

In situ Ion Irradiation Transmission Electron Microscope at Sandia National Laboratories

SAND2016-1927PE

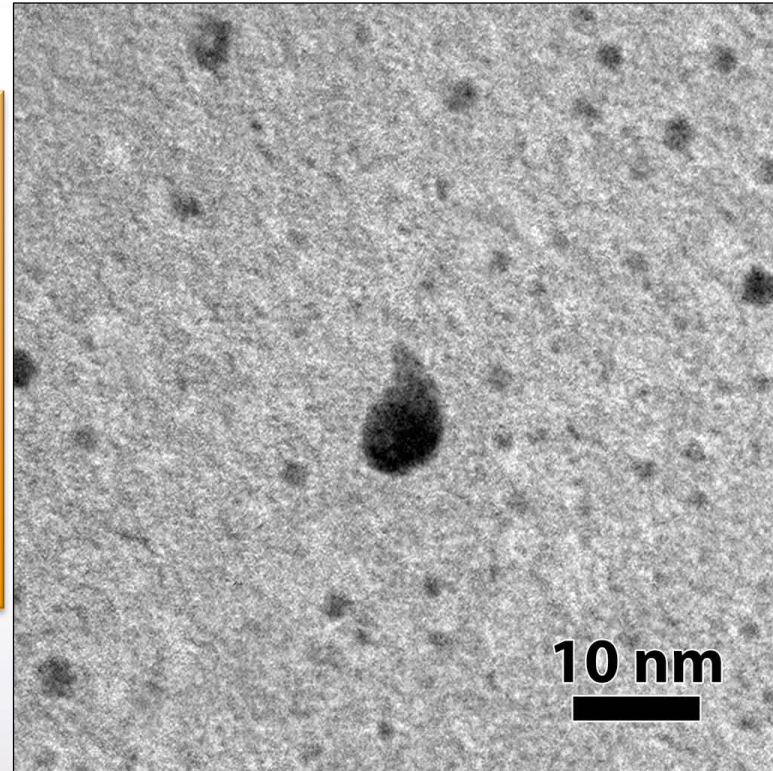
K. Hattar

Ion Beam Lab at Sandia National Laboratories

March 23, 2016

Outline

- 1) *Recent results in Au (NP and NC films)*
- 2) *Recent results in Ni films*
- 3) *Future directions*
- 4) *Far-out future directions*



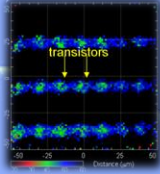
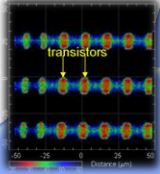
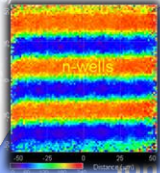
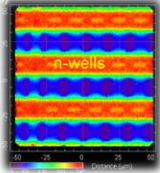
Collaborators:

- IBL: **D.C. Bufford, D. Buller, C. Chisholm, B.G. Clark, J. Villone, S. H. Pratt, M. Steckbeck, J. Kolar & M.T. Marshall**
- Sandia: **B. Boyce, T.J. Boyle, P.J. Cappillino, J.A. Scott, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, 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**

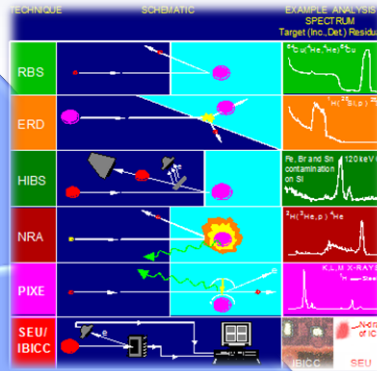
This work was supported by the US Department of Energy, Office of Basic Energy Sciences.

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

Sandia's Ion Beam Laboratory



Ion Beam Analysis (IBA)

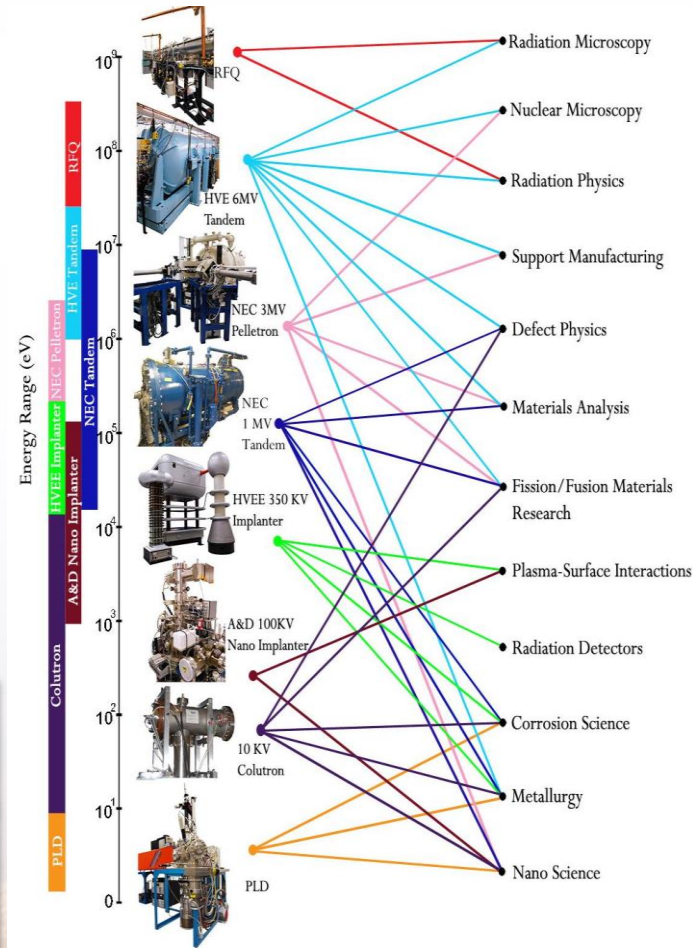
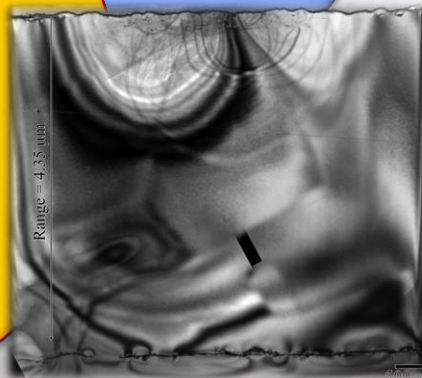


Radiation Effects Microscopy (REM)



Ion Beam Modification (IBM)

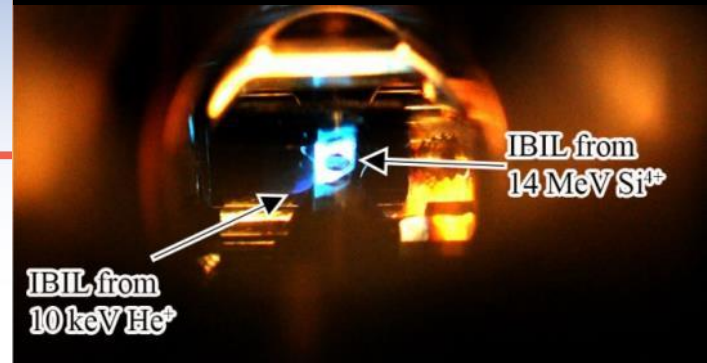
Radiation Electron Microscopy (REM)



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

Collaborator: D.L. Buller

IBIL from a quartz stage inside the TEM

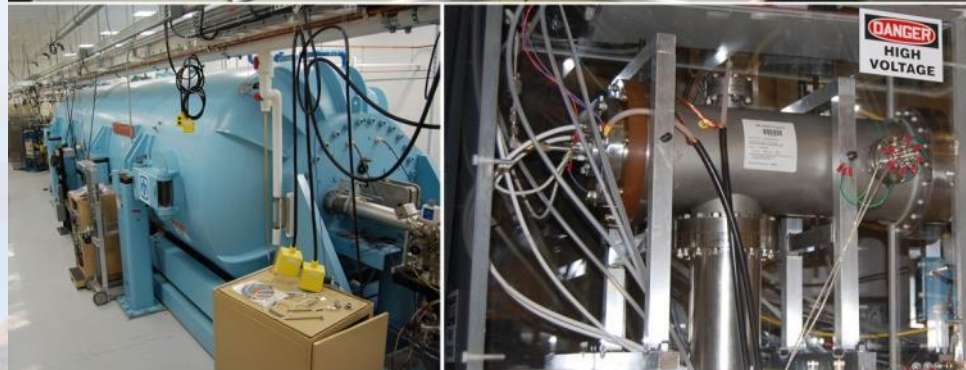
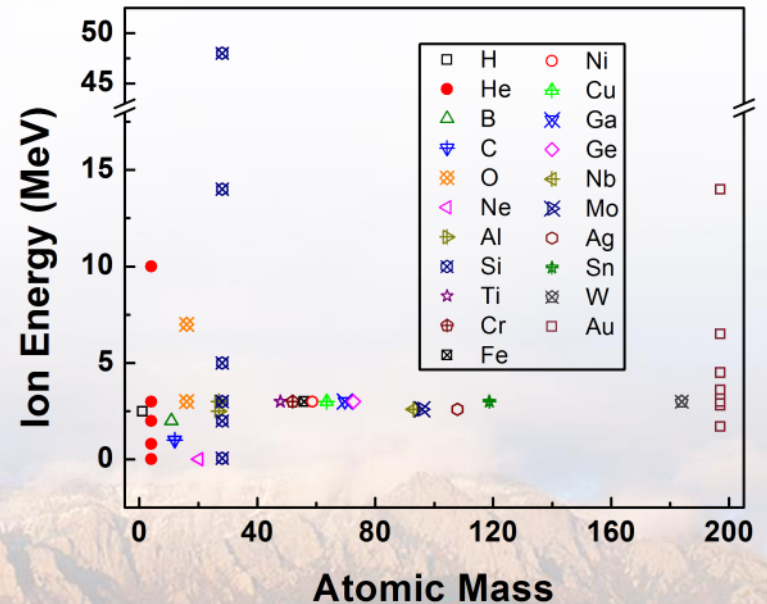


Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution

10 kV Colutron - 200 kV TEM - 6 MV Tandem

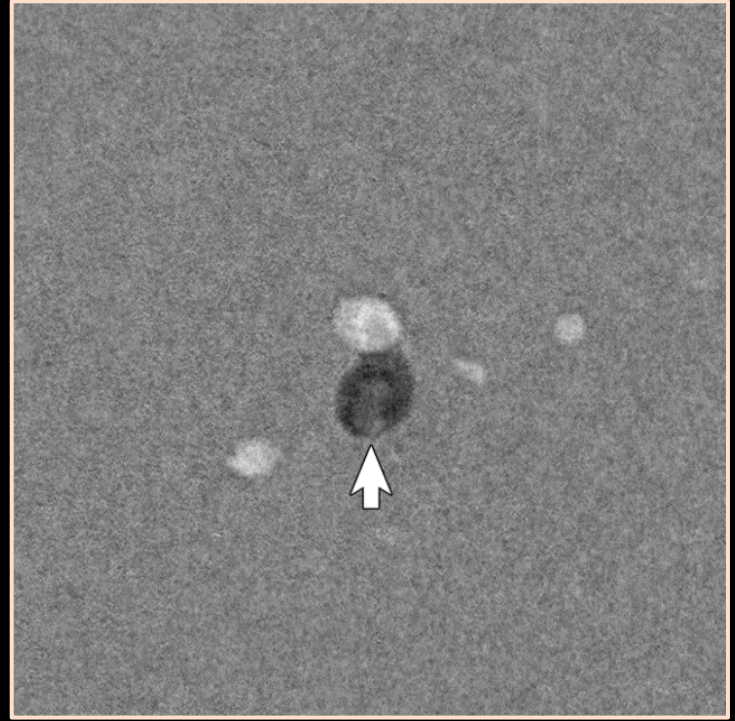
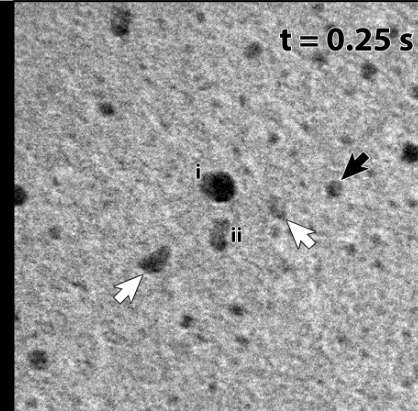
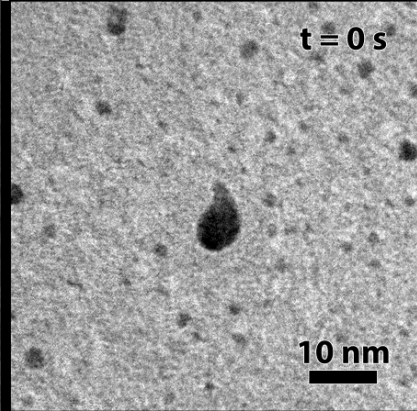
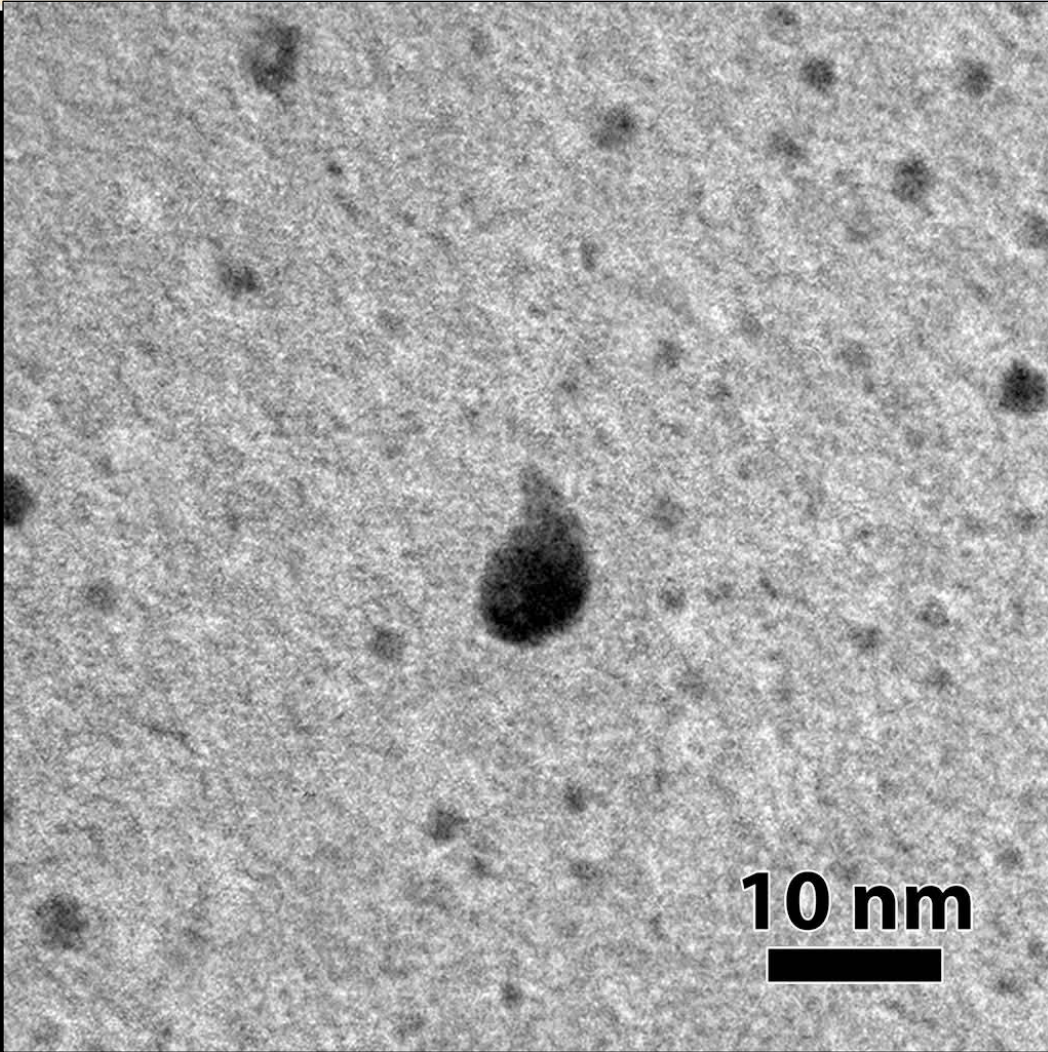


Ion species & energy introduced into the TEM



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

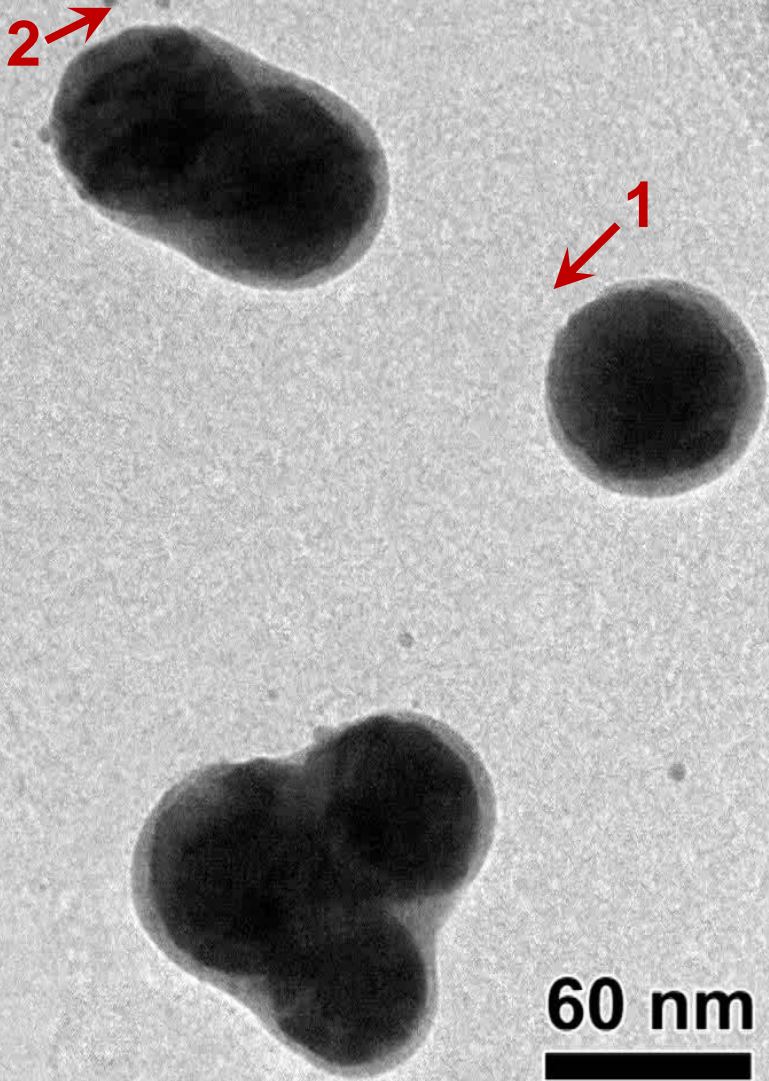
Collaborator: D.C. Bufford



Single Ion Strikes:

2.8 MeV Au⁴⁺ ions into 60 nm Au nanoparticles

Collaborator: D.C. Bufford



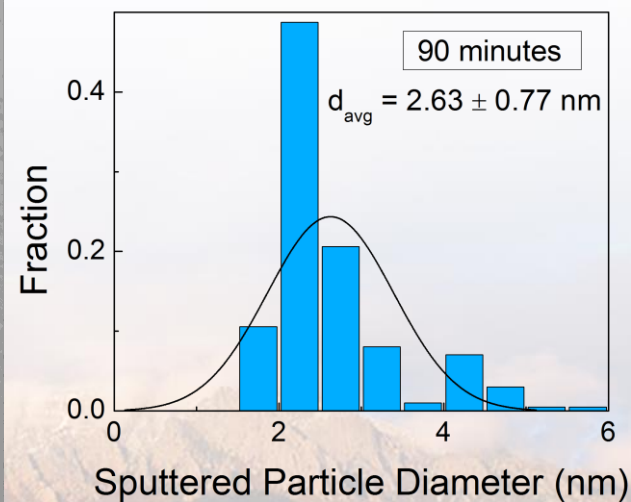
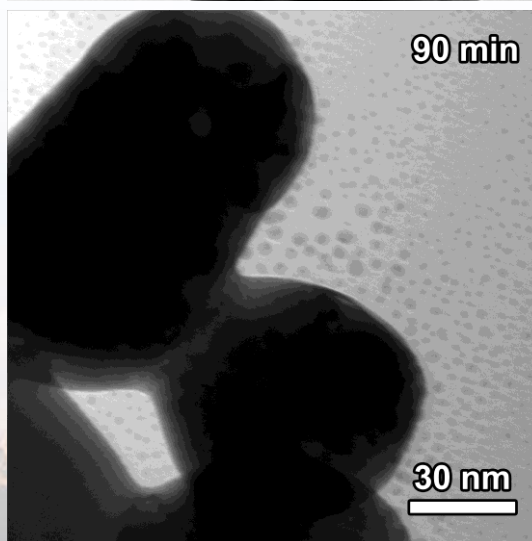
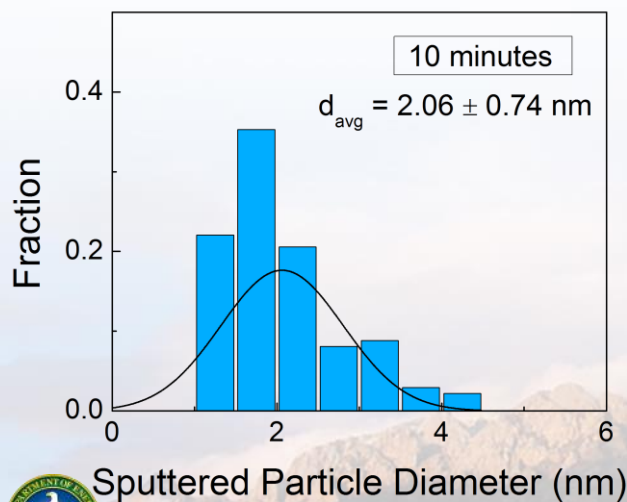
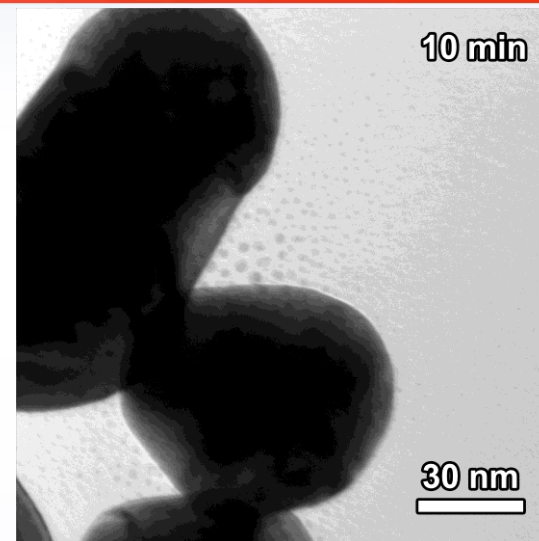
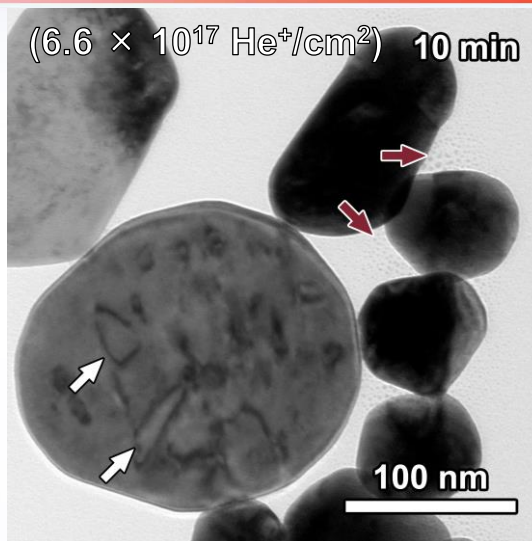
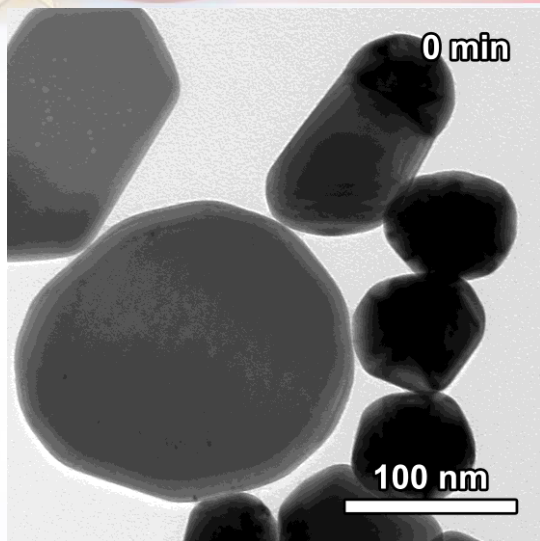
- 2.8 MeV Au⁴⁺ ions into 60 nm diameter Au nanoparticles
- 100 kx magnification
- Nanoscale filaments created by individual ions

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



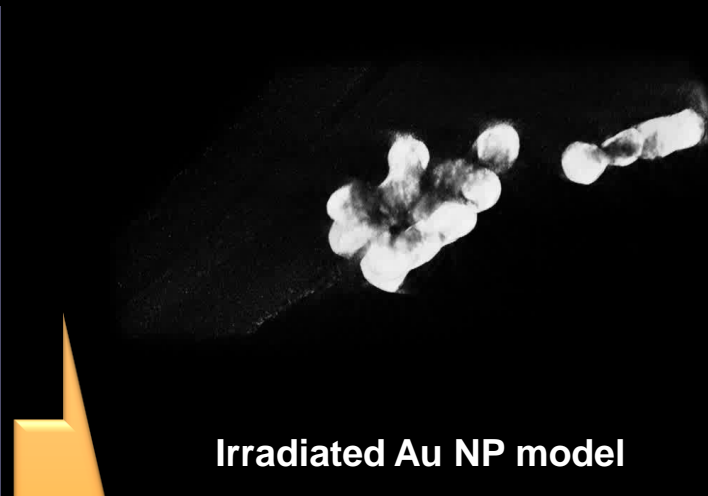
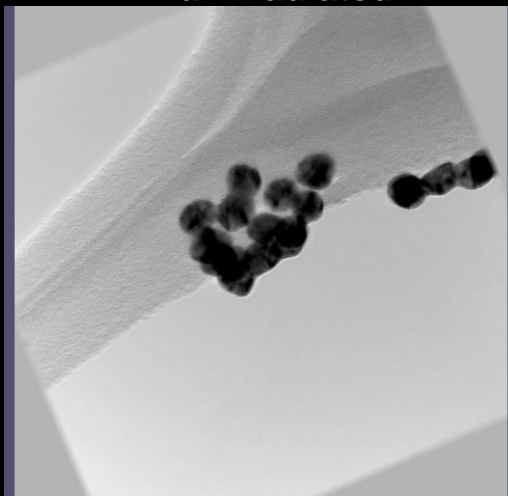
Electron Tomography Provides 3D Insight

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

In situ Ion Irradiation TEM (I³TEM)

Aligned Au NP tilt series -
unirradiated

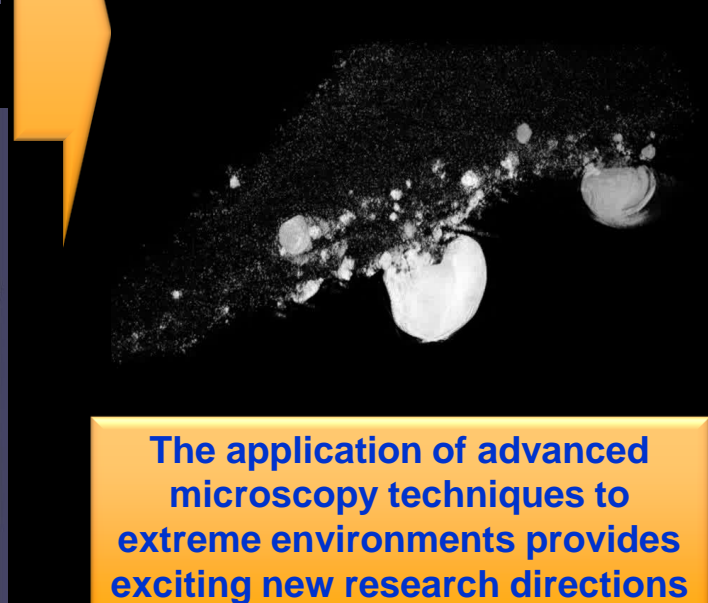
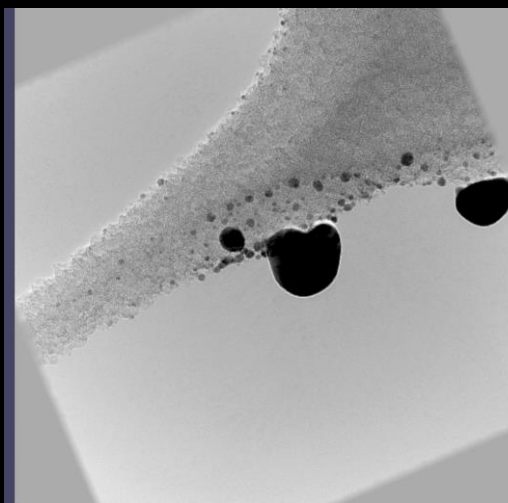
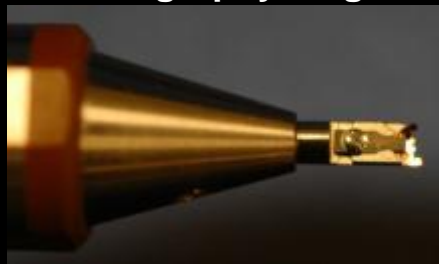
Unirradiated Au NP model



Hummingbird
tomography stage

Aligned Au NP tilt series -
irradiated

Irradiated Au NP model

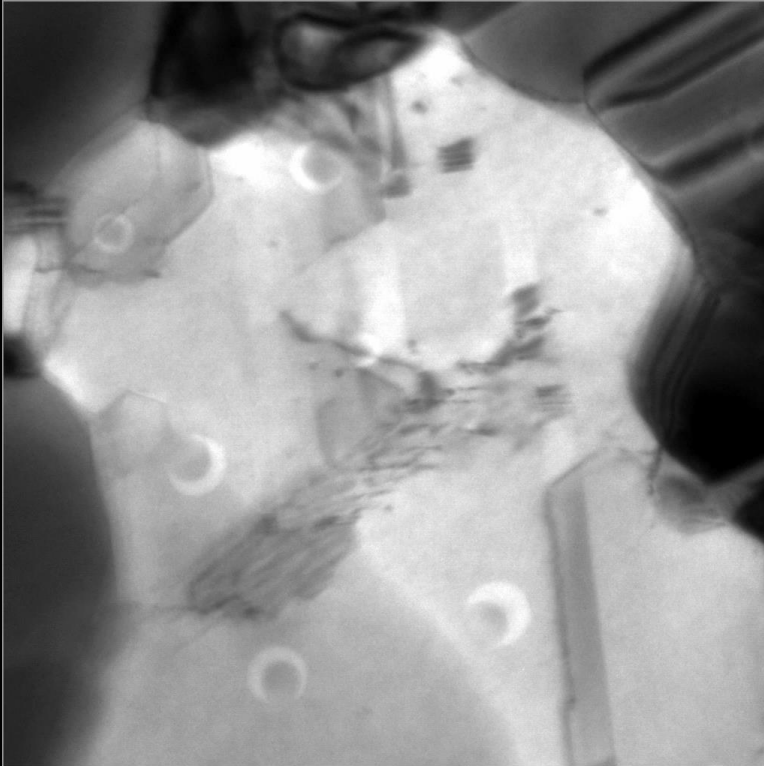


The application of advanced
microscopy techniques to
extreme environments provides
exciting new research directions

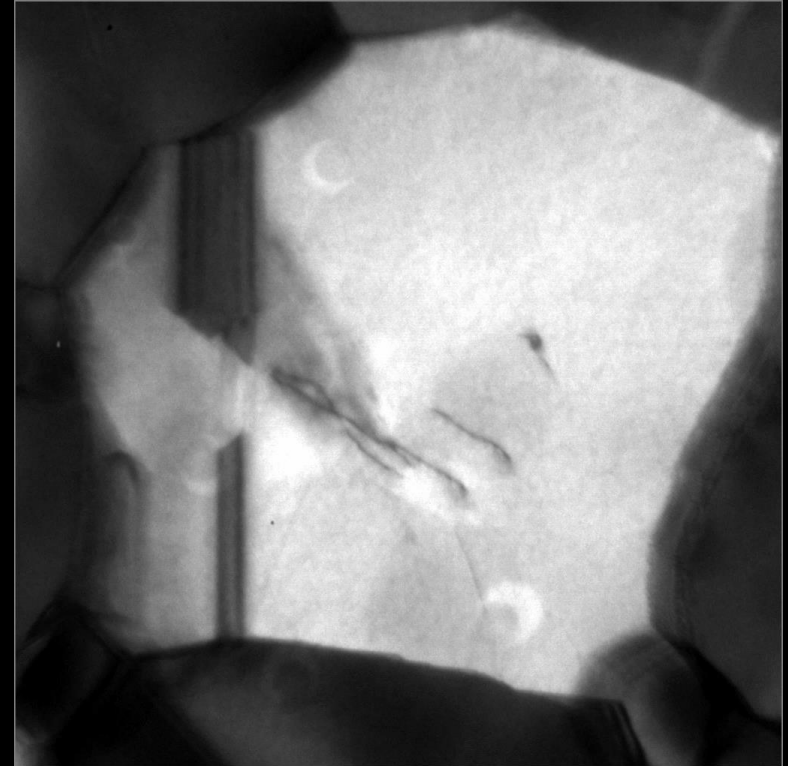
Dose Rate Effects

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

7.9×10^9 ions/cm²/s



6.7×10^7 ions/cm²/s

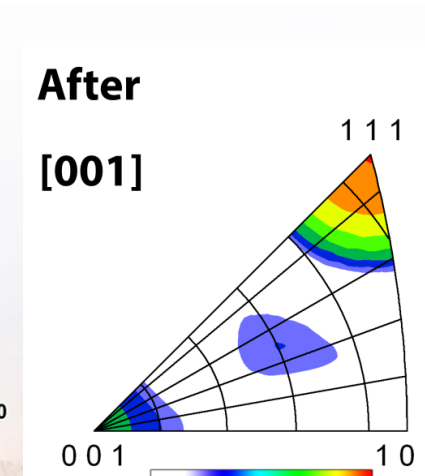
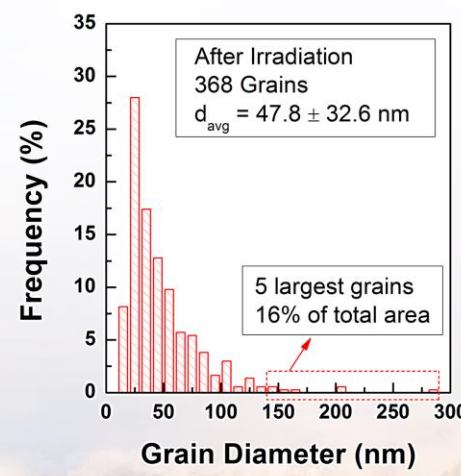
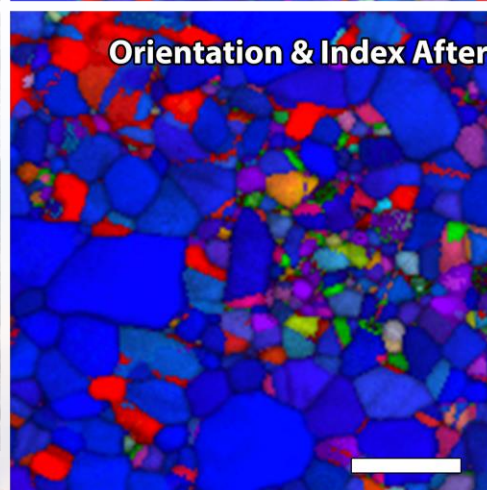
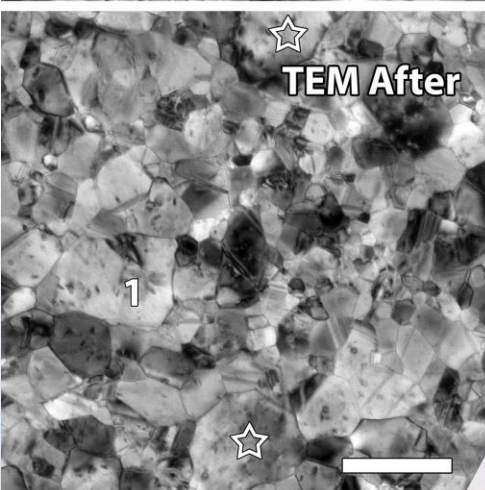
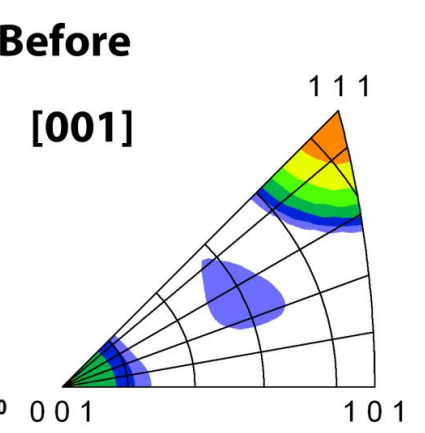
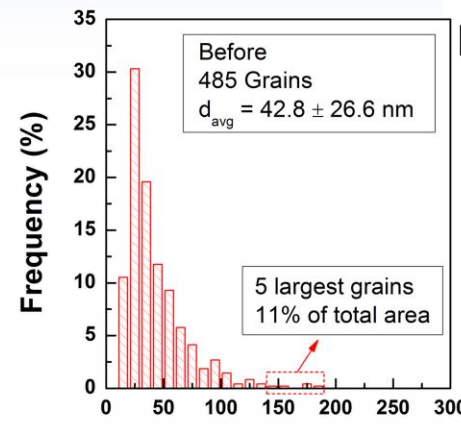
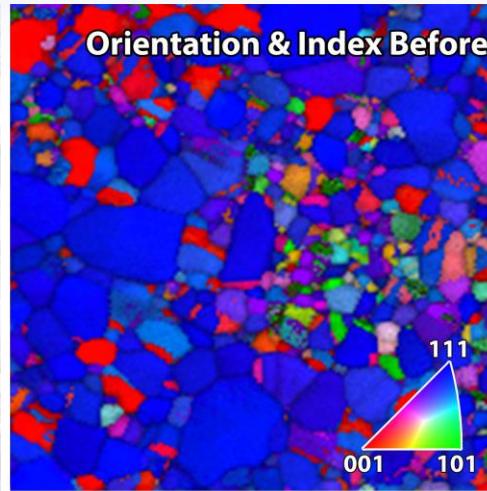
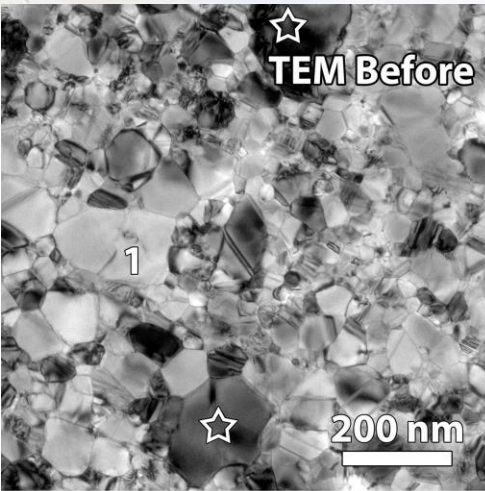


VS

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

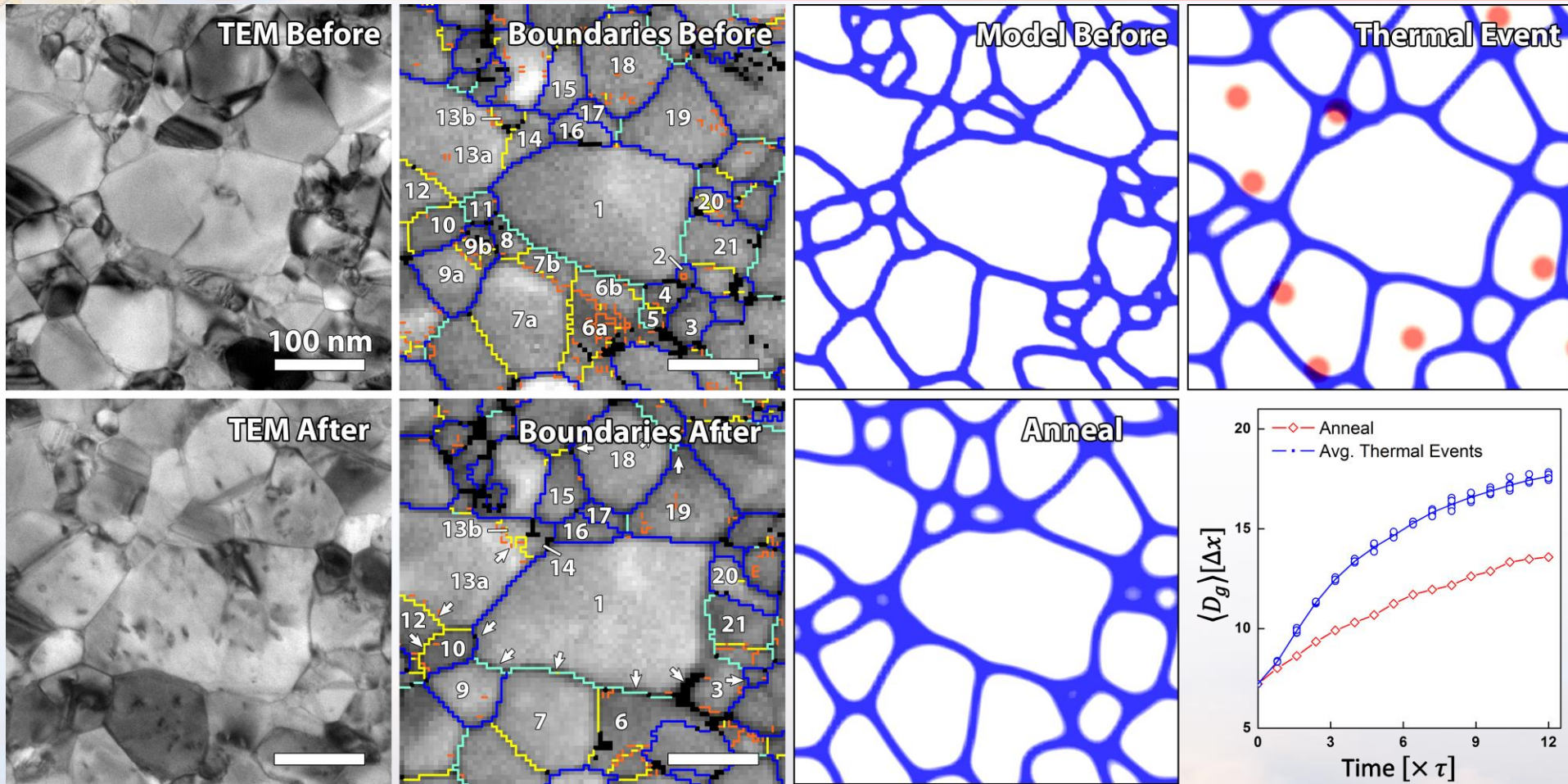


Any texture or grain boundary evolution can be directly observed and quantified



Direct Comparison to Mesoscale Modeling

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

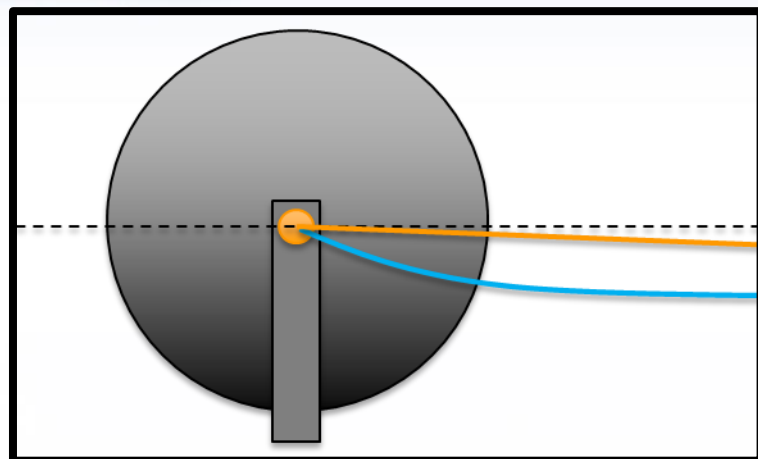


Because of the matching length scale, the initial microstructure can serve as direct input to either MD or mesoscale models & subsequent structural evolution can be directly compared.

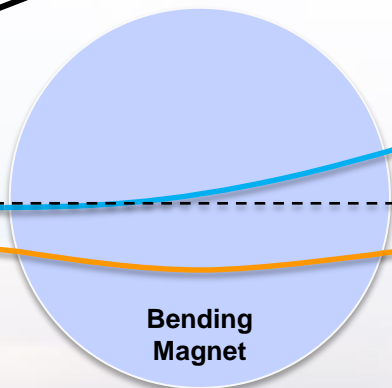
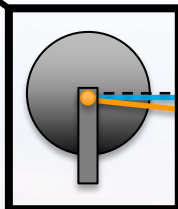


Modeling Beam Mixing and Deflection Necessary to Develop a *In situ* Triple Beam Facility

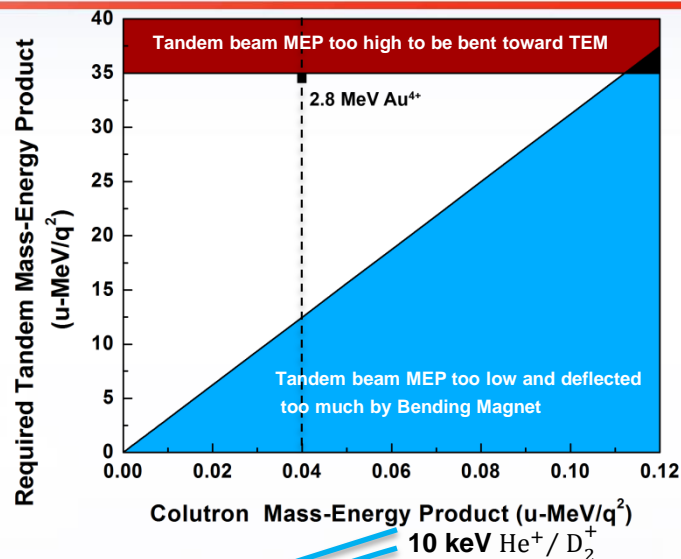
Collaborators: M. Steckbeck, D.C. Bufford, & B.L. Doyle



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



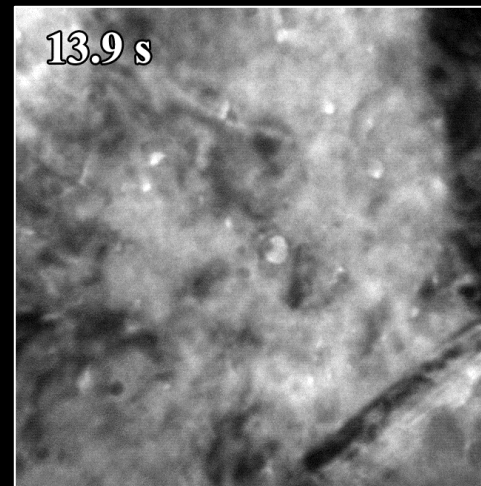
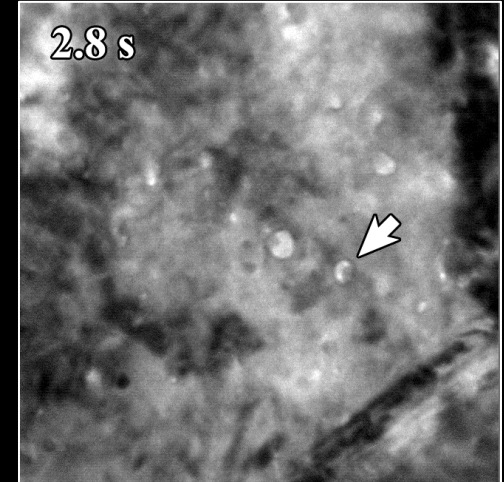
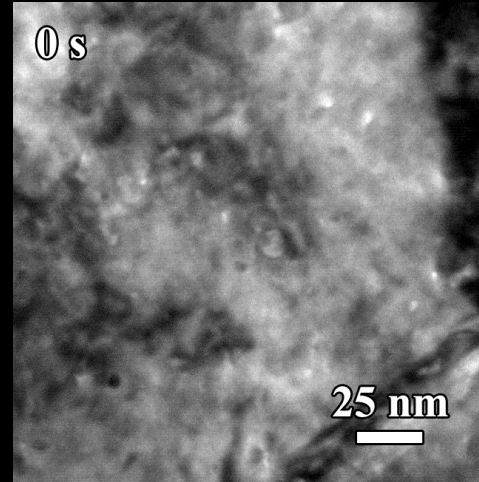
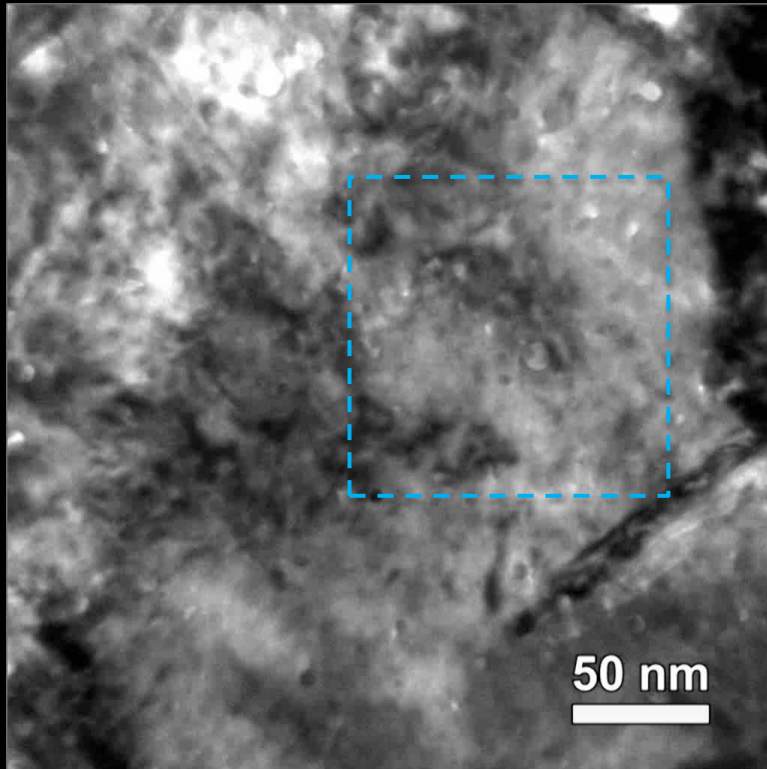
Sandia National Laboratories

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.

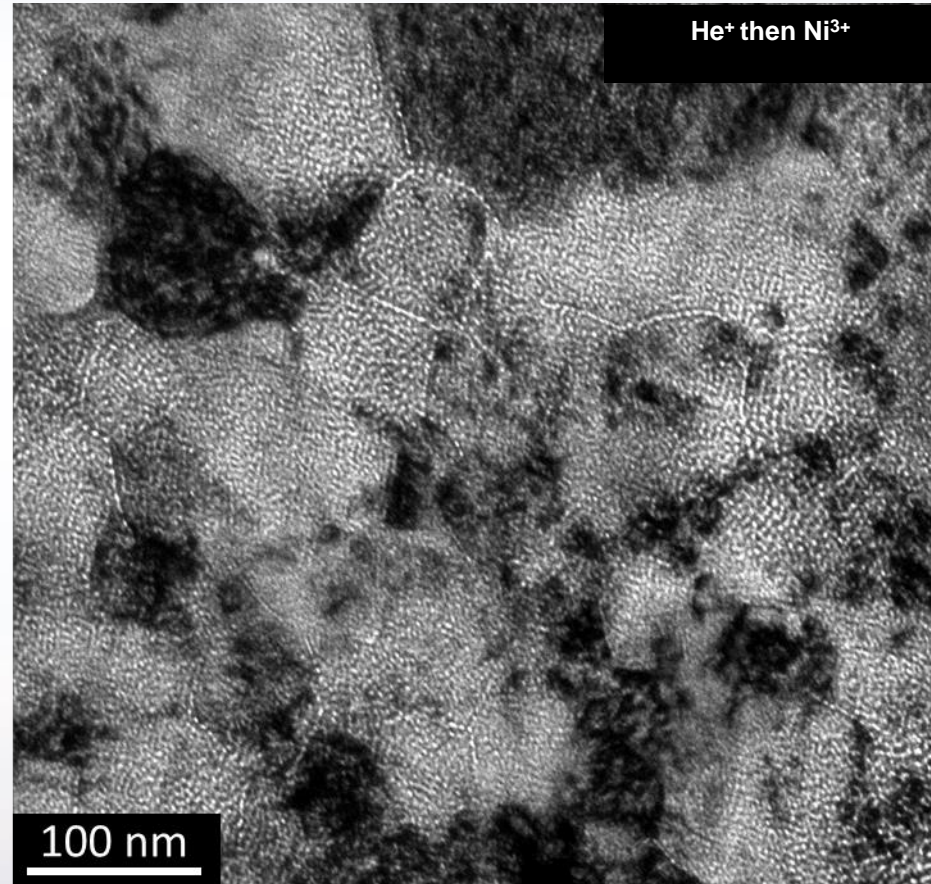
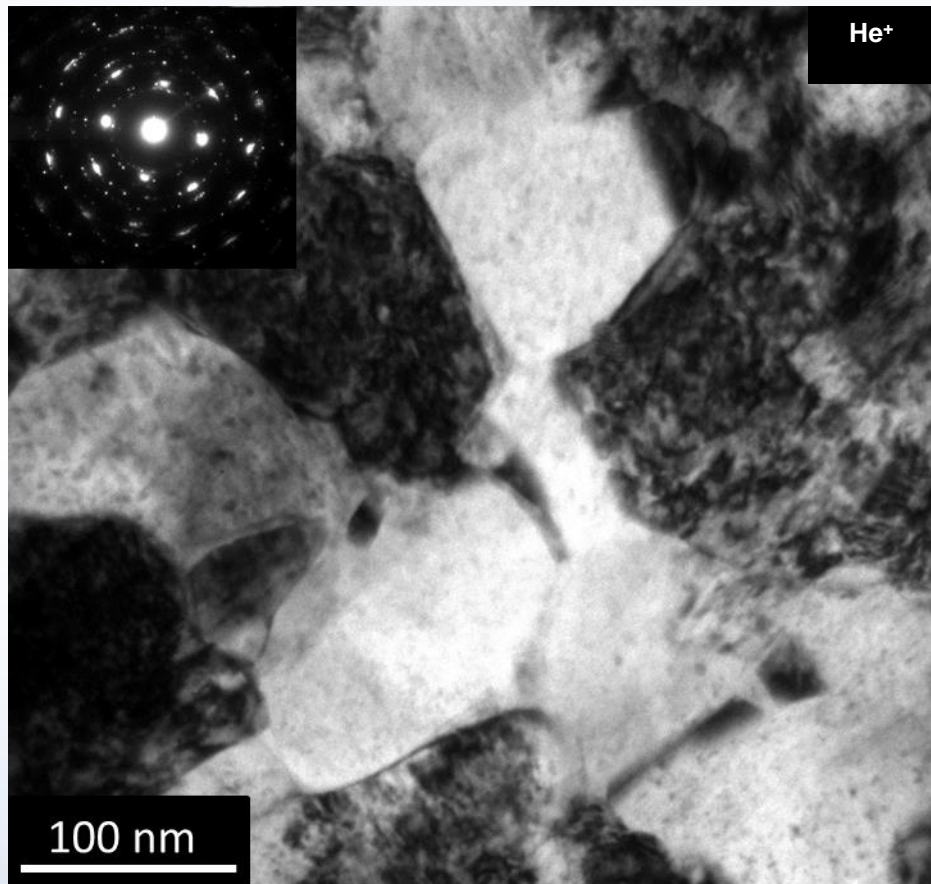
■ Approximate fluence:

- 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

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

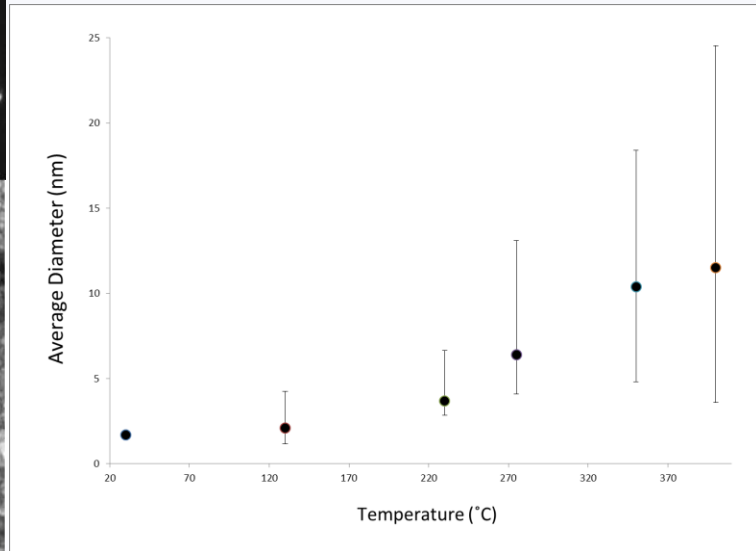
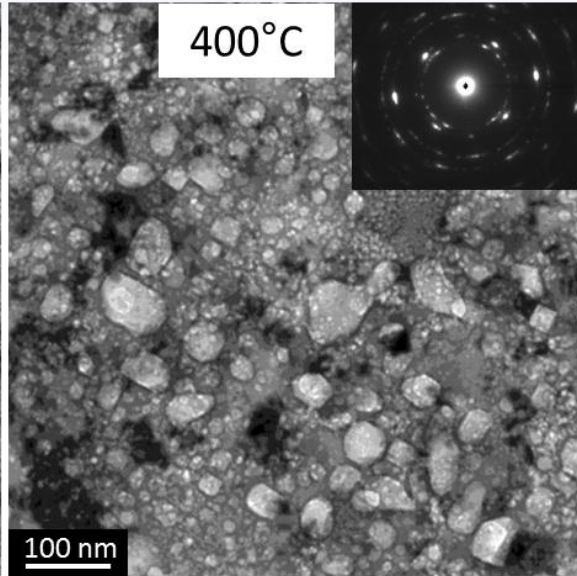
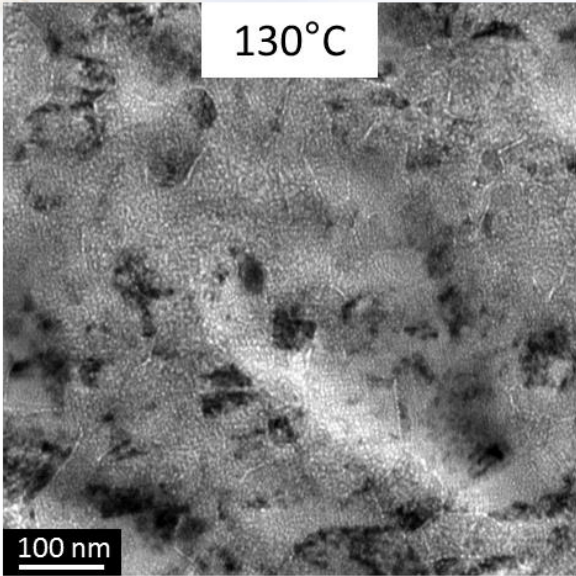
Collaborator: B. Muntifering & J. Qu



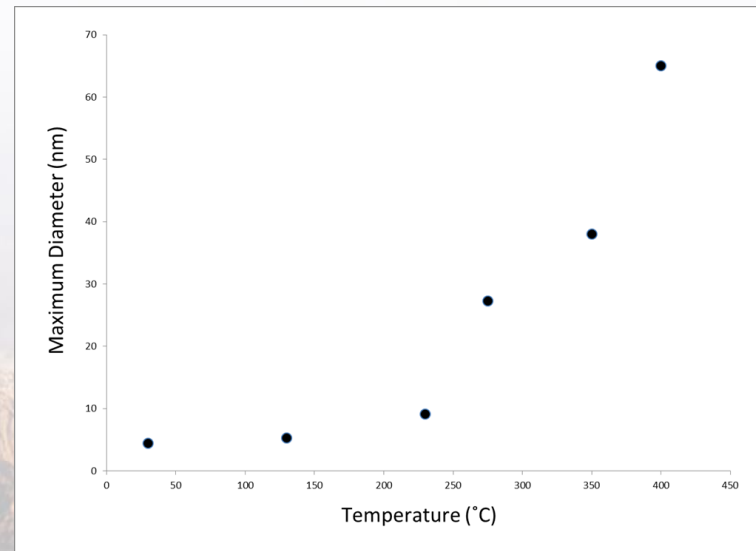
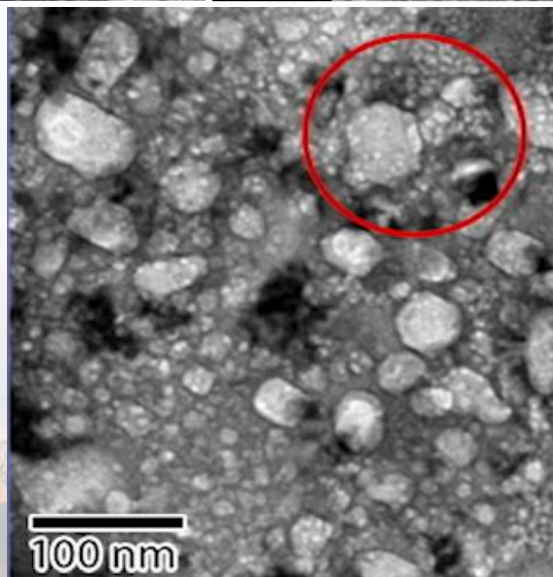
10¹⁷ He⁺/cm²
Visible damage to both the
sample and the source

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⁺

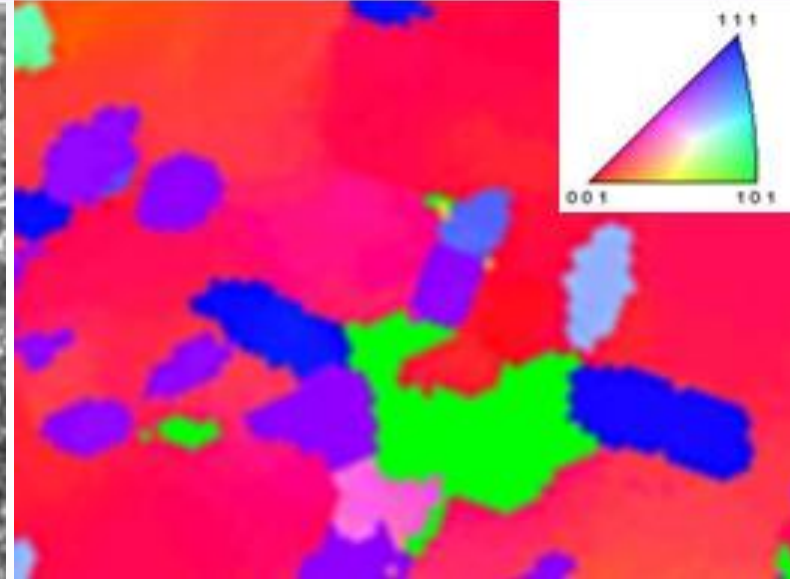
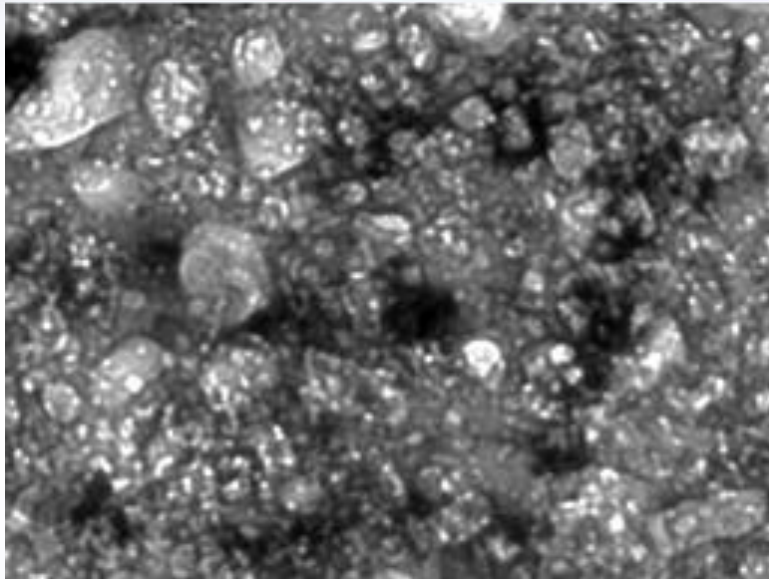


Bubble to cavity transition and cavity evolution can be directly studied

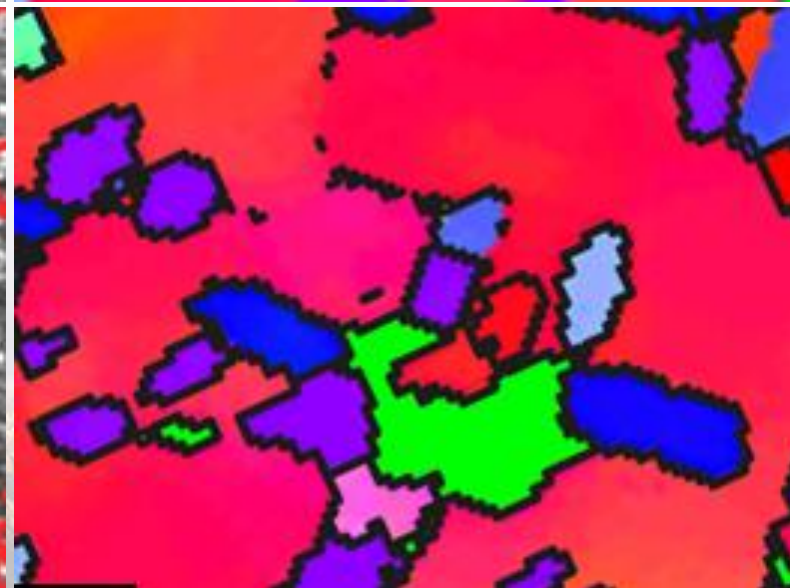
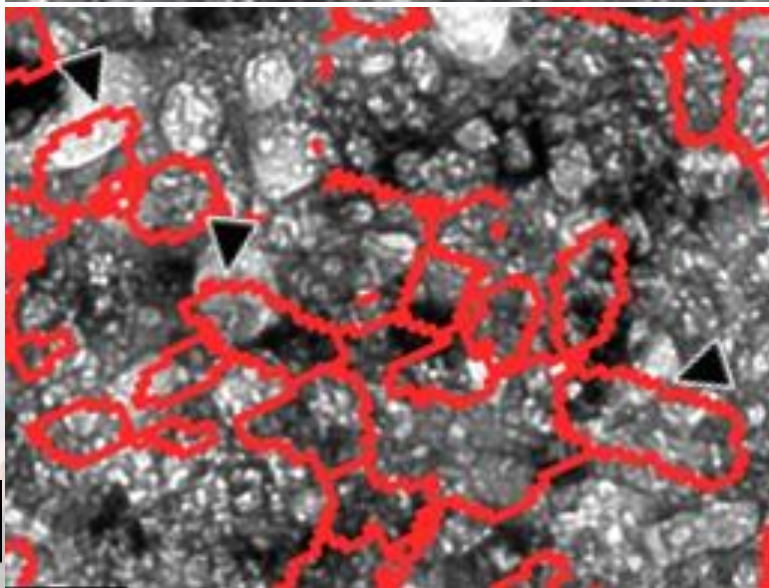


Precession Electron Diffraction Reveals Hidden Grain Structure

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



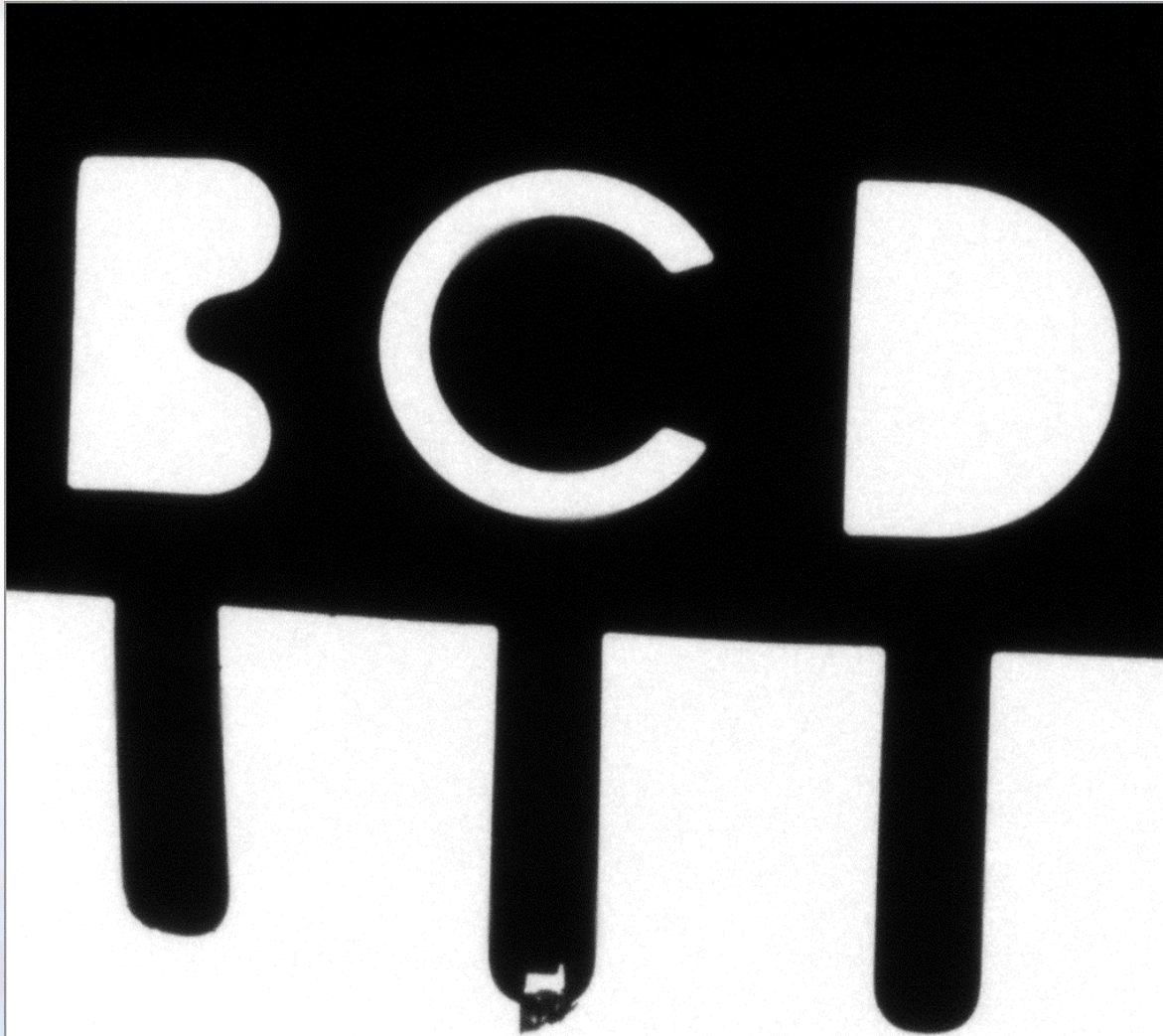
Cavities span multiple grains at identified grain boundaries



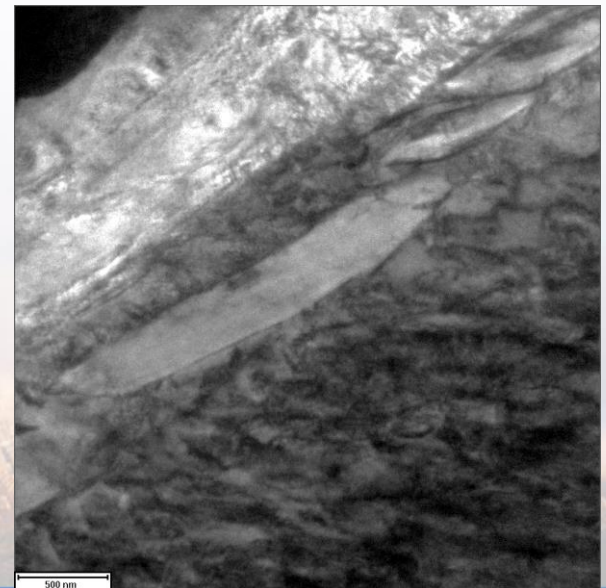
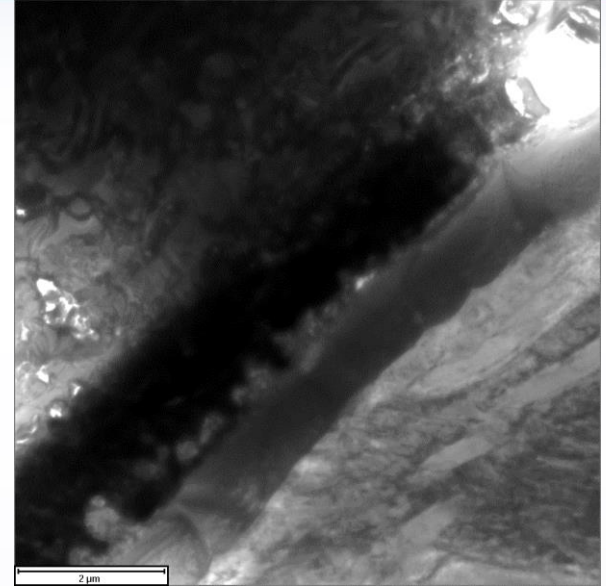
100 nm

First “Hot” sample into I³TEM

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

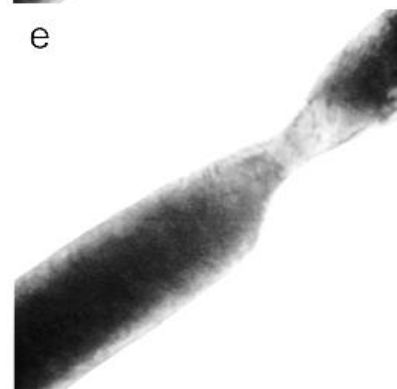
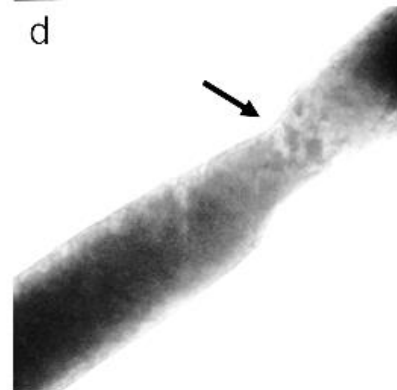
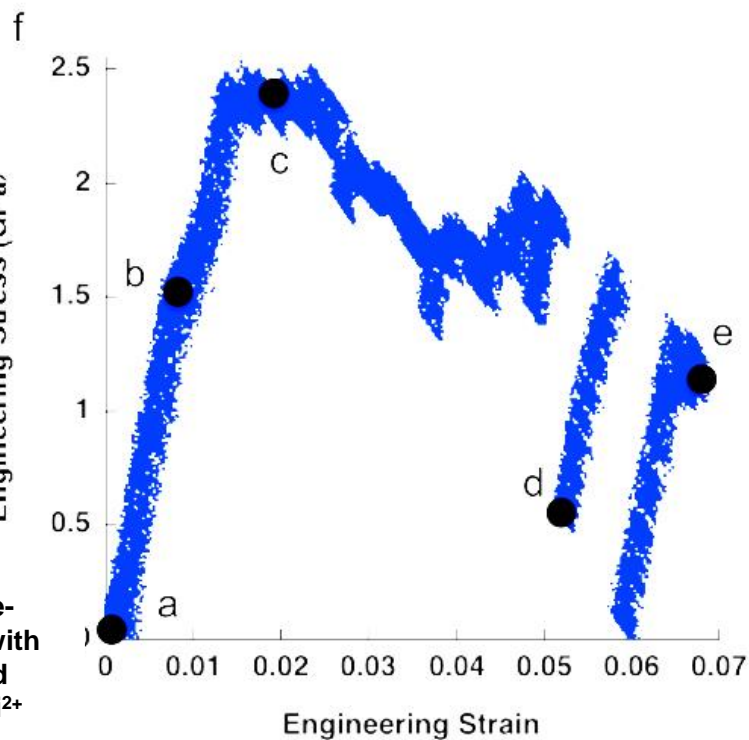
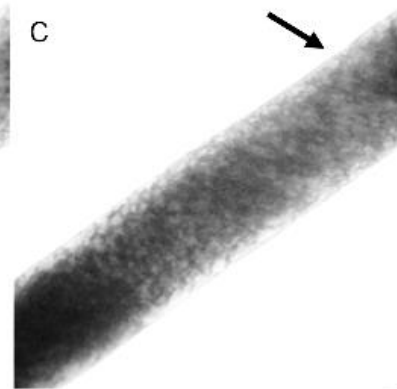
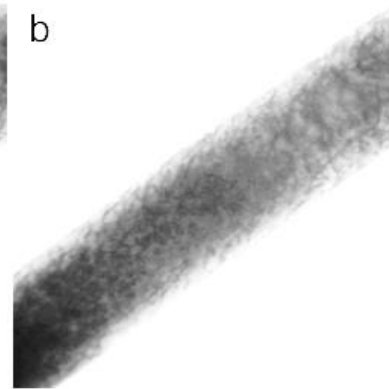
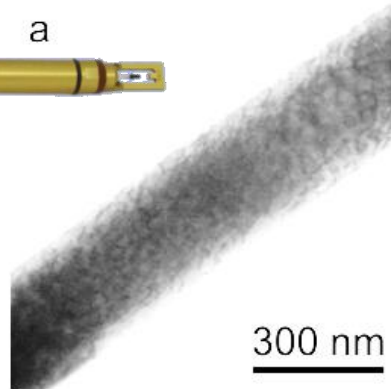
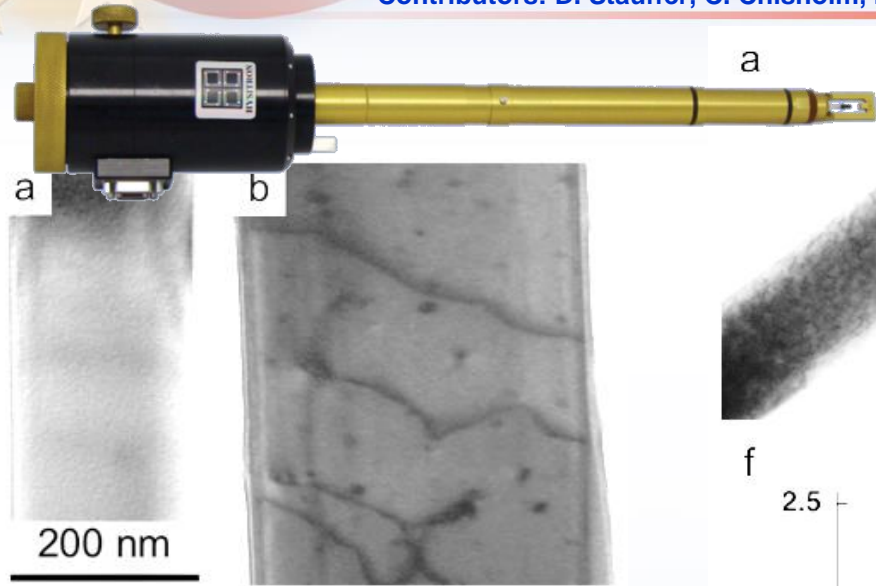


This Pellet Cladding Interface is the first “hot” TEM sample approved for study in Sandia’s I³TEM



In situ TEM Quantitative Mechanical Testing

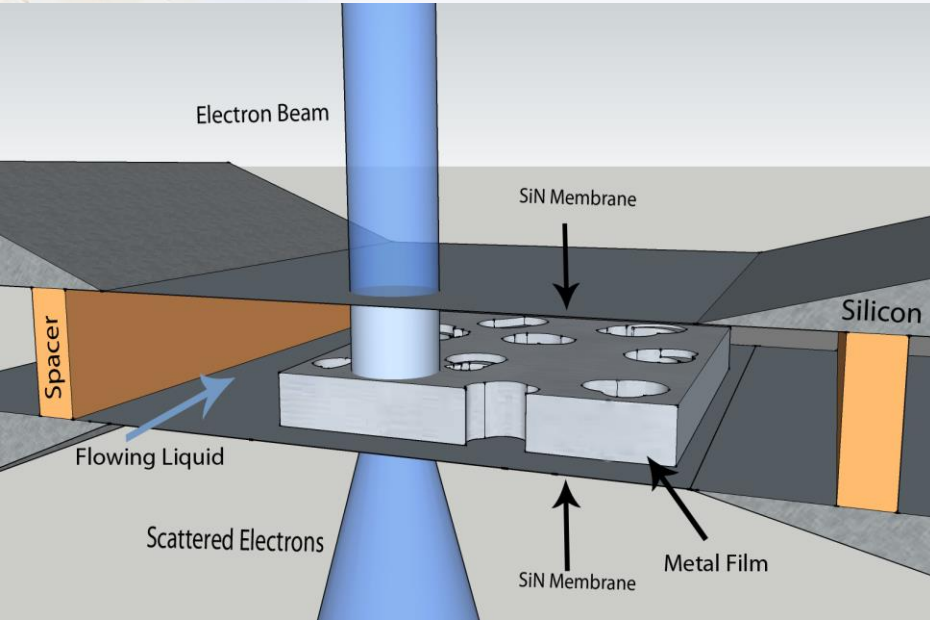
Contributors: D. Stauffer, C. Chisholm, H. Bei, E.P. George, P. Hosemann, & A.M. Minor



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.

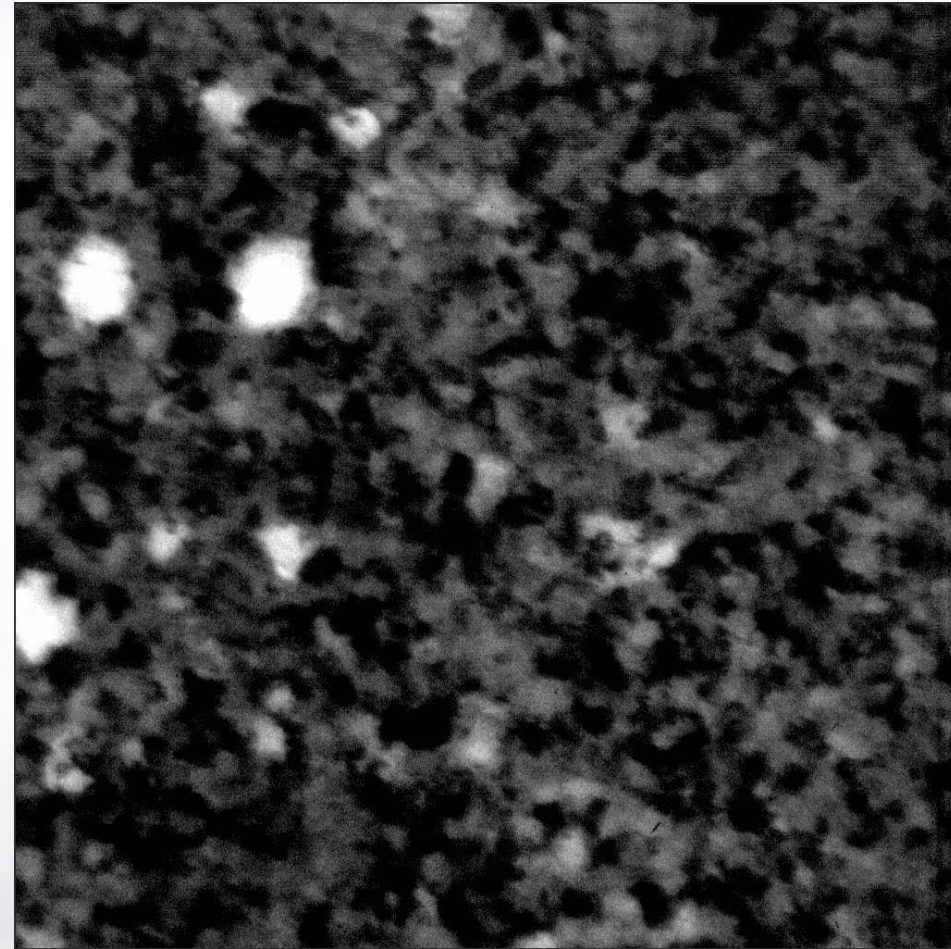
Future Direction: *In situ* TEM Corrosion Direction

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.

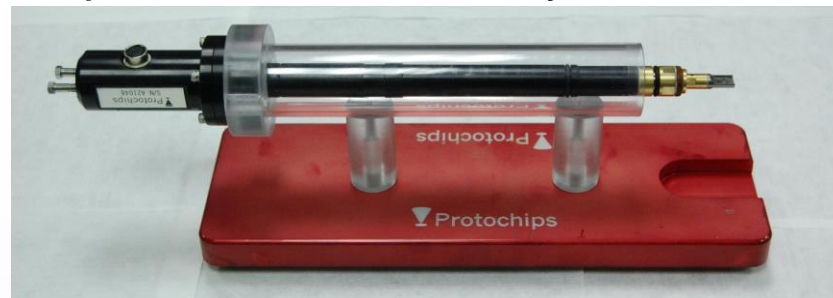


Future Direction: *In situ* TEM Hydrogen Exposure

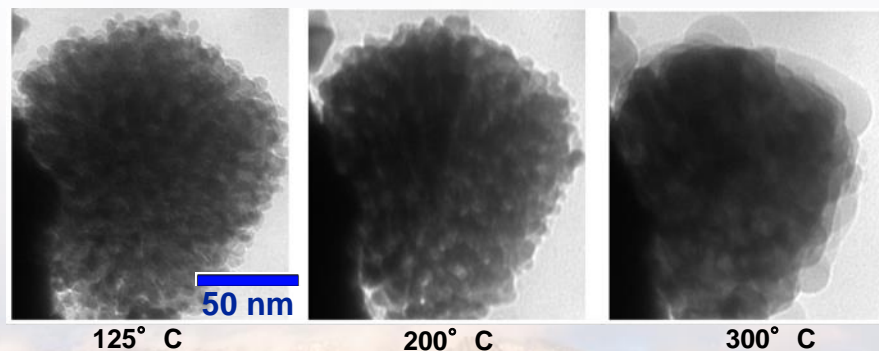
Contributors: B.G. Clark, P.J. Cappillino, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, L.R. Parent, I. Arslan. & Protochips, Inc.

Vapor-Phase Heating TEM Stage

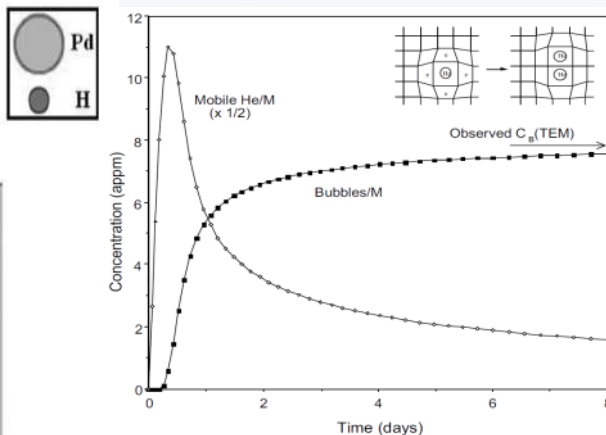
- Compatible with a range of gases
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Compatible with MS and other analytical tools



- 1 atm H₂ after several pulses to specified temp.



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



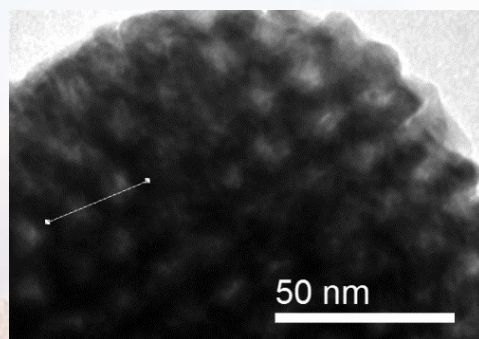
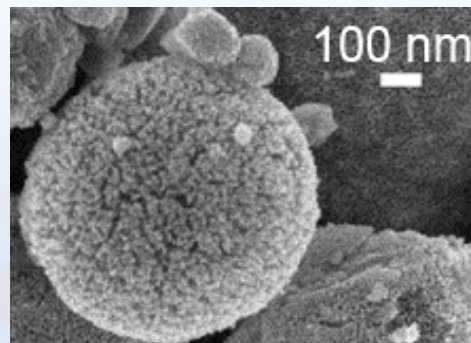
Cowgill, D., *Fusion Sci. & Tech.*, 28 (2005) p. 539

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

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

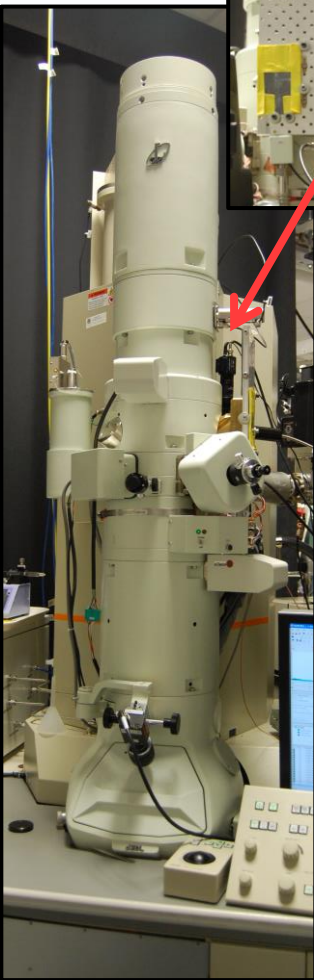
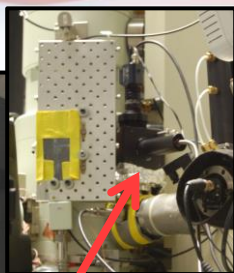
R. Delmelle, J., *Phys. Chem. Chem. Phys.* (2011) p.11412

Harmful effects may be mitigated in nanoporous Pd

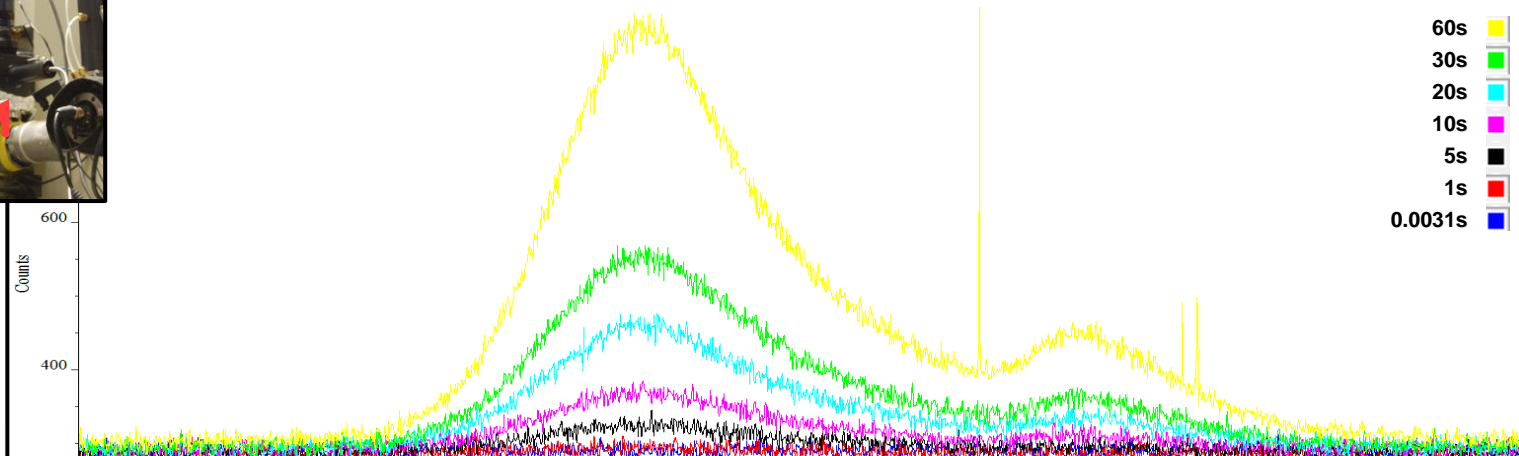


Future Direction: *In situ* TEM Ion beam Induced Luminescence (IBIL)

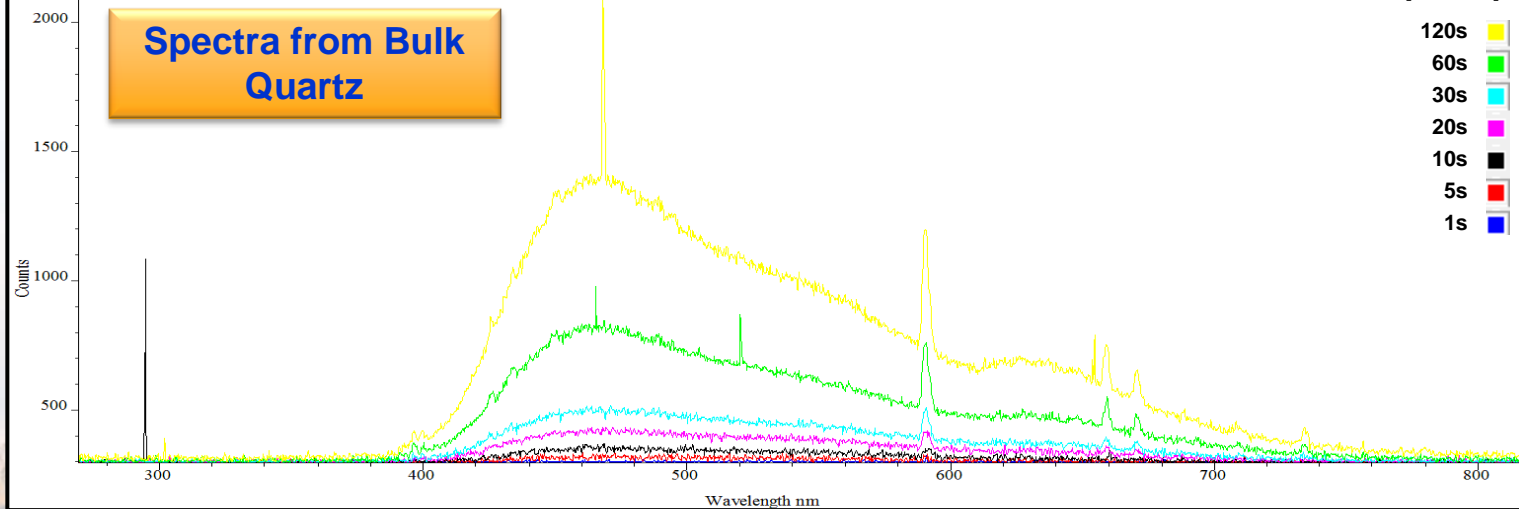
Collaborator: J. Gutierrez-Kolar



Cathodoluminescence (CL)



Ion Beam Induced Luminescence (IBIL)

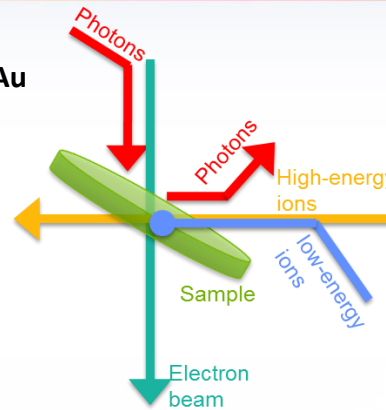


Significant optimization is still needed; potential is promising

Summary & Still Father-out 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
- Quantitative and bulk straining
- Two-port microfluidic cell
- Gas flow/heating stage
- Electron tomography
- Precession Electron Diffraction



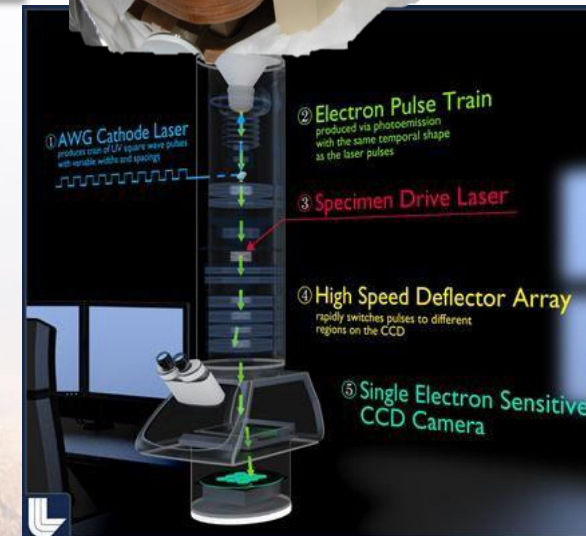
Currently applying the current I³TEM capabilities to various material systems in sequential or combined harsh environmental conditions

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, S. H. Pratt, M. Steckbeck, J. Kolar & M.T. Marshall
- Sandia: B. Boyce, T.J. Boyle, P.J. Cappillino, J.A. Scott, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, 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 partially funded by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.