AN INVESTIGATION INTO THE COMPACTION OF POWDERS*

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The mechanisms by which a mass of powder is compressed into a coherent tablet, compact or pellet are not yet fully understood. One subject requiring clarification is the knowledge of the transmission of forces through the compacting mass and of the effects of these forces within the mass. Several workers have touched on this problem[1–6] but the most useful contributions have been made from the field of powder metallurgy[7–12]. However, there is conflicting evidence about several aspects of the process[7–11], and it is possible that some of the theories which have been advanced to explain the mechanism of pressing, based on the behaviour of metals, will not necessarily hold when applied to non-metals. In the work described in this paper, compacts have been prepared from a suitable material under controlled conditions, partly in order to obtain additional evidence on the sequence of events in the compaction of powders and partly to provide information for use in determining operating conditions for further work[13].

Experimental

Apparatus

A horizontally split die was designed (Fig. 1), in which there were eight sections each of 2·5 cm. depth which permitted a maximum fill of 13·5 cm. An accurate location was achieved by an overlapping skirt having a taper of 3° on the lower edge of each segment. This engaged on a corresponding taper on the upper edge of the lower segment to give an exact fit. All segments were made of K.9 steel (Edgar Allen and Co. Ltd.) but were not hardened; they were located and clamped together to form a die by means of three studs screwed into the bottom segment. The upper punch, also made of K.9 steel, was a sliding fit in the die and had a diameter of 5·32 cm. and length 20 cm. The base plate and bottom punch were made of mild steel. A location slot was milled in the base plate in order to prevent relative movement between base, die and lower punch. This die-set was used between the platens of a 50-ton hydraulic press to give a maximum punch pressure of over 2000 kg./sq. cm.

The Powder

Heavy Magnesium Carbonate B.P. was selected as the material most suitable for the pressings because it had constant powder characteristics of shape and size, it was free flowing, packed consistently and did not

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cake during storage; it did not affect the materials of the die-set; it cohered when compressed, so that the resultant compact generally could be taken intact from the die for subsequent measurements; the particle size was negligible compared with the diameter of the die so that dimensional effects were reduced to a minimum. In order that different

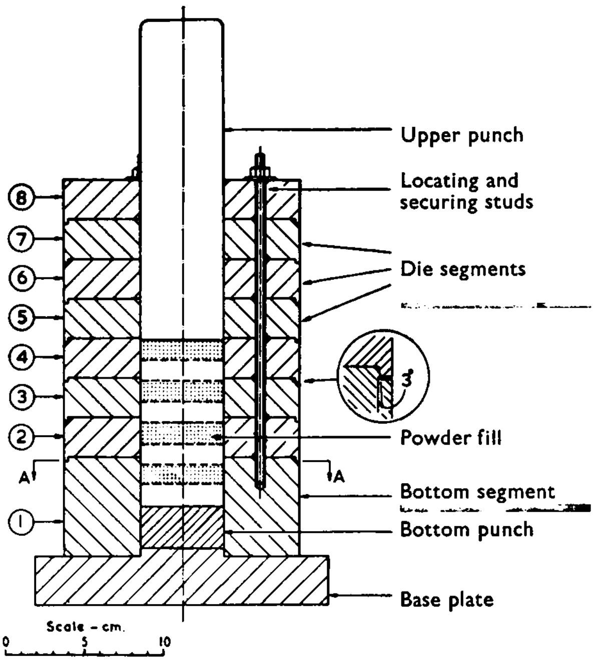

FIG. 1. Split-die assembly.

powder layers could be distinguished, 0·25 per cent. ammoniated carmine was used to produce a powder of satisfactory colouration to contrast with the natural white colour of the material. The particle size was standardised for the powder of both colours by using the 200–240 mesh fraction after sifting on an Inclyno sifter. The experiments were planned so that the powder fill could be made up of alternate layers of different colour.

The Compacts

The main factors affecting compaction are applied pressure, depth of powder and condition of die wall surface. A manageable experimental series to include all these factors was made to the following plan.

(*a*) *Three levels of pressure.* Final mean pressure of top punch, (i) 671 kg./sq. cm., (ii) 1358 kg./sq. cm., and (iii) 2040 kg./sq. cm.

(*b*) *Three depths of powder.* Controlled by weight and subsequent hand tamping to predetermined levels, (i) 80 g. in 20·0 g. layers (2 white, 2 pink), (ii) 160 g. in 20·0 g. layers (4 white, 4 pink), and (iii) 240 g. in 20·0 g. layers (6 white, 6 pink).

(*c*) *Two surface conditions.* (i) "unlubricated" plain wall, and

(ii) "lubricated" wall—lubricated with colloidal graphite (acetone "dag" (Acheson Colloids Ltd.)).

This gave $3 \times 3 \times 2 = 18$ experiments for which a suitable experimental pattern was devised (see Table I).

TABLE I

EXPERIMENTAL PATTERN FOR PRESSING STUDIES

Pressing	Compacting pressure (kg./sq. cm.)	Weight of powder fill (g.)	Type of surface
P.1	1358	80	lubricated
2	671	80	unlubricated
3	671	160	unlubricated
4	671	160	lubricated
5	1358	80	unlubricated
6	2040	160	lubricated
7	1358	240	lubricated
8	2040	80	lubricated
9	2040	240	lubricated
10	671	240	unlubricated
11	671	80	lubricated
12	1358	240	unlubricated
13	671	240	lubricated
14	1358	160	lubricated
15	2040	240	unlubricated
16	2040	80	unlubricated
17	2040	160	unlubricated
18	1358	160	unlubricated

Method

All surfaces to be in contact with the powder were degreased with equal parts acetone and carbon tetrachloride mixture and then polished with "Bluebell" metal polish; the internal surfaces of the die segments, including the part of segment No. 1 next to the bottom punch, were coated with a thin layer of colloidal graphite in those experiments employing lubrication.

The powder was poured into the die a layer at a time with frequent pauses for hand tamping to ensure a constant initial condition of packing of the particles. Each 20·0 g. layer was levelled to a predetermined height using a depth gauge. After the die had been assembled and the top punch slipped into position, pressure was applied with two minute intervals between increments after each of which the exposed punch length was measured. On reaching the required pressure, the load was released and measurements again taken of the exposed punch length and the height which the die had risen from the base plate. The die was taken off its base, placed on suitable supports and the compact forced out through its lower end, that is in the same direction as the applied pressure. Also, a note was made of the force required to produce the first detectable movement.

The height of the resulting compact was measured accurately and it was cut longitudinally using a fret-work machine; if the compact was broken or cracked, it was embedded in paraffin-wax before cutting. Photographs of some of the results are shown in Figure 2.

RESULTS AND DISCUSSION

The Sections

An inspection of the longitudinal sections brings out several interesting features. The most striking observation is the differences in relative

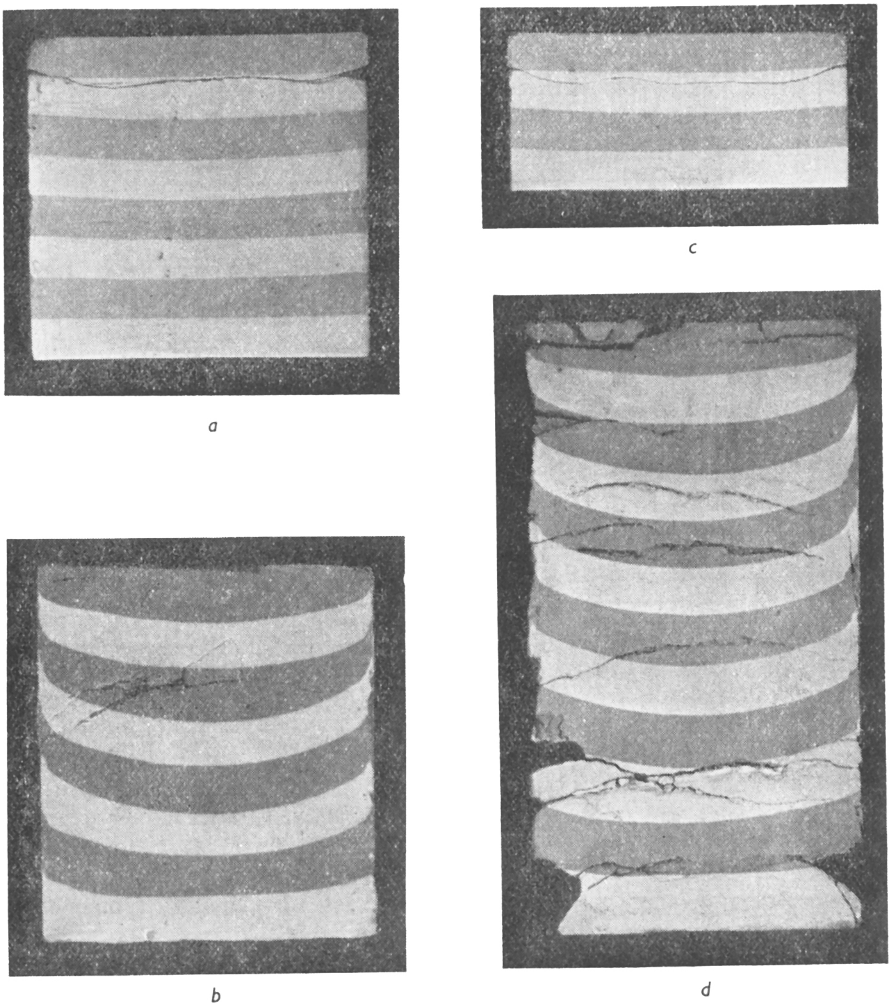

FIG. 2. Cut sections of compacts after ejection from die.

a. Pressing,	p. 6,	compacting pressure		2040	kg./sq. cm.	in	lubricated die
b. ,,	p. 17,	,,	,,	2040	,,	,,	unlubricated die
c. ,,	p. 8,	,,	,,	2040	,,	,,	lubricated die
d. ,,	p. 12,	,,	,,	1358	,,	,,	unlubricated die

displacement of the layers of material when compacted to the same level of pressure, but using different conditions of lubrication (Fig. 2*a* and *b*). This feature has been noted by various workers in the powder metallurgy field[7,8,14]. The differential displacement is caused by the greatly increased frictional forces occurring at the face of an unlubricated die wall, opposing the movement of the adjacent compact material which is tending to move as a result of the thrust from the top punch. On the other hand, the material at the centre is relatively free to move, being subject only to normal interparticulate friction. Consequently the layers are unevenly displaced in the manner shown in the sections.

A second interesting observation is the presence in the compact of cracks of two types. The first can be seen as a split near the top of a compact (Fig. 2*a*). The shape is slightly concave in an upward direction along the axis of symmetry and roughly follows the line of demarcation between the top two layers of material. This type of crack is common in the lubricated pressings but is also found in the unlubricated pressings. The second type is concave in a downward direction and is to be seen in the sections of the unlubricated compacts which were subjected to the heavier pressures (Fig. 2*b* and *d*). How these cracks may arise is discussed later.

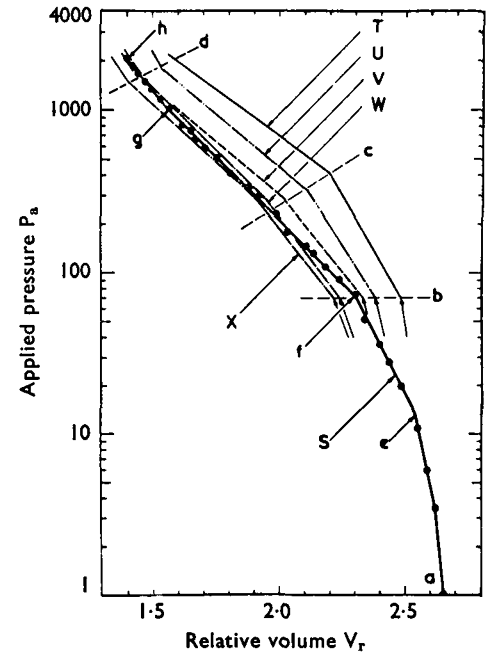

FIG. 3. Relation between applied pressure and relative volume of compact. In the case of curve S,

Stage I extends from a to e
" II " " e to f
" III " " f to g
" IV " " g to h

Applied Pressure-Relative Volume Relationships

From the measurements the exposed punch length taken during a pressing, the height of the compact within the die after each pressure increment was calculated, allowance being made for the shortening due to elastic compression of the top punch. Since a simple cylindrical shape was used, the relative volume V_r, defined by Walker[1] and used by Bal'shin[7] is given by dividing the observed length, L, by the length of the material when present as a true solid, L_s, i.e.,

$$V_r = \frac{L.\pi.D^2/4}{L_s.\pi.D^2/4} = \frac{L}{L_s}$$

where D is the diameter of the compact.

This ratio was computed for each height and the relations between the relative volume and the applied pressure P_a, for the 18 pressings are summarised in Figure 3 by lines T to X. T, U and V represent the results obtained when 12, 8 and 4 layers of powder are pressed in an unlubricated die, whilst W represents 12 and 8 layers, and X, 4 layers of powder pressed in a lubricated die. For this series, the average relative volume of the powder fill was 2·66 ("a" in Fig. 3) and the first stage, represented by short arrows at the base of each curve, for each pressing was an applied pressure of 70 kg./sq. cm. This level has been shown as the horizontal line "b" and must be considered the datum line for this series, even though these compacts have a previous history of formation up to that pressure. It will be noted that each curve consists of three sections delineated by abrupt changes of slope. These breaks occur along roughly the same lines which have been labelled "b", "c" and "d".

Curve S is derived from a further series of 10 lubricated pressings in which readings were taken at a larger number of pressure levels. In this case a mean value of V_r for each value of P_a was obtained and the results plotted in Figure 3. There are eight readings below line "b", thus providing information for a region not included in the preliminary pressings.

It is possible to suggest an explanation of the changes in slope in the curves in terms of the changing physical condition of the material being pressed. It will be seen that such changes occur in curve S at points "e", "f" and "g". Stage I occurs in the region "a" to "e", i.e., up to 13 kg./sq. cm.; here decrease in the relative volume is most probably caused by interparticulate slippage of the powder, leading to closer packing. This process, however, is limited since the particles soon become immobile relative to one another and give rise to Stage II. This condition, found between "e" and "f", is characterised by the formation of temporary struts, columns and vaults[15] protecting small voids and generally supporting the imposed load. Stage III extends from "f" to "g", when the structure of the material fails either by crushing or by plastic flow. This takes place because there are point and line contacts between the asperities and angular edges of the particles, and, as P_a increases, the stresses are transmitted from particle to particle through these contacts. Although the force transmitted between two particles may be relatively low, it takes place over such a small area that high stresses are imposed locally causing the material to fail. For the system under examination, this failure begins at about 70 kg./sq. cm., and extends to about 1000 kg./sq. cm.

However, since this failure takes place in a confined space, the freshly produced surfaces will be held in close proximity to one another under the influence of smaller transverse stresses. This is a favourable condition for bonding and "cold welding". Whether or not bonding actually does take place, depends on the coherent and adherent properties of the material. With magnesium carbonate, there is a strong coherent property; thus, although the powder is being broken down by the increasing load, simultaneously it will be rebonding to form a more closely packed structure. For a time the rate of breakdown will greatly exceed the rate of rebonding and this idea is supported by the work of Higuchi and others[3] who have shown that the specific surface area of a material increases during the initial stages of compaction. However, as the stresses increase, a condition eventually develops, Stage IV, when the structure formed is strong enough to support the imposed load and any further reduction in volume of the compact involves the normal compressibility of the solid material. This stage is probably developing at point "g" on curve S and any permanent decrease in the voids of a still porous compact in this state can only be achieved either by exceeding the crushing strength of the structure or by plastic deformation, or both.

It must be emphasised, however, that this explanation is based on evidence obtained from external measurements and that as pressure is applied to a powder, various zones within the pressing will be subjected to different reliefs of pressure so that the particles in these zones will be at different stages of compaction. Thus the combined condition, given by external measurements, may not truly reflect the internal measurements.

In the preliminary pressings, due to the fact that the first pressure increment was 70 kg./sq. cm., the system was not able to accommodate itself for Stages I and II. Consequently in the next increment (to 110 kg./sq. cm.) imposed forces were being applied which could cause crushing and breakdown of powder near the top punch, whilst that near the bottom punch was still only being packed more closely. Thus the region "b" to "c" in Figure 3 is really a condition when there is a mixture of elastic and plastic deformation taking place and the result is a slope midway between that given by Stages II and III of curve S. It is not until the region between "c" and "d" is reached that the slope of the curves for the preliminary pressings approaches that of Stage III of curve S.

Observations of the compact state after compression serve to support the suggested explanation. The individual particles of powder of a pressing made at 28·5 kg./sq. cm. were only packed together and were easily removed from the surface of a cut cross-section using a camel hair brush; in a pressing made at 62 kg./sq. cm., the packing was firmer and the particles harder to dislodge; at 90 and 145 kg./sq. cm., there was a definite keying and bonding of the particles; at 336 and 671 kg./sq. cm., the compacts had become quite firm and the outline of the individual particles was difficult to trace even with the use of a low powered microscope; for pressings made to 1358 and 2040 kg./sq. cm., the compact to all appearances was a solid block, and, using a microscope, only occasionally could the outlines of the original particles be seen.

There is supporting evidence for this explanation in published work. Various descriptions of the compaction sequence have been noted[7,8,16,17], but as far as can be ascertained, only Huffine[17] has attempted to trace to any extent the various stages using the relation between applied pressure, P_a, and the relative volume, V_r. The author considers that probably too much attention has been directed to the exponential relation which usually exists at some stage of the process, and as a result evidence for the existence of other relations was missed.

A further experimental point is that at all stages a "skin" was produced where the compacts had been in contact with the die-walls, and the particles in this region were always relatively much more distorted and bonded together compared with those situated within the compact. This condition is attributed to the local shear forces produced during compaction and extrusion causing breakdown with simultaneous rebonding of particles to give a denser structure of skin thickness only. In compacts pressed above 145 kg./sq. cm. the particles in contact with the punches appear to be bonded together more strongly than the internal particles adjacent to them. This may be explained by the development of a special boundary condition, where the particles are being crushed against a hard surface, the fragments filling up the voids and rebonding. An analysis of the forces which cause crushing under these conditions was first made by Hertz[18]. It must be emphasised that the bonding near the punch surfaces was never so marked as that at the die walls.

Elastic Recovery of the Compacts

There is a stage at which the powder has formed a structure which, although still porous, is strong enough to support the applied pressure and the natural elastic properties of the material become important as is shown by the fact that the relative volume exhibits a recovery when the pressure is released, especially with the lubricated compacts (Table II).

TABLE II

RECOVERY IN LENGTH AFTER RELEASE OF APPLIED PRESSURE

Compact size g.	Lubricated die		Unlubricated die	
	Per cent. recovery on release of P_a	Per cent. recovery when extruded	Per cent. recovery on release of P_a	Per cent. recovery when extruded
80	2·3	4·4	2·5	4·2
160	3·3	4·8	1·6	4·6
240	2·5	4·2	1·0	Not measurable

Further increase in volume takes place as the compact is ejected from the die when the total recovery is the same for all compacting conditions of this series and seems independent of the condition of the wall surface. It would appear that elastic recovery takes place only to a limited extent within the die, frictional forces on the walls opposing complete relaxation especially in the unlubricated die, and full recovery is possible only after the compact has been ejected.

Force Required to Produce First Movement

Inspection of the values for the force, F, in units kg. weight, required to produce first movement of a completed compact in a die shows two trends. The first is that there is an exponential relation between F and the applied pressure, P_a, provided all other conditions are kept constant. The second is that F is related exponentially to the height, L, and therefore to the final wall surface, S, of the compact, all other conditions being kept constant. It was found advantageous to combine P_a with S as a product and the logarithm of this function (log. $P_a.S$) was plotted against log F as shown in Figure 4. Results of 24 other pressings using lubricated die-walls and a wide range of pressure conditions have also been included in this figure.

There are two sets of results according to the condition of the die surface. For the unlubricated die, the results fall about a "best straight line", line A, which fits the equation:

$$\log F = 1{\cdot}43\ (\log P_a.S) - 2{\cdot}68$$

i.e., $$F = 0{\cdot}0021(P_a.S)^{1.43} \quad \ldots \quad (1)$$

In the case of the lubricated die, the line fitting the points is B, the equation of which is:

$$\log F = 1{\cdot}43\ (\log P_a.S) - 3{\cdot}39$$

i.e., $$F = 0{\cdot}00041\ (P_a.S)^{1.43} \quad \ldots \quad (2)$$

Conclusions which may be drawn from this correlation can be only tentative, but two points are worthy of comment. The first is that the equations derived from the graph are dimensionally homogenous, which indicates that the correlation may have a physical basis. The second is that it is possible to develop a theoretical approach to aid the understanding of the correlation and of the mechanism associated with the frictional effects developed during compaction and ejection.

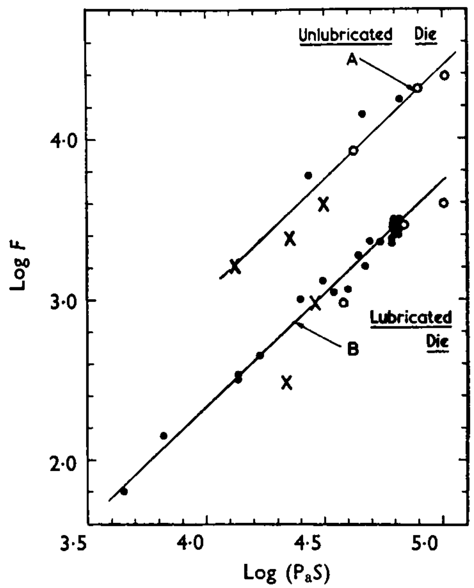

FIG. 4. Relation between the force required to produce first movement, F, and the product of maximum applied pressure, P_a and the final surfaces S.

× 80 g. compacts
● 160 g. ,,
○ 240 g. ,,

If the compacting system is analysed at the time at which the maximum applied pressure has been reached, it will be seen, Figure 5, that this pressure, P_a, will be transmitted to the material immediately below the top punch. Let the mean vertical stress on a horizontal plane at distance, z, from the top punch face be defined as P_z: then it is reasonable to assume that

$$P_z = f_1(P_a,z),$$

and that the nature of the function, $f_1(P_a,z)$ will be the same for similar conditions of pressing.

If the radial stress along the horizontal plane, distance, z, from the datum surface and radius, r, from the axis of symmetry, is $P_{r(z)}$ there will be a relation between P_z and $P_{r(z)}$ depending on the physical properties of the material and possibly on the condition of the die-wall surface.

If these conditions are held constant for a particular experimental series, then we may write

$$P_{r(z)} = f_2(P_z, r),$$

and it follows

$$P_{r(z)} = f_3(P_a, z, r).$$

Let $d(F_z)$ be the frictional force at the die wall tending to prevent motion of the element of thickness, dz, and which is subject to the radial stress, $P_{R(z)}$ at the wall surface of the compact of radius, R: then,

$$d(F_z) = \mu_z P_{R(z)} \pi 2R\, dz$$
$$= \mu_z f_3(P_a, z, R) \pi 2R\, dz,$$

where μ_z is the coefficient of friction between the compact and the die-wall at z.

Over the whole wall surface of the compact the force, F, resisting movement will be

$$F = \int d(F_z) = \int_O^L \mu_z f_3(P_a, z, R) \pi 2R\, dz, \quad .. \quad ..(3)$$

where L is the length of the compact under the conditions considered.

Equation (3) is a general equation for the system and can only be solved when the relation between the various factors are known. This requires a complete knowledge of the mode of stress transmission within the material and a knowledge of the change in value of the coefficient of friction, μ_z, with z. The former information is not known at present and the variation in the coefficient of friction along the length of a compact has only recently been demonstrated. Thus Unckel[14] in testing his theory of stress distribution within a compact used a coefficient of 0·2, whereas Kamm, Steinberg and Wulff[9] found in some of their pressings that the value of μ_z varied from 0·625 near the top punch to less than 0·07 near the bottom.

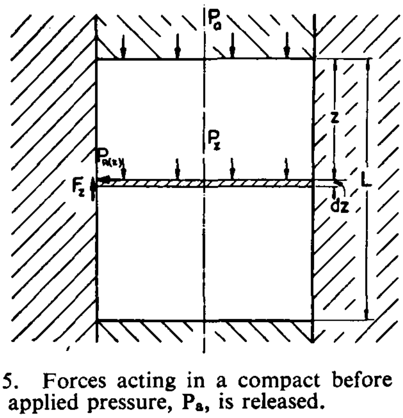

FIG. 5. Forces acting in a compact before maximum applied pressure, P_a, is released.

Before extrusion can take place the applied pressure is released and the material exhibits an elastic relaxation to which attention has already been drawn in this discussion. Residual stresses remain in the compact and certain other stresses are reintroduced when the force, F necessary to produce first movement for extrusion is applied. This gives rise to a new set of stresses acting in the compact and it is a function of these stresses, distinguished in the following formulæ by the index ^{e}, which combined with the coefficient of friction, μ_z^e, acting on the walls, tends to oppose motion when the extrusion process takes place.

$$\text{That is, } F = \int d(F_z) = \int_O^L \mu_z^e f_4(P_z^e, R)\pi\, 2R\, dz \quad .. \quad ..(4)$$

It is probable that $\mu_z = \mu_z^e$
and also that

$f_4(P_z^e,R)$ is simply related to $f_3(P_a,z,R)$.

This is supported by evidence obtained from a paper by Nelson and others[4] who published data (Table III in their paper) on the forces developed during the production of tablets of 0·954 cm. diameter, using compacting forces of up to 1300 kg. weight. All experimental conditions were kept constant, except the amount and type of lubricant. From the information supplied, it was possible to calculate the force, F, resisting movement at the die wall at maximum applied pressure, and also the force, F, to produce first movement for ejection, and, as shown in Figure 6, there appears to be a simple relation between F and F. This graph means that, if μ_z is proportional to μ_z^e, then $f_4(P_z^e,R)$ must be of the same form as $f_3(P_a,z,R)$.

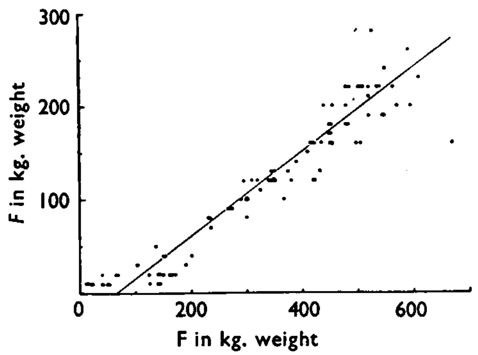

FIG. 6. Relation between the force, F, resisting movement at the die wall at maximum applied pressure and the force, F, to produce first movement for ejection (From Nelson and others[4]).

The results of the present work point to a relationship of the form

$$F = c(P_a S)^m \quad .. \quad .. \quad .. \quad .. \quad .. \quad ..(5)$$

This equation is consistent with the analysis above. The constant m is a property of the material used and possibly of the physical dimensions of the system, whereas c is a property of the surface condition of the walls. It is probable that in making other series of pressings of the same nature, but varying the lubricant on the walls, other lines parallel to A and B in Figure 4 would be obtained. For example, if a good lubricant such as stearic acid were used, the resulting line would lie near that of line B and if a material like colophony resin were used to coat the walls the line would be parallel to and lie near line A.

Apparent Density Calculations from Sections

By reason of symmetry, the rotational movement of particles in an evenly packed cylindrical powder mass has no significance. Several workers[7,9,14,19] have stated that there is little evidence to indicate radial movement in a powder mass when an even compacting force is applied in a die with well lubricated walls. It follows that the only significant movement that the material can make is along the z-axis. That being so, if a given weight of material is evenly distributed horizontally in layers before pressing, it will be possible to calculate the degree of compaction in various regions of the resulting compact from measurements of the vertical heights of the appropriate layers seen in a half section of the compact.

The cut sections of the lubricated compacts were inspected and the heights of the various layers measured using a Universal measuring machine (Cambridge Instrument Co.); the verticals chosen were the centre-line, together with lines parallel to and at a distance from it of R/4, R/2, 3R/4, 7R/8 and R where R is the radius. These latter readings were taken on both sides of the centre-line and a mean reading recorded. From the measurements the per cent. solid present was calculated and these figures were transferred to an outline of the compact; lines of equal apparent density were then drawn in the three diagrams on the left of Figure 7. Evidence confirming the validity of this procedure will be presented in another paper[13].

For sections of compacts made with unlubricated walls, the assumption that no significant radial movement takes place is not necessarily true. Because of this any apparent densities determined by the above procedure can be used only as a guide or a first approximation and cannot be considered to be correct until proved by some other means. However, it was of interest to see what picture would emerge if the method were used on some of the sections to hand, so a group of 8-layered "unlubricated" compacts was measured and the results are presented in the right-hand column of diagrams in Figure 7.

Inspecting the results of the lubricated compacts first (Fig. 7*a*, *b* and *c*), it can be seen that there is a region of greater apparent density in the top corners of all pressings, and a region of lower apparent density in the bottom corners. The greatest difference between the figures closest to the wall (line 7R/8) is only about 5 per cent., but those on the surface show up to 11·5 per cent. In the body of the pressings, there is a region of lower density near the top centre and a region of higher density about two-thirds of the way down. The greatest difference amounts to 6 per cent. The interesting feature is that the region of higher density on the centre-line is roughly of the same magnitude as that in the top corners and these regions are connected by "ridges" exhibiting a lower density. In the more heavily pressed compacts, *b* and *c*, the low density region in the centre is about the same apparent density as that in the bottom corners. In general these diagrams are similar to the examples given by Kamm, and others[9].

A similar picture is seen in the results of the unlubricated pressings but it may be distorted due to undetected radial movement. Here, the range in densities is greater and the higher density region on the centre-line has not the same magnitude as that found in the top corners. When

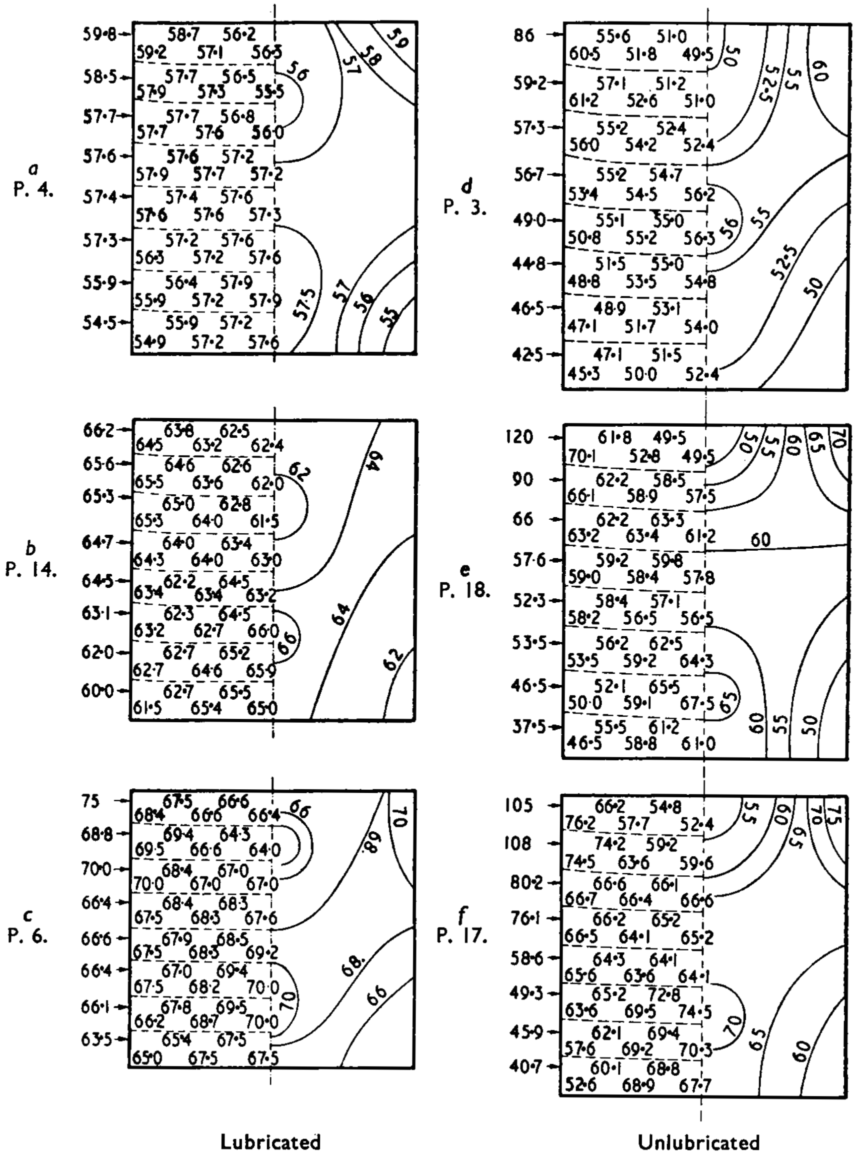

FIG. 7. Apparent density distributions in preliminary pressing expressed as per cent. solid present.

compared with densities just within the pressing, those on the edge show sufficient discrepancy to indicate that movement of material takes place during extrusion, causing a concentration towards the top end. This shows particularly in the unlubricated compacts where apparent densities of "120" per cent. would appear to have been achieved. For this reason the surface figures have not been taken into account when drawing the contours.

There is an interesting point about the region of higher density found in the lower centre portion of the compacts. The presence of a denser structure such as this in a normal tablet could explain the well-known empirical observation in a disintegration test where the outer layers of the tablet disintegrate and slough away quite rapidly leaving a core which requires a longer period before it breaks up.

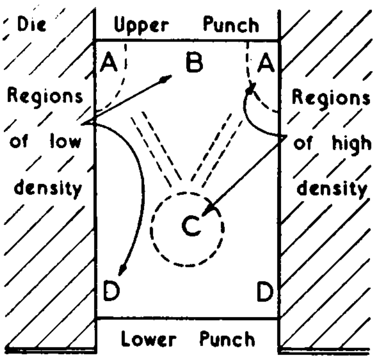

FIG. 8. Conditions in compact before applied pressure is released.

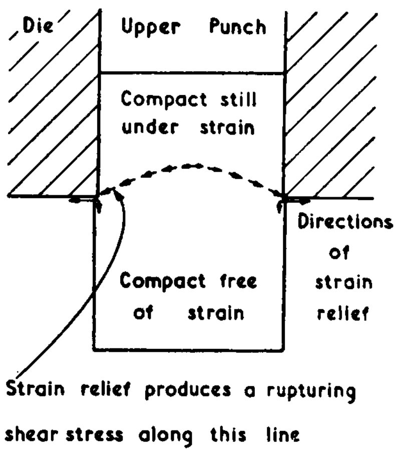

FIG. 9. Extrusion of unlubricated compact.

The variations in apparent density that exist in different regions of a compact have been noted. Evidence of and comment on the formation of these regions will be given elsewhere[13], but their effect on the subsequent history of the compact will be discussed now. On the release of the maximum applied pressure, there will be a peripheral elastic relaxation along the longitudinal axis in zone A (Fig. 8) and this will induce a longitudinal stress in B, where the material is less dense and therefore probably weaker. Since the structure of the compact is such that it is not strong in tension, a relief of weakness will be produced. Any additional stress due to some other factor will tend to cause a laminar crack to develop through the material. Attention has already been drawn to cracks of this form in pressings made for this work, and it is probable that this condition is present to a greater or lesser extent in all compacts. It is suggested that this is the basic cause of the phenomenon called "capping". Capping is that condition where the top of the pellet or tablet "becomes detached and even when this is not immediately apparent, the 'cap' can be removed with the thumb nail or it will fall off when a few tablets are shaken in a bottle"[20]. The reasons for capping are various, and amongst those listed by Silver and Clarkson[21] are (*a*) too much pressure, (*b*) entrapped air, and (*c*) insufficient binder. In each case the "reason" is the obvious or visible factor which is sufficient to increase the stress at the capping site to breaking point.

The relaxation of the elastic strains in the lower part of the pressing (C in Fig. 8) takes place in a downward direction causing the die-block to be lifted off the base plate. Induced tensile stresses are probably developed in the peripheral parts, D, but, since there are no cracks in the lubricated pressings in this region, the breaking or cracking stress is probably not exceeded. The stress pattern in the case of the unlubricated pressings is very complex even after the maximum applied pressure has been released. Since the elastic strains are only partially relieved at this stage (Table II), full relaxation can only take place on extrusion. Further, even during extrusion, the stresses induced by friction at the walls hold the strained condition in the material until the edge of the die is reached when a spontaneous expansion in two directions takes place (Fig. 9). Thus the material outside the die is relaxed whereas that inside the die is still stressed. The imposed strain exceeds the shear strength of the material resulting in the series of cracks to which attention has already been drawn in Figure 2.

Summary

1. Using Heavy Magnesium Carbonate B.P., the differential movement of a powder has been investigated in a cylindrical die when subjected to applied pressures of up to 2000 kg./sq. cm. from one end.

2. The relation between the relative volume of the material and the applied pressure has been determined for selected conditions of pressing, and a possible explanation in terms of the changing physical condition of the material is advanced for the changes in slope which occur in the curves.

3. Studies have been made correlating the force to produce first movement for extrusion, the maximum applied pressure and the final wall surface of the compact.

4. The variations in relative density within a compact have been estimated and a tentative explanation has been made of the cause of the phenomenon known as "capping".

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DISCUSSION

The paper was presented by the AUTHOR.

DR. F. HARTLEY (London) enquired why the author had selected a basic substance, heavy magnesium carbonate, as the material for his work. Was the same batch used throughout and was the moisture content uniform?

MR. E. W. RICHARD (Upminster) said the author had used material and procedures which were far removed from the normal conditions of pharmaceutical tablet manufacture. The die walls and punch faces were lubricated, whereas in tablet manufacturing practice the granules were lubricated. One could foresee that that would make a considerable difference to the internal stresses set up during compression. Then, again, pressure was applied at intervals of two minutes, and the increments of pressure were not stated. Flat punches were used, but the internal stresses in a biconvex tablet would be very different: also contrary to normal procedure, the compact was extruded from the die in the same direction as that of the applied pressure. It was not clear whether the author was aware of the difference between capping and lamination. By "capping" was meant the phenomenon whereby the surface layer of the tablet broke away when in a certain form, and by "lamination" was meant the horizontal splitting open of a tablet. In his experience he had found those two conditions to be quite distinct.

MR. H. BURLINSON (Ashton-under-Lyne) said inorganic powders did not present the same problems in compression as organic ones. Had the author any experience of using organic compounds?

MR. A. AXON (Dartford) asked how the ammoniated carmine was incorporated. Could some information be given about the shape and size of the particles, and whether there was any aggregation. It was not clear from the paper why the author had used a horizontally split die. It would seem that in the method of compaction the uppermost layer would be the least compacted. He wondered whether it was significant that the split near the top of the compact occurred between the two least compacted layers in that region. It was also difficult to appreciate why there were the marked changes in slope of curve S in Figure 3.

DR. D. TRAIN, in reply, said he had chosen heavy magnesium carbonate because he wanted a material which was easy to handle. He had extensively reviewed the literature, and had come to the conclusion that for this initial work all he needed was a simple, readily available substance. A sample of 200 to 240 mesh powder was selected, dried for 24 hours at 110° C., allowed to humidify for 2 hours under standard conditions and then 80 g. quantities were placed in sealed containers. The containers were opened just before pressing was carried out; the moisture content was about 0·5 per cent. under those conditions. He had been taken to task for departing from the normal procedure of tablet manufacture but he had not intended to follow this. He had chosen a cylinder because it was the simplest practical shape on which to make measurements, he wanted a size which would facilitate manipulation of the powder fill, so he chose a die of 5 cm. diameter, and because he found difficulty in packing the powder fill in horizontal layers, he had split the die horizontally so that it could be built up as it was filled. He felt that the rôle of lubricants in the compaction process needed clarifying. The pressures were applied in roughly a logarithmic increment. Over the total range he had approximately 30 pressure levels. So far as extrusion in the same direction was concerned, again he did not want to complicate a simple picture. He wanted to find out what the compact looked like when pressing was continued in the same direction and he felt that by pressing in the opposite direction the contour of the layers within the compact would be altered because the direction of applied force had been reversed. Ammoniated carmine was used because it was simply made and easily incorporated. The pink and white powders under microscopic examination, appeared to be identical in shape and the constituent particles were not aggregated and had a shape factor of about 80 per cent. sphericity. It would be seen from Figure 7 that the uppermost layer of the compact showed quite a difference in its final condition. There was a wide difference in the apparent density distribution across a horizontal line near the top. It could not be said that any one of the layers was less compacted before compression because they were all the same. Each layer was hand tamped to a predetermined level so that every compact consisted of layers of powder with the same distance between them. With regard to the question of rigid stages, although line S (Fig. 3) represented the mean of approximately 15 pressings and each point had been put in separately, the relation between the series of points in each experimental run was so characteristic and reproducible that it was felt that attention should be drawn to the sharp changes of slope.