The Effect of Diffuse Scattering on the Dark-Field Contrast from Precipitates

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Precipitate contrast due to Bragg interactions in the diffuse scattering has been studied. Both ordinary dark-field contrast and moiré contrast were found to be preserved at quite large distances from the Bragg spots, especially along the direction normal to the reciprocal lattice vector. The effects, which are similar to those seen in thickness fringe contrast from perfect crystals, may be disturbing in the search for faint precipitate reflections with the dark-field technique, especially in thicker parts of the crystal.

1. Introduction

As was first shown by Kamiya & Uyeda (1961), the diffuse background in electron diffraction may contribute to the diffraction contrast in electron micrographs. The consequences of this effect in contrast experiments vary, however. When the objective aperture is placed around a strong spot, the contribution from diffuse scattering may be quite similar to that from the Bragg reflection, and hence does not call for any special consideration. The situation becomes different when dark-field imaging is used in the search for very faint precipitate spots and the crystals from which they originate.

We encountered this problem in a study of small copper precipitates in silicon (Solberg, 1978). It was found that considerable care had to be taken in order to ensure that the dark-field contrast really did originate from precipitate spots within the aperture and not from Bragg interactions in the diffuse scattering. It is felt to be of some interest to present and discuss some of the contrast effects with reference to studies of weak reflections from small particles.

2. Experimental results

The specimens were made from silicon, containing 2–3 p.p.m. of copper, which through a suitable heat treatment precipitates as small particles of the phase $\eta''$ (Solberg, 1978); see Solberg & Nes (1978) for details about the preparation. The micrographs were taken in a Philips EM 300 operating at 100 kV. Since the $\eta''$ particles constitute a very small fraction of the material, considerable areas had to be searched for precipitates, and also relatively thick specimens had to be used.

Fig. 1. Precipitate contrast due to diffuse scattering from the beam 28.0, 7, . The position of the objective aperture during the exposure is shown in (b).
Fig. 1 shows considerable precipitate contrast in dark-field taken from an aperture position where no precipitate reflection was apparent. The $\eta''$ structure may include a split spot at this position, but calculations indicate this to be extremely weak, and the contrast seemed much too strong to originate from such an invisible reflection. It was also found that the maximum contrast appeared when the nearby $28,0,7_{\eta''}$ reflection satisfies the Bragg condition, and that the contrast increases when the aperture is moved towards this, quite strong, precipitate spot. A further study of background contributions to dark-field contrast was

![Figure 2: Moiré contrast due to diffuse scattering from the beams $\bar{0}22_{\eta''}$ and $19,1,9_{\eta''}$. The positions of the objective aperture during exposures (a)-(f) are shown in (g).](image)

carried out by exploiting the moiré patterns which arise through interference between the 19,1,9,, spot and 022,, see Fig. 2(g). The moiré contrast was found to be preserved at quite a large distance away from the two spots (Fig. 2d) in a direction normal to the reciprocal lattice vector g. The contrast fades quite quickly, however, when the aperture is moved along the direction of g.

3. Discussion

The diffraction contrast described above stems from Bragg interactions in the diffuse scattering. Theoretical treatment of such interactions followed the original observations by Kamiya & Uyeda (1961), see, for example, Fujimoto & Kainuma (1963), Fukuhara (1963), Howie (1963) and Gjønnes (1966), and led to the generally accepted conclusion that diffraction contrast is preserved by small-angle diffuse scattering, especially along a line through the Bragg spot and normal to the reciprocal lattice vector of the spot. Detailed calculations of thickness fringes pertaining to this effect were recently presented by Rez, Humphreys & Whelan (1977).

The present case may appear slightly more involved, since Bragg scattering in both precipitate and matrix should ideally be included, leading to more terms than in the usual three-stage scattering expression relating to a perfect crystal. Since the effects are indeed the same as found in previously studied cases and the interest in the cases such as the present one is expected to be limited to recognizing the effects and taking steps to eliminate them, such a detailed treatment is not given here.

The contrast effects found in the present study will be most pronounced when the crystal is quite thick, and are therefore most likely to be encountered when there are few precipitates, so that a large volume of crystal has to be investigated. In such cases the present type of contribution to diffraction contrast should be considered.

References