Effect of Heavy Ion Irradiation on Microstructural Evolution in CF8 Cast Austenitic Stainless Steel

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ABSTRACT

The microstructural evolution in ferrite and austenite in cast austenitic stainless steel (CASS) CF8, as received or thermally aged at 400°C for 10,000 h, was followed under TEM with in situ irradiation of 1 MeV Kr ions at 300 and 350°C to a fluence of 1.9×10^{15} ions/cm² (~ 3 dpa) at the IVEM-Tandem Facility. For the unaged CF8, the irradiation-induced dislocation loops appeared at a much lower dose in the austenite than in the ferrite. At the end dose, the austenite formed a well-developed dislocation network microstructure, while the ferrite exhibited an extended dislocation structure as line segments. Compared to the unaged CF8, the aged specimen appeared to have lower rate of damage accumulation. The rate of microstructural evolution under irradiation in the ferrite was significantly lower in the aged specimen than in the unaged. This difference is attributed to the different initial microstructures in the unaged and aged specimens, which implies that thermal aging and irradiation are not independent but interconnected damage processes.

1. Introduction

The austenitic stainless steel welds and cast austenitic stainless steels (CASSs) are widely used in light water reactor (LWR) for joints and internals. They have a duplex structure of austenite (fcc) and ferrite (bcc) where the ferrite is typically 5-30% [1]. The presence of the ferrite improves the corrosion and mechanical properties. However, its phase instability during thermal aging at 300-500°C may, depending on the ferrite content, result in an increase in the yield and tensile strength, and a decrease in the ductility, impact energy and fracture toughness [1-6].

Certain welds near the reactor core might experience fast neutron fluence as high as 10^{22} n/cm² [7]. Compared to aging, however, the irradiation effects on the welds are much less understood. A recent study suggested that the combined effect of thermal aging and irradiation results in further embrittlement of CASSs [6]. Therefore the

potential interaction of the two degradation processes is of concern. The complex interaction of irradiation-induced defects, duplex structure and phase instability need to be investigated in order to evaluate the degradation of mechanical properties under long-term irradiation at high temperature.

Two major questions are to be addressed in this study. Firstly, how differently are the ferrite and the austenite in CF8 reacting to the irradiation under the same condition? The austenitic (fcc) and ferritic (bcc) alloys and metals behave quite differently during irradiation. Irradiation produces mobile $\frac{1}{2}\langle 111 \rangle$ and immobile $\langle 100 \rangle$ perfect loops in bcc Fe [8], while it produces immobile faulted dislocation loops and stacking fault tetrahedral in fcc metals [9]. Victoria et al. showed that a much lower dose is required to produce the same amount of dislocation loops in the fcc metals than in the bcc metals [9]. A difference in cascade production efficiency between the two crystalline structure was suggested [10].

Another question is how the presence of the second phases in the ferrite affects the evolution of defect clustering. The aging in CASSs at 270-400°C causes spinodal decomposition of the ferrite into Fe-rich α phase and Cr-rich α' phase, and the precipitation of G-phase ($M_6Ni_{16}Si_7$, $M = Mn, Cr$) in the ferrite [11]. Previous studies reported different findings about the irradiation effect in Fe-Cr alloy systems where irradiation can accelerate or reduce spinodal decomposition and G-phase precipitation in the ferrite [7, 12-15]. Irradiation introduced supersaturated point defects that can enhance the diffusivity needed for phase transformation. Meanwhile, irradiation ballistic mixing can recoil atoms from precipitates and increase disordering [16]. On the other hand, the precipitates can act as trapping sites for vacancies and interstitials to recombine [17], affecting the defect concentration, diffusivity and the evolution of defect microstructure during irradiation [18].

This study used thermally-aged and as-received (unaged) CASS of grade CF8 as a surrogate of austenitic stainless steel welds to study the combined effects of thermal aging and irradiation damage on the microstructure. Irradiation experiments were performed using 1 MeV Kr at 300 and 350 °C with in-situ TEM observations. Ion irradiation is an accelerated tool to understand the neutron irradiation effect. The observed dose dependence of defect clustering and G-phase precipitation in CF8 showed distinctive microstructural evolution in ferrite and austenite phases and in varied thermal-aging conditions, which was important microstructural information for understanding the long-term mechanical property degradation of this class of materials under elevated temperature and irradiation in LWRs.

2. Experimental Procedure

2.1 Materials

Thermally-aged and unaged CF8 were examined in this study. The nominal chemical composition (wt%) of CF8 was Fe–20.46Cr–8.08Ni–0.64Mn–0.31Mo–1.07Si–0.063C–0.062N–0.021P–0.014S [6]. Thermal aging treatment was conducted at 400°C for 10,000 hours. Accelerated aging at 400°C was used to evaluate aging in LWR service temperature at 280-340°C [19]. CF8 has a duplex structure of ferrite and austenite as shown in Figure 1. **Metallographic examinations showed similar morphology of ferrite (~17%) in CF8 before and after the thermal aging treatment.**

2.2. Irradiation Experiments

Disc specimens of 3 mm in diameter were punched from a thin sheet and subsequently electropolished to perforation using a Tenupol twin-jet polishing unit with an electrolyte of 5% perchloric acid plus 95% methanol at -35°C. The electron-transparent thin foil specimens were irradiated in situ with 1 MeV Kr ion at 300°C and 350°C in the IVEM-Tandem Facility at Argonne National Laboratory. The specimens were irradiated with an incident angle around 15° from the specimen normal, while the microstructure was observed *in situ* with 200 keV electrons. A Faraday cup in the microscope at 2 cm from the specimen was used to measure the ion dosimetry. The irradiation temperature was controlled within $\pm 3^\circ\text{C}$.

The specimens were irradiated to an ion fluence of 6.3×10^{14} ions/cm² (~1 dpa) with an ion flux of 1.6×10^{11} ions/cm²/sec (~0.00025 dpa/s), and to a high fluence with an increased ion flux of 6.2×10^{11} ions/cm²/sec (~0.001 dpa/s). The final fluence was 1.9×10^{15} ions/cm² (~3 dpa). As shown in **Figure 2**, the damage profile and Kr ion distribution of 1 MeV Kr ion irradiations in the CF8 was calculated with SRIM-2012 [20] using the quick damage mode with the density of 7.8 g/cm³ and displacement energies of 40 and 45 eV for Fe and Cr atoms, respectively [21]. The calculation indicates a reasonably uniform damage (Frenkel pair production) and little Kr ion retention (~0.4%) in the TEM foil of a thickness around 100 nm. In order to examine the dose rate effect, an irradiation experiment with lower ion flux of 1.6×10^{10} ions/cm²/s (~0.000025 dpa/s) to a fluence of 1.9×10^{14} ions/cm² (~0.3 dpa) was performed.

The following equation was used to calculate the dpa values:

$$dpa = \frac{\Phi \times 10^8 \times D}{N}$$

where Φ is the fluence in ions/cm², D is the damage rate in vacancies/ion/Å and N is the atomic number density in atoms/cm³. An average damage rate of 1.3 vacancies/ion/Å was estimated according to the SRIM calculation in **Figure 2**.

2.3. Transmission Electron Microscopy

The irradiation-induced dislocation loops and G-phase precipitates were imaged under bright field (BF) and weak-beam dark field (WBDF) diffraction conditions. Examinations with dark field images from G-phase reflection, which shows only G-phase precipitates, were reported previously in Ref. [15]. The Burgers vectors of dislocation loops in the ferrite were determined by $g \cdot b$ analysis in WBDF with $g = 110$, $g = 200$ and $g = 020$ near (001) zone axis [8]. The size and density of dislocation loops and precipitates were measured manually. The foil thickness was estimated by counting the thickness fringes in WBDF. A 50% confidence factor on defect clusters or precipitates of relatively weak contrast was applied to estimate the uncertainty in density measurements [22]. An uncertainty of 1 nm was estimated for the uncertainty in size measurement. The foil surface effect to the formation of defect clusters was evaluated by measuring the areal density of defect clusters or precipitates as a function of foil thickness [23]. Under and over focused BF images were used to show voids or bubbles induced by irradiation [24].

3. Results

3.1. Microstructure prior to irradiation

For the unaged specimens, the dislocation density was low in both ferrite and the austenite. Figure 3 (A) shows an example of the ferrite. $M_{23}C_6$ carbides were found partially decorating the ferrite-austenite phase boundary with a cube-on-cube orientation to the austenite. No G-phase precipitates or spinodal decomposition were observed.

After aging, fine G-phase precipitates of ~ 3 nm were uniformly distributed in the ferrite matrix as shown in Figure 3 (B). Larger precipitates were found at dislocations and in the vicinity of $M_{23}C_6$ carbides. G-phase denuded zone of ~ 50 nm wide was observed at phase boundaries. Detailed TEM and APT characterization of the G-phase precipitates in aged specimens can be found in Ref. [13, 15]. In addition to G-phase precipitates, the mottled image in Figure 3 (B) indicates the spinodal decomposition of δ ferrite into Fe-rich α phase and Cr-rich α' phase, which was confirmed previously by a companion APT study [13]. In contrast to the ferrite, the microstructure in the austenite was not changed after aging.

3.2. Microstructural evolution in unaged CF8 under irradiation

Figure 4 and Figure 5 shows the microstructural evolution in the ferrite in the unaged CF8 irradiated at 300°C and 350°C, respectively. Nanometer-sized clusters formed, and their density and size increased with increasing ion fluence. Quantitative measurements are shown in Figure 6. The microstructural evolution was similar between the two temperatures. At 1.9×10^{15} ions/cm² (~ 3 dpa), the extended structure of loop strings consisting of small $\frac{1}{2}\langle 111 \rangle$ loops (see Ref. [25]) was observed in relatively thick regions (> 100 nm) as shown in Figure 7(a).

After irradiation, the diffraction pattern of the ferrite was carefully examined to look for satellite spots from G-phases. As a result, no G-phase diffraction spots was observed, indicating that irradiation caused few well-developed G-phase precipitates, and dislocation loops were the primary defects in the unaged CF8 under the examined irradiation conditions. In addition, no voids or bubbles were observed after irradiation.

The $g \cdot b$ analysis was performed on an unaged CF8 irradiated to 1.9×10^{14} ions/cm² (~0.3 dpa) with a lower flux of 1.6×10^{10} ions/cm²/s. Dislocation loops with a mixture of $\frac{1}{2}\langle 111 \rangle$ and $\langle 100 \rangle$ Burgers vectors (58% and 42%, respectively) were observed, which is consistent with the literature [8, 25-26]. No evident dose rate effect on the size and density of dislocation loops was observed between the two fluxes, 1.6×10^{10} and 1.6×10^{11} ions/cm²/s.

The microstructural evolution in the austenite irradiated at 300°C and 350°C is shown in Figure 8 and Figure 9, respectively. The size and density measurements of dislocation loops are shown in Figure 6 along with the ferrite data. Visible dislocation loops appeared in the austenite at a very low dose (~0.01 dpa), and the density increased rapidly afterward. Beyond 6.3×10^{14} ions/cm² (~ 1 dpa), the density became too high to be measured. The loop density was consistently higher in the austenite than in the ferrite for both irradiation temperatures. The loop size, however, was similar between the two phases.

Figure 7(B) shows the microstructure of a relatively thick (> 100 nm) region of the austenite in an unaged CF8 irradiated at 300°C to 1.9×10^{15} ions/cm² (~ 3 dpa). Unlike the extended structure observed in the ferrite, a well-developed dislocation network was observed in the austenite.

3.3. Microstructural Evolution in the ferrite of Aged CF8 under irradiation

Figure 10 shows the microstructural evolution in the ferrite of aged CF8 irradiated with 1 MeV Kr at 300°C. The initial microstructure consisted of G-phase precipitates (white dots) and spinodal decomposition (mottled

background [27]). With increasing ion fluence, the number density of white dots, which could be G-phase precipitate or dislocation loops now, increased. Distinguishing the precipitates and the loops in WBDF and BF images was difficult due to their equally small size. As a result, the total density and mean size was measured.

Figure 11 shows the total density in aged CF8 along with the loop density in the unaged CF8. Both density increased with ion fluence. The slope n of density versus ion fluence is lower for the aged CF8 ($n = 0.44$) than for the unaged CF8 ($n = 0.94$). The defect size in unaged specimen (mostly dislocation loops) increased with ion fluence. For the aged specimen, however, the defect size (a mixture of dislocation loops and G-phase precipitates) is relatively unchanged with increasing ion fluence.

G-phase precipitates can be distinguished from dislocation loops in TEM by dark field imaging with precipitate reflection. As shown in Ref. [15], the density of G-phase precipitates increased from 1.9×10^{22} to $2.7 \times 10^{22} \text{ 1/m}^3$ in the ferrite of aged CF8 after $6.3 \times 10^{14} \text{ ions/cm}^2$ fluence ($\sim 1 \text{ dpa}$) of 1 MeV Kr ion irradiation at 300°C. In this study, the total density of G-phase precipitates plus dislocation loops in the same specimen was measured with BF and WBDF to be $3.5 \times 10^{22} \text{ 1/m}^3$, indicating a dislocation loop density of $8.0 \times 10^{21} \text{ 1/m}^3$.

4. Discussion

4.1. The surface sink effects on the in-situ ion irradiation experiment

The surface sink effect is a unique characteristic of in-situ ion irradiation experiments in TEM. The specimens used in the in-situ experiments have a thickness of typically $\sim 100 \text{ nm}$. The surface of the TEM foil is effective sink for interstitials, vacancies and small mobile defect clusters. The kinetics and the spatial profiles of microstructural evolution would be significantly changed when point defects and small clusters are sufficiently mobile.

In bcc Fe and Fe-Cr, the motion of $\frac{1}{2}\langle 111 \rangle$ dislocation loops and their loss to the foil surface have been observed previously [8]. The Cr appeared to suppress the loop mobility [8]. In this study, detectable motion of dislocation loops were rarely observed with real time video using a Gatan 622 camera, indicating limited foil surface effect on visible dislocation loops. Apparently, the alloying elements (Cr, Ni, Si etc.) decreased the loop mobility similar to the Cr in Fe-Cr alloys.

For the point defects and tiny defect clusters (< 1 nm), their interaction with foil surface cannot be revealed by the video because of the insufficient TEM resolving power. Therefore, the areal density of loops or precipitates as a function of foil thickness was measured in order to understand the surface sink effect [23].

Figure 12 shows the areal density of dislocation loops and G-phase precipitates as a function of foil thickness in unaged and aged CF8 irradiated at 300°C. As shown in Figure 12 (A), the unaged CF8 specimen was measured in BF/WBDF and had a positive thickness intercept for 1.9×10^{14} ions/cm² (~ 0.3 dpa) and 6.3×10^{14} ions/cm² (~ 1 dpa), respectively, which indicates loss of point defects and small defect clusters to the foil surface [23]. A surface denuded zone of ~ 13 nm can be estimated for the unaged CF8 specimen irradiated to 1.9×10^{14} ions/cm² (~ 0.3 dpa). Since the density measurements shown in Figure 6 were obtained by directly dividing the areal density with the foil thickness without the consideration of foil surface effect, its value should be an underestimate to the loop density at the foil center.

The aged CF8 was measured in DF using G-phase reflection (Figure 12 (B)), which showed only G-phase precipitates, and in BF/WBDF (Figure 12 (C)), which showed both G-phase precipitates and loops. As shown in Figure 12 (B), before irradiation the data fit intercepts the origin. This is reasonable because the precipitates were uniformly distributed spatially and the areal density should be proportional to the foil thickness. At 6.3×10^{14} ions/cm² (~ 1 dpa), the thickness intercept remained close to the origin, which suggested that the foil surface had little effects on G-phase precipitate distribution under irradiation. As shown in Figure 12 (C), the data fit in BF/WBDF measurement also had a thickness intercept close to the origin for no irradiation, 1.9×10^{14} ions/cm² (~ 0.3 dpa) and 6.3×10^{14} ions/cm² (~ 1 dpa). The foil surface effect on the dislocation loops was not pronounced in aged CF8, either. The grown in G-phase precipitates may act as defect traps that competed with the foil surface for defects. In addition, the matrix strain field from the spinodal decomposition, although small, might hinder the migration of point defects and tiny defect clusters toward the foil surface.

4.2. Effects of irradiation and thermal aging on the microstructural evolution

The microstructural evolution under irradiation depends on the production and accumulation of irradiation defects, the diffusion of point defects, and the interactions between irradiation defects and pre-existing microstructure [18].

The initial sink/trap density in a material is an important factor that determines the microstructural evolution under irradiation.

For the ferrite, the irradiation temperature of 300°C is well beyond the temperature at which interstitials and vacancies become mobile (the stage III temperature in Fe is roughly -30°C [28]). Due to the high sink density of G-phase precipitates, the ferrite of the aged CF8 is subjected to interstitial and vacancy annihilation at sinks [29].

As shown in Figure 11, the defect evolution kinetics was slower in aged CF8 specimens ($n = 0.44$) than in un-aged CF8 specimens ($n = 0.94$). In addition, the defect size in the unaged specimen increased with ion fluence, while it was relatively constant in the aged specimen. It indicates that the pre-existing G-phase precipitates in the aged specimens acted as effective defect traps that enhanced the recombination of irradiation-induced point defects and slowed down the evolution of defect clustering. The different evolution rates of ferrite microstructure in the unaged and aged specimens suggest that irradiation-induced and aging-induced microstructural processes are not independent but interconnected. The large sink effect of G-phase precipitates introduced by thermal aging before irradiation may undermine the combined effect of two degradation mechanisms, thermal aging and irradiation, induced concurrently in a reactor environment.

The number density of G-phase precipitates increased in the ferrite of aged CF8 after irradiation (Figure 12 (B) and Ref. [15]) implies that the precipitation process was not complete by the end of the thermal-aging treatment (400°C for 10000 hours), and was accelerated under irradiation due to irradiation-enhanced diffusivity. This observation is qualitatively consistent with the results of aged CF8M irradiated with Fe ions at 300°C to 1 dpa [7].

In contrast to aged CF8, no apparent G-phase precipitates were observed in the unaged CF8 after irradiation. In literature, Ni/Si clusters were observed by APT in unaged CF8M (Ref. [7]) and in ferritic Fe-Cr steels (Ref. [12]) irradiated at about 300°C to a dose comparable this study. It is likely that Ni/Si clusters had formed in our unaged CF8 after irradiation, but had not developed into G-phase precipitates compositionally and crystallographically. Small Ni/Si clusters may not make changes on crystal structure, resulting in a lack of strain field and diffraction pattern for TEM observations [30]. The fact that the G-phase precipitation was observed in aged CF8 specimens but not in unaged specimens under the same irradiation condition may be attributed to the spinodal decomposition in the aged specimens. As was suggested by Danoix and Auger [31], the α - α' unmixing results in the rejection of Ni and Si from, respectively, α' and α domains, which facilitates G-phase precipitation at α - α' interface. The

unaged CF8, in contrast, lacks such nucleation sites and therefore may have higher barrier for G-phase precipitation.

The spinodal decomposition in the ferrite is another important factor affecting the degradation in mechanical properties of CASSs. In this study, it was difficult to characterize spinodal decomposition with TEM due to the very weak contrast. Previous studies have shown mixed irradiation effect on the spinodal decomposition in the ferrite. The spinodal decomposition was accelerated by neutron irradiation in unaged Fe-Cr ferrite and austenitic stainless steel welds [14, 32]. In contrast, ion irradiation was reported to reduce the spinodal decomposition in the aged CASSs [7, 13]. The conflicted observation may be attributed to the large variation in the dose rate between neutron and ion irradiation, which can result in distinct balance between competing micro-processes of irradiation-enhanced diffusivity and atomic mixing [13].

4.3. Comparison of the irradiation damage in the ferrite and the austenite

Ion irradiation at 300°C and 350°C induced a high density of dislocation loops in both the ferrite and the austenite in CF8. However, the defect accumulation with ion fluence was much earlier and faster in the austenite (fcc) than in the ferrite (bcc), which is consistent with literature observations in bcc and fcc metals [9, 33]. The more open crystal lattice structure of bcc compared to fcc may attribute to the lower cascade production efficiency of dislocation loops in irradiated ferrite [10]. In addition, small $\frac{1}{2}\langle 111 \rangle$ dislocation loops gliding to and annihilating at foil surface may also contribute to the lower loop density in the ferrite.

Unlike thermal aging where only the ferrite is subjected to change and is responsible for the embrittlement, irradiation causes microstructural modification in both the ferrite and the austenite. Therefore, attention needs to be paid to the austenite when evaluating the irradiation and its synergistic effects with thermal aging in CASSs and austenitic stainless steel welds. This is especially true for the tensile property because it was thought to be affected more by the phase of larger volume (i.e. the austenite) [34-35].

Conclusion

In-situ TEM observation revealed distinct microstructural evolution between the ferrite and the austenite in the as-received (unaged) CF8 irradiated with 1 MeV Kr at 300°C and 350°C to an ion fluence up to 1.9×10^{15} ions/cm² (~ 3 dpa). Irradiation produced a high number density of nanometer-sized dislocation loops in both austenite and ferrite.

Dislocation loops appeared in the austenite at a dose nearly one order of magnitude lower than in the ferrite, and at a given dose the density of dislocation loops was more than one order of magnitude higher in austenite than in ferrite. The mean sizes of dislocation loops were similar in both phases in unaged CF8 when irradiated at 300 and 350°C. At the end dose, the austenite formed a well-developed dislocation network microstructure, while the ferrite exhibited an extended dislocation structure as line segments.

In aged CF8, irradiation produced both dislocation loops and G-phase precipitates in the ferrite. The total number density of nanometer-sized dislocation loops and G-phase precipitates in the aged specimen was significantly higher than that in the unaged specimen. The increase rate of defect density and size was slower in the aged specimens than in the unaged specimens.

Acknowledgments

This work was supported by Electric Power Research Institute. The ion irradiation and microscopy was accomplished at Argonne National Laboratory at the IVEM-Tandem Facility, a user facility funded by the US Department of Energy Office of Nuclear Energy, operated under Contract No. DE-AC02-06CH11357 by UChicago Argonne, LLC. Yiren Chen at ANL is thanked for providing the materials, helpful discussion, and conducting the paper review.

Figure 1. The optical image of the aged CF8 specimen. The grey area is ferrite and the bright area is austenite. The label Fer and Aus correspond to the ferrite and the austenite, respective.

Figure 2. Quick damage calculation showing the depth profile of the irradiation damage and the Kr ion deposition in CF8 stainless steel irradiated by 1 MeV Kr

Figure 3. Bright field TEM micrographs of the ferrite in (A) unaged and (B) aged CF8 austenitic stainless steels. The dark line contrasts in (A) and (B) are dislocations. The dark dot contrasts in (B) are G-phase precipitates. The images were taken under $g = 110$ near 001 zone.

Figure 4. The microstructural evolution of the same specimen area of a ferrite in an unaged CF8 irradiated with 1 MeV Kr at 300°C. Images were taken under WBDF $g = 110$ close to $(1\bar{1}1)$ zone.

Figure 5. The microstructural evolution of the same specimen area of the ferrite in an unaged CF8 irradiated with 1 MeV Kr at 350°C. Images were taken under WBDF $g = 110$.

Figure 6. The density and size of defect clusters as a function of irradiation fluence in unaged CF8 irradiated with 1 MeV Kr at 300 and 350°C.

Figure 7. The microstructure of the (A) ferrite and (B) the austenite of unaged CF8 irradiated with 1 MeV Kr at 300°C to 1.9×10^{15} ions/cm² (~3 dpa). The ferrite image (A) was taken under $g = 110$ close to (111) zone, and the austenite image (B) were taken under $g = 111$ close to (112) zone.

Figure 8. The microstructural evolution of the austenite in an unaged CF8 irradiated with 1 MeV Kr at 300°C. Images were taken under WBDF $g = 200$.

Figure 9. The microstructural evolution of the austenite in an unaged CF8 irradiated with 1 MeV Kr at 350°C. Images were taken under WBDF and BF $g = 200$.

Figure 10 The WBDF images of the same specimen area of an aged CF8 irradiated with 1 MeV Kr at 300°C.

Figure 11. Volume density and size measurements of total defects in the ferrite of aged and unaged CF8 irradiated by 1 MeV Kr at 300 °C. Value n of the slopes is shown. The initial volume density and the mean size of total defects observed in aged CF8 specimen are 2.1×10^{22} 1/m 3 and 2.9 nm, respectively.

Figure 12. The areal density of (A) dislocation loops (B) G-phase precipitates only and (C) G-phase precipitates plus dislocation loops as a function of the foil thickness in the ferrite of unaged and aged CF8 specimens irradiated with 1 MeV Kr at 300°C. The line is the linear least square fit to the data points.

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