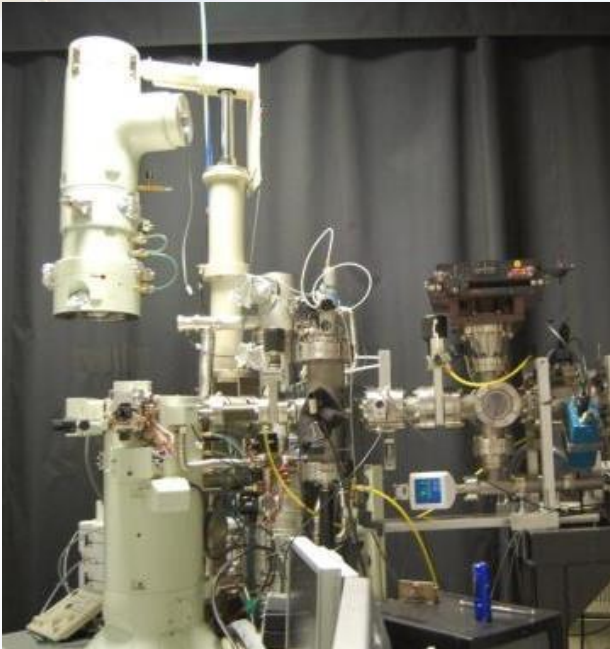


In situ Ion Irradiation Transmission Electron Microscope at Sandia National Laboratories

SAND2016-4924PE



Khalid Hattar

- Staff Member at Sandia National Laboratories' Ion Beam Lab since 2008 working in:
 - Development of advanced in-situ TEM techniques
 - Ion beam modification and
 - Accelerated aging in harsh environments
- PhD in Materials Science from University of Illinois, Urbana-Champaign (UIUC) under Ian M. Robertson on:
 - “Thermal and mechanical stability of nanograined face-centered cubic free-standing films.”
- BS in Chemical Engineering with an emphasis in Materials Science from University of California, Santa Barbara (UCSB)

EXTERNAL COLLABORATORS and Co-Editors (last 5 years)

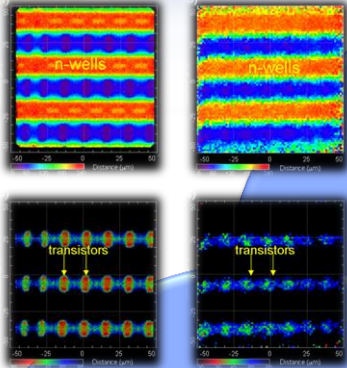
- ANL: Mark Kirk, Meimei Li, Pete Baldo (retired); LANL: Osman Anderoglu, J. Kevin Baldwin, Patricia Dickerson, Nan Li, Stuart A. Maloy, Cheng Sun, Blas P. Uberuaga, Yong Wang; ORNL: Philip D. Edmondson; Drexel: Osman El-Atwani, Christopher Barr, James E. Nathaniel, Mitra L. Taheri, Christopher Weinberger, Garrett Tucker; Georgia Tech: Kacher, Josh, Arron Dunn; Northwestern: Brittany R. Muntifer; RPI: Robert Hull; Stony Brook U.: Olivia Donaldson, Jason Trelewicz; Texas A&M: Michael Demkowicz; Tufts Univ: Jianmin Qu; U. of Alabama: Gregory B. Thompson, Luke N. Brewer; UCB: Chisholm, Claire, Andrew M. Minor, Peter Hosemann; UCSB: Daniel S. Gianola; UIUC: J.P. Allain; UM: Amit Misra; UNT: Jon-Erik Mogonye, Thomas W. Scharf; UT-Austin: Paulo J. Ferreira; UVa: Ramez Cheaito, Caroline S. Gorham, Patrick E. Hopkins; U. Wisc.: Ian M. Robertson; Oxford: Xiaou Yi, Michael L. Jenkins, Steve G. Roberts; Univ. of Huddersfield: Jonathan A. Hinks, Steve E. Donnelly; Exxon-Mobile: Jamey A. Fenske IBM: Frances M. Ross; Intel: Shreyas, Rajasekhara, K.J. Ganesh, Janelle Villone; Nanomegas: Amith Darba; Protochips: Benjamin W. Jacobs; TerraPower: Gregory A. Vetterick;

SANDIA COLLABORATIONS

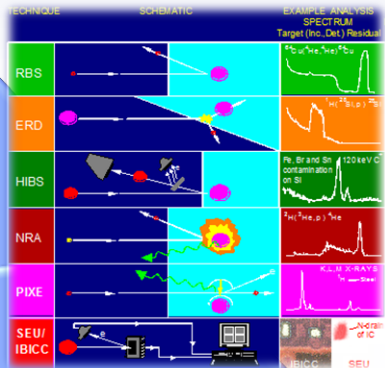
- Fadi F. Abdeljawad, Mark D. Allendorf, Corbette Battaile, Thomas E. Beechem III, Brad L. Boyce, Timothy J. Boyle, Darren W. Branch, Thomas Buchheit, Daniel C. Bufford, Daniel, L. Buller, Jay D. Carroll, Michael Chandross, Blythe G. Clark, Remi P.M. Dingreville, F. Patrick Doty, Barney L. Doyle, Matthew Eichenfield, Patrick L. Feng, Stephen M. Foiles, David M. Follstaedt (retired), Ronald Goeke, Steven H. Goods (retired), Aaron C. Hall, Michelle A. Hekmaty, Bernadette A. Hernandez-Sanchez, Sara J. Homeijer, Jon F. Ihlefeld, James A. Knapp (retired), Paul G. Kotula, Jonathan Madison, Douglas L. Medlin, Joesph Michael, William M. Mook, Tina M. Nenoff, Roy H. Olsson III, Henry A. Padilla II, Edward S. Piekos, S. V. Prasad, Sarah H. Pratt, Joseph D. Puskar, David B. Robinson, Mark A. Rodriguez, Pylin Sarobol, Darryl Y. Sasaki, Peter A. Sharma, Vitalie Stavila, Vina Tikare, Gyorgy Vizkelethy, Michael Wiwi, Pin Yang, and Jonathan A. Zimmerman

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Sandia's Ion Beam Laboratory



Ion Beam Analysis (IBA)

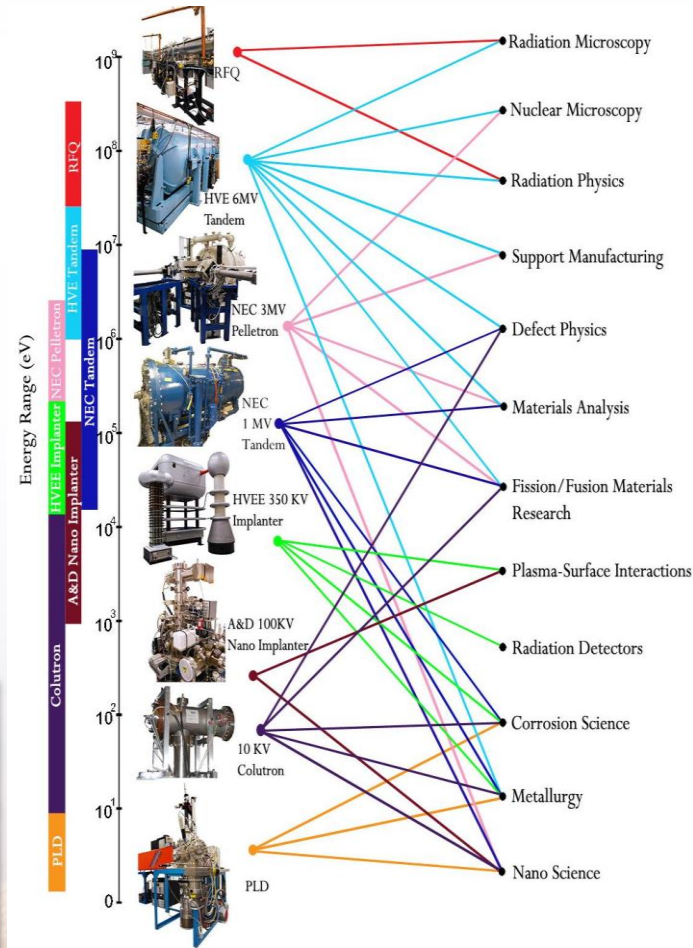
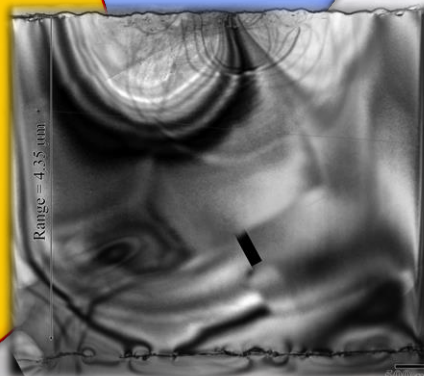


Radiation Effects Microscopy (REM)



Ion Beam Modification (IBM)

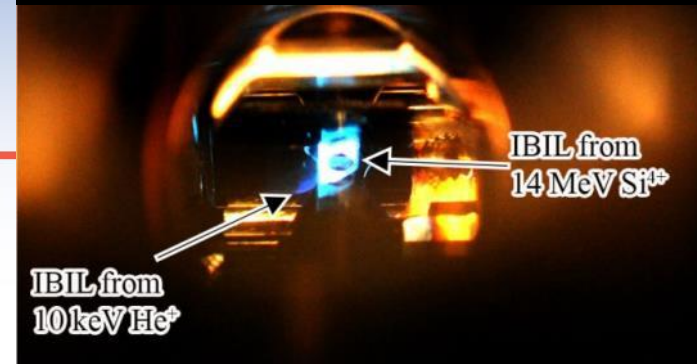
In situ Ion Irradiation Transmission Electron Microscopy (³TEM)



Sandia's Concurrent I³TEM Facility

Collaborator: D.L. Buller

IBIL from a quartz stage inside the TEM

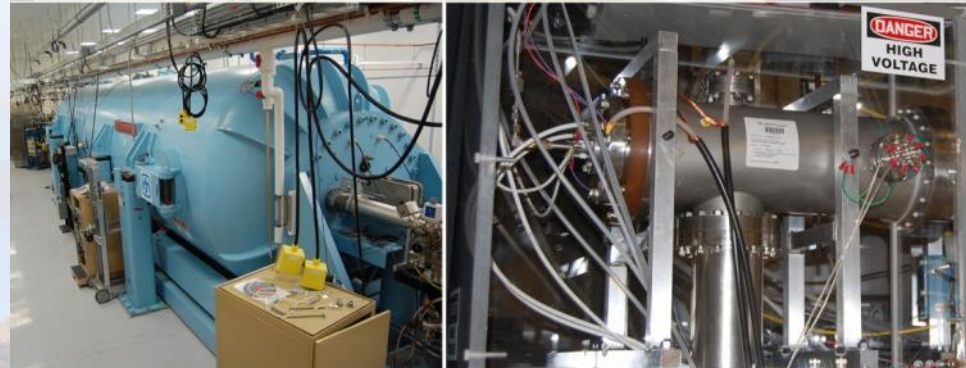
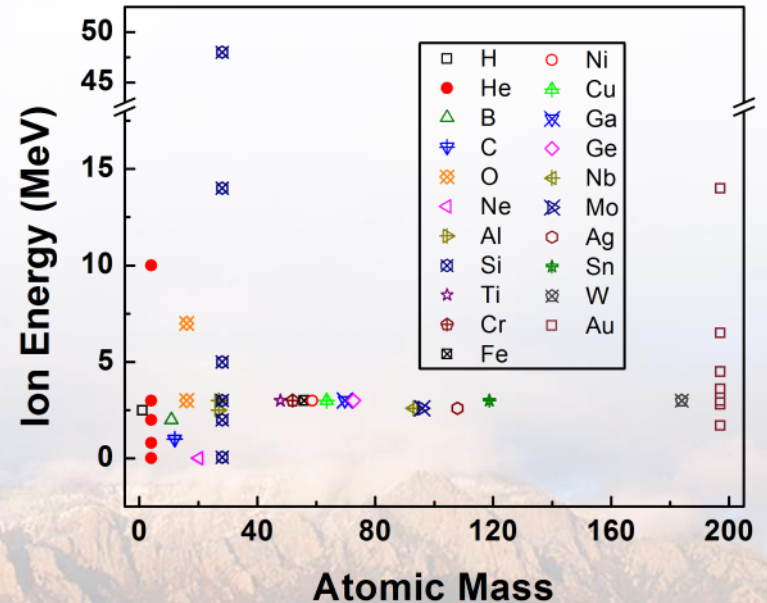


Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution

10 kV Colutron - 200 kV TEM - 6 MV Tandem



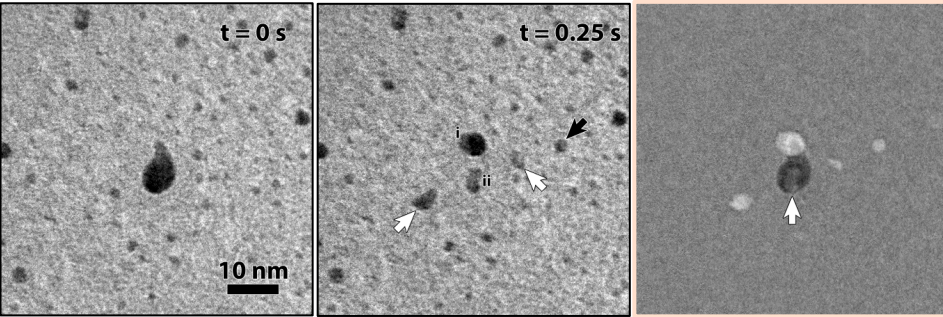
Ion species & energy introduced into the TEM



I³TEM capabilities

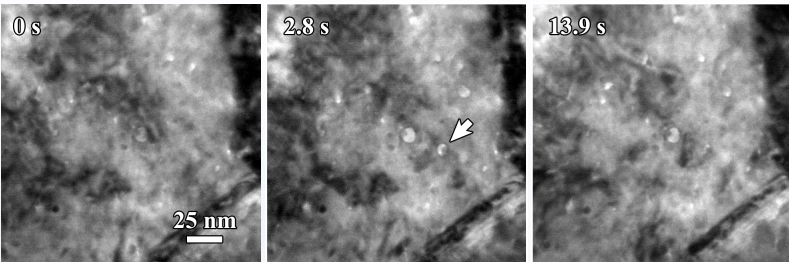
Radiation Environments

- Single Ion Strikes through 100s of dpa



A 46 keV Au¹⁻ ion into 5 nm Au nanoparticles

- Including Multiple Concurrent Ion Beams



Simultaneous TEM Triple Beam: 2.8 MeV Au⁴⁺ + 10 keV He⁺/D₂⁺

Characterization Tools

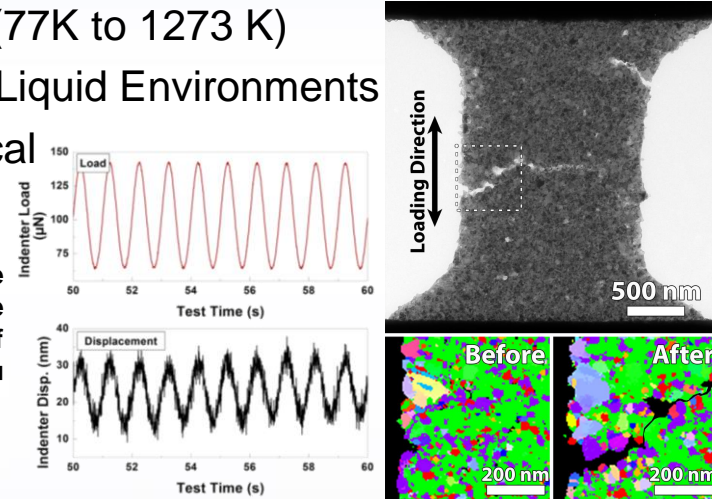
- Tomography ($\pm 81^\circ$ & double-tilt rotate)
- Precision Electron Diffraction (PED)
- Cathodoluminescence and Ion Beam Induced Luminescence

PED mapping of Nanocrystalline Au before and after 10 MeV Si irradiation

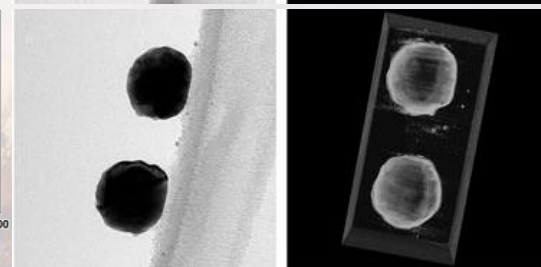
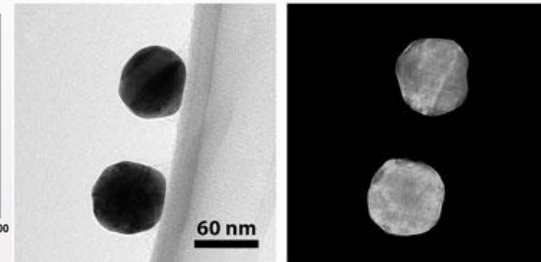
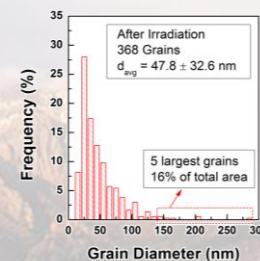
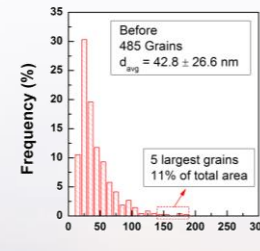
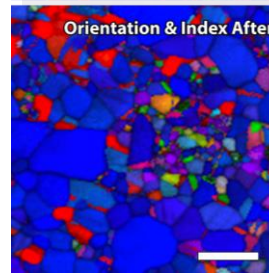
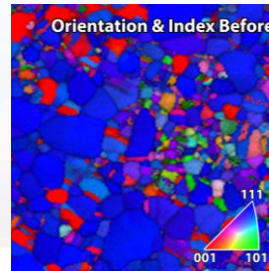
Other Extreme Environments

- Thermal (77K to 1273 K)
- Gas and Liquid Environments
- Mechanical

Quantitative High Cycle Fatigue of nc-Cu



BF images and 3D reconstructions before and after 2.8 MeV Au Irradiation of 60 nm Au NPs



Recent Publications Highlighting These Capabilities

1. Chen, Y., N. Li, D. C. Bufford, J. Li, K. Hattar, H. Wang, and X. Zhang. "In situ study of heavy ion irradiation response of immiscible Cu/Fe multilayers." *Journal of Nuclear Materials* (2016).
2. Muntifering, Brittany, Sarah Jane Blair, Cajer Gong, Aaron Dunn, Remi Dingreville, Jianmin Qu, and Khalid Hattar. "Cavity Evolution at Grain Boundaries as a Function of Radiation Damage and Thermal Conditions in Nanocrystalline Nickel." *Materials Research Letters* (2015): 1-8.
3. Bufford, D. C., F. F. Abdeljawad, S. M. Foiles, and K. Hattar. "Unraveling irradiation induced grain growth with in situ transmission electron microscopy and coordinated modeling." *Applied Physics Letters* 107, no. 19 (2015): 191901.
4. Sun, C., M. Kirk, M. Li, K. Hattar, Y. Wang, O. Anderoglu, J. Valdez, B. P. Uberuaga, R. Dickerson, and S. A. Maloy. "Microstructure, chemistry and mechanical properties of Ni-based superalloy Rene N4 under irradiation at room temperature." *Acta Materialia* 95 (2015): 357-365.
5. Yi, Xiaou, Michael L. Jenkins, Khalid Hattar, Philip D. Edmondson, and Steve G. Roberts. "Characterisation of radiation damage in W and W-based alloys from 2MeV self-ion near-bulk implantations." *Acta Materialia* 92 (2015): 163-177.
6. Cheaito, Ramez, Caroline S. Gorham, Amit Misra, Khalid Hattar, and Patrick E. Hopkins. "Thermal conductivity measurements via time-domain thermoreflectance for the characterization of radiation induced damage." *Journal of Materials Research* 30, no. 09 (2015): 1403-1412.
7. El-Atwani, O., S. Gonderman, S. Suslov, M. Efe, G. De Temmerman, T. Morgan, K. Bystrov, K. Hattar, and J. P. Allain. "Early stage damage of ultrafine-grained tungsten materials exposed to low energy helium ion irradiation." *Fusion Engineering and Design* 93 (2015): 9-14.
8. Zhao, Xinyu, Daniel J. Strickland, Peter M. Derlet, Mo-rigen He, You-Jung Cheng, Jue Pu, Khalid Hattar, and Daniel S. Gianola. "In situ measurements of a homogeneous to heterogeneous transition in the plastic response of ion-irradiated <111> Ni microspecimens." *Acta Materialia* 88 (2015): 121-135.
9. Cheaito, Ramez, Khalid Hattar, John T. Gaskins, Ajay K. Yadav, John C. Duda, Thomas E. Beechem, Jon F. Ihlefeld et al. "Thermal flux limited electron Kapitza conductance in copper-niobium multilayers." *Applied Physics Letters* 106, no. 9 (2015): 093114.
10. Barr, Christopher M., Leland Barnard, James E. Nathaniel, Khalid Hattar, Kinga A. Unocic, Izabela Szlurfarska, Dane Morgan, and Mitra L. Taheri. "Grain boundary character dependence of radiation-induced segregation in a model Ni-Cr alloy." *Journal of Materials Research* 30, no. 09 (2015): 1290-1299.
11. El-Atwani, O., K. Hattar, J. A. Hinks, G. Greaves, S. S. Harilal, and A. Hassanein. "Helium bubble formation in ultrafine and nanocrystalline tungsten under different extreme conditions." *Journal of Nuclear Materials* 458 (2015): 216-223.
12. Mogonye, J-E., K. Hattar, P. G. Kotula, T. W. Scharf, and S. V. Prasad. "He implantation for improved tribological performance in Au electrical contacts." *Journal of Materials Science* 50, no. 1 (2015): 382-392.
13. Chee, See Wee, Sarah H. Pratt, Khalid Hattar, David Duquette, Frances M. Ross, and Robert Hull. "Studying localized corrosion using liquid cell transmission electron microscopy." *Chem. Commun.* 51, no. 1 (2014): 168-171.
14. El-Atwani, Osman, A. Suslova, T.J. Novakowski, K. Hattar, M. Efe, S.S. Harilal, and A. Hassanein, "In-situ TEM/heavy ion irradiation on ultrafine- and nanocrystalline-grained tungsten: Effect of 3 MeV Si, Cu, and W ions" *Mater Charact* (2014), 99 (2015): 68-76.
15. Bufford, Daniel, and Khalid Hattar. "Physical response of gold nanoparticles to single self-ion bombardment" *Journal of Materials Research*, 29:20 (2014): 2387-2397. Invited
16. Hattar, Khalid, Bufford, Daniel C., Buller, Daniel, L. "Concurrent in situ ion irradiation transmission electron microscope." *Nuclear Instruments and Methods in Physics Research B* (2014), pp. 56-65
17. Gorham, Caroline S., et al. "Ion irradiation of the native oxide/silicon surface increases the thermal boundary conductance across aluminum/silicon interfaces." *Physical Review B* 90.2 (2014): 024301.
18. Chisholm, Claire, Khalid Hattar, and Andrew M. Minor. "In Situ TEM Concurrent and Successive Au Self-Ion Irradiation and He Implantation." *Materials Transactions* 55, no. 03 (2014): 418-422. Invited
19. Barr, Christopher M., Gregory A. Vetterick, Kinga A. Unocic, Khalid Hattar, Xian-Ming Bai, and Mitra L. Taheri. "Anisotropic radiation-induced segregation in 316L austenitic stainless steel with grain boundary character." *Acta Materialia* 67 (2014): 145-155.
20. Brons, J. G., J. A. Hardwick, H. A. Padilla II, K. Hattar, G. B. Thompson, and B. L. Boyce. "The role of copper twin boundaries in cryogenic indentation-induced grain growth." *Materials Science and Engineering: A* 592 (2014): 182-188.





R&D Topics of Interest

Current R&D Topics Under Investigation:

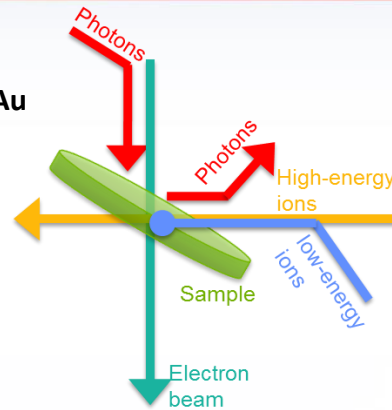
- Accelerated aging testing in harsh environments,
- Development and testing of surrogate fuels,
- Validation and refinement of pellet cladding interface properties,
- Validation and testing of current and advanced radiation detectors,
- Fundamentals of hydrogen isotope production, transportation, and storage,
- Exploration of radiation tolerant structural materials for cladding and duct systems,
- Determination of radiation tolerance in microelectronics,
- Predicting the performance margins in structural metals,
- Basic Science understanding of grain boundary stability under thermal, mechanical, and radiation environments,
- Exploring the microstructural stability in complex wear environments,



Summary & Future Directions

Sandia's I³TEM capabilities:

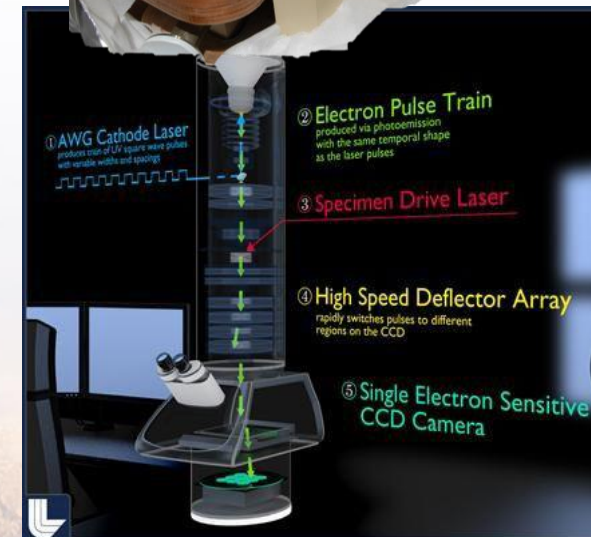
- *In situ* high energy ion irradiation from H to Au
- *In situ* gas implantation
- Heating up to 1,000 °C
- Cooling down to 77 K
- Quantitative and bulk straining
- Two-port microfluidic cell
- Gas flow/heating stage
- Electron tomography
- Precession Electron Diffraction



Sandia's I³TEM future capabilities being developed:

- In situ ion irradiation TEM in liquid or gas (currently capable)
- DTEM: Nanosecond resolution (laser optics being developed)
- Adding 1 MV NEC Tandem to complete the ion energy range
- Converting a 90° switching magnet to bend beams at 45° to obtain 88 MeV Au

Currently applying the current I³TEM capabilities to various material systems in sequential or combined harsh environmental conditions to elucidate the governing mechanisms and inform computational models





Technical Back-up Slides



Current Status of the *In situ* TEM Beamline

Collaborators: D.L. Buller & J.A. Scott



Beam burn from
14 MeV Si

Gas Heating TEM
Stage Controls

Microfluidic TEM
Stage Controls

Double tilt stage
needs to tilt only
12°

10 kV
Colutron

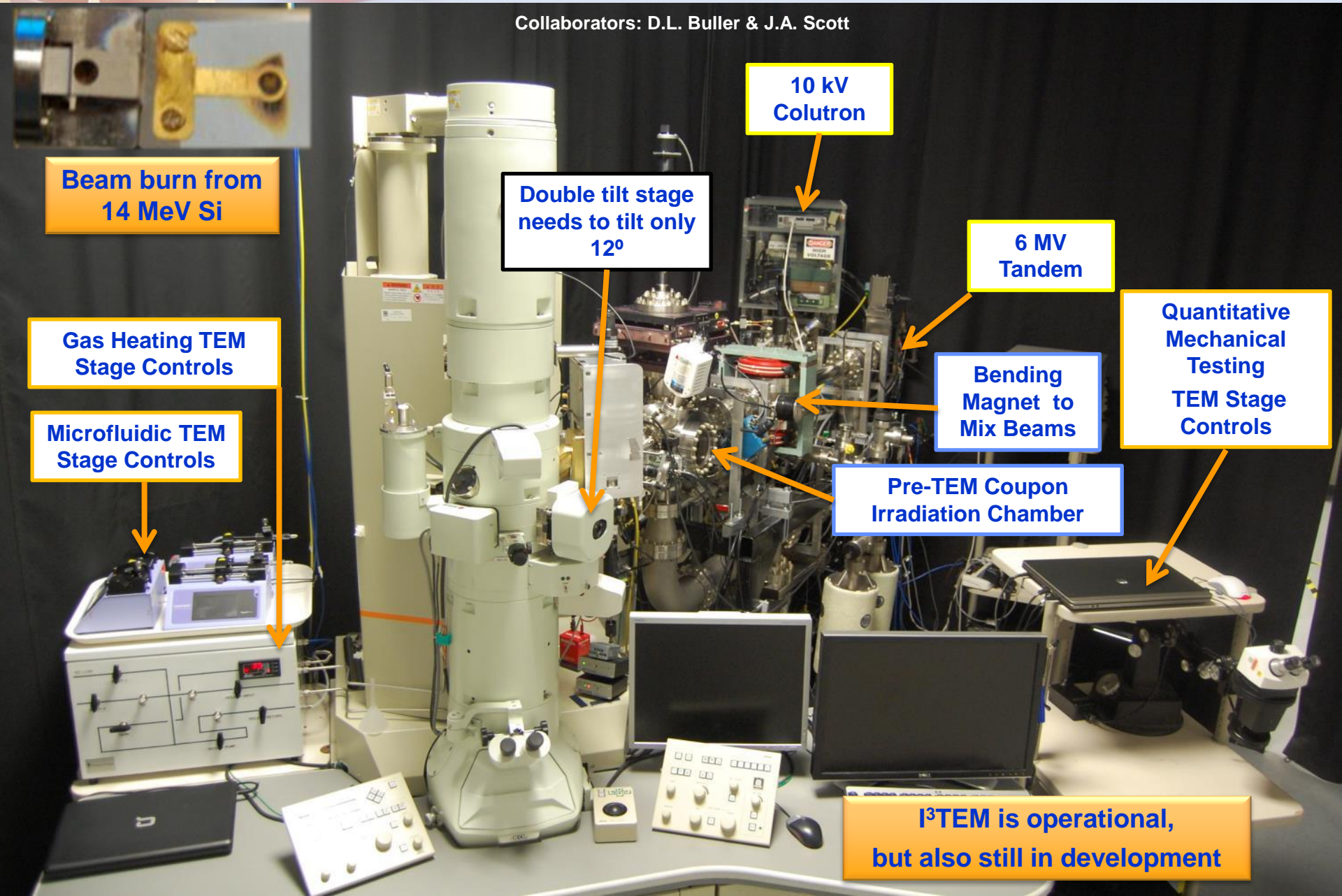
6 MV
Tandem

Bending
Magnet to
Mix Beams

Quantitative
Mechanical
Testing
TEM Stage
Controls

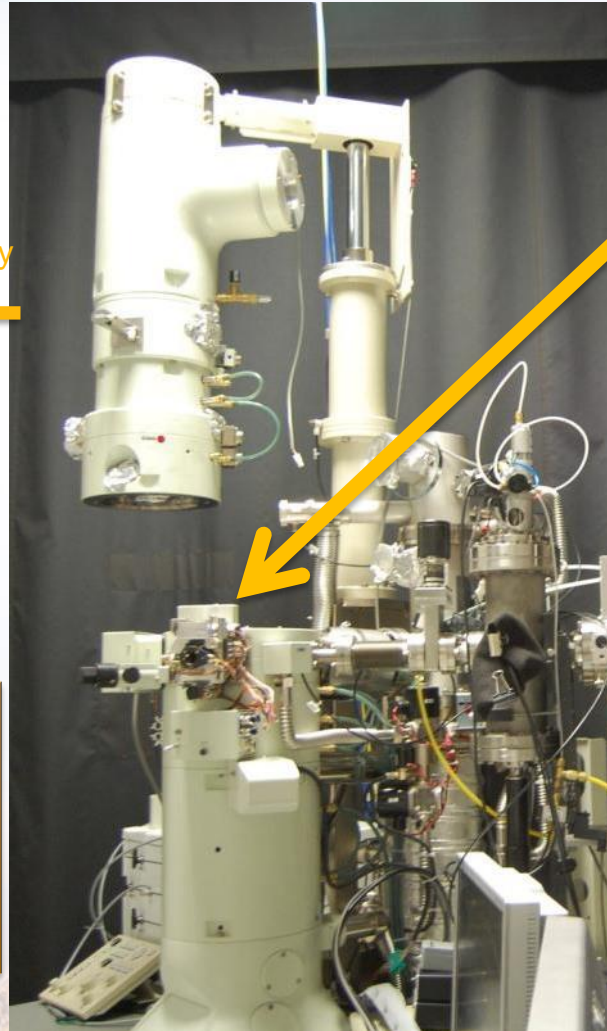
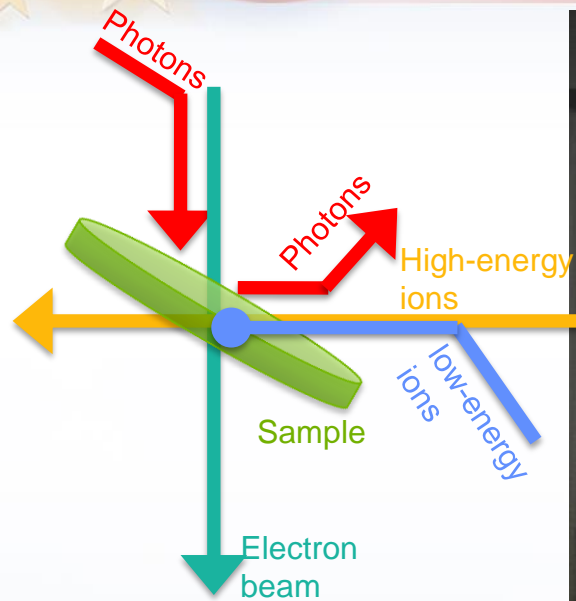
Pre-TEM Coupon
Irradiation Chamber

I³TEM is operational,
but also still in development



In situ TEM Luminescence

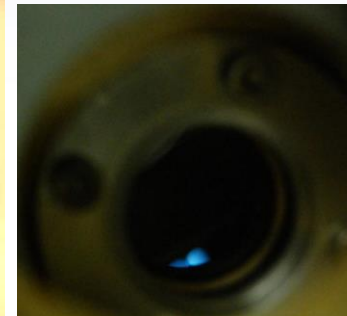
Collaborators: D. Masiel, D. Buller, and C. Chisholm



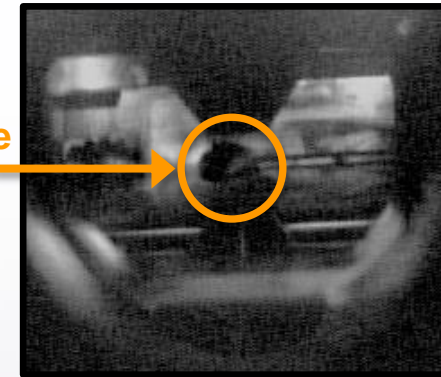
Optical Mirror in TEM



First IBIL in TEM



Sample



Two optical ports were added to the I³TEM already containing an electron beam and two ion beams, which permits *in situ* TEM luminescence studies

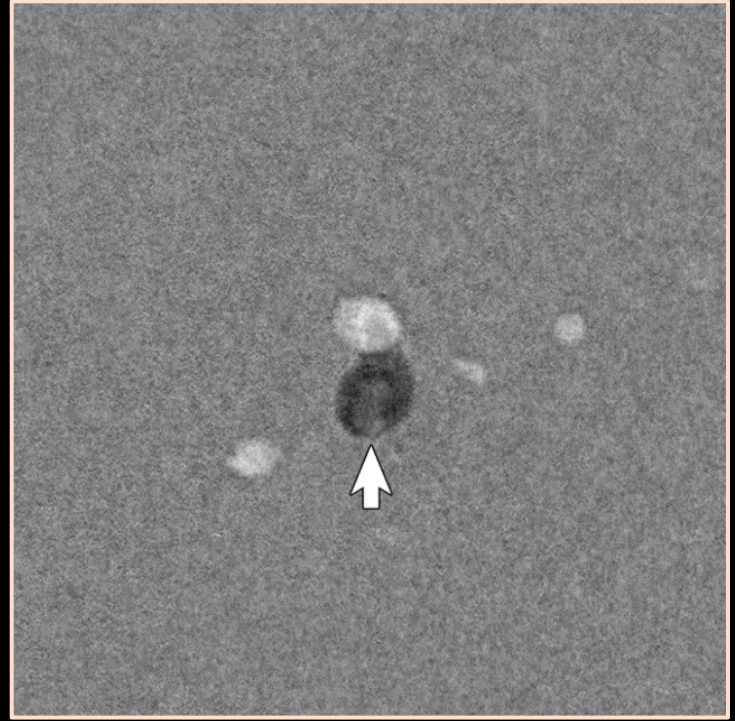
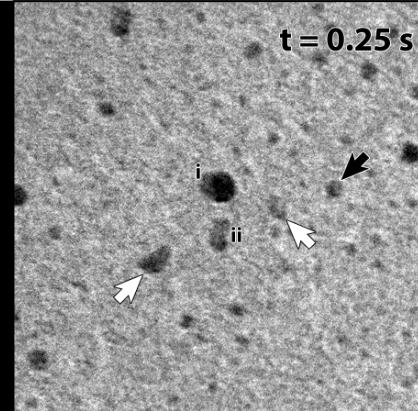
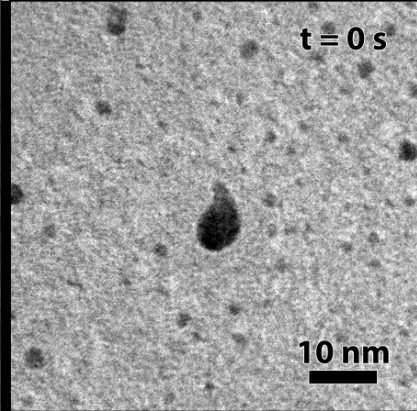
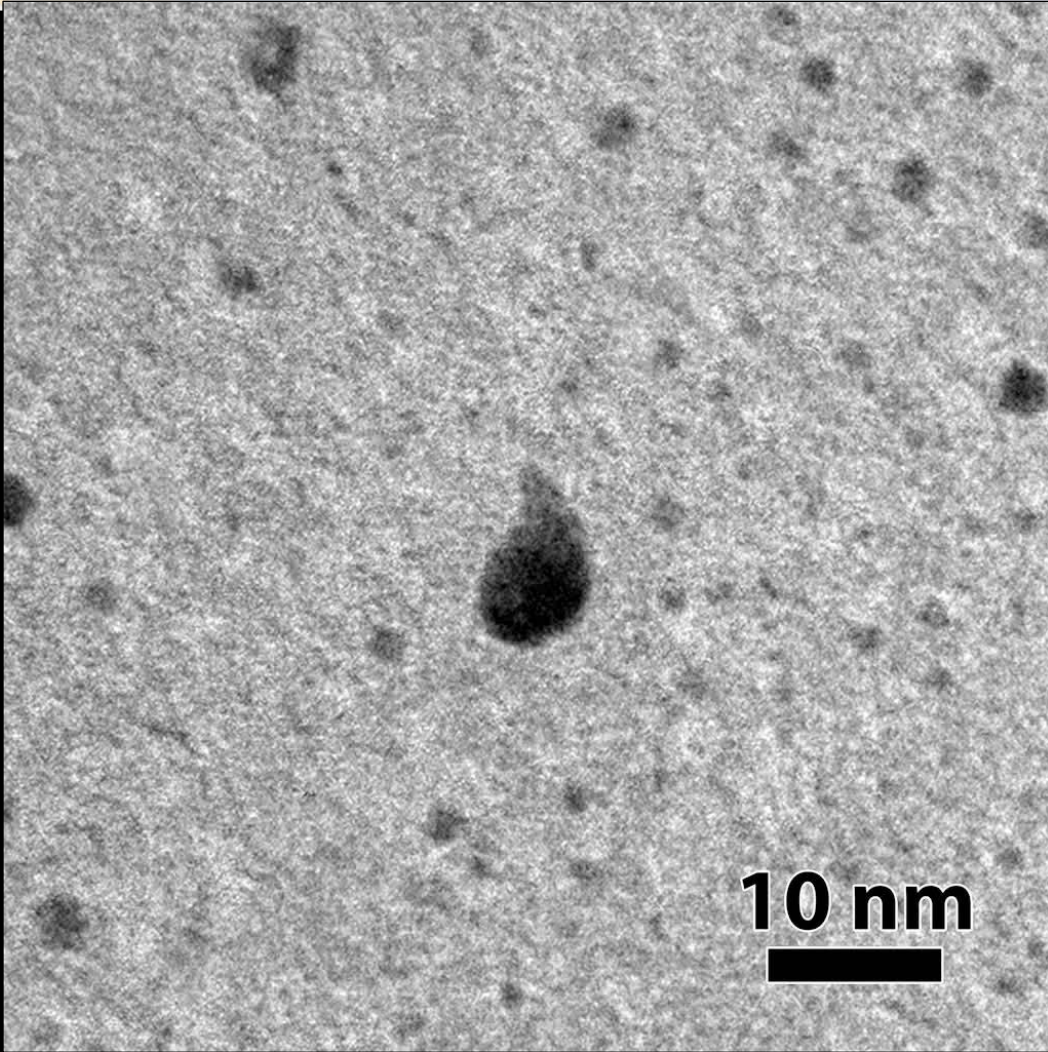
Optical Pathway in an I³TEM

- Angled mirror with bore hole for the electron path was installed above the polepiece
- Another mirror is located just above the ion beams in the beamline
- Two perspectives of the sample are possible
- Permits *in situ* IBIL and CL.



Single Ion Strikes: 46 keV Au¹⁺ ions into 5 nm Au nanoparticles

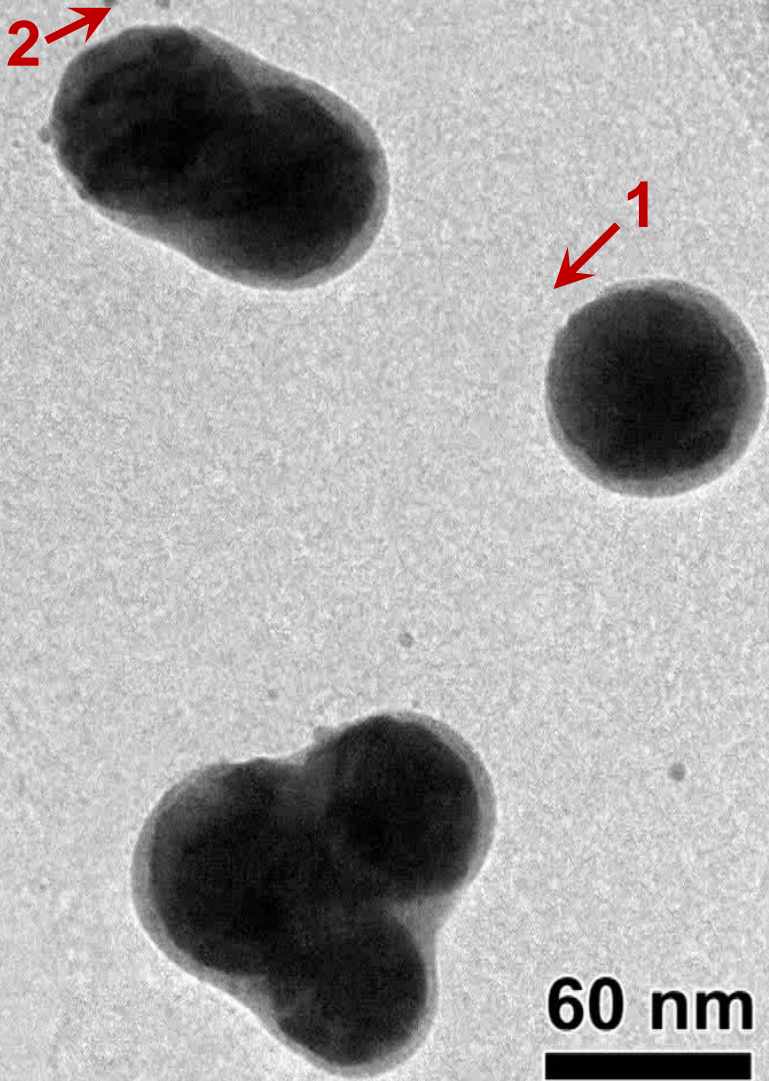
Collaborator: D.C. Bufford



Single Ion Strikes:

2.8 MeV Au⁴⁺ ions into 60 nm Au nanoparticles

Collaborator: D.C. Bufford



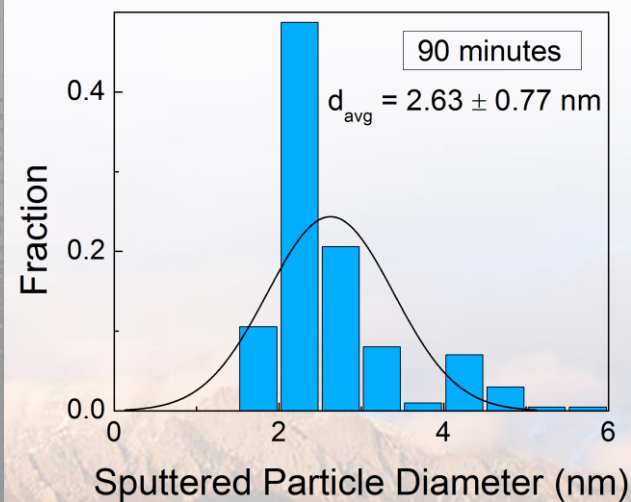
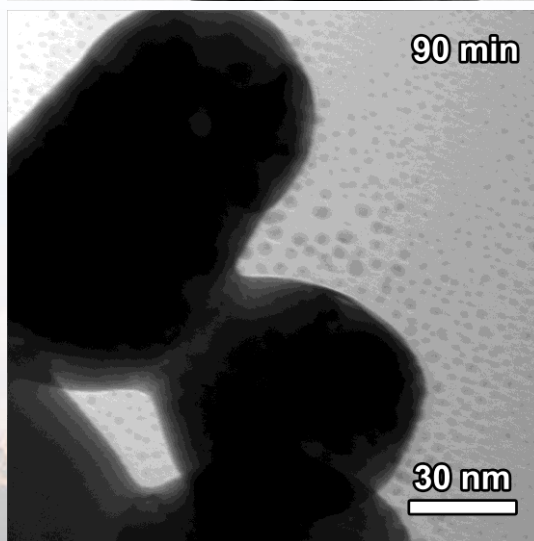
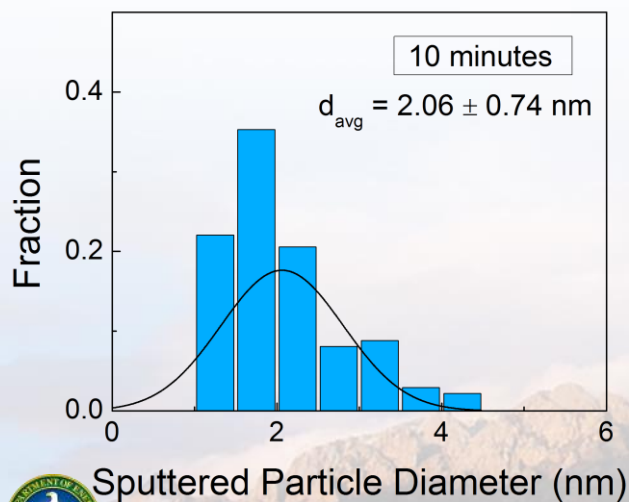
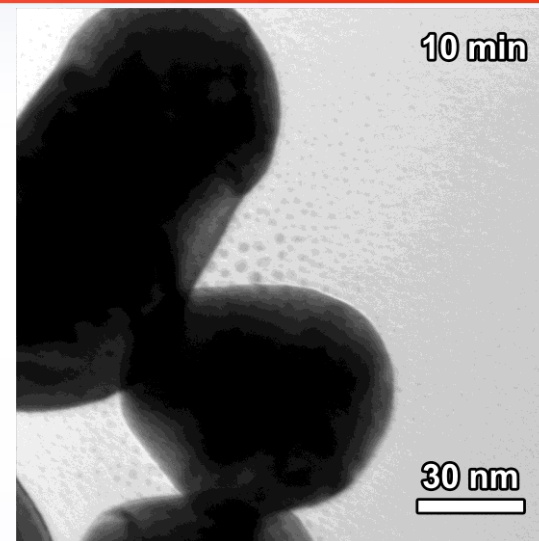
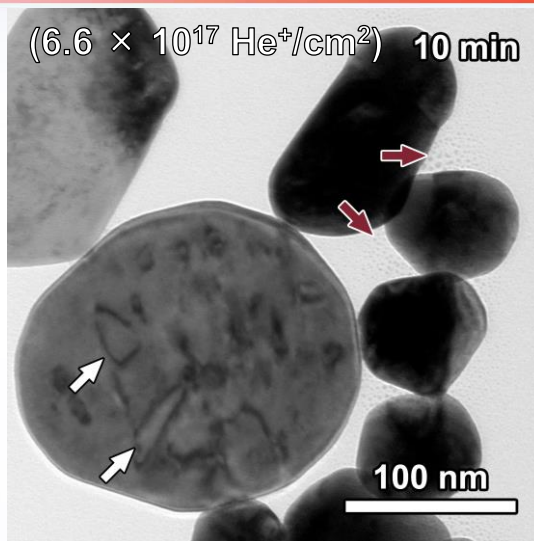
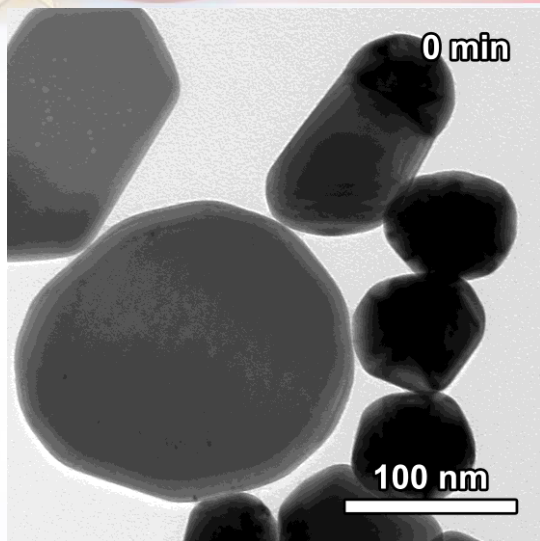
- 2.8 MeV Au⁴⁺ ions into 60 nm diameter Au nanoparticles
- 100 kx magnification
- Nanoscale filaments created by individual ions

The permanent and transient structures resulting from single ion strikes can be directly observed

Video playback at 2x real time.

Formation of Dislocation Loops & Sputtered Particles due to He implantation

Collaborators: D.C. Bufford, S.H. Pratt & T.J. Boyle



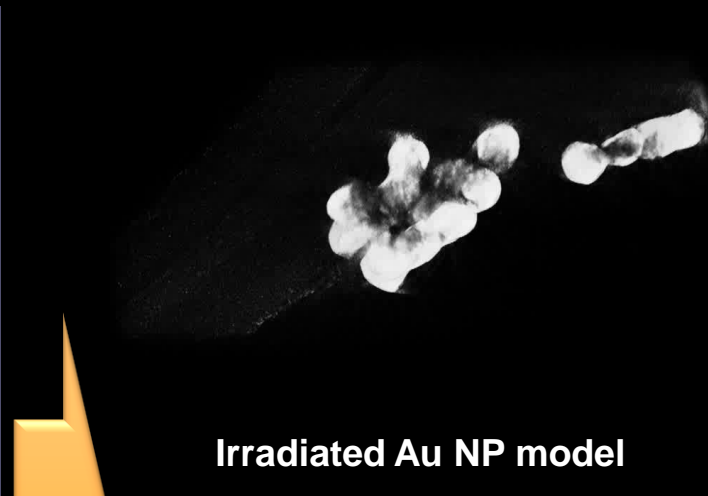
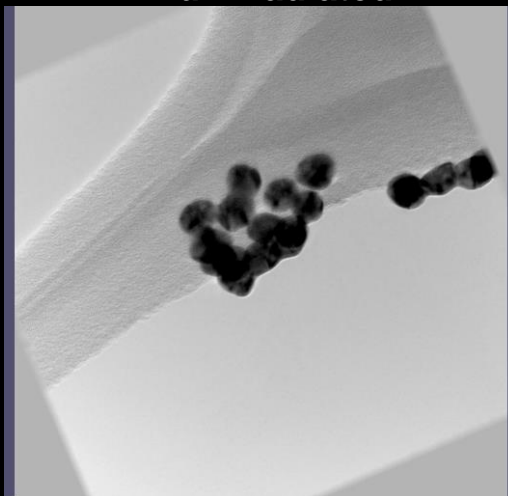
Electron Tomography Provides 3D Insight

Collaborators: S.H. Pratt & T.J. Boyle

In situ Ion Irradiation TEM (I³TEM)

Aligned Au NP tilt series -
unirradiated

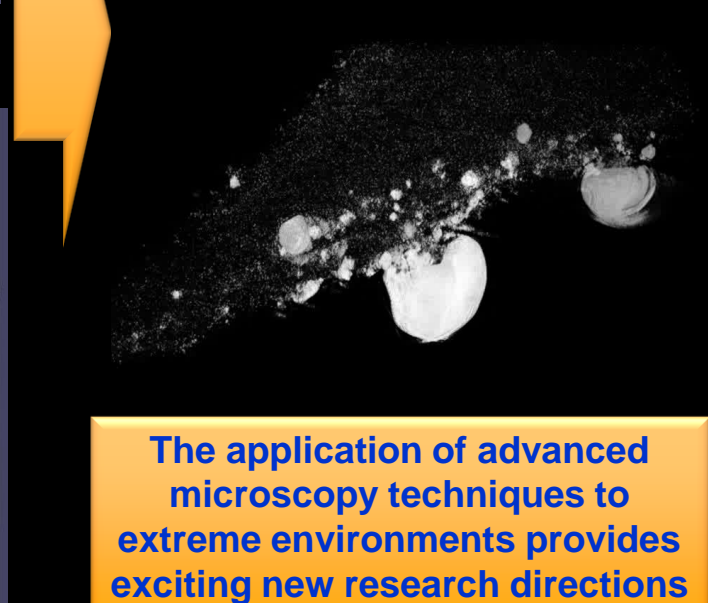
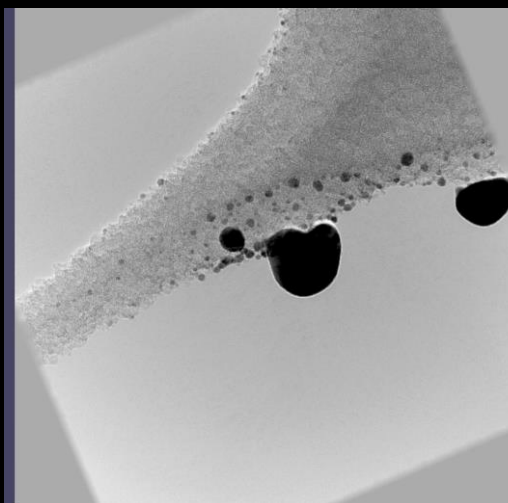
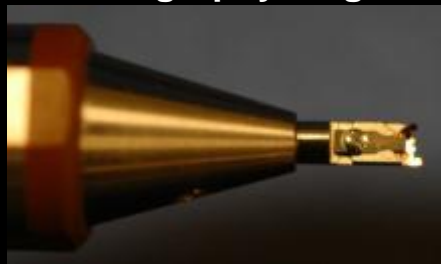
Unirradiated Au NP model



Hummingbird
tomography stage

Aligned Au NP tilt series -
irradiated

Irradiated Au NP model

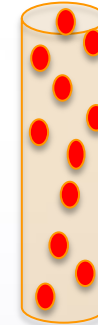
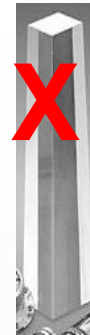


The application of advanced
microscopy techniques to
extreme environments provides
exciting new research directions

Need for Advanced Scintillators

Goal: determine the utility of size-selected high Z transition metal tungstates (MWO_4) as novel scintillating materials

- Characterize novel nanotungstates and their radiation hardness
- Investigate size effects on radiation interaction
- Elucidate radiation interaction mechanisms

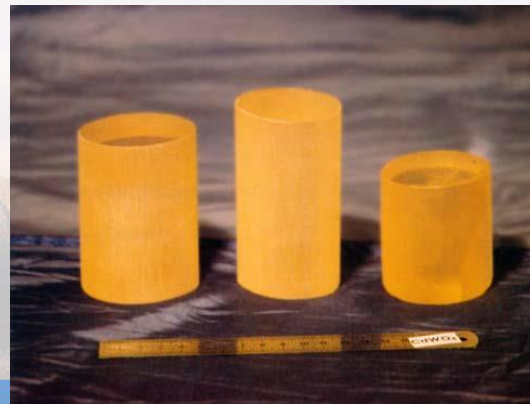


High Z Nanocomposite



Single crystal scintillators suffer from : low luminosity, volume restrictions, mechanical ruggedness, and chemical instability

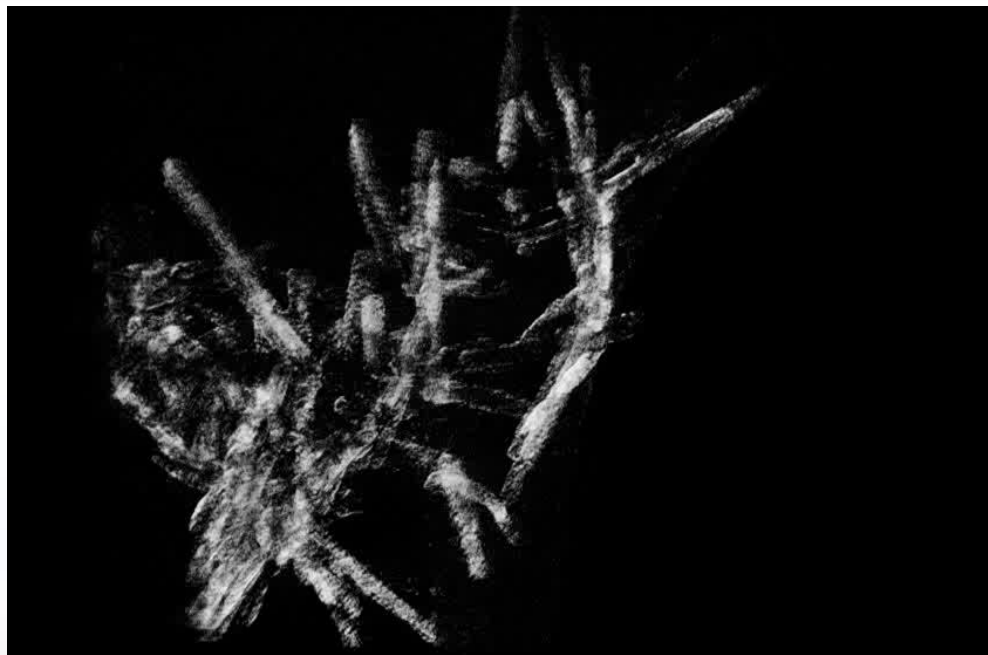
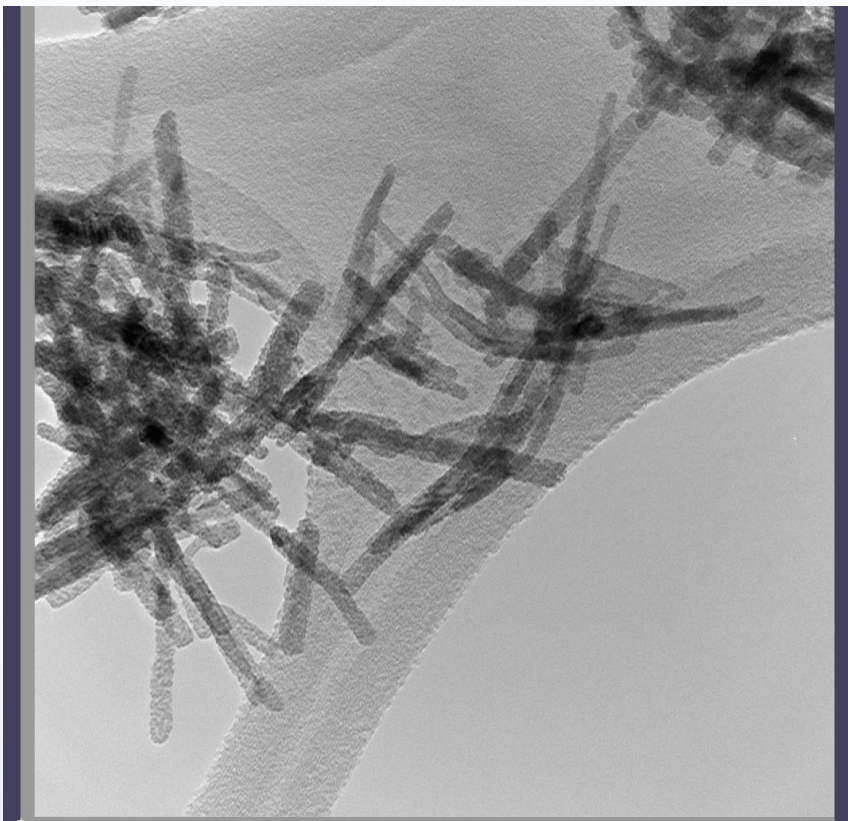
$CdWO_4$ single crystal scintillators



Nanorods of $CdWO_4$
Improvement in scintillation properties?

New scintillating materials are critically needed to protect the United States.

Tomographic Reconstruction of CdWO₄

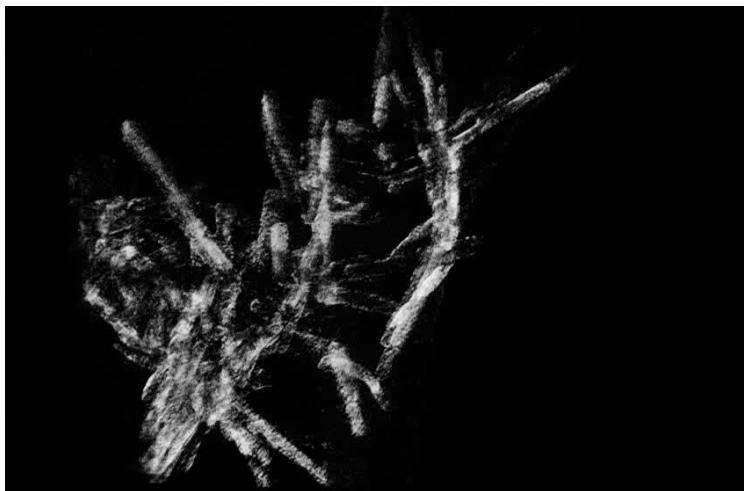


Recent advancements in TEM control and reconstruction software permit collection and production of 3D model of the “transmission” micrograph.

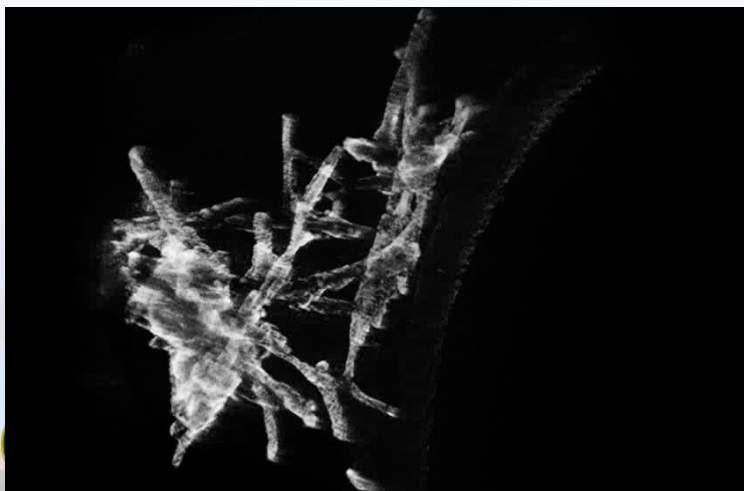


Tomography of CdWO_4 irradiated with 30 nA of 3 MeV Cu^{3+}

Unirradiated



5 minutes



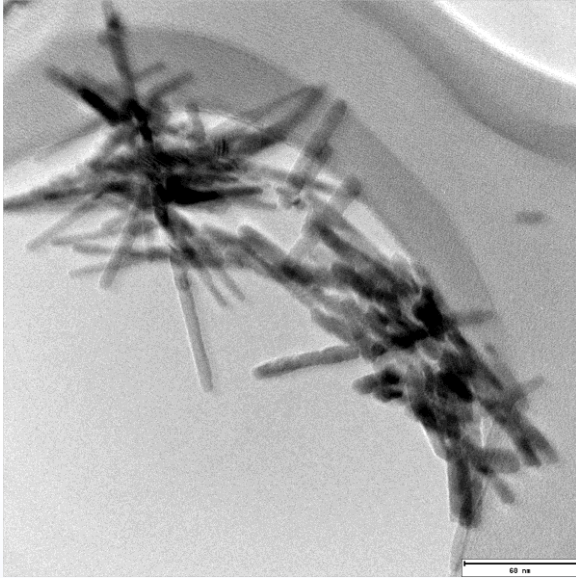
30 minutes



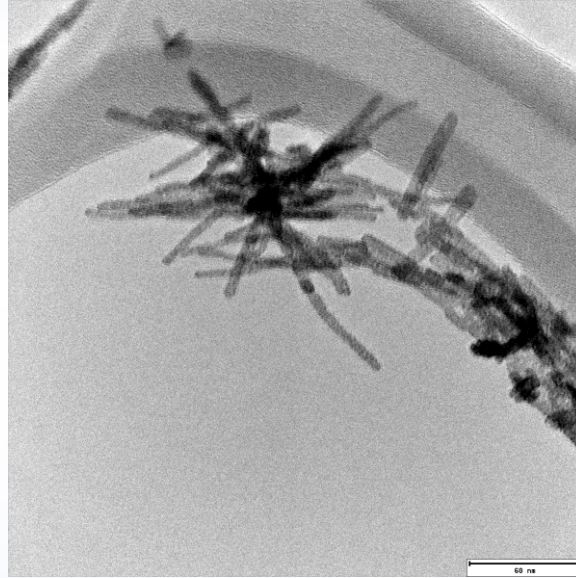
Tilt series were collected after each dose of irradiation resulting in 4D tomography with 3D reconstructions showing radiation damage over time.

Current work: *In situ* Proton Irradiation as First Order Simulation of Neutrons

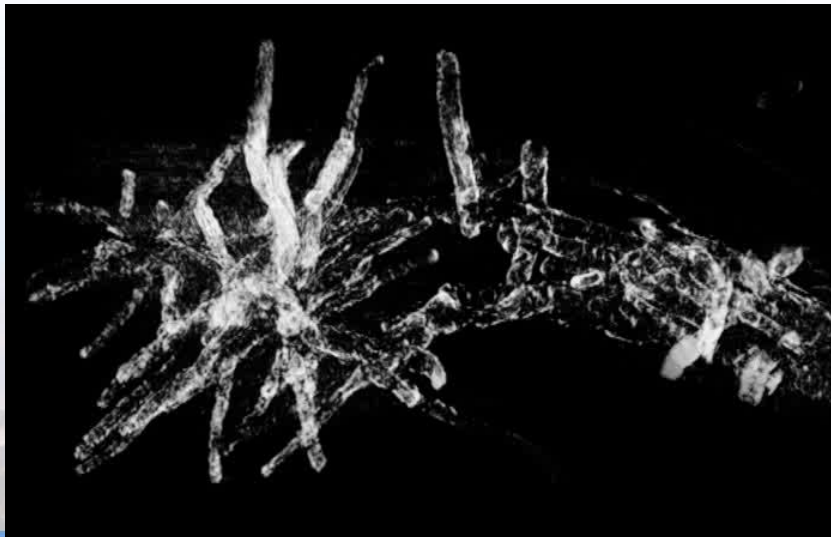
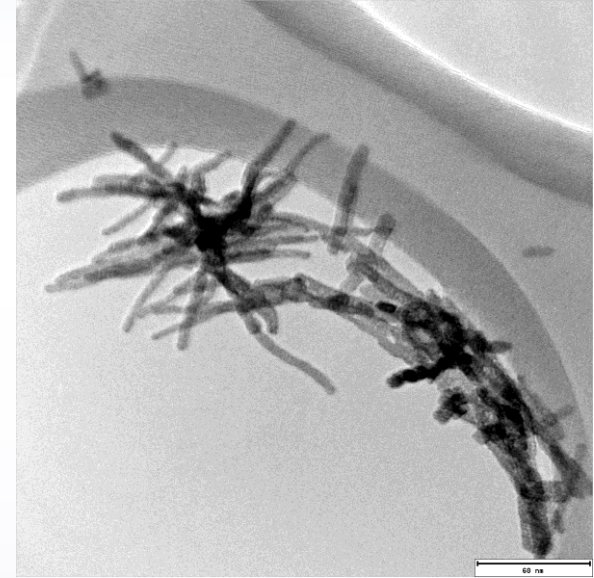
Unirradiated



15 minutes



60 minutes

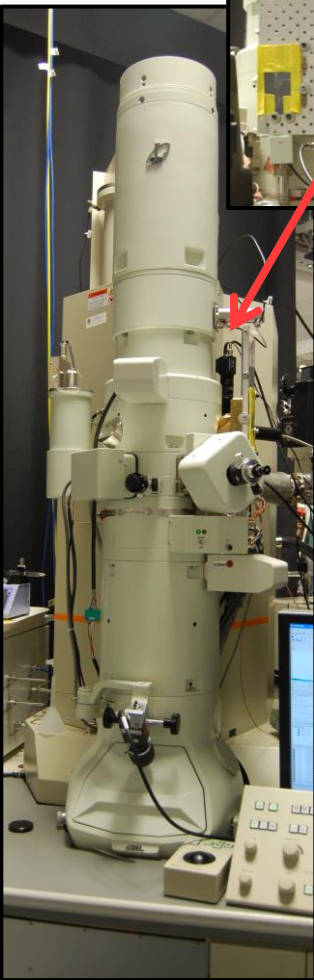
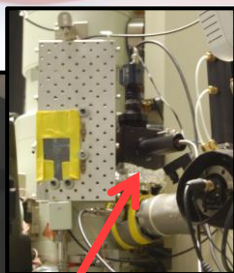


160 nA of 2.5 MeV H⁺ used to simulate neutron radiation shows less change. Results suggest good radiation hardness for tungstate nanorod-composite scintillators.

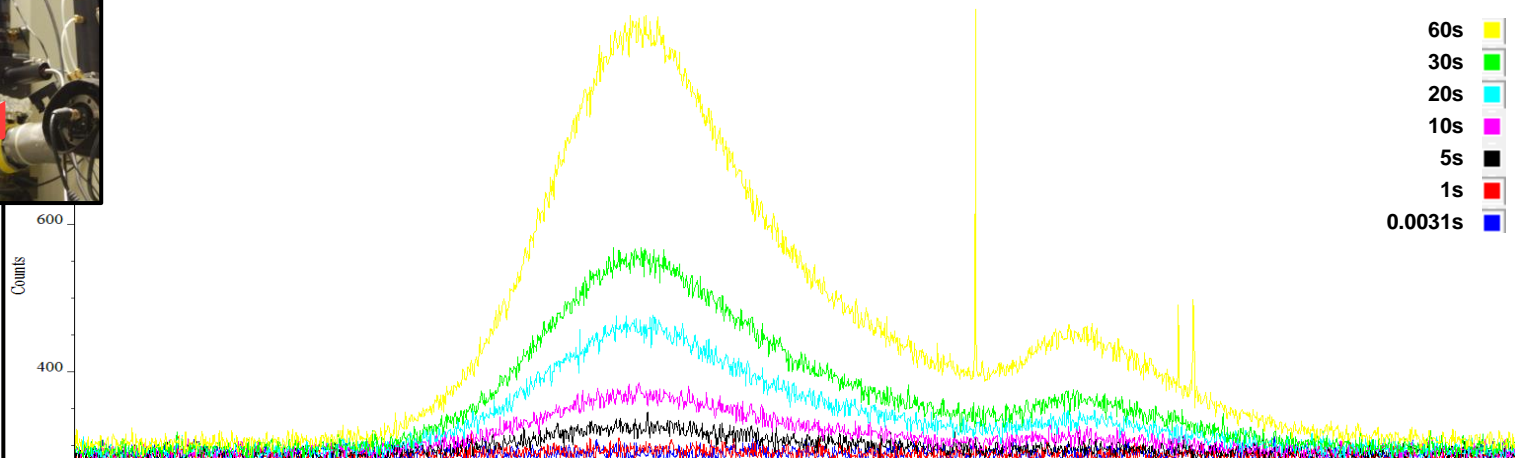


Future Direction: *In situ* TEM Ion beam Induced Luminescence (IBIL)

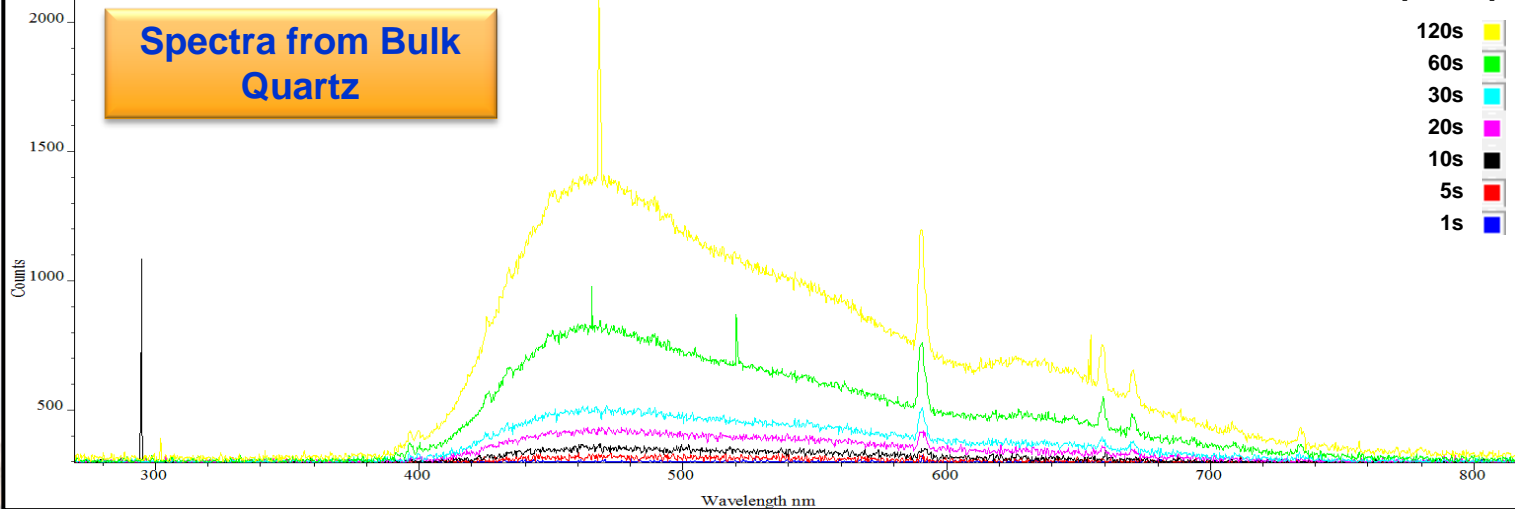
Collaborator: J. Gutierrez-Kolar



Cathodoluminescence (CL)

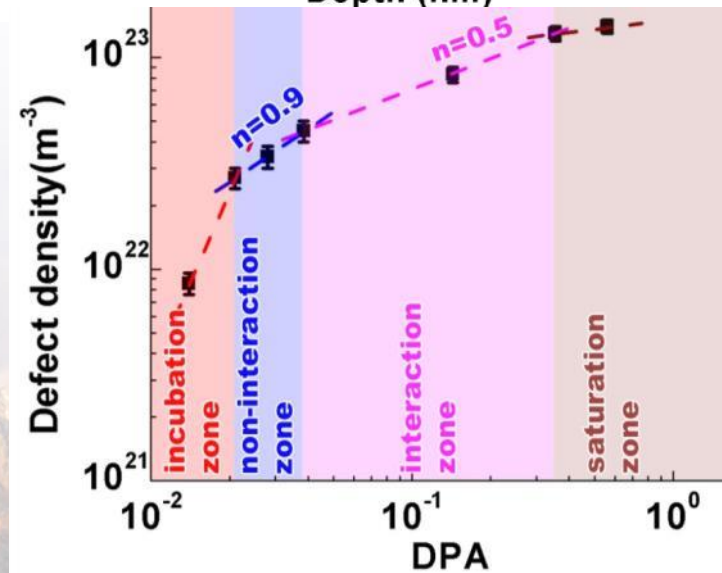
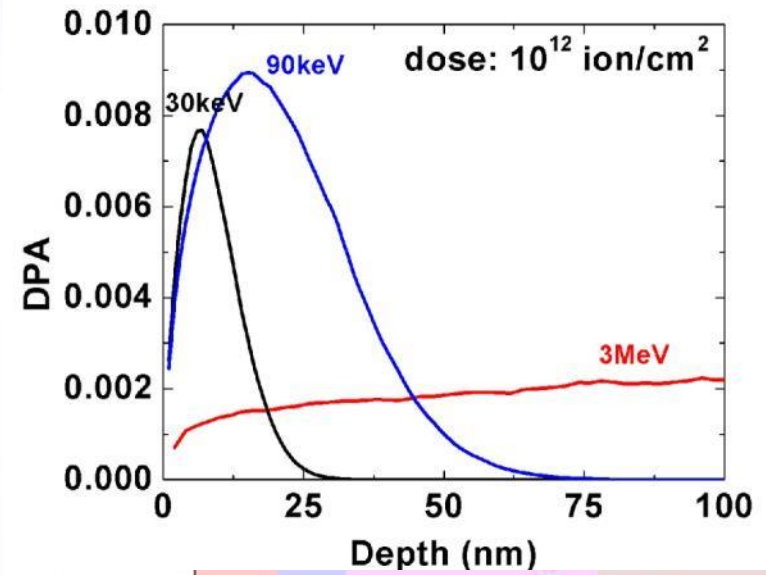
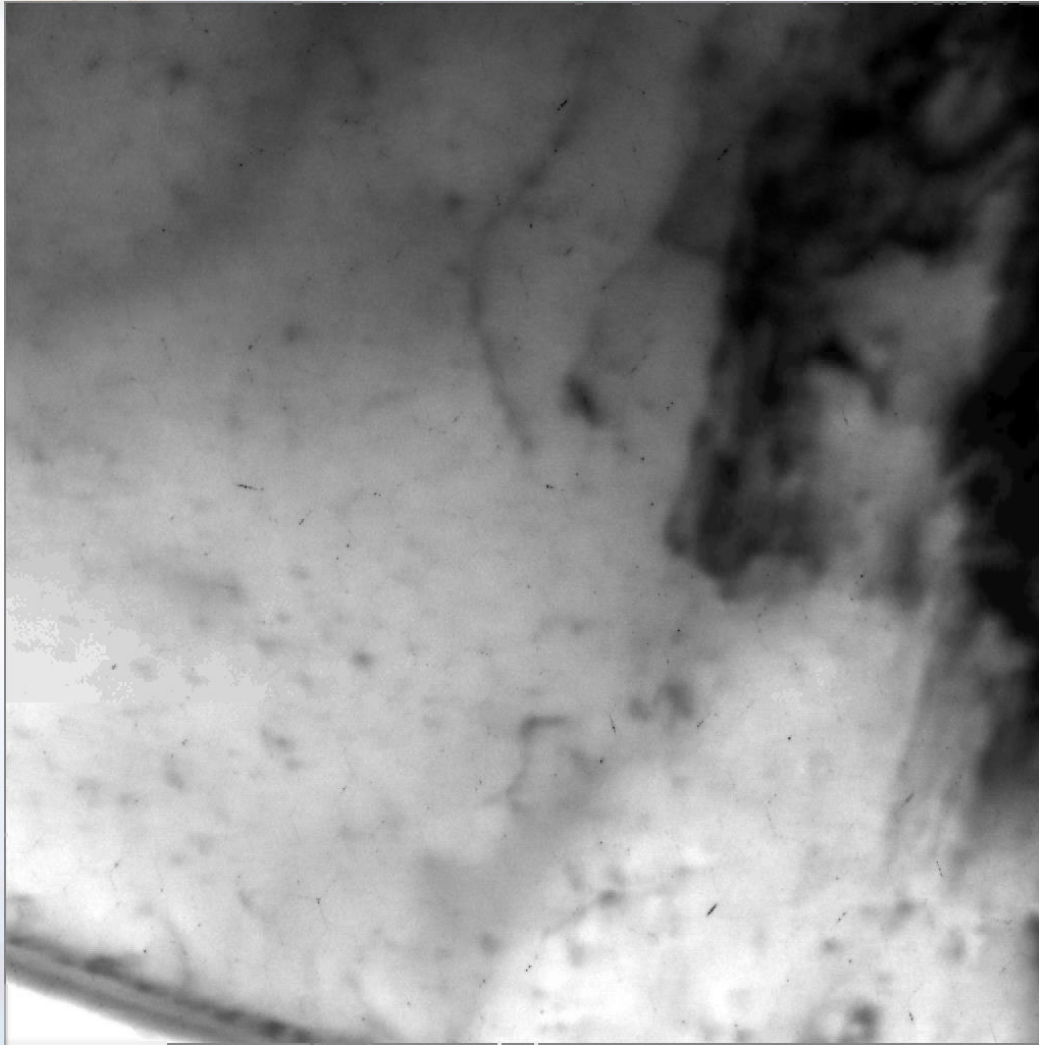


Ion Beam Induced Luminescence (IBIL)



Significant optimization is still needed; potential is promising

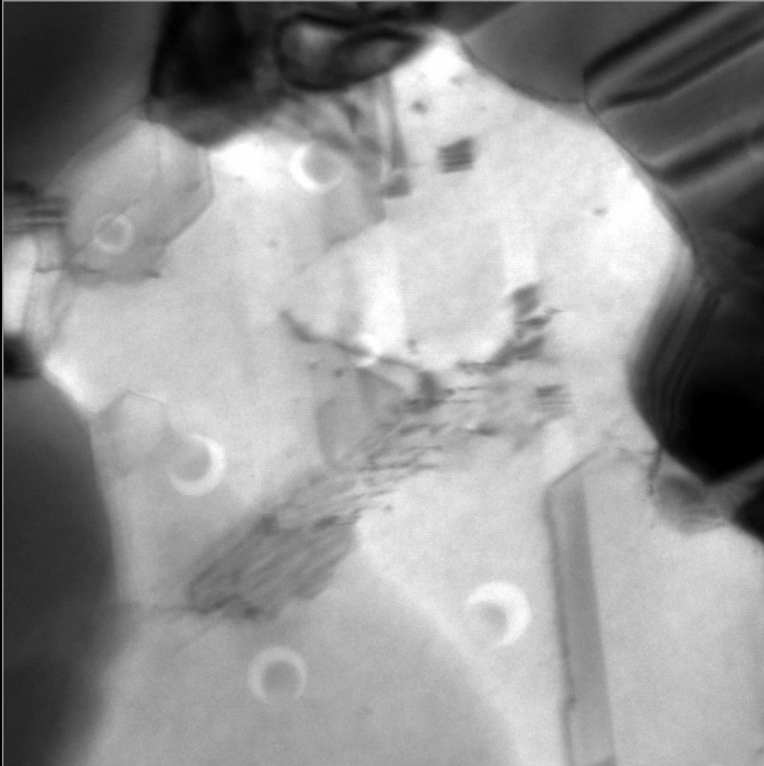
Quantifying Defect Evolution



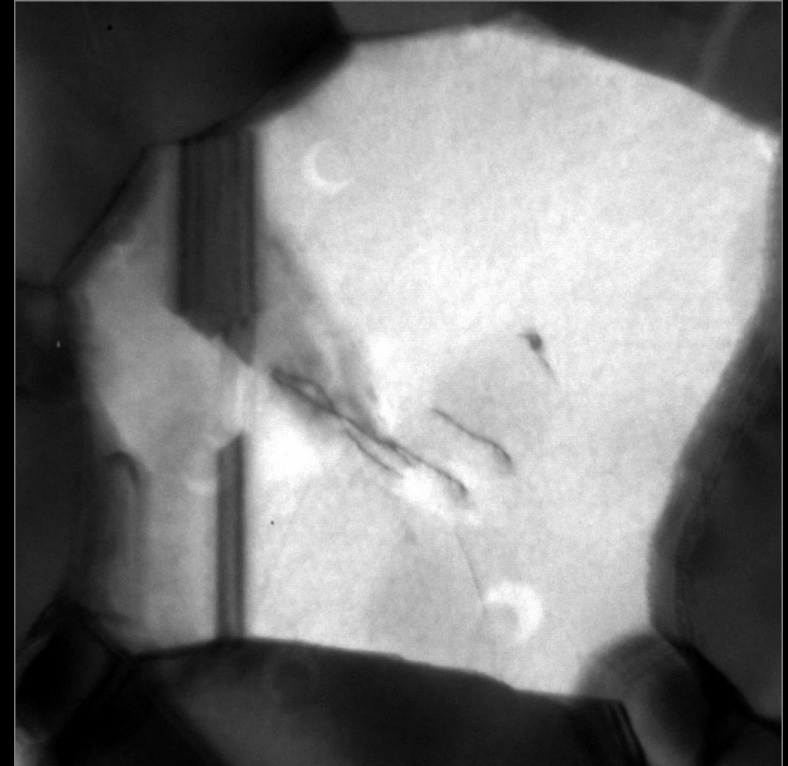
Dose Rate Effects

Collaborators: C. Chisholm , P. Hosemann, & A. Minor

7.9×10^9 ions/cm²/s



6.7×10^7 ions/cm²/s

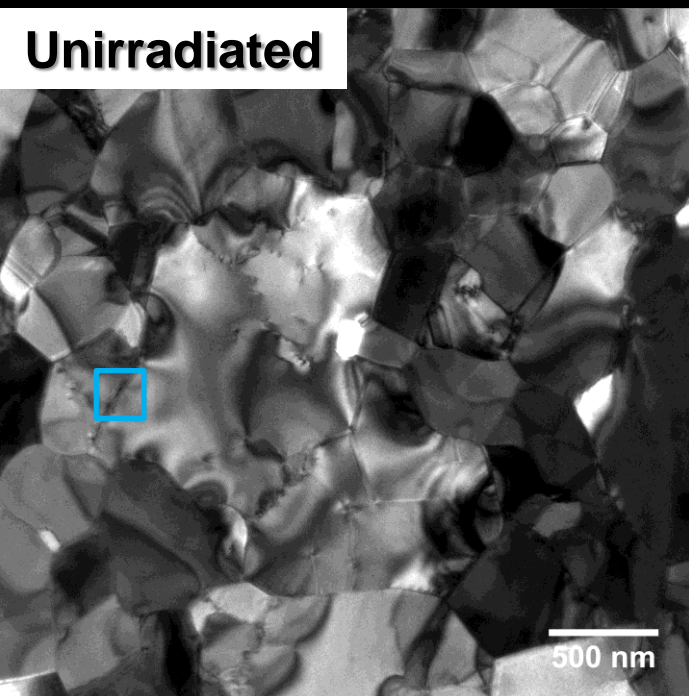


VS

Improved vibrational and ion beam stability permits us to work at 120kx or higher permitting imaging of single cascade events

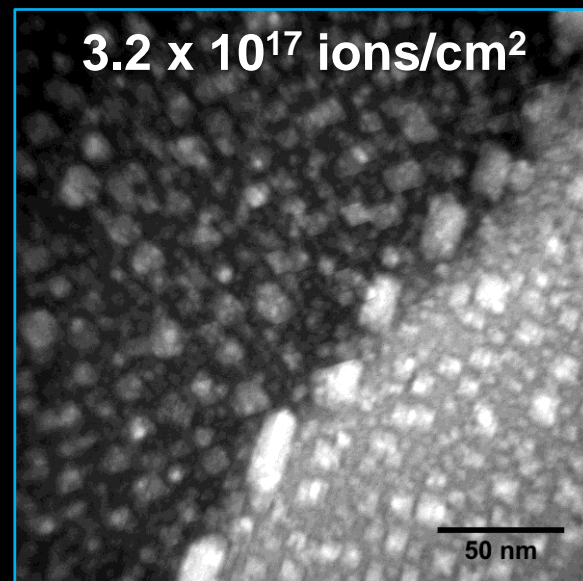
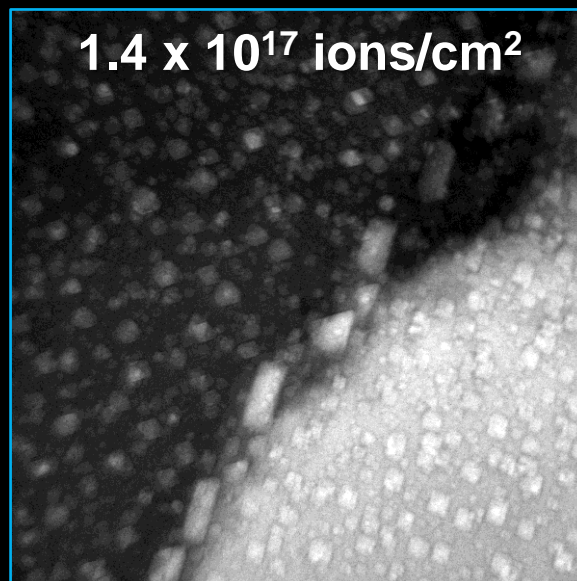
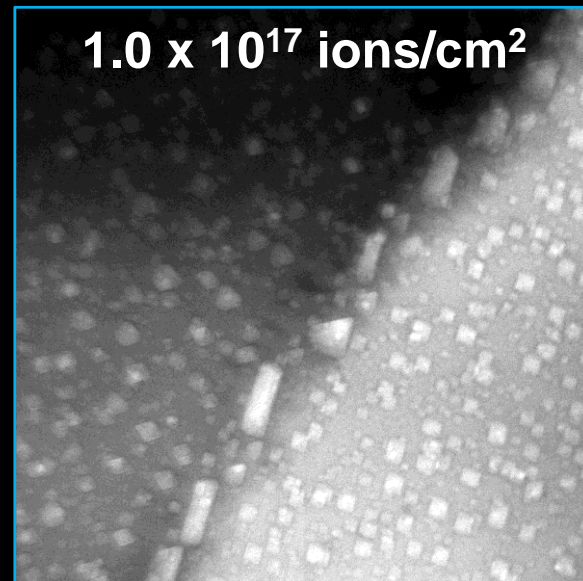
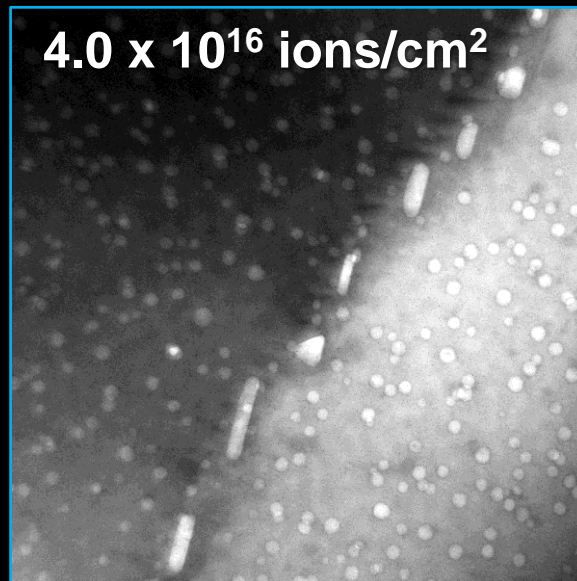
In situ Implantation

Collaborators: C. Chisholm & A. Minor



**Gold thin-film implanted
with 10keV He²⁺**

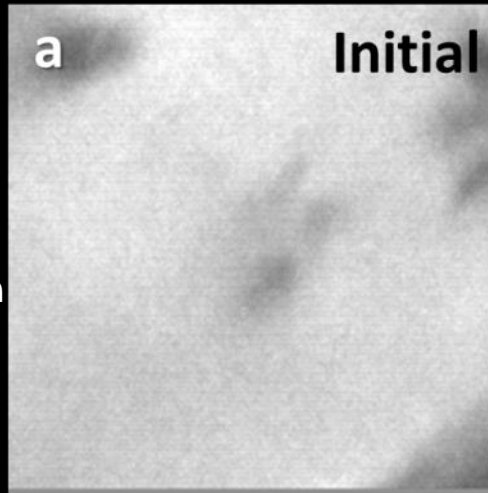
**Result: porous
microstructure**



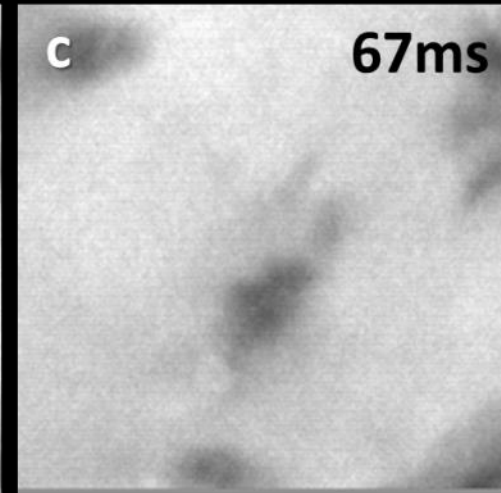
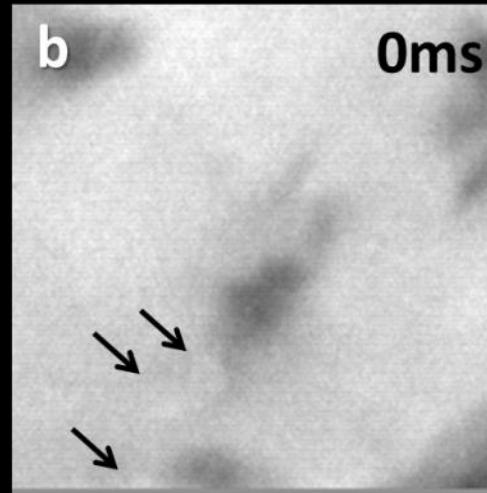
Single Ion Strikes During Concurrent Irradiation: Nucleation of Helium Cavities

Collaborators: C. Chisholm & A. Minor

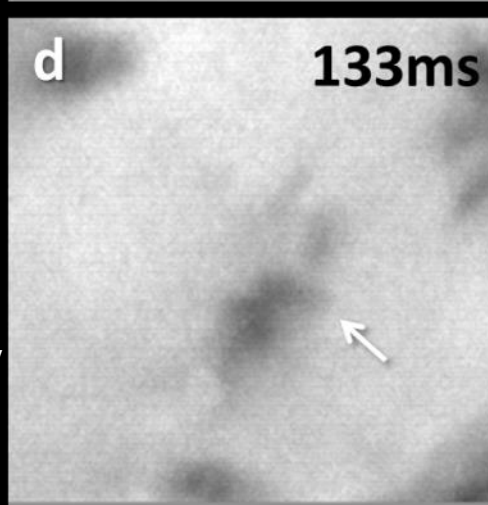
a) Initial microstructure



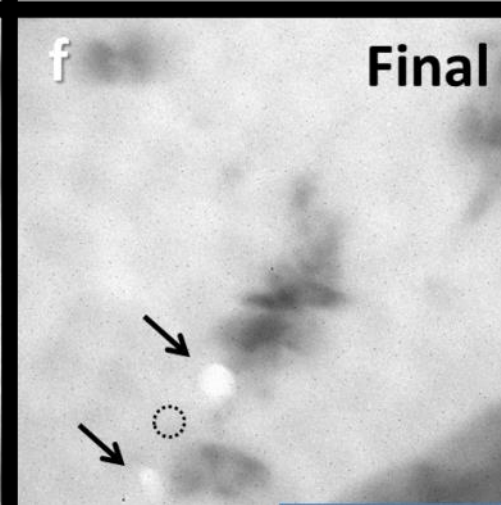
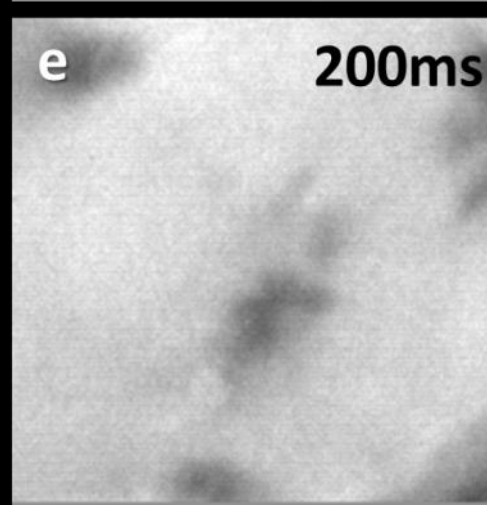
b) Cascade: Creation of dislocation loops, vacancy clusters, and three cavities



d) Cascade damage still evolving



e) Apparent stability



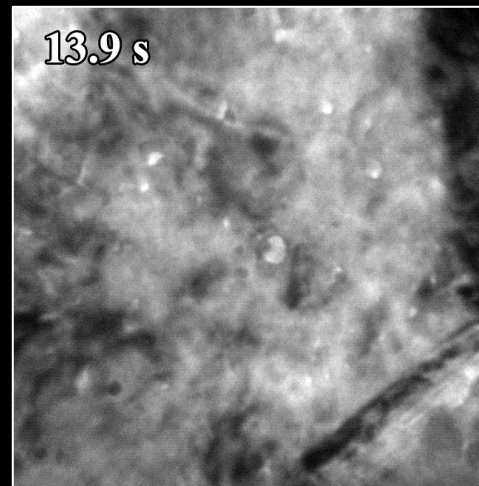
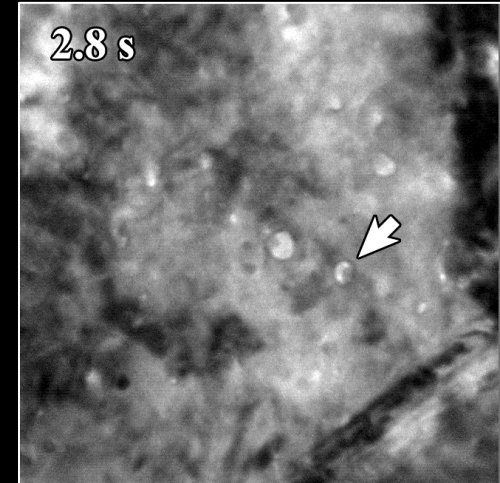
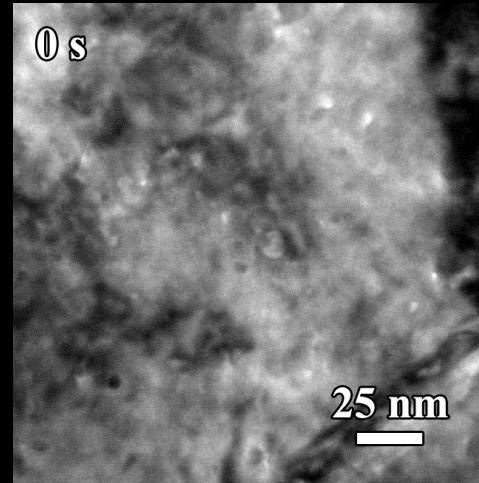
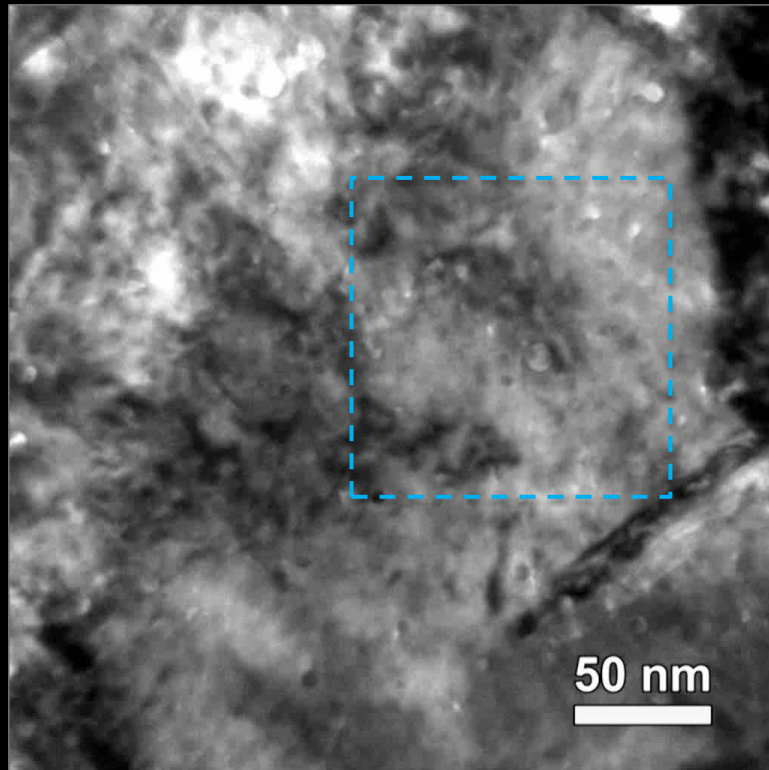
f) Final microstructure: Only two remaining cavities

Simultaneous *In situ* TEM Triple Beam:

2.8 MeV Au⁴⁺ + 10 keV He⁺ / D₂⁺

Collaborator: D.C. Bufford

Video playback speed x1.5.



In-situ triple beam He, D₂, and Au beam irradiation has been demonstrated on Sandia's I³TEM!

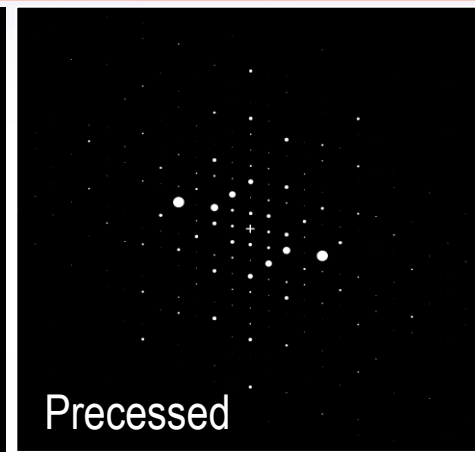
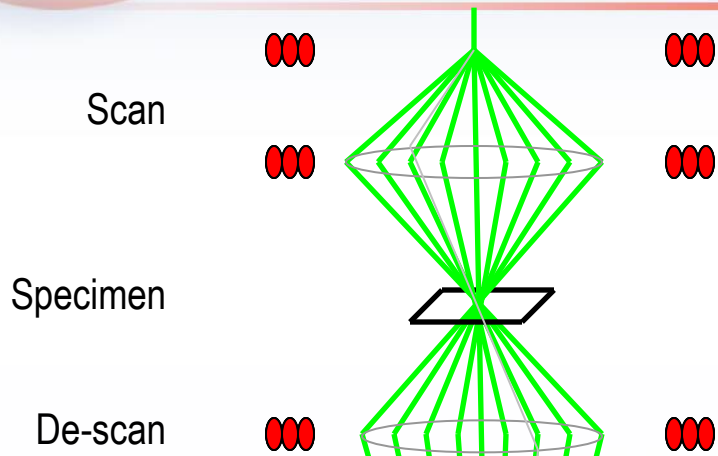
Intensive work is still needed to understand the defect structure evolution that has been observed.

■ Approximate fluence:

- Au 1.2×10^{13} ions/cm²
- He 1.3×10^{15} ions/cm²
- D 2.2×10^{15} ions/cm²

■ Cavity nucleation and disappearance

Precession Electron Diffraction Microscopy



Unprocessed

Processed

(Diffracted amplitudes)

Advantages:

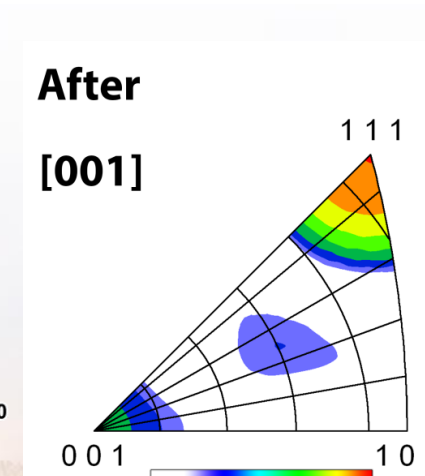
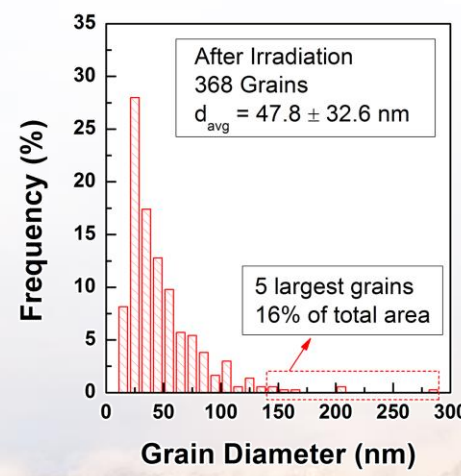
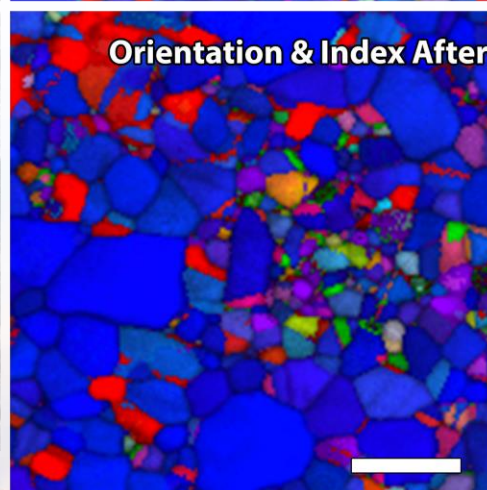
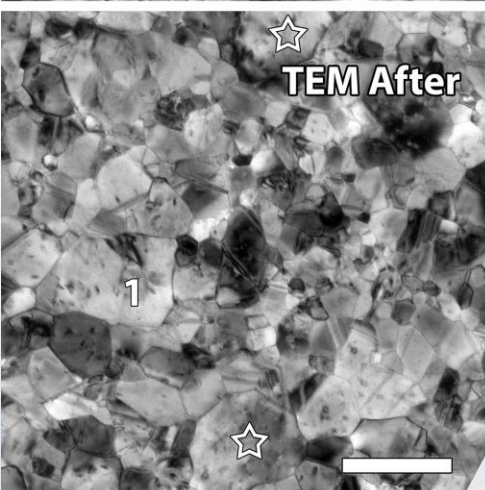
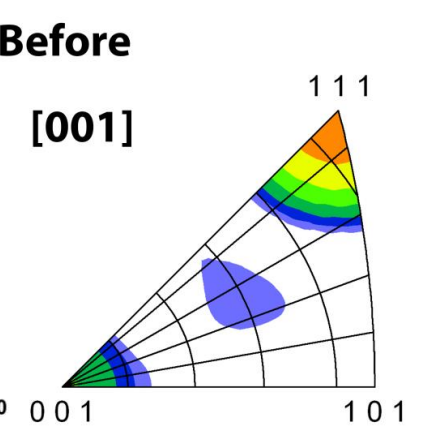
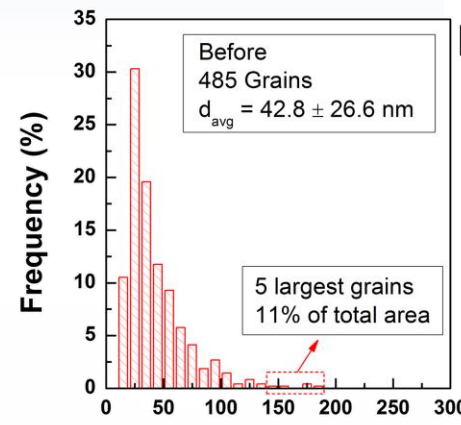
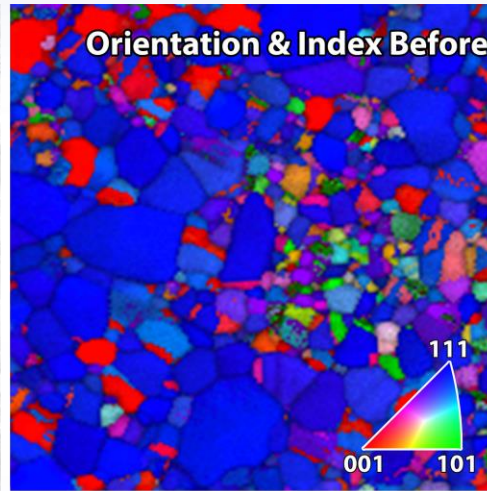
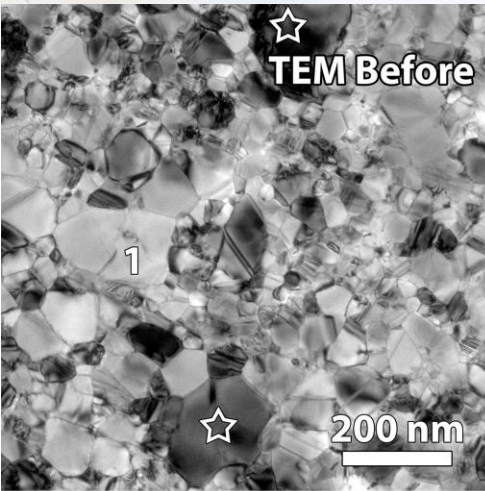
- < 10 nm spatial resolution
- Near kinematical electron diffraction
- Symmetry ambiguities are resolved
- Fast and automated acquisition
 - ≈ 200 grains in 15 min.
 - Angular resolution $\approx 1^\circ$



Sandia National Laboratories

Quantifying Stability of Nanocrystalline Au during 10 MeV Si Ion Irradiation

Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles

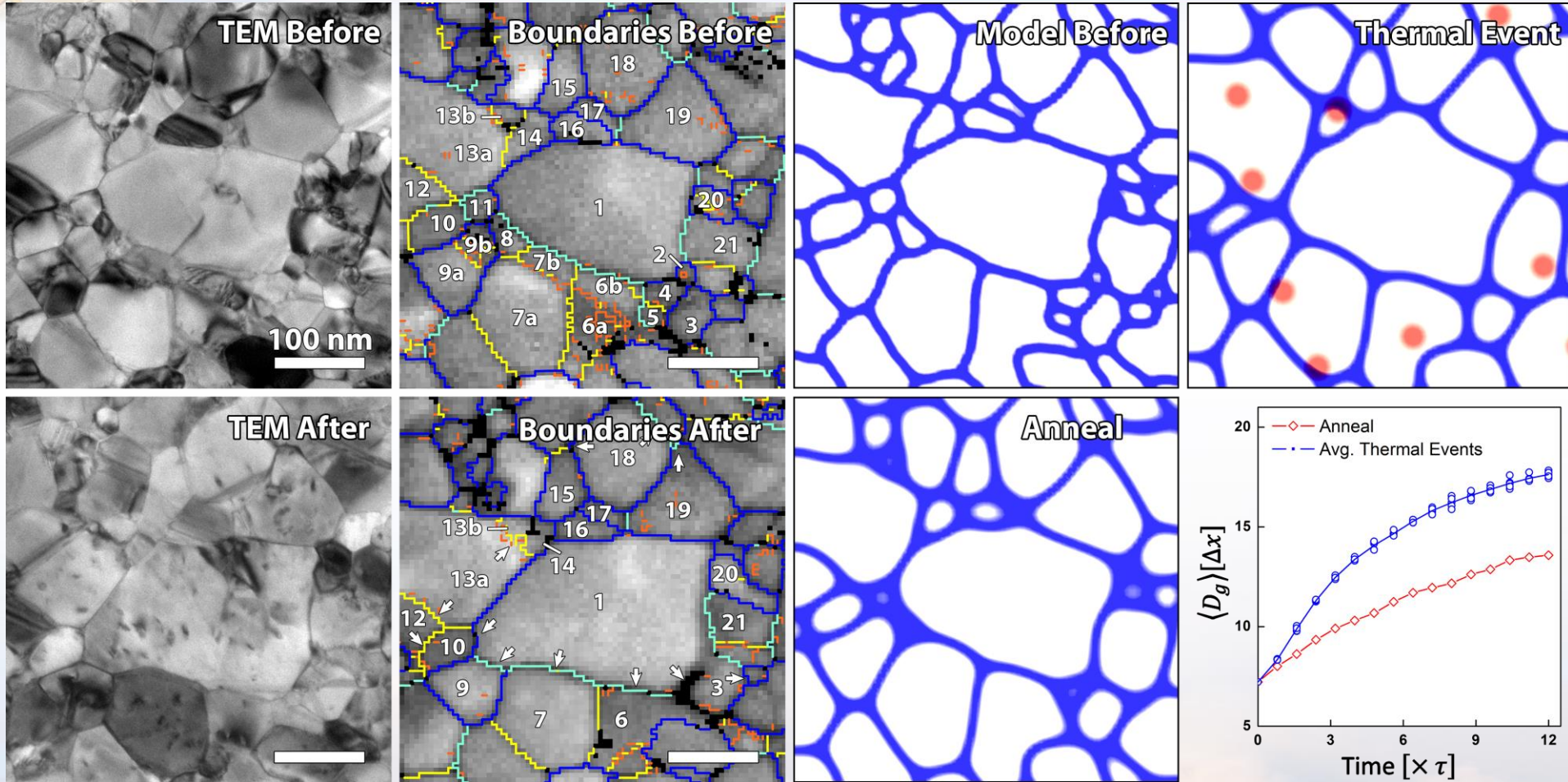


Any texture or grain boundary evolution can be directly observed and quantified



Direct Comparison to Mesoscale Modeling

Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles

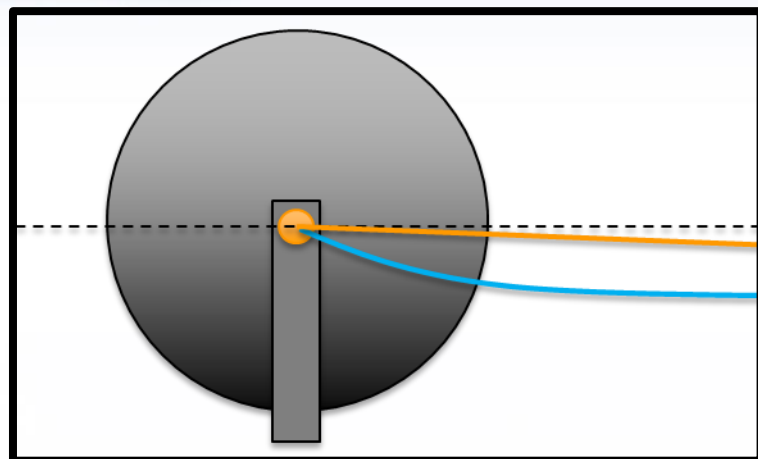


Because of the matching length scale, the initial microstructure can serve as direct input to either MD or mesoscale models & subsequent structural evolution can be directly compared.

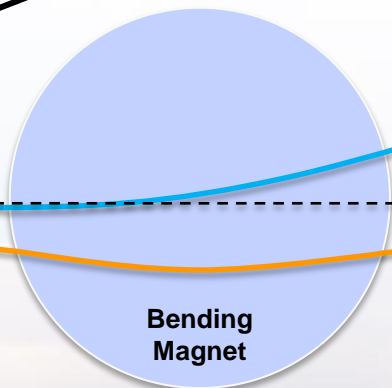
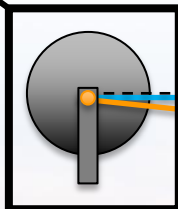


Modeling Beam Mixing and Deflection Necessary to Develop a *In situ* Triple Beam Facility

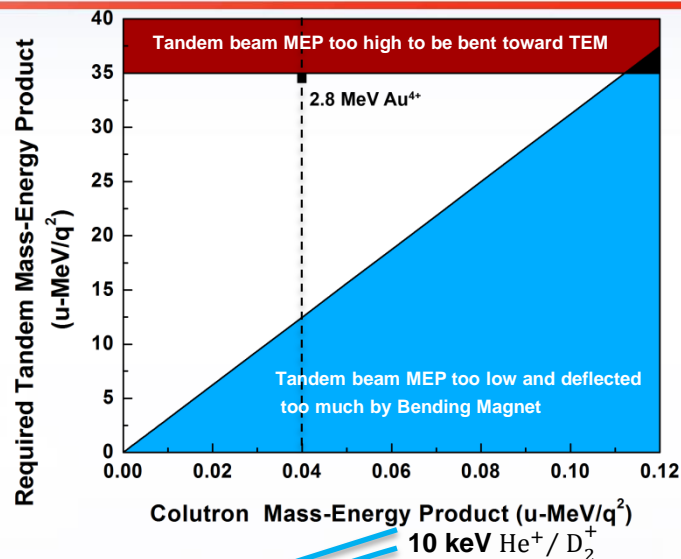
Collaborators: M. Steckbeck, D.C. Bufford, & B.L. Doyle



TEM
Obj. Lens



Bending
Magnet



Colutron Mass-Energy Product (u-MeV/q²)
10 keV He⁺ / D₂⁺

Steering Magnet

20°

2.8 MeV Au⁴⁺

- Must compensate for deflection of Tandem beam by bending magnet
Colutron beams deflected by the TEM objective lens
- Insignificant deflection of Tandem beams
- With 10 keV He/D₂ we can use Tandem beams $\approx 13 \text{ MeV/q}^2$

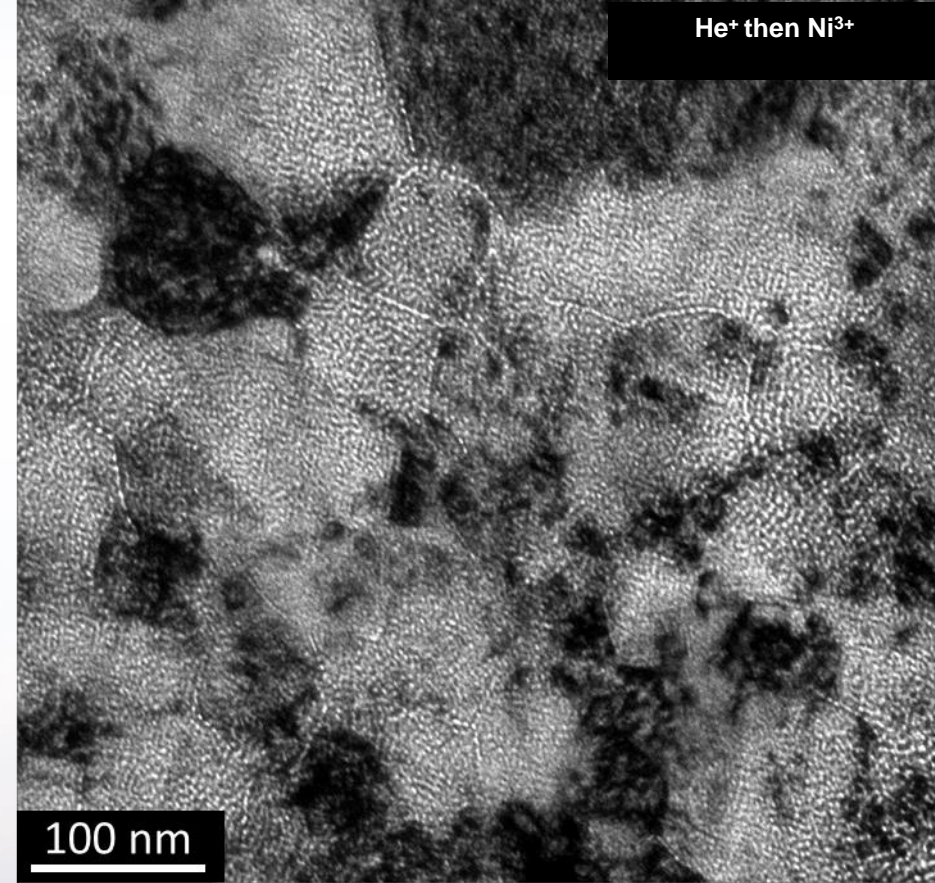
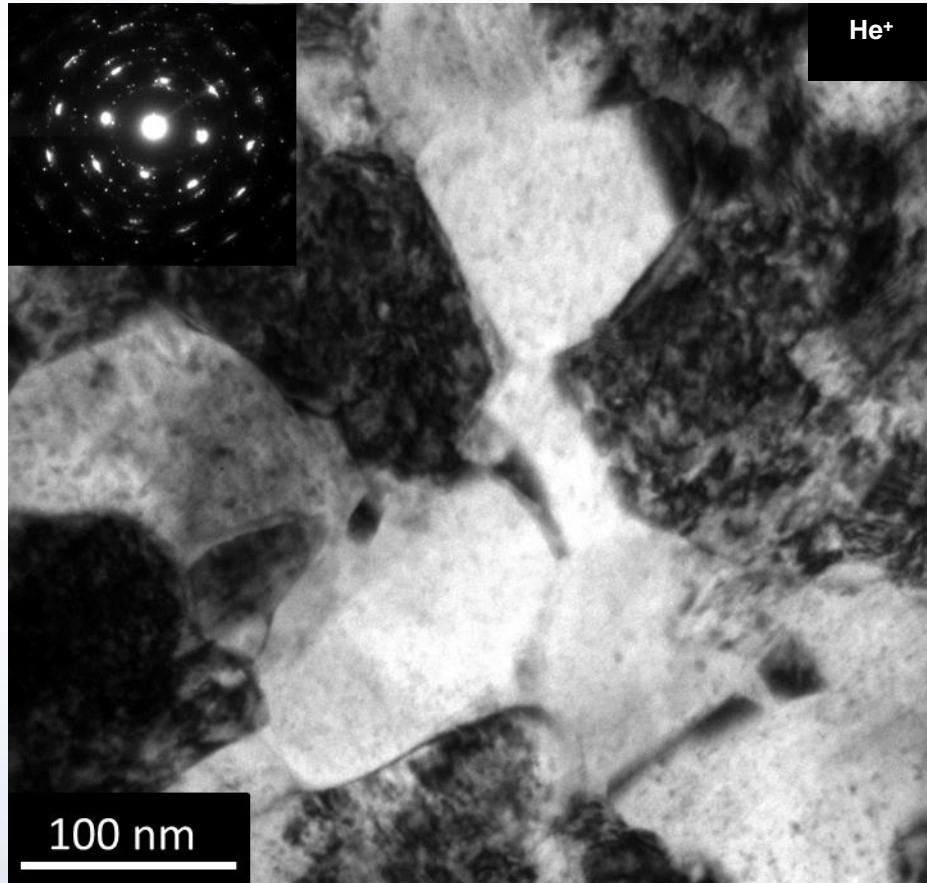
Au, He, and D₂
ions can all
reach the
sample
concurrently



Sandia National Laboratories

10 keV He⁺ Implantation followed by 3 MeV Ni³⁺ Irradiation

Collaborator: B. Muntifering & J. Qu



10¹⁷ He⁺/cm²

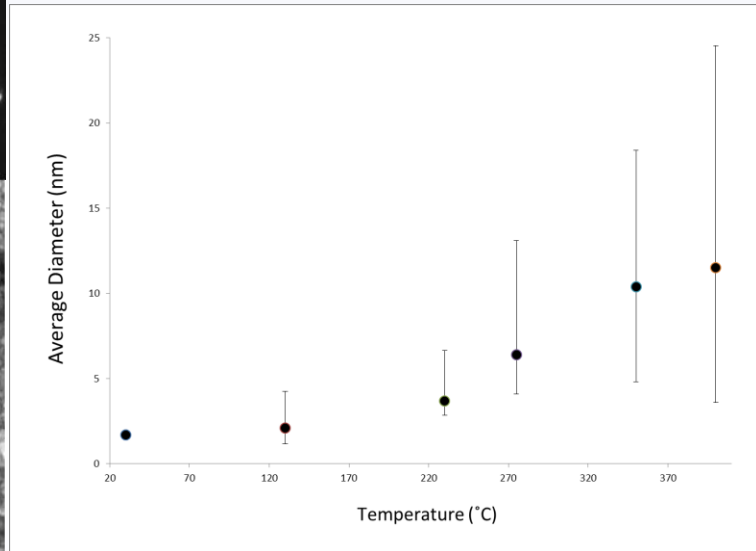
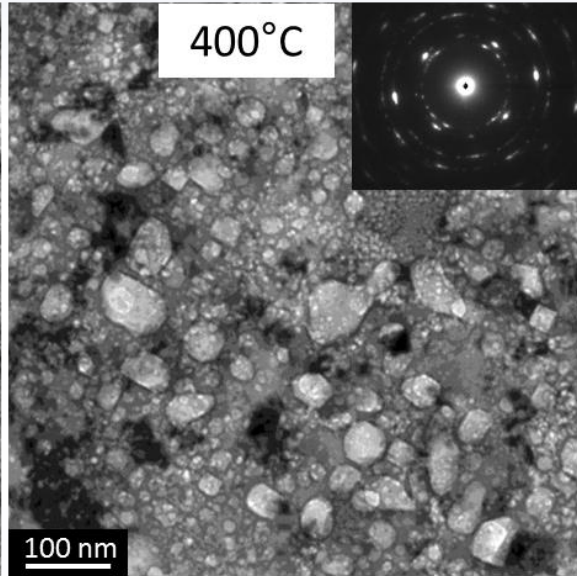
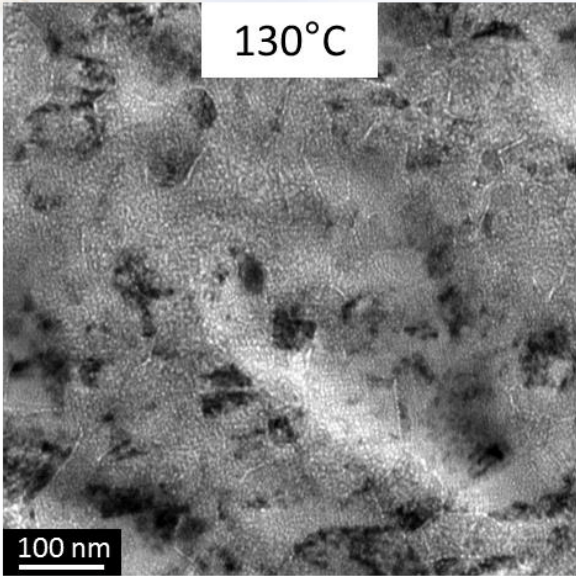
Visible damage to the sample

0.7 dpa Ni³⁺ irradiation

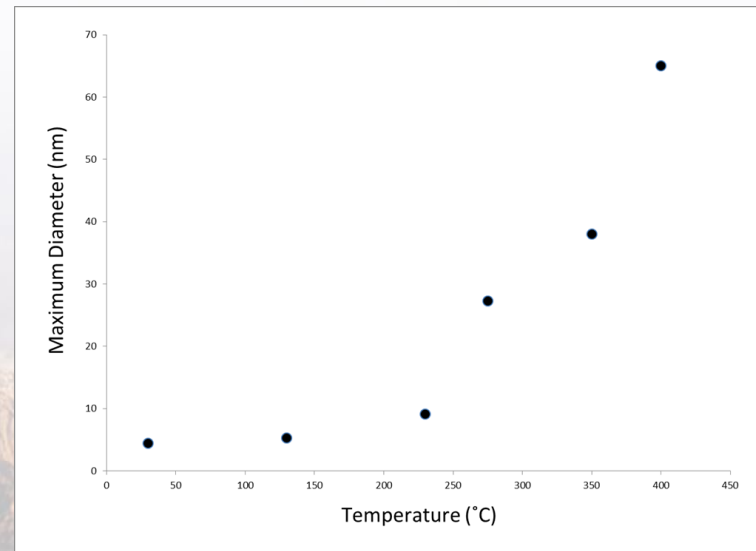
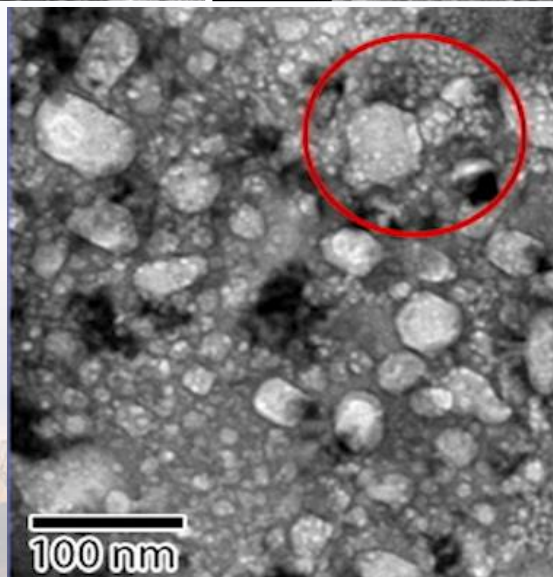
High concentration of cavities along
grain boundaries



Cavity Growth during In-situ Annealing of 10 keV He⁺ Implanted and then 3 MeV Irradiated Ni³⁺

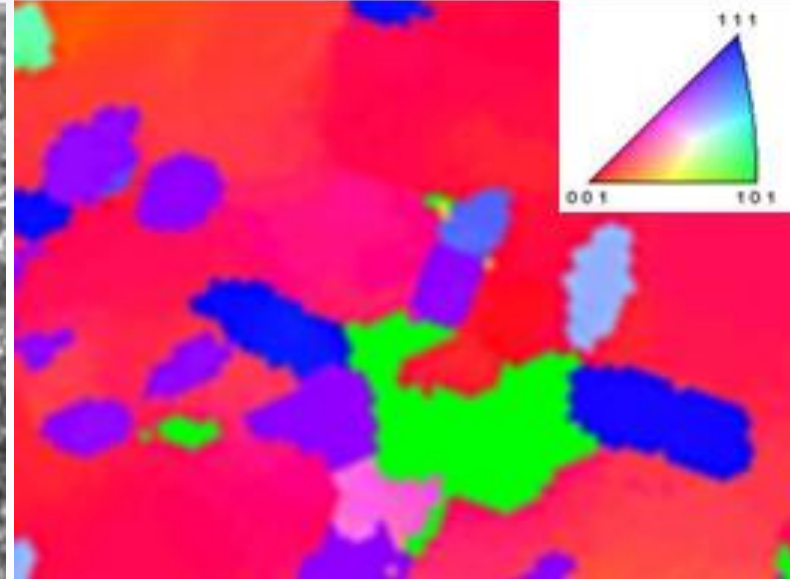
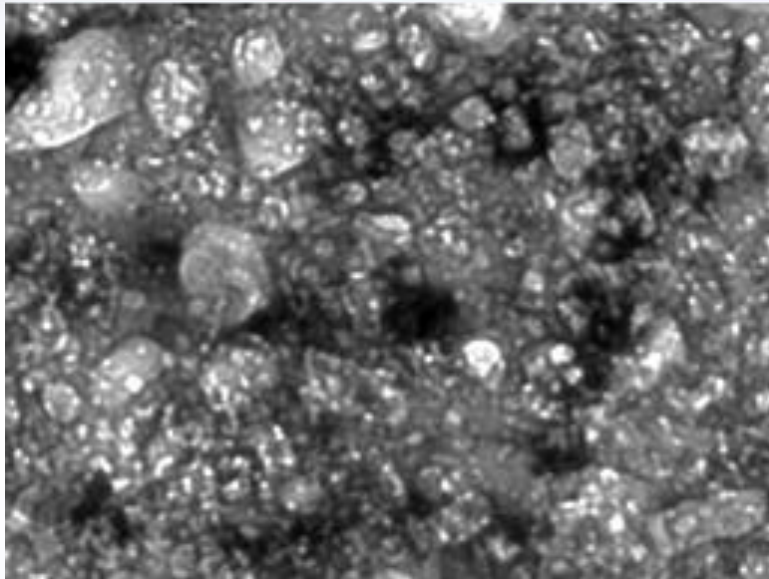


Bubble to cavity transition and cavity evolution can be directly studied

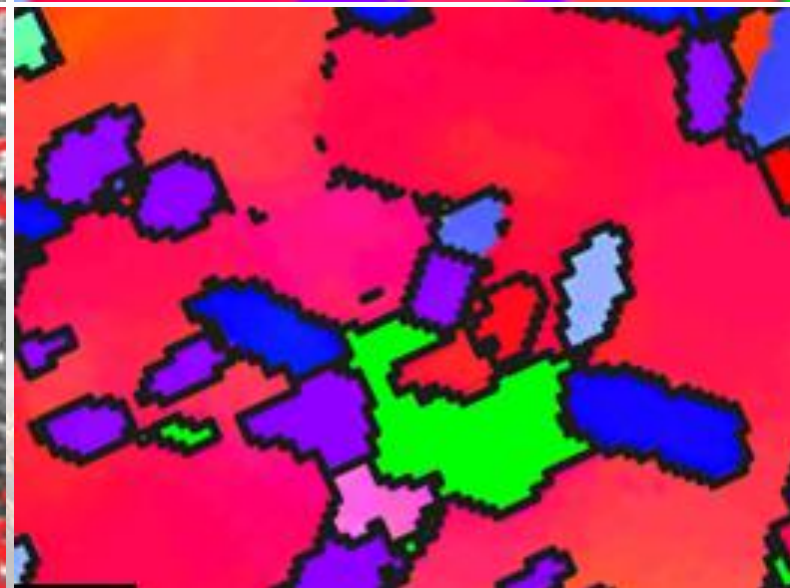
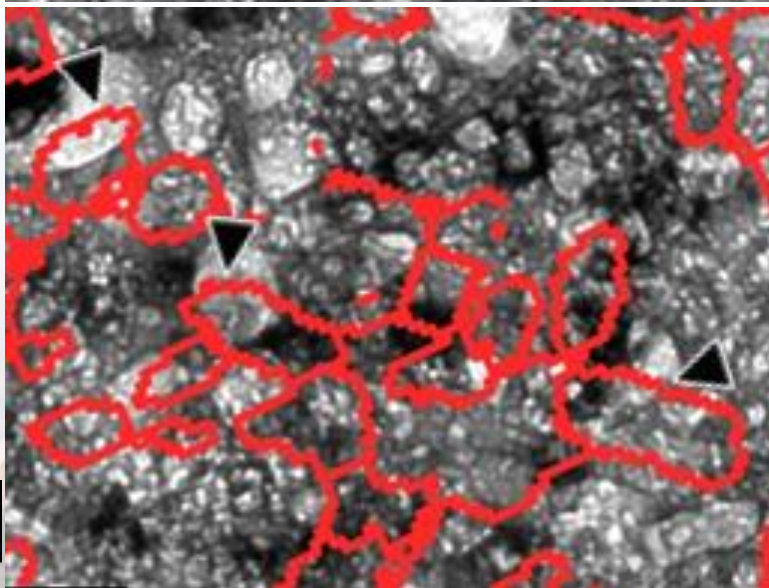


Precession Electron Diffraction Reveals Hidden Grain Structure

Cavities in helium implanted, self-ion irradiated, nc nickel film annealed to 400 °C



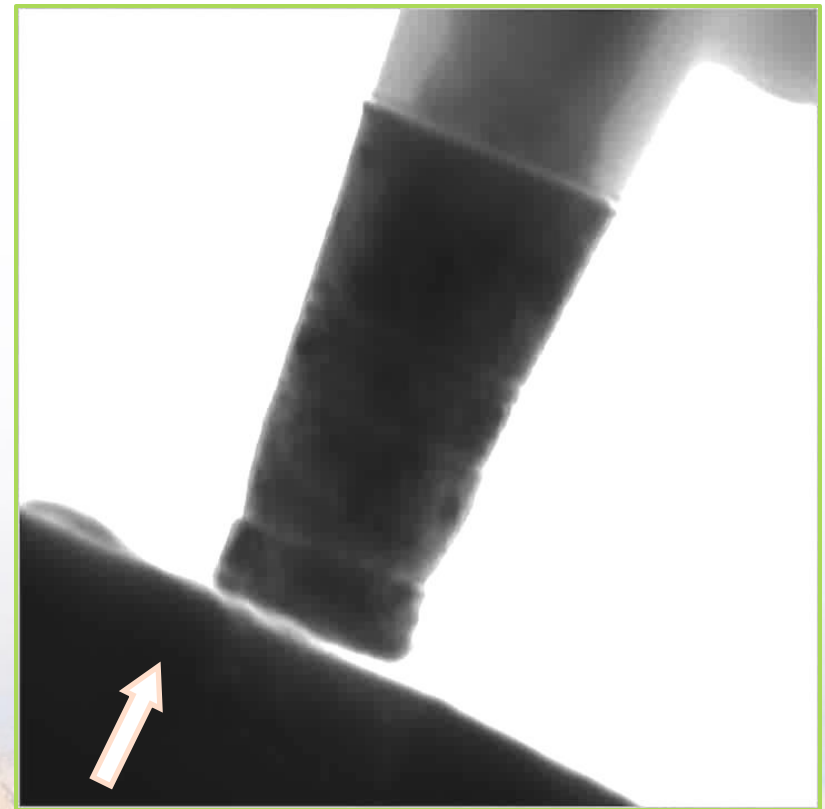
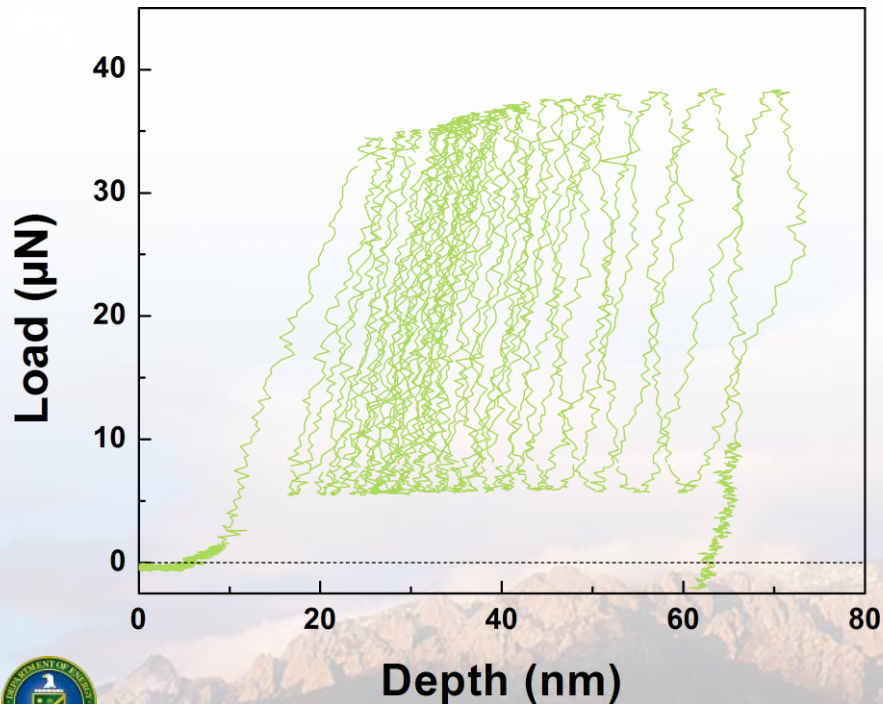
Cavities span multiple grains at identified grain boundaries



100 nm

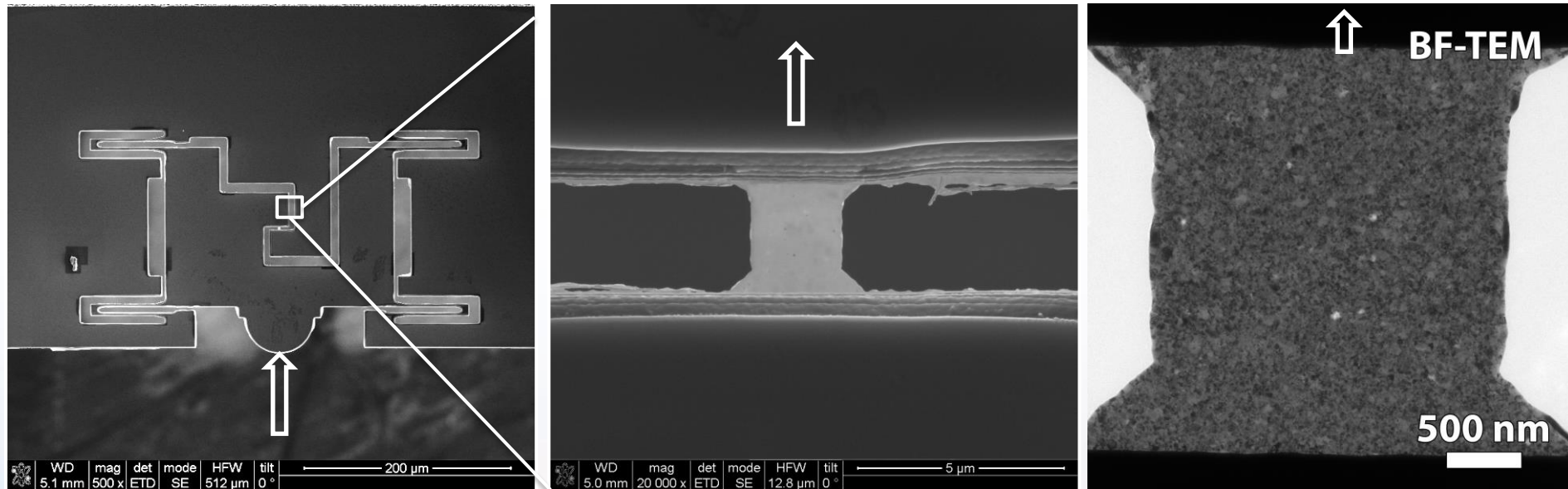
Cyclic Pillar Compression

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



Tension Specimen Fabrication

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



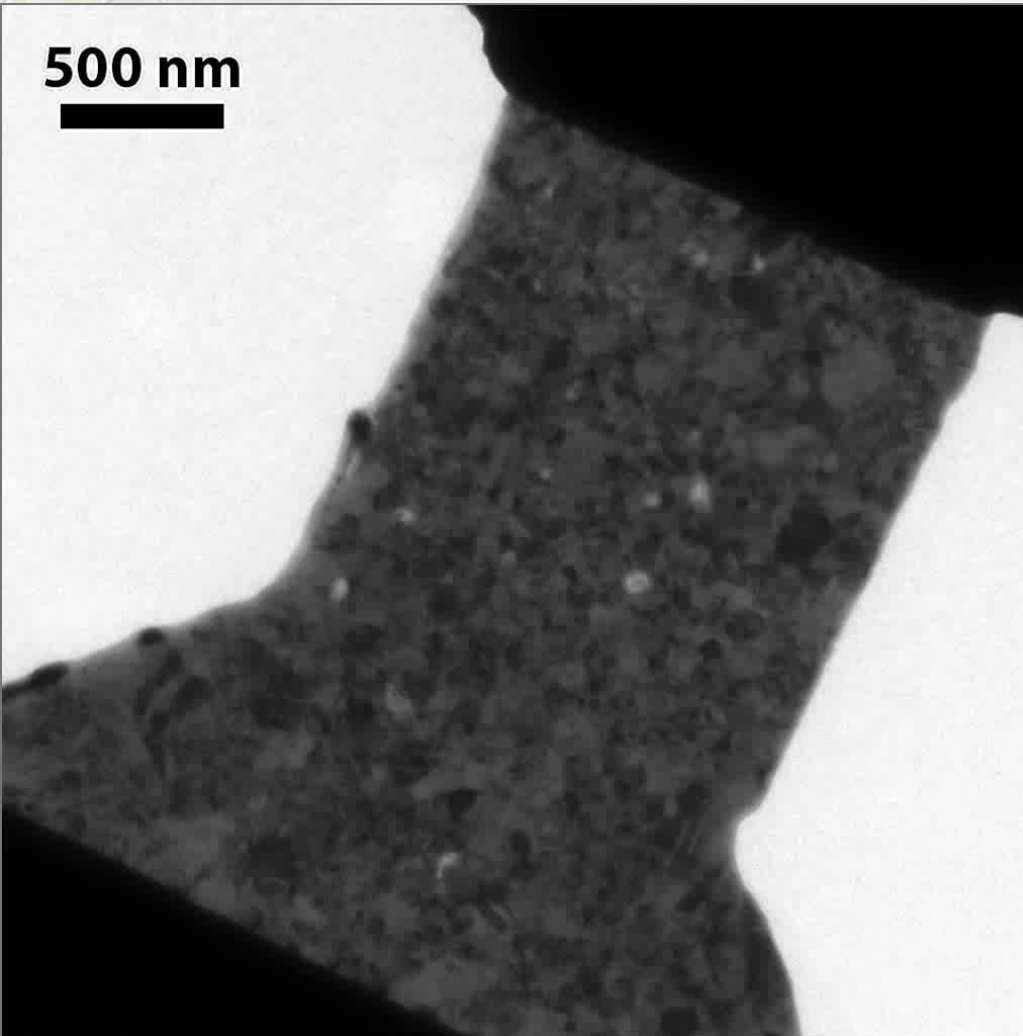
- Nearly pure tension, uniform cross sectional area, stable load frame
- Sensitive to shape of edges, issues with magnetic materials



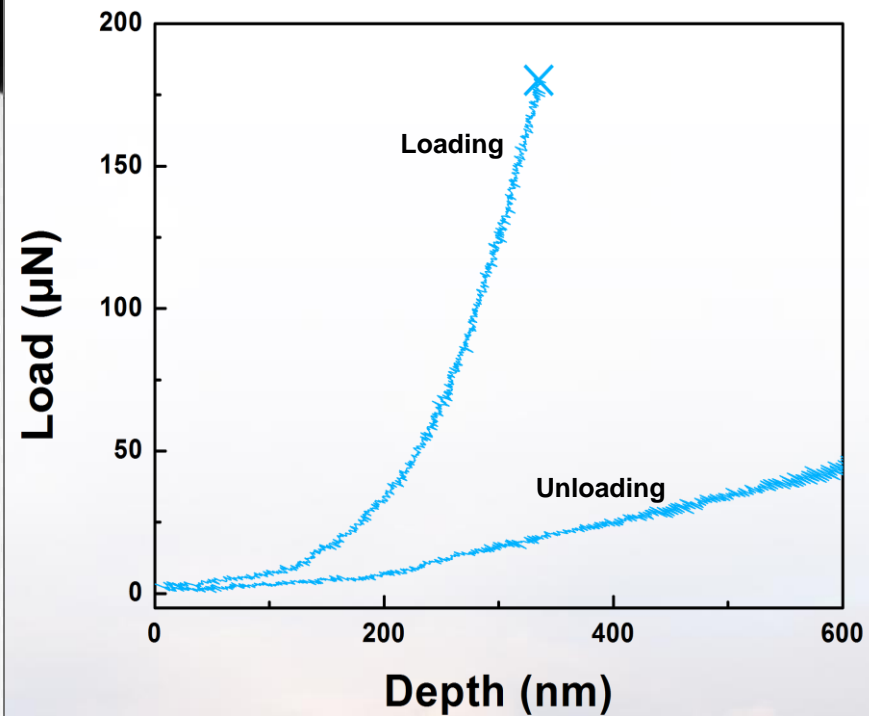
In situ TEM Monotonic Tension Testing

Video playback × 0.5

500 nm



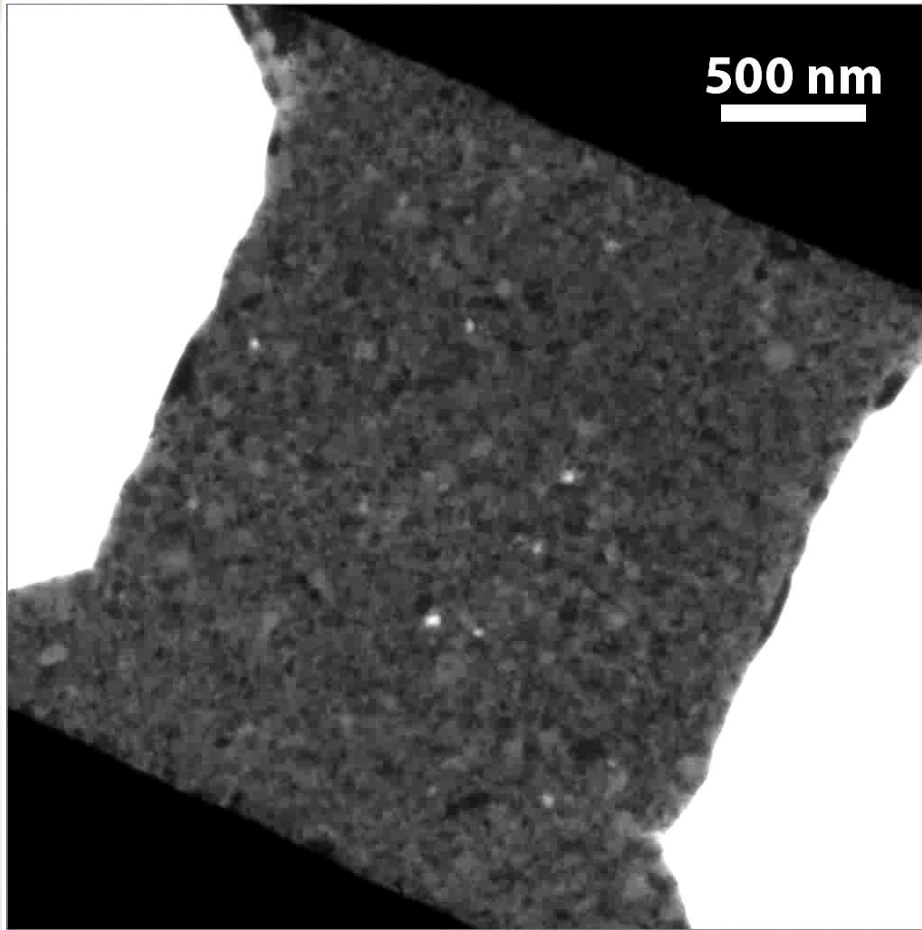
Raw Mechanical Property Data



In situ TEM Cyclic Tension Testing

Video playback × 10

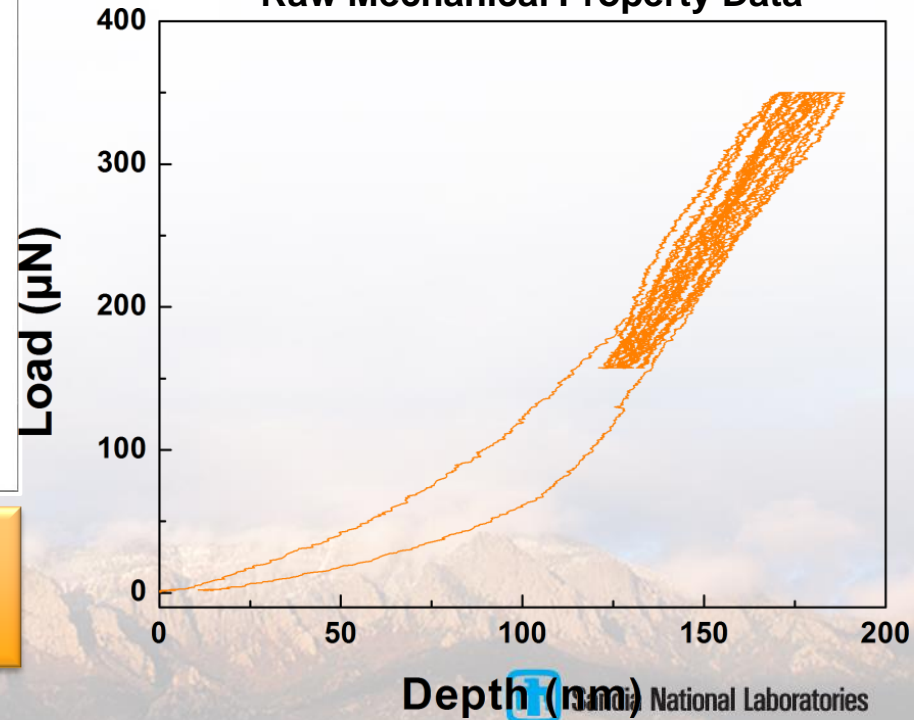
Collaborator: D. Stauffer



■ Cyclic loading:

- Crack initiated in previous monotonic test
- 9 cycles to $\approx 87.5\%$ of that load
- 50% unloading
- Slow crack propagation

Raw Mechanical Property Data

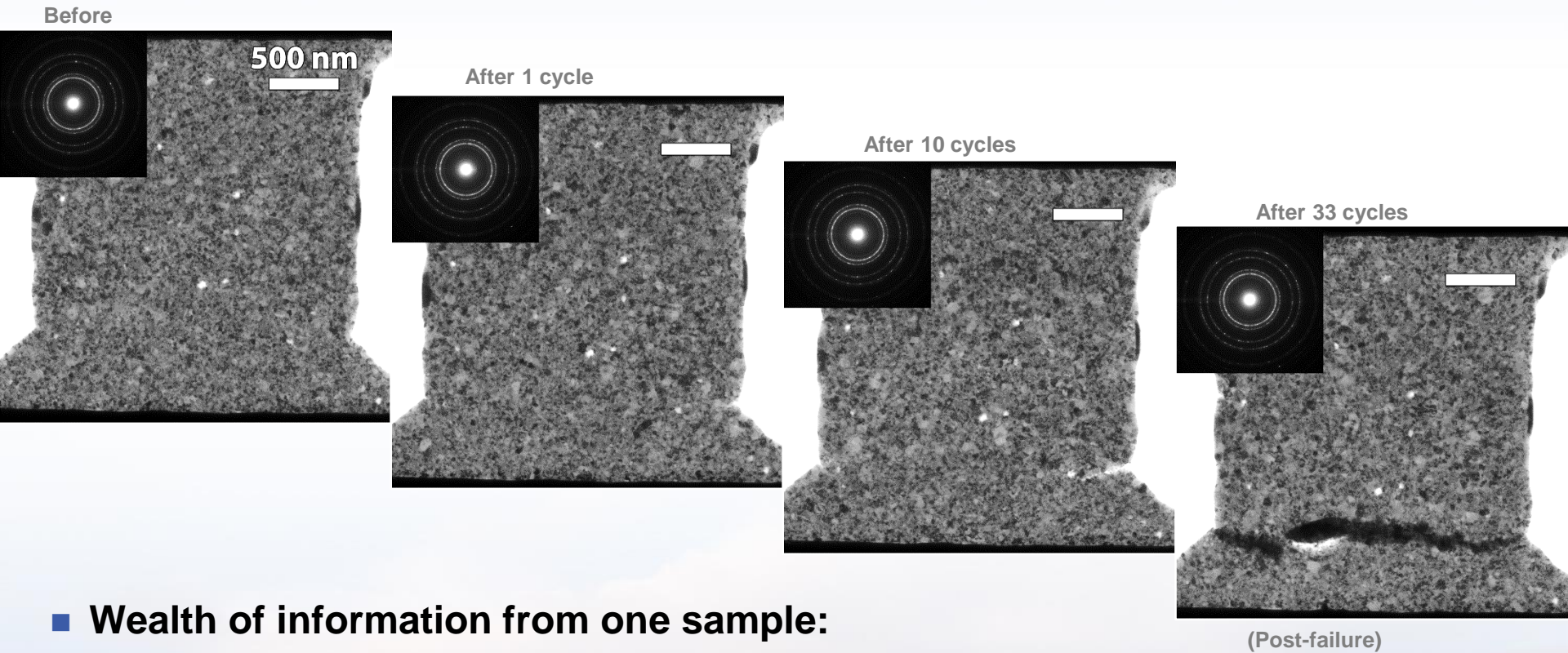


- Slow crack propagation
- Evidence of grain growth



Cyclic Tension *In Situ*

Collaborator: D. Stauffer

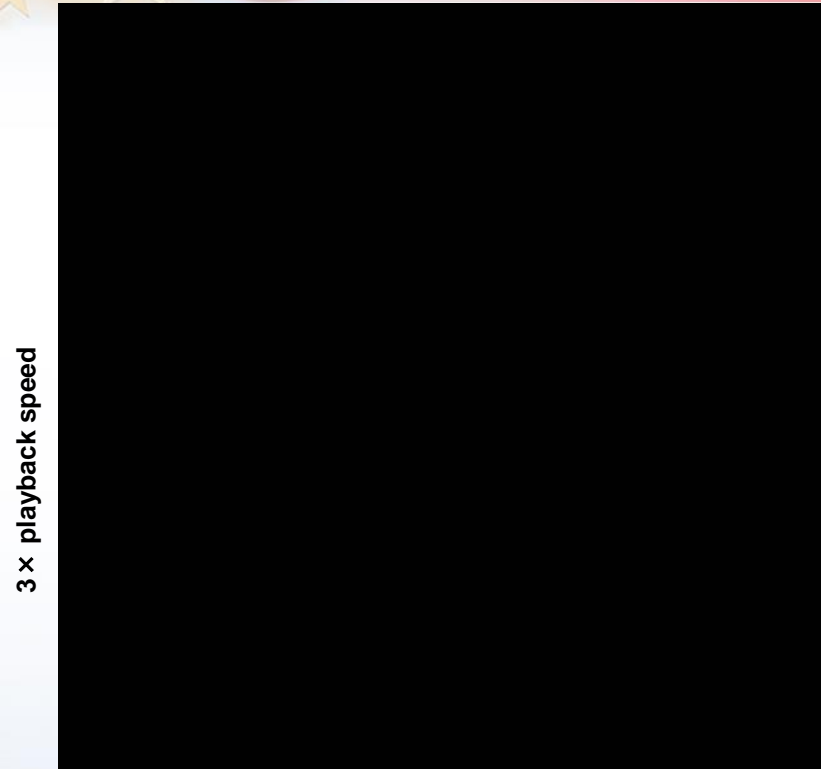


- **Wealth of information from one sample:**
 - Images and electron diffraction at each stage
 - Video and force/displacement during load cycles
- **Microstructural change still elusive**
 - Difficult to confirm and quantify

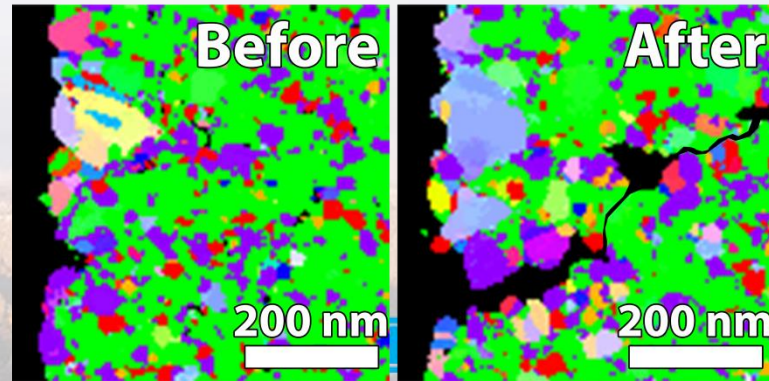
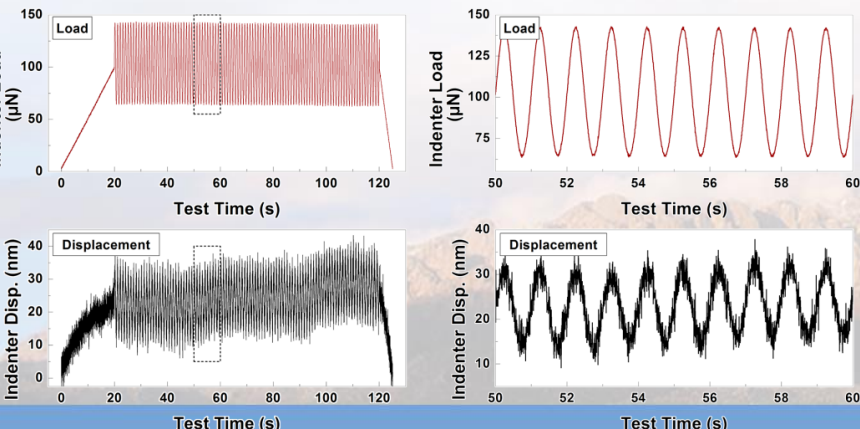
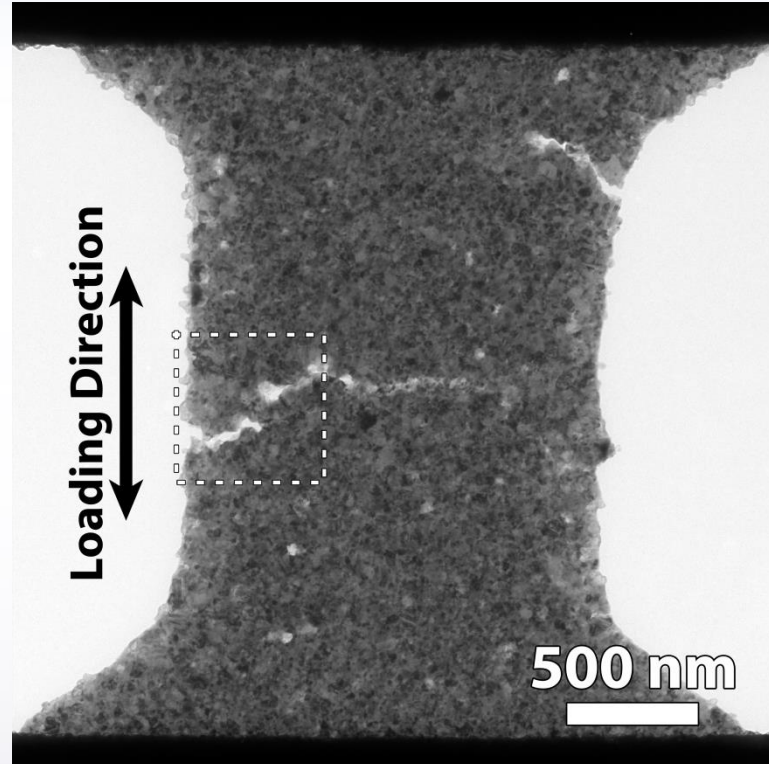


In situ TEM Quantitative Fatigue Testing

Contributors: D.C. Bufford, D. Stauffer, W. Mook

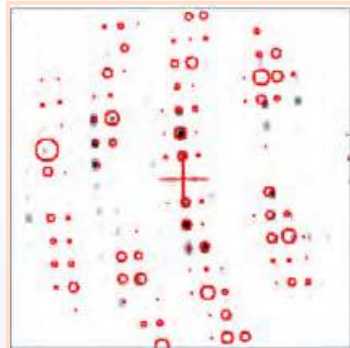
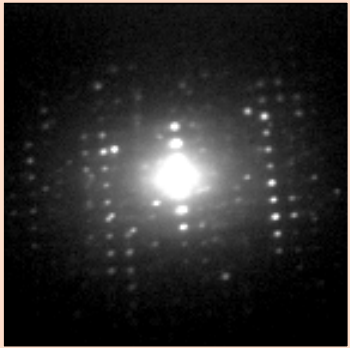


*High cycle
fatigue in
real time
with
nanometer
resolution*



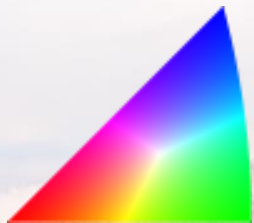
Quantifying Microstructural Change

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

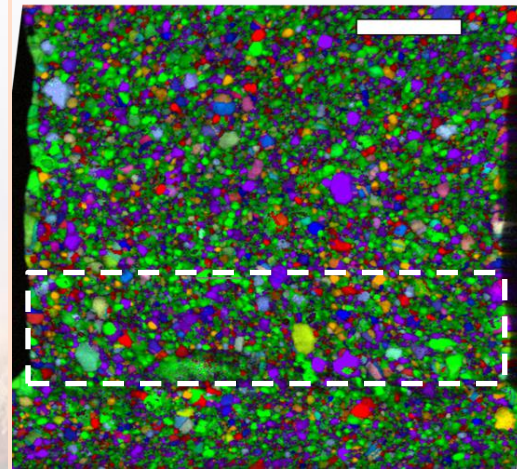
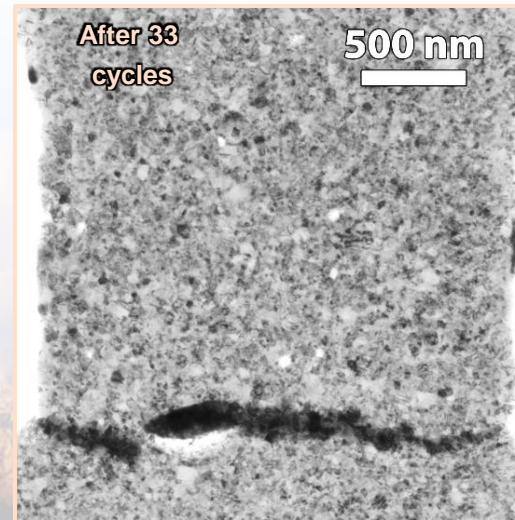
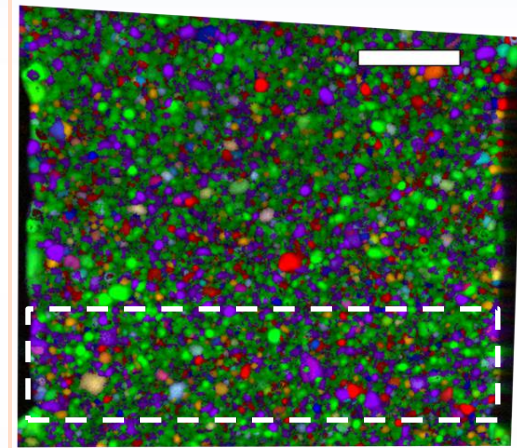
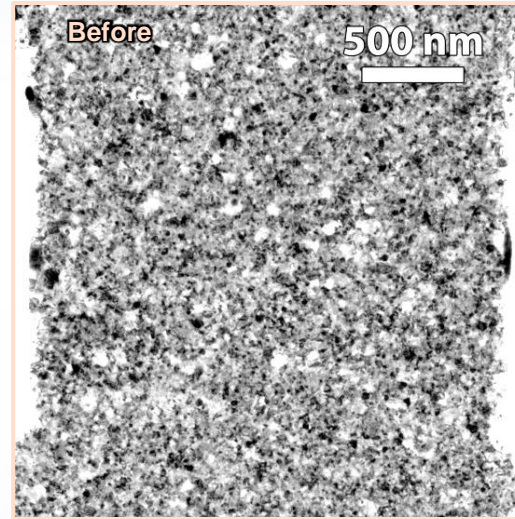


111

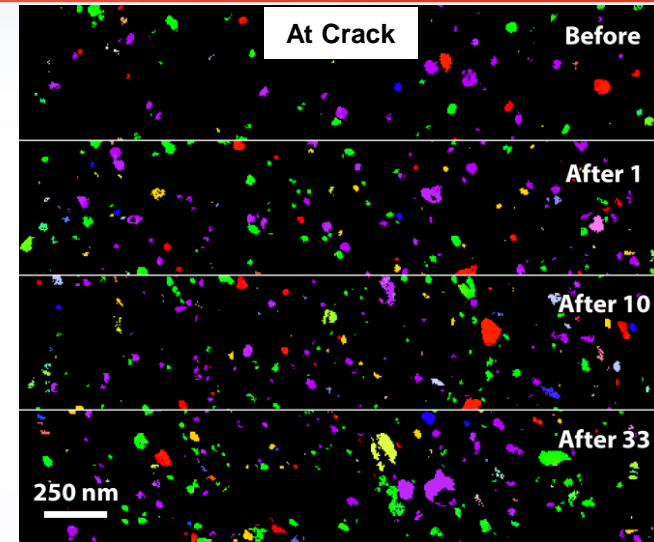
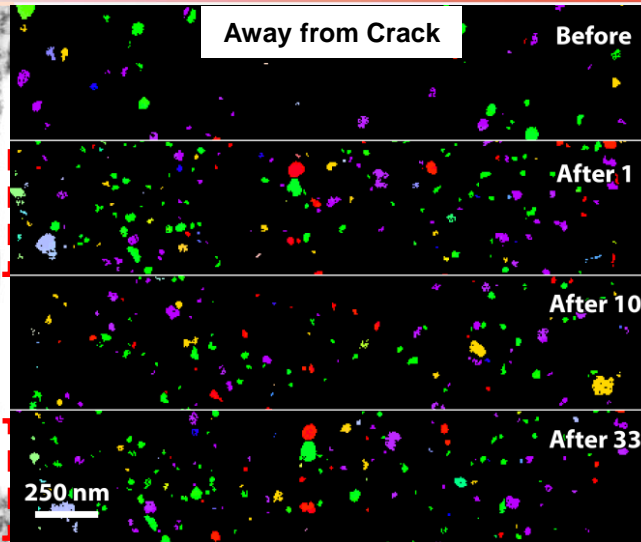
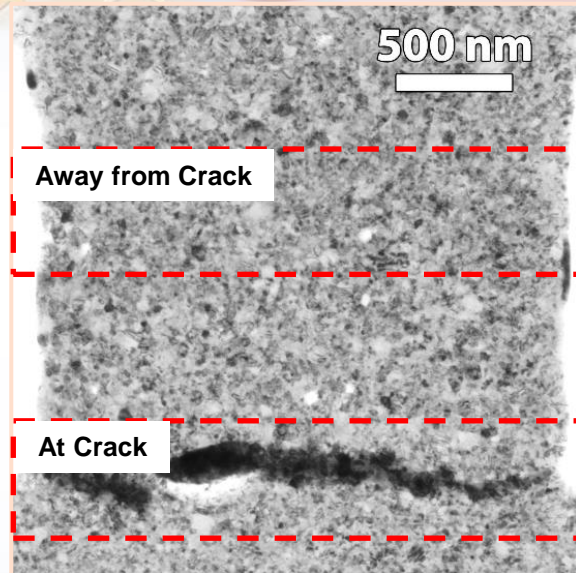
100



110

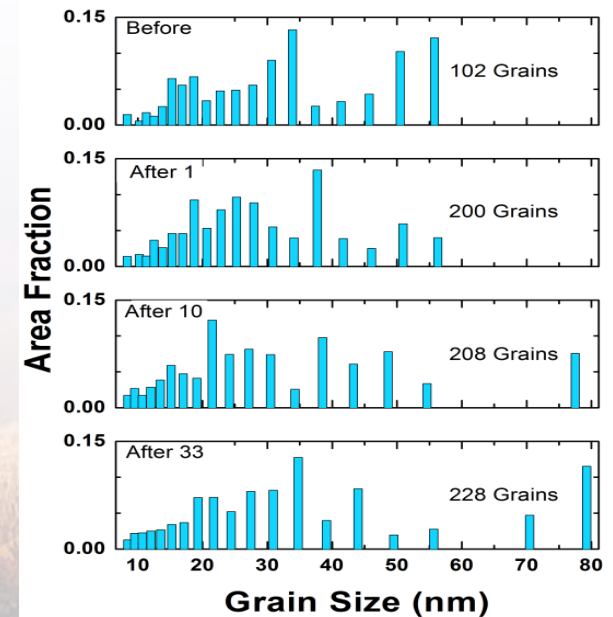
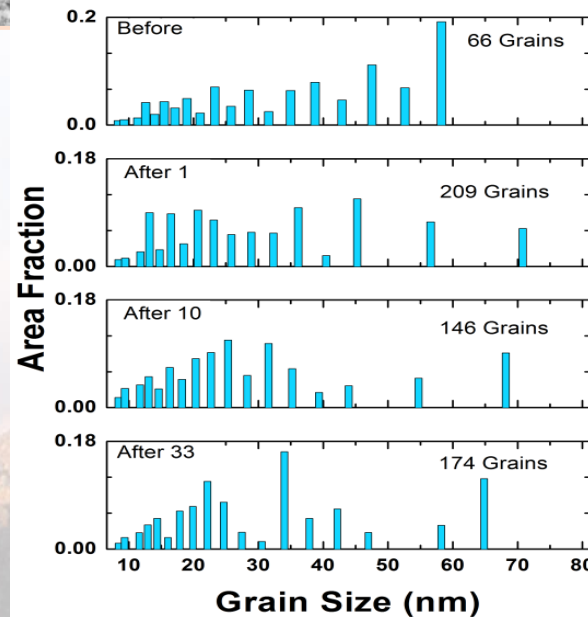


Quantifying Microstructural Change



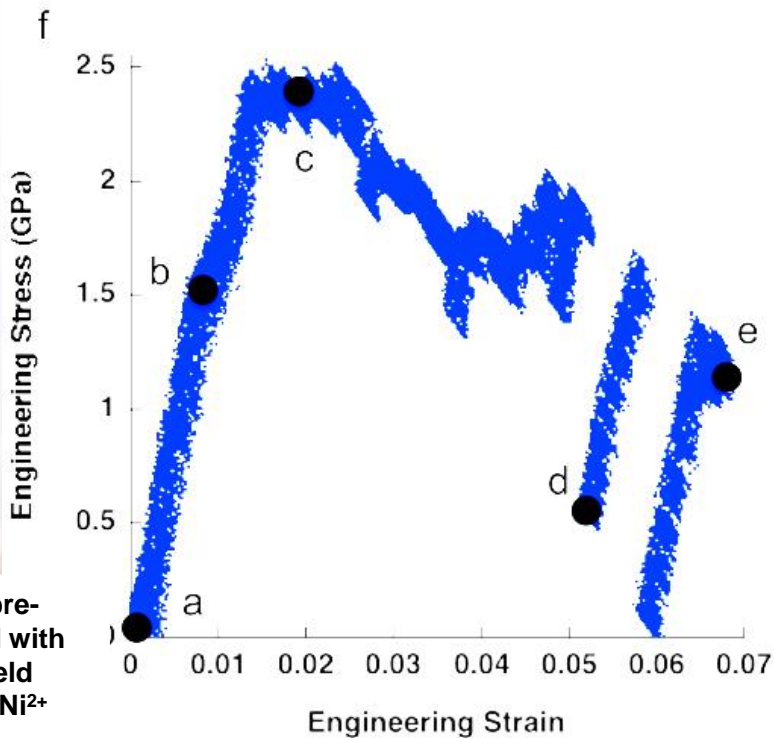
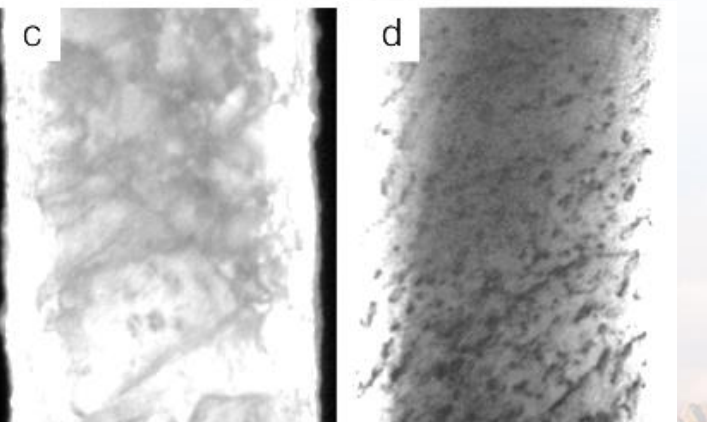
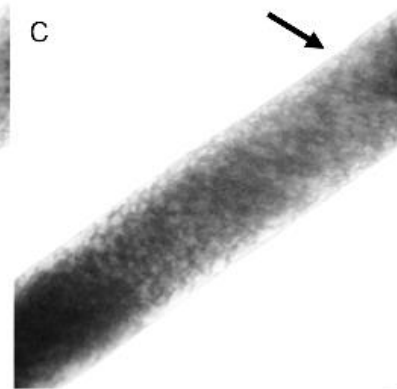
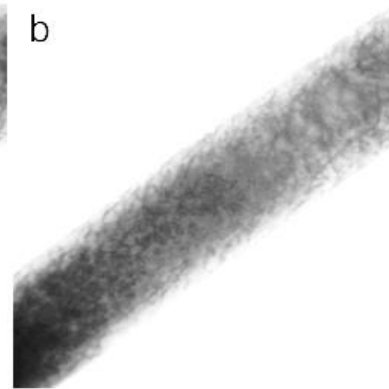
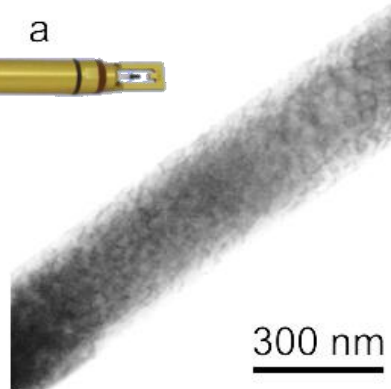
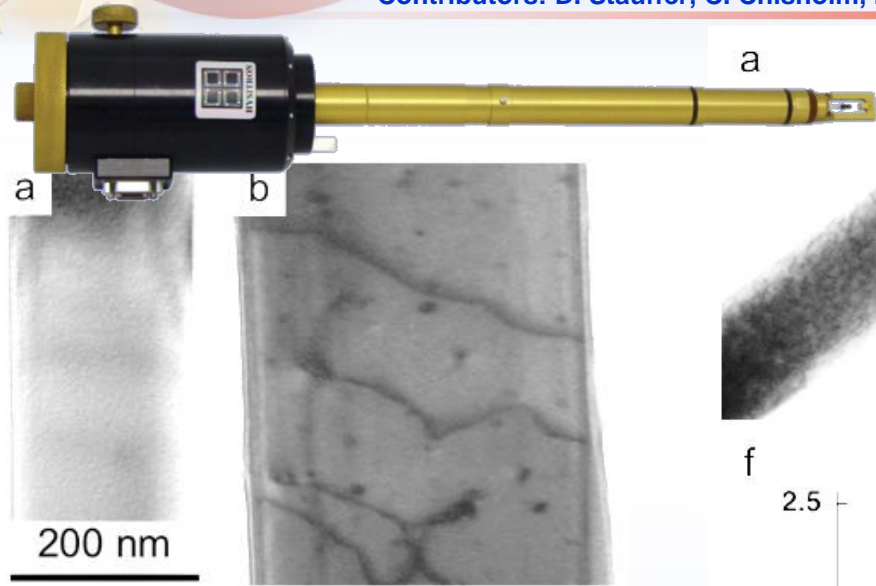
Texture and Grain size analysis can target:

- Various regions
- Quality of PED patterns
- Particular orientations
- Set of grain sizes
- Etc.



In situ TEM Quantitative Mechanical Testing

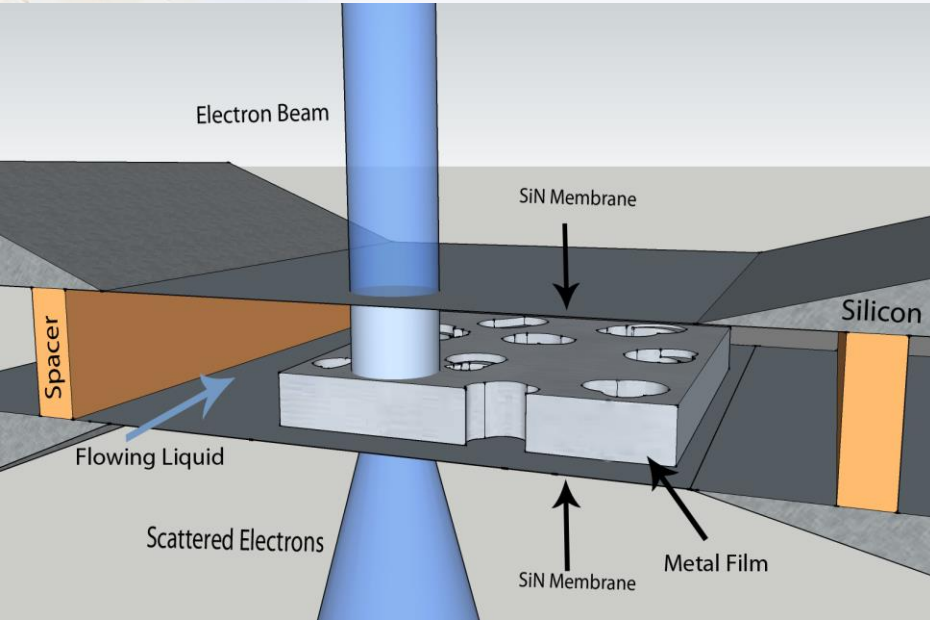
Contributors: D. Stauffer, C. Chisholm, H. Bei, E.P. George, P. Hosemann, & A.M. Minor



TEM micrographs of Mo-alloy nanofibers. (a) 0% pre-strained, no observable defects. (b) 9% pre-strained with mobile dislocations and sessile loops. (c) Dark-field micrograph of 15% pre-strained fiber. (d) He¹⁺ and Ni²⁺ irradiated, 9% pre-strained fiber.

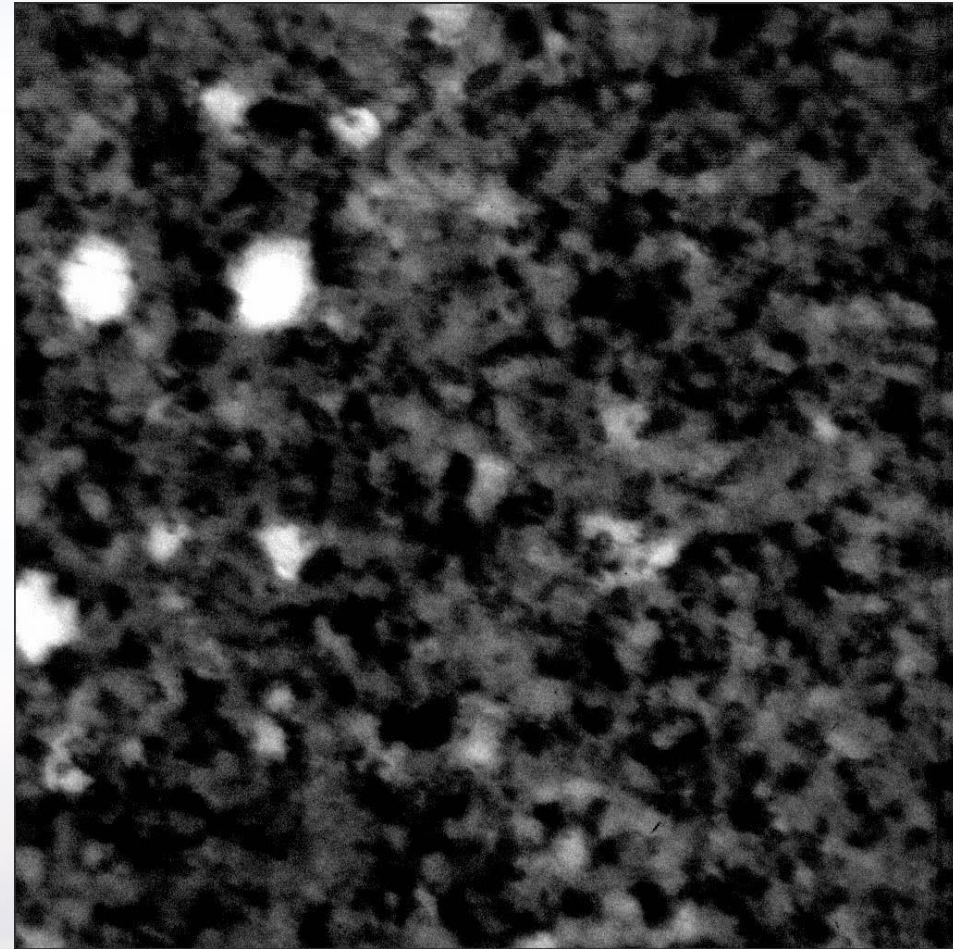
Future Direction: *In situ* TEM Corrosion Direction

Contributors: D. Gross, J. Kacher, & I.M. Robertson



Microfluidic Stage

- Mixing of two or more channels
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Films can be directly deposited on the electron transparent SiN membrane



Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.

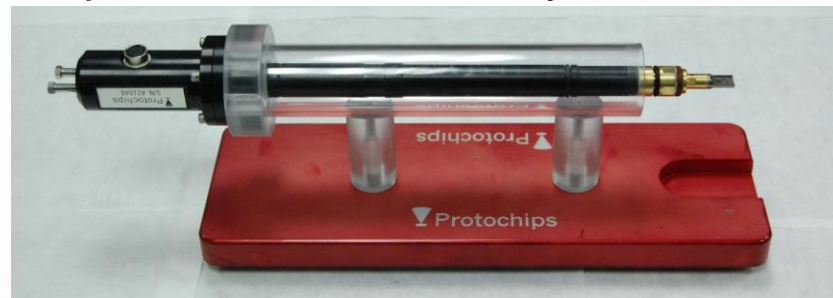


Future Direction: *In situ* TEM Hydrogen Exposure

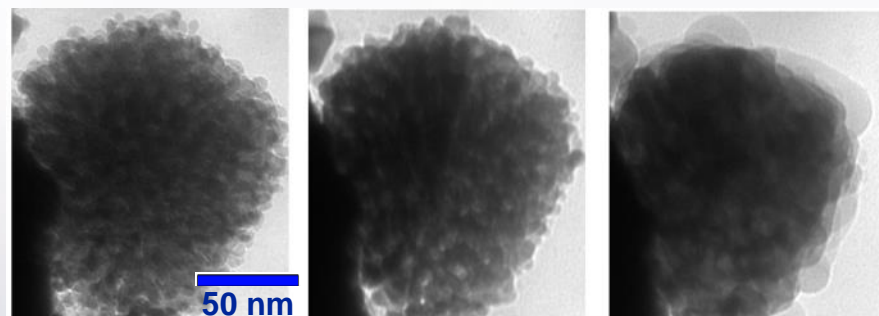
Contributors: B.G. Clark, P.J. Cappillino, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, L.R. Parent, I. Arslan. & Protochips, Inc.

Vapor-Phase Heating TEM Stage

- Compatible with a range of gases
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Compatible with MS and other analytical tools



- 1 atm H₂ after several pulses to specified temp.

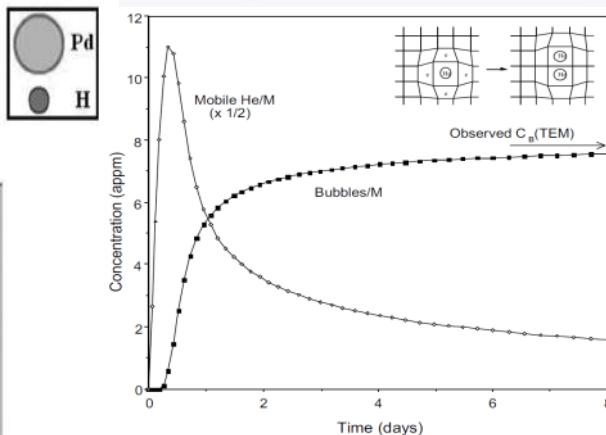


125° C

200° C

300° C

New *in situ* atmospheric heating experiments provide great insight into nanoporous Pd stability



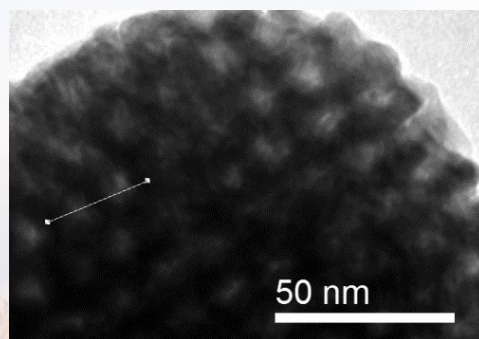
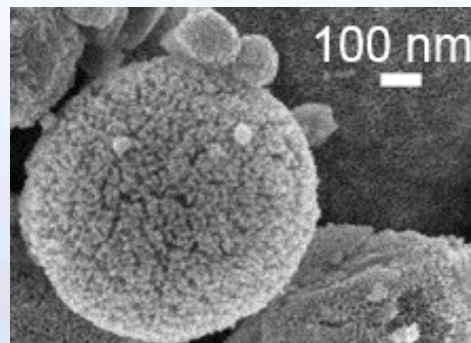
Cowgill, D., *Fusion Sci. & Tech.*, 28 (2005) p. 539

Trinkaas, H. *et al.*, *JNM* (2003) p. 229

Thiebaut, S. *et al.* *JNM* (2000) p. 217

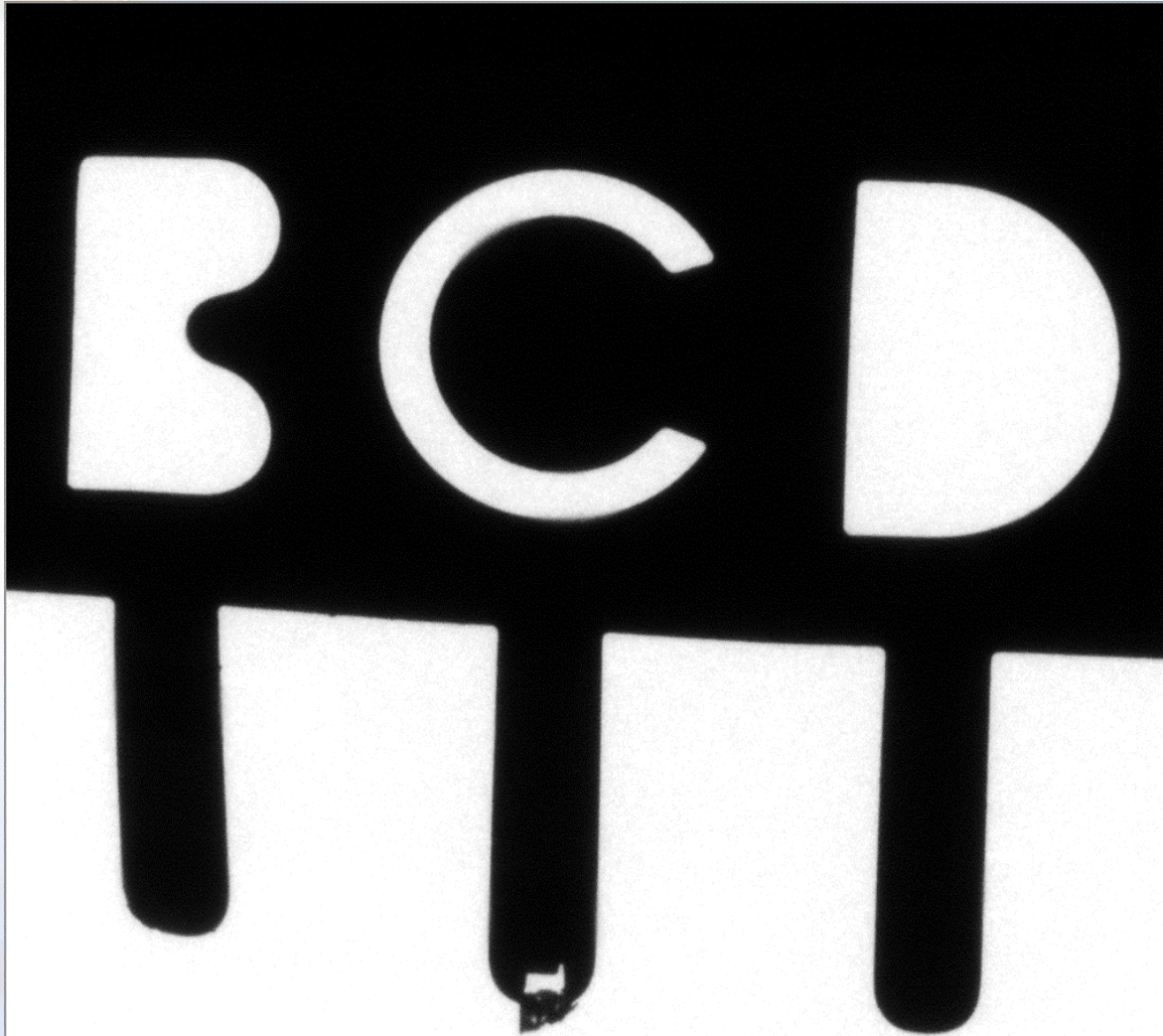
R. Delmelle, J., *Phys. Chem. Chem. Phys.* (2011) p.11412

Harmful effects may be mitigated in nanoporous Pd

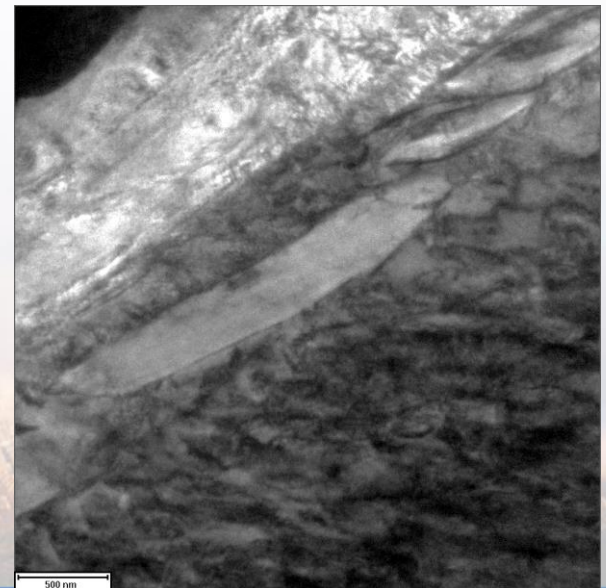
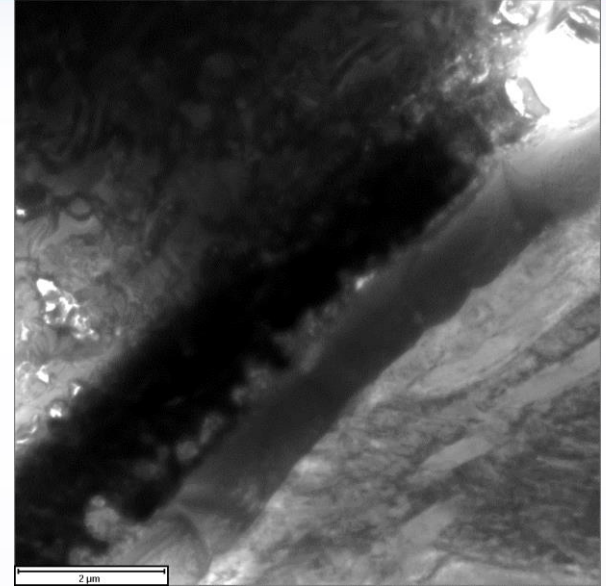


First “Hot” sample into I³TEM

Contributors: A. Aitkaliyeva, R. Hess, R. Dingreville



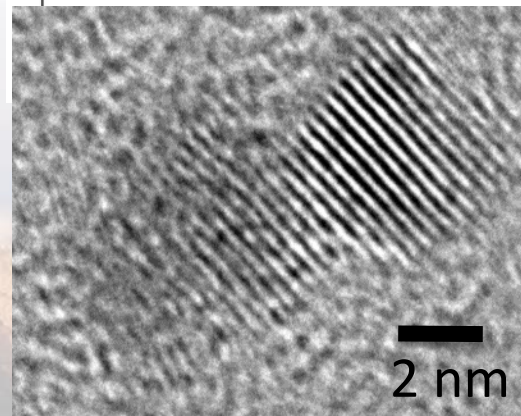
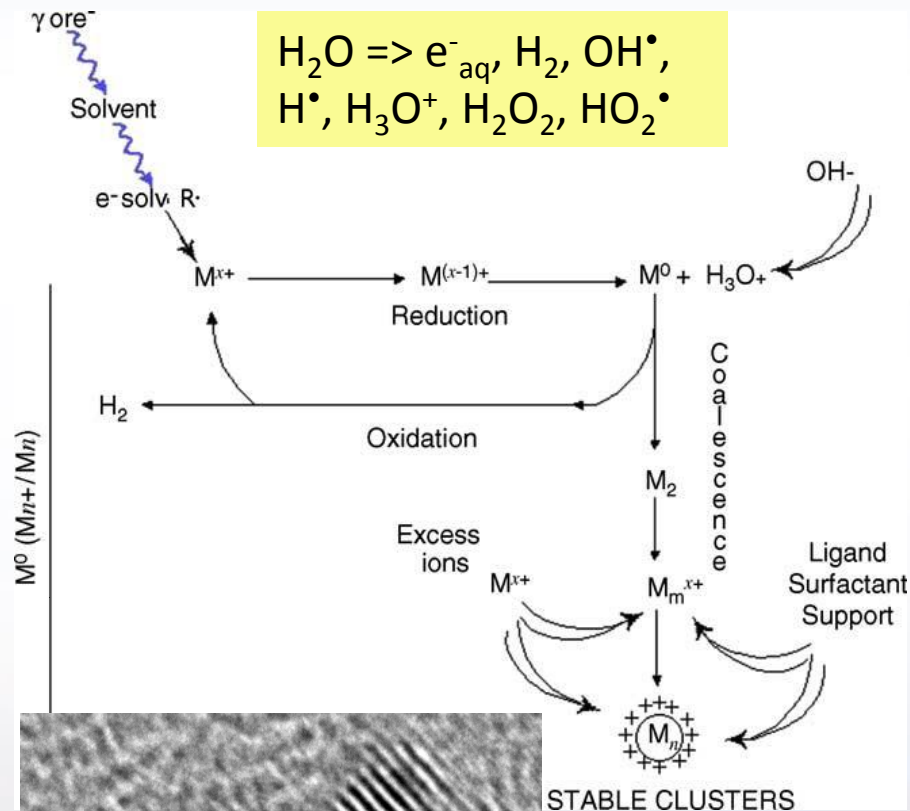
This Pellet Cladding Interface is the first “hot” TEM sample approved for study in Sandia’s I³TEM



Low Temperature Synthesis of Lanthanide and Actinide Nanoparticles

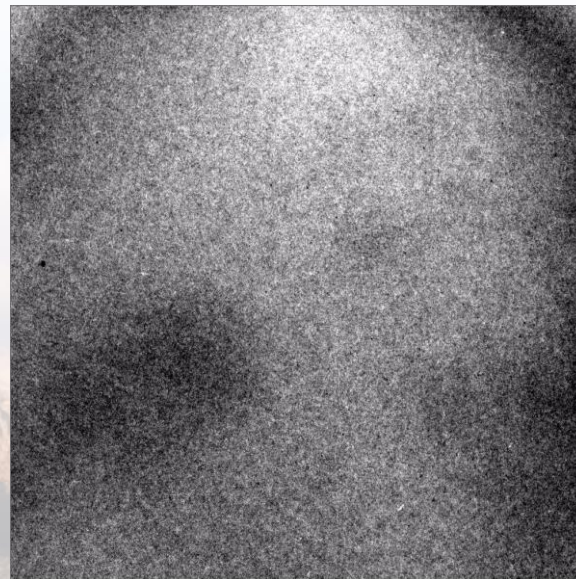
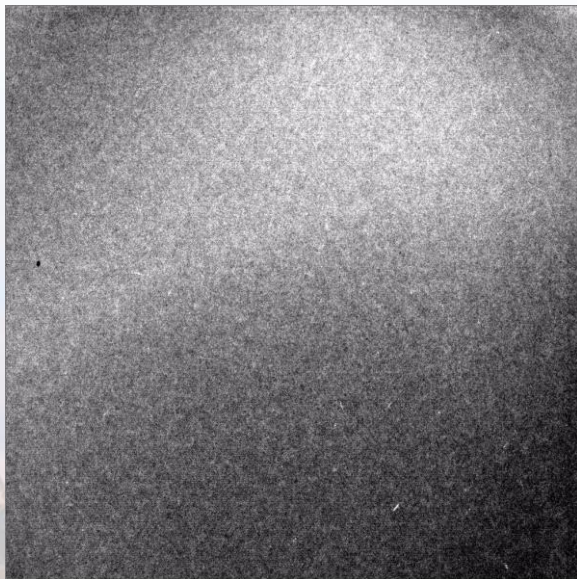
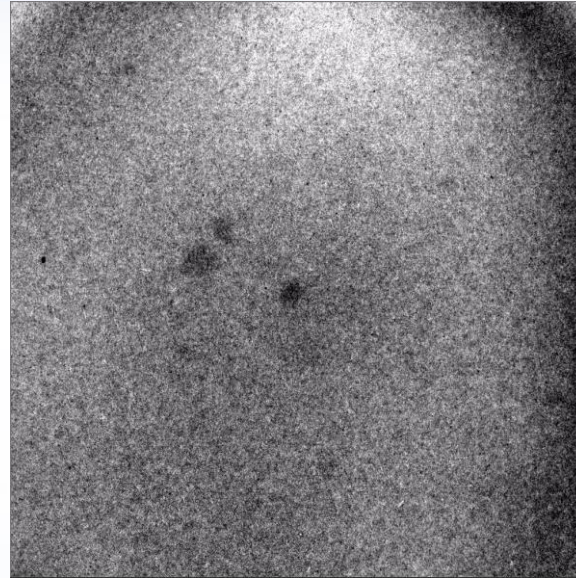
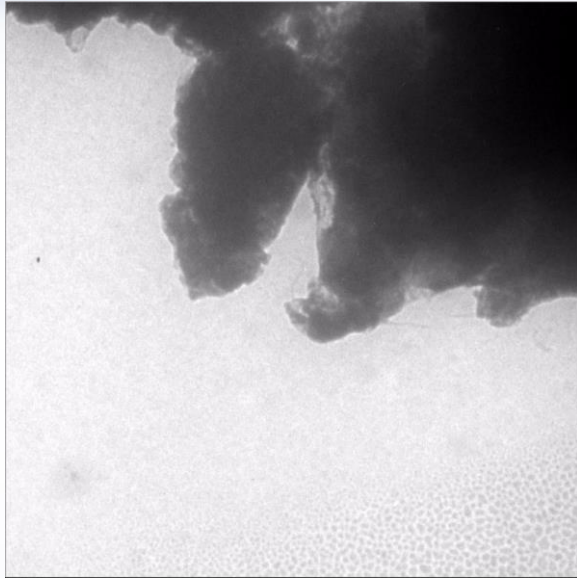
- Fabrication of nuclear fuels at low temperatures allowing for the formation of complex Uranium metal and alloys with minimum volatility of alloy compounds in the process
- Potentially permits the recycling of dissolved uranium and lanthanide ions from acidic solutions (used fuel) into metals and alloys
- Gamma (γ) irradiation is used to reduce uranium and lanthanide ions to metal in solution at room temperature
- result in Limits potential release to the environment and/or other ES&H issues

Radiolytic formation of actinide nanoparticles
 RT approach for nuclear fuels synthesis reducing defect formations in bulk metals when compared to normal sintering at 1500-1700°C



T.M. Nenoff, *et al.* J. Nuc. Mater. 442 (2013)
 T.M. Nenoff, *et al.* Chem. Mater. 2011, 23,
 Sandia National Laboratories

In situ Observation of the Various Structural Evolutions



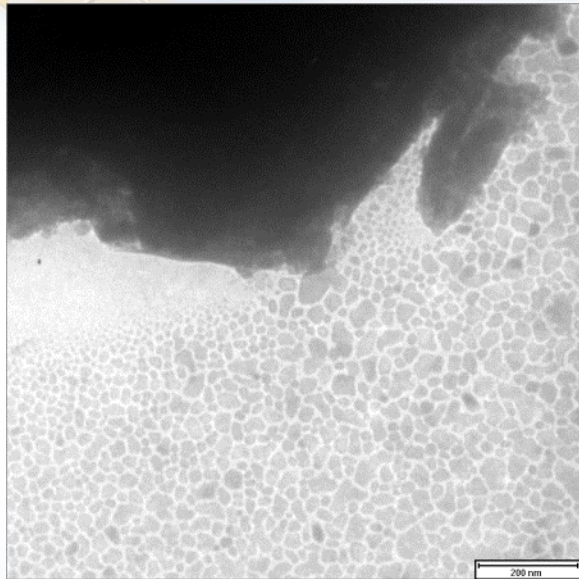
Structural evolution is highly dependent on:

- the lanthanide salt
- local salt conc.
- flow rate
- proximately to other nanostructures
- e⁻ beam dose
- e⁻ beam dose rate
- etc.

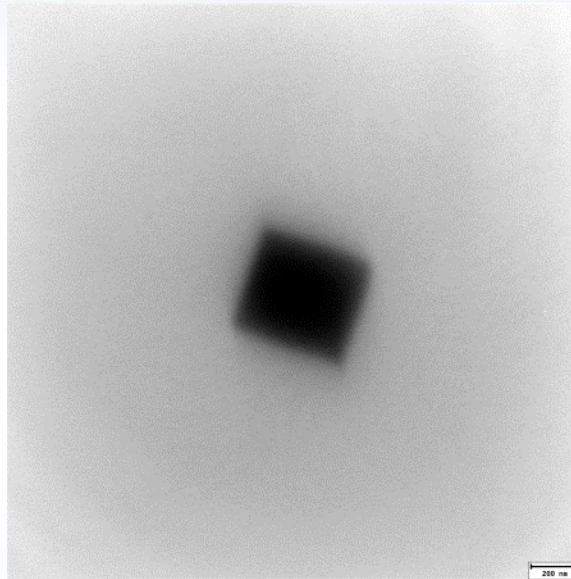


The Resulting Microstructures

EuCl_3 in H_2O



EuCl_3 in H_2O



LaCl_3 in H_2O

