LATTICE FORMATION IN TWO-DIMENSIONAL DISPERSIONS

by

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ABSTRACT

The formation of ordered structures in monolayers of spheres, rods and discs on a horizontal vibrating plate have been studied over a range of surface concentration and particle axis ratio. The number and orientation distributions of the aggregates of cylindrical particles either resting on a solid surface or floating at a liquid surface were computed from photographs of the system at various times. It was found that a dynamic equilibrium was established where, at a given concentration, the degree of aggregation depended on the frequency of the vibrations but was independent of the axis ratio.

The number distribution of close packed hexagonal crystallites of spheres with time during vibration of a monolayer was studied as well as the formation and propagation of dislocations resulting in V-type and equilateral triangle-type crystallites when an initially close packed assembly was vibrated.

The number and orientation distributions of cylindrical chains or rouleaux of discs were measured in 3-dimensional assemblies subjected to vibration.

FOREWORD

This thesis describes the formation of highly organized structures in monolayers of spheres, rods and discs and in multiple layers of discs subjected to vibratory motion.

Part I is an introduction to the problem giving a brief review of the general background and setting forth the scope of the investigation. The main portion of the experimental work is presented in Part II, which has been written in a manner suitable for publication in the scientific literature with little or no further modification. Thus, the main part is complete with its abstract, introduction, discussion and references.

Additional details of the experimental apparatus and procedure are given in Appendices I and II.

Some concluding remarks and suggestions for further work are presented in Part III.

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PART I

GENERAL INTRODUCTION

Patterns of order resulting from arrays of geometrically regular figures have attracted the attention of man throughout the ages[1] and have stimulated his advance in the field of the physical and mathematical sciences as well as in the arts.

In many areas of scientific investigation such patterns of order are visible under the microscope as with clusters of living cells in a colony[2] or as tactoids in dispersions of colloidal particles[3]; in the submicroscopic region they may be inferred from X-ray diffraction patterns in the hexagonal or cubic close packed arrangement of atoms in the crystal lattice or in the rod-like structures of soap molecules within micellae in aqueous solution[4].

This thesis is concerned with a study of the ordering which results in two and three dimensional assemblies of uniform macroscopic particles of regular geometry when these are brought into relative motion by the application of mechanical vibrations. It is known that under conditions where the particles in the assembly have limited room to move and frequently collide with neighbouring members of the group, they can be aggregated into highly organized structures variously called lattices, mosaics and crystallites in which they are arranged in some kind of maximum density or minimum potential energy state.

A prerequisite for the formation of such structures appears to be the regular and uniform geometry of the particles. Thus an assembly of roughly cylindrical pulpwood logs in a holding boom or towing raft, in which the particles have approximately the same length but different diameters, forms a mosaic of aggregates (see Figure 1a, Part II) in which a number of logs are aligned with their long axes parallel. Litwiniszyn[5] showed that

a similar mosaic pattern of rods could be formed by vibrating pieces of wire of uniform length on a horizontal flat plate. He considered the pattern to be characterized by a distribution density function $g(\phi,t)$ giving the fraction of particles in the total assembly which at time t form an angle ϕ contained in the interval $(\phi,\phi+\Delta\phi)$ with an arbitrarily fixed direction.

Other examples of rod like particles forming ordered structures are the lamellar micelles of soap molecules in solution such as sodium oleate [4] and dispersions of colloidal particles such as FeO.OH aggregated into tactoids[3].

Regular structures are also formed by assemblies of disc-like particles such as the biconcave mammalian red blood cell (see Figure 1c, Part II) which aligns itself with its neighbours in a face to face orientation resulting in cylindrical chains called rouleaux[6]. Rouleaux of biconcave, non nucleated red blood cells have been observed <u>in vitro</u> and <u>in vivo</u> in many mammals but not in the nucleated amphibian biconvex cells in which the geometry is unfavourable for such aggregation. It was also shown in a very early investigation of the phenomenon[7] that small cork discs poised to float vertically on a water surface quickly arranged themselves in rouleaux.

The arrangement of spheres in two and three dimensions has been studied in connection with both liquid and crystalline structure. Bernal[8] used models of randomly packed uniform ball bearings to account for certain of the properties of liquids; a similar model was used by Scott[9] in his work on liquid structure.

Arrays of regular cubic or hexagonal close-packed spheres have been used as models of crystals. It became apparent, however, that such

regular structures could not explain certain observed properties such as plasticity, electrolytic conductivity and crystal strength[10]. Consequently it was necessary to postulate departures from the ideally perfect crystal in which uniform lattice arrays of atoms are interrupted by flaws. Darwin showed that lattice arrays of atoms in real crystals are not continuous throughout the crystals but are broken up into blocks by discontinuities at the block boundaries; such an ideally imperfect crystal was thought to be like a mosaic in which each part is perfect relative to its neighbours. Later investigators showed that though actual crystals are not perfect, Darwin's ideally imperfect crystal is not an accurate representation of crystals existing in nature. The present understanding of the nature of the deviations from perfect crystals really began when Prandel and Dehlinger[11] independently suggested that the mechanical properties of crystals were related to the presence of linear imperfections in such crystals. Taylor[12] proposed that linear deviations from true periodicity now called dislocations, were responsible for the relative ease with which most crystals could be deformed by an externally applied stress. Soon thereafter Burgers[13] extended these notions, and within a few years many investigators firmly established the existence of dislocations in crystals. At about the same time Smekal pointed out that the explanation of properties such as electrolytic conductivity and diffusion required imperfections of a different kind. Although obviously dependent on the actual crystal structure, the motion of atoms through crystals cannot be explained without requiring an atom to leave its normal position in the structure. Such a displacement produces an imperfection on an atomic scale as distinct from dislocations which affect large groups of atoms collectively.

A model of crystal behaviour, first suggested by Marshall[14] and later extended by Bragg[15,16] is the bubble raft in which an assemblage of a large number of small air bubbles about 1 mm. or less in diameter float on the surface of a soap solution. They are held together by surface tension either in a single layer as shown in Figure 1b of Part II, or in a three dimensional mass. Such rafts show hexagonal and cubic close packing, as well as the propagation of dislocations and recrystallization of the assemblage after stirring.

SCOPE OF THE THESIS

In the present work, described in Part II, assemblies of rods, discs and spheres were used in the measurement of particle aggregation during vibration of the system. The following is an outline of the objectives of the study and the scope of the thesis:

1) To measure the number distribution and orientation of aggregates in monolayers of rods on a solid plate at various vibration frequencies, surface concentrations and particle axis ratios.

2) To conduct a similar study in vibrating monolayers of wooden rods and poised, vertically floating discs at a liquid surface with special reference to the formation and break-up of rouleaux.

3) To measure the number distribution and orientation of rouleaux in a 3-dimensional assembly of discs after vibrating the system.

4) To study the growth of a monolayer of hexagonally close-packed crystallites of spheres with time during vibration; as well as to demonstrate the formation of dislocations and their propagation in an originally perfect close-packed structure.

The experimentation and results obtained are described in Part II which forms the main body of the thesis. A general summary and suggestions for further work are given in Part III.

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PART II

THE FORMATION OF ARRAYS BY UNIFORM PARTICLES

ABSTRACT

The formation of organised structures in vibrating monolayers of spheres, rods and discs, either resting on a horizontal solid plane or floating at a liquid surface, has been studied.

The size and orientation distributions of aggregates within mosaics of cylindrical particles were measured over a range of surface concentration, axis ratio and degree of vibration. With spheres, the growth of close packed hexagonal crystallites with time was studied as well as the development of dislocation boundaries on vibrating an initially close packed assembly.

The size and orientation distributions of rouleaux of discs were measured in 3-dimensional assemblies subjected to vibration.

The phenomena considered are of interest in connection with particle aggregation in dispersions of particles of regular geometry, such as rouleaux formation of red blood cells.

INTRODUCTION

It is known that under certain conditions, randomly distributed assemblies of macroscopic uniform spheres, rods and discs can be ordered into structures variously called mosaics, lattices and crystallites. Examples of such structures, illustrated in Figure 1, are the mosaic patterns of floating pulpwood logs in a boom, the hexagonal close packed arrangement of crystallites in a raft of small air bubbles floating on the surface of a soap solution[1] and the formation of regular cylindrical or rouleaux chains of biconcave red blood cells in plasma[2]. A variety of forces are responsible for bringing about such order - the restraint at the periphery of the log boom, the capillary attraction in the bubble raft and short range attractive forces in the case of the red cells.

The bubble raft has been used[1] as a model for demonstrating the existence of structures supposed to exist in metal crystals where the atoms are held together by the binding force of the free electrons, and to simulate imperfections such as grain boundaries and line dislocations[3] which are important in considering plastic deformation of crystals[4].

The process of forming an ordered structure of rods has been analysed by Litwiniszyn[5] who showed that a randomly distributed set of rigid straight wire segments on a horizontal plate could be ordered into a mosaic pattern by vibrating the plate. Statistically, the ordering process was described by a distribution density function $g(\phi,t)$ where $g(\phi,t)\Delta\phi$ expressed the share of those elements of the set which at time t form an angle contained in the interval $|\phi,\phi+\Delta\phi|$ with a fixed direction. Perfect order results when all the elements of the set form the same angle ϕ. The

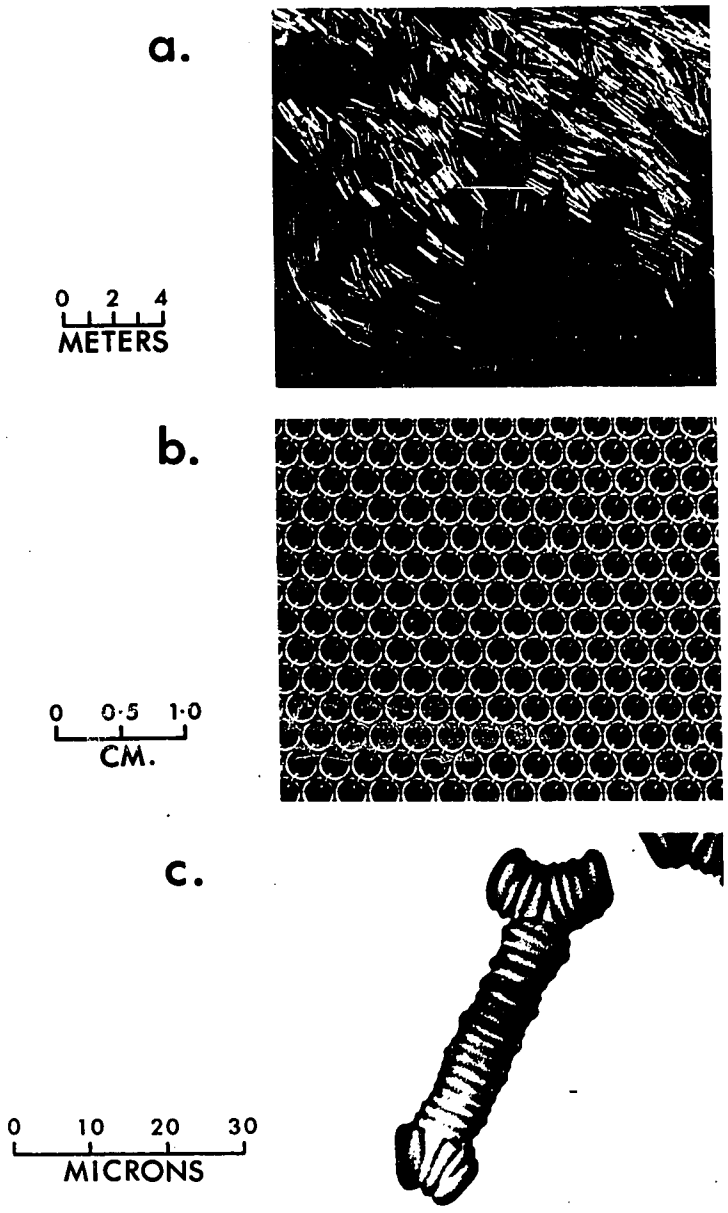


Figure 1. Examples of ordered arrays of particles having uniform geometry. (a) A towing raft of pulpwood logs on the river showing a mosaic pattern of aggregates much like those produced by vibrating a monolayer of steel rods as in Figure 2a. (b) A raft of air bubbles at the surface of a soap solution forming a two-dimensional hexagonal close-packed layer of spheres. (c) Two rouleaux of biconcave human red blood cells in plasma showing the characteristic face to face linear stacking of the discoid particles.

distribution in aggregate sizes within the mosaic was, however, not considered.

The formation of rouleaux in model systems was first shown by Norris[6,7] using, (i) poised vertically floating cork discs which ran together into cylindrical rolls where strewn onto a liquid, (ii) water wetted cork discs stirred into a water immiscible oil which on colliding formed piles or rouleaux.

To date, no quantitative measurements on the number and orientation distributions of structures in 2- and 3-dimensional assemblies of cylindrical particles subjected to an ordering restraint have been reported. The purpose of the present investigation was to study the number, size and orientation distribution of aggregates in vibrating monolayers of rods and discs resting on a horizontal solid plane or floating at a liquid surface as a function of the vibration frequency, particle size and surface concentration. The work also includes a study of vibrating monolayers of spheres and 3-dimentional assemblies of discs.

2. EXPERIMENTAL PART

(a) Monolayers

Assemblies of rods, discs and spheres resting on a flat horizontal plate bounded by a low wall, or floating on a liquid layer within a container set on the plate, were subjected to mechanical vibrations. The distribution of particles in aggregates and their orientation was studied as a function of time, plate size, particle surface concentration c, and axis ratio r_p = 2a/2b = axis of revolution/diametrical axis. Table 1 summarizes the range of experiments carried out.

(i) Cylindrical particles: Uniform roller bearings, diameter 2b = 0.315 cm, and the axis ratios from 3 to 10 were used to study the behaviour of rods on a vibrating surface. Wooden dowell pins of the same size were used in experiments on the surface of water.

Uniform circular discs, 2a = 0.32 cm, 2b = 1.27 cm. were prepared from low density (ρ = 0.92 gm.cm.$^{-3}$) polyethylene sheets. To ensure that the discs floated with the faces vertical, each was poised by inserting a small brass slug, 0.15 cm. diameter and 0.32 cm. long, on the edge. They were immersed in an approximately 6% aqueous $Cd(NO_3)_2$ solution whose density was adjusted so that all the discs floated either just below the air interface or with 1/10-2/10 of the diametrical axis above the surface, all particles assuming the same vertical position ($\rho = 0.998 \pm 0.014$).

(ii) Spheres: Steel ball bearings of 0.476 cm. diameter were used to study the behaviour of hexagonally close packed monolayers on a solid surface, the plate being painted black to accentuate the dislocations between aggregates formed during vibration.

TABLE 1

Summary of experiments on monolayers

Particle	Surface	Particle Axis Ratio a/b	2a cm.	2b cm.	Plate Diameter cm.	Surface Concentration c	N
Steel Roller Bearing	Solid	10	3.15	0.315	50.8	0.40 0.59 0.89	620 890 1370
Steel Roller Bearing	Solid	5.97	1.88	0.315	30.0-40.8	0.39 0.59 0.82	680 1040 1475
Steel Roller Bearing	Solid	2.85	0.897	0.315	26.0	0.37 0.60 0.74	590 910 1220
Wooden dowell pin	Water	5.97	1.88	0.315	30.0	0.80	770
Steel ball bearing	Solid	1.0	-	0.476	26.0	0.41-0.91	600-1500
Poised Polythene Disc	6% Cadmium nitrate solution	0.25	0.315	1.27	25.5	0.39 0.59 0.78	400 600 810

(b) Three dimensional assemblies

Polythene discs having the dimensions given above, but unweighted, were used for the 3-dimentional experiments.

(c) Procedure

(i) Monolayers. A weighed sample containing a known number of particles was placed in a heap on the solid plate or strewn at random on to the liquid surface. The plate, which was attached to the base of a Fisher Vibromatic Polisher[8], was then set into mechanical vibration at frequencies which could be varied from 10 to 100 sec^{-1}. The plate diameters could be varied from 26 to 41 cm. diameter. The vibration consisted of a reciprocating up, down and turning motion causing the particles to move in a counterclockwise direction around the plate and to arrange themselves into a monolayer consisting, in the case of cylinders, of a mosaic of aggregates; this is illustrated both for solid and liquid surfaces by the photographs in Figure 2.

The plate was photographed at various time intervals using a Nikon F 35 mm. camera, or continuously with a Bolex 16 mm. reflex cine camera at 16 frames/second, the optical axes of the cameras being normal to the plane (XY-plane, Figure 3) of the monolayer. The films were subsequently analysed frame by frame by projecting them onto a drafting table.

For the purposes of analysis, aggregates of cylinders in a mosaic pattern were defined as those structures in which the particles were all aligned at the same angle ϕ with respect to the Y-axis (Figure 3a) and touching, the discs face to face, the rods lengthwise. Aggregates, or crystallites of spheres were defined as those structures in which the particles assumed a hexagonal close-packed structure.

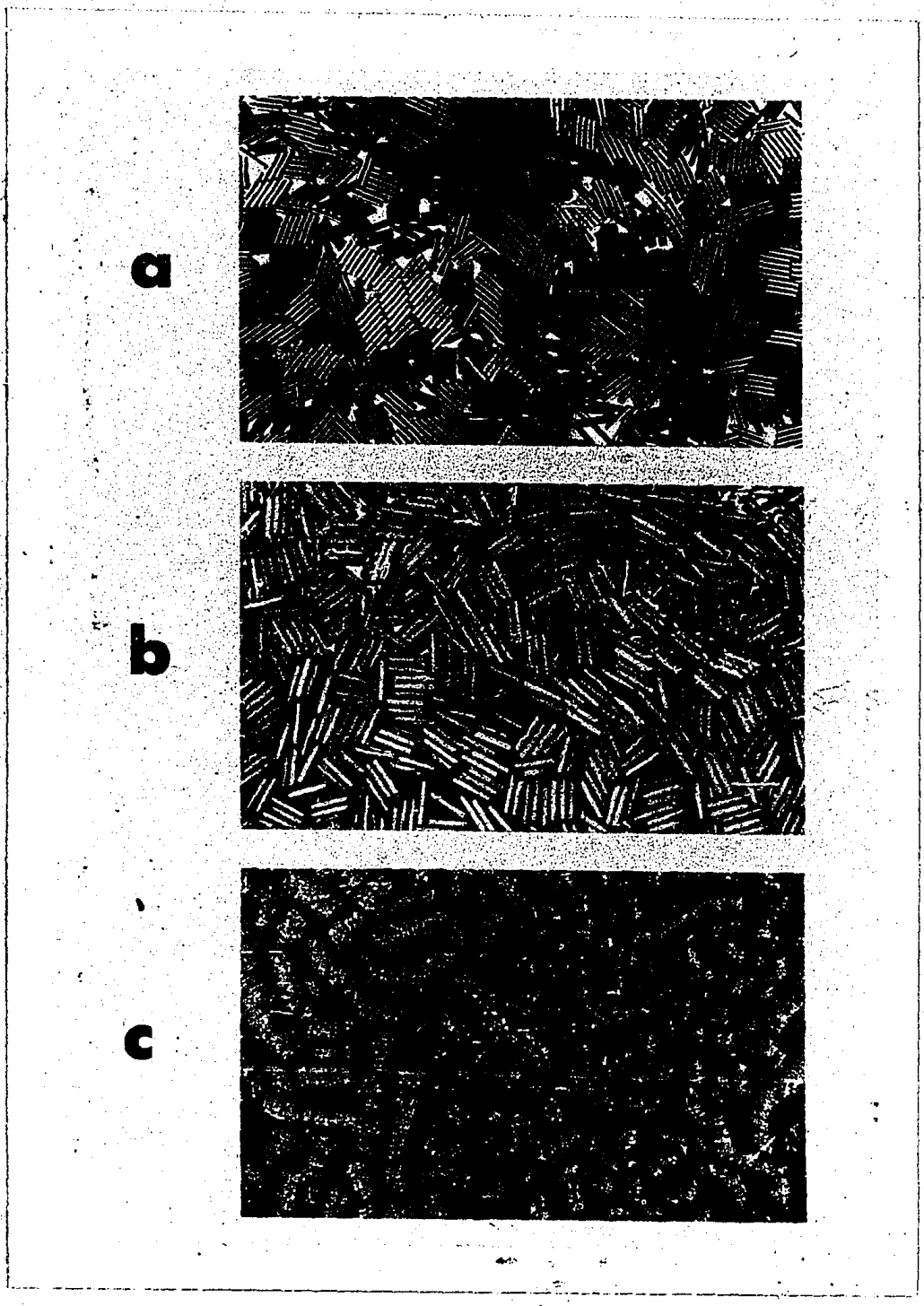
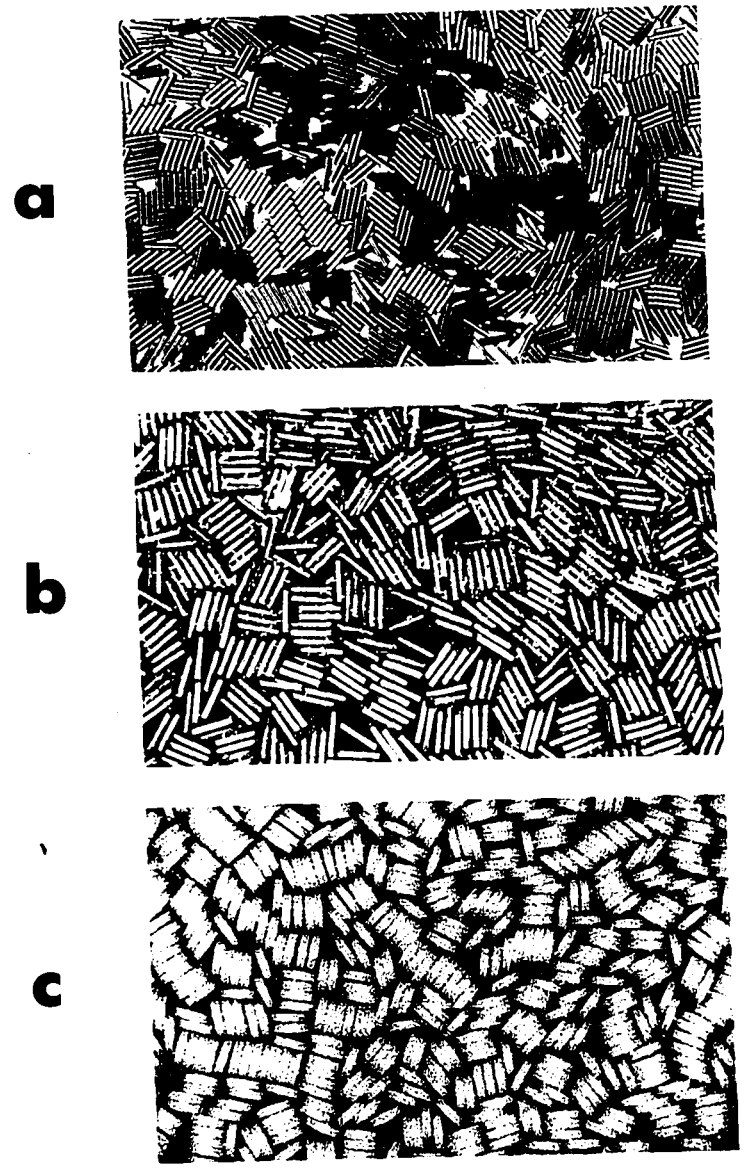


Figure 2. Formation of a mosaic of structures in a vibrating monolayer of cylinders. (a) Steel rods, $r_p = 5.97$, $c = 0.82$, resting on a solid surface. (b) Wooden rods, $r_p = 5.97$, $c = 0.78$ floating at a water surface. (c) Polyethylene discs $r_p = 0.25$, $c = 0.78$, poised to float vertically in a 6% aqueous cadmium nitrate solution. The aggregates here resemble rouleaux of red blood cells[2,7].



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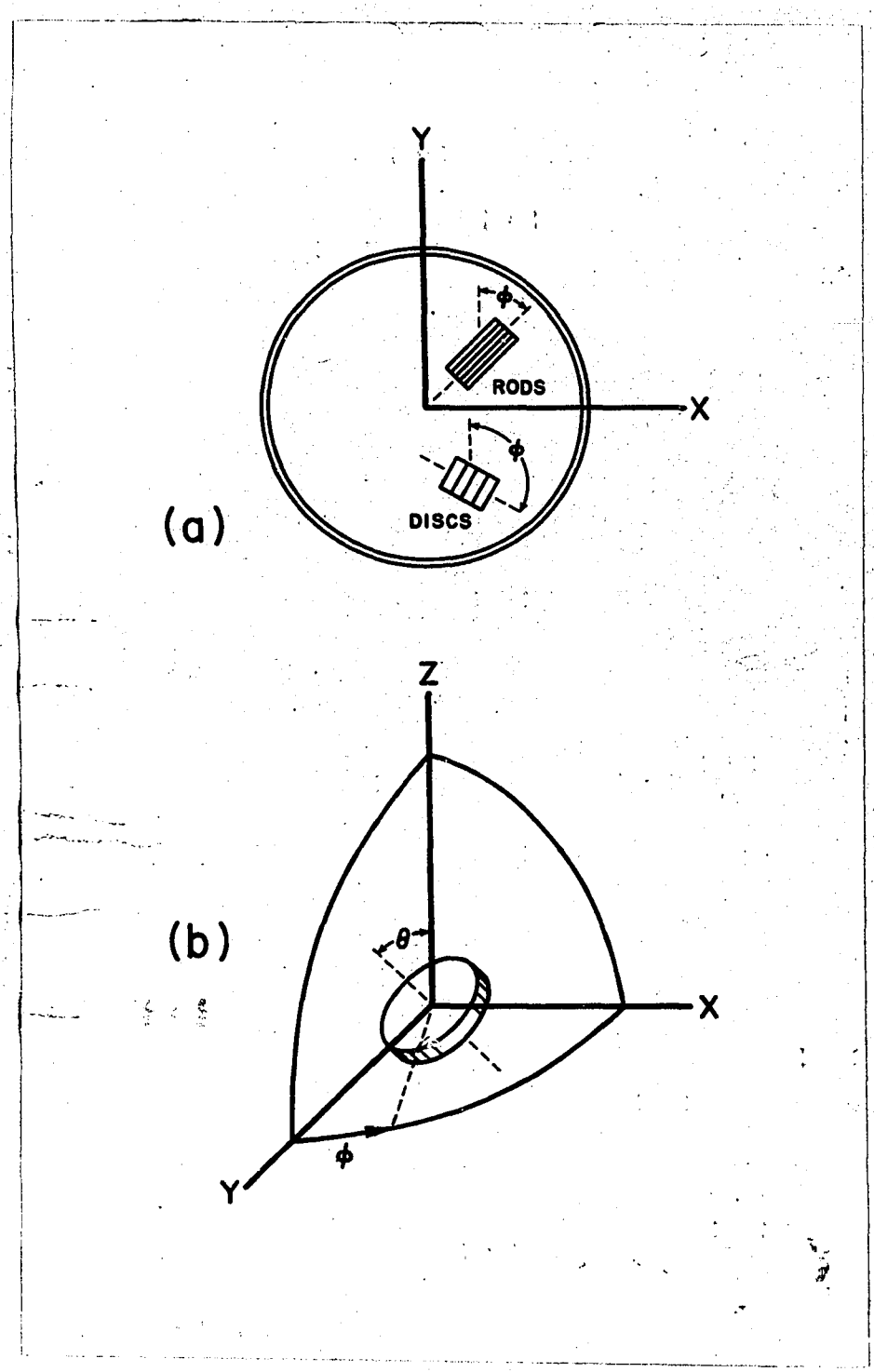


Figure 3. Coordinate system used to analyse orientations of aggregates of cylindrical particles. (a) Monolayers on solid plate or liquid surface showing the angle ϕ of aggregates of rods and discs with the Y-axis arbitrarily chosen at the beginning of an experiment. (b) Orientation of discs in multilayers showing the polar angles Θ and ϕ with respect to the Z-axis as the polar axis.

The surface concentration in each experiment was calculated from the total number of particles, the area of their median plane and the surface area of the vibrating plate. However, at the wall of the plate or container there was a noticeable tangential alignment of single cylinders and aggregates of 2 and 3 particles (see Figure 8 below). In these experiments the particle count was therefore limited to those cylinders whose centers were at a distance greater than a in the case of rods, and b in the case of discs, from the wall. The total particle count was never less than 600. For the spheres, a central rectangular domain on the plate containing ca. 1500 particles when hexagonally close packed was used to analyse the particle distributions.

(ii) 3-dimensional assemblies. The polythene discs were allowed to accumulate at random in a glass beaker, 13.2 cm. diameter and 17.0 cm. height up to a level of approximately 15 cm. The beaker was securely clamped with its base on the plate of the vibrometer and then subjected to vibration. The particles were shaken down into rouleaux-like structures which were fixed in situ by pouring in an epoxy-type resin as monomer (Waldex 1202, Waldor Enterprise Ltd.) to which a catalyst had been added 15 secs. previously, resulting in a very rapid polymerization so that after 1 minute at room temperature the resin had the consistency of tar. To avoid disturbing the discs a thick circular plate fitting tightly into the beaker was placed over the particles and the resin poured in through a centrally located hole. No obvious disturbances among the discs could be observed during this operation.

The cylindrical cast of the beaker contents was isolated by breaking the glass and then cut along the median, XY-plane into five 1"

thick discs using a band saw. These pieces were further cut into strips (see Appendix II) along an axis, previously chosen to be the Y-axis.

The number and orientation distribution of discs and rouleaux was carried out in each strip. The ϕ-orientation of the axis of revolution of those particles close to the surface of the cut section was obtained from measurements of the angle of the edge of the disc with respect to the Y-axis ($= \phi \pm \pi/2$). The θ-orientation of the axis of revolution with respect to the Z-axis (Figure 3b) was obtained from the measured thickness $2a'$ in the XY-plane and the relation

$$\sin\theta = a'/a ,$$

or from the angle of the disc edge with the Z-axis ($\pi/2 - \theta$) for those particles visible on the cut edge in the YZ-plane. A number of discs and aggregates comprising ca. 35% of all discs were situated in the interior of the strips and for these the orientations could not be estimated.

3. RESULTS

(a) General

Upon vibrating the assembly of rods on a solid surface, the mosaic pattern of aggregates shown in Figures 1a and 2a was formed very quickly. The individual structures could be seen continually shifting their Ø-orientation and to break up and reform elsewhere. Subsequent analysis showed that a condition of dynamic equilibrium was reached within 1 minute at a vibration frequency of 100 sec^{-1} and in 2 minutes at 10 sec^{-1}. A similar pattern of behaviour was observed with the wooden rods on the surface of water (Figure 2b), although the time required to reach equilibrium was somewhat longer.

In the case of the discs floating beneath the surface in cadmium nitrate solution the situation was quite different. When the particles were stirred into the container and brought into collision by the velocity gradients in the suspending solution, they tended to approach each other in a face to face orientation and to form rouleaux[6,7] (Figure 2c). The aggregates thus formed at first increased in length very quickly and then more slowly as the solution appeared to have come to rest. The effect of vibrating the plate in this case was to break up the long chain rouleaux into smaller aggregates and many single discs. As with the rods, a dynamic equilibrium was reached after a certain time, depending on the vibration frequency. Upon arresting vibration, longer rouleaux rapidly reformed and after the liquid had come to rest slowly increased further in length. The same behaviour was observed when the discs were only 80% submerged. The various stages of the above described process are illustrated by the photographs in Figure 4.

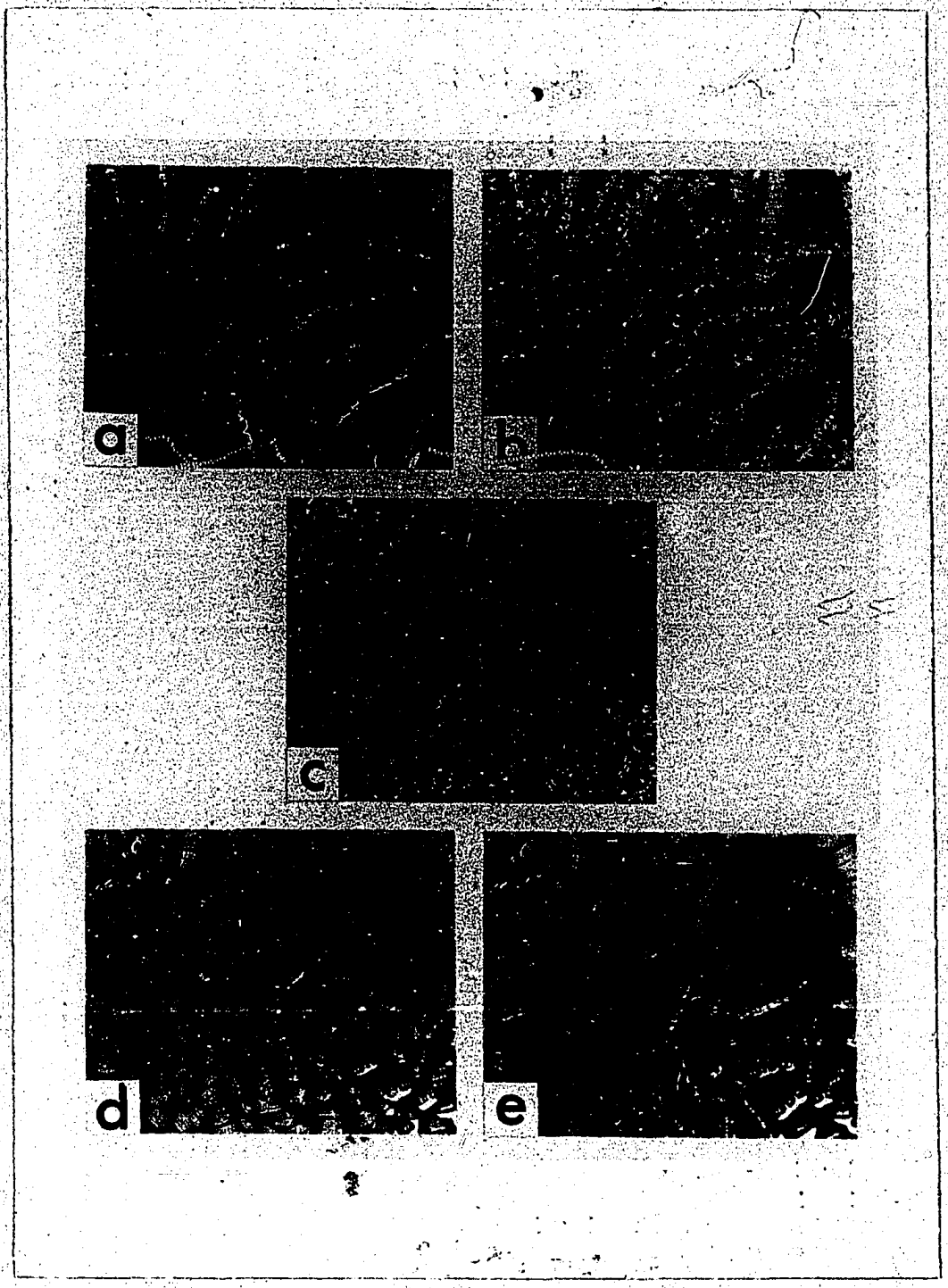
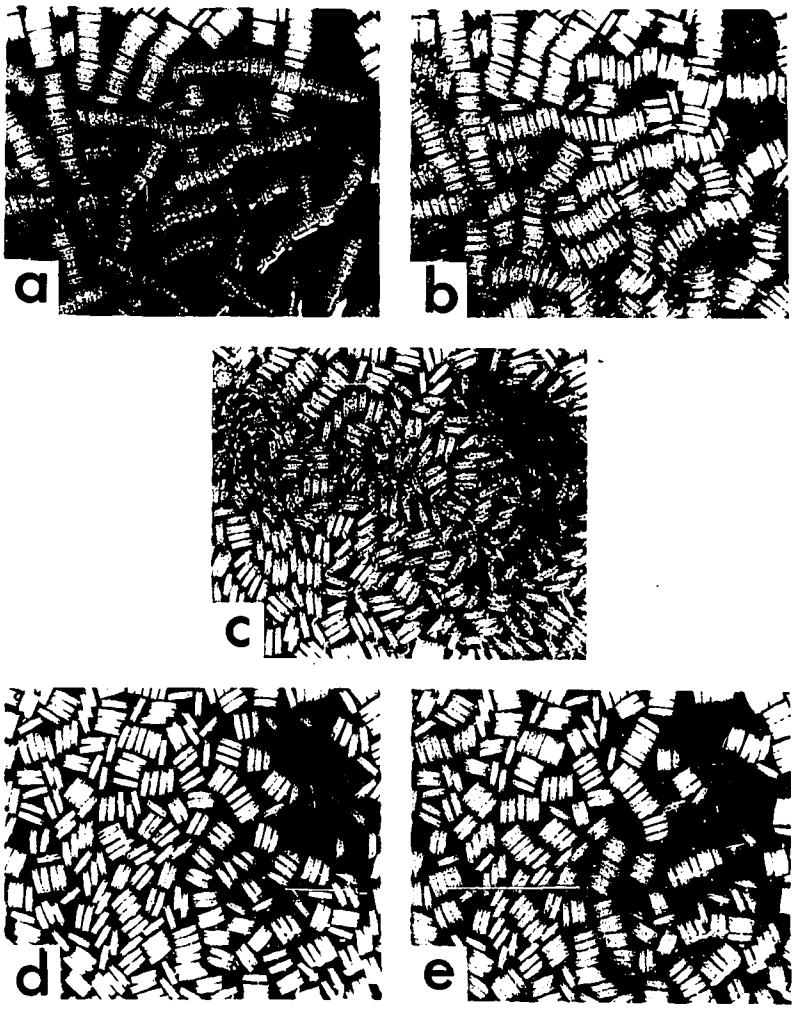


Figure 4. Rouleaux of polythene discs, $r_p = 0.25$, $c = 0.78$, as in Figure 2c, initially at rest (a), then 1 second (b) and 25 seconds (c) after vibration commenced showing break up of aggregates. (d) and (e) show the assembly 1 second and 60 seconds after arresting the vibration with the reformation of larger aggregates.



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(b) Monolayers of rods on solid surfaces

The results of the analysis of the numbers, sizes and orientation of aggregates of steel rods were plotted in terms of the differential and integral distribution functions defined as follows:

Let a_1, a_2, a_3 ..... a_n be the numbers of aggregates having sizes 1, 2, 3 ..... n containing a_1, $2a_2$, $3a_3$ ..... na_n particles at time t. Let the total number of aggregates $\sum a_n = A$, and the total number of particles $\sum na_n = N$.

We define two number distribution functions.

(i) The distribution density function f(n,t) which expresses the fraction of those aggregates of the set which at a given time t contain n particles.

(ii) The distribution density function g(n,t) which expresses the fraction of particles in the set which at a given time t are contained in aggregates of number n.

In comparing the results obtained at different axis ratios and surface concentrations the system was allowed to come to equilibrium. The time independent functions are then

$$F(n) = \sum_{n=1}^{n} f(n)$$

where F(n) and f(n) are the respective aggregate number integral and differential distributions; and similarly for G(n) and g(n), the particle number distribution functions.

Moreover, since

$$f(n) = \frac{a_n}{A} \quad \text{and} \quad g(n) = \frac{na_n}{N},$$

$$f(n) = \frac{Ng(n)}{nA}.$$

Similarly we define the orientation distribution functions $f(\phi,t)$ and $g(\phi,t)$ which give the respective share of those aggregates and particles of the set which at the moment t form with a fixed coordinate an angle ϕ contained in the interval $|\phi,\phi + d\phi|$. Unlike the number distribution functions, they are assumed to be continuous and are periodic in ϕ with the period π[5]. Thus

$$f(\phi,t) = f(\phi \pm m\pi,t) \qquad m = 1,2, \ldots ,$$

and

$$F(\phi,t) = \int_0^{\phi} f(\phi,t)d\phi ,$$

or at equilibrium

$$F(\phi) = \int_0^{\phi} f(\phi)d\phi, \quad \text{with}$$

$$\int_0^{\pi} f(\phi)d\phi = 1 .$$

Typical number distribution functions at a vibration frequency of 10 sec^{-1} are shown in Figure 5 in which are plotted the arithmetic mean values and root mean square deviations in f(n), g(n) and F(n), G(n) from 15 experiments with steel roller bearings $r_p = 5.97$ and $c = 0.82$ at equilibrium. Whereas the differential particle distribution function at first increases with n, reaching a peak at $n = 4$, the differential aggregate distribution function decreases continuously with increasing n to the largest aggregate size found, $n = 17$. As is evident from Figure 5a and Table 2 which lists values of the functions obtained in a typical experiment, the R.M.S. deviations in f(n) and g(n) are large compared to those in F(n) and G(n), the latter were therefore used in comparing results obtained

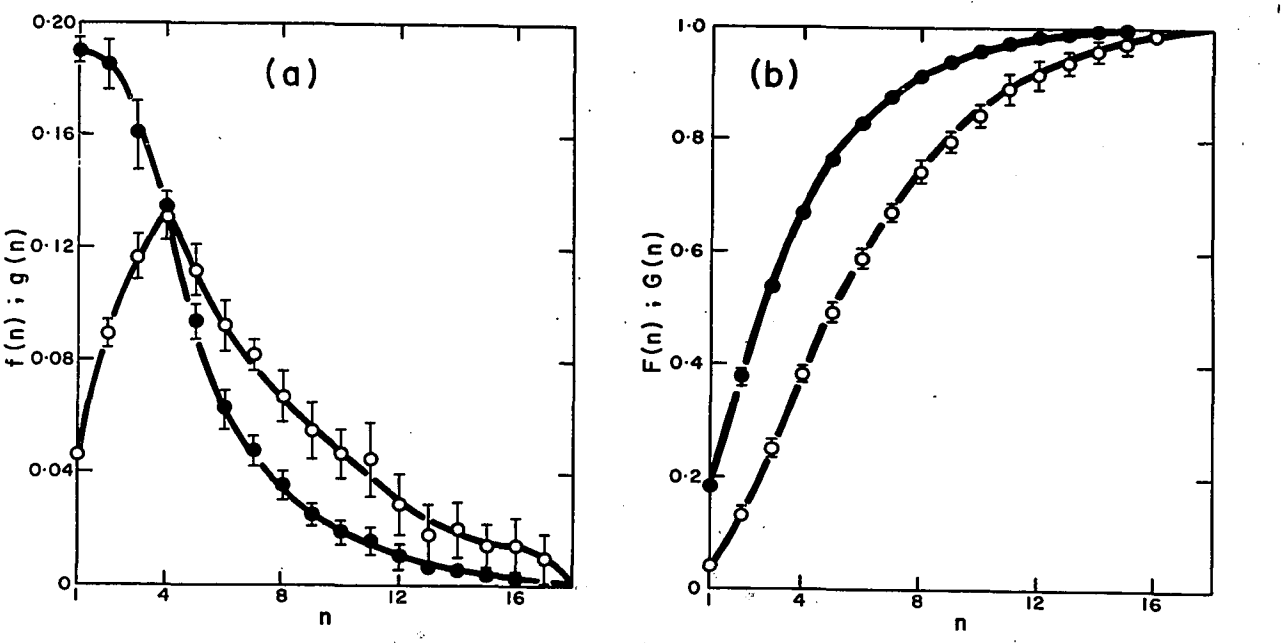

Figure 5. Differential (a) and integral (b) number distribution functions obtained at equilibrium in a monolayer of rods at a solid surface, $r_p = 5.97$, $c = 0.82$ vibrating at 10 sec^{-1} shown in Figure 2a. The values plotted are the mean of 15 experiments; the standard deviations are shown by the bars. The open and closed circles refer to the particle and aggregate distributions respectively.

TABLE 2

Equilibrium number distribution of particles and aggregates of cylinders, r_p=5.97, c=0.82, 10 vibrations/second

n	a_n	na_n	f(n)	g(n)	F(n)	G(n)
1	49	49	0.189±0.005[a]	0.046±0.002[a]	0.189±0.005[a]	0.046±0.002[a]
2	46	92	0.177±0.009	0.026±0.005	0.366±0.011	0.132±0.006
3	43	129	0.165±0.012	0.121±0.009	0.531±0.006	0.253±0.008
4	35	140	0.135±0.009	0.131±0.010	0.666±0.007	0.384±0.011
5	25	125	0.096±0.006	0.117±0.009	0.762±0.007	0.501±0.012
6	16	96	0.062±0.008	0.090±0.011	0.824±0.007	0.591±0.016
7	13	91	0.050±0.005	0.085±0.006	0.874±0.008	0.676±0.016
8	9	72	0.035±0.005	0.067±0.011	0.909±0.007	0.743±0.018
9	6	54	0.023±0.004	0.050±0.010	0.932±0.008	0.793±0.017
10	4	40	0.015±0.004	0.037±0.009	0.947±0.006	0.830±0.020
11	4	44	0.015±0.005	0.041±0.013	0.962±0.006	0.871±0.028
12	3	36	0.012±0.005	0.034±0.011	0.974±0.006	0.905±0.023
13	2	26	0.008±0.004	0.024±0.011	0.982±0.005	0.929±0.020
14	2	28	0.008±0.003	0.026±0.010	0.990±0.004	0.955±0.017
15	1	15	0.004±0.003	0.014±0.008	0.994±0.004	0.969±0.014
16	1	16	0.004±0.002	0.015±0.010	0.998±0.004	0.984±0.009
17	1	17	0.004±0.002	0.016±0.009	1.000	1.000
18	0	0	0	0	1.000	1.000

A = 260 N = 1070

[a] R.M.S. Standard deviations calculated from 15 experiments.

at different r_p and c. The particle and aggregate number distributions were unaffected by changes in the diameter of the vibrating plate (and hence N).

As illustrated in Figure 6a, the change in particle distribution at a vibration frequency 10 sec^{-1} after the first minute is quite small showing that equilibrium is rapidly established. This was found to be true whether the particles were initially heaped onto the plate or all aligned in parallel rows with $\phi = 0$, and the final equilibrium distributions were identical in both cases. As the vibration frequency was increased from 10 to 80 sec^{-1} the larger aggregates broke up as shown in Figure 6b with the most probable particle density shifting from $n = 4$ to $n = 3$.

At each axis ratio, the distributions were shifted toward larger n as the surface concentration was increased from 0.39 to 0.82 (Figures 7 and 8), although as shown in Table 3, the peak in g(n) still occurred at $n = 4$. At a given c, however, the distribution curves were not significantly affected by changing r_p (Table 4).

The distribution of particles with respect to the angle ϕ, $g(\phi)$, was not significantly different from random and independent of concentration and axis ratio. The scatter in the measured values of $G(\phi + 20) - G(\phi)$, the mean of which are listed in Table 5, is quite large (S.D.$\sim \pm 20\%$), and there is a tendency for the particle density to be higher in the region $\phi = 40° - 80°$.

(c) Monolayers of rods and discs on liquid surfaces

Table 3 shows the mean equilibrium distribution G(n) for rods on a liquid surface to be very close to that obtained for rods on the solid surface when the frequency, r_p and c are the same.

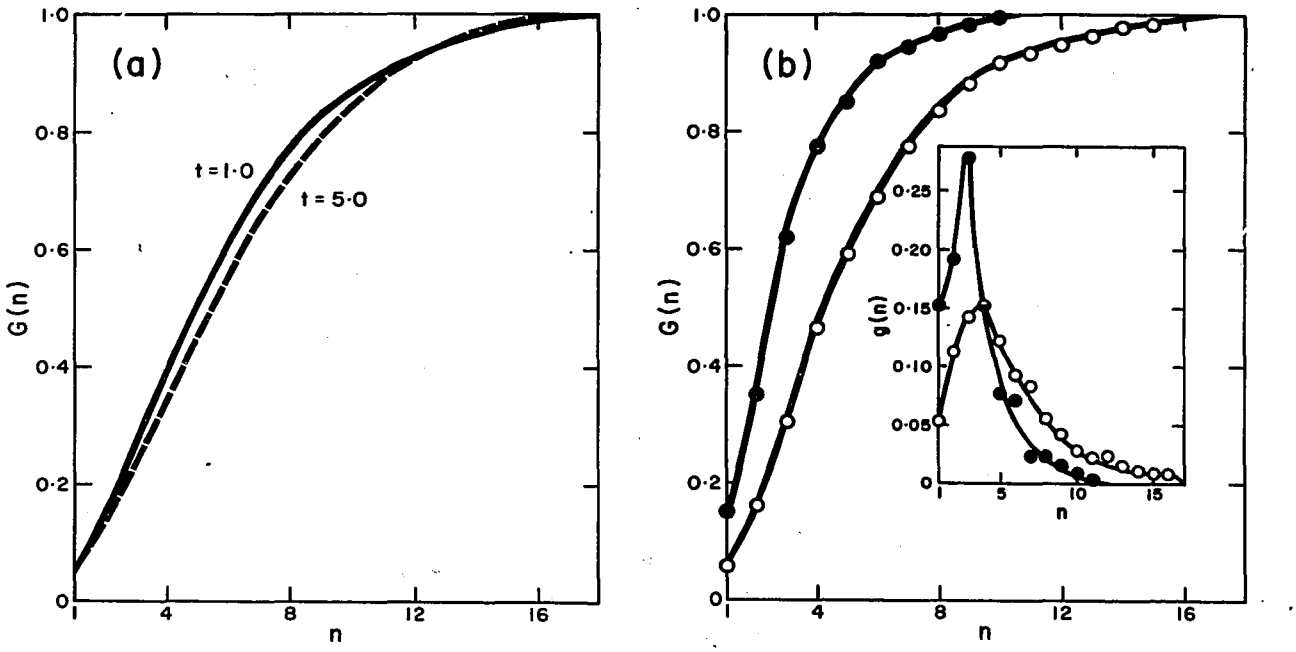

Figure 6. (a) Change in particle number distribution with time (minutes) for steel rods $r_p = 5.97$, $c = 0.82$ on a solid surface at 10 vibrations/second.
(b) The effect of increasing the vibration frequency from 10 sec^{-1} (open circles) to 80 sec^{-1} (closed circles) on the integral and differential (inset) particle number distribution at equilibrium.

TABLE 3

Integral particle number distributions for rods, $r_p = 5.97$ as a function of concentration, 10 vibrations/second

n	G(n) solid surface c = 0.39	c = 0.59	c = 0.82	G(n) water surface c = 0.80
1	0.064	0.055	0.046	0.048
2	0.179	0.154	0.132	0.141
3	0.323	0.278	0.253	0.250
4	0.477	0.418	0.385	0.382
5	0.611	0.527	0.500	0.499
6	0.713	0.634	0.592	0.586
8	0.857	0.795	0.742	0.746
10	0.929	0.887	0.840	0.858
14	1.00	0.974	0.962	0.967
17	1.00	1.00	1.00	1.00
N =	685	1055	1475	760

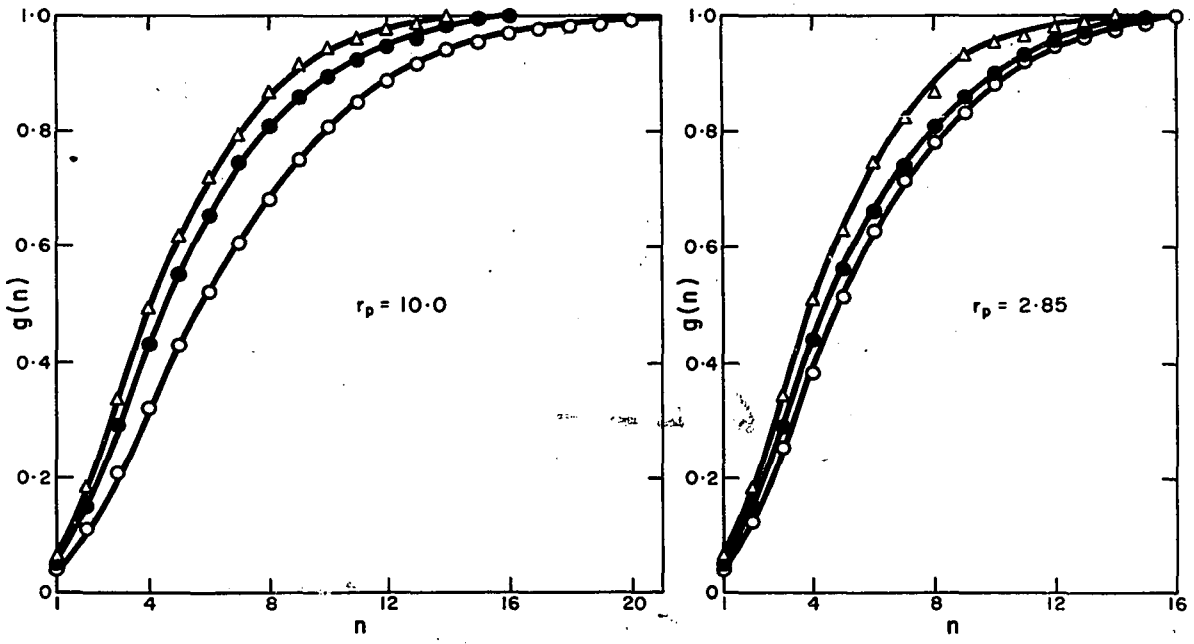

Figure 7. The effect of increasing surface concentration on the integral distribution of particles in aggregates at $r_p = 10.0$ and $r_p = 2.85$. The curves, in order of increasing aggregation (from top to bottom) refer to c = 0.40, 0.59 and 0.89 at $r_p = 10.0$, and c = 0.37, 0.60 and 0.74 for $r_p = 2.85$.

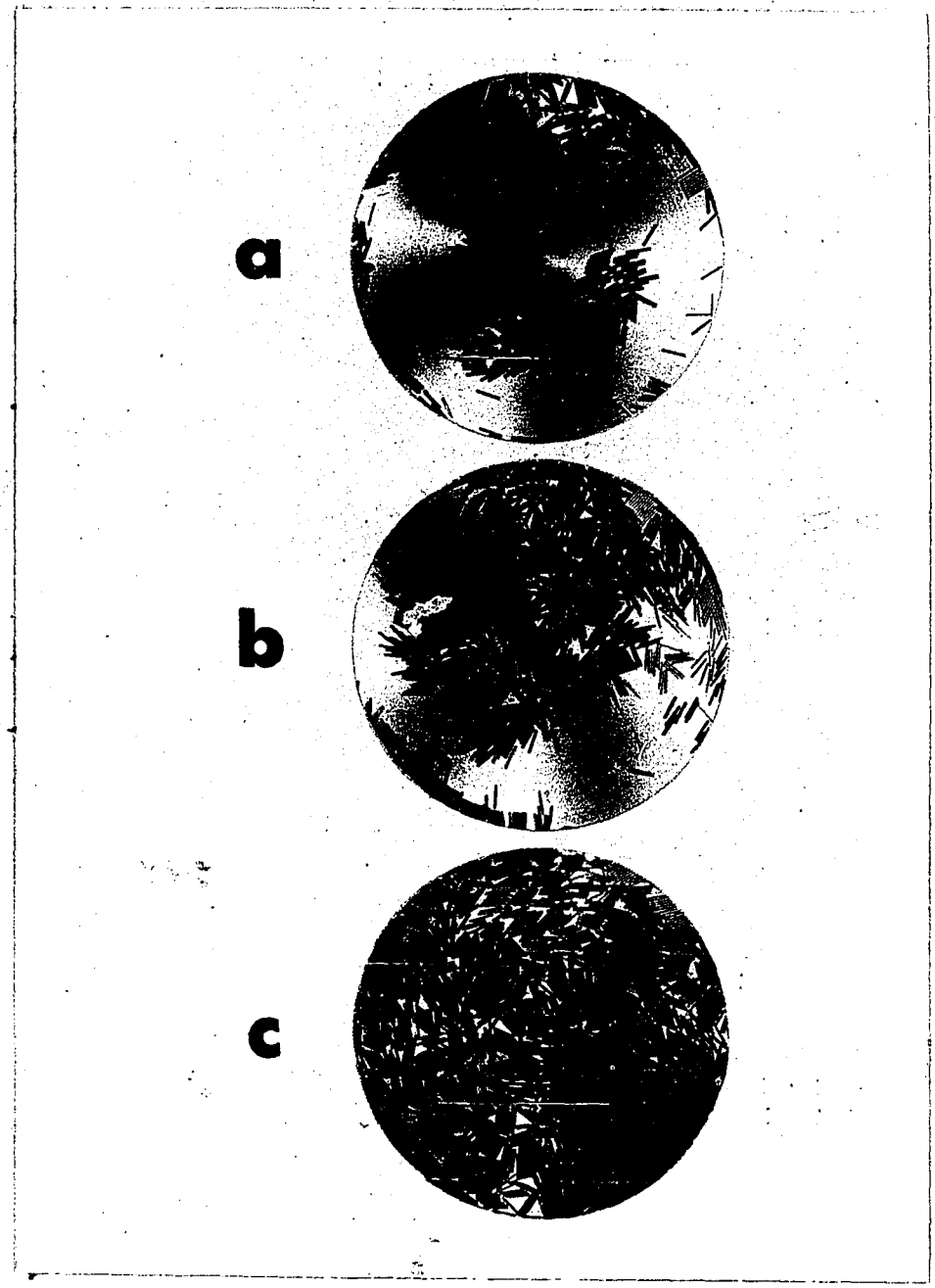

Figure 8. Photographs of aggregates of steel rods $r_p = 10.0$ at increasing mean surface concentrations $c = 0.40$ (a), 0.59 (b), and 0.89 (c) corresponding to the curves given in Figure 7.

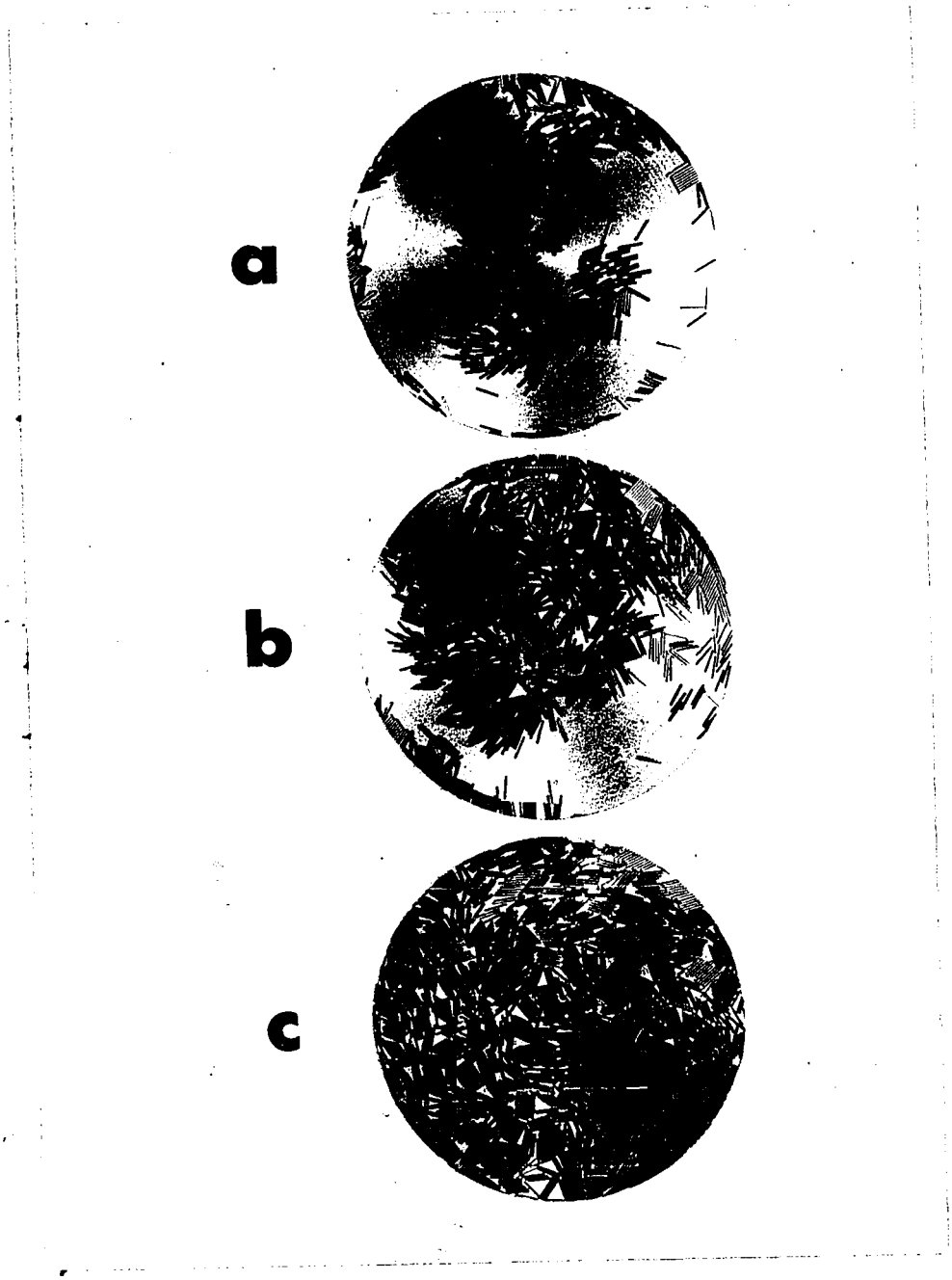

Figure 8. Photographs of aggregates of steel rods $r_p = 10.0$ at increasing mean surface concentrations $c = 0.40$ (a), 0.59 (b), and 0.89 (c) corresponding to the curves given in Figure 7.

TABLE 4

Number distribution of particles and aggregates of rods, c = 0.60, as function of axis ratio, 10 vibrations/second

n	F(n) r_p= 2.85	F(n) r_p= 5.97	F(n) r_p= 10.0	G(n) r_p= 2.85	G(n) r_p= 5.97	G(n) r_p= 10.0
1	0.188	0.210	0.219	0.049	0.055	0.059
2	0.379	0.401	0.398	0.151	0.154	0.155
3	0.550	0.555	0.568	0.286	0.278	0.291
4	0.696	0.687	0.701	0.438	0.418	0.433
5	0.792	0.772	0.793	0.562	0.527	0.555
6	0.854	0.842	0.855	0.660	0.634	0.655
7	0.896	0.890	0.904	0.739	0.722	0.741
8	0.929	0.926	0.929	0.809	0.795	0.806
9	0.950	0.949	0.950	0.861	0.847	0.857
10	0.967	0.963	0.962	0.893	0.887	0.890
11	0.979	0.974	0.975	0.933	0.921	0.922
12	0.988	0.982	0.983	0.954	0.944	0.954
15	1.00	0.993	0.995	1.00	0.974	0.989
17	1.00	1.00	1.00	1.00	1.00	1.00
	A=240	272	241	N=925	1055	915

TABLE 5

Ø-Orientation distributions of cylincers on solid and liquid surfaces

Ø degrees	$G(\emptyset + 20) - G(\emptyset)$ [a] $r_p = 0.25$, $c = 0.78$	$r_p = 2.85$, $c = 0.74$	$r_p = 5.97$, $c = 0.82$
0	0.110	0.095	0.113
20	0.106	0.103	0.111
40	0.130	0.112	0.137
60	0.107	0.131	0.123
80	0.097	0.108	0.102
100	0.115	0.119	0.109
120	0.118	0.114	0.101
140	0.114	0.105	0.093
160	0.102	0.110	0.109
N	810	1220	1510
S.D. [b]	$\pm$0.025	$\pm$0.020	$\pm$0.022

[a] Average of 5 experiments

[b] Standard deviation from random distribution $G(\emptyset + 20) - G(\emptyset) = 0.111$

Measurements of the number distributions were made with the weighted polythene discs in $Cd(NO_3)_2$ solution at various c, before, during and after vibrating the system. The state of aggregation after initially stirring in the discs and waiting 10 minutes is shown by curves 1 and 4 of Figure 9a at c = 0.59 and 0.39 respectively. At the lower concentration there was a much higher proportion of aggregates having $n > 4$; 40% of particles being in aggregates having $n > 8$, these comprising 15% of all aggregates; at c = 0.59 the respective proportions are 16% and 4%. The time course of break up and reformation during and after vibrating at 60 sec^{-1} for 39 seconds at c = 0.59 is illustrated in Figure 9b for g(1) and g(6) and the values of f(n) are listed in Table 6. It is evident that there is first a rapid increase in single discs and rouleaux, $n \leq 4$, at the expense of larger rouleaux (see also Figure 4), the maximum g(n) being at n = 3. As expected the degree of break up increased with increasing frequency. After a slight reaggregation the system appeared to reach equilibrium after approx. 27 seconds. On stopping vibration, longer rouleaux reformed rapidly. The initial distribution, in which the maximum g(n) is at n = 4, was reached after about 10 minutes.

At equilibrium during vibration the ϕ-orientation distribution was close to random (Table 5).

(d) Monolayers of spheres on solid surfaces

Two types of experiment were carried out:

(i) The spheres were randomly distributed over the horizontal plate at a surface concentration of 65%. The assembly consisted of a number of aggregates in which the spheres were hexagonally close packed. These may be called two-dimensional crystallites and, as illustrated in

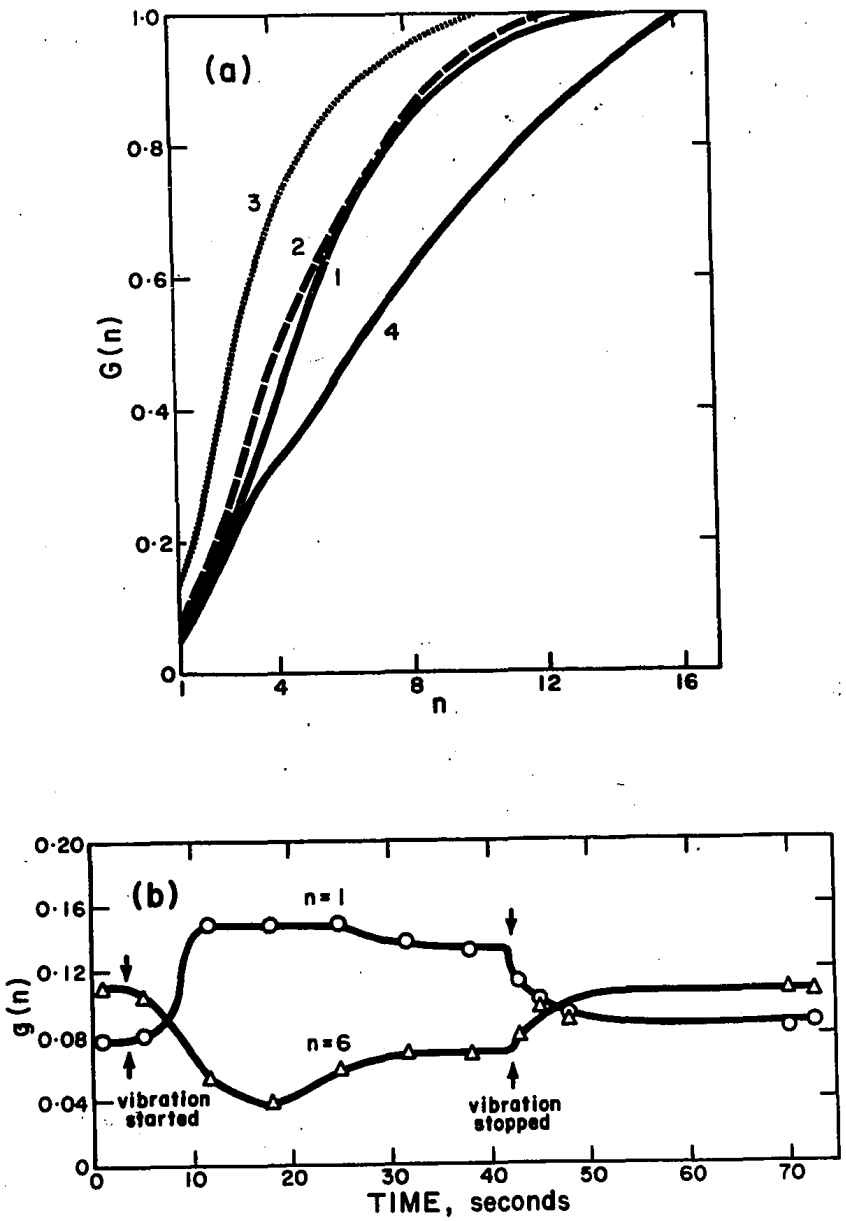

Figure 9. (a) Integral particle number distributions of discs in cadmium nitrate solution, c = 0.59, before (1), 1 second, after beginning vibration (2), and at equilibrium 25 seconds later (3). Curve 4 is the initial distribution at c = 0.39 showing higher proportion of large rouleaux.

(b) Time course of break up during and after vibration for the same system as in (a) at c = 0.59. The values of the differential distribution functions g(1) and g(6) are plotted against time. The vibrations bring about an increased formation of single discs at the expense of rouleaux of n > 4.

TABLE 6

Number distribution of rouleaux of discs floating in cadmium nitrate solution, c = 0.59, 60 vibrations/second

n	f(n) At rest t = -3.1 sec.	f(n) vibrating[a] t = 7.6 sec.	f(n) vibrating[a] t = 25.0 sec.[b]	f(n) At rest t = 69.3 sec.
1	0.276	0.364	0.345	0.258
2	0.171	0.256	0.260	0.274
3	0.129	0.176	0.176	0.159
4	0.129	0.104	0.096	0.108
5	0.095	0.056	0.059	0.089
6	0.065	0.026	0.029	0.054
7	0.041	0.011	0.011	0.020
10	0.018	0.004	0.004	0.005
A =	170	269	242	200
F(10)=	0.948	0.990	0.983	0.970

[a] Vibrations at 60 sec^{-1} commenced at t = 0, ceased at t = 39 sec.

[b] System at equilibrium

Figure 10a were separated from each other by non crystalline or "grain" boundaries two to three particle diameters wide. The boundaries[3] contained numerous "fault" lines where the spheres were not in close packing, as well as singlet spheres and vacant sites.

When the system was subjected to vibration at a low frequency of 10 sec^{-1}, recrystallization took place[1], i.e. the crystallites grew in area at the expense of the grain boundaries from which singlet spheres and small aggregates become attached in hexagonal close packing. (Figures 10b and c). The process of ordering continued by depleting the peripheral area of the plate of particles until the great majority of spheres had collected in a few close packed crystallites of over 100 particles each.

The particle distributions were followed in a central rectangular domain 19 cm. x 13.5 cm. and the results of a typical experiment are given in Table 7. It can be seen that whereas at zero time, c = 0.41, there were 86 crystallites, the largest of which contained only 32 spheres; after 2 minutes there were but 10 crystallites of which the largest contained 647 spheres and the concentration in the domain was 0.81.

(ii) The spheres were arranged in a hexagonal close-packed monolayer, i.e. one single crystallite as illustrated in Figure 11a with the surface concentration (area of circle/area of hexagon circumscribing sphere[9]) = 0.907. When the system was subjected to vibration at a frequency 10 sec^{-1}, linear deviations[3] or dislocations from the periodic arrangements were observed (Figure 11b). These came about by displacements of spheres from their regular position in the structure. Cinefilms of the process showed that it was similar to that postulated by Taylor[4] and observed by Bragg[1] in the spherical bubble raft. Thus, it began with the

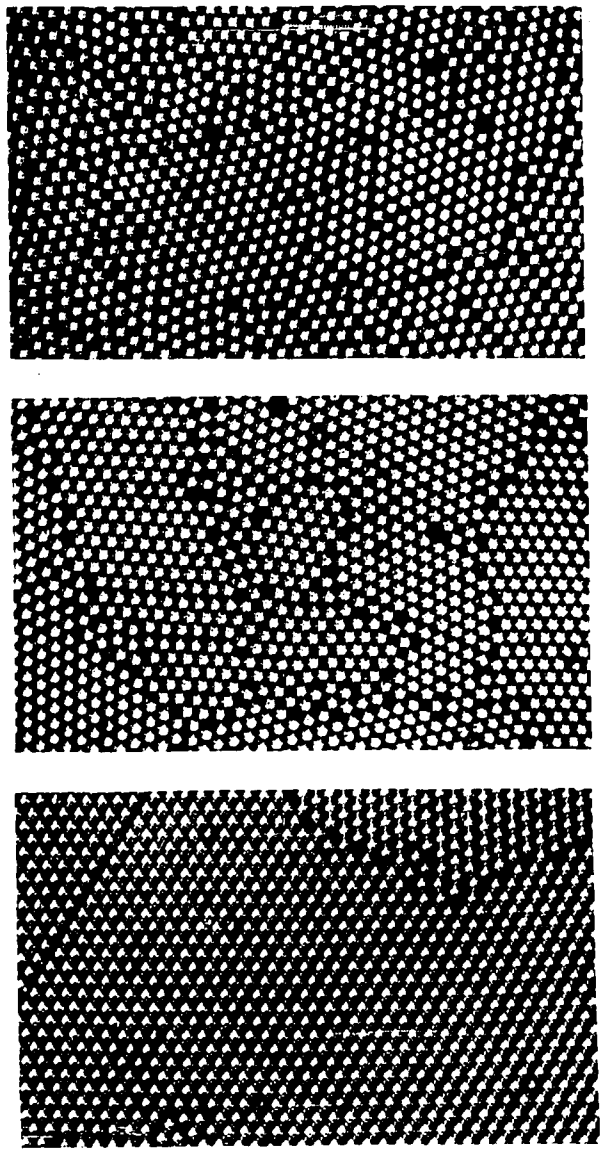

Figure 10. The effect of vibrations on the size of crystallites of hexagonally close-packed spheres (ball bearings 0.48 cm. diameter) resting on a solid surface. The photographs show a central domain of the plate. (a) t = 0; small crystallites separated by grain boundaries can be seen. (b) t = 1 min. (c) t = 2 min. after vibration started: the crystallites increase in size at the expense of the grain boundaries and the surface concentration has grown from 0.41 in (a) to 0.81 in (c).

TABLE 7

Number distribution of spheres in crystallites during vibration

t = 0		t = 1 min.		t = 2 min.	
n	a_n	n	a_n	n	a_n
2	3	3	5	3	2
3	7	4	2	4	1
4	9	5	2	6	1
5	9	7	1	7	1
6	14	11	1	12	1
7	13	13	1	106	1
8	8	14	1	156	1
9	8	16	1	231	1
10	5	20	1	647	1
12	3	27	1		
13	1	39	1		
14	1	48	1		
15	1	63	1		
16	1	102	1		
22	1	121	1		
25	1	161	1		
32	1	172	1		
		189	1		
N = 642		N = 923		N = 1275	
A = 86		A = 24		A = 10	
c = 0.41		c = 0.71		c = 0.81	

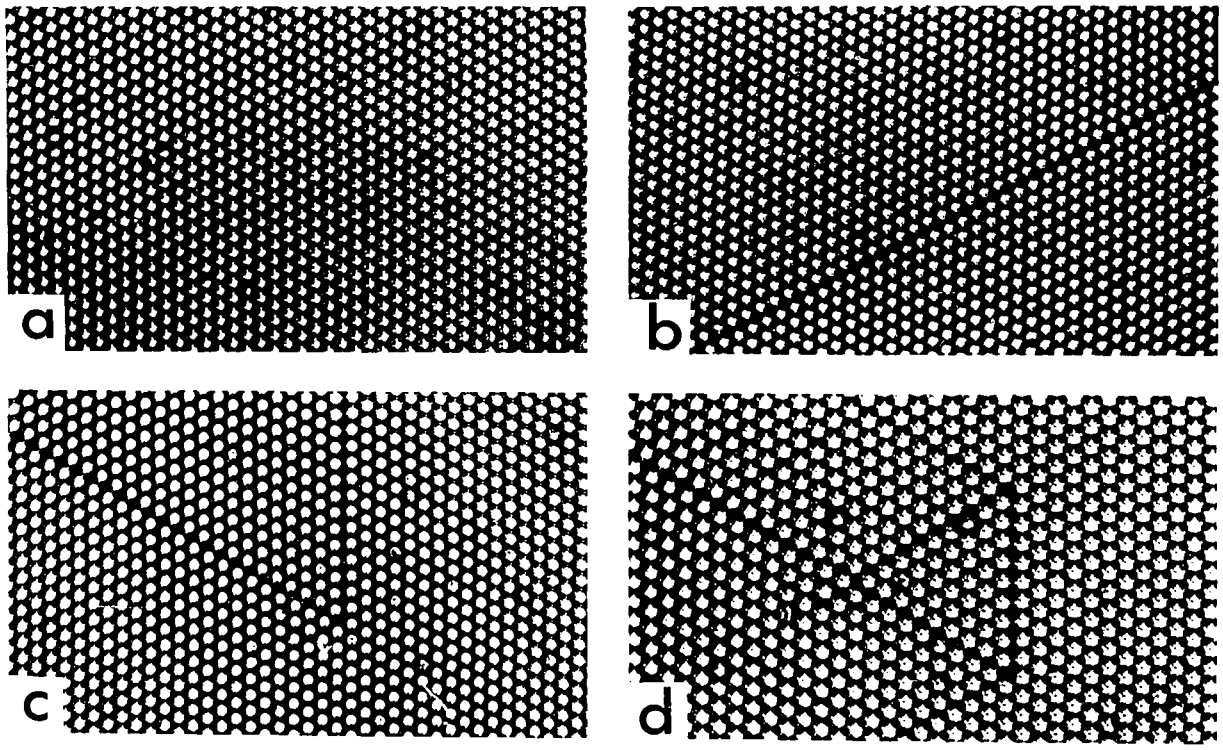

Figure 11. Linear dislocations arising from the vibration of a hexagonally close-packed monolayer of spheres. (a) Close-packed layer, c.f. Figure 1b. (b) Dislocations in adjacent rows. (c) Dislocations meeting at 60° to produce V-structure. (d) Formation of Δ-structure through meeting of 3 dislocations at 60°.

appearance of a point dislocation in which a sphere moved to the left or right by an amount equal to the distance between neighbours. The dislocation then moved from one side of the crystallite to the other, the end result being a slip by one particle diameter with the structures on either side of the line boundary remaining undisturbed (Figure 11b). Line dislocations were continually formed during vibration and often intersected each other at an angle of 60° to form V-type (Figure 11c) and equilateral triangle-type (Figure 11d) crystallites. However, their existence was temporary and they disappeared again behind the path of the advancing dislocations.

(e) 3-dimensional assemblies of discs

The number and orientation distributions of the discs found in the epoxy resin cast of the beaker after vibrating at 100 sec^{-1} for 12 hours are given in Table 8 and plotted in Figure 12.

The mean volume concentration of discs in the cast was calculated to be 0.47; this compares with the maximum possible concentration of 0.91 for discs in hexagonal close packed layers. In contrast to the aggregates found in monolayers of cylinders, the rouleaux-like structures in the multilayers were of small size, 90% of all particles being found in aggregates having $n \leqslant 5$. As with the monolayer experiments g(n) had a maximum (n = 3) whereas f(n) decreased with increasing n.

The Ø-orientation distribution showed a small deviation from randomness. The values for $G(\theta)$ given in Table 8 illustrate a marked orientation of discs with their faces close to the vertical ($\theta = 90°$), 31% of all particles having orientations $\theta > 70°$.

TABLE 8

Number and orientation distribution of discs,

$r_p = 0.25$, in 3-dimensional assembly

Aggregates			Orientation			
n	a_n	G(n)	ø degrees	G(ø+20)-G(ø)	θ degrees	G(θ+10)-G(θ)
1	135	0.126	0	0.132	0	0.054
2	125	0.360	20	0.127	10	0.051
3	88	0.606	40	0.112	20	0.061
4	53	0.805	60	0.084	30	0.112
5	22	0.907	80	0.082	40	0.141
6	9	0.958	100	0.128	50	0.158
7	5	0.990	120	0.111	60	0.111
8	-	0.990	140	0.122	70	0.097
9	-	0.990	160	0.102	80	0.216
10	-	1.00				
N = 1070			N = 660		N = 893	

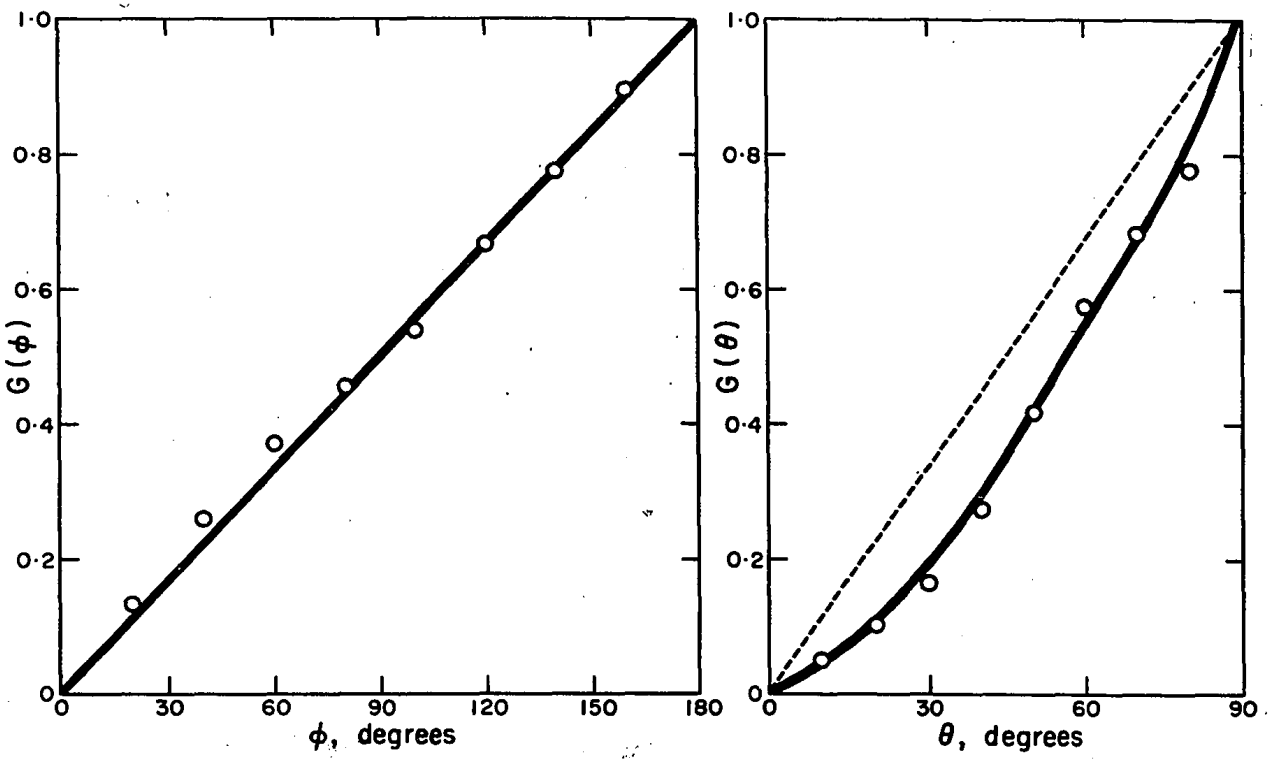

Figure 12. Ø- and Θ- integral orientation distribution functions in a 3 dimensional assembly of discs, $r_p = 0.25$, after vibrating the particles in a large beaker. The solid lines drawn are the best fit of the experimental points. The G(Θ) curve shows that a preferred orientation of particles with their faces close to the vertical ($\Theta = 90°$) has occurred; the dashed line corresponds to random distribution.

4. DISCUSSION

These qualitative experiments demonstrate that a two-dimensional assembly of particles in which the freedom of each member to move is limited by collisions with others, and in which rods (and discs) brought into relative motion can form mosaic structures and in the case of spheres, hexagonal close-packed crystallites.

The uniform size of the particles and their geometry must be regarded as cardinal features of the patterns of order. Thus it was found that at a given surface concentration and degree of vibration, the number distribution of aggregates of the steel rods was independent of their axis ratio.

It also appears that it is the shape and uniform size of the polythene discs and the biconcave red blood cells which allows them to form linear chains or rouleaux[7]. However, the immediate cause of aggregation is different in the two systems. With red blood cells it is probably due to short range attractive forces dependent on the presence of the protein fibrinogen in the plasma, for the cells when washed free of plasma and dispersed in physiological saline, while still biconcave in shape, do not form rouleaux[11]. The polythene discs, brought into collision by the flowing liquid or by convection currents, form loosely associated rouleaux resulting in a lowering of the potential energy of the system. But they are easily dispersed again by vibrating the assembly; similarly rouleaux of red blood cells which are more firmly associated may be broken up by shearing the blood[12] as in flow through a tube or in the annulus of a Couette viscometer. The rouleaux of discs are analogous to the flocs or 3-dimensional aggregates seen in aqueous flowing suspensions of pulpwood

fibres - irregular rodlike flexible particles of axis ratio about 50. Here a dynamic equilibrium is established as the flocs are being constantly broken down and built up by the action of the velocity gradient[13]. As the gradient increases so the average size of the floc in the suspension decreases.

The formation of aggregates in the towing rafts and holding booms of pulpwood logs again illustrates the importance of geometry for here, the logs, while of approximately uniform length have a wide distribution of diameters.

Finally, it should be noted that ordered arrays of rodlike units are also found on the molecular scale as in the lamellar micelles[14] present at and near the surface of soap solutions, as well as in liquid crystals or tactoids[15] found in certain polymer solutions.

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LIST OF SYMBOLS

$2a$	length of axis of revolution of rod or disc
a_n	number of aggregates containing n particles
A	total number of aggregates
$2b$	length of diametrical axis of rod or disc, diameter of sphere
n	number of particles in aggregates
N	total number of particles
$f(n), F(n)$	respective differential and integral number distributions of aggregates
$g(n), G(n)$	respective differential and integral number distributions of particles
r_p	particle axis ratio = a/b
t	time
ϕ	orientation of axis of revolution with respect to Y-axis
θ	orientation of axis of revolution with respect to Z-axis
$g(\phi), G(\phi); g(\theta), G(\theta)$	respective differential and integral particle orientation distributions with respect to ϕ; distributions with respect to θ.

PART III

GENERAL SUMMARY

AND

SUGGESTIONS FOR FURTHER WORK

GENERAL SUMMARY

The main conclusions drawn from the experimental work, and some general similarities observed between aggregates of particles and certain aggregates met in practice such as atoms and molecules in a crystal, rouleaux of red blood cells etc., is presented briefly followed by suggestions for further work.

(1) The periodic hexagonal arrangement of spheres in 2-dimensions can be used as a model for the arrangement of atoms in a crystal[1]. The formation of dislocations resulting in V-type and equilateral △ -type structures in a close-packing model, indicated that similar behaviour may be expected in a crystal when subjected to strain. As a result of strain, atoms may slide over each other on certain crystal planes producing plastic deformation[2]. Thus to obtain real strength from the crystal, the main problem is to eliminate all dislocations from the crystal. The dislocations can be blocked by work-hardening[3] in which it is hammered so that dislocations mutually entangle one another's movement in various directions with the result that none can move easily. Furthermore the formation of numbers of small crystallites, numerous dislocations and other faults in the model bubble raft[1] indicated that the crystal is in a state of non-homogeneous strain. The growth of close-packed hexagonal crystallites with time showed that strain has disappeared in the process of recrystallization. This is in qualitative agreement with the experimental model of a monolayer of spheres described in Part II of this thesis.

(2) In the assembly of discs, floating beneath the surface in $Cd(NO_3)_2$ solution, the particles have a tendency to approach each other in a face to face orientation. The aggregates thus formed first increased

in length very quickly and then more slowly as the solution appeared to have come to rest. This physical model of rigid discs is of interest in connection with blood rheology, and demonstrates the importance of shape in the formation of linear chains of red cells in the blood, called rouleaux[4]. It is of interest to note that the formation of rouleaux in a model system was first shown by Norris[5,6], using small cork discs so poised as to float in a vertical position. He observed that they arranged themselves in "rolls" or "cylindrical masses" i.e. rouleaux. From the marked analogy existing between the modes of arrangement of the discs and the red blood cells, he considered that the phenomena were influenced by the same universal law of cohesive attraction.

(3) Upon vibrating the assembly of rods on a solid surface, organized structures[7] as in the log boom were formed. Subsequent analysis showed that at a low frequency of vibration, the number differential particle distribution function at first increased with n, reaching a peak at $n = 4$ whereas the number differential aggregate distribution function decreased continuously with n and that both distributions were unaffected by changes in diameter of the vibrating plate. Furthermore, the most probable particle density shifted to lower n with increasing frequency of vibration. At each axis-ratio, the distributions were shifted to larger n with increasing surface concentration but at a given surface concentration remained unaffected by changing the particle axis-ratio.

SUGGESTIONS FOR FURTHER WORK

The following recommendations for further studies are made:

1. To study the effect of spheres of different sizes on the distribution of aggregates in 2-dimensions.

2. To study the number of contacts and near contacts existing in random packing of equal sized spheres in 3-dimensions.

3. To study the aggregates formed in monodisperse vibrating suspensions of drops at known concentration in a pure liquid by measuring the distribution in drop size resulting from coalescence. The work could be extended to systems containing various amounts of surface active materials.

4. To conduct preliminary studies of the behaviour of aggregates in the Poiseuille flow of dilute suspensions of rigid and deformable spheres.

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APPENDIX I

Fisher Vibrometic Polisher

General Description

A photograph of the vibrometic polisher is given in Figure I. It consists of a flat-bottomed metal bowl, in which the particles are placed. The bowl is supported above the base casting by four heavy leaf-springs. The base is attached to one half of a driving magnet, while the other half is attached to the bowl. When the magnet is pulsed, the bowl tends to move down towards the base, but at the same time because of the heavy springs the bowl is also twisted about the vertical through its centre. Consequently it undergoes a reciprocating combined up and down and at the same time turning motion.

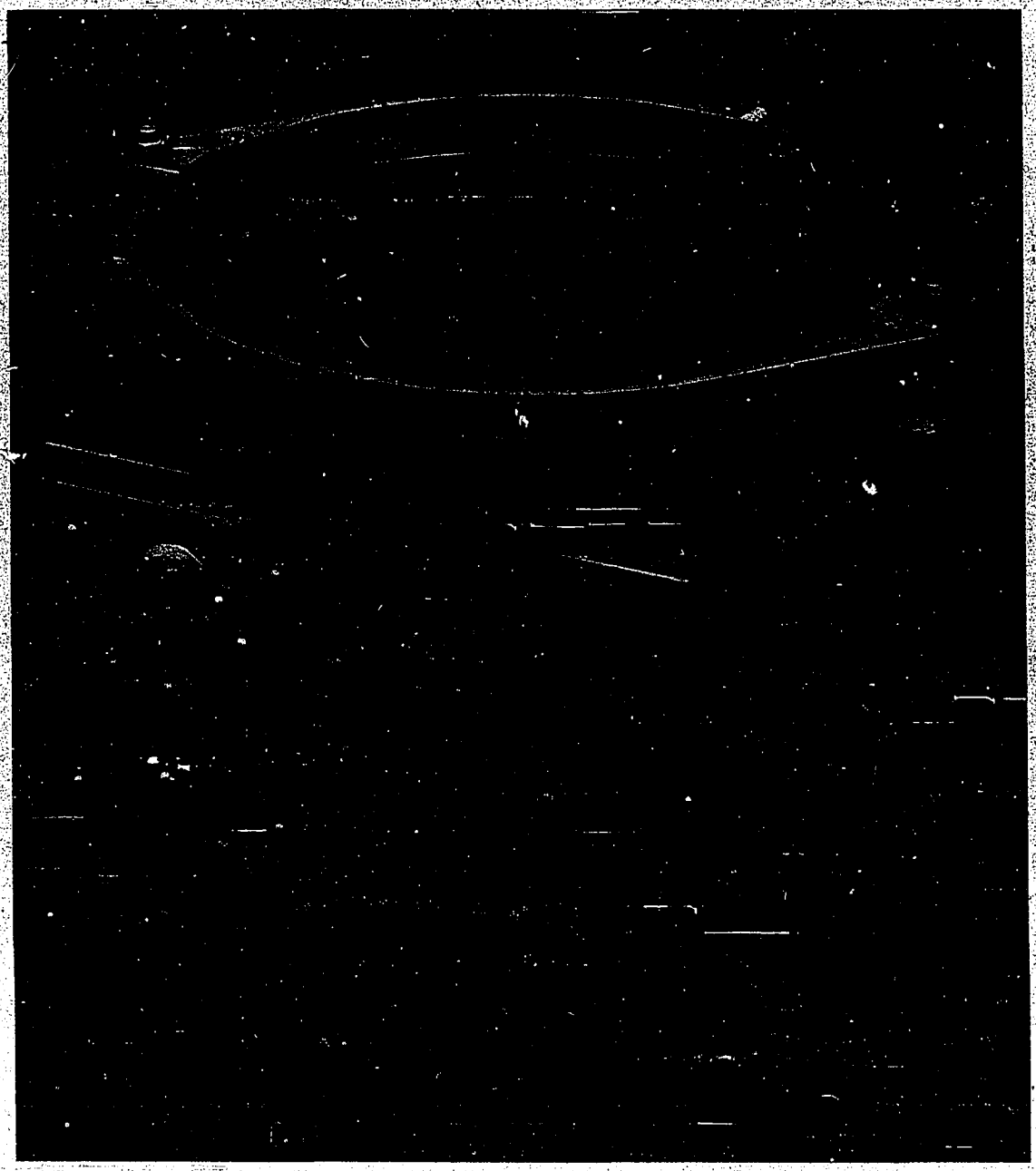
Operational Procedure

The apparatus is placed on a rigid support such as a table or bench. The feet on the base casting may be adjusted in order to bring it to a level position, a spirit level being incorporated in the top of the base part.

A rheostat control is located on the front panel of the polisher. This control varies the rate of vibrations of the bowl, and hence indirectly the speed with which elements move in the bowl. The range of frequency of the vibrations is from 0 to 100 sec^{-1}.

Figure I.

Photograph of Fisher Vibrometic Polisher. The picture shows the instrument mounted with a flat bottomed metal bowl of 26 cm. diameter. It was possible to replace this by different size bowls to accommodate larger numbers of rods of higher axis ratio.




FISHER
LABORATORY
POLISHER

APPENDIX II

Photographs of Strips Cut from the Polymerized Cast of Polythene Discs

The details of the experimental procedure in fixing and analysing the orientation and distribution of rouleaux in the vibrated multilayers of discs were given in Part II of the thesis.

The Figure overleaf shows four successive 1" strips in order from top to bottom cut along the Y-axis (Figure 3b, Part II) at the same distance from the centre from four 1" thick discs sectioned along the median, XY-plane of the polymerized beaker contents. The photographs were taken along the X-axis and show discs and small rouleaux (the most probable aggregate having three discs) sectioned by the cut in the YZ-plane.

Figure II. Photograph of four strips taken from the epoxy cast of vibrated discs in multilayers.

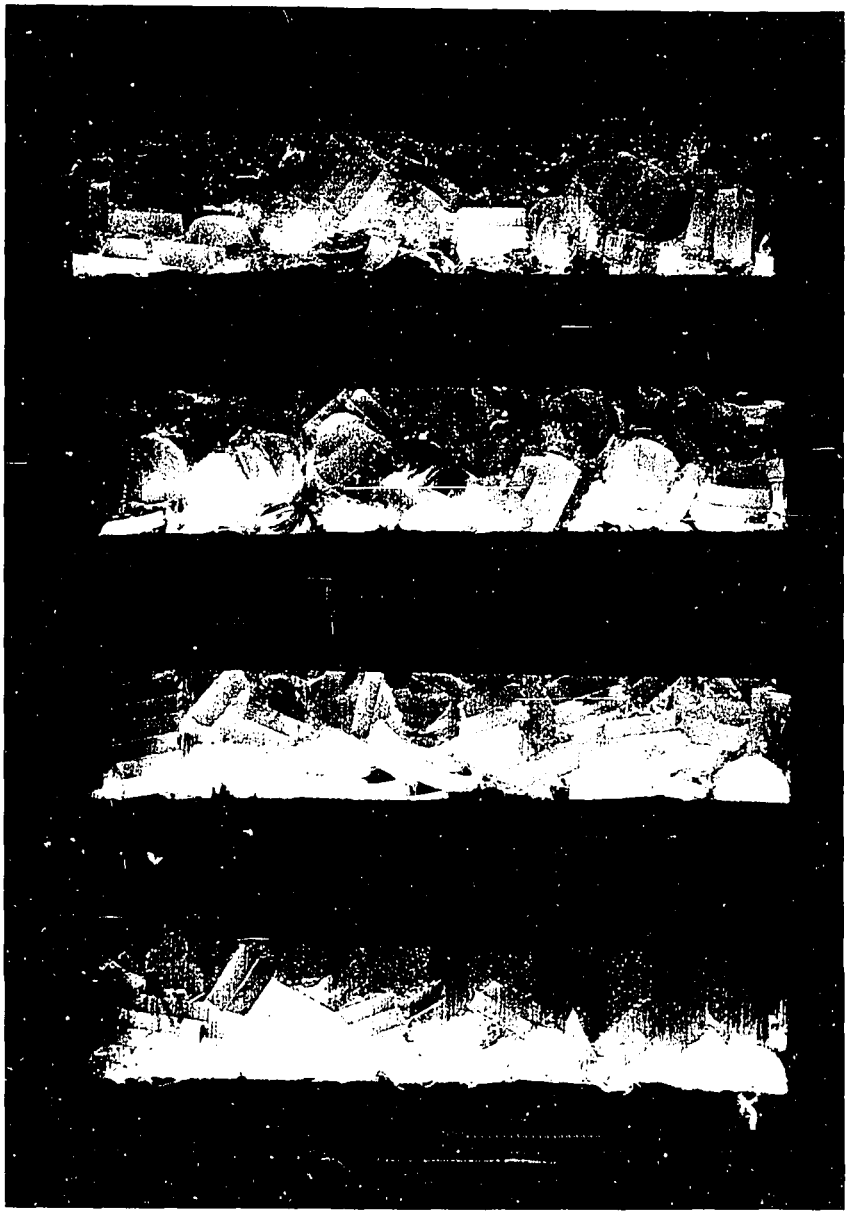


Figure 11 Photograph of four strips taken from the epoxy cast of vibrated discs in multilayers

APPENDIX III

Calculation of Fraction of Surface Area Occupied by Spheres in Closest Packing

A closest packing of like spheres arranged on the points of a triangular lattice is shown in Figure IIIa. The structure is peculiar in the sense that each sphere is in intimate contact with six spheres in its neighbourhood, thus occupying the available space most efficiently. In 2-dimensions, the spheres become circles.

To calculate the portion of the total surface area occupied by the circles we need to find the area of the regular hexagon circumscribing the sphere, radius R.

Each of the six isosceles triangles dividing the hexagon in the figure has height R and base 2R tan 30, hence their area is

$$R^2 \tan 30 = R^2/\sqrt{3} .$$

The efficiency with which the available surface is utilized in this close packing is then given by

$$\frac{\text{Area of sphere}}{\text{Area of hexagon}} = \frac{\pi R^2 \sqrt{3}}{6R^2}$$

$$= \frac{\pi\sqrt{3}}{6} = 0.907 .$$

Similarly, it is easy to show that in cubic close packing (Figure IIIb) the fraction is given by

$$\frac{\text{Area of sphere}}{\text{Area of circumscribed square}} = \frac{\pi R^2}{4R^2} = \frac{\pi}{4}$$

$$= 0.786 .$$

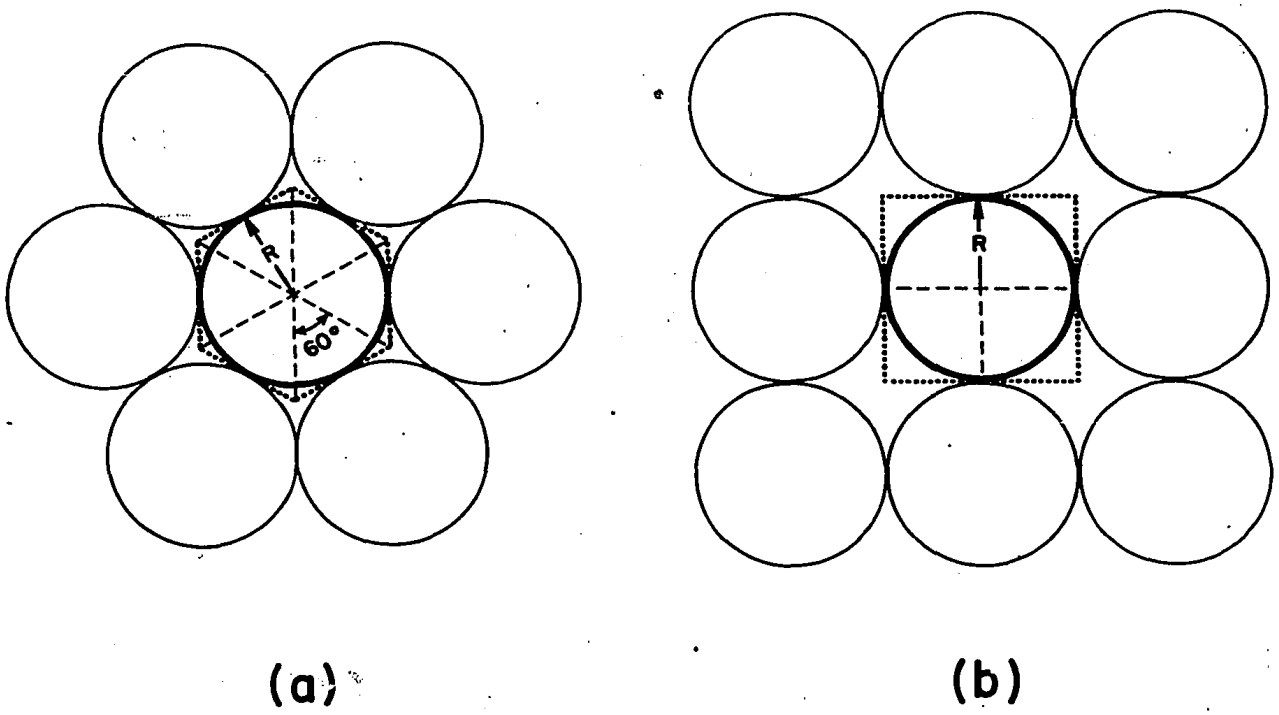


Figure III. Hexagonal (a) and cubic (b) close packing of spheres. It is evident from mere inspection that the packing in the former is more efficient than in the latter. To calculate the fraction of the total area occupied by spheres resting on a horizontal surface it is necessary to find the areas of the respective circumscribed hexagon and square shown in the figure.