Characterization of Neutron Irradiated CANDU Inconel X-750 Garter Springs Via Lift-Out, Three-Point Bend Tests

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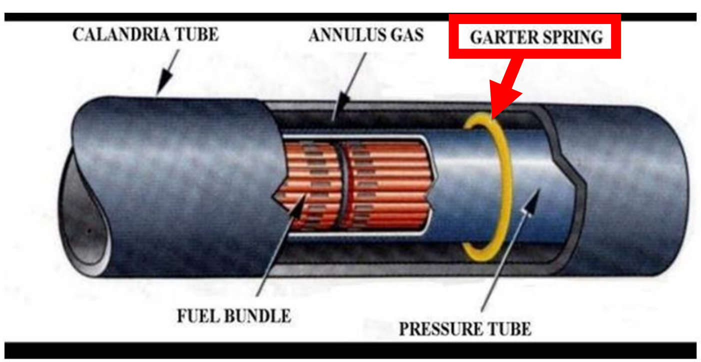
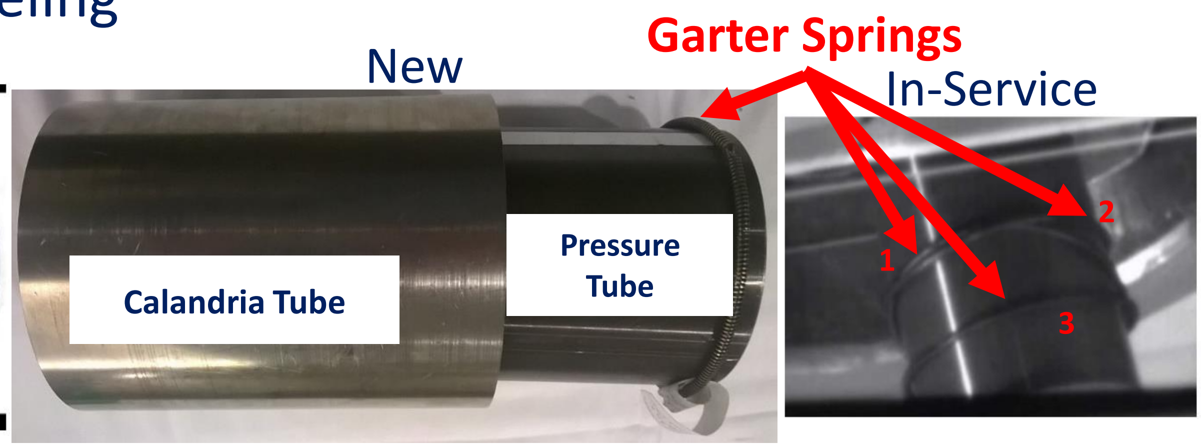
Outline

1. Background
2. Motivation
3. Specimen Preparation Methods
4. *In-Situ* Three-Point Bend Testing
5. Mechanical Data
6. Post Test STEM Observations
7. Summary
8. Future Work

Background

- CANDU reactor design
  - D_2O moderator & coolant -> natural U fuel
  - 380-480 hot pressure tubes housed in low pressure cold calandria tanks
    - ***separated & supported by Inconel X-750 garter springs (~1200-1500 in a reactor !!!)***
  - robotic online refueling





M. Griffiths. "The Effect of Irradiation on Ni-containing Components in CANDU® Reactor Cores: A Review." AECL Nuclear Review Vol. 2 Num. 1 (2013).

Background

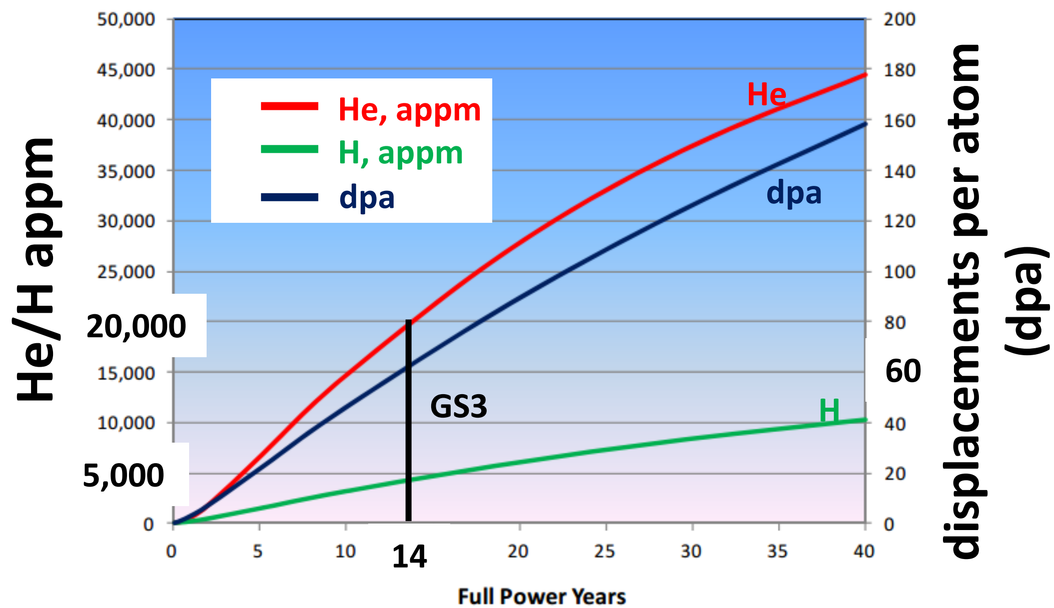
- Irradiation effects on Ni superalloys in CANDUs
  - naturally 68% ^{58}Ni

$$^{58}Ni + n \rightarrow {}^{59}Ni + \gamma \quad (1)$$

$$\mathbf{^{59}Ni + n \rightarrow {}^{56}Fe + {}^{4}He} \quad \mathbf{(2)}$$

$$^{59}Ni + n \rightarrow {}^{59}Co + {}^{1}H \quad (3)$$

$$^{59}Ni + n \rightarrow {}^{60}Ni + \gamma \quad (4)$$



M. Griffiths, et al. 2013, "Degradation of Ni-alloy Components in CANDU Reactor Cores", submitted to 16th Int. Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, Asheville, North Carolina, USA, August 11-15, 2013.

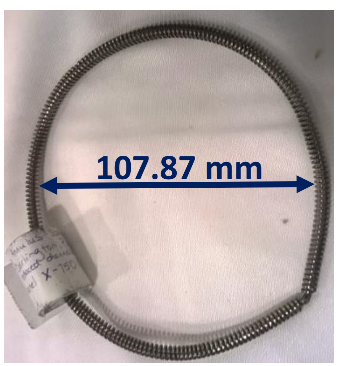
Background

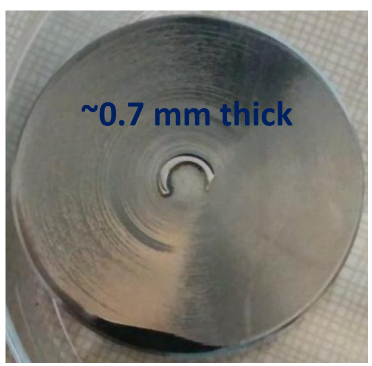
- Unirradiated Inconel X-750 garter springs

5.6 mm

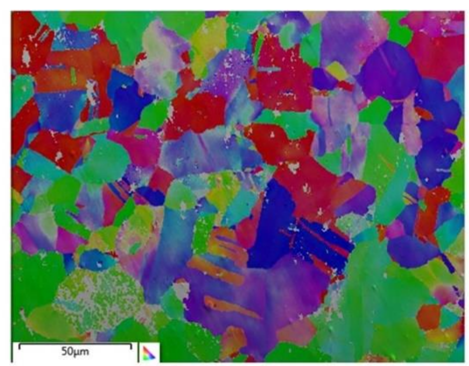
M. Griffiths, D. Poff, Z. Yao and K. Huang, 2013, "Performance of Ni alloy Components in CANDU Reactors", published in Proceedings of Int. Conf. on Materials Science & Technology(MS&T'13), Montreal, Quebec, Canada, October 27-31, 2013.

Element	Concentration (wt%)
Al	0.4-1
C	.08
Co	1
Cr	14-17
Cu	0.5
Fe	5-9
Mn	1
Ni	**70**
S	0.01
Si	0.5
Ti	2.25-2.75
Nb + Ta	0.7-1.2
Solution Treatment	1093-1204 °C
Precipitation Hardening	732 ± 14 °C for 16.5 hrs, air cool



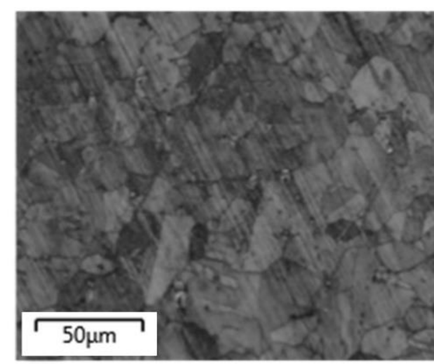


Grain Structure



EBSD

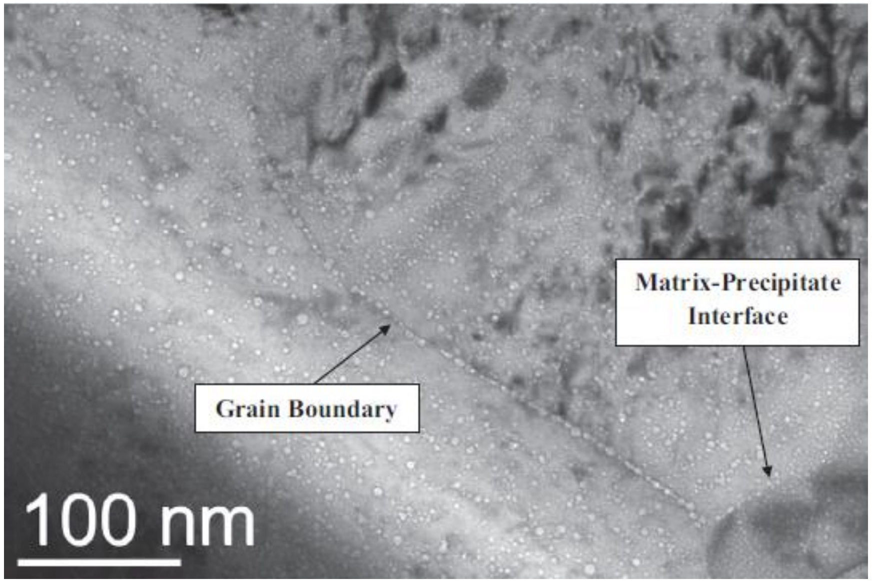
d ≈ 12 ± 8 µm



Kikuchi Band Contrast

Motivation

- Springs removed from CANDU-6 reactors after 14 service years exhibit severe intergranular failure upon handling
  - excessive He bubble accumulation on GBs



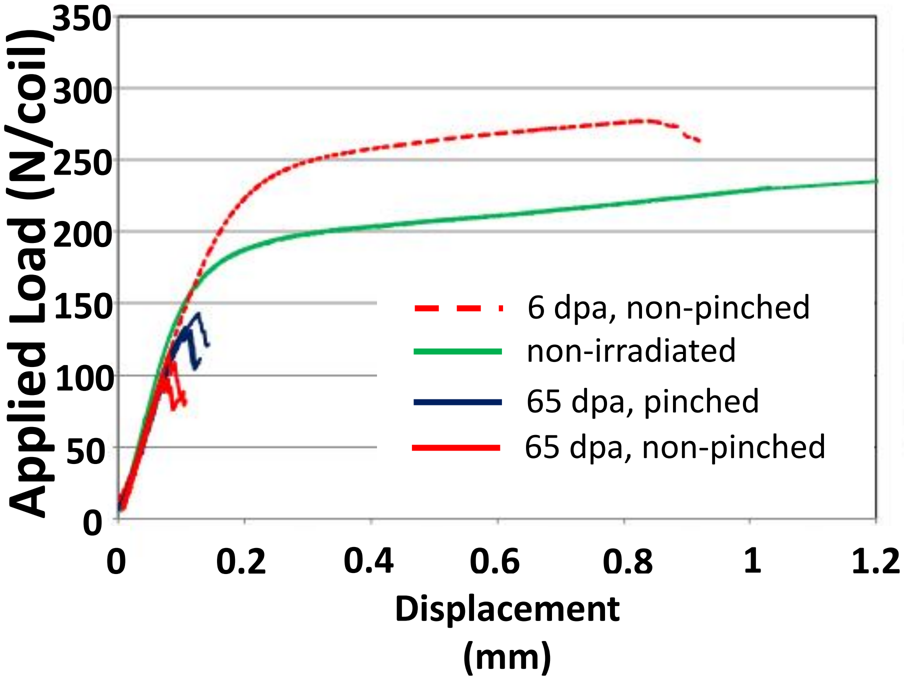
C.D. Judge et al. Journal of Nuclear Materials 457 (2015) 165–172

Motivation

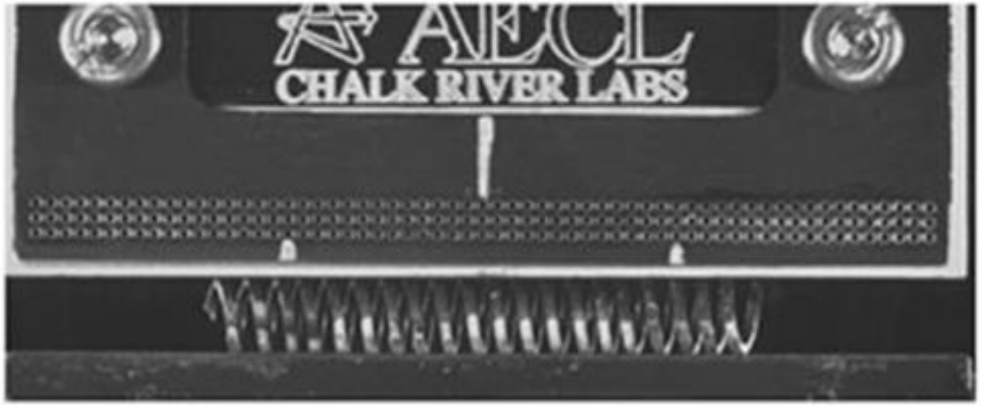
- Profound T effects (6 o’clock/pinched/low T vs. 12 o’clock/non-pinched/high T)

Motivation

- 0.7 mm thick -> difficult + expensive to quantify mechanical properties w/ conventional testing & get good statistics



Bulk Crush Test Apparatus



presumed weakest GB causes failure

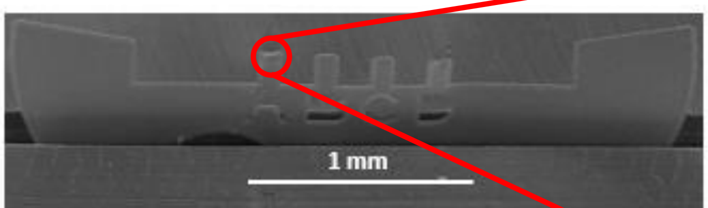
Motivation

- Use ***Small Scale Mechanical Testing*** to produce σ vs. ε to quantitatively compare:
  - mechanical properties differences caused by T effects (6 o’clock vs. 12 o’clock)
  - matrix strengths vs. GB strengths

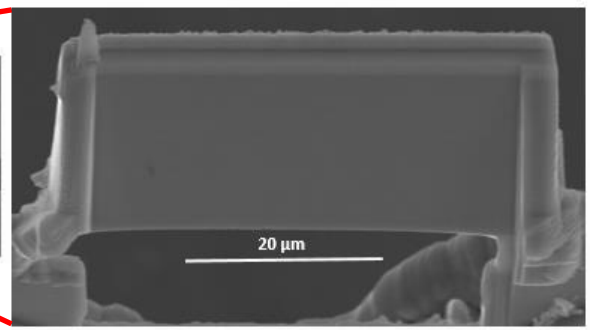
- Extend quantitative picture of mechanical properties degradation to all nickel alloy power reactor components

Specimen Preparation Methods

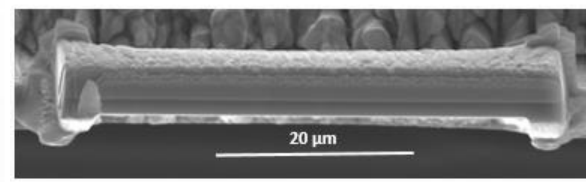
- Two initial “stock” foils FIB milled, lifted out, mounted in TEM grids and shipped to UC Berkeley



GS3-6



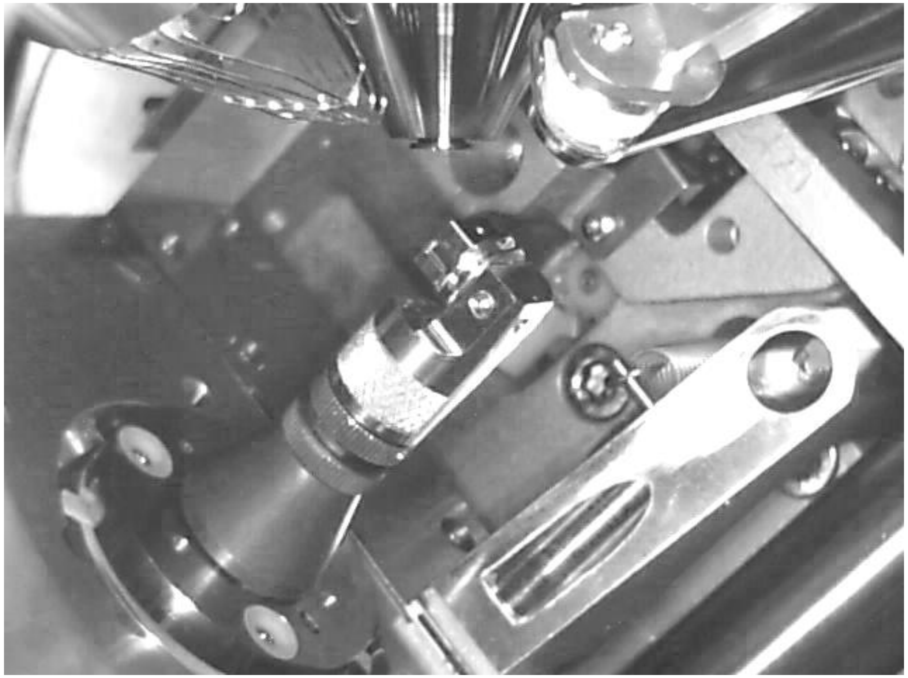
Side View

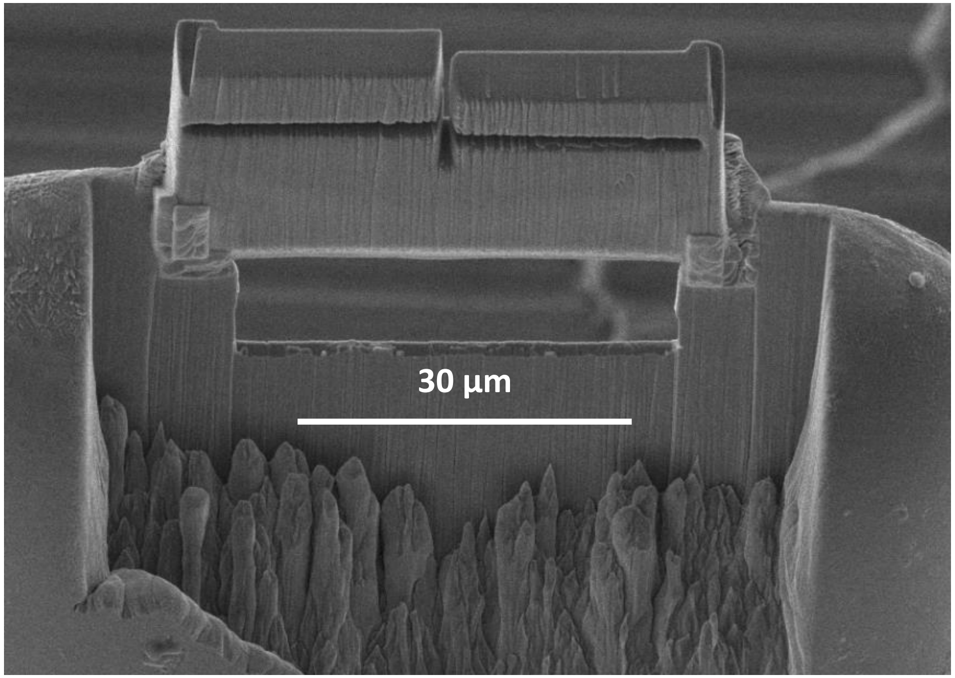


Top View

Specimen Preparation Methods

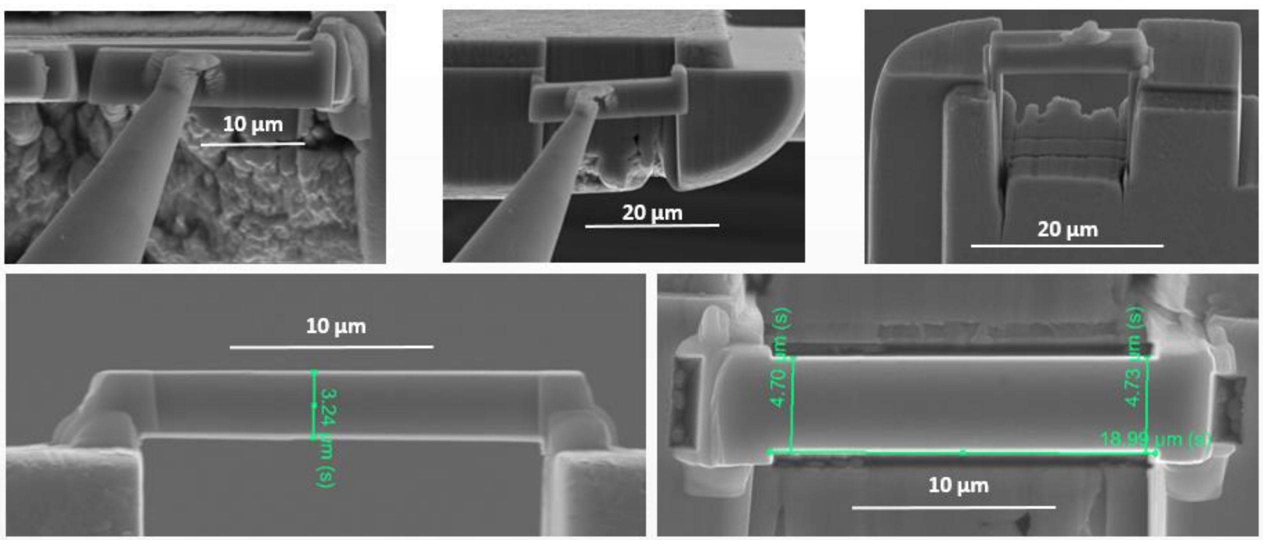
- Three point bend specimens manufactured using a homemade 90^{o} mount with a Ga^{69+} FIB in a FEI Quanta 3D FEG at the UC Berkeley BNC

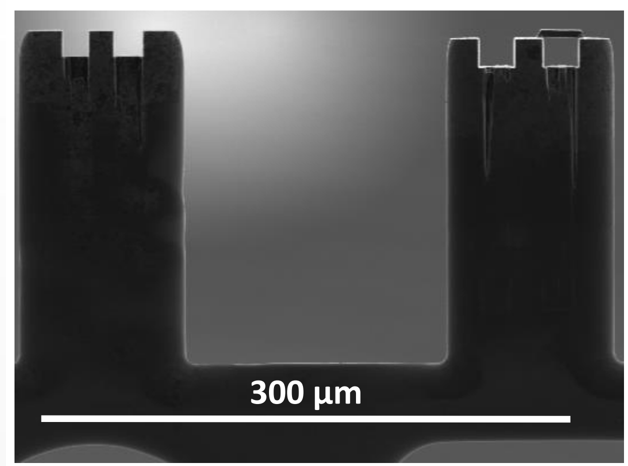




Specimen Preparation Methods

- Each three point bend specimen removed from "stock" foil and mounted across pre FIB fabricated bridge testing area using Kleindiek Nanotechnik manipulator and FIB cleaned to final dimensions

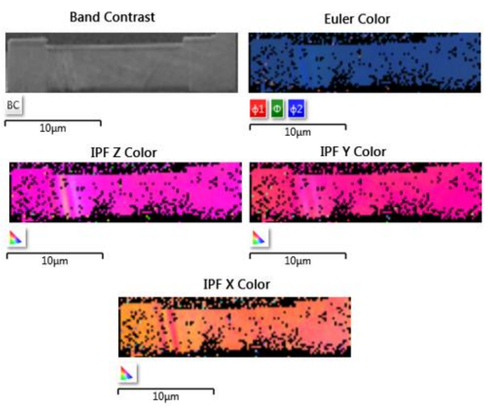




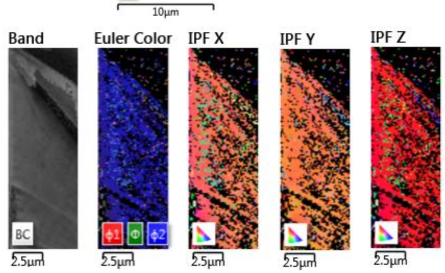
Pre-Test EBSD

GS3-6 (No GBs)

Top:

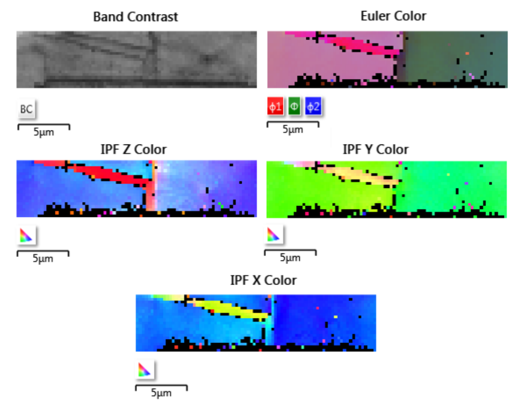


Front:

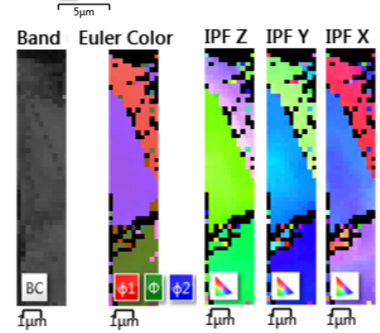


GS3-12 (GBs)

Top:

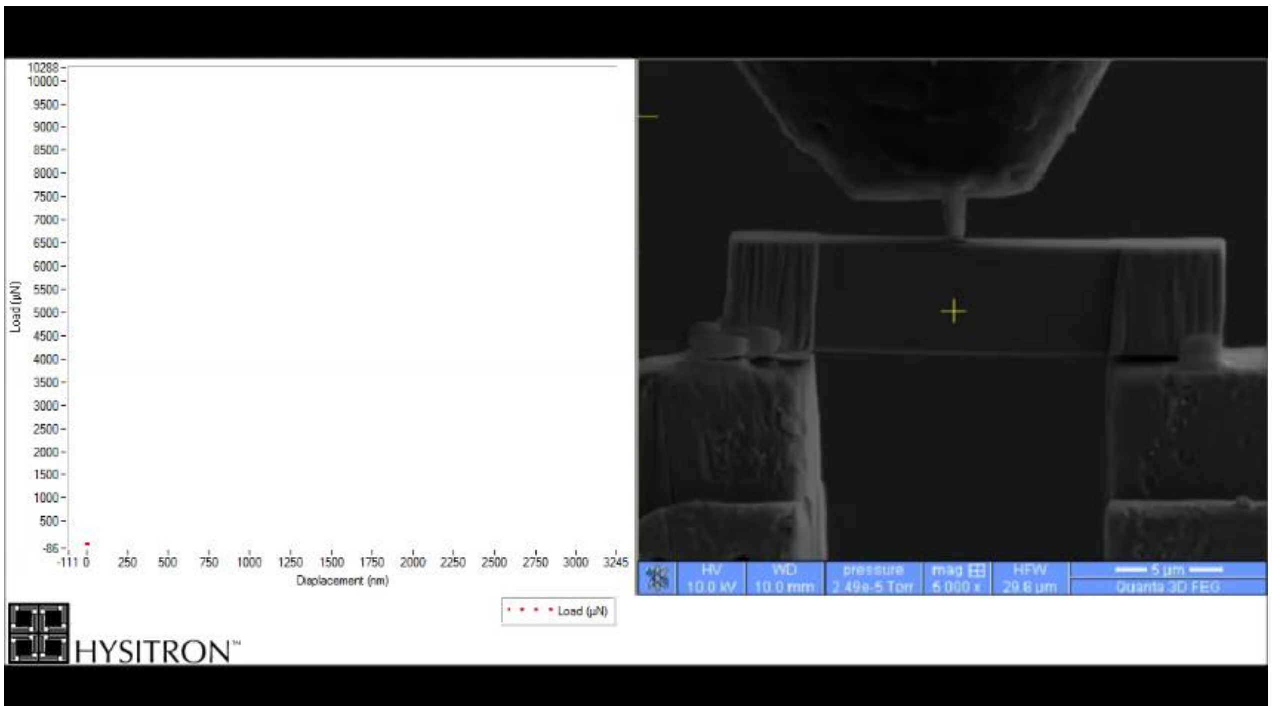


Front:



In Situ Three Point Bending

- Displacement controlled tests performed at 15 nm/s loading and 30 nm/s unloading using Hysitron PI 85 Pico Indenter



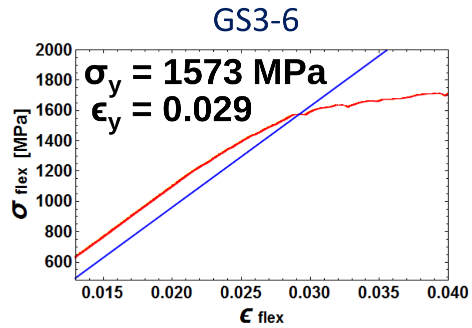
Initial Mechanical Data: Analysis

- Convert recorded F vs. D curves to σ vs. ε using the following:

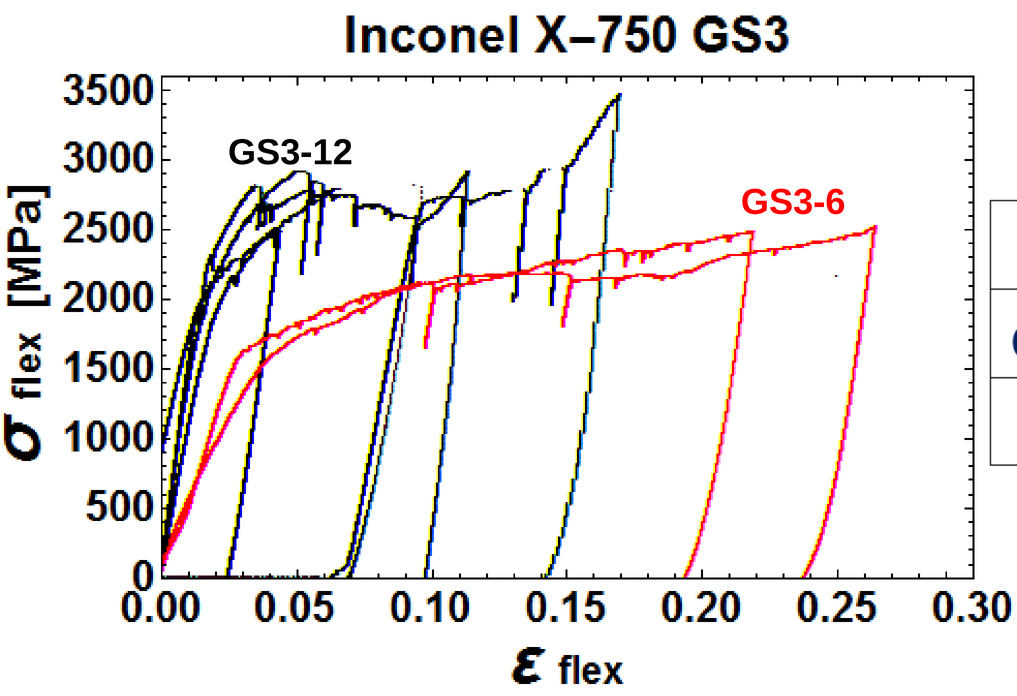
$$\sigma = \frac{3\,F\,L}{2\,b\,d^2} \qquad \varepsilon = \frac{6\,D\,d}{L^2}$$

σ = stress at midpoint (MPa)
ε = strain in outer surface at midpoint
F = load at midpoint (N)
D = displacement at midpoint (mm)
d = beam height (mm)
L = beam length (mm)
b = beam width (mm)

- Fit linear elastic portion and apply 0.2% offset, intersection is σ_y



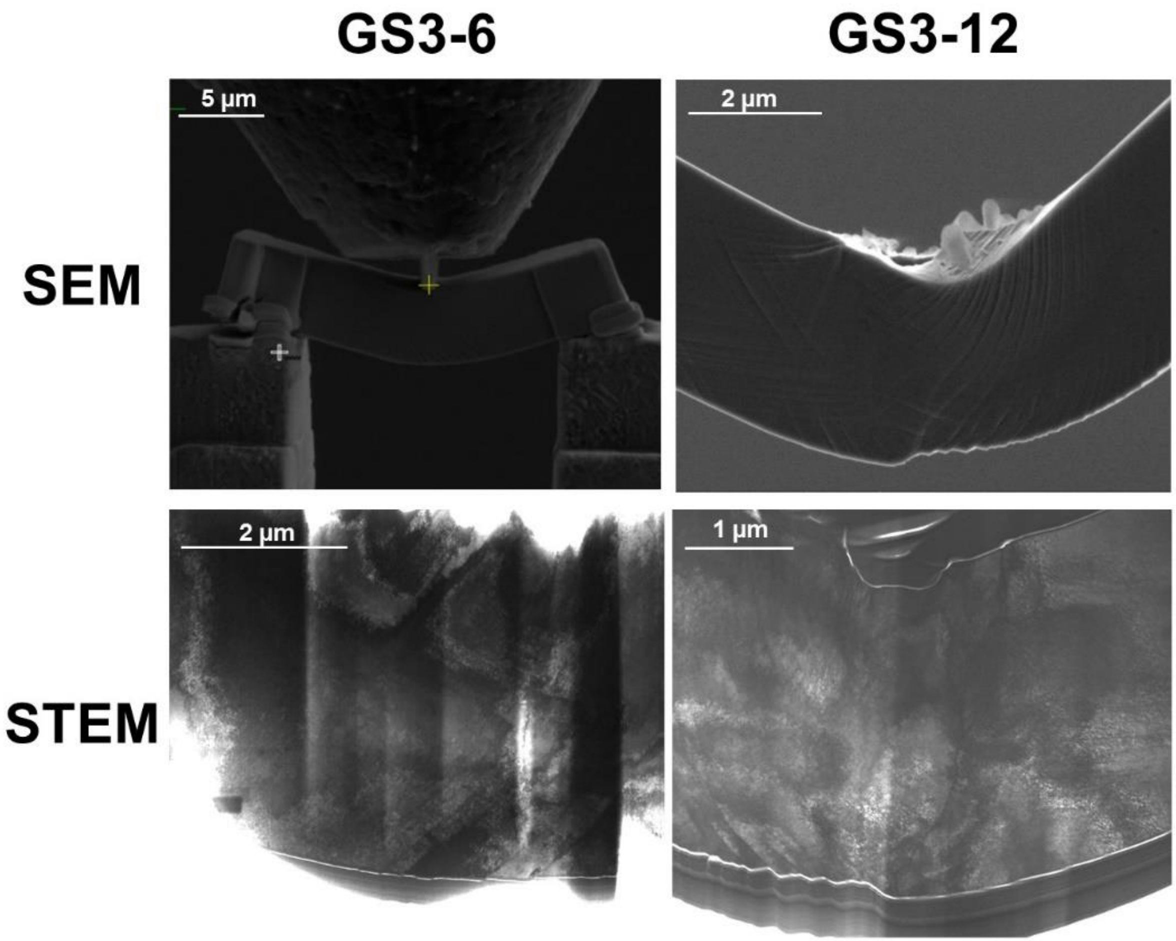
Initial Mechanical Data: Results



GS3-12 High Temperature (GB): 4 tests
GS3-6 Low Temperature (Sxx): 2 tests

	GS3-6	GS3-12
σ_y [MPa]	1561 ± 12	1871 ± 77
ε_y [%]	3.4 ± 0.5	1.6 ± 0.3

Post Test STEM Observations (BF)



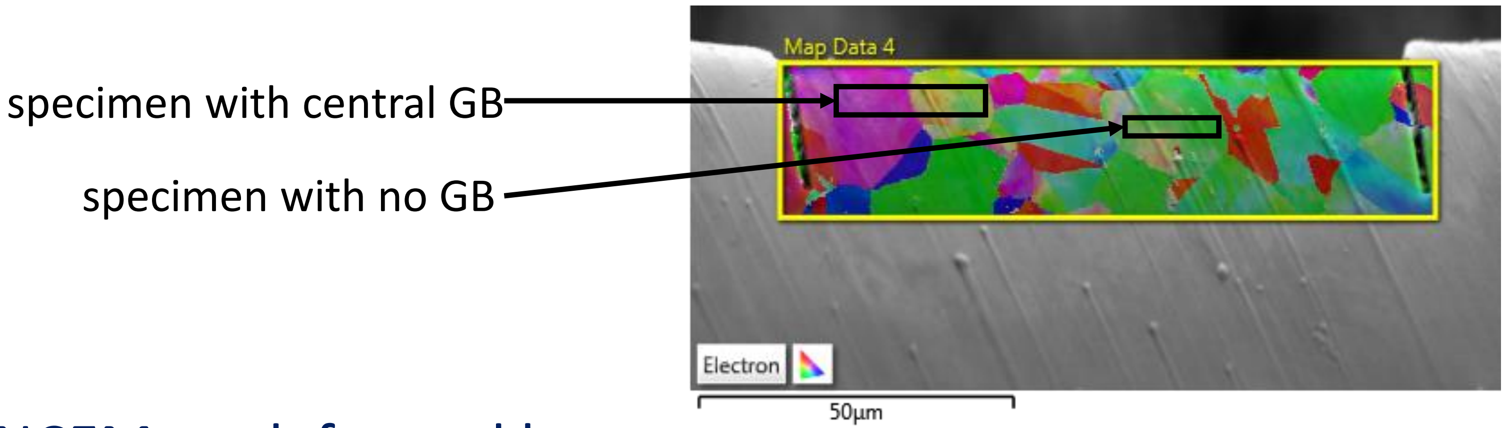
Summary

- developed novel ***Small Scale Mechanical Testing*** technique for quantitatively characterizing reactor components (Inconel X-750 garter springs) with challenging geometries using tiny amounts of material!
- measured bulk matrix properties (σ_y) of GS-6 and GS-12 in good agreement with hardness testing
  - GB effects yet to be determined

Future Work

- Obtain a full sample matrix: GS3-6, GS3-12, and unirradiated three point bend tests that both contain GBs and do not contain GBs in order to compare the effects of GBs, T, and n radiation on σ_y and ductility.

EBSD on unirradiated Inconel X-750



- TEM at NCEM on deformed bars
- Lift Out Tensile Tests to measure GB strength
- He implantation & mech. tests of unirradiated Inconel X-750 at different doses

Acknowledgements

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INL Idaho National Laboratory

NSUF Nuclear Science User Facilities

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