Acta Materialia 147 (2018) 277–283

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Detailed transmission electron microscopy study on the mechanism of dislocation loop rafting in tungsten

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ARTICLE INFO

Article history:
Received 16 August 2017
Received in revised form
19 December 2017
Accepted 6 January 2018
Available online 3 February 2018

Keywords:
Loop rafting
Transmission electron microscopy
Burgers vector
Heavy ion irradiation
Nanocrystalline

ABSTRACT

Dislocation loop rafting and dislocation decoration have been previously observed in neutron and heavy ion irradiated materials. Understanding the fundamental aspects of these phenomena assist in evaluating irradiation damage of nuclear materials. Multiple different mechanisms have been suggested to explain loop rafting. Here, we performed a detailed transmission electron microscopy study on dislocation loop rafts in heavy ion irradiated tungsten. Different imaging conditions showed that the rafts are of <111> Burgers vector type and specifically the same <111> Burgers vector variant (<$1\bar{1}1$>) in the particular grain analyzed. Some rafts were associated with dislocation lines while some form as a result of alignment of dislocation loops. They were shown to form at both room temperature (RT) and high temperature with stronger rafts forming at RT. These observations confirm the mechanism previously suggested by Wen et al. which explains raft formation due to loop glide, cluster-cluster and grown-in dislocation-cluster interaction with subsequent Burgers vector rotation. Similar irradiation studies on nanocrystalline tungsten showed that these materials are more resistant to raft formation at RT irradiations.

1. Introduction

The change in the microstructure of irradiated nuclear materials under irradiation at the mesoscale has consequences to their performance in nuclear reactors [1]. Irradiation damage has been shown to alter the microstructure [2,3] and the mechanical properties [1,4–6] of the irradiated materials. Such consequences should be studied through controlled laboratory testing to fundamentally understand the damage mechanism, and evaluate the overall performance of the materials by examining property (eg. mechanical properties) changes to prevent failure during real reactor operation [1,7]. Neutron irradiation damage occurs in both fission and fusion (output of the fusion reaction) type materials [8]. While neutron irradiation is not the only damaging aspect of irradiation in the reactors, and synergistic effects can occur between different species, examining the effect of neutron irradiation independently assists in understanding the fundamental aspects and kinetic processes of neutron irradiation damage. Irradiation with particles (e.g. neutrons or high energy protons), however, can make the material radioactive which creates significant challenges in the characterization and the evaluation processes [9]. Moreover, high dose rate neutron irradiation facilities are limited [1]. Simulation of neutron irradiation can also be performed via the use of heavy ions [10–14]. The main drawbacks (differences) in simulating neutron damage with heavy ions are the higher dose rates and the shallower depth of damage from the surface [15].

Neutron and heavy ion irradiations on materials can lead to several microstructural changes in BCC materials (dislocation loops with both vacancy and interstitial type, voids, irradiation induced segregation, precipitation, etc.) [1,16]. One of the most interesting phenomena which was shown to occur during neutron and heavy ion irradiation is the orientation of loops into parallel lines called dislocation rafting [15,17–25]. Understanding the irradiation hardening and embrittlement in materials suffering from loop rafting is necessary to evaluate the performance for these materials. Quantification of irradiation damage is also challenging since loops are not uniformly distributed but rather they are organized in parallel linear lines and the loops are often too close to each other to distinguish individual ones. Discovering the formation mechanism of the rafting can guide the quantification process by assisting in understanding the defect accumulation behavior and any correlation with defect concentration. Several mechanisms are suggested to explain the formation of these rafts based on different

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https://doi.org/10.1016/j.actamat.2018.01.003
1359-6454/Published by Elsevier Ltd on behalf of Acta Materialia Inc.

experimental observations [15,17,18,20,21] which are illustrated and compared to our results later in this paper.

Here, we examine raft formation in tungsten irradiated with different displacement per atom (dpa) rates at room temperature (RT) and high temperature (1050 K) to different doses. Tungsten is a nuclear material with particular application in fusion reactors as a plasma facing component (PFC) [26]. Tungsten, in a fusion reactor, will be exposed to a neutron damage of hundreds of dpa for an anticipated 3–5 year lifetime [27] which can result in raft formation.

Detailed transmission electron microscopy investigations are performed on the raft microstructures to determine the exact Burgers vector of the dislocations in the rafts and examine orientation effects. Raft formation is also examined in commercial and nanocrystalline tungsten at RT and 1050 K and the different suggested mechanisms are compared in our conclusions. Our findings support the mechanism suggested by Wen et al. [20] described in detail later in the paper.

2. Experimental

Two tungsten grades were irradiated with heavy ions in this work. The first grade (identified as coarse grained tungsten CGW throughout this manuscript) is a commercial tungsten grade provided by ESPI, USA. This grade has an average grain size of over 1 μm. The second grade (identified as nanocrystalline tungsten NCW) is a nanocrystalline and ultrafine tungsten grade (both ultrafine and nano-sized grains co-exist) which is formed via orthogonal machining process [28]. The nanocrystalline grains are defined as those that have the shortest distance between two boundaries to be less than 100 nm and the ultrafine grains are grains with the shortest distance of 100–500 nm as defined by Wei et al. [29,30]. Both grades are high tungsten purity of 99.95 and 99.99 for the nanocrystalline and the commercial grades, respectively.

TEM samples were obtained from both grades prior to irradiation. The TEM foils were prepared via electropolishing (with 0.5% NaOH solution) [2] to specifically provide surfaces that are not affected by irradiation (e.g. irradiation from focused ion beam).

Irradiation was performed ex-situ in the Ion Beam Materials Laboratory (IBML) at Los Alamos National Laboratory (LANL) using the Tandem accelerator with 3 MeV Cu^{+} ions at nominal incidence with two different dpa rates (0.0167 and 0.000167 dpa/s) and two different temperatures (RT and 1050 K). The dpa and the dpa rates were determined using the Stopping Range of Ions in Matter (SRIM) Monte Carlo computer code (version 2013) [31]. The damage event was 0.76 vacancy/Ion/Å in the 100 nm sample thickness of the foils.

Characterization of the samples was performed via TEM using an FEI-Tecnai-G2-F30 transmission electron microscope with electron beam energies of 300 keV in the Electron Microscopy Laboratory (EML) at LANL. Quantification was performed using 2-beam conditions with g vectors of $(1\bar{1}0)$ and (200).

3. Results and discussion

3.1. Rafting mechanisms (literature)

Dislocation loop rafting (needle-like aggregates of dislocation loops) was previously observed in tungsten and other BCC materials as mentioned above. It was also observed in copper [17]. Zhang et al. [25] irradiated tungsten with dual beam (MeV Fe^{+3} and MeV He^{+}) and observed loop rafting at different temperatures (ranging from 300 °C to 1000 °C) which was suggested to be correlated with special crystal orientations. Brimhall and Mastel [18] observed dislocation loop rafting in neutron irradiated molybdenum. The rafts were shown to occur at temperatures over 400 °C where dislocation loop glide and climb can occur with ease. Based on the contrast of TEM images, the authors in that work concluded that dislocation loops in the raft should have the same Burgers vector.

Sikka and Moteff [21] observed dislocation loop rafting in fast neutron irradiated tungsten samples irradiated at 430 °C and 580 °C. The Burgers vector of the dislocation loops in the rafts, using the g·b analysis of [110] zone axis, was concluded to be of <111> type. The rafts were demonstrated to be associated with linear dislocations. Voids were also present in the samples. The authors suggested the preferential drift of free interstitials to dislocations and glide and self-climb of loops (as the mechanism suggested by Brimhall and Mastel) to be the mechanism for raft formation. The arguments by Bullough et al. [32] which suggests that preferential drift of self-interstitials to dislocations produces a supersaturation of vacancies for void formation, and the association of rafts with dislocations in their TEM images were used to support their conclusions. Eldrup et al. [19] observed stronger rafting in BCC Fe than FCC Cu in neutron irradiated specimens at 70 °C. The rafts were suggested to be formed by dislocation loop alignment and were shown to occur clearly at 0.72 dpa. The segregation and coalescence of clusters start to occur at doses higher than 0.01 dpa. In another paper by Zinkle and Singh [24] on neutron irradiated iron at a similar temperature, it was demonstrated that the formation of rafts occurs beyond 0.3 dpa and the Burgers vector of the loops associated with the rafts were mainly of <111> type. In addition, a higher number of rafts was observed in thick foils than thin foils in that study. Yi et al. [23] observed rafting after tungsten irradiation with 150 keV W^{+} at doses over 0.4 dpa and temperatures higher than 500 °C in grains close to [001] orientation. In an in-situ TEM/irradiation (2 MeV W^{+}) study of the same author [22], rafts were observed in tungsten at temperatures over 500 °C and a dose of 1.2 dpa for grains with [001] orientation. In other orientations, the authors mentioned that rafts occur at temperatures over 300 °C. They attributed the effect of orientation to surface traction effect (glide of different variants of <111> Burgers vector loops to the surface).

Wen et al. [20], through Kinetic Monte Carlo (KMC) simulations, suggested that raft formation could be achieved through loop glide and rotation of the Burgers vectors of self-interstitial clusters which interact with dislocation microstructure without the need for self-climb (as suggested by Brimhall et al. [18]). The process of rafting was shown to occur more readily at RT.

Dudarev et al. [15] described the raft formation or the “ordered nano-scale dislocation loops” to be a result of Brownian motion of loops and their elastic interaction through angular dependent elastic forces (loops attracting or repelling each other) when high density of defects exists. Barnes et al. [17] suggested the formation mechanism of rafting to be a result of loop-loop interactions where loops adjust their positions and orientation to form a low energy configuration. Therefore, several mechanisms are suggested and raft formation was claimed to be dependent on the grain orientation and the sample thickness.

It is then concluded that in most suggested mechanisms above, rafts were often associated with high temperature irradiations. Rafts were also thought to be facilitated at conditions where voids are present. Experimental results are needed to verify one or some of the above mechanisms. For example, the effect of irradiation temperature on raft formation and whether strong rafting can be observed at RT can eliminate the validity of some mechanisms. Moreover, some mechanisms suggested that loops in rafts are of the same Burgers vector type (eg. <111> vs <100>). However, experimental determination whether the Burgers vector in the rafts are of the same variant (eg. one of the four possibilities of <111> type Burgers vector) can provide sufficient evidence regarding the