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DETECTION OF SOLUTE SEGREGATION AT GRAIN BOUNDARIES

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Studies of grain boundary segregation in metallurgical systems are tradition-ally based upon the premise that grain boundaries are more likely sites for solute atoms than their surrounding grains. This idea is manifested in experimental studies which distinguish the solute concentration at boundarie from that of grain interiors using various spectroscopic techniques, includin more recently, energy dispersive X-ray analysis in TEM/STEM instruments. A typical study therefore usually consists of spot or line scans across a grain boundary plane in order to detect concentration gradients at the boundary region. It has also been pointed out that there are rather severe problems in quantitatively determining the absolute solute concentration within the grain boundary, and data correction schemes for this situation have been proposed.[1]

The present paper is concerned with an alternative study of grain boundary segregation where the distribution of solute atoms along the boundary plane (as opposed to that across the boundary plane) is sought. The interest here is to establish whether or not a relationship exists between the structural defect configuration of the boundary plane and site preference for solute segregation.

As an initial attempt in this research, controlled aluminum bicrystals were prepared and their boundaries subsequently doped with zinc according to the scheme illustrated in Fig. 1. Near-coincidence boundaries were selected for analysis because of their relatively large-period defect structures which maximize the probability of detecting an in-plane solute variation at the boundary. Discs from the boundary region were then spark-cut and electro-chemically thinned to electron transparency. In order to overcome the problems of thickness variations, only those boundaries which ran parallel to the edge of the foil (through regions of constant thickness) were analyzed by EDS in a Philips EM 400 TEM/STEM, equipped with an LaB_6 gun. The electron probe size was 50Å at the specimen surface and spot readings were taken at intervals of a few hundred angstroms along the boundary plane over a total distance of a few microns. Working STEM magnification was 100,000 x and a 150 second count time was employed for each data point. An example of the output from a high Zn region is shown in Figure 2.

The results of the study for a single boundary are presented in Fig. 3, whic shows the Zn/Al count rate ratio as a function of distance along the boundar plane. Although quantitative corrections have not been performed, a relati. change in Zn concentration of approximately 20% is evident at periodic intervals of approximately 4000 angstroms. Diffraction analysis of the bicrystal indicates that the two grains are misoriented by a 58.73° rotatio about the direction $[1.0 \quad -1.28 \quad 1.11]$. This is very close to a $\langle 111 \rangle /60°$ CSL boundary which characterizes a coherent twin. It would not be unusual therefore to observe a 4000Å periodicity in the defect structure for this boundary and further work is underway to correlate structural studies with these analytical results.

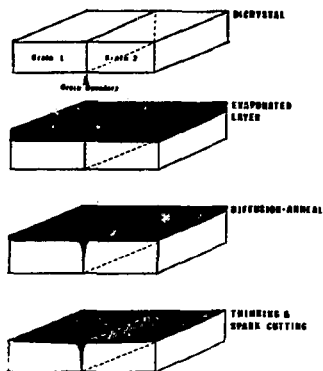1. P. Doig and P.E.J. Flewitt, J. Microscopy 110, 107 (1977).

Figure 1 – Schematic of specimen preparation method. The Al bicrystal is first coated with an evaporated layer of solute, then subjected to a diffusion anneal. Foils are prepared from the edge-on doped boundary region after surface residue is removed.
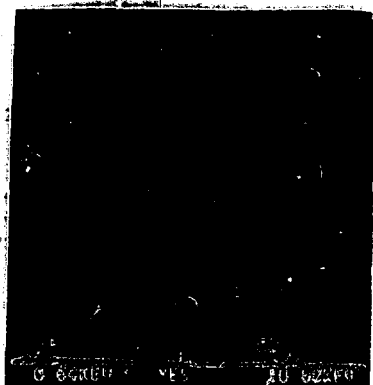

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Figure 2 – Typical EDS output from the resultant specimen; 150 second count time, 50Å focused spot size, over the grain boundary region. The Cu signal arises from systems background in the EM 400.
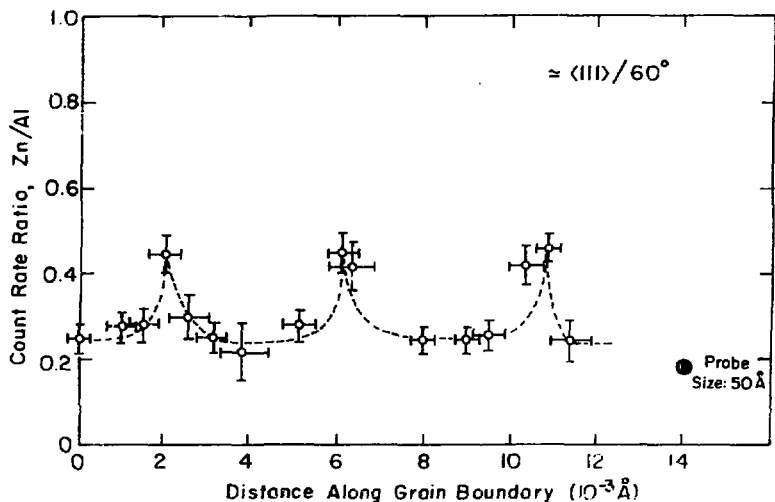

Figure 3 – Summary plot of the composition data expressed in count rate ratio as a function of distance along the grain boundary plane. The bicrystal has a boundary structure very nearly characterized by a ⟨111⟩/60°, axis/angle pair. The periodicity of the solute segregation peaks is ~4000Å and this agrees very well with the anticipated periodicity of the defect structure for deviations from a coherent twin boundary.

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