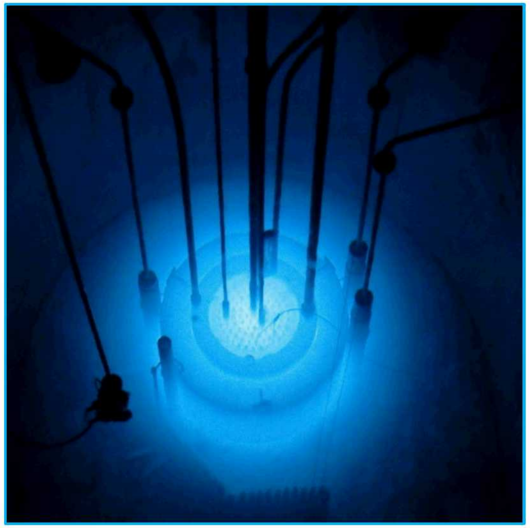
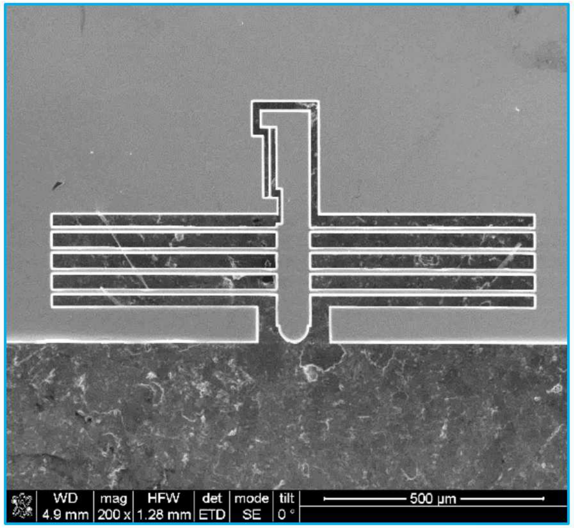
SAND2018-2850C

Investigating Irradiation Creep by *In Situ* TEM





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Nanocrystalline Metals and Radiation

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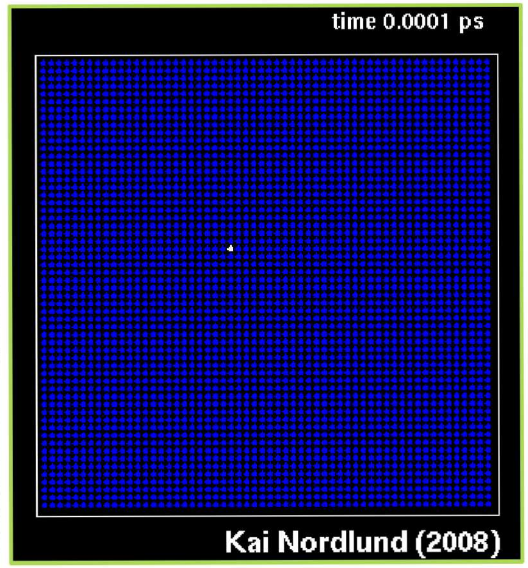
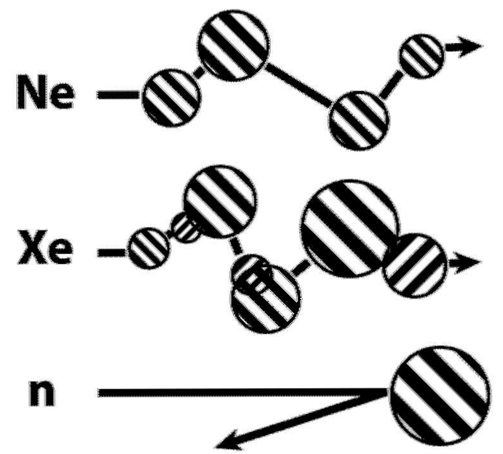
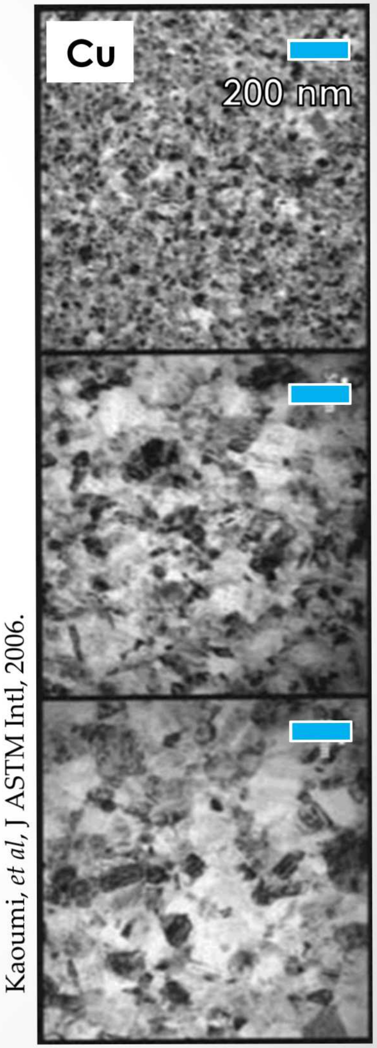
- Exemplary mechanical properties.
- Abundant sinks for structural and chemical defects.
- …but provide ample diffusion pathways
- …and may undergo coarsening with irradiation



10 keV Au in Au, *via* Wikimedia Commons.



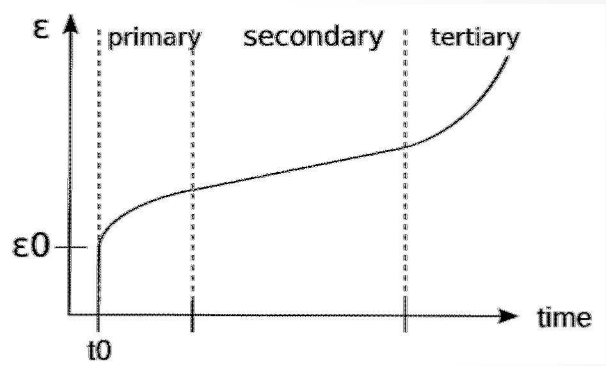
Averback, J Nucl Mater, 1994.



Kaoumi, *et al*, J ASTM Intl, 2006.

Radiation Creep

- Still many questions regarding irradiation creep in nanocrystalline metals
- Typical creep effects superposed with radiation effects
  - Swelling and growth phenomena
- Complex phenomenon!

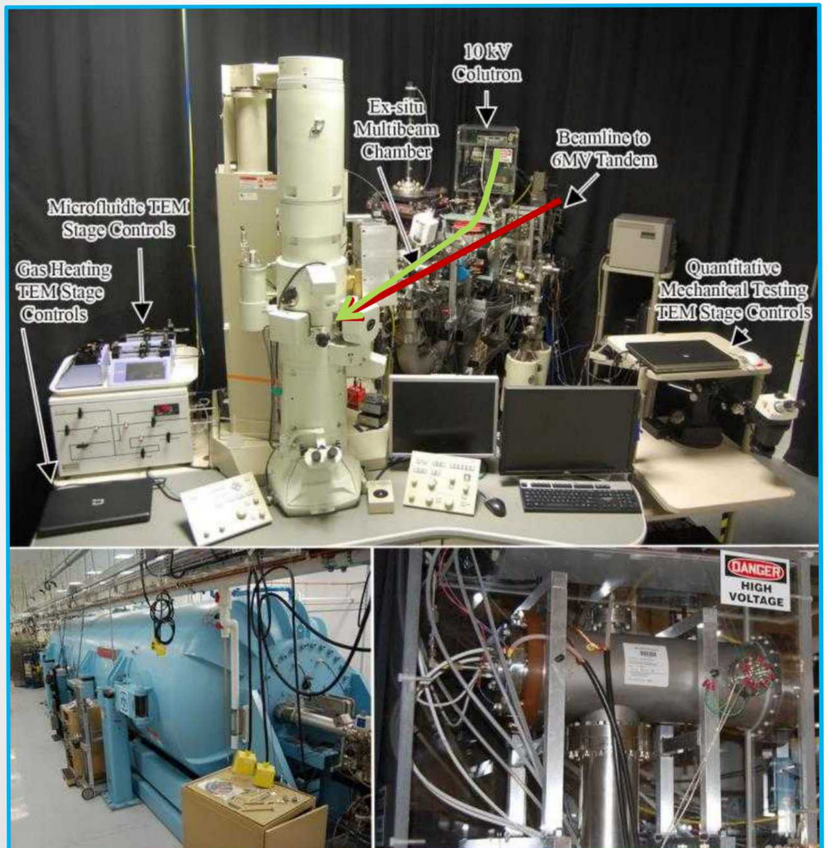


How does the creep behavior change with accumulated dose and concurrent irradiation?

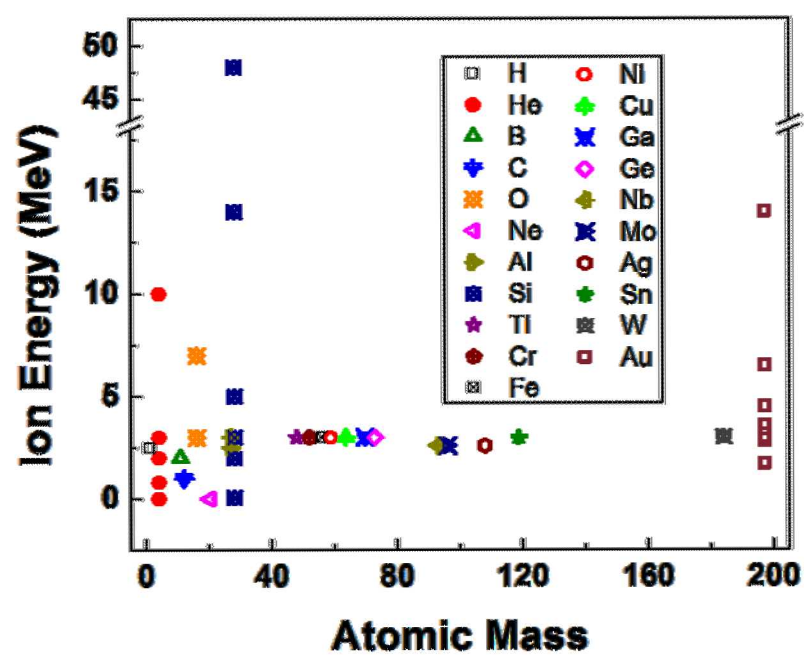
Sandia's *In situ* Ion Irradiation TEM (I^3TEM)

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem



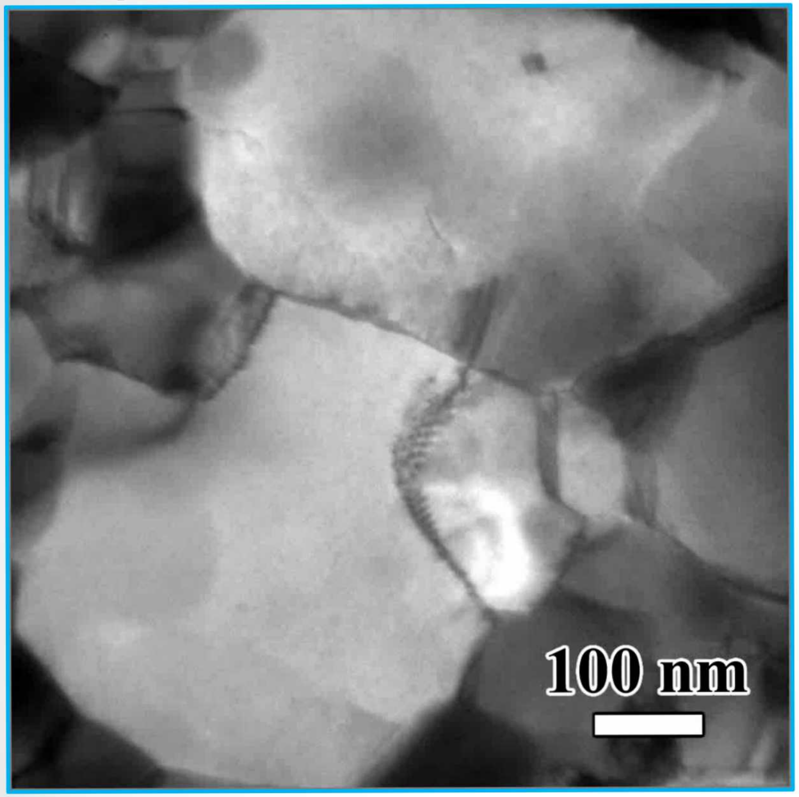
Hattar, *et al*, Nucl Instr Meth Phys Res B, 2014.

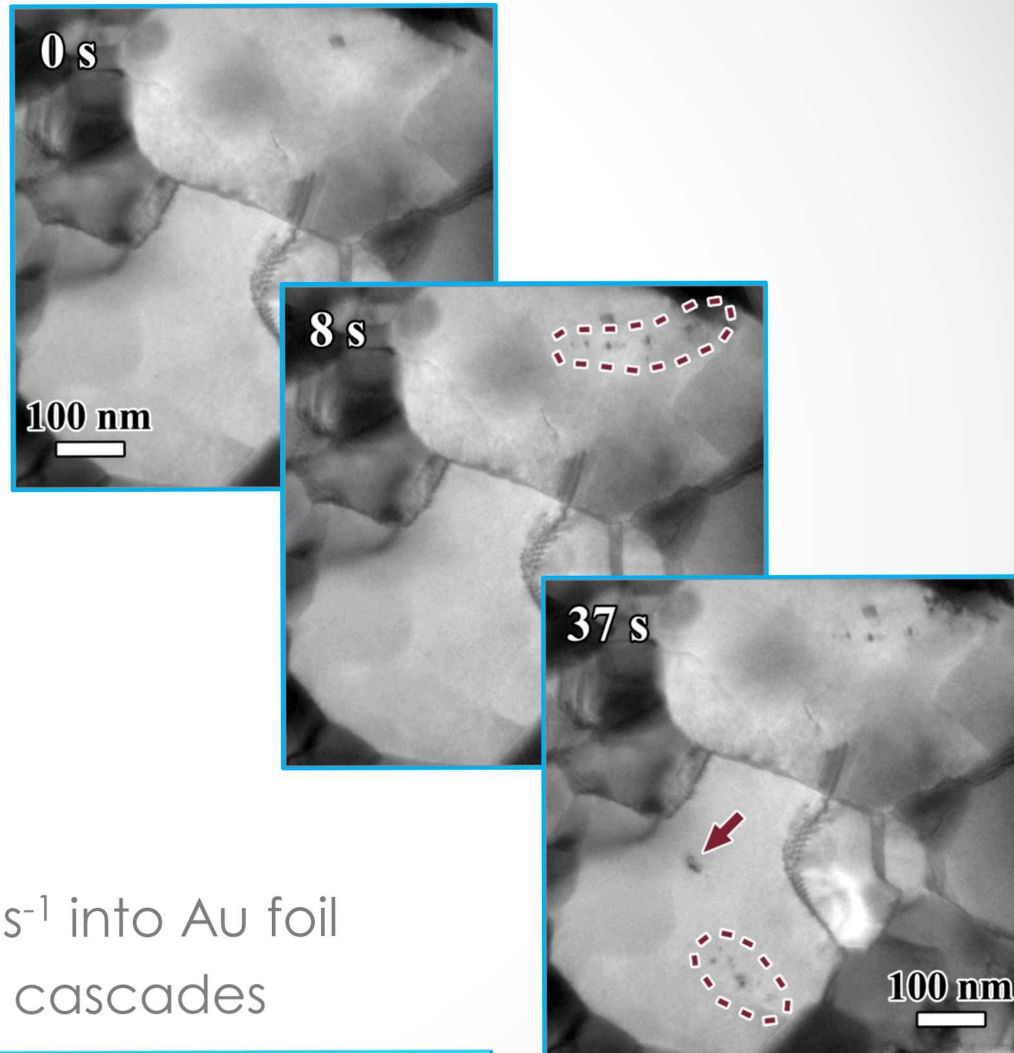


Enables real-time studies of samples under irradiation and other stimuli.

Defects during *in situ* irradiation

Video speed ×5.



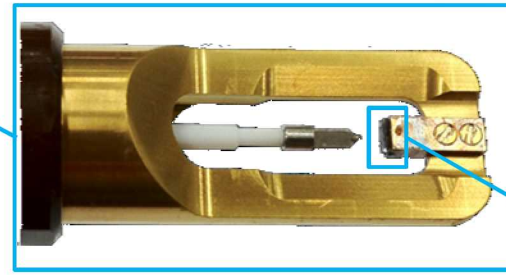


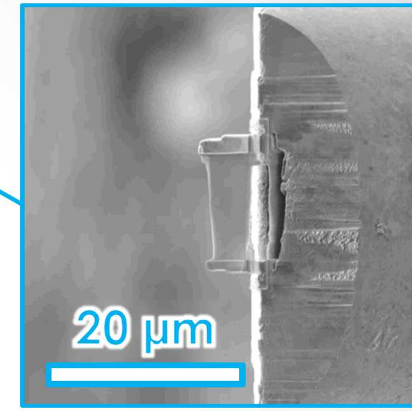
- Au^{6+} at 2.1×10^{8} ions cm^{-2} s^{-1} into Au foil
- Large defect clusters from cascades

Effects from even individual ions captured

Hattar, *et al*, Nucl Instr Meth Phys Res B, 2014.

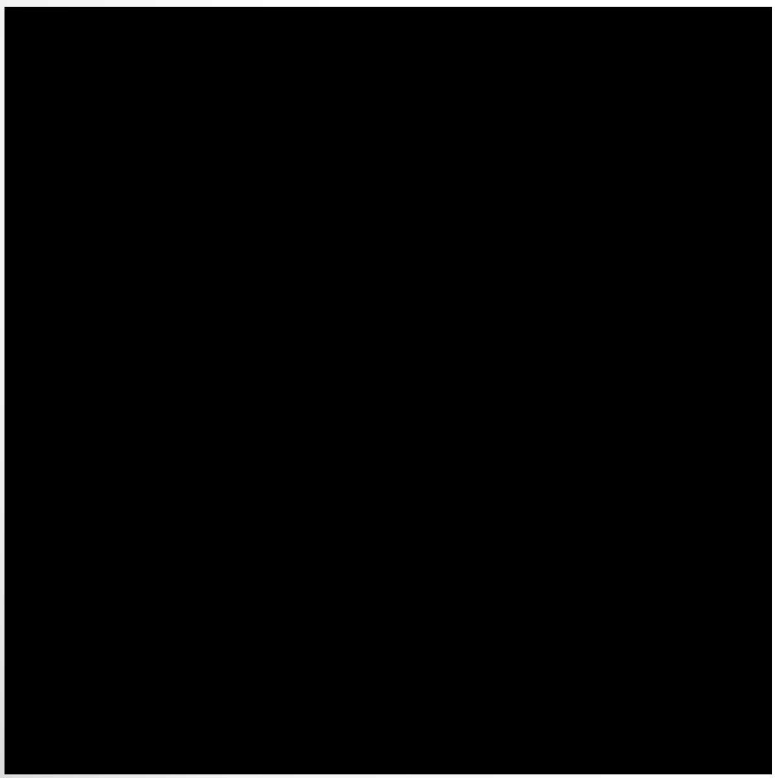
In Situ TEM Nanoindentation

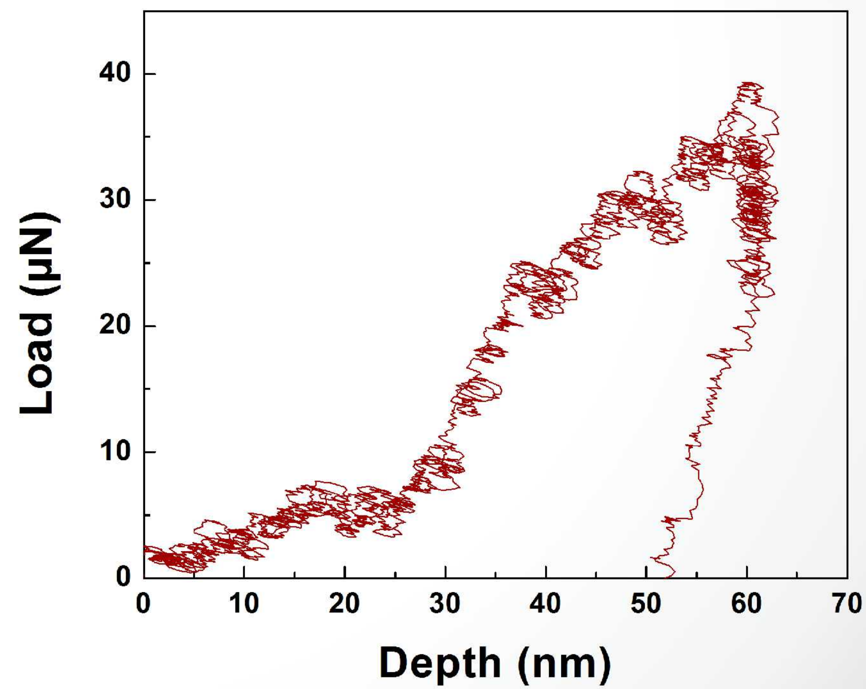




Hysitron PI95 *In Situ* Nanoindentation TEM Holder

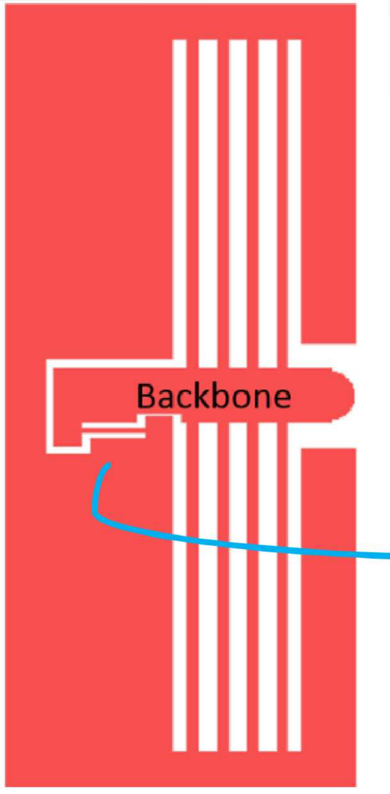
- Sub nanometer displacement resolution
- Quantitative force information with µN resolution

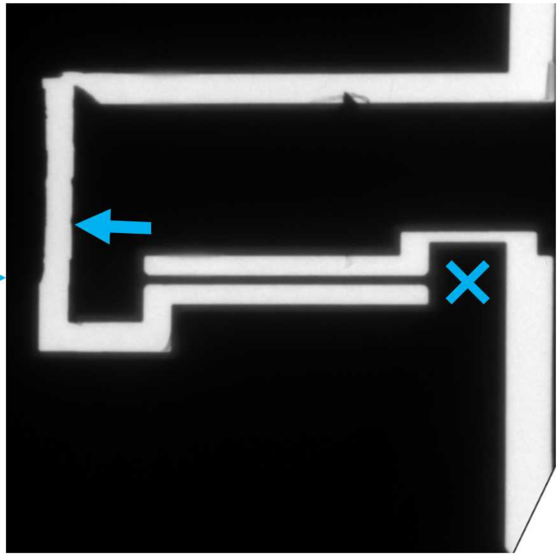




Quantitative deformation & observation at the nanoscale

Micro Tension Frames

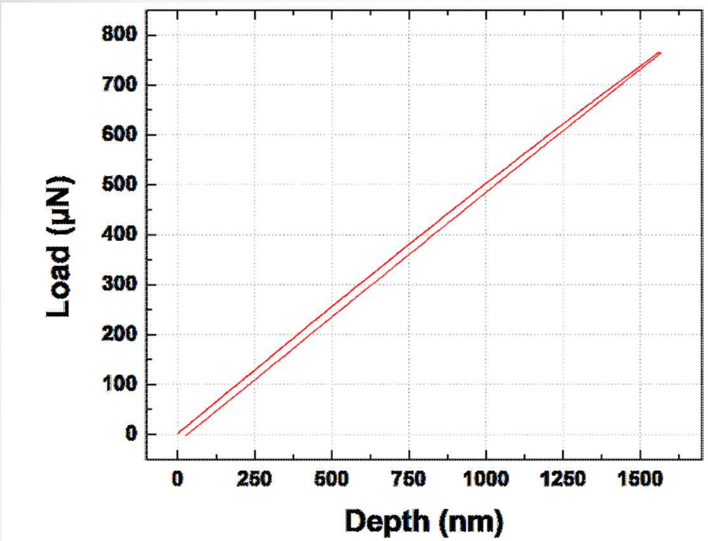


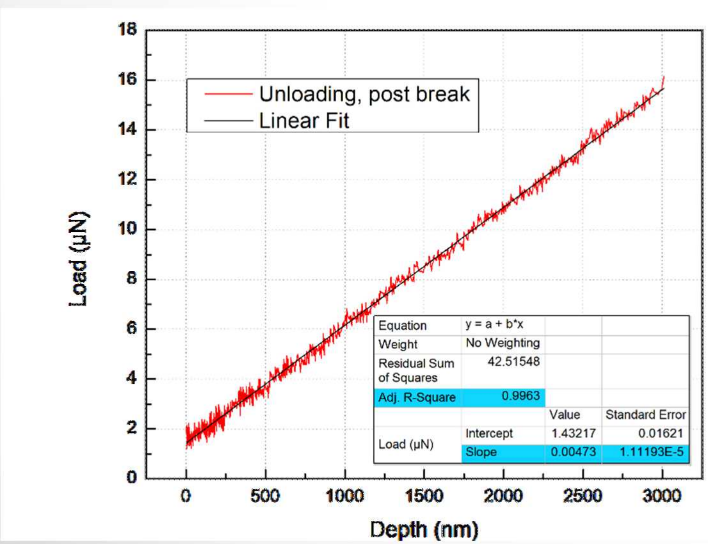


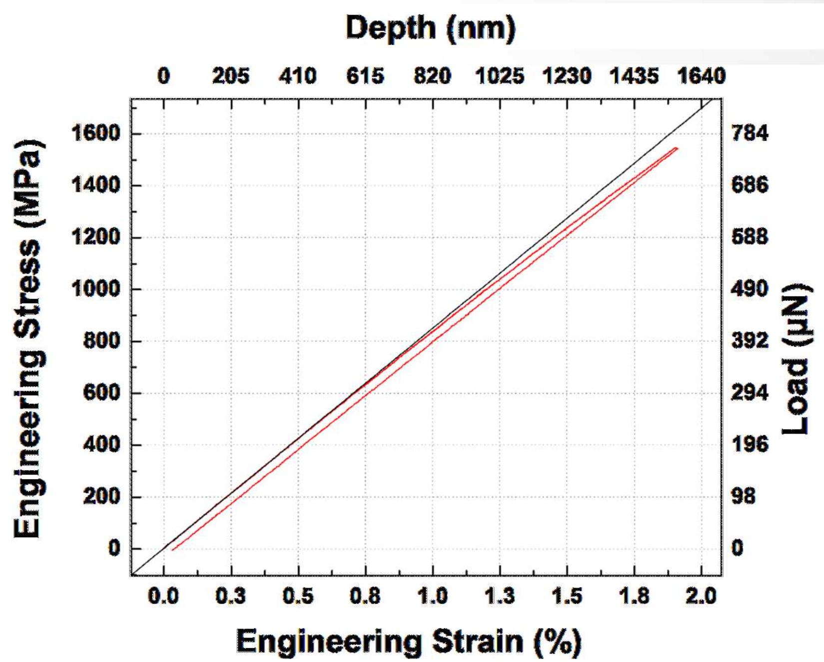
- Si frame with freestanding metal film
- Actuated by nanoindenter
- Spring design mitigates misalignment of the indenter

Test frame enables tension testing at the micro/nanoscale!

Monotonic Response





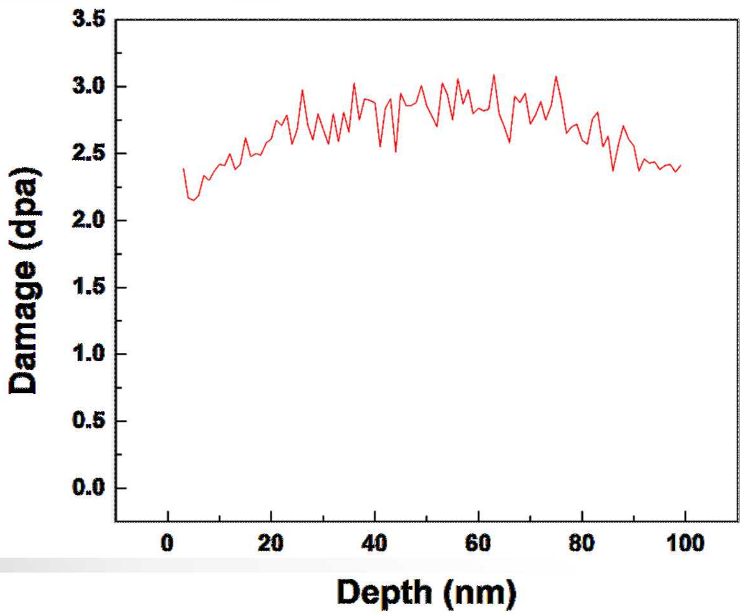


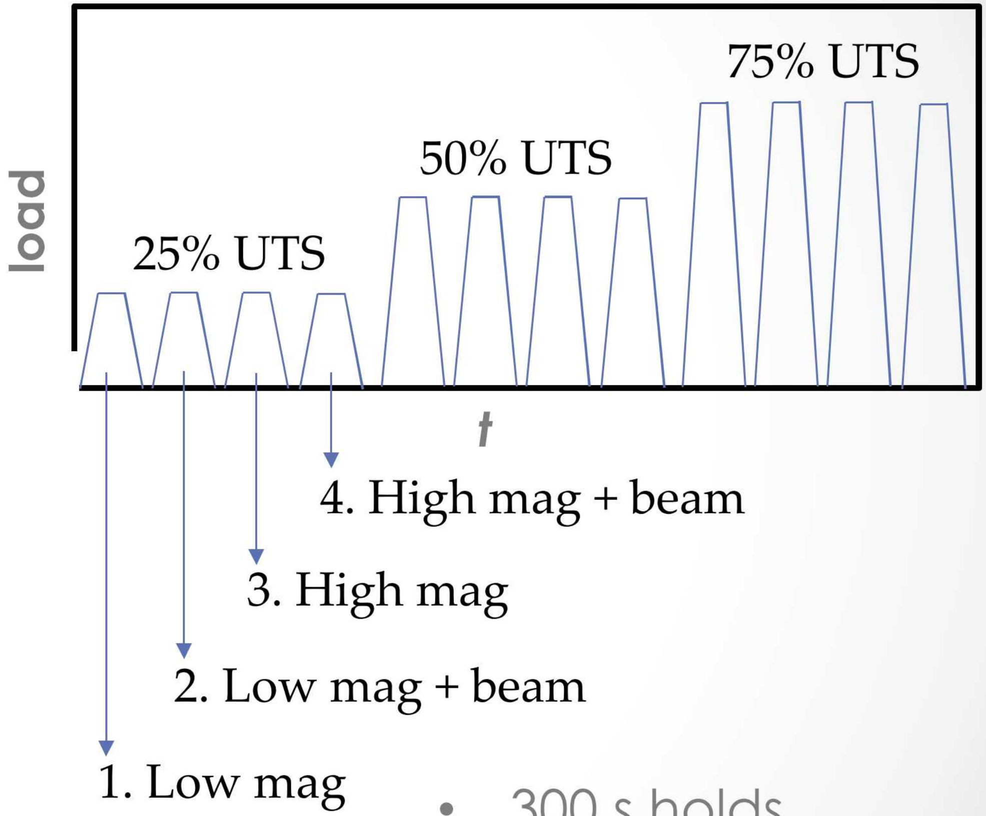
- Linear elastic frame response
- Depending on design, frame contributes 1-10% of measured load

Removal of frame response allows us to calculate the stress-strain response of the sample alone.

Irradiation Creep

- 1.4 MeV Zr
- 4.6×10^{13} and 10^{14} ions cm^{-2}
- Nominally 0.26 and 2.6 dpa
- 4.2×10^{10} ions cm^{-2} s^{-1}
- 2.5×10^{-4} dpa s^{-1}

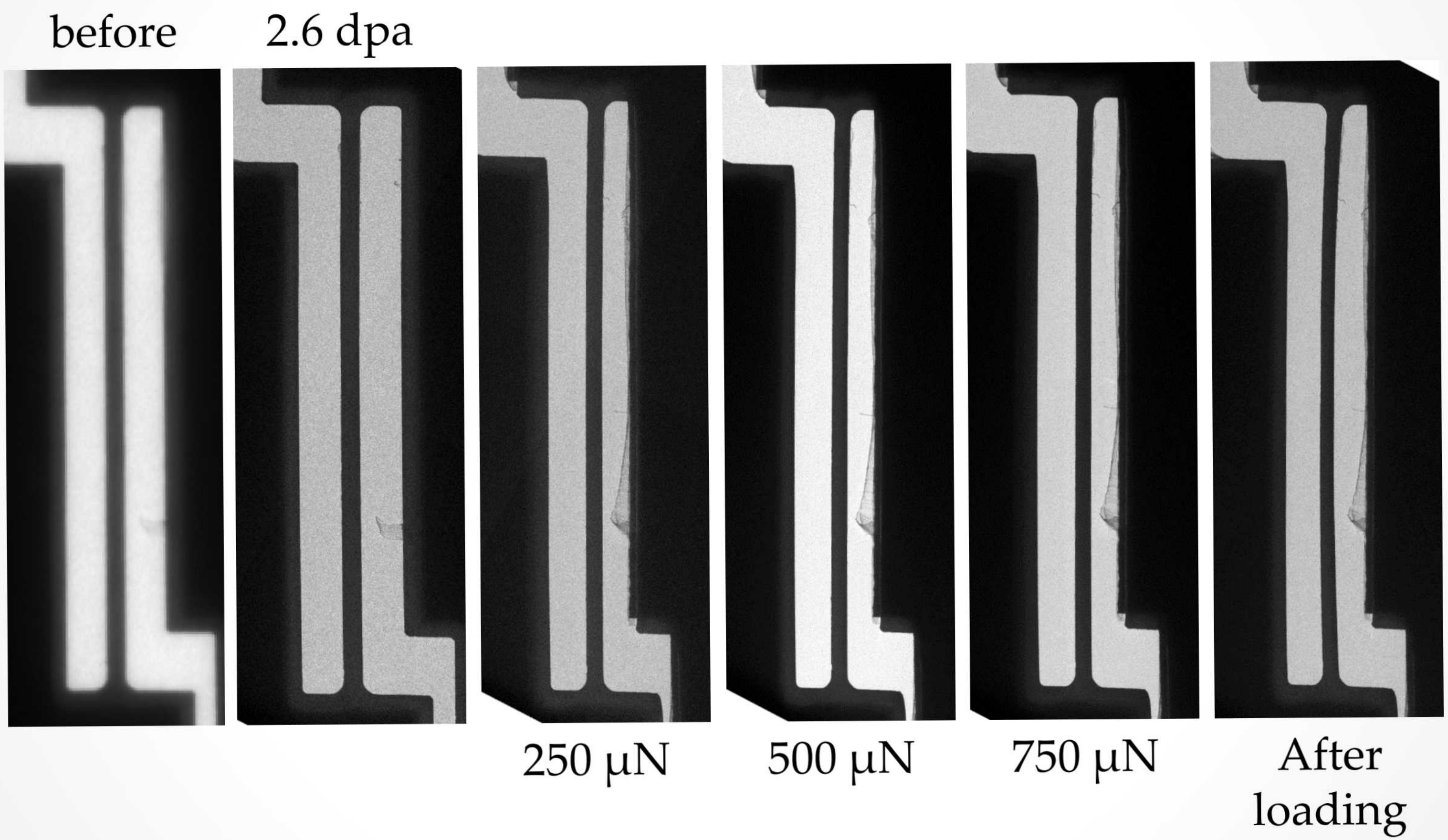




- 300 s holds
- 7.5×10^{-2} dpa $cycle^{-1}$

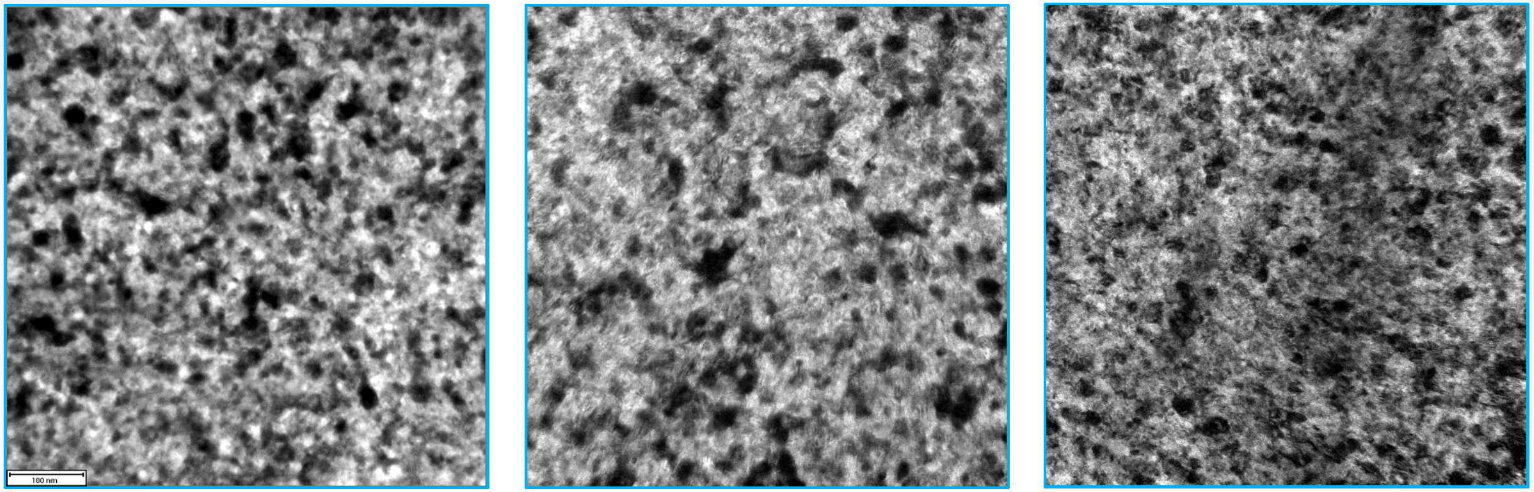
Measuring overall changes and microstructural changes with and without irradiation.

Elongation: 2.6 dpa

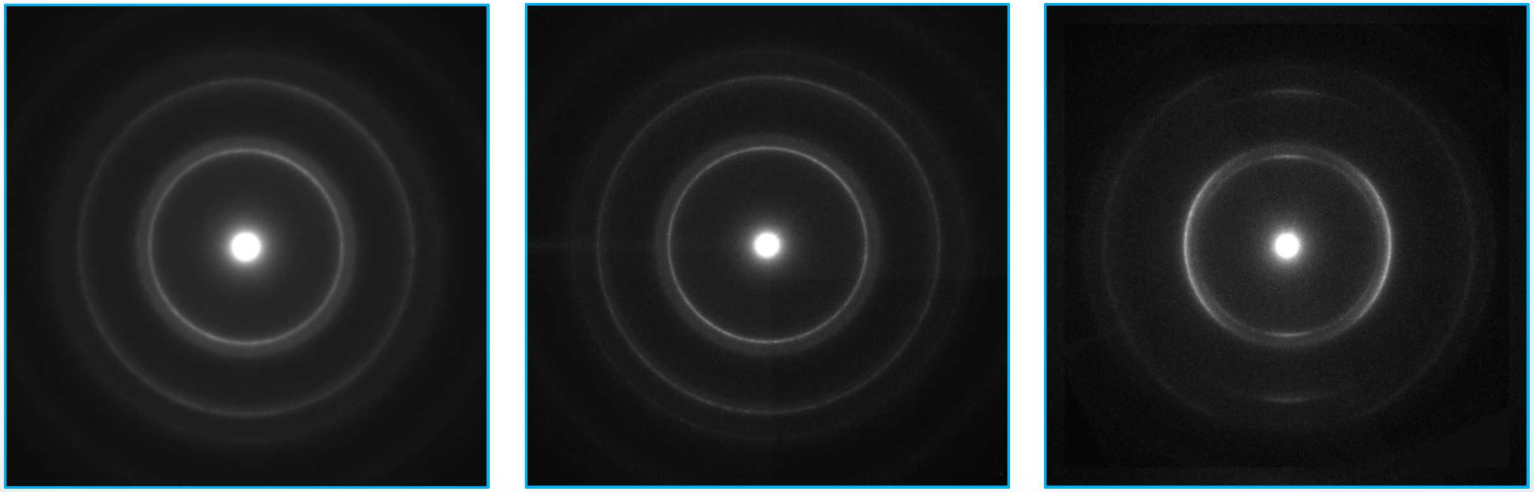


Clear extension, particularly during the 750 μN loadings.

Structure: 2.6 dpa



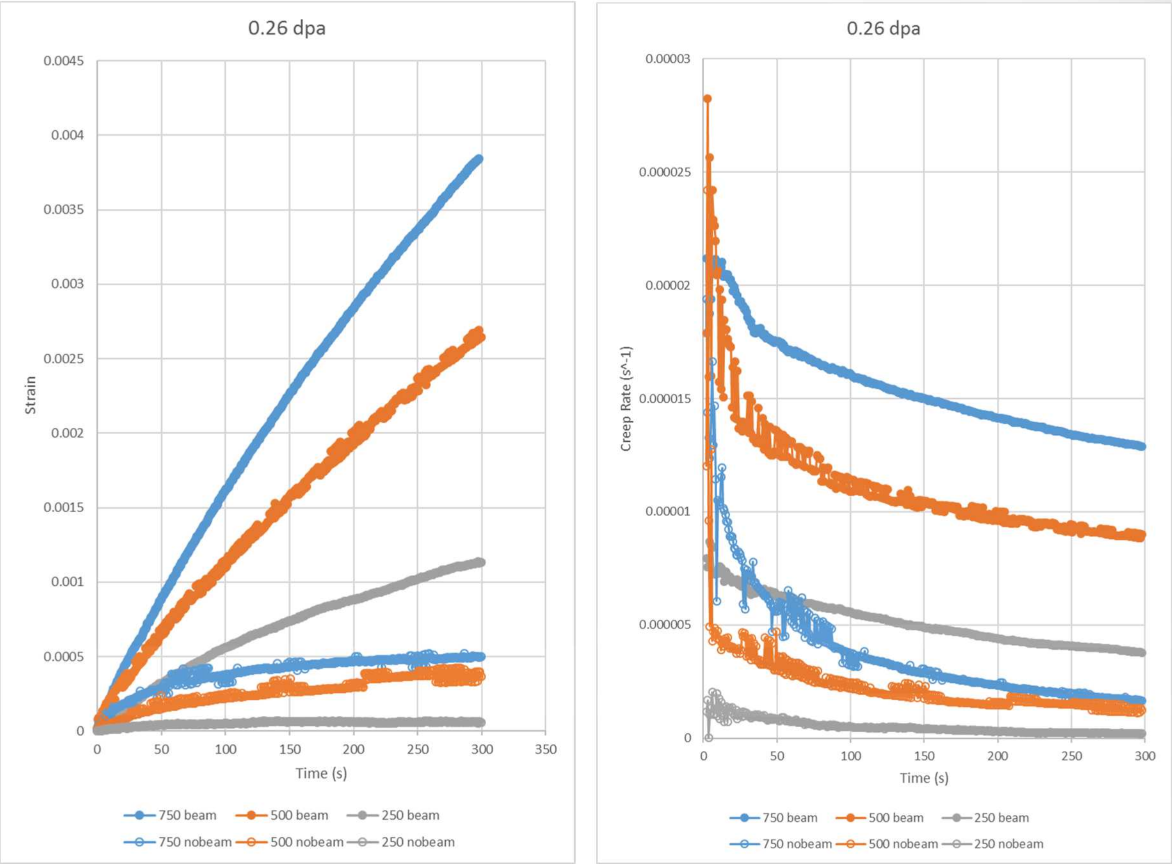
before 2.6 dpa After loading



Little change with irradiation, but deformation texture developed during loading.

Creep Rates

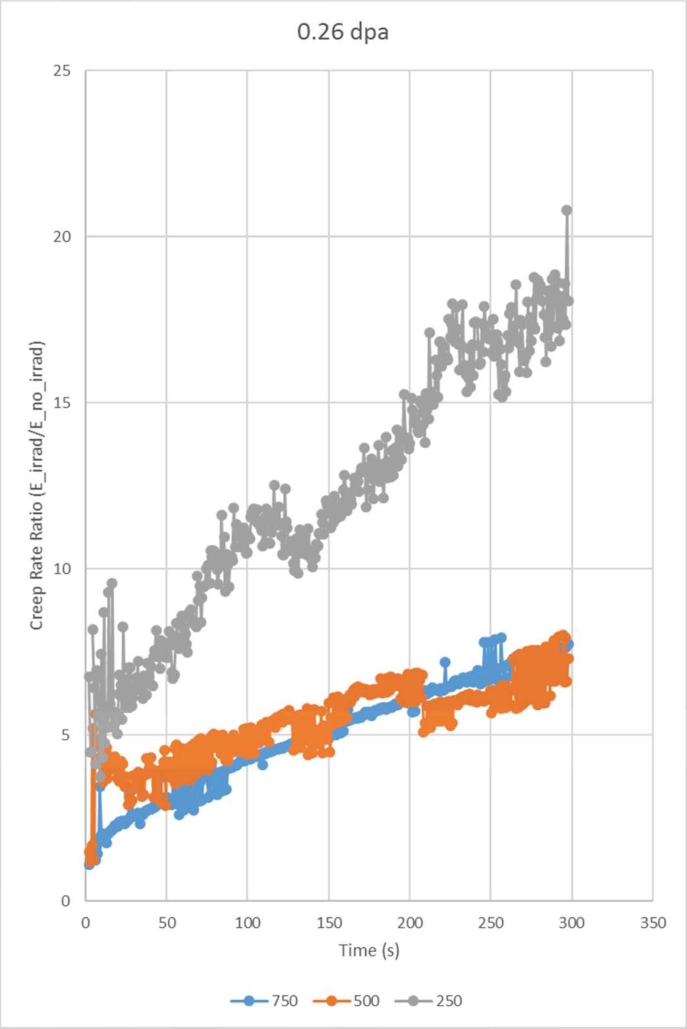
- Automated frame-by-frame position measurement
- B_0 found to be approximately 3 MPa^{-1} dpa^{-1}



$$\dot{\varepsilon}_{irrad} = B_0 \sigma \varphi$$

Rapid extraction of a key irradiation creep parameter

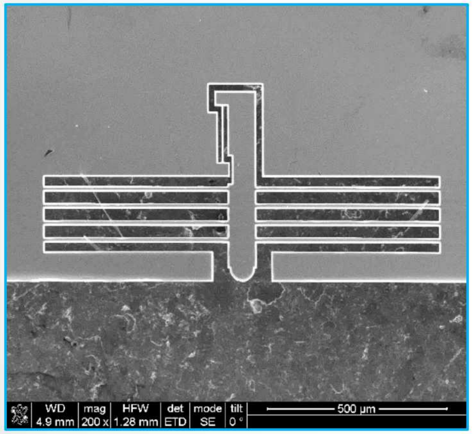
Creep Rate Comparison

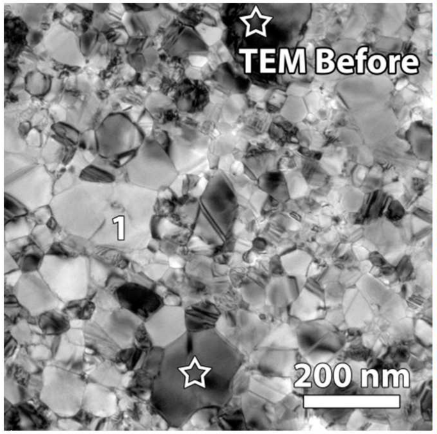


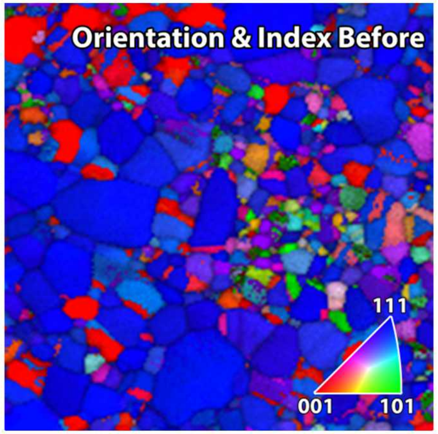
- Relative enhancement is greatest for low load conditions

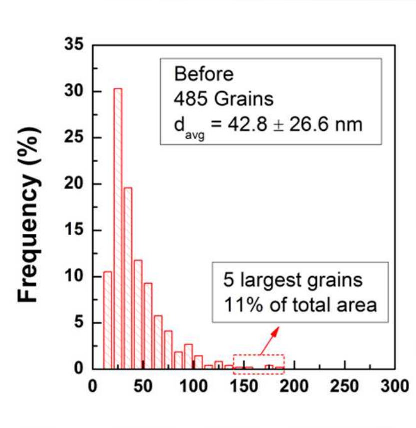
Summary

- Demonstrated a capability for quantifying tensile creep rates in small scale samples in radiation environments
- Built-in microstructural analysis capability
- Extraction of key radiation creep parameters





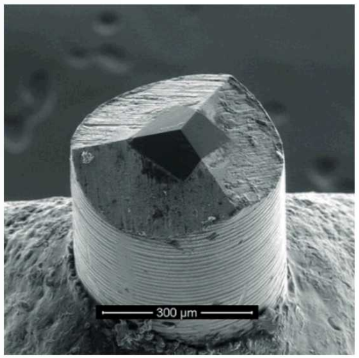


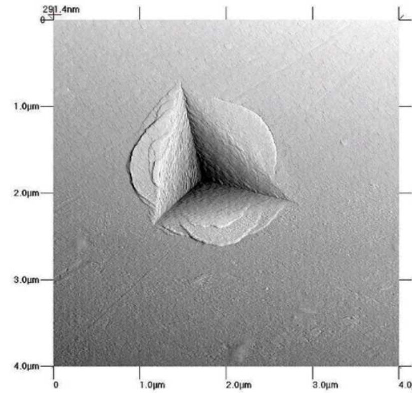


Acknowledgements: A. Darbal (AppFive), D. Kaoumi (University of South Carolina), A. Leff (Drexel University), and C.M. Barr, B.L. Boyce, D.L. Buller, T.A. Furnish, C. Gong, H. Lim, M.T. Marshall, W.M. Mook, and B.R. Muntifering (Sandia National Laboratories). This work was fully supported by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy.

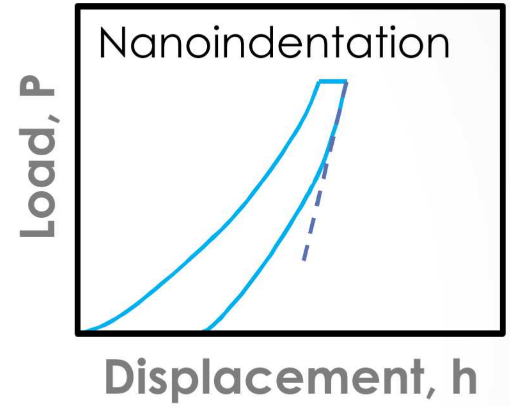
Small-Scale Mechanical Testing

- Lots of data from small volumes of material
- Length scales overlap with ion ranges

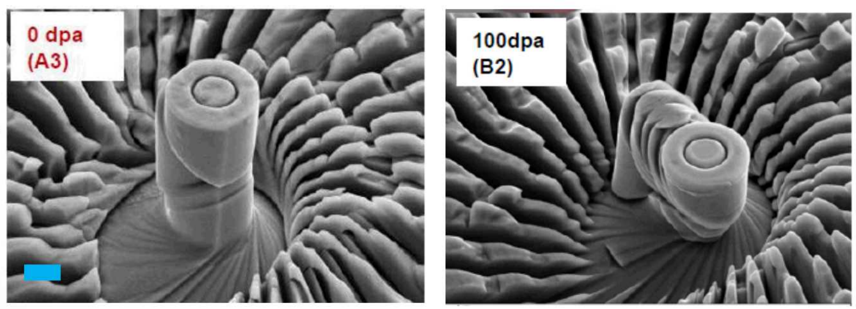




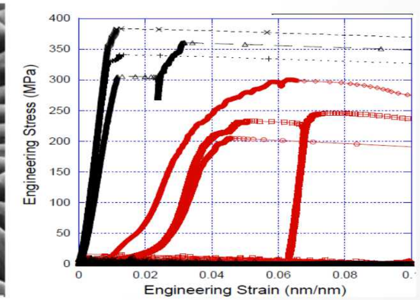
J. Puthoff, *via* Wikimedia Commons.



30 MeV Cu^{5+}
100 DPA

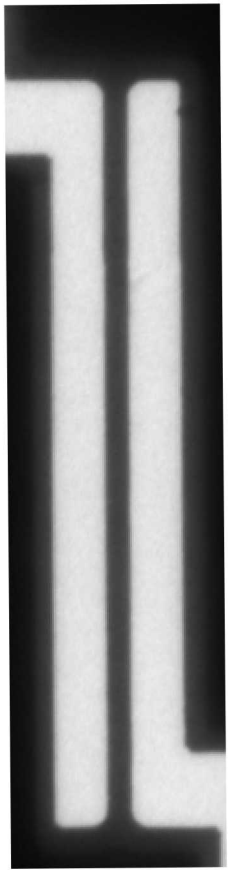


Sharon, *et al*, Mater Res Lett, 2014.

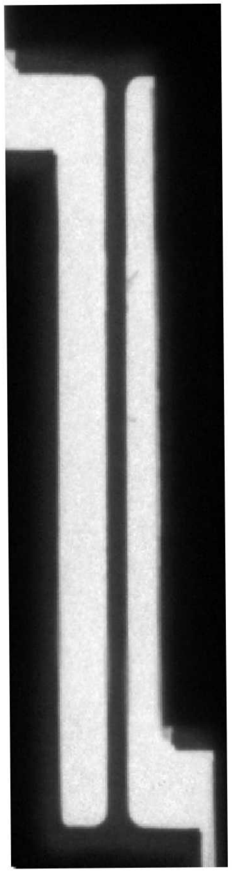


Powerful tools for evaluating radiation effects on mechanical properties!

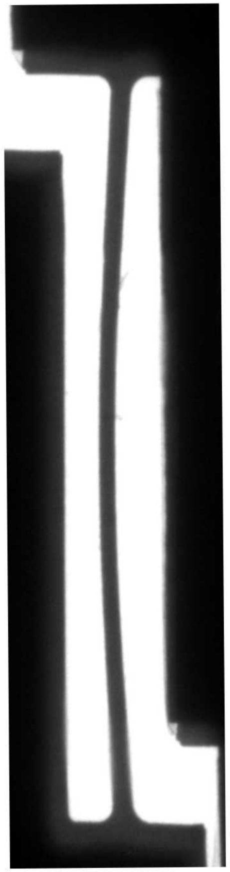
Elongation: 0.26 dpa



before



0.26 dpa



After loading

Minimal change with irradiation alone, but clear extension during loading.

Structure: 0.26 dpa

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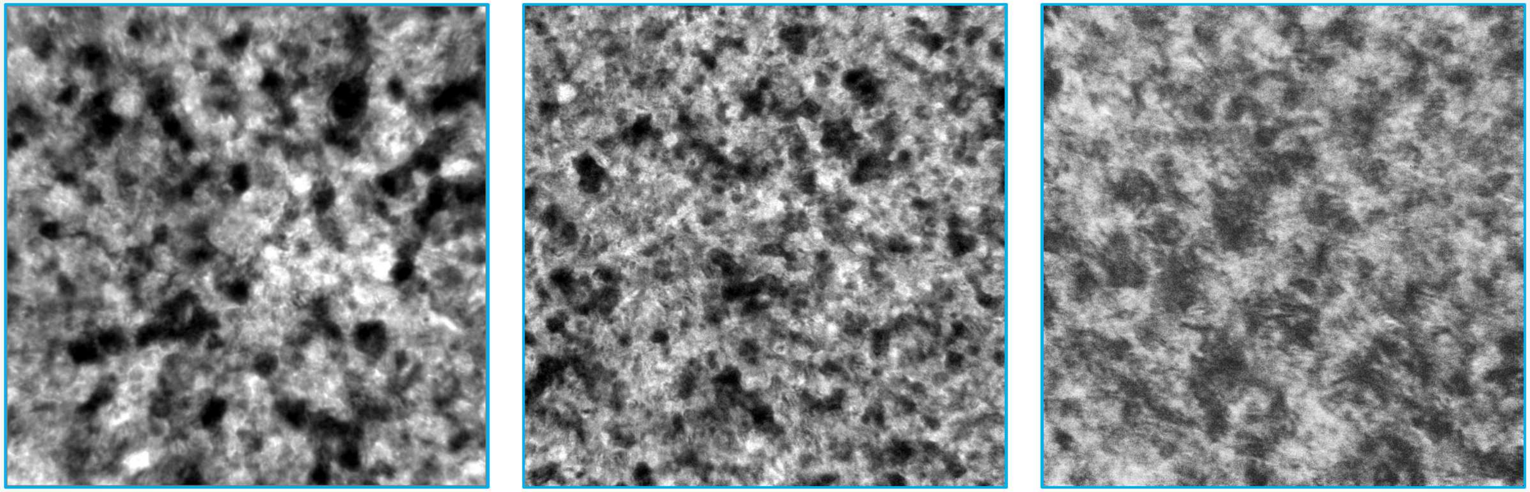
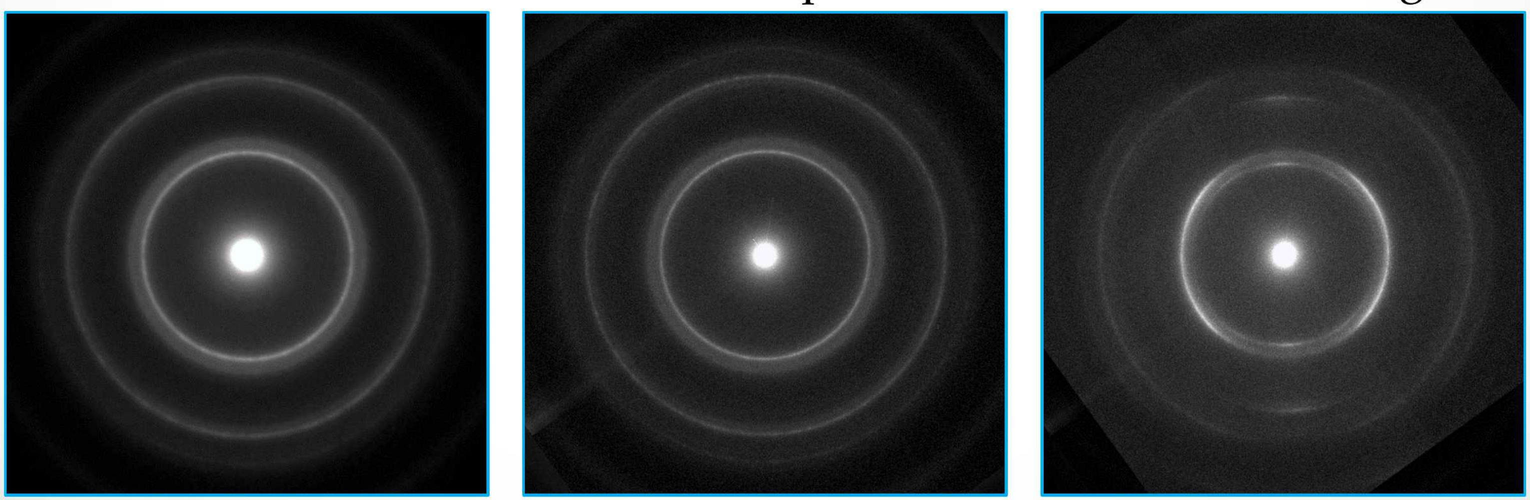
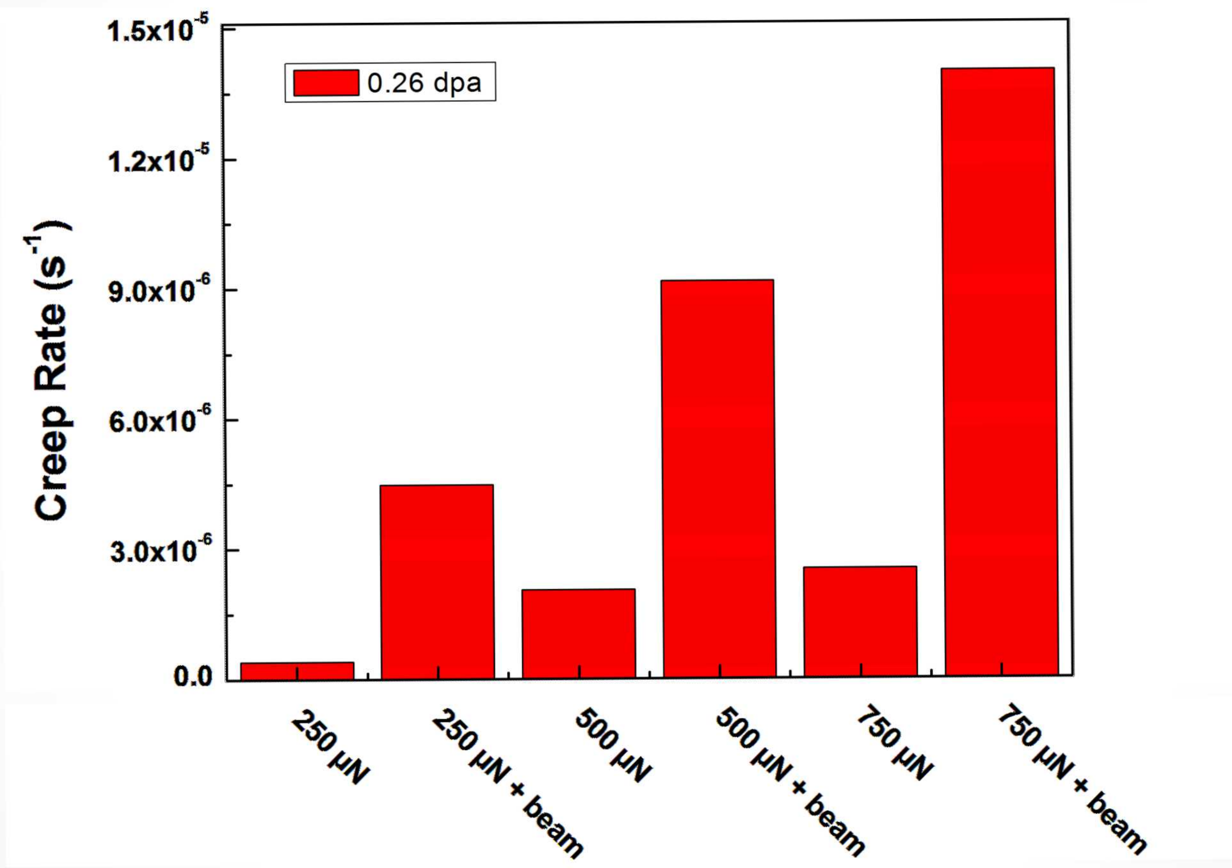


before | 0.26 dpa | After loading



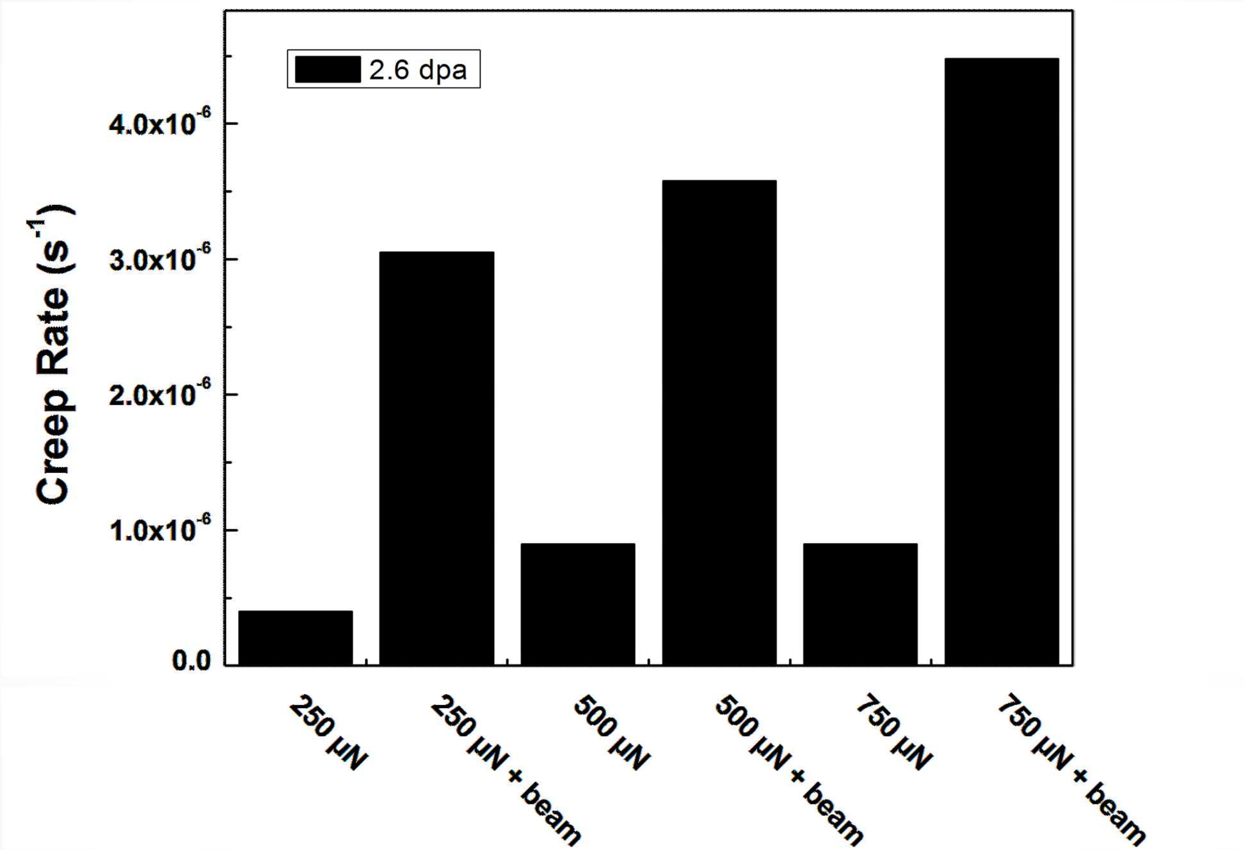
Little change with irradiation, but deformation texture developed during loading.

Creep Rates: 0.26 dpa



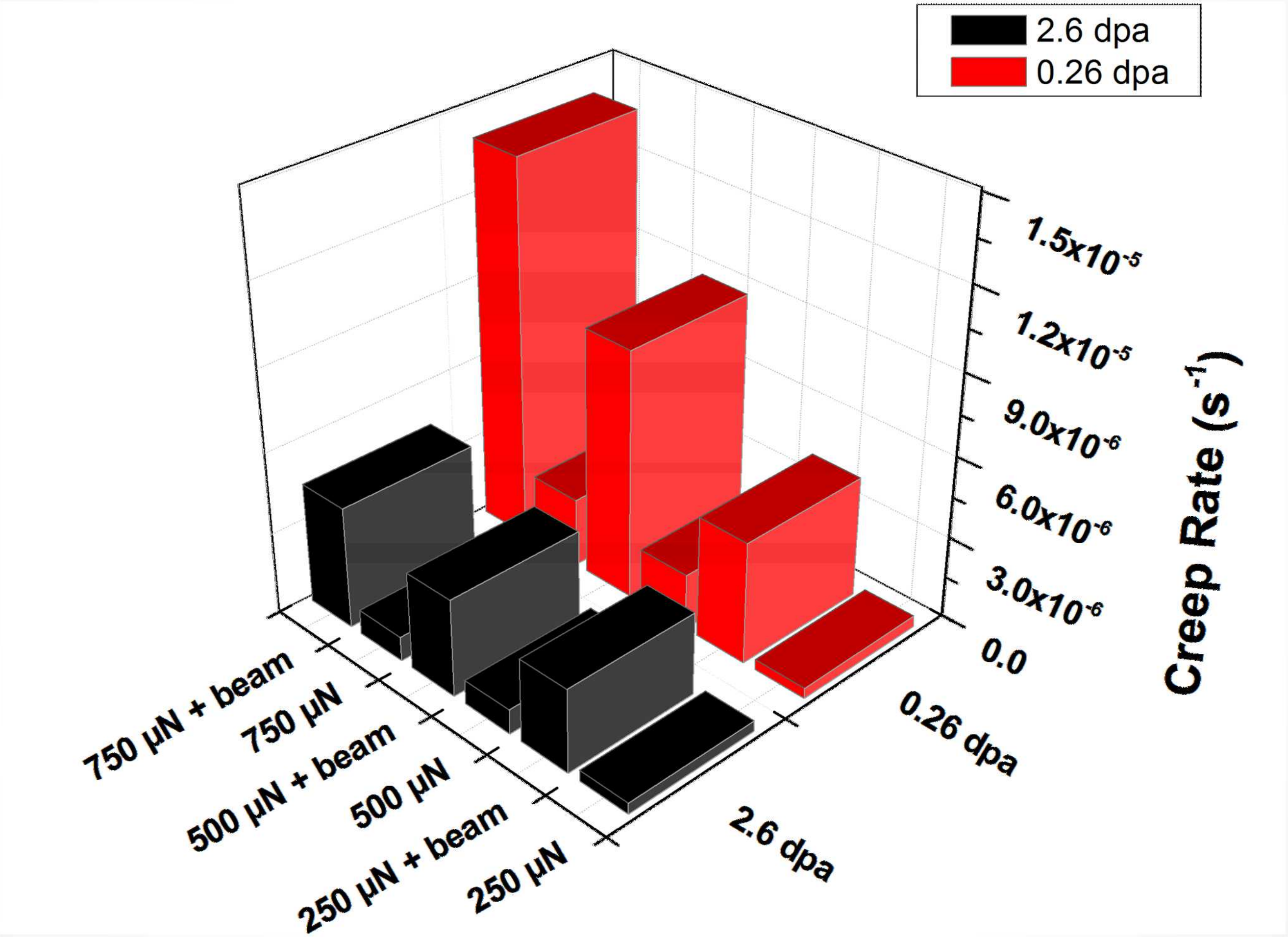
At a given load, creep proceeds substantially more quickly with beam on.

Creep Rates: 2.6 dpa



At a given load, creep proceeds substantially more quickly with beam on.

Creep Rate Comparison



Substantially higher creep rates in sample with less accumulated damage.