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Variability in Chromium Segregation at Lath Boundaries in Proton-Irradiated 9 wt.% Cr Model Steel Determined by Quantitative X-Ray Mapping

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Ferritic-martensitic (F/M) steels are under consideration as structural materials for advanced nuclear reactors. F/M steels are preferred for advanced reactor designs over current austenitic stainless steels due to their superior resistance to swelling while maintaining excellent thermal properties [1]. One matter of concern is that radiation-induced segregation (RIS) may occur at grain boundaries. Segregation of Cr at grain boundaries, either alone or by initiating the nucleation and growth of embrittling precipitate phases, can impact the mechanical properties of the steel. Previous work on F/M steels presents no conclusive trends in the RIS behavior at grain boundaries [2]. It is proposed that a major reason for the apparent inconsistency in RIS behavior is variability in the presence of precipitates, grain boundary orientation and defects from boundary to boundary. These microstructural variations at boundaries may lead to differences in microchemisty among boundaries in F/M steels. In this investigation, quantitative compositions extracted from energy-dispersive X-ray spectroscopy (EDS) spectrum images are used to provide insight on how variations in local microstructure affects microchemistry at lath boundaries in irradiated F/M steels.

The steel used in this work is a 9 wt.% Cr model alloy designed to approximate the microstructure of commercially available steels but having a simplified chemistry. The composition of the steel is 0.015 wt.% O, 0.72 wt.% C, 8.68 wt.% Cr, balance Fe. Sample coupons were irradiated up to 3 displacements per atom (dpa) using 2.0 MeV protons at 400°C. Disk samples were extracted from irradiated coupons and prepared using standard dimpling, electro-polishing and ion-milling techniques. With the use of a Philips CM200-FEG operated in scanning transmission electron microscopy (STEM) mode and equipped with an EDAX EDS detector and Emispec Vision system, drift-corrected spectrum images of 32 x 64 1.5-nm pixels were acquired with an incident probe size of ~1.5 nm full width at half-maximum (FWHM) and a dwell time of 1 s/pixel. Quantitative Fe and Cr contents were generated using the Cliff-Lorimer method with experimentally determined 'k' factors [3].

Four lath boundaries were investigated per irradiation condition. Only one lath boundary in material irradiated to 1 dpa showed Cr depletion at the boundary, as seen in Fig. 1. All other boundaries in the 1 dpa condition showed no segregation of Cr at the boundary. All lath boundaries in material irradiated to 3 dpa showed slight enrichment of Cr. Fig. 2 shows the composition enrichment profiles which were extracted from spectrum images by binning data over 5 pixels parallel to the boundary for the material irradiated to 3 dpa. For the limited number of lath boundaries investigated in the model steel, it was found the enrichment of Cr at the lath boundaries increases with increasing dose at 400°C.

Quantitative EDS mapping allows for a direct coupling between diffraction contrast images and distribution of Cr in the sample. This provides insight on how changes along and around the grain boundary affect the Cr segregation at the boundary. It was found that Cr-rich carbides on lath boundaries altered segregation behavior in the 1 dpa condition but not in the 3 dpa condition, but due to the low number of lath boundaries that were characterized it is difficult to determine any conclusive trends. Results for each irradiation condition also suggest minor variability in the Cr segregation response between lath boundaries with similar microstructure as observed in annular dark field (ADF) images. From the observations, it appears that not only Cr-rich carbides but also grain boundary crystallography (misorientation and habit plane) must be key factors driving the varying responses observed at lath boundaries. Composition mapping by STEM-EDS spectrum imaging provides greater insight than one-dimensional profiles on the link between microstructure and microchemistry in irradiated F/M steels but with additional analysis of grain boundary orientation an even more complete understanding of RIS responses in F/M steels is expected [4].

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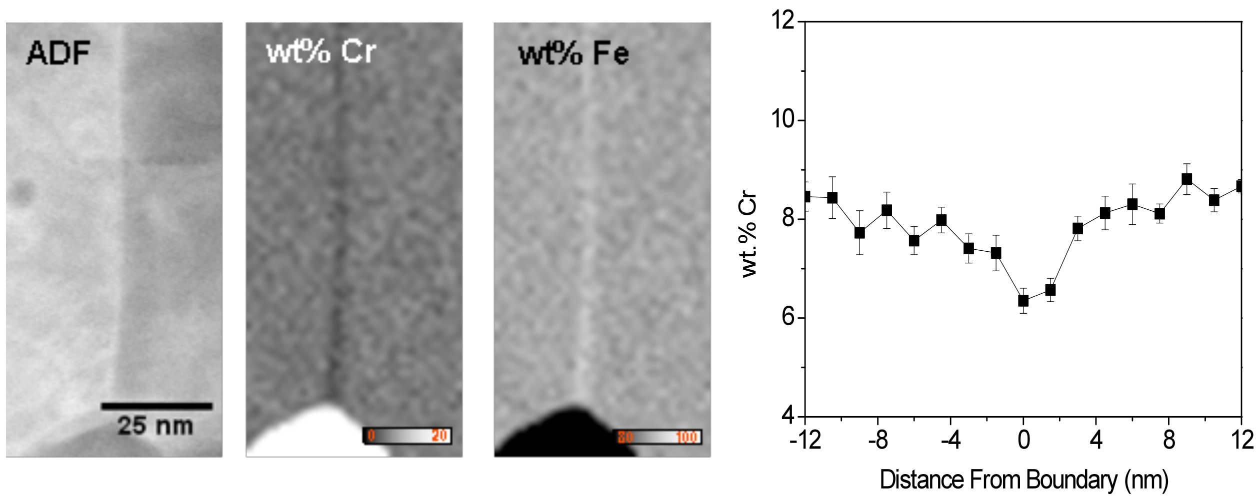

FIG. 1. ADF image of a lath boundary (1 dpa, 400°C) with corresponding wt.% Cr and Fe maps. Plot shows line profile extracted from Cr concentration map averaged over 5 pixels parallel to boundary.

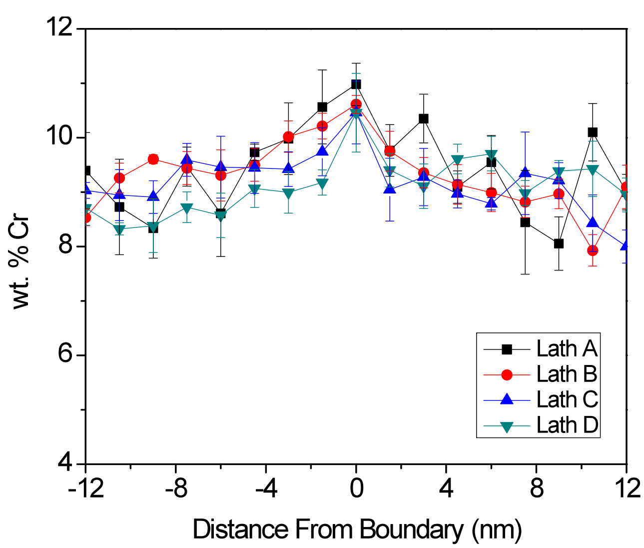

FIG 2. Extracted Cr line profiles (averaged over 5 pixels parallel to boundary) across lath boundaries in material irradiated to 3 dpa at 400°C. Plot shows Cr enrichment at all lath boundaries.

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