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Fresnel effects at grain-boundary dislocations

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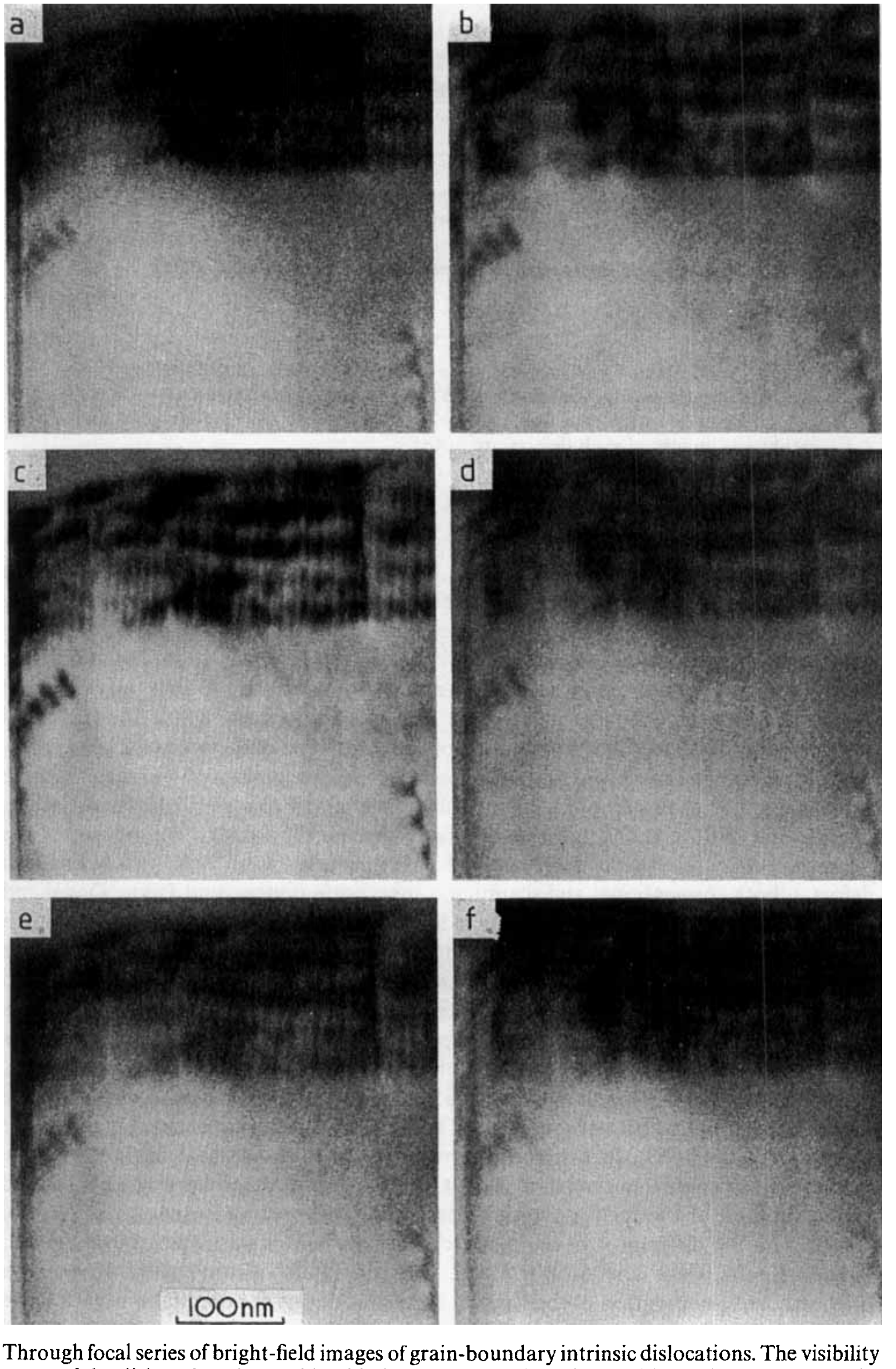
ABSTRACT

It is demonstrated that intrinsic grain-boundary dislocations of spacing 12 nm exert sufficiently strong Fresnel effects to show periodic changes in contrast over a limited defocus range. The implications of this for the interpretation of high-resolution images of such defects are noted.

The way fringes are formed at discontinuities, such as the edge of a specimen, in out-of-focus electron microscope images has attracted attention since the work of von Borries and Ruska (1939) and von Ardenne (1940). This has been partially because early calculations (for example, Hillier and Ramberg 1947) failed to explain the strong contrast of these fringes at low defoci, despite taking into account the amplitude change and phase shift produced by a thin specimen in modifying Born's wave optical expressions (Born 1933) for an opaque half-plane specimen. Once the effects of refraction were included, by taking into account the shape of the specimen edge cross-section, good agreement was obtained between experimental and theoretical fringe profiles (see, for example, Fukushima, Kawakatsu and Fukami 1974). Thus Colliex, Craven and Wilson (1977) have been able to discuss the relative importance of the 'refraction effect', classically described by Joy, Maher and Cullis (1976), at low and high defoci in both conventional and scanning transmission images, and Peyre, Davai and Henry (1980) have predicted the effects of inelastic scattering on the fringes. Also, with this improved understanding of the contrast phenomena, it has been used in the characterization of defects such as voids (see, for example Stobbs 1979, Foreman, von Harrach and Saldin 1982) and in the determination of the size of very small etch pits (Iijima 1977). The inherent problems in the unique determination of larger step heights by the method have, however, been demonstrated by the work of Boulesteix, Colliex, Mory, Renard and Yangui (1978). The technique has also been applied to the assessment of the widths of amorphous zones between grains in ceramics (see, for example, Clarke 1979). In this connection it has been shown that, while the fringe positions in an image at a given underfocus for such a region are relatively insensitive to the magnitude of the local potential discontinuity, the fringe contrast can depend strongly on the differences in the phase changes for waves passing through the two regions (Jepps, Page and Stobbs 1982). It is this which prompted us to make a qualitative re-examination of the Fresnel effects produced at potential discontinuities such as dislocations. Ferreira-Lima, Howie and Linington (1972) first demonstrated that an out-of-focus matrix dislocation exhibits Fresnel fringes and here we have examined whether or not intrinsic and extrinsic grain-boundary dislocations can be shown to exhibit similar effects.



Through focal series of bright-field images of grain-boundary intrinsic dislocations. The visibility of the dislocations is considerably lower in (*b*) and (*d*) than in (*a*) and (*e*). This cannot be attributed to specimen drift as in all cases the grain boundary on the left is sharp. Approximate values of Δf: (*a*) $-17\,\mu$m; (*b*) $-5\,\mu$m; (*c*) $0{\cdot}6\,\mu$m; (*d*) $7\,\mu$m; (*e*) $13\,\mu$m; and (*f*) $19\,\mu$m.

A through focal series of bright-field images of grain-boundary dislocations in an austenitic steel is shown in the figure. The images were taken with one grain (~ 130 nm in thickness) in a strongly diffracting condition, as can be seen from the matrix dislocation contrast. A Phillips EM300 electron microscope was used, operated at an accelerating voltage of 100 kV and fitted with a standard hair-pin filament. The low convergence required, $\lesssim 2 \times 10^{-4}$ rad, necessitated fairly long exposure times of between 30 s and 1 min despite the images being obtained at the low direct magnification of 21 000. The magnitude of the focal steps between the images was $\sim 6\ \mu$m and the examination of the 'in focus' Fresnel fringe profile at the specimen edge showed it to be approximately 0·6 μm over-focus. It can be seen that the images obtained at $\Delta f \simeq 7$ and $-5\ \mu$m show the intrinsic dislocation array of spacing ~ 12 nm at considerably lower visibility than do the images at $\Delta f \simeq 13\ \mu$m and $-17\ \mu$m. Similar effects were obtained in a through-focal series of dark-field images. In both types of image there was some indication that isolated boundary dislocations with very weak contrast when in focus, have enhanced contrast for defoci in the 5 to 10 μm range. That specimen drift was not the cause of the changes in dislocation visibility observed was checked by examination of the Fresnel fringes at a variably oriented specimen edge.

It is clear that the cyclic behaviour of the contrast between defoci of $+18$ and $-18\ \mu$m is not directly caused by the intrinsic dislocation array forming a fairly regular grating. At a spacing, d (here ~ 12 nm), enhanced contrast from this effect might first be expected at a defocus of $\sim 37\ \mu$m (d^2/λ where the wavelength $\lambda = 0{\cdot}0037$ nm). That the defoci at which reinforcement occurs ($\sim 13\ \mu$m and $\sim 17\ \mu$m) are so much smaller than would be expected on the above argument suggests that the Fresnel contrast at the individual dislocations might be responsible. There is some uncertainty as to whether the fringe displacements to be expected are proportional to Δf or $\Delta f^{1/2}$. However, in this case Fresnel diffraction is likely to be more important than refraction and large defoci are employed, consequently a dependence of the displacement on $\Delta f^{1/2}$ has been assumed (see, for example, Colliex *et al.* 1977 and Jepps *et al.* 1982). The positions of the Fresnel fringes as a function of defocus at the specimen edge, where refraction would be expected to be more important than at a dislocation, both confirmed this assumption and allowed a check on the relative magnitudes of image defoci. As a first approximation we might thus expect the first fringe position for each defect to be at $(\Delta f\lambda/2)^{1/2}$ so that superposition of these fringes, at $d/2$, would be expected to give enhanced visibility for $\Delta f \simeq 19\ \mu$m. Bearing in mind the work of Fukushima *et al.* (1974) demonstrating the way the displacement of the first fringe at an edge alters when realistic assumptions are made about the local phase and amplitude variations, the above figure is in fair agreement with our observed contrast reinforcement at a defocus of between 12 and 18 μm. Another simple approach is to treat each dislocation image as a single 'slit' and to use a Cornu spiral (defined by the Fresnel integrals as a function of V) to see whether or not self-consistent values of V and ΔV can be found for bright-fringe superposition at d. Given that reinforcement occurs at $\Delta f \simeq 15\ \mu$m, consistent values require a 'slit width', defining ΔV, of, not unreasonably, about half the dislocation spacing. The cyclic behaviour with defocus of the visibility of the dislocation array in the figure can thus be associated qualitatively with the overlap of Fresnel-like contrast due to each defect.

It has been suggested (for example, Balluffi, Woolhouse and Komem 1972) that it is often difficult even to distinguish between the contrast of the moiré fringes found between grains and that of grain-boundary intrinsic dislocations. The observation of Fresnel effects, as described here, unequivocally differentiates the periodic image as being associated with defects. Also the nature of the displacement field of a grain or phase boundary defect is notoriously difficult to determine (see, for example, Donovan and Stobbs 1983) and the result described here suggests that it might be possible to gain some insight into the magnitude of the potential discontinuity at such a defect by comparison with that at a matrix dislocation by this technique (the intensities of the first Fresnel fringe being a function of the relative phase differences from column to column). At the same time work on the Fresnel-like effects at perfect twin boundaries (D. J. Smith, W. M. Stobbs and G. W. Wood 1983, private communication) has demonstrated that only full atomistic contrast calculations would be likely to explain details of the contrast unequivocally. It is, of course, artificial to distinguish this type of contrast at a discontinuity from that generally associated with the variable local atomic positions and, in principle, accurately described by a full Bloch wave or multislice computation.

In conclusion it is interesting to note that an important application of high-resolution electron microscopy is the examination of grain-boundary defects, end-on, at such low spacings ($\lesssim 2{\cdot}0$ nm) that weak-beam techniques can provide no useful information (see, for example, Penisson, Gronsky and Brosse 1982). Typically if such images were to be observed at Scherzer defocus $(C_s\lambda)^{1/2}$, using either a typical 500 kV machine or a low-C_s 100 kV microscope, the analysed images would have defoci of between 50 and 100 nm and Fresnel-like effects, in particular at the dislocation cores, would then be important. The fringe displacement at Scherzer defocus is $\sim(C_s^{1/2}\lambda^{3/2}/2)^{1/2}$: equal to 0·3 nm for $C_s=0{\cdot}7$ mm, $\lambda=0{\cdot}0037$ nm and 0·2 nm for $C_s=3$ mm, $\lambda=0{\cdot}00142$ nm. That Fresnel effects as strong as we have observed do, in fact, occur confirms their importance in high-resolution defect image simulations.

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