Recent advancements in Sandia's *In situ* Ion Irradiation Transmission Electron Microscope

SAND2016-1656C

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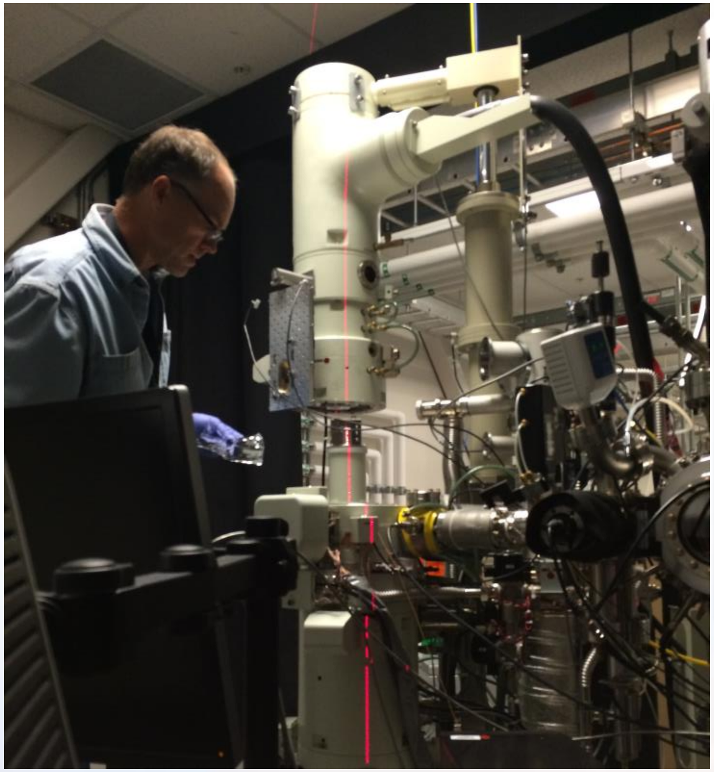
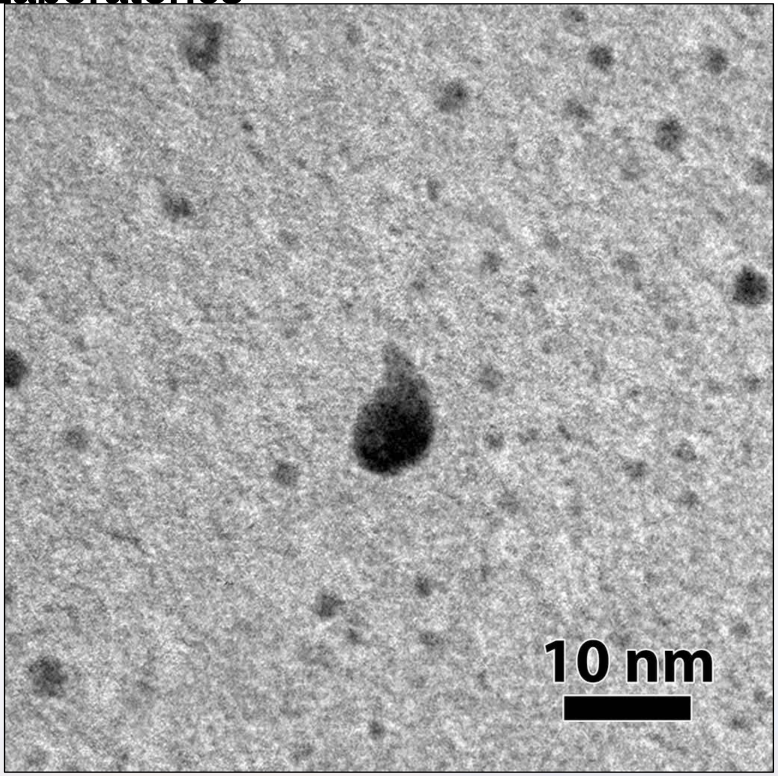
Ion Beam Lab at Sandia National Laboratories

March 17, 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 & 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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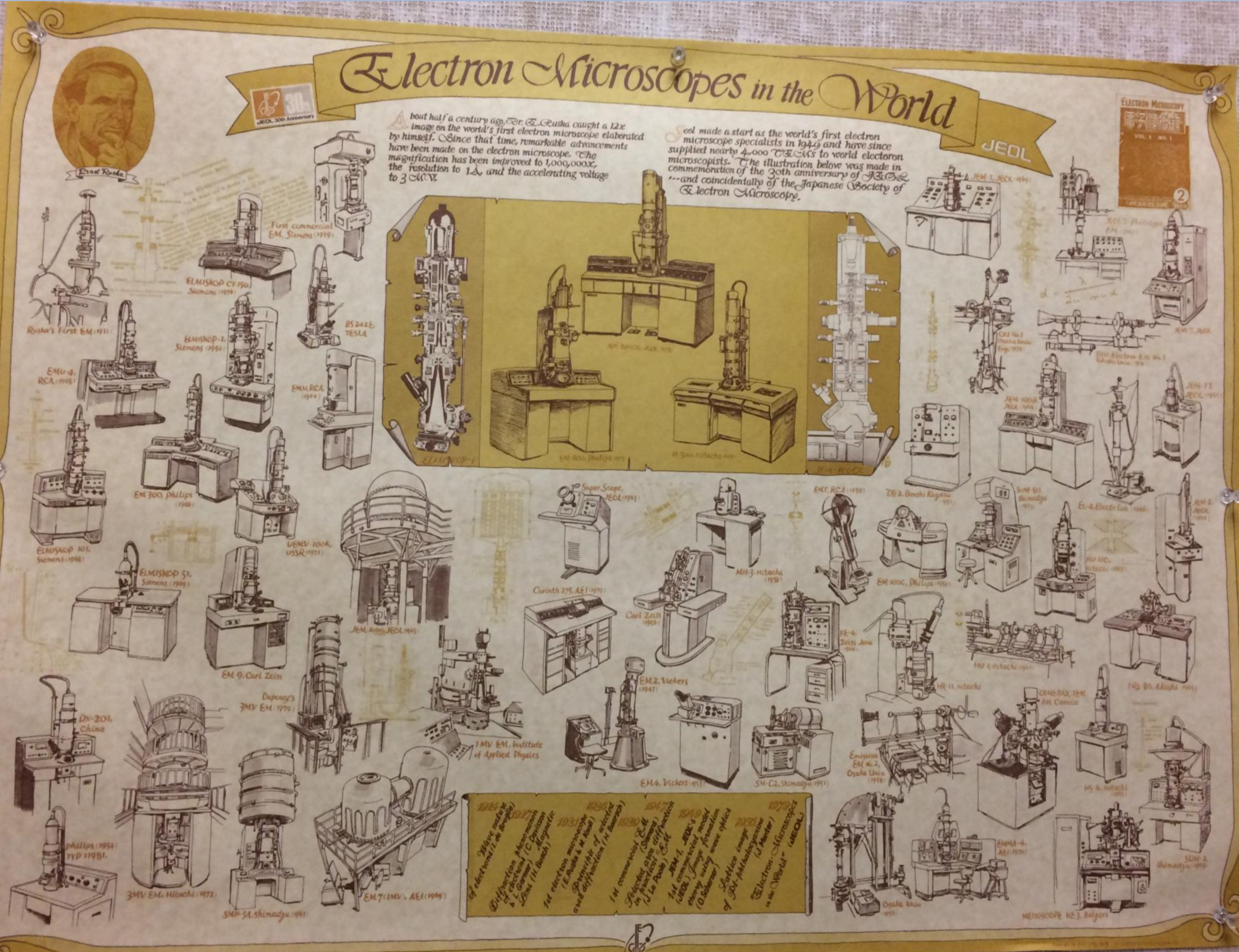
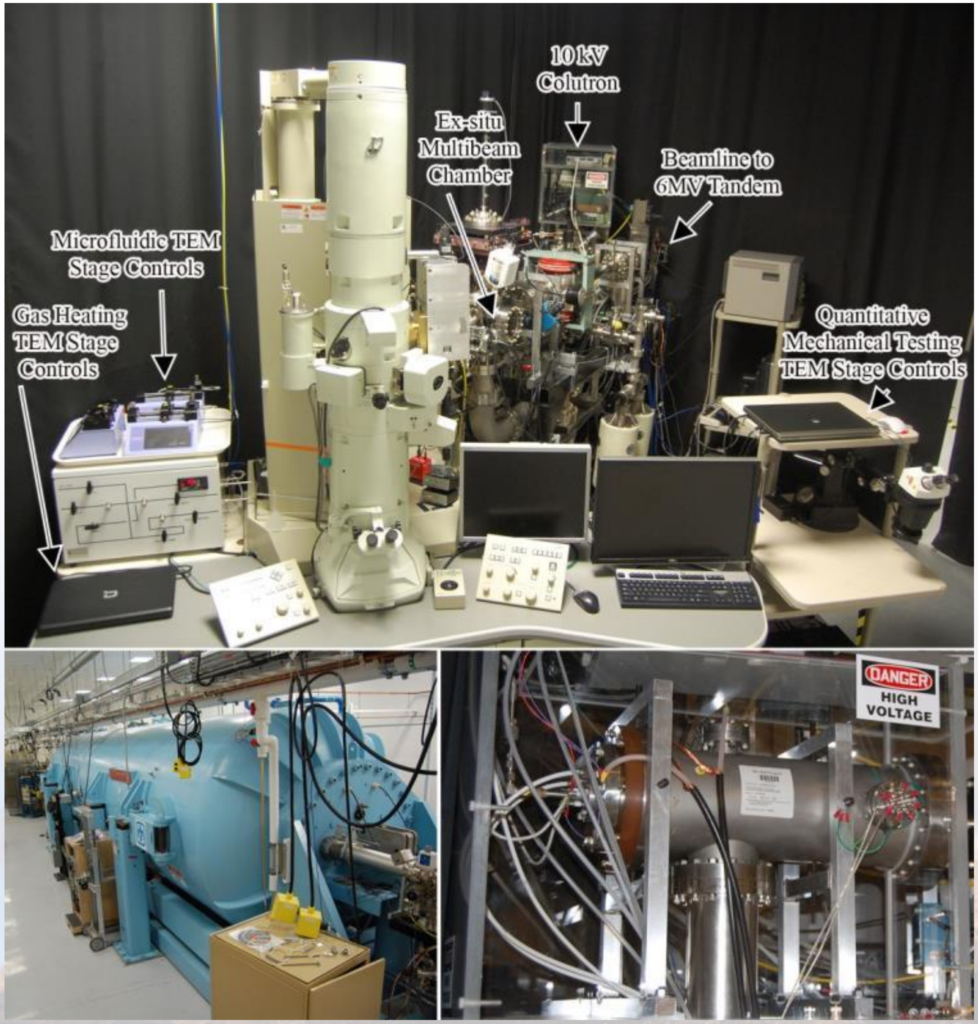


Electron Microscopes in the World
JEOL 30th Anniversary
JEOL
Ernst Ruska
About half a century ago, Dr. E. Ruska caught a 12x image on the world's first electron microscope elaborated by himself. Since that time, remarkable advancements have been made on the electron microscope. The magnification has been improved to 1,000,000x, the resolution to 1Å, and the accelerating voltage to 3 MV.
Jeol made a start as the world's first electron microscope specialists in 1949 and have since supplied nearly 4,000 TEMs to world electoron microscopists. The illustration below was made in commemoration of the 30th anniversary of JEOL ---and coincidentally of the Japanese Society of Electron Microscopy.
Ruska's First EM (1931)
First commercial EM, Siemens (1939)
ELMISKOP I, Siemens (1954)
EMU-4, RCA (1958)
EM 300, Philips (1968)
ELMISKOP 101, Siemens (1968)
ELMISKOP 51, Siemens (1964)
EM 9, Carl Zeiss
BS 242E, TESLA
UEMV 100K, USSR
JEM 1000, JEOL (1965)
Dupouy's 3MV EM (1970)
3MV EM, Hitachi (1972)
1MV EM, Institute of Applied Physics
EM 7 (1MV, AEI (1966)
DX-201, China
Super Scope, JEOL (1963)
Corinth 275, AEI (1970)
Carl Zeiss (1956)
MH-3, Hitachi (1958)
EM 2, Vickers (1947)
EM 4, Vickers
SM-C2, Shimadzu
FE-4, Zeiss Jena
EM 100C, Philips
HU-11, Hitachi
HS-6, Hitachi
JEM-1, JEOL
JEM-7, JEOL
JEM-100B
EPMA-4, AEI
METIOSCOPE KS3, Balzers
Emission EM No. 2, Osaka Univ.
Osaka Univ.
Electron Microscopy VOL. 1 NO. 1

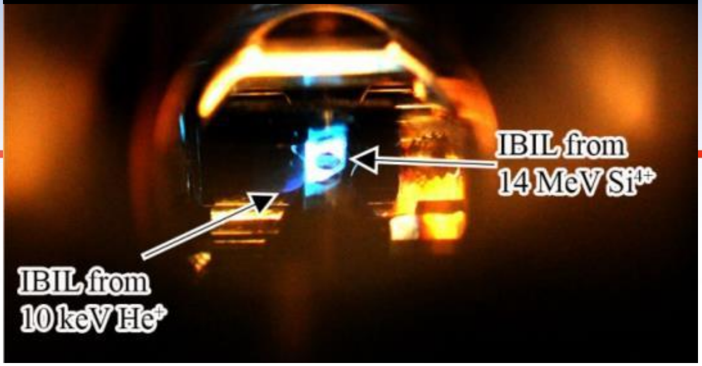
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem

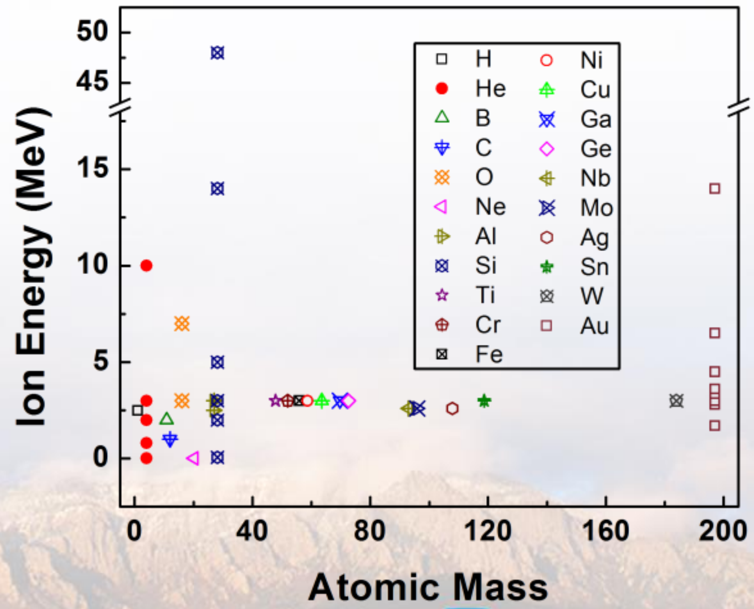


IBIL from a quartz stage inside the TEM



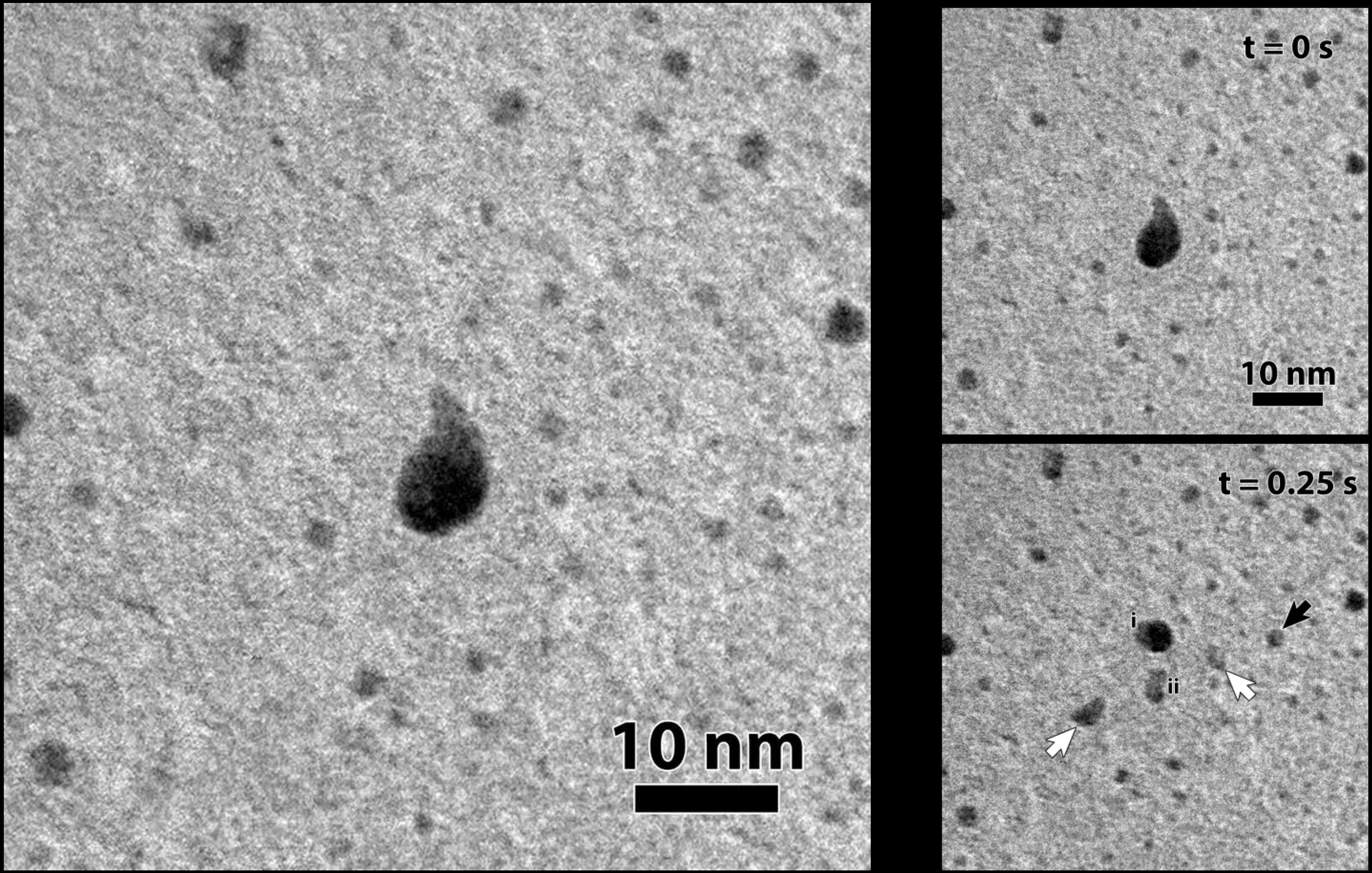
Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution

Ion species & energy introduced into the TEM



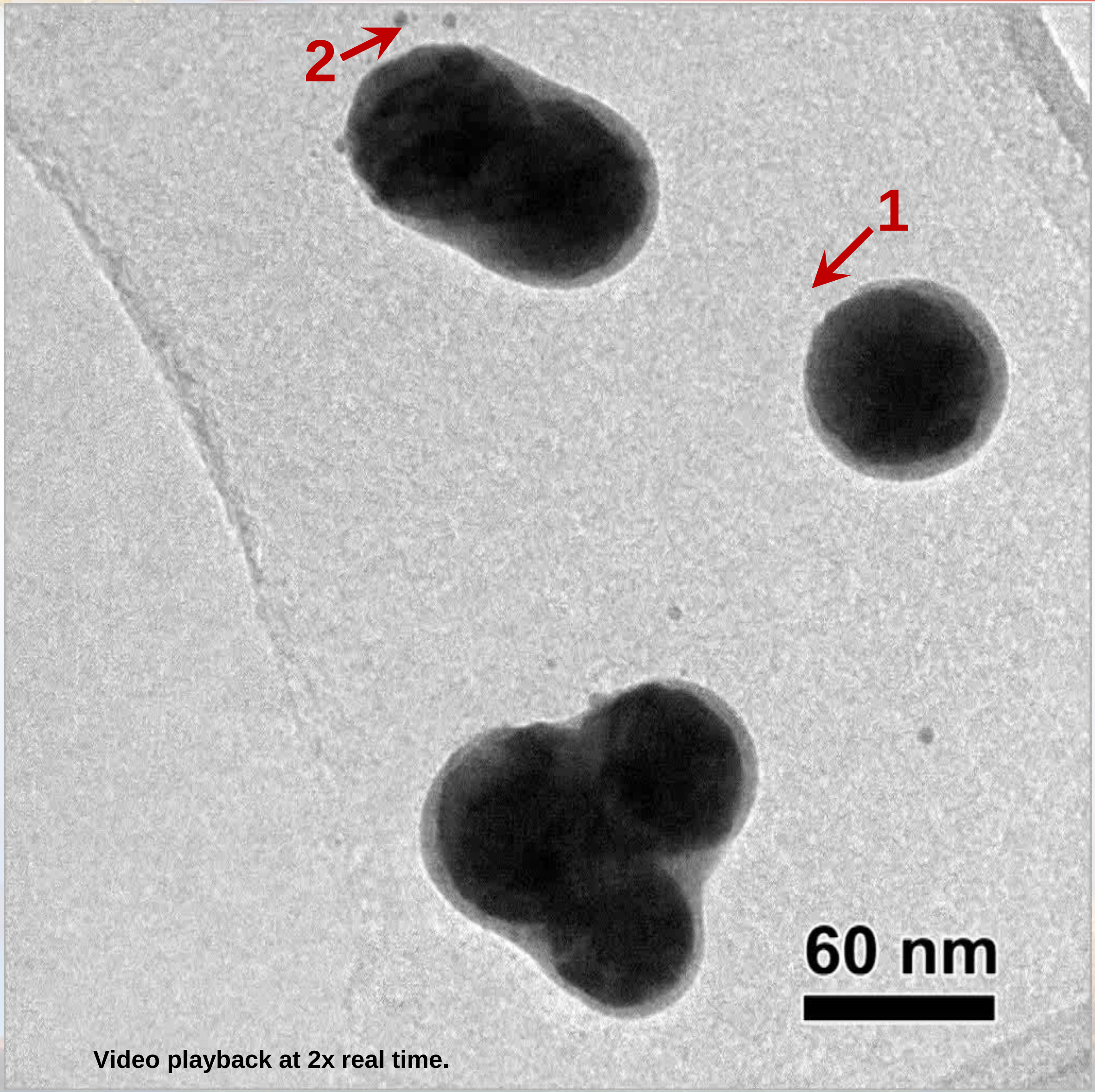
Single Ion Strikes:
46 keV Au^{1-} ions into 5 nm Au nanoparticles

Collaborator: D.C. Bufford



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

Collaborator: D.C. Bufford

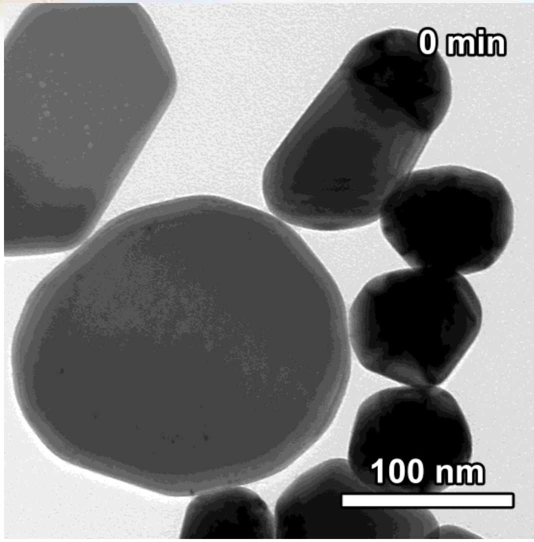


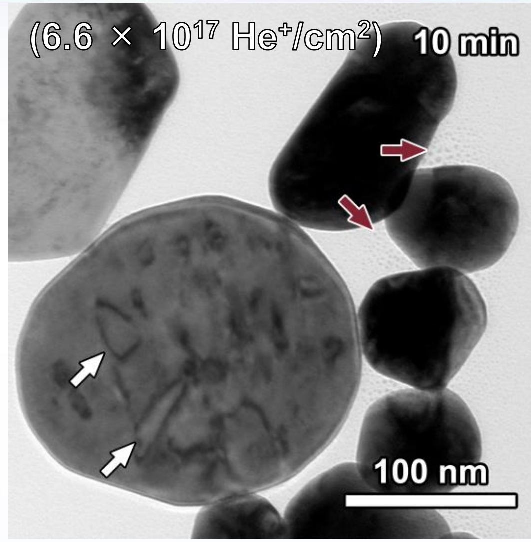
- **2.8 MeV Au^{4+} ions into 60 nm diameter Au nanoparticles**
- **100 kx magnification**
- **Nanoscale filaments created by individual ions**

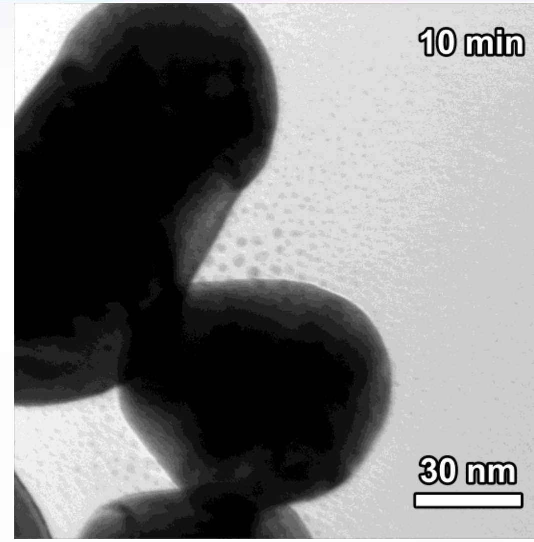
The permanent and transient structures resulting from single ion strikes can be directly observed

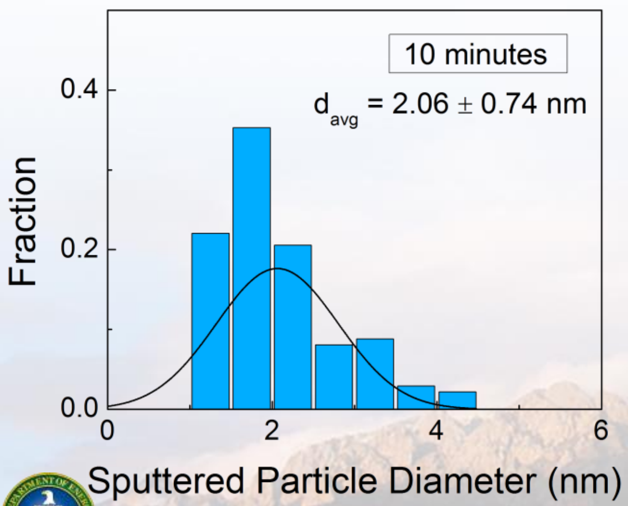
Formation of Dislocation Loops & Sputtered Particles due to He implantation

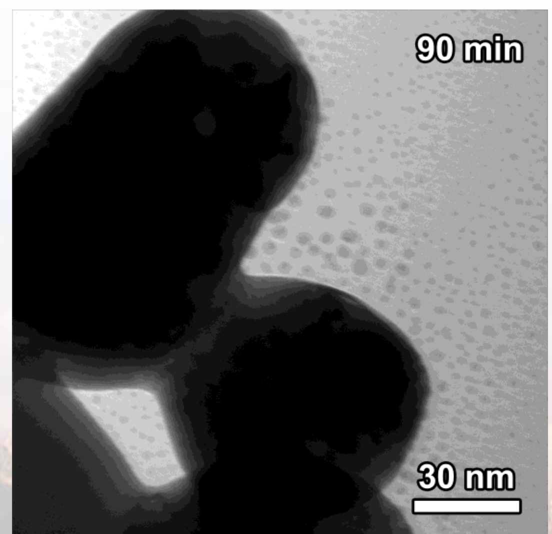
Collaborators: D.C. Bufford, S.H. Pratt & T.J. Boyle

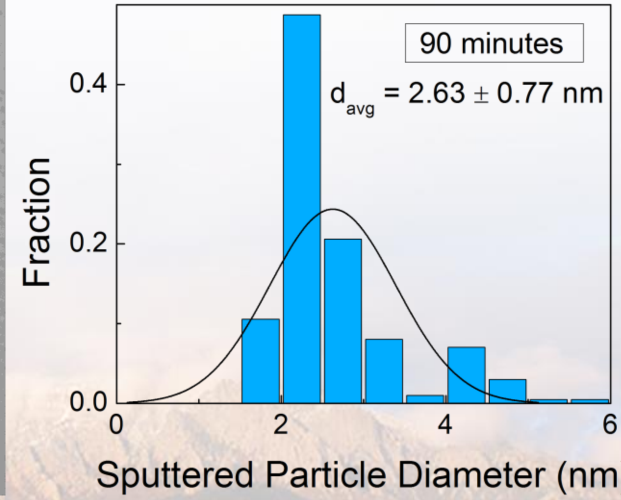












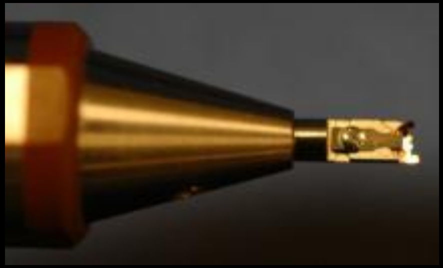
Electron Tomography Provides 3D Insight

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

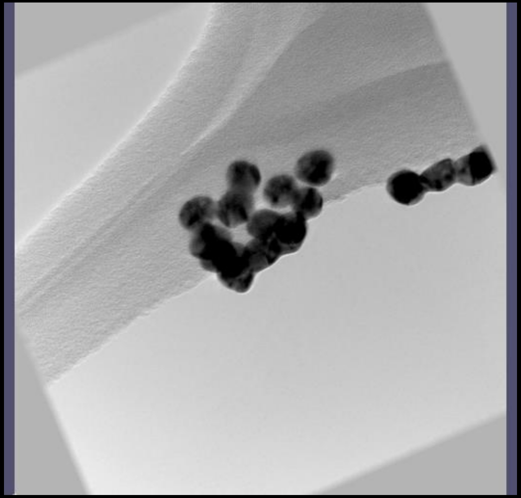
In situ Ion Irradiation TEM (I[3]TEM)

+

Hummingbird tomography stage



Aligned Au NP tilt series - unirradiated



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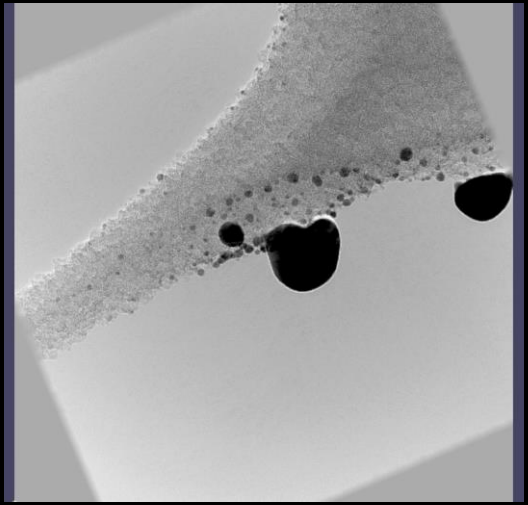
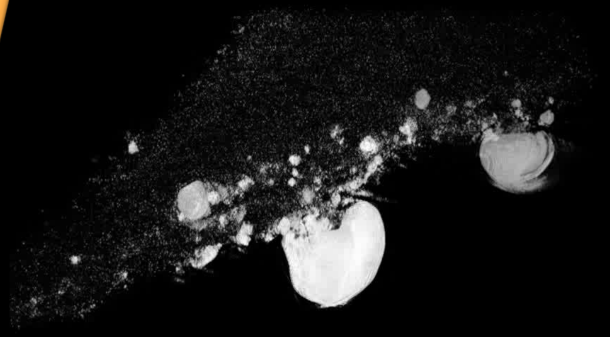


Unirradiated Au NP model

Irradiated Au NP model

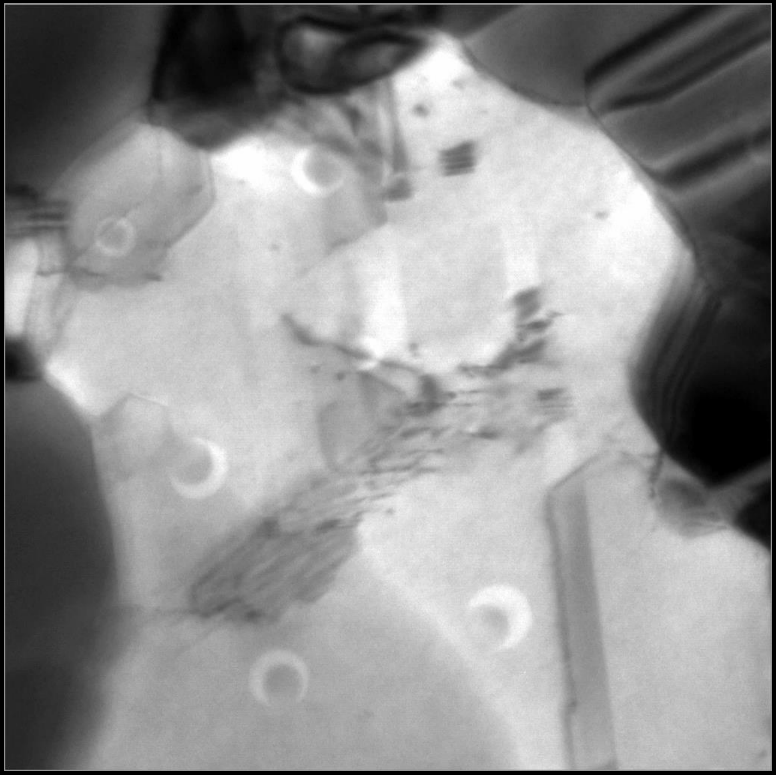


The application of advanced microscopy techniques to extreme environments provides exciting new research directions

Dose Rate Effects

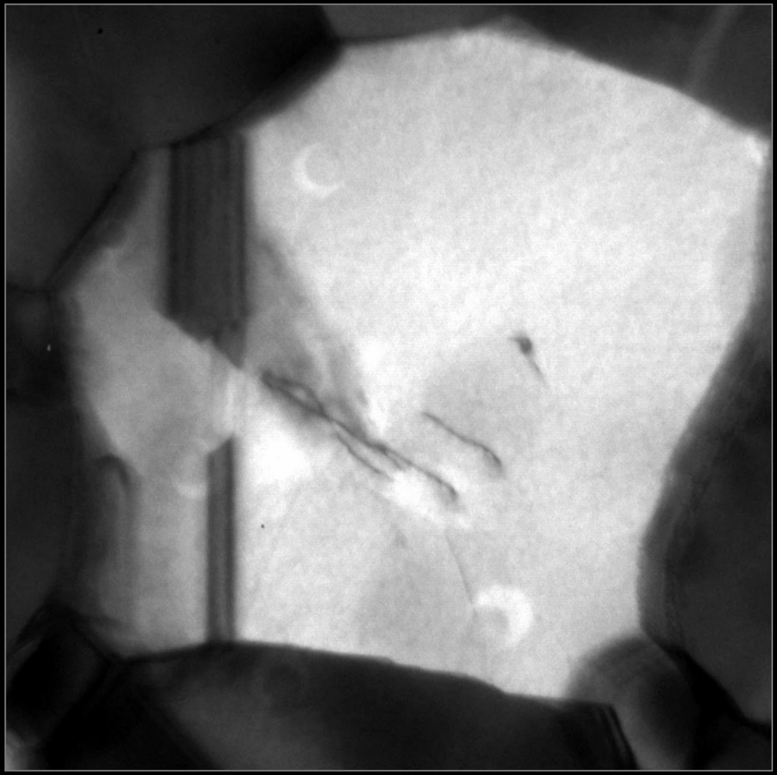
Collaborators: C. Chisholm , P. Hosemann, & A. Minor

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



VS

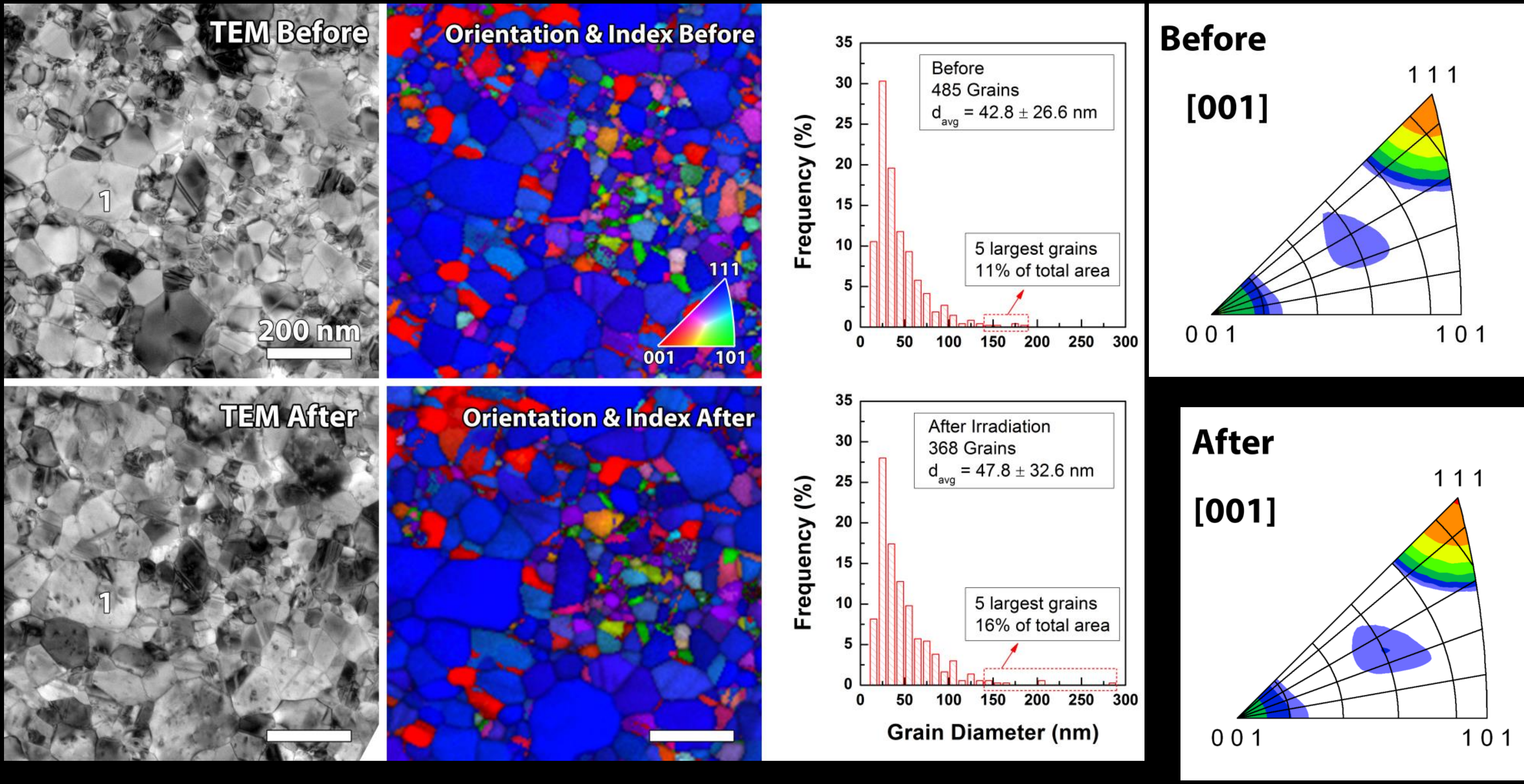
6.7 x 10^7 ions/cm^2/s



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

Quantifying Stability of Nanocrystalline Au during 10 MeV Si Ion Irradiation

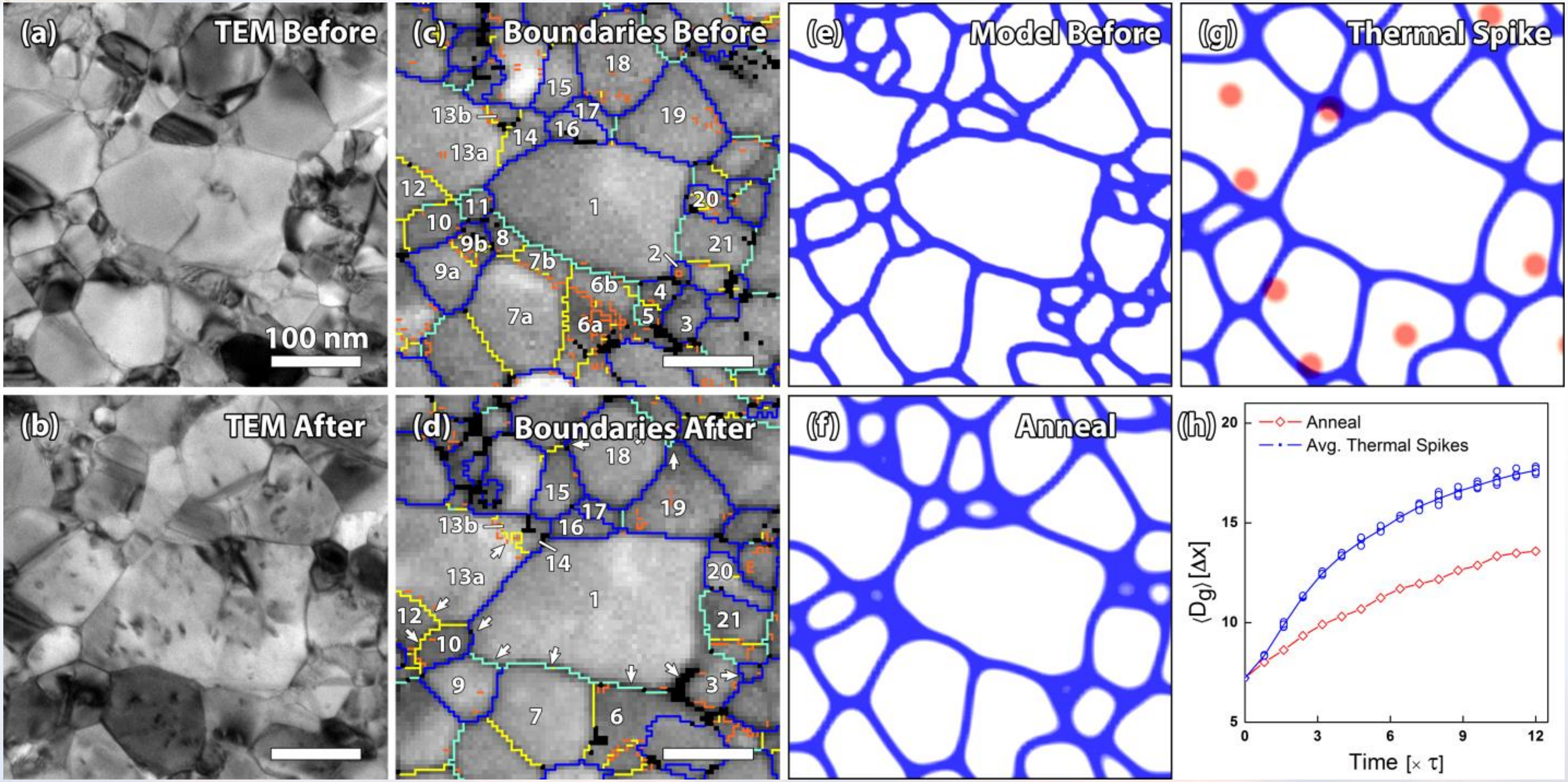
Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles



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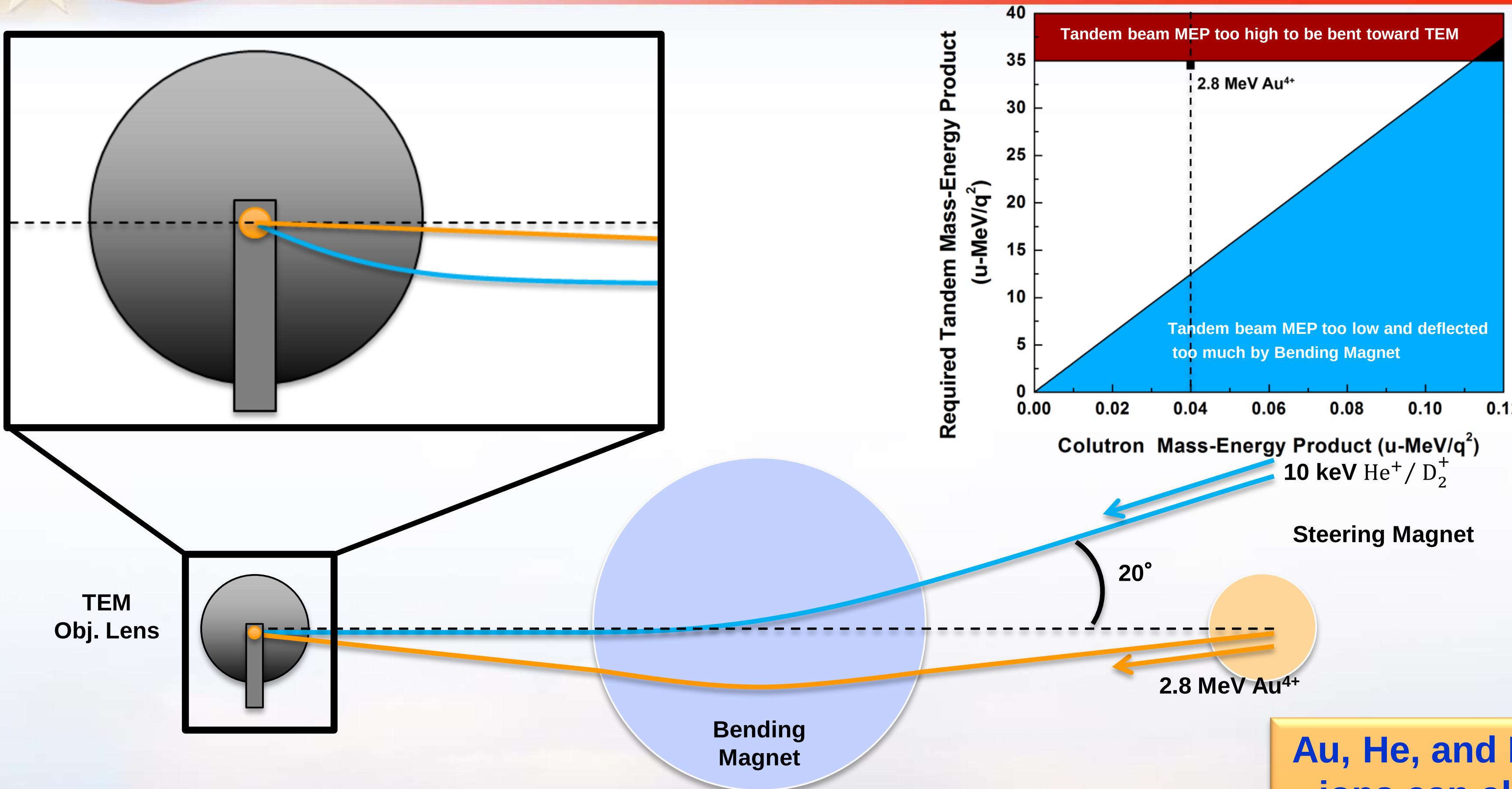
Modeling Beam Mixing and Deflection Necessary to Develop a *In situ* Triple Beam Facility

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



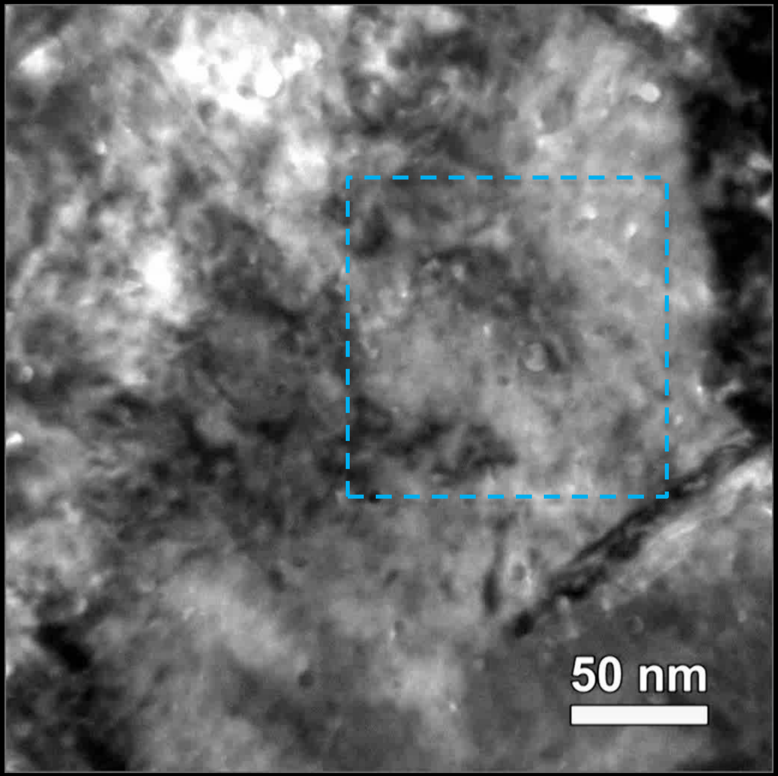
- Must compensate for deflection of Tandem beam by bending magnet Colutron beams deflected by the TEM objective lens
- Insignificant deflection of Tandem beams
- With 10 keV He/D_2 we can use Tandem beams ≳13 MeV/q^2

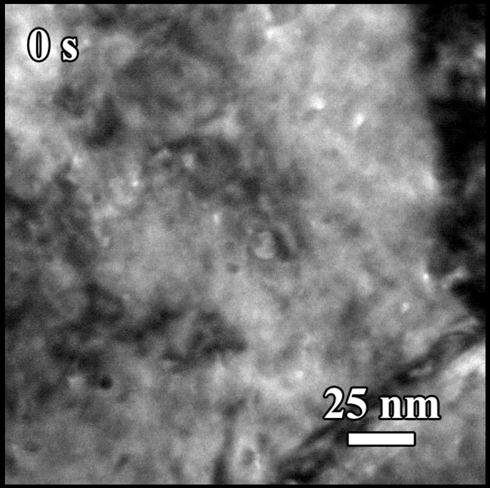
Au, He, and D_2 ions can all reach the sample concurrently

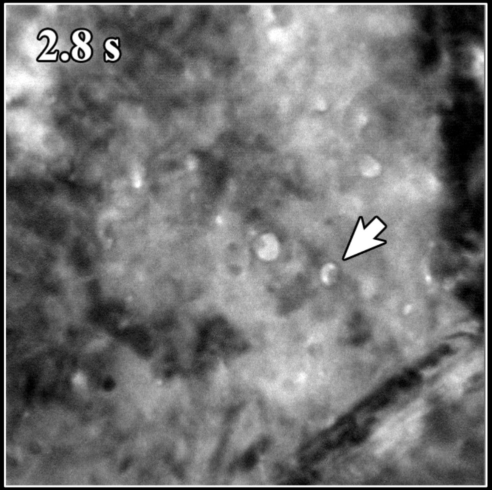
Simultaneous *In situ* TEM Triple Beam: 2.8 MeV Au^{4+} + 10 keV He^+/D_2^+

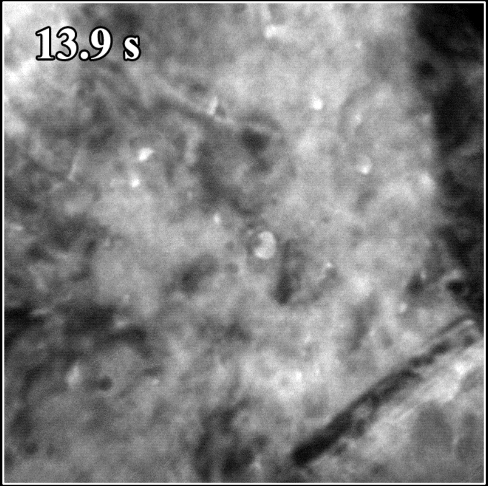
Collaborator: D.C. Bufford

Video playback speed x1.5.









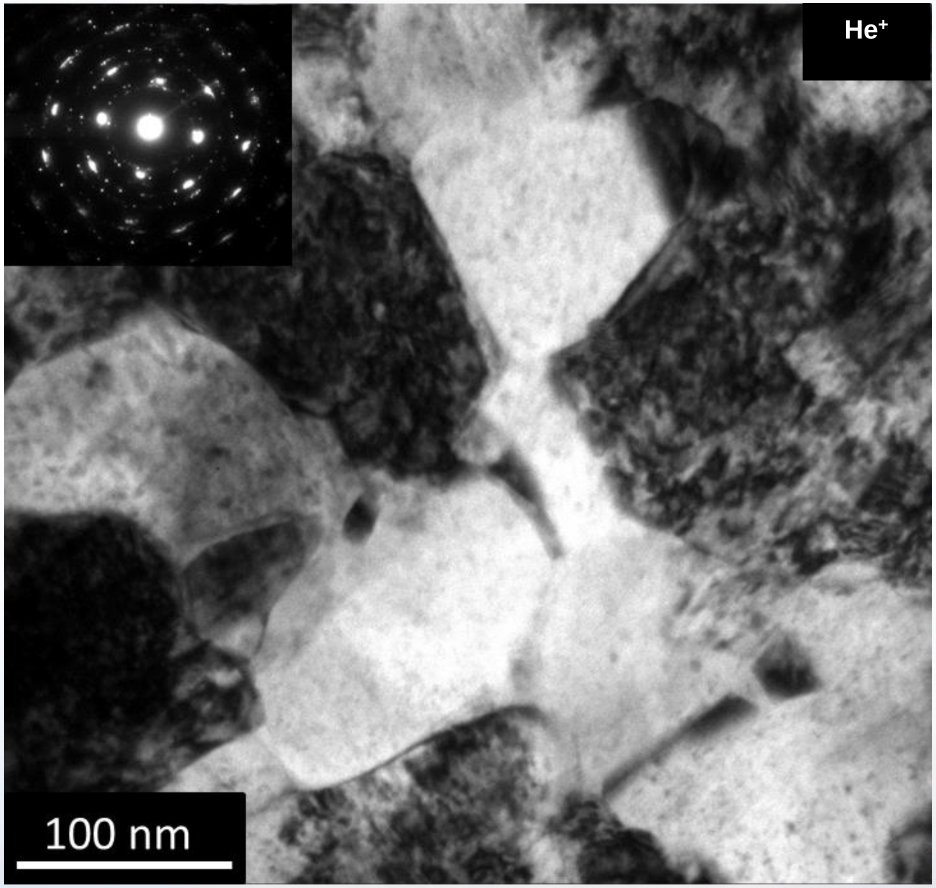
In-situ triple beam He, D_2, and Au beam irradiation has been demonstrated on Sandia's I^3TEM!

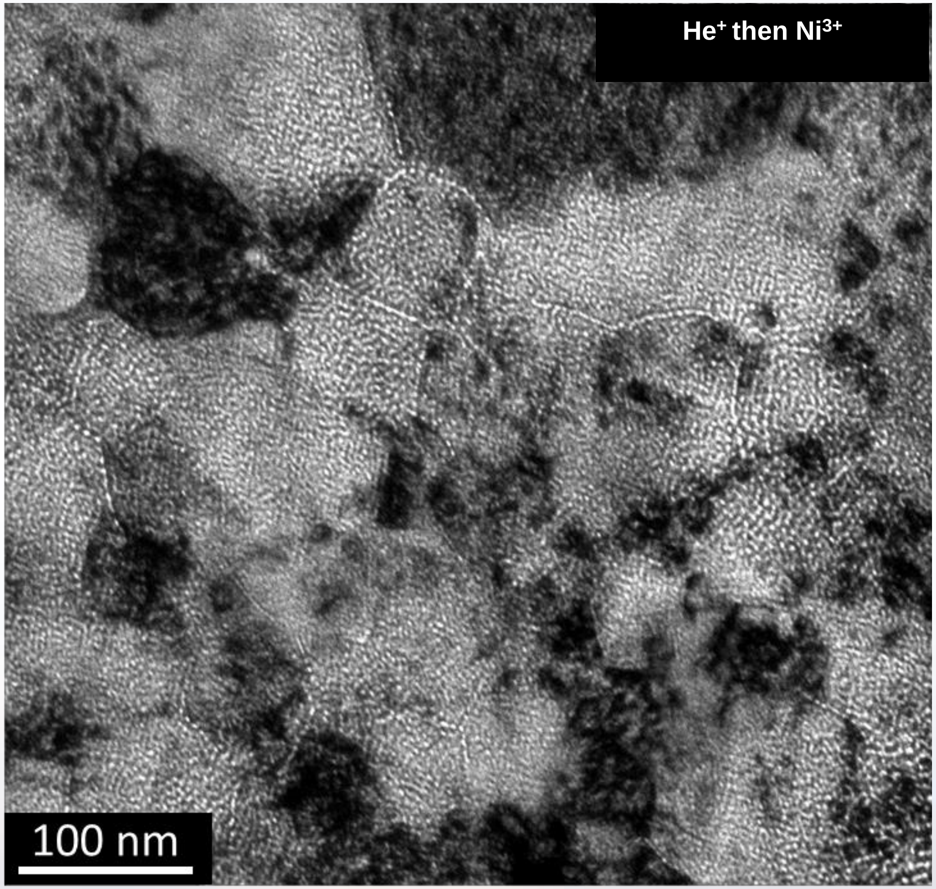
Intensive work is still needed to understand the defect structure evolution that has been observed.

- **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**

10 keV He^+ Implantation followed by 3 MeV Ni^{3+} Irradiation

Collaborator: B. Muntifering & J. Qu





10^{17} He^+/cm^2

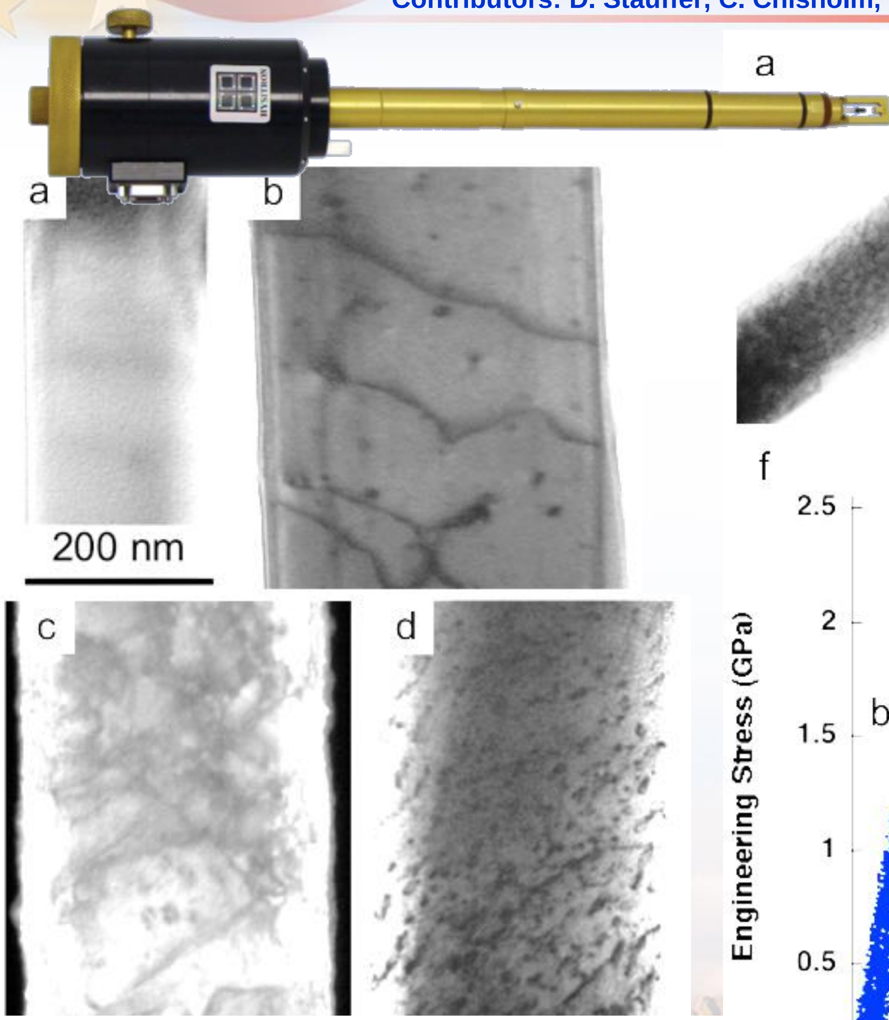
Visible damage to both the sample and the source

0.7 dpa Ni^{3+} irradiation

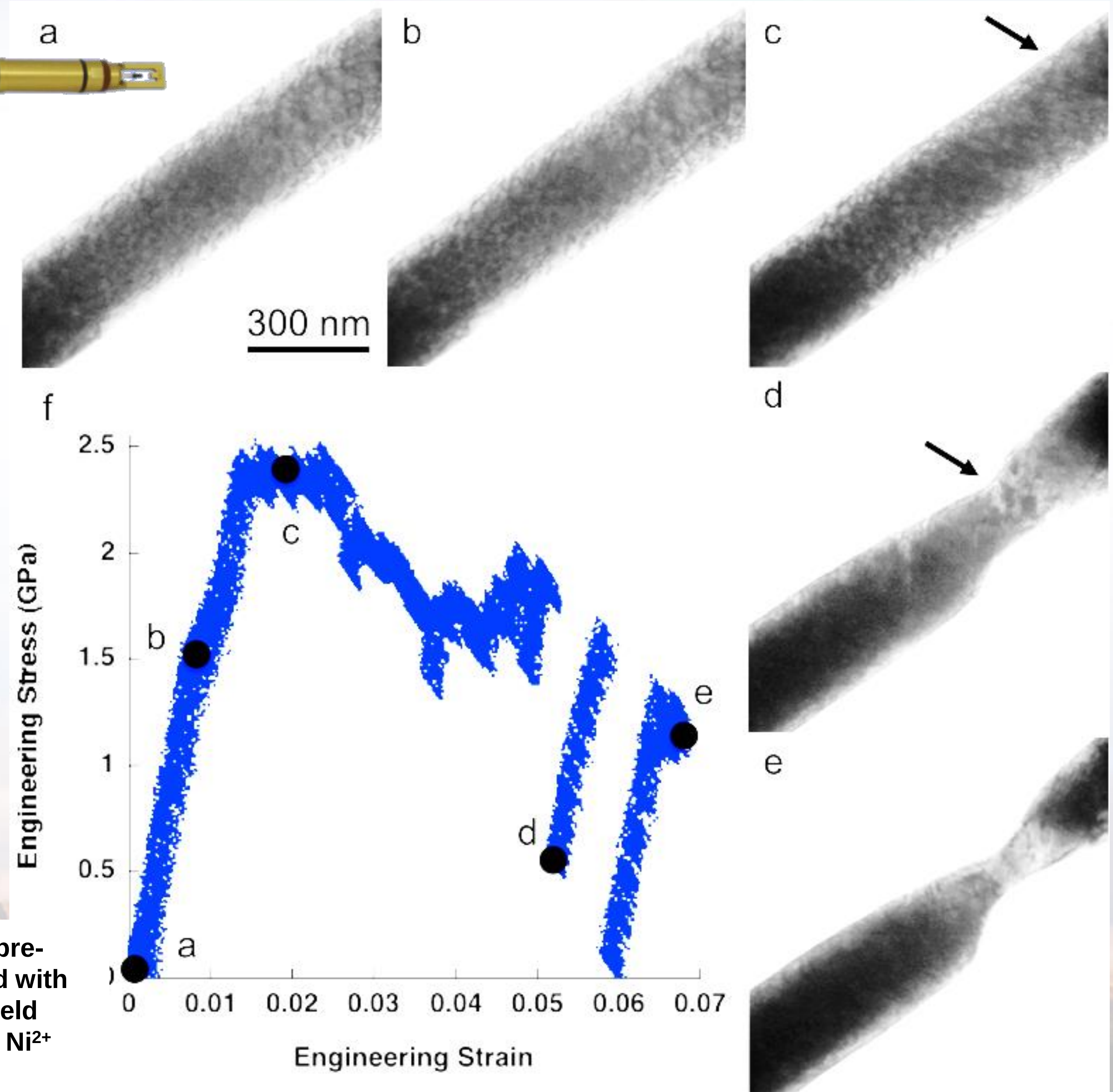
High concentration of cavities along grain boundaries

In situ TEM Quantitative Mechanical Testing

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

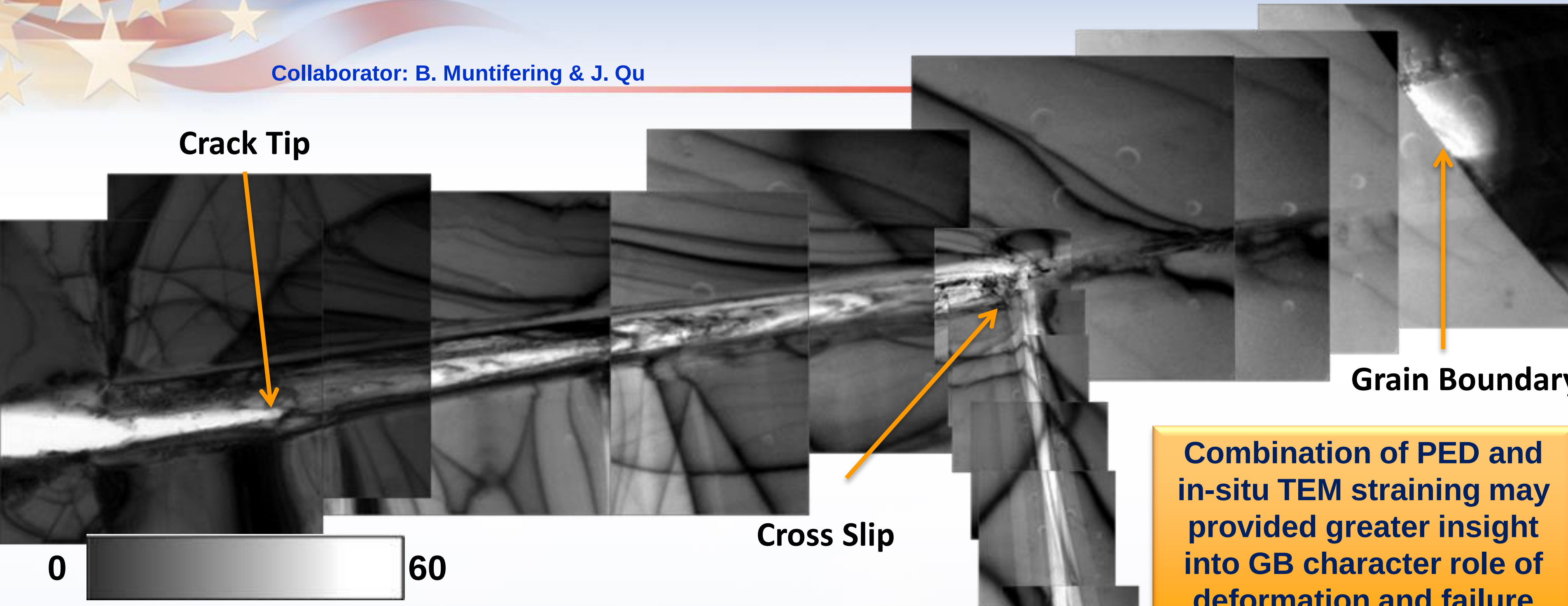


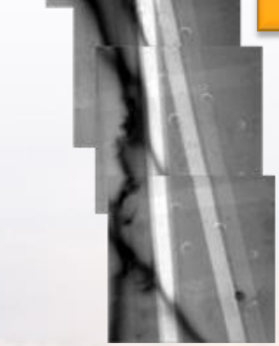
TEM micrographs of Mo-alloy nanofibers. (a) 0% pre-strained, no observable defects. (b) 9% pre-strained with mobile dislocations and sessile loops. (c) Dark-field micrograph of 15% pre-strained fiber. (d) He^{1+} and Ni^{2+} irradiated, 9% pre-strained fiber.



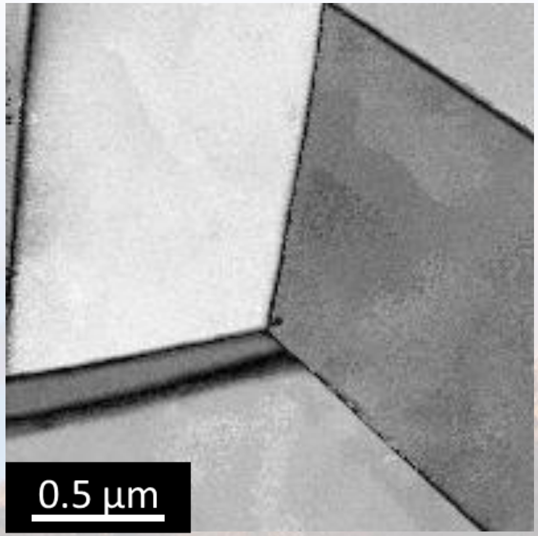
Future Direction: PED and *In situ* TEM Deformation

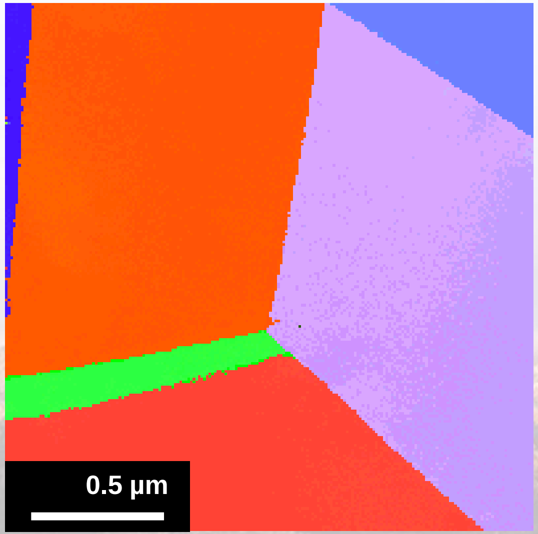
Collaborator: B. Muntifering & J. Qu

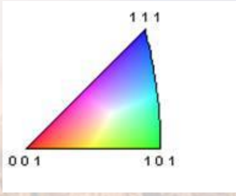




Combination of PED and in-situ TEM straining may provided greater insight into GB character role of deformation and failure

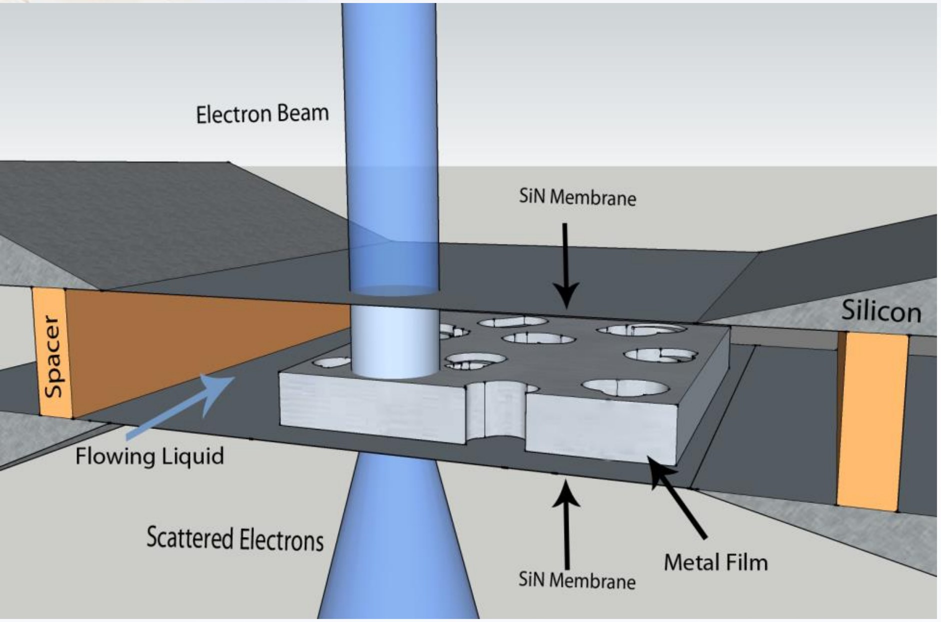






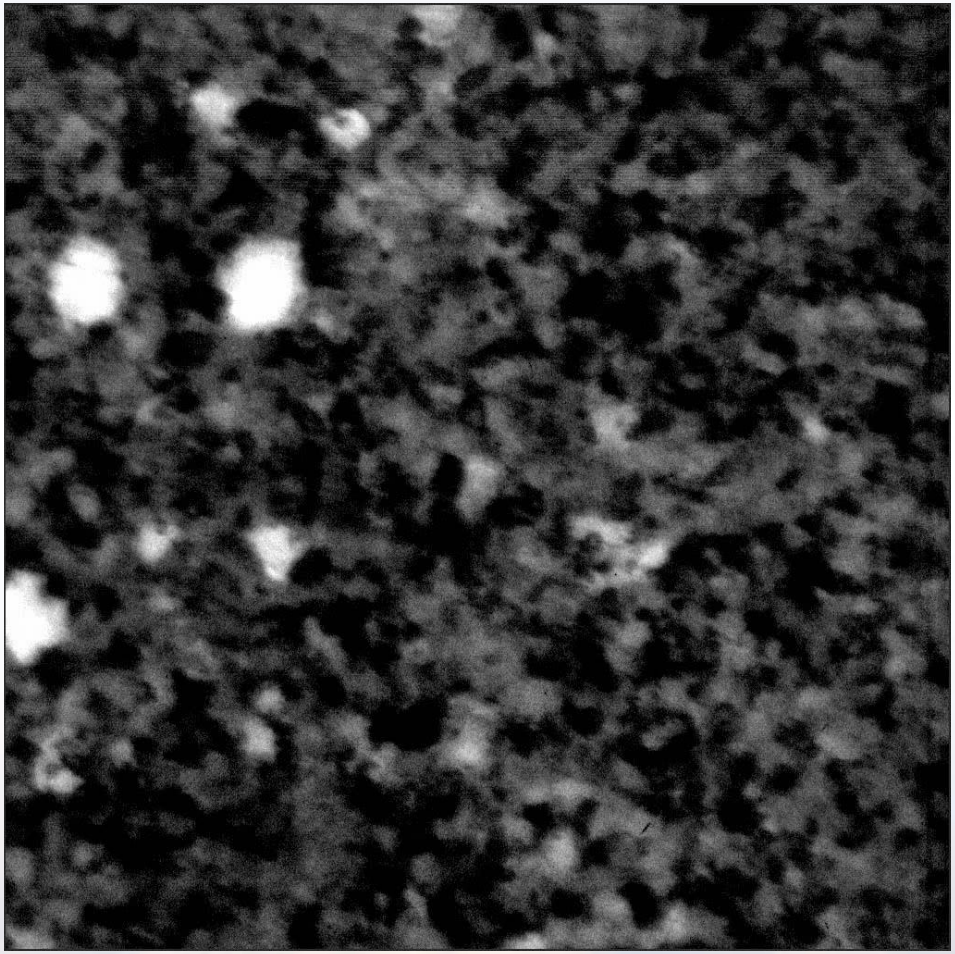
Future Direction: *In situ* TEM Corrosion Direction

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



Microfluidic Stage

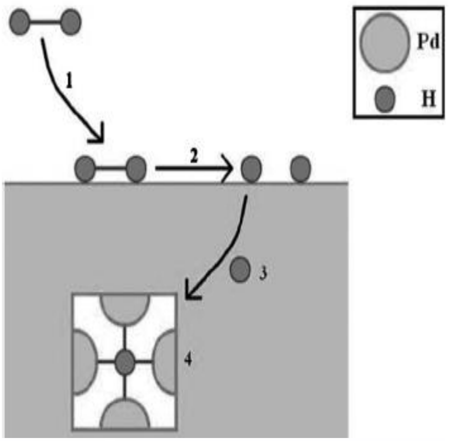
- Mixing of two or more channels
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Films can be directly deposited on the electron transparent SiN membrane



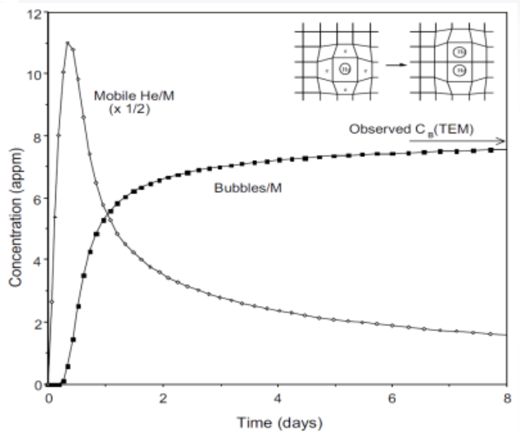
Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.

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.



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

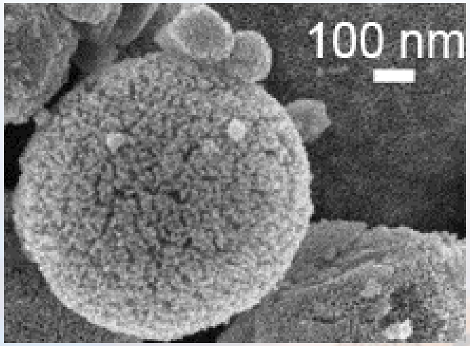


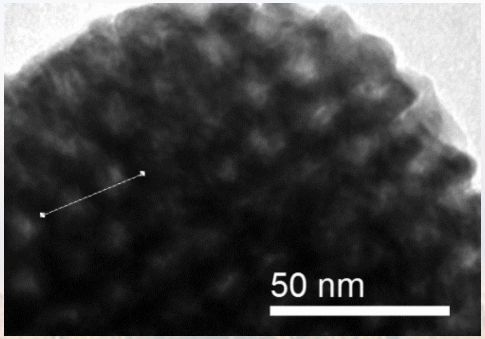
Cowgill, D., Fusion Sci. & Tech., 28 (2005) p. 539

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

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

Harmful effects may be mitigated in nanoporous Pd

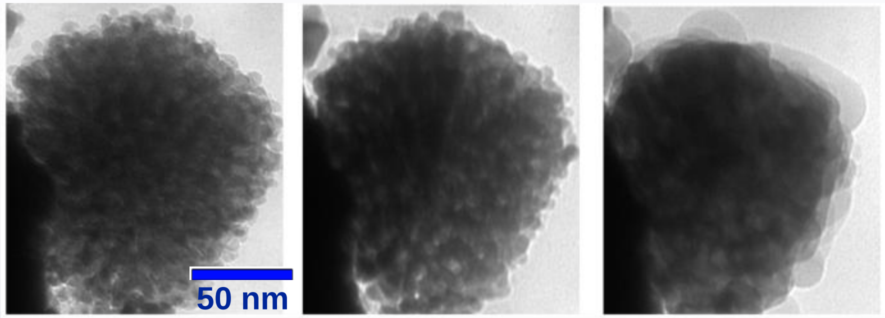




Vapor-Phase Heating TEM Stage

- 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.

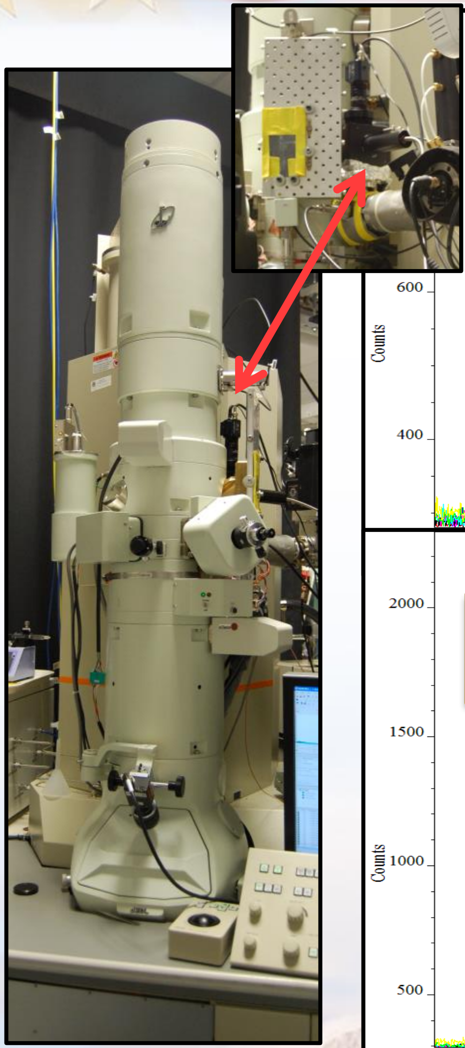


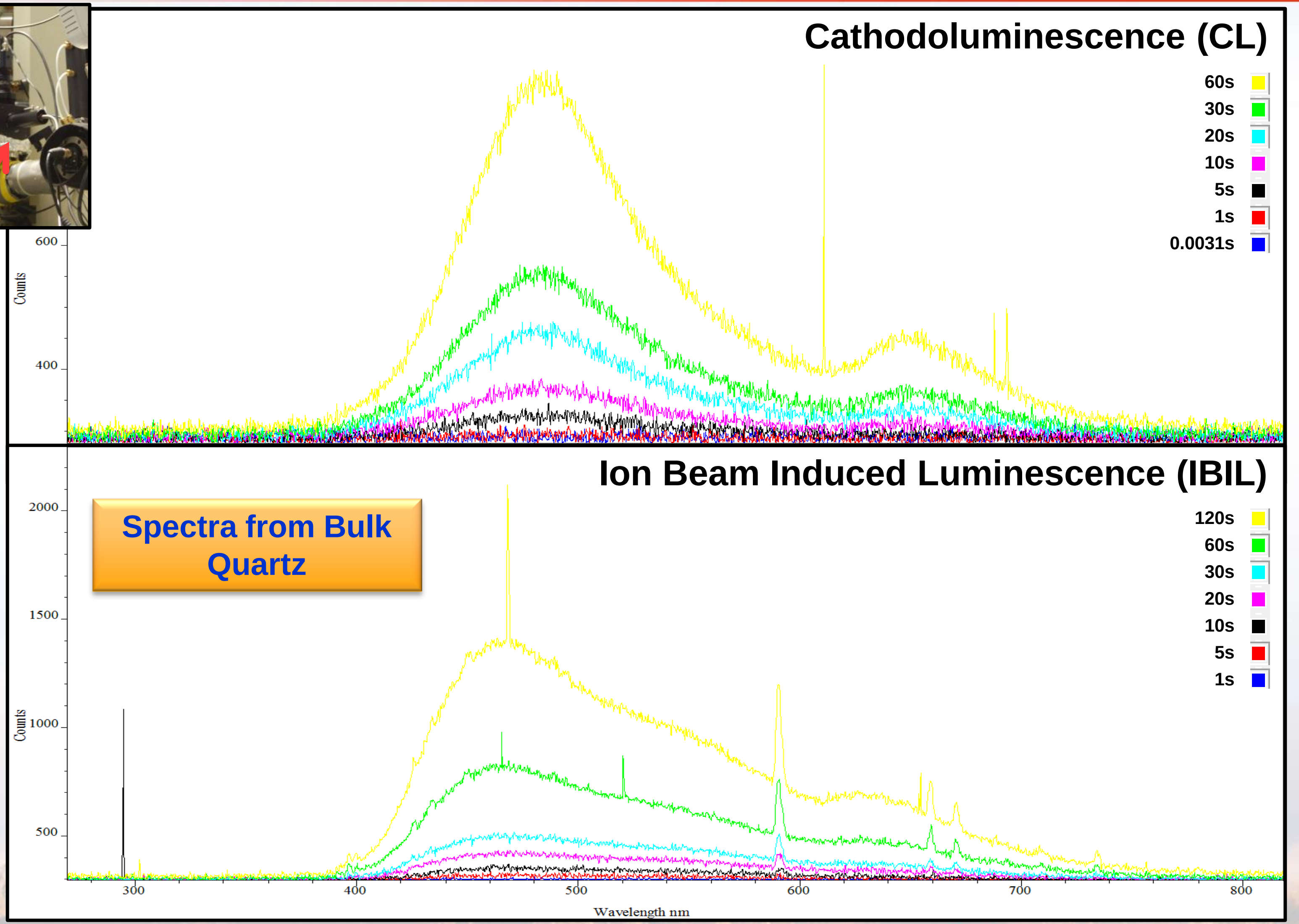
125° C 200° C 300° C

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

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

Collaborator: J. Gutierrez-Kolar

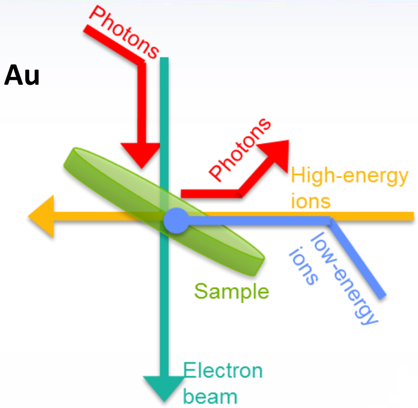




Significant optimization is still needed; potential is promising

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˚**

<u>Collaborators:</u>

- **IBL: D.C. Bufford, D. Buller, C. Chisholm, B.G. Clark, J. Villone, B.L. Doyle, S. H. Pratt, M. Steckbeck & M.T. Marshall**
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- **External: A. Leff, M. Taheri, A. Minor, L.R. Parent, I. Arslan, H. Bei, E.P. George, P. Hosemann,**

. Gross, J. Kacher, & I.M. Robertson

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