IMAGE CONTRAST OF DISLOCATION LOOPS IN BCC METALS*

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Abstract

Electron microscope image contrast for small dislocation loops in niobium has been calculated from the dynamical theory of electron diffraction using the displacement field corresponding to both prismatic and shear components of the Burgers vector. For dynamical diffraction conditions, in addition to the usual black-white contrast, some loops show a triple white-black-white or a black-white-black contrast depending on the depth and the orientation of the loop. The results indicate that care should be taken in determining the vacancy or interstitial nature of dislocation loops in irradiated BCC metals.

Introduction

Defect structures encountered in irradiated BCC metals are frequently in the form of small unresolved dislocation loops which are formed by the condensation of point defects of either vacancy or interstitial type. The presence of these dislocation loops is believed to be responsible for some of the property changes observed following irradiation. The identification as to the vacancy or interstitial nature of the loops is uncertain at present because the analyses employed are based on the contrast behavior expected of a pure edge prismatic loop. Whereas in FCC metals prismatic loops are frequently observed, in BCC metals some of the loops are expected to have their Burgers vectors inclined to the plane of the loop. A typical example is a loop lying on the (110) plane with an $\frac{a}{2}[111]$ Burgers vector.

Calculation of image contrast in the electron microscope requires a knowledge of the displacement field around the loop. In the past, the elasticity solutions for the displacement field have been available under the assumption of isotropic elasticity only for

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a pure edge prismatic dislocation loop (1). For a loop with its Burgers vector inclined to the plane of the loop, it is also necessary to have the displacement field arising from the shear component of the Burgers vector which lies in the plane of the loop. Recently, solutions have been obtained for the displacement field due to the shear component of a circular dislocation loop by applying the Green's function method (2). In the present paper, these solutions have been incorporated into the two-beam dynamical diffraction equations and the image contrast of dislocation loops in niobium has been calculated.

Dislocation Loops in BCC Metals

In BCC metals, the condensation of point defects is expected to occur on the most densely packed {110} planes resulting in a faulted loop of Burgers vector $\frac{a}{2}[110]$. The stacking fault in the loop is expected to have a high stacking fault energy. The energy of this configuration can be reduced by the nucleation and growth of a shear dislocation loop inside the loop eliminating the stacking fault. A shear along the [001] direction will result in a perfect loop with the Burgers vector $\frac{a}{2}[111]$, while a shear along the $[1\bar{1}0]$ direction will lead to an a[100] Burgers vector. These loops lie in the (110) plane with Burgers vectors inclined to the loop plane. It is possible for these loops to glide along their glide cylinders and rotate into pure edge orientations giving unfaulted prismatic loops on (111) and (100) planes. The possible dislocation loops that are expected in an irradiated BCC metal are:

Pure Edge Prismatic Loop

$\vec{b} = \frac{a}{2}[110]$ on (110)

$= \frac{a}{2}[111]$ on (111)

$= a[100]$ on (100)

Sheared Loop

$\vec{b} = \frac{a}{2}[111]$ on (110)

$= a[100]$ on (110).

The expressions for the displacement field around a circular dislocation loop of vacancy type with its shear component of the Burgers vector along the x axis (the x-y plane taken as the plane of the loop) are given by:

$$u_x = \frac{1}{4(1-\nu)} \{ \frac{b_p x}{R\,\rho} [(1-2\nu)\ I_1^o - |\zeta|\ I_1^1] - b_s \zeta [\frac{2(1-\nu)}{|\zeta|} I_o^o + \frac{(x^2-y^2)}{\rho^3 R^2} I_1^o - \frac{x^2}{\rho^2 R^2} I_o^1]\} \qquad (1)$$

$$u_y = \frac{y}{4(1-\nu)} \{\frac{b_p}{R\,\rho} [(1-2\nu)\ I_1^o - |\zeta|\ I_1^1] - \frac{b_s x\zeta}{\rho^3 R^2} (2\ I_1^o - \rho\ I_0^1)\}$$

$$u_z = -\frac{1}{4(1-\nu)} \{\frac{b_p|\zeta|}{\zeta} [2(1-\nu)\ I_o^o + |\zeta|\ I_o^1]$$

$$+ \frac{b_s x}{R\,\rho} [(1-2\nu)\ I_1^o + |\zeta|\ I_1^1]\} \quad ,$$

where b_p and b_s are the prismatic and shear components, respectively, of the Burgers vector, R is the loop radius, ν is Poisson's ratio (=1/3), $\rho = (x^2+y^2)^{1/2}/R$, $\zeta = z/R$, and I_m^n is the Lipschitz-Hankel integral defined as

$$I_m^n = \int_o^\infty t^n\ J_1(t)\ J_m(\rho t)\ \exp(-|\zeta|\ t)\ dt \qquad (2)$$

where J_m is the Bessel function of first kind and order m. The function I_m^n can be expressed in terms of elliptic integrals (3). Figures 1 and 2 show equi-displacement contour plots in the plane y = 0 of u_x and u_z in units of Burgers vector. The Burgers vector taken as $\frac{a}{2}[111]$ makes an angle of 35° 16' with the $[\bar{1}10]$ loop normal. Compared to similar plots for a pure edge prismatic loop, the contour lines are stretched out considerably along the direction parallel to the Burgers vector.

Image Contrast

The contrast profiles of dislocation loops of various Burgers vectors and orientation have been computed numerically by integrating the two-beam dynamical diffraction equations of Howie and Whelan (4) on the basis of the displacement field given in Eq. (1). The foil plane is taken as the (113) plane oriented for exact Bragg condition for the $(12\bar{1})$ reflection. The foil thickness is taken as 1000 Å and the loop radius 25 Å. The diffraction parameters employed are ξ_g = 460 Å, ξ_o' = 3000 Å, and ξ_g' = 4700 Å, where ξ_g is the extinction distance, and ξ_o' and ξ_g' are the absorption distances (5,6).

1. $\frac{a}{2}[110]$ on (110):

   The depth dependence of the image contrast under the dynamical diffraction condition of this prismatic loop agrees with the prediction of Rühle et al (7). For the vacancy type of loop, the black-white direction is along the projected direction of the Burgers vector for $\vec{g}\cdot\vec{b} > 0$ in bright field within $\xi_g/4$ of the top surface. The black-white direction reverses at $\xi_g/4$ and again at $3\xi_g/4$ as the depth of the loop is varied.

2. $\frac{a}{2}[111]$ on (110):

   The contrast behavior of this sheared loop as a function of the depth from the top surface is shown in Fig. 3. The loop shows the usual black-white contrast at a depth about one-half of the extinction distance. The loops located quite close to the foil surface as well as at about one extinction distance depth exhibit either white-black-white or black-white-black contrast. The dotted areas correspond to a weak white contrast while the cross-hatched areas represent a weak black contrast. The detailed appearance of this weak contrast will depend on which (110) type plane contains this loop, as well as the background intensity which varies with the foil thickness and the diffraction condition. Another new feature is the presence of white contrast at the depths of $\xi_g/4$ and $3\xi_g/4$. These calculations also indicate that the direction of the maximum black-white streaking may deviate as much as 15° from the projected direction of the Burgers vector depending on the orientation of the loop.

3. a[100] on (100) and (110):

   The image contrasts of these a[100] loops are nearly identical to those of the prismatic loop of $\frac{a}{2}$[110]/(110) type.

4. $\frac{a}{2}$[111] on (111):

   This loop formed as a consequence of rotation into the pure edge orientation retains the image contrast of the $\frac{a}{2}$[111] sheared loop described above.

Discussion

The present work has shown that the depth dependence of the image contrast of dislocation loops in BCC metals is expected to be more complicated than that encountered previously in FCC metals. The most striking feature predicted is the appearance of triple white-black-white and black-white-black contrasts. These triple contrasts are characteristics of the loops with $\frac{a}{2}$<111> Burgers vectors. Since one of the triple contrasts is weaker than the other two, these spots may simply appear as black-white spots in some cases. When this occurs, the depth dependence of the black-white contrast is similar to that predicted for a prismatic loop in FCC metals.

In view of these results, re-examination of some of the electron micrographs obtained in an earlier study of niobium irradiated to a dose of 10^{18} neutrons/cm^2 (E > 1 MeV) at room temperature (8) has been carried out to see if contrast predictions could be verified. A number of white-black-white and black-white-black contrast configurations were noted for diffraction conditions comparable to those employed in the present calculations. However, the white contrast described earlier was not observed. There was insufficient information (i.e., stereo depth measurements) to verify in detail the calculations or the vacancy-interstitial nature of the loops. The detailed verification of the calculated contrast and determination of the loop character will require careful experiments coupled with calculations in order to delineate the extent of agreement between theory and experiment.

The recent work of Wilkens et al (9,10) on the black-white contrast configurations is in general agreement with the present result in that the image contrast of a dislocation loop in BCC metals is more complicated than that in FCC metals. An exact comparison cannot be made at present because the loop configurations employed by them are not identical to that used in the present work. In their calculation, they used an asymptotic displacement field based on the infinitesimal loop approximation which is only valid in regions more than twice the loop radius away from the loop. This approximation is not expected to predict all details of the image contrast, but it should correctly predict the symmetry properties of the black-white contrast.

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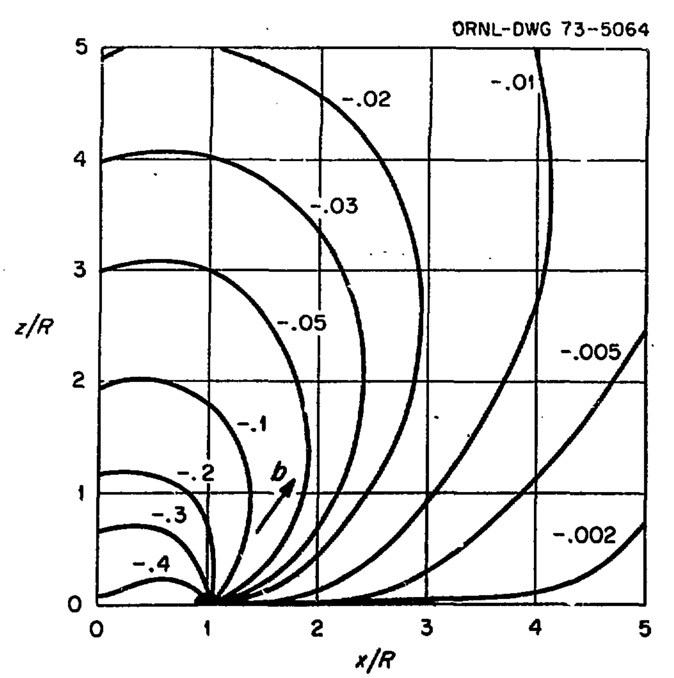

FIG. 1

Equi-displacement contour plot of u_x in units of b for a sheared dislocation loop in an isotropic medium.

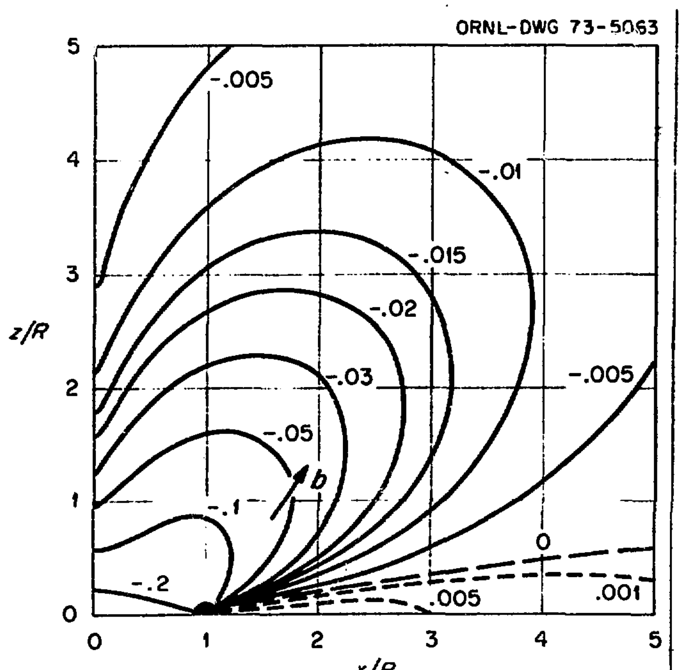

FIG. 2

Equi-displacement contour plot of u_z in units of b for a sheared dislocation loop in an isotropic medium.

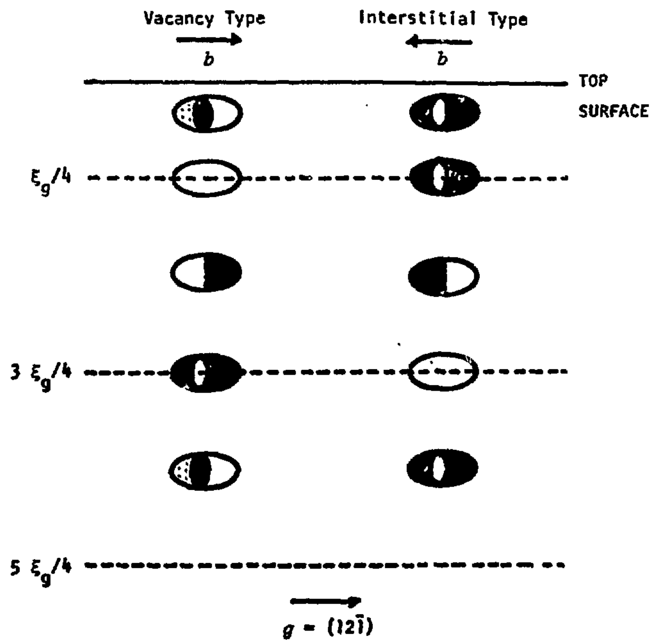

FIG. 3

Bright field image contrast of a dislocation loop with Burgers vector $\pm\frac{a}{2}[111]$ in niobium.