In situ Ion Irradiation TEM at Sandia's IBL

SAND2012-0648 C

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K. Hattar, B.G. Clark, J. Custer

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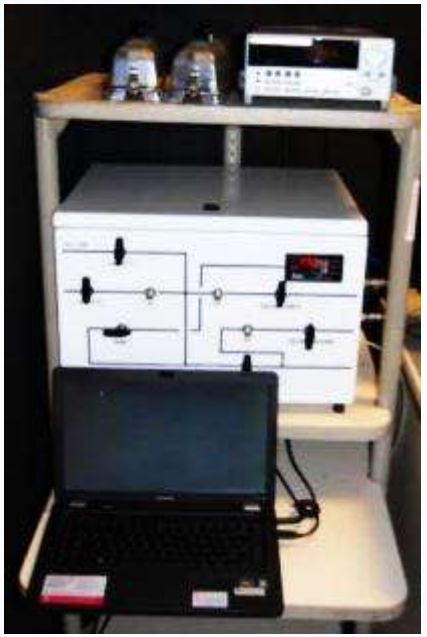
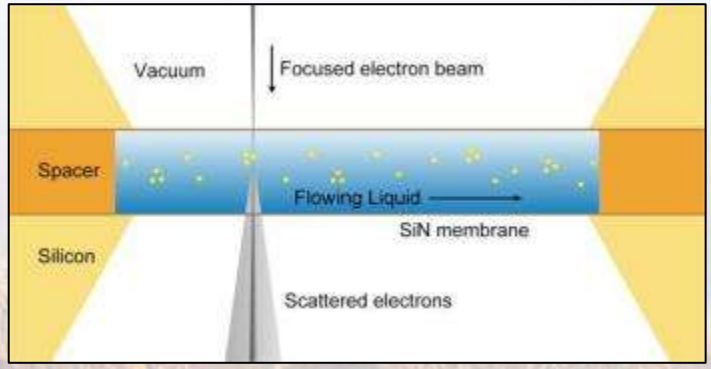


Ion Beam Lab (IBL)

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



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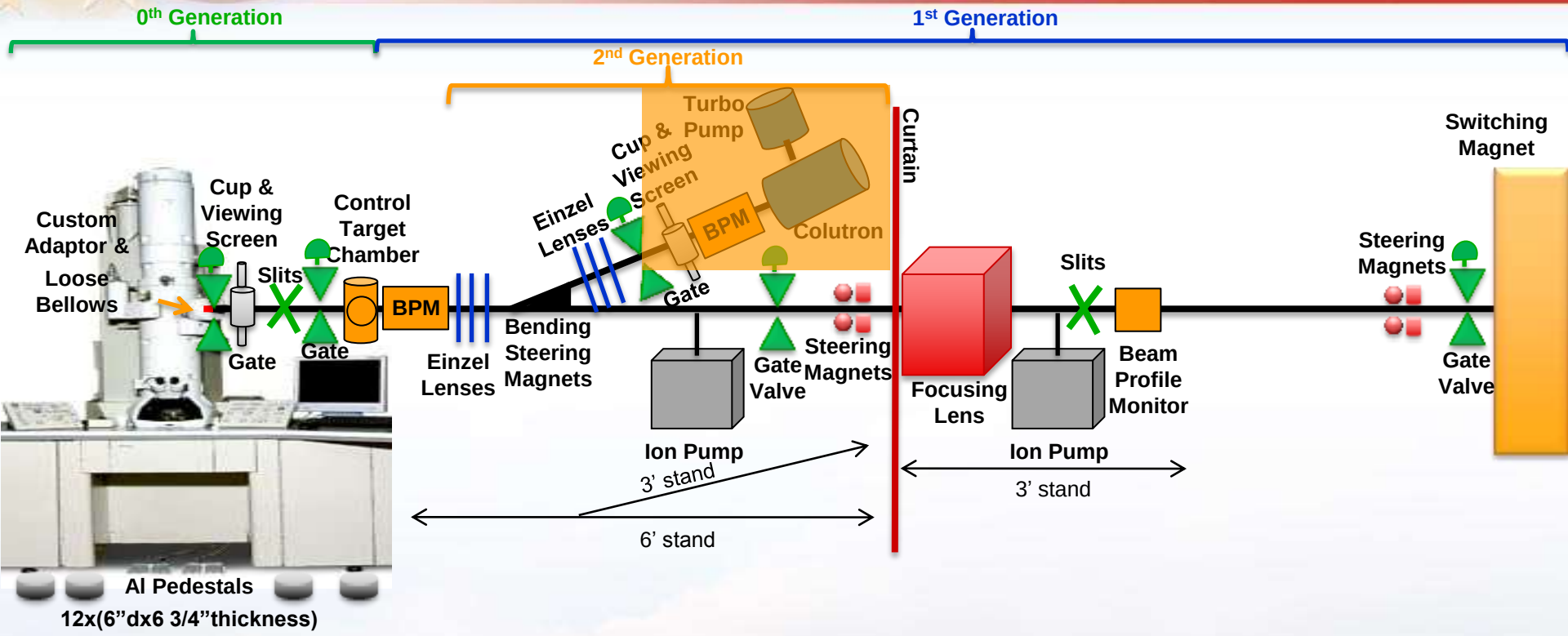
Current Status of the *In situ* TEM Beamline

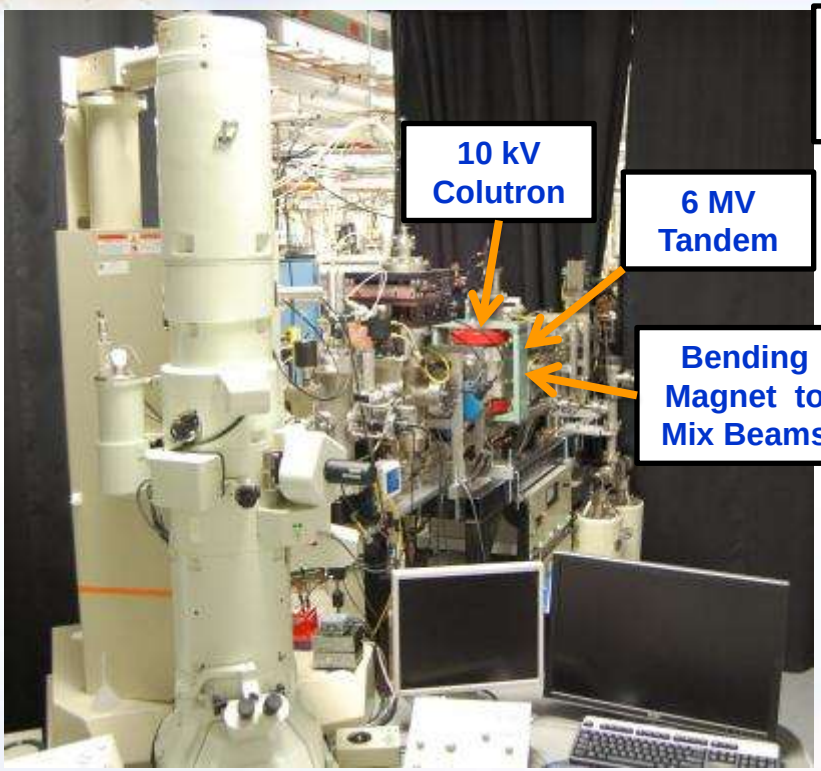


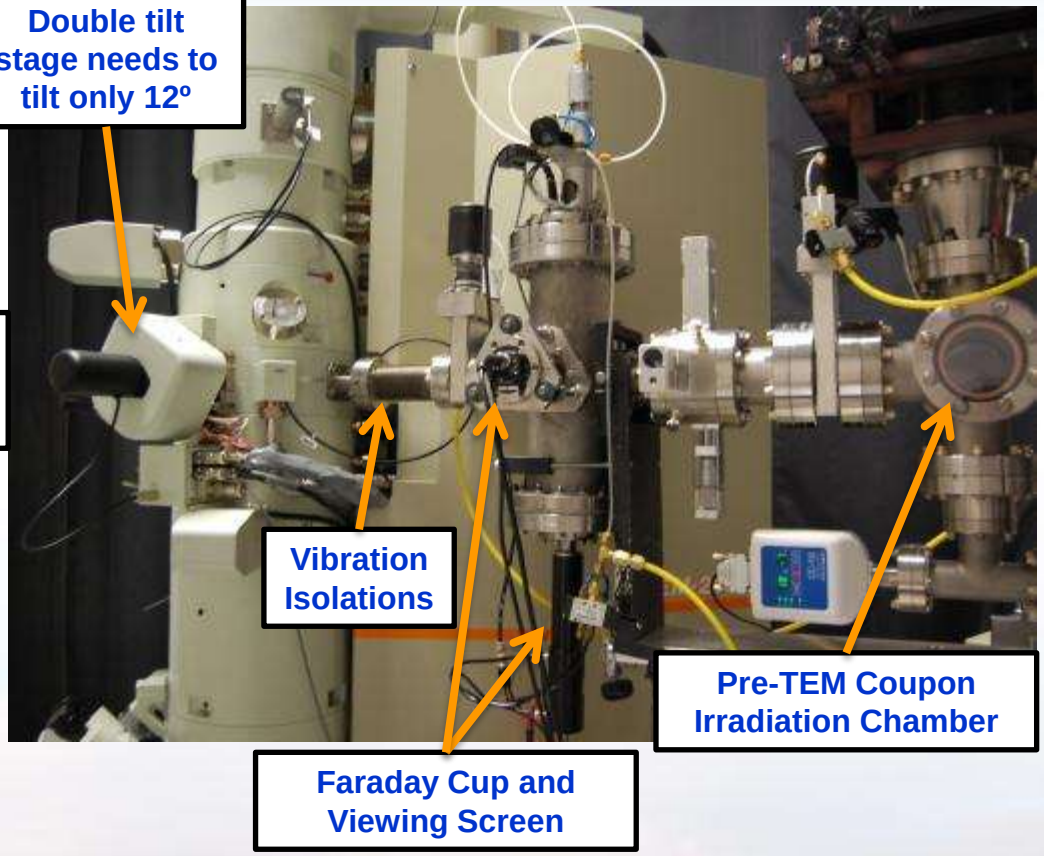
0th and 1st Generation are completed and operated regularly

2nd Generation was assembled and placed under vacuum on 12/23/2011

We hope to have concurrent heavy and light ion irradiation facility operational in 2012

Current Status of the *In situ* TEM Beamline





Beam burn from
14 MeV Si

0^{th} and 1^{st} Generation are completed and operated regularly

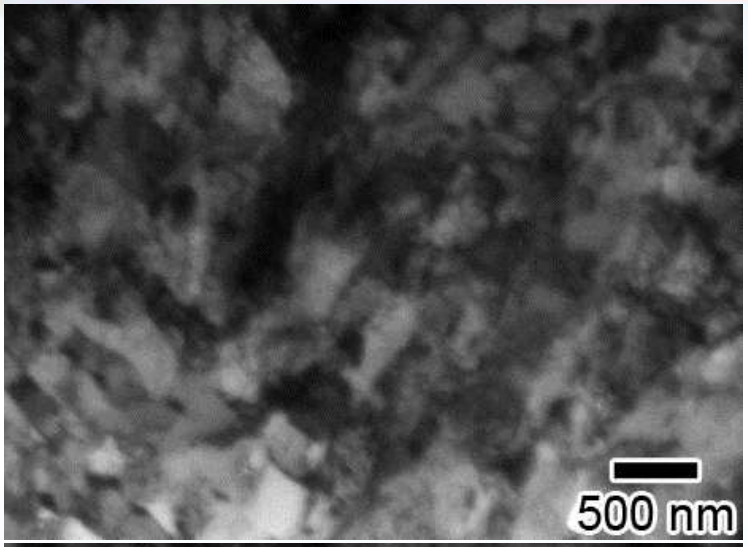
2^{nd} Generation was assembled and placed under vacuum on 12/23/2011

We hope to have concurrent heavy and light ion irradiation facility operational in 2012

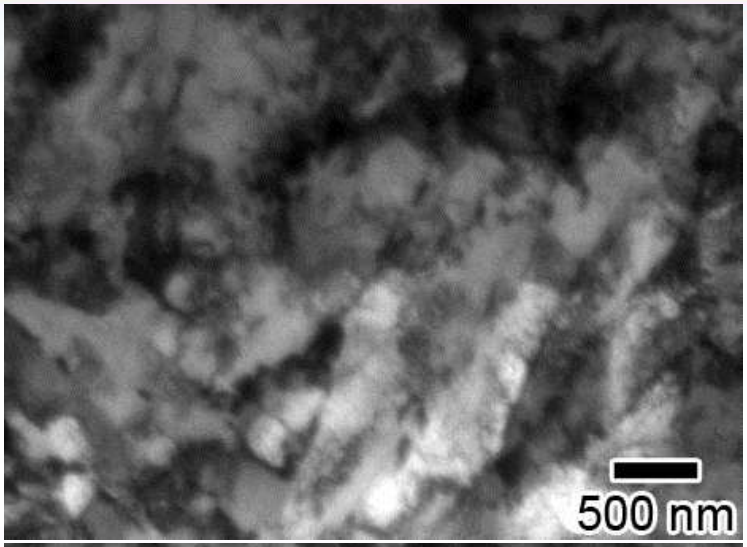
In situ TEM Ion Irradiation

HT9 3 MeV Cu^{3+} at ~10 nA RT

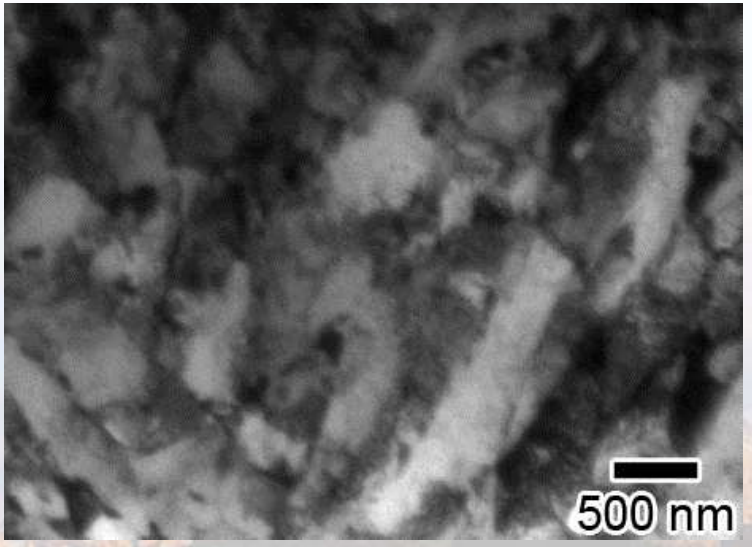
Initial



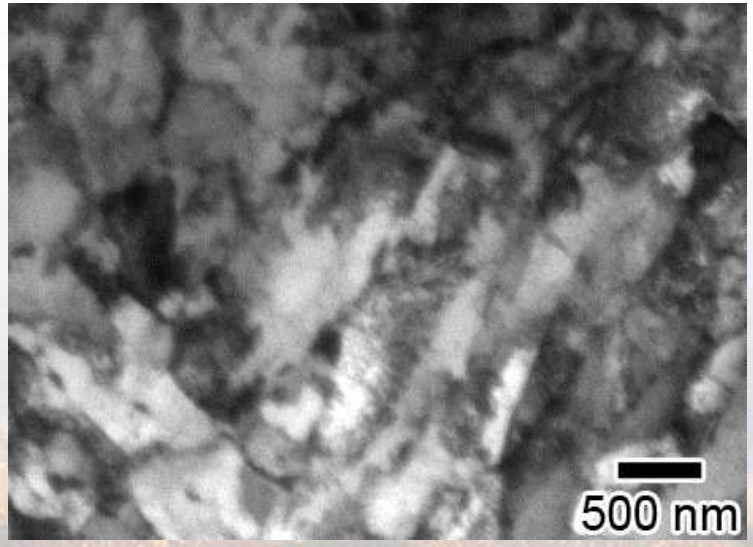
2 hrs



3 hrs



6 hrs



These initial studies show the I³TEM facility is operational, but sample prep and imaging conditions were not optimal

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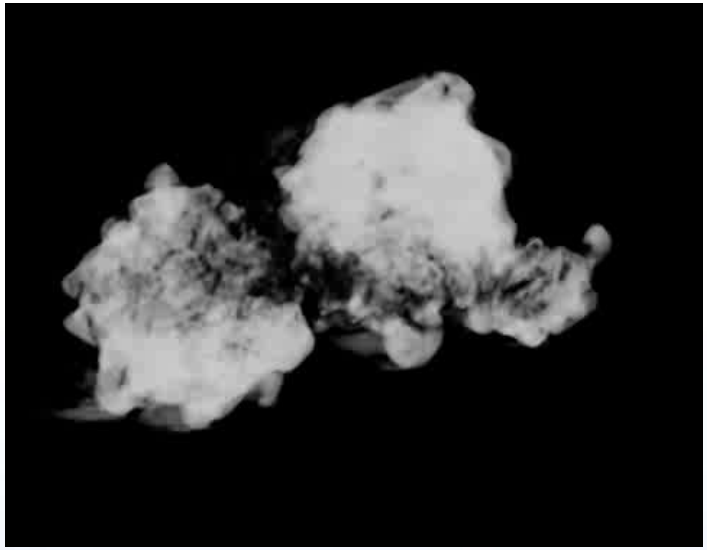
Aiming Towards 4D Irradiation Studies

Tomography of Pd NP

In collaboration with: B. Yates, J.Villone, D. Robinson

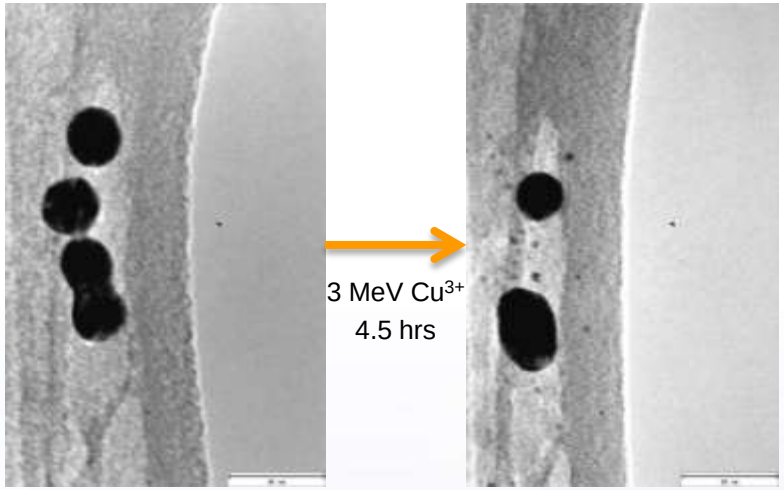
Porous Pd nanoparticles reconstructed in 3D

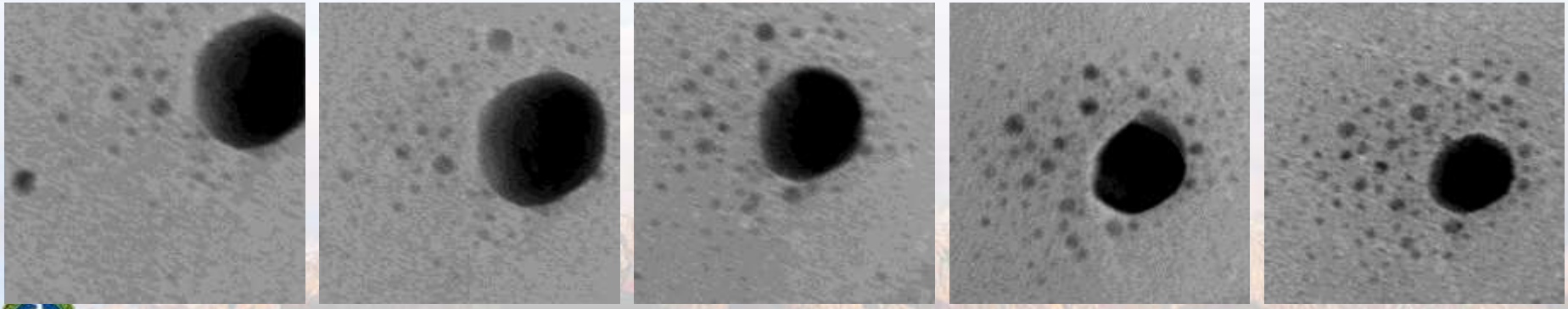


Ion Irradiation of Au NP

In collaboration with: S. Hoppe, S. Rajasekhara

Sintering and sputtering of Au nanoparticles during ion irradiation





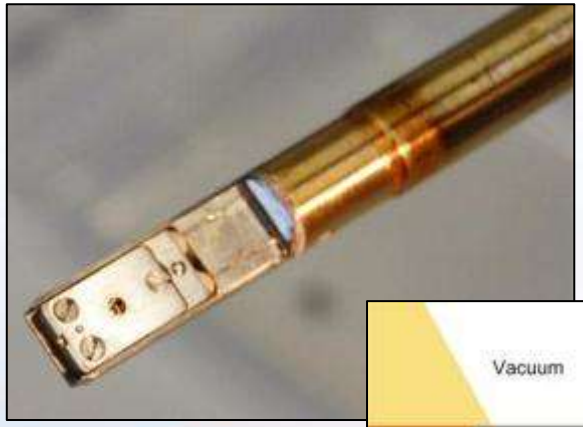
The combination of new capabilities permit better understanding at the nanoscale of systems in real environments

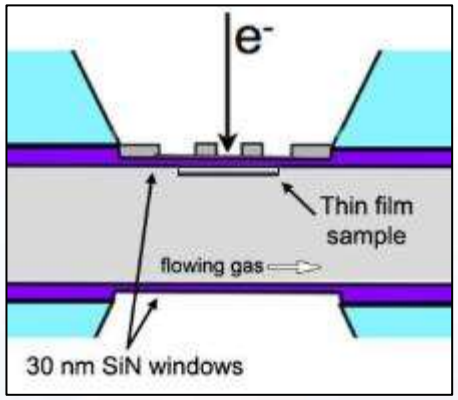
Gas/Vapor and Liquid Stages

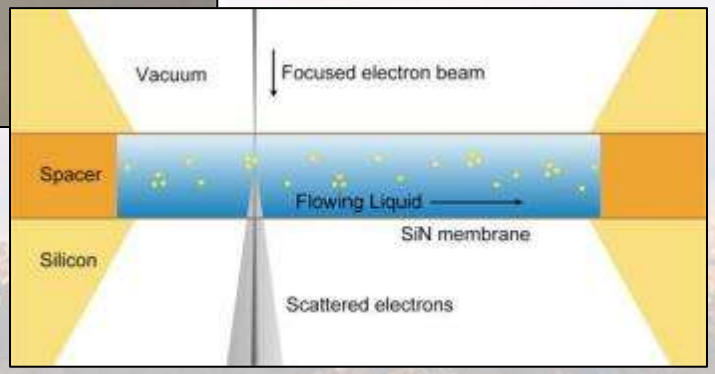
Samples must fit within the limited space of the cell

Both stages have microfabricated chips that create the cell volume for gas/vapor/liquid

Nanoparticles, thin films, FIB'ed samples, and biological samples

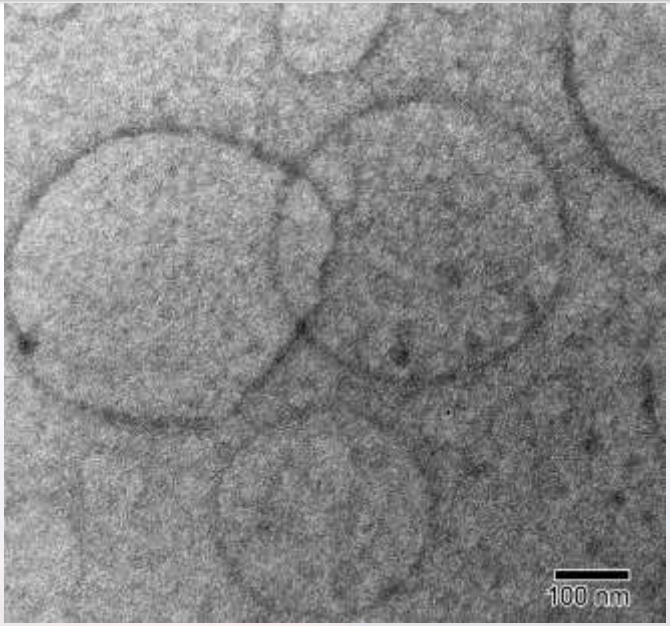






On their own or combined with ion irradiation, these stages offer access to new experimental realms

Liposome imaged in aqueous solution without stain or cryogenic techniques



In collaboration with: S. Hoppe, D. Sasaki

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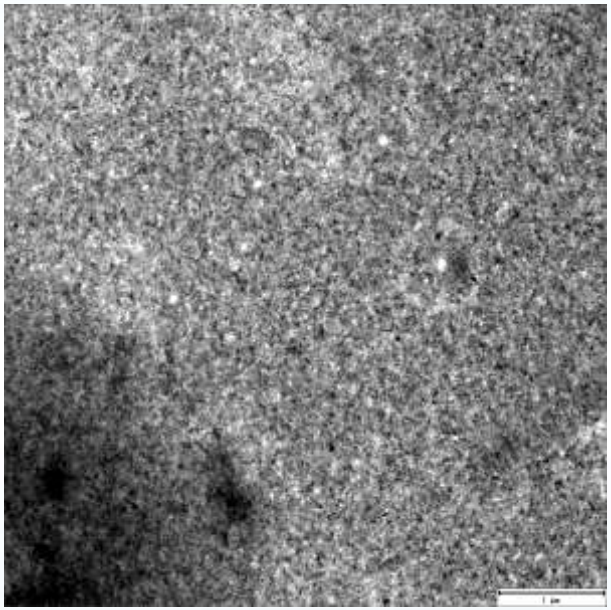
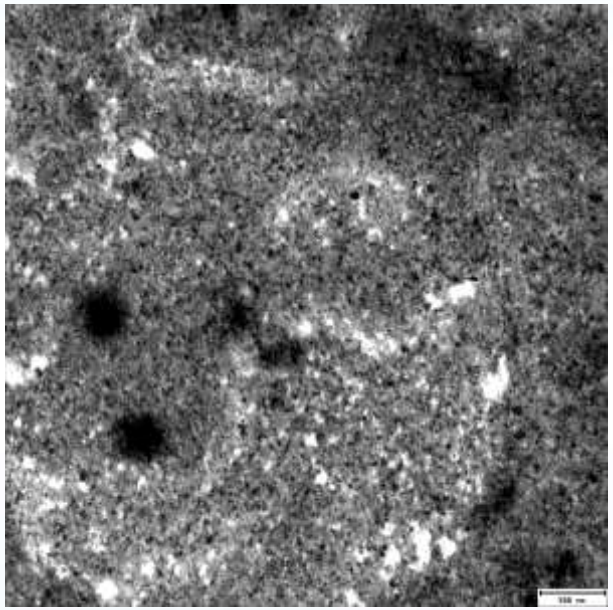
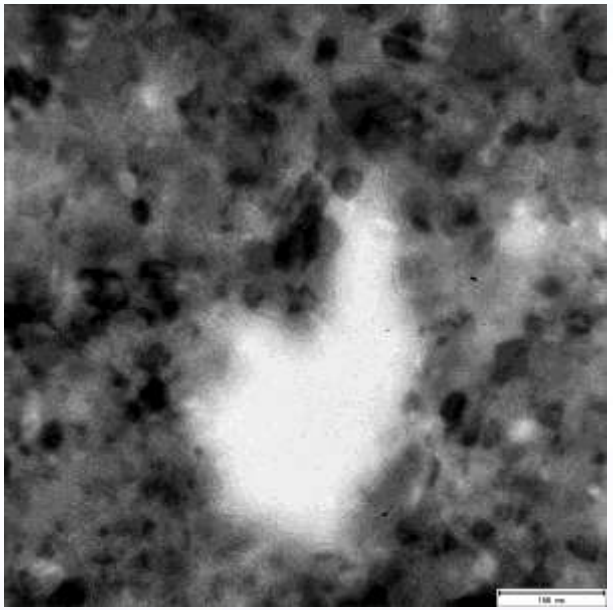
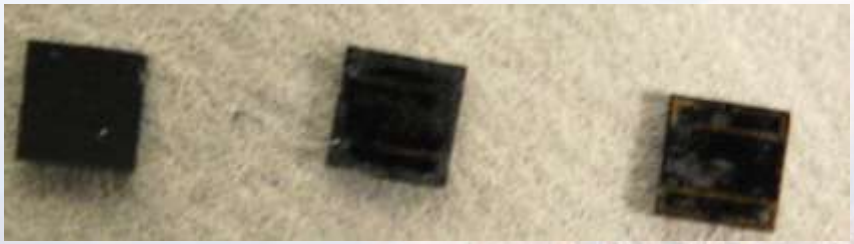
Corrosion of PLD Nanograined Cu

In collaboration with: S. Hoppe, B. Hernandez-Sanchez



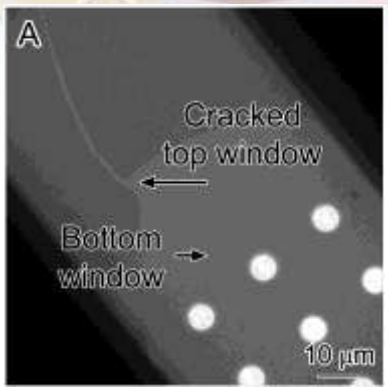


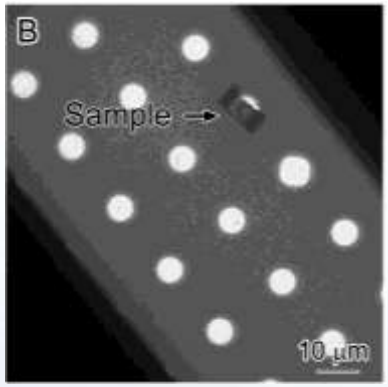


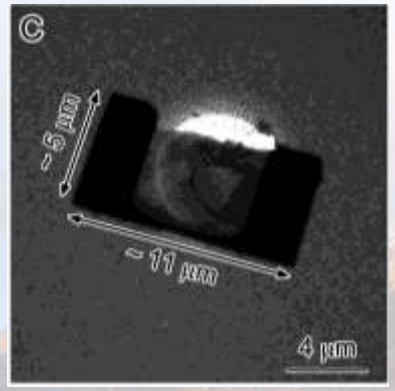


Initial results with PLD Cu films exposed to water and saline solution in the microfluidic stage suggests that fundamental studies into the active corrosion mechanism can be evaluated in either liquid or vapor environments.

Feasibility of studying Zircaloy 2 at nominally 1 atm

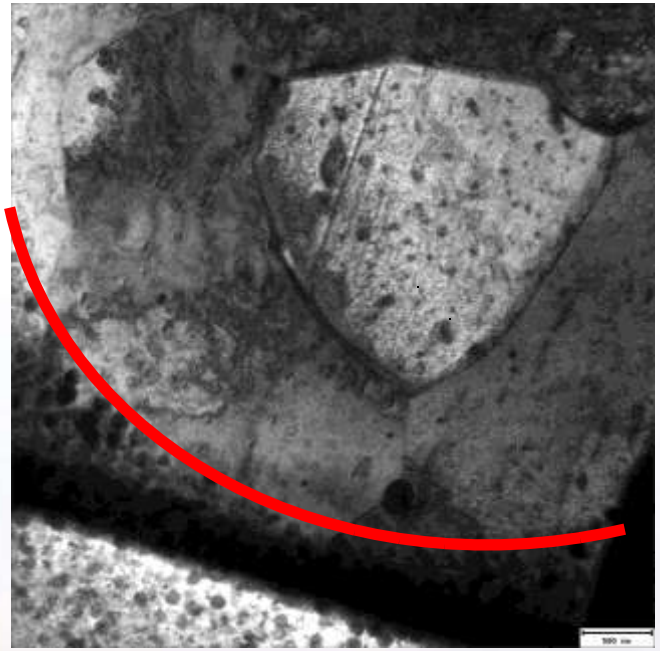




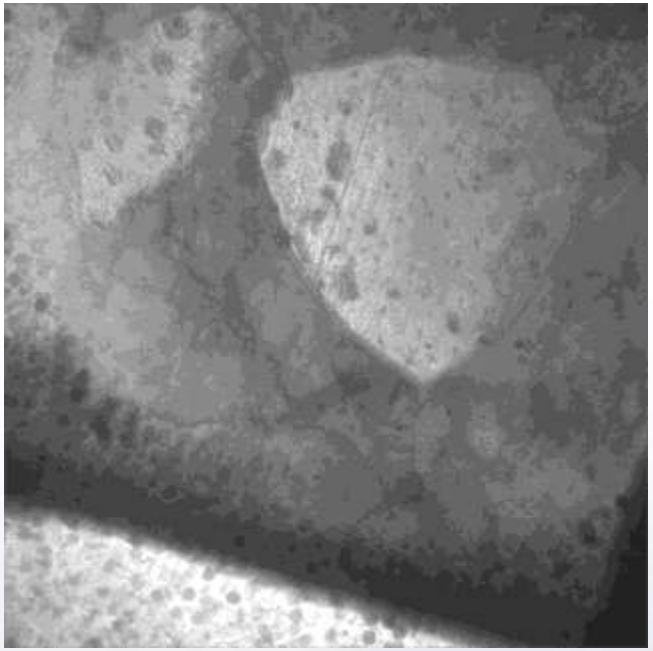


In collaboration with: S. Rajasekhara and B.G. Clark

Vacuum & Single Window



Nominally 1 atm & Two Windows



Most features are observed in both despite the decreased resolution resulting from the additional SiN window and 5 µm of air

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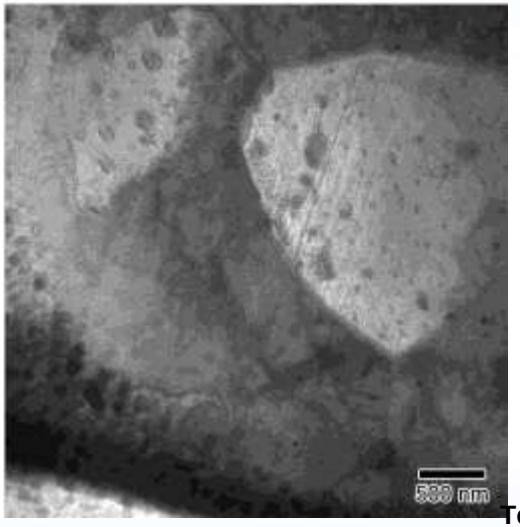
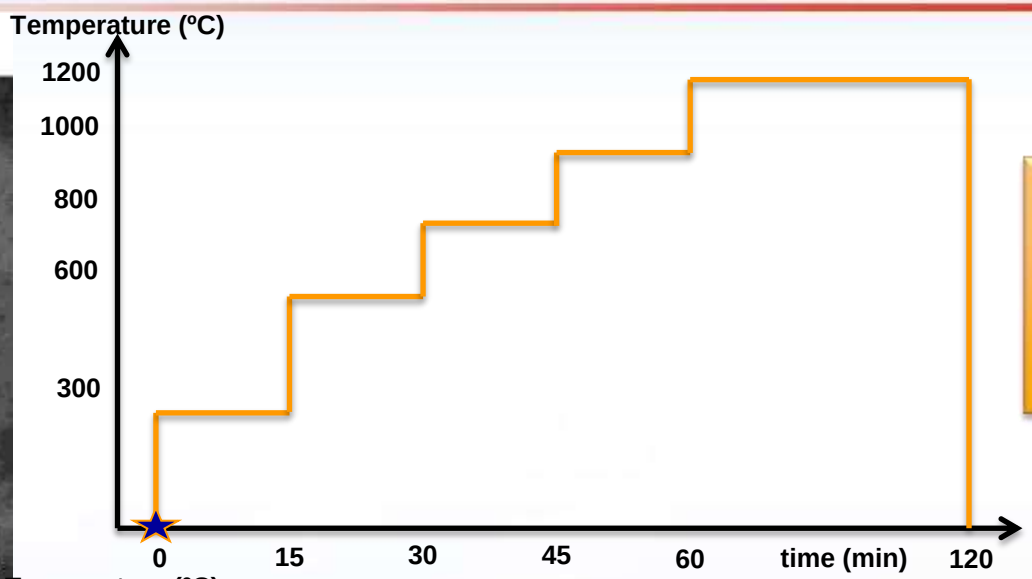
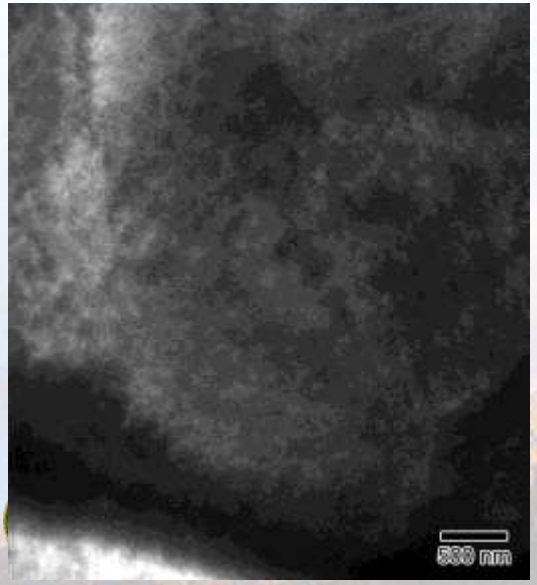
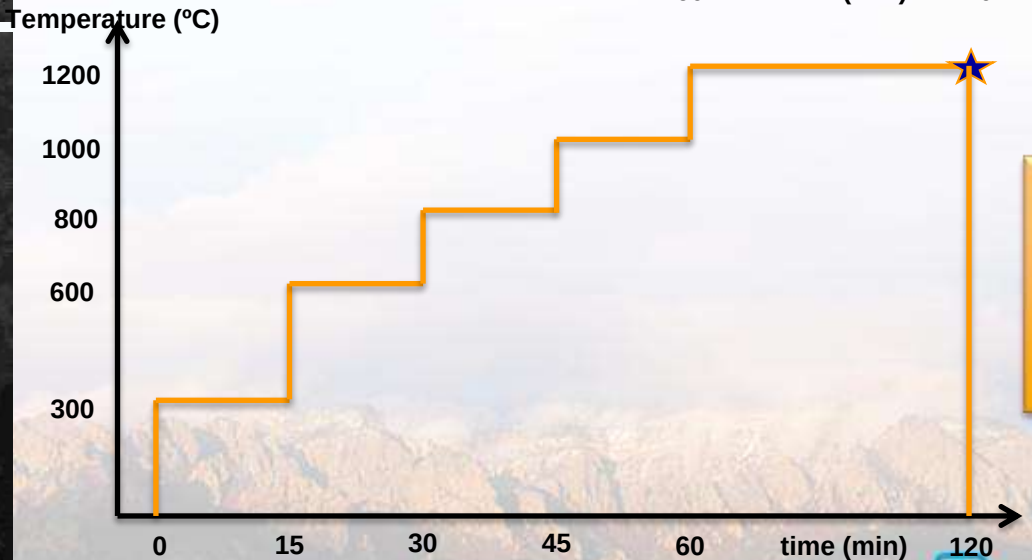
In situ TEM heating of Zircaloy





Zircaloy underwent rapid oxidation between 300ºC and 600ºC





Both stage and FIB foil are stable for 1 hour at 1,200ºC

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Summary and Future Work

- **The new ion beam lab at Sandia is operational**
  - 4 accelerators ranging from 1 keV to 100s of MeV
- ***In situ* ion irradiation and gas exposure are operational provides insight into the microstrucutral evolution under various extreme conditions**
- **Collaborations can be started by contacting**
  - Khalid Hattar khattar@sandia.gov
  - Blythe Clark blyclar@sanida.gov
  - Jon Custer jscute@sandia.gov

Future Work

- **Concurrent *in situ* ion irradiation**
  - Dual beam exposure
  - Triple beam exposure
- **Hydrogen exposure at elevated temperature**

Sandia is developing a suite of *in situ* TEM tools for studies applicable to nuclear materials

Acknowledgements

- All staff and technologists of the IBL at SNL
- D. Nakashi and J. Domiano, Protochips Inc

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