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DEFORMATION THEORY OF HOT PRESSING

ABSTRACT

The possible deformation behaviour of spheres in a compact has been theoretically analyzed and experimentally verified. The change in contact area radius 'a' relative to the particle radius R has been related to the bulk density and bulk strain for four possible modes of packing: simple cubic ($Z = 6$), orthorhombic ($Z = 8$), rhombohedral ($Z = 12$), and body-centered cubic ($Z = 8$).

An equation relating the above parameters can be represented by $D - D_0 = \frac{3}{2} D_0 (a/R)^2$ for different types of packings, D and D_0 being the densities at any a/R and at a/R = 0, respectively. It has been shown experimentally by deforming monosized lead spheres at room temperature, 50 and 100°C in a cylindrical die, that the overall deformation is similar to that of the orthorhombically packed spheres. A change in the coordination number Z during the deformation process was also observed and may partially account for the deviation from the theoretically predicted values.

Similar experiments using sapphire and K-Monel spheres were also carried out in the temperature ranges 1570 - 1700°C and 800 - 1000°C respectively. The results showed that the deformation behaviour was very similar to that of the lead spheres.

A study of the geometry of deformation revealed that most of the spheres deformed in a random manner, although individual colonies of orthorhombic, tetragonal and rhombohedral packings were observed. It was also observed that the deformed faces, that were approximately perpendicular to the direction of pressing were about 2.2 times larger than those parallel to the direction of pressing. This observation has been subsequently used to modify the theoretical models. The particle rearrangement and plastic flow have been found to be the predominant mechanisms for the densification of lead, K-Monel, and sapphire spheres under the experimental conditions used in this investigation.

The criterion for yielding of two hemispheres of the same material in contact was used to incorporate the yield strength in the basic density equation. This equation has been found to fit the data obtained during the hot-pressing of the spheres.

It has been observed that the deformation of sapphire single crystal spheres takes place by a complex deformation process. The presence of the basal and prismatic slip has been identified in the spheres deformed at 1570 and 1700°C. Presence of cross slip is also confirmed by the optical and electron micrographs at these temperatures.

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by

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We accept this thesis as conforming to the
required standard

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A B S T R A C T

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I

I N T R O D U C T I O N

Hot-pressing of particulate compacts has recently gained importance since it has an added advantage of giving greater densification at lower temperatures and shorter times than is possible by normal sintering methods. Despite the fact that hot-pressing methods have been in use since 1900, it is only during the last twenty years that efforts have been made towards understanding the mechanisms of the hot pressing process.

Many empirical equations¹⁻⁸, first proposed to express the relationship between the density (volume) of the powder compact and the applied pressure, led to speculations concerning the mechanism of hot-pressing. Modern theories of hot-pressing are derived primarily either from sintering theories or from the stress-enhanced diffusional creep mechanism. The sintering theories are based on either plastic flow^{9,10} or viscous flow¹¹ as the mechanism of deformation of solids. Clark and White⁹ considered the open-pore stage of sintering in which the material is assumed to flow into the lens between the original spherical particles. The later or the closed-pore stage of sintering was investigated by Mackenzie and Shuttleworth¹⁰. Their theory rests on a model consisting of closed pores in a homogeneous matrix which are reduced in size with time as a result of surface forces.

A. THEORIES BASED ON PLASTIC FLOW MECHANISM

Among the notable contributions to the theory of hot-pressing, the best known is, perhaps, that of Murray, Rodgers and Williams¹² (also

Murray, Livey and Williams¹³) who modified the sintering theory of Mackenzie and Shuttleworth to explain the observed behaviour in hot-pressing of various oxides and carbides. Murray included the applied pressure along with the surface tension as the driving force for the rate of densification. The final form of their equation, neglecting the contribution from sintering, is

$$\frac{dD}{dt} = \frac{3P}{4\eta} (1-D)$$

where

D = the relative density at time t

P = the applied pressure

and η = the viscosity at infinite rate of shear.

Their plastic flow theory succeeded in explaining the increase of rate of densification with pressure, and the effect of pressure on the end-point density at constant temperature. Murray's equation has been employed to interpret data on alumina by Mangsen, Lambertson and Best¹⁴; on silica glass by Vasilos¹⁵; in liquid-phase sintering of copper-bismuth, sodium chloride-water and ice-menthol by Kingery, Woulbroun and Charvat¹⁶; and on potassium niobates by Jaeger and Egerton¹⁷. The studies on several carbides by Lersmacher and Scholz¹⁸ also showed that Murray's equation based on the plastic flow model can describe the early stages of hot-pressing, but deviations occur at longer times, particularly at higher temperatures.

McClelland^{19,20} observed that the kinetics of hot-pressing of BeO and Al₂O₃ did not follow the equation proposed by Murray. In his theory, McClelland also considered that the plastic flow mechanism is operative during hot-pressing, but he included the pressure effective in closing the pores instead of the applied pressure in the Mackenzie and

Shuttleworth's equation. His modified equation agreed well with his experimental data on BeO and Al_2O_3 .

B. THEORIES BASED ON THE NABARRO-HERRING CREEP MECHANISM

Koval'chenko and Samsonov²¹ proposed a hot-pressing equation based on the Nabarro²²-Herring²³ creep mechanism, which was verified by studies on tungsten carbide and chromium carbide. Scholz and Lersmacher²⁴ simplified Koval'chenko and Samsonov's equation and showed that it took a form similar to the equation developed by Murray, namely

$$\frac{dQ}{dt} = - \frac{3P}{4\eta} Q$$

where Q = porosity.

C. THE PRESENT CONCEPTS OF HOT-PRESSING

During the past few years, there has been little agreement among investigators regarding the mechanism or mechanisms operative during hot-pressing. Their results differ from those of Murray and they all suggest certain complex mechanisms of hot-pressing. The kinetics of densification during hot-pressing at relatively low temperatures was studied by Felten²⁵ using alumina powders. Although not explicitly described, Felten concluded that the most probable initial densification mechanism was particle rearrangement. This rearrangement may be due to particle sliding, fragmentation, plastic flow and diffusional processes.

Direct microstructural evidence for grain-boundary sliding and fragmentation was obtained by Chang and Rhodes²⁶. They considered that the densification was controlled by particle sliding, fragmentation, plastic flow and volume diffusion. The role of fragmentation in the densification process has been further revealed by Hashimoto²⁷ using calcium

fluoride spheres of uniform size, and by Shapiro²⁸⁻³⁰ using various types of powders. Using single crystal spheres of sapphire, Coble and Ellis³¹ showed that for alumina the contribution of plastic flow is limited by the hot-hardness of the material. Their results show that plastic flow can contribute to the densification process in the early stages of hot-pressing only. Further densification is ascribed to enhanced diffusion under the influence of stress, e.g., diffusional creep.

Vasilos and Spriggs³² also proposed diffusion-controlled creep as the mechanism for densification of pure alumina and magnesia beyond the initial stages. In contrast to the work of Murray and McClelland, they observed that high purity magnesia could be pressure-sintered continuously to essentially full density over a range of temperatures. Rossi and Fulrath³³ studied the kinetics of the final stage of densification of alumina under vacuum hot-pressing conditions. They also suggested that plastic flow may be operative at an intermediate stage but definitely not during the final stage, where diffusion-controlled creep is proposed as the mechanism responsible for densification. Rossi and Fulrath further concluded that there is no particle rearrangement in the final stage.

D. SCOPE OF PRESENT INVESTIGATION

From the preceeding review of the literature, it appears, that three densification mechanisms have been considered to be operative in the hot-pressing process:

1. Particle rearrangement aided by sliding and fragmentation
2. Plastic flow and
3. Stress-enhanced diffusional creep.

It appears that because of the complexities of the variables involved during hot-pressing, no quantitative study has been made, so far, to account for each individual stage. To treat the different stages mathematically, it is necessary to know or to control the geometry of the particles and the packing configurations. Pelzel³⁴ attempted to correlate the percentage bulk density of a spherical compact with the total surface-contact area produced by continuous deformation. He calculated his models on the basis of six-fold and twelve-fold coordinations.

An attempt has been made in the present work to separate the various stages and to study the contribution from the plastic flow mechanism and the particle reorientation to the densification process during the early stages of hot-pressing. Theoretical models have been formulated on the basis of the plastic deformation of spheres in a compact. The starting point for the models is various modes of simple and systematic packing of uniform spheres of equal size.

The validity of these models has been tested by deforming uniform spheres of lead, K-Monel and single crystal sapphire.

II

THEORETICAL MODELS

PART ONE-THEORY

A quantitative study of the hot-pressing process is greatly simplified by assuming that the compact is composed of uniform spheres of equal size and that the spheres are packed in some regular arrangement. The packing of spheres of uniform size has been studied by many workers in different fields. The findings of Graton and Fraser³⁵ are most significant in this respect. The basic systems of packing are described in Appendix I and can be summarised as follows:

1. Simple cubic ($Z=6$)
2. Body-centered cubic ($Z=8$)
3. Orthorhombic ($Z=8$)
4. Tetragonal ($Z=10$)
5. Rhombohedral ($Z=12$)

where Z is the coordination number.

Of these, the b.c.c. packing is an unstable arrangement in a unidirectional field of force (i.e., gravitational force), and is considered here for theoretical interests only. Furthermore, the following assumptions have been made to develop the models quantitatively:

1. Each of the above packings is stable and maintains its symmetry upon the application of pressure
2. The pressure is applied equally from all sides
3. The material is isotropic and
4. Deformation takes place by the plastic flow mechanism.

Under these conditions, on application of the pressure, plastic deformation will take place at the points of contact and flat circular faces will form on each particle. As the radius 'a' of the face increases, the radius 'R' of the sphere must increase to maintain a constant volume. This is true only if the material at the faces spreads symmetrically to maintain the sphericity of the particle. Under such conditions, the spheres will be deformed into uniformly shaped polyhedra depending upon the coordination number. Thus, simple cubic packing will give rise to cubes, orthorhombic packing will give hexagonal prisms, and rhombohedral packing will produce rhombic dodecahedrons on complete deformation. In the b.c.c. case, the spheres begin to deform in the shape of an octahedron, but a critical stage is reached when six spheres at the face-centered positions begin to touch. On further deformation, the polyhedron formed is known as a tetrakaidecahedron ($Z=14$). Coble³⁶ used a model (based on the tetrakaidecahedron) in deriving the sintering equations for the intermediate and final stages of densification. The tetragonal packing does not seem to conform to a regular polyhedron on deformation and so cannot result in complete densification; therefore it has not been considered here.

A. GEOMETRIC RELATIONSHIPS

Figure 1 shows the formation of a flat face between two spheres in contact. The geometry of the deformation process can be considered as follows.

The volume of the particle at any deformation is given by

$$V_p = V - Z \times V_s$$

where

V = volume of the sphere

Z = the coordination number

V_s = volume of a segment of the sphere.

If R is the radius of the sphere, and h is the height of the segment of the sphere, then we have

$$V = \frac{4}{3} \pi R^3$$

$$\text{and } V_s = \frac{\pi h^2}{3} (3R - h)$$

Therefore,

$$V_p = \frac{4}{3} \pi R^3 - Z \left[\frac{\pi h^2}{3} (3R - h) \right]$$

It can be seen from Fig. 1, that

$$h = R - y \quad (1)$$

$$\text{and } R^2 = y^2 + a^2 \quad (2)$$

1. Cubic Model

There are six points of contact in this case giving rise to six flat faces.

The volume of the particle at any deformation will be given by

$$V_p = \frac{4}{3} \pi R^3 - 6 \left[\frac{\pi h^2}{3} (3R - h) \right]$$

Using Eq. (1) and (2), and on simplification it can be

SPHERES IN CONTACT

Fig. 1 Geometry of Deformation of Two Spheres in Contact.

shown that

$$V_p = 2\pi(2R^2 + a^2) (R^2 - a^2)^{1/2} - \frac{8\pi R^3}{3} \quad (3)$$

This also equals the original volume of the sphere $\frac{4\pi R_o^3}{3}$, where R_o is the original radius of the sphere. Therefore, from Eq. (3)

$$\begin{aligned} 2\pi(2R^2 + a^2) (R^2 - a^2)^{1/2} - \frac{8\pi}{3} R^3 &= \frac{4\pi R_o^3}{3} \\ &= \text{const.} = 1 \quad (\text{e.g.}) \quad (4) \end{aligned}$$

The radius of the face increases as the deformation proceeds and a critical radius is reached when the six circular faces begin to touch (Fig. 2a). For this condition, from Fig. 2b

$$\begin{aligned} a_{\text{Crit}} &= y_{\text{Crit}} \\ &= \frac{1}{\sqrt{2}} R_{\text{Crit}} \quad \text{for } y^2 + a^2 = R^2 \end{aligned}$$

Substituting this in Eq. (4) gives

$$R_{\text{Crit}} = 0.71553 \text{ (arbitrary units)}$$

2. Hexagonal Prismatic Model

There are eight points of contact. In this case we have

$$\begin{aligned} V_p &= \frac{4\pi R^3}{3} - 8\left[\frac{\pi h^2}{3} (3R-h)\right] \\ &= \frac{8\pi}{3} (2R^2 + a^2) (R^2 - a^2)^{1/2} - 4\pi R^3 \\ &= \frac{4\pi R_o^3}{3} = 1 \quad (\text{e.g.}) \quad (5) \end{aligned}$$

The critical stage is reached in this case when the six faces in the close-packed layer begin to touch (Fig. 3a). For this condition,

CUBIC MODEL

Fig. 2 Geometric Relationship of Cubic Model Showing the Critical Stage.

HEXAGONAL PRISMATIC MODEL

Fig. 3 Geometric Relationship of Hexagonal Prismatic Model Showing the Critical Stage.

$$a_c = \frac{1}{\sqrt{3}} y_c$$

$$= 0.5 R_c$$

Substituting this in Eq. (5) gives

$$R_c = 0.64321 \text{ (arbitrary Units).}$$

3. Rhombic Dodecahedron Model

There are twelve points of contact in this case. Therefore, we have

$$V_p = \frac{4\pi R^3}{3} - 12 \left[\frac{\pi h^2}{3} (3R-h) \right]$$

$$= 4\pi (2R^2 + a^2) (R^2 - a^2)^{1/2} - \frac{20\pi R^3}{3}$$

$$= \frac{4}{3} \pi R_o^3 = 1 \text{ (e.g.)} \quad (6)$$

The critical stage is reached in this case when the twelve faces begin to touch (Fig. 4a). Also in this model six faces in the close-packed layer begin to touch (Fig. 4b). The second condition is the same as that obtained in the hexagonal prismatic model, giving

$$a_c = 0.5R_c$$

Alternately, from Fig. 4c

$$a_c = \frac{D_1}{2} \cos \theta \quad (7)$$

From geometry, it can be shown that

$$D_1 = 2y$$

$$\text{and } \theta = 54^\circ 44'$$

Substituting these values in Eq. (7), gives

$$a_c = y_c \cos 54^\circ 44'$$

$$= 0.5R_c$$

RHOMBIC DODECAHEDRON MODEL

Fig. 4 Geometric Relationship of Rhombic Dodecahedron Model Showing the Critical Stage.

which on substitution in Eq. (6) gives

$$R_c = 0.65560 \text{ (arbitrary units).}$$

4. Tetrakaidecahedron Model

Initially this model has the configuration of the b.c.c. packing. Since there are two critical stages in this case, subscripts 1 and 2 have been used to designate the two stages.

a) B.C.C. Model

There are eight points of contact in this case.

Therefore, we have

$$\begin{aligned} V_p &= \frac{4}{3} \pi R^3 - 8 \left[\frac{\pi h_1^2}{3} (3R - h_1) \right] \\ &= \frac{8\pi}{3} (2R^2 + a_1^2) (R^2 - a_1^2)^{1/2} - 4\pi R^3 \\ &= \frac{4\pi}{3} R_o^3 = 1 \text{ (e.g.)} \end{aligned} \quad (8)$$

The critical stage is reached in this case when the six spheres in the face-centered positions begin to touch (Fig. 5b). This is considered as the first critical stage. From Fig. 5b, it can be seen that

$$y_2 = 2y_1 \sin \theta \quad (9)$$

and it can be shown, from geometry, that

$$\theta = 35^\circ 16' 30''$$

Now, at this first critical stage,

$$y_2 = R$$

Therefore, Eq. (9) becomes

$$R = 2 (R^2 - a_1^2)^{1/2} \sin \theta$$

which on simplification gives

$$(a_1)_{c_1} = 0.5 (R_c)_1$$

Substituting this in Eq. (9) gives

$$(R_c)_1 = 0.64321 \text{ (arbitrary units)}$$

b) Tetrakaidecahedron Model

After the first critical stage, the deformation proceeds until the second critical stage is reached when all fourteen faces come into contact (Fig. 6a). For this condition, from Fig. 6b

$$(a_1)_{c_2} = \frac{1}{2\sqrt{3}} L$$

where L = edge length of the octahedron shown in Fig. 5a.

It can be shown that

$$L = \sqrt{6} y_1$$

This gives $(a_1)_{c_2} = \frac{1}{\sqrt{2}} (\bar{y}_c)_1$

$$= \frac{1}{\sqrt{3}} (R_c)_2 \quad (10)$$

Also, from Fig. 6c

$$\begin{aligned} (a_2)_c &= \frac{1}{6} L \\ &= \frac{1}{\sqrt{6}} \bar{y}_1 \end{aligned}$$

Since from Eq. (9)

$$\bar{y}_2 = 2\bar{y}_1 \sin \theta$$

B.C.C. MODEL

Fig. 5 Geometric Relationship of B.C.C. Model Showing the Critical Stage.

TETRAKAIDCAHEDRON MODEL

Fig. 6 Geometric Relationship of Tetrakaidecahedron Model Showing the Critical Stage.

therefore, we have

$$\begin{aligned}
 (a_2)_c &= \frac{1}{2\sqrt{6}} \sin \theta (y_c)_2 \\
 &= \frac{1}{2\sqrt{2}} (y_c)_2 \\
 &= \frac{1}{3} (R_c)_2
 \end{aligned} \tag{11}$$

Alternately, from Eq. (9) we have

$$(R^2 - a_2^2)^{1/2} = 2(R^2 - a_1^2)^{1/2} \sin \theta$$

Substituting $(a_1)_{c_2} = \frac{1}{\sqrt{3}} (R_c)_2$ from Eq. (10) and simplifying we have,

$$(a_2)_c = \frac{1}{3} (R_c)_2$$

The volume of the particle in this case is given

by

$$\begin{aligned}
 V_p &= \frac{4\pi}{3} R^3 - 8 \left[\frac{\pi h_1^2}{3} (3R - h_1) \right] - 6 \left[\frac{\pi h_2^2}{3} (3R - h_2) \right] \\
 &= \frac{8\pi}{3} (2R^2 + a_1^2) (R^2 - a_1^2)^{1/2} + 2\pi (2R^2 + a_2^2) \\
 &\quad \times (R^2 - a_2^2)^{1/2} - 8\pi R^3 \\
 &= \frac{4\pi}{3} R_o^3 = 1 \text{ (e.g.)}
 \end{aligned} \tag{12}$$

Substituting the values of the critical radii from Eqs. (10) and (11) into Eq. (12), we get

$$(R_c)_2 = 0.66940 \text{ (arbitrary units)}$$

It can be seen that all the foregoing calculations can be summarized into a single equation, namely

$$\begin{aligned}
 V_p &= \alpha (2R^2 + a^2) (R^2 - a^2)^{1/2} - \beta R^3 \\
 &= \frac{4\pi}{3} R_o^3 = 1 \text{ (e.g.)}
 \end{aligned} \tag{13}$$

where α, β are constants with different values for each model. These values are given in Table I.

For each of the models, the change in R values at various stages of deformation can be calculated from the constant-volume equation. These values (ALL THEORETICAL CALCULATIONS HAVE BEEN CARRIED OUT USING AN IBM COMPUTER) are summarized in the form of a graph relating a/R and R as shown in Fig. 7. It is seen that up to a value of $a/R = 0.25$, R is approximately equal to R_0 (the initial radius) and for this reason all experimental work was limited to $a/R = 0.25$.

The geometry of the deformation for all the models is no longer the same after the critical stage has been reached. Beyond the critical stage, the spheres are further deformed to the respective polyhedra giving 100% densification.

B. DEDUCED RELATIONSHIPS

From the above derivations the following relationships can be deduced.

1. Bulk Density

Bulk density is defined as the ratio of the volume of the particle to that of the volume of the unit cell, the volume of the unit cell being the sum of the volume of the particle and its associated void space volume. The volume of the unit cell for the various models can be calculated from the respective schematic diagrams (Figs. 2 to 6) in terms of the particle radius R and the face radius a at any stage of deformation. The bulk densities for the various models can be calculated

Fig. 7 Theoretical Relationship of R vs a/R for the Proposed Models.

as follows.

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a) Cubic Model

In this case the unit cell is a cube. The volume of the unit cell at any stage of deformation can be calculated from Fig. 2a.

The volume of the unit cell is given by

$$V_u = L^3$$

where L = edge length

$$\begin{aligned} &= 2y \\ &= 2(R^2 - a^2)^{1/2} \end{aligned}$$

therefore, we have

$$V_u = 8(R^2 - a^2)^{3/2}$$

Now,

$$\text{the bulk density} = \frac{\text{volume of the particle}}{\text{volume of the unit cell}}$$

$$\text{or} \quad D = \frac{1}{8(R^2 - a^2)^{3/2}}$$

$$[\text{or in percent,} \quad D = \frac{1}{8(R^2 - a^2)^{3/2}} \times 100\%] \quad (14)$$

We know that,

when $a = 0$, then $R = R_0$. Substituting this condition in Eq. (4) gives

$$\frac{4}{3} \pi R_0^3 = 1$$

$$\text{or} \quad R_0 = 0.62035 \text{ (arbitrary units)}$$

therefore, for $a = 0$, we have

$$\begin{aligned} D = D_0 &= \frac{1}{8R_0^3} \times 100 \\ &= 52.36\% \end{aligned}$$

This value of the percentage bulk density agrees with the value of the density of packing of uniform spheres in a cubic array (Table I).

When $a = a_c$, then $R = R_c = \sqrt{2} a_c = 0.71553$ (arbitrary units).

therefore, we have

$$\begin{aligned} D = D_c &= \frac{1}{8(R_c^2 - a_c^2)^{3/2}} \times 100 \\ &= \frac{1}{\sqrt{8} R_c^3} \times 100 \\ &= 96.51\% \end{aligned}$$

The values of the bulk density for other a/R values have been similarly calculated and are given in Appendix II and plotted in Fig. 8.

b) Hexagonal Prismatic Model

In this case the unit cell is a hexagonal prism. The volume of the unit cell at any stage of deformation can be calculated from Fig. 3a.

The volume of the unit cell is given by

$$V_u = \frac{3\sqrt{3}}{8} D_1^2 H$$

where, from geometry

D_1 = diameter of the base of the hexagonal prism

$$= \frac{4y}{\sqrt{3}}$$

$$\text{and } H = 2y$$

therefore, we have

$$\begin{aligned} V_u &= 4\sqrt{3} y^3 \\ &= 4\sqrt{3} (R^2 - a^2)^{3/2} \end{aligned}$$

this gives,

$$D = \frac{1}{4\sqrt{3} (R^2 - a^2)^{3/2}} \times 100\% \quad (15)$$

We know that,

when $a = 0$, then $R = R_o = 0.62035$ (arbitrary units).

Substituting this in Eq. (15) gives

$$D_o = 60.46\%$$

and

when $a = a_c$, then $R = R_c = 2a_c = 0.64321$ (arbitrary units).

therefore, from Eq. (15)

$$D_c = 83.51\%$$

The values of the bulk density for other a/R values have been similarly calculated and are given in Appendix II and plotted in Fig. 8.

c) Rhombic Dodecahedron Model

The unit cell for this case is a rhombic dodecahedron. The volume of the unit cell at any stage of deformation can be calculated from Fig. 4a.

The volume of unit cell is given by

$$V_u = \frac{D_1^3}{\sqrt{2}}$$

where

$$\begin{aligned} D_1 &= \text{larger diagonal of the rhomb} \\ &= 2y \\ &= 2(R^2 - a^2)^{1/2} \end{aligned}$$

therefore, we have

$$V_u = \frac{4\sqrt{2}}{3} (R^2 - a^2)^{3/2}$$

This gives

$$D = \frac{1}{4\sqrt{2} (R^2 - a^2)^{3/2}} \times 100 \quad (16)$$

We know that,

when $a = 0$, then $R = R_o = 0.62035$ (arbitrary units)

Substituting this in Eq. (16) gives

$$D_o = 74.05\%$$

and

when $a = a_c$, then $R = R_c = 2a_c = 0.65560$ (arbitrary units)

therefore, from Eq. (16),

$$D_c = 96.40\%$$

The values of the bulk density for other a/R values have been similarly calculated and are given in Appendix II and plotted in Fig. 8.

d) Tetrakaidecahedron Model

The unit cell for this case is a tetrakaidecahedron. The volume of the unit cell at any stage of deformation can be calculated from Fig. 6.

The volume of the unit cell is given by

$$V_u = \frac{8\sqrt{2}}{27} L^3$$

where $L = \text{edge of the octahedron}$

$$= \sqrt{6}y_1$$

$$= \sqrt{6}(R^2 - a_1^2)^{1/2}$$

therefore, we have

$$V_u = \frac{32\sqrt{3}}{9} y_1^3$$

This gives

$$D = \frac{1}{\frac{32\sqrt{3}}{9} (R^2 - a_1^2)^{3/2}} \times 100\% \quad (17)$$

Fig. 8 Theoretical Relationship of D vs $\frac{a}{R}$ for the Proposed Models.

We know that,

When $a = 0$, then $R = R_o = 0.62035$ (arbitrary units)

Substituting this in Eq. (17) gives

$$D_o = 68.02\%$$

and when $a = (a_1)_{c_1}$, then $R = (R_c)_1 = 2 (a_1)_{c_1} = 0.64321$ (arbitrary units)

therefore, from Eq. (17)

$$D_{c_1} = 93.95\%$$

also when $a = (a_1)_{c_2}$, then $R = (R_c)_2 = \sqrt{3}(a_1)_{c_2} = 3(a_2)_c = 0.66490$
(arbitrary units)

This on substitution in Eq. (17) gives

$$D_{c_2} = 99.45\%$$

The values of the bulk density for other a/R values have been similarly calculated and are given in Appendix II and plotted in Fig. 8.

e) Relationship Between D and D_o

It can be seen from the foregoing calculations that

$$D = \frac{1}{A(R^2 - a^2)^{3/2}} \quad (18)$$

where A is a constant having different values for different models.

For $a = 0$, $R = R_o$, and

$$D = D_o = \frac{1}{AR_o^3} \quad (19)$$

Combining Eqs. (18) and (19),

$$\frac{D_o}{D} = \left[\frac{R^2 - a^2}{R_o^2} \right]^{3/2} \quad (20)$$

for $a/R = 0.25$, R is essentially a constant $= R_o$

Therefore,

$$\frac{D_0}{D} = \left[1 - \frac{a^2}{R^2} \right]^{3/2}$$

or

$$D = D_0 \left[1 - \frac{a^2}{R^2} \right]^{-3/2}$$

which, on expansion by the binomial theorem (neglecting squares and higher power), gives

$$D = D_0 \left(1 + \frac{3}{2} \frac{a^2}{R^2} \right) \quad (21)$$

or

$$D - D_0 = \frac{3}{2} D_0 \frac{a^2}{R^2} \quad (22)$$

Also, it comes out empirically that $(D - D_0)$ is very nearly equal to $101.5 \times \log Z (a/R)^2$ for cubic, hexagonal prismatic, and rhombohedral models. ^{ic dodecahedron}

2. Bulk Strain

Bulk strain ϵ_b can be defined as

$$\epsilon_b = (1 + \epsilon)^3 - 1 \quad (23)$$

where

$$\epsilon = \frac{y - R_0}{R_0}$$

therefore,

$$\begin{aligned} \epsilon_b &= \frac{y^3}{R_0^3} - 1 \\ &= \left[\frac{R^2 - a^2}{R_0^2} \right]^{3/2} - 1 \end{aligned} \quad (24)$$

Alternately,

Bulk strain can be defined as equal to the volume of the unit cell at any deformation minus the volume of the unit cell at no deformation divided by the volume of the unit cell at no deformation. From the foregoing calculations, we know that the volume of the unit cell at any deformation is given by $A(R^2 - a^2)^{3/2}$ and the volume of the unit cell at no deformation is given by AR_0^3 , where A is a constant with different values

for different models.

Therefore, we have

$$\begin{aligned}\epsilon_b &= \frac{A(R^2 - a^2)^{3/2} - AR_o^3}{AR_o^3} \\ &= \left[\frac{R^2 - a^2}{R_o^2} \right]^{3/2} - 1\end{aligned}$$

which is the same as Eq. (24). The bulk strain values for different a/R values have been calculated using Eq. (24) and are given in Appendix II and plotted in Fig. 9.

3. Bulk Density and Bulk Strain

Combining Eqs. (20) and (24) we get,

$$\begin{aligned}\frac{D_o}{D} &= 1 + \epsilon_b \\ \text{or} \quad D &= \frac{D_o}{1 + \epsilon_b}\end{aligned}\tag{25}$$

For small values of bulk strain, Eq. (25) can be expanded to give

$$D = D_o (1 - \epsilon_b)\tag{26}$$

This gives a straight-line relationship between the bulk density and the bulk strain, for small values of bulk strain.

4. D, a/R, and Z

In order to correlate all these parameters, a three dimensional graph has been constructed between D , a/R , and Z . (Fig. 10). It is interesting to note that the b.c.c. model does not seem to fall on the same surface as the one drawn through the other models.

Fig. 9 Theoretical Relationship of ϵ_b vs a/R for the Proposed Models.

Fig. 10 Theoretical Relationship of D vs a/R vs Z for the Proposed Models.

TABLE I Deformation Models and Their Characteristics

No.	Type of packing	Deformation model	α^*	β^*	Volume of the unit cell	% Bulk Density at	
						$R = R_o$	$R = R_c$
1.	Simple Cubic	Cubic	2π	$8\pi/3$	$8(R^2 - a^2)^{3/2}$	52.36	96.51
2.	Orthorhombic	Hexagonal Prismatic	$8\pi/3$	4π	$4\sqrt{3}(R^2 - a^2)^{3/2}$	60.46	83.51
3.	Rhombohedral	Rhombic Dodecahedron	4π	$20\pi/3$	$4\sqrt{2}(R^2 - a^2)^{3/2}$	74.05	96.41
4.	B.C.C.	Tetrakaidecahedron	$8\pi/3$	4π	$(32\sqrt{3}/9)(R^2 - a_1^2)^{3/2}$	68.02	(1) 93.95 (2) 99.45

* Ref. to Eq. (13).

PART TWO - EXPERIMENTAL VERIFICATION OF MODELS

A. PARTICLE REARRANGEMENT

In actual practice, the ideal modes of packing assumed earlier in the theoretical models and discussed in Appendix I are not obtained. The cubic, orthorhombic, tetragonal, or rhombohedral packings, e.g., do not exist across the diameter of the die, because the width of the die does not permit an integral number of spheres. Also, the wall appears to hold up a certain number of spheres. On the application of a small pressure, spheres which have been lightly held up by friction or by bridges will collapse to lower positions; some others will follow shortly afterwards to fill the void space created by the movement. Thus, some restacking of the particles always takes place on the initial pressure application.

The particle restacking was studied in a few preliminary experiments using glass spheres of 380 to 400 μ diameter to take this into account while testing the models experimentally. The glass spheres were rolled down an inclined panel to separate the uniform and regular spheres from the irregular ones. Figure 11 shows such an arrangement. The roundest spheres rolled down the panel through the selector and were subsequently used for the experiments. A heat-treated cylindrical die (1.25 in. diam. and 0.25 in. height) made from steel (Keewatin) was used in conjunction with a specially designed strain gauge having an accuracy of ± 0.001 in. The whole assembly was loaded in a floor-model testing machine (Instron)⁴ for deformation purposes. The glass spheres were pressed to a total load of 1000 lb using a strain rate of 0.04 in./in./min. No fragmentation of the glass spheres was observed. The strain remaining

Fig. 11 Apparatus for Separating Uniform Spheres from the Irregular Ones.

Fig. 12 Volume Strain (%) vs the Initial Applied Load.

on the removal of the load was noted and was considered to be due to restacking or repacking. In addition, separate experiments were carried out using arbitrary "initial loads" of 2, 5, 10, 20, and 40 lbs and then subsequently pressing to a final load of 1000 lb. On decreasing the load to the initial values, the amount of permanent strain was noted for each "initial load". The strain remaining for each initial applied load was then plotted against the initial applied load and, on extrapolation, produced the value of strain at zero initial applied load. The results (Fig. 12) show that a volume change of about 1.25% can be produced by the application of 10 psi. Lubricating the spheres with graphite powder had no effect on the strain produced. Experiments carried out using copper spheres by Duffield and Grootenhuis³⁷ produced a volume change of 1.3- 4.3% for an applied pressure of 10 psi. The discrepancy between these two results may be due to the mode of packing, the size of the spheres, and the manner in which stress is applied.

B. PLASTIC DEFORMATION OF SPHERES

1. Materials Used

The theoretical models were tested using lead (2% Sb) spheres because of the convenience of working with a metal having a low melting point. The nature of the study required that sufficiently low temperatures should be used so that there is no interaction at the contact points. This is particularly important for accurate measurements of 'a', the contact face radius. In addition, the range of pressure should be large enough to produce deformation within the intended range of study. i.e. up to $a/R \approx 0.25$.

The study of the contribution of plastic flow to the mechanisms of densification during hot-pressing of ceramic oxides was carried out using single crystal sapphire spheres of 1 mm diameter. The high cost of conducting each experiment limited the number of experiments to three only. Spheres of K-Monel were also used to test models further. Extension of the models showed that it is possible to incorporate in the basic density equation material properties, such as the yield strength.

The results in this section will be presented and discussed with respect to lead and sapphire, though some results for K-Monel will also be given.

2. Experimental Techniques

a) Lead

Lead (2 wt.% Sb) shots 0.120 - 0.125 in. diameter were used for the investigation. The most regular spheres, separated by rolling them down the glass panel using the assembly shown in Fig. 11, were used for the experiments. A heat-treated cylindrical die (2 in. diam and 0.564 in. height) made from steel (Keewatin) was used in conjunction with the strain gauge described earlier. The maximum initial density was obtained by packing the die in small installments with intermittent shaking and tapping. An initial load of 10 lb gave a good reference point. A strain rate of 0.35 in./in./min. was used for the compaction. The spheres were pressed to the various final loads and the actual permanent strain was obtained from the difference in the initial and final readings on the chart at the same "initial loads". A typical load-compaction curve obtained during the experiment is shown in Fig. 13.

Fig. 13 Load-Compaction Curve in Case of Lead Spheres.

Fig. 14 Assembly for Hot-Pressing Lead Spheres.

Fig. 15 Schematic Representation of the Die used for Hot-Pressing of Lead Spheres.

Experiments at 50° and 100°C were carried out by fixing resistance heating coils in the upper and lower plungers. The temperature was measured at the bottom of the die using a chromel-alumel thermocouple and a potentiometer. A drop in the temperature was observed during deformation and was counterbalanced by increasing the current. It was possible to control the temperature to within $\pm 3^\circ\text{C}$. The initial load in the high temperature tests was reduced to 2 lb to avoid any initial deformation. Figure 14 shows the assembly used in the experiment while Fig. 15 shows the schematic representation of the die.

b) Single Crystal Sapphire

Sapphire spheres of 1 mm diam. (supplied by A. Miller and Co. Libertyville, Illinois) were deformed in a graphite die (0.75 in. diam) at 1570°, 1600° and 1700°C at 2000 psi in a specially designed vacuum hot-press (the vacuum was 0.5 atm.). The desired temperature was reached within 15-20 minutes. It was possible to control the temperature to within $\pm 10^\circ\text{C}$. The strain produced was measured using a dial gauge which had a sensitivity of 0.001 in. The schematic diagram of the assembly is shown in Fig. 16.

An instantaneous deformation was observed on application of the load which slowed down significantly within a few seconds. To be sure that deformation at the points of contact occurred by plastic flow rather than by diffusional creep, the applied load was maintained for only 30 seconds. The permanent strain was noted from the difference in the dial gauge readings before applying the load and after removing the load. The compact was cooled rapidly, after removing the load, by flushing Argon gas through the system so that no sintering occurred during cooling.

Fig. 16 Schematic Representation of the Vacuum Hot-Press
Used for Sapphire and K-Monel Spheres.

c) K-Monel

K-Monel spheres of $1/16 \pm 0.002$ in. diameter were vacuum hot-pressed in the same assembly as used for sapphire. They were deformed in a graphite die (1 in diam) at 800°, 900° and 1000°C at pressures of 625, 937.5, 1250, and 1562.5 psi. The load was held for 2 minutes only. It was found that the deformation was complete within a few seconds, as the dial gauge did not record any further change in the reading after this period. The desired temperature was reached within 5-10 minutes.

3. Experimental Procedures

After the spheres in the die were deformed by the application of a certain predetermined load, the spheres were taken out systematically to measure the flat faces formed from which an average value of Z was obtained. The average diameter of the faces was also measured using a specially designed goniometer. The assembly used for studying the flat faces is shown in Fig. 17. Typical flat faces formed during deformation are shown in Fig. 18. The density of the spheres was determined by the pycnometer method and the percentage bulk density of the compact was calculated from the weight and the volume of the compact.

The pressure within the die was not the same at all points contrary to the hydrostatic conditions assumed in the theoretical models. In an attempt to determine the variation in a/R and Z values in the whole compact, spheres from layers (three rows only) in two perpendicular directions were examined for the average value of a/R and the coordination number Z . Figure 19 shows the statistical distribution of the number of spheres vs the coordination number. It can be seen that the

Fig. 17 Apparatus for Studying the Deformed Spheres.

LEAD

K-MONEL

SAPPHIRE

Fig. 18 Photomicrographs of the Deformed Spheres Showing the Typical Flat Faces.

Fig. 19 Statistical Distribution of the Number of Spheres vs the Coordination Number.

mean coordination number ($Z = 7.9$) of the spheres, including those touching the die-walls is about 8% smaller than those from the central portion of the compact (leaving the die-walls) ($Z=8.5$). Because of the nature of the applied stress, which was uniaxial, instead of hydrostatic as assumed in the models, the different contact face radii on the individual spheres varied widely, as expected. This variation was particularly evident when the faces formed normal to the direction of the applied stress were compared with the faces formed parallel to the direction of the applied stress. However, the average value of a/R for all the spheres (omitting the sides, top and bottom layers) was within 3 - 4% of the mean value as determined from 20 spheres selected randomly from the central portion of the die. It was also found that the accuracy of measurement could have been improved to within 2 - 3 % of the mean value by using 50 spheres instead of selecting 20. In the case of the spheres from the sides, top and bottom layers, the a/R values were slightly larger (about 5 - 7%) than the rest of the compact, but the Z values were relatively smaller (about 10%), so that the overall error introduced by not considering the spheres in contact with the die-walls would be very small.

From all these considerations, it was decided that 20 spheres of lead, randomly selected from the central region, could be considered as representative of each experiment. The error introduced by studying only 20 spheres from the central region of the compact was considered acceptable in view of the time saved in not studying all the spheres (about 800) from each experiment.

4. Results and Discussion

Twenty spheres (40 for sapphire and K-Monel) from the central region of the die were studied for each compaction experiment. On each sphere, the faces had a wide variation of 'a' values sometimes deviating as much as 100 - 150% from the average value. Higher deviations were often observed for higher a/R values, when it was observed that the newly formed faces gave low values of a/R. In addition, the following experimental conditions may have contributed to this scatter:

- i) Hydrostatic conditions were not obtained in the uniaxial stressing of the compact. It was observed that faces that were approximately perpendicular to the direction of pressing gave larger values than those parallel to the direction of the pressing.
- ii) Difference in the hardness of different parts of the sphere.
- iii) Crystal orientation in the case of sapphire.

In the case of some spheres, an indentation rather than a flat face was occasionally observed and was probably due to the difference in hardness at the contact point. Very little fragmentation was observed during hot-pressing of sapphire spheres.

The theoretical models were tested by comparing the following results.

a) D vs a/R

Figure 20 shows the plot of percentage bulk density vs a/R for lead at room temperature. The experimental curve follows the general trend of the theoretical curves with the rising slope probably due to the coordination change observed. The sudden shift of the curve

Fig. 20 D vs a/R for Lead at Room Temperature and Sapphire at ● 1570°, ▲ 1600°, and ■ 1700°C.

Fig. 21.. D vs a/R for Lead at 50 and 100°C.

Fig. 22 D vs a/R for K-Monel.

at $a/R = 0.15$ corresponds to the sliding observed in the load vs compression curve. Figure 21 shows similar plots for the results obtained at 50 and 100°C. The theoretical plots are superimposed for comparison. The results for the sapphire spheres are shown in Fig. 20. Figure 22 also shows that the increase of densification of the K-Monel spheres with deformation follows the theoretical curves.

b) D vs Z

The deviation from the theoretical curves is attributed to the change of coordination number as the deformation proceeds. A change of Z was observed in all the experiments, and this is reflected in the general increase in the value of Z. Figure 23 shows the increase of Z as a function of $(D - D_0)$ for the lead spheres. The break in the line at $a/R = 0.15$ for the room temperature curve corresponds to the point of sudden shift in Fig. 20. The change of Z with the deformation is also evident from Fig. 24 which shows the change in the statistical distribution of the number of contact points for the spheres from the central portion of the die.

c) ϵ_b vs a/R

Figures 25 and 26 show the plots of percentage bulk strain vs a/R for both the lead and the sapphire spheres. The large values of the experimental strains are due to the contribution of particle rearrangement and coordination number change to the experimental strain, which has not been considered in the theoretical curves. The curve at 100°C shows that there was a larger contribution from the particle rearrangement.

Fig. 23 (D-D₀) vs Z for Lead.

Fig. 24 Change of Z with the Increasing Deformation for K-Monel Spheres.

Fig. 25 ϵ_b vs a/R for Lead at Room Temperature and Sapphire at $\blacktriangle$ 1570°, $\bullet$ 1600°, and $\blacksquare$ 1700°C.

Fig. 26 ϵ_b vs a/R for Lead at 50 and 100°C.

d) D vs ϵ_p

The straight line relationship between the percentage bulk density and the percentage bulk strain in the theory as well as experimental observations (shown in Figs. 27 and 28), verifies that the proposed models are operative during the deformation process. The different slope is again indicative of the change in the coordination number. Both the theoretical and experimental strains are negative, consequently, the data as plotted produced a positive slope and this is consistent with Eq. (26).

e) D vs a/R vs Z

In order to support further the argument that the deviation of the experimental data from the theoretical curves was primarily due to the gradual change of coordination with increased deformation, plots were made with the three variables, percentage bulk density, a/R and Z as shown in Figs. 29 and 30. The experimental data as superimposed on these plots appear to lie on the same surface formed by the cubic, orthorhombic and rhombohedral models. The deviation of the experimental plots is probably due to the change in Z. It is interesting to note that the curve for the b.c.c. model does not seem to lie on the same surface drawn through the curves for other models.

f) D vs $(a/R)^2$

Figure 31 is a plot of D vs $(a/R)^2$ for the lead and the sapphire spheres and Fig. 32 shows the same plot for the K-Monel spheres. It can be seen that a good fit is obtained between the theoretical predictions and the experimental observations. The change of the slope

Fig. 27 D vs ϵ_b for Lead at Room Temperature and Sapphire at $\blacksquare$ 1570°, $\blacktriangle$ 1600°, and $\bullet$ 1700°C.

Fig. 28 D vs ϵ_b for Lead at 50 and 100°C.

Fig. 29 D vs $\frac{a}{R}$ vs Z for Lead at Room Temperature and Sapphire at ▲ 1570°, ● 1600°, and ■ 1700°C.

Fig. 30 D vs a/R vs Z for Lead at 50 and 100°C.

at 100°C during the later stage may be due to the change in the nature of the contribution from the particle rearrangement to the overall densification process.

In order to normalise the differences in the initial packing densities for the individual experiments, plots between $(D - D_0)/D_0$ vs $(a/R)^2$ were plotted and are shown in Figs. 33 to 35 for lead. Again a good fit is obtained with the theoretical predictions.

It can be seen from Eq. (22), namely

$$D - D_0 = \frac{3}{2} D_0 (a/R)^2 \quad (22)$$

that it holds for a fixed coordination number. This does not include any contribution from the particle rearrangement to the densification process. It can be seen that according to this equation, a plot of D vs $(a/R)^2$ or between $(D - D_0)/D_0$ vs $(a/R)^2$ should give a slope of $3/2 D_0$ or $3/2$, respectively. But the calculated experimental slopes are much higher than the theoretically predicted slopes. Such a difference can be explained on the basis of the experimental observation that the plastic flow is accompanied by particle rearrangement and little fragmentation in the case of sapphire.

If ΔD is the increase in the density during hot-pressing then it must be composed of two parts i.e.

$$\Delta D = \Delta D_p + \Delta D_r$$

where ΔD_p = contribution from plastic flow

and ΔD_r = contribution from rearrangement

The contribution from the particle rearrangement will depend upon:

- i. the initial packing density and
- ii. the applied pressure.

Fig. 31 D vs $(\frac{a}{R})^2$ for Lead at Room Temperature, 50° and 100°C and Sapphire at ● 1570°, ▲ 1600°, and ■ 1700°C.

Fig. 32 D vs $(a/R)^2$ for K-Monel.

The complex nature of this contribution prevented any quantitative evaluation of this factor. But it can be seen that its inclusion in the plastic flow equation will increase the value of the constant. As observed by Duffield and Grootenhuis³⁷, apart from the initial repacking on immediate application of load, a transitional repacking persists causing a further volume change of about $1 \frac{1}{2} \%$ up to a pressure of 1000 psi. This is supported by the straight line relationship between D vs $(a/R)^2$ or $(D - D_0)/D_0$ vs $(a/R)^2$, which otherwise would not be so if the contribution from particle rearrangement was much different with increasing values of the load.

Fig. 33 $(D - D_0)/D_0$ vs $(a/R)^2$ for Lead at Room Temperature.

Fig. 34 $(D-D_0)/D_0$ vs $(a/R)^2$ for Lead at 50°C.

Fig. 35 $(D-D_0)/D_0$ vs $(a/R)^2$ for Lead at 100°C.

III

GEOMETRY OF DEFORMATION

IN THE DIE

A. INTRODUCTION

The simple and systematic packings of uniform spheres of equal size have been discussed in detail by Graton and Fraser³⁵ and were assumed in the theoretical models. The relative stability of the various packings depends upon the type of packing, the shape of the container, the mode of filling the die, and a few other factors like the die-wall effect etc. It can be shown that under the gravitational field of force alone, the conditions for stable equilibrium are satisfied by only the rhombohedral packing. The tetragonal and orthorhombic (from the rhombic base only) packings are less stable and have a tendency to change to the rhombohedral packing. The cubic and orthorhombic (from cubic base) packings are even more unstable.

1. The Die-Wall Effect

The theoretical densities obtained for the simple and systematic packings (shown in the Table IX, Appendix I) have been calculated on the assumption that the system is infinitely large in comparison with the diameter of the spheres. It is evident that the systematic packings cannot be attained throughout unless the die has identically the same shape and a very special exact relation to the size of the unit cell of the type of packing involved. This condition is extremely unlikely to be encountered in the die; hence, the systematic packing is rarely found throughout the die.

When the ratio of the container size to the particle diameter is less than 50 in case of the orthorhombic, or 100 in the case of the tetragonal and rhombohedral packings, the effect of the reduced density resulting from misalignments at the side walls is of the order of 1% or more³⁸. Also the shape of the container has a bearing on the type of packing e.g., a rectangular container will favour packings with square layer arrangement whereas a cylindrical container will have a simple rhombic arrangement in the first layer.

McGeary³⁹ has studied the effect of the container size on the packing density. His results are shown in Fig. 36. He found that at $D/d = 200$, the packing density reached a value of 62.5% of the theoretical density which agreed very closely with his experimental maximum values. It can be seen from Fig. 36 that the packing density is little affected by an increase in the container size beyond the ratio $D/d = 10$.

2. Mode of Filling the Die

The packing of spheres is greatly influenced by the way the die is filled. When a packing of a certain degree of stability is subjected to shaking and tapping, there is a tendency for it to change or translate into a packing of a higher degree of stability. If the lateral support is adequate, this tendency is reduced. Attainment of a more stable packing, which involves lateral spreading of the body, is prevented by adequate support from the sides. Therefore, the degree of stability of the packing is maintained inspite of the unstable configuration. Obviously, the degree of rigidity and adequacy of the lateral support will determine the extent to which the body will conform to a more stable packing

Fig. 36 Effect of Container Size on the Efficiency of Packing One-Size Spheres (After McGeary³⁹).

configuration.

The various modes of filling the die and the effect of vibration on the initial packing density have been studied extensively by Smith, Foote and Busang⁴⁰. They studied the change in the number of contact points of the spheres with the increase in the density of the compact resulting from shaking and tapping. They employed a corrosion technique which developed small rings on each sphere indicating the points of contact. They excluded the spheres touching the side-walls in their observations in order to minimise the die-wall effect. Their results are shown in Table II (from Jones⁴¹). It can be seen that there is a Gaussian distribution of the number of spheres with a given coordination number. Figure 37 shows such a graph and it can be seen that the maxima are obtained around the coordination number 8.

McGeary, using a transparent container and a low power stereoscopic microscope, showed that the vertical vibratory translation of a bed of spheres tends to produce the orthorhombic packing and to a small extent the tetragonal packing. He suggested that the rhombohedral packing cannot occur under such conditions because the vertical force will tend to change it to the tetragonal packing.

3. Haphazard Packings

There are various other schemes of arrangement which, while yet systematic and orderly, are more arbitrary and less simple than the ones considered under the simple and the systematic modes of packing. The most readily visualised examples are the arrangements in which the angle between the rows and spacing of the sphere-centers differ in some

TABLE II

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Correlation of Co-ordination Number and Density for Lead Shot, 3.78 mm Diameter

Treatment	Num- ber counted	Distribution of Numbers of contacts (z)										Mean co- ordina- tion number	Density	
		4	5	6	7	8	9	10	11	12	Ob- served		calcu- lated	
1. Poured into glass beaker	905	6	78	243	328	200	48	2	0	0	6.92	0.553	0.562	
2. Poured and shaken	906	3	54	173	309	233	118	14	2	0	7.34	0.560	0.575	
3. Poured and shaken	887	0	14	69	182	316	212	87	7	0	8.06	0.574	0.609	
4. Shaken to maximum density	1494	0	14	86	192	233	193	161	226	389	9.51	0.628	0.662	
5. Added in small quantities and tamped inter- mittently	1562	1	13	77	245	322	310	208	194	192	9.14	0.641	0.646	

The results are calculated from experimental data of Smith, Foote, and Busang. (After Jones⁴¹)

Fig. 37 Statistical Distribution of the Number of Spheres with the Coordination Number (Data from Jones⁴¹).

systematic manner and degree from those instances described earlier.

Other types of packings are possible in which no orderly or systematic repetitive arrangement can be discovered. All these packings can be grouped together under the general term chaotic or haphazard packings. Also in a given assembly of spheres packing is likely to vary from place to place. At certain places, one of the simple and systematic packings may be developed, at other places another type, while elsewhere various phases of haphazard arrangements can exist. Some of these arrangements may be entirely chaotic while others show some degree of orderliness.

The most probable result is, therefore, that layer-colonies of simple and systematic packings will be separated by zones of different random packings.

In the present study, it was intended to investigate the geometry of the deformed contact faces produced by deformation during hot-pressing. It is apparent that such a geometry will be different from the initial packing because of the continued repacking observed during compaction. But the overall geometry will depend upon the type of initial packings, systematic or haphazard.

B. EXPERIMENTAL TECHNIQUES AND RESULTS

The distribution of the contact faces on the sphere can be easily studied by measuring the angles between them using a goniometer. The apparatus shown in Fig. 17, with the specially designed goniometer to measure the rotation of the sphere with respect to the microscope alignment, was used for the purpose. It should be recalled that all

the theoretical models assume the spheres to be uniform and of equal size. Unless this condition is satisfied, some spheres packed in a certain manner may not produce contact points at low loads. This situation is quite different from measuring the coordination number using the corrosion technique. While the latter will show the total number of contact points, the former may not produce such a contact point by deformation. Such an effect was readily seen during the deformation of the lead spheres. It was found that the deviation from the perfect sphericity and the difference in the size of the lead spheres occasionally, caused the deformed sphere to show, lesser number of contact points.

In order to study the geometry of the deformation, K-Monel spheres with ball-bearing precision were used for the purpose. About 160 spheres, randomly chosen from the central region from four different experiments (40 from each) were used to study the angular relationship between the flat faces formed on each sphere. The spheres were first divided into groups according to their coordination numbers. Using angular relationships, stereographic plots were made in order to identify any symmetry between the faces. From the comparison of the distribution of the faces in each group, it is concluded that most of the spheres deform randomly i.e. the neighbouring spheres are randomly distributed, although the coordination number of the spheres may be the same. The evidence for this random geometry for the spheres having the same coordination number is shown schematically in Fig. 38. The knowledge of the size of the face and the fact that maximum contact points should lie in the middle layer, were used to determine the orientation of the spheres with respect to the direction of pressing (Fig. 38).

PROBABLE DIRECTION OF PRESSING

(A)

(B)

(C)

(D)

(Cont.)

(E)

(F)

(G)

(H)

(Cont.)

Fig. 38 Stereographic Representation of the Geometry of Deformation of Spheres in the Die.

It can be seen from Fig. 38 that not only the geometry of the flat faces is different from sphere to sphere but also it does not correspond to the hex. prism. model except in the case of one or two spheres. The fact that the coordination number varies from 4 to 12 inside the die (as also observed by others), suggests that the geometry of the deformation is random to a large extent.

The other observations made in this part of the investigation can be summarized as follows:

1. Individual colonies of the rhombohedral, the tetragonal and the orthorhombic packings were observed. In the central portion of the die, there was found to be a greater proportion of the rhombohedral and the tetragonal packings. The orthorhombic arrangement was found to be more pronounced near the walls of the die.

2. It was found that though the general packing arrangement should have been one of the above packings, sometimes one or two of the contact points were missing. This will be true if the spheres in the horizontal layer are not close enough to produce contact points at low loads. Also if the spheres are held at one place or the other in a disorderly manner, the coordination number will be affected.

3. Few of the spheres in contact or neighbouring to the die-wall were found to deform in a manner similar to the cubic model.

4. In some cases, especially those near the die-wall, dodecahedral type of deformation ($Z=4$) was observed.

5. In order to examine the shape of the deformed spheres at very high deformation, lead spheres were deformed under high pressures. On examination, it was found that the spheres had deformed more in the direction of the load and large variations in a/R values were observed. Figures 39 and 40 show the deformed spheres. It can be seen that the geometry of the deformation in Fig. 39 is similar to that of the hexagonal prismatic model and in Fig. 40 is similar to the rhombic dodecahedron model.

Also it was observed that at such high pressures, most of the spheres in the middle of the die had $Z=12$, corresponding to rhombic dodecahedron model.

6. In the case of packing the die without intermittent tapping and shaking, the deformation produced was more disorderly than when the die was filled in installments with some shaking and tapping.

7. The effect of the container size was determined by deforming brass spheres (1/8 in. diam) in a graphite die (1 in. diam). It was found that the deformation produced was very random in nature and very few spheres deformed according to the proposed models. The cubic and the orthorhombic packings were occasionally observed.

8. The fact that the initial bulk density of the compact varies from 60% to 62%, suggests that the overall packing arrangement i.e. the mode of deformation corresponds to the $Z=8$ coordination i.e. the hexagonal prismatic model.

Fig. 39 Photomicrograph of the Lead Sphere Deformed in Accordance with Hexagonal Prismatic Model.

Fig. 40 Photomicrograph of the Lead Sphere deformed in Accordance with Rhombic Dodecahedron Model.

IV

M E C H A N I C A L P R O P E R T I E S

A. INTRODUCTION

The theoretical models discussed previously (Chapter II) are general in the sense that the equations have been developed from geometrical considerations alone. The basic equations should hold for any material within the limits of the assumptions made in their derivation. The formation of a flat face on any sphere can take place only by two mechanisms:

1. Plastic deformation
2. Stress-enhanced diffusional creep.

The contribution from the diffusional process is calculable, if the diffusion coefficients under the existing stress are known. However, from the consideration of the time used for hot-pressing, no significant contribution from the diffusional mechanism towards the flat-face formation was expected. The alternative mechanism i.e. plastic deformation, involves the material properties such as viscosity, yield strength, etc.

An attempt has been made to include the yield strength into the basic density equation, so that the knowledge of the properties of any material can be used in predicting the densification behaviour during the hot-pressing process.

1. Deformation Under Static Conditions

The mechanism of deformation of two spheres in contact under a static load can be better understood by first considering the deformation

occurring between a hard hemispherical indenter and a flat face of a softer metal (the results are similar for the deformation occurring between a hard flat face and a hemispherical surface of a softer metal).

If the surfaces are pressed together with a load L , they will, at first, deform elastically according to Hertz's⁴² classical equations. The region of contact is a circle of radius a , where

$$a = 1.1 \left[\frac{Lr}{2} \left(\frac{1}{E_1} + \frac{1}{E_2} \right) \right]^{1/3}$$

where

r = radius of the hemisphere

E_1 = Young's modulus of the hemisphere

E_2 = Young's modulus of the flat surface.

It can be seen that the area of contact ($A = \pi a^2$) will increase as $L^{2/3}$ while the mean pressure over the circle of contact ($P_m = L/\pi a^2$) will vary as $L^{1/3}$. The deformations involved in this range are elastic and reversible. As the load is increased, a stage is reached at which the maximum shear stress in the softer material exceeds its elastic limit and plastic deformation just occurs. Timoshenko⁴³ has shown, using the Hertzian analysis, that this region is situated at a point about $0.5a$ below the centre of the circle of contact. At this stage, the mean pressure is given by

$$P_m = 1.1 Y$$

where Y is the yield stress or the elastic limit of the softer metal.

The plastic deformation is limited to a small region.

The material outside this region has not reached the condition for plasticity and its deformation is still essentially elastic.

As the load is further increased, the region of plasticity increases until the whole of the material around the region of contact flows plastically. Hencky⁴⁴ and Ishlinsky⁴⁵ have attempted to correlate the mean pressure with the elastic limit of the material when plastic yielding just occurs. The basic equation used in both studies was von Mises's criterion for plastic flow. According to this criterion, plastic flow occurs when

$$(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2 = 2Y^2$$

where σ_1 , σ_2 , σ_3 are the principal stresses in the solid and Y is the elastic limit of the material in pure tension (or frictionless compression) experiments. The analysis of both Hencky and Ishlinsky yields essentially the same result: i.e. to a first approximation, plastic yielding occurs when

$$P_m = c Y$$

where c has a value of approximately 3 (Hencky, $c = 2.82$ and Ishlinsky, $c = 2.6$).

If the load is further increased, the area over which plastic flow occurs increases and the mean yield pressure remains approximately constant at a value of $3Y$, provided, that;

a) the deformed area is not too large compared with the size of the specimen. There is a slight increase in P_m when the indentations are large compared to the size of the specimens, probably due to the increased confinement of the displaced materials⁴⁶.

b) the material does not work-harden as a result of plastic deformation i.e. the yield strength Y does not increase.

However, if the material work-hardens during the course of deformation, the effective value of Y may be considerably higher than the

value of Y at an earlier stage of deformation. A close approximation can be reached by using metals which have been so highly worked that further deformation produces no appreciable change in their elastic limit.

Many workers have confirmed the validity of the equation $P_m = 3Y$. Tabor⁴⁷ has given results for a hard steel sphere pressing against flat faces of other metals (Table III). The mean pressure P_m , which is also referred to as the "yield pressure" or the "pressure of fluidity" is also found to be independent of the indentation, and therefore the load. The term "pressure of fluidity" seems to have⁴⁸ been first introduced by Tresca to designate the true stress (on the enlarged area) necessary to induce a certain critical condition of plasticity. Works along these lines have also been carried out by Unwin⁴⁹, Coe⁵⁰, Sachs⁵¹, and by others. O'Neill and Greenwood⁵² summarize the results for various metals and showed that the condition $P_m = 3Y$ is satisfied (Table IV).

2. Effect of Work-Hardening

In the simple model of a hard spherical surface penetrating the surface of a softer metal $P_m = 3Y$ is constant only if the material does not work-harden. The yield strength or the elastic limit will increase, if the material work-hardens, on the formation of the indentation. Theoretical considerations and experimental measurements show that the elastic limit will not be constant around the indentation since the amount of deformation or strain will, in general, vary from point to point⁴⁷. A detailed experimental investigation by Tabor showed that, the elastic limit Y_e at the edge of the indentation can be used as an

TABLE III Mean Pressure Vs Elastic Limit

Work-hardened metal	Y Kg./mm ²	$P_m = \frac{L}{\pi d^2}$ Kg./mm ²	$c = P_m/Y$
Tellurium-Lead	2.1	6.1	2.9
Copper	31	88	2.8
Steel	65	190	2.8
(After Tabor ⁴⁷)			

TABLE IV Results Showing Validity of Equation $P_m = 3Y$

Metal	Y kg/mm ²			$3Y$	Mean Pressure of Fluidity kg/mm ²
	Ludwick ⁵³	Korber ⁵⁴ & others	Mean		
Lead	1.95	-	1.95	5.9	6.9
Tin	4.55	-	4.55	13.6	12.1
Cadmium	7.75	-	7.75	24	31
Aluminium	14.3	13.7	14	42	41
Zinc	11.9	-	11.9	36	38/71
Copper	33.5	32.8	33	99	108
Iron(Krupp soft)	42.5	42.3	42	126	132
Nickel	66.5	75.4	71	213	237

(After O'Neill and Greenwood⁵²)

average or representative value of the elastic limit which is related to the mean pressure P_m by a relation

$$P_m = c' Y_e$$

where c' has a value lying between 2.7 and 3.

It can be seen that the elastic limit is independent of the size of the indentation and the area over which plastic flow occurs is directly proportional to the load for fully worked metals. It can be shown that the area of contact for a work-hardening material is proportional to $L^{0.8}$ and for partially work-hardened e.g., extruded brass or mild steel, the area of contact is proportional to $L^{0.92}$ (Bowden and Tabor⁵⁵).

O'Neill⁵⁶ has obtained similar results for two hemispheres of the same metal pressing against one another.

B. THEORY

The foregoing considerations show that the contact areas grow to stable sizes, fixed by a constant stress, ($P_m = 3Y$). This fact will be utilized here to extend Eq. (22) to a more general form and thus calculate the contribution of the plastic flow mechanism to the densification process.

Assuming that the contact areas grow to stable sizes under a fixed stress, Coble³¹ has obtained a relationship between a/R , coordination number and the applied stress. In his paper, Coble assumed that if

σ_p = the fixed stress

Z = the coordination number

σ = the applied stress (assumed to be hydrostatic)

then the load transferred to each particle is given by

$$\sigma_p Z \pi a^2 \quad \text{and}$$

the load applied to each particle is given by

$$\sigma 4 \pi R^2.$$

He equated these two loads getting

$$\sigma_p Z \pi a^2 = \sigma 4 \pi R^2$$

or

$$\frac{a^2}{R^2} = \frac{4}{Z \sigma_p} \sigma \quad (27)$$

Equating $\sigma_p Z \pi a^2$ to $\sigma 4 \pi R^2$ is based on the assumption that the forces operating on a volume element of a medium under hydrostatic conditions can be unequal but that σ must be the pressure (Coble⁵⁷). Furthermore, he believes that the error introduced by assuming such conditions is probably within a factor of 2.

It can be seen that the surface area of the sphere ($4\pi R^2$) will no longer be the same after some deformation has taken place. The surface area of the sphere can be calculated at any stage of deformation for the specific models described in Chapter II. It can be shown that for small deformations ($a/R \leq 0.3$), the surface area of the sphere essentially remains constant.

The constant or the fixed stress σ_p (same as P_m) in Eq. (27), can be equated to three times the yield strength of the material, if it does not work-harden, or three times the representative value of the yield

strength of the material, if it does not work-harden, or three times the representative value of the yield strength, if the material work-hardens.

Substituting $\sigma_p = 3Y$ in Eq. (27) gives

$$\frac{a^2}{R^2} = \frac{4}{3YZ} \sigma \quad (28)$$

Introducing this value of a^2/R^2 into Eq. (22) gives

$$D - D_o = \frac{3}{2} D_o \frac{4}{3YZ} \sigma$$

or

$$D = D_o + \frac{2D_o}{YZ} \sigma \quad (29)$$

where

D = the bulk density at any deformation

D_o = the initial bulk density at $a/R = 0$

Y = the representative value of the yield strength
of the material

Z = the coordination number of the particles.

σ = the applied hydrostatic stress.

C. EXPERIMENTAL VERIFICATION

Equations (28) and (29) are based on the assumptions

1. the applied stress is hydrostatic in nature
2. Z remains constant

There is considerable experimental evidence which shows that the hydrostatic conditions are not obtained during the conventional hot-pressing process. Under these conditions, as expected, there was found to be a large variation in the contact areas on each particle. An average value of the contact areas has been assumed to be representative

Fig. 41 $(\frac{a}{R})^2$ vs L for Lead at Room Temperature.

Fig. 42 $(a/R)^2$ vs L for Lead at 50°C.

Fig. 43 $(a/R)^2$ vs L for Lead at 100°C.

Fig. 44 $(a/R)^2$ vs σ for K-Monel.

of the experiment.

Figures 41-44 show the plot of $(a/R)^2$ vs σ (or applied load) for lead at room temperature, 50° and 100°C, and K-Monel at 800, 900, and 1000°C. Similarly, Figs. 45 to 47 show the plot of $(D - D_0)/D_0$ vs σ (or the applied load) for the lead and K-Monel spheres. According to Eqs. (28) and (29), these relationships should be linear provided Z remains constant. It can be seen that the experimental results give a good fit with the theoretical predictions. The deviation from the linearity at low load values may be due to the change of Z during the initial stages of hot-pressing. Such a conclusion is consistent with the observations of Felten²⁵, Shapiro²⁸⁻³⁰, and Duffield and Grootenhuis³⁷, namely that the contribution from particle reorientation is mostly during the initial application of loads.

It is theoretically possible to calculate the yield strength of the material from the modified density equation - Eq. (29), provided Z remains constant. However, experimentally it was observed that Z changes continuously with the progressive deformation during hot-pressing which makes it difficult to calculate the yield strength from the data. But the change in Z , as experimentally observed, was only about 8 to 10%. Under these circumstances, the values of the yield strength were computed from the slopes of the Figs. 41 to 47 using the mean value of Z . The calculated yield strength values for lead, K-Monel and sapphire are given in Table V.

It can be seen from Table V that the calculated yield strength values from the plots $(a/R)^2$ vs σ (or L) and $\frac{D - D_0}{D_0}$ (or D) vs L (or σ)

Fig. 45 $(D - D_o)/D_o$ vs L for Lead at Room Temperature.

Fig. 46 $(D-D_0)/D_0$ vs L for Lead at 50 and 100°C

Fig. 47 D vs σ For K-Monel.

TABLE V Yield Strength Values from the Slopes of Plots in Figs. 41 to 47

Relationship	Yield Strength (psi)								
	Lead (2%Sb)			K-Monel			Sapphire		
	Room Temp.	50°C	100°C	800°C	900°C	1000°C	1570°C	1600°C	1700°C
(D-Do) _{Do} vs L or D vs σ	3842	4070	2095	8800	5320	4245	16,570	15,520	9635
σ or L vs $(a/R)^2$	7210	7470	4010	14,280	10,090	6,910	35,600	35,420	22,210

differ nearly by a factor of 2 in all cases. The values obtained from the plots of σ vs $(a/R)^2$ appear to be about two times the value of the yield strength reported by Kronberg⁶² for sapphire in this temperature range. It is believed that Eq. (27) is accurate possibly within a factor of 2 (as also stated by Coble⁵⁷); therefore the calculations based on this equation give larger yield strength values. That means the results obtained from the plots $(D - \frac{D_0}{2})$ vs L would have to be correspondingly reduced. But in the later case, the values obtained should be increased by a factor of about 1.5 because of the contribution from particle rearrangement contained in $(D - D_0)/D_0$ values. It is also possible that the difference between the two results is affected by i) $(D - D_0)/D_0$ values which represent the bulk of the compact and ii) $(a/R)^2$ values which represent the mean of 20 or 40 spheres only.

In order to compare the yield strength value obtained from these calculations for the lead spheres at room temperature with that of the actual yield strength, microhardness measurements were made on lead spheres. Following Tabor's observations, the yield strength of the material is approximately 2.9 to 3 times the Vicker's hardness number (same as Microhardness number). Although no conclusions can be drawn from this analysis without doing an extensive study, the yield strength value determined from the microhardness tests (about 6000 psi at 23°C) for the lead (2% Sb) spheres lies between those values calculated from the experimental plots (Table V).

It is interesting to compare Eq. (29) with the other empirical and semi-empirical relationships given in the literature. These are

summarized in Table VI. Equations (i) to (vi) seem to take into account only one mechanism of compaction i.e. plastic deformation and have been supported by observations on metal powders. Equation (vii) comprises of two parts

- a) Filling of large size holes by particle sliding
- b) Filling of holes which are smaller than the particle size probably by plastic flow or by fragmentation.

All these equations seem to be applicable for the entire range of densification. But the Eq. (29) obtained in the present investigation is applicable for limited range of densification process i.e. early stages of compaction; and a deviation from linearity is expected at the later stages of densification with the higher values of a/R (≥ 0.3) and also from other mechanism of densification, such as stress enhanced diffusion, which may be rate determining at a later stage. It is preferred, however, to leave any formal comparison of the relationship with the others given in the literature, as the results herein are not sufficiently detailed to warrant critical assessment of the density-pressure relationship.

TABLE VI Equations Relating Density (or Volume)
of the Compact with the Applied Pressure

No.	Equation	Reference
(i)	$w = k_1 - k_2 \log P$	1
(ii)	$\frac{dP}{dw} = -kP$	2
(iii)	$Pw^k = \text{constant}$	3
(iv)	$P = k \log \frac{1 - D_0}{1 - D}$	6,8
(v)	$D - D_0 = kP^{1/3}$	7
(vi)	$D - D_0 = kP^2$	58
(vii)	$V = a_1 \exp - (k_1/P)$ $+ a_2 \exp - (k_2/P)$	59
(viii)	$D = D_0 + kP$	Present work

where

w = the apparent volume

P = the applied pressure

D = the density at pressure P

D_0 = the density at zero pressure

V = fractional volume

k_1, k_2, k = constants

a_1, a_2 = fractional coefficients.

MECHANISM OF — DEFORMATION OF
SINGLE CRYSTAL SAPPHIRE SPHERES BY
HOT — PRESSING

A. INTRODUCTION

In the present study it has been observed that permanent strain can be produced during the hot-pressing of single crystal sapphire spheres in the temperature range 1570°-1700°C. The densification process has been interpreted in terms of the particle movements giving rise to repacking, and the plastic flow mechanism. Although the contribution from the particle rearrangement cannot be quantitatively determined, the overall densification behaviour of the sapphire spheres is very similar to that of the lead spheres, where both the particle rearrangement and the flat face formation by plastic flow contributed to the densification process. The contribution from plastic flow is calculable from the proposed geometric models, knowing the average coordination number Z and the average face radius ' a '.

Coble³¹ has studied the flat face formation between two single crystal sapphire spheres (1 mm diam) in contact under a static load at 1530°C. He speculated that the deformation might be due to both basal and non-basal slip, twinning or some other complex mechanism. The nature of the deformation of the sapphire spheres has been investigated qualitatively in the present work by examining the deformed spheres under optical and electron microscopes and relating the slip lines to the crystal orientation.

In this respect, it would be desirable to make a survey of the present day knowledge of the deformation characteristics of single crystal sapphire.

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McCandless and Yenni were the first to observe basal slip in single crystal sapphire rods deformed by bending around an axis normal to the hexagonal axis at 1300-1400°C. Wachtman and Maxwell⁶¹ subsequently studied the tensile creep of single crystal rods of Linde synthetic sapphire. They showed that sapphire could be deformed plastically above 900°C. The onset of creep near this temperature was found to be preceded by a pronounced incubation period. With increasing temperature and stress, however, the length of the delay period was found to decrease sharply. From the orientation study, Wachtman and Maxwell determined that the slip system (0001) $[11\bar{2}0]$ was operative. Kronberg⁶² confirmed both this slip plane and the slip direction in tensile deformation of sapphire between 1200° and 1700°C. He also studied the deformation as a function of the strain rate between 10^{-3} to 10^{-1} in./in./min. His work showed the existence of a relatively sharp transition between complete brittle fracture and massive plastic flow, the specific transition temperature being sensitive to the strain rate. For the range of the rates used, the transition temperature increased from approximately 1270 to 1520°C. He also found that with only a few degrees difference in the transition temperature, it was possible to obtain either no plastic flow on the low temperature side or a relatively large amount of extension (about 100%) on the high temperature side. He also observed pronounced yield-point drops i.e. the subsequent flow stresses were found to be approximately half the stresses required to initiate macroscopic flow.

Kronberg⁶³ had earlier presented in the form of a model the justification for the experimentally observed elements of the slip system (0001) $[11\bar{2}0]$ and twinning in the sapphire single crystals. It is interesting to note that $[11\bar{2}0]$ direction in sapphire is at 30° to the row of closest packed oxygen ions as different from $[11\bar{2}0]$ direction in zinc parallel to the closest packed row of atoms. Figure 48 shows the two cases for a comparative study.

Nye⁶⁴ has also observed basal slip in corundum crystals bent into the form of a U-shaped loop. The Prismatic slip system $\{11\bar{2}0\} [10\bar{1}0]$ has been observed by Klassen-Neklyundova⁶⁵ during deformation studies on corundum crystals above 2000°C . The relation between the two slip systems observed in corundum is illustrated schematically in Fig. 49. Scheuplein and Gibbs⁶⁶ have studied the basal and prismatic dislocations using an etch pit technique. They found that basal slip could occur more readily than prismatic. The latter was found to be induced more readily at temperatures near 2000°C in crystals oriented unfavourably for basal glide. In their later paper⁶⁷, they showed that both dislocations could be activated concurrently and that important interactions between them could occur.

Lommel and Kronberg⁶⁸ observed dislocations in sapphire and ruby single crystals by means of a high resolution Berg-Barrett x-ray technique. Their thermal etching data were found to be in agreement with the data of Scheuplein and Gibbs. Stephens and Alford⁶⁹ used the chemical polishing and etching techniques to reveal the dislocation structures of sapphire and ruby crystals grown by the flame-fusion techniques. They observed both the basal slip and the prismatic slip around 1800°C .

Fig. 48 Representation of $[11\bar{2}0]$ Direction in Case of Zinc and Sapphire Crystals (After Kronberg⁶³).

Fig. 49 Schematic Representation of Basal and Prismatic Slip Systems in Sapphire (After Scheuplein and Gibbs⁶⁶).

Also basal twinning was observed in all flux grown crystals.

B. EXPERIMENTAL TECHNIQUES, RESULTS, AND DISCUSSION

1. Optical Microscopy

The contact faces formed on deformation of the single crystal sapphire spheres together with the undeformed surfaces of the deformed spheres were examined for any slip lines, deformation or fracture markings. It was noted that the deformation markings shown in Fig. 50 were not observed on all the faces. The sets of markings suggest the presence of more than one slip system under the present deformation conditions.

Figures 50(c) and 50(d) show that extensive slip has taken place in the free surface near the deformed face. The two sets of slip lines shown in Fig. 50(c) were identified from the orientation of the crystal determined by the back-reflection Laue technique using the standard projection for the corundum crystal⁷⁰. Similar techniques helped in analyzing one or both of these slip planes responsible for deformation of sapphire.

Figure 50(e) shows that inhomogeneous deformation has taken place (left of the flat face). The circular marking on the right of the contact face seems to be present in the undeformed spheres produced by grinding.

2. Electron Microscopy

It was found that the optical microscopy helped very little in studying the details of the flat face formed. For this reason, it was decided to study the deformation characteristics of the single crystal

(a) X600

(b) X600

(Cont.)

(c) X900

(d) X400

(Cont.)

(e) X600

Fig. 50 Photomicrographs of the Typical Contact Faces.

spheres using replica techniques. Because of the very small size of the surface to be studied, replicas were made with about one third to one half of the surface of the sphere making an impression in the cellulose acetate film. The replica was expected to contain at least one flat face.

Figures 51 to 53 provide enough evidence for the presence of the plastic flow mode of deformation during hot-pressing of the sapphire single crystal spheres. Figure 51(a) shows the presence of slip lines possibly representing the basal slip. Presence of wavy slip in Figs. 51(b) and 51(c) strongly supports the conclusion that complex slip has taken place.

Figures 52 and 53 show a stepped structure similar to one observed in α -brass⁷¹ and copper⁷² crystals. Following the interpretation used for these materials, cross slip is thought to be the operating mechanism. Scheuplein and Gibbs⁶⁷ have advanced a mechanism for cross-slip in corundum based on the earlier model of Kronberg⁶³ describing the interaction of basal and prismatic slips. They postulate that the screw segments of prismatic dislocations can decompose into basal dislocations. Further decomposition of basal dislocation into quarter partials and stacking faults restricts the motion of prismatic dislocation entering the basal plane.

3. Fractured Surfaces

At some places on the free surface of the sapphire sphere, chips were seen to be flaked-off. Figure 54 shows such a chipped-off surface on a sapphire sphere. The markings show that fracture has taken place. No attempt was made to study the nature or the characteristics of the fracture surface any further.

(a) X5000

(b) X5000

(Cont.)

(c) X 10,000

Fig. 51 Electronmicrographs Showing Evidence of Simple and Wavy Slip.

Fig. 52 Cross Slip in Sapphire at 1570°C. X12,000

Fig. 53 Cross Slip in Sapphire at 1700°C. X5000

Fig. 54 Fractured Surface on the Sapphire Sphere. X400

DISCUSSION

The theoretical models have been derived on the assumption that the pressure is applied equally from all directions. Though it is well established that such an assumption is hardly met in practice during hot-pressing, it seems to simplify the quantitative analysis of the problem. Thus before any discussion of the present results is attempted, it is desirable to see how far this assumption is valid under the present experimental conditions.

A. PRESSURE DISTRIBUTION IN THE DIE

It is a well known fact that the distribution of density within cold-pressed powder compacts is influenced by the type of powder, shape of the die, lubrication during the process, etc. The analysis of the density and the strain distribution in the compact is very helpful in obtaining information about the stress distribution within a powder compact.

Kamm, Steinberg and Wulff⁷³ used a deformable grid within a powder compact to study the density distribution in the compact. From their measurements, they found that the densest part of a compact pressed from one side is at the top outer circumference and the least dense region at the bottom circumference near the stationary plunger. The density gradients are attributed to the die-wall friction. They found that as the ratio of the diameter to the height of the compact is increased the die-walls play a correspondingly smaller role.

Duwez and Zwell⁷⁴ have also studied the pressure distribution based on density measurements. They represent the distribution of pressure

within the compact by tracing lines of equal pressure in a longitudinal direction. Figure 55 shows the approximate location of the lines of equal pressure in a copper compact of thickness to diameter ratio of 0.42. The variation of die-wall pressure with height observed by them is substantiated by the results of many other workers. Results on the studies of the pressure distribution in the die on these lines lead to a most important practical consequence of the die-wall friction i.e. uniform distributions of density within a powder compact are the function of the height of the compact.

Fig. 55 Approximate Lines of Equal Pressure in Copper Compact. Pressure 100,000 psi (After Duwez and Zwell⁷⁴).

The unequal distribution of pressure throughout the compact arises from the wall frictional force which opposes transmission of the applied pressure in its neighbourhood. Such pressure variations at local regions within the compact can lead to relative sliding of particles. Thus considerable movements of particles can be expected to take place near the walls of the die. This relative movement, particularly at high pressures, can cause distortion of the particles, produce fragmentation by shearing forces in these areas, and may cause dislodgement of surface films in metals.

It can be concluded from the works of several investigators that the maximum uniform distribution of pressure within a powder compact can be obtained by fixing the ratio of the thickness to the diameter of the die to about 0.5. In the present work this ratio was respectively, 0.25 for lead, 0.40 for K-Monel and 0.30 for sapphire.

It can be seen that above conclusions are based on measurements of the density of very small portions of the compact. These may not necessarily apply to the distribution of pressure at each contact point of the powder particle. It is observed in the present work that there is a large variation in contact face radii for each particle. By deforming lead spheres to larger values of strain it was observed that the the deformed faces that were normal (or within 30°) to the direction of pressing were about 2.2 times larger than those parallel to the direction of pressing. Since the deformation takes place under a fixed stress (as discussed in Chapter IV), the variation in 'a' values suggests that the forces acting at each contact point are unequal contrary to the assumption made in the theoretical models. This observation will

be used to modify the theoretical models.

B. CORRECTED THEORETICAL MODELS BASED ON EXPERIMENTAL

RESULTS

Experimentally it was observed that

$$a_2 = 2.2 a_1 \text{ (approximately)}$$

where

a_2 = radius of the faces normal to the direction of pressing

a_1 = radius of the faces parallel to the direction of pressing.

Using this observation, theoretical models have been modified to take into account the algebraic mean radius of the contact faces.

1. Corrected Cubic Model

In this case there will be 4 a_1 's and 2 a_2 's. Therefore, the average or the mean radius a_m will be given by

$$a_m = (4a_1 + 2a_2)/6$$

Substituting $a_2 = 2.2 a_1$, we get $a_m = 1.4 a_1$,

$$\text{or } a_1 = 0.71 a_m$$

$$\text{and } a_2 = 1.57 a_m$$

Furthermore, the constant volume equation can be rewritten as

$$\frac{4}{3} \pi R^3 - \frac{4\pi h_1^2}{3} (3R - h_1) - \frac{2\pi h_2^2}{3} (3R - h_2) = 1 \text{ (e.g.)}$$

$$\begin{aligned} \text{or } \frac{4\pi}{3} (2R^2 + a_1^2) (R^2 - a_1^2)^{1/2} + \frac{2\pi}{3} (2R^2 + a_2^2) (R^2 - a_2^2)^{1/2} \\ - \frac{8\pi R^3}{3} = 1 \end{aligned} \quad (30)$$

Substituting the values of a_1 and a_2 in terms of a_m , the radius of the particle at any deformation can be obtained from Eq. (30) for the different values of a_m . The unit cell in this case will be a rectangular parallelepiped. The volume of the unit cell will be given by

$$\begin{aligned} V_c &= 8 y_1^2 y_2 \\ &= 8 (R^2 - a_1^2) (R^2 - a_2^2)^{1/2} \end{aligned}$$

and the bulk density will be given by

$$D = 1/V_c \cdot 100\%$$

Table VII summarises the values of R , V_c , D , and $(D - D_o)$ for the values of a_m up to $0.3R$. It can be calculated from the data in Table VII that

$$D - D_o = 1.78 D_o (a_m/R)^2 \quad (31)$$

2. Corrected Hexagonal Prismatic Model

In this case there will be $6a_1$'s and $2a_2$'s. Therefore, the average or the mean radius will be given by

$$a_m = (6a_1 + 2a_2)/8$$

Substituting $a_2 = 2.2 a_1$, we get $a_m = 1.3 a_1$

$$\text{or} \quad a_1 = 0.77 a_m$$

$$\text{and} \quad a_2 = 1.69 a_m$$

The constant volume equation, in this case, becomes

$$\begin{aligned} &\frac{4}{3}\pi R^3 - \frac{6\pi h_1^2}{3} (3R - h_1) - \frac{2\pi h_2^2}{3} (3R - h_2) = 1 \text{ (e.g.)} \\ \text{or} \quad &2\pi(2R^2 + a_1^2)(R^2 - a_1^2)^{1/2} + \frac{2\pi}{3}(2R^2 + a_2^2)(R^2 - a_2^2) - 4\pi R^3 = 1 \quad (32) \end{aligned}$$

Substituting the values of a_1 and a_2 in terms of a_m , the radius of the particle at any deformation can be calculated from Eq. (32) for different values of a_m . The unit cell in this case will be again a hexagonal prism with different height. The volume of the unit cell will be given by

$$\begin{aligned} V_c &= 4\sqrt{3} y_1^2 y_2 \\ &= 4\sqrt{3} (R^2 - a_1^2) (R^2 - a_2^2)^{1/2} \end{aligned}$$

and the bulk density will be given by

$$D = 1/V_c \cdot 100\%$$

Table VII summarizes the values of R , V_c , D , and $(D - D_0)$ for the values of a_m up to $0.3R$. The following modified density equation

$$D - D_0 = 2.03 D_0 (a_m/R)^2 \quad (33)$$

can also be calculated from the data given in Table VII.

3. Corrected D- σ Equation

Equation (28) will also be affected because of the two values of a 's. In this case we can write the new equality equation for the two loads as:

a) Corrected Cubic Model

$$3 Y (4\pi a_1^2 + 2\pi a_2^2) = 4\pi R^2$$

or in terms of a_m , this can be simplified to

$$\frac{a_m^2}{R^2} = \frac{\sigma}{5.21 Y} = \frac{\sigma}{0.87 Z Y} \quad (34)$$

b) Corrected Hexagonal Prismatic Model

$$3 Y (6\pi a_1^2 + 2\pi a_2^2) = 4\pi R^2$$

or in terms of a_m , this can be simplified to

$$\frac{a_m^2}{R^2} = \frac{\sigma}{6.96 Y} = \frac{\sigma}{0.87 Z Y} \quad (35)$$

4. Comparison with the Results

These modified equations for the cubic and the hexagonal prismatic models were used to replot the data given in Figs. 20 to 22 and are shown in Figs. 56 and 57. The original theoretical plots are also shown for comparison. It can be seen that the modified theoretical curve for the hexagonal prismatic model gives a better fit with the experimental data. In addition, it can be seen that the value of the constant $1.5 D_o$ in the Eq. (22) changes to $1.78 D_o$ for the cubic and $2.03 D_o$ for the hexagonal prismatic models. A comparison of these corrected theoretical slopes with the experimental slopes of the curves $(D - D_o)/D_o$ vs $(a/R)^2$ or D vs $(a/R)^2$ shows that the corrected values of the theoretical slopes are much closer to the experimental values. Table VIII summarizes these results.

Also the Eq. (33) changes to

$$D = D_o + \frac{2.33}{ZY} D_o \quad (\text{for } Z = 8) \quad (36)$$

and this corrected equation will give different values of the predicted yield strength (about 16% larger).

It can be concluded from the good agreement between the corrected theoretical plots and the experimental results that the contribution to the densification may be chiefly from the plastic flow mechanism in the later stages of the experimental observations. As the maximum initial packing density of the spheres was obtained before any application of the

Fig. 56 D vs $\frac{a}{R}$ for Lead (Comparison with the Corrected Theoretical Models).

Fig. 57 D vs a/R for K-Monel (Comparison with the Corrected Theoretical Models).

TABLE VII Corrected Cubic and Hexagonal Prismatic Models and Their Characteristics

Model	a_m/R	R (arb. Units)	V_c	D (%)	$D - D_o$ (%)
Corrected	0.1	0.6203	$7.843R^3$	53.30	0.94
Cubic	0.2	0.6213	$7.365R^3$	56.02	3.66
Model	0.3	0.6248	$6.556R^3$	60.81	8.45

Corrected	0.1	0.6204	$6.787R^3$	61.70	1.24
Hex. Prism.	0.2	0.6219	$6.363R^3$	56.36	4.90
Model	0.3	0.6267	$5.654R^3$	71.66	11.20

TABLE VIII Theoretical and Experimental Slopes of Plots

$$\frac{(D - D_o)}{D_o} \text{ vs } (a/R)^2 \text{ and } D \text{ vs } (a/R)^2$$

Material	Temp °C	$\frac{(D - D_o)}{D_o} \text{ vs } (a/R)^2$		$D \text{ vs } (a/R)^2$	
		Theoretical	Experimental	Theoretical	Experimental
Lead	Room Temp.	3/2	2.75, 2.20	3 $D_o/2$	2.15 D_o , 2.69 D_o
	50	modified	2.57	modified	2.92 D_o
	100	to	4.8, 2.3	to	3.68 D_o , 2.28 D_o
K-Monel		2.03 for		2.03 D_o	
	800	Hex. Prism.		for	2.72 D_o
	900			Hex. Prism.	2.73 D_o
	1000	Model		Model	1.99 D_o

load the contribution from particle rearrangement was expected to be small during the later stages of the experiment.

C. HOT-PRESSING-MECHANISMS OF DENSIFICATION

1. General

Many theories have been put forward in recent years to describe the densification mechanism during hot-pressing process. Most of these theories are supported by works on ceramic oxides. The densification process as a whole has been suggested by the recent investigators to be a complex mechanism. It has been shown that the entire process of densification cannot be represented by a single equation. While fragmentation and particle rearrangement are considered to be responsible for the densification in the initial stages^{16,25-30,33}; the final stages of hot-pressing have been concluded to be controlled by the stress enhanced diffusional creep³¹⁻³³. The contributions from the different mechanisms at different stages of densification have been proposed (hypothetically) by Spriggs and Atteraa⁷⁵ (shown in Fig. 58). According to them, the predominant mechanisms of densification during the very early stages of the hot-pressing process are particle rearrangement and plastic flow. And the maximum contribution from these two mechanisms occurs within the first 30 seconds of the hot-pressing process. Obviously, these maxima or peak contributions either from the particle rearrangement or from the plastic flow will vary with different materials and the initial packing configuration. Whether the densification will take place by fragmentation, particle rearrangement, plastic flow, or stress-enhanced diffusional creep will depend upon the type of material,

Fig. 58 Densification Rate as A Function of Time Calculated From the Densification of MgO shown at the Upper Right Hand Corner.. (After Spriggs and Atteraa⁷⁵).

the temperature and the stress level used during the hot-pressing experiments. Metal compacts will have a much greater contribution from plastic flow than ceramic oxides. Also plastic flow is enhanced by high temperatures and high stresses.

It is evident from the above discussion that densification during hot-pressing is a net result of several mechanisms. The contribution from one mechanism alone, i.e. particle rearrangement, plastic flow or stress-enhanced diffusional creep is thus not easily measured. One of the easiest methods is to consider models based on some simple and systematic packings of one-sized spherical particles. The present work essentially describes the densification of such compacts by the plastic flow mechanism alone. Because of the difficulty in starting the compaction experiments with one of the ideal packings, particle rearrangement is also active although the particle rearrangement was kept to a minimum by shaking and tapping the spheres as they were packed in the die, the contribution from particle rearrangement with increasing deformation has been well demonstrated in the present work. The good agreement between the theoretical predictions (based on ideal packing) and the experimental results (random packing) leads to the conclusion that the densification is taking place chiefly by plastic flow in the early stages of the hot-pressing process. In the intermediate and final stages, calculations of the contribution of plastic flow to the densification process are complicated by the simultaneous occurrence of stress-enhanced diffusional creep; therefore, was not attempted in the present work.

2. Hot-Pressing of Ceramic Oxides — Contribution from Plastic Flow

von Mises⁷⁶ and Taylor⁷⁷ have shown that five independent slip systems are necessary for plastic flow within polycrystalline materials. von Mises's criterion also assumes slip to be homogeneous and capable of occurring on all the possible slip systems in any given volume of the material. However, in materials where available independent slip systems are limited, deformation by twinning, bending and kinking can reduce the number of distinct systems required.

In case of ceramic oxide materials like MgO, only two independent slip systems $\{110\} \langle \bar{1}10 \rangle$ are available at low temperatures⁷⁸. But at temperatures above 600°C, it has been shown by Hulse, Copley, and Pask⁷⁹ that three other slip systems of the form $\{100\} \langle 110 \rangle$ become operative in compression. Though the availability of these five independent slip systems satisfies the criterion for plastic flow, slip in single crystals of MgO has been found to be inhomogeneous up to about 1700°C (in tension experiments⁸⁰).

In sapphire single crystals, only basal slip has been observed by other investigators up to a temperature of 1800°C. Evidence for the presence of both basal slip and prismatic slip and possibly twinning has been demonstrated in the present investigation.

It is important to keep in mind the chief difference between the plastic flow as it applies to hot-pressing and plastic flow during tensile or compressive deformation of single crystals. In case of hot-pressing, the compact is composed of single crystal spheres with an initial bulk density of about 58%. Thus the spheres can relieve

themselves of high stresses by particle sliding or rotation of the spheres. Also the available free surface allows the spheres to deform with little constraint from the neighbouring spheres. However, as the densification proceeds, the spheres will be less liable to orient and slide because of the constraint imposed by denser packing geometry.

Thus in materials, particularly ceramic oxides, where the number of available active slip systems are few at a particular temperature, contribution from dislocation glide will be limited. This is believed to be the only contribution from plastic flow in such materials. Further densification should take place by stress-enhanced diffusional creep. That the contribution from plastic flow in ceramic oxides is limited by such a factor, has been substantiated by the work of Coble and Ellis³¹. They studied the increase in the contact area between two single crystal sapphire spheres (1 mm diam) as a function of time at 1530°C. They found that at each load, the value of radius of the contact face formed remained constant after about 10 minutes.

The spheres in the die will have random crystal orientation with respect to their neighbours. The effective stresses at the contact points which are quite large compared to the flow stress can cause fracture, brittle or ductile, at unfavourably oriented places. It has been reported earlier that a small amount of fragmentation was also observed during the hot-pressing of single crystal sapphire spheres. Thus the densification of sapphire spheres is mainly attributed to plastic flow and particle rearrangement.

3. Creep of Materials (Under Hot-Pressing Conditions)

The mode of deformation of materials during hot-pressing can be easily related with the mechanisms of high temperature creep deformation of materials under constant compressive stress. Most of the experiments on the creep of materials are conducted under simple tension and as a result little information is available on the creep of materials under simple shear, torsion or compression. It has been shown from the limited information available that the creep rates may be sensitive to the nature of stress applied. It has been found that the creep rate in tension is greater than that in compression for stainless steel and lead⁸¹, and nickel⁸², whereas the reverse is true for the Woods metal⁸¹.

The hot-pressing experiments are mostly carried out at temperatures above $0.5T_m$. The total creep strain produced will be composed of the strains produced in the individual stages i.e., the initial extension, transient flow and steady state creep. The initial instantaneous strain is obtained on immediate loading. The high temperature creep (above $0.5T_m$) results on materials such as stainless steel⁸³, Fe-Ni-Cr-Mn alloy⁸⁴, copper⁸⁵, brass⁸⁶, lead⁸⁷ etc. have shown that the instantaneous strain is due to plastic deformation. It can be expected that the large contact stresses during the hot-pressing of the spheres, for short times, would account for most of the deformation taking place.

The transient creep is characterized by the decreasing rate of creep strain with time. In this stage, the theory of work-hardening proposed by Mott⁸⁸ has been found to be in agreement with

results of many workers. The activation energy for this stage was found to be 7.7 kcal/g atom for lead by Feltham⁸⁷ compared to the activation energy for volume diffusion in lead about 25.7 - 27 kcal/g atom. It has been observed that the strain rate is accelerated towards the end of the transient creep. This acceleration in the creep rate has been described to mark the start of recrystallization. Gifkins⁸⁹ has explained this acceleration of creep rate to be dependent upon the creep of new grains formed during recrystallization and not their creation. This has been explained semiquantitatively resulting from initial extension and primary creep of new grains. That the soft new grains do deform readily is shown by the new coarse slip lines that appear very soon after the formation of the grains and which have been taken as an indication of the onset of recrystallization⁹⁰. Gifkins⁹¹ has also shown that the instantaneous strain has an effect on the creep strain to initiate recrystallization during his studies on the effect of prior strain on the recrystallization of lead.

Steady state creep is characterized by the constancy of the strain rate under conditions of constant stress. Recovery and strain hardening can occur simultaneously here, as shown by Cottrell and Aytakin⁹², giving a constant creep rate. Experimental evidence strongly indicates that the high temperature creep is diffusion-controlled.

In the present set of experiments, the stress was applied for only a short period of time. In case of lead spheres, both at room temperature and 100°C, it was found that practically the same strains were obtained irrespective of whether the required load was applied in 5 minutes or 5 seconds. In case of sapphire and K-Monel spheres, the total load

was applied within about 30 seconds. It was found that the instantaneous strain observed on loading slowed down quickly within a few seconds. These observations suggest that most part of the observed strain during hot-pressing, is due to the instantaneous and transient strains. Although no conclusions can be drawn regarding the basic mechanisms involved during hot pressing of lead and K-Monel spheres, it can be assumed that the strain produced during a very short period of hot-pressing is due to plastic flow mechanism. Since the hot-pressing experiments are being carried out above $0.5T_m$, recovery and recrystallization can be assumed to occur concurrently in the later parts of the experiments.

VII

S U M M A R Y A N D C O N C L U S I O N S .

1) The deformation behaviour of spheres in a compact can be predicted quantitatively by assuming the spheres to be packed in simple and systematic modes of packing. On application of hydrostatic pressure, plastic deformation takes place at the points of contact and the spheres are deformed to regular polyhedra depending upon the coordination number.

2) The densification produced during compaction can be described in terms of an equation relating the contact face radius a , particle radius R , initial packing density D_o , and the density D , at any deformation. The fact that the radius of the sphere remains practically constant for small deformations (for $a/R \leq 0.3$) leads to the final density equation given by

$$D = D_o + \frac{3}{2} D_o (a/R)^2.$$

3) The bulk strain can be related to the sphere parameters by the relationship

$$\epsilon_b = \left[(R^2 - a^2)/R_o^2 \right]^{3/2} - 1$$

and the bulk density and the bulk strain can be related by the relationship

$$D = \frac{D_o}{1 + \epsilon_b}$$

which on expansion gives, to a first approximation,

$$D = D_o (1 - \epsilon_b)$$

- 4) It has been shown by deforming uniform monosized spheres of lead, K-Monel and sapphire that the deformation of spheres in a compact is similar to that of the orthorhombically packed spheres.
- 5) The initial stages of densification are attributed to the combined effect of the particle rearrangement and plastic flow mechanisms. Very little fragmentation was observed in the case of the sapphire spheres.
- 6) A large variation in the contact face radii was observed during hot-pressing. It was found that the radii of the faces that were approximately perpendicular (or within 30°) to the direction of pressing are 2.2 times the radii of the faces formed in the direction parallel to the direction of pressing.
- 7) A study of the geometry of the deformation showed that most of the spheres deformed in a random manner. Individual colonies of orthorhombic, tetragonal and rhombohedral packings were occasionally observed.
- 8) On the basis of the fact that the contact areas grow to a stable size fixed by a constant stress equal to 3 times the yield strength, the density equation was modified to include the yield strength. A linear relationship between density and pressure was quantitatively established and experimentally tested.
- 9) The densification of sapphire single crystals by plastic deformation was established using optical and electron microscopy. The presence of the basal, and prismatic slip and also other complex modes of deformation ~~have~~^{has} been demonstrated. The contribution of plastic flow to the densification of sapphire (like other ceramic oxides)

is limited by the state of stress, temperature used, and the number of available slip systems under the experimental conditions.

VIII

SUGGESTIONS FOR FUTURE WORK

- 1) A study in which the diameter of the spheres is integrally related to the diameter of the die may be useful as such an arrangement will restrict large scale particle movement. A particular packing can be obtained by fixing the geometry of the die and thus densification of this packing may test the theoretical models more accurately.
- 2) The deformation of spheres under isostatic conditions may elucidate the mechanisms of hot-pressing.
- 3) A study under fixed geometry to identify the predominant mechanism of hot-pressing during the later stages of hot-pressing.
- 4) The extension of the deformation range beyond a/R equal to 0.3 should show the limit of densification by plastic flow and initiation of diffusion-controlled densification during the hot-pressing process.

A P P E N D I X I

THE PACKING OF SPHERES OF UNIFORM SIZE

The packing of spheres of uniform size has been studied by many workers in different fields. Gratton and Fraser³⁷ have discussed this subject extensively and their findings are most significant in this respect. There are five simple and systematic modes of packing of uniform spheres. These are shown in Fig. 59 (from Morgan⁹³) and can be described as follows.

1. Cubic Packing

This packing is constructed by placing spheres in square formation (Fig. 60b). Spheres in second and subsequent layers are placed vertically over those in the preceeding layers. Such an arrangement makes this packing most open and least stable.

2. Orthorhombic Packing

Figure 59 shows that this type of packing can be obtained either by stacking spheres in second layer horizontally offset with respect to those in the first layer by a distance R (sphere radius) along the direction of one set of rows, or by stacking vertically over those in first simple rhombic layer. It turns out that these two ways of packing are identical in nature though of different orientation in space.

3. Body-Centered-Cubic Packing

Figures 59b and 60c show this type of packing. It can be seen that if the spheres in the third layer have to lie vertically over those in the first, it forms a very unstable arrangement under gravitational

force alone.

4. Tetragonal Packing

This is constructed by placing spheres in second layer horizontally offset with respect to those of the first layer, by a distance R along the direction of one of the sets of rows. All the layers are simple rhombic in this case.

5. Rhombohedral Packing

As in case of orthorhombic packing, this can be constructed either from square layer type base or from simple rhombic layer type base. But in this case the spheres in second layer are horizontally offset with respect to those in the first layer in a direction bisecting the angle between two sets of rows by a distance of $R/\sqrt{2}$ in case of square layer formation, and of $2R/\sqrt{3}$ in case of simple rhombic layer formation. These two ways of packing are identical in nature though of different orientation in space.

Table IX summarises the basic methods of simple and systematic packing of uniform spheres.

TABLE IX : Basic Methods of Packing and their Construction

Method of Packing	Coordination number	Density of the Unit Cell	Density %
Simple Cubic	6	$\pi/6$	52.36
Orthorhombic	8	$\pi\sqrt{3}/9$	60.46
Body-Centered-Cubic	8	$\pi\sqrt{3}/8$	68.02
Tetragonal	10	$2\pi/9$	69.81
Rhombohedral	12	$\pi\sqrt{2}/6$	74.05

(a) Cubic	(b) Body-centred cubic
(c) Orthorhombic (from Cubic Base)	(d) Orthorhombic (from Rhombic base)
(e) Rhombohedral (from Cubic Base). Face-Centred Cubic	(f) Tetragonal
(g) Rhombohedral (from Rhombic base). Face-centred cubic.	(h) Rhombohedral (from Rhombic base) Close-packed hexagonal

Fig. 59 Basic Systems of Spherical Packings (After Morgan⁹³).

Fig. 60 Shapes of the Pores in Rhombic, Cubic, and B.C.C. Packings
(After Morgan⁹³),

A P P E N D I X II

A. THEORETICAL CALCULATIONS

1. Cubic Model

a/R	R	Bulk Density (%)	Bulk Strain (%)
0.00	0.620350	52.360	0
0.05	0.620352	52.556	0.37
0.10	0.620374	53.149	1.48
0.15	0.620469	54.145	3.30
0.20	0.620728	55.565	5.77
0.25	0.621282	57.423	8.82
0.30	0.622307	59.749	12.37
0.35	0.624038	62.576	16.32
0.40	0.626787	65.937	20.59
0.45	0.630972	69.869	25.06
0.50	0.637180	74.393	29.62
0.55	0.646268	79.498	34.14
0.60	0.659573	85.085	38.46
0.65	0.679356	90.843	42.36
0.70	0.709891	95.936	45.42
0.7071	0.715532	96.506	45.74

2. Hexagonal Prismatic and Tetrakaidecahedron Models

a/R	R	Bulk Density (%)	Bulk Density (%)	Bulk Strain (%)
		Hex. Prism.	B.C.C. Model	
0.00	0.620350	60.460	68.017	0
0.05	0.620352	60.687	68.273	0.37
0.10	0.620382	61.369	69.040	1.48
0.15	0.620509	62.511	70.325	3.28
0.20	0.620854	64.121	72.137	5.71
0.25	0.621593	66.207	74.483	8.68
0.30	0.622964	68.774	77.371	12.09
0.35	0.625287	71.824	80.802	15.82
0.40	0.628993	75.340	84.757	19.75
0.45	0.634679	79.272	89.181	23.73
0.50	0.643213	83.507	93.946	27.60
a ₁ /R	a ₂ /R	R	Bulk Density (%)[Z = 14]	
0.577350	0.333333	0.669404	99.450	

3. Rhombic Dodecahedron Model

a/R	R	Bulk Density (%)	Bulk Strain (%)
0.00	0.620350	74.048	0
0.05	0.620353	74.325	0.37
0.10	0.620397	75.156	1.47
0.15	0.620588	76.531	3.24
0.20	0.621107	78.437	5.95
0.25	0.622218	80.842	8.40
0.30	0.624288	83.697	11.53
0.35	0.627815	86.908	14.80
0.40	0.633499	90.317	18.01
0.45	0.642361	93.646	20.93
0.50	0.655600	96.410	23.19

B. EXPERIMENTAL RESULTS

1. Lead at room temp.

Load Lbs.	D_o %	D %	$\frac{D-D_o}{D_o}$	ϵ_b %	Z	$\frac{a}{R}$	$\left(\frac{a}{R}\right)^2$
1000	60.77	62.43	.022	2.65	7.67	0.0993	0.0099
1000	60.67	62.29	.021	2.56	7.55	0.1100	0.0121
1000	60.96	62.84	.031	3.00	7.65	0.1180	0.0139
1500	60.67	63.02	.039	3.70	7.80	0.1350	0.0182
2000	60.40	63.23	.047	4.45	7.60	0.1419	0.0201
2000	60.19	63.19	.050	4.73	7.56	0.1468	0.0216
2000	60.85	63.93	.051	4.81	7.56	0.1491	0.0220
3000	60.73	63.80	.051	4.80	8.40	0.1487	0.0221
3000	60.71	64.59	.064	6.00	8.42	0.1495	0.0224
3000	60.85	64.89	.067	6.22	8.26	0.1597	0.0256
4000	60.43	65.67	.087	7.95	8.72	0.1583	0.0251
4000	60.18	65.19	.083	7.82	8.61	0.1640	0.0269
4000	60.75	66.13	.089	8.12	9.06	0.1695	0.0287
4500	61.41	66.60	.084	7.82	8.39	0.1850	0.0342
5000	61.20	67.96	.110	9.93	8.30	0.2030	0.0412
5000	60.95	67.48	.107	9.67	8.73	0.2030	0.0412
5000	61.03	68.41	.121	10.79	8.80	0.2100	0.0441
6000	60.75	68.50	.128	11.30	8.87	0.2112	0.0446
6000	61.31	69.50	.134	11.80	9.03	0.2276	0.0518

2. Lead at 50°C

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Load lbs.	D_o %	D %	$\frac{D-D_o}{D_o}$	ϵ_b %	Z	$\frac{a}{R}$	$\left(\frac{a}{R}\right)^2$
600	61.10	63.39	.036	3.59	8.60	0.1176	0.0138
750	61.28	63.52	.037	3.51	8.65	0.1178	0.0139
1000	61.08	62.87	.029	2.85	8.63	0.1139	0.0130
1000	61.27	63.76	.041	3.92	8.70	0.1267	0.0160
2000	60.81	63.85	.050	4.73	9.05	0.1446	0.0209
2000	61.25	64.31	.050	4.76	8.35	0.1469	0.0216
2000	60.59	63.62	.050	4.76	8.72	0.1547	0.0234
3000	61.13	66.12	.082	7.54	9.25	0.1629	0.0266
3000	60.53	65.61	.084	7.73	8.95	0.1675	0.0280
3000	61.30	65.82	.074	6.85	8.70	0.1797	0.0323
4000	61.29	66.74	.089	8.15	8.85	0.1915	0.0366
4000	61.42	67.26	.095	8.70	9.25	0.1983	0.0393
4000	60.96	67.45	.106	9.61	9.50	0.2030	0.0412
5000	60.75	67.47	.111	9.96	8.75	0.1979	0.0392
5000	61.31	68.19	.112	10.09	9.30	0.1979	0.0392
5000	60.98	68.32	.120	10.73	9.50	0.1984	0.0394
5000	61.79	68.84	.114	10.25	9.75	0.2063	0.0426
6000	61.66	68.08	.104	9.41	9.35	0.2000	0.0400
7000	62.40	70.50	.130	11.46	9.35	0.2242	0.0523

3. Lead at 100°C

Load lbs.	D_o %	D %	$\frac{D-D_o}{D_o}$	ϵ_b %	Z	$\frac{a}{R}$	$(\frac{a}{R})^2$
500	61.75	63.66	.031	3.01	8.30	0.1011	0.0102
500	61.53	63.72	.036	3.41	8.33	0.1059	0.0112
500	61.25	63.96	.044	4.23	8.43	0.1114	0.0124
1000	60.99	65.16	.068	6.39	8.75	0.1308	0.0171
1000	61.12	65.18	.066	6.22	8.74	0.1319	0.0174
1000	61.01	65.23	.069	6.45	8.60	0.1348	0.0182
2000	61.30	66.63	.087	8.02	9.05	0.1432	0.0205
2000	61.44	66.73	.088	7.90	9.35	0.1485	0.0220
2000	60.67	66.61	.098	8.92	8.85	0.1650	0.0273
3000	61.12	68.25	.117	10.44	9.75	0.1767	0.0312
3000	61.06	68.68	.125	11.07	9.40	0.1893	0.0358
3500	61.59	69.95	.136	11.96	9.40	0.2166	0.0468
3500	61.05	69.82	.144	12.54	9.65	0.2227	0.0496
4000	61.01	70.84	.161	13.87	10.05	0.2232	0.0498
4000	61.35	70.22	.145	12.62	9.95	0.2262	0.0512
4000	60.91	70.03	.150	13.02	10.35	0.2302	0.0530
4000	61.12	70.96	.161	13.85	9.43	0.2334	0.0545

4. Sapphire

D_o	D	ϵ_b	Z	a/R	$(a/R)^2$	Temp °C
58.05	59.92	3.11	7.50	0.0999	0.00998	1570
57.68	59.67	3.34	7.48	0.0997	0.00994	1600
57.80	60.98	5.23	7.55	0.1261	0.0159	1700

5. K-Monel at 800°C

a/R	Z	σ psi	$(a/R)^2$	D %
0.099	8.2	625	0.0098	62.50
0.110	8.8	937.5	0.0121	63.14
0.121	9.9	1250	0.0146	63.50
0.124	9.1	1250	0.0154	63.20
0.133	9.6	1562.5	0.0178	64.00
0.138	9.5	1562.5	0.0190	64.13

6. K-Monel at 900°C

a/R	Z	σ_{psi}	$(a/R)^2$	D %
0.113	8.8	625	0.0128	63.02
0.125	9.6	937.5	0.0156	63.70
0.137	9.6	1250	0.0188	64.80
0.139	9.8	1250	0.0193	64.43
0.151	9.8	1562.5	0.0228	65.10
0.160	9.9	1562.5	0.0256	65.31

7. K-Monel at 1000°C

a/R	Z	σ_{psi}	$(a/R)^2$	D %
0.128	9.0	625	0.0164	64.69
0.129	9.3	625	0.0166	64.40
0.141	9.0	937.5	0.0199	65.41
0.145	9.5	937.5	0.0210	65.70
0.156	10.1	1250	0.0243	66.24
0.164	9.4	1250	0.0269	66.30
0.184	9.7	1562.5	0.0339	66.81
0.194	9.9	1562.5	0.0394	67.61

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