RADIATION-INDUCED SEGREGATION IN AUSTENITIC STAINLESS STEELS

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Radiation-induced segregation (RIS) is driven by fluxes of point defects to sinks. RIS can induce composition fluctuations in irradiated stainless steels, which can result in microstructural and property changes, including precipitation, austenite instability, strengthening, embrittlement, and irradiation-assisted sensitization and stress corrosion cracking. Analytical electron microscopy (AEM) provides a powerful technique to study such segregation. RIS in several irradiated stainless steels has been investigated. AEM was performed in a Philips EM400T/FEG equipped with an EDAX 9100/70 analysis system. The specimens were neutron irradiated to 15 displacements per atom (dpa) at 520 °C in the Fast Flux Test Facility (FFTF) and were only mildly radioactive ($<50 \mu\text{Ci} \equiv 1.85 \text{ MBq}$), thus permitting high spatial resolution X-ray microanalysis to be employed. Typical acquisitions were performed for 100 s in the STEM mode with $<2\text{-nm}$ -diam probes containing $>0.5 \text{ nA}$ current. Subtraction of "in-hole" spectra from the measured spectra corrected for both the normal "in-hole" counts and those associated with the radioactivity of the specimen.

A curved grain boundary in an irradiated, Ni-stabilized steel is shown in Fig. 1(a). The high local curvature of the boundary and the lower dislocation density to its right indicate that this boundary migrated to the left during the irradiation. Fig. 1(b) shows the RIS profiles measured at this boundary. In the region swept relatively clear of dislocations, nickel is enriched and chromium is depleted to $\sim 11 \text{ at.}\%$, as a result of RIS to the boundary. There is a narrow region $\sim 70 \text{ nm}$ wide to the left of the boundary which is depleted of nickel and slightly enriched in chromium. The observed nickel depletion in this zone arises from RIS of nickel to the boundary from the left, whereas chromium rejected from the boundary region diffuses ahead of the migrating boundary.

Figure 2(a) shows three nearly edge-on dislocation loops (A-C) in an irradiated, Ni-stabilized steel with diameters of ~ 100 , 50 , and 25 nm , respectively. Their relative sizes indicate that loop A probably nucleated first, followed by loop B, and then loop C. Profiles were measured perpendicular to loops A and B across their centers. For loop A, nickel and silicon are enriched at the loop, whereas chromium and iron are depleted [Fig. 2(b)]. The magnitude of the composition change at the fault plane is noteworthy, as nickel exceeds iron. However, as the distance from the loop increases, the magnitude of the segregation decreases and at $\sim 40 \text{ nm}$, the profiles cross the level of the unperturbed matrix. This effect is a little clearer to the right of loop A where the RIS profiles are not disturbed by RIS to other defects. The origin of this reversal in segregation is twofold — the conservation of solute and limited diffusion distances. Solute which segregates to the loop must be removed from the adjacent matrix producing a depleted zone just beyond the enriched zone. The opposite effect occurs for solute removed from the vicinity of the boundary. The RIS profile for loop B is given in Fig. 2(c), which surprisingly indicates slight depletion of silicon and nickel and enrichment in iron. The general trend is for silicon and nickel to increase and for chromium and iron to decrease as one approaches loop A. As loop B is $\sim 40 \text{ nm}$ from loop A, it is growing in the zone of reversed segregation associated with loop A. Similar effects are observed for loop C. The RIS profiles near loop B are dominated by the point defect fluxes to loop A. As such, loop A perturbs the matrix composition out to at least 70 nm from the fault plane.

Figure 3(a,b) indicates the RIS profiles measured at a migrating boundary in an irradiated, Mn-stabilized EP838 steel. The asymmetry of the Mn and Fe profiles is the result of the motion of the boundary from right to left. In contrast to the Ni-stabilized steel (Fig. 1), manganese and molybdenum are strongly depleted and there is only a slight depletion of chromium at the boundary, whereas nickel, silicon, and iron are enriched. The local RIS zone width at the boundary is $\sim 10 \text{ nm}$ (FWHM). In the region swept by the migrating boundary, similar depletions or enrichments to those at the boundary are observed, though at reduced levels. The low degree of chromium depletion for the EP838 steel relative to the Ni-stabilized steel may result in a greater resistance to radiation-induced sensitization. Manganese depletion at grain boundaries may induce a loss of austenite stability, but the effect will be offset by the nickel enrichment.¹

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FIG. 1.--(a) Curved, migrating grain boundary in FFTF-irradiated USPCA steel (b) RIS profiles. FIG. 2.--(a) Nearly edge-on dislocation loops (A-C) in FFTF-irradiated USPCA steel (b) RIS profiles at loop A. (c) RIS profiles at loop B. FIG. 3.--(a,b) RIS profiles at boundary in FFTF-irradiated EP838 steel.