: or two degrees centigrade of

:o the upper plunger using a 7:1
was preferred to the more
tems because the applied pres-
accurately and would remain
g the entire course of a pressing
ment of the upper plunger could
during pressing using a sensitive
e top of the steel frame: changes
s one micron could be detected.
sembly (Figure 2) were made of

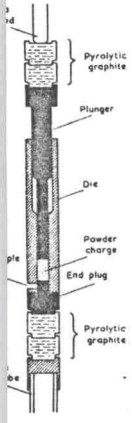

FIGURE 2
t arrangement.

Morganite Carbon Ltd, London
3 cm long and 2·5 cm O.D., was
diameter bore through its lower
m by a plug, and a 15 mm dia-
pper half to act as an alignment
ne plunger was shaped as shown
nsions such that its upper part
ed the die body before its tip
. A recess was drilled in the die
tion of the pressed compact, to
of the Pt–20%Rh/Pt–40%Rh
easure the die temperature.
hin the apparatus the die-and-
stacked with pyrolytic graphite
(the total thickness of pyrolytic
ing 4 cm) to reduce loss of heat
ion. The stacked assembly stood
anvil and pressure was transmit-
r by a 12 mm diameter alumina
gh an O-ring piston seal at the
n envelope tube.

NTAL PROCEDURE

nominal particle size 0·3 μm.
de Ltd, London W.1.) was first
g, sufficient to produce a pellet
densification) and poured into
unger was then placed loosely in
pressing assembly was placed
The envelope system was then
ssure less than 10^{-2} torr) and
e was raised to about 350 C.

After about 12 h outgassing, the temperature was raised to the value required for pressing, with a pressure of 5000 lb.in^{-2} applied to the plunger to ensure adequate particle packing during this period. After 20 min. at the final pressing temperature the load was removed for at least one hour, to allow the apparatus to reach thermal equilibrium without further significant densification of the pellet occurring. After that, the pressure required for final pressing was applied, and dial gauge readings (of plunger displacement) were taken at intervals throughout the remainder of the experiment. Readings were taken at 1-min. intervals during the initial period of rapid shrinkage, but at longer intervals (up to $\frac{1}{2}$ h) as the densification rate decreased.

At the end of the experiment the load was removed and the furnace was switched off. Having cooled, the pressed pellet was removed from the die and its thickness was measured with a precision micrometer. The pellet density was then determined by weighing in air and in absolute alcohol, the density of a high-quality specimen of single-crystal alumina being determined at the same time to act as a calibration. The final thickness and density of the pellet could be combined with the periodic measurements of plunger displacement during pressing, to obtain a series of pellet densities at regular intervals throughout the experiment. The data so obtained were then plotted on a graph of $(\rho/P)^{\frac{1}{3}}$ *versus* time. These plots were always straight lines for most of their length, indicating that the porosity factor in Equation (4) was being obeyed.

5. EFFECT ON DENSIFICATION OF CHANGES IN APPLIED PRESSURE

Equation (4) predicts that the shrinkage rate of a compact during pressure-sintering should be proportional to the applied stress, but it is difficult to test this prediction by comparing the shrinkage rates of different compacts to which different pressures are applied. This is because small differences in mean pore separation (which markedly affect shrinkage rates) arise amongst compacts even when great care is taken to reproduce the experimental conditions exactly, and it is difficult to allow for the effect because of the difficulty of obtaining accurate values of pore separation. For this reason the pressure-dependence of shrinkage rate is best determined by changing the pressure applied to a particular compact at some time during the experiment.

Experiments were attempted in which the initial applied pressure was 5000 lb.in^{-2}, which was then reduced to lower values as the experiment progressed. The results were confusing, because the shrinkage rate tended to fall much more rapidly than expected for some hours after the load had been reduced. The reason was not clear; possibly some grain growth occurred which had been inhibited under the higher stress. Whatever the reason, it was clear that stress-dependence comparisons could best be made in experiments in which the applied load was initially low, and then increased in stages. A further difficulty was the discovery that an increase of pressure applied to a compact with a relative density lower than about 0·9 resulted in an unexpectedly large increase in shrinkage rate: possibly the higher pressure imposed caused a change of microstructure through grain-boundary sliding or some other effect which did not occur at higher densities.

Figures 3 to 5 show the data obtained in two stress-dependence experiments, in which all the measurements were obtained after the compacts had achieved densities greater than 90%.

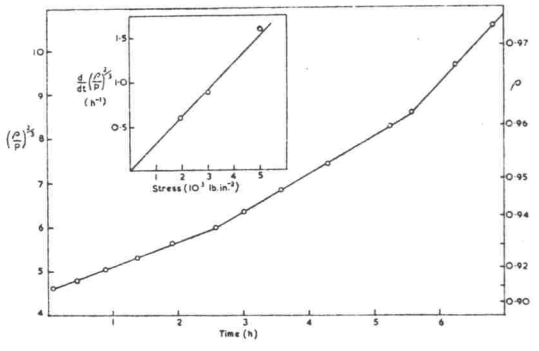

FIGURE 3
Densification data: 1st pressure-dependence experiment.

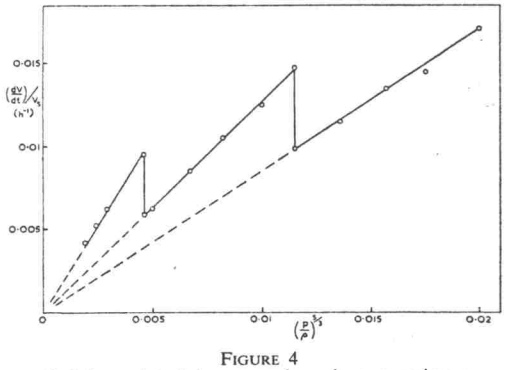

FIGURE 4
Shrinkage plot: 1st pressure-dependence experiment.

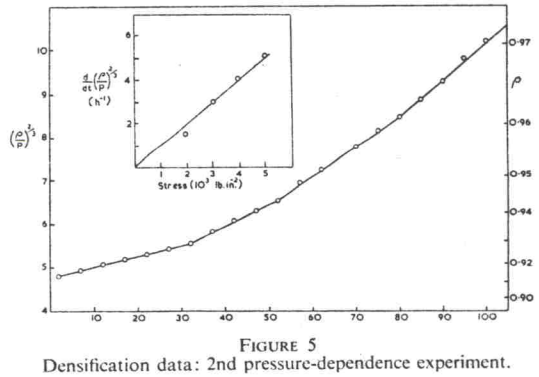

FIGURE 5
Densification data: 2nd pressure-dependence experiment.

The first of these experiments was performed at 1300°C. After initial pressing to reach the required starting density, followed by a period of 2 h to allow the apparatus to reach thermal equilibrium, a pressure of 2000 lb.in^{-2} was applied. After $2\frac{1}{2}$ h, the pressure was increased to 3000 lb.in^{-2}, and after a further 3 h to 5000 lb.in^{-2}. Regular measurements of plunger displacement were, of course, taken in the usual fashion throughout the experiment. A plot of plunger displacement against time displayed a discontinuity each time the load was changed, because of