In situ study of defect migration kinetics in nanoporous Ag with enhanced radiation tolerance

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Defect sinks, such as grain boundaries and phase boundaries, have been widely accepted to improve the irradiation resistance of metallic materials. However, free surface, an ideal defect sink, has received little attention in bulk materials as surface-to-volume ratio is typically low. Here by using in situ Kr ion irradiation technique in a transmission electron microscope, we show that nanoporous (NP) Ag has enhanced radiation tolerance. Besides direct evidence of free surface induced frequent removal of various types of defect clusters, we determined, for the first time, the global and instantaneous diffusivity of defect clusters in both coarse-grained (CG) and NP Ag. Opposite to conventional wisdom, both types of diffusivities are lower in NP Ag. Such a surprise is largely related to the reduced interaction energy between isolated defect clusters in NP Ag. Determination of kinetics of defect clusters is essential to understand and model their migration and clustering in irradiated materials.

The successful development of advanced nuclear reactors calls for the discovery of advanced materials that can endure unprecedented neutron irradiation damage to hundreds of displacements-per-atom (dpa)1–3. A high density of irradiation-induced defect clusters, including dislocation loops and networks, voids, bubbles and stacking fault tetrahedra (SFTs), can significantly degrade mechanical properties of materials4–7. Several types of defect sinks have been explored to achieve enhanced radiation tolerance, such as high-angle grain boundaries (GBs)8–13, immiscible interfaces in nanolayer composites14–17, twin boundaries18,19 and phase boundaries20,21. Metallic nanoporous (NP) materials with large surface-to-volume ratios have applications for energy storage, catalysts, filters and gas sensors22. Their mechanical, catalytic and optical properties have been widely investigated23–25. Lee et al.26 reported mechanical strength of NP Au and suggested that NP metals could be used as high strength, low density materials. Kucheyev et al.27 concluded that the pronounced time-dependent creep of NP silica at room temperature was attributed to the stress corrosion fracture of nanoscale ligaments. The impact of free surface on irradiation-induced damage in bulk materials has also been studied. In general a larger number of defect clusters (mostly vacancy loops) were observed near-surface compared to those in materials interior (inside bulk materials)28–31. Such disparity in defect distribution has been explained by preferential removal of mobile interstitials by free surfaces, leaving less mobile vacancy clusters behind (near surfaces)29. MD simulations also observed viscous flow of atoms to free surfaces32. Norris et al.33 reported void denuded zones near the free surface of Ni foil under high voltage electron beam irradiation. Bringa et al.34 predicted via MD simulations that, at a given dose rate, the Au foam would be resistant to radiation if the ligament diameter is within an optimum window. The ligament, if too small (several nm) would melt and break during radiation, and, if greater than 100 nm, would accumulate damage rapidly, similar to bulk materials. Using ex situ Ne ion irradiation, Fu et al.35 showed the formation of SFTs in porous Au is dose rate dependent.

Despite prior studies on surface sinks, several significant issues remain unaddressed. First, there is no in situ evidence to unambiguously reveal defect removal and accumulation mechanisms via surfaces in NP metals. Second, defect migration kinetics, or diffusivity of nanoscale defect clusters in porous as well as conventional metals remains largely unknown36–38. Matsukawa and Zinkle36 reported a maximum instantaneous diffusivity of ~3000 nm²/s of vacancy clusters (3 nm in diameter) in Au during 1D migration along a dislocation line at ~113 K. Arakawa et al.37 showed global diffusivity (considering dwell time) of isolated dislocation loops (6 nm in
dramatically increased at low dose and reached saturation, studies show that defect cluster density in CG Ag (Fig. 2a) finally by 1.5 dpa, the average defect cluster size in CG Ag appeared only a handful of activities for defect clusters were captured in NP Ag. In situ ex situ few defect clusters were observed in the ligaments of NP Ag. the size and density of defect clusters in CG Ag (Fig. 1c). In parallel few defect clusters were observed in the ligaments of NP Ag. By 1 dpa, in situ radiation (see supplementary Video 3) shows abundant defect migration activities in irradiated CG Ag, whereas only a handful of activities for defect clusters were captured in NP Ag. Finally by 1.5 dpa, the average defect cluster size in CG Ag appeared much greater than that in NP Ag (Fig. 1d and Fig. 1d’). Statistical studies show that defect cluster density in CG Ag (Fig. 2a) dramatically increased at low dose and reached saturation, ~2 × 10^22 m^-2 at ~0.25 dpa, while NP Ag approached a 50% lower saturation defect density, 1 × 10^22 m^-2 at 0.5 dpa. Meanwhile the average size of defect clusters in NP Ag shown in Fig. 2b reached a saturation value of ~3.5 nm, much lower than that in CG Ag, ~8 nm. Statistical studies in Fig. 2c–d show that after irradiation to 1.5 dpa, both the average and maximum sizes of defect clusters in NP Ag were much smaller than those in CG Ag.

For validation purposes, Kr ion irradiations at 800 keV at room temperature were performed ex situ on CG Ag TEM thin foils with nearly no preexisting defect clusters. SRIM simulations (Suppl. Fig. S2) show the radiation damage level in Ag within the depth of the first 100 nm is very similar between the two irradiation conditions (ex situ 800 keV vs. in situ 1 MeV Kr ions). The microstructural evolution with irradiation doses up to 1 dpa (as shown in suppl. Fig. S3) suggests ex situ irradiated CG Ag exhibited nearly identical defect density compared to the in situ irradiated CG Ag at the same dose level. Bright field and weak beam dark field (WBDF) images (suppl. Video 1 (0–0.014 dpa). By 0.02 dpa, the CG Ag was already swamped with a large number of defect clusters (Fig. 1b), whereas no detectable defect clusters formed in NP Ag (see supplementary Video 3 at 0.038 dpa)). Up to 0.25 dpa, there was a significant increase in both the size and density of defect clusters in NP Ag (Fig. 1a). In comparison, NP Ag was basically free from obvious defect clusters prior to radiation (Fig. 1a’). In situ videos show that within the first 5–10 sec, defect density in CG Ag increased rapidly, whereas no detectable defect clusters formed in NP Ag (see supplementary Video 1 (0–0.014 dpa)). By 0.02 dpa, the CG Ag was already swamped with a large number of defect clusters (Fig. 1b), whereas NP Ag remained intact (Fig. 1a). After irradiation to 0.02 dpa, CG Ag was swamped with a large number of defect clusters, whereas NP Ag remained intact (see Supplementary Video 2). (c–c’) By 0.25 dpa, there was a significant increase in both the size and density of defect clusters in CG Ag. In parallel few defect clusters were observed in the ligaments of NP Ag. (d–d’) By 1.5 dpa, the average defect cluster size in CG Ag appeared much greater than that in NP Ag.

Fig. S4) of irradiated CG Ag (at 1 dpa) show that a majority of defect clusters were dislocation loops and SFTs.

In situ evidence of defect cluster removal in NP Ag. We now present several typical examples in NP Ag where defect clusters were absorbed by defect sinks under irradiation (see supplementary Video 4 for details). First we report the removal of individual dislocation loops by free surface (Fig. 3a–c). A radiation induced dislocation loop (~4 nm in diameter) was located at 7.5 nm from free surface (Fig. 3a). By 2 s, the loop migrated 1.8 nm towards the free surface (Fig. 3b). The SFT remained stable till 7 s (Fig. 3c). However by 9 s, the SFT was gradually removed by the free surface (Fig. 3f). Third, in situ video snap shots (Fig. 3g–i) captured formation and rapid absorption of a dislocation segment. Several

![Figure 1](https://example.com/figure1.png)
individual dislocation loops observed in ligament (Fig. 3g) at 15 s combined to form a dislocation segment, which was \(\sim 4.2 \text{ nm}\) from free surface (Fig. 3h). Within merely 0.1 second, the dislocation segment was instantly absorbed by the free surface (Fig. 3i). Finally we look into the absorption of a discrete dislocation loop by triple junctions (TJs) in NP Ag (Fig. 3j–l). A dislocation loop outlined by a dash circle formed near a GB at 46 s (Fig. 3j). The dislocation loop rapidly migrated towards the GB (within 0.1 s) and then diffused along the GB towards a TJ for 0.6 s (Fig. 3k), and eventually was fully absorbed by the TJ (Fig. 3l).

**Drastic difference in defect migration kinetics between CG and NP Ag.** Besides vivid examination of various types of defect removal mechanisms, the *in situ* radiation study also provides abundant information to investigate defect migration kinetics in irradiated Ag. In particular we were able to determine global and instantaneous diffusivity of defect clusters. The global diffusivity \(D_g\) is the diffusivity averaged over a long period of time (including migration and dwell time) for numerous defect clusters, whereas instantaneous diffusivity \(D_i\) is measured only during the migration process. Fig. 4a shows typical examples of measured migration distance (diffusion length \(X\)) of individual defect clusters (4 nm in diameter) in both CG and NP Ag. In order to determine the global diffusivity of defect clusters, the migration of a large number of defect clusters was studied statistically. Fig. 4b shows diffusion length square \(X^2\) vs. accumulative time for numerous defect clusters with similar size (4 nm in diameter) in both CG and NP Ag. Assuming one dimensional diffusion, the diffusivity of defect clusters \(D\) can be estimated by \(D = X^2/2t\), where \(t\) is the diffusion time. Although there is error on the \(X^2\) measurement for both CG and NP Ag, a linear fit of \(X^2\) vs. accumulative time for numerous defect clusters with 0.25 dpa at twice higher value, 2 \(\times 10^{-23}/\text{m}^3\). (b) At increasing dose, the average size of defect clusters in CG Ag increased monotonically to \(\sim 8 \text{ nm}\), whereas the defect cluster size in NP Ag remained small, \(\sim 3.5 \text{ nm}\) by 0.5 dpa. (c–d) Statistical size distributions of defect clusters (by 1.5 dpa) show that CG Ag has both greater average and maximum defect size, \(8\) and \(13 \text{ nm}\) respectively, comparing to \(3.5 \text{ nm}\) and \(6 \text{ nm}\) in irradiated NP Ag.

**Discussion**

NP Ag has excellent radiation resistance as it contains enormous free surface that removes point defects and defect clusters and consequently significantly reduces the density and size of defect clusters in NP Ag compared with CG Ag. The absorption of point defects by free surface appears to result in defect concentration gradient and several nm wide surface-affected-zones (SAZs). The continuous flux of point defects towards free surfaces leads to drainage of overall point defect concentration in interior of NP Ag. Thus the formation and growth of defect clusters internally are significantly retarded.

Mechanisms for removal of SFTs, individual dislocation loop and dislocation segment by free surface might be different. (1) SFTs are highly stable defects in irradiated fcc metals with low stacking fault energy, and their removal typically requires high temperature annealing or mobile dislocations. SFTs within SAZs in NP Ag could be removed via numerous mechanisms. One of them could be accelerated migration of mobile SIAs in SAZ. These SIAs could interact with SFTs as observed during *in situ* irradiation. (2) The image force on discrete dislocation loops adjacent to free surface provides driving force for its migration towards free surface. The image force arises from a virtual dislocation required to satisfy the free surface criteria (or balance the force induced by a real dislocation near free surface).

![Figure 2](https://www.nature.com/scientificreports/)
defect cluster density is negligible, consistent with previous study on 
SFT defect sinks, such as free surfaces. Rapid exit of interstitials and 
to support homogeneous cluster nucleation and growth next to pla-

By calculating image force on an edge dislocation without losing 
the speed of CCD camera (15 frame/s). The lack of size depend-
and SFTs. We also determined global and instantaneous diffusion of 
defect clusters in both systems. The observed much lower diffusivity 
defect clusters in irradiated NP Ag than that in CG Ag, though 
counterintuitive at its first glance, is in fact consistent with enhanced 
radiation tolerance of NP Ag. Experimental determination of defect 
migration kinetics via in situ radiation provides crucial information 
to understand defect accumulation in irradiated metallic materials 
and could drastically improve the reliability of a variety of modeling 
Studying on radiation damage.

Methods

Figure 3 | In situ video snap shots revealing several representative defect 
capture events by free surface or triple junctions (TJs) over 1.18– 
1.27 dpa. (See supplementary video 4 for details). (a–c) Evidence of rapid 
absorption of individual dislocation loops by free surface. A loop, 
~7.5 nm away from free surface, migrated leftwards in 2 s, and was 
immediately removed by the free surface by 2.1 s. (d–f) A stacking fault 
tetrahedron (SFT) was gradually removed by the free surface from 7 to 9 s. 
(g–i) Formation of a dislocation segment and its rapid absorption by 
free surface. At 15 s, several individual dislocation loops were in contact with 
one another. The loops then combined to form a dislocation segment in 2 s 
at ~4.2 nm from free surface. Within 0.1 s, the dislocation segment was 
completely absorbed by the free surface. (j–l) Absorption of a dislocation 
loop by a TJ in NP Ag. At 46 s, a dislocation loop was generated near a grain 
boundary (GB). After 5 s, the loop was rapidly attracted towards the GB 
within 0.1 s, and the loop migrated along the GB for 0.6 s before being 
captured by the TJ.

By calculating image force on an edge dislocation without losing 
general applicability (see supplementary information for details of 
calculation), we estimate the vacancy formation energy near SAZ is 
~0.04 eV. (3) In general there is insufficient defect supersaturation to 
support homogeneous cluster nucleation and growth next to plan-
lar defect sinks, such as free surfaces. Rapid exit of interstitials and 
vacancies in certain cases towards free surface leads to SAZ, where 
defect cluster density is negligible, consistent with previous study on 
void denuded zones close to surface of Ni foils45. Correspondingly, 
the density of defect clusters in NP Ag saturated at greater dose, 
~0.5 dpa, compared to that in CG Ag, ~0.25 dpa. (4) Several indi-
vidual dislocation loops in SAZ form a segment instead of being 
absorbed separately by free surface. The dislocation loops may have 
mutually attractive interaction forces (to be discussed in the next 
section), which balance with the image force from free surface. The 
combination of discrete dislocation loops to a single segment might
For Ar ion milling, which was performed at 3.7 keV at an incident angle of 5°.

Low energy (2 keV) ion polishing was applied to remove ion milling induced damage in Ag.

In situ and ex situ Kr ion irradiation. In situ Kr ion irradiation at 1 MeV was performed for CG and NP Ag at room temperature in the Intermediate Voltage Electron Microscope (IVEM) at Argonne National Laboratory, where an ion accelerator was attached to a HITACHI H-9000NAR microscope. The microscope was operated at 200 kV and kept on during radiation in order to record the microstructural evolution. The average dose rate was about $1.8 \times 10^2$ dpa/s. A CCD camera was used to capture microstructural evolution during radiation at 15 frame/second. SRIM simulation was used to estimate the displacement damage profile and Kr ion distribution. To check reproducibility and validate in situ radiation studies, ex situ Kr ion irradiation was performed at 800 keV at Ion Beam Materials Laboratory at Los Alamos National Laboratory. SRIM simulations (see supplementary Fig. S2) show that most of the Kr ions at 1 MeV and 800 keV will penetrate through the first 100 nm (the thickness of TEM foil is 100 nm), whereas displacement damage will be contained primarily in the TEM foil; and the magnitude of depth dependent dose profile applied at both energy was controlled to be similar to each other.

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**Figure 4** Determination of global and instantaneous diffusivity in Kr ion irradiated CG and NP Ag at a dose of $-1$ dpa. (a) Representative plots of diffusion length (X) versus accumulative time for individual defect clusters with a diameter of 4 nm in CG and NP Ag. Defect clusters migrate in a similar “stick-jump” jerky pattern. (b) Plots of diffusion length square ($X^2$) vs. accumulative time for a large number of defect clusters with a similar average size of 4 nm. The average global diffusivity was estimated to be $-78$ and 12 nm²/s in CG and NP Ag, respectively. (c) The global diffusivity ($D_g$) in both CG and NP Ag reduces monotonically with increasing defect cluster size. For nearly identical defect cluster size, the value of $D_g$ of NP Ag is consistently lower than its CG counterpart. (d) However instantaneous diffusivity ($D_i$) for both systems has no defect size dependence. The average value of $D_i$ in CG and NP Ag was estimated to be 1200 and 350 nm²/s, respectively.

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**Figure 5** Schematic illustration on migration of defect clusters in CG and NP Ag. (a) In CG Ag, the average loop size and separation distance ($L_s$) are 8 and 17 nm. The yellow zone outlines the region in which interactive stress field between the newly generated small defect clusters and preexisting large defect clusters is significant. As the stress field of these closely spaced large defect clusters tends to overlap, there is a greater probability to capture fresh radiation induced small defect clusters (indicated by open circles). (b) In NP Ag with ligament size ($D_l$) of 40 nm, $L_s$ among defect clusters (3 nm in diameter) is $\sim 22$ nm due to the lower defect cluster density. The width of the surface-affected-zones (SAZs) is $\sim 5$ nm. Radiation induced fresh small defect clusters can be removed by free surface (if they are located within SAZs). Compared with CG Ag, the preexisting defect clusters are much smaller, and have rather limited stress field (indicated by a narrower yellow zone). Furthermore these defect clusters are also more isolated, and consequently the possibility of capturing fresh radiation induced loop by preexisting loop is much less than in CG Ag.

**In situ and ex situ Kr ion irradiation.** In situ Kr ion irradiation at 1 MeV was performed for CG and NP Ag at room temperature in the Intermediate Voltage Electron Microscope (IVEM) at Argonne National Laboratory, where an ion accelerator was attached to a HITACHI H-9000NAR microscope. The microscope was operated at 200 kV and kept on during radiation in order to record the microstructural evolution. The average dose rate was about $1.8 \times 10^2$ dpa/s. A CCD camera was used to capture microstructural evolution during radiation at 15 frame/second. SRIM simulation was used to estimate the displacement damage profile and Kr ion distribution. To check reproducibility and validate in situ radiation studies, ex situ Kr ion irradiation was performed at 800 keV at Ion Beam Materials Laboratory at Los Alamos National Laboratory. SRIM simulations (see supplementary Fig. S2) show that most of the Kr ions at 1 MeV and 800 keV will penetrate through the first 100 nm (the thickness of TEM foil is $\sim 100$ nm), whereas displacement damage will be contained primarily in the TEM foil; and the magnitude of depth dependent dose profile applied at both energy was controlled to be similar to each other.
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Author contributions

C.S. and D.B. prepared specimens for in situ Kr ion irradiation; C.S. and Y.C. performed the in situ Kr ion irradiation experiments with assistance from M.K. and M.L. at Argonne National Laboratory; Y.Q.W. and C.S. performed ex situ Kr ion irradiation experiments at Los Alamos National Laboratory, X.Z., H.W. and S.A.M. assisted with interpreting the data. X.Z. developed the concept and directed the project. All authors discussed the results and commented on the manuscript.

Additional information

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