Role of Interfaces on the Trapping of He in 2D and 3D Cu-Nb Nanocomposites

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Abstract

The role of interface structure on the trapping of He in Cu-Nb nanocomposites was investigated by comparing He bubble formation in nano-multilayers grown by PVD, nanolaminates fabricated by accumulative roll bonding (ARB), and 3D nanocomposites obtained by high pressure torsion (HPT). All samples were implanted with 1 MeV He ions at room temperature and characterized by cross section transmission electron microscopy (TEM). The critical He concentration leading to bubble formation was determined by correlating the He bubble depth distribution detected by TEM with the implanted He depth profile obtained by SRIM. The critical He dose per unit interfacial area for bubble formation was largest for the PVD multilayers, lower by a factor of ~1.4 in the HPT nanocomposites annealed at 500 °C, and lower by a factor of ~4.6 in the ARB nanolaminates relative to the PVD multilayers. The results indicate that the (111)FCC||{(110)BCC Kurdjumov-Sachs (KS) interfaces predominant in PVD and annealed HPT samples provide more effective traps than the (112)KS interfaces predominant in ARB nanolaminates; however, the good trapping efficiency and high interface area of 3D HPT structures make them most attractive for applications.

Keywords: Copper; Niobium; Nanocomposite; Interfaces; Radiation effects; He bubbles

1. Introduction

Implanted helium is known to have deleterious effects on the mechanical properties of most structural materials, as He is insoluble in most materials, coalesces into gas-filled bubbles, and helps to nucleate voids [1]. Extensive research has focused on mitigating the detrimental effects of He, particularly in reactor materials where He is produced in n- α reactions [2-5]. Nanostructured metals should be advantageous for this purpose, as their high densities of grain boundaries and interphase boundaries can trap large amounts of He and thus suppress bubble nucleation [6-9]. Ultrafine-grained single phase materials have been shown to trap helium at grain boundaries, but they are not particularly efficient and can coarsen at high temperatures or during irradiation [10-13]. Interphase boundaries in oxide dispersion strengthened (ODS) steels have also been shown to be good defect sinks, and they are stable in the extreme environment of a nuclear reactor [14-16]. Owing to their complexity, however, they are not suitable for elucidating the relationship between interface structure and He trapping. Interphase boundaries between immiscible metals with semi-coherent interfaces, such as in Cu-Nb [17, 18], Cu-V [19, 20], Cu-Fe [21], and Ag-V [22], are more appropriate for this latter purpose as they are good defect sinks, are stable under irradiation, and their interface structures can be controllably varied, characterized, and modelled [6, 23-25]. Previous research on Cu-Nb and Cu-V produced by physical vapor deposition (PVD) has indeed shown that the Kurdjumov-Sachs (KS) orientation relationship with a dominant (111)FCC||(110)BCC interface habit plane, often referred to as (111)KS, effectively suppresses He-filled bubble formation. This finding has been attributed to the large density of in-plane misfit dislocation intersections in this interface, which provide localized excess volume to capture He atoms [23, 26]. This research examined specimens grown by physical vapor deposition (PVD) and thus just one specific interphase boundary structure. The

present work examines the efficiency of interphase boundaries for trapping He more broadly: still focusing on nanocomposites of Cu-Nb, while also comparing trapping efficiencies for samples prepared by various severe plastic deformation (SPD) techniques, as these procedures are highly conducive to bulk processing of materials [27].

Bulk nanocomposites of Cu-Nb have been fabricated in the present study predominantly by means of SPD using accumulative roll bonding (ARB) and high pressure torsion (HPT) [28-30]. ARB materials are nano-layered composites similar to PVD films [29]. These materials have the same KS orientation relationship as PVD films but with a different habit plane $(112)_{\text{FCC}} \parallel (112)_{\text{BCC}}$, thus referred to as the $(112)_{\text{KS}}$ interface. The density of interface misfit dislocation intersections in this plane is similar to that of $(111)_{\text{KS}}$, but due to the different structure of the misfit dislocation intersections, this interface does not contain significant amounts of localized excess free volume, referred to as constitutional vacancies [23, 28, 29]. Nevertheless, recent studies have concluded that the ARB material has a trapping efficiency for point defects similar to that of the PVD-grown material [31, 32], and it is therefore reasonable to suppose that they will also be good traps for He. In addition to studying the ARB directly, we also investigate ARB Cu-Nb material that has been subjected to subsequent treatment by HPT. The accumulated strain in this case is more than two orders of magnitude greater than during ARB, and it results in a three-dimensional nanocomposite structure. The interfacial area in these ARB/HPT samples is more than five times greater than that of the initial ARB material [28], and thus it is potentially more effective in trapping He on a volumetric basis. We also study He trapping in these ARB/HPT samples but after heat treatment at 500 °C. The heat treatment reduces the total interface area due to coarsening, somewhat, but it also changes the interface structure. As we will show, this treatment has a very positive effect on the He trapping

efficiency. Lastly, we investigate He trapping in PVD Cu-Nb samples. This was done for direct comparison to the bulk Cu-Nb samples, as well as for comparison with PVD Cu-Nb samples studied in ref. [26].

2. Experimental Methods

2.1. Fabrication of Cu-Nb nano-layered composites by ARB and PVD

Nano-layered ARB Cu-Nb bulk composites were prepared by a combination of rolling and annealing steps, following the method detailed in ref. [33]. The samples were composed of 50/50 volume fraction Cu/Nb, using starting materials of 99.99% (Cu) and 99.97% (Nb) purity, corresponding to a 60/40 ratio based on molar fractions. The material selected for the present work was processed to a rolling strain of 10.93, resulting in a nominal layer thickness of 18 nm for each of the Cu and Nb layers, although the average individual layer thickness measured in this work is ≈ 25 nm. The average specific Cu/Nb interfacial area, or Cu/Nb interface area per unit volume, is thus 0.04 nm^{-1} .

For comparison, nano-layered Cu-Nb composites were also prepared by PVD using magnetron sputtering. Films were grown using argon gas at a pressure of 2.1×10^{-3} torr with a background vacuum pressure of 2×10^{-8} torr. The films were grown to a thickness of $2.5 \text{ }\mu\text{m}$ with individual layer thicknesses of 20 nm and 90 nm on $[11\bar{2}0]$ single crystal sapphire, thus resulting in specific interfacial areas of 0.050 nm^{-1} and 0.011 nm^{-1} , respectively.

2.2. Fabrication of 3D nanocomposites by high pressure torsion and annealing

High pressure torsion was applied to the ARB nanocomposite as described in ref. [28]; the shear strain was ~ 2000 . This high strain stabilizes a 3D nanocomposite structure, with a Cu grain size of ≈ 6.2 nm, and a Nb grain size of ≈ 4.7 nm, as determined by transmission electron microscopy (TEM). The specific interfacial area for these samples, based on atom probe

tomography (APT) reconstructions described in ref. [28], is $\sim 0.30 \text{ nm}^{-1}$. After HPT, some samples were annealed for one hour at 500°C under high vacuum conditions, 1×10^{-7} torr. From x-ray diffraction using the Scherrer equation, the Cu grain size was found to increase from 6 nm to 25 nm and the Nb grain size from 5 nm to 19 nm. Based on this increase of grain sizes by a factor 4, the specific interfacial area was estimated to be reduced to $\sim 0.075 \text{ nm}^{-1}$.

2.3. He-ion implantation and SRIM profile

The four sample sets, ARB, PVD, HPT, and HPT-annealed, were irradiated with 1 MeV He ions using a current density of $\approx 1 \times 10^{13} \text{ cm}^{-2}\text{-s}^{-1}$ to a dose of $0.8\text{-}1.6 \times 10^{17} \text{ ions-cm}^{-2}$. The specific dose for each sample is listed in Table 1. The depth distribution of the implanted He in each sample was determined by SRIM simulation and is shown in Figure 1 for the four samples with similar implant dose.

Figure 1: He concentration profile in a 60-40 at% Cu-Nb alloy. Total dose ranges from $1.4 \times 10^{17} \text{ ions/cm}^2$ to $1.6 \times 10^{17} \text{ ions/cm}^2$. Horizontal lines in inset show distances between first and last bubble for each sample.

2.4. Characterization methods

Through-focus transmission electron microscopy (TEM) was used to image the helium bubbles in the implanted samples. Cross-sectional areas of the implanted regions were obtained using a focused ion beam (FIB) (FEI Strata Dual Beam 235 and Helios NanoLabTM 600i) and conventional lift-out methods. Operating voltages from 2 kV to 30 kV and currents from 20 nA to 1 pA were selected to optimize sample quality while minimizing gallium implantation and damage. The thicknesses of the foils were between 50 and 100 nm, as measured by electron energy loss spectroscopy (EELS) using a JEOL 2010EF scanning transmission electron microscope (STEM). As the He bubbles were only a few nm in diameter, they were observed in a JEOL 2010 LaB6 using phase contrast, by acquiring bright-field images for different depths of focus, ranging from -500 nm to +500 nm [34]. In kinematic diffraction conditions, the He-filled bubbles appear bright with dark circular Fresnel fringes in under-focus and dark with bright Fresnel fringes in over-focus. Since it is difficult to achieve optimal imaging conditions everywhere at one time in nano-grained composites, the emphasis was put on achieving best visibility conditions near the transition zones defined in the next paragraph.

For each sample, under-focus and over-focus TEM images were taken to determine the depths at which the first and last bubbles are visible. The widths of these regions were then compared to the He implantation profile from SRIM as shown in Figure 1. The critical He concentrations for bubble formation could then be obtained from the SRIM profile as the He concentration where the measured width of the bubble distribution equals the calculated width in the He implantation profile. The effectiveness of the interface for suppressing He bubbles was then determined by dividing the critical He concentration for bubble formation by the specific interfacial area, or interfacial area per unit volume, with the assumption that all He atoms at concentrations below the critical He concentration are absorbed at the phase boundaries.

3. Results

The five sample sets investigated in this study include the ARB sample with an average individual layer thickness of 25 nm, two PVD-grown samples with 20 nm and 90 nm individual layer thicknesses, the ARB material that was subsequently processed by HPT, and this same HPT-processed material subsequently annealed at 500 °C for an hour. The implant dose, the distance between the first and last visible bubbles, and the effectiveness for trapping He are collected in Table 1 for the five sample sets.

Table 1: The characteristics of each sample viewed in through-focus TEM. Error bars correspond to local variations in visibility range of He bubbles.

	Implant Dose (cm ⁻²)	Visible He- filled Bubble Range (nm)	Critical He Concentration (He-nm ⁻³)	Specific Cu/Nb Interfacial Area (nm ⁻¹)	Interfacial He Storage Capacity (He-nm ⁻²)
ARB	1.4×10^{17}	950 ± 165	0.06 ± 0.04	0.040	1.4 ± 1.0
ARB + HPT	1.4×10^{17}	580 ± 40	0.33 ± 0.06	0.30	1.1 ± 0.2
ARB + HPT + Annealed	1.6×10^{17}	555 ± 25	0.42 ± 0.08	0.075	5.8 ± 1.0
PVD – 20nm	1.6×10^{17}	600 ± 60	0.31 ± 0.12	0.050	6.4 ± 2.6
PVD – 90nm	0.8×10^{17}	730 ± 55	0.08 ± 0.02	0.011	7.0 ± 2.0

The PVD samples are the most efficient in terms of suppressing bubble formation per unit interfacial area. The ARB sample is significantly less efficient among the three 2D samples. It holds the least amount of helium before bubbles are visible, about five times less per unit interfacial area than that of the PVD samples. The qualitative differences in the bubble structure in the PVD and ARB samples, however, appear small; for example, the bubble sizes and densities are similar in the regions where bubbles are observed and they preferentially form on interfaces. Figure 2, specifically, shows under-focus and over-focus TEM images of the PVD – 20 nm sample and the ARB (without HPT) sample near the deep end of the range where bubbles are observed. In both cases the bubbles are $\approx$ 1-2 nm in diameter. As seen on each image in

Figure 2, bubbles form at the interfaces and within the layers at the peak of the helium implant profile, but mostly at or near interfaces when the helium concentrations are reduced. In these lower He concentration regions, bubbles “decorate” the Cu/Nb interfaces, forming on the Cu-side of the interface; similar behavior was reported in ref. [23]. Note that He bubbles were not found at Cu/Cu and Nb/Nb grain boundaries within these regions, and thus grain boundary area was not taken into account for the He storage capacity calculation, see below and Table 1. The He bubble density within the Cu layers is higher than in Nb layers except at the highest He concentration, in which case the density is approximately the same in the two layers.

Figure 2: Under-focus bright field TEM images of (A) ARB sample and (B) PVD-20nm sample at deep end of visible bubble range. Sample surface and peak of implant profile is to the right. Images (C) and (D) are of the same regions in over-focus bright field TEM.

Figure 3 shows the critical He concentration per unit volume as a function of specific interfacial area in PVD grown films. Here data from samples with 20 nm and 90 nm individual layer thicknesses are plotted together with data from ref. [26] for samples with much smaller layer thicknesses, 2.8 nm and 5.6 nm. The critical He concentration decreases inversely with layer thickness, and thus increases linearly with specific interfacial area. This finding agrees with the conclusion in ref. [26] that the critical He concentration per unit interfacial area does not depend on layer thickness. The current work extends this conclusion to much thicker films. This is significant since it demonstrates that for room temperature implantation He reaches the interfaces for layer thicknesses greater than 90 nm. This critical He concentration per unit area was determined from the slope of the linear fit of the present data, see Figure 3, and the value of 6.25 He-nm^{-2} agrees well with that found in ref. [26], $8.5 \pm 2.5 \text{ He-nm}^{-2}$.

Figure 3: Critical He concentration per volume as a function of specific interfacial area of samples with (111)KS orientation relationship; the samples with 0.18 nm^{-1} and 0.36 nm^{-1} specific interfacial area are from ref. [26].

The three-dimensional nanocomposite Cu-Nb sample obtained from the ARB material using HPT had a critical helium concentration per unit volume that was ~ 5 times larger than that of the untreated ARB material. The HPT treatment, however, also increased the interfacial area by a factor of 7.5 such that its critical interfacial efficiency is somewhat reduced from that of the as-prepared ARB materials, $1.1 \text{ He ions/ nm}^2$ compared to $1.4 \text{ He ions/ nm}^2$. The TEM images in Figure 4 show the region of the sample spanning from near the peak He concentration to the end of the visible range. Bubbles are clearly observed in Figure 4(a) (under-focus) and 4(b) (over-focus), but the three-dimensional, interconnected nature of the microstructure prevents a definitive evaluation of whether the bubbles reside at interfaces or in the bulk. It is quite clear that the bubble density decreases quickly from the peak He concentration on the right to the end of range on the left.

Figure 4: (A) Under-focus and (B) over-focus bright field TEM images of HPT sample with strain of ~ 2000 . Implanted surface is towards the bottom-right. Due to the small interconnected three-dimensional nature, it is not possible to tell if bubbles form primarily at interfaces.

Annealing the HPT sample increased the grain size and decreased the specific interfacial area by a factor of ~ 4 . The critical helium concentration, nevertheless, increased slightly. The TEM images in Figure 5 show the region at the peak He concentration and at the end of range in

this sample. Again it is difficult to assess whether bubbles form predominantly at Cu/Nb interfaces, but in some locations there is a tendency to reside at boundaries; whether these are phase boundaries or grain boundaries is presently unknown.

Figure 5: (A) Under-focus and (B) over-focus bright field TEM images of the HPT and annealed sample. Implanted surface is towards the bottom-right. In some regions, identified by circles, bubbles seem to “decorate” boundaries.

4. Discussion

Among the studied materials, He-storage capacity has a distinct dependence on both the type of interface and the quantity of interfacial area. This phenomenon is best represented by the HPT and PVD samples. Although the HPT sample is capable of storing large quantities of helium per unit volume, the interfaces are considerably less efficient in storing He than those of samples with effective interfaces such as the (111)KS , which the PVD samples possess.

To further illustrate the evidence provided, the bubble density distributions for the four differently treated samples are plotted together in Figure 6. As can be seen in the plot, the distributions are asymmetric about the mean range, falling off faster at the deep end following the asymmetry of the helium implant profile, see Figure 1. The asymmetry is most dramatic in

the ARB sample. Each set is fitted with a quadratic fit, which shows that the HPT-annealed sample has the narrowest region of bubbles followed by the PVD – 20 nm sample and the HPT sample, and the ARB sample clearly has the widest range of bubbles. Furthermore the widths of the quadratic fits are very close to the range where bubbles are no longer seen, in table 1.

Figure 6: Bubble density per area vs. depth. Polynomial fits for each sample set are indicated.

Bubbles form preferentially on the Cu-Nb interfaces of the ARB sample, which has dominant (112)FCC||(112)BCC habit plane, but this is not the best interface for trapping He atoms. Computer simulations have shown that the local structure where misfit dislocations intersect plays a very important role, and that it is different for the two interfaces, (111)KS and (112)KS [23]. The (111)KS interface has constitutional vacancy concentrations up to 5 at.% in Cu-Nb, as opposed to zero in the (112)KS interface, and it is at these vacancies where He is strongly trapped [23]. It is thus reasonable that the PVD sample with (111)KS interfaces has a higher efficiency for trapping helium than the ARB sample of comparable layer thickness; consequently it suppresses bubble formation more effectively.

An important finding in this work is that three-dimensional nanocomposite Cu-Nb structures formed by SPD are also very effective in suppressing He bubble formation. In fact, the critical He concentration in the sample processed by HPT was over five times larger than the ARB sample with 25 nm individual layer thickness, and indeed comparable to the PVD sample with similar individual layer thickness. In the present experiment, the HPT samples were processed from the ARB samples, but we point out that the steady state microstructure of Cu-Nb during SPD is independent of its initial structure [35]. Thus, bulk nanocomposites of Cu-Nb with microstructures very similar to the ones investigated in this study can be obtained without the need for ARB. For example, samples prepared by ball milling followed by HPT, will have similar microstructures. A second important finding of this study is that the HPT sample retains its ability to suppress bubble formation, even after thermal annealing at 500 °C. In this case some grain growth occurs, which leads to a reduction in specific interfacial area, but the interfacial storage capacity increases by nearly the same factor. This suggests that such materials can be employed for high temperature applications without loss of its ability to suppress He bubble formation.

In order to better understand He trapping in annealed HPT samples, texture analysis was performed on HPT samples annealed at 500 °C for 1 hour. By using a micro-beam XRD system, areas as small as $100\text{ }\mu\text{m}^2$ were analyzed. This small spot size is important because the strain in our HPT samples varies linearly with radial position, to a maximum radius of only $\approx 4\text{ mm}$. The procedures employed for texture measurements and analyses were reported elsewhere [28]. The results are displayed as orientation distribution functions (ODF) represented by three Euler angles ($\varphi_1, \Phi, \varphi_2$) in Bunge notation; the frequently used ODF sections $\varphi_2 = 0^\circ$ and $\varphi_2 = 45^\circ$ are

displayed in Figure 7. Common shear components for BCC and FCC metals are included for reference.

Figure 7: ODF sections for Cu and Nb phases in the HPT and annealed sample for $\phi_2 = 0^\circ$ and $\phi_2 = 45^\circ$. Scale bars shown are in multiples of random distribution (min=0, max=16 for Cu, min=0, max=4 for Nb). Locations of common shear texture orientations are shown below each ODF section.

The present results are compared to the final texture reported previously on an as-deformed HPT sample [28]. The texture analysis of the annealed sample indicates an overall increase in texture intensity compared to as deformed HPT sample, more significantly for Cu where the maximum intensity rose from about 4 times to about 16 times the random intensity. As seen in Figure 7, copper texture developed a strong A-fiber texture with ND// $\{111\}$, where ND is the direction normal to the surface of the sample (i.e., parallel to the torsion axis during HPT). The main texture components present for Cu were the A_1^*/A_2^* components, corresponding to $(111)[11\bar{2}]$ and $(111)[\bar{1}12]$, and the $A/\bar{A}$ ones, corresponding to $(\bar{1}\bar{1}1)[110]$ and

(111)[$\bar{1}\bar{1}0$]. The texture intensity of Nb also increased after annealing, transforming from a nearly random texture in as-deformed HPT samples to the main texture components at $J/\bar{J}$, corresponding to (110)[$\bar{1}\bar{1}2$] and ($\bar{1}\bar{1}0$)[$\bar{1}\bar{1}2$] and at $E/\bar{E}$, corresponding to (110)[$\bar{1}\bar{1}1$] and ($\bar{1}\bar{1}0$)[$\bar{1}\bar{1}1$], followed by a weak F component corresponding to $\{110\} < 001 >$.

After annealing, the texture of the HPT sample, with predominantly (111)FCC||ND||(110)BCC, is consistent with the KS-relationship, although the three-dimensional phase co-existence at small scale prevented us from determining the dominant interface habit planes. Nevertheless, this texture change results in a 30% increase in the trapping efficiency of the sample, despite the ≈ 4 times decrease in interfacial area. The total processing of the ARB sample by HPT plus annealing thus leads to an increase in the critical He concentration by a factor of seven.

5. Conclusion

The type and orientation of the Cu/Nb interphase interface was shown to be of utmost importance **in the trapping of He atoms before bubbles formed**. There is good agreement with previous research that the ability to store helium in PVD grown samples with the (111)KS orientation relationship scales with layer thickness and is constant per interfacial area. The (111)KS interface is the preferred interface character in the PVD samples, compared to the (112)KS interface in the ARB samples and the more random orientation relationship found in the HPT 3D nanocomposites. A sample with a reduced ability to store helium, such as the ARB sample, can be processed by severe plastic deformation and annealing to greatly improve the storage ability of the sample. This shows great promise for the design of bulk alloys to improve properties that previously could only be achieved by thin film growth.

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