

means of a proportional controller system. Constant loads could be applied to the die plunger by means of a weighted lever, and plunger displacement could be measured at any time during an experiment using a sensitive dial gauge.

3. EVALUATION OF THE CONSTANT Z

A value of the constant Z in Equations (1) and (2) is required before diffusion coefficients can be evaluated from pressure-sintering shrinkage data.

An attempt was made to estimate Z by assuming that the compact contained equal-sized equally-spaced pores, and that the average length of the vacancy diffusion path was about one quarter the pore separation. Using these assumptions, a value $Z \sim 20$ was obtained, but tentative evaluations of diffusion coefficients from some of the experimental data yielded values several orders of magnitude higher than tracer diffusion coefficients. It was therefore decided to evaluate Z by comparison with the geometrical constant in the Nabarro-Herring creep equation, by obtaining creep data with a pressure-sintered sample.

3.1 Experimental Procedure

A compact of Linde A alumina was pressed at 1304°C with an applied pressure of 5000 lb. in⁻², shrinkage data being obtained throughout the experiment. After the compact had cooled, its density was measured by weighing in air and in alcohol, so that the density at any time during shrinkage could be calculated. The pressing time was 7½ h, and the pellet produced was 3.19 mm thick, 9.595 mm diam., with a relative density of 0.990. A plot of $(\rho/P)^{1/3}$ versus time (cf. Equation (2)) was a straight line of gradient $3.25 \pm 0.15 \text{ h}^{-1}$.

The pellet was then inserted in a modified die, arranged so that it would experience no lateral constraint during pressing. The assembly was placed within the pressure-sintering apparatus and the temperature was raised to the value used previously for compacting the pellet (1304°C). After 1 h at this temperature, to allow the

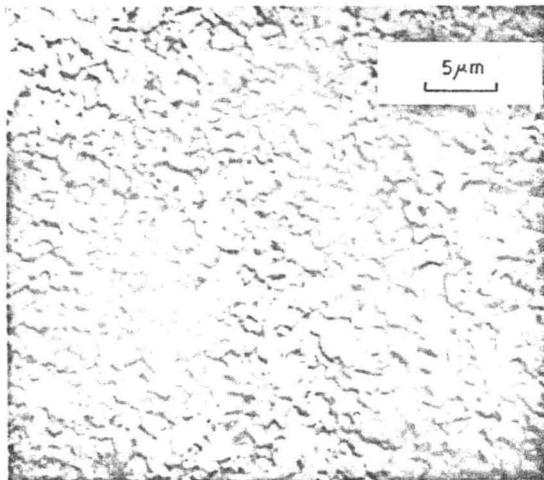


FIGURE 1
Etched section of pressure-sintered specimen used for creep experiment.

apparatus to attain thermal equilibrium, a pressure of 5000 lb. in⁻² was again applied to the plunger, and the change in length of the pellet was recorded during the following 3½ h. Because of the absence of restraint on the sides of the pellet, the observed deformation was presumably the result of Nabarro-Herring creep under a uniaxial compressive stress: this mechanism has been confirmed consistently for stressed dense alumina.^{3,4,5} The observed strain rate was 5.10^{-3} h^{-1} .

On completion of the creep experiment the pellet was sectioned and polished (using 6-μm and 1-μm diamond paste on successive lead laps). The diffraction patterns generated by pores lying below the polished surface were then sought using reflected light with a ×140 oil-immersion microscope objective. The pore separation was estimated to be 1 μm approximately.

The polished surface of the specimen was then etched in orthophosphoric acid (30 sec. at 300°C) and examined microscopically (×3000 magnification, reflected light). The etched grain boundaries were easily distinguished (Figure 1), and the grain size was estimated to be about 1.5 μm.

3.2 Results

From Equation (2),

$$\frac{d}{dt} \left(\frac{\rho}{P} \right)^{1/3} = \frac{2 Z \sigma D_M \Omega_s}{3 l^2 k T} \quad (4)$$

The theory of Nabarro-Herring creep, re-written as applying to a binary ionic polycrystal, predicts that the strain rate

$$\dot{\epsilon} = \frac{40 D_M \Omega_s \sigma}{3 k T L^2} \quad (5)$$

where L is the grain size. In the case where $d/dt (\rho/P)^{1/3}$ and $\dot{\epsilon}$ are measured under similar conditions of temperature and pressure, as in the present experiment, Equations (4) and (5) can be combined to give

$$Z = \frac{20}{\dot{\epsilon}} \left(\frac{l}{L} \right)^2 \frac{d}{dt} \left(\frac{\rho}{P} \right)^{1/3}$$

so that, by inserting the appropriate values for the various quantities, as obtained in the present experiment, one obtains

$$Z = 6 (\pm 3) 10^3$$

where the uncertainty is caused mainly by the uncertainties in l and L .

This experimentally-determined value of Z is unexpectedly high, and implies that the vacancies migrating from the pore surfaces follow very short diffusion paths. Because a given grain boundary must receive an equal flux of vacancies over all parts of its area if it is to collapse uniformly, it is further implied that a major part of the total vacancy flux within the pressed compact does not originate at pore surfaces. In other words, it seems possible that pellet shrinkage during pressure-sintering is greatly enhanced by change of grain shape by Nabarro-Herring creep (or by grain-boundary diffusion). The pore surfaces, however, are the only net source of vacancies, so that the shrinkage rate must be governed by the total pore surface area within the compact. The arguments leading to Equation (1) thus remain valid.

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