Hardening due to Interfacial He Bubbles in Nanolayered Composites

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A series of helium (He) implantations with varying energies and doses were used to introduce bubbles into interfaces in Cu/Mo, Cu/V and Cu/Nb nanolayered composites. Micro-pillar compression testing revealed that the interfacial He bubbles give rise to modest hardening as compared to those in grain interiors. The flow stress enhancement is proportional to the strength of the un-implanted sample. We discuss the influence of the structure of the interfacial dislocation network, the shear resistance of the interfaces, and atomic-level interface steps on hardening. Interfaces with higher density of misfit dislocation intersections and lower shear resistance tend to provide greater hardening.

Keywords: Interfacial He bubbles, dislocation slip transmission, interface shear resistance

1. Introduction

The mechanical response of metallic materials significantly degrades in the presence of helium (He)-filled cavities and is of particular concern for materials in nuclear power reactors.[1–4] At room temperature, He solubility in metals or alloys is extremely low.[5] He atoms introduced by nuclear transmutation can easily diffuse and aggregate at boundaries, dislocations, or other internal defects.[3,6] They stabilize cavity nuclei that grow into bubbles by absorbing radiation-induced vacancies or by punching out self-interstitial loops.[7–9] Growth and interaction of He bubbles at high temperature can change material microstructure, leading to volumetric swelling and blistering at the surfaces.[10–15] Moreover, He bubbles, distributed either inside grains or at certain interfaces, increase the stress required for yield, leading to hardening, reduced fracture toughness,[16] and embrittlement.[17] Ultimately, He may reduce the service time of nuclear reactor materials dramatically.[4,18–20]

Hardening induced by He bubbles has been widely reported and is thought to be due to pinning of glide dislocations at He bubbles.[21,22] The pinning strength is related to dislocation character, testing strain rate, temperature, bubble size, and the He/vacancy ratio of the bubble, which, in turn, is determined by the rate of He introduction and other external conditions. One strategy for describing the hardening effect of He bubbles is to use the Orowan hardening equation,[23–26] which contains an empirical constant to describe the
obstacle pinning strength. Unlike other barriers, such as voids, the pinning strength of He bubbles must be adjusted depending on the He/vacancy ratio inside the bubble.[27–29] For example, molecular dynamics (MD) simulations have revealed that a 2-nm void is a stronger obstacle than a 2-nm He bubble with low He pressure, whereas when the He/vacancy ratio increases to 5, the He bubble becomes stronger than the void. The increased internal pressure distorts the surrounding lattice and initiates loop punching, hindering the passage of glide dislocations.[30]

How to manipulate the distribution of He bubbles in materials and suppress the corresponding detrimental effects on materials has become a critical issue when designing advanced radiation tolerant materials. Our group proposed an approach that uses interphase boundaries to trap He atoms, thereby removing them from the crystalline layers and making them unavailable for forming either bubbles or dislocation obstacles.[31–40] The trapped He, however, does precipitate at the interfaces.[34,41] By contrast with He bubbles in grain interiors, the influence of interfacial He bubbles on hardening has not been explored. In the present work, we describe experimental investigations of hardening induced by He bubbles located at interfaces. Two prerequisite conditions have to be satisfied in this study: (1) Most of the He bubbles must be located at interphase boundaries; and (2) Dislocation–boundary interaction must be the dominant deformation mechanism.

Nanolayered composites (NLCs) of face-centered cubic and body-centered cubic metals with an interface spacing of ∼5 nm are ideal model systems for studying the hardening mechanisms introduced by interfacial He bubbles. The flow strength of He-free multilayers increases with decreasing thickness of the individual layers. Simultaneously, the mechanisms responsible for deformation in these materials also change: dislocation pile-ups govern deformation in multilayers with layer thickness larger than ∼100 nm, confined layer slip dominates for layer thickness in the range of 5–100 nm, and crossing of interfaces by single dislocations is the primary deformation mechanism for multilayer with layer thickness less than 5 nm. Thus, in our study, the layer thickness should be small enough (≤5 nm) so that dislocation cutting across He bubble-decorated interfaces is dominant.[35,42–44] Moreover, for such low layer thicknesses, the spacing between interfaces is smaller than the mean distance between any He bubbles that may remain in the crystalline layers.[42,43] Thus, Orowan bowing around these bubbles is not the dominant strengthening mechanism for NLCs with layer thicknesses of ∼5 nm and below.

The structure of interfaces in NLCs may be described as a misfit dislocation network.[31] Previous studies revealed that defect formation/solution energies along the interfacial misfit dislocation, especially at the misfit dislocation intersections (MDIs), are lower than the corresponding values in crystalline grain interiors.[41,45] In particular, He bubbles preferentially nucleate at MDIs.[15,41] In the current study, the implanted He concentration is designed so that every interface has an equal number of He atoms at each MDI. Since Cu/Nb, Cu/V and Cu/Mo interfaces each have different numbers of MDIs per unit area,[34] different total He concentrations must be implanted into each interface to achieve equal numbers of He atoms per MDI. These concentrations have been determined previously [34]: ∼8.5 atoms/nm² for Cu/Nb interfaces,[46] ∼3 atoms/nm² for Cu/Mo,[36] and ∼1.9 atoms/nm² for Cu/V.[47]

2. Experiments Cu/Mo, Cu/V and Cu/Nb NLCs with an interface spacing of 5 nm were synthesized using magnetron sputtering at room temperature (referred to as Cu/Mo 5 nm NLC, Cu/V 5 nm NLC, and Cu/Nb 5 nm NLC hereafter). For such low layer thicknesses, the dominant deformation mechanism is dislocation crossing at interfaces.[44] The total thickness of the film was approximately 1.2 μm. A series of He implantations were performed, with varying energies and doses chosen based on SRIM (stopping and range of ions in matter, http://www.srim.org) [48] calculation to produce a controlled distribution of bubbles, in which most of the He bubbles decorate the interfaces. The implanted He concentration is chosen to be the critical concentration at which bubbles are first detected via transmission electron microscopy (TEM) for different NLC systems. As presented in Figure 1, a nearly constant 2 at.% He concentration over a 1 μm film thickness has been implanted into Cu/Nb 5 nm NLC. For Cu/V 5 nm NLC, Cu/Mo 5 nm NLC, and Cu/Nb 5 nm NLC.

![Figure 1](Colour online) SRIM calculations of 2 at.% He concentration profile in Cu/Nb multilayer films at multiple energies and fluences.
and Cu/Mo 5 nm NLC, the implanted He concentration is 0.5 and 0.8 at.%, respectively. Based on our previous studies,[36,46,47] at this critical concentration, most of the He bubbles nucleate at interfaces, thus the interaction of dislocation with He bubbles in layer interiors is minimized. To avoid the non-uniform plastic zone in conventional nanoindentation measurements with a sharp tip, focused ion beam (FIB) has been used to machine micro-pillars with an aspect ratio (height divided by diameter) of 2:1. Uniaxial compression loading was applied at an approximately constant strain rate of $2 \times 10^{-3}$ s$^{-1}$ in a Hysitron Triboindenter. To check for repeatability, multiple as-deposited and He-implanted pillars were compressed. True stress–strain curves are calculated accounting for pillar taper correction.[49]

3. Results and Discussion

A FIBed micro-pillar with a top diameter of 557 nm and a bottom diameter of 613 nm is shown in Figure 2(a). The height of the pillar is 1.2 μm. The distribution of implanted He bubbles at a different depth beneath the surface are presented in Figure 2(b)–(d) with under-focused bright-field cross-sectional TEM (XTEM) images. Close to the top of the surface, the layers present a wave-like morphology, which results from the shadowing process during the sputtering. The relationship between curved morphology and deposition parameters has been studied in detail by Wei.[50] Most He bubbles decorated the interface, with a limited number of bubbles visible inside the layer. Meanwhile, typical XTEM micrographs of implanted Cu/V 5 nm and Cu/Nb 5 nm have been shown in Figure 2(e) and 2(f), respectively. After He implantation to a critical dose, He bubbles, approximately 2 nm in diameter, are preferentially distributed at the interface. The He bubble density within the Cu layers (not at interfaces) has been quantified in all three cases. It is approximately $1.7 \times 10^{23}$/m$^3$ for Cu/Mo, $2.4 \times 10^{23}$/m$^3$ for Cu/V, and $3.7 \times 10^{23}$/m$^3$ for Cu/Nb. The bubble densities are over 10 times lower than in He-implanted multilayers with higher fluences.[35,43,47]

The deformability of Cu/Mo, Cu/V and Cu/Nb 5 nm NLC systems before and after implantation has been explored by the micropillar compression tests and the corresponding stress–strain curves are presented in Figure 3. Red or pink solid lines present the mechanical response of implanted pillars, whereas blue or black dotted lines present that of as-deposited samples. In order to minimize the influence of surface plasticity, multiple loading–unloading schemes have been designed. In the case of Cu/Mo 5 nm pillar compression, three important phenomena have been uncovered:

1. The yield strength increases after implantation. As mentioned before, at the length scale of 5 nm, the mechanical behavior of Cu/Mo NLC micropillar is interface governed. Yielding of an unimplanted NLC initiates when dislocations pass between adjoining crystals through the interfaces. MD simulations revealed that this process occurs through dislocation nucleation along interface misfit dislocation line segments aligned with the trace of a glide plane in the interface.[51,52] Thus, the increase in strength
can be interpreted as an increased barrier to nucleation of lattice dislocation from the He bubble-decorated interfaces.

(2) After yielding, strain hardening is observed in both as-deposited and He implanted pillars. The fiber texture of \{1 1 1\} Cu || \{1 1 0\} Mo is able to provide symmetric slip on multiple slip systems, leading to stable plastic flow at a low strain level (~6%). After 6% compressive strain, shear instability is observed at the top of the pillar and the corresponding scanning electron microscopy (SEM) image in Figure 3(a) captured the formation of a mushroom-shaped top. At large plastic strains, barreling of the pillar is generally expected since the bottom end is fixed and there is high friction between the diamond tip
Figure 4. (Colour online) Flow stress enhancement introduced by interfacial He bubbles of Cu/Mo, Cu/V, and Cu/Nb 5 nm NLC pillars versus the strength of un-implanted counterparts. The increment is proportional to the strength of the un-implanted sample.

and the top surface. The observation here is consistent with the earlier pillar compression study of Al–Al3Sc multilayers.\[53\]

(3) The maximum value of the flow stress, correlated to the critical stress required for dislocation slip transmission across the interface, is increased from 2.8 to 3.1 GPa. The enhancement of the maximum flow stress after implantation indicated a higher barrier strength of He bubbles decorated interfaces to dislocation transmission. As presented in Figure 3(b) and 3(c), the deformation behavior of Cu/V and Cu/Nb 5 nm NLC pillars fell into the same trend. The measurements showed that the flow stress enhancements were 0.3 GPa in Cu/Mo 5 nm (10.7% increase); 0.14 GPa in Cu/V 5 nm (9.8% increase); and 0.2 GPa in Cu/Nb 5 nm (10.5% increase). As shown in Figure 4, the hardening increment is proportional to the strength of the un-implanted sample. The representative SEM micrographs in Figure 3(b) and 3(c) captured the mushroom pillar head with either the crack or shear instability over quite a large strain.

To uncover the mechanical influence of He bubbles at interfaces, true stress—plastic strain curves are shown in Figure 5. In addition, the average true stress enhancement has been calculated within a certain range of strain and plotted as green dashed lines. The average true stress enhancement is calculated using the average true stress value of the implanted sample minus that of the as-deposited sample at the same plastic strain. In all three cases, the hardening rate is larger for implanted pillars at the onset of yield. Onset of shear instabilities occurs at lower strains, as well.

Compared to the separated bubbles in bulk, the dislocation–bubble interaction becomes more complicated at interfaces. Factors such as the He/vacancy ratio inside the bubble, the interface structure, the interface shear resistance, the bond strength, the size of column domain, the waviness of interface morphology, testing temperature, and shear direction, and others will greatly influence the mechanical response of He bubble templated interfaces. Here, we will discuss four important factors in detail: (1) the He/vacancy ratio inside the bubble; (2) interface shear resistance; (3) the structure of the dislocation network at interfaces; and (4) atomic-level
Table 1. The distribution of implanted He atoms at Cu/Nb, Cu/Mo, and Cu/V interfaces.

| Multilayer systems | Implanted He concentration (at.%) | Interfacial He concentration (nm\(^{-2}\)) | Areal density of MDIs (nm\(^{-2}\)) | Average He atoms per MDI |
|--------------------|-----------------------------------|------------------------------------------|-----------------------------------|--------------------------|
| Cu/Nb              | 2                                 | 10                                       | 0.34                              | 29                       |
| Cu/Mo              | 0.8                               | 4                                        | 0.15                              | 27                       |
| Cu/V               | 0.5                               | 2.7                                      | 0.06                              | 45                       |

In addition to their individual influence on strength, these four factors may also exhibit synergistic effects. Our previous studies indicated that the critical He concentration at interfaces to form bubbles detectable via TEM is solely determined by the structure of dislocation network at interfaces, or specifically the areal density of MDIs. However, as measured in our pillar compression tests, the flow stress enhancements were not monotonically related to the density of MDIs. Other factors will also contribute to the flow stress enhancement. MD simulations uncovered that the concentration of He in bubbles will influence zero-temperature interface shear strength.\[^{54}\] For example, the Cu/Nb interface shear strength reaches a maximum when the interface He concentration is \(\sim 6\) atoms/nm\(^2\).

The influence of the He/vacancy ratio in bubbles has been discussed in detail elsewhere.\[^{43}\] In our study, the implanted He concentration is designed to make the average number of He atoms at each MDI nearly equal. As shown in Table 1, around 30 He atoms aggregate on each MDI at Cu/Nb interfaces, 27 at Cu/Mo interfaces, and 45 at Cu/V interfaces. If the number of vacancies to form a bubble of given size is the same at each interface, then the He/vacancy ratio is also nearly equal at each MDI in all three cases.

Another factor that may influence the interaction between dislocations and the interfacial bubbles is the interface shear resistance.\[^{31}\] As illustrated in Figure 6, the process of dislocation slip transmission across interfaces can be divided into three steps. At the beginning, with the glide dislocation approaching the interface, owing to the lower shear resistance of interfaces, the interface will be sheared under the stress field of the glide dislocation. As a consequence, the core of the lattice glide dislocation spreads into a complicated pattern within the interface. The area of the spread region is associated with the interface shear strength. When the shear strength is low, the sheared region will be larger. The first step describes how dislocation cores evolve at interfaces. In the second step, before the onset of dislocation transmission, the strain field associated with the spread dislocation core needs to constrict to a width suitable for slip in the adjacent layer. Thus, if the spread region is larger, the constriction process requires higher applied stress. Finally, a merged dislocation line will be bowed out in-between two intersections, which is similar to the Orowan model (as shown in Figure 6(b)).

The strengthening from templated He bubbles is closely connected to the spacing of two adjoining bubbles and higher density is inclined to provide a higher barrier to slip.

In addition, atomic-level interface steps, which are responsible for a slightly wavy morphology of the interface, may influence interface shear behavior at experimentally accessible temperatures and strain rates.
Recent MD simulations revealed that as the temperature increases, interfacial shear resistance drops rapidly, with lower strain rates giving steeper reductions in shear resistance with increasing temperature. The shear resistance of atomically flat Cu/Nb interfaces approaches zero at room temperature and at experimentally accessible strain rates. However, real Cu/Nb interfaces are not atomically flat: steps at the interface result in non-zero shear resistance. Moreover, similar to MDIs, steps may also be favored locations for nucleating He bubbles. Thus, as illustrated in Figure 6, dislocation-related mechanical response, including dislocation nucleation and transmission, at stepped and atomically flat He bubble templated interfaces may be different.

Compared to the earlier micro-pillar compression results, the maximum flow stress is lower here for Cu/Nb 5 nm.[42] The deviation may result from the different quality of the as-deposited thin film. Factors, such as porosity, internal stress, column size or in-layer grain size, will affect the measured value. However, the magnitude of He bubble-induced hardening is consistent. After 2 at.% He implantation, the flow stress increased 0.2 GPa in comparison to 0.3 GPa after 7 at.% implantation.

We notice that there is no way to isolate each factor from the results of these experimental measurements alone. Here by implanting designed amount of He into three Cu-based multilayer systems, the He/vacancy ratio is nearly equal at each MDI in all three cases. The comparison of the mechanical response of three different dislocation networks with the same amount of He per trapping site becomes very instructive. For equal numbers of He atoms per MDI, the hardening increment is proportional to the initial hardness. Investigating hardening as a function of the number of He atoms per interface MDI would be valuable.[56] In order to clarify the contribution of each factor, further experiments as well as atomistic simulations are planned.[54]

4. Conclusions In summary, Cu/Mo, Cu/V and Cu/Nb NLC with individual layer thickness of 5 nm were synthesized by magnetron sputtering. A series of He implantations with varying energies and doses were performed to generate a distribution of He bubbles at interfaces. The implanted He concentration was tuned to correlate with the density of MDIs, so that the average He concentration at each MDI was approximately equal. The micro-pillar compression tests have been performed and we found after implantation, the flow stress enhancement is proportional to the strength of the un-implanted sample. The interfaces with higher density of MDIs and lower shear resistance tend to provide enhanced hardening. Atomistic simulations of dislocation nucleation and propagation across nanometer scale interfaces decorated with He bubbles at MDIs may provide more insights on the management of He in nanocomposites to suppress the degradation in mechanical response due to He bubbles.

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