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Electron Microscopy of Spinodal Structures at High Resolution

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I. INTRODUCTION

Alloys which have undergone spinodal decomposition from a super-saturated solid solution are characterized by a composition variation that is highly periodic. By suitable control of the aging sequence, the resulting modulated structure can be made to occur on an extremely fine scale throughout the entire specimen, thereby imparting a desirable homogeneity of properties. Even though the theoretical description of the spinodal reaction is highly developed¹, there are deficiencies in the experimental characterization of spinodal structures which arise from their sampling of rather large specimen volumes. In the present paper, a high resolution analytical method is described which is based upon direct imaging of the modulated lattice.

II. EXPERIMENTAL PROCEDURE

An alloy of Au-77 at .% Ni was chosen for study due to its large difference in component lattice parameters. Sheet specimens were homogenized at 900°C, quenched, and aged for twenty-one hours at 150°C prior to jet electropolishing (final thickness $\sim 100\text{-}300 \text{ \AA}$) for transmission electron microscopy foils. This aging scheme was utilized previously² to impart maximum decomposition kinetics. All foils were viewed in a [100] zone axis orientation for best visibility of the cube planes affected during decomposition. The microscope used for this study was a Siemens Elmiskop 102 equipped with a double tilt-lift goniometer stage and pointed cathode.

High resolution images were formed by the two-beam tilted-illumination method³ using the forward-scattered and 002 Bragg-scattered beams. Subsequent analysis of image detail was made using microdenistometer scans⁴ and optical diffraction⁵.

III. RESULTS AND DISCUSSION

A typical lattice image of the decomposed alloy is shown in Fig. 1. The fine scale fringes exhibit the spacing of the (002) planes (on average, 1.8 Å), while the large scale background intensity variation reflects the modulation wavelength. This spacing modulation was measured from microdensitometry of the photographic negatives, and a smoothed plot of peak-to-peak distances is shown in Fig. 2. The modulation wavelength determined in this way (29 ± 8 Å) agrees well with that indicated in the electron diffraction patterns (29 ± 3 Å) of the same specimen.

In analyzing electron diffraction patterns, it is usually the separation of satellite reflections bordering the Bragg beams which indicate the spinodal wavelength. Similarly, an objective assessment of the wavelength of the spacing modulation in a lattice fringe image can be made from the satellite spacings in optical diffraction patterns taken directly from the negative. During the present study this technique produced excellent quantitative agreement with the electron diffraction results, i.e., a wavelength of 29 ± 2 Å was obtained. There was also significant qualitative agreement between the observed relative intensities of the satellites in both cases; the high angle satellite was more intense due to the asymmetrical, Ni-rich composition of the alloy.

This analysis was also extended by incorporating optical microdiffraction⁵, which utilizes a small field limiting aperture in the path of the incident laser illumination. The aperture diameter was chosen to be smaller than the

spinodal wavelength, viz. $20 \text{ \AA}$ at the specimen plane, and by translation normal to the fringes in $20 \text{ \AA}$ steps, a sequence was obtained which showed a systematic variation in the spacing of the scattered beams. From this latter study, an objective verification of the results in Fig. 2 was obtained.

Throughout these analyses, the choice of optimum focus setting for imaging of the modulated lattice was reinforced by complementary optical diffractograms. In each case a choice was made from among an extended through-focus series of images taken from the same specimen area. Examination of the diffractograms gave an immediate impression of the effect of focusing on the image detail. The example shown in Fig. 3 shows that underfocusing the objective lens by $240 \text{ \AA}$ produces an image which exaggerates the information content of the high angle satellite (Fig. 3(b)), while the optimum imaging condition is shown in Fig. 3 (a)).

These results clearly indicate that the range of harmonics present in a modulated microstructure are directly revealed by lattice imaging. Additional information is also present on the amplitude of the individual composition peaks and their localized distribution with respect to other microstructural features. Such capabilities are essential to establish the atomistic processes involved in spinodal decomposition.

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REFERENCES

1. J. W. Cahn, Trans. Met. Soc. AIME 242, 166 (1968).
2. J. E. Woodilla and B. L. Averbach, Acta Met. 16, 225 (1968).
3. W. C. T. Dowell, Optik 20, 535 (1963).
4. R. Gronsky, M. Okada, R. Sinclair and G. Thomas, 33rd. Annual Proc. Elec. Mic. Soc. Amer., (G. W. Bailey, ed.) Claitor's, New York, 22, (1975).
5. R. Gronsky, R. Sinclair and G. Thomas, 34th. Ann. Proc. Elec. Mic. Soc. Amer., (G. W. Bailey, ed.), Claitor's, New York, 494, (1976).

FIGURES

- Fig. 1. Lattice image showing (002) planes in Au-77 at .% Ni modulated in spacing at a wavelength of approximately 30 angstroms.
- Fig. 2. Plot of d-spacing vs. distance obtained from microdensitometer scan of negative used in Figure 1. Each point represents an average of five measurements, with corresponding error indicated.
- Fig. 3. Optical diffractograms from Au-Ni lattice images taken at:
(a) optimum focus, Fig. 1; and (b) -240 angstroms underfocus, which enhances the intensity of the high-angle satellite reflections.

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Fig. 1

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Fig. 2

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Fig. 3

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