

## Atom Probe Tomography of Oxidised Grain Boundaries in Highly Irradiated SS316

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Structural materials in a nuclear reactor are exposed to a demanding environment, posing requirements on the materials in order to maintain the structural integrity for safe operation. The austenitic components inside of the reactor pressure vessel are exposed to high neutron fluences during the lifetime of the reactor, resulting in radiation induced segregation (RIS) at grain boundaries, and hardening of the matrix by formation of loops and precipitates. At the same time the environment is corrosive. Irradiation assisted stress corrosion cracking (IASCC) appears as intergranular cracks, and it is a phenomenon that these austenitic materials might experience when subjected to a significant fast neutron fluence [1, 2]. Thus, it is important to understand the processes that affect the grain boundaries, especially at the surface in contact with reactor water where cracks initiate.

In this study, Type 316 steel from thimble tubes of the pressurized water reactor (PWR) Ringhals 2 has been characterized. The thimble tubes, used as guide tubes for neutron flux measurements, were in service from the commissioning of the reactor in 1975 until 2009 (205,891 effective full power hours) at a temperature of around 315°C. The accumulated dose during this time is as high as 100 dpa. The irradiated material is compared to reference material that was exposed at a similar temperature for the same time, but without neutron irradiation.

Atom probe tomography (APT) was used to characterize the material, as it gives high resolution (nanometre) three-dimensional chemical information. For sample preparation, a focused ion beam/scanning electron microscope (FIB/SEM) located in a hot lab was used in order to do site specific analysis of grain boundaries close to the surface oxide.

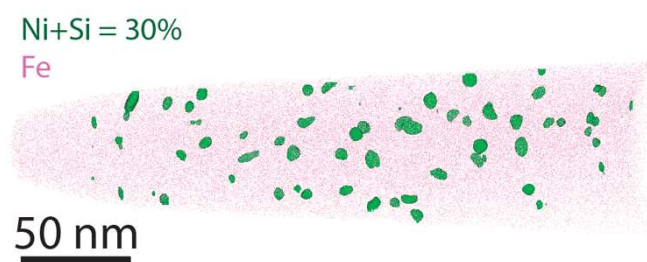
After 100 dpa of irradiation, the metal matrix was full of precipitates of Ni and Si that are a few nanometres in diameter, see Figure 1. These precipitates are assumed to be  $\gamma'$  ( $\text{Ni}_3\text{Si}$ ) or its precursors, as the Ni/Si ratio was less than 3 in most cases ( $2.4 \pm 0.2$ , except one analysis where the ratio was found to be 3.1). The number density of precipitates was  $0.9 \pm 0.3 \times 10^{23}/\text{m}^3$ . These precipitates are known to increase the hardness of the material [3] and appear as a result of the irradiation, as no precipitates were observed in the reference material.

An overview (from the FIB/SEM during the sample preparation) of an analysed grain boundary can be seen in Figure 2. The oxide is growing into the boundary. A few micrometres from the outer oxide, the APT needle was prepared (arrow in the SEM micrograph). The reconstruction of the APT analysis can be seen in the same figure. The grain boundary is enriched in Ni and Si, and depleted in Cr and Mn. There is no O detected at the boundary. There are a number of Ni- and Si enriched particles on the boundary. These

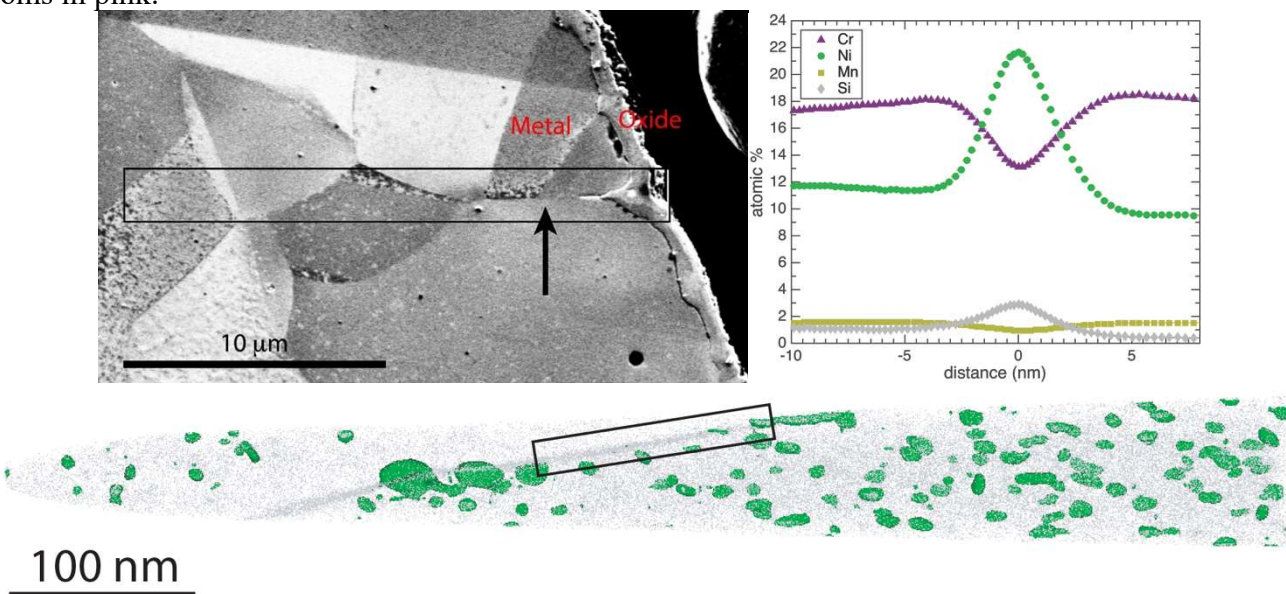
are larger than the ones detected in the matrix of the grains, but the composition is similar (Ni/Si ratio of around 2.4). The boundary composition a few micrometres from the oxide was in essence similar to other grain boundaries from the same material, further away from the oxide [4].

#### References:

- [1] GS Was and JT Busby, *Philosophical Magazine* **85** (2006), p. 443.  
 [2] EA Kenik and JT Busby, *Materials Science and Engineering: R: Reports* **73** (2012), p. 67.  
 [3] T Toyama et al., *Journal of Nuclear Materials* **418** (2011), p. 62.  
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**Figure 1.** Atom probe reconstruction of the material irradiated to 100 dpa. Green isoconcentration surfaces represent Ni-Si rich precipitates. The extent of the analysis is shown by a small number of the Fe atoms in pink.



**Figure 2.** SEM micrograph of the material irradiated to 100 dpa. The box marks the volume that was initially lifted out and made into a number of APT needles. The arrow marks the approximate position of the analysis of the grain boundary. Furthermore, the figure also shows the APT reconstruction of the grain boundary, with Si atoms in grey and Ni+Si= 30% as green isoconcentration surfaces. The profile through the grain boundary (marked with a box) is shown.