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Complementary Techniques for Quantification of α′ Phase Precipitation in Neutron-Irradiated Fe-Cr-Al Model Alloys

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The substandard performance of Zircaloy LWR cladding materials under loss-of-coolant accident (LOCA) conditions has prompted the search for a more well-suited material for these conditions. Initial investigations of Fe-Cr-Al alloys have demonstrated their superior high temperature oxidation and corrosion resistance compared to Zr-based alloys [1]. However, questions still remain regarding the radiation tolerance of Fe-Cr-Al alloys which, similar to other high-Cr ferritic alloys, are susceptible to embrittlement due to the precipitation of a Cr-rich α′ phase.

Quantification of α′ phase precipitation has historically been limited to bulk average analysis using small angle neutron scattering (SANS) techniques, as no contrast is seen using conventional TEM imaging techniques due to the semi-coherency of the α′ phase with the α-Fe matrix [2]. However, the advent of local electrode atom probe tomography (APT) and improvements in STEM/EDS chemical mapping have allowed for a more localized investigation of the morphology and composition of these precipitates. Coupling of these techniques allows one to overcome the individual limitations of each approach, resulting in a more comprehensive determination of precipitate evolution and structure. A greater understanding of the mechanisms underlying this precipitation phenomenon allows for a more informed alloy development process as well as the formulation of increasingly robust predictive models of radiation response.

This work presents the results of a multifaceted study of precipitate morphology in four model Fe-Cr-Al alloys with compositions ranging from 10-18 wt.% Cr and 2.9-4.8 wt.% Al irradiated to several nominal damage dose levels up to 7 displacements per atom (dpa) in the High Flux Isotope Reactor (HFIR). Sample forging and heat treatment details can be found in Field *et al.* [3]. SS-J2 sub-sized tensile specimens were cut from the resulting materials and irradiated at a nominal target temperature of 320°C. Following room temperature tensile testing, SANS data was collected using the broken half-tensile specimens, resulting in measurements spanning a momentum transfer (Q) range of $0.01 < Q < 10$ nm^{-1}. Materials for APT and TEM investigations were prepared from the broken half-tensile heads (grip areas where no deformation occurred) using FIB liftout techniques. Atom probe data was collected in a Cameca LEAP 4000X HR operated in laser mode, and STEM/EDS spectral imaging was performed on the FEI Talos F200X S/TEM located at the ORNL LAMDA laboratory.

Clustering in the atom probe data was quantified using the maximum separation method [4]. Overlap of peaks at 27 and 54 Da in the time-of-flight (TOF) spectrum was accounted for using manual peak decomposition with assumed isotopic abundances based on calculations using the ORIGEN-2.2 isotope generation and depletion code to account for neutron transmutation effects. Figure 1 shows Cr atom maps observed in the APT analysis demonstrating how precipitate morphology changes with

composition in the 7 dpa Fe-Cr-Al specimens [5]. SANS quantification assumes pure nuclear scattering with clusters assumed to be spheres interacting with an exclusion volume, with scattering contrast determined using composition data from the APT analysis. Comparisons of SANS scattering intensities between dose conditions and compositions are shown in Figure 2. Finally, a composite STEM/EDS micrograph for Fe-18Cr-2.9Al illustrating a homogeneous nucleation of precipitates in the presence of dislocation loops is shown in Figure 3.

In short, APT has allowed for very in-depth assessment of composition and spatial distribution of precipitates in a very small volume. SANS demonstrated that this understanding can be extrapolated to bulk average morphologies, but requires the composition data generated by APT. Finally, STEM/EDS showed no evidence of precipitate interaction with dislocations and other defects that cannot be easily seen using the other methods. As such, this research demonstrates that the coupling of these techniques results in a much broader understanding of the precipitation phenomena observed in Fe-Cr-Al alloys [6].

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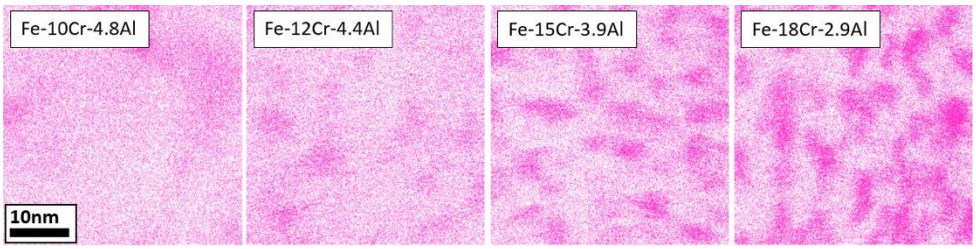

Figure 1. 40nm×40nm×20nm APT Cr atom maps showing signs of Cr-rich α′ precipitation in all Fe-Cr-Al compositions irradiated to 7 dpa at 320°.

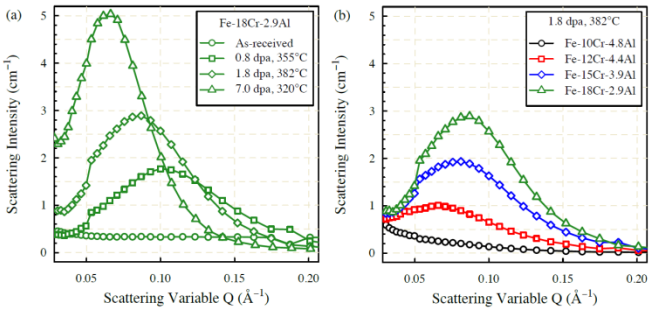

Figure 2. Comparison of scattering intensities for (a) varied irradiation conditions for the Fe-18Cr-2.9Al composition and (b) varied Fe-Cr-Al compositions irradiated to 1.8 dpa at 382°C. Adapted from [3].

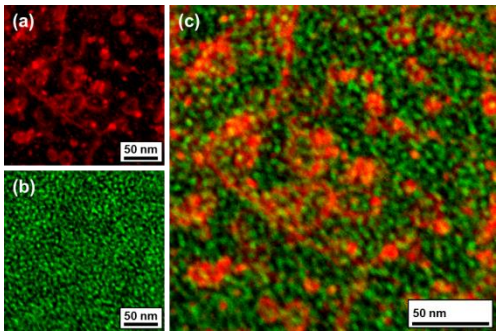

Figure 3. (a) STEM-ADF image, [110] on-zone. (b) STEM/EDS map for Cr-K_α x-rays. (c) Color overlay of ADF and EDS map. All images from Fe-18Cr-2.9Al, 7.0 dpa, 320°C.

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