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

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Background
Inconel X-750 Program History

• The work presented, along with additional microstructural characterization, accelerated irradiation programs (proton irradiations & HFIR), and multi-scale modelling, is a compilation of 10+ years assessing long term radiation effects on ex-service Inconel X-750.

• The goal of this work is to link the evolving microstructure and mechanical properties of irradiated Inconel X-750 at multiple length scales to determine deformation & embrittlement mechanisms.
Outline

1. Introduction
2. Material
3. Component Scale Investigations
   a. Crush Tests
   b. Fractography
4. Meso-Scale Investigations
   a. Microhardness
   b. Meso-Tensile Tests
5. Micro-Tensile Tests
6. Nano-Scale Investigations
Introduction
CANDU Core, Fuel Channel, & Inconel X-750 Spacer
Introduction
CANDU Inconel X-750 Radiation Environment

1) Heavily thermalized n flux spectrum

2) Enhanced radiation damage: 1.7-4.0 dpa/yr

3) Inflated levels of H & He
Material

Inconel X-750 Composition & Microstructure

Chemical Composition [wt%] & Heat Treatments

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>C</th>
<th>Co</th>
<th>Cr</th>
<th>Cu</th>
<th>Fe</th>
<th>Mn</th>
<th>Ni</th>
<th>S</th>
<th>Si</th>
<th>Ti</th>
<th>Nb + Ta</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration</td>
<td>0.4-1</td>
<td>.08</td>
<td>1</td>
<td>14-17</td>
<td>0.5</td>
<td>5-9</td>
<td>1</td>
<td>70</td>
<td>0.01</td>
<td>0.5</td>
<td>2.25-2.75</td>
<td>0.7-1.2</td>
</tr>
</tbody>
</table>

Solution Anneal (SA) | 1093-1204 °C
Single Step Ageing | 732 ± 14 °C for 16.5 hrs., air cool

Strengthening Nanoprecipitates

\( \gamma' : \text{Ni}_3(\text{Al},\text{Ti}) \)

8.5 ± 1.3 μm avg. grain size
Material
Inconel X-750 Gamma Prime Stability

Non-Irradiated

Softens the material at intermediate & high doses!

5 dpa

superlattice reflections

20 dpa

superlattice reflections

53 dpa

120-280 °C

No γ' superlattice reflection detected (i.e. γ' disordered)

300-330 °C

Weak γ' superlattice reflection detected (i.e. γ' not fully disordered)

67-84 dpa

completely disordered & dissolved (both temps)

Material Inconel X-750 Gamma Prime Stability
Material

Radiation Defect & He Bubble Size Distributions

Radiation defects: 120-280 °C 300-330 °C

He Bubbles:

Higher temperature = larger defects & bubbles
Material

He Bubbles on Grain Boundaries

53 dpa, 1.7 at% He

Helium Bubble
Depletion Zone
Component Scale Investigations

Crush Tests

![Graph showing load vs. displacement for different conditions. The graph includes data points for non-irradiated samples and samples irradiated to 53 dpa and 67 dpa at temperatures of approximately 180 °C and 300 °C.]

![Image of crush test setup with 'CHALK RIVER LABS' label visible.]
Component Scale Investigations

Fractography

Non-Irradiated
TEM Investigations of Bulk Fracture Surfaces

Intergranular Feature

localized plasticity adjacent to IG crack
TEM Investigations of Bulk Fracture Surfaces

Transgranular Feature:
Nano-twinning & sheared He bubbles
Meso-Scale Investigations

Microhardness

\[ HV \approx 2.9 \sigma_y \]
Meso-Scale Investigations

Tensile Tests

Non-Irradiated

67 dpa, 2.1 at% He, $T_{irr} = 120-270 \, ^{\circ}C$
Micro-Tensile Tests

Push-to-Pull Micro-Tensile Sample Preparation
Micro-Tensile Tests

Push-to-Pull Micro-Tensile Set-Up and Test Geometries

**Specimen Geometries**
- Single-Grained 1 µm
- 45° Mixed-Mode 1 µm
- Vertical Boundary 1 µm
- Decohesion 1 µm

**Isometric View**
- Spring
- Si Push-to-pull (P2P) device
- Mounting location for a test specimen
- Location for compressive loading

**Test Specimen**
- 2.5 µm
Micro-Tensile Tests

Single-Grained Specimens -> Critical Resolved Shear Stress

53 dpa, 1.7 at% He
Micro-Tensile Tests
High Angle Grain Boundary Specimens

84 dpa, 2.6 at% He debond notched at GB

53 dpa, 1.7 at% He 45 degree notched at GB

Graph showing stress ($\sigma_Y$) vs. dose (dpa) for different conditions:
- Non-Irradiated
- Pinched > 3 dpa/EPFY
- Pinched < 3 dpa/EPFY
- Non-Pinched < 3 dpa/EPFY
- Non-Pinched > 3 dpa/EPFY
Micro-Tensile Tests

Deformation Mechanism #1: Transgranular He Bubble Shear, Elongation & Coalescence

severe bubble elongation in adjacent grain interior

severe bubble shear/elongation
Deformation Mechanisms
Deformation Mechanisms Summary

Deformation Initiation: Localized Plasticity

Shear magnitude determined by nano-twin width: limited
(a few lattice planes = ~10% bubble elongation)

Shear magnitude determined by number of dislocations passing through: unrestricted

> 100% bubble elongation
Transgranular Deformation Slip Mechanism

defformation increases

Slip Band Formation
Bubble Elongation
Bubble Elongation/Coalescence
Fracture

bulk: flat facet fracture features in grain interiors

μm: Slip step traces in sub-size tensile specimens

nm: severely elongated/coalesced He bubbles in slip steps
Intergranular Crack Initiation Mechanism

1. Slip bands intersect rigid GB carbide interfaces
2. Dislocations pile up at interface & increase pressure
3. He bubbles elongate in GB plane, coalesce and create IG crack

Voids at GB-carbide interfaces

nm: TEM lamella from bulk component test
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Questions? Comments?
Backup Slides
Introduction
CANDU Reactor System
Material
Inconel X-750 Temperature History

In Contact

Out of Contact

![Temperature History Graphs](image-url)
Material

He Bubble Distribution Plots

Grain boundary bubbles consistently larger than grain interior bubbles!
Material

He Bubble Grain Interior Dose Evolution

Bubble Size

Bubble Density

Bubble Volume

120-280 °C

Diameter Range [nm]

Number Density [10^{24} m^{-3}]

Volume %

Size remains constant, density increases

300-330 °C

Diameter Range [nm]

Number Density [m^{-3}]

Volume %

Size, volume, & density all increase
Material

He Bubble Grain Boundary Area Coverage Dose Evolution

**120-280 °C:**

**300-330 °C:**

![Graph showing fractional area coverage vs. dose for two temperature ranges.](image-url)
Mechanism of Fracture #2 (Intergranular)

- Slip bands intersect with compromised grain boundaries/twins.
  - Dislocation pile-up at boundaries cause grain boundary bubbles to compress, increase in pressure, resulting in either dislocation emission to neighbouring grain or bubble expansion along boundary plane, leading to coalescence and crack initiation.
Mechanism of Fracture #2 (Intergranular)

Low dose, $\downarrow AC\% + \uparrow \varepsilon = IG$ Fracture

- Grain Boundary
- Helium Bubbles
- Slip bands hit Boundary
- Bubbles compress, increase pressure, and expand on plane of boundary
- Fracture

High dose, $\uparrow AC\% + \downarrow \varepsilon = IG$ Fracture

- Grain Boundary
- Helium Bubbles
- Slip bands hit Boundary
- Bubbles compress, increase pressure, and expand on plane of boundary
- Fracture
Mechanism of Fracture #4 (Intergranular)

Once a crack is initiated, or a flaw at a boundary is created, propagation along a boundary can occur, assisted by the previously discussed mechanisms, or by growth and coalescence of GB He bubbles by local plasticity in the inter-bubble regions (requiring a critical coverage).

Courtesy of M Demkowicz, (Texas A&M)