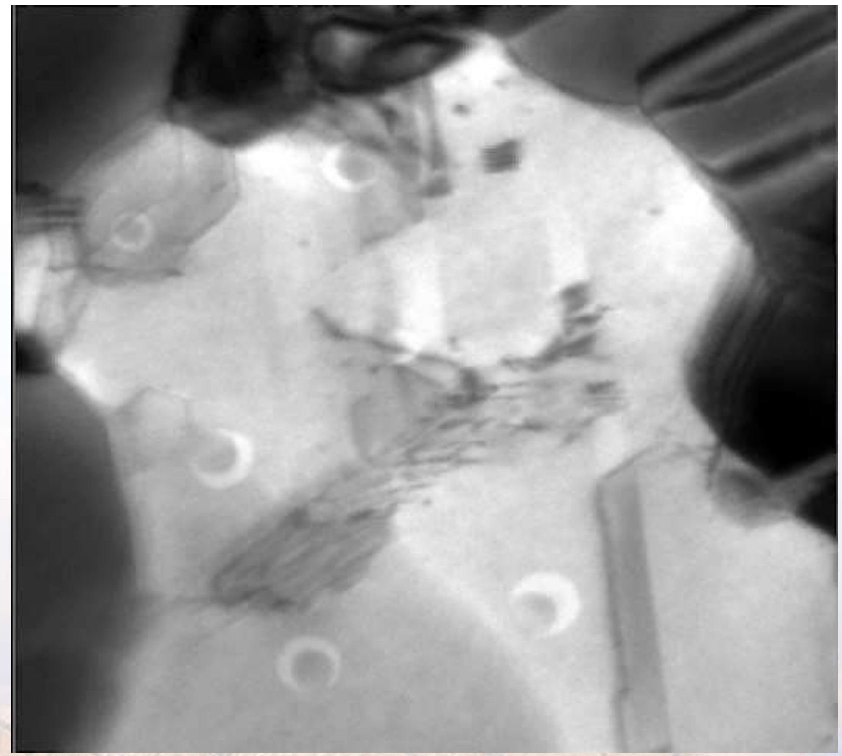
Recent Developments in Sandia's *In situ* Ion Irradiation TEM

SAND2013-6570C

K. Hattar, S.H. Pratt, C. Chisholm, O.J. El-Atwani, B.A. Hernandez-Sanchez, H. Bei, E.P. George, P. Hosemann, & A.M. Minor

Sandia's Ion Beam Lab

August 7, 2013



Sandia's *In situ* Ion Irradiation TEM is operational, but still in development

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

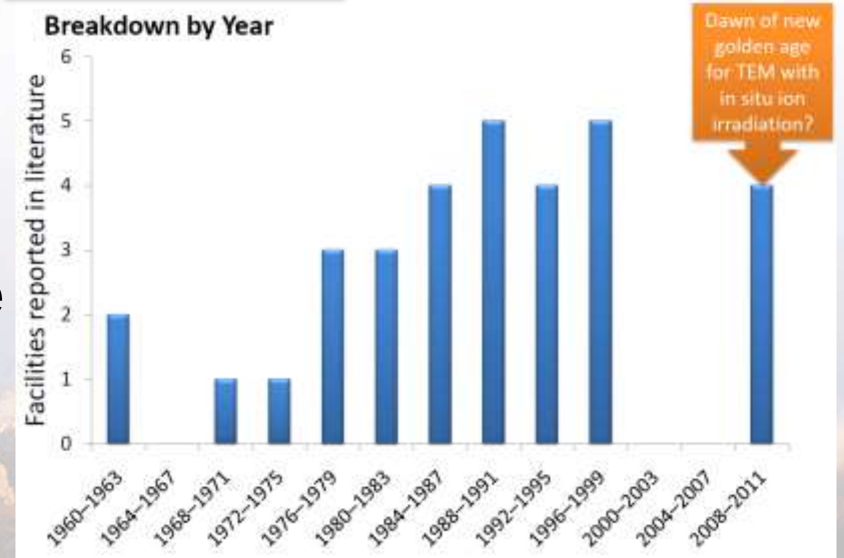
1968
First TEM beamline combination by Thackery, Nelson, and Sansom at AERE Harwell, UK

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"

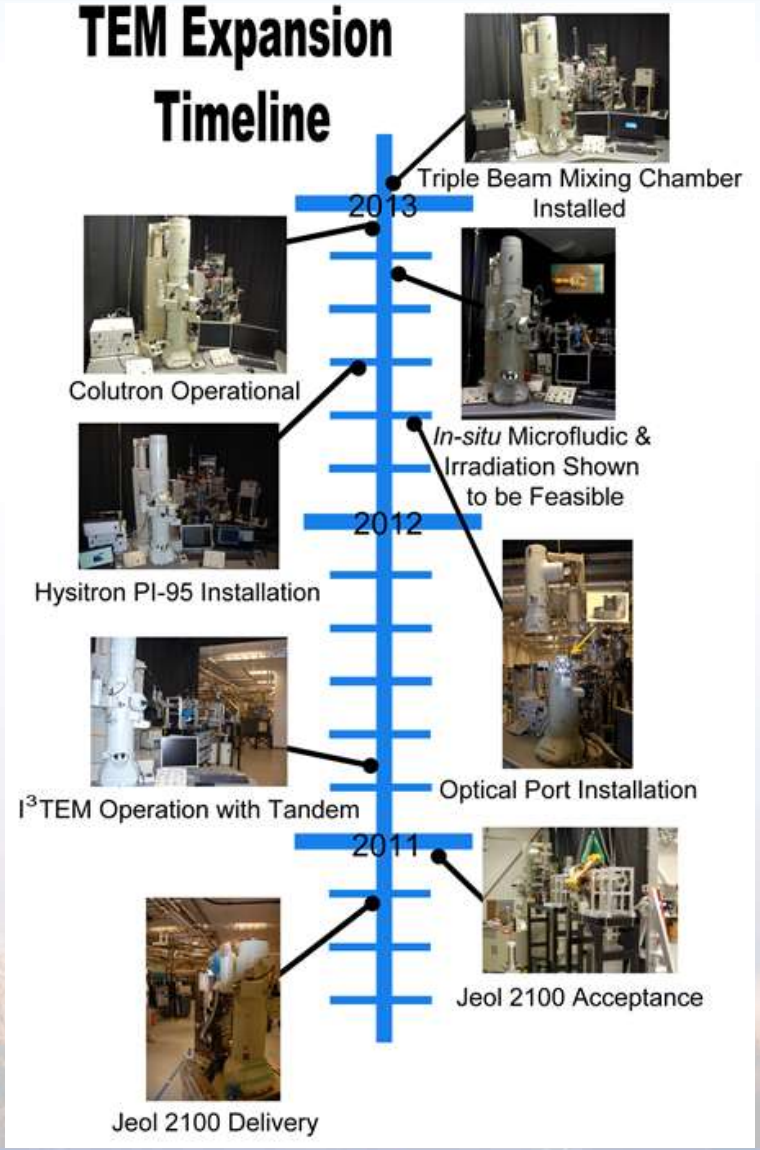
D.W. Pashley and A.E.B. Presland Phil Mag. 6(68) 1961 p. 1003

The Development of an *In situ* Ion Irradiation TEM in Sandia's New Ion Beam Lab

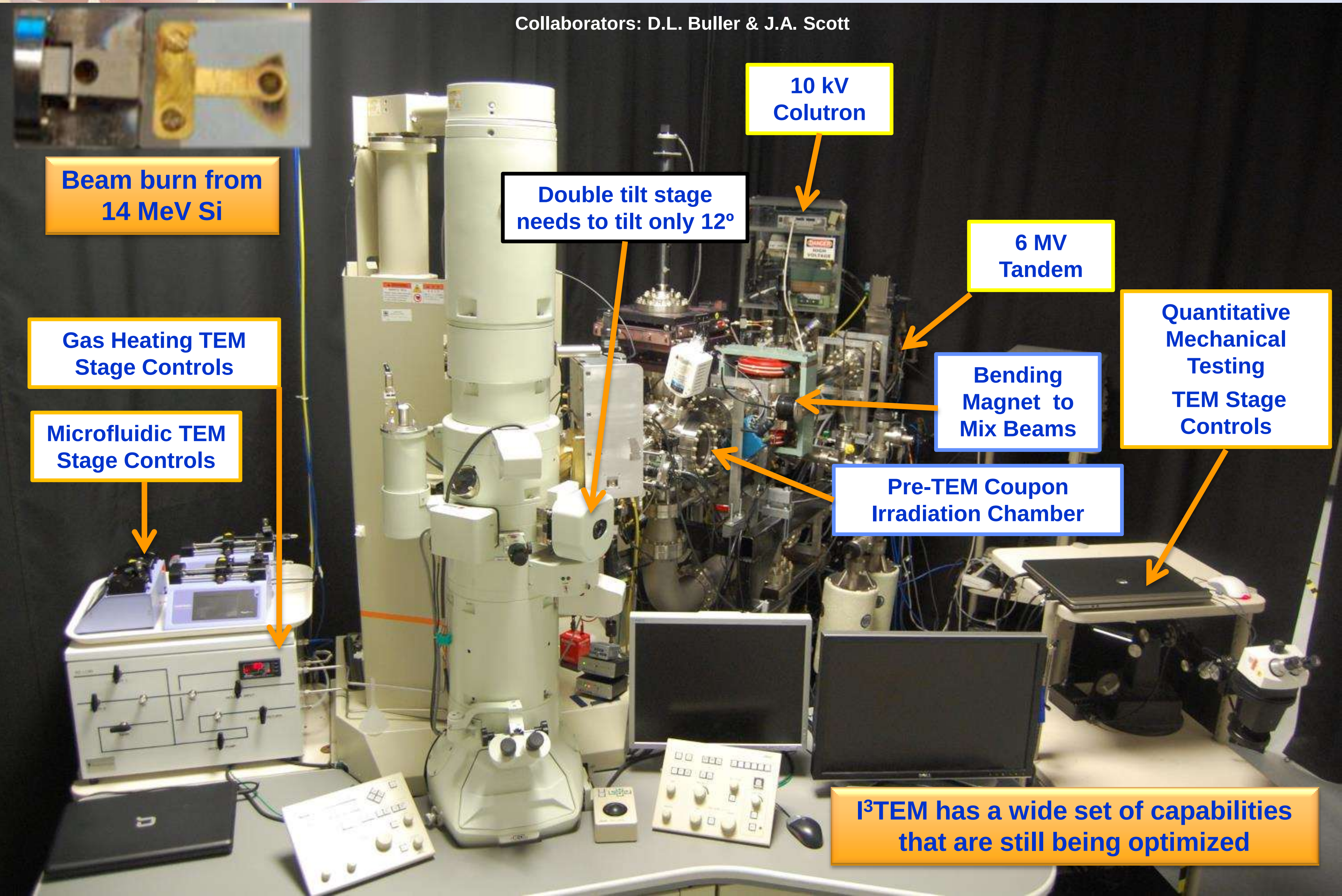
A special 72 wheeled vehicle with independent steering for each pair of wheels was used to move the Tandem accelerator

<u>New Facility</u>
laboratory space
1850 m^2
office space
650 m^2
<u>Old Facility:</u>
1300 m^2 total

Building: $20M
<u>Equipment: $11M</u>
Total: $40M

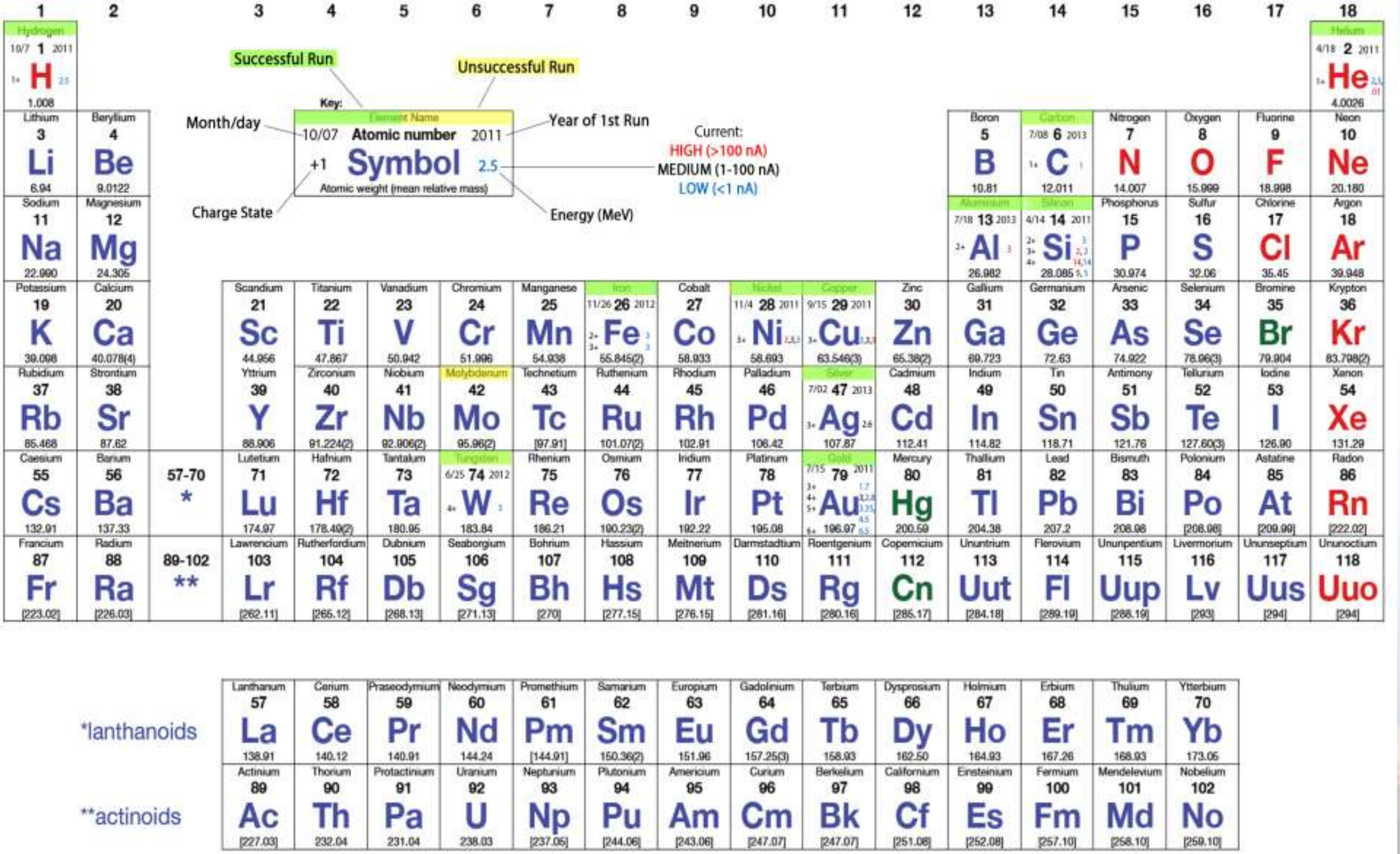


Current Status of the *In situ* TEM Beamline



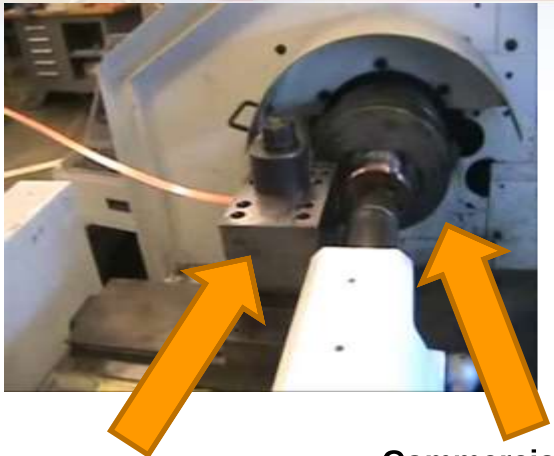
Ion Species Attempted to Date

Collaborators: M. Steckbeck



What Insight into Structural Stability is Gained from I³TEM Experiments?

Collaborators: O. El-Atwani, J. P. Allain, D. Buller, & J.A. Scott

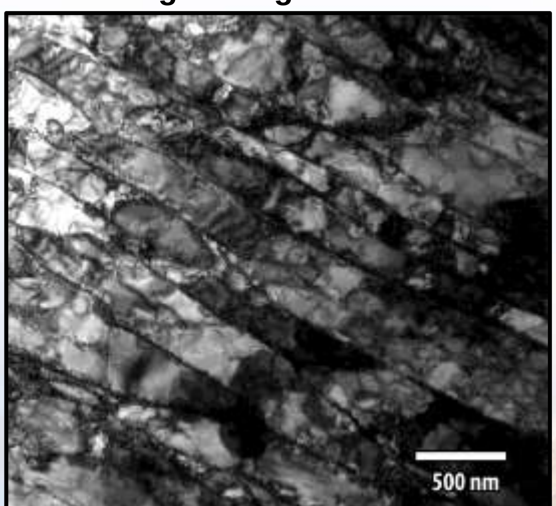


Extrusion machining tooling

Commercially available lathe

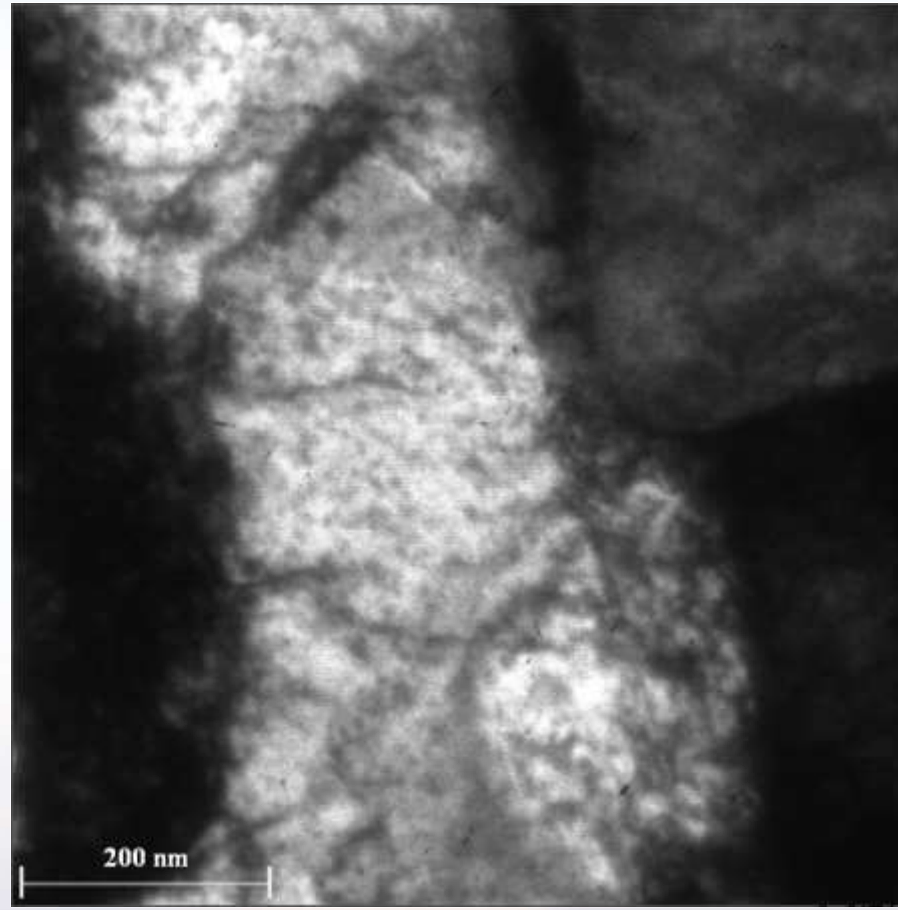
From space application through nuclear reactor cladding to waste storage:

Understanding Radiation Damage is Essential



UFG Tungsten

- **I³TEM W irradiation and He implantation of SPD-W developed for ITER applications**

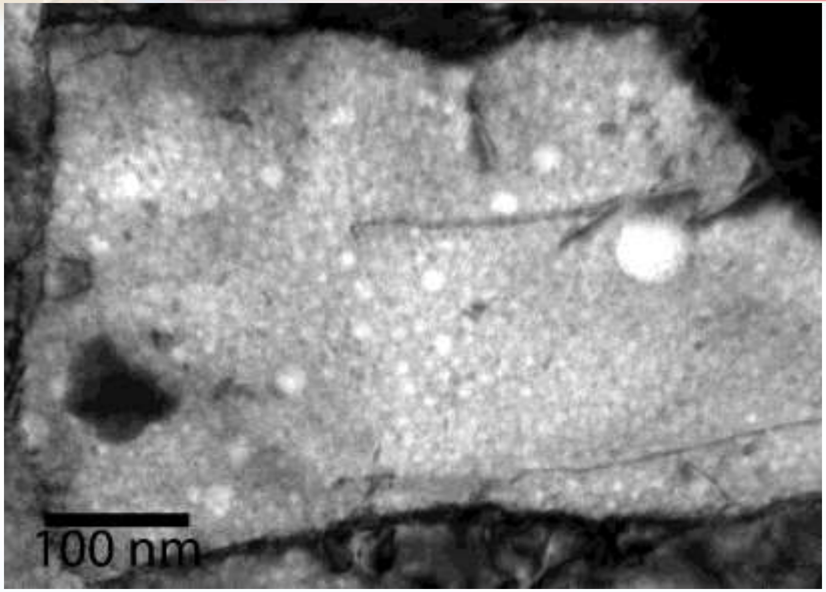


I³TEM is providing insight into:

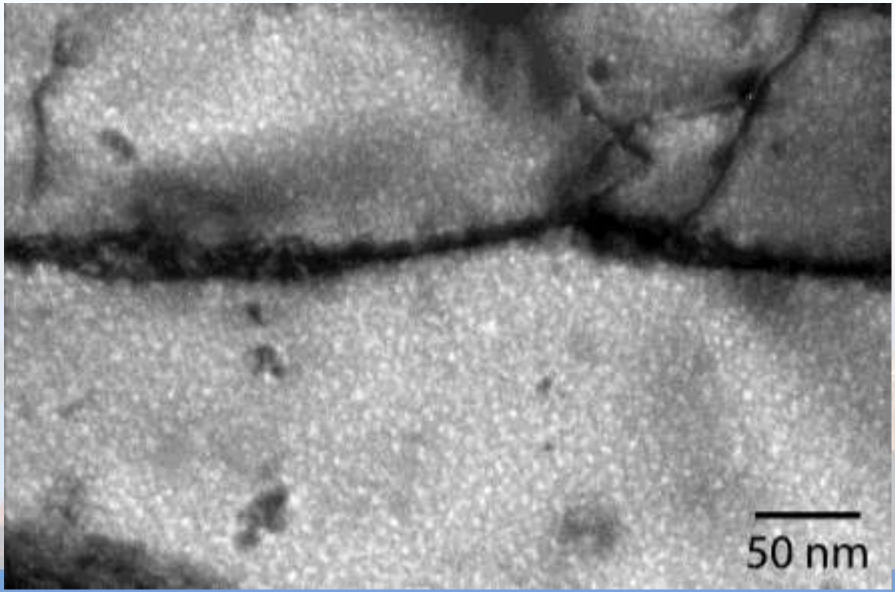
1) **Loop formation**
2) **Loop stability & migration**
3) **Rad & structural defect interactions**

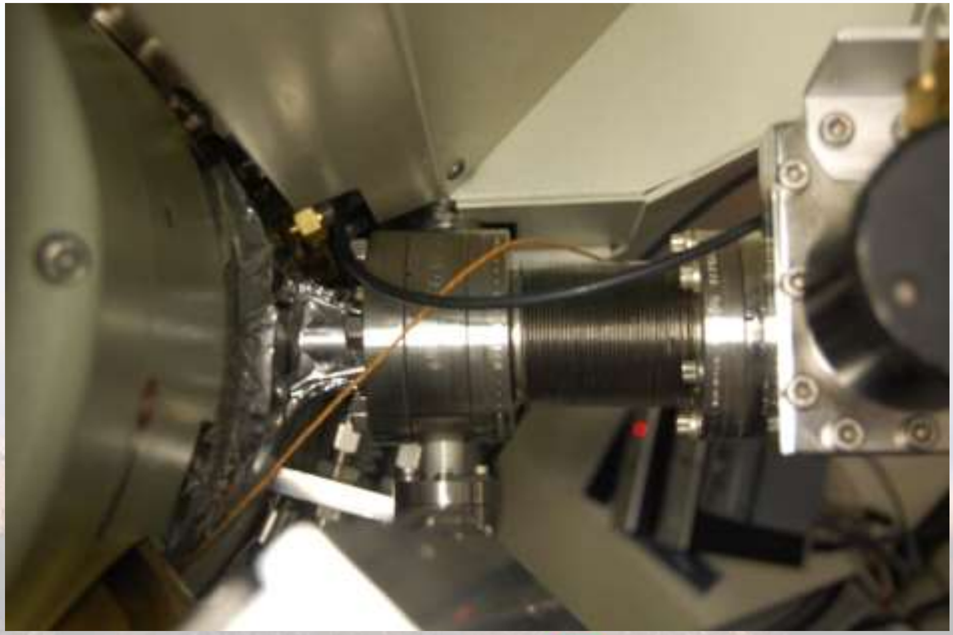
What Insight into Helium Bubble Formation is Gained from I^{3}TEM Experiments?

Collaborators: O. El-Atwani, J. P. Allain, D. Buller, & J.A. Scott



Electrostatic steering magnets were added into the bellows vicinal to the TEM. Should permit real time high magnification imaging of He bubbles.

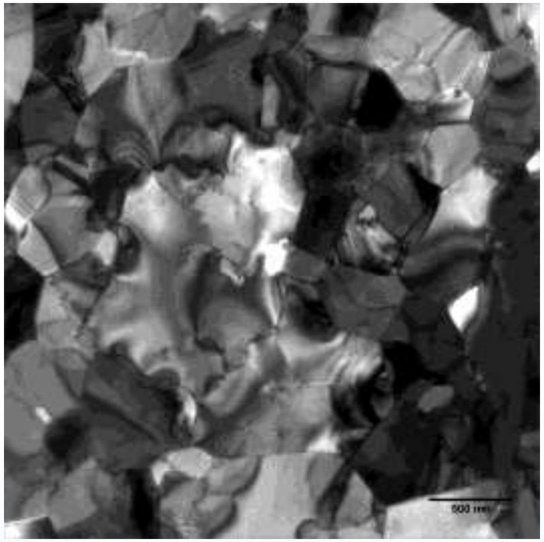




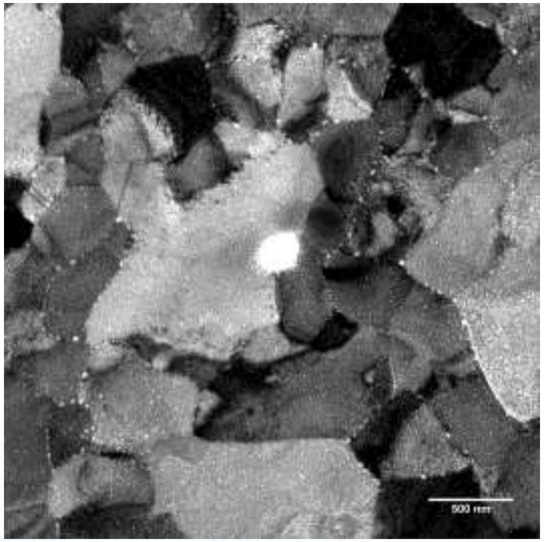
He Implantation and Subsequent Self-ion Irradiation of Au films

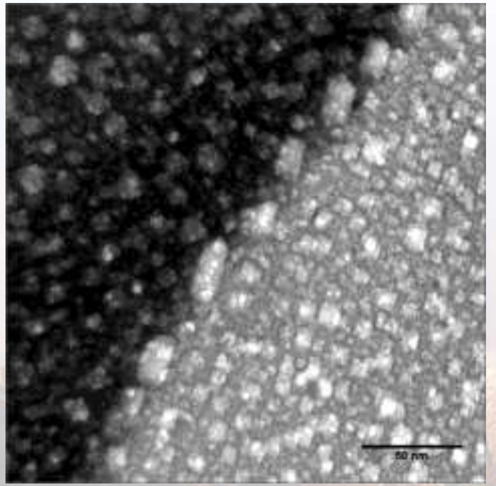
Collaborators: C. Chisholm & A.Minor

Unirradiated

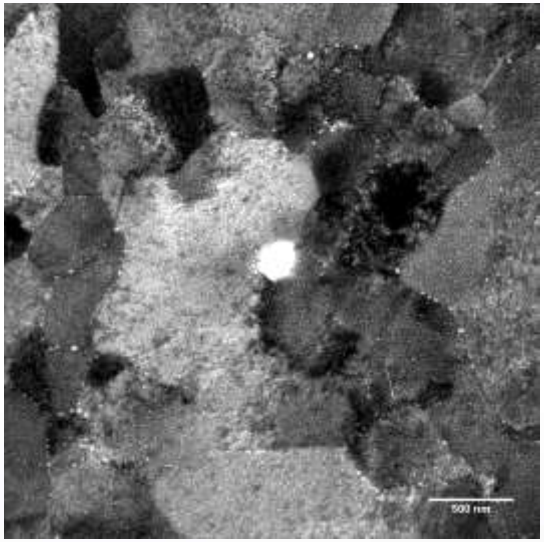


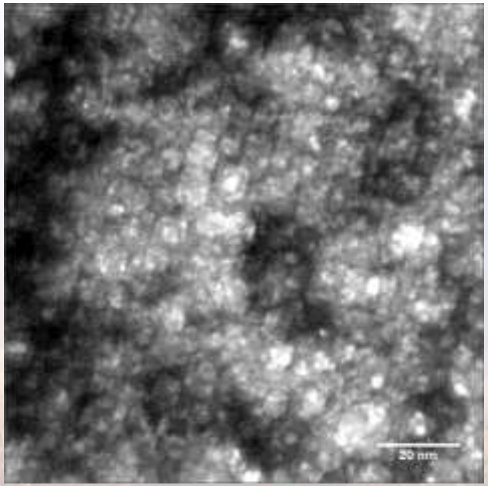
$1.5x10^{17}$ ions/cm^2 10keV He^{2+}





$1.5x10^{17}$ ions/cm^2 10keV He^{2+} then $5.8x10^{10}$ ions/cm^2 2.8MeV Au^{4+}



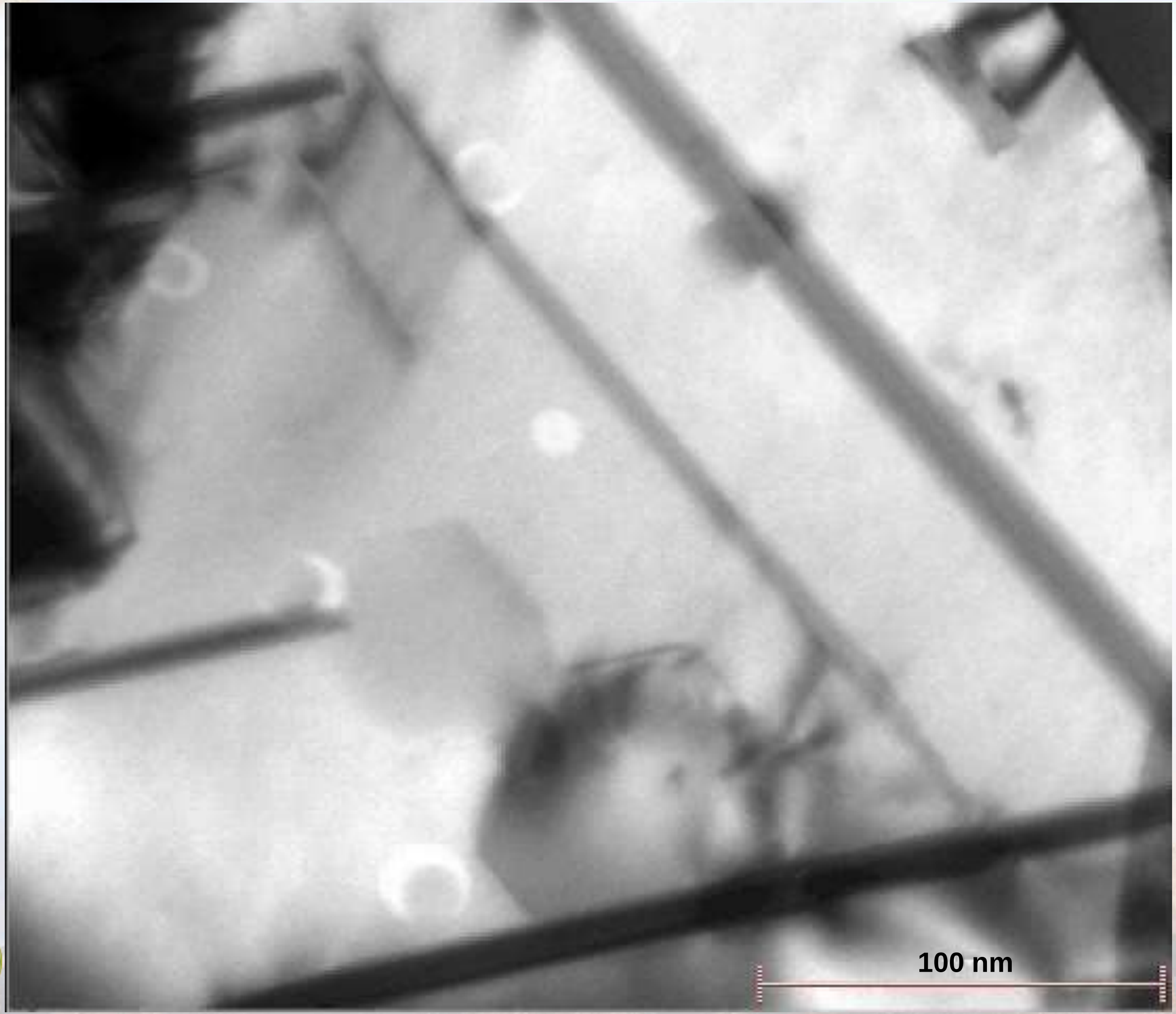


Gold thin-film was implanted with 10keV He^{2+} with a resulting porous microstructure

The thin-film was then irradiated with 2.8MeV Au^{4+}, adding dislocation loops

In situ Observations of Single Cascade Events?

Collaborators: C. Chisholm, A. Kinghorn, & A. Minor



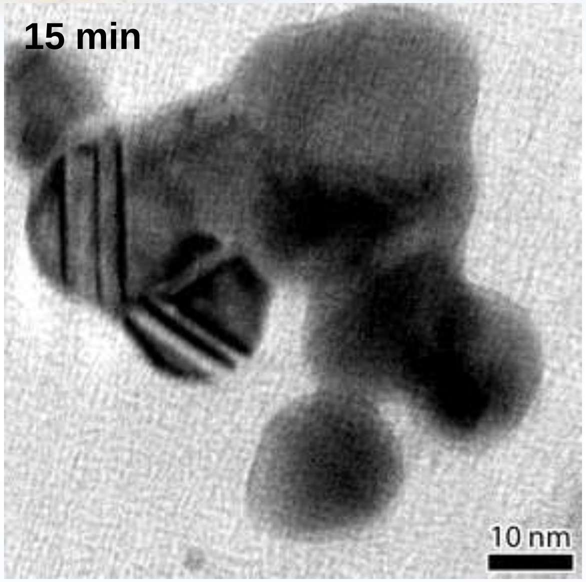
2.8MeV Au^{4+}

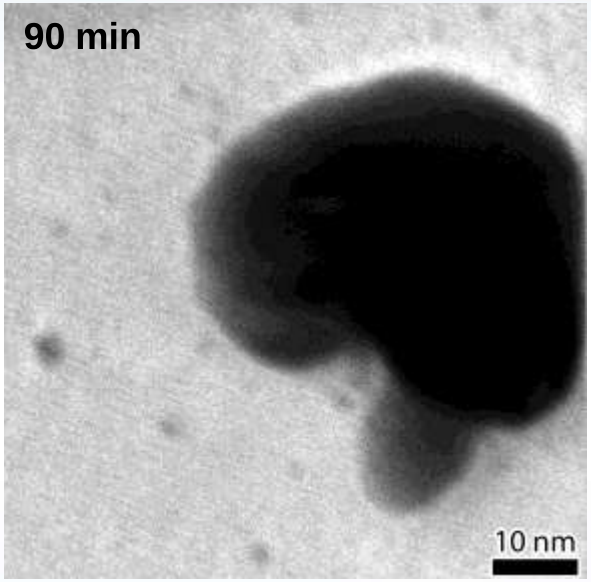
Very preliminary observations suggest that single ion strikes can be observed during self-ion irradiation of Au thin foils.

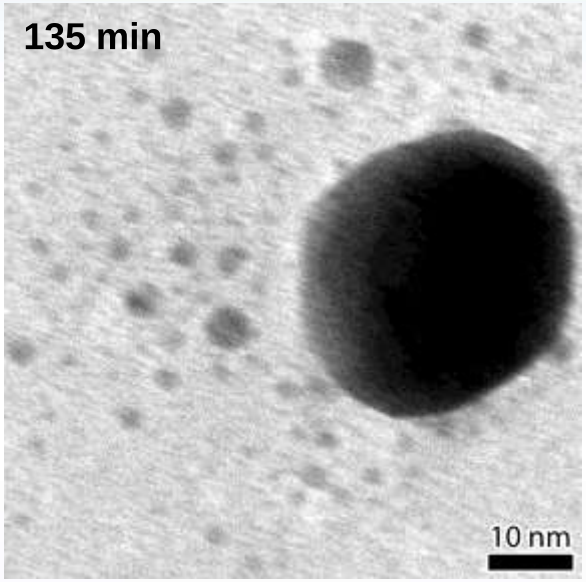
Current work is underway to confirm that each event is a single ion strike.

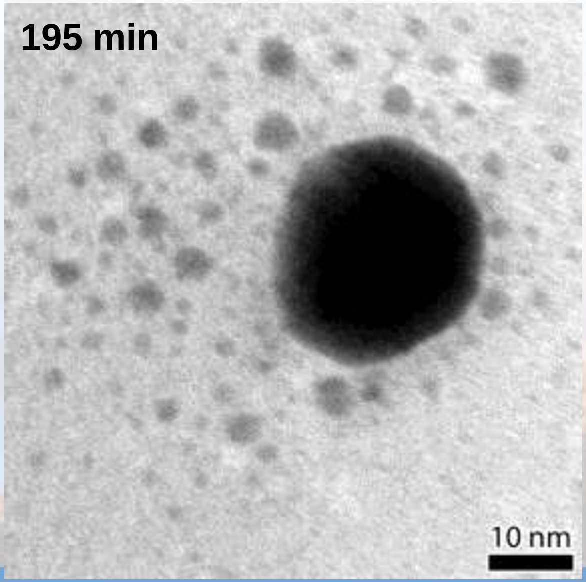
Au NP Irradiated with 100 nA of 3 MeV Cu^{3+}

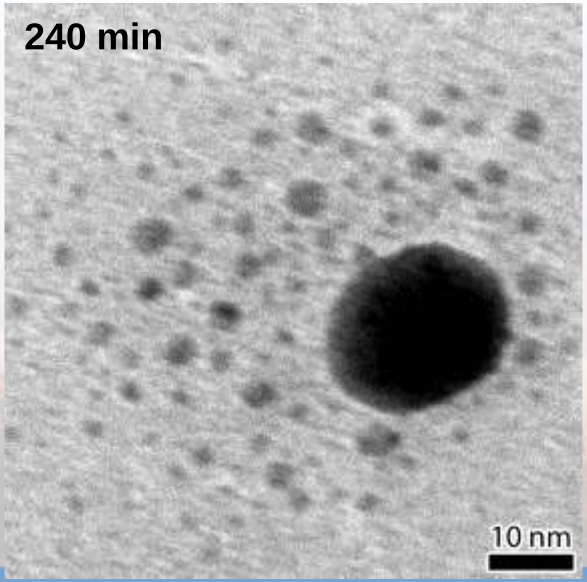
Collaborators: S.H. Pratt











Over 4 hours

a group of Au NP coalesced and smaller particles sputtered off, then grew

Advanced Microscopy Techniques Applied to Nanoparticles in Radiation Environments

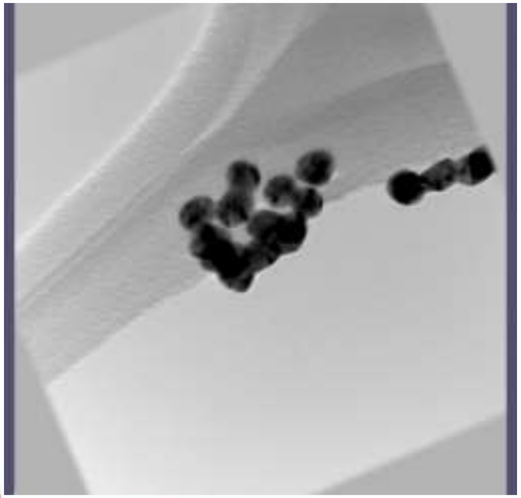
Collaborators: S.H. Pratt

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

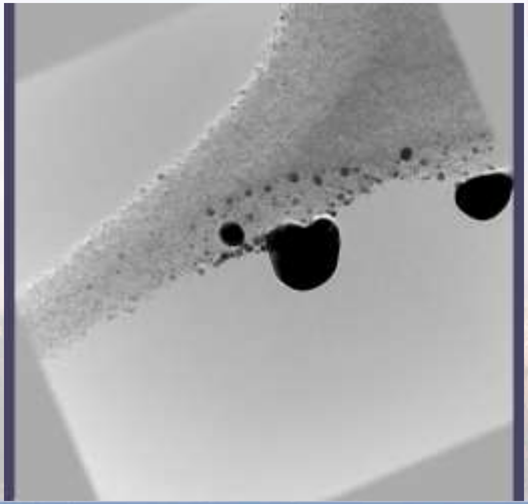
+

Hummingbird tomography stage

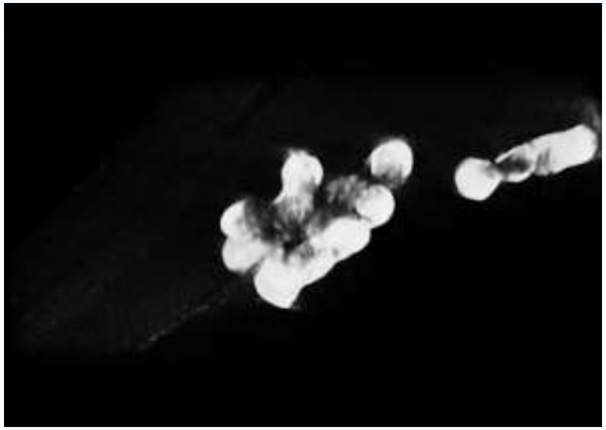
Aligned Au NP tilt series - unirradiated



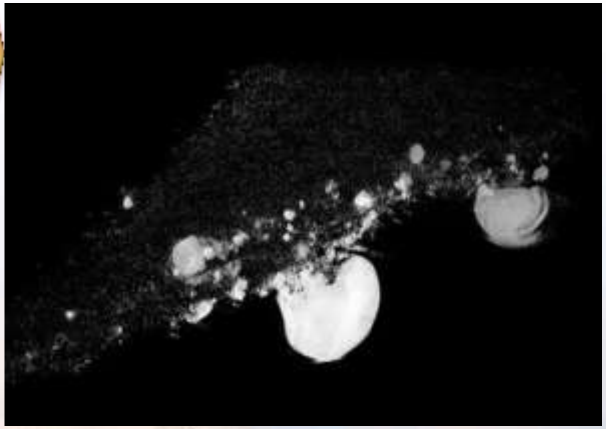
Aligned Au NP tilt series - irradiated



Unirradiated Au NP model



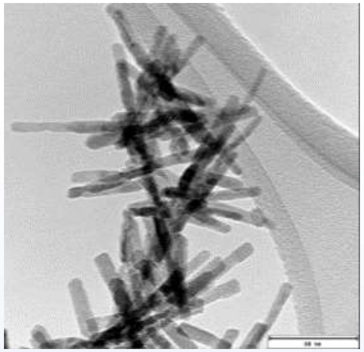
Irradiated Au NP model

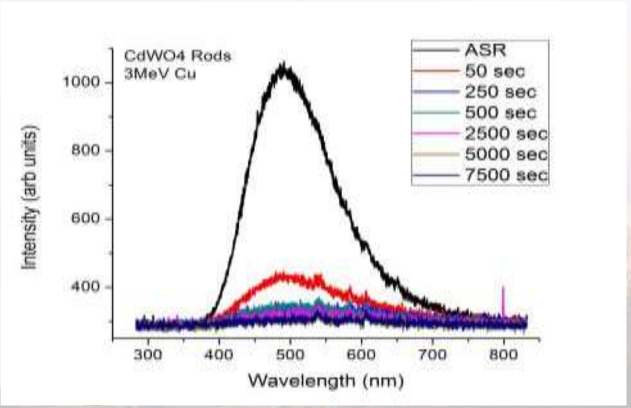


The application of advanced microscopy techniques to extreme environments provides exciting new research directions

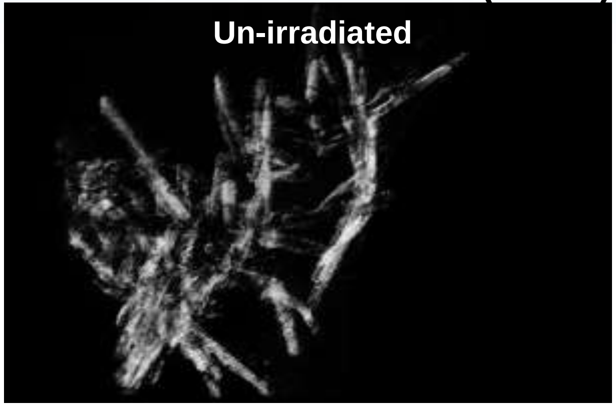
Radiation Tolerance is Needed in Advanced Scintillators for Non-proliferation Applications

Contributors: S.H. Pratt, B.A. Hernandez-Sanchez, T. Boyle

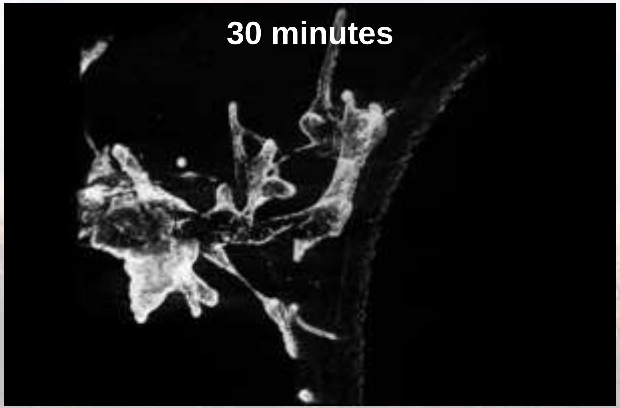




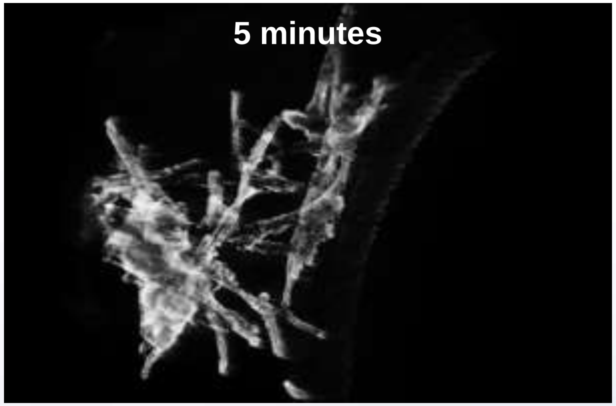
In situ Ion Irradiation TEM (I³TEM)



High-Z nanoparticles ($CdWO_4$) are promising, but are radiation sensitive



Tomography of Irradiated $CdWO_4$: 3 MeV Cu^{3+} at ~30 nA



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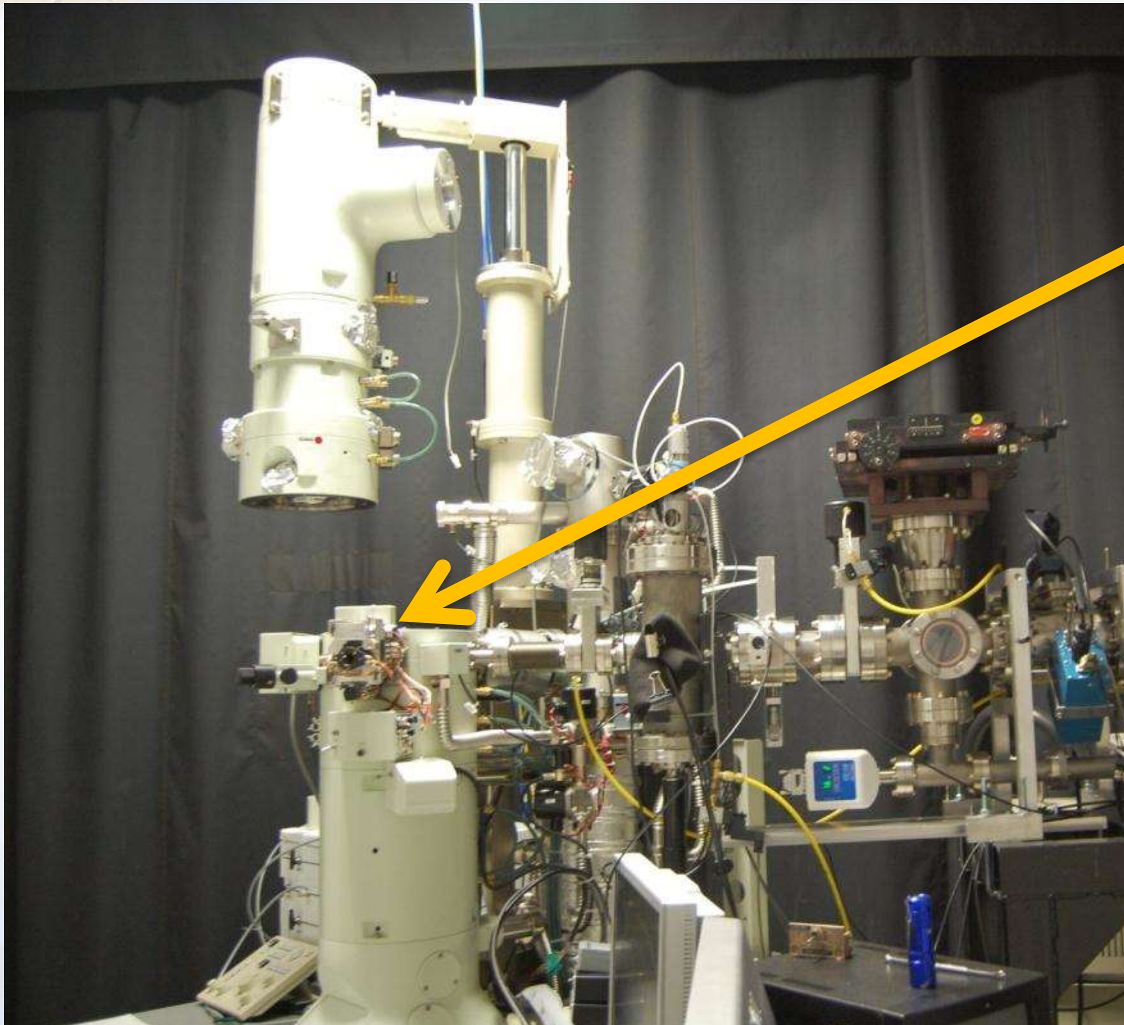
In situ TEM Luminescence

Collaborators: D. Masiel



Optical Mirror in TEM

First IBIL in TEM

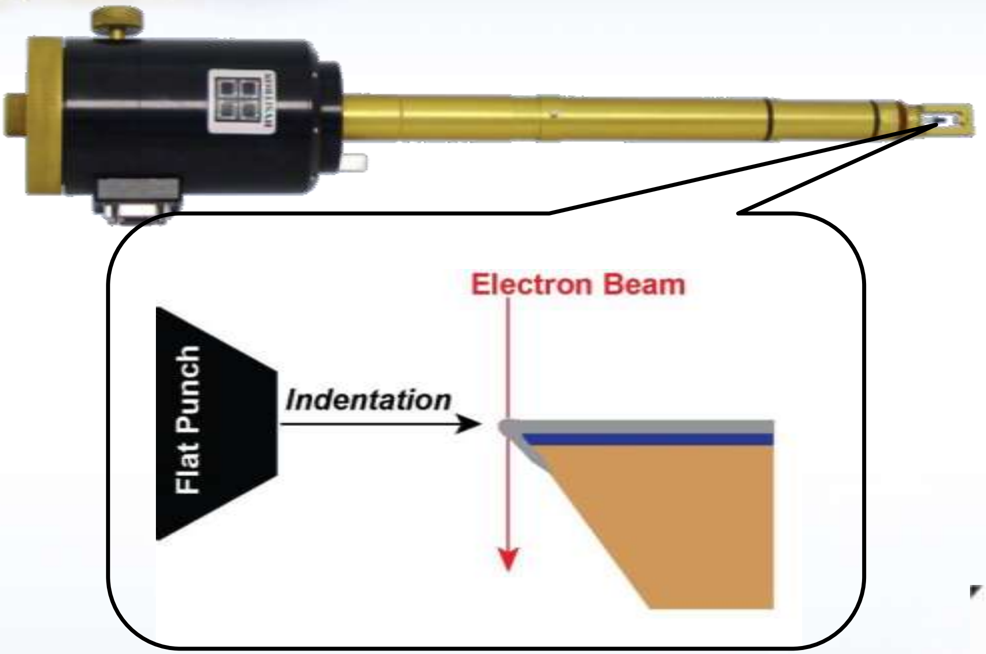
Optical Pathway in an I^3TEM

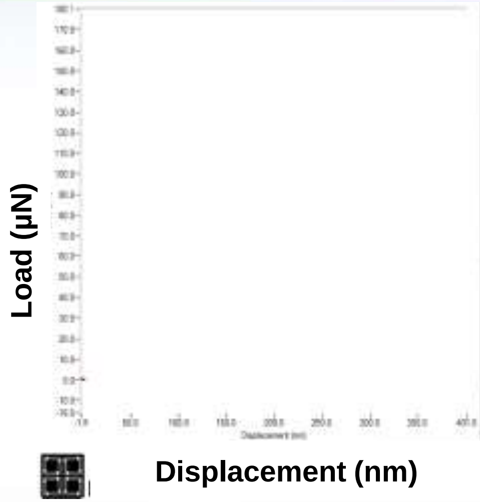
- Angled mirror with bore hole for the electron path was installed.
- Mirror is located on top of the objective polepiece
- Port was constructed with thick leaded glass to permit light through, but limited x-rays created by ion or electron beams.
- Permits *in situ* IBIL.

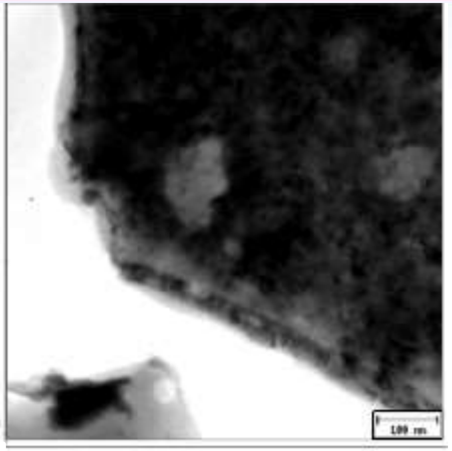
An optical port was added to the I^3TEM, which permits *in situ* TEM luminescence studies

In situ TEM Quantitative Mechanical Testing

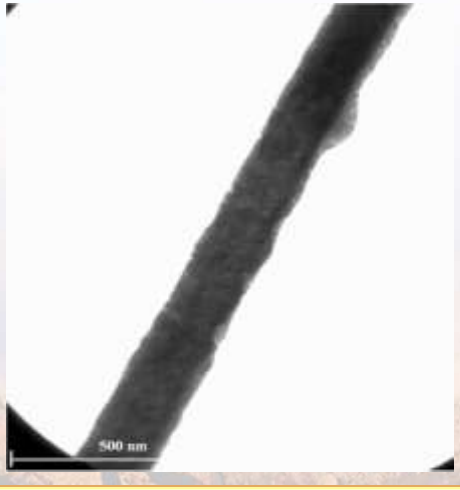
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

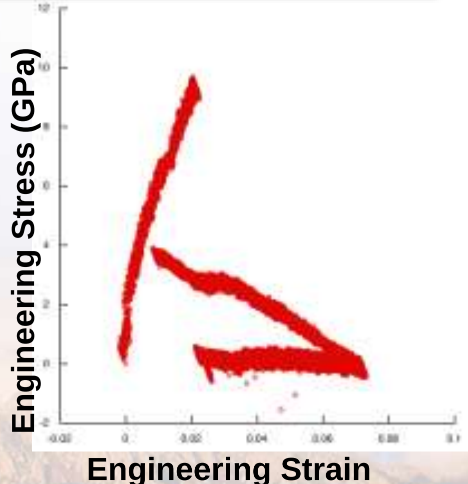






Fundamentals of Mechanical Properties





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

Range of Mechanical Testing Techniques

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

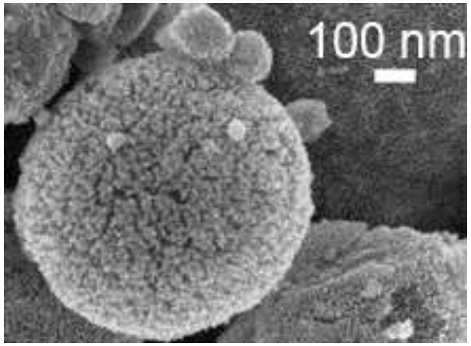
In situ TEM Gas Environments

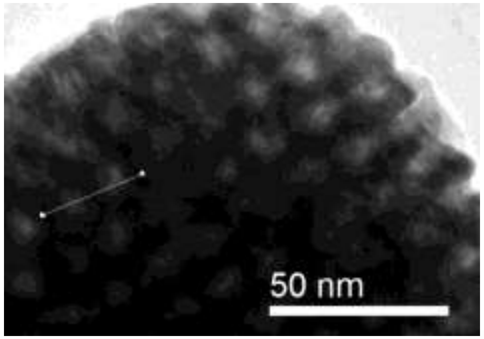
Contributors: B.G. Clark, S. Rajasekhara, P.J. Cappillino, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, L.R. Parent, I. Arslan. & Protochips, Inc.

Vapor-Phase Heating TEM Stage

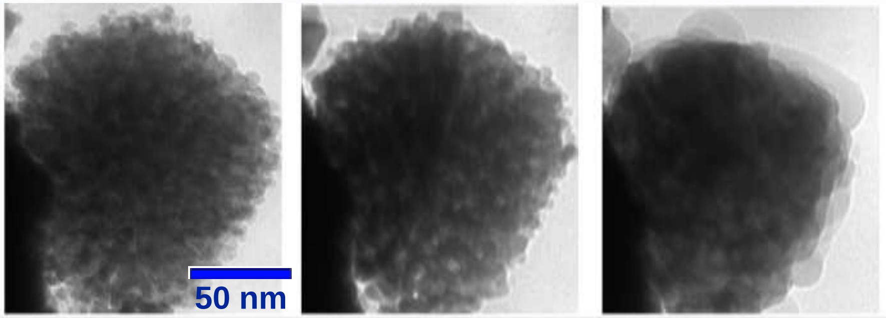
- Compatible with a range of gases
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Compatible with MS and other analytical tools

New *in situ* atmospheric heating experiments provide great insight into range of environmental studies





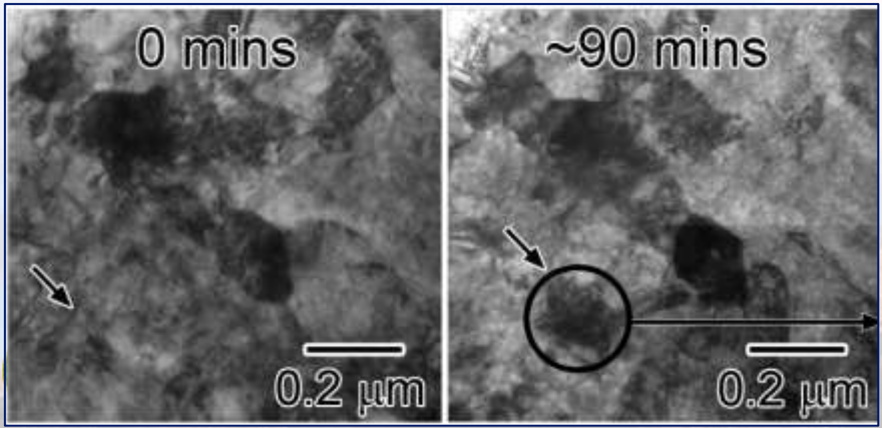
- 1 atm H_2 after several pulses to specified temp.



125° C 200° C 300° C

Absolute hydrogen pressure: 327 torr,

Ramp rate: 1 ^{o}C/s, Final temperature: ~ 400 C,



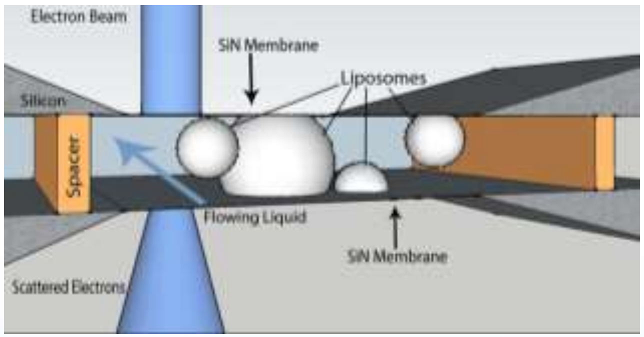
Even greater potential is envisioned when it is combined with *in situ* ion irradiation.

In situ TEM microfluidic Enviroments

Contributors: S.H. Pratt, E. Carnes, J. Brinker, D. Sasaki, D. Gross, J. Kacher, I.M. Robertson & Protochips Inc.

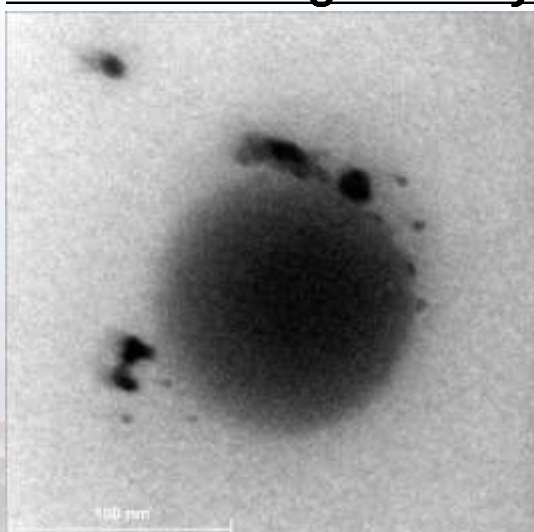
Microfluidic Stage

- Mixing of two or more channels
- Continuous observation of the reaction channel
- Chamber dimensions are controllable



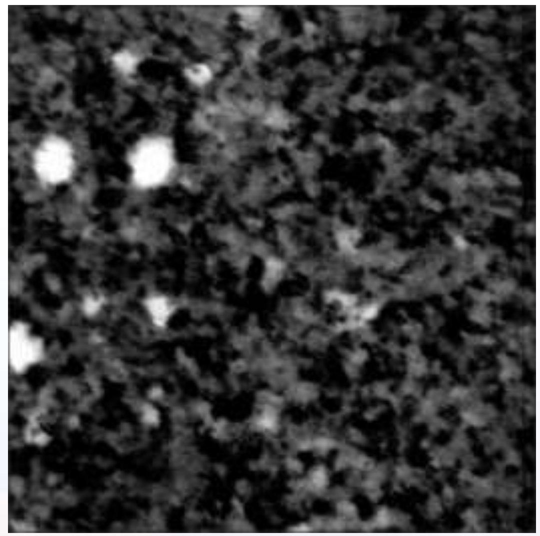
Protocell Drug Delivery

Liposome encapsulated Silica destroyed by the electron beam in aqueous environment



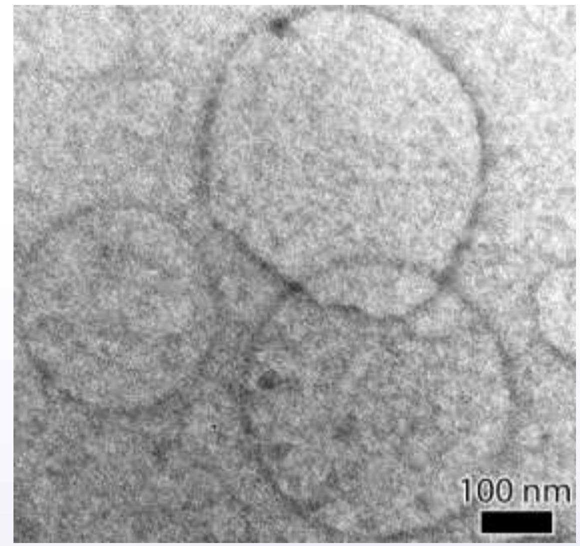
***In situ* microfluidic TEM can provide insight in events as diverse as corrosion and drug delivery**

Fe Corrosion



Dilute flow of acetic acid over 99.95% nc-PLD Fe

Liposomes in Water



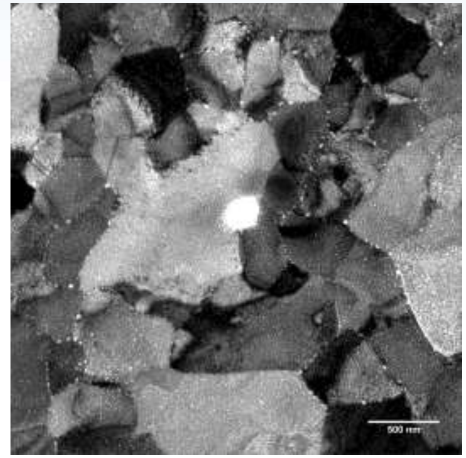
Liposomes imaged in flowing aqueous channel

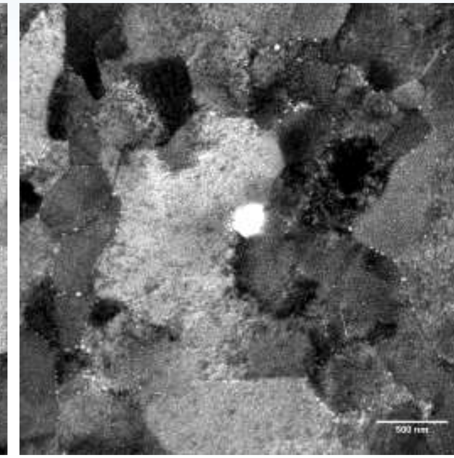
We hope to go a step further and combine it with ion implantation and irradiation environments

Summary

<u>Current Status of the I[3]TEM</u>

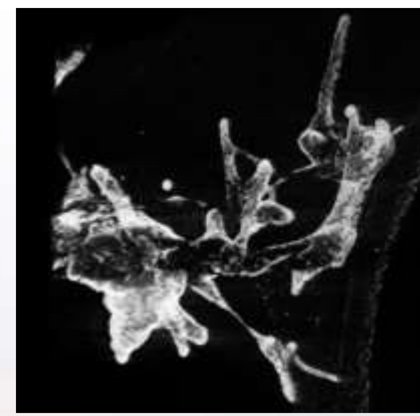
- Perform real team heavy ion irradiation
- Ion implantation capability is operational
  - Still being optimized
- Can operate at elevated temperatures
- Combined with tomography
  - Provides 4D insight as a function of dose





<u>Future Combinations with I[3]TEM</u>

- Quantitative mechanical property testing
- High temperature gas environments
- Microfluidic environments
- …



Sandia's I[3]TEM, although still under development, is testing the frontiers of *in situ* ion irradiation microscopy and providing a wealth of interesting initial observations along the way

Contributors & Collaborators:

J. Hinks, S.H. Pratt, A. Kinghorn, M. Steckbeck, B.A. Hernandez-Sanchez, T. Boyle, E. Carnes, J. Brinker, D. Sasaki, D. Gross, J. Kacher, I.M. Robertson, Protochips, B.G. Clark, S. Rajasekhara, P.J. Cappillino, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, L.R. Parent, I. Arslan, J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, Hysitron, & D. Masiel

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