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AEM AND AES OF RADIATION-INDUCED SEGREGATION IN PROTON-IRRADIATED STAINLESS STEELS

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Irradiation-assisted stress corrosion cracking (IASCC) of stainless steels has been attributed in part to radiation-induced segregation (RIS) of both major alloying and impurity elements at grain boundaries. There are phenomenological similarities observed between IASCC and intergranular stress corrosion cracking (IGSCC) of thermally-sensitized stainless steels. One concern for both IGSCC and IASCC is the localized loss of corrosion resistance associated with chromium depletion at grain boundaries. In order to avoid complications related to the long-term, induced radioactivity of neutron-irradiated specimens, four type 304L alloys were irradiated to 1 dpa (displacements per atom) with 3.4 MeV protons at 400°C. Both analytical electron microscopy (AEM) in a Philips EM400T/FEG and Auger electron spectrometry (AES) in a Perkin Elmer (PHI) 660 were employed to measure composition at or near grain boundaries in unirradiated and irradiated specimens of four controlled purity alloys [ultra-high purity (UHP), UHP+S (0.03 at.%), UHP+P (0.08 at.%), and UHP+Si (0.87 at.%)]. A sufficient number of boundaries were analyzed via AEM and AES to result in the standard deviation of the mean boundary composition of less than ~0.5 at.%. Further experimental details are presented elsewhere.¹

Grain boundary RIS profiles typical for the irradiated alloys are presented in Fig. 1. The major alloying elements (i.e., Cr and Ni) exhibit consistent behaviors in all four alloys, in that chromium was depleted near the boundary; whereas Ni was enriched. Manganese concentrations in the alloys are low (~1.2 at.%) and there is an overlap between the small Mn K_{α} peak with the larger Cr K_{β} peak. However, there were reproducible differences in the apparent Cr K_{α} / K_{β} ratio and position of the Cr K_{β} /Mn K_{α} composite peak for spectra acquired at and away from the boundaries, indicating that there were consistent changes in the manganese levels relative to the grain boundary. The intensities of the overlapped Mn K_{α} /Cr K_{β} peaks were deconvoluted based on a fixed Cr K_{α} / K_{β} ratio, which was confirmed from a pure chromium standard. Though the precision in the calculated Mn concentration was poorer than that for the other major alloying elements, it was sufficient to consistently indicate a significant depletion of manganese (2-3 fold) at grain boundaries in all four irradiated alloys. The relative depletion of manganese by RIS was greater for manganese than it was for chromium. The nickel RIS profiles were narrower than that of chromium; whereas the manganese RIS profiles tended to be slightly wider.

The exact magnitude of the RIS of the major alloying elements is dependent on the impurity level of the specific alloy (Fig. 1). The measured RIS in the UHP alloy was relatively small. Averaged over a number of boundaries, the enrichment of nickel was ~2 at.% and the depletion of chromium and manganese were ~3.5 and ~0.4 at.%, respectively. A slight phosphorus enrichment at boundaries was observed in the irradiated UHP and was attributed to the residual phosphorus present in the alloy (~0.01 at.%). For the UHP+P (and the UHP+S), the nickel enrichment and the chromium and manganese depletion averaged approximately 3.1, 4.7, and 0.8 at.%, respectively. Though there was phosphorus enrichment induced by RIS in the UHP+P (~1.4 at.%), no detectable change in sulfur level was indicated in the UHP+S, even by AES. A slight phosphorus enrichment at grain boundaries was observed in the unirradiated UHP+P and was attributed to equilibrium segregation of phosphorus during thermomechanical treatment of the alloy. A further increase in the degree of RIS was observed in the UHP+Si; nickel and silicon were enriched by ~4.5 and 2.3 at.%, respectively, and chromium and manganese were depleted by ~6 and 0.7 at.%. Similar or slightly higher degrees of RIS at the grain boundaries were indicated by AES. However, the UHP+Si material could not be fractured intergranularly, thus preventing AES.

As a result of the narrow RIS profiles (<20 nm width) at grain boundaries induced in these materials by low temperature irradiation and the finite size of the excited volume for X-ray microanalysis, the measured profiles are convolutions of these two factors. Closer approximations of the actual composition profiles were estimated by comparing measured profiles with simulated RIS profiles convoluted with an X-ray generation

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function. The simulated RIS profiles were calculated from a model of the point defect fluxes and solute interactions with those fluxes.² The x-ray generation function was estimated from the measured incident probe size and the degree of beam broadening characteristic of the foil thickness specific for the measured profile. The broadening was estimated by a Monte Carlo simulation of electron scattering.³ The RIS profiles estimated in this manner indicated slightly increased levels of segregation for Cr, Fe, and Ni as compared to that measured via AEM and similar to that measured via AES. However, the large difference in measured grain boundary phosphorus levels from AEM and AES (1.4 and 8.7 at.%, respectively) indicates that the phosphorus is concentrated in a very narrow region at the boundary, approaching a monolayer.⁴

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FIG. 1.--RIS profiles at high-angle grain boundaries measured by X-ray microanalysis in a) irradiated UHP, b) UHP+P, c) UHP+S, and d) UHP+Si alloys. Arrows indicate ordinate axis used.

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