# DISLOCATION DIPOLES IN ELASTICALLY ANISOTROPIC CRYSTALS

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#### Abstract

The major features of the images of dislocation dipoles obtained by two-beam electron microscopy which enable dipoles to be identified and distinguished from other defects are discussed. The positions of stable equilibrium of dipoles in various f.c.c. and b.c.c. materials have been derived using linear anisotropic elasticity. The results are presented as curves of equilibrium orientation versus screw-edge orientation of the dipole and in most cases these show marked differences from the equivalent curves deduced using isotropic elasticity. The above results have been combined and applied to the case of high purity nickel where dipoles having dislocation orientations of  $30^{\circ}$ ,  $60^{\circ}$ , and  $90^{\circ}$  from screw orientation have been identified. It has been shown by image matching that the  $30^{\circ}$  and  $60^{\circ}$  dipoles are very close to the equilibrium positions predicted by theory. In fact, the accuracy of determination of the alignment angle in the case of the  $30^{\circ}$  dipole indicates that the forces on the dislocations resolved in their slip planes must be less than 0.5 dyne cm<sup>-1</sup>. This may be taken as a measure of the lattice frictional force in nickel. For many screw-edge orientations there are two possible positions of equilibrium and it has been shown from energy considerations that for dipoles formed by slip, in f.c.c. materials, one of these positions will be favoured. The 90° dipole has a particularly small separation and it was found necessary to consider each dislocation as a Shockley pair separated by stacking fault. The faults were identified as intrinsic and, from the observed separation of the Shockley partial dislocations and the evaluation of the forces on these in the stable equilibrium position, it was estimated that the stacking fault energy was  $\sim 80 \,\mathrm{erg}\,\mathrm{cm}^{-2}$ . This is less than half the value obtained by other methods, but it is suggested that it might be representative of the values which apply to configurations such as those identified here in which the dissociation of the dislocations is small.

## I. INTRODUCTION

A dislocation dipole (i.e. parallel segments of dislocations with opposite Burgers vectors) constitutes the simplest stable dislocation array. Dipoles are often formed during the initial stages of plastic deformation of a crystal, and when they are present in large numbers they can have a significant influence on the electrical and thermal, as well as mechanical properties of crystals (see e.g. Gilman 1964). The correlation between these properties and the presence of dislocation dipoles must rely, to some extent, upon a knowledge of the nature and geometry of the dipoles present in the material and on the way dipoles are formed. A suitable means of detecting and identifying dislocation dipoles is by the use of transmission electron microscopy of thin metal foils. In Section II, the essential features of the two-beam electron diffraction image produced by a dipole are derived. This makes it possible for the image of a dislocation dipole to be readily distinguished from that of a single dislocation. In Section III, a study is made of the positions of stable equilibrium for dipoles formed on the most common slip systems of different cubic metals. These

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results show that, even in metals which have only slight elastic anisotropy, positions of stable equilibrium can differ markedly from those predicted by isotropic calculations. Both these points are demonstrated in Section IV by observations on dipoles in nickel. Using electron microscopy and the visual matching technique of Head (1967*a*) and Humble (1968) involving experimental and theoretical electron micrographs, dipoles whose dislocations are  $30^{\circ}$ ,  $60^{\circ}$ , and  $90^{\circ}$  from screw orientation have been identified. In the first two cases the dipoles are almost certainly in their stable equilibrium configurations as predicted by anisotropic elasticity theory, and in the third case probably so. For the  $90^{\circ}$  case the dislocations were found to be dissociated into Shockley partial dislocations and it was possible to make an estimate of the stacking fault energy of nickel. In the discussion in Section V, the examples of dislocation dipoles observed in nickel are used to indicate how dipoles may be employed to obtain information about various properties of metals.

In this paper dislocation dipoles in pure screw orientation will not be considered since they can annihilate by cross slip, and therefore would not be expected to be observed in practice.



Fig. 1.—Generalized cross section for a dislocation dipole in a foil tilted with respect to the electron beam direction Oz. The origin O is midway between the component dislocations of the dipole which have Burgers vectors + b and - b.

II. ELECTRON DIFFRACTION IMAGES OF DISLOCATION DIPOLES

In this section we consider the properties of the bright-field images of a dislocation dipole. It will be assumed that the dislocations are straight and parallel and run completely through a foil of uniform thickness. The properties discussed hold for materials of any elastic anisotropy, but it is assumed that the crystals are centro-symmetric.

The main feature of the images of dislocation dipoles which distinguishes them from single dislocations is that they all possess a centre of inversion. Such symmetry has been shown for a line of dilation (Chadderton 1964; Humble 1969) which may be regarded as an edge dipole of one atomic plane separation, and may easily be deduced for a dipole of wide separation (i.e. two oppositely signed dislocations whose images do not overlap) from the properties of isolated dislocations (Howie and Whelan 1961). It may be demonstrated in the general case by examining the form of the combined displacement fields of the two dislocations. This, in turn, can be done for instance by considering the way in which a computed micrograph of a dipole is formed. Figure 1 shows the generalized cross section (a plane containing the beam direction and onto which all displacements may be uniquely projected (Head 1967a; Humble 1968)) for two dislocations of Burgers vectors **b** and  $-\mathbf{b}$ . The origin of coordinates O is midway between the dislocations which are coming out of the paper but are not normal to it. Consider columns at -y and +y and consider the vector displacement fields at points +z and -z in these columns respectively. It is clear from the symmetry of the situation about the origin that the displacement  $\mathbf{R}$  at (-y,z) is equal and opposite to that at (y, -z) and indeed this is true for all points which are symmetrically related in this manner. That is,  $\mathbf{R}(z)$  for the column at -y is equal to  $-\mathbf{R}(t-z)$  for the column at +y, where t is the thickness of the generalized cross section. It follows from the properties of centro-symmetric crystals (Howie and Whelan 1961) that the bright-field intensities in the row of the picture which result from integrating the differential equations\* governing the diffraction of the electrons down the column at -y will be equal to those resulting from the column at +y, but will be in the reverse order. Thus, since the rows are equally spaced on either side of the dipole, the image will have a centre of symmetry. This is amply demonstrated by the experimental and theoretical micrographs shown in this paper.

Although the above discussion is based on the use of the generalized cross section, it is important to note that the result is not affected by the main limitation of that construction, namely that relaxations of the surface are not allowed. Providing the relaxations occur equally on both surfaces (and this may not be the case if



Fig. 2.—Four theoretical micrographs of dislocation dipoles in aluminium illustrating the variation in the images for two values of separation with  $\pm g$  (see text for details).

contamination of the surfaces takes place unequally, for example), then the images will still have a centre of inversion. This is true for any degree of anisotropy. In addition, the arguments also hold if the dislocations are dissociated into partial dislocations provided that the configuration of separations, Burgers vectors, and type of fault are symmetric about the origin. Images having a centre of inversion will not, of course, be obtained if the dislocations are not parallel (see e.g. the defect C in Fig. 9).

Another property which the bright-field images of dislocation dipoles possess is that in general the image formed with a reflecting vector g has no relation to the image formed with -g. This is obviously not the case for large separations, since then the displacement fields of the two dislocations do not overlap appreciably and the images are essentially those of isolated dislocations. In this case the image for -g may be obtained approximately by rotating the image of each dislocation for +g

\* The property of inversion symmetry of the bright-field image deduced here holds for many-beam diffracting conditions although the experimental and computed micrographs shown in this paper all refer to two beams. through 180°. These points are illustrated in Figure 2, which shows four theoretical micrographs of a dipole that lies along the direction  $\boldsymbol{u} = [\overline{112}]$  and whose Burgers vectors are  $\pm \frac{1}{2}[\overline{101}]$ . Figures 2(a) and 2(c) have a reflecting vector of  $\overline{111}$  and 2(b) and 2(d) have a reflecting vector of 111. In (a) and (b) the separation of the two dislocations is 35 Å and in (c) and (d) it is 1400 Å.

The diffraction conditions necessary to put a dipole completely out of contrast are, of course, the same as those for single dislocations in elastically anisotropic media. Since in this paper we are not considering dipoles whose dislocations have screw orientation, the only case in which no contrast can occur is when the dislocations are pure edge. The conditions that these will be completely out of contrast are: (i) that they lie normal to an elastic symmetry plane in the crystal\* and (ii) that the reflecting vector lies along the line of the dislocations. It should be mentioned that there may be other occasions when the contrast from a dipole is so weak that it cannot be distinguished from background intensity. However, it is not possible to predict when these cases will occur.

### III. STABLE EQUILIBRIUM OF DIPOLES

The interaction between the displacement fields of two distant parallel dislocations with opposite Burgers vectors situated in an infinite crystal is such that the dislocations are attracted towards each other. If the dislocations are constrained to move on parallel non-coincident slip planes then they will eventually take up a stable configuration of minimum energy. Following Stroh (1958), who investigated



Fig. 3.—Schematic diagrams of dislocation dipoles with (a) undissociated dislocations and (b) symmetrically dissociated dislocations.

the positions of equilibrium of edge dislocations of like Burgers vectors, we have found the positions of equilibrium of dipoles by locating one dislocation at the origin and examining the force, resolved in the slip plane, on the other dislocation as this is moved from a large distance on one side of the origin to a large distance on the other side. In general there is at least one and often two positions of stable equilibrium. In cases where there are two positions of stable equilibrium these are always separated by a position of unstable equilibrium corresponding to where two dislocations of like Burgers vector would align. The fact that, in a general anisotropic elastic solid, there is always an odd number of equilibrium positions (both stable and unstable) was originally shown by Stroh (1958).

\* For crystals which are elastically anisotropic, the elastic symmetry planes are those which possess mirror crystal symmetry (e.g. {100} and {110} in cubic crystals). For elastically isotropic crystals, any plane in the crystal is an elastic symmetry plane.

#### IMAGES OF DISLOCATION DIPOLES

### (a) Elastically Isotropic Crystals

In the case of an elastically isotropic solid, positions of stable equilibrium for dipoles can be easily evaluated using the expressions given by Cottrell (1953). Consider two dislocations 1 and 2, as shown in Figure 3(a), which are parallel to Oz with respective edge and screw components of Burgers vectors  $\pm b_e$  along Oxand  $\pm b_s$  along Oz so that both dislocations have slip planes parallel to zOx. Let dislocation 1 be positioned at the origin and dislocation 2 at the point (x, y). Then the component of interaction force,  $F_x$ , parallel to Ox which acts on 2 due to the presence of 1 and which can cause relative motion between the two dislocations is given by

$$F_x = rac{-\mu b_{
m e}^2 x}{2\pi (1-
u)(x^2+y^2)^2} \left(x^2 - y^2 + rac{b_{
m s}^2}{b_{
m e}^2}(1-
u)(x^2+y^2)
ight),$$

where  $\mu$  is the shear modulus and  $\nu$  is Poisson's ratio. Positions of equilibrium are given when  $F_x = 0$ ; that is, when

$$x = 0$$
 or  $x = \pm y\{1 - K^2(1 - \nu)\}^{\frac{1}{2}}/\{1 + K^2(1 - \nu)\}^{\frac{1}{2}},$  (1)

where  $K = b_s/b_e$ , so that the character of the dipole can be specified as  $\cot^{-1} K$  from screw orientation. x = 0 is the only position of stable equilibrium when  $K^2 \ge 1/(1-\nu)$ , and there are no real positions of unstable equilibrium. For  $K^2 < 1/(1-\nu)$ , the positions of stable equilibrium are given by expressions (1) and in this case x = 0is a position of unstable equilibrium.



Fig. 4.—Curves showing positions of stable equilibrium versus screw-edge orientation for dislocation dipoles in three different elastically isotropic materials with Poisson's ratio  $\nu$  equal to  $\frac{1}{2}$ ,  $\frac{1}{3}$ , and -1.

In Figure 4 positions of stable equilibrium, denoted by the angle  $\phi = \tan^{-1} y/x$  between the slip plane and the plane containing the two dislocations, have been plotted against the screw-edge character of the dipole,  $\cot^{-1} K$ . In the figure, three curves given by expressions (1) are shown which correspond to three values of Poisson's ratio ( $\nu = \frac{1}{2}, \frac{1}{3}, -1$ ). These curves show that the dislocations close to screw orientation which form dipoles are aligned normal to their slip planes,  $\phi = 90^{\circ}$ .

This condition holds until the dislocation orientation exceeds  $\cot^{-1}\{1/(1-\nu)^{\frac{1}{2}}\}$  from screw, when each curve divides into two branches corresponding to two positions of stable equilibrium. These are symmetrical about the normal to the slip plane and there are positions of unstable equilibrium at  $\phi = 90^{\circ}$ . As expected, for elastic isotropy, the dipole equilibrium curves are symmetrical about screw and edge orientations.

#### (b) Elastically Anisotropic Crystals

In the case of anisotropic crystals an analytic solution for the positions of stable equilibrium of dipoles is not in general possible. This is because the evaluation of the stress field around a dislocation requires the solution of a sextic algebraic equation involving the elastic constants of the crystal. However, the sextic equation can be solved numerically and a set of parameters resulting from this solution can then be substituted into an expression for  $F_x$  given by Stroh (1958). The necessary set of parameters was obtained from a computer program originally written by Head (1967a) as part of a program for computing theoretical micrographs. The parameters were substituted into the expression for  $F_x$  and positions of dipole equilibrium were found in the way described at the beginning of this section. Since

VALUES OF ELASTIC CONSTANTS USED						
Metal	c <sub>11</sub> (10	$c_{12}$ <sup>11</sup> dyne cn	$c_{44}$ n <sup>-2</sup> )	Reference		
Nickel	$24 \cdot 65$	14.73	$12 \cdot 47$	Huntington (1958)		
Lead	4.66	$3 \cdot 92$	$1 \cdot 44$	Huntington (1958)		
Indium(28·13%)thallium	$4 \cdot 01$	$3 \cdot 95$	0.83	Novotny and Smith (1965)		
Niobium	$24 \cdot 6$	$13 \cdot 40$	$2 \cdot 87$	Hearmon $(1966)$		
Iron	$23 \cdot 31$	$13 \cdot 54$	11.78	Ravne and Chandrasekhar (1961)		
$\beta$ -brass	$12 \cdot 91$	$10 \cdot 97$	$8 \cdot 24$	Huntington (1958)		

TABLE 1

all the calculations were carried out numerically, only specific examples of dipoles have been considered and these have been confined to the most common dipoles which occur in f.c.c. and b.c.c. metals. The positions of equilibrium for these dipoles are represented by the sets of curves shown in Figures 5, 6, 7, and 8 which have been plotted in a manner consistent with those for elastic isotropy. The elastic constants used to calculate the curves in Figures 5 and 6 are given in Table 1. The variation in screw-edge orientation has been achieved by varying the line vector  $\boldsymbol{u}$  of the dipole whilst its Burgers vectors have been kept fixed. The angle  $\phi$  is measured from the slip plane to the plane containing the dislocations in the sense of a righthanded screw whose axis is along the  $\boldsymbol{u}$  of the dipole.

Figure 5 shows the curves of stable equilibrium for the dipole  $\pm \frac{1}{2}[011](11\overline{1})$ in three f.c.c. metals. Close to screw orientation the dislocations which form the dipole are not aligned normal to the slip plane, as is the case for elastic isotropy, but have a single position of equilibrium that is acute with the slip plane. As the orientation from screw increases, the positions of equilibrium become more acute with the slip plane and at a particular orientation a second branch of the curve is



Fig. 5.—Curves showing positions of stable equilibrium versus screw-edge orientation for  $\pm \frac{1}{2}[011](111)$  dislocation dipoles in three f.c.c. metals: nickel, lead, and indium(28.13 at.%)-thallium. The dashed regions of the curve for indium-thallium are screw-edge orientations where single dislocations are unstable against kinking.



Fig. 6.—Curves showing positions of stable equilibrium versus screw-edge orientation for dislocation dipoles in three b.c.c. metals. The curves for niobium and iron refer to  $\pm \frac{1}{2}[111](\overline{110})$ dipoles and that for  $\beta$ -brass refers to  $\pm [111](\overline{110})$  dipoles. The dashed regions of the curve for  $\beta$ -brass are screw-edge orientations where single dislocations are unstable against kinking.



Fig. 7.—Curves showing positions of stable equilibrium versus screw-edge orientation for  $\pm \frac{1}{2}[011](11\overline{1})$  dislocation dipoles in five f.c.c. materials with different degrees of elastic anisotropy. The elastic constant ratio  $\mathscr{A}$  is indicated for each curve and the value of  $\mathscr{A} = 8.0$ . The dashed regions of the curve for  $\mathscr{A} = 8.0$  are screw-edge orientations where single dislocations are unstable against kinking.



Fig. 8.—Curves showing positions of stable equilibrium versus screw-edge orientation for  $\pm \frac{1}{2}[111](\overline{110})$  dislocation dipoles in five b.c.c. materials with different degrees of elastic anisotropy. The elastic constant ratio  $\mathscr{A}$  is indicated for each curve and the value of  $\mathscr{A}$  is  $8 \cdot 0$ . The dashed regions of the curves for  $\mathscr{A} = 4 \cdot 0$  and  $8 \cdot 0$  are screw-edge orientations where single dislocations are unstable against kinking.

generated which corresponds to positions of stable equilibrium on the other side of the slip plane normal. However, the positions of equilibrium are not symmetrical either side of the slip plane normal. There are positions of unstable equilibrium at or close to  $\phi = 90^{\circ}$  between the two branches of stable equilibrium, but at no orientation is there a position of stable equilibrium normal to the slip plane.

Figure 6 contains a set of curves of stable equilibrium for dipoles of the type  $\pm \frac{1}{2}[111](\overline{1}10)$  in niobium and iron, and for dipoles of the type  $\pm [111](\overline{1}10)$  in  $\beta$ -brass. As in the case of elastic isotropy these curves have a range of screw-edge orientation in which the dislocations comprising the dipole are aligned normal to their slip plane. However, this range of orientation is not necessarily close to screw orientation as can be seen from the curve for  $\beta$ -brass. Outside this orientation range the curves divide into two branches which are symmetrical about  $\phi = 90^{\circ}$ . Between these two branches there are positions of unstable equilibrium normal to the slip plane.

In the case of  $\beta$ -brass (Fig. 6) and indium-thallium (Fig. 5), which are very elastically anisotropic, there are ranges of screw-edge orientation for which the dislocations that form dipoles will be unstable against kinking (Head 1967b), and these ranges are shown as dashed lines in Figures 5 and 6.

The general form of the equilibrium curves calculated using anisotropic elasticity has changed from that calculated by isotropic elasticity in a manner determined by the relationship between the crystal symmetry and the slip system. The curves are symmetrical about any pure screw or pure edge orientation which is normal to an elastic symmetry plane. In addition, they will be symmetrical about  $\phi = 90^{\circ}$ when the slip plane is an elastic symmetry plane. Thus, the curves for f.c.c. metals (Figs 5 and 7) are symmetrical about screw orientation only, and the curves for b.c.c. metals (Figs 6 and 8) are symmetrical about  $\phi = 90^{\circ}$  only.

Figures 7 and 8 are curves of stable equilibrium for  $\pm \frac{1}{2}[011](11\overline{1})$  and  $\pm \frac{1}{2}[111](\overline{1}10)$  dipoles respectively, which illustrate the general change of dipole equilibrium with increasing elastic anisotropy for f.c.c. and b.c.c. metals. They are referred to the dimensionless ratios of elastic constants  $\mathscr{A} = 2c_{44}/(c_{11}-c_{12})$  and  $\mathscr{B} = (c_{11}+2c_{12})/c_{44}$ , where an elastically isotropic crystal with a Poisson's ratio  $\nu$  of  $\frac{1}{3}$  can be designated by  $\mathscr{A} = 1 \cdot 0$ ,  $\mathscr{B} = 8 \cdot 0$ . It is quite clear that a slight deviation from isotropy, particularly in the case of the  $\pm \frac{1}{2}[011](11\overline{1})$  dipoles, can have a marked effect on the positions of stable equilibrium. For instance, when  $\mathscr{A}$  changes from  $1 \cdot 0$  to  $1 \cdot 2$  the stable equilibrium of a  $\pm \frac{1}{2}[011](11\overline{1})$  dipole which is 38° from screw orientation changes from  $\phi = 90^{\circ}$  to  $\phi = 75^{\circ}$ . Since linear elasticity has been used to calculate the positions of equilibrium the absolute magnitude of the Burgers vector is unimportant in determining the equilibrium curves. Therefore, the curves in Figures 7 and 8 apply equally well to dipoles of the type  $\pm [011](11\overline{1})$  and  $\pm [111](\overline{1}0)$  which can sometimes occur in ordered metals.

## IV. Identification of Dipoles in Nickel

The above findings have been applied to the identification of dipoles in nickel using two-beam transmission electron microscopy. The elastic constants used for nickel are those given in Table 1 and these give values of the elastic ratios of  $\mathscr{A} = 2 \cdot 52$  and  $\mathscr{B} = 4 \cdot 35$  so that nickel would not be considered to be very elastically anisotropic.

Figure 9 is an electron micrograph of a region from a nickel specimen which has been quenched from a temperature close to its melting point under a vacuum of the order of  $10^{-6}$  torr into silicone oil. It was found that this treatment often produced dipoles and three examples A, B, and D, which may be identified as such from their centre of inversion images, can be seen in Figure 9. The defect at C is probably also a dipole whose dislocations are not parallel. It can be seen from the dipole images that the individual dislocations comprising the dipoles can be resolved easily in the case of A, with difficulty in the case of B, and not at all in the case of D.

The dipoles at A, B, and D have been identified by comparing their experimental images, taken under various diffracting conditions, with theoretical micrographs computed for the same diffracting conditions but scanned over all possible Burgers vectors. When computing the theoretical micrographs, the displacement fields around the dislocations were, of course, determined using anisotropic elasticity. The appropriate equilibrium curves for nickel (Figs 5 or 15) were used as the basis for determining the relative geometry of the dislocations.

## Dipole A

Figure 10(a) shows five images of A taken under different reflecting conditions. The u of the dipole was found to be [ $\overline{112}$ ]. Figure 10(b) shows the five corresponding theoretical images computed on the basis that A is a 30° dipole with Burgers vectors of  $\pm \frac{1}{2}[10\overline{1}]$  which has an equilibrium angle  $\phi$  of 60°. It is apparent that the theoretical images are a good match to the experimental ones. Theoretical images computed for other Burgers vectors of the type  $\pm \frac{1}{2}\langle 110 \rangle$  and their corresponding equilibrium angles proved to be gross mismatches. The defect A was therefore identified as a  $30^{\circ}$  dipole with a  $u = [\overline{112}]$ , Burgers vectors of  $\pm \frac{1}{2}[10\overline{1}]$ , and a separation between the slip planes of 416 Å.

The accuracy to which the alignment angle agrees with the equilibrium angle  $\phi$  may be estimated by examining theoretical micrographs for a range of angles about  $\phi = 60^{\circ}$  and then comparing these with the experimental images. This has been done in Figures 11(a), 11(b), and 11(c) for the experimental images (1), (2), and (3) shown in Figure 10(a). In Figure 11, the alignment angle varies from  $55^{\circ}$  to  $65^{\circ}$ in steps of  $2\frac{1}{2}^{\circ}$ . In Figure 11(a) where the dislocations comprising the dipole are clearly resolved, the character of the dislocation images is almost unchanged by the variation in alignment angle but the degree of "off-set" of one dislocation image with respect to the other does alter. Using this geometrical off-set criterion it can be seen that the images corresponding to alignment angles of  $55^{\circ}$  and  $65^{\circ}$  are not a match to the experimental image. In Figures 11(b) and 11(c) the images of the component dislocations overlap considerably and the character of the resultant dipole image varies markedly with variations in angle. For instance, the main features of the images in Figure 11(b) which change are: the shape of the light central region and the width, intensity, and inclination to the dipole axis of the dark regions on either side of this central region. The images corresponding to alignment angles of 55° and 65° are bad matches to the experimental image and of the remaining three the one for an alignment angle of 60° is by far the best match. Similar considerations applied to the central features of Figure 11(c), where changes in image character are most sensitive to changes in alignment angle, lead to the rejection of images for 65° and 55°.

Thus it can be concluded that  $\phi = 60^{\circ} \pm 2\frac{1}{2}^{\circ}$  and to this accuracy therefore the dipole A is in the equilibrium configuration predicted by anisotropic elasticity for the given elastic constants of nickel.

It is interesting to note that in the case where nickel is considered to be elastically isotropic with  $\nu = 0.32$  ( $\mathscr{A} = 1.0$ ,  $\mathscr{B} = 7.34$ ) dipole A would be aligned with  $\phi = 90^{\circ}$ , and the computed micrograph that would then correspond to image (2) in Figure 10(a) is that shown in Figure 12. It is quite apparent that this is no match.



Fig. 9.—Bright-field transmission electron micrograph ( $\times 42000$ ) of a thin foil of quenched nickel containing dislocation dipoles at A, B, and D. The beam direction is close to [213], the foil normal is close to [419], and the diffracting vector  $11\overline{1}$  is indicated.

### Dipole B

In the case of the 30° dipole discussed above, there is only one possible equilibrium angle. However, preliminary computations for defect B in Figure 9 whose u is [011] showed that it could be identified as a 60° dipole and in this case there are two possible equilibrium angles,  $\phi = 45 \cdot 5^{\circ}$  and  $123 \cdot 5^{\circ}$  (see Fig. 5). In Figure 13, (a) shows five images of the defect B taken under different reflecting conditions, and (b) shows the corresponding set of theoretical images computed on the basis that  $u = [0\overline{1}1]$ ,  $b = \pm \frac{1}{2}[10\overline{1}]$ ,  $\phi = 45 \cdot 5^{\circ}$ , and the separation between the slip planes is 113 Å. It can be seen that these theoretical micrographs are a good match to the experimental images. Theoretical images computed for the other Burgers vectors of the  $\pm \frac{1}{2}\langle 110 \rangle$  type and for the other possible equilibrium angle of a 60° dipole,  $\phi = 123 \cdot 5^{\circ}$ , did not match the experimental images. As in the previous case,

theoretical images were computed for a range of alignment angles about the calculated equilibrium value of  $45 \cdot 5^{\circ}$ , but due to the insensitivity of the available images in this case,  $\phi$  could only be verified to  $\pm 5^{\circ}$ . Theoretical micrographs calculated on the basis that nickel was elastically isotropic, with  $\nu = 0.32$  and  $\phi = 51 \cdot 5^{\circ}$ , did not match the experimental images.



Fig. 10.—Comparison of (a) five bright-field transmission electron micrographs of dipole A (Fig. 9), whose  $\boldsymbol{u} = [\overline{112}]$ , with (b) corresponding computed images of a 30° dislocation dipole. In (a) the beam directions are close to [101] for (1), (2), (4), and (5) and close to [112] for (3). The diffracting vectors are indicated. In (b) the Burgers vectors are  $\pm \frac{1}{2}[10\overline{1}], \phi = 60^{\circ}$ , the separation between the (111) slip planes is 416 Å, and the diffracting conditions are those of the corresponding micrographs in (a).

## Dipole D

The defect D in Figure 9, whose u was determined as  $[1\overline{2}1]$  was considerably more difficult to identify than the previous two cases. Five experimental images of dipole D taken under different reflecting conditions are shown in Figure 14(*a*). Preliminary computations indicated that the experimental images were approximately matched on the assumption that the component dislocations were in edge orientation with Burgers vectors  $\pm \frac{1}{2}[10\overline{1}]$  and separated by a distance of about

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20 Å at  $\phi = 44^{\circ}$ . All other possible configurations of dipoles with Burgers vectors of the type  $\pm \frac{1}{2} \langle 110 \rangle$  proved to be very bad matches to the experimental images. In order to improve the approximate match it was decided to investigate configurations where the  $\pm \frac{1}{2} [10\overline{1}]$  dislocations were split into Shockley partial dislocations



Fig. 11.—Three sets of five computed electron micrographs for the 30° dislocation dipole with  $u = [\overline{112}]$  and Burgers vectors  $\pm \frac{1}{2}[10\overline{1}]$ . The alignment angles are (1) 55°, (2) 57.5°, (3) 60°, (4) 62.5°, and (5) 65°. The diffracting conditions of (a), (b), and (c) correspond to those of (1), (2), and (3) respectively in Figure 10(a).



Fig. 12.—Theoretical micrograph for the  $30^{\circ}$  dipole with  $\boldsymbol{u} = [\overline{11}2]$  and Burgers vectors  $\pm \frac{1}{2}[10\overline{1}]$ . It is computed on the assumption that nickel is isotropic with Poisson's ratio  $\nu = 0.32$  and  $\phi = 90^{\circ}$ . The diffracting conditions correspond to those of (2) in Figure 10(*a*).

which bound the appropriate stacking faults. Such a configuration was considered to be of importance in this case because, even with the relatively large stacking fault energy of nickel (see Section V), the dissociation of the edge dislocations would be comparable with their 20 Å spacing. This in itself could have a marked effect upon the dipole images and in addition would affect the equilibrium position of the dipole. Each dislocation can dissociate to produce intrinsic or extrinsic faulting and thus there are four possible cases to consider: where dislocations 1 and 2 in Figure 3(a)dissociate as (i) intrinsic-intrinsic, (ii) extrinsic-extrinsic, (iii) intrinsic-extrinsic, or (iv) extrinsic-intrinsic. A computer program of the type described in Section III but which takes into account the equilibrium of four dislocations and two stacking



Fig. 13.—Comparison of (a) five bright-field transmission electron micrographs of dipole B (Fig. 9), whose  $\mathbf{u} = [0\bar{1}1]$ , with (b) corresponding computed images of a 60° dislocation dipole. In (a) the beam directions are close to [101] for (1), (2), (3), and (5) and close to [112] for (4). The diffracting vectors are indicated. In (b) the Burgers vectors are  $\pm \frac{1}{2}[10\bar{1}]$ ,  $\phi = 45 \cdot 5^{\circ}$ , the separation between the slip planes is 113 Å, and the diffracting conditions are those of the corresponding micrographs in (a).

faults (Morton and Forwood, to be published) was used to determine the positions of stable equilibrium. It was found that, in stable equilibrium, the dissociations of dislocations 1 and 2 were equal for case (i) and equal for case (ii), but unequal for case (iii) and unequal for case (iv) even if identical values of intrinsic and extrinsic stacking fault energies were used in the last cases.

The magnitude of the dissociation depends of course on the stacking fault energy, and sets of dislocation dissociations and dipole separations were obtained from the equilibrium calculations for a range of stacking fault energies. These were then used in turn to compute theoretical micrographs under the diffraction conditions corresponding to the experimental micrographs. Although the approximate match obtained was for a dipole with Burgers vectors  $\pm \frac{1}{2}[10\overline{1}]$ , when considering dissociated dipoles, all possible  $\pm \frac{1}{2}\langle 110 \rangle$  Burgers vectors were examined. Matches could only



Fig. 14.—Comparison of (a) five bright-field transmission electron micrographs of dipole D (Fig. 9), whose  $\boldsymbol{u} = [1\overline{2}1]$ , with (b) corresponding computed images of a dissociated edge dislocation dipole of the intrinsic-intrinsic type. In (a) the beam directions are close to [101] for (1), (2), and (3), to [112] for (4), and to [001] for (5). The diffracting vectors are indicated. In (b) the Burgers vectors are  $\pm (\frac{1}{6}[\overline{1}12] + \frac{1}{6}[\overline{2}11]), \ \theta = 46 \cdot 5^{\circ}, \ S/Y = 1 \cdot 1, \ Y = 22 \cdot 4$ Å (see Fig. 3(b)), and the diffracting conditions are those of the corresponding micrographs in (a).

be obtained for intrinsic-intrinsic dipoles whose total Burgers vectors were  $\pm \frac{1}{2}[10\overline{1}]$ . If the separation of the partial dislocations is S, the separation between the slip planes is Y, and the equilibrium angle  $\theta$  is defined as shown in Figure 3(b), then an equilibrium curve of S/Y versus  $\theta$  can be evaluated. This is shown in Figure 15. For a fixed value of separation Y, each point on the equilibrium curve corresponds to a different value of stacking fault energy which decreases as S/Y increases. However, for a given point on the equilibrium curve (i.e. for fixed values of S/Y and  $\theta$ ), the stacking fault energy is inversely proportional to the separation Y (Morton and Forwood). The equilibrium curve in Figure 15 consists of two distinct regions of stability,  $0 < S/Y \leq 1.18$  (region 1) and  $S/Y \geq 1.08$  (region 2), separated by a region of unstable equilibrium. Theoretical micrographs computed for region 1 with various values of Y did not match the experimental images, whereas those computed for region 2 with  $1.1 \leq S/Y \leq 1.25$  and 18 Å  $\leq Y \leq 24$  Å proved to be reasonable matches. This range of variables corresponded to values of intrinsic stacking fault energy  $\gamma$  in the range 100 to 73 erg cm<sup>-2</sup>. The best match to the images in Figure 14(*a*) is shown in Figure 14(*b*) for which S/Y = 1.1, Y = 22.4 Å, and  $\gamma = 79$  erg cm<sup>-2</sup>.\*



Fig. 15.—Curve showing positions of equilibrium for a  $\pm \frac{1}{2}\langle 110 \rangle$  edge dislocation dipole in nickel dissociated on {111}. Both dislocations are dissociated intrinsically according to the geometry of Figure 3(b). The values at the points on the curve are values of stacking fault energy in erg cm<sup>-2</sup> when  $Y = 5a_0 = 17.62$  Å.

#### V. Discussion

In the preceding section it has been shown how dislocation dipoles in nickel can be precisely identified in terms of their nature and geometry by using image matching in conjunction with equilibrium calculations based on linear anisotropic elasticity. In this section it is indicated how observations made in this way can be used to investigate certain properties of metals which contain dipoles. The topics discussed include: (a) the measurement of elastic constant ratios  $\mathscr{A}$  and  $\mathscr{B}$ ; (b) the measurement of lattice frictional stresses or other small forces acting on the dislocations; (c) the magnitude and asymmetry of the forces acting on the dislocations during deformation; (d) the relative importance of the two branches of the equilibrium curve; and (e) the measurement of stacking fault energy.

\* The dimensions of all three dipoles A, B, and D considered above have been obtained directly from the matching sets of theoretical micrographs in terms of two-beam extinction distances. The dimensions quoted have been converted into Angstrom units using values for two-beam extinction distances in nickel of:  $\xi_{111} = 236$  Å,  $\xi_{200} = 275$  Å,  $\xi_{220} = 409$  Å, and  $\xi_{311} = 499$  Å. These values are based solely on theoretical calculations but a comparison of the matching set of theoretical micrographs for dipole A with measurements from the experimental micrographs showed that the extinction distances were applicable to within  $\pm 5\%$ .

#### IMAGES OF DISLOCATION DIPOLES

The properties under discussion are illustrated using the particular dipoles which have been identified in nickel. A possible objection to this procedure is that although the calculations apply to dipoles attaining equilibrium by slip, the observations have been made on quenched material in which equilibrium might have been influenced by the presence of a high vacancy concentration. It should be pointed out, however, that it follows from the detailed identification which we have made that all three dipoles are of the type which would annihilate by the accretion of vacancies. Thus it is likely that they were formed by the stresses produced in the metal during quenching.

#### (a) Measurement of Elastic Constant Ratios

If it is assumed that dipoles are in equilibrium, observation of their images produced in the electron microscope may be used to determine the elastic constant ratios of the localized pieces of crystal in which they lie. The way to do this is to use the information from equilibrium curves, computed for a range of values of  $\mathscr{A}$  and  $\mathscr{B}$  for the particular slip system under consideration (e.g. Figs 7 and 8), to compute sets of theoretical micrographs (which themselves depend on  $\mathscr{A}$  and  $\mathscr{B}$ ). These are then compared with the experimental micrographs until a match is obtained; the dipole is thus identified and the values of  $\mathscr{A}$  and  $\mathscr{B}$  determined. The accuracy of the values is governed by the range over which a tolerable match exists. The accuracy is obviously increased if the dipole chosen has a screw-edge orientation where the equilibrium angle  $\phi$  at that point changes rapidly with small changes in  $\mathscr{A}$  and  $\mathscr{B}$ . This method of determining  $\mathscr{A}$  and  $\mathscr{B}$  can be applied to crystals of a wide range of elastic anisotropy and supplements the method due to Morton and Head (1970) using kinked dislocations which can only be applied to crystals of large anisotropy.

This method has been applied to the case of the 30° dipole in nickel (dipole A) and the resulting values for the elastic constant ratios of nickel are  $\mathscr{A} = 2 \cdot 6 \pm 0 \cdot 2$  and  $\mathscr{B} = 4 \cdot 4 + 2 \cdot 2$ .

## (b) Measurement of Lattice Frictional Stress

If the elastic constants of nickel are assumed to be those given by Huntington (1958) then the experimental error of  $\pm 2\frac{1}{2}^{\circ}$  in the equilibrium angle obtained by image matching for dipole A corresponds to positions of the dislocations in their slip planes where any resolved force acting on the dislocations would have to be less than or equal to about 0.5 dyne cm<sup>-1</sup>. This may be taken as a measure of the lattice frictional force for nickel operating on these dislocations, and corresponds to a value of frictional stress equal to about  $3 \times 10^{-5}$  of the shear modulus.

This value of the frictional stress is extremely small, and clearly observations on dipoles made in conjunction with equilibrium calculations may be used quite generally to determine the frictional force or any other small force acting on dislocations. One factor which would determine the accuracy with which the force could be measured is the magnitude of the restoring force which acts on the dislocations and this is inversely proportional to the separation of the dipole. Thus it would be advantageous to work with widely separated dipoles where the restoring forces are small. A second factor which introduces a source of uncertainty is, of course, the accuracy with which the alignment angle of the dislocations can be measured. The accuracy can probably be increased to the order of  $\pm 1^{\circ}$  with more careful image matching and by selecting electron micrographs which are most sensitive to this parameter. From the examples analysed in Section IV, it is apparent that the alignment angle can be most accurately determined when the dislocation images interact strongly (e.g. Figs 11(b) and 11(c)). Although this point has not been investigated in depth, it is thought that this condition would be best satisfied for images which have beam directions close to the plane of the dislocations.

### (c) Magnitude and Asymmetry of Forces during Deformation

Another aspect of the forces acting within a dipole concerns the maximum values of the force (resolved in the slip plane) which one dislocation exerts on the other as it is moved past, i.e. the maximum value of  $F_x$ . These maxima occur on each side of the positions of unstable and stable equilibrium, and are, of course, related to the stress which must be applied to deform a crystal containing dislocations of opposite sign. In Table 2 the values of the maxima in  $F_x$  for the  $\pm \frac{1}{2}\langle 110 \rangle \{111\}$  slip system in nickel are listed for the cases of the 30°, 60°, and 90° dipoles. They

TABLE 2						
MAXIMA IN ${oldsymbol{F}}_x$ for	$\pm \frac{1}{2} \langle 110 \rangle \{111\}$ slip system in nickel compared v	wiтн				
ISOTROPIC ELASTICITY VALUES						

Screw-Edge Orientation of Dipole	Maxima in $F_x$			
	Anisotropic Elasticity (in units of $c_{44} a_0 \mathrm{cm}^{-1}$ )	Isotropic Elasticity (in units of $\mu a_0 \mathrm{cm}^{-1}$ )		
<b>3</b> 0°	-0.0199, +0.0215	-0.0328, +0.0328		
60°	-0.0173, +0.0114, -0.013, +0.0180	-0.0292, +0.0152, -0.0152, +0.0292		
90°	-0.0173, +0.0163, -0.0163, +0.0173	-0.0292, +0.0292, -0.0292, -0.0292, +0.0292		

are compared with the corresponding maximum values of  $F_x$  obtained using isotropic elasticity theory with  $\nu = 0.32$ . The separation of the slip planes has been arbitrarily chosen to be  $a_0$ , the lattice constant. The positive or negative signs assigned to these values of force indicate opposition or assistance respectively to the motion of the second dislocation when this is moved past the one at the origin in a direction along the minus x axis, and the values of  $F_x$  are listed in this order. The signs and the order are reversed when the second dislocation is moved along the positive x axis. It can be seen, therefore, that the maximum values of force which would oppose the motion for the isotropic case are symmetrical regardless of the direction from which the second dislocation approaches the first. However, this is not generally the case for elastic anisotropy. For  $30^{\circ}$  and  $60^{\circ}$  dipoles in nickel the maximum value of the force which opposes the motion is different, depending on the direction from which the second dislocation approaches the first. In the case of the edge dipole, because it is lying normal to an elastic symmetry plane, the forces again become symmetrical.

This asymmetry in the maximum values of  $F_x$  is common to all slip systems whose dipole equilibrium curves are asymmetric about the slip plane normal. Thus asymmetries exist for the  $\langle 110 \rangle \{111\}$  slip system in f.c.c. crystals but not for the (111){110} slip system in b.c.c. crystals. Consider an f.c.c. crystal containing isolated  $\pm \frac{1}{2}$  (110){111} dipoles which can be divided into two types: those where the dislocation with positive Burgers vector is (i) above or (ii) below the slip plane of the one with negative Burgers vector. If the distribution curve of the total number of dipoles of type (i) versus slip plane separation is significantly different from that for type (ii), then the crystal would be easier to deform in one direction than it would in the opposite direction. If, however, the two distribution curves are approximately equal, no such asymmetry in the mode of deformation would be observed. In this case the value of force for a dipole of a particular screw-edge orientation which would bear most relevance to the stress required to deform the crystal is the largest value of  $F_x$ . In the case of nickel, it can be seen that the largest values of  $F_x$  calculated on the basis of anisotropic elasticity are approximately equal to those estimated from isotropic elasticity, when due account is taken of the relative values of  $c_{44}$ and  $\mu$ .

### (d) Relative Importance of the Two Branches of the Equilibrium Curve

In the case of dipoles in f.c.c. metals, the asymmetry of the maximum forces, the asymmetry of the equilibrium curves about the normal to the slip plane, and the depths of the potential wells in which the dislocations are situated at equilibrium are all interrelated. The difference in depths of the potential wells for a dipole which has two possible positions of equilibrium is greatest at the screw-edge orientation where the second branch of the equilibrium curve first appears. This difference gradually decreases with increasing screw-edge orientation as the two branches of the curve become more symmetrical about the normal to the slip plane. At pure edge orientation where the equilibrium curves are symmetrical about the slip plane normal, the depths of the potential wells become equal.

The difference in the depths of the potential wells for the two possible positions of equilibrium for dipoles of a given screw-edge orientation will affect the relative numbers of dipoles in each position. Even though the component dislocations moving on their slip planes are equally likely to approach one another from either direction, they are more likely to be trapped in the deeper of the potential wells and this will give rise to an imbalance in the population of the two branches of the equilibrium curve. The magnitude of the imbalance is difficult to estimate directly after the formation of the dipoles but, if we consider the limiting case when finally a state of thermal equilibrium has been achieved, the imbalance can be calculated.

To illustrate the order of magnitude of the population imbalance under conditions of thermal equilibrium, consider the case of a 60° dipole in nickel. In this case the potential well for the equilibrium angle  $\phi = 45 \cdot 5^{\circ}$  is deeper than that for  $\phi = 123 \cdot 5^{\circ}$  and the difference between the two energies is  $0 \cdot 09$  eV per atomic spacing. Since the lengths of dislocation which are observed in the electron microscope are of the order of 1000 atomic spacings then, under conditions of thermal equilibrium at room temperature, for every one dipole in equilibrium at  $\phi = 45 \cdot 5^{\circ}$  there will be  $\sim e^{-3600}$  at  $\phi = 123 \cdot 5^{\circ}$ . Thus, under these conditions, a major portion of the equilibrium curve for  $\phi > 90^{\circ}$  would be almost non-operative. However, it should be emphasized that calculations of this kind probably provide a gross overestimate of the actual population imbalance, since in general there is a large energy barrier between the two potential wells and this, together with the difficulty of moving large segments of dislocation from one well to the other, will make the time for attainment of thermal equilibrium extremely long.

# (e) Measurement of Stacking Fault Energy

The  $90^{\circ}$  dipole which has been identified in nickel (dipole D, Fig. 9) has been used to deduce a value of stacking fault energy for that metal. Before discussing the significance of the value itself, it is interesting to investigate the several features peculiar to this dipole which all combined to make the determination possible.

The dislocations which constitute dipole D are very close together, and it was found during the image matching procedure that the images were extremely sensitive to small differences in separation of the slip planes. It is probable that this arises because the strain field from such a narrowly separated dipole varies very rapidly with separation, since for zero separation the strain field is zero. In the particular example shown here it was considered that the best match to the experimental images occurred at a slip plane spacing of 11*d* rather than 10*d* or 12*d*, where  $d = a_0/\sqrt{3}$ .

As mentioned earlier in Section IV, the close proximity of the dislocations and the fact that they were of edge character meant that dissociation of the dislocations into partials was important. Indeed, the separation of the partials ( $\sim 25$  Å) was of the same order as the separation of the slip planes and this, together with the change in equilibrium angle for dissociated dislocations, resulted in the sensitivity of the images to these factors also. It was therefore a combination of the dissociation, and the change in equilibrium angle which gave rise to the overall sensitivity of the images and enabled the dissociation width to be measured. The estimation of stacking fault energy followed from the equilibrium curve in Figure 15 adjusted for Y = 11d.

The value of 79 erg cm<sup>-2</sup> for the stacking fault energy of nickel deduced here is itself interesting since it is considerably lower than previous estimates which range from about 160 to 450 erg cm<sup>-2</sup> (see e.g. Seeger, Berner, and Wolf 1959; Howie and Swann 1961; Mader 1963; Dillamore and Smallman 1965; Häussermann and Wilkens 1966; Humble, Loretto, and Clarebrough 1967). It should be pointed out that there are some fundamental differences between the methods used in the previous estimates and that used here. For instance, all the previous estimates rely wholly or in part on linear isotropic elasticity theory. Some of these are based on the determination of values of stacking fault energy for different nickel-cobalt alloys followed by the extrapolation of these values to zero cobalt content to predict a value for pure nickel. A method where observations on pure nickel have been made is that due to Dillamore and Smallman (1965) which relies on an empirical relation between stacking fault energy and deformation textures. The estimate of Häussermann and Wilkens (1966), where observations on faulted dipoles in nickel have been made, is based on a consideration of the energy of the defect and includes the uncertain core energy term. The present estimate, however, is based on observations in pure nickel; the observations and calculations are made entirely within the framework of linear anisotropic elasticity, and only the forces between dislocations have been considered thus making core energy evaluations irrelevant.

There are, however, several other possible reasons why the present estimate of 79  $m erg\,cm^{-2}$  for the stacking fault energy of nickel is smaller than those estimated previously. First, dipole D may not be in equilibrium. Owing to the large number of variable parameters involved in computing micrographs corresponding to dipole D, only those cases which are in stable equilibrium have been considered. Thus the possibility of dipole D not being in equilibrium is difficult to assess. However, the evidence from the other dipoles which have been analysed would suggest that it is in equilibrium. Second, there may be impurities segregated to the stacking fault. The nickel used was zone refined with a quoted purity of 99.999%, but the relatively high dilational component of the strain field around a dipole compared with that of a single dislocation, for example, would make a dipole a favoured site for the accommodation of impurities. In this particular case, the dilation is compressive and the dipole would tend to attract substitutional impurities with smaller atomic volumes than nickel. Third, it could be that the low value of stacking fault energy derived here is not that appropriate to infinite strain free crystals, but that which applies to regions very close to dislocations. In a simple way, such anomalous values can be thought to occur when the separation of the partial dislocations is small and when the dislocation cores are spread out along the slip plane for distances of the order of the separation of the partials. In the neighbourhood of the dislocations, displacements due to the cores will affect the mis-positioning of atoms across the stacking fault. Although the final atomic displacements cannot be attributed wholly to the cores or to the stacking fault, if we do choose to express the energy of the displacement field conventionally by separating it into contributions from the elastic interaction of the dislocations and the stacking fault energy, then the effect of the spread-out cores will have a large influence on the value deduced for stacking fault energy. Indeed, it will lead to a value of stacking fault energy which is not constant but varies with the extent of the dissociation. This type of effect has been found theoretically by Englert and Bullough (1969), who, for a fixed value of stacking fault energy for copper, showed that Shockley partial dislocations were closer together than would have been expected from calculations using linear anisotropic elasticity. However, the experimental observations in our case for nickel show that the dissociation of the partials is wider than would have been expected for stacking fault energies in the range 160 to 450 erg cm<sup>-2</sup>.

If this last effect is in fact operative here, and if it can be applied to other cases, processes involving dissociation of dislocations in high stacking fault energy materials (e.g. aluminium) will now have to be seriously considered.

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