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Dislocation loop formation in model FeCrAl alloys after neutron irradiation below 1 dpa☆

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ABSTRACT

FeCrAl alloys with varying compositions and microstructures are under consideration for accident-tolerant fuel cladding, but limited details exist on dislocation loop formation and growth for this class of alloys under neutron irradiation. Four model FeCrAl alloys with chromium contents ranging from 10.01 to 17.51 wt % and aluminum contents of 4.78 to 2.93 wt % were neutron irradiated to doses of 0.3–0.8 displacements per atom (dpa) at temperatures of 335–355 °C. On-zone STEM imaging revealed a mixed population of black dots and larger dislocation loops with either $a/2\langle 111\rangle$ or $a\langle 100\rangle$ Burgers vectors. Weak composition dependencies were observed and varied depending on whether the defect size, number density, or ratio of defect types was of interest. Results were found to mirror those of previous studies on FeCrAl and FeCr alloys irradiated under similar conditions, although distinct differences exist.

1. Introduction

High-temperature oxidation resistance has been identified as a key material characteristic needed for accident-tolerant fuel (ATF) claddings for commercial light water reactor (LWR) operation [1,2]. FeCrAl alloys have been shown to exhibit the necessary high-temperature oxidation resistance, with oxidation rates three orders of magnitude or more slower than those of currently deployed zirconium-based alloys at temperatures of 1050 °C–1500 °C [3,4]. This excellent oxidation resistance at elevated temperatures in the FeCrAl alloys class occurs when specific concentrations of chromium (Cr) and aluminum (Al) are contained within the alloy, giving them the ability to form a uniform α-Al_2O_3 layer during exposure [5]. Although the oxidation resistance of the FeCrAl alloy class has led to their adoption as candidate ATF claddings, the alloys must also have reasonable radiation tolerance.

Several studies have already been conducted investigating the radiation tolerance of FeCrAl alloys [6–11]. These studies have shown the behavior of FeCrAl alloys to closely mimic the behavior of more traditional FeCr alloys, including the formation of dislocation loops with Burgers vectors of either $a/2\langle 111\rangle$ or $a\langle 100\rangle$ and precipitation of the Cr-rich α' phase under neutron irradiation. As in FeCr alloys, the formation of these radiation-induced/enhanced microstructural features leads to hardening and loss of ductility in FeCrAl alloys. The severity of the hardening response has been directly linked to both the sizes and number densities of both the dislocation loops and α' phase [6].

Previous studies have established the initial groundwork for determining the radiation tolerance of FeCrAl alloys; however, little detail exists on the nucleation of these defects and their subsequent growth under neutron irradiation, as most studies have been conducted above 1 dpa. Determining the defect production and evolution—including the rate of change in the size and number

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density, as well as defect-sink interactions—will enable further optimization of the FeCrAl alloys class for radiation tolerance. Furthermore, low-dose irradiations provide easier benchmarks for advanced modeling and simulation techniques, as reaching significantly higher doses can drastically increase the computational expense and/or reduce the fidelity of these techniques. Future modeling and simulation efforts could lead to predictive models for the radiation hardening and embrittlement of FeCrAl alloys.

This work evaluates the dislocation loop morphologies in four model FeCrAl alloys after elevated-temperature (334–355 °C) neutron irradiation below 1 dpa in the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory. Scanning transmission electron microscopy (STEM) was used to assess the microstructures of dislocations resulting from neutron irradiation. The use of four candidate alloy compositions enables the determination of any composition dependence on the formation of the radiation-induced/enhanced defects. Additionally, combining this study with previous higher-dose studies provides further insight into the subsequent growth of dislocation loops.

2. Materials and methods

The same model alloys investigated in previous studies [5–8,12] were used in this study. The model alloys have the designations F1C5AY, B125Y, B154Y-2, and B183Y-2, which have nominal Cr contents of 10.01 11.96, 15.03, and 17.51 wt %, respectively, and Al contents of 4.78, 4.42, 3.92, and 2.93 wt %, respectively. The alloys represent a series of FeCrAl alloys with yttrium additions (see Table 1) that span compositions known to be oxidation resistant in high-temperature steam [5] without complicated chemistry introduced via minor alloying additions. All specimens were received with 10% cold-working introduced after hot forging, rolling, and heat treatment, resulting in a grain size of 20–30 μm and a line dislocation density of $6.3 \pm 1.0 \times 10^{13}$ m^{-2} to $1.5 \pm 0.7 \times 10^{14}$ m^{-2} [6].

Wire electric discharge–machined dog-bone–shaped SS-J2 specimens with a gauge section of 5.0 × 1.2 × 0.5 mm [6] were subjected to low-dose HFIR irradiation during cycle 450b of its operation. HFIR irradiations for the F1C5AY and B183Y-2 alloys were conducted to a damage dose of 0.8 dpa with a nominal capsule average specimen temperature of 355.1 ± 3.4 °C. The B125Y and B154Y-2 specimens were irradiated to a damage dose of 0.3 dpa with a nominal capsule average specimen temperature of 334.5 ± 0.6 °C. All specimens were irradiated with a dose rate of 7.7×10^{-7} dpa/s. Capsule average specimen temperatures were determined from dilatometric analysis of silicon carbide (SiC) specimens co-irradiated with the tensile specimens. Median average temperatures from the dilatometric analysis were determined using the algorithms developed within Campbell et al. [13]; error is reported as one standard deviation of the mean for at least three separate SiC samples per capsule.

Tensile testing of the nonirradiated and irradiated specimens has been previously reported [6,9]; here, the broken tensile heads from those studies were used for microstructural characterization. Broken tensile heads were mechanically polished using standard procedures to produce flat, damage-free surfaces for focused ion beam (FIB) sample preparation. FIB was performed on an FEI Quanta 3D 200i FIB using typical lift-out procedures. FIB lift-out specimens were extracted from the widest portion of the tensile head (far from the gauge-head transition) resulting in specimens free of deformation [14]. All electron transparent samples were finished with a 5 kV low-energy rastered gallium ion beam followed by a 2 kV beam to remove any surface damage to the specimens induced during subsequent sample thinning procedures.

Dislocation microstructures were determined using on-[100] zone axis STEM. Orientating a body-centered-cubic (BCC) specimen toward the [100] zone axis results in imaging of $a/2\langle 111\rangle$ dislocation loops as open ellipses, whereas $a\langle 100\rangle$ dislocation loops are either edge-on or in-plane; the result is stark line contrast or diffuse contrast of circular/faceted loops, respectively [15]. STEM enables all **g**-vectors to be excited simultaneously, allowing all possible Burgers vectors to be imaged at once, while also relaxing the $\mathbf{g}\cdot\mathbf{b}$ criterion and enabling in-plane $a\langle 100\rangle$ loops to be imaged as well [16]. The result is easily distinguishable Burgers vectors based on dislocation loop morphology with micrographs containing a strong signal-to-noise ratio, and no introduction of systematic error because of the $\mathbf{g}\cdot\mathbf{b}$ invisibility correction factors.

On-[100] STEM imaging was completed using simultaneous bright-field (BF) and annular dark-field (ADF) detection on a JEOL JEM-2100F field emission gun STEM operating at 200 kV. Specimen thicknesses for number density measurements were determined using convergent beam electron diffraction (CBED) techniques [17]. Slopes of areal-density versus foil thickness plots were used to account for sample thickness variability [18–20].

3. Results

Example micrographs of the radiation-induced microstructure for each alloy are given in Fig. 1. Fig. 1 shows dislocation loop morphologies consistent with those expected of dislocation loops with Burgers vectors of both $a/2\langle 111\rangle$ (open ellipses) and $a\langle 100\rangle$ (stark, straight, edge-on line segments and faint faceted circles), as well as the presence of dislocation networks. Systematic two-beam imaging was not completed to identify whether the observed loops were interstitial or vacancy in nature. Small ($\lesssim$10 nm), unidentifiable dislocation loops based on morphology were also found randomly distributed throughout the matrix; they are classified here as “black dots.” Additionally, previous studies of the same irradiated specimens found precipitation of the Cr-rich α' randomly distributed in the matrix using small-angle neutron scattering (SANS) and atom probe tomography (APT) [8]. The α' was not visible in either the BF- or ADF-STEM imaging modes. Imaging of α' using either diffraction-based or Z-contrast imaging was not expected due to the similar lattice parameters [21] and average atomic numbers [8] between α' and the matrix, respectively. As expected based on previous studies [6,22], no cavity-induced swelling was observed in any specimen analyzed after the low-dose neutron irradiation. A limited effect on the dislocation loop

Table 1
Summary of model FeCrAl alloy compositions in weight percent (wt %).

Alloy	Composition (wt %)									
	Fe	Cr	Al	Y	C	S	O	N	P	Si
F1C5AY	85.15	10.01	4.78	0.038	0.005	0.001	0.0013	0.0003	0.006	<0.01
B125Y	83.56	11.96	4.42	0.027	0.005	0.0013	0.0017	0.0009	0.0	0.01
B154Y-2	80.99	15.03	3.92	0.035	0.005	0.0004	0.0025	0.0007	<0.002	0.01
B183Y-2	79.52	17.51	2.93	0.017	0.005	0.0006	0.0015	0.0011	<0.002	<0.01