DOI: 10.1017/S1431927606067924
Microsc Microanal 12(Supp 2), 2006

Diffraction-Contrast Analysis of Dislocation Loops in BCC Alloys

J. Bentley

Metals & Ceramics Division, Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6064

Soon after the first observations of dislocations by transmission electron microscopy (TEM), dislocation loops in MgO were analyzed by Groves and Kelly and their character determined as interstitial; some months later, after Mike Whelan pointed out the extra 180° rotation between image and diffraction pattern, the loop character was changed to vacancy [1]. It can be argued that loop analysis has been in a continuing state of confusion ever since. In the first of five seminal papers on irradiation damage in molybdenum, Maher and Eyre described in detail the analysis of non-edge perfect dislocation loops, including the concept of "safe orientations" where contrast is in the same sense as that of a pure edge loop [2]. Needless to say, some confusion followed, even in print [3].

Dislocation loop analysis was an integral part of a TEM characterization study of Mo neutron-irradiated at elevated temperatures to fluences of 1 and 3 x 10^{20} fission neutrons.cm^{-2} [4]. With one exception, all clearly resolved loops were interstitial in character for materials irradiated at ≥475°C. Remarkably, in TZM alloy (Mo-0.5%Ti-0.1%Zr) irradiated at 750 and 850°C high concentrations of small (<20 nm diameter) vacancy loops with Burgers vector **b** = a/2<111> constituted the dominant component of the damage microstructure. The 100-kV diffraction-contrast analyses followed a procedure derived from Maher and Eyre [2] that accounted for foil normals near <011>. All dislocation loops with **b** = a/2<111> were imaged with diffracting vector **g** = ±200 at a beam direction (image-plane normal) **B** = [023]. Images recorded with **g** = 01$\bar{1}$ at **B** = [$\bar{1}$55], **g** = 1$\bar{2}$1 at **B** = [135] and **g** = 12$\bar{1}$ at **B** = [137] were used to identify loops with **b** = ±a/2[111] and ±a/2[$\bar{1}$11] from **g**.**b** = 0 invisible or residual contrast. Both +**g** and –**g** diffracting vectors were used (with s_g constant and positive) so that the invariance in strength of contrast and position of images satisfying **g**.**b** = 0 conditions could be confirmed. Diffracting vectors in safe orientations which gave **g**.**b** = ±2 conditions for the appropriate loops were used for inside/outside contrast analyses to determine the sense of **b** and thus the nature of the loop. For **b** = ±a/2[111], ±**g** = 310 at **B** = [$\bar{1}$36] and for **b** = ±a/2[$\bar{1}$11], ±**g** = $\bar{3}$10 at **B** = [136] were used. Weak-beam dark-field images were also recorded for the inside/outside **g**.**b** = ±2 analyses with $s_{<310>}$ = 2.4 to 2.9 x 10^{-2} $Å^{-1}$ (Ewald sphere intersecting the reciprocal lattice 1/4 to 1/2 of the distance from <620> to <930>). Outside contrast occurs for (**g**.**b**)s_g > 0, inside contrast for(**g**.**b**)s_g < 0. With **b** defined by the FS/RH perfect crystal convention, the positive dislocation direction as clockwise when viewed from above, and **n** = upward loop normal, for interstitial loops **n**.**b** > 0 and for vacancy loops **n**.**b** < 0. The presence of vacancy loops and their stability during post-irradiation annealing was rationalized on the basis of segregation of oversized Ti and Zr solutes to the dilated near-core regions, or through the formation of Ti-Zr-C complexes [4].

V-4%Cr-4%Ti has been of interest for the last decade as a candidate structural material for proposed fusion reactors. For a series of oxygen-doped alloys, annealing at 950°C resulted in the formation of large (diameter >1 μm), disk-shaped, TiC-rich Ti(C,O,N) precipitates ~2 nm thick on {001} [5]. The misfit normal to the habit gives rise to misfit dislocation loops. Diffraction-contrast analyses with ±**g** = <110> and <200> reveal that **b** ≈ a<001>, although the exact magnitude of **b** is uncertain since displacement fringes are commonly present with **g** = <110> (figure 1). Since the loops are of pure edge type, no consideration of safe orientations is needed and inside/outside contrast analyses with **g**.**b** = ±2 are achieved with **g** = <112>, as shown in figure 2. The loops have interstitial character, as expected from the bi-phase crystallography. The results are directly relevant to analysis of similar precipitates and secondary point-defect clusters in neutron-irradiated V-4%Cr-4%Ti. Even with the added complications of ferromagnetism, similar analyses of interstitial loops with **b** = a<100> were performed by Horton and Bentley on neutron-irradiated Fe and ion-irradiated Fe-10%Cr [6]. In summary, although fraught with potential pitfalls and seemingly countless opportunities for getting the wrong answer, traditional diffraction-contrast analysis still has much to offer for the characterization of dislocation structures, especially for point-defect clusters such as dislocation loops [7].

1. G.W. Groves and A. Kelly, *Philos. Mag*. **6** (1961) 1527; *Philos. Mag*. **7** (1962) 892.
2. D.M. Maher and B.L. Eyre, *Philos. Mag*. **23** (1971) 409.
3. C.M. van der Walt, *Philos. Mag*. **24** (1971) 999; D.M. Maher and B.L. Eyre, *Philos. Mag*. **26** (1972) 1233; C.M. van der Walt, *Philos. Mag*. **26** (1972) 1237.
4. J. Bentley, PhD thesis, University of Birmingham, England (1974).
5. J. Bentley and B.A. Pint, *Microsc. Microanal*. **7**(Suppl.2) (2001) 1246.
6. L.L. Horton et al., *J. Nucl. Mater*. **108/9** (1982) 222; *J. Nucl. Mater*. **103/4** (1981) 1085.
7. Research at the ORNL SHaRE User Facility (JB) supported by the Office of Basic Energy Sciences, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

1

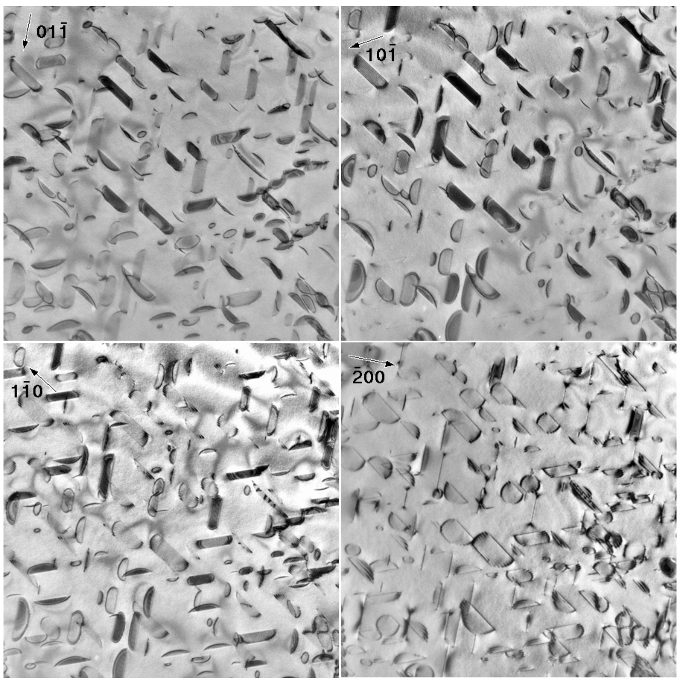
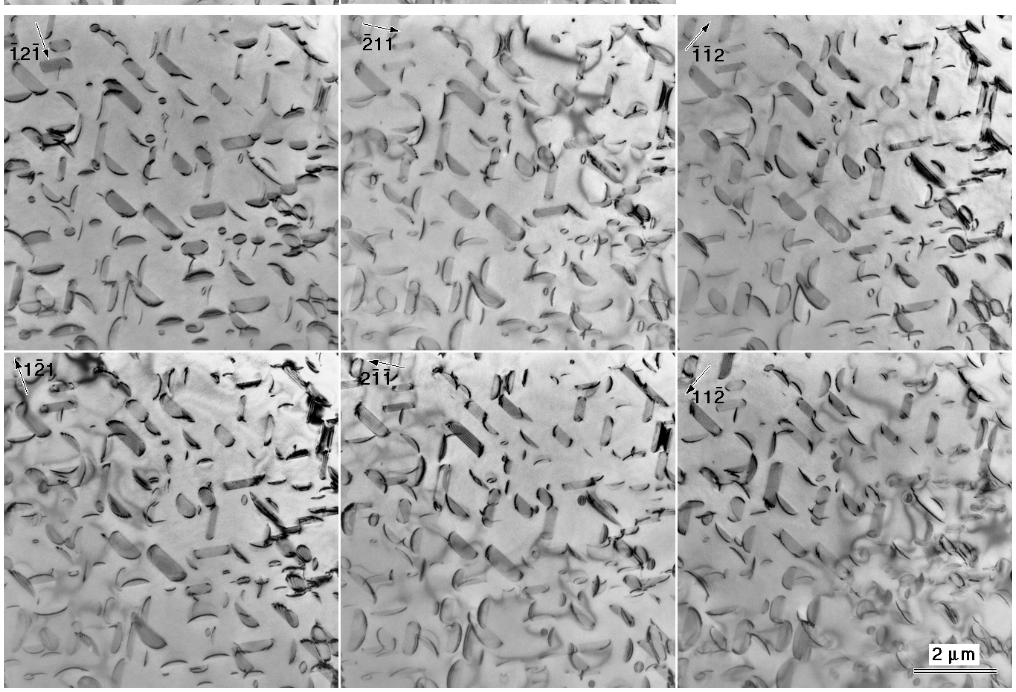


Fig. 1. Disk-shaped TiC-rich Ti(C,O,N) precipitates on {001} in oxygen-doped V-4%Cr-4%Ti annealed at 950°C exhibiting misfit dislocation loop contrast. Pairs of **g.b** = 0 conditions with **g** = $\pm 01\bar{1}$, $\pm 1\bar{1}0$, $\pm 10\bar{1}$ and 200 (±**g** recorded but not shown) at **B** = [122], [223], [212] and [023], respectively, reveal that **b** ≈ a<001>.

Fig. 2. Same area as Fig. 1 showing **g.b** ≈ ±2 inside/outside contrast analyses with **g** = $\pm\bar{1}2\bar{1}$, $\pm 21\bar{1}$ and $\pm\bar{1}\bar{1}2$ at **B** = [234] [146] and [243], respectively. The loops have interstitial character as expected from the bi-phase crystallography.

2



https://doi.org/10.1017/S1431927606067924 Published online by Cambridge University Press