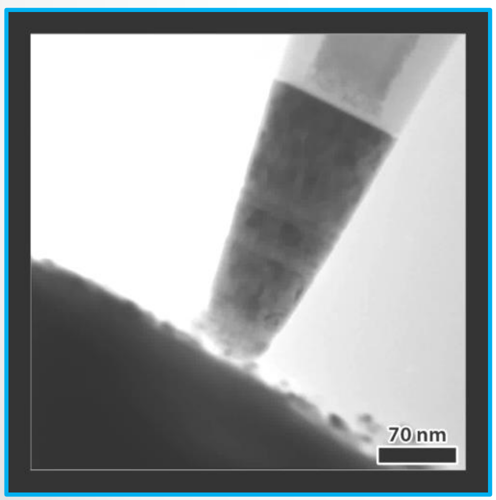
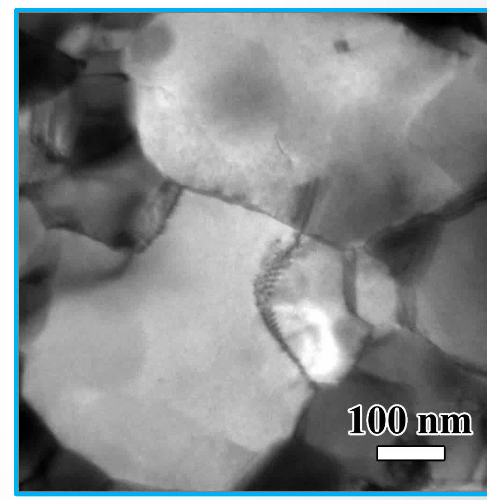
SAND2016-10557PE

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.



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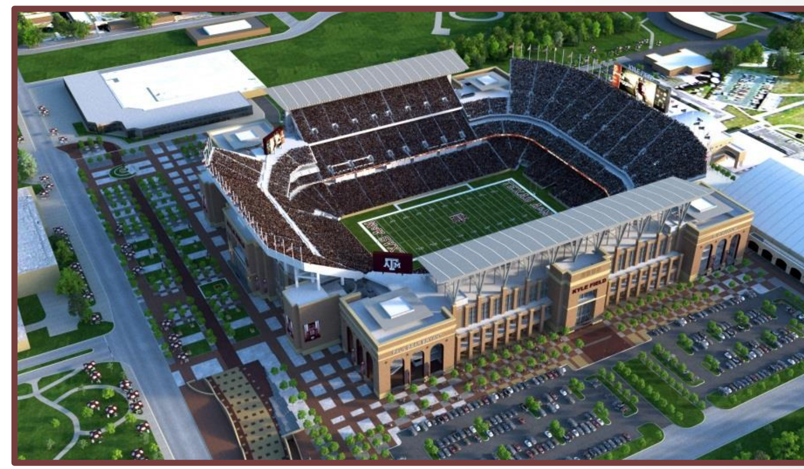
13 October, 2016

My Background

Sandia National Laboratories

Olin College of Engineering



TEXAS A&M UNIVERSITY

Sandia National Laboratories

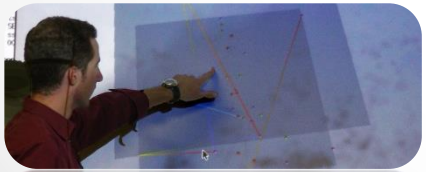
Sandia's National Security Mission

Sandia National Laboratories

Nuclear Weapons

International, Homeland & Nuclear Security

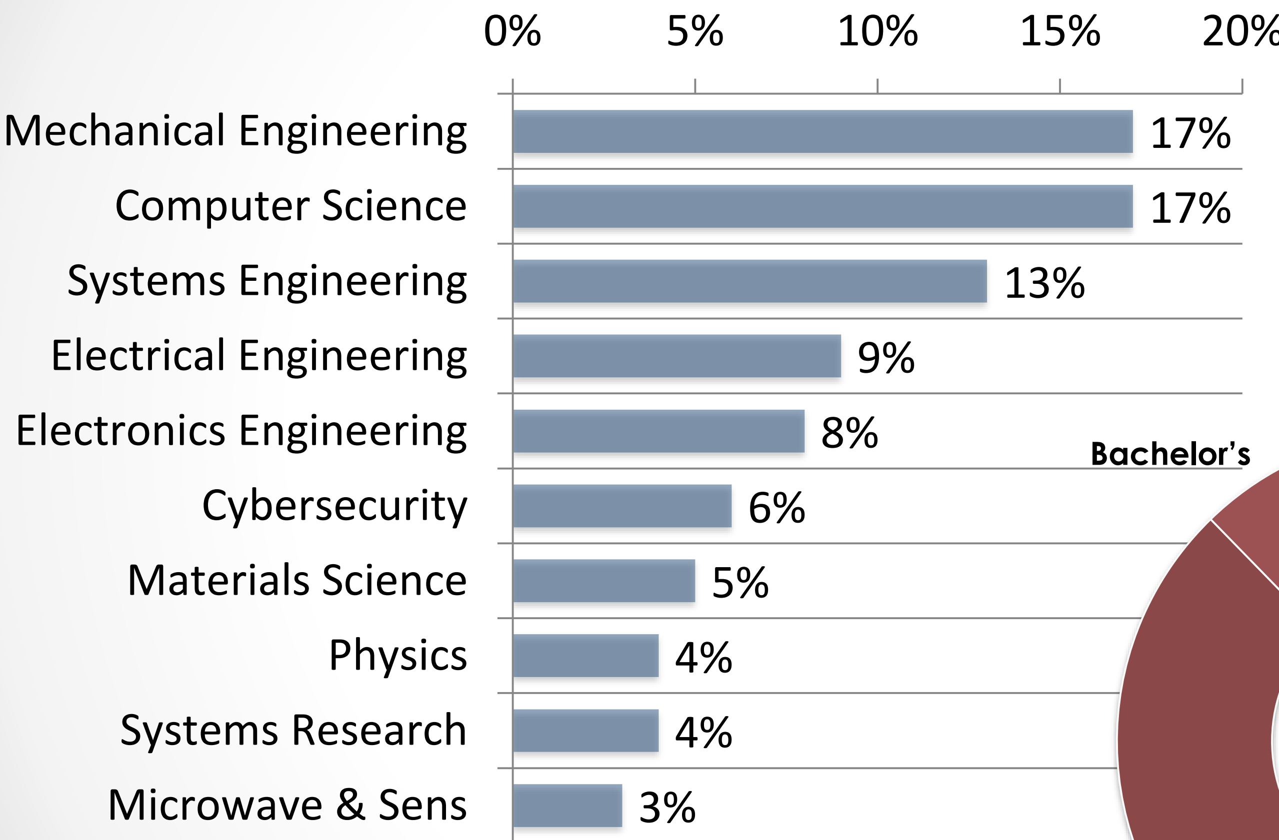
Energy & Climate



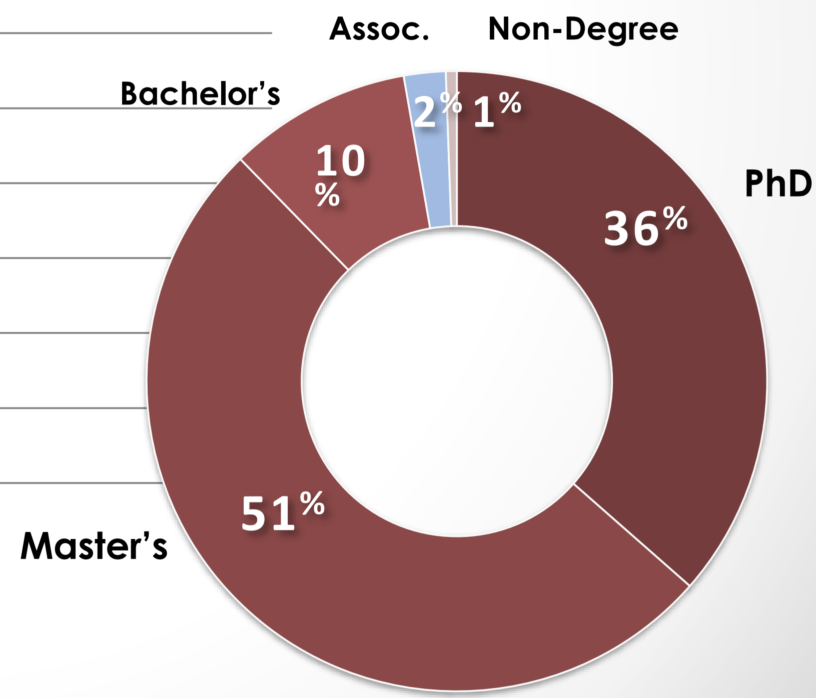
Defense Systems & Assessments

DOE National Laboratories

R&D by Discipline & Degree



Top 10 job descriptions shown, Regular exempt non-management employees only



Internship opportunities for high school through graduate students.

Data as of June 26 end of Q3 of the FY15

CINT: a DOE Office of Science National User Facility

Sandia National Laboratories

Core Facility (SNL)

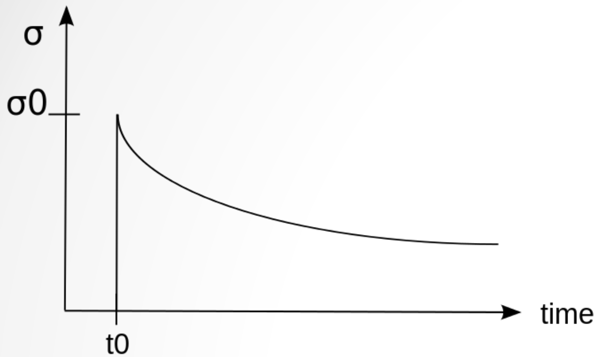
- TEM, SEM, FIB, XRD
- Low Temp Transport
- Scanning Probe Microscopy
- Ultra-fast Spectroscopy
- Molecular Beam Epitaxy
- Chem & Bio labs
- Molecular films
- E-beam lithography
- Photolithography
- Deposition & Etch

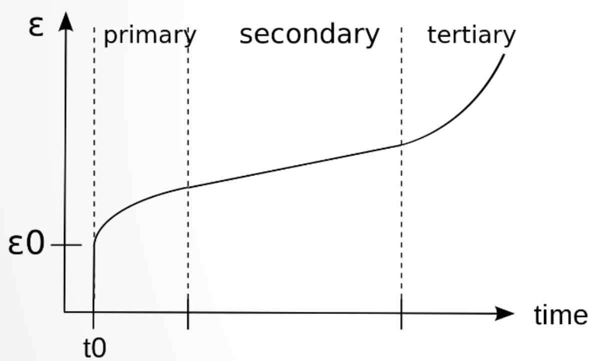
Gateway Facility (LANL)

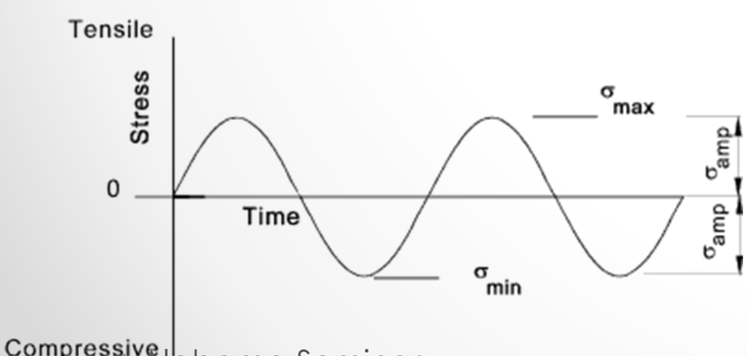
- Biomaterials & Chem synthesis
- XRD, SEM
- UV-vis, ellipsometry
- Nano-indentation
- Nanoscale optical probes
- Microscopies
- Physical Synthesis
- Pulsed Laser Deposition
- Ultra-fast Spectroscopy
- Computer Cluster
- Visualization Lab

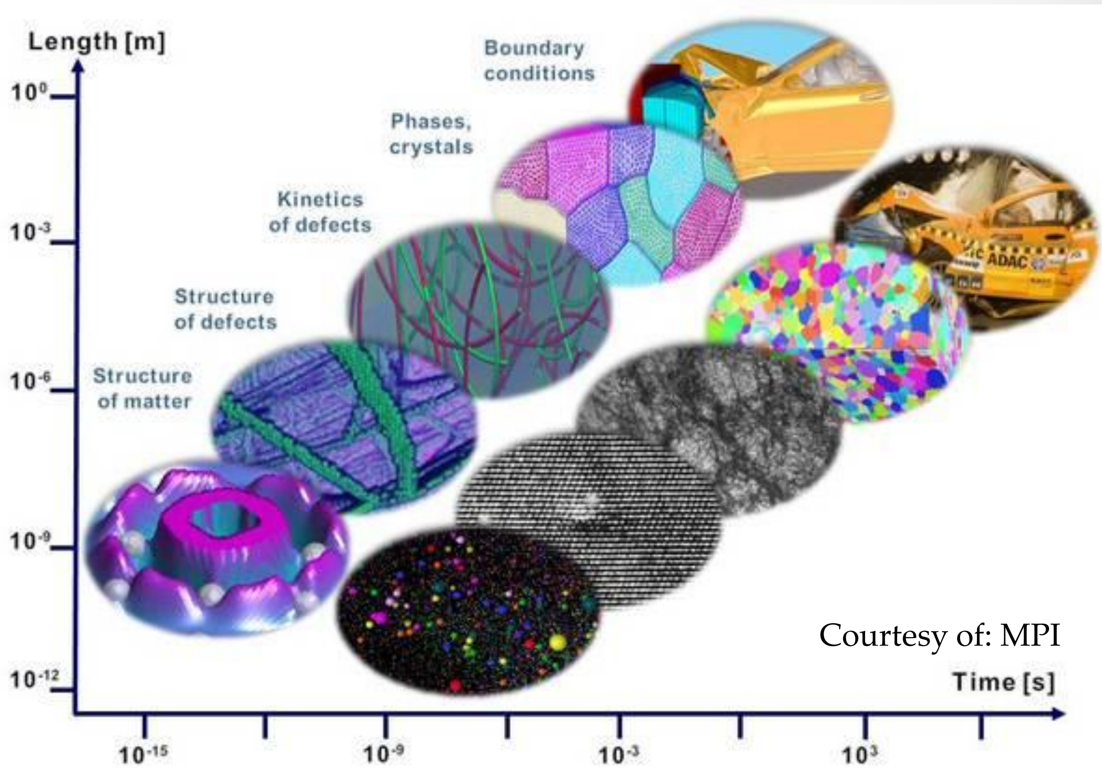
Many instruments/techniques in this talk available for use through CINT's competitive proposal-based system.

Material Evolution Over Time



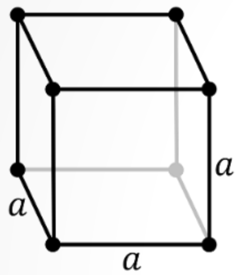




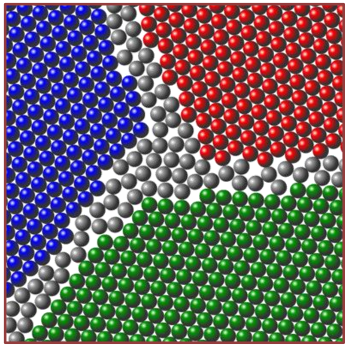


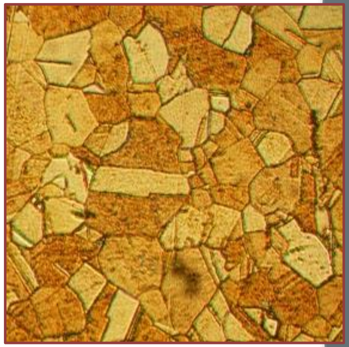
To develop predictive physics-based models, a fundamental understanding of the structure of matter, defects, and kinetics of structural evolution in the environments of interest are needed.

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

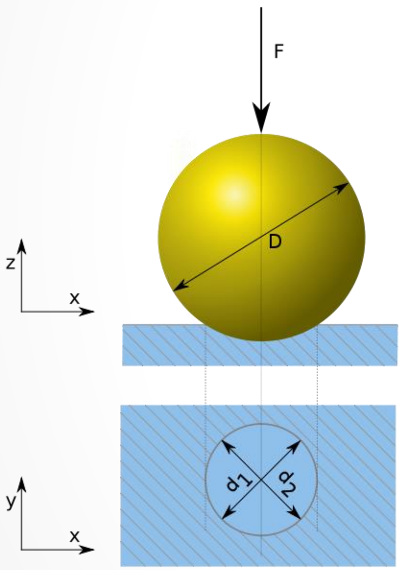
How does the internal structure affect material properties?

Roadmap

- Mechanical Testing
  - Bulk→ Micro → Nano
- *In Situ* TEM Mechanical Testing
- Irradiation
  - Grain growth

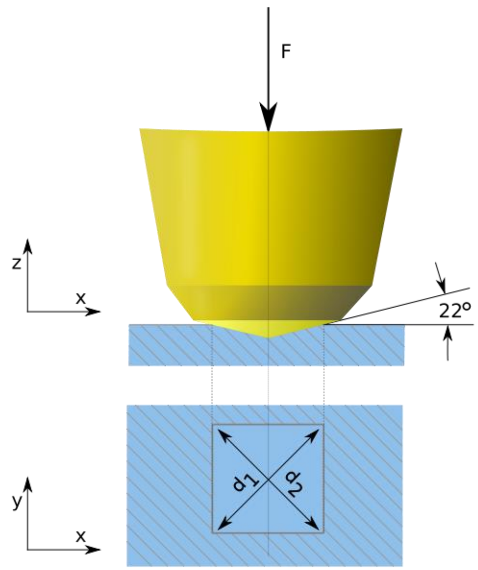
Macro/Microindentation

- 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

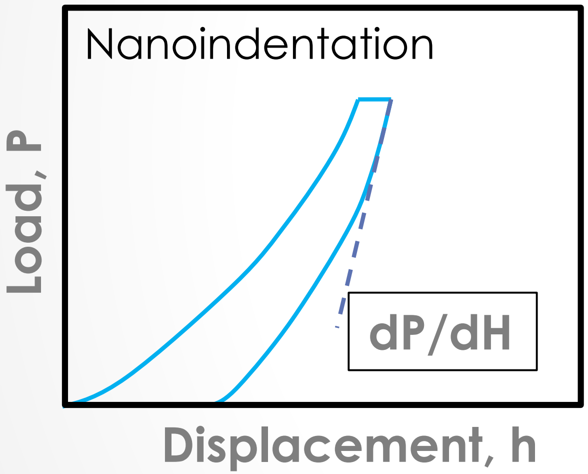
R. Tanaka, *via* Wikimedia Commons.

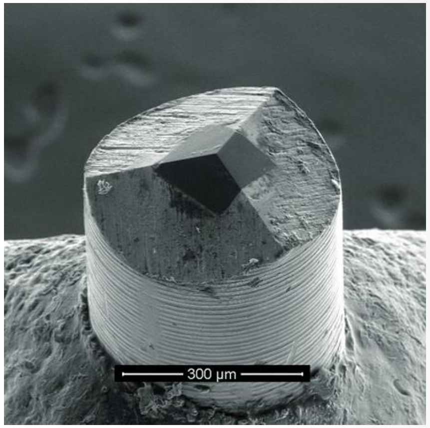
- Hardness, $H = \frac{P_{max}}{A_r}$

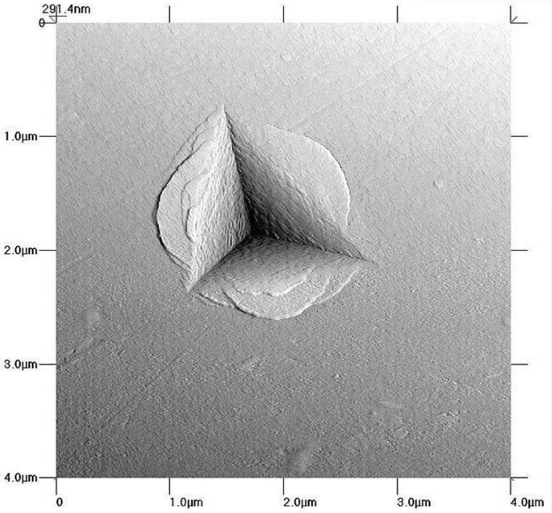
Infer bulk properties from local resistance to plastic deformation in "small" volumes.

Nanoindentation

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







J. Puthoff, *via* Wikimedia Commons.

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

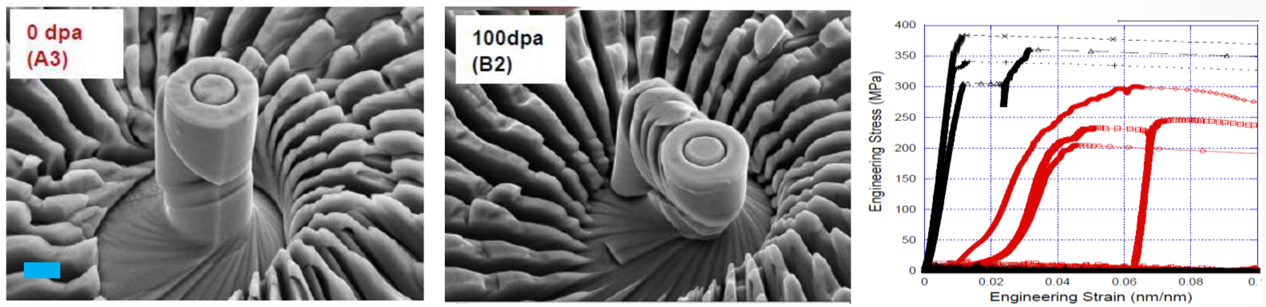
Quantifying mechanical response at the nanoscale.

Micro- and Nanoscale Testing

Sandia National Laboratories

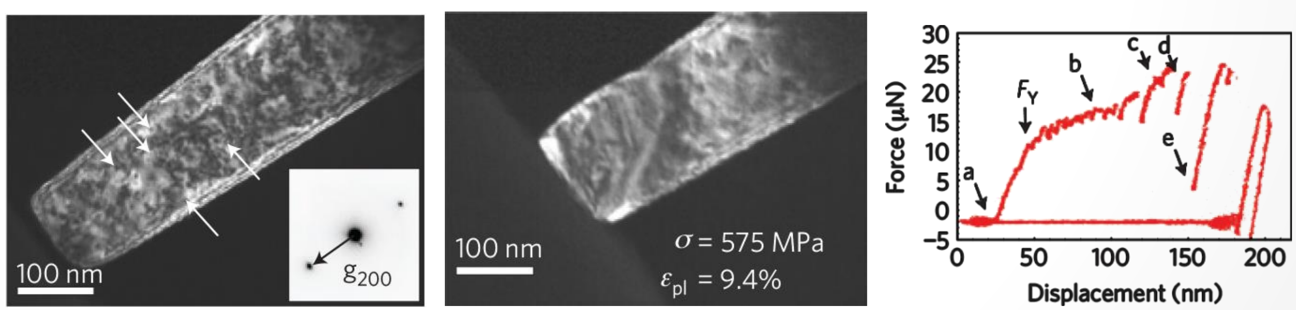
- Wealth of small-scale mechanical testing methods developed in last decade
- Capitalizing on advances in specimen preparation and testing instrumentation

30 MeV Cu^{5+}
100 DPA



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

1.1 MeV H^+
0.8 DPA



Kiener, *et al*, Nat Mater, 2011.

- **Approaching length scales of small grains, dislocation sources, etc.**
- **Small enough to be placed in a microscope**

Nanotwinned Metals

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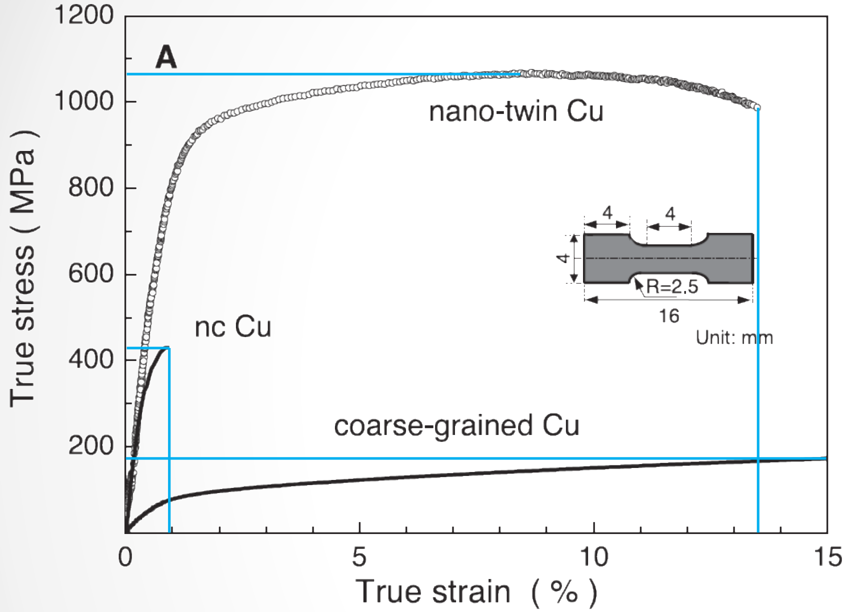
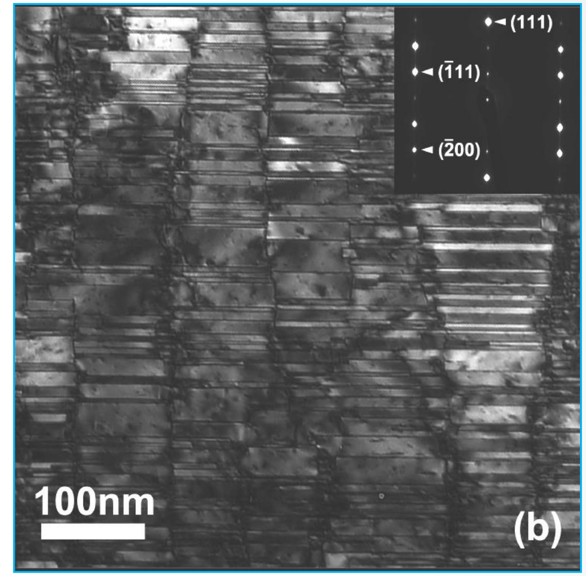
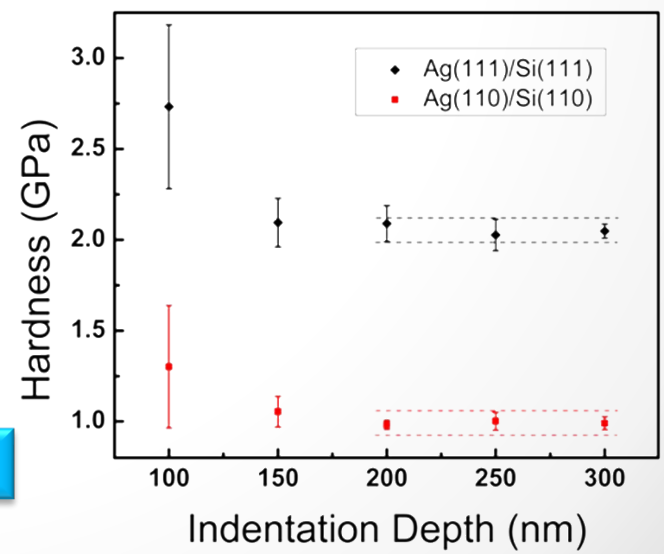


Lu, L. et al, *Science* **304, 2004.**



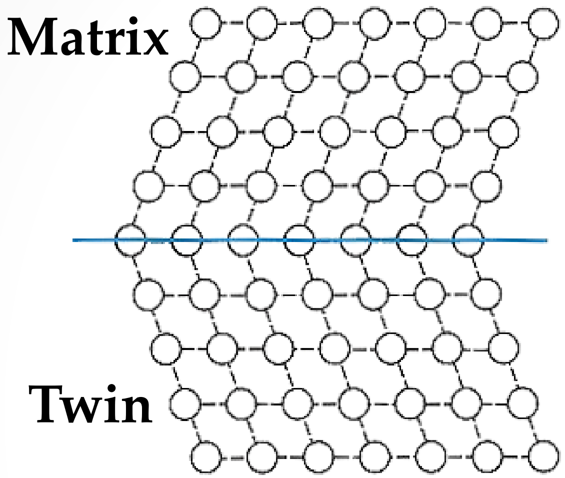


Bufford, D., et al. *Acta Mater*, **Vol. 59, 1, 2011.**

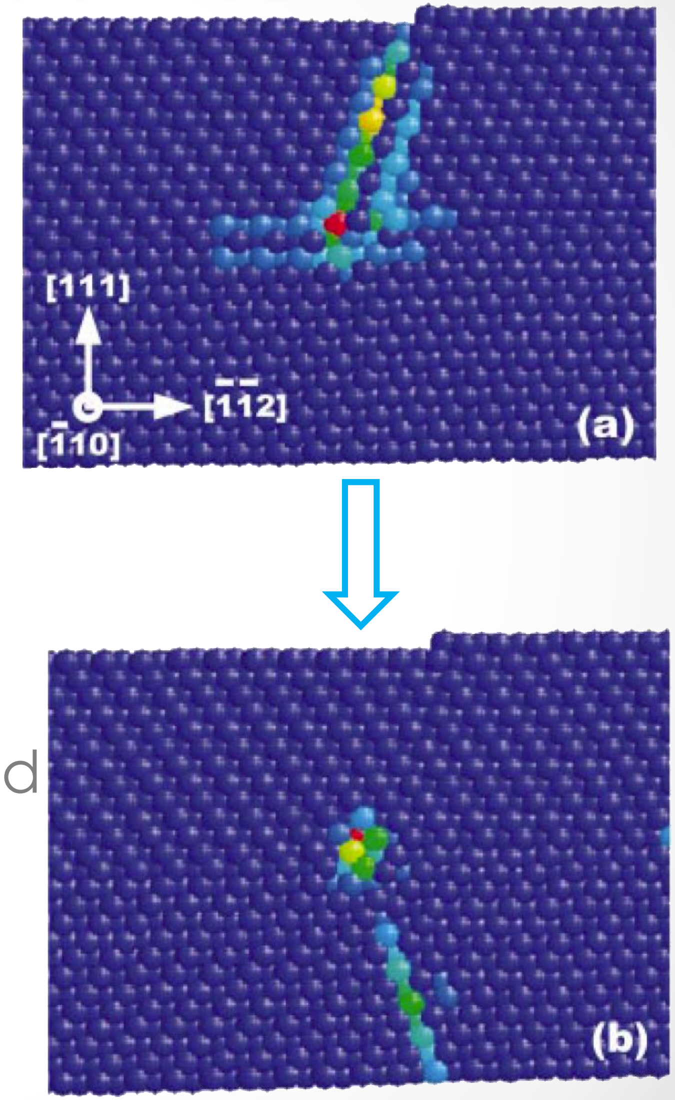
- Microstructures dominated by twins spaced <100 nm
- High mechanical strength
- Considerable plasticity

Structural origins of this mechanical behavior?

Twin vs. Grain Boundaries



Imperial College Rock Library.

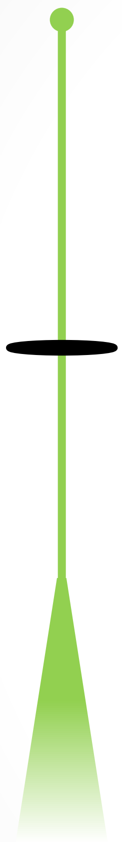


Zhang, X., et al, *Appl Phys Lett* **84, 2004.**

- 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

TEM and *In Situ* Experiments

Electron Beam Generation



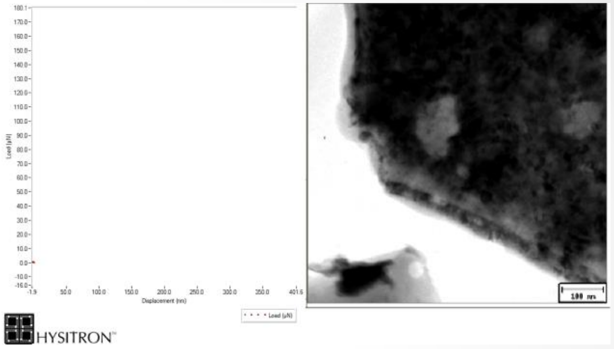
Sample Interaction

Magnification

Sub-nm imaging
Electron diffraction

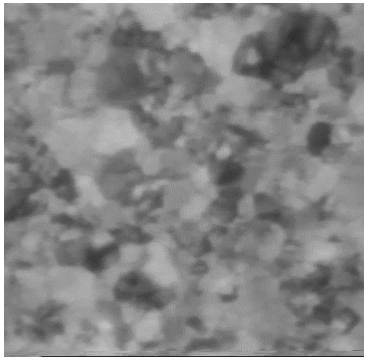
Mechanical

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



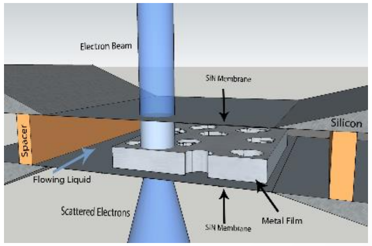
Thermal

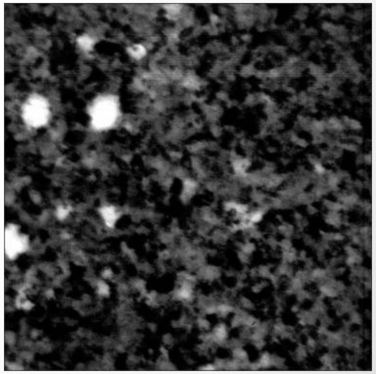
Effects of temperature on microstructural evolution up to 800 °C



Environmental

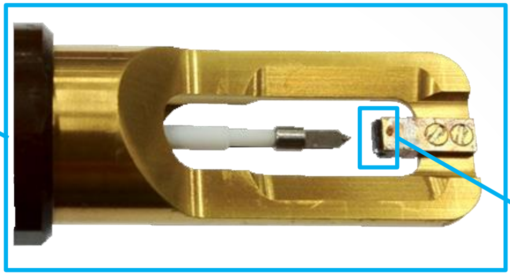
Effects of corrosion and gas loading at the grain level

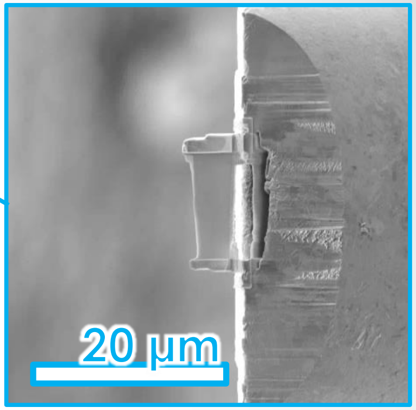




- **Enables real-time studies of samples under various stimuli**
- **Limited to electron-transparent, vacuum & electron beam-compatible samples**

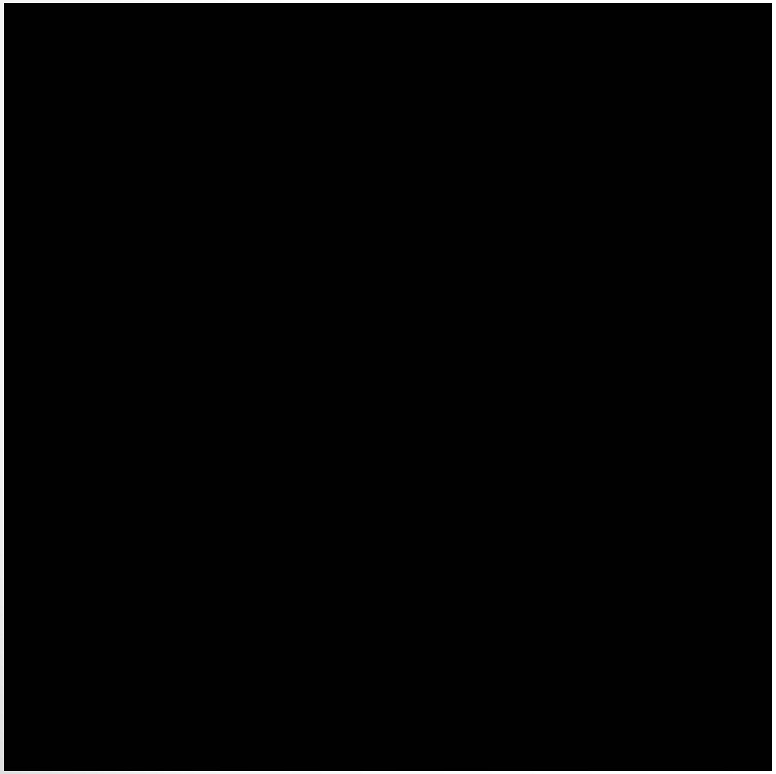
In Situ TEM Nanoindentation

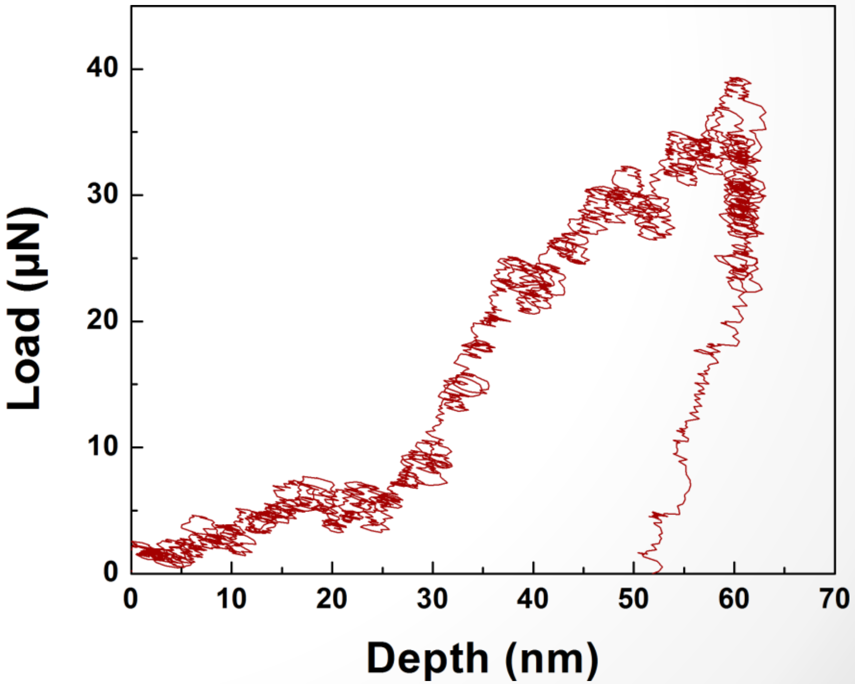




Hysitron PI95 *In Situ* Nanoindentation TEM Holder

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



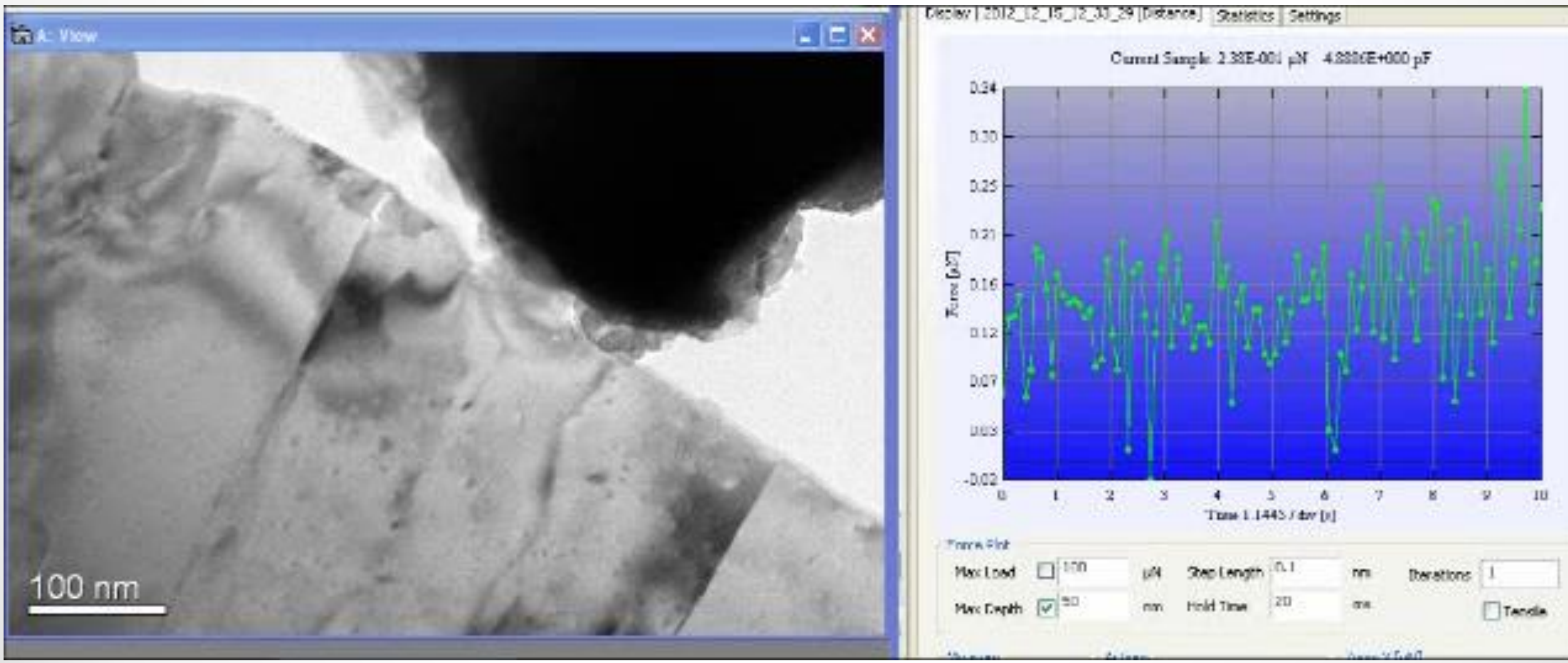


Quantitative deformation & observation at the nanoscale

Initial Plasticity in Al

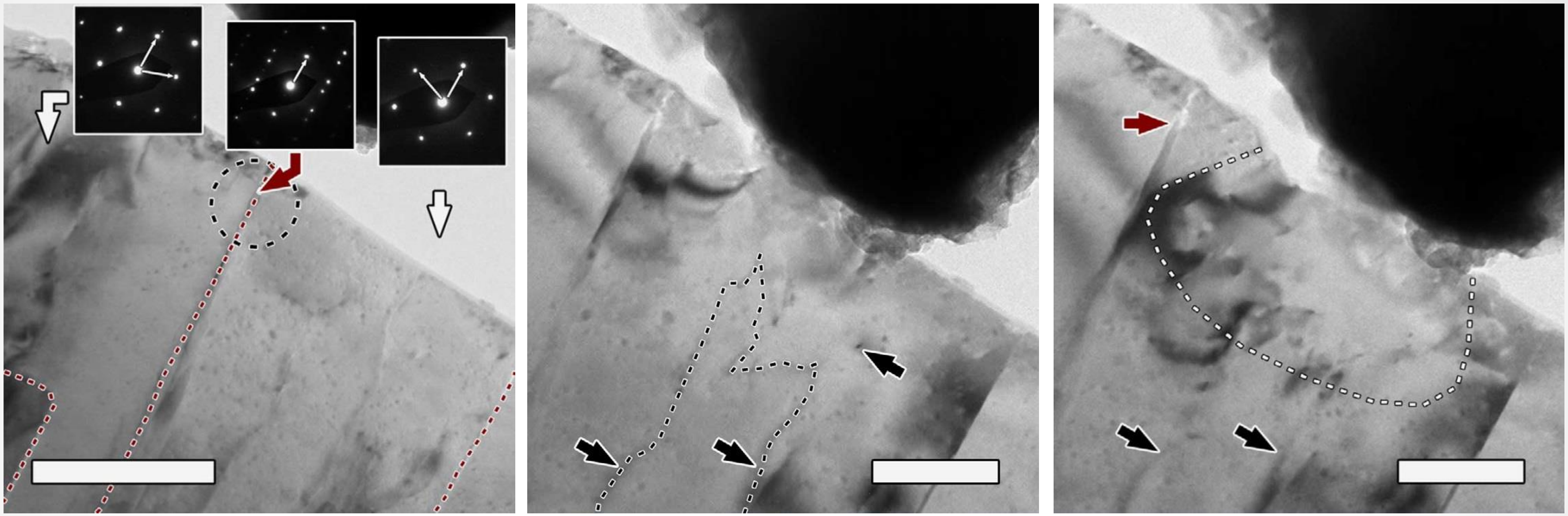
- *In situ* nanoindentation of Al near a Σ3{112} twin boundary
- Initial cycle

Video playback ×3



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

Initial Plasticity in Al



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

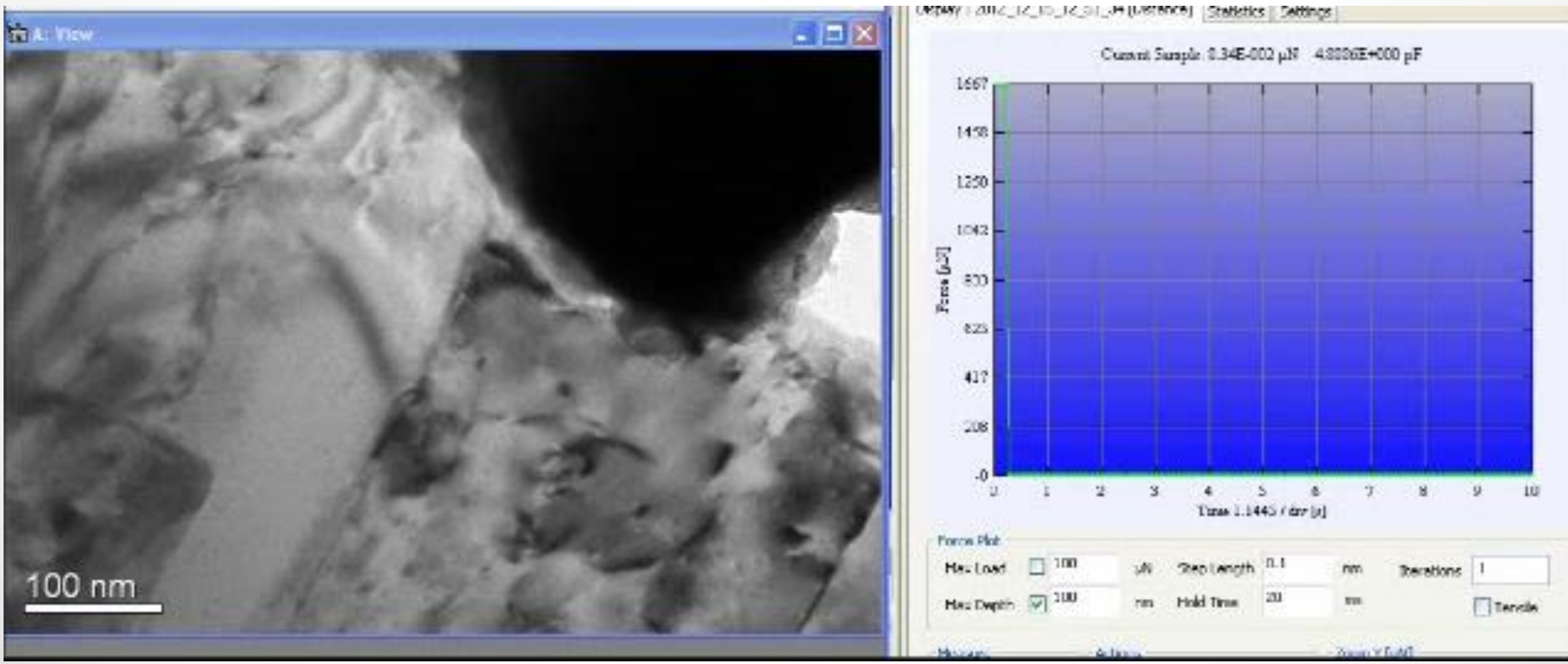
- Grains initially mostly free of large defects
- Movement of existing dislocation observed
- Deformation confined by twin to single grain

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

Dislocation Transmission

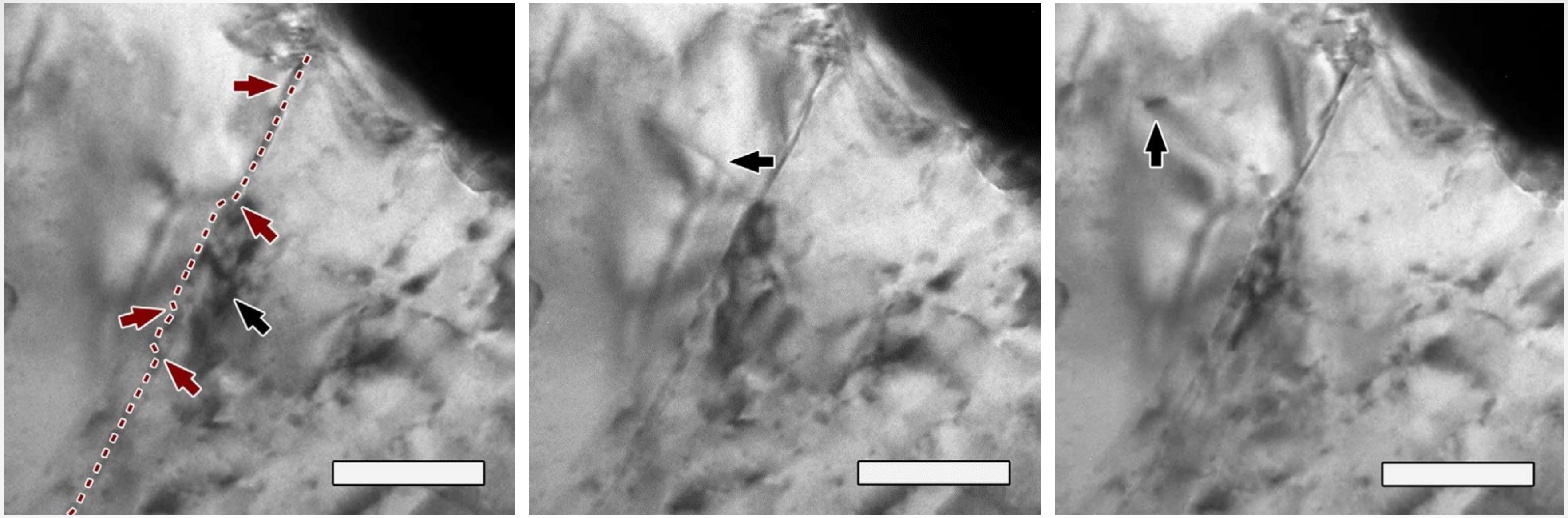
- In situ nanoindentation of Al near a Σ3{112} twin boundary
- Cycle #4, after 3 previous cycles to progressively higher loads

Video playback ×3



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

Dislocation Transmission



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

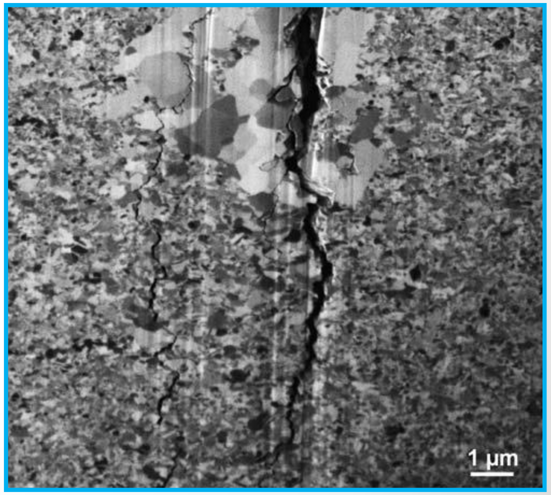
- Boundary deformed by dislocation interactions in previous cycles
  - But no obvious plastic deformation in adjacent grain
- First *observable* plasticity event in second grain captured
- Measured forces associated with the event

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

Cyclic Loading

- 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

Execcharter, 2011.



Padilla and Boyce, Exp Mech 2006.

- ***In situ*** **TEM deformation is a tool capable of investigating these questions.**

Tensile Testing

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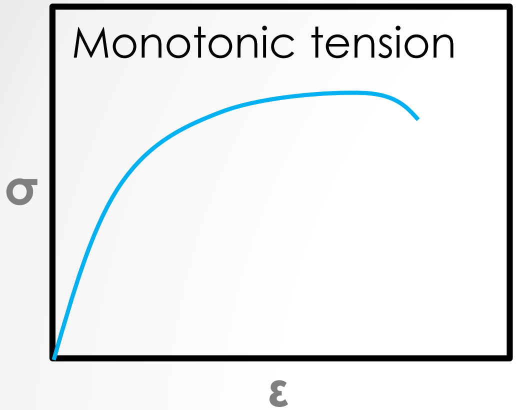
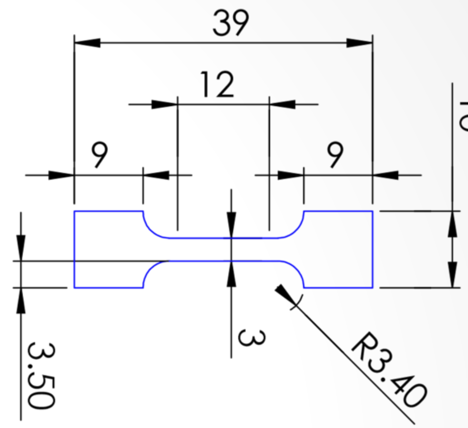
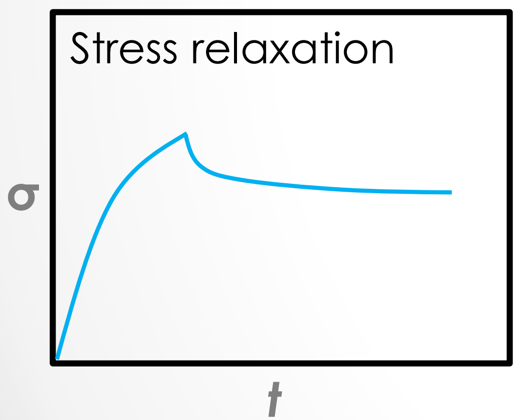
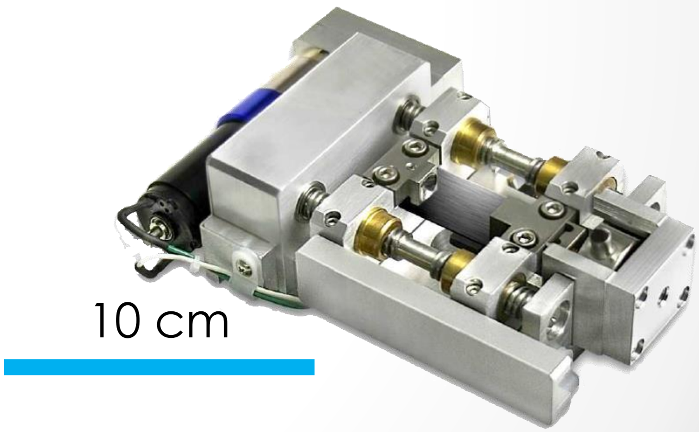


- Lots of information:
  - E, σ_y, σ_{UT}, elongation, toughness, n, m





- More information:
  - m, ΔV, creep

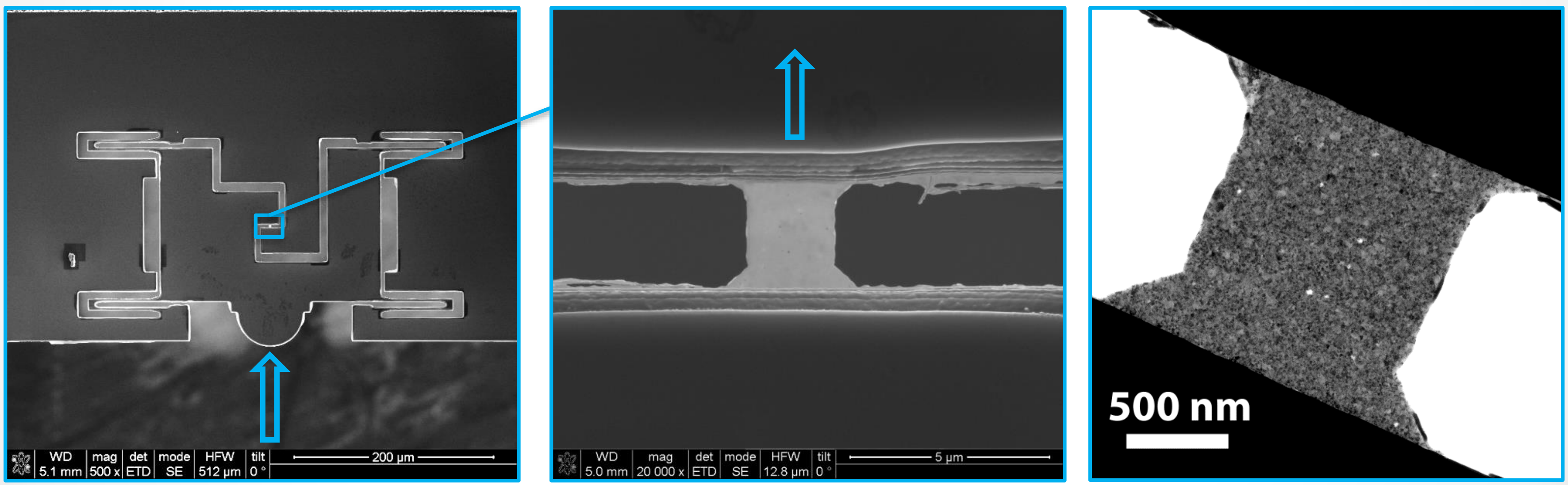


- **Gold standard for bulk mechanical properties**
- **Requires well formed specimens**

Tension Specimen Fabrication

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

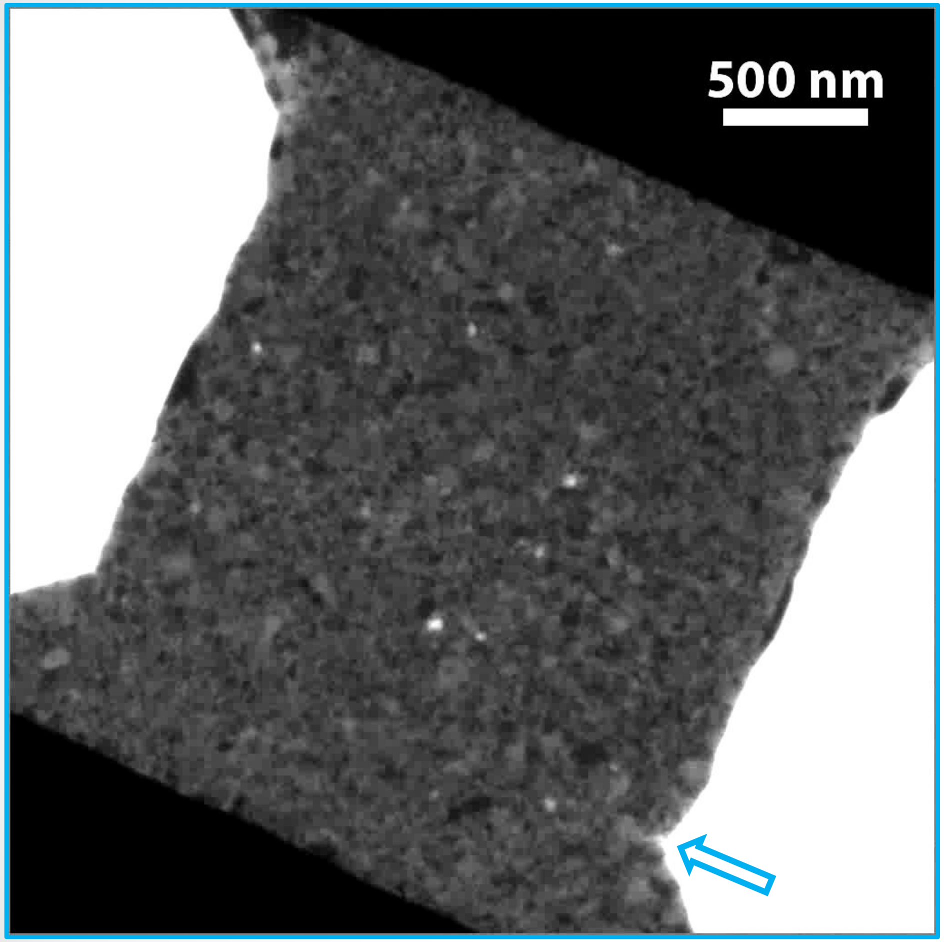
Collaborators: D. Adams, K. Hattar, W. Mook, C. Sobczak



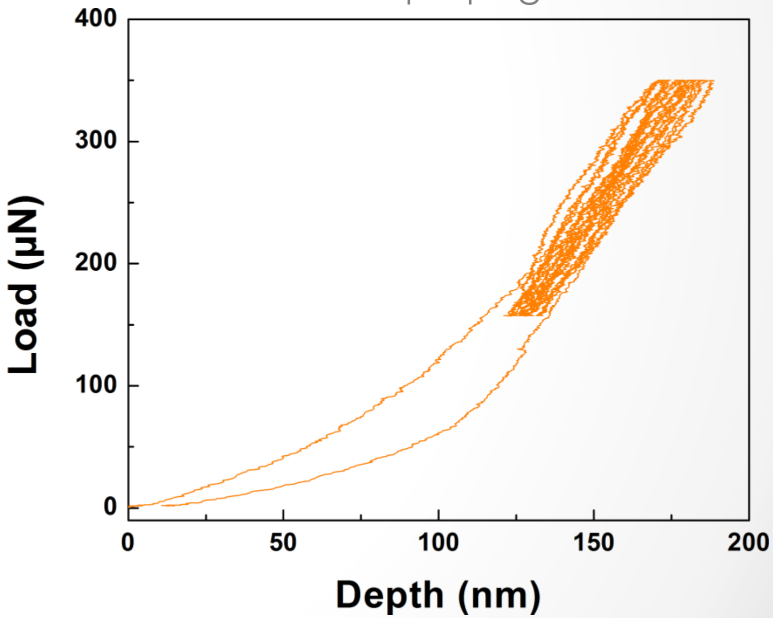
- Nearly pure tension, uniform cross sectional area, stable load frame
- Thin foil geometry not ideal for mechanics, but is electron-transparent

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



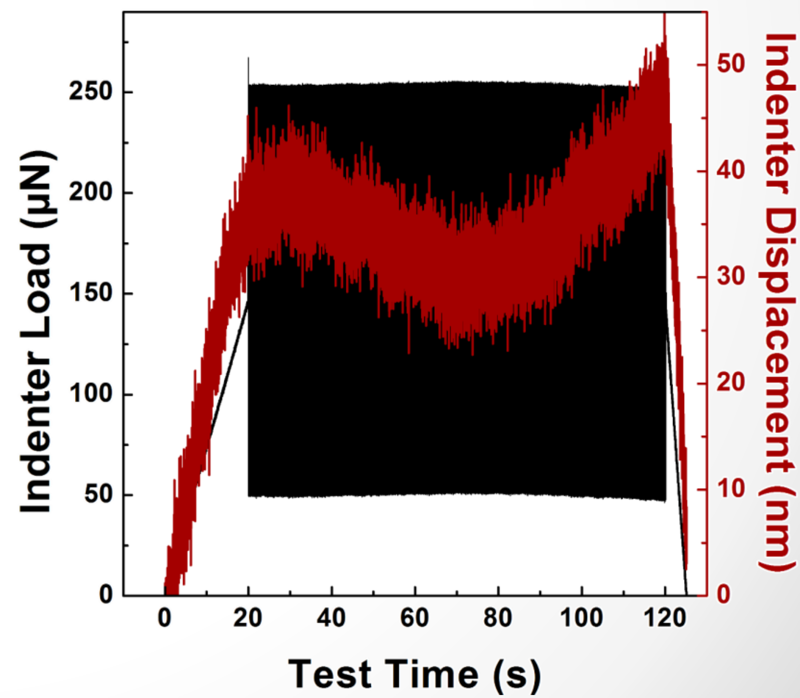
- **Direct measurements of fracture parameters**
- **Structural evolution at the crack tip**

High Cycle Fatigue

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

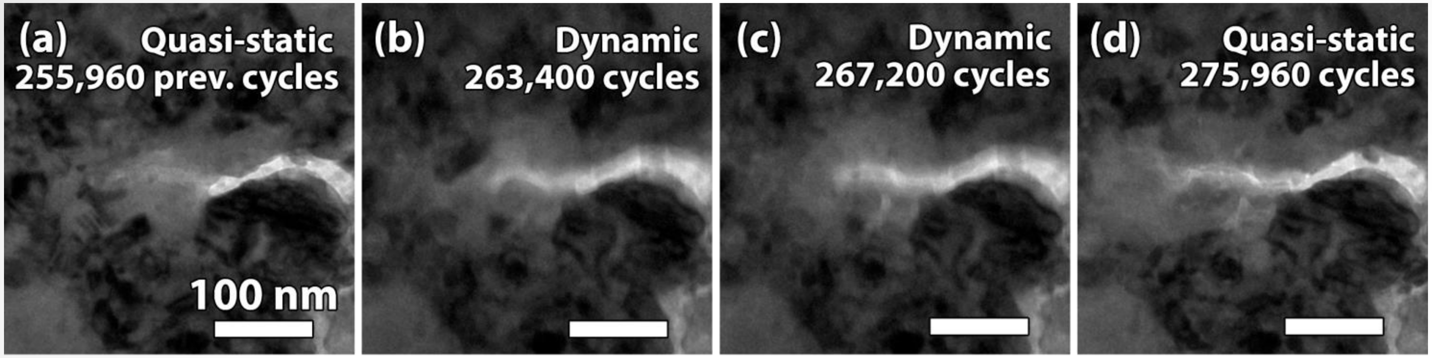
Video playback ×3

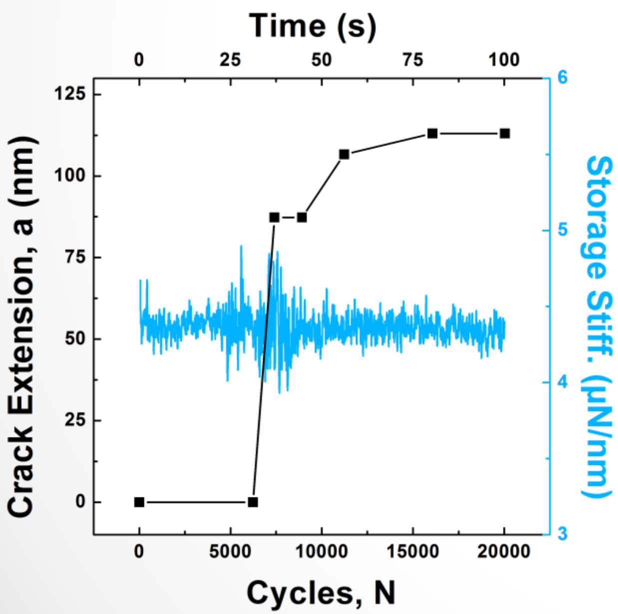
- Cyclic loading:
  - 200 hz
- Structural change at crack tip captured

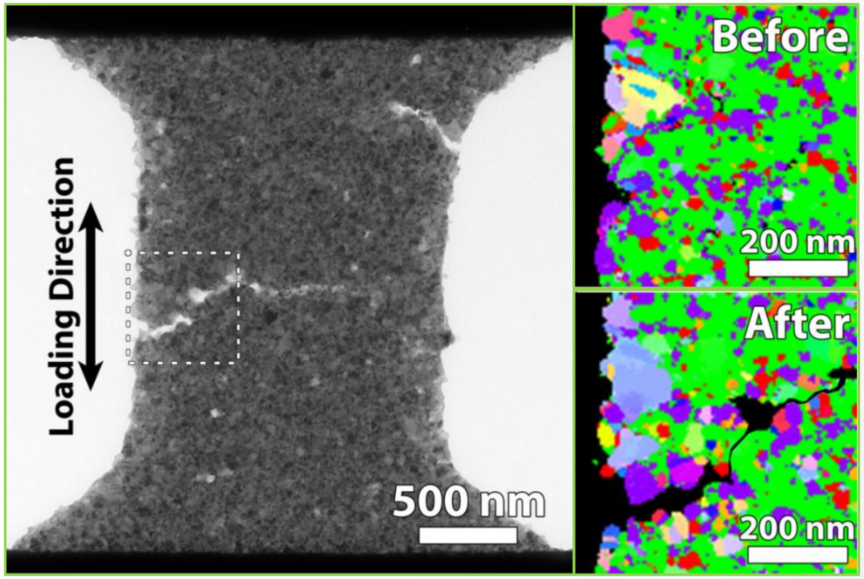


Crack Growth Quantified

Sandia National Laboratories



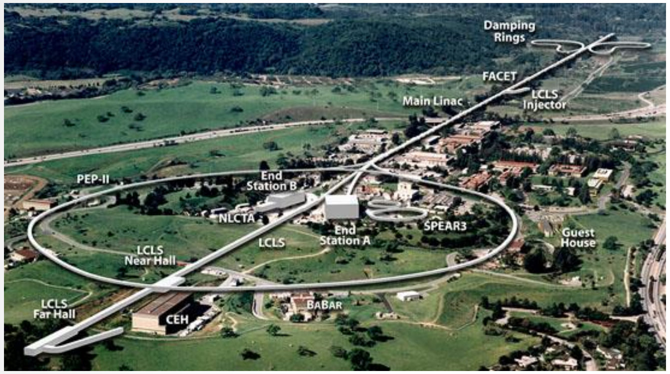


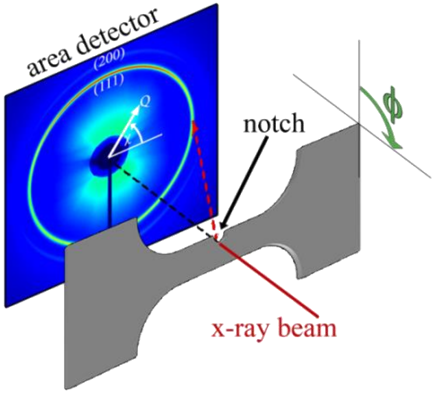


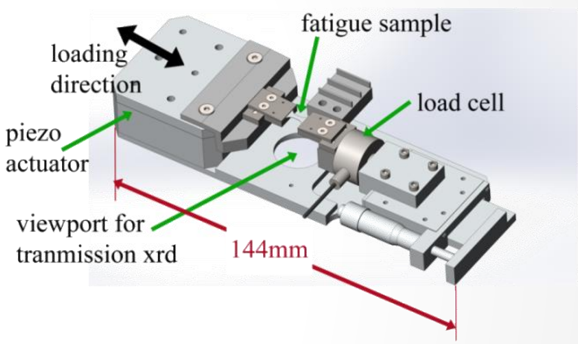
- Crack growth rate measured at 6×10^{-12} m/cycle!
- Evidence of fatigue-induced grain growth.

Another Approach…

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





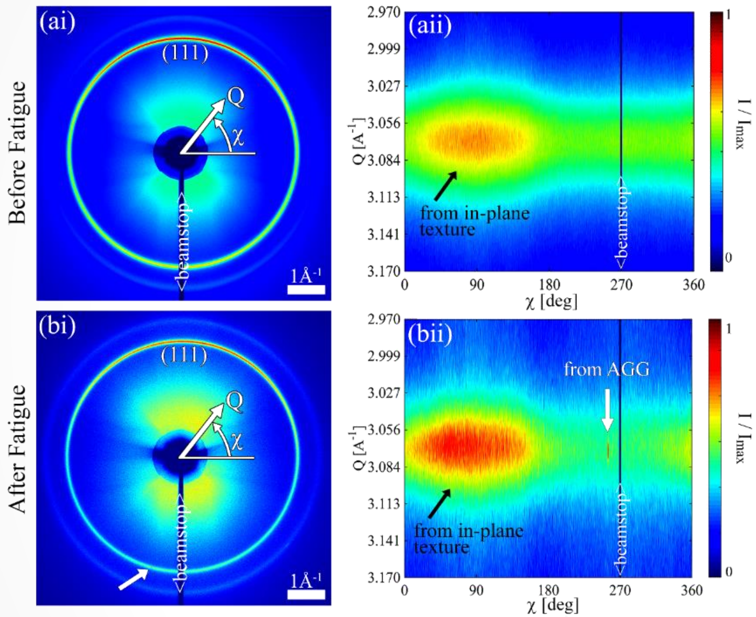


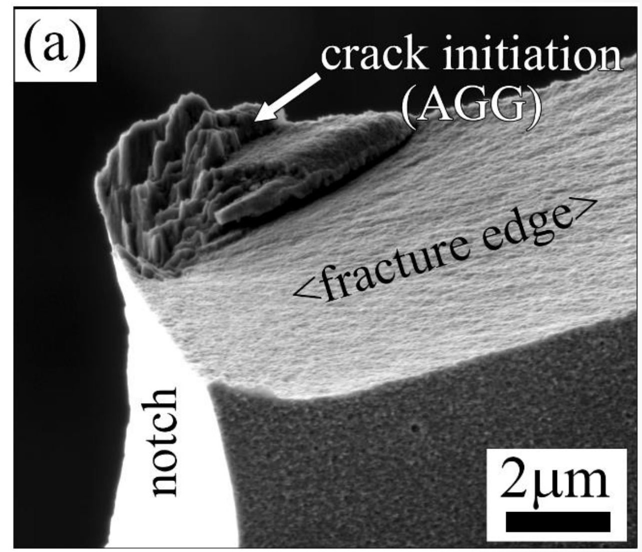
- *In situ* fatigue performed during X-ray diffraction analysis
- Experiments performed at the Stanford Synchrotron Radiation Lightsource (SSRL)

Which comes first, grain growth or crack initiation?

Another Approach…

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



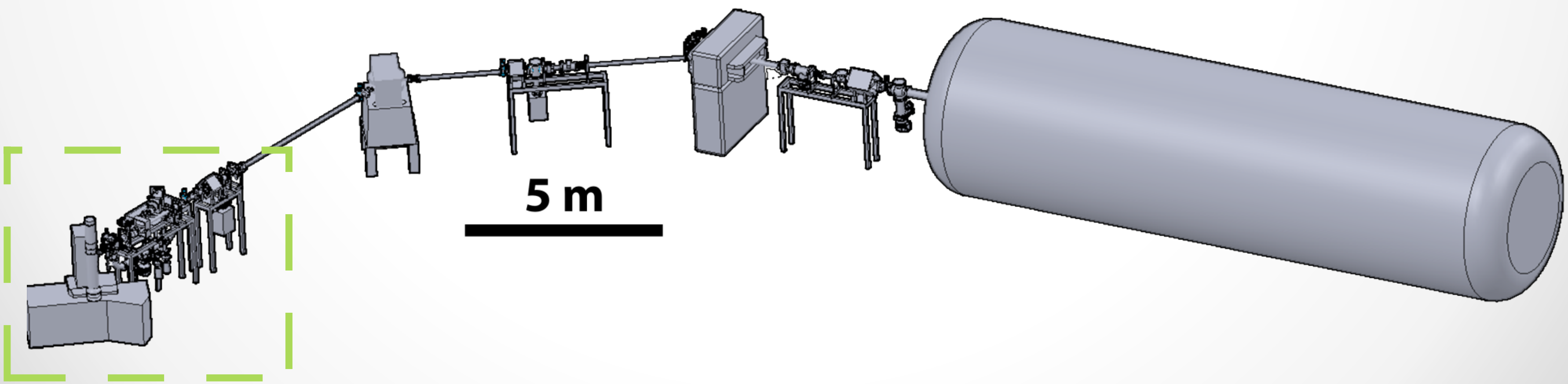


- Patterns processed and analyzed for outliers
- Fatigue-induced grain growth captured before failure
- Analysis of fracture surface suggests grain growth precedes crack initiation

Detection of abnormalities in only ~0.00001% of the sampled volume!

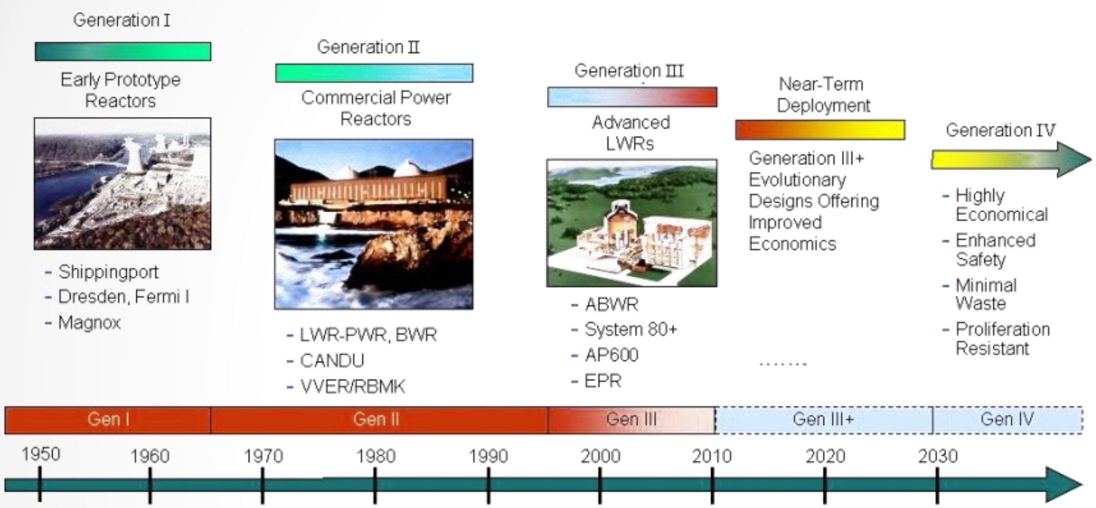
Roadmap

- Mechanical Testing
- *In Situ* TEM Mechanical Testing
  - Nanoindentation
  - Cyclic tension
- Irradiation
  - Grain growth



Radiation Effects

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

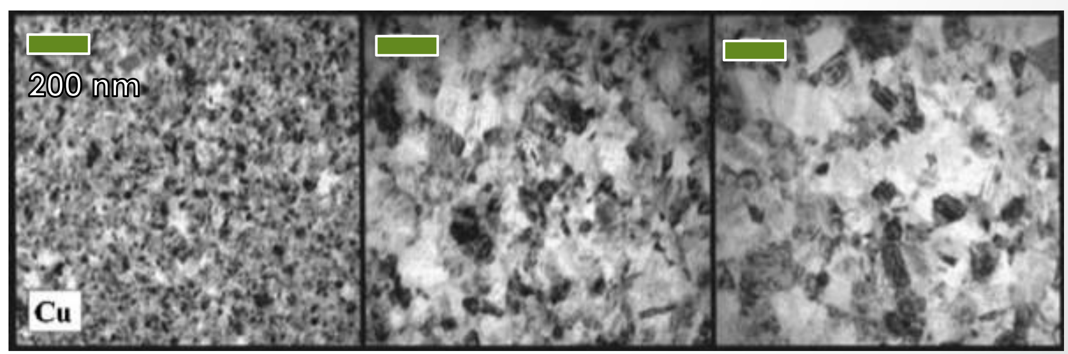


Argonne National Lab, *via* Wikimedia Commons.

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?



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

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

Radiation-Solid Interactions

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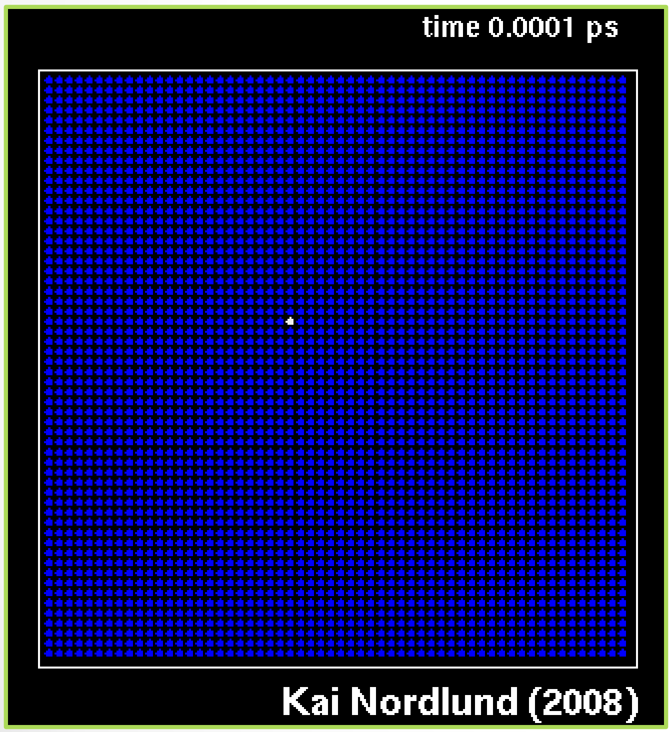
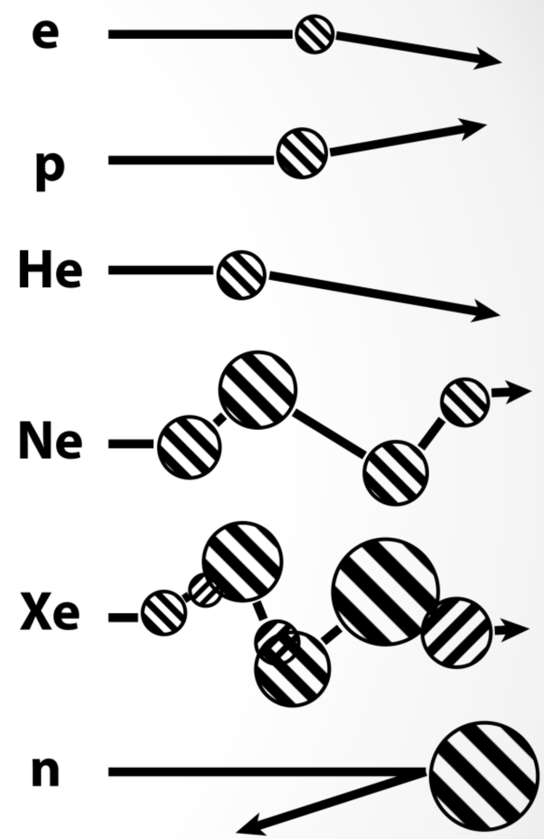
- Energetic ion displaces one or more target atoms
  - Frenkel (vacancy-interstitial) pair
  - Collision cascade
  - Nuclear and electronic interactions



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

Effective transient temperatures ~<u>thousand(s)</u> of K!

Affected volumes vary based on radiation species, energy, and target material.



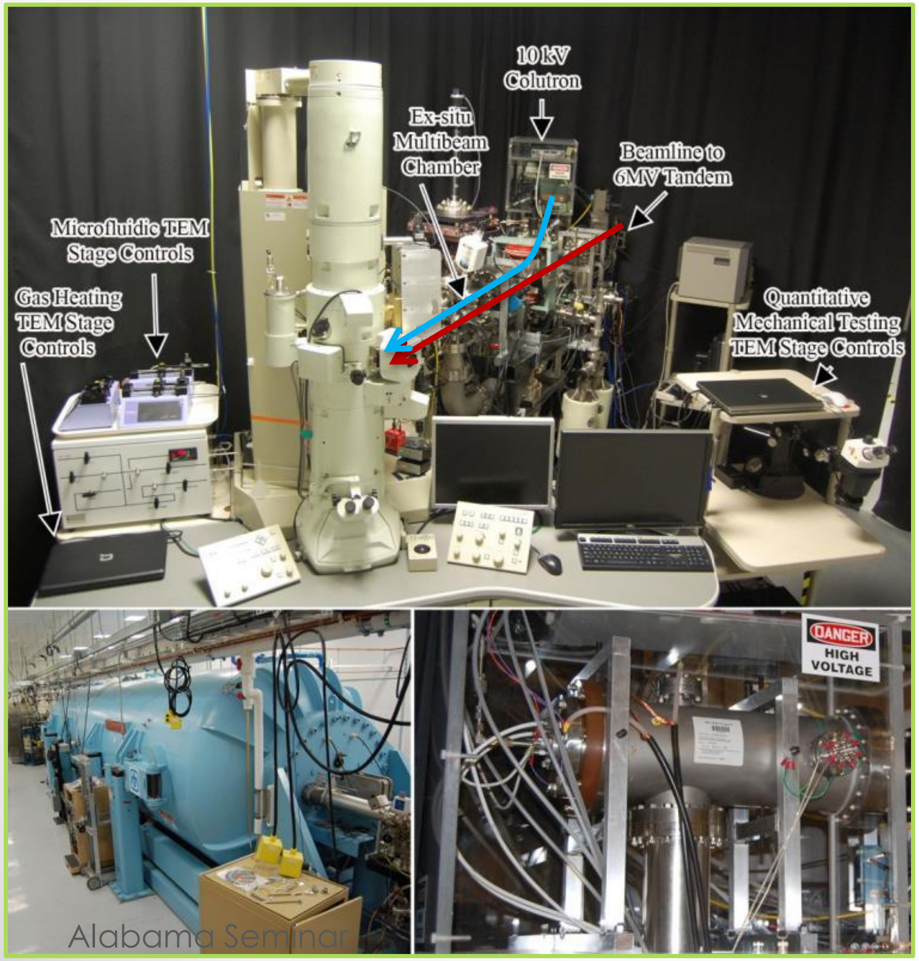
Schematic recoil spectra for 1 MeV particles in Cu. Sizes represent recoil energies. After Averback, J Nucl Mater, 1994.

Highly temporally and spatially localized energy transfer drives microstructural change.

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

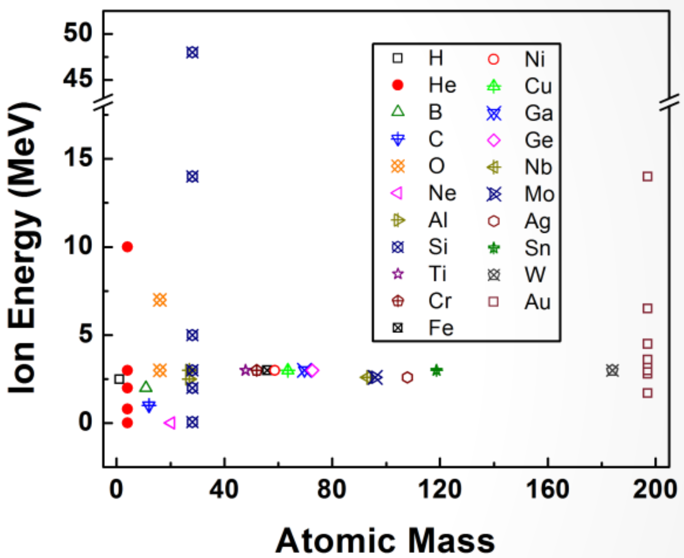
Collaborators: D. Buller, K. Hattar, J. Scott

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

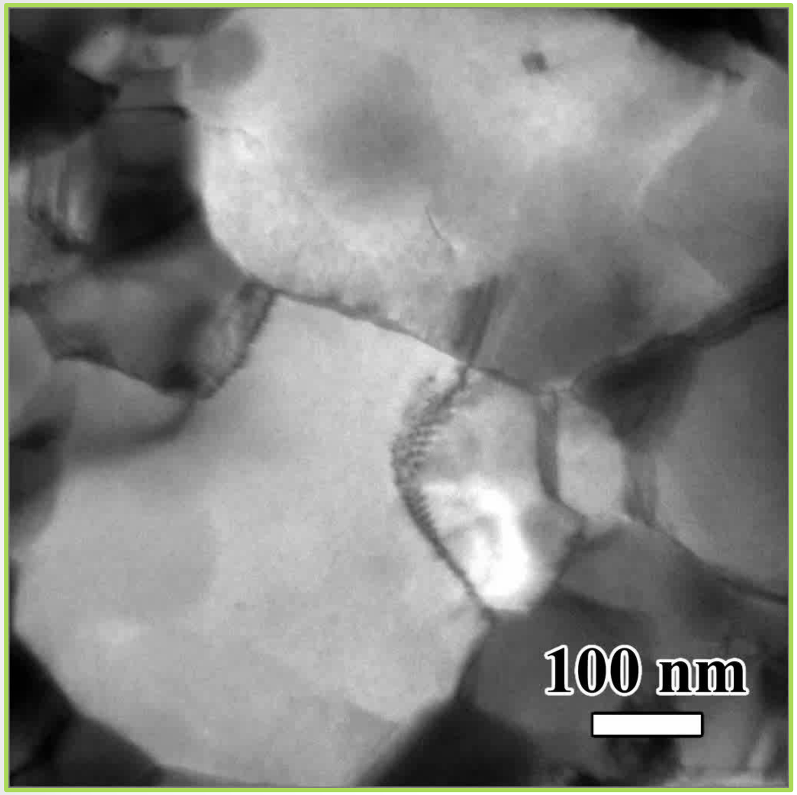


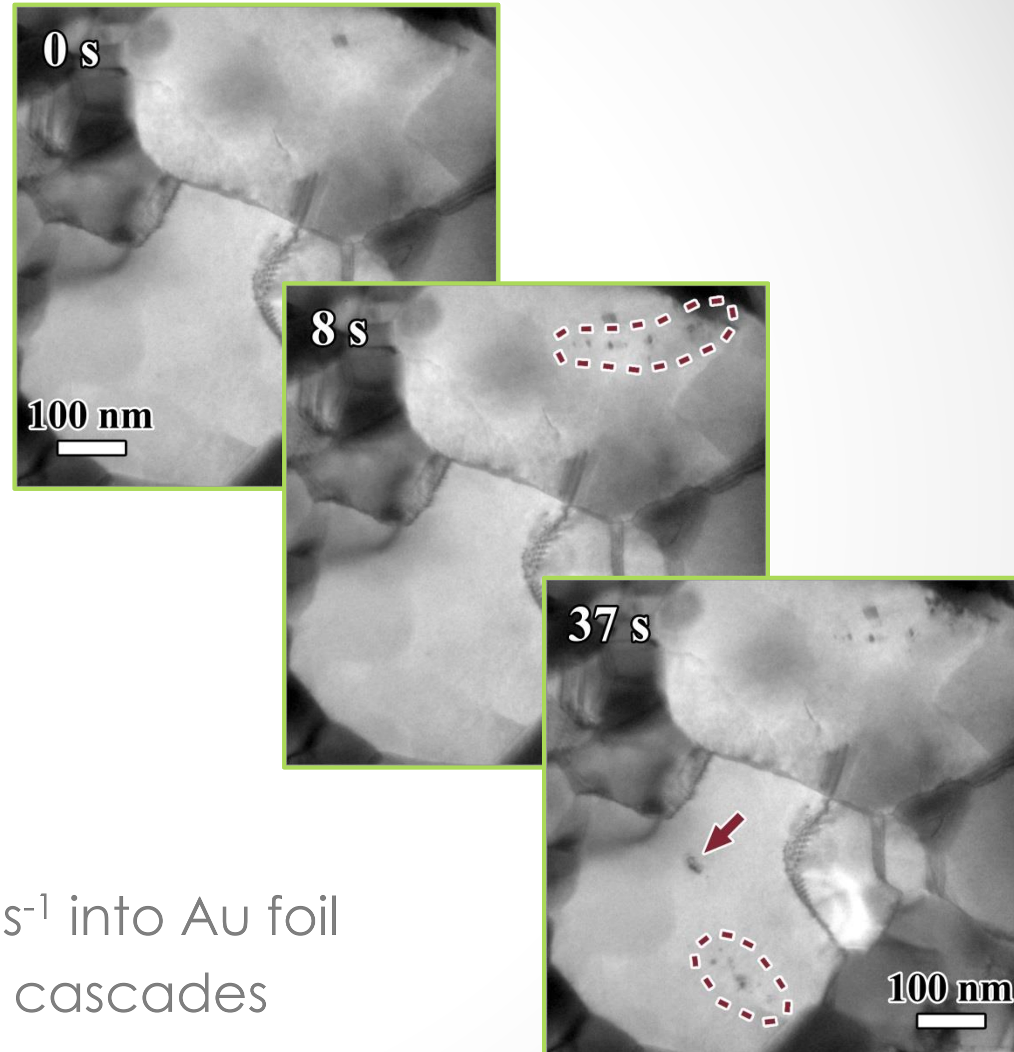
Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution.

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

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

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+}
- ~22 s of 4000s total experiment time

In situ ion irradiation
TEM: 10 MeV Si into nanocrystalline Au.

Playback at 2 × real time.

2× real time

Locations of single ion strikes and resulting microstructural change captured!

Quantification: Overall

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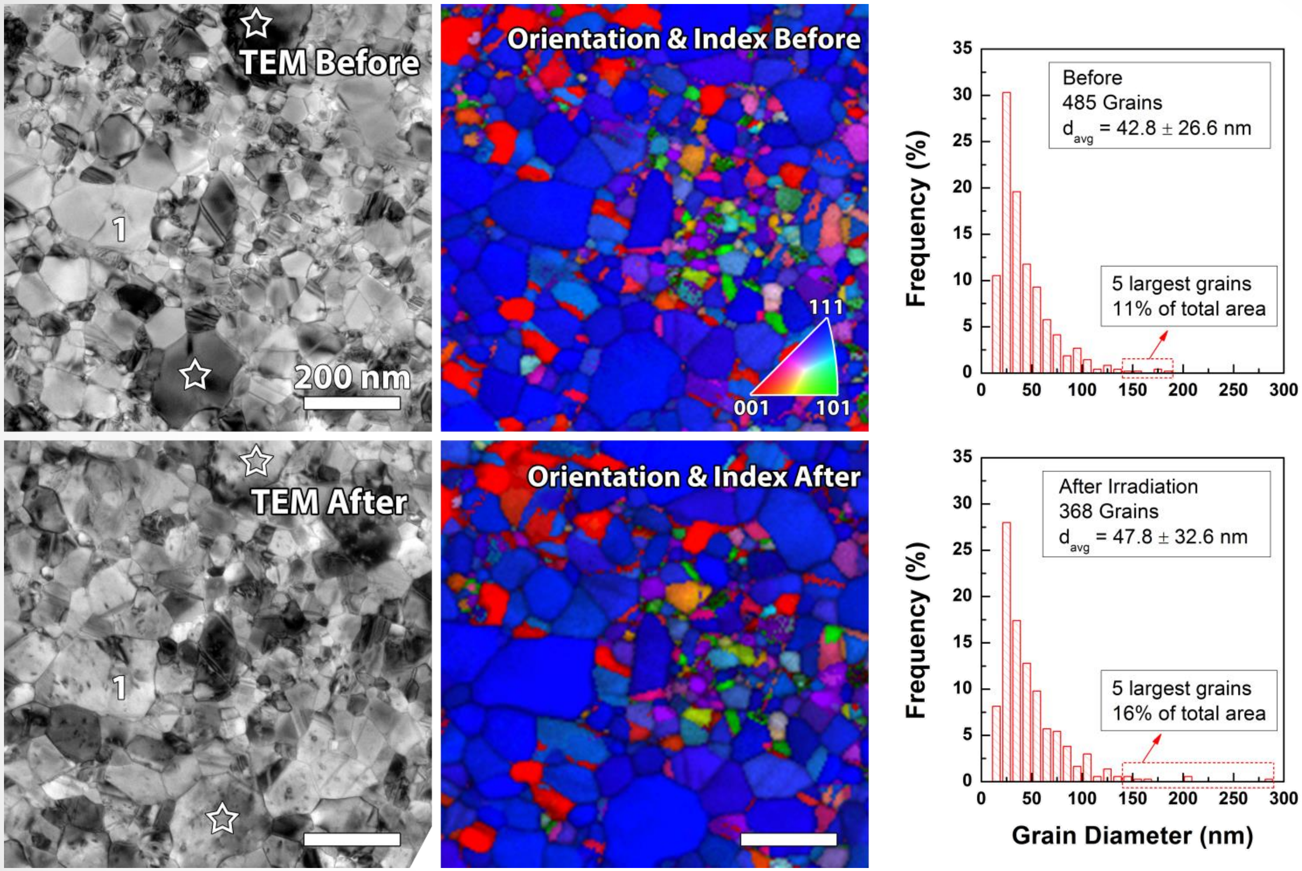
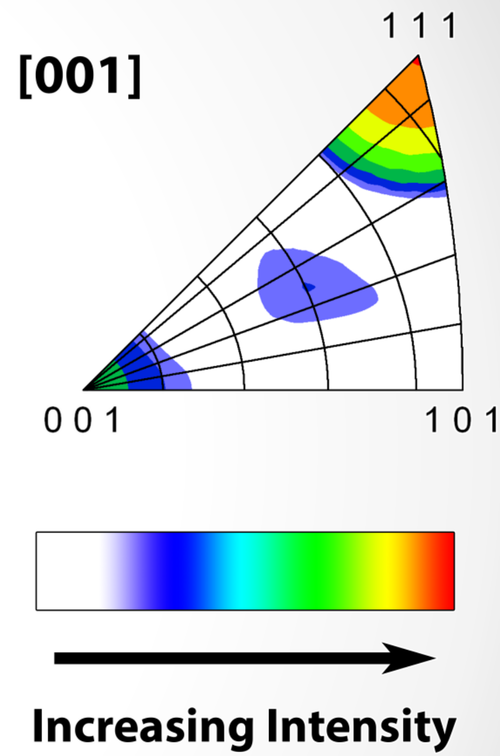




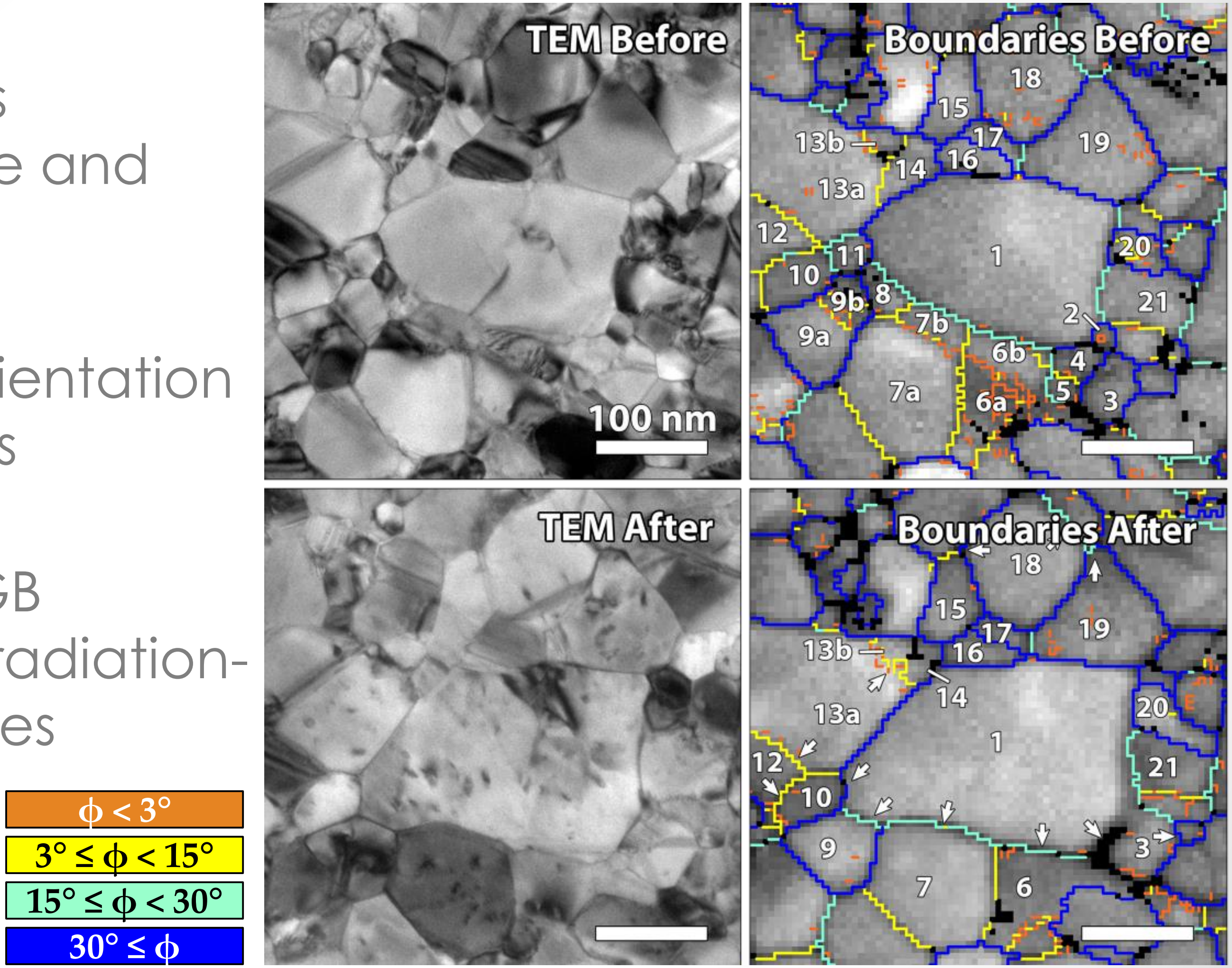
Bufford, *et al*, J Appl Phys, 2015.

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

Rapid quantification of statistically relevant numbers of grains and boundaries.

Individual Boundaries

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

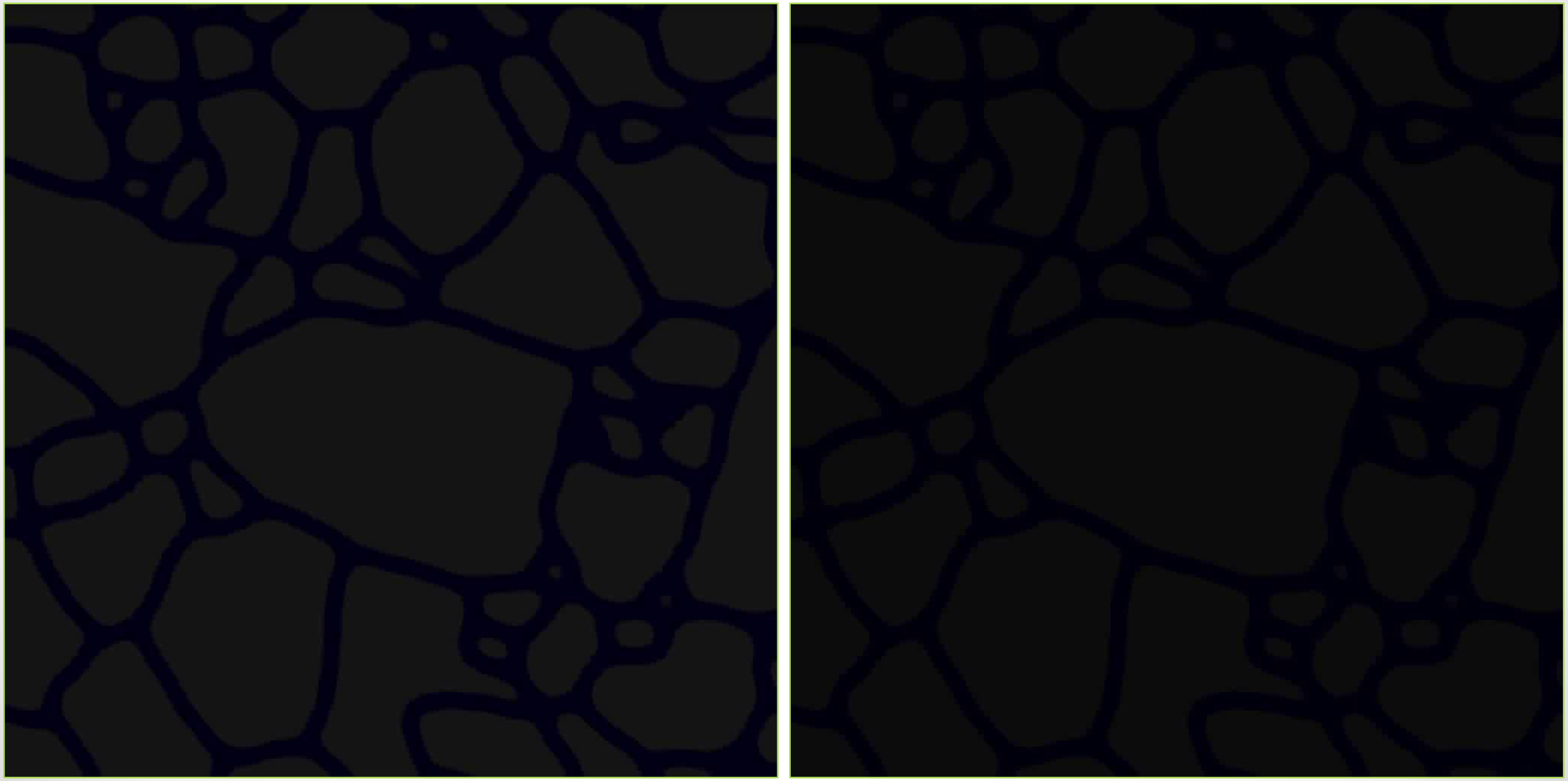


Bufford, *et al*, J Appl Phys, 2015.

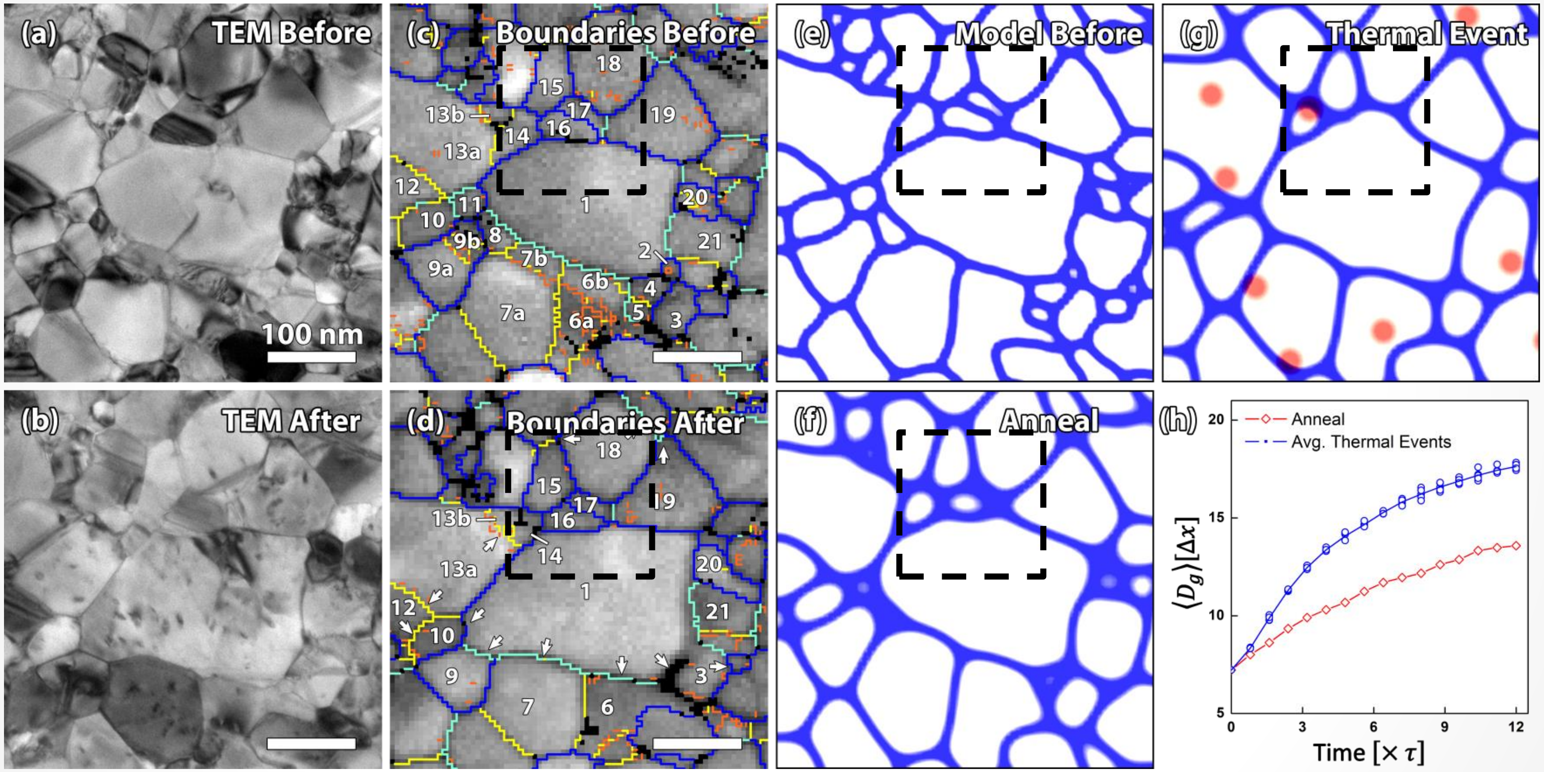
Grain boundary misorientation angle and axes quantified

Simulated Irradiation and Annealing

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



Experiment/Model Discrepancies?



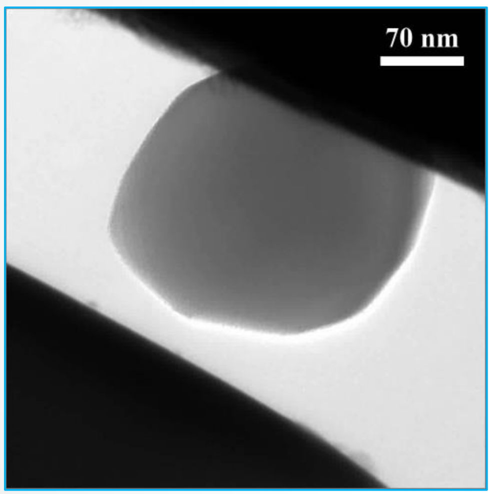
Bufford, *et al*, Appl Phys Lett, 2015.

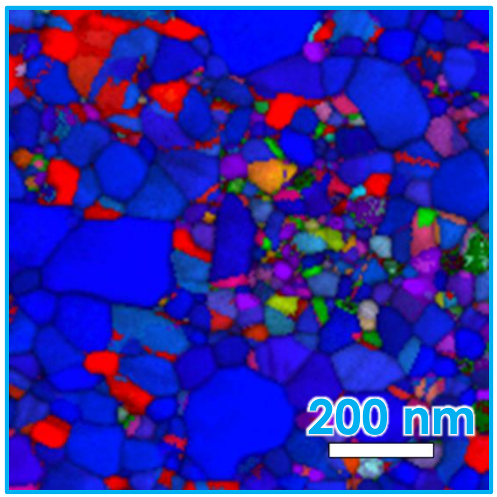
- Subtle deviations from homogenous grain growth
- Overall scaling laws appear consistent

Immobile boundaries suggest importance of non-thermally activated mobility

Summary and Conclusions

- 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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Synergistic In Situ Capabilities

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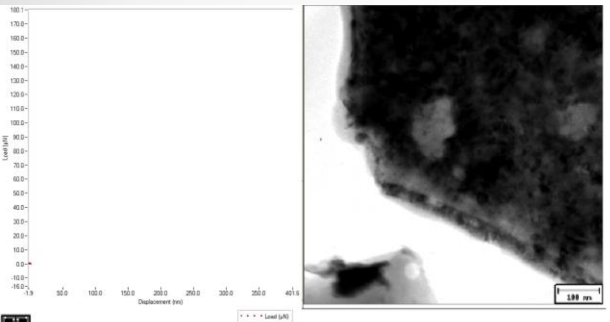
Mechanical

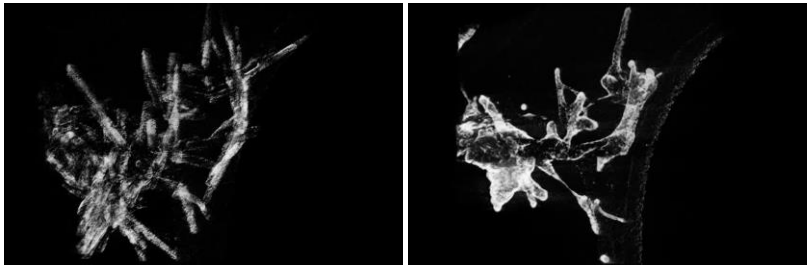


HYSITRON

Hysitron PI95 TEM Picoindenter
Gatan 654 Straining Holder
Direct correlation of dose and defect density with resulting changes in strength, ductility, and defect mobility

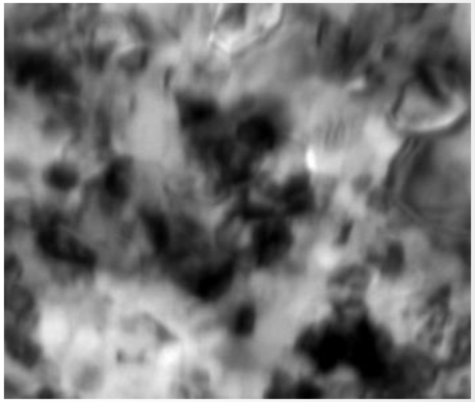
Structural

Hummingbird Tomography Stage
Gatan 925 Double Tilt Rotate
Morphology changes as a result of radiation damage



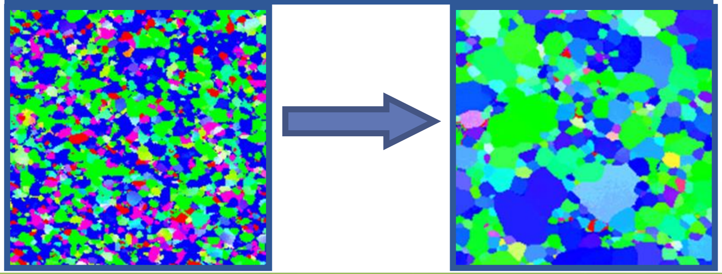
Thermal

Hummingbird Heating Stage
Coupling effects of temperature and irradiation on microstructural evolution up to 800 °C



Texture

Nanomegas ASTAR
Quantifying orientation changes as a result of radiation, implantation, and heat.



Environmental

Protochips Liquid and Gas Flow
Effects of radiation on corrosion and gas loading at the grain level

The application of advanced microscopy techniques to characterize synergistic effects in a variety of extreme environments

Precession Electron Diffraction Microscopy

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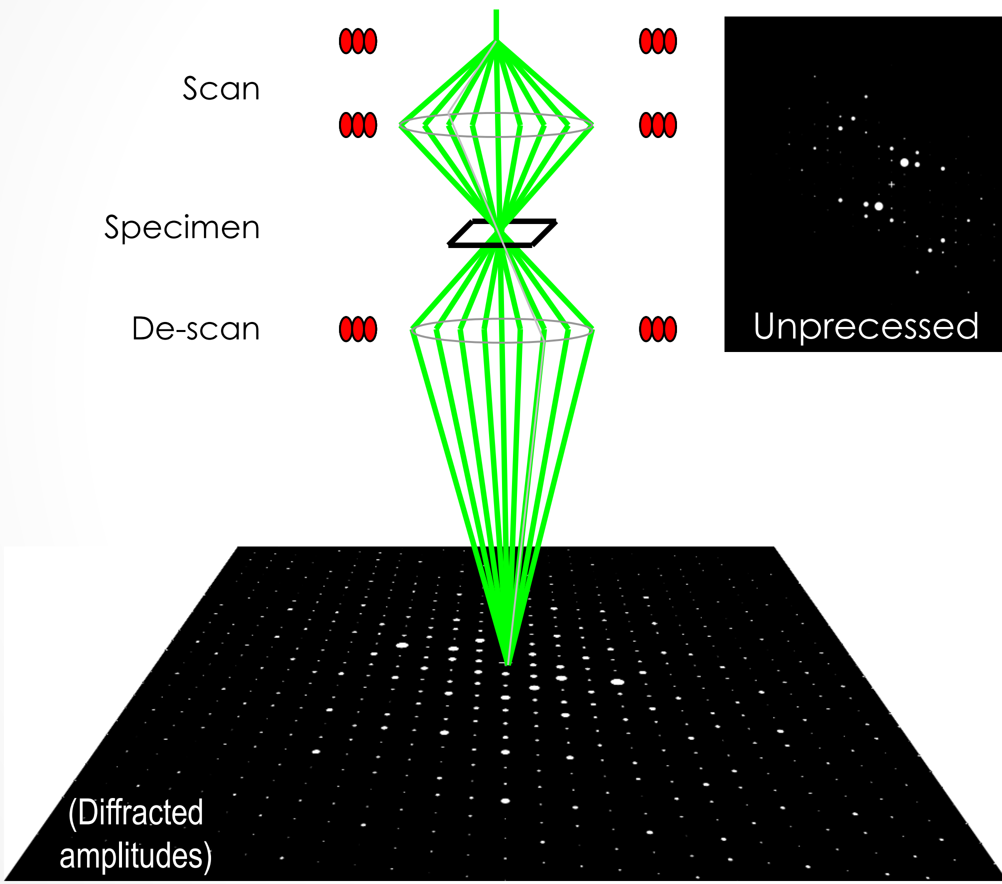
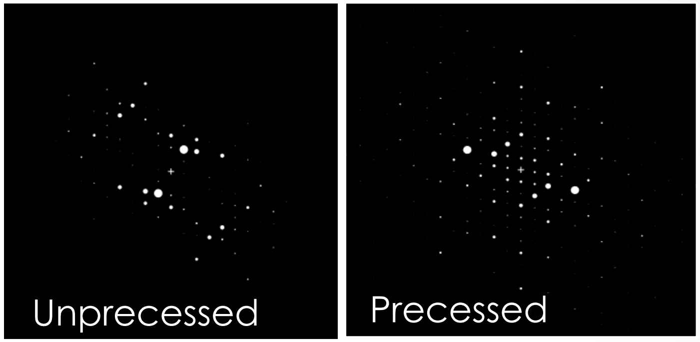
Collaborators: K.J. Ganesh, S. Rajasekhara, P.J. Ferreira





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

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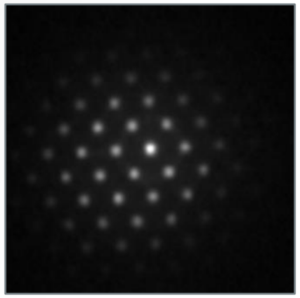
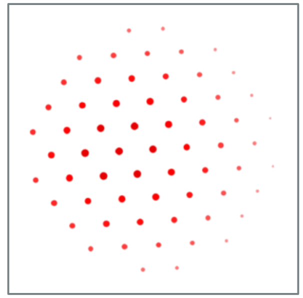
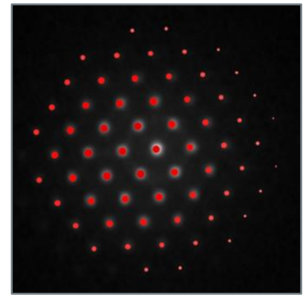


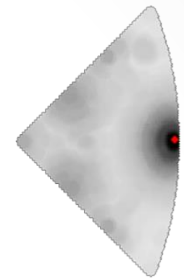
Experimental Pattern



Theoretical Template

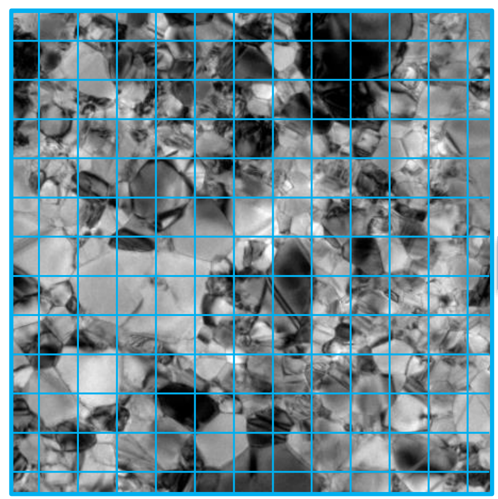


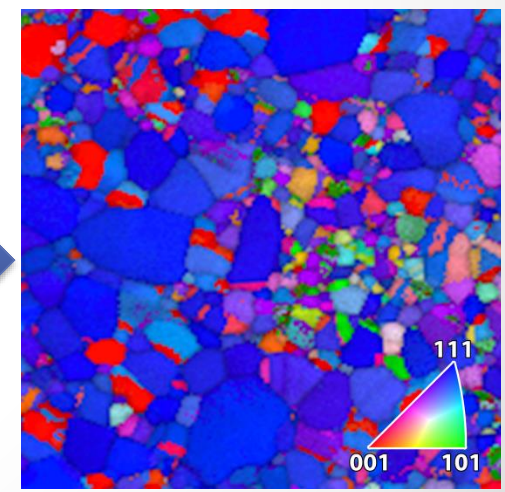
Template Matched



Point Mapped To IPF

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

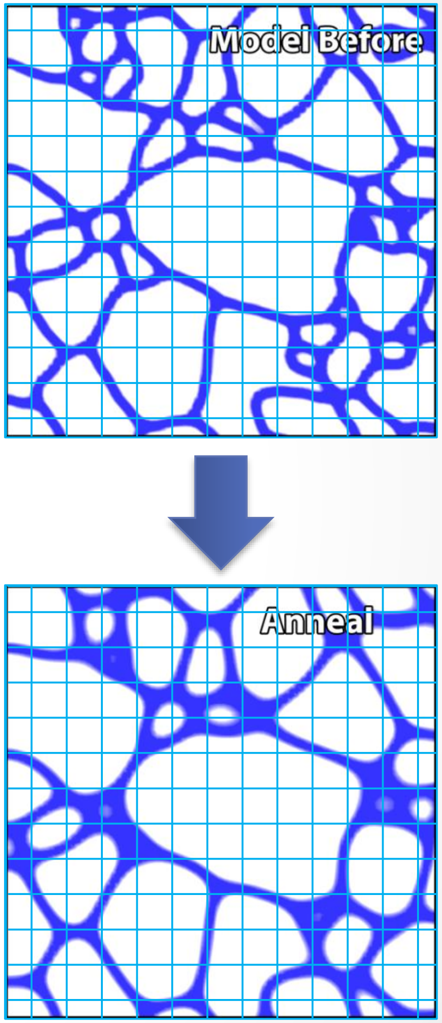




Point diffraction data

Approach: Modeling

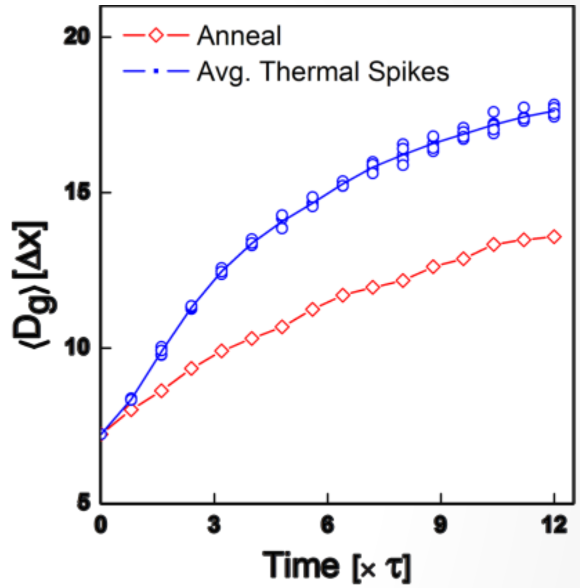
- What is phase field modeling?
  - Mathematical model for solving interfacial problems, like solidification, growth, etc.
- Example grain growth model
  - Thermodynamic free energy function
    - $dF = d(\gamma A) = \gamma dA$ (γ: GB energy, A: GB area)
  - Model for kinetics
    - $V = M\gamma h$ (M: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

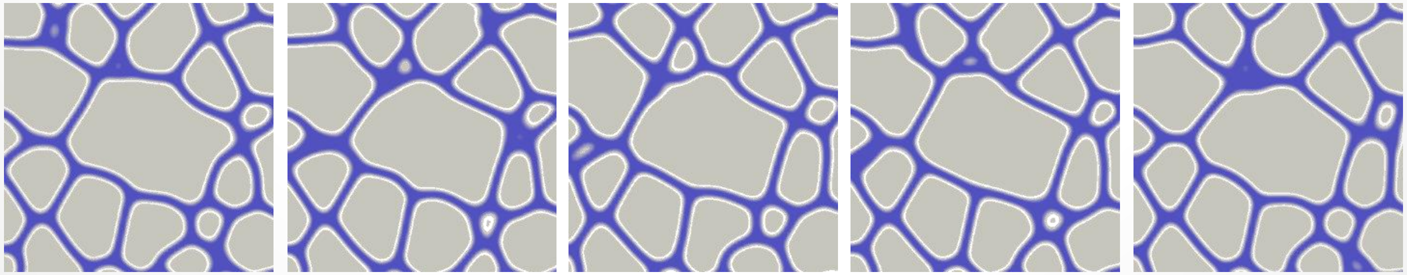


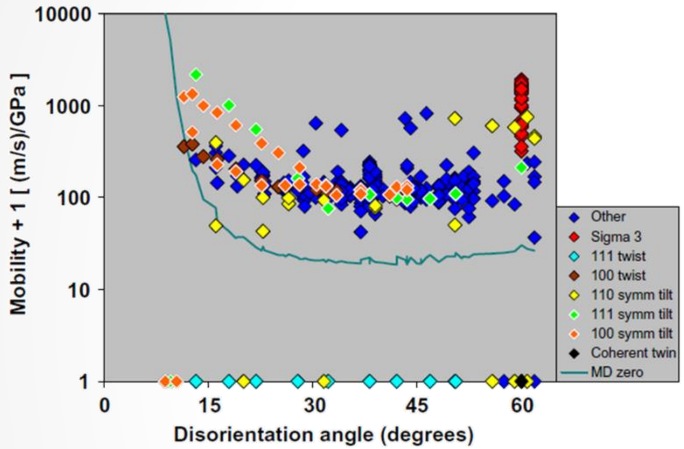
Can directly use experimental maps as input structures, and then compare evolutions!

Model Data Analysis

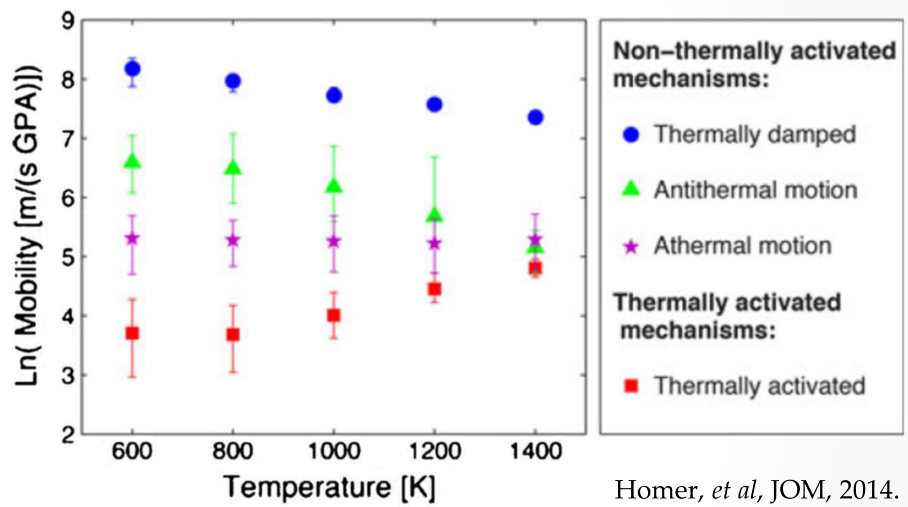
- During simulated annealing grain growth scales approximately with $T^{1/2}$
  - Expected for homogenous grain growth
- During simulated irradiation, grain growth scales with $T^{1/n}$, where $n \approx 3$
  - Initially faster, but stagnates sooner







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



Homer, *et al*, JOM, 2014.

Time scaling

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

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

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

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

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

$$= K\Phi t.$$

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

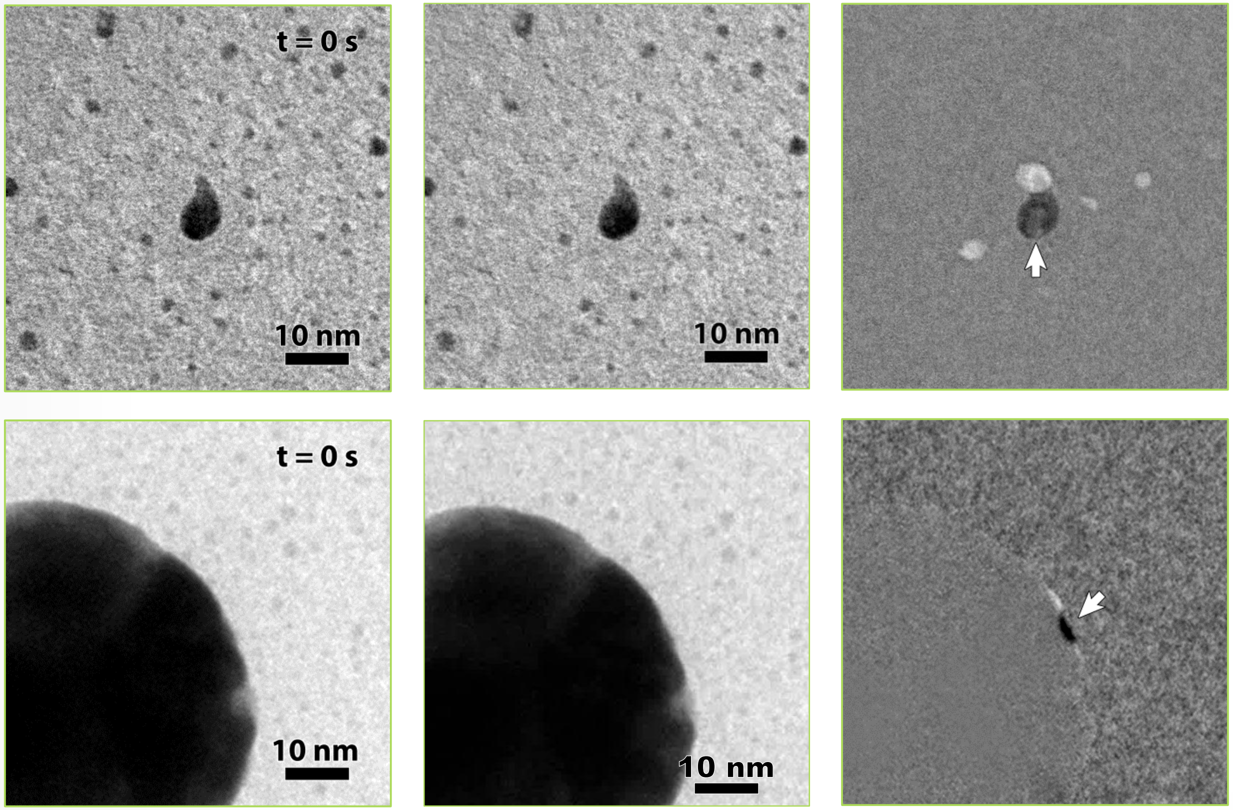
Kaoumi, *et al*, J Appl Phys, 2008.

Single Ions in Nanoparticles

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

5 nm

60 nm



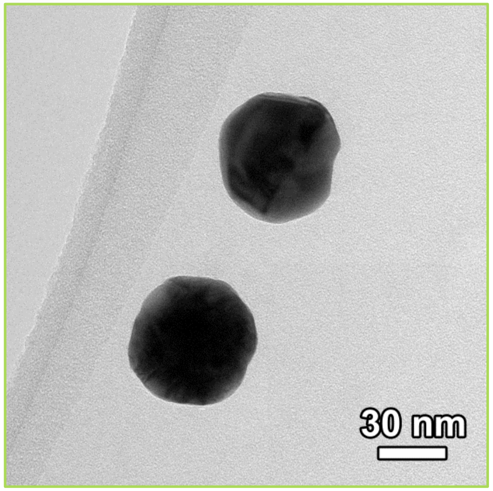
Difference Images

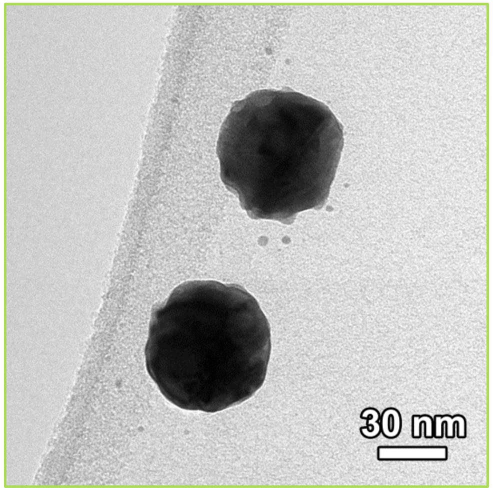
- **Dark**: Only present before
- Light: Only present after
- Gray: unchanged

- Single 46 keV Au^{-} ions into Au nanoparticles

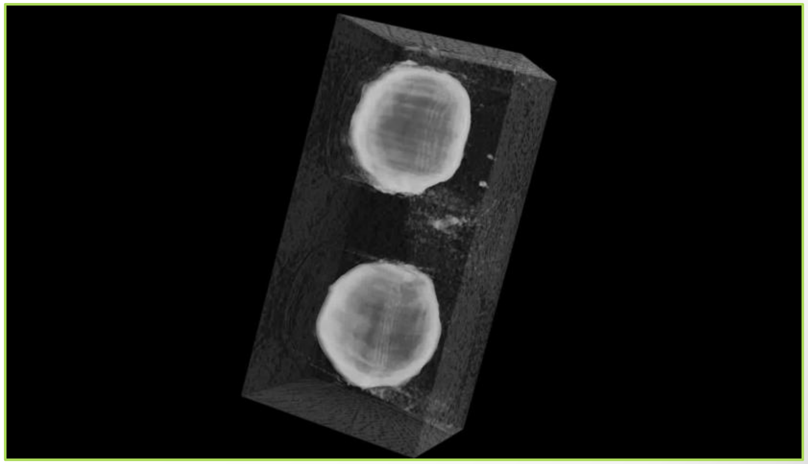
Effects of similarly sized cascades vary dramatically with particle size.

Surface Effects of Heavy Ions



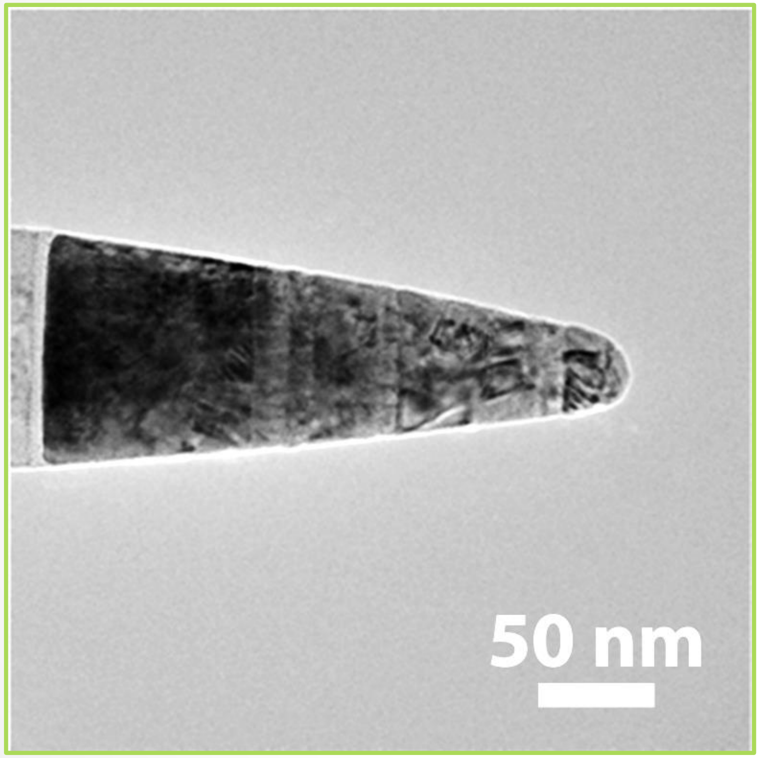


- 60 nm Au NPs before/after 2.0 × 10^{14} ions/cm^2 of 2.8 MeV Au^{4+}



Nanopillar Fabrication

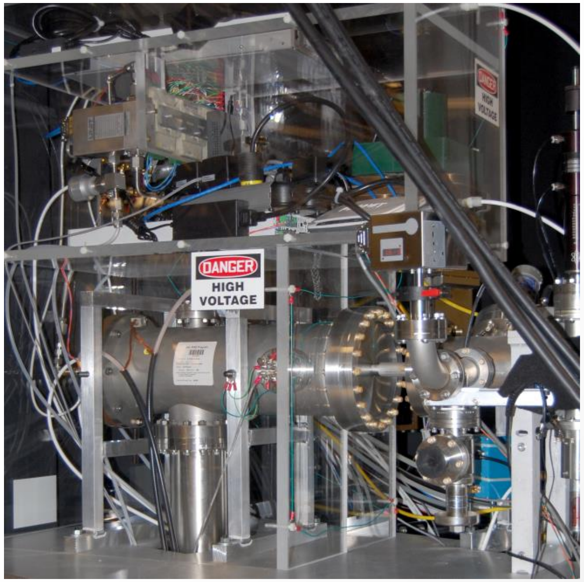
- FIB-milled from 500 nm PLD Ni on Si
  - ~100 nm base diameter
  - Conical geometry



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

Accelerators

- HVE EN Tandem
    - 0.8 – 6 MV
    - SNICS, Alphatross, Hiconex 834 sputter, and duoplasmatron sources
        - H, He, most elements except other noble gases
        1. ~3 MeV Au
    - Displacement damage
- Colutron G-1
    - 0.5 – 10 kV
    - Hot filament source
        - Gases
        2. 10 keV He
        3. 10 keV D_2
    - Implantation



Ions in the TEM

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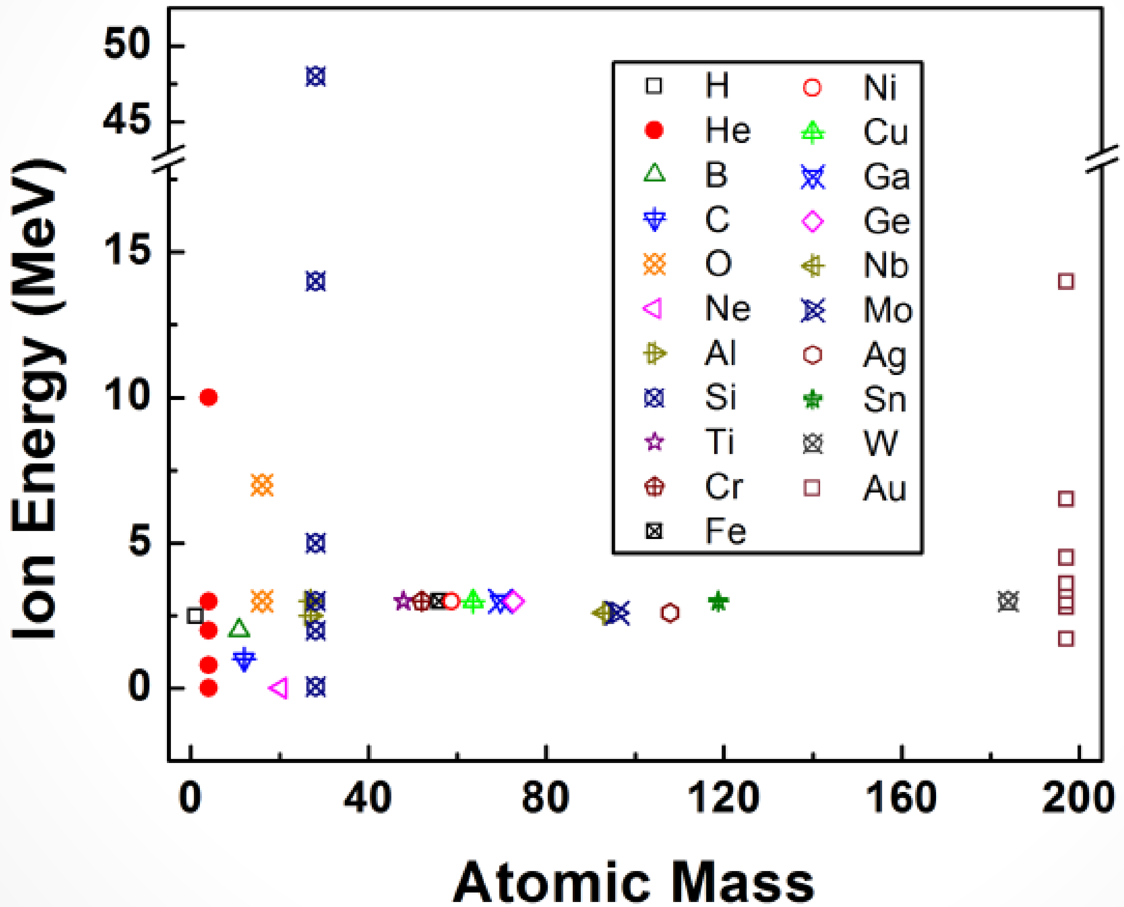


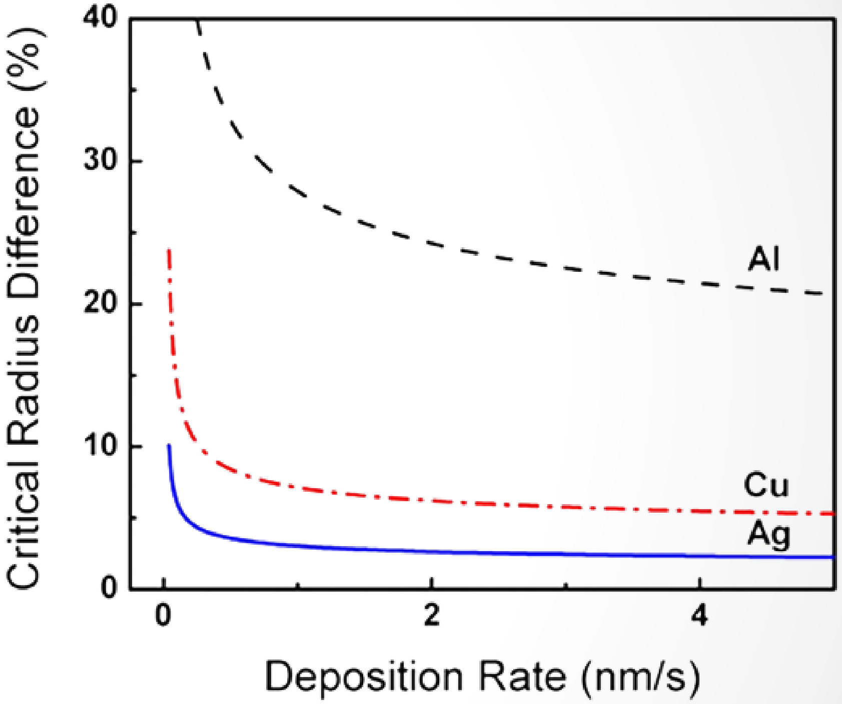
Hattar, *et al*, Nucl Instr Meth Phys Res B, 2014.

Probability of Formation

$$r^*_{perfect} = \frac{\gamma}{\left(\frac{kT}{\Omega}\ln\left[\frac{J\sqrt{2\pi mkT}}{P_s}\right]\right)}$$

$$r^*_{twin} = \frac{\gamma}{\left(\frac{kT}{\Omega}\ln\left[\frac{J\sqrt{2\pi mkT}}{P_s}\right] - \frac{\gamma_t}{h}\right)}$$

γ - surface energy
k - Boltzmann constant
T - substrate temperature
Ω - atomic volume
m - atomic mass of the film species
P_s - vapor pressure above the target
γ_t - twin boundary energy
J - deposition flux
h - height of the nucleus

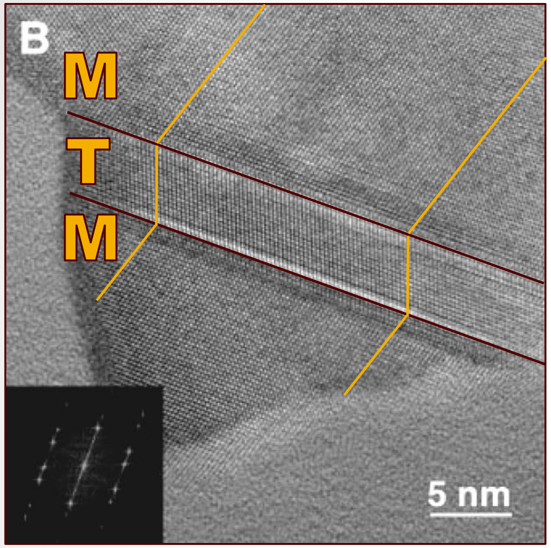


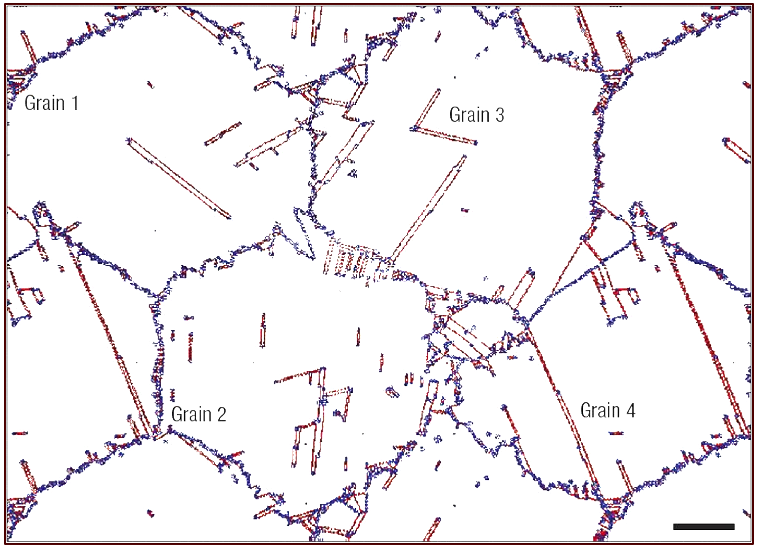
- Higher deposition rates and lower twin boundary energy favor twin formation

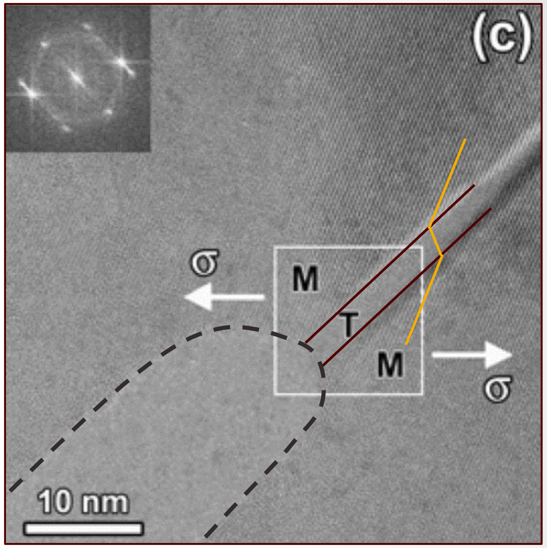
Bufford, D., et al. *Acta Mater,* **Vol. 59, 1, 2011.**

53

Deformation twins in Al



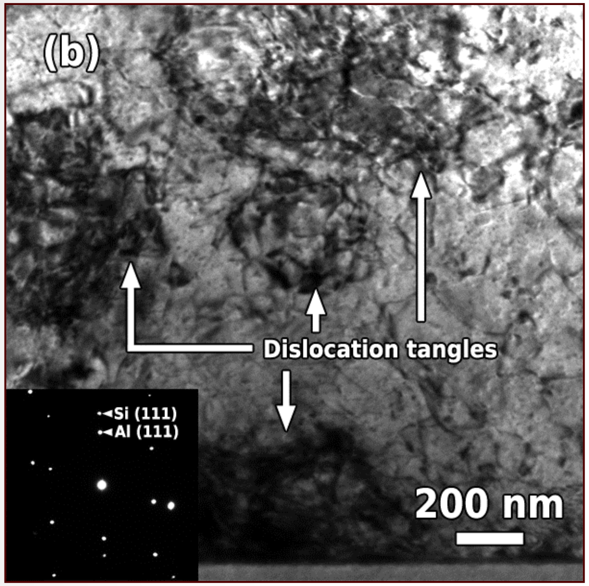


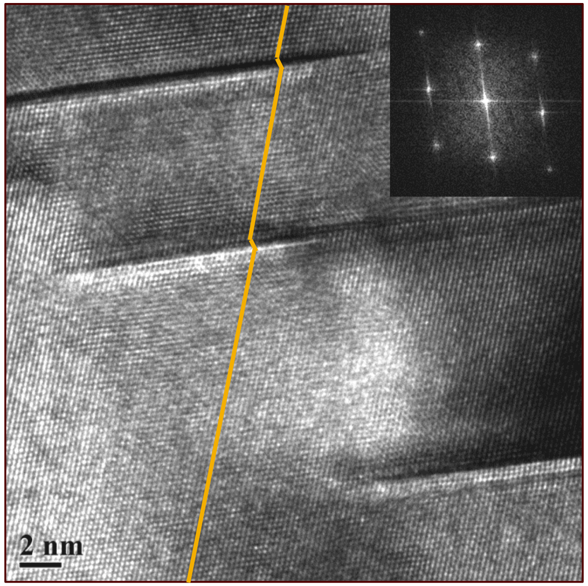


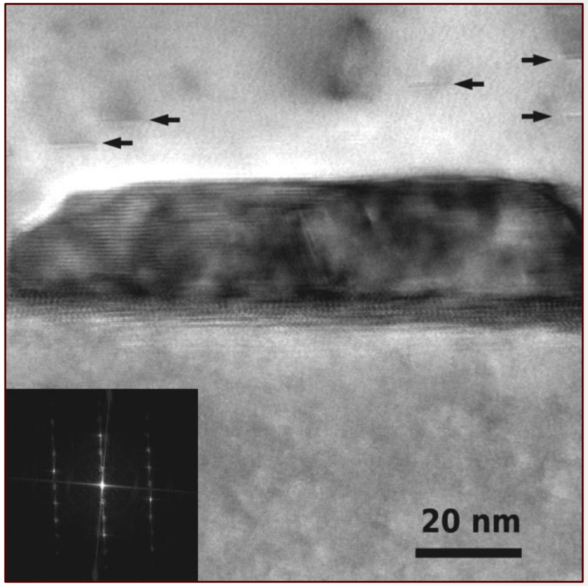
- Associated with extremely…
  - Low temperature……………. (77 K)
  - High strain rate………………. (>10^3 s^{-1})
  - Small grain size………………. (<8 nm)
  - High stress concentration

Chen, M. *et al*, *Science* **300, 2003.**
Li, B.Q. *et al*, *Phys Rev Lett* **102, 2009.**
Yamakov, V. *et al*, *Nat Mater* **1, 2002.**

Epitaxial Al / Si(111)







- Distinct lack of twins
- A few small, unstable stacking faults
- Rare twins at Al/Si interface

Bufford, D., et al. *Mater Res Lett*, **Vol. 1, 1, 2013.**

Mechanical Properties

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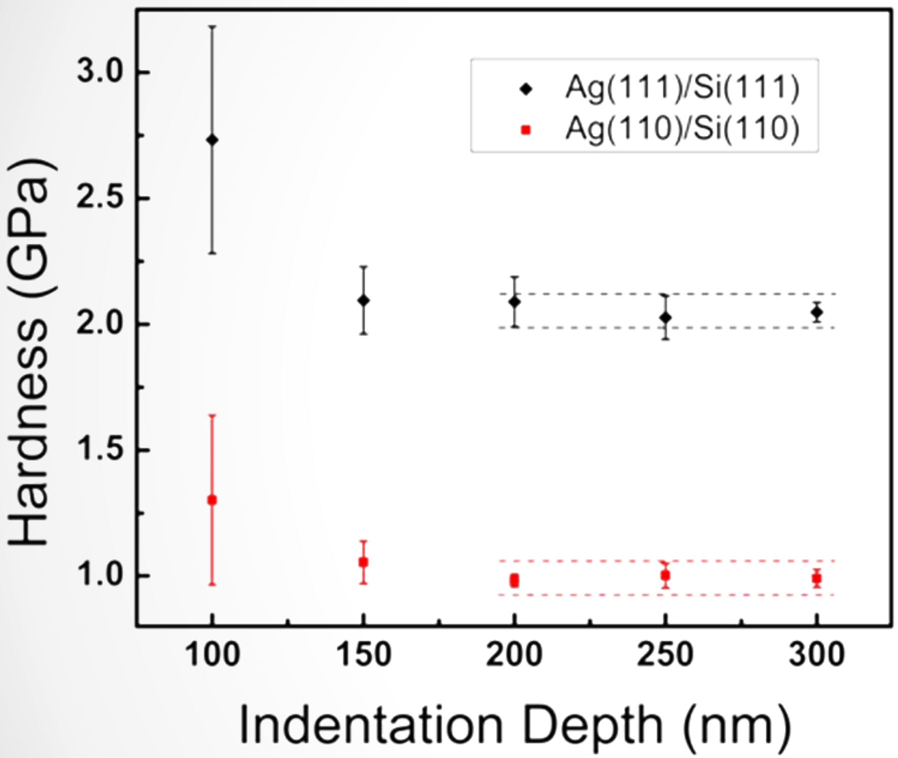
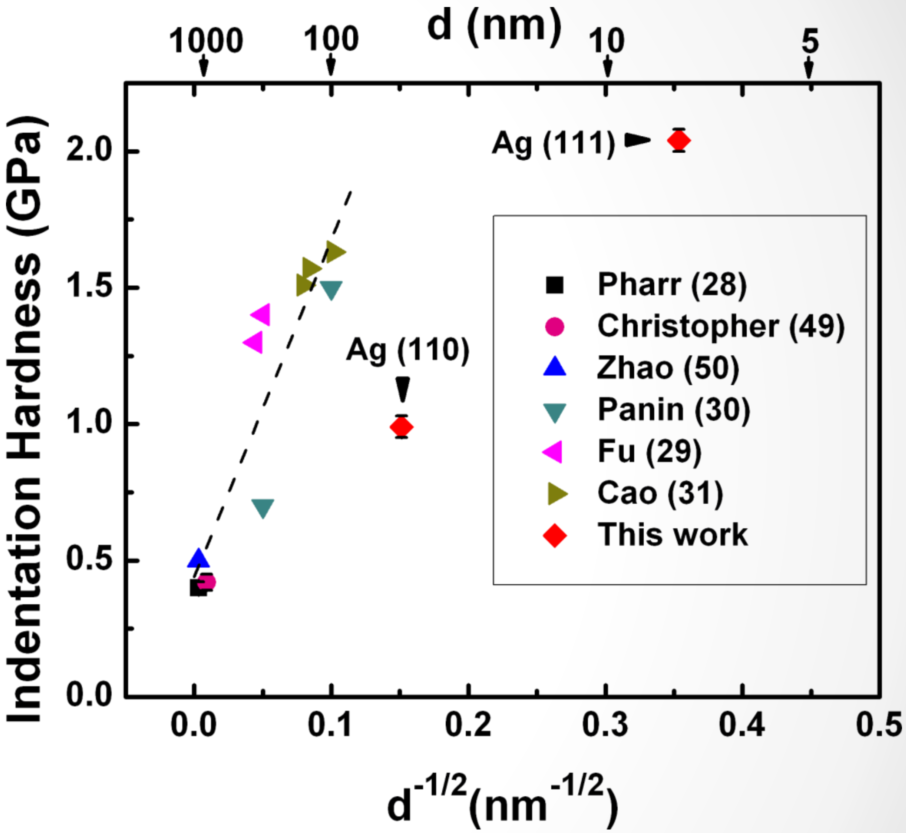




Hardness vs. depth profile

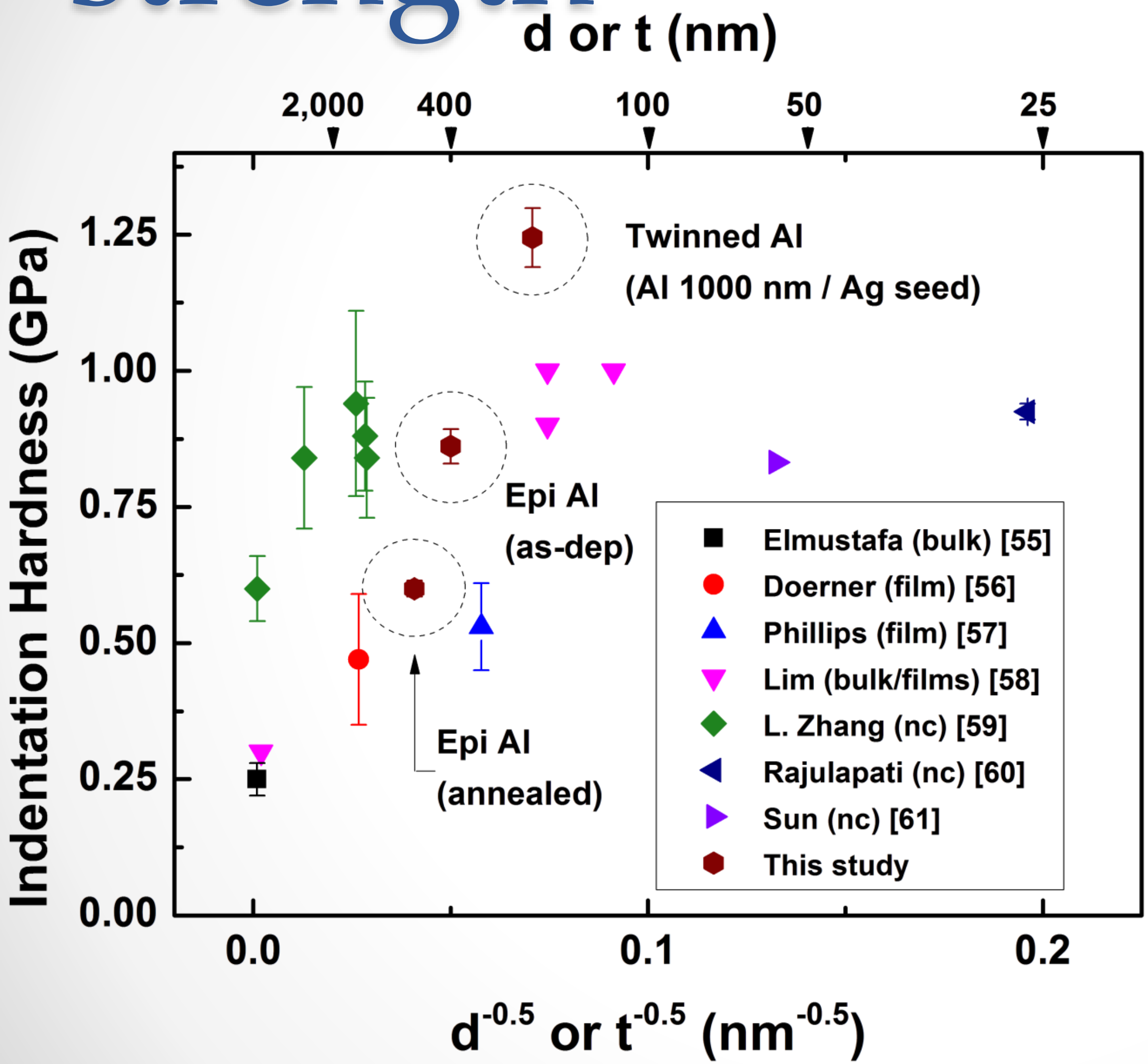
- Plateau achieved by ~200nm
- (111) and (110) films differ greatly
- Both films are harder than bulk

Hall-Petch type plot

- (111) film is among hardest reported
- Positions are anomalous

Bufford, D., et al. *Acta Materialia*, **Vol. 59, 1, 2011.**

Nanotwinned Al strength

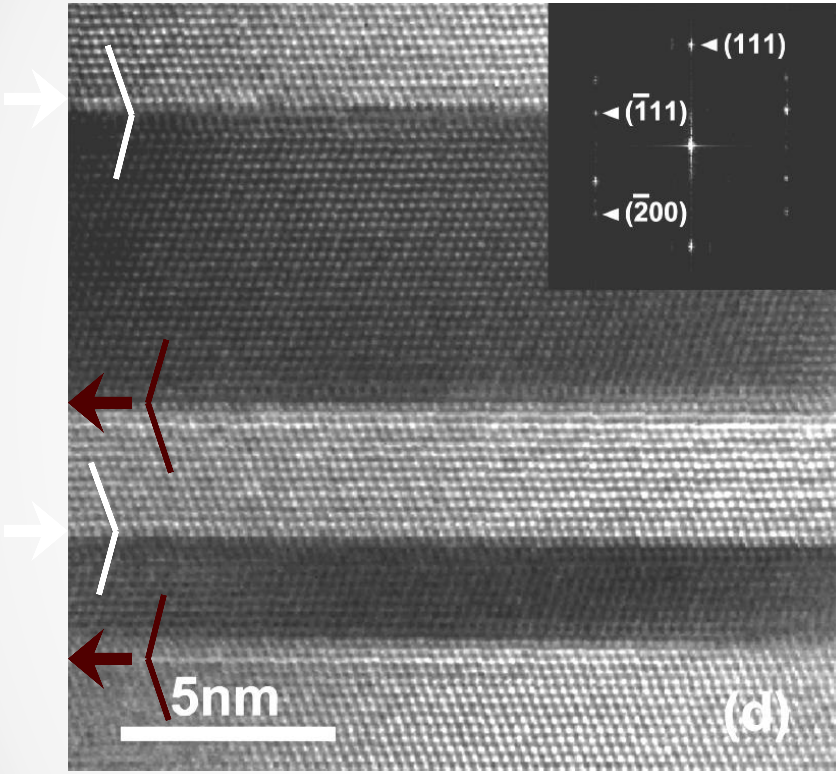


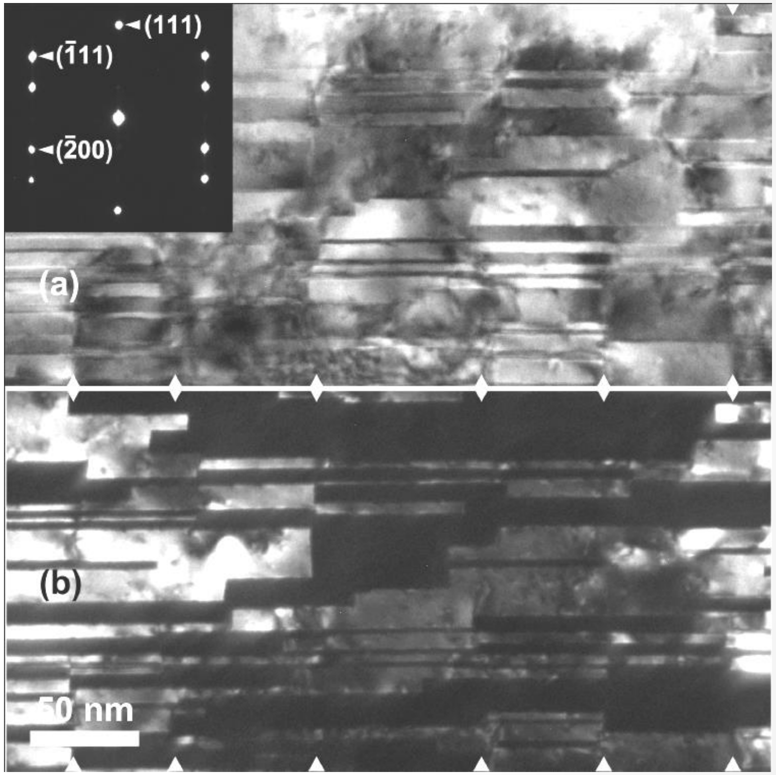
High Strength

- nt Al among hardest reporte

Bufford, D., *et al*, *Mater Res Lett*, **Vol. 1, 1, 2013.**

Epitaxial Ag(111)/Si(111)





High resolution XTEM

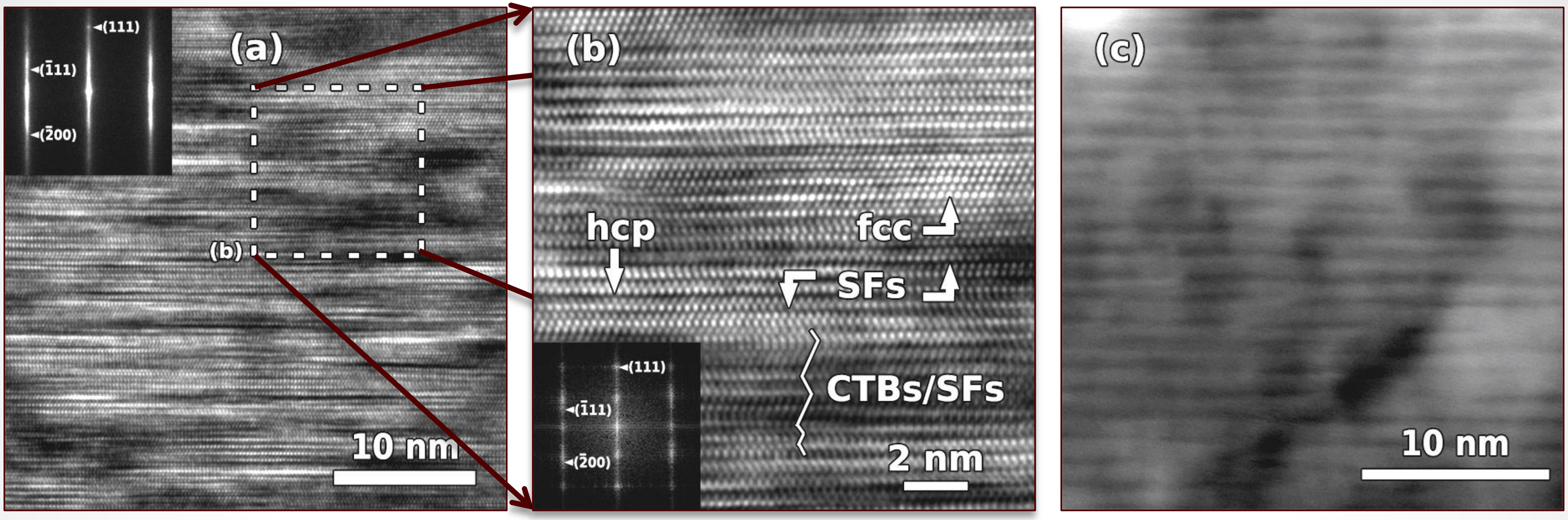
- Coherent TBs obvious in micrograph and FFT

Bright/Dark-field XTEM

- Same area
- Many twin variants pass through the domain boundaries

Bufford, D., et al. *Acta Mater,* **Vol. 59, 1, 2011.**

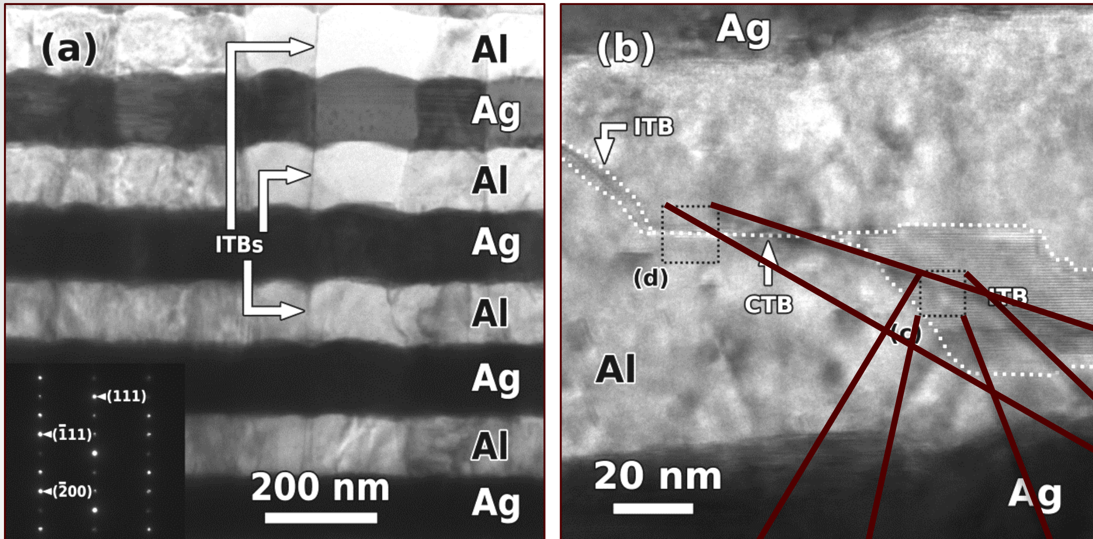
Ag/Al multilayers: h = 1 nm



- Extremely dense stacking defects
- Local areas with twins, stacking faults, and hcp stacking
- STEM shows that the layers are still chemically modulated

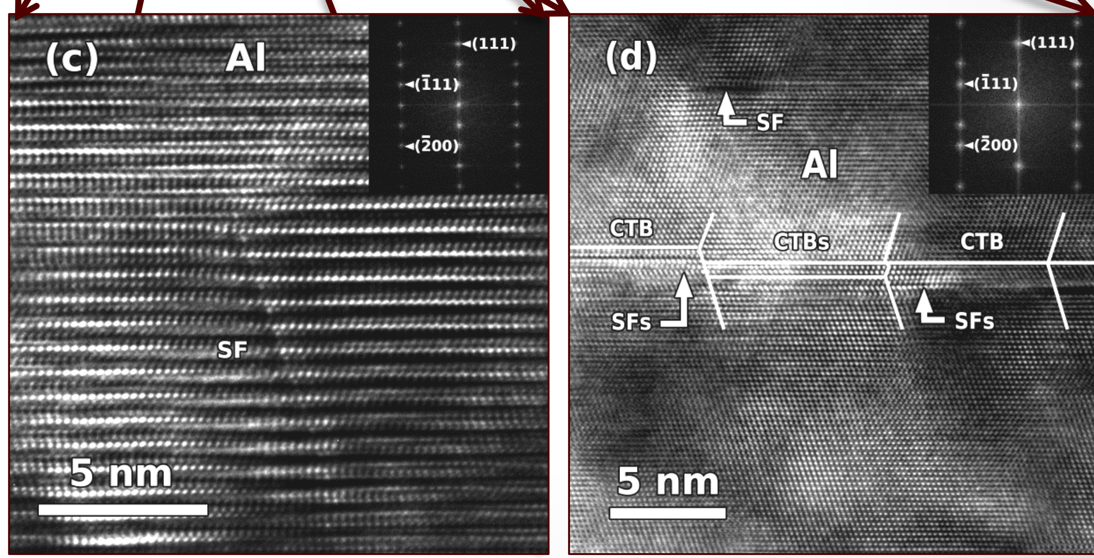
Bufford, D., *et al*, *Appl Phys Lett* **101, 2012**

Ag/Al multilayers: h = 100 nm



Low magnification

- Mixed TBs present in Al layers
- Penetrate through layers

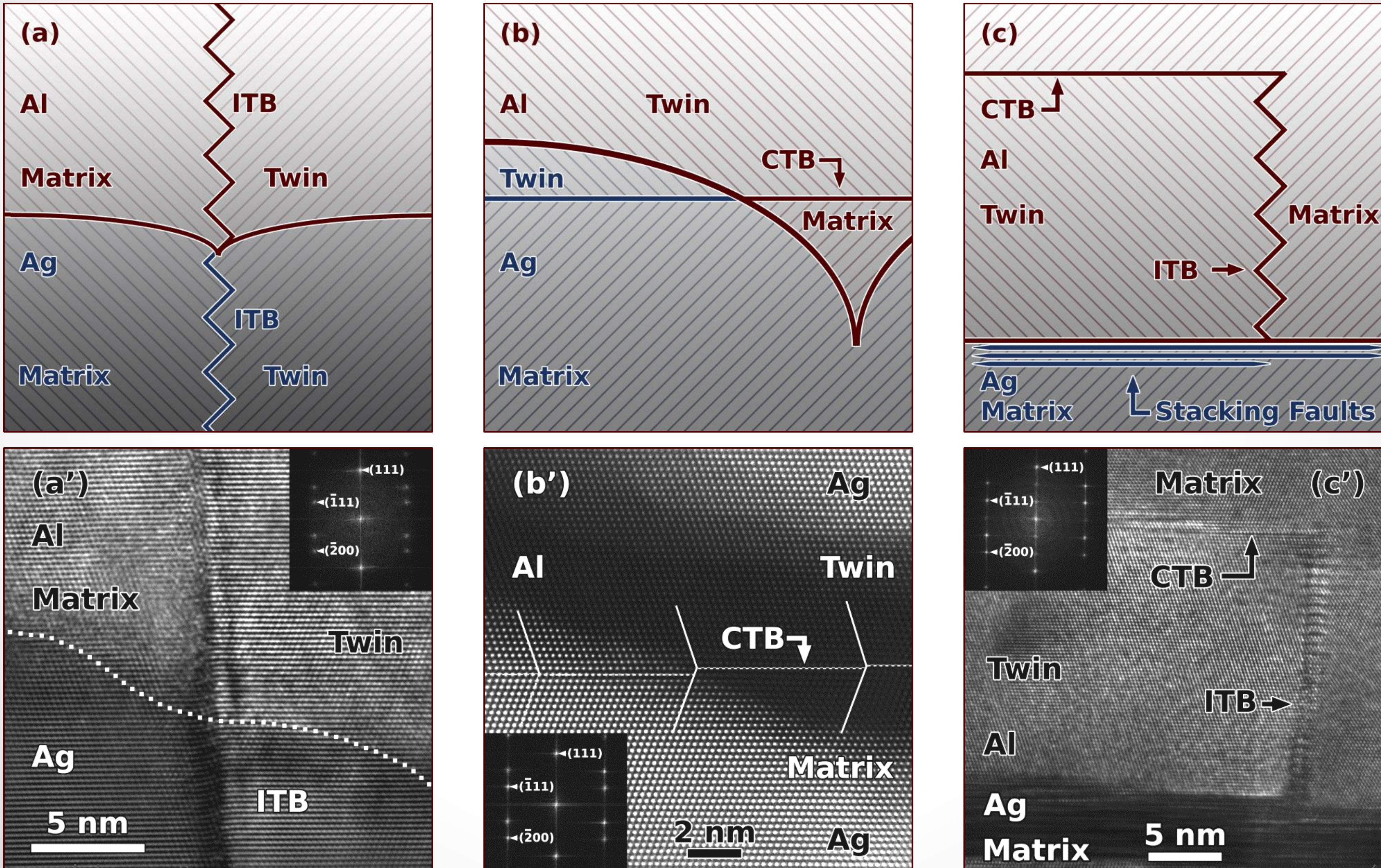


Higher magnification

- ITB and CTB structures confirmed

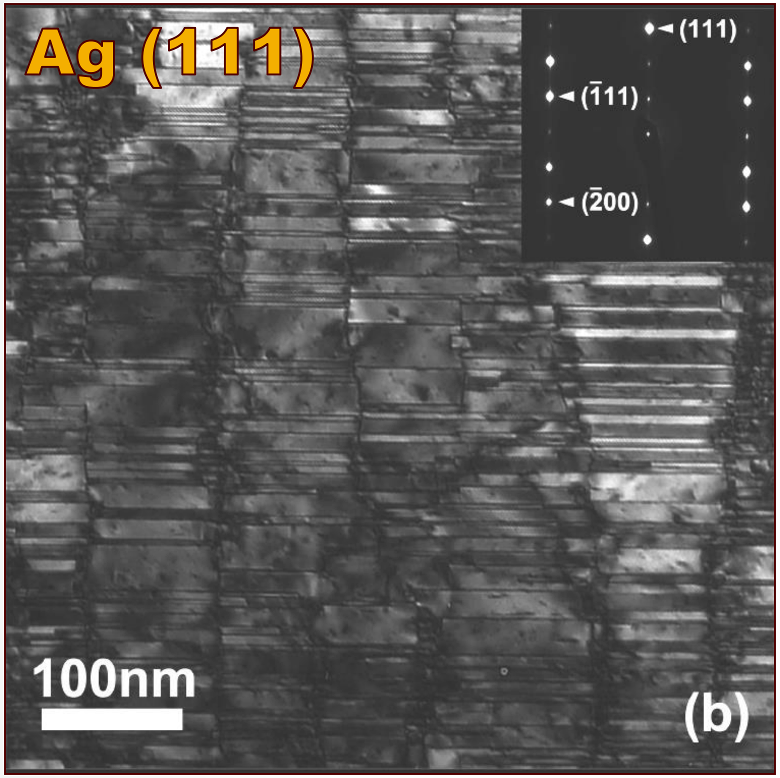
Bufford, D., *et al*, *Mater Res Lett*, **Vol. 1, 1, 2013.**

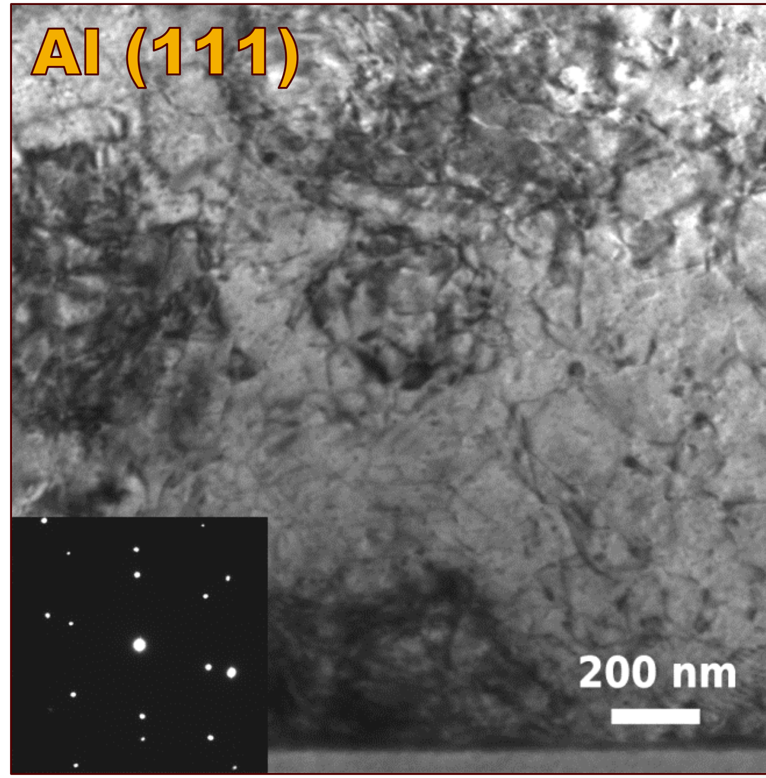
Twin replication mechanisms



Bufford, D., *et al, Mater Res Lett,* **Vol. 1, 1, 2013.**

Growth twin formation problems

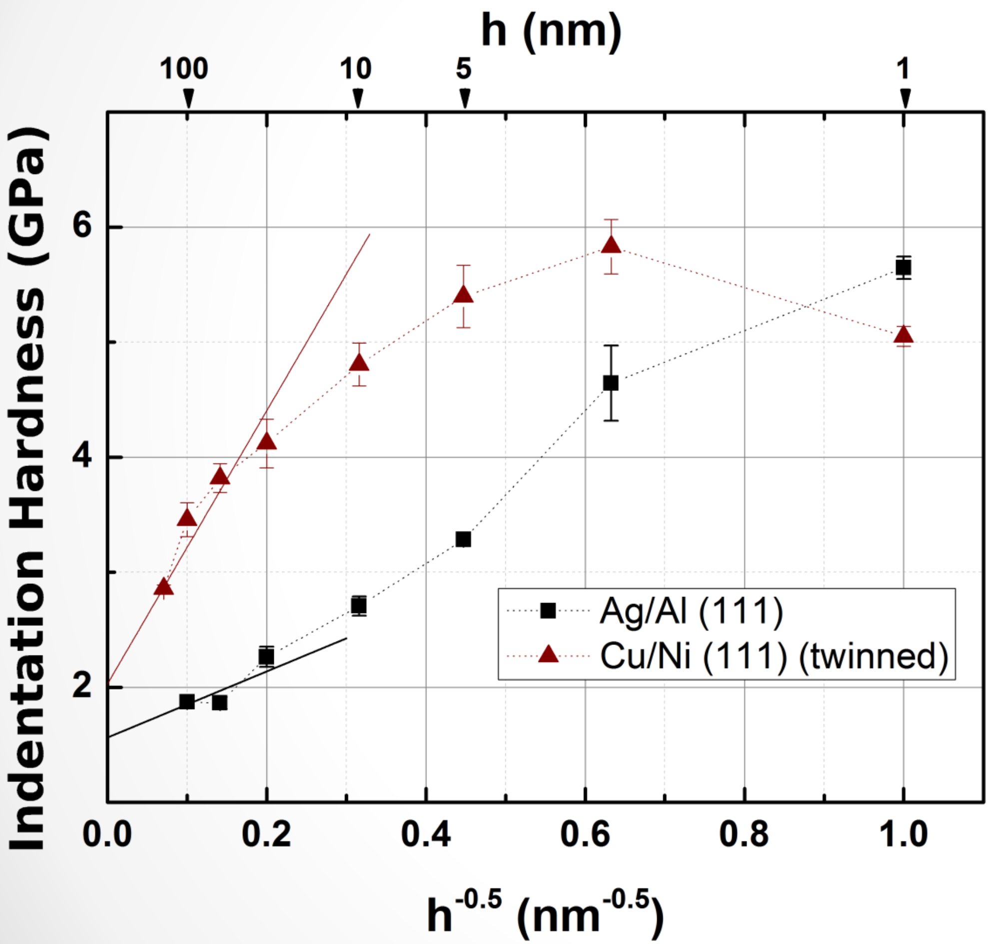




- Similar deposition conditions yield dramatically different results

Bufford, D., et al. *Acta Mater,* **Vol. 59, 1, 2011.**
Bufford, unpublished.

Ag/Al Multilayer strength



Unexpected behavior

- Very high hardness
- No softening
- Explained by multilayer film strengthening models
  - Shear modulus mismatch
  - SFE mismatch
  - Slip system discontinuity

Bufford, D., *et al*, *Appl Phys Lett* **101, 2012.**

Ag/Al Multilayer Film Strength

The important contributors to strength in Ag/Al films are…

$$\tau^* = \tau_K^* + \tau_{ch}^* + \tau_\omega^*$$

Koehler stress (shear modulus)

$$\tau_K^* = \frac{\mu_1(\mu_2 - \mu_1)b}{4\pi(\mu_2 + \mu_1)h} \longrightarrow 0.05 \text{ GPa}$$

Chemical stress (SFE)

$$\tau_{ch}^* \approx \frac{\delta\gamma}{b} \longrightarrow 0.5 \text{ GPa}$$

ω interaction (Slip system continuity)

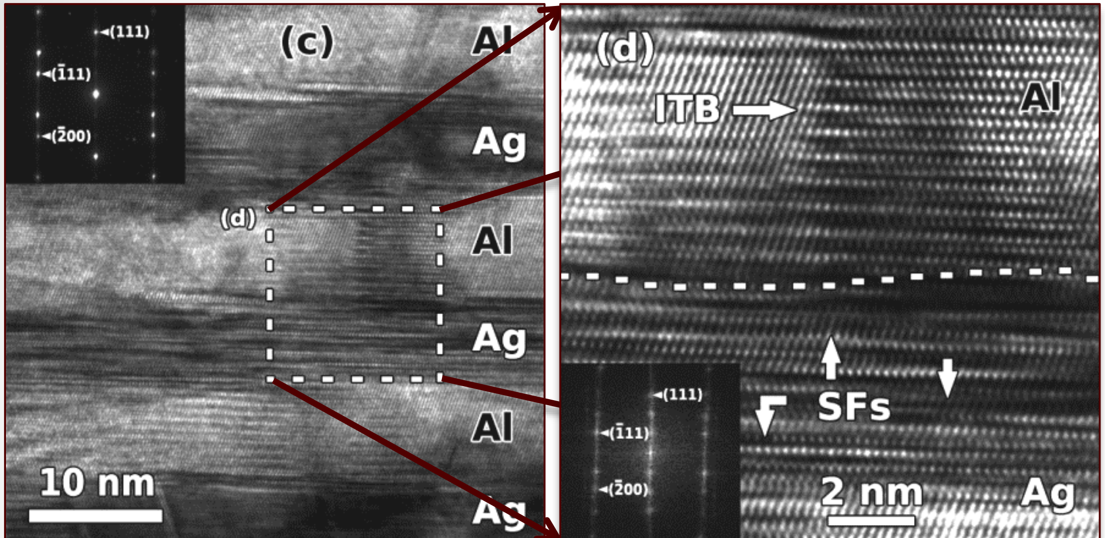
$$\tau_\omega^* \longrightarrow 0.1 \text{ GPa}$$

H_{max} ≈ 5.2 GPa

Measured H_{max} ≈ 5.5 Gpa

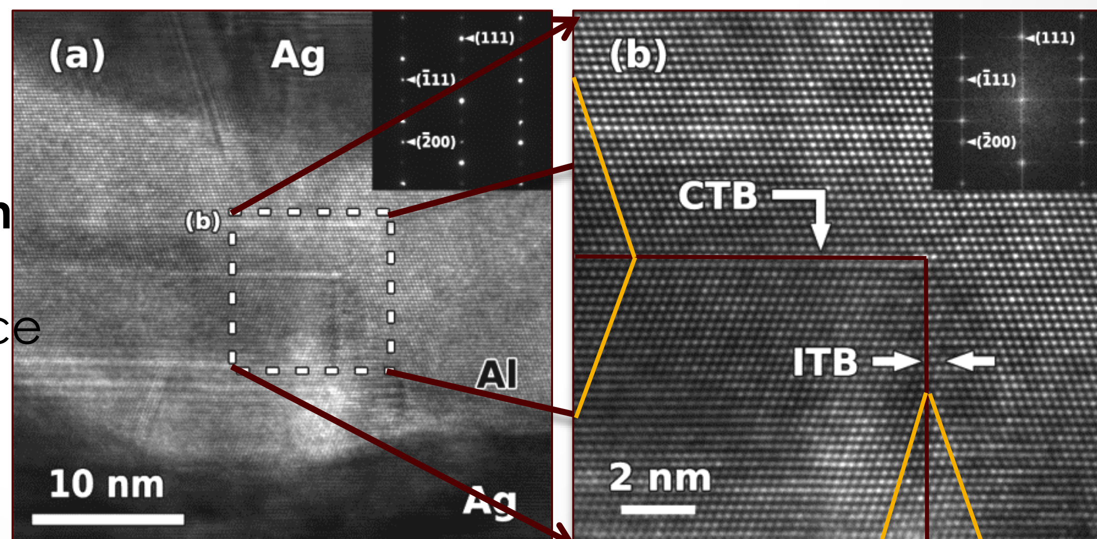
…consistent with experimental observations

Ag/Al multilayers: *h* = 10 and 25 nm



10 nm: high magnification
- Wide ITBs present in Al layers
- Penetrate through the layer

25 nm: high magnification
- CTBs appear in Al
- Away from layer interface



Bufford, D., *et al*, *Appl Phys Lett* **101, 2012**

Ag/Al multilayers: mixed *h*

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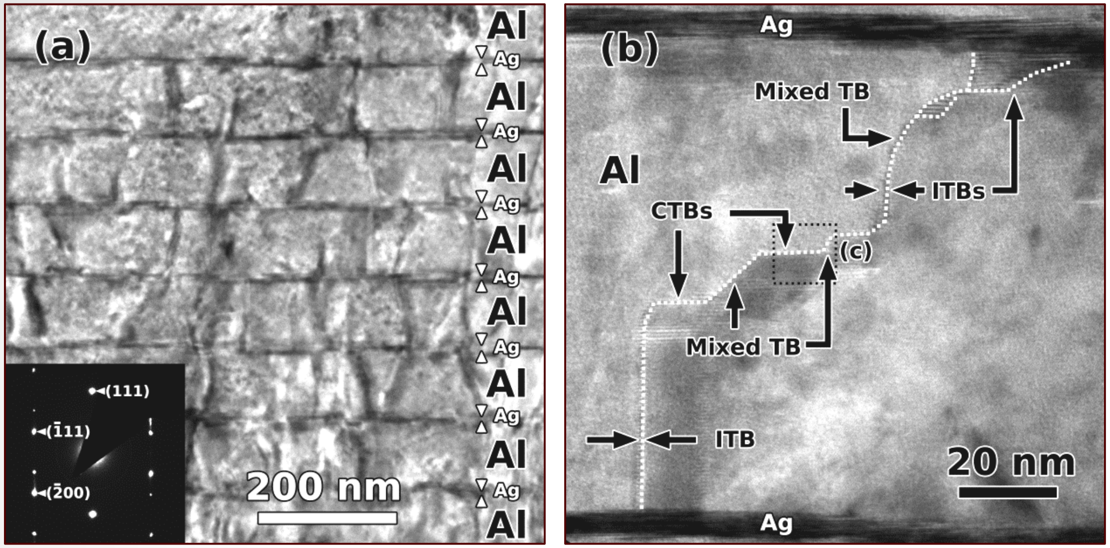
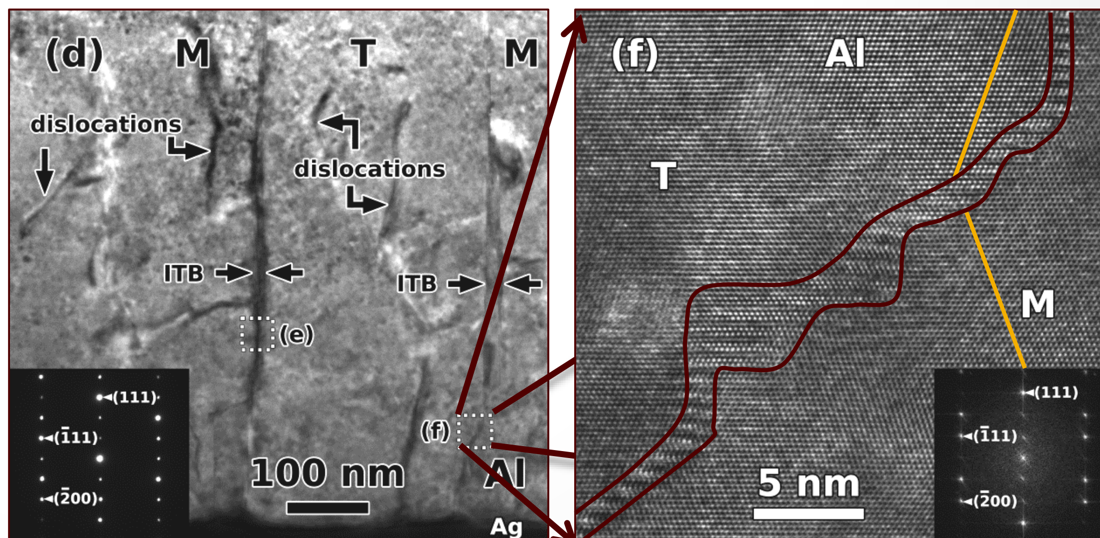


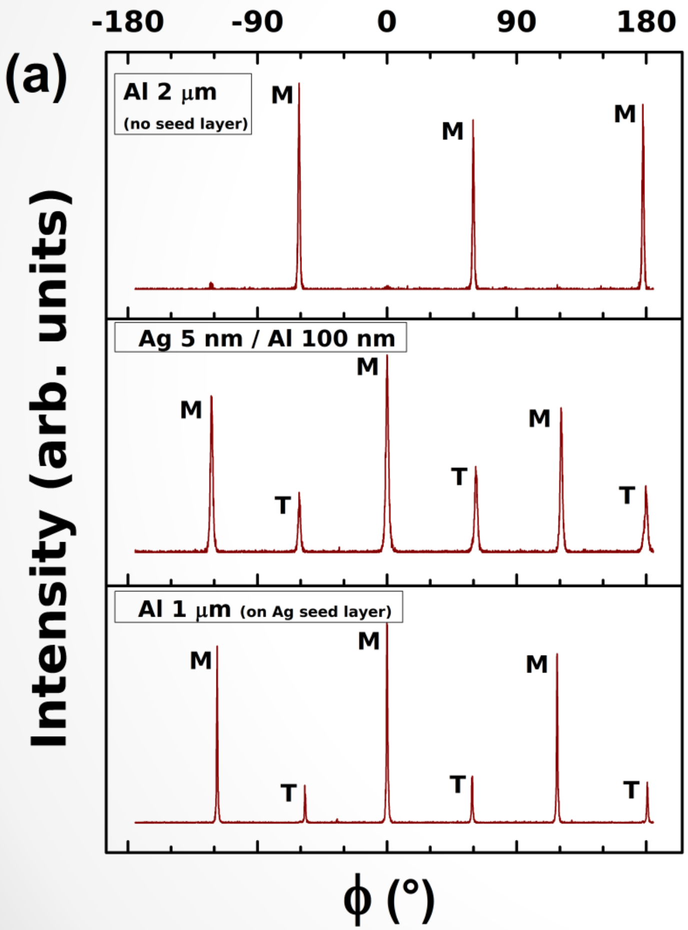
Ag 5 nm / Al 100 nm
- Similar to 100 / 100nm films

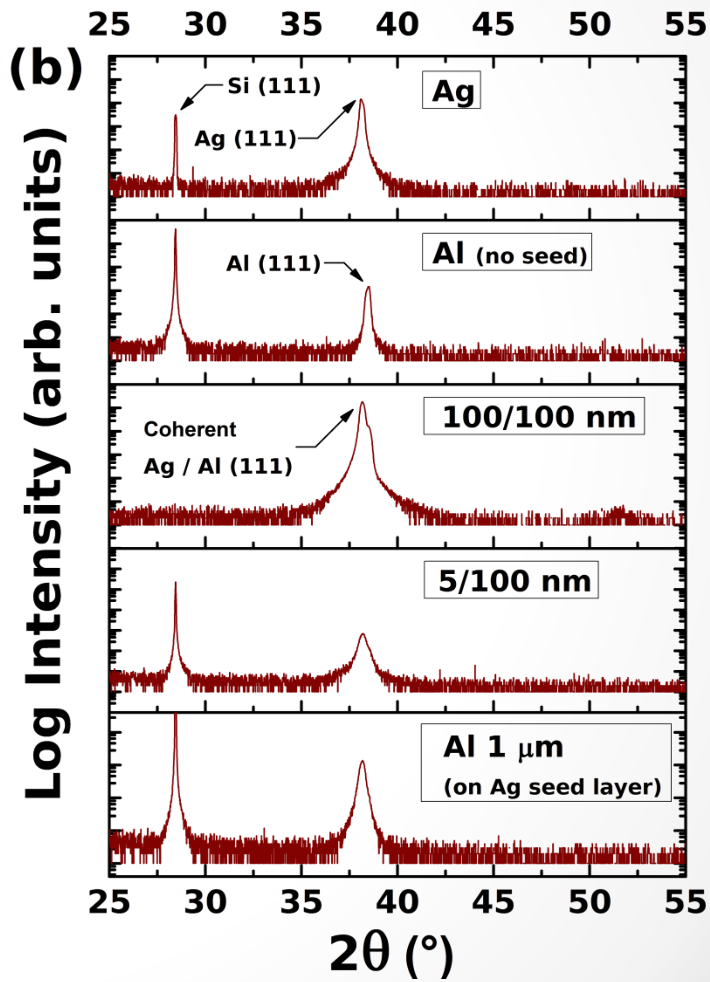
Ag 100 nm / Al 1000 nm
- Preference for ITBs
- Some mixed ITB/CTB segments



Bufford, D., *et al*, *Mater Res Lett*, **Vol. 1, 1, 2013.**

Twins in thicker Ag/Al layers





Bufford, D., *et al*, *Mater Res Lett*, **Vol. 1, 1, 2013.**

Material Data

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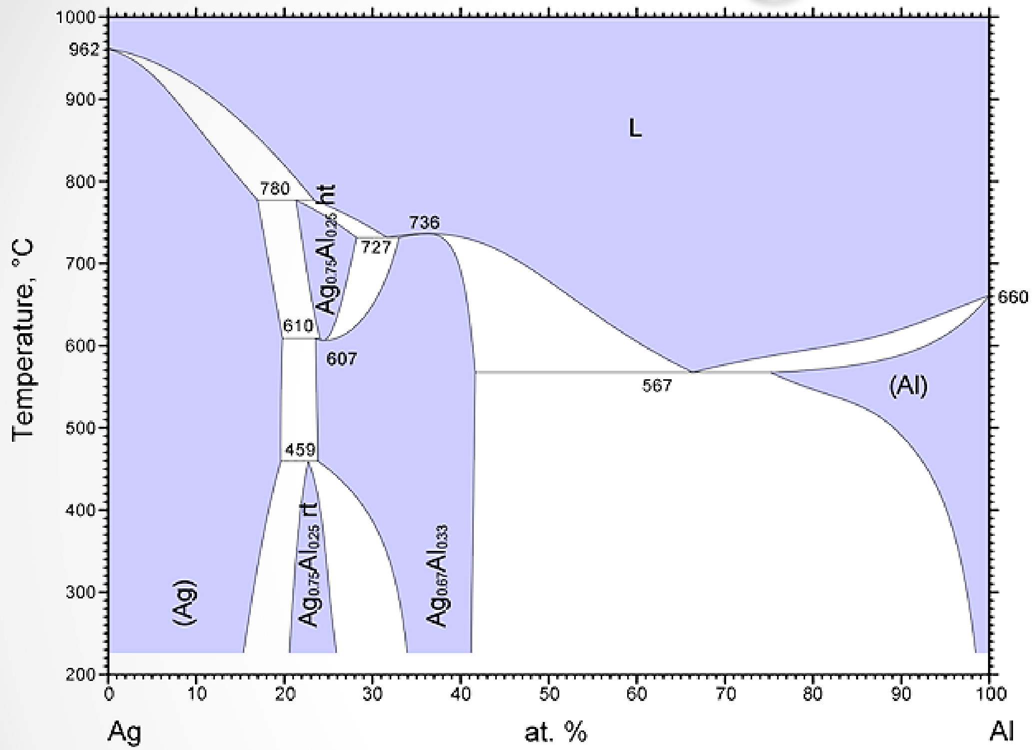
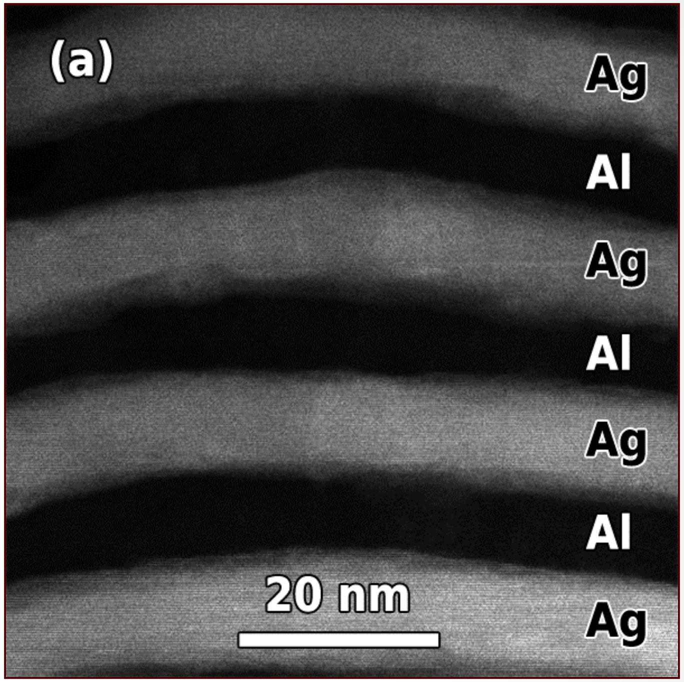
	Ag	Al	Cu
Young's modulus (GPa)	83	69	117
Shear modulus (GPa)	30	26	48
Lattice parameter (Å)	4.09	4.05	3.61
Burgers vector (Å)	2.89	2.86	2.55
Poisson's ratio	0.37	0.35	0.34
Stacking Fault energy (mJ/m^2)	22	120-165	45
CTB energy (mJ/m^2)	8	75	24-39
ITB energy (mJ/m^2)	126	223-357	550-714

Intermixing





- Guns shuttered between layers
- Heating avoided
- HRSTEM micrographs show chemical modulation

Lim, S.S., et al, 1995 (via ASM Alloy Phase Diagrams Center)
Bufford, D., et al, *Mater Res Lett,* **Vol. 1, 1, 2013.**

CINT: a DOE Office of Science National User Facility

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