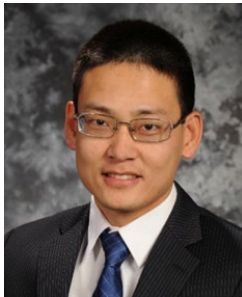


## Understanding Swelling Related Embrittlement of AISI 316 Stainless Steel Irradiated in EBR-II

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Neutron irradiation causes degradation of the mechanical properties of materials due to the formation of irradiation defects that can change the deformation mechanisms. Many years of experimental and modeling efforts on irradiation damage and effects have generated a substantial knowledge database on the evolution of microstructure, microchemistry, and mechanical properties of materials under low-dose neutron irradiation. However, due to the limited database of high-dose neutron-irradiation studies, a complete understanding remains unclear concerning microstructural evolution and corresponding mechanical property changes under high-dose neutron irradiation. This project investigated swelling-induced embrittlement of American Iron and Steel Institute (AISI) 316 SS irradiated in Experimental Breeder Reactor II, a sodium-cooled fast reactor. The research team examined the microstructure, microchemistry, and mechanical properties of high-dose, neutron-irradiated AISI

316 SS. They observed a strong correlation between elongation after yielding and void swelling. The role of void swelling on the deformation mechanisms was made clear. Understanding the mechanical behaviors of high-dose, neutron-irradiated core internals is vital to developing advanced materials for the next generation of nuclear reactors.

### Introduction

Austenitic AISI 316 SS has been widely used for the structural components in LWRs and proposed as critical structural materials for the next generation of nuclear reactors due to their excellent mechanical properties. However, irradiation-induced defects and precipitation could lead to hardening and embrittlement. At low irradiation temperatures ( $<360^{\circ}\text{C}$ ) and low irradiation doses ( $<10$  dpa), neutron irradiation typically creates black dots and dislocation loops in 316 SS. The interaction between mobile dislocations and dislocation loops causes dislocation channeling, a

localized deformation mode. At higher irradiation temperatures and higher irradiation doses, irradiation-induced voids could cause severe embrittlement. Some studies have shown that AISI 316 SS could lose ductility when swelling reaches 10 percent. Such severe embrittlement arises from the intense deformation localization and void-to-void cracking. Despite the efforts, a direct correlation between microstructure, chemical redistribution, and mechanical properties is still missing, and the effect of void swelling on deformation mechanisms remains unclear.

### Project Hypothesis

Neutron irradiation of austenitic 316 is known to cause embrittlement. However, the deformation mechanism of 316 SS after high-dose neutron irradiation remains unclear. In this project, the research team studied the microstructure and microchemistry of 316 SS irradiated in a fast reactor to high neutron fluences and the influence of void swelling on the deformation mechanisms.

*Understanding the deformation mechanism of high dose, fast neutron irradiated materials is essential for the development of advanced structural materials for advanced reactors.*

### Experimental or Technical Approach

Researchers used transmission electron microscopy (TEM), atom-probe tomography, and ring tensile testing.

### Results

The research team performed TEM characterizations of the irradiated 316 SS specimens. A high density of voids and precipitates formed at each irradiation condition. The team measured void size and void density to calculate volumetric swelling. They performed chemical analysis of irradiated specimens using energy-dispersive spectroscopy (EDS) equipped on Titan Themis. Researchers observed Ni-Si precipitates with an average diameter of ~10 nm, which had formed in the matrix close to the voids, and enrichment of Ni, Si, and P near the voids. The chemical segregation near the voids leads to the changes in sink strength to irradiation defects and furthers the embrittlement of the specimens. Fe,

Cr, Mn, and Mo are depleted along grain boundaries, while Ni and Si are enriched along the grain boundary. The dark-field scanning transmission electron microscope (STEM) image also reveals a void-denuded zone near the grain boundary. The formation of voids and chemical redistribution (e.g., precipitation, chemical segregation on voids, and grain boundaries) cause severe changes in the mechanical properties of the irradiated specimens.

The research team characterized the fractography of deformed specimens using a scanning electron microscope (SEM). The fracture surface of the reference sample mainly has dimple-like morphologies, suggesting a ductile failure of materials. With increasing swelling from 1.8–13.33%, the dimple-like failure gradually becomes cleavage failure, which is depicted by a river pattern in SEM images. Cleavage fracture results in embrittlement. Fractography

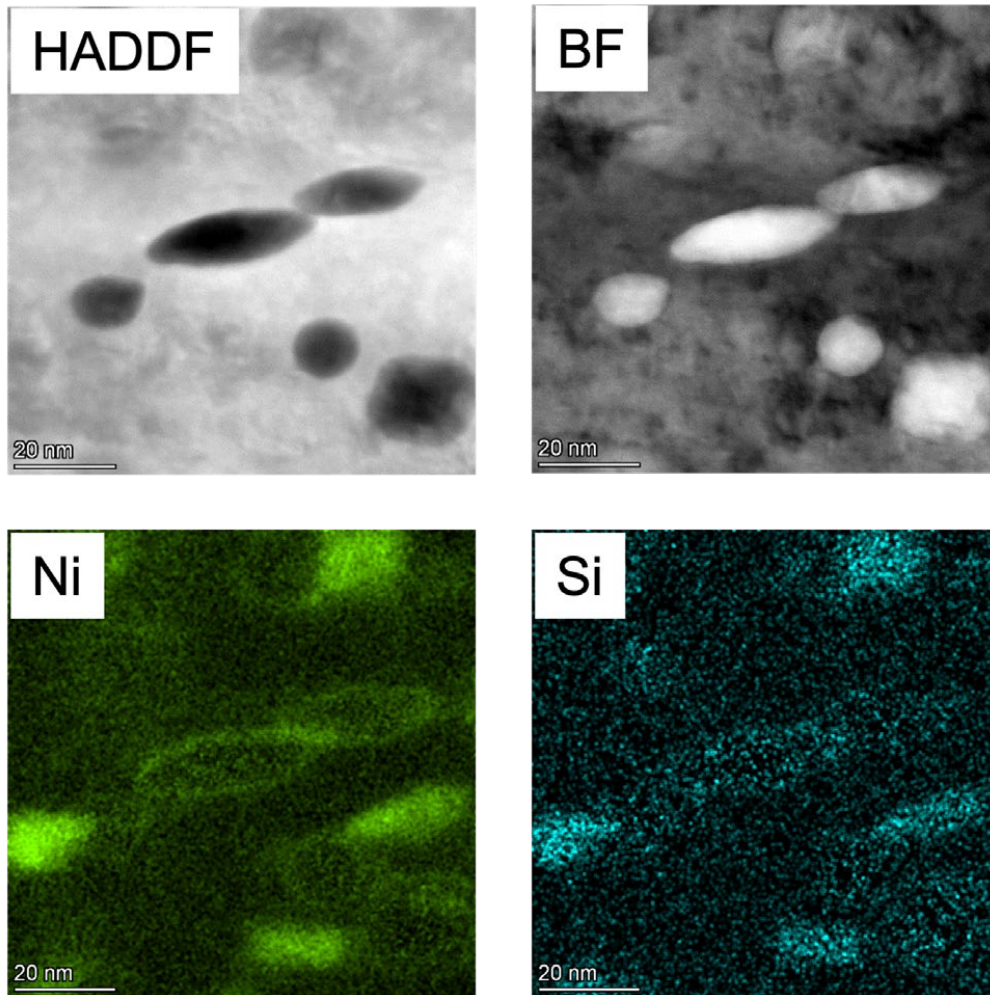


Figure 1. Microstructure and microchemistry of high dose, fast neutron irradiated 316L SS after tension test.

examination is consistent with the ring tensile tests of irradiated specimens; i.e., the elongation-to-failure decreases from ~75 to 5%. Although the irradiated specimen became brittle, an enlarged SEM micrograph shows band structures in the grain interiors. TEM characterization in the next section reveals that these band structures are deformation-induced twins. Researchers examined the chemical analysis of deformed reference and irradiated specimens using EDS and wavelength-dispersive spectroscopy. The EDS mapping of Fe, Cr, Ni, Mn, Mo, and Si indicates the marked region in the SEM image. There was no clear evidence of chemical variations within the dimple features in the deformed reference sample. Researchers prepared TEM lamellae from the deformed region of the tested ring specimens. Microstructure and chemical analysis were performed for the eight specimens (one reference and seven irradiated specimens) through TEM and EDS. TEM micrographs reveal that deformation twinning is predominant in the irradiated specimen. EDS mapping of the deformed irradiated specimen shows that the previous spherical voids become elongated along the twin interfaces. In addition, the Ni-Si

enriched precipitates were sheared and became elongated along twin interfaces. Voids with Ni and Si enrichment are also sheared in the twin domain and become elongated.

### **Discussion:**

Understanding the deformation mechanisms of fast-neutron-irradiated alloys is essential to developing novel structural materials for fast nuclear reactors. This research provides new insights into the swelling-induced embrittlement in austenitic 316 SS subjected to high-dose, fast-neutron irradiation. PIE reveals a high density of voids, decorated by Ni and Si phase formed in the microstructure. Voids provide a strong strength barrier for dislocation movement. During tensile tests at room temperature, deformation twinning is the predominant deformation mechanism in irradiated 316 SS at high swelling levels. The interaction of twins and voids causes void elongation, shrinkage, and linkage, leading to localized deformation. The fundamental understanding gained in this project provides a scientific basis for developing irradiation tolerant materials for fast reactors.

### **Conclusion:**

In this project, the team characterized the microstructure and microchemistry of high-dose, neutron-irradiated AISI 316 SS. Researchers identified irradiation-induced void swelling and chemical redistribution (i.e., chemical segregation near voids, grain boundaries, and precipitation). The high density of voids and Ni-Si precipitates formed in the grain interiors, and voids are decorated with the Ni-Si phase. Ring hoop tensile testing showed that elongation after yielding strongly depends on the void swelling. The fractography of fracture surfaces revealed that the dimple-like failure mode gradually turns to cleavage failure mode as the swelling increases, suggesting the vital role of void swelling in the deformation mechanisms. Deformation twinning is predominant in the irradiated specimens during the tension test at room temperature. The shearing of Ni- and Si-enriched voids and precipitation during twinning could further cause the hardening and embrittlement. This work provides new insights into the deformation behavior of fast-neutron-irradiated SS and can be applied to design and synthesize novel materials for advanced nuclear systems.

### **Future Activities:**

Further understanding of deformation mechanism at high dose by atomistic modeling and simulation.

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**Publications**

Two manuscripts in preparation.

**Distributed Partnership at a Glance**

NSUF Institution	Facilities and Capabilities
Idaho National Laboratory	Electron Microscopy Laboratory Hot Fuel Examination Facility Irradiated Materials Characterization Laboratory
Westinghouse Electric Company	Churchill Laboratory Services
Collaborators	
Idaho National Laboratory	Cheng Sun (Principal Investigator), Douglas L. Porter (Co-Principal Investigator), Wen Jiang, (Co-Principal Investigator)
Idaho National Laboratory	Boopathy Kombaiah (Team Member), Mukesh N. Bachhav (Team Member)
Texas A&M University	Frank Garner (Co-Principal Investigator)
Westinghouse Electric Company	Michael Ickes (Team Member)