Self-organization of helium precipitates into elongated channels within metal nanolayers

Di Chen,1 Nan Li,2 Dina Yuryev,3 J. Kevin Baldwin,2 Yongqiang Wang,1* Michael J. Demkowicz4*

Material degradation due to precipitation of implanted helium (He) is a key concern in nuclear energy. Decades of research have mapped out the fate of He precipitates in metals, from nucleation and growth of equiaxed bubbles and voids to formation and bursting of surface blisters. By contrast, we show that He precipitates confined within nanoscale metal layers depart from their classical growth trajectories: They self-organize into elongated channels. These channels form via templated nucleation of He precipitates along layer surfaces followed by their growth and spontaneous coalescence into stable precipitate lines. The total line length and connectivity increases with the amount of implanted He, indicating that these channels ultimately interconnect into percolating "vascular" networks. Vascularized metal composites promise a transformative solution to He-induced damage by enabling in operando outgassing of He and other impurities while maintaining material integrity.

RESULTS

Plan-view imaging of He channels in a metal trilayer composite

We perform our investigation on a specially designed model material: vanadium-copper-vanadium (V/Cu/V) trilayers synthesized by physical vapor deposition. As shown in the cross-sectional transmission electron microscopy (TEM) image in Fig. 1A, the first V layer is approximately 150 nm thick and is deposited directly onto a single-crystal MgO substrate. A thin, ~5-nm Cu layer is deposited upon the first V layer, and a second, ~50-nm V layer is deposited upon the Cu layer (see Materials and Methods). This sample geometry provides an atomically flat, high-perfection Cu layer sandwiched between two much thicker V layers. Previous investigations have demonstrated that He precipitates nucleate and grow much more rapidly in face-centered cubic (fcc) materials, such as Cu, than in body-centered cubic (bcc) metals, such as V (20), niobium (Nb) (13, 14), or molybdenum (Mo) (21). Moreover, although He precipitates may preferentially adhere to FCC/BCC interfaces, they remain confined within the Cu and do not cross into the adjacent BCC material (13, 14). Thus, the V layers in our sample confine He precipitates within the Cu layer.

We introduce He into these samples using a He⁺ ion beam directed perpendicular to the sample surface, as shown in Fig. 1 (see Materials and Methods). The selected beam energy (20 keV) yields a peak implanted He concentration centered on the Cu layer, whereas the elevated...
implantation temperature (250°C) accelerates the nucleation and growth of He precipitates. To determine how He precipitate morphologies depend on the total amount of He, we implanted samples to four different fluences: $10^{15}$, $3 \times 10^{15}$, $5 \times 10^{15}$, and $10^{16}$ ions/cm$^2$. Figure 1 (A and B) shows edge-on views of the Cu layer after He implantation to $10^{16}$ ions/cm$^2$. The He precipitates appear dark because of their low atomic number under the dark-field scanning TEM (STEM) imaging conditions in Fig. 1A, whereas in the defocused strain contrast image in Fig. 1B, they appear bright. Comparing these figures confirms the presence of He precipitates and demonstrates that they are confined within the Cu layer and do not cross over into the adjacent V, as expected. Spherical He bubbles also nucleate in the V layers but do not grow beyond the nanometer scale.

The cross-sectional TEM foils in Fig. 1 only allow us to observe the Cu layer edge-on. To fully characterize the shapes of the He precipitates, we prepare samples for imaging from a plan-view perspective, that is, looking perpendicular to the V/Cu/V interface planes (see Materials and Methods), as shown in Fig. 2. At the lowest fluence—$10^{15}$ ions/cm$^2$ (Fig. 2A)—we only see isolated, nanometer-scale spherical He bubbles. However, at $3 \times 10^{15}$ ions/cm$^2$ and above (Fig. 2B to D), we observe bright, elongated features and fewer isolated spherical bubbles. We confirm that the elongated features are He-filled cavities—that is, He “channels”—by comparing their appearance in under- and overfocus conditions. Figure 2 (E and F) shows such images for one feature selected from the sample with a fluence of $3 \times 10^{15}$ ions/cm$^2$. Because of its low atomic number, He interacts weakly with the electron beam in TEM. Therefore, a He precipitate in a metal matrix appears as if it were an empty cavity: bright in underfocus and dark in overfocus (22), consistent with Fig. 2 (E and F).

As the fluence rises from $3 \times 10^{15}$ ions/cm$^2$ (Fig. 2B) to $5 \times 10^{15}$ ions/cm$^2$ (Fig. 2C) and $10^{16}$ ions/cm$^2$ (Fig. 2D), the length and number of He channels increase. We characterize these changes by measuring the projected area of the channels in plan-view images and plot it as a function of fluence in Fig. 3. Because the channels are confined to the Cu layer and span its entire thickness, as shown in Fig. 1A, there is no possibility of channel overlap in the plan-view images. Thus, area fraction does not under- or overcount the channels. Figure 3 also shows the average He concentration in the Cu layer—computed using the SRIM (Stopping and Range of Ions in Matter) code (23)—corresponding to each fluence. The absence of visible channels at the lowest fluence suggests that a critical He concentration in the Cu layer of approximately 0.15 to 0.3 atomic % is required to form the first channels. This observation is consistent with previous investigations—which found that a critical He concentration per unit interface area of ~1.9/nm$^2$ is needed to form visible bubbles at Cu/V interfaces (24, 25)—provided that the Cu/V interfaces in our sample sequester all of the He in the Cu layer as well as within a ~10-nm distance of the interfaces in the V layers. This deduction is consistent with the view that fcc/bcc interfaces are excellent sinks for He (25).

Figure 3 also shows that the He channel area coverage does not increase at a constant rate with fluence. Rather, at higher fluences, its rate of increase is smaller than at lower fluences. This effect may be due to enhanced sequestration at high fluences of He into traps too small to image in TEM, such as vacancies or vacancy clusters, dislocations, or high-volume sites at Cu/V interfaces (25). Alternatively, it may also indicate that a higher fraction of He is being transported out of the sample. Because of its extremely low solubility, we expect that He trapped in precipitates cannot reenter solution and diffuse through the metal matrix. Therefore, any significant He transport necessitates high-mobility paths that do not require He reentering into solution.

He channels may serve as such high-mobility pathways, provided that they interconnect into a system-spanning network that enables long-range He transport. In this context, it is intriguing that the channel area coverage shown in Fig. 3 appears to approach ~10 to 12% asymptotically as fluence increases, a value consistent with the expected area...
coverage at the bond percolation threshold in two dimensions (26). Furthermore, we expect that channels that link up into networks form kinks (when two channels join at their end points) and junctions (when the end point of one channel connects to a midpoint of another). Figure 4 shows that the total number of these kinks and junctions per unit channel length increases with fluence, consistent with rising interconnectivity of He channels.

**Phase-field modeling of He channel formation**

The stability of He channels in our trilayer sample is due to their confinement within the Cu layer. Unconstrained cylindrical precipitates (for example, those in uniform single crystals or on flat interfaces) break up via the P-R instability (11, 12, 15) because their surface energy decreases monotonically at every stage of their transformation into discrete, equiaxed precipitates, that is, there is no energy barrier for this transformation. By contrast, a shape perturbation imposed upon an elongated precipitate pinched between two impenetrable planar surfaces initially incurs a surface energy increase (27). Thus, even if the breakup of these elongated precipitates ultimately reduces their total surface energy, it cannot occur without first surmounting an energy barrier. This barrier stabilizes He channels confined within a metal layer against the P-R transformation.

Although confinement within a layer explains the morphological stability of He channels, it leaves open the question of how they form. The area-minimizing—and therefore lowest-energy—shape for He precipitates confined within our Cu nanolayer is a “pancake” sandwiched between the adjacent V layers. Why do He precipitates not take on this shape as they grow? The answer relies on the preferential nucleation of He precipitates at specific, high-energy locations within Cu/V interfaces.

Far from being featureless dividing surfaces between adjacent crystals, these interfaces have definite, location-dependent internal structures consisting of intrinsic (misfit) (25, 28) and extrinsic (20) dislocations as well as constitutional vacancies (25). Associated with these non-uniform structures are location-dependent interface energies (16). He precipitates wet the highest-energy regions within an interface, resulting in preferential nucleation and growth at interface dislocations (20) and on intersections between them (16, 29). Additional factors, such as local impurities, may also influence the distribution of He bubble nucleation sites at interfaces and the kinetics of subsequent precipitate growth (30).

To elucidate the mechanism of He channel formation, we use a recently developed phase-field model that accounts for preferential wetting of interface defects by He precipitates (see Materials and Methods) (31). Our simulation domain consists of a layer bounded by two impenetrable interfaces, as shown in Fig. 5A. Each interface contains a ribbon that is wettable by He precipitates (with a contact angle of 120°), whereas the interface area outside the ribbon is nonwettable. The ribbons represent straight, parallel interface dislocations at adjacent interfaces, similar to those created by threading dislocations gliding through a layer (20). The layer is under periodic boundary conditions in the directions parallel to the interface planes.

We initiate the simulation by creating a series of equally spaced He precipitate nuclei along the wettable ribbons, as shown in Fig. 5B. Additional He is then introduced into the precipitates quasi-statically, causing them to grow, impinge upon their neighbors, and eventually coalesce into larger precipitates extended along the wetting ribbons, as illustrated in Fig. 5C. These coalescence events first occur at precipitate volumes per channel length of ~10 nm³/nm, corresponding to bubble radii of ~1.8 nm. As they continue to grow, precipitates wetting opposing interfaces begin to coalesce as well (at a precipitate volume of ~34 nm³/nm), ultimately connecting up into elongated He channels spanning the entire layer thickness, such as the one in Fig. 5D.

Thus, the wetting ribbons serve as a template that aligns He precipitate nuclei, guiding their subsequent growth and coalescence into He channels. We repeat the foregoing simulation for contact angles of 30° and 90°, a wettable ribbon on only one interface, and wettable ribbons replaced by rows of discrete wettable patches. All of these simulations lead to the formation of elongated channels following a similar sequence of steps as in Fig. 5, demonstrating that the proposed channel formation mechanism applies to a wide range of interface structures.

**DISCUSSION**

The self-organization of He into elongated channels is a profound departure from the classical life cycle of He precipitates in metals (3).
In conventional, coarse-grained alloys, He precipitates nucleate as nanoscale spherical bubbles and later grow by vacancy capture into large, He-filled voids while remaining equiaxed (3, 5). The physical mechanisms governing the formation of elongated He precipitates are distinct from those governing the growth of equiaxed He precipitates. Therefore, He channels are not bubbles or voids in the classical sense. Rather, they are best described as a new class of He precipitates.

He channels have potentially far-reaching consequences for the retention and transport of implanted He in metals. He trapped in equiaxed precipitates is effectively immobilized and escapes only once the precipitates break through the surface of the material in micrometer-scale blisters (7, 8). By contrast, the coalescence of He bubbles into elongated channels and the subsequent linkup of channels into systemspanning vascular networks may provide pathways for He to outgas from the material without damaging its surfaces.

Indications of damage-free He outgassing were previously found by Demkowicz et al. (32) in the case of Cu/Nb multilayer composites. The authors introduced He by implantation at temperatures ranging from 490° to 660°C into composites with individual layer thicknesses from 4 to 100 nm. Using elastic recoil detection analysis, they demonstrated progressively lower He retention as temperature increased and as layer thickness decreased. Scanning electron microscopy imaging of sample surfaces found remarkably few blisters, indicating that the outgassing mechanism did not involve the creation of surface damage.

McPhie et al. (33) and Dunn et al. (34) developed dedicated models to assess the likelihood that accelerated outgassing in the Cu/Nb experiments occurs via short-circuit atomic diffusion along Cu/Nb interfaces. They concluded that this mechanism cannot explain the observed outgassing trends. In particular, they showed that, under the assumptions of their model, reductions in layer thickness predict greater He retention, rather than faster outgassing. They therefore propose that a mechanism other than interfacial diffusion must be responsible for the experimentally observed outgassing trends. He outgassing through vascular networks of nanochannels is a viable alternative hypothesis to explain the Cu/Nb experiments of Demkowicz et al. (32). According to the results presented here, the primary role of reduced layer thickness in He outgassing may not be to elevate interface area per unit volume but rather to confine He precipitates into narrow layers, increasing their likelihood of linking up into elongated channels.

Previous TEM imaging of Cu/Nb multilayers implanted with He at elevated temperatures found indications of precipitate linkup within Cu layers (13, 14). However, these previous studies only imaged the Cu layers looking parallel to Cu/Nb interface planes and so were not capable of observing any elongated channels that may have formed. By contrast, we are able to observe channels in the present work because we image the Cu layer in our sample looking perpendicular to the Cu/V interface plane. Additional plan-view imaging of He precipitate morphology evolution is needed to gain further insight into nanochannel formation, in particular to identify the factors that determine their lengths and directions. In situ imaging during He implantation may further illuminate the mechanisms of their formation and linkup as a function of time.

Nuclear fusion is one application area where He outgassing through vascular networks may have important technological consequences. A key concern in fusion is the degradation of PFM due to precipitation of implanted He (9). In single-element PFMs such as tungsten (35, 36), equiaxed He precipitates link up and form a foam-like structure (37–39), devastating mechanical cohesion and thermal conductivity (40). However, vascularized solids may allow He and other impurities such as tritium (41, 42) to escape while maintaining material integrity.

There are several promising routes for bulk-scale processing of nanocomposite materials that support self-organized vascular structures. One option is to synthesize highly textured laminates using severe plastic deformation (43, 44). Another route uses liquid metal dealloying to create composites with three-dimensional interpenetrating metal phases (45, 46). Finally, continuous irradiation may drive spontaneous phase patterning into steady-state nanocomposite structures in some alloys (47, 48). All of these processing methods result in multiphase metal nanocomposites that provide the conditions needed for the formation of He channels, that is, nanoscale confinement of He precipitates between interfaces with location-dependent energies. Each of them requires extensive additional testing to determine their overall performance under plasma exposure (49).

**Fig. 5. Phase-field simulations of He precipitate growth and coalescence.** (A) Simulation domain with top and bottom boundaries representing Cu/V interfaces, each of which contains one wettable strip (red). (B) Initial distribution of He precipitate nuclei on wettable strips. (C) Growth and coalescence of He precipitates along individual strips. (D) Growth and coalescence of He precipitates wetting opposing interfaces.
MATERIALS AND METHODS

Sample synthesis
V/Cu/V samples were deposited using electron beam evaporation from high-purity (99.999%) Cu and V targets at the Center for Integrated Nanotechnologies (CINT) at Los Alamos National Laboratory. The base pressure was 6.66 × 10⁻⁶ mbar, and the process pressure while the substrate was heated to 650°C was 6.66 × 10⁻² mbar. The first layer of V was deposited at a substrate temperature of 650°C. The following two layers (Cu and V) were both deposited at a temperature of 100°C. The deposition rate for all three layers was 0.2 nm/s.

He implantation
Helium ion implantation was conducted using the 200-kV Danysfik ion implanter at Ion Beam Materials Laboratory at Los Alamos National Laboratory. The implantation was performed at 250°C using 20-keV He⁺ ions to fluences of 3 × 10¹⁵, 5 × 10¹⁵, and 10¹⁶ ions/cm² with a flux of ~3 × 10¹² ions/cm²/s. The ion beam direction was perpendicular to the sample surface. The depth profile of implanted He was determined using the SRIM code (23).

Plan-view TEM imaging
Both plan-view and cross-sectional TEM foils were prepared using the focused ion beam (FIB) lift-out technique (20). For plan-view foils, the MgO substrate was carefully sputtered away completely. Both microstructure and chemical analyses were conducted using an FEI Tecnai F30 field emission gun TEM. We used through-focus imaging to observe the He precipitates (22).

To determine whether the Ga ion beam in our FIB causes any material damage, we created TEM foils of as-prepared (that is, not implanted with He) V/Cu/V samples. We prepared both cross-sectional and plan-view foils using identical techniques as were used for the He-implanted samples. In particular, we used FIB exactly the same way in both sets of samples. We then compared TEM images from these two sets of samples to determine whether we could observe any FIB-induced damage (see figs. S1 and S2). The as-prepared samples exhibited no evidence of any defect structures, such as those seen in the He-implanted samples. In particular, there is no evidence of Ga beam–induced damage in any of these samples. Therefore, we conclude that the Ga beam is not responsible for the elongated precipitates that we term “He channels.”

Phase-field modeling
We simulated the growth and coalescence of He precipitates using a phase-field model that represented the Cu-He binary system with a single order parameter governed by the Cahn-Hilliard equation. A detailed description of this model may be found in the study of Yuyerb and Demkowicz (31). Location-dependent wetting and nonwetting behavior on the two interfaces bounding the simulation domain was imposed using classical Dirichlet and specially developed Neumann boundary conditions, respectively. He was loaded directly into the precipitates by increasing the volume integral of the order parameter in a quasi-static manner. The simulations were carried out using the MOOSE (Multiphysics Object Oriented Simulation Environment) software package (30) and required approximately 2 million CPU (central processing unit) hours to complete on the Ada machine at the Texas A&M University High Performance Research Computing Center.

SUPPLEMENTARY MATERIALS
Supplementary material for this article is available at http://advances.sciencemag.org/cgi/content/full/3/11/eaao2710/DC1

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