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Three-dimensional scanning transmission electron microscopy of dislocation loops in tungsten

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ABSTRACT

Scanning transmission electron microscopy (STEM) imaging using diffraction contrast is a powerful technique to assess crystal defects. In this work it is used to assess the spatial distribution of radiation induced defect in tungsten. In effect, its irradiation leads to the formation of nanometric dislocation loops that under certain conditions may form intriguing 3-D rafts. In this study, we have irradiated thin tungsten samples *in situ* in a TEM with 1.2 MeV W ions to 0.017 dpa at room temperature (RT) and at 700 °C. Besides the Burgers vector analysis, the number density and size of the dislocation loops with their spatial arrangement were quantitatively characterized by stereo imaging in STEM mode. Most of the loops have a Burgers vector ½ a_0 ⟨111⟩, with some a_0 ⟨100⟩ at room temperature. Loops are located mainly in the simulated damage profile but there is also a significant portion in deeper regions of the sample, indicating that loops in W diffuse easily, even at RT. At 700 °C, loops form elongated rafts that contain dislocation segments having a Burgers vector ½ a_0 ⟨111⟩. The rafts are narrow and reside on {111} planes; they are elongated along ⟨110⟩ directions, which correspond, when combined to the rafts' Burgers vector, to the lines of edge dislocations. Compared to conventional TEM, 3-D analysis in STEM appears thus as a powerful technique for quantitative analyses of defects in tungsten, as it allows reducing the background diffraction contrast and reaching thicker areas of the electron transparent foil, here 0.5 µm of tungsten at 200 kV.

1. Introduction

Scanning transmission electron microscopy based on diffraction contrast is now a well-established method to evaluate crystal defects, such as dislocation lines, and is continuously improving (see e.g. (Yoshida et al., 2017)). Thermonuclear fusion is a promising source of energy for the future, but the subsequent large loads of heat and irradiation on the first wall of the reactor due to the resulting high-energy 14 MeV fusion neutrons would strongly impact the plasma facing materials microstructure and hence their physical properties. In the coming International Thermonuclear Experimental Reactor (ITER), plasma-facing material may be in tungsten (Rieth et al., 2011). Fusion neutrons impinging in tungsten generate gases by nuclear transmutation and cascades of atomic displacements, leaving defects made of interstitials and vacancies. These defects can be dislocation loops, voids and gas bubbles (Stiegler and Mansur, 1979). Many studies have shown that these defects degrade materials' mechanical properties, inducing hardening, embrittlement, loss of ductility and of fracture toughness (Baluc et al., 2000; Spatig et al., 2009). Dislocation loops are of 'the most common irradiation induced defects that can determine changes of mechanical properties by irradiation' (Seeger, 1959). It is thus critical to quantify these loops, in terms of their number density, size and Burgers vector, in order to allow the prediction of the evolution of the microstructure, using e.g. kinetic Monte Carlo, and, further, the prediction of the induced change in mechanical properties. Irradiation induced dislocation loops in tungsten appear to have a Burgers vector ½ a_0 ⟨111⟩ or a_0 ⟨100⟩, the former being the most commonly observed (Fikar and Schaublin, 2009; Mason et al., 2014; Sikka and Moteff, 1973). Molecular dynamics simulations have shown that the ½ a_0 ⟨111⟩ loops are energetically more favorable than the a_0 ⟨100⟩ ones (Fikar and Schaublin, 2009).

Spatial ordering such as raft formation of dislocation loops is one of the well-observed phenomena in irradiated bcc metals, such as Mo (Brimhall and Mastel, 1970; Sikka and Moteff, 1974; Yamakawa and

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Shimomura, 1999) and Fe (Zinkle and Singh, 2006) it occurs in irradiated tungsten as well (Sikka and Moteff, 1973; Van Renterghem and Uytdenhouwen, 2016). However, the information on the observed rafts in W that would allow understanding their formation is lacking. Besides, there is still a lack of knowledge on its dependence on irradiation temperature, despite some published results (Ferroni et al., 2015; Yi et al., 2015; Yi et al., 2016). Transmission electron microscopy of metals irradiated in situ with high energy electrons or energetic ions showed that rafts form through the diffusion of the irradiation induced loops and their mutual interaction. Using Langevin dynamics simulation, Dudarev et al. showed that loops with collinear burgers vector have a large elastic interaction and so a tendency towards local ordering and eventually raft formation. This does not exist for non-collinear Burgers vectors. Moreover, rafts of dislocation loops appear in the aforementioned simulations to be aligned in preferential crystallographic directions (Dudarev et al., 2010).

In this work we characterize the dislocation loops structure in pure tungsten induced by low dose self-ion irradiations made *in situ* in a transmission electron microscope (TEM). The impact of temperature on the tungsten's microstructure is assessed by comparing room temperature (RT) and 700 °C irradiations using TEM, which remains the tool of choice in the evaluation of radiation induced damage (Linsmeier et al., 2013). Conventional tomography in TEM can be applied for the 3-D reconstruction of dislocation lines (Barnard et al., 2006; Liu and Robertson, 2011). Recently, the use of scanning TEM (STEM) mode, and in particular annular dark-field STEM, has been extended to dislocation imaging and analysis (Perovic et al., 1993; Phillips et al., 2011a; Phillips et al., 2011b). In addition, the advantages that STEM imaging offers – for the potential reduction of the diffraction contrast background and for the lack of optical aberrations (chromatic mainly) after the specimen that allows the investigation of thicker areas – made this technique the method of choice for tomographic and stereoscopic imaging of dislocations (Agudo Jácome et al., 2012; Humphreys, 1981; Maher and Joy, 1976; Norfleet et al., 2008), and recently a method based on such imaging was developed which allows for a tilt-less 3-D reconstruction of dislocation lines (Oveisi et al., 2017; Oveisi et al., 2018).

In the present work, besides quantifying the density, size and Burgers vector of the dislocation loops in the irradiated W using conventional TEM based on diffraction contrast, a methodology, combining stereo-imaging in STEM mode and image analysis, is developed and applied to assess the 3-D distribution of the loops, as well as the crystallographic orientation of the loops rafts. The derived spatial distribution of loops is compared to the defect distribution according to Monte Carlo simulations obtained with SRIM code (Ziegler et al., 2010). The Burgers vector of the dislocation loops in rafts is explored in relation with the crystallographic orientation of the host raft. Results are presented here and the formation of dislocation loops raft is discussed, mainly in terms of the mobility of the observed loops.

2. Experimental methods

2.1. Sample preparation

Pure tungsten sample was provided by FZK Karlsruhe in the frame of EFDA (European Fusion Development Agreement, now EUROfusion). It was produced by sintering tungsten nanopowder at 1273 K. The resulting sample was then heat treated at 1873 K during 1 h under vacuum (2.10^{-8} mbar). Foils were cut into $\sim 2 \times 2$ mm^2 squares (with a diagonal fitting the typical 3 mm TEM disk). Samples were then mechanical polished down to a thickness of about 100 µm. For the in situ TEM irradiations, the W square samples were thinned down to electron transparency with a twin-jet electrochemical polisher (Tenupol 5 of Struers®) using a 0.5 vol.% NaOH aqueous solution at room temperature and 20 V.

2.2. Ion irradiation of tungsten foils

Samples were irradiated in JANNuS Orsay facility at CSNSM, University Paris Sud, France, with ARAMIS ion accelerator coupled to a TEM FEI Tecnai G2 operated at 200 kV and equipped with a LaB_6 source (Serruys et al., 2009). The electron transparent tungsten foils were irradiated with 1.2 MeV W^+ ions at an angle of 45° relative to the sample surface, a fluence of $1.8.10^{12}$ at cm^{-2} corresponding to 0.017 displacements per atom, at room temperature and 700 °C. The thickness of the observed area was determined by electron energy loss spectroscopy in the TEM.

2.3. STEM stereo imaging

For the detailed analysis of the loops a FEI Tecnai Osiris at CIME EPFL Lausanne operated at 200 kV and equipped with a field emission gun was used. Images were acquired in scanning TEM (STEM) mode with a central bright-field detector of 10 mrad collection range. The incident beam convergence semi-angle was set to 6.2 mrad using a nominal 50 µm probe-forming aperture in the nanoprobe mode. Pairs of images were acquired to form so-called stereo pairs by mechanically tilting the specimen in the microscope. In the stereo-imaging method, the relative depth, z, of an object can be calculated by measuring the shift of its image position from one image to the other composing the stereo pair, with the knowledge of the angle, φ, between them:

$$z = \frac{x_1 - x_2}{2\sin\left(\frac{\phi}{2}\right)}. \tag{1}$$

The typical angle, φ, applied here is between 5° to 15°. It should be noted that stereo-pairs of dislocation loops should be acquired using the same diffraction vector **g** and the same diffraction condition in order to avoid a change in contrast from one image to the other and to avoid a change in the invisibility of the loops (due to **g**·**b** = 0). For the acquisition of the stereo images and **g**.**b** analysis, the specimen has been mechanically tilted to excite a near two-beam condition with a slightly positive deviation from the exact Bragg condition. As an example, to illustrate the great efficiency of the technique, a typical pair of stereographic micrographs is shown in Fig. 1.

Dislocation loops can overlap each other from one image of the stereo pair to the other. To overcome this obstacle, a series of images (about 15 image with 1.5° increment) was acquired within the angle applied to form the two images composing the stereo pair. The images of the tilt series were aligned using the software ImageJ. The displacement of the loops was carefully identified in the tilt-series, allowing the identification of overlapping loops. The loops that were unambiguously not overlapping in the two extreme images within the tilt series were manually marked and their positions (x_1, y_1) and (x_2, y_2) in respectively the first and the last image of the stereo pair were recorded. In case one loop is masked by other loops in one or both images of the stereo pair, we did the analysis on the images of smaller stereo tilt angles. With the knowledge of the parallax shifts of dislocation loops (x, y) in the two images composing the stereo pair (images of largest stereo tilts for which the loop do not overlap) and the angle between them the relative depth of dislocation loops is derived from the equation 1, yielding a full 3-D assessment of the loops spatial distribution. Using the crystallographic coordinate system of the specimen from diffraction pattern the crystallographic orientation of dislocation loops and glide planes can be derived. Note that the 3-D assessments of loop's positions are done for the stereo images of only one **g** vector each time.

The precision of the reconstructed depth depends mainly on the tilt angle between the two extreme views and the precision of the lateral loop's position. The latter in turn depends on the image pixel size but also on the imaging conditions which will affect the smallest measurable displacement d_s between two images as well as the absolute precision in the position of the dislocation, noting that the width of the

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