



Multi-scale Effects of Defects and Microstructure on Mechanical Properties of Nuclear Graphite

July 2024

Changing the World's Energy Future

Gongyuan Liu, William E Windes



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ADVANCED REACTOR TECHNOLOGIES PROGRAM



07/17/2024

Multi-scale Effects of Defects and Microstructure on Mechanical Properties of Nuclear Graphite

Gongyuan (Patrick) Liu

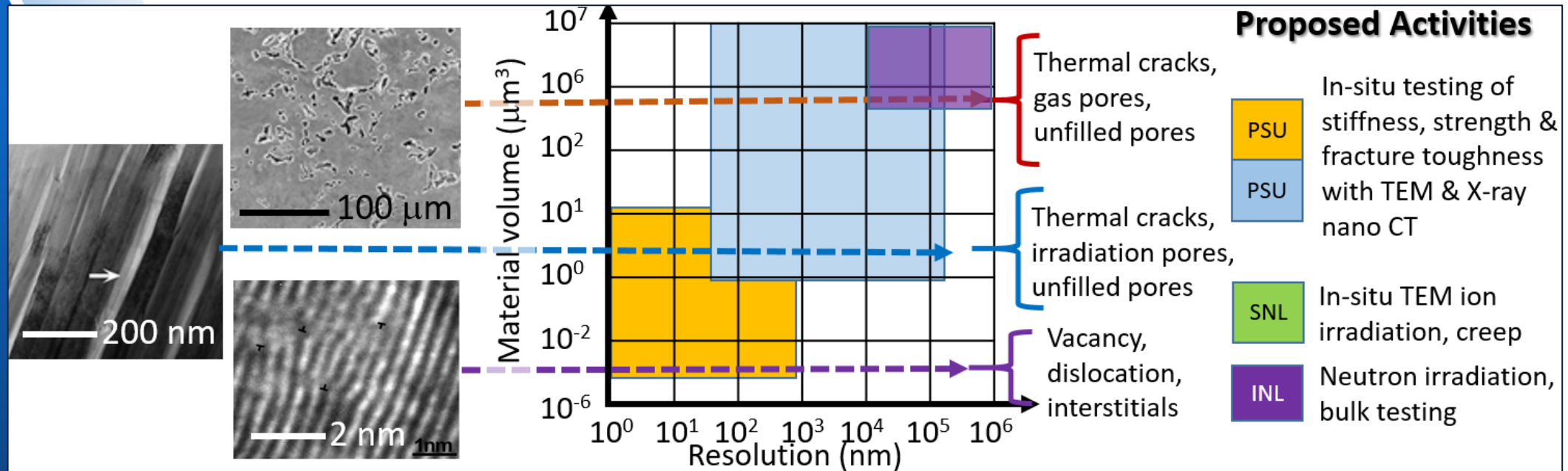
Department of Mechanical Engineering, The Pennsylvania State University

DOE ART GCR Review Meeting

Hybrid Meeting at INL

July 16–18, 2024

Research Goals



- Multi-scale interaction mechanisms among pre-existing and irradiation defects → Connecting to the bulk scale deformation and failure
- Deformation micro-mechanisms influenced by the constituent (filler, binder, interface), radiation displacement damage and temperature
- Stress localization (due to defect/microstructural heterogeneity and internal stress buildup during radiation) on the above-mentioned mechanisms

Outline

- **Micro-CT split disc fracture test**
- In situ TEM compression/creep testing after ion irradiation
- Room temperature annealing of graphite



Objectives of Preliminary Study

- Establish a new experimental method to investigate crack initiation and propagation.
- Neutron irradiated samples will be tested.

Material: NBG-17 Nuclear Graphite

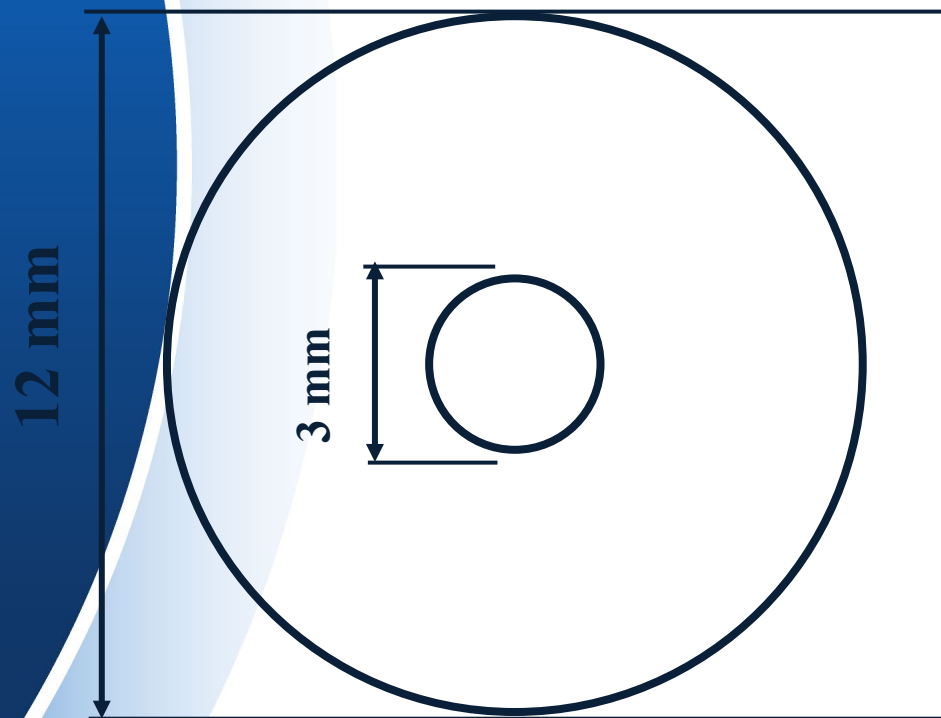
- Vibration molded with a grain size of 800 μm
- **Filler:** Pitch coke
- **Binder:** coal tar pitch



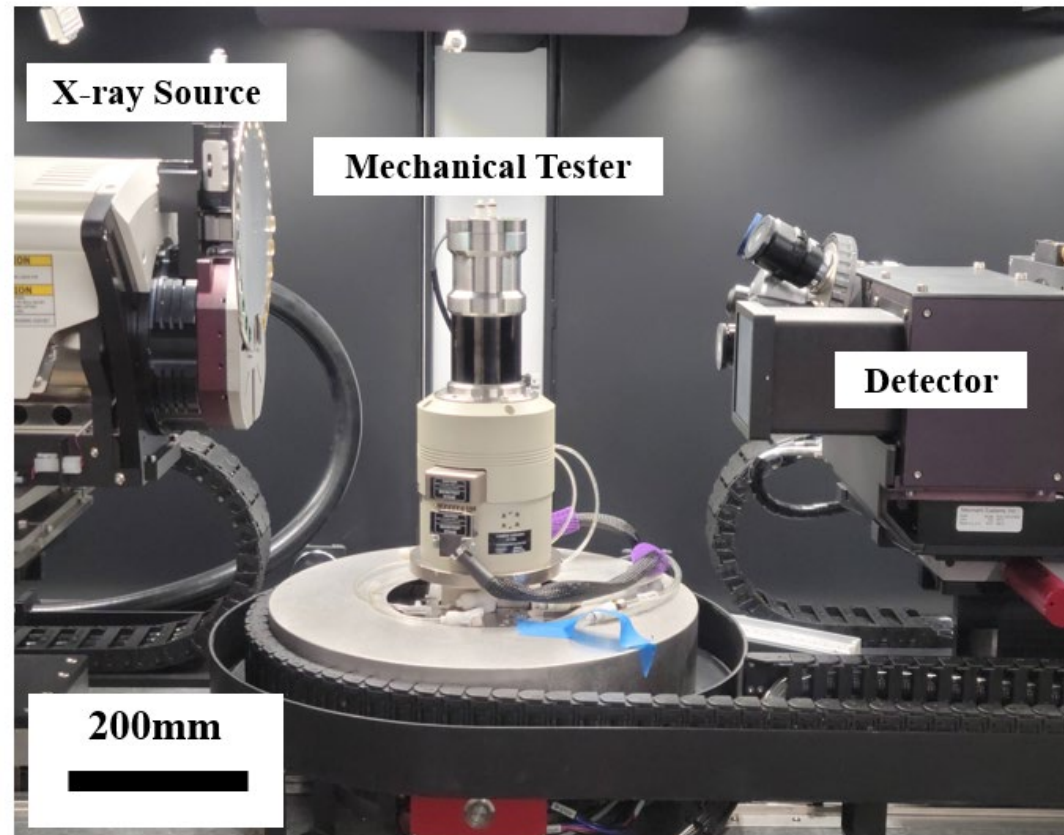
Methodology: Experimental Setup

- Incremental split disc test on NBG-17 button sample under load control.

Dimensions of button specimens



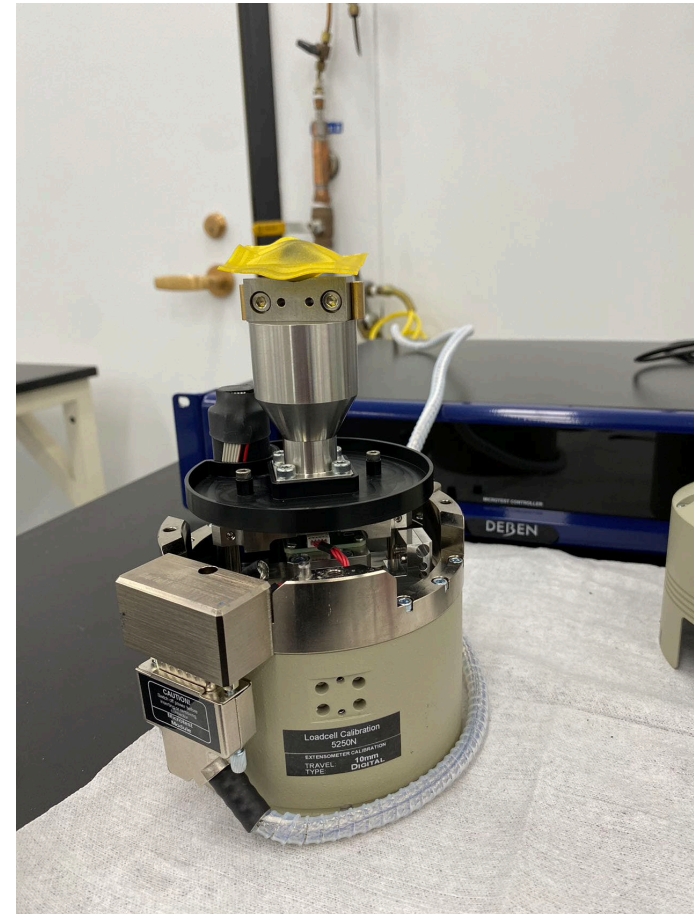
In-situ micro-CT mechanical testing



Mechanical tester (CT5000, Deben, Suffolk, UK)
Micro-CT (Xradia 620 Versa, ZEISS, Jena, Germany)

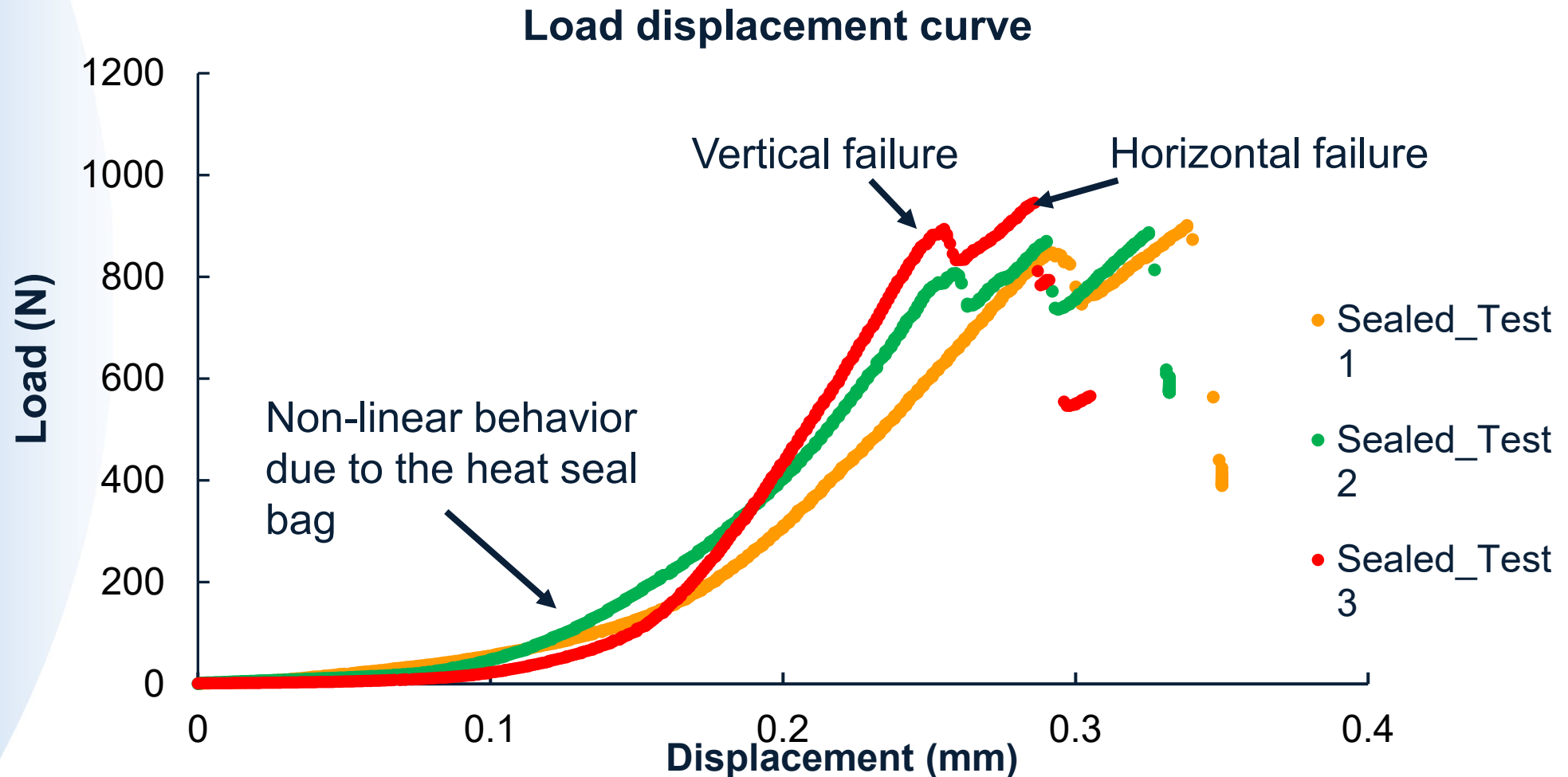
Micro-CT split disc test with heat seal bag

- Due to the safety requirement of Idaho National Lab, the irradiated samples need to be completely sealed by the heat seal bag for mechanical testing.



Load-Displacement Curve

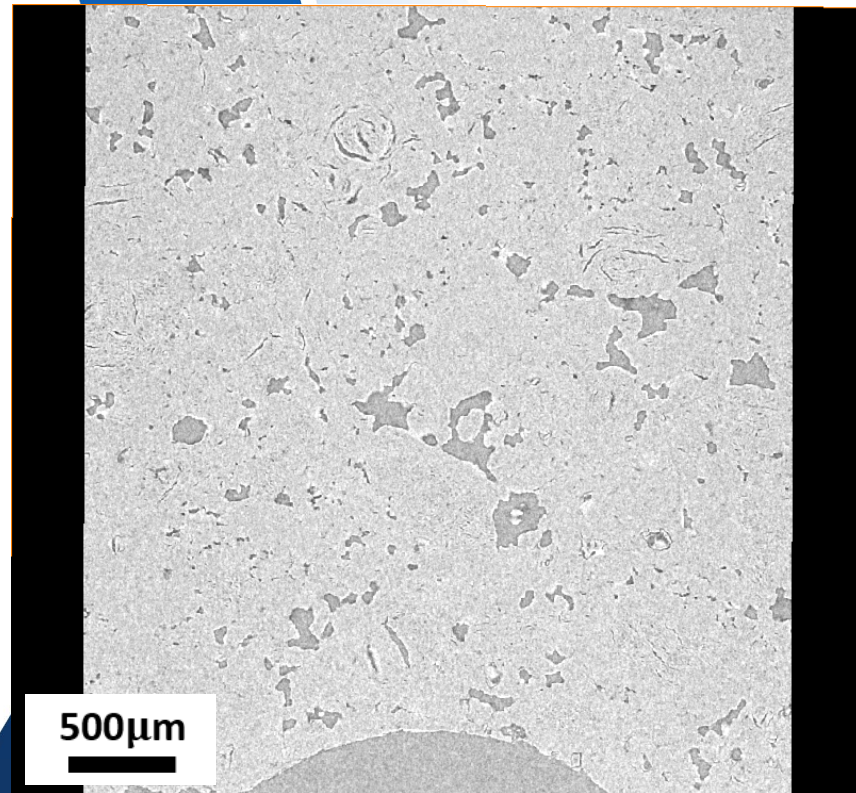
- All curves have a similar trend despite subtle differences in maximum loads



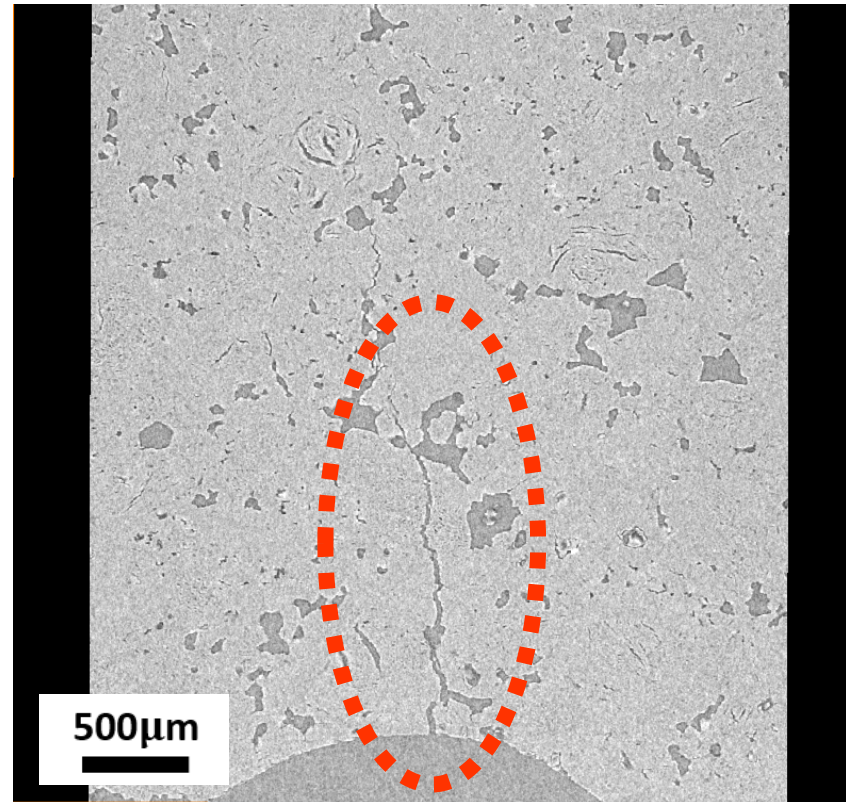
Micro-CT and *in situ* micro-CT

The NBG-17 graphite was scanned three times under different loads.

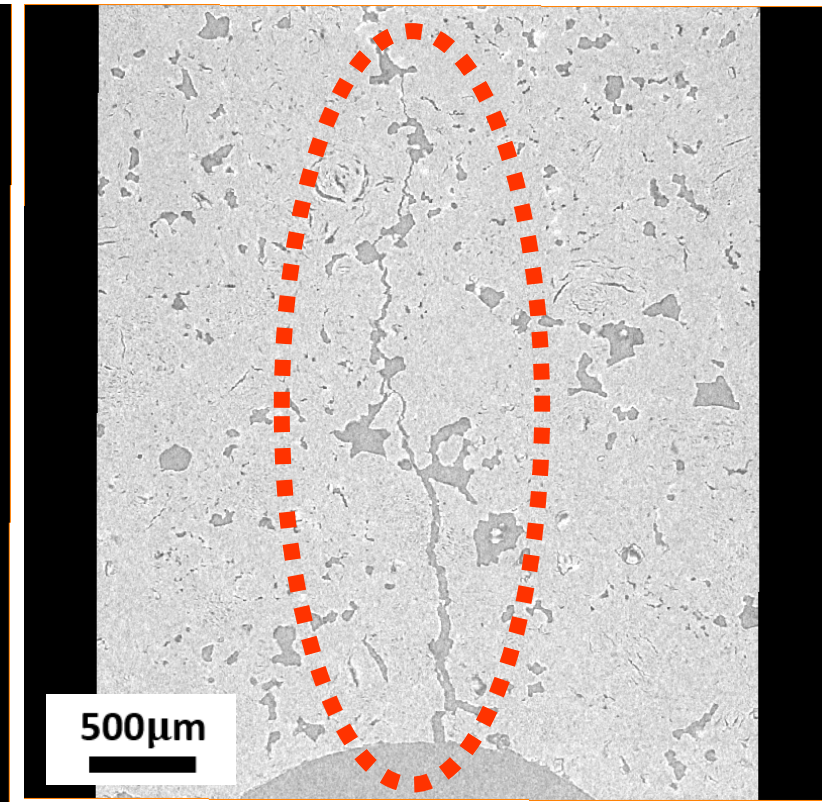
Scan 1: 0 N



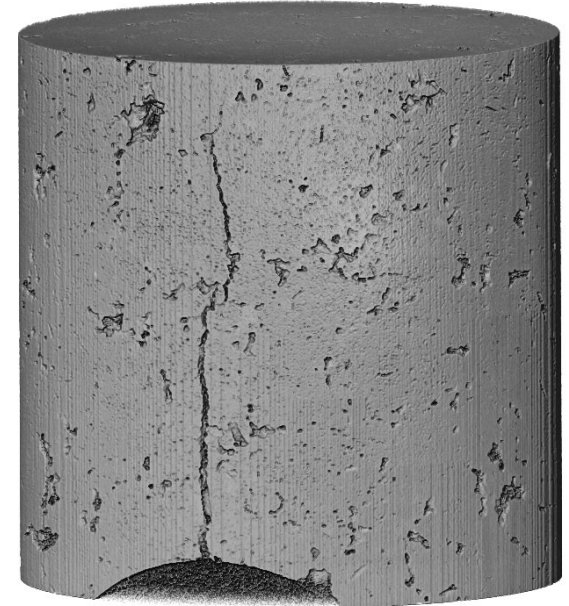
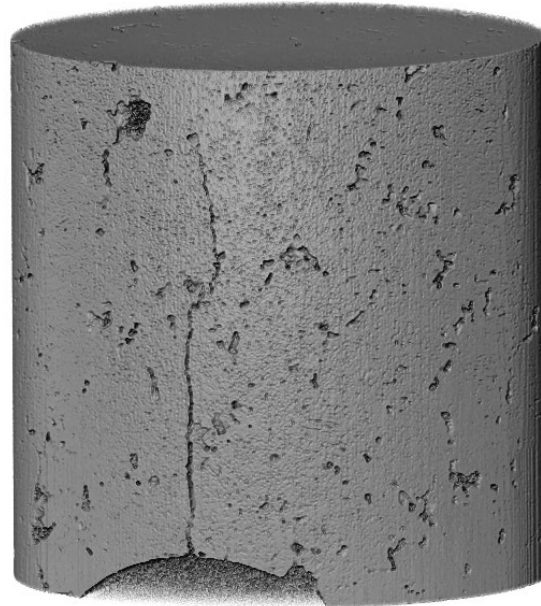
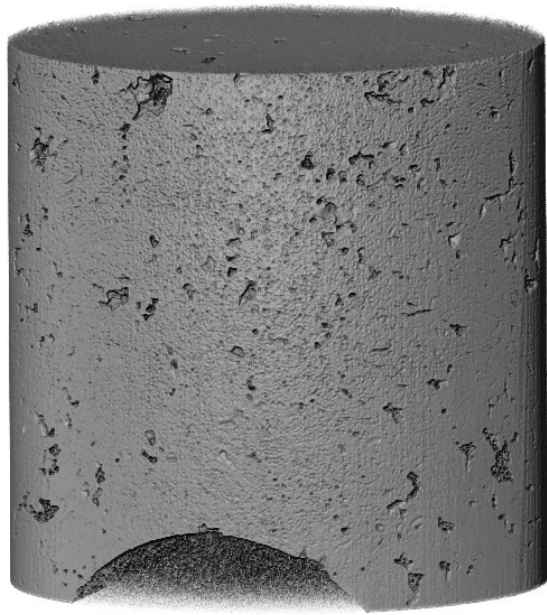
Scan 2: 890 N



Scan 3: 790N



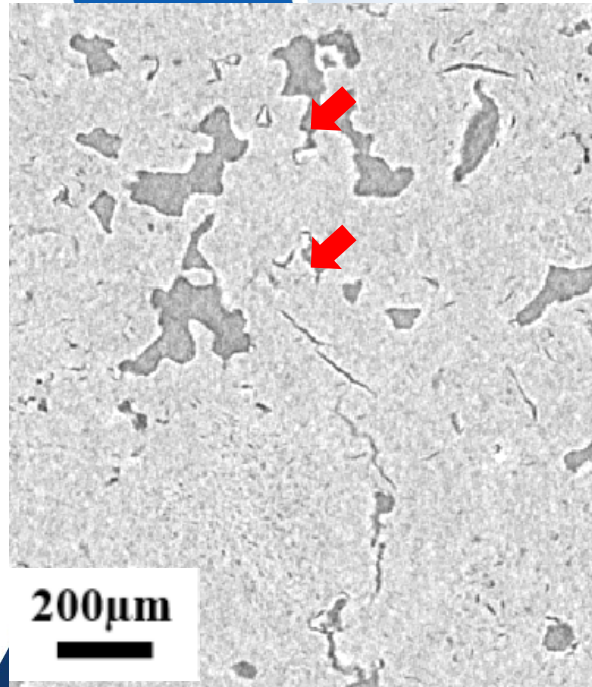
3D volume rendering image of each scan



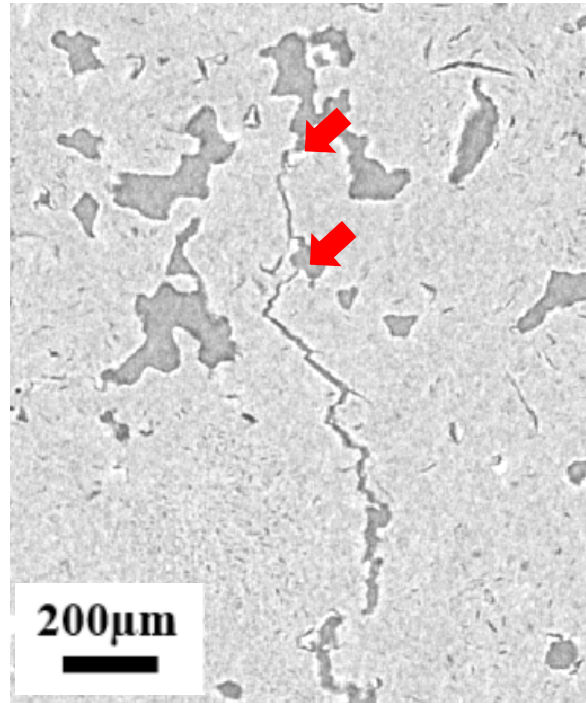
Crack Propagation

- Crack propagated along and deflected by the pre-existed defects
- Crack propagating through the filler particle was also observed

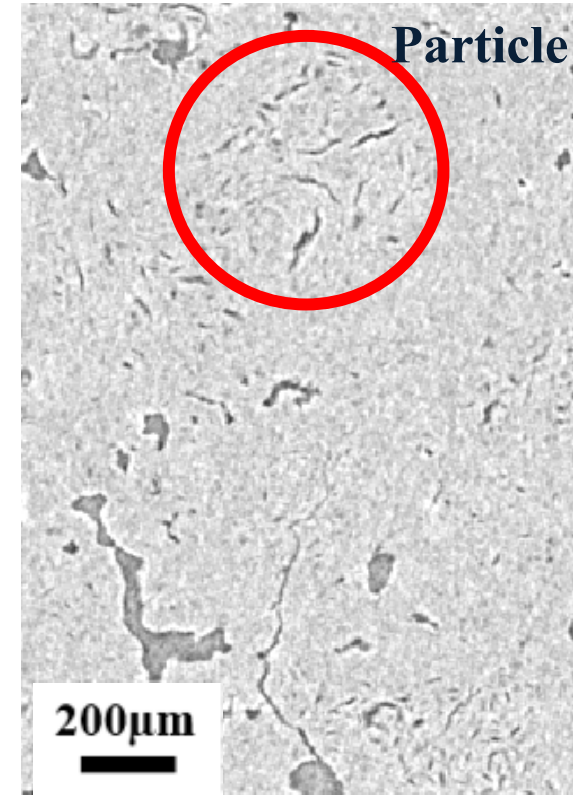
Scan 2



Scan 3



Scan 2



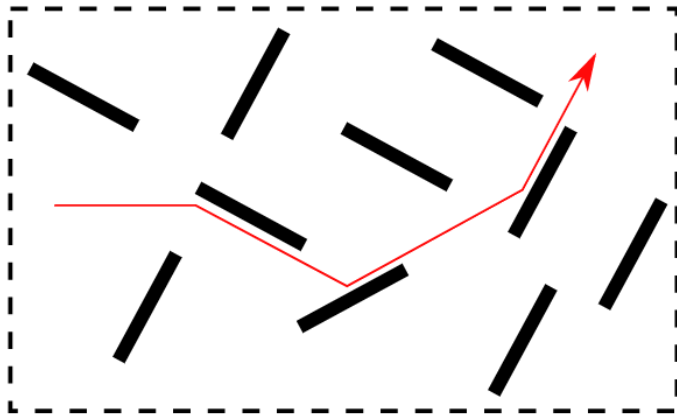
Scan 3



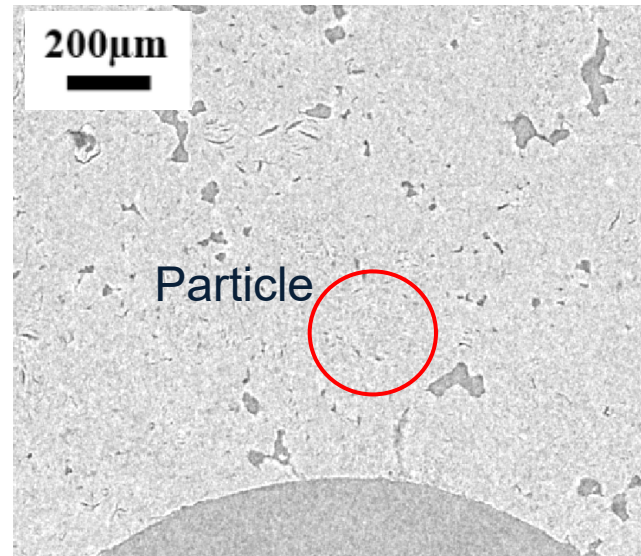
Result: Toughening Mechanism

- **Toughening mechanism:** the process of making a material more resistant to the propagation of cracks.
- **Crack deflection** due to thermal crack, gas entrapment pores, and filler particles.

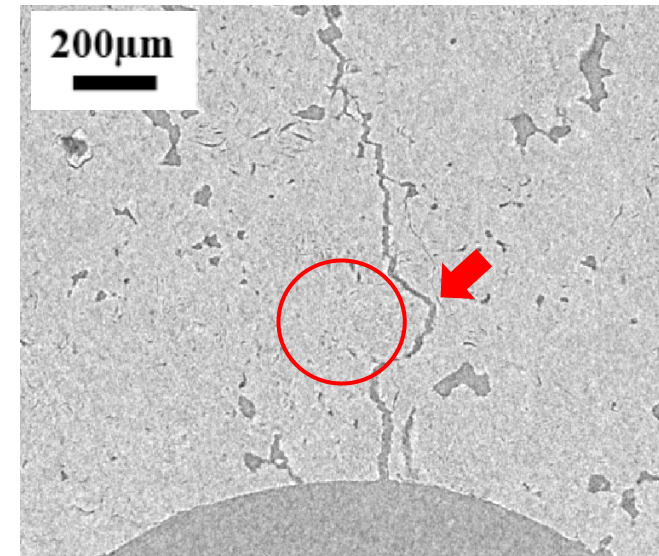
Schematic of crack deflection



0 N



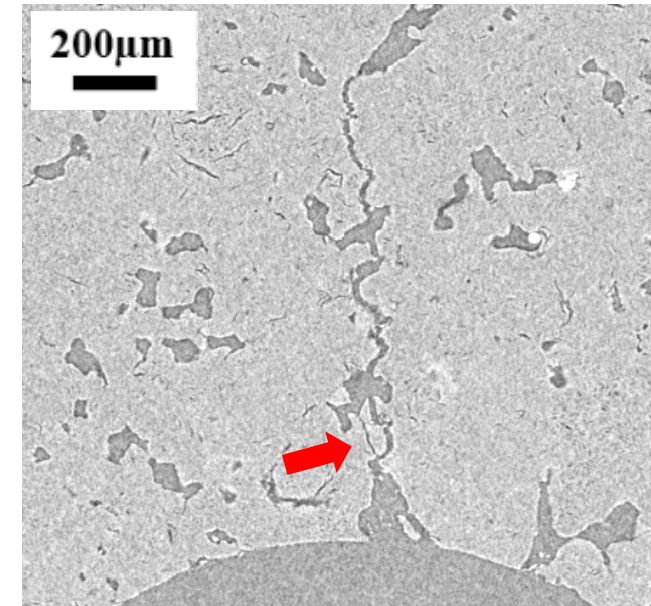
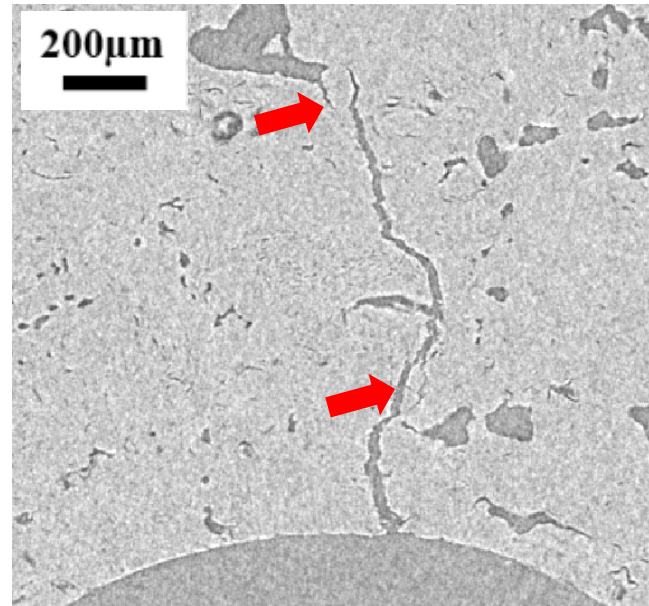
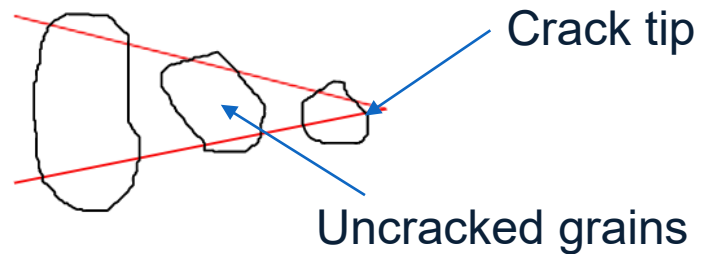
890 N



Result: Toughening Mechanism

Crack bridging by uncracked ligament:

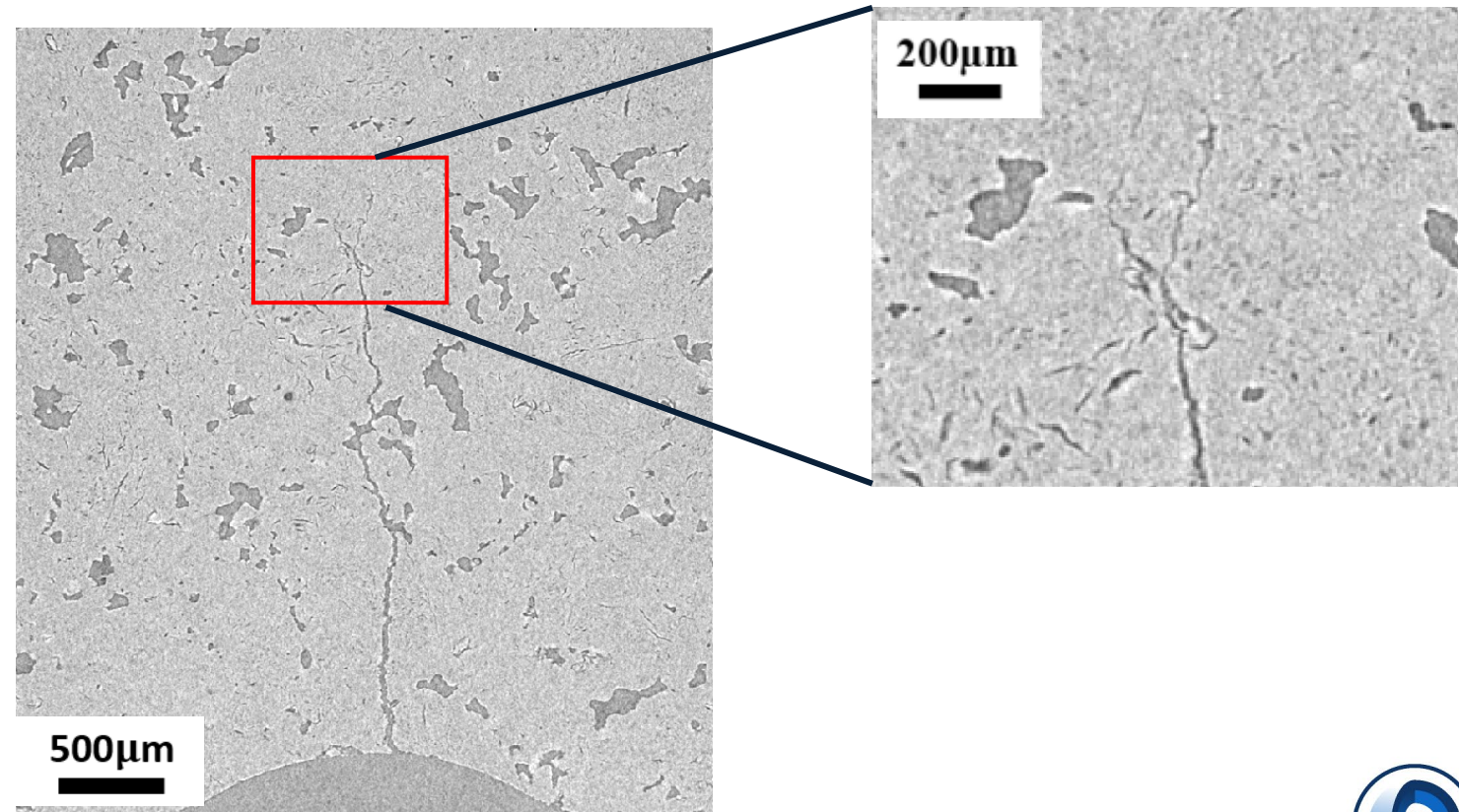
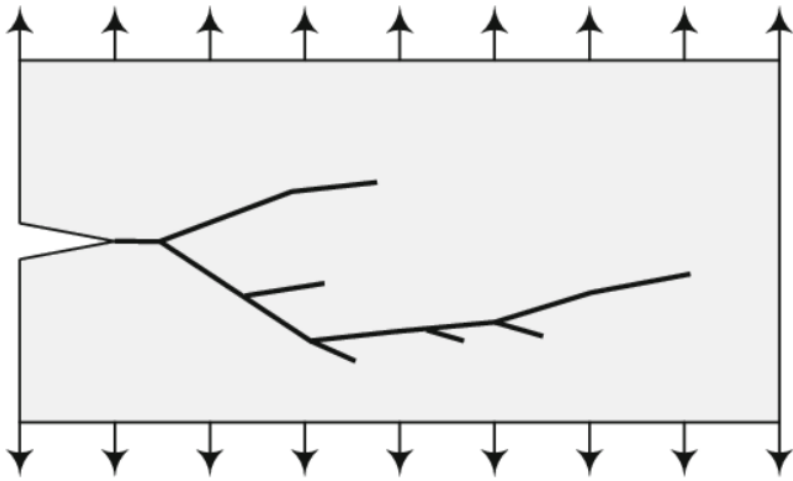
Schematic of crack bridging



Result: Toughening Mechanism

- **Crack tip bifurcation** was also observed but it was not common.

Schematic of crack tip bifurcation



Outline

- Micro-CT split disc fracture test
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- Room temperature annealing of graphite

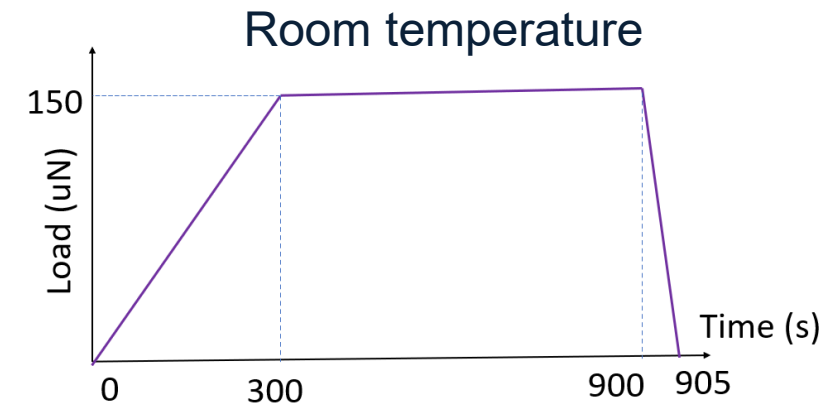
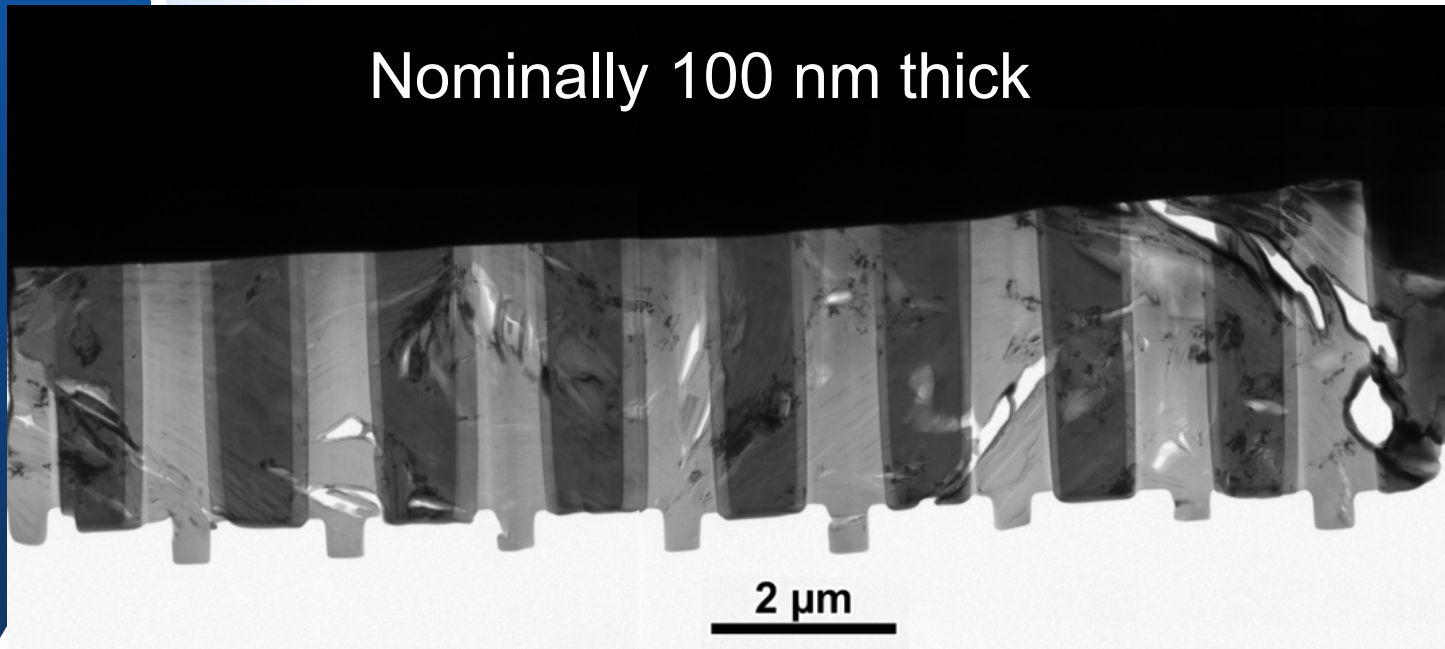


In-situ TEM Creep Testing

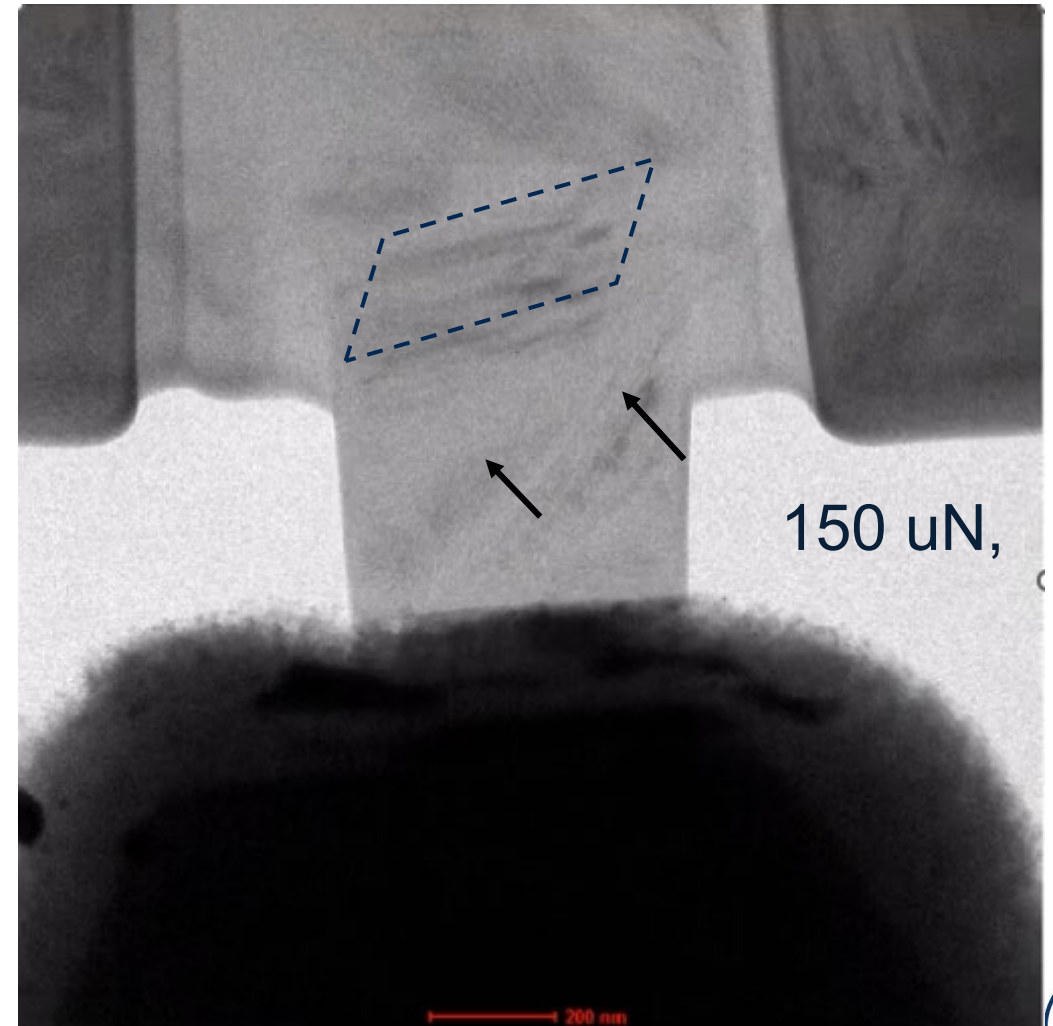
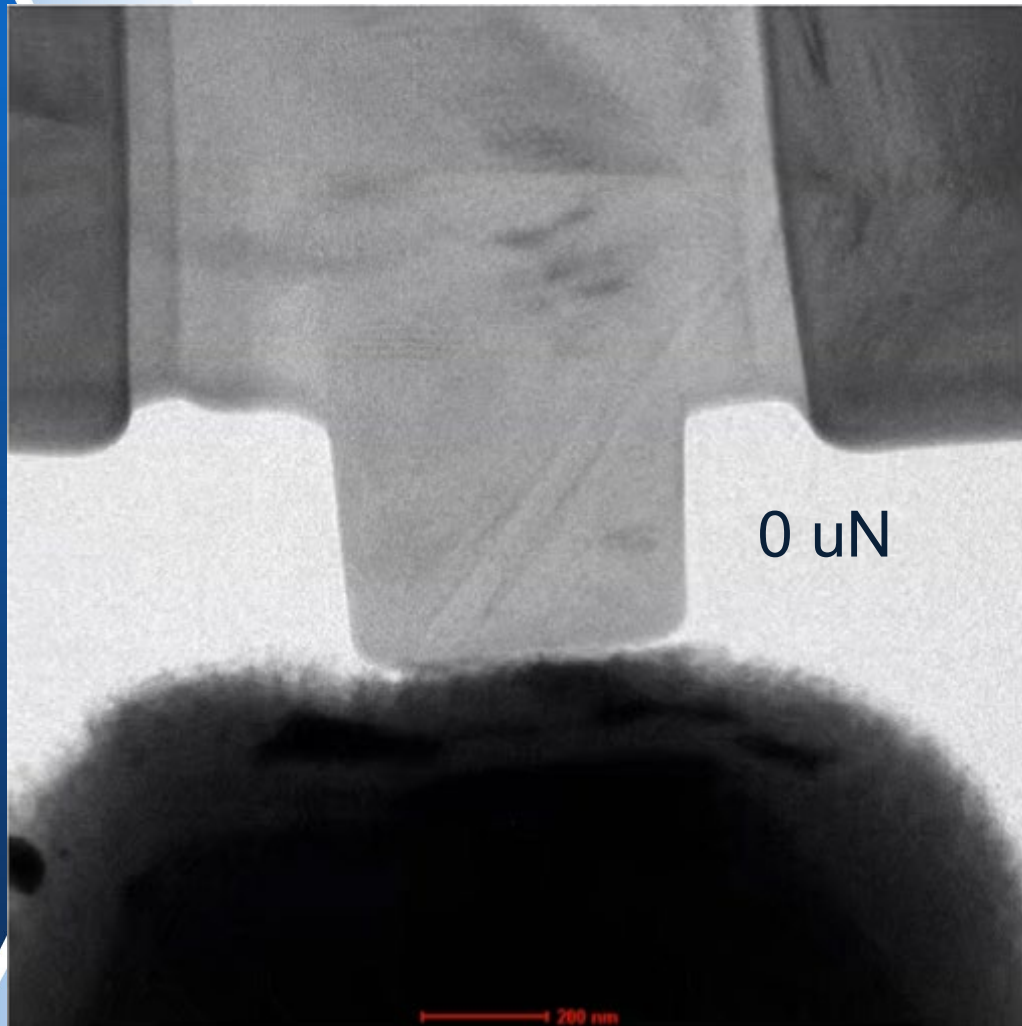
Creep plays the important role of relieving the stresses originating from irradiation at the reactor core.

The fundamental mechanism is often ascribed to basal plane slip, which is not well understood – particularly from other properties (coefficient of thermal expansion, Young's modulus) perspectives.

Nominally 100 nm thick

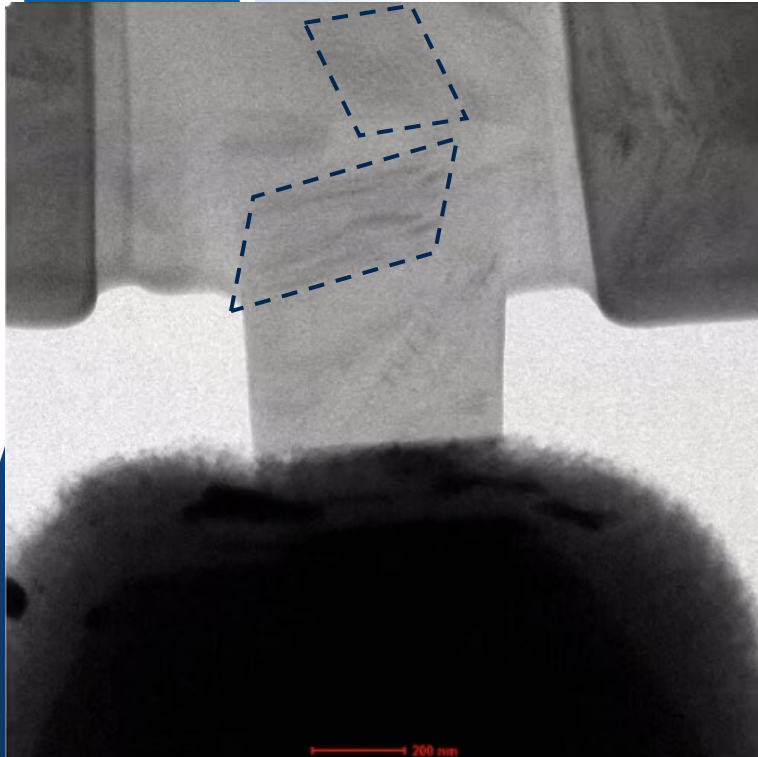


In-situ TEM Creep (Pristine IG110)

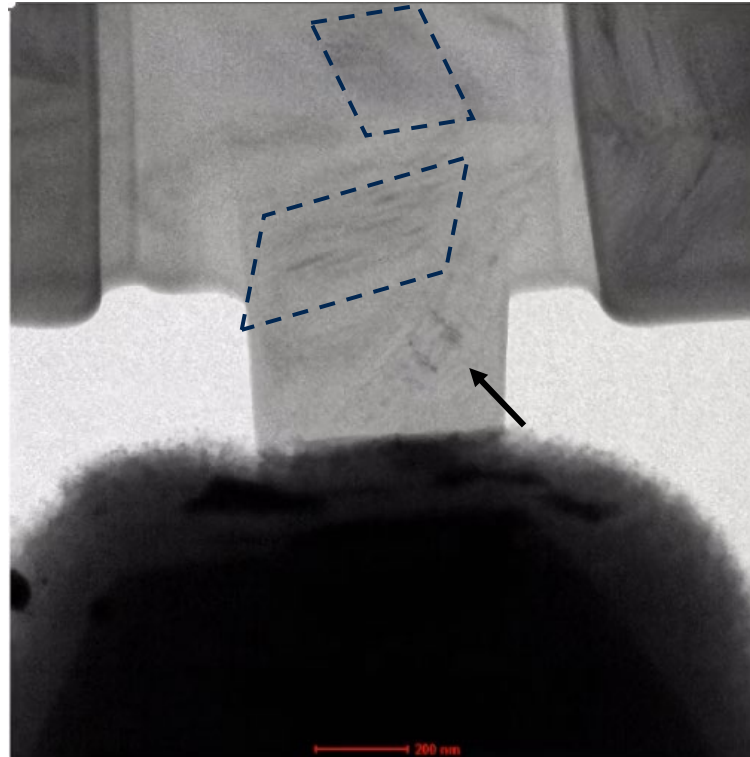


In-situ TEM Creep (Pristine IG110)

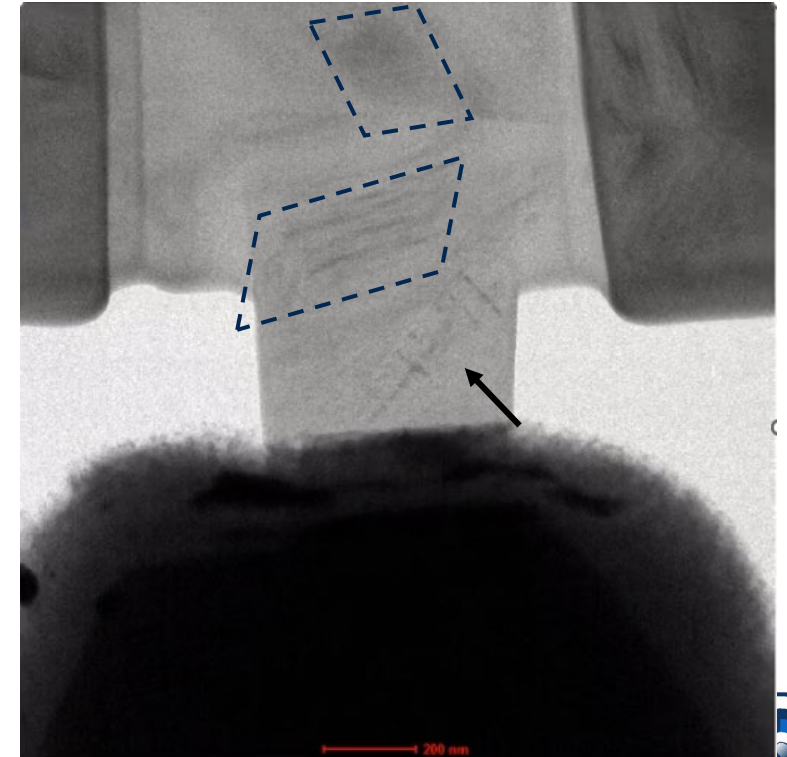
150 μ N, 100 sec



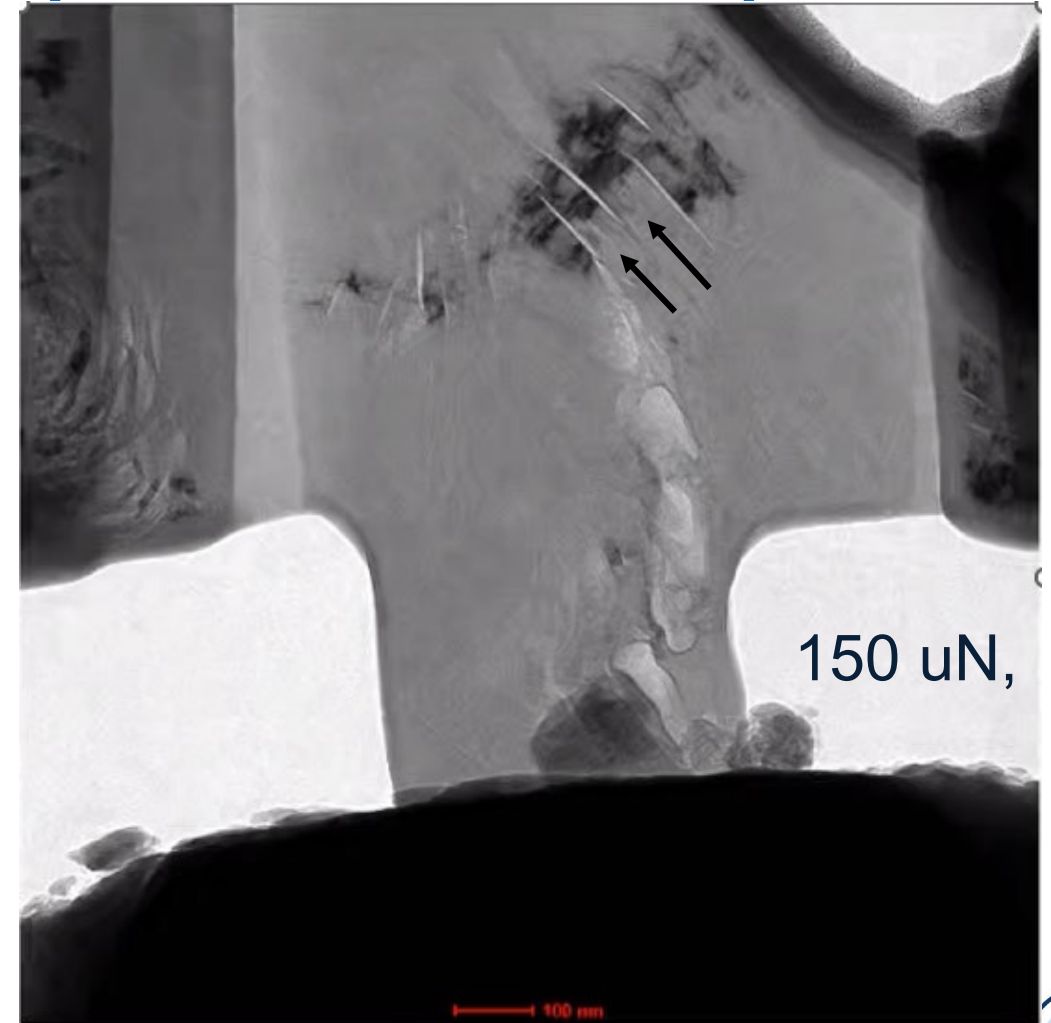
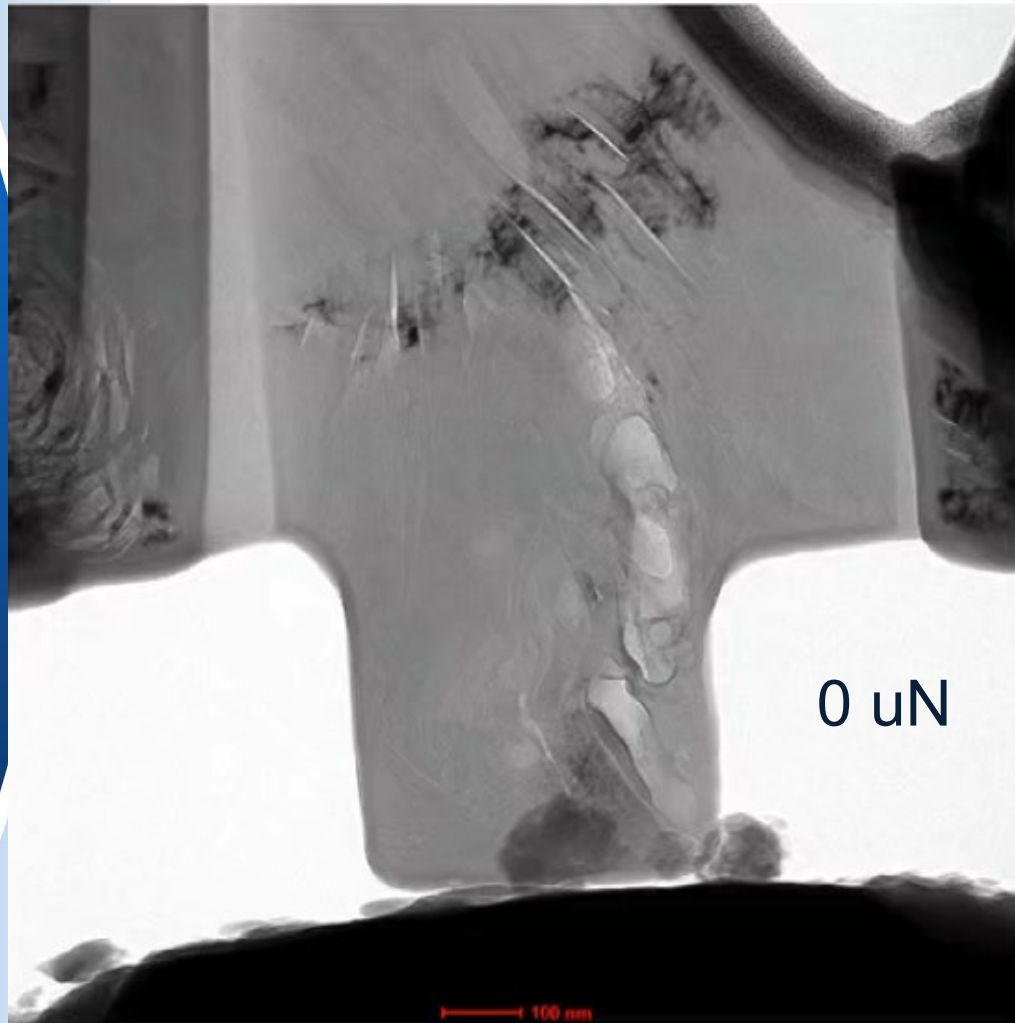
150 μ N, 200 sec



150 μ N, 250 sec

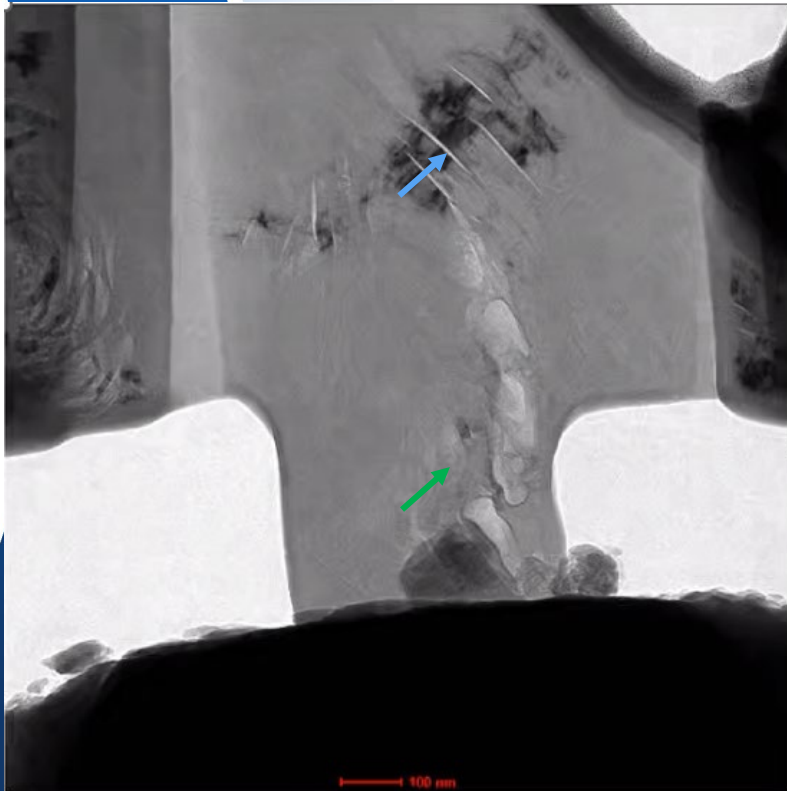


In-situ TEM Creep (Ion Irradiated)

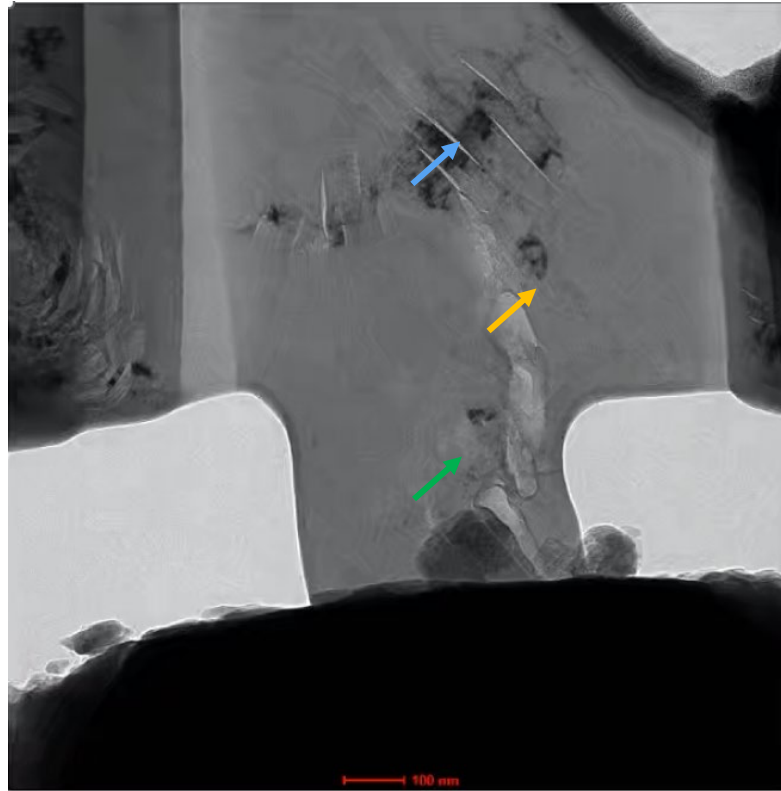


In-situ TEM Creep (Ion Irradiated)

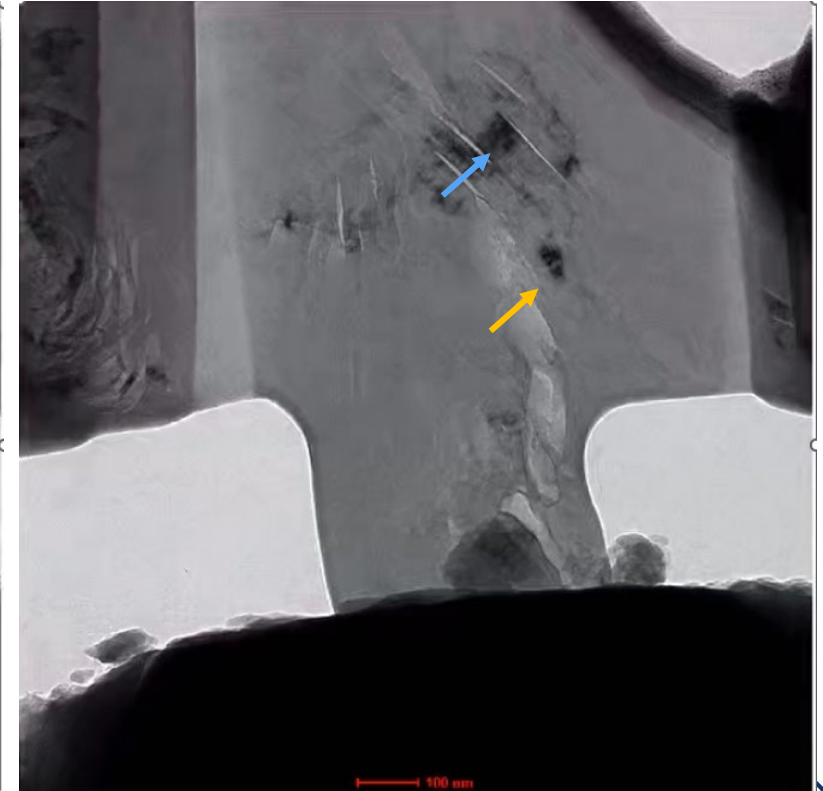
150 uN; 300 sec



150 uN; 600 sec



150 uN; 900 sec



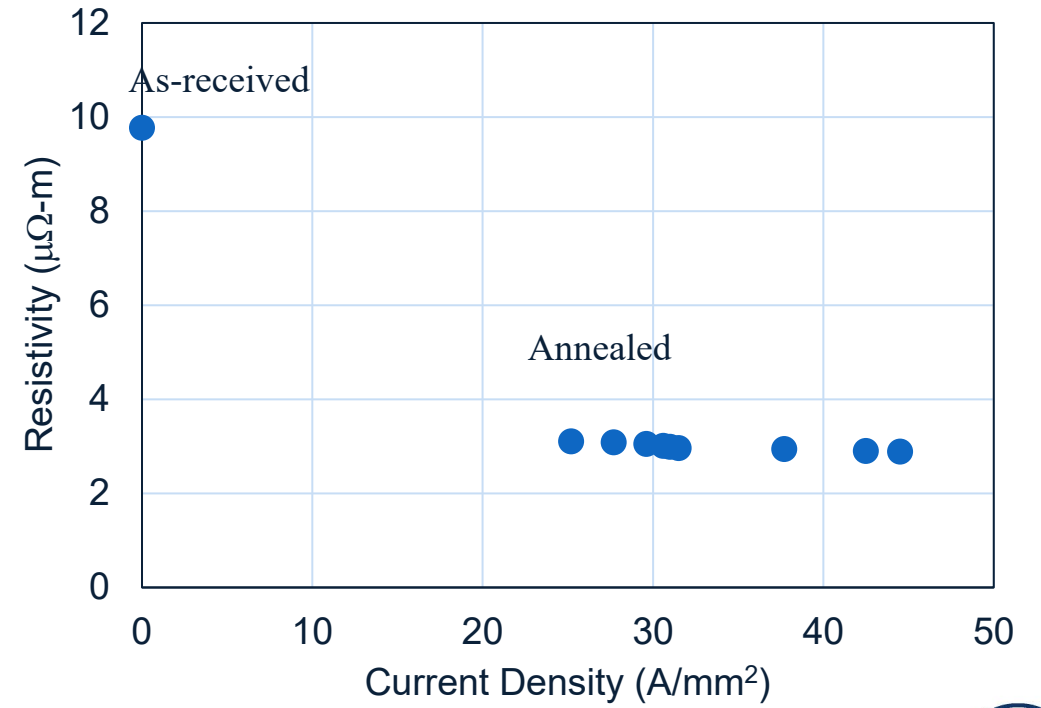
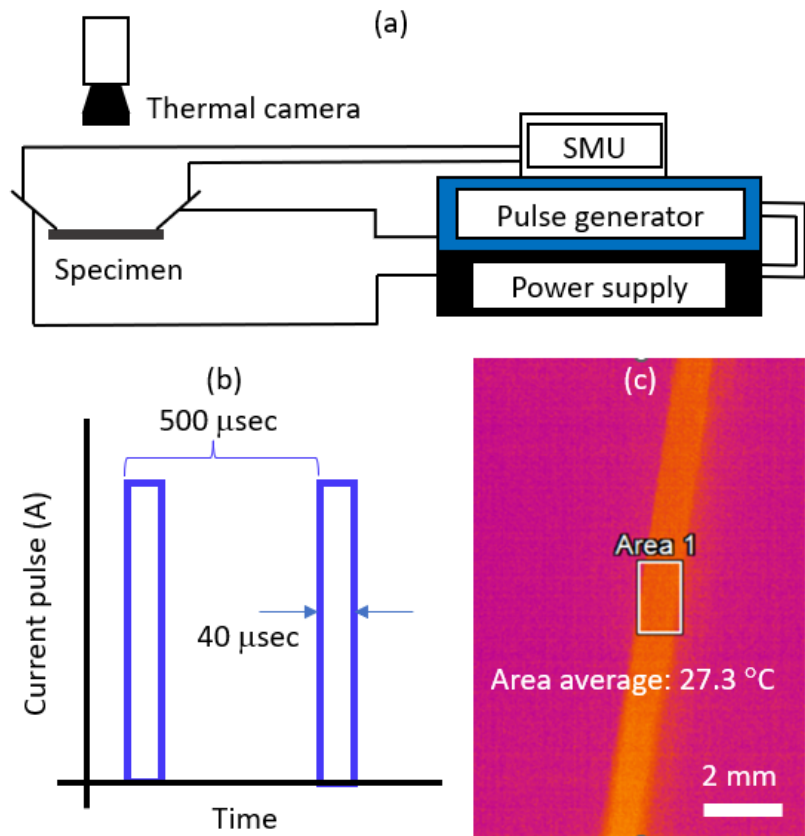
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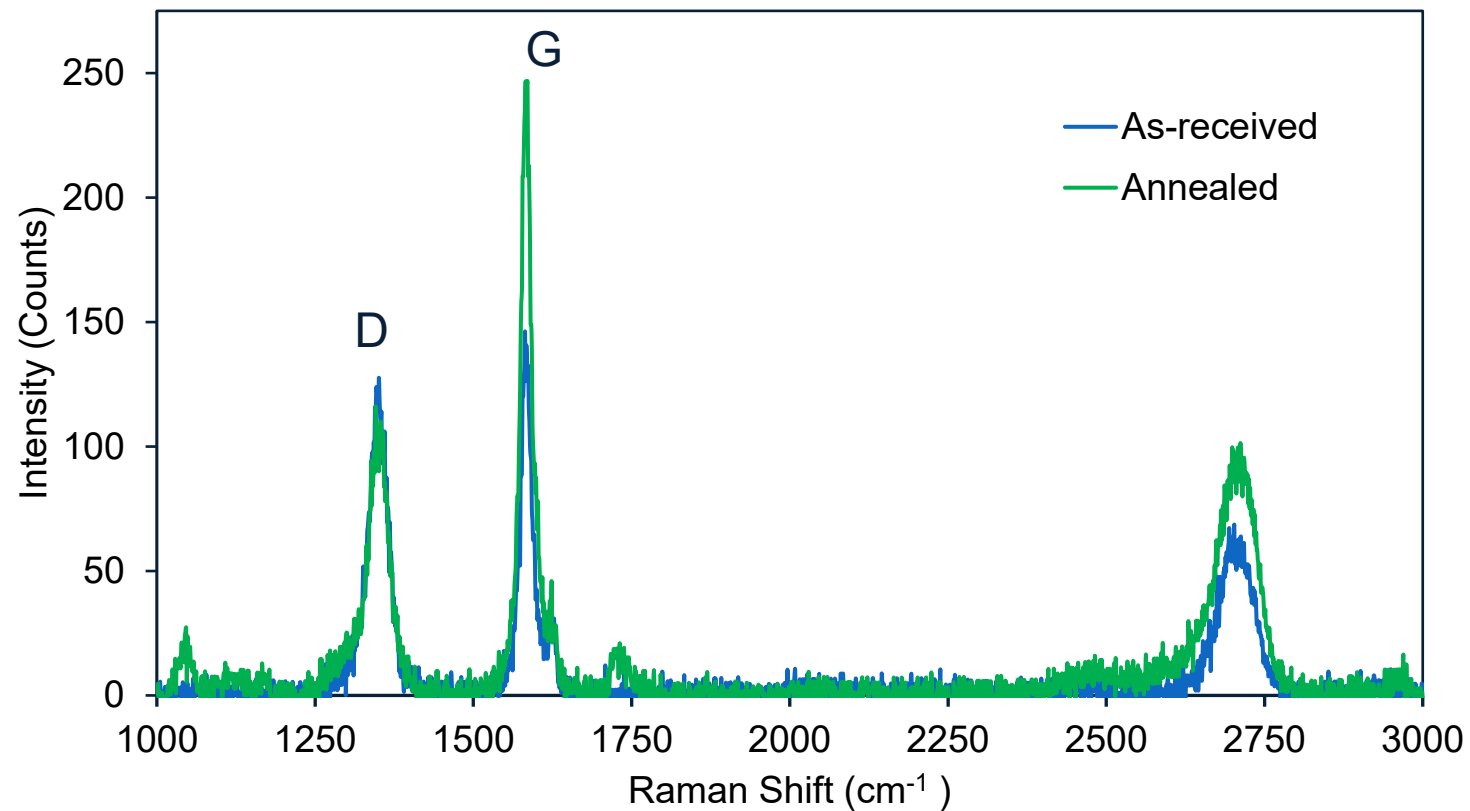
Room Temperature Annealing

Complete annealing is not possible even $> 2000\text{ }^{\circ}\text{C}$.
What if the annealing was not thermal?



Electropulsing Room Temperature Annealing

Raman spectra of the same sample before and after annealing



Future work

- In-situ TEM and SEM mechanical testing on neutron-irradiated nuclear graphite
- Creep studies bulk neutron-irradiated graphite



Acknowledgements



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- TPOC: Dr. William Windes (INL)
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Thank you



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