Radiation induced detwinning in nanotwinned Cu

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Abstract

Superior radiation tolerance has been experimentally examined in nanotwinned metals. The stability of nanotwinned structure under radiation is the key factor for advancing the application of nanotwinned metals for nuclear reactors. We thus performed *in situ* radiation tests for nanotwinned Cu with various twin thicknesses inside a transmission electron microscope. We found that there is a critical twin thickness (10 nm), below which, radiation induced detwinning is primarily accomplished through migration of incoherent twin boundaries, in particular, detwinning is faster for thinner twins in this range, while above which, twins are stable.

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Irradiation of metals results in abundant point defects [1-3] and their clusters [4-9], which cause severe void swelling and mechanical deterioration of materials [10-12]. Interfaces, such as grain boundaries (GBs) [13-17], heterophase interfaces [18-20] and free surfaces [21-23], have proven to be effective in alleviating radiation damage. Nanograined materials gather intense interest as a result of drastic enhancement of radiation tolerance shown both experimentally [15, 17] and theoretically [14, 24-27]. However, nanograins tend to coarsen rapidly at elevated temperature or under irradiation [28, 29], compromising the radiation tolerance of nanocrystalline materials. In contrast to nanograins, nanotwinned (nt) metallic materials with predominantly TBs have outstanding microstructural stability under both radiation [30] and annealing conditions [31, 32], as coherent twin boundaries (CTBs) are thermodynamically stable at elevated temperatures.

Recent studies show that CTBs can serve as defect sinks to reduce radiation damage in nt metals [33, 34]. *In situ* radiation studies and molecular dynamics (MD) simulations show that CTBs can effectively interact with radiation induced stacking fault tetrahedra (SFTs), resulting in the removal of SFTs in irradiated fcc metals [33-36]. Furthermore, deliberate introduction of nanovoids in conjunction with nanotwins establishes a network of defect sinks in nt metals. *In situ* TEM observation and atomistic simulations demonstrate that TBs can rapidly transport radiation induced defects to nanovoids, where defects are stored and annihilated, leading to significant enhancement of radiation tolerance in nt metals [37, 38]. The stability of twins under radiation is thus one key factor in retaining the high radiation tolerance in nt metals. However, does radiation induce detwinning in nt metals?

Detwinning under mechanical deformation has been frequently observed in metals [33, 39-42] and the deformation induced detwinning mechanisms can be categorized to at least two

cases. The first case is characterized by the migration of steps/dislocation disconnections at CTBs, where the steps derive from interaction between CTBs and inclined dislocations [43-48]. The second case is associated with the collective glide of Shockley partial dislocations at $\Sigma 3$ {112} incoherent twin boundary (ITB) [49, 50]. It is anticipated that radiation induced detwinning could be accomplished through different mechanisms from deformation induced detwinning because the internal stress arisen from radiation process is not as significant as the case of mechanical deformation. In this paper, we studied the radiation response of epitaxial nt Cu with various twin thicknesses by using *in situ* Kr ion irradiation technique inside a transmission electron microscope. The migration of both CTBs and ITBs was investigated and the radiation induced detwinning mechanisms are discussed.

Epitaxial nt Cu films were deposited on HF etched Si (110) substrates using the magnetron sputtering technique at room temperature. The total film thickness was $\sim 1 \mu\text{m}$ for nt Cu. The deposition rate was $\sim 0.5 \text{ nm/s}$. Cross-sectional transmission electron microscopy (XTEM) samples were prepared by dimpling and low energy (3.5 keV) Ar ion milling and subsequent ion polishing. *In situ* Kr⁺⁺ ion irradiation at 1 MeV was performed for nt Cu 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 transmission electron microscope. The microscope was operated at 200 kV continuously during radiation in order to record the microstructural evolution. The average dose rate was $0.43\text{-}1.98 \times 10^{-3} \text{ dpa s}^{-1}$. A CCD camera was used to capture microstructural evolution during radiation at 15 frame s^{-1} . More details are referred to [34].

Detwinning occurs via migration of CTBs and ITBs during radiation. Fig. 1 compares snap shots of nt Cu subjected to *in situ* Kr ion irradiation of up to 1.56 dpa to illustrate the ITB

migration. The ITBs of thinner twins have higher tendency to migrate than those of thicker ones. As shown in Fig. 1, during radiation, the migration of ITBs occurs for the thinner twins (twin 1 and 3), whereas no significant ITB migration was observed for the thicker twin 2. The average twin spacing slightly increased from 13 nm (at 0.01 dpa) to 15 nm after irradiation to 1.56 dpa, showing that fine twins have been eliminated.

One should note that in this study the definition of twins and matrices is relative to each other as the volume fraction of twins is very close to that of the matrix and the average twin thickness and matrix thickness is similar, ~ 13 nm. Irradiation of nt Cu also induced a high density of stacking faults (SFs) decorating along the TBs. TEM micrographs in Fig. 2(a-b) compare the positions of CTBs at the same location of irradiated nt Cu before and after radiation to 1.56 dpa. The phase with bright contrast is defined as matrices and the dark regions as twins. Statistical studies (Fig. 2(c-d)) show the radiation induced the increase of the twin thickness and corresponding decrease in the matrix thickness, due to the migration of CTBs along its normal direction. Overall the thickness variation of twins and matrices (or the magnitude of CTB migration) shows little dependence on the original twin thickness. The thickness variations for both twins and matrices are close, ~ 3 nm up to 1.56 dpa, and therefore the CTB migration speed is estimated to be ~ 1 nm/dpa.

The prominent migration of thin twins leads to two interesting questions: How does ITB migrate? is there a quantitative relation between the migration of ITBs and twin thicknesses? Extensive studies have been performed to investigate migration of ITBs by analyzing the *in situ* radiation videos. A typical example shown in Fig. 3a captures the migration of four ITBs (T1-T4) with respective twin thicknesses of 2.8, 1.4, 5.2 and 6.4 nm. The migration traces of the four ITBs are shown in Fig. 3b. It is evident that ITBs migrate in a “stick-slip” fashion, i.e., the

migration processes consist of instantaneous movement and dwelling. Fig. 3b also shows that instantaneous migration speed could be as high as 21 nm/s. Fig. 4 shows ITB migration distances as a function of radiation dose with respect to twins with various thicknesses in the range of 2.8 - 23 nm. When twin thickness is below a critical value (~ 10 nm), the velocity of ITB migration (described in unit of nm/dpa) decreases progressively with the increasing in twin thickness (Fig. 4b). Above the critical thickness, detwinning is not obviously observed.

Previous studies show that TBs in nt metals can actively engage and absorb radiation induced defect clusters, including dislocation loop and vacancy clusters [36, 37]. Our current study shows that both CTBs and ITBs not only interact with defects, but also migrate. In other words, the defect sinks (TBs) are mobile. CTBs migrate along $\langle 111 \rangle$ at a velocity of ~ 1 nm/dpa and its migration velocity is independent of twin thickness. This is different from detwinning associated with the migration of ITBs. In contrast, ITBs migrate along $\langle 112 \rangle$ at a much faster velocity, varying from several to 60 nm/dpa. The migration of ITBs leads to detwinning. Two potential mechanisms may contribute to the migration of CTBs. First, radiation induced defects, such as dislocation loops and SFTs, accumulate and interact with CTBs, forming stacking fault ribbons at CTBs [33, 34]. Second, steps/dislocation disconnections at CTBs that formed due to the interaction of CTBs and inclined dislocations could migrate along the direction normal to CTBs.

The high migration velocity of ITBs with small twin thickness (t) is related to their defect structures. $\Sigma 3 \{112\}$ ITB can be represented as an array of Shockley partial dislocations on each $\{111\}$ plane, containing three repetitive partial dislocations: $\mathbf{b}_1 = 1/6[11\bar{2}]$, $\mathbf{b}_2 = 1/6[\bar{2}11]$ and $\mathbf{b}_3 = 1/6[1\bar{2}1]$, where $\mathbf{b}_1$ is an edge partial dislocation and $\mathbf{b}_2$ and $\mathbf{b}_3$ are mixed partial dislocations with screw components of opposite sign. Without radiation, the ITBs with the thicknesses of 2.8

- 23 nm are stable at room temperature, as demonstrated in our previous study [49] in Cu that ITBs are stable when the twin thickness is larger than 1.9 nm. Energetically ITBs tend to migrate to reduce the area of CTBs (and consequent energy stored in CTBs) with the driving force F ($=2\gamma_T$, where γ_T is excess energy of CTB). We note that friction force (F_p , or Peierls barrier), for the migration of ITBs increases with the twin thickness, as partial dislocations in the ITB migrate collectively [49]. When the driving force is larger than the friction stress ($F > F_p$), ITB could migrate to reduce the area of CTBs (case 1 in Fig. 4b); when $F < F_p$, ITB would not migrate. Fig. 4b shows that when $t > 10$ nm, ITBs barely migrate, suggesting that the critical thickness (t^*) for ITB migration is ~ 10 nm. When $t > t^*$, detwinning could begin from the corner (case 2 in Fig. 4b). The driving force for ITB migration in case 2 is negligible as the detwinning does not change the total area of CTBs (does not reduce the energy stored at TBs). A further increase of twin thickness would make segment of ITBs to migrate randomly (case 3 in Fig. 4b), that is in this case both twinning and detwinning may occur, but both process would increase the area of CTBs. The direction of ITB migration is random as such is mainly determined by both radiation induced local shear stress [49] and the density and type of radiation induced defects across the ITBs [51].

In contrast, when $t < t^*$, the migration velocity of TBs decreases with the increase of twin thickness, which is rationalized as follows. During radiation, the driving force on individual Shockley partial dislocation in the ITB can be treated as $F_1 = F/L = 2\gamma_T/L$. (L is the number of atomic layers of the twin, $L = t/d_{111}$, d_{111} is the interplanar spacing of $\{111\}$ planes). For fcc and hcp metals, the dislocation velocity and applied stress follows $v = A\tau^m$ [52], where A is a material constant, m is approximately 1 at 300 K in pure metals. Therefore, the velocity of ITB migration can be rationalized as $v = A\tau = \frac{AF_1}{b} = B/t$ (b is the magnitude of Burgers vector of

partial dislocation) which qualitatively explains the trend that the migration velocity decreases with the increase of twin thickness when $t < t^*$.

It is noted that there is scattering for data points, particularly for ITBs with small thicknesses. One possibility is the variation of the TEM foil thickness across the wedge sample. The driving force discussed previously is evaluated thermodynamically. During *in situ* radiation, a kinetic process is also involved, that is the migration of different segments on a dislocation line may not occur simultaneously. ITBs may migrate at a slower speed when the TEM foil becomes thicker, causing the scattering of data in Fig. 4b.

It is worth pointing out that ITB migration velocity also depends on stacking fault energy (SFE) [49]. Our current study only discussed detwinning in Cu with low SFE. For ITBs in metal with high SFE, the driving force ($2\gamma_T/bL$) for ITB migration is large. However, the Peierls barrier associated with the three TDs in metal with high SFE is also high as high SFE prevents the dissociation of the three TDs at ITBs [53]. Therefore, future experiments are required to examine the ITB response under radiation in metal with high SFE.

In summary, detwinning occurs in irradiated nanotwinned Cu via migration of both ITB and CTB during radiation. In particular, the migration speed of CTBs is sluggish, ~ 1 nm/dpa, independent of twin thickness. In comparison, ITBs migrate aggressively at a much greater speed when the twin thickness is below 10 nm. The migration speed of ITBs decreases progressively with increasing twin thickness, while ITBs with large twin thickness are stable during radiation. The ITB migration consists of instantaneous movement and dwelling during radiation. Having mobile ITBs in irradiated metallic materials could be beneficial to enhance radiation tolerance, as these mobile defect sinks can have a larger territory to capture, trap and eliminate radiation

induced defects. However it is also important to retain a high density of ITBs so that these defects sinks can operate over a long period of time to eliminate radiation induced defects.

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Figure captions

Fig.1. (a-d) TEM studies on migration of incoherent twin boundaries (ITBs) in nanotwinned Cu subjected to *in situ* Kr ion irradiation of up to 1.56 dpa. Three typical ITBs are labeled with respective twin thickness of 6, 23 and 8 nm in (a). During radiation, ITB migration occurs for the thinner twins (twin 1 and 3), whereas no significant ITB migration was observed for the thicker twin 2. (e-f) Comparison of the distribution of twin spacings shows that the average twin spacing slightly increased from 13 nm (at 0.01 dpa) to 15 nm after irradiation to 1.56 dpa.

Fig. 2. *In situ* study on radiation induced CTB migration. (a-b) TEM snap shots document the positions of CTBs at the same location before and after radiation to 1.56 dpa. The bright and dark areas correspond to the matrix and twin, respectively. (c-d) Statistical studies show that radiation induced the increase of the twin thickness and decrease in the matrix thickness, due to migration of CTB along its normal direction. Overall the thickness variation of twins and matrices (or the magnitude of CTB migration) shows little dependence on the original twin thickness. The thickness variations for both twins and matrices are close, ~ 3 nm up to 1.56 dpa, leading to an average CTB migration speed of ~ 1 nm/dpa.

Fig. 3. *In situ* TEM snap shots showing the migration of ITBs consisting of instantaneous ITB movement and dwelling during radiation. Four ITBs (T1-T4) with respective thicknesses of 2.8, 1.4, 5.2 and 6.4 nm were selected to track their migration processes. At 0.07 s, the tip of T1 became blunted. At 0.14 s, T1 migrated rightward by 1 nm. At 0.21 s and 0.28 s, T1 migrated by another 0.5 and 1.5 nm (1.5 nm and 3 nm away from initial position). T1 then stalled afterwards. Meanwhile T2 migrated over 1 nm from 0.21 to 0.28 s, while T3 and T4 did not migrate. (b) Plots of ITB migration distance vs. accumulative time for the four ITBs show that stick-slip type of ITB migrations consisting of instantaneous movement and dwelling of the ITBs. Instantaneous migration speed could be as high as 21 nm/s.

Fig. 4. The dependence of ITB migration process on twin thickness. (a) The plot of ITB migration distance over radiation dose for numerous twins with twin thickness of 2.8 - 23 nm shows that in general the thinner ITBs have greater migration distance. (b) The velocity of ITBs in unit of nm/dpa decreases progressively with the increasing twin thickness. The critical film thickness, t^* , is ~ 10 nm. For case 1 ($t < t^*$), detwinning reduces the area of CTBs and thinner twins appear to migrate at a greater velocity. For case 2, when t is moderately greater than 10 nm, detwinning doesn't change the area of CTBs, and hence TB migration is less likely; In case 3, both twinning and detwinning would increase the area of CTBs, reducing the likely for ITB migration.

Figure1

Figure2

(a) 0 dpa

(b) 1.56 dpa

Figure3

(a) Instantaneous ITB migration

Figure4-R

(a) 0 dpa

(b) 1.56 dpa