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H. A. MOHAMED
*Energy and Environment Division,
Lawrence Berkeley Laboratory,
University of California,
Berkeley, California 94720, USA*

Welding of magnesium oxide single crystals by H_3PO_4

Bicrystals of magnesium oxides have been fabricated by hot pressing [1, 2] from single crystals or by heating at very high temperatures [3]. For some purposes, however, it is useful simply to weld crystals with an appropriate binder. This note describes the welding of magnesium oxide single crystals by orthophosphoric acid.

Sheets approximately 2mm thick were cleaved on {1 0 0} faces from crystals purchased from the Tateho company. H_3PO_4 solution at 120° C was applied with a brush made of glass fibres to crystals heated to the same temperature. After cooling the crystals to room temperature, they were bound with Pt wire, as shown in Fig. 1. The values of θ vary from 5° to 45°. The crystals were

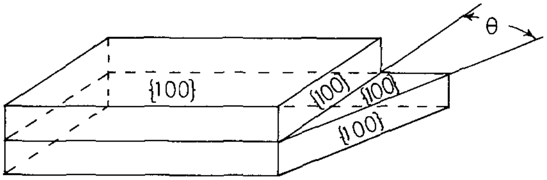

Figure 1 Schematic diagram of welding samples of magnesium oxide single crystals.

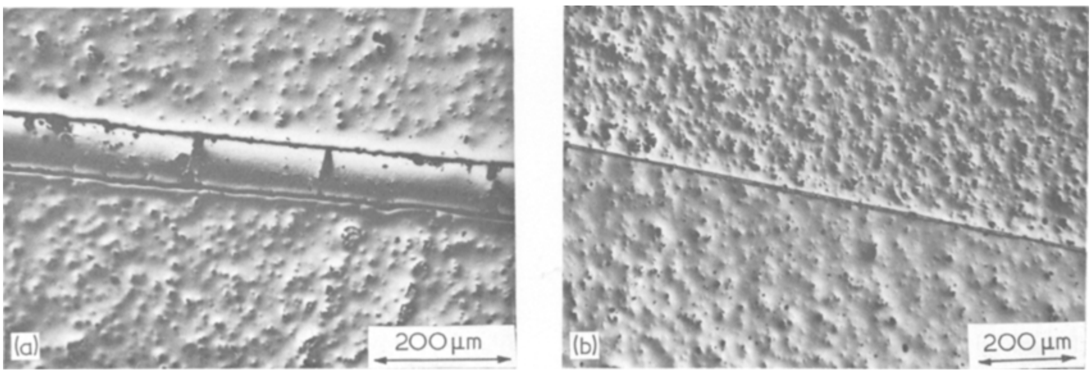

Figure 2 Photomicrographs of polished surface in a sample heated at 1400° C for 20 h in air. θ is 20°.

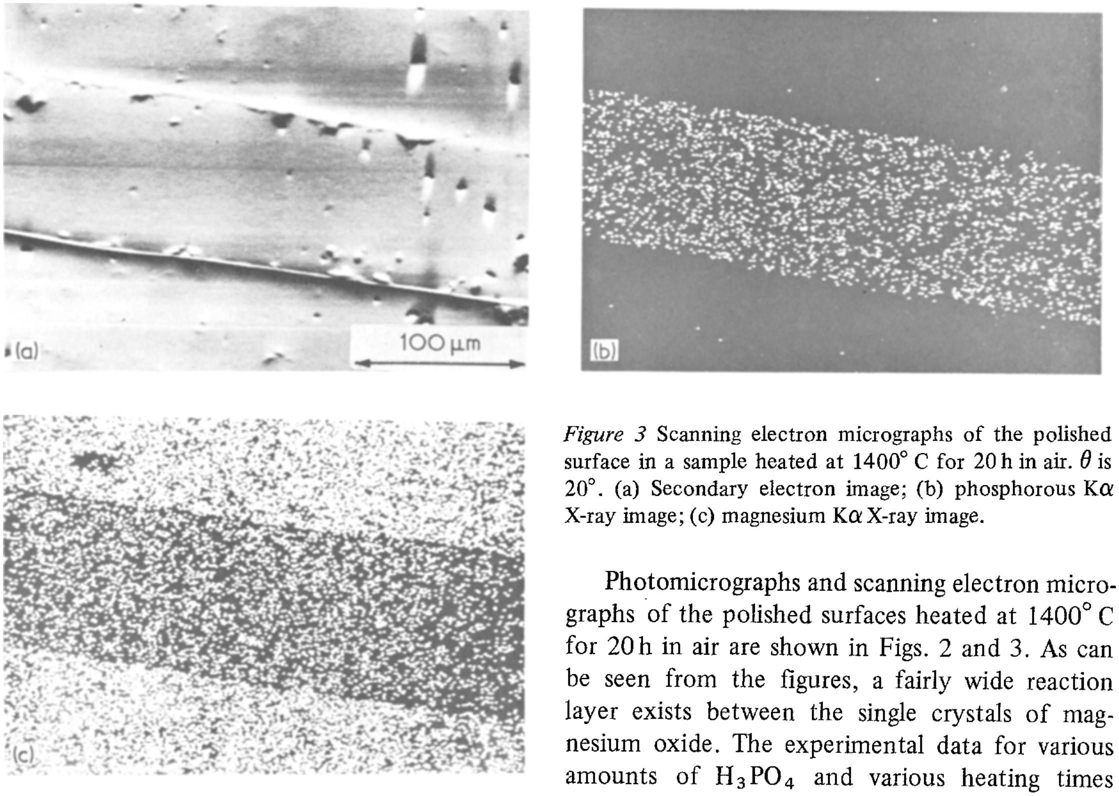

Figure 3 Scanning electron micrographs of the polished surface in a sample heated at 1400° C for 20 h in air. θ is 20°. (a) Secondary electron image; (b) phosphorous Kα X-ray image; (c) magnesium Kα X-ray image.

then heated at 1400° C in air for given periods of time. The resultant crystals were cut at right angles to the bonding faces with a diamond saw, and the cut surfaces finally polished with diamond paste. The polished surfaces were observed microscopically in order to identify the properties of the boundary between the two single crystals.

Photomicrographs and scanning electron micrographs of the polished surfaces heated at 1400° C for 20 h in air are shown in Figs. 2 and 3. As can be seen from the figures, a fairly wide reaction layer exists between the single crystals of magnesium oxide. The experimental data for various amounts of H_3PO_4 and various heating times showed that the thickness of the layer depended on the amount of H_3PO_4 and was independent of heating time. X-ray distribution images as shown in Figs. 3b and c indicate that Mg and P ions exist in the layer, but P ions are not present in magnesium oxide single crystals. Data line analysis, as shown in Fig. 4, indicated that there was no concentration gradient of P ions in the

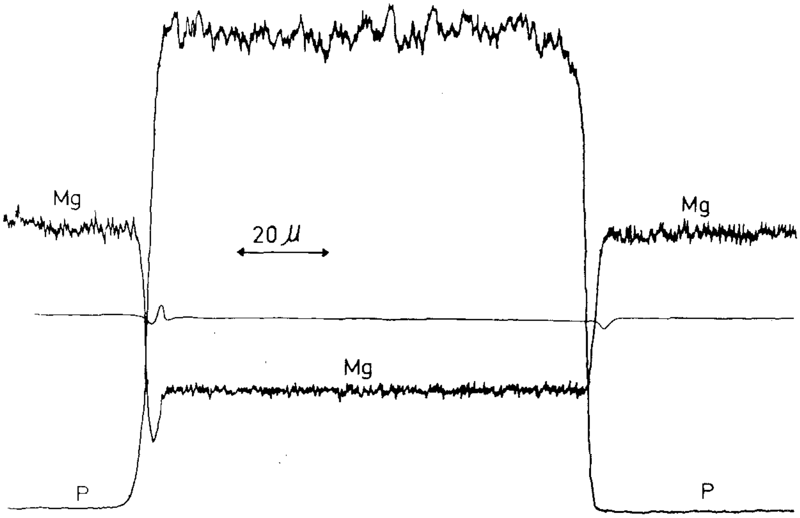

Figure 4 Line analysis of magnesium and phosphorous.

magnesium oxide single crystals, nor a concentration gradient of Mg ions in the reaction layer. This may mean that P and Mg ions do not form a solid solution with magnesium oxide and the reactant, respectively. Results of electron probe microanalysis of the specimens prepared with various ratios of magnesium oxide to P_2O_5, showed that the reactant had the approximate composition $3MgO{\cdot}P_2O_5$, and the composition was constant for heating times from 1 to 20h.

According to the phase diagram of the $MgO{\cdot}P_2O_5$ system [4], $3MgO{\cdot}P_2O_5$ is liquid at 1400° C. The wetting test of magnesium oxide single crystals by $3MgO{\cdot}P_2O_5$ at 1400° C showed spreading of the liquid on the magnesium oxide; $3MgO{\cdot}P_2O_5$ liquid completely wets the surface of magnesium oxide. The preferred interpretation of the welding is that $3Mg{\cdot}P_2O_5$ acts as a binder to connect the two single crystals of magnesium oxide.

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Y. MORIYOSHI
M. TSUTSUMI
S. MATSUDA
T. IKEGAMI
H. YAMAMURA
S. SHIRASAKI
National Institute for Researches in
Inorganic Materials, Kurakake,
Sakura-Mura, Niihari-Gun
Ibaraki, 300–31, Japan.

Effect of precursory solution anion on sintering of magensium aluminate

Highly homogeneous and reactive magnesium aluminate powder can be prepared by spray drying various precursor solutions followed by calcining [1]. A conventional ball-milling step was eliminated. The magnesium aluminate formed by using the precursor of $[MgSO_4 \cdot 7H_2O + Al_2(SO_4)_3 \cdot 18H_2O]$ can be sintered to a high density at a lower temperature than the temperature used to sinter conventional magnesium aluminate powder. The purpose of this note is to show the effect of sulfate precursor solution anion on the sintering property of magnesium aluminate.

Reagent grade $Mg(NO_3)_2 \cdot 6H_2O$, $Al(NO_3)_3 \cdot 9H_2O$, $MgSO_4 \cdot 7H_2O$ and $Al_2(SO_4)_3 \cdot 18H_2O$ were used to make the precursor solution. Precursor solutions (25 wt % of nitrate) of magnesium and aluminium nitrate $Mg^{2+}/Al^{3+} = 1:2$ were prepared by mixing 25 wt % $MgNO_3$ and $Al(NO_3)_3$ aqueous solutions which were obtained by dissolving $Mg(NO_3)_2 \cdot 6H_2O$ and $Al(NO_3)_3 \cdot 9H_2O$ in de-ionized water. Other precursor solutions with 0.5:0.5 mole ratios of $[MgNO_3 \cdot 6H_2O + 2Al(NO_3)_3 \cdot 9H_2O]$ to $[MgSO_4 \cdot 7H_2O + Al_2(SO_4)_3 \cdot 18H_2O]$ and 1:1 of $MgSO_4 \cdot 7H_2O$ to $Al_2(SO_4)_3 \cdot 18H_2O$ were prepared in the same way as a mixed nitrate solution. The various precursor solutions were spray dried in a laboratory spray drier. The spray drier was held at 390° C. Spray-dried powders were calcined at 1150° C for 1 h. A single phase of $MgAl_2O_4$ was obtained. Compacts formed by isostatic pressing the calcined powder to 20 000 p.s.i. were sintered at 1450° C for 20 h. The specific area of the powders was determined by nitrogen adsorption using the B.E.T. technique. Sintered density was measured by mercury immersion. Spray-dried sulfate powder was further investigated by differential thermal and thermogravimetric analyses.

Fig. 1 shows the effect of precursor sulfate solution on the sintered density of $MgAl_2O_4$ and surface area of calcined spray-dried powder. Results indicate that the sintered density of $MgAl_2O_4$ and the surface area of the calcined powder were increased as the precursor sulfate solution anion was inceased. Bratton shows that sintering of high purity $MgAl_2O_4$ is governed by a volume diffusion equation [2]: