Atom Probe Characterization of Neutron Irradiated Commercial ZIRLO® and AXIOM X2® Alloys

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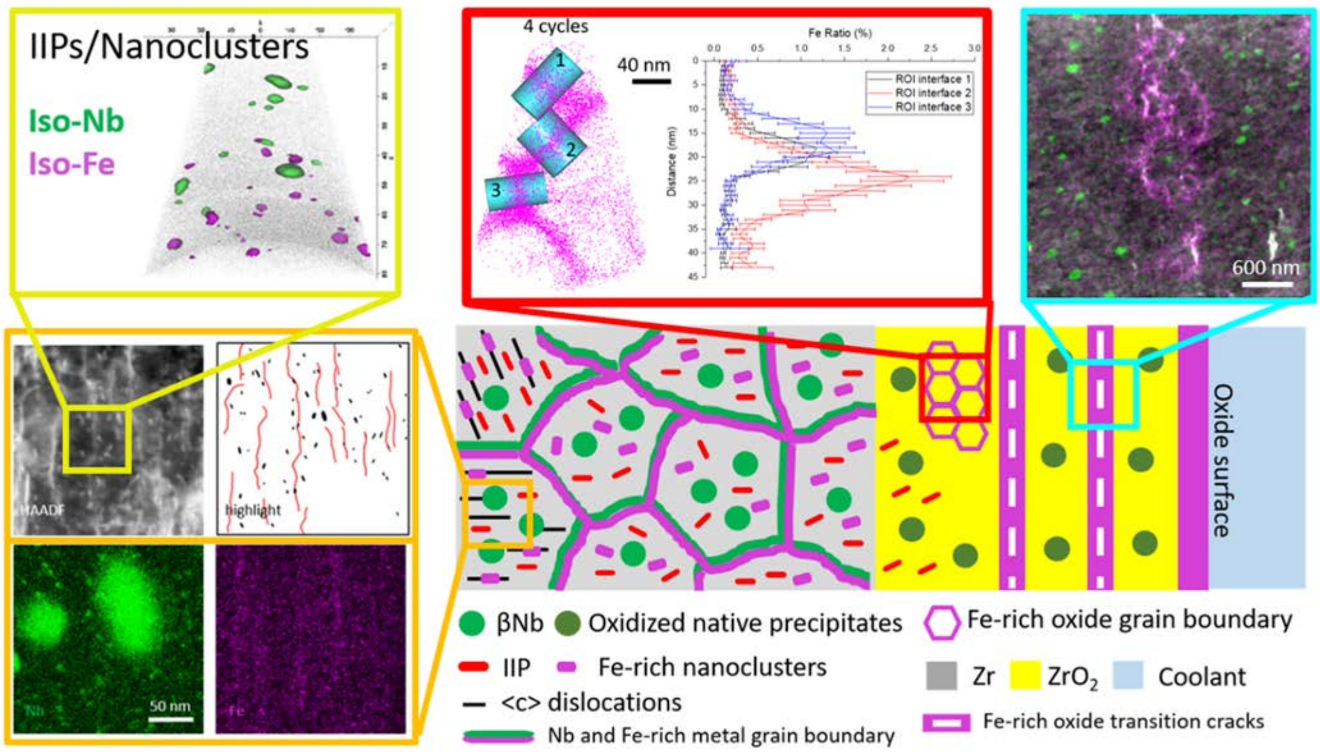
The hypothesis validated in this study is then reduced in reactor corrosion kinetics of Zr-Nb alloys, compared to other Zr-based fuel clad alloys is due to the irradiation induced reduction of Nb content in the Zr solid solution by the precipitation of Nb rich irradiation induced platelets (IIPs)/nanoclusters. The measurement of Nb concentration in the metal solid and oxide layer is key in understanding precipitation behavior and its role on corrosion kinetics. Although the instantaneous oxide/metal velocity is relatively fast, the diffusion of Nb atoms is likely faster for Nb to reach an equilibrium state in the oxide by rejecting Nb atoms back into the metal matrix.

Experimental or Technical Approach

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. This study specifically aims at investigating the neutron irradiation induced Nb redistribution in AXIOM® X2® to understand the effect of neutron irradiation induced microchemistry changes and the irradiation-enhanced corrosion resistance observed in Zr-Nb alloys. The uniqueness of these sets of neutron irradiated alloy samples is that they are at the same two extremes of the fuel cycles, allowing the microchemistry evolution to be studied as a function of irradiation doses, exposure time, and Sn content. To study the neutron IIP/nanoclusters and Nb concentration in the solid solution, atom probe tomography (APT) is the primary tool to obtain reliable chemical information. Sample preparation for the APT study was performed using the shielded focused ion beam at the Irradiated Materials



Characterization Laboratory (IMCL) at Idaho National Laboratory. This was followed by APT analysis in the MaCS at the Center for Advanced Energy Studies (CAES) facility to elucidate microstructural and chemistry changes in all samples.

Results

1. The evolution of precipitates in the metal matrix of in pile X2® do not show much irradiated difference compared to other neutron irradiated recrystallized Zr-Nb alloys, such as M5®. Both Nb rich native precipitates and IIPs

Figure 1. Graphical abstract on microstructural and microchemistry changes in neutron irradiated X2 alloy using Atom Probe Tomography and Transmission Electron Microscopy along with schematic of overall microstructure in the oxide and matrix region.

were found in the metal matrix. Their composition tends to reach an equilibrium state of about 40 at.% Nb. The Nb rich IIPs/nanoclusters density and sizes in X2® follow the same trend reported in literature. There is no evidence showing Nb segregation to <c> dislocation loops, whereas Fe rich nanoclusters may decorate the <c> dislocation loops.

2. In the oxide, the Nb rich nanoclusters are likely to only exist at regions close to an oxide/metal (O/M) interface as observed by APT analysis. However, their oxidation state is still uncertain. At further distance from an O/M interface, the Nb rich nanoclusters may have fully dissolved. While Fe rich nanoclusters were not found in the oxide, Fe rich transition cracks and Fe rich oxide grain boundaries were observed. Those Fe rich features may be due to (1) trapping of coolant water with dissolved Fe or (2) segregation of Fe from supersaturated oxide solid solution. The second mechanism is supported by the nonequilibrium capture of Fe in the suboxide.

Discussion/Conclusion

The major hypothesis validated in this study is 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 Nb content in the oxide is even lower than in the metal matrix. The lower Nb content in the oxide solid solution led to a higher oxide space charge density, which induces a higher electric field across the oxide and lowers the corrosion rate.

References

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Publications

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Distributed Partnership at a Glance

NSUF Institution	Facilities and Capabilities
Center for Advanced Energy Studies	Microscopy and Characterization Suite
Idaho National Laboratory	The Intermediate Voltage Electron Microscopy – Tandem Facility
Collaborators	
University of Wisconsin	Zefeng Yu (collaborator), Adrien Couet (collaborator)