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On the Dose Rate Dependence of Cr Clustering in Ion-Irradiated Fe-18Cr Alloys

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In high Cr ferritic alloys, the α' phase has been observed in a number of model and commercial alloys and under a variety of irradiation conditions, e.g.[1], and has been associated with increased hardening. Recent studies focused on binary Fe-Cr model alloys as surrogates for more complex Cr-containing ferritic steels. Atom probe tomography (APT) studies reported the presence of near-equilibrium nanoscale α' precipitates in alloys containing above 9 % Cr that had been neutron irradiated at ≈ 315 ºC [2] at intermediate dose rates to 1.8 dpa. Post irradiation annealing at of a Fe-18%Cr binary alloy at 500°C partially dissolves and coarsens the precipitates, while annealing at 600°C fully dissolves them. These results are consistent with recent assessments of the Fe-Cr phase diagram. In contrast, well-formed nanoscale α' precipitates have generally not been found under ion irradiation, that produce more diffuse diffuse and dilute Cr clusters instead [3].

To test the hypothesis that α' precipitation is strongly affected by dose rate, and perhaps the irradiating particle, two high energy ion irradiations at different dpa rates, to the same nominal peak dose of 3 dpa, were performed at the Center for Accelerator Mass Spectrometry at the Lawrence Livermore National Laboratory using 70 MeV Fe ions at 290 ±5 °C. The dose rate varied from $6x10^{-6}$ dpa/s to $3x10^{-4}$ dpa/s.

Atom probe tomography (APT) was performed on the ion-irradiated Fe-18%Cr alloys. APT specimens were prepared following standard liftout and milling procedures using a FEI Helios 650 Nanolab scanning electron microscope and focused ion beam instrument. The specimens were extracted from the ≈ 6 µm ion implanted layer with their tips located at different selected distances from the sample surfaces. Approximately the same series depths were examined to assess the effects of the dpa rate at the same dpa. The APT data were collected using a Cameca LEAP 4000XHR instrument operated in voltage mode at 50 K, with a pulse fraction of 20%, a pulse repetition rate of 200 kHz, and a collection rate of 5 ions per 1000 pulses. The CAMECA Integrated Visualization & Analysis Software version 3.6.10 was used for the data reconstruction.

The APT spatial distributions of Cr atoms in the ten ion irradiation conditions is shown in **Figure 1**. The extent of clustering has been quantified and is used to characterize the effects of dpa and dpa rates on the character and evolution of Cr clusters and α' precipitates [5].

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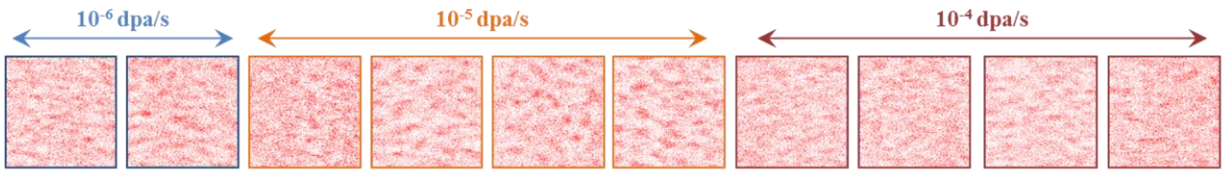

Dose (dpa)	0.2	0.6	0.3	1.1	1.5	2.4	0.6	1.2	1.6	3.1
Dose rate (dpa/s)	2.4×10^{-6}	6.1×10^{-6}	2.3×10^{-5}	1.1×10^{-5}	1.6×10^{-5}	2.5×10^{-5}	5.8×10^{-5}	1.1×10^{-4}	1.5×10^{-4}	2.8×10^{-4}

Figure 1. Cr spatial distributions in the ten ion irradiation conditions ranging from 0.3 to 3.1 dpa and 6×10^{-6} to 3×10^{-4} dpa/s. Atom maps are 5 nm slices of the reconstructed APT volumes. Each map has dimensions of 25 x 25 x 5 nm.

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