

Equation (4) has little in common with, and is in no way related to, any earlier theory of pressure-sintering.³ At present it is probably best regarded as a semi-empirical equation, although it is hoped to present a detailed analysis of its theoretical basis in a later publication.

2.2 The Integrated Form of the Equation

The results obtained in previously-reported experiments¹ were plotted as $-dV/dt$ versus $(P/\rho)^{2/3}$, which necessitated measuring gradients on a graph of pellet length versus time. This plotting process was lengthy and liable to error, and the gradients of densification plots could be compared only after the solid volume V_s of each pellet had been determined. The integrated form of Equation (4), derived below, provides a much simpler method of plotting experimental data.

Equation (4) may be rewritten

$$\frac{d}{dt} (1/\rho) = -Z \frac{\sigma}{l^2} \frac{D_M \Omega_s}{kT} \left(\frac{1}{\rho} - 1 \right)^{2/3}$$

so that

$$\int \left(\frac{1}{\rho} - 1 \right)^{-2/3} d \left(\frac{1}{\rho} \right) = -Z \frac{D_M \Omega_s}{l^2 kT} \int dt$$

or

$$\left(\frac{\rho}{P} \right)^{2/3} = \frac{2Z}{3l^2} \frac{D_M \Omega_s}{kT} \sigma t + \text{constant} \quad (5)$$

Thus a plot of $(\rho/P)^{2/3}$ versus time will yield a straight line with a gradient equal to two-thirds that of a plot of $(dV/dt)/V_s$ versus $(P/\rho)^{2/3}$.

3. APPARATUS

The general arrangement of the pressure-sintering apparatus is shown in Figure 1. The die-and-plunger as-

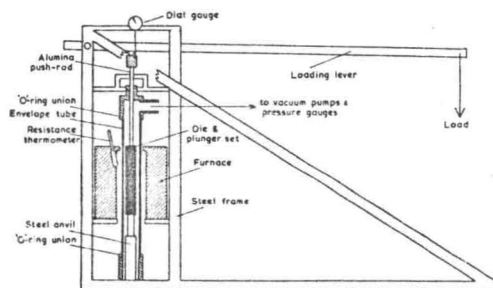


FIGURE 1
Schematic diagram of pressure-sintering apparatus.

sembly was arranged vertically within the steel pressing-frame and was enclosed within a sintered-alumina vacuum envelope tube. This in turn was encircled by an eight-element molybdenum-in-alumina furnace constructed in two halves, hinged together. When the furnace was opened and the envelope tube was removed, the die assembly was easily accessible. A miniature platinum resistance thermometer, mounted between two of the furnace elements, was coupled through a proportional controller to a saturable reactor wired into the power supply to the furnace. This equipment enabled the temperature to be

controlled to within one or two degrees centigrade of the set value.

The load was applied to the upper plunger using a 7:1 lever. This arrangement was preferred to the more customary hydraulic systems because the applied pressure could be calculated accurately and would remain essentially constant during the entire course of a pressing experiment. The displacement of the upper plunger could be measured at any time during pressing using a sensitive dial gauge mounted at the top of the steel frame: changes of displacement as little as one micron could be detected.

The die and plunger assembly (Figure 2) were made of

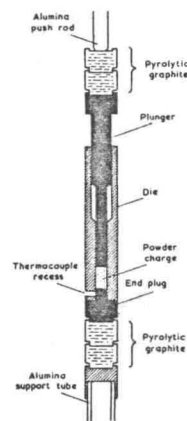


FIGURE 2
Die-set arrangement.

graphite (grade EY 110, Morganite Carbon Ltd, London S.W.11). The die body, 13 cm long and 2.5 cm O.D., was furnished with a 9.5 mm diameter bore through its lower half, closed at the bottom by a plug, and a 15 mm diameter bore through its upper half to act as an alignment guide for the plunger. The plunger was shaped as shown in Figure 2, with dimensions such that its upper part (15 mm diameter) engaged the die body before its tip entered the 9.5 mm bore. A recess was drilled in the die wall just below the position of the pressed compact, to accommodate the bead of the Pt-20%Rh/Pt-40%Rh thermocouple used to measure the die temperature.

When in position within the apparatus the die-and-plunger assembly was stacked with pyrolytic graphite spacers above and below (the total thickness of pyrolytic graphite at each end being 4 cm) to reduce loss of heat from the die by conduction. The stacked assembly stood upon a tubular alumina anvil and pressure was transmitted to the upper plunger by a 12 mm diameter alumina push rod, passing through an O-ring piston seal at the upper end of the vacuum envelope tube.

4. EXPERIMENTAL PROCEDURE

The alumina powder nominal particle size 0.3 μ m. (Linde A, Union Carbide Ltd, London W.1.) was first weighed out (about 0.9 g, sufficient to produce a pellet about 3 mm thick after densification) and poured into the die body. The die plunger was then placed loosely in position and the whole pressing assembly was placed within the apparatus. The envelope system was then evacuated (ultimate pressure less than 10^{-2} torr) and the furnace temperature was raised to about 350 C.

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