

Multi-Detector STEM-EDS Mapping of Ion-Irradiated Nanostructured Ferritic Alloys

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Materials used in advanced fission or fusion reactors must be able to withstand radiation damage doses of several hundred displacements per atom (dpa) under high temperature and elevated creep stresses, in corrosive environments. Because actual reactors can only apply a few dpa/year of neutron irradiation to specimen capsules, heavy ion irradiation at dose rates of hundreds of dpa/day are useful for rapid screening of candidate alloys. Heavy ions produce a damaged region of a few thousand nanometers from the surface of a test specimen and this dose profile results in large damage gradients in the material volume. To characterize the damage regions, an FEI Titan ChemiSTEM scanning transmission electron microscope with an aberration-corrected probe and large-area, four-sensor X-ray detector, was used to map a 14YWT nanostructured ferritic alloy (NFA) implanted at -100 to 750°C with 10 MeV Pt³⁺ ions, with a peak damage depth of ~1000 nm and a damage tail extending to ~1500 nm. By rapidly mapping >500×2000 nm regions, the high brightness probe and high-efficiency detector of the ChemiSTEM permitted the NFA response over the full damage range (i.e., zero to peak to zero dpa) to be recorded.

FIB-prepared surface cross-sections prepared from the as-fabricated and irradiated NFAs were mapped using the following parameters: 6-7 nA, ≈0.5 nm, 200 keV corrected probe and 252×1000 pix, 528×2078 nm spectrum images acquired over 3600 sec. This ≈2 μm deep map captures the entire radiation response of the NFA, extending from the surface to the maximum dpa (at ~1000 nm) and back to zero dpa (at a depth of ~1500 nm) [1]. For the highest fluence specimen (4×10¹⁶ Pt/cm²), the peak dpa is ~320 and the SRIM-estimated peak Pt is ~1 at. %. Maps for the as-fabricated NFA, irradiated 4×10¹⁶ Pt/cm² at -100°C, and 4×10¹⁶ Pt/cm² at 750°C, are shown in Fig. 1. The as-fabricated NFA shows Cr and W segregation to the grain boundaries (GB), Ti-rich ~50 nm precipitates [2] dispersed in the matrix, and Ti-Y-enriched particles at the GBs. In the -100°C specimen, the 1 at. % peak Pt implantation profile is clear, and matches the SRIM prediction. Above ~30 dpa, all GB segregation and Ti-Y GB precipitates are homogenized, and the larger Ti-rich particles are blurred by ballistic mixing into the matrix [1]. The 750°C specimen (close to proposed fusion reactor service conditions) retains Cr and W segregation and indicates growth of the Ti-Y-O particles at the GBs. This behavior points to a self-healing process active at 750°C but not at -100°C, indicating 14YWT is promising for in-reactor service.

Combining a high brightness aberration-corrected probe with high-efficiency X-ray detectors allow the entire response of heavy-ion-irradiation specimens to be studied in a single map, which was a previously impossible experiment.

[1] C. M. Parish et al., *J. Nucl. Mater.*, **V418** (2011) 106.

[2] M. K. Miller et al., *Mater. Sci. Tech.*, **V27** (2011) 729.

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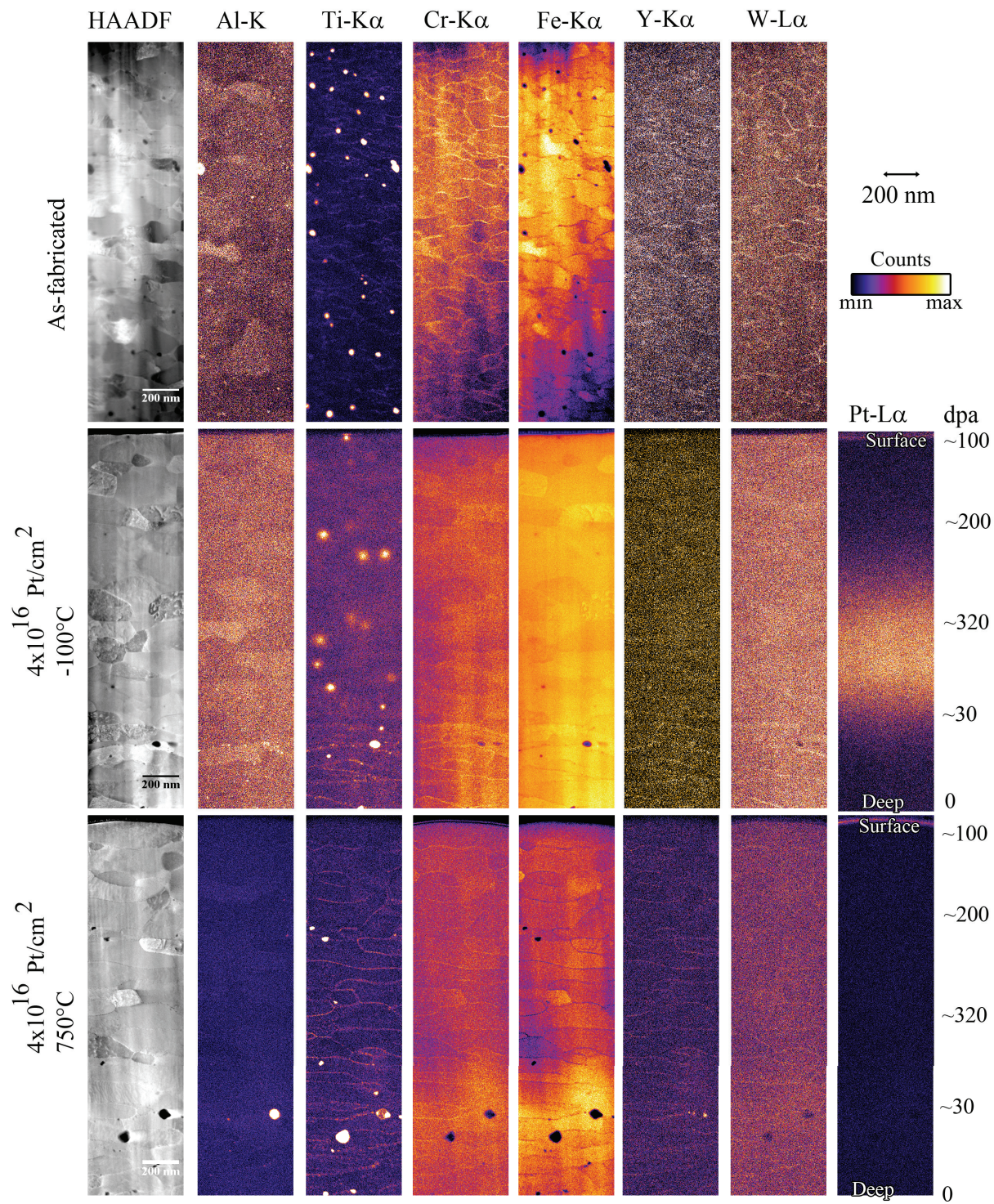


Figure 1. As-fabricated, high-fluence Pt ion irradiated at -100°C and 750°C maps of 14YWT NFA.