Correlating Grain Orientation and Grain Boundary Character to the Failure Path in Nanocrystalline Metals

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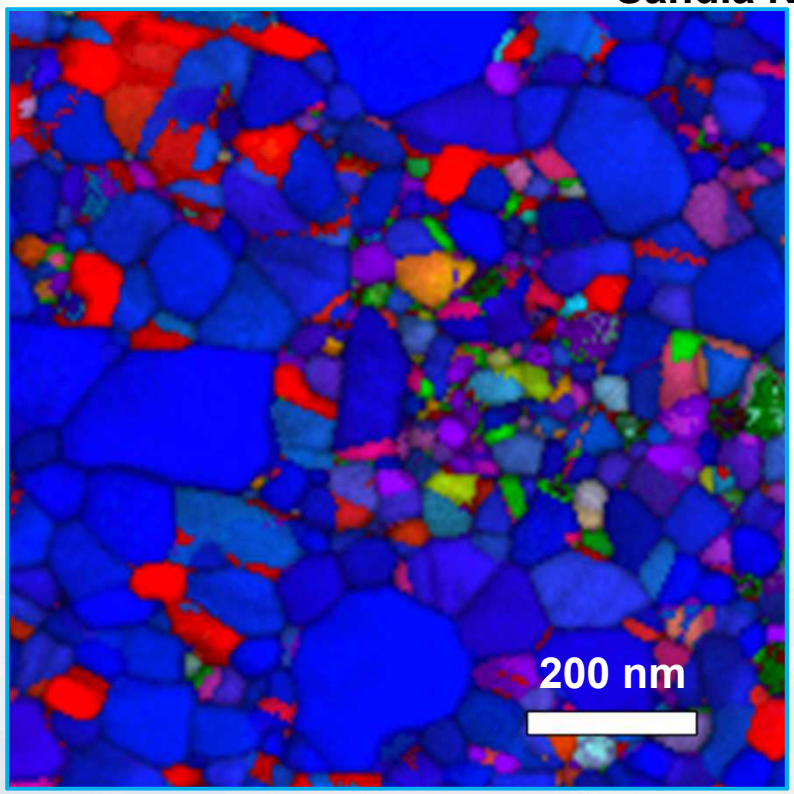

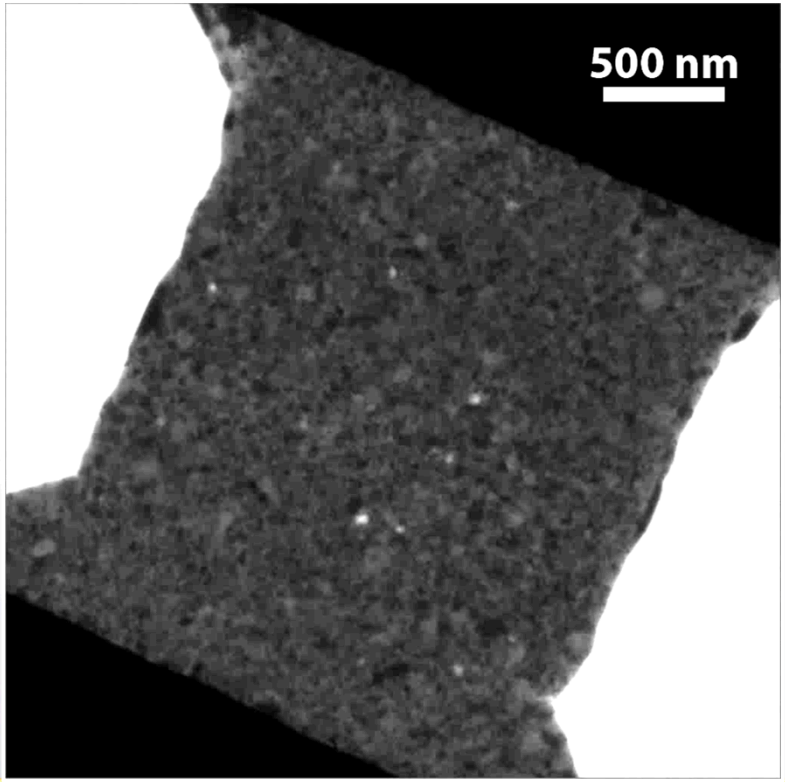

Preliminary work to experimentally correlate the grain boundary stability with local grain orientation or grain boundary character in cyclic loading or radiation environments.

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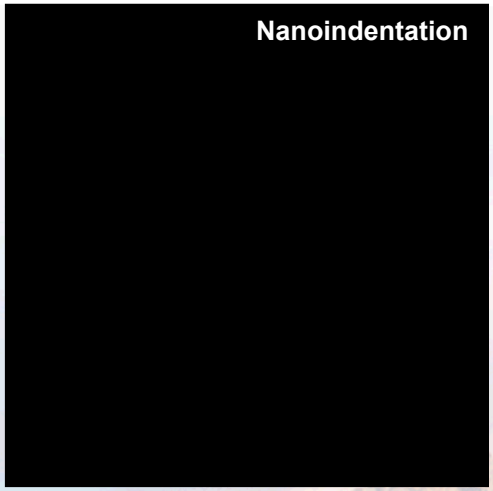
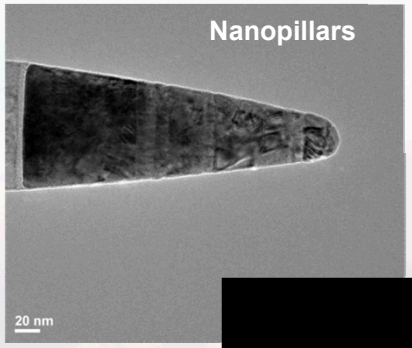
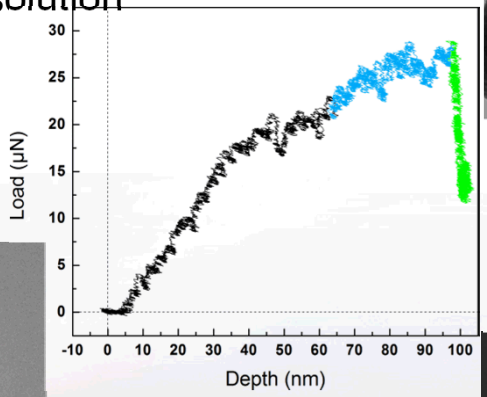
In situ Quantitative Mechanical Testing

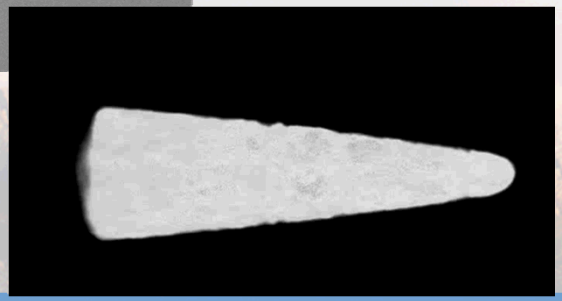
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

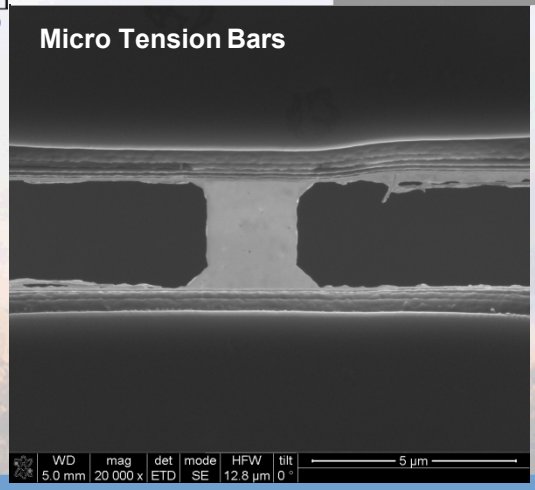
- Sub nanometer displacement resolution
- Quantitative force information with μN resolution
- **Concurrent real-time imaging by TEM**





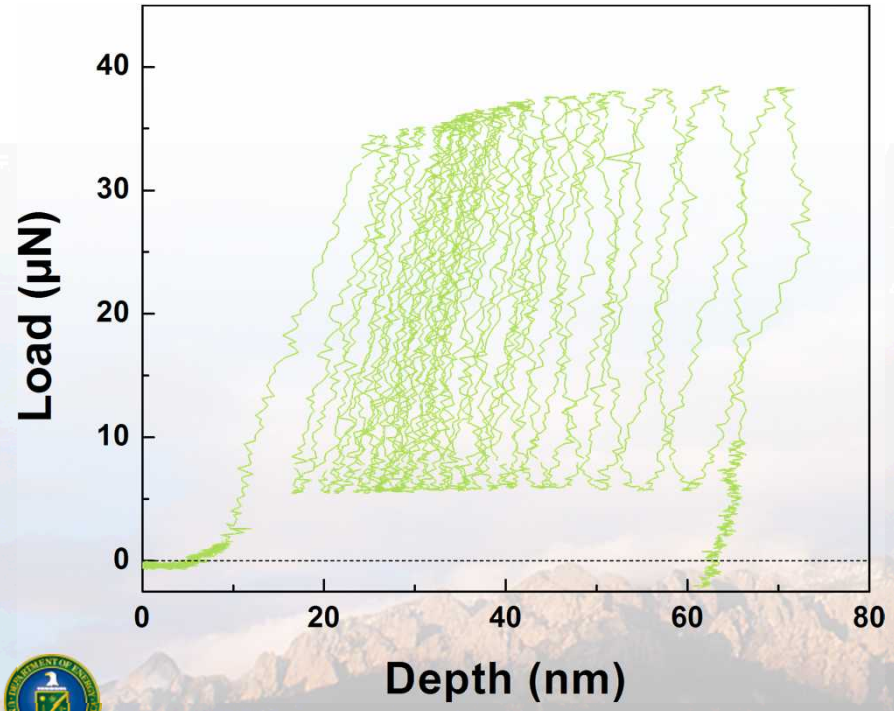


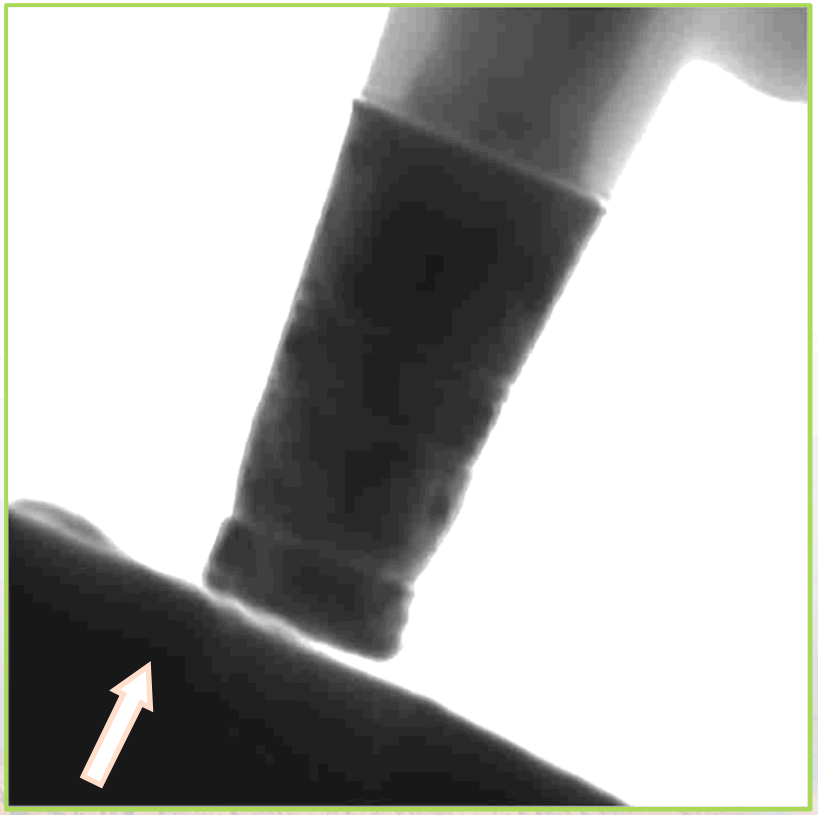




Cyclic Pillar Compression

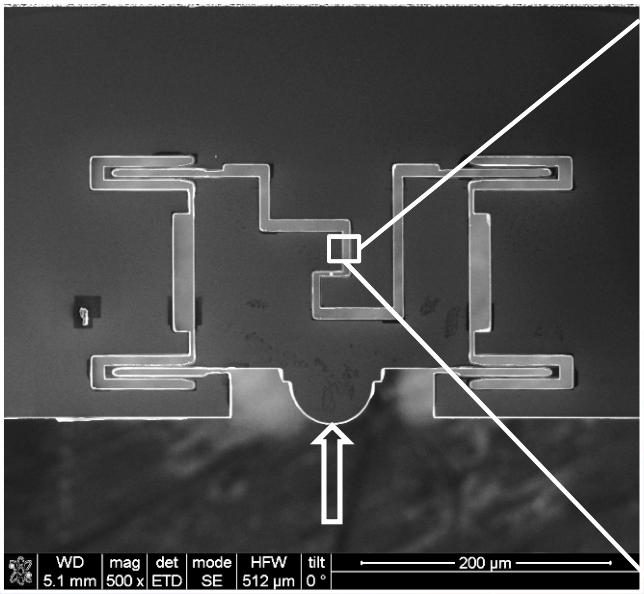
- **Cyclic loading with increasing force amplitude**
  - After a previous monotonic compression
  - 23 cycles to failure
- **Failure initiated at notches due to fabrication defects**

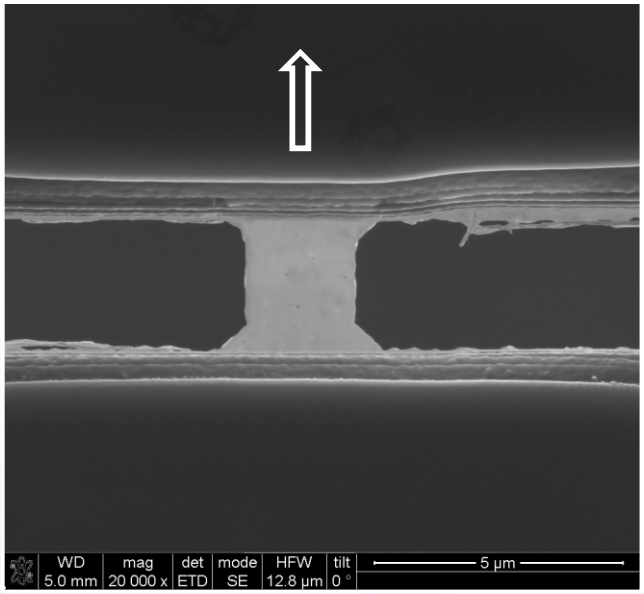


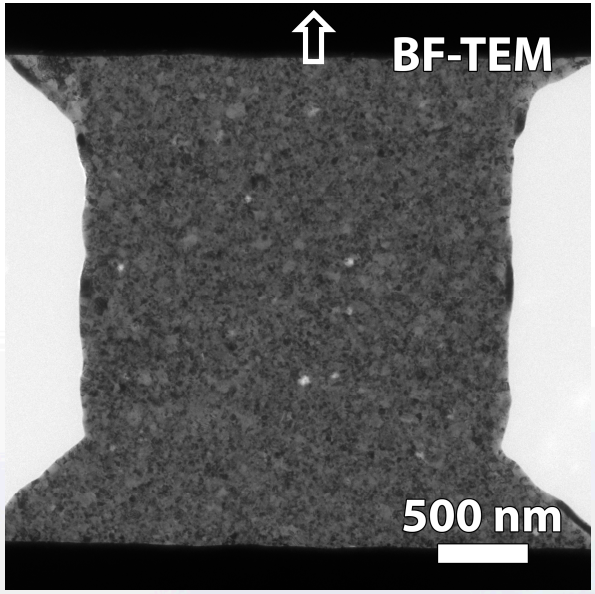


Tension Specimen Fabrication

- **Hysitron "Push-to-Pull" devices**
  - **Microfabricated Si test frame**
  - **Cu film (75 nm) floated onto device, then FIB milled**



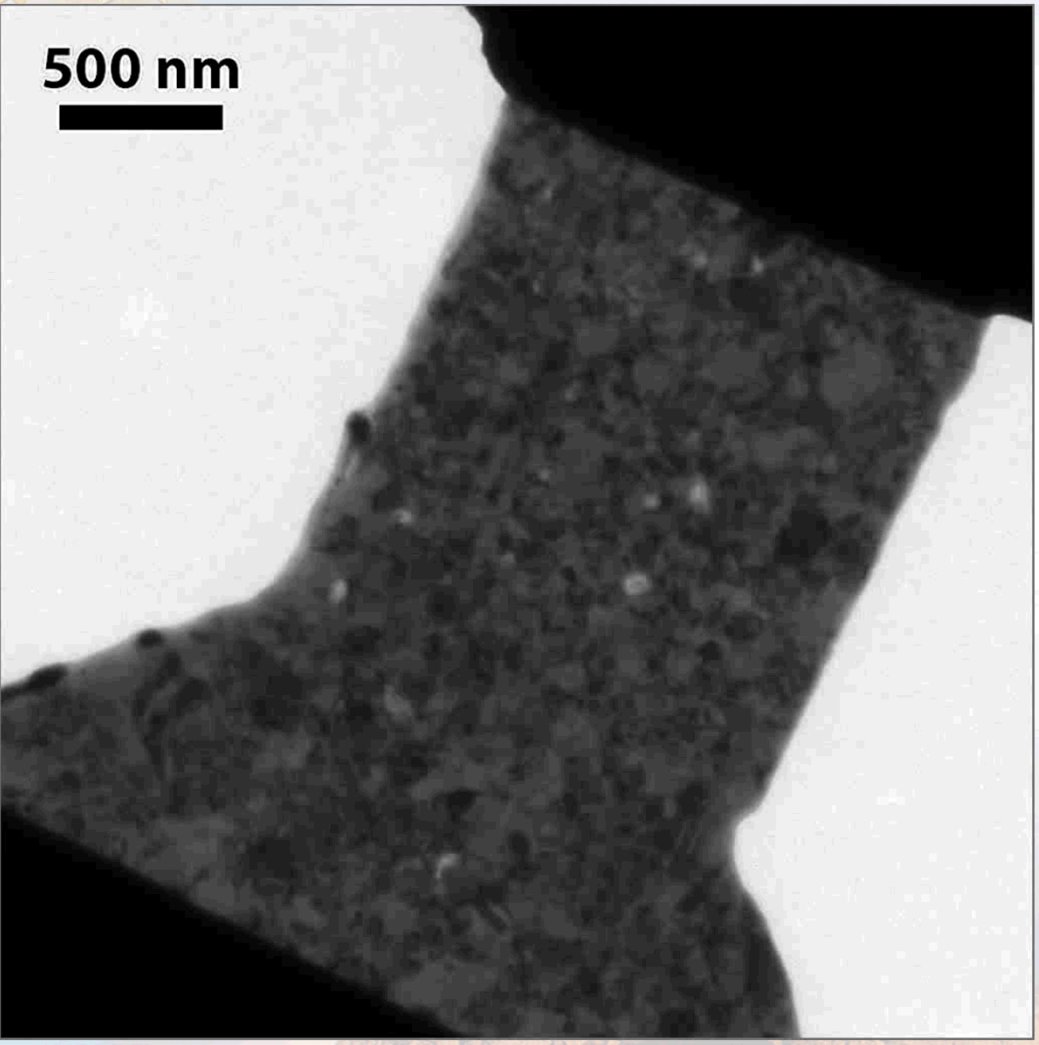


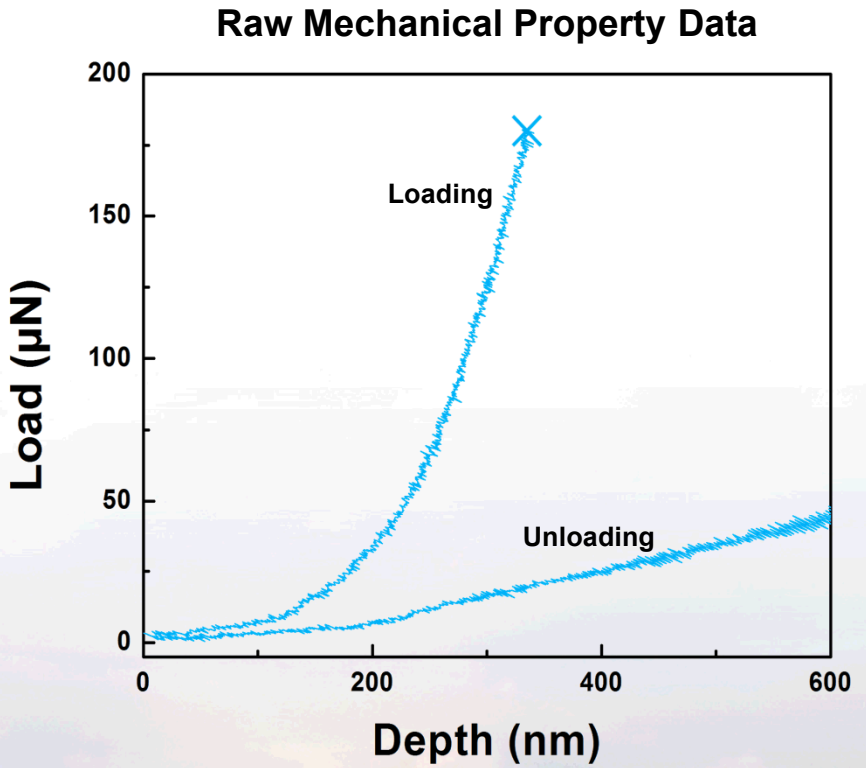


- **Nearly pure tension, uniform cross sectional area, stable load frame**
- **Sensitive to shape of edges, issues with magnetic materials**

In situ TEM Monotonic Tension Testing

Video playback × 0.5

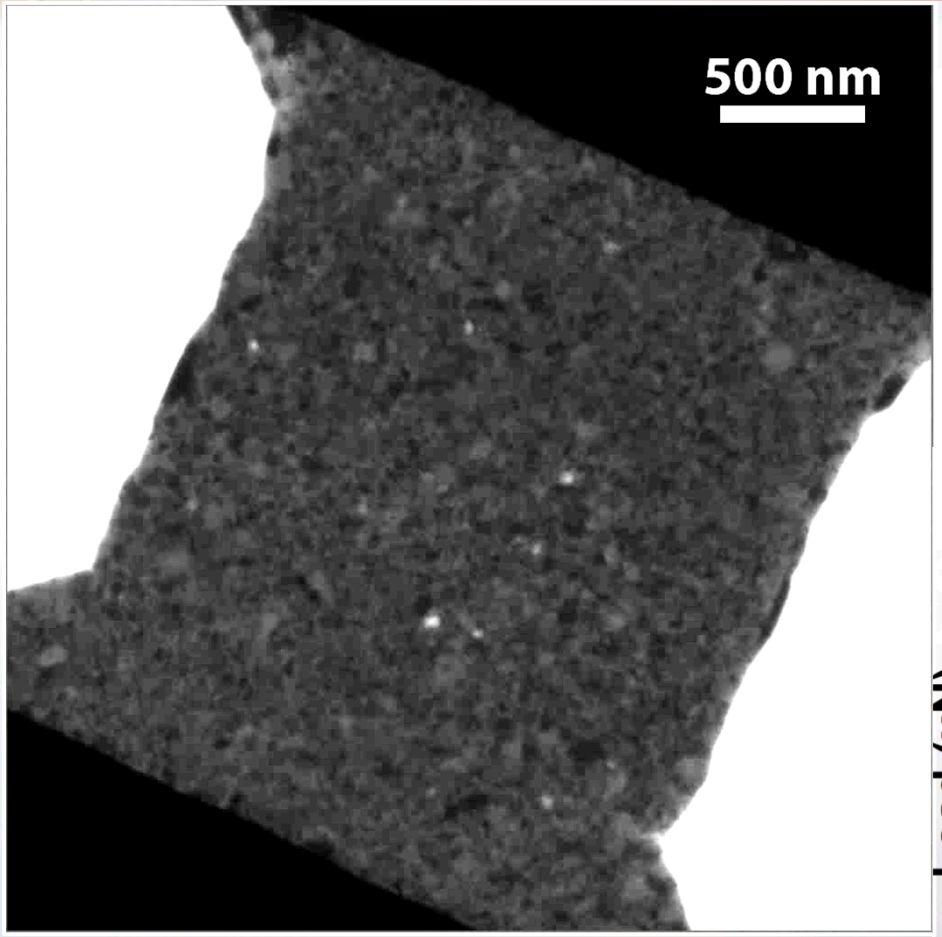




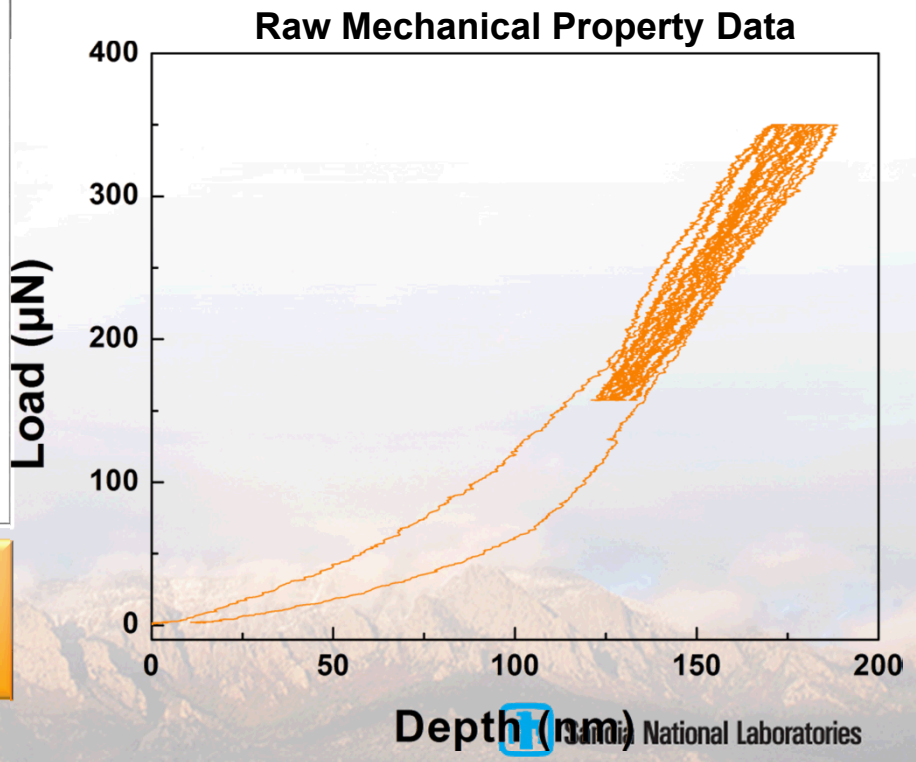
In situ TEM Cyclic Tension Testing

Video playback ×10

Collaborator: D. Stauffer



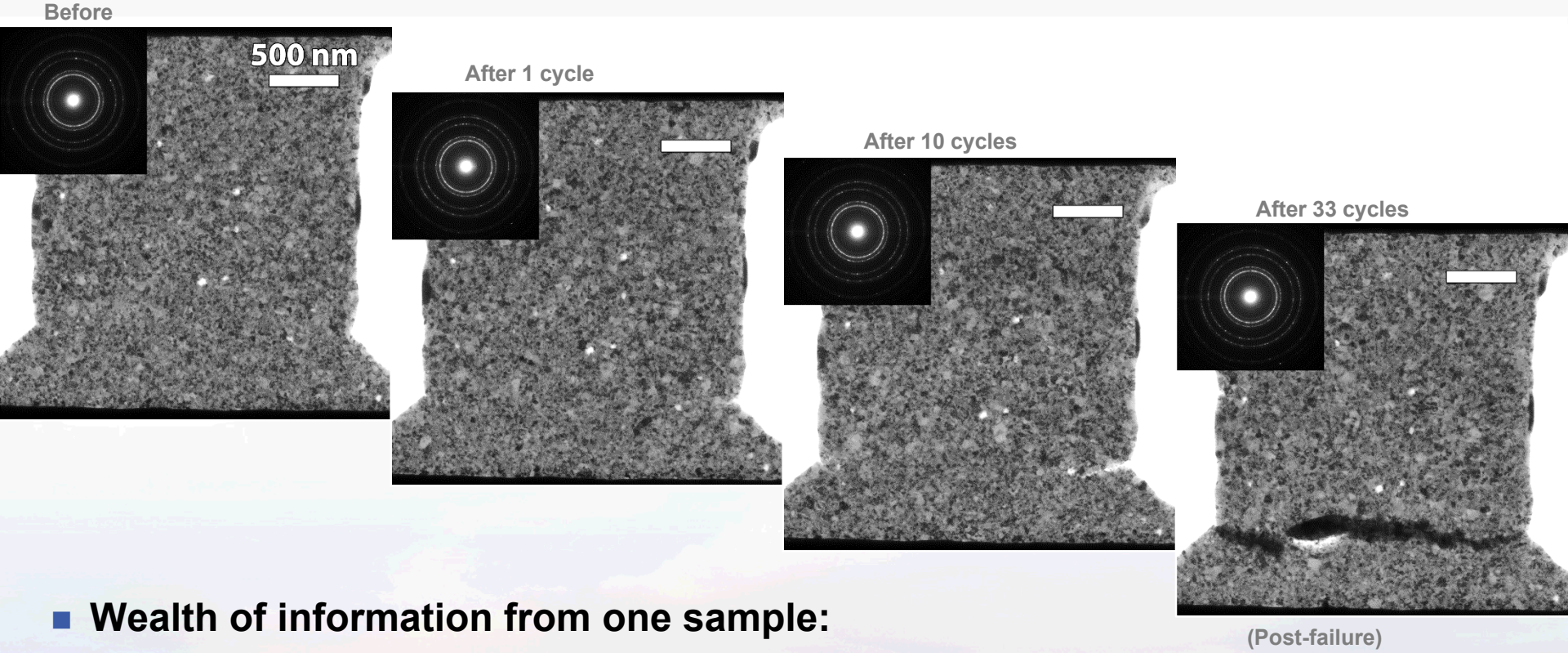
- **Cyclic loading:**
  - Crack initiated in previous monotonic test
  - 9 cycles to ≈87.5% of that load
  - 50% unloading
  - Slow crack propagation



- **Slow crack propagation**
- **Evidence of grain growth**

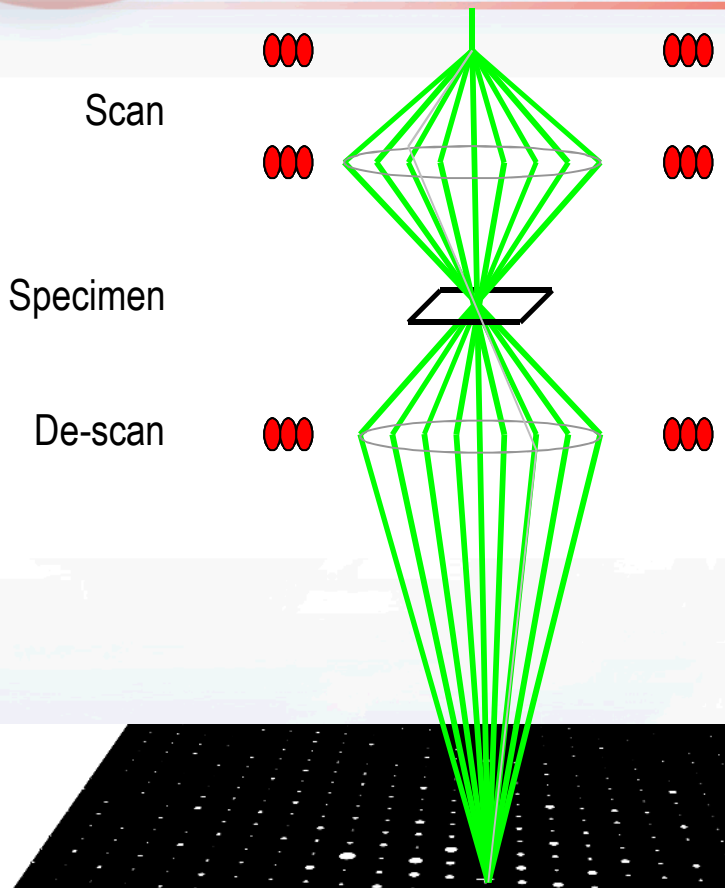
Cyclic Tension *In Situ*

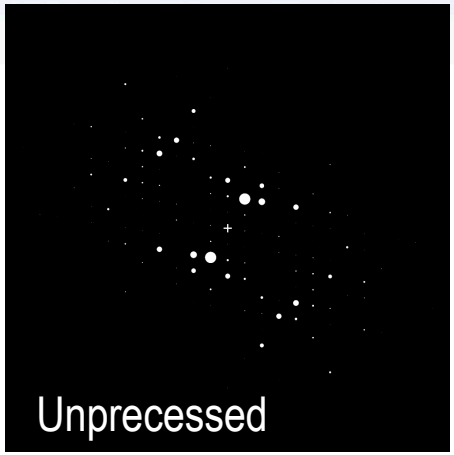
Collaborator: D. Stauffer

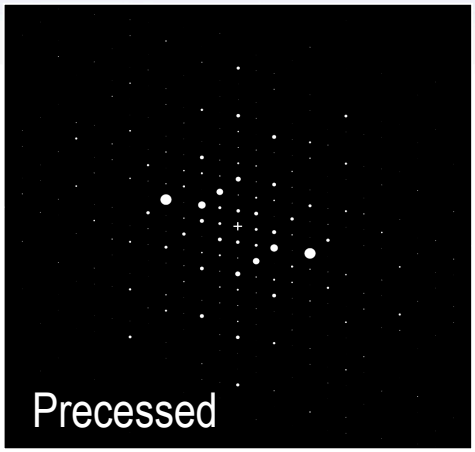


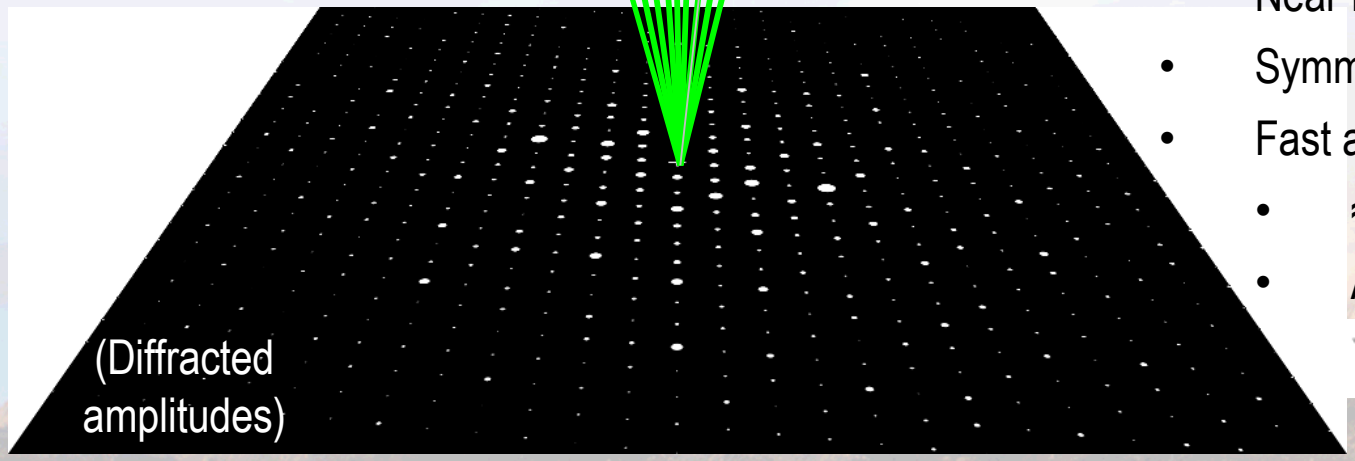
- **Wealth of information from one sample:**
  - Images and electron diffraction at each stage
  - Video and force/displacement during load cycles
- **Microstructural change still elusive**
  - Difficult to confirm and quantify

Precession Electron Diffraction Microscopy









Advantages:

- < 10 nm spatial resolution
- Near kinematical electron diffraction
- Symmetry ambiguities are resolved
- Fast and automated acquisition
  - ≈200 grains in 15 min.
  - Angular resolution ≈1°

App Five

NanoMEGAS
Advanced Tools for electron diffraction

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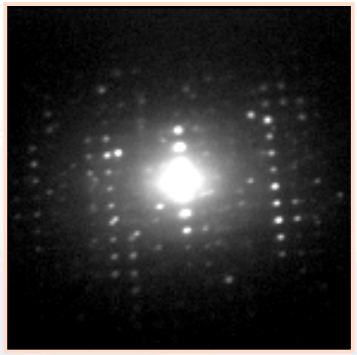
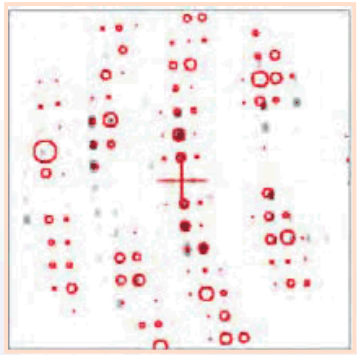
Quantifying Microstructural Change

- **Combining orientation mapping with deformation**
- **EBSD-like capability in the TEM**
  - Powerful analytical tools available





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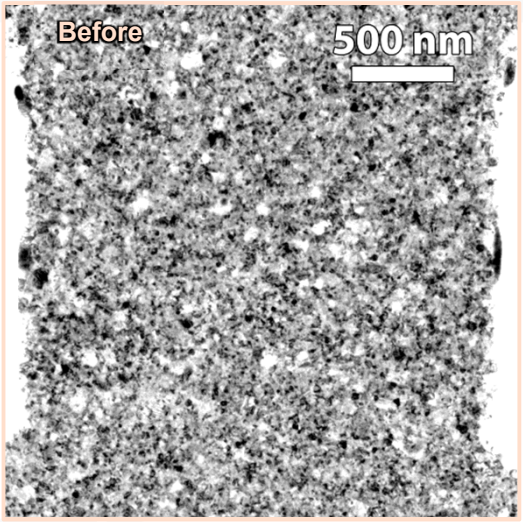

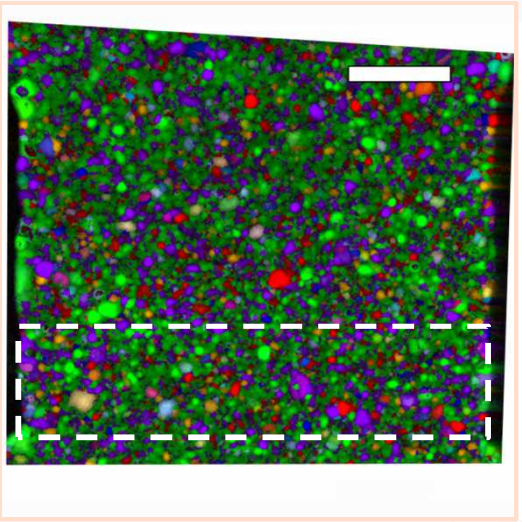

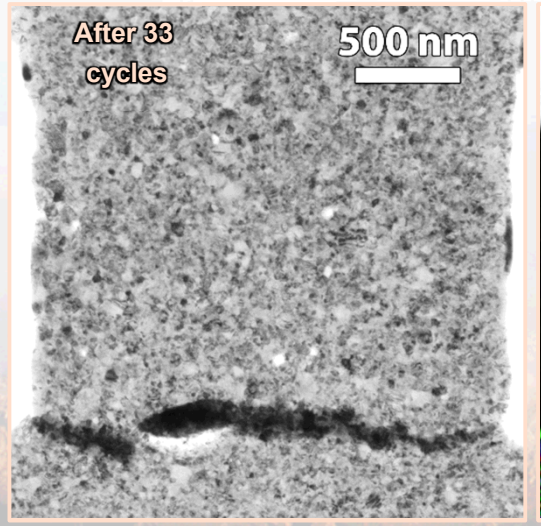

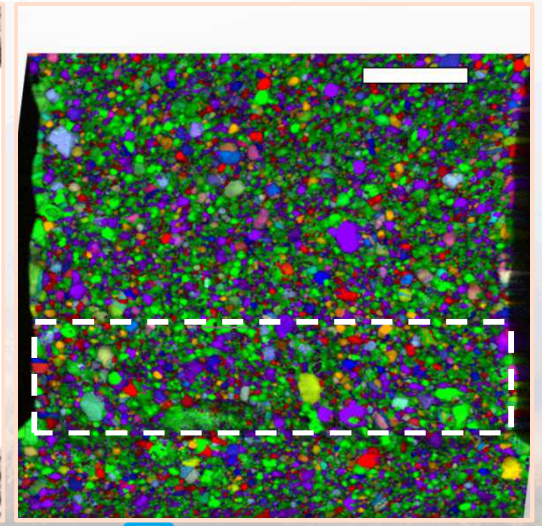

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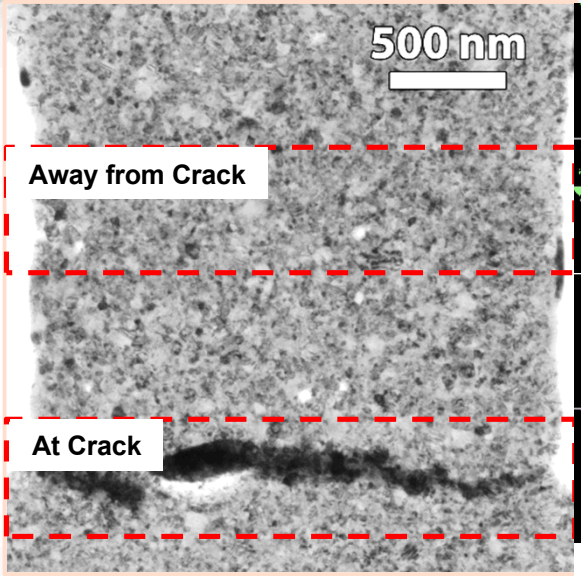
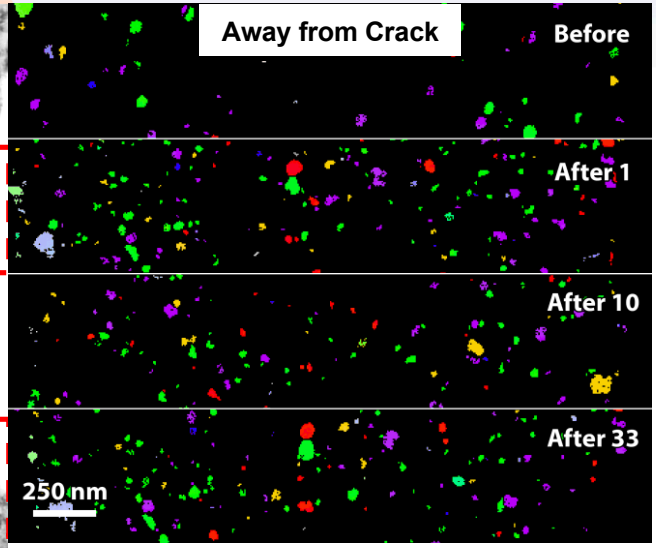
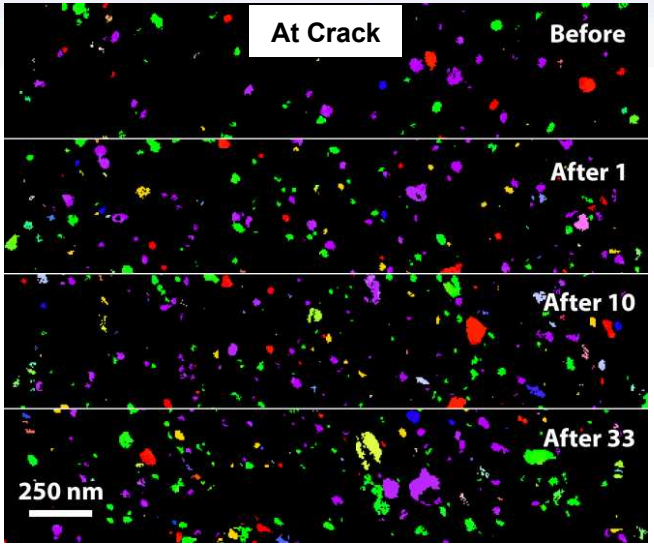
Quantifying Microstructural Change

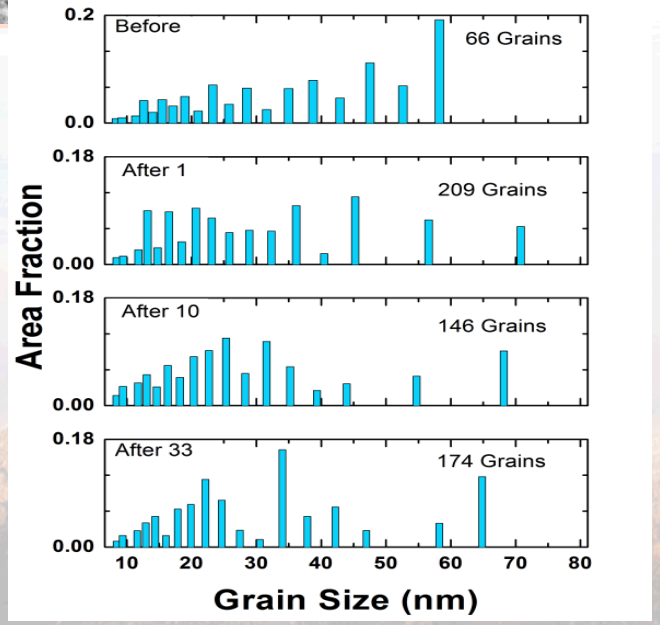


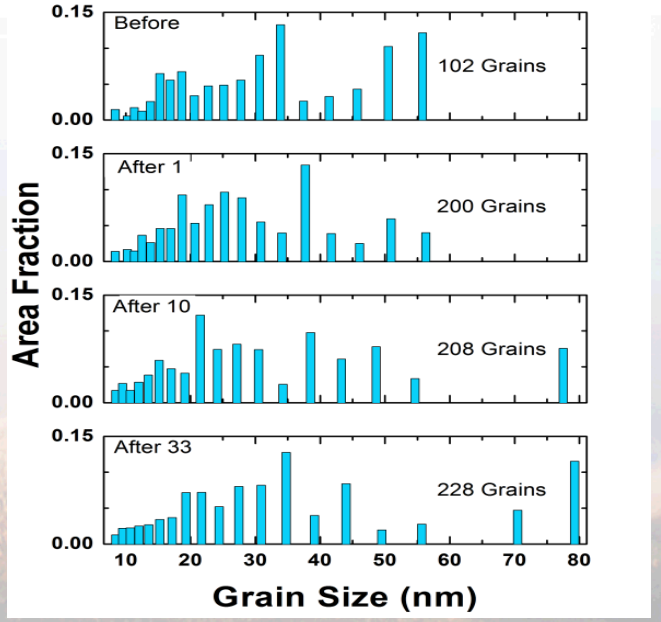




Texture and Grain size analysis can target:

- Various regions
- Quality of PED patterns
- Particular orientations
- Set of grain sizes
- Etc.





Future Directions

- *In situ* TEM high cycle fatigue
- *In situ* TEM creep
- *In situ* TEM radiation-induced creep
- *In situ* TEM stress-corrosion cracking
- *In situ* TEM implantation stress measurements
- Etc.

Combining the precision of Hysitron's Pico-indenter with harsh environments capable in Sandia's In-situ Ion Irradiation TEM a wealth of previously impossible experiments are now feasible.

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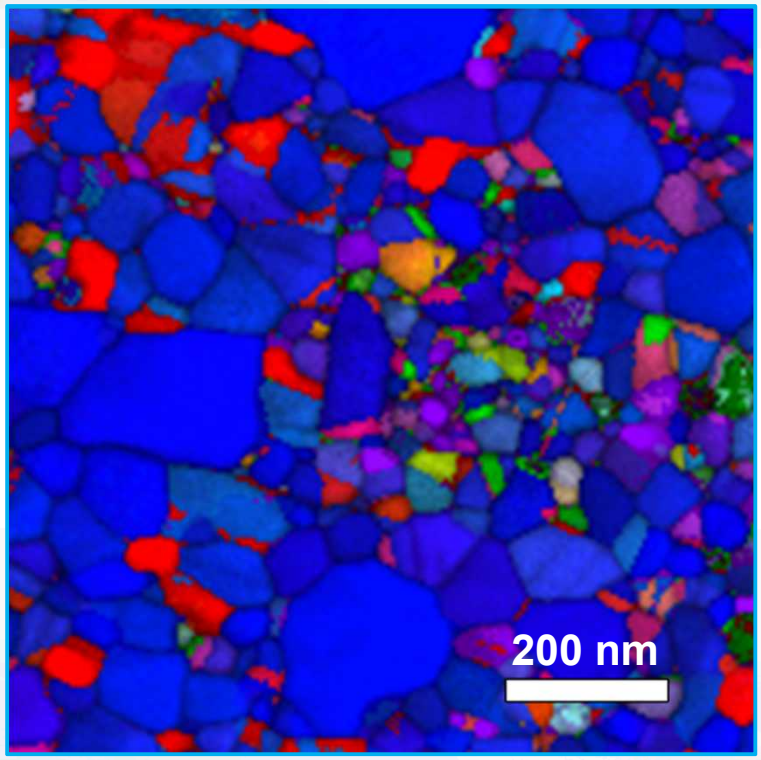
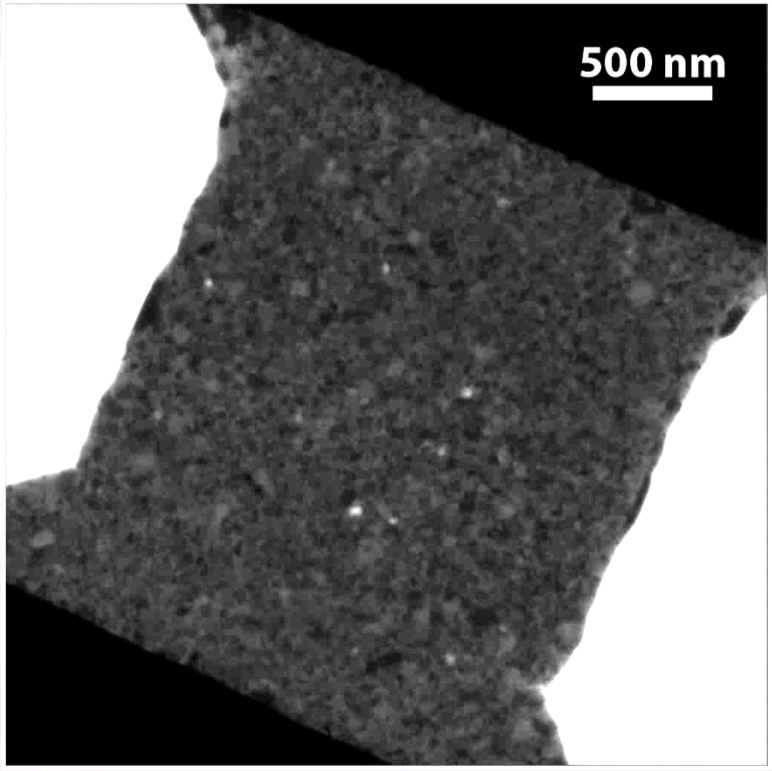
Summary





Collaborators:

- S. Bhowmick, L. Kuhn., & D. Stauffer (Hysitron)
- A. Darbal (AppFive)
- D.P. Adams, M. Marshall, B.L. Boyce and C. Sobczak (Sandia)

Combining precession electron diffraction with quantitative mechanical testing provides new correlations between structure-property relationships

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