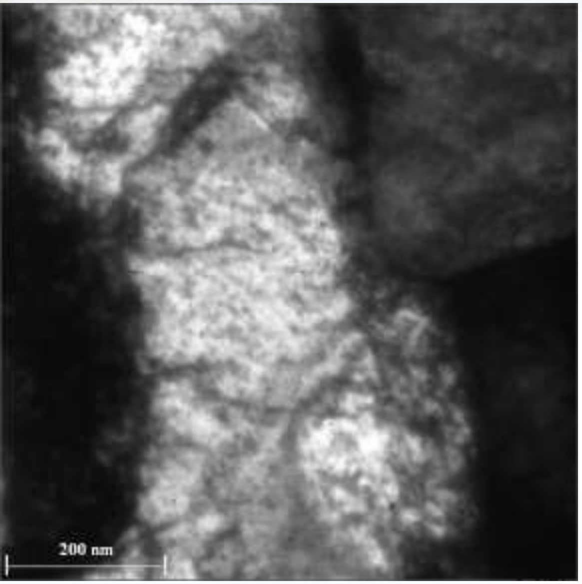
In situ TEM Studies of Ion Beam Radiation Effects

SAND2013-3496C

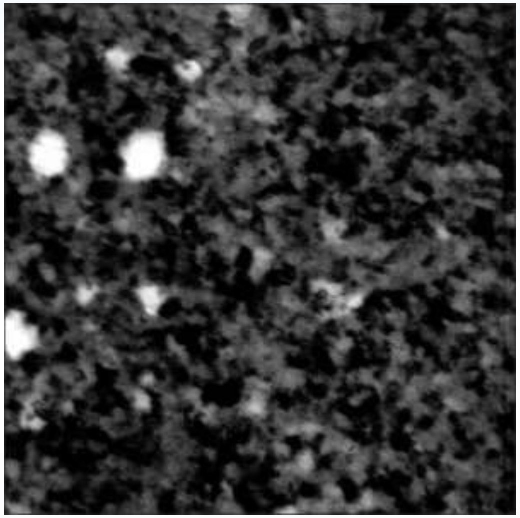


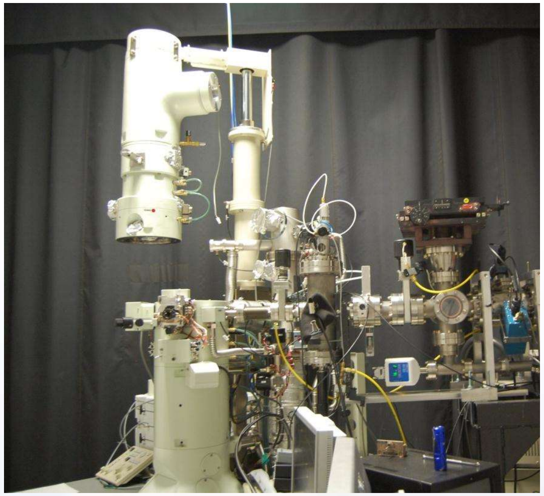
K. Hattar

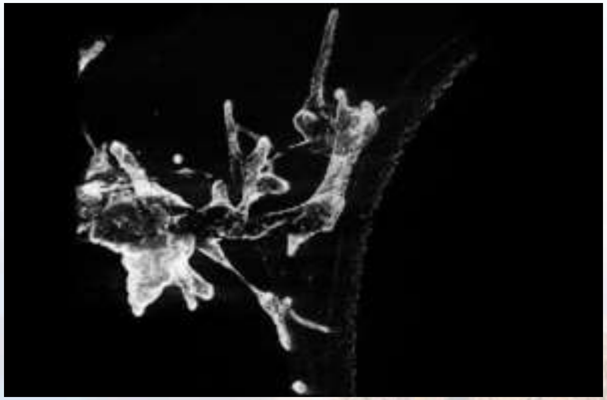
Ion Beam Lab

Sandia National Laboratories

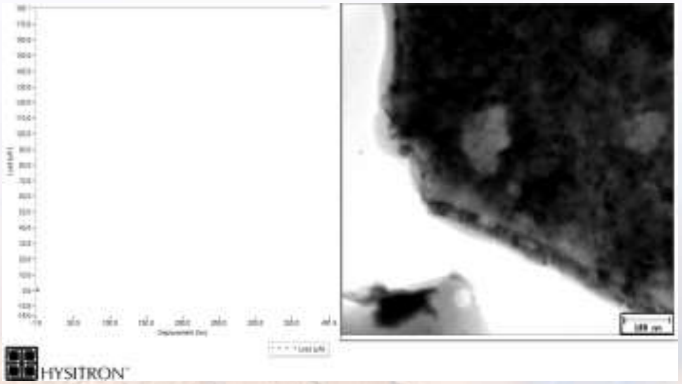
May 14, 2013







In situ Ion Irradiation TEM is operational, but still in development



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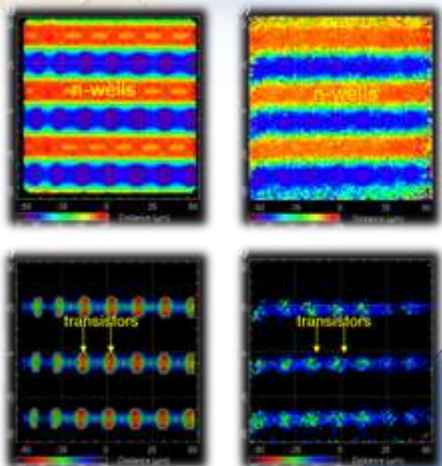
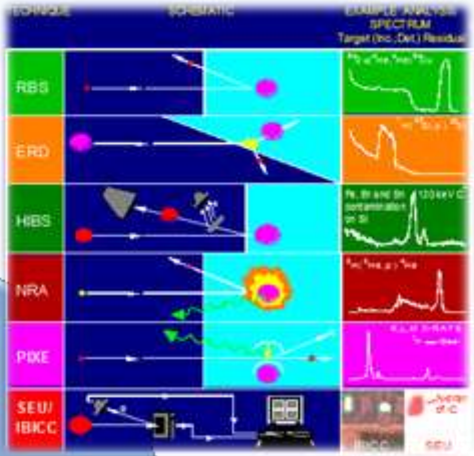
Sandia's Ion Beam Laboratory

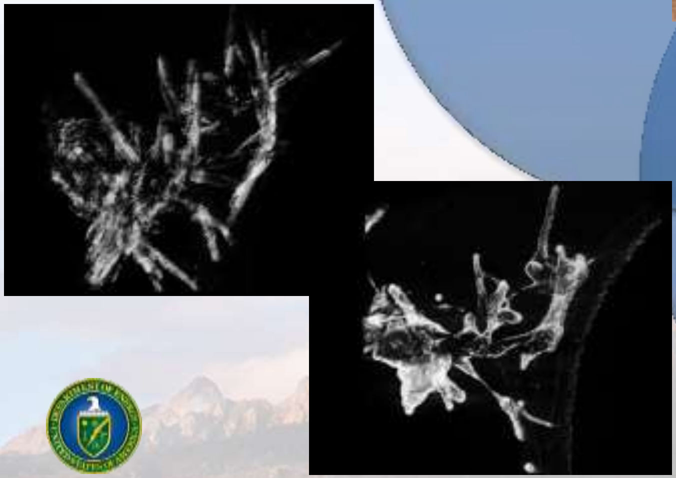


Ion Beam Analysis (IBA)



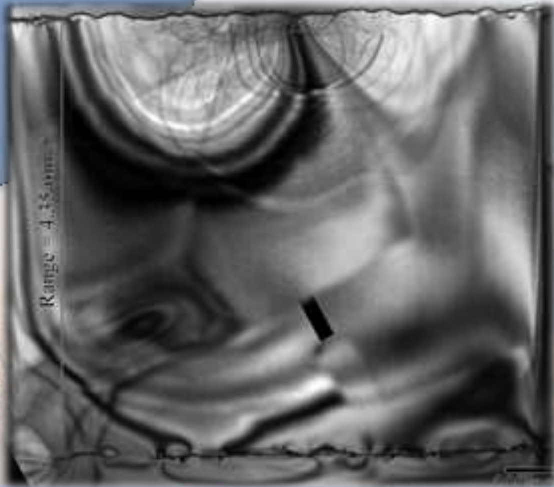
Radiation Effects Microscopy (REM)

Ion Beam Modification (IBM)



In situ Ion Irradiation Transmission Electron Microscopy

(I^3TEM)

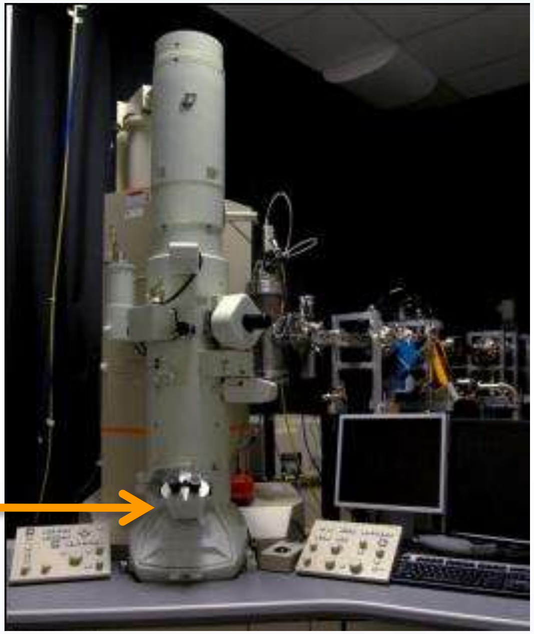


In situ Ion Irradiation TEM (I^{3}TEM)

Collaborators: D. Buller, B.G. Clark, & B.L. Doyle

Proposed Capabilities

- 200 kV LaB_6 TEM
- Ion beams considered:
    - 1 MeV H^{1+}
    - 3 MeV He^{1+} Si^{3+} Ni^{3+} Cu^{3+} Au^{3+} W^{3+}
    - 14 MeV Si^{3+}
    - 10 keV D^{2+}
    - 8 to 10 keV He^{+}
  - All beams hit the same location
- Electron tomography
- Nanosecond time resolution (DTEM)
- Precession scanning (EBSD in TEM)
- *In situ* PL, CL, and IBIL
- *In situ* heating and cooling stages
- *In situ* electrical measurement stage
- *In situ* quantitative mechanical testing
- *In situ* vapor phase stage
- *In situ* liquid mixing stage



TVIPS

Hummingbird

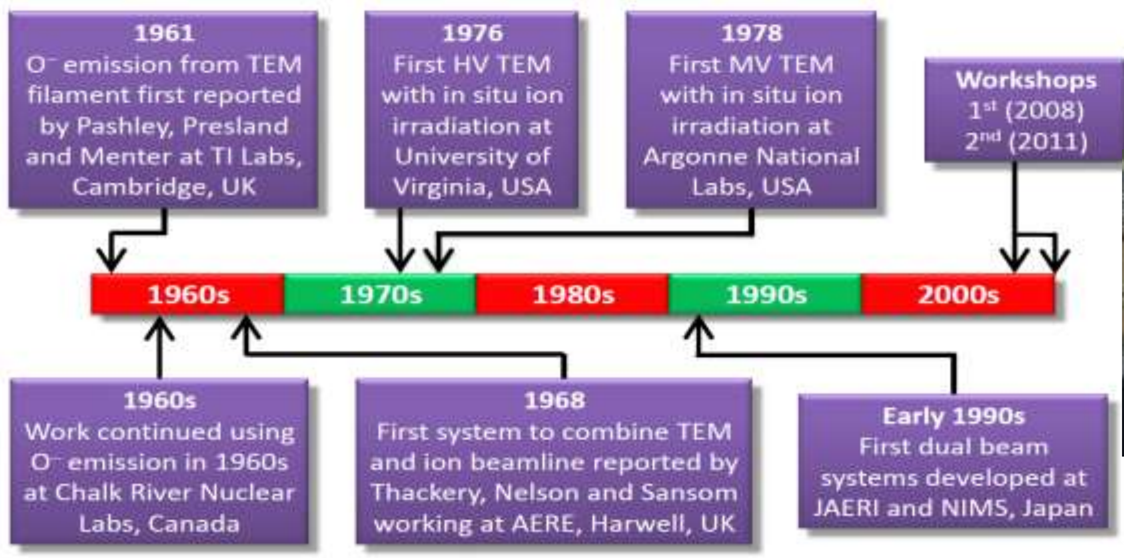
In situ IBIL

We have completed the Tandem accelerator connection and Colutron accelerator connection.

Many potential additions are being considered

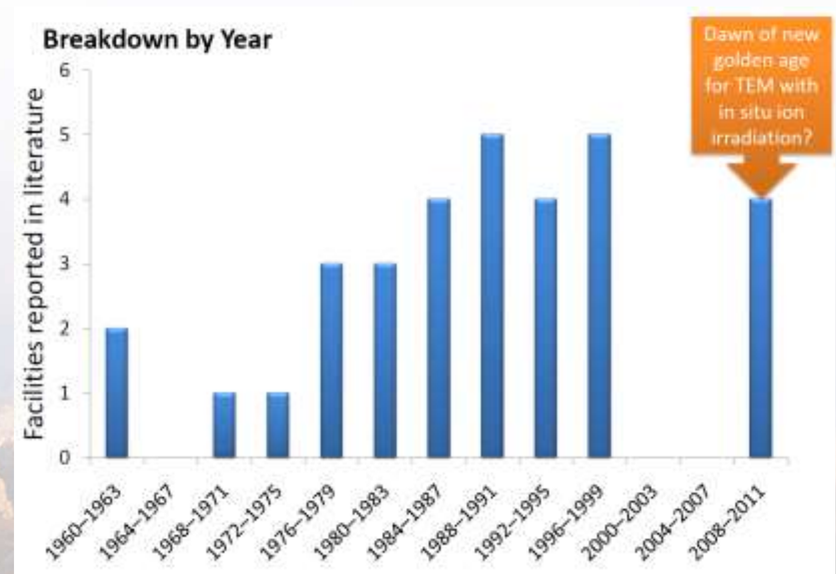
History of *in situ* Irradiation TEM

Courtesy of: J. Hinks

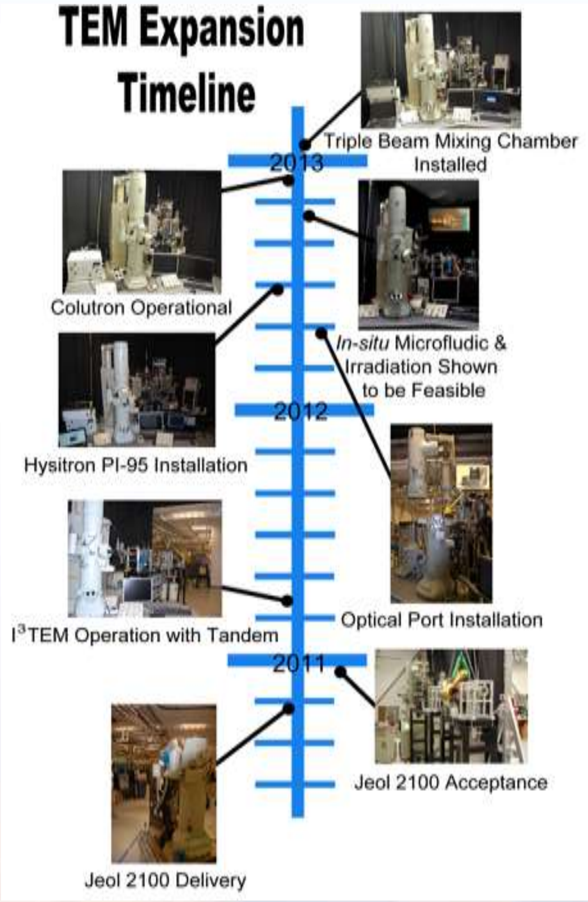


"The direct observation of ion damage in the electron microscope thus represents a powerful means of studying radiation damage"

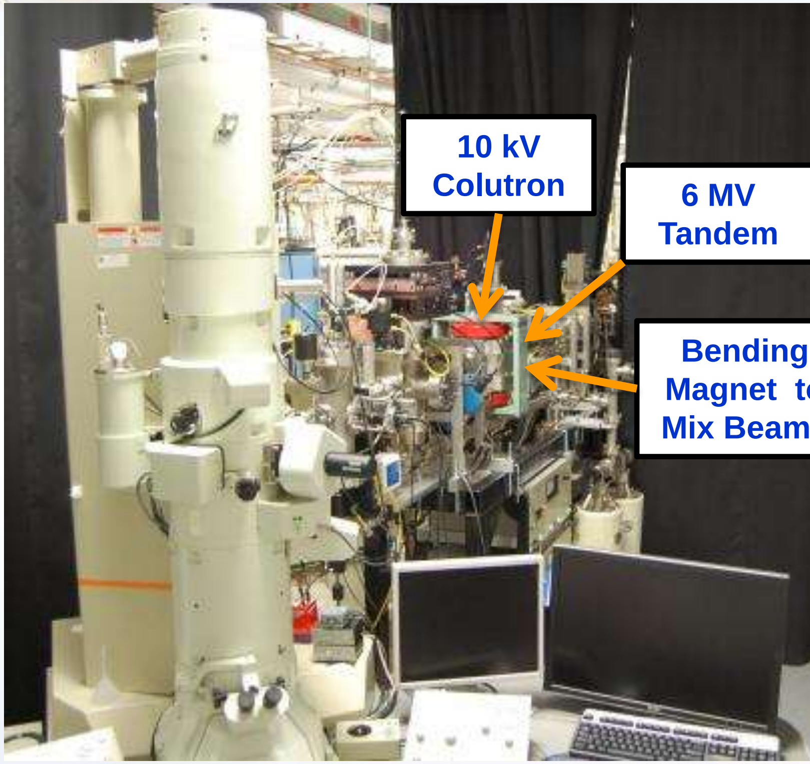
D.W. Pashley and A.E.B Presland *Philosophical Magazine* **6 (68)** (1961) p1003

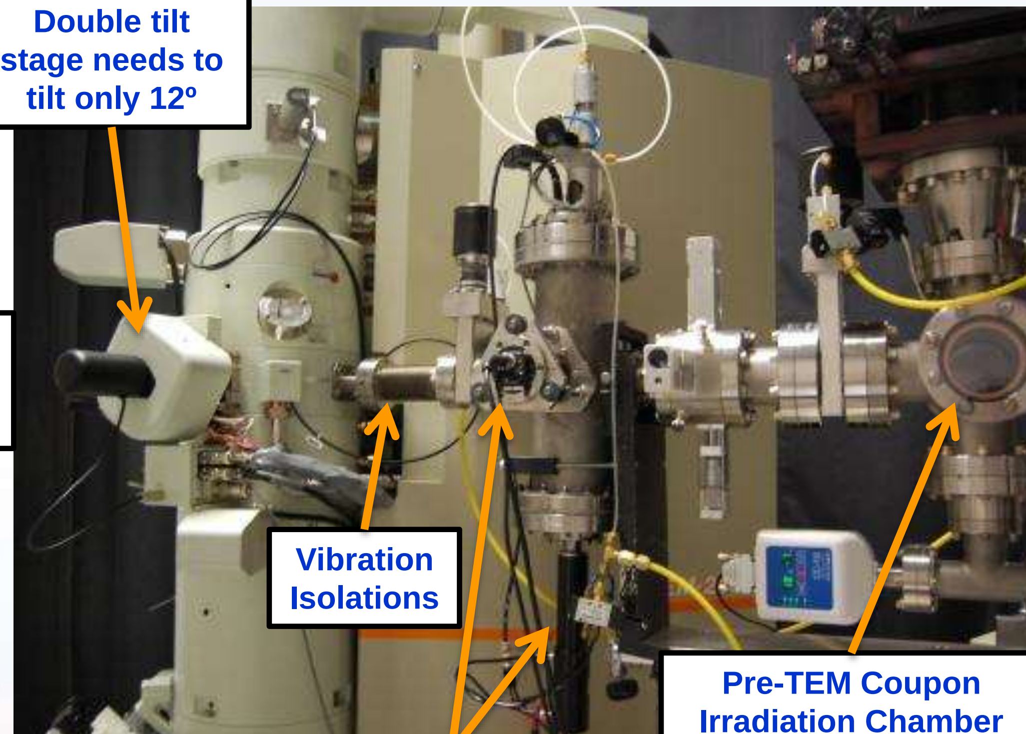


I³TEM History at the Ion Beam Lab



Current Status of the *In situ* TEM Beamline





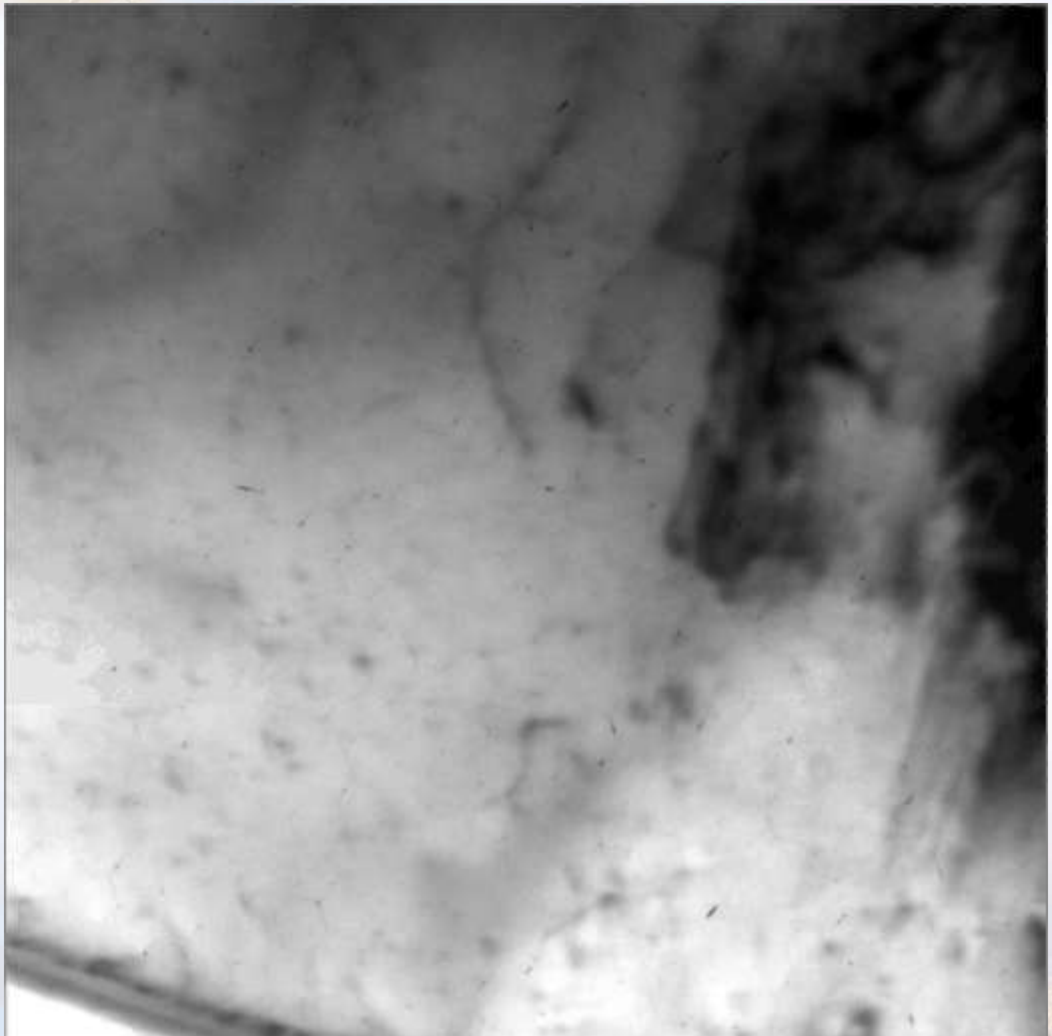
Beam burn from 14 MeV Si

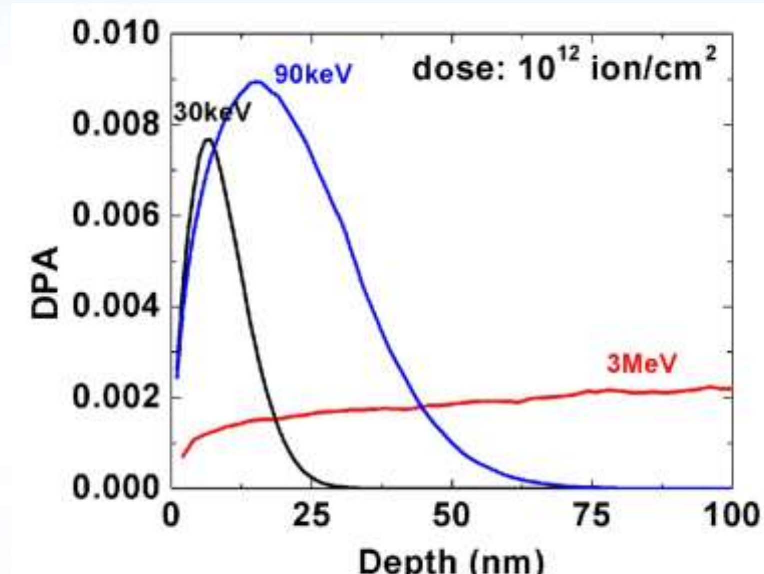
I^3TEM is operational, but also still in development

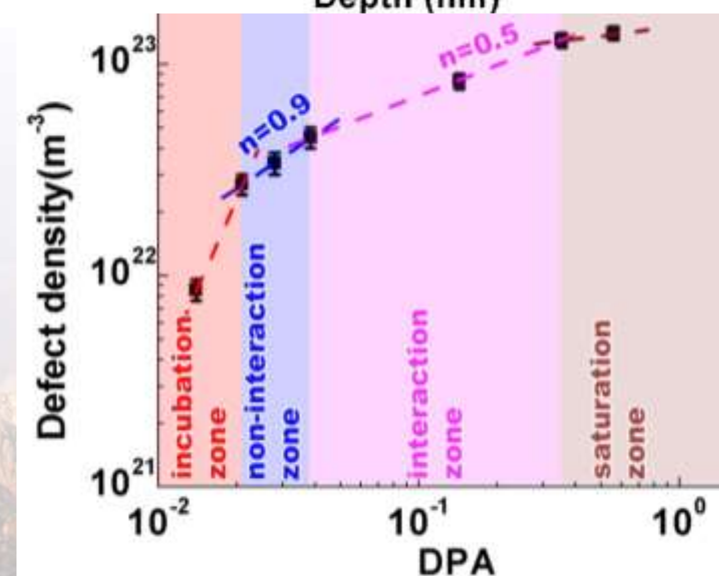
Microfluidic Holder Beam Burn

Evolution of Radiation Defects in Cu TEM Foil

Collaborators: N. Li, A. Misra



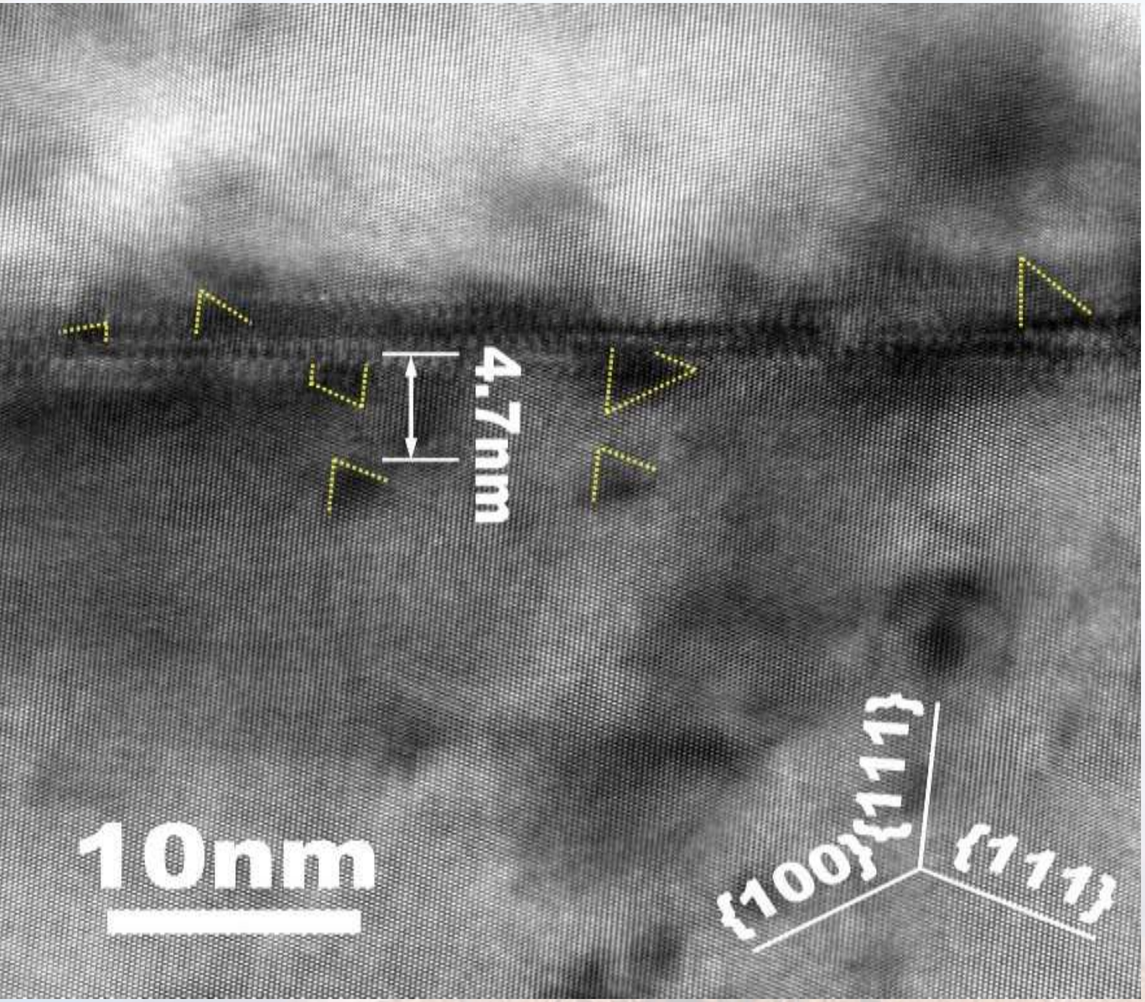


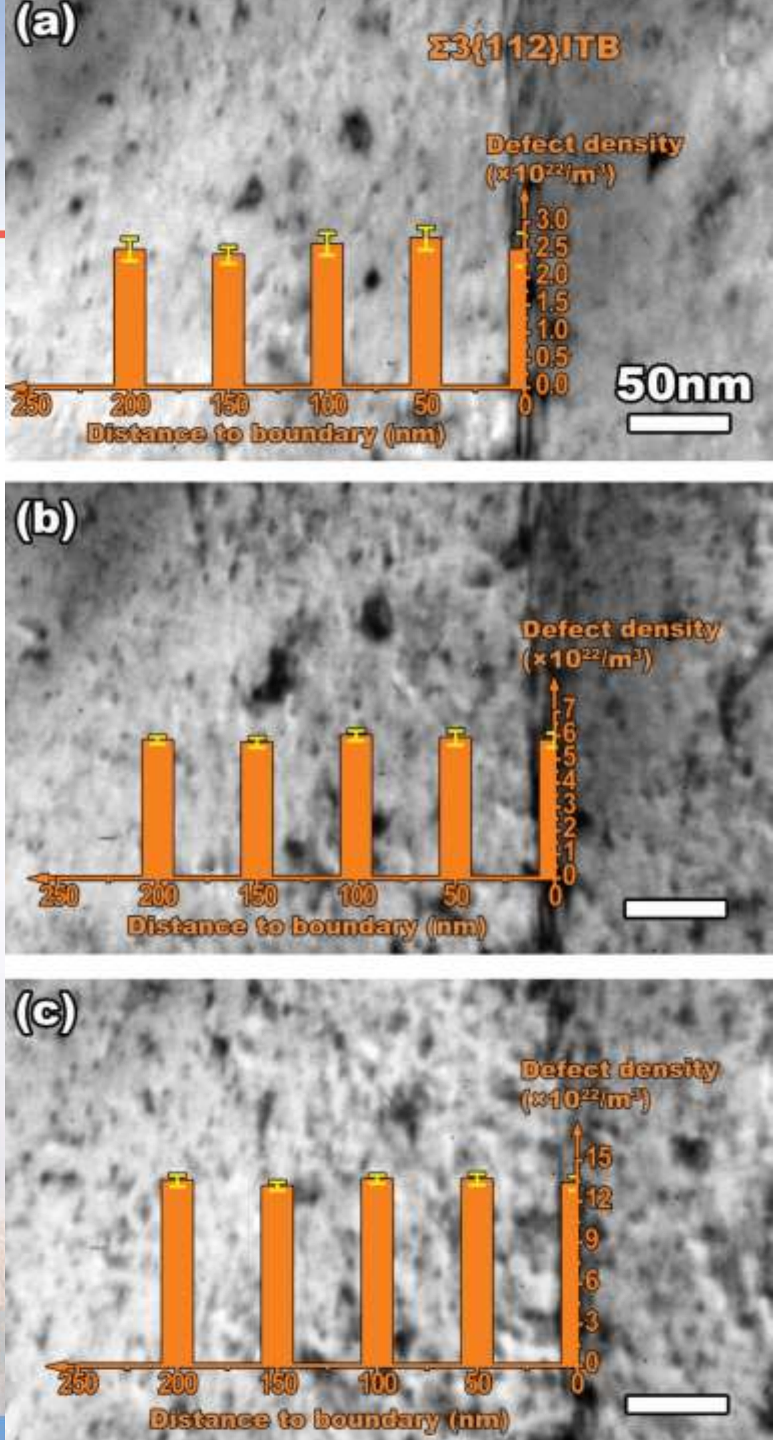


- FIB processed sputter deposited high purity Cu foils
- Tailored to have high density of Σ3 boundaries

Defect are Altered Little by the Presence of Grain Boundaries

Collaborators: N. Li, A. Misra



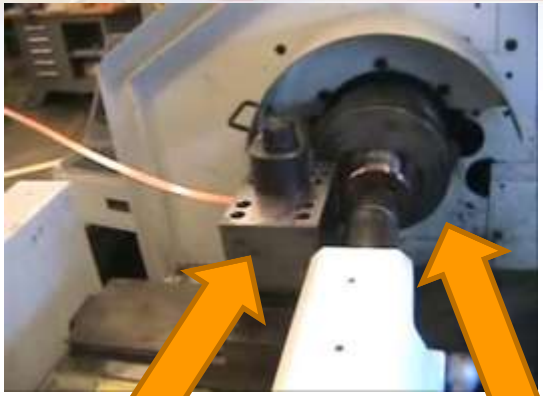


SFT appear to be directly at GB

No change in defect density is observed near GB

What Insight into Structural Stability is Gained from I^3TEM Experiments?

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

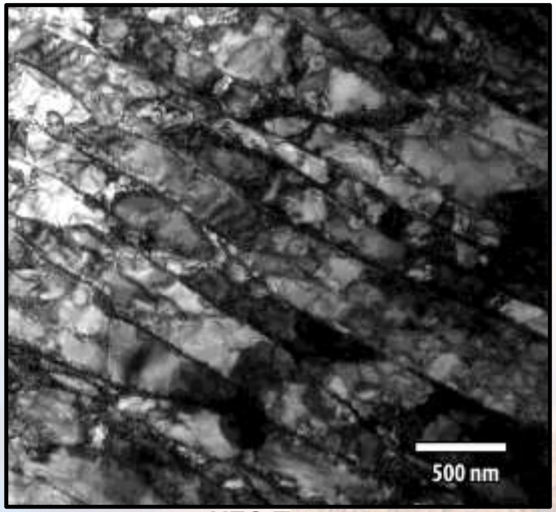


Extrusion Machining tooling

Commercially available lathe

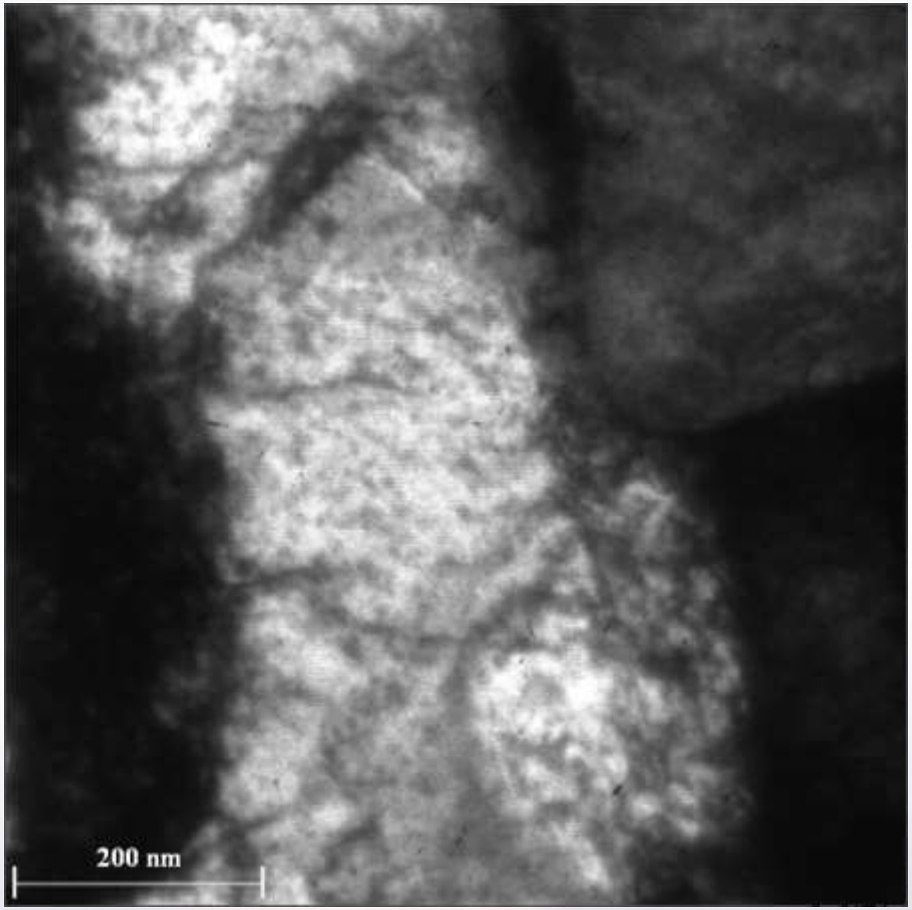
From NW components through proposed NE cladding to waste storage:

Understanding Radiation Damage is Essential



UFG Tungsten

- I^3TEM W irradiation and He implantation of SPD-W developed for ITER applications

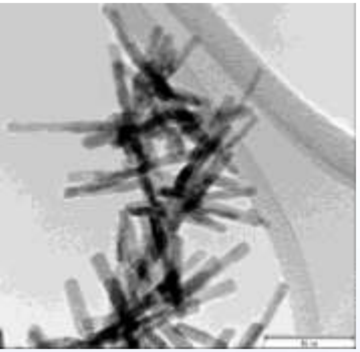


I^3TEM is providing insight into:

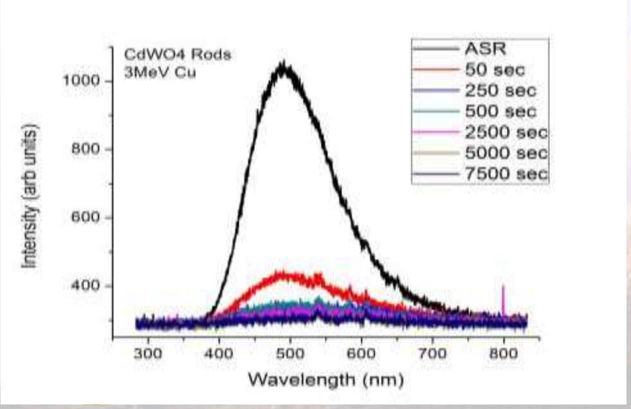
1) Loop formation
2) Loop stability & migration
3) Rad & structural defect interactions

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

Contributors: S.M. Hoppe, B.A. Hernandez-Sanchez, T. Boyle



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

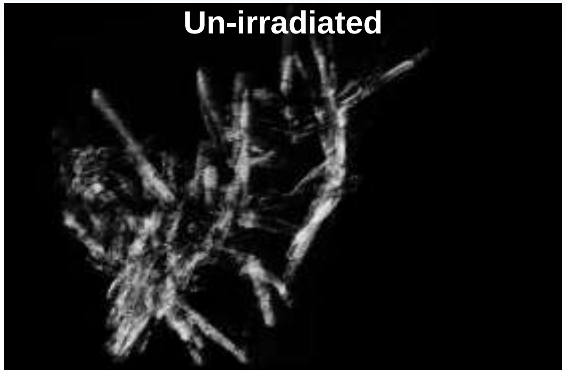


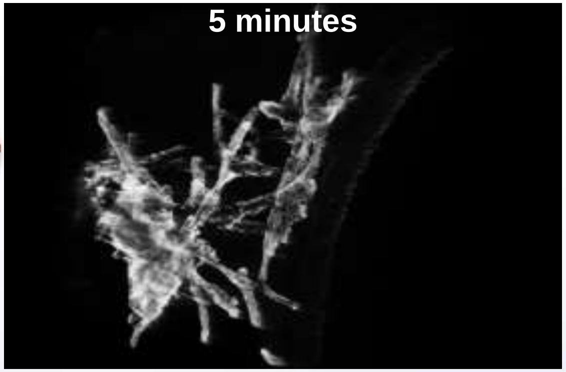
In situ Ion Irradiation TEM (I[3]TEM)

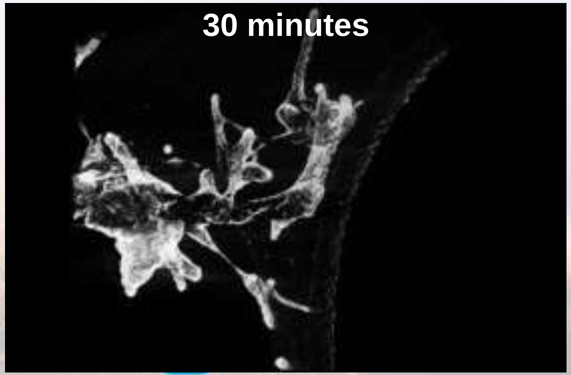
+

Hummingbird tomography stage

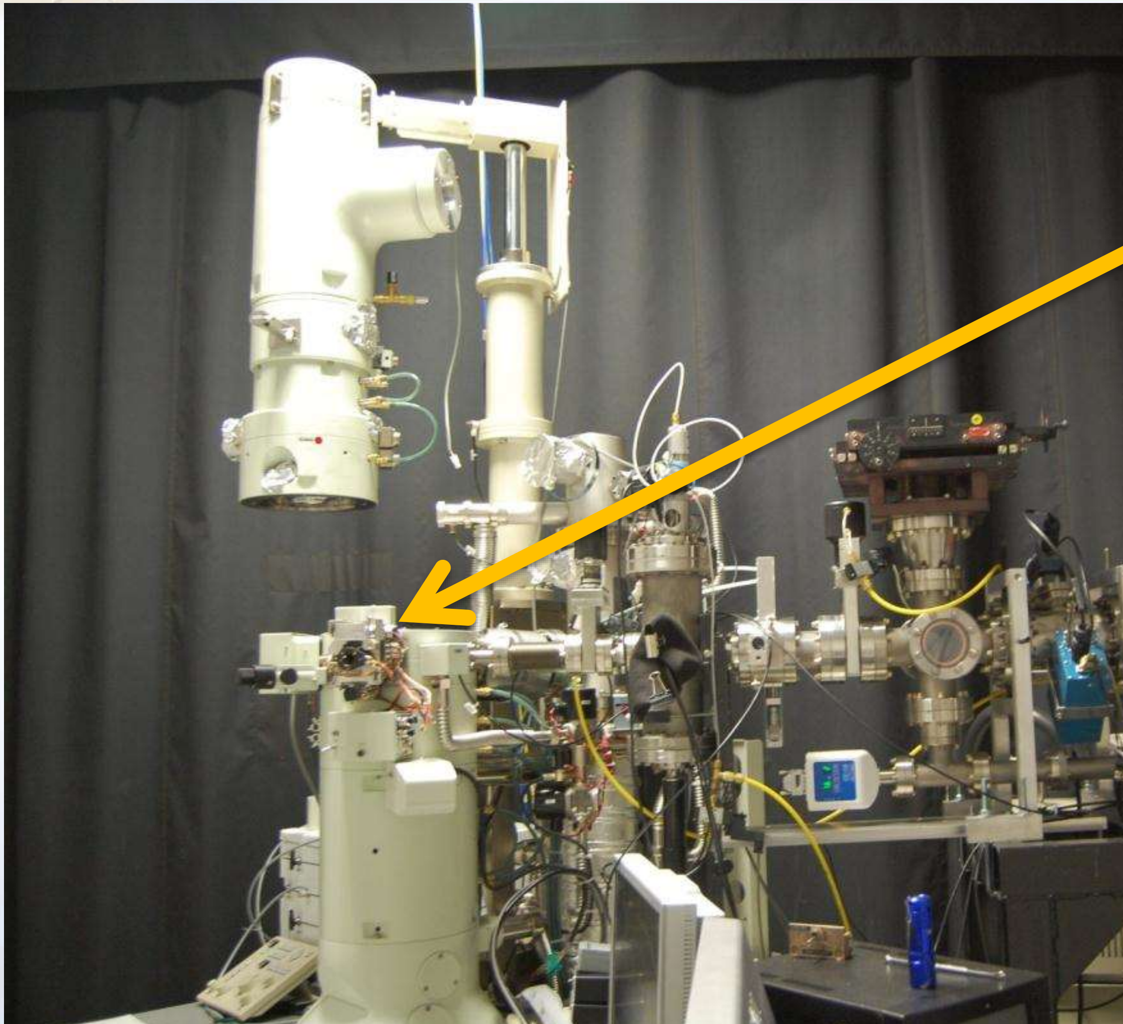
Tomography of Irradiated $CdWO_4$:
3 MeV Cu^{3+} at ~30 nA


Un-irradiated


5 minutes


30 minutes

In situ TEM Luminescence



Optical Mirror in TEM

First IBIL in TEM

Optical Pathway in an I^{3}TEM

- **Angled mirror with bore hole for the electron path was installed.**
- **Mirror is located on top of the objective polepiece "heart of the TEM"**
- **Port was constructed with thick leaded glass to permit light through, but not x-rays created by ion or electron beams.**
- **Permits *in situ* IBIL.**

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

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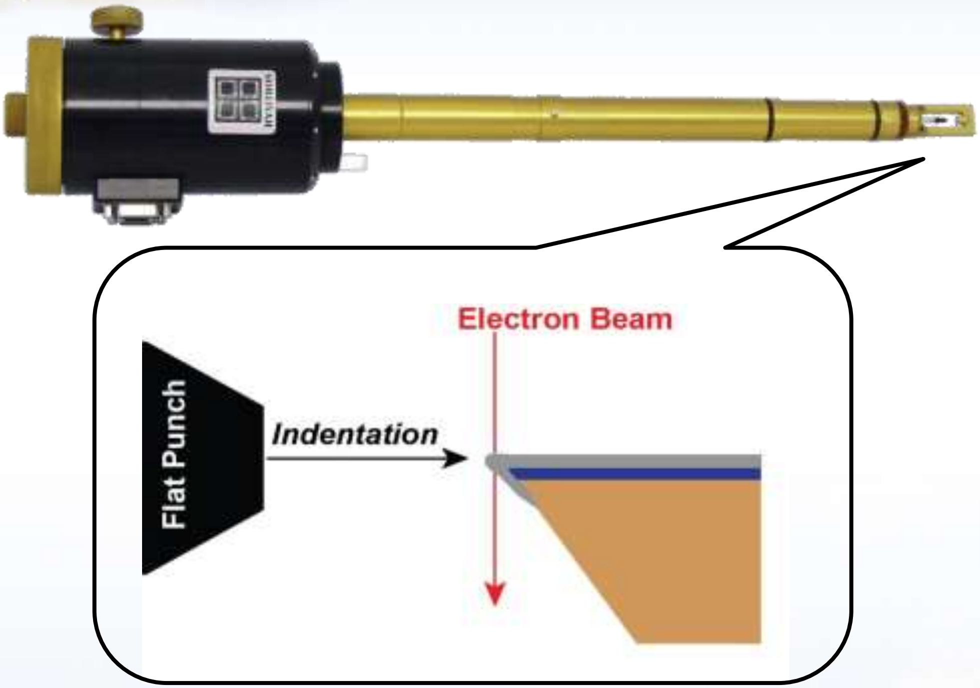
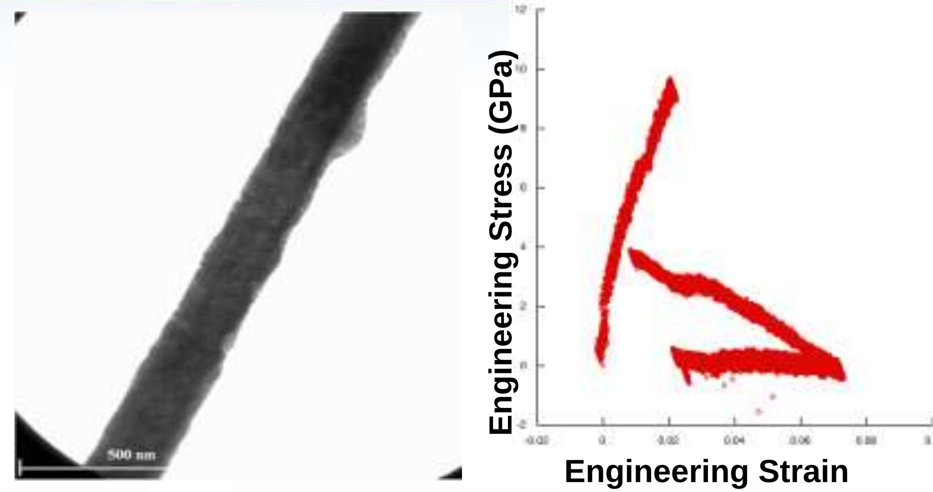
Collaborators: D. Masiel

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.





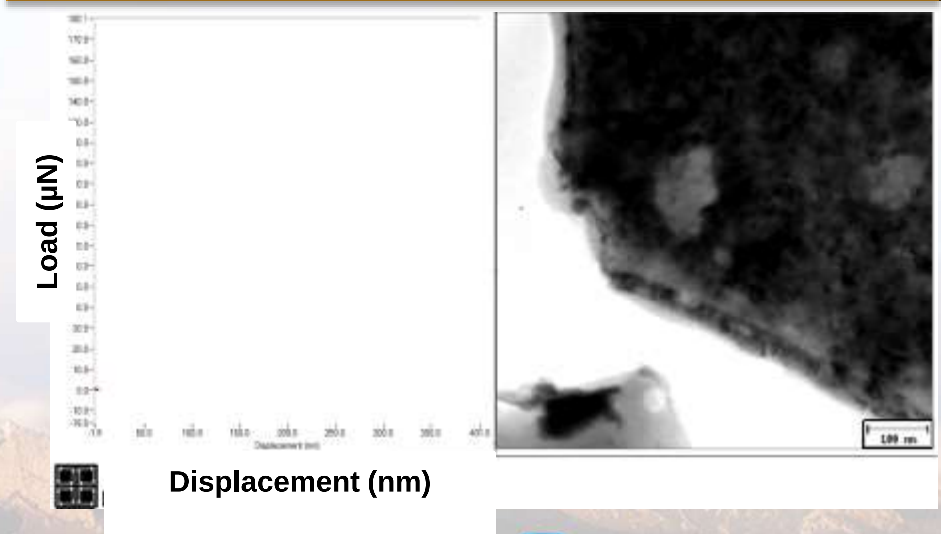
Displacement damage effect on mech. prop.

Radiation effect on mechanical properties

- Direct correlation of dose and defect density with resulting change in strength and ductility
- Failure of Mo-wire after 3 MeV Cu irradiation

Contact effect on structure

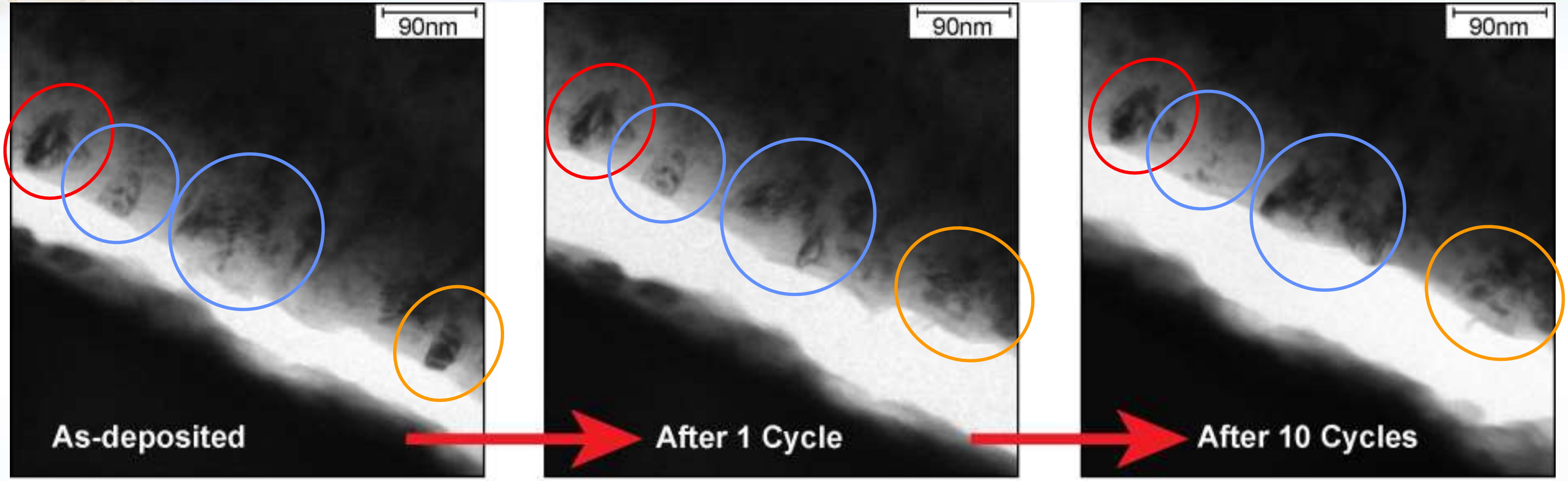
- Associate change in local hardness and fatigue with nanocrystalline Cu film structural evolution

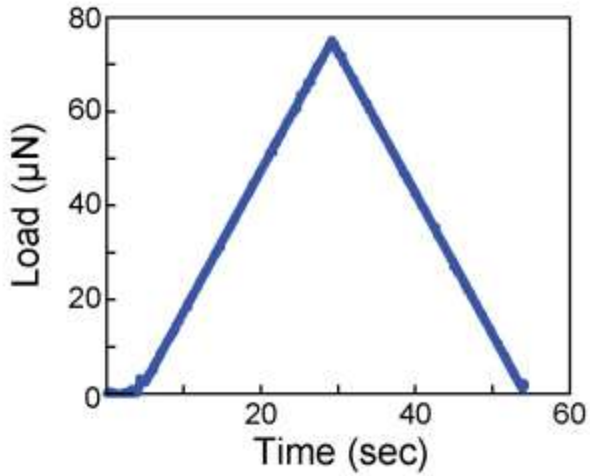


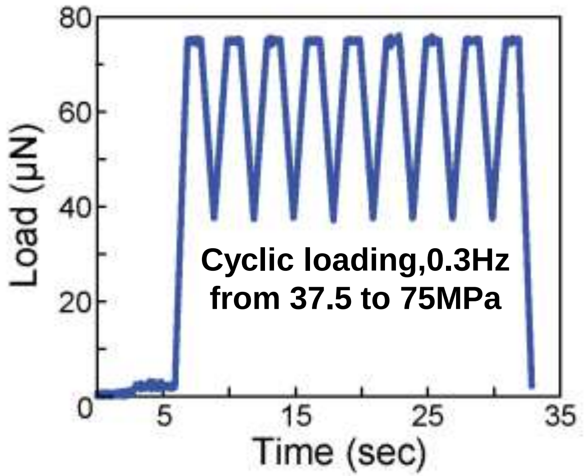
Fundamentals of contact reliability

No, Monotonic, or Cyclic Evolution

Contributors: J. Sharon, B. L. Boyce



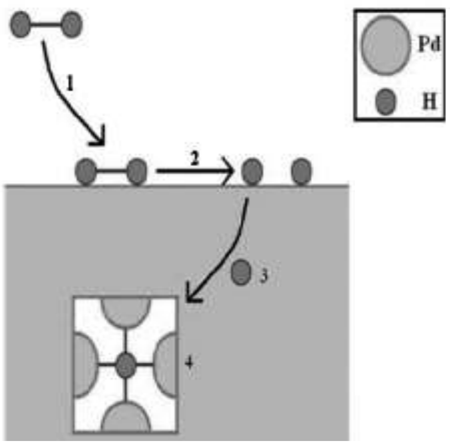




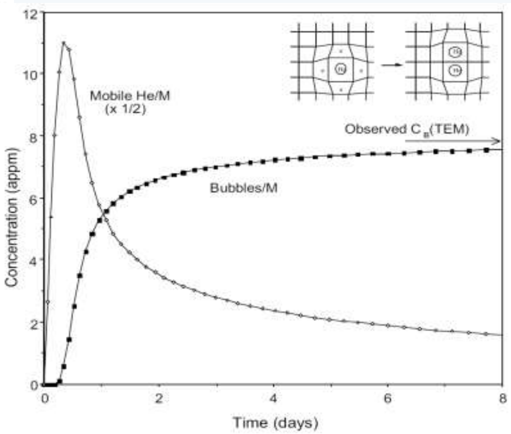
375 MPa nominal contact stress

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.

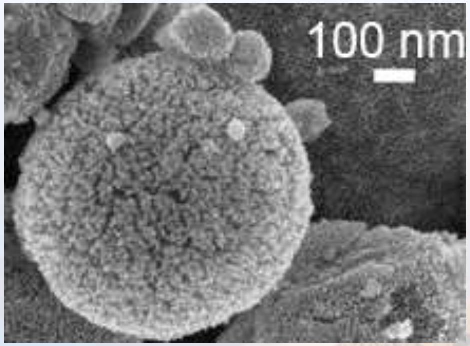


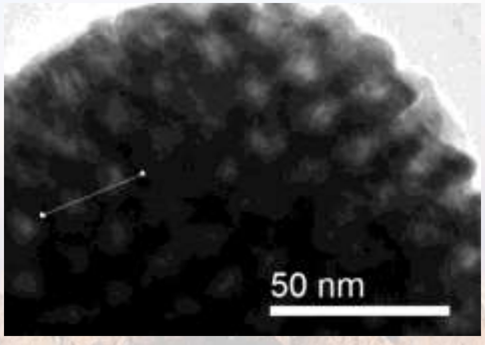
R. Delmelle, J., Phys. Chem. Chem. Phys. (2011) p.11412



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

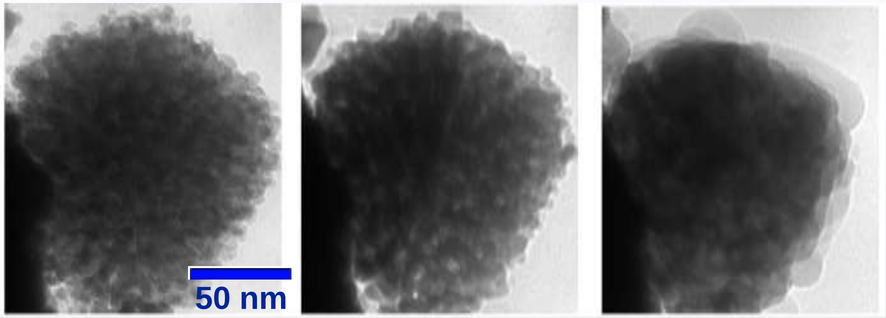




Vapor-Phase Heating TEM Stage

- 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

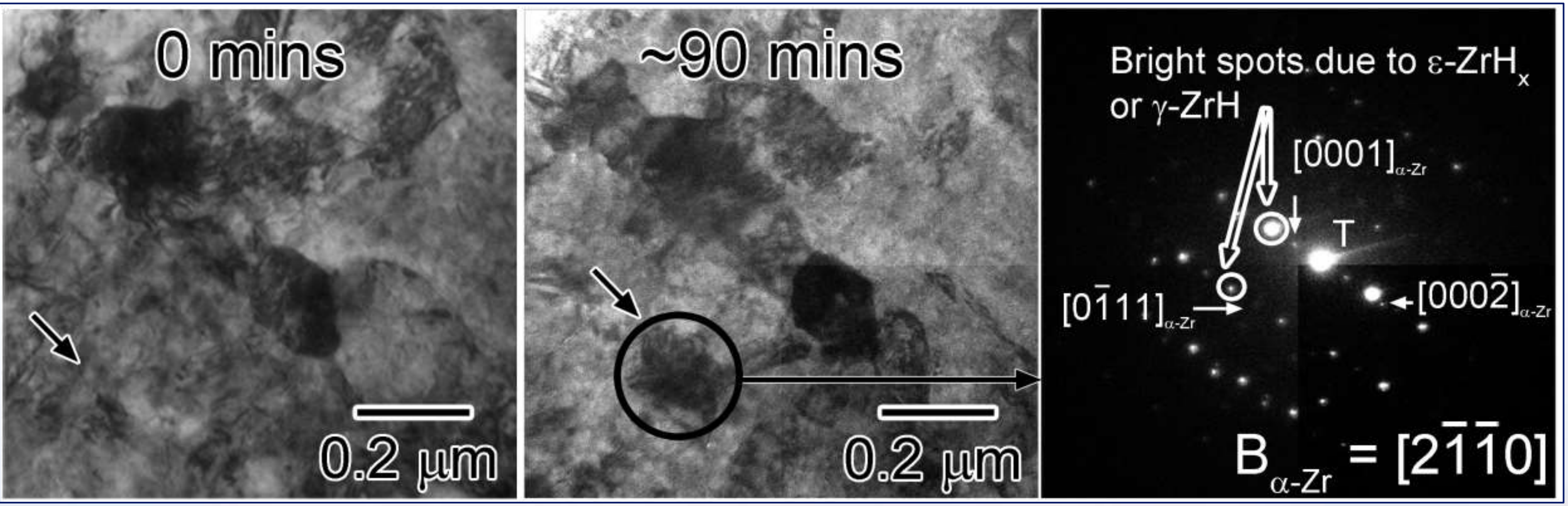
New *in situ* atmospheric heating experiments provide great insight into nanoporous Pd stability

In situ Observation of Hydride Formation in Zirlo

Collaborators: S. Rajasekhara and B.G. Clark

Absolute hydrogen pressure: 327 torr (~ 0.5 atm),

Ramp rate: 1 ^{o}C/s, Final temperature: ~ 400 C, Dwell time: ~ 90 mins



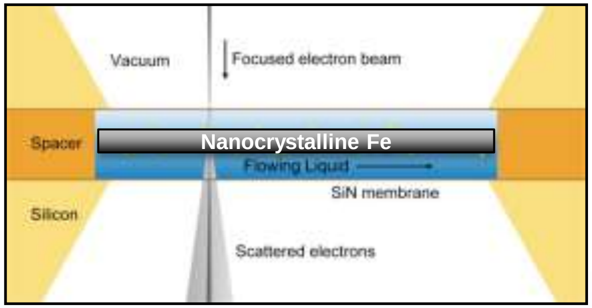
Hydride formation shown, for the first time by use of a novel TEM gas-cell stage, at elevated temperature and hydrogen pressure

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

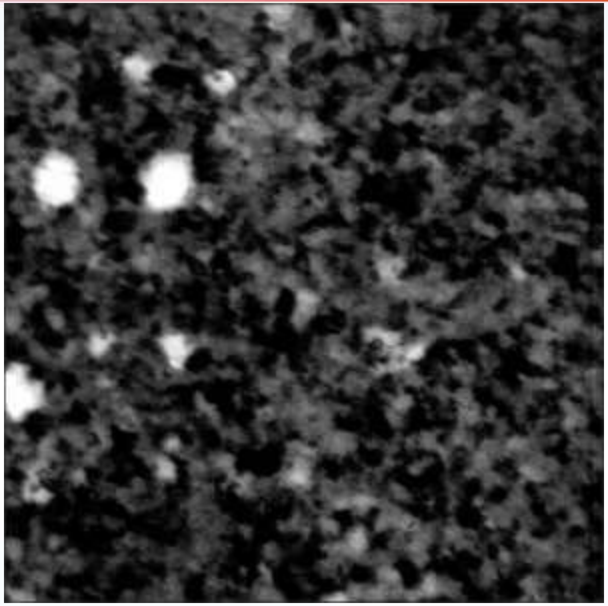
Contributors: D. Gross, J. Kacher, I.M. Robertson & Protochips, Inc.

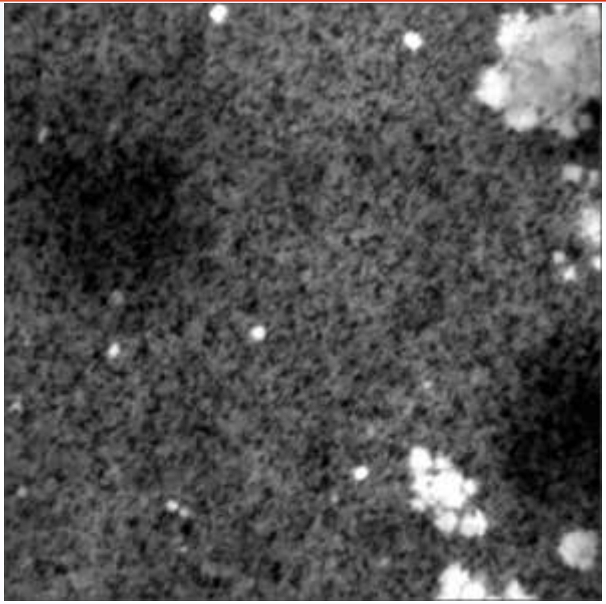
Microfluidic Stage

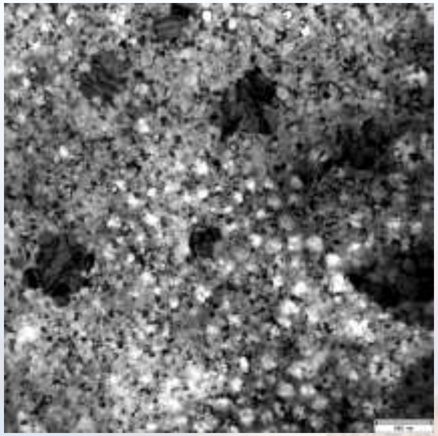
- Mixing of two or more channels
- Continuous observation of the reaction channel
- Chamber dimensions are controllable

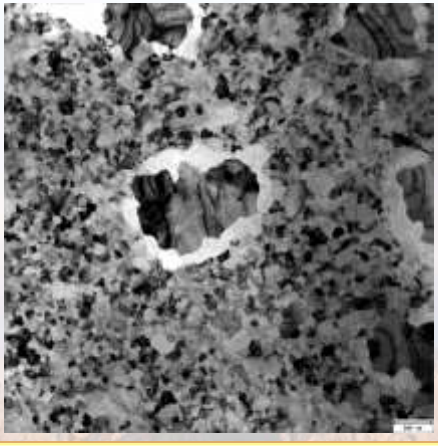


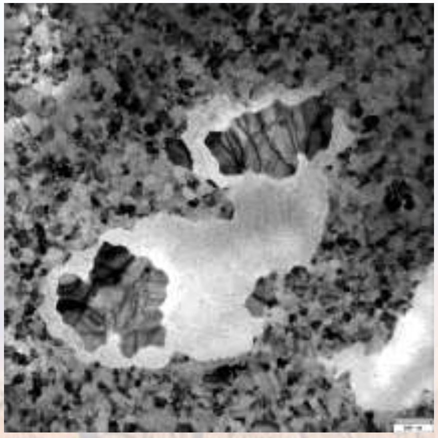
Cross-sectional schematic











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

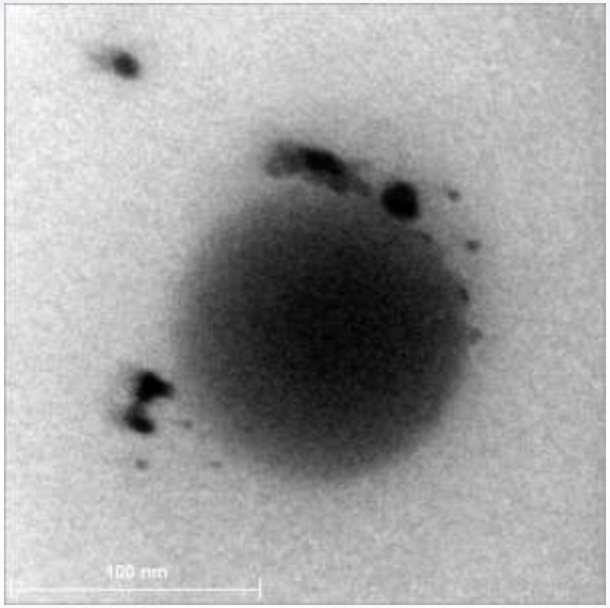
Large grains resulting from annealing appear more corrosion tolerant

Other Fun Uses of Microfluidic Cell

Protocell Drug Delivery

S. Hoppe,
E. Carnes,
J. Brinker

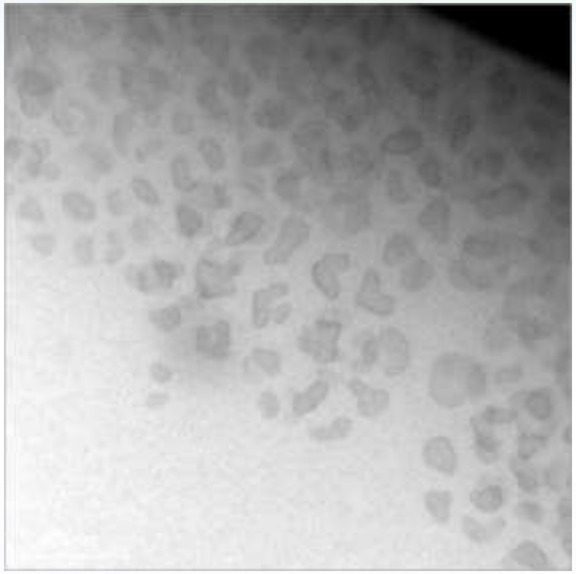
Liposome encapsulated Silica destroyed by the electron beam



BSA Crystallization

S. Hoppe

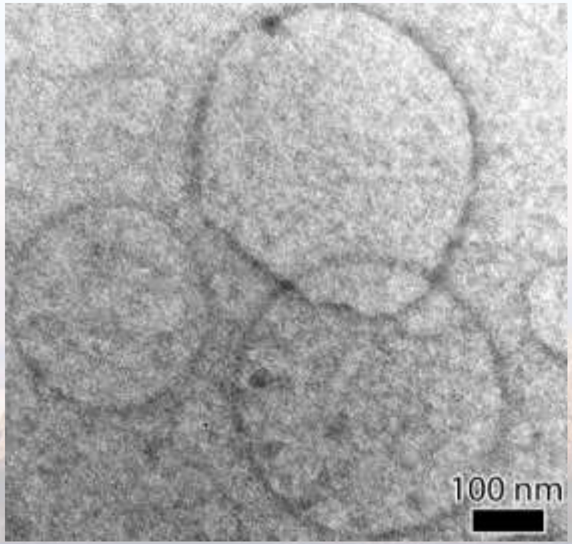
Crystallization of excess Bovine Serum Albumen during flow



Liposomes in Water

S. Hoppe,
D. Sasaki

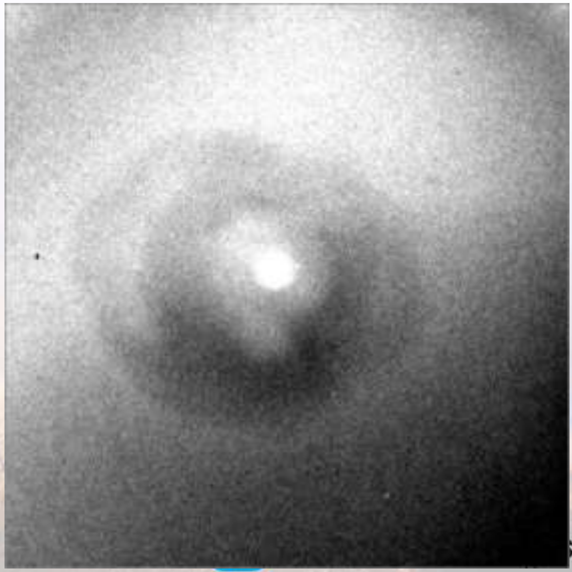
Liposomes imaged in flowing aqueous channel



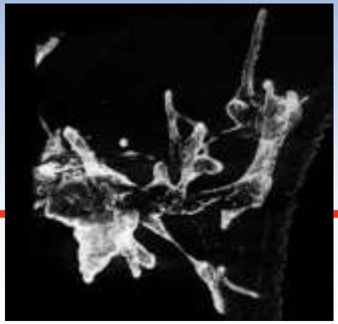
La Structure Formation

S. Hoppe,
T. Nenoff

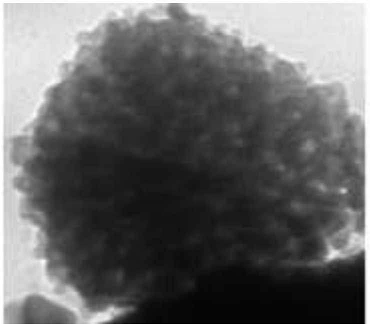
La Nanostructure form from $LaCl_3$ H_2O in wet cell due to beam effects

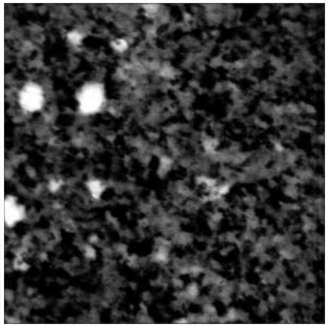


Summary



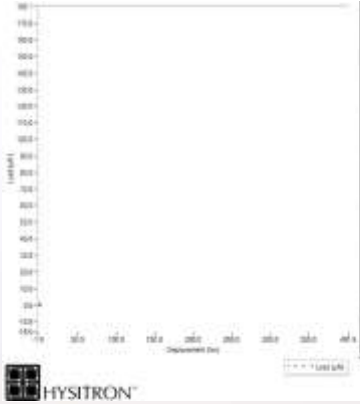
- Sandia's I[3]TEM is one of only two facilities in the US
  - Only facility in the world with a wealth of dual *in situ* capabilities
  - *In situ* irradiation from H to Au
  - *In situ* gas implantation
  - Eight TEM stages with various capabilities (two beta-testing)

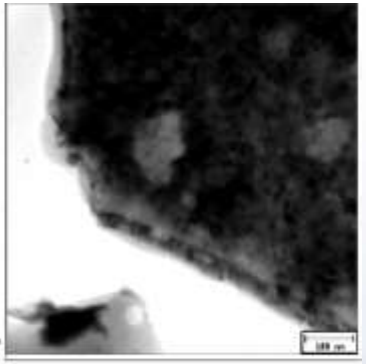




Future Interests

- Apply the current I[3]TEM capabilities to various material systems of interest to internal and external customers
- Expand the I[3]TEM capability to include
  - Nano to microsecond imaging
  - Laser heating and/or shock to the sample
  - Addition of precession electron diffraction





Sandia's I[3]TEM although still under development is providing a wealth of interesting initial observations

Contributors & Collaborators:

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