SAND2016-7313PE

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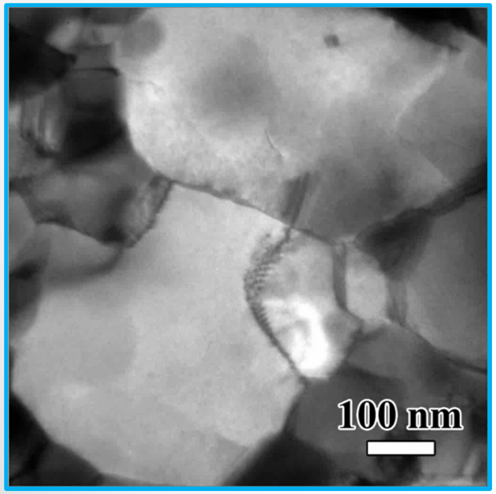
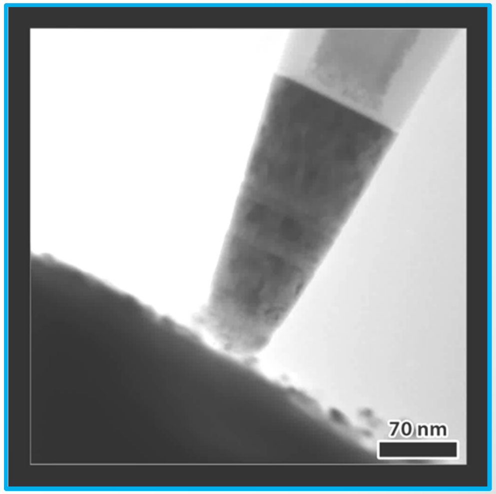
Radiation and Mechanical Responses of Materials Revealed by *In Situ* Transmission Electron Microscopy (TEM)



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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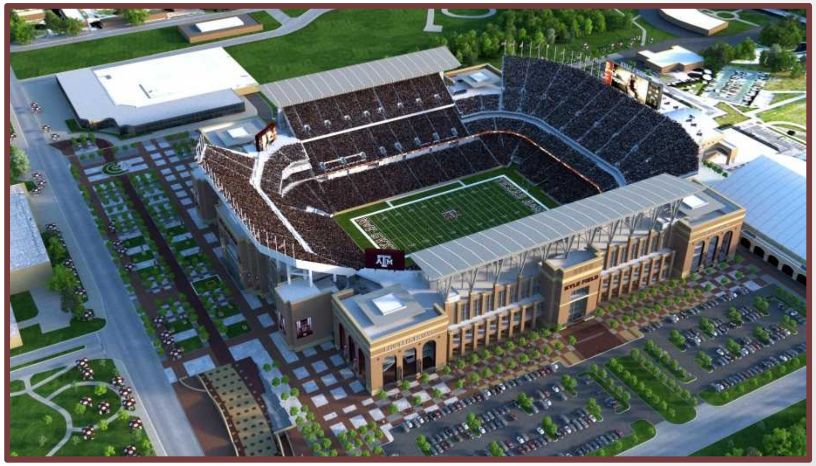
3 August, 2016

My Background

Sandia National Laboratories

Olin College of Engineering



TEXAS A&M UNIVERSITY

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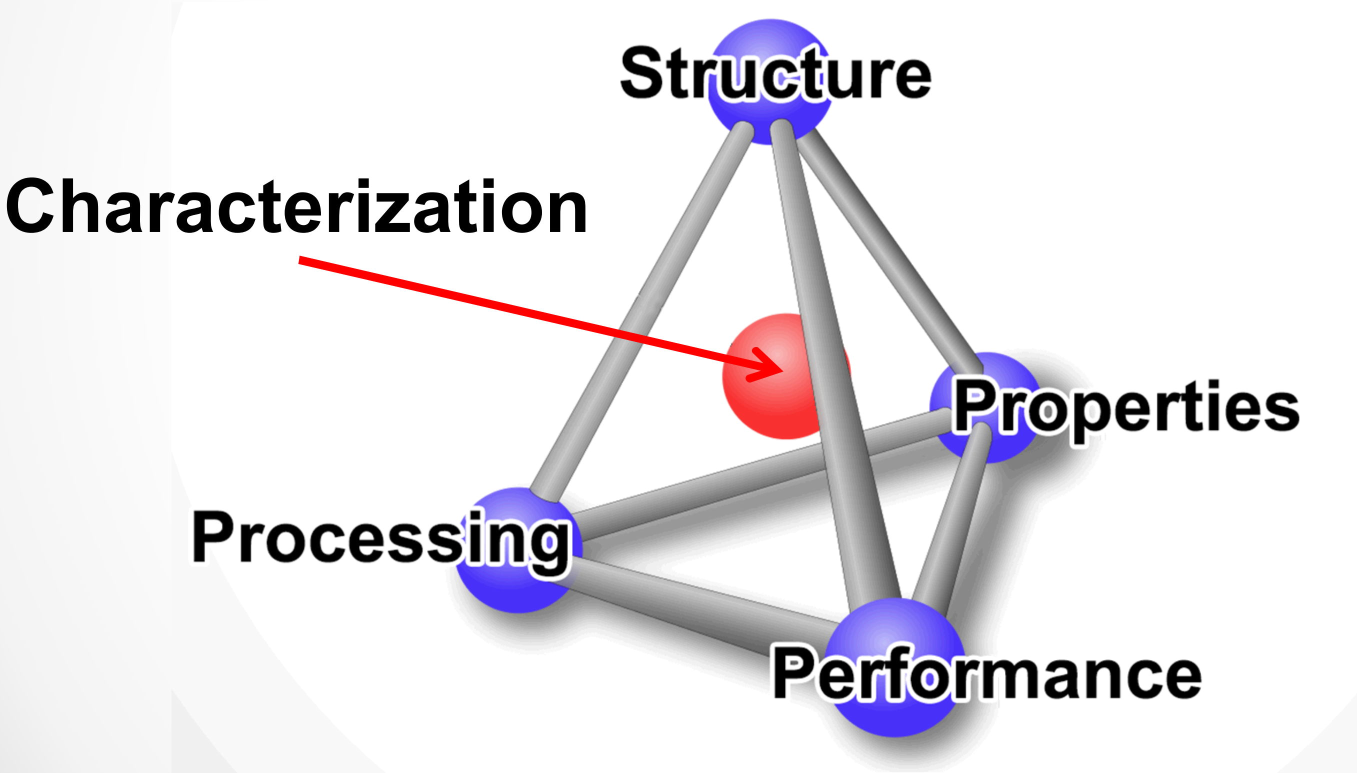
The Core of Materials Science



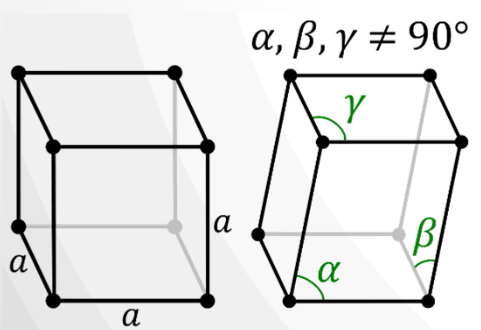
Crystalline Materials

nm

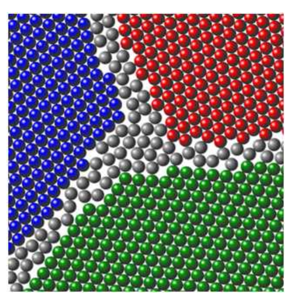
Tens of nm

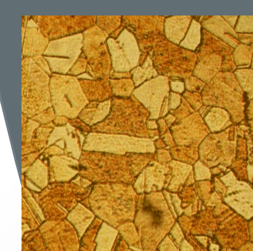
Hundreds of nm

Thousands of nm



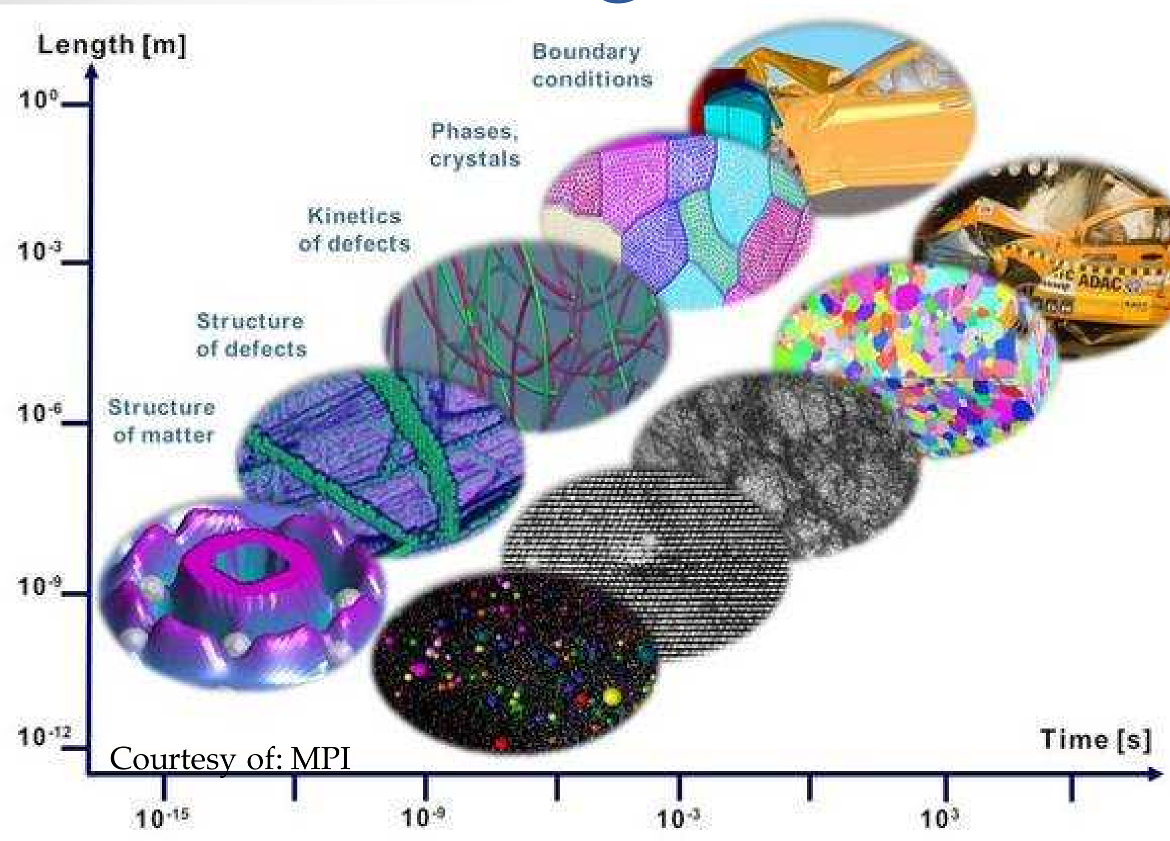
Stannered, 2007, *via* Wikimedia Commons.





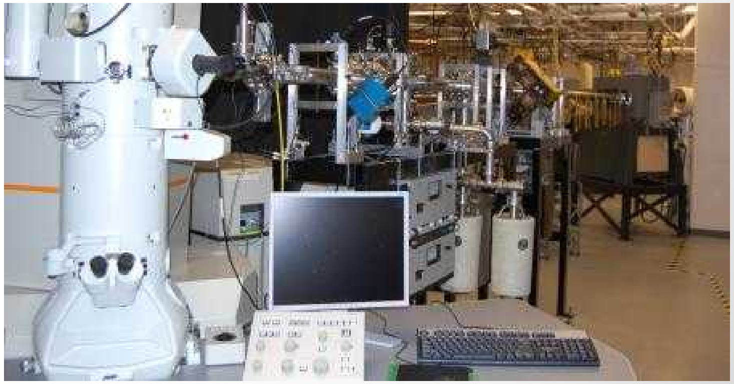
Strangerhahaha, 2008, *via* Wikimedia Commons.

Material Behavior Across Time & Length Scales



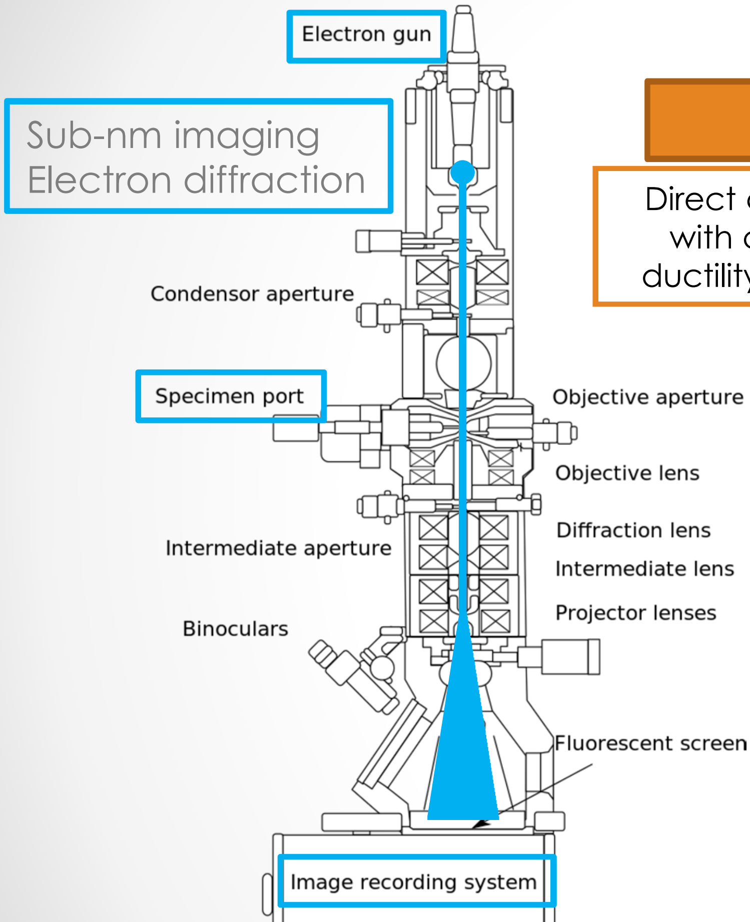
Ion Beam Lab (IBL)

In situ Ion Irradiation TEM (I³TEM)



To develop predictive physics-based models, a fundamental understanding of the structure of matter, defects, and the kinetics of structural evolution in the environments of interest are needed.

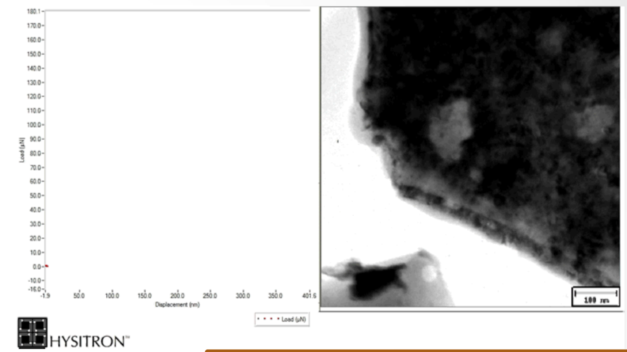
TEM and *In Situ* Experiments



Gringer, 2009, *via* Wikimedia Commons.

Mechanical

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

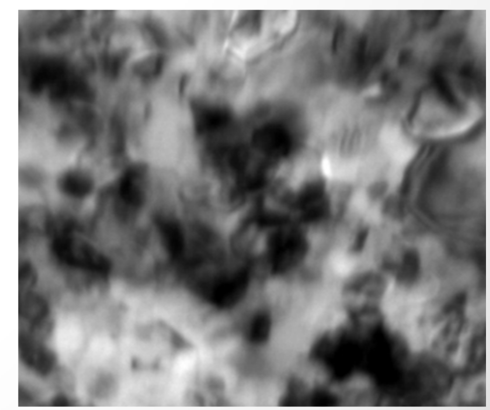


Environmental

Effects of corrosion and gas loading at the grain level

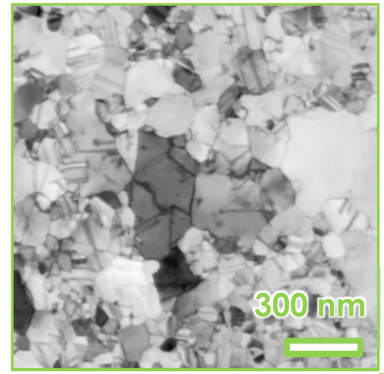
Thermal

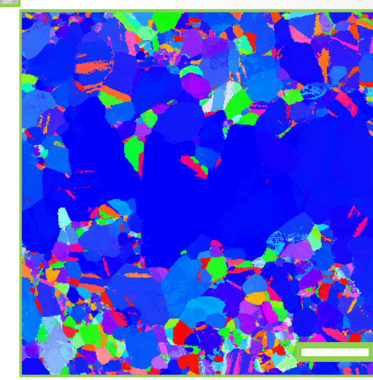
Effects of temperature on microstructural evolution up to 800 °C

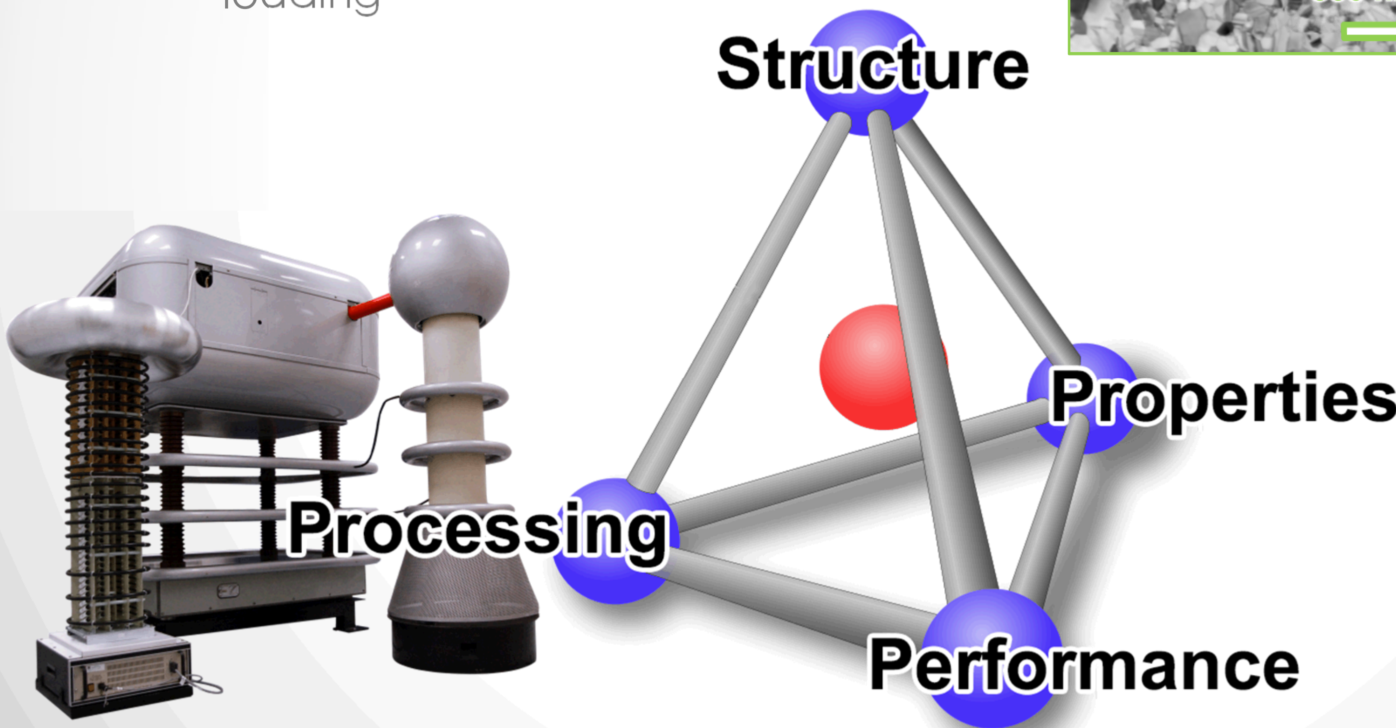


- Enables real-time studies of samples under various stimuli
- Limited to electron-transparent, vacuum & electron beam-compatible samples

- Ion irradiation
  - Ion beam modification
- Mechanical behavior
  - Monotonic and cyclic loading

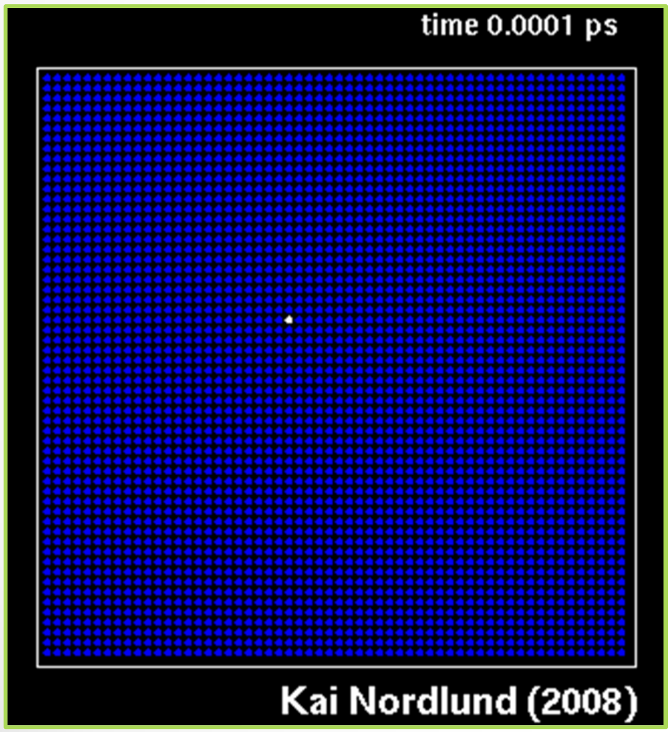




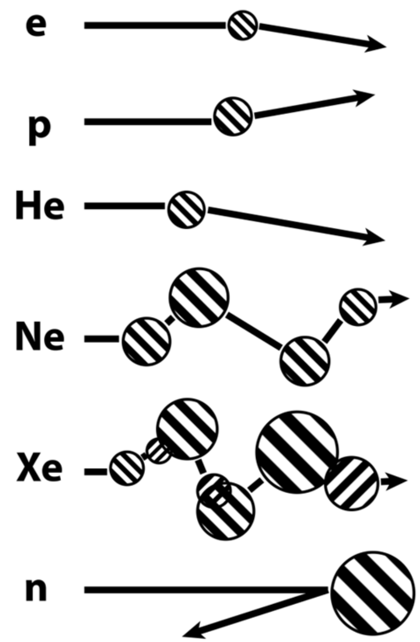


Radiation-Solid Interactions

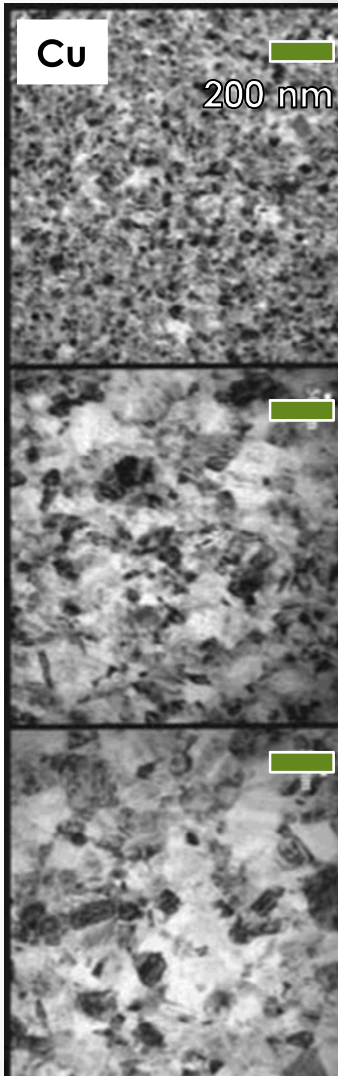
- Energetic ion displaces one or more target atoms
  - Frenkel (vacancy-interstitial) pair
  - Collision cascade
  - Nuclear and electronic interactions



10 keV Au in Au, *via* Wikimedia Commons.



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



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

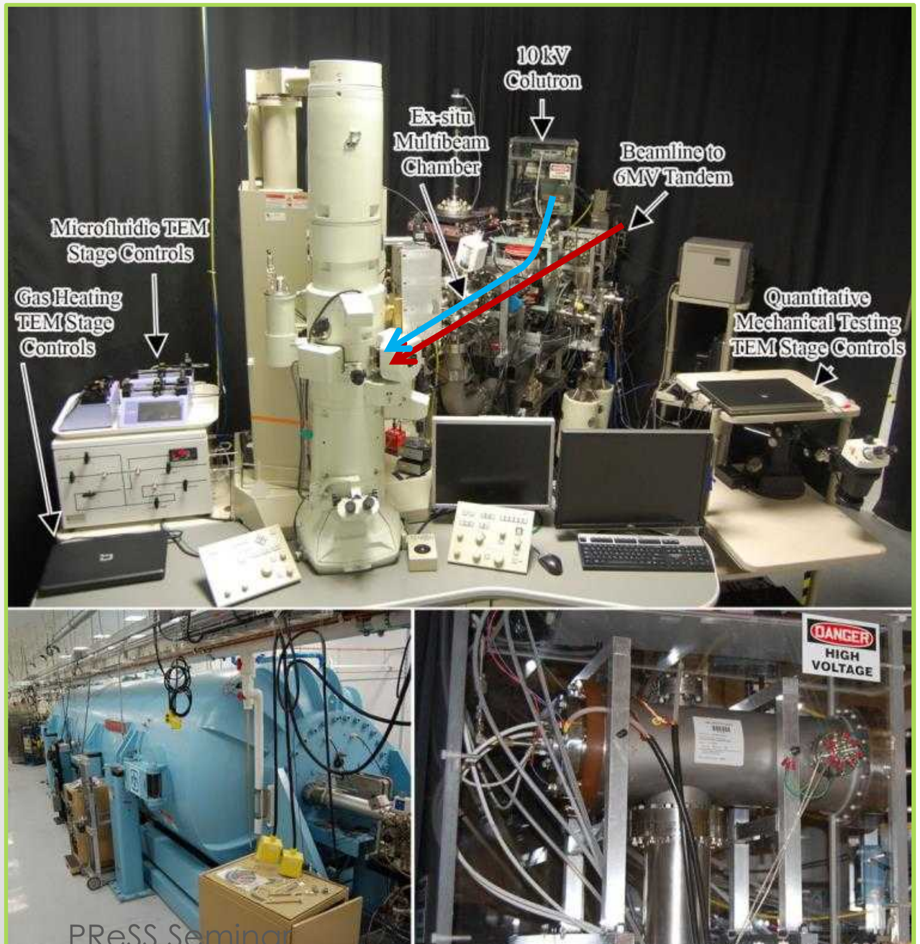
Highly temporally and spatially localized energy transfer drives microstructural change.

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

Sandia National Laboratories

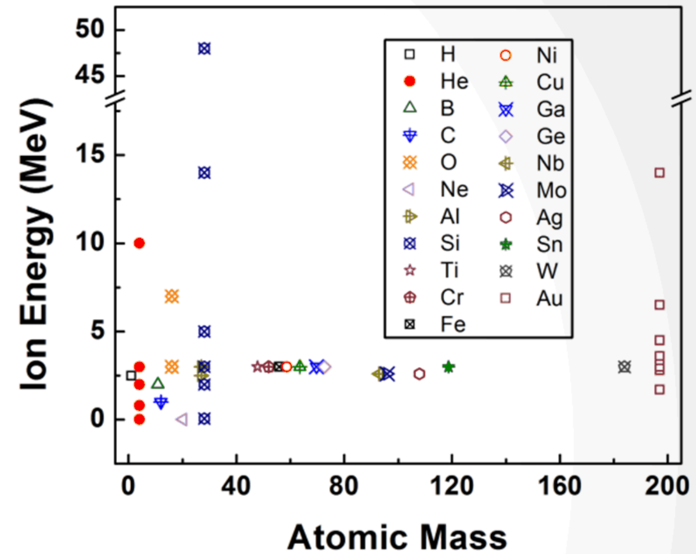
Collaborators: D. Buller, K. Hattar, J. Scott

10 kV Colutron - 200 kV TEM - 6 MV Tandem

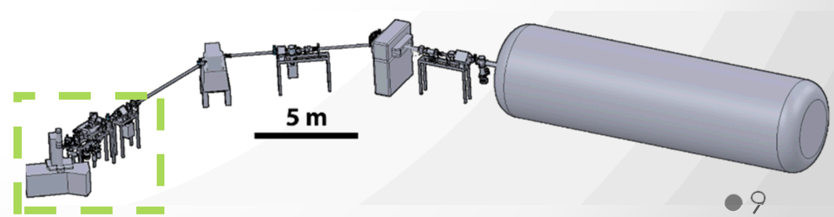


Hattar, *et al*, Nucl Instr Meth Phys Res B, 2014.

Ion species & energy introduced into the TEM



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

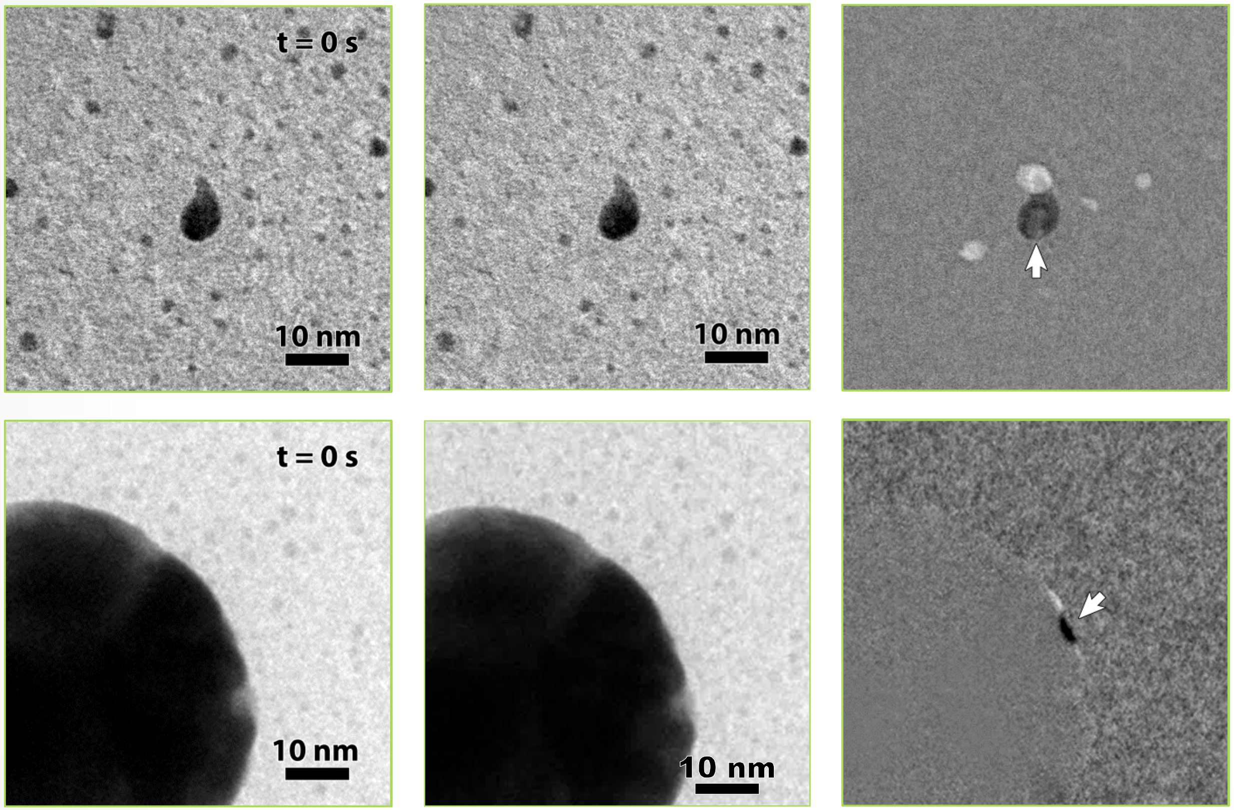


Single Ions in Nanoparticles

Collaborators: T.J. Boyle, K. Hattar, S. Pratt

5 nm

60 nm



Difference Images

- **Dark**: Only present before
- Light: Only present after
- Gray: unchanged

- Single 46 keV Au^- ions into Au nanoparticles

Effects of similarly sized cascades vary dramatically with particle size.

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

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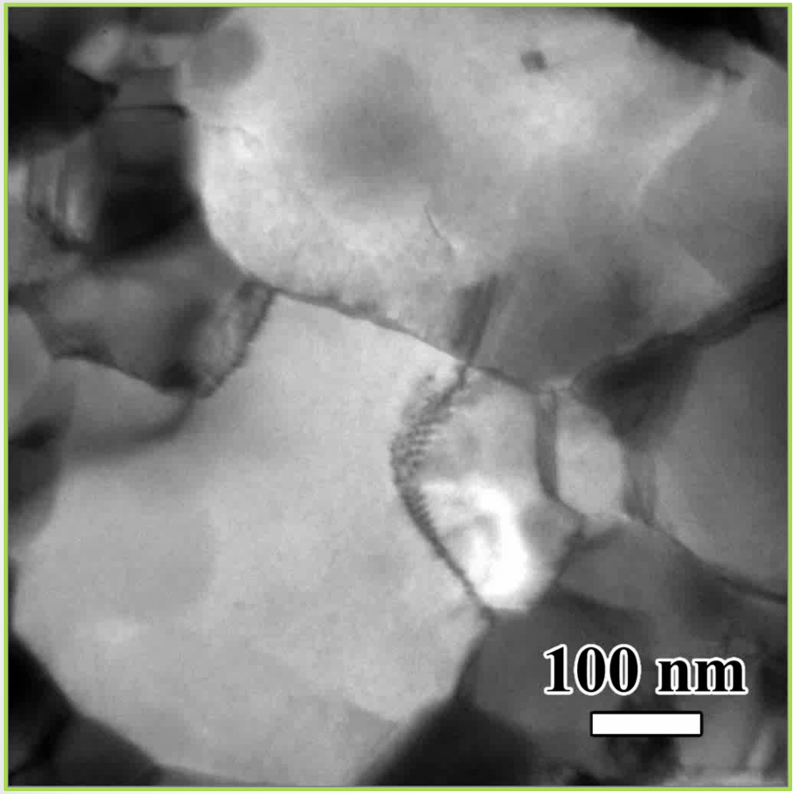
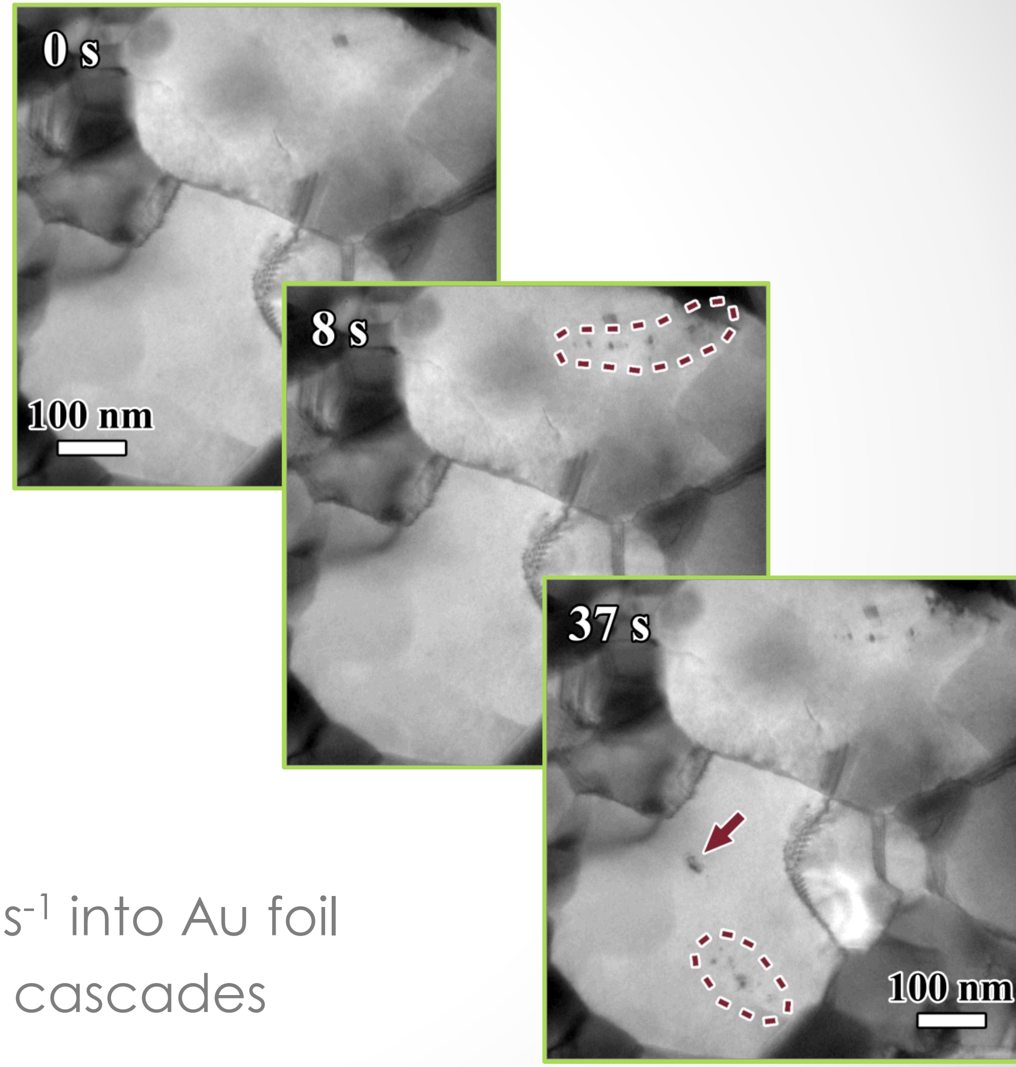
Video speed ×5.





- 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

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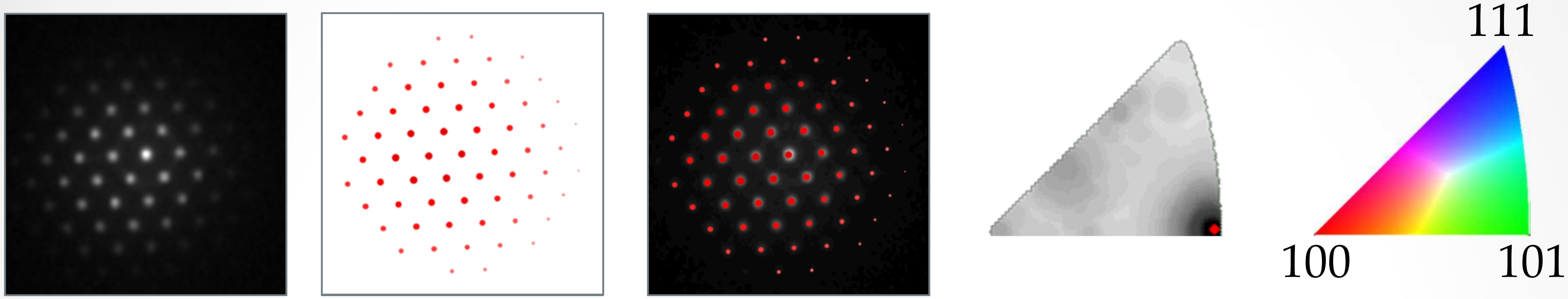
- Au foil during bombardment with 10 MeV Si^{3+}
- ~10 s of 4000s total experiment time
- Can reproduce previous results, but with greater energy range and expanded capabilities for analysis.

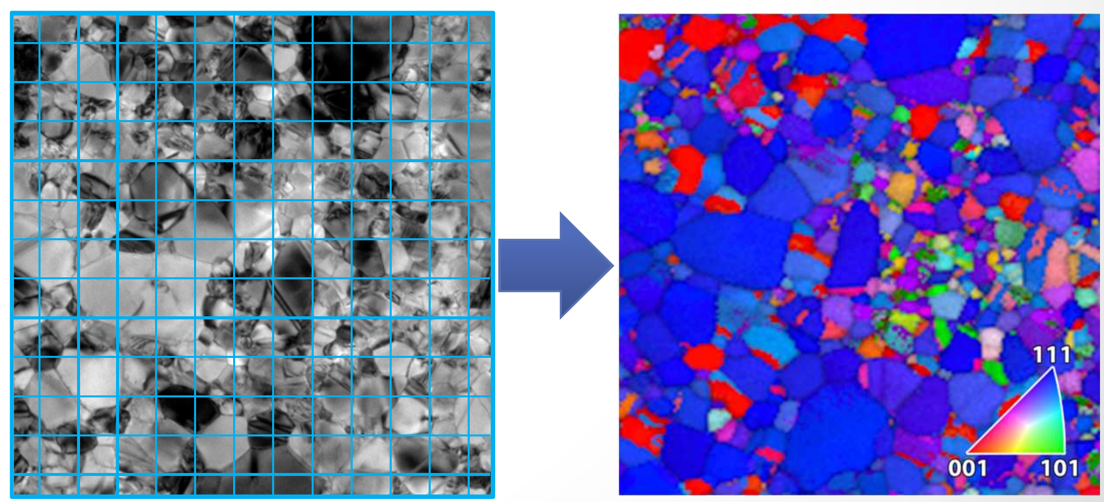
Locations of single ion strikes and resulting microstructural change captured.

Microstructure Digitization



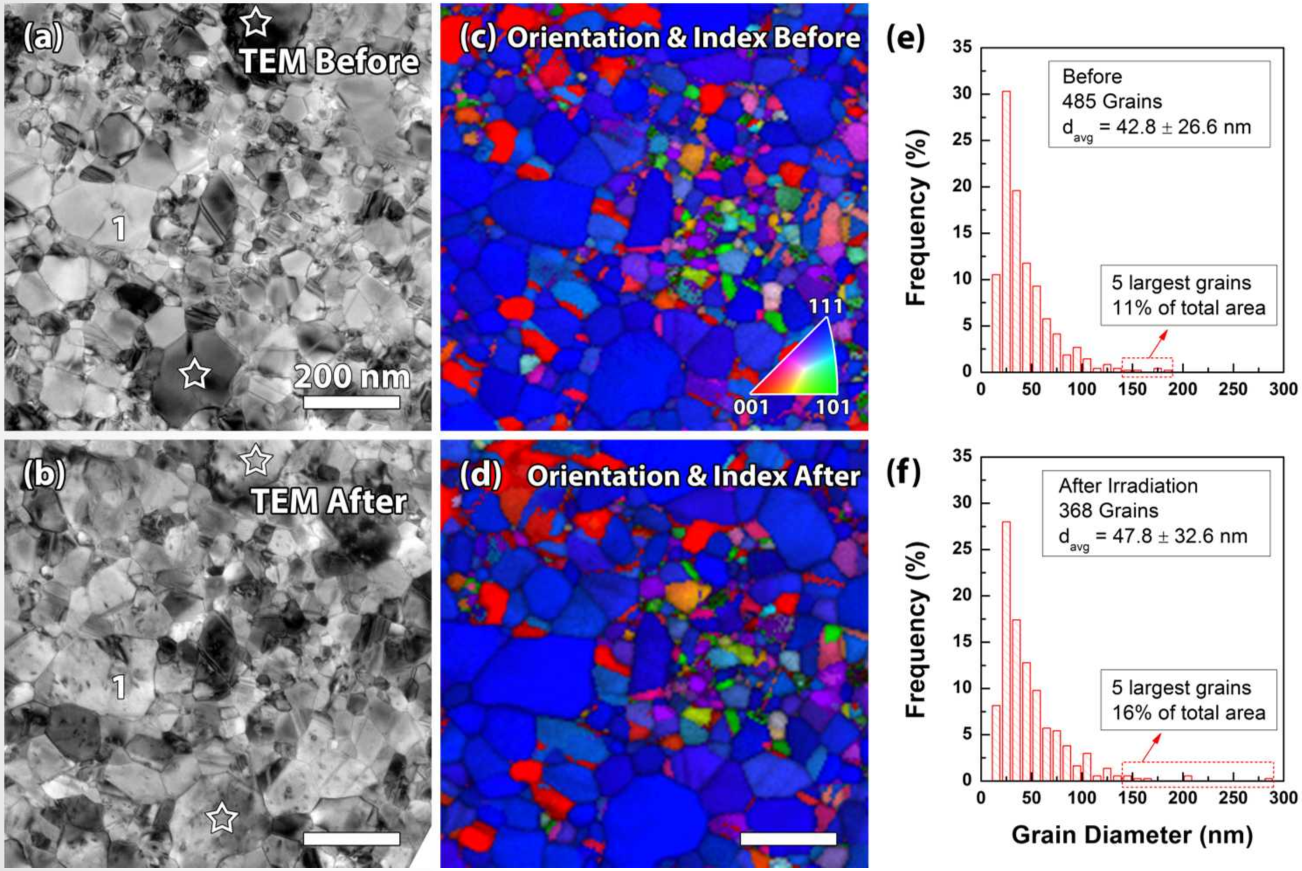
Experimental Pattern — Theoretical Template — Template Matched — Point Mapped To IPF

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



Point diffraction data

GB Movement: Statistical



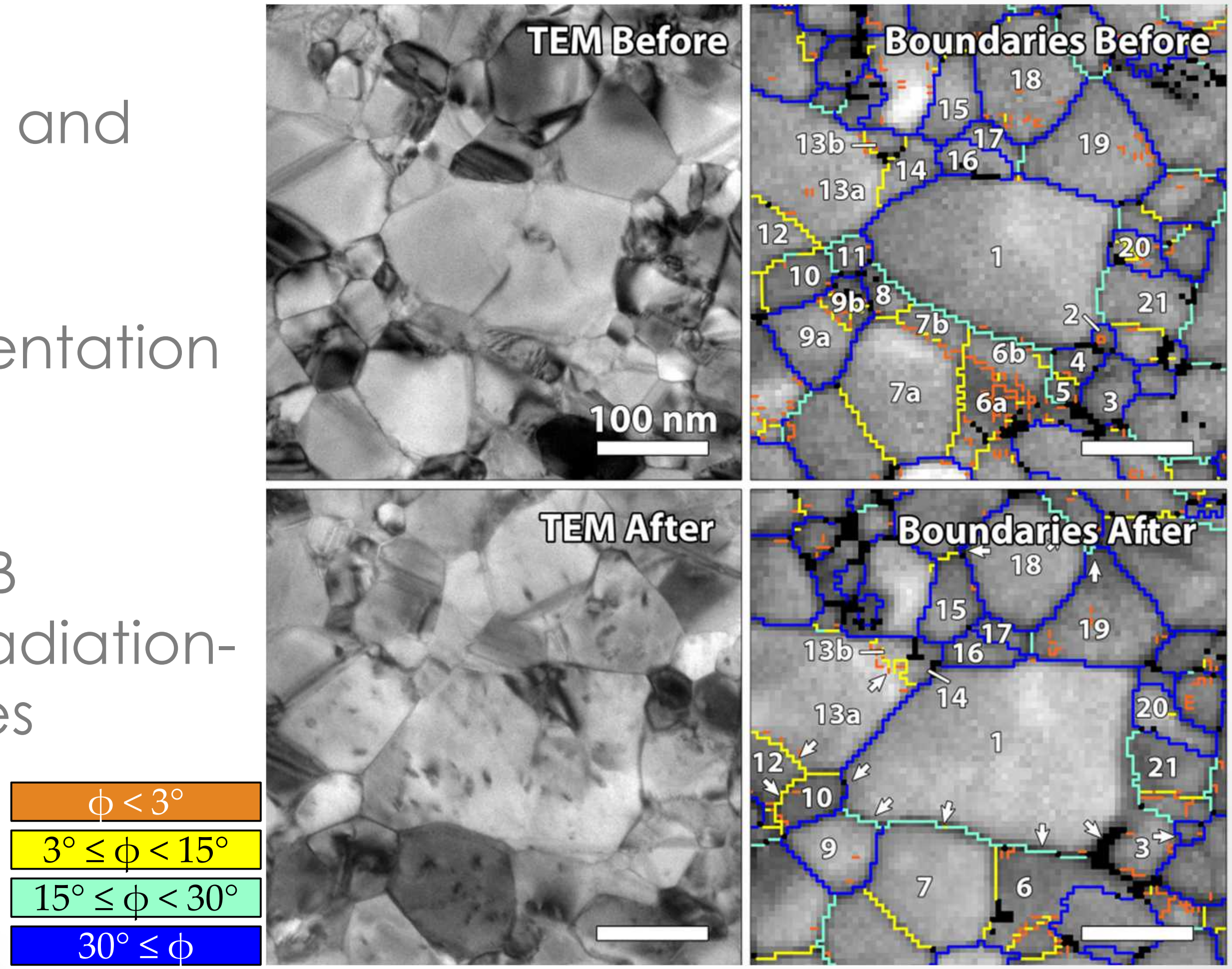
Bufford, *et al*, Appl Phys Lett, 2015.

- Same area characterized before and after irradiation.
- Standard TEM
- Orientation maps
  - Local grain size, orientation, boundary character
  - Hundreds of grains counted in minutes

Rapid quantification of statistically relevant numbers of grains and boundaries.

GB Movement: Local

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

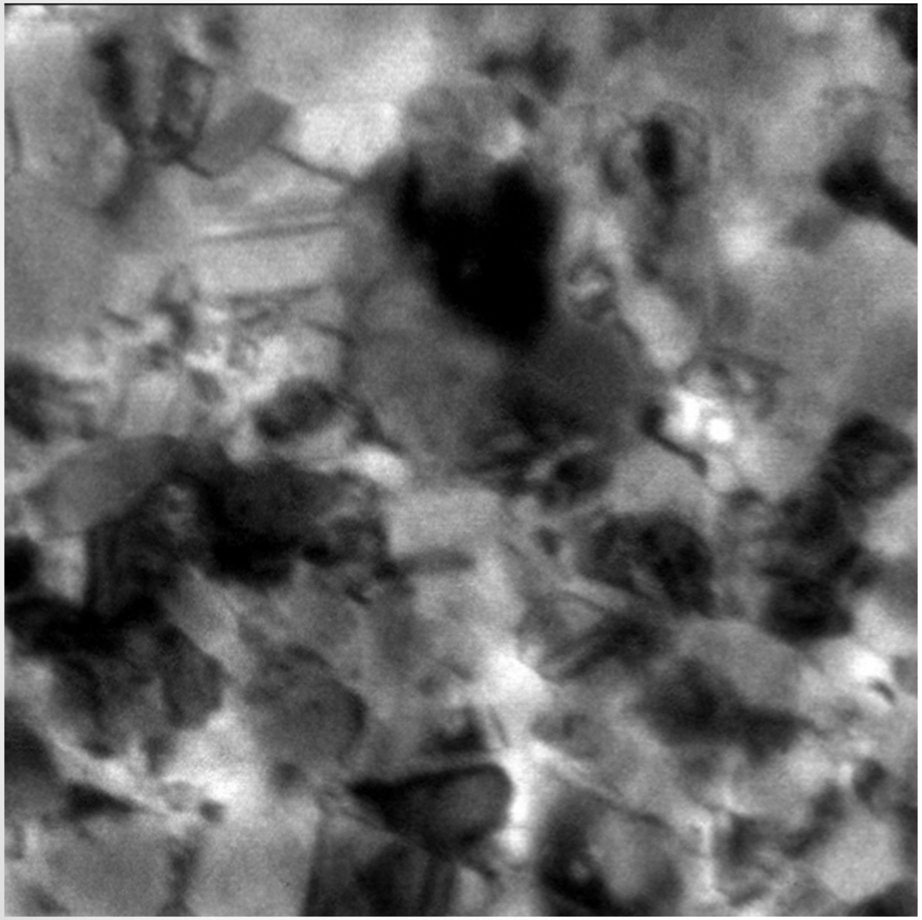


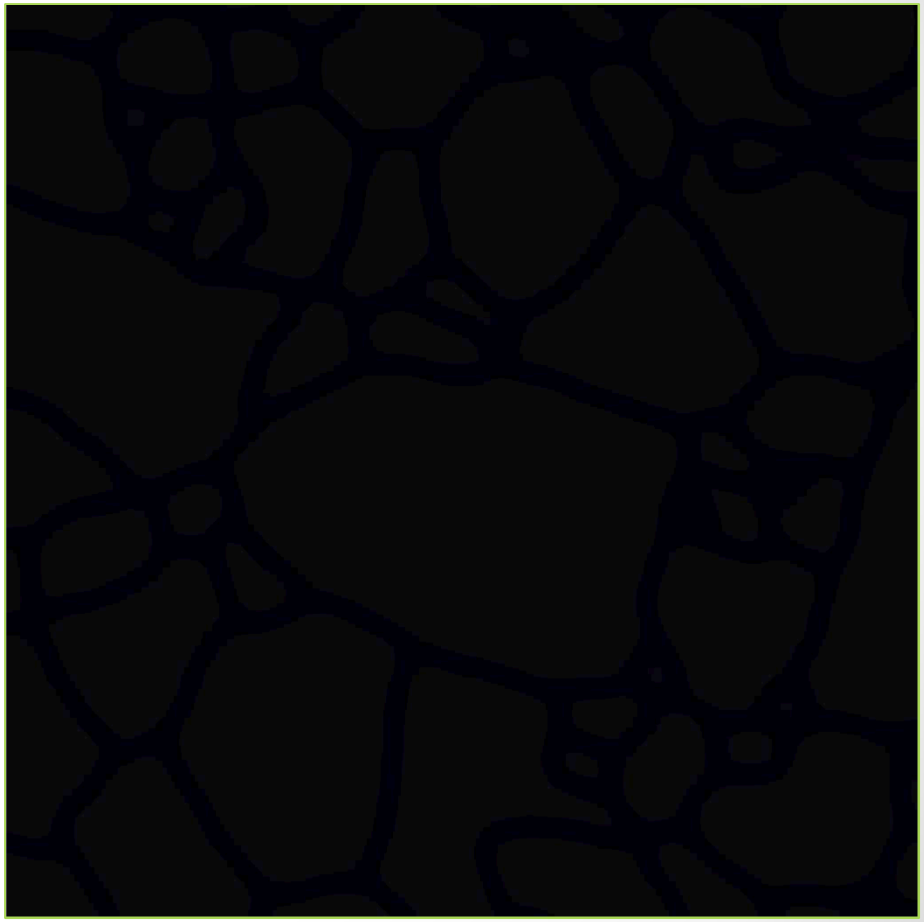
Bufford, *et al*, Appl Phys Lett, 2015.

Grain boundary misorientation angle and axes quantified

Simulated Irradiation

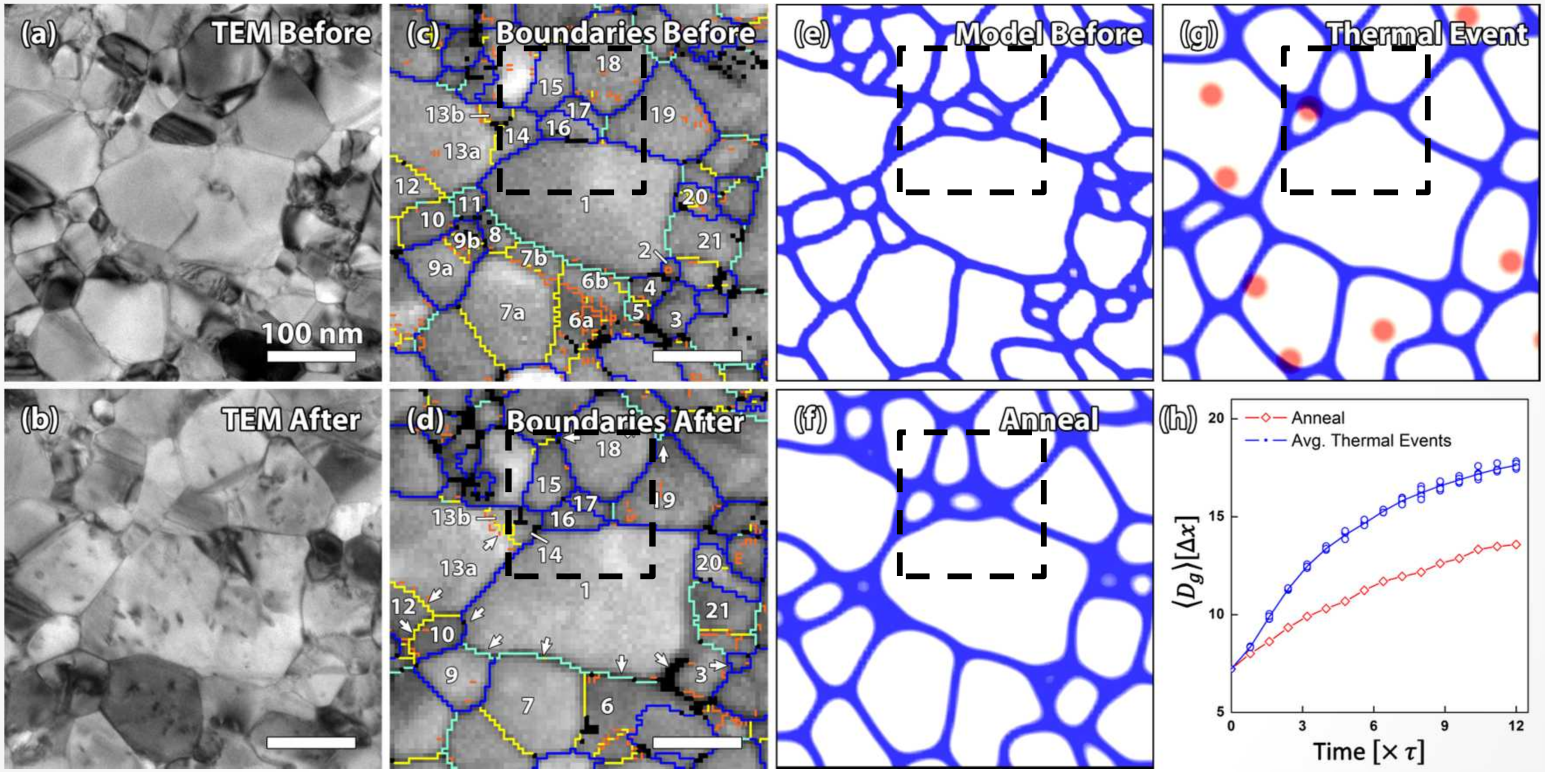
Collaborators: F.F. Abdeljawad and S.M. Foiles





2× real time

Experiment/Model Discrepancies?

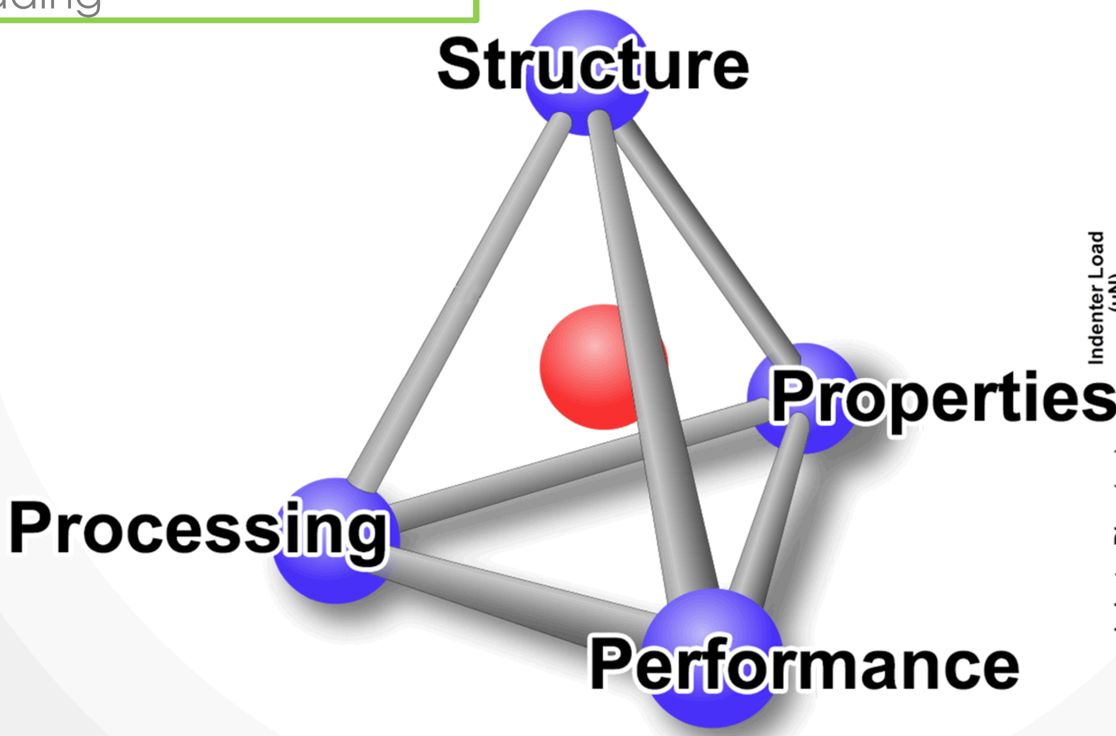


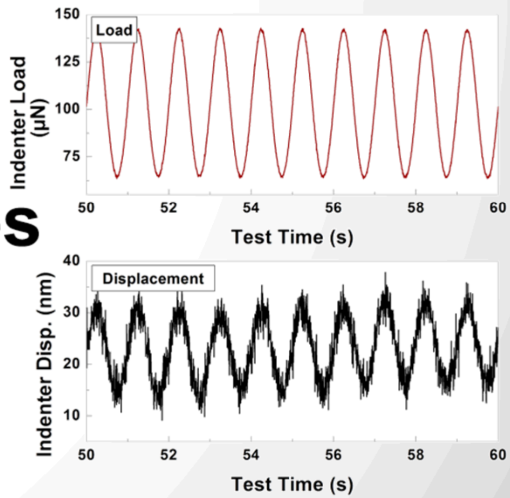
Bufford, *et al*, Appl Phys Lett, 2015.

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

Immobile boundaries suggest importance of non-thermally activated mobility

- Ion irradiation
  - Ion beam modification
- Mechanical behavior
  - Monotonic and cyclic loading





In Situ TEM Nanoindentation

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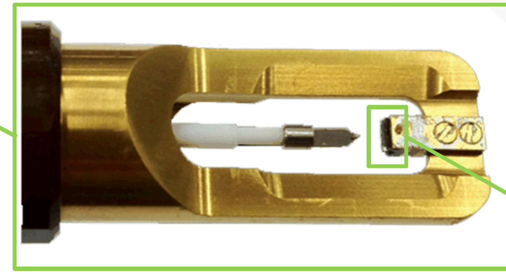
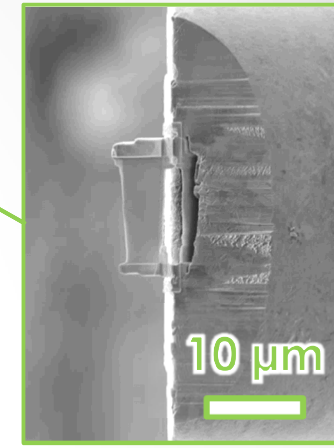
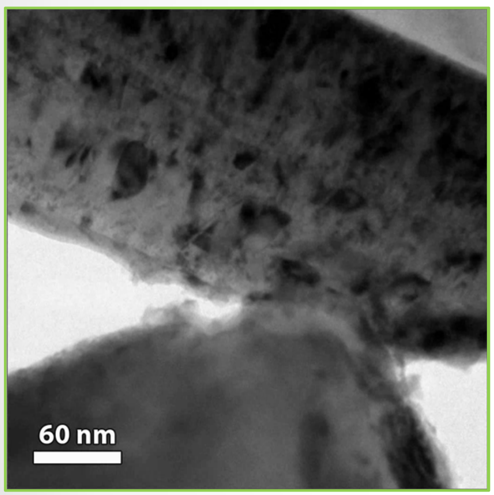
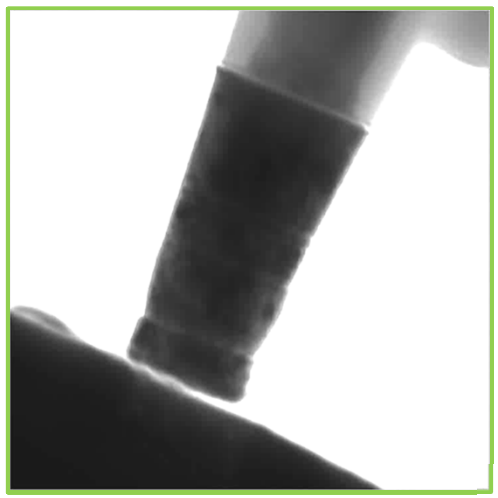


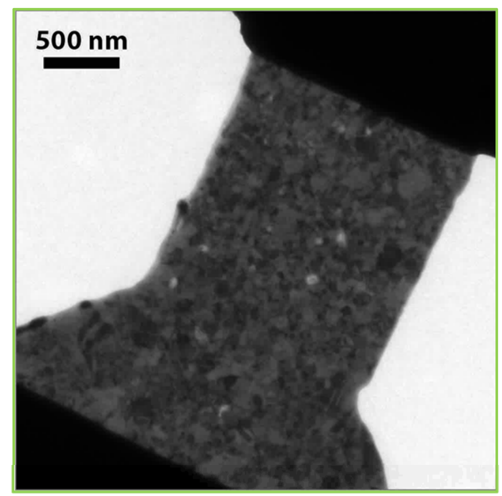


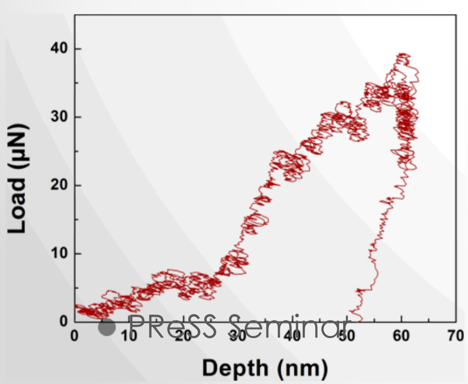
In Situ Nanoindentation TEM Holder

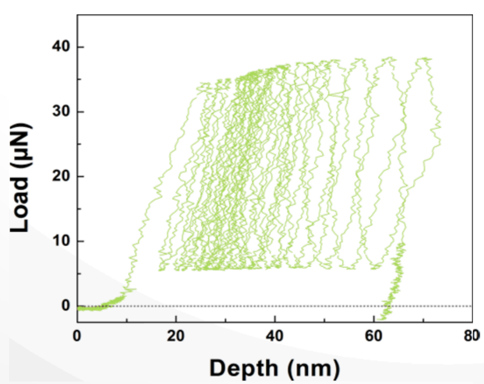
- Sub nanometer displacement resolution
- Quantitative force information with µN resolution

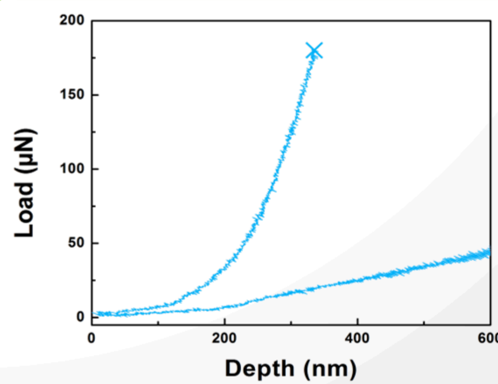






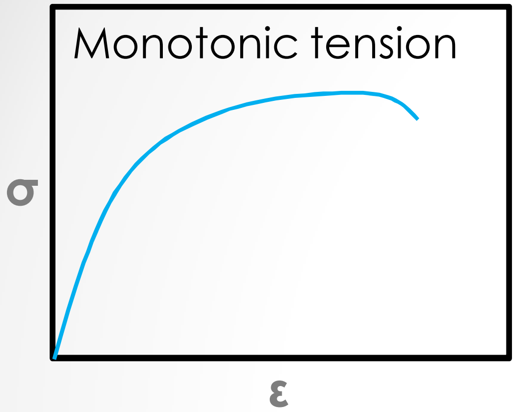




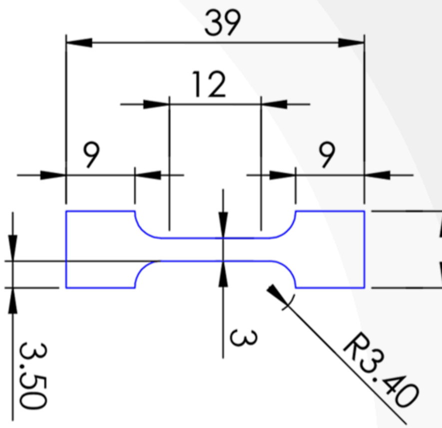


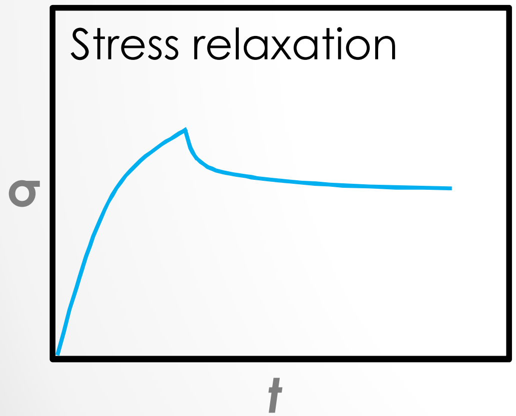
- A variety of sample geometries
- Load functions: monotonic, cyclic/fatigue, creep, stress relaxation.

Tensile Testing

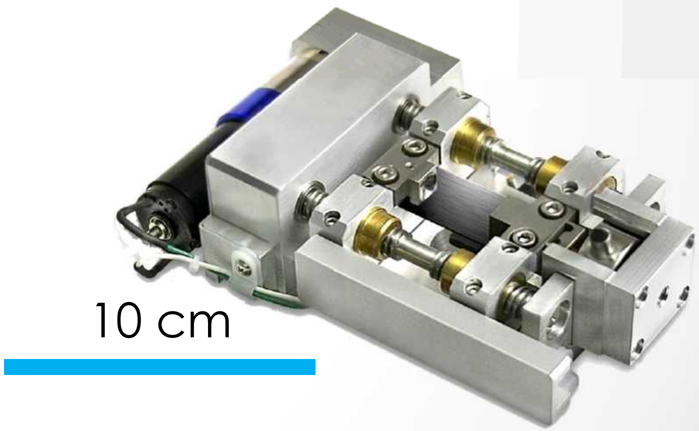


- Lots of information:
  - E, σ_y, σ_{UT}, elongation, toughness, n, m





- More information:
  - m, ΔV, creep

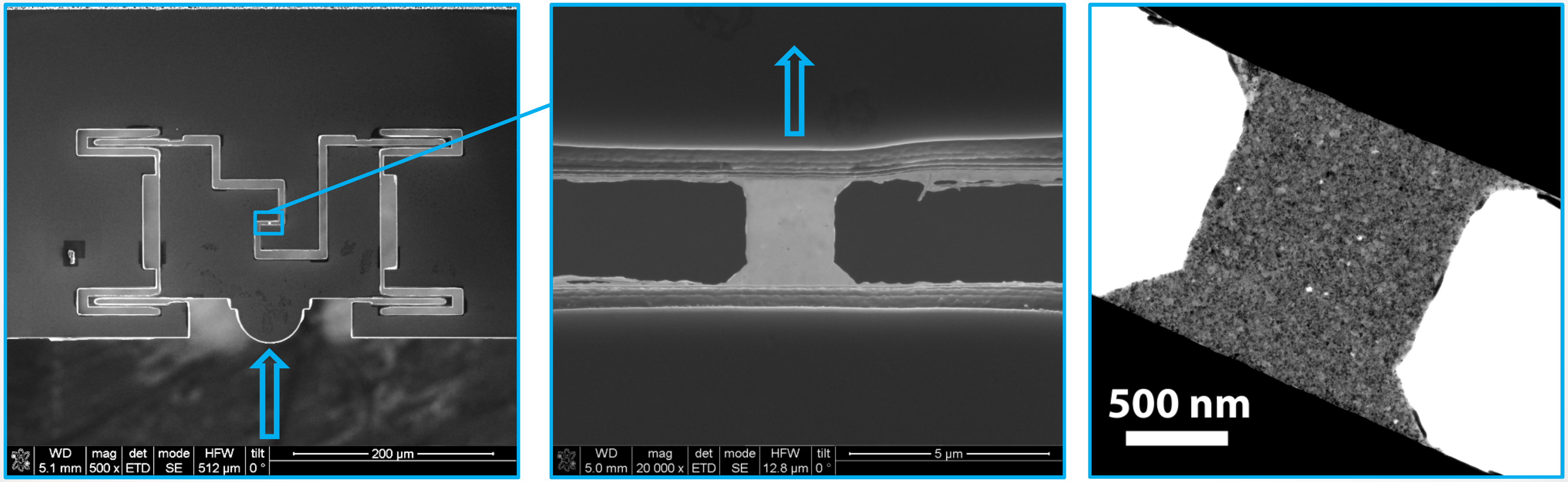


- **Gold standard for bulk mechanical properties**
- **Requires well formed, destructible specimens**

Tension Specimen Fabrication

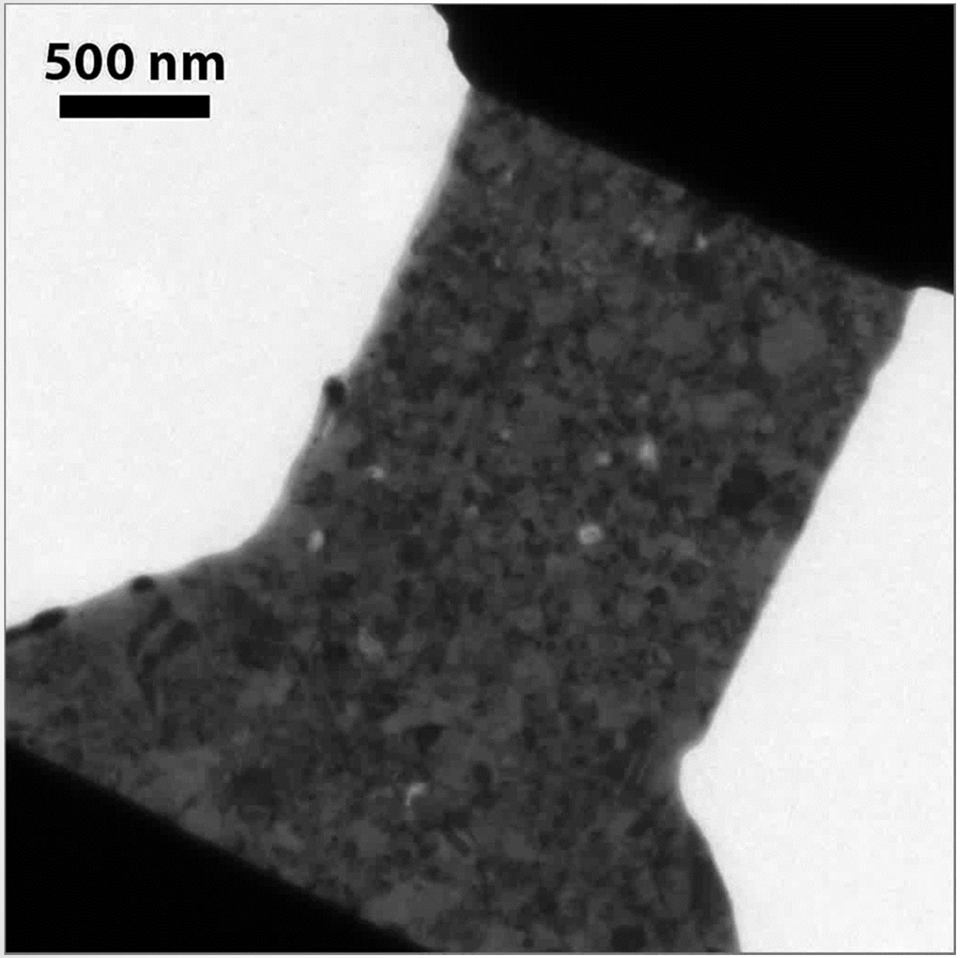
- Hysitron "Push-to-Pull" devices
  - Microfabricated Si test frame
  - Cu film (75 nm) floated onto device, then FIB milled

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

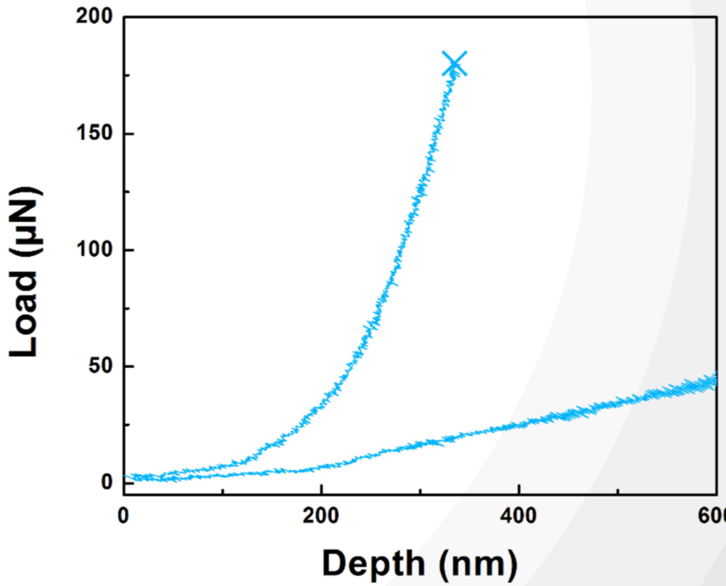


- Nearly pure tension, uniform cross sectional area, stable load frame
- Thin foil geometry not ideal for mechanics, but is electron-transparent

Monotonic



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



Successful quantitative tensile testing in the TEM

Cyclic Loading

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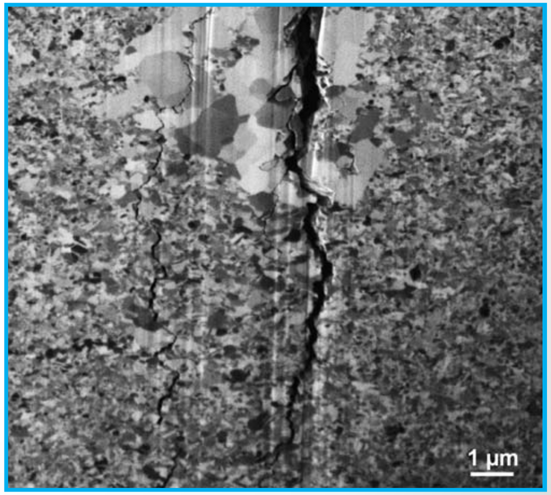
- Fatigue in bulk metals
  - Progressive microstructural change with cyclic loading, often below yield stress
- Fatigue in nc metals
  - Grain boundary migration and grain growth
  - Crack initiation
- What are the underlying mechanisms associated with these phenomena?
  - Pre-deformation microstructure
  - Grain and grain boundary orientations

***In situ* TEM deformation techniques are capable of investigating these questions.**

Execcharter, 2011.

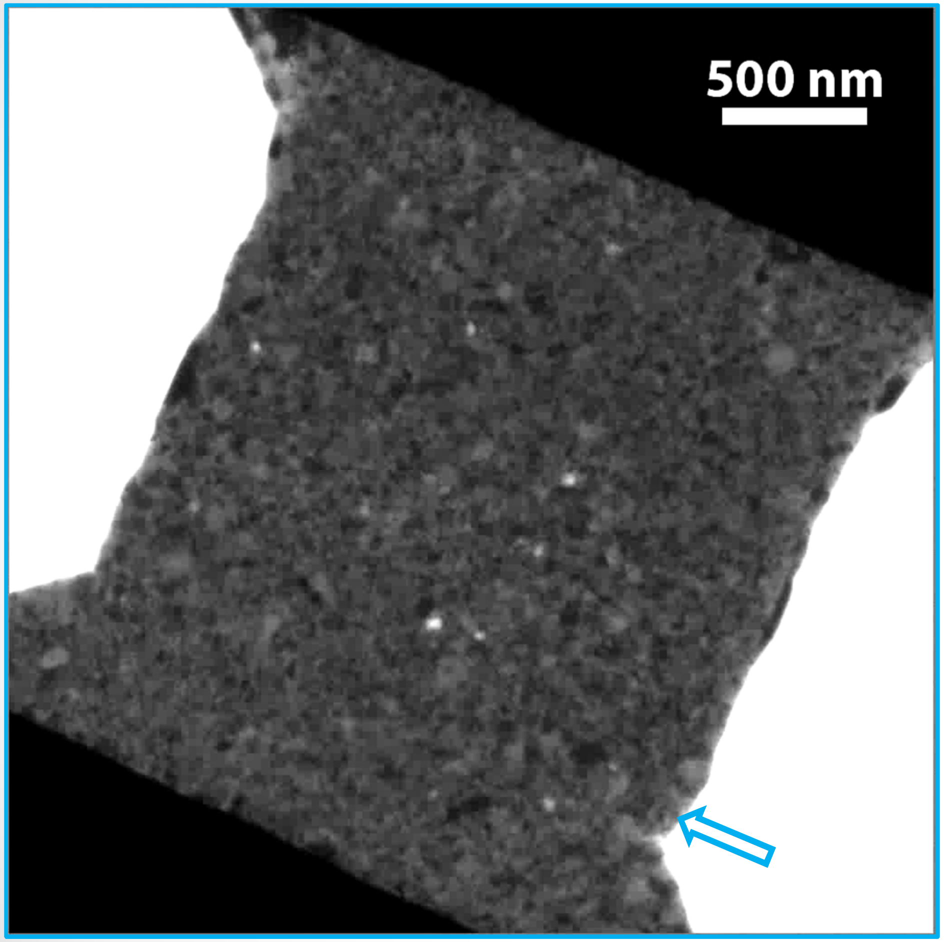


Padilla and Boyce, Exp Mech 2006.

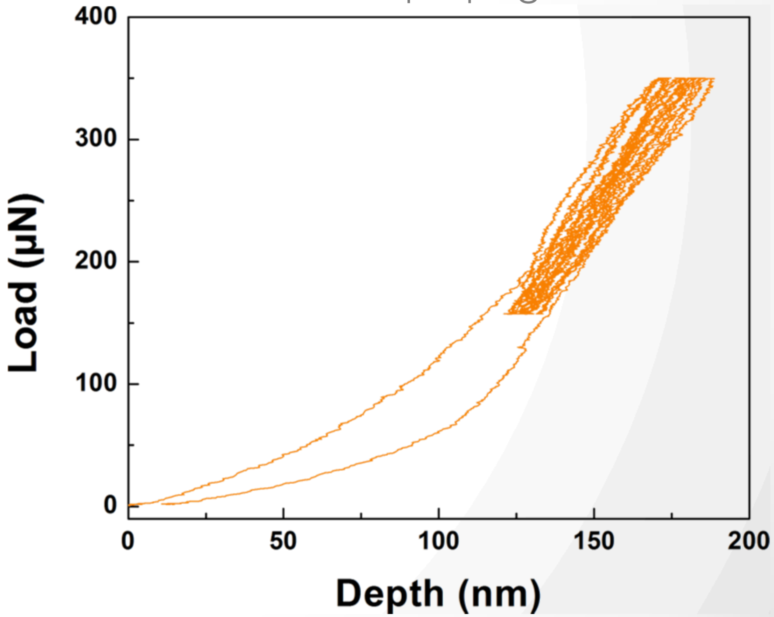
Low Cycle Fatigue

Video playback ×10

Collaborator: W. Mook



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



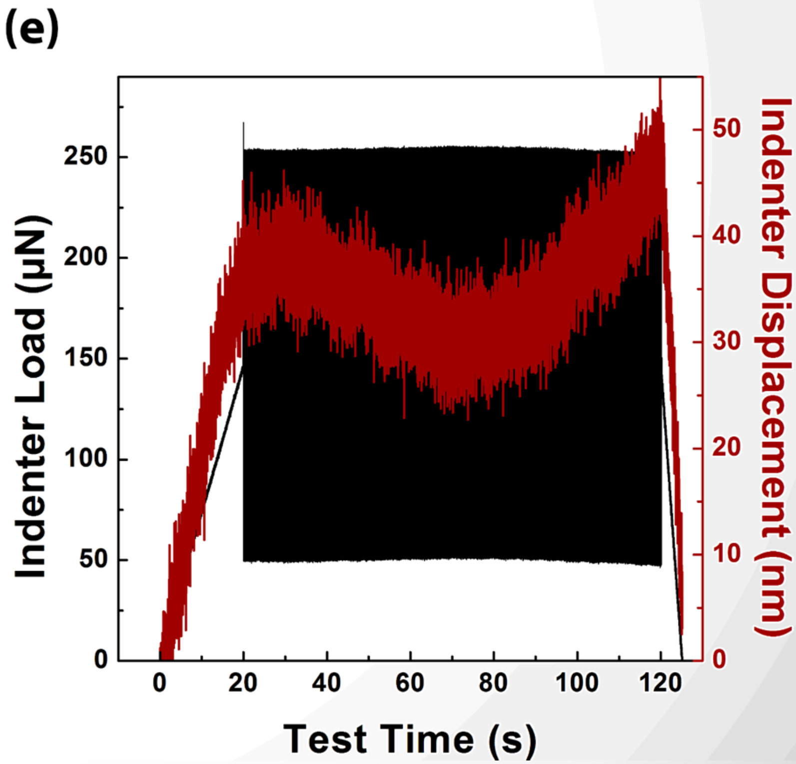
- Direct measurements of fracture parameters
- Structural evolution at the crack tip

High Cycle Fatigue

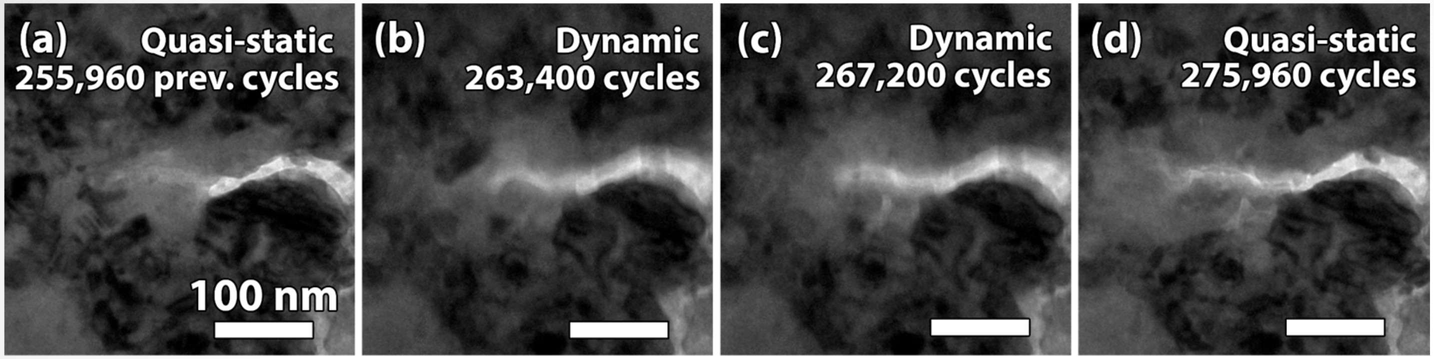
Collaborators: D. Stauffer, B. Boyce, K. Hattar, W. Mook

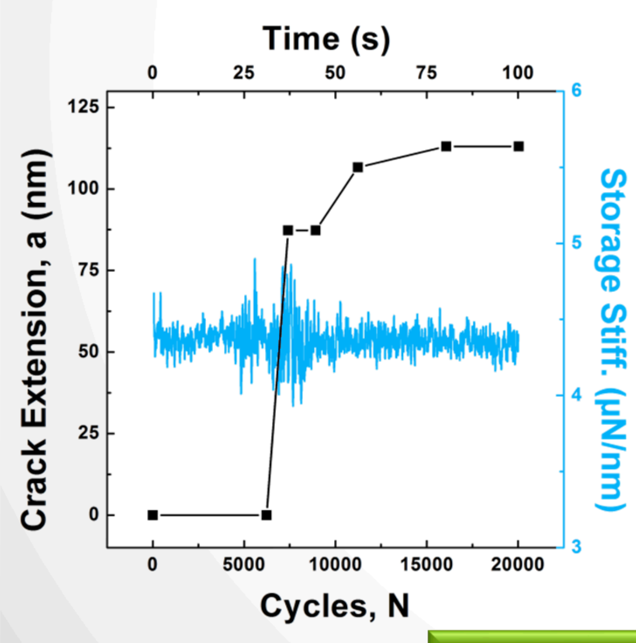
Video playback ×3

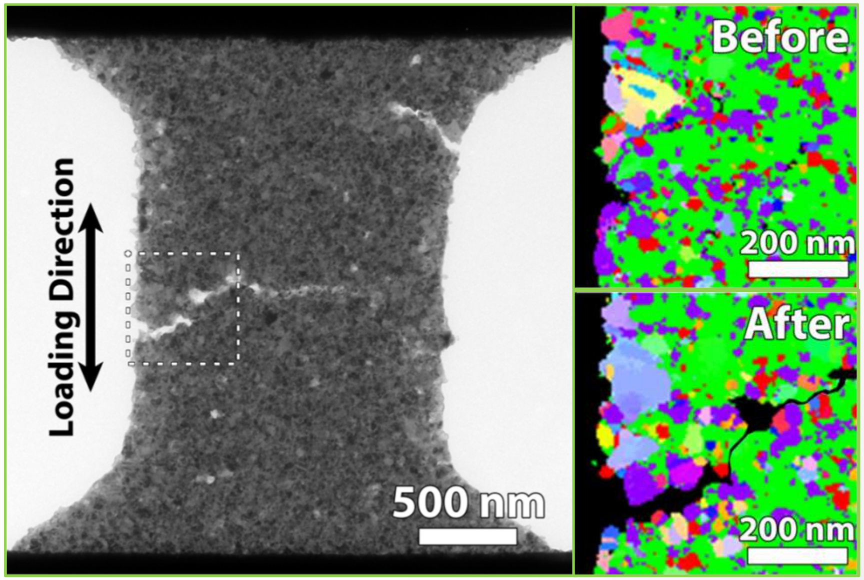
- Cyclic loading:
  - 200 hz



Crack Growth Quantified







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

Summary and Conclusions

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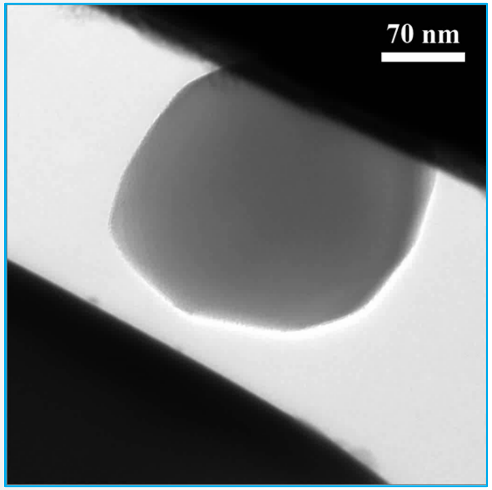
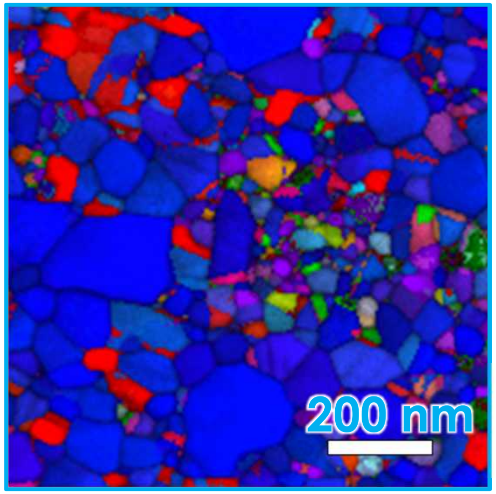
- Mechanical deformation and irradiation-induced grain growth studied with quantitative *in situ* TEM techniques.
- Immediately relevant to small-scale devices.
- Fundamental knowledge of processes at the nanoscale informs models and improves understanding at longer length scales.





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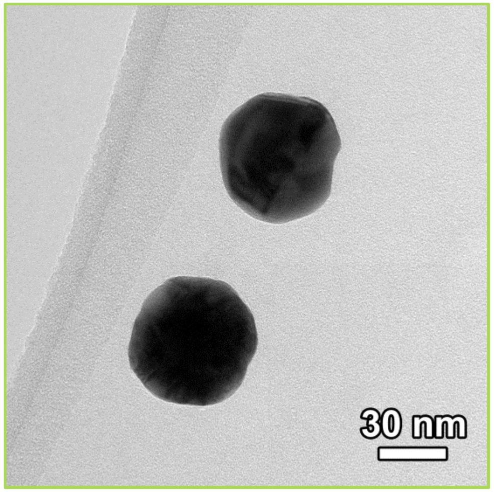
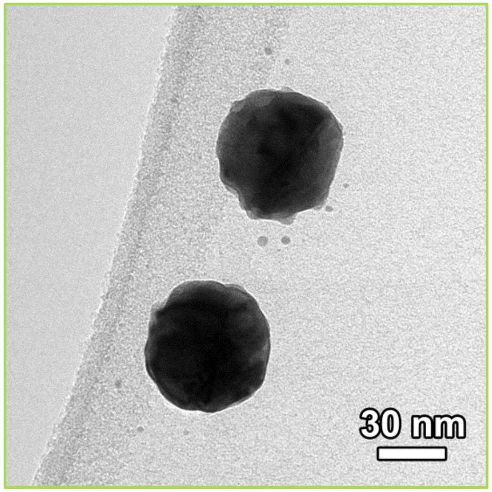
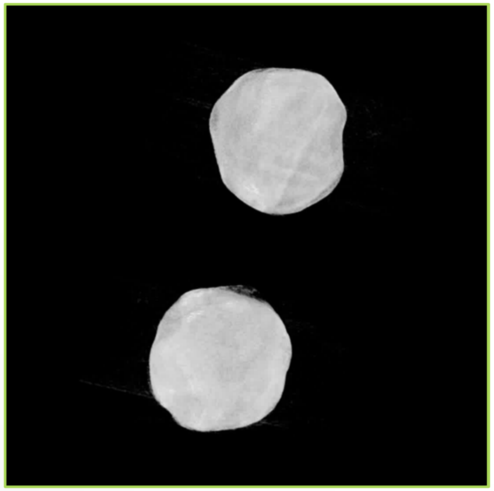
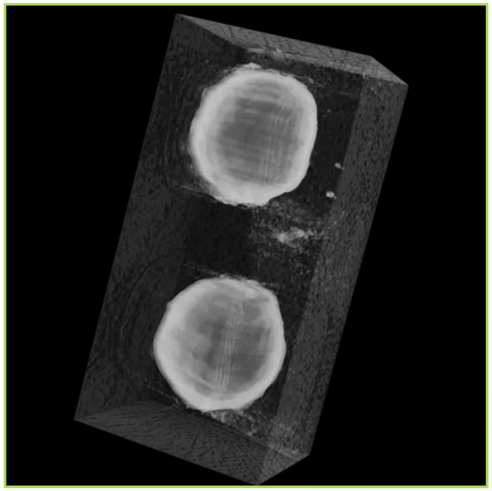
Surface Effects of Heavy Ions





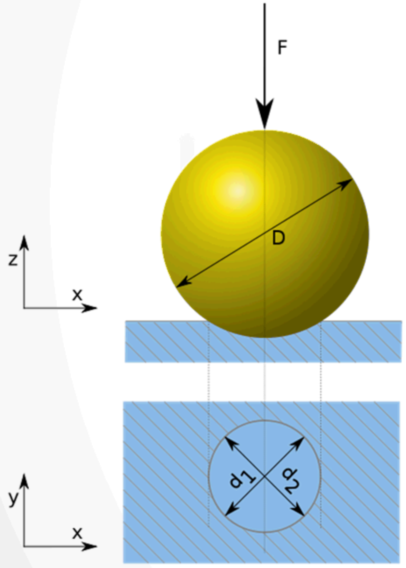
- 60 nm Au NPs before/after 2.0×10^{14} ions/cm^2 of 2.8 MeV Au^{4+}





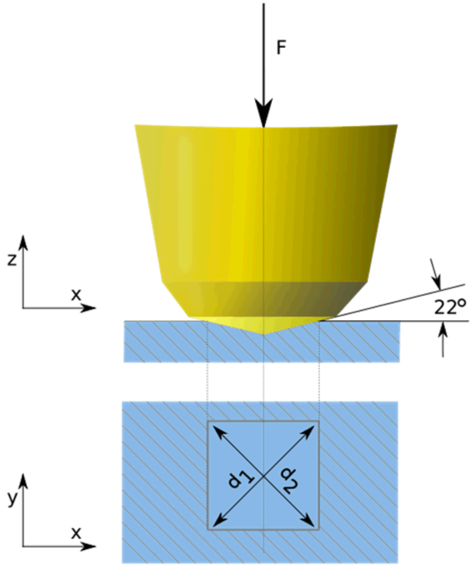
Macro/Microindentation

- 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.

Nanoindentation

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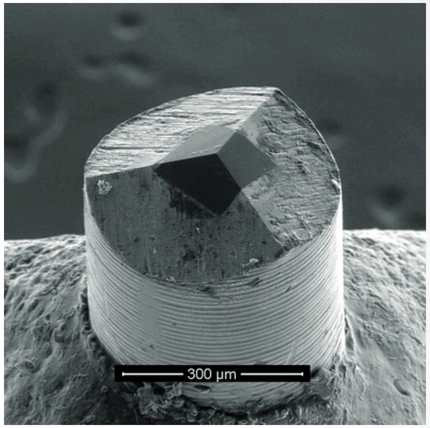
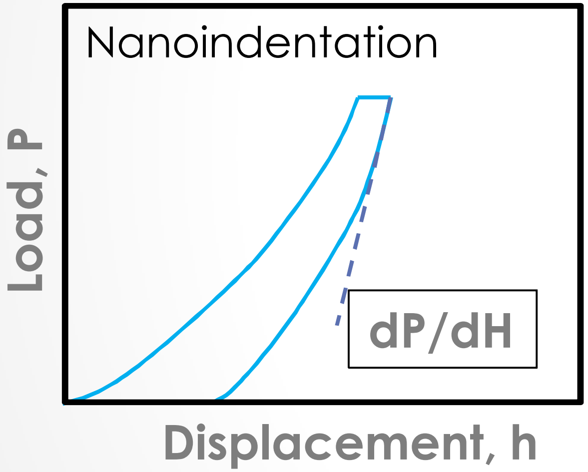
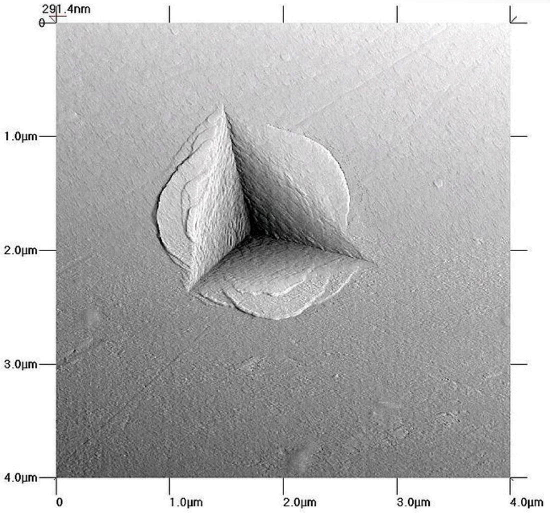
- Apply a load
- Measure force and depth continuously
- Measure or compute residual area





- Depths from tens of nm to µm
- Modulus and rate sensitivity

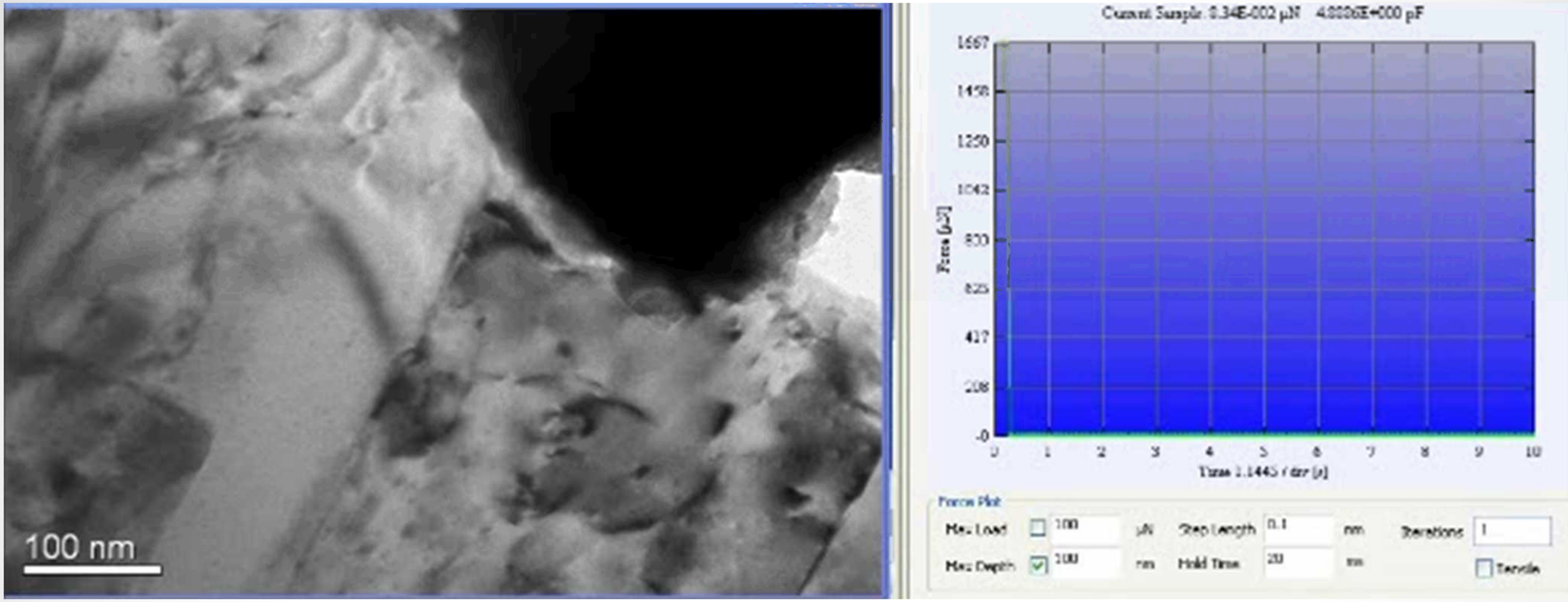


J. Puthoff, *via* Wikimedia Commons.

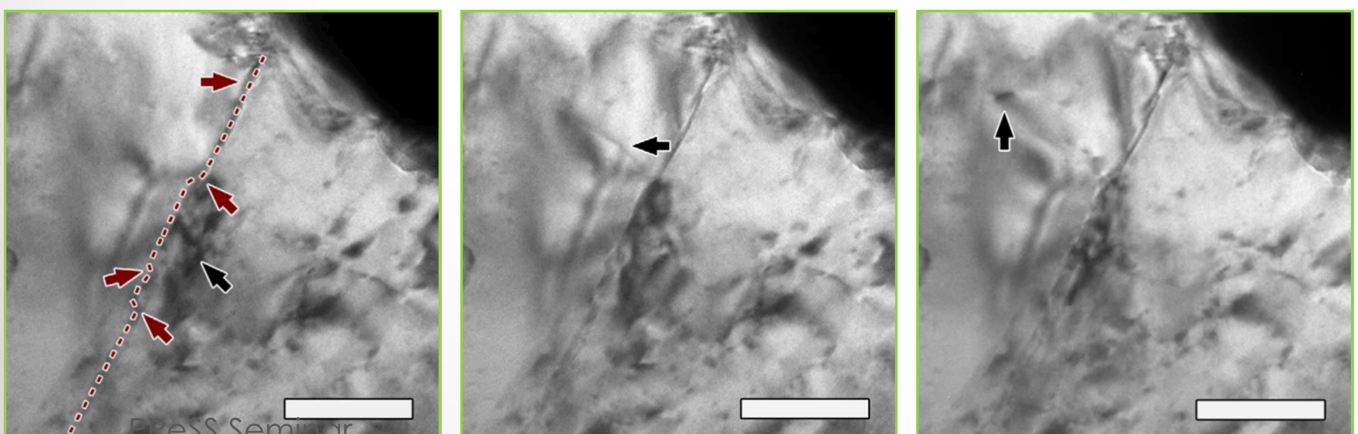
Quantifying mechanical response at the nanoscale.

Grain Boundary Yielding

Sandia National Laboratories



Video playback ×3



First observable intergranular plasticity event captured, with quantitative force measurement.

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

In Situ TEM Nanoindentation

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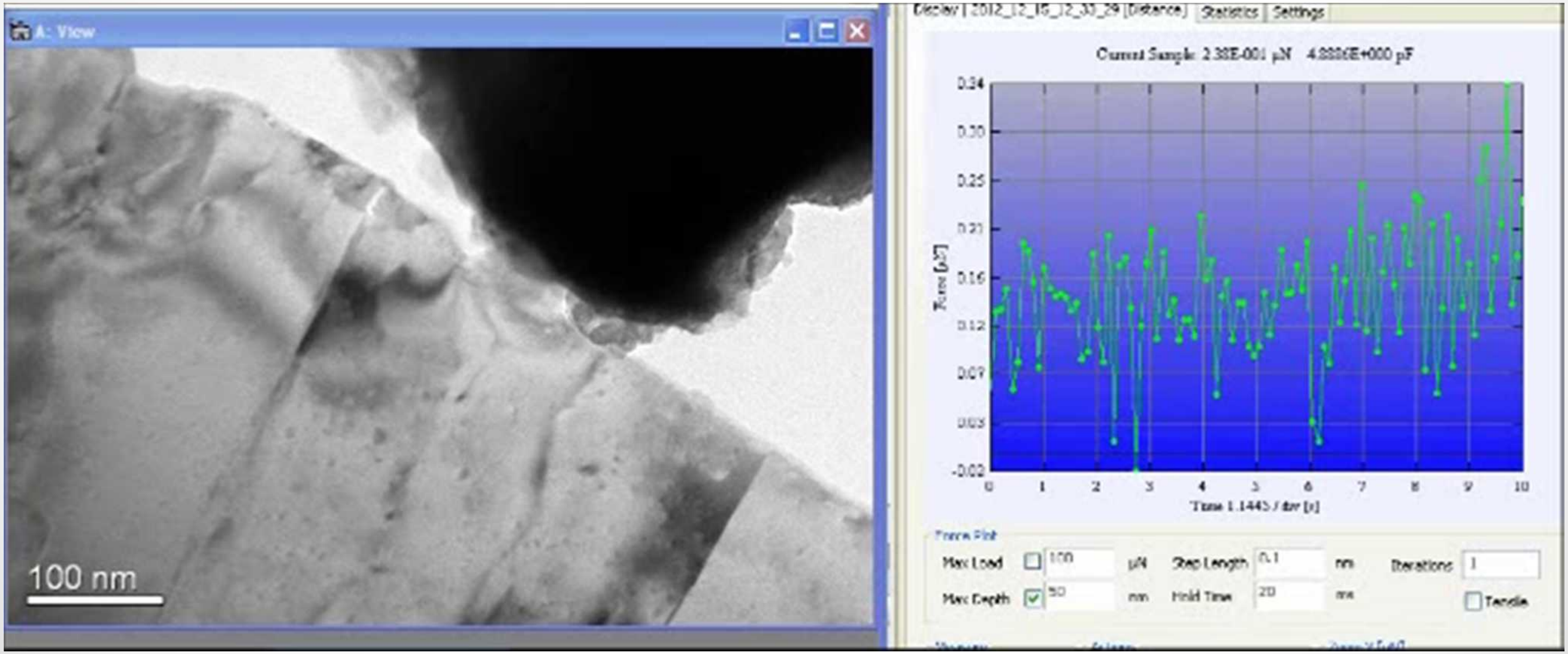
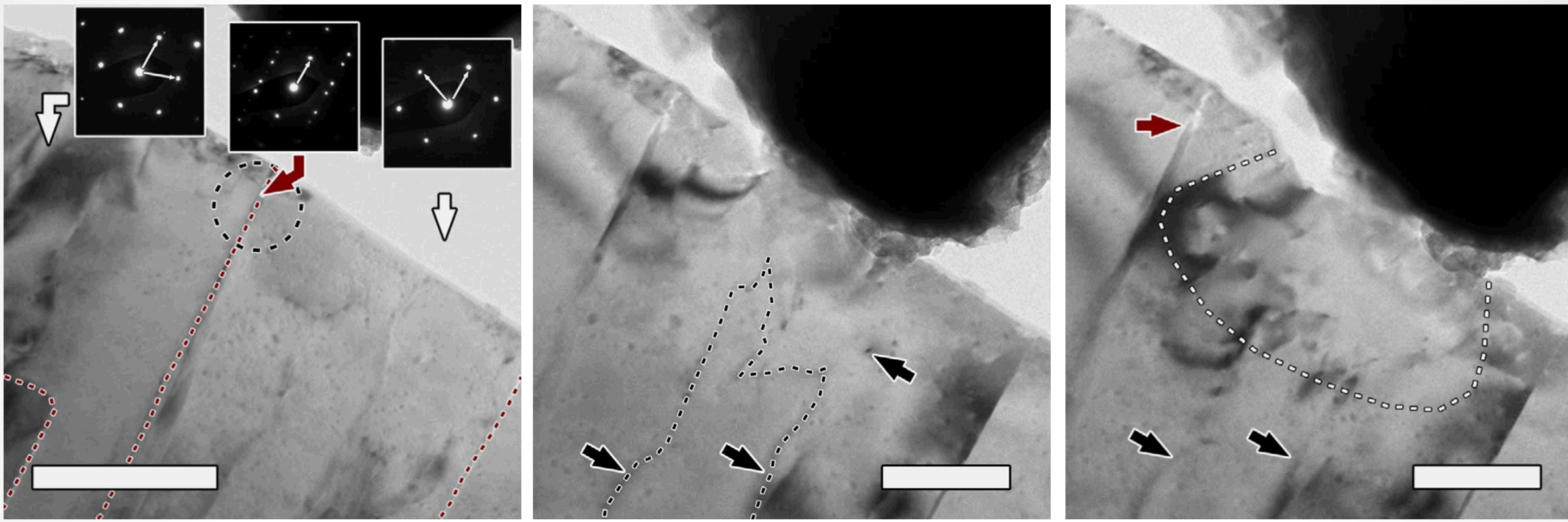
- *In situ* nanoindentation of Al near a Σ3{112} twin boundary
- Initial cycle

Video playback ×3



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

Video Snapshots



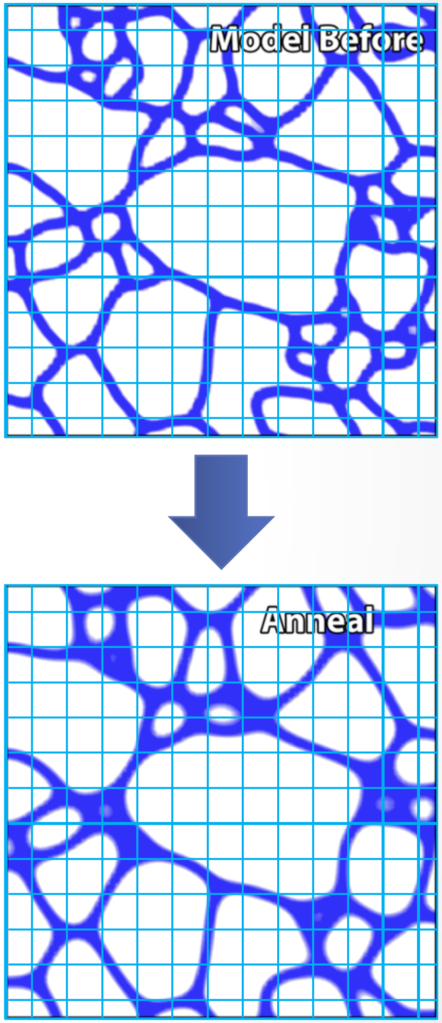
Bufford, *et al.*, Nat Commun 2014.

- 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.

Approach: Modeling

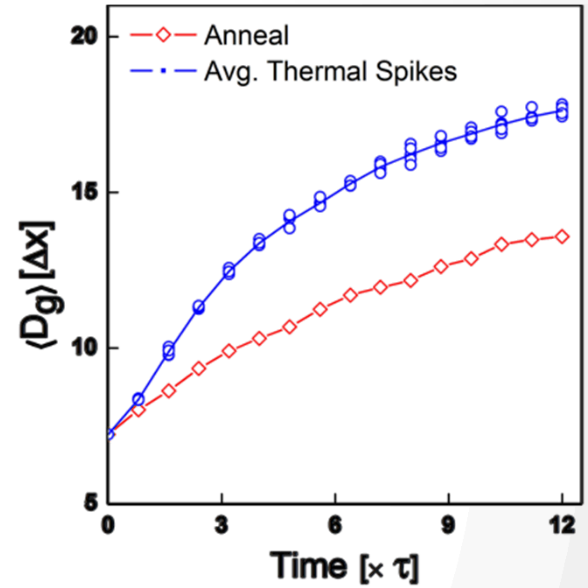
- What is phase field modeling?
  - Mathematical model for solving interfacial problems, like solidification, growth, etc.
- Example grain growth model
  - Thermodynamic free energy function
    - $dF = d(\gamma A) = \gamma dA$ (γ: GB energy, A: GB area)
  - Model for kinetics
    - $V = M\gamma h$ (M: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

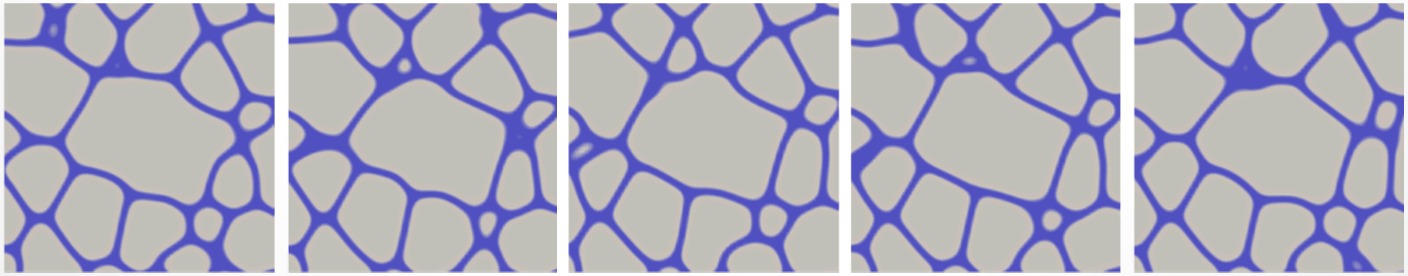


Can directly use experimental maps as input structures, and then compare evolutions!

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





Simulated Irradiation and Annealing

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