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ELECTRON MICROSCOPY STUDIES OF GRAIN BOUNDARY SEGREGATION IN $\alpha$ Al$_2$O$_3$

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Solute segregation at grain boundaries is a common phenomenon in ceramic systems. (1) Under certain circumstances it can lead to a reduction in the boundary migration rate by an impurity drag mechanism. (2) This favours pore attachment to grain boundaries during the later stages of sintering and allows high densities to be achieved. (3) To interpret the sintering data one needs to know the spatial distribution of the additives, and how the additives affect the defect structure of the material. Spatial distributions can be measured by Auger electron spectroscopy, but this technique cannot distinguish additives present as separate second phase from those in solid solution, and is restricted to materials that fracture intergranularly.

In alumina, the standard sintering additive is MgO, but TiO$_2$ can be used (4) where hardness of the sintered material is important. We have studied Al$_2$O$_3$ doped with 0.1 w/o TiO$_2$. Thin foils were prepared by ion beam milling, and examined by 1) conventional electron microscopy to investigate the grain and pore structure of the material, 2) high resolution lattice imaging to test for the presence of intergranular phases at grain boundaries, and 3) X-ray microanalysis to probe the Ti concentration profile across grain boundaries and near pore surfaces.

Fig. 1 shows a low magnification image of the grain structure of the $\alpha$Al$_2$O$_3$ matrix. The framed trijunction is imaged at a higher magnification in Fig. 2. One of the three grains is out of contrast. The two remaining grains show lattice fringes leading right up to the edge-on boundary, thus showing that no intergranular phase wider than 3Å was present.

Fig. 3 shows X-ray spectra recorded with a 100Å diameter probe. The upper trace is the sum of five spectra taken at different points along a boundary with the probe on the boundary, and the lower trace is the sum of five spectra taken at points 300Å away from the boundary. The peak in the boundary trace corresponds to 1 w/o Ti concentration of Ti, as determined by comparing the Ti peak height to the Al peak present in a different part of the spectrum (not displayed). The absence of the Ti peak in the lower trace shows that within the matrix, the Ti concentration is below the detectable minimum of about 0.2 w/o. Investigation of a pore surface revealed a large concentration of impurities (Ta, Fe, Ar) that are probably due to ion milling contamination, but no trace of Ti.

We therefore conclude that in the present sample doped with 0.1 w/o TiO$_2$ segregation of Ti occurred within about 300Å from grain boundaries, but there was no separate intergranular phase. The small width of the enriched layer lends support to the theoretical width of the Al$_2$O$_3$ grain boundary derived from a variety of sintering data (5), but disagrees with the 5000Å wide enrichment layer derived by analysis of Ti solubilities in Al$_2$O$_3$ of different grain size. (6)

These findings form part of a more comprehensive study on the sintering behaviour of alumina being carried out by M.P. Harmer, R.J. Brook and E.W. Roberts at the University of Leeds, England. Financial support from NSF Contract DMR 77-24022, access to facilities provided by the Division of Basic Sciences, U.S. Department of Energy, and encouragement from Professor G. Thomas are gratefully acknowledged.

References.
Fig. 1  Low magnification bright field image of TiO₂ doped with Al₂O₃.

Fig. 2  Lattice image from the framed area of Fig. 1. Note the boundary region at left where no intergranular phase wider than 3Å appears to be present.

Fig. 3  X-ray spectra from along a boundary. Upper trace: probe on boundary.
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