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Effect of heavy ion irradiation on microstructural evolution in CF8 cast austenitic stainless steel

Wei-Ying Chen [a, *], Meimei Li [a], Marquis A. Kirk [a], Peter M. Baldo [a], Tiangan Lian [b]

[a] Argonne National Laboratory, Argonne, IL 60436, USA
[b] Electric Power Research Institute, Palo Alto, CA 94304, USA

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ABSTRACT

The microstructural evolution in ferrite and austenitic 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/cm^2 (~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.

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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/cm^2 [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 ½<111> and immobile <100> 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

* Corresponding author.
E-mail address: wychen@anl.gov (W.-Y. Chen).

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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 h. 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 Fig. 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 ±3 °C.

The specimens were irradiated to an ion fluence of 6.3×10^{14} ions/cm^2 (~1 dpa) with an ion flux of 1.6×10^{11} ions/cm^2/sec (~0.00025 dpa/s), and to a high fluence with an increased ion flux of 6.2×10^{11} ions/cm^2/sec (~0.001 dpa/s). The final fluence was 1.9×10^{15} ions/cm^2 (~3 dpa). As shown in Fig. 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/cm^3 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/cm^2/s (~0.000025 dpa/s) to a fluence of 1.9×10^{14} ions/cm^2 (~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/cm^2, D is the damage rate in vacancies/ion/Å and N is the atomic number density in atoms/cm^3. An average damage rate of 1.3 vacancies/ion/Å was estimated according to the SRIM calculation in Fig. 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

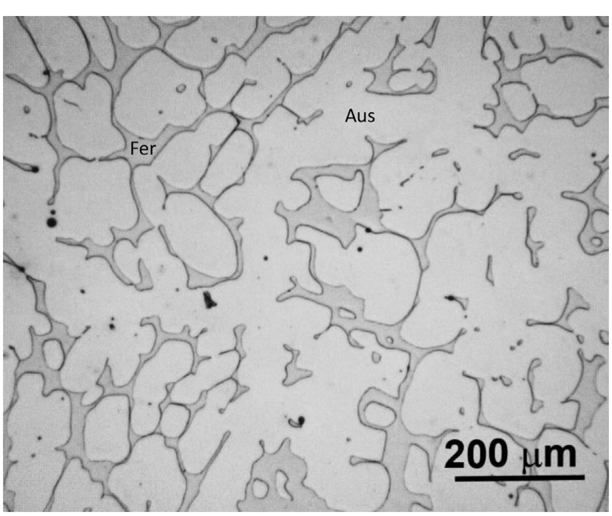

Fig. 1. The optical image of the aged CF8 specimen. The gray area is ferrite and the bright area is austenite. The label Fer and Aus correspond to the ferrite and the austenite, respective.

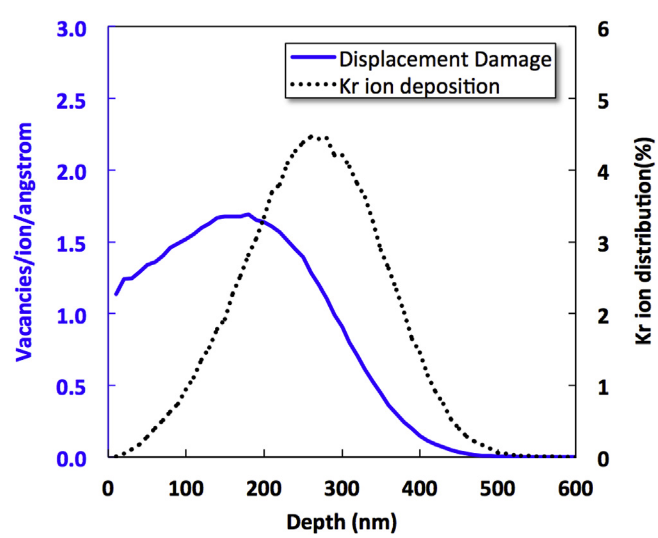

Fig. 2. Quick damage calculation showing the depth profile of the irradiation damage and the Kr ion deposition in CF8 stainless steel irradiated by 1 MeV Kr.