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A METHOD FOR DETERMINING DISLOCATION BURGERS' VECTORS IN ICE

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ABSTRACT

It is shown that the usual method of Burgers' vector analysis in the Transmission Electron Microscope (TEM) using invisibility criteria is unlikely to succeed for ice. A method is outlined using a novel application of computer-simulated imaging which can be used to perform such an analysis.

BACKGROUND

The standard method for determining the Burgers' vector, b, of a perfect dislocation is to obtain two diffraction conditions for which the dislocation is invisible (that is g . b = 0 (1), where g is the diffraction vector). A third diffraction condition (not necessarily a 2-beam condition) must, of course, be obtained in order to record an image of the dislocation.

There are two problems associated with performing such a Burgers' vector analysis in ice. One is technical, the other is intrinsic to the material: -

1. In order to prevent sublimation of an ice specimen in the TEM, it must be held in a cold stage. Most available cold stages (especially if equipped with a cryotransfer device which prevents frost accumulation prior to insertion into the TEM) have only a single tilt axis capability. For such a single tilt axis holder, a Burgers' vector analysis of a dislocation is not possible except under unique circumstances; see appendix.

2. Irradiation of ice by a 100 KeV electron beam rapidly produces defects, notably cavities, which soon overwhelm the unirradiated defect structure (2-4). This defect accumulation is such that only three 2-beam conditions could be obtained and photographed together with the associated diffraction patterns in ice, by Falls et al. (4), before the original defect structure was no longer observable.

METHOD

The proposed technique attempts to minimize the electron irradiation of the ice and to make use of the 2-beam conditions which are available.

Several routines have been used to produce computer-simulated images of a dislocation (5-7) starting from a knowledge of the crystal structure and the Howie-Whelan equations for 2-beam dynamical diffraction (8). These routines are useful for providing an analysis of perfect dislocations present in an anisotropic medium (the g . b = 0 invisibility criterion is only strictly valid for an isotropic material) or for analysis of dislocation partials (5). Head (9) originally demonstrated that generally only three matches between computer-simulated and experimental images were necessary in order for a Burgers' vector to be identified. However, McConnell (10) subsequently demonstrated that in many circumstances two, or even one, matches were sufficient for a unique identification.

Thus, practically, a 2-beam condition can be set up in a region A away from the region of interest, I, but in the same grain. The image can also be coarsely focussed there. The region I, which contains the dislocations to be analyzed, can then be translated to the electron beam, finely focussed and photographed. The diffraction pattern must also be recorded from region I since, the value of the deviation parameter, w, must be obtained from the diffraction pattern for the computer-simulation. This procedure can then be repeated for a second or if necessary (and possible) a third 2-beam condition. Possible dislocation images can

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then be derived computationally and matching performed in the usual way (5) until the dislocation has has been uniquely identified.

In ice, the setting up of 2-beam conditions away from the region of interest in the same grain will be easy since grain sizes are relatively large, typically $\geq$ 1mm. The technique has the advantage that only two of three 2-beam conditions need to be accessed, and the 2-beam conditions accessed need to be such that $\underline{g} \cdot \underline{b} \neq 0$. The latter is the usual situation (see appendix) and this technique will work for all orientations of Burgers' vector except under the restrictive condition when the single tilt axis is parallel to the Burgers' vector. Note that even if a two-axis tilt capability was available, many 2-beam conditions normally have to be examined before two 2-beam conditions are found for which $\underline{g} \cdot \underline{b} = 0$, again meaning that a Burgers' vector analysis cannot be performed before radiation damage becomes a problem.

Whilst this proposed technique is specifically designed for the examination of dislocation in ice, it is obviously useful for other defects (using appropriate computer programs) and other materials which are sensitive to electron beam damage or which must be examined at low temperature.

REFERENCES

1. P.B. Hirsch, A. Howie, R.B. Nicholson, D.W. Pashley and M.J. Whelan, Electron Microscopy of Thin Crystals, Krieger, New York, 1977, P178
2. G. Honjo, N. Kitamura, K. Shimaoka and K. Mihama, J. Phys. Soc. Japan 11 (1956) 527
3. P.N.T. Urwin and J. Muguruma, Phys. Stat. Sol. (a) 14 (1972) 207
4. A.H. Falls, S.T. Wellinghoff, Y. Talmon and E.L. Thomas, J. Mat. Sci. 18 (1983) 2752
5. A.K. Head, P. Humble, L.M. Clarebrough, A.J. Morton and C.T. Forwood "Computed Electron Micrographs and Defect Identification", North Holland, Amsterdam, 1973
6. A.R. Thölén, Phil Mag 22 (1970) 175
7. K. Scheerschmidt and J. Heydenreich, Phys. Stat. Sol. (a) 42 (1977) 47
8. A. Howie and M.J. Whelan, Proc. Roy Soc. A 263 (1961) 217
9. A.K. Head, Aust. J. Phys. 22 (1969) 43
10. W.H. McConnell, Phil Mag. 33 (1976) 863

APPENDIX

Consider, three 2-beam diffraction conditions $\underline{g}_1$, $\underline{g}_2$, $\underline{g}_3$ which can be obtained by rotation about an axis defined by the vector $\underline{A}$ such that

$$\underline{g}_i \cdot \underline{A} = 0 \text{ for } i = 1,2,3 \tag{1}$$

Consider, first the situation when two of these reflections, $\underline{g}_1$ and $\underline{g}_2$, produce invisibility when a dislocation of Burgers' vector $\underline{b}$ is examined. That is,

$$\underline{g}_i \cdot \underline{b} = 0 \text{ for } i = 1,2 \tag{2}$$

Now equations (1) and (2) can only simultaneously be true if $|\underline{b}| = |\underline{A}|$ that is, if the Burgers' vector of the dislocation and the tilt axis are parallel (or anti-parallel). Thus, for any other (2-beam) diffraction vector $\underline{g}_3$ which is accessible using this tilt axis

$$\underline{g}_3 \cdot \underline{A} = \underline{g}_3 \cdot \underline{b} = 0$$

That is, using a single tilt axis, if two 2-beam diffraction conditions produce dislocation invisibility then all other 2-beam conditions which are accessible must also produce invisibility. Note, though that the dislocation can be imaged under multi-beam conditions.

Consider now the situation when a dislocation is visible for a 2-beam condition $\underline{g}_1$ but invisible for a 2-beam condition $\underline{g}_2$, that is

$$\underline{g}_1 \cdot \underline{b} \neq 0 \text{ and } \underline{g}_2 \cdot \underline{b} = 0$$

Then $|\underline{b}| \neq |\underline{A}|$ and in this case the only 2-beam condition which can give invisibility is $\underline{g}_2$.

Practically, this second situation is the usual case and a Burgers' vector analysis cannot be completed using a single tilt axis.