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Cavity Evolution at Grain Boundaries as a Function of Radiation Damage and Thermal Conditions in Nanocrystalline Nickel

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Enhanced radiation tolerance of nanostructured metals is attributed to the high density of interfaces that can absorb radiation-induced defects. Here, cavity evolution mechanisms during cascade damage, helium implantation, and annealing of nanocrystalline nickel are characterized via *in situ* transmission electron microscopy (TEM). Films subjected to self-ion irradiation followed by helium implantation developed evenly distributed cavity structures, whereas films exposed in the reversed order developed cavities preferentially distributed along grain boundaries. Post-irradiation annealing and orientation mapping demonstrated uniform cavity growth in the nanocrystalline structure, and cavities spanning multiple grains. These mechanisms suggest limited ability to reduce swelling, despite the stability of the nanostructure.

Keywords: *In situ* TEM, Radiation, Helium Implantation, Cavity Evolution, Nanocrystalline Nickel

Growing interest in nanostructured materials has revealed that, when structurally stable, such materials show a high tolerance to radiation damage compared to conventional coarse-grained materials.[1,2] These properties are attributed to the high density of grain boundaries and interfaces that serve as traps for irradiation-induced defects [3–5] and retard dislocation climb and glide.[6,7] Recent experimental studies of irradiated nanocrystalline metals have demonstrated that the resulting cavities are preferentially distributed along grain boundaries with a significantly lower density in the grain interior,[8] indicating the propensity of grain boundaries to act as efficient defect and helium sinks. Radiation-induced swelling may be reduced if the boundaries act to hinder cavity growth, as has been seen experimentally in irradiated nanostructured materials.[5] However, the opposite effect has also been observed experimentally, where, for certain size [9] or temperature [10] ranges, smaller grains resulted in

more defect accumulation or larger swelling compared to larger grains of the same material. Apparently, the efficiency of planar defects (grain boundaries and interfaces) to act as helium and defect sinks and limit the resulting cavity growth may be significantly affected by the character of the boundaries themselves as well as the irradiation-induced defects and the radiation environment.

This manuscript investigates the effectiveness of grain boundaries to act as cavity sinks and to limit the cavity growth in nanocrystalline pulsed laser deposited (PLD) nickel upon successive self-ion irradiation, helium implantation, and annealing. Considered as a model nanocrystalline face-centered cubic (FCC) system, PLD nickel films were selected for this study due to the extensive available literature on microstructural characterization and investigation into their associated response upon thermal treatments and ion irradiation.[11]

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It is well documented that a significant difference in final defect microstructure can occur when performing self-ion irradiation and helium implantation sequentially vs. concurrently.[12–14] The difference is thought to be a function of the density of sinks for helium trapping as it is implanted. Self-ion irradiation results in collision cascades and large immobile clusters,[15] which may act as sinks for helium. In contrast, the self-interstitial atoms (SIAs) created during helium implantation are highly mobile at room temperature,[16,17] allowing for recombination with vacancies and a reduced density of sinks for helium. In this manuscript, self-ion irradiation and helium implantation are performed sequentially, in both orders, which allows for the characterization of mechanisms that influence helium mobility as well as the interaction between helium and other damage types such as Frenkel pairs and cascades. Through this systematic study, we intend to probe the role of irradiation sequence on the damage behavior at grain boundaries and the effect of grain boundaries on the evolution of cavity structure with temperature. The results of this study indicate that a reversal of the sequence of self-ion irradiation and helium implantation significantly affects the final defect microstructure, with preferential cavity formation along grain boundaries resulting only in the helium pre-implantation case. Grain boundaries were found to have negligible ability to limit the growth of cavities with temperature.

Electron transparent nanocrystalline nickel films were produced by PLD to a thickness of approximately 80 nm on a NaCl substrate at nominally room temperature. Additional information on the deposition process can be found in Ref. [18]. The salt substrate was then dissolved in a deionized water bath to obtain freestanding nickel films, which were then floated onto a copper clamshell transmission electron microscope (TEM) grid. To establish columnar grain structure, the films were annealed at 550°C for 5 min, resulting in a strong {100} texture.

To determine the structural evolution associated with He⁺ implantation and displacement damage, careful attention needs to be paid to irradiation conditions. Thus, the Stopping Range of Ions in Matter (SRIM) recoil simulation in the ‘detailed calculation with full damage cascade’ mode [19] was used to estimate the displacement damage caused by the ions and the penetration depth of the helium within a TEM film. The simulations suggested that 3 MeV nickel ions (Ni³⁺) would pass fully through the film and create a nearly uniform damage profile throughout the film, and that 10 keV helium ions (He⁺) would reach a maximum concentration approximately 20 nm from the entrance surface of the film and a maximum depth of approximately 60 nm. Irradiation and implantation experiments were conducted utilizing the *in situ* ion irradiation TEM (I³TEM)

facility at Sandia National Laboratories.[20] A 3 MeV Ni³⁺ beam and a 10 keV He⁺ beam were aligned onto the TEM holder of a JEOL 2100 TEM operating at 200 kV. The two ion beams both entered the TEM along nominally the same path, nearly orthogonally to the electron beam. The nickel foil was tilted approximately 30° toward the ion beams, permitting real-time observation.

To investigate the effect of successive helium implantation and self-ion irradiation and the consequence of the sequence on the final microstructural features, two separate experiments were performed with the irradiation parameters summarized in Table 1. The goal in both experiments was to create nanometer-sized cavities which were visible in the TEM at room temperature, and final doses were adjusted to achieve this in both irradiation sequences.

In the first experiment, the nickel foil was irradiated with 3 MeV Ni³⁺ ions at an average flux of 1.5×10^{11} Ni³⁺/cm²s to a dose of 5.4×10^{14} Ni³⁺/cm² which resulted in approximately 1.8 displacements per atom (DPA). The sample was then implanted with 10 keV He⁺ ions at an average flux of 10^{13} He⁺/cm²s until visible cavities were present, at an approximate concentration of 2×10^{16} He⁺/cm², corresponding to an additional 1.5 DPA, for a total of approximately 3.3 DPA. See supplemental Figure S1(a) for the SRIM simulated damage profile and implanted helium concentration profile.

The second sample was subjected to the reverse order of irradiation, such that it was first implanted with helium to a concentration of approximately 8×10^{16} He⁺/cm², corresponding to nearly 5.5 DPA. The sample was then irradiated with 3 MeV Ni³⁺ ions until cavities were visible, which corresponded to an additional 0.9 DPA. A higher helium dose than the first sample was chosen because 10 keV helium ions create only Frenkel pairs, which are more likely to annihilate than the cascade damage created by the self-ions. The decrease in helium sinks compared to the first experimental conditions likely resulted in an increase fraction of helium atoms escaping the free surfaces; therefore, a higher concentration was chosen in attempt to achieve similar cavity structures as the first experiment. See supplemental Figure S1(b) for the SRIM simulated damage profile and implanted helium concentration profile.

The thermal stability of the cavity structures created by irradiation was evaluated by post-irradiation annealing experiments carried out in a Philips CM30 TEM at 300 kV using a Gatan 628 single tilt heating stage. The ImageJ software was used to measure the diameter of visible cavities. Orientation mapping of the irradiated and annealed samples was performed at room temperature in a JEOL 2100 TEM equipped with Nanomegas ASTAR system mapping (Automated Crystal Orientation/Phase mapping for TEM). Data were collected in

Table 1. Self-ion irradiation and helium implantation parameters.

	Ni ³⁺ Rate	Ni ³⁺ Dose	Damage	He ⁺ Rate	He ⁺ Dose	Damage	Total damage
Sequence	Ni ³⁺ /cm ² s	Ni ³⁺ /cm ²	DPA	He ⁺ /cm ² s	He ⁺ /cm ²	DPA	DPA
Ni ³⁺ , He ⁺	1.5×10^{11}	5.4×10^{14}	≈ 1.8	10^{13}	2×10^{16}	≈ 1.5	≈ 3.3
He ⁺ , Ni ³⁺	1.5×10^{11}	2.7×10^{14}	≈ 0.9	10^{13}	8×10^{16}	≈ 5.5	≈ 6.4

TEM diffraction mode with Alpha 3, 10 μm condenser aperture, and 5 nm step size and a procession angle of 0.3°. Grain boundaries were analyzed using TSL OIM software. The cubic 60° pseudo-symmetry boundaries were removed and grain boundaries with less than 3° misorientation were removed to reduce noise. The combination of these techniques permitted the observation of the structural response of nanocrystalline nickel to self-ion irradiation, helium implantation, and annealing in real-time with nanometer resolution.

The sequence of self-ion irradiation and helium implantation is known to have a significant impact on the final defect structure in many materials.[12–14] The first part of this study involved room temperature self-ion irradiation and helium implantation, in both forward and reverse order, to study the effect of the sequence of ion irradiation on the final defect microstructure and, particularly, the cavity structure in relation to grain boundaries in nanocrystalline nickel. Figure 1(a) shows the defect microstructure after room temperature self-ion irradiation to a final dose of 5.4×10^{14} Ni³⁺/cm² after which significant displacement damage is seen. After this first stage of the sequence, through-focus imaging revealed no visible cavities. Helium ions were then implanted into the sample to a final dose of 2×10^{16} He⁺/cm². No change was noted in the loop structure; however, as illustrated in Figure 1(b), nanometer-sized cavities formed, ranging from 1.5 to 4 nm in size, and were evenly distributed throughout the grain, with no observed preference for the grain boundaries. The cavity size and apparent density were similar to that seen in other room temperature helium implantation studies into nickel.[21] The density of cavities is high enough that there is frequently overlapping cavities through the thickness of the film, making accurate quantification challenging [22] and was not performed here.

The reverse order of implantation and irradiation is demonstrated in Figure 1(c) and 1(d). Helium was implanted first to a concentration of 8×10^{16} He⁺/cm², which resulted in visible defect damage but no visible cavities (Figure 1(c)). The sample was then self-ion irradiated to a final dose of 2.7×10^{14} Ni³⁺/cm² at which point significant damage as well as a high density of cavities was visible (Figure 1(d)). In contrast to the previous experiment, the cavities appeared larger at the grain boundaries than in the interior of the grain, making the grain boundaries particularly visible in Figure 1(d)

(several examples are marked with arrowheads). The cavities at the grain boundaries appear to be elongated and ellipsoidal, forming connected strings of cavities with little or no distance between them. Supplemental Figure S2 is higher magnification over-focus and under-focus images of ellipsoidal cavities located along a vertical grain boundary.

Both sequences of irradiation and helium implantation resulted in a large density of nanometer-sized cavities; however, larger, connected strings of cavities at grain boundaries were only observed for the case of helium implantation followed by self-ion irradiation. A comparison of Figure 1(b) and 1(d) demonstrates the difference in cavity structure as a result of implantation and irradiation sequence. This is likely the result of different densities of sinks for helium in the different irradiation conditions. In the first case, Figure 1(a) and 1(b), self-ion irradiation resulted in displacement cascades and the formation of vacancy clusters evenly distributed throughout the foil. Vacancies and vacancy clusters are known to be immobile in nickel at room temperature.[17] When the helium was later implanted, these immobile vacancy clusters acted as sinks, preventing the helium from reaching the grain boundaries and resulting in a uniform distribution of visible cavities. When the order of irradiation and implantation was reversed, Figure 1(c) and 1(d), fewer stable sinks for helium were present in the nanocrystalline nickel, and thus the helium ions had a higher probability of diffusing to larger sinks such as grain boundaries. This is consistent with several other experimental studies which have observed a preferential accumulation of helium at grain boundaries in nickel.[23–25] When the nickel sample was then self-ion irradiated, the resulting irradiation-induced vacancies formed visible cavity structures, particularly in areas where there was already a high density of helium, such as the grain boundaries.

The preferential distribution of cavities along planar defects has been hypothesized to decrease the total density and volume of cavities and the overall effect of swelling.[5] However, this effect may be dependent on the thermal stability of the cavities at the grain boundaries or interfaces. Post-irradiation annealing of the nanocrystalline nickel film which was first helium implanted and then self-ion irradiated was carried out to characterize the thermal stability of the cavity structure along the grain boundaries. Figure 2(a)–(d) shows the

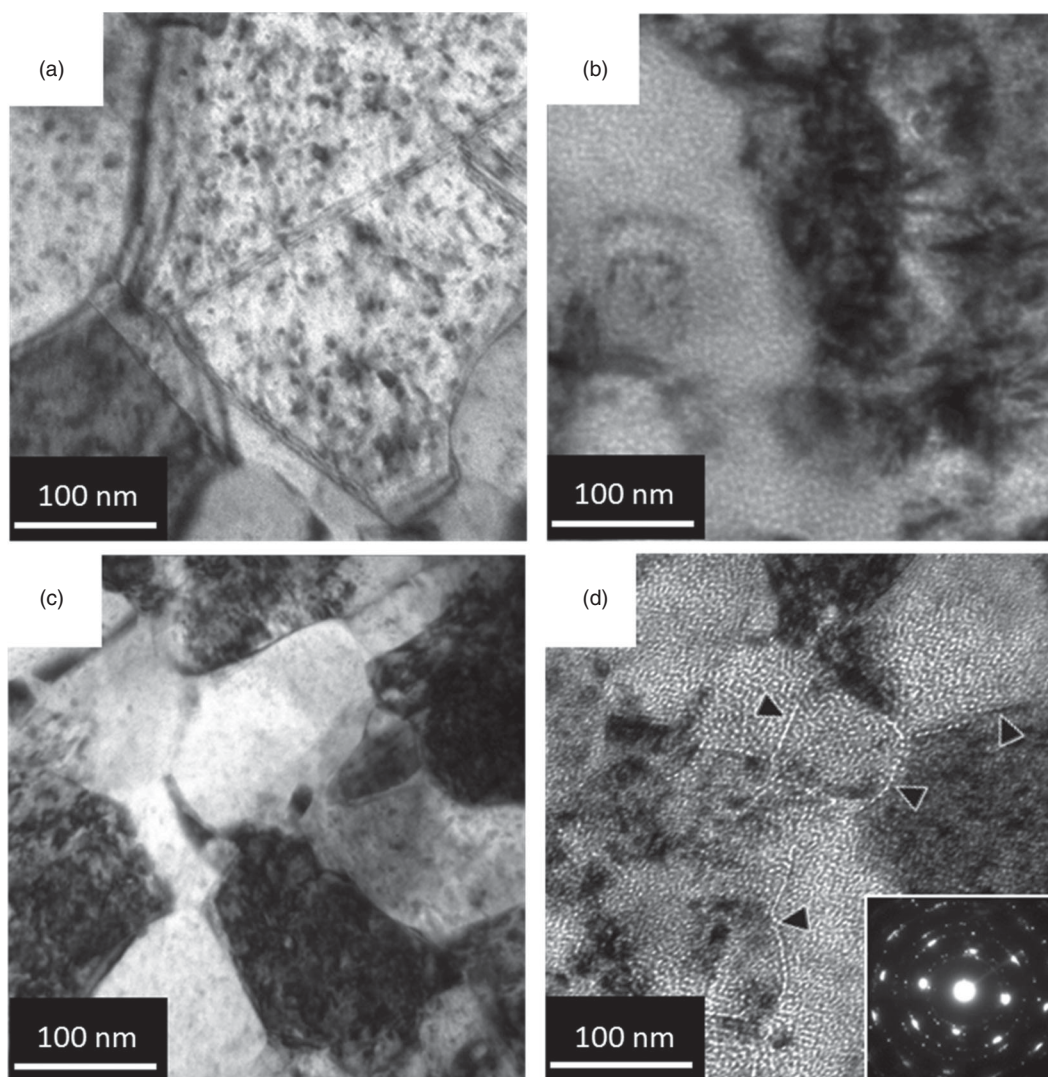


Figure 1. (a) Nanocrystalline nickel irradiated with 3 MeV Ni^{3+} and (b) the same film after the additional implantation of $2 \times 10^{16} \text{ He}^+/\text{cm}^2$. Nanometer size voids are evenly distributed throughout the grains. (c) Nanocrystalline nickel implanted with 10 keV He^+ to a total concentration of $8 \times 10^{16} \text{ He}/\text{cm}^2$ and (d) the same film after additional self-ion irradiation. Nanometer-sized voids were present throughout the film, but with a higher concentration along grain boundaries (black arrowheads point at several examples).

evolution of the cavity structure present in Figure 1(d) as the sample was annealed to 400°C. Supplemental Figure 3 shows the cavity structure at smaller temperature intervals. The large, elongated cavities along grain boundaries remained visible until above 200°C. As the temperature was increased to 230°C (Figure 2(a)), the average cavity diameter increased to 4 nm with some cavities as large as 12 nm. Supplemental Figure S4 displays histograms of cavity size as a function of temperature. No preferential cavity growth was observed between cavities in the interior of the grain and cavities at the grain boundaries despite the initially larger size and elongated shape apparent at the grain boundaries. In addition, the larger size of cavities along the grain boundaries was no longer discernible. At 350°C

(Figure 2(b)), the cavities had an average size of over 10 nm, with several cavities having diameters larger than 40 nm. It is thought that, in this temperature regime, many of the He^+ bubbles present at room temperature have attracted significant amounts of the now mobile vacancies and have transformed into cavities dominated by the accumulated vacancies. The vacancies could have been absorbed from Frenkel pair damage created during He^+ implantation and the cascade damage created during self-ion irradiation, or even from the far-from-equilibrium boundaries resulting from the PLD process.[18] Increasing the annealing temperature to 400°C (Figure 2(c)) resulted in a multimodal cavity size distribution, visible in Figure 2(d) and supplemental Figure 2. In this distribution, a high concentration

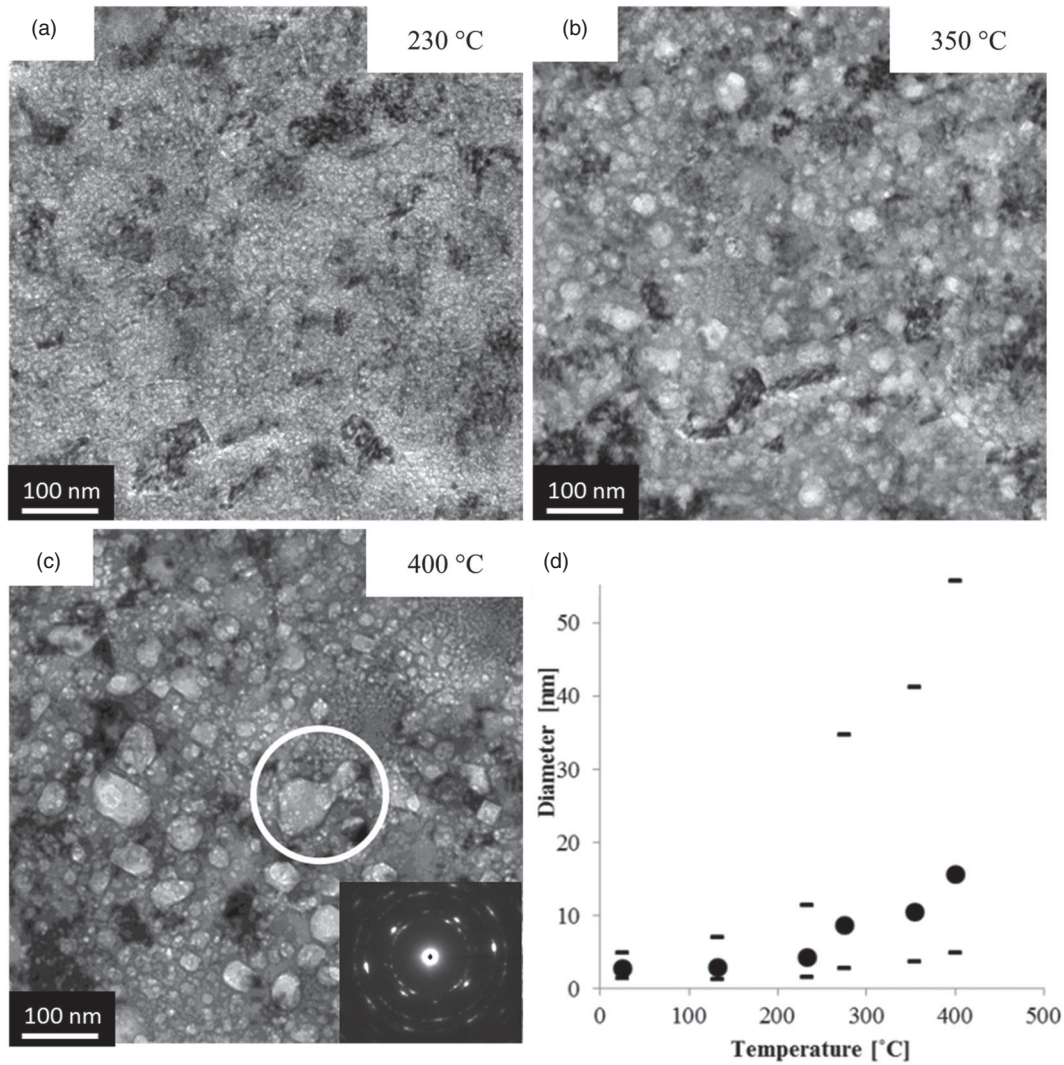


Figure 2. Annealing of the helium implanted and then self-ion irradiated film from Figure 1(d). (a) At 230°C, the cavities have grown large enough that the grain structure is no longer visible. (b) Annealed film at 350°C. (c) Annealed film at 400°C. Inset diffraction pattern shows the {100} grain texture has been maintained. (d) Evolution of the diameter of cavities as a function of the temperature with the maximum, mean, and minimum displayed.

of cavities with diameters centered around 10 nm and a small number of large cavities with diameters ranging between 40 and 70 nm, most of which display a faceted structure, were present. Several cavities had reached or surpassed the size of the grains and were nearly as large as the thickness of the film. Figure 2(d) shows a plot of the average cavity diameter at several annealing temperatures. Lines indicate the maximum and minimum diameters of cavities at each temperature. During annealing from room temperature to 400°C, the minimum diameter grew by less than 4 nm, while the maximum diameter increased by nearly 60 nm. The average cavity diameter remained below 5 nm with little change until above 250°C, at which point some cavities grew in size rapidly.

In coarse-grained nickel, large octahedral and/or cuboidal cavities are formed during high temperature

neutron [26,27] and ion [28–30] irradiation, and the cavities maintain a faceted structure during annealing. In contrast, annealing of cavities in nanocrystalline nickel at 400°C resulted in large oblong-shaped cavities, such as that circled in Figure 2(c). Video recording of the film at 400°C revealed that, at least in part, the growth of the cavities occurred via coalescence (see supplemental videos). In the video, cavities as large as 20 nm in diameter are mobile and seen to coalesce with larger cavities to create large, less symmetric-shaped cavities. Coalescence is also seen to occur by a mechanism in which only one side of the cavity expands to combine with nearby cavities, resulting in the large oblong-shaped cavity circled in Figure 2(c).

Following annealing, the grain structure was no longer easily identified in Figure 2(c) due to the large density of cavities. However, selected area diffraction

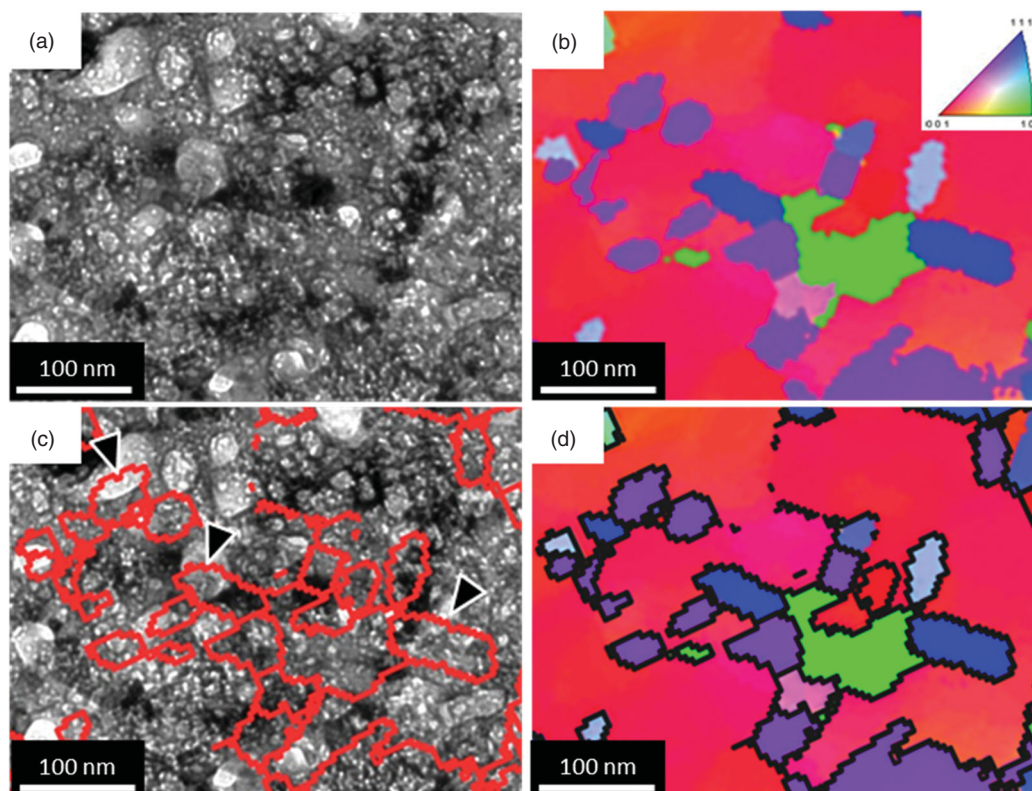


Figure 3. (a) Under-focus BF image of cavities in helium implanted then self-ion irradiated nanocrystalline nickel film after annealing to 400°C. (b) Orientation map of (a) revealing the grain structure and strong {100} texture, inverse pole figure color chart is inset. (c) Grain boundaries (red) overlaid on Figure 3(a) demonstrating that many cavities cross grain boundaries, several examples of which are highlighted with black arrows. (d) Orientation map with grain boundaries emphasized in black.

demonstrates that the nanocrystalline structure with a strong {100} texture is preserved, as illustrated in the inset of Figure 2(c). To further characterize the post annealing grain structure, ASTAR orientation was performed on the region shown in the BF-TEM image in Figure 3(a). Because the film had already been annealed, it was assumed that no HCP grains were present, and all grains were indexed as FCC.[18] Orientation information for this region shown in Figure 3(b) revealed that the {100} grain texture and nanocrystalline grains structure were preserved after the self-ion irradiation, He⁺ implantation, and annealing process, consistent with the diffraction pattern in Figure 2(c). Surprisingly, the grain structure can be indexed with a relatively high level of reliability even in areas with large cavities. The information on grain boundary location provided by the ASTAR orientating mapping is overlaid on the BF-TEM image and orientation map in Figure 3(c) and 3(d), respectively. The grain diameters identified by the orientation map ranged from approximately 15–150 nm with a small number of grains surpassing 200 nm, similar to the initial grain size distribution (see grain size histograms in supplemental Figure 5). Many cavities grew as large as or larger than the grains themselves, without causing significant grain growth. In addition, arrows in Figure 3(c)

show regions in which the large cavities are observed to pass through grain boundaries. Because the *in situ* TEM annealing and orientation map were not performed sequentially on the same area, the growth dynamics could not be directly correlated with the final structure. However, the final structure and *in situ* TEM observations independently suggest that the grain boundaries and the defects present on them after He⁺ implantation and self-ion irradiation did not significantly stabilize the cavities on the grain boundary under the thermal and radiation environments presented in this study. Instead, the cavities resemble the size of large faceted cavities experimentally characterized in ion-irradiated large grained nickel at elevated temperature.[31,32] These results suggest that similar macroscopic swelling may be expected in this system. It is hypothesized that this is due to the relatively low angle of grain boundaries present in this film and the large amount of vacancies present in the film due to the PLD process, the Frenkel pairs created during He⁺ implantation, and the cascade damage from self-ion irradiation. In contrast to other nanostructured metals,[1–5] these results suggest that single component nanocrystalline metals dominated by low-angle grain boundaries will not provide significant radiation tolerance compared to large grained systems.

In this letter, sequential *in situ* 3 MeV self-ion irradiation and 10 keV helium implantation were performed, in forward and reverse orders, on nanocrystalline PLD nickel films in a TEM. The order of the irradiation and implantation was seen to have a significant impact on the distribution of the resulting cavity structure. When the films were first implanted with helium, the room temperature microstructure after subsequent self-ion irradiation showed an increased density of cavities at the grain boundaries. In contrast, the samples that were first self-ion irradiated and subsequently implanted with helium resulted in an even distribution of cavities through the grain structure. It is thought that the difference in cavity structure is due to the fact that self-irradiation provides additional sink locations for the helium to cluster uniformly without having to migrate to the grain boundaries or surfaces. In addition, the *in situ* TEM annealing of the cavities resulted in large cavities with diameters on the order of the grain size and film thickness. Orientation mapping revealed that the nanocrystalline structure was largely maintained up to 400°C and many cavities spanned multiple grains. The grain boundaries did not limit cavity growth, suggesting that PLD nanocrystalline nickel, which is dominated by low-angle grain boundaries, will not provide significant radiation tolerance compared to large grained systems. These results provide a greater insight into the active mechanisms governing the evolution of radiation damage in nanocrystalline nickel and other nanostructured metal systems.

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Supplementary online material A more detailed information on experiments is available at <http://dx.doi.org/10.1080/21663831.2015.1121165>.

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