Characterization of grain boundary segregation in ferritic/martensitic steel after irradiation in SINQ Target 4

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Ferritic/martensitic (FM) steels are major structural materials irradiated in the targets of Swiss Spallation Neutron Source (SINQ). The post-irradiation examination (PIE) of irradiated FM specimens show strong degradation of mechanical properties after irradiation, particularly at dose \textgreater 10 dpa. As it is known radiation-induced segregation (RIS) could greatly affect the fracture properties of irradiated materials, RIS at grain boundary has been studied with atom probe tomography (APT) in this work. Some interesting results have been obtained from 3 FM steels (F82H, Eurofer 97, and ODS Eurofer) irradiated to 20 dpa.

\textbf{KEYWORDS:} Radiation-induced segregation (RIS), Grain boundary, Atom probe, SINQ target, PIE (post-irradiation examinations)

1. Introduction

FM steels are promising structural materials for spallation targets and the key materials studied in the fusion and Generation-IV reactor materials programs. RIS may have a strong impact on the ductility of materials. Historically the evolution of elements segregation at interfaces (GBs \& phase boundaries) was studied with analytical electron microscopy and Auger-spectroscopy. However, it should be noted that these techniques usually are not sufficiently accurate for the investigation of elements with low concentrations. With the improvement of the APT technique in the last decades, nowadays the RIS behavior can be investigated with higher accuracy.

RIS can be influenced by irradiation temperature, dose and the mis-orientation angle of grain boundaries [1-2]. In this work, we have studied RIS behavior of solute elements and spallation transmutants in 3 FM steels in different irradiation conditions.

2. Experimental results

2.1 Sample preparation
Firstly, a pillar sample including grain boundaries is extracted from bulk material with focused ion beam (FIB) milling. Then, electron backscatter diffraction (EBSD) is carried out after polishing the top surface of the pillar sample by FIB. From the EBSD map (Fig. 1) one can roughly determine the angle of a grain boundary. Finally, the pillar is sharpened to a needle-like sample containing the selected grain boundary by annular milling. Additionally resharpening progress would be necessary when the grain boundary is too far away from the apex.

![Fig. 1. Left: the polished top surface of a pillar. Right: EBSD map of the top surface.](image)

### 2.2 Grain boundary mis-orientation characterization

During annular fibbing only long and straight grain boundary could be tracked and kept in the center of the apex. For some samples as shown in Fig. 1, the grain structure is complex and the grain boundaries are tortuous. A more accurate characterization method, the precession electron diffraction (PED) equipped in a transmission electron microscope (TEM), needs to be used before APT experiments.

In this work, the PED measurements were performed in JEOL Grand ARM operated at 300 keV. In this high voltage TEM, electrons can traverse very thick (~500 nm) samples. This helps to find the grain boundary position and guide the resharpening to make the grain boundary in the apex, as shown in Fig. 2.

![Fig. 2. PED measurement of an APT ample. The grain boundary has a large mis-orientation angle with 54 degree.](image)

### 2.3 Grain boundary segregation

APT experiments were conducted using a Cameca Instruments LEAP 4000X HR of ScopeM at ETHZ. During the APT experiment, a sample expose in an extremely high
electric field ($10^{10}$ V/m) at a temperature of 50 K, which induces a stress on the sample and may cause it fractured. As an example, Figure 3 shows a result obtained from F82H irradiated to 20 dpa in SINQ. The APT sample contains a grain boundary of about 60 degrees. The segregation behavior at the grain boundary for two selected elements V and Si is demonstrated. The atomic concentration across the grain boundary was also measured, as shown in Fig. 4. It can be seen that Si is enriched, while V is depleted at the grain boundary. It should be mentioned that no V depletion was observed in unirradiated F82H steel.

3. Discussion and summary

The observed Si and V segregation behavior is in agreement with solute atoms transporting described by the coupled point-defects fluxes theory [3-5]. Si atoms are smaller and V atoms are bigger than that of the solvent element (Fe). In general, the under-size atoms bind strongly to interstitials and segregate to sinks, while over-size atoms have weaker binding with vacancies and move away from sinks.

In this work, 3 FM steels in different irradiation conditions have been analyzed. The RIS behavior of all solute elements and spallation transmutants Ti, Ca, Sc, K have been studied. The results are of significance to the understanding of radiation damage induced by neutrons and high energy protons.

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