Microstructure of α -U and δ -UZr₂ phase uranium-zirconium alloys irradiated with 140-keV He⁺ ion-beam

Sangjoon Ahn^{a,*}, Sandeep Irukuvarghula^b, Sean M. McDevitt^c

^a*Department of Nuclear Engineering, School of Mechanical and Nuclear Engineering, Ulsan National Institute of Science and Technology, Ulsan, 44919, Republic of Korea*

^b*Manchester Materials Science Centre, School of Materials, University of Manchester, Manchester, M1 7HS, United Kingdom*

^c*Department of Nuclear Engineering, Texas A&M University, College Station, TX 77843, United States*

Abstract

Ion-beam irradiation has been conducted to demonstrate and assess solid phase dependence of fission gas bubble nucleation and growth in metallic nuclear fuels. Differential formation and growth of irradiation-induced voids and bubbles were observed from as-cast uranium-zirconium (U-Zr) alloys including 0.1 and 40wt% zirconium. The U-Zr alloys were irradiated with 140-keV He⁺ ions with fluences ranged from 1×10^{14} ions/cm² to 5×10^{16} ions/cm². Several larger spherical bubbles ($< \sim 70$ nm) were revealed from an irradiated single α -U phase alloy (U-0.1Zr) in which observed bubbles were mobile under 200-keV electron beam of transmission electron microscope (TEM). *In-situ* mergence and leakage of the bubbles in the uranium-rich alloy was also evident during the observation. In contrast, numerous number of small (~ 6 nm) angular voids, or low pressure bubbles, were found in an irradiated δ -UZr₂ phase alloy (U-40Zr), in

* Corresponding author. Tel.: +82-52-217-2351
E-mail address: sjahn99@unist.ac.kr (Sangjoon Ahn)

which the radiation-induced nanostructure was stationary throughout the observation. This study may reaffirm higher radiation-susceptibility of the α -U phase than the δ -UZr₂ phase, which could strengthen the needs of separate accounting of solid phase effects for metallic fuel performance modeling particularly regarding the fuel restructuring phenomena, such as fission gas swelling and constituent redistribution, since conventional models are commonly adopted homogeneous medium assumption, not in accordance with the results of post-irradiation examinations of used metallic nuclear fuel in which the formation of several concentric phase zone was distinct corresponding to a steep radial temperature gradient in the fuel pin and multiple isotherm lines in the binary phase diagram of U-Zr alloy system.

Keywords: Uranium-zirconium alloy; metallic nuclear fuel; irradiation-induced void and bubble; swelling; ion beam irradiation.

1. Introduction

Uranium-zirconium (U-Zr) alloy is a promising nuclear fuel demonstrated high burnup of circa 20 at% in the Experimental Breeder Reactor II (EBR-II) [1]. The high burnup was enabled with an optimized fuel element design employing a low smeared density (~75%) fuel and an enlarged cladding plenum, taken together permitting fuel swelling mainly due to gaseous fission product accumulation without significant fuel-cladding interaction and subsequent cladding performance degradation that could potentially lead to cladding rupture and radioactivity release to the environment. Thus, fission gas swelling behavior of metallic nuclear fuel is a key to model

its performance that can be a crucial prerequisite to pursue even higher fuel burnup to achieve technical feasibility for closed fuel cycle annexed with fast reactor systems in the near future.

Extensive post-irradiation examinations (PIE) on the metallic used nuclear fuel discharged from the EBR-II revealed concentric phase zone formation within the fuel pins due to a steep radial temperature gradient [2]. Fission gas bubble morphologies also differentiate each phase zone in the alloy fuel pins; large interconnected bubbles were formed in central γ phase zone in contrast to swirled two-phase lamellar structure in periphery zone consisting of the (α -U, δ -UZr₂) phase formed due to small bubbles alongside the phase boundaries. This characteristic phase effects, however, are not fully reflected in current performance models for metallic nuclear fuel, since the models commonly assume homogeneous medium for entire fuel pin against the experimental observations [3-12]. Consequently, the modification of the model to account the phase effects is a matter of course to narrow the gap between the model prediction and in-pile fuel properties and dimensions while simultaneously pushing the burnup limit.

Aforementioned modification to include phase-dependent criterion into the modeling could also be beneficial in case of varying alloy fuel composition to enhance fuel performance. Historically, as-fabricated U-Zr alloy nuclear fuel composition was limited at 10 wt% zirconium due to technical limitation of injection casting, the fuel fabrication method used at the time [13]. This limitation, high melting temperature of Zr-rich (>10 wt%) fuel, could be circumvented by utilizing recently developed, or under developing, fuel fabrication methods, such as α -U phase extrusion [14, 15]. The validity of the current empirical models adopting the homogeneous medium assumption would certainly become more questionable with non-conventional composition fuel. Following the post-irradiation studies, Zr content of each phase zone changes along with the burnup increase due to fuel constituent redistribution; Zr depletion from

intermediate (or central) β -U phase zone decreased the local Zr composition down to ~ 2 wt%, on the other hand, the fuel periphery comprised of (α -U, δ -UZr₂) phase mixture was enriched up to ~ 24 wt% Zr [16, 17]. Therefore in the outer zone of a high burnup U-Zr alloy fuel pin, volume fraction of the δ -UZr phase would certainly increase, while phase boundary density may fluctuate. This phenomenon would bring uncertainties for the current fuel performance codes that are not directly taking account of each solid phase effects on the fuel restructuring, but commonly using empirical formulations to reproduce the existing material properties versus the burnup, especially in case which the responses of the two phases (α -U and δ -UZr₂) to radiation damage are substantially differential.

This study provides nano-scale experimental observations using transmission electron microscopy (TEM) on the phase-dependent gas bubble nucleation and growth in two single-phase U-Zr alloys, i.e., U-0.1Zr for α -U phase and U-40Zr for δ -UZr₂ phase, irradiated with 140-keV helium ions at various fluences ranging from 1×10^{14} to 5×10^{16} ions/cm² to experimentally evidence phase-differential irradiation-induced microstructure evolution implicating the very needs for reconsideration of the application of homogeneous medium assumption for metallic nuclear fuel performance modeling.

2. Materials and Methods

2.1 Alloy Preparation and Characterization

The U-0.1Zr and U-40Zr alloys were melt-cast from nitric acid-washed depleted uranium chunks and high-purity zirconium crystal bars in cylindrical yttrium oxide crucibles using a high

temperature furnace. The furnace was held at ~ 1900 °C for 1 h and then cooled down to 25 °C at a rate of 30 °C/min under an argon atmosphere. The cast alloy slugs were flipped and re-melted under identical conditions to improve the alloy homogeneity. The resulting dimensions of the cylindrical alloy slugs were ~ 15 mm both in length and diameter [18].

The as-cast U-Zr alloys were sectioned into ~ 1 mm thick buttons and preliminarily characterized at a micro-scale using a Cameca SX-50 electron probe microanalyzer (EPMA) equipped with an energy dispersive spectrometer (EDS) and a wavelength dispersive spectrometer (WDS) to affirm the homogeneity and acceptable degree of secondary phase formation in the alloys. The nano-scale metallographic characterization of the EPMA-characterized U-Zr alloys was conducted using JEOL JEM-2010.

2.2 Ion-beam Irradiation and TEM Specimen Preparation

Thin alloy buttons (~ 0.5 mm thick) sectioned from the EPMA characterized alloy slugs were mechanically thinned to ~ 150 μm thick foils using gradually smaller grit of SiC papers and stepwisely finished with 3, 1, and 0.25 μm polycrystalline diamond suspensions without additional mechanical force applied to relieve mechanical damage perhaps induced from the thinning process.

The prepared alloy foils were irradiated with 140 keV helium ions in a high vacuum ($\sim 10^{-7}$ Torr) implant chamber of an accelerator equipped with Danfysik ion source. The ion-beam flux was maintained around 1×10^{16} ions/ $\text{cm}^2 \cdot \text{h}$ and the tested fluences were 1×10^{14} , 1×10^{15} , 1×10^{16} , and 5×10^{16} ions/ cm^2 , that are conveniently referred as to 0.01, 0.1, 1, and 5, respectively, for the notation of TEM specimens hereinafter.

Several 3 mm diameter disks were punched out of the alloy foils using a FISCHIONE Model 130 specimen punch. The punched alloy disks were electrochemically thinned using a single vertical jet electropolisher, South Bay Technology Model 550D. For the electropolishing of the alloy specimen, a 5% perchloric acid/95% methanol electrolyte was used and ~40V of electric potential was applied. The prepared specimens were immediately preserved in a plastic vial filled with acetone to delay surface oxidation during the specimen transportation to the TEM facility [19].

Irradiation-induced radiation damage and implanted helium concentration in the U-Zr alloys were assessed by using the Stopping and Range of Ions in Matter (SRIM) [20]. Note Fig. 1 shows the distribution of radiation damage and helium concentration for the reference case (1×10^{16} ions/cm²) and microstructures and solid phases of the alloys were disregarded during the calculation due to the limitation of the SRIM. Practical distribution of helium atoms can also be off from the theoretical prediction, since radiation damage and slightly elevated alloy foil temperature during the ion-beam irradiation both enhance the diffusion of implanted helium atoms, again not considered in the program.

The sizes and densities of observed bubbles in TEM bright field (BF) images of the alloys were measured using ImageJ (NIH).

3. Results

For two U-Zr alloys (U-0.1Zr and U-40Zr) irradiated at the same dose as shown in Fig. 1, approximately two-fold larger radiation damage and helium concentration were resulted in U-0.1Zr alloy than in its Zr-rich counterpart (U-40Zr alloy) from the alloy foil surface to 100 nm

depth, where have likely provided electron transparent areas in the TEM specimens assuming negligible loss of the irradiated alloy surface because single-sided electropolishing was conducted from the unirradiated surface of the alloy foils.

Figure 2 shows undamaged as-cast microstructures of the U-Zr alloys; (a) U-0.1Zr and (b) U-40Zr. In both alloys, single-phase homogeneous medium was clearly observed that is commensurate with U-Zr alloy phase diagram. An intact crystalline structure with some edge dislocations is vivid from the U-0.1Zr alloy bulk shown in Fig. 2(a). The dark rim appeared on the perforated boundary shown in Fig. 2(b) seem to be an edge-rolled thin alloy foil which was constantly waving during the TEM observation, thus indicating ultra-thin nature of the specimen.

Figure 3 shows a group of intermediate to large size bubbles observed in the as-cast U-0.1Zr alloy irradiated at 5×10^{16} ions/cm², or referred as to U-0.1ZrAi5 hereinafter ('A' and 'i' stand for 'as-cast' and 'irradiated', respectively.). Figure 3(b) was imaged 190 sec after the moment of Fig. 3(a) to visualize short-term evolution of irradiation-induced bubbles in U-0.1ZrAi5 under high energy (200-keV) electron beam of TEM, which largely enhanced the mobility of the bubbles. Most notably, several small bubbles were merged into the largest bubble located left center of the figures, which was grown and migrated toward the opposite direction of the deflection on the right-hand side of the bubble. Pseudo-spherical shapes of the bubbles imply the high internal pressure enough to overcome the surface tension of the U-rich medium. Figure 3(c) is given to emphasize the fact that always the brighter contrasts are resulted from three-dimensional overlap of the bubbles. From Fig. 3(d), thinner effective thickness of the specimen, i.e., the bubble diameter subtracted TEM specimen thickness, at the bubble center is unmistakable from the two bubbles about to merge; the areas having the bubbles are effectively too thin, therefore significant noise on the charge-coupled device (CCD) camera of the TEM was

unavoidable due to relatively unattenuated electron beam penetrating partially hollow medium, while the adjacent area to the bubbles shows darker contrast without any noise.

Figure 4(a) shows small ($< \sim 20$ nm) angular (or indefinite) shapes of the bright features sparsely scattered in U-40ZrAl, i.e., the as-cast U-40Zr alloys irradiated at 1×10^{16} ions/cm², that are likely radiation-induced bubbles. The bubble-like features ought not to be confused with the darker and larger ($> \sim 100$ nm) features at the periphery of the TEM BF image, that are re-deposition type surface contamination from the electropolishing of the specimen. Moreover, Fig. 4(b) clearly shows several nanometer-scale holes (surrounded by bright Fresnel fringe) located within the polycrystalline area; note the randomized atomic crystal orientation nearby the holes, indicating significant radiation damage induced in the observed area. It is very unlikely that those smaller dimension holes were haphazardly and concurrently formed due to electropolishing-induced macroscopic perforation of the specimen.

Figure 5 shows numerous bubbles were formed in the higher dose irradiated U-40Zr alloy (U-40ZrAl5). The bubble sizes and densities in Figs. 4 and 5 measured using ImageJ (NIH) are summarized in Table 1. A trend is clear from Table 1 that bubble densities are virtually linearly proportional to the irradiation dose, yet the dimensions of the bubbles were nearly constant.

From U-40ZrAl5 specimens, a ‘hex-mesh’ structure (porous area comprised of numerous hexagonal, or somehow polygonal, rims interconnected to each other) were often observed as shown in Fig. 6. The TEM BF image analysis confirmed similar dimensions, but low density, of the central holes of the hex-mesh structure in Fig. 6 compared to the counterparts of the bubbles in Fig. 5; the results are also included in Table 1. Therefore, it may be reasonable to suspect that the hex-mesh structures were formed due to over-polishing of the highly irradiated specimen, that could have been resulted in very thin, yet mechanically rigid, observable area, since the U-

40Zr alloy is Zr-rich, thus chemically more resistive to the electropolishing conditions than the U-0.1Zr alloy [19].

4. Discussion

The tested irradiation doses were selected to be stepwisely increased with at least five- or ten-fold intervals from 1×10^{14} ions/cm² to 0.1, 1, and 5×10^{16} ions/cm² to ensure the order of irradiation damage was not reversed between the adjacent-dose-irradiated different Zr composition alloys (the TEM specimens of U-0.1ZrAl1, for instance, have been exposed to certainly less radiation damage than those of U-40ZrAl5), since the differential stopping power per unit thickness of the two U-Zr alloys is well noted from Fig. 1 that can be intuitively understood by referring to about a half density of the U-40Zr alloy compared to that of the U-0.1Zr alloy.

Unfortunately, the TEM specimen preparation was largely unsuccessful for low-to-intermediate dose (below 1×10^{16} ions/cm²) irradiated U-0.1Zr alloys, maybe due to double-side thinning attempted in order to simultaneously remove surface oxidation and less radiation-damaged area. The irradiated alloy foil surface thinning may have been resulted in the loss of entire radiation-damaged layer of the specimens instead of appropriately exposing the peak damage area, even when a relatively low electric potential of ~40 V was applied for less than 3 sec after stabilized electrolytic flow was achieved. As a consequence, clear observation of irradiation-induced features was limited to U-0.1ZrAl5, U-40ZrAl1, and U-40ZrAl5 as shown in Fig. 3-6, among all alloy specimens prepared utilizing electropolishing.

From the highest tested dose-irradiated alloys (5×10^{16} ions/cm²), a greater α -U susceptibility compared to δ -UZr₂ phase is evident from the comparison between Figs. 3 and 5, which are obtained from the same dose-irradiated U-0.1Zr and U-40Zr alloys. High pressure of the large bubbles up to ~70 nm diameter in U-0.1ZrAl₅ (Fig. 3) was apparent from the bubble diffusion activated under 200 keV electron beam. During the TEM observation, the bubbles were coalescing to each other and often escaped from the thin electron transparent area either after growing beyond the area thickness or after being diffused into the specimen surface. In addition, it could be speculated that the deflections on the bubble surface affected their movement. Note the bubble locations in Fig. 3(b) compared to Fig. 3(a). The movement of the bubbles guided by their geometrical deflection is a clear evidence that shows the observed features are not any type of surface contamination. Also spheroid shapes with varying diameter of the bubbles are the strong indication of which instantaneous new balance between the surface tension of U-rich medium and helium gas pressure inside the bubbles were being made inside the irradiated alloy medium initiated due to high energy electron beam from the TEM. Finally, the bright contrast of the features and the brighter contrast unexceptionally obtained from their overlapping further strengthens the identification of the features as the radiation damage-induced high-pressure helium gas bubbles. Moreover, the similar bubble morphologies obtained from the ion-beam irradiation on uranium-based alloys from some references [21, 22] can weigh the conclusion above.

In contrast, smaller (~6 nm) faceted, not spherical, voids numerous observed in U-40ZrAl₅ (Fig. 5) were stationary throughout the observation even under focused electron beam cross-over, which was able to jiggle the larger bubbles in U-0.1ZrAl₅. The dimensions, shapes, and contrasts of the features jointly indicate that the features are voids or low-pressure bubbles

near vacuum. This low pressure could be attributed to deeper penetration of helium (concentration peak at ~ 450 nm) than radiation damage peak (~ 350 nm) as indicated in Fig. 1(b). Also, the smaller dimensions could imply the relative lack of diffusion of the nucleated bubbles in intermetallic δ -UZr₂ phase dominant medium of the U-40Zr alloy compared to α -U phase of the U-0.1Zr alloy.

To ensure the morphologic and dimensional differences between the bubbles in the two alloys were due to two different room temperature phases comprise the U-Zr alloys, ion-beam heating effect was considered to estimate the maximum temperature of the alloy foil during the irradiation. Although ion-beam irradiation was conducted at an ambient temperature, yet the alloy foil temperature can only be slightly elevated by the implanted high energy (140-keV) helium ions, because the foils were attached using a carbon adhesive on a low thermal conductivity ceramic wall of the implant chamber of the accelerator. A simplistic 1-D energy conservation model, which assumes energy balance between the beam influx and the heat dissipation through the carbon adhesive, estimated the foil temperature during the irradiation can be ~ 150 °C higher than an ambient temperature, which suggests the alloy phase have never been changed throughout the irradiation. This approach could associate with relatively large error due to several uncertainties, i.e., thermal conductivity of the carbon adhesive and gap conductance between the foil and the wall, thus the most conservative assumptions were adopted for the calculation.

The bubble densities in the U-40Zr alloys were almost linearly proportional to the radiation damage, while the bubble sizes were constant, as shown in Figs. 4 and 5 and as summarized in Table 1. The lower bubble densities in the hex-mesh structure (Fig. 6) can be attributed to the thinner nature of the porous area that was exposed to excessive thinning. This

density proportionality to the radiation damage could suggest that vacancy cluster formation in the δ -UZr₂ phase is fairly local, thus the bubble growth could be halted for the low temperature/low dose regime, while the bubble formation is continued. In contrast, relatively large bubbles were already started to form in α -U phase alloy irradiated at the same dose. The observed dimensions of the bubbles, up to ~70 nm, are in fact the maximum size can be observed using a TEM, since the TEM specimen thickness to be electron transparent cannot much exceed ~100 nm. Also, considering the large bubbles were sensitive to the stimulation from the electron beam, bubble coalescence in U-rich medium can likely be activated at relatively lower temperatures than the threshold temperature for the Zr-rich δ -UZr₂ phase medium.

Overall, it can be envisioned from the above observations that the bubble nucleation and growth in the periphery (α -U, δ -UZr₂) phase zone of a low burnup U-Zr alloy fuel pin could be concentrated in the α -U phase lamellae other than the δ -UZr₂ phase precipitates; the α -U/ δ -UZr₂ phase boundary effects on the fission gas bubble nucleation and growth certainly deserves a future study, but it is out-of-scope of this thesis. It should be noted that this observation and prediction would be rather valid for very low burnup fuel, therefore can be limitedly referred for as initial inputs for the fuel performance modeling, when gaseous fission products concentration is low enough to not alter the observed characteristic of the bubbles formed in Zr-rich intermetallic δ -UZr₂ phase medium.

5. Conclusions

Irradiation-induced microstructure evolution of α -U and δ -UZr₂ phase U-Zr alloys (U-0.1Zr and U-40Zr) irradiated using 140-keV helium ion-beam was observed using TEM. The U-

0.1Zr alloy comprised of the α -U phase appeared to be more susceptible to the radiation damage to allow the formation of larger spherical bubbles, while the U-40Zr alloy have exhibited relatively smaller and faceted void formation instead. Thus, in the periphery of the U-10Zr alloy fuel at low burnup the fission gas bubble nucleation is suspected to rather be concentrated in the α -U matrix phase than in the δ -UZr₂ phase precipitates. This finding would urge solid-phase-based modeling of the metallic nuclear fuel performance to embody better physical integrity for those models that are currently assuming homogeneous medium for the entire fuel pin against the historical post-irradiation studies of spent U-Zr alloy fuels.

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Figure caption

Fig. 1. SRIM simulation on (a) the induced radiation damage and (b) the implanted helium distribution in U-0.1Zr and U-40Zr alloys.

Fig. 2. As-fabricated microstructures of U-Zr alloys; (a) U-0.1Zr and (b) U-40Zr.

Fig. 3. Large bubbles observed in U-0.1ZrAi5 mobilized under 200-keV electron beam; a group of bubbles at (a) reference time and (b) 190 sec after, respectively; (c) brighter contrasts resulted from the overlapped bubbles, and (d) two bubbles about to merge.

Fig. 4. Bubbles in U-40ZrAi1.

Fig. 5. Bubbles in U-40ZrAi5.

Fig. 6. Over-polished porous, hex-mesh, structures in U-40ZrAi5.

Table caption

Table 1 Average sizes and densities of bubbles observed in 140-keV He⁺ ion-beam irradiated U-40Zr alloys estimated from TEM BF image analysis using ImageJ (NIH).

Table 1 Average sizes and densities of bubbles observed in 140 keV He+ ion-beam irradiated U-40Zr alloys estimated from TEM BF image analysis using ImageJ (NIH).

Dose (ions/cm ²)	Bubble zone			Hex-mesh zone		
	Size (nm)	Density (#/μm ²)	Swelling (area%)	Size (nm)	Density (#/μm ²)	Swelling (area%)
1 × 10 ¹⁶	6.0 ± 1.5	250 ± 40	0.7 ± 0.6	-	-	-
5 × 10 ¹⁶	5.2 ± 1.2	1460 ± 30	3.1 ± 1.7	5.8 ± 1.5	670 ± 70	1.8 ± 1.3

Figure 5

Figure 6

Figure 85

Figure 86

Figure 5

Figure 6

Figure 7

Figure 8

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Figure) 5

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