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Precipitation of Second-Phase Particles in a Proton-Irradiated Model Alloy

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Precipitation of second-phases is one form of microstructural change observed following irradiation of austenitic alloys in nuclear reactor cores [1]. Precipitation of γ' has been observed in irradiated austenitic steels, including a heat of type 316 steel irradiated as low as 270°C [2], although precipitation is not regularly observed below irradiation temperatures of 400°C. At 400-500°C, γ' precipitation forms abundantly in most SA and CW 316 and Ti-modified steels during fast breeder reactor irradiation [1]. G-phase is another cubic precipitate that has been observed in irradiated austenitic alloys. As noted by Maziasz [1], the dominant cause of radiation-induced precipitation is radiation-induced segregation of solutes to a sink.

Proton irradiation has been used to accurately emulate the radiation-induced segregation, radiation-induced microstructure, hardening and irradiation-assisted stress corrosion cracking found in neutron-irradiated austenitic stainless steels [3]. However, radiation-induced precipitation has not been observed in austenitic alloys following proton irradiation under conditions relevant to light water reactor cores. The objective of this work is to examine precipitation of second phase particles in a model alloy following proton irradiation.

An ultra-high-purity Fe-18Cr-12Ni-1.2Si (wt%) alloy was used in this study and the bulk composition is listed in Table 1. Irradiation to 5.5 dpa was conducted with 3.2 MeV protons (E_d = 40 eV) at 360° ± 10°C, resulting in a nearly uniform damage rate throughout the first 35 μm of the proton range (40 μm). Further details of the sample preparation and irradiation are given elsewhere [3]. Microchemical analysis was performed using a scanning transmission electron microscope with energy-dispersive x-ray analysis (STEM/EDS). Microstructural characterization and STEM/EDS analysis were performed in the Philips CM200/FEG at Oak Ridge National Laboratory.

Following irradiation to 5.5 dpa, precipitation was observed in the high-Si alloy. A dark-field image of the second phase particles is illustrated in Figure 1. X-ray microanalysis indicated that the precipitates were enriched in Ni and Si relative to the matrix. Analysis of selected-area diffraction patterns (shown in the inset of Figure 1) reveals the presence of a second-phase. The second phase has been identified as γ' phase (Ni_3Si), which is consistent with both the SAD pattern and indicated enrichment. The second phase particles range in size from 2-9 nm. The mean size of the precipitates is 2.2 nm and the number density is 1.9 x 10^{23} m^{-3}. This corresponds to a volume fraction of 0.22 %. The volume fraction of precipitates is then used to calculate the weight fraction of precipitates within the alloy. The calculated weight fraction of precipitates is then multiplied by the fraction of Si within the precipitates (13.7 wt%). For Ni_3Si and the measured volume fraction, 0.02 wt% of the total Si content (1.05 wt% Si bulk) is contained within the irradiation-induced γ' particles.

Radiation-induced segregation was observed within ~5nm of grain boundaries. In addition, segregation of Cr, Ni, and Si was also observed away from the grain boundary. Composition profiles were taken in the matrix, both perpendicular and parallel to the grain boundary (a typical profile perpendicular to the grain boundary is given in Figure 2). Significant periodic Ni and Si enrichments are found in the matrix, as high as 20 and 7 wt%, respectively, although, the actual composition are likely higher. The width of the Ni and Si peaks is consistent with the mean size of the particles illustrated in Figure 1. Despite similar levels of segregation at the boundary and in the matrix, no precipitates were observed at the grain boundaries.

References

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TABLE 1. Bulk composition of experimental alloy determined by electron microprobe (in wt%).

Alloy	Cr	Ni	Fe	Mn	Mo	Si	C	N	P	S
HP-304+Si	18.2	12.4	67.3	1.0	0.02	1.05	0.020	<0.005	0.01	0.002

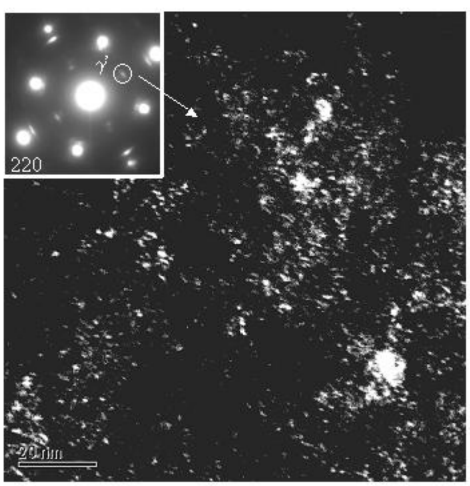

FIG. 1: Darkfield image of γ' particles observed in specimen proton-irradiated to 5.5 dpa at 360°C.

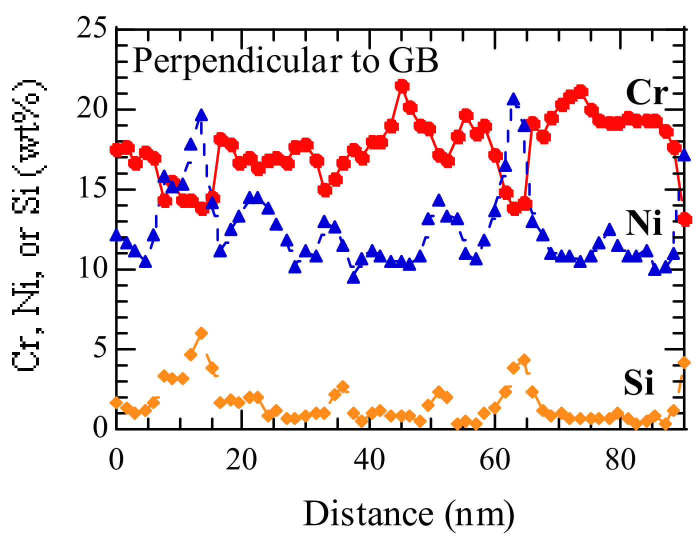

FIG. 2: Segregation of Cr, Ni, and Si in matrix in proton-irradiated to 5.5 dpa at 360°C.

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