Novel STEM-EDX Analysis of Radiation-Induced Precipitates in a Self-Ion Irradiated Cold-Worked 316 Austenitic Stainless Steel Used for PWR Baffle-Bolts

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Abstract. High neutron irradiation doses exceeding 100 dpa are estimated for reactor vessel internal components such as baffle-bolts, when the pressurized water reactor (PWR) life extension beyond 60 years will take a place. At doses above 100 dpa, there are no literature data of the radiation-induced precipitates in austenitic stainless steels irradiated at the PWR in-core conditions and the information about radiation-induced precipitates evolution is still limited even at intermediate doses. Furthermore, the high neutron dose data of the other types of radiation damage (such as Frank loops, swelling, or radiation-induced segregation (RIS)) are very limited. The missing experience about the radiation damage at the high doses and temperatures can be achieved with use of self-ion irradiations. In comparison with neutrons, they have high dose rates on the order of 10^{-3} dpa/s and shorten the irradiation times of 100 dpa from decades to days. Also they are low cost and do not produce activated samples.

To achieve better mechanistic understanding about the radiation-induced precipitates at the intermediate and high doses, 5 MeV Ni^{++} and Fe^{++} ion irradiations were performed on a cold-worked 316 austenitic stainless steel. The irradiation conditions were 23 dpa at 380°C and 130 dpa at 380°C. Complementary techniques, TEM selected electron diffraction (SAED), TEM dark-field imaging, STEM energy dispersive X-ray (EDX) spectroscopy mapping and atom probe tomography (APT) were used for extensive characterization of fine radiation-induced precipitates with size of few nanometers. Firstly, TEM-DF imaging using extra spots of precipitates from SAED patterns with specific two-beam diffraction conditions was performed to detect presence of the precipitates. Secondly, these precipitates were analyzed without any tilt or movement of the sample by STEM-EDX using a powerful SUPER-X EDX detector with a high solid angle.

This approach allowed for the first time to characterize crystallography, morphology and chemical composition of exactly the same fine-scale precipitates only by TEM techniques. It also allowed investigating of substantially larger volume of material in comparison with APT.

The precipitates characterized in the irradiated material were predominantly in form of the Ni-Si rich γ' phase at both irradiation conditions. The EDX analysis further determined Ni-Si-Mo-P rich nano particles indicating possible presence of G-phase precipitates. It was found out a saturation of precipitates size and density before irradiation dose of 23 dpa at 380°C which is an important new insight to evolution process of radiation-induced precipitates in the studied material. Furthermore, it was found out by STEM-EDX and APT, that the original dislocation network introduced by cold-working prior the irradiation was a primary sink where nucleation of radiation-induced precipitates was initiated via intra-granular solute RIS.