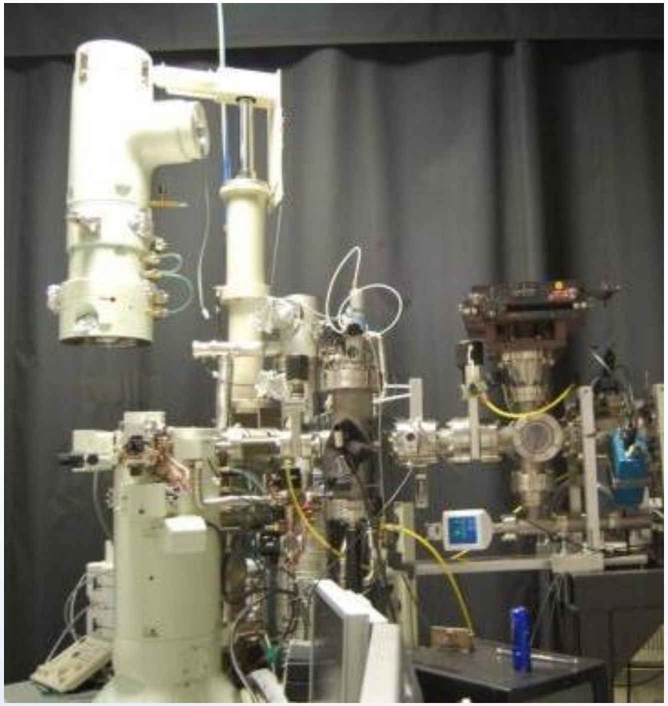
Recent Developments in *In situ* TEM to Probe Mechanical, Radiation, and Corrosive Response of Materials

SAND2014-17423PE

K. Hattar

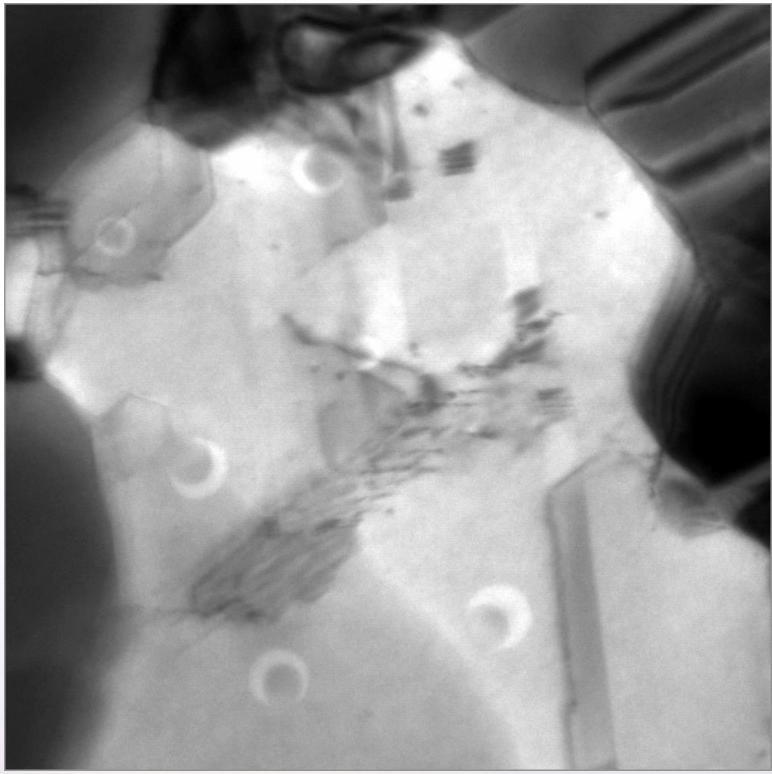
Ion Beam Lab at Sandia National Laboratories

September 22, 2014



***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, B.L. Doyle, S. H. Pratt, & M.T. Marshall**
- **Sandia: B. Boyce, T.J. Boyle, P.J. Cappillino, J.A. Scott, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, E. Carnes, J. Brinker, D. Sasaki, J.A. Sharon, T. Nenoff, W.M. Mook**
- **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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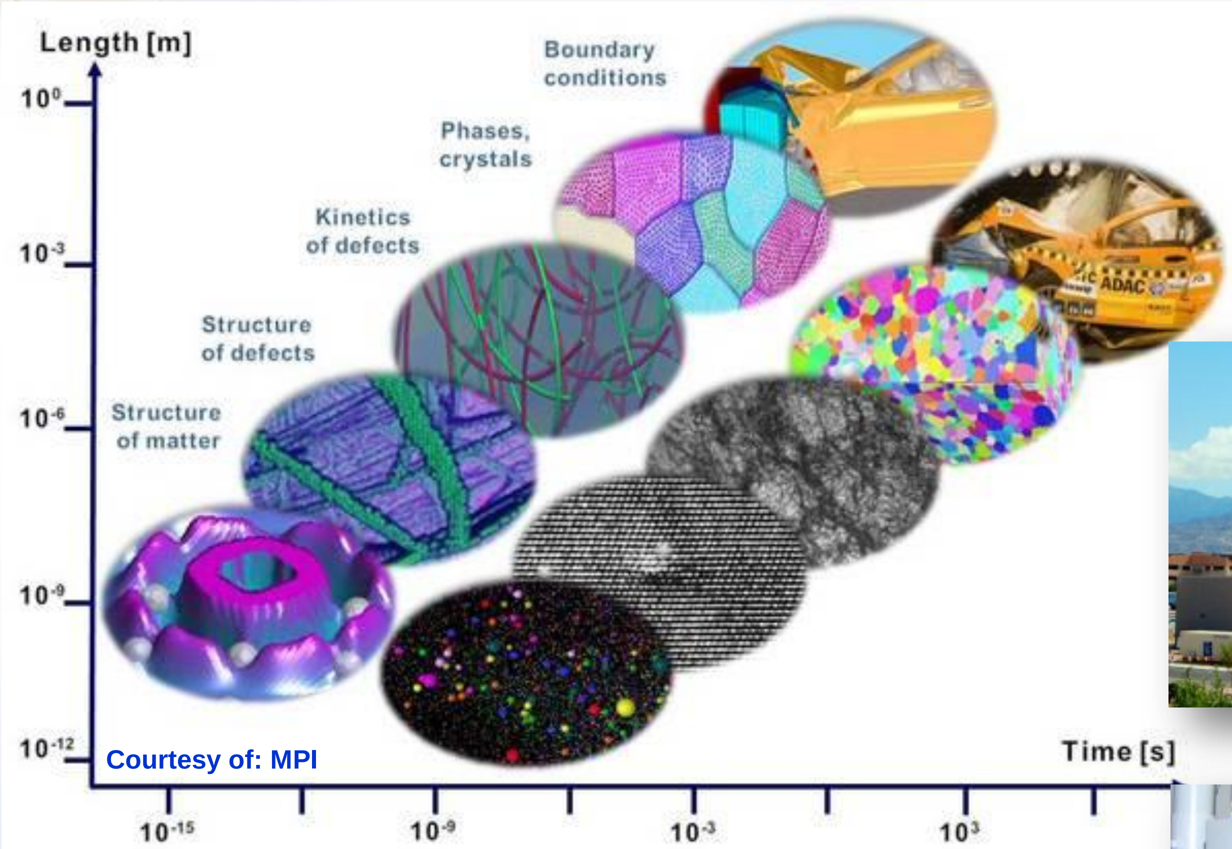
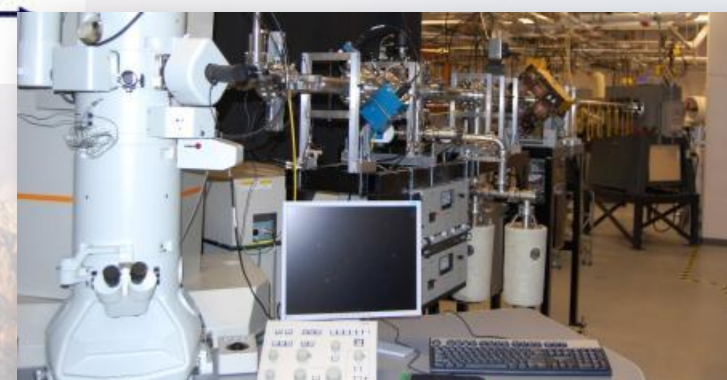
Investigating the nm Scale to Understand the km Scale to Understand Materials Response in the Extremes



Ion Beam Lab (IBL)

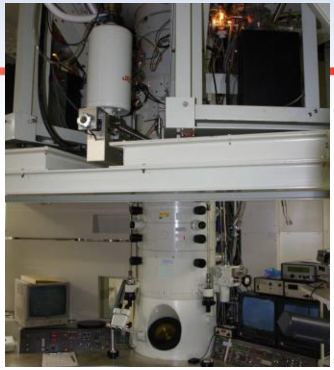
In situ Ion Irradiation TEM (I^3TEM)



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

History of *In situ* Ion Irradiation TEM

Courtesy of: J. Hinks



1930s… → 1960s → 1970s → 1980s → 1990s → 2000s → 2010s

1931
The invention of the TEM

1961
O$^-$ emission reported from a TEM filament by Pashley, Presland, and Meneter at TI Labs, Cambridge, UK

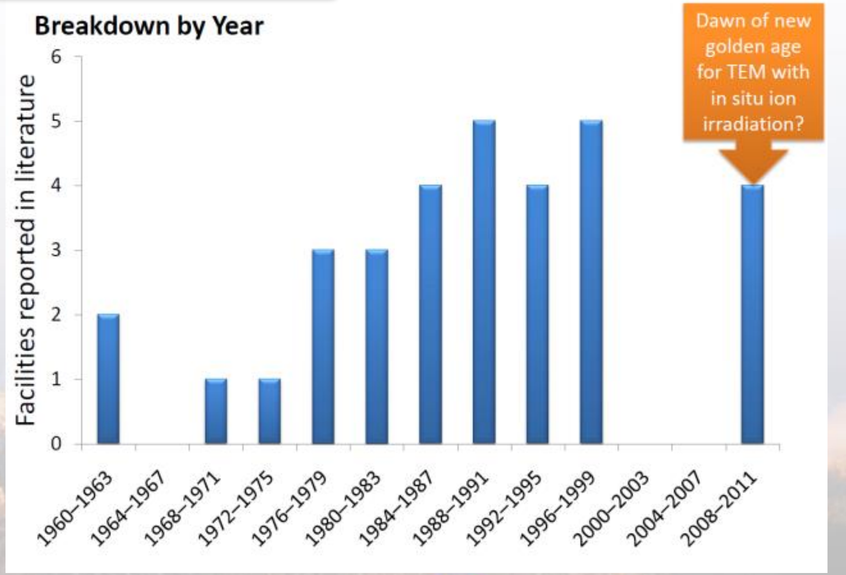
1968
First TEM beamline combination by Thackery, Nelson, and Sansom at AERE Harwell, UK

1976
First HVEM with ion irradiation at UVA, USA

1978
First in-situ ion irradiation experiments at ANL

1990s
First dual beam system developed at JAERI and NIMS, Japan

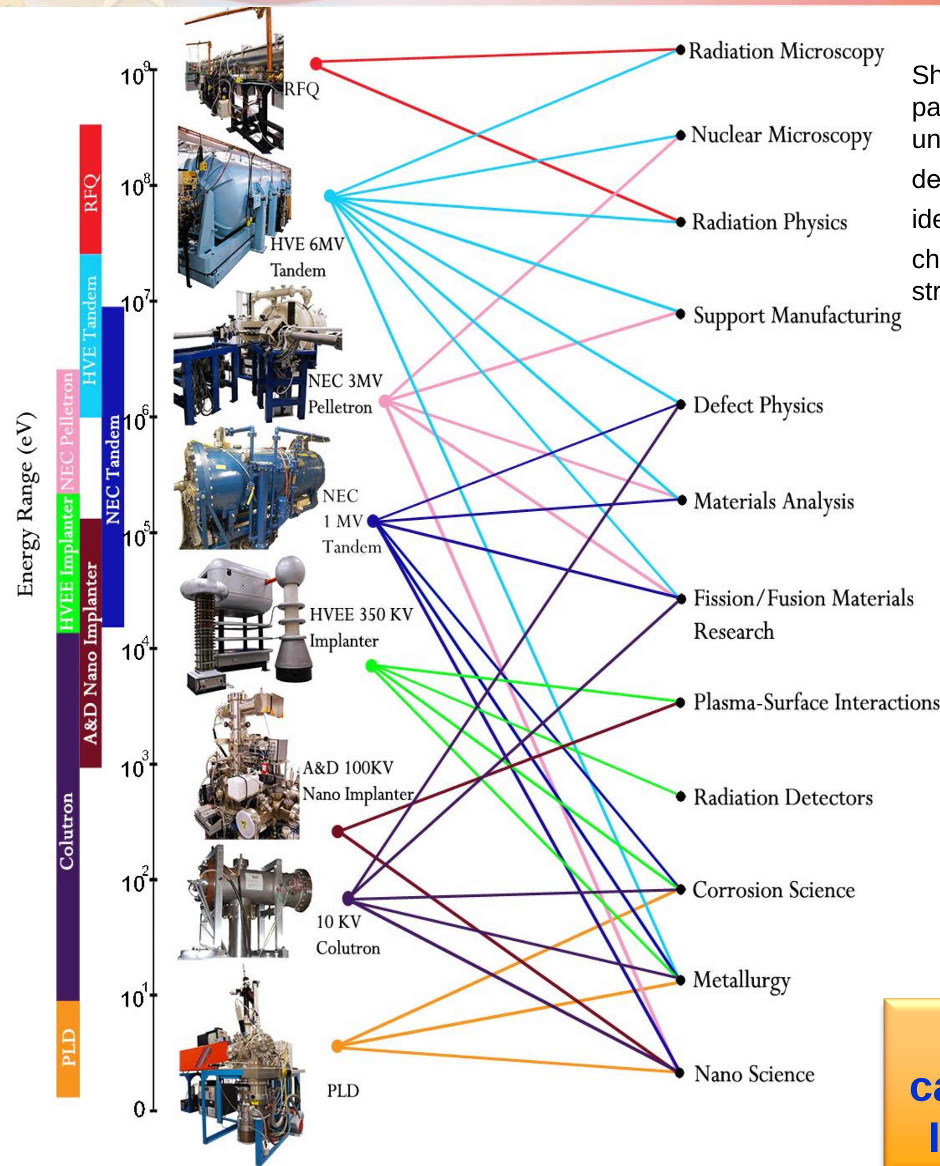
Workshop on Ion Irradiation TEM
Huddersfield, UK (2008)
Albuquerque, USA (2011)
Saporro, Japan (2013)



"The direct observation of ion damage in the electron microscope thus represents a powerful means of studying radiation damage"

D.W. Pashley and A.E.B. Presland Phil Mag. 6(68) 1961 p. 1003

Sandia's Ion Beam Laboratory

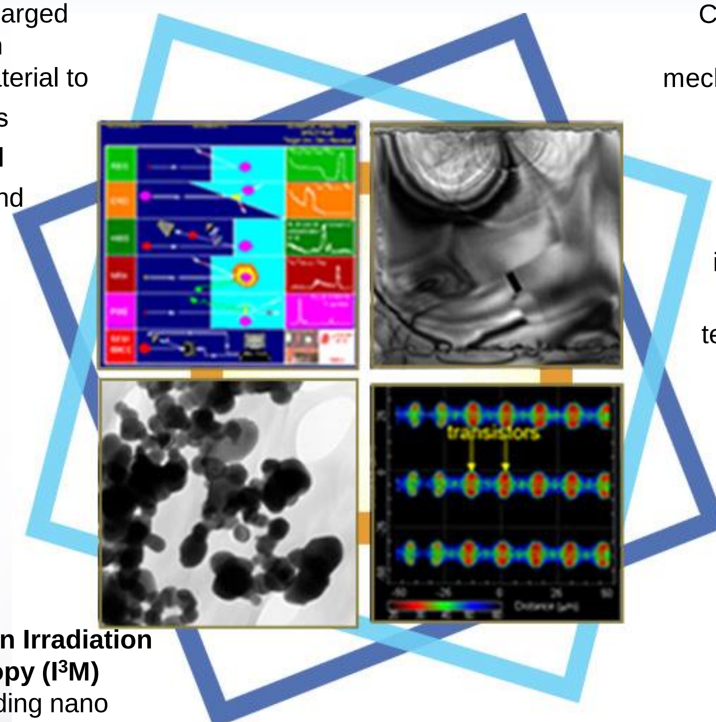


Ion Beam Analysis (IBA)

Shootinga charged particle at an unknown material to determine it's identity, local chemistry, and structure.

Ion Beam Modification (IBM)

Changing the optical, mechanical, and chemical properties of materials via ion implantation to meet technological needs



***In Situ* Ion Irradiation Microscopy (I³M)**

Bombarding nano samples with various particles and observing the changes in real time to understand how materials will behave in extreme environments.

Radiation Effects Microscopy (REM)

Using ion emissions to determine the Radiation hardness of microelectronics, identifying potential weaknesses.

The IBL has a unique and comprehensive capability ion beam set including and *In situ* Ion Irradiation Transmission Electron Microscopy.

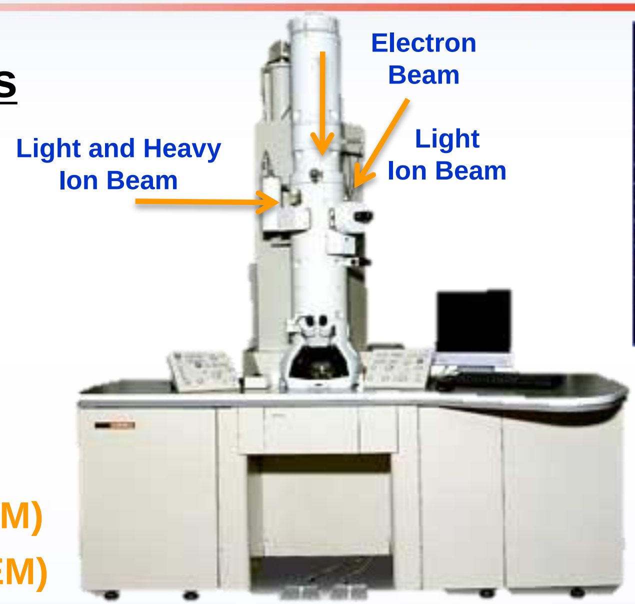
In situ Ion Irradiation TEM Facility

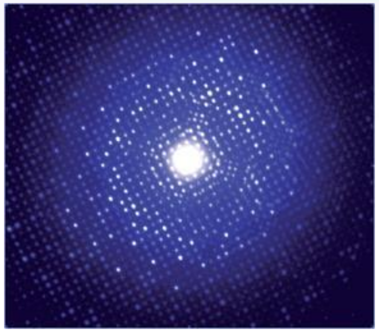
Proposed Capabilities

- 200 kV LaB_6 TEM
- Ion beams considered:
  - Range of Sputtered Ions
  - 10 keV D^{2+}
  - 10 keV He^{+}
- All beams hit same location

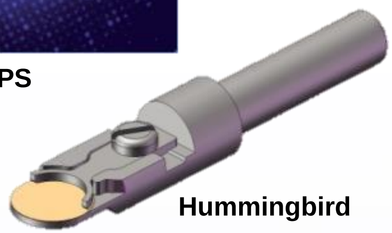
- Nanosecond time resolution (DTEM)
- Procession scanning (EBSD in TEM)
- *In situ* PL, CL, and IBIL

- *In situ* vapor phase stage
- *In situ* liquid mixing stage
- *In situ* heating
- Tomography stage (2x)
- *In situ* cooling stage
- *In situ* electrical bias stage
- *In situ* straining stage

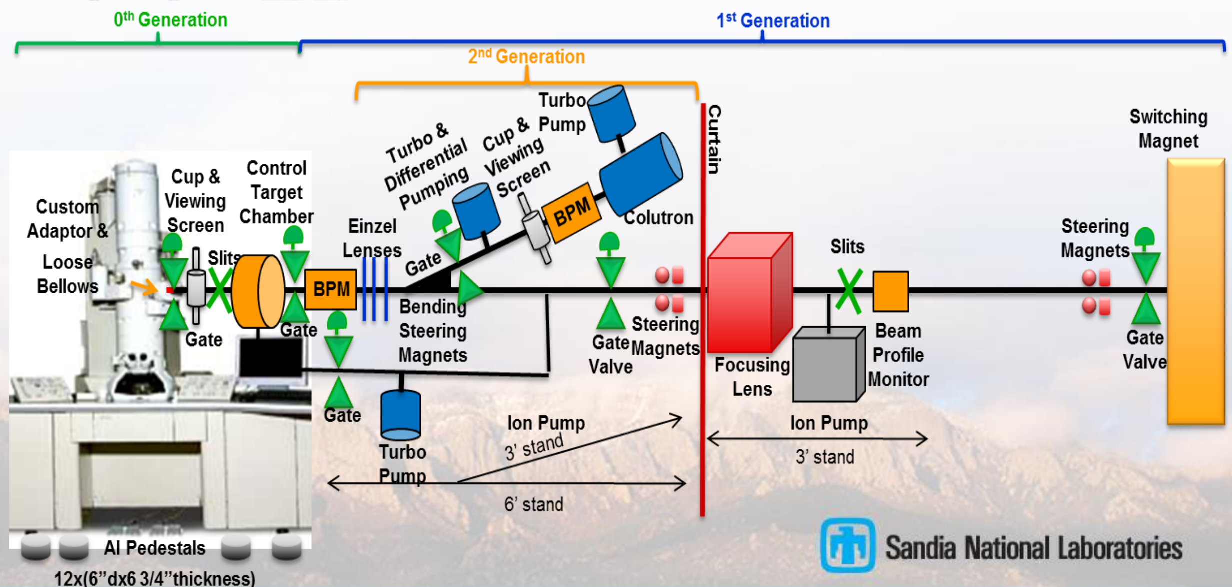




TVIPS

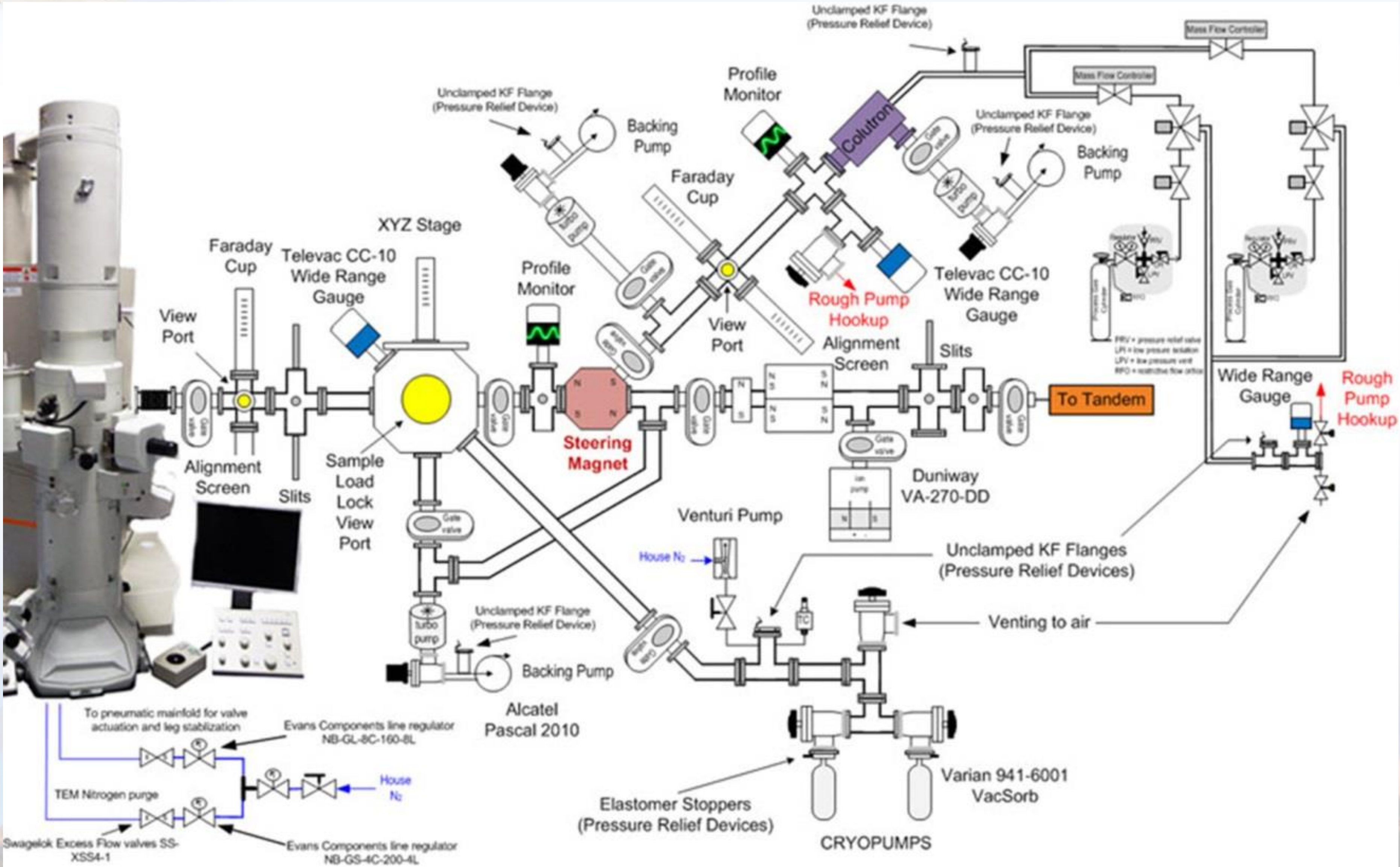


Hummingbird



Schematic of the *In situ* TEM Beamline

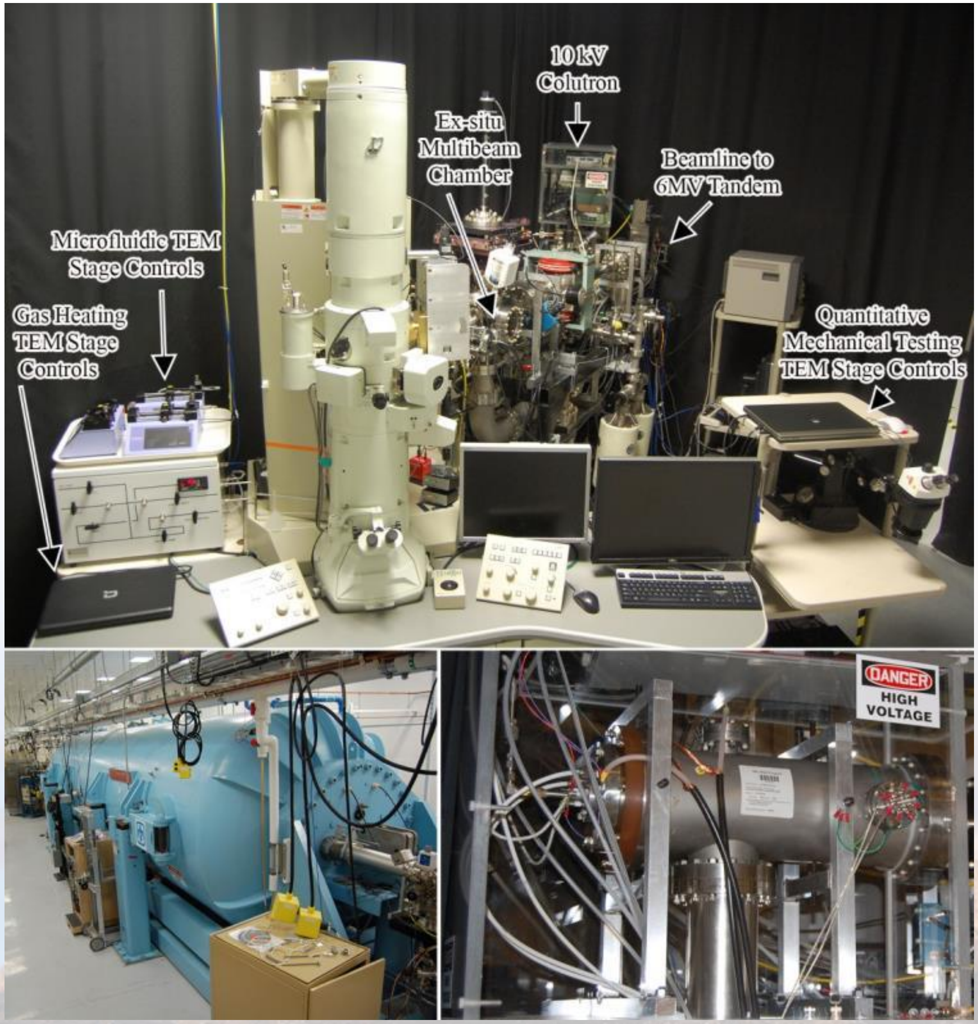
Collaborators: M.T. Marshall J.A. Scott, & D.L. Buller



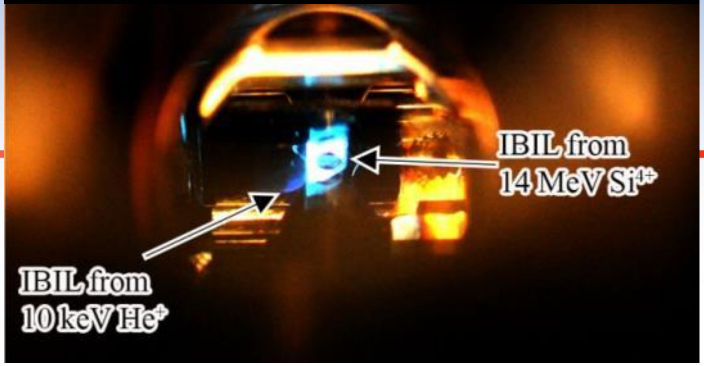
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem

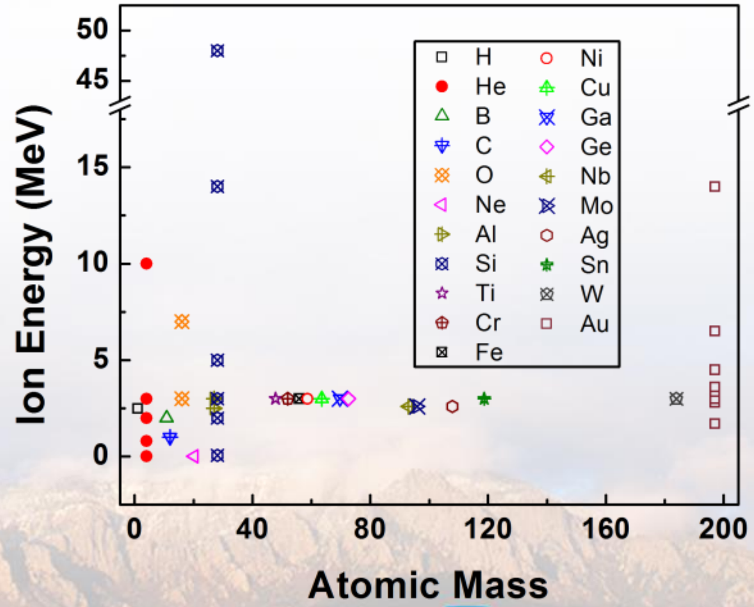


IBIL from a quartz stage inside the TEM



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

Ion species & energy introduced into the TEM



Sandia National Laboratories

Cumulative Effects of Ion Irradiation as a Function of Ion Energy and Au Particle Size

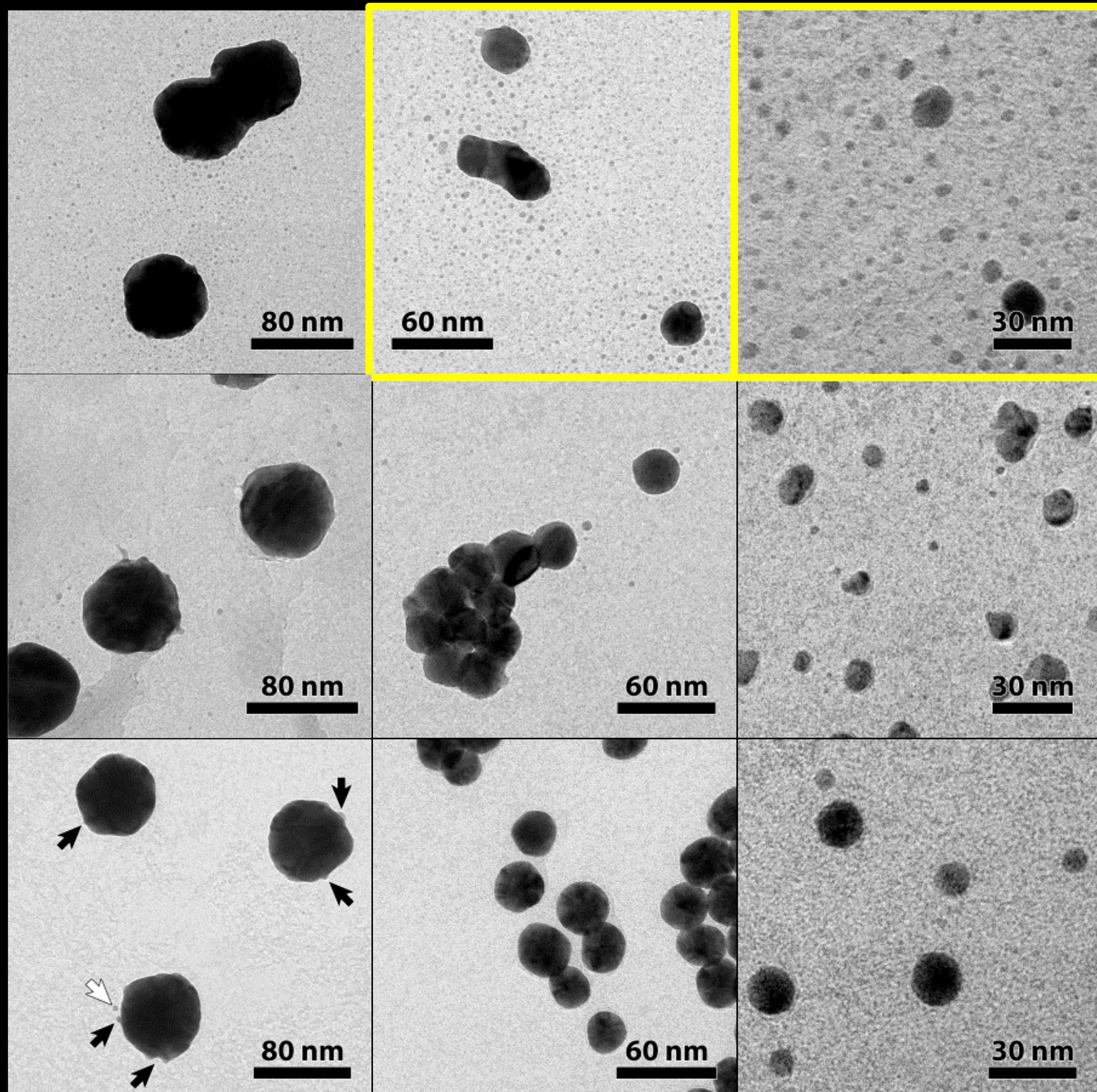
Collaborator: D.C. Bufford

60 nm | 20 nm | 5 nm

46 keV Au^{1-}
3.4×10^{14} /cm²

2.8 MeV Au^{4+}
4×10^{13} /cm²

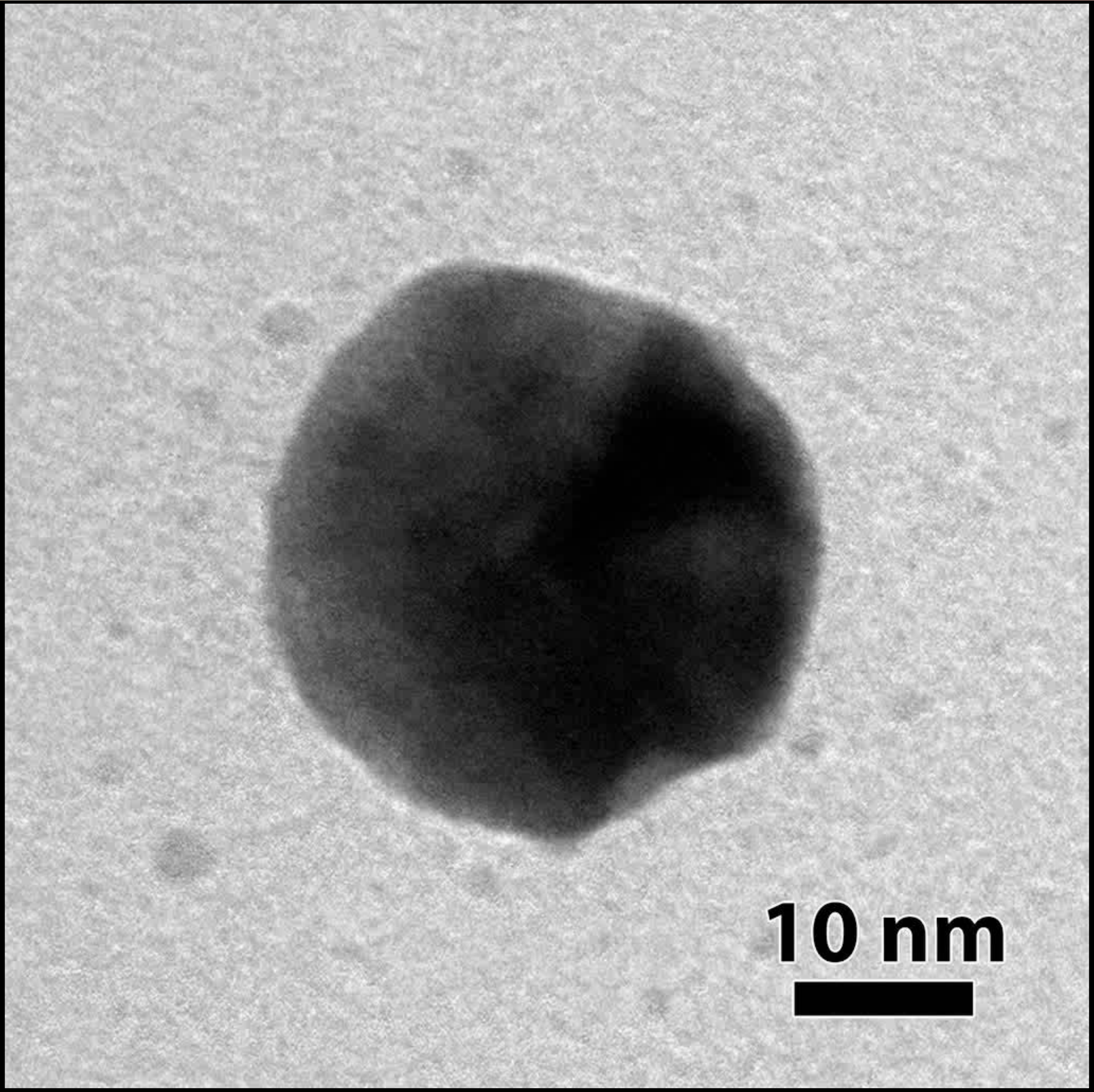
10 MeV Au^{8+}
1.3×10^{12} /cm²

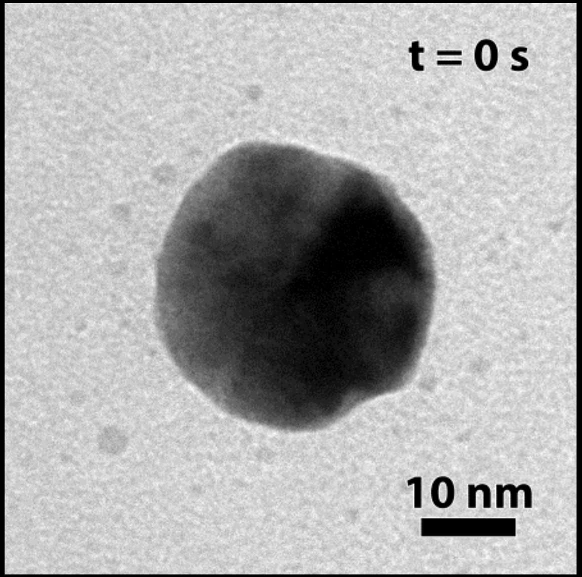


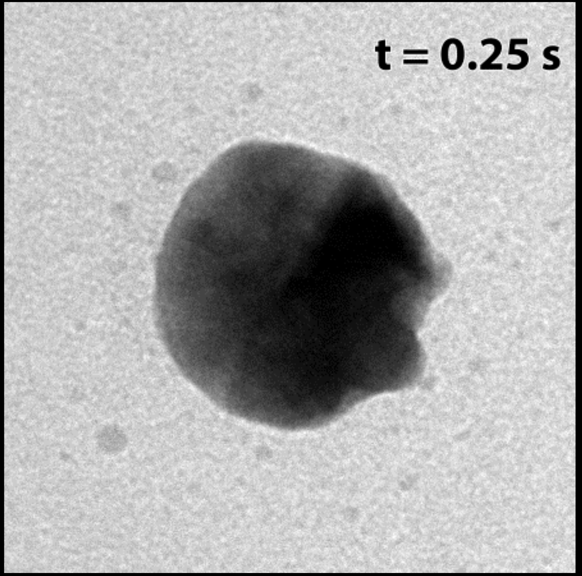
Particle and ion energy dictate the ratio of sputtering, particle motion, particle agglomeration, and other active mechanisms

Single Ion Effects with 46 keV Au^{1-} ions: 20 nm

Collaborator: D.C. Bufford

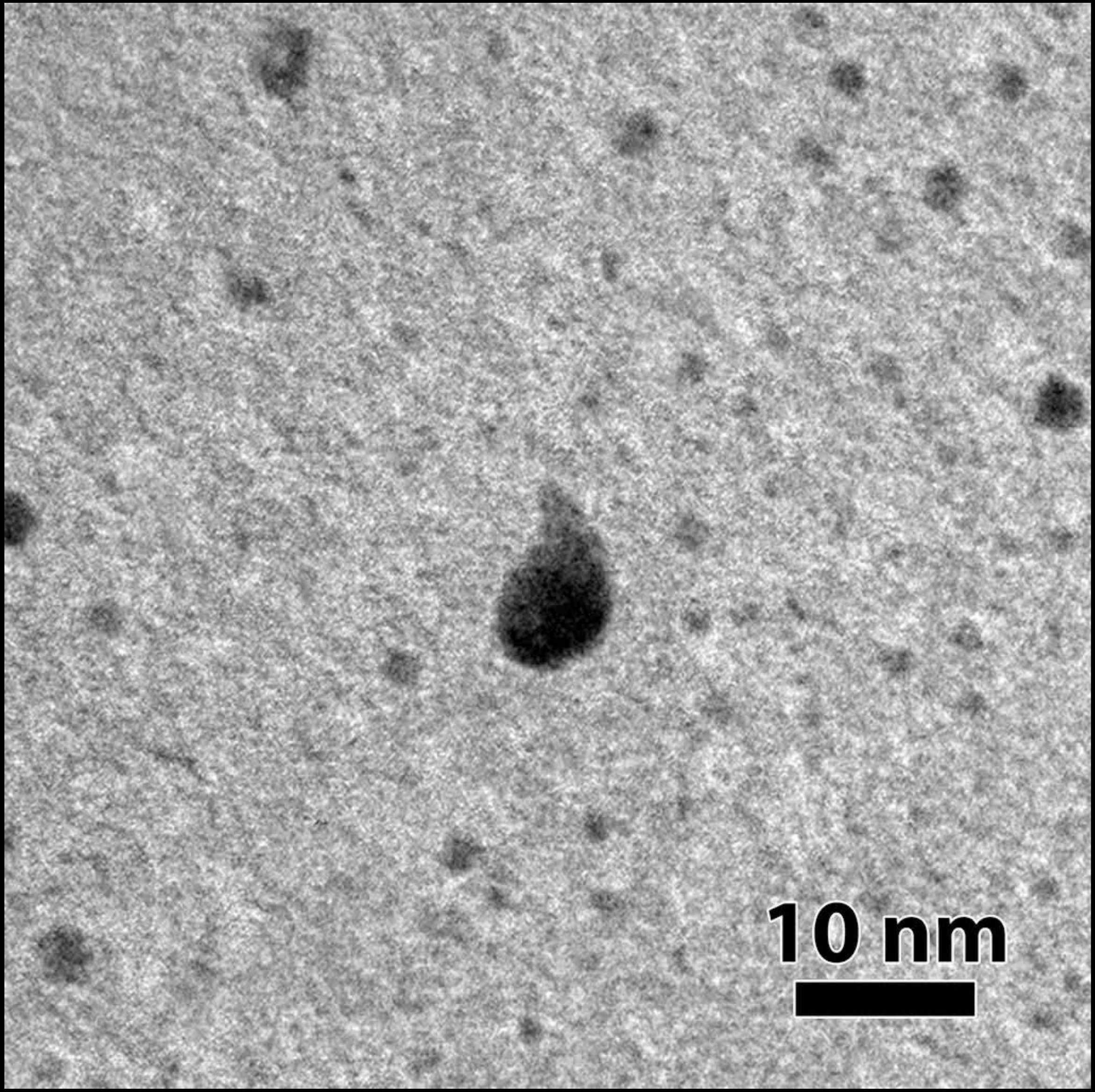


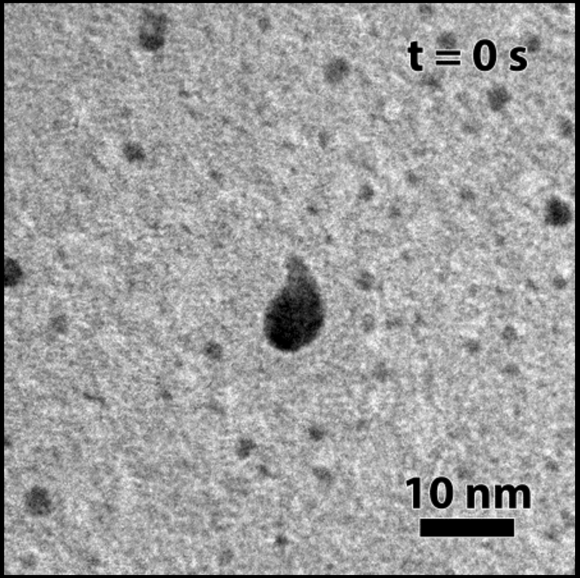


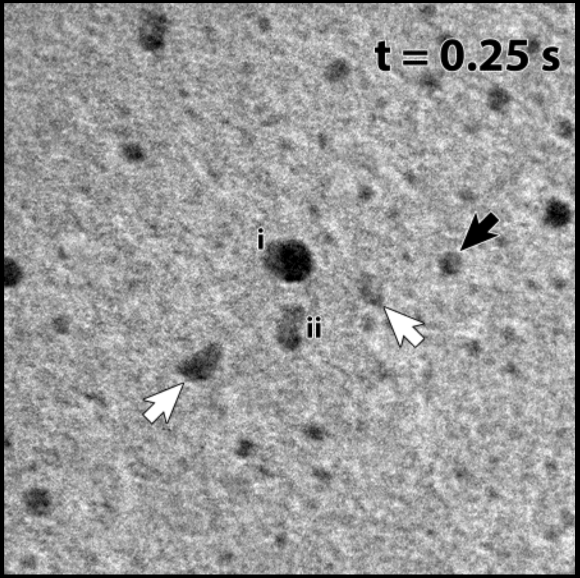


Single Ion Effects with 46 keV Au^{1-} ions: 5 nm

Collaborator: D.C. Bufford







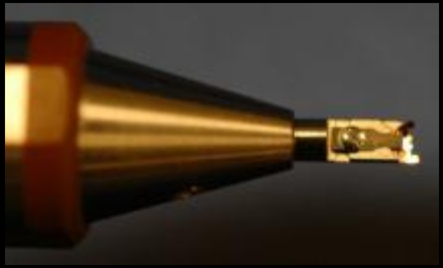
Advanced Microscopy Techniques Applied to Nanoparticles in Radiation Environments

Collaborators: S.M. Hoppe & T.J. Boyle

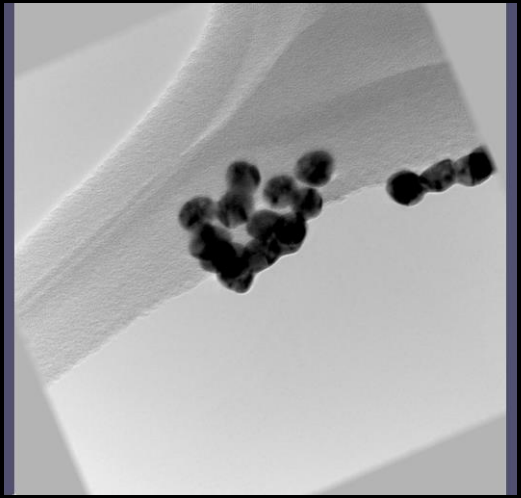
In situ Ion Irradiation TEM (I³TEM)

\+

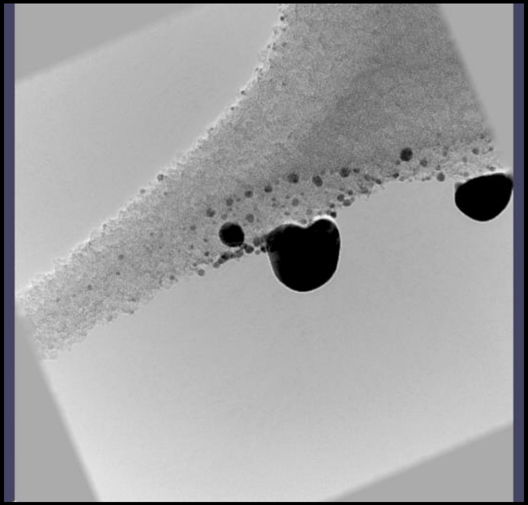
Hummingbird tomography stage



Aligned Au NP tilt series - unirradiated

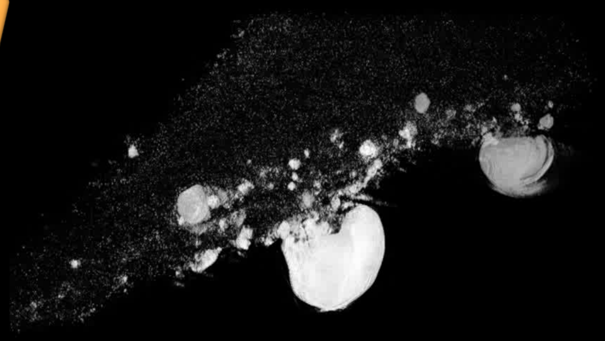


Aligned Au NP tilt series - irradiated



Unirradiated Au NP model

Irradiated Au NP model

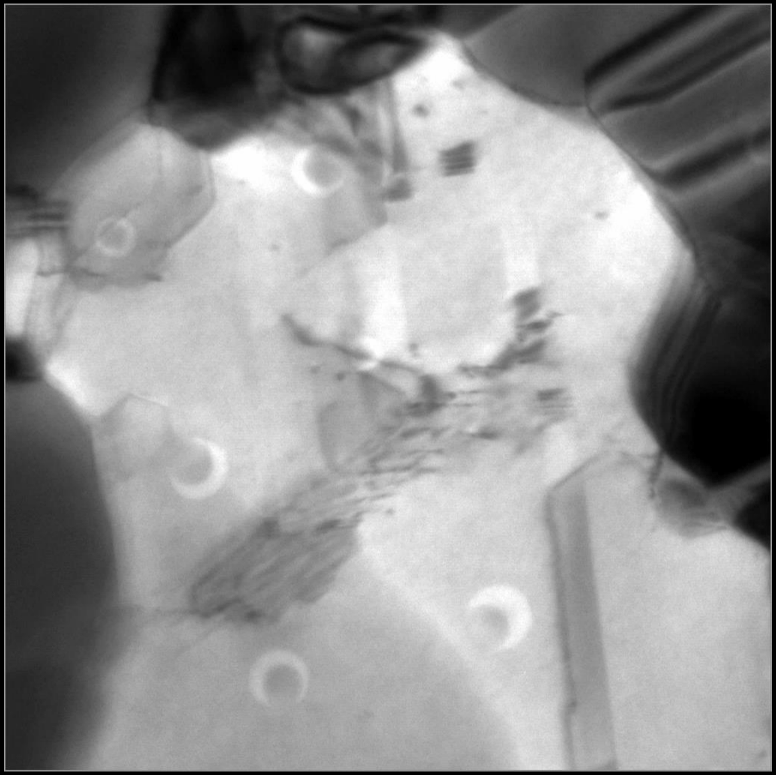


The application of advanced microscopy techniques to extreme environments provides exciting new research directions

Single Ion Strikes

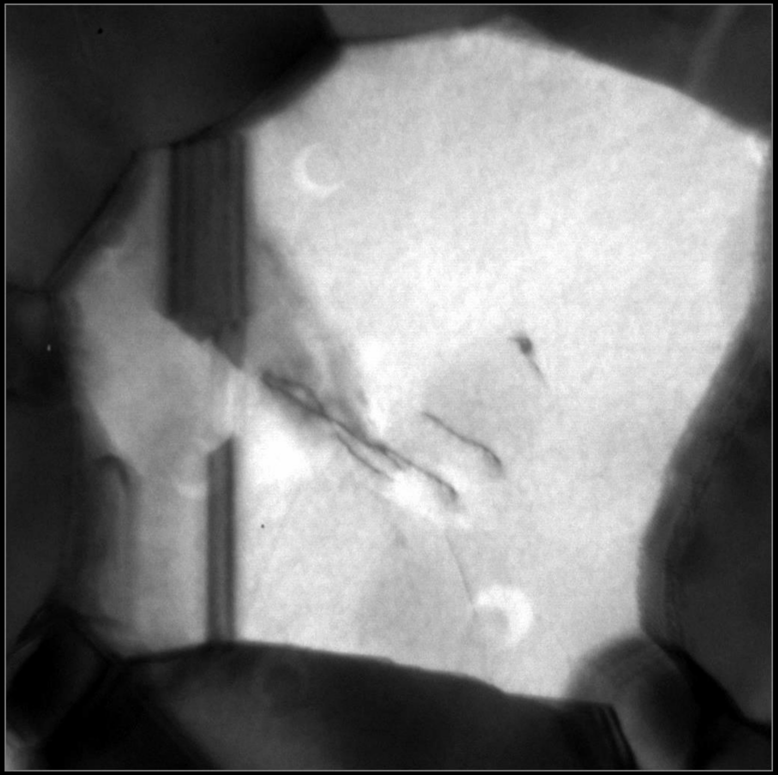
Collaborators: C. Chisholm & A. Minor

7.9 x 10^9 ions/cm^2/s



VS

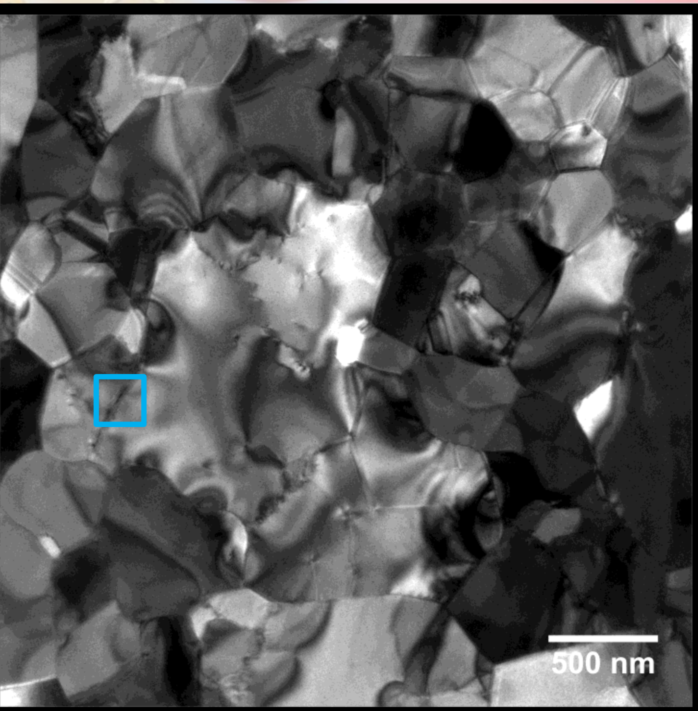
6.7 x 10^7 ions/cm^2/s

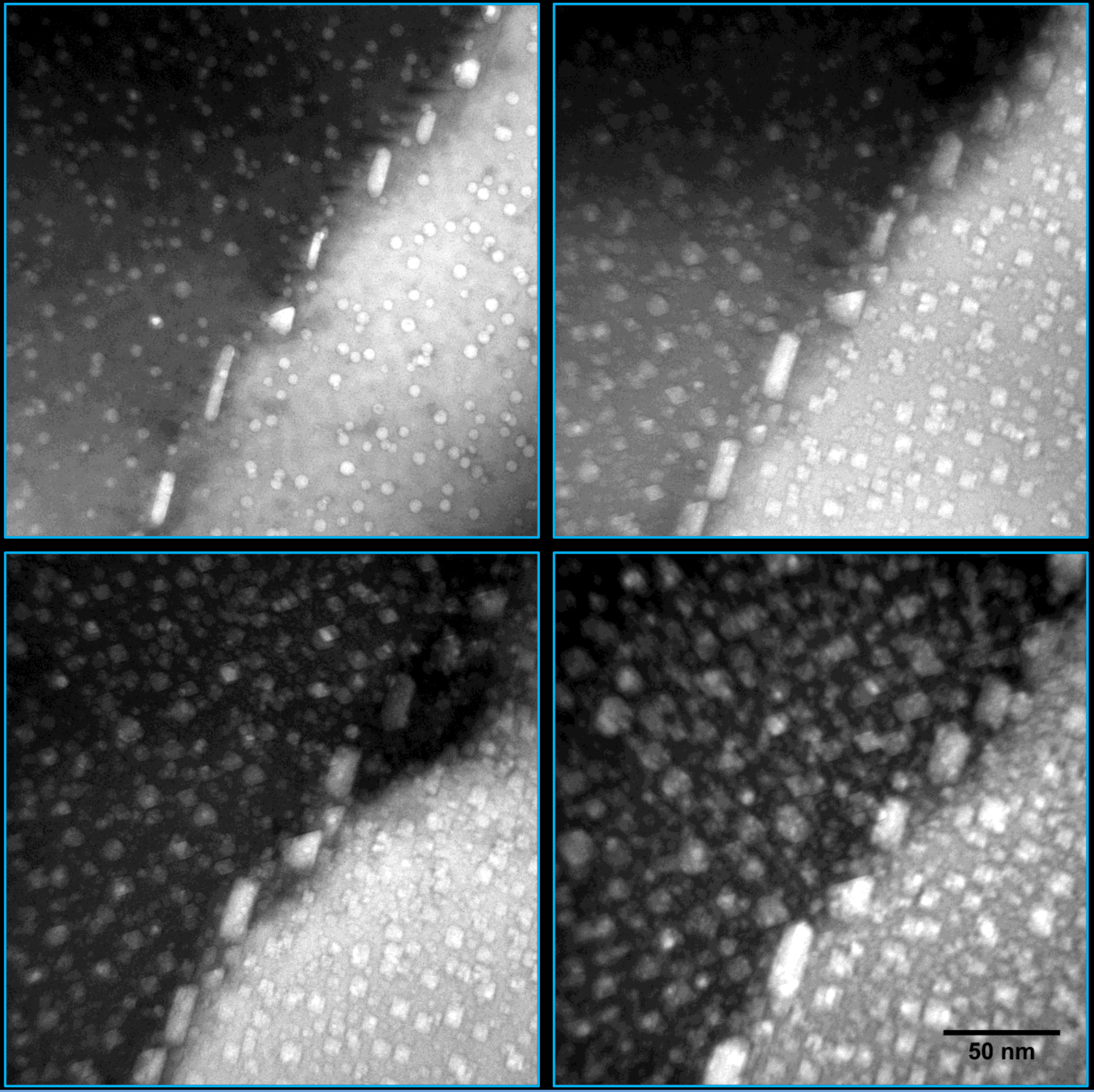


Improved vibrational and ion beam stability permits us to work at 120kx or higher permitting imaging of single cascade events

In situ Implantation

Collaborators: C. Chisholm & A. Minor

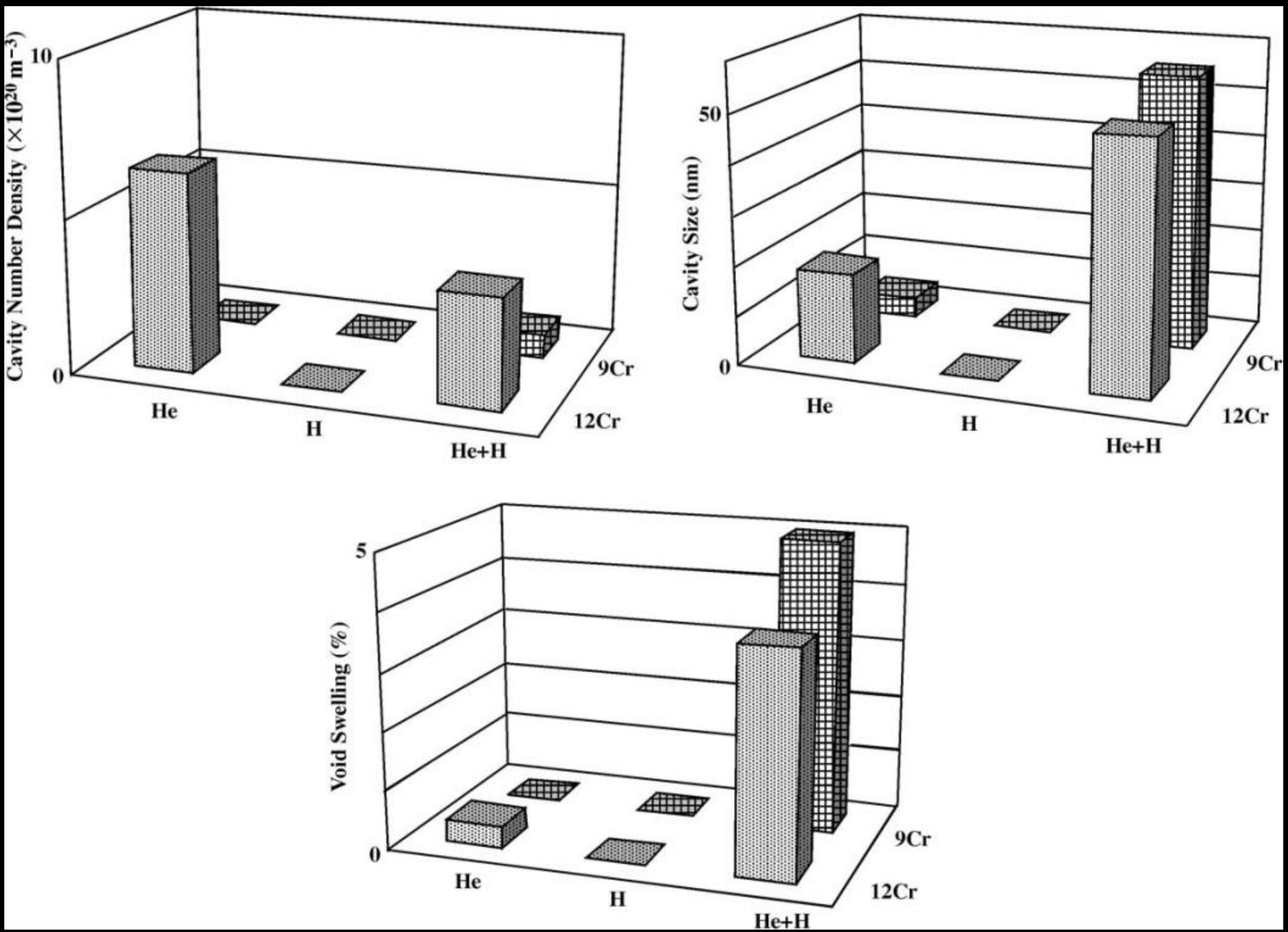




Gold thin-film implanted with 10keV He^{2+}

Result: porous microstructure

H, He, and Displacement Damage Synergy



T. Tanaka et al. "Synergistic effect of helium and hydrogen for deffect evolution under milt-ion irradiation of Fe-Cr ferritic alloys"

J. of Nuclear Materials 329-333 (2004) 294-298

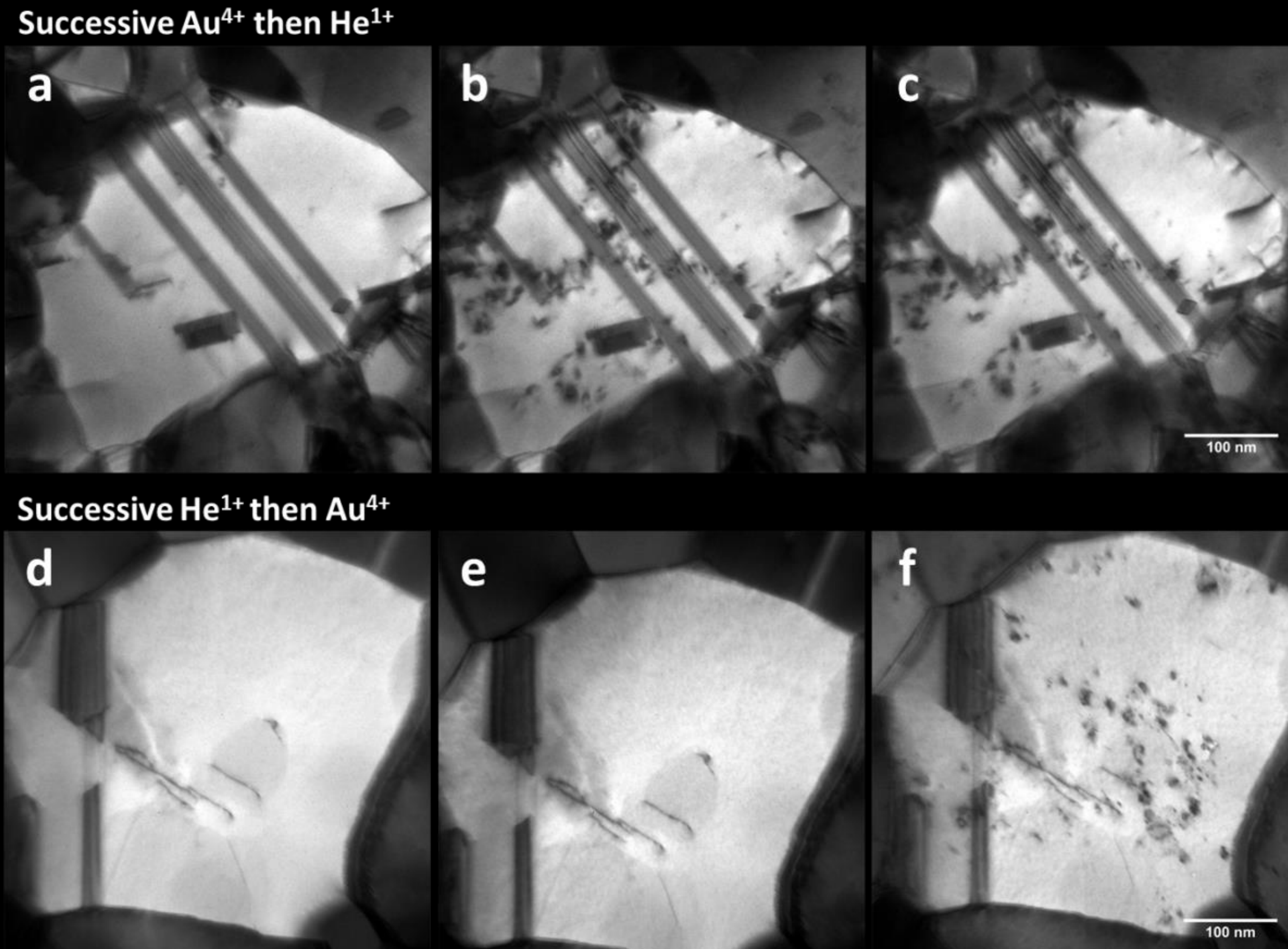
Coupling Effect

- H and He are produced as decay products
- The relationship between the point defects present, the interstitial hydrogen, and the He bubbles in the system that results in the increased void swelling has only been theorized.
- The mechanisms which governs the increased void swelling under the presence of He and H have never been experimental determined

No capability currently exist for triple beam irradiation in the U.S. and No capability for tripple beam TEM ion irradiation exists in the world

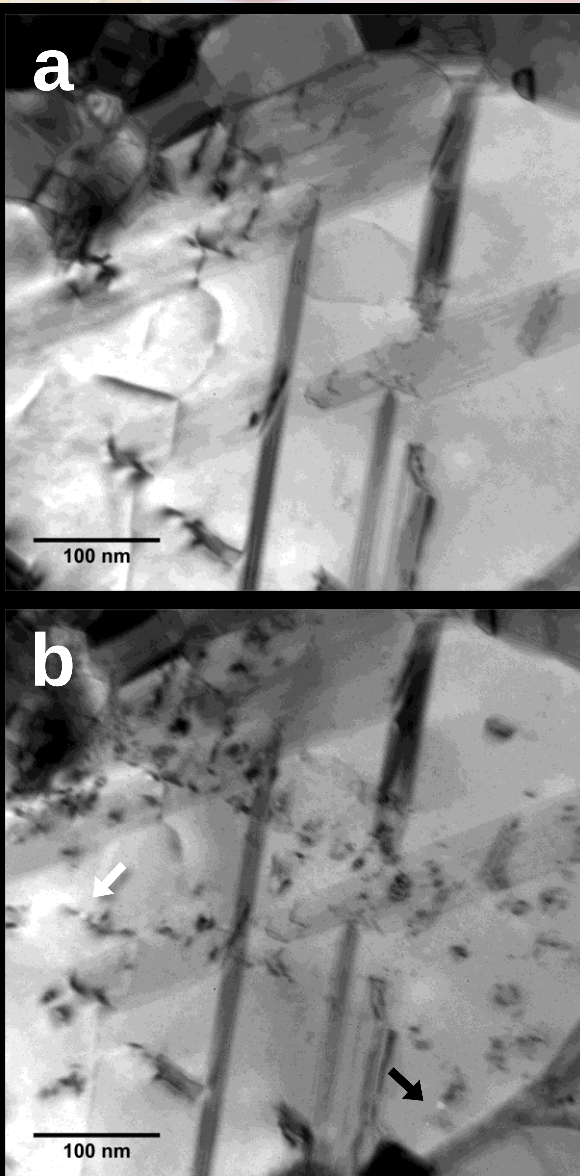
In situ Successive Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor

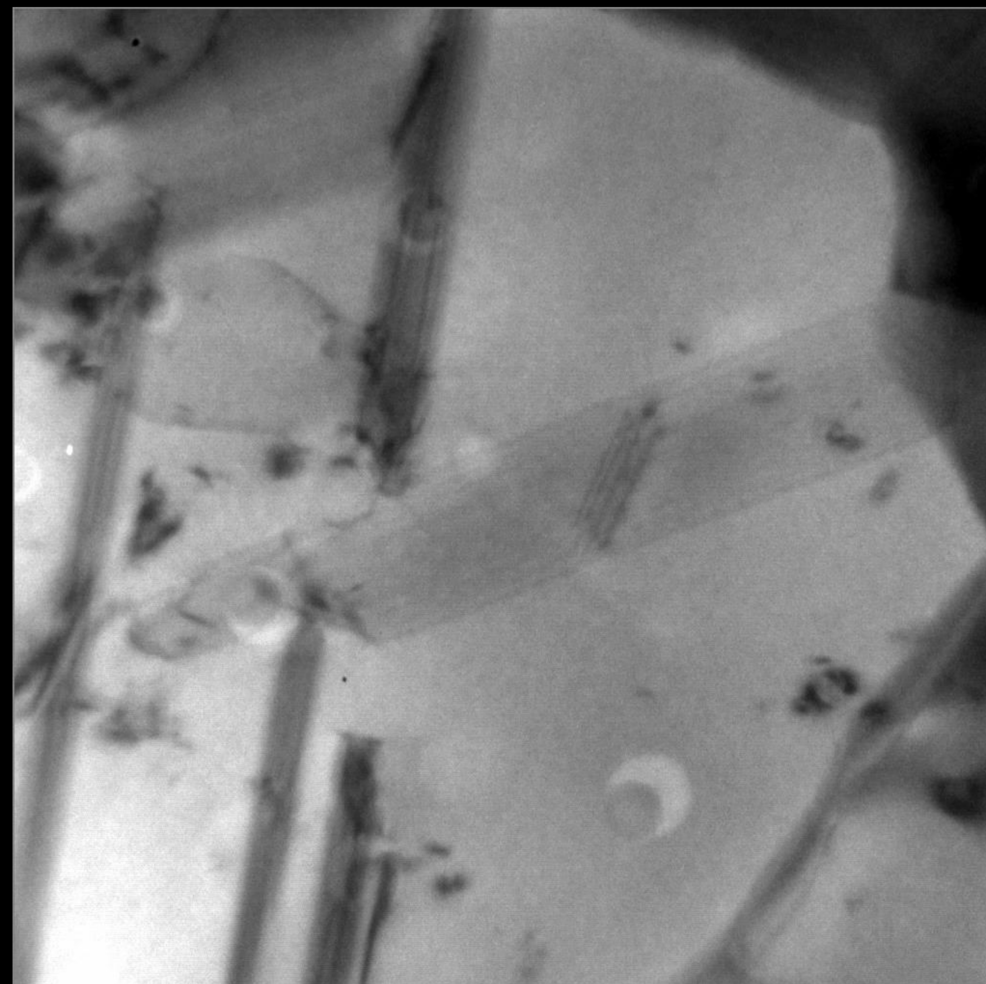


In situ Concurrent Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor

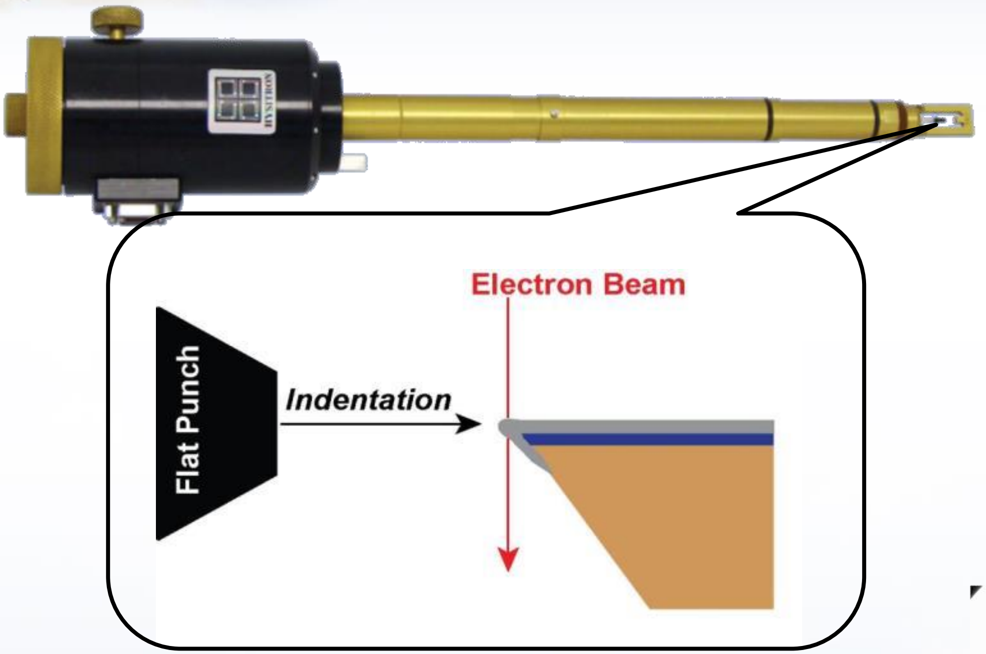


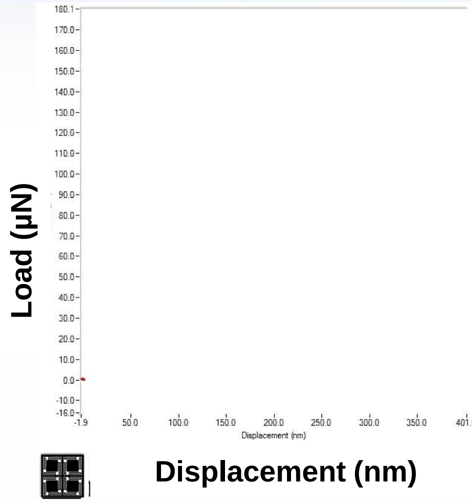
He^{1+} implantation and Au^{4+} irradiation of a gold thin film

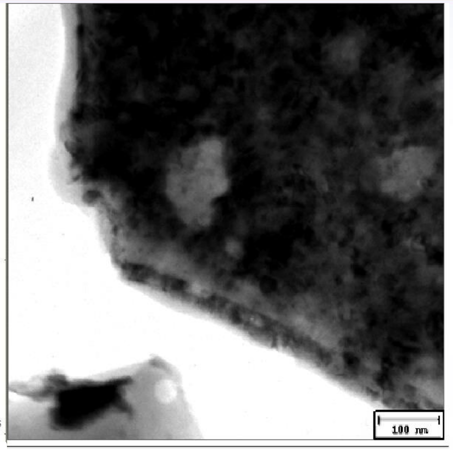


In situ TEM Quantitative Mechanical Testing

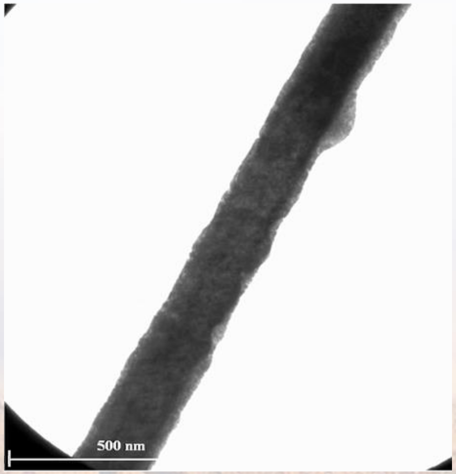
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

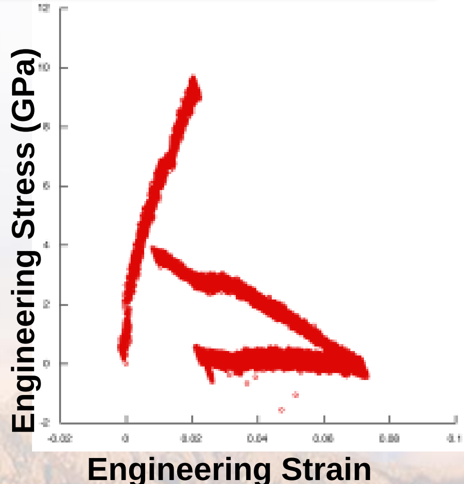






Fundamentals of Mechanical Properties





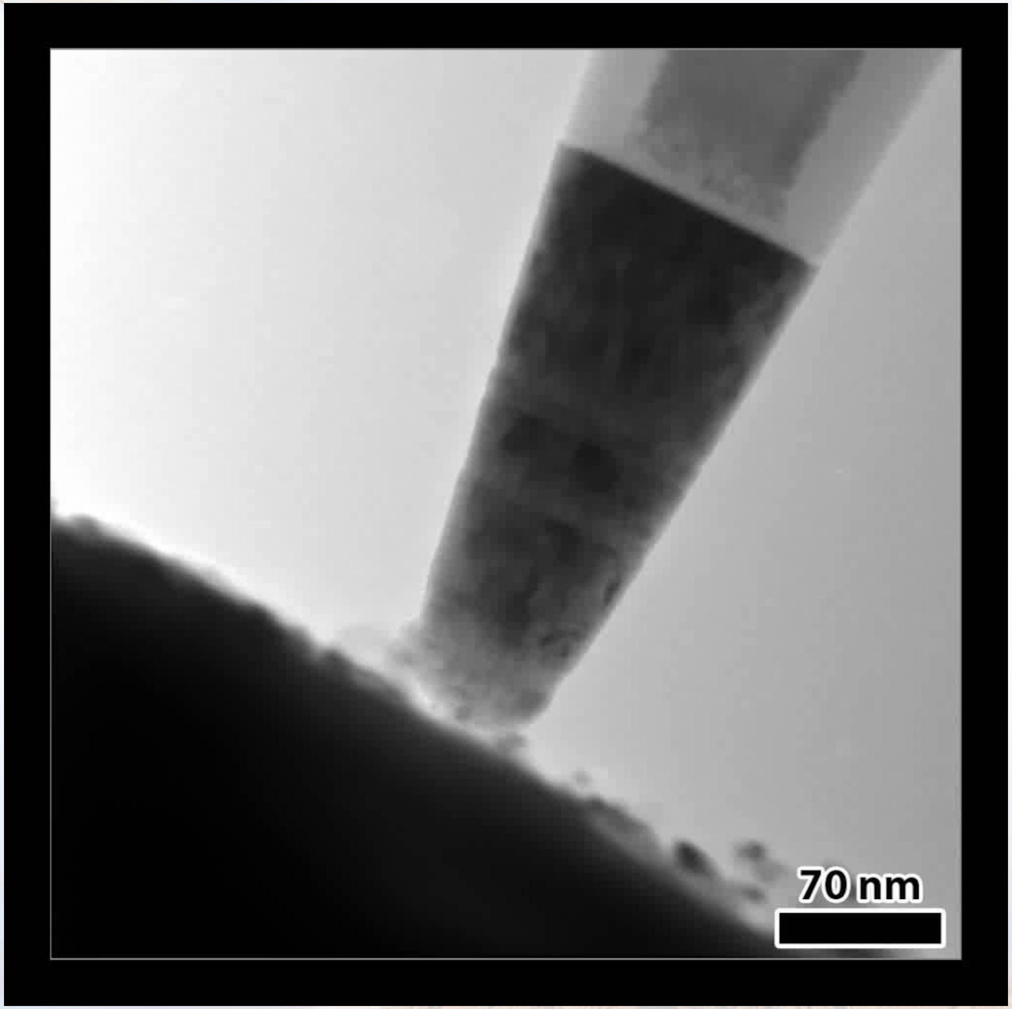
Range of Mechanical Testing Techniques

- Indentation
- Compression
- Tension
- Bending
- Wear
- Fatigue
- Creep

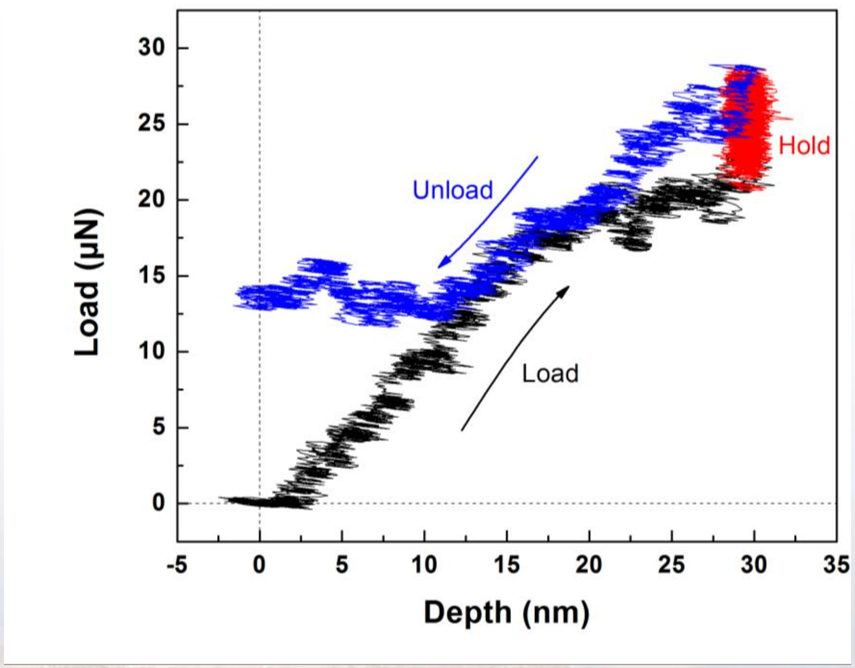
We have started looking at the effects of ion irradiation on mechanical properties

NC Ni Pillar Indentation

Collaborator: D.C. Bufford & W.M. Mook



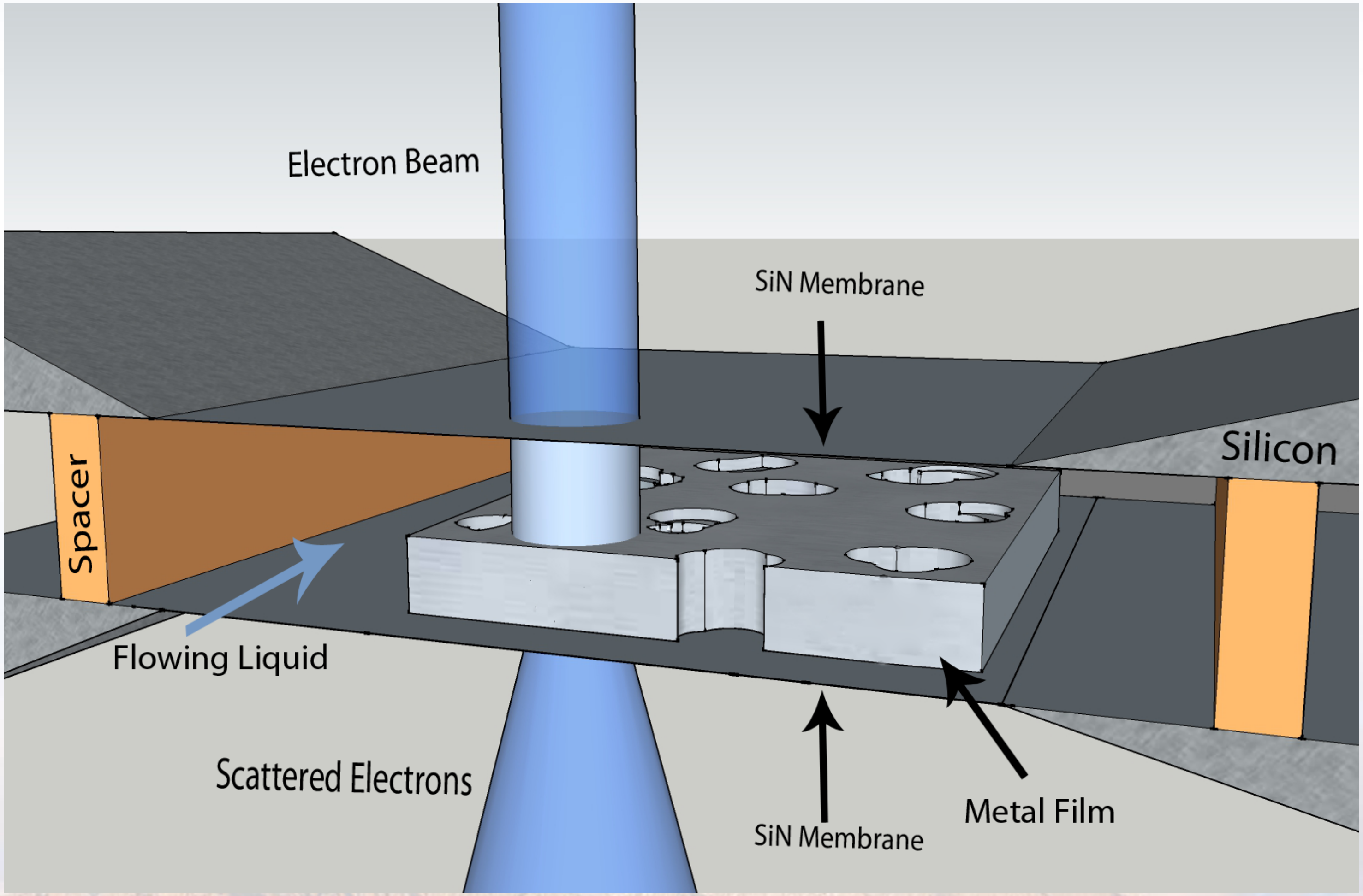
- 0.5 nm/s loading rate
- Trapezoid load function
- 60s load/60s hold/60s unload



Can We Gain Insight into the Corrosion Process through *In situ* TEM?

<u>Microfluidic Stage</u>

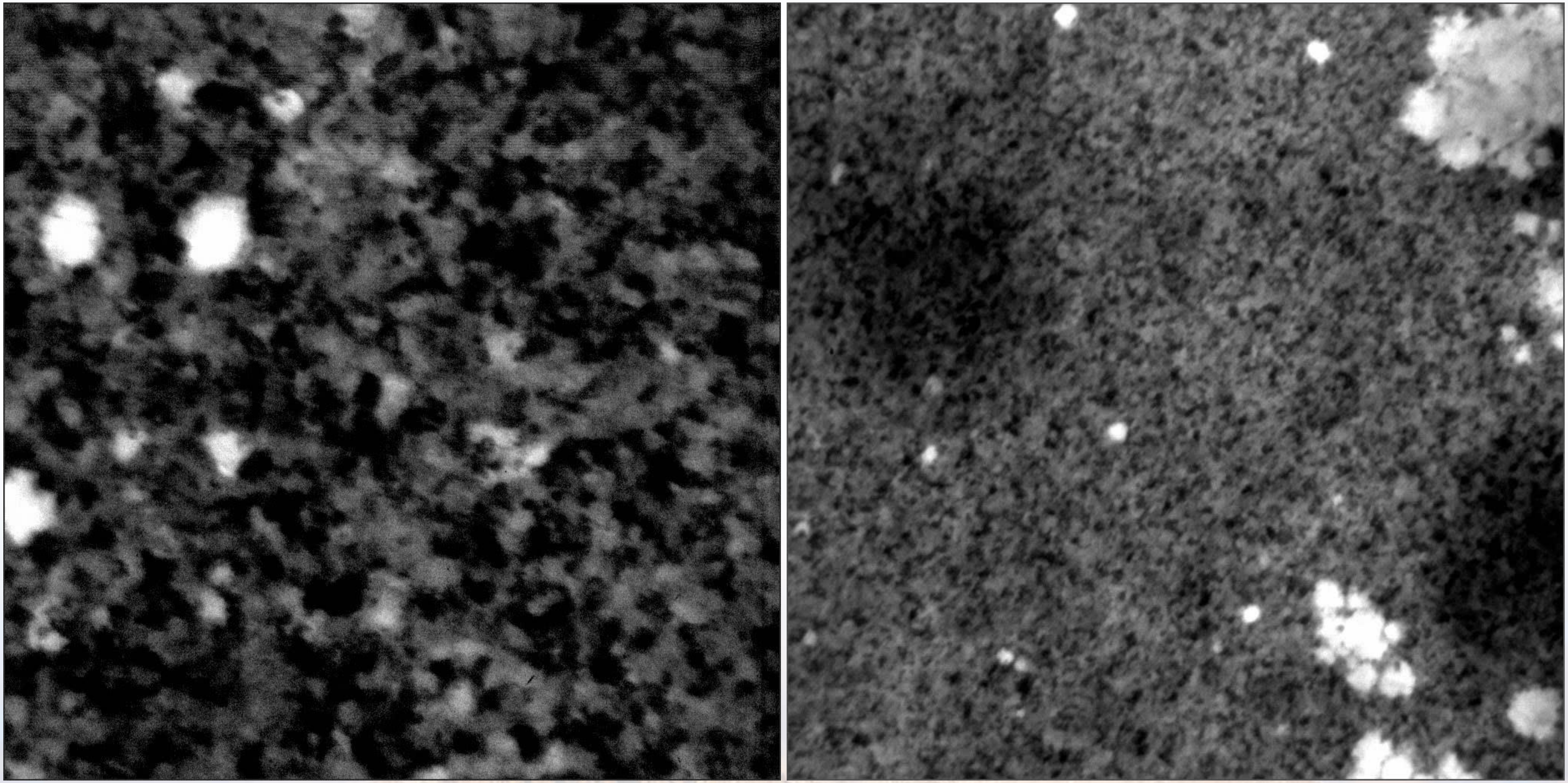
- **Mixing of two or more channels**
- **Continuous observation of the reaction channel**
- **Chamber dimensions are controllable**
- **Films can be directly deposited on the electron transparent SiN membrane**



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

Acetic Acid Corroding Nanograined Iron

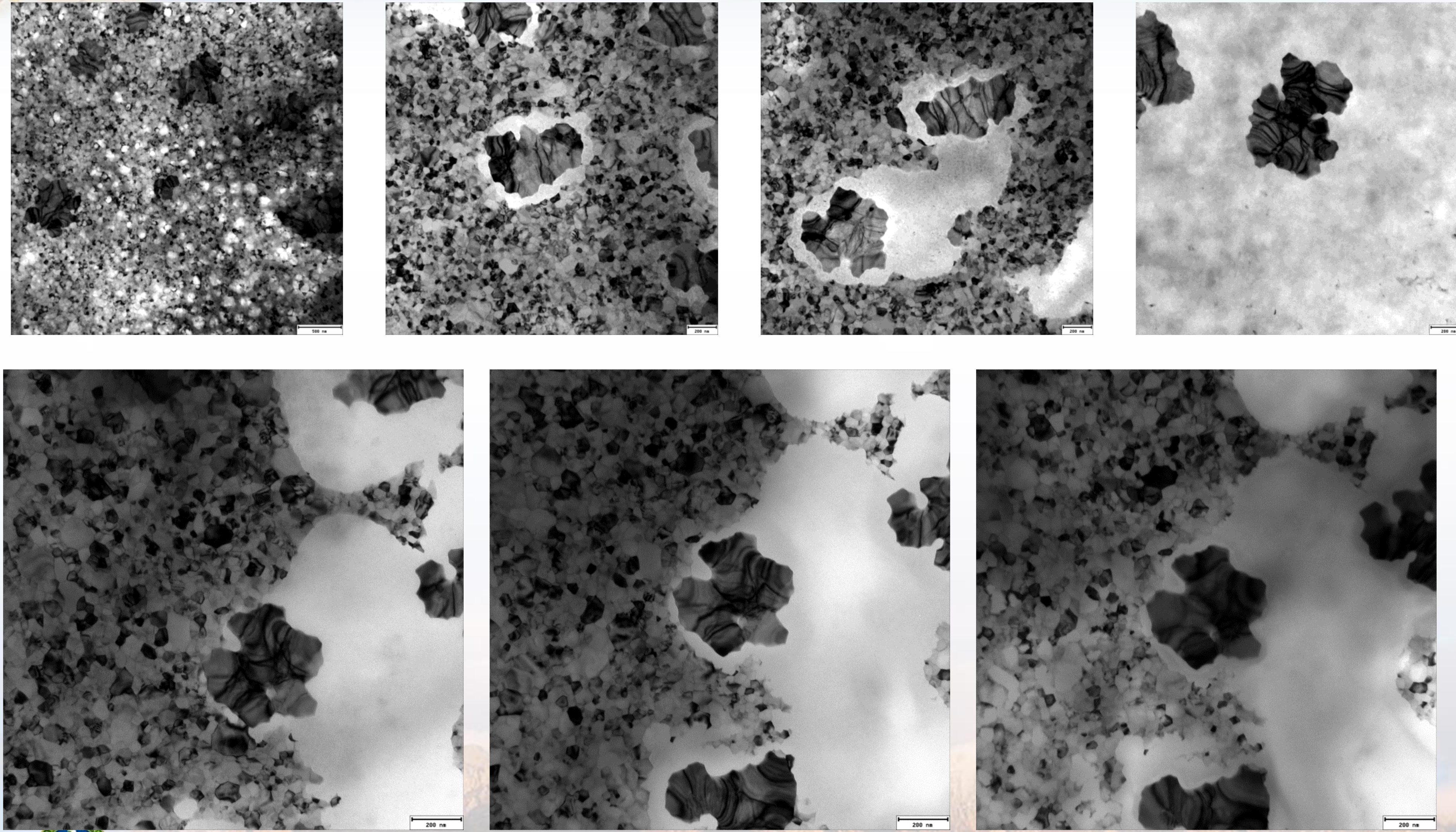
Collaborators: D. Gross, J. Kacher, & I.M. Robertson



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

Acetic Acid Corroding in Annealed Nanograined Iron

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



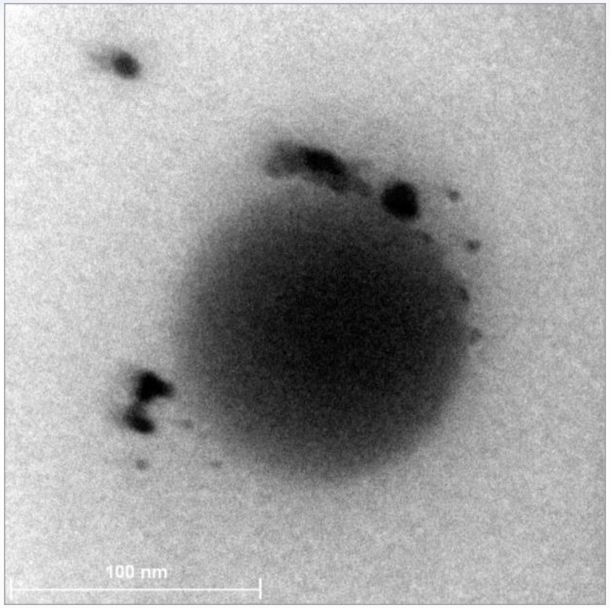
Large grains resulting from annealing appear more corrosion tolerant

Other Fun Uses of Microfluidic Cell

Protocell Drug Delivery

S. Hoppe,
E. Carnes,
J. Brinker

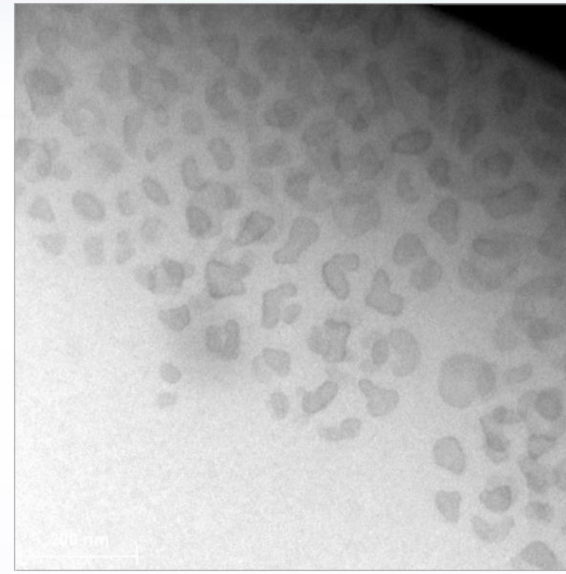
Liposome encapsulated Silica destroyed by the electron beam



BSA Crystallization

S. Hoppe

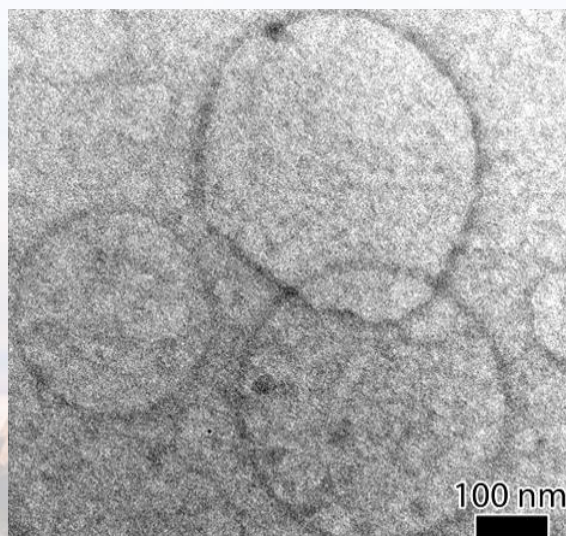
Crystallization of excess Bovine Serum Albumen during flow



Liposomes in Water

S. Hoppe,
D. Sasaki

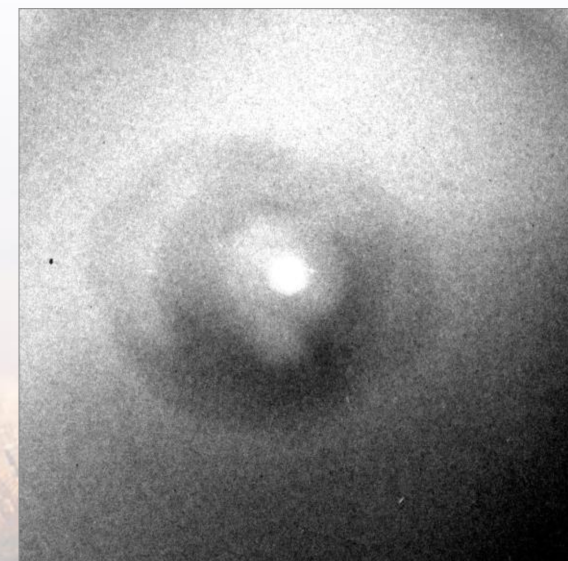
Liposomes imaged in flowing aqueous channel



La Structure Formation

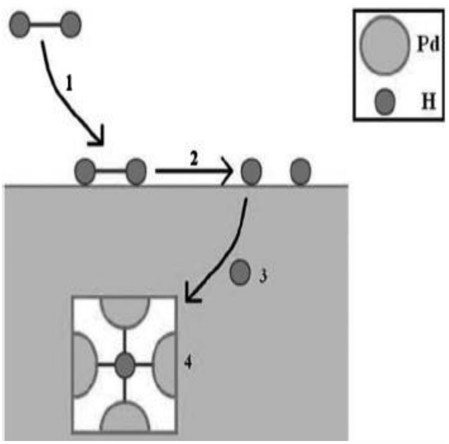
S. Hoppe,
T. Nenoff

La Nanostructure form from $LaCl_3$ H_2O in wet cell due to beam effects

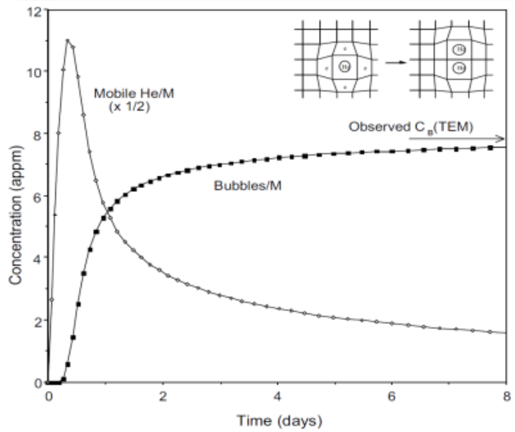


Can *In situ* TEM Address Hydrogen Storage Concerns in Extreme Environments?

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

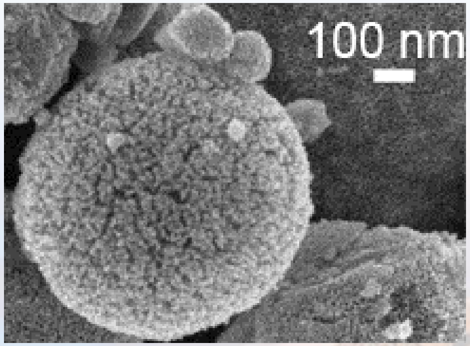


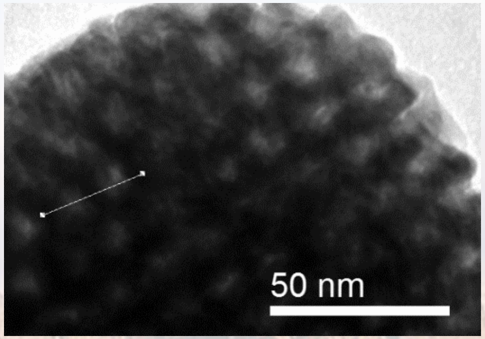
Cowgill, D., Fusion Sci. & Tech., 28 (2005) p. 539

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

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

Harmful effects may be mitigated in nanoporous Pd

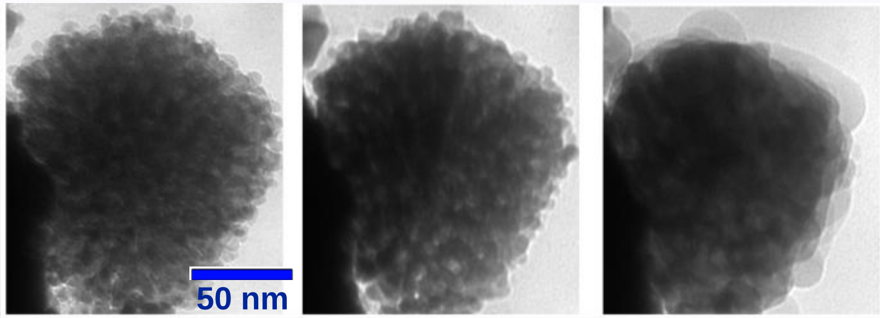




Vapor-Phase Heating TEM Stage

- 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 H_2 after several pulses to specified temp.

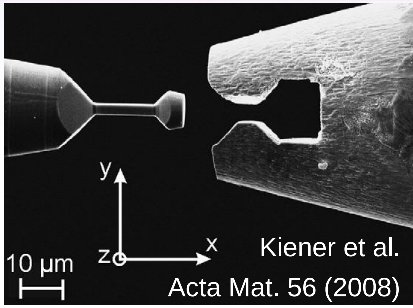


125° C | 200° C | 300° C

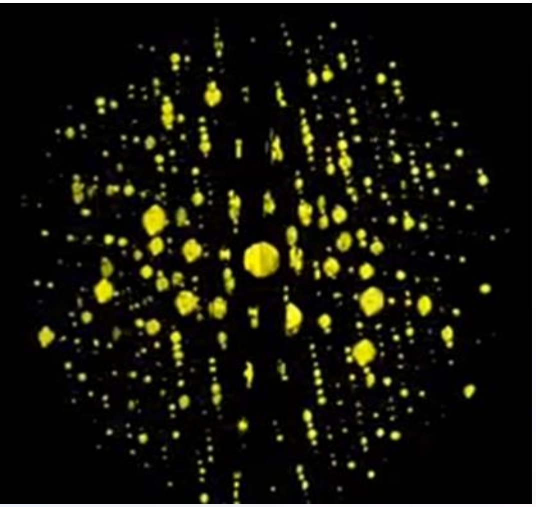
New *in situ* atmospheric heating experiments provide great insight into nanoporous Pd stability

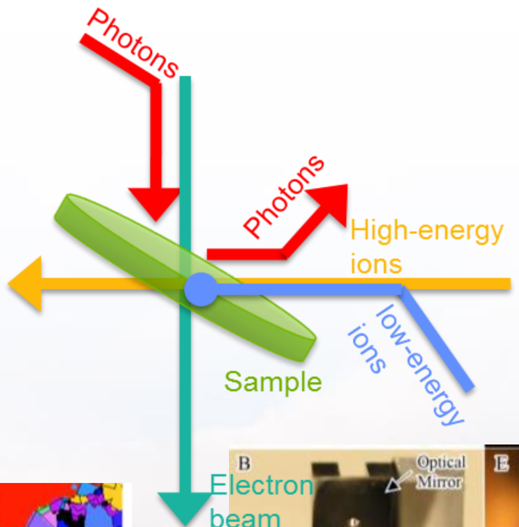
Future Directions Under Pursuit

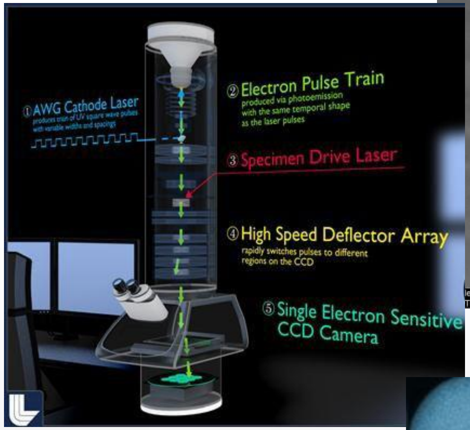
1. In-situ TEM CL, IBIL (currently capable)
2. *In situ* ion irradiation TEM in liquid or gas (currently capable)
3. PED: Local texture characterization (arriving FY15)
4. Quantative in-situ tensile/creep experiments (Sample in development)
5. DTEM: Nanosecond resolution (laser optics needed)

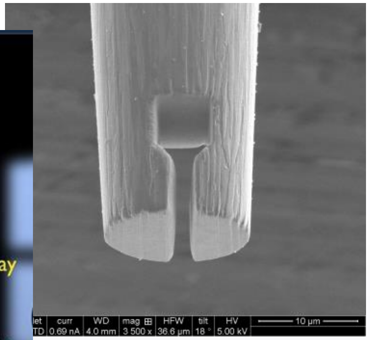


Kiener et al. Acta Mat. 56 (2008)

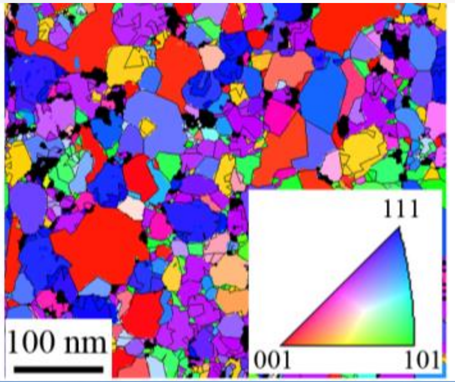


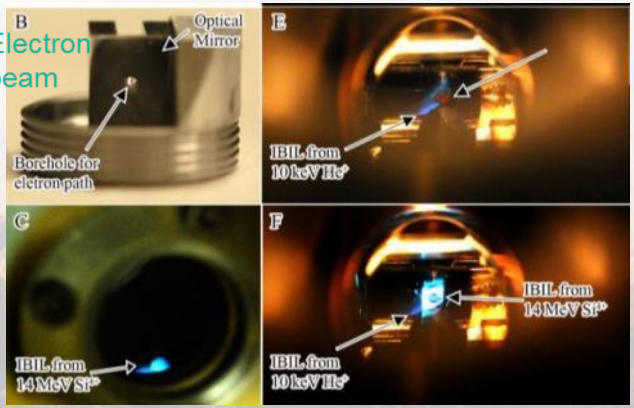


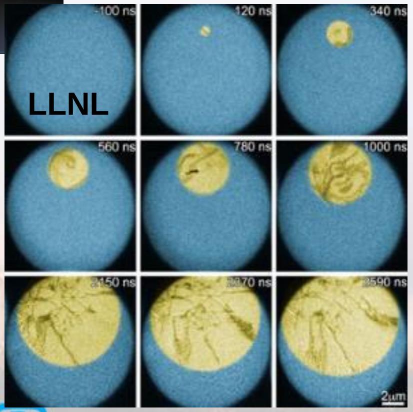




AppFive NanoMegas







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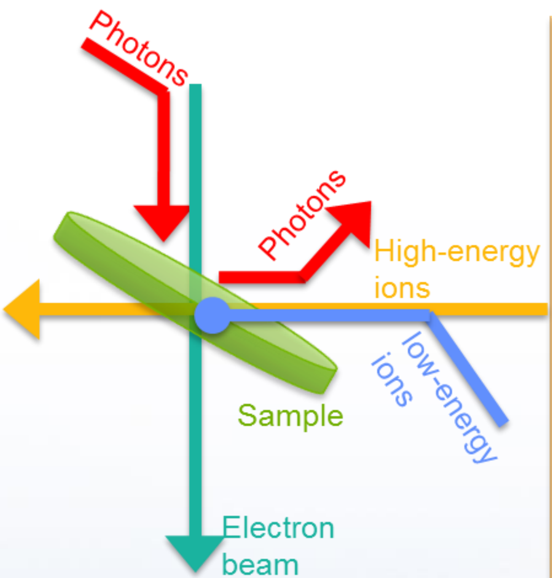
Summary

The Ion Beam Lab at Sandia National Laboratories applies a variety of nanoscale tools to a wealth of problems

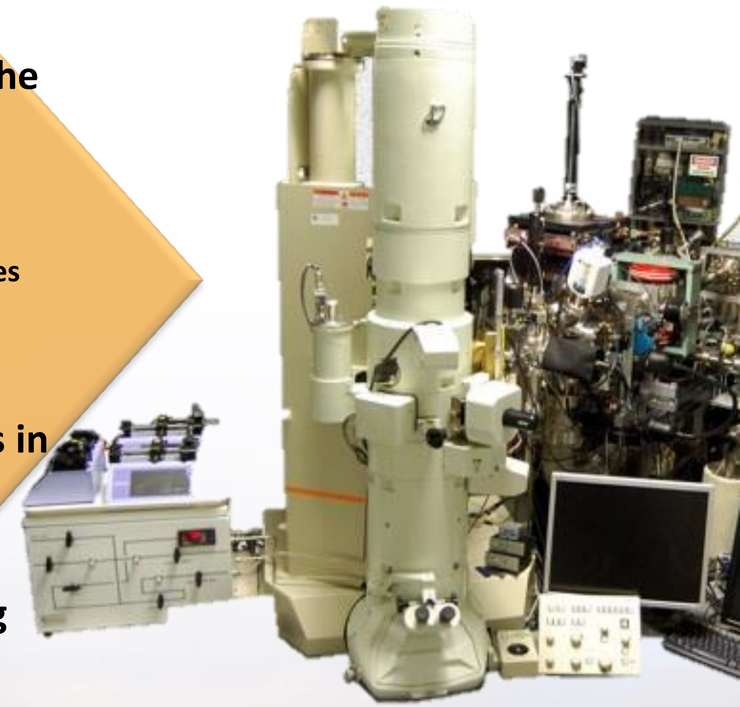


Sandia's I^3TEM is one of a few in the world

- *In situ* irradiation from H to Au
- *In situ* gas implantation
- Combinations of in-situ techniques

I^3TEM can provide fundamental understanding to key mechanisms in a variety of extreme conditions

The I^3TEM capability are still being expanded…



Collaborators:

- IBL: D.C. Bufford, D. Buller, C. Chisholm, B.G. Clark, B.L. Doyle, S. H. Pratt, & M.T. Marshall
- Sandia: B. Boyce, T.J. Boyle, P.J. Cappillino, J.A. Scott, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, E. Carnes, J. Brinker, D. Sasaki, J.A. Sharon, T. Nenoff, W.M. Mook
- 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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