Diffraction-Contrast Analysis of Dislocation Loops in BCC Alloys

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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 \( \mathbf{b} = a/2<111> \) 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 \( \mathbf{b} = a/2<111> \) were imaged with diffracting vector \( \mathbf{g} = ±200 \) at a beam direction (image-plane normal) \( \mathbf{B} = [023] \). Images recorded with \( \mathbf{g} = 011 \) at \( \mathbf{B} = [155] \), \( \mathbf{g} = 121 \) at \( \mathbf{B} = [135] \) and \( \mathbf{g} = 121 \) at \( \mathbf{B} = [137] \) were used to identify loops with \( \mathbf{b} = ±a/2[111] \) and \( ±a/2[111] \) from \( \mathbf{g}, \mathbf{b} = 0 \) invisible or residual contrast. Both \( +\mathbf{g} \) and \( -\mathbf{g} \) diffracting vectors were used (with \( s_\mathbf{g} \) constant and positive) so that the invariance in strength of contrast and position of images satisfying \( \mathbf{g}, \mathbf{b} = 0 \) conditions could be confirmed. Diffracting vectors in safe orientations which gave \( \mathbf{g}, \mathbf{b} = ±2 \) conditions for the appropriate loops were used for inside/outside contrast analyses to determine the sense of \( \mathbf{b} \) and thus the nature of the loop. For \( \mathbf{b} = ±a/2[111] \), \( ±\mathbf{g} = 310 \) at \( \mathbf{B} = [136] \) and for \( \mathbf{b} = ±a/2[111] \), \( ±\mathbf{g} = 310 \) at \( \mathbf{B} = [136] \) were used. Weak-beam dark-field images were also recorded for the inside/outside \( \mathbf{g}, \mathbf{b} = ±2 \) analyses with \( s_\mathbf{g} < 0 \) and for vacancy loops \( \mathbf{n}, \mathbf{b} > 0 \) and for vacancy loops \( \mathbf{n}, \mathbf{b} < 0 \). 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 \( ±\mathbf{g} = <110> \) and <200> reveal that \( \mathbf{b} \approx a<001> \), although the exact magnitude of \( \mathbf{b} \) is uncertain since displacement fringes are commonly present with \( \mathbf{g} = <110> \) (figure 1). Since the loops are of pure edge type, no consideration of safe orientations is needed and inside/outside contrast analyses with \( \mathbf{g}, \mathbf{b} = ±2 \) are achieved with \( \mathbf{g} = <112> \), 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 \( \mathbf{b} = a<100> \) 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].
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**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 \( g \cdot b = 0 \) conditions with \( g = \pm 01\overline{1}, \pm 110, \pm 10\overline{1} \) and 200 (+\( g \) recorded but not shown) at \( B = [122], [223], [212] \) and [023], respectively, reveal that \( b \approx a<001> \).

**Fig. 2.** Same area as Fig. 1 showing \( g \cdot b \approx \pm 2 \) inside/outside contrast analyses with \( g = \pm \overline{1}2\overline{1}, \pm 21\overline{1} \) and \( \pm \overline{1}12 \) at \( B = [234] [146] \) and [243], respectively. The loops have interstitial character as expected from the bi-phase crystallography.