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Dynamic Void Growth and Shrinkage in Mg under Electron Irradiation

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We report *in situ* atomic-scale investigation of late-stage void evolution, including growth, coalescence and shrinkage, under electron irradiation. With increasing irradiation dose, the total volume of voids increased linearly, while the nucleation rate of new voids decreased slightly and the total number of voids decreased. Some voids continued to grow while others shrank to disappear, depending on the nature of their interactions with nearby self-interstitial loops. For the first time, surface diffusion of adatoms was observed to be largely responsible for the void coalescence and thickening. These findings provide fundamental understanding to help with the design and modeling of irradiation-resistant materials.

Keywords: Void, Coalescence, Growth, Electron Irradiation, Magnesium

The irradiation resistance of a material determines its performance and service life in many applications such as nuclear energy, [1–5] outer space systems [6] or other industrial applications.[7,8] Radiation produces crystalline and microstructural defects in materials, which causes degradation of their properties. Extensive effort has been taken to investigate the evolution of irradiation-induced defects, including dislocations, stacking fault tetrahedrons, voids, element segregation and precipitation in metals and ceramics.[9–27] Particularly, void formation has attracted extensive attention since it may cause volumetric swelling and eventual material failure.

Voids have been reported to form in almost all crystalline materials under irradiation.[9–12,28–32] They are formed from the aggregation of vacancies or vacancy clusters in irradiated materials.[9] Voids usually take the morphology of faceted polyhedrons, bounded with low-energy surfaces. The growth process of voids is found complex and dynamic,[33,34] influenced by defect production, temperature, crystal anisotropy and the density of crystalline defects such as dislocations and grain boundaries.[9,16,17,35–41] Dislocations, in particular, are believed to facilitate the void nucleation and growth, since they act as stronger sinks for self-interstitial atoms (SIA) than for vacancies, known as dislocation bias.[42] Consequently, voids may evolve simultaneously with dislocations. The interaction of voids and dislocations is thus believed to significantly affect the evolution and stability of voids in materials.[9,42,43]

SIA dislocation loops on a basal plane are usually produced under electron irradiation in hexagonal close-packed (hcp) Mg as an additional (0001) interstitial layer.[22,31,44] The SIA dislocation loops are also called c-component dislocation loops since their Burgers vector contains the $\frac{1}{2}[0001]$ component. The formation of the c-component loop is believed to promote void formation and growth in hcp Ti and Zr metals.[32,45] However, little is known about the relationship between voids and the c-component SIAs loops in Mg so far.

We have recently reported the nucleation and early-stage growth of voids in Mg, which were observed *in situ* at atomic scale using high-resolution transmission electron microscopy (HRTEM).[46] Voids were readily formed under electron radiation in a commercial 200 kV electron microscope at room temperature due to the relatively low melting point and low electron damage

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threshold of Mg.[47] The early-stage growth of voids involves lengthening to a plate-like shape, and then thickening to a more equiaxed shape, which is determined by the growth kinetics and thermodynamics. Interestingly, the evolution of surface ledges on various void facets was observed to affect void evolution. Such a process on void facets is rarely reported before and can only be observed *in situ* at the atomic scale.

In this paper, we further report on void evolution behaviors during their late-stage growth. This stage is characterized with the interaction between voids and SIA dislocation loops. Void coalescence is also observed, which involves surface diffusion of adatoms. These findings represent new fundamental understandings of void growth mechanisms.

1. Experimental Procedures Commercial purity magnesium (99.9%) was used in this study. Major impurities include (wt%) 0.0510% Fe, 0.0320% Mn, 0.0089% C, 0.0054% Al, 0.0027% Na, 0.0026% Zn. The transmission electron microscopy (TEM) foil was prepared using a Struers TenuPol-2 electro-polishing machine in an electrolyte of 5.3 g lithium chloride, 11.16 g magnesium perchlorate, 100 ml 2-butoxy-ethanol and 500 ml methanol at -30°C and 200 mA, then low energy ion-milled on a cold stage and plasma cleaned for high-resolution TEM observations.

The electron irradiation and *in situ* observation were carried out in a JEM-2010F TEM operating at 200 kV at room temperature. The electron beam flux is about $8.2 \times 10^{23}\ \mathrm{e \cdot m^{-2} \cdot s^{-1}}$, which corresponds to a damage rate of $\sim 1.4 \times 10^{-3}\ \mathrm{dpa \cdot s^{-1}}$. The listed times in Figures 3–6 are times from the starts of video recording, which corresponds to the time listed in the Supplementary Movies 1–4, respectively. The recording time in the movie is expressed as minutes:seconds.

A series of HRTEM images taken along a $[2\bar{1}\bar{1}0]$ orientation are used for void volume calculations. According to previous studies,[29,46] the voids are found to have a polyhedron shape, bounded by $\{0001\}$, $\{01\bar{1}1\}$ and $\{01\bar{1}0\}$ facets. The schematic illustration of the void shape is shown in Supplementary Figure S1. Volume V of a void is calculated using the following equation, which is derived from the void geometry:

$$V = 2\sqrt{3}L^2W - 2\sqrt{3}L(W-S)^2\tan\theta + \sqrt{3}(W-S)^3\tan^2\theta, \qquad (1)$$

where L is the void length, which is the void dimension on the (0001) basal plane, W is the void thickness, which is the void dimension along [0001] direction, S is the projected length of the $\{01\bar{1}0\}$ facets, θ is the acute angle between $\{01\bar{1}1\}$ and $\{01\bar{1}0\}$ plane, which is 33. 8°.

2. Statistics of Void Evolution The data reported here are from an irradiated region observed under *in situ* HRTEM. It has an approximate area of about 180 nm × 100 nm and a thickness of ~70–100 nm. Figure 1(a) shows that the total volume of the voids steadily increased with irradiation dose. Figure 1(b) shows that the average void length increased from 12 to 15.8 nm, and the average thickness increased from 2.2 to 5.7 nm. In other words, the average thickness increased sharply by about 160%, while the average length increased by only 32%. This is similar to the previous finding that the void nuclei tend to grow in the thickness direction after first lengthening to a plate-like shape.[46]

While the total volume of voids increased, the total number of voids decreased during the irradiation as shown in Figure 2. This indicates that some voids grew larger while others became smaller and eventually disappeared. Interestingly, new voids continued to nucleate,

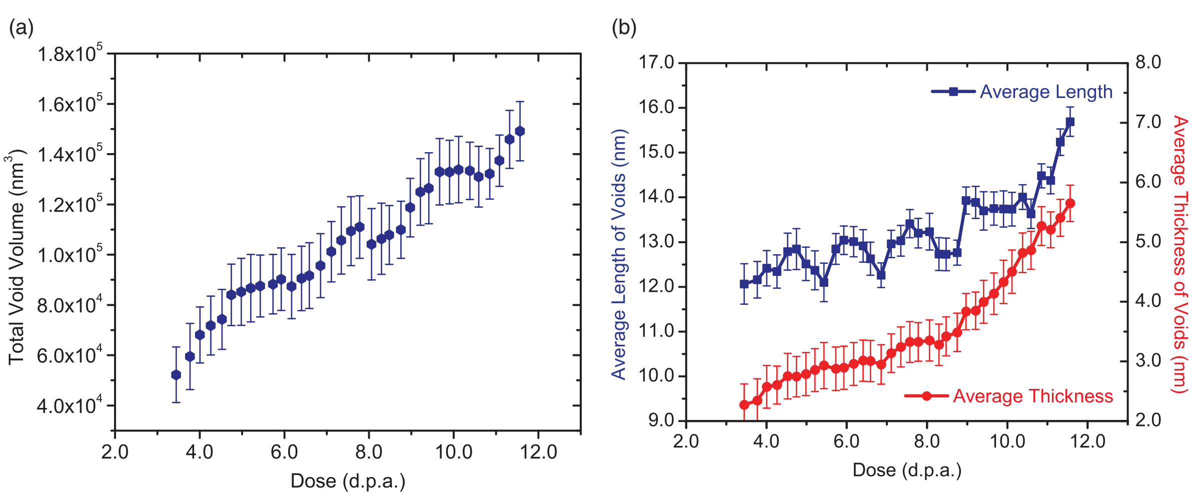

Figure 1. Statistics of void evolution behavior in the observed region with increasing irradiation dosage (a) total void volume, (b) average void length and thickness.

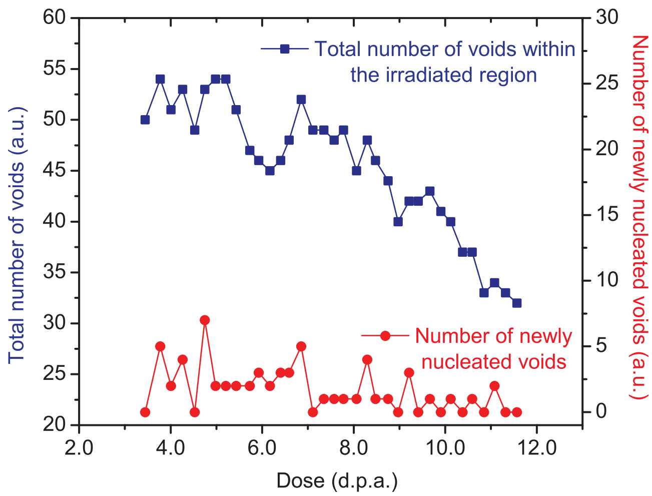

Figure 2. Evolution of (a) total number of voids and (b) number of newly nucleated voids within the irradiated region with increasing irratiation dosage.

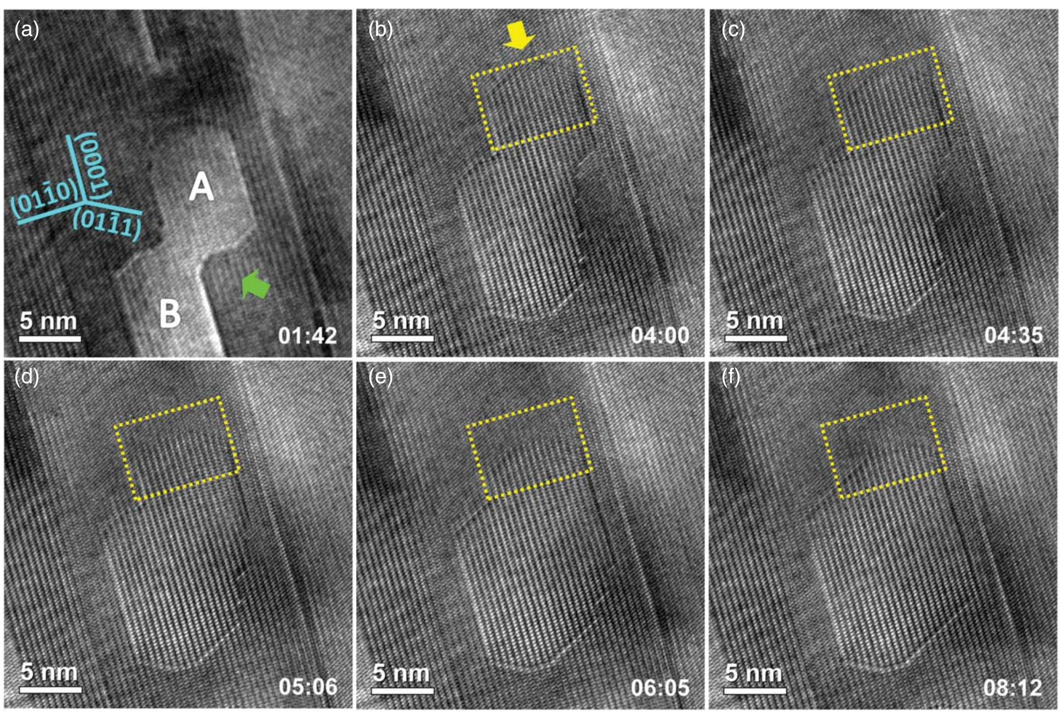

Figure 3. The coalescence of two voids (a) Ledges were formed when two voids met each other, as indicated by the green arrow. (b)–(f) Atoms on the ledges gradually diffused out and filled the cavity in the dashed frame. The total length of the two voids decreased after coalescence (see Supplementary Movie 1).

which caused the fluctuation of the total number of voids. Such simultaneous nucleation of new voids suggests a very different evolution kinetics from the conventional growth and ripening process.[48]

3. Void Coalescence As shown in Figure 3, when voids A and B initially contacted each other, a big step was formed on the (0001) facet that bridged the two voids, as shown in Figure 3(a). This big step evolved into many small steps along the (0001) facet (Figure 3(b)). The small steps moved along (0001) plane and merged to the side of the neighboring step or the facet. As shown in Figure 3(b)–(f), six steps merged to four steps, two steps, one step and finally formed one void without any step, respectively. As seen, the stepped (0001) facet evolved to form a flat facet in the end.

It is interesting that the length of void A shortened as the steps on the (0001) facet vanished, as shown in Figure 3(b) and 3(d). Moreover, the removal of steps during the void coalescence occurred much faster than a

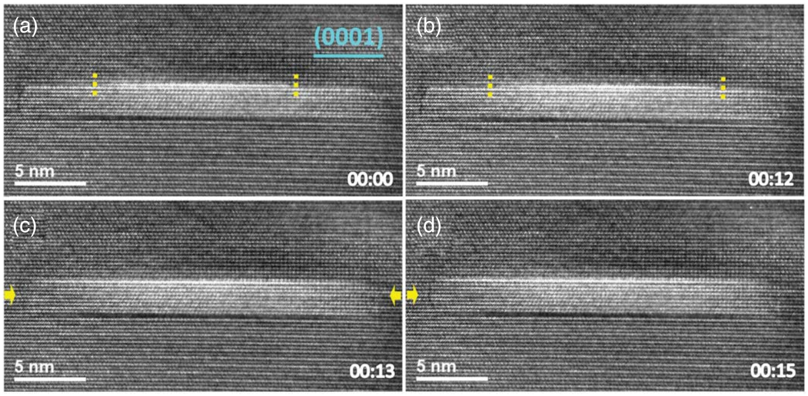

Figure 4. The shortening of void length during Stage 2 growth. (a) and (b) A vacancy layer on a (0001) facet was extending to the sidewalls, as marked by the dashed yellow lines. (c) and (d) The shortening of void length happened when this vacancy layer extended over the (0001) facet (see Supplementary Movie 2).

typical void thickness growth process via the nucleation and extension of vacancy layers on the (0001) facet.[46] These observations indicate that the atoms on surface steps most likely diffused along the void's inner surfaces [46] (from $\{0001\}$ facets to $\{01\bar{1}1\}$ and $\{01\bar{1}0\}$ sidewalls) to reshape the void. The fast diffusion of adatoms leads to fast coalescence of the voids, because the energy barrier is much smaller for adatom diffusion than for vacancy diffusion.[49]

The adatom diffusion on the inner surfaces of voids also occurred during the void thickening process. Figure 4(c) and 4(d) show that one or a few atomic layers of void length suddenly vanished during its thickness growth as marked by the yellow arrow (see Supplementary Movie 2). This occurred when the vacancy cluster was extending on a (0001) facet. In other words, the remaining atoms on the edge of the (0001) facet diffused away quickly to the sidewalls of the void, which is quite similar to what occurred during void coalescence. The diffusion of these excess atoms on the (0001) facet to the void sidewalls not only directly contributed to the length shortening but also might have reduced the vacancy concentration on the sidewalls. This will make it more difficult for the nucleation of new vacancy layers on the sidewall facets, which hinders the void growth in the length direction. In Figure 4(a) and 4(b), the extension of a vacancy layer on the (0001) facet involves vacancy diffusion from the matrix to the void surface, which is expected to be a slow process. The phenomenon of length shortening during the void thickness growth was also observed in Zirconium under 1 MeV electron irradiation.[20,50] The detailed evolution process in Zr could be very similar to the finding here in Mg, which needs to be clarified in future studies.

4. Interaction Between SIA Loop and Void

Figure 5 shows an SIA loop eating away a void while extending along a basal plane from the up-left corner to bottom-right side. The void happened to lie in front of the loop. The void gradually shrank in length as it interacted with the SIA loop edge (Figure 5(b)–(d)), and eventually disappeared after the SIA loop passes through it (see Supplementary Movie 3). It should be noted that there is a transient thickening process of the void observed in Figure 5(a) and 5(b). The void thickness largely remained un-shrunk during the process until the length and thickness are comparable in size, and then the thickness is reduced. The void volume is measured as a function of time to confirm that it is a void shrinkage process rather than a geometry change as shown in Supplementary Figure 2.

Figure 6(a) and 6(b) shows another observation that the void shrank in its size when it was located in front of the SIA loop edge similar to the previous observation in Figure 5. Interestingly, the void did not vanish after the loop passed it, but rather increased again in length (Figure 6(c) and 6(d)). The void growth began when it was located at the broad side of the loops indicated in Figure 6(e), suggesting such a void position could promote void growth. The broad sides of the loop are also found to be typical locations of void nucleation.[46]

The above observation indicates that the evolution of a void is significantly affected by its interaction with nearby SIA loops. A void tends to grow if it is located near the broad side of an SIA loop, and shrink if located in front of an SIA loop edge. This can be understood by considering the stress-induced diffusion of point defects around the loops. The SIA loop can be regarded as a Frank dislocation with compressive stress field near the broad side and the tensile stress field near the outer side. The compressive stress field would attract vacancies,[51] thus helping void grow larger in this region. However, the tensile stress field would attract SIAs, causing the void to shrink. Therefore, a transition from shrinkage to growth is expected when the void is positioned from the tension region to compression region, as shown

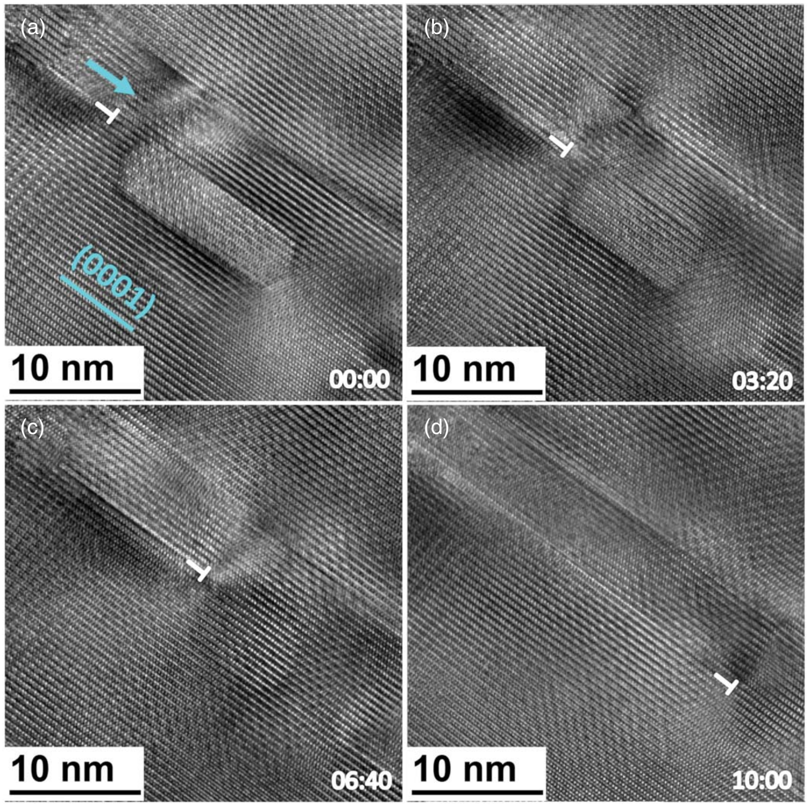

Figure 5. The void shrank in length when it contacted the edge of an extending SIA loop. (a) The loop is extending along (0001) basal plane toward the void. The direction of loop extension is indicated by the blue arrow. (b) and (c) The void shrank along length direction. (d) The void vanished after the SIA loop extends over. (see Supplementary Video 3).

in Figure 6(a)–(c). In addition, pipe diffusion through an SIA dislocation loop may occur when it comes in direct contact with the void, which was suggested to be responsible for the void shrinkage observed in an earlier study.[52]

Void growth or shrinkage is closely related to the dynamic evolution of SIA loops. The diameters of SIA loops were observed to grow. SIA loops were also observed to migrate along the $\langle c \rangle$ direction in some occasions. The SIA loops are not stationary in the matrix. They continue to grow or climb until they meet other dislocation loops [53] or a void. The dynamic evolution of SIA loops is also commonly seen in other materials under electron irradiation.[9,43,54]

The dynamic evolution of SIA loops could also result in the nucleation of new voids if the required condition of local vacancy super-saturation is attained. As shown in Figure 2, the nucleation of new voids could simultaneously occur during the entire irradiation period. These newly nucleated voids follow the same growth pattern as reported in our previous paper on early-stage void growth,[46] and then evolved as observed in this study.

The voids were observed to shrink mostly in length, and rarely in thickness (Figure 5(a) and 5(b)). The void shrinkage in thickness only occurred when the void became small and equiaxed in shape. These phenomena were caused due to the following reasons. First, the length shortening process is energetically favorable, since it causes a void to evolve into a more equiaxed shape, which lowers the overall surface energy.[29,46] The thickness reduction, on the contrary, is thermodynamically unfavorable. Second, in order to reduce void thickness, an atom layer should form on the void (0001) facet. However, this process turned out to be very difficult. On the one hand, the formation energy of an adatom on the $\{0001\}$ surface is 0.61 eV in Mg, which is much larger than those on the void sidewalls, which are 0.46 eV on the $\{01\bar{1}1\}$ facets and $\{01\bar{1}0\}$ 0.34 eV on the facets.[49] In other words, it is more difficult to form an adatom on the $\{0001\}$ surface than on the $\{01\bar{1}1\}$ or $\{01\bar{1}0\}$ surface. On the other hand, for a typical elongated void, a (0001) facet is larger than a $(01\bar{1}1)$ facet or a $(01\bar{1}0)$ facet. Therefore, it takes more adatoms to fill the entire (0001) facet, and a step needs to form first. As seen in the void coalescence, the step on the (0001) facet is energetically unfavorable and will be quickly removed via adatom diffusion through the inner facets to the sidewalls. This makes the formation of an atom layer on the (0001) surface more difficult.

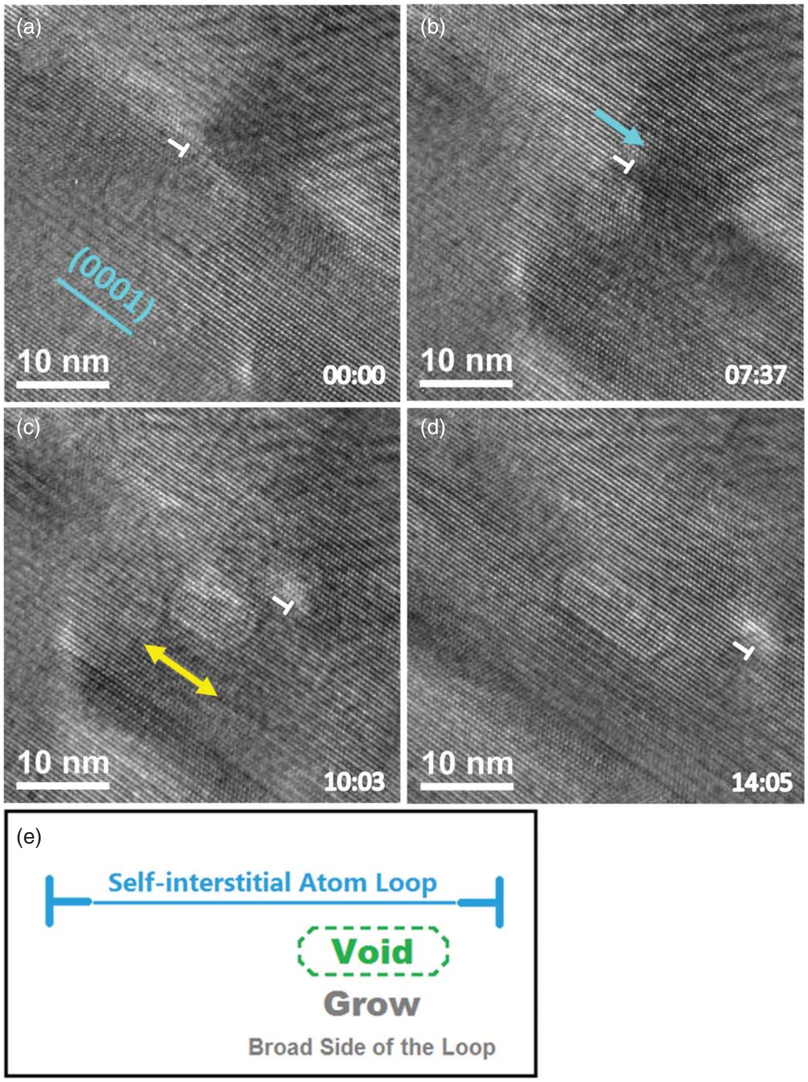

Figure 6. The void length grew as an SIA loop was passing through it. (a) and (b) The void shrank in size when it was located in front of an extending SIA loop edge. The extending direction of the SIA loop is indicated by the blue arrow. (c) and (d) The void began to grow in length when the SIA loop moved to its side (see Supplementary Movie 4). (e) Schematic illustration of a void located at the broad side of an SIA loop. A void tends to grow on this side.

5. Conclusion With increasing irradiation dosage, the total volume of voids increases and the number of voids decreases, while new voids still nucleate but with decreasing nucleation rate. The evolution of voids in Mg under active irradiation is significantly affected by their interactions with SIA loops. A void will grow if it is on the broad side of an SIA loop, and shrink if it is located in front of the loop edge. Two voids close to each other may coalesce with each other via the formation and elimination of steps on the {0001} facets. The fast diffusion of adatoms on the {0001} facets is believed to be largely responsible for the fast coalescence of voids. Void growth in thickness is usually accompanied by shrinking in length. Elongated voids usually do not shrink in thickness (*c*-axis) until their length is reduced to an extent that the void becomes equiaxed. This is attributed to the difficulty in nucleating an atomic layer on the {0001} facets as well as to thermodynamics, which favor equiaxed voids.

Supplementary Online Material. A more detailed information on experiments is available at http://dx.doi.org/10.1080/21663831.2014.904826.

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