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Institut für Physik am Max-Planck-Institut für Metallforschung, Stuttgart

A Simple Method for the Analysis of Dislocation Loops by Means of the Inside–Outside Contrast on Transmission Electron Micrographs

By

H. FÖLL and M. WILKENS

In honour of Prof. Dr. Dr. h. c. P. GÖRLICH's 70th birthday

A new method is proposed for the analysis (vacancy or interstitial) of dislocation loops by means of the so-called inside–outside contrast. A simple and straightforward recipe is developed which is applicable in the same manner to loops of edge and of non-edge type irrespective of the loop orientation within the transmission foil; in particular, there is no reason for a subdivision of the loop orientations into "safe" and "unsafe" orientations as required when applying the method of Maher and Eyre.

Es wird eine neue Methode vorgeschlagen, um anhand des sogenannten "inside–outside" Kontrastes von Versetzungsringen deren Natur (Leerstellen- bzw. Zwischengitteratom-Typ) zu bestimmen. Es ergibt sich eine einfache und einheitliche Verfahrensvorschrift, die in gleicher Weise auf Versetzungsringe mit und ohne Scherkomponente angewandt werden kann, unabhängig von der Ringorientierung in der Durchstrahlungsprobe. Insbesondere bedingt diese Vorschrift keine Aufteilung der Ringorientierungen in „sichere" und „unsichere" Orientierungen, wie dies bei Anwendung der Methode von Maher und Eyre erforderlich ist.

1. Introduction

The analysis of the type of a dislocation loop, vacancy or interstitial, is a common task in transmission electron microscopy of crystalline specimens. If the loops are large enough in order to give rise to a well-resolved loop image or at least to a "double-arc" contrast figure the analysis is best performed by application of the so-called "inside–outside" contrast method (I–O-method) first introduced by Groves and Kelly [1] and used with minor variations by Mazey et al. [2] and Edmondson and Williamson [3]. This method is based on the fact that under kinematical diffraction conditions the image contrast of a dislocation loop lies either inside or outside the true loop position as projected onto the image plane, depending upon (i) the type of the loop, (ii) its orientation with respect to the electron beam and the operating diffraction vector $\boldsymbol{g}$, and (iii) the sign of the excitation error s.

The application of the I–O-method, for instance in the form as described by Hirsch et al. [4], is straightforward in the case of pure edge loops (Burgers vector $\boldsymbol{b}$ perpendicular to the loop plane). A rigid application of this procedure to non-edge loops (loops with $\boldsymbol{b}$ non-perpendicular to the loop plane, sometimes called "loops with shear components") may lead to incorrect results. This was first pointed out by Maher and Eyre [5]. In order to avoid these difficulties Maher and Eyre and, in a slightly different form, Kelly and Blake [6] have worked out somewhat more complicated variants of the I–O-method which should yield correct results also in the case of non-edge loops. An essential point of both these variants of the I–O-method is the use of the so-called FS/RH-rule ("finish-start/right-hand") [7, 4] which is applied in order to define the Burgers vector of the loop.

In the present paper we will show that the I–O-method can be cast into a form which is equally suitable for edge and non-edge loops and which is, to our understanding, more easily applied in practical cases than those of [5, 6]. The essential step in our approach is the replacement of the FS/RH-rule by another definition of the Burgers vector of a dislocation loop which was introduced by Kröner [8] and Kroupa [9].

2. Definitions

The crystallographic nature of a dislocation loop is fully characterized by the normal, $\boldsymbol{n}$, of the loop plane and the Burgers vector, $\boldsymbol{b}$, of the loop dislocation. In this section we introduce definitions of $\boldsymbol{n}$ and $\boldsymbol{b}$ which will be used later.

1. For a given vector, say $\boldsymbol{v}$, we distinguish between the "axis" and the "direction" of $\boldsymbol{v}$. The difference between these two notions is recognized easily, if one considers that two vectors of opposite directions, i.e., $\boldsymbol{v}$ and $-\boldsymbol{v}$, have a common axis.

2. We define as the loop normal $\boldsymbol{n}$ that unit vector perpendicular to the loop plane which points upwards in the electron microscope, i.e., towards the electron source.[1])

3. We may define a positive and a negative surface of the loop area, with the positive surface showing upwards, cf. Fig. 1. Thus $\boldsymbol{n}$ points from the negative to the positive surface. Now the positive direction of the Burgers vector $\boldsymbol{b}$ of the loop is defined as follows:

The loop is formed by shifting the negative surface against the positive surface by a displacement $\boldsymbol{b}$ (of course, at the same time matter has to be removed or added, depending on $(\boldsymbol{n} \cdot \boldsymbol{b})$). From this definition it follows[2]):

$$(\boldsymbol{n} \cdot \boldsymbol{b}) > 0 \Rightarrow \text{loop of vacancy type},$$
$$(\boldsymbol{n} \cdot \boldsymbol{b}) < 0 \Rightarrow \text{loop of interstitial type}.$$

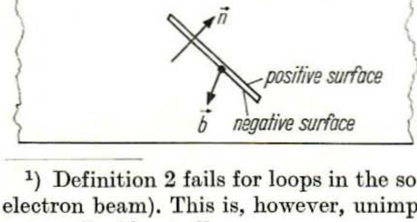

Fig. 1. Definition of the direction of $\boldsymbol{n}$ and $\boldsymbol{b}$ for a loop of interstitial type. The Burgers vector describes the shift of the negative against the positive surface

[1]) Definition 2 fails for loops in the so-called edge-on orientation ($\boldsymbol{n}$ perpendicular to the electron beam). This is, however, unimportant since in this orientation the I–O-method is not applicable at all.

[2]) For dislocation loops which are formed by clustering of point defects we have always $(\boldsymbol{n} \cdot \boldsymbol{b}) \neq 0$. Loops with $(\boldsymbol{n} \cdot \boldsymbol{b}) = 0$ (pure shear loops) may be produced, e.g., by pinching-off of a slip dislocation after passing around an impenetrable obstacle ("Orowan-mechanism"). In this case, which will not be considered further, the definition of the direction of $\boldsymbol{b}$ describes the direction of the shear over the loop area.

For the experimental determination of the axis of $\boldsymbol{n}$ we refer to the methods proposed in the literature [5, 10, 11]. Once the axis of $\boldsymbol{n}$ is determined the direction of $\boldsymbol{n}$ follows from the definition 2.

The *axis* of the Burgers vector $\boldsymbol{b}$ is usually derived from experiments resulting in contrast extinction ($\boldsymbol{g} \cdot \boldsymbol{b} = 0$). In the following we shall show that the correct *direction* of $\boldsymbol{b}$ can be deduced directly from the inside–outside contrast experiments.

3. The $(\boldsymbol{g} \cdot \boldsymbol{b}) \cdot s$-Rule

If a dislocation line is imaged with an excitation error $s \neq 0$ the contrast line is laterally shifted with respect to its geometrically projected position. This lateral shift depends only on the component of $\boldsymbol{b}$ parallel to the axis of $\boldsymbol{g}$ (see, e.g., [4]). Therefore, for a description of the I–O-method it is sufficient to consider a cross-section through the loop, where the section plane (= drawing plane) is parallel to the electron beam and to $\boldsymbol{g}$, irrespective of the components of $\boldsymbol{n}$ and $\boldsymbol{b}$ perpendicular to the drawing plane.

3.1 Pure edge loops

We consider a pure edge loop of interstitial type in two different orientations imaged with $s > 0$, cf. Fig. 2 and 3. The directions of $\boldsymbol{n}$ and $\boldsymbol{b}$ are drawn according to the definitions of Section 2. In Fig. 2a and 3a the dislocations cut by the drawing plane are characterized by the corresponding symbols of the edge components of their Burgers vector parallel to the drawing plane. In Fig. 2b and 3b these symbols are further reduced to the components of $\boldsymbol{b}$ parallel to the axis of $\boldsymbol{g}$. The latter figures also show the bending of the reflecting lattice planes as caused by the reduced Burgers vectors. By well-known arguments the maximum contrast is produced on that side of a dislocation on which the reflecting lattice planes are locally bent towards the Bragg diffraction position. Then it follows immediately that in Fig. 2 an inside contrast and in Fig. 3 an outside contrast is produced. In terms of $\boldsymbol{g}$, $\boldsymbol{b}$ and s we arrive for these particular cases of inter-

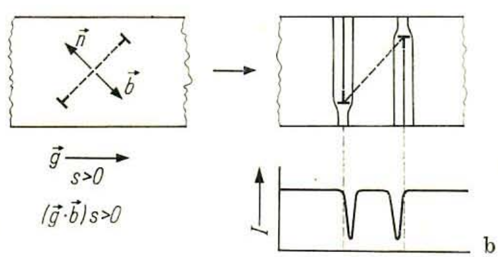

Fig. 2. Edge loop in an orientation which gives rise to an inside contrast. For details see text (I intensity)

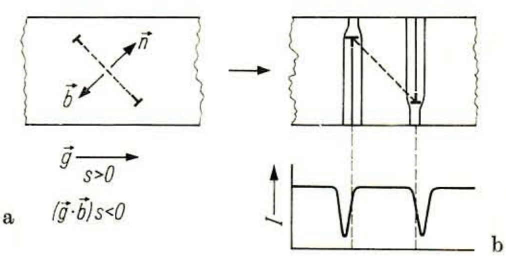

Fig. 3. Edge loop in an orientation which gives rise to an outside contrast. For details see text

stitial loops of pure edge type at the following $(\boldsymbol{g} \cdot \boldsymbol{b}) \cdot s$-rule:

$$(\boldsymbol{g} \cdot \boldsymbol{b}) \cdot s > 0 \Rightarrow \text{inside contrast} ,$$

$$(\boldsymbol{g} \cdot \boldsymbol{b}) \cdot s < 0 \Rightarrow \text{outside contrast} .$$

In this form the $(\boldsymbol{g} \cdot \boldsymbol{b}) \cdot s$-rule is a direct consequence of the definitions of Section 2.

Since the contrast changes from an "inside" position to an "outside" position (or vice versa) if the sign of $\boldsymbol{g}$, the sign of $\boldsymbol{b}$ (change from an interstitial loop to a vacancy loop or vice versa), or the sign of s is changed, we conclude that this $(\boldsymbol{g} \cdot \boldsymbol{b})$ s-rule is generally applicable at least for pure edge loops. In the next section we shall show that this is true also for loops in non-edge configurations.

3.2 Extension to non-edge loops

We start from a pure edge loop of interstitial type with the Burgers vector $\boldsymbol{b}$ inclined to the image plane (perpendicular to the electron beam) by an angle γ, cf. Fig. 4a. For $s > 0$, according to Fig. 2a, this loop produces an inside contrast. In a next step we change the loop into a non-edge configuration preserving the interstitial nature of the loop. This can be done, e.g., by extending the loop on its glide cylinder. In this way the number of point defects stored in the loop remains the same, while the axis of the loop normal $\boldsymbol{n}$ changes. We describe these changes of the axis of $\boldsymbol{n}$ by an angle α (Fig. 4b) or, if the loop is extended in the opposite direction, by an angle α' (Fig. 4c, d). Since the loop is extended only on its glide cylinder, α and α' are restricted to $0 \leqq \alpha, \alpha' < \pi/2$.

In the case of Fig. 4b it is immediately obvious that, irrespective of the value of α, an inside contrast is produced with $(\boldsymbol{g} \cdot \boldsymbol{b}) \cdot s > 0$ in all cases. The same is

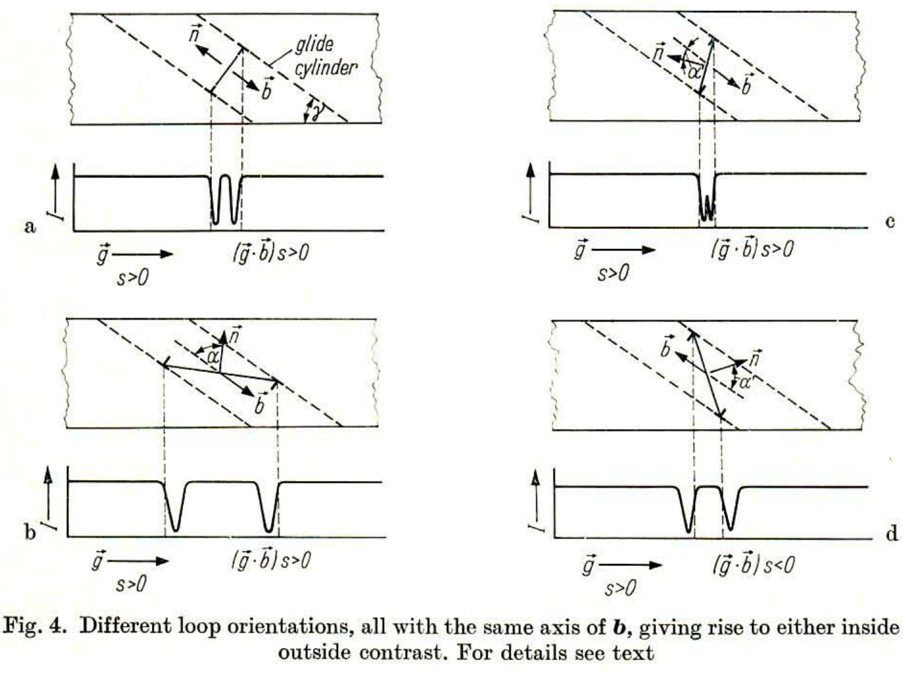

Fig. 4. Different loop orientations, all with the same axis of $\boldsymbol{b}$, giving rise to either inside or outside contrast. For details see text

true for the case of Fig. 4c where $\alpha' < \gamma$. For $\alpha' = \gamma$ (edge-on position of the loop) no inside–outside contrast is produced. If α' exceeds γ the two dislocation segments drawn in the figure interchange their mutual lateral position. Thus the contrast changes from "inside" for $\alpha' < \gamma$ to "outside" for $\alpha' > \gamma$. This change from "inside" to "outside" contrast is correctly described by the $(\boldsymbol{g} \cdot \boldsymbol{b}) \cdot s$-rule of Section 3.1: For α' exceeding γ the direction of $\boldsymbol{n}$ has to be reversed according to the definition 2 of Section 2; in order to preserve the interstitial nature of the loop the direction of $\boldsymbol{b}$ must be reversed too (cf. definition 3 of Section 2). Accordingly we have $(\boldsymbol{g} \cdot \boldsymbol{b}) \cdot s > 0$ for $\alpha' < \gamma$ and $(\boldsymbol{g} \cdot \boldsymbol{b}) \cdot s < 0$ for $\alpha' > \gamma$.

We conclude that for all non-edge configurations which can be derived from the pure edge configuration as schematically drawn in Fig. 4a (or Fig. 2a) the $(\boldsymbol{g} \cdot \boldsymbol{b}) \cdot s$-rule of Section 3.1 is applicable. It is easy to show that the same is true for all non-edge configurations of interstitial type which are derived, in the same manner as treated above, from the pure edge configuration as schematically drawn in Fig. 2b.

The same considerations apply to loops of vacancy type where, according to the reversal of the direction of $\boldsymbol{b}$, the inside contrast has to be changed into an outside contrast and vice versa. Therefore we conclude that for *all* loop orientations giving rise to an inside–outside contrast the $(\boldsymbol{g} \cdot \boldsymbol{b}) \cdot s$-rule of Section 3.1 together with the definitions of $\boldsymbol{n}$ and $\boldsymbol{b}$ as outlined in Section 2 gives a self-consistent indexing of the loop type, vacancy or interstitial.

4. Determination of the Loop Type[3])

Let us assume that the inclination of the loop in the foil (axis of the loop plane normal $\boldsymbol{n}$) has been determined with sufficient accuracy. Then the direction of $\boldsymbol{n}$ is given by definition 2 of Section 2. The axis of the Burgers vector $\boldsymbol{b}$ of the loop can be determined by contrast experiments using the $\boldsymbol{g} \cdot \boldsymbol{b} = 0$ criterion for contrast extinction. Then, by observing whether the contrast is "inside" or "outside" for a particular diffraction vector $\boldsymbol{g}$ and a given sign of the excitation error s, the direction of $\boldsymbol{b}$ can be determined using the $(\boldsymbol{g} \cdot \boldsymbol{b}) \cdot s$-rule of Section 3.1. Finally, once the direction of $\boldsymbol{b}$ is known, the loop type, vacancy or interstitial, follows from the definition 3 of Section 2.

5. Final Remarks

The inside–outside method described in the present paper is based on the definition introduced by Kröner [8] and Kroupa [9]. Accordingly the loop type, vacancy or interstitial, is unequivocally characterized by the angle ($<\pi/2$ or $>\pi/2$, respectively) between the normal $\boldsymbol{n}$ of the loop plane and the Burgers vector $\boldsymbol{b}$ of the loop. This sign convention, which has been shown to be very convenient also in the field of the black–white contrast analysis of small dislocation loops [12, 13] allows one to develop a very simple recipe for the interpretation of the inside–outside contrast of loops. For all cases giving rise to an inside–outside contrast of dislocation loops it is shown that this recipe can be applied in a straightforward manner and does not lead to complications for particular loop orientations, irrespective of whether the loops are in a pure edge or in a non-edge configuration.

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[3]) This recipe is valid for a loop viewed from above; i.e. we are discussing photographic negatives, or positives printed with emulsion side up.

In the inside–outside method proposed by Maher and Eyre [5] (cf. also Kelly and Blake [6]) the directions of $\boldsymbol{n}$ and $\boldsymbol{b}$ are defined according to the FS/RH-rule [4, 7]. This rule, originally established for the definition of the Burgers vector of a single dislocation, requires, as an intermediate step, the definition of the sense of circulation of the loop dislocation. Such an intermediate step, which may be a source of errors, is not required in the Kröner-Kroupa-definition.

In the method of Maher and Eyre the possible loop orientations are subdivided into "safe" and "unsafe" orientations depending on the mutual orientations of the axes of $\boldsymbol{n}$ and $\boldsymbol{b}$ with respect to the diffraction vector $\boldsymbol{g}$ and the direction of the electron beam. For "safe" orientations the loop behaves like a fictitious pure edge loop of the same Burgers vector and of the same type, vacancy or interstitial, whereas for loops in an "unsafe" orientation the fictitious pure edge loop has apparently changed its type. Obviously the method of Maher and Eyre, although in principle correct, is fairly complicated and has, indeed, led to some confusions in the literature [14 to 16].

As a matter of fact all variants of the inside–outside method become questionable either (i) for physical reasons if the loop is close to a pure shear loop ($(\boldsymbol{n} \cdot \boldsymbol{b}) \approx 0$) or (ii) for experimental reasons if the loop is close to an edge-on orientation ($\boldsymbol{n}$ nearly perpendicular to the electron beam). Then the result of the analysis of the inside–outside contrast depends sensitively on the accuracy by which the axes of $\boldsymbol{n}$ and $\boldsymbol{b}$ can be determined.

In a subsequent paper [17] the method proposed in the present paper will be applied to the analysis of dislocation loops (commonly called "swirls") in nearly perfect silicon crystals. Some preliminary results of this analysis were reported elsewhere [18].

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