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ELECTRON MICROSCOPY OF SPINODAL ALLOYS

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Transmission microscopy, electron diffraction and scanning microscopy were used to study directly the microstructure of spinodal alloys and to correlate the fracture mode of aged tensile specimens with the microstructural changes. The kinetics of spinodal decomposition for two copper-nickel-iron alloys were experimentally studied by heat treating specimens as described by Butler and Thomas (1). The aging times inside the spinodal ranged from one minute to 1000 hours. Thin foils for transmission electron microscopy were prepared by first using a chemical polish containing nitric, hydrochloric and acetic acids and then by electropolishing in a chromic oxide-acetic acid solution. The average interparticle spacing for each aging time was determined by direct measurements from micrographs, e.g. figs. 1 and 2 which were taken of specimens aged six minutes and 1000 hours respectively. The contrast in fig. 1 results from atomic displacements in the direction of the composition fluctuations. For interparticle spacings less than $150\text{\AA}$, sidebands or satellites are observed on both sides of the main diffraction spots along $\langle 100 \rangle$ directions (see fig. 3); and from the inverse relationship between the sideband spacing and the compositional periodicity in the crystal, the interparticle spacing could be determined directly from the diffraction pattern(1). The change in composition of the ferromagnetic Ni-Fe phase was followed by measuring the Curie temperature of the aged specimens.

A second part of this study was the correlation of microstructural changes during aging with the changes in yield stress and fracture mode of the aged specimens. The yield stress varied directly with the difference in composition of the two decomposing phases and was independent of the interparticle spacing. The fractured tensile specimens were examined directly in a scanning electron microscope; and because the specimens were conductive, no special preparation was necessary. The as quenched specimens and the specimens aged one minute fractured in a ductile manner characterized by dimples as shown in fig. 4. The spherical particles seen in the dimples initiated the dimple formation during the fracture process. For specimens aged six minutes and longer, the fracture mode was intercrystalline with some areas of ductile tearing as shown in fig. 5, which was taken of a specimen aged six minutes.

Spinodal decomposition is a clustering type reaction occurring in some fluid and solid systems where a homogeneous solution decomposes spontaneously into two phases upon cooling below the spinodal temperature. On a binary phase diagram the boundary of the spinodal region is defined by the locus $(d^2G/dc^2)_{T,P} = 0$ where G is the Gibbs free energy and c is composition. The spinodal is the limit of metastability for the homogeneous solution because inside the spinodal where $(d^2G/dc^2)_{T,P} < 0$ there is no activation barrier for the nucleation of the new phases which form initially as small composition fluctuations throughout the entire solution. In metallic crystals that are elastically anisotropic, the new phases form as periodically spaced platelets or rods that are preferentially aligned along the elastically soft directions of the crystal in order to minimize the elastic strain energy component of the total free energy. In face centered cubic Cu-Ni-Fe crystals, the cube directions are the elastically soft directions; and the alignment of precipitates along the $\{100\}$ planes is clearly seen in fig. 2.

1. E. P. Butler and G. Thomas, *Acta Met.* 18, p. 347, (1970).

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Fig. 1 Cu-Ni-Fe foil aged six minutes showing early stages of spinodal decomposition. Interparticle spacing is 75Å.

Fig. 2 Cu-Ni-Fe foil aged 1000 hours showing particle coarsening and alignment along {100} planes.

Fig. 3 Sidebands on 200 diffraction spot along [100] corresponding to microstructure shown in fig. 1.

Fig. 4 Scanning micrograph showing ductile fracture of Cu-Ni-Fe tensile specimen aged one minute.

Fig. 5 Scanning micrograph showing intercrystalline fracture of Cu-Ni-Fe tensile specimen aged six minutes.

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Fig. 1

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Fig. 2

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Fig. 3

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Fig. 4

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Fig. 5

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