Structural Stability of Nanocrystalline Cu Films during Various In-situ TEM Indentation Conditions

SAND2013-8943C

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October 29, 2013

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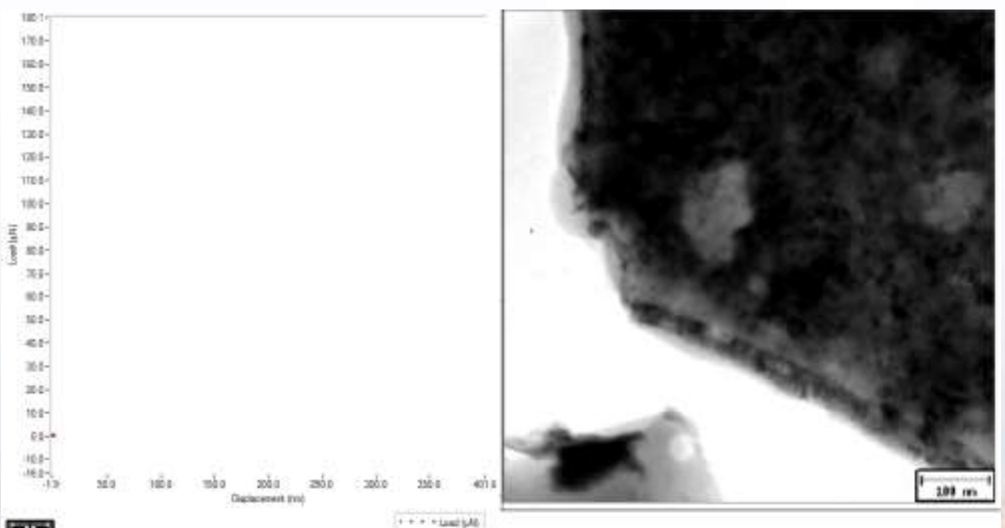

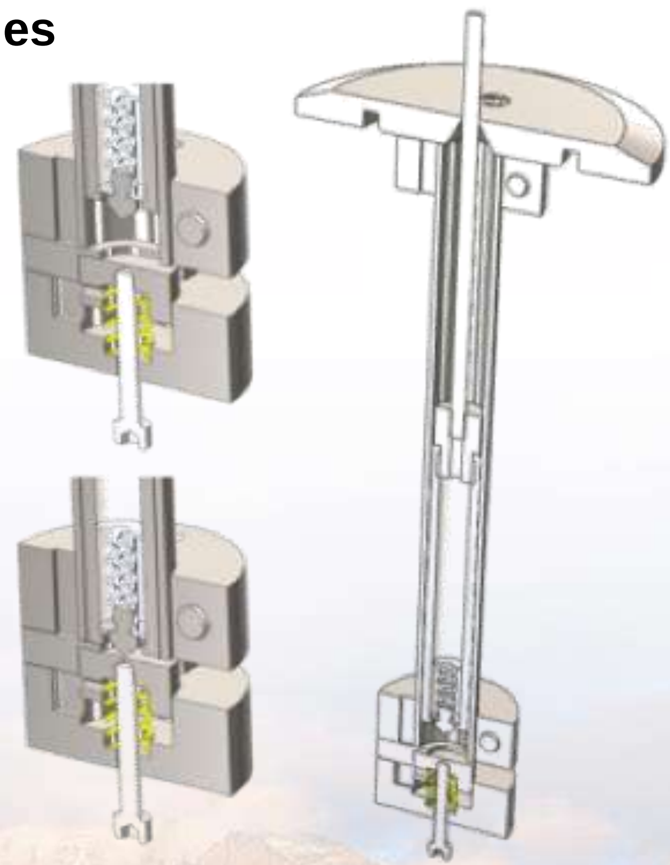

HYSITRON

This work was partially funded by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

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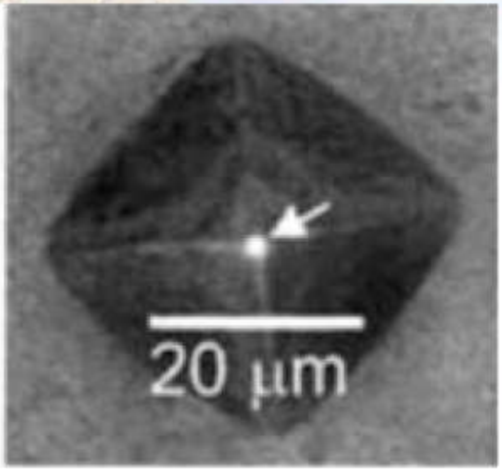
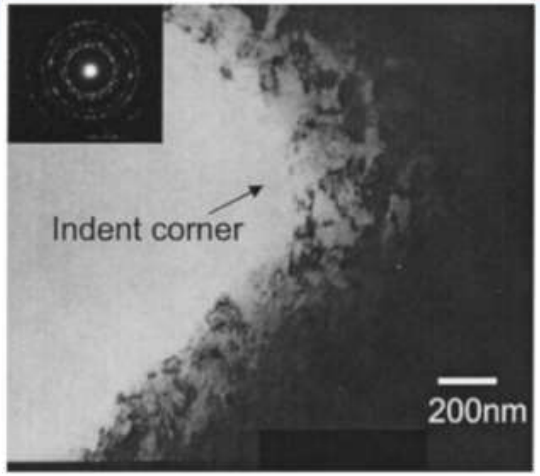
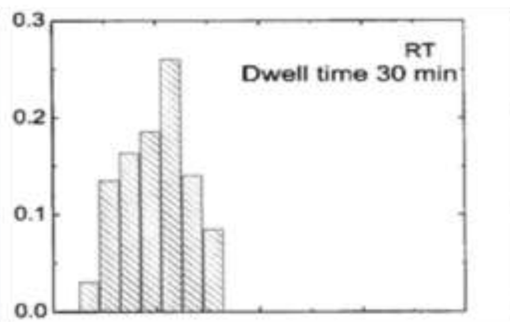
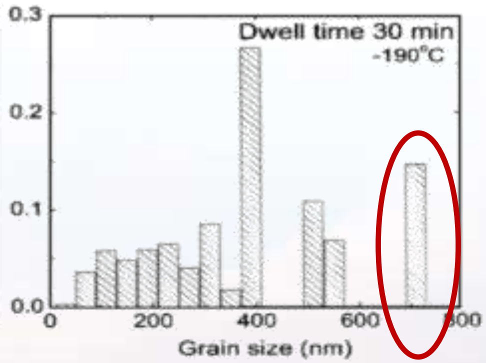
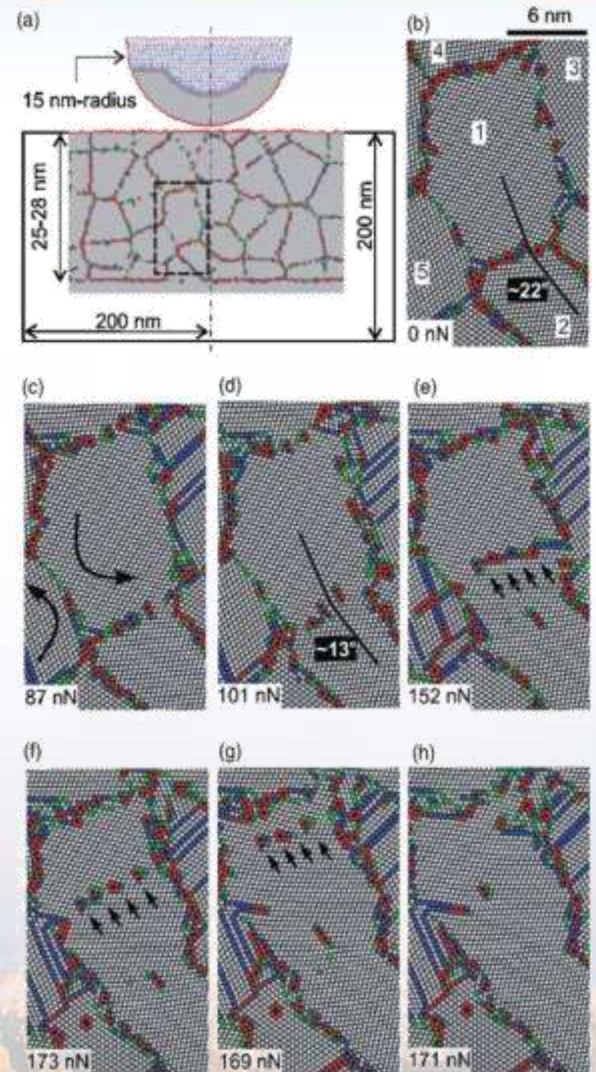
Early Reports of Cryogenic Grain Growth











Simulations at 0 K, there appears to be a process for stress-driven grain growth - *shear-coupled boundary motion*

Zhang, Weertman, and Eastman (APL 2005) reported grain growth in nanocrystalline Cu films under indentation at 77 K.

- ✓ **When the temperature is sufficient and a driving force is present, it is understood why the system exhibits growth.**
- ✓ **In their report, where *mobility* is significantly hindered, the kinetics of grain growth are poorly understood.**

Initial Observation and proposed mechanisms exist for cryogenic GG

Zhang *et al.*, *APL*, 87 (2005) 6192
Sansoz and Dupont. *APL* 2006.

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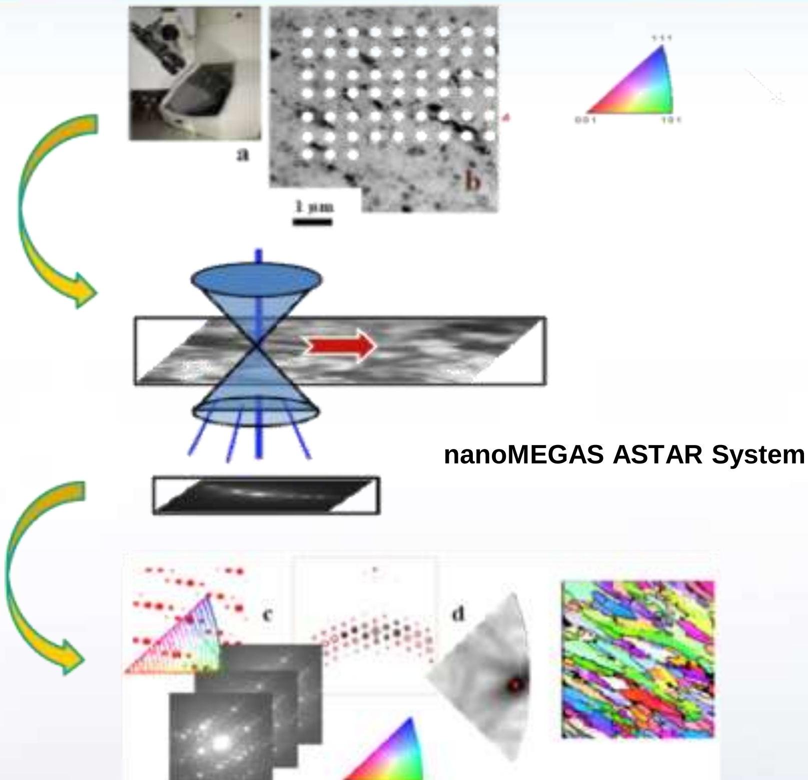
Precession-Enhanced Diffraction for Orientation Mapping

Conventional electron diffraction:

- ➢ e$^-$ interactions result in dynamical scattering, not kinematic.
- ➢ Diffracted intensities are altered such that they cannot be used for crystal structure identification.

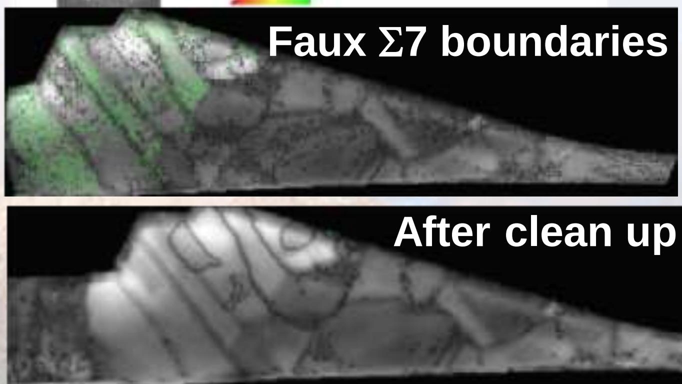
Precession-enhanced electron diffraction:

- ➢ Decreases the dynamical behavior of the e-beam by precessing the beam along the optical axis.



As a result….

- ➢ More reflections in the reciprocal space
- ➢ Diffracted intensity is closer to the integrated intensity value
- ➢ Resulting precession-enhanced diffraction pattern is close to that of kinematical diffraction
- ➢ Orientation analysis can be performed without intensive calculation

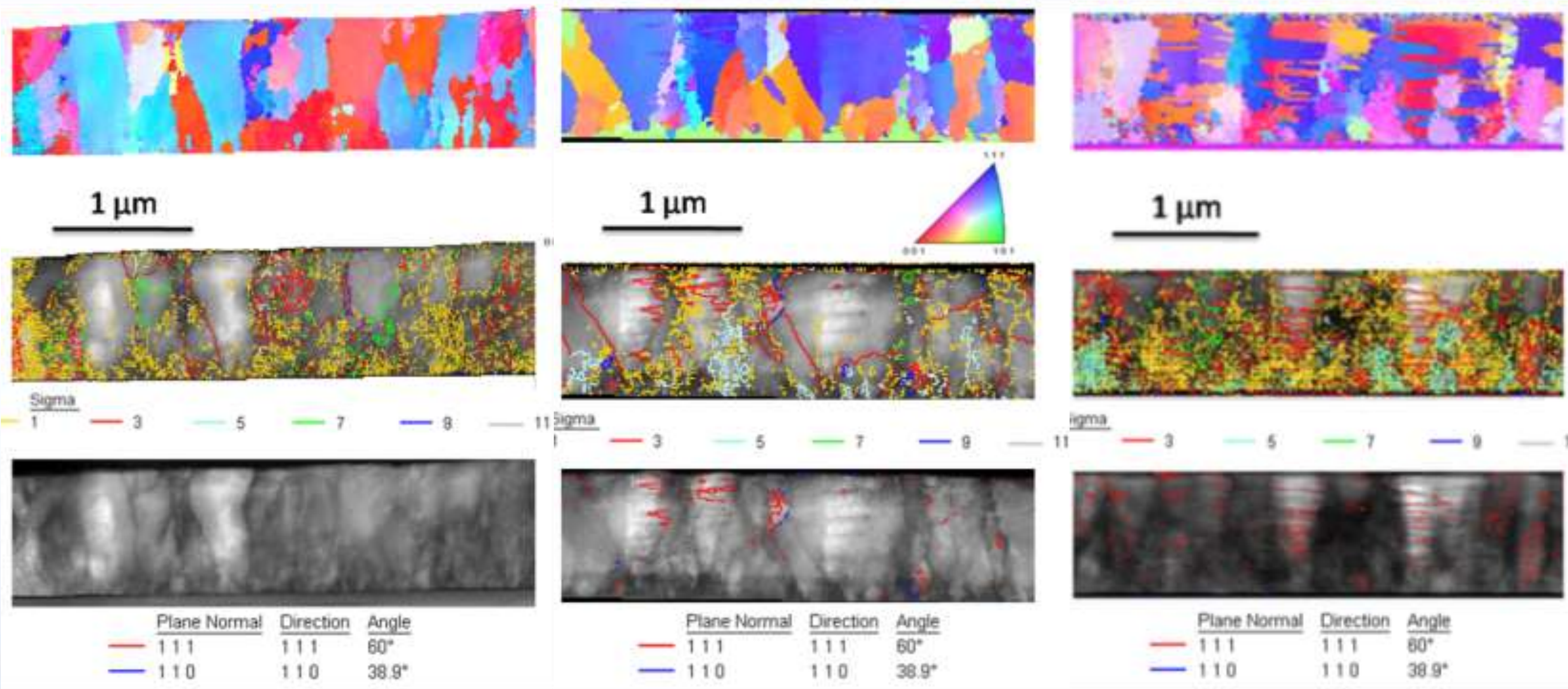


Recent advancements in precession provide a much needed capability to understand nanostructured metals

Vincent, Midgely. *Ultramicroscopy*. 1996.

Microstructure of As-deposited Films

Nanostructured Cu ***<100> Nanotwinned Cu*** ***<111> Nanotwinned Cu***



Sigma: 3 5 7 9 11

Twins plotted in bottom set

Deposition parameters during PLD can be used to control local texture and twin density

Brons, et al. *Scripta Materilia*, 68 (2013) 781-784

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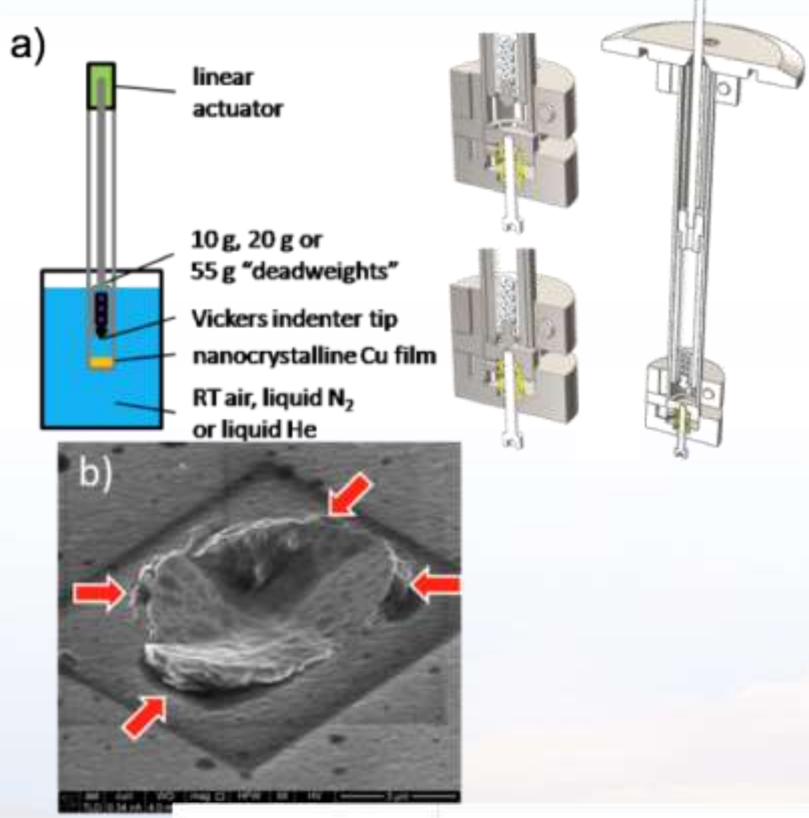
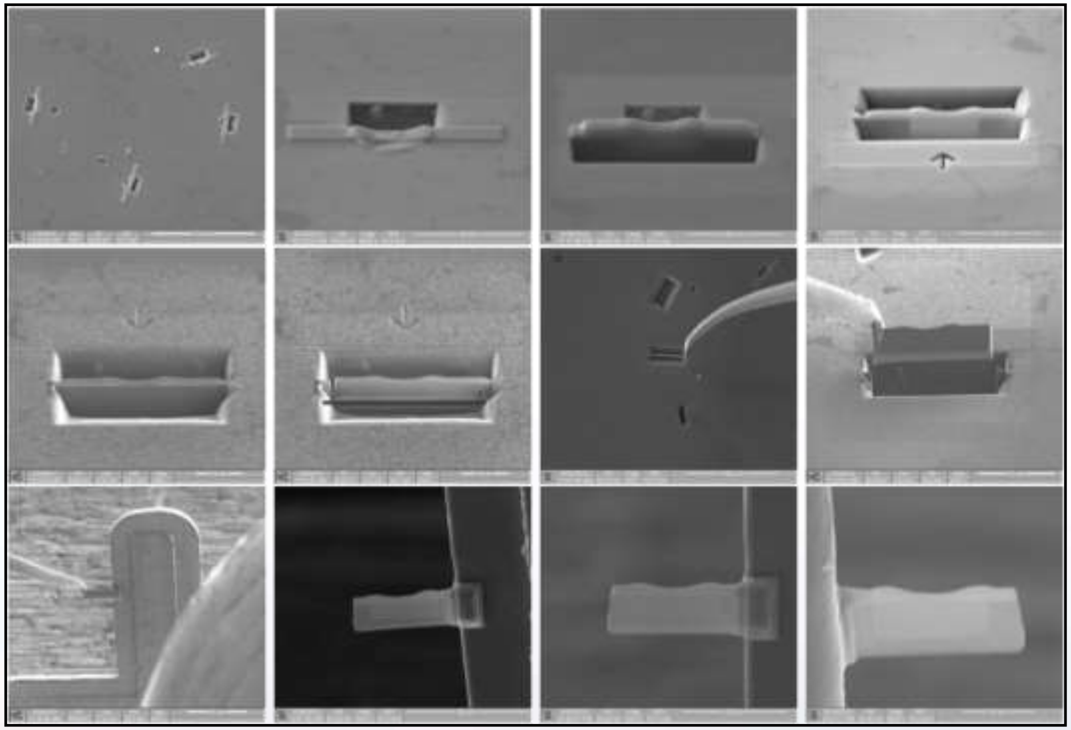
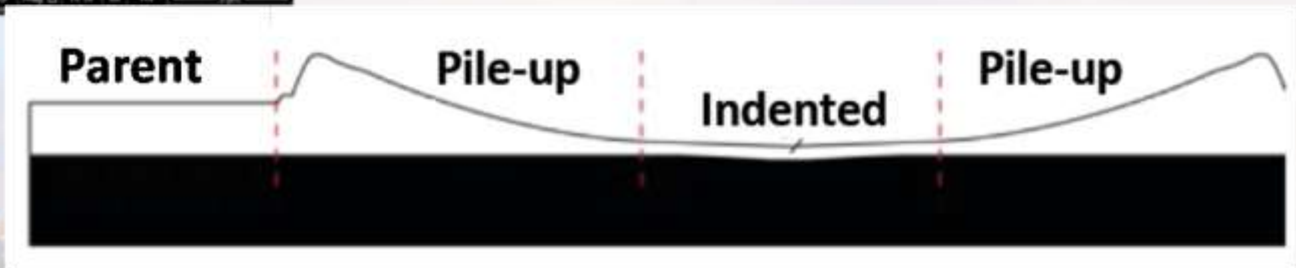
Cryogenic Indentation & TEM Foil Preparation

Untwinned and nanotwinned Cu films were indented using a Vickers indentation tip at a load of 55g while submerged in liquid N or He for 600 seconds.



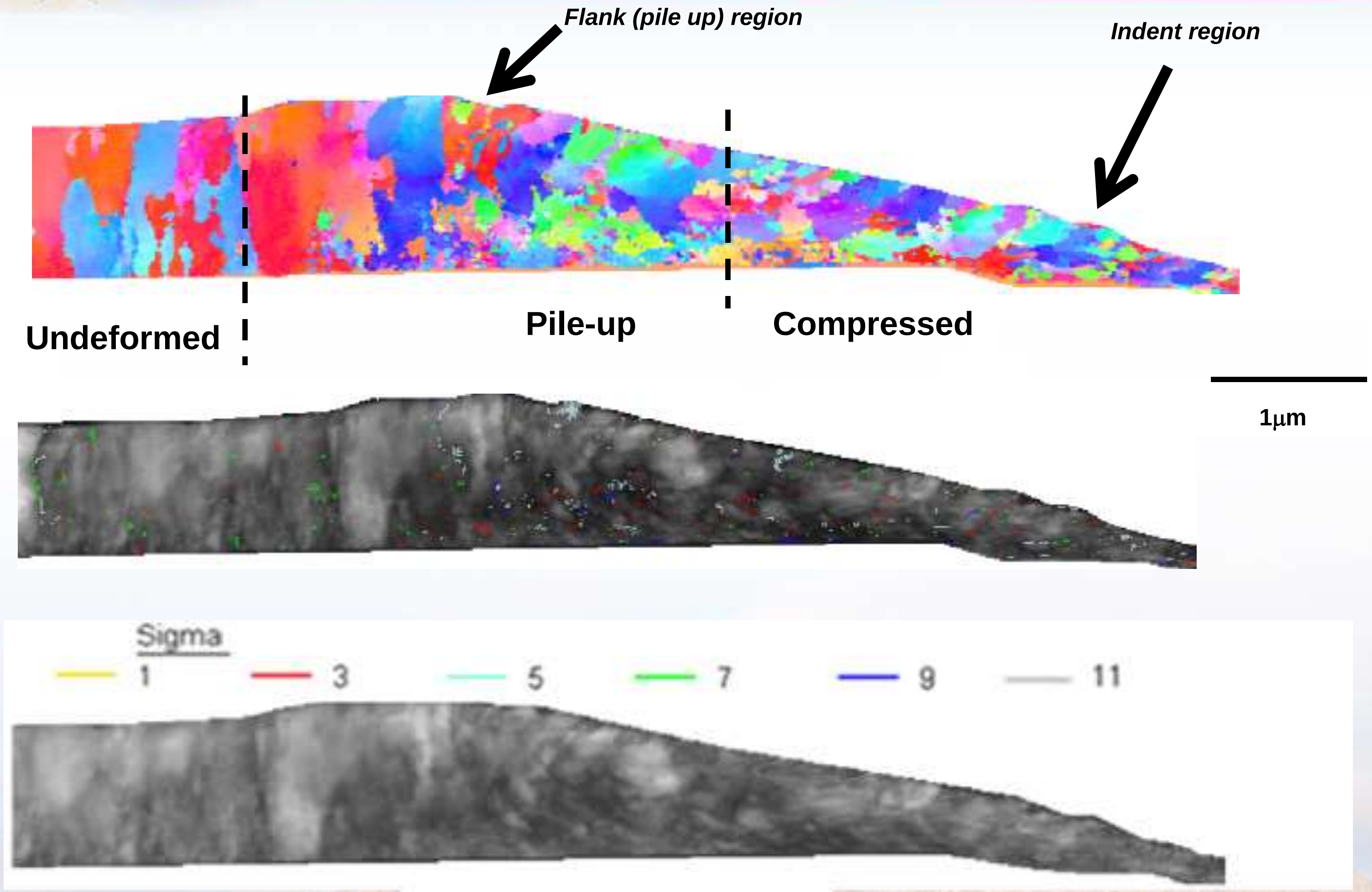




Foils were prepared that allowed for the characterization of both sides of the indent as well as the parent material. Post-FIB, PIPS milling done to remove any surface damage

Brons, et al. *Scripta Materilia*, 68 (2013) 781-784

Nanostructured Cu Indent



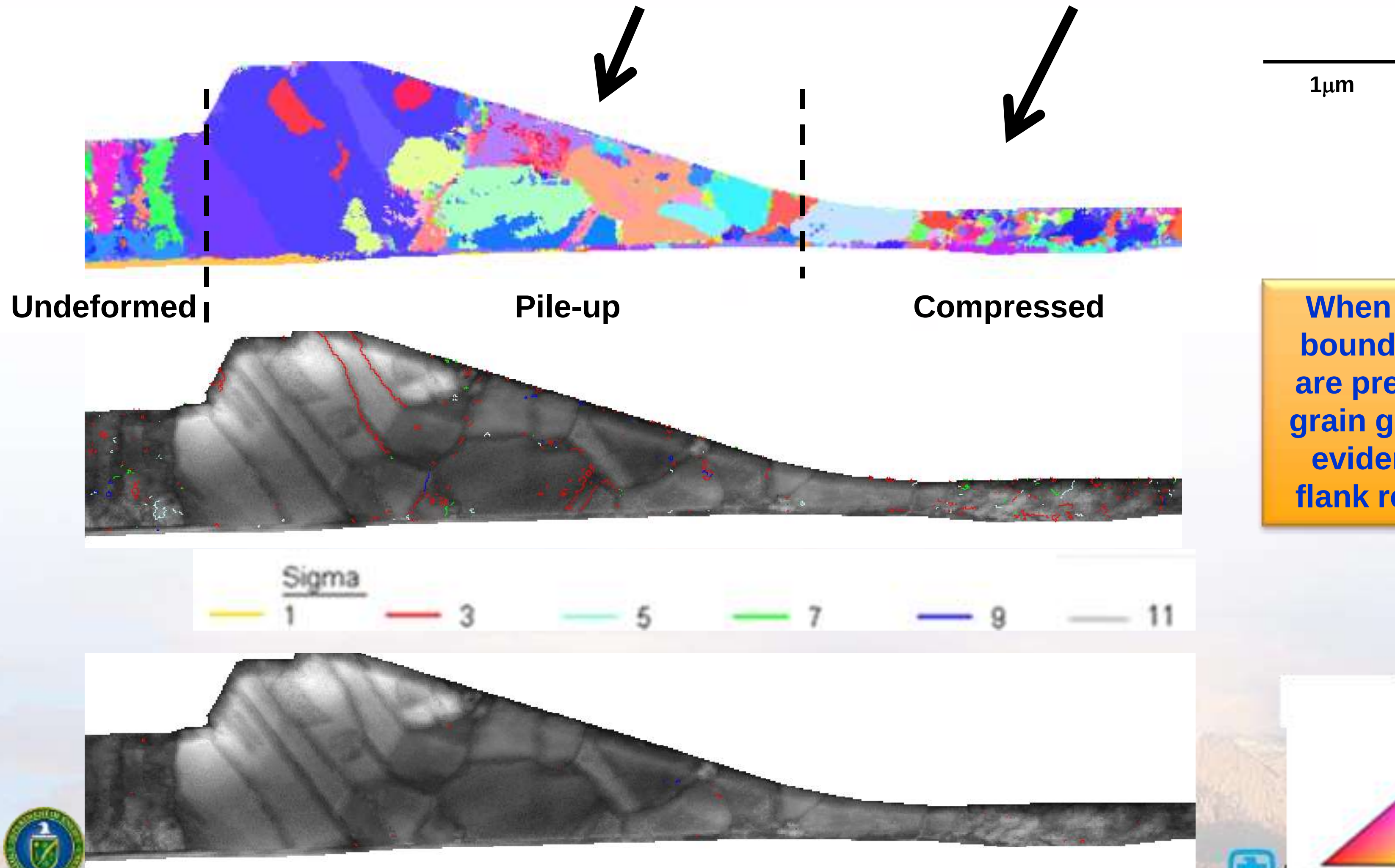
Grain distortion occurs in the flank of the indent - along with low angle boundary formation. No evidence of grain growth

Nanotwinned Cu Indent

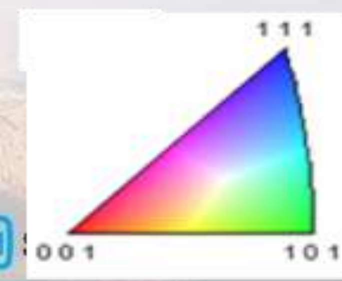
<111> Nanotwinned Cu

Flank region

Indent region



When twin boundaries are present, grain growth evident in flank region



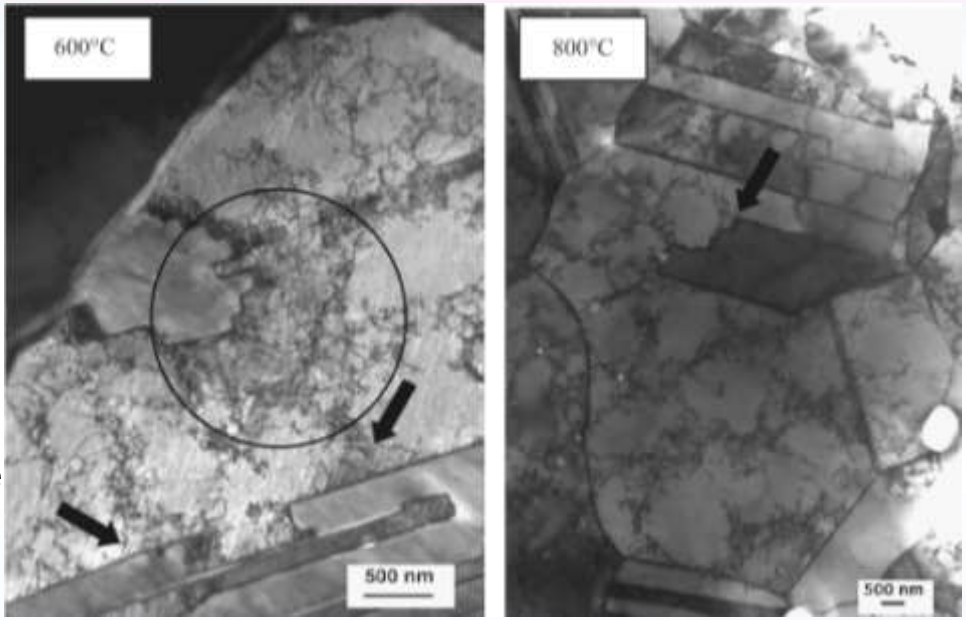
Brons, et al. *Scripta Materilia*, 68 (2013) 781-784

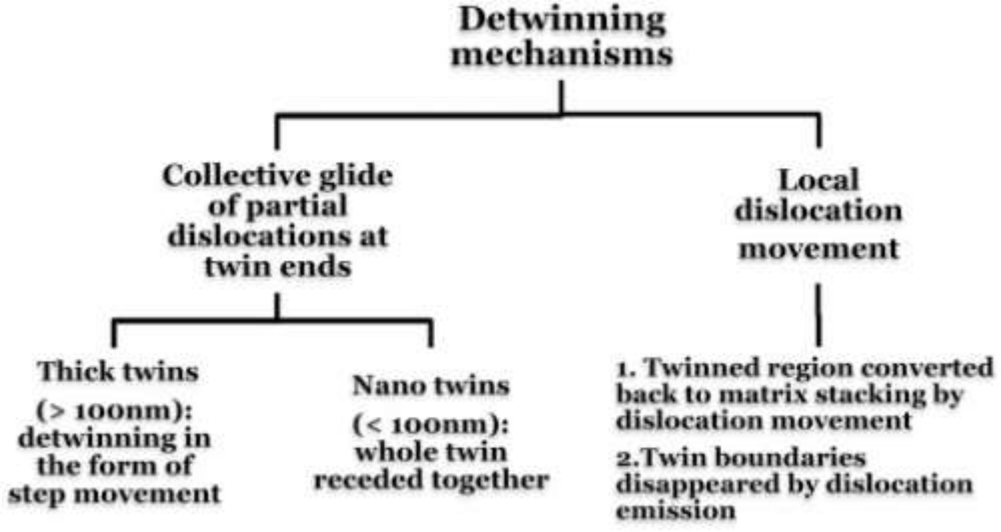
De-twinning Mechanism

Although twin boundaries are more resistant to migration than other boundaries, Chen *et al.* explained that apparent detwinning processes are found to exist.

Dislocation arrays are often found at the migrating twin boundary front.

This allows twin boundaries to annihilate locally and convert themselves into single dislocation lines through detwinning mechanisms:





The driving force for detwinning was attributed to the interaction between two twin boundaries, arising from the variation in the energy of the twin boundaries.

Shear stresses enable the twin boundaries to migrate easily.

Chen *et al*. *Materials Science and Engineering A*. 2012.

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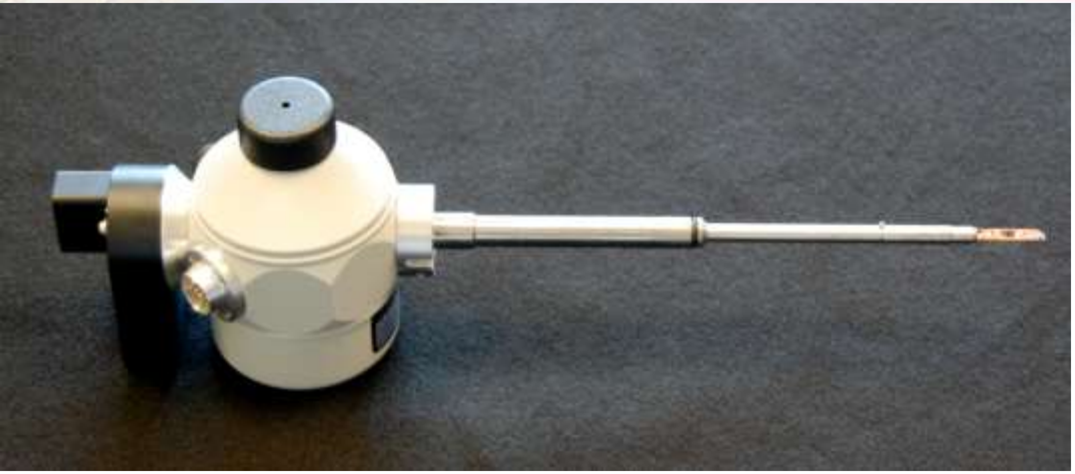
In-situ TEM Cryoindentaiton



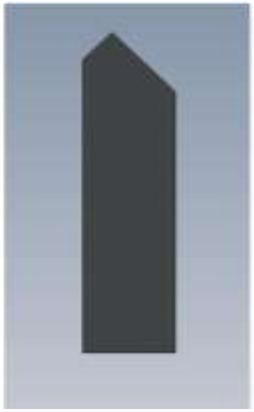
<u>Substrates etch control</u>

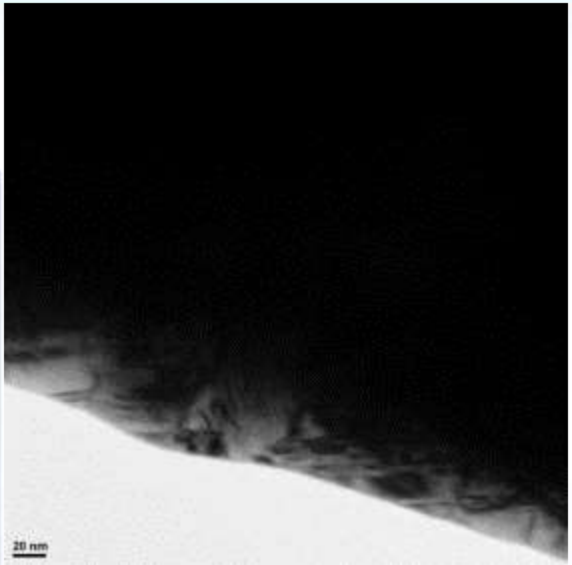
Nitrided Silicon substrates have been single etched or double etched using a TMAH process to yield two profiles. The single etch gives larger thin areas, the double etch is thicker. The profile seems to affect the film growth.

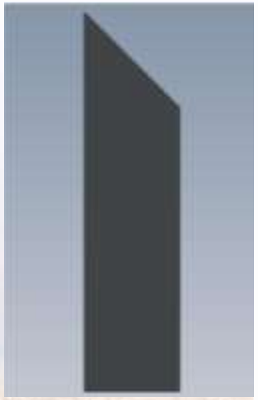
2) Cu Film Deposition Parameters

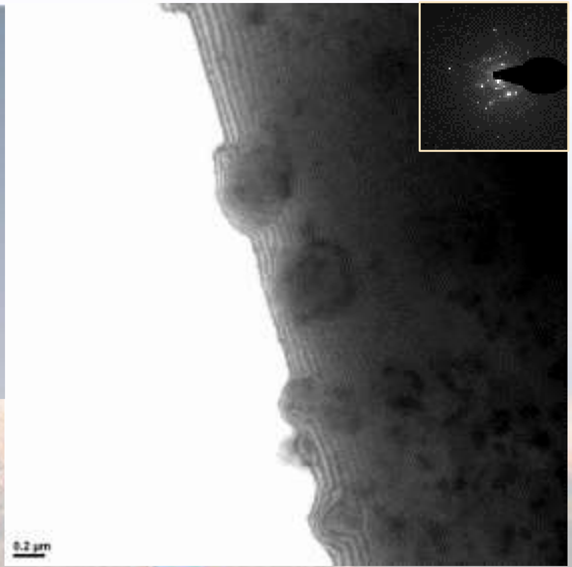
Double etched, 230 nm Cu showing twins but limited thin region, comparable to a XTEM sample.

Single etched, 100 nm Cu showing thickness fringes over a large area, comparable to a Plan View TEM image of PLD Cu.

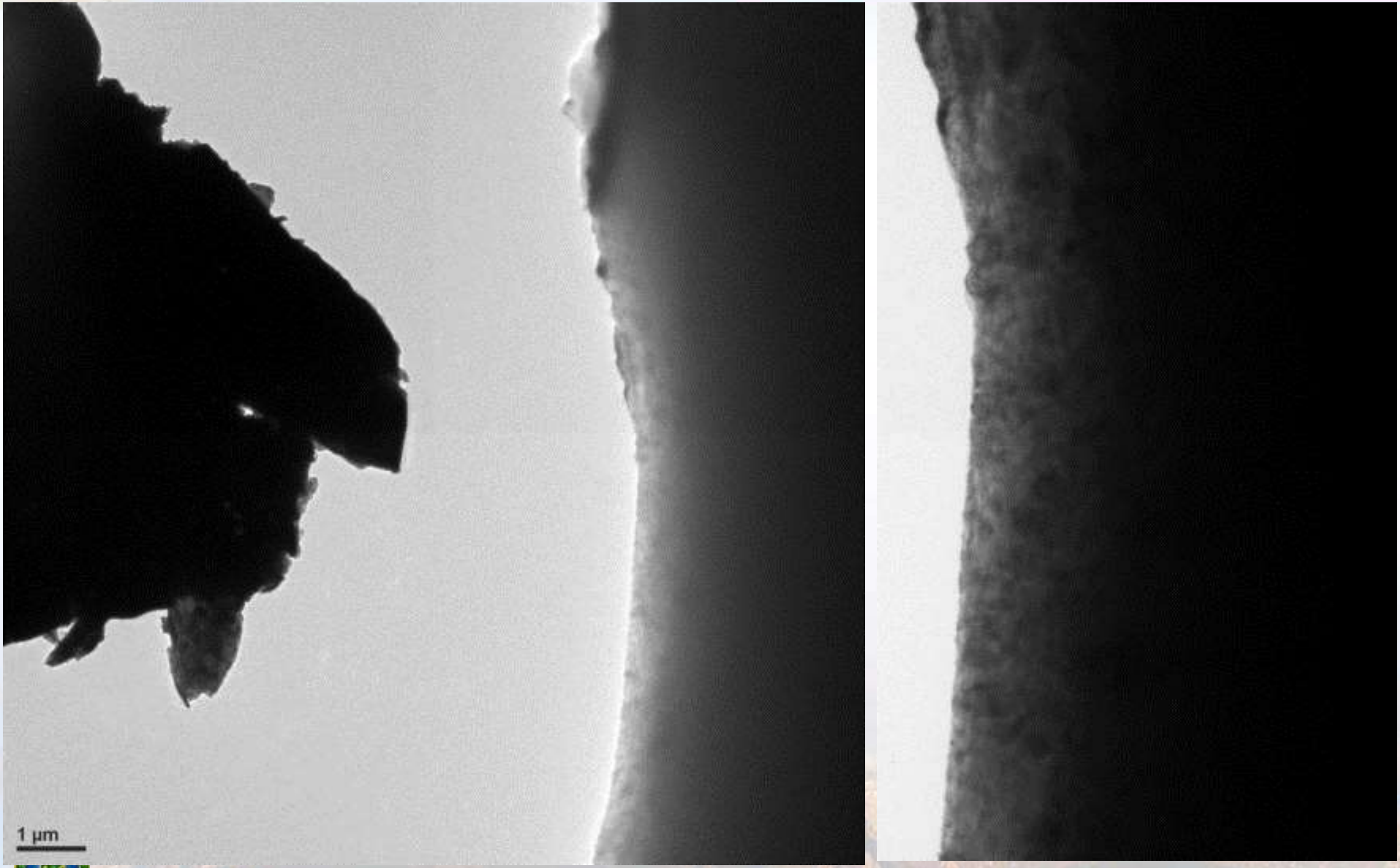






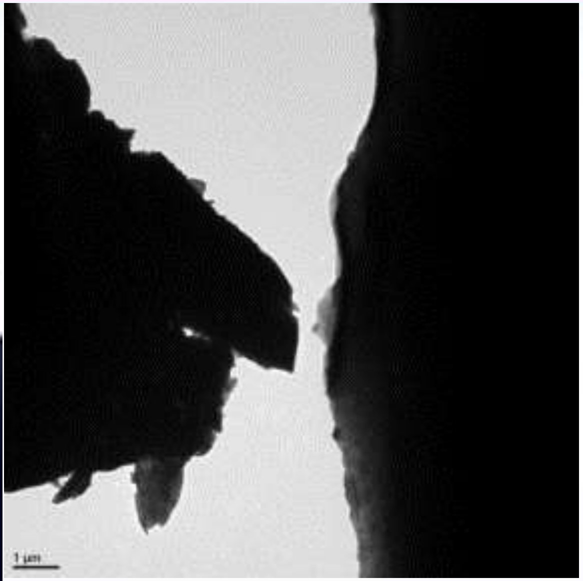


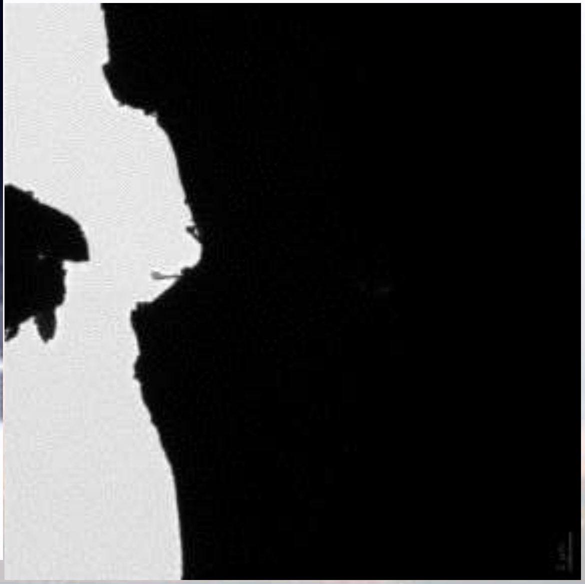
RT Pre-identation



Tip quality, shadowing, and electron transparent region are limiting

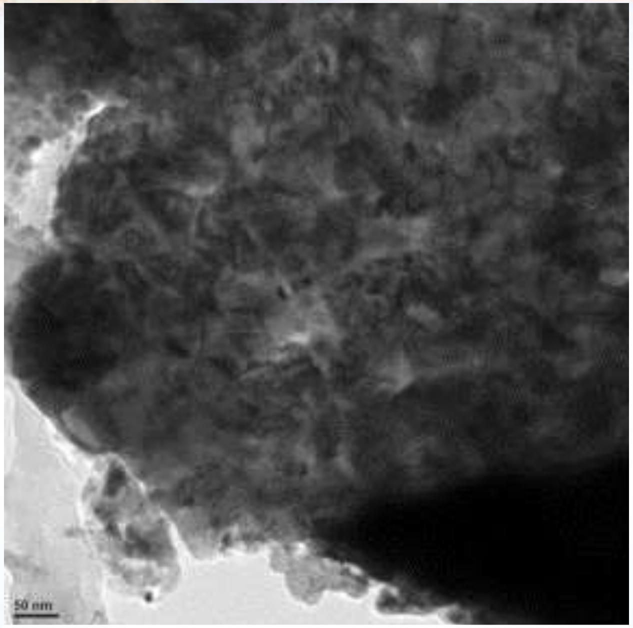
Cryo indent of Cu films



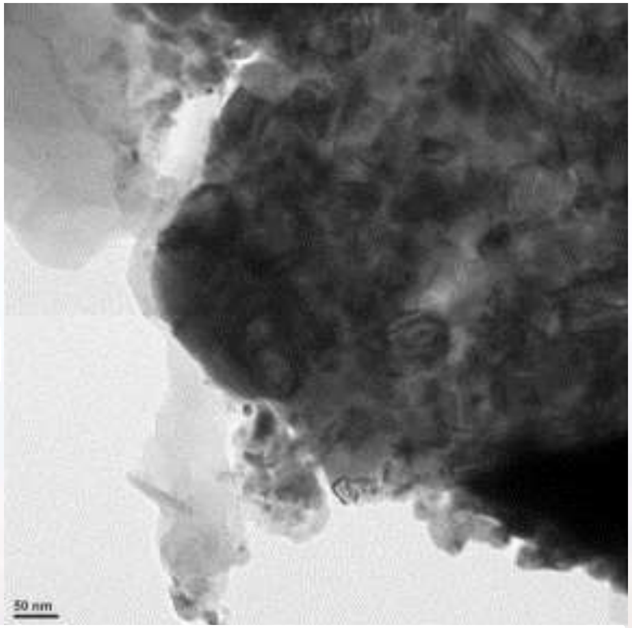


Warming from Cryogenic Temperatures to Room Temperatures

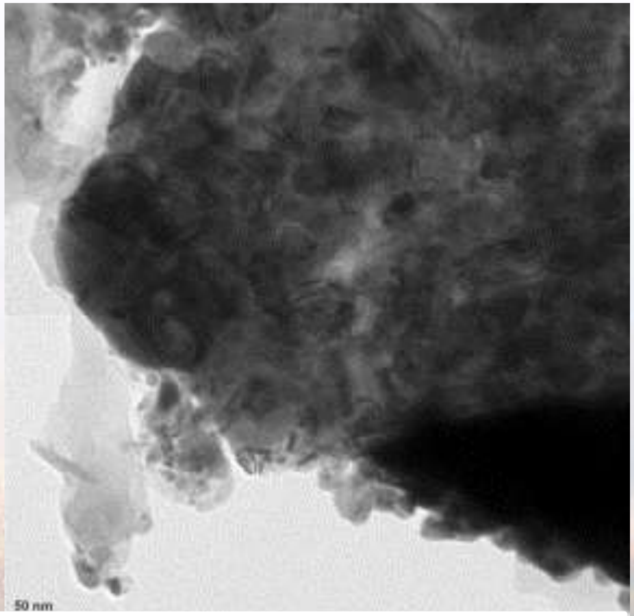
Structural changes are observed due to indentation at cryogenic temperatures, but no grain growth



Pile-up region after cryo-indent



Same pile-up region during warming

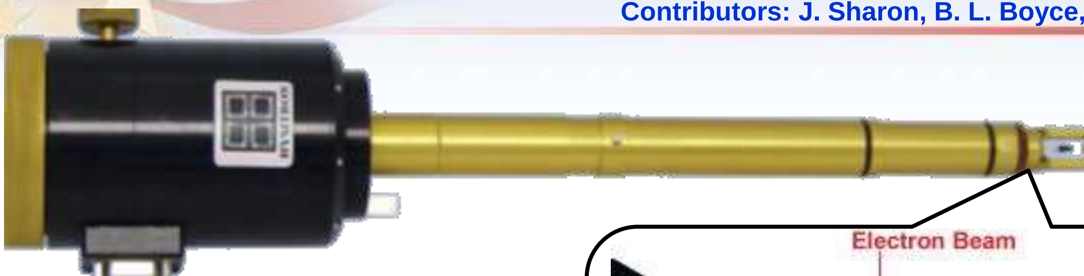


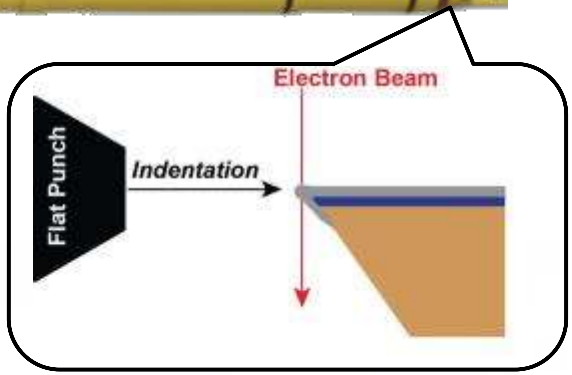
Same pile-up region at room temperature

Further evolution occurs during warming, but no obvious abnormal grain growth was observed

In situ TEM Quantitative Mechanical Testing

Contributors: J. Sharon, B. L. Boyce, & Hysitron Inc.

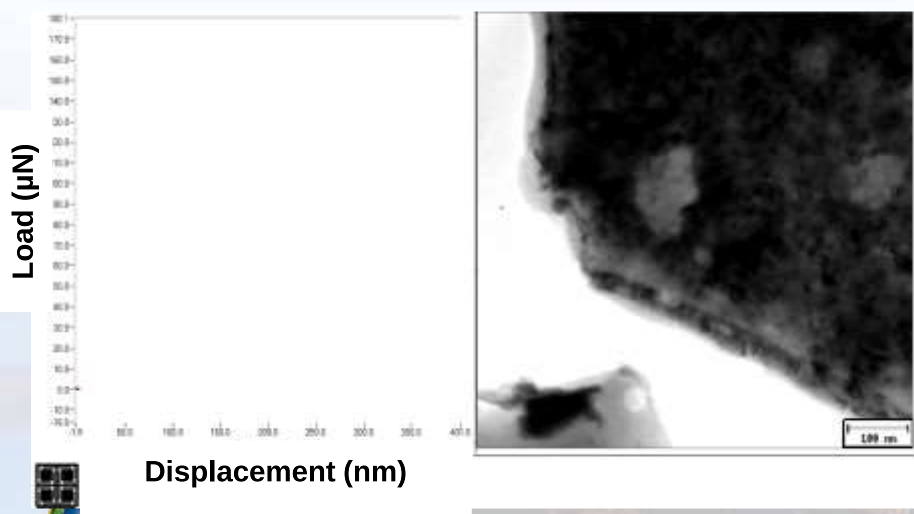




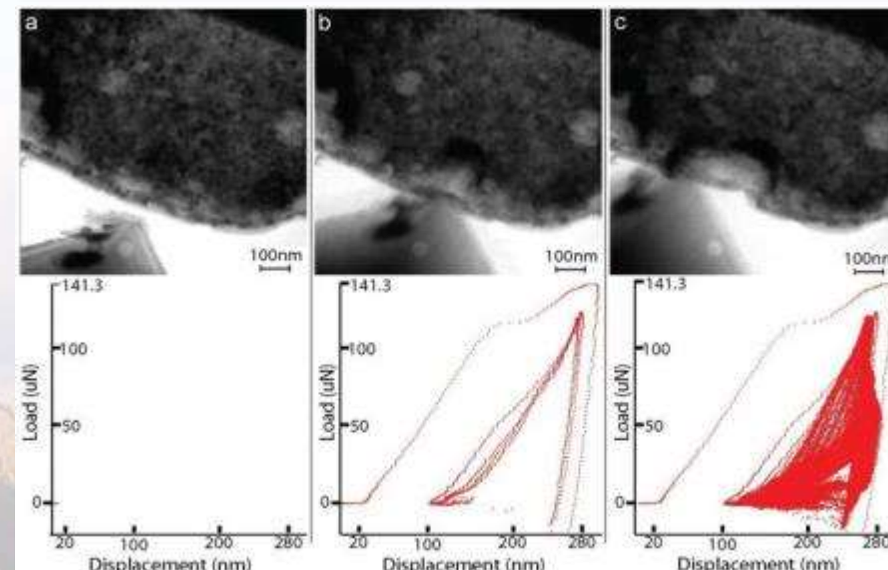
Indentation and fatigue effects

- Associate change in local hardness and fatigue with corresponding nanostructure
- Indent and Fatigue of nanocrystalline Cu film

Single Indents in nc-Cu



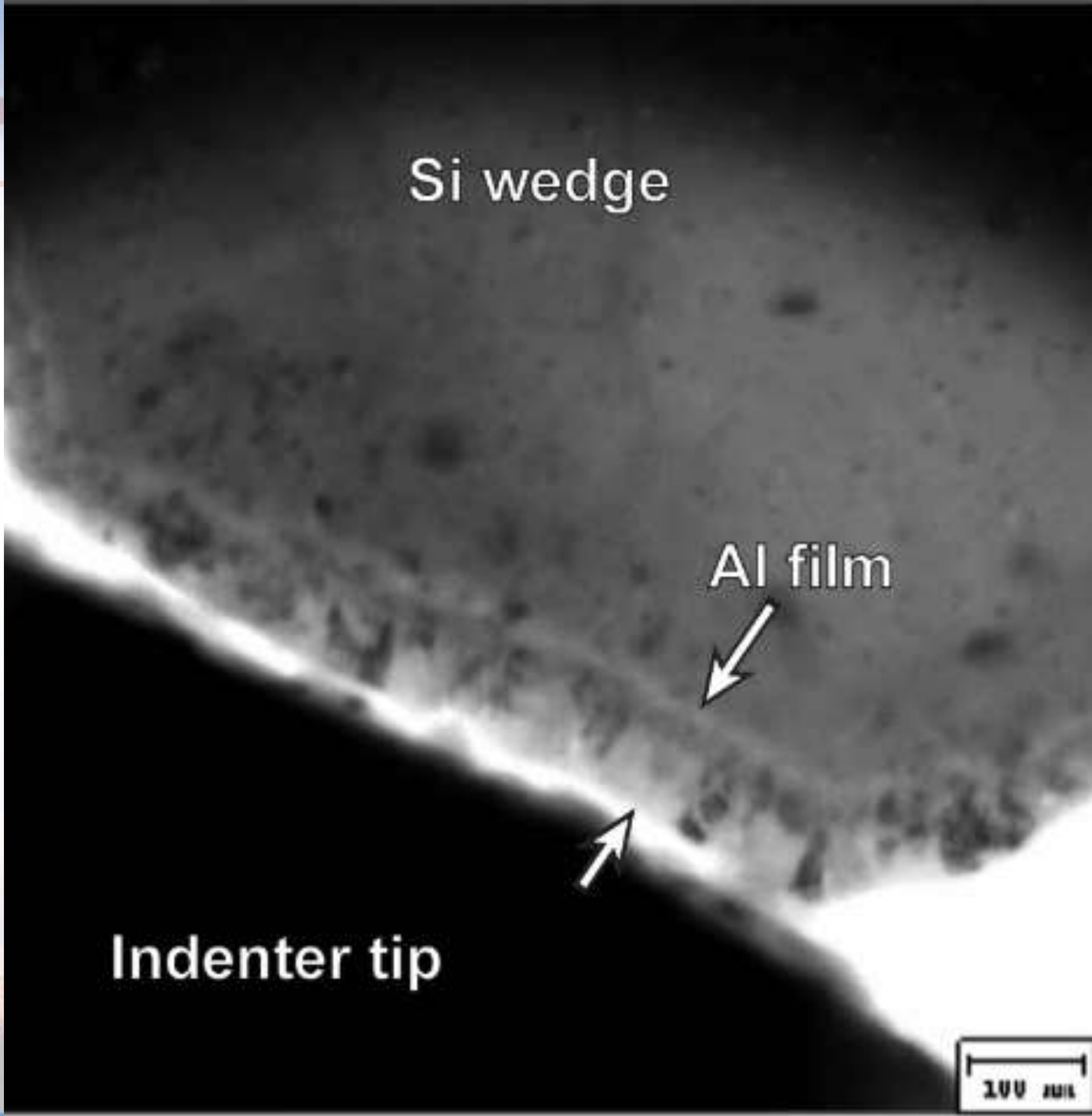
Cycled Multiple Indents in nc-Cu



In Situ TEM Cycling Indentation

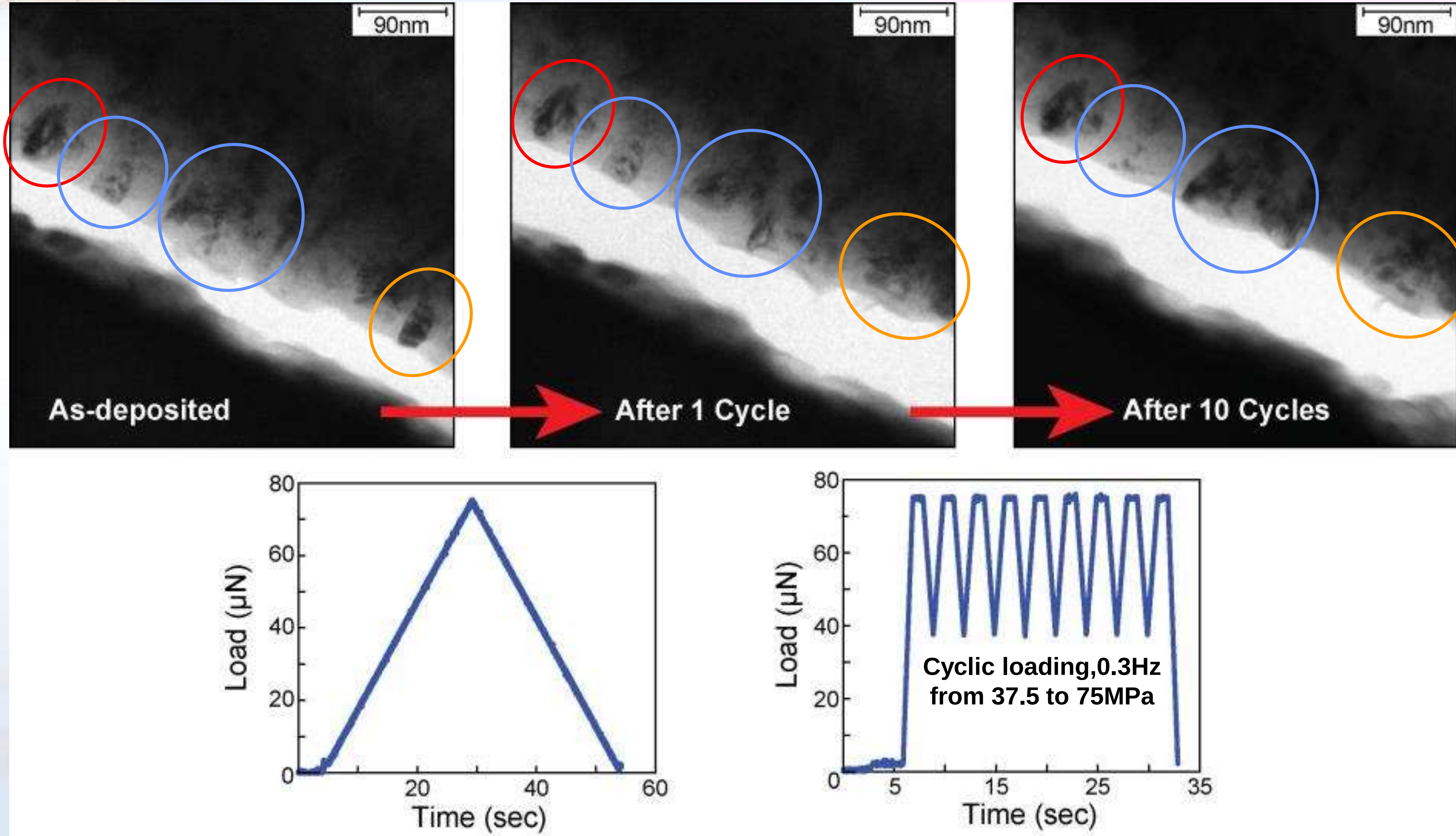
- Low load cyclic loading
- Local asperities play a major role
- Some changes are observed after the first contact
- Others require multiple cycles

In-situ TEM fatigue experiments are feasible and do provide some insight into the structural evolution



No, Monotonic, or Cyclic Evolution

Contributors: J. Sharon, B. L. Boyce



375 MPa nominal contact stress

Summary

Cryo ex-situ indents

- Local texture dictates whether grain growth will occur under cryogenic indentation``

Cryo in-situ TEM indents

- Is feasible
- No abnormal grain growth was observed

RT in-situ TEM indents

- Significant indentation is possible with minimal structural evolution

RT in-situ fatigue experiments

- Asperities play a large role in fatigue process
- Different structural evolution occurs as a function of number of cycles.

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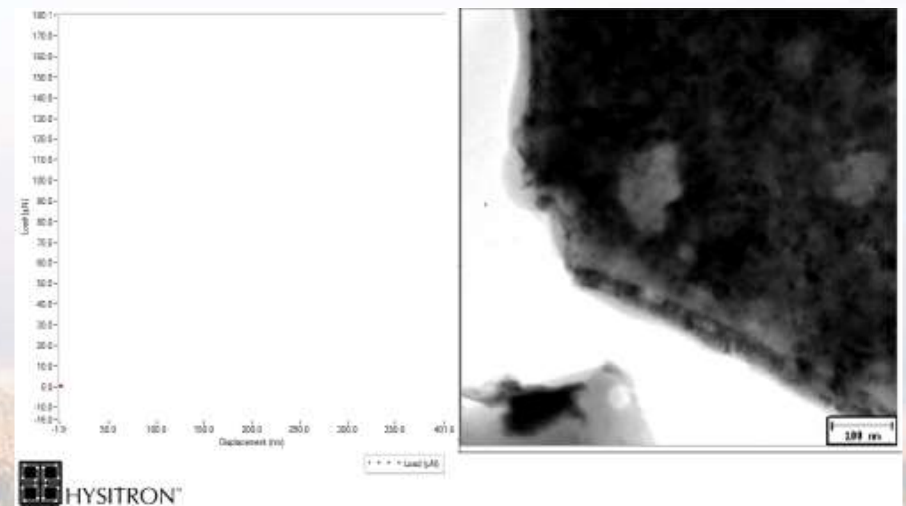

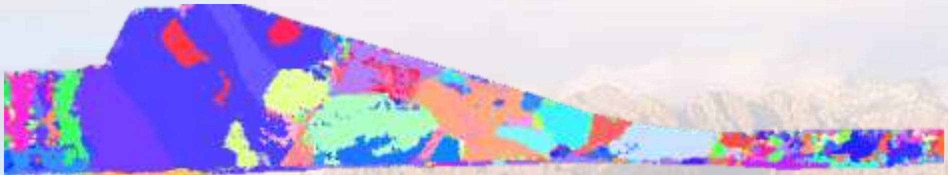

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