Atom Probe characterization of neutron irradiated commercial ZIRLO® and AXIOM X2® alloys (#2986)

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Advanced commercial Zirconium-Niobium (Zr-Nb) alloys, such as ZIRLO® and AXIOM®, have been developed by Westinghouse to enhance the corrosion resistance of fuel cladding material for longer service time. As part of the Mechanistic Understanding of ZIrconium Corrosion (MUZIC) program, EPRI and Westinghouse sponsored the shipment sets of neutron-irradiated samples of ZIRLO® and of X2® from Studsvik (Sweden) to Idaho National Laboratory (INL). This study investigated the neutron irradiation induced Nb redistribution in AXIOM® X2® to understand irradiation induced microchemistry changes and irradiation enhanced corrosion resistance. Detailed sample information is shown in Table 1. Sample preparation was performed using the shielded focused ion beam (FIB) at the Irradiated Materials Characterization Laboratory (IMCL) and was followed by APT analysis at CAES facility to elucidate microstructural and chemistry changes.

Table 1: Summary of accomplishment for this proposal 2986.

Materials	Sample ID	Irradiation cycles	FIB for APT sample preparation	Acquisition of APT data
SRA ZIRLO	XL42	1	√	√
SRA ZIRLO	X4	3	√	√
SRA ZIRLO	X33	2	√	√
SRA ZIRLO	XL47	4	√	√
RXA X2	N42	4	√	√
RXA X2	N43	1	√	√

APT data was collected to study effect of neutron irradiation dose on the distribution of Nb and other solute elements like Fe and Cr. Selected datasets (from X2, 4 cycles) are shown in Figure 1 to highlight the type of information obtained and the data analysis carried out to elevate understanding of microstructural changes. The size, shape, and number density of Fe and Nb enriched cluster are crucial to understanding the effects of irradiation and the corrosion kinetics of advanced Zr alloys.

This RTE study was designed to test the hypothesis that the reduced in-reactor corrosion kinetics of Zr-Nb alloys is due to the reduction of Nb content in the Zr solid solution by the precipitation of Nb-rich IIPs/nanoclusters. The APT data suggest that the Nb concentration in the metal solid solution after 4 irradiation cycles has been reduced to about 0.15 at.%. The Nb content in the oxide is even lower than in the metal matrix.

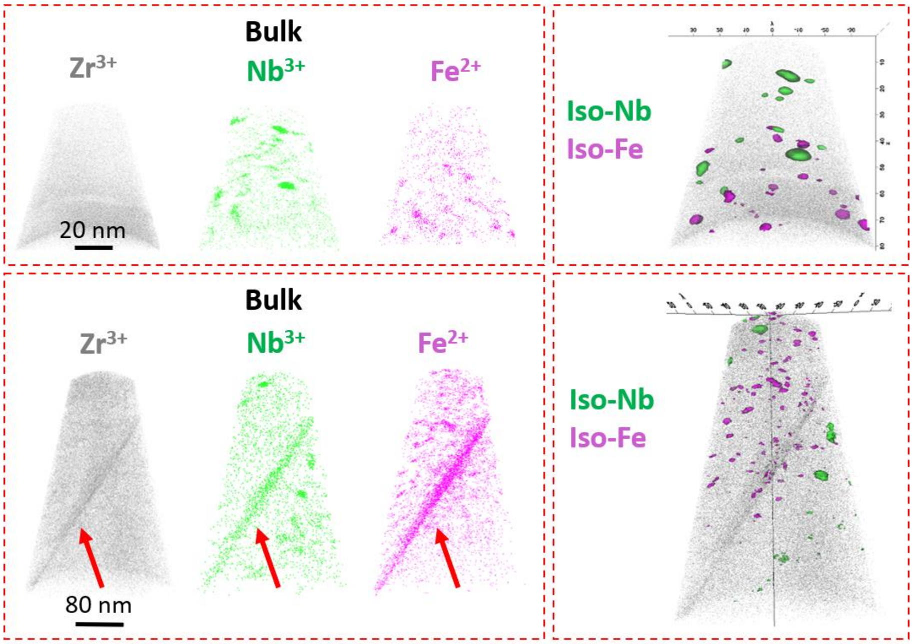

Figure 1. 3D reconstruction of two APT tips in the metal matrix of 4 cycles X2®. Red arrows point to the grain boundary that Nb and Fe have segregated to. The two APT tips locate at about 1-1.5 µm below the O/M interface.

Program relevance:

The research conducted in this project is a critical contribution by INL researchers to the internationally important, highly visible MUZIC program. It is critical for the nuclear industry to understand the evolution of the properties of zirconium alloys during neutron irradiation. One of the primary factors limiting the utilization of fuel rods is hydrogen pickup by the zirconium clad during oxidation. As a result of oxidation and hydrogen pickup, hydrides form and accumulate in the cladding, causing embrittlement and limiting fuel lifetime. Proposed Nuclear Regulatory Commission loss-of-coolant-accident (LOCA) criteria are likely to be hydrogen-dependent, allowing less oxidation of the cladding during a LOCA as hydrogen content increases. The Electric Power Research Institute estimates that transitioning to the new regulatory requirements will cost the U.S. nuclear industry of the order $100M. Other issues related to oxidation and hydrogen pickup in Zircaloy include: (i) channel distortion in boiling water reactors that can lead to control blade binding, and (ii) BWR fuel-assembly structural failures.