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DETERMINATION OF THE STRUCTURAL ASPECTS OF FAVORED GRAIN

BOUNDARY REACTION SITES

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Of the techniques which have been employed in the study of grain boundary precipitation reactions, none have demonstrated sufficient resolution to directly reveal the structural characteristics of active nucleation sites. This information is vital since many existing interpretations of conventional TEM data are in conflict.^{1,2} The present paper describes an application of high resolution electron microscopy which distinguishes atomic level details in both the boundary and growth front regions of grain boundary precipitates, and indicates the structure and mechanisms responsible for enhanced reaction kinetics.

Experiments were performed on an Al-9.5 at. % Zn alloy aged for 30 mins. at 180°C to encourage heterogeneous nucleation at grain boundaries. Two examples of early stage grain boundary precipitates are shown in Fig. 1, both of which have been nucleated in the lower grain. It is seen that in (b) the precipitate is much more sharply faceted than in (a). Notably the orientation of the grain boundary plane is very near to that of the close-packed [111] matrix planes of the lower grain for precipitate (b), whereas the boundary is sharply inclined to [111] for precipitate (a).

Lattice images corresponding to the boundary region for both of these precipitates are shown in Fig. 2 (a) and (b) respectively. The images were formed under tilted illumination conditions using a strong excited $\bar{1}\bar{1}1$ reflection. Measurements of both images reveal a larger fringe spacing within the precipitate region, indicative of a higher Zn content; however

a variation in spacing is observed for (a) as the boundary is approached from either side. This result suggests the presence of a composition gradient across the boundary. To the same experimental accuracy ($\pm 0.5\%$), no fringe spacing variation was detected on either side of the boundary for precipitate (b).

The structural details responsible for this difference are revealed in the enlargements shown in Fig. 3. It is found that the two primary facets in the growth front of the precipitate in Fig. 1 (b) are comprised of monolayer ledges which maintain the macroscopic facet orientations. These orientations are: (a), parallel to the close-packed planes of the grain in which nucleation occurred, and (b), parallel to the close-packed planes of the grain into which growth is occurring. Such ledges are most likely responsible for rapid solute redistribution and growth, which prevent a gradient in composition from becoming established. The orientation of the close-packed planes for the precipitate in Fig. 1 (a) does not favor the propagation of such ledges and the reaction is correspondingly more sluggish.

Optical microdiffractograms of the same precipitate (Fig. 1(b)) further confirm the extent of decomposition. The outermost diffracted intensities represent the smaller matrix fringe spacings which are parallel to the larger spacing precipitate fringes at the grain boundary (Fig. 4(a)), and rotated within the contiguous grain at the reaction front (Fig. 4(b)). The arrowed large periodicity reflection is due to Zn, arising from the (0001) basal planes within the precipitate regions, and indicating that equilibrium composition has been attained. A similar analysis of the precipitate in Fig. 1(a) shows no evidence of Zn. Rather the orientation and spacing change of lattice planes from matrix to precipitate in Fig. 2(a) indicates a state of rhombohedral distortion characteristic of the intermediate phase.³

It is concluded that boundary regions oriented parallel to close-packed martix planes offer a kinetic advantage in the growth of heterogeneously nucleated precipitates. The enhanced kinetics are brought about by a microledge mechanism.

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FIGURE CAPTIONS

- Fig. 1. Low magnification images of grain boundary precipitates in an Al-9.5 at .% Zn alloy aged 30 mins. at 180 C. Note that the grain boundary plane is approx. normal (a) and parallel (b) to the close-packed planes of the lower matrix grain.
- Fig. 2. Lattice images of the grain boundary vicinity corresponding respectively to Fig. 1 (a) and (b) above. Measurements of fringe spacing variations indicate a solute gradient within both matrix and precipitate for (a), but none for (b).
- Fig. 3. Details of the growth front in Fig. 1 (b) showing monolayer ledges in both facets.
- Fig. 4. Optical microdiffractograms of (a) the grain boundary region and (b) the growth edge of the precipitate in Fig. 1 (b).

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Fig. 1(a) and (b)

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Fig. 2(a) and (b)

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Fig. 3(a) and (b)

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Fig. 4(a) and (b)