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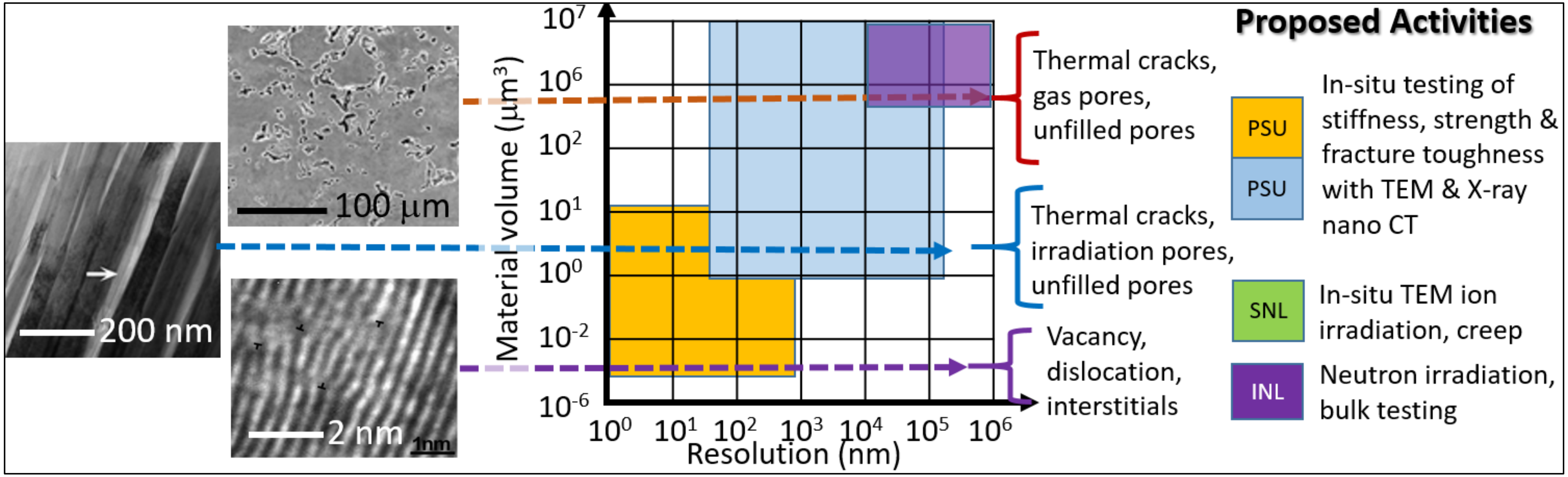
Multi-scale Effects of Irradiation Damage on Nuclear Graphite Properties

DE-NE0009129

U.S. Department of Energy

Research Goals

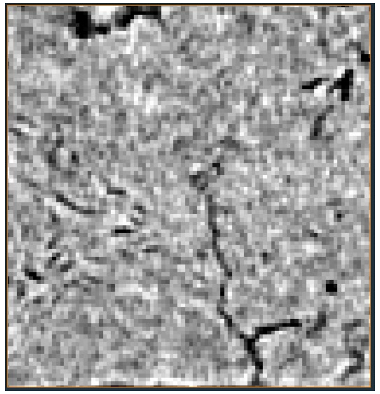


- Multi-scale interaction mechanisms between pre-existing and irradiation defects
- Influence of the constituent (filler, binder, interface), radiation displacement damage and temperature
- Role of stress localization on the above-mentioned mechanisms

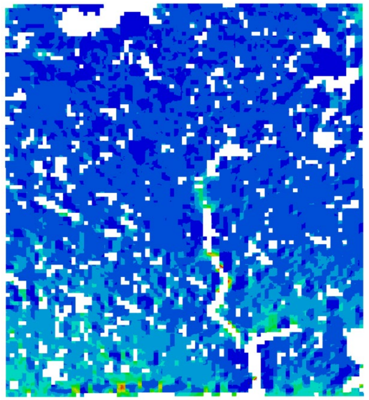
Outline

- In situ X-ray CT fracture testing & finite element analysis
- In situ TEM compression testing after ion irradiation
- High temperature ion irradiation effects

X-CT

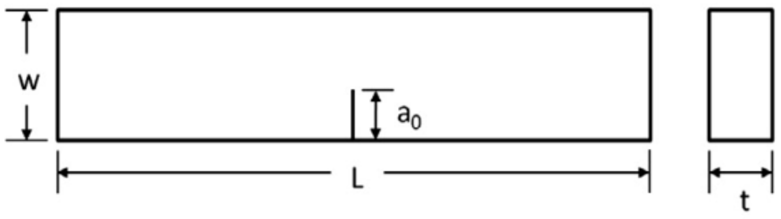


FEM

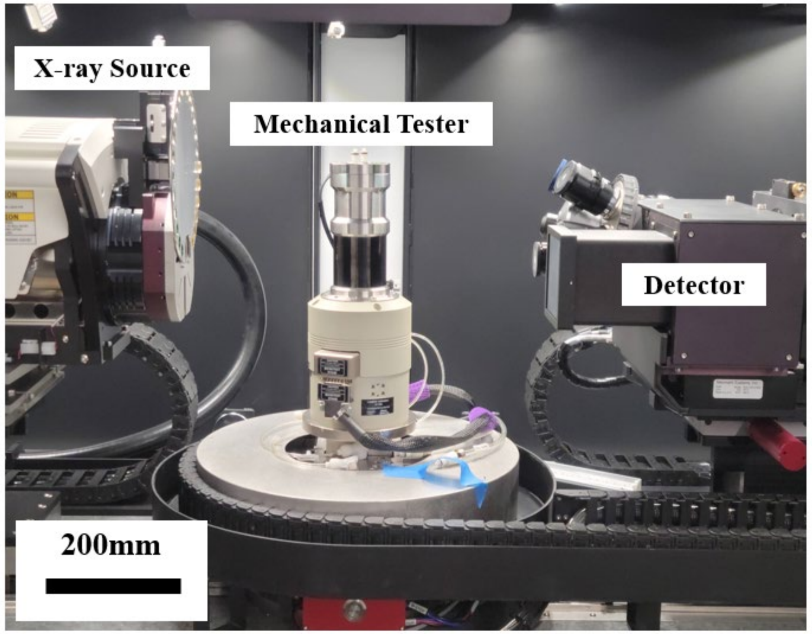


In situ X-ray CT fracture testing on NBG-18

- Measure fracture toughness while observing pre-existing defects, crack nucleation and propagation
- Three-point bending under displacement control (ASTM D7779-20)



Sample	W (mm)	t (mm)	a (mm)	a/W
I	3.980	4.937	1.9465	0.489
II	3.907	4.893	1.879	0.481
III	3.997	4.980	1.958	0.490
IV	3.860	5.020	1.727	0.447



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

In situ X-ray CT fracture testing

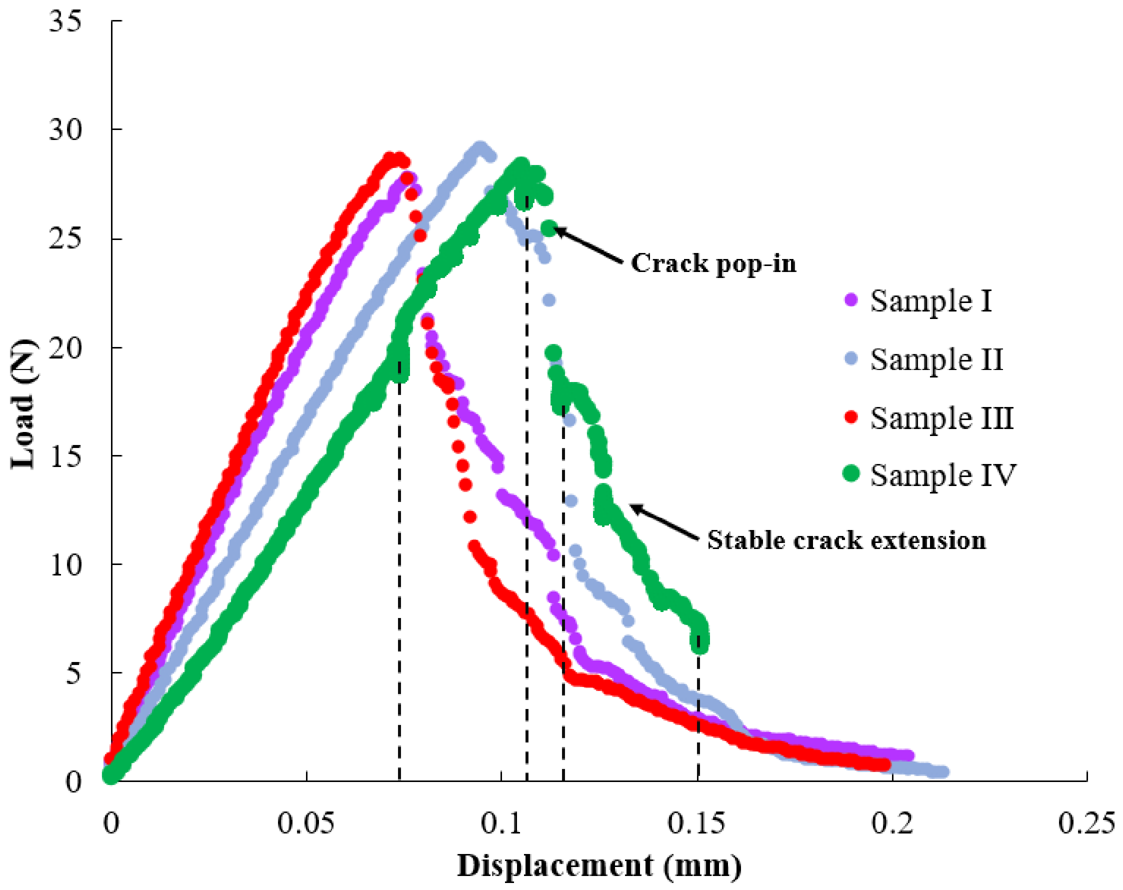
Fracture toughness of the NBG-18 specimens:

$1.17 \pm 0.06\ \mathrm{MPa}\sqrt{\mathrm{m}}$

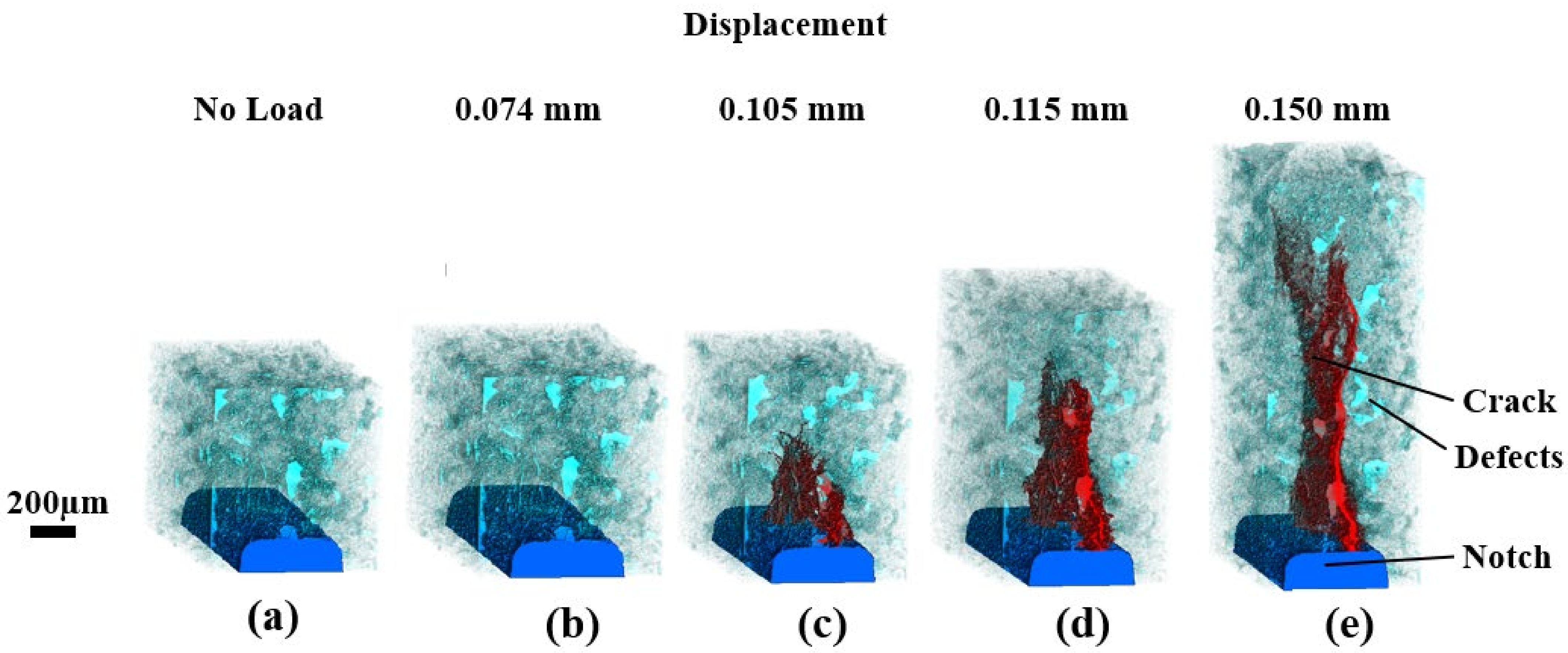
P: applied force
S: loading span
a: crack length
B and W : width and depth respectively
A_0 to A_5 : geometry of the specimen.

$$K_I = g\left[\frac{PS10^{-6}}{BW^{3/2}}\right]\left[\frac{3[a/W]^{3/2}}{2[1-a/W]^{3/2}}\right]$$

$$g = A_0 + A_1(a/W) + A_2(a/W)^2 + A_3(a/W)^3 + A_4(a/W)^4 + A_5(a/W)^5$$



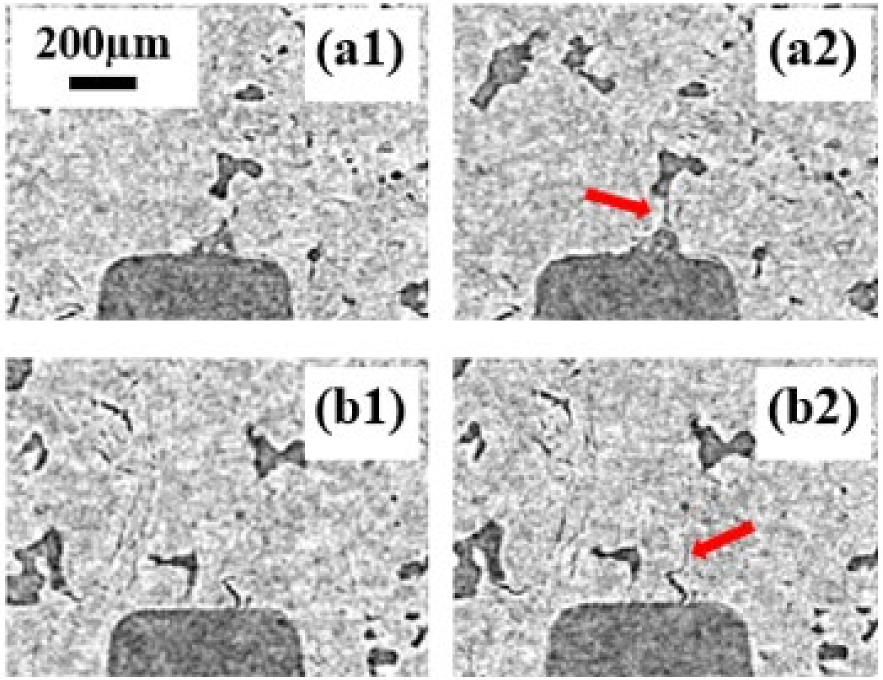
3D volume rendering



Crack nucleation

No Load

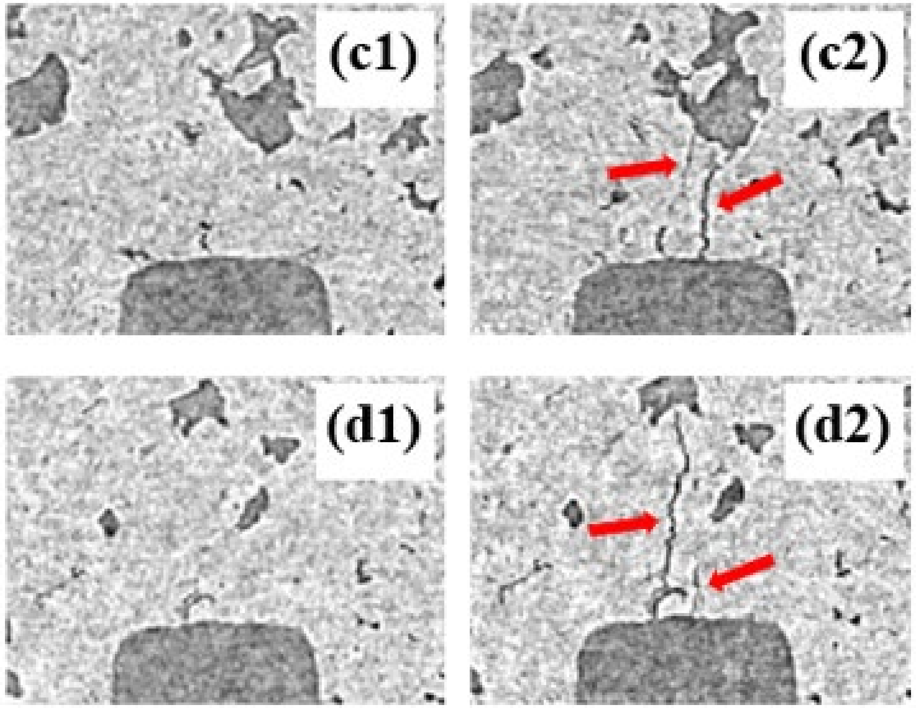
Displacement: 0.105 mm



Primary crack

No Load

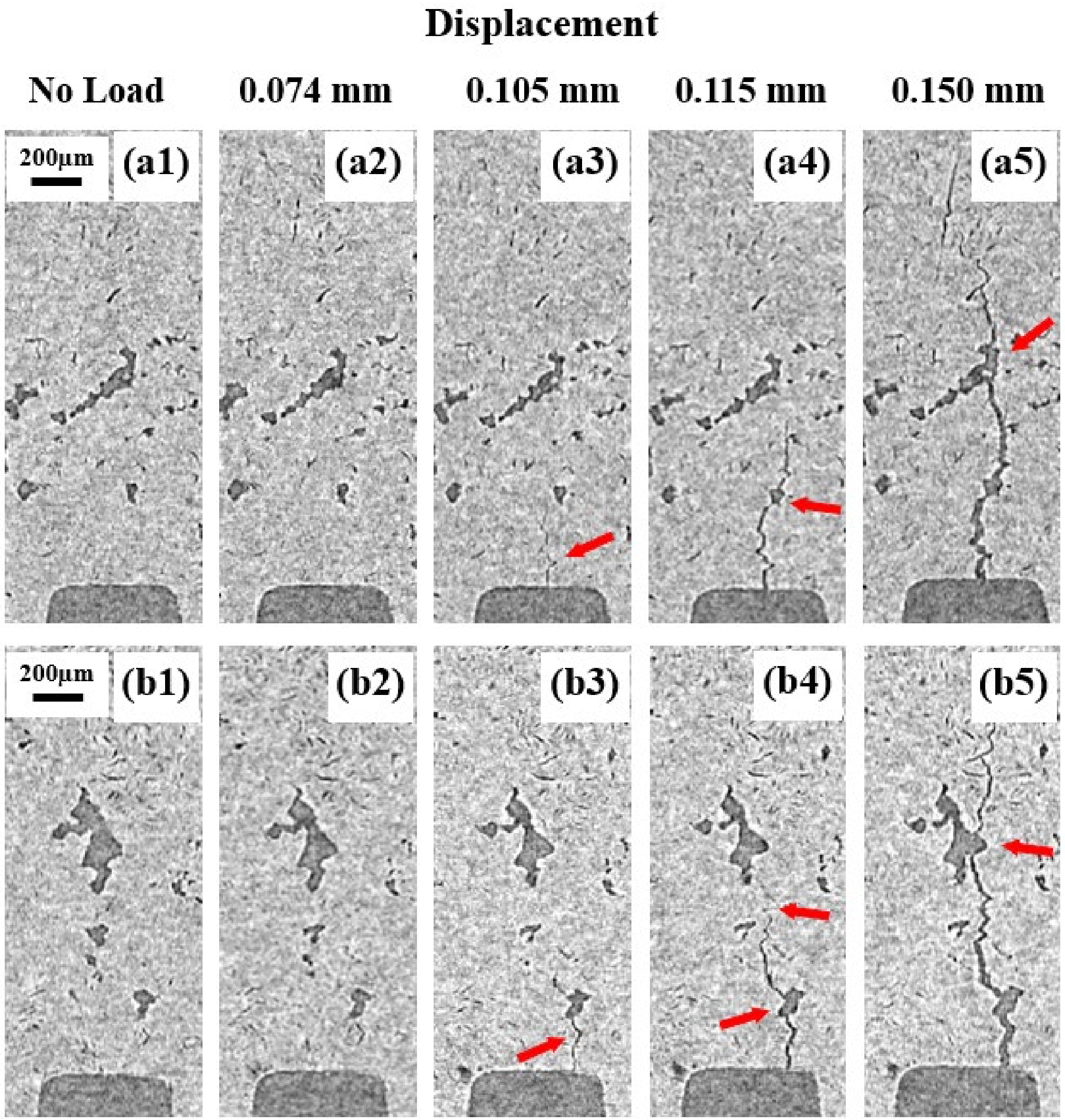
Displacement: 0.105 mm



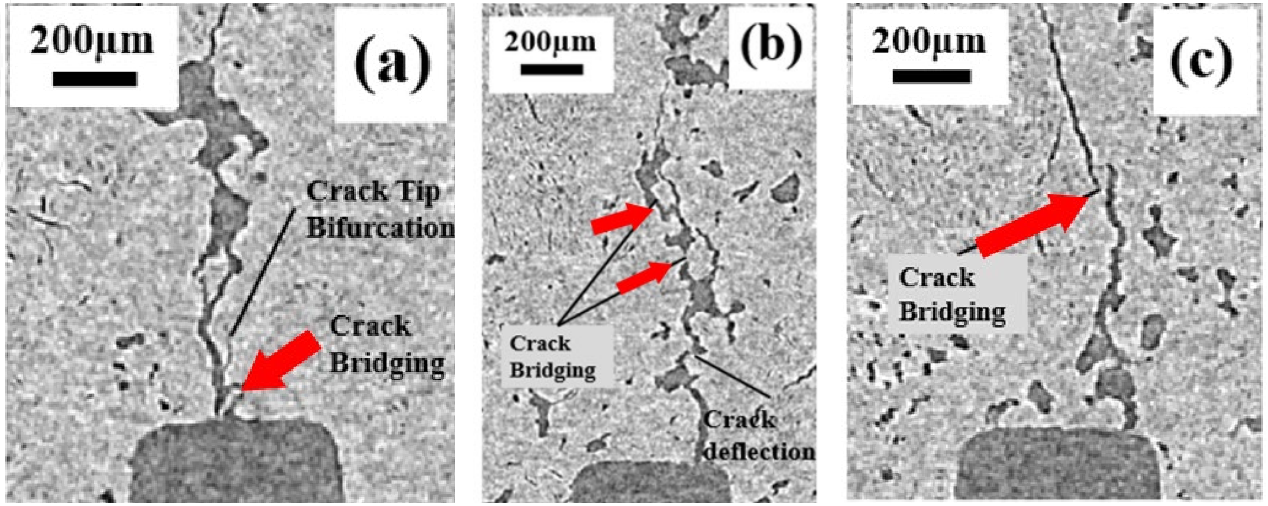
Secondary crack

Crack propagation

Crack propagated along and deflected by the thermal cracks (a3), gas entrapment pores (a4 and b3), and the unfilled voids (a5 and b5).



Toughening mechanisms

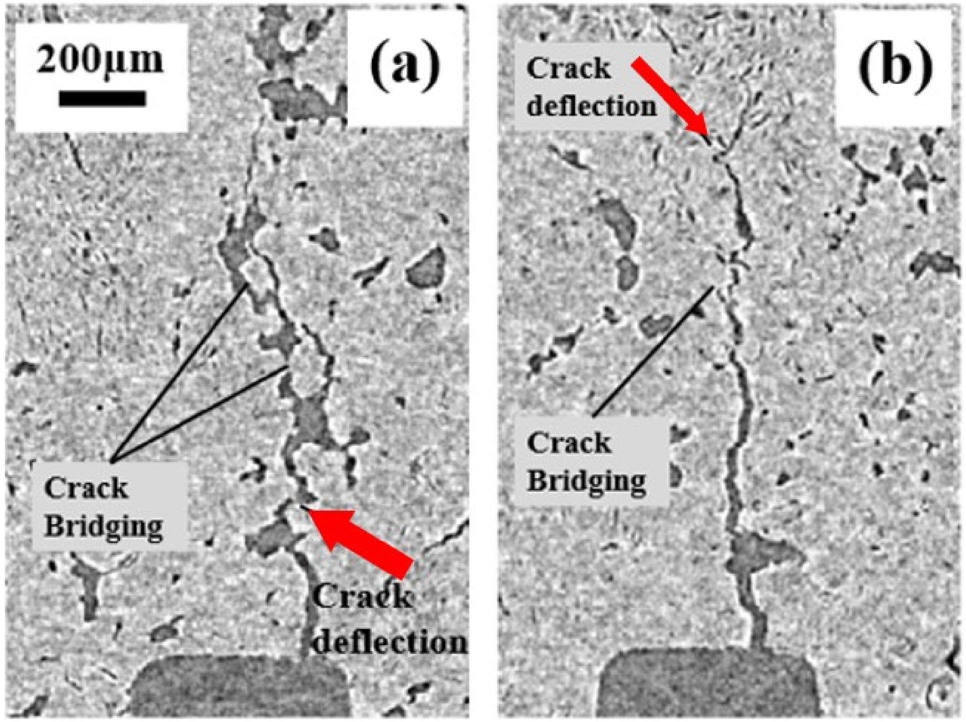


Crack bridging by uncracked ligament: (a)in the filler, (b)in the binder, (c) at the filler-binder boundary

Journal of MATERIALS RESEARCH
Article
DOI:10.1557/s43578-023-00929-7

An investigation of fracture behaviors of NBG-18 nuclear graphite using in situ mechanical testing coupled with micro-CT

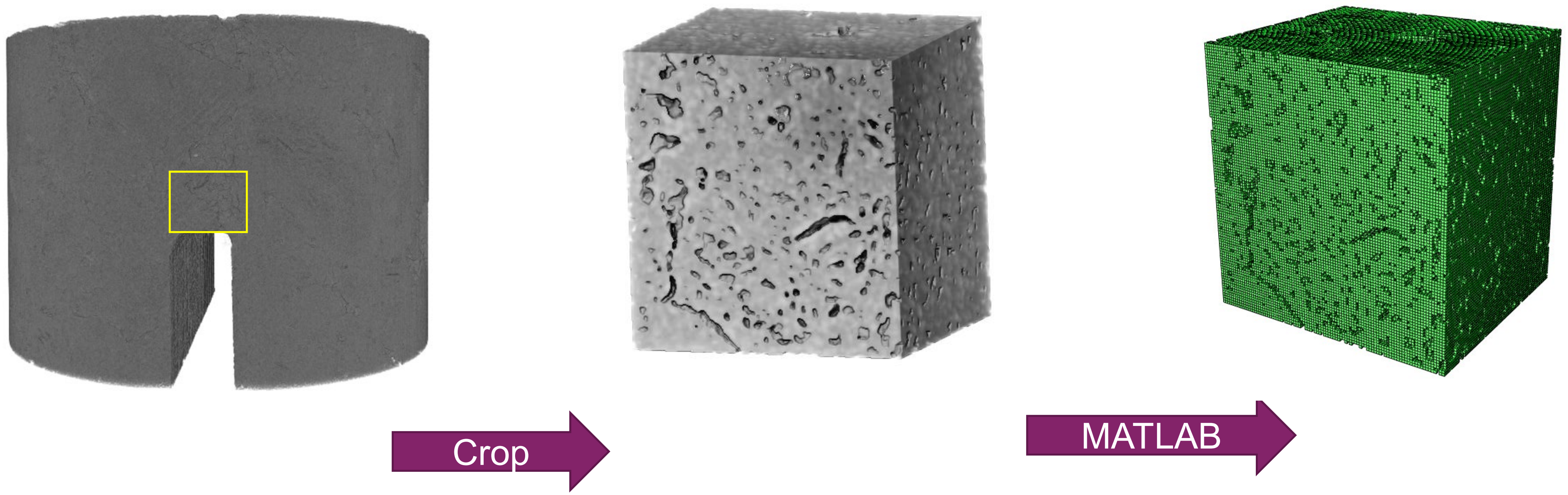
Gongyuan Liu[1], Yichun Tang[1], Khalid Hattar[2,3], Yuzhou Wang[4], William Windes[4], Aman Haque[1], Jing Du[1,a)]



Crack deflection due to thermal crack and gas entrapment pores

Finite element analysis

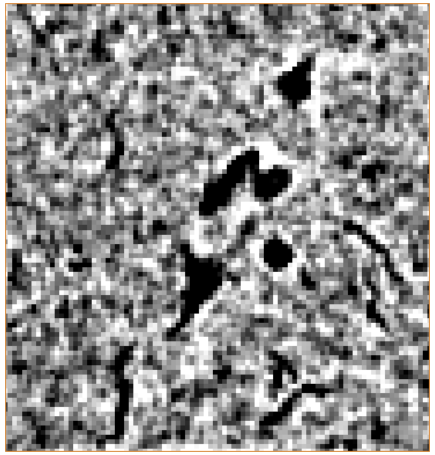
- Voxel-based method to mesh the complex microstructures of nuclear graphite.



Gray-scale based model

- Knudsen’s expression used to approximate constituents

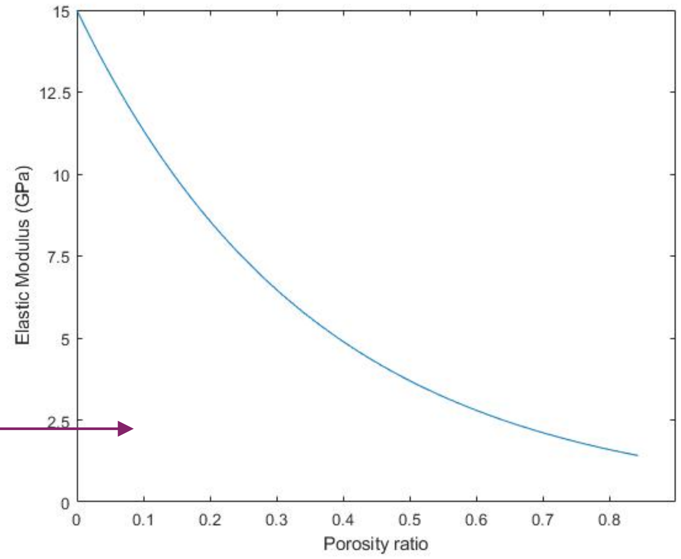
CT data



256 element sets created based on the grayscale intensity (0-255)

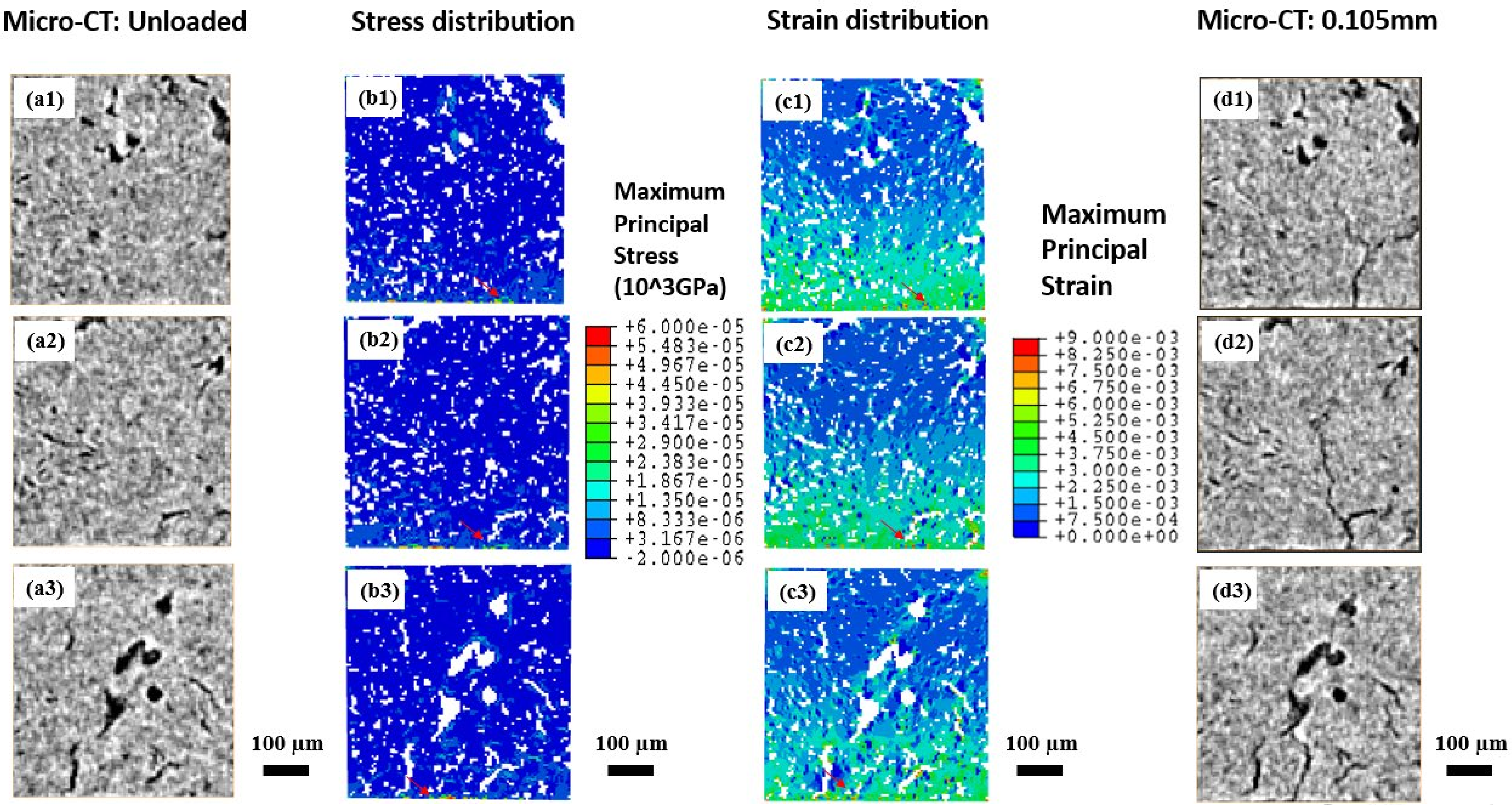
Below 40 → Considered to be visible pores → 0.5GPa

Above 40 → Porosity ratio at each voxel →

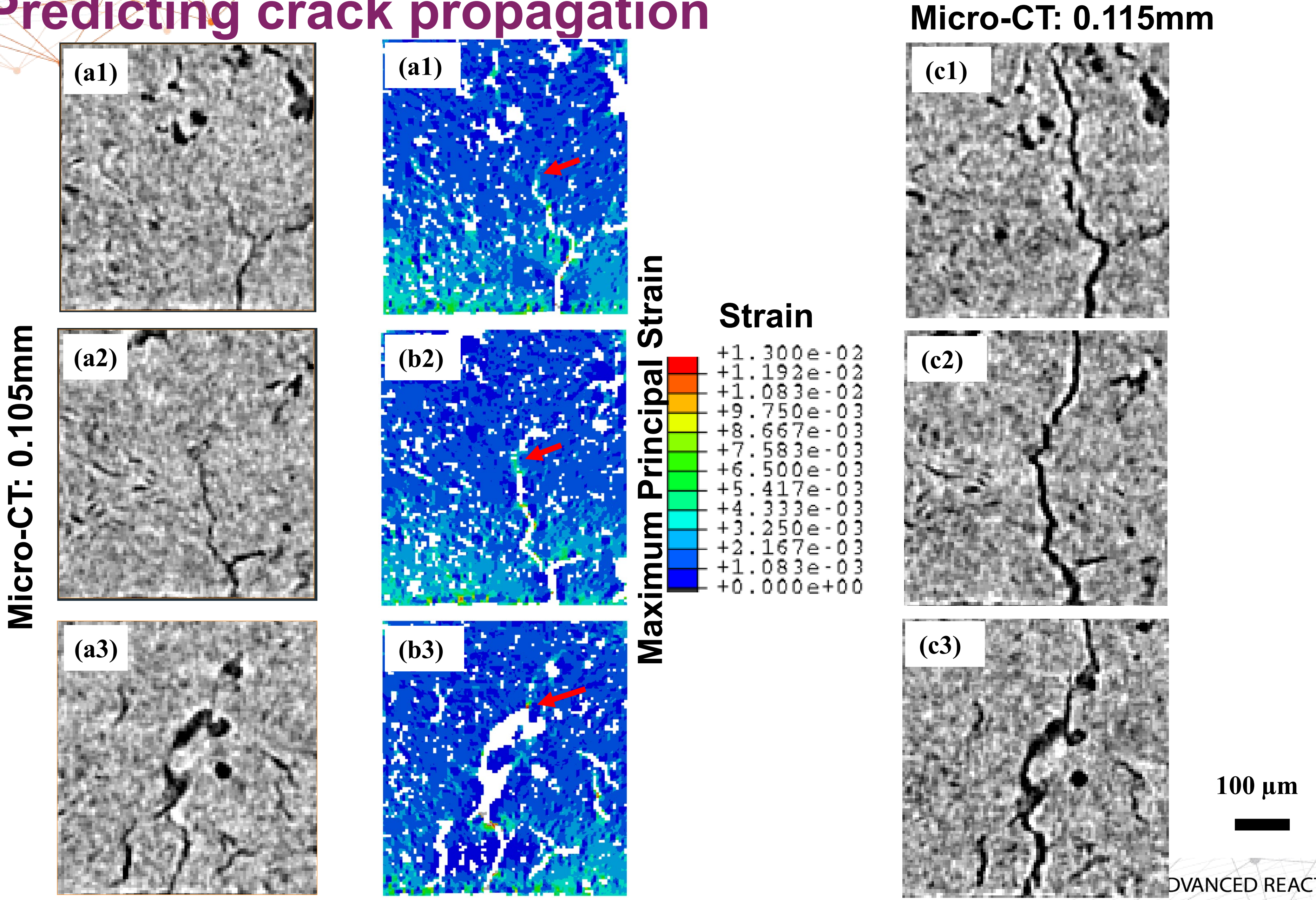


Predicting crack nucleation

- Local stress concentrations not enough to predict accurately



Predicting crack propagation



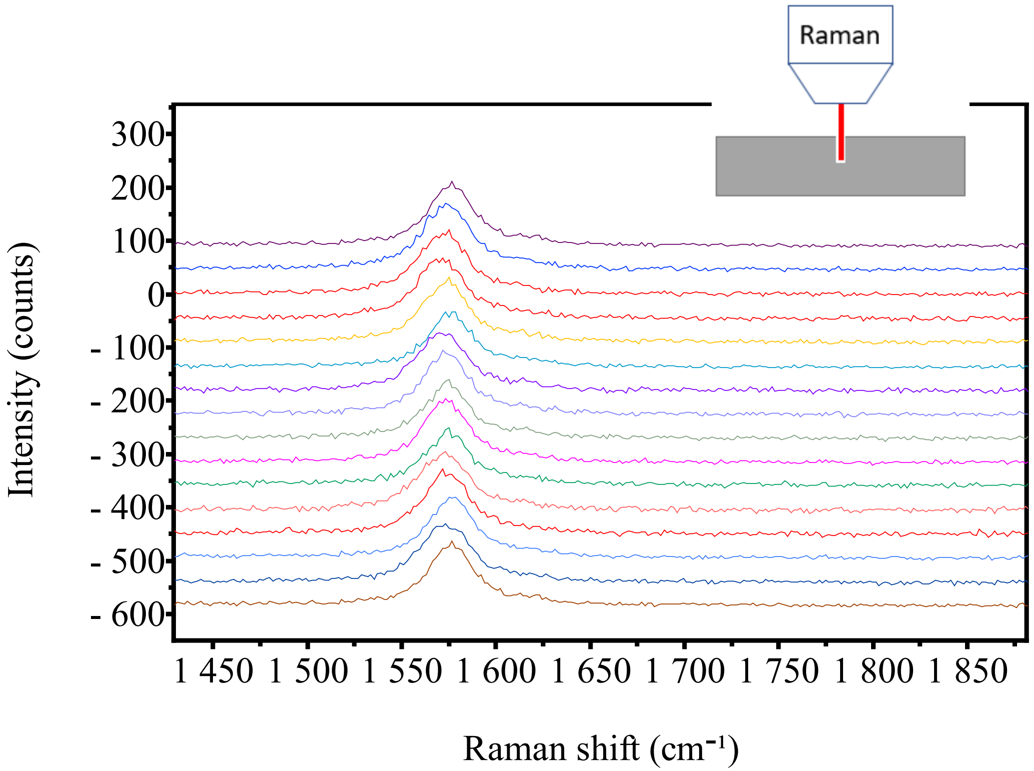
Discussion

- FEA model can easily predict crack propagation – but not its nucleation site. Can residual stress due to machining be the clue?

- G peak shift ranges from 1571.54 cm^{-1} to 1576.43 cm^{-1}

$$\Delta\varpi_G = -\frac{5}{\varpi_{G0}}\sigma$$

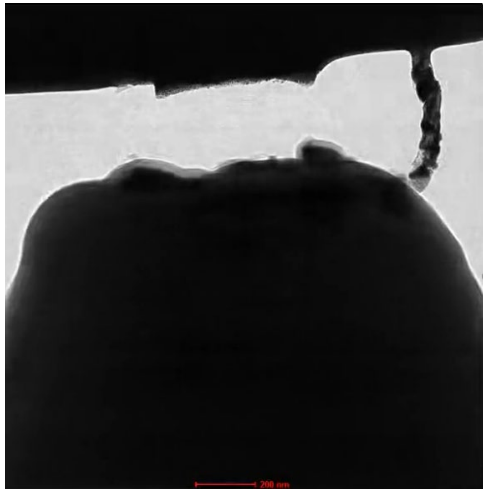
- Localized residual stress: 1.1 GPa to 2.6 GPa (Tension).



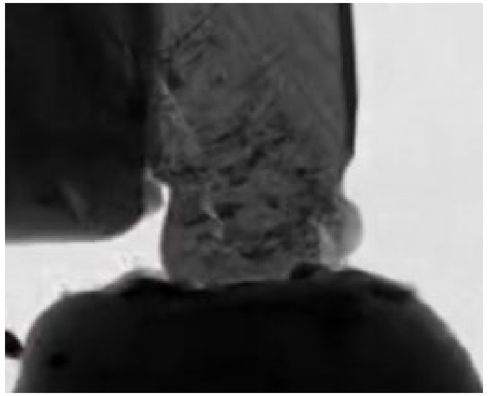
Outline

- In situ X-ray CT fracture testing & finite element analysis
- In situ TEM compression testing after ion irradiation
- High temperature ion irradiation effects

Pristine



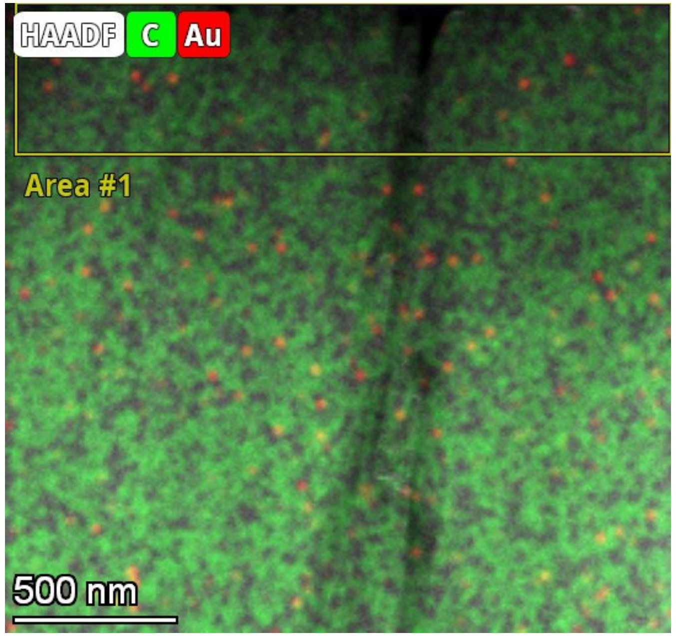
Irradiated



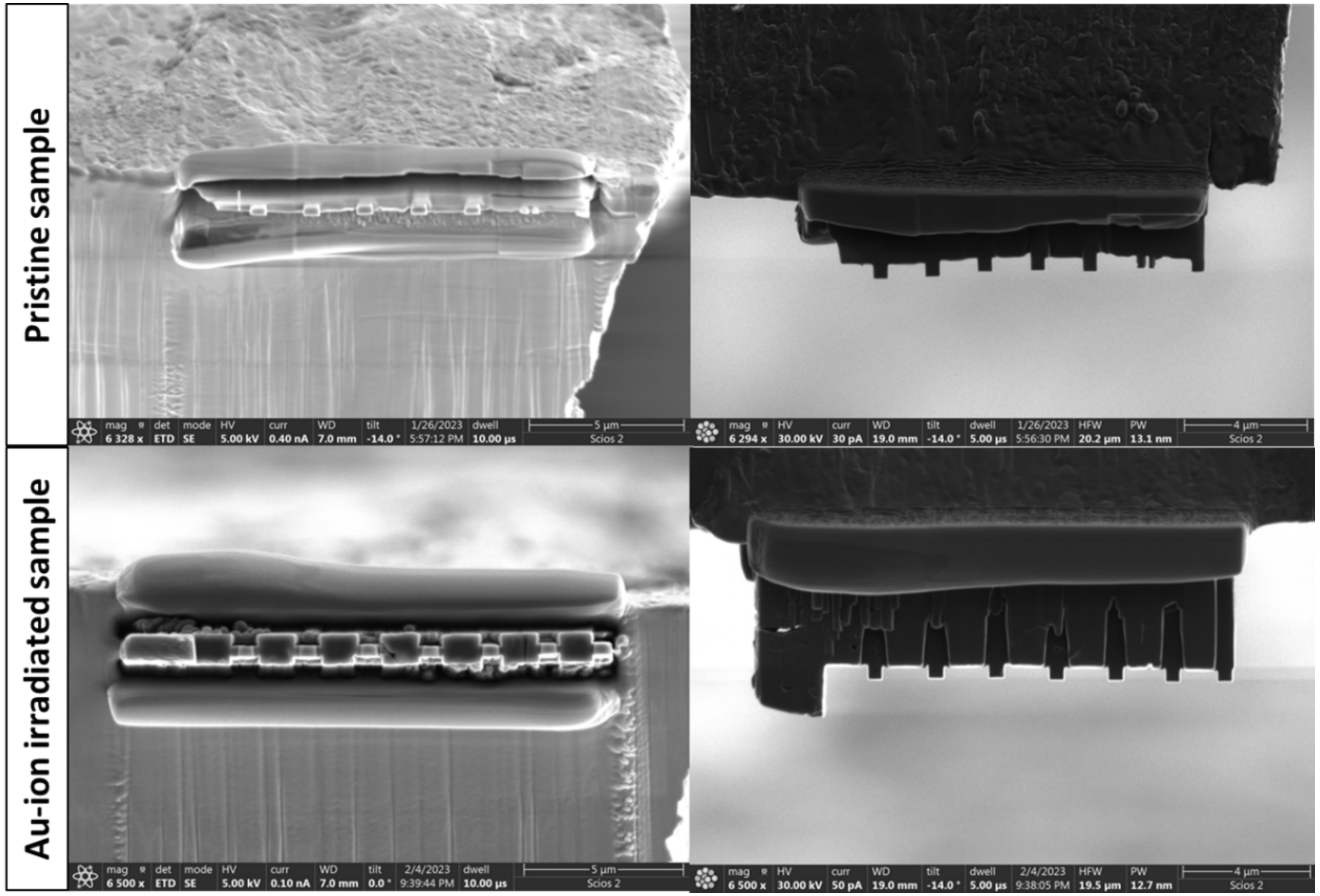
In situ TEM compression on IG-110

- 2.8 MeV Au^{+2} (fluence of 4.378×10^{14} cm^{-2})

- Cuboid (~200 nm) micro pillars milled with FIB



TEM EDS



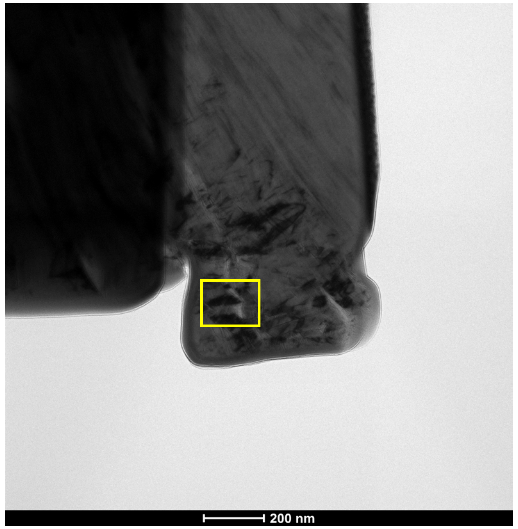
Pristine IG-110 micro pillar

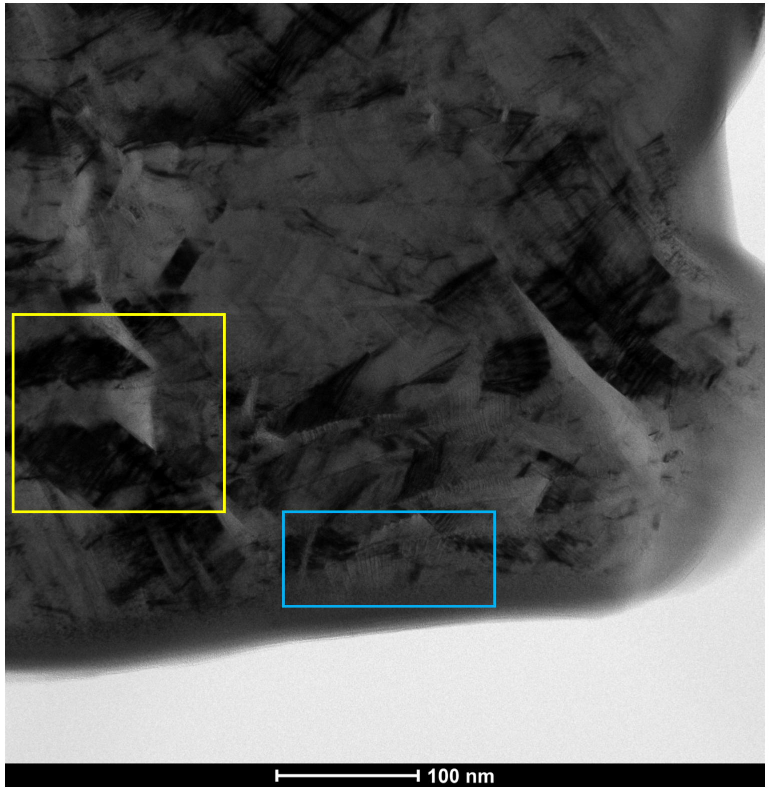
- FEI Titan ETEM G2 at 300 kV, using the Hysitron PI 95 in displacement-controlled mode

Ion irradiated micro pillar

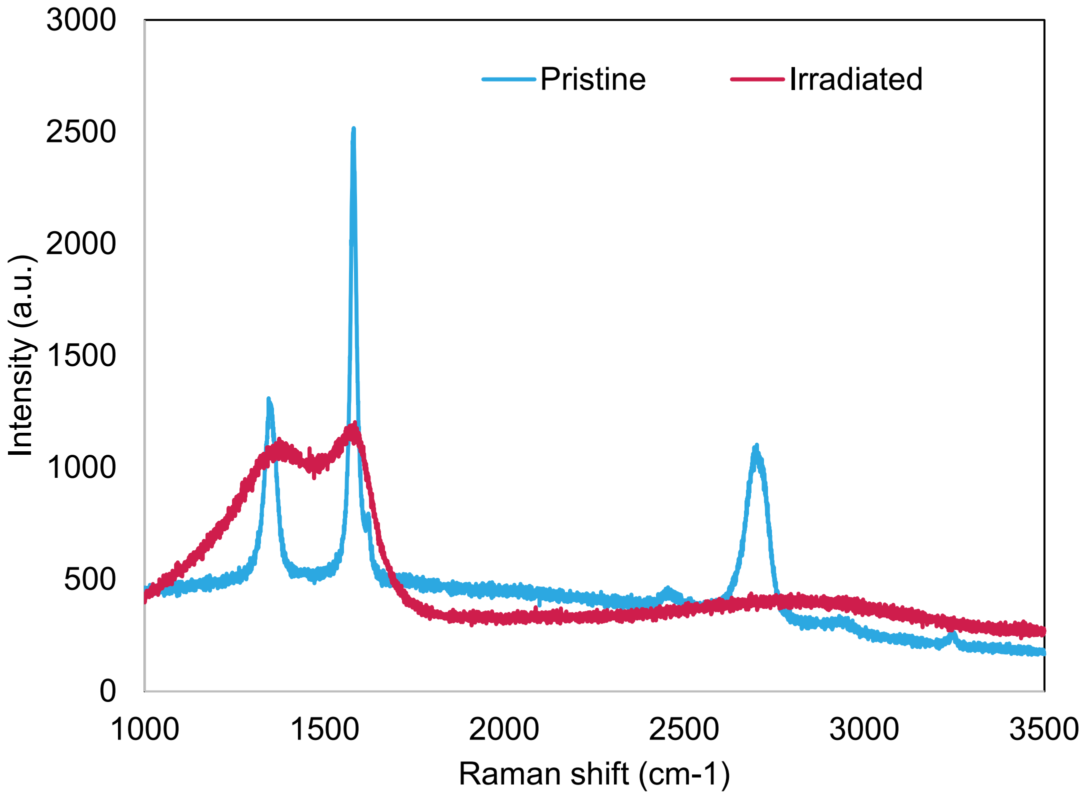
- FEI Titan ETEM G2 at 300 kV, using the Hysitron PI-95 in displacement controlled mode

Ion irradiated micro pillar



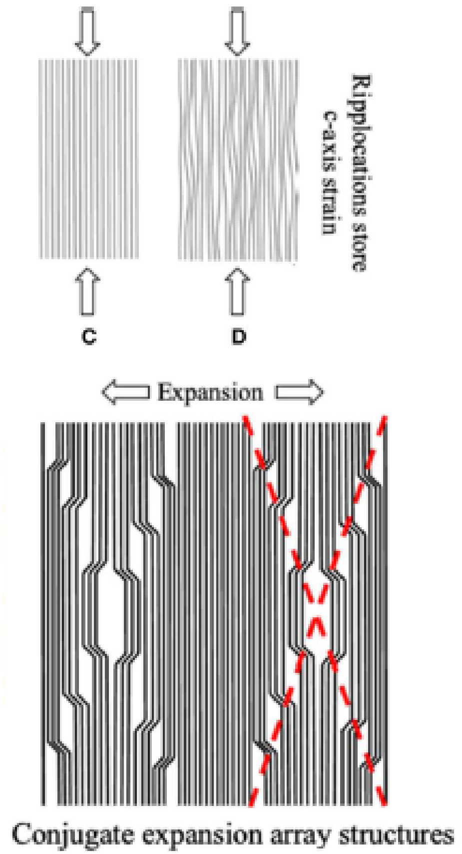


Raman spectra for IG-110

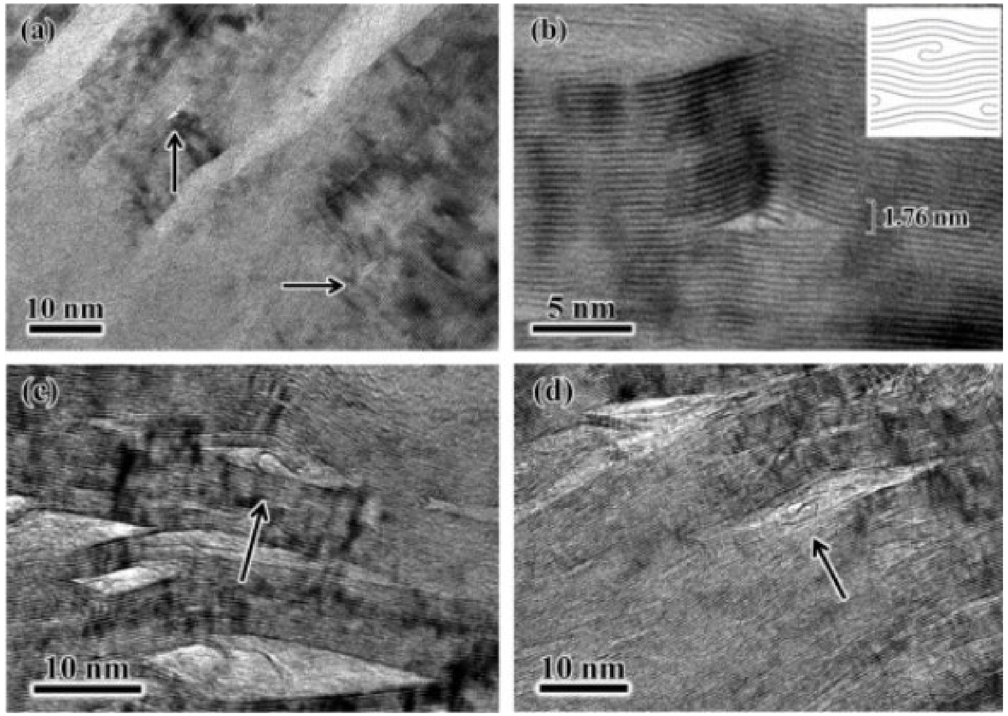


Underlying mechanisms

- Radiation displacement causes contraction in the a/b direction (i.e., parallel to the basal planes)
- From ruck/tuck, ripplocations to microscale kink bands generated



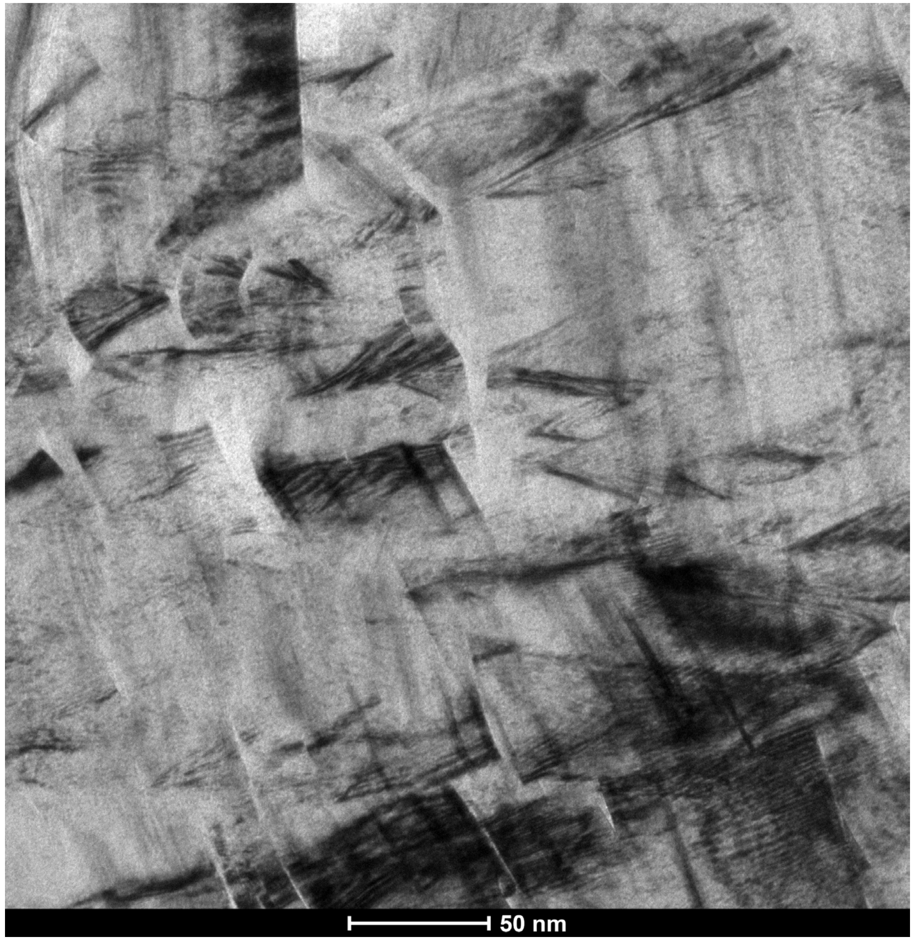
Barsoum, Frontiers in Materials, 2020



Johns et al., Carbon 2020

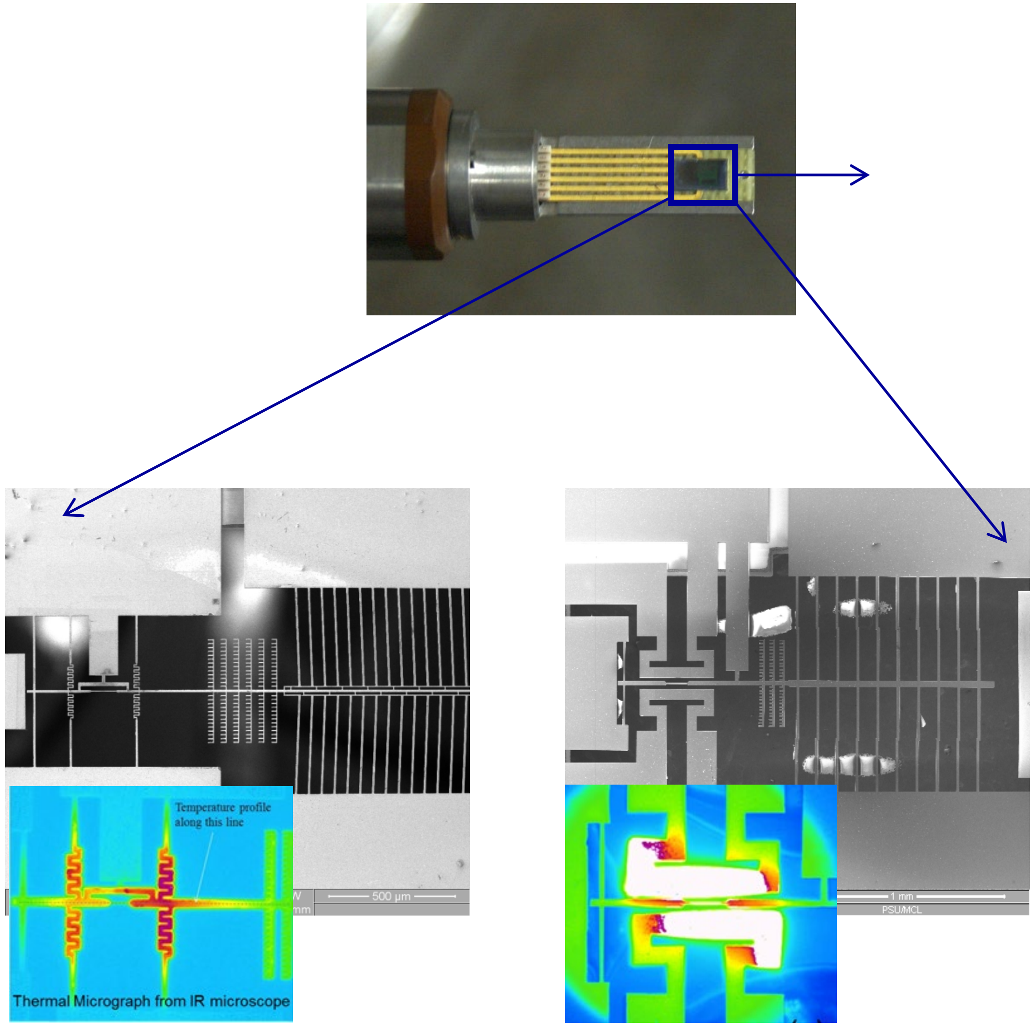
Mechanisms under external force

- External force causes extreme localization in the ripplocations and kink bands.
- Shear band generation becomes natural response to accommodate the localization
- Multitudes of shear bands pile up similar to dislocations (against GB), acting as ‘plastic zones’ impeding the crack front



Discussions

- In situ mechanical tests need to be HRTEM at very low kV to elucidate atomic origin of shear banding.
- Mechanical behavior at high temperature needs to be studied.



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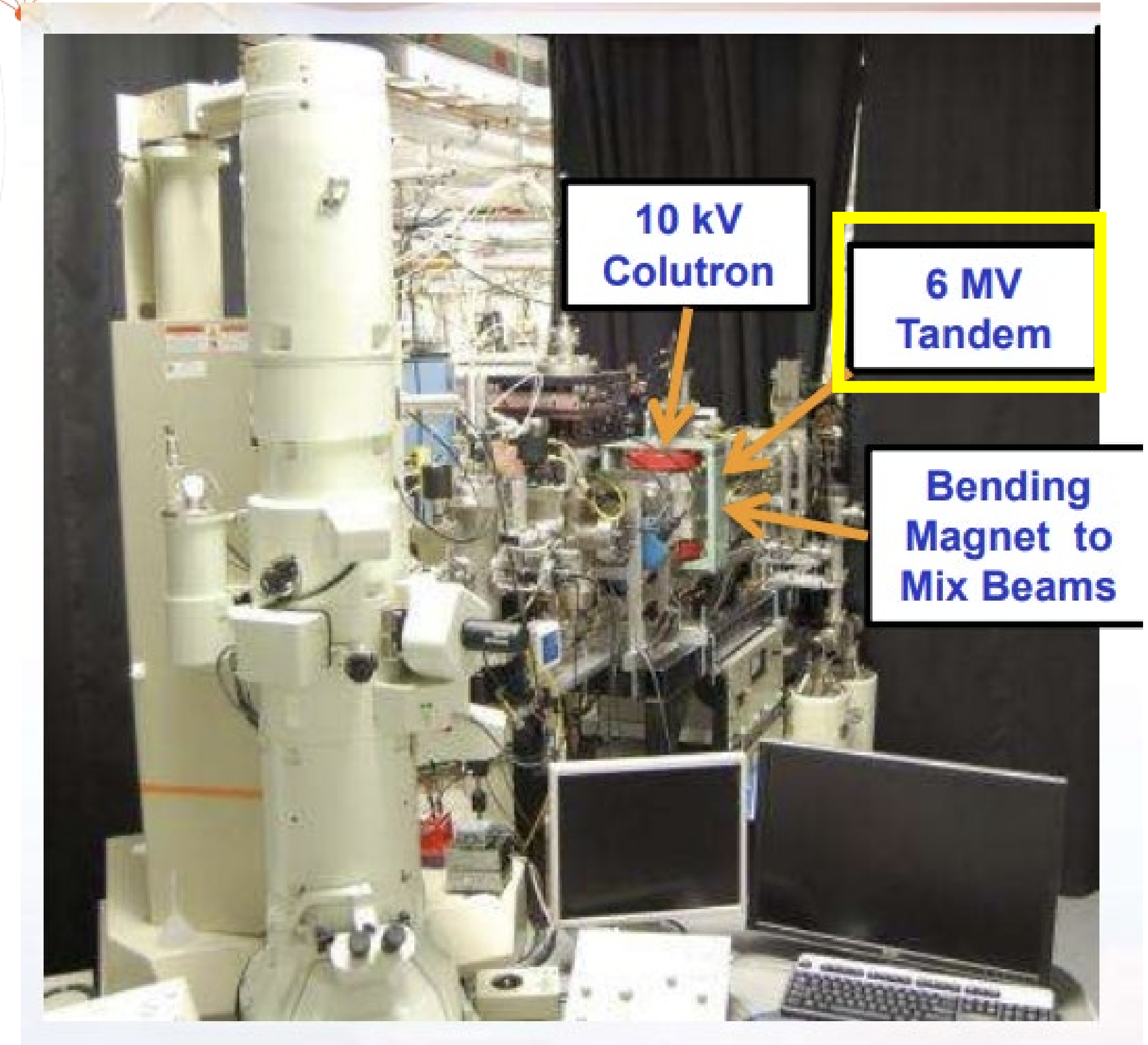
In situ TEM heating & irradiation

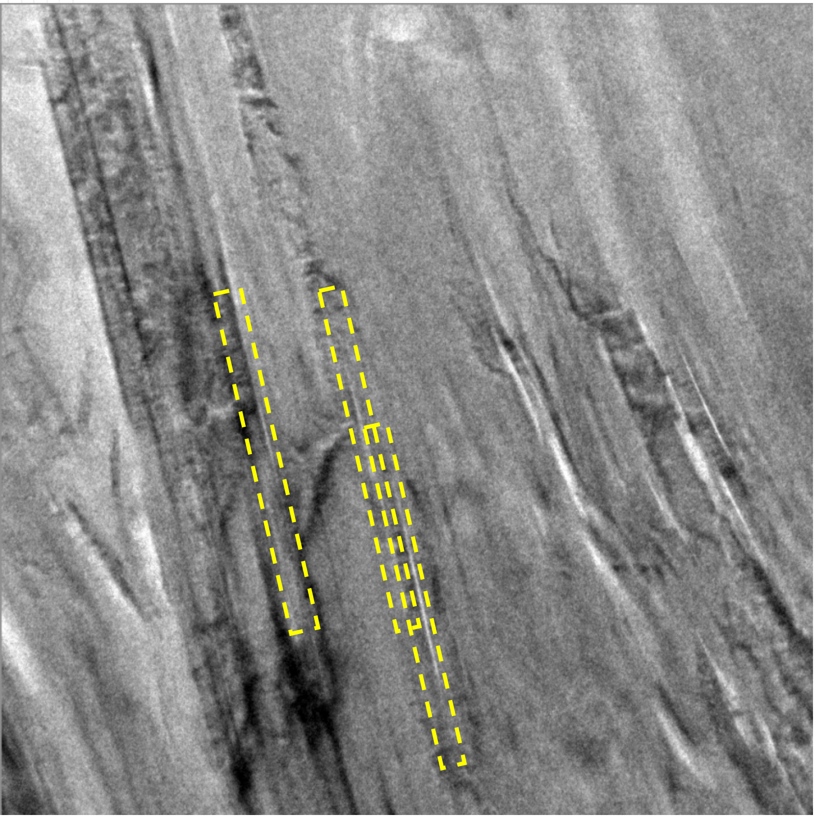


- Pristine IG 110 lamella heated to 800 ^{o}C in 100 ^{o}C increments in Jeol I^{3}TEM at 200 kV.
- Micrograph and a diffraction pattern were acquired in the intervals.
- At 800 ^{o}C, sample is exposed to the 2.8 MeV Au 4^{+} ion beam for 1 hour and 10 minutes (final fluence of 1.6762 x 10^{14} ion cm^{-2}) at a flux of 3.991 x 10^{10} ion cm^{-2} s^{-1} with

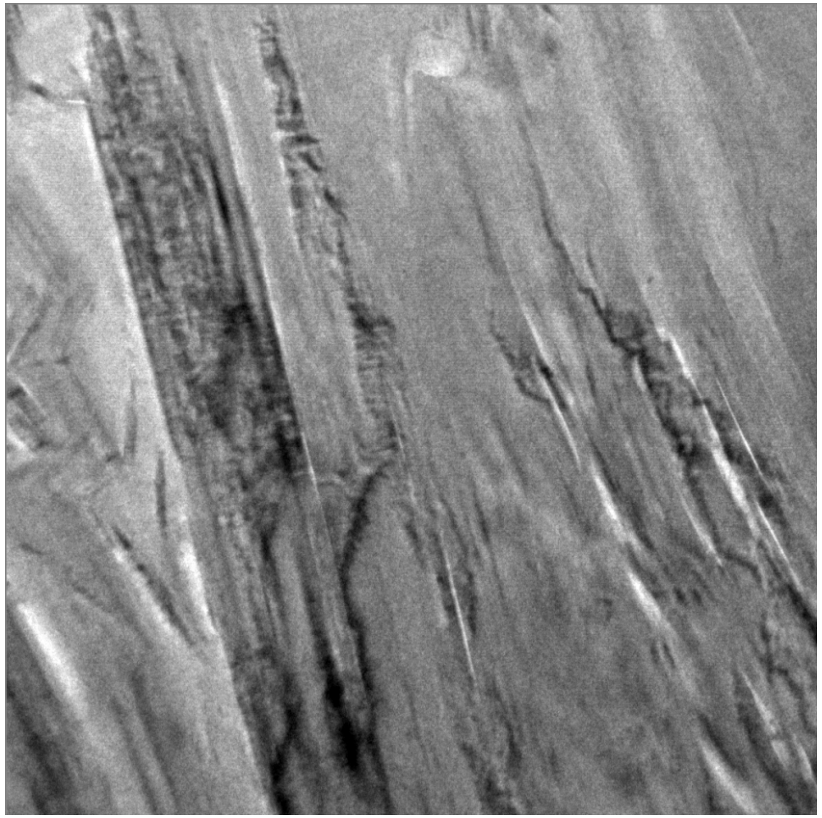
Hattar, Khalid Mikhiel, Blythe Clark, and Jonathan S. Custer. *In situ Ion Irradiation TEM at Sandia's IBL*. No. SAND2012-0648C. Sandia National Lab.(SNL-NM), Albuquerque, NM (United States), 2012.

Micro-crack closure upon irradiation

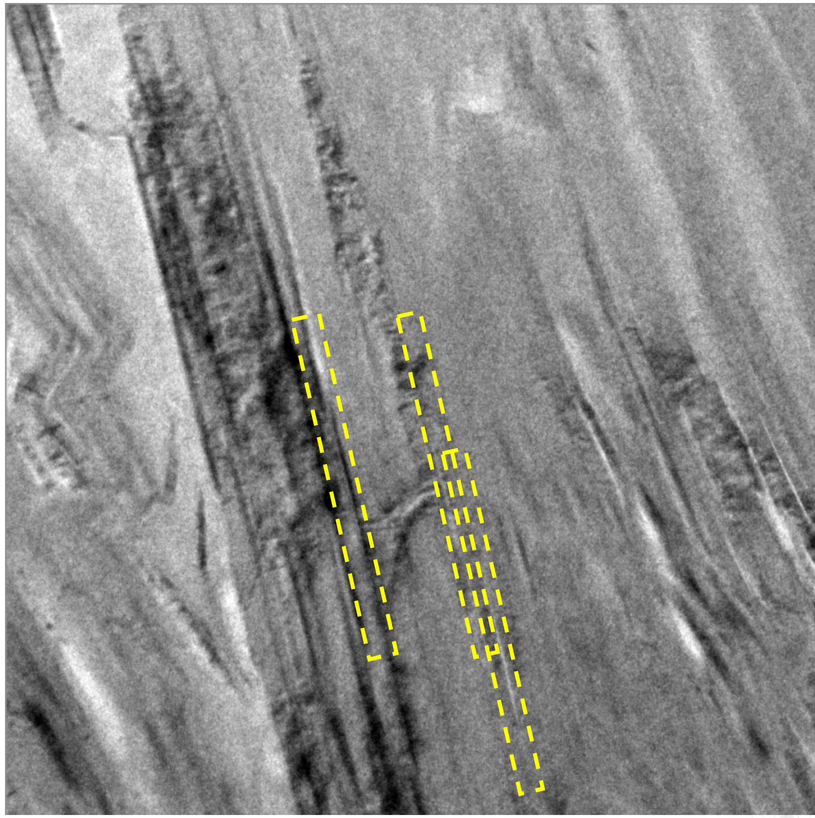
Fluence: 0



Fluence: 7.423 x 10^{13} ion cm^{-2}

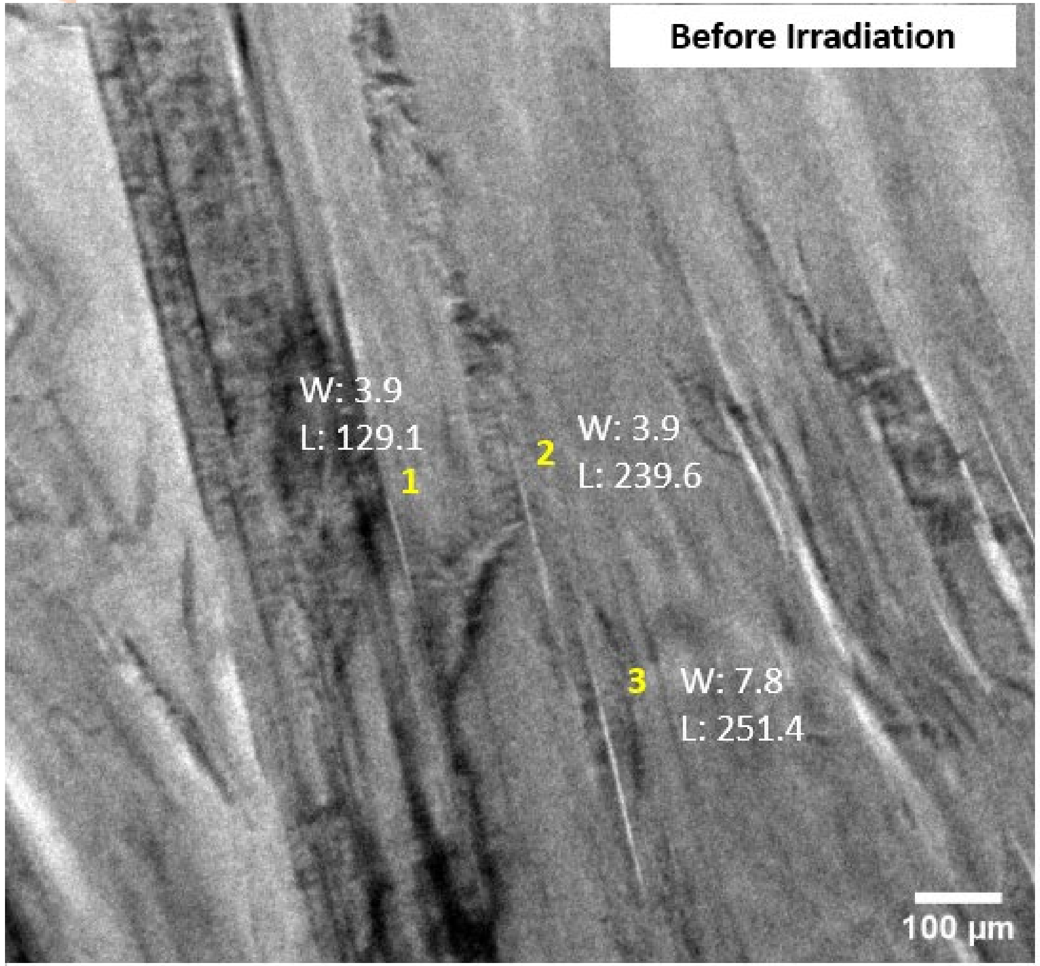


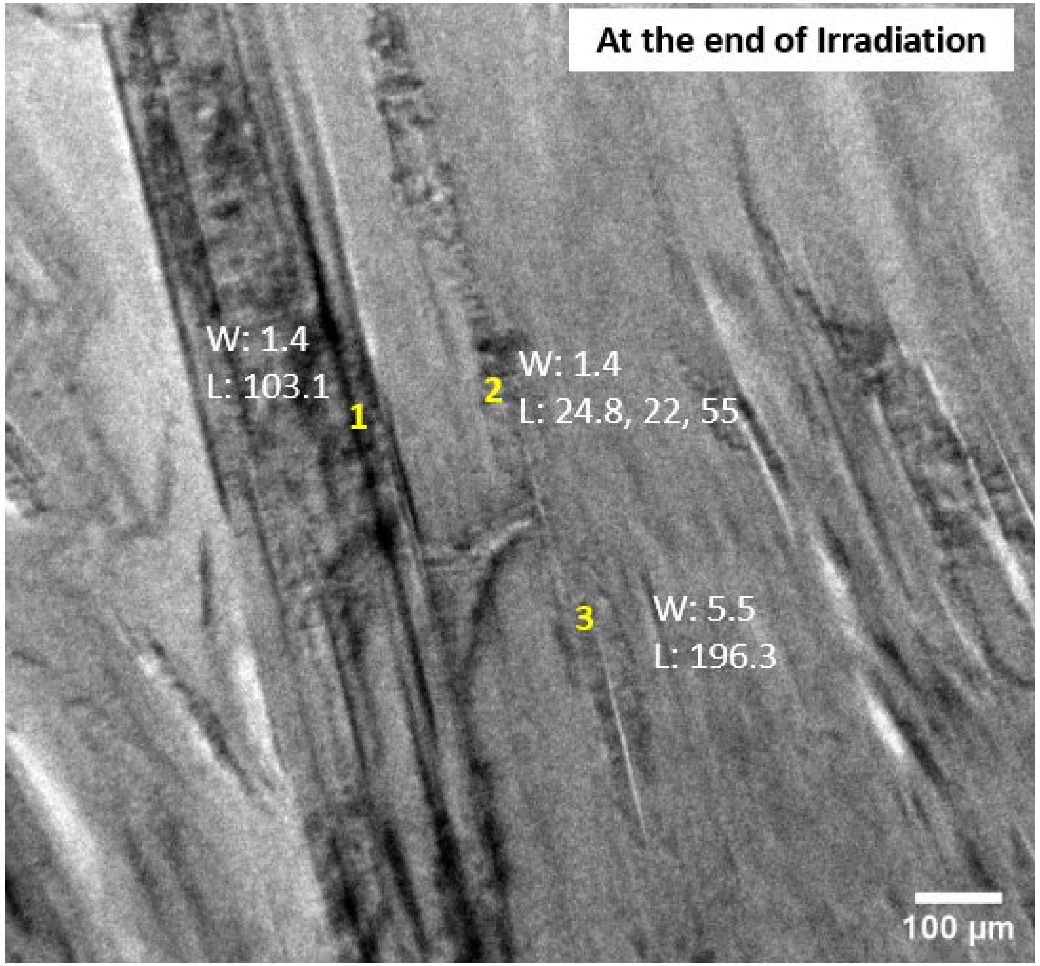
Fluence: 1.486 x 10^{14} ion cm^{-2}



Scale bar: 100 um

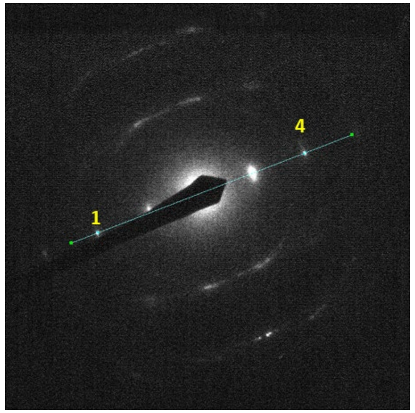
Micro-crack closure upon irradiation

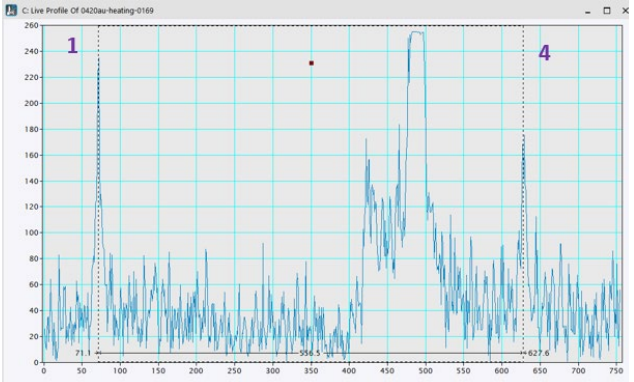


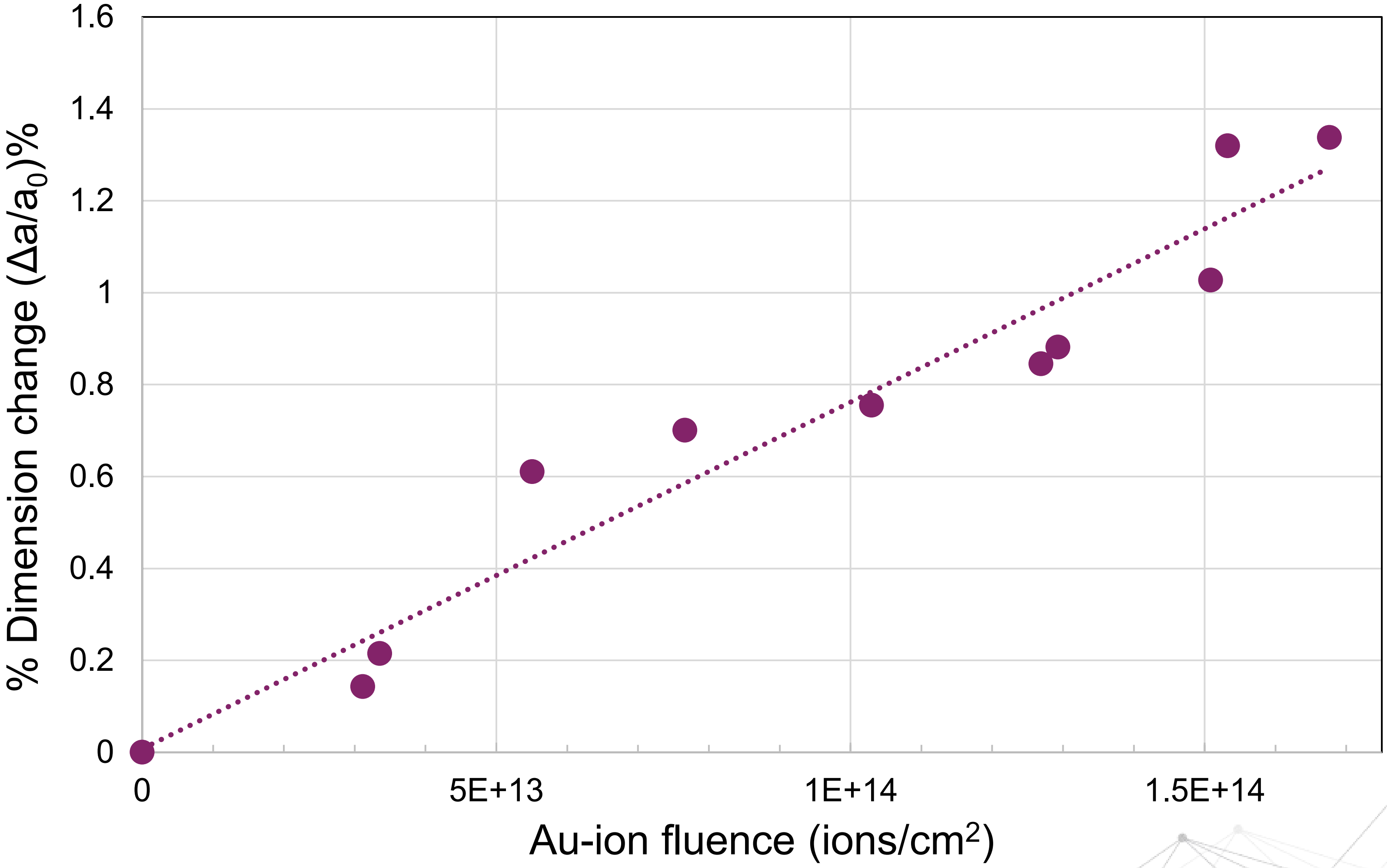


All measurements are in um W: Width of the crack L: Length of the crack

Expansion upon heating and irradiation







Conclusions

- Combination of experiment and FEA modeling was pursued. The model reasonably predicts crack propagation – but not crack nucleation. This highlights importance of localized residual stress (mechanical hotspots)

- Defects originating from ion irradiation showed very high mobility under external stress. Shear banding appeared to be the mechanism for accommodation of localized deformation. These bands produce ‘strain hardening’ behavior by impeding further defect motion

- Opportunities exist to explore atomic origins of defect generation and dynamics under external mechanical load and temperature as function of radiation dosage.

Acknowledgements

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