Development of a Concurrent *In situ* Ion Irradiation TEM

SAND2014-2629C

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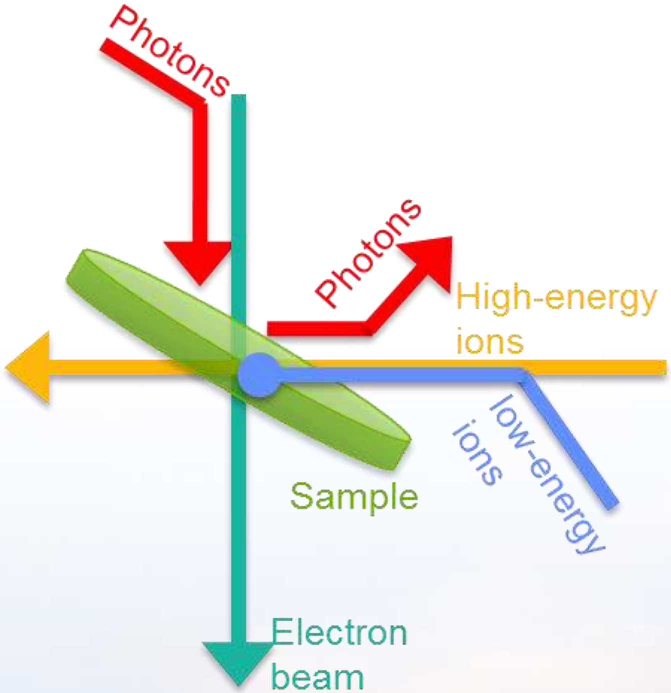
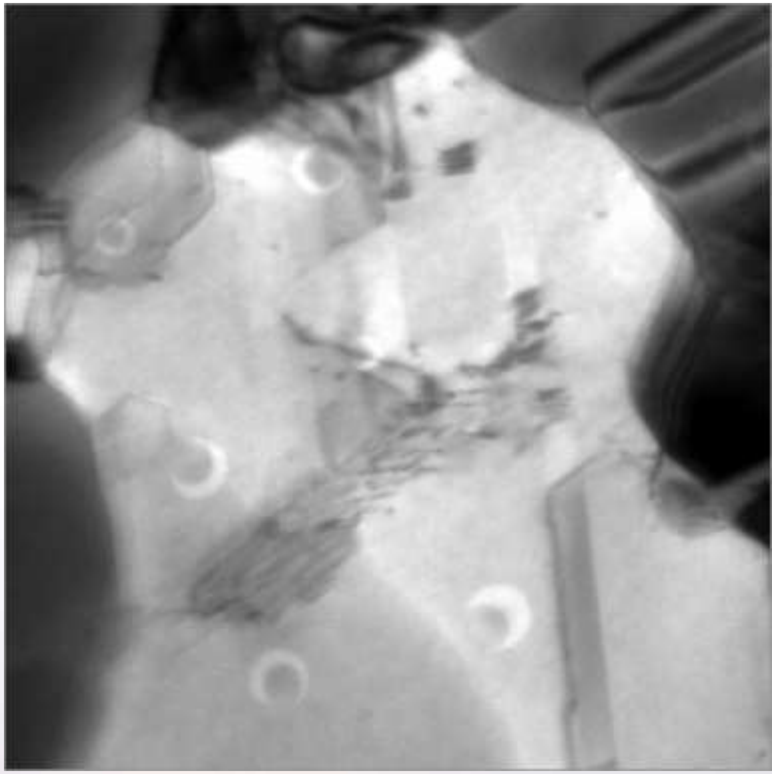
Sandia National Laboratories

April 23, 2014



Triple beam *In situ* Ion Irradiation TEM:

1) Provides exceptional insight into extreme environments
2) Is completely dependent on the ion, electron, and photon optics.



Collaborators:

J.A. Scott, D. Masiel, N. Li, A. Misra, B.A. Hernandez-Sanchez, T.J. Boyle, J. Sharon, B. L. Boyce, C. Chisholm, A.M. Minor, D. Sasaki, B.G. Clark, D. Gross, J. Kacher, P.J. Cappillino, B.W. Jacobs, L.R. Parent, I. Arslan, M.A. Hekmaty, D.B. Robinson, & I.M. Robertson,

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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

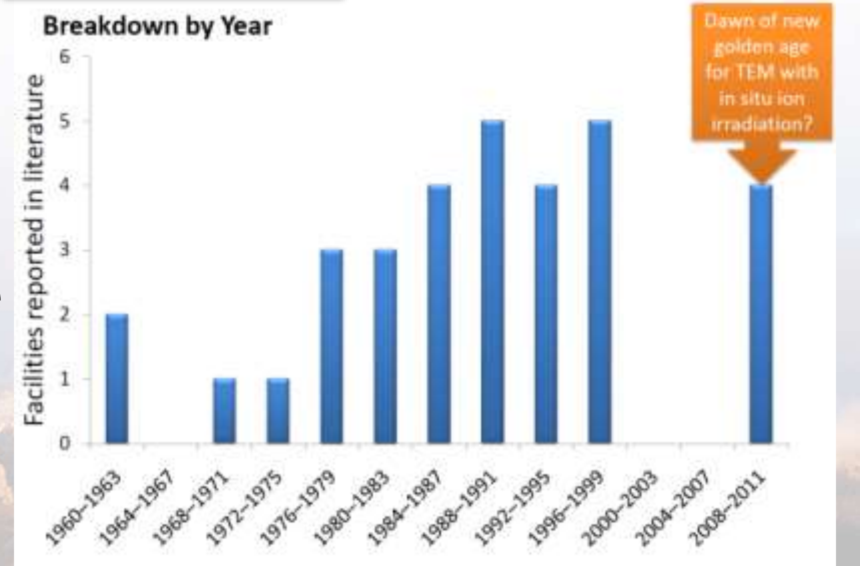
1968
First TEM beamline combination by Thackery, Nelson, and Sansom at AERE Harwell, UK

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

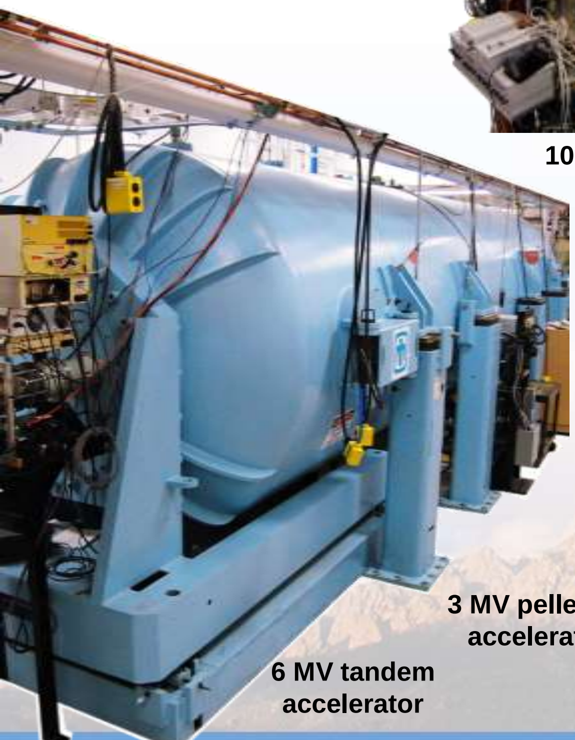
Ion Irradiation Capabilities at Sandia's Ion Beam Lab

- Highest Energy in situ TEM ion irradiation facility
- Capable of studying end of range interactions

350 kV implanter

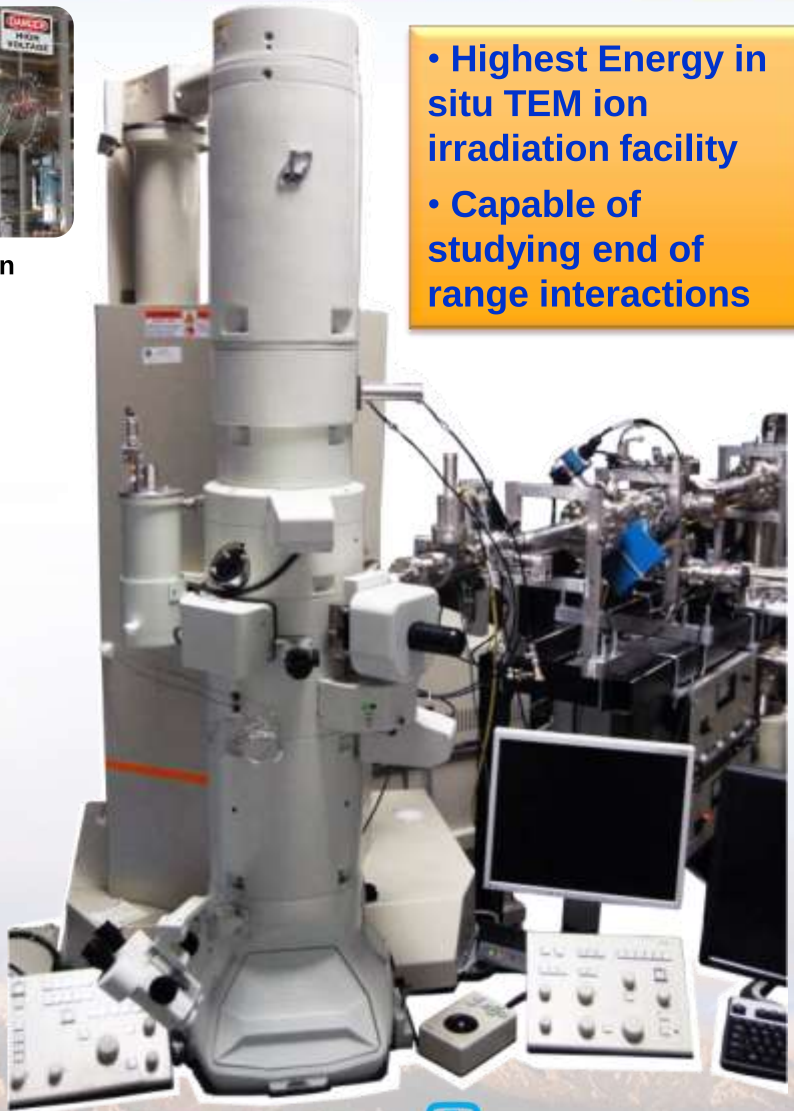
100 kV nanoimplanter

10 kV Colutron



6 MV tandem accelerator

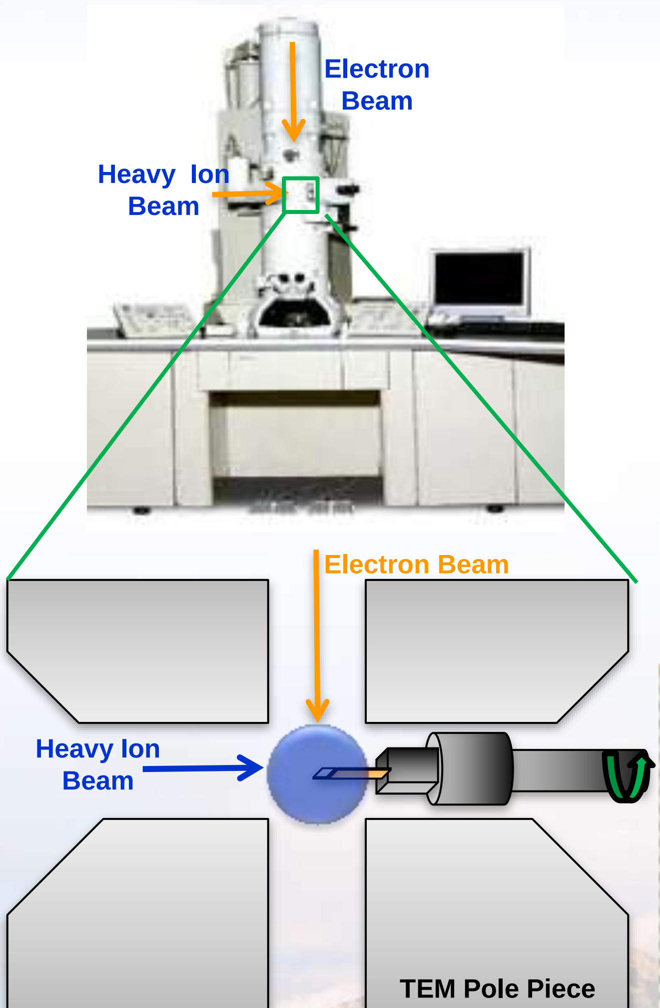
3 MV pelletron accelerator



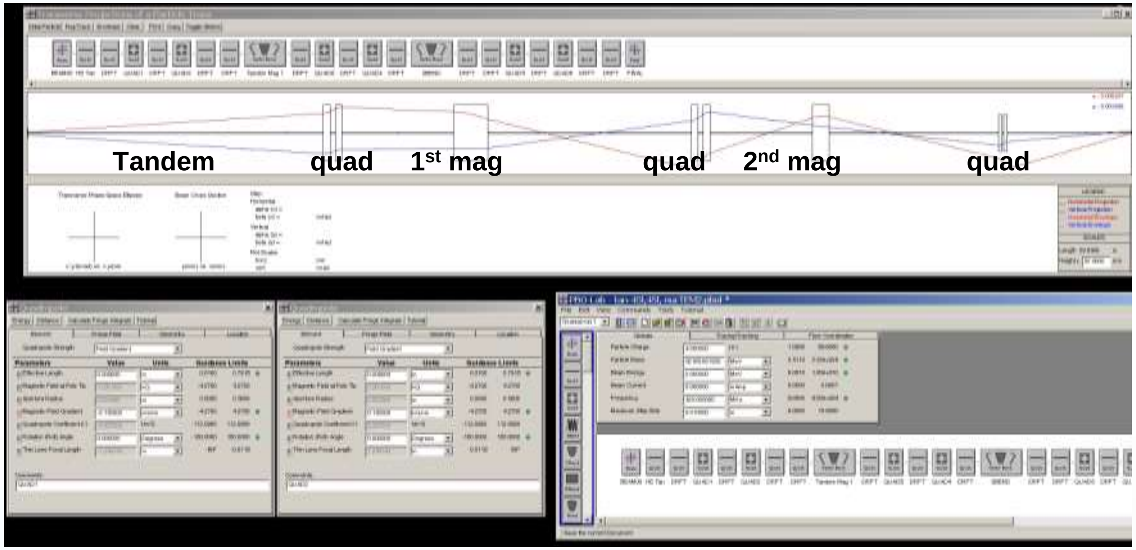
200 kV TEM attached to 6 MV tandem

Beam Optic Models and Image Simulation of the *In situ* Ion Irradiation TEM

Collaborators: P. Rossi

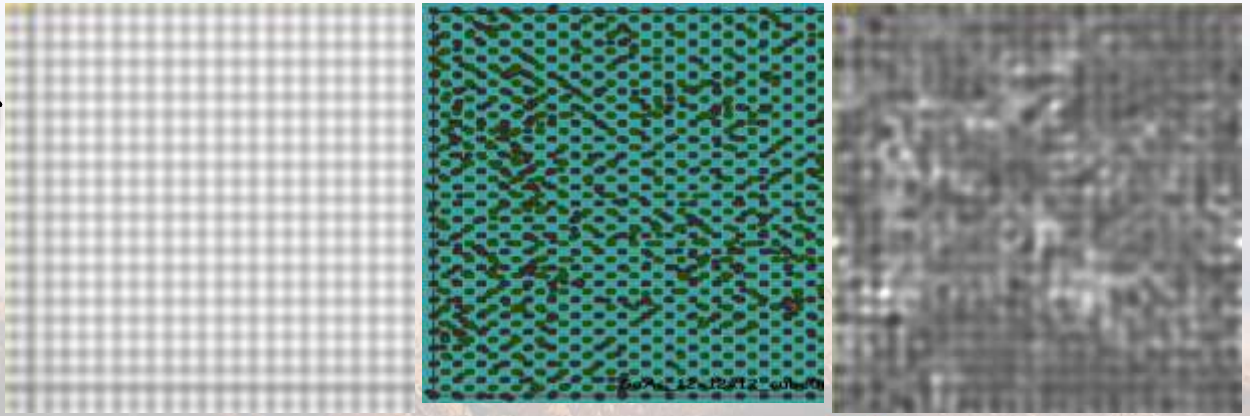


Ion Optics Feasibility Study



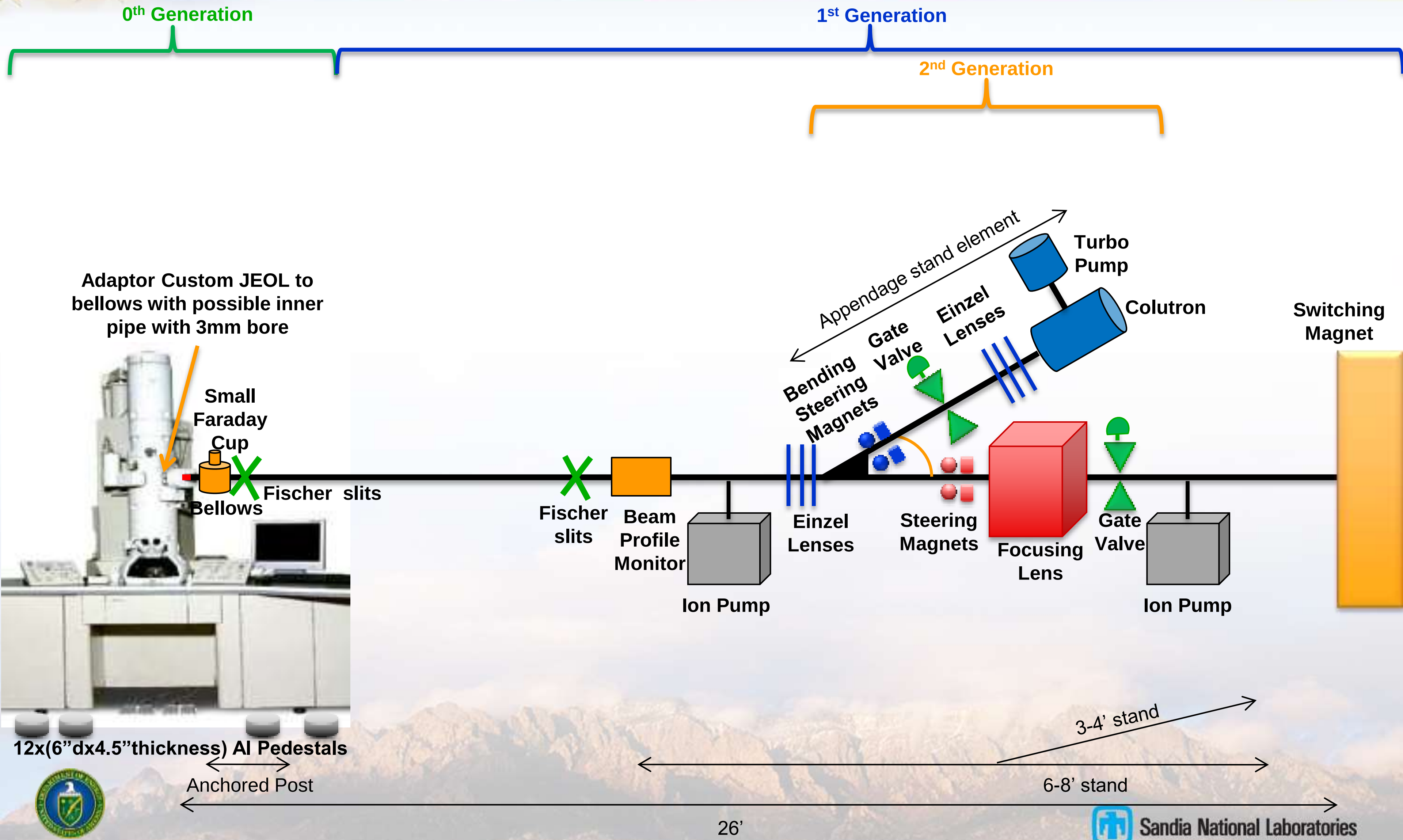
TEM image simulation of radiation damage

JEMS undamaged | Marlowe | JEMS damaged

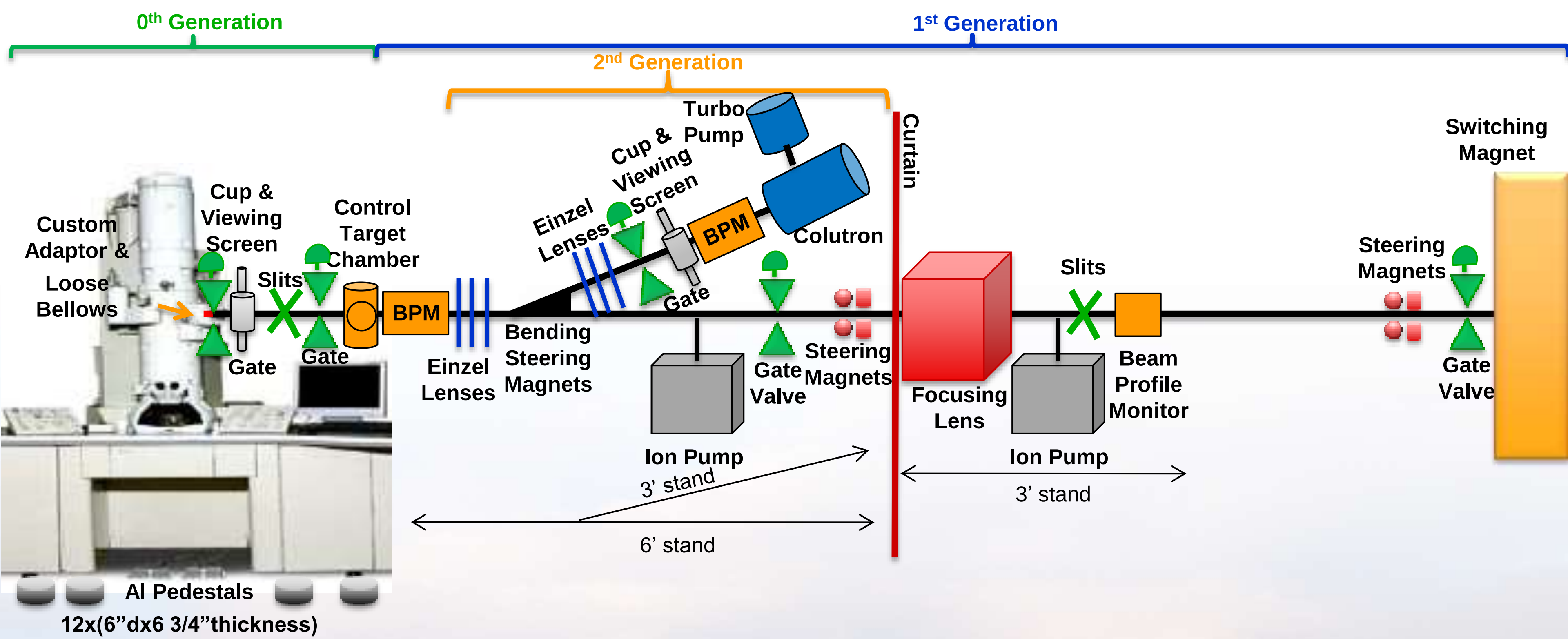


Simulation (Marlowe+JEMS) shows the project is viable (30 MeV Cu -> GaAs)

Schematic of the *In situ* TEM Beamline



Schematic of the *In situ* TEM Beamline



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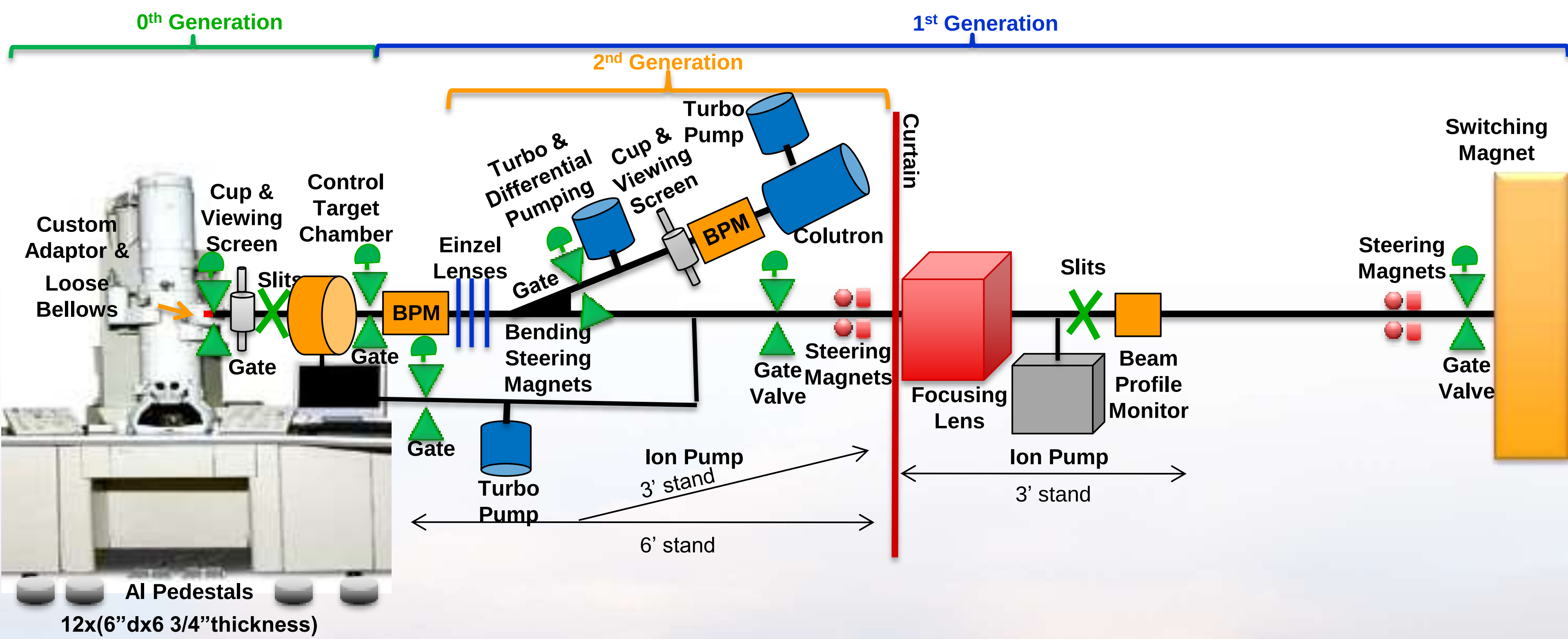
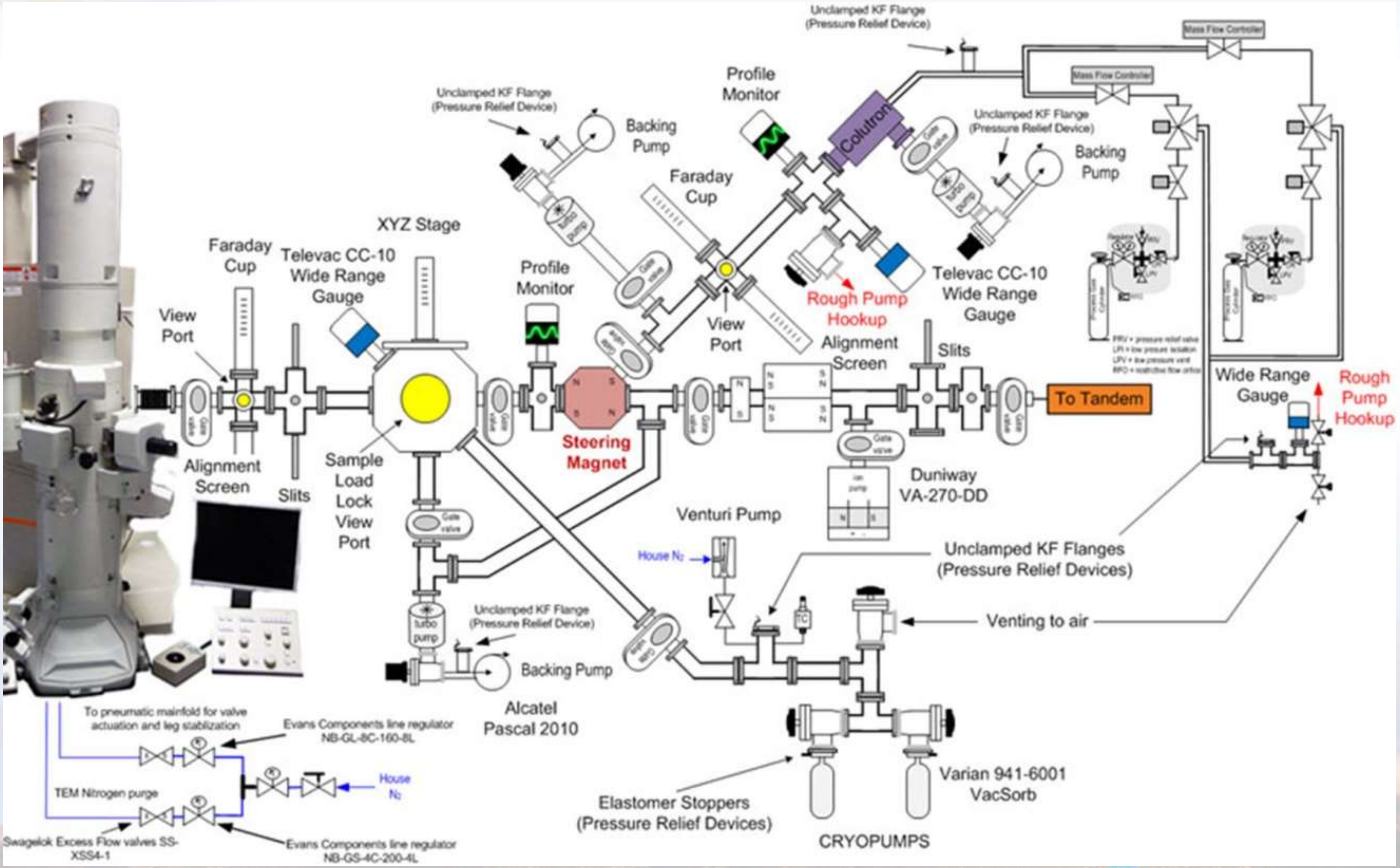
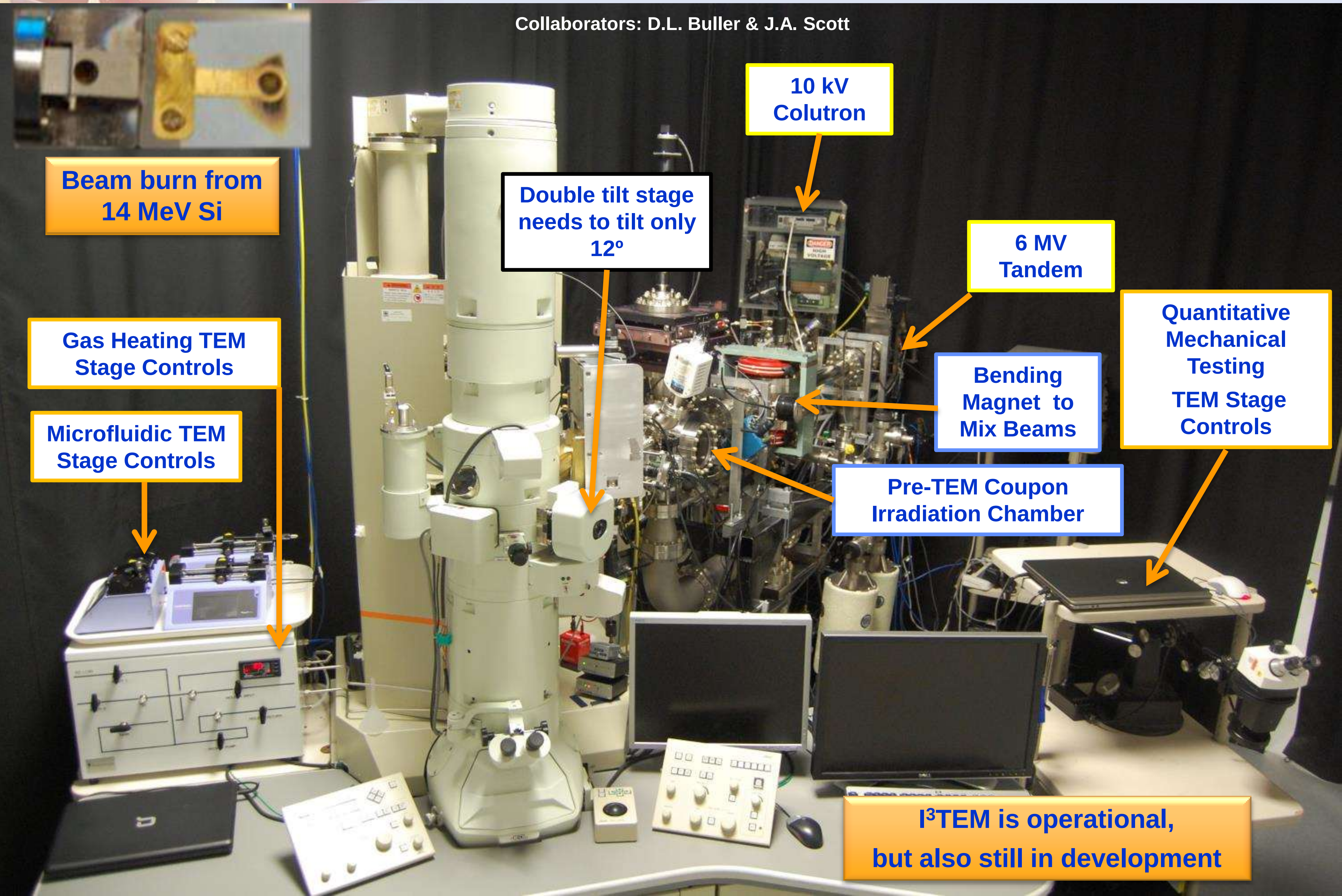


Schematic of the *In situ* TEM Beamline

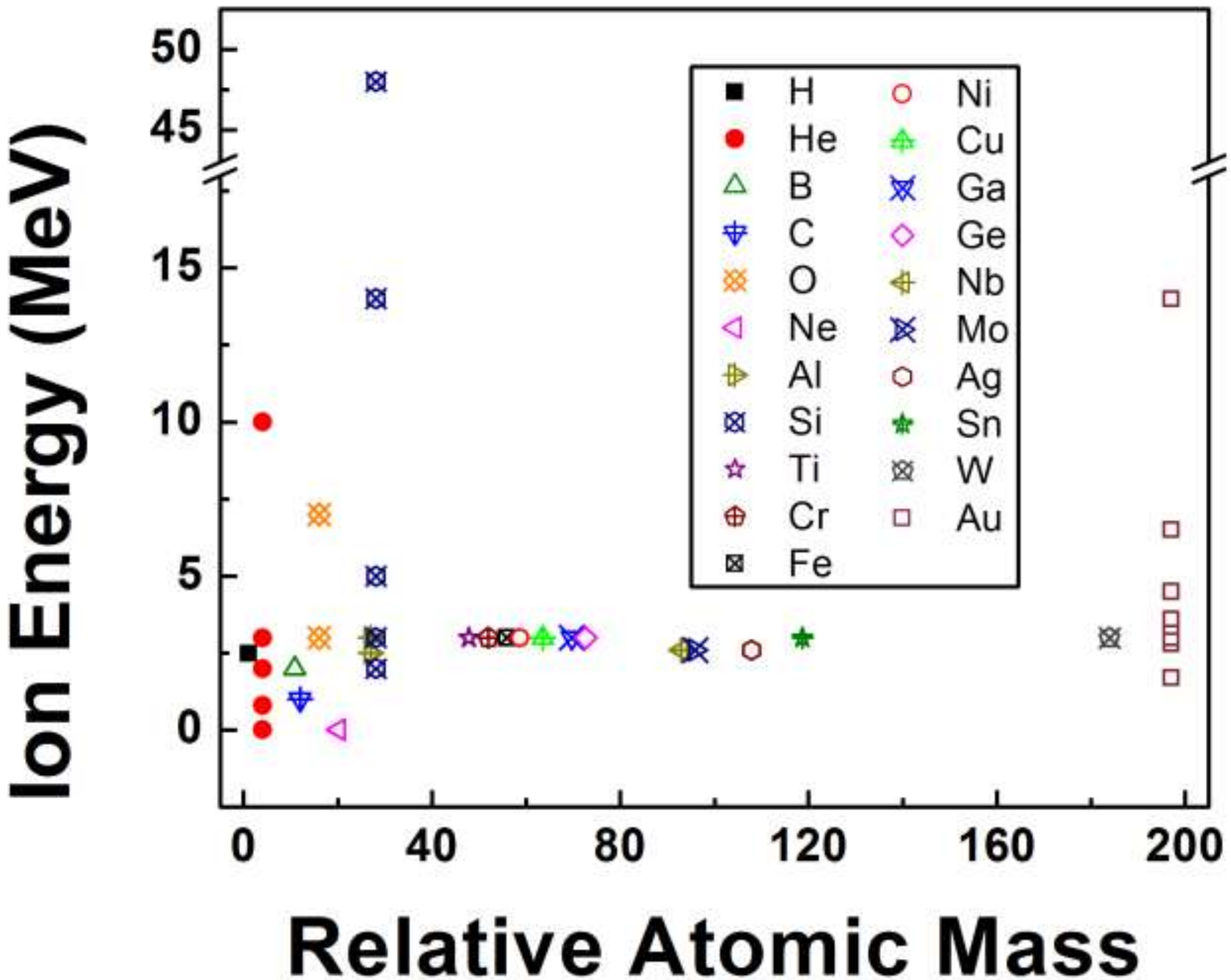


Current Status of the *In situ* TEM Beamline



Ion Species Recently Attempted

Collaborators: M. Steckbeck



Colutron ion beams possible:

0.8-20 keV energies

Any gas species

Colutron ion beams to date:

8, 10, & 20 keV energies

He, D_2, & Ne

Tandem ion beams possible:

0.8-88 MeV energies

Any sputtered or alpha sources

Tandem ion beams to date:

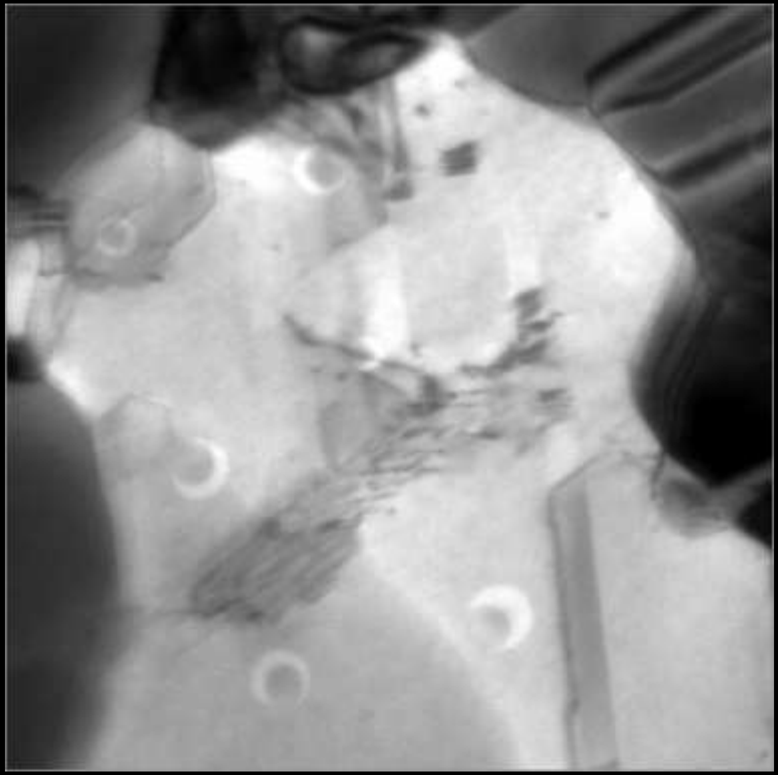
0.8-48 MeV energies

Large range

Single Ion Strikes

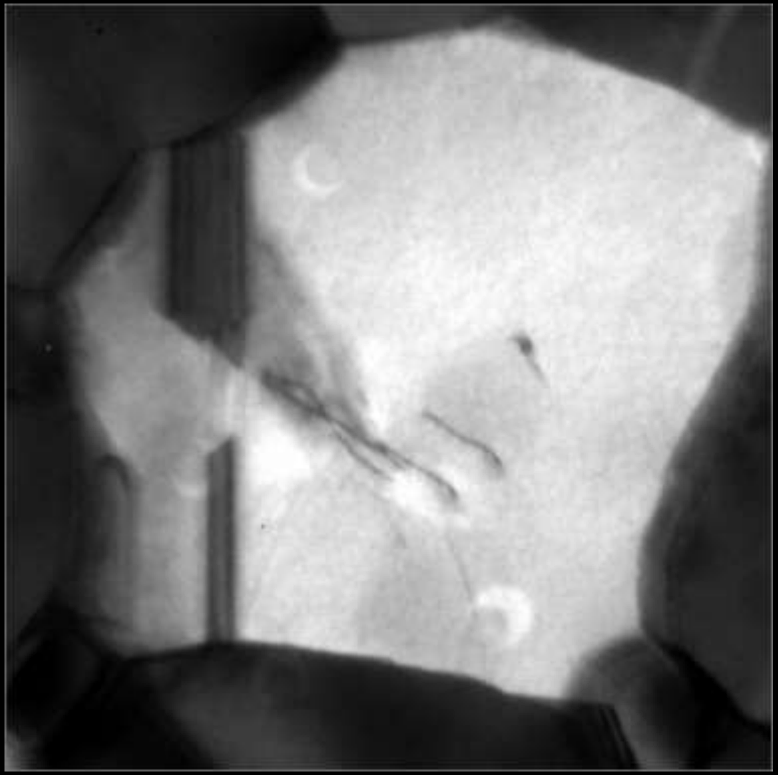
Collaborators: C. Chisholm & A. Minor

7.9×10^9 ions/cm^2/s



VS

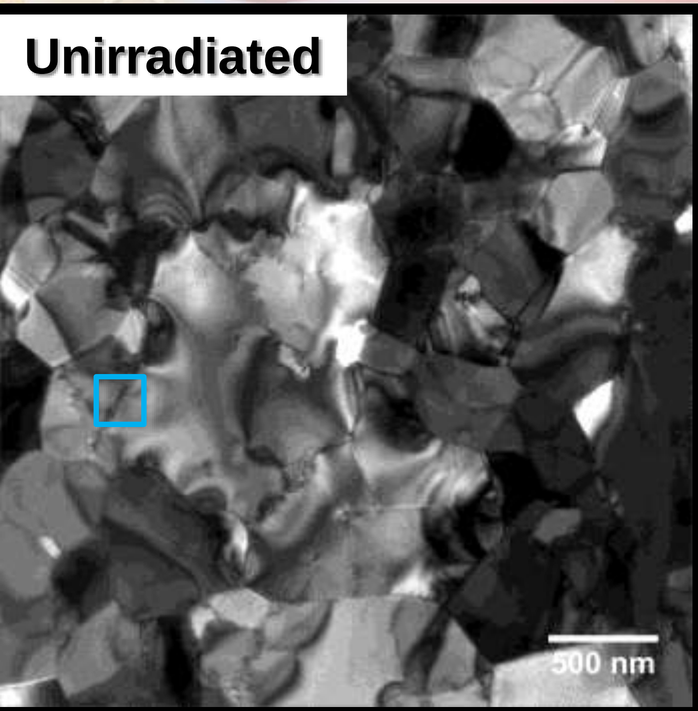
6.7×10^7 ions/cm^2/s

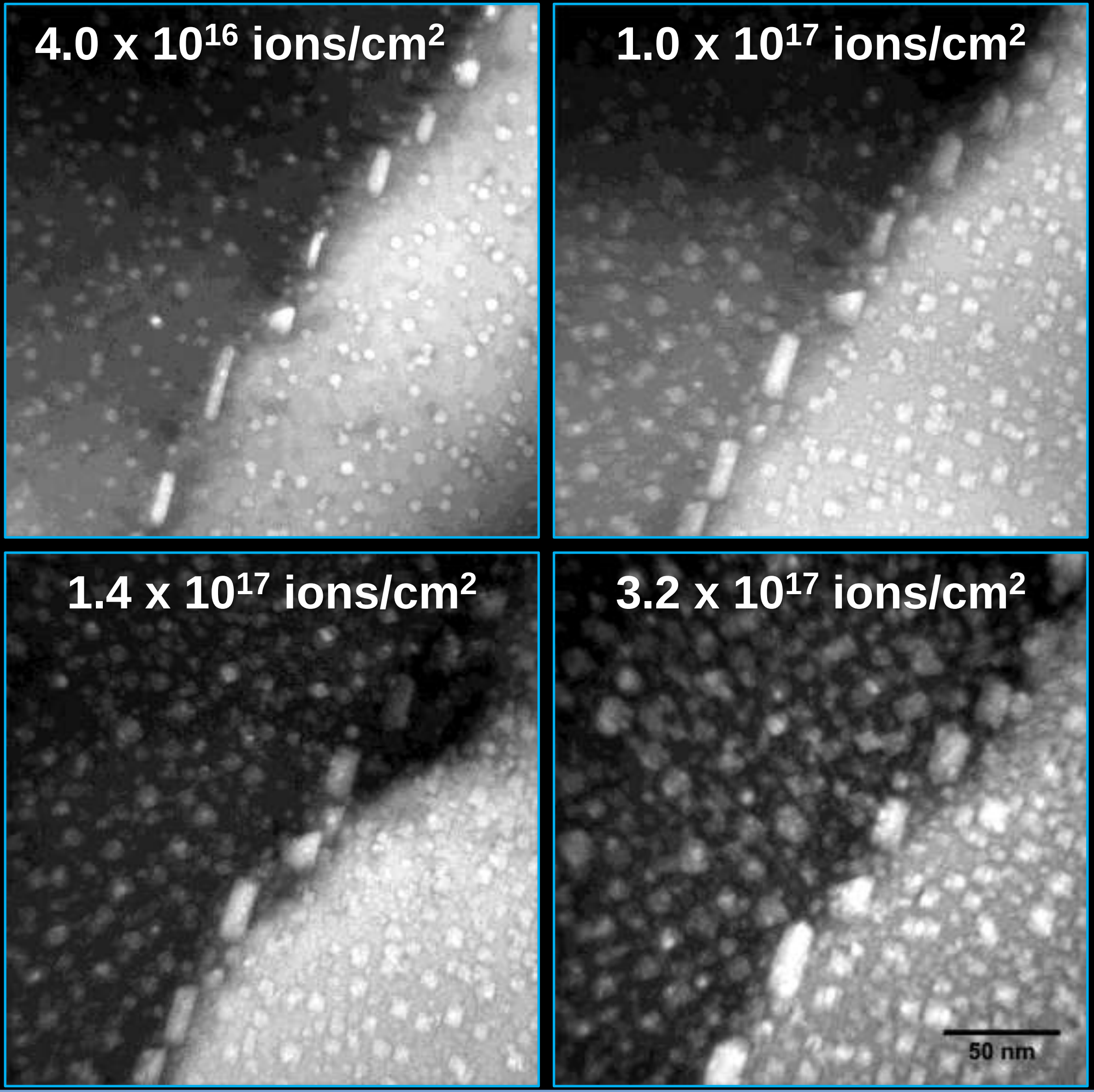


Improved vibrational and ion beam stability permits us to work at 120kx or higher permitting imaging of single cascade events

In situ Implantation

Collaborators: C. Chisholm & A. Minor



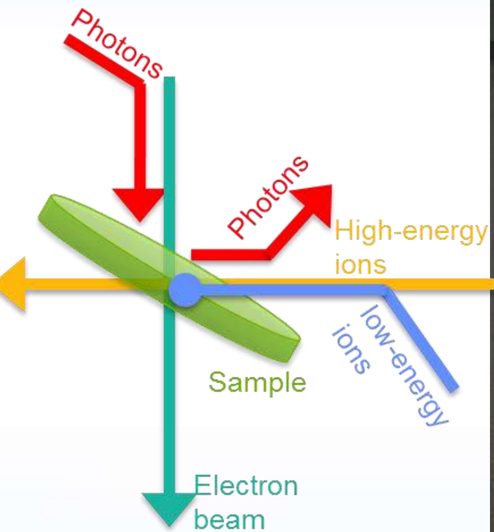


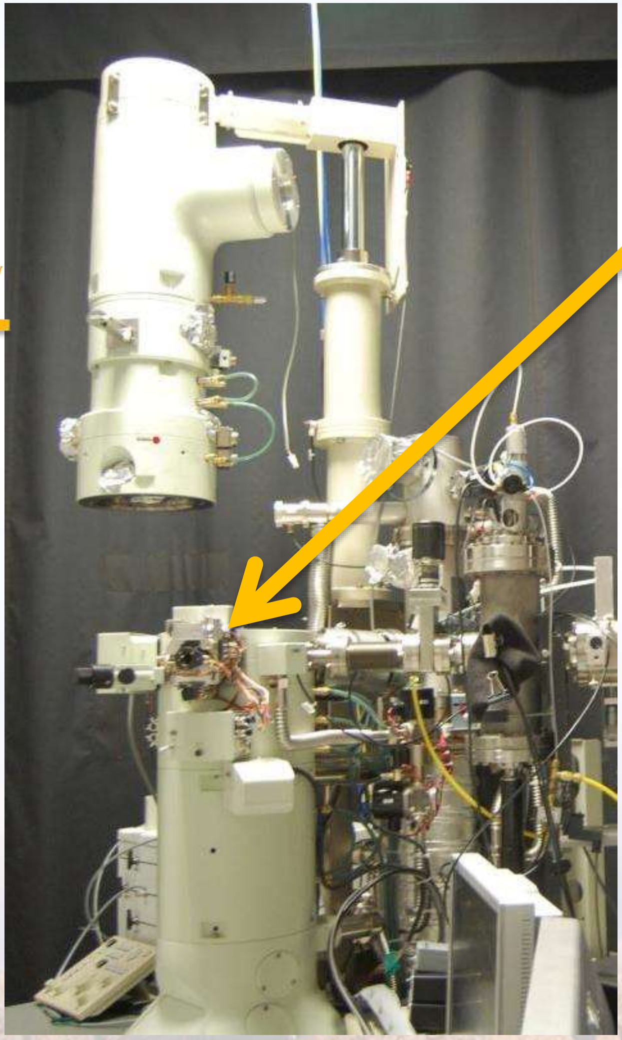
Gold thin-film implanted with 10keV He^{2+}

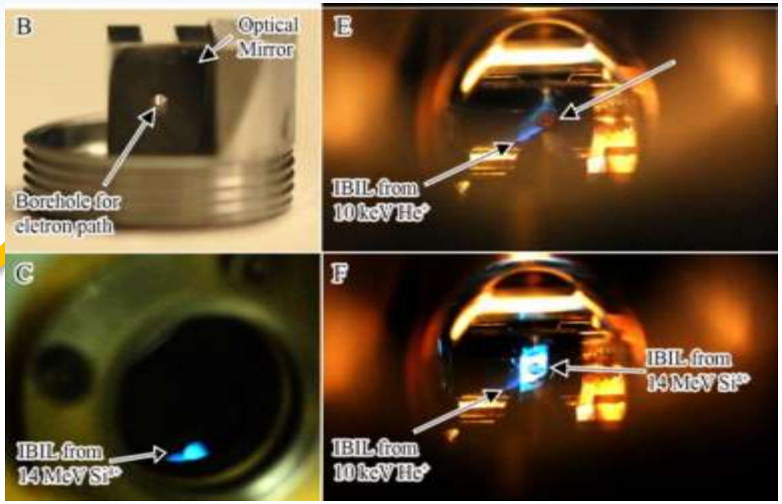
Result: porous microstructure

In situ TEM Luminescence

Collaborators: D. Masiel and C. Chisholm







Two optical port were added to the I³TEM already containing a electron beam and two ion beams, which permits *in situ* TEM luminescence studies

Optical Pathway in an I³TEM

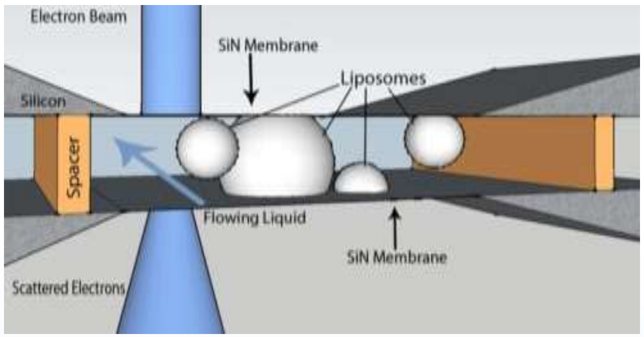
- **Angled mirror with bore hole for the electron path was installed above the polepiece**
- **Another mirror is located just above the ion beams in the beamline**
- **Two perspective of the sample are possible**
- **Permits *in situ* IBIL and CL.**

In situ TEM microfluidic Environments

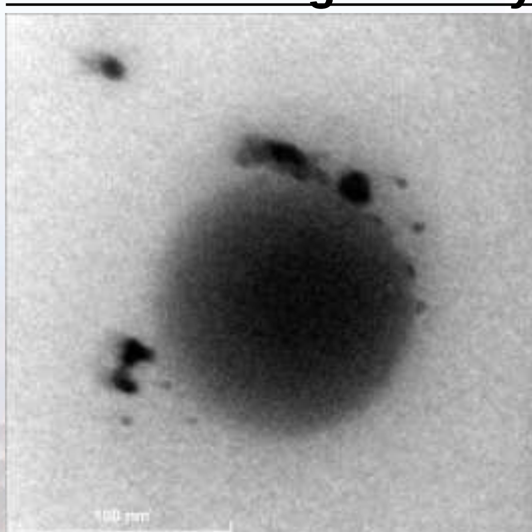
Contributors: S.H. Pratt, E. Carnes, J. Brinker, D. Sasaki, D. Gross, J. Kacher, I.M. Robertson & Protochips Inc.

Microfluidic Stage

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



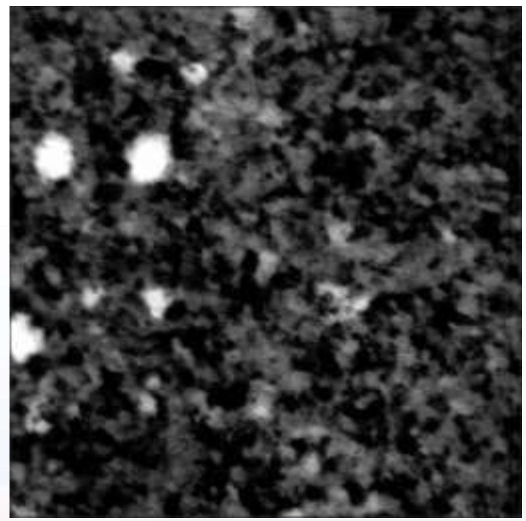
Protocell Drug Delivery



Liposome encapsulated Silica destroyed by the electron beam in aqueous environment

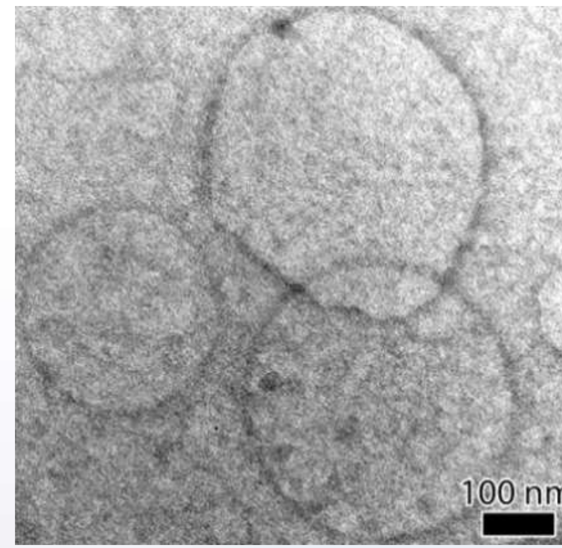
***In situ* microfluidic TEM can provide insight in events as diverse as corrosion and drug delivery**

Fe Corrosion



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

Liposomes in Water



Liposomes imaged in flowing aqueous channel

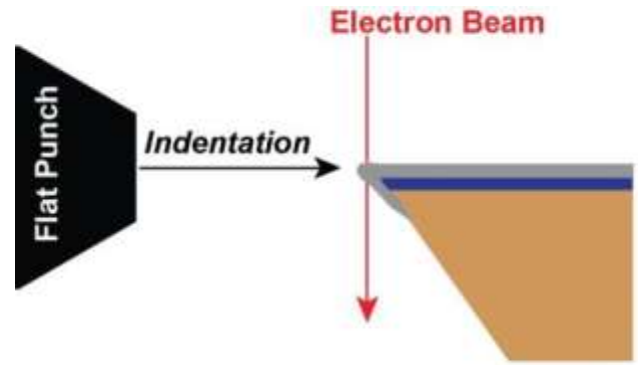
We hope to go a step further and combine it with ion implantation and irradiation environments

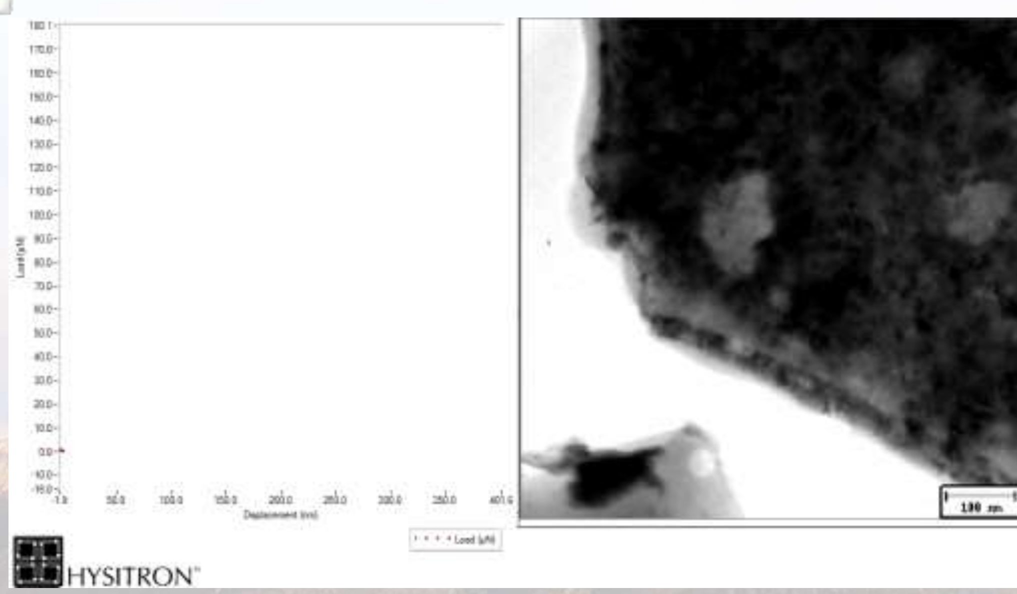
In situ TEM Quantitative Mechanical Testing

Contributors: J. Sharon, B. L. Boyce, C. Chisholm, A.M. Minor, & Hysitron Inc.

In situ TEM Fatigue

- **Microscope: JEOL JEM 2100 LaB_6 TEM**
  - **Holder: Hysitron PI-95 PicoIndenter with a 1µm flat tip punch**





Summary

The capabilities range drastically from facility to facility.

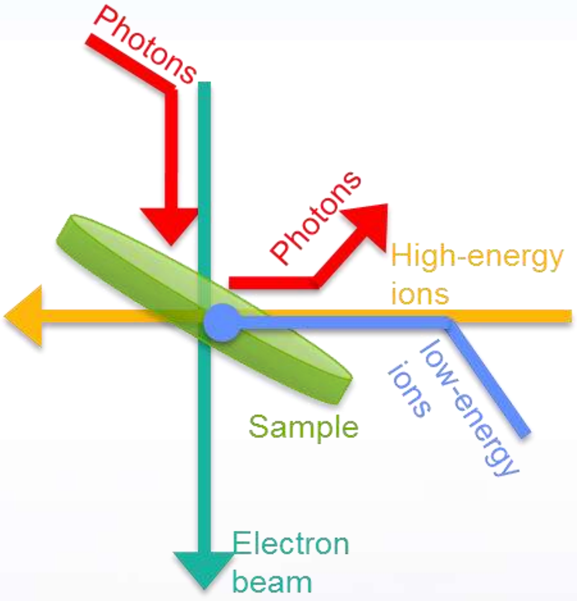
Sandia's I^3TEM is one of only two facilities in the US

- *In situ* irradiation from H to Au
- *In situ* gas implantation

I^3TEM can provide fundamental understanding to key mechanisms in a variety of extreme conditions

The capabilities are limited by the optics.

The I^3TEM capability are still being expanded



Optics design, development, and iterations are essential to explore multi-beam facilities

Collaborators:

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