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Preliminary Atom Probe Tomography Evidence for Hydrogen Trapping at a β-Nb Second Phase Particle in a Neutron-irradiated Zirconium Alloy

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Understanding how hydrogen behaves in different materials during service is a key challenge for materials scientists. However, experimentally imaging low levels of hydrogen has proven to be very difficult and this has limited our ability to create or validate models that predict in-service hydrogen behaviour and, therefore, material property changes.

Zr-based alloys used for fuel cladding in nuclear reactors are susceptible to hydride embrittlement during operation. However, we currently lack a mechanistic understanding of how hydrogen behaves in these alloys [1]. Atom probe tomography (APT), with its unique combination of high spatial and chemical resolution, offers the opportunity to image hydrogen in zirconium and characterise its evolution in the metal after exposure to conditions analogous to those experienced in a reactor.

APT has been applied to study a neutron-irradiated zirconium-based alloy that, during analysis, was found to contain a β-Nb second phase particle (SPP) in one specimen. Within the SPP, an increase in hydrogen levels compared to the surrounding matrix was observed (Figure 1). This is consistent with modelling work, which indicates that Nb-rich SPPs should act as weak hydrogen sinks in zirconium [4], [5]. However, uncertainties remain in determining whether this observation is truly representative of increased hydrogen content within the SPP after service, or if the apparent increase is due to artefacts that are inherent to APT and affect the characterisation of hydrogen [2, 3].

A common approach in APT studies on hydrogen involves isotopic labelling, where samples are prepared with deuterium instead of naturally abundant hydrogen in order to facilitate differentiation between sample and contaminant hydrogen [6-8]. This was not feasible in our study, which aimed to replicate real reactor conditions and involved no "artificial" hydrogen/deuterium charging step. Therefore, it is necessary to understand potential artefacts that may influence the data and to very carefully interpret results [9]. Whilst accurate quantitative conclusions of the hydrogen's behaviour is not possible from this data, qualitative results may still provide interesting information regarding the evolution of hydrogen. This presentation will focus on providing a thorough analysis of the integrity of the apparent increase in hydrogen levels, and the implications of our results will be discussed [10].

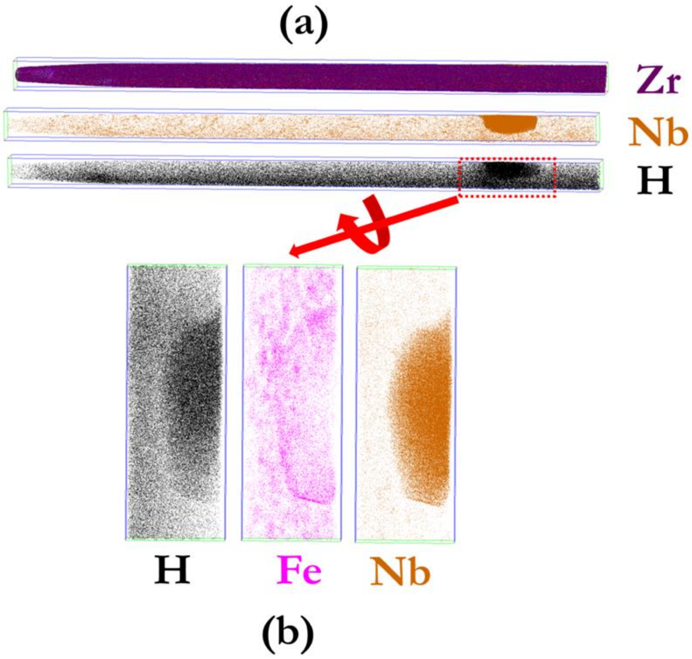

Figure 1. (a) Atom maps of Zr, Nb, and H in low Sn ZIRLO sample irradiated to 3 dpa (bounding box dimensions 70 nm x 72 nm x 1460 nm). (b) Atom maps of H, Fe, and Nb from region of interest highlighted by red box in (a) (bounding box dimensions 70 nm x 72 nm x 300 nm).

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