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Cyclic Loading of Nanocrystalline FCC Metals *In Situ* in the TEM

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Daniel Bufford[1], John Sharon[1,2], William Mook[1], Brad Boyce[1], and Khalid Hattar[1]

[1.] Sandia National Laboratories, Albuquerque, NM, USA
[2.] Currently at United Technologies Research Center, East Hartford, CT, USA

U.S. DEPARTMENT OF ENERGY | Office of Science

NNSA National Nuclear Security Administration

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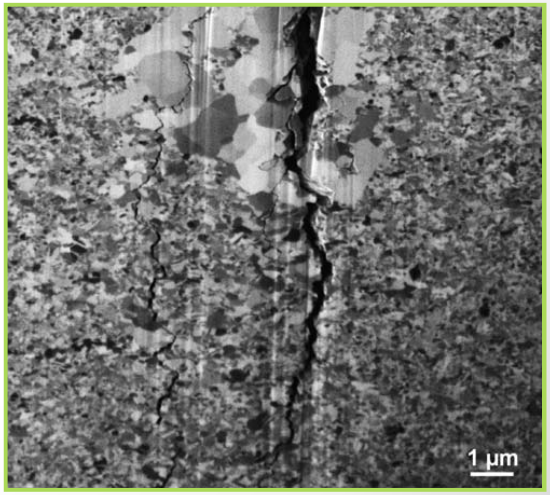
Motivation

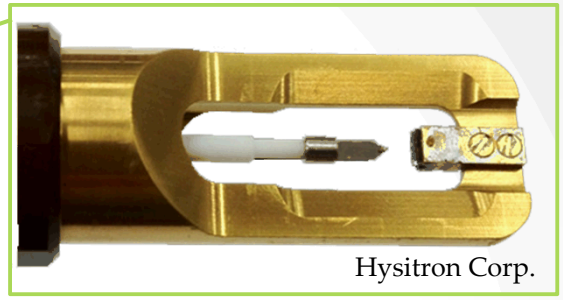
- Fatigue in bulk metals
  - Progressive microstructural change with cyclic loading
  - Often at loads below yield stress
- Fatigue in nc metals
  - Grain boundary migration and grain growth
  - Crack initiation
- What are the underlying mechanisms associated with these phenomena?
  - Pre-deformation microstructure
  - Grain and grain boundary orientations
- *In situ* TEM deformation is a tool capable of investigating these questions.

Execcharter, 2011.



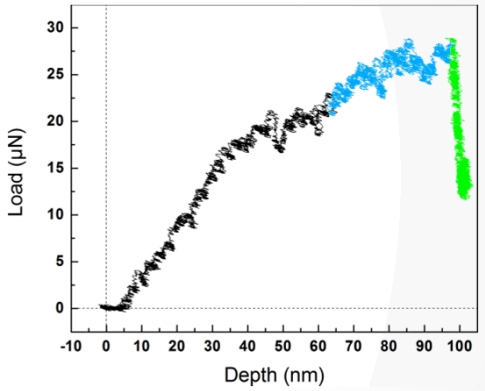
Padilla and Boyce, Exp Mech 2006.

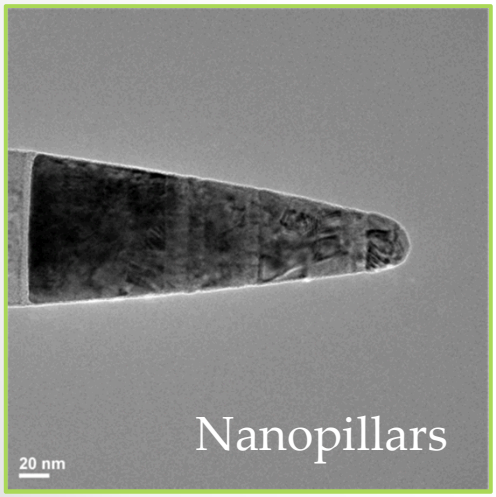
Experimental Tools

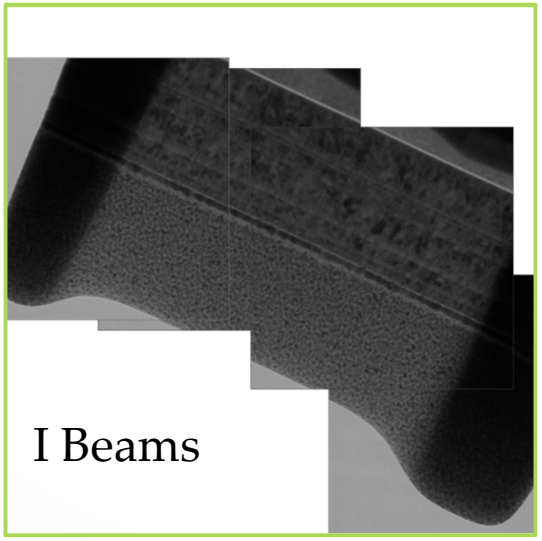


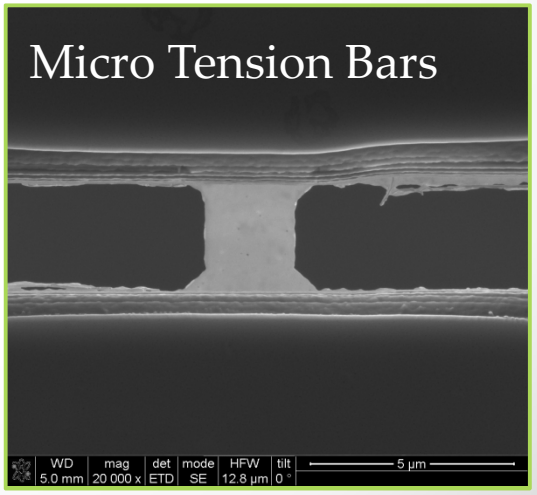
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

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



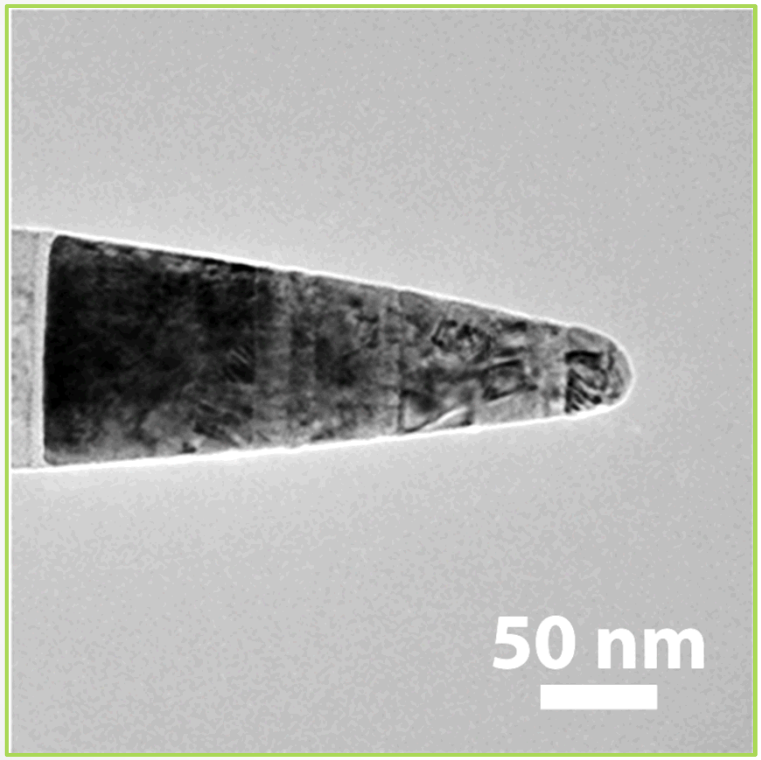






Nanopillar Fabrication

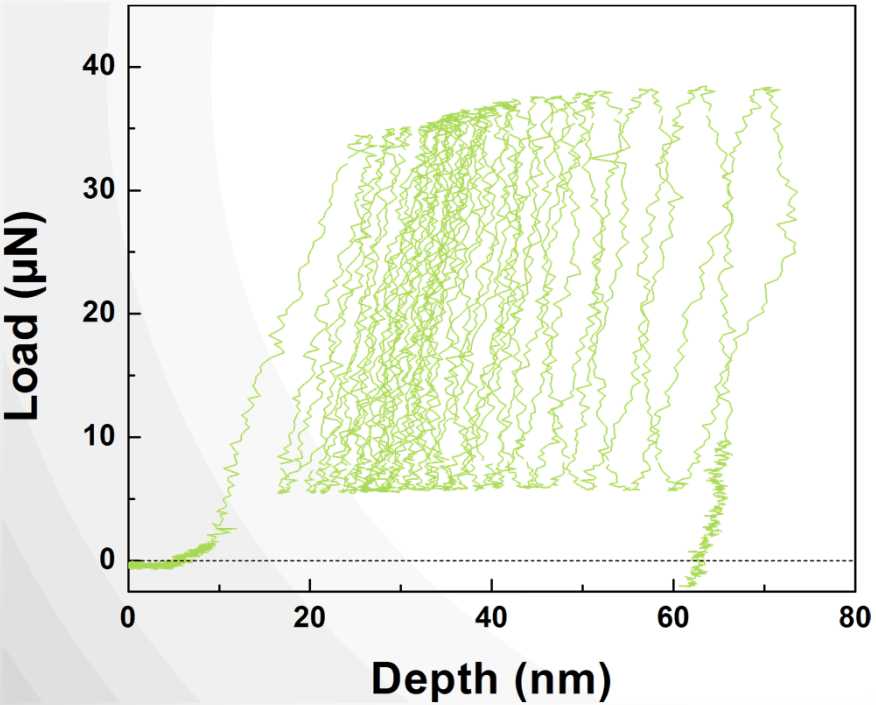
- FIB-milled from 500 nm PLD Ni on Si
  - ~100 nm base diameter
  - Conical geometry

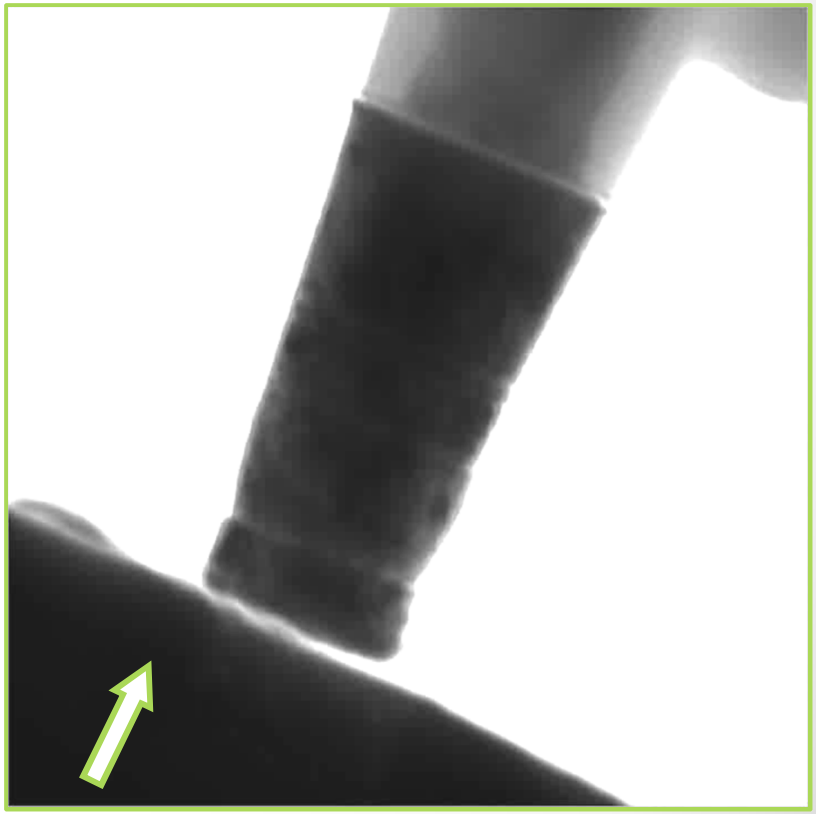


- Mostly electron-transparent, easy geometry for stress calculations
- Small volume, susceptible to vibration and shear

Pillar Compression

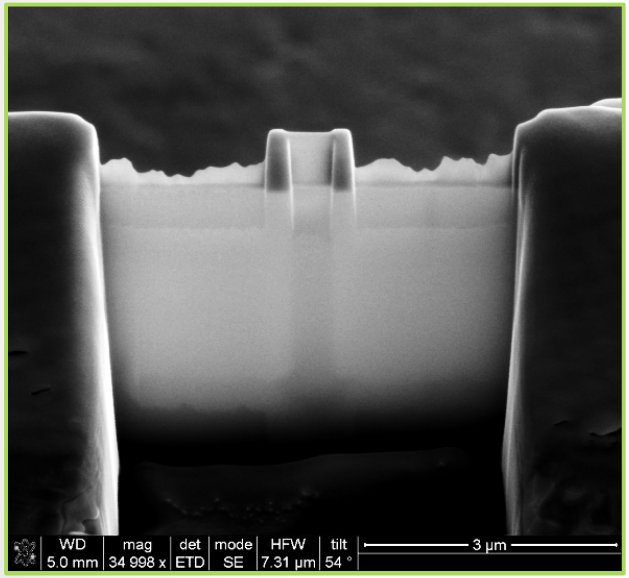
- Cyclic loading with increasing force amplitude
  - After a previous monotonic compression
  - 23 cycles to failure
- Failure initiated at notches

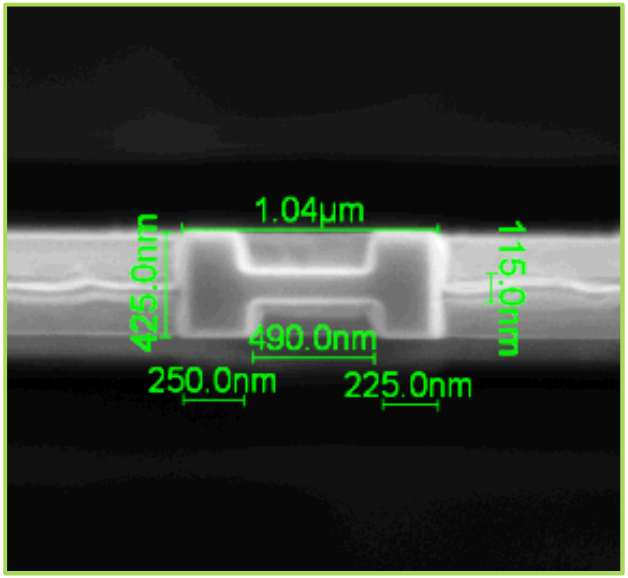


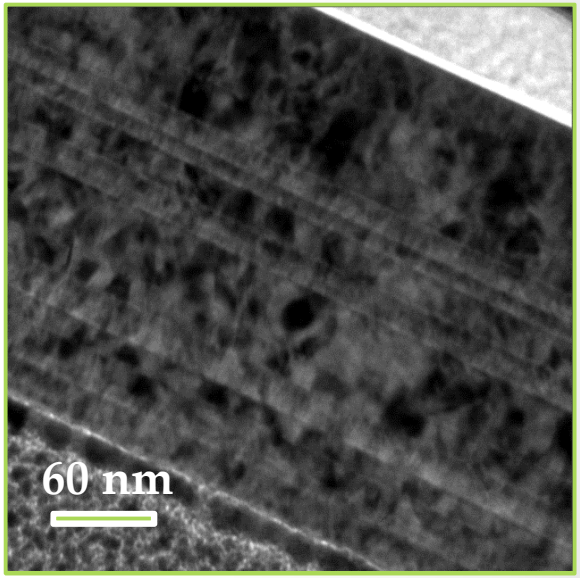


“I-Beam” Fabrication

- Wide pillars with transparent center and thicker side supports
- FIB-milled from same 500 nm PLD Ni on Si

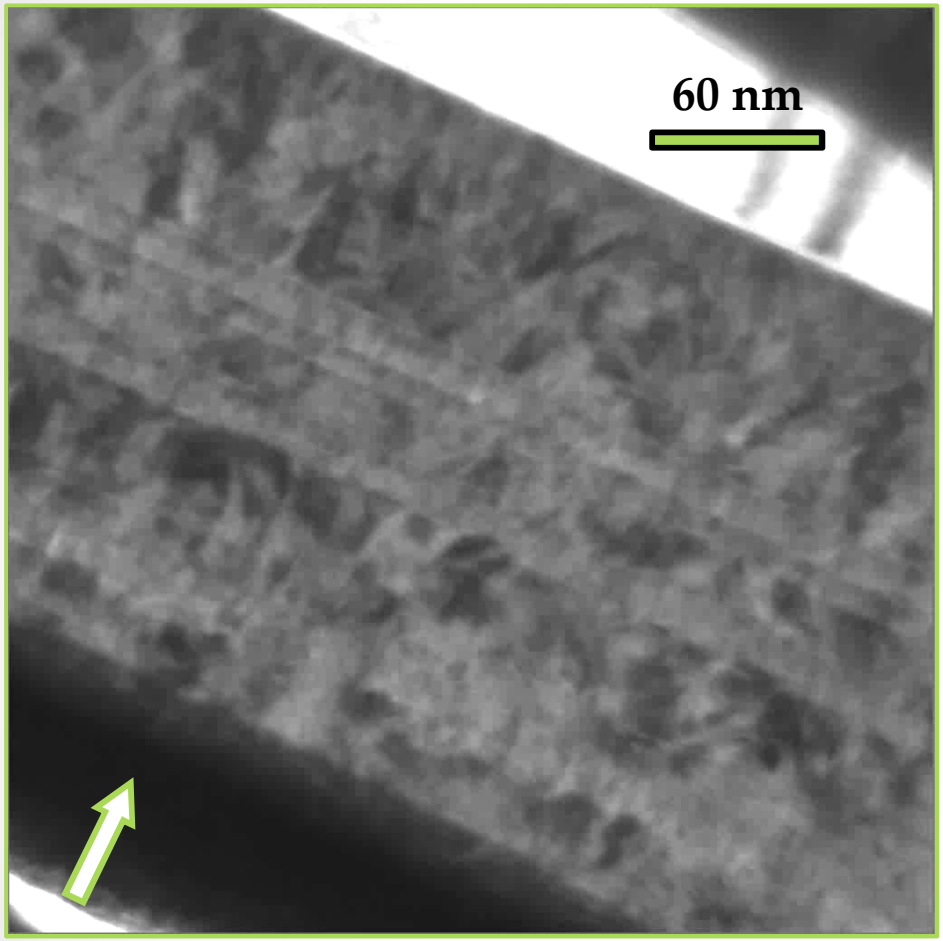


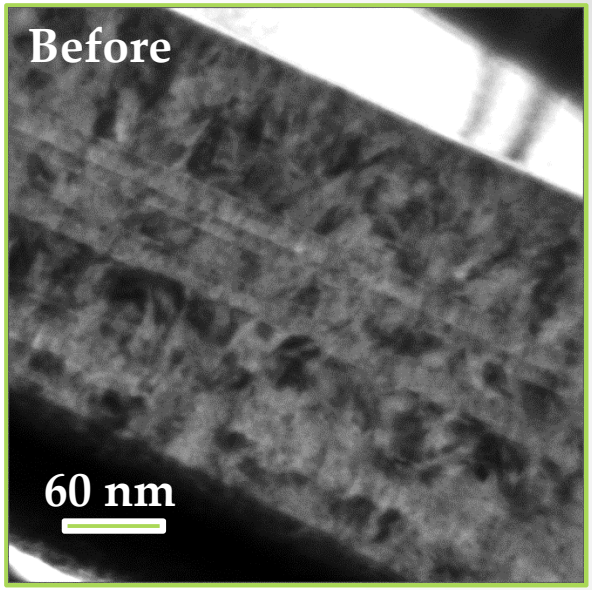


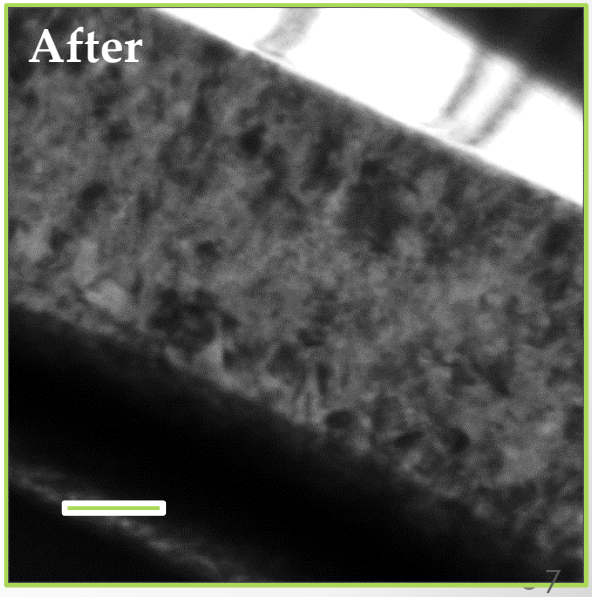


- Enhanced mechanical stability and larger viewable area
- More complicated force-displacement interpretation

I Beam Compression



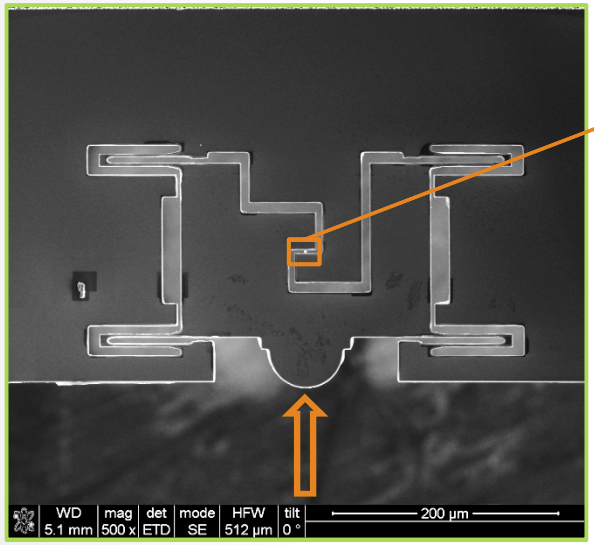


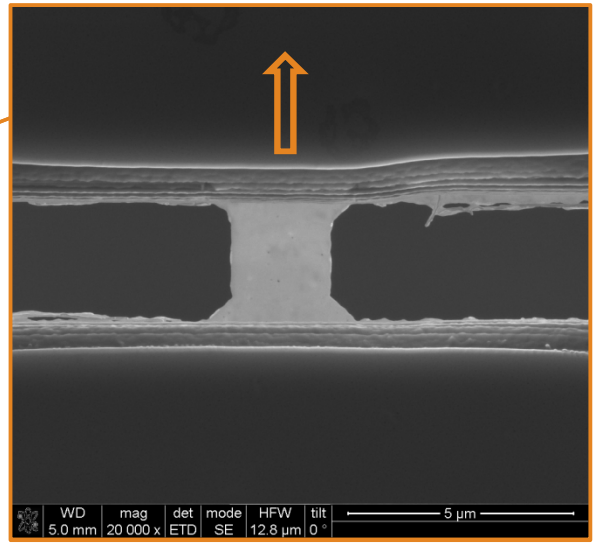


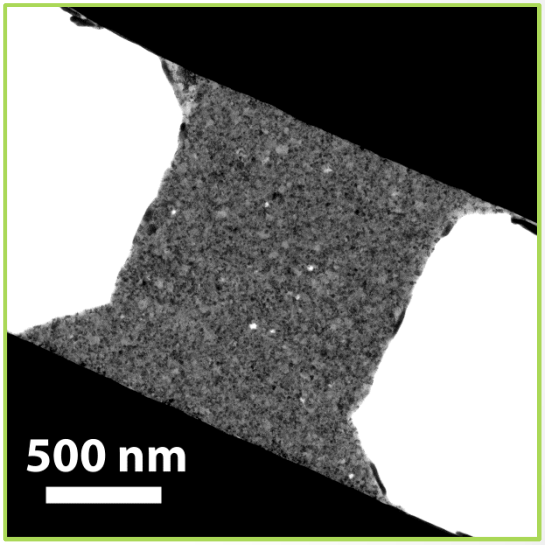
- Improved visualization
- Evidence of grain growth

Tension Specimen Fabrication

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

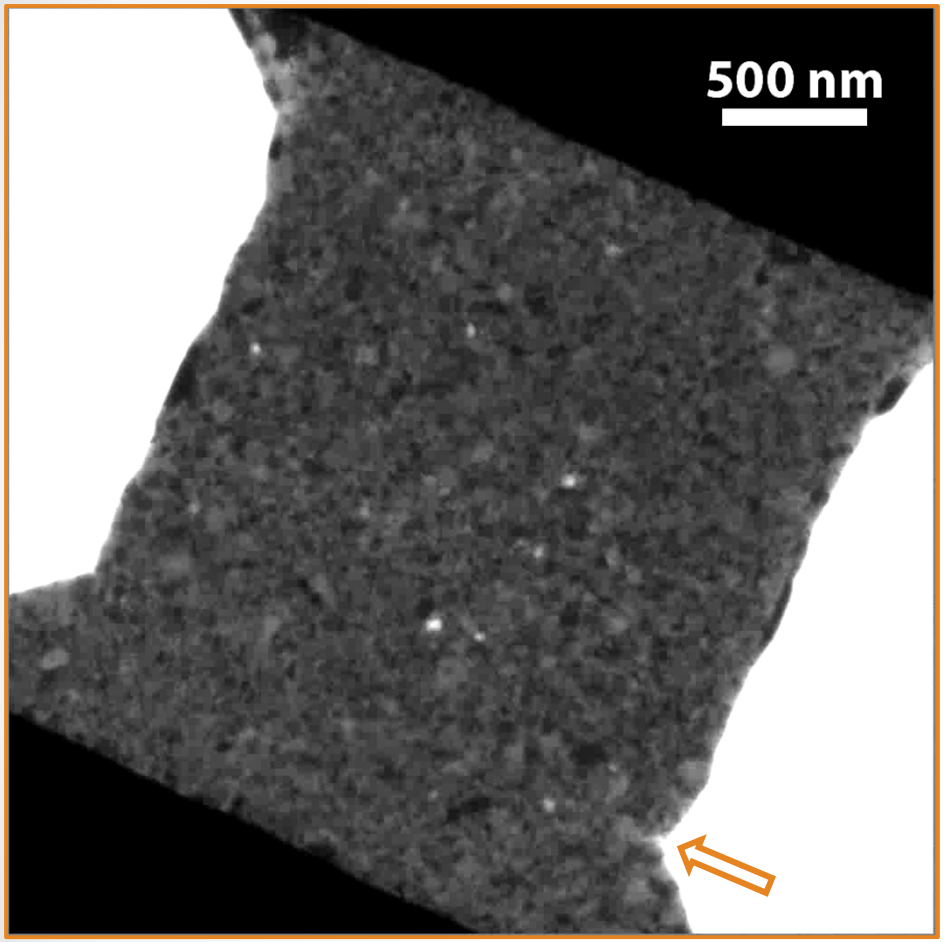






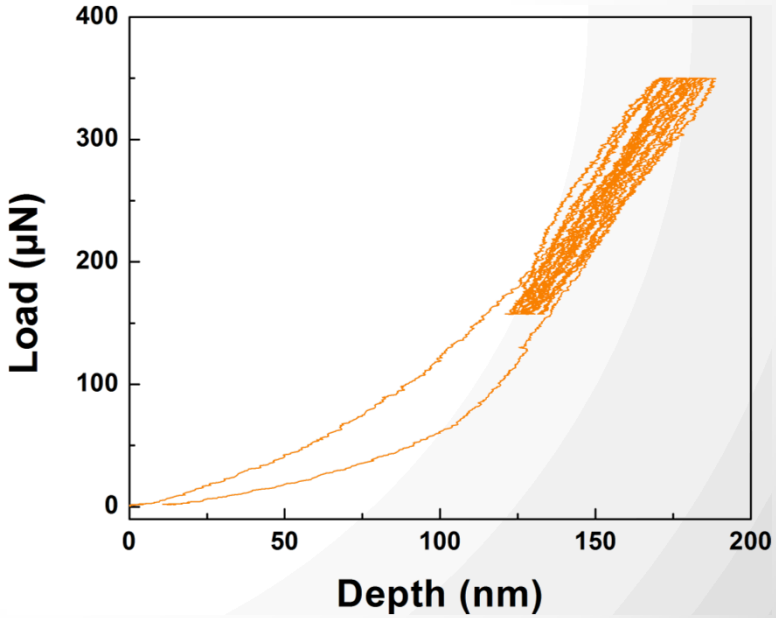
- Nearly pure tension, uniform cross sectional area, stable load frame
- Fragile, sensitive to shape of edges, issues with magnetic materials

Tension Testing



- Slow crack propagation
- Evidence of grain growth

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



Cyclic Tension *In Situ*

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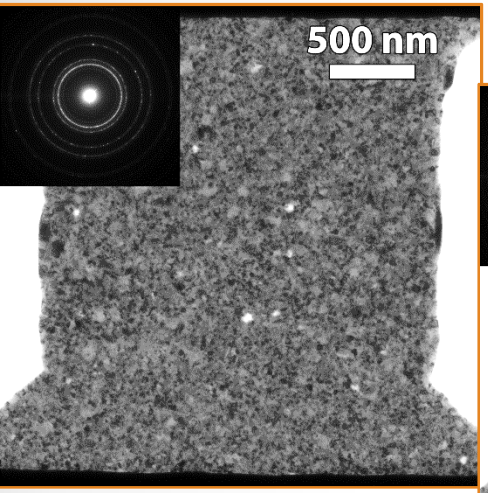
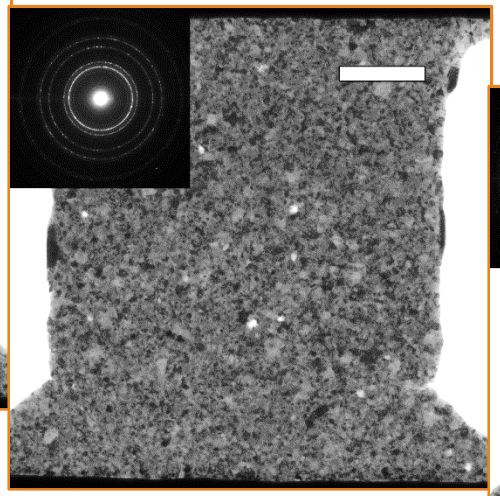
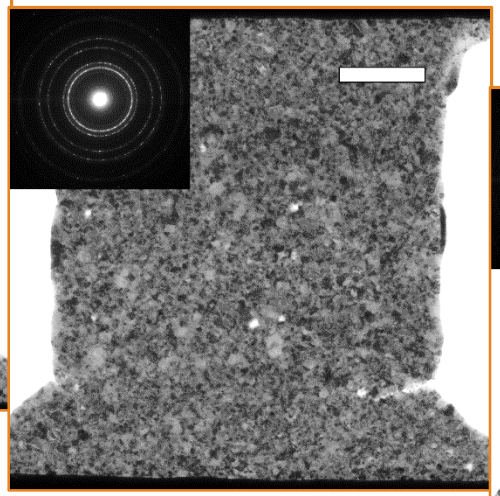
Before



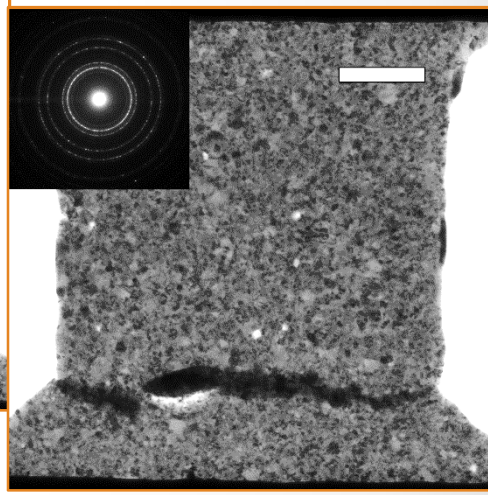
After 1 cycle



After 10 cycles



After 33 cycles

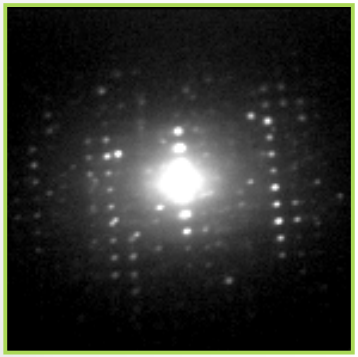


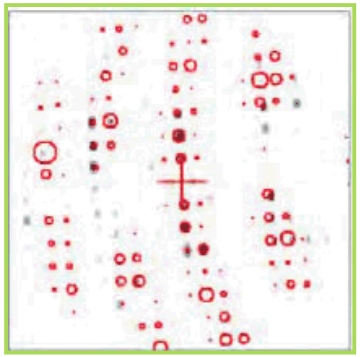
(Post-failure)

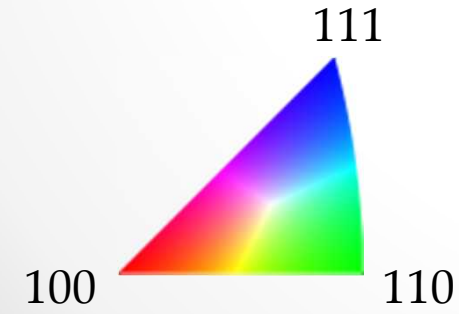
- Wealth of information from one sample:
  - Images and electron diffraction at each stage
  - Video and force/displacement during load cycles
- Microstructural change still elusive
  - Difficult to confirm and quantify

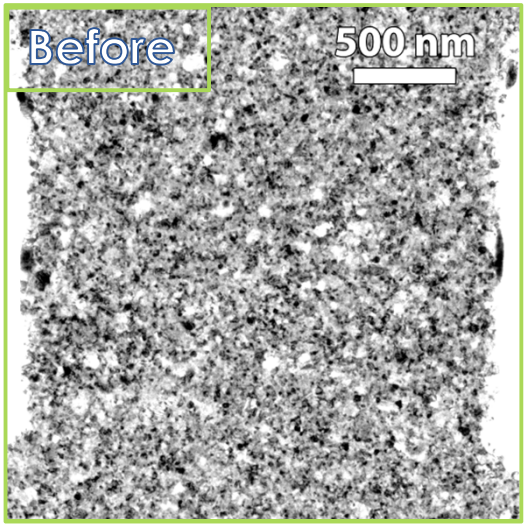
Quantifying Microstructural Change

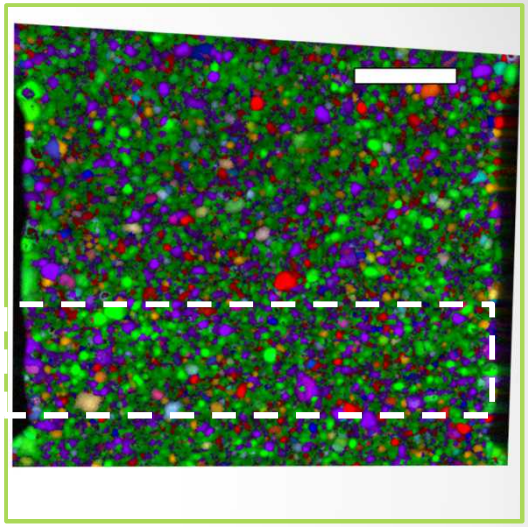
- Combining orientation mapping with deformation
- EBSD-like capability in the TEM
  - Powerful analytical tools available

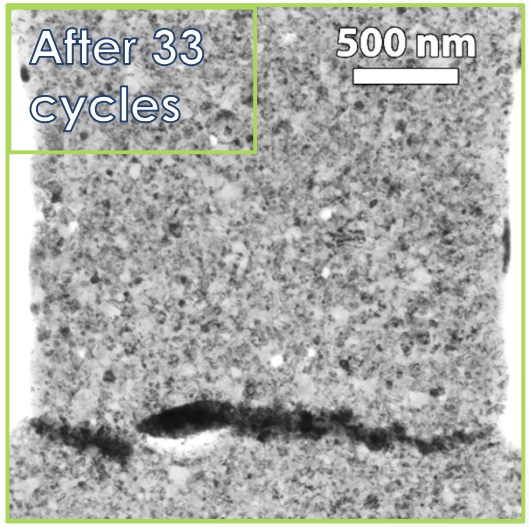


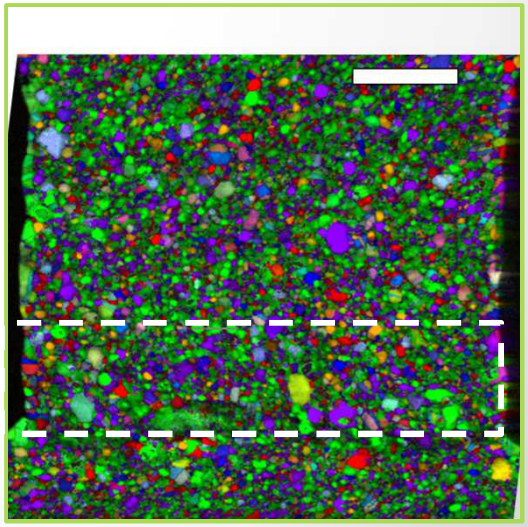






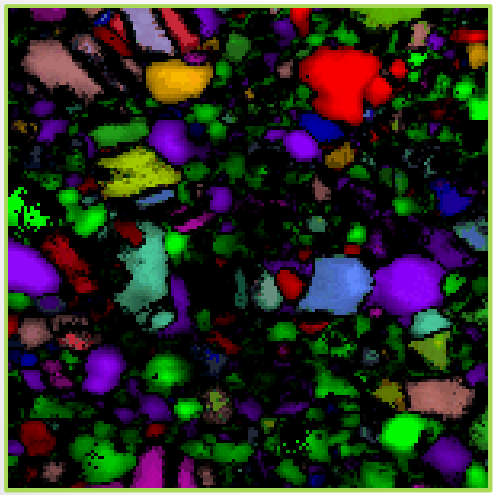


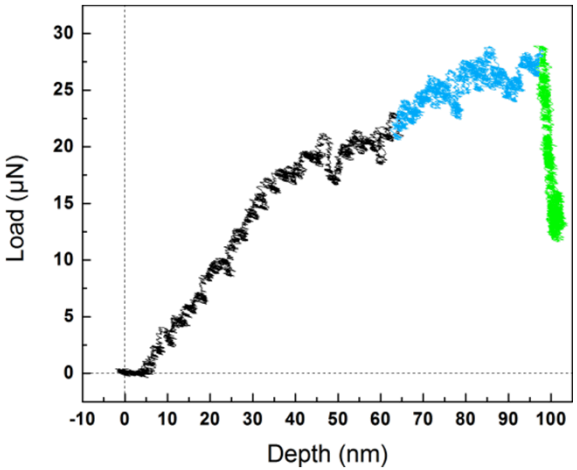


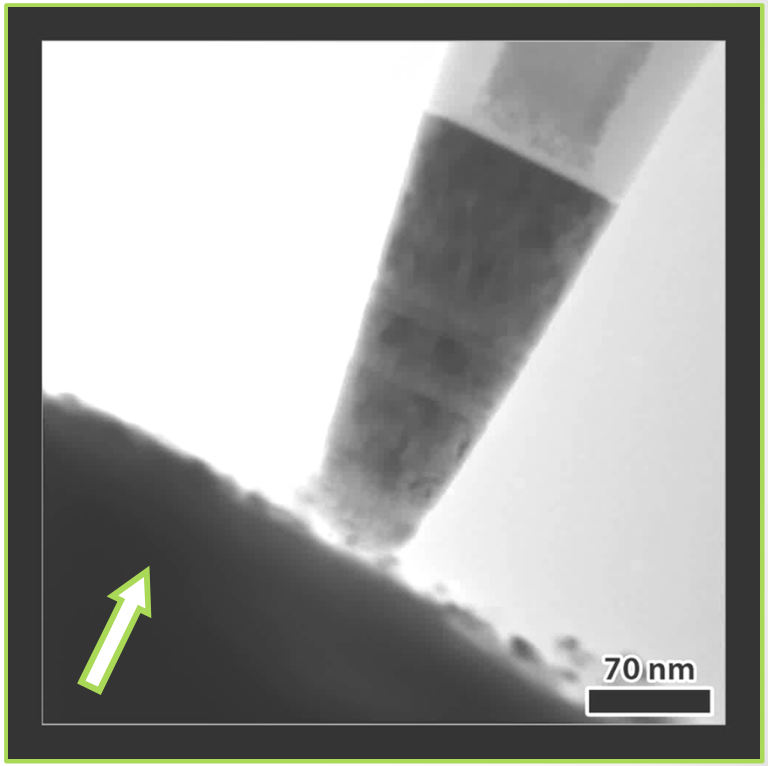


Summary and Conclusions

- Preliminary low-cycle fatigue tests of nanocrystalline Ni and Cu performed in the TEM
  - Grain growth observed
  - Demonstrated feasibility of these methods







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