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Atom Probe Tomography of Radiation-induced Precipitation in Reactor Cladding and Structural Steels

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300 serials stainless steels are used extensively for core internal components in light water reactors. The formation and evolution of precipitates under irradiation in the stainless steel core components may degrade the mechanical properties to a degree that it may affect the goal of the extension of the current LWR fleet beyond 60 years [1]. Ferritic-martensitic (F-M) steels are candidate cladding and structural materials for advanced fast reactors and fusion reactors. However, possible radiation-induced phases such as a brittle α' phase may severely limit the application of these alloys. A better understanding of radiation-induced precipitation is needed in order to provide guidance for mitigation strategies and alloy modification to survive the extreme environment of advance reactors.

In this study, three stainless steel alloys HP304, HP304+Si, CP304 and three F-M steels T91, HT9, HCM12A were irradiated to 5 dpa at 360ºC and 7 dpa at 400ºC, respectively, using 2 MeV protons in a Tandetron accelerator at the Michigan Ion Beam Laboratory. The penetration depth of 2 MeV protons in both types of steels was calculated to be ~20 μm using SRIM 2006 [2]. Characterization was done at a depth of 5-10 μm from the irradiated surface to avoid the damage peak. After having reached the desired depth of the proton-irradiated region by electropolishing, atom probe tomography (APT) tips were made using the focused ion beam (FIB) lift-out method [3]. The tips were analyzed using the local electrode atom probe (LEAP) system at the Central Analytical Facility at the University of Alabama.

Ni/Si-rich precipitates with a core (~2 nm) composition close to that of the γ' phase were observed in irradiated HP304+Si and CP304. Interestingly, Ni/Si- rich precipitates were also observed in all three F-M steels containing Si (0.2-0.3 wt%) and Ni (0.2-0.5 wt%). The Ni/Si-rich clusters were also enriched in other elements such as Mn and P. Some of the precipitates were found to be associated with dislocation lines and loops. Although the majority of the Ni/Si- rich precipitates do not have the stoichiometric composition of the γ' phase, they are probably precursors. Ni/Si-rich phase was reported in reactor pressure vessel (RPV) steels (referred to "late blooming phase" [4]). Therefore, Ni/Si- rich clusters form readily in steels containing Ni and Si under irradiation regardless of the crystal structure (FCC, BCT or BCC) of the matrix. In addition to Ni/Si-rich clusters, Cu-rich and Cr-rich precipitates were also observed in F-M steels. The type of precipitate is dependent on the bulk composition as shown in FIG. 1. For instance, Cu-rich precipitates were observed in T91 and HCM12A with higher Cu content and Cr-rich precipitates only appeared in HCM12A and HT9 with higher Cr content. Cr-rich precipitates will likely develop into the α' phase which was reported at high dose [5]. Neither Cu-rich nor Cr-rich precipitates are favorable phases in F-M steels as both cause embrittlement of the alloys. However, further investigation of the evolution of these precipitates at high dose and high temperature is still needed. [6]

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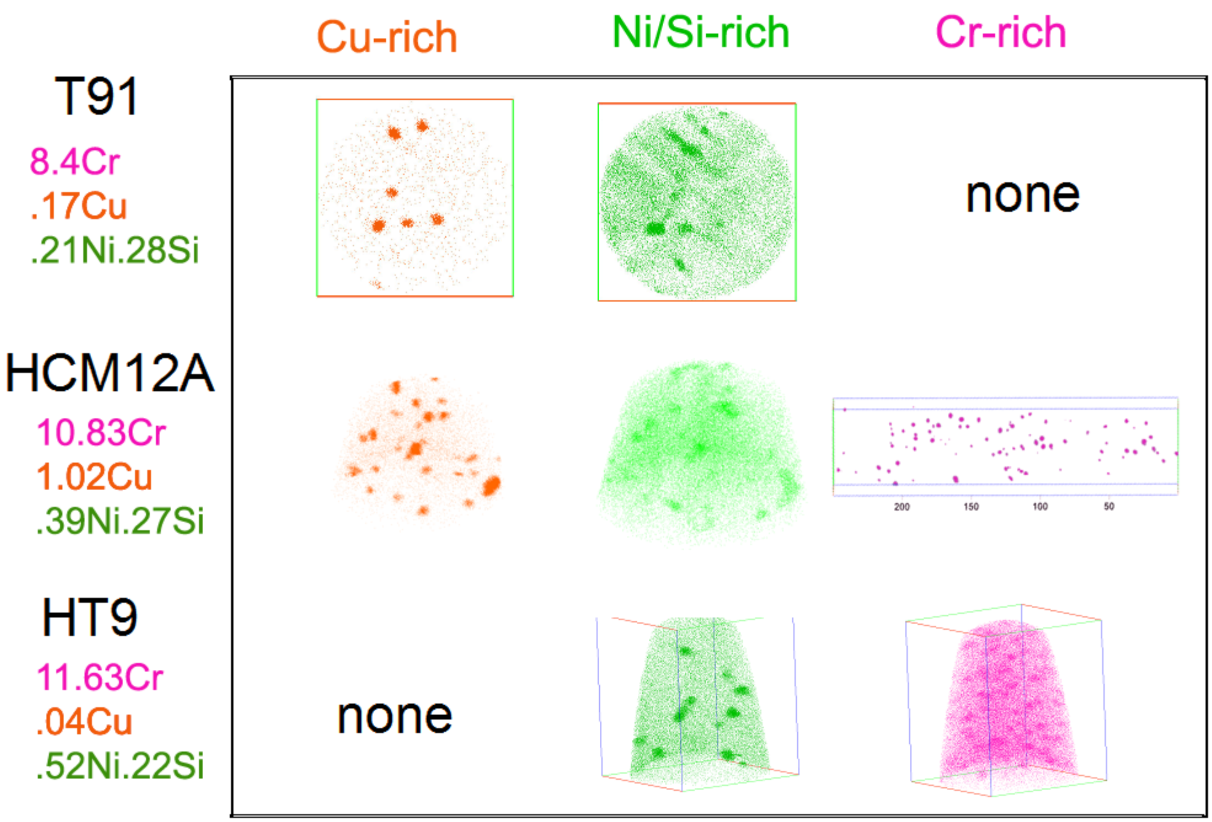

FIG. 1. Three types of radiation-induced precipitates as revealed by APT in F-M steels T91, HCM12A and HT9 following irradiation to 7 dpa at 400ºC. The bulk contents of Cr, Cu, Ni and Si (in wt%) in the three steels are shown on the left.

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