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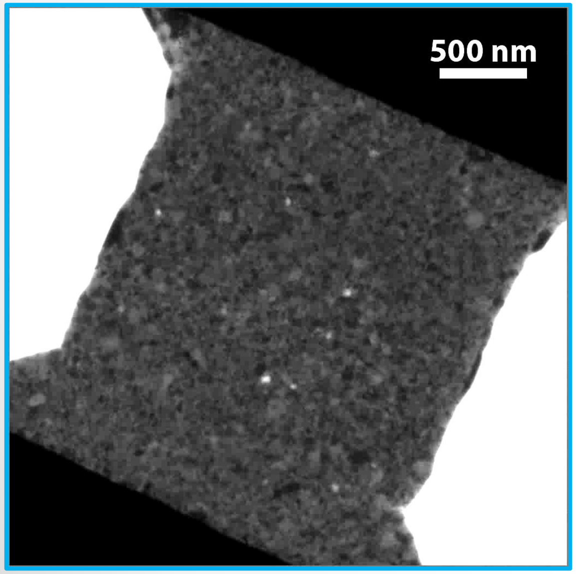
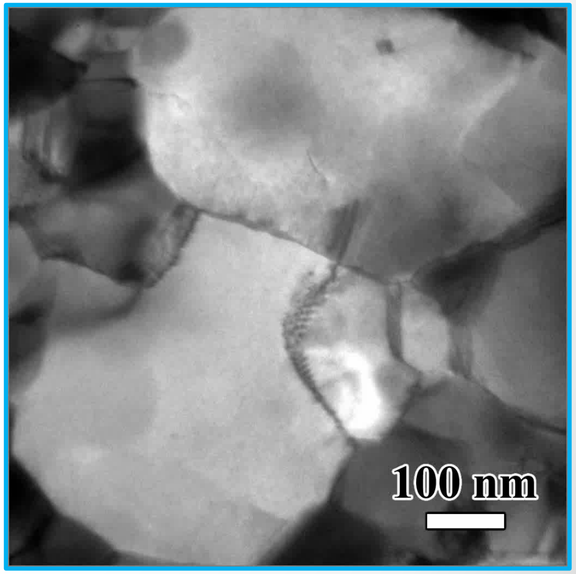
SAND2016-10933PE

Origins of material degradation and failure: real time, nanoscale observations of deformation, fatigue, and radiation damage



Daniel Bufford*
Sandia National Laboratories
Albuquerque, NM, USA

*and many collaborators!



U.S. DEPARTMENT OF ENERGY   NNSA National Nuclear Security Administration

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18 October, 2016

My Background

Sandia National Laboratories

Olin College of Engineering

NATIONAL RECONNAISSANCE OFFICE
UNITED STATES OF AMERICA

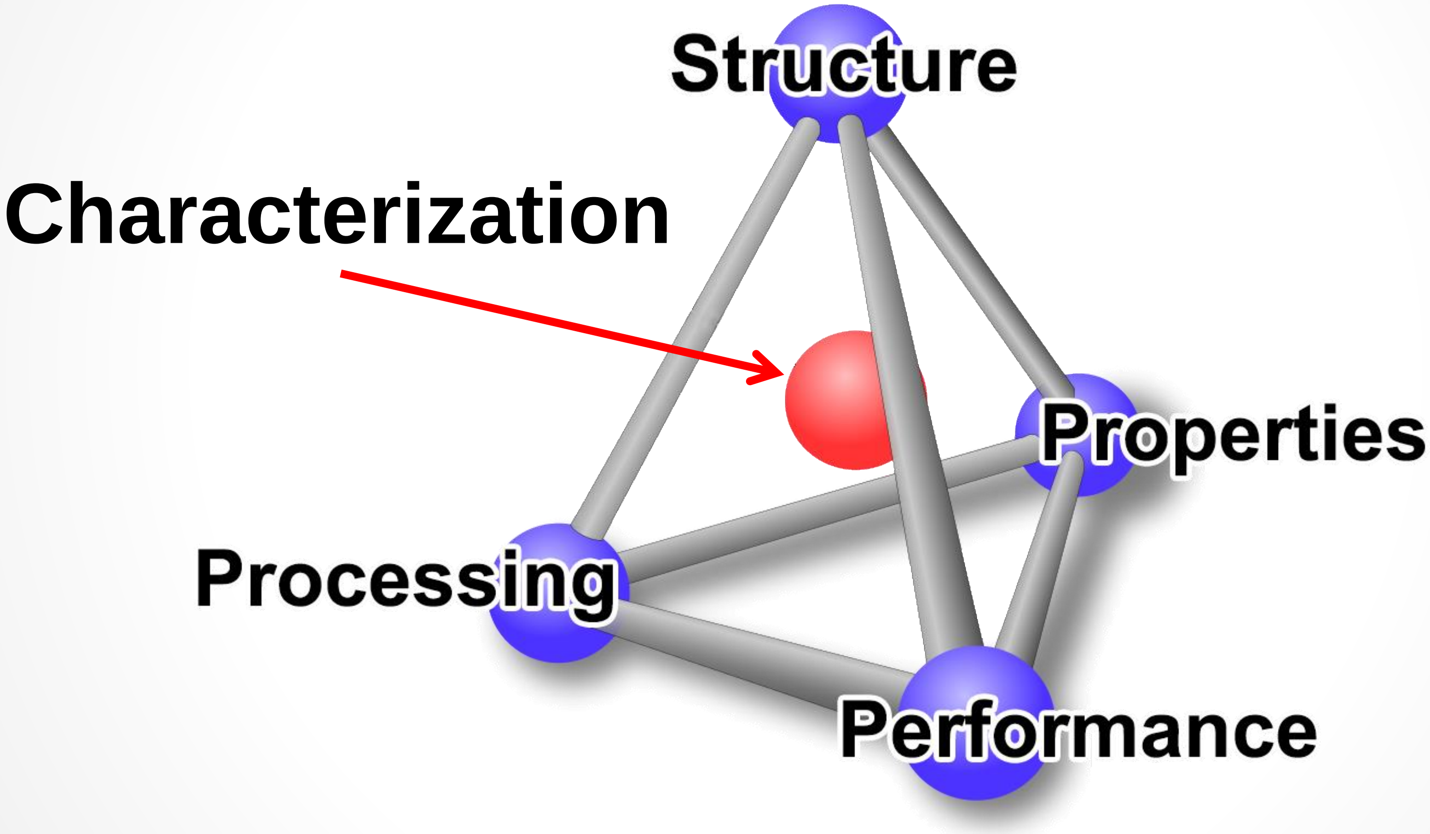
CENTRAL INTELLIGENCE AGENCY
UNITED STATES OF AMERICA

TEXAS A&M UNIVERSITY

Sandia National Laboratories

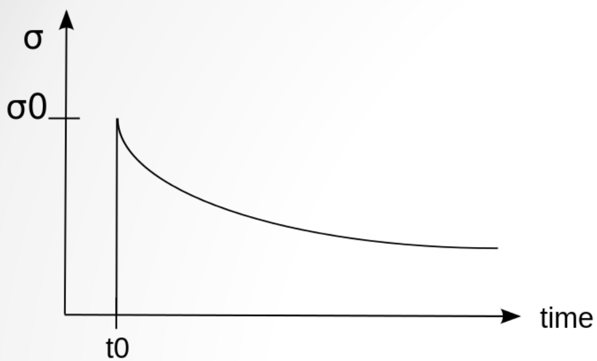
- MRS Graduate Student Silver Award
- Member of MRS, TMS, Microscopy and Microanalysis
- 10 presentations and 2 posters at international conferences
- Visiting scientist at Los Alamos, Sandia, and Stanford

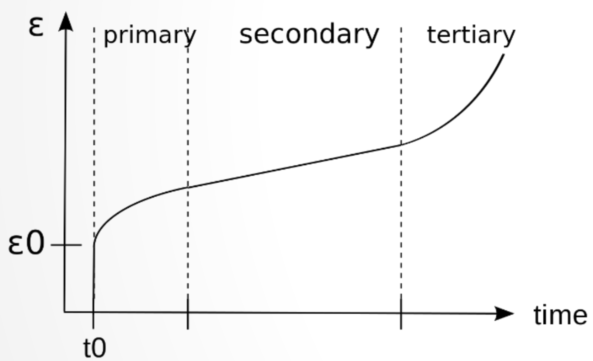
The Heart of Materials Science and Engineering

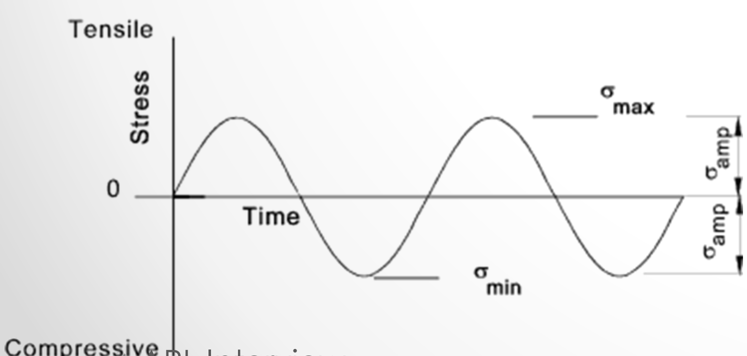


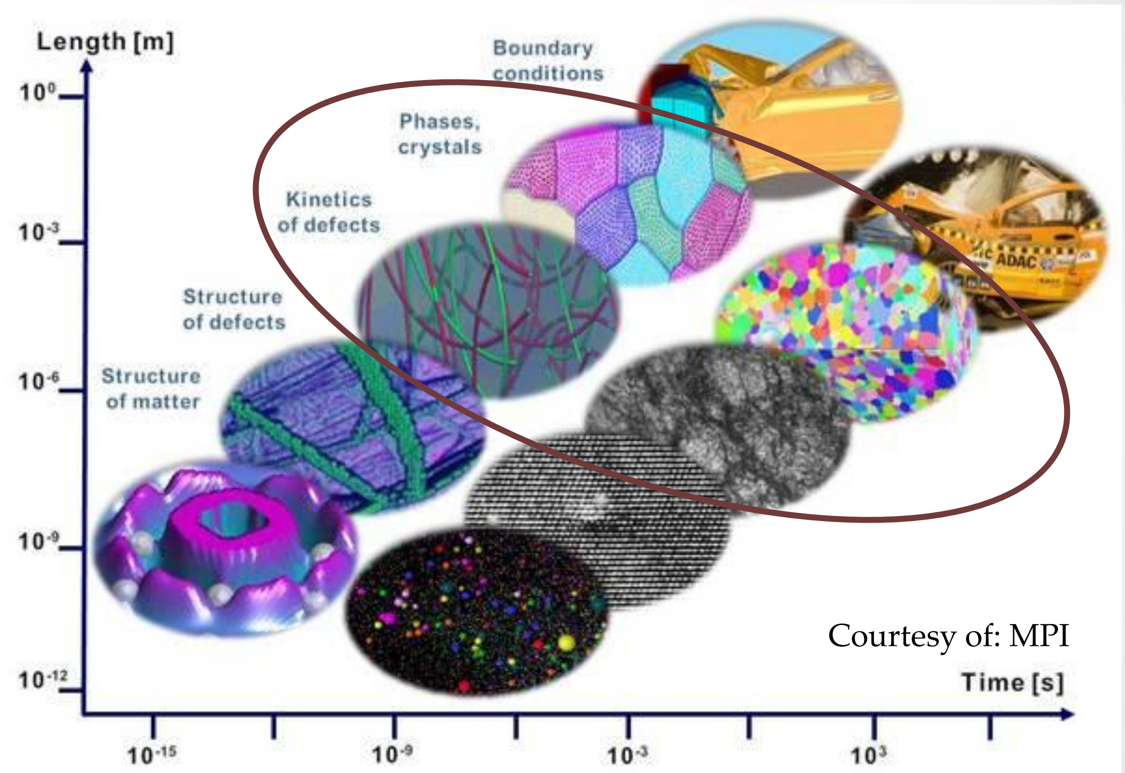
How do we ensure that our material behaves in desired, predictable ways?

Material Evolution Over Time



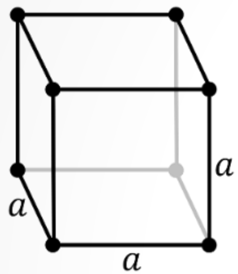




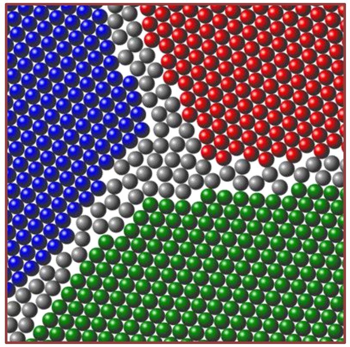


Predictive physics-based models require a fundamental understanding of the structure of matter, behavior of defects, and kinetics of structural evolution in the environments of interest.

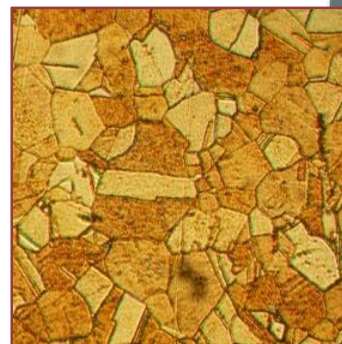
Microstructures



Stannered, 2007, *via* Wikimedia Commons.



Strangerhahaha, 2008, *via* Wikimedia Commons.



Vassil, 2008, *via* Wikimedia Commons.

nm

Tens of nm

Hundreds of nm

Thousands of nm

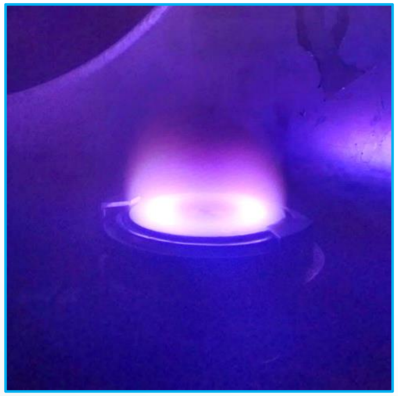
- **How does the internal structure affect material properties?**

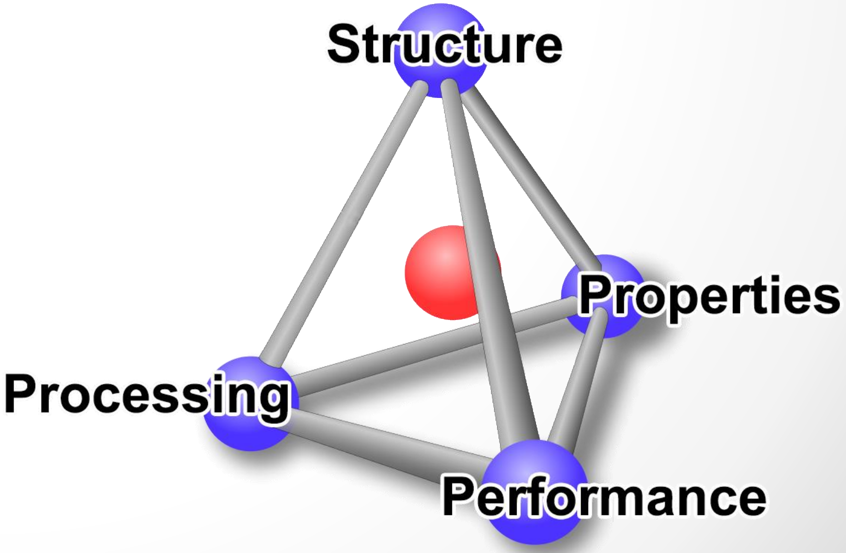
- At the grain boundary level
  - Slip transfer and modification of boundaries?
  - Fatigue-induced grain boundary migration and coarsening?
  - Radiation-induced grain boundary migration and coarsening?

How do we quantify these processes in nanocrystalline metals, and what do we learn in doing so?

Roadmap

- Mechanical Loading
  - Grain boundary – dislocation interactions
  - Nanoscale Fatigue
- Irradiation
  - Grain growth





Nanotwinned metal strength and plasticity

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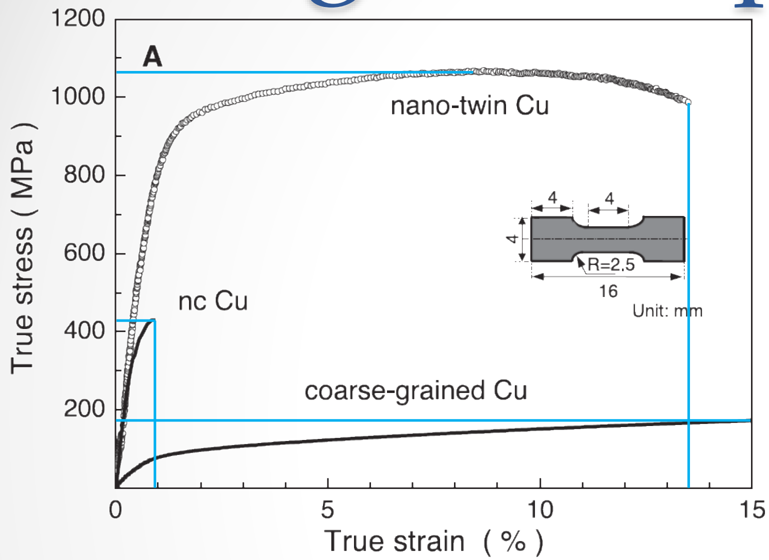
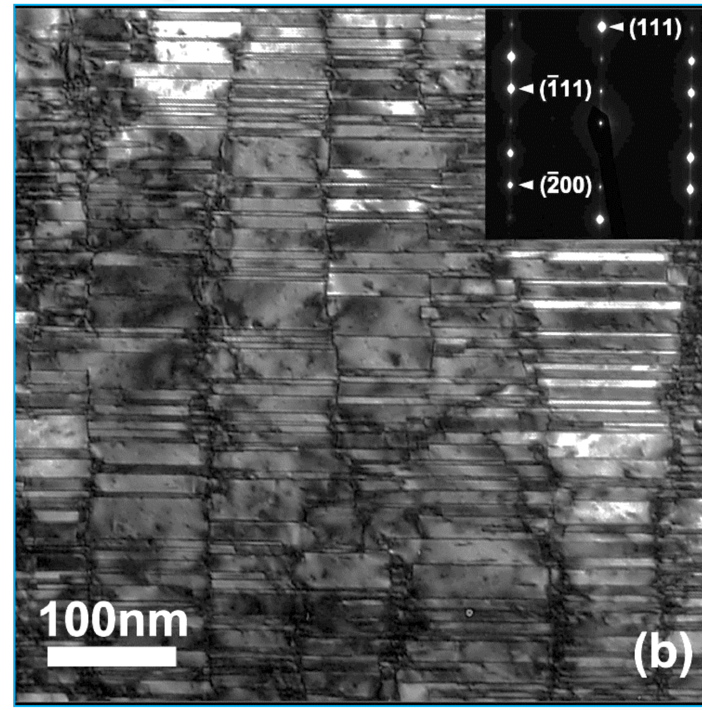
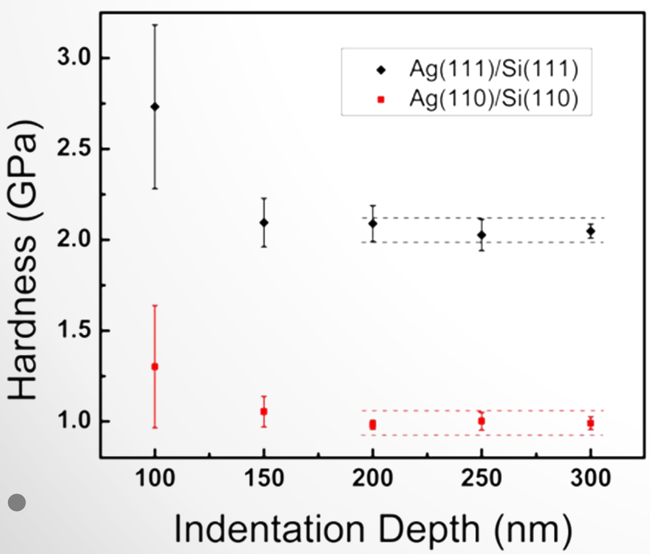


Lu, et al, *Science* 2004.



Bufford, et al. *Acta Mater*, 2011.



- Microstructures dominated by twins spaced <100 nm
- High mechanical strength
- Considerable plasticity

What is happening at the grain boundary level?

Twin vs. Grain Boundaries

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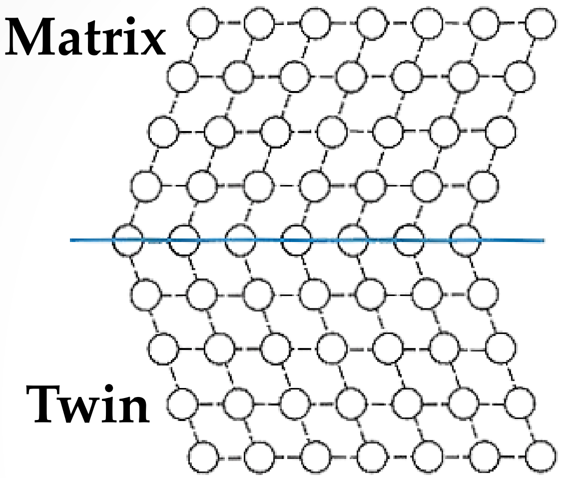
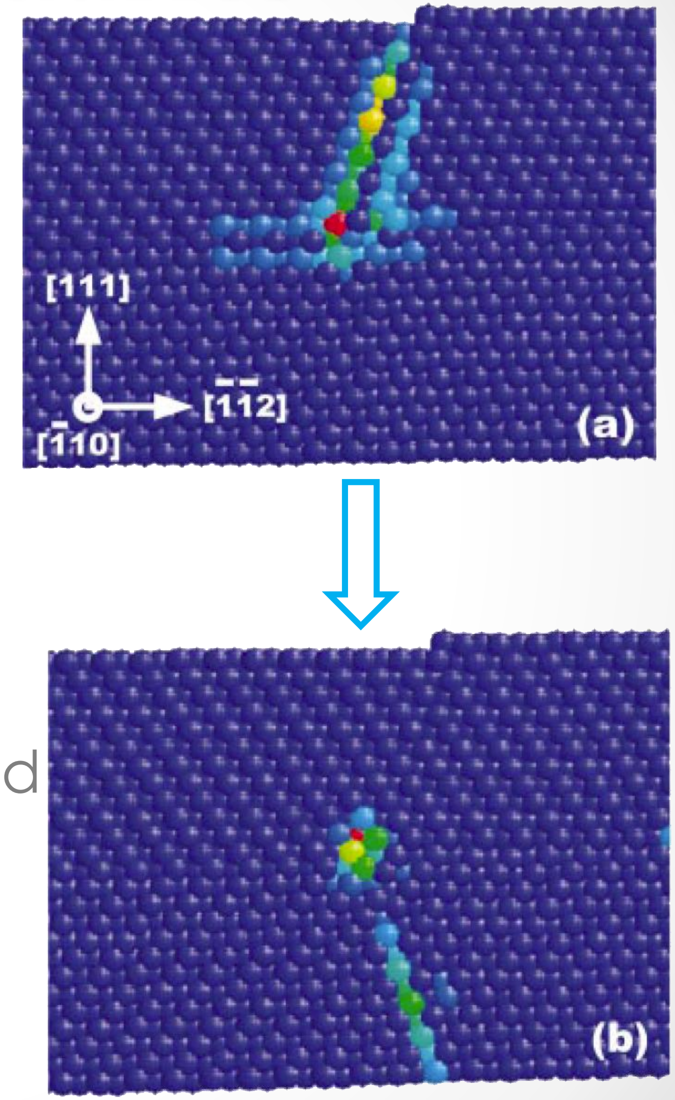


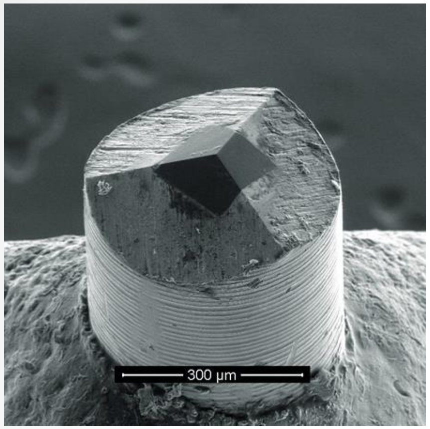
Imperial College Rock Library.

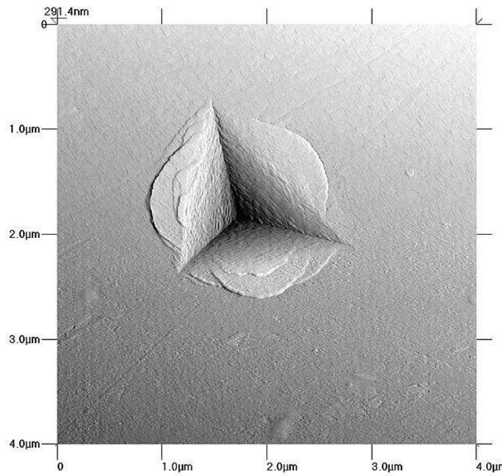


Zhang, et al, *Appl Phys Lett* 2004.

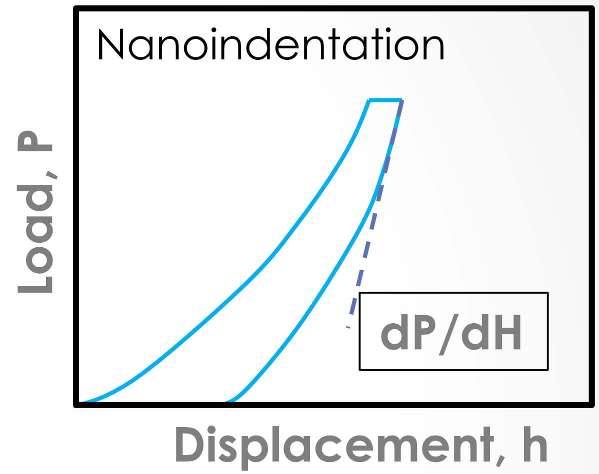
- Twin boundaries are less disordered than most other boundaries
- During simulated deformation:
  - Existing dislocation cross slips at high stress
  - Sessile dislocation left at the boundary

Small-scale testing

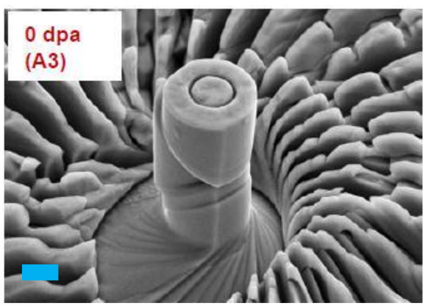


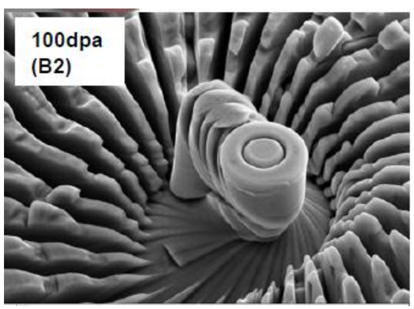


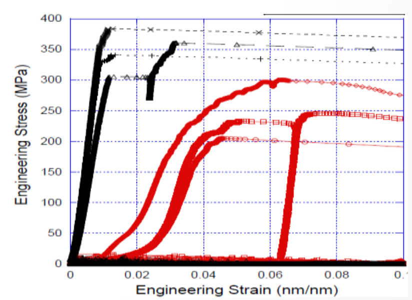
J. Puthoff, *via* Wikimedia Commons.



30 MeV Cu^{5+}
100 DPA







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

Quantifying mechanical response at the nanoscale.

TEM and *In Situ* Experiments

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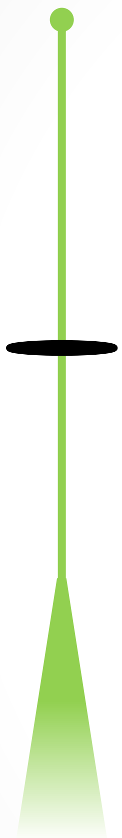
Electron Beam Generation

Sample Interaction

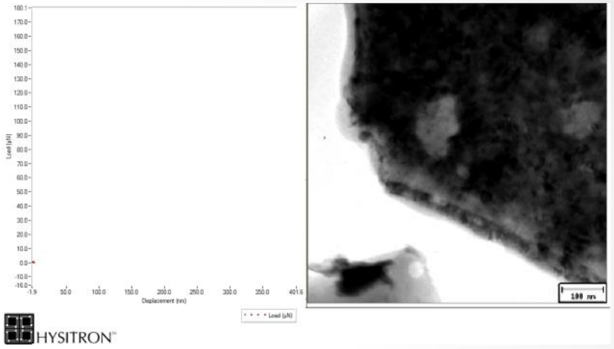
Magnification



Sub-nm imaging
Electron diffraction

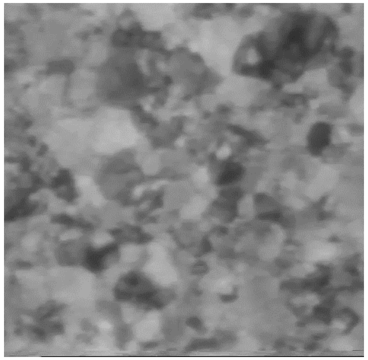
Mechanical

Direct correlation of loading with changes in strength, ductility, and defect mobility



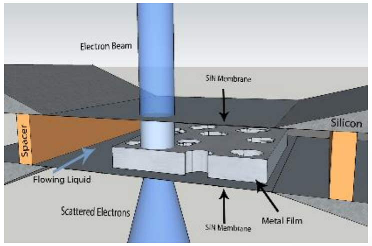
Thermal

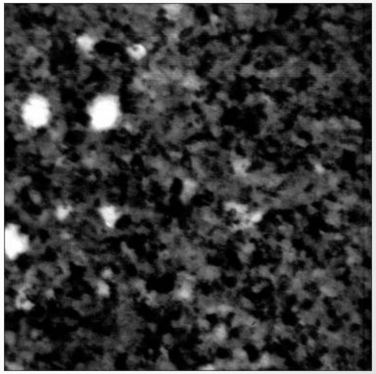
Effects of temperature on microstructural evolution up to 800 °C



Environmental

Effects of corrosion and gas loading at the grain level





- **Limited to appropriate samples**
- **Enables real-time studies of samples under various stimuli**

In Situ Nanoindentation

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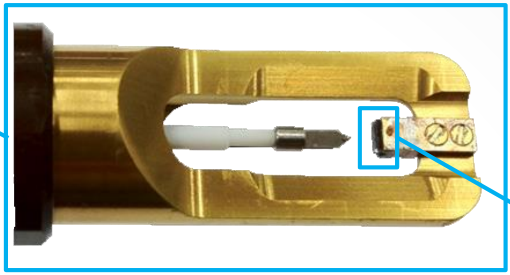
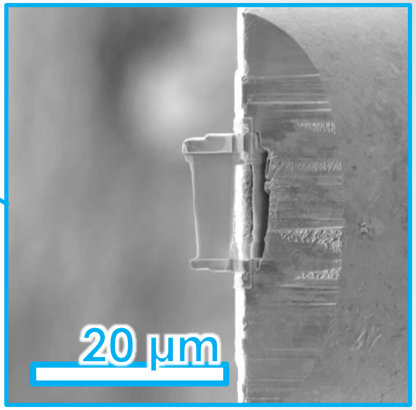
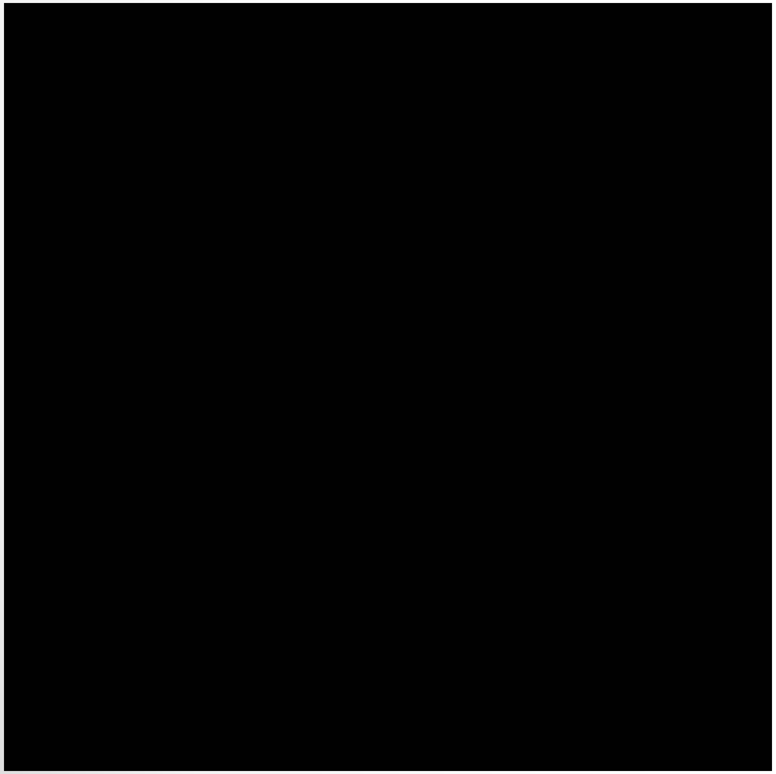
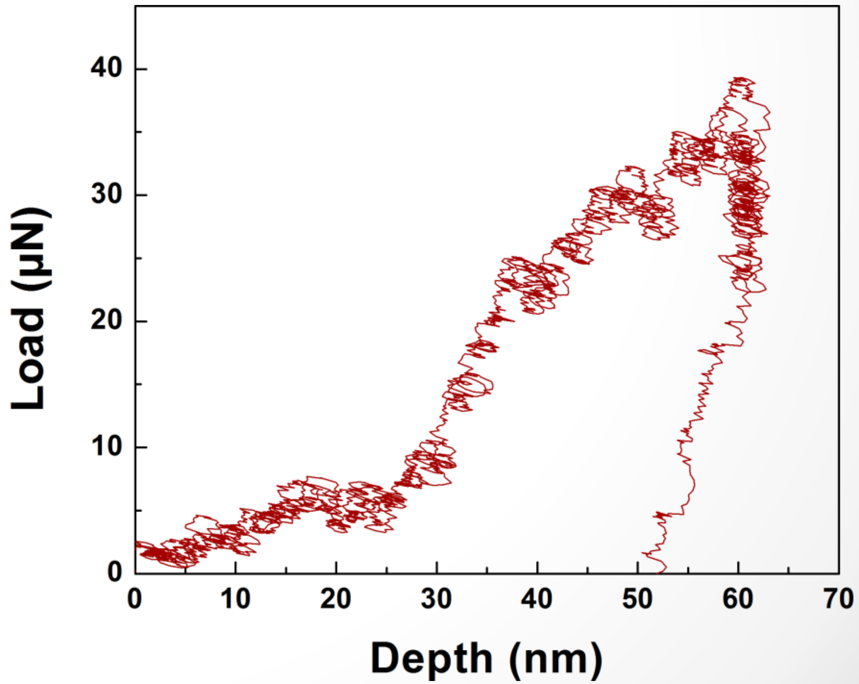




Hysitron PI95 *In Situ* Nanoindentation TEM Holder
- Sub nanometer displacement resolution
- Quantitative force information with µN resolution



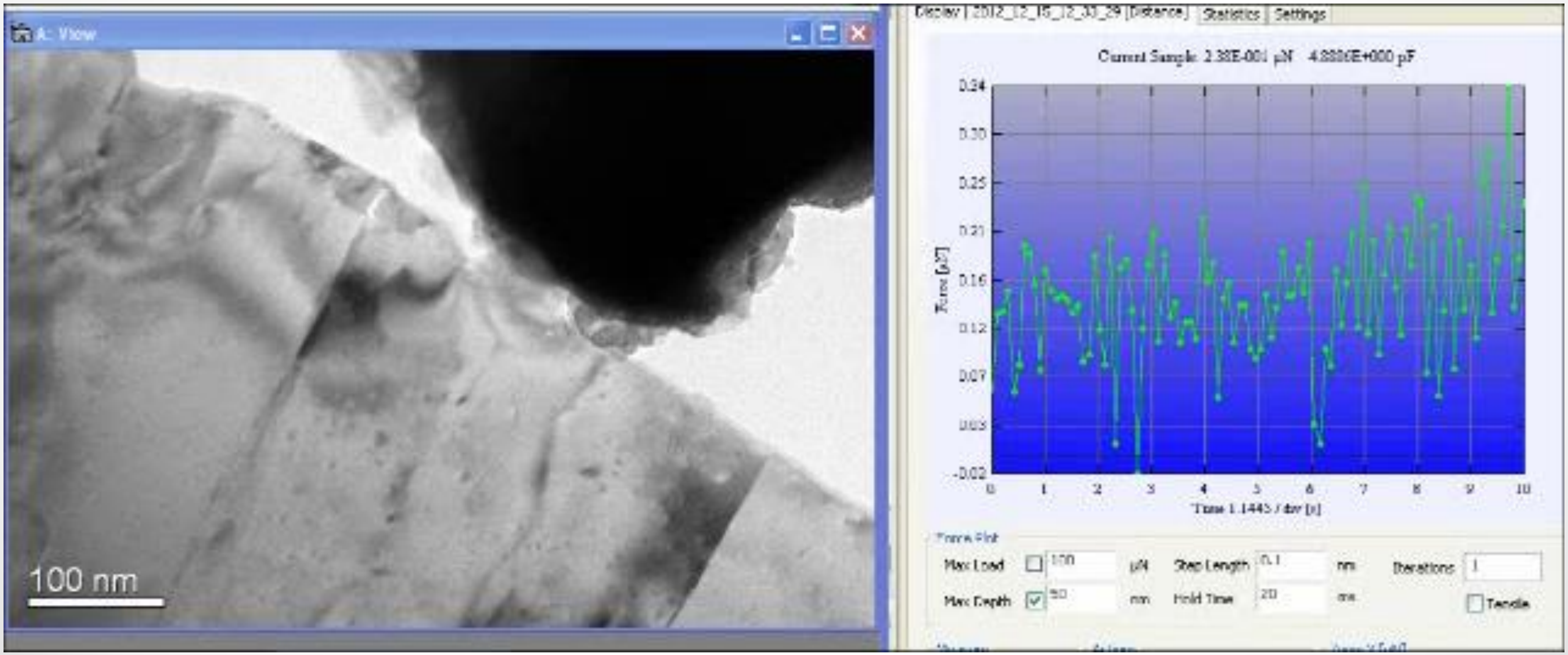


Correlation of real-time imaging and quantitative mechanical response.

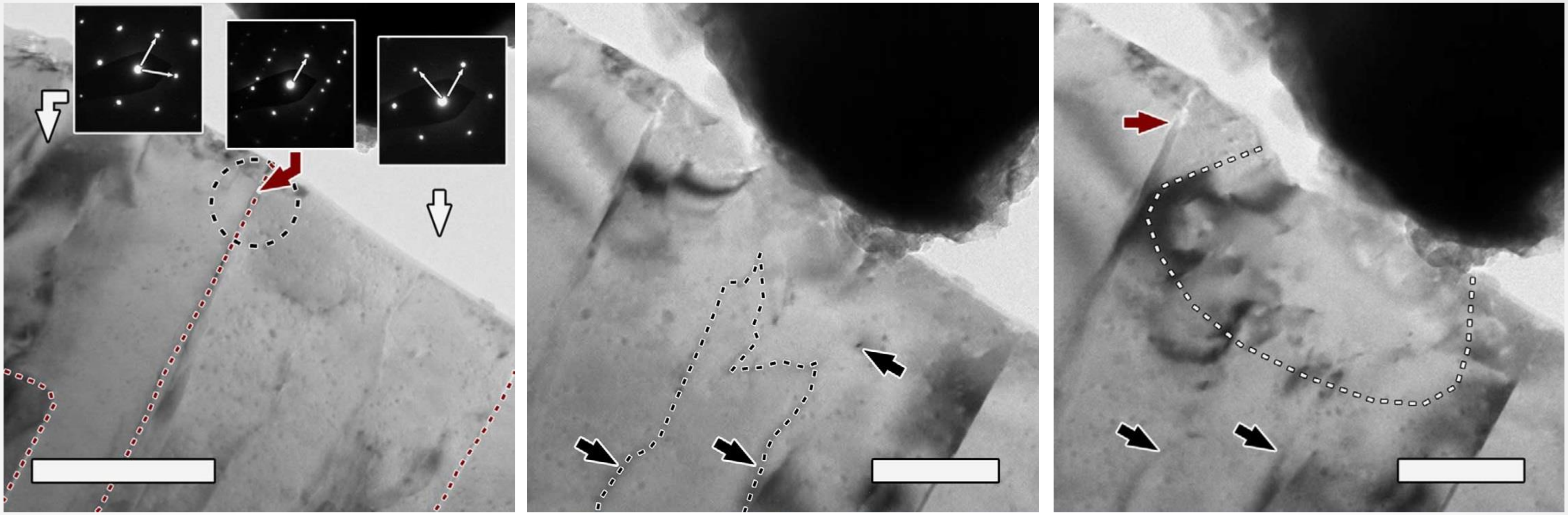
Plasticity in Al

- *In situ* nanoindentation of Al near a Σ3{112} twin boundary
- Initial cycle

Video playback ×3



Plasticity in Al



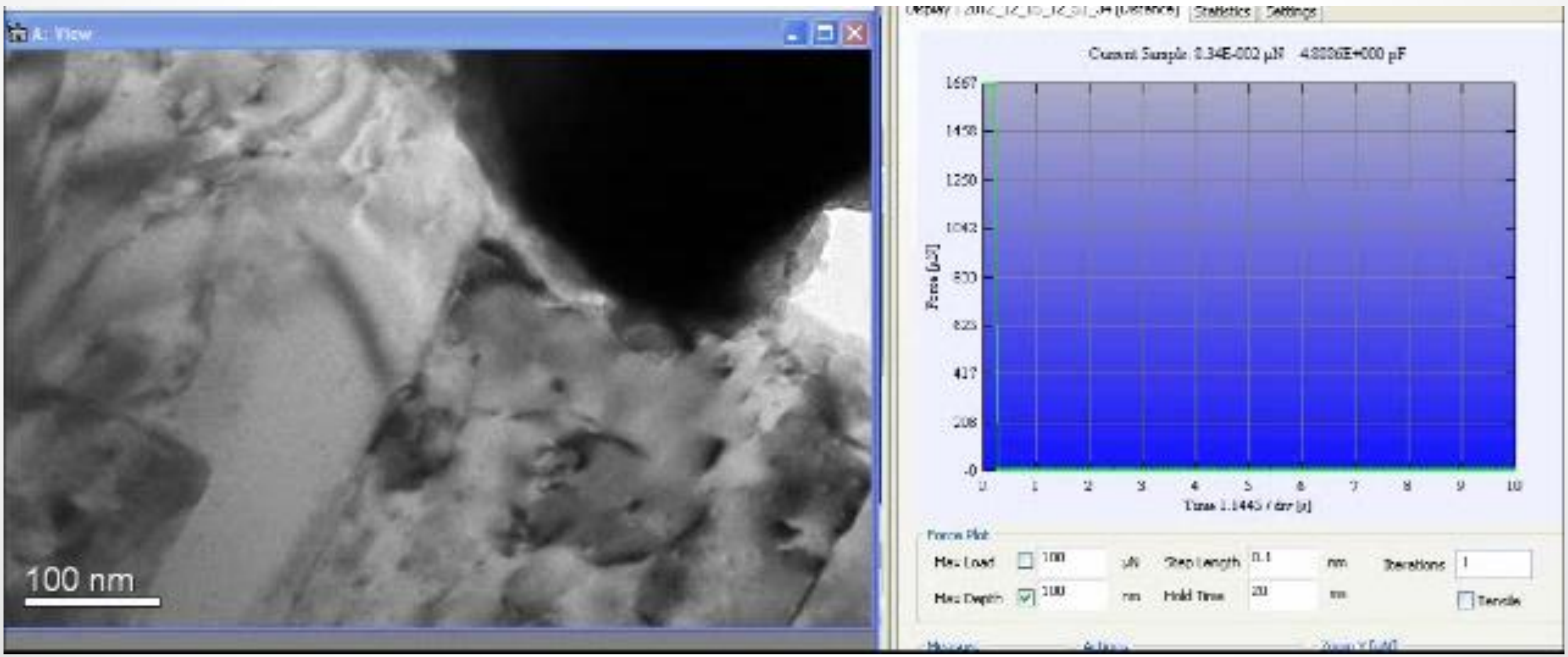
- Grains initially mostly free of large defects
- Movement of existing dislocation observed
- Deformation confined by twin to single grain

Confinement of dislocation activity suggests barrier (Hall-Petch) strengthening.

Dislocation Transmission

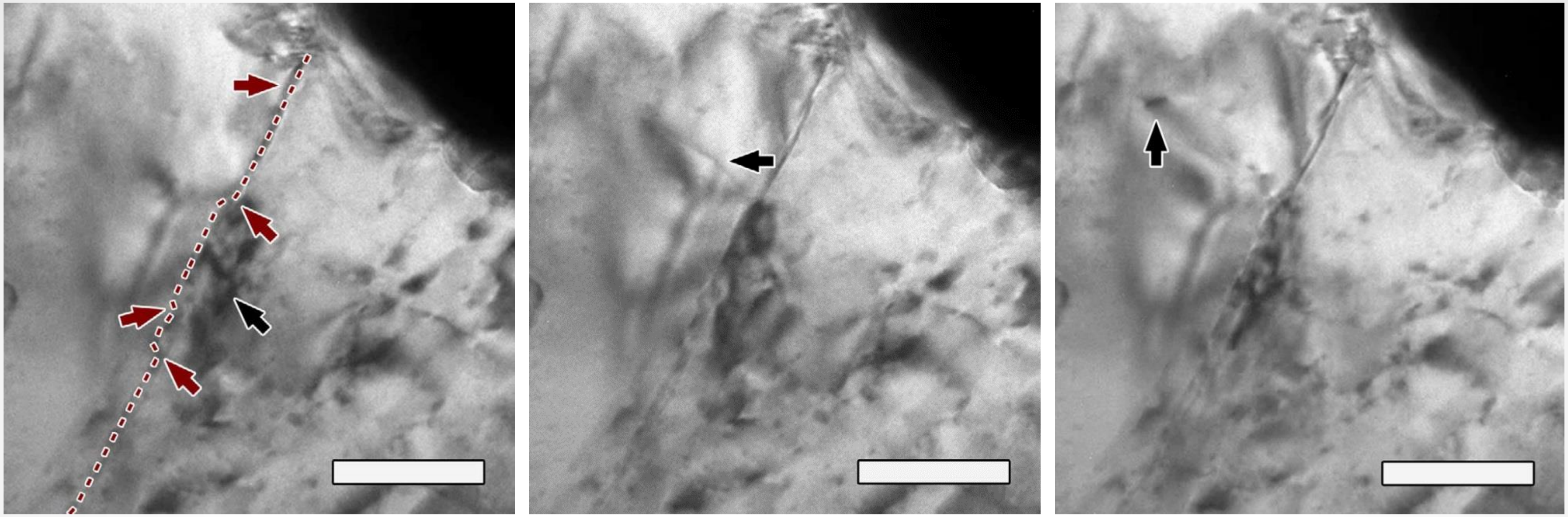
- In situ nanoindentation of Al near a Σ3{112} twin boundary
- Cycle #4, after 3 previous cycles to progressively higher loads

Video playback ×3



Dislocation Transmission

Sandia National Laboratories



Bufford, *et al.*, Nat Commun 2014.

- Boundary deformed by dislocation interactions in previous cycles
  - But no obvious plastic deformation in adjacent grain
- First *observable* plasticity event in second grain captured
- Measured forces associated with the event

Known bicrystal geometry and quantitative force measurements provide bounds for determining likely dislocation reactions and estimating associated local stresses.

Nanotwinned metal strength and plasticity

Sandia National Laboratories

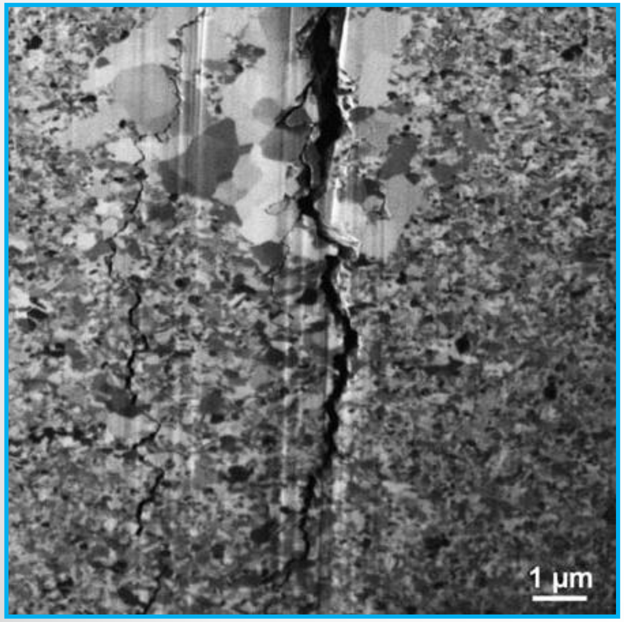
- Grain boundary structure change resulting from dislocation pile-up
- Dislocation transmission appears to be spatially correlated with defects formed in the boundary

Fatigue-induced grain growth

Sandia National Laboratories

- Progressive microstructural change with cyclic loading

Execcharter, 2011.



Padilla and Boyce, Exp Mech 2006.

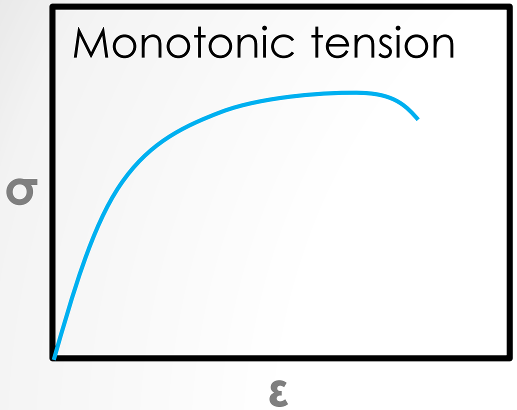
- Fatigue in nanocrystalline metals
  - Grain boundary migration and grain growth
  - Crack initiation
- Substantial hurdle to practical adoption of these materials

What are the underlying mechanisms associated with these phenomena?

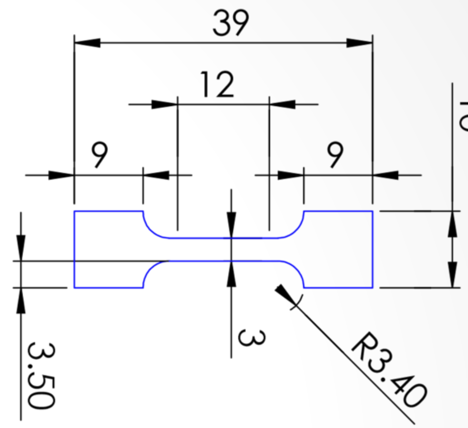
Plausible mechanisms

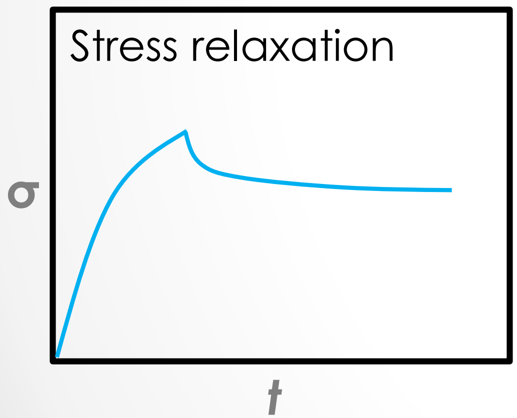
- Grain boundary migration
- Pre-deformation microstructure
- Grain and grain boundary orientations

Tensile Testing

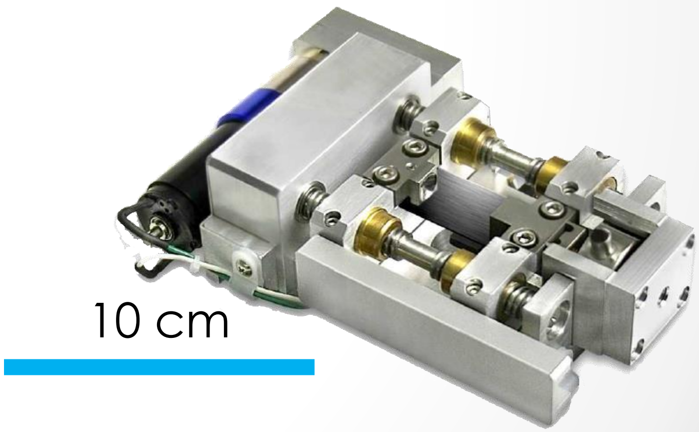


- Lots of information:
  - E, σ_y, σ_{UT}, elongation, toughness, n, m





- More information:
  - m, ΔV, creep

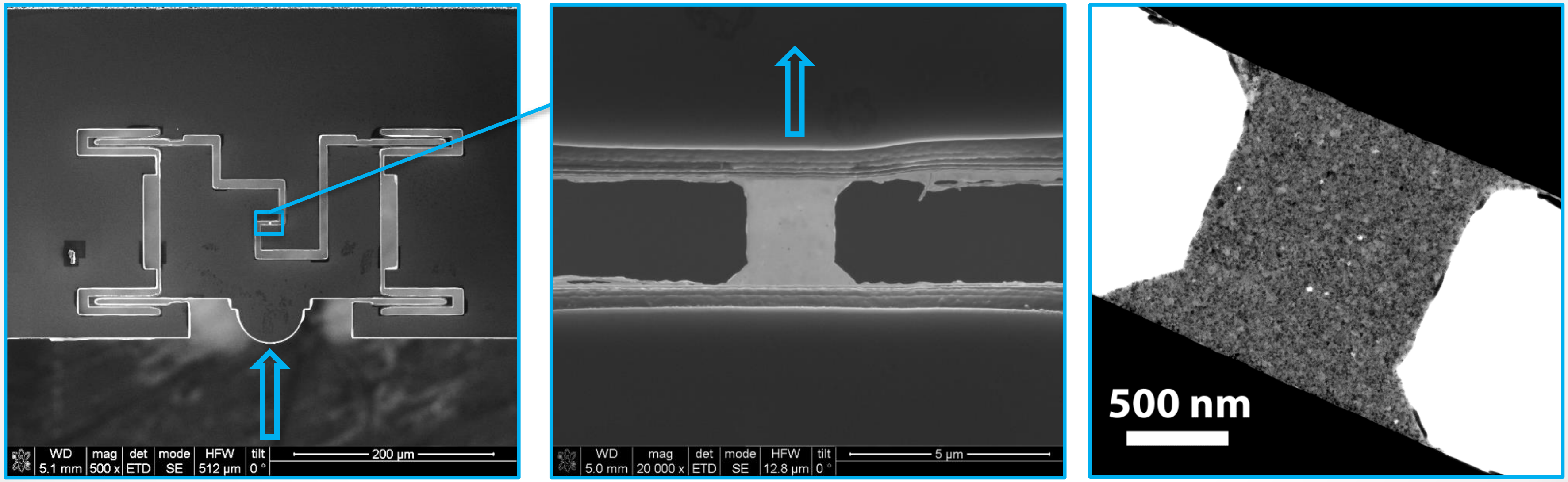


- **Requires well formed specimens**
- **Gold standard for mechanical properties**

Small-Scale Tension Specimens

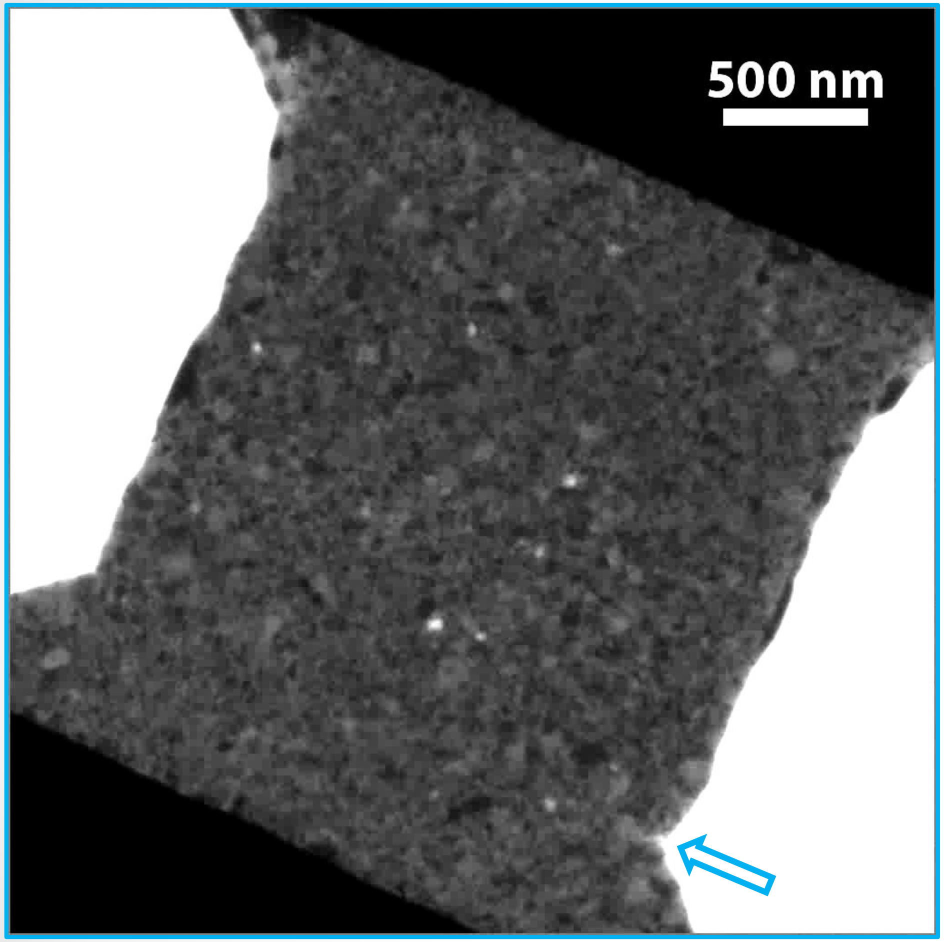
- Hysitron "Push-to-Pull" devices
  - Microfabricated Si test frame
  - Cu film (75 nm) floated onto device, then FIB milled

Collaborators: D. Adams, K. Hattar, W. Mook, C. Sobczak



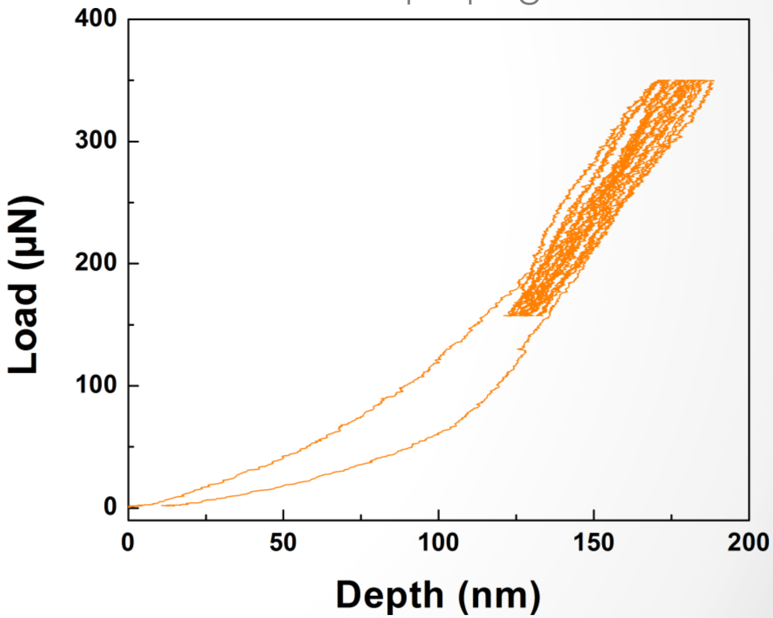
- Thin foil geometry not ideal for mechanics, but is electron-transparent
- Nearly pure tension, uniform cross sectional area, stable load frame

Cyclic Tension *In Situ*



Video playback ×10

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



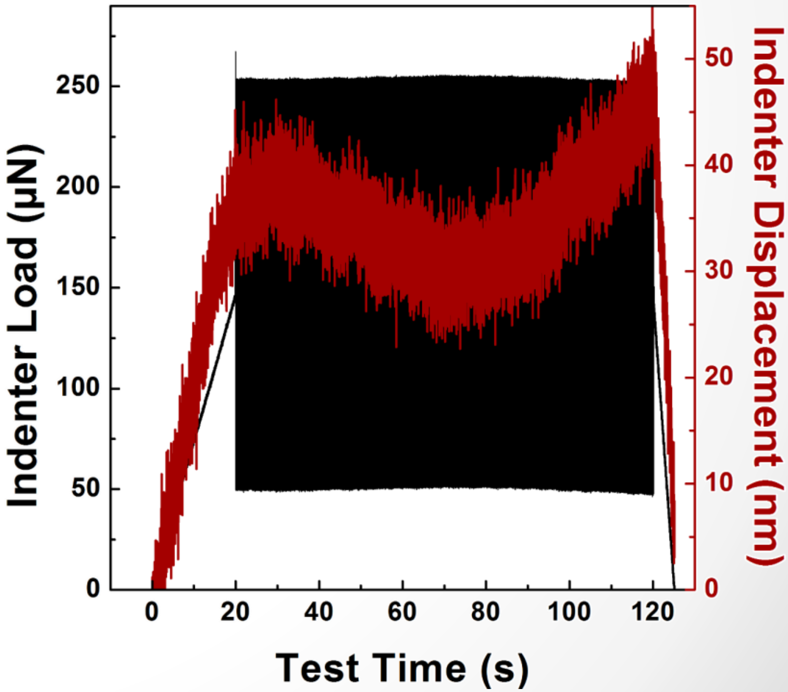
- **Direct measurements of crack growth**
- **Little structural evolution at the crack tip**

High Cycle Fatigue *In Situ*

Collaborators: D. Stauffer, B. Boyce, K. Hattar, W. Mook

Video playback ×3

- Cyclic loading:
  - 200 hz
- Structural change at crack tip captured



First high-cycle fatigue in the TEM!

Crack Growth Quantified

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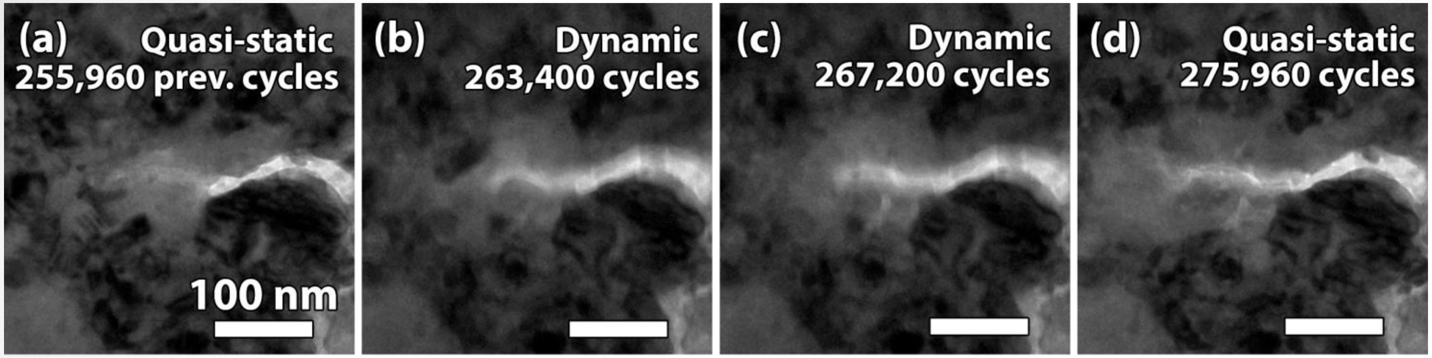
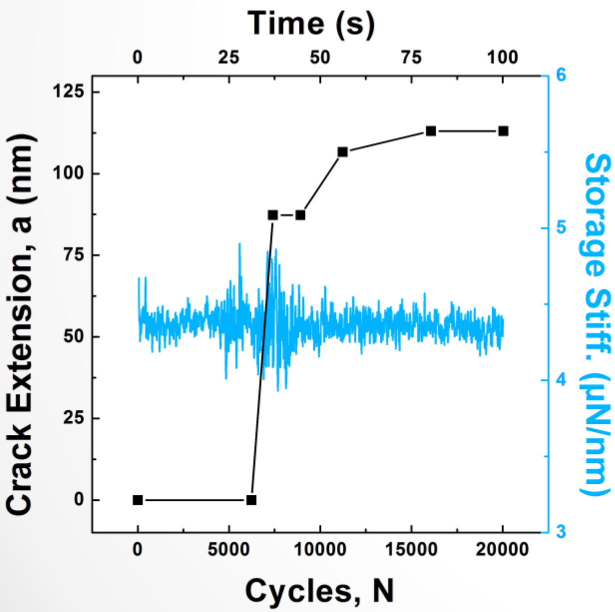
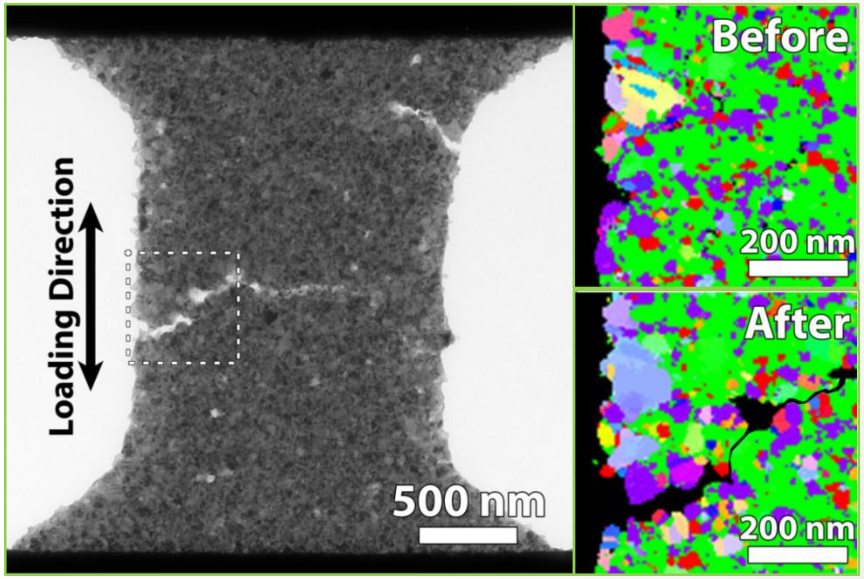


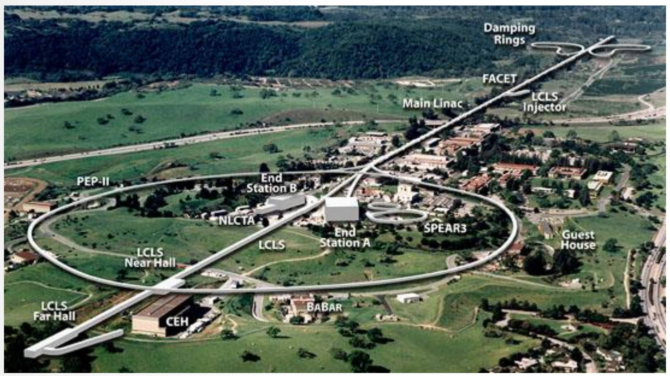


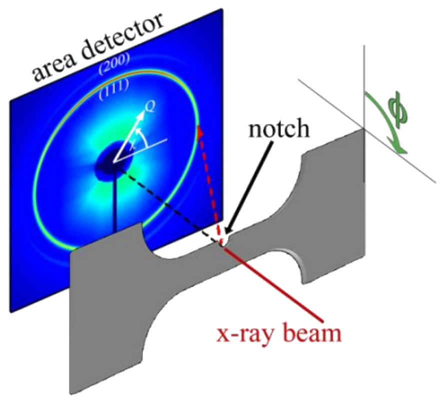


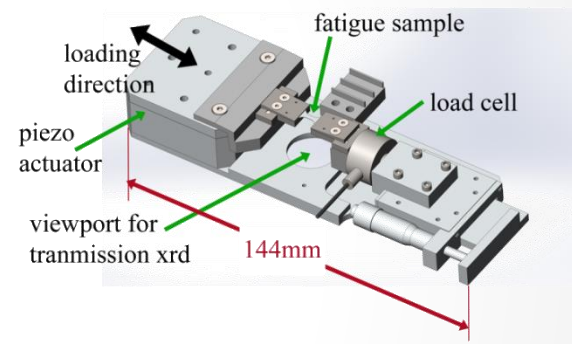
- Average crack growth rate measured at 6×10^{-12} m/cycle!
- Evidence of fatigue-induced grain growth.

Moving up in length scale

Collaborators: A. Mehta, D. Van Campen, T.A. Furnish, B.L. Boyce







- Fatigue performed *in situ* during X-ray diffraction analysis
- Experiments performed at the Stanford Synchrotron Radiation Lightsource (SSRL)

Which comes first, grain growth or crack initiation?

Moving up in length scale

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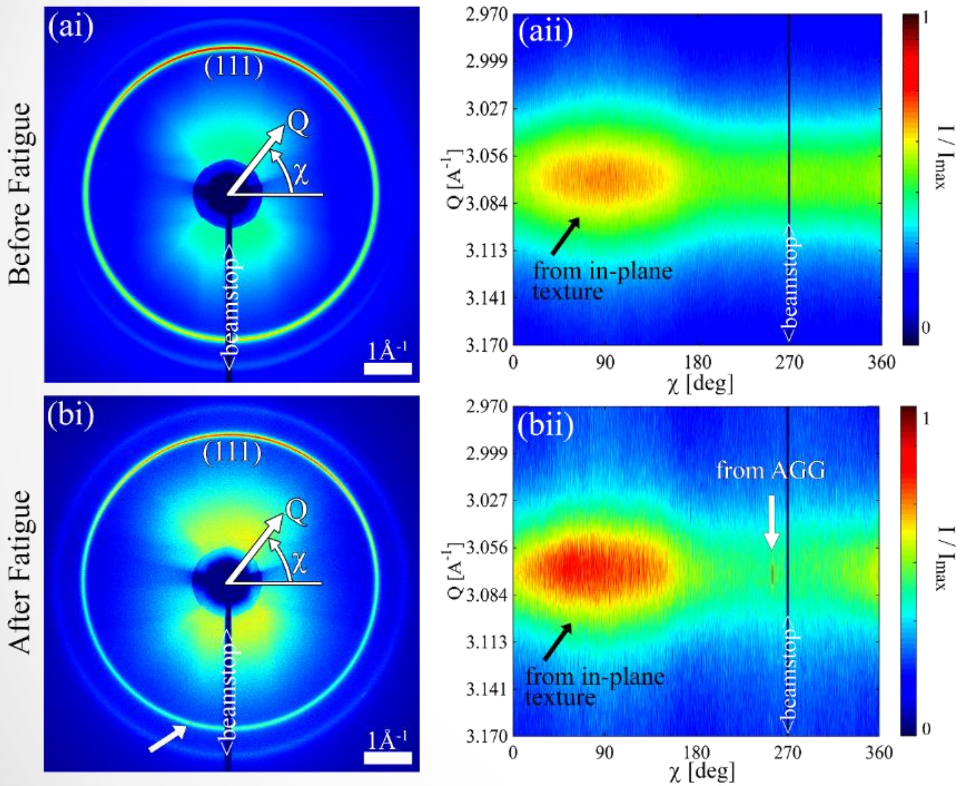
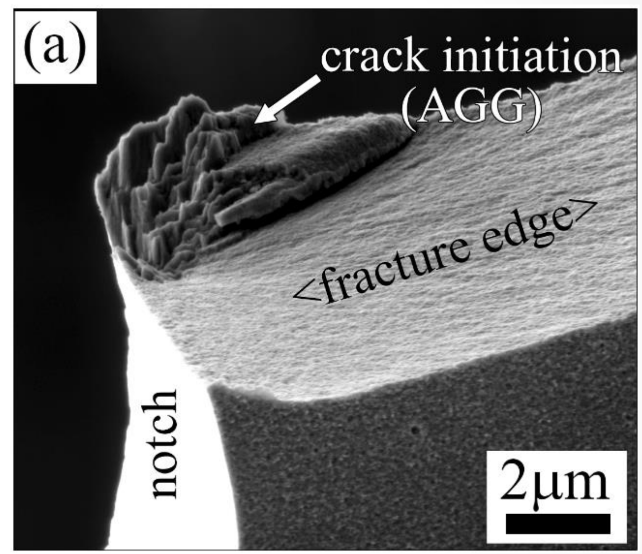
Collaborators: A. Mehta, D. Van Campen, T.A. Furnish, B.L. Boyce





- Patterns processed and analyzed for outliers
- Fatigue-induced grain growth captured before failure

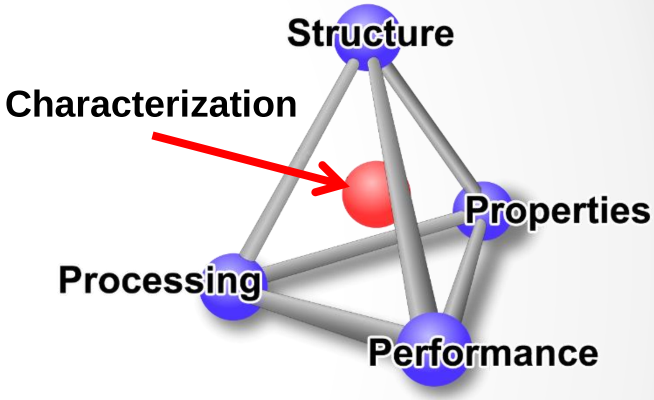
- **Detection of abnormalities in only ~0.00001% of the sampled volume!**
- **Analysis of fracture surface indicates that grain growth preceded crack initiation**

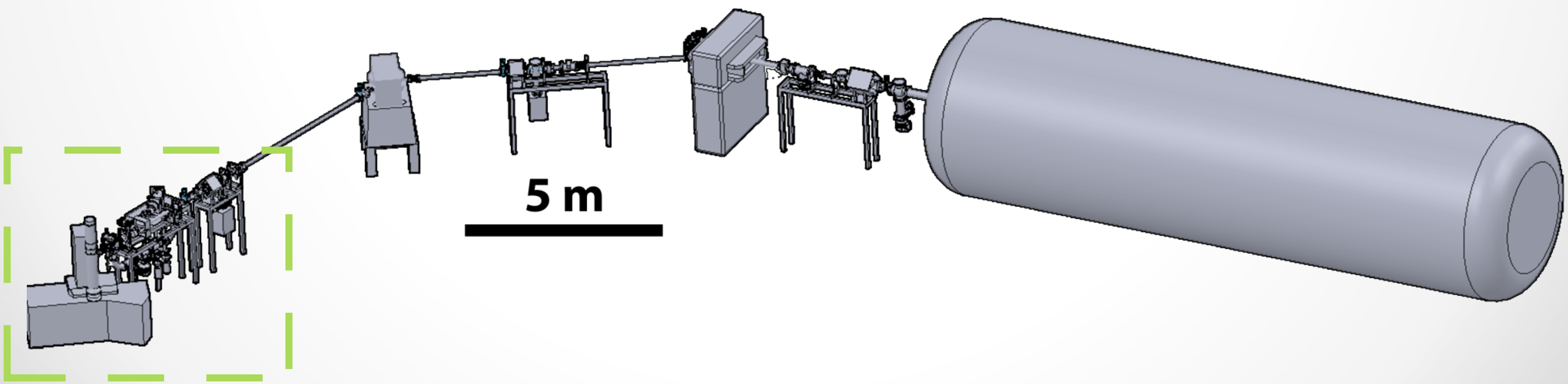
Fatigue-induced grain growth

- Demonstrated capabilities for capturing local fatigue-induced structural changes as they occur
- Ongoing work to reveal orientation dependencies at the grain boundary level

Roadmap

- Mechanical Loading ✓
  - Grain boundary – dislocation interactions
  - Nanoscale Fatigue
- Irradiation
  - Grain boundary migration





Radiation Tolerant Materials

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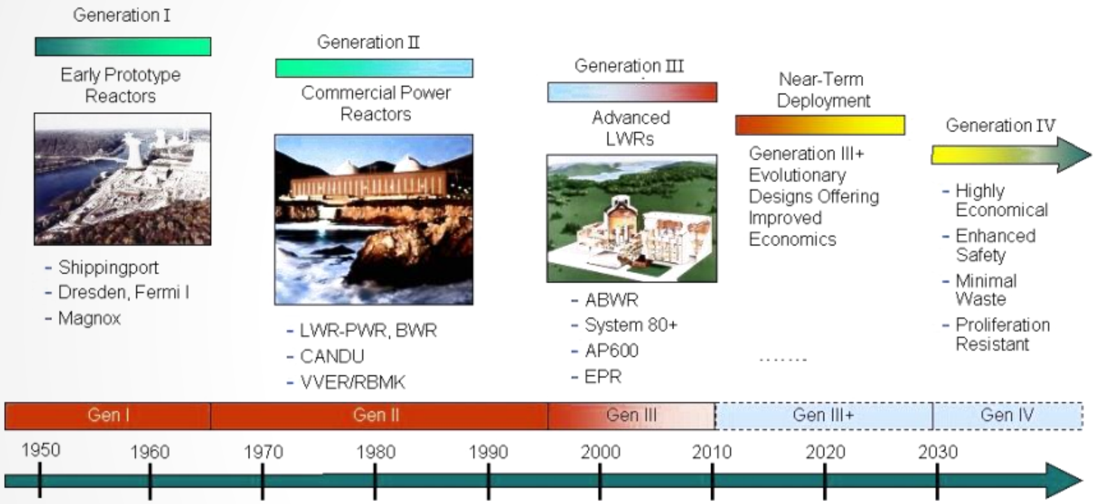
Generation IV: Nuclear Energy Systems Deployable no later than 2030 and offering significant advances in sustainability, safety and reliability, and economics



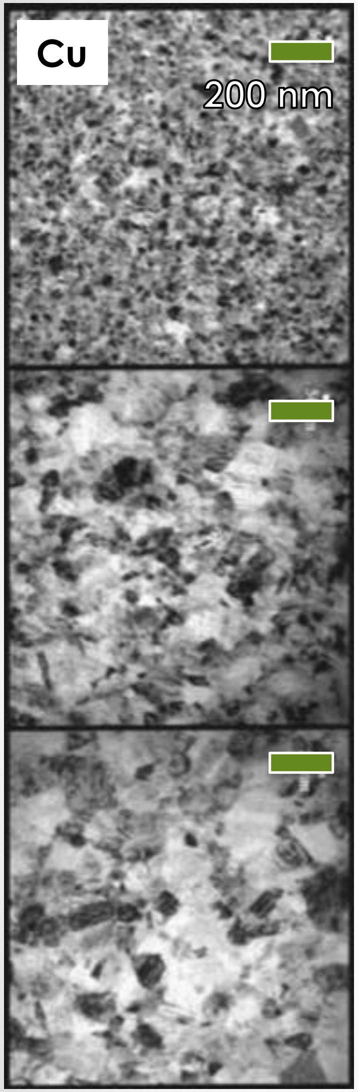
Argonne National Lab, *via* Wikimedia Commons.

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

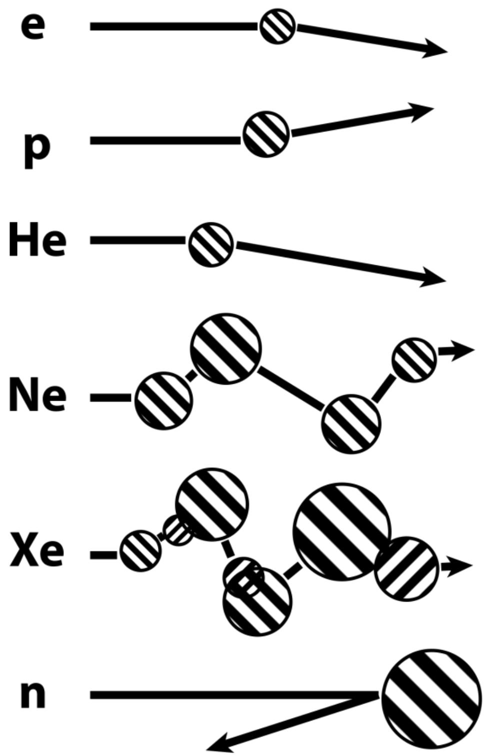
Nanocrystalline metals

- Exemplary mechanical properties
- Abundant sinks for structural and chemical defects
- Ideal candidates for radiation-tolerant materials?

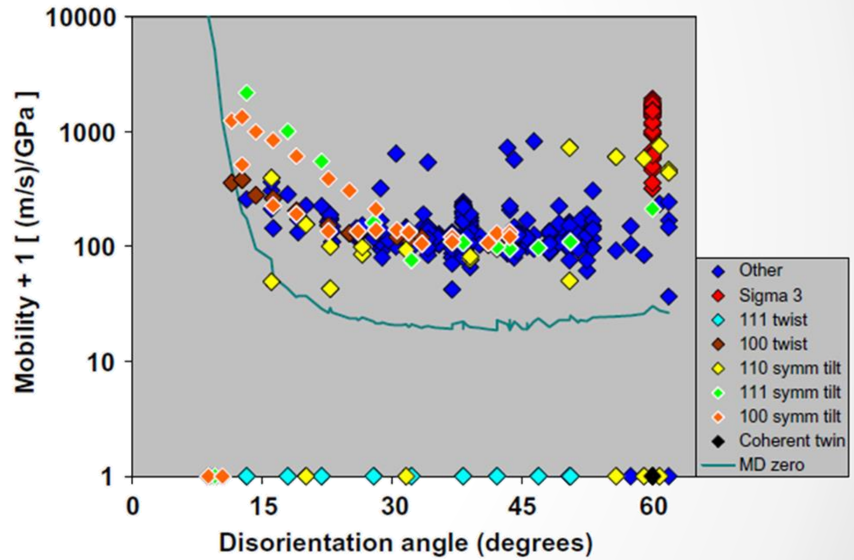
Radiation-Induced Grain Growth



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



Schematic recoil spectra for 1 MeV particles in Cu. Sizes represent recoil energies. After Averback, J Nucl Mater, 1994.



Olmstead, *et al*, Acta Mater, 2009.

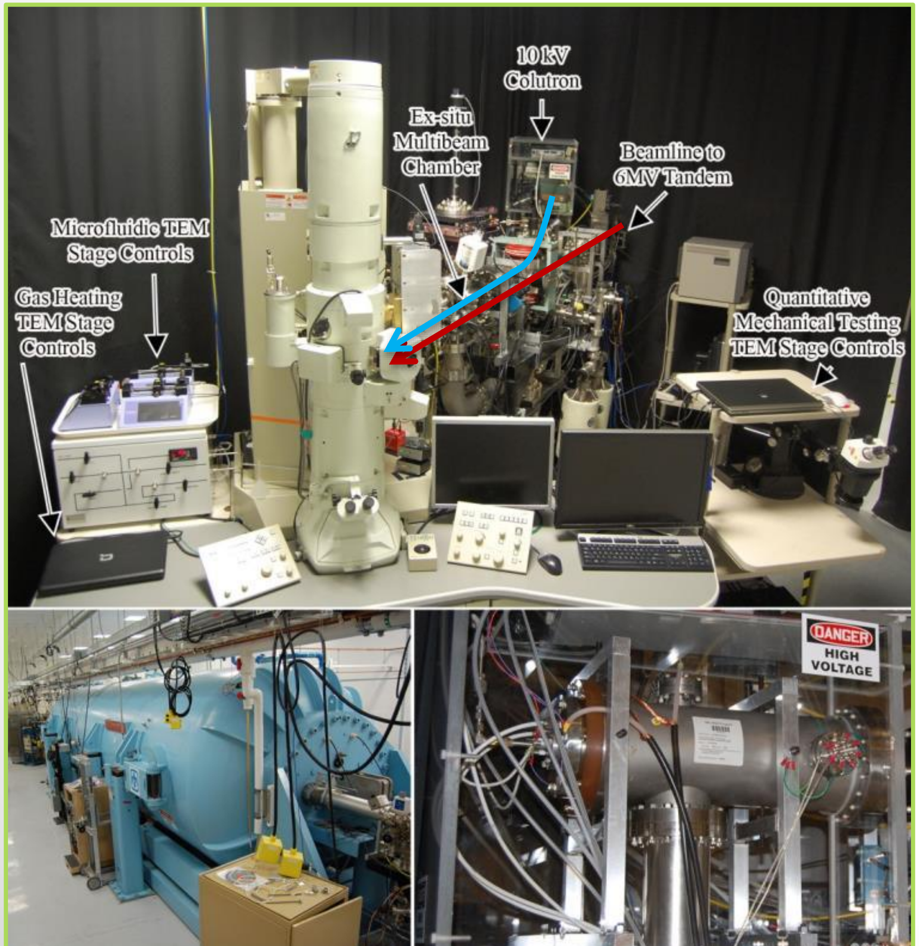
What are the relationships among ion damage, grain boundary character, and grain growth?

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

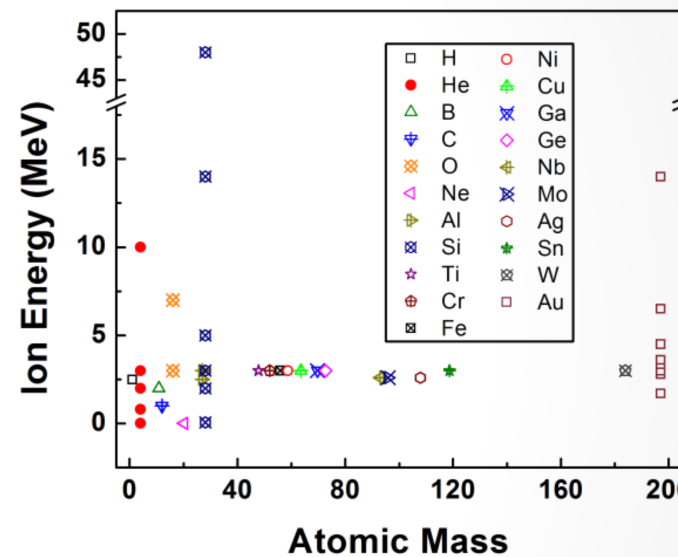
Sandia National Laboratories

Collaborators: D. Buller, K. Hattar, J. Scott

10 kV Colutron - 200 kV TEM - 6 MV Tandem



Ion species & energy introduced into the TEM



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

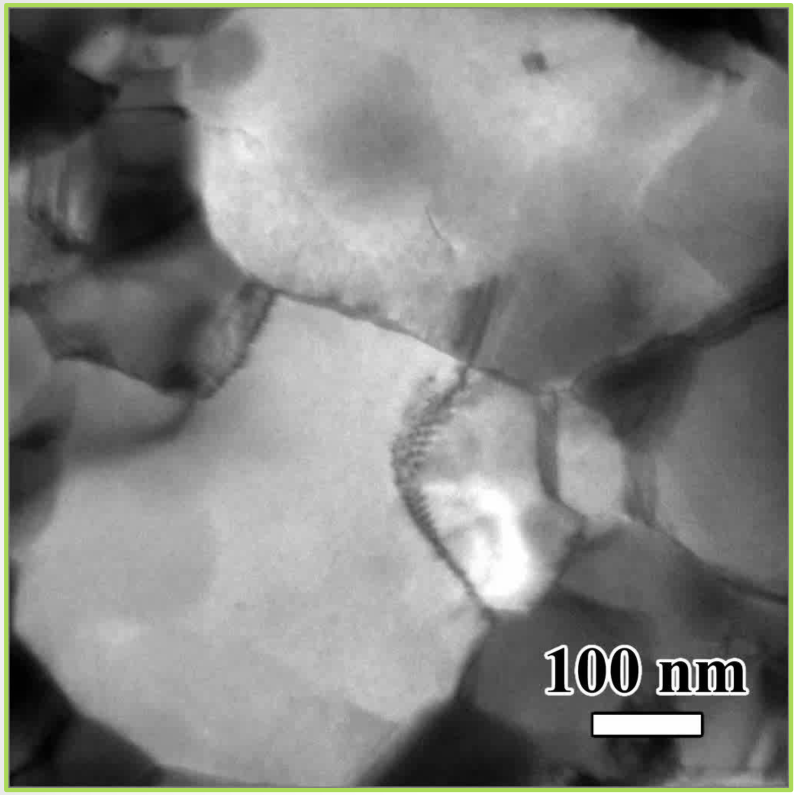
Similar beams can be directed to the TEM and end stations.

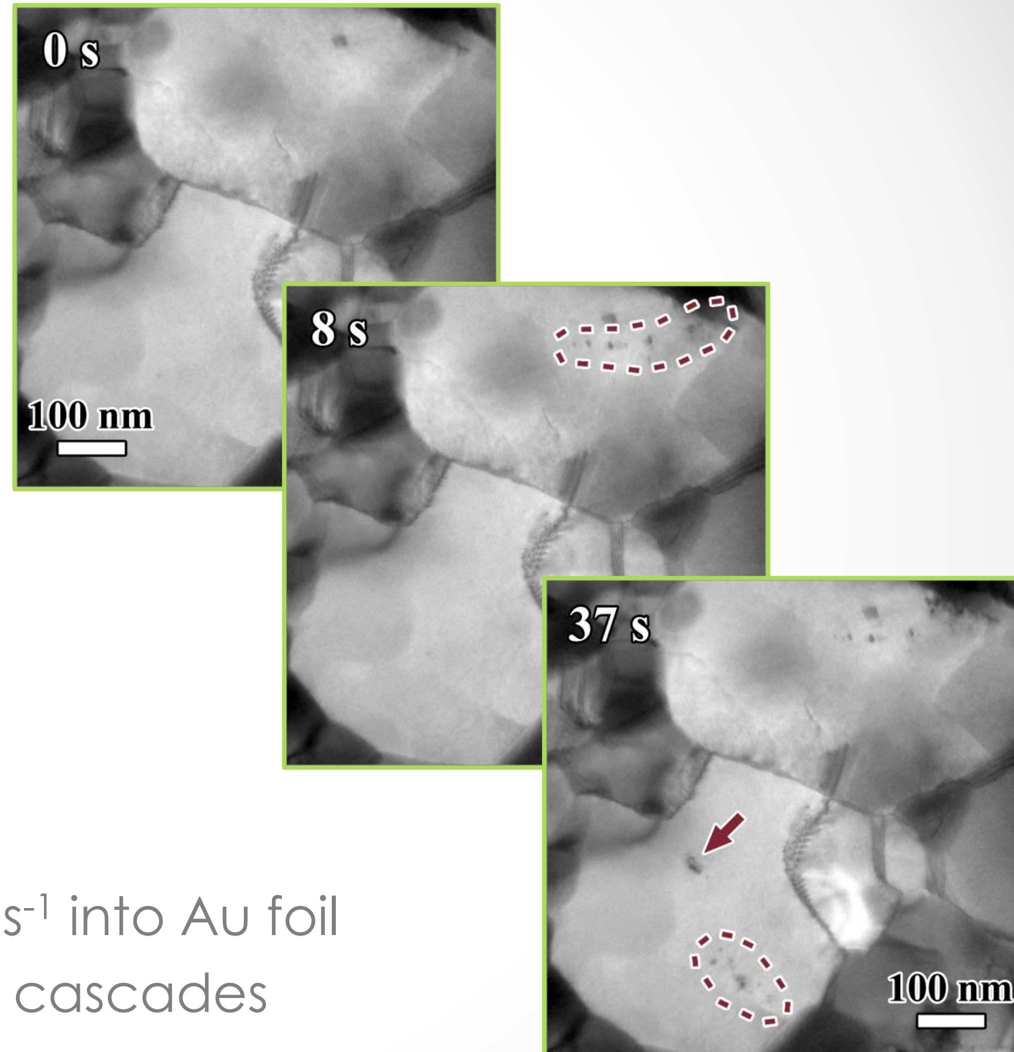
Hattar, *et al*, Nucl Instr Meth Phys Res B, 2014.

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

Sandia National Laboratories

Video speed ×5.





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

What happens near grain boundaries?

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

In Situ Irradiation

- Au foil during bombardment with 10 MeV Si^{3+}
- ~22 s of 4000s total experiment time

2× real time

Structural evolution on the timescale of seconds after ion strikes.

Quantification: Overall

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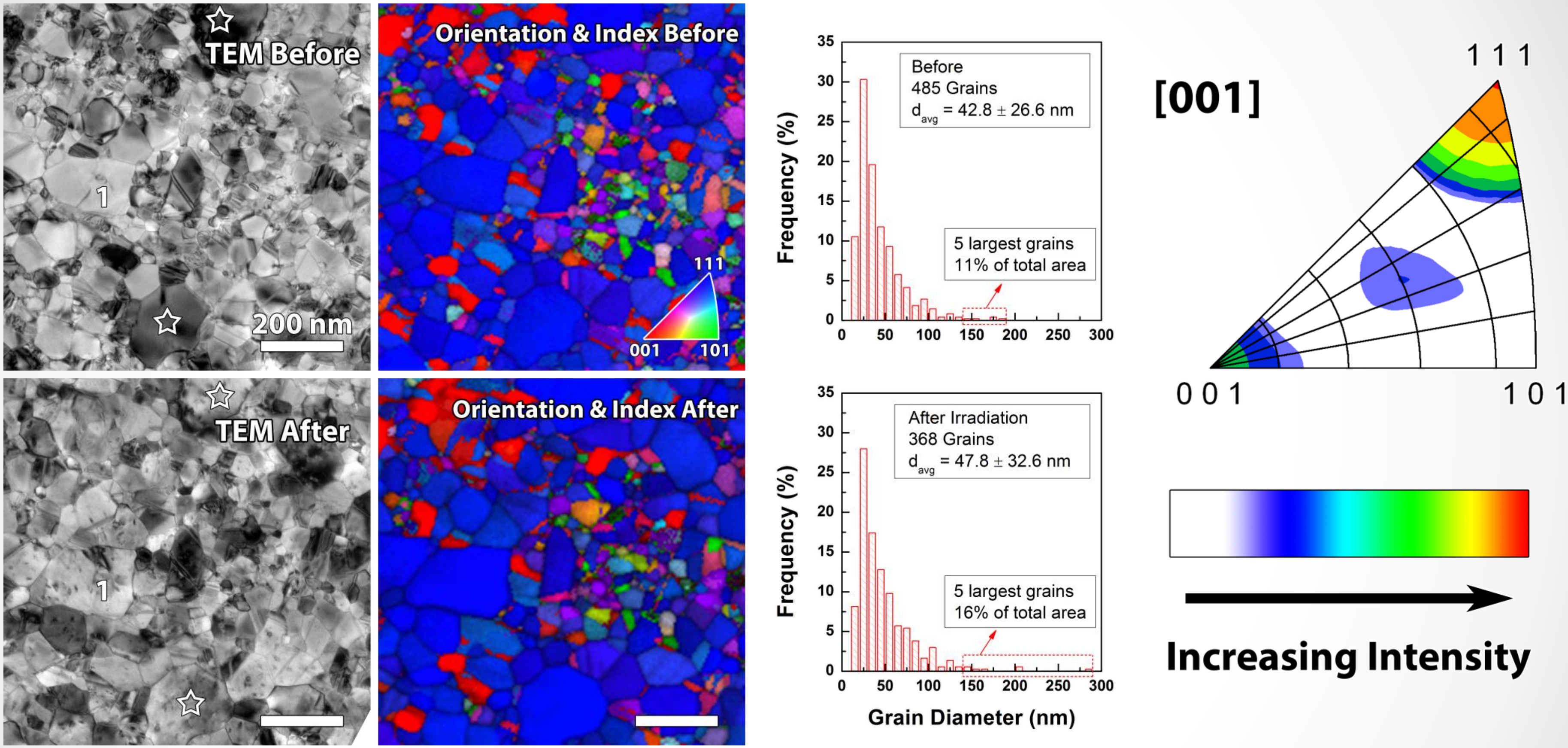
Bufford, *et al*, J Appl Phys, 2015.



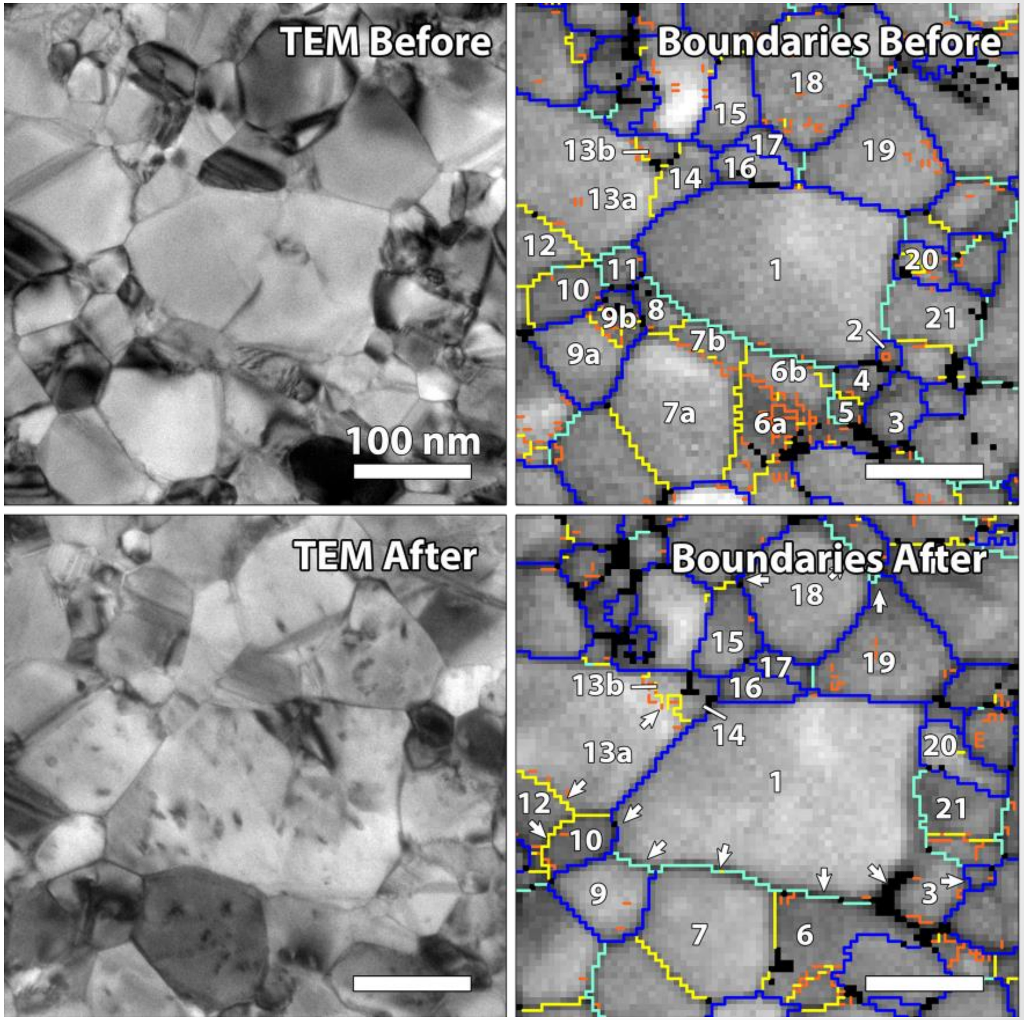
- Same area characterized before and after irradiation.
  - Hundreds of grains counted in minute
  - Grain size, orientation, boundary characters

Rapid quantification of statistically relevant numbers of grains and boundaries.

Individual Boundaries

Sandia National Laboratories

- The same grains identified before and after irradiation
- Individual grain boundary misorientation angles and axes quantified
- Correlation of GB properties and radiation-induced changes



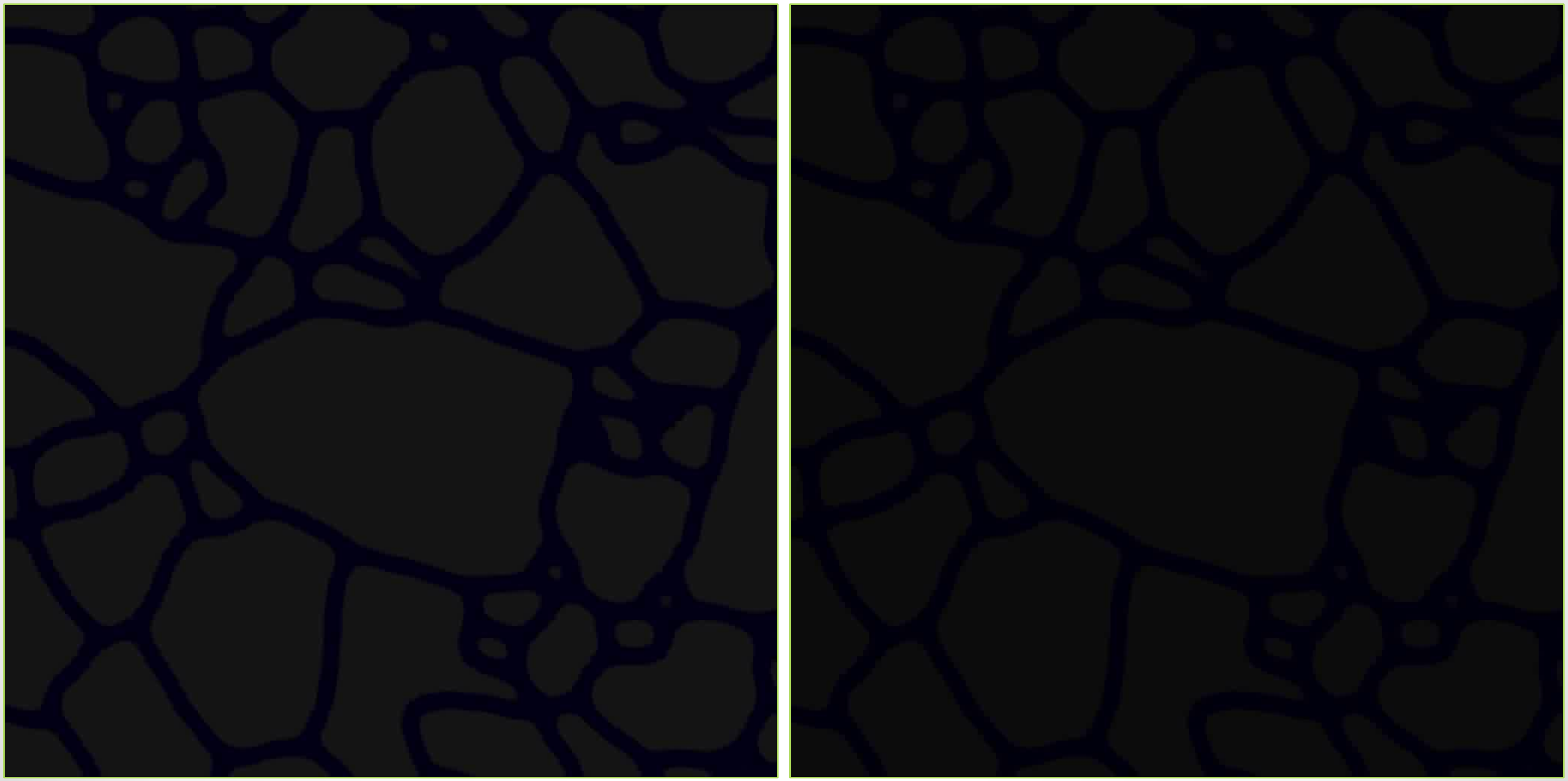
ϕ < 3°
3° ≤ ϕ < 15°
15° ≤ ϕ < 30°
30° ≤ ϕ

Bufford, *et al*, J Appl Phys, 2015.

Changes in individual boundaries quantified!

Simulated Irradiation and Annealing

Collaborators: F.F. Abdeljawad and S.M. Foiles



Exp. & Model Comparison

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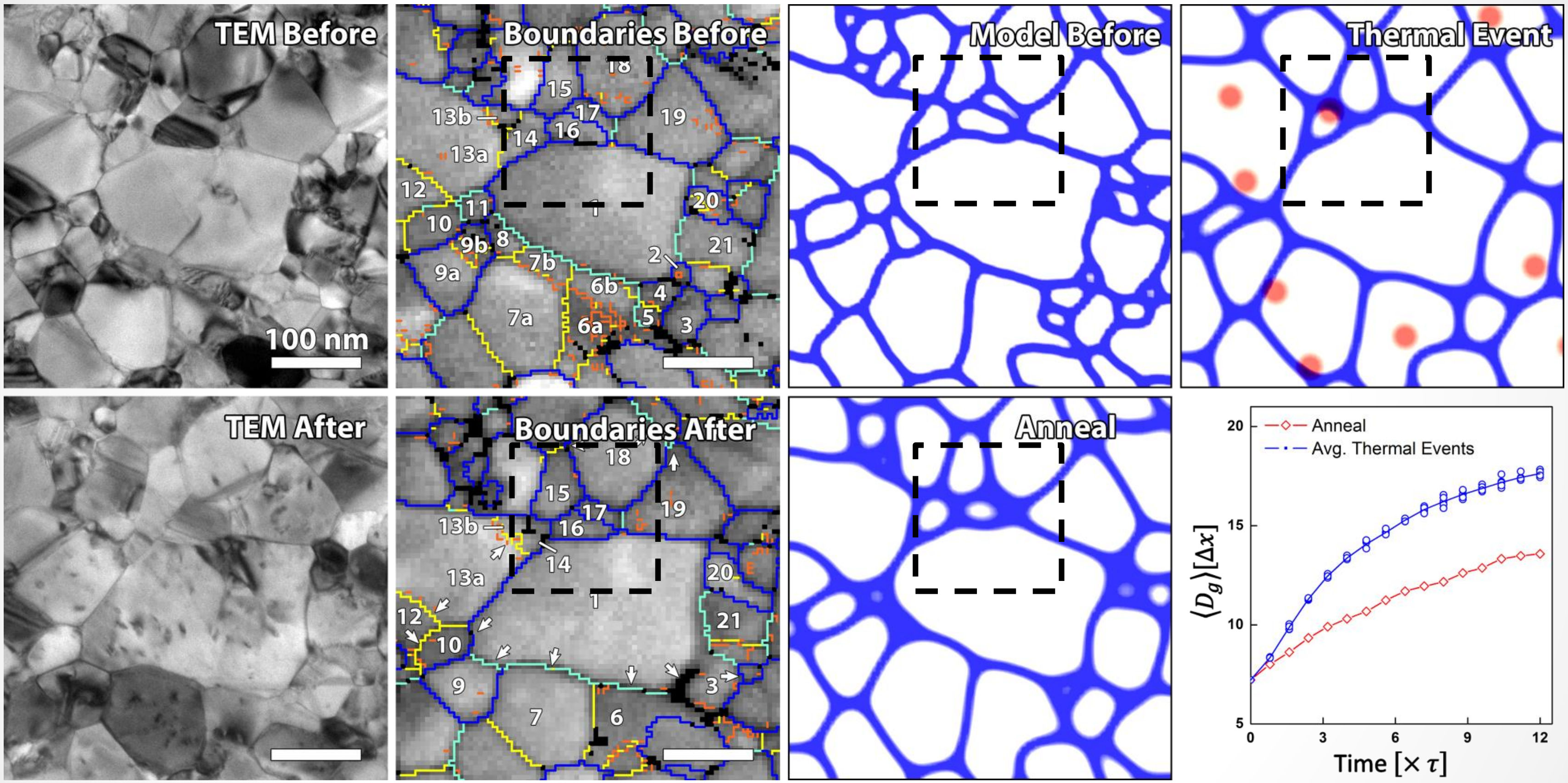


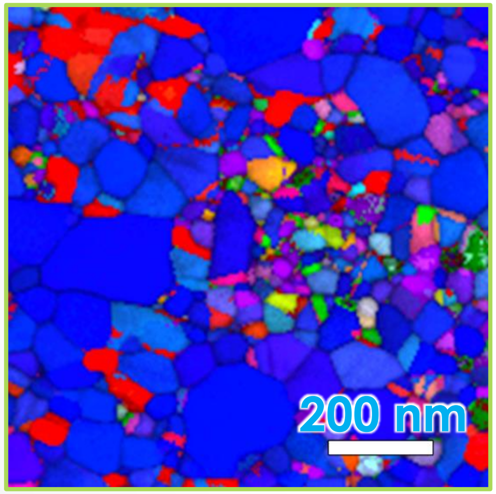
Bufford, *et al*, J Appl Phys, 2015.

- Overall scaling laws appear consistent
- Subtle deviations from homogenous grain growth

Stable boundaries suggest importance of non-thermally activated mobility.

Summary

- Response at the grain and grain boundary level to deformation, fatigue, and radiation in nanocrystalline materials
- Immediately relevant to small-scale devices.
- Fundamental knowledge of processes at the nanoscale informs models and improves understanding at longer length scales.



Microstructural-level underpinnings of macroscopic problems.

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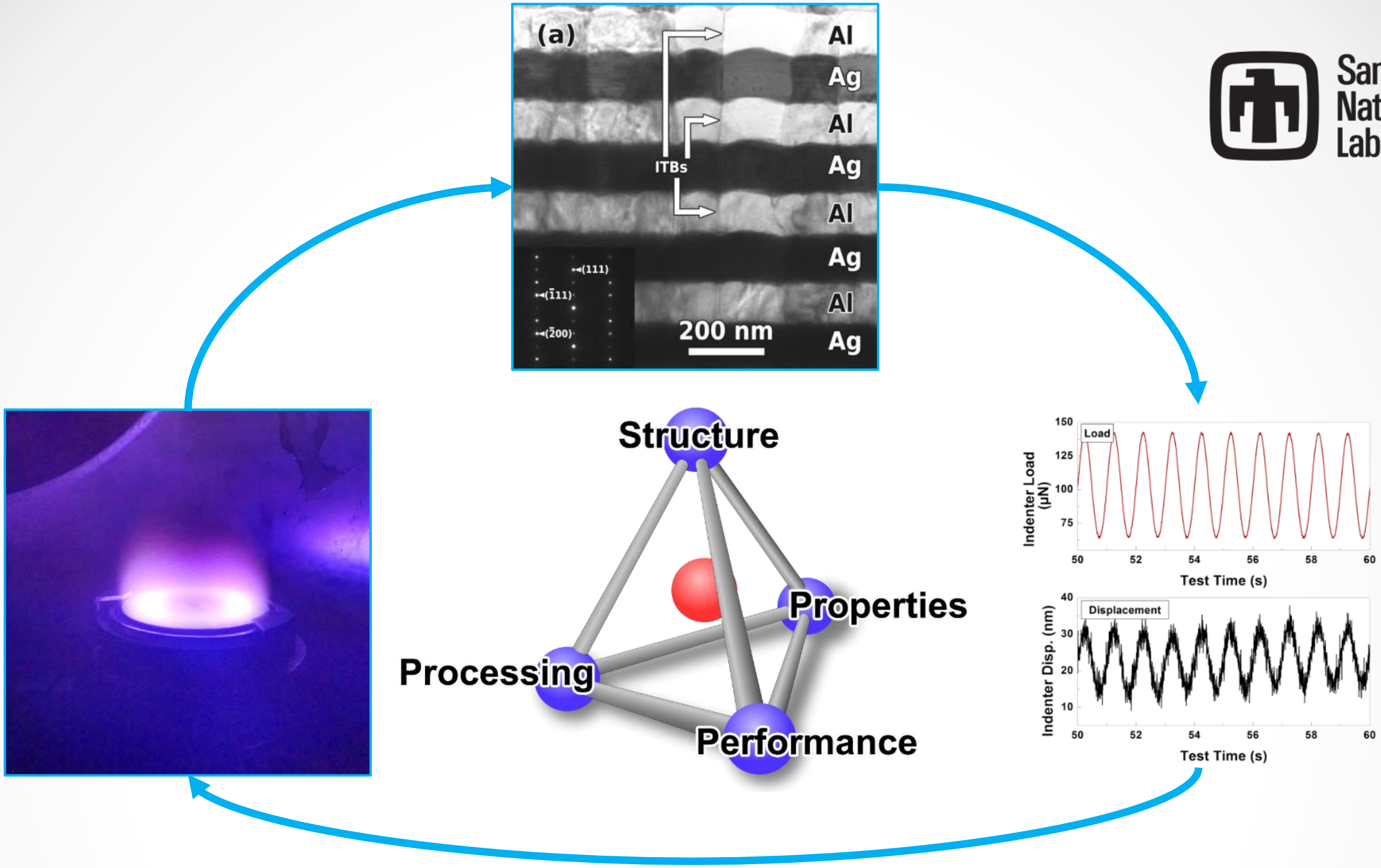


Coming full-circle to produce better materials.

Acknowledgements: **IBL**: D.L. Buller, B.L. Doyle, C. Gong, K. Hattar, M.T. Marshall, B.R. Muntifering, M. Steckbeck. **Sandia**: F.F. Abdeljawad, D.P. Adams, B. Boyce, T.J. Boyle, S.M. Foiles, H. Lim, W.M. Mook, J.A. Scott, J.A. Sharon, S.H. Pratt, M. Rye, C. Sobczak. **External**: S. Bhowmick, L. Kuhn (Hysitron), A. Minor, C. Chisholm, P. Hosemann (UC Berkley), Z. Bi, Q.X. Jia, Y. Liu, (Los Alamos National Lab), A. Darbal (AppFive), D. Kaoumi (University of South Carolina), A. Leff (Drexel University), H. Wang, X. Zhang (Purdue University). Work performed by DCB at Sandia was fully supported by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science under proposal #U2014A0026.

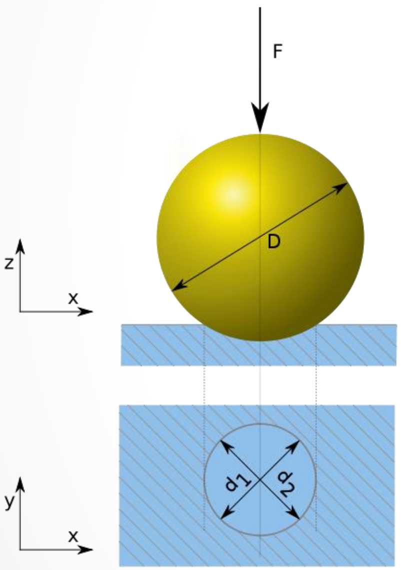
U.S. DEPARTMENT OF ENERGY | Office of Science

NSF

Macro/Microindentation

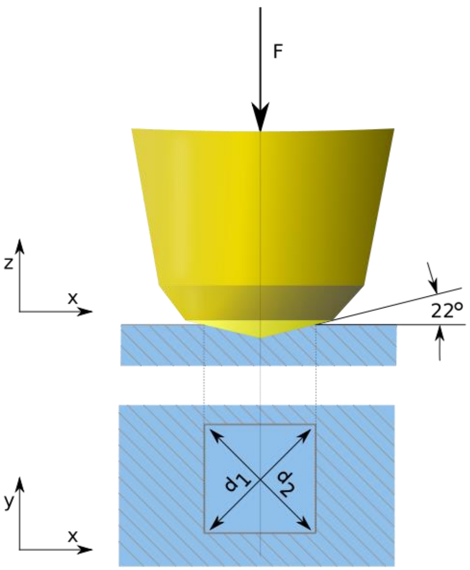
- Apply a static load
- Measure residual indentation area
- Depths from tens of µm to mm

Lokilech, *via* Wikimedia Commons.



Brinell

A1, *via* Wikimedia Commons.



Vickers

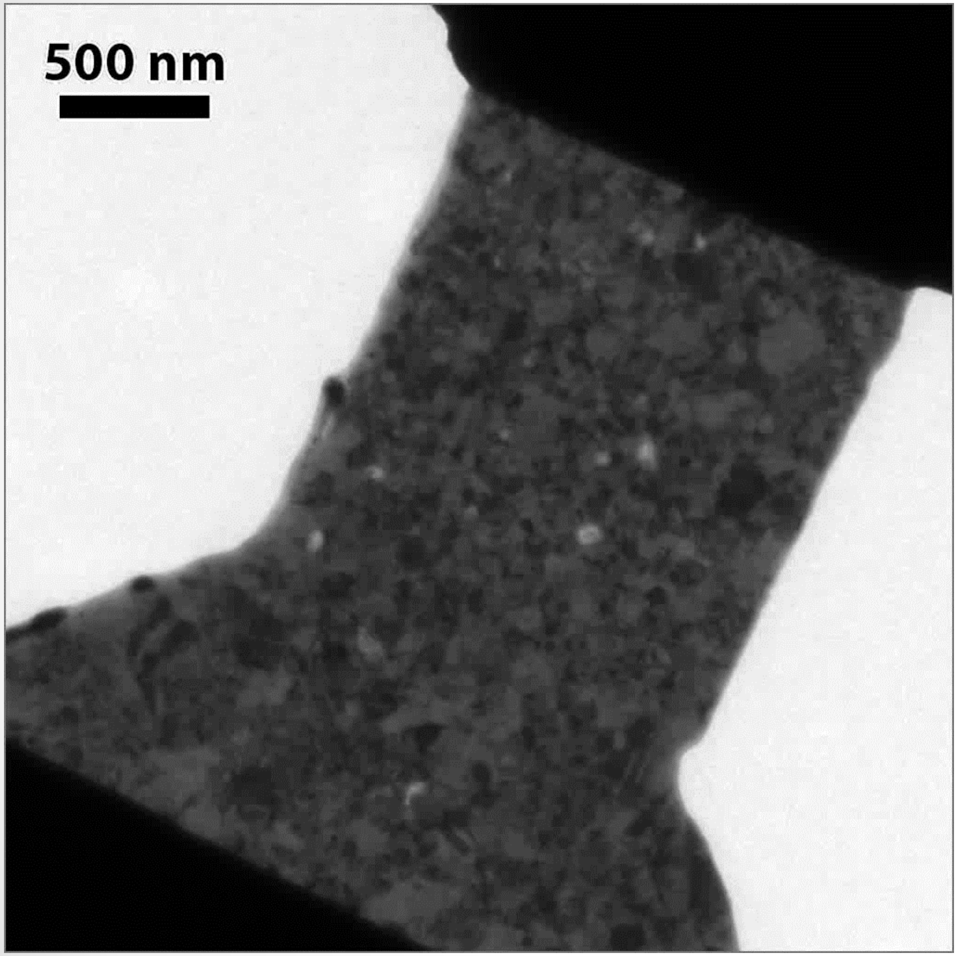
Diamond Vickers Tip

R. Tanaka, *via* Wikimedia Commons.

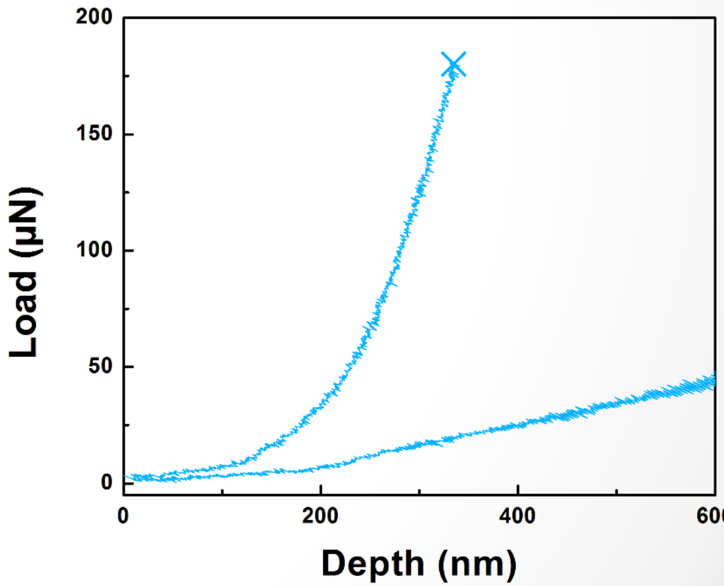
- Hardness, $H = \frac{P_{max}}{A_r}$

Infer bulk properties from local resistance to plastic deformation in "small" volumes.

Monotonic



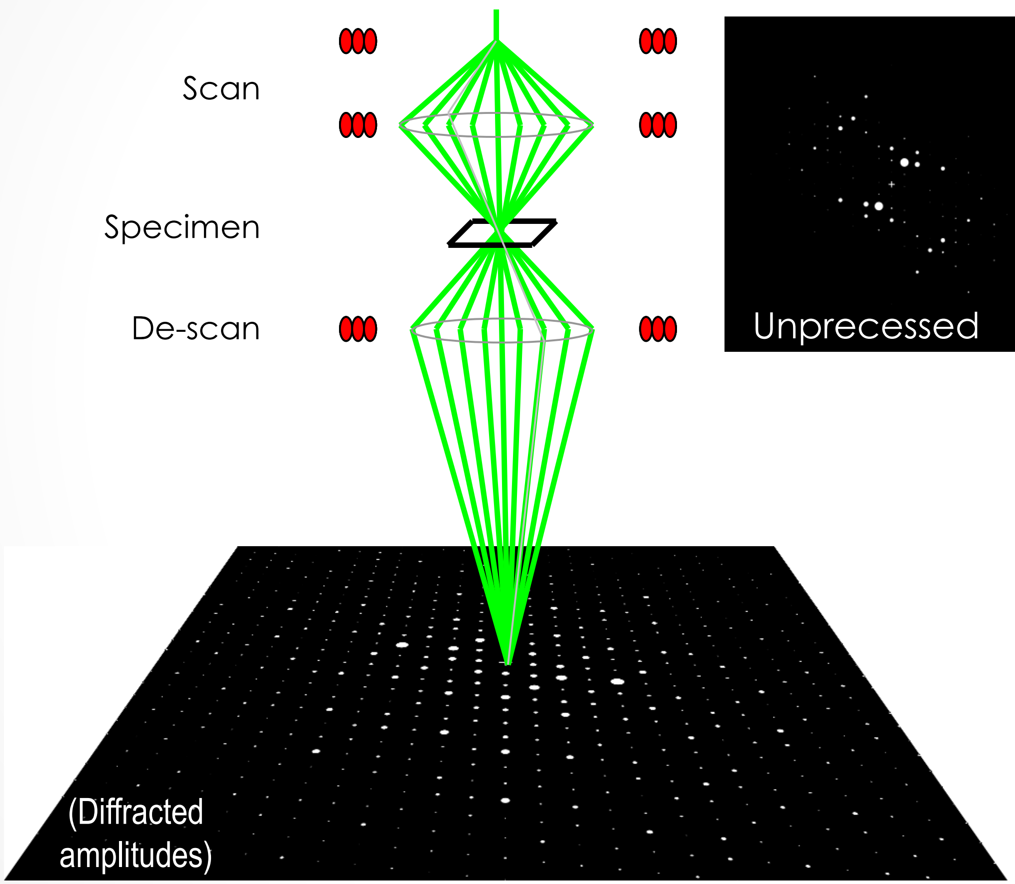
- Monotonic loading
  - Negligible plasticity before failure
  - Rapid crack propagation

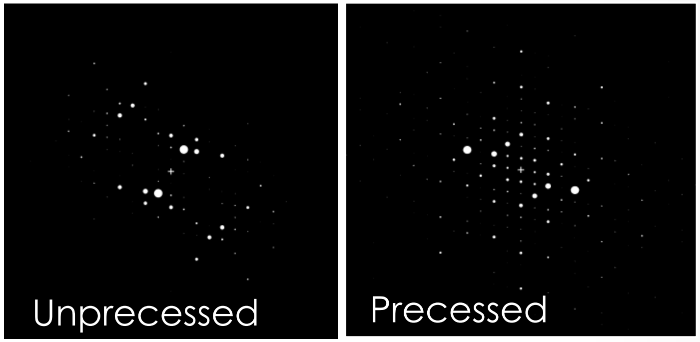


Successful quantitative tensile testing of a micrometer-scale sample!

Precession Electron Diffraction Microscopy

Collaborators: K.J. Ganesh, S. Rajasekhara, P.J. Ferreira



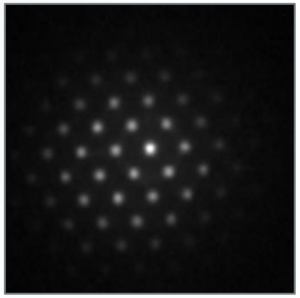


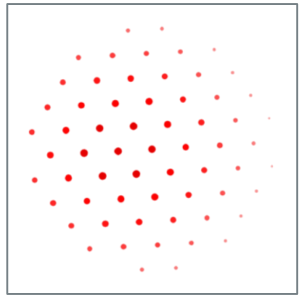
Advantages:

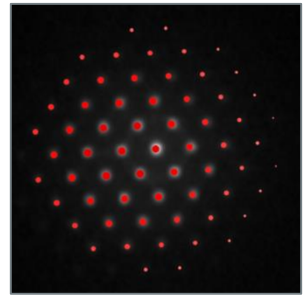
- < 10 nm spatial resolution
- Near kinematical electron diffraction
- Symmetry ambiguities are resolved
- Fast and automated acquisition
  - ~200 grains in 15 min.

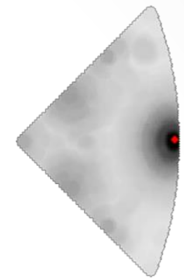
Slide courtesy of K.J. Ganesh

Approach: Experimental

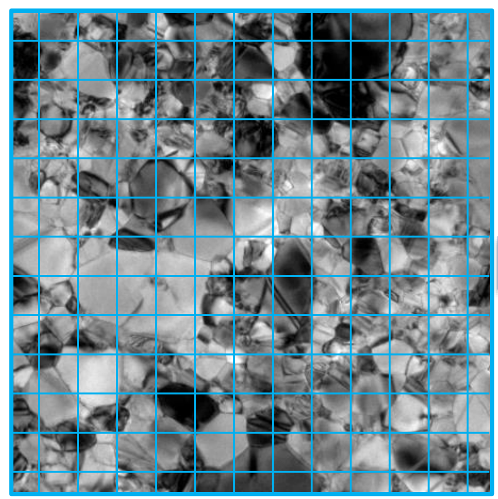

Experimental Pattern

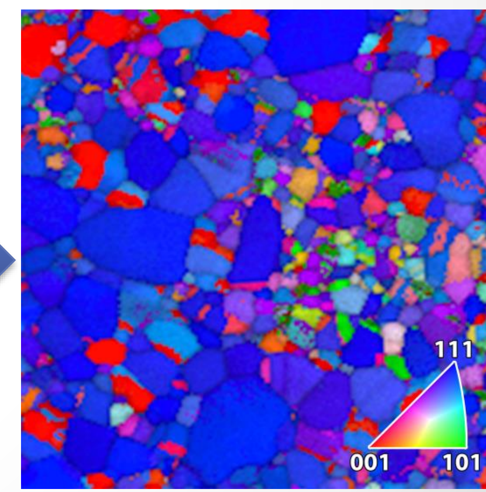

Theoretical Template


Template Matched


Point Mapped To IPF

- Automated diffraction orientation mapping
  - Point by point grid of orientations mapped
  - 5 nm resolution
- Analogous to EBSD

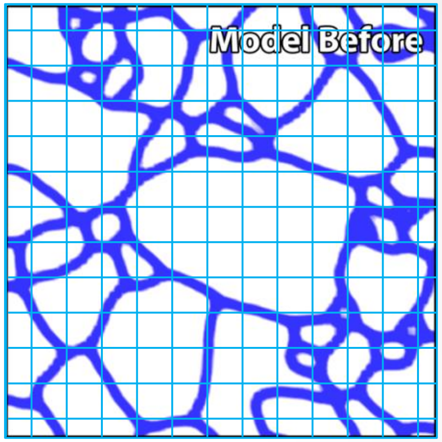


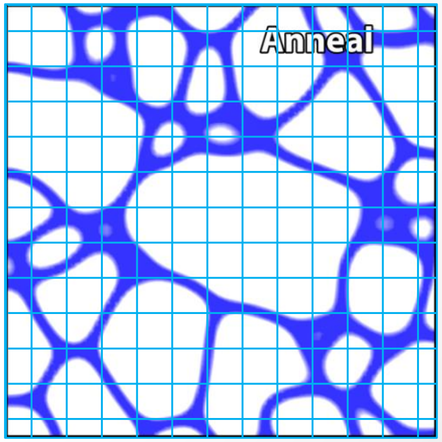


Point diffraction data

Approach: Modeling

- What is phase field modeling?
  - Mathematical model for solving interfacial problems, like solidification, growth, etc.
- Example grain growth model
  - Thermodynamic free energy function
    - $dF = d(\gamma A) = \gamma dA$ (γ: GB energy, A: GB area)
  - Model for kinetics
    - $V = M\gamma h$ (M: GB mobility, h: GB curvature)
  - Solve at each pixel for a predetermined timestep
- See Abdeljawad and Foiles, Acta Mater, 2015 for more information

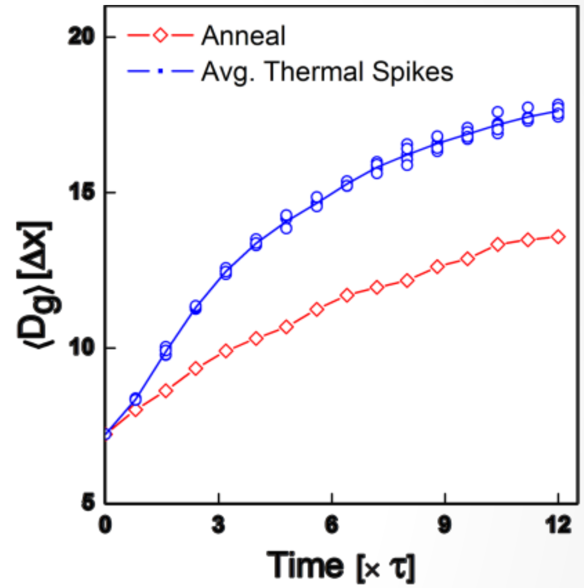


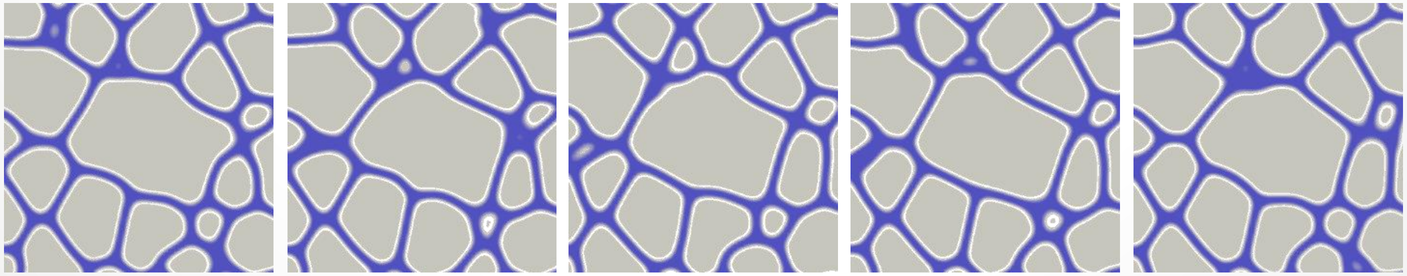


Can directly use experimental maps as input structures, and then compare evolutions!

Model Data Analysis

- During simulated annealing grain growth scales approximately with $T^{1/2}$
  - Expected for homogenous grain growth
- During simulated irradiation, grain growth scales with $T^{1/n}$, where $n \approx 3$
  - Initially faster, but stagnates sooner





Growth time scaling

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Fraction of ion strikes that intersect grain boundaries (thus contributing to grain growth):

$$f_{GB} = \frac{\pi D^2 \frac{d_{\text{spike}}}{2}}{\frac{\pi D^3}{6}} = \frac{3d_{\text{spike}}}{D},$$

- Incorporation of this *D* term leads to scaling proportional to t$^{(1/3)}$.
- Consistent with experimental observations.

$$\frac{dD}{dt} = \frac{\Phi\chi\delta}{N_{\text{at}}}\frac{3d_{\text{cas}}}{D}\left[\frac{4\gamma V_{\text{at}}N_{\text{at}}\nu}{Dk_B}\frac{\sqrt{\frac{3}{5}}\Gamma\left(\frac{8}{3}\right)k_B^{8/3}}{10\pi C_0^{2/3}\kappa_0}\frac{Q^{5/3}}{E_a^{8/3}}\right].$$

$$D^2 dD = \left[12\gamma d_{\text{spike}}\Phi\chi\delta\frac{V_{\text{at}}\nu\sqrt{\frac{3}{5}}\Gamma\left(\frac{8}{3}\right)k_B^{5/3}}{10\pi C_0^{2/3}\kappa_0}\frac{Q^{5/3}}{E_a^{8/3}}\right]dt.$$

$$D^3 - D_0^3 = \left[36\gamma d_{\text{spikes}}\chi\delta\frac{V_{\text{at}}\nu\sqrt{\frac{3}{5}}\Gamma\left(\frac{8}{3}\right)k_B^{5/3}}{10\pi C_0^{2/3}\kappa_0}\frac{Q^{5/3}}{E_a^{8/3}}\right]\Phi t$$

$$= K\Phi t.$$

Kaoumi, *et al*, J Appl Phys, 2008.