Advanced Multi-Scale Post-Irradiation Experiments Link the Mechanical Properties and Deformation Mechanisms of In-Core Inconel X-750 Spacers

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1. Abstract / Introduction

Inconel X-750 has excellent high temperature strength and creep properties, making it suitable as a structural component in the form of fuel-assembly hold-down springs, control guide-tube support pins, jet pump beams, and core internal bolts in LWRs, and as a fuel channel annulus spacer in CANDUs. CANDU spacers function in an elevated thermal neutron flux spectrum, almost an order of magnitude higher than the average thermal neutron flux spectrum in a PWR core. Spacer regions separating the pressure tube and calandria tube operate at 120-280°C, whereas regions not in contact with the fuel channel operate at 300-330°C. More extreme thermal neutron environments increase the number of (n,a), (n,p), and (n,γ) transmutations, more than doubling the total displacement damage seen by the material, which reaches 1.74-3.95 dpa/yr, when summed with contributions from fast-neutron direct collision cascades. Total displacement damage in core Inconel X-750 material will reach ~100 dpa; components removed for post-irradiation experiments in this study range from 0-84 dpa. In addition, inflated levels of internal He and H gas, exceeding 3.5 at% He and 0.6 at% H, are expected in the highest dose material. He bubbles formed during irradiation were linked to grain boundary embrittlement and intergranular failure in the material [1] and are expected to play a major role in the evolution of mechanical properties and deformation mechanisms.

An extensive program of post-irradiation experiments on ex-service Inconel X-750 has led to the development of novel component-scale, meso-scale, and micro-scale mechanical tests, employing advanced techniques in the Fuels and Materials Cells (FMC), shielded SEM, active FEI Versa Focused Ion Beam (FIB) workstation, active TEM, and Hysitron PI 88 Picoindenter used for in-situ SEM testing at CNL; as well as the active FEI Helios plasma FIB workstation in IMCL at INL. The test procedures and results from these mechanical tests, coupled with pre and post-test SEM and TEM microscopy to link the mechanical properties and deformation mechanisms of in-core Inconel X-750 will be presented in this study.

2. Component-Scale Testing and Fractography Coupled with Post-Test TEM

Sections of spacers are compressed in a modified MTS Insight 50 device at a displacement controlled rate of 0.025 mm/min until a drop in the load-displacement curve is observed to measure the material's load bearing capacity. Subsequent fracture surface imaging in the shielded SEM reveals mixed-mode intergranular and transgranular features. Figure 1 depicts fracture surfaces at various doses; a ductile
ligament is found at 5 dpa, but at higher doses only brittle surfaces are observed. Post-test TEM lamella extracted from fracture surfaces similar to those in Figure 1 display evidence of local plasticity in the form of nanotwinning and slip band deformation. Nanotwins only shear the He bubbles by several lattice planes, whereas more severe bubble elongation was seen within each deformation slip band, as shown in Figure 2.

![Fracture surfaces from ex-service crush tested Inconel X-750 spacers.](image)

**Figure 1.** Fracture surfaces from ex-service crush tested Inconel X-750 spacers.

![TEM lamella extracted from bulk fracture surfaces of an Inconel X-750 spacer irradiated to 84 dpa at 300-330°C, 3.91 dpa/yr.](image)

**Figure 2.** TEM lamella extracted from bulk fracture surfaces of an Inconel X-750 spacer irradiated to 84 dpa at 300-330°C, 3.91 dpa/yr: a) A nanotwin in the deformed region elongates a He bubble by several lattice planes. b) A deformation slip band (between the red dashed lines) severely elongates He bubbles.

### 3. Microhardness Testing

Vickers microhardness testing performed at 500 gf on cross-sections of ex-service Inconel X-750 material ranging from 0-84 dpa is presented in Figure 3. Using the relationship provided in [2], hardness values are converted to 8% flow stress of the material. An initial hardness increase up to 5 dpa can be attributed to the introduction of dislocation loops. The subsequent decrease, 5-40 dpa, is a result of the disordering and dissolution of γ' strengthening nanoprecipitates. Radiation effects appear to saturate > 40 dpa, with the lower temperature material exhibiting lower hardness/flow stress due to smaller radiation defects serving as weaker barriers to dislocation motion.

![Representative ex-service Inconel X-750 cross-section with Vickers microhardness indents (left). Microhardness and 8% flow stress of Inconel X-750 as a function of dose (right).](image)

**Figure 3.** Representative ex-service Inconel X-750 cross-section with Vickers microhardness indents (left). Microhardness and 8% flow stress of Inconel X-750 as a function of dose (right).

### 4. Micro-Tensile Testing Coupled with Post-Test TEM

Micro-tensile specimens (1.25 μm x 1.25 μm x 2.5 μm) were fabricated using the active FEI Versa FIB workstation with 4 geometries as shown in Figure 4 in order to investigate the decohesion and shear strength of the grain boundaries, observe the interactions of dislocation slip bands intersecting grain boundaries, and obtain the critical resolved shear stress to initiate plasticity independent of crystallographic orientation of the material as a function of dose and irradiation temperature. Specimens of all geometries shown in Figure 4 yielded in their grain interiors and subsequently failed along a heavily deformed slip trace, indicating that the grain boundaries are stronger than the matrix at all doses and irradiation temperatures.
and suggesting that the yield stress of the material can be used as a lower bound of its strength. However, in several cases, specimens with vertically oriented grain boundaries developed voids where slip bands intersected the boundary. Post-test TEM of one of these deformed specimens revealed that these voids nucleated at the interfaces of grain boundary carbides, implying that stress concentrations may develop here and promote intergranular failure in a fully constrained environment.

5. **Meso-Tensile Testing**

Tensile specimens (20 μm x 20 μm x 50 μm) were fabricated on the edge of the cross-section of a 67 dpa Inconel X-750 spacer that operated at 120-280°C as shown in Figure 5a-b using the plasma FIB workstation in IMCL at INL. In-situ SEM testing is expected to commence soon using a grip and pull test set up shown in Figure 5c. Meso-scale tensile specimens contain whole grains fully constrained within the interior of their gauge, and will address scaling effects associated with an increased surface area to volume ratio in the micro-tensile specimens. Direct observations of interactions between grain interior and grain boundary deformation with full constraint will show how the deformation propagates, and if certain grain boundary types are preferred. For Inconel X-750 spacers that have a complex geometry, large meso-scale tensile tests are the most representative way to measure a bulk like ductility parameter such as total elongation.

References
