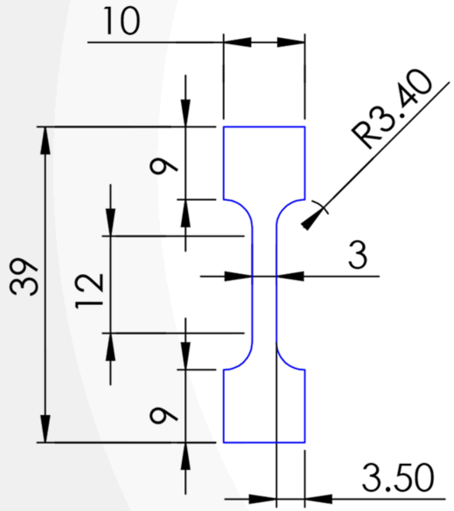
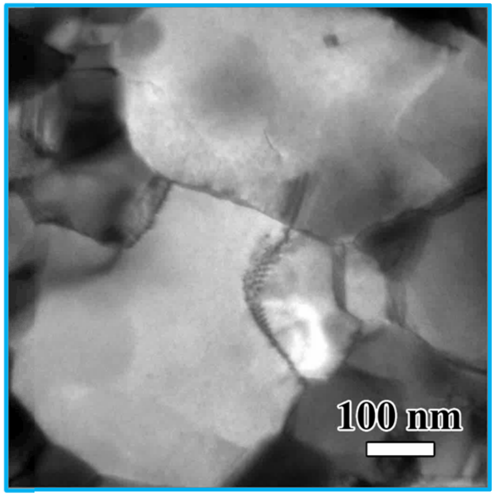
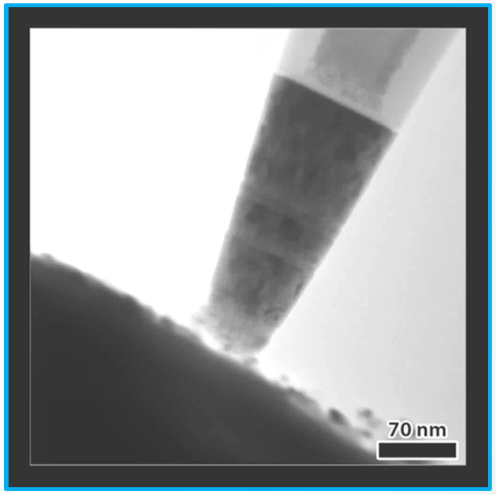
SAND2015-2193C

Combined Thermomechanical and Irradiation Testing of Materials







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2 April, 2015

Motivation

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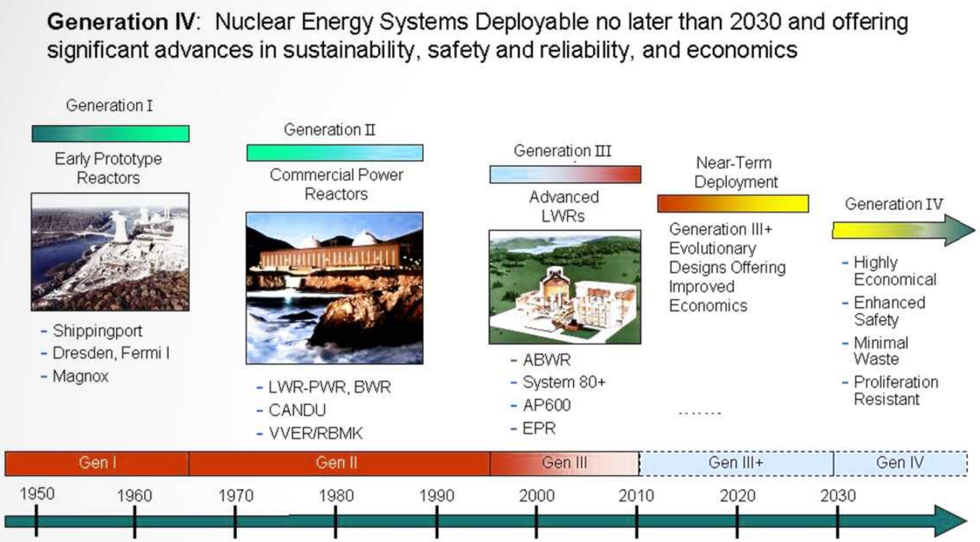


Argonne National Lab, *via* Wikimedia Commons.

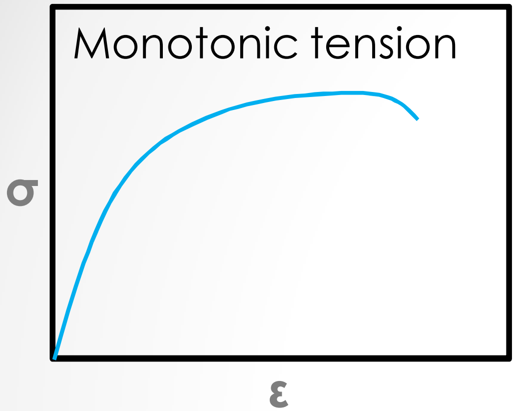
Future Reactors

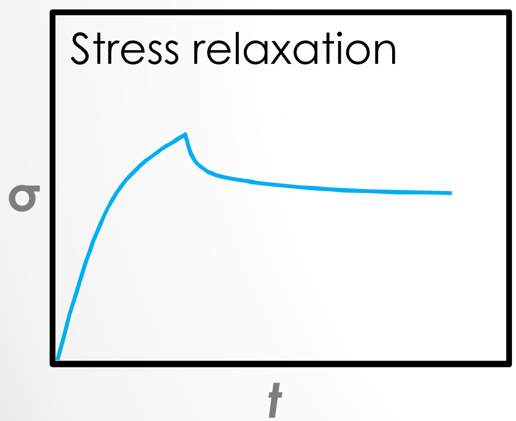
Aleš Buršic, *via* World-Nuclear-News.org

- Less data than ideal available for irradiation creep in new alloys
- Ion beams allow for rapid simulated testing

- **We want to perform experiments with simultaneous mechanical loading, specimen heating, and ion irradiation**

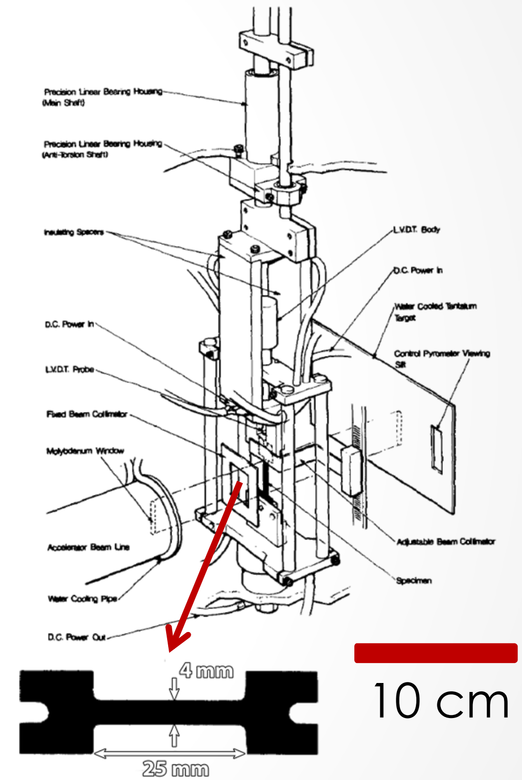
Tension Testing





- Lots of information:
  - *E*, σ_y, σ_{UT}, elongation, toughness, *n*, *m*
- More information:
  - *m*, ΔV, creep

- AERE Harwell, 1977

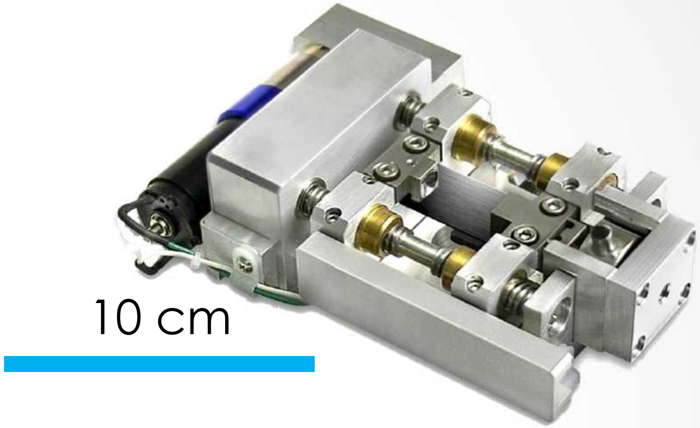


- **Gold standard for bulk mechanical properties**
  - **Nontrivial to implement in an end station**
- **Complementary *in situ* TEM experiments for microstructural mechanisms**

Hudson, *et al*, J Nucl Mater, 1977.

Major Components

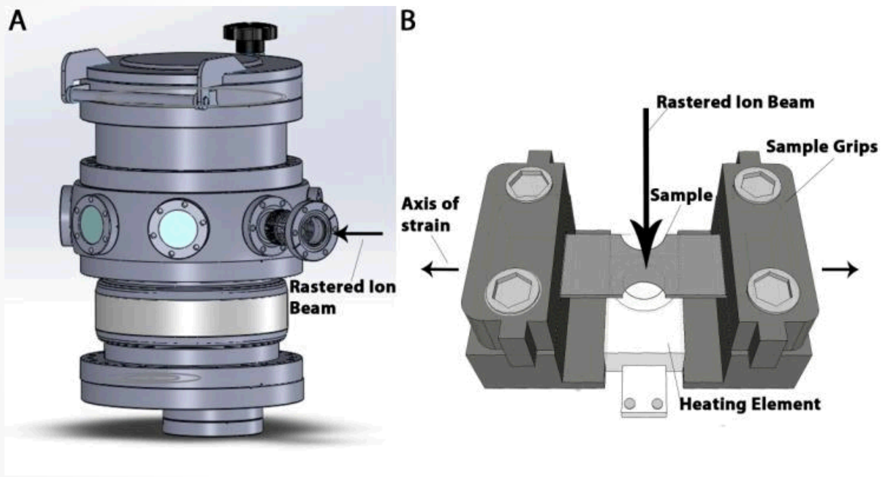
- Tension stage: MTI/Fullam SEMTester
  - 4500 and 450 N load cells
    - ±0.2% full scale sensitivity
  - Ambient to 1200 °C

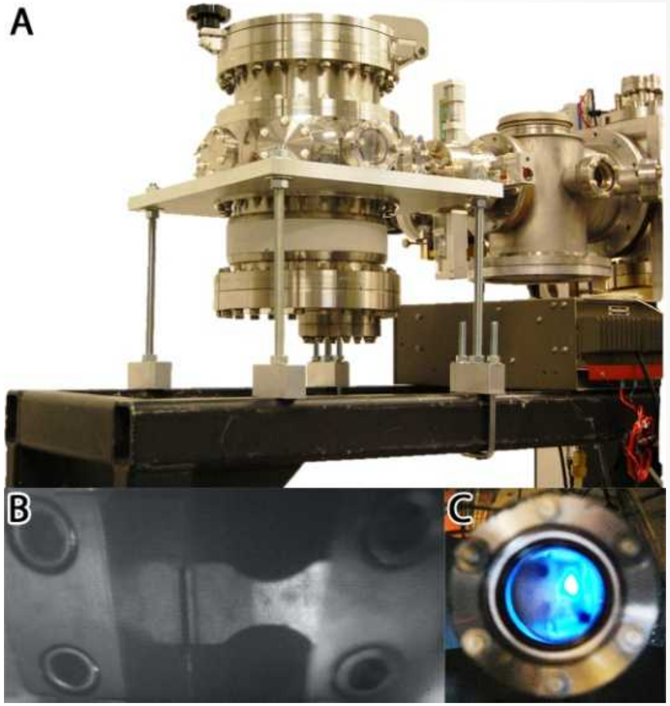


- Accelerator: HVE EN Tandem
  - 0.8 to 6 MV terminal voltage
  - SNICS, Alphatross, Hiconex 834 sputter, and duoplasmatron ion sources

- Array of beamline components

Design and Construction

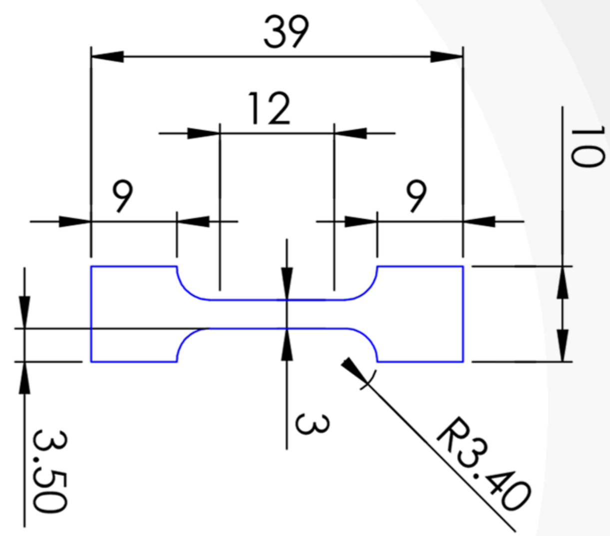




- Components created and assembled in SolidWorks prior to actual construction
  - Assured fit of components and alignment of the beam
- Viewports for beam alignment and sample monitoring

Sample Considerations

1. Beam transparency
2. Stage compatibility
   - Load/extension limits
3. Standard geometry
   - Based on ASTM-E8
   - Literature comparison
4. Proton activation concerns

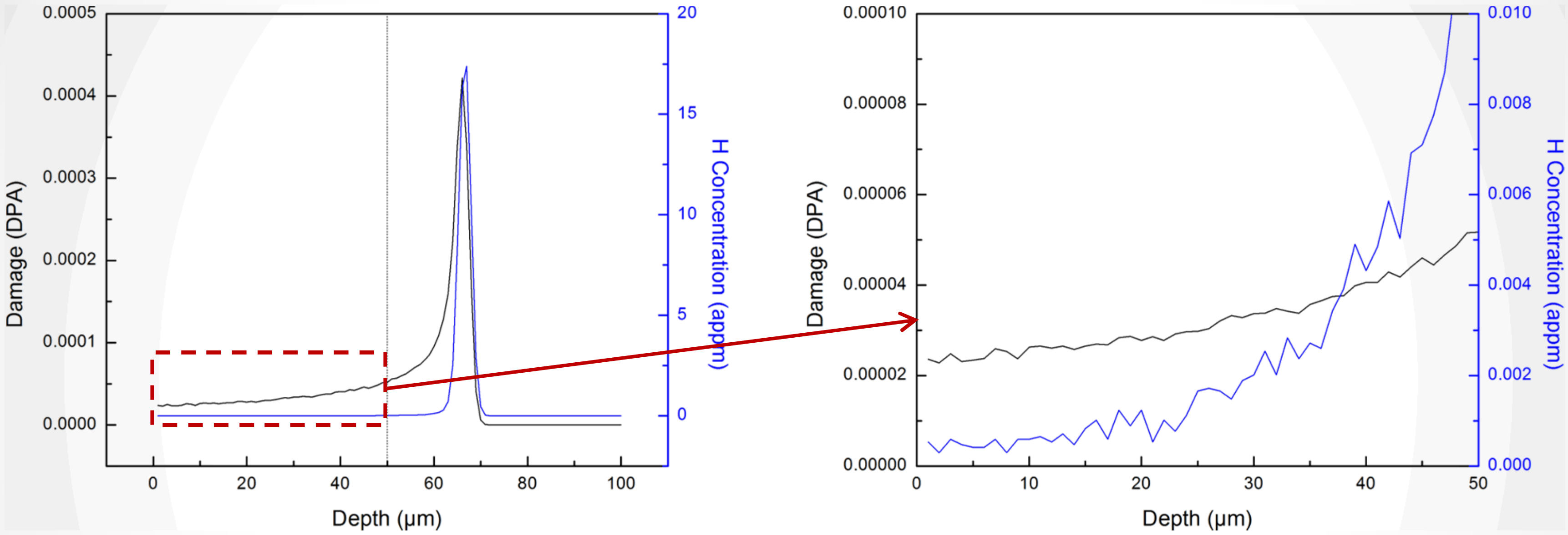


Can apply up to 30 GPa stress and accommodate 58% strain

Commercial OFHC Cu used as a model system

- **50 and 100 μm thicknesses**

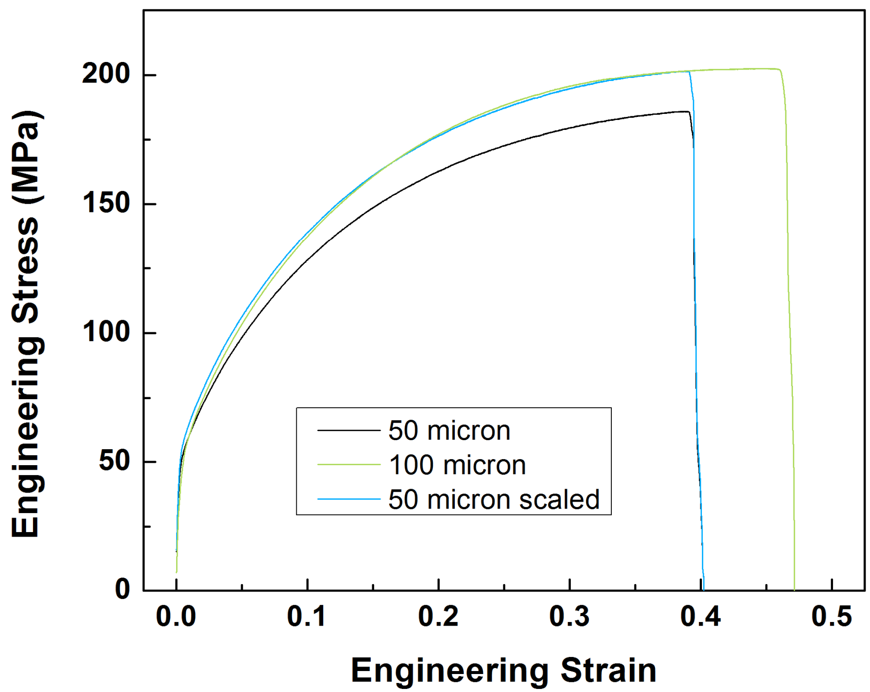
SRIM Damage Estimation



- 4.5 MeV H^+ into 50 μm thick Cu
  - Limited for radiation safety, not by the accelerator capability
- 2.1×10^{11} ions $cm^{-2}s^{-1}$ ➔ 1.4×10^{-8} mean DPA s^{-1}
- Ion energy and type can be changed to vary damage rate and type, with consideration for film thickness and density

Basic Tension Testing

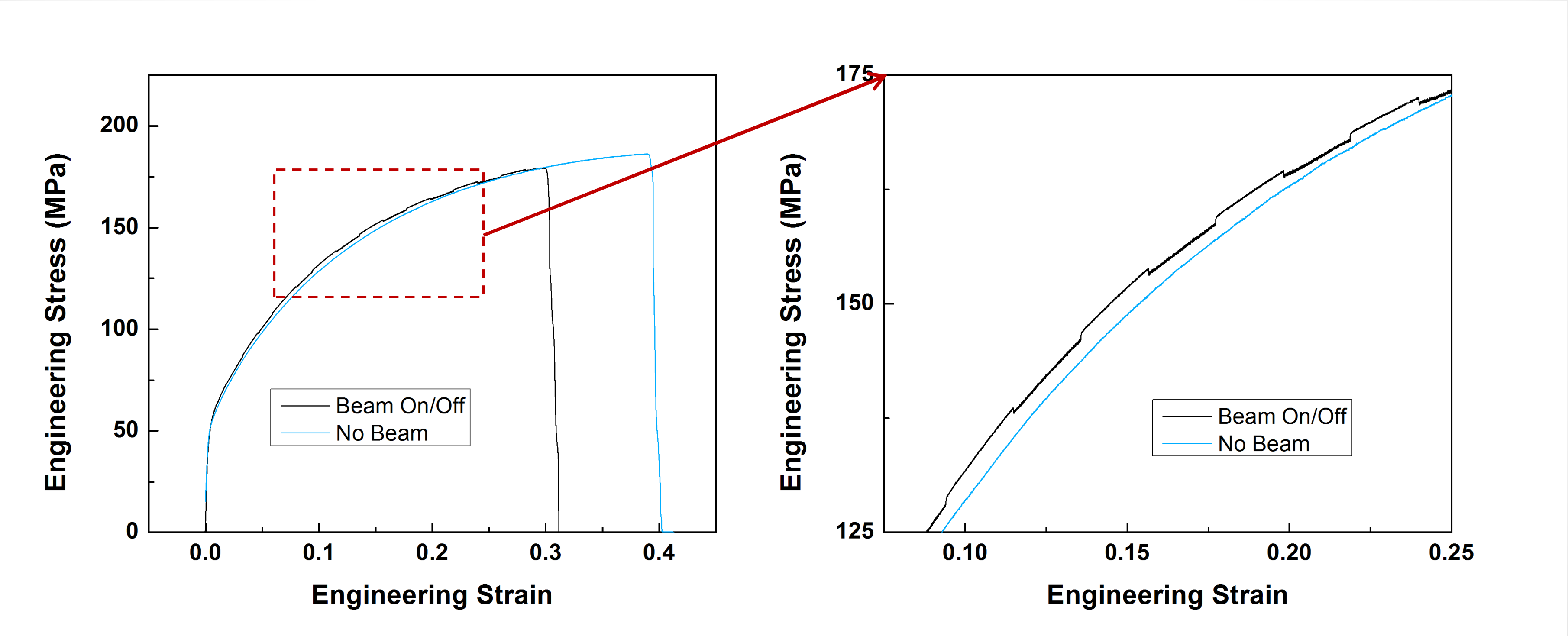
- 0.25mm/min elongation rate (initial engineering strain rate $2.1 \times 10^{-2}\ s^{-1}$)
  - ~17 minute tests



- **Reproducible results and reasonable strain**
- **In agreement with bulk annealed Cu (70 MPa yield, 220 MPa UTS)**

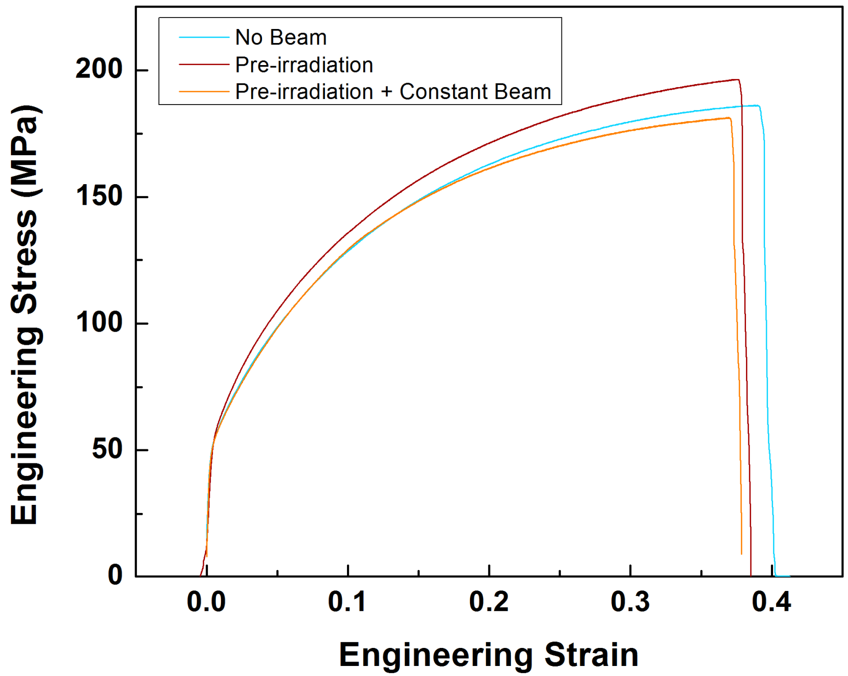
Tension + Irradiation

- Similar elongation rate
- Beam cycled on/off at 60 s intervals



- **Measured temperature rise of ~0.25 °C when beam is turned on**
- **Small but measureable change in stress-strain curve when beam cycled on/off**

Tension + Irradiation

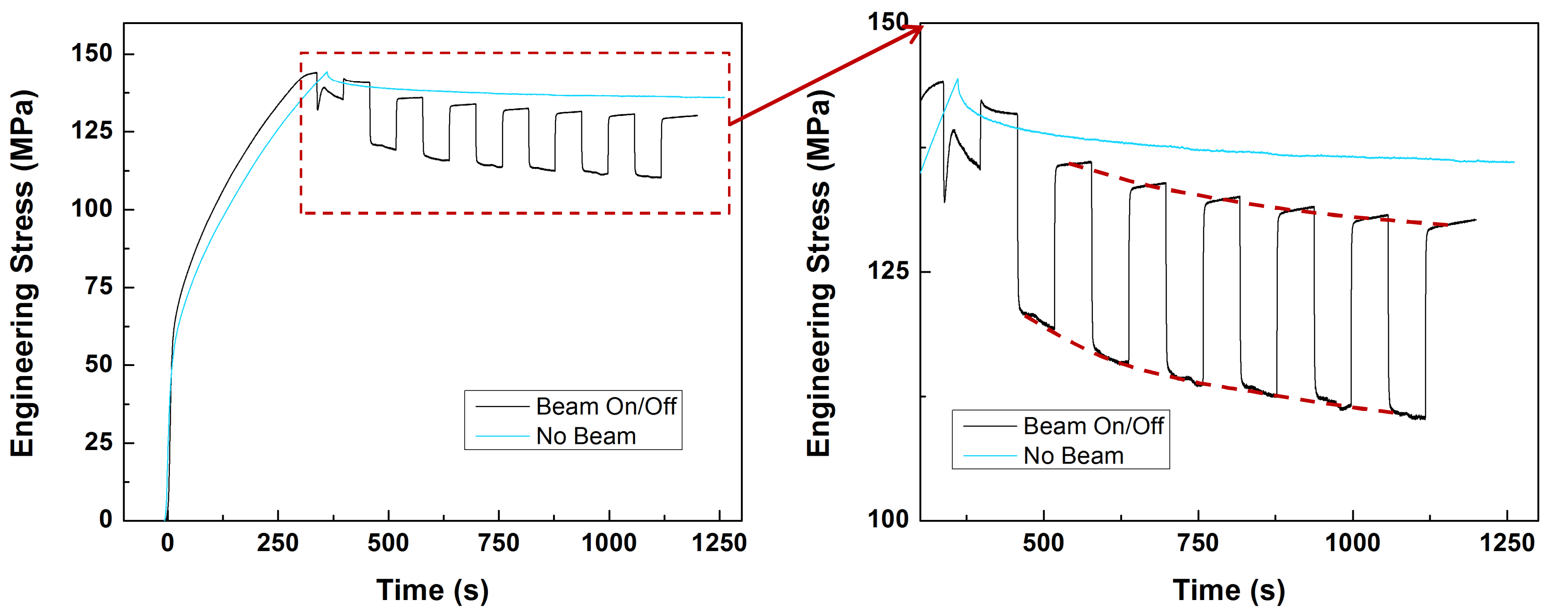


- Pre-irradiation to 6 × 10^{14} ions cm^{-2} (~4 × 10^{-5} DPA)
- Pre-irradiation followed by irradiation during test (additional ~1.3 × 10^{-5} DPA)
- Minimal change in elongation to failure

- **Overall strengthening after pre-irradiation to 4-5 × 10^{-5} DPA**
- **Effects of continuous irradiation during the test are not yet clear**

Stress Relaxation

- Similar 0.25mm/min elongation rate to 22.5 N load
  - Approximately 75% of typical ultimate tensile load
  - 900 s hold at constant position
  - Beam cycled on/off at 60 s intervals

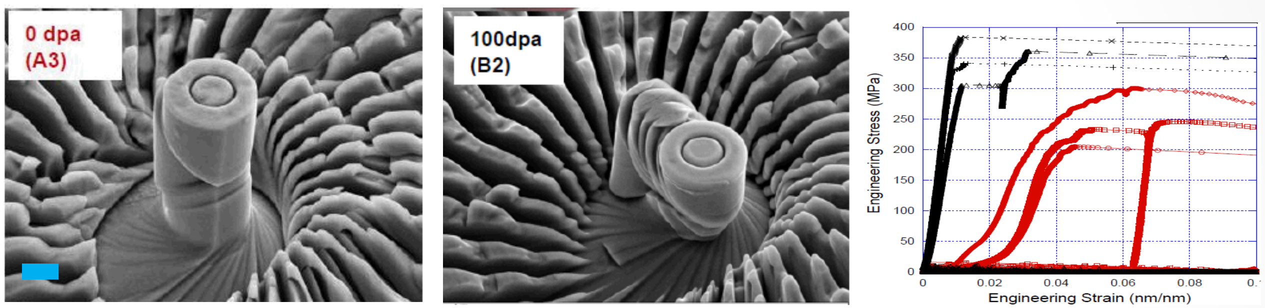


- **Offset likely an artifact**
- **Different relaxation rates with beam on and off**

Micro- and Nanoscale Testing

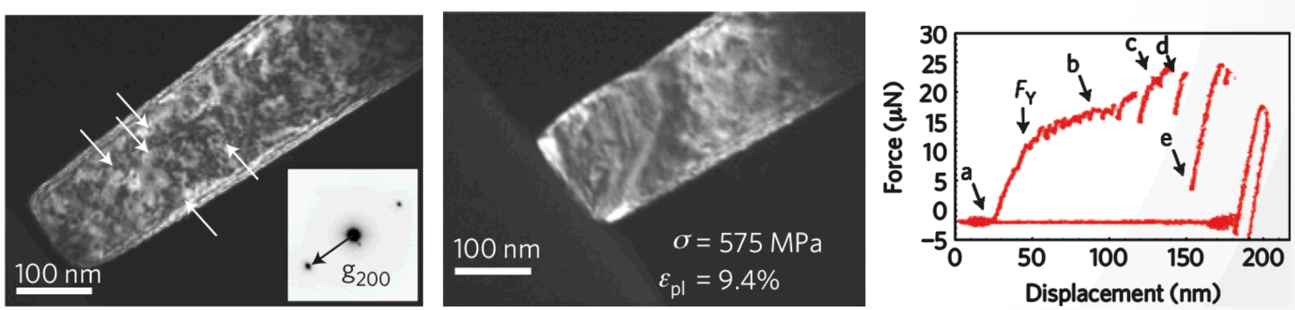
- Wealth of small-scale mechanical testing methods developed in last decade
- Recent experiments with radiation-damaged materials

30 MeV Cu^{5+}
100 DPA



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

1.1 MeV H^{+}
0.8 DPA



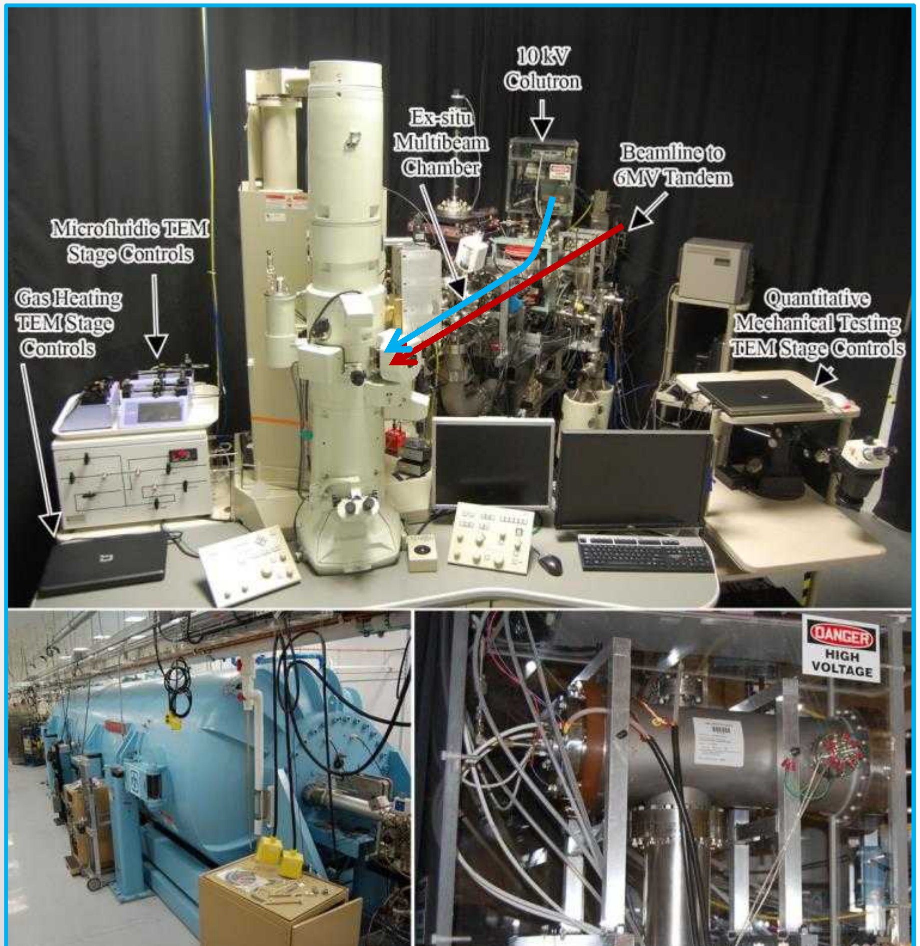
Kiener, *et al*, Nat Mater, 2011.

- **Combining these methods with *in situ* irradiation**

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

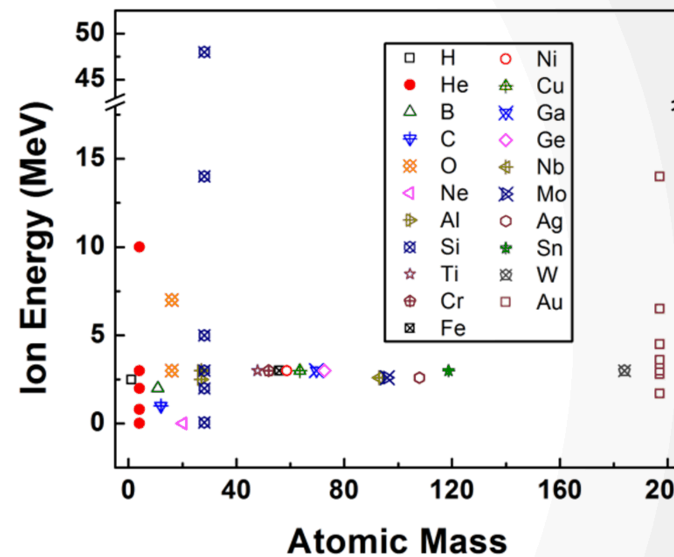
Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem



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

Ion species & energy introduced into the TEM

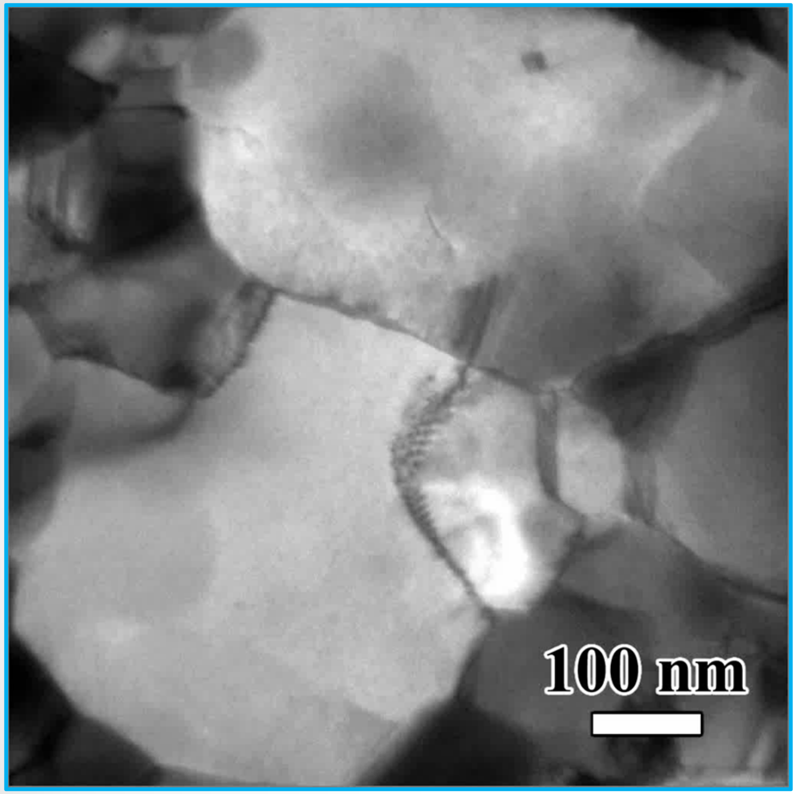


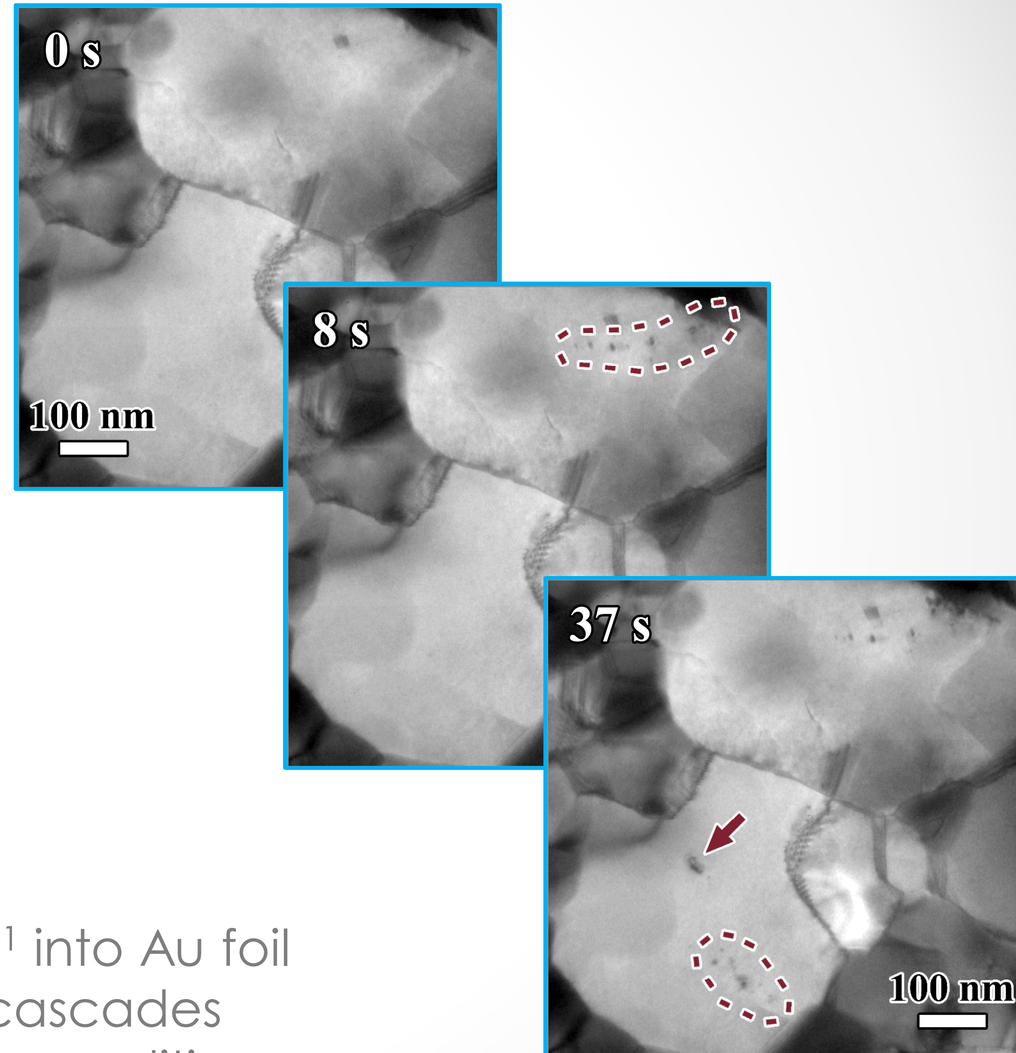
Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution.

Similar beams can be directed to the TEM and end stations.

In Situ Irradiation: 3.6 MeV Au^{6+}

Video speed ×5.

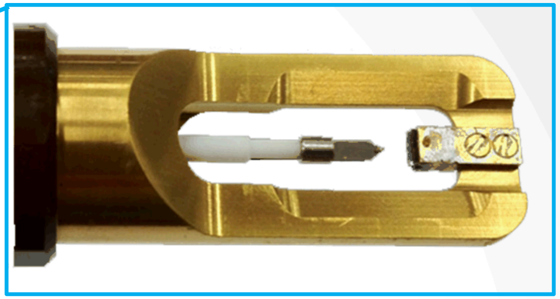




- Au^{6+} at 2.1 × 10^8 ions cm^{-2} s^{-1} into Au foil
- Large defect clusters from cascades
- Sample stability under these conditions

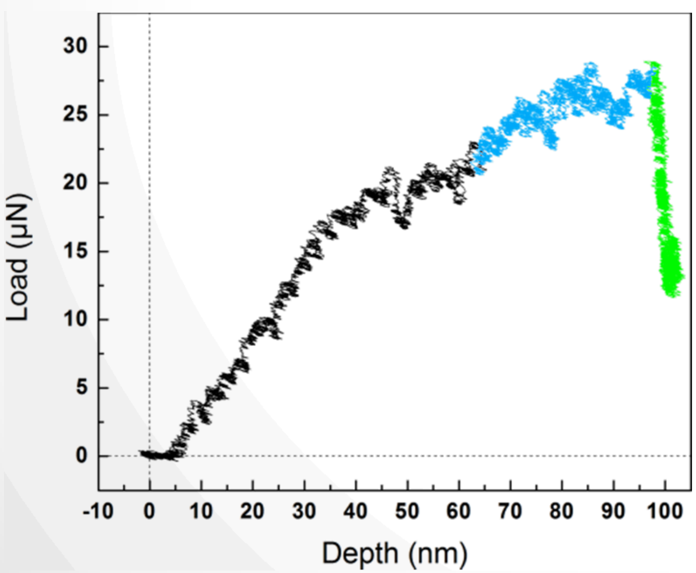
Hattar, *et al*, Nucl Instr Meth Phys Res B, 2014.

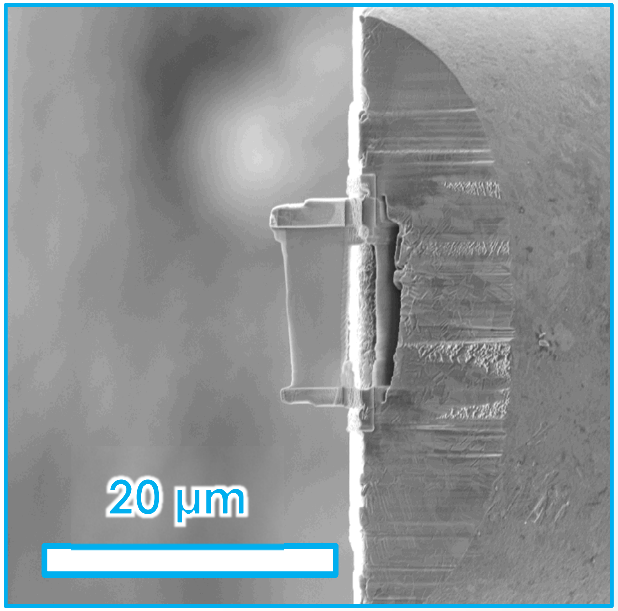
In Situ TEM Nanoindentation



Hysitron PI95 *In Situ* Nanoindentation TEM Holder

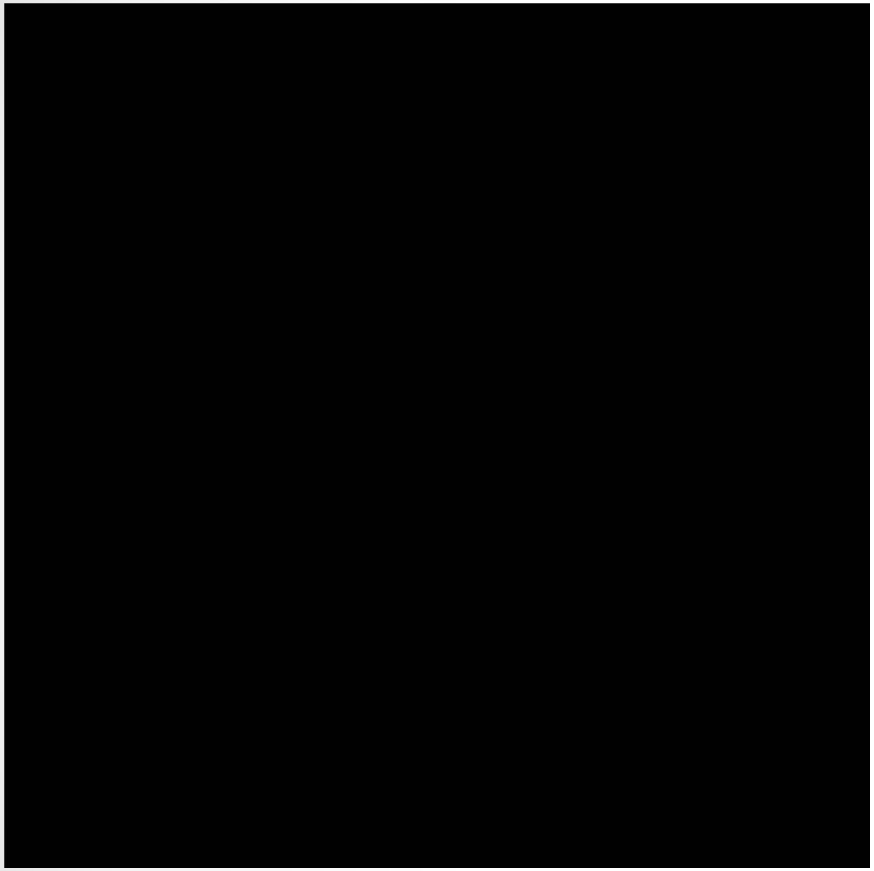
- Sub nanometer displacement resolution
- Quantitative force information with µN resolution
- Concurrent real-time imaging by TEM



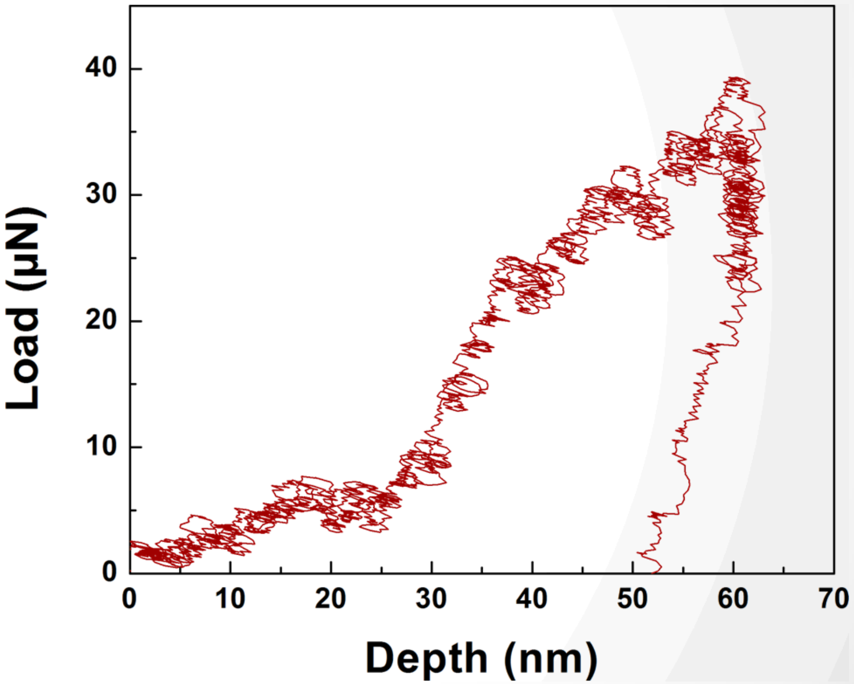


FIB-lift out from the same Cu stock material

In Situ TEM Nanoindentation



- FIB window from PLD Ni film
- Single indentation to 60 nm



- **Able to sequentially irradiate and perform indentations**
- **Investigating feasibility of concurrent irradiation + deformation**

Quantifying Radiation-Induced Microstructural Change

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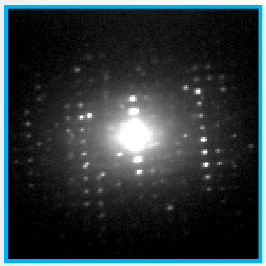
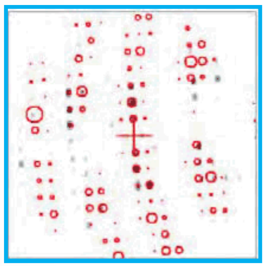
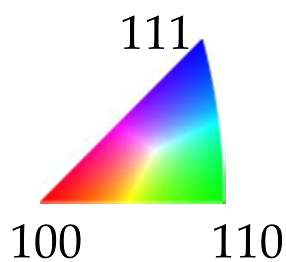
- Automated crystallographic orientation mapping
  - EBSD-like maps from the TEM

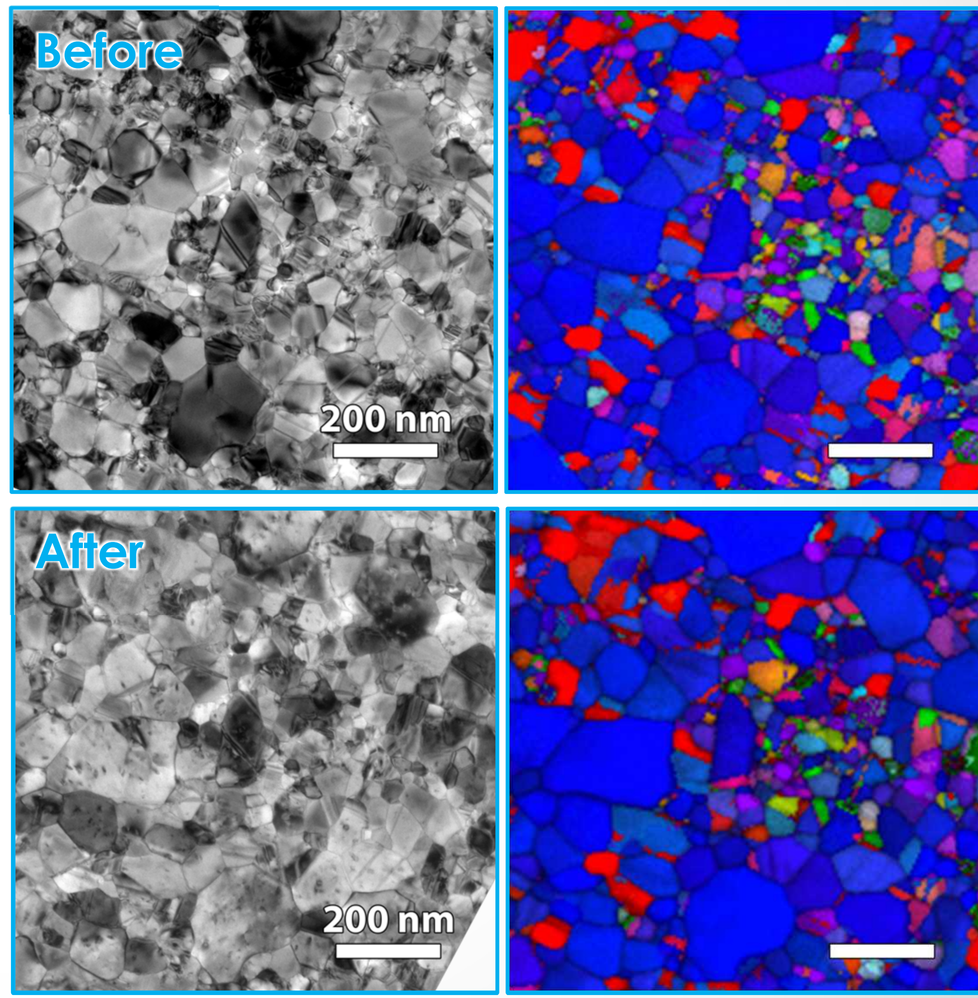






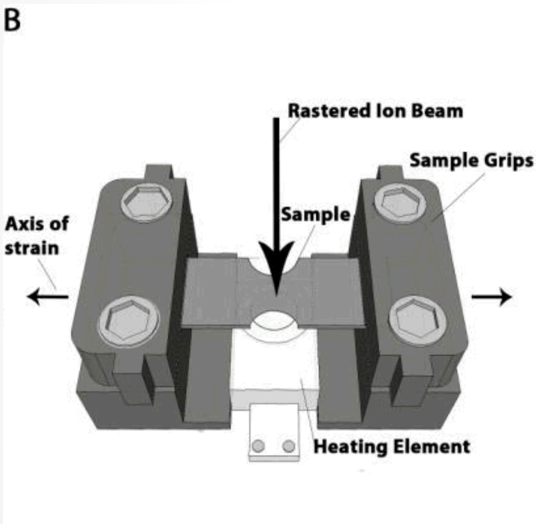
- Less sensitivity to strain, diffraction, and defect contrast
- Established data analysis tools
  - Grain size
  - Grain boundary character
  - Pole figures

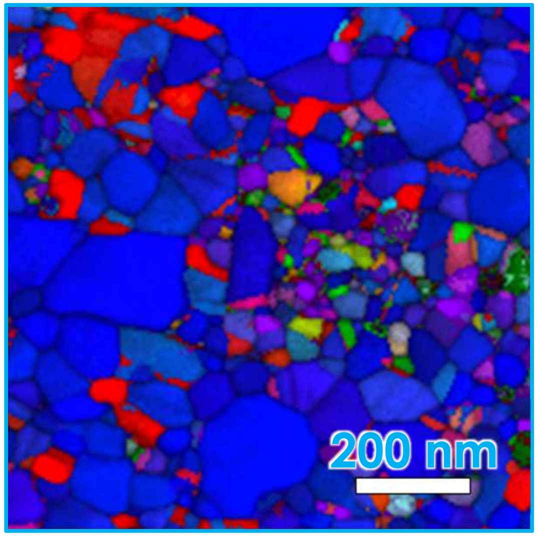
nc Au before and after xx × 10^y 10 MeV Si^{3+} cm^{-2}

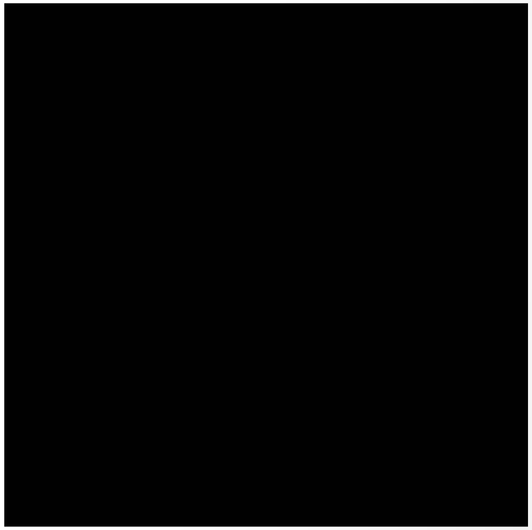


Summary and Conclusions

- Successfully integrated a tensile load frame into an end station
  - Proof-of-concept demonstrations with Cu specimens and 4.5 MeV H^+
- Developing *in situ* TEM methods with combined irradiation + deformation







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