Journal of Nuclear Materials 487 (2017) 311–316

Contents lists available at ScienceDirect

Journal of Nuclear Materials

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

Effects of He radiation on cavity distribution and hardness of bulk nanolayered Cu-Nb composites

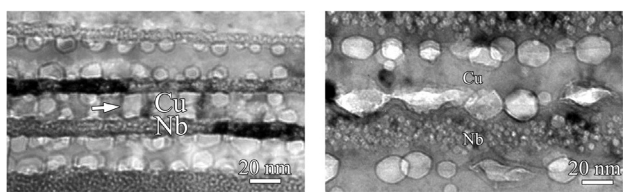
L.X. Yang [a], S.J. Zheng [a, *], Y.T. Zhou [a], J. Zhang [b], Y.Q. Wang [c], C.B. Jiang [a], N.A. Mara [c], I.J. Beyerlein [d], X.L. Ma [a]

[a] Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China
[b] School of Energy Research, Xiamen University, Xiamen 361005, China
[c] LANL Institute for Materials Science and the Center for Integrated Nanotechnologies, Los Alamos National Laboratory, Los Alamos, NM 87545, USA
[d] Mechanical Engineering Department, Materials Department, University of California at Santa Barbara, Santa Barbara, CA 93106, USA

HIGHLIGHTS

- Effects of He radiation temperature, fluence, and layer thickness on damage of ARB nanolayered Cu-Nb composites have been investigated.
- Whether cavities cross the interface depends on layer thickness and temperature.
- He radiation could generate softening mainly owing to recovery of dislocations.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 2 November 2016
Received in revised form
13 February 2017
Accepted 14 February 2017
Available online 21 February 2017

Keywords:
Interface
Cavity
Softening
Radiation
Nanolayered composite

ABSTRACT

Interface engineering is an important strategy for developing radiation tolerant materials. In prior work, bulk nanolayered composites fabricated by accumulative roll bonding (ARB) showed outstanding radiation resistance. However, the effects of layer thickness and radiation conditions on damage distributions and their effect on hardness have not been explored. Here, we use transmission electron microscopy (TEM) and nanoindentation to investigate the effects of radiation on the distribution of radiation-induced cavities and post-radiation hardness in ARB nanolayered Cu-Nb composites. We show that whether the cavities cross the interface depends on layer thickness and temperature, and that, remarkably, radiation could generate softening, not always hardening. We posit that the softening mainly results from the recovery of dislocations stored in the crystal after the bulk forming ARB processing due to He radiation and this phenomenon offsets radiation-induced hardening as layers become finer and temperatures rise.

1. Introduction

Structural materials in nuclear reactors suffer damage due to long-term exposure to high temperatures, high stress, corrosion, and radiation. Voids and bubbles, which are some of the major forms of irradiation-induced damage, are generated by the He radiation that accompanies endothermic (n, α) reactions. These He bubbles and voids lead to detrimental changes in the material microstructure, dimensions, and structural performance, such as swelling, hardening, and embrittlement [1–6]. Hardening, for example, can be severe. In 316LN stainless steel, He radiation that leaves peak He concentrations of 10% in the material has been reported to cause enhancements in hardening ΔH up to 90% percent [7].

The enhancement in hardness, ΔH, is thought to result from the

* Corresponding author.
E-mail address: sjzheng@imr.ac.cn (S.J. Zheng).

http://dx.doi.org/10.1016/j.jnucmat.2017.02.022
0022-3115/

bubbles or voids, or hereinafter generically called cavities, remaining after irradiation and their ability to hinder dislocation motion. Accordingly, ΔH would increase with the size and/or density of cavities in the crystal as they would present larger and/or more frequent obstacles to dislocation glide. This relationship between cavity properties and ΔH has been described by the Friedel-Kroupa-Hirsch (FKH) relation [8,9],

$$\Delta H \approx 3\Delta\sigma_{cavity} = \frac{1}{8} MGbdN^{\frac{2}{3}}_{cavity}, \quad (1)$$

where M is Taylor factor reflecting crystal orientation; G is the shear modulus (GPa); b is the Burgers vector (nm) of the dislocation; d is the cavity diameter (nm) and N is the cavity density (m^{-3}). Thus, for a given material, the larger the cavity size and/or cavity density in the crystal, the higher ΔH is.

Extensive studies have demonstrated that interfaces and grain boundaries can serve as excellent sinks for point defects [10,11], and can store He ions efficiently [12], therefore reducing cavity sizes and densities in the adjoining crystals and minimizing post-irradiation swelling and hardening [13–16]. Using deposited Cu-Nb multilayer thin films as an interface-dominant model material system, Li et al. [16] demonstrated that when the layer thickness (or interface spacing) reduced from 70 nm to 5 nm, the He cavity volume fraction within the layers decreased with concomitant reductions in hardening enhancement ΔH. This study is just one of the many examples suggesting that introduction of biphase interfaces can be an important strategy to design radiation-resistant materials.

Most of these studies on the promising mitigation effects of biphase interfaces were carried out in thin films fabricated by bottom-up techniques, such as physical vapor deposition [15]. Recently, simultaneous high strength and outstanding thermal stability were demonstrated in bulk nanolayered Cu-Nb composites fabricated by a top-down processing method, called accumulative roll bonding (ARB Cu-Nb) [15,17,18]. Unlike bottom-up deposition techniques, the ARB process can be scaled up to manufacture sheet metal in quantities suitable for structural components. Microscopic analyses revealed that just like the prior Cu-Nb nanocomposite studies, no voids formed in the interfaces and only within the crystalline layers, suggesting that void formation was hindered in the interfaces [19]. Moreover, the voids that formed in the crystal near the interfaces tended to stay on the Cu side of the interface and predominately where non-parallel misfit dislocations in the interface intersected [20]. The preference of the Cu phase arises because Cu has a smaller surface energy than Nb and wets the regions of highest interface energy, which is where the misfit dislocations in the Cu-Nb interface intersect [20,21]. Thus, like the thin films, the bulk sheet material exhibits similar behavior, but with it, we have much more versatility in processing and potential for achieving target interface properties [18]. Furthermore, the correlation between void spacing along the boundaries and interface dislocation structure suggests that the distribution of He bubbles or voids could be tuned by engineering the interface.

It should be mentioned that in these previous studies, He was directly implanted into transmission electron microscope (TEM) samples [19,20]. TEM samples are thin films, in which the thicknesses of observable regions are usually less than 100 nm. For such fine thicknesses, He ions transmit across the samples and generating damage in their wake. The type of damage, however, should be different from that generated when the He ions terminate within the bulk material.

Also, as mentioned, temperature, fluence, and grain size (layer thickness) are dominant factors influencing cavity size and distribution [1,4,5,22], and hence ΔH. Such effects have not been investigated in these bulk structural Cu-Nb composites. Here, in order to evaluate the radiation resistance of bulk Cu-Nb composites, we investigate interface effects on cavity distribution and ΔH. As the damage could include either bubbles or voids, hereinafter we use the term cavity to generically encompass both types of damage. Our analyses find that whether the cavities cross the interface depends on layer thickness and temperature, and that, remarkably, radiation could generate softening, not hardening. We rationalize that softening results from annihilation of the dislocations stored within the crystal after the bulk forming ARB processing by interactions with radiation-induced defects, as well as from thermal annealing. For finer layers, such as < 20 nm, there is less room for dislocations to move and dislocation annihilation at cavities dominates over dislocation blockage by cavities. These effects, such as cavity formation in interfaces and softening, were not reported before for nanolayered composites, since other temperatures and fluence conditions were not considered.

2. Materials and methods

The ARB Cu-Nb composites used for this work started with polycrystalline sheets of reactor grade Nb (99.97% pure, ATI-Wah Chang) and oxide-free high conductivity Cu (99.99% pure, Southern Copper and Supply) in a 50-50% volume ratio. Details on the ARB process, which included repeatedly cleaning, stacking, roll-bonding, and cutting, can be found in Ref. [17]. ARB Cu-Nb composites chosen for this radiation study are 300 μm thick sheets with nominal layer thicknesses of 16 and 58 nm respectively. These materials have similar grain structures, with one grain spanning the layer thickness, and textures [23]. He-ion irradiation was conducted using a Danfysik 200 kV ion implanter at Los Alamos National Laboratory at room temperature (RT) and 450 °C. All the samples were mounted on a stage with cooling and heating systems which can monitor the temperature precisely. To reach a fluence of 2×10^{17} ions/cm^2, the radiation process lasted for about 5 h. The SRIM calculation for the radiation of the 58 nm sample shown in Fig. 1a indicates that the most intense damage and He concentration are about 16 dpa and 11 at.% at depths of about 500 and 550 nm respectively. The SRIM calculations for the 16 nm samples showed a similar damage and He-concentration profile vs. depth as those of the 58 nm samples and hence are not shown here for compactness. For comparison, radiation with fluences of 2×10^{17} ions/cm^2 and 6.5×10^{17} ions/cm^2 at RT were performed on the 58 nm ARB Cu-Nb composites, which required about 10 h. More detailed information for the sample can be found in Table 1.

To investigate the distribution of voids and bubbles after irradiation, TEM samples were prepared by a conventional cross-sectioning method, consisting of low-speed saw cutting, mechanical polishing, dimpling, and ion milling on a Gatan precision ion polishing system (PIPS) operated at 3.5 kV. TEM was performed on a Tecnai F30 (FEI) operated at 300 kV.

To test for hardening enhancements, the hardness before and after radiation was measured using nano-indentation on a Nano-indenter G200 (Agilent). Indents were performed to a depth of 500 nm with a target strain rate of 0.05 s^{-1}. Hardness measurements were made in the continuous stiffness measurement mode. Average hardness values were calculated from 16 separate indents with a depth range of 460–480 nm.

Under the He radiation conditions applied here, He concentrations can be detected up to depths of 400–700 nm in both the 16 and 58 nm ARB Cu-Nb samples. Taking the 58-RT sample as an example, as shown in Fig. 1b, the maximum He concentration appears at a depth of 550 nm, which is consistent with the SRIM calculations. This He distribution also agrees well with a previous study using the same He radiation conditions [24]. Hereinafter, we