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The cold compaction of several mineral powders

Part 2 *The time-dependent deformation*

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Four mineral powders with widely different mechanical properties have been studied in prolonged cold compaction. An analysis of the time-dependent deformation has been attempted by splitting the finite strain into two components, one a linear function of time. A single compaction equation has been developed and applied to all four powders. The empirical constants of this equation have been examined, proposals concerning the mechanism of compaction under the prolonged application of pressure have been advanced and a correlation between the empirical parameters and the mechanical constants of the materials has been attempted. Suggestions for future work have been made.

1. Introduction

The time-dependent behaviour of powders cold compacted by a constant applied pressure has received little attention compared to the extensive literature on the cold compaction under increasing pressure (see the comprehensive review by Bockstiegel and Hewing [1]). Amar [2] made an experimental study of the room temperature pressing of several ionic materials which included investigating the creep of a compact under a constant applied pressure. One conclusion was that the creep of the compact increased with pressure and particle size.

Most of the work on the creep of compacted powdered materials is concerned with the pressure sintering of ceramics at elevated temperatures (Vasilos and Spriggs [3]) although Habberjam [4] did investigate hot-char briquetting and the effect of the prolonged application of pressure. He also proposed a rheological model for the compaction behaviour of hot char.

This investigation deals with the time-dependent behaviour in cold compaction of the four mineral powders described in a previous paper (Lawrence [5]). The experimental details of this present investigation were outlined in that paper and for further details the reader is referred to that paper.

2. Results

The experimental results have been analysed using the basic compaction equation of Habberjam [4] to define the finite strain of a compact.

For a powder compacted to a pressure P in a constant cross sectional die

$$P = Q\epsilon_{\text{total}} \qquad (1)$$

where ϵ_{total}, the total finite strain, is given by

$$l_\infty \left(\frac{1}{l - l_\infty} - \frac{1}{l_0 - l_\infty} \right)$$

and l, l_0 and l_∞ are the compact length, extrapolated compact length at zero pressure and extrapolated compact length at infinite pressure. Previously [5] the instantaneous component of the total finite strain has been defined by the equation

$$P = Q'\epsilon_{\text{i}} \qquad (2)$$

where ϵ_{i}, the instantaneous finite strain, is given by

$$l_\infty \left(\frac{1}{l_{\text{i}} - l_\infty} - \frac{1}{l_0 - l_\infty} \right)$$

and Q' is that value of the modulus Q for the instantaneous compact length l_{i}. Thus the time dependent finite strain ($\epsilon_{\text{total}} - \epsilon_{\text{i}}$) is given by

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$$l_\infty\left(\frac{1}{l-l_\infty}-\frac{1}{l_\mathrm{i}-l_\infty}\right)$$

and in Fig. 1 an example of this component of the finite strain, calculated from the experimental results, is plotted against time. This finite strain is shown in Fig. 2 for the point A on a idealized curve along with the instantaneous strain component. It is evident from examining such experimental curves that the change in strain with respect to time measured from the attainment of the maximum applied pressure is not linear, however at long times the curves do tend towards a linear form. It is proposed to split the time dependent strain into two components in such a way that one of them is a linear function of time. The total finite strain of the compact at the point A can then be represented by the sum of three components ϵ_i, ϵ_s and ϵ_t where ϵ_s, a steady state component fixed as a linear function of time, is given by

$$l_\infty\left(\frac{1}{l_\mathrm{s}-l_\infty}-\frac{1}{l_\mathrm{i}-l_\infty}\right)$$

and ϵ_t, a transient component, is given by

$$l_\infty\left(\frac{1}{l-l_\infty}-\frac{1}{l_\mathrm{s}-l_\infty}\right),$$

see Fig. 2.

Since ϵ_s has by design been made a linear function of time then $\dot{\epsilon}_\mathrm{s}$ will be a constant for a given curve and the strain is split into the two components ϵ_s and ϵ_t by obtaining an estimate of $\dot{\epsilon}_\mathrm{s}$ from the linearity of the strain curves at long times. It is assumed that for the approximately linear part of the curve

$$\dot{\epsilon}_\mathrm{s} \doteqdot \partial/\partial t\,(l_\infty/l - l_\infty),\ \text{i.e. } \dot{\epsilon}_\mathrm{s} = \dot{\epsilon}_\mathrm{total}\ .$$

For the deformation of newtonian liquids the deforming stress and the resulting strain-rate are related by a constant of proportionality, the viscosity, so by anology a constant η, with dimensions of viscosity, is derived from the relationship

$$\eta = P/\dot{\epsilon}_\mathrm{s}\ . \qquad (3)$$

From the estimate of $\dot{\epsilon}_\mathrm{s}$ the value of $l_\infty/(l_\mathrm{s} - l_\infty)$ at various times may be determined. Thus ϵ_s and ϵ_t may be calculated from the above expressions for ϵ_s and ϵ_t in terms of $l_\infty/(l_\mathrm{s} - l_\infty)$, $l_\infty/(l_\mathrm{i} - l_\infty)$ and $l_\infty/(l - l_\infty)$ which are known.

The value of $\dot{\epsilon}_\mathrm{s}$ has been fixed by reference to the long time linearity of the plot of $l_\infty/(l - l_\infty)$ against time. As time tends to infinity then it is clear from Fig. 2 that

$$\dot{\epsilon}_\mathrm{t} \rightarrow 0$$

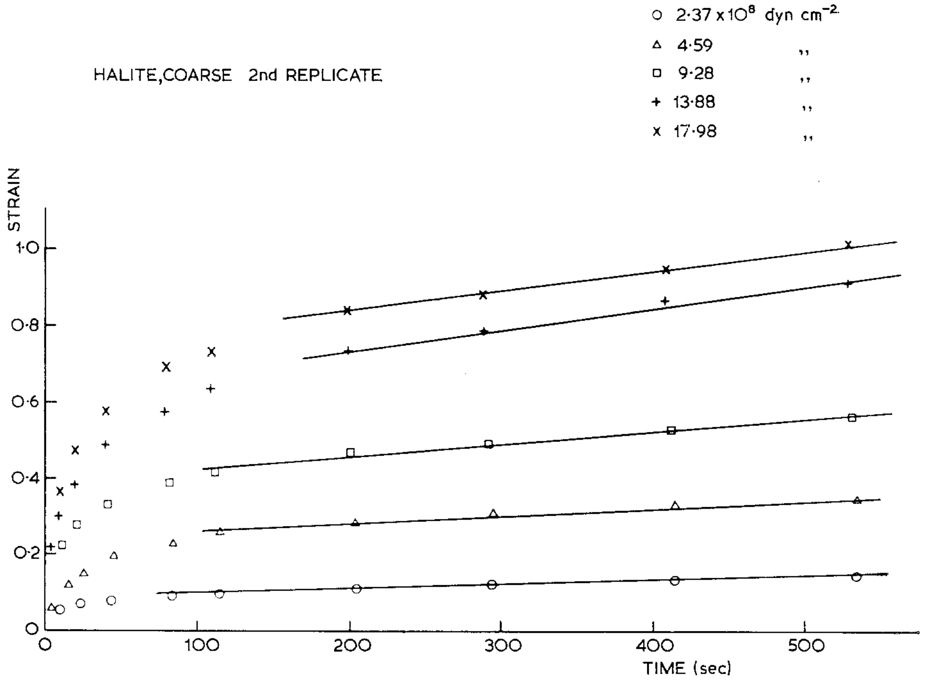

Figure 1 Experimental curve for the variation in strain with time from the attainment of maximum applied pressure for the halite coarse powder at five pressure levels.

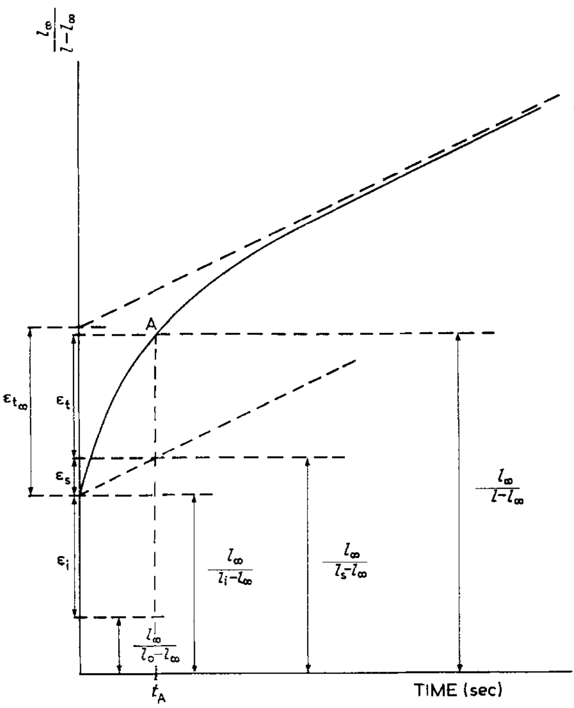

Figure 2 Theoretical curve for the variation in strain with time from the attainment of the maximum applied pressure.

and consequently at long times ϵ_t is no longer a function of time. For a given curve, ϵ_t tends towards a constant ϵ_{t_∞}, which can be identified with a particular value of the transient strain component given by

$$l_\infty \left(\frac{1}{l - l_\infty} - \frac{1}{l_s - l_\infty} \right)_{t\infty},$$

see Fig. 2.

Since $\epsilon_{t\infty}$ is a dimensionless constant derived at a constant compacting pressure P, we can further identify it with the ratio P/Q'', where Q'' is a constant with dimensions of stress. Thus

$$P = Q'' \epsilon_{t\infty} \, . \qquad (4)$$

From an examination of the trend of the values of ϵ_t an estimate can be made of $\epsilon_{t\infty}$ and a value of Q'' determined for each curve.

More generally ϵ_t can be expressed as

$$\epsilon_t = P/Q_t \qquad (5)$$

where Q_t is some function of time. Thus from Equations 4 and 5 we can write

$$\epsilon_{t\infty} - \epsilon_t = P \left(\frac{1}{Q''} - \frac{1}{Q_t} \right) = \frac{P}{Q_t''} \qquad (6)$$

where Q_t'' is some new function of time. Expressing $1/Q_t''$ differently thus

$$1/Q_t'' = 1/Q'' \; (Q''/Q_t'') \qquad (7)$$

The dimensionless ratio Q''/Q_t'' must satisfy certain conditions, namely:

$$\text{as time} \rightarrow \infty$$
$$Q''/Q_t'' \rightarrow 0$$

to fulfil Equation 6, and at time = 0, $Q''/Q_t'' = 1$, since at time = 0, $\epsilon_t = 0$ by definition and Equation 6 reduces to Equation 4 which is then satisfied. These conditions are met if,

$$Q''/Q_t'' = (\eta_t \, \dot{\epsilon}_t)/P \qquad (8)$$

where η_t has dimensions of viscosity and is defined at time = 0 as equal to $P/\dot{\epsilon}_t$. Then from Equation 6

$$\epsilon_{t\infty} - \epsilon_t = (1/Q'') \, \eta_t \, \dot{\epsilon}_t \, . \qquad (9)$$

Thus using Equation 9 we can now deduce a value for η_t at any time t since we can obtain $\dot{\epsilon}_t$ by numerical differentiation of ϵ_t, and the constants Q'' and $\epsilon_{t\infty}$ are known.

The values for η_t determined in this way can be plotted against the strain rate $\dot{\epsilon}_t$. An examination of such curves indicates that η_t tends towards a maximum value, say η_0, at very small values of $\dot{\epsilon}_t$ and tends towards zero at large values of $\dot{\epsilon}_t$. The shape of the curves suggest a relationship between η_t and $\dot{\epsilon}_t$ of the form

$$\eta_t = \eta_0 \, (\sinh^{-1} \dot{\epsilon}_t/k)/(\dot{\epsilon}_t/k) \qquad (10)$$

where k is a constant with dimensions of reciprocal time. An example of the ratio η_t/η_0 has been plotted against $\dot{\epsilon}_t$ in Fig. 3 and the curves fitted to the experimental points are derived theoretically from the $\sinh^{-1}$ curve of Equation 10. The fitting constant k, can be varied to alter the position of the theoretical curve along the strain-rate axis in order to fit the observed data at each pressure level. Consider the point on the $\sinh^{-1}$ curve when $\dot{\epsilon}_t/k = 1$, i.e. $\eta_t/\eta_0 = 0.88$. From this point on the curve we can obtain the best value of k for each pressure level. As can be seen the agreement of the observed data with the theoretical $\sinh^{-1}$ curve is reasonable.

The values of the constants η, Q'', η_0 and k were analysed statistically for variance between compacts pressed to different final applied pressures, different size fractions, different materials and repeated replicates. The significance of these variances tested against the residual variance was achieved using the variance ratio test (Brownlee [6]).

The analysis of variance demonstrates that none of the powders show any significant

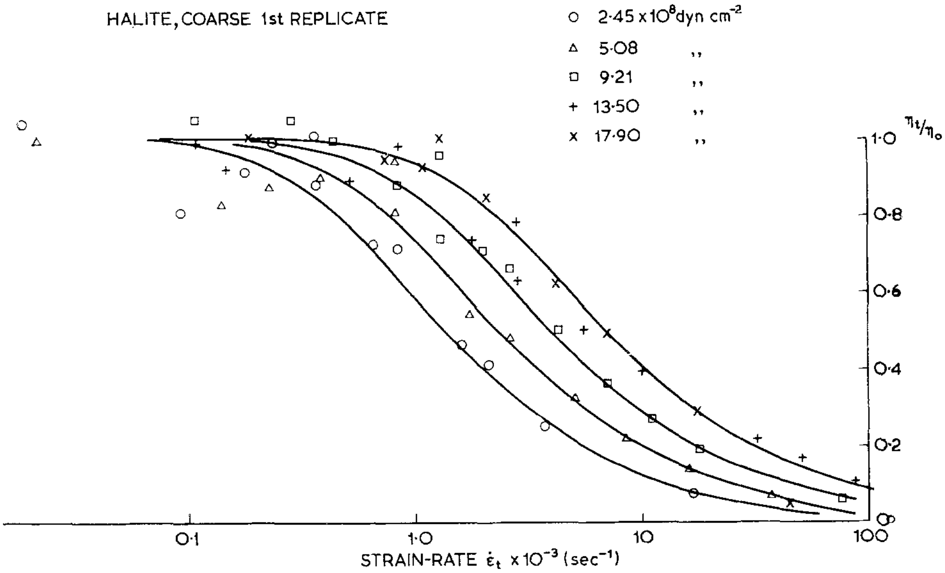

Figure 3 The experimental variation of η_t/η_0 with the transient strain-rate showing the trend towards unit value at very small strain-rates with the theoretical sinh curves superimposed on the experimental data.

variance (5 % level) in any of the fitting constants due to replication and that all the powders show a high level of significant variance (1 % level) with pressure for the constant k. The analysis does show some differences in the behaviour of the powders for the variation of the other constants with applied pressure. Q'', η and η_0 generally show no variance which is significant (5 % level) with the change in applied pressure. Exceptions do occur in the case of calcite powder for the constants η and Q'' and hematite powder for the constant η_0 where the significant variance reaches the 1 % level.

Though the variance due to the differences between powders does not reach the 5 % level of significance for any of the four constants it only just falls short and is worth a further examination. The analysis of variance between size fractions attains a high level of significance (1 % level) for all four constants for the quartz and calcite powders, for the constants Q'' and η_0 for halite and for none of the constants for hematite.

It is proposed that for the limited pressure range investigated the significant variance with applied pressure for the minority constants η and Q'' will not be considered and only mean values will be examined. This approximation does not involve large adjustments (but the size significance in η for calcite is lost) and consequently from the analysis of variance the constants may be summarized as in Tables I and II.

3. Discussion

The steady state viscosities of the constant η are very low, of the order of 10^{12} poise, when compared with the known room temperature creep viscosities of the original solid materials which range from 10^{17} poise for halite to greater than 10^{22} poise for calcite, hematite and quartz. However, the effective viscosity of the solid as a small grain of material in the environment of a compact may be considerably reduced and the viscosity determined from the empirical description of the compact deformation may be a reflection of this fact. The quantitative effect of the possible contributory factors, such as size and stress concentration, in lowering the effective value of the viscosity of the material can only be estimated from the relative values of the normal creep viscosity and the compact viscosity.

A structural factor of importance in the creep of rocks, particularly sedimentary rocks, is porosity and stress concentrations occurring around the pores will enhance the creep rate. This is typical of the situation in a powder compact and this phenomena may account for the low values of the compact viscosity η. In this event the viscosity for the softer easily deformed materials with a lower compact viscosity would be depressed far less than the harder materials with a high compact viscosity. The reduction of the halite viscosity by approximately five magni-

TABLE I Empirical fitting constants

		Halite		Calcite		Hematite	Quartz	
		Coarse	Fine	Coarse	Fine		Coarse	Fine
$\eta \times 10^{11}$	poise		35.8 ±5.7		65.0 ±34.0	62.0 ±20.8	135.0 ±42.7	61.1 ±10.4
$Q'' \times 10^{8}$	dyn cm^{-2}	16.2 ±2.9	30.5 ±6.4	19.4 ±6.9	25.7 ±7.1	29.0 ±3.1	78.5 ±20.5	34.7 ±3.2
$\eta_0 \times 10^{11}$	poise	2.3 ±0.3	3.0 ±0.7	1.6 ±0.2	2.3 ±0.2	2.5 ±0.7	6.7 ±2.0	2.7 ±0.2
$\eta_k \times 10^{12}$	poise		1.12	0.65	1.04	0.53	2.85	1.0

TABLE II Empirical fitting constant k at five pressure levels

	Halite	Calcite		Hematite	Quartz	
		Coarse	Fine		Coarse	Fine
				10^{-4} sec^{-1}		
P_1	1.6	4.0	1.8	3.2	0.8	2.2
P_2	4.0	4.6	4.2	7.6	2.4	4.2
P_3	7.6	9.4	10.4	12.4	4.4	7.0
P_4	12.6	17.0	11.6	27.8	4.4	9.4
P_5	15.8	30.6	16.6	36.0	4.4	22.0
	±1.80	±3.2	±3·2	±4.0	±3.2	±5.2

tudes and the reduction of the other materials by at least ten magnitudes would appear to confirm this view.

It is clear that further more closely controlled investigations are necessary to determine the more likely process controlling the steady state compact viscosity η.

At high strain-rates it is likely that air is entrained within the compact but is able to escape over a period of time during the prolonged application of pressure. The gradual escape of air from within the compact would give rise to a further deformation of the compact under a constant pressure as the voidage decreases. This would be a transient effect and it is proposed that the fitting constant Q'' is a measure of the magnitude of this transient strain at infinite time. Clearly from the plot of Q'' against the nominal yield stress, see Fig. 4, the more easily deformed the material the lower the Q'' value (i.e., the more entrained air) for the coarse size fraction. This is not observed in the fine size fraction where no significant difference is observed between the materials. However, it is worth noting that the Q'' value for the coarse size fraction, compared to the fine size fraction, is significantly lower for the soft materials halite and calcite, but is significantly higher for the hard material, quartz. More air is entrained in the coarse size fraction than the fine size fraction of the softer materials since the particles in both size fractions prefer to deform rather than fracture thus trapping air. Quartz is brittle and more likely to fracture, moreover the breaking strength has a marked size dependence (Lawrence [5]), with the coarse size fraction having a considerably lower breaking strength. Consequently the coarse size fraction of quartz will entrain less air than the fine size fraction which will result in a larger Q'' value as confirmed by the experimental value.

The non-newtonian viscosity which tends towards η_0 at low strain-rates is a result of time-dependent deformation occurring as the powder grains take up the volume occupied by the expelled air either by sliding grain over grain or by grain deformation. The viscosity η_0 correlates with the Q'' value, see Fig. 5.

The fourth empirical fitting constant k shows a significant variance with applied pressure and it was found that a constant with dimensions of viscosity η_k may be derived from the ratio P/k.

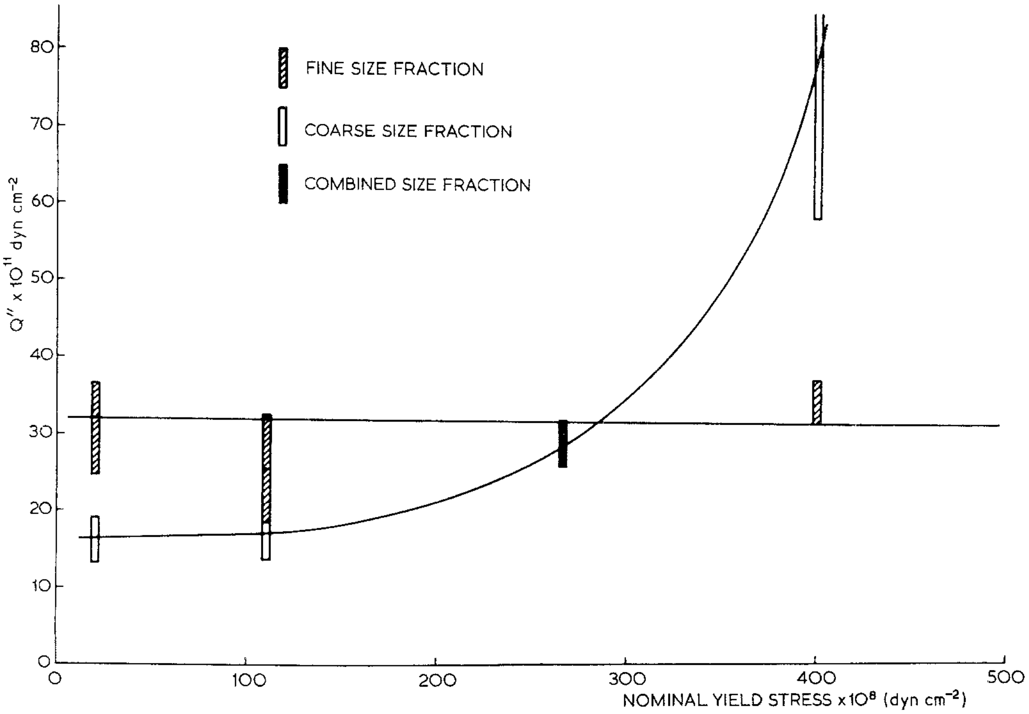

Figure 4 The correlation of Q'' with the nominal yield stress for coarse and fine size fractions.

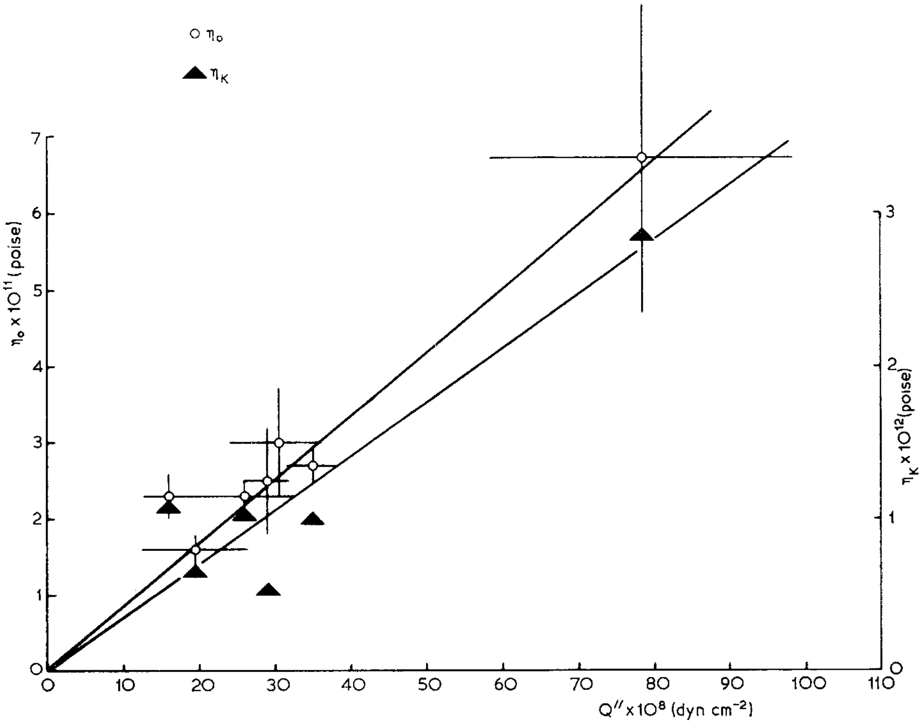

Figure 5 The correlation of η_0 and η_k with Q''.

This derived constant shows a similar correlation with Q'', see Fig. 5.

4. Conclusions

A compact creep has been identified in all four powders whether hard or soft. This time-dependent deformation or creep has been analysed by using a single time-dependent relation where the time-dependent strain of the compact

$$\epsilon_{\text{time}} = l_\infty \left(\frac{1}{l - l_\infty} - \frac{1}{l_\text{i} - l_\infty} \right)$$

is given by the equation

$$\epsilon_{\text{time}} = P \left[\frac{t}{\eta} + \frac{1}{Q''} \left(1 - \frac{\eta_0}{\eta_k} \sinh^{-1} \frac{\dot{\epsilon}_\text{t}}{k} \right) \right] .$$

The empirical parameters of this equation have been examined for correlation with the mechanical properties of the bulk material and the viscosity parameter η is open to a wide interpretation from a diffusion process to a stress concentration effect and clearly further experimentation is needed to determine with more certainty the controlling mechanism. It has been postulated that the parameter Q'', is due to the escape, from within the compact, of entrained air and, with η_0 and η_k, has been correlated with the nominal yield stress. As a result the hardness of the material can be considered as an indication of the air entrainment likely to occur in compaction at the strain-rates employed in this investigation.

The marked size-dependence of the quartz powder does not confirm the findings of Amar [2] that creep increased with particle size. It was found that the two viscosities associated with the creep in this case point to an increase in creep with a decrease in particle size. A further investigation involving wider size fractions of the other three materials should be considered with additional experimentation at temperatures above room temperature.

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