APPARENT ANOMALOUS ABSORPTION AND IMAGE MATCHING

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Summary

Visual matching of experimental and theoretical electron micrographs can be used to determine the apparent anomalous absorption constant. The method has been applied to a copper–silicon (8 at.% Si) alloy by matching experimental and theoretical images of a Shockley partial dislocation and the main and subsidiary fringes of the associated stacking fault. The value of $0\cdot067$ which is obtained is considerably less than the value of $0\cdot1$ usually assumed for metals and alloys. In foils approximately $3\xi_{111}$ thick details of the image are very sensitive to the value of the anomalous absorption constant and the value of $0\cdot1$ is inadequate for image matching. However, in foils approximately $6\xi_{111}$ thick the general topological features of an image are little altered by varying the anomalous absorption constant over a wide range and the approximate value of $0\cdot1$ is adequate for image matching.

I. Introduction

In the formal two-beam dynamical theory of electron diffraction (Howie and Whelan 1961) two absorption parameters appear in the differential equations: ξ_g/ξ_0' the normal absorption parameter and ξ_g/ξ_g' the anomalous absorption parameter (where ξ_g is the two-beam extinction distance for the operative reflection $\boldsymbol{g}$, ξ_0' is the distance related to normal absorption, and ξ_g' is the distance related to anomalous absorption). In this paper $\mathscr{A}$ is used to denote the ratio ξ_g/ξ_g'. In any image computation technique which computes the intensity of the image of a defect relative to background intensity (e.g. Head 1967; Humble 1968*a*) the results are independent of normal absorption, but anomalous absorption is still a significant parameter.

Hashimoto, Howie, and Whelan (1962) found from comparisons of experimental micrographs and theoretical profiles of thickness and stacking fault fringes that $\mathscr{A}$ was in the range $0\cdot07$–$0\cdot10$ for several face-centred cubic metals and alloys. However, a value of $\mathscr{A}$ of $0\cdot1$ has usually been used when comparing theoretical profiles with experimental images (Humphreys and Hirsch 1968) and this value has also been used in comparisons of experimental images with images computed using the methods of Head (1967) and Humble (1968*a*) (e.g. France and Loretto 1968; Morton and Clarebrough 1969). Humphreys and Hirsch (1968) have shown theoretically that the assumption $\mathscr{A} = 0\cdot1$, irrespective of $\boldsymbol{g}$ and the material, is often a very poor approximation. However, they suggest that for normal values of $\boldsymbol{g}$ and for elements of medium atomic weight the assumption is valid as a first approximation.

For fringe contrast at stacking faults, it is well known that increasing anomalous absorption suppresses all subsidiary fringes and also the main fringes near the centre of the foil (Hashimoto, Howie, and Whelan 1960, 1962; Humphreys, Howie, and Booker 1967). For dislocations, the effect of increasing anomalous absorption is to

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reduce oscillating contrast effects near the centre of the foil (Howie and Whelan 1962), but little is known about the effect on other characteristics of the image. In this paper $\mathscr{A}$ for a 111 reflection in a copper–silicon (8 at.% Si) alloy is determined by image matching for a Shockley partial dislocation and its associated stacking fault in a thin foil at a small value of w ($= \xi_g S_g$, where S_g is the distance in reciprocal space of the operating reciprocal lattice point from the sphere of reflection), a case where subsidiary fringes are pronounced. For this case it is shown that $\mathscr{A} = 0\cdot1$ is a poor approximation. However, in thicker foils and at larger values of w, $\mathscr{A} = 0\cdot1$ is a reasonable approximation.

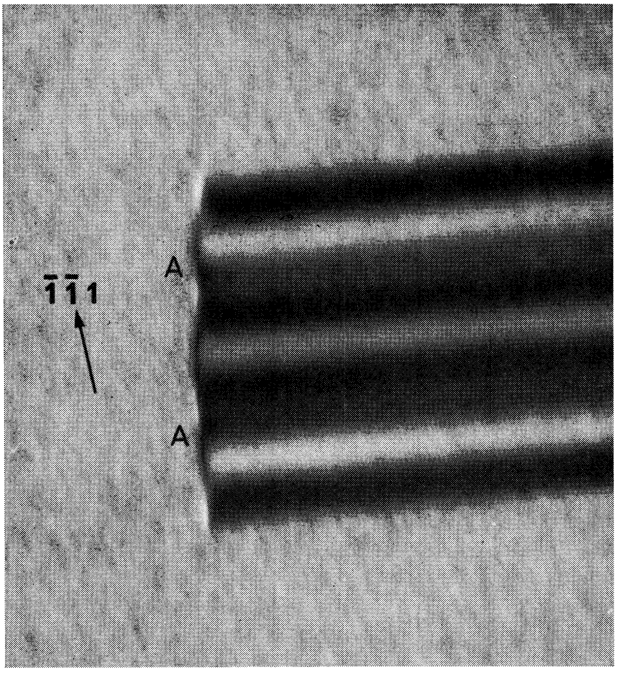

Fig. 1(a)

Fig. 1.—Images of a Shockley partial dislocation and the associated stacking fault in a copper–silicon (8 at.% Si) alloy. The beam direction is [347], the foil normal is [9 9 14], and $w = 0\cdot15$. In the experimental image (a) above ($\times$ 125 000) the operative reflection is indicated, while the computed images (b) opposite are for the indicated values of t and $\mathscr{A}$.

II. Observations and Image Matching

Figure 1(a) shows a region of fault in a lightly deformed copper–silicon (8 at.% Si) alloy. The sense of the reflecting vector was determined from the known sense of rotation from the [011] to the [123] beam direction and this rotation also fixed the plane of the fault as (111). The outermost fringe in Figure 1(a) is black, so that $\boldsymbol{g}.\boldsymbol{R} = -\frac{1}{3}$ ($\boldsymbol{R}$ is the displacement vector of the fault) (Hashimoto, Howie, and Whelan 1962).* From the known sense of $\boldsymbol{g}$, $\boldsymbol{R}$ is $\frac{1}{3}$[111] and the fault is intrinsic.

* For the range of validity of this rule, see the following paper by A. K. Head (present issue, pp. 569–71).

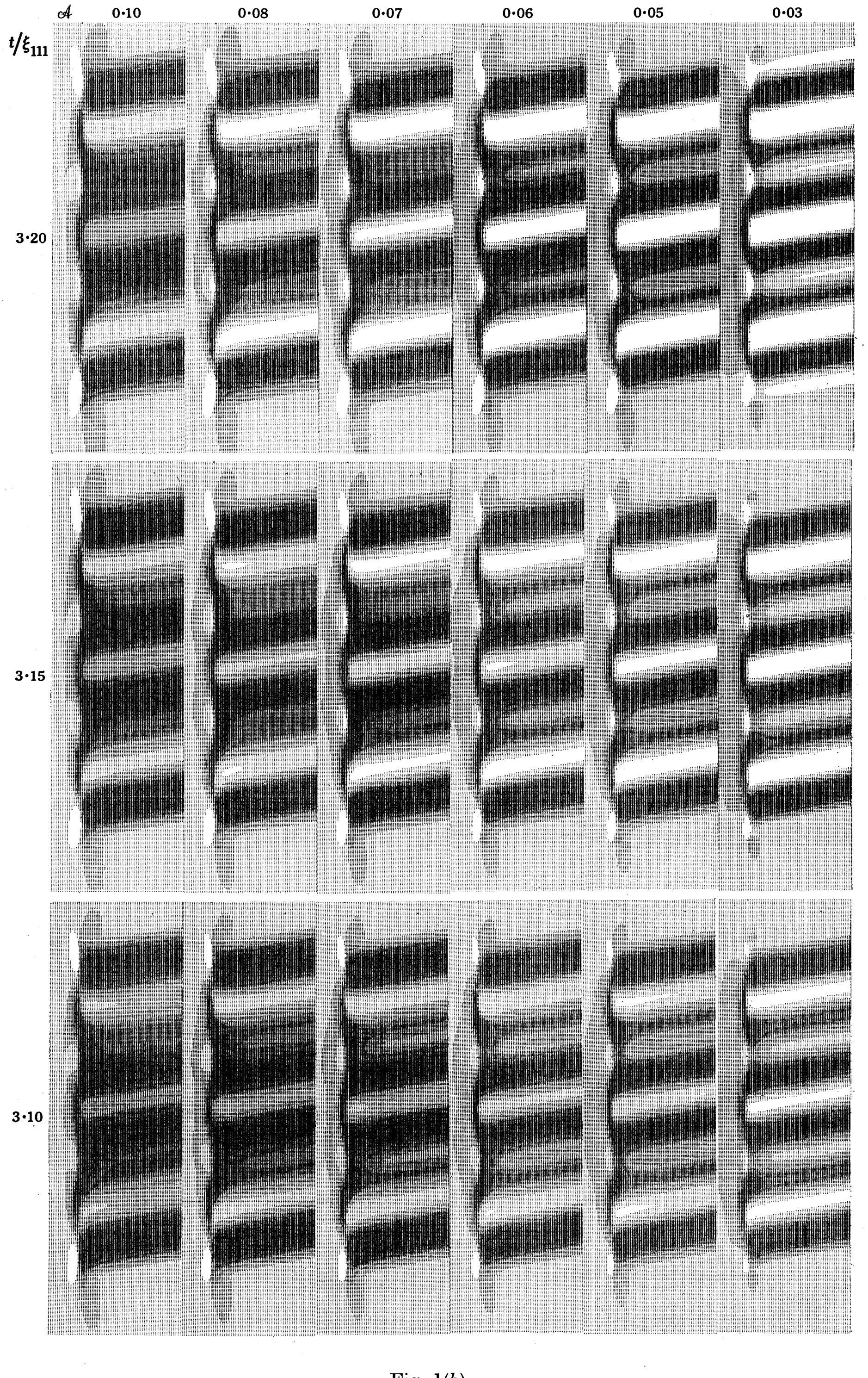

Fig. 1(*b*)

The beam direction in Figure 1(a) is [347] and the fault is bounded by a Shockley partial dislocation with $\boldsymbol{u}$ (a vector along the dislocation line) close to $[\bar{2}\bar{1}3]$. The dislocation is in contrast in Figure 1(a) so that $\boldsymbol{g}.\boldsymbol{b} = \pm\frac{2}{3}$ (where $\boldsymbol{b}$ is the Burgers vector) and, for the above $\boldsymbol{u}$ direction, $\boldsymbol{b} = \frac{1}{6}[11\bar{2}]$. The foil normal determined from three slip traces was [9 9 14]. The foil thickness was estimated from the projected widths of the (111) and $(\bar{1}11)$ planes and from the projected length of the intersection of $(1\bar{1}1)$ and $(\bar{1}11)$ using the relation

$$t = (l\cos\alpha)/\sin\beta\,,$$

where t is the foil thickness, l is the projected length of a known direction $[uvw]$, α is the angle between $[uvw]$ and the foil normal, and β is the angle between $[uvw]$ and the beam direction. The three values obtained for foil thickness were 884, 901, and 902 Å respectively. The extinction distance for the 111 reflection in the alloy was calculated from the atomic scattering amplitudes for electrons for copper and silicon and a value of 250 Å was obtained after applying a relativistic correction for 100 keV electrons (Hirsch *et al.* 1965). With this value of ξ_{111} the mean foil thickness corresponding to the above values is $3\cdot5\xi_{111}$. However, owing to the error in magnification when using a specimen holder with large tilt angles, this value of t could be in error by $\pm10\%$. The value of w determined from the diffraction pattern corresponding to Figure 1(a) was $0\cdot15$.

In the image in Figure 1(a) the dark fringe on the bottom of the image corresponds to the intersection of the faulted plane with the bottom of the foil. Subsidiary fringes are clear at A and it can be seen that the image of the fault is asymmetric about mid foil, the first light fringe near the bottom of the foil being above background intensity and the corresponding fringe near the top of the foil being close to background intensity. This asymmetry was confirmed by microdensitometer traces. The contrast at the dislocation shows strong oscillations with light regions near the top and bottom of the foil and at depths where the light subsidiary fringes appear. The contrast at the dislocation is dark where it crosses the main light fringes, but is lighter where it crosses the subsidiary light fringes. The image of the dislocation does not show a marked asymmetry about mid foil.

In matching experimental images the technique of Head (1967) and Humble (1968a) based on the Howie–Whelan two-beam column approximation (Howie and Whelan 1961) was used. With this technique a scale of grey is used in which the various intensities in the image are represented by a combination of single and overprinted characters on the computer line printer output. For image matching in the cases discussed here 7% below and 15% above background intensity have been taken as the visibility limits. The atomic displacements are calculated using anisotropic elasticity (Head 1967) and for image computation the elastic stiffness constants for a copper–silicon (7·69 at.% Si) alloy, namely $c_{11} = 16\cdot58$, $c_{12} = 12\cdot64$, $c_{44} = 7\cdot41 \times 10^{11}$ dyn cm^{-2} (Neighbours and Smith 1954), were used.

In matching the image of Figure 1(a), theoretical images were computed for values of t/ξ_g in the range $3\cdot0$–$3\cdot8$, $\mathscr{A}$ in the range $0\cdot03$–$0\cdot1$, and w in the range $0\cdot1$–$0\cdot2$. It was clear from initial computations that within these ranges (which include the experimentally determined values of t and w) there was only a small region of the combination of the three parameters in which a match to the experimental image could be obtained. This region is represented by the computed images in Figure 1(*b*) for which t/ξ_g varies between $3\cdot1$ and $3\cdot2$ and $\mathscr{A}$ between $0\cdot03$ and $0\cdot1$ with $w = 0\cdot15$. The influence of varying w in the range $0\cdot1$–$0\cdot2$ was small, so that the experimental value of $w = 0\cdot15$ is used for the theoretical images in Figure 1(*b*). The results in Figure 1(*b*) show that the topology of the image is very sensitive to variations in t and in $\mathscr{A}$. The subsidiary fringes develop at larger values of $\mathscr{A}$ as t decreases and at a given t the oscillatory nature of the contrast at the dislocation increases with decreasing $\mathscr{A}$. It is clear that $\mathscr{A} = 0\cdot10$ does not give a match to the experimental image in Figure 1(*a*) at any of the values of t.

Consider now the computed images in Figure 1(*b*) in more detail. The experimental image shows an asymmetry in fringe intensity whilst the computed images are symmetrical about mid foil (apart from small effects, see Section III). As discussed below, the asymmetry in the experimental image is most likely due to a change in foil thickness across the fault so that it is appropriate to match a particular portion of the experimental image with the computed images which are for constant foil thickness. Therefore, in the more detailed comparison we will consider only the portion of the experimental image from and including the central light fringe to the bottom of the foil. For $t/\xi_{111} = 3\cdot2$ subsidiary fringes do not appear until $\mathscr{A} = 0\cdot06$ and for this value the main light fringes are above background intensity, contrary to the experimental result. Further, the subsidiary fringes appear stronger than in the experimental image. For $t/\xi_{111} = 3\cdot1$ subsidiary fringes appear at $\mathscr{A} = 0\cdot08$, but the main light fringes are at or below background intensity and remain so until $\mathscr{A} = 0\cdot05$, at which stage only a small portion of the light fringe near the bottom of the foil is above background intensity. Further, the change in intensity across the subsidiary fringe for $t/\xi_{111} = 3\cdot1$ and $\mathscr{A} = 0\cdot05$ appears too great. The best agreement with experiment for the images in Figure 1(*b*) is considered to be at $t/\xi_{111} = 3\cdot15$ and $\mathscr{A} = 0\cdot07$. For these values the light fringe at the bottom of the foil is above background intensity and the central light fringe is at background, with the change in intensity across the subsidiary fringe appearing very similar to that in the experimental image.

At $t/\xi_{111} = 3\cdot15$ and $\mathscr{A} = 0\cdot07$, however, the computed image is too dark at the position where the dislocation crosses the subsidiary light fringe and by reducing $\mathscr{A}$ to $0\cdot067$ this contrast becomes lighter without any observable change in intensity of the main or subsidiary fringes. From all the computed images it is concluded that a value of $\mathscr{A} = 0\cdot067$ gives the best match to the contrast of the dislocation and stacking fault. Computations on the influence of w indicate that this value of $\mathscr{A}$ would also apply for $w = 0\cdot1$, but would have to be reduced to $0\cdot065$ for $w = 0\cdot2$.

The marked change in the topology of the images in Figure 1(*b*) with very small change in thickness suggests the reason for the asymmetry in the image in Figure 1(*a*). The taper on the foil is such that the thickness is increasing most rapidly in a direction approximately at right angles to the direction of the fault fringes so that an increase in thickness is to be expected on going from top to bottom across the fault. From the image of Figure 1(*a*) the light fringe near the bottom of the foil is above background intensity, the central light fringe is below background, and the light fringe near the top of the foil is close to background. Comparison of the experimental image with the computed images in Figure 1(*b*) suggests that these details could be matched by

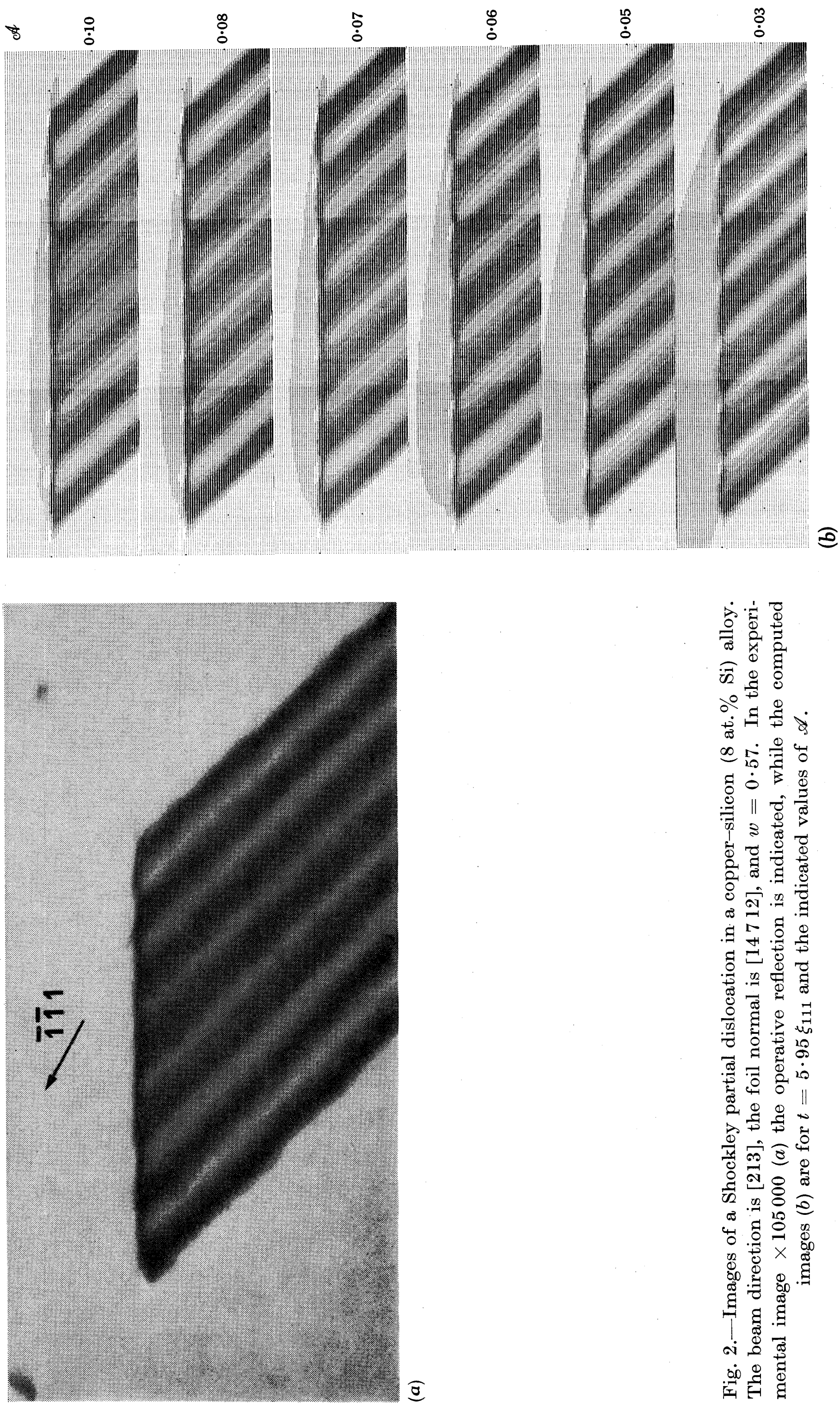

Fig. 2.—Images of a Shockley partial dislocation in a copper–silicon (8 at.% Si) alloy. The beam direction is [213], the foil normal is [14 7 12], and $w = 0{\cdot}57$. In the experimental image $\times 105\,000$ (*a*) the operative reflection is indicated, while the computed images (*b*) are for $t = 5{\cdot}95\,\xi_{111}$ and the indicated values of $\mathscr{A}$.

a change in thickness across the fault as small as $0 \cdot 05\,\xi_{111}$ with the thickness at the intersection of the fault plane with the top of the foil being $3 \cdot 10\,\xi_{111}$ and at the intersection with the bottom of the foil being $3 \cdot 15\,\xi_{111}$, that is, a change in foil thickness of approximately 12 Å.

Figure 2(*a*) shows a region of intrinsic fault in a thicker foil of the same alloy and in this case the fault is bounded by a Shockley partial dislocation for which $\boldsymbol{u}$ is $[0\bar{1}1]$ and the Burgers vector is the same as that for the dislocation in Figure 1(*a*), namely $\boldsymbol{b} = \frac{1}{6}[11\bar{2}]$. Measurements give a foil thickness of approximately $6\,\xi_{111}$ and $w = 0 \cdot 57$. Taking $\mathscr{A} = 0 \cdot 07$ from the previous result, a good match is obtained to the image in Figure 2(*a*) at $t/\xi_{111} = 5 \cdot 95$. The computed images in Figure 2(*b*) show the effect of varying $\mathscr{A}$ in the range $0 \cdot 03$–$0 \cdot 10$ for these larger values of t and w. Although changes in intensity can be detected in the computed images for a decrease in $\mathscr{A}$ from $0 \cdot 1$ to $0 \cdot 08$, the general topology of the image is not affected until $\mathscr{A}$ is reduced to $0 \cdot 03$. At this small value of $\mathscr{A}$ the contrast at the dislocation where it crosses the light fringes is very weak and subsidiary fringes are starting to develop. In contrast to the case of the thin foil, the topology of the image in foils approximately $6\xi_{111}$ thick is not nearly so sensitive to changes in thickness. Apart from changes in intensity in the fault fringes, the general topology of the image is not changed on increasing t from $5 \cdot 4\xi_{111}$ to $5 \cdot 95\xi_{111}$.

III. Discussion

The results presented here illustrate the strength of the technique of visual matching of experimental and computed images. Even though the example in Figure 1 involved three variable parameters (t, w, and $\mathscr{A}$) the apparent anomalous absorption could be determined very precisely. The theoretical values of $\mathscr{A}$ at 300°K for copper and silicon taken from Figure 2(*a*) of Humphreys and Hirsch (1968) give a value of $\mathscr{A} = 0 \cdot 044$ for this alloy. The theoretical results of Humphreys and Hirsch apply to zero aperture and, as they point out, must be increased for comparison with experimental results at particular aperture sizes. In the present experiments, the angle of the objective aperture was $2\theta_B$ (where θ_B is the Bragg angle for 111 reflection). An extrapolation of the results of Metherell and Whelan (1967) on the dependence of $\mathscr{A}$ on the aperture angle for the 111 reflection in aluminium suggests that $\mathscr{A}$ should be increased by approximately 100% for an increase in aperture angle from 0 to $2\theta_B$. However, the present result suggests that the theoretical value should be increased by approximately 50%.

It is clear from the results in Figure 1 that in a thin foil at small values of w the approximation $\mathscr{A} = 0 \cdot 1$ is a bad one as the computed images for this value do not match the details of the contrast of the fault fringes or at the dislocation. However, for the larger values of thickness and w considered in Figure 2, the general character of the contrast at the dislocation and of the fault fringes is little influenced by decreasing $\mathscr{A}$ from $0 \cdot 1$ to $0 \cdot 05$ and in this case the approximation $\mathscr{A} = 0 \cdot 1$ gives a good match to the topology of the image. However, even in this case, the approximation would be inadequate if quantitative matching of fringe intensity were required.

Clarebrough and Morton (1969) and Morton and Clarebrough (1969) have used the approximation $\mathscr{A} = 0 \cdot 1$ in image matching to determine the Burgers vectors and separations of stair-rod dislocations in complex Frank dislocation loops and the extent of dissociation of Frank dislocations in copper–aluminium alloys and in silver. In their experiments no account was taken of changes in absolute intensity in the image, but rather the required information was determined by matching the topological character of experimental images with computed images, and in many cases this matching involved very fine detail. In their experiments also the foil thicknesses used were in general in the range 6–8ξ_{111} and the values of w in the range 0·3–0·7. At such values of t and w the present results suggest that the topology of images is relatively insensitive to small changes in $\mathscr{A}$. Several of the images of Morton and Clarebrough for the copper–aluminium (9·4 at.% Al) alloy taken with 020 reflections and the same aperture angle as used here have been recomputed at various values of $\mathscr{A}$. No change in the general character of the images occurs until $\mathscr{A}$ is reduced from 0·1 to 0·05. The theoretical values of $\mathscr{A}$ for copper and aluminium from Figure 2(a) of Humphreys and Hirsch (1968) give a value of $\mathscr{A}$ for the 020 reflection in the copper–aluminium alloy of 0·047. Assuming that this value must be increased by approximately 50% to take account of aperture angle, then the present computations indicate that for the cases considered by Morton and Clarebrough $\mathscr{A} = 0 \cdot 1$ is an adequate approximation.

The bright field image of a stacking fault should be symmetrical about mid foil. However, the strain field of a Shockley partial dislocation can influence the details of fringe contrast for a considerable distance from the dislocation (Humble 1968b) and may cause an asymmetry in the fault fringes even in foils of constant t. Small asymmetries of this type can be seen in the light fringes at the top and bottom of the foil in Figure 1(b) for $t/\xi_{111} = 3 \cdot 10$ and values of $\mathscr{A}$ of 0·1 and 0·05. The asymmetry of the fault fringes in the experimental image in Figure 1(a) is very pronounced and is most likely due to a change in foil thickness, as discussed in Section II, rather than to effects arising from the strain field of the dislocation.

The theoretical images presented here are based on the two-beam column approximation of Howie and Whelan (1961). In determining the value of $\mathscr{A}$, it is therefore assumed that there is no contribution to the experimental image from systematic or non-systematic reflections. A contribution from non-systematic reflections can cause marked changes in an image, so that a two-beam approximation is no longer adequate (Humphreys, Howie, and Booker 1967; Howie and Basinski 1968). However, the present images were taken in beam directions which minimize any contribution from non-systematic reflections (Howie and Basinski 1968). Some contribution from systematic reflections cannot be prevented, but the influence of such reflections for both dislocations (Howie and Basinski 1968) and stacking faults (Humphreys, Howie, and Booker 1967) appears to involve changes in intensity without any change in the general topology of the image on which the image matching technique is based.

IV. Acknowledgments

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