In situ Ion Irradiation and Cyclic Indentation TEM Experiments of Nanocrystalline Metals

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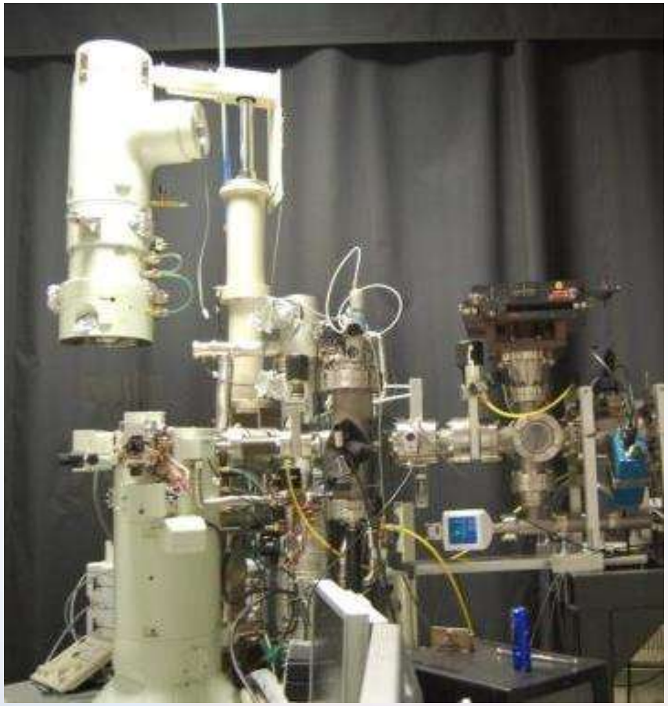
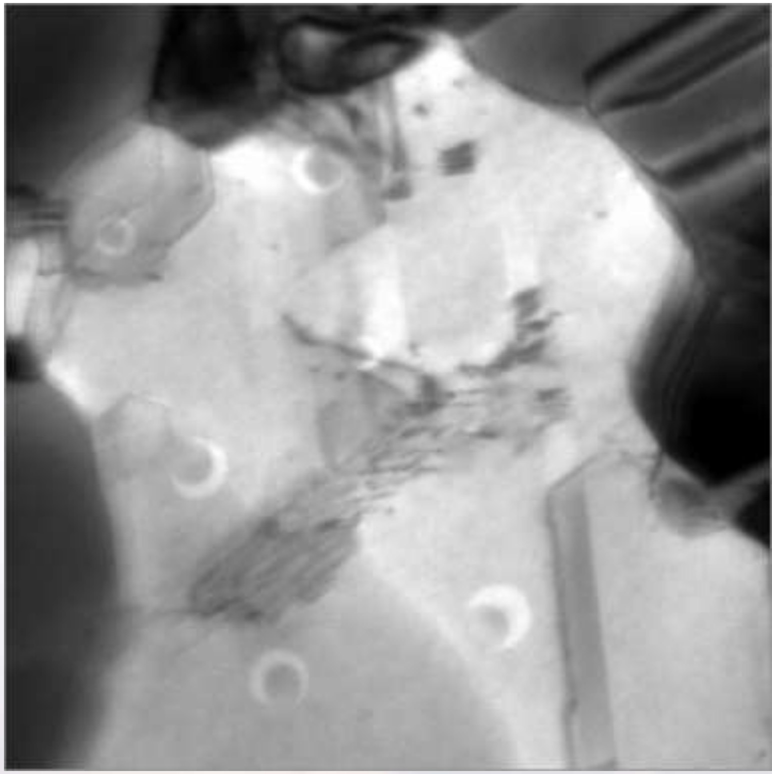
Sandia National Laboratories & Lawrence Berkeley National Laboratory

February 18, 2014



In situ **TEM ion irradiation and cyclic indentations is providing initial insight into the stability of nanocrystalline metals in extreme environments.**



Collaborators:

D. Buller, J.A. Scott, D. Masiel, N. Li, A. Misra, S.M. Hoppe, B.A. Hernandez-Sanchez, T. Boyle, H. Bei, E.P. George, D. Gross, J. Kacher, & I.M. Robertson

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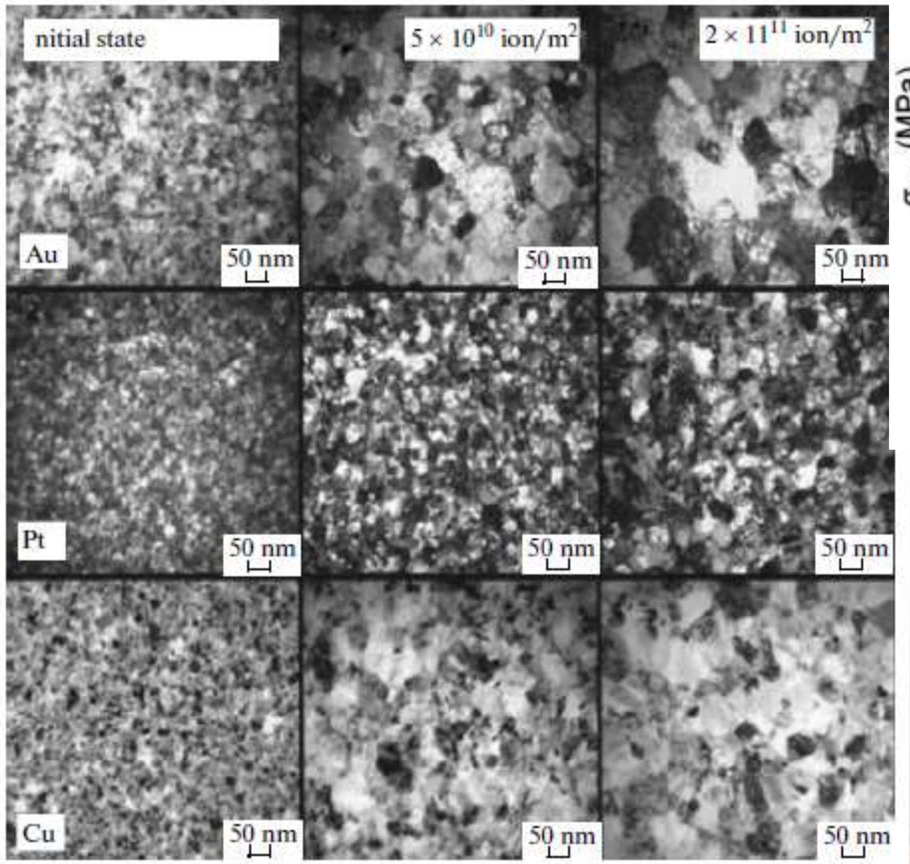
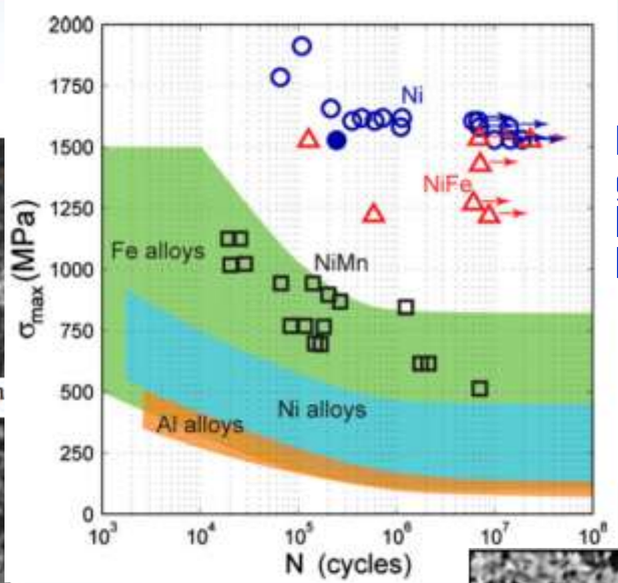
Grain Boundary Stability in Nanocrystalline Metals in Radiation and Cyclic Environments

Extent of grain growth varies as a function of system and radiation environments.

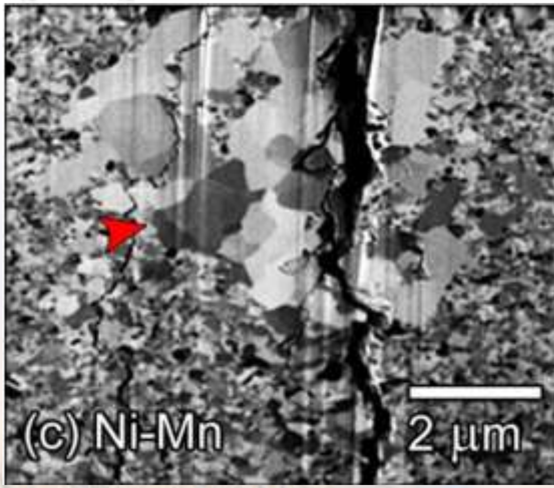


Kaomi et al., JAP: 2008. 104 073525



Boyce & Padilla, Met.&Mat. Trans. A: 2011. 42A

Fatigue induced grain growth limiting the fatigue lifetime



To a first order mean grain size comparison, these reports appear conflicting.

Not necessarily the case if initial microstructural details and associated properties are considered

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

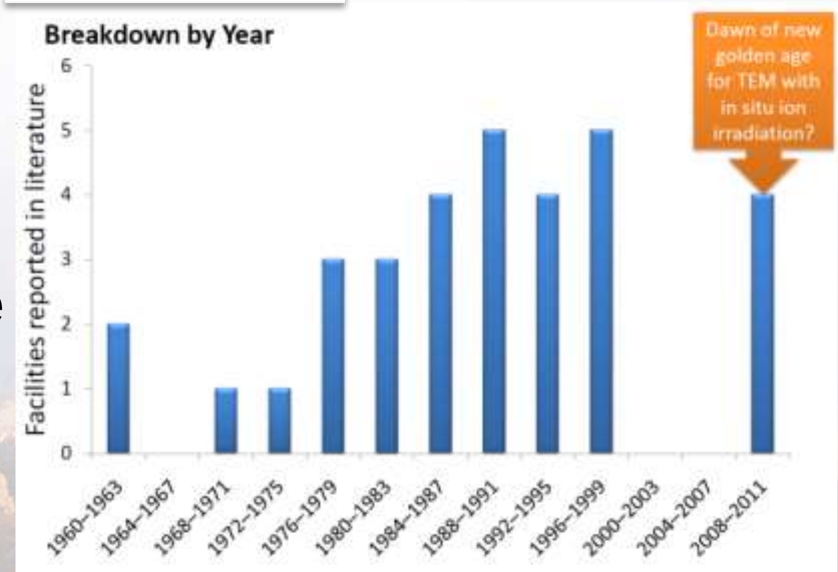
1968
First TEM beamline combination by Thackery, Nelson, and Sansom at AERE Harwell, UK

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"

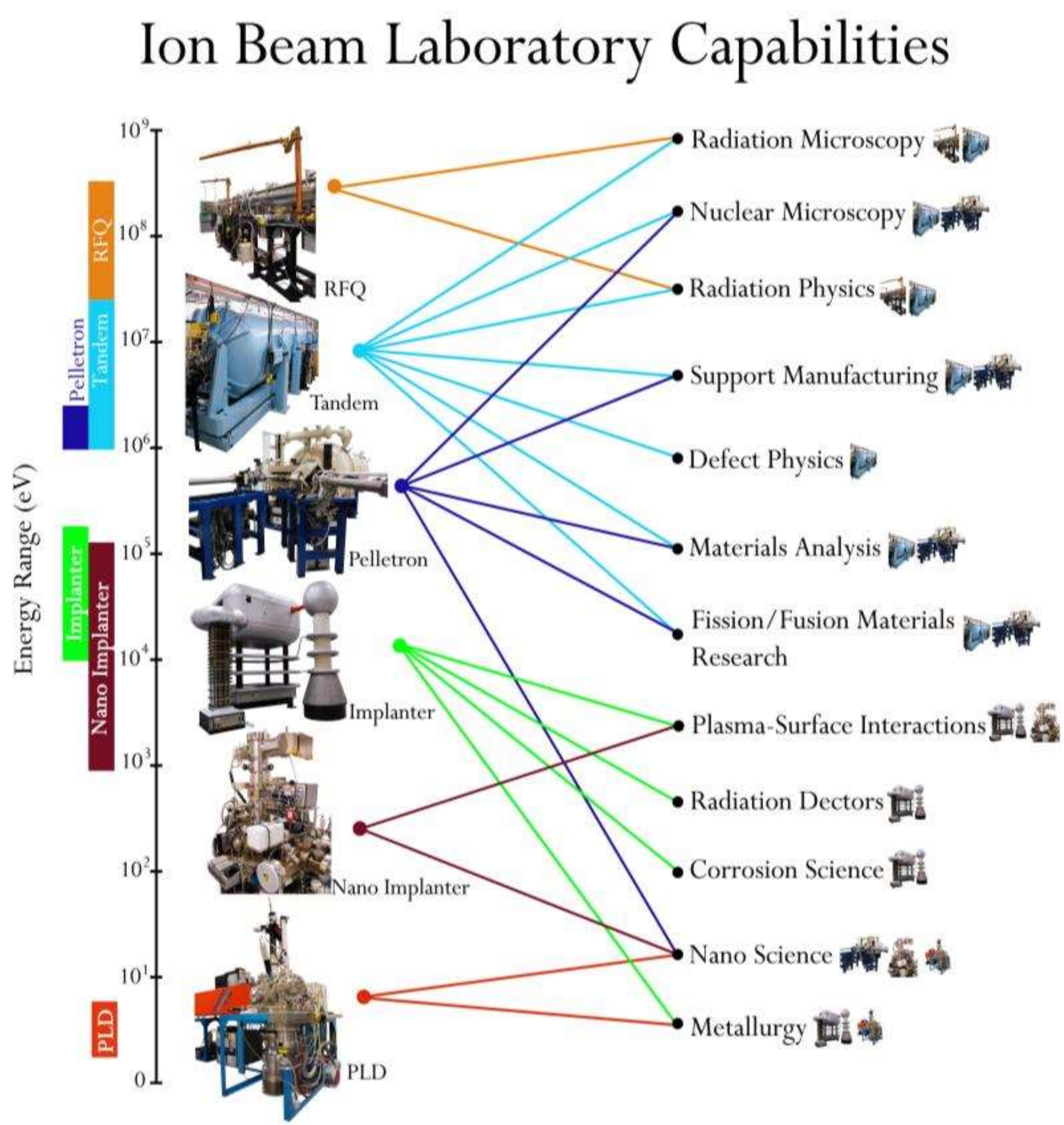
D.W. Pashley and A.E.B. Presland Phil Mag. 6(68) 1961 p. 1003

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

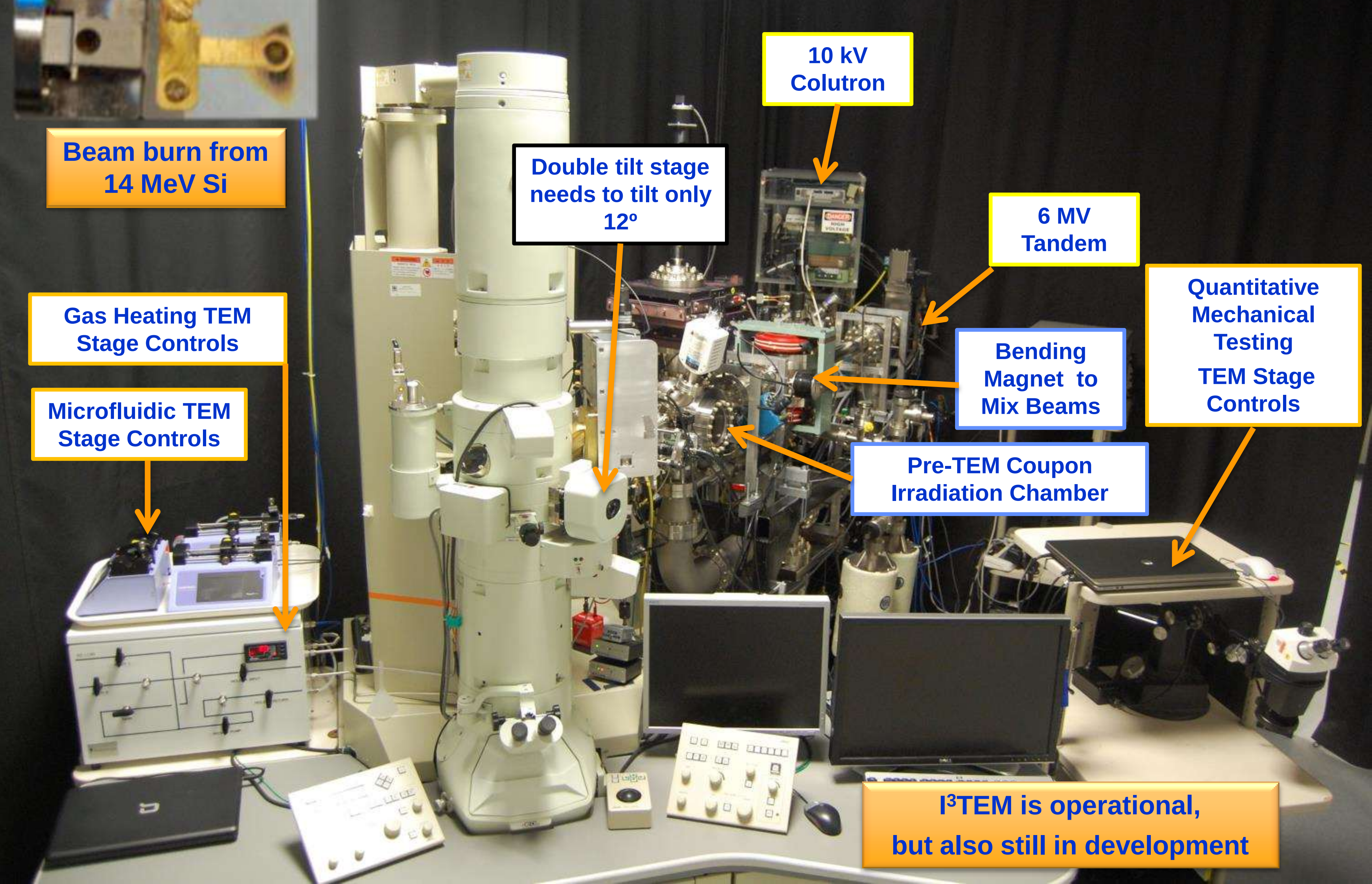
<u>New Facility</u>
laboratory space
1850 m^2
office space
650 m^2
<u>Old Facility:</u>
1300 m^2 total

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



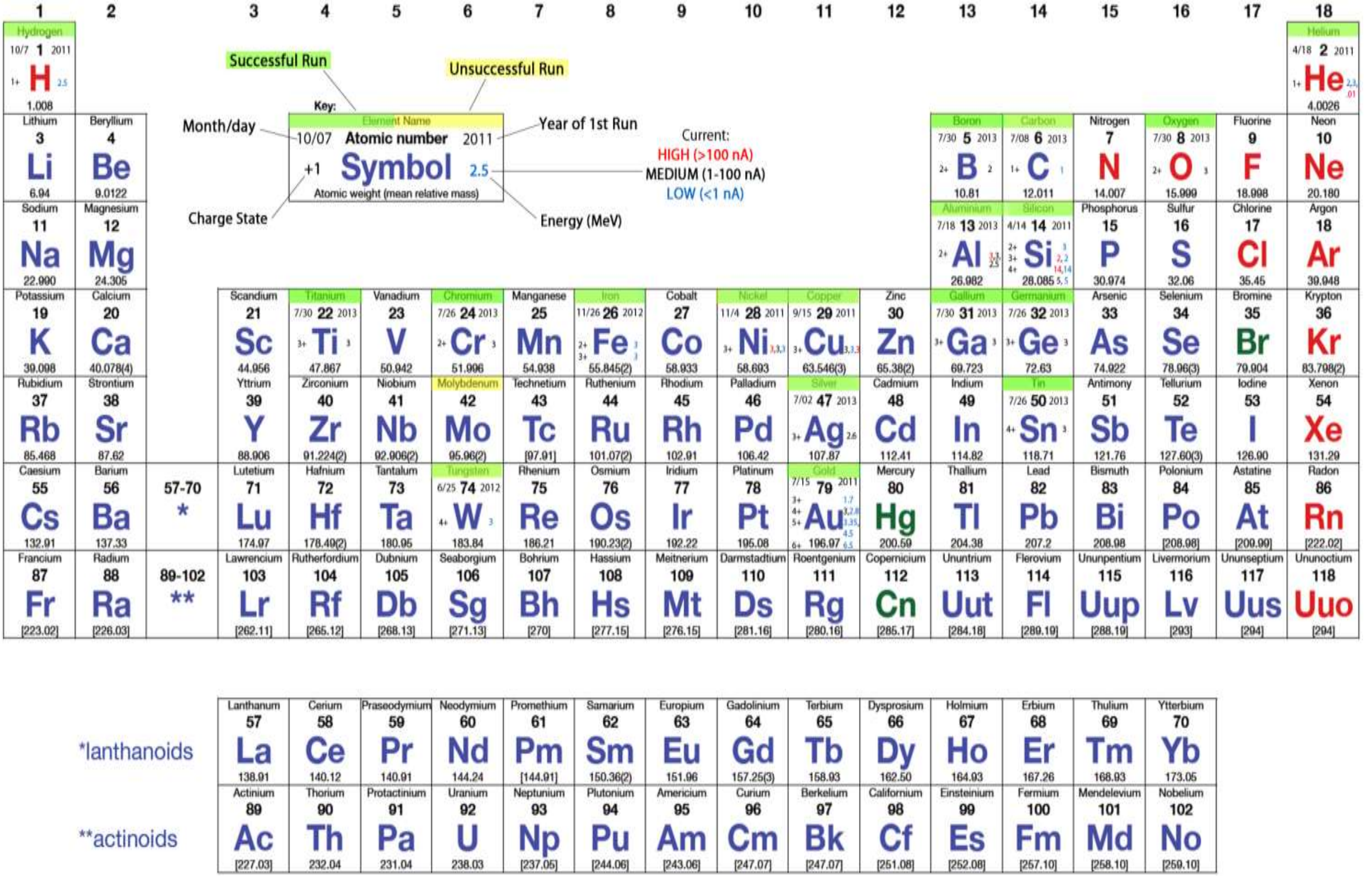
Current Status of the *In situ* TEM Beamline

Collaborators: D.L. Buller & J.A. Scott



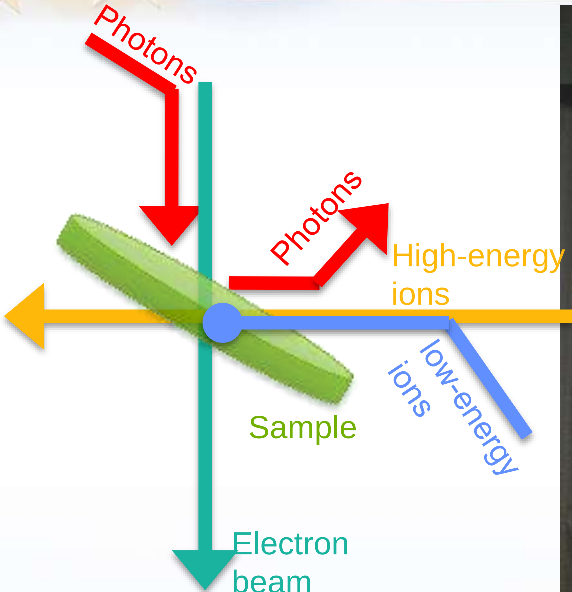
Ion Species Attempted as of 7/23/2013

Collaborators: M. Steckbeck, D. Buller, & D. Bufford

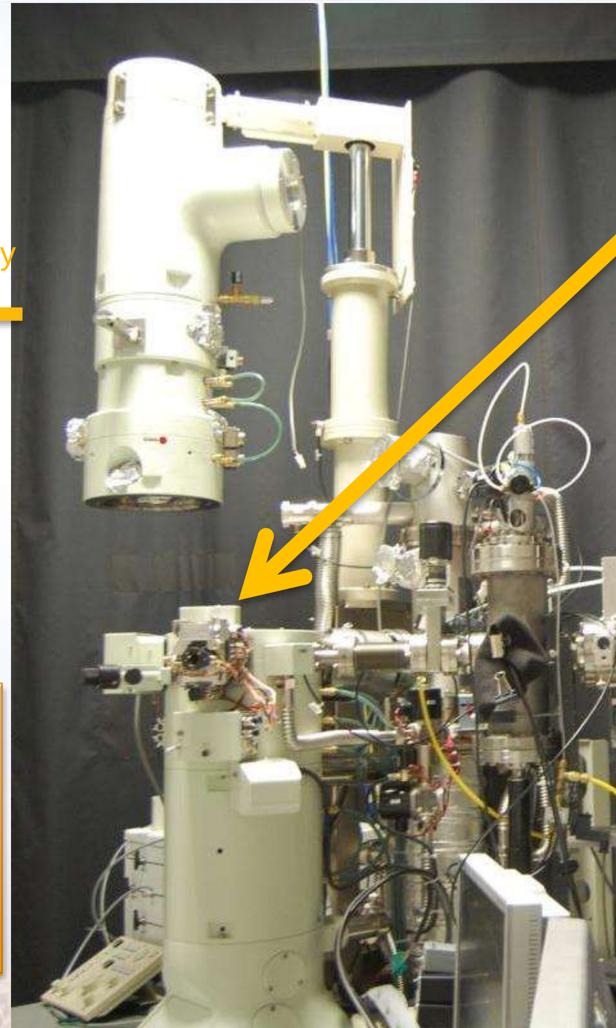


In situ TEM Luminescence

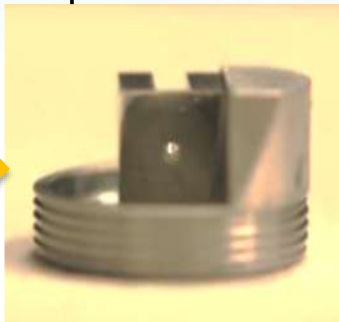
Collaborators: D. Masiel & D. Buller



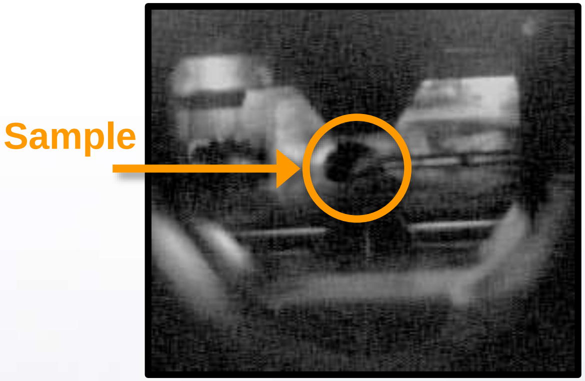
Two optical port were added to the I^3TEM already containing a electron beam and two ion beams, which permits *in situ* TEM luminescence studies



Optical Mirror in TEM



First IBIL in TEM

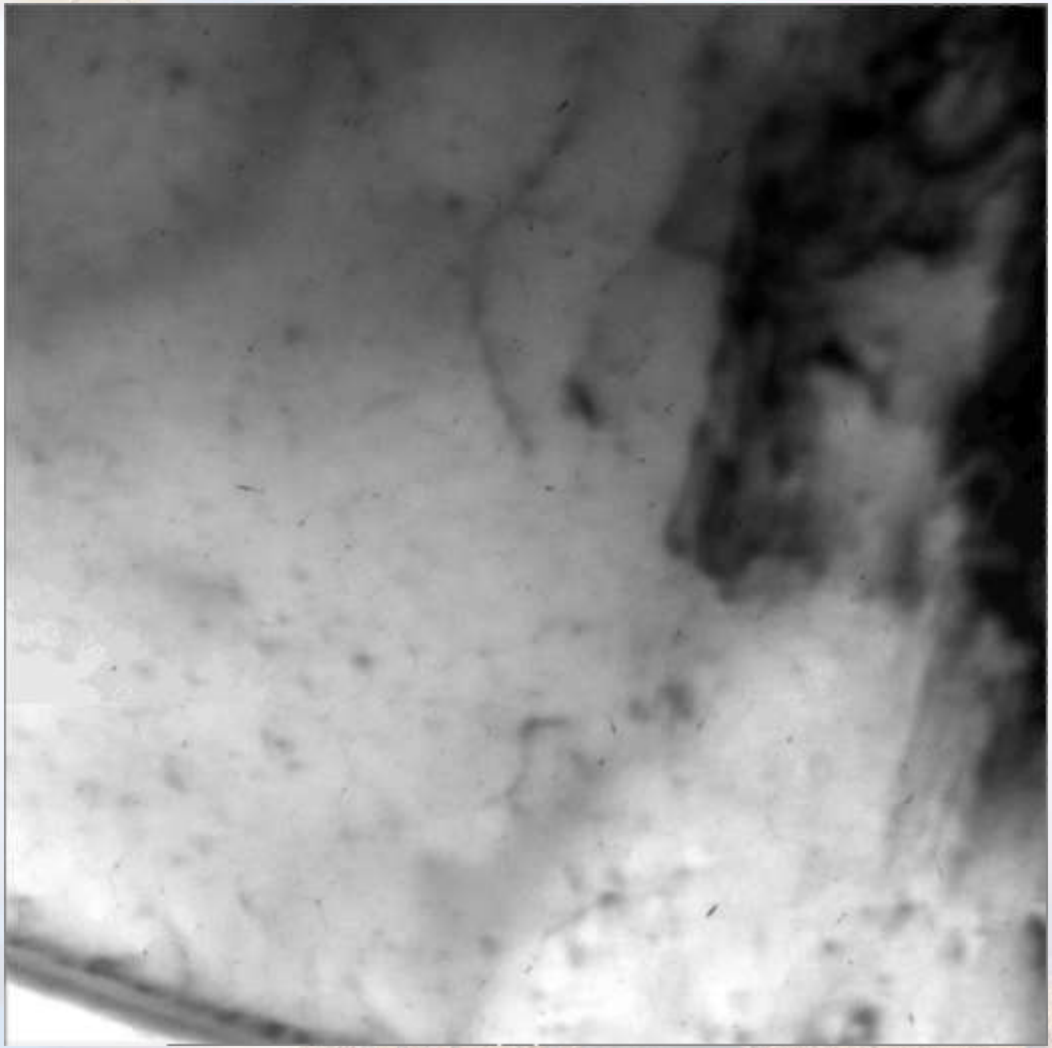


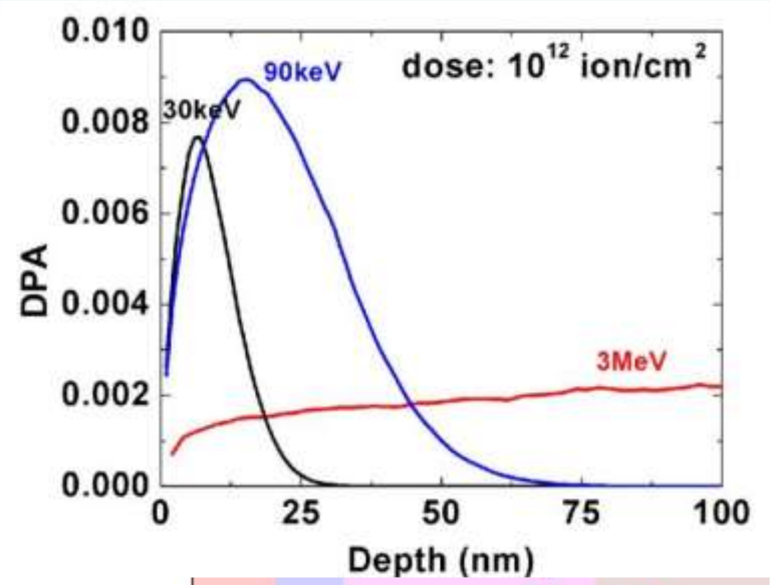
Optical Pathway in an I^3TEM

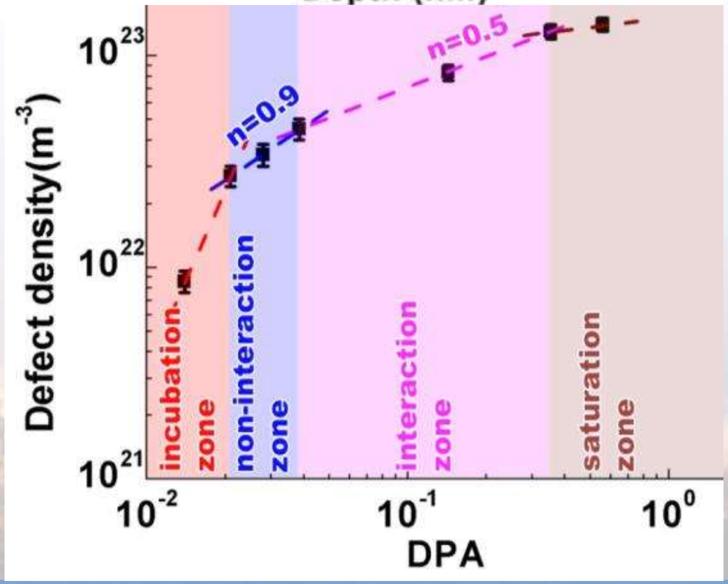
- Angled mirror with bore hole for the electron path was installed above the polepiece
- Another mirror is located just above the ion beams in the beamline
- Two perspective of the sample are possible
- Permits *in situ* IBIL and CL.

Quantifying Defect Evolution

Collaborators: N. Li & A. Misra

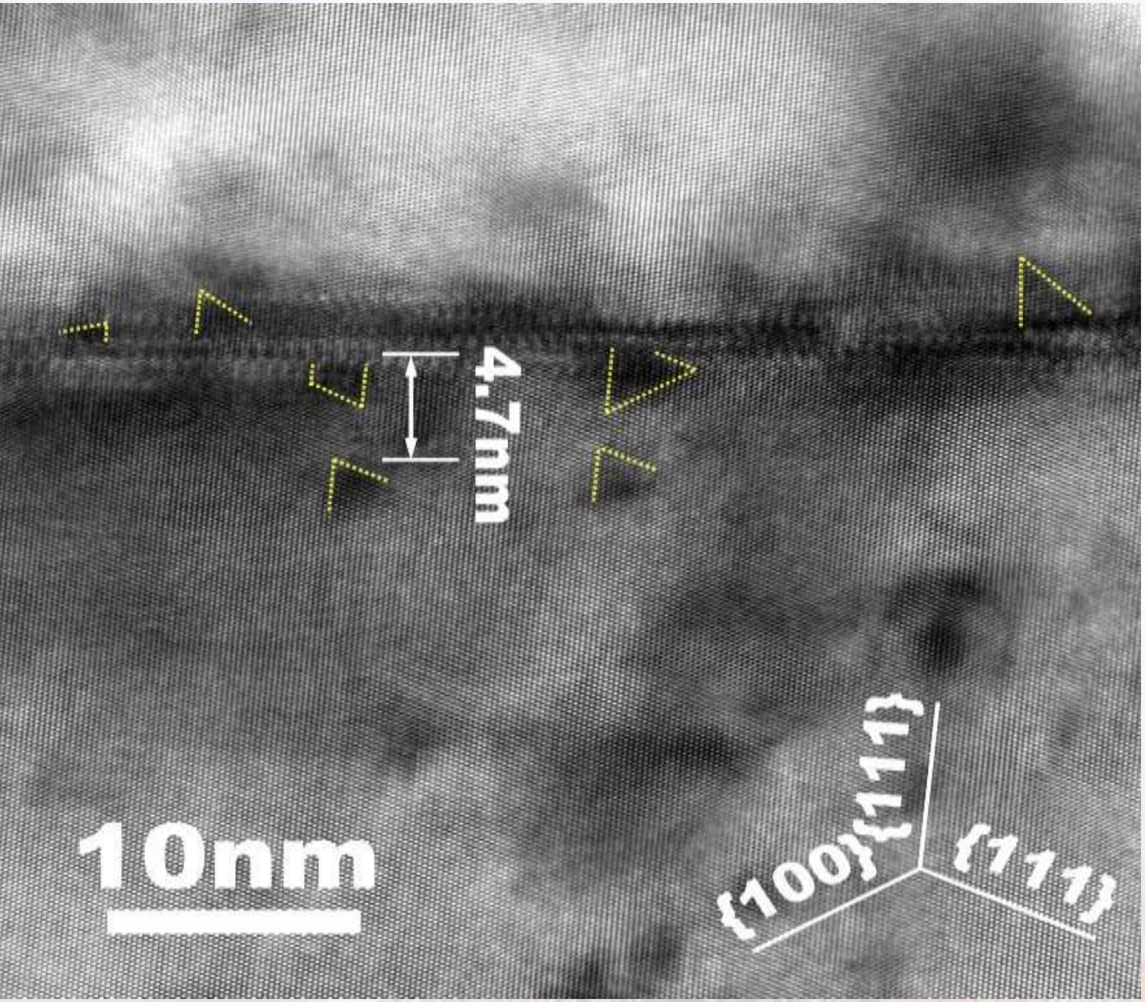


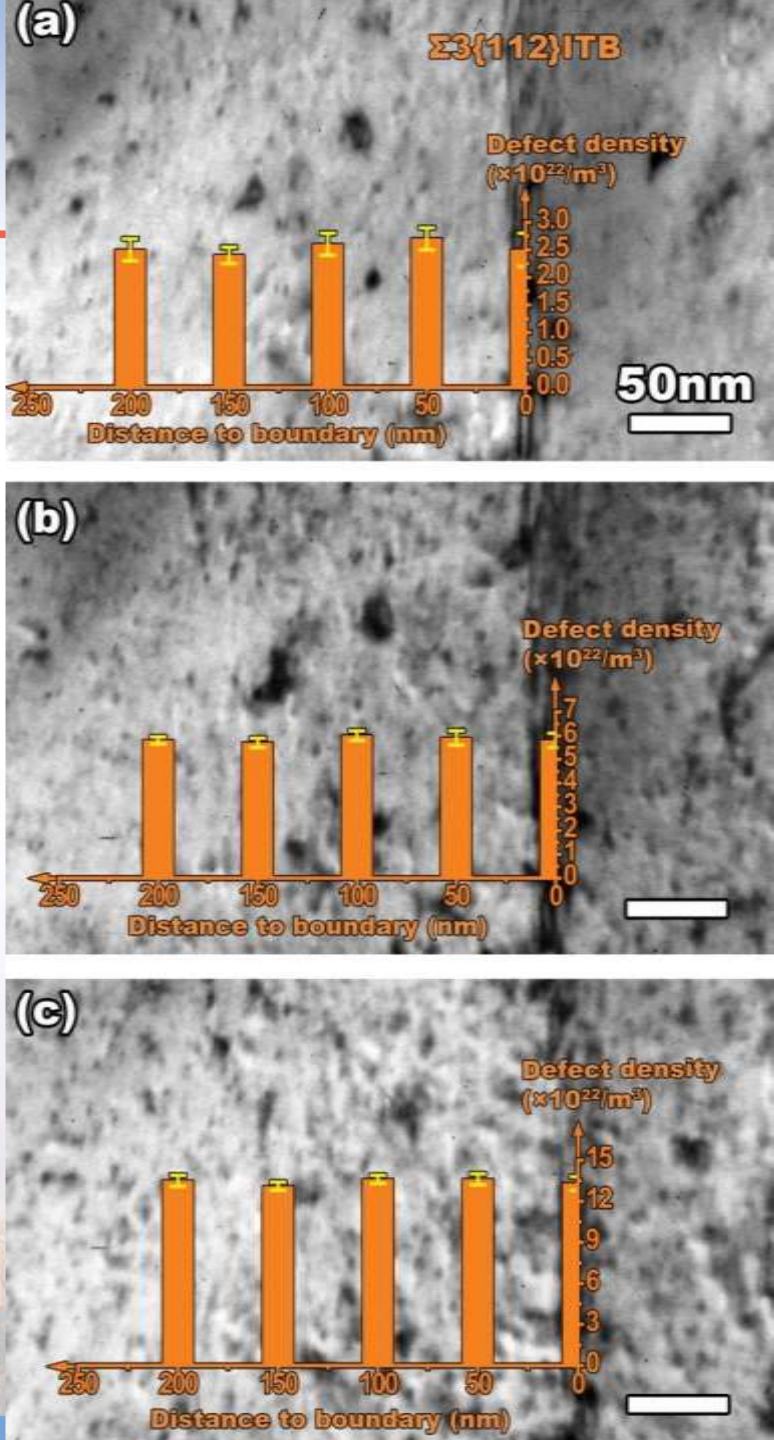




Defects are Altered Little by the Presence of Grain Boundaries

Collaborators: N. Li & A. Misra



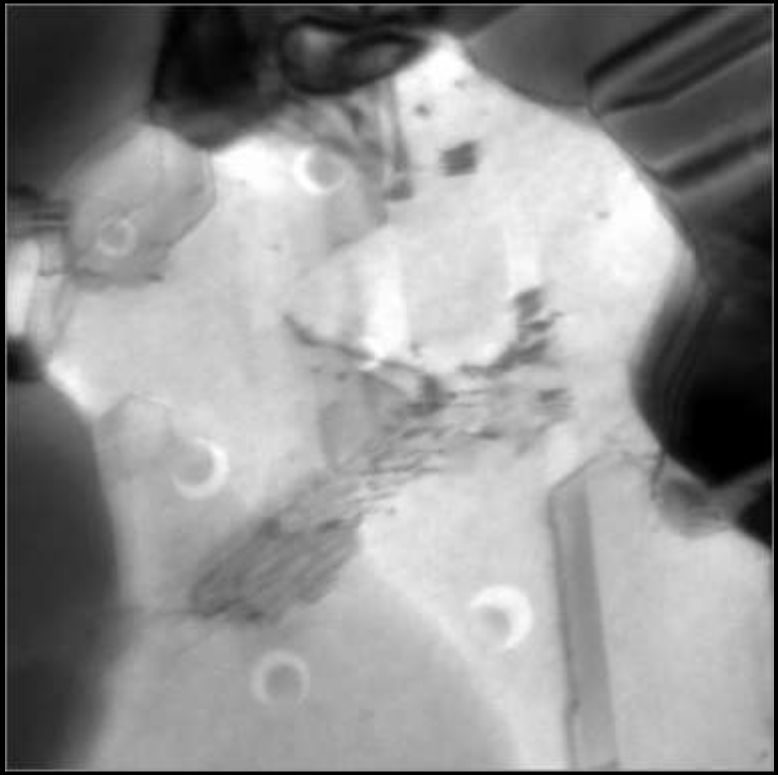


SFT appear to be directly at GB

No change in defect density is observed near GB

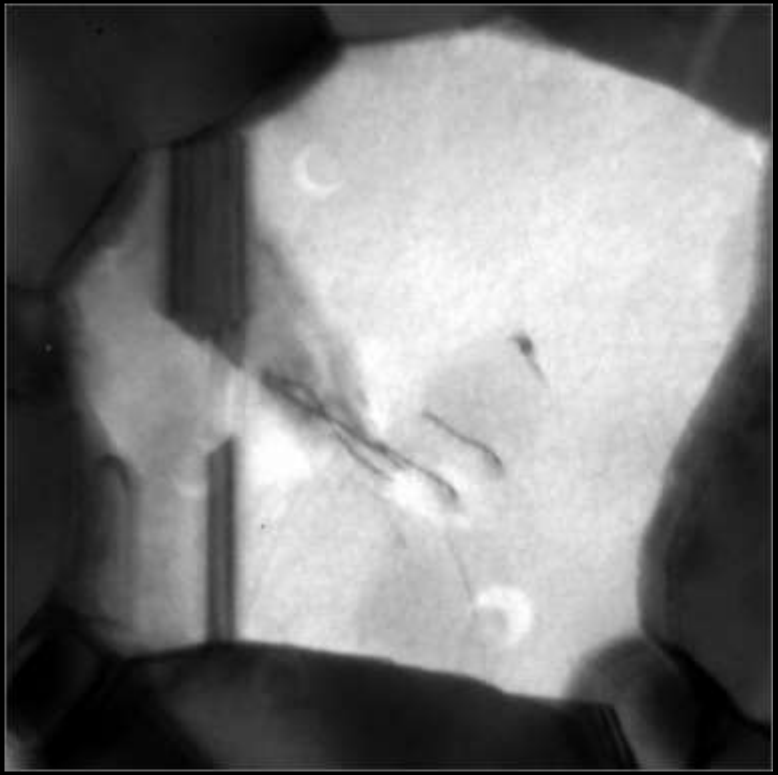
Single Ion Strikes

7.9×10^9 ions/cm²/s



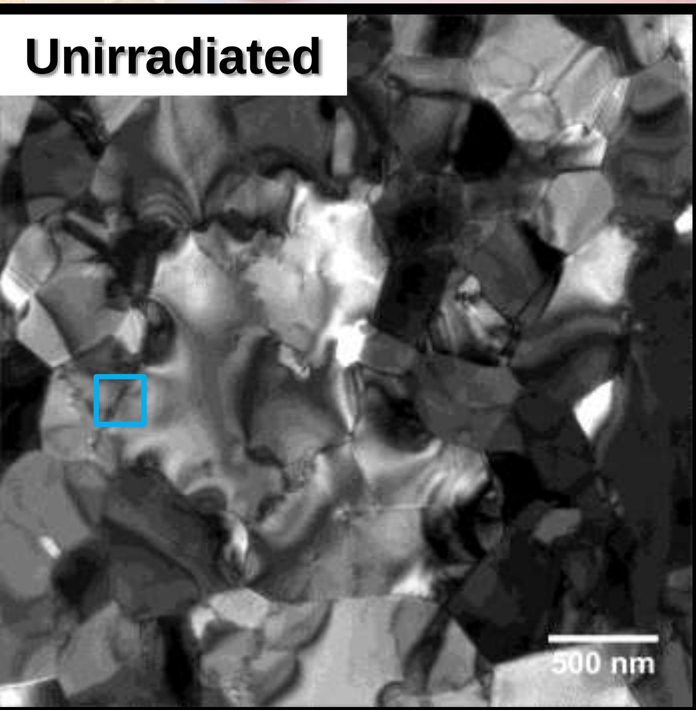
VS

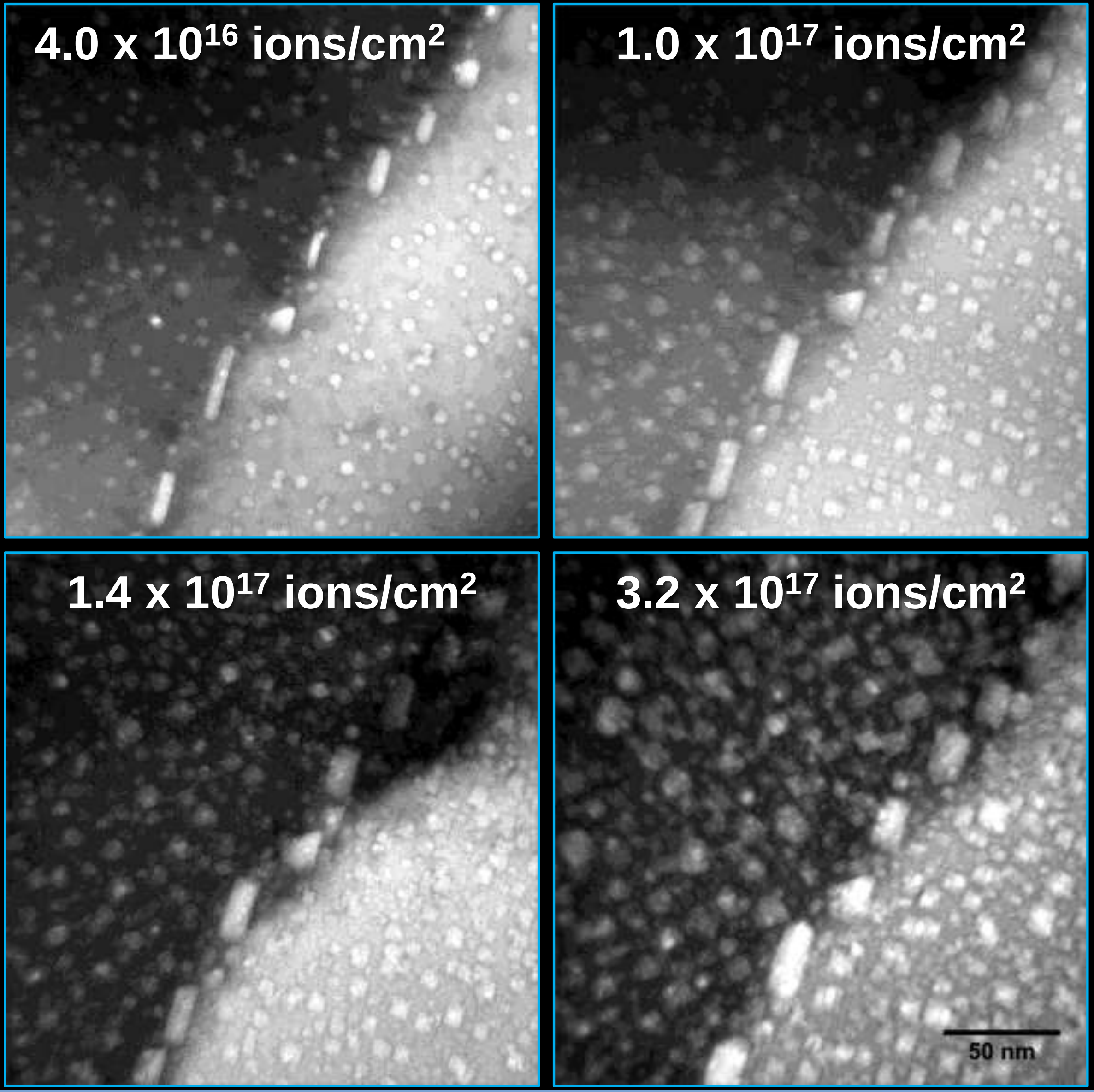
6.7×10^7 ions/cm²/s



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

In situ Implantation

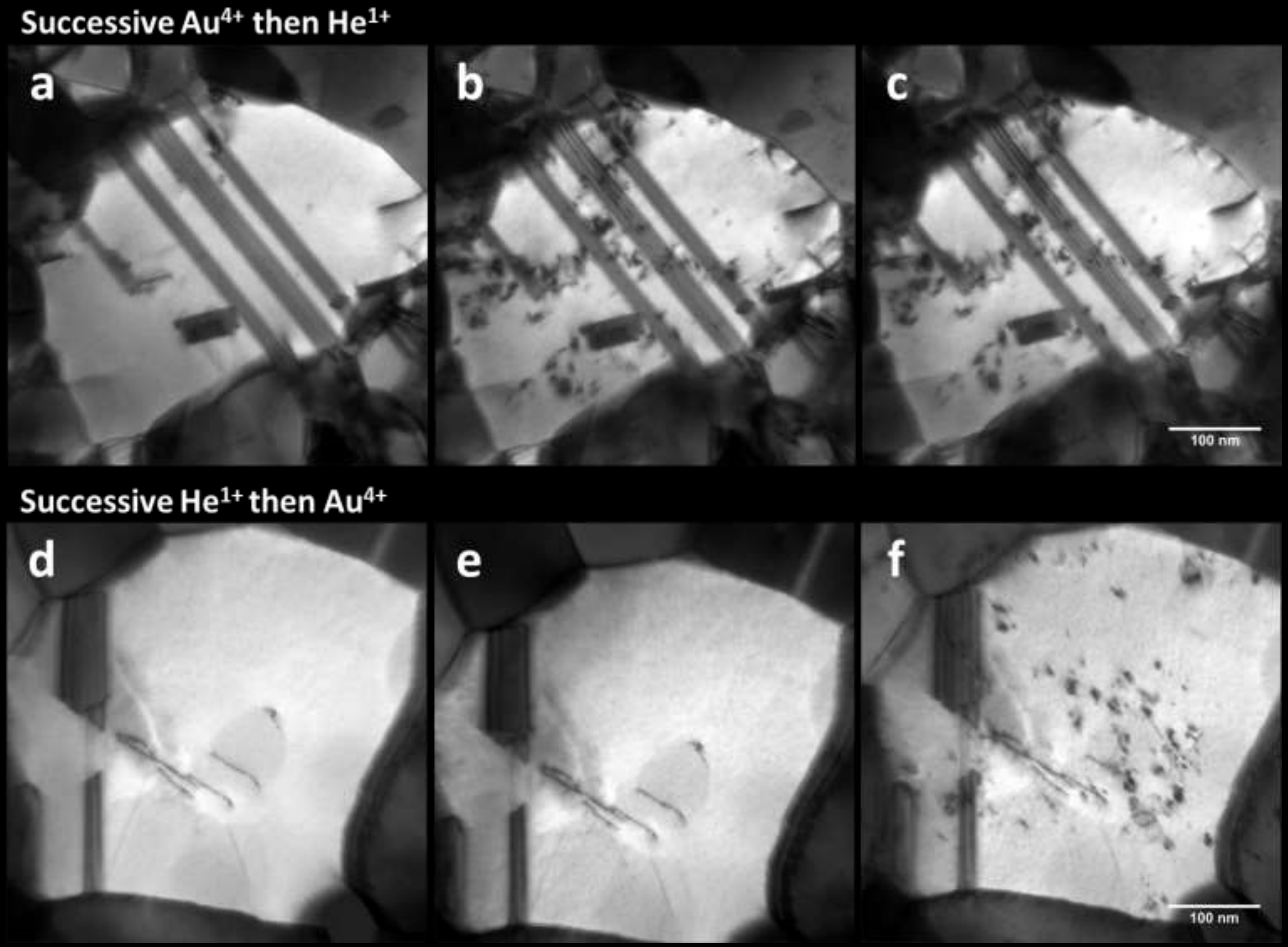




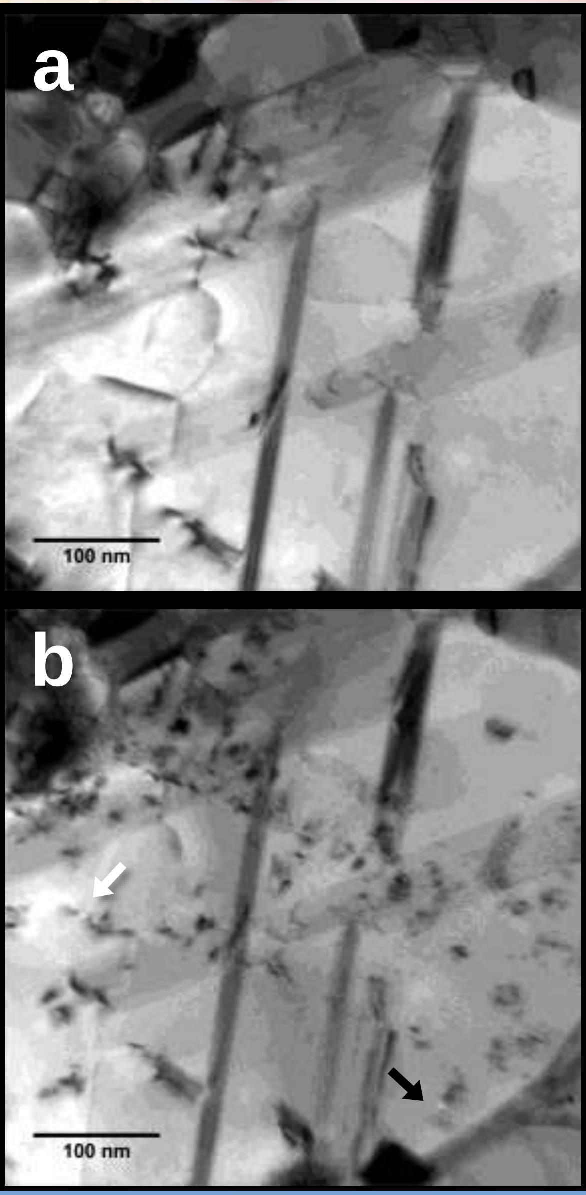
Gold thin-film implanted with 10keV He^{2+}

Result: porous microstructure

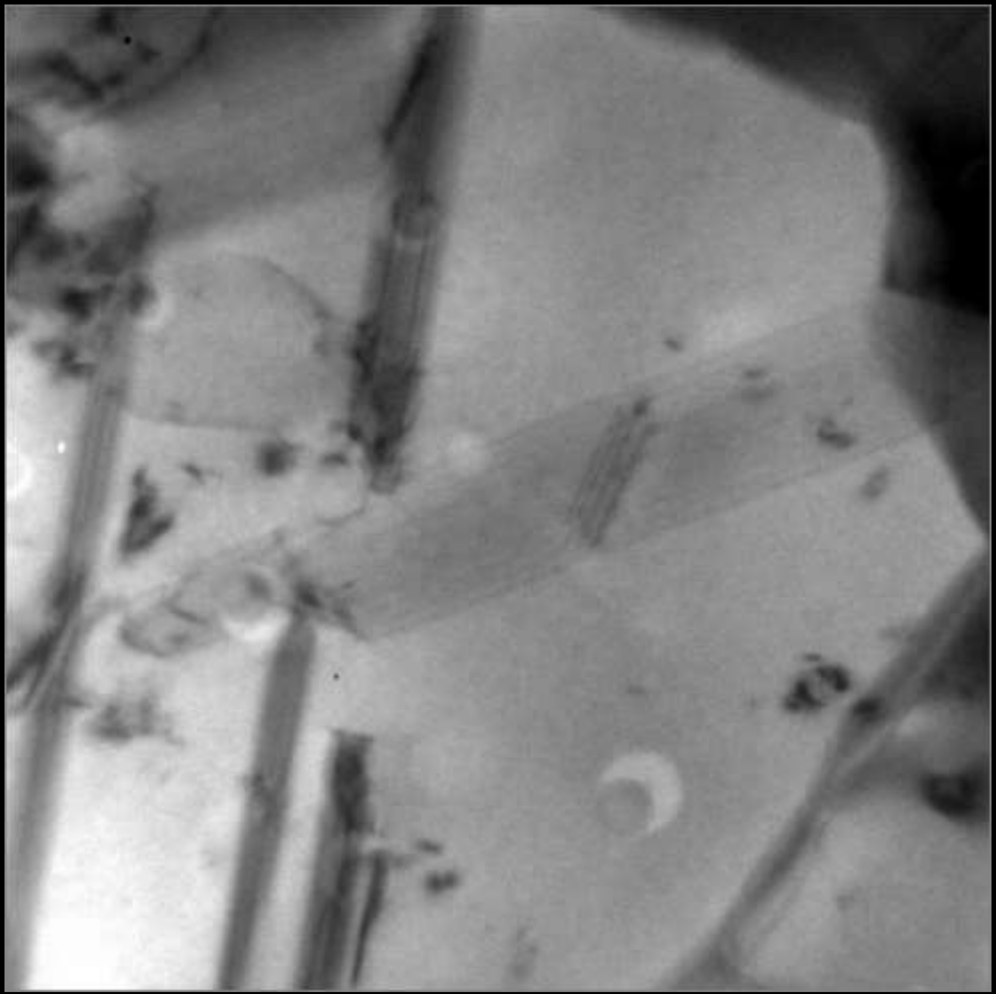
in situ Successive Implantation & Irradiation



In situ Concurrent Implantation & Irradiation



He^{1+} implantation and Au^{4+} irradiation of a gold thin film



Single Ion Strikes During Concurrent Irradiation: Nucleation of Helium Cavities

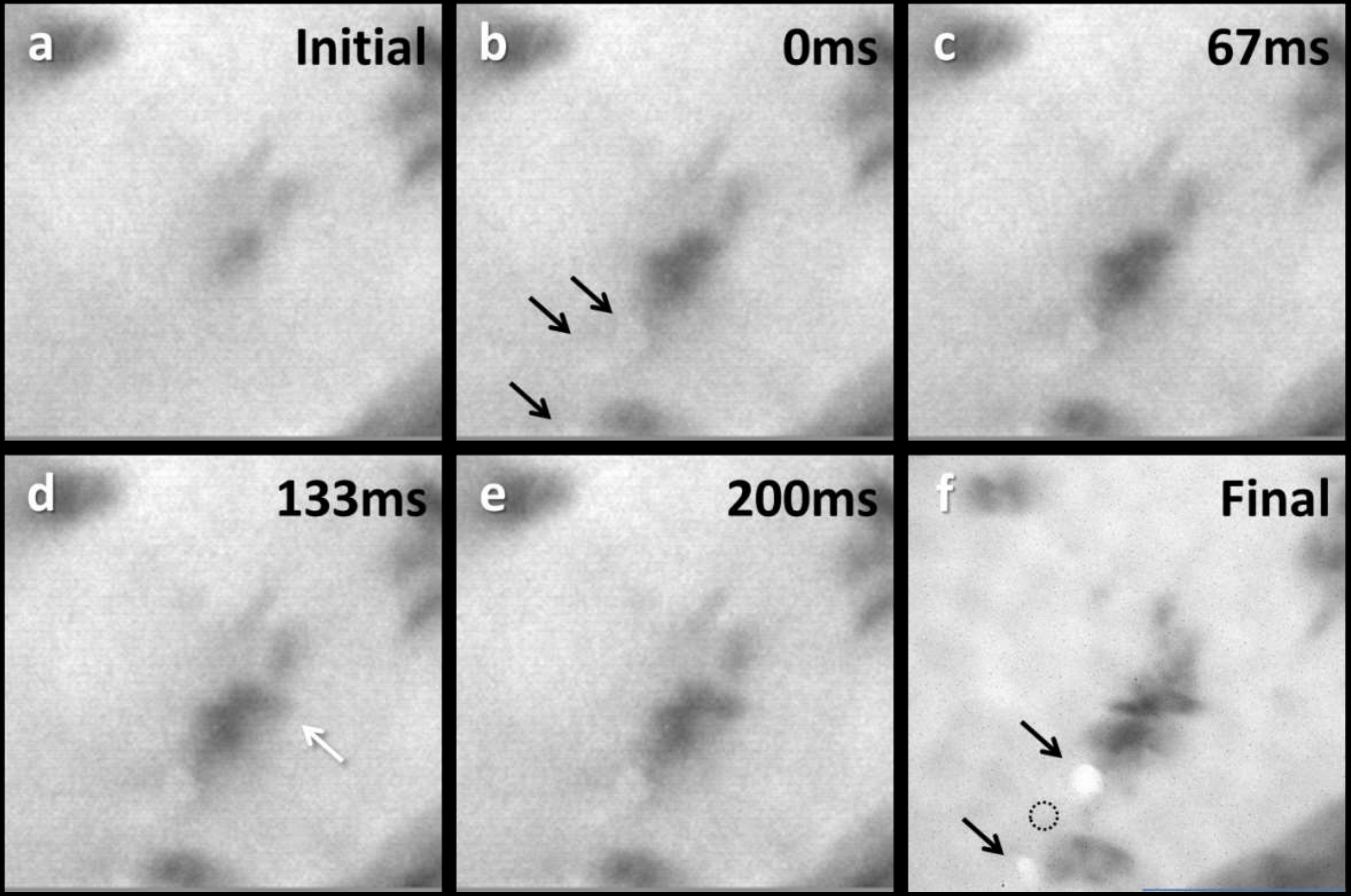
a) Initial microstructure

b) Cascade: Creation of dislocation loops, vacancy clusters, and three cavities

d) Cascade damage still evolving

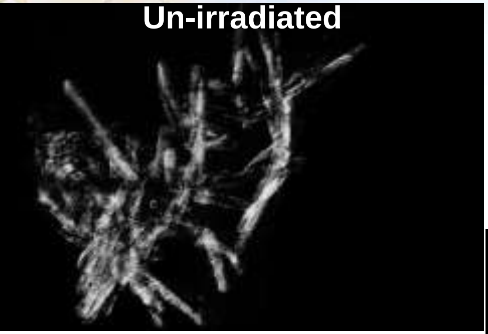
e) Apparent stability

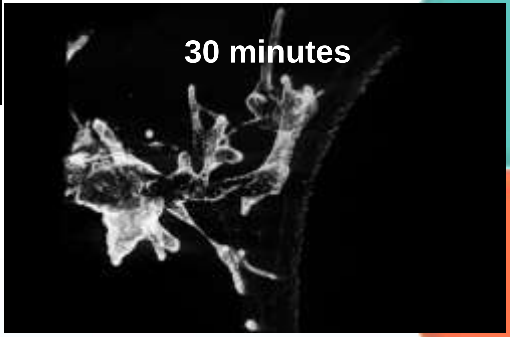
f) Final microstructure: Only two remaining cavities

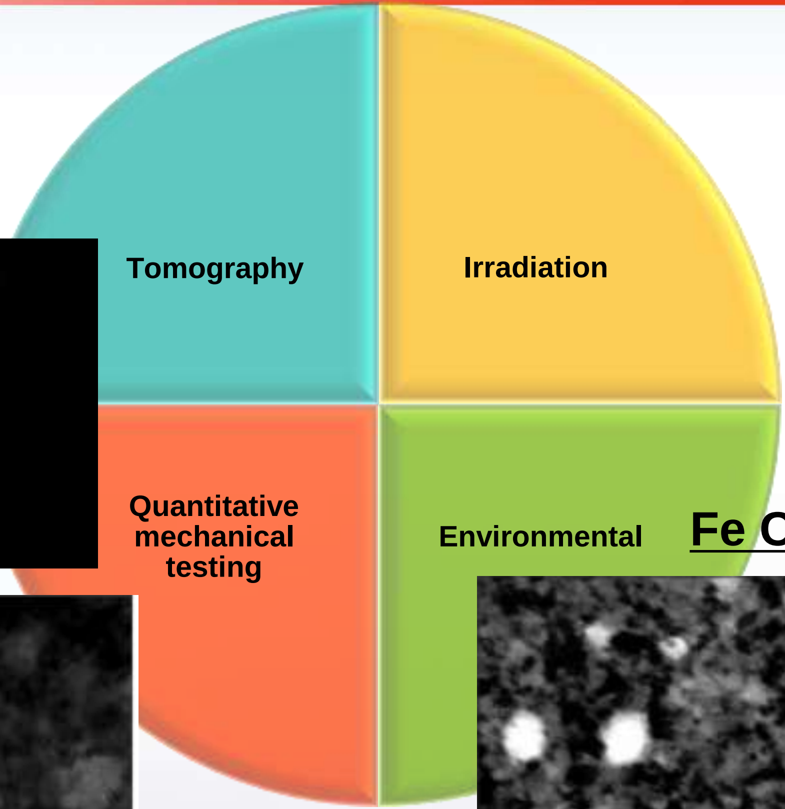


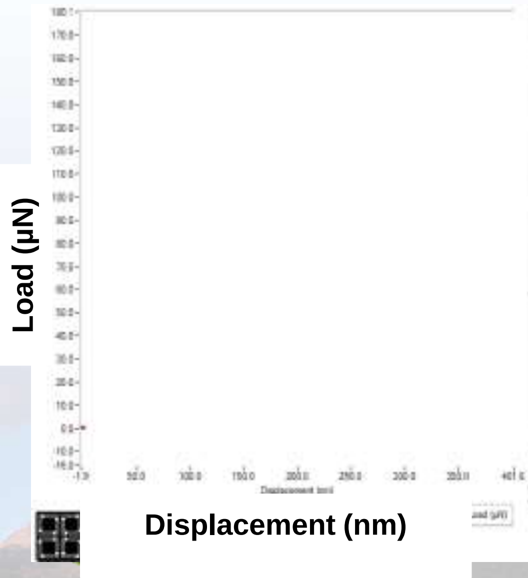
Combination of *in situ* Capabilities

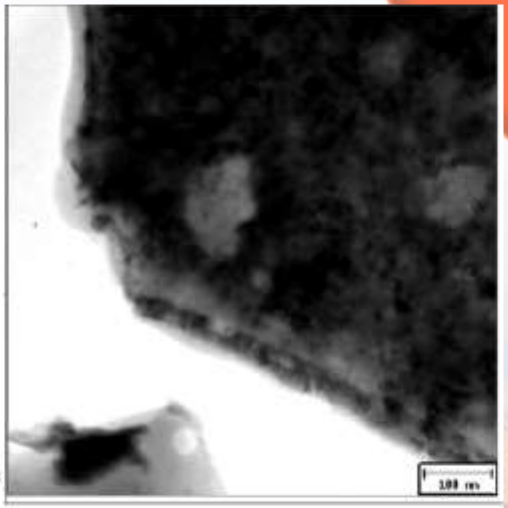
Contributors: S.M. Hoppe, B.A. Hernandez-Sanchez, T. Boyle, D. Gross, J. Kacher, & I.M. Robertson



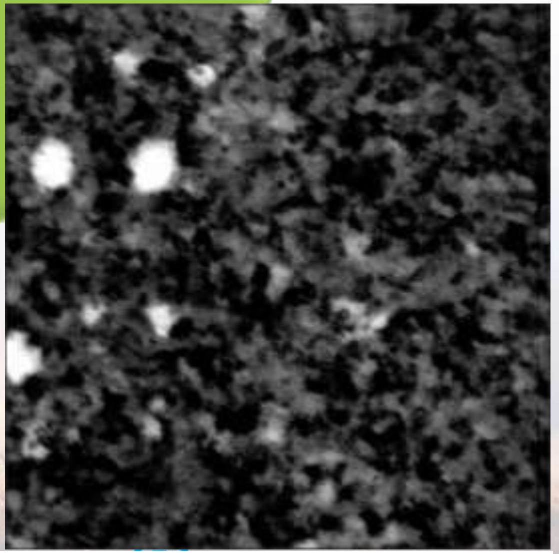








Fe Corrosion

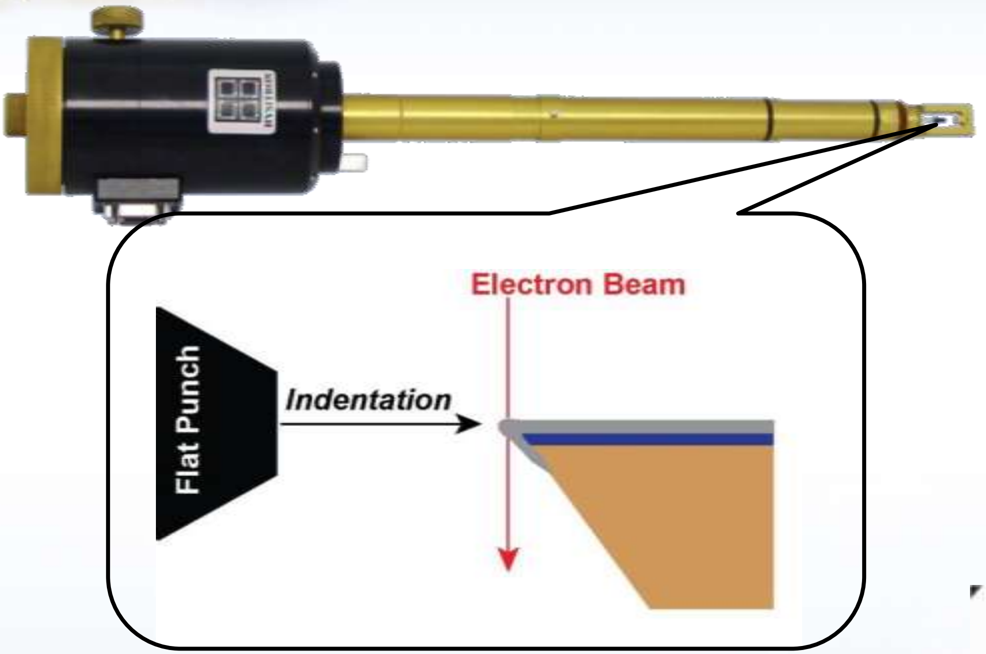


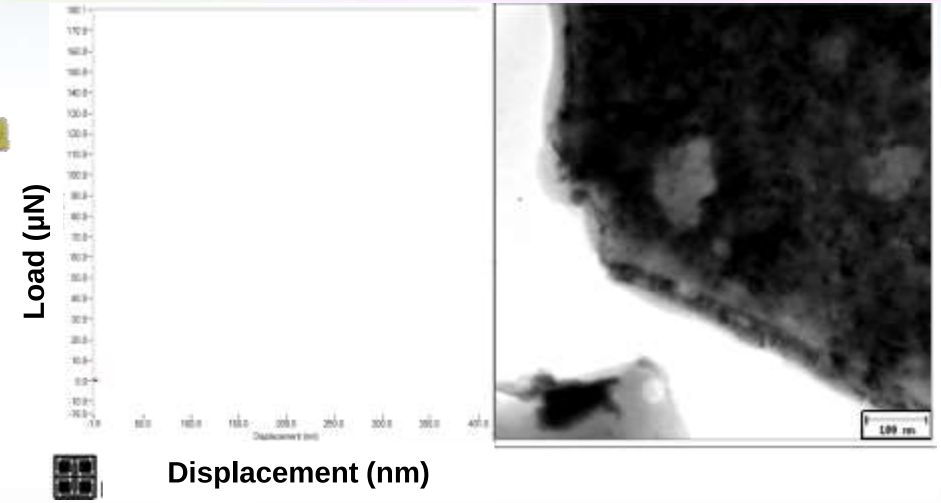
Dilute flow of acetic acid over 99.95% nc-PLD Fe

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

In situ TEM Quantitative Mechanical Testing

Contributors: H. Bei, & E.P. George

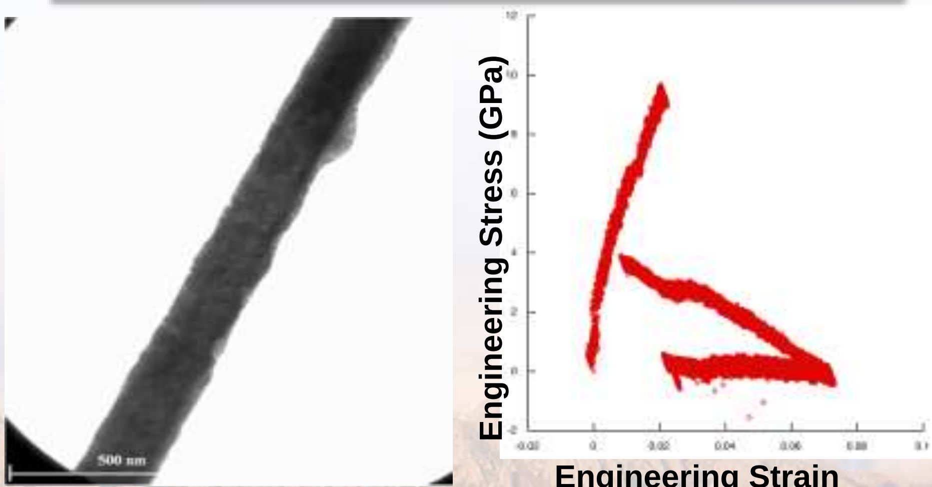




Fundamentals of Mechanical Properties

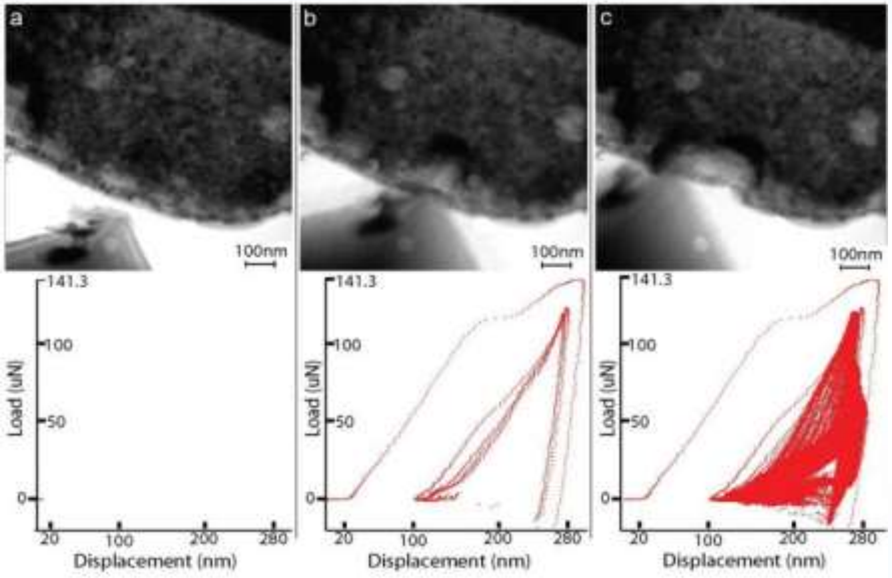
Range of Mechanical Testing Techniques

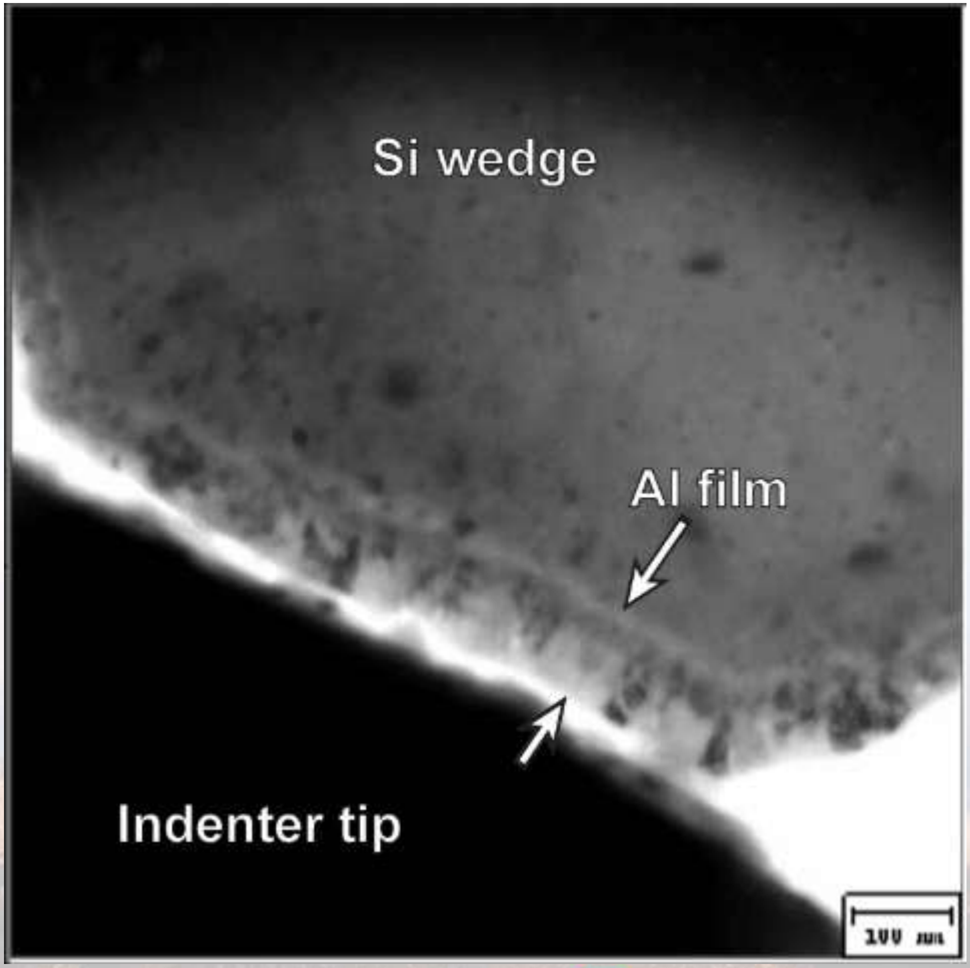
- Indentation
- Compression
- Tension
- Bending
- Wear
- Fatigue
- Creep



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

In situ TEM Cyclic Loading

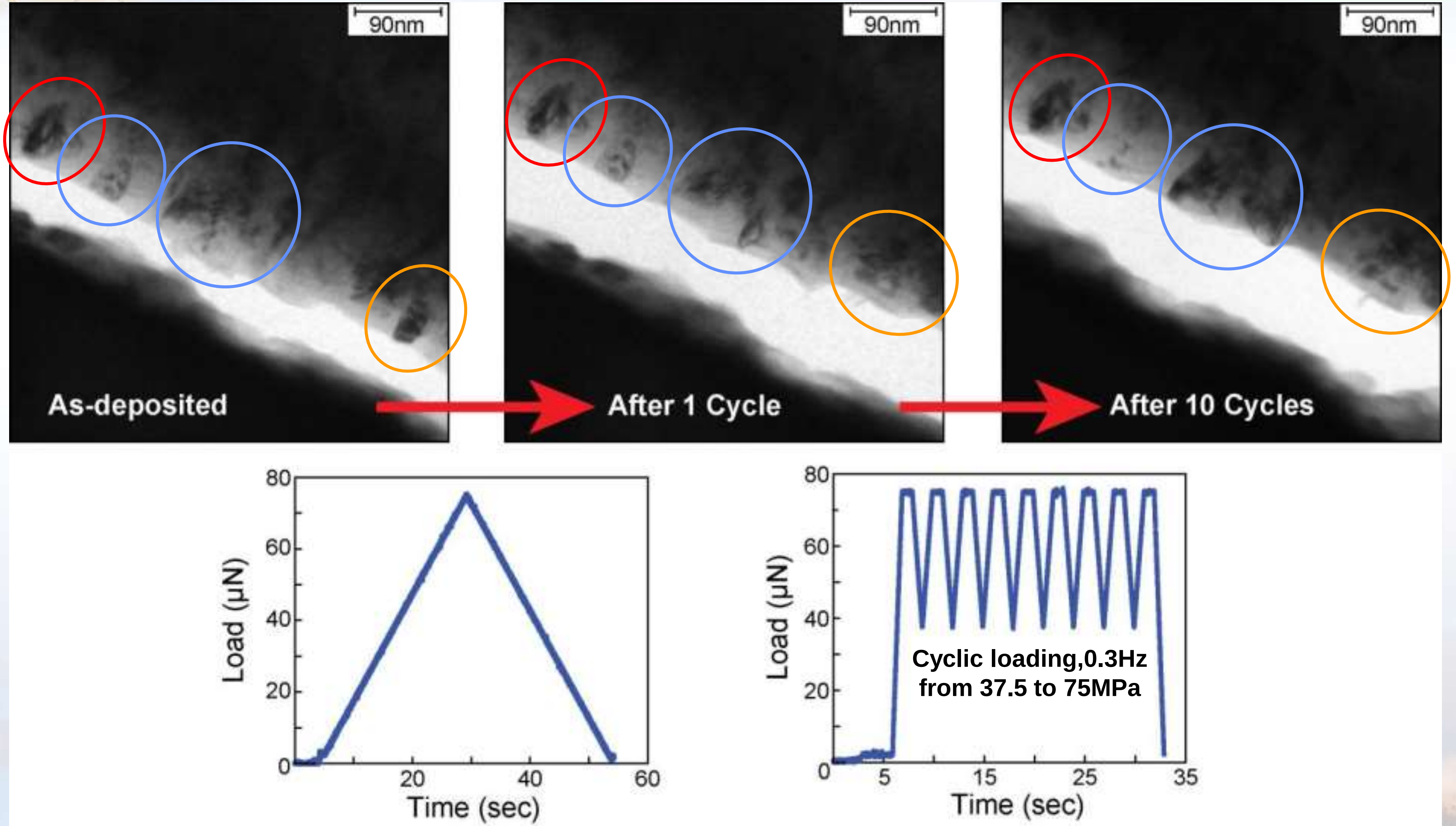




<u>Cyclic contact effect on structure</u>

- **Associate change in local hardness or compression strength with corresponding structural evolution in either:**
  - **Indentation of nanocrystalline Cu films**
  - **Compression of nanocrystalline Al films**

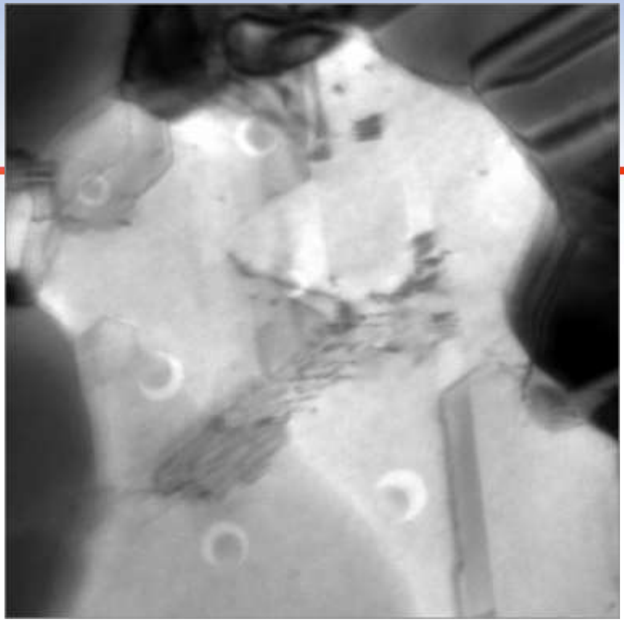
No, Monotonic, or Cyclic Evolution in Nanocrystalline Al

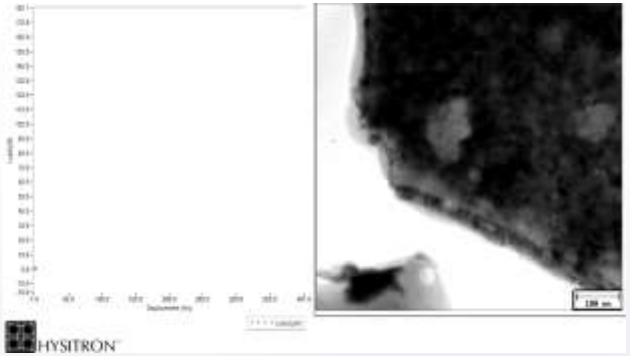


375 MPa nominal contact stress

Summary & Future Interests

- Sandia's I^3TEM has a range of *in situ* capabilities for extreme enviroments
- Have applied the current I^3TEM capabilities to various nanocrystalline metals for:
  - Irradiation
    - Single ion strikes
    - Defect migration
  - Implantation
    - Bubble formation
    - Defect cavity interactions
  - Cyclic loading
    - Grain growth occurs under different cycles
  - And combinations thereof





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

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