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CONTRAST AND RESOLUTION OF SMALL DISLOCATION LOOPS
IN HIGH VOLTAGE ELECTRON MICROSCOPY

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ABSTRACT

A study has been made of contrast from small loops in ion implanted silicon using high voltage electron microscopy. Theoretical calculations have been carried out to determine the optimum imaging conditions using many beam dynamical theory for systematic orientations. It was found that the best imaging conditions are obtained when the crystal is set for excitation near the third order reflection ($ng = \langle 111 \rangle$) and second order reflection ($ng = \langle 220 \rangle$) conditions respectively. It is also shown that for small loops or dipoles, defect visibility in bright and dark field as well as in the weak-beam condition is very sensitive to small changes in the deviation parameter and effective foil thickness.

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1. INTRODUCTION

In our continuing program of study of ion-implanted silicon, it has been necessary to characterise the small loop ($60\text{\AA} - 500\text{\AA}$) and rod shaped defects that are produced following annealing treatments in the $600^\circ\text{C} - 800^\circ\text{C}$ range [1,2]. These defects occur in high densities ($\sim 10^{15}/\text{cm}^3$) and in conventional two beam images the contrast is complex thus making characterisation of these defects extremely difficult. Consequently a study has been made of the visibility and contrast of such defects using high voltage electron microscopy for the many beam systematic orientation case, since this is known to enhance resolution of strain contrast images in bright field [3,4], and also in weak-beam dark field images [5].

2. EXPERIMENTS

Images at exact ng Bragg conditions (ng from $-6g$ to $+5g$ for $g = [111]$ and $-4g$ to $+3g$ for $g = [220]$) i.e. systematic bright field and weak-beam images, at 650kV have been obtained using a Hitachi 650kV electron microscope. In this study all dark-field images were obtained using the $(-g, ng)$ weak-beam excepting in Figs. 6 and 7 where the $(+g, ng)$ dark-field image was used.* The foil was also tilted gradually from $lg s_g = 0$ to $4g s_g = 0$ and the same defects observed in bright and dark-field conditions in order to compare images at slightly

* In this paper we shall use the convention $(000, ng)$ to refer to bright field imaging conditions and (g, ng) for weak-beam conditions; for conventional images $n = 1$, i.e. first order reflections are strongly excited.

differing values of s_g , the deviation from the exact Bragg condition [6a]. Image widths were measured on electron microscope plates by densitometry across specific defects.

Defect Models Used for Calculations

For the purpose of the calculations the elongated rod-like defects were assumed to be closely spaced dislocation dipoles lying on {111} planes with Burgers vectors of the type $\frac{a}{2} \langle 110 \rangle$. Image profile calculations were performed for dipole separations of 60 to 100 Å. Except where visibility was calculated as a function of depth, the dipoles were assumed to be at one half the extinction distance ($1/2 \xi_g = 625 \text{ Å}$) below the top surface in a foil which is two extinction distances thick (2500 Å).

3. RESULTS

Fig. 1 [7] shows bright-field and weak-beam ($-g$) images of an area of elongated rods. Various reflection conditions from $1g$ excited were used. The maximum image separation i.e. defect resolution, in both bright field (B.F.) and weak-beam (W.B.) occurs at the $2g$ condition. This is shown in Fig. 2 where the separation of the two image peaks (as measured by the microdensitometer traces) of defect (a) in Fig. 1 are plotted. Defect (c) in the same figure, shows similar image contrast. This is in contradiction with an earlier speculation [8], that the image separation continues to enlarge for higher order reflection conditions. The fact that dipole image separation decreases beyond $2g$ in W.B. may be expected from the analysis of high voltage

systematic images [4]. The higher the order of reflection, the larger is the deviation parameter s_g , and the closer is the image to the core. When the outside contrast condition is satisfied [6b], this implies the image separation of two dislocations should decrease as s_g increases.

Calculated image profiles of dipole images ($60\text{\AA}$ and $100\text{\AA}$) for bright field ($0,ng$) and weak-beam conditions ($-g,ng$) are shown in Fig. 3a for $\vec{g} = [2\bar{2}0]$ and in Fig. 3b for ($0,ng + 0.05$) BF and ($-g,ng + 0.05$) WB, ($n = 1,4$). These graphs yield data on changes in the image as higher orders of reflection are excited. These correspond to the experiment shown in Fig. 1. The variance in image characteristics for small changes in diffracting conditions can also be studied from these graphs.

In accordance with experiment, the dipoles are not resolved for the $1g$ condition (Fig. 4a). The maximum separation of the dipole image, in agreement with experiment, occurs both in bright and dark field at the $2g$ exact Bragg condition. The effect of exciting higher orders is mainly to decrease the image width.

Computed image visibility and width at different reflecting conditions are shown in Figs. 4b,c. The Michelson criterion [9] has been used for visibility: $\text{Visibility} = \frac{I_{\text{max}} - I_{\text{min}}}{I_{\text{max}} + I_{\text{min}}}$. The graphs show that maximum visibility and slightly wider images are associated with the $2g$ Bragg condition. This provides the best imaging conditions for the small defects under investigation. The asymmetry of the image profile makes it difficult to calculate visibility for the bright field case.

Figure 5 shows the case for $\vec{g} = [111]$ with $n\vec{g}$ satisfied ($n = 2, 5$). For $n = 1$, there is very poor image contrast and is not shown. For $n = 2$, the contrast is also quite poor as shown in the figure. However, for $n = 3$, the contrast becomes distinctly better and the images of the line defects are quite narrow. In fact, the partials of the dissociated elongated loop which have the separation of about $75\text{\AA}$ are all resolved in these cases [10]. Since in practice, exact systematic reflections are much more difficult to obtain for higher order reflections than for lower, and the intensity of the weak beam for the very high order reflection is very low, the practical use of the method is limited. The lowest order of reflection which gives good contrast and resolution is considered to be the best imaging condition, such as the $3g$ reflection ($g = 111$) in the present case. It is interesting to note that these best imaging conditions are close to those of maximum transmission for perfect crystals at 1MeV [11], although the detailed nature of the correlation is not yet clear.

The experiments where the foil was tilted by small amounts showed that the visibility of small defects are markedly affected by the value of the deviation parameter s_g ; at 650kV both bright field and dark field images can show this strong visibility dependence on s_g . An example of this at the $3g$ $[111]$ condition is shown in Fig. 6; [12]. The circled loop is only seen at $3g$ ($s_g = 0$) in Figs. 6c,d. It disappears anomalously at the other conditions. Such a disappearance may also be seen in the dipole images of Fig. 1(8). In the $3g$ weak beam images (Fig. 1(8) near the white arrow) the image of one segment of the elongated loop is seen to disappear.

Computer calculations were carried out to find out how well the experiments correlate with the contrast theory and what other parameters may have a strong influence on this effect. Figure 7a shows a plot of visibility vs. diffracting conditions near $2g$ for a $60\text{\AA}$ wide dipole. It shows, with slight change in s_g , that the visibility of the W.B. image decreases sharply (from 0.96 to 0.46). Figure 7b shows the depth dependence of the visibility; when the depth of the dipole was changed from $0.5\xi_g$ to $1\xi_g$ the visibility fluctuated between 0.88 and 0.97. For a fixed depth ($d = 1/2\xi_g$), but differing thickness from $2\xi_g$ to $3.75\xi_g$, the visibility varies from 0.34 to 0.9, which is a much more pronounced effect (Fig. 7c). Further computations for thicker specimens, varying thickness ($t = 5-6\xi_g$, $d = 1\xi_g$) and depth ($t = 5\xi_g$, $d = 1-2\xi_g$), show similar image behavior. The dependence on accelerating voltages (300-650kV) and defect size (30-500 $\text{\AA}$) were found to be much less than those of thickness and depth.

6. SUMMARY AND CONCLUSION

The conclusion that can be drawn from this investigation is that the optimum imaging conditions for small defects in silicon is for systematic orientations with excitations near $2g_{220}$ and $3g_{111}$ at 650kV. These are also the conditions of maximum transmission found by Humphreys [11].

For imaging closely-spaced dipoles it was found that the maximum dipole-image separation occurs at $2g_{220}$ for both bright field as well as weak-beam images. Contrary to an earlier expectation, further

tilting to higher orders of reflection does not increase image separation [8]. This contradiction may be resolved by noting that in the systematic, high voltage, case the shift of the image from core position decreases, the higher the order of the reflection [4]. The calculations for the bright-field case show similar results (Figs. 3a,b). Consequently, at higher orders the image separation is more nearly equal to the core separation, i.e. the image separation decreases. Therefore, it is concluded that for high resolution imaging where images and cores most closely coincide (e.g. dissociated dislocations) high order systematic bright field and weak-beam images are beneficial.

The experiments on tilting the foils through small angles show that images of small defects are extremely sensitive to changes in s_g . At certain s_g values the images of some defects disappear anomalously ($\vec{g} \cdot \vec{b} \neq 0$). Computer calculations confirm this speculation.

Strong contrast differences in dark field by varying s_g at 100kV in silicon have been observed [13]. An anomalous disappearance of line defects in bright field in α -ion is also reported and ascribed to changes in s_g [14]. Our experiments show the anomalous disappearance effect occurs in both bright field and weak beam under systematic high voltage conditions. Theoretical calculations confirm the strong s_g dependence. Thickness and defect depth also have considerable influence on defect visibility. Therefore, caution should be taken in the determination of Burgers vector in high order bright field and weak beam work at high voltages.

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FIGURE CAPTIONS

Fig. 1. Shows several bright-field (BF) and weak-beam (WB) images in B^+ ion implanted silicon with $g = [2\bar{2}0]$ under the following conditions:

1. $(0,g)$ BF, 2. $(0g,g)$ WB 3. $(0,1.75g)$ BF
4. $(0g,1.75g)$ WB 5. $(0,2g)$ BF 6. $(0g,gs+)$ WB
7. $(0,3g)$ BF, 8. $(-g,3g)$ WB - A segment of loop (b) as shown by a white arrow, anomalously disappears. (Courtesy Claitors Publishers, Baton Rouge).

Fig. 2. Shows experimentally measured image separations for defect (a) in Fig. 1 vs. reflecting condition. The maximum image separation occurs at the $2g$ reflecting condition.

Fig. 3 (a,b) Shows calculated dislocation dipole profiles for $g = [\bar{2}20]$ in bright field (BF) and weak-beam $(-g,ng)$, 1-8 for a $60\text{\AA}$ wide dipole and 9-16 for a $100\text{\AA}$ dipole. In Fig. 3b the foil is tilted slightly away from the exact Bragg condition, as indicated in the captions. The foil thickness is $2500\text{\AA}$, the defect is at depth $d = 625\text{\AA}$ of Burgers vector $b = 1/2[101]$. These profiles show that bright field and weak-beam images are very sensitive to defect size (Compare Fig. 3a 1 and 9) and diffracting condition (Compare Fig. 3a, 13 and 3b 13).

Fig. 4 (a,b,c) Shows several parameters as calculated in Fig. 3a,b. In Fig. 4a image separation vs. the Bragg condition is shown. The maximum separation, in agreement with experimental result (Fig. 2), occurs at the $2g$ condition.

In Fig. 4b image visibility and in Fig. 4c image width is plotted vs. reflecting condition. Notice that the $2g$ condition combines maximum visibility and image width.

Fig. 5 Shows several bright-field and weak-beam images in B^+ ion-implanted silicon with $g = [111]$ under the following conditions:

1. $(-g, 5g)$ WB	2. $(0, 5g)$ BF
3. $(-g, 4g)$ WB	4. $(0, 4g)$ BF
5. $(-g, 3g)$ WB	6. $(0, 3g)$ BF
7. $(-g, 2g)$ WB	8. $(0, 2g)$ BF

The partials in the elongated loop (a) is resolved for the $(-g, ng)$, $n = 3$ condition as explained in the text.

Fig. 6 Shows several bright and dark-field images of P^+ ion-implanted silicon with $g = [111]$ under the following conditions:

a) BF $(0, 2g \ s_g +)$	b) DF $(g, 2g \ s_g +)$
c) BF $(0, 3g)$	d) DF $(g, 3g)$
e) BF $(0, 3g \ s_g +)$	f) DF $(g, 3g \ s_g +)$

The circled loop is seen only at the exact $3g$ Bragg condition (c and d) and disappears "anomalously" both in bright and dark field $s_g \neq 0$. (Courtesy Claitor's Publishing Division Baton Rouge).

Fig. 7 Shows calculated weak-beam visibility of a $60 \text{ \AA}$ dipole a) vs. reflecting condition at two different depths d_1 and d_2 for a foil thickness of $2500 \text{ \AA}$, b) vs. defect depth (625 – $1250 \text{ \AA}$) for the same reflecting condition and foil thickness as in (a)

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and c) vs. foil thickness with dipole fixed at a depth of $625 \text{ \AA}$.

These plots show that visibility varies drastically with reflecting condition and effective foil thickness and simulates the experimentally observed phenomenon in Fig. 6.

XBB 732-1225

Fig. 1

-13-

XBL742-5581

Fig. 2

Fig. 3a

XBL 737-1574

Fig. 3b

XBL 742-5583

Fig. 4a

Fig. 4b

Fig. 4c

XBB 736-3777

Fig. 5

XBB 7210-5175

Fig. 6

XBL 749-1686

Fig. 7

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