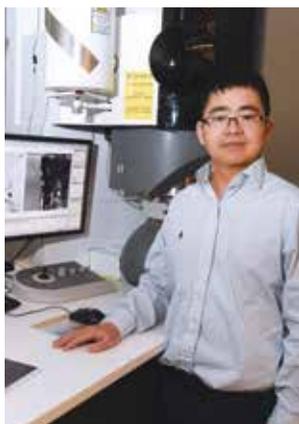


## Short Communications

### Characterization of Grain Boundaries of Alloy X-750 and SS 304 Irradiated in Experimental Breeder Reactor-II

Lingfeng He – Idaho National Laboratory – [Lingfeng.He@inl.gov](mailto:Lingfeng.He@inl.gov)



This project investigates the dose rate effect on the microstructural evolution of stainless steels, which is currently not well understood. Moreover, this project also tries to characterize the elemental segregation behavior at different types of grain boundaries and establish the possible relationship between elemental segregation and grain boundary strength as part of an Idaho National Laboratory (INL) Laboratory-Directed Research and Development project. The grain boundary strength will be measured using similar setup as previous studies [1,2]. The specimens were chosen from the NSUF library [3]. For both unirradiated and irradiated specimens, focused ion beam (FIB) was used to prepare site-specific lamellae for characterization by transmission electron microscopy (TEM). The Titan Scanning TEM (STEM) at the Irradiated Materials Characterization Laboratory (IMCL), equipped with ChemiSTEM technology was used to quantify radiation-induced defects (i.e., dislocation loops, voids, and precipitates) and the segregation at defect sinks (dislocations and grain boundaries). Electron backscatter diffraction (EBSD) was carried out to distinguish different types of grain boundaries. Atom probe tomography (APT) measurement of the segregation

at the selected grain boundary was conducted on a LEAP 4000X HR at the Center for Advanced Energy Studies (CAES) Microscopy and Characterization Suite (MaCS). STEM, TEM, and APT data were then combined to achieve better understanding of the irradiated microstructure and microchemistry.

#### Results

STEM data on the irradiated microstructure, including the size and number density of voids, Frank loops, and gamma-prime precipitates were gathered as well as APT data. APT data on microchemistry at different types of grain boundaries. The irradiated microstructures of stainless steel (SS) 304 and nickel-base alloy X-750 were compared to existing data in literature of similar alloys irradiated at different dose rates.

Our results show [4] that dose rate has a significant effect on the loop size and density in SS 304 i.e., lower dose rate leads to larger loop size but lower loop density. Besides dislocation loops, voids were also found in SS 304 [4]. Ni and Si segregation was identified at the void-matrix interface, and (Ni,Si)-rich clusters were also identified. The HyperSpy non-negative matrix factorization (NMF) multivariate statistical analysis (MVSA) was used to better quantify the Ni and Si content in the (Ni,Si)-rich clusters.

**Conclusion**

The results of this project provide new insights and advanced microstructure data on neutron-irradiated SS 304 and nickel-base alloy X-750. It also provides specific segregation behavior of different types of grain boundaries, both before and after irradiation. These data are being incorporated into molecular dynamics (MD) and density functional theory (DFT) simulations of corresponding alloys.

**References**

[1.] K. Fujii and K. Fukuya: Development of micro tensile testing method in an FIB system for evaluating grain boundary strength, Mater. Trans., 52 (2011) 20-24.  
 [2.] T. Miura, K. Fujii, and K. Fukuya: Micro-mechanical investigation for

effects of helium on grain boundary fracture of austenitic stainless steel, J. Nucl. Mater. 457 (2015) 279-290.

[3.] J.I. Cole: NSUF Fuels and Materials Library, INL/EXT-15-36345, 2015  
 [4.] L. He, X.M. Bai, J. Pakarinen, B. Jaques, J. Gan, A.T. Nelson, A. El-Azab, and T.R. Allen: Bubble Evolution in Kr-irradiated UO<sub>2</sub> during Annealing, J. Nucl. Mater., 496 (2017) 242-250.

**Publications**

[1.] X. Liu, L. He, H. Yan, M. Bachhav, J.F. Stubbins: A transmission electron microscopy study of EBR-II neutron-irradiated austenitic stainless steel 304 and nickel-base alloy X-750, J. Nucl. Mater., 528 (2020) 151851.

Distributed Partnership at a Glance	
NSUF and Partners	Facilities and Capabilities
Idaho National Laboratory	Electron Microscopy Laboratory, Irradiated Materials Characterization Laboratory
Collaborators	
Idaho National Laboratory	Lingfeng He (principal investigator), Mukesh Bachhav (co-principal investigator), Sebastien Teyseyre (co-principal investigator), Xiang Liu (collaborator)