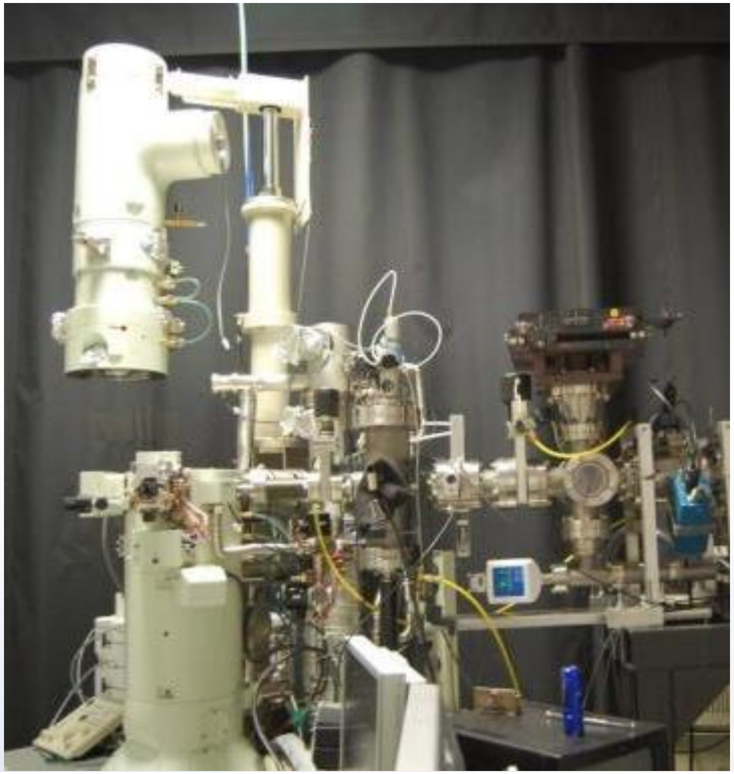
Investigating Overlapping and Harsh Environments via *In situ* TEM

SAND2015-11117C

K. Hattar

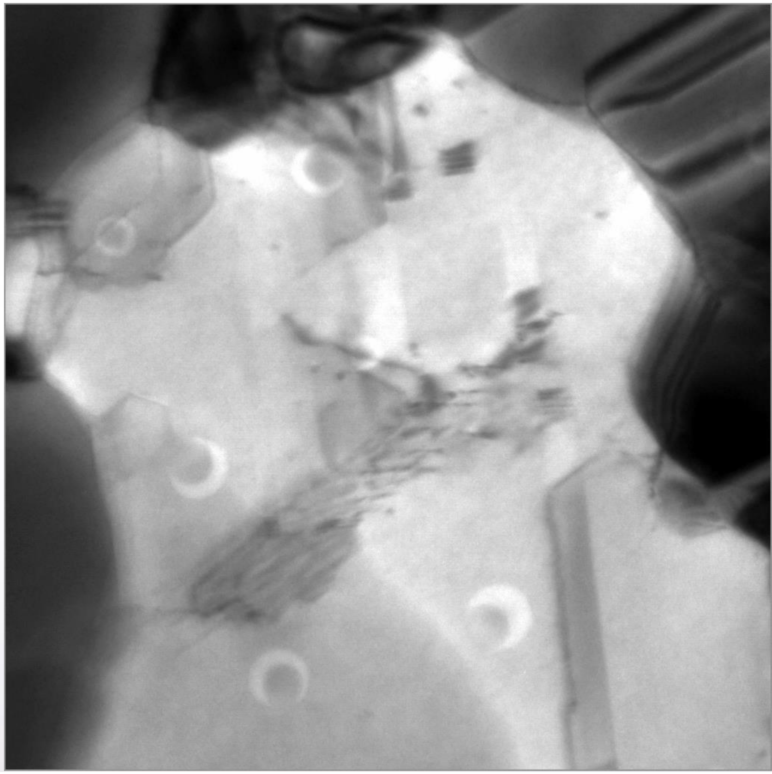
Ion Beam Lab at Sandia National Laboratories

January 3, 2016



***In situ* TEM microscopy**

has recently undergone significant growth providing capabilities to investigate the structural evolution that occurs due to various extreme environments and combinations thereof



Collaborators:

- **IBL: D.C. Bufford, D. Buller, C. Chisholm, B.G. Clark, J. Villone, B.L. Doyle, S. H. Pratt, M. Steckbeck & M.T. Marshall**
- **Sandia: B. Boyce, T.J. Boyle, P.J. Cappillino, J.A. Scott, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, J.A. Sharon, W.M. Mook, F. Abdeljawad, & S.M. Foiles**
- **External: A. Minor, L.R. Parent, I. Arslan, H. Bei, E.P. George, P. Hosemann, D. Gross, J. Kacher, & I.M. Robertson**

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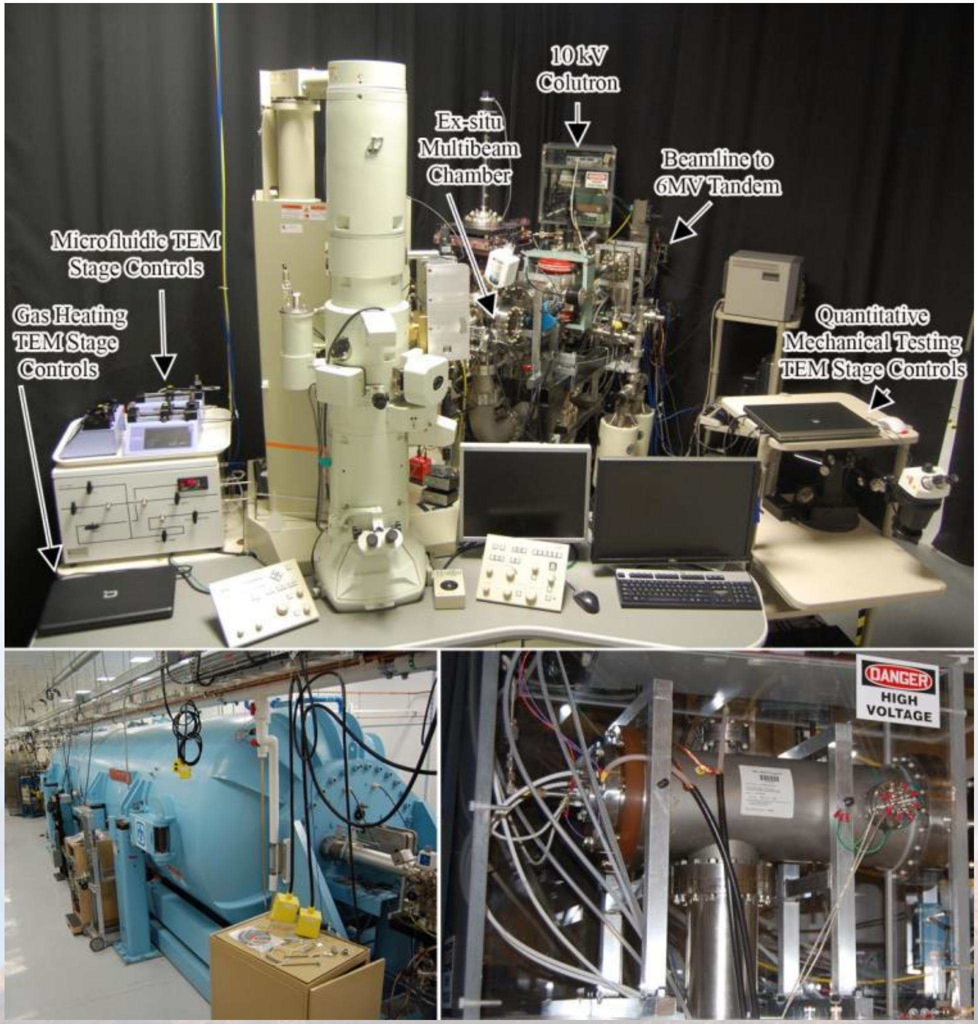
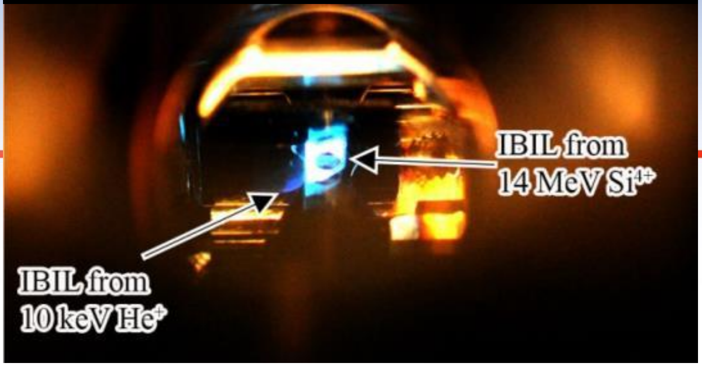
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem

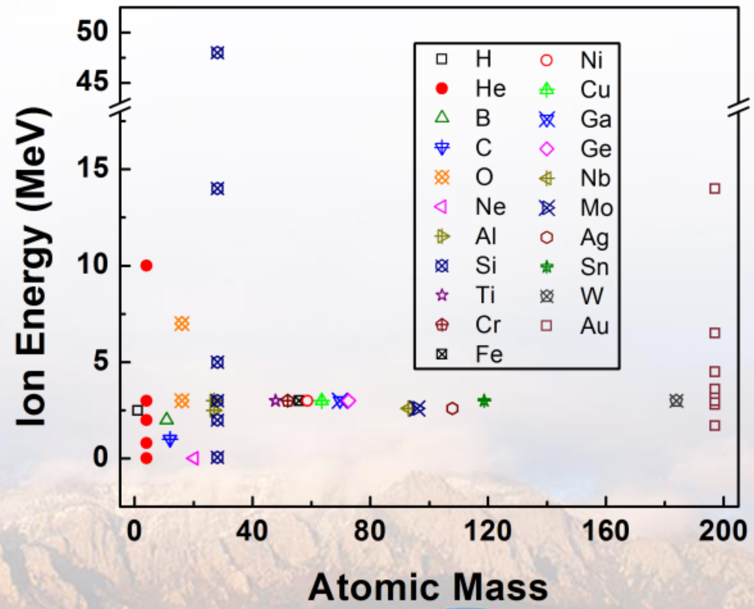


IBIL from a quartz stage inside the TEM



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

Ion species & energy introduced into the TEM



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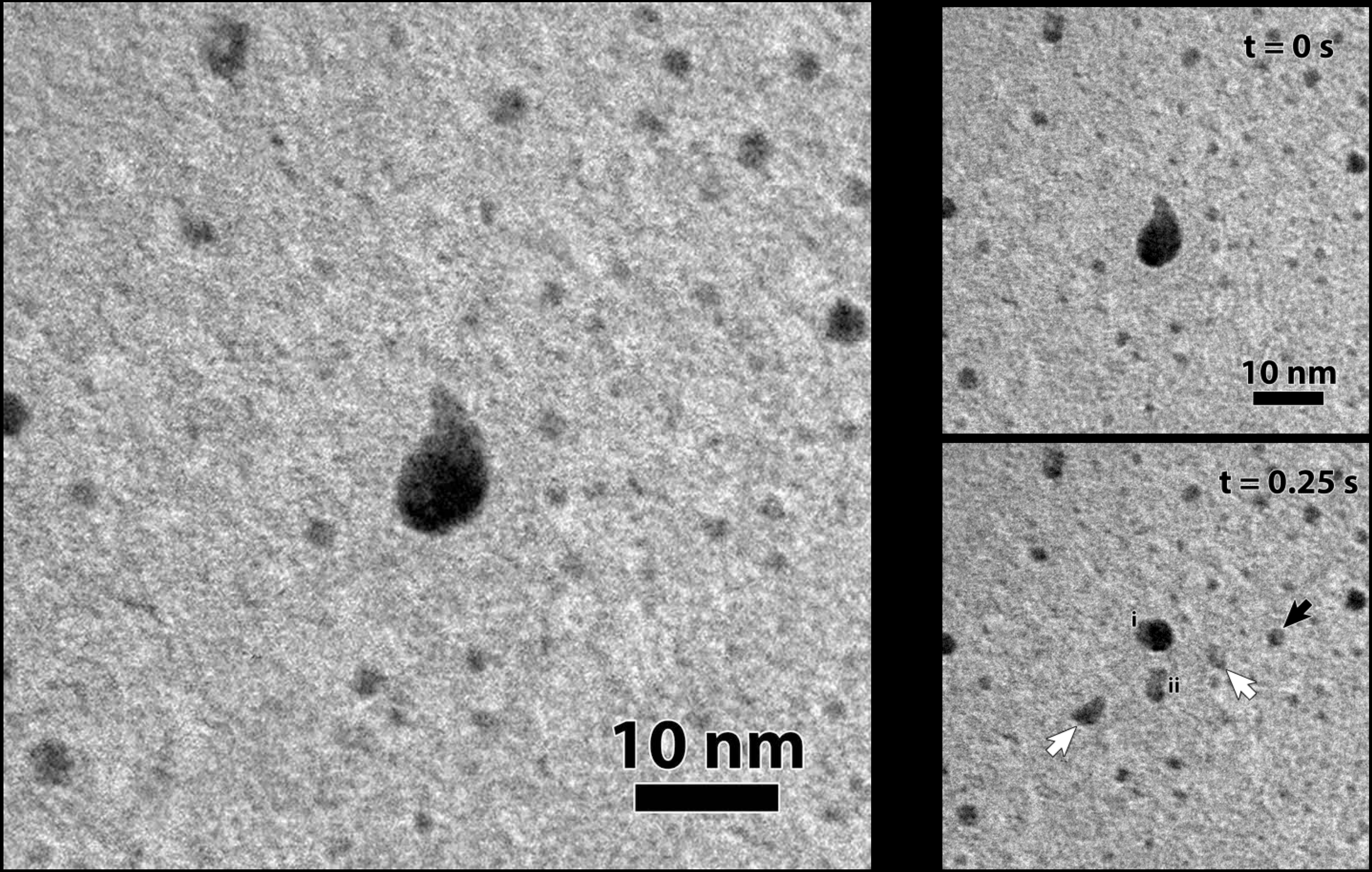
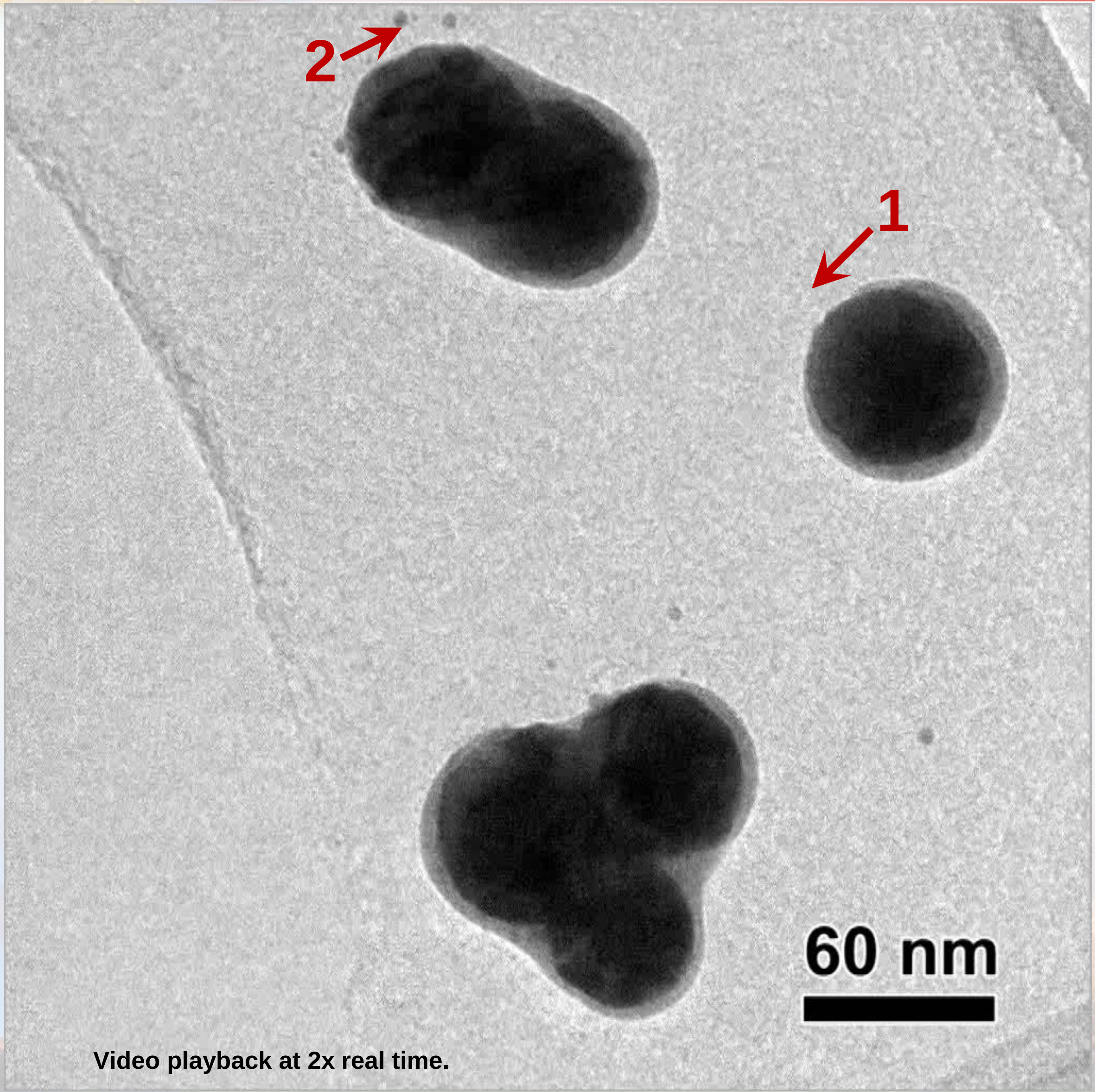
Single Ion Strikes:
46 keV Au^{1-} ions into 5 nm Au nanoparticles

Collaborator: D.C. Bufford



Single Ion Strikes: 2.8 MeV Au^{4+} ions into 60 nm Au nanoparticles

Collaborator: D.C. Bufford

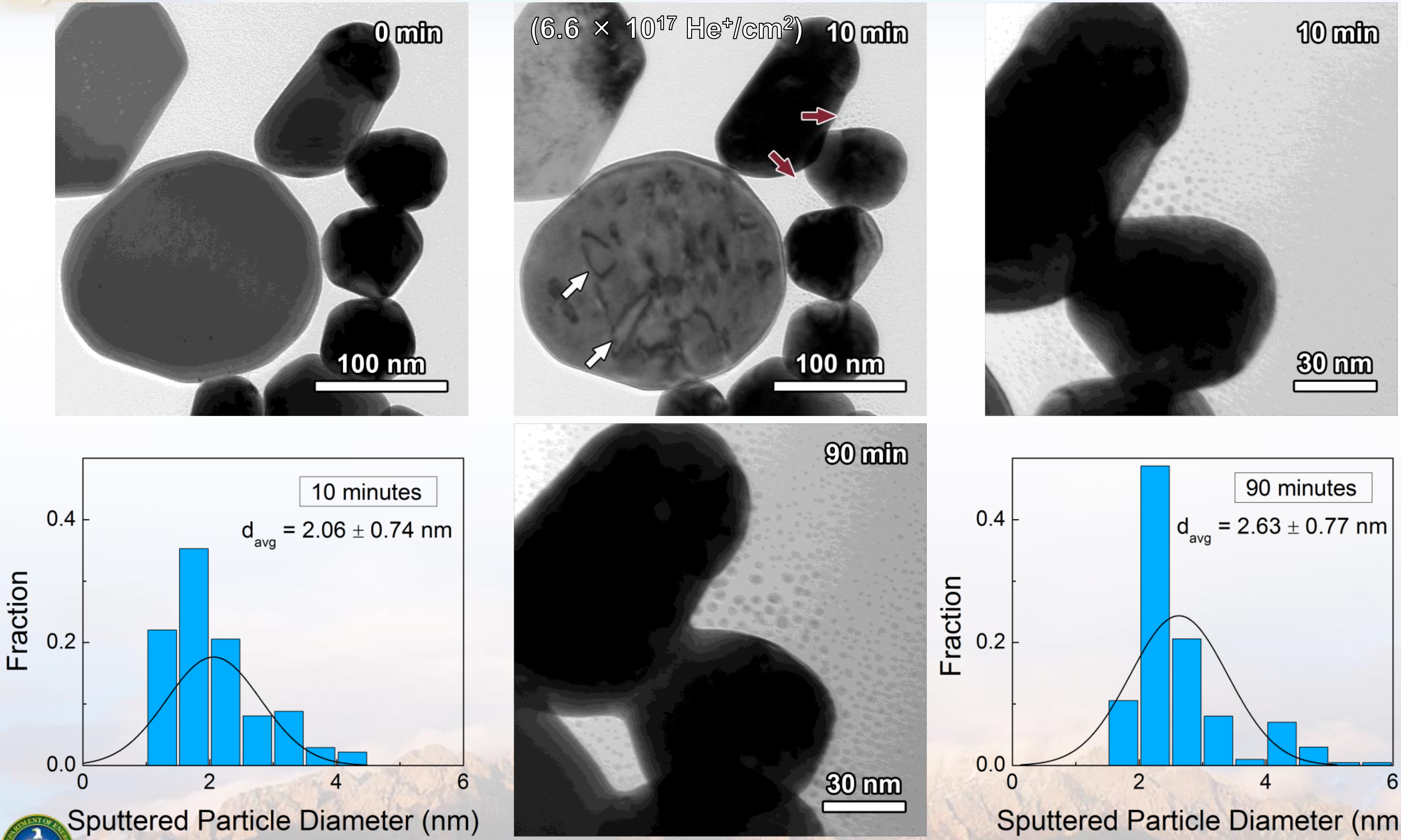


- 2.8 MeV Au^{4+} ions into 60 nm diameter Au nanoparticles
- 100 kx magnification
- Nanoscale filaments created by individual ions

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

Formation of Dislocation Loops & Sputtered Particles due to He implantation

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

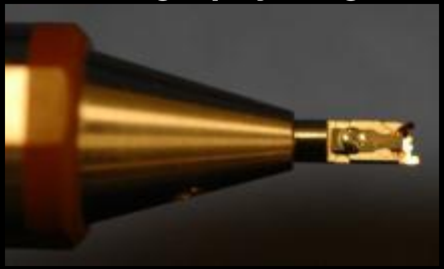


Electron Tomography Provides 3D Insight

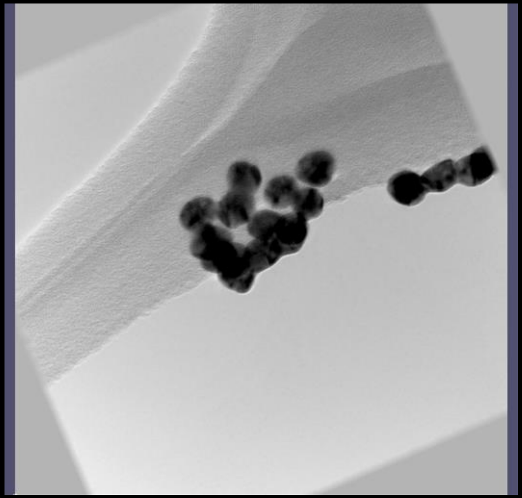
Collaborators: S.H. Pratt & T.J. Boyle

In situ **Ion Irradiation TEM (I^3TEM)**

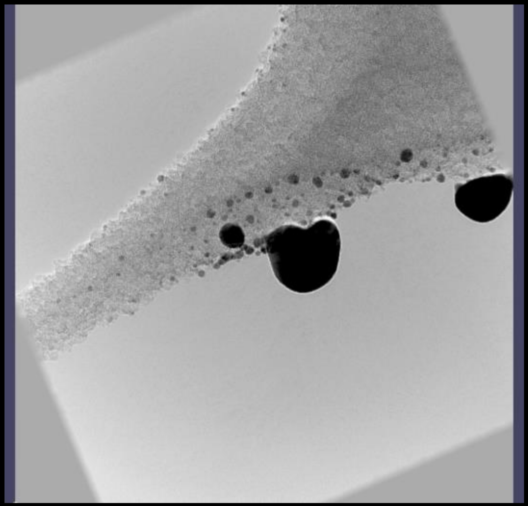
Hummingbird tomography stage



Aligned Au NP tilt series - unirradiated

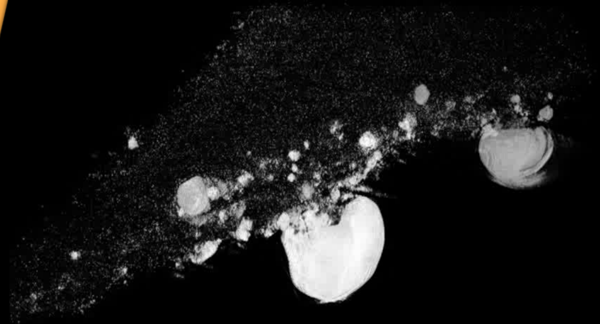


Aligned Au NP tilt series - irradiated



Unirradiated Au NP model

Irradiated Au NP model

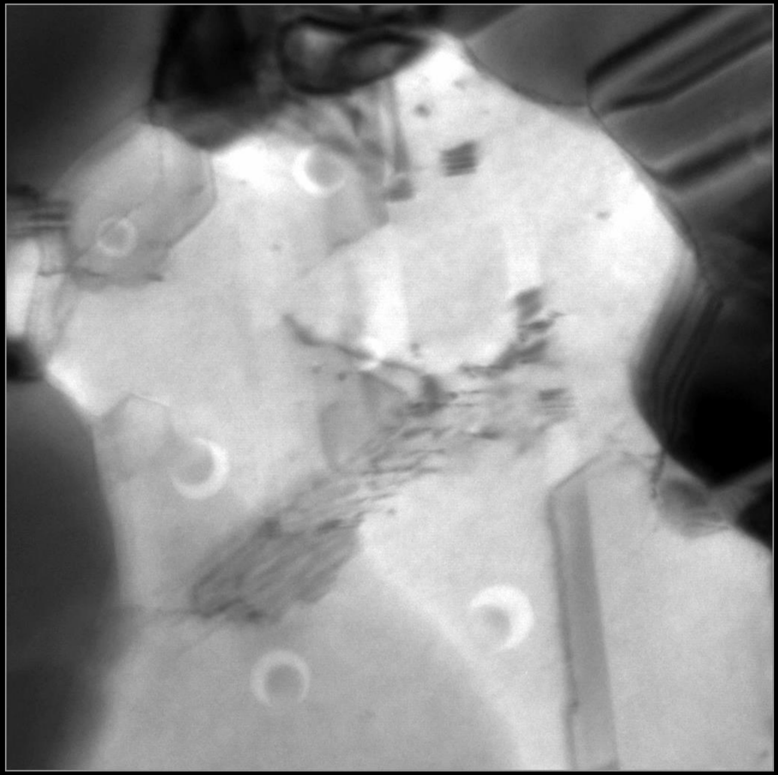


The application of advanced microscopy techniques to extreme environments provides exciting new research directions

Dose Rate Effects

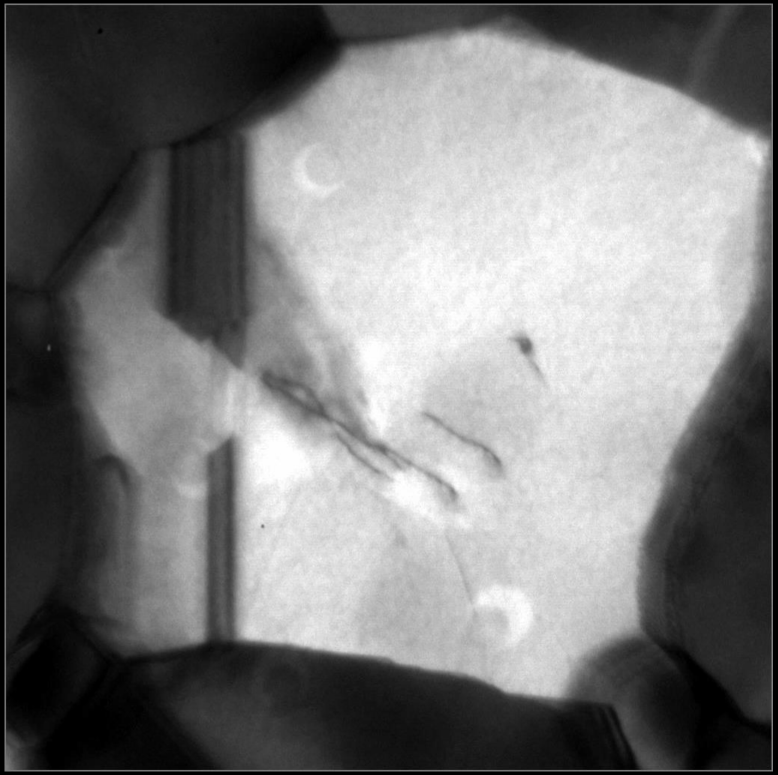
Collaborators: C. Chisholm , P. Hosemann, & A. Minor

7.9×10^9 ions/cm^2/s



VS

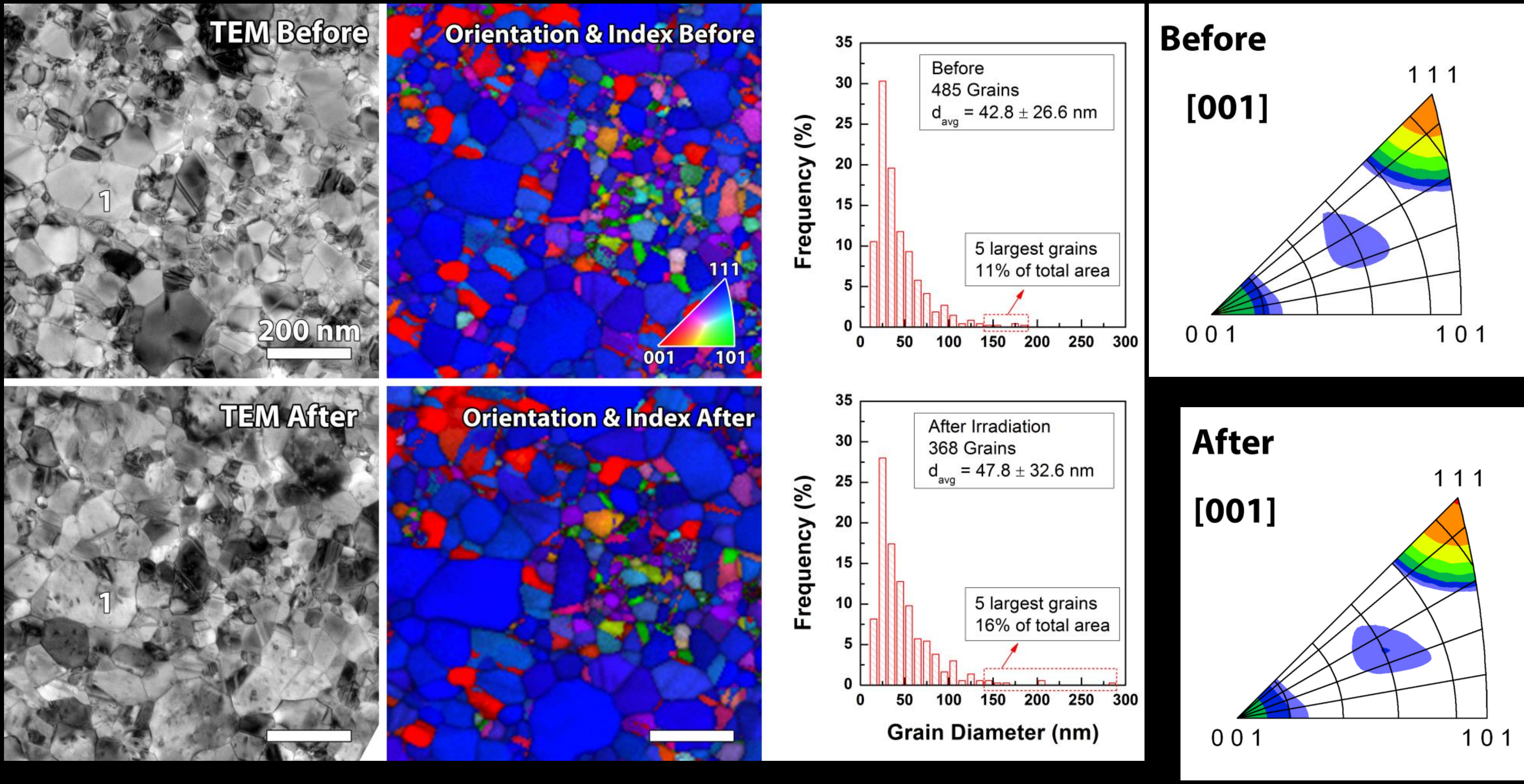
6.7×10^7 ions/cm^2/s



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

Quantifying Stability of Nanocrystalline Au during 10 MeV Si Ion Irradiation

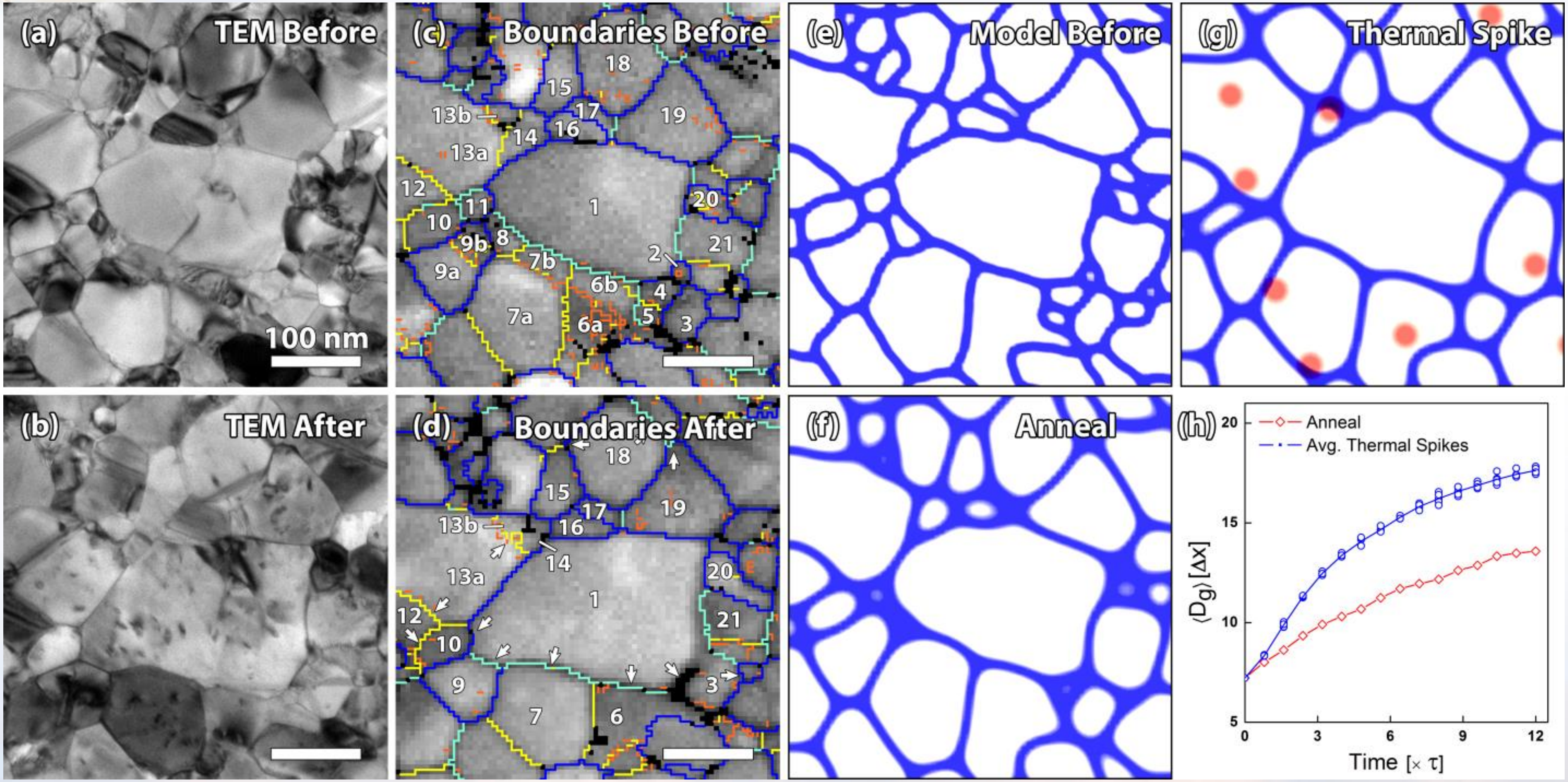
Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles



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 of mesoscale models & subsequent structural evolution can be directly compared.

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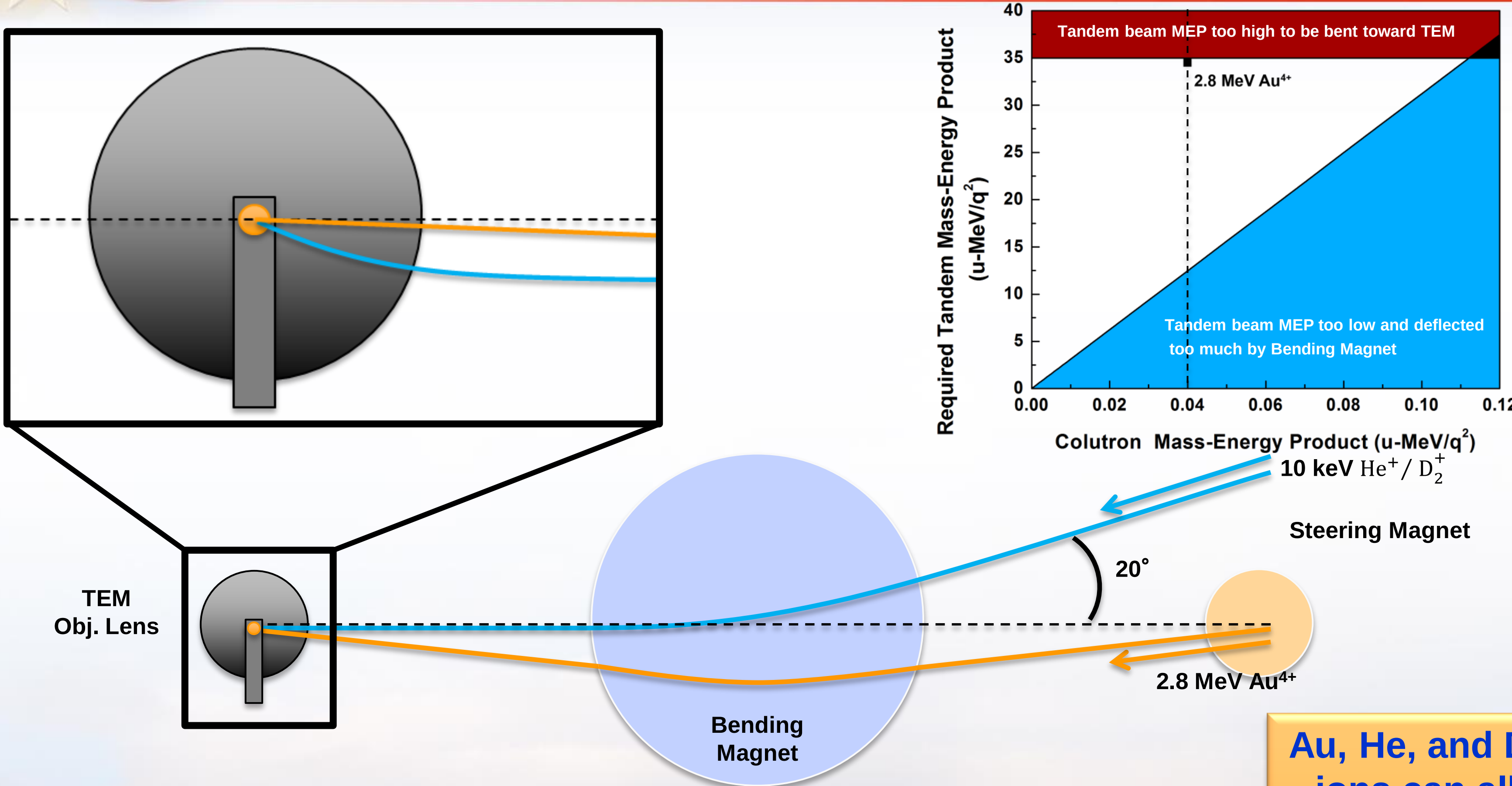
Modeling Beam Mixing and Deflection Necessary to Develop a *In situ* Triple Beam Facility

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



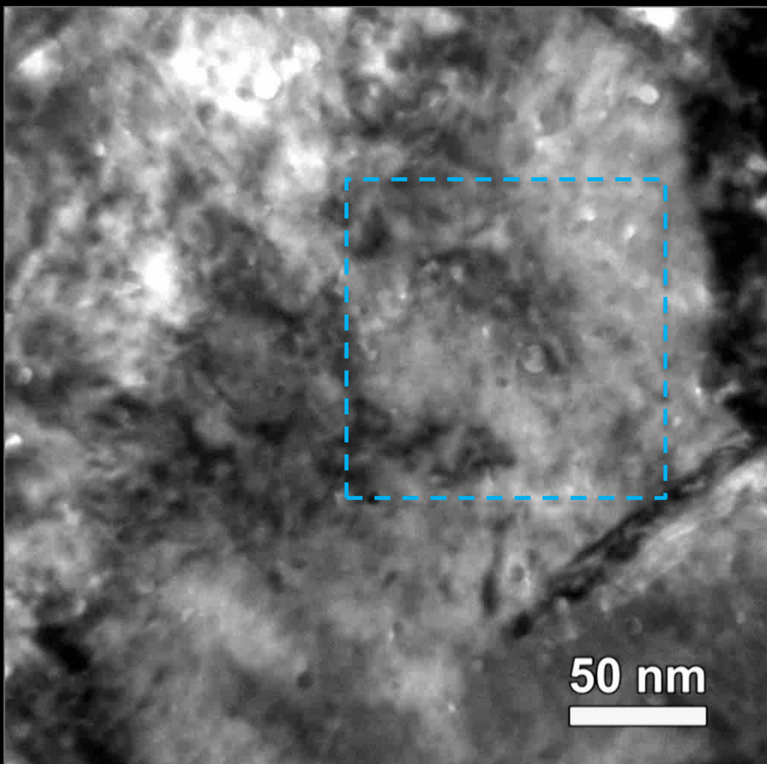
- Must compensate for deflection of Tandem beam by bending magnet Colutron beams deflected by the TEM objective lens
- Insignificant deflection of Tandem beams
- With 10 keV He/D_2 we can use Tandem beams ≳13 MeV/q^2

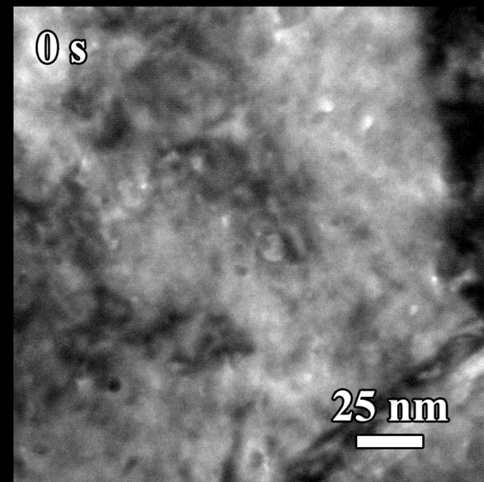
Au, He, and D_2 ions can all reach the sample concurrently

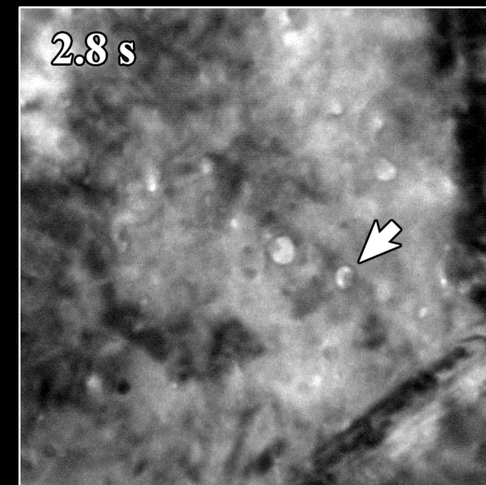
Simultaneous *In situ* TEM Triple Beam: 2.8 MeV Au^{4+} + 10 keV He^{+}/D_2^{+}

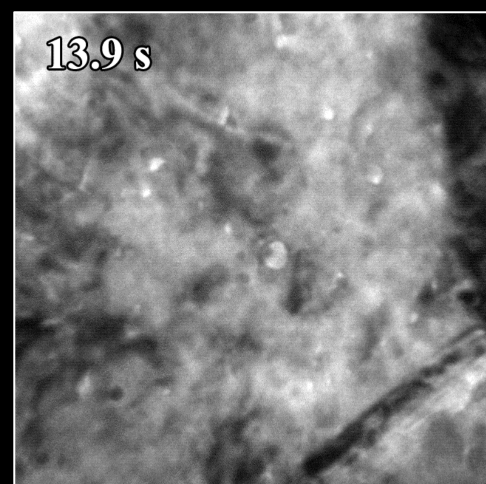
Collaborator: D.C. Bufford

Video playback speed x1.5.









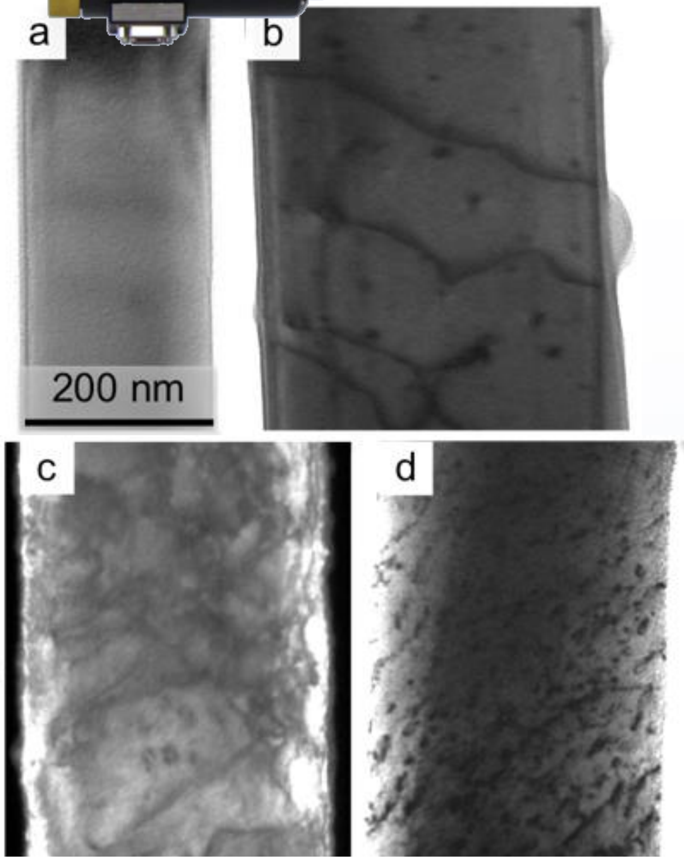
In-situ triple beam He, D_2, and Au beam irradiation has been demonstrated on Sandia's I^3TEM!

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

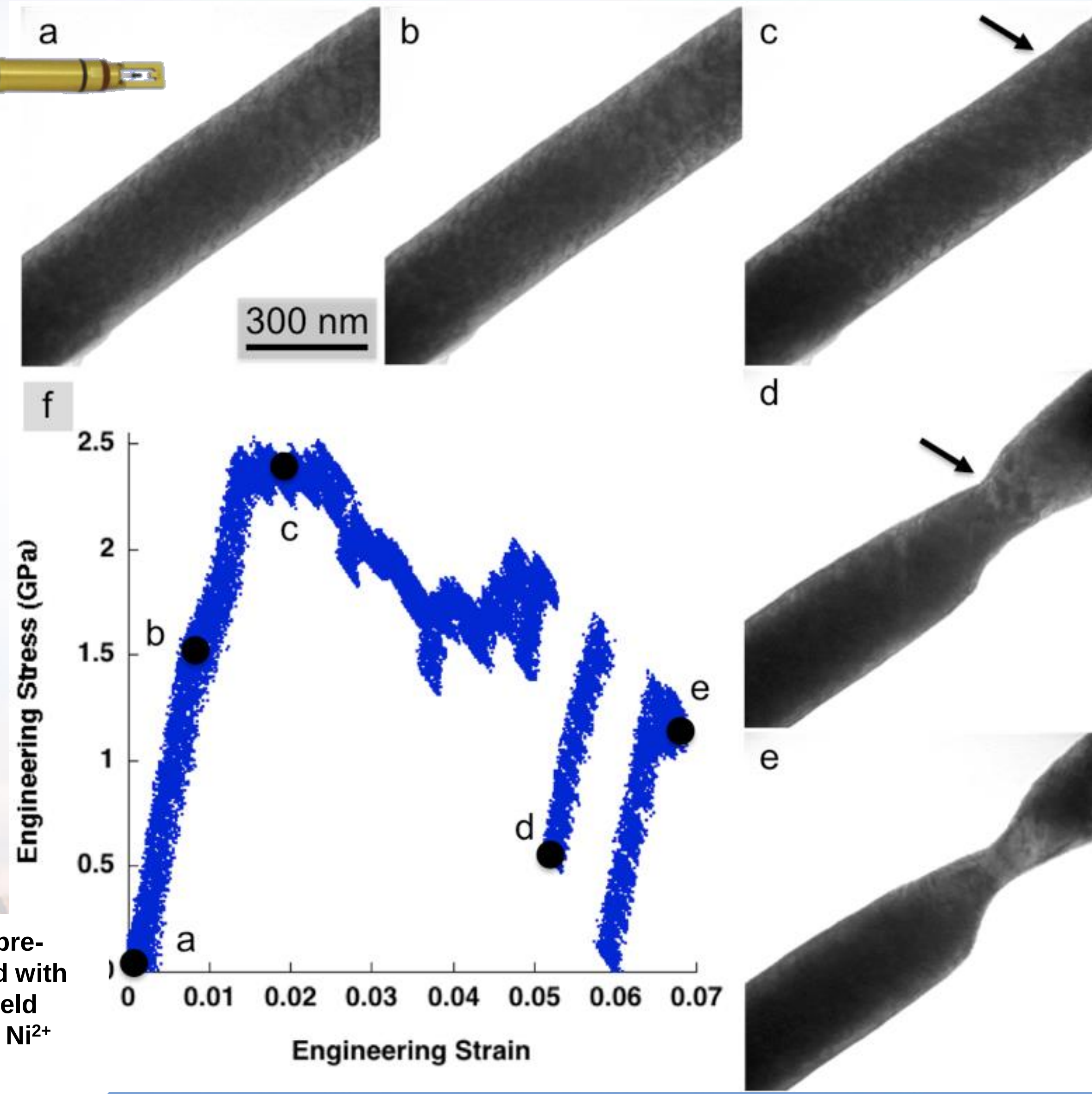
- **Approximate fluence:**
  - Au 1.2×10^{13} ions/cm^2
  - He 1.3×10^{15} ions/cm^2
  - D 2.2×10^{15} ions/cm^2
- **Cavity nucleation and disappearance**

In situ TEM Quantitative Mechanical Testing

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

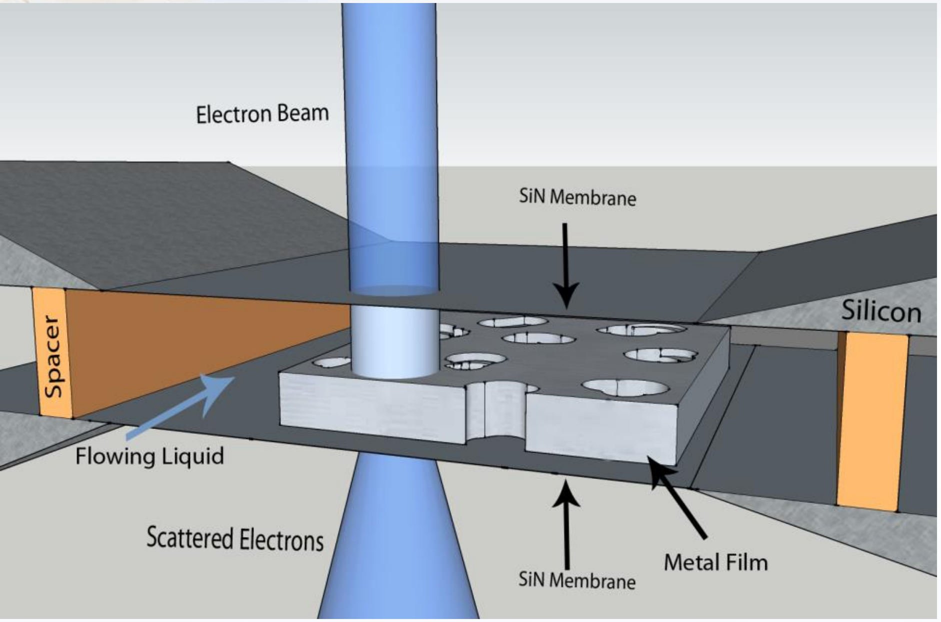


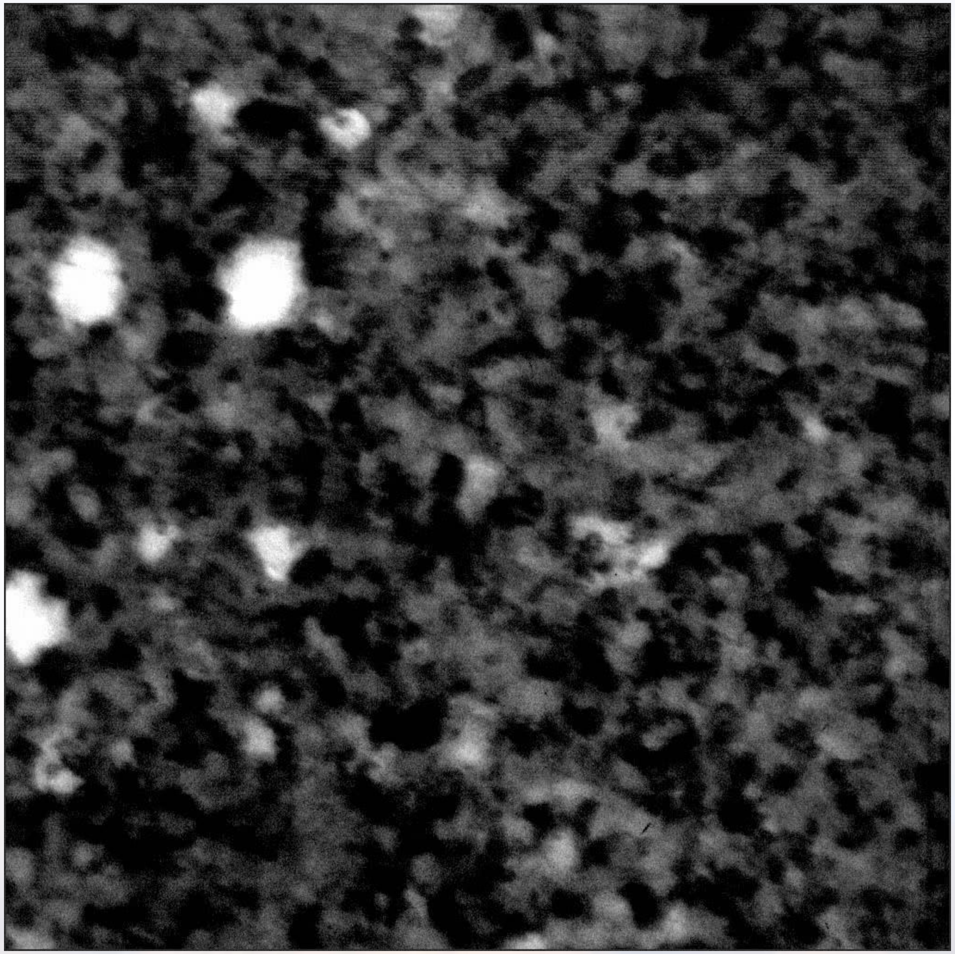
TEM micrographs of Mo-alloy nanofibers. (a) 0% pre-strained, no observable defects. (b) 9% pre-strained with mobile dislocations and sessile loops. (c) Dark-field micrograph of 15% pre-strained fiber. (d) He^{1+} and Ni^{2+} irradiated, 9% pre-strained fiber.



In situ TEM Corrosion

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





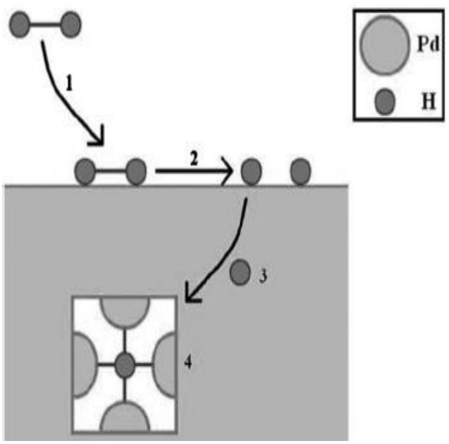
Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.

Microfluidic Stage

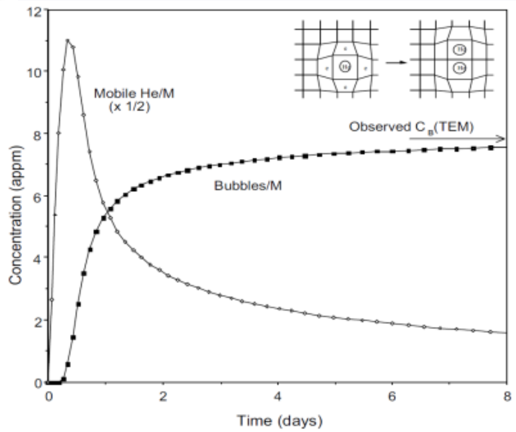
- **Mixing of two or more channels**
- **Continuous observation of the reaction channel**
- **Chamber dimensions are controllable**
- **Films can be directly deposited on the electron transparent SiN membrane**

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.



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

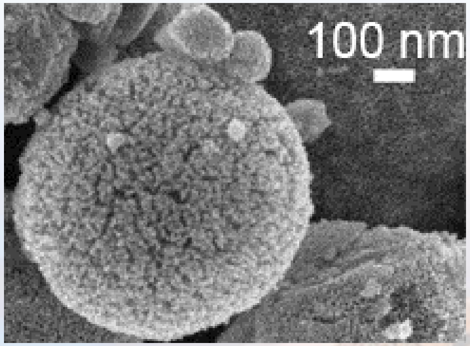


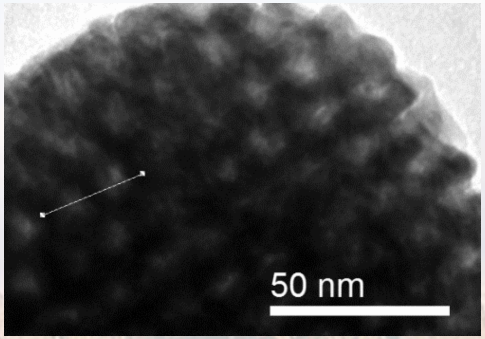
Cowgill, D., Fusion Sci. & Tech., 28 (2005) p. 539
Trinkaus, H. *et al*., JNM (2003) p. 229
Thiebaut, S. *et al*. JNM (2000) p. 217

Vapor-Phase Heating TEM Stage

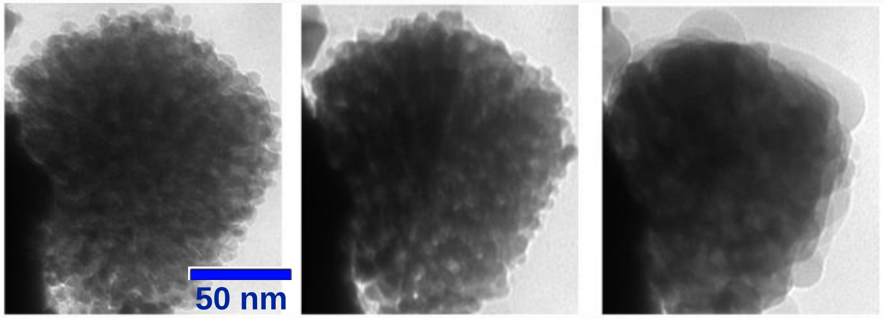
- Compatible with a range of gases
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Compatible with MS and other analytical tools

Harmful effects may be mitigated in nanoporous Pd





- 1 atm H_2 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

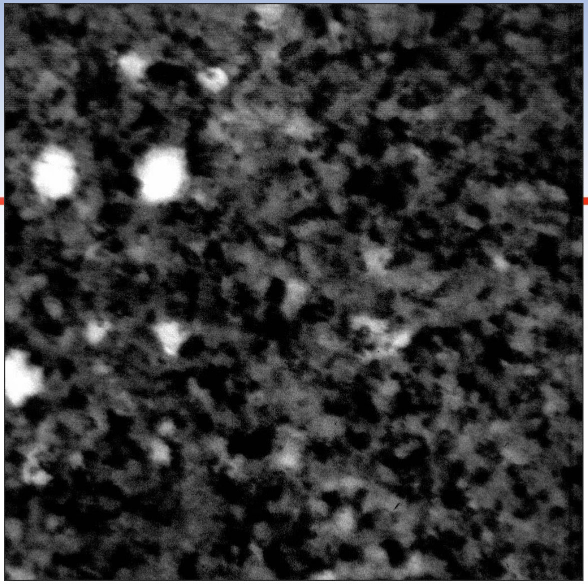
Summary

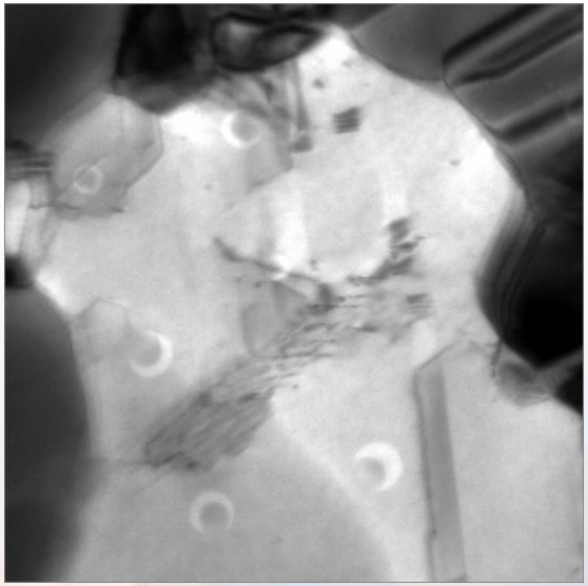
- **Sandia's I[3]TEM is one of only two facilities in the US of this type:**
  - ***In situ*** **high energy ion irradiation from H to Au**
  - ***In situ*** **gas implantation**
  - **Heating up to 1,000 ˚C**
  - **Quantitative and bulk straining**
  - **Two-port microfluidic cell**
  - **Gas flow/heating stage**
  - **Electron tomography**
  - **Precession Electron Diffraction**

Sandia's I[3]TEM although still under development is providing a wealth of interesting initial observations and harsh environments

- **Currently applying the current I[3]TEM capabilities to various material systems in combined and harsh environmental conditions**

- **ThM-5 (11:00-13:15)** ***Room: Mauna Loa "Correlative nanomechanical measurements for complex engineered systems"***
- **ThM-8 (11:00-13:30)** ***Room: Keauhou IV*** **"Correlating grain orientation and grain boundary character to the failure path in nanocrystalline metals"**
- **FM-3 (8:30-10:30)** ***Room: Keauhou III "ultrafine and nanocrystalline metals under extreme heat loading and irradiation conditions"***





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