IRRADIATION-INDUCED MICROSTRUCTURAL CHANGES IN ALLOY X-750

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Alloy X-750 is a γ' -strengthened Ni-base alloy that is often used in nuclear power systems for its excellent corrosion resistance and good mechanical properties.¹ However, radiation-induced segregation (RIS) and irradiation-assisted stress corrosion cracking (IASCC) can occur under neutron irradiation.² The present study examines the microstructure and composition profiles in a heat of Alloy X-750 before and after neutron irradiation.³

The material was in the HTH commercial heat treatment (1h at 1121°C + air cooling + 20h at 704°C). Specimens were prepared from both control material and material irradiated at 360°C to a fluence of 2.3×10^{20} n/cm² ($E > 1$ MeV), which was estimated to produce ~ 0.4 displacements per atom. High spatial resolution analytical electron microscopy was performed in a Philips EM400T/FEG operated in the STEM mode with a probe diameter of ~ 1.4 nm (FWHM). Composition profiles were acquired in two different ways: all energy dispersive X-ray spectrometry (EDS) and some parallel-detection electron energy-loss spectrometry (PEELS) profiles were acquired as individual spot analyses, whereas the majority of the PEELS profiles were acquired as spectrum lines with ~ 10 s acquisition times per point. The quoted compositions are as measured (wt%); no deconvolution for the effect of the excited volume was performed.

The control material exhibited a complex, multiphase microstructure including fine (~ 20 nm) intragranular γ' precipitates along with several intergranular phases. Some regions of intergranular precipitation exhibited $M_{23}C_6$ and γ' precipitates (e.g., Fig. 1), whereas others exhibited $M_{23}C_6$ and a co-precipitate containing both $M_{23}C_6$ and a structurally-related phase (Fig. 2). This precipitate had overall electron diffraction patterns consistent with the $M_{23}C_6$ structure. Some regions exhibited a composition (>85 Cr, ~ 3 Fe, ~ 7 Ni) consistent with $M_{23}C_6$, whereas other regions exhibited a distinctly different composition (~ 7 Ti, ~ 2 Fe, ~ 2 Cr, ~ 2 Nb, and >80 Ni). Convergent beam electron diffraction indicated that the Cr-rich and Ni-rich regions exhibited very similar, parallel-oriented diffraction patterns. PEELS light element analysis indicated that the chromium-rich phase contains carbon with little or no boron; whereas the nickel-enriched phase contains boron with little or no carbon. This analysis identified the Ni,Ti-enriched phase as the τ boride ($M_{23}B_6$) which is isostructural with $M_{23}C_6$. The structurally similar boride forms cube-on-cube with the $M_{23}C_6$ and appears as a single, homogeneous precipitate under most diffracting conditions. However, slight defocussing of the image permits the different regions to be visualized in the TEM mode (inset in Fig. 2), where the τ phase appears mottled and the $M_{23}C_6$ phase exhibits more uniform contrast. This contrast difference may be a surface-related effect associated with electropolishing.

Composition profiles were measured in the control material at precipitate-free sections of grain boundaries and at intergranular precipitate interfaces. An apparently precipitate-free grain boundary exhibited a narrow (<10 nm) region depleted to $\sim 7\%$ Cr and enriched to $\sim 6\%$ Ti (PEELS - Fig. 3). X-ray microanalysis on a similar boundary indicated similar Cr depletion and enrichment of Al and Nb, as well as Ti. This behavior was attributed to the undetected presence of a thin precipitate enriched in these three elements (i.e., γ' phase). This assumption was confirmed by the presence of other precipitate-free grain boundaries that exhibited Ti levels $<1\%$. The average Cr level for such boundaries was measured as 13.3% (EDS) and 15.8% (PEELS).

Faulted dislocation loops (5-25 nm) and isolated cavities (1.5-8 nm) were present in the irradiated matrix. Depletion of Cr and Fe, Ni enrichment and no Ti enrichment were observed for a precipitate-free grain boundary in the irradiated material (Fig. 4). The average Cr level for such low-Ti grain boundaries was measured as 11.0% (EDS) and 11.5% (PEELS). The change in minimum chromium level at non- $M_{23}C_6$ interfaces for the control and the irradiated materials as measured by PEELS is 4.3 wt%, larger than measured by EDS (2.3 wt%). This difference may in part reflect the better spatial resolution of PEELS because of the reduced influence of beam broadening. Further high spatial resolution AEM will be required to clarify the role of RIS in IASCC of Alloy X-750.⁴

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FIG. 1 Grain boundary in control material exhibiting predominantly $M_{23}C_6$ (C) with some γ' interspersed (arrowed).
 FIG. 2 Grain boundary in control material with mixed particles of $M_{23}C_6$ carbide/ τ ($M_{23}B_6$) boride ($M_{23}C_6$ arrowed).
 FIG. 3 Composition profile (PEELS) at an apparently precipitate-free grain boundary in the irradiated material.
 FIG. 4 Composition profile (PEELS) at a precipitate-free grain boundary in the irradiated material.