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APPLICATIONS OF MANY BEAM SYSTEMATIC DIFFRACTION CONTRAST
IN HIGH VOLTAGE TRANSMISSION ELECTRON MICROSCOPY

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ABSTRACT

The increase in number of reflections that occur as the voltage is increased above 100 kV to 650 kV leads to several new applications of contrast analysis and improved resolution. In systematic orientations two practical examples of contrast analysis are described. In the first, it is shown how the nature of stacking faults (intrinsic or extrinsic) can be found from the contrast of dark field images formed in $1g$ when the $2g$ systematic condition is satisfied. The contrast is high if the phase factor $\alpha = -\frac{2\pi}{3}$ and considerably lower when $\alpha = +\frac{2\pi}{3}$. This is a general method and does not have the limitations of current methods of analysis, unless defects are very small ($\ll$ the extinction distance).

In the second method further exploration is made of the improved resolution that can be obtained for diffraction contrast at defects such as dislocations by lowering the effective extinction distance, e.g. by dark field weak-beam, or bright field high order reflection, imaging. These methods have been applied to defects in boron implanted silicon in which for the first time, dissociated loops have been resolved. From the separation of the partials, the stacking fault energy is estimated to be $45 \pm 6 \text{ ergs/cm}^2$ (isotropic elasticity) or $51 \pm 7 \text{ ergs/cm}^2$ (anisotropic elasticity theory).

1. Introduction

The excitation of many-beam systematic reflections is particularly pronounced in high voltage electron microscopy (HVEM), since the radius of curvature of the Ewald sphere increases as the electron wavelength decreases at higher energies. Also the scattering factor increases so that dynamical effects become more pronounced ^(1,2) than at lower voltages.

For the study of defects in crystals, the problem of solving the many-beam dynamical equations ⁽²⁾ involving many variable parameters is essentially intractable by analytical techniques, and numerical computation using a digital computer is usually required. Furthermore, the solutions of many-beam dynamical equations usually involve various approximations such as the column approximation, displacements in an infinite isotropic medium, etc. While these approximations appear to be generally applicable, they have not been extensively tested, so that supporting experiments are necessary before generalizations can be made of the suitability of many-beam dynamical theory for the quantitative characterization of crystal lattice defects.

In the present paper, which is part of a continuing study of defect contrast under many beam systematic conditions as observed in the HVEM, a method is described to determine the nature of stacking faults in fcc and diamond cubic crystals. The applicability of the weak-beam method ⁽³⁾ at high voltage has also been tested experimentally, and compared to resolutions obtainable by the high order bright field imaging method. ⁽⁴⁾ The results of theory and experiment have been compared and it is shown how such systematic reflections can be used to resolve partial dislocations and to determine stacking fault energies and the nature of stacking faults.

2. Determination of the Nature of Stacking Faults in fcc and dc Crystals

From two beam dynamical theory, rules have been derived for the determination of the nature of stacking faults in fcc and dc crystals^(1,5) which rely on the observation of the outermost fringes when the stacking fault intersects the foil surface. Subsequent calculations using the systematic many-beam dynamical equations have shown that the same information can be obtained without as many restrictions. Bell⁽⁶⁾ suggested a many-beam method suitable in high voltage microscopy that would determine the nature of faulted loops without requiring faults to intersect the foil surface. The method depends upon the fact that different contrast is observed in bright field (B.F.) images if the phase angle change at the fault $\alpha = 2\pi \vec{g} \cdot \vec{R}$ is positive or negative, ($\vec{g}$ is the diffraction vector and $\vec{R}$ the fault displacement vector).

This observation was thought originally to be a critical voltage effect⁽⁷⁾ but was subsequently shown to be due to systematic many-beam interactions⁽⁸⁾ with the contrast difference at the critical voltage being particularly pronounced. However, Tan⁽⁹⁾ showed that the contrast in bright-field is thickness dependent, which obviously limits the usefulness of the method, since the thickness of the foil has to be known before the correct sign of $\alpha = 2\pi \vec{g} \cdot \vec{R}$ can be determined.

In an attempt to sort out the exact relationship between the contrast of the stacking fault and thickness of the foil, the present results have shown that whilst the relative contrast of B.F. fringes are always dependent on the thickness of the foil, if an image is formed in lg dark field (D.F.)

then the contrast is uniquely determined by $\alpha = 2\pi \vec{g} \cdot \vec{R}$ when the 2g Bragg condition is exactly satisfied (1g is the first order reflection of the systematic set). The D.F. 1g stacking fault image exhibits higher contrast when the phase change $\alpha = 2\pi \vec{g} \cdot \vec{R}$ at the fault is $-\frac{2\pi}{3}$ and an observably lower contrast when $\alpha = +\frac{2\pi}{3}$ with modulus 2π , independently of foil thickness. Similar contrast effects can be found when the symmetry diffraction condition is satisfied, but its application is less practical, as will be discussed in a later section.

2.1. Computer Calculations

Using the systematic 8-beam dynamical approximation,⁽²⁾ the B.F. and D.F. image profiles of stacking faults in silicon have been calculated for different diffraction conditions, accelerating voltages and thickness of the foil. The parameter $\xi_g |\xi_g|$ was taken to be 0.06 for 100 kV and corrected to 0.022 for 650 kV for all g corresponding approximately to that determined by Bell.⁽¹⁰⁾ The column approximation has been used.⁽²⁾ The results are shown in Figures 1 and 2.

Since the phase angle change α appears only in the argument of the trigonometric function in the dynamical equations, the solution is invariant under a change of phase angle by a multiple of 2π . All the phase angles referred below will be in the principal value region, i.e. $-\pi \leq \alpha \leq \pi$ with modulus 2π .

For the calculated image intensity profiles, the maximum intensity has been normalized to unity to show the details of the fringe contrast.

Since the relative intensity determines the contrast, the visibility of the fringes are not changed when the scale is varied.

Figures 1(a) shows $1g$ ($2g$, $s=0$) D.F. image profiles of a stacking fault. The foil thickness varies from 4 to 5 extinction distances (ξg) with increments of $1/4 \xi g$. It can be seen clearly that the visibility of the fringes $\left(\frac{I_{\max} - I_{\min}}{I_{\max} + I_{\min}} \right)$ for $\alpha = -\frac{2\pi}{3}$ is generally much higher than that for $\alpha = +\frac{2\pi}{3}$ at all thicknesses. For $\alpha = +\frac{2\pi}{3}$, at most for 1 or 2 fringes because of the extremely low intensity at minimum, the visibility has about the same value as that for $\alpha = -\frac{2\pi}{3}$, but this is unlikely to be observed in practice due to its low intensity.

Figure 1(b) shows the B.F. image profiles corresponding to those in Figure 1(a). The contrast difference of fringes for $\alpha = \pm \frac{2\pi}{3}$ are large at foil thicknesses (t) of $4.25\xi g$ and $4.75\xi g$. At all other thicknesses, the contrast of both cases is much lower.

We have also calculated the effects to be expected if $2g$ is not quite exactly satisfied, i.e. for $s > 0$ or < 0 . This was done at reflecting conditions of $1.95g$ and $2.05g$ at all the thicknesses stated above. Whilst in many instances the contrast difference for $\alpha = \pm \frac{2\pi}{3}$ cases is large, as at $s = 0$, the effect is thickness dependent for both B.F. and D.F. images. From a practical viewpoint, therefore, the foil must be oriented at exactly $s = 0$ for $2g$ if the value of α is to be found, which can be readily obtained if sharp kikuchi lines are present.

Calculations have also been carried out for symmetrical reflecting conditions. At the exact symmetry position, pronounced contrast differences can be found between D.F. images for the first order reflections on both sides of the transmitted beam, (i.e. for $\pm g$). But for slightly deviated positions from the exact symmetrical case, the contrast difference changes

drastically and no general rule can be used. Because it is difficult to judge whether the symmetry reflection condition is exactly satisfied, the method is considered to be unimportant experimentally. By comparison of image profiles of stacking faults at 100 kV and 650 kV, we have found that the contrast difference of images for $\alpha = \pm \frac{2\pi}{3}$ at 100 kV is much less pronounced than their counterpart at 650 kV, confirming Bell's conclusion that the contrast truly arises from many-beam dynamical interactions, the strengths of which increase with increasing voltage.

Figure 2 is a graphical representation simulating the contrast effects shown in Figure 1. These were obtained by the overprinting method of image processing.⁽¹¹⁾ Eight grey levels (which are linearly proportional to the intensity of the image) were used.

2.2. Experiments

The features of the D.F. and B.F. images predicted by theory and shown in Figures 1 and 2 have been verified experimentally. This new method has been checked using the conventional method of analysing fault contrast.^(1,5)

Stacking faults in $\langle 111 \rangle$ epitaxial silicon have been used for the investigation. Figure 3(a) shows a normal D.F. image of a stacking fault for $\vec{g} = [\bar{2}20]$. The fault is readily identified to be extrinsic since if $\vec{g}$ is placed at the center of the fault, it points toward the light fringe and the method of Amelinckx et al. applies.^(1,5) The top edge of the fault is determined to be at the light fringe edge in Figure 3(a). This can be found by several well-known methods.^(1,12) The fault plane is therefore (111) and the geometrical configuration is

shown in Figures 3(b) and 3(c). Since the fault is extrinsic, $\vec{R} = \frac{1}{3}[\bar{1}\bar{1}\bar{1}]$, $\alpha = 2\pi\vec{g}\cdot\vec{R} = 2\pi \times \frac{4}{3} = +\frac{2\pi}{3}$ (modulus 2π). From Figure 1, the lg D.F. stacking fault images with $2g$ satisfied should show observably lower contrast for $\alpha = +\frac{2\pi}{3}$, than for $\alpha = -\frac{2\pi}{3}$. Figure 3(d) ($\alpha_g = +\frac{2\pi}{3}$) and 3(e) ($\alpha_g = -\frac{2\pi}{3}$) clearly show that this is indeed the case. Figures 4(a) and 4(b) show lg, ($2g, s = 0$) D.F. images of a stacking fault for $\alpha = -\frac{2\pi}{3}$ and $+\frac{2\pi}{3}$ respectively when $2g$ is satisfied for a foil of varying thickness. The fringe contrast for $\alpha = -\frac{2\pi}{3}$ case is much stronger than that for $\alpha = +\frac{2\pi}{3}$ at all thicknesses. However, the corresponding B.F. images shown in Figures 4(c) and 4(d), clearly show the oscillations in intensity with changing foil thickness. Since the contrasty or non-contrasty nature of the dark field fault fringes is determined by the sign of α only, and is independent of the thickness, the method is particularly useful for analysing the nature of faulted loops which do not intersect the foil surfaces. Thus, the method can be useful for analysing small loops inside the foil provided the dark field conditions are satisfied.

3. Determination of Stacking Fault Energy

The weak beam technique has now become a well-established method for the determination of stacking fault energy through the resolution of partials of extended dislocations. (3, 13-17) So far the experimental investigations (3, 13-17) have primarily been done at 100 kV with a number of preliminary studies at high voltages (18-20). It is of interest to determine if the weak beam method is also useful for high resolution at high voltage, especially since at high voltage thicker specimens are normally used because they are more representative of bulk materials.

Theoretical calculations and experiments⁽¹⁸⁾ have indicated that relatively narrow dislocation images can be obtained at high voltages. Theoretical analyses by Cockayne et al.,⁽³⁾ Cockayne⁽²¹⁾ and Sandström^(22,23) showed that, if the deviation parameter $|sg| \geq 2 \times 10^{-2} \text{Å}^{-1}$ for the weak-beam is satisfied, the width of a dislocation image at 1 MeV should be as narrow as that at 100 kV. However, in practice, for low index reflections such as $\langle 111 \rangle$, $\langle 200 \rangle$, $\langle 220 \rangle$ etc., which are most often used for contrast studies, the values of n (n for $n\vec{g}$ reflecting condition satisfied) required to obtain a large enough $|sg|$ to satisfy the weak-beam condition at high voltage can be very large, e.g. $n = 44$ for the (111) reflection of Si (the approximate n value for typical elements are listed in Table I).

Thus at high voltages, weak-beam imaging leads to the following difficulties:

- 1) Very high order reflections are often difficult to obtain, even with the existence of clear Kikuchi patterns.
- 2) Non-systematic reflections, whose influence on the image contrast are not yet well-treated by theoretical analysis, are much more likely to be excited to an extent such that they cannot be ignored.
- 3) The image intensity for weak-beam work may well be too weak to be practical, even at high voltages, because of other limitations in resolution (mechanical and electrical stability, etc.).

In order to find out whether the weak-beam method is practically useful at high voltages, we have applied the method to investigate dislocation loops formed in ion-implanted silicon. The data is compared to those obtained using the high order bright field method.^(8, 18)

3.1 Experiments

Specimens of boron-implanted silicon were annealed at 700°C for 1/2 hour in vacuum and chemically thinned. The resulting thin foils were then examined in a Hitachi-650 HVEM at 650 kV. Specimens were tilted to near (101) orientation, and care was taken to ensure that the $\vec{g} = [11\bar{1}]$ systematic reflection conditions were maintained. Micrographs of high order bright field (from $+5\bar{g}$ to $-6\bar{g}$) and corresponding weak-beam images (in $\pm g$) were taken. Examples are shown in Figure 5. For loops lying on the (111) plane, with edges elongated along $\langle 112 \rangle$, the Burgers vectors were identified to be $\frac{a}{2}\langle 110 \rangle$, (Figure 5d). Examination of the series of micrographs showed that these loops are dissociated, although constricted at some places, and the partials of the edge parts of the dislocations are resolved when the 3g, 4g, 5g, conditions are satisfied for both B.F. and weak-beam images. The weak-beam micrograph obtained in $5g_s = 0$ is shown in Figure 5c. It can be seen that the loop A with $\vec{b} = 1/2[10\bar{1}]$ and loops B and C with $\vec{b} = 1/2[01\bar{1}]$ are dissociated. Loop D with $\vec{b} = 1/2[\bar{1}10]$ shows residual contrast, since $g \cdot b = 0$ in this case. The average separation of the non-constricted part of the extended dislocations was measured to be $75 \pm 10 \text{ \AA}$ from microdensitometer traces taking into account possible magnification errors from the electron microscopy.

The measured separation of partials is about the same as that obtained by Ray and Cockayne⁽¹³⁾ for single dislocations in pure silicon measured at 100 kV. The stacking fault energy can then be calculated from elasticity theory of dislocations. In the isotropic case⁽²⁴⁾ and anisotropic case,⁽²⁵⁾ the values are determined to be $45 \pm 6 \text{ erg/cm}^2$ and $51 \pm 7 \text{ erg/cm}^2$, respectively. These values are close to the theoretical value calculated by pseudopotential

method 55 erg/cm^2 (26) for pure silicon.

3.2 Computer Calculations

The contrast of dissociated dislocation has also been calculated using many-beam dynamical equation. The calculation has been performed for 650 kV conditions using a 13-beam approximation. The foil thickness was estimated to be $4300\text{\AA}$ corresponding to that for Figure 5. Contrast for various depth of the defect from the foil surface at different diffraction condition were calculated. Figure 6 shows the B.F. and weak beam D.F. image profiles of extended dislocations for the 5th order reflection satisfied, for a dislocation with Burgers vector $\frac{1}{2}[01\bar{1}]$ lying on the (111) plane. The separation of the two partials is $75\text{\AA}$. The general shape of the dissociated dislocation profiles match well with the microdensitometer traces of the micrographs and the corresponding separation of the partials is also close to what was measured in the weak beam case (Figure 5). Thus confidence can be placed on the usefulness on the 13 beam dynamical contrast calculations at high voltages.

4. Summary and Conclusions

4.1 Stacking Faults

A method to determine the nature of the stacking fault in HVEM has been presented. The method is developed from the results of experiments and computer calculations using many-beam dynamical theory. The experimental procedure can be summarized as follows:

- 1) Obtain one systematic set of reflection conditions, e.g. $\vec{ng} = \langle 111 \rangle$
- 2) Take lg D.F. micrographs when $\pm 2g$ condition is satisfied
- 3) Determine the slope of the fault plane by the usual method (the actual fault plane indices are not required).

For analysis, from (2) the sign of the phase angle change α at a fault can be determined by applying the rule that the fringe contrast is much stronger for $\alpha = -\frac{2\pi}{3}$ case than $\alpha = +\frac{2\pi}{3}$ case. Combined with the knowledge of the slope of the fault plane, the nature of the fault can be readily determined.

Since the rule of contrasty and non-contrasty is strictly thickness independent only when exact 2g conditions are satisfied, the presence of Kikuchi lines is most helpful. The method is useful for defects contained inside foils.

4.2 Resolution of Defects

Weak beam and high order bright field imaging methods have been applied to dislocation loops in Boron-implanted silicon. The loops have been resolved to be dissociated, and the stacking fault energy has been calculated to be $45 \pm 6 \text{ erg/cm}^2$ for the isotropic case and $51 \pm 7 \text{ erg/cm}^2$ for anisotropic case. The resolution of the projected separation of partials is about $60\text{\AA}$, when the third to the fifth order reflection conditions are satisfied for $\vec{g} = [111]$. This indicates that weak beam imaging is practical and advantageous in high voltage electron microscopy, as well as at 100 kV.

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Table I

Numbers of the Order of Reflection Satisfying the Criterion

$$|sg| = 2 \times 10^{-2} \text{\AA}^{-1} \text{ for Typical Elements}$$

Element	Reflection	1 MeV	650 keV
Si ($a_0 = 5.43\text{\AA}$)	111	44	32
	220	16	11
Al($a_0 = 4.04\text{\AA}$) Au($a_0 = 4.07\text{\AA}$) Ag($a_0 = 4.08\text{\AA}$)	111	24	17
	200	18	13
	220	7	6
Cu($a_0 = 3.61\text{\AA}$)	111	19	14
	200	14	10
	220	7	5

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FIGURE CAPTIONS

- Fig. 1. (a) Computed profiles of D.F. images for $\alpha = \pm \frac{2\pi}{3}$ when second order reflection conditions are satisfied. The thickness of the foil varies from 45g to 55g in $\frac{1}{4}$ 5g increments;
(b) B.F. image profiles corresponding to Fig. 1(a).
- Fig. 2. Pictures produced by imaging processing technique corresponding to the cases shown in Fig. 1.
- Fig. 3. (a) 1g D.F. micrograph for $g = [\bar{2}20]$; (b) and (c) Geometrical configuration; (d) 2g D.F. micrograph $\alpha_g = (+\frac{2\pi}{3})$;
(3) -2g D.F. micrograph $\alpha_g = -\frac{2\pi}{3}$.
- Fig. 4. Micrographs showing contrast differences for 2g D.F. and B.F. for $\alpha = \pm \frac{2\pi}{3}$ at various thickness.
- Fig. 5. Dissociated dislocation loops in boron-implanted silicon
(a) 5g W.B.; (b) 5g B.F. for $\vec{b} = \frac{1}{2}[10\bar{1}]$; (c) 5g W.B.;
(d) Geometrical configuration.
- Fig. 6. Computed dissociated dislocation image profiles (a) B.F.; (b) W.B.

XBL 737- 6568

Fig. 1

XBB 737-4385 B

Fig. 2

Fig. 3

XBB 743-1597

XBB 743-1598

Fig. 4

(a)

(b)

(c)

(d)

XBB 743-1596

Fig. 5

(a)

(b)

Fig. 6

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