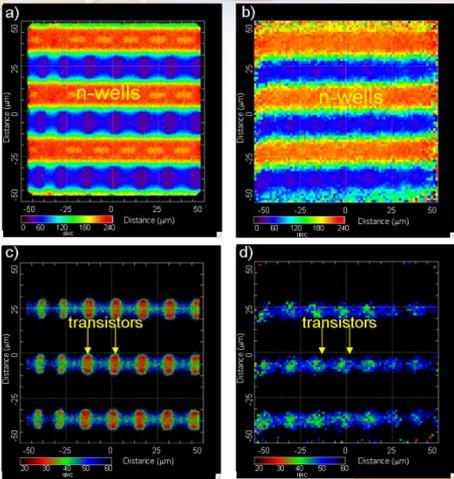


# Sandia's Ion Beam Laboratory



Ion Beam Analysis (IBA)

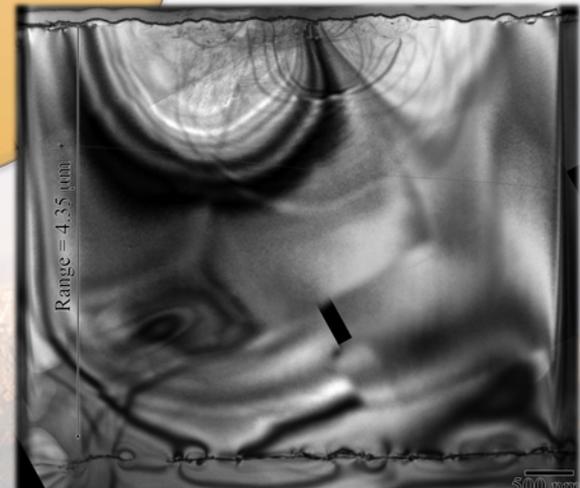
TECHNIQUE	SCHEMATIC	EXAMPLE ANALYSIS SPECTRUM
RBS		
ERD		
HIBS		
NRA		
PIXE		
SEU/IBICC		

Radiation Effects Microscopy (REM)



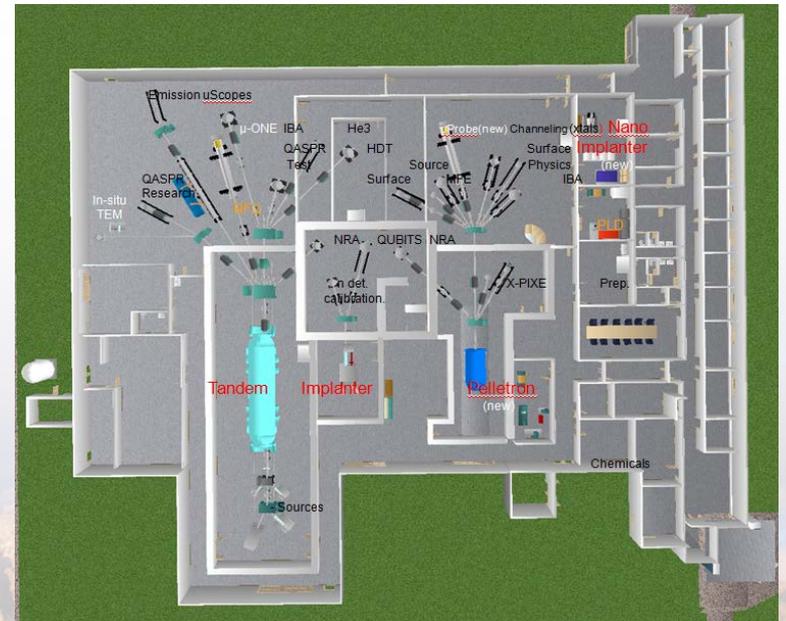
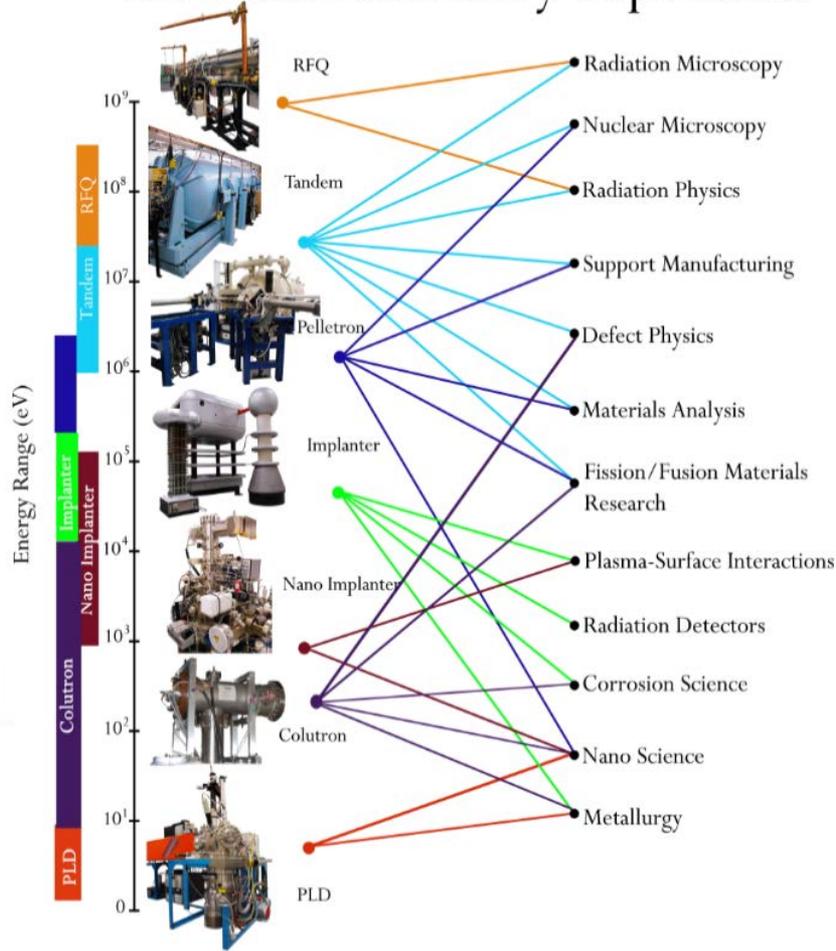
Ion Beam Modification (IBM)

*In situ* Ion Irradiation Transmission Electron Microscopy (I<sup>3</sup>TEM)



# Overview

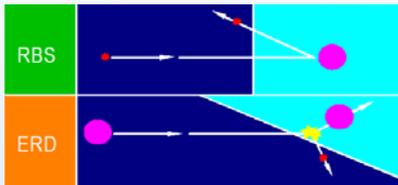
## Ion Beam Laboratory Capabilities



# Overview

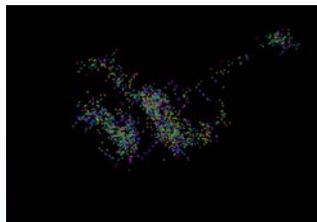
## Ion Beam Analysis

- *The basic concept:*  
A charged particle interacts with a material and one of a variety of signals produced gives information on the local chemistry and structure
- Each beam has associated benefits and limitations

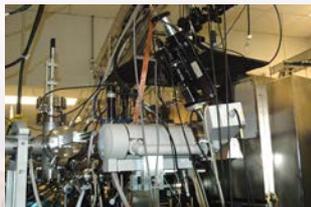


## Radiation Effects Testing

- Space and other nuclear environments demand radiation hardness
- Can simulate radiation damage from a single ion strike to up to 500 dpa

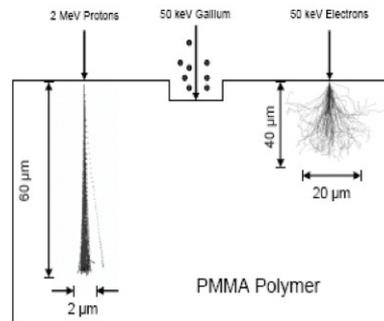


Blistering in CuNb multilayers



## Ion Beam Modification

- *The basic concept:*  
Alteration of the structure through ion beam interactions
  - Implantation of dopants
  - Sputtering of material
  - Decomposition of gasses



<http://www.pbeam.com>

## *In-situ* Ion Irradiation Transmission Electron Microscopy

- *The basic concept:*  
Characterization of materials exposed to various types of particle bombardment in real time, at the nanoscale.
- Current capabilities include
  - Heating
  - Straining
  - Tomography



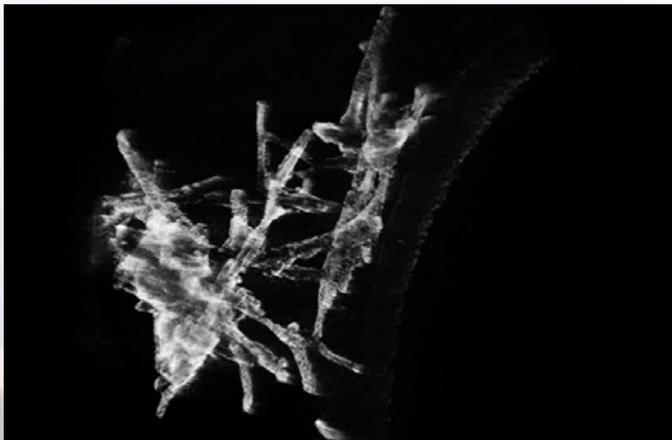
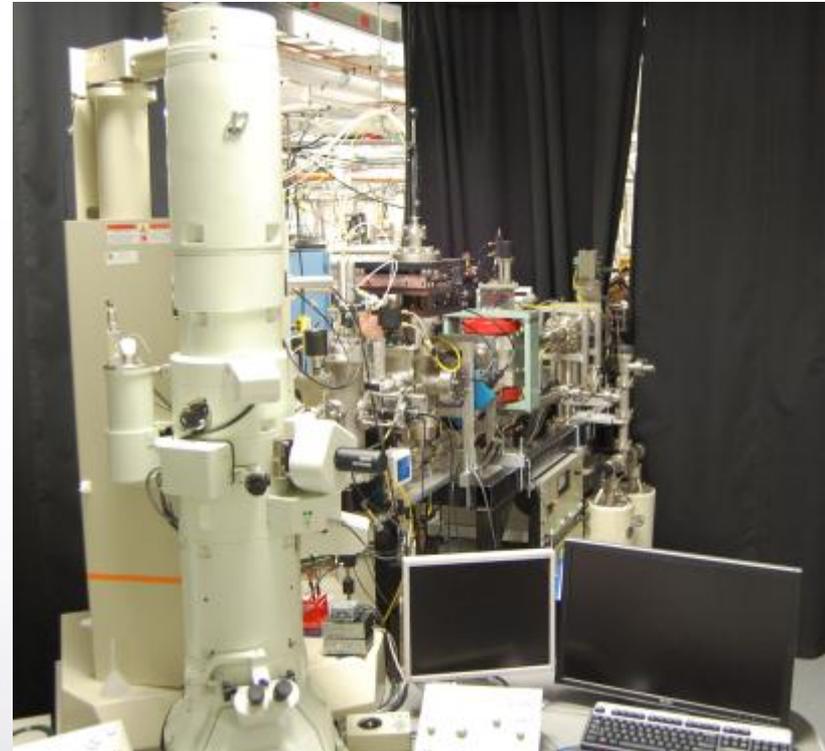
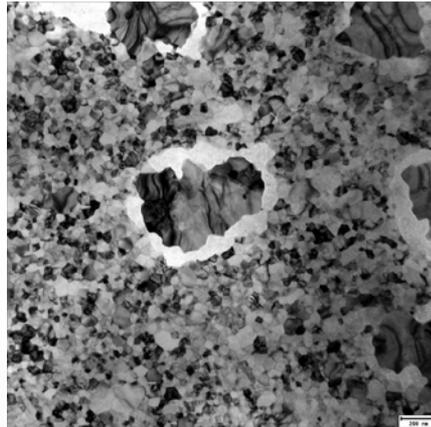
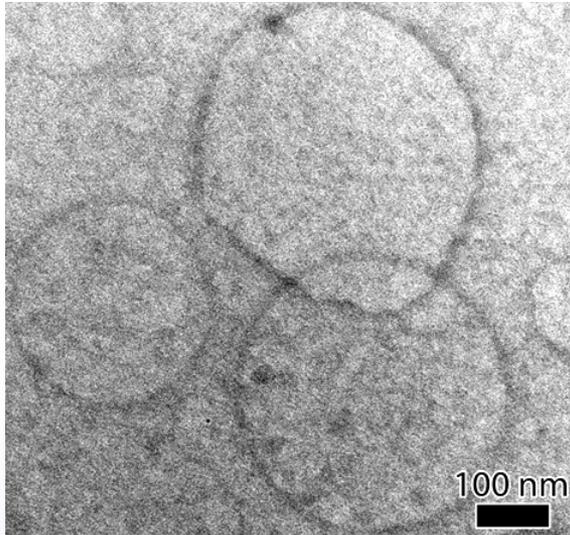
IBA, radiation effect testing, and ion beam modification are all widely used in research and industry



Sandia National Laboratories

# *In Situ* Transmission Electron Microscopy (I<sup>3</sup>TEM)

Characterization of materials exposed to various types of particle bombardment in real time, at the nano scale



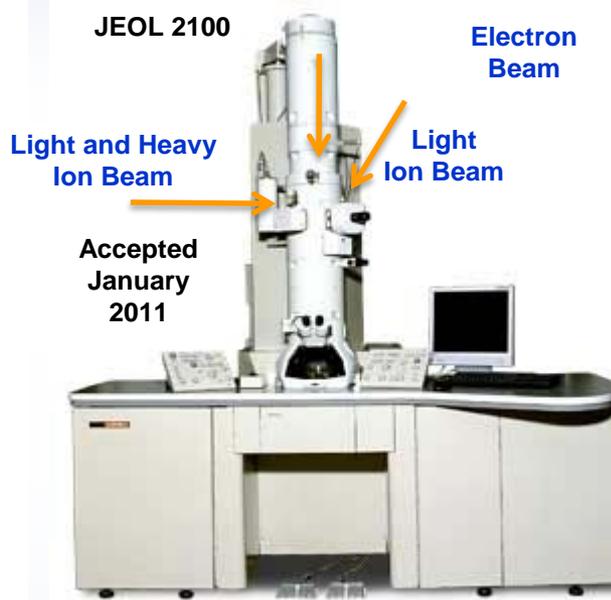
The IBL is one of only 11 facilities *worldwide* with this capability



# In situ Ion Irradiation TEM (I<sup>3</sup>TEM)

## Proposed Capabilities

- 200 kV LaB<sub>6</sub> TEM
- Ion beams considered:
  - 1 MeV H<sup>1+</sup>
  - 3 MeV He<sup>1+</sup> Si<sup>3+</sup> Cu<sup>3+</sup> Au<sup>3+</sup>W<sup>3+</sup>
- 14 MeV Si<sup>3+</sup>
- 10 keV D<sup>2+</sup>
- 10 keV He<sup>+</sup>
- 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

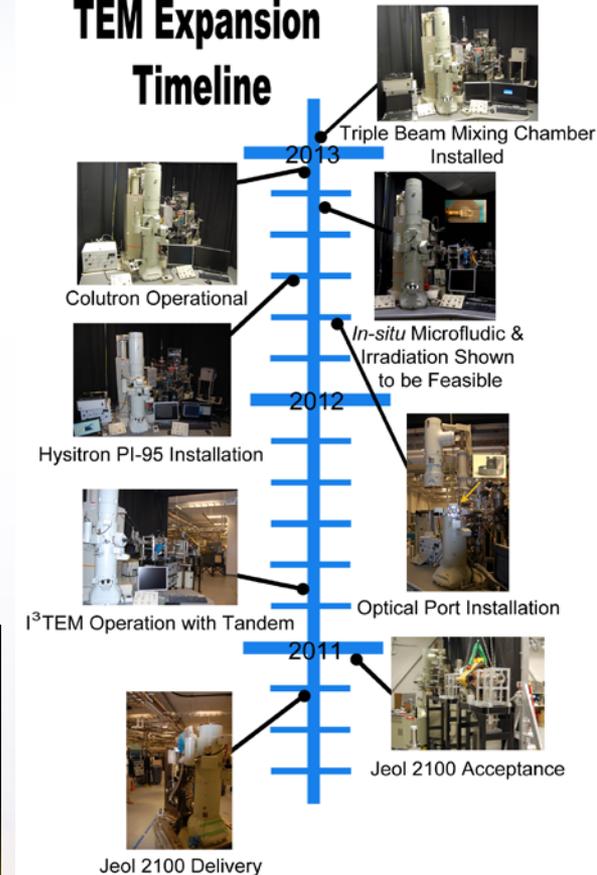


TVIPS



*In situ* IBIL

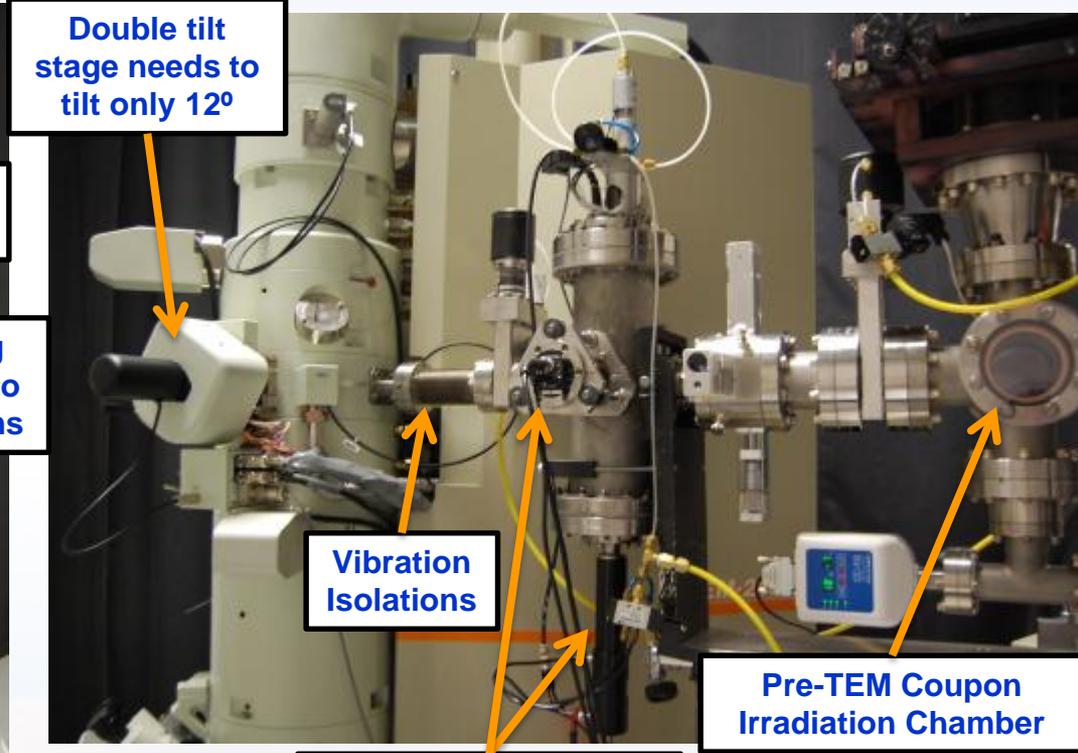
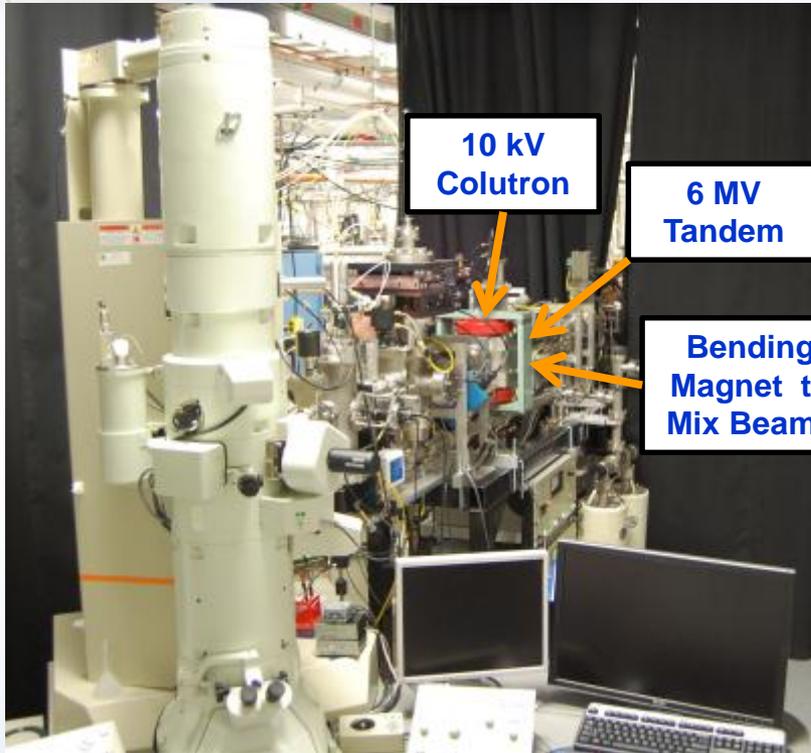
## TEM Expansion Timeline



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

**Many potential additions are being considered**

# Current Status of the *In situ* TEM Beamline



Beam burn from 14 MeV Si

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

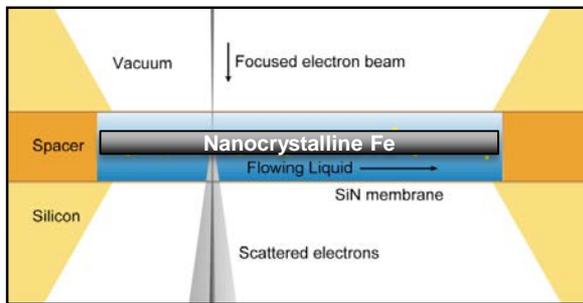
**Microfluidic Holder Beam Burn**



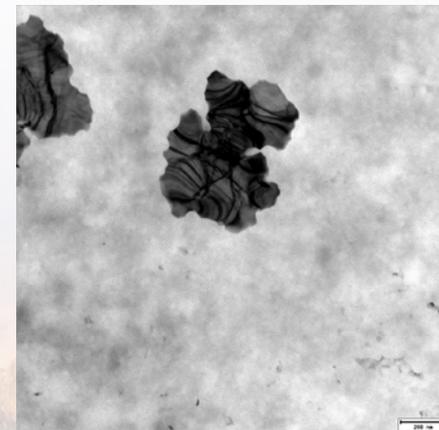
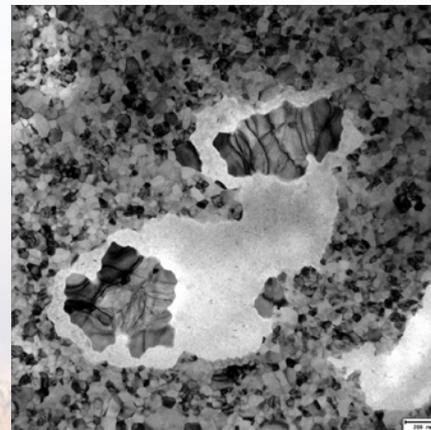
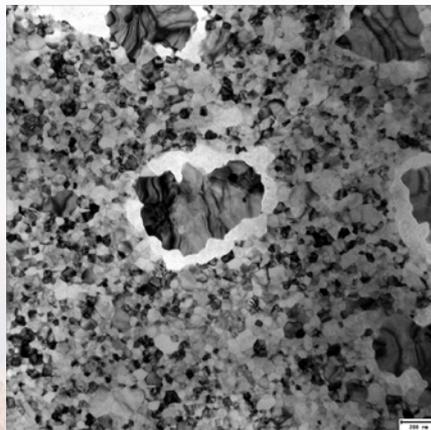
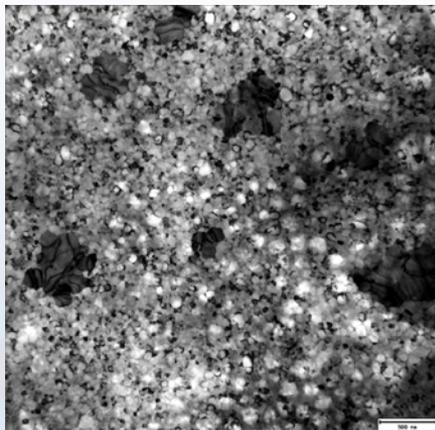
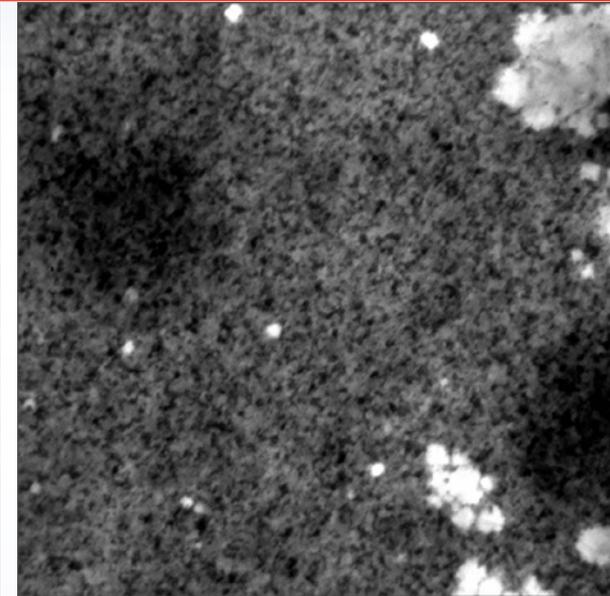
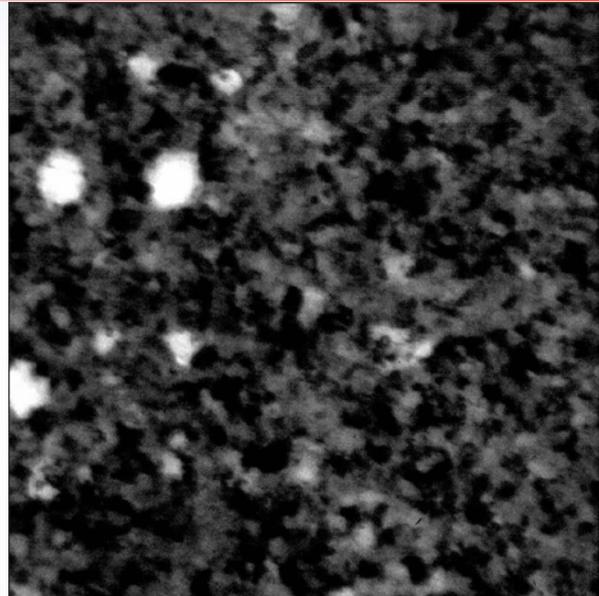
# Can We Gain Insight into the Corrosion Process through *In situ* TEM?

## Microfluidic Stage

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



Cross-sectional schematic



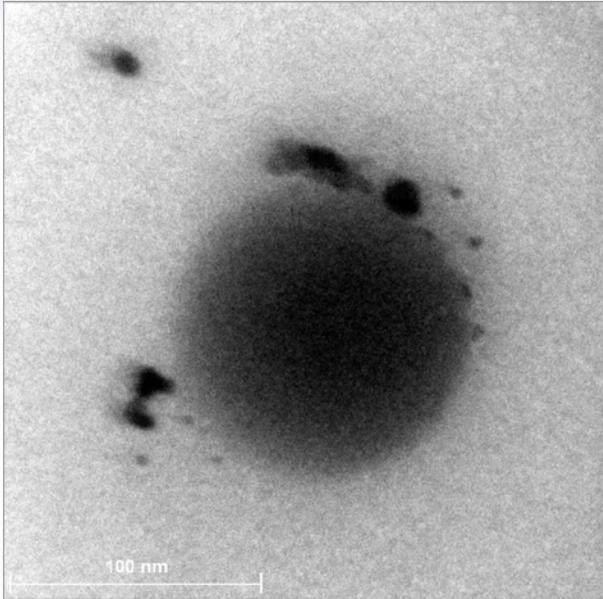
**Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.  
Large grains resulting from annealing appear more corrosion tolerant**

# Other Fun Uses of Microfluidic Cell

## Protocell Drug Delivery

S. Hoppe,  
E. Carnes,  
J. Brinker

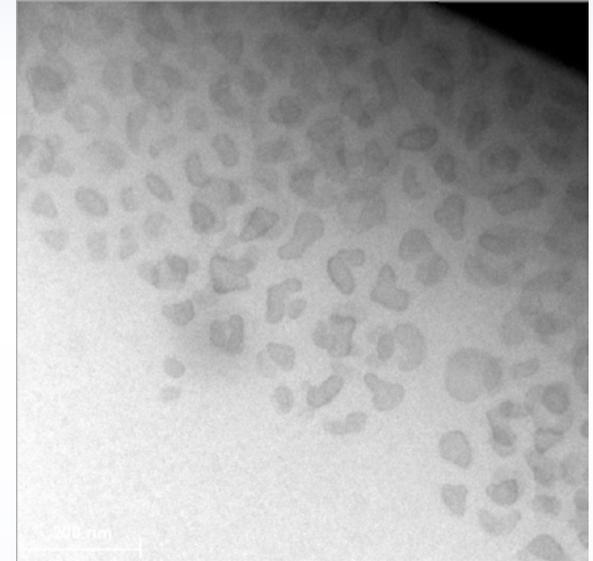
Liposome  
encapsulated  
Silica destroyed  
by the electron  
beam



## BSA Crystallization

S. Hoppe

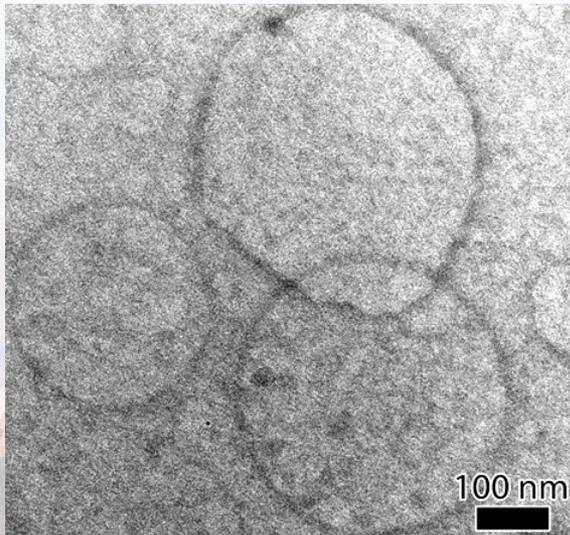
Crystallization of  
excess Bovine Serum  
Albumen during flow



## Liposomes in Water

S. Hoppe,  
D. Sasaki

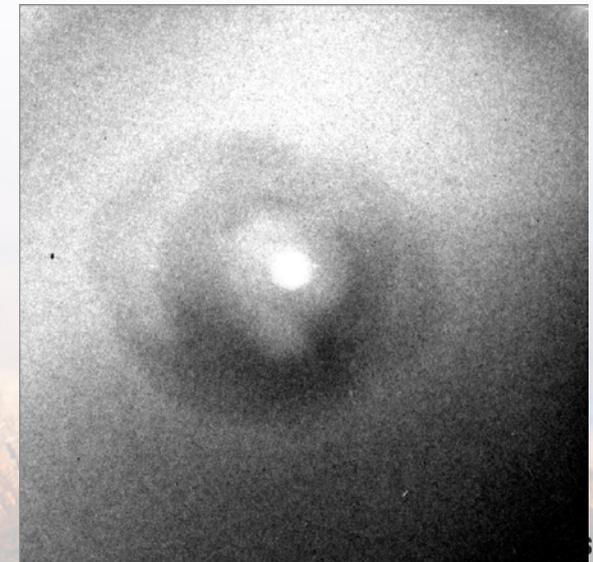
Liposomes  
imaged in  
flowing aqueous  
channel



## La Structure Formation

S. Hoppe,  
T. Nenoff

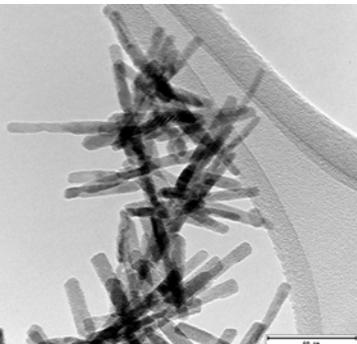
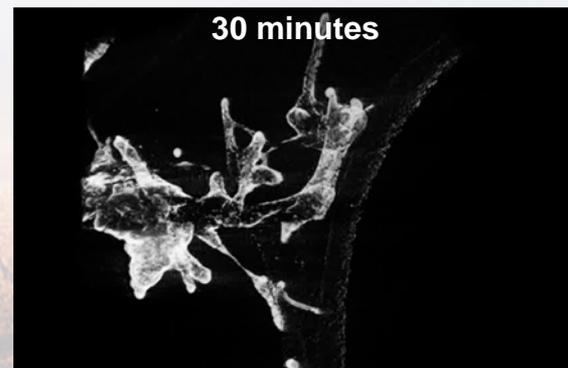
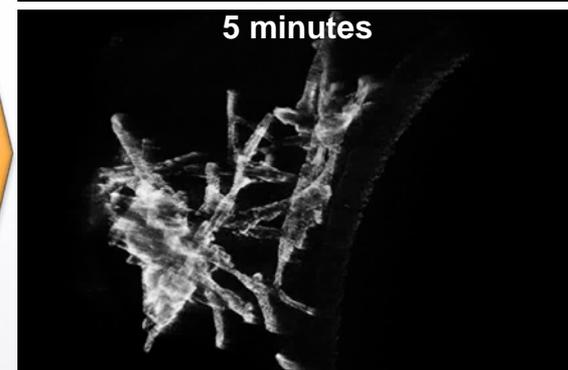
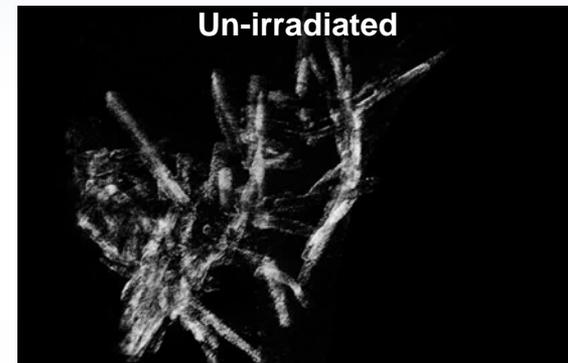
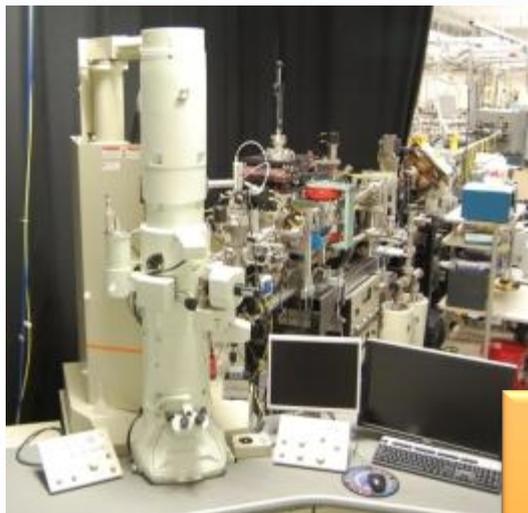
La  
Nanostructure  
form from  $\text{LaCl}_3$   
 $\text{H}_2\text{O}$  in wet cell  
due to beam  
effects



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



## In situ Ion Irradiation TEM (I<sup>3</sup>TEM)



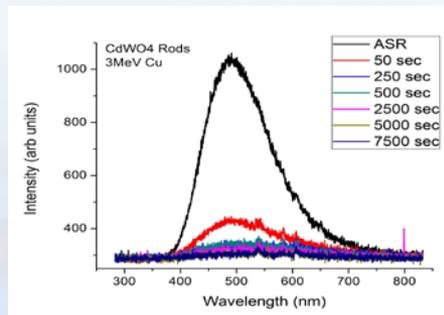
High-Z nanoparticles (CdWO<sub>4</sub>) are promising, but are radiation sensitive



Hummingbird tomography stage



Tomography of Irradiated CdWO<sub>4</sub>:  
3 MeV Cu<sup>3+</sup> at ~30 nA



First IBIL in TEM



# In situ TEM Quantitative Mechanical Testing



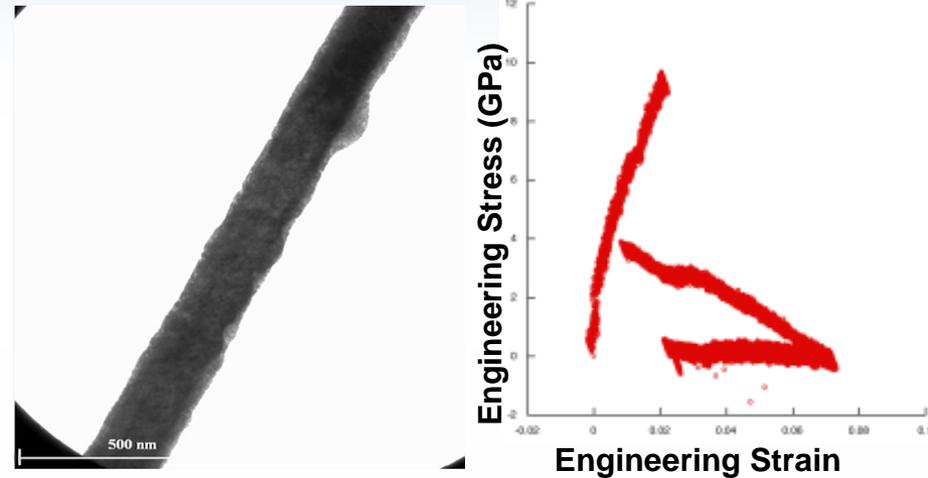
Hysitron Stage

## 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

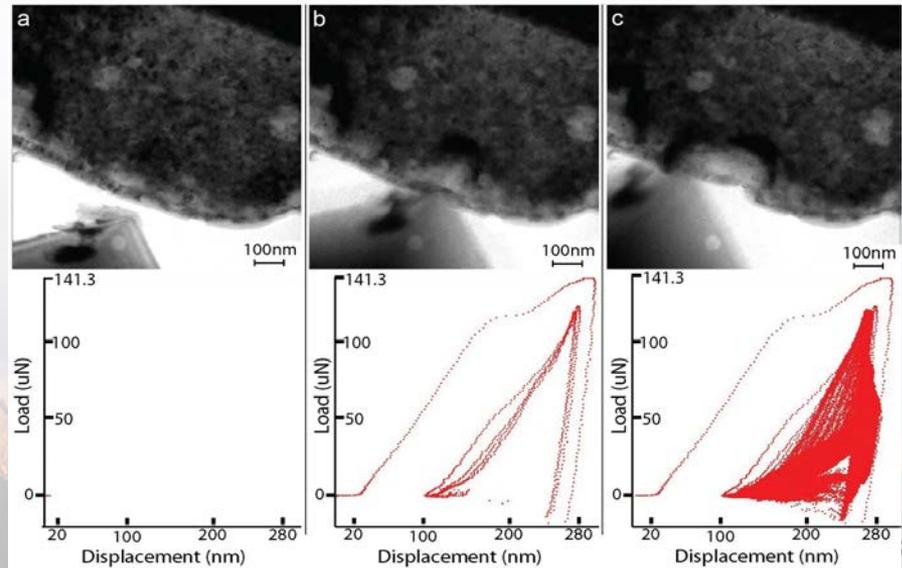
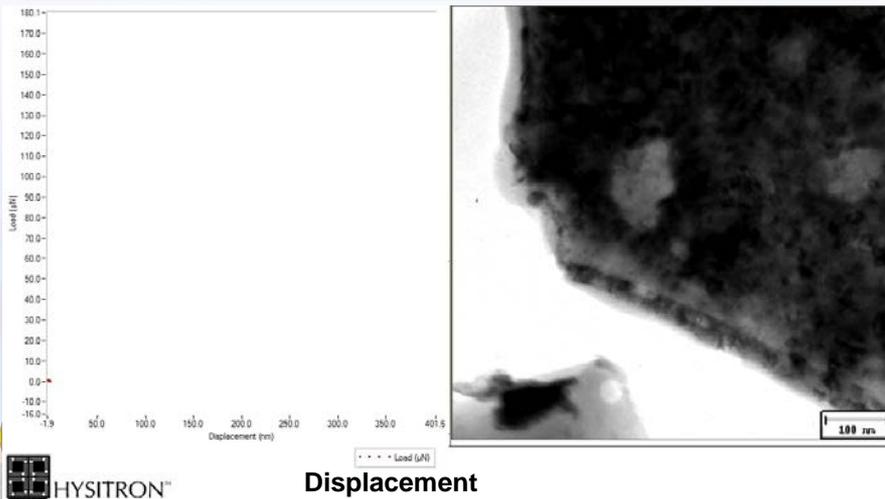
## Contact and fatigue effect on structure

- Associate change in local hardness and fatigue with corresponding nanostructure
- Indent and Fatigue of nanocrystalline Cu film

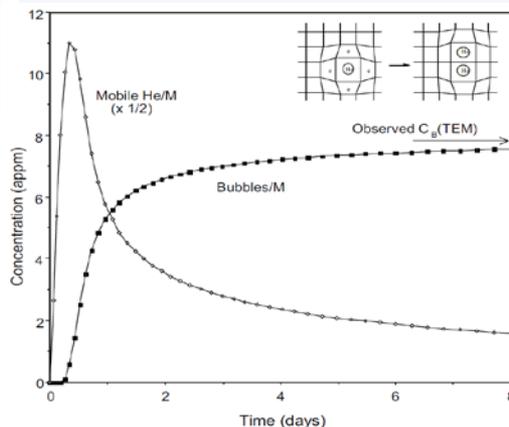
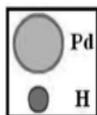
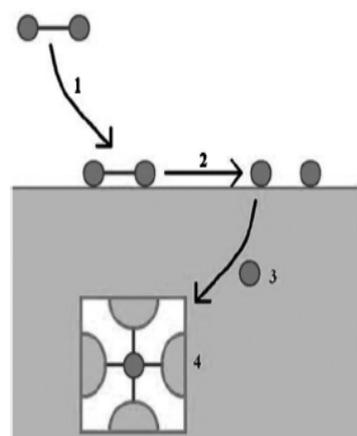


Displacement damage effect on mech. prop.

## Fundamentals of contact reliability



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



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

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

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

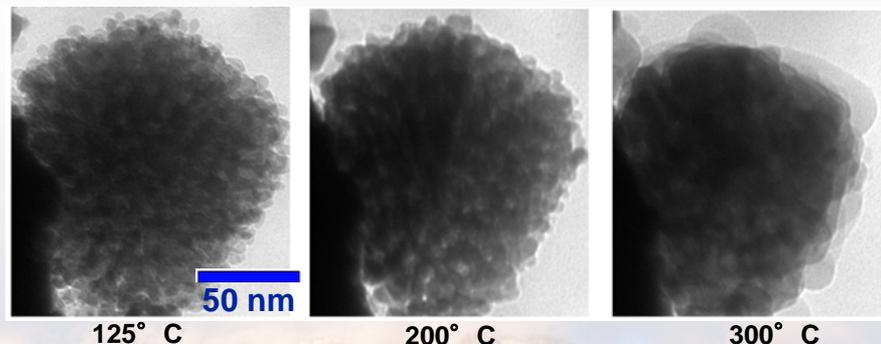
Thiebaut, S. et al. JNM (2000) p. 217

## 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<sub>2</sub> after several pulses to specified temp.

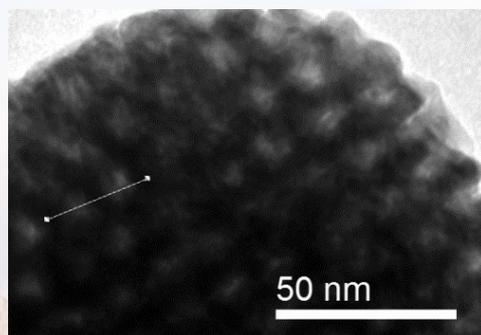
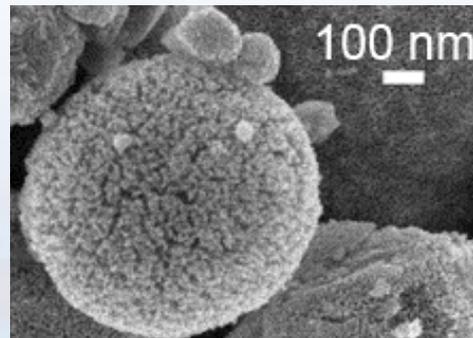


125° C

200° C

300° C

## Harmful effects may be mitigated in nanoporous Pd



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

