

In situ TEM Investigations of Overlapping and Harsh Environments

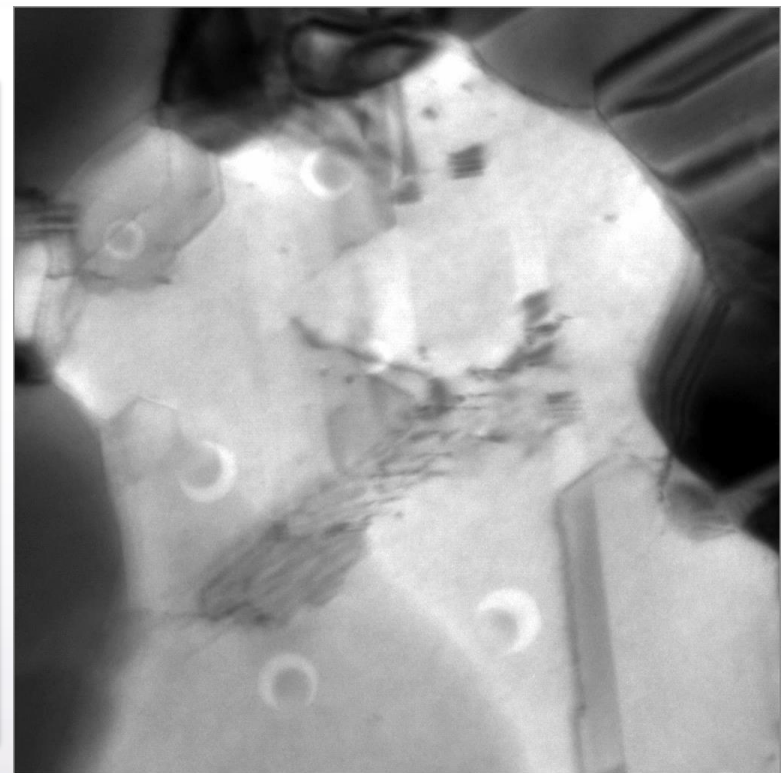
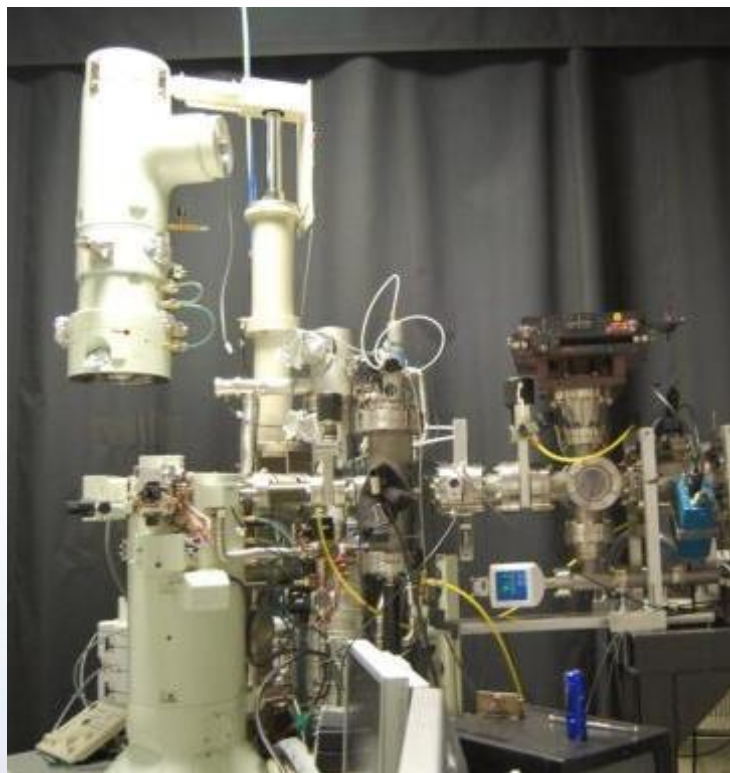
SAND2016-0984PE

K. Hattar

Ion Beam Lab at Sandia National Laboratories

February 10, 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**

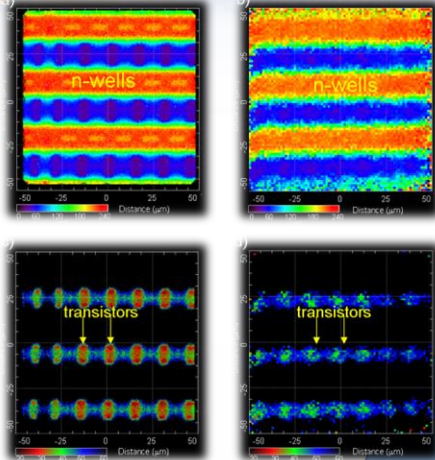
This work was supported by the US Department of Energy, Office of Basic Energy Sciences.

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Sandia National Laboratories

Sandia's Ion Beam Laboratory



Ion Beam Analysis (IBA)

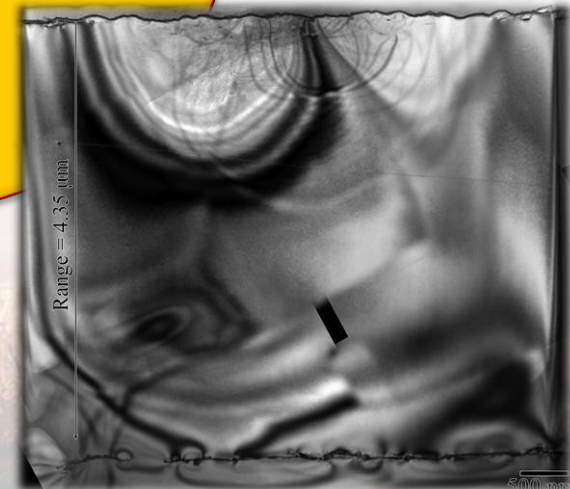
TECHNIQUE	SCHEMATIC	EXAMPLE ANALYSIS SPECTRUM Target (Ino., Det) Residual
RBS		
ERD		
HIBS		
NRA		
PIXE		
SEU/IBICC		

Radiation Effects Microscopy (REM)



Ion Beam Modification (IBM)

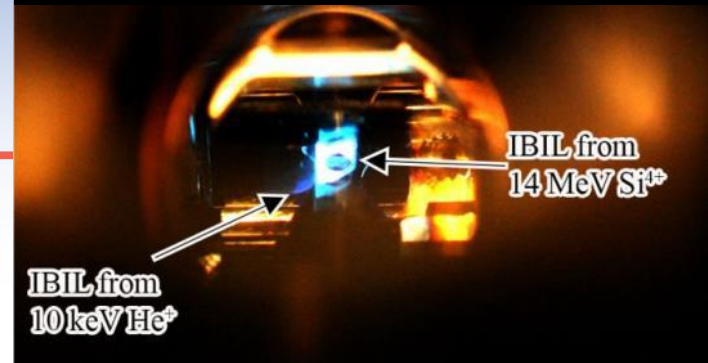
In situ Ion Irradiation Transmission Electron Microscopy (I³TEM)



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

Collaborator: D.L. Buller

IBIL from a quartz stage inside the TEM

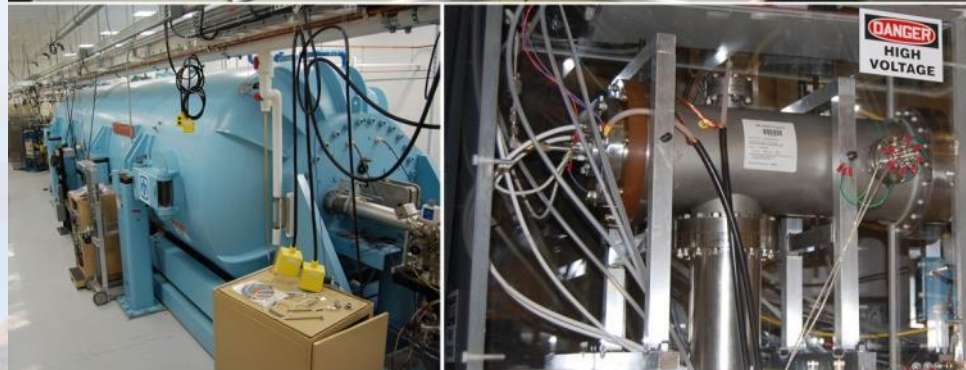
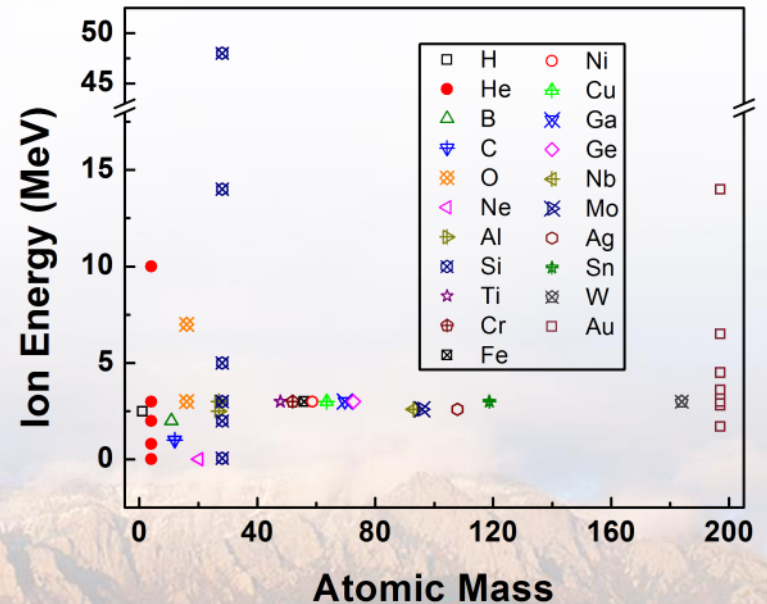


Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution

10 kV Colutron - 200 kV TEM - 6 MV Tandem



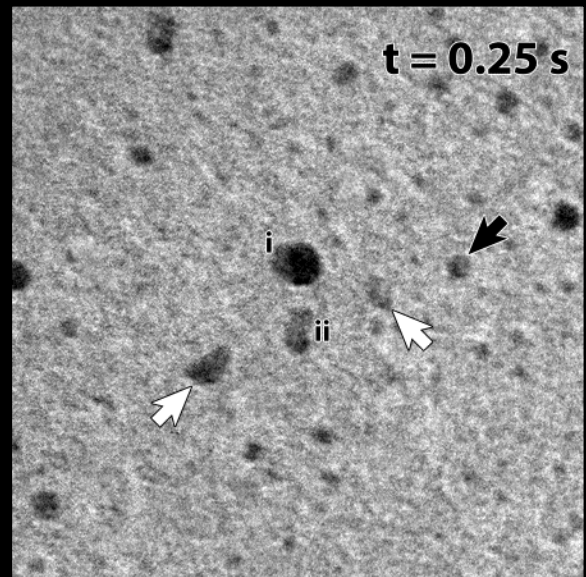
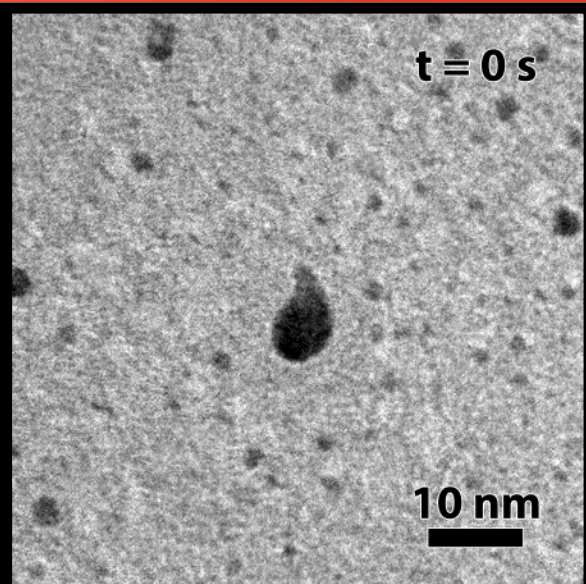
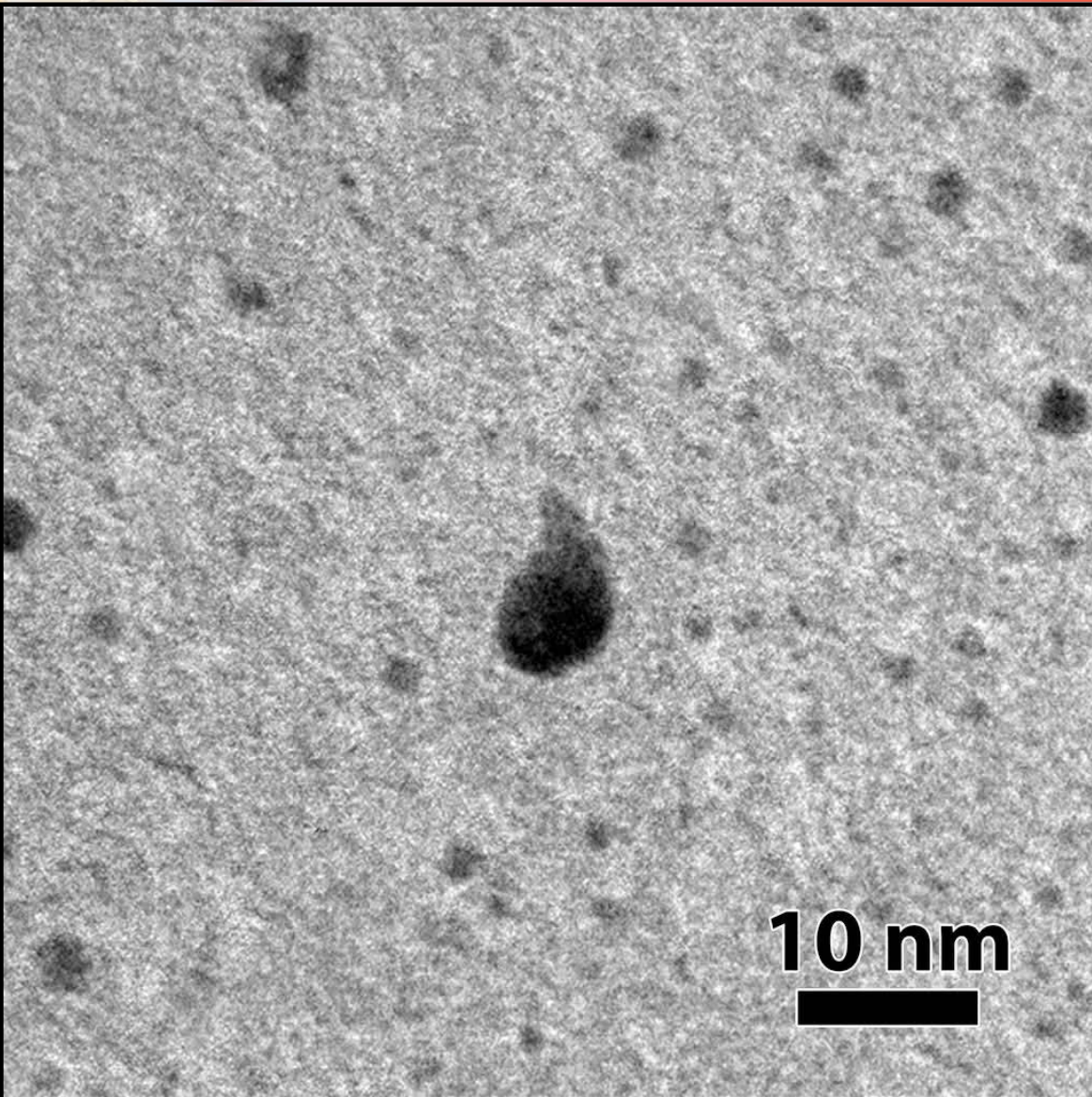
Ion species & energy introduced into the TEM



Single Ion Strikes:

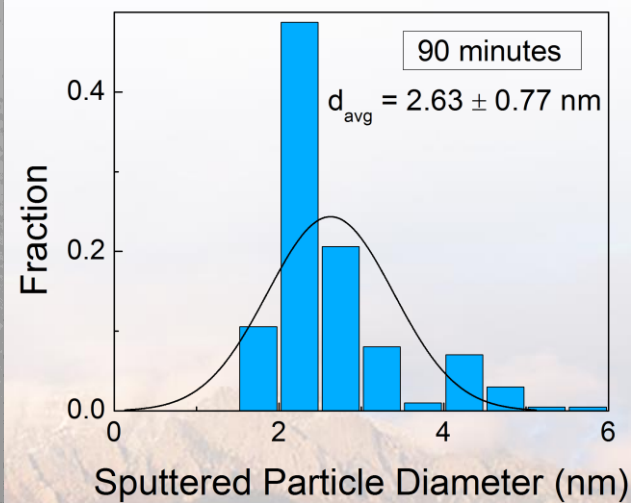
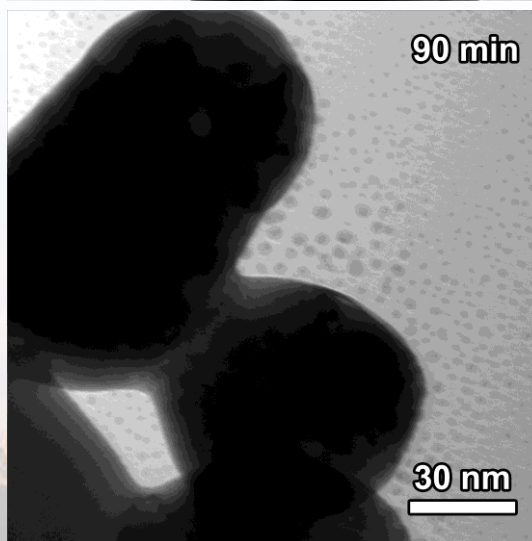
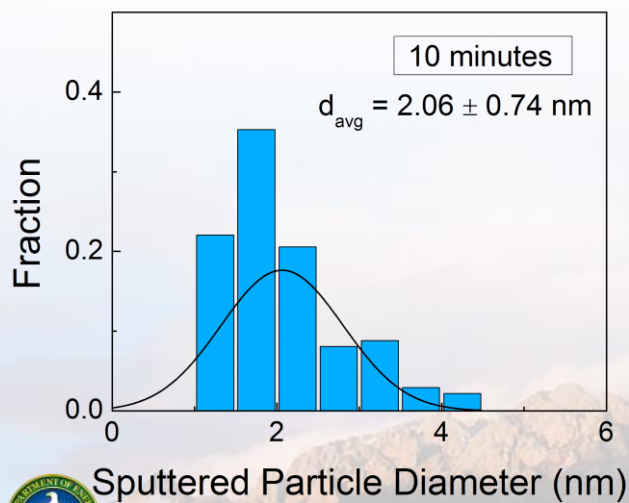
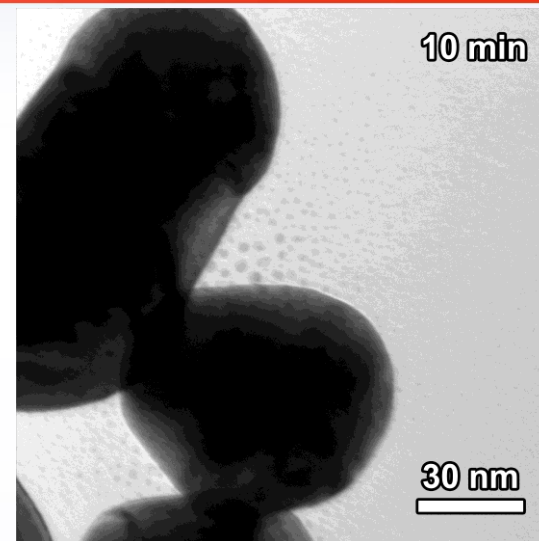
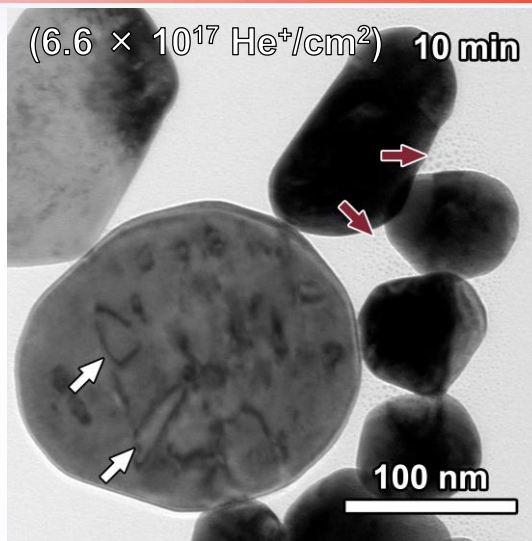
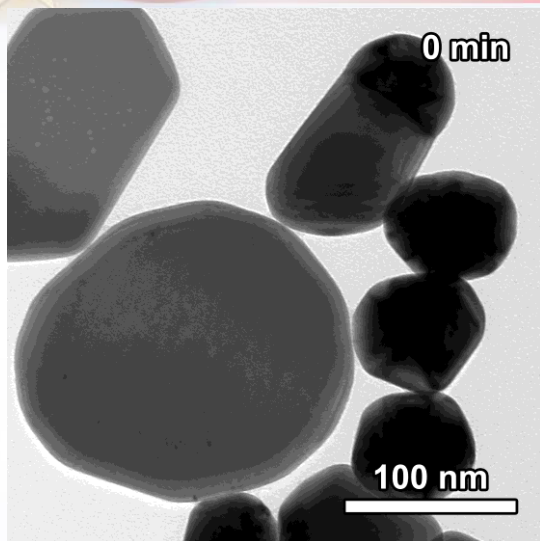
46 keV Au¹⁺ ions into 5 nm Au nanoparticles

Collaborator: D.C. Bufford



Formation of Dislocation Loops & Sputtered Particles due to He implantation

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



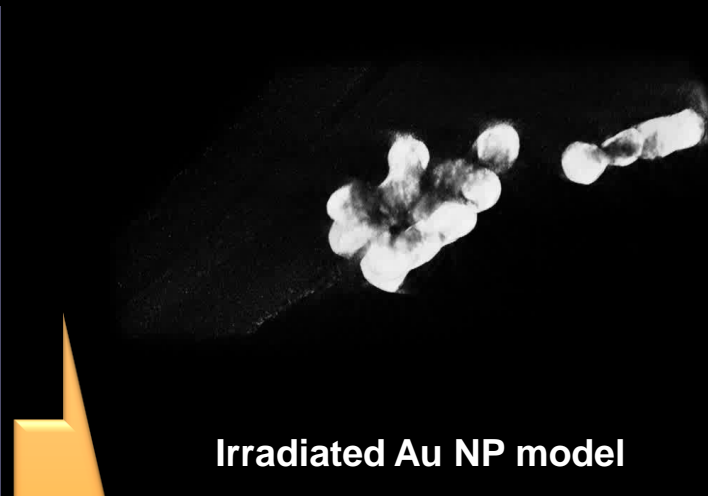
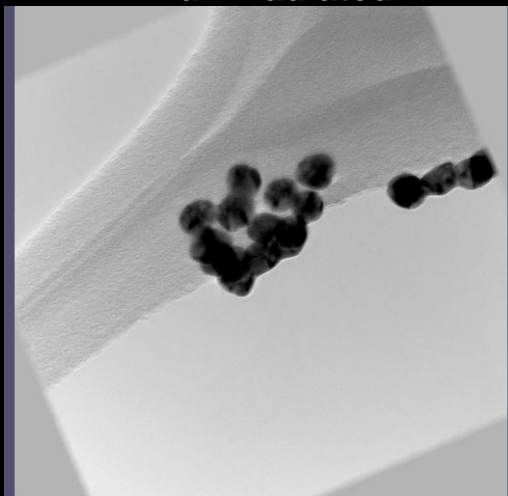
Electron Tomography Provides 3D Insight

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

In situ Ion Irradiation TEM (I³TEM)

Aligned Au NP tilt series -
unirradiated

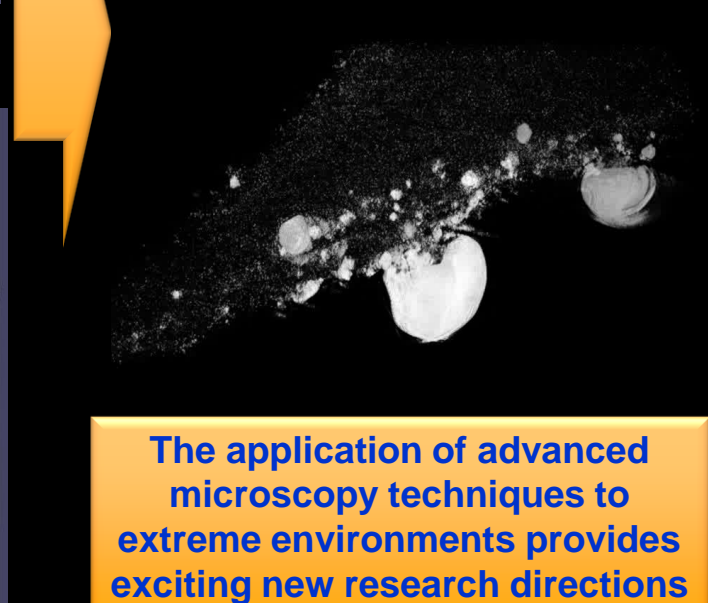
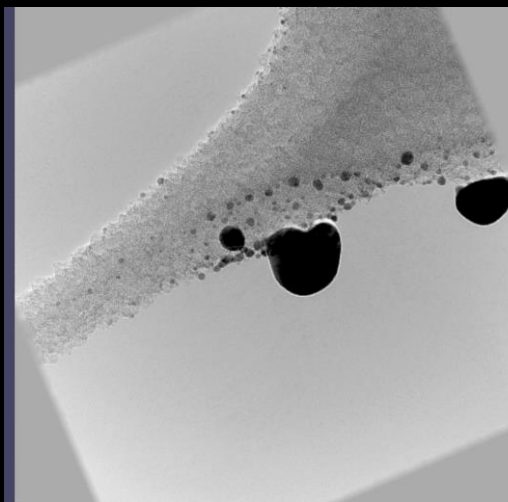
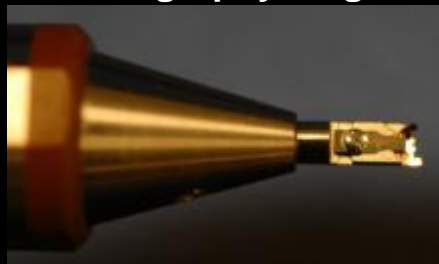
Unirradiated Au NP model



Hummingbird
tomography stage

Aligned Au NP tilt series -
irradiated

Irradiated Au NP model

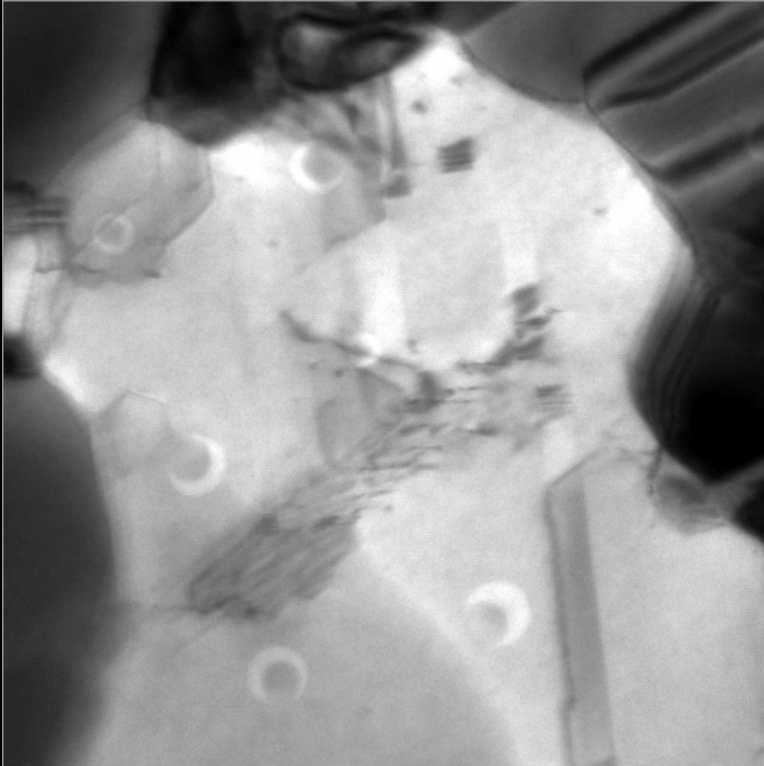


The application of advanced
microscopy techniques to
extreme environments provides
exciting new research directions

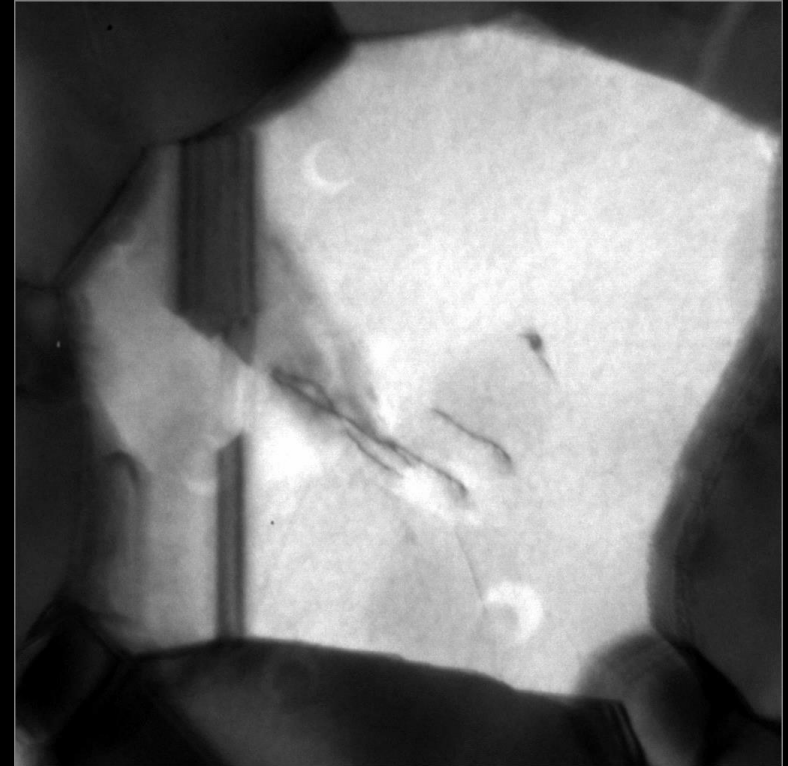
Dose Rate Effects

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

7.9×10^9 ions/cm²/s



6.7×10^7 ions/cm²/s

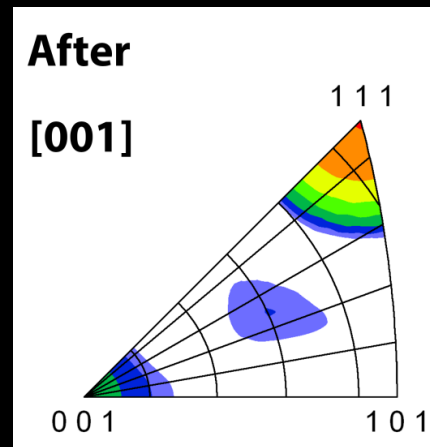
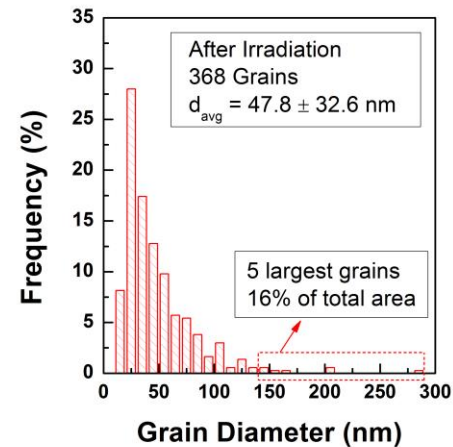
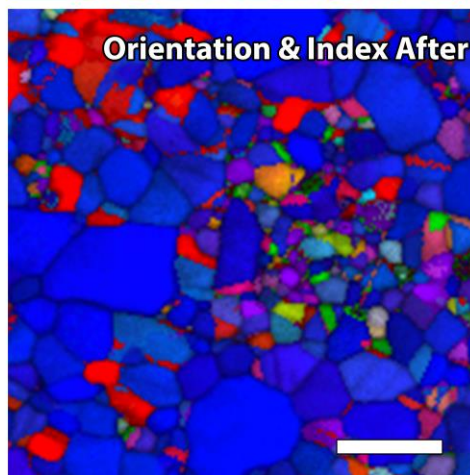
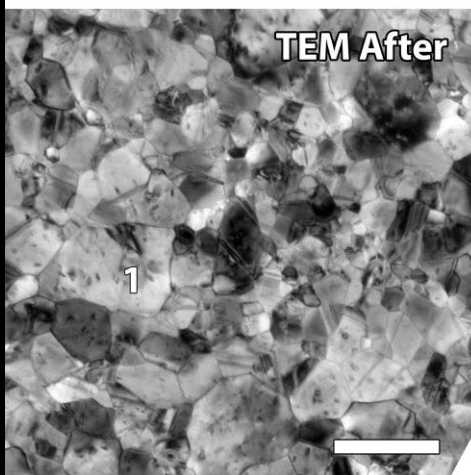
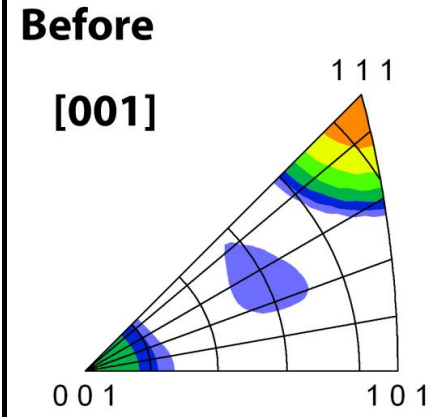
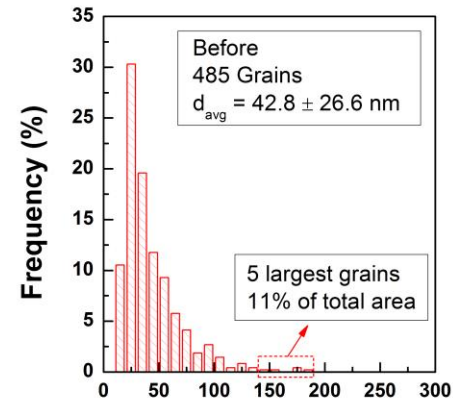
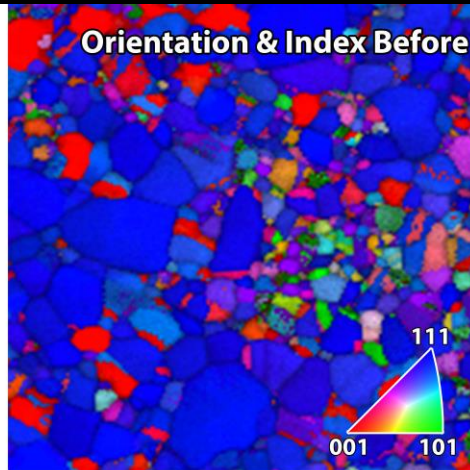


VS

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

Quantifying Stability of Nanocrystalline Au during 10 MeV Si Ion Irradiation

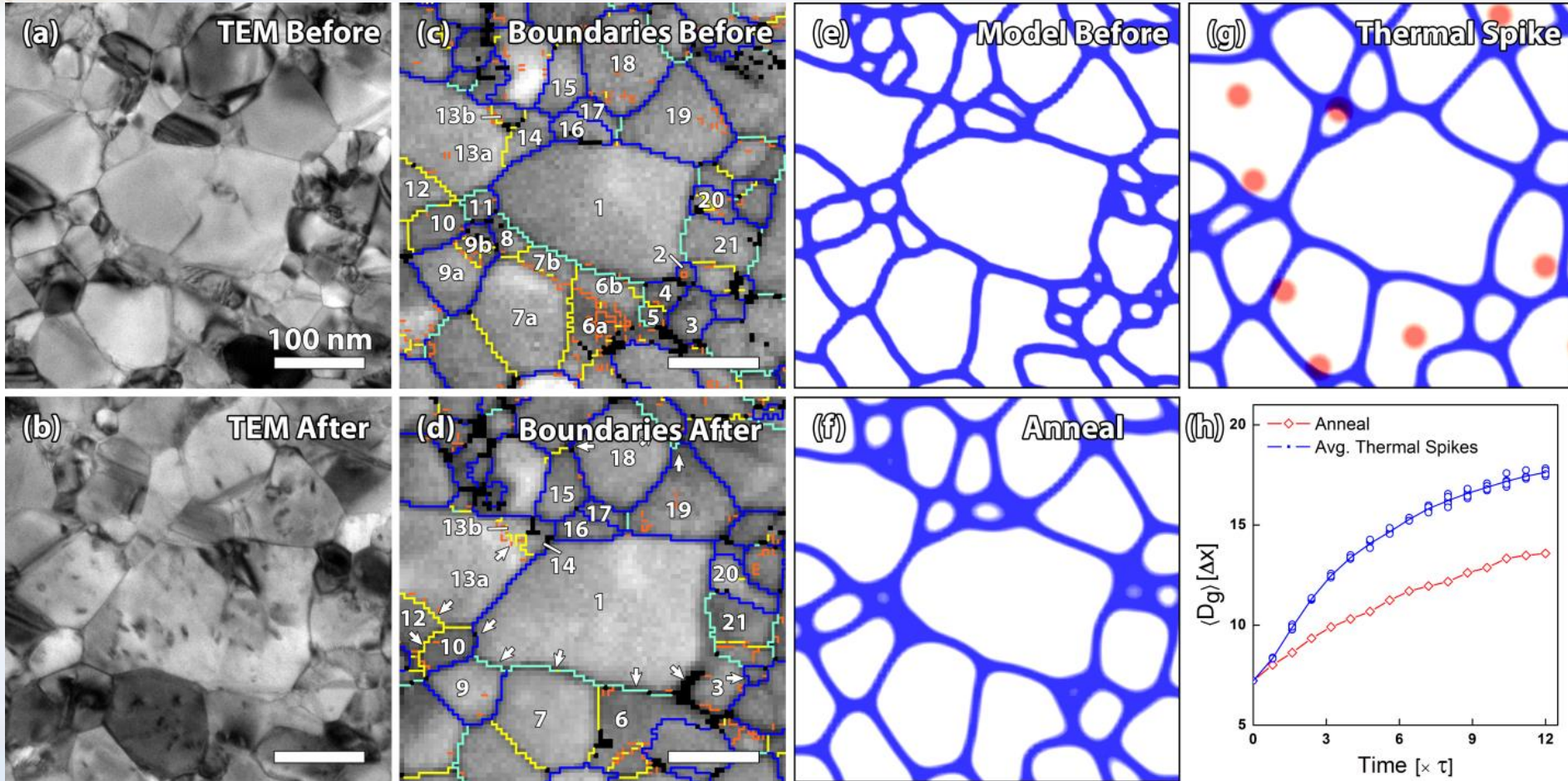
Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles



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



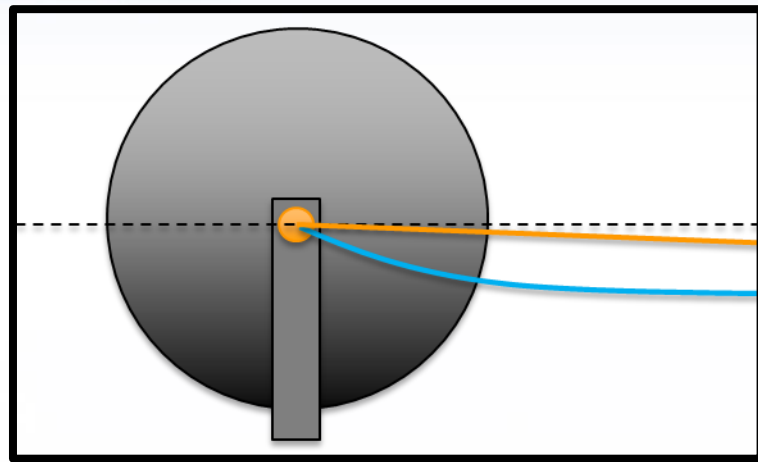
Because of the matching length scale, the initial microstructure can serve as direct input to either MD or mesoscale models & subsequent structural evolution can be directly compared.



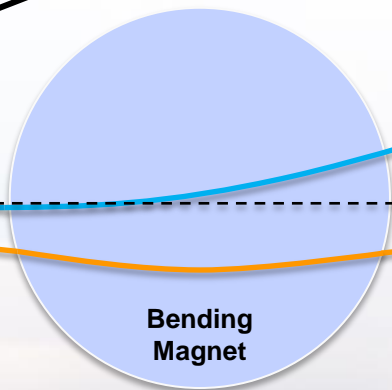
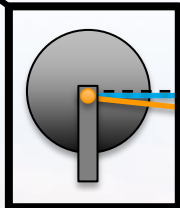
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Modeling Beam Mixing and Deflection Necessary to Develop a *In situ* Triple Beam Facility

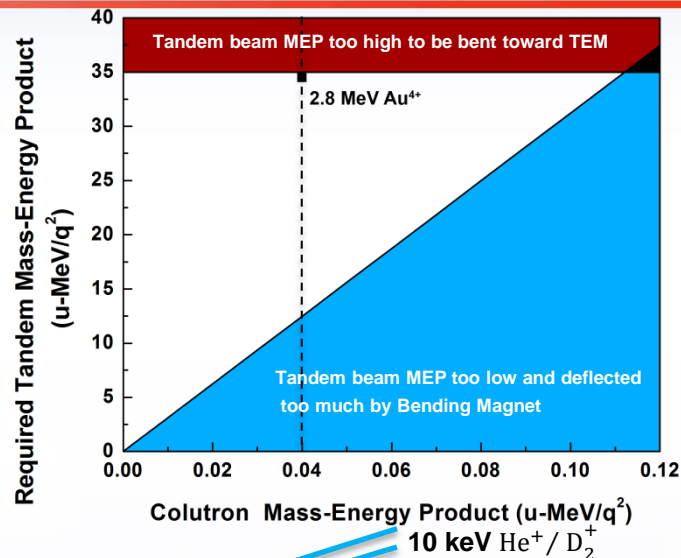
Collaborators: M. Steckbeck, D.C. Bufford, & B.L. Doyle



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



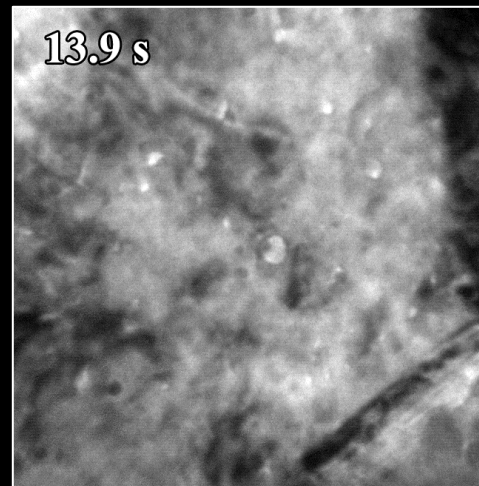
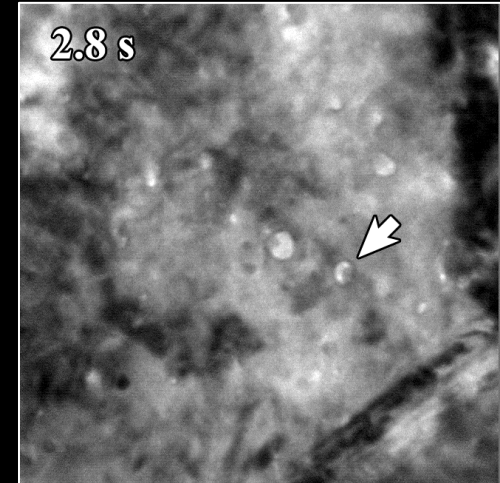
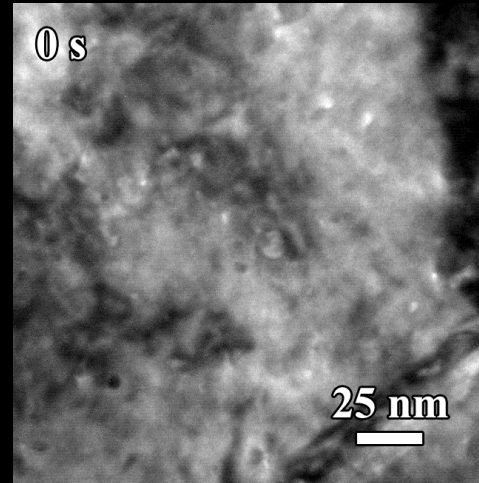
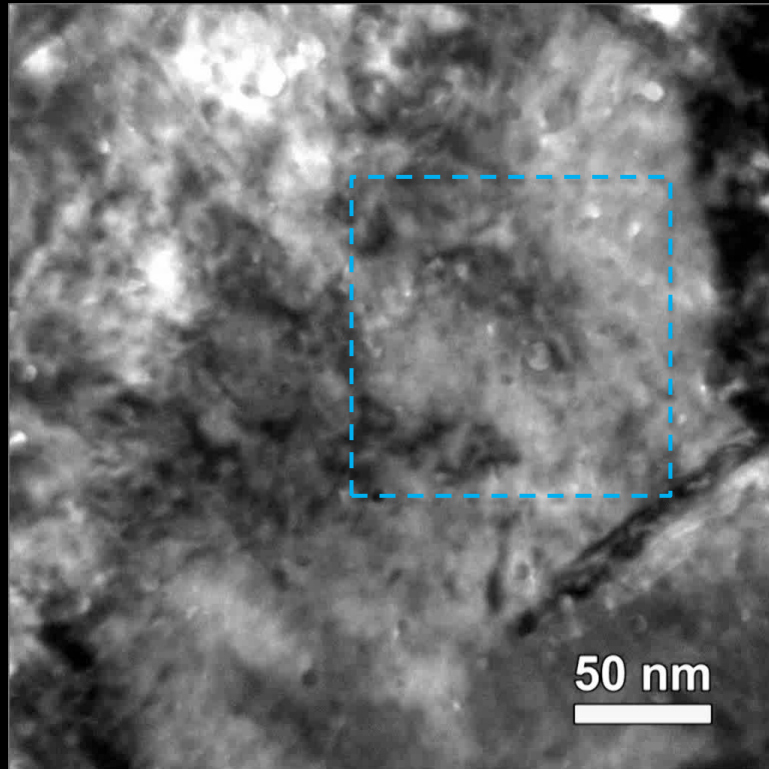
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Simultaneous *In situ* TEM Triple Beam:

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

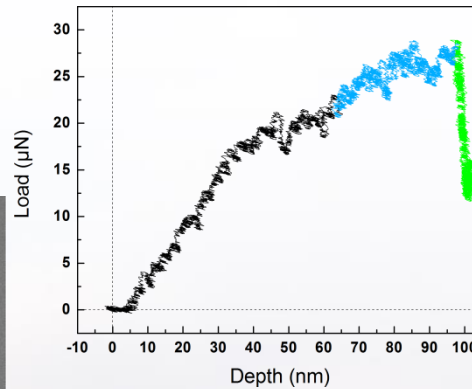
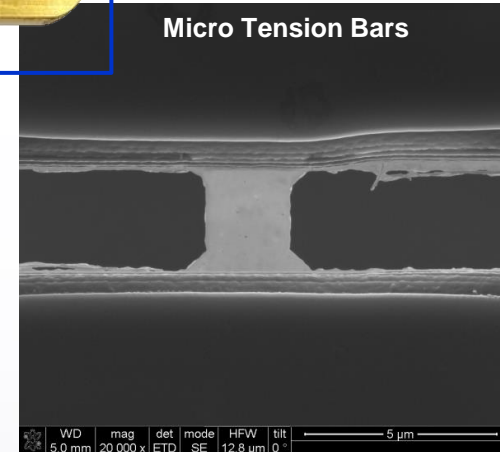
In situ Quantitative Mechanical Testing



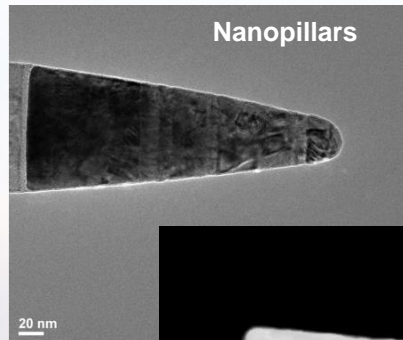
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

- Sub nanometer displacement resolution
- Quantitative force information with μN resolution
- **Concurrent real-time imaging by TEM**

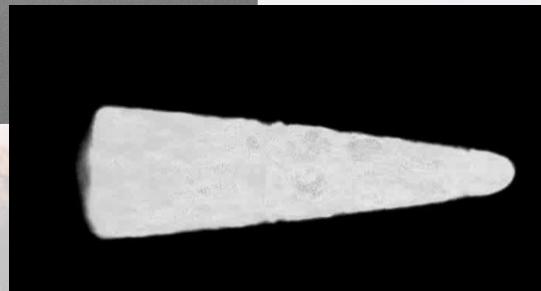
Micro Tension Bars



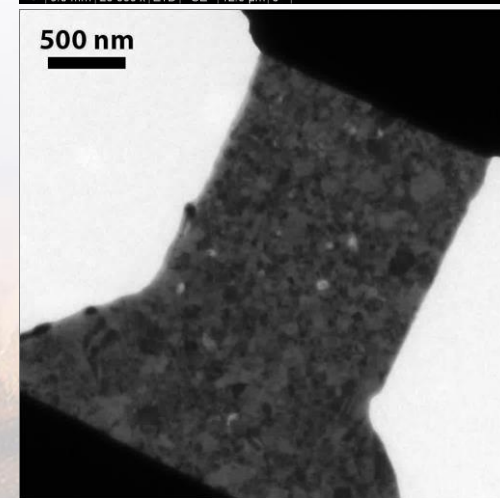
Nanoindentation



Nanopillars

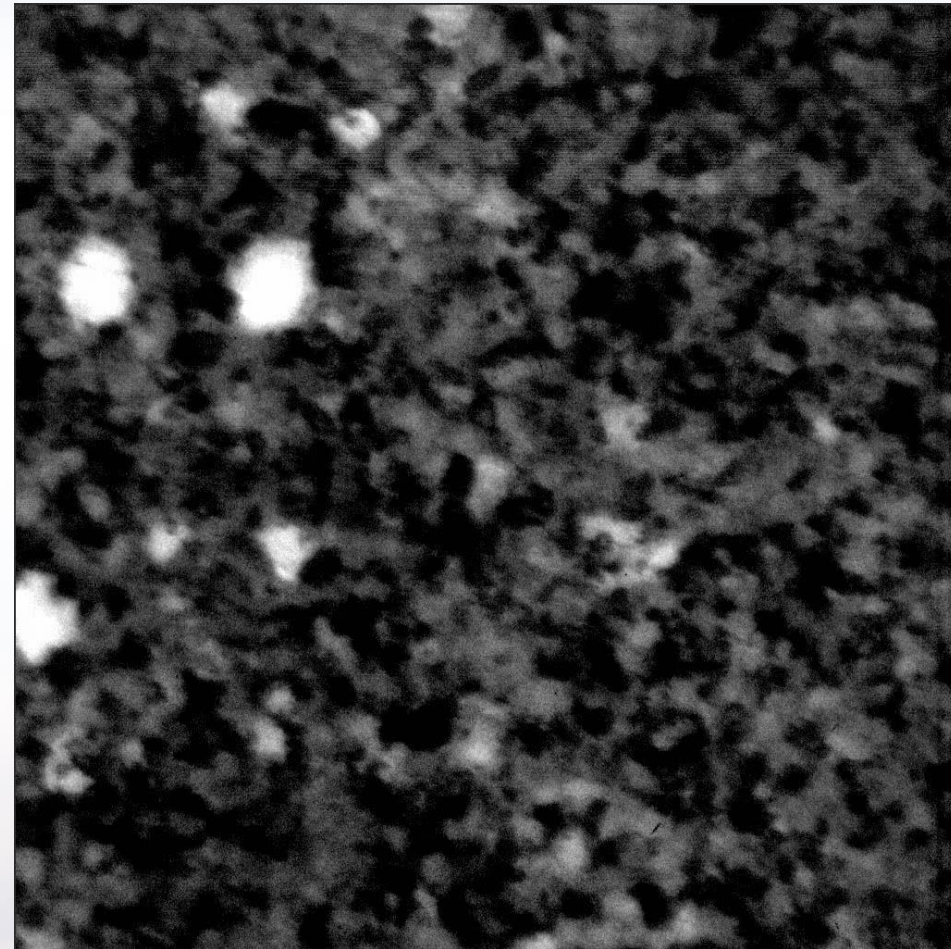
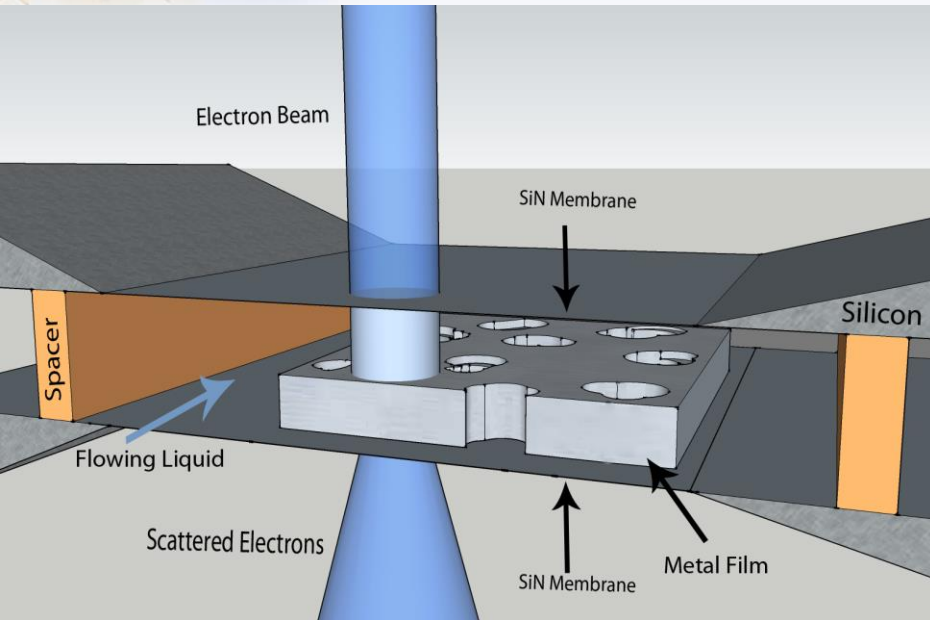


500 nm



In situ TEM Corrosion

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.

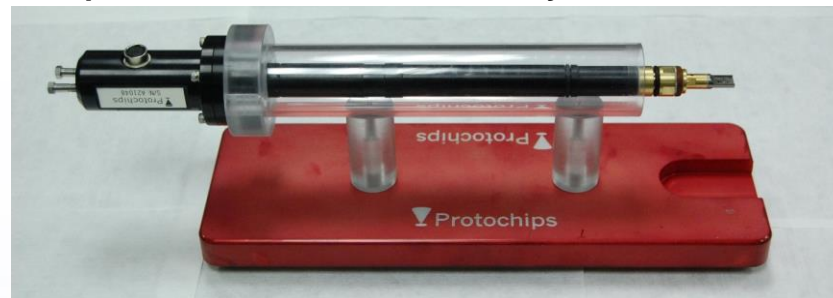


In situ TEM Hydrogen Exposure

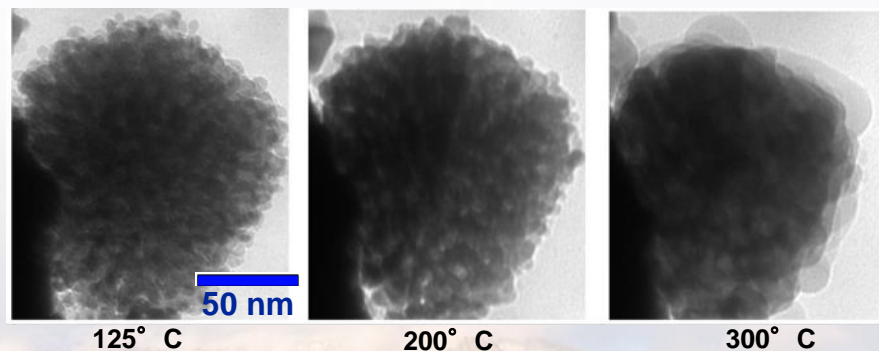
Contributors: B.G. Clark, P.J. Cappillino, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, L.R. Parent, I. Arslan. & Protochips, Inc.

Vapor-Phase Heating TEM Stage

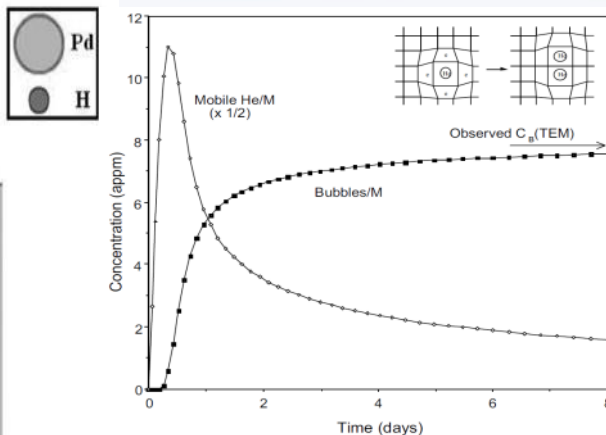
- Compatible with a range of gases
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Compatible with MS and other analytical tools



- 1 atm H₂ after several pulses to specified temp.



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



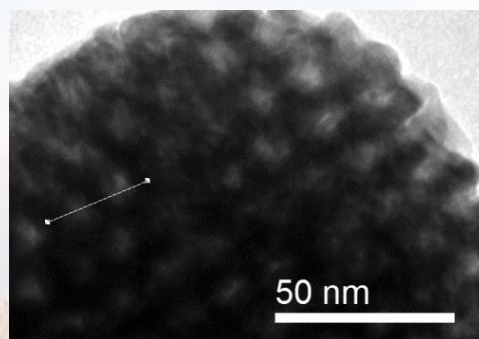
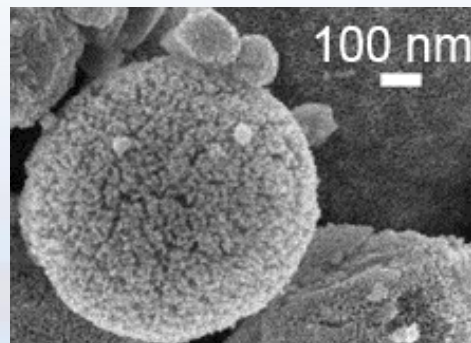
Cowgill, D., *Fusion Sci. & Tech.*, 28 (2005) p. 539

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

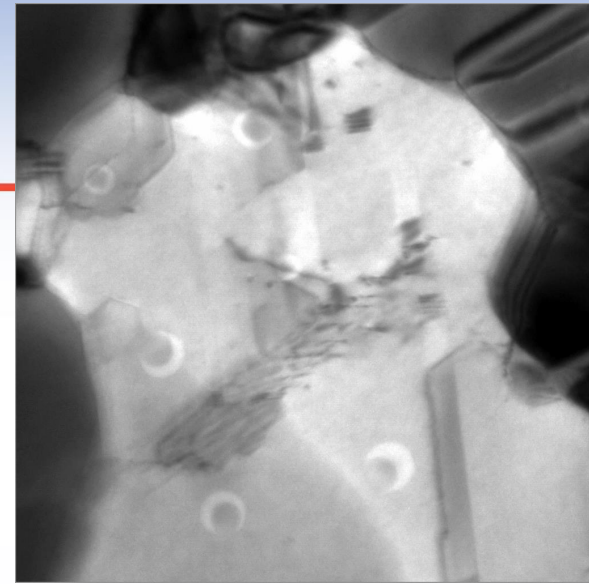
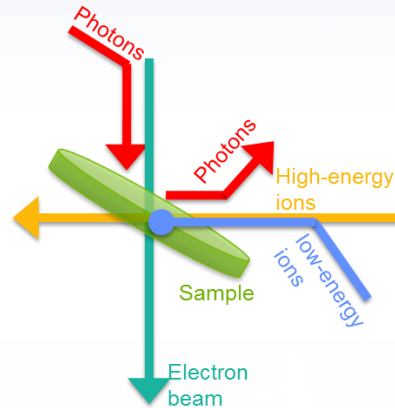
Harmful effects may be mitigated in nanoporous Pd



Summary

Sandia's I³TEM capabilities:

- *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 I³TEM although still under development is providing a wealth of interesting initial observations and harsh environments

Currently applying the current I³TEM capabilities to various material systems in combined and harsh environmental conditions

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)
- Beamline: Add 1 MV NEC Tandem & convert 90° magnet to bend beams 45°

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
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