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Grain Boundary Chemistry Before and After Ion Implantation in ODS Steels

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Reduced-activation (RA) oxide dispersion strengthened (ODS) ferritic/martensitic steels (FM) are potential candidate materials for structural components in fusion reactors for their improved mechanical properties over the austenitic steels, higher operating temperatures, resistance to swelling and helium embrittlement [**1**]. The complex microstructure of RAFM steels provides a high density of lattice defects allowing for the absorption of point defects created by irradiation and the accumulated damage is less than in conventional steels. However, the detailed behaviour of microstructural features and grain boundaries (GB) in particular in the ODS alloys is still unclear. During irradiation, a non-equilibrium Cr segregation profile can develop as a result of enhanced diffusion in austenitic steels, either by a vacancy or interstitial mechanism [**2**]. In more complex steels such as ODS steels, RIS and/or RID are expected to take place as well, but the synergies resulting from complex microstructures (nano-oxide particles, carbide phases, small grain size, alloying elements) which possibly lead to more complex behaviours under irradiation need to be clarified.

The limited knowledge of Cr depletion at grain boundary arises from the experimental difficulties in both controlling implantation conditions and in analyzing grain boundaries as three-dimensional structures. The combination of atom probe tomography analyses and analytical electron microscopy provide unprecedented information of the 3D character of grain boundary chemistry.

A model ODS Fe-12 wt.% Cr steel containing 0.4 wt.% Y_2O_3 was processed by mechanical alloying followed by hot isostatic pressing (HIP) as described in [**5**]**.** The alloy was thermally annealed at 750^{o}C for 4 hours. Ion implantation at temperatures between 300 and 500^{o}C was performed at the EPSRC Ion Beam Center of Surrey University. Fe ions of 2MeV and 0.5MeV energy respectively were implanted with doses of 1.10^{15} /cm^2 and 5.10^{14} /cm^2 respectively to create a flat damage profile over ~750nm. Atom-probe tomography samples were prepared by standard electropolishing methods. Atom-probe analyses at 30K were performed using a LEAP-3000X and 3000HR using laser pulses with a spot size of ~10 µm and energy of 0.4nJ at 200 kHz repetition rate. TEM samples from flat implanted surfaces were prepared using a Focused Ion Beam lift-out technique. TEM characterization and electron energy loss spectroscopy (EELS) analyses were carried out in a JEOL 3000F scanning-transmission electron microscope operated at 300kV and equipped with a high angle annular dark field detector (HAADF) and a Gatan image filter.

Segregation of Cr to grain boundaries is observed in the as-HIPed state and after annealing at 750^{o}C (Fig. 1). Carbon is also found at some grain boundaries. Both Cr segregation and complete depletion were observed after heavy-ion implantation (Fig. 2). Depletion occurs over 3-10nm at the grain boundaries. The effect of temperature and dose will be discussed.

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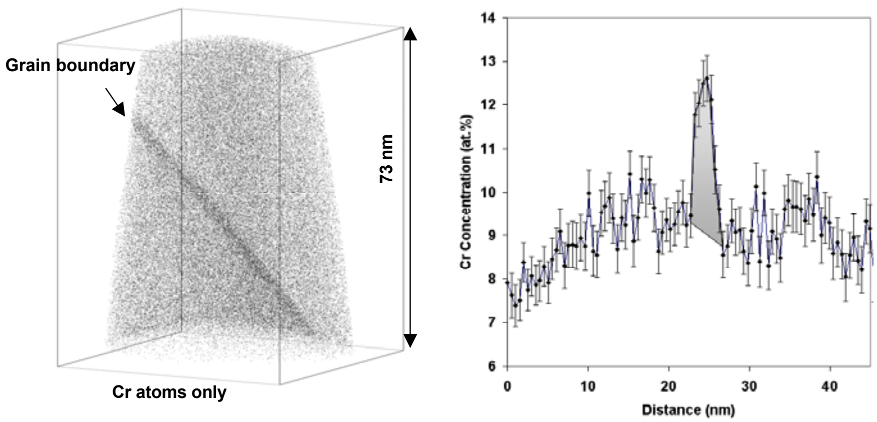

FIG 1: (a) 3D atom probe reconstruction (b) Concentration profiles across grain boundaries showing Cr segregation before irradiation.

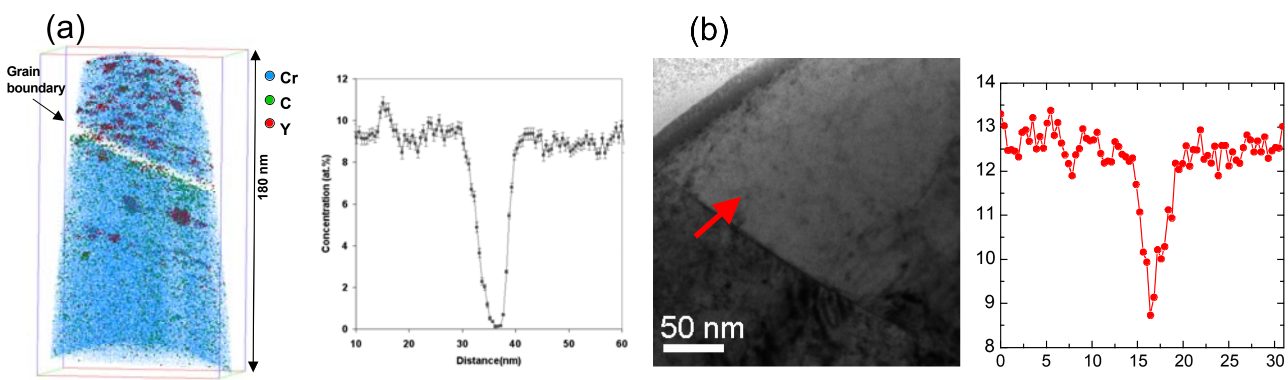

FIG 2: 3D atom probe reconstruction and concentration profile across grain boundaries showing Cr depletion after irradiation (b) BF-TEM image of a grain boundary located ~ 60 nm from the irradiated surface, with EELS Cr $L_{2,3}$ relative concentration showing Cr depletion across the grain boundary.

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