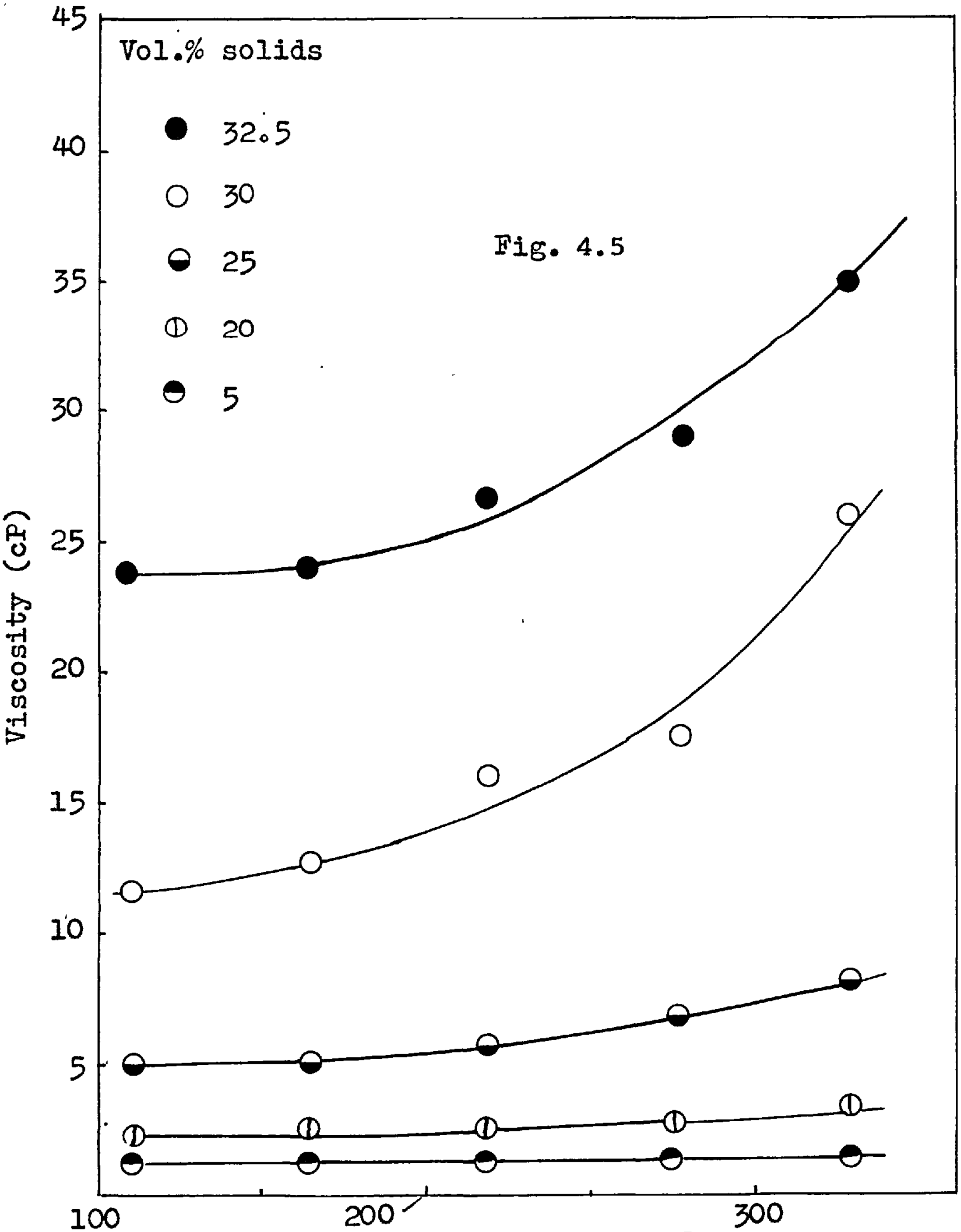


Fig. 4.3

Effect of concentration on viscosity of quartz suspensions of different particle size (at a shear-rate of 109.7 sec^{-1})



Effect of shear-rate on viscosity of $-72^{\#}$ $+100^{\#}$ quartz suspensions.



Effect of shear-rate on viscosity of -350# +10μ quartz suspensions

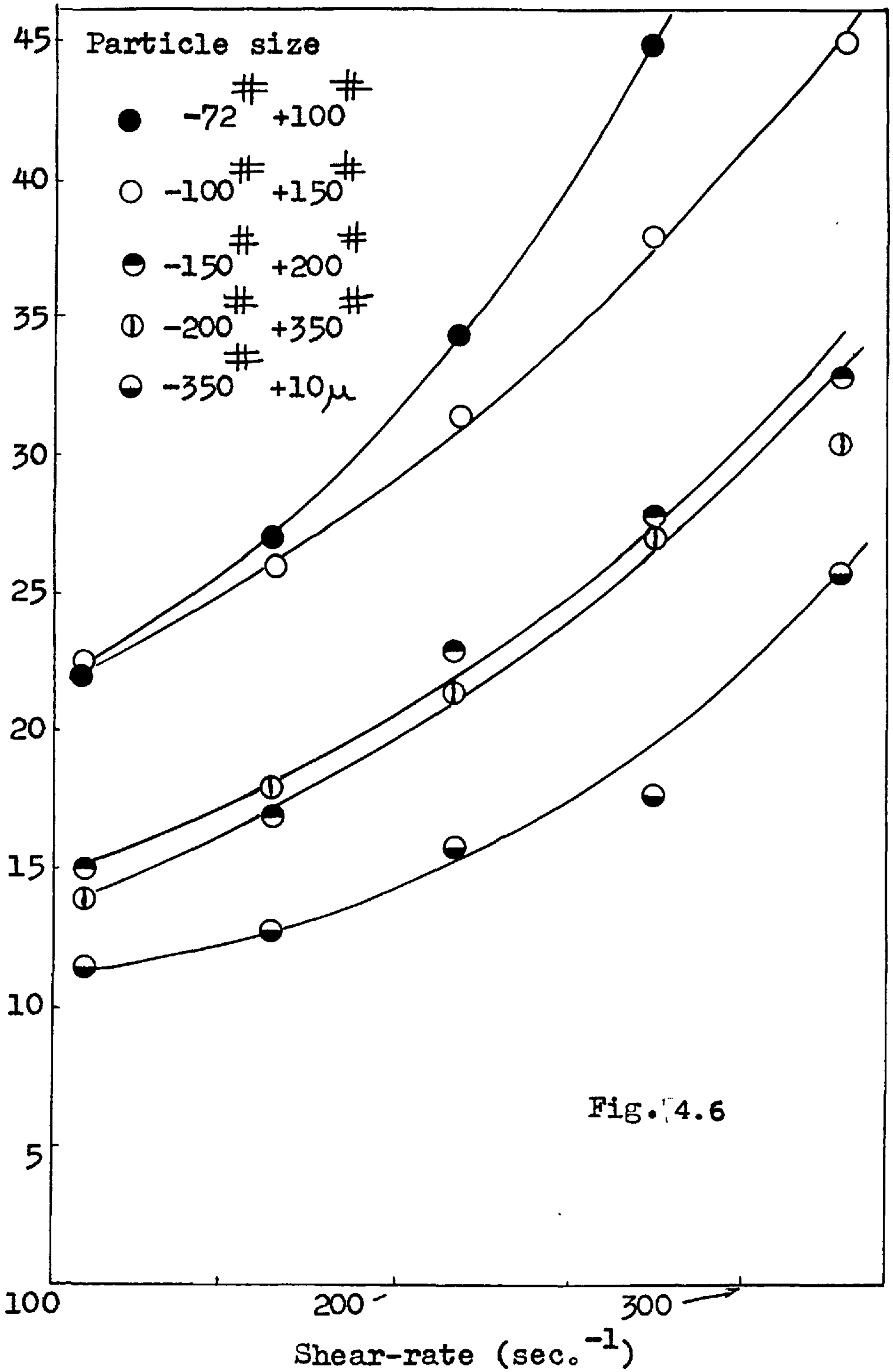
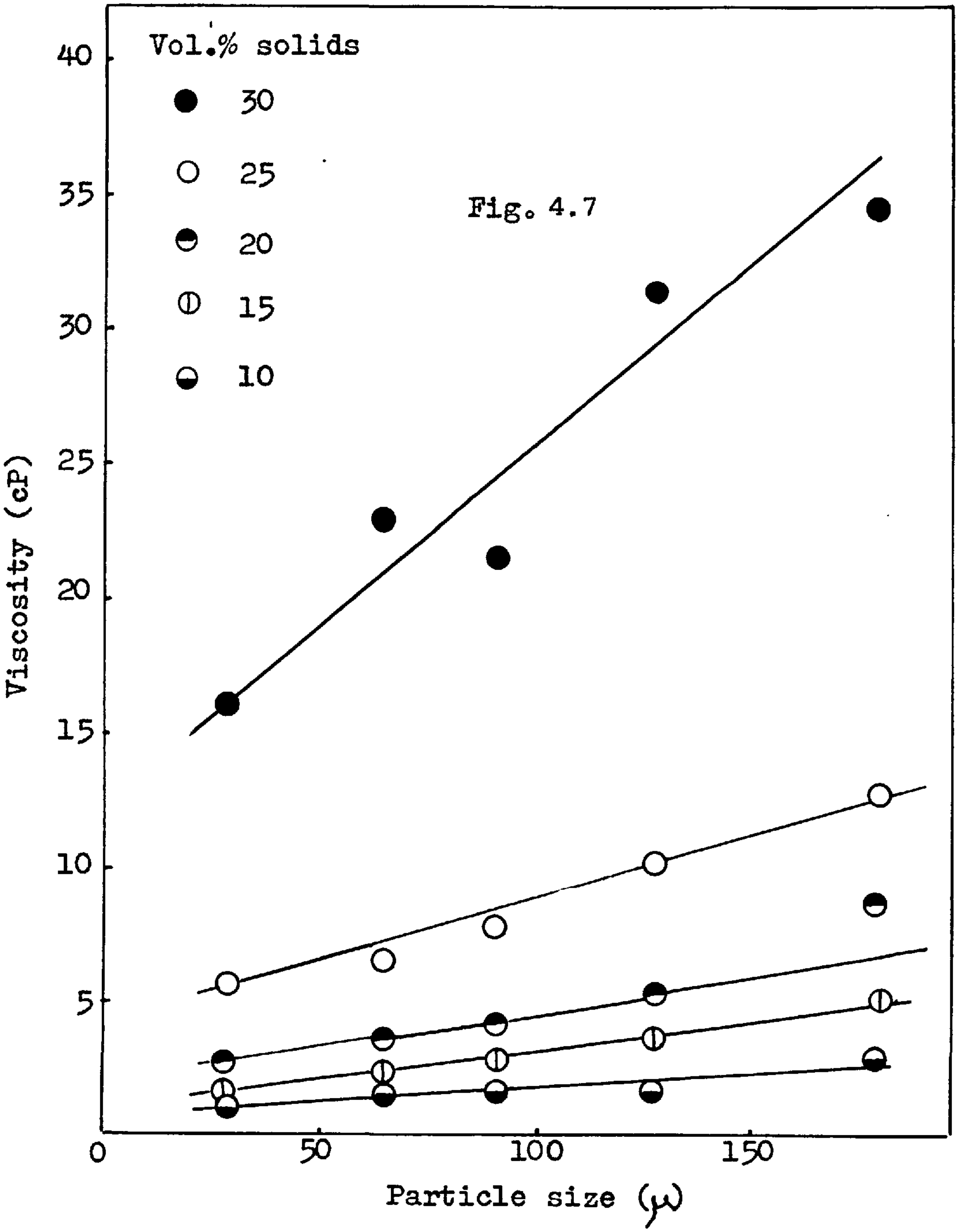


Fig. 4.6

Effect of shear-rate on various size fractions of quartz at 30% solids.



Effect of particle size with quartz grains
(shear-rate 216.6 sec.$^{-1}$)

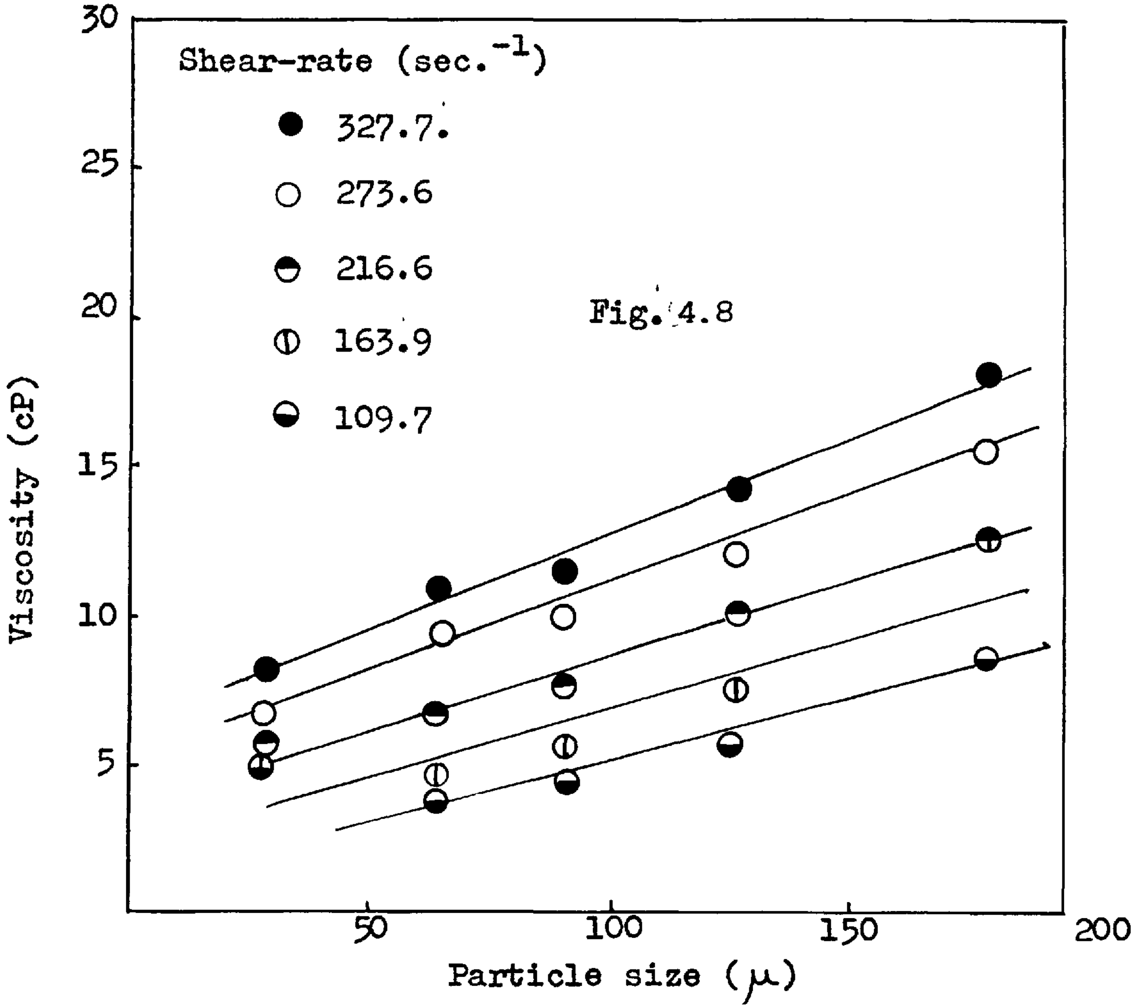
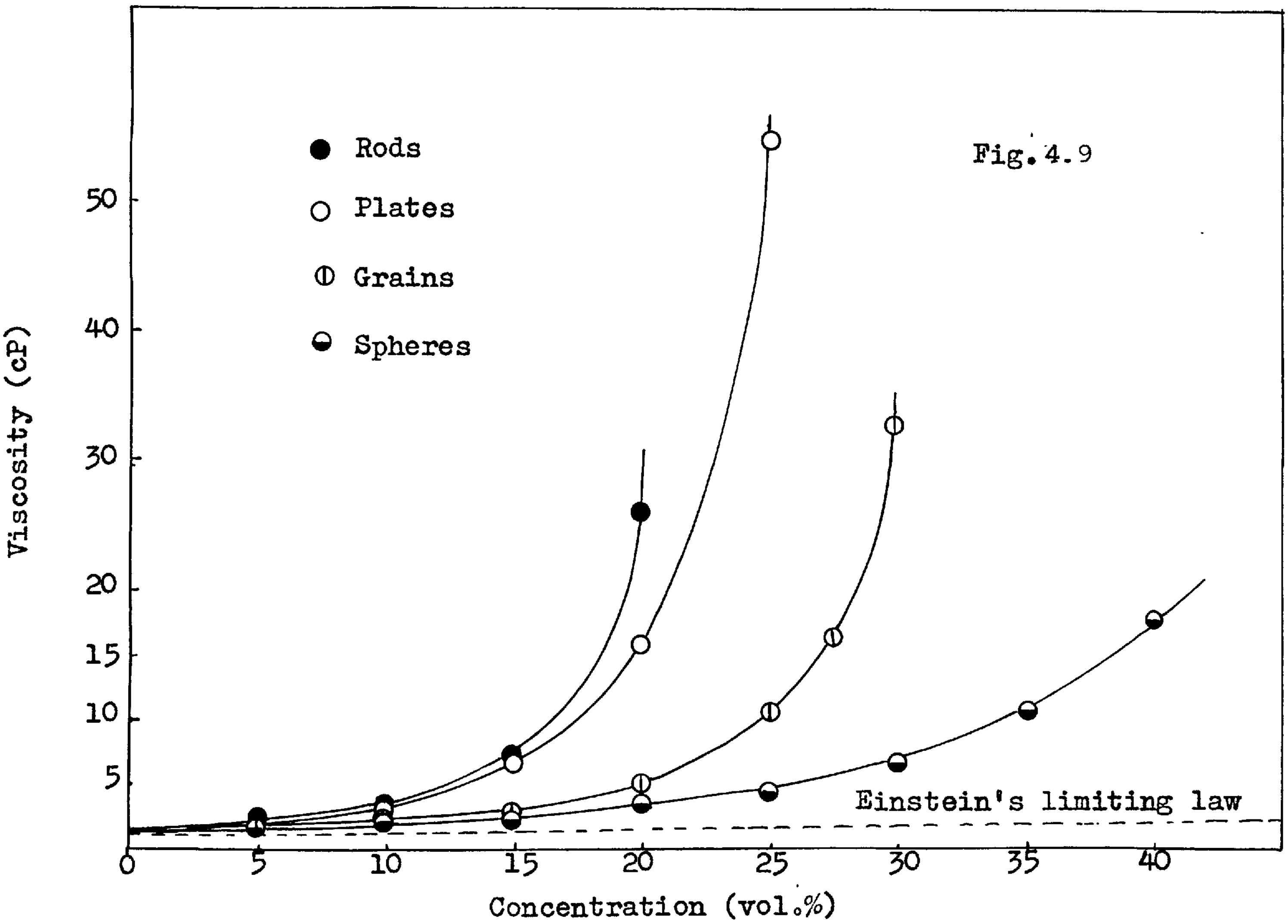


Fig. 4.8

Effect of particle size on the viscosity of 25 vol.% quartz suspensions at five shear-rates.



Effect of concentration on differently shaped particles (shear-rate 327.7 sec.$^{-1}$).

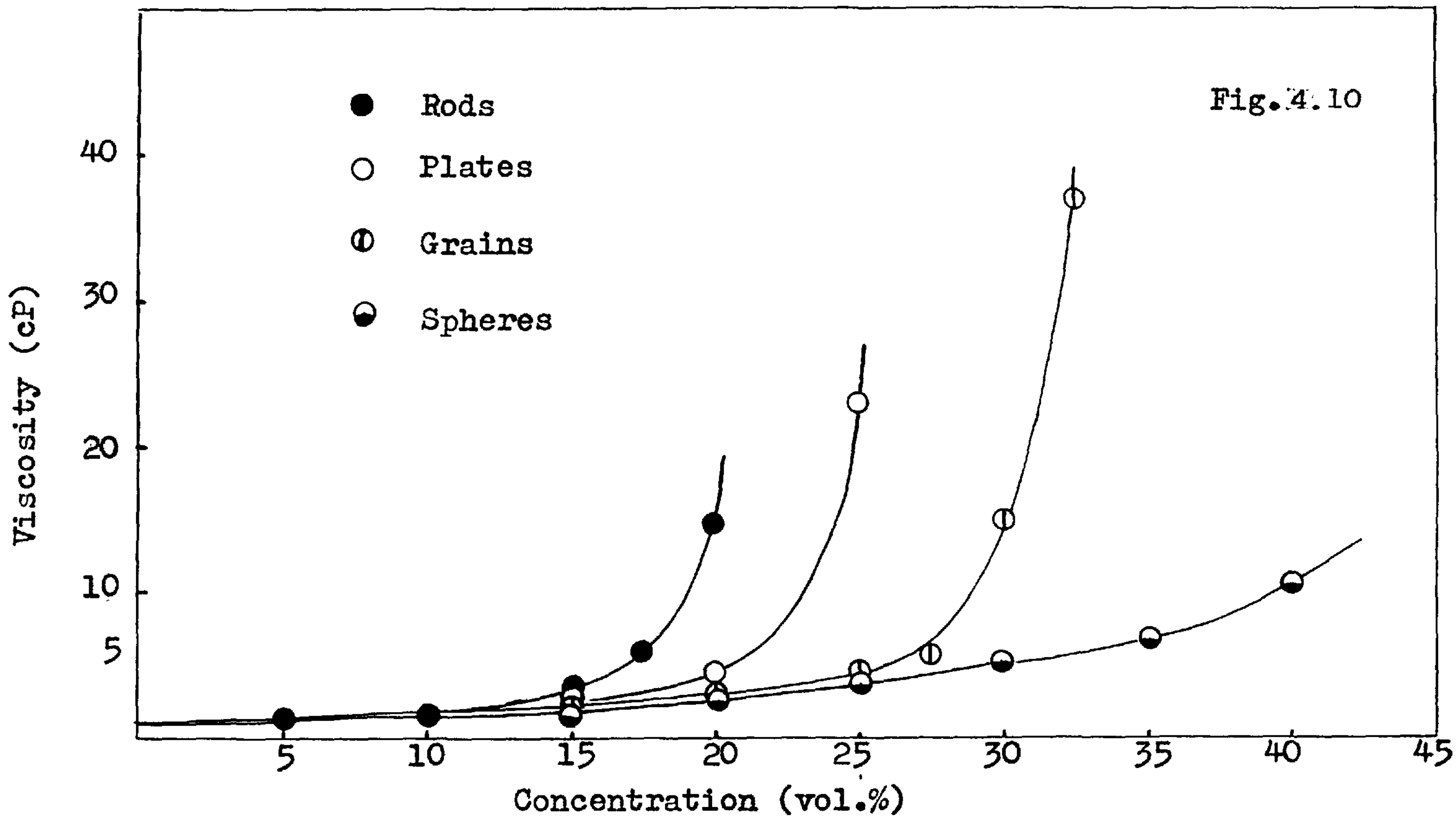
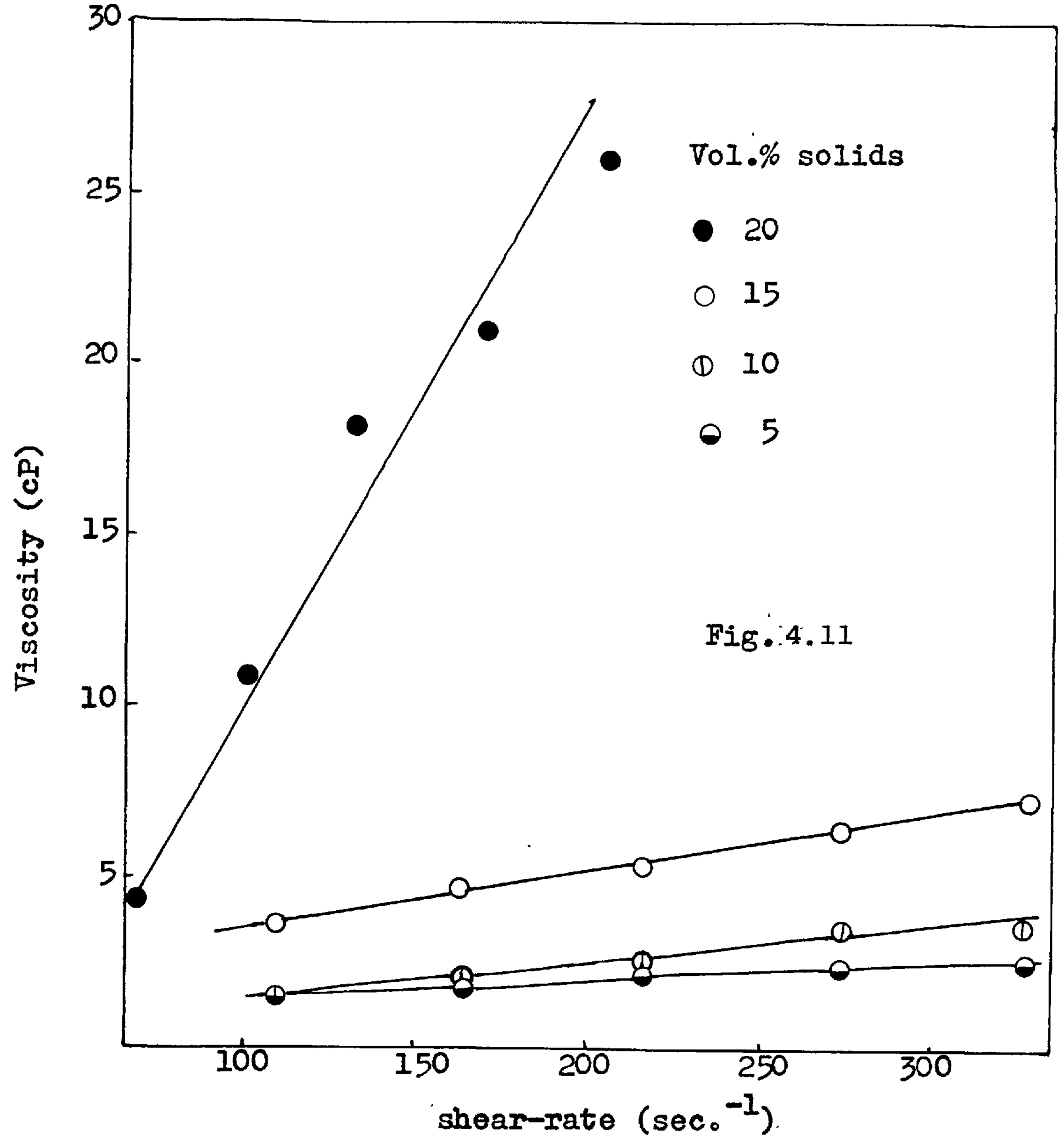
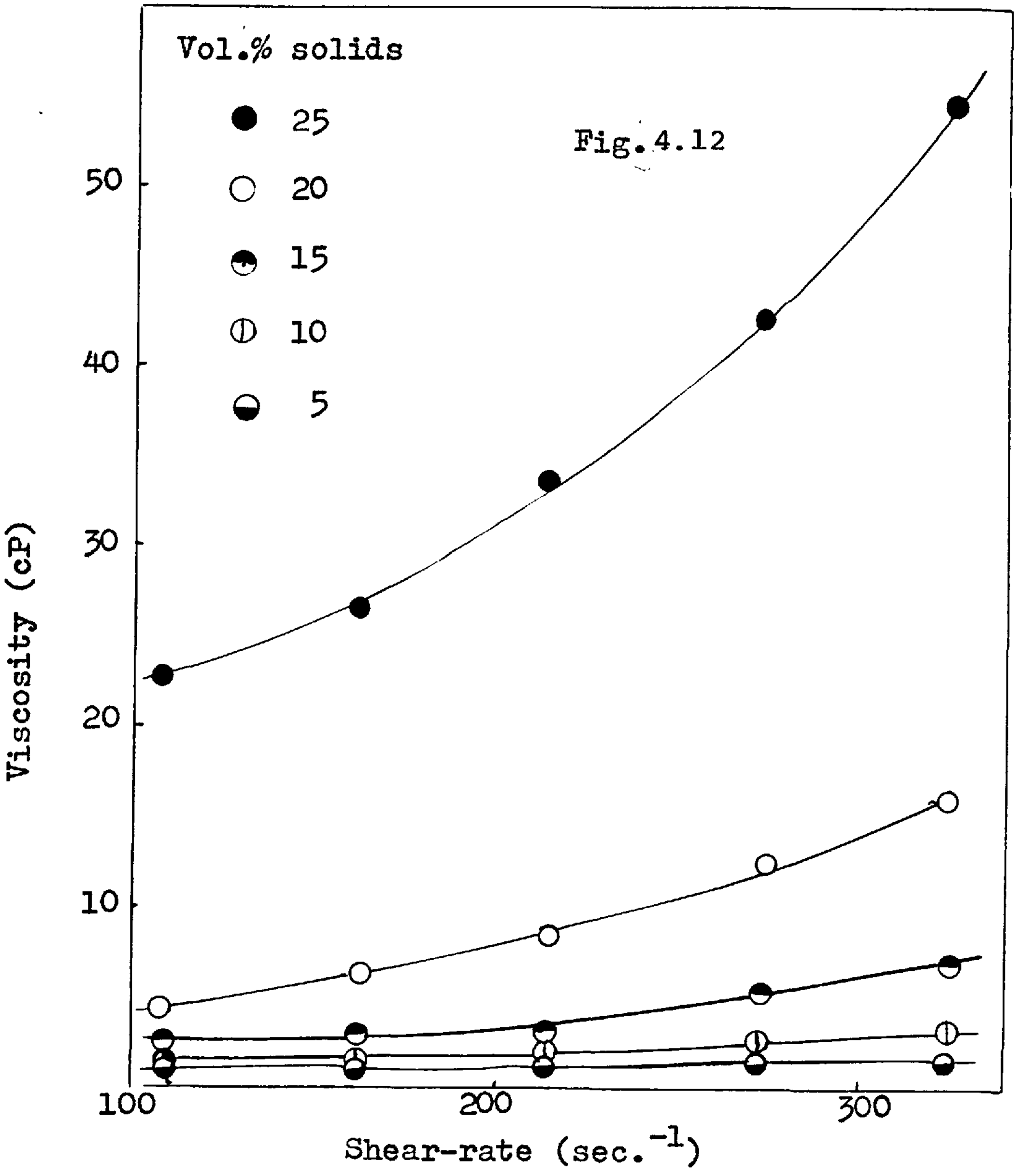


Fig. 4.10

Effect of concentration on differently shaped particles (shear-rate 109.7 sec.$^{-1}$)



Effect of shear-rate on rod-shaped particle suspensions



Effect of shear-rate on plate-shaped particle suspensions

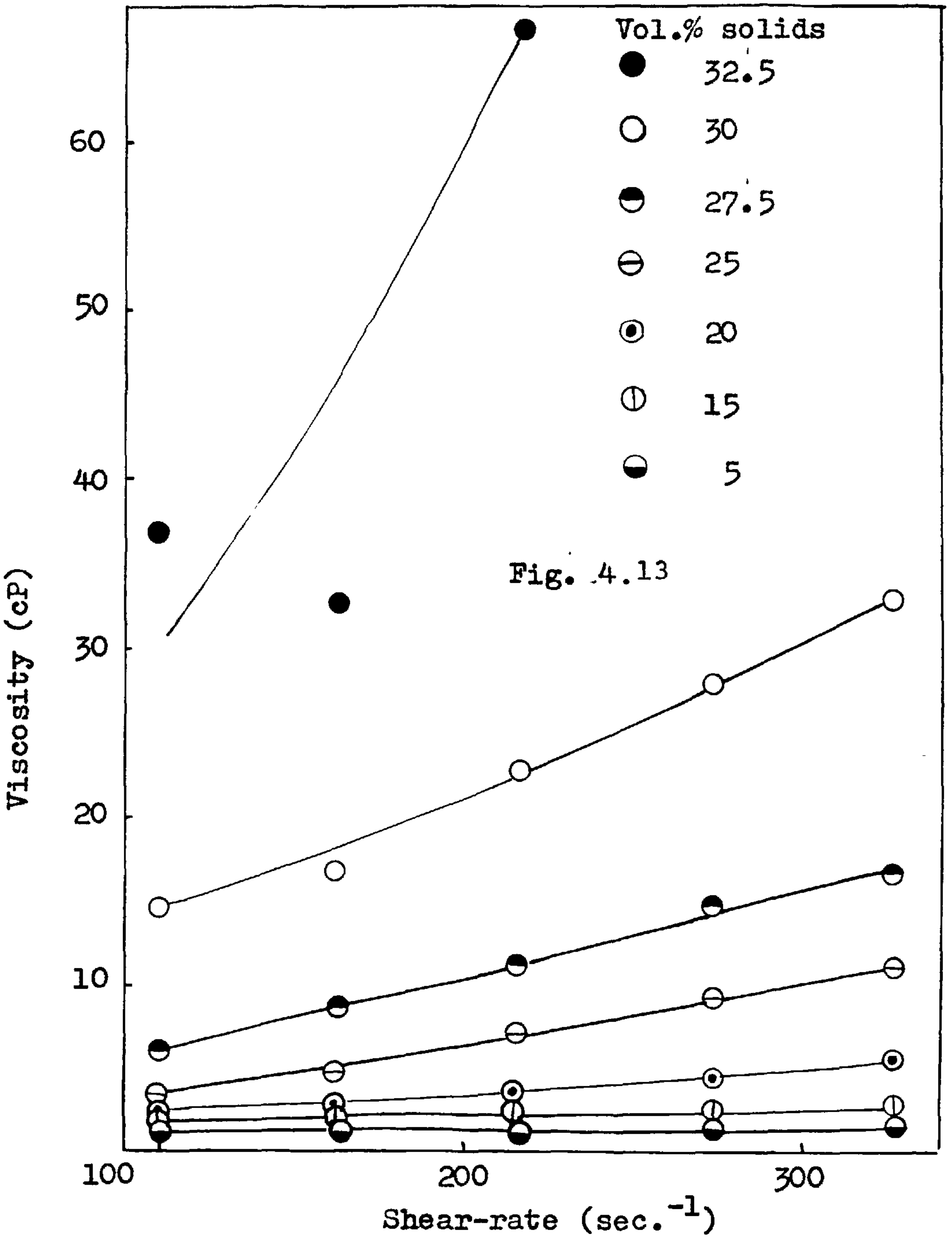
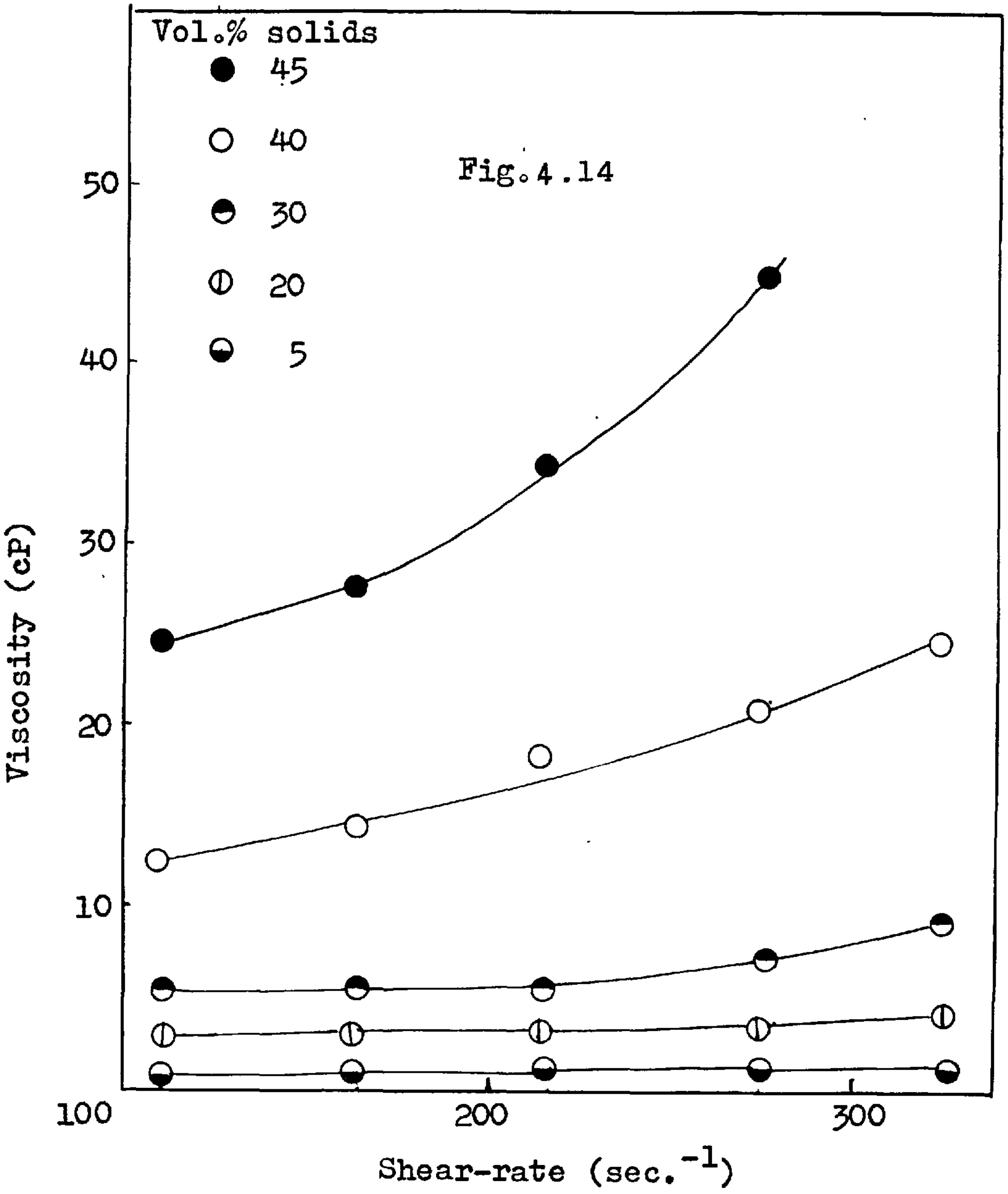


Fig. 4.13

Effect of shear-rate on granular shaped particle suspensions (-200# + 300#).



Effect of shear-rate on a spherical glass-particle suspension ($-200^{\#}$ $+300^{\#}$, ρ = 2.36).

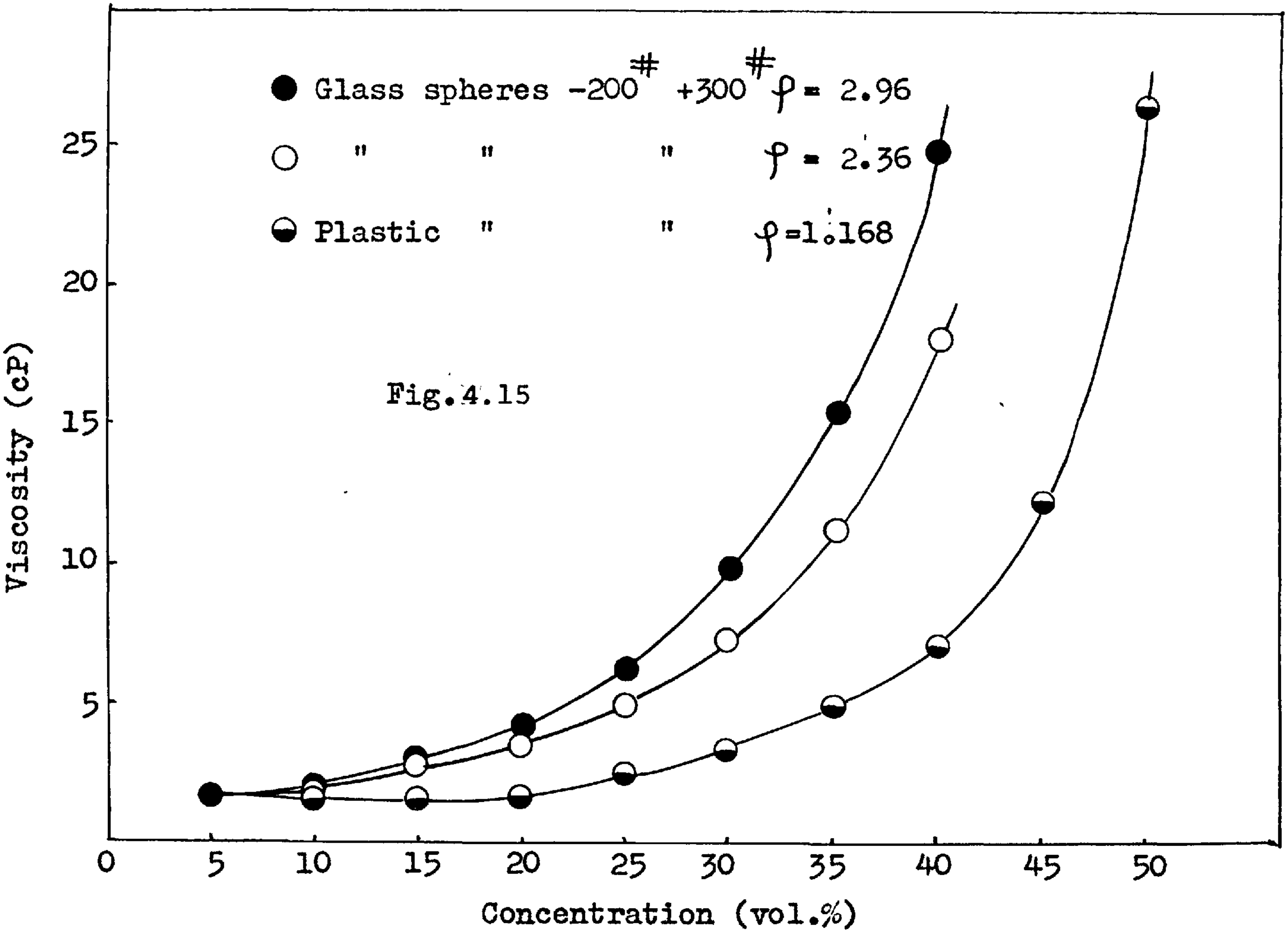
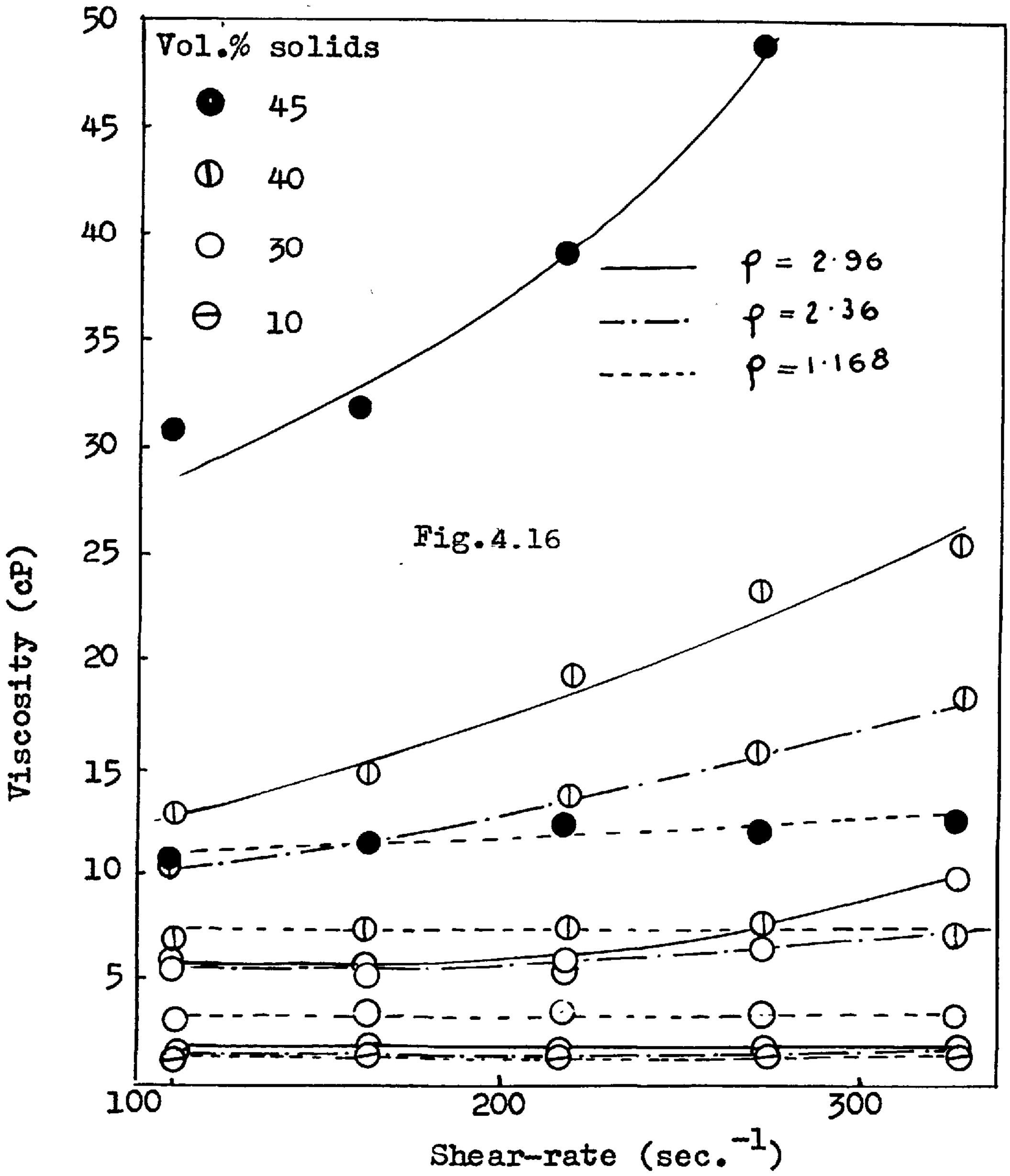
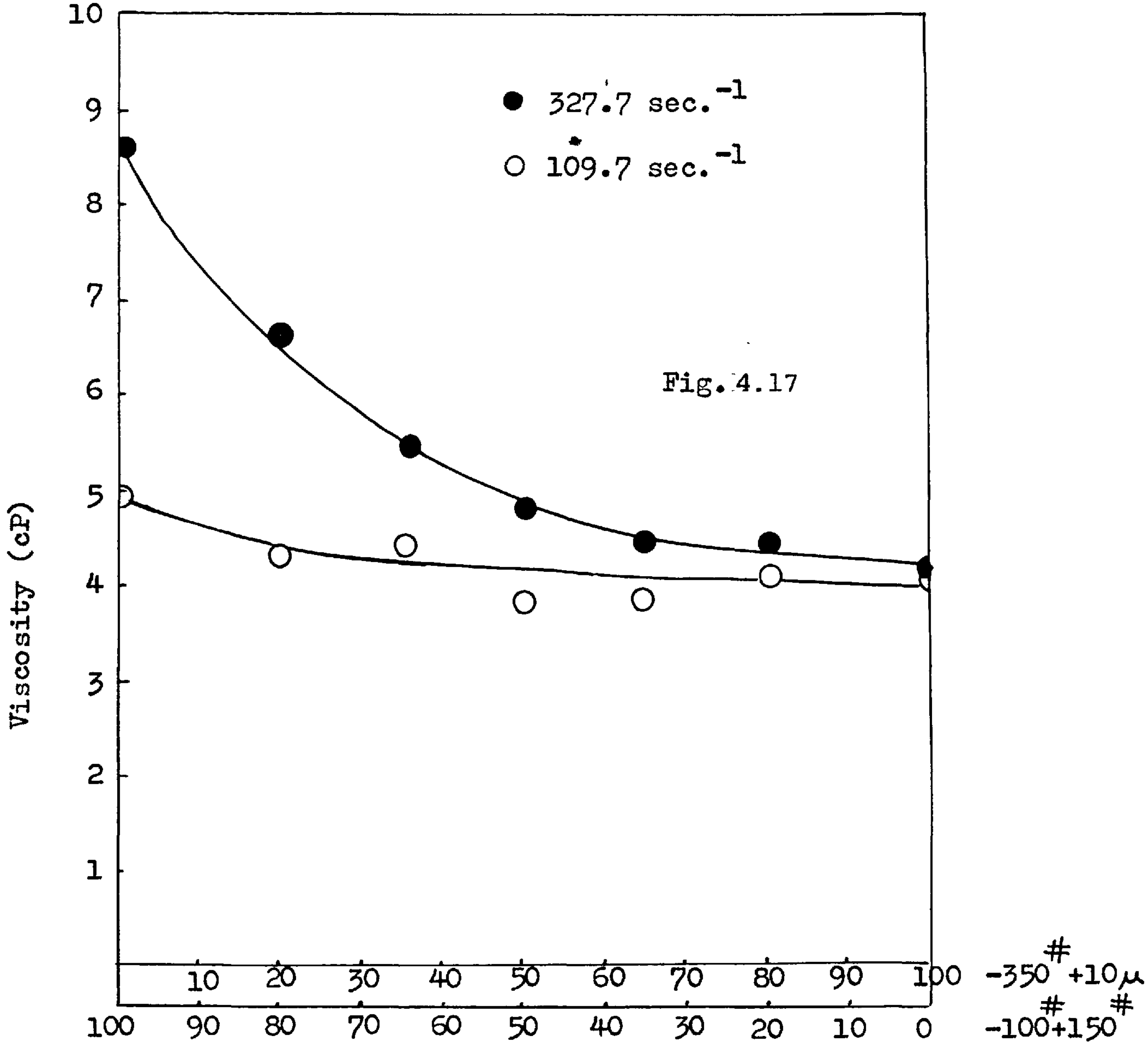


Fig. 4.15

Effect of concentration on viscosity using glass and plastic spheres at a shear-rate of 327.7 sec.$^{-1}$.



Effect of shear-rate on the viscosity of suspensions of glass spheres and polymethylmethacrylate spheres.



Effect of varying proportions of two size fractions of quartz at 20% total solids content and at two shear-rates.

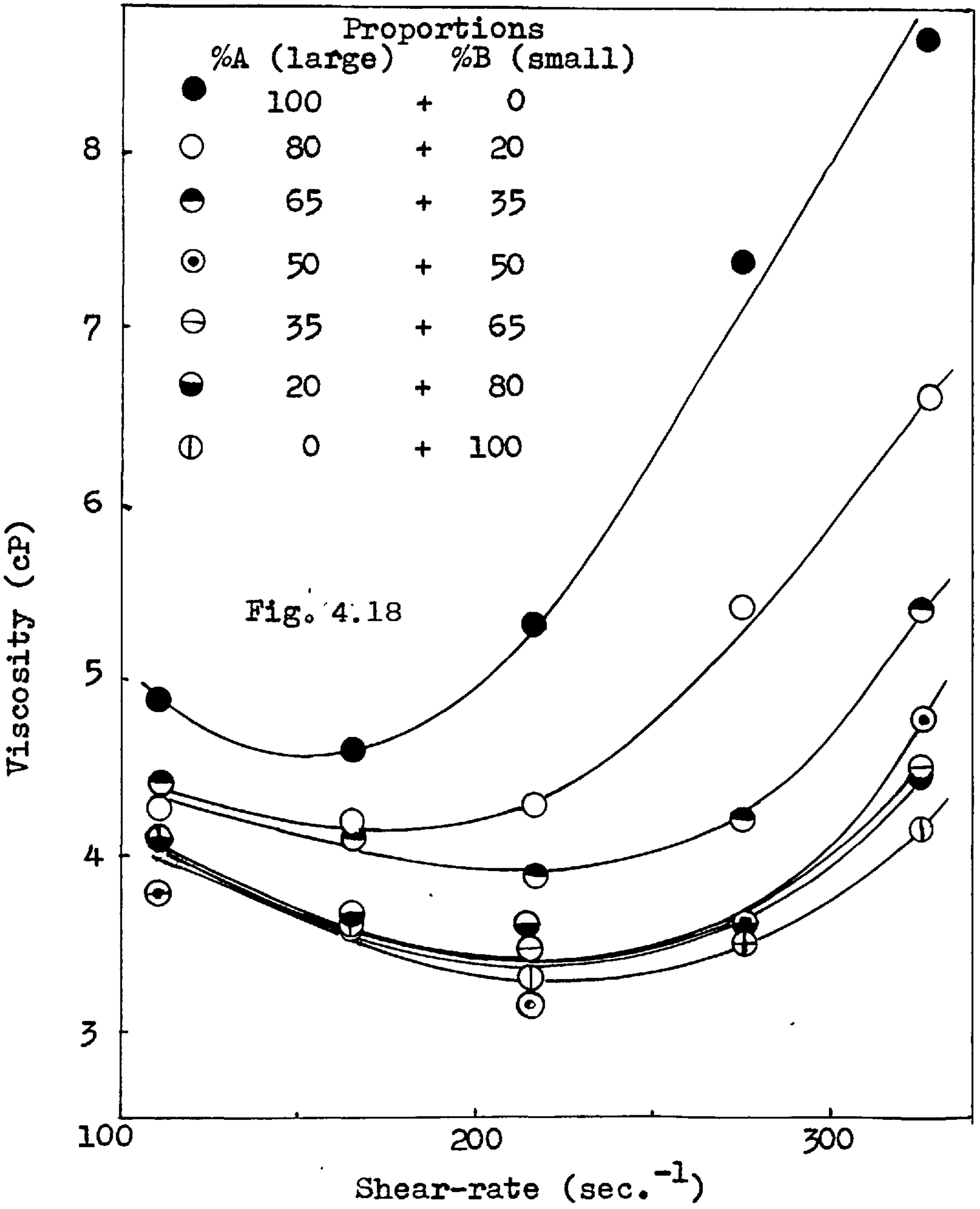
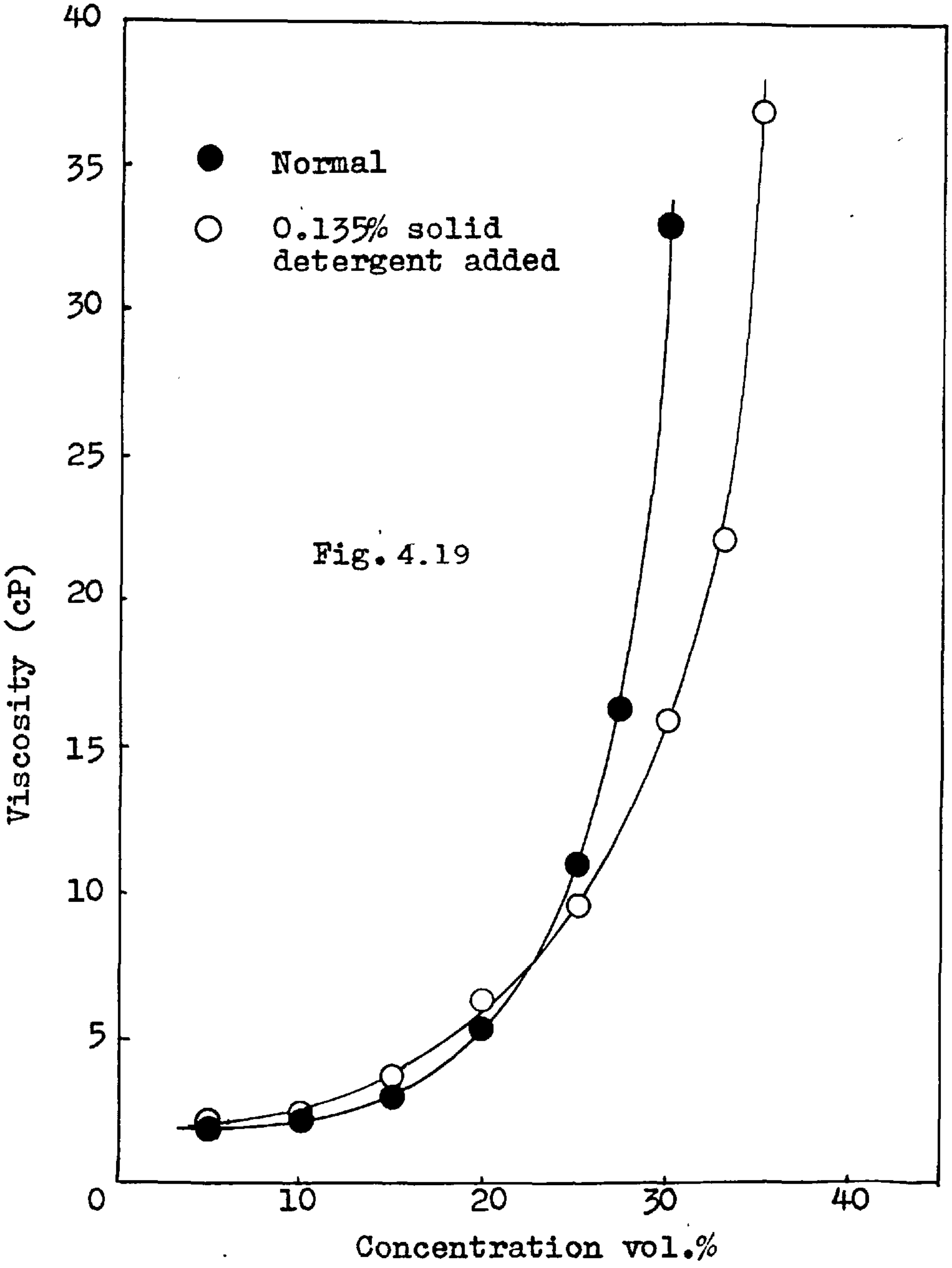


Fig. 4.18

Effect of shear-rate on size mixtures of quartz.
(A = -100$^{\#}$+150$^{\#}$: B = -350$^{\#}$ +10μ), at 20 vol.% solids.



Effect of anionic detergent addition to $-200^{\#}$ $+300^{\#}$ quartz (shear-rate 327.7 sec.$^{-1}$)

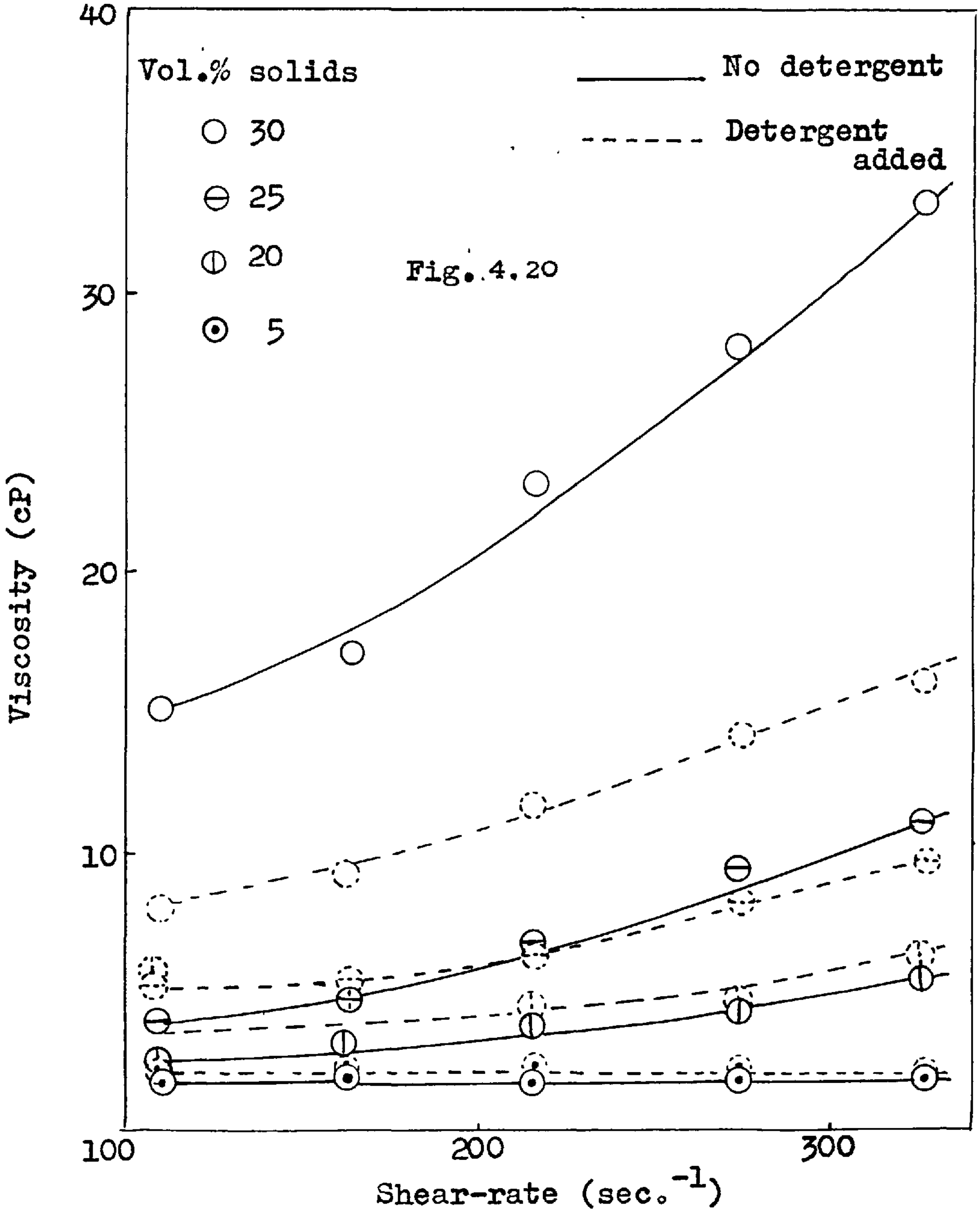


Fig. 4.20

Effect of shear-rate on $-200^{\#}+300^{\#}$ quartz with and without 0.135 vol.% solid detergent.

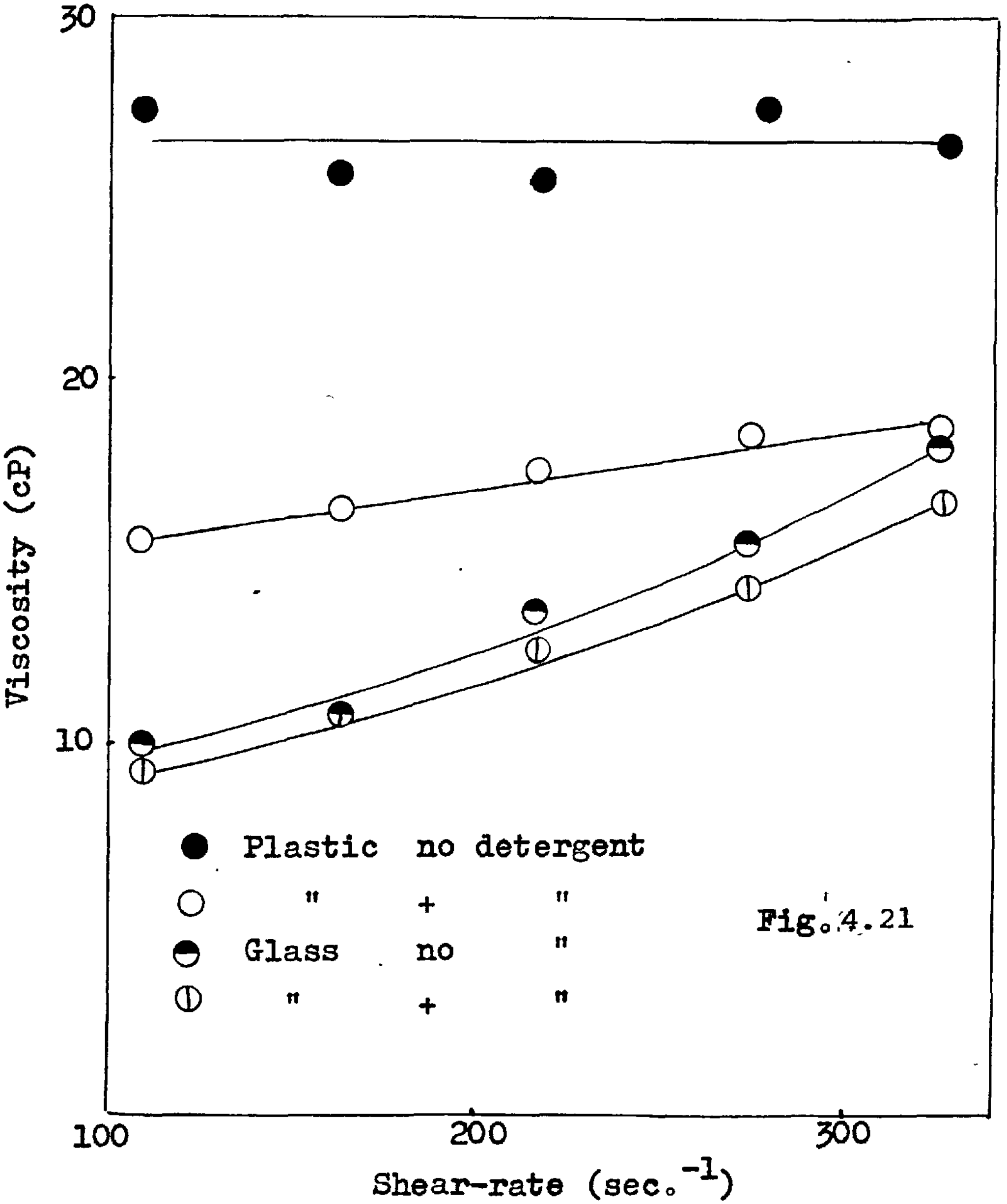


Fig. 4.21

Effect of shear-rate on viscosity with glass spheres ($\rho = 2.36$, $-200^{\#} +300^{\#}$) at 40 vol.% solids and polymethylmethacrylate spheres ($\rho = 1.168$, $-200^{\#} +300^{\#}$) at 50 vol.% solids, both with and without 0.6% active detergent.

Chapter 5.

INFLUENCE OF VISCOSITY ON BALL-MILLING.

The general opinion has commonly been expressed (103 to 106) that the viscosity of ball-mill pulps should be sufficiently high to provide a fairly coherent, even coating of medium on all the metal surfaces within the mill, and yet permit full mobility of the balls. If the pulp is too dilute, then the particles settle and centrifuge outward; the coating of the balls becomes patchy and voids between the outer layers of balls become overpacked, resulting in a "choked grind" (106). In addition, according to Taggart (104), with a dilute pulp, a large percentage of balls will be above the surface of the pulp, leaving only a small proportion of the balls to crush the material. Conversely, the more viscous the pulp, then the thicker the coating, and the more particles will be crushed, provided the balls are heavy enough, and the impacts are not cushioned.

Practical operators usually control the 'consistency' of the charge empirically, by variation of the pulp density. Studies have revealed, however, that viscosity is not simply a function of pulp density. Many other factors are involved which have to be considered, such as the actual dimensions of the mill, power absorbed, speed of rotation, density of, and friction between the grinding bodies, nature of the material, temperature rise, etc..

Investigations made by Fischer (107), on the rate of pigment dispersion, revealed that, as the diameter of the mill increases, the viscosity may be increased without loss of ball mobility. Viscosities of 200 poises are permissible in mills of 5'-0" diameter, while laboratory mills of about 6" diameter operate well at 10 poises. He notes that mills using pebbles or porcelain balls should be charged with pulps of lower viscosity than those using heavier, steel balls. One would not expect to encounter viscosities as high as 200 poises when grinding mineral ores in an aqueous medium, except under extreme conditions, although the same principle holds. Fischer (107) pointed out that the flow properties of a pulp may change as the comminution progresses. The investigation of viscosity, described in Chapter 4 of this thesis, also illustrates the effect of particle size reduction on a suspension of mineral particles. Apart from changes of particle size, there may also be changes of particle shape, size distribution and degree of dispersion, each of which may alter the viscosity of the pulp.

Variations in temperature may have a small effect, although the oleoresinous media studied by Fischer (107) were more susceptible to changes in temperature than the aqueous pulps normally used in mineral dressing.

Fischer (108) found, when dispersing pigments, that variations in viscosity could cause changes in the power requirements of a mill. He obtained very little change in the power absorbed up to 2000 cP, but further increases in viscosity up to 2250 cP caused increases in the power consumed of about 30%.

Gow, Guggenheim, Campbell and Coghill (109) recorded increases in power from 1.3 to 1.5 h.p., when the concentration of solids was increased from 64 to 80 wt.%, although they do not quote values of viscosity. Taggart (110), on the other hand, stated that thick pulps crowd the ball charge nearer to the mill axis, shortening the lever arm of the charge and consequently reducing the power required. Of these conflicting viewpoints one would expect Gow et al (109) to be correct because of the enhanced adhesion of the balls to the liner. This produces a higher ball trajectory and requires additional potential energy. When the viscosity reaches a point at which the balls adhere permanently to the shell of the mill, then part of the charge is spread over the shell, and is thus balanced about the axis of rotation. This causes a drop in the power consumed (111). Conversely, one would expect a fluid pulp of small particles to "lubricate" the ball charge and so reduce its equilibrium displacement, with a consequent drop in the power consumption. These factors have led some to the conclusion that the most efficient grinding takes place when there is a maximum power consumption (112).

Rose and Sullivan (113) include a viscosity factor in their formula for the calculation of the power requirements for mills, assuming a linear relationship between power consumption and viscosity: this is based partly on the results of Gow et al (109).

The trajectory of a ball charge is, therefore, a function of viscosity and speed, in addition to particle size, coefficient of friction etc.. Accordingly, Kendall (114), in his formula for predicting the optimum operating speed for ball-mills with smooth liners, includes a consistency factor 'c'. This factor varies between 0.63 for viscous pulps and 1.5 for fluid materials. The speed (r.p.m.)

is given by:

$$S = \frac{140 \times M \times C \times F}{\sqrt{D}}$$

where D is the diameter of the mill, M is a factor dependent on the surfaces of the balls and the liner, F is a factor dependent on the diameter of the mill, and C is the consistency factor.

There is sufficient evidence to conclude, therefore, that investigations of the influence of viscosity on grinding efficiency, which do not take into account either the variation of the power or the optimum speed, are of dubious value.

Another phenomenon referred to by Rose and Sullivan (111) is "slumping" of the charge, caused by low viscosity; this results in a reduction in the power requirements. They suggest that it is probably in some way linked with the variation in frictional characteristics of the mill charge. They state that, for large mill fillings, the effects of the variation of the pulp characteristics would be largely in accordance with what would be expected to result from consequential changes in the coefficient of friction involved. It is conceivable, therefore, that the coefficient of friction is an interdependent variable as well as viscosity. Tests by Rose and Sullivan (115), however, on the effect of friction showed no significant difference. Values of the coefficient of friction which ranged between 0.3 and 0.05 were obtained by using clean, dry, steel balls alone or with molybdenum disulphide, oil or a small charge of quartz powder.

It is suggested by Rose and Sullivan (116) that, on purely mechanical grounds, dry milling may be regarded as comparable with wet milling, the medium having the viscosity and density of air. The horsepower absorbed by wet grinding is, however, generally quoted (117)(118) to be 60 to 90% of that required for dry grinding. This is contrary to the normal trend of viscosity effects on power and again appears to be a frictional effect. The need for further study on the influence of pulp viscosity on the coefficient of friction of the charge is, therefore, clearly illustrated.

An important function of the liquid medium is to disperse the mineral particles and prevent settling. From Stokes's law it may be assumed that the more viscous the pulp, the slower the settling rate of the heavier mineral particles. Mular (119) studied the effect of upward settling particles. He obtained reductions in efficiency by grinding quartz in tetrabromoethane rather than in water. It may, therefore, be concluded that any increases in efficiency, due to effects of viscosity, are not purely a matter of its buoyancy properties.

Experiments were conducted by Creyke and Webb (120) on the ratio of solids to water in cylinder grinding. Constant speed tests were carried out with a 16¼" diameter by 7¼" long mill, charged with porcelain balls. The material used for grinding was a soft flint (-16 +20 mesh), which was ground in water for 7 hours. The viscosity of the pulp was measured each hour by using a torsional viscometer. 100 c.c. was retained each time for size distribution analysis. No attempt was made to vary the optimum operating speed or measure the torque required to drive the mill. The results showed that there was an optimum solid-liquid ratio for maximum grinding efficiency. This was expressed in terms of pint weight and relative efficiency (E) (Fig.5.1) which is given by:

$$E = \frac{W(S_1 - S_o)}{W_1(N_1 - N_o)}$$

where $\underline{W}$ and $\underline{W_1}$ are the weights of the charges: $\underline{S_1}$ and $\underline{S_o}$

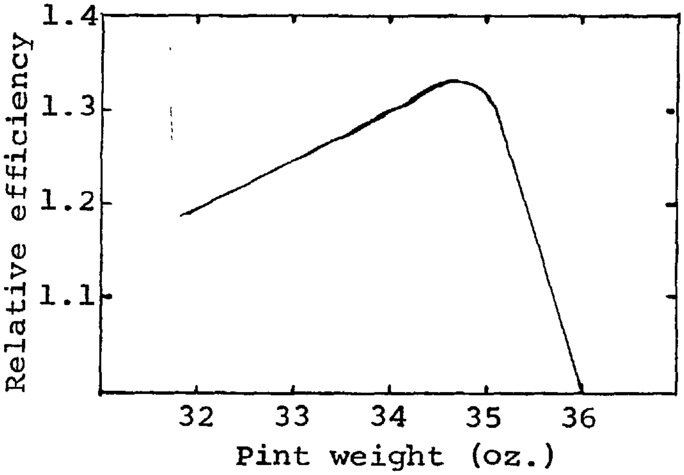


Fig.5.1 - Creyke and Webb.
Variation of relative grinding efficiency with pint weight of slip.

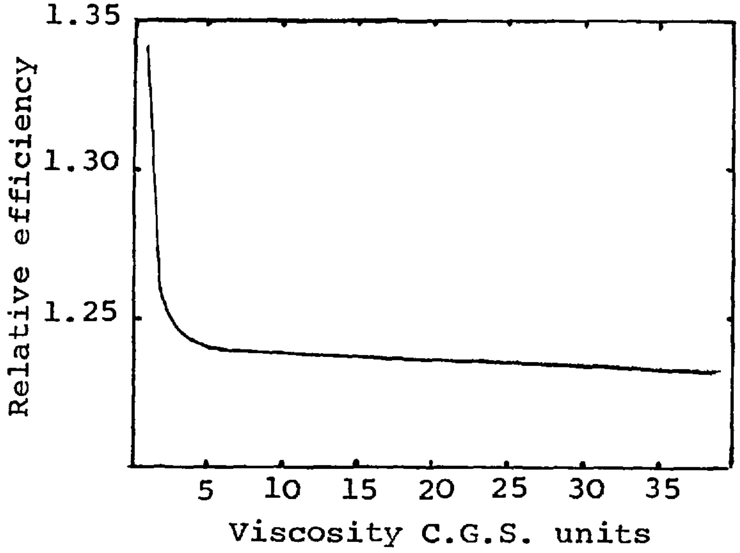


Fig.5.2 - Creyke and Webb.
Grinding at constant pulp density.

are surface factors before and after grinding weight $\underline{W}$. $\underline{N_o}$ and $\underline{N_1}$ are the corresponding factors for grinding $\underline{W_1}$.

The change of efficiency was, however, attributed to the viscosity which this optimum solid-water ratio attained. This hypothesis was tested by the addition of 5% clay. This lowered the efficiency while maintaining the same pint weight and increasing the viscosity. Further tests were done to examine the effect of viscosity at constant solid-water ratio by the addition of flocculants or dispersants. Fig. 5.2 shows that the maximum efficiency was obtained at the lowest viscosity and that efficiency fell rapidly over a narrow range of viscosity. Thus, by careful addition of a flocculant or deflocculant, the correct viscosity could be obtained, or, by the addition of a deflocculant, maximum efficiency could be maintained for a greater grinding capacity. The addition of a deflocculant was also recommended by Fischer (121) to prevent the formation of a pulp having a high yield value and hence, to permit maximum mobility of the balls. Materials such as stone and feldspar with inherently alkaline properties could also be used at greater solid-liquid ratio. Creyke and Webb found that the production of fines was greater at low viscosities. No allowance was made for variations in the power required or the optimum speed in any of these experiments.

An investigation was made by Schweyer (122) on the influence of the viscosity of the medium on the rate of grinding in pebble mills. Sand was ground in porcelain mills of one gallon capacity with aqueous glycerol media over a range of viscosities from 1 to 400 cP, for each of 4, 16 and 168 hours at constant speed. The products were analysed by means of a nest of sieves and a Wagner turbidimeter.

Schweyer differentiated between two phases of grinding — an initial rapid development of surface, and a subsequent grinding period during which the rate of surface development decreased. The initial phase was dependent on viscosity in that the impact grinding was faster for a less viscous medium up to 2000 revolutions; although there was little difference between 1 to 50 cP which Schweyer quoted as the normal commercial range of grinding.

Contrary to the work of Creyke and Webb, the fine grinding appeared to be independent of viscosity. Schweyer did not measure the energy input to the mill, however, because of the scale of the operation. He recognised the large error due to contamination from the grinding surfaces, especially with the longer grinding periods. The results were based on measurements of the viscosity of the suspending fluid rather than the pulp as in the present investigation.

The lengths of grinding time used by Schweyer were far longer than those of the present study, where shorter residence times have been used, as they are of more practical interest in the field of mineral processing.

Salman and Sirois (86) sought to establish relationships between density, surface area and viscosity of pulps in grinding. Quartz was ground in an Abbe - type ball-mill with water as the medium. The grinding time and power input to the mill were kept constant. (No mention of speed variation is made, however, under changing load conditions). A rotational viscometer was used, in a special tank with stirrer and baffles, to measure the viscosity of the pulp after the grind. The pulps were found to be non-Newtonian and dilatant in nature. The pulp density was varied between 5 to 70% weight of solids, producing consequent changes in viscosity from 6 to 205 cP.

Salman and Sirois found that the more dilute the pulp (and presumably the lower the viscosity) then the finer was the grind produced by the mill, in agreement with Creyke and Webb. This result was to be expected because of the increased ratio of ball to material weight. These workers found that small increases in the surface area at high concentrations of solids produced substantial increases in viscosity.

The role of pulp density in ball-mill operation was outlined by Salmân and Sirois. They stated that it affects the internal friction of the load, the load friction of the wall surface and the buoyant and spreading effects of the pulp on the tumbling media. The optimum operating requirement in a mill is the uniform flow of a pulp (which should be dense enough to maintain substantial homogeneity) over all the surface of the tumbling media. However, the roles of pulp density and viscosity largely overlap, and many of these effects are primarily caused by the viscosity of the media.

The effect of the viscosity of the suspending fluid on batch mill grinding was studied by Hockings, Volin and Mular (123). Constant speed tests, of up to 4 minutes duration, were made in a laboratory rod mill, with -10 +14 mesh quartzite. The viscosity of the suspending fluid was varied with corn-syrup -- water mixtures over a range from 1 to 1760 cP. No measurements were made of the pulp viscosity because it was thought that the grinding times were too short to alter the apparent viscosity appreciably. No allowance was made for any change in power requirements which may have occurred.

The results of the work of Hockings et al are illustrated in Figs. 5.3 and 5.4. Although considerable scatter is evident in the graph for 60 seconds, this curve shows that at low viscosities most of the feed disappeared in the first grinding interval. The weight fraction broken was found to be independent of viscosity up to about 20 cP but decreased at higher viscosities. For grinding times larger than 60 secs., the weight fraction broken was independent of viscosity up to about 55 cP, but decreased at higher viscosities.

The size modulus 'k', from the Schumann (124) size distribution equation, $y = \left(\frac{x}{k}\right)^{\alpha}$, was plotted against the viscosity (Fig. 5.4). It was found that, again, the formation of finer sizes was relatively independent of viscosity up to 50 cP and decreased at higher levels. The decrease in grinding efficiency with increasing viscosity

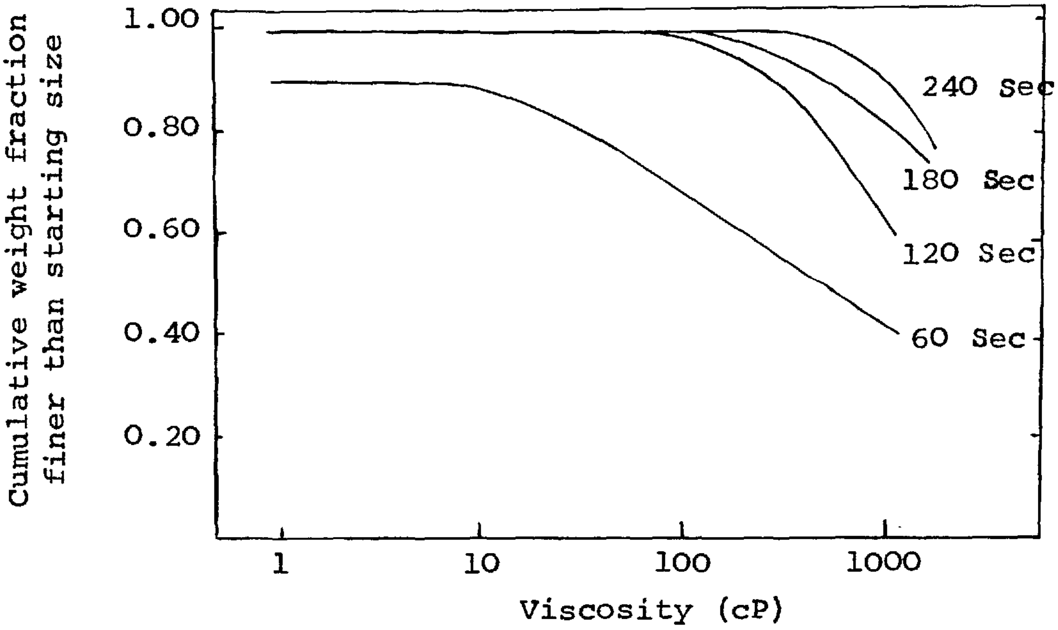


Fig.5.3 - Hockings, Volin and Mular
Efficiency of grinding quartz in a rod-mill.

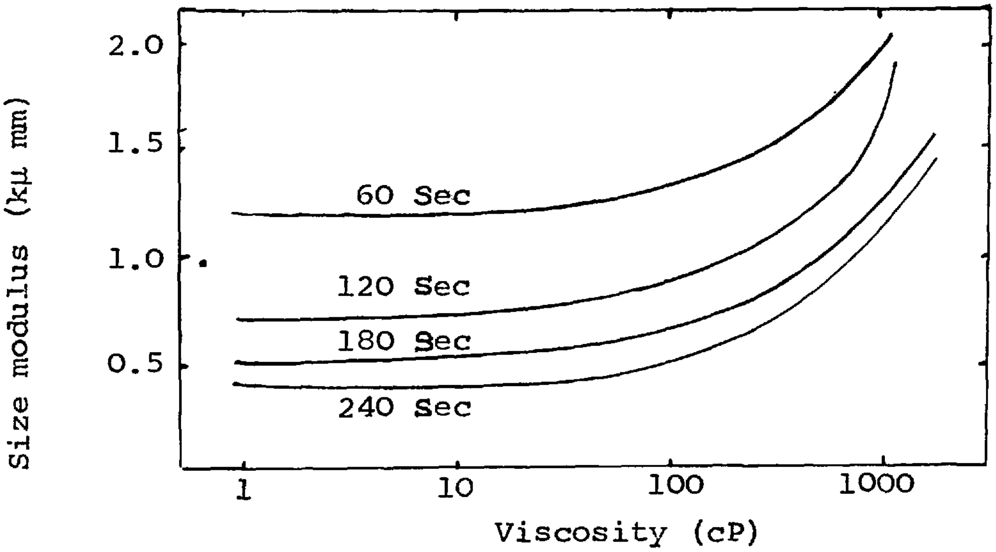


Fig.5.4 - Hockings, Volin and Mular
Variation of size modulus with viscosity of pulp when grinding quartz in a rod-mill

was attributed to four possible effects. The inhibition of settling of the particles meant a reduction of the amount of pulp at the shell of the mill which would result in a reduction of efficiency, if that was where much of the grinding took place. Much of the material that was at the periphery of the mill was taken up the ascending mill wall and out of the grinding zone. Also, increasing viscosity would cause a decrease in velocity of the rods in the pulp, causing a reduction in the frequency and energy of impacts. It was also suggested that in the range where grinding was independent of viscosity (0 to 50 cP), the flow was turbulent and the drag forces were independent of viscosity, and so the probability of capture of a particle by a rod would also be independent of viscosity. In the higher range of viscosity, laminar flow prevails, where the drag forces on a particle, and also the grinding efficiency, would be dependent on viscosity.

The reasons suggested by Hockings et al are surprising in some ways. One would expect most of the grinding to take place between the rods, and both the coating of the rods and the mill surfaces ought to cause an increase in grinding efficiency. Secondly, a reduction in the energy and frequency of impacts is unlikely at values of viscosity of 50 cP. On the contrary, the higher rod trajectory would cause the opposite effect owing to the greater falling distances. There would also be an increase in the frequency of impacts, as more of the grinding bodies were taken up the wall of the mill by the increased adhesion, rather than undergoing pure rotation. Adherence to the shell and cushioning could, of course, predominate at the highest viscosities used.

The work of Hockings and co-workers is the most nearly comparable with that described in this thesis (which was planned and mostly carried out before the publication of that work). Although the work of Hockings et al was with a rod-mill rather than a ball-mill, many of their conclusions are applicable to either. The reason for many of their results was the influence of the viscosity of the pulp on the optimum operating speed of the mill, by affecting the trajectory of the rods. In order to study the influence of the viscosity of the pulp on breakage alone, experiments must be done at equivalent rod or ball paths.

In conclusion, the influence of the viscosity of the pulp on grinding in ball-mills is complex, involving almost every other variable in the milling process. The extent of its influence on the dynamics of the charge and on the different phases of grinding is obscure. It is known that the viscosity of the pulp, or the suspending fluid can cause differences in the trajectory of the balls, and hence alter the optimum operating speed and the power consumed by the mill.

The influence of viscosity appears to cover four main phases of grinding for a particular size of mill; namely, impact and attrition grinding at high and low viscosities. The rate of production of fine material is generally agreed to be higher at low viscosities, whereas the impact breakage is independent of viscosity in the 0 to 50 cP range. In the higher ranges of viscosity impact breakage decreases, while the results of different workers do not agree on the rate of abrasion grinding. Creyke and Webb(120) agree with Hockings et al (123) that there is a point beyond which further increases in the viscosity cause decreases in the grinding efficiency. Schweyer, on the other hand, claimed that the rate of production of fine material was practically independent of the viscosity of the medium.

The amount of work done on this aspect of grinding is very limited. The few results available are not sufficiently comprehensive to yield a clear answer. Little work has been done to control the viscosity of the pulp by means of additives because of the lack of appreciation of the importance of the role of viscosity in grinding.

Further work is required to clarify the results already obtained in fine grinding, using not only artificial media such as glycerol and corn syrup,but practical grinding media. Breakage of coarse material (>50 µ) is of greater interest to the mineral technologist, and has been largely neglected in ball milling in all viscosity ranges. This aspect is therefore particularly worthy of further attention.

The explanations offered for the influence of the viscosity of the pulp on the mechanisms of breakage appear plausible. Results have been obscured, however, by the influence of viscosity on the optimum operating speed of a mill. Hence, effects of coating, turbulence, cushioning, impact frequency etc. have been difficult to isolate, and the explanations have been largely conjectural. The need for tests with variable speed and controlled power is, therefore, clear. When the influence exerted by the various factors enumerated above has been clarified, then an investigation will be required to seek to control the viscosity throughout the milling process by means of chemical additives.

Chapter 6.

THE DESIGN AND DEVELOPMENT OF THE MILLING APPARATUS.

The review of literature revealed the need of an experimental ball-mill possessing certain special features, which would permit a practical investigation into the influence of viscosity on grinding.

The apparatus needed to be large enough to simulate practical, industrial mills so that the scale-up factor would be practicable in the event of a wide application of the results. The measurement of power was expected to be simpler for a large, rather than a smaller, mill. Facilities for the control and measurement of speed and power were required under changing load conditions. Thus, changes in the power requirements as the grind progressed were anticipated as well as local fluctuations because of a shifting charge.

The means for a nominal, noise-level indication was needed to permit the study of any relationships between noise-level and viscosity, which may have offered an indirect method of viscosity measurement during operation of the mill. Several mechanical methods of continuous measurement of the viscosity of the pulp, as the mill operated, were considered but abandoned because of technical difficulties.

The final design of the mill and its associated instrumentation, which emerged after laboratory trials, is shown in Figs. 6.1 and 6.2, Plates 6.1 and 6.2 and as described below.

The inside dimensions of the mill were 18" long by 12" diameter. A length-to-width ratio of 1:1.5 would have permitted the use of rods instead of balls, had the need arisen, and it also minimised end effects. Fillets were welded into the inside corners of the mill of 1" radius, in order to prevent material from packing in the corners and so escaping grinding. The mill walls were $\frac{3''}{8}$ thick and smooth (rather than fitted with "lifters") in order to prevent changes in their lifting qualities throughout the project, and also to make the tests as general and reproducible as possible.

One end of the mill was a detachable door, locked into place by a key which screwed through a bar, fitted across the door, to apply a central pressure. The bar fitted into two brackets which were welded to opposite sides of the body of the mill. Thus, uneven fastening of the door

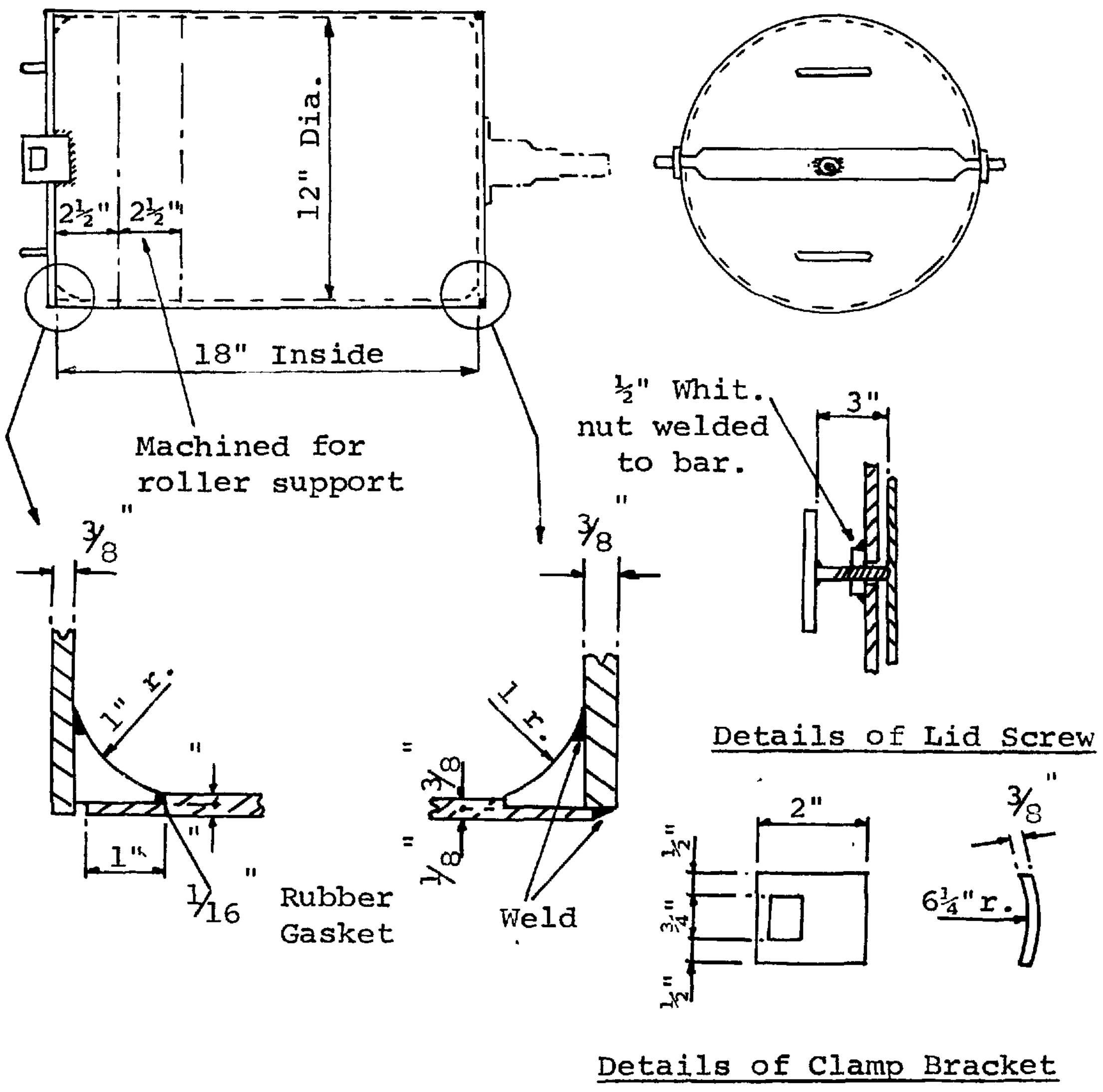
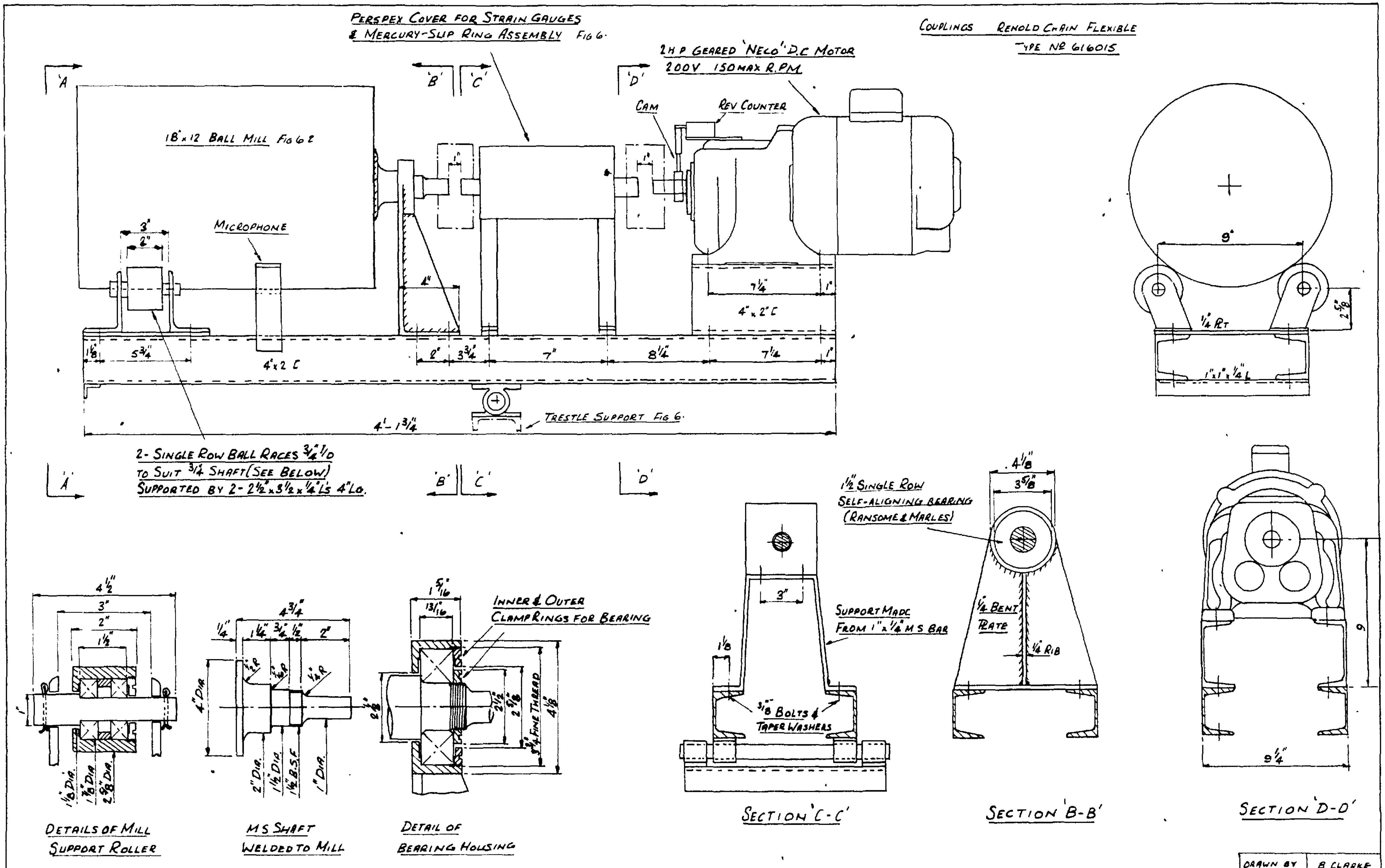


Fig.6.1 Details of 18" x 12" M.S. Ball Mill.



SUPPORTS & DRIVE FOR 18" x 12" BALL MILL - FIG 6·1

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DATE	10-4-63

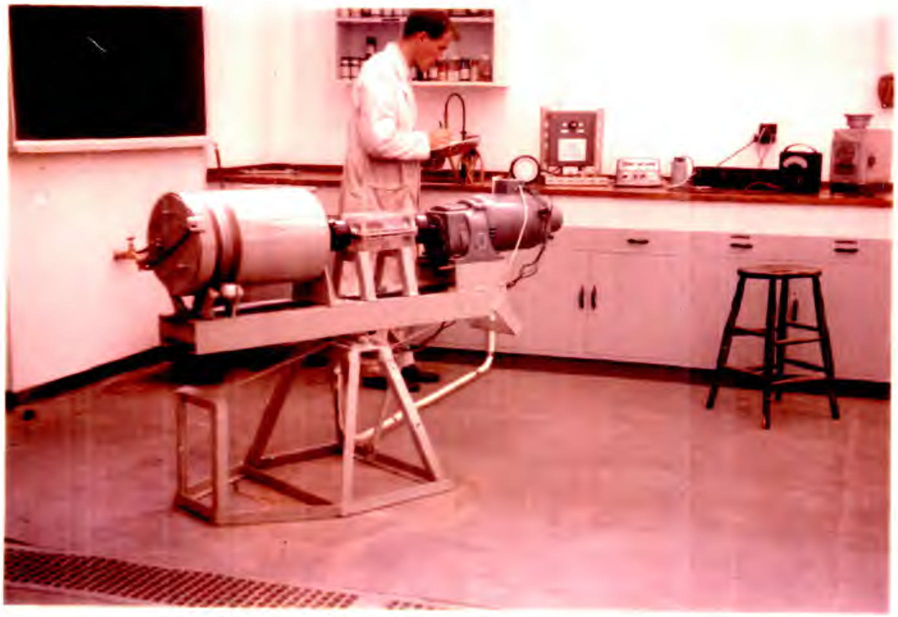


Plate 6.1

Experimental Ball-mill

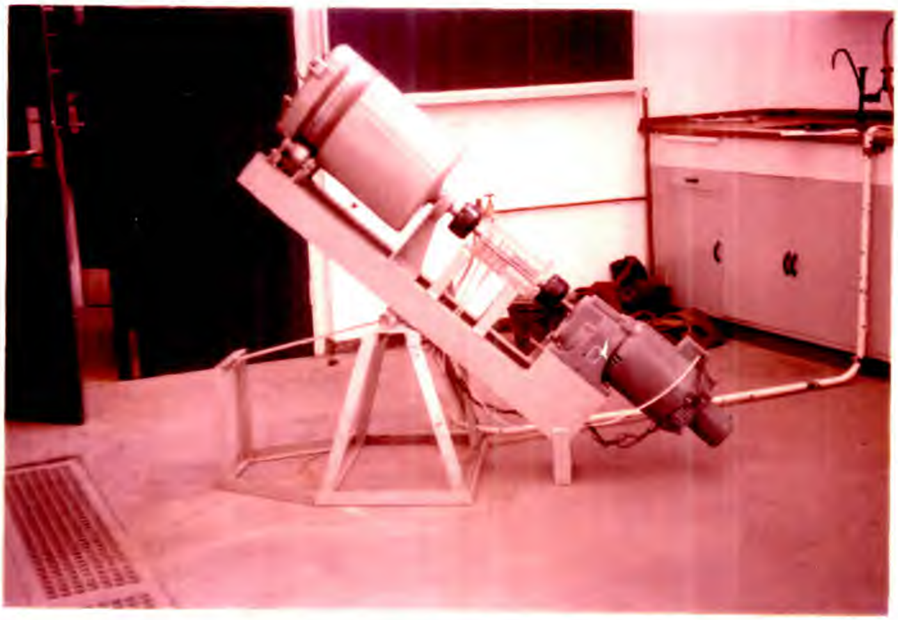


Plate 6.2

Ball-mill in charging position.

and consequent leakage of the medium was avoided. A rubber sealing-ring, which was fitted around the rim of the door, completed the water-tight compartment.

Provision was made for discharge of the material by replacement of the door of the mill by a perforated end-plate, which released the pulp but retained the balls.

The ball charge, including voids, under conditions of random packing (129), was calculated to fill 50% of the total volume of the mill. The charge, consisting of 168 lb of steel balls, was graded from $1\frac{1}{8}$" diameter to $\frac{9}{16}$" diameter, namely 5 lb of $1\frac{1}{8}$", 51 lb of 1", 56 lb of ¾" and 56 lb of $\frac{9}{16}$" diameter balls. In order to minimise the effect of size reduction of the balls after many tests, the total weight of balls was checked regularly, and new $1\frac{1}{8}$" and 1" balls added if necessary. The large balls were selected, round, smooth balls from old mills, while the smaller balls were reject ball-bearings, which were ground during preliminary tests to attain a settled surface finish.

The mill was supported on two 2½" diameter roller assemblies, one to each side of the mill. A 1½" diameter shaft, through which the apparatus was driven, was welded to the centre of the end-plate of the mill, and held by a self-aligning bearing. Thus, the mill was provided with three-point support.

The mill was driven by a 2 h.p., shunt-wound, d.c. motor (manufactured by Normand Electrical Co. Ltd.) through a reduction gearbox (supplied with the motor) with a maximum output speed of 150 r.p.m. The horse-power rating of the motor was well in excess of the theoretical power requirements, based on the maximum displacement of the charge at 150 r.p.m. The output speed of the gearbox was calculated to allow for over 100% slip of the charge. The theoretical speed for operation of the mill was estimated by equating the forces of gravity and centrifuging on the balls(126)(127).

Control of speed and power was provided by an electronic (transistorized) regulator ("Bercotrol" model 12, manufactured by British Electric Resistance Co. Ltd.). Closed loop control was obtained by feeding a 0 - 5 d.c. voltage back to the regulator from a d.c. tacho-generator (Type no.SA 740A-2, rating 7v/1000 r.p.m., made by Servo-Tek Products Co. Inc.). Fig.6.3 shows the electrical circuit for the regulator. The instrument could be operated manually to provide infinite speed variation between 0 and 150 r.p.m. ± 1% under changing load conditions. A constant torque could be provided, if necessary, under

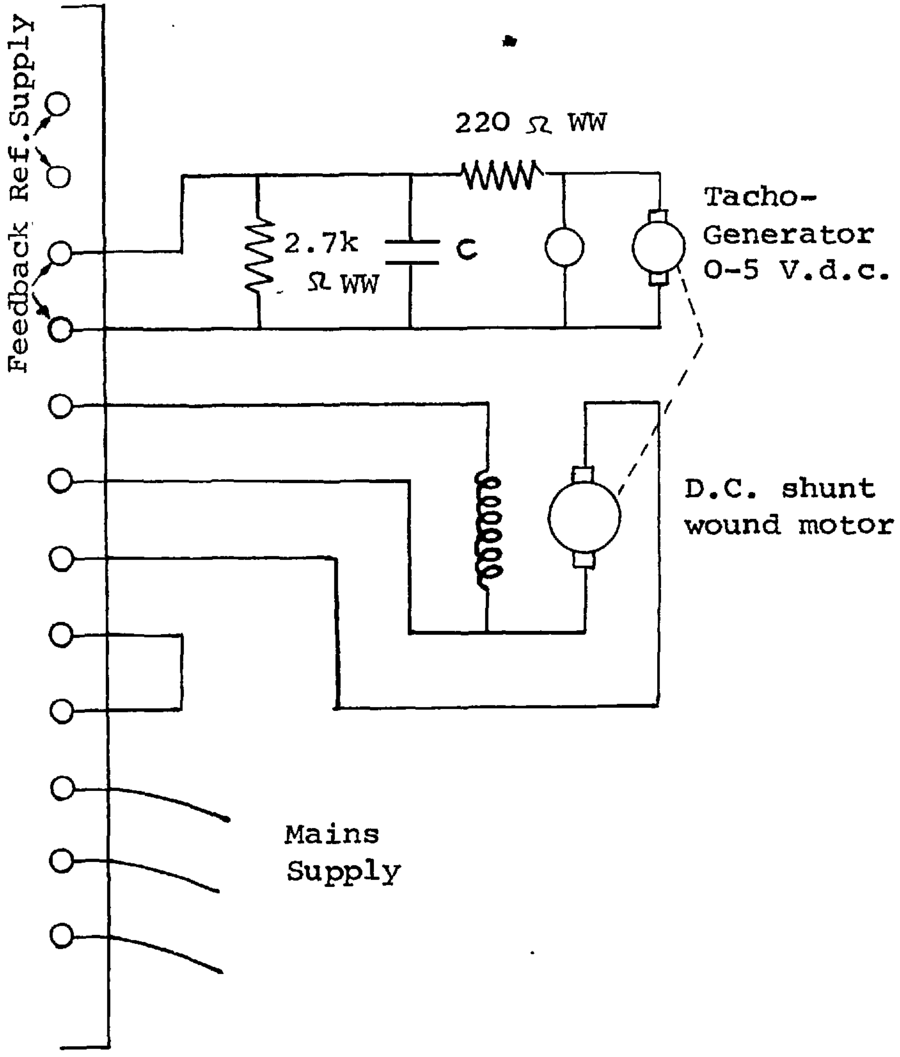


Fig.6.3 - Electrical Circuit for the Barcotrol power and speed regulator.

changing power requirements, with resultant variation of the speed, by adjustment of the "current limit" of the instrument. A calibrated voltmeter, connected across the tach-generator, provided a continuous indication of the speed of the mill. This was calibrated beforehand by reference to a stop-watch at the lower speeds and a mechanical tachometer at the higher speeds.

The power consumption of the mill was determined by torque measurements on a 1¼" diameter by 9" long, hollow, steel shaft. It was connected, by flexible couplings (made by Renold Chains Ltd. type no.616015) to the gearbox and the mill in order to include only power supplied to the mill body (excluding losses in the motor and gearbox). The torque was measured by means of four, matched, semi-conductor (128) strain-gauges, each of 357Ω resistance (manufactured by J. Langham Thompson Ltd., type no. 3A-1A-350N). The gauges were bonded around the shaft along its plane of maximum stress (i.e. at 45° to the main axis of the shaft(129)), as shown in Fig.6.4. They were connected in a Wheatstone bridge circuit (Fig.6.5) with two arms in compression and the other two in tension, thus producing the maximum sensitivity, and compensating for temperature and bending

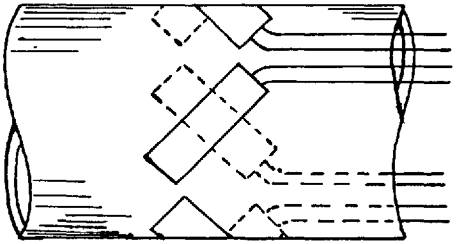
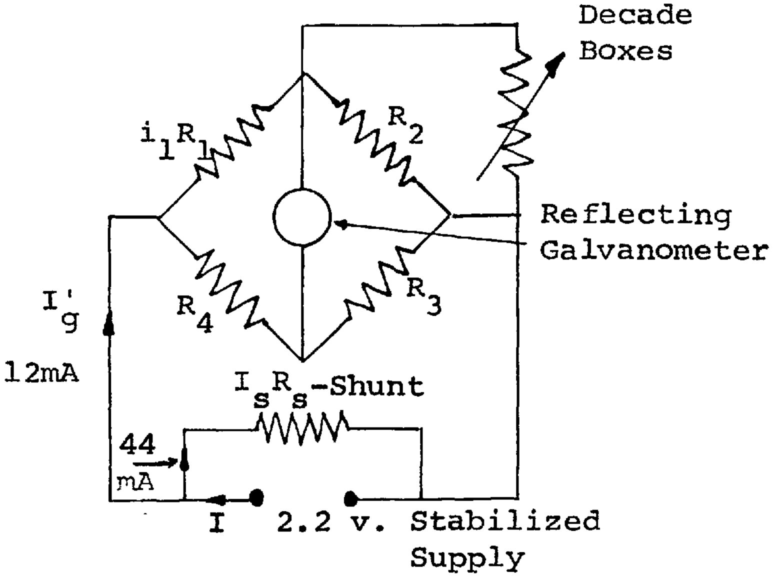


Fig.6.4 Arrangement of strain gauges on the shaft.



Gauges $R_1 = R_2 = R_3 = R_4 = 357\Omega$

Fig.6.5 Electrical circuit for strain gauges.

effects (130). The large signal produced by semi-conductor gauges obviated the need for amplification and permitted the use of a robust, reflecting galvanometer (Type 45, "Unigalvo", made by Evans Electro Selenium Ltd.) over the range 0 to 50 mV.

The gauges were bonded to the shaft diametrically opposite to each other, using epoxy resin. The shaft was prepared by abrasion with emery paper, and cleaned with trichlorethylene. A pressure of 2 lb/sq.in. was applied to the gauges while the cement was drying, using p.t.f.e. tape, $\frac{1}{32}$" thick (as advised by the gauge manufacturer), suspended over the shaft with weights attached to the loose ends.

Connections were made to the strain gauges by means of mercury-slip-rings (131). Four, 3" diameter, copper discs were clamped to the shaft, 1" apart, with "Perspex" spacers and two steel collars with grub screws. A "Perspex" tube, fitted over the shaft, provided insulation between the discs and the shaft. The discs rotated with their lower edges dipping in separate baths of mercury which contained fixed terminals. The whole strain gauge assembly and slip-rings were enclosed in a "Perspex" box for protection against accidental damage, and to prevent the ingress of dust into the mercury. The complete slip-ring assembly is shown in Fig. 6.6. The amalgam formed between the copper and the mercury, and deposited on the discs, was found to have no effect on their efficiency.

The minimum diameter shaft to which the gauges could be bonded was 1", according to the manufacturers' recommendations. A small margin of safety of ¼" was allowed, giving the outside diameter of the shaft as 1¼". The shaft was bored out to give a hollow tube of wall thickness $\frac{1''}{8}$, thus providing a shaft which gave a reasonable deflection within its elastic limit, which presented no machining problems and which transmitted the power requirements of the mill.

The inside diameter of the tube was calculated by the formula$^{(132)}$:

$$\frac{T}{J} = \frac{G\theta}{\ell} = \frac{q}{r}$$

where T is the torque, J the polar moment of inertia, G the modulus of rigidity, θ the angle through which the shaft is deflected, ℓ the length, q the shear-stress and r the radius of the shaft. The maximum static torque that the charge of 168 lb could exert was approximately $168 \times \frac{3}{12} = 42$ ft. lb. Allowing an arbitrary factor of

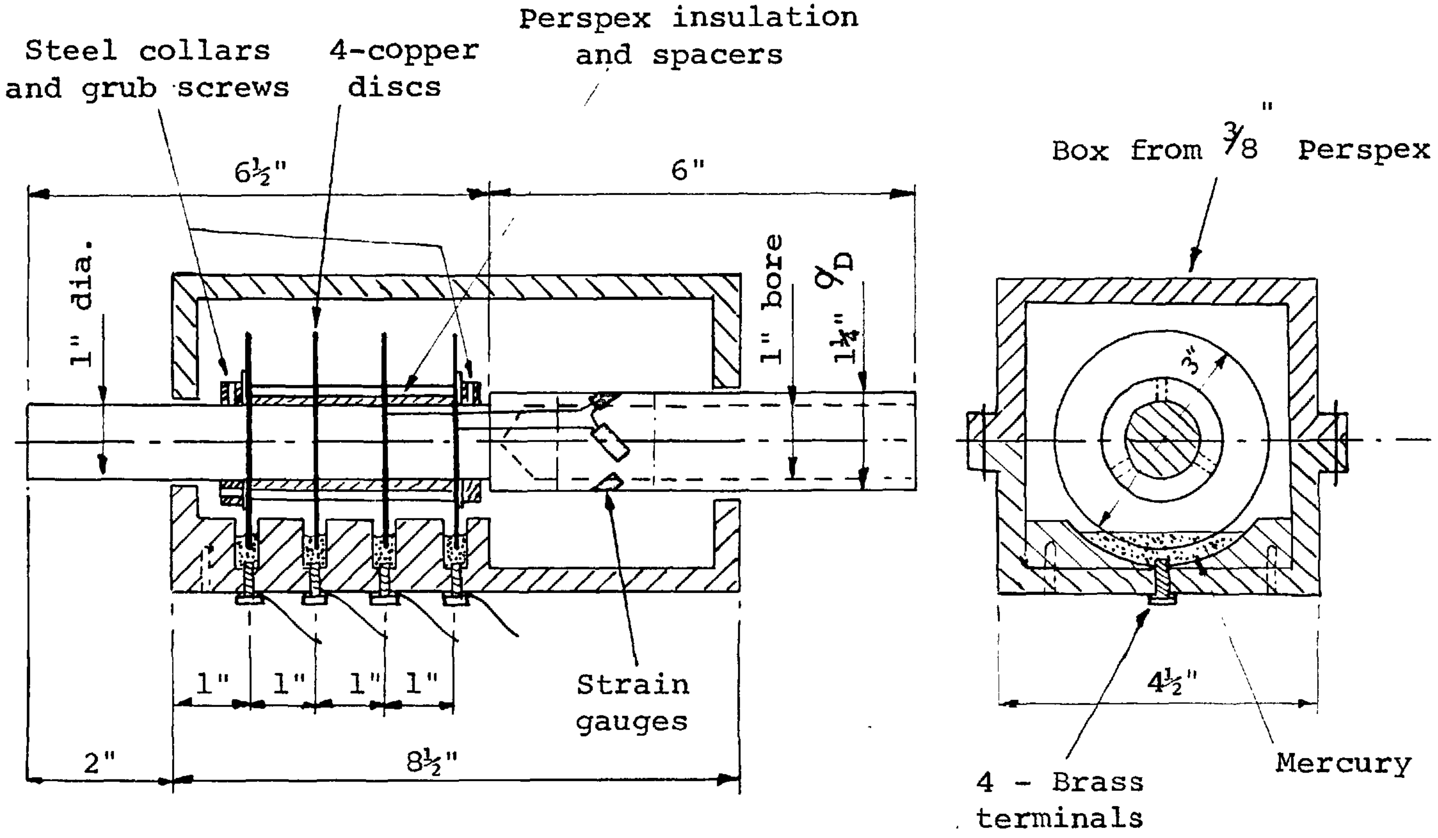


Fig.6.6 Arrangement of Mercury -Slip - rings on M.S. shaft.

safety of 6 for dynamic and acceleration loading, the maximum torque anticipated was 252 ft. lb., at a maximum speed of 150 r.p.m. The maximum shear-stress assumed for mild steel was one half of the tensile stress (133) = 14,000 lb/sq.in.

$$\text{Thus} \quad J = \frac{Tr}{q} = \frac{252 \times 12 \times 0.625}{14,000} = \frac{\pi\left(D_o^{\,4} - D_1^{\,4}\right)}{32}$$

$$D_1 = \sqrt[4]{1.25^4 - \frac{252 \times 12 \times 0.625 \times 32}{14,000 \times \pi}}$$

$$D_1 = 1.012'' \simeq 1''$$

The anticipated deflection was calculated from

$$\theta = \frac{T\ell}{GJ} \quad \text{where} \quad \theta \simeq \frac{\Delta L}{R}$$

for estimated running conditions requiring a torque of 40 ft. lb. at 100 r.p.m.

$$\therefore \Delta L = \frac{Tr\ell}{GJ}$$

where L = Gauge Length
ΔL = Change in gauge length
R = Gauge resistance
ΔR = Change in gauge resistance

$$\underline{\Delta L = 0.56 \times 10^{-4} \text{ in.}}$$

The gauge factor given by the strain-gauge manufacturers was

$$\frac{\Delta R}{R} \Big/ \frac{\Delta L}{L} = 110 \pm 2\%$$

$$\therefore \Delta R = 110 \times \frac{\Delta L}{L} \times R$$

$$\therefore \Delta R = 0.670\ \Omega$$

The recommended continuous working current for the gauges was 6 mA, which was provided by a mains operated, stabilized supply (manufactured by Croydon Precision Instrument Co., type no. P3/S), accurate to within 1 part in 20,000 after 30 minutes.

The optimum working load of the supply unit was 56 mA. A shunt of 50Ω was, therefore, connected in parallel with the strain gauge bridge to take the excess current. The bridge was balanced by means of a series of decade boxes which were connected across one of the gauges (Fig. 6.5). The circuit produced a deflection on the galvanometer of about 3", on its 50 mV range, for a torque of 40 ft.lb. The galvanometer deflection was calibrated with a torque arm, fixed to the mill body, on which weights were hung. The calibration was checked before each series of tests. The constancy of the gauges, over changes in temperature of 10°C, was found to be within ± 1%. The frictional properties of the rollers, however, were found to vary throughout the project, giving an absolute value of power measurement accurate to ± 5%, although tests within a particular series would attain accuracies within ± 2%, considering the calibration of the gauges, the sensitivity of the galvanometer and the estimation of an unsteady reading.

A revolution counter (made by Veeder-Root Ltd. type no.KA/1353) mounted on the gearbox, was operated by a smooth lift-and-lower cam clamped to the shaft (Fig. 6.7) The total number of revolutions required in an experiment could be preset on the counter, and a micro-switch connected to the motor automatically stopped the mill.

All the wiring associated with the motor, strain gauges, etc., was taken through conduit tubing to a bench, where all the instruments were assembled and from whence the mill was controlled.

In accordance with the theory that the noise produced by the mill is a function of the viscosity (134), provision was made for measurement of the sound level. An ordinary, crystal, audio microphone was fixed at 1" from the toe of the mill casing (135). The signal from it was amplified by a 3W, 12v, 3Ω audio amplifier (made by Newmarket Transistors Ltd., type PC5) which gave a response ranging from 0 to 2.0 mA a.c. measured on an "Avometer"

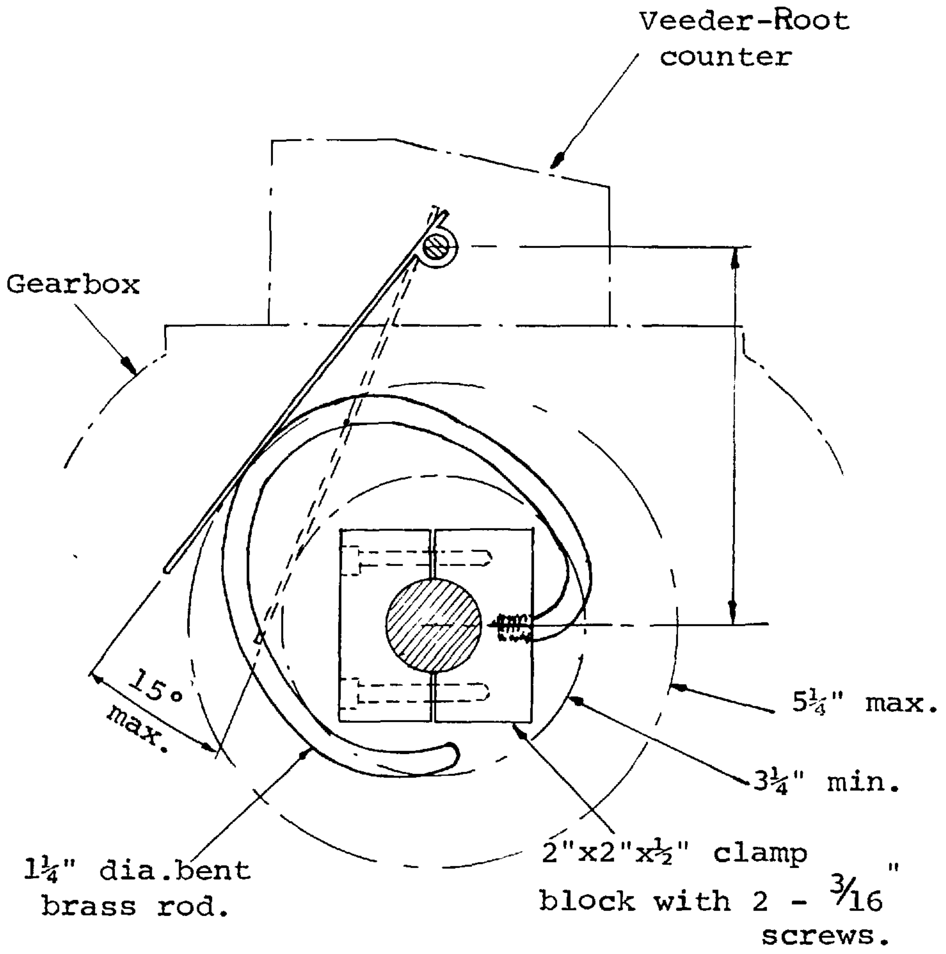


Fig.6.7 Arrangement of cam on gear-box to operate counter.

(model 8). The circuit diagram is shown in Fig.6.8. The meter did not analyse the tone or frequency of the noise, only the average intensity. The accuracy of the reading depended on a visual estimation of a fluctuating signal and was accurate to approximately ± 5%.

The motor, gearbox, torque-shaft and mill were mounted in line upon two 4" x 2" mild steel channel sections. The strength of these support beams was checked from a knowledge of the maximum bending moments (Fig. 6.9).

Referring to Fig. 6.9, and taking moments about X - X

$$200(1\tfrac{1}{2} + x) + 50x = 150(2\tfrac{1}{2} - x)$$

$$\underline{x = \tfrac{1}{4}\text{ft.}}$$

Hence, the maximum bending moment was given by:

$$(200 \cdot 1\tfrac{3}{4}) + (50 \cdot \tfrac{1}{4})$$

$$\doteqdot \underline{363 \text{ lb.ft.}}$$

Considering two 4" x 2" channels

$$\frac{M}{I} = \frac{f}{y}^{(136)}$$

$$f = \frac{363 \times 1.5}{3.91 \times 2} = 64 \text{ lb/sq.in}$$

Bending Stress.

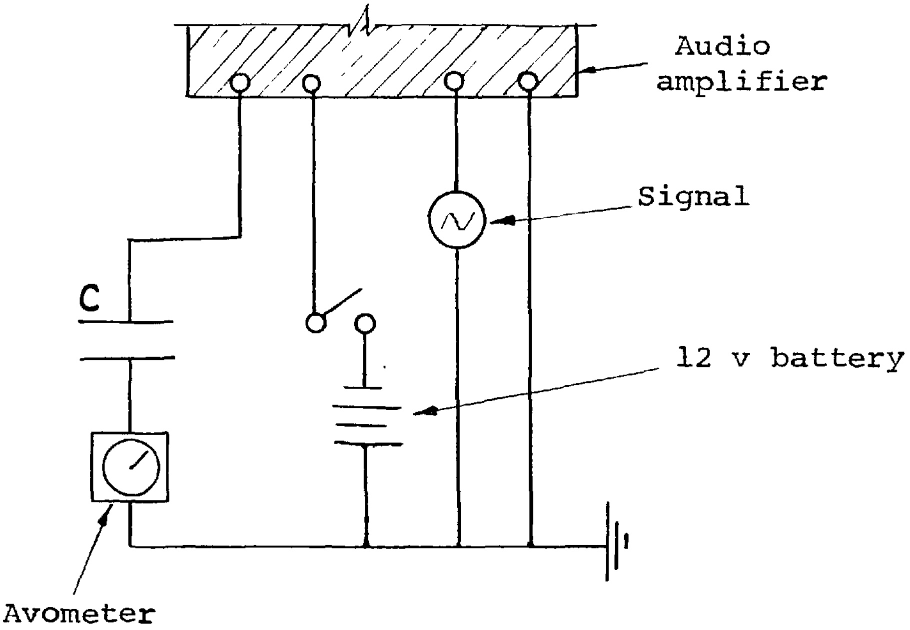


Fig.6.8 Electrical circuit for measurement of the noise level.

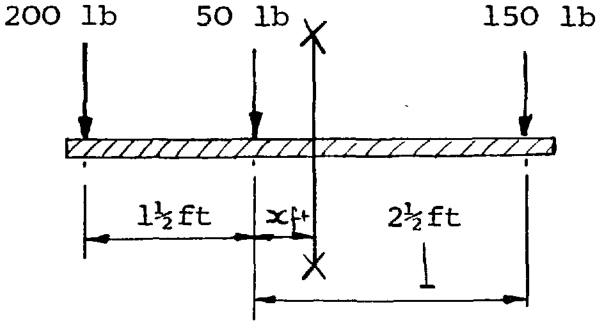


Fig.6.9 Loading diagram for the support beams for the mill and drive.

The maximum safe bending stress from B.S. 15 (1961)

= 22,400 lb/sq.in.

Thus two 4" x 2" channels were easily adequate from a strength consideration.

The support beams were clamped and balanced on a 1" diameter steel rod which was supported by a steel framework. Hence , the whole assembly could be tilted backwards, forwards or be held horizontal by a locking screw (Fig. 6.10).

The contents of the mill were discharged into a bucket which was hung, by its handle, on to the central door screw, (when the perforated plate was in place). The bucket swung vertically underneath the lip of the mill, independent of the position of the mill. When the apparatus was tilted forward the pulp poured into the bucket.

In order to avoid carrying out size analyses on the whole of the ground product, a wet-pulp splitter was used for the finer fractions (Fig. 6.11). A stirred reservoir drained through a small adjustable hole (maximum diameter ¼") into a 1" diameter rotating cup. A ¼" diameter pipe attached to the bottom of the cup swung in a circle, feeding the pulp into twelve adjacent cans which were constructed to fit together as sectors of a circle. Although the percentage

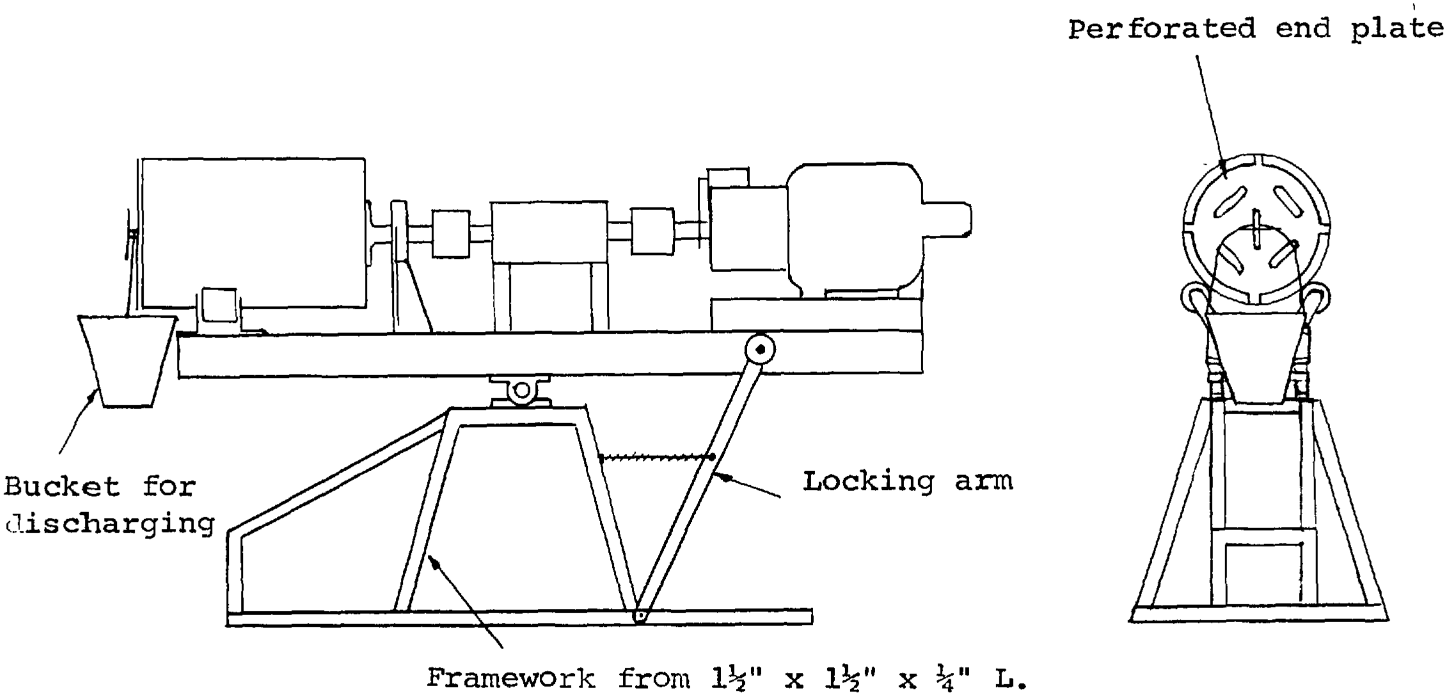


Fig.6.10- General arrangement of mill and support structure.

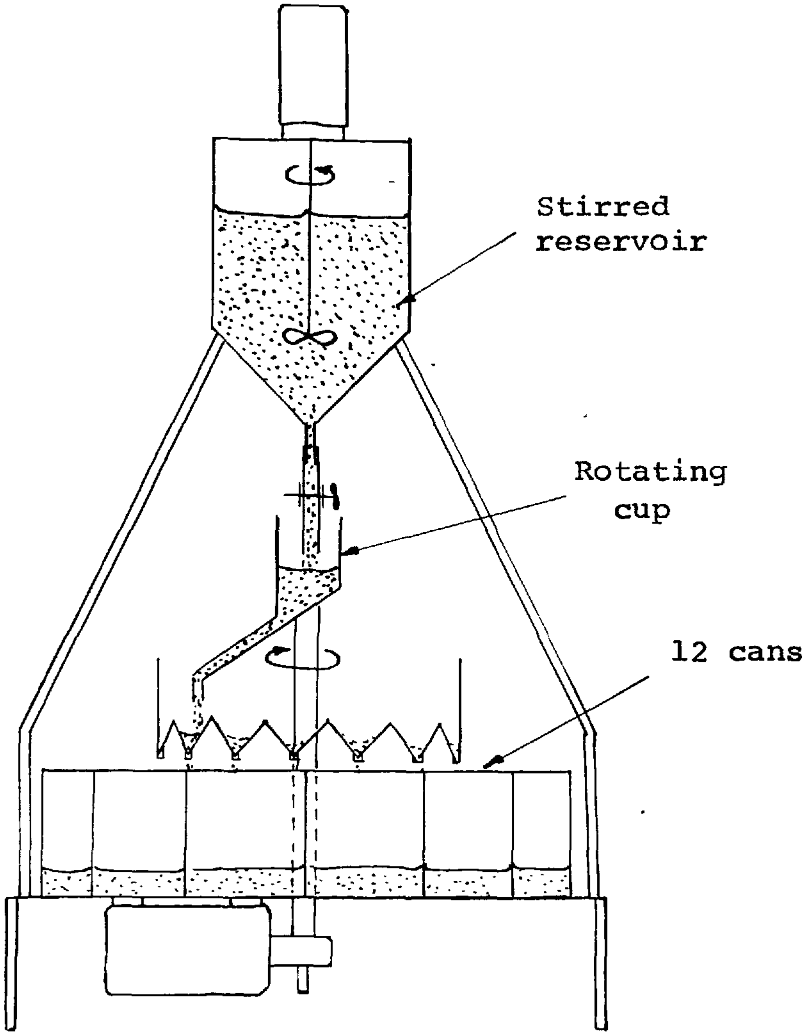


Fig.6.11 - Sketch of wet-pulp splitter.

weight in the cans varied slightly, the accuracy of the size analysis was tested and found to be representative of the whole sample, to within ± 2% or ± 3 gm on each size fraction.

A set of 8" diameter B.S.S. test sieves were used throughout the whole series for size analysis of the ground products.

Other laboratory equipment used during the milling tests were a screen shaker, a set of riffles for sampling the initial feed, an oven for drying the sample, and a laboratory balance of ± 0.1 gm accuracy.

Chapter 7.

EXPERIMENTAL - GRINDING.

Details of samples.

The standard charge used throughout the majority of the grinding tests was 1 kg of high purity Norwegian quartz of size -¼" +14 mesh. The quartz was received as a consignment of ½ ton of size -7/16". The requisite fraction was screened out, thoroughly mixed and sampled down to portions of 5 kg. These were stored in bags ready for use, and further sampled to 1 kg portions as necessary. Riffles and standard techniques (137) were used for the sampling process. A check was made of the amount of dust and fine particles present in the quartz owing to handling and attraction forces. A sample of 5 kg was wet-screened and the minus 14 mesh weighed: the fine material was found to be approximately 4.4 gm ($<$0.1%) which was considered to be negligible.

Many of the tests were conducted with certain artificial media in order to keep close control over the viscosity throughout the grind. Glycerol ($>$98.8% pure) was used mainly because of its ease of mixing with water, its similarity in density to that of water, its Newtonian property and its wide range of viscosities when mixed with water. The proportions of glycerol and water were prepared by volume and mixed in a bucket. The viscosity of the solutions was checked with the Ferranti viscometer (with an original set of cylinders), and adjusted if necessary.

The quantity of pulp used for all the tests was sufficient to fill the voids between the balls, namely, 6700 ml (both calculated and observed).

Other non-Newtonian, synthetic pulps were used to investigate their relative influence on the efficiency of grinding. These were constituted from starch, bentonite and calcium carbonate ("light") in water, producing dilatant, thixotropic and plastic media respectively. They were fully dispersed prior to testing, and their viscosities controlled by solid or water addition to be approximately equal (Fig.7.1). These viscosities were intended to be as near as possible to that which yielded the maximum efficiency with aqueous glycerol (i.e. 300 cP) in the tests at constant speed.

Further artificial pulps were made up from fines of quartz (<200 mesh) to produce a viscous slurry. This type of medium permitted a study of the breakage of a feed charge under more practical conditions. The fine material was prepared by sieving the -14# fraction from the original consignment and material recovered from previous tests. An 18" diameter screen was used on a mechanical shaker to prepare this material. Samples of the -200 mesh quartz were checked on the reference set of 8" B.S. screens.

Additions of an anionic detergent ("Quix" - 23% solid) and pine oil were used for certain tests in order to produce weakly plastic pulps of foam. Pine oil was also used in further tests in an attempt to influence the viscosity of the pulp by affecting the friction between the particles of quartz. Liquid concentrations of 1% and 0.01% were used for the detergent and pine oil respectively.

The final series of tests was carried out in water alone with full charges from 6 kg to 9½ kg of the original feed size of quartz (-¼ +14 mesh).

Experimental procedure.

The mill was prepared for use before each test by operating it with only the balls present. It was rotated at about 50 r.p.m. for 100 revolutions to loosen any rust which had formed on the grinding surfaces. A compressed air line was then directed into the mill to remove the rust.

This initial operation also served to warm the bearings and permit accurate checking of the instruments, and adjustment if necessary. The measured volume of medium (usually aqueous glycerol) was placed with the charge of quartz into the mill and its temperature taken. After setting the required number of revolutions on the counter, the mill was set in motion. It was brought as soon as possible to the operating speed (in about 15 revolutions) where it was maintained ± 1 r.p.m. throughout the remainder of the test. Observations were made of the torque required and the noise produced every 100 revolutions, until the mill automatically stopped. For the tests at constant energy, the average torque required throughout each test was estimated, and, from a brief calculation, the number of revolutions were adjusted, accordingly, to give a constant work input (usually 15,000 ft.lb.revs.). As soon as the grinding operation was complete, the temperature of the pulp was taken. The mill was tipped backwards and the door was replaced by the perforated end-plate. The bucket was hung in place and the pulp was poured into it by tipping the mill forwards. Measurements of viscosity were rechecked actually inside the mill, before discharging in a pool of pulp, in the tests with aqueous glycerol. A hose-pipe was used to assist the pulp in flowing out of the mill. The end-plate was removed when as much pulp as possible had left the mill, and the balls and remaining material were discharged into other buckets in a similar manner to the pulp. The hose-pipe was used again to wash the mill out carefully. The buckets containing the balls were filled with water, and the balls were washed manually in the supernatent liquid, before returning to the mill. Alternatively, for highly viscous and non-Newtonian media, the balls were washed in batches on a wire screen under a continuous stream of water. The screen was suspended over a large vessel which caught all the ground material. During the washing the balls were checked for cracking, deformation or excessive wear and replaced if necessary. The balls and the mill were dried as quickly as possible, by means of an electric fire, in order to minimize the formation of rust.

The contents of the buckets were wet-screened (52 mesh) to remove all the coarse material which might block the pulp splitter. The coarser fraction, which normally represented about 15% of the charge, was dried and given a complete size analysis: the remainder was put through the pulp splitter. The contents of one of the cans (the same can being used for every test) were wet-screened (300 mesh) to remove as much sub-sieve material as possible. When aqueous glycerol was used, care was taken to remove all traces of it from the quartz by washing. A flocculant ("Superfloc 16" supplied by Cyanamid Co. Ltd.) was used to flocculate the fine material in order to assist the settling, before decanting the supernatent liquid. Both fractions of quartz were then placed in shallow bowls and dried in an oven at about 200°C. The plus 300 mesh material was fractioned after cooling, with a set of 8" B.S. test sieves for 15 minutes on an automatic shaker. This time was found to be adequate without causing excessive abrasion of the particles or wear of the screens. Finally, the complete size analysis for the whole charge was computed from the weights and analyses of the various fractions examined, and the weight of the initial feed charge

Details of grinding experiments.

Preliminary tests.

Before tests could be carried out with synthetic media, it was necessary to establish the charge of quartz required to simulate a full charge. This was done by observation of the speed at which the balls centrifuged. Hence, an indication was obtained of the movement of the grinding bodies. When no quartz was present in the mill, the centrifuging speed was beyond the maximum speed of operation (i.e. > 200 % slip at 150 r.p.m.). Small amounts of quartz caused marked reductions in the speed of centrifuging, until at ¾ kg a constant speed was attained. Additions of quartz beyond 1½ kg caused further slight decreases in the centrifuging speed: this was presumably due to viscosity-concentration effects. This result was checked at three viscosities, namely, 800, 200 and 1 cP. Previous tests on viscosity showed that below a concentration equivalent to 1½ kg (about 8½ vol.%) the solids of such a suspension have little effect on the viscosity. Therefore, 1 kg of quartz was used for all synthetic pulp tests. Hence, close control was kept of the viscosity of the media throughout the grinding operation, while realistic conditions of ball movement, friction, etc. were maintained.

A short preliminary test was carried out to discover a suitable grinding time which would produce neither excessive fine nor unground material. With a feed charge of 1 kg, this was found to be about 500 revolutions. This number was, therefore, used as a basis for many subsequent tests.

Tests on centrifuging speed of the balls.

The results in the literature and the preliminary tests revealed that the 'critical' speed (as it is commonly called) is influenced by the viscosity of the pulp. Tests were conducted, therefore, to study the variation of critical speed over a range of Newtonian viscosities from 1 to 885 cP. A charge of 1 kg of quartz was added to provide practical operating conditions. The onset of centrifuging was observed by the fall-off in noise level and torque requirements. A curve was prepared relating centrifuging speed to viscosity of pulp. This was used later for the tests run at a constant proportion of the centrifuging speed.

Tests at constant speed.

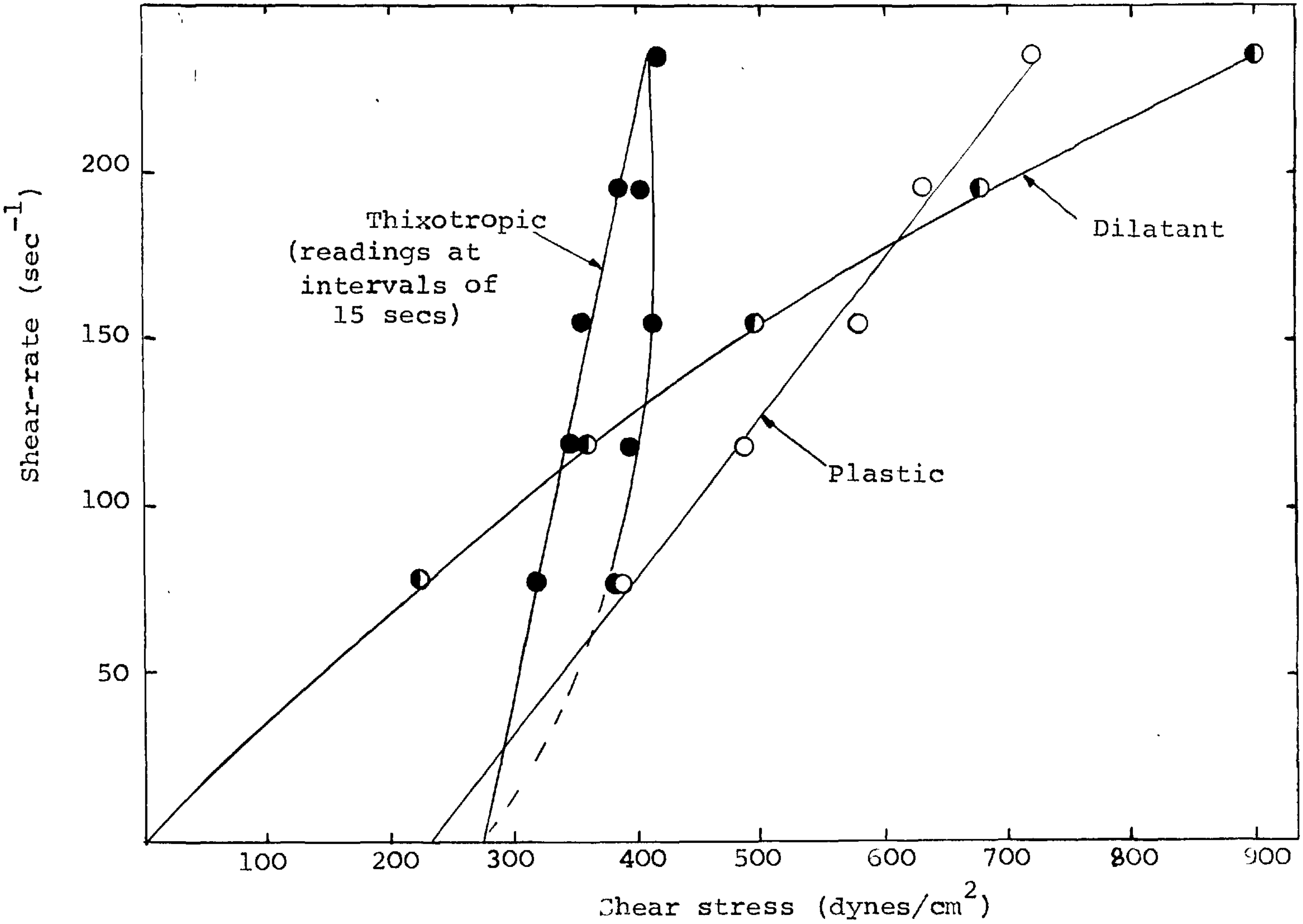
Charges of 1 kg of quartz were ground over a range of viscosities of aqueous glycerol from 1 to 760 cP at 70 r.p.m. for 500 revolutions. The purpose of this series was to study the influence of a range of Newtonian media on the rate of grinding. Furthermore, it was intended to lay a foundation for subsequent tests by obtaining a general understanding of the subject. The operating speed was based on the results from the centrifuging tests. The speed had to be below that which would centrifuge the highly viscous pulps and yet be as near as possible to 75% (138) of the centrifuging speed for the majority of the tests. Observations were made of the torque required and noise level after every 100 revolutions, and the temperature rise over the whole grind. A size analysis was carried out of the ground product.

The results from this series revealed that 300 cP was perhaps the optimum viscosity at which to grind. Constant speed tests at 70 r.p.m. were carried out, therefore, with non-Newtonian media at approximately 300 cP (at 110 sec^{-1}) (Fig.7.1).

Tests at a constant proportion of the centrifuging speed.

The results obtained so far, and those in the literature, indicated that a major influence on tests at constant speed was the change in the trajectory of the balls. The present series of tests was designed, therefore, to permit a study of the influence of viscosity on the rate of

Fig.7.1 Diagram showing properties of synthetic pulps for tests at 70 r.p.m.



breakage at similar ball trajectories. The experiments were carried out over a range of aqueous glycerol media from 1 - 685 cP. The constant speed tests were not directly comparable with one another because the energy input varied with each test. The variable speed tests, therefore, were carried out at a constant energy input of 15,000 ft.lb. revs, which was maintained by adjustment of the number of revolutions (usually around 500). The tests were run at 76% of the centrifuging speed of the balls. This percentage was chosen on the basis of the results of the previous tests because the viscosity which yielded the highest efficiency in the constant speed tests (300 cP) was run at 76% of the centrifuging speed. It was assumed, therefore, that 76% would be a suitable (although not necessarily ideal) running speed for all the variable speed tests.

Fines tests.

It was realised that the results of the previous tests were not directly applicable to practice, owing to the use of artificial media. It was considered essential, therefore, to repeat the last series of tests, using fine particles of quartz (-200 mesh), instead of glycerol, to constitute viscous pulps. The experiments were carried out at 76% of the centrifuging speed and with a constant work input of 15,000 ft.lb. revs. A range of viscosities was achieved by variation of the concentration of fine quartz. A check was kept of changes in viscosity throughout the grinding operation by measuring it before and after the grind.

An introductory test was necessary to discover the centrifuging speed of the balls over a range of viscosities with this type of pulp, in order to find the optimum operating speed. This preliminary test and subsequent "fines" tests were, again, carried out with a feed charge of 1 kg of quartz (-¼ +14 mesh). Size analysis of the ground product was not, of course, carried out below 200 mesh.

Full charge tests.

Having established a relationship between grinding efficiency and viscosity, using various closely controlled pulps, it remained to examine its validity under normal grinding conditions. A range of viscosities was achieved by variations in the pulp density. The question arose here, and to a small extent with the fines tests, of whether any effects observed might be due to changes in viscosity or just pulp density. The answer to this problem lay in the interpretation of the results. If the viscosity - efficiency relationship for this test coincided with that of constant pulp density tests, then it would be reasonable to assume that the relationship had been fully established. If the relationships had not coincided, then an alternative solution would have been sought.

A further problem was the change in viscosity throughout the grinding operation. The viscosity at the start of the test was assumed to be that of water (1 cP). Measurements of viscosity were not taken during tests because stopping and starting to extract samples would have upset the speed schedule. Furthermore, it would have been very difficult to extract a representative sample from the mill, without removing the complete charge. Viscosity measurements were made, therefore, after grinding, in the modified Ferranti viscometer and hence, can only be regarded as nominal. Also, only average measured values are plotted owing to the non-Newtonian nature of the pulps.

The experiments were carried out over a range of pulp densities with 6 - 9 kg of quartz. This represented a viscosity range of approximately 18 - 300 cP. Below 18 cP the charge was clearly too uneconomical: the main purpose of the series was to establish the viscosity - efficiency relationship over the latter part of the curve. This was with a view to a practical application of the results.

The tests were approximately four times longer in duration (about 2,000 revs.) than the previous tests. This was done to enable the mill to produce a viscous slurry from the feed charge, which was necessary before viscosity would be likely to influence the grind.

Preliminary tests were conducted to investigate the variation in centrifuging speed throughout the milling operation, for each pulp density. The initial feed charge was found to lock the balls together, causing them to centrifuge at a low speed. As the comminution progressed the centrifuging speed fell markedly. Now 76% of the maximum critical speed would have centrifuged the initial feed charge: the tests were carried out, therefore, at 70% of the maximum. The energy input for these tests was 50,000 ft.lb. revs.

Tests with foaming additives.

This series was prompted by the non-Newtonian pulp experiments. These suggested that a weakly plastic pulp which would just support unbroken material and coat the grinding bodies, would be the most efficient. Detergent was used to give an indication of the broad effects because of its greater foaming power. The pine oil was used, on the other hand, because the foam produced would collapse as soon as the mill stopped, instead of completely filling it - as did that from Quix. Furthermore, pine oil would be less likely to interfere with subsequent processes in a practical ore processing plant. Both additives were used for variable speed tests with fines, and the pine oil with tests having a full charge. Introductory experiments were conducted to find the optimum concentration of additive required to produce a foam capable of supporting coarse material. These experiments were done simply by agitation of the various constituents of the pulp in a measuring cylinder, with a range of concentrations of the additive. Preliminary centrifuging tests were again conducted to discover the optimum operating speed.

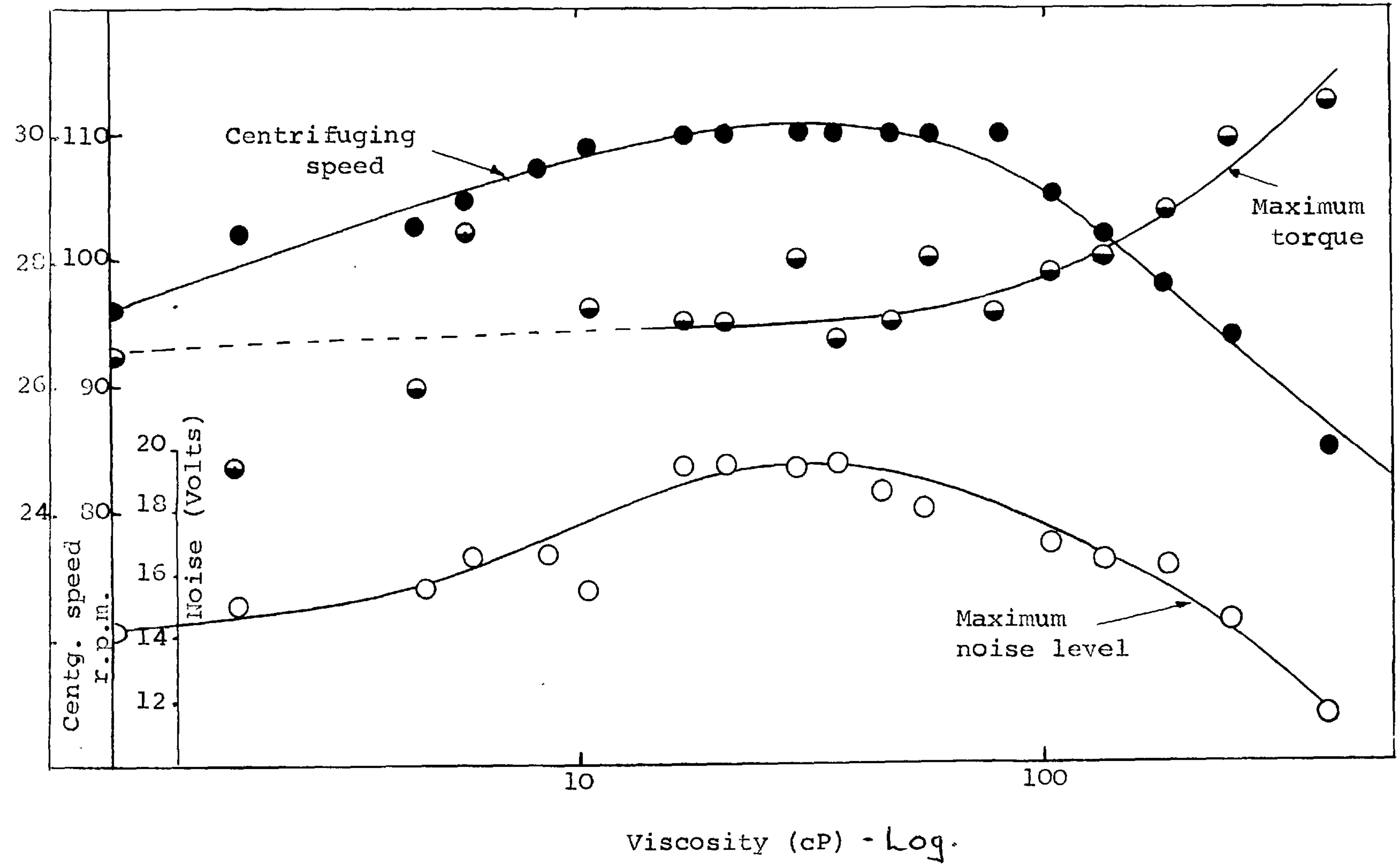
Results.

Tests on centrifuging speed of the balls.

Increases in viscosity from 1 to 12 cP were found to produce increases in the centrifuging speed from 96 to 110 r.p.m. (Fig.7.2). The value then remained constant (±1 r.p.m.) up to about 80 cP. Further increases in the viscosity up to 885 cP caused a drop in the centrifuging speed to 78 r.p.m. (The calculated "critical" value was 80 r.p.m.). The figures quoted here and plotted in Fig.7.2 represent the onset of centrifuging of the balls: a further 3 r.p.m. (approximately) was required for fully developed conditions of centrifuging.

Measurements were made during this series of the maximum noise level and torque required. The results illustrated on Fig.7.2 show that the noise level varied with viscosity in a similar manner to the centrifuging speed. There was a 37% increase in the noise up to 140 cP before it decreased to 30% of the original noise at 885 cP. The readings of torque tended to be rather scattered, although, generally, they increased with increasing viscosity from 26.5 ft.lb. at 1 cP to 30.5 ft.lb. at 400 cP.

Fig.7.2 Results of centrifuging tests



Tests at constant speed.

Fig.7.3 shows the results of this series plotted in the form of viscosity against cumulative percentage smaller than the stated sieve size). There is a clear relationship between viscosity and efficiency of grinding. The quantity of unbroken grains was minimal (about 2%) at high viscosities (300 - 760 cP). The largest percentage of minus 52 mesh was obtained with a viscosity of about 400 cP, and for minus 100 mesh and smaller sieve sizes the maximum efficiency was about 300 cP. Above these values the efficiency decreased gradually in a linear manner. Thus, the viscosity which yielded the maximum efficiency of grinding depended, to a large extent, on the size fraction required. The efficiency for all sieve sizes, however, was a minimum at about 10 cP from which there was a steady increase up to 300 cP. Below 10 cP to 1 cP there was another increase. The quantity of unbroken grains at 1 cP was well below the minimum but the volume of fines produced was about the same as those at 300 cP. The minimum in the efficiency at 10 cP is remarkable: it represented a drop in efficiency of about 15%, with respect to that with pure water, for each sieve size (plotted cumulatively). Although the minimum was mainly due to the minus 300 mesh

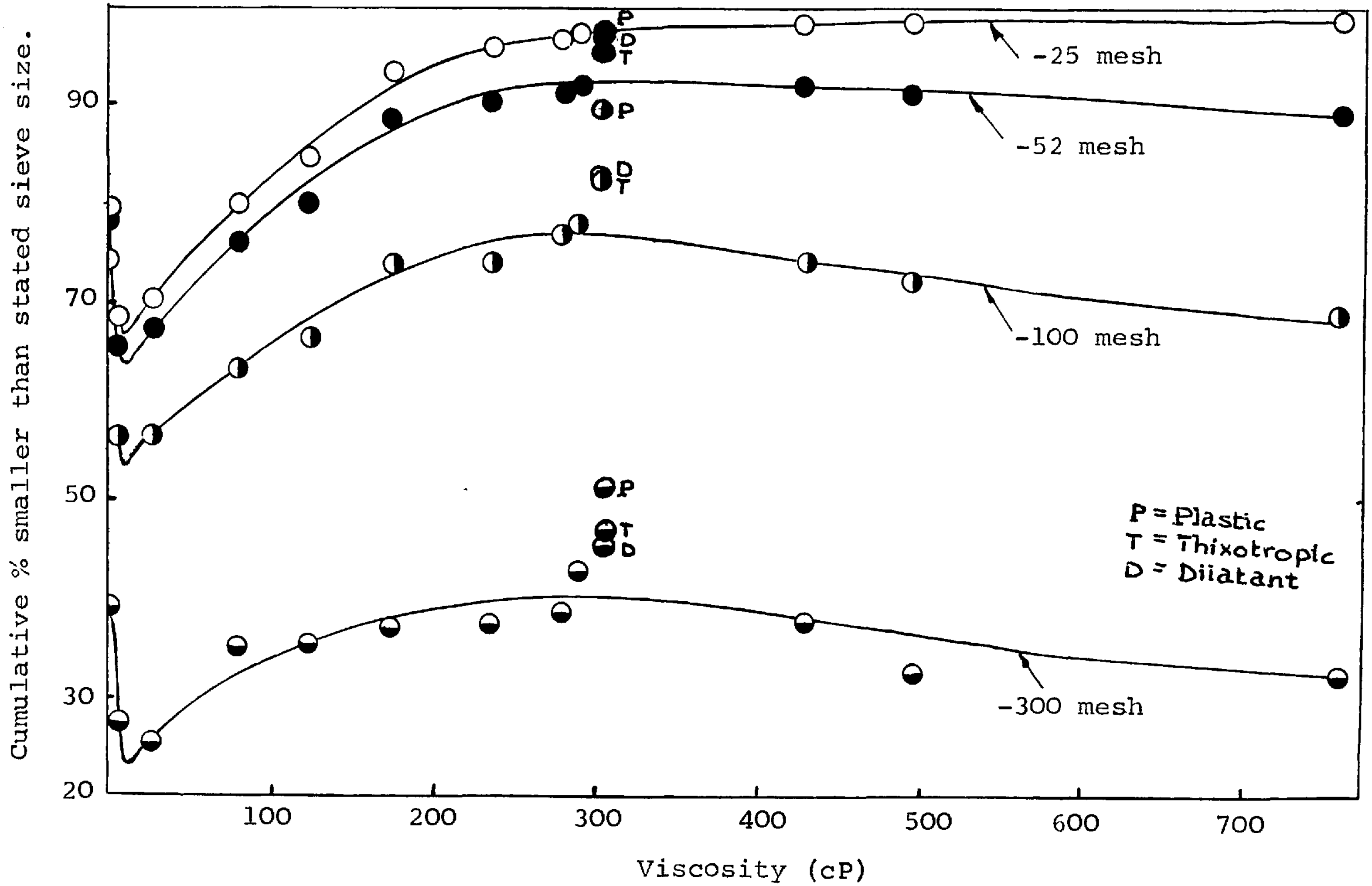


Fig.7.3 Efficiency of constant speed tests (70 r.p.m. for 500 revs.)

fraction, both abrasion and impact grinding were at their least.

The torque required to drive the mill increased generally with viscosity from 23 ft.lb. (at 1 cP) to 27.5 ft.lb. (at 760 cP). The noise level did not show any clear trends with changes in viscosity. During any particular experiment, however, the torque required usually decreased slightly and the noise increased; therefore, the figures quoted are only average values. The average power absorbed during these tests was about 0.35 h.p. No relationship appeared to exist between viscosity and change in temperature, which was of the order of 1.2°C over the 500 revolutions.

The results for the constant speed tests with non-Newtonian media are illustrated in Fig.7.3. Increases of 25%, 13% and 9% of minus 300 mesh material were obtained with plastic, thixotropic and dilatant media respectively, over the maximum efficiency obtained with aqueous glycerol. The torque required for these tests was 31.2 ft.lb. for the dilatant and thixotropic and 29.3 ft.lb. for the plastic.

Tests at constant proportion of centrifuging speed.

The general trends observed in the previous tests were again obtained but with overall increases in efficiency (Fig.7.4). The peak efficiency was shifted from 300 cP to about 100 cP for all sieve sizes, although a maximum was obtained at 1 cP for all sizes below 100 mesh. The surprising minimum at 10 cP - 15 cP still represented a decrease of about 15% in efficiency, over that of pure water, despite the overall increase in efficiency. Beyond 100 cP, the efficiency decreased in a linear manner for all sieve sizes. The decrease, however, was only gradual and much of the variation obtained in the constant speed tests was eliminated.

Fig.7.4 shows that the minimum efficiency of grinding was accompanied by the maximum noise, and, also, maximum temperature change. The torque required, however, did not follow any clear pattern.

Fines tests.

The results were similar to those obtained with aqueous glycerol. The efficiency curve (Fig.7.5) shows the main differences between these and previous tests. The maxima and minima of efficiency were magnified again in a

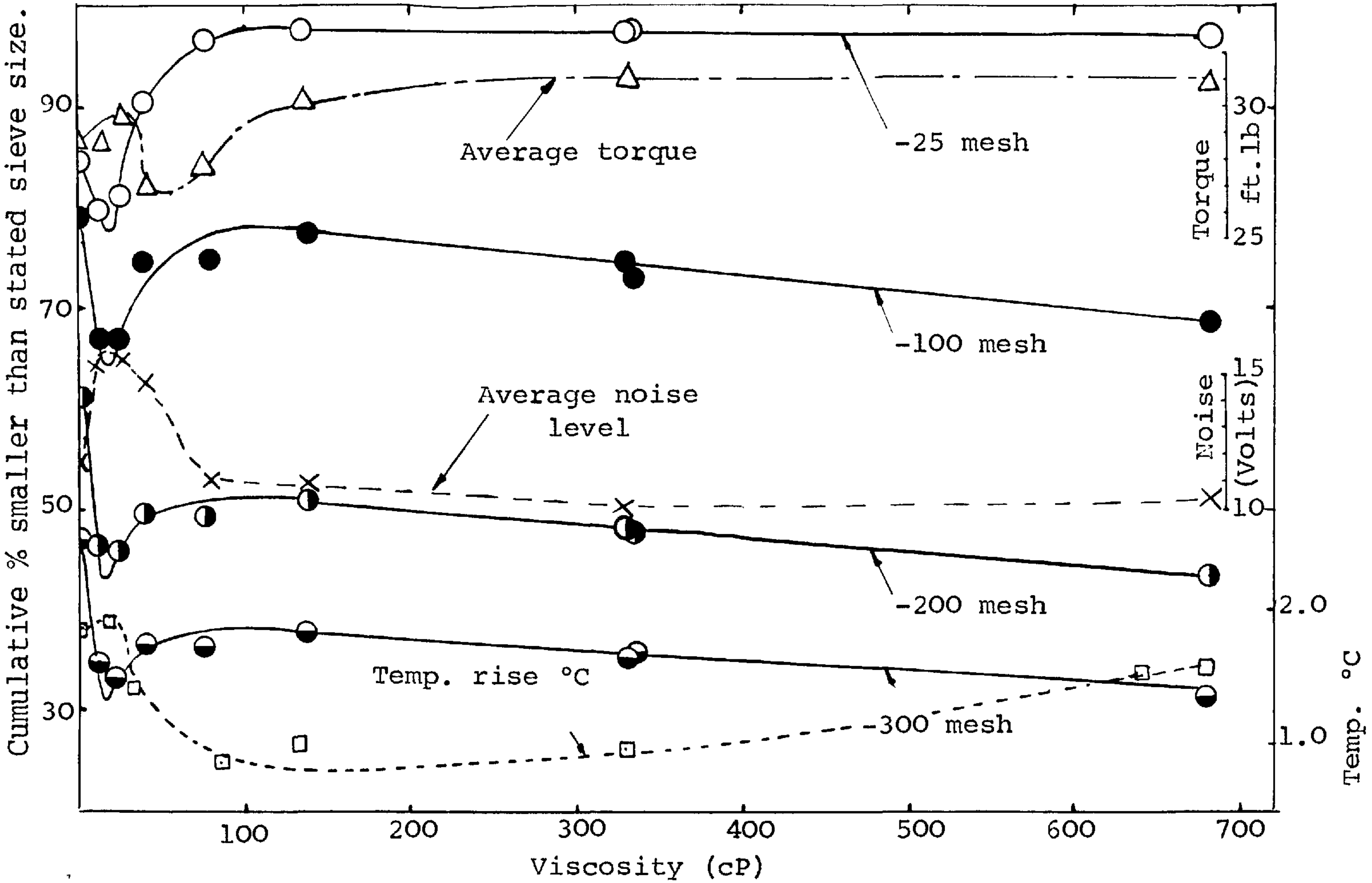


Fig.7.4 Efficiency of tests at 76% of centrifuging speed with Newtonian fluids.

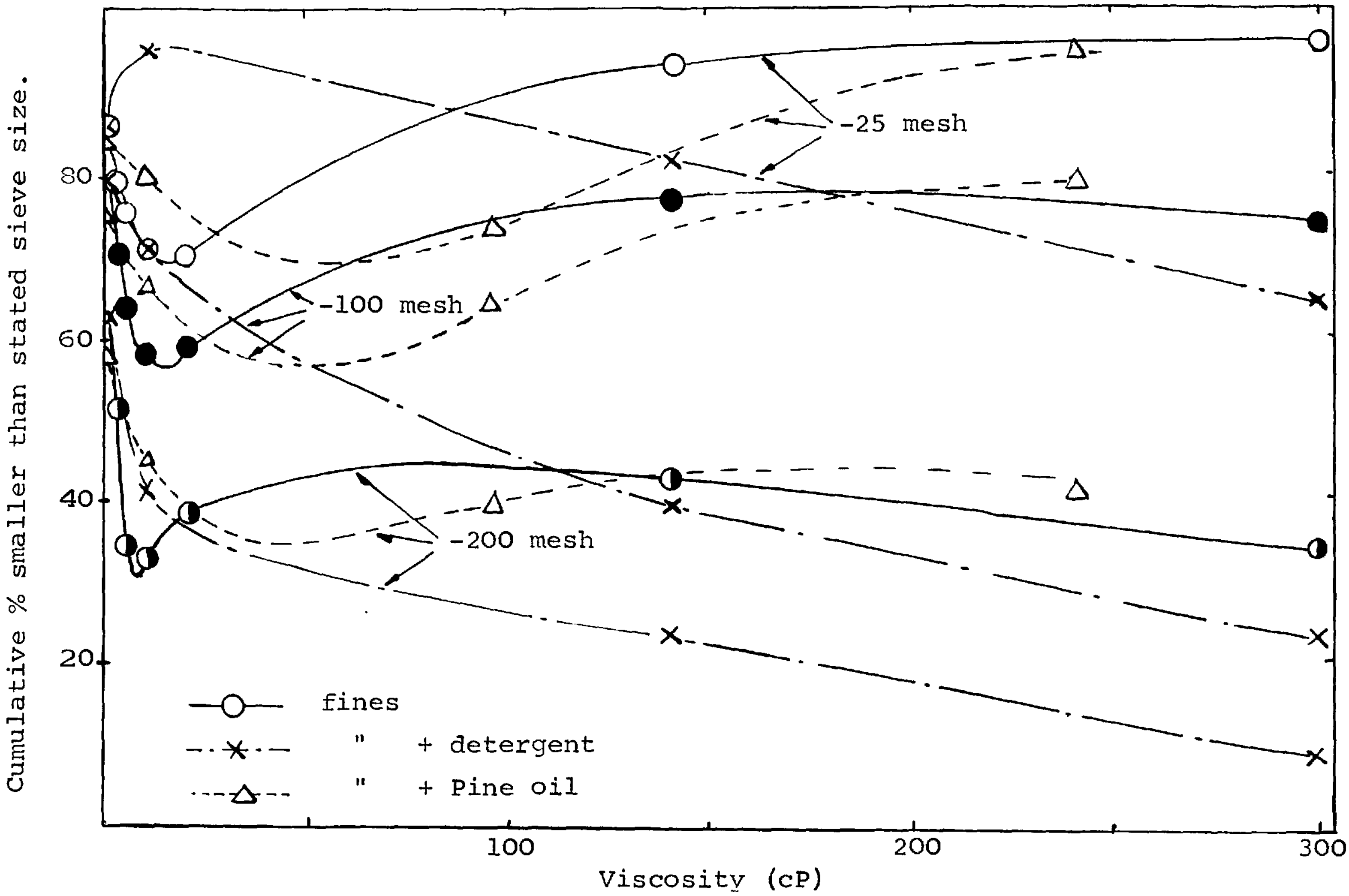


Fig.7.5 Efficiency of tests at 76% of critical speed with fines of quartz and with detergent and Pine oil.

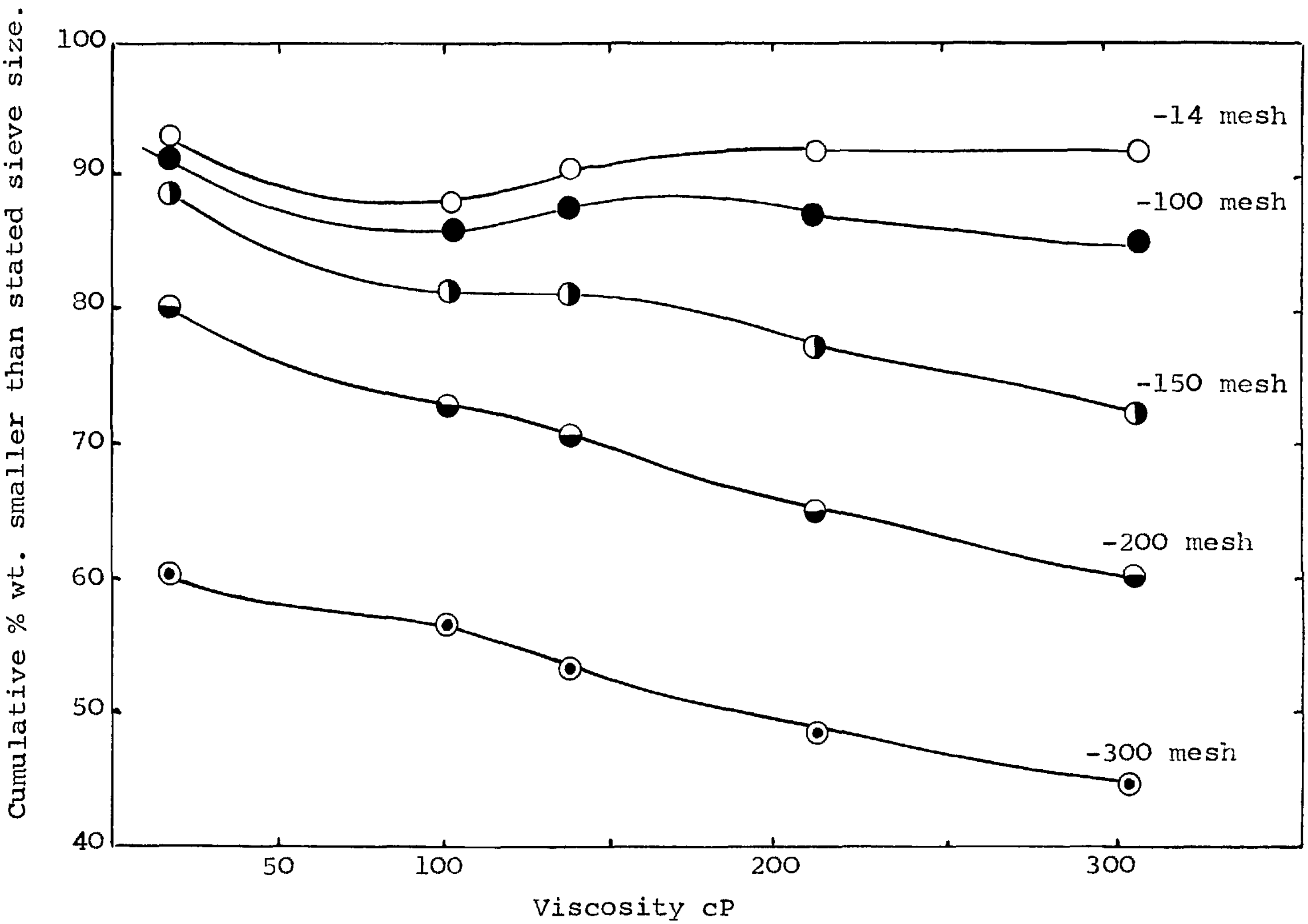
similar manner to the constant speed tests. The drop in efficiency (-300 mesh) at 10 cP represented about 25%, rather than the previous 15%, and at 1 cP the production of fines was 16% above the secondary maximum at 120 cP. A further result from these tests was the displacement of the minima and maxima of efficiency with respect to the screen size. The maximum for 100 mesh, for example, was at about 180 cP, and the minimum at 23 cP.

There were no clear trends with respect to the noise produced, the torque required or the change in temperature over the grinding operation.

Full charge tests.

These tests have been analysed on two bases: firstly, "cumulative weight percentage minus the stated sieve size" (Fig.7.6), and secondly, "cumulative actual weight minus the stated sieve size" (Fig.7.7). The plot on an actual weight basis (which is the more practical for mill output assessment) shows a maximum efficiency (-300 mesh) at about 140 cP. The maximum weight minus 150 mesh, however, was at about 190 cP. Beyond these values of viscosity the efficiency gradually decreased. The minus 100 mesh fraction appeared to have just reached a maximum at 300 cP, but

Fig.7.6 Efficiency of tests with a full charge of material at 76% of the critical speed.



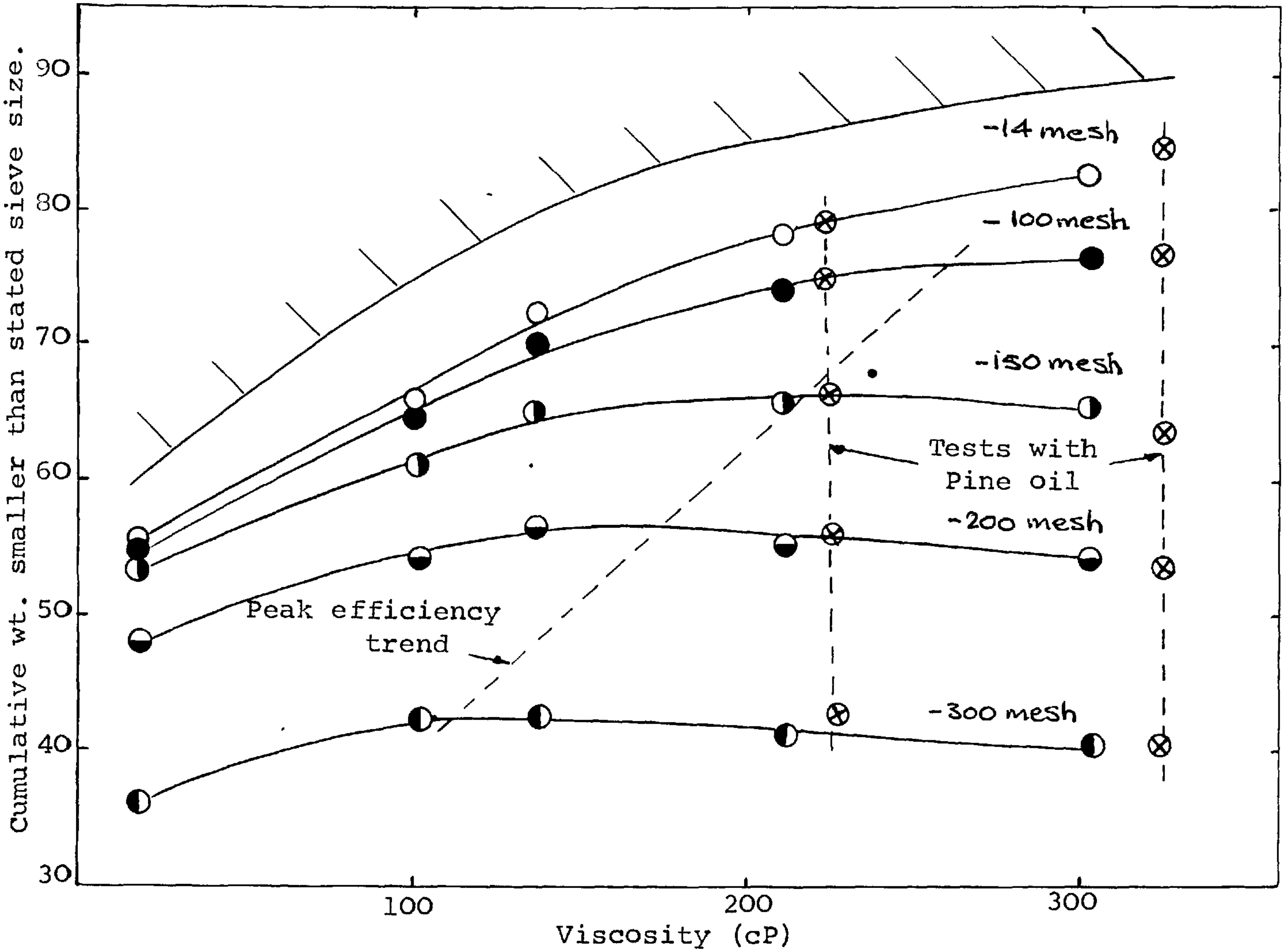


Fig.7.7 Efficiency of tests with a full charge of material at 76% of the 'critical' speed.

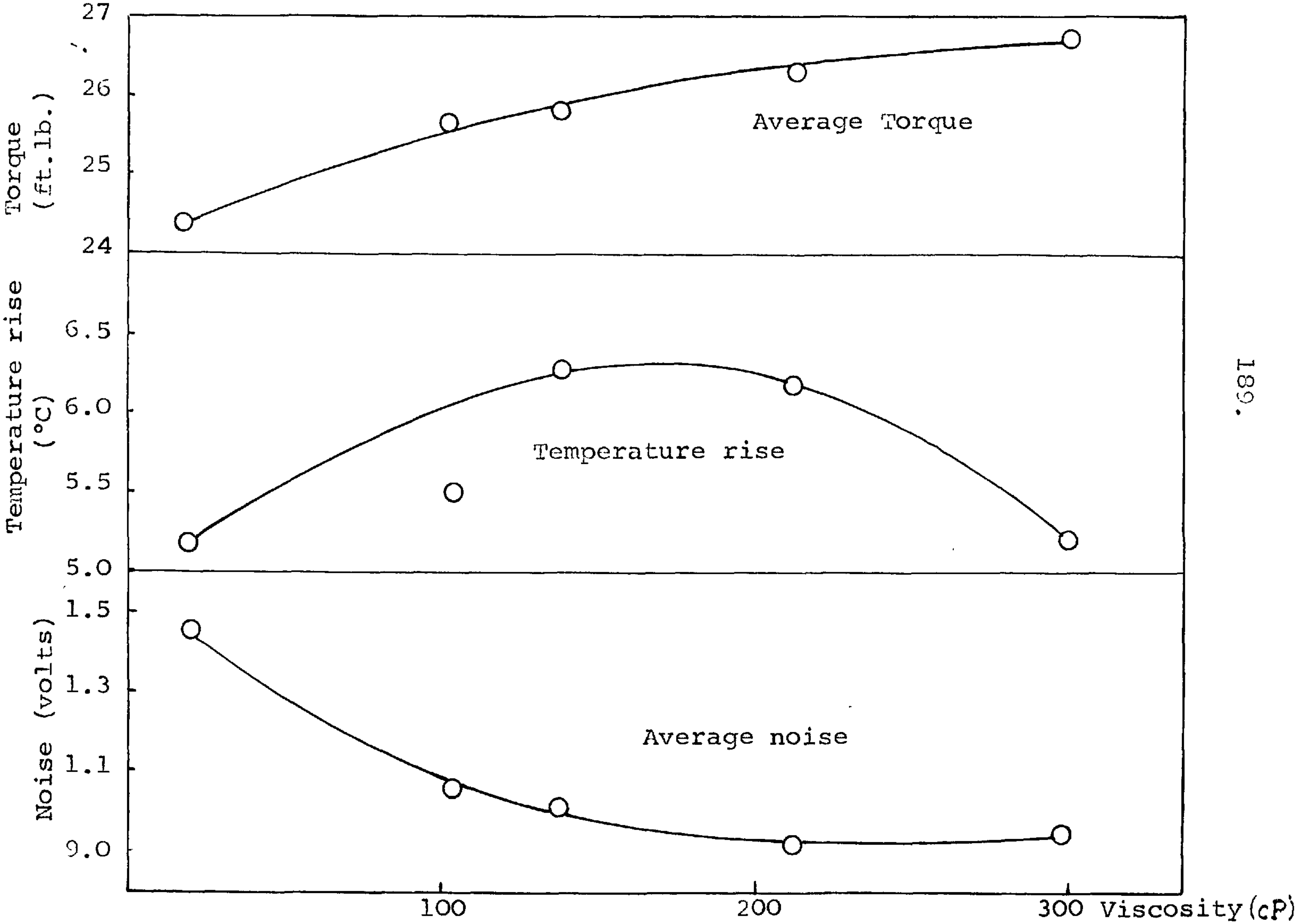
measurements beyond this point were impossible in the modified Ferranti viscometer.

Fig.7.6 shows that on a percentage basis the efficiency generally decreased with increasing charge, and particularly for the finer sizes (-150 mesh). There were, however, up to 4% increases in the minus 100 mesh and coarser sizes with increasing weight of charge and increasing viscosity (100 to 300 cP).

These results cannot be compared on a quantitative basis with the other series of tests owing to the differences in grinding time. The same general trends and shapes of viscosity - efficiency curves, however, have been obtained.

The torque, temperature and noise effects are shown in Fig.7.8. The torque required increased in a linear manner with increasing weight of solids from 24.5 to 26.4 ft.lb.. The noise decreased as shown from 1.45 v to 0.92 v with increases in the weight of charge from 6 kg to 9 kg. Changes in temperature, over the duration of the grind, followed, approximately, the efficiency curve for minus 300 mesh, possessing a peak at 140 cP. The changes, however, were small, ranging between 5.2°C and 6.3°C.

Fig.7.8 Torque, Noise and Temperature effects for full charge tests at 76% of centrifuging speed.



Tests with foaming additives.

Fig.7.5 shows that detergent was effective at low concentrations of fines, producing the desired effect by eliminating the minimum at 10 - 15 cP. However, the mill soon became choked and efficiency (-300 mesh and finer) fell below the original for media of over 40 cP, although marked increases in the percentage smaller than the original feed size were obtained up to about 115 cP.

The influence of the pine oil was quite different from that of detergent (Fig.7.5). The original efficiency curve was shifted to the right. The minimum appeared around 50 cP instead of 10 cP, and the peak efficiency (-300 mesh) was at about 200 cP instead of 100 cP. The measurements of viscosity, for the higher concentrations (>6½ kg) were lower when pine oil was used. These measurements, however, were only nominal owing to their non-Newtonian character. The viscosities of those suspensions with detergent were impossible to measure because of the foam, so those values of viscosity plotted (Fig.7.5) are for the original media without detergent. This introduces some uncertainty, but the efficiency trends were so marked that this uncertainty does not obscure any of the effects.

At the highest concentration the overall efficiency was greater when pine oil was used. This was because the original efficiency of the grinds without pine oil were past their peak, whereas the pulps with pine oil were still grinding at peak efficiency owing to the reduction in viscosity. Additions of pine oil to the charge of 7 kg produced an increase of 22% in the weight of material below 300 mesh, and for 52 mesh material the increase was 65%. With smaller charges, however, the pine oil caused a drop in the efficiency by displacing the viscosity of the pulp into the low efficiency region. The measure of viscosity for the higher concentrations was lower when pine oil was added. For example, the viscosity with 7 kg of fine material was reduced from 285 cP to 235 cP.

Additions of pine oil to the full charge pulps again produced increases in efficiency. The peak efficiency, however, was not sharp and the increases were only marginal. There was an increase of 3.7% in the weight of material below 300 mesh with 8½ kg, but for other screen sizes the cumulative increase in material was about . 1%. On the other hand, with 9 kg the increases were greater for the coarser sizes. There was an average increase of 2.5% of the cumulative weight of quartz coarser than 72 mesh, but the finer material did not show any clear increase. The actual measurement of viscosity with an 8½ kg charge was higher with the pine oil tests. This was probably because of the increase in the concentration of ground material. The measurements with 9 kg, however, were approximately the same with and without pine oil. The pulps were again non-Newtonian and the additives may have produced different instantaneous viscosities when subjected to the high shear-rates within the mill. The values of viscosity quoted and plotted in connection with these tests are only approximate because of limitations of the viscometer.

Conclusions

Movement of the grinding bodies.

On a basis of centrifuging of the balls, aqueous glycerol solutions are satisfactory to give model suspensions to simulate ground pulps. This is only true, however, when there is sufficient mineral present to provide realistic conditions of friction.

The critical speed is dependent upon two factors, firstly, the speed of rotation of the mill and, secondly, the slip between the balls and the liner. The slip is affected by the coefficient of friction and viscosity of the pulp. Thus, high friction and high viscosity tend to raise the balls higher up the mill wall. The high friction occurs at low viscosities before the contacts between the balls are 'lubricated' by a continuous layer of particles or synthetic pulp. Hence, a regime of low friction and medium viscosities cause high critical speeds. The torque requirements are not only related to the height of the ball path, but to increases in the viscosity. This suggests the importance of the distribution of the charge, as affecting the displacement of the centre of gravity of the charge from the axis of the mill.

Size reduction of particles.

The similarity in response of the synthetic and the practical pulps recommends their use as satisfactory model media.

The efficiency of grinding is affected by the trajectory of the balls, which, in turn, is a function of the viscosity. Hence, for a given speed there is an optimum viscosity. The path of the balls is not the only important factor; but, nevertheless, for the optimum viscosity there is only one speed which will give maximum efficiency.

The efficiency of comminution cannot be described in terms of one parameter, as the maximum production of fines occurs at a different viscosity from the maximum production of coarser material. The influence of viscosity affects the rate of breakage in four different ways. Firstly, at low viscosities the contacts between the balls are not lubricated and high friction prevails. The result is high grinding efficiency of the fine material in particular, which is largely produced by the frictional rolling of the balls at the bottom of the mill. Secondly, the grinding surfaces are coated with a layer of particles, which presents the material more efficiently between the points of impact. At medium viscosities it is possible to reach a stage at which the friction is reduced by the increased viscosity, but the coating is not thick enough to present the material efficiently. The value of viscosity for low efficiency is higher for coarse pulps. This is because the pulps have to be even more viscous to support heavy particles. Hence, a region of low grinding efficiency for all sieve sizes is obtained, though at slightly different viscosities. The third factor is the mobility of the balls. As the viscosity of the pulp increases, the balls at the bottom of the mill cannot roll together as much because of the restrictions imposed by the viscous media. Hence, the rate of attrition grinding which produces the fines declines steadily, although the rate of production of coarser material remains constant. Finally, at very high viscosities the rate of impact grinding gradually decreases because of 'cushioning'. The coating reaches a certain thickness when the force of impacts are not sufficient to break the particle. With a graded ball charge the coating thickness is not critical. Nevertheless, there is clearly a point where the efficiency declines, for all sieve sizes, when the coating is too thick for the heaviest balls to penetrate.

Power requirements in milling.

The torque required to rotate the mill is mainly a function of the ball path. Extra work is necessary to continually raise the balls up to a higher trajectory. It follows that greater power input will not necessarily mean an increase in the rate of breakage, as there is an optimum ball path. As shown previously, the torque is also dependent on the distribution of the charge. This affects the displacement of the centre of gravity of the charge from the axis of the mill.

There is a linear relationship between the power required and the volume of the charge, for a given set of conditions. This is clearly because of the extra weight which is displaced from the axis.

As the grinding operation progresses, the torque required falls, because of the higher ball path with coarse material. Large particles lock and wedge the balls together, and prevent any smooth rolling until comminution has progressed somewhat.

Influence of additives on the viscosity of pulps.

Certain chemical additives may be used to reduce the viscosity of ball-mill pulps. This reduction permits a higher charge to be used while maintaining the viscosity of the pulp at the optimum value. With a graded ball charge the peak efficiency is not critical, but it is possible to obtain increases of up to about 4%. A suitable additive for this purpose is pine oil. A further effect of additives may be the production of an artificial coating on the grinding surfaces. The viscosity, naturally, changes throughout the grinding operation because of the size reduction of the mineral. The coating is, therefore, pure water for a coarse feed in the initial stages. The production of a coating may be assisted artificially by chemical additives to produce a weakly plastic foam. Hence, the low efficiency at low friction values may be practically eliminated. For the complete removal of this low efficiency a high foaming agent such as detergent may be used. This may, however, have an adverse effect on higher viscosity pulps or subsequent handling of the material.

Effect of viscosity of the pulp on the noise produced by a mill.

For a particular type of pulp the variation of noise with viscosity follows a certain pattern. As the viscosity increases the impacts are softened and the noise level decreases. For tests at constant speed the problem becomes complicated: as the viscosity increases the ball path is raised but the impacts are softened. It can be difficult, therefore, to follow a clear pattern. With variable speed tests, however, the noise can be used to give an indication of whether the mill is operating efficiently, or whether energy is wasted in loud metal - to - metal impacts.

Chapter 8.

DISCUSSION; WITH SUMMARY OF CONCLUSIONS.

Rheology

The modified Ferranti viscometer, and the results obtained from it, can be considered some advance in the study of suspensions, quite apart from any application to ball-milling.

The design of the instrument is unusual, in that it permits free settling of the particles while they are undergoing shear. This technique has been used before (92), but a conical bob then employed does not maintain homogeneity with a settling suspension; the open-topped principle of the present investigation is preferable. The design of the sample cup to produce a vertical flow pattern with a minimum of rotation of the sample is new in the design of viscometers. A slight drawback in the design of the sample cup was that the action of the stirrer shaft, in the presence of mineral slurries, tended to wear the rubber gland and the shaft at the bottom of the cup. This problem was overcome partly by providing a hard, stainless steel collar which protected the shaft, and partly by replacement of the collar and gland at regular intervals, when leakage occurred.

Few investigations have been made with the type of coarse, settling suspensions described in Chapter 4 of this thesis. The results of such investigations are discordant, mainly because of slip and other weaknesses inherent in the various instruments used. Hence, one of the main features of the modified Ferranti viscometer was the nature of the cylinder surfaces. The importance of preventing slip between the particles of the suspension and the walls of the cylinders was discovered empirically. This problem was overcome by making shear take place between surfaces of a similar "roughness" to the suspension itself. This was done by using projections on the surfaces of the cylinder, which trapped samples of the suspension, providing a defined surface of shear. On a microscopic scale even smooth surfaces are relatively rough compared to the size of molecules of pure liquids. Thus, the molecules are trapped and molecular slip is prevented. With the larger particles of a suspension, therefore, surfaces need to be sufficiently 'rough' to trap the solids. Vertical grooves are preferable for a free-settling suspension; but for non-settling suspensions other profiles may be acceptable. This technique could be used with either type of viscometer.

The results obtained with the modified Ferranti viscometer help to explain some of the discrepancies between the results of other workers. The effect of concentration of solids is generally in agreement with all who have studied this variable, in that viscosity increases with increased concentration. The extent of the increase, however, varies between the results of different workers. The values of viscosity recorded by those who used DeVaney-Shelton type viscometers (56)(60) are generally greater than the results obtained here. This is probably because of excessive turbulence, which can be observed with these instruments, and the lack of wall corrections. On the other hand, the values obtained by Purohit and Roy (47), with a concentric cylinder viscometer, were well below those of the present investigation. This was because they probably had slip, when operating their instrument at high speeds, with smooth cylinder walls and large particles.

The "critical concentration", which some workers (56)(59) quote, was found to be dependent on the shear-rate. This fact did not reveal itself in the efflux type viscometers because of the lack of variation in the rate of shear. It is probably caused by the dilatancy of the pulps, which caused an increase in the viscosity at high shear-rates with lower percentage solids, hence, obscuring the critical concentration. This concentration is reached when the particles can no longer circulate freely without mutual interference. The factor of interaction is important, governing the influences of concentration, particle size, shape, density and friction. Thus, the standard suspension may be regarded as one which fulfills the conditions for the Einstein equation, apart from the range of concentration. Any deviations from these conditions to increase the number and force of interactions, produce increases in the viscosity.

The viscosity was found to increase in a linear manner with increasing particle size. This disagrees with Schack, Dean and Molloy (56), DeVaney and Shelton (59) and Pakianathan (60) who found a decrease. On the other hand, increases in viscosity with increasing particle size were obtained by De Bruijn and Meerman (71) and Purohit and Roy (47) (whose results were published after the completion of the present work). Both of these sets of results, which are in agreement with the present work, were obtained with coarse, settling particles in rotational viscometers. It was shown in Chapter 2, that when particles are in the size range which may be affected by colloidal forces (-10 μ), the viscosity increases with decreasing size of particles. It would appear, therefore, that there is a minimum viscosity at about 10 μ, and increases with both larger and smaller particles.

The effect of particle shape is well known for the smaller sizes but the extent of the increase had not been clarified for coarse, settling particles. Smooth, spherical particles give the minimum viscosity. Departures from this ideal shape cause greater effective concentrations. The particles rotate when undergoing shear and sweep out larger volumes. Hence the chance of interaction is increased and the critical concentration occurs at lower values of viscosity. For example, the rods exhibited a sharp increase in viscosity at 15 vol.%. Interaction between spheres (apart from interaction of fluid flow lines) is probably a series of simple impacts and rebounds. Roughened grains, rods and plates, however, rotate to a greater extent, resulting in frictional scraping and locking between the particles, and hence higher viscosities. This was established by using the four standard shapes mentioned above, which represented the ideal and three grades of departure from it. Attention is drawn, therefore, to the importance of changes in particle shape on the viscosity of a pulp or slurry in a unit operation. For example, at a concentration of 25 vol.% it was possible to attain an increase in the viscosity by a factor of 10 when using plates rather than spheres.

It has been known for some time that, under certain conditions, mixtures of particles may be of lower viscosity than a mono-disperse system of the same mass concentration (49)(67). This is because of the ability of small particles to circulate freely in the voids between the large. A new relationship has been introduced which relates the effect of the relative concentrations of the constituents on the viscosity of the suspension. The relationship agrees with the theory that a small percentage of large particles does not affect the viscosity, except from a concentration and particle size standpoint. Small percentages of fine particles, however, can reduce the viscosity of a coarse suspension markedly, by intermingling and circulating in the voids between the larger particles. This relationship is in agreement with that of Ward and Whitmore (67) with non-settling suspensions of spherical particles in a rising sphere viscometer. These authors did not study the effect of relative concentrations, however, only the width of the size distribution.

There does not appear to be any published work on the influence of the density of particles. Purohit and Roy (47) realised that density did have some effect, when the particles of coal responded in a different manner to the particles of quartz that they used, in particle size tests. The results shown here, however, illustrate the fact that as the density increases there is a corresponding increase in viscosity. This effect is presumably a function of the force of the impacts between the particles.

The importance of the nature of the interactions is illustrated by the results from the addition of detergent. The viscosity was reduced substantially with the rough particles by reducing the coefficient of friction between the quartz particles. The difference in the viscosity of the smooth (hard) glass spheres with and without detergent was barely measurable.

The data which have been obtained are relevant in several industries. For example, a knowledge of particle size and shape effects is necessary in the handling and transport of solids in liquids. The use of a detergent to reduce the viscosity is a cheap, available method which could be used to increase capacity of existing apparatus for the transport of coarse suspensions. The instrument developed and the results from it could be of value in mixing and processing of pulps in the food, paint, paper, cement and ceramic industries. Further work would probably be required, however, to analyse the behaviour of specific suspensions under a particular set of conditions. The present work has also a bearing on the mineral processing industry, notably, as regards continuous milling, gravity concentration, flotation, etc..

Summary of Conclusions: Rheology.

1) The instrument developed has made it possible to extend the study of the rheology of coarse settling suspensions to particles up to 211 μ diameter at concentrations of 20 - 50 vol.%, depending on the shape and density of the particles.

2) Coarse, settling suspensions are non-Newtonian in nature: the viscosity increases with increasing shear-rate. This dilatancy increases with density, anisometry of form and, to a small extent, size of particles.

3) The "critical concentration" for increase of viscosity is not clearly defined at high shear-rates with dilatant suspensions. At low shear-rates there is a fairly sharp increase in the viscosity (between 25 - 30 vol.% for grains of quartz) with linear regions above and below it. It occurs at a lower viscosity with greater density or greater anisometry of particles and at higher shear-rates.

4) The viscosity of coarse settling suspensions increases linearly with particle size for a given concentration and shear-rate.

5) Friction between particles contributes largely to the viscosity of suspensions. Hence, if a detergent is added to a suspension of rough particles the viscosity is appreciably reduced.

6) At a given solids content, suspensions of mixtures of particle sizes are of lower viscosity, when the

size distribution is such that small particles can circulate freely in the voids between large particles.

Grinding.

The present work has directed attention to the mechanics of the operation of ball-mills, and particularly to the influence of the viscosity of the pulp. The results of others have been clarified, and phenomena have been discovered which explain some of the discrepancies between the results of different workers.

The results obtained here are in agreement with those of Creyke and Webb (120) and Salman and Sirois (86) in that the production of fines is greater at low viscosities. In addition, however, a secondary zone was discovered which could account for high production of fines over a certain range of higher viscosities. The results disagree with those of Schweyer (122) who claimed that there was little difference in the rate of grinding from 1 to 50 cP. This is a range in which much variation took place in the present investigation. The low efficiency at about 10 cP was not discovered by Schweyer because his increments of viscosity were too large. His results were also obscured by over-grinding and contamination from grinding for long periods. Schweyer (122) found that the rate of impact grinding was faster for a less viscous medium. This is true according to the present results beyond the peak efficiency (say 140 cP), but the breakage of coarse material by impact, below this value, generally decreased whether with tests at constant speed or with equivalent ball paths.

The peak efficiency described by Creyke and Webb (120) is in agreement with the present work. It is suggested, however, that their results were affected by variations in the optimum running speed. The tests of these workers were conducted at 83% of the theoretical critical speed. The centrifuging tests described in Chapter 6 showed that there was no slip of the charge at 760 cP with steel balls. One would have expected, therefore, at least partial centrifuging of the charge, with the porcelain balls that they used, at 2000 cP. The balls were of similar diameter to those used in the present investigation. This would account for the sharp decrease in efficiency which occurred (Fig. 5.1), rather than the more gradual effects obtained at equivalent ball paths. Creyke and Webb (120) did not investigate the influence of viscosity below 82 cP and, hence, did not record the reduction in efficiency at 10 - 15 cP. Their work was mainly with sub-sieve material, and so they did not differentiate between the influences of the viscosity of the pulp on attrition and impact grinding.

The zone of minimum grinding efficiency was not obtained by Hockings, Volin and Mular (123). This could be because the increments of viscosity that they used were too great. On the other hand, the minimum may have been eliminated because of fundamental differences in the operation of ball-mills and rod-mills. They did, however, note the gradual decrease in efficiency when the viscosity was increased beyond about 100 cP.

The results obtained here agree with the conclusion of Fischer (121) regarding ball mobility. He did not, however, differentiate between the varying effects of mobility on impact or attrition grinding, as affecting the breakage of feed material or the production of fines. This was no doubt because he was mainly interested in pigment dispersion with very fine particles.

Fischer observed that the power consumption went up with increasing viscosity with tests at constant speed. He also noted the change in viscosity throughout a grind.

Fischer did not obtain the same "locking" effect on the balls, however, which coarse particles produce, as he was grinding only small particles.

The linear relationship between power and pulp-consistency of Gow, Guggenheim, Campbell and Coghill (109), and quoted by Rose and Sullivan, is considered to be doubtful as a general application. If pulp 'consistency' is taken to represent the viscous properties of the medium then the relationship obtained in the tests at constant speed was not linear. The tests with a full charge showed that the power increased in a linear manner with increases in the weight of the charge at equivalent ball paths. This was, however, clearly due to mill loading effects.

No evidence was found to support the conclusions of Taggart (110) who claimed that there was a decrease in power with increasing viscosity. The only occasion when this result was obtained was when enhanced viscosity caused centrifuging of the balls.

The significance of friction between the grinding surfaces was suspected by Rose and Sullivan (115), but they were unable to fully establish their hypotheses experimentally. Further evidence has now been provided which supports the theory that friction is important (Appendix I). The resistance to sliding is shown to follow approximately the same trend as the efficiency of grinding. Thus, when the grinding bodies can slide easily over one another the efficiency is low. When movement is restricted by a layer of particles, however, then a harsh contact is made, and the balls can trap the particles, resulting in efficient grinding. This is not a true coefficient of friction; it is resistance to sliding in the presence of various media. At low concentrations, the particles appear as a series of separate obstructions which produce high friction, whereas at higher concentrations the bodies can slide easily over a continuous layer of particles. As the concentration is increased further, the irregular packing of particles between the surfaces again produces high 'friction'. The friction experiments give an indication of the contacts between the balls. They give a reason for the minimum efficiency zone, and add further support for the coating theory. Thus, not only does an even coating present the material to the grinding surfaces, but it also provides a layer of particles which the balls can "bite" into rather than slide over.

These results are in agreement with those of von Szantho (6) who obtained a response to friction similar to that of grinding efficiency with changes in the percentage of chemical reagent added to the pulp. He also suggested that additives may be used to affect the friction to produce increases in the efficiency as well as by adsorption phenomena.

The results from the friction experiments are in agreement with those from the centrifuging tests. Hence, the friction between the balls and the liner influences the height of the ball path. At very low viscosities, the friction was high and, therefore, the critical speed was low and the grinding efficiency high. Conversely, at values of viscosity around 10 cP, the centrifuging speed was high because of low friction, and low efficiency of grinding resulted. Thus, the friction between the grinding surfaces is one of the major influences on the efficiency of grinding.

The simplified model, used for the friction measurements, was satisfactory in giving an indication of the efficiency of grinding within the mill. This is only true within specified limits, however, because frictional effects were obscured by cushioning beyond the peak value of efficiency.

The variation of noise with viscosity is well known (139), and it was found to be an excellent indicator for centrifuging. On the other hand, within the limits of the information obtained the variation of noise with efficiency under closely controlled laboratory conditions was not found to be a reliable guide to the control of the mill.

The "buoyancy" effect of viscosity studied by Mular (119) may be linked with the coating effect. In other words, the property of the pulp which prevents the mineral from settling is important in presenting the ore in an efficient manner to the grinding bodies. However, upward settling is no better than downward, in that the material does not <u>evenly</u> coat the grinding surfaces.

The use of additives to lower viscosity (140) or prevent the formation of a pulp with a yield stress (121) has been discussed earlier. The reasons for the additives used here, however, have been different from those in previous investigations. Flocculants and dispersants have been used previously to lower the viscosity by influencing the surface forces on the fines. It has been discovered, however, that a detergent or foaming agent may be used to lower the viscosity of a <u>coarse</u> suspension by lowering the coefficient of friction between the particles. Thus, it is possible to maintain a higher concentration of solids within the range of peak efficiency by the addition of a detergent. It has been found (Fig.4.19) that a detergent may be used to reduce the viscosity of a pulp by 50%. The grinding results also show that changes in the viscosity of the pulp may produce increases of up to 65% in the grinding efficiency (Fig.7.5). It is possible, therefore, that changes in efficiency of 65% or less previously ascribed to the Rehbinder effect, could have been due to simultaneous changes in the viscosity of the pulp. Increases greater than 65% in the cumulative weight-minus-a-particular-sieve-size are not, however, accounted for by changes in the viscosity of the pulp. The results quoted earlier, obtained by Gilbert and Hughes (12) with Armac T, could well have been due to variations in the viscosity of the pulp.

The main difficulty in pursuing this problem further is probably the measurement of the viscosity of the pulp. The modified Ferranti viscometer was found to be satisfactory within specified limits in this respect. It is assumed from the scatter of results that the absolute accuracy fell at high concentrations of coarse particles, though a nominal figure could always be obtained and, hence, the change in viscosity due to additives etc. could be estimated. An efflux type of viscometer would not have indicated the non-Newtonian nature of the pulp. But dilatancy presents a problem of a different kind. The shear-rate in the viscometer could not be made to match the "average" operating shear-rate within the ball-mill. The instantaneous viscosities at certain points within the mill are probably very high with a dilatant pulp, and far beyond the normal range of any conventional viscometer. The actual values of shear-rate within an operating ball-mill are, however, unknown. Approximate values could be calculated from a knowledge of the falling speed, the size and the density of the balls, etc.. Alternatively, the following experimental method might provide some insight. A fluid would be required which exhibited a permanent reduction of viscosity with high shear. This property is said to be possessed by certain oils (141) containing long-chain molecules which break with high rates of shear and do not rejoin. The breakdown of such oils could be calibrated with a high pressure orifice viscometer (141). The viscosity of a sample of this oil should be measured before and after a period of grinding in a ball-mill. Hence, the degree of breakdown in viscosity would be a function of the shear-rates within the mill. Several periods of varying time may be necessary to obtain the full relationship between the average shear-rate and the residence time.

Despite the errors in the measurements of viscosity due to variations in the shear-rates, general trends and effects of viscosity of the pulp on the efficiency of grinding have been clarified. In support of the validity of the measurements of the viscosity of coarse pulps, it was found that the equivalent maxima and minima of efficiency with aqueous glycerol or full charge tests appeared at approximately the same measure of viscosity. This suggests that the response of the ball-mill to changes in viscosity corresponded to that of the viscometer for all types of pulp.

The present study has shown the importance of operating a mill out of the low-efficiency zone, where reductions of up to 40% in the volume of ground material have been recorded. The major factor which influences the viscosity of the pulp is the percentage of solids. The earlier work (Chapter 4) showed how the viscosity was greatly affected by small changes in the percentage solids at high concentrations. The weight of charge is, therefore, critical. It would be safer to operate a mill having a pulp which is too viscous than too fluid, because efficient grinding would take place before all the particles were small enough to contribute to the viscosity. The fall in efficiency is also less sharp beyond the peak than before it.

Further work would be required to apply the principles to a particular mill, as the figures obtained apply only to the particular set of experimental conditions used here. Checks could be made to ascertain whether a mill was operating at maximum efficiency. In view of the high cost of milling, even improvements of about 1% could result in substantial savings. Naturally, other factors would have to be taken into account, such as ball and liner wear, before the techniques were implemented practically.

In mineral processing, grinding is mainly carried out in continuous flow mills. Control of the viscosity in this case is more difficult. A continuously operating, viscosity-sensing instrument (possibly on the lines of an efflux viscometer) could be used to record the nature of the output material. A signal sent to an input regulator could then automatically adjust the feed to the mill to provide the optimum viscosity of the pulp.

Summary of Conclusions: Grinding.

1) The speed at which the balls of a mill start to centrifuge varies with the viscosity of the pulp. The centrifuging speed increases with increasing viscosity if the pulp is too fluid. The increase continues until enhanced viscosity causes adhesion between the balls and the mill lining when there is a steady fall in the centrifuging speed with further increase of viscosity.

2) There is a certain speed at which a mill may be operated to yield maximum efficiency. This speed depends on the fineness of the product required and the viscosity of the pulp. Thus, for a particular size of ground product there is also an optimum viscosity of pulp. The efficiency of grinding in ball-mills follows a reproducible relationship with the viscosity of the pulp. At very low viscosities there is high grinding efficiency because of the high coefficient of friction between the grinding surfaces. Slight increases in the viscosity of the pulp reduce the friction and hence the efficiency.

Further increases in the viscosity produce a thicker coating of material on the balls and also increase resistance to sliding. Eventually the coating becomes too thick and the impacts are cushioned, with a resultant gradual fall in efficiency.

3) The grinding of the material is by the processes of impact and attrition. The coarse feed is generally broken by impact and the fines are produced by impact and attrition. Hence, as the viscosity is increased, the energy of the impacts is just as great if ball paths are equivalent, but the mobility of the balls at the bottom of the mill is reduced. Thus, the production of fines decreases before the breakage of coarse material falls off as the viscosity of the pulp increases.

4) It is possible to increase the output of a mill by increasing the pulp density, while maintaining the optimum viscosity by the addition of a detergent.

5) The power consumed by a mill increases with the viscosity of the pulp over the normal operating range of pulp densities. This is because of variations in the average height of the path of the balls, the

distribution of balls throughout the mill and the weight of the charge. The power consumed throughout a batch grinding process decreases as an operation progresses.

6) The noise produced by a mill during grinding gives a good indication of the onset of cascading and centrifuging of the balls. The noise generally decreases with increasing viscosity, and increases as a grinding operation progresses.

Appendix I.

FRICTION BETWEEN GRINDING SURFACES.

Introduction

This study of friction was prompted by the anomalous results of grinding tests obtained at low viscosities. Firstly, ordinary viscous effects did not explain the decrease in the critical speed for centrifuging at low viscosities. Secondly, friction effects appeared to be the most likely reason for the minimum in the grinding efficiency at 10 - 15 cP.

Friction is a major factor affecting the relative motion between solid bodies which are in contact. The degree of friction can be significantly influenced by the nature of the lubricant between the bodies. Thus, it was reasonable to suppose that friction played a part in governing the motion of the balls within the mill under different conditions of 'lubrication'. The work of von Szantho (6) and Rose and Sullivan (115), discussed in Chapters 5 and 8, gave more evidence for this supposition. Their work, however, did not cover the effects at low viscosity obtained in the present investigation. It was considered worthwhile, therefore, to carry out experiments to investigate the influence of various pulps on the friction between steel surfaces. Hence, it was hoped to clarify the extent to which the ball mechanics and the grinding efficiency were affected by the nature of the grinding surfaces.

Apparatus for the measurement of friction.

The coefficient of friction was obtained by measuring the time of traverse of a trolley along a flat mild steel plate (Fig.AI.1). The trolley was dragged by a thread which was attached to a balance weight via a light pulley. The plate was machined flat in both directions (-.005") and the surface prepared by abrasion with FF grade emery paper and acetone before each test. It was necessary to de-grease the surface before each test otherwise it was not wetted by the glycerol solutions. A spirit level was used to check the levelling of the plate in two directions before each test. The approximate dimensions of the plate were 18" long by 3½" wide by ¾" deep. The distance of traverse of the trolley was about 14½".

The trolley consisted of a flat plate, 3" long by 2¼" wide by ¼" deep, to which was attached three 1" diameter

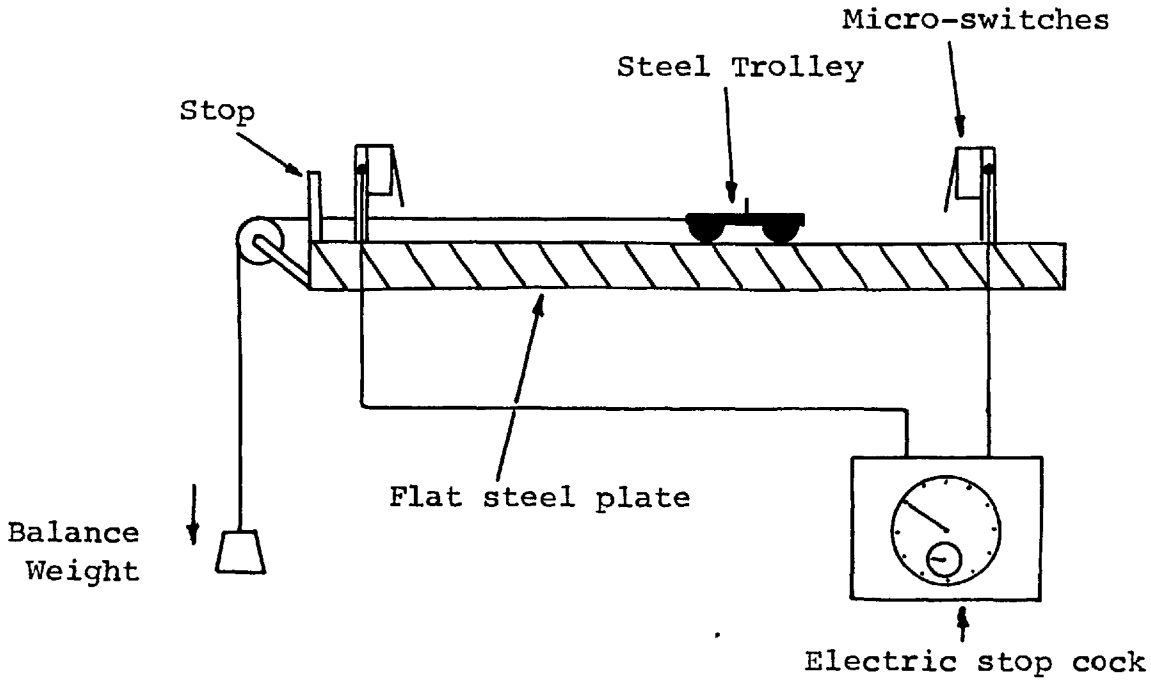


Fig.A1.1 Apparatus for the measurement of the apparent coefficient of friction.

hemispheres. The hemispheres were reject balls from the mill, sawn in half, drilled, tapped and screwed to the underside of the trolley plate in a triangular arrangement. Thus the plate was supported evenly, with each point of contact able to travel in its own path. The hemispheres were prepared before each test in the same way as the bottom plate with emery paper and acetone. A striker plate was fitted to the top of the trolley to operate the levers of the micro-switches. The switches (1 oz pressure, made by Burgess Products Co. Ltd., type no.CRW2) were fitted one to each end of the bottom plate and arranged in circuit with an electric stop-clock (made by Venner Ltd. as in leaflet no.AD/19). By these contacts, the time of travel of the trolley was automatically timed to ±0.005 sec. Over-travel of the trolley was prevented by a rubber-covered stop which was welded to the end of the plate.

The accuracy of the measurements was dependent on the thickness of the coating, the preparation of the surfaces, the viscous and hydrodynamic effects and the measurement of the time of traverse. In order to keep the errors as small as possible, each test was repeated at least ten times. It is estimated that the overall accuracy of the absolute values of the coefficient of friction was ±25% for fluids below 25 cP and ±50% for those around 1000 cP, but the comparative results within a particular series, with equivalent balance weights, were accurate to ±2%.

Experimental.

The plate was prepared before each test by removing the score marks and medium from the previous tests with new emery paper and paper tissues with acetone. The suspension to be tested was prepared and brushed thinly on the plate before the surface had a chance to corrode in the atmosphere. A balance weight was selected which would overcome any tendency for the trolley to 'stick' when released, but which only accelerated the trolley slowly in order to minimize hydrodynamic forces. The trolley was held against the starting switch and released. Ten readings were recorded for each set of conditions. The quartz and aqueous glycerol used for these tests were those fractions and calibration solutions used for the rheology work described in Chapter 4.

The quartz was suspended in a solution of aqueous glycerol of 2 cP instead of water in order to reduce the rate of corrosion of the clean metal surfaces.

In view of the large number of tests to be carried out, it was inconvenient to use the exact weight each time which was just sufficient to keep the trolley in motion. The coefficient of friction was, therefore, calculated from the following formula which allows for the acceleration of the trolley:

$$\mu = \frac{w}{W} - \frac{(w + W)}{Wg} \cdot \frac{2x}{t^2}$$

where μ is the coefficient of friction, W is the trolley weight, w is the balance weight, x is the distance of traverse, t is the time of traverse and g is the acceleration due to gravity.

Experiments were conducted to study the effects of a range of solutions of aqueous glycerol (1 - 1210 cP), various suspensions of aqueous glycerol and quartz and suspensions with the detergent "Quix" or pine oil added. The effect of concentration and size of the quartz particles were also studied. Corrosion of the surfaces was found to have a significant influence on the coefficient of friction. A short series of tests was carried out, therefore, to examine the rate of increase of the coefficient of friction with time, using distilled water alone or with pine oil. The initial series of experiments with aqueous glycerol alone was carried out with two different balance weights, and hence different trolley speeds, in order to check the validity of the results.

Results.

Figure AI.2 shows the relationship between the coefficient of dynamic friction (μ) between steel surfaces and the viscosity of the aqueous glycerol lubricant, with two different balance weights. A striking result was the rapid decrease in the frictional resistance over the range of viscosity 1 - 10 cP. Beyond 10 cP the friction continued to decrease in a gradual manner up to 1210 cP. The values of the coefficient of friction were found to be higher with the lighter balance weight and the slower acceleration. Thus, at 25 cP the two coefficients were 0.162 and 0.208 for the heavier and the lighter weight respectively. The values of friction recorded ranged between 0.226 to 0.45 for the 172 gm balance weight and 0.26 to 0.132 for the 122 gm weight over the range of viscosities from 1 to 1210 cP.

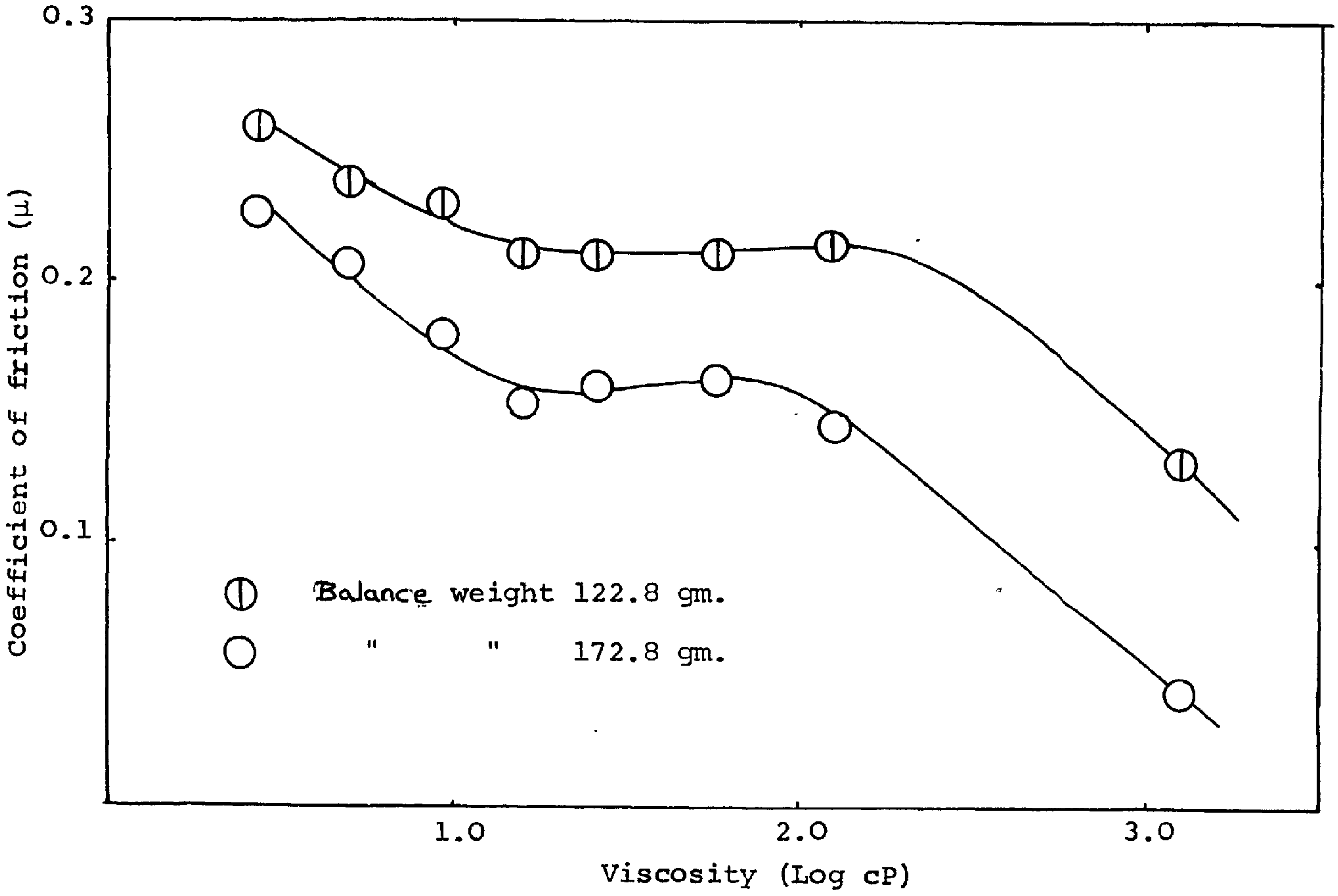


Fig.A1.2 Apparent coefficient of friction with glycerol water mixtures.

The influence of additions of quartz particles to solutions of aqueous glycerol of 2 cP was to increase the coefficient of friction (Fig.A.I.3). As the concentration of particles was increased to produce a higher viscosity, the frictional forces decreased in a similar manner to that of the previous test (1 - 10 cP). Further increases, however, produced increases in the coefficient of friction to a constant value of about 0.25 for viscosities greater than 200 cP.

The effect of particle size on the friction was negligible under the conditions of the test. The concentration of quartz used was high in this series (equivalent to 8½ kg in the mill), but the variation in the friction was only 0.249 to 0.259 over the range 300 to 52 mesh.

The influences of neither "Quix" nor pine oil addition on the friction were significant. Both additives produced slight increases in the friction at a high concentration of particles (equivalent to 8½ kg in the mill, i.e. 48 vol.%). The increases, however, were not consistent and in some cases were only marginal. The additives were used over a range of sizes of particles from 300 - 52 mesh.

The results of the study of the influence of pine

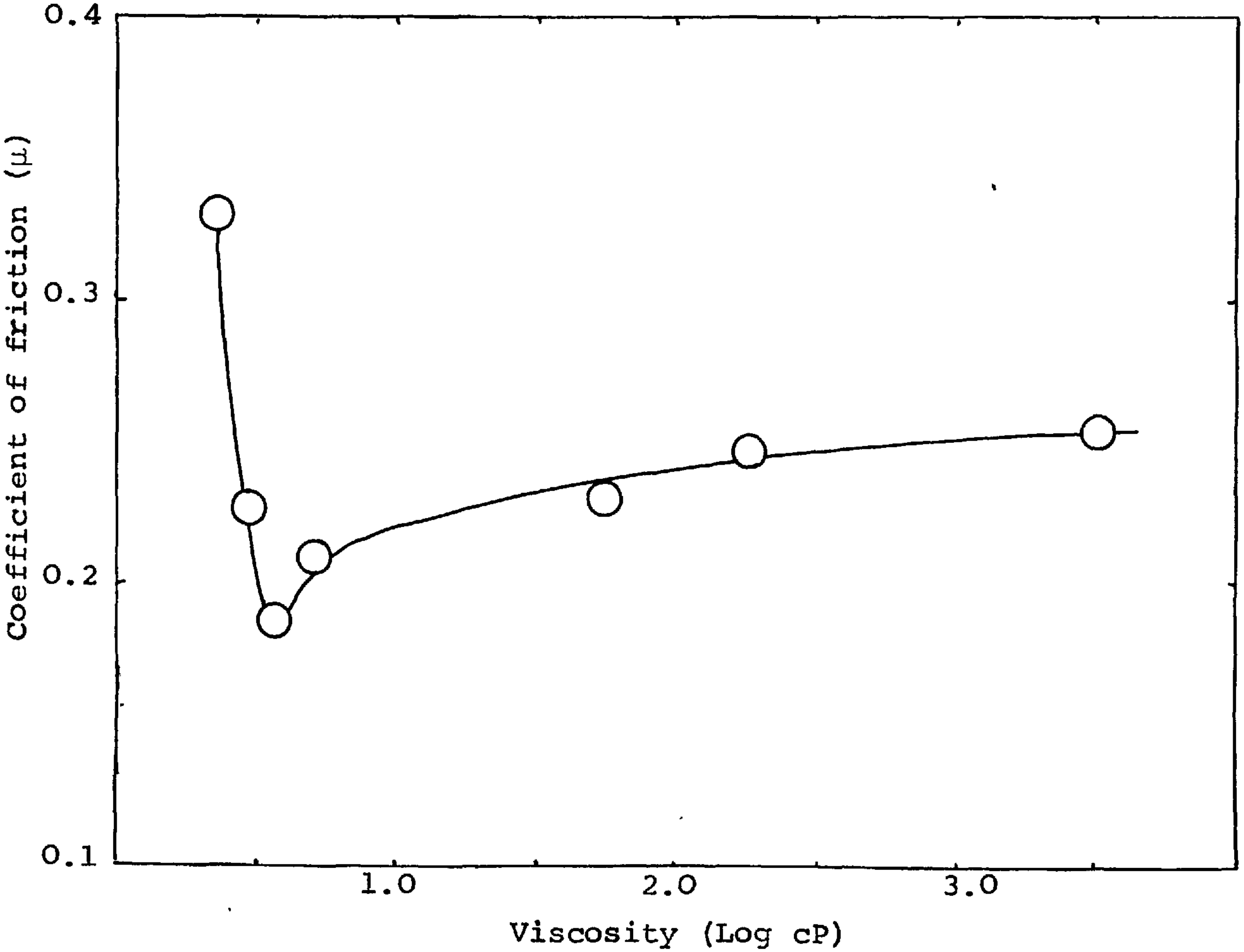


Fig.A1.3 Coefficient of friction with glycerol-water-quartz mixtures oil in distilled water on the friction between steel surfaces are shown in Fig.AI.4. The pine oil had no measurable effect on the friction, relative to that with water only, over a period of time. It can be seen that, over a period of 2 minutes, the coefficient of friction increased in a linear manner with time from 0.33 to 0.4 approximately.

Discussion.

The results of the friction tests largely support those of the grinding tests. The centrifuging speed was thought to be a function of the friction. This hypothesis is supported by the fact that at low viscosities high values of friction prevail, with or without the presence of quartz. The high friction accounts for the low critical speed at around 1 cP. Increases in the viscosity produce decreases in the friction which give a reason for the higher critical speed from 10 to 100 cP. Experiments above 100 cP with aqueous glycerol showed that the critical speed is not entirely dependent on frictional forces which show a further decrease. The tests with aqueous glycerol alone, however, do not give a complete picture. A surprising result was the decrease in friction with a heavier balance weight, and hence, faster speed of traverse. The results were of the

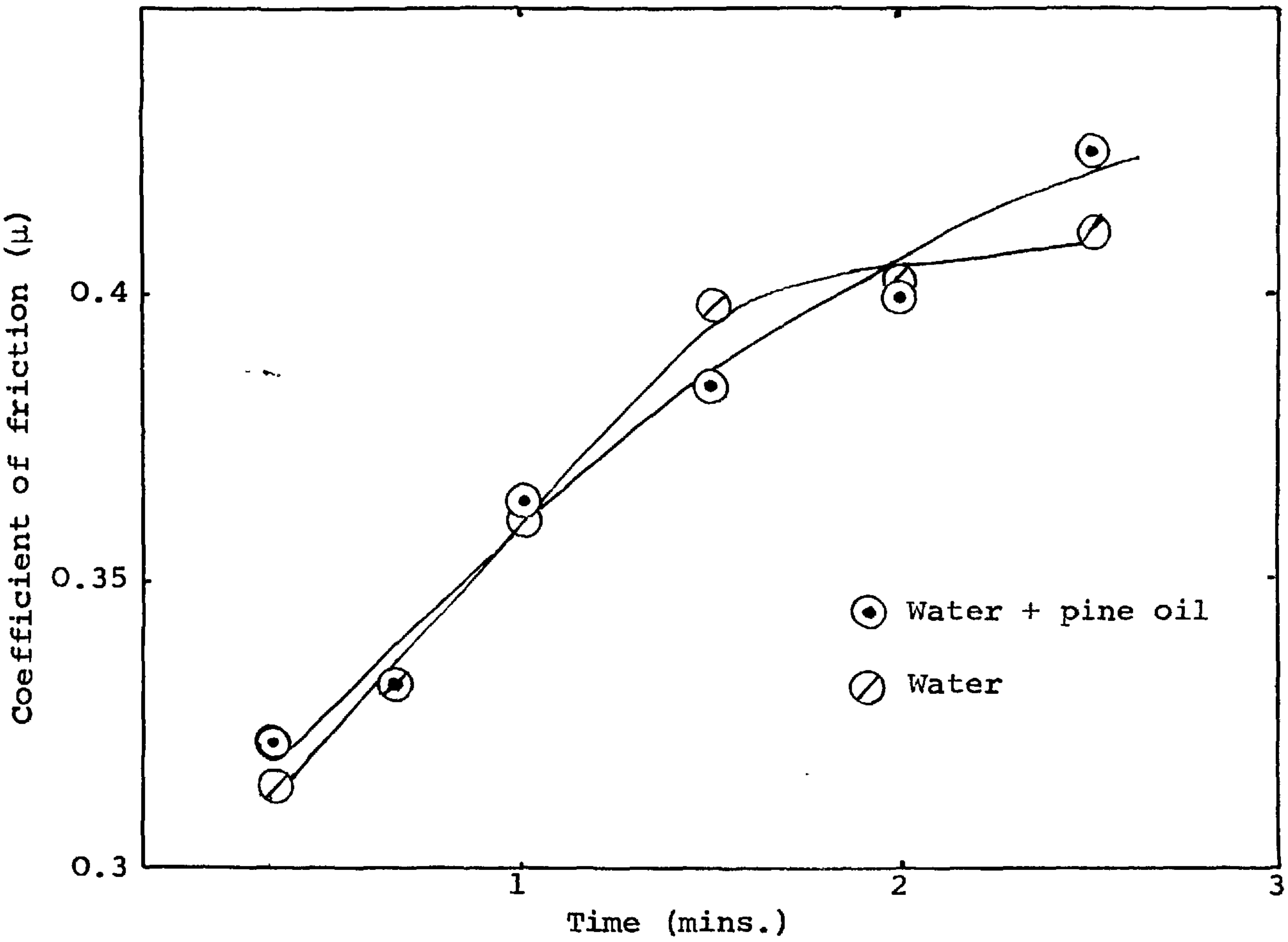


Fig.A1.4 Increase in friction with time.

same order, which helps to confirm the measurements. The difference was probably due to hydrodynamic influences at high speeds. A suggested explanation is that the wedge shape of the leading face of the hemispheres (which was chosen to simulate the form of the balls at the mill lining) probably induced a lifting effect over the layer of media. This tended to reduce the effective pressure on the points of contact of the trolley. The fact that the discrepancy widened as the viscosity increased supported this theory.

The series of tests with a range of concentrations of quartz particles gave results which exhibited a striking resemblance to those obtained for the efficiency of grinding. In other words, the variation of the coefficient of friction with the viscosity of the quartz suspension followed a similar pattern to the variation of the efficiency of grinding with the viscosity of the pulp. This is particularly true in the range 0 - 100 cP. Beyond 100 cP the effects of cushioning and ball mobility become important and exert additional influences. The gradual decrease in efficiency of grinding beyond about 150 cP may be partly due to a gradual decrease in the coefficient of friction. This decrease was only observed, however, with aqueous glycerol and not with suspensions with quartz.

The results of the concentration experiments show that the frictional resistance is affected significantly by the nature of the layer of media between the metal surfaces. Firstly, at very low concentrations (equivalent to $<$ 1 kg in the mill) the particles are separate and represent individual obstructions in the path of a moving body. There is an optimum concentration when the number of obstructions, and hence, the friction, is a maximum yet the particles are still separate (about 1 kg). Secondly, as the concentration becomes such that the particles represent a bed of unit thickness, then the resistance to motion decreases because the trolley slides smoothly over a continuous layer. Eventually, the bed becomes too thick and the hemispheres are forced to plough a way through it. This model gives a clear representation of the influence of the viscosity of the pulp on the operation of ball-mills.

The results of particle size tests were inconclusive, possibly because of the concentration used. One would have expected some variation in the results, however, even at high concentrations if there was any effect due to variations in the particle size.

The influence of additives on the friction was not in agreement with the conclusions from the grinding tests. The foam produced by the "Quix" appeared to reduce the effective concentration of particles per unit volume of coating applied. Hence, the reduction in concentration increased the friction. The influence of the pine oil on the quartz suspensions was probably obscured by the presence of glycerol. A possible solution to this problem would be the use of stainless steel surfaces which would obviate the need for the glycerol.

The influence of pine oil on the friction between steel surfaces was found to be negligible. Thus, the effect exerted by pine oil in the grinding tests was surely due to its influence on the quartz.

Although it does not affect the main investigation, the increase in the coefficient of friction with the rate of corrosion was interesting. This could provide a clue to differences in the performance of mills with stainless steel grinding surfaces and those of mild steel, for example.

The present investigation gives a further reason for the difference in operation of ball-mills and rod-mills from a consideration of the friction. The ball-mill makes use of point contacts between the grinding bodies which are affected by low concentrations of particles. Rod-mills on the other hand, use line contacts which may possibly eliminate the high friction of separate obstructions. This would not, however, account for the results obtained by Hockings, Volin and Mular (123), who obtained no variation in the efficiency response with a rod mill over the range of viscosity 1 - 50 cP, as they used a constant charge of material.

The results obtained agree with those of von Szantho (6) who showed that the coefficient of friction followed a similar response to the production of new surface with varying percentages of chemical additive. There does not, however, appear to be any other published work on the influence of the nature of the pulp on the friction between metal surfaces.

The friction-measuring technique gives an indication of the influence of the nature of the pulp on the contacts between the grinding surfaces. Hence, the influence of pulp viscosity, additives, different minerals etc. may be studied quickly and easily. In other words, an approximate indication may be obtained of how efficiently a mineral would be ground without the necessity for carrying out a full grinding test in a mill.

The results from the present series of tests, although subject to many limitations of experimental error, show clearly the importance of the degree of friction between the grinding surfaces.

Tables of results of friction experiments.

Values of μ, the nominal coefficient of dynamic friction, are calculated from the formula:

$$\mu = \frac{w}{W} - \frac{(w + W)}{Wg} \cdot \frac{2x}{t^2}$$

where w is the balance weight (172.8 or 122.8 gm), W is the trolley weight (388.2 gm), x is the distance of traverse (36.4 cm), t is the time of traverse (secs) and g is the acceleration due to gravity (981 cm/sec^2).

CONT/...

Table 1

Media: aqueous glycerol.

Balance weight 172.8 gm.

Viscosity (cP)	2.63	4.99	9.29	15.59	25.22	56.30	121.17	1210
Stop-clock readings (secs)	.75	.66	.63	.62	.62	.62	.62	.51
	.68	.70	.66	.61	.63	.61	.58	.51
	.69	.69	.62	.65	.63	.61	.65	.52
	.67	.70	.63	.63	.62	.63	.58	.51
	.72	.69	.64	.61	.61	.63	.59	.53
		.65	.64	.63	.64	.61	.61	.52
		.66	.65	.59	.64	.62	.60	.54
		.66	.64	.62	.59	.63	.59	.52
		.67	.66	.58	.58	.62	.59	.52
		.65	.61	.62	.61	.62	.61	.52
Average Time	.702	.673	.638	.617	.617	.62	.60	.52
μ	0.226	0.207	0.18	0.162	0.162	0.164	0.145	0.045

Table 2

Media: aqueous glycerol.

Balance weight 122.8 gm.

Viscosity (cP)	2.63	4.99	9.29	15.59	25.22	56.3	121.17	1210
Stop-clock readings (secs)	1.41	1.1	1.04	0.9	0.92	1.00	1.1	0.75
	1.23	1.15	1.00	0.91	0.96	0.96	0.99	0.74
	1.15	1.14	1.07	0.98	0.98	0.96	1.02	0.74
	1.40	1.12	1.22	0.98	0.92	0.91	1.03	0.71
	1.44	1.12	1.07	1.03	1.03	0.95	0.99	0.72
		1.13	1.13	0.96	0.92	0.96	0.89	0.77
		1.16	1.01	1.00	0.94	0.94	0.98	0.73
		1.14	1.04	0.95	0.99	0.98	0.91	0.73
		1.14	1.08	1.00	0.93	0.93	0.99	0.72
		1.10	1.00	0.99	0.92	0.96	0.93	0.70
Average time	1.326	1.13	1.066	0.973	0.951	0.955	0.967	0.731
μ	0.26	0.239	0.230	0.212	0.208	0.209	0.211	0.132

Table 3

Media: aqueous glycerol + quartz (150 - 200 mesh).

Balance weight 172.8 gm.

Vol. % Solid	5.8	11.6	17.3	23.1	34.6	46.1	52.0
Stop clock readings	1.03	0.73	0.67	0.68	0.72	0.74	0.76
	0.97	0.68	0.63	0.67	0.74	0.74	0.79
	1.02	0.67	0.68	0.68	0.72	0.75	0.75
	1.03	0.68	0.64	0.69	0.70	0.75	0.77
	1.00	0.71	0.65	0.68	0.70	0.75	0.78
	0.98	0.75	0.64	0.68	0.71	0.75	0.76
	0.93	0.72	0.65	0.68	0.71	0.77	0.79
	1.12	0.67	0.62	0.67	0.71	0.78	0.75
	0.98	0.74	0.65	0.68	0.70	0.73	0.75
	0.95	0.68	0.64	0.67	0.71	0.76	0.73
Average time	1.001	0.703	0.647	0.678	0.712	0.752	0.763
μ	0.337	0.226	0.187	0.210	0.232	0.254	0.259

Table 4

Media: aqueous glycerol + quartz.

Balance weight 172.8 gm.

Vol. % solid: 48.

Particle size	200-300#	150-200#	100-150#	72-100#	52-72#
Stop-clock readings (secs)	0.77	0.74	0.75	0.73	0.8
	0.78	0.74	0.75	0.77	0.8
	0.74	0.76	0.77	0.74	0.82
	0.77	0.75	0.74	0.75	0.77
	0.77	0.78	0.76	0.75	0.79
	0.75	0.74	0.73	0.74	0.82
	0.76	0.74	0.70	0.77	0.78
	0.75	0.73	0.74	0.74	0.77
	0.76	0.74	0.75	0.75	0.78
	0.76	0.70	0.74	0.75	0.76
Average time	0.761	0.742	0.743	0.749	0.745
μ	0.259	0.249	0.249	0.252	0.250

Table 5

Media: aqueous glycerol + quartz and pine oil.

Balance weight: 172.8 gm.

Vol. % solid: 48.

Particle size	200-300#	150-200#	100-150#	72-100#	52-72#
Stop-clock readings (secs)	0.78	0.76	0.79	0.85	0.69
	0.77	0.72	0.78	0.82	0.81
	0.75	0.76	0.79	0.83	0.82
	0.77	0.69	0.81	0.81	0.75
	0.79	0.75	0.80	0.84	0.78
	0.81	0.74	0.84	0.80	0.81
	0.81	0.75	0.83	0.83	0.81
	0.77	0.76	0.81	0.86	0.76
	0.77	0.75	0.82	0.82	0.76
	0.76	0.73	0.78	0.84	0.75
Average time	0.778	0.743	0.805	0.830	0.774
μ	0.266	0.249	0.278	0.288	0.265

Table 6

Media: aqueous glycerol + quartz with detergent "Quix".

Balance weight: 172.8 gm.

Vol. % solid: 48.

Particle size	200-300#	150-200#	100-150#	72-100#	52-72#
Stop-clock readings	0.83	0.81	0.71	0.73	0.75
	0.81	0.86	0.73	0.74	0.75
	0.74	0.87	0.73	0.76	0.77
	0.78	0.81	0.75	0.80	0.73
	0.76	0.77	0.75	0.75	0.79
	0.75	0.78	0.75	0.75	0.80
	0.83	0.77	0.77	0.77	0.78
	0.86	0.78	0.77	0.78	0.76
	0.81	0.82	0.76	0.78	0.76
	0.82	0.86	0.78	0.80	0.81
Average time	0.799	0.813	0.750	0.766	0.770
μ	0.276	0.282	0.253	0.261	0.263

Table 7

Media: distilled water with and without pine oil.

Balance weight: 172.8 gm.

Time (secs) between readings	Stop-clock readings. water	Stop-clock readings. water + pine oil.	μ water	μ water + pine oil.
20	0.91	0.88	0.314	0.322
40	1.01	1.01	0.338	0.338
60	1.13	1.33	0.361	0.364
90	1.52	1.78	0.398	0.384
120	1.61	2.40	0.403	0.400
150	1.78	5.42	0.411	0.425

Appendix 2.

TABLES OF RESULTS.

Viscosity

The values of viscosity for the six aqueous glycerol, calibration solutions were (1) 2.085 (2) 7.446 (3) 19.55 (4) 42.66 (5) 86.3 and (6) 152.38 cP. Typical calibration curves are shown in Figs. A2.1 and A2.2.

Table 1. Calibration readings for cylinder VLB with stirrer speed 400 r.p.m.

Cylinder speed	1	2	3	4	5
Sol$^{\underline{n}}$ (1)	6.5	6.0	5.5	4.9	4.5
" (2)	19.0	16.2	13.6	11.0	9.0
" (3)	45.6	38.8	32.1	25.3	17.8
" (4)	95.0	81.3	67.0	52.0	36.5
" (5)	-	-	-	93.2	65.1
" (6)	-	-	-	-	98.5

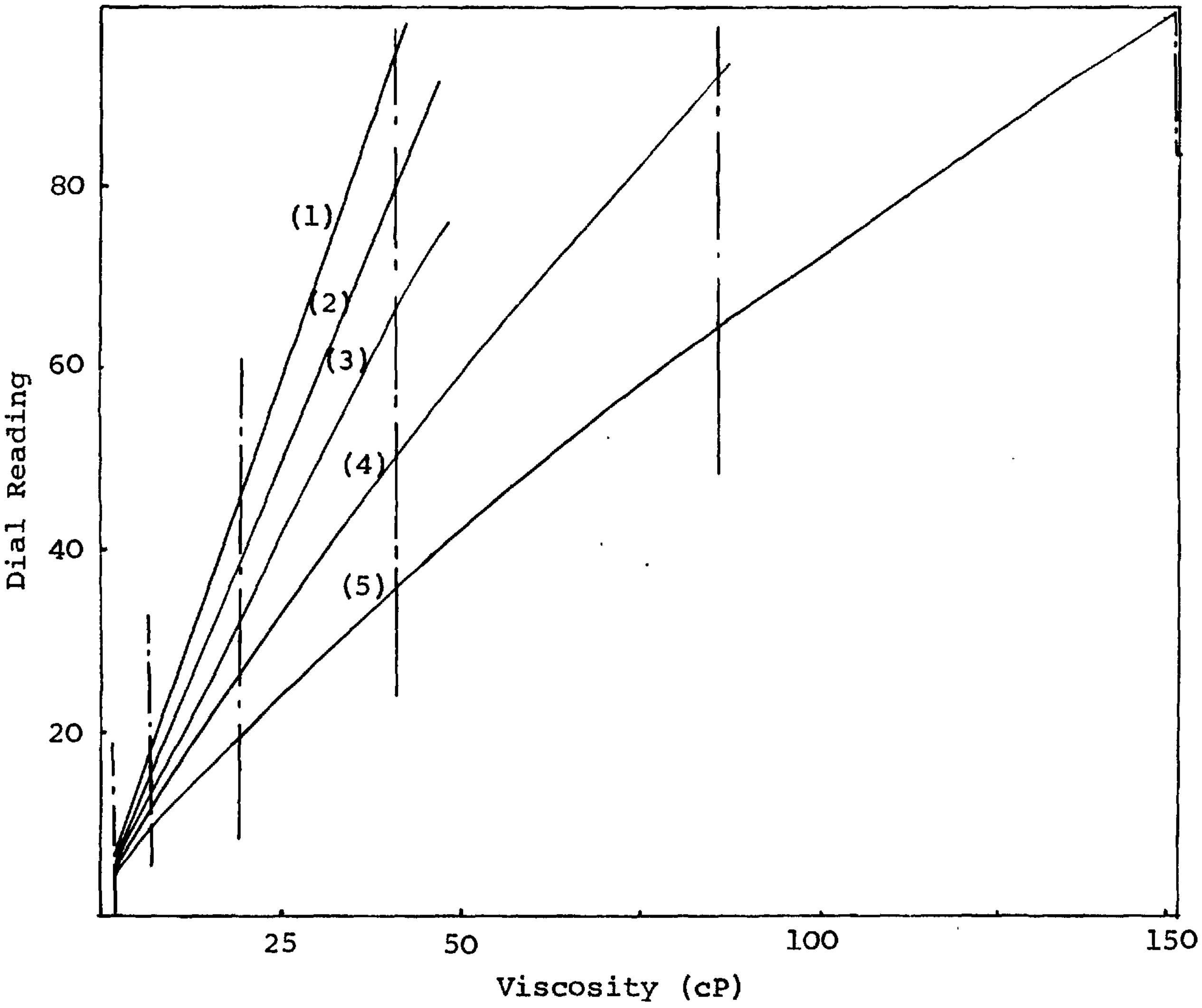


Fig.A2.1 Calibration Chart for Inner Cylinder VLB at 400 r.p.m. stirrer speed with 5 shear-rates.

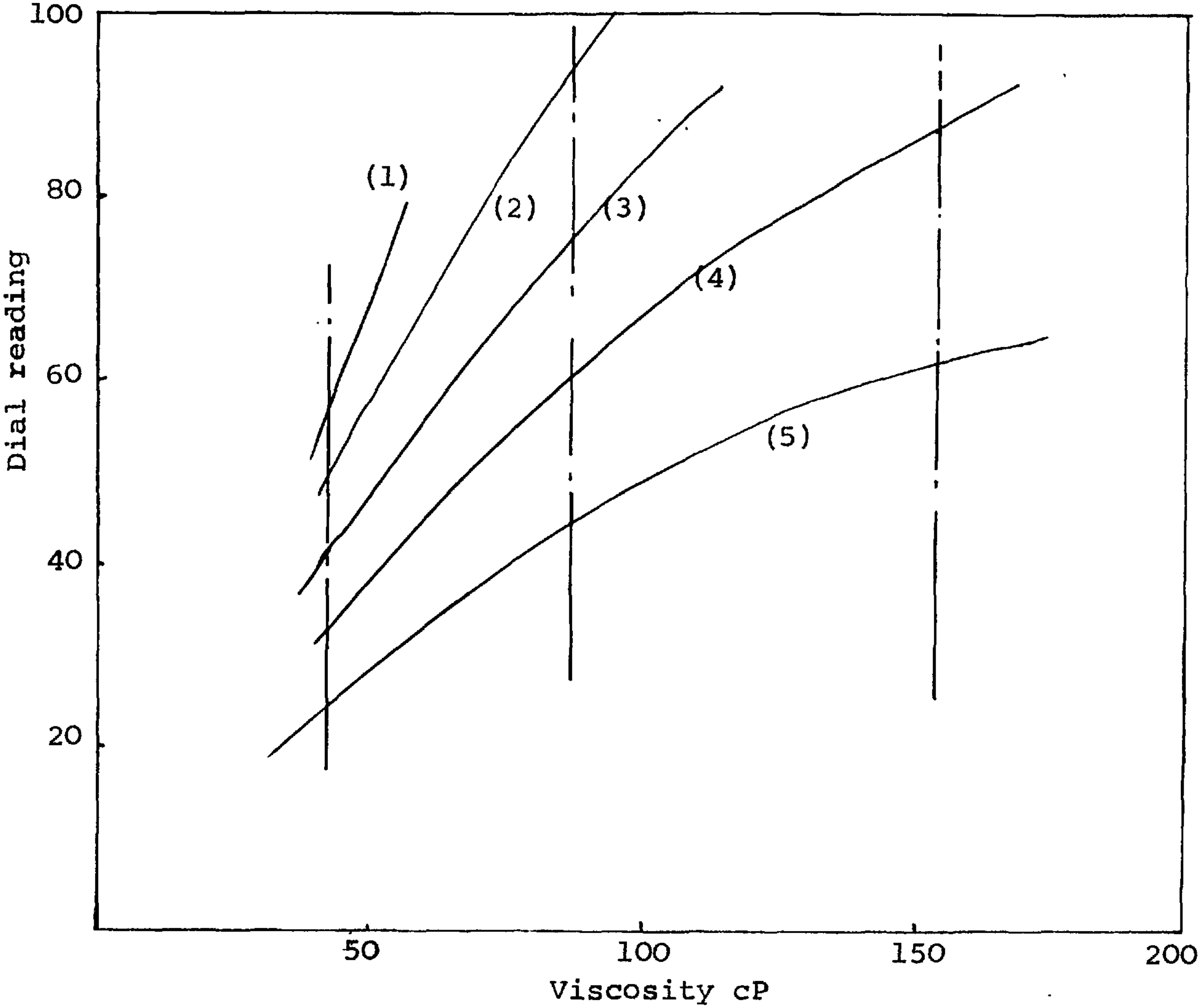


Fig.A2.2 Calibration chart for inner cylinder VLC at 500 r.p.m. Stirrer speed

Table 2. Calibration readings for cylinder VLC with stirrer speed 400 r.p.m.

Cylinder speed	1	2	3	4	5
Sol$^{\underline{n}}$ (1)	3.8	3.6	3.4	3.2	2.8
" (2)	8.7	8.2	7.8	6.8	5.4
" (3)	25.5	22.3	18.9	15.5	10.8
" (4)	54.0	46.8	39.1	31.5	22.7
" (5)	98.0	84.2	69.4	56.0	41.6
" (6)	-	-	-	81.0	56.9

Table 3. Calibration readings for cylinder VMC with stirrer speed 400 r.p.m.

Cylinder speed	1	2	3	4	5
Sol$^{\underline{n}}$ (1)	-	-	-	-	-
" (2)	5.5	4.65	4.4	3.9	3.0
" (3)	14.0	12.4	10.5	8.5	7.0
" (4)	32.0	28.0	23.0	19.0	14.5
" (5)	58.0	51.4	43.0	35.0	26.0
" (6)	89.9	76.5	62.2	48.5	34.0

Table 4. Calibration readings for cylinder VLC with stirrer speed 500 r.p.m.

Cylinder speed	1	2	3	4	5
Sol$^{\underline{n}}$ (1)	-	-	-	-	-
" (2)	-	-	-	-	-
" (3)	-	-	-	-	-
" (4)	57.0	50.0	41.5	33.25	24.5
" (5)	-	97.5	76.0	61.5	45.0
" (6)	-	-	-	88.5	63.5

Table 5.

Quartz suspended in water (Tables 5 - 10)
Particle size:- $-72^{\#} + 100^{\#}$
Stirrer speed:- 400 r.p.m.
Inner cylinder:- VLB

Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
5%	1	8.6	2.9	10%	1	12.5	4.6
	2	7.5	2.8		2	9.5	3.8
	3	7.0	3.0		3	7.8	3.5
	4	6.5	3.3		4	7.1	3.8
	5	5.6	3.3		5	6.5	4.2
15%	1	18.5	7.1	20%	1	27.5	11.25
	2	13.5	5.0		2	20.5	9.6
	3	10.2	5.1		3	15.3	8.7
	4	8.5	5.1		4	10.5	6.9
	5	7.6	5.6		5	8.5	6.8
25%	1	43.0	18.3	27.5%	1	72	31
	2	31.5	15.5		2	50	26
	3	21.5	12.7		3	31.2	19.6
	4	17.0	12.7		4	18.5	13.9
	5	10.0	8.8		5	11.5	10.6
30%							
	2	88.0	45				
	3	57.0	34.5				
	4	36.0	27				
	5	21.0	22				

Table 6

Particle size:- $-100^{\#} + 150^{\#}$

Stirrer speed:- 400 r.p.m.

Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
5%	1	6.2	1.9	10%	1	9.5	3.3
	2	5.3	1.75		2	7.0	2.5
	3	4.8	1.6		3	5.8	2.3
	4	4.5	1.7		4	5.5	2.5
	5	4.0	1.6		5	5.0	2.6
15%	1	13.5	5.0	20%	1	21.5	8.6
	2	9.6	3.9		2	15.7	7.1
	3	7.6	3.45		3	10.5	5.3
	4	6.7	3.45		4	7.8	4.4
	5	5.9	3.6		5	6.5	4.3
25%	1	34.5	14.4	27.5%	1	55.0	23.3
	2	25.5	12.3		2	37.5	18.8
	3	17.7	10.2		3	26.0	15.6
	4	11.2	7.7		4	16.5	12.3
	5	7.7	5.65		5	10.5	9.3
	500	r.p.m.					
30%	1	100	45				
	2	77	38				
	3	52	31.5				
	4	34.5	26.0				
	5	21.5	22.5				

Table 7.

Particle size:- $-150^{\#} + 200^{\#}$

Stirrer speed:- 400 r.p.m.

Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
5%	1	6.5	2.0	10%	1	8.0	2.7
	2	5.4	1.75		2	6.3	2.3
	3	4.9	1.68		3	5.4	2.0
	4	4.5	1.7		4	4.7	1.85
	5	3.5	1.4		5	4.0	1.6
15%	1	11.5	4.2	20%	1	18.0	7.0
	2	8.4	3.2		2	13.5	5.9
	3	6.9	2.9		3	8.8	4.15
	4	6.3	3.2		4	7.3	3.9
	5	5.1	2.8		5	6.0	3.8
25%	1	28.0	11.5	27.5%	1	36.0	15.1
	2	21.0	10.0		2	28.0	13.6
	3	14.0	7.7		3	20.5	12.0
	4	9.0	5.6		4	12.3	8.7
	5	6.8	4.6		5	8.8	7.1
30%	1	71·0	30.5	32.5%	1	-	-
	2	54.0	27.0		2	98.5	52.5
	3	36.5	21.5		3	67.5	43.0
	4	23.5	18.0		4	46.0	37.0
	5	14.3	14.0		5	29.5	33.0

Table 8

Particle size:- - 200$^{\#}$ + 300$^{\#}$

Stirrer speed:- 400 r.p.m.

Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
5%	1	6.1	1.9	10%	1	6.8	2.2
	2	5.6	1.8		2	5.8	1.9
	3	5.0	1.7		3	5.3	1.9
	4	4.4	1.7		4	4.8	1.9
	5	4.0	1.6		5	4.1	1.8
15%	1	8.8	3.0	20%	1	14.5	5.4
	2	7.2	2.7		2	10.2	4.3
	3	6.0	2.4		3	7.0	3.8
	4	5.4	2.5		4	6.0	3.0
	5	4.4	2.0		5	4.7	2.3
25%	1	27.0	11.0	27.5%	1	39.0	16.4
	2	20.0	9.5		2	29.5	14.4
	3	12.6	6.7		3	20.0	11.8
	4	8.5	4.7		4	12.7	9.0
	5	6.0	3.8		5	8.0	6.2
30%	1	75.0	33				
	2	55.5	28				
	3	37.5	23				
	4	23.0	17				
	5	14.5	15				

Table 9.

Particle size:- − $350^{\#}$ + 10μ

Stirrer speed:- 400 r.p.m.

Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
5%	1	4.8	1.3	10%	1	5.2	1.5
	2	4.5	1.3		2	4.9	1.5
	3	3.8	1.2		3	4.6	1.5
	4	3.5	1.2		4	4.0	1.5
	5	3.0	1.2		5	3.3	1.4
15%	1	5.6	1.6	20%	1	9.5	3.3
	2	5.1	1.6		2	7.2	2.7
	3	4.8	1.6		3	6.4	2.6
	4	4.3	1.6		4	5.5	2.5
	5	3.6	1.5		5	4.5	2.1
25%	1	20.5	8.2	30%	1	48.0	26.0
	2	15.0	6.8		2	36.0	17.9
	3	11.0	5.7		3	26.0	16.0
	4	9.0	5.0		4	17.4	12.9
	5	7.2	5.0		5	12.3	11.6
32.5%	1	80.0	35.0	35%	1		
	2	61.5	29.0		2		
	3	42.5	26.7		3	100	70
	4	30.0	23.7		4	64.5	56
	5	20.5	23.9		5	51	62

Table 10.

Particles:- glass rods $\rho = 2.01$
Stirrer speed:- 400 r.p.m.
Inner cylinder:- VLB

Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
5%	1	7.8	2.6	10%	1	10.2	3.6
	2	6.8	2.4		2	7.9	3.5
	3	5.8	2.2		3	6.2	2.6
	4	4.8	1.9		4	5.0	2.1
	5	3.8	1.6		5	3.8	1.6
15%	1	18.5	7.2				
	2	13.5	6.4				
	3	10.3	5.1				
	4	8.2	4.8				
	5	6.0	3.8				

Inner cylinder:- VLC

Stirrer speed: 500 r.p.m. Stirrer speed: 600 r.p.m.

17.5%	1	9.0	7.6	20%	1	37.0	26.0
	2	7.5	6.0		2	26.0	21.0
	3	5.0	6.0		3	17.5	18.2
	4	3.9	5.0		4	10.5	11.0
	5	3.3	5.0		5	5.0	4.0

Table 11.

Particles:- glass plates $\rho = 2.52$
Stirrer speed:- 400 r.p.m.
Inner cylinder:- VLB

Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
5%	1	6.0	1.8	10%	1	9.5	3.3
	2	5.0	1.6		2	7.0	2.6
	3	4.5	1.4		3	5.4	2.0
	4	3.7	1.3		4	4.5	1.7
	5	3.0	1.2		5	3.7	1.4
15%	1	18.0	7.0	20%	1	38.0	16.0
	2	12.0	5.15		2	26.0	12.6
	3	7.7	3.3		3	15.3	8.6
	4	6.0	3.0		4	9.6	6.2
	5	5.0	2.7		5	6.8	4.6
25%	1	-	-				
	2	94.0	43.0				
	3	60.0	34.0				
	4	33.5	26.5				
	5	20.0	23.0				

Table 12.

Particles:- glass spheres $-200^{\#} + 300^{\#}$ $\rho = 2.36$
Stirrer speed:- 400 r.p.m.
Inner cylinder:- VLB

Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
5%	1	5.5	1.6	10%	1	7.0	2.3
	2	5.2	1.6		2	6.5	2.3
	3	5.0	1.7		3	5.5	2.1
	4	4.6	1.7		4	4.9	2.0
	5	3.6	1.2		5	4.3	1.9
15%	1	8.3	2.8	20%	1	11.0	4.0
	2	7.1	2.6		2	8.5	3.3
	3	6.5	2.7		3	7.2	3.1
	4	5.8	2.8		4	6.5	3.4
	5	5.0	2.7		5	6.0	3.8
25%	1	13.5	5.0	30%	1	18.4	7.2
	2	10.3	4.2		2	14.5	6.5
	3	8.5	4.0		3	10.9	5.5
	4	7.5	4.2		4	8.7	5.3
	5	6.5	4.0		5	7.5	5.5
35%	1	28.0	11.5	40%	1	43.0	18.2
	2	20.5	9.7		2	31.8	15.6
	3	14.5	8.1		3	23.0	13.7
	4	11.0	7.4		4	15.5	11.5
	5	8.2	6.5		5	11.0	10.0

Table 13.

Particles:- glass spheres - 200# + 300# ρ = 2.96

Stirrer speed:- 400 r.p.m.

Inner cylinder:- VLB

Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
5%	1	5.2	1.5	10%	1	6.2	2.0
	2	4.9	1.5		2	5.9	2.0
	3	4.5	1.4		3	5.6	2.0
	4	3.5	1.0		4	4.8	2.0
	5	3.3	1.0		5	4.2	1.9
15%	1	8.0	2.7	20%	1	24.5	9.9
	2	6.7	2.3		2	16.8	7.8
	3	6.2	2.5		3	11.4	5.9
	4	5.5	2.5		4	9.4	5.9
	5	4.5	2.2		5	7.8	5.9
25%	1	16.0	6.1	30%	1	24.5	9.9
	2	12.0	5.1		2	16.8	7.8
	3	9.0	4.3		3	11.4	5.9
	4	7.1	4.0		4	9.4	5.9
	5	6.3	4.1		5	7.8	5.9
35%	1	37.2	15.6	40%	1	59.0	23.6
	2	27.0	13.1		2	46.0	21.0
	3	17.4	10.1		3	32.0	19.4
	4	12.0	8.8		4	19.8	15.0
	5	9.6	8.2		5	13.2	12.8
45%	1			50%	1		
	2	90.0	45.5		2		
	3	60.0	35.0		3		
	4	40.0	28.0		4		
	5	27.0	25.0		5	95	180

Table 14.

Particles:- polymethylmethacrylate spheres ρ = 1.168

Stirrer speed:- 400 r.p.m.

Inner cylinder:- VLB

Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
5%	1	5.5	1.6	10%	1	5.5	1.6
	2	5.2	1.7		2	5.2	1.7
	3	4.5	1.4		3	4.8	1.6
	4	4.0	1.3		4	4.3	1.6
	5	3.7	1.6		5	4.0	1.7
15%	1	5.5	1.6	20%	1	5.7	1.7
	2	5.2	1.7		2	5.2	1.7
	3	4.8	1.6		3	4.9	1.7
	4	4.3	1.6		4	4.5	1.7
	5	4.0	1.7		5	4.3	1.8
25%	1	7.5	2.5	30%	1	9.6	3.4
	2	7.0	2.5		2	8.7	3.4
	3	6.5	2.7		3	7.6	3,6
	4	5.5	2.4		4	6.4	3.4
	5	5.0	2.6		5	5.8	3.5
35%	1	13.5	5.0	40%	1	18.5	7.2
	2	11.4	4.8		2	16.0	7.3
	3	10.3	5.2		3	13.8	7.5
	4	8.6	5.1		4	10.9	7.3
	5	6.8	4.7		5	8.5	6.8
45%	1	30.0	12.4	50%	1	61.5	26.5
	2	25.0	12.0		2	52.0	27.5
	3	20.8	12.2		3	41.0	25.6
	4	16.6	11.4		4	32.5	25.8
	5	11.5	10.6		5	22.5	27.6

Table 15.

Particles:- polymethylmethacrylate spheres (ρ = 1.168)
Stirrer speed:- 400 r.p.m.
Inner cylinder:- VLB
Suspending fluid:- distilled water + anionic detergent.

0% detergent				2% detergent			
Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
50%	1	61.5	26.5	50%	1	44.0	18.8
	2	52.0	27.5		2	37.0	18.5
	3	41.0	25.6		3	29.4	17.6
	4	32.5	25.8		4	22.0	16.8
	5	22.5	27.6		5	15.3	15.6
4% detergent				6% detergent + 2gm BaCl			
50%	1	45.5	19.5	50%	1	54	24.2
	2	37.5	18.7		2	45.5	23.3
	3	30.0	18.1		3	36.5	22.5
	4	22.5	17.2		4	27.0	21.0
	5	15.5	17.0		5	18.5	19.8
6% detergent + 5gm BaCl							
50%	1	54.0	24.2				
	2	45.5	23.3				
	3	36.5	22.5				
	4	27.0	21.0				
	5	19.0	20				

Table 16.

Particles:- Quartz mixture -100# + 150# and -350# + 10μ
Stirrer speed:- 400 r.p.m.
Inner cylinder:- VLB

Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
100% -100# + 150#				80% -100# +150# 20% -350# +10μ			
20%	1	21.5	8.6	20%	1	17.2	6.6
	2	16.0	7.3		2	12.5	5.4
	3	10.5	5.3		3	9.0	4.3
	4	7.8	4.6		4	7.4	4.2
	5	7.0	4.9		5	6.5	4.3
65% -100# +150# 35% -350# +10μ				50% -100# +150# 50% -350# +10μ			
20%	1	14.4	5.4	20%	1	12.9	4.7
	2	10.2	4.2		2	9.0	3.6
	3	8.2	3.9		3	7.2	3.1
	4	7.4	4.1		4	6.6	3.1
	5	6.6	4.4		5	6.0	3.8
35% -100# +150# 65% -350# +10μ				20% -100# +150# 80% -350# +10μ			
20%	1	12.2	4.5	20%	1	12.0	4.4
	2	8.8	3.5		2	9.0	3.6
	3	7.7	3.5		3	7.8	3.6
	4	7.0	3.7		4	6.9	3.7
	5	6.0	3.8		5	6.4	4.1
100% -350# + 10μ							
20%	1	11.4	4.1				
	2	8.8	3.5				
	3	7.4	3.3				
	4	6.9	3.6				
	5	6.4	4.1				

Table 17.

Particles:- $-200^{\#}$ $+300^{\#}$ Quartz.
Stirrer speed:- 400 r.p.m.
Inner cylinder:- VLB
Suspending fluid:- distilled water + 0.14% solid anionic detergent.

Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.%	Cyl. speed	Dial reading	Viscosity (cP)
5%	1	6.8	2.1	10%	1	7.8	2.5
	2	6.3	2.2		2	6.4	2.3
	3	5.8	2.2		3	5.9	2.3
	4	5.3	2.2		4	5.3	2.4
	5	4.6	2.1		5	4.7	2.2
15%	1	10.5	3.7	20%	1	16.5	6.3
	2	8.1	3.1		2	11.0	4.6
	3	7.1	3.1		3	9.0	4.3
	4	6.7	3.4		4	8.1	4.7
	5	6.0	3.8		5	7.8	5.6
25%	1	24.0	9.6	30%	1	38.0	16.0
	2	17.5	8.2		2	29.0	14.1
	3	12.2	6.5		3	20.0	11.7
	4	9.0	5.5		4	13.0	9.3
	5	7.3	5.3		5	9.5	8.0
32.5%	1	51.5	22.2	35%	1	84.0	37.0
	2	39.5	19.9		2	67.5	35.0
	3	25.7	17.0		3	48.5	29.5
	4	17.3	13.0		4	28.0	22.0
	5	13.0	12.5		5	18.5	20.0

Table 18.

Particles:- Glass spheres $-150^{\#} + 200^{\#}$ $\rho = 2.96$.
Stirrer speed:- 400 r.p.m.
Inner cylinder:- VLB
Suspending fluid:- distilled water and detergent.
Concentration of solids:- 40 vol.%

Conc. Vol.% solid detergent	Cyl. speed	Dial reading	Viscosity (cP)	Conc. Vol.% solid detergent	Cyl. speed	Dial reading	Viscosity (cP)
0.0	1	67	29.0	0.4	1	55.5	23.6
	2	49.5	25.5		2	37.6	19.0
	3	33.0	20.0		3	29.0	17.5
	4	23.0	17.8		4	20.0	15.1
	5	15.5	16.0		5	13.5	13.7
0.04	1	57.5	25.0	0.7	1	57.0	24.0
	2	38.0	19.0		2	37.0	18.5
	3	30.0	18.0		3	29.0	17.5
	4	21.0	15.0		4	20.5	15.5
	5	14.5	14.5		5	13.6	13.7
0.1	1	54.5	23.3				
	2	36.5	17.9				
	3	29.0	17.4				
	4	20.5	15.6				
	5	14.0	13.9				
0.2	1	54.0	23.0				
	2	35.3	17.5				
	3	28.0	16.8				
	4	19.5	14.8				
	5	13.5	13.2				

Grinding

Table 19. Results of tests to find optimum charge of quartz with artificial media.

Medium	Centrifuging speed - r.p.m.
Water	150+
" + ½ kg. quartz	108
" + ¾ "	103
" + 1 "	100
" + 1½ "	100
" + 2 "	99
Aq. glycerol (250 cP Approx.)	150+
" + ½ kg. quartz	102
" + 1 "	90
" + 1½ "	87
" + 2½ "	82
Aq. glycerol (800 cP Approx.)	150+
" + ½ kg. quartz	90
" + 1 "	78
" + 1½ "	78
" + 2½ "	76

Table 20

Results of centrifuging speed tests with aqueous glycerol + 1 kg of quartz.

Viscosity cP	Centrifuging speed r.p.m.	Max.Noise (Volts)	Max.Torque (ft.lb.)
1	96	1.42	26.5
1.9	102	1.5	24.7
4.42	103	1.55	26.0
5.85	105	1.65	29.0
8.1	107	1.65	28.5
10.6	110	1.95	27.3
17.6	110	1.95	27
22.4	110	1.95	27
29.8	110	1.95	28
35.5	110	1.95	26.8
42.2	110	1.95	27
58.0	110	1.86	28
80.0	110	1.80	27.2
102.6	105	1.70	27.75
137	102	1.65	28.0
182.5	98	1.62	28.8
262	94	1.45	29.9
400	85	1.15	30.5
885	78	1.03	

Table 21

Cumulative analysis % weight smaller than stated sieve size for constant speed tests (70 r.p.m., 500 revs.).

	Viscosity cP							
mesh	1cP	4.8	10	28	80	125	175	239
-¼	100	100	100	100	100	100	100	100
-14	80.8	79.3	69.5	71.3	81.0	85.9	93.9	96.8
-18	80.4	78.7	69.0	70.9	80.6	85.6	93.7	96.6
-25	79.9	78.2	68.4	70.4	80.1	84.9	93.2	96.0
-36	79.2	77.6	67.3	69.5	79.0	83.6	92.1	94.8
-52	78.0	75.9	65.4	67.2	76.0	80.2	88.8	90.9
-72	76.9	72.9	61.8	63.1	71.2	74.4	82.9	84.0
-100	74.3	67.1	56.1	56.2	64.0	66.4	74.0	74.1
-150	64.3	55.8	47.0	45.3	53.4	54.5	60.0	60.3
-200	53.8	46.2	39.8	36.0	45.0	45.4	49.3	49.5
-300	39.0	34.7	27.6	25.2	35.0	35.1	37.0	37.2
Average Torque ft.lb.	23	23	24	23.2	26.5	25.8	26.5	27.8
Average Noise - V	1.3	1.32	1.3	1.5	1.6	1.4	1.3	1.5
Temp. Incr. °C	1.1	1.0	1.1	0.9	1.1	1.2	1.3	1.4

Table 21, contd.

	Viscosity cP							
mesh	280	288	430	495	760	A*	B*	C*
-¼	100	100	100	100	100	100	100	100
-14	96.9	97.1	98.5	98.4	99.3	99.7	97.4	98.5
-18	96.6	97.0	98.4	98.2	99.2	99.7	97.3	98.4
-25	96.1	96.4	97.8	97.9	98.4	99.5	97.1	98.3
-36	95.0	95.3	96.5	96.2	95.8	99.0	96.7	98.2
-52	91.4	91.6	92.2	91.2	89.5	97.1	95.0	97.5
-72	85.0	85.5	84.9	83.1	78.7	91.8	90.7	95.0
-100	75.9	77.0	74.5	72.2	69.1	83.4	83.3	89.7
-150	61.9	64.0	60.0	57.4	54.4	69.1	70.7	78.5
-200	51.1	54.2	49.2	45.9	44.0	57.5	59.8	66.8
-300	38.7	43.1	37.6	33.0	32.5	45.4	46.7	51.3
Aver. Torque ft.lb.	26.4	27.2	27.4	27.4	27.5	30.5	29.5	29.2
Aver. Noise. Volts	1.2	1.3	1.3	1.3	1.4	0.9	0.8	1.0
Temp. Incr. °C	1.2	1.2	1.2	1.3	1.3	1.4	1.0	0.8

* A = Starch, B = Bentonite and C = Calcium Carbonate.

Table 22. Cumulative size analyses % weight smaller than stated sieve size for tests at 76% of the centrifuging speed.

	Viscosity cP								
mesh	1	11.81	20	35	80	132	325	326	685
-¼	100	100	100	100	100	100	100	100	100
-14	85.3	80.6	81.8	90.9	97.0	97.7	98.0	98.3	98.3
-18	85.0	80.2	81.5	90.7	96.9	97.6	97.9	98.2	98.1
-25	84.6	79.8	81.0	90.5	96.7	97.3	97.4	97.7	97.3
-36	84.2	79.0	80.2	89.8	95.8	96.6	96.1	96.1	95.1
-52	83.5	77.1	78.1	87.6	92.1	93.4	91.5	90.6	88.6
-72	82.2	73.5	74.0	82.8	85.2	87.0	84.4	82.6	79.9
-100	79.2	67.0	66.9	74.6	75.0	77.4	74.6	72.1	68.9
-150	70.7	55.7	55.2	61.0	60.4	62.7	59.5	58.2	54.3
-200	60.8	46.2	45.5	49.9	49.4	51.1	48.2	47.9	43.3
-300	46.9	35.0	33.4	36.7	36.5	37.7	35.3	35.9	31.7
Crit. speed r.p.m.	96	110	110	110	110	102	90	90	82
Aver. torque ft.lb.	28.5	28.5	29.9	26.6	27.7	30.1	31	30.8	31
Aver. noise volts	1.2	1.5	1.6	1.5	1.1	1.1	1.0	1.0	1.0
Temp. Incr. °C.	1.7		1.8	1.4	0.7	1.0	0.9	0.9	1.5

Table 23. Cumulative analyses % weight smaller than stated sieve size for "fines" tests at 76% of the centrifuging speed.

	Viscosity cP						
mesh	1	2	7	15	36	140	300
-¼	100	100	100	100	100	100	100
-14	85.3	79.1	74.9	70.4	70.0	93.1	96.3
-18	84.9	78.4	74.0	69.5	69.4	92.7	96.0
-25	84.6	77.8	73.4	68.7	68.6	92.3	95.4
-36	84.2	77.1	72.5	67.7	67.6	91.6	94.4
-52	83.5	75.9	70.8	65.8	65.9	89.9	91.4
-72	82.2	74.0	68.0	62.7	63.3	85.9	84.7
-100	79.2	70.0	62.9	57.5	58.4	76.8	73.0
-150	70.7	61.4	52.0	46.5	48.2	57.7	53.1
-200	60.8	51.0	34.4	32.6	37.7	42.4	32.9
Charge of 'fines'-kg.	0	2	4	5	6	6½	7
Critical speed r.p.m.	96	99	102	101	94	90	86
Average Torque ft.lb.	28.5	30.5	30	30.6	31.4	31.8	32.1
Average Noise - V	1.2	1.55	1.55	1.52	1.6	1.04	1.1
Temp. Incr. °C.	1.7	1.5	1.6	2.7	1.4	1.2	1.3

Table 24. Cumulative analyses % weight smaller than stated sieve size, for "fines" tests at 76% of the centrifuging speed with detergent or pine oil added.

	Detergent ("Quix") added				Pine oil added			
	0 kg	5 kg	6½ kg	7 kg	0 kg	5 kg	6½ kg	7 kg
Mesh	1 cP				1cP	15cP	96cP	245cP
-¼	100	100	100	100	100	100	100	100
-14	86.2	94.4	81.3	63.7	83.4	79.7	73.7	95.0
-18	85.9	93.6	77.3	58.0	83.0	79.0	75.9	94.8
-25	85.6	92.6	72.1	51.6	82.6	78.1	75.1	94.3
-36	85.2	90.7	66.1	45.2	82.1	77.1	74.1	93.7
-52	84.3	86.6	56.3	36.4	80.7	75.1	72.3	92.0
-72	82.8	79.7	47.5	29.3	77.7	71.7	69.3	88.0
-100	79.4	70.3	39.2	23.0	74.6	66.0	64.0	79.1
-150	71.5	55.8	29.9	15.6	66.8	55.3	52.5	59.2
-200	62.2	40.9	23.1	8.1	57.3	44.3	39.4	40.2
Crit. Speed R.P.M.	86	88	81	79	96	102	99	93.
Average Torque Ft.lb.	24.4	23.1	22.2	22.5	23.3	25.0	26.1	27.7
Average Noise Volts	15.6	7.3	12.7	8.5	12.8	10.4	15.1	9.9
Temp. Incr. °C	1.1	1.2	1.2	1.2	1.1	1.3	-	1.6

Table 25. Cumulative analyses % weight smaller than stated sieve size for full charge tests at 70% of critical speed.

						Pine oil added.	
Mesh	6 kg	7½ kg	8 kg	8½ kg	9 kg	8½ kg	9 kg
-¼	100	100	100	100	100	100	100
-14	92.9	88.1	90.5	92.1	92.0	92.9	94.3
-18	92.8	87.9	90.3	91.7	91.7	92.7	94.0
-25	92.7	87.7	90.0	91.5	91.5	92.4	93.6
-36	92.7	87.5	89.8	91.1	91.0	92.1	93.2
-52	92.5	87.3	89.5	91.0	90.3	91.6	92.4
-72	92.3	86.9	89.0	89.6	89.0	90.7	90.7
-100	91.7	86.1	87.7	87.2	85.2	88.1	85.5
-150	88.7	81.6	81.2	77.4	72.3	78.0	71.1
-200	80.1	72.9	71.0	65.1	60.3	66.2	59.2
-300	60.5	56.7	53.2	48.6	44.6	50.4	45.0
Torque (Av)	24.4	25.6	25.8	26.28	26.7	25.9	26.4
Viscosity (cP)	18.5	100	138	212	303	223	324
Noise (Av)	14.5	10.7	10.2	9.1	9.2	10.1	8.0
Critical speed r.p.m.	96	81	79	74	72	77	74
Temp. incr. °C	5.2	5.5	6.3	6.2	4.7	4.0	5.2

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