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Publication Date
1974-02-01
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February 1974

Prepared for the U. S. Atomic Energy Commission
under Contract W-7405-ENG-48
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TRANSMISSION ELECTRON MICROSCOPY STUDIES OF GRAIN BOUNDARY PRECIPITATION IN SPINODALLY DECOMPOSED CU-NI-Fe ALLOYS

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In many alloy systems the grain boundaries play an important role in controlling properties but little is known about the mechanism of grain boundary precipitation reactions. Transmission electron microscopy and diffraction are necessary to distinguish reactions such as discontinuous precipitation and preferential coarsening. The present paper describes current progress on the Cu-Ni-Fe system which is known to be embrittled at the grain boundaries (1). Two alloys are considered, having their compositions on a binary section through the Cu-Ni-Fe ternary with terminal values at the Cu corner and the point Ni0.7Fe0.3. Alloy A (51.5 at% Cu-33.5 at% Ni-15 at% Fe) lies very near to the center of the miscibility gap (2). Phase identification in the electron micrographs is facilitated by comparison with alloy B (69.3 at% Cu-19.4 at% Ni-11.1 at% Fe) of asymmetrical composition.

The microstructure shown in Fig. 1 results from aging alloy B within the spinodal (650°C) for 10 hrs. Enhanced coarsening of the spinodal structure in the vicinity of the grain boundary is immediately evident e.g. the particle size is ~1300 Å compared to that within the matrix (~600 Å). Continued aging of alloy B (1000 hrs.) causes the coarsened structures to lose coherency, marked by the formation of interfacial dislocations to accommodate lattice mismatch, as shown in Fig. 2. The large particle near the center of the micrograph exhibits enhanced growth at the expense of nearby matrix depletion of the Ni-Fe rich phase. By selective excitation of appropriate reflections across the boundary it is shown that the depletion occurs only within the grains containing the augmented particles.

Fig. 3 shows a low magnification image of a large cellular constituent obtained after aging alloy B for 500 hrs. (750°C). Optical micrographs of similar structures are plentiful but give no indication of their actual origin. In this study the combined use of imaging and selected area diffraction has shown the relationships of the cell with its contiguous grains. As indicated in the figure, regions marked A and B are in the same orientation, while C is not; hence a grain boundary is located only at the leading edge of the cell. These findings indicate that the precipitates are not formed by heterogeneous nucleation, but rather from a selective migration of the grain boundary, proving that the mechanism of the grain boundary reaction is not the discontinuous one. A higher magnification image of a cell segment produced in alloy A (aged 1000 hrs. at 650°C) is shown in Fig. 4. The particles are semicoherent and contrast analyses show that the interfacial dislocations have \( \mathbf{b} = \frac{\mathbf{a}}{2} \langle 110 \rangle \) i.e. identical to those found within the matrix. Thus the process of coarsening must be identical for grain boundary and matrix particles, but the rates at the boundary are much greater due to increased diffusivity and potential for reducing the interfacial energy.

In conclusion it has been shown that quantitative electron microscopy and diffraction are essential for understanding the mechanisms of boundary precipitation and growth reactions.

The author gratefully acknowledges the continued guidance and encouragement of Professor G. Thomas. This research is sponsored by the United States Atomic Energy Commission through the Lawrence Berkeley Laboratory.

Fig. 1. B.F. micrograph showing enhanced coarsening at grain boundary in alloy B aged 10 hrs. at 650°C; [110] foil.
Fig. 2. Matrix depletion and accelerated growth of semicoherent particles at boundary; alloy B, 1000 hrs at 650°C. Zone [110], (B.F.).
Fig. 3
Cellular structure produced in alloy B aged for 500 hrs at 750°C. Areas marked M and N are in same orientation, separated from O by a grain boundary, (B.F.).
Fig. 4. B.F. image showing semicoherent particles within grain boundary constituent of alloy A aged 1000 hrs at 650°C;