Acta Materialia 149 (2018) 206–219

Contents lists available at ScienceDirect

Acta Materialia

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

Full length article

Loop and void damage during heavy ion irradiation on nanocrystalline and coarse grained tungsten: Microstructure, effect of dpa rate, temperature, and grain size

O. El-Atwani [a, *], E. Esquivel [a], M. Efe [b], E. Aydogan [a], Y.Q. Wang [a, c], E. Martinez [a], S.A. Maloy [a]

[a] Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM, USA
[b] Metallurgical and Materials Engineering, Middle East Technical University, Ankara, Turkey
[c] Center for Integrated Nanotechnology, Los Alamos National Laboratory, Los Alamos, NM, USA

ARTICLE INFO

Article history:
Received 8 September 2017
Received in revised form
31 January 2018
Accepted 13 February 2018
Available online 20 February 2018

Keywords:
Heavy ion irradiation
Grain size
Electron microscopy
Voids
Dislocation loops
Burgers vector

ABSTRACT

Displacement damage, through heavy ion irradiation was studied on two tungsten grades (coarse grained tungsten (CGW) and nanocrystalline and ultrafine grained tungsten (NCW)) using different displacement per atom rates and different irradiation temperatures (RT and 1050 K). Percentage of <111> and <100> type loops at the irradiation conditions was determined. Irradiation damage in the microstructure was quantified using average loop areas and densities (method A) and loop areal fraction in the grain matrices under 2-beam diffraction conditions (method B). Average values of <111> and <100> loops were calculated from method A. Loop coalescence was shown to occur for CGW at 0.25 dpa. Using both methods of quantifying microstructural damage, no effect of dpa rate was observed and damage in CGW was shown to be the same at RT and 1050 K. Swelling from voids observed at 1050 K was quantified. The loop damage in NCW was compared to CGW at the same diffraction and imaging conditions. NCW was shown to possess enhanced irradiation resistance at RT regarding loop damage and higher swelling resistance at 1050 K compared to CGW. For irradiation at 1050 K, the NCW was shown to have similar defect densities to the CGW which is attributed to higher surface effects in the CGW, vacancy loop growth to voids and a better sink efficiency in the CGW deduced from the vacancy distribution profiles from Kinetic Monte Carlo simulations. Loop density and swelling was shown to have similar values in grains sizes that range from 80 to 600 nm. No loop or void denuded zones occurred at any of the irradiation conditions. This work has a collection of experiments and conclusions that are of vital importance to materials and nuclear energy communities.

Published by Elsevier Ltd on behalf of Acta Materialia Inc.

1. Introduction

Tungsten is considered to be the primary choice as a plasma material interface (PMI) in fusion reactors [1]. For these applications, tungsten will be exposed to high doses of low energy helium ($10–10^4$ eV) and 14.1 MeV neutron irradiation of approximately hundreds of dpa for an anticipated 3–5 year lifetime [2,3] which leads to irradiation damage denoted by the formation of interstitial loops, dislocations, vacancy clusters, helium bubbles [4,5], and cavities [6–10]. The irradiation damage alters the thermal [11,12] and mechanical properties [13,14] of the material and changes its microstructure [15–17]. The degradation of the mechanical properties can lead to serious issues such as failure of the components [13]. Such degradation is manifested by the embrittlement and hardening of the material, creep, and changes in the ductile to brittle transition temperature [18,19]. These challenges prompted the investigation of other alternative materials such as tungsten alloys and ultrafine (UF) and nanocrystalline (NC) tungsten (refined grained tungsten) [20,21].

NC materials are considered to be highly radiation tolerant due to their high density of grain boundaries which, in turn, act as defect (i.e. irradiation induced defects) [10,22–24] and particle (e.g. the helium ash from the deuterium-tritium reaction in fusion reactors) [25] sinks and enhance defect annihilation [26]. This is

* Corresponding author.
E-mail address: oelatwan25@gmail.com (O. El-Atwani).

https://doi.org/10.1016/j.actamat.2018.02.035
1359-6454/Published by Elsevier Ltd on behalf of Acta Materialia Inc.

believed to lead to a high dose threshold for the aforementioned undesired changes in the material matrix. The enhanced irradiation tolerance performance of nanocrystalline materials was illustrated on several BCC and FCC metallic materials as well as ceramic materials [6,27–35].

Recently, studies regarding helium irradiation on NC tungsten and other BCC materials demonstrated a logarithmic trend of bubble density and swelling as a function of grain size over a temperature threshold [32,33,36]. Heavy ion irradiation on tungsten at RT demonstrated a surprising trend of defect density decrease as the grain size increased in the ultrafine (100–500 nm) regime. This was demonstrated to be a result of defect coalescence in the large grains [37]. Different NC materials, however, showed a variation in their response to irradiation damage. Their response depends on the irradiation conditions, synergistic effects, and their sink efficiency (grain boundary character) type which is usually experimentally characterized by the width of denuded zones (loop-free zones near the grain boundaries) [38].

Heavy ion irradiation is used to simulate neutron irradiation in materials [39–42]. However, the dpa rate during ion irradiations is orders of magnitude higher than the dpa rate during neutron irradiation [43] and therefore, heavy ion irradiations have to be performed at higher temperatures to introduce similar defect density and size distributions [44]. Recently, Dunn et al. [44] performed spatially resolved stochastic cluster dynamics (SRSCD) to study the temperature shift required during heavy ion irradiation to simulate neutron irradiation damage. Their results showed that a temperature shift of 100–200 °C is needed to correlate heavy ion irradiation with neutron irradiation results on BCC Fe.

Several studies have used different heavy ions to investigate the early stage neutron damage on tungsten [8–10,45–48]. Defect configurations, geometries and distributions were studied by Yi et al. [8,9]. Effect of the heavy ion type, the damage stages and grain size effect in the ultrafine regime were examined by El-Atwani et al. [10]. Synergistic effect with He ions were also studied by Zhang et al. [48]. However, there are still several outstanding questions that need to be answered regarding irradiation damage in tungsten. The first outstanding question is the Burgers vector type of the loops formed during heavy ion irradiation. Sand et al. [49] analyzed MD simulations for 150 keV collision cascades on W. In D-D potential simulations, small loops (with 35–80 self-interstitials, SIAs) were shown to be 50% <111> Burgers vector and fifty percent <100> Burgers vector, while in A-T potential simulations only one <100> type loop was predicted. No vacancy loops were found using D-D potential while one vacancy loop formed from the cascade collapse with the A-T potential. Setyawan et al. [50], via MD simulations, predicted the formation of rare <100>{110}SIA loops at 1025 K (similar to the high temperature in this study) but not at 300 K (similar to the room temperature in this study). However, vacancy loops of <100>{100} or cavities were shown to occur at both 300 K and 1025 K. Such loops were shown to form directly in the higher energy cascade. Others [51–53] used 30–60 keV Au^{+} to irradiated tungsten foils at RT and have found vacancy type <111> {110} loops and few pure edge <110> loops which are explained to form from cascade vacancy core collapse. Yi et al. [9] demonstrated that all loops at 1075 K are of <111> type (using 150 keV self-ion irradiation). At higher energies (2 MeV), Yi et al. [8] found the percentage of <100> loops to be approximately 10 at 1023 K. The authors argue that these <100> loops are not stable at high temperatures. Yi et al. used weak beam dark field imaging and loops of less than 4 nm were disregarded. The second outstanding question is the effect of grain size (direct comparison between nanocrystalline or ultrafine grained tungsten with coarse grained tungsten), dpa rata and temperature on the irradiation damage and the methods in which a total irradiation damage can be quantified. The third outstanding question is the mechanism of loop rafting (a phenomenon which is observed in heavy ion and neutron irradiated BCC metals) [9,43,48,54,55]. Lastly, the fourth outstanding question is void formation under heavy ion or neutron irradiation and the stability of these voids as a function of dpa. Evans et al. [56] who showed a decrease in void sizes on neutron irradiated molybdenum as a function of dpa. The shrinkage of the voids was shown to occur due to the change in the rate of vacancy jumps to voids. This can occur when vacancy loops start to grow at the sites of the voids. The voids will add another interstitial biased microstructure (in addition to dislocation networks) thus absorbing interstitials and giving rise to net absorption of vacancies to the vacancy loops. Swelling will probably decrease as a function of dpa.

This work attempts to answer these outstanding questions by performing heavy ion (Cu^{+}) irradiation on W thin foils. We first determine the Burgers vector of the loops via bright field transmission electron microscopy (TEM) images at different two beam conditions on all visible loops. Second, we perform two different methods to quantify irradiation damage (taking into account both loop density and sizes) and compare the damage for two dpa rates (using two dpa rates of 100 magnitude difference), and two different extreme temperatures (RT and 1050 K). The performance of nanocrystalline and ultrafine tungsten is also compared with coarse grained tungsten at low dpa rates. Kinetic Monte Carlo (KMC) simulations are performed to plot the steady state vacancy profile across an interface and discuss the sink efficiency vs. grain boundary density effects on the performance of nanocrystalline- and coarse-grained tungsten. Third, we summarize detailed TEM results (details are presented elsewhere) on the discovery of the rafting mechanism. Finally, void swelling and void shrinkage is studied.

2. Experimental

2.1. Samples

Two tungsten sample grades were used in this study. The first grade is a nanocrystalline and ultrafine tungsten (NCW) grade produced by an orthogonal machining process. The details of this process were mentioned elsewhere [20]. Both nanocrystalline (< 100 nm) and ultrafine (100–500 nm) [57,58] grains coexist in this grade. The second tungsten grade is a coarse grained tungsten (CGW with grains that are larger than 1 μm) provided by ESPI metals, USA. Both grades are high purity (nominally 99.95 and 99.99% for the NCW and the CGW respectively).

TEM samples were prepared from both grades via electropolishing with 0.5% NaOH solution. The average samples thickness is ~100 nm. The grain morphology and texture of the samples prior to irradiation are shown in Fig. 1. Both grades have a high fraction of low angle grain boundaries (LAGBs) (~50% and 65% for the CGW and NCW grades respectively).

2.2. Irradiation

Irradiation of the thin foil samples was performed in the Ion Beam Materials Laboratory (IBML) at Los Alamos National Laboratory (LANL). Irradiation was performed using the Tandem accelerator with 3 MeV Cu^{+} ions at normal incidence. The irradiation was performed *ex-situ* using two different displacement per atom (dpa) rates (0.0167 and 0.000167 dpa/s) at two different temperatures (RT and 1050 K). The samples were irradiated to different dpa values ranging from 0.2 to 4 dpa. To determine the dpa and the dpa rate, the Kinchin-Pease model in the Stopping Range of Ions in Matter (SRIM) Monte Carlo computer code (version 2013) [59] was used to determine the damage event (which was 0.76 vacancy/Ion/