JOURNAL OF MATERIALS SCIENCE 5 (1970) 663-668

The Cold Compaction of Several Mineral Powders

P. LAWRENCE*
Department of Applied Mineral Sciences, Houldsworth School of Applied Science, The University, Leeds, UK

Four mineral powders with widely different mechanical properties have been studied in cold compaction. The effect of final compacting pressure and particle size have been investigated. An analysis of the experimental data indicates that the same basic empirical compaction equation can be applied to the compaction of all four powders. A theoretical model for the compaction process is developed and the empirical parameters are correlated with the mechanical constants of the materials.

1. Introduction

The cold compaction of powders has been widely investigated in the past. The scope of the work can be judged from the comprehensive review made by Bockstiegel and Hewing [1], where reference is made to more than 270 original papers. Many empirical descriptions of the compaction process have been developed and experimental parameters have been derived but in many cases the description has been applied to only one material, or one type of material, and no detailed interpretation of the experimental parameters has been attempted on the basis of the mechanical properties of the material involved. Several possibilities have been indicated.

Heckel [2] investigated Konopicky's [3] relationship between the relative density D of a metal powder compact and the applied pressure P

$$\log \frac{1}{1 - D} = KP\,.$$

He concluded that on an experimental basis K, the proportionality constant, correlated with the reciprocal of the nominal yield stress of the material.

Cooper and Eaton [4] examined the cold compaction behaviour of several ceramic powders of widely different hardness values and they drew the conclusion that the compaction behaviour depended largely on the hardness of the material.

Although using a hot pressing technique Habberjam [5], investigating hot char briquetting, indicated similar correlations between the stress constant, Q, of his basic compaction equation and the breaking strengths and yield stresses of the chars. The basic compaction equation is

$$P = Q\left(\frac{1}{\phi} - \frac{1}{\phi_0}\right)$$

where P is applied pressure and ϕ is the porosity of the briquette, the subscript 0 referring to zero applied pressure.

This investigation deals with the cold compaction of four mineral powders with widely varying bulk material properties. The cold compaction of the powders has been investigated in two size fractions up to pressures of 2×10^9 dyn cm^{-2} and a pressure application time of up to 600 sec. The previously developed compaction equation of Habberjam [5] has been successfully applied to the instantaneous compaction of the four powders. A modified compaction equation has been developed to describe the time-dependent compaction behaviour. This is outlined in a later paper; here an analysis of the instantaneous compaction equation parameters indicates correlations with the bulk material properties.

2. Experimental

Table I shows the materials selected for this investigation together with their bulk material properties. The halite is ICI grade A ground rock salt. The hematite was obtained from Millom, Cumberland. The calcite was obtained from

---

*Now at Building Research Station, Garston, Watford, Herts.

TABLE I Material constants

	Halite	Calcite	Hematite	Quartz
Vickers hardness number	20	110	800	1200
Nominal yield stress $\times 10^8$ dyn cm^{-2}	6.6	37	266	400
K, bulk modulus (from β-tables) $\times 10^{11}$ dyn cm^{-2}	2.44	7.15	16.7	3.70
K, bulk modulus (from elastic constants) $\times 10^{11}$ dyn cm^{-2}	1.95	4.05	11.1	3.74
G, shear modulus (from elastic constants) $\times 10^{11}$ dyn cm^{-2}	2.85	4.51	13.7	6.87
E, Young's modulus (from K and G) $\times 10^{11}$ dyn cm^{-2}	5.76	9.89	26.2	12.8

Appletreewick, Yorks, and the natural quartz was supplied commercially.

The materials were crushed, milled and sieved to the size fractions $-60/+100$ mesh and $-120/+300$ mesh giving nominal particle size limits of 250 to 150 μm and 125 to 53 μm respectively.

The powders were compacted by a laboratory hydraulic press at room temperature in a hardened steel, uniaxial, single punch, fixed die. Two dial gauges, either side of the punch mounted on a rigid suspension arm connected to the punch, record the displacement of the punch. The dial gauges were read to 0.001 mm by estimation in the absence of any appreciable backlash. The mean of the two readings from the dial gauges determined the displacement of the punch and hence enabled the compact length to be calculated. To correct the displacement readings for the elastic deformation curves were determined by pressing without materials in the die.

Compacts 38 mm in diameter and of the order of 5 mm thick were produced. The weight of the compact was fixed by the need to produce thin compacts in order to keep the wall friction to a minimum and avoid significant variations in density along the compact length. The die was tap-filled with the weight of powder which would give approximately identical compact lengths for each of the powders at a theoretical relative density of 1. The pressure was applied at the maximum strain rate attainable with the press and held at one of five fixed pressure levels for 600 sec, and then released. A simultaneous and continuous recording of displacement, thrust and time was taken, using a cine camera, by filming the displacement dial gauges, the thrust indicator and a stopclock. The values of the displacement, thrust and time were then obtained by reading the developed film frame by frame.

3. Results

The experimental results have been analysed using the basic compaction equation of Habberjam [5]:

$$P = Q\left(\frac{1}{\phi} - \frac{1}{\phi_0}\right).$$

For a powder compacted in a constant cross-sectional die and for the instantaneous application of the pressure the above equation may be written thus:

$$P = Q' l_\infty \left(\frac{1}{l_i - l_\infty} - \frac{1}{l_0 - l_\infty}\right).$$

In this equation Q' is that value of the modulus Q for instantaneous compact lengths l_i, l_0 is the extrapolated compact length at zero pressure and l_∞ the extrapolated compact length at infinite pressure.

Using the above equation a linear curve for the applied pressure P against $1/(l_i - l_\infty)$ can be fitted to the experimental results if the correct value of l_∞ is known. The correct value for l_∞ is obtained by a series of successive approximations until the best linear fit is obtained using Gauss' closeness of fit criterion (Worthing and Geffner [6]). The final estimate of l_∞ for the halite powders is used to plot P against $1/(l_i - l_\infty)$ in fig. 1. The agreement between the experimental results and the empirical equation gives an excellent linear fit for all the powders. Using these fitting curves the empirical constants l_0 and Q' may be determined.

The values of l_∞, l_0 and Q' were analysed statistically for variance between compacts pressed to different final applied pressures, of different size fractions, from different materials and repeated replicates. The significance of these variances tested against the residual variance was achieved using the variance ratio test (Brownlee [7]). Since each of the compacts for different materials were pressed from different powder masses the constants l_0 and l_∞ were reduced to packing density constants $1/l_0/L$ and $1/l_\infty/L$ before analysis, where L is the compact length for each powder mass at zero porosity. L is estimated from the density of the individual powder grains.

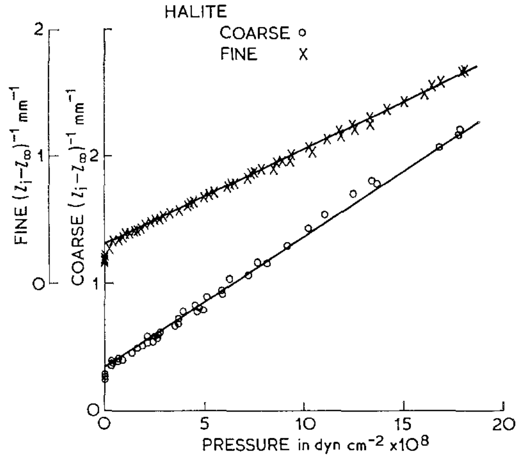

Figure 1 Variation of compaction with pressure for halite powders.

The analysis of variance demonstrates that none of the powders show any significant variance (5 % level) in any of the fitting constants due to differences in the final applied pressure or replication. The variance due to size fraction differences was significant at the 5 % level for the constant Q' in the case of quartz powder only. The constant L/l_0 showed no variance between materials, L/l_∞ has a significant variance between materials at the 1 % level and the constant Q' a significant variance between materials at the 5 % level. From this analysis of variance the constants may be summarised as in table II.

4. The Grain-Pore Model of Cold Compaction

This model has previously been developed by Habberjam [5]. A further extension is proposed here.

Consider the compact as a system of incompressible grains which can, however, undergo changes of shape. Let the rigidity be G and the yield stress Y. Ignoring any packing and fracture for a first approximation the reduction of the compact length, i.e. of the pore volume, must result from the change in shape of the grains. Assuming that G and Y are finite and constant all the work done on the system is used in distorting the grains. Such a system is equivalent, energetically, to one where the grains are incompressible and without rigidity and the pores are filled with a material of bulk modulus K and a yield stress Y', with zero rigidity. The model expresses K in terms of G and Y' in terms of Y on the basis that equivalence requires that the energy changes must be the same in both systems. Thus

$$K = G\frac{V_g}{V_p}$$

$$Y' = -\,Y\frac{V_g}{V_p}$$

where V_g and V_p are the total grain and pore volumes respectively. Considering only the elastic deformation of the model, the model yields the equation

$$\frac{P}{G} = l_\infty\left(\frac{1}{l_i - l_\infty} - \frac{1}{l_0 - l_\infty}\right)$$

where l_i is the instantaneous compact length at pressure P, l_∞ is the compact length at zero porosity, i.e. $V_p = 0$, and $l_\infty \equiv V_g$; hence $(l_i - l_\infty) \equiv V_p$, and l_0 is the compact length at zero pressure. Considering only plastic deformation the model yields the equation

$$\frac{P}{\frac{2}{3}Y} = l_\infty\left(\frac{1}{l_i - l_\infty} - \frac{1}{l_0 - l_\infty}\right).$$

For a combination of elastic and plastic deformation occurring simultaneously and instantaneously

$$\frac{P}{Q} = l_\infty\left(\frac{1}{l_i - l_\infty} - \frac{1}{l_0 - l_\infty}\right)$$

where

$$Q = \frac{\frac{2}{3}YG}{\frac{2}{3}Y + G}.$$

This is the stage reached by Habberjam in his development of the model but it can be extended further to the fracture of the grains.

For the same model the grains can now only

TABLE II Empirical constants

	Halite	Calcite	Hematite	Quartz coarse	Quartz fine
L/l_0	0.61	0.62	0.60	0.61	0.61
$L/l\infty$	1.13	0.92	0.83	0.87	0.87
$Q \times 10^8$ dyn cm^{-2}	3.44	2.35	1.90	4.45	2.32

be strained up to the point of fracture. The average strain energy within the system is $B^2/2G$ where B is particle breaking stress. The equivalence of the energy changes leads to the equation

$$K = \frac{\frac{1}{2}BV'_g}{V'_p}$$

where V'_g and V'_p are the total grain + unexcludable and *excludable* pore volumes respectively.

Consider a compact under a compaction pressure P and having an excludable pore volume V'_p at this pressure; an additional pressure $\mathrm{d}P$ will result in a contraction $\mathrm{d}v'_p$ in the pore volume given by

$$\mathrm{d}P = K\left(-\frac{\mathrm{d}v'_p}{V'_p}\right) = -\tfrac{1}{2}B\frac{V'_g}{V'^2_p}\,\mathrm{d}v'_p$$

or in terms of compact length,

$$\mathrm{d}P = -\tfrac{1}{2}B\frac{l_\infty}{(l_i - l_\infty)^2}\,\mathrm{d}l$$

where l_∞ is now the compact length at infinite pressure (but not necessarily zero porosity), i.e. $V_p' = 0$ and $l_\infty \equiv V_g'$ hence $(l_i - l_\infty) \equiv V_p'$. Integration between l_0 and l_∞ yields the relation

$$\frac{P}{\frac{1}{2}B} = l_\infty\left(\frac{1}{l_i - l_\infty} - \frac{1}{l_0 - l_\infty}\right).$$

If the original Habberjam definition of l_∞ is amended then for a combination of elastic, plastic and fracture deformation occurring simultaneously and instantaneously

$$\frac{P}{Q} = l_\infty\left(\frac{1}{l_i - l_\infty} - \frac{1}{l_0 - l_\infty}\right)$$

where

$$Q = \frac{\frac{2}{3}YGB}{\frac{2}{3}YB + GB + \frac{4}{3}YG}.$$

For the materials used in this investigation $G \gg Y$ and B, thus we can write

$$Q = \frac{\frac{2}{3}YB}{B + \frac{4}{3}Y}.$$

If the materials are divided into two broad classes of soft yielding materials and harder brittle materials various estimates of Q may be made. For a soft yielding material the particle breaking stress is likely to be either very much greater than, or of the same order of magnitude as, the yield stress:

$$\text{with } B \gg Y,\ Q = \tfrac{2}{3}Y\ ,$$
$$\text{with } B \approx Y,\ Q = \tfrac{2}{7}Y\ .$$

For a harder brittle material the yield stress is generally much greater than the particle breaking stress:

$$\text{with } Y \gg B,\ Q = \tfrac{1}{2}B\ .$$

5. Discussion

The values of L/l_0 are the extrapolated values of the compact packing density at zero pressure. For every powder the L/l_0 value is less than the experimental tap-filled loose powder packing density in the die before compaction. The tap-filled loose powder packing density varies widely from powder to powder. This is to be expected since the loose particle packing depends on the shape, bridging and orientation of the particles, which vary from powder to powder. In fact the loose powder packing density cannot easily be duplicated even with a standard die filling procedure. However within the experimental errors for all the powders the L/l_0 values correspond to the constant theoretical hexagonal packing density of 0.60. Thus the empirical parameter L/l_0 can be regarded as a constant for all the powders.

In the grain pore model the theoretical parameter l_0 is the length of the powder compact at zero pressure. This is a constant independent of the material since no conditions on the powder particles size and shape are specified. Consequently the grain-pore model indicates a theoretical constant packing density L/l_0 in agreement with the constant empirical parameter.

The values of L/l_∞ derived from the empirical description are, by definition, the density of the compact at infinite pressure. For a compact of incompressible but distortable grains and compressible pores L/l_∞ should be 1. In fact for all the powders except halite the value of L/l_∞ is less than 1. For halite the value of L/l_∞ is greater than 1. However, halite is highly compressible and as one of the above assumptions is that the materials are incompressible it is possible that this is the reason for the difference. The actual effect of compressibility cannot be quantitatively assessed as the transmission of hydraulic pressure in a powder compact is not clearly understood, although various estimates do exist (Train [8]). For a soft material like halite the grains are easily distortable and unlikely to fracture within the compact, consequently the application of a pressure to a halite powder compact reduces the porosity by the distortion of the grains. Extrapolating to extremely high pressures the voids will eventually be entirely eliminated.

The grain-pore model theoretical parameter l_∞ is identified with the grain + unexcludable pore volume. The unexcludable pore volume depends on the extent of particle fracturing within the compact, and the packing density L/l_∞ varies between materials with different degrees of particle fracturing. For the more rigid and brittle materials the porosity is more likely to be reduced by the grains fracturing and the voids being filled by the fracture products. Extrapolating the results to higher pressures the porosity is reduced but can never be entirely eliminated by this mechanism and there will always be some interstitial voids left unfilled and which can only be filled by distortion.

The density L/l_∞ correlates with both the rigidity modulus and the Youngs' modulus, see fig. 2. The moduli were calculated from the mean of the Voigt and Reuss estimates derived from the elastic constants of the material (Clark [9]).

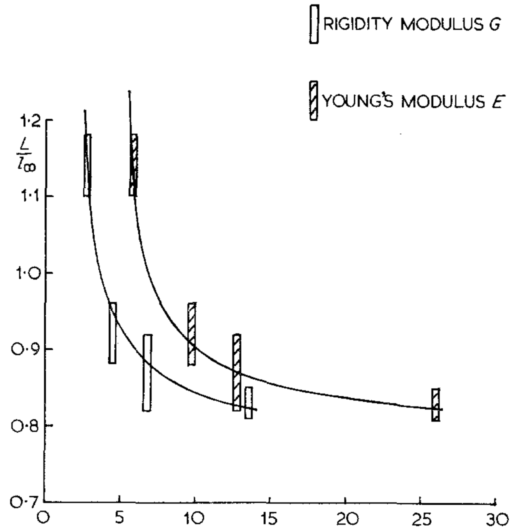

Figure 2 Variation of the compact density L/L_∞ with elastic constants.

The constant Q' in units of stress is obtained in the empirical description in such a way that it could be considered analogous to a stress-strain modulus. For the purposes of correlation the significance of the two sources of variance have to be compared: these are due to the material differences and the size fraction-material interaction indicated by the significant size fraction variance in the case of quartz.

We can examine the variation of Q' with material with respect to the modulus Q determined from the grain-pore model according to the most likely dominant mechanism of yielding or fracture. For halite Q is estimated as being between $\frac{2}{7}Y$ and $\frac{2}{3}Y$, for the other three materials Q is estimated as $\frac{1}{2}B$. This data is given in table III and plotted in fig. 3. The three materials halite, calcite and hematite show a good correlation between Q' and Q. The Q'-values for the quartz powders diverge from the estimates of Q.

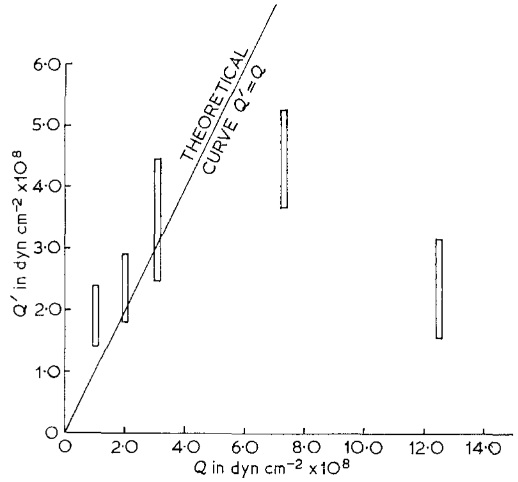

Figure 3 Comparison of the empirical pressure modulus Q' with the theoretical modulus Q.

However, a subsidiary experiment on the crushing of single powder particles showed no variation of the breaking stress (as defined by

TABLE III Pressing modulus

	Halite	Calcite	Hematite	Quartz coarse	 fine
$Q \times 10^8$ dyn cm^{-2}					
$\frac{2}{7}Y$	1.8				
$\frac{2}{3}Y$	4.4				
$\frac{1}{2}B$		2.0	1.0	7.5	12.5
$Q' \times 10^8$ dyn cm^{-2}	3.44	2.35	1.90	4.45	2.32

Hiramatsu and Oka [10]) with particle size for halite, calcite and hematite. Quartz showed a significantly higher breaking stress and a marked increase of magnitude from the coarse to fine size fraction, consequently it is likely that greater interparticle slipping occurs with fracturing and yielding taking a less dominant rôle in the compaction of the harder, more rigid and stronger quartz particles. Additionally the finer the particle size the less the interparticle forces (Huffine and Bonilla [11]) and therefore the easier the interparticle slipping.

The grain-pore model does not take into account any interparticle slipping and for a material such as quartz with marked interparticle slipping likely the value of Q' is consequently reduced. For the coarse quartz powder with a particle breaking stress of 15×10^8 dyn cm^{-2} Q' is reduced from an estimated 7.5×10^8 dyn cm^{-2} to 4.45×10^8 dyn cm^{-2}. The reduction for the fine quartz powder, particle breaking stress 25×10^8 dyn cm^{-2}, is from 12.5×10^8 dyn cm^{-2} to 2.32×10^8 dyn cm^{-2}. Empirically if the ratio of the particle breaking stresses is n the multiplying factors by which Q' is reduced are in the ratio $1/n^2$.

6. Conclusions

A single basic compaction equation has been developed which defines the deformation of the four mineral powders with widely varying mechanical properties. Although the dominant mechanism of compaction varies between soft and hard powders the same basic compaction equation fits the experimental data for both types of powders. The empirical parameters derived from the basic compaction equation are constant over the pressure range investigated and the extrapolated packing density at zero pressure is a constant independent of the powder compacted. The extrapolated packing density at infinite pressure correlates well with the elastic constants of the material. The pressing modulus Q' correlates with the theoretical modulus Q of the grain-pore model except for the quartz powder. In the case of quartz the mechanism of particle slipping reduces the pressing modulus. Quartz is the only powder of those investigated that shows a size-dependence of Q' between the size fractions of this investigation. Empirically the increase in the quartz particle breaking strength correlates with this size-dependence of Q'.

Acknowledgement

The work described formed part of a Ph.D thesis and was carried out in the Department of Applied Mineral Sciences, Houldsworth School of Applied Science, The University, Leeds.

The author wishes to acknowledge the assistance of Dr G. M. Habberjam of the University of Leeds.

References

1. G. BOCKSTIEGEL and J. HEWING, *Arch. Eisenhuttenes.* **36** (1965) 751.
2. R. W. HECKEL, *Trans. Met. Soc. AIME* **221** (1961) 611, 1001.
3. K. KONOPICKY, see R. KIEFFER and W. HOTOP "Sintereisen and Sinterstabl" (Springer-Verlag, Wien, 1948.)
4. A. R. COOPER and L. E. EATON, *J. Amer. Ceram. Soc.* **45** (1962) 97.
5. G. M. HABBERJAM, *Brit. J. Appl. Phys.* **15** (1964) 1233.
6. A. G. WORTHING and J. GEFFNER, "Treatment of Experimental Data" (Wiley, New York, 1948).
7. K. A. BROWNLEE, "Industrial Experimentation", 4th edition (HMSO, London, 1960).
8. D. TRAIN, *J. Pharm. Pharmacol.* **8** (1956) 745.
9. S. P. CLARK, "Handbook of Physical Constants", revised edition. Geological Society of America Memoir 97. (1966).
10. Y. HIRAMATSU and Y. OKA, *Int. J. Rock. Mech. Min. Sci.* **3** (1966) 89.
11. C. L. HUFFINE and C. F. BONILLA, *AIChEJour.* **8** (1962) 490

Received 13 April and accepted 18 May 1970