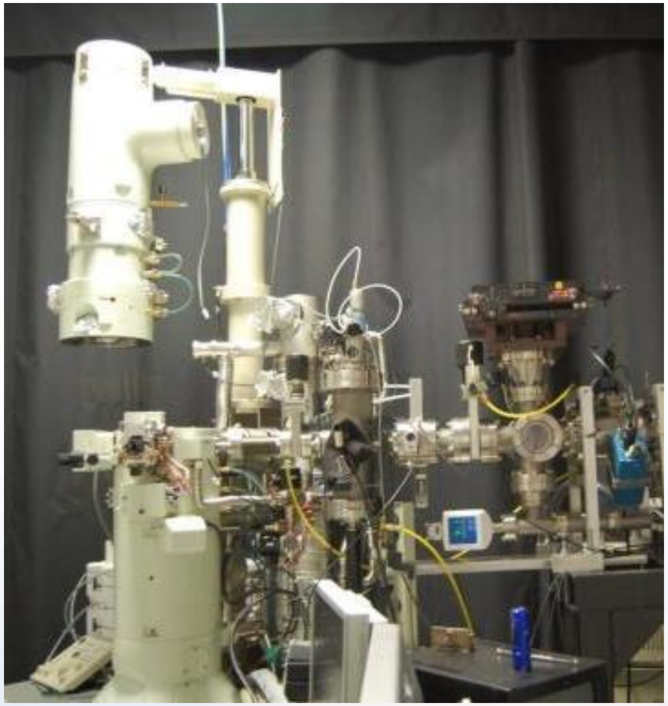
Development of Advanced Ion Beam End Stations & Their Potential for Informing Models

SAND2014-17713PE

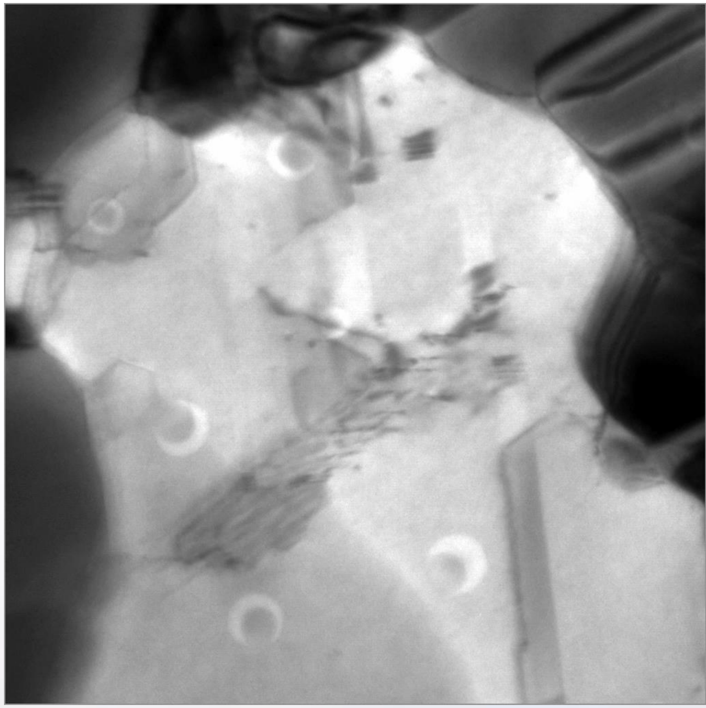
K. Hattar

Ion Beam Lab at Sandia National Laboratories

September 17, 2014



Recent advancements in small scale testing, thermal measurements, *In situ* TEM microscopy provides capabilities to investigate the structural evolution that occurs due to various extreme environments and combinations thereof



Collaborators:

- **IBL: D.C. Bufford, D. Buller, C. Chisholm, B.G. Clark, B.L. Doyle, S. H. Pratt, & M.T. Marshall**
- **Sandia: B. Boyce, T.J. Boyle, P.J. Cappillino, J.A. Scott, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, E. Carnes, J. Brinker, D. Sasaki, J.A. Sharon, T. Nenoff, W.M. Mook**
- **External: A. Minor, L.R. Parent, I. Arslan, H. Bei, E.P. George, P. Hosemann, D. Gross, J. Kacher, & I.M. Robertson**

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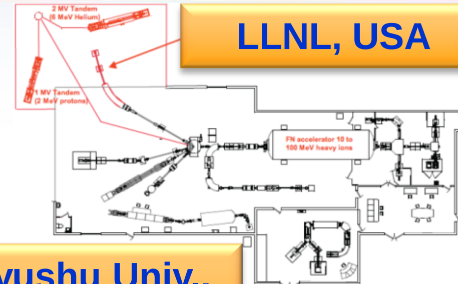
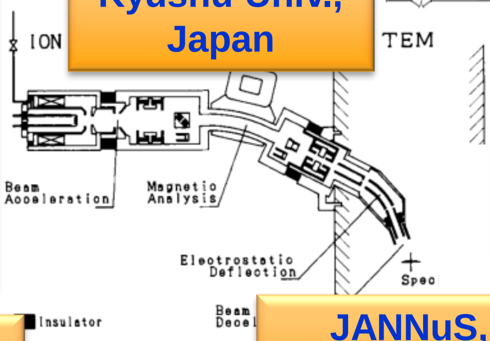
Examples of Proposed and Developed Ion beam Facilities Around the World with Complex End Stations

IVEM, ANL, USA

LLNL, USA

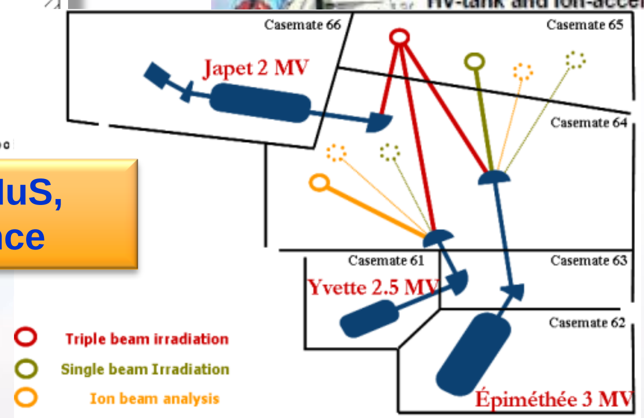


Kyushu Univ., Japan



Hokkaido Univ. Japan

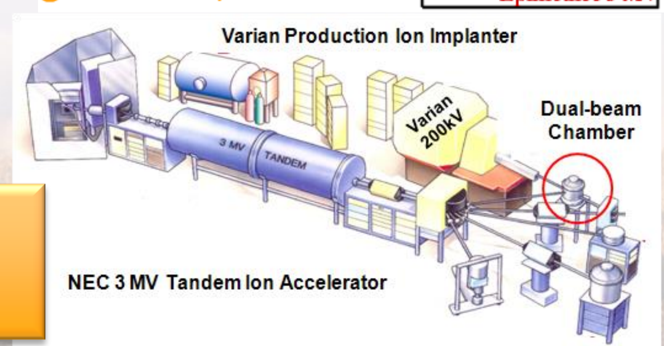
JANNuS, France



IBL, SNL, USA

MAIMI, Univ. Huddersfield, UK

IBML, LANL, USA



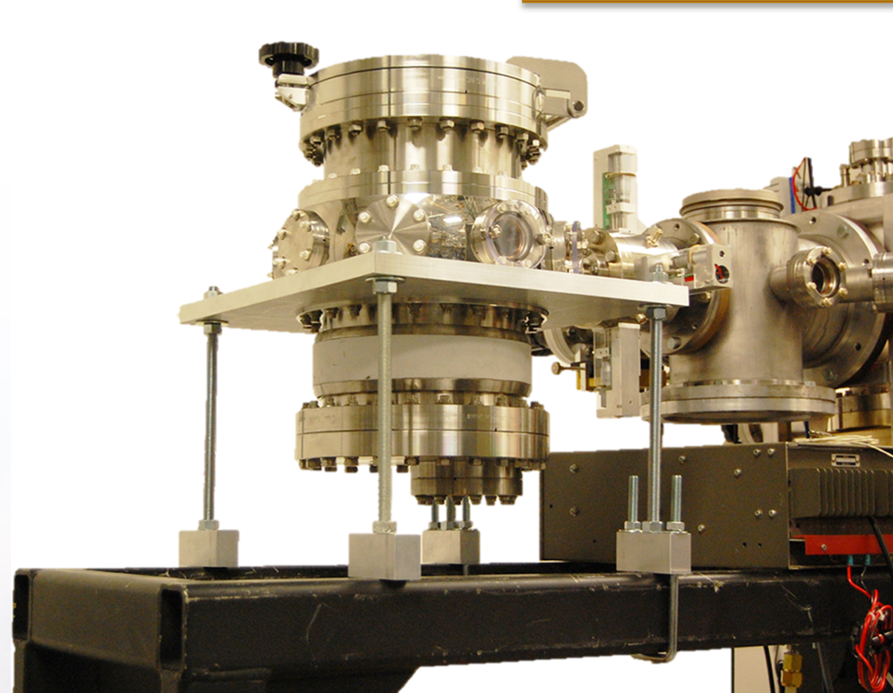
Extreme Environments at Ion Beam End Stations

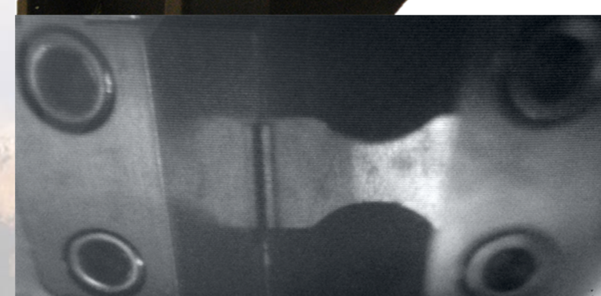
Courtesy of: M. Caro, Y.Q. Wang, J. Tesmer, M. Bourke, S. Qvist, B. McWaters and P. Hosemann

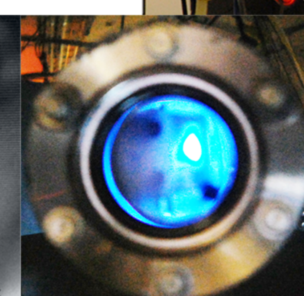
Low Dose Neutrons, SNL, USA

Corrosion (ICE-2), LANL, USA

Creep SNL, USA



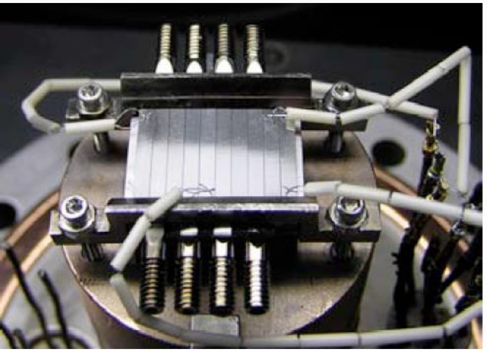


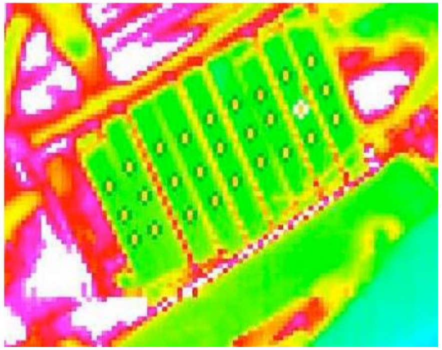


In-situ Non-destructive Characterization

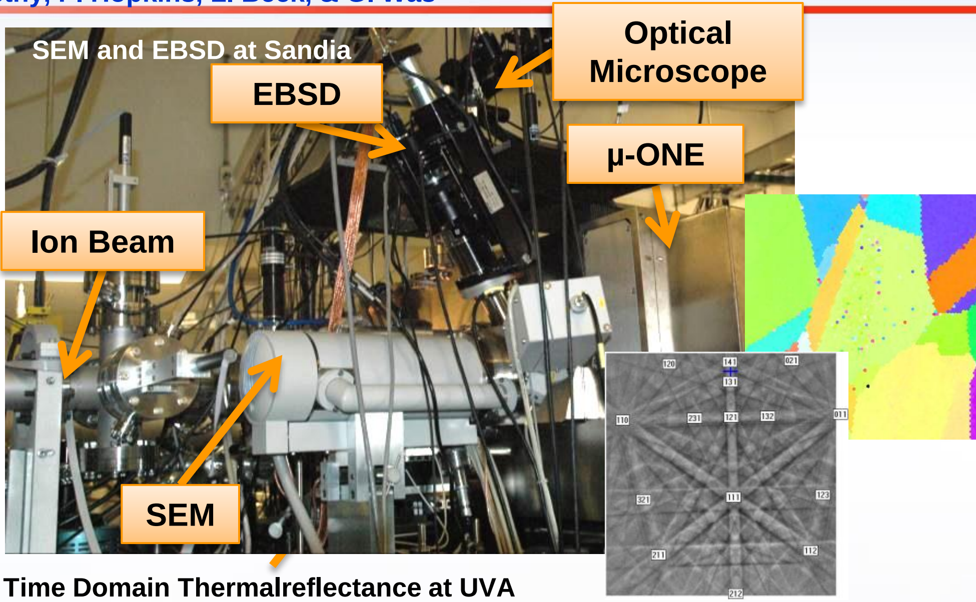
Courtesy of: J. Henry, G. Vizkelethy, P. Hopkins, L. Beck, & G. Was

Thermal Imaging at University of Michigan

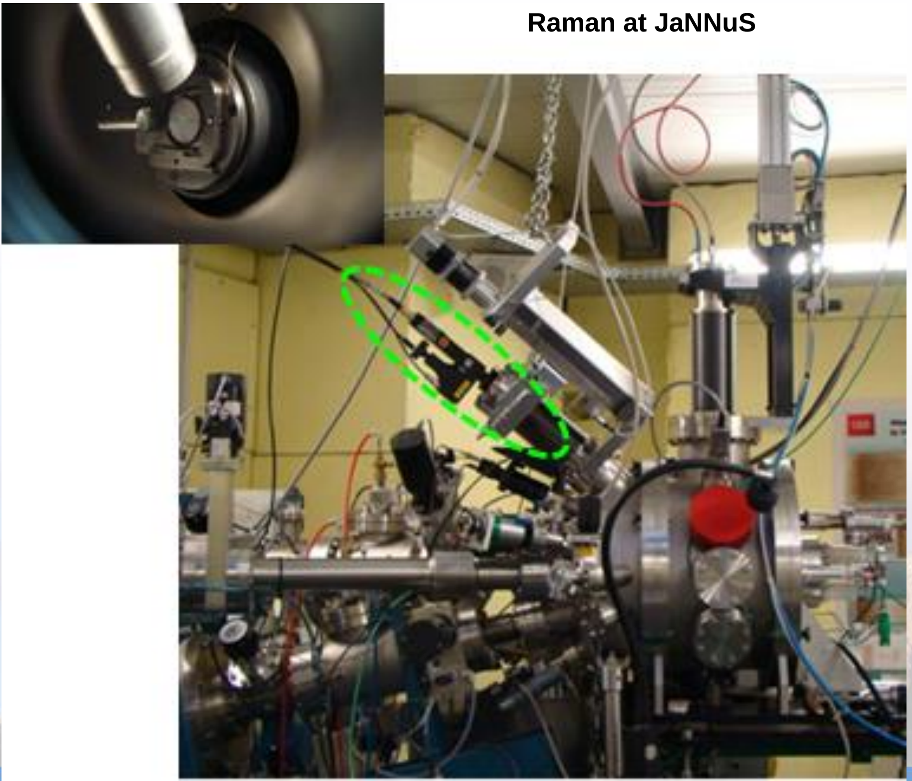




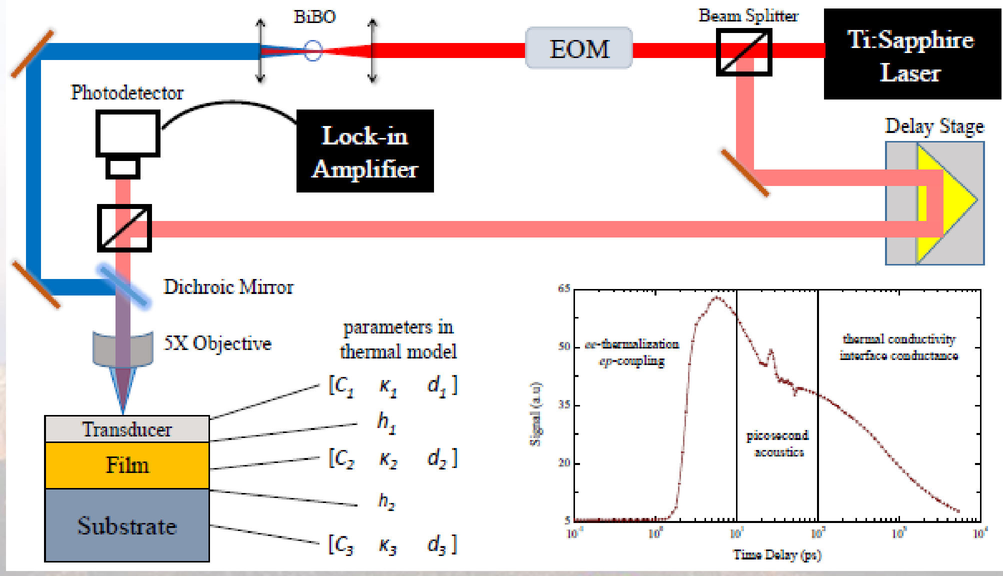
SEM and EBSD at Sandia



Raman at JaNNuS

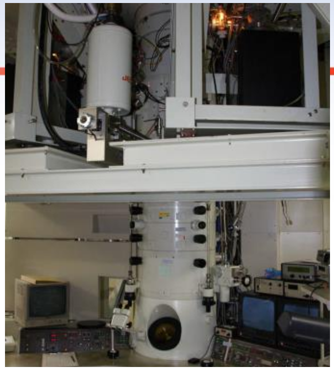


Time Domain Thermalreflectance at UVA



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

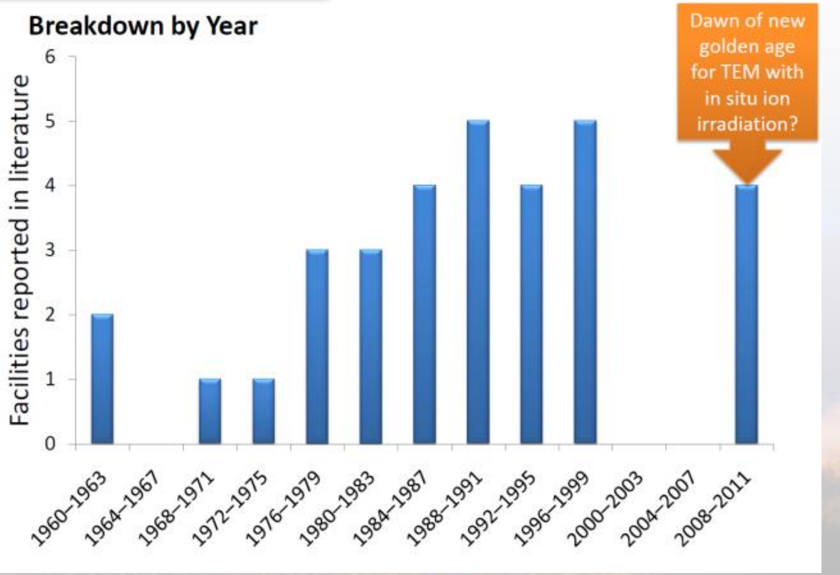
1968
First TEM beamline combination by Thackery, Nelson, and Sansom at AERE Harwell, UK

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)



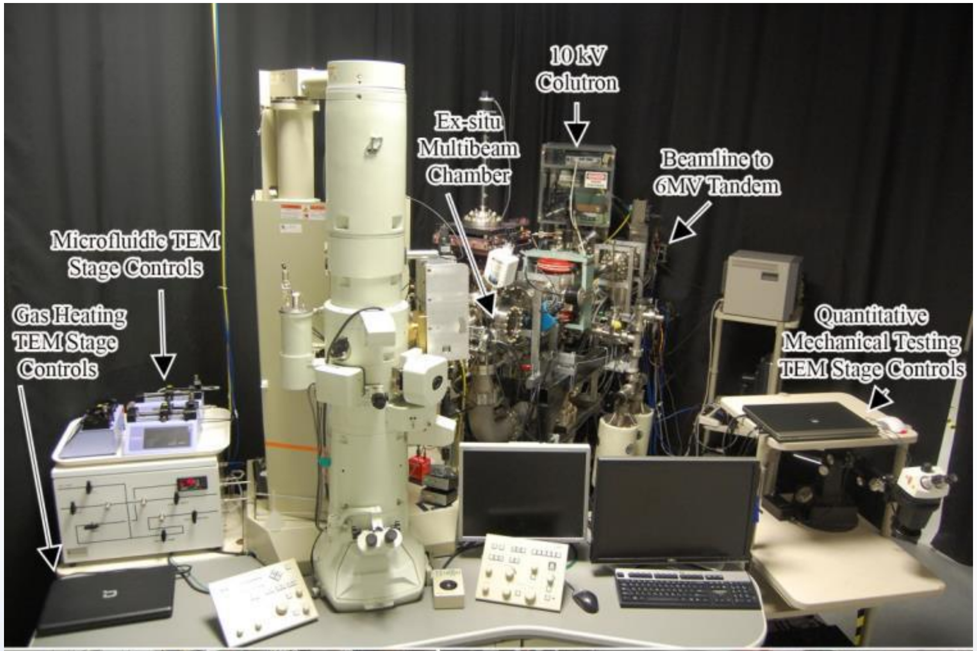
"The direct observation of ion damage in the electron microscope thus represents a powerful means of studying radiation damage"

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

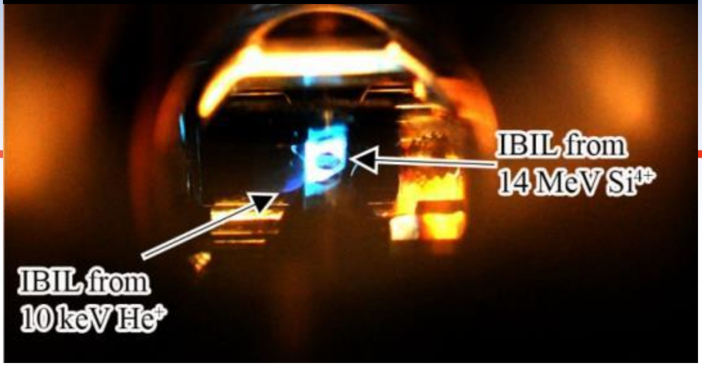
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem

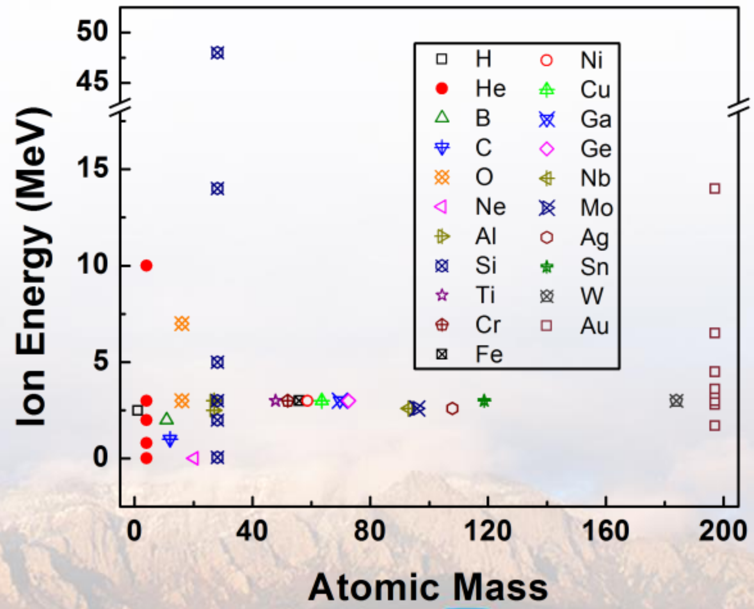


IBIL from a quartz stage inside the TEM



Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution

Ion species & energy introduced into the TEM



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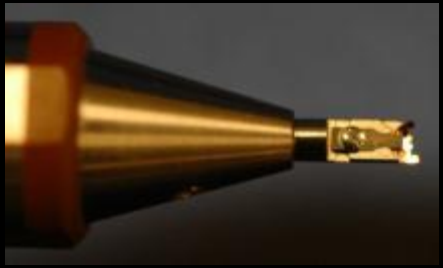
Advanced Microscopy Techniques Applied to Nanoparticles in Radiation Environments

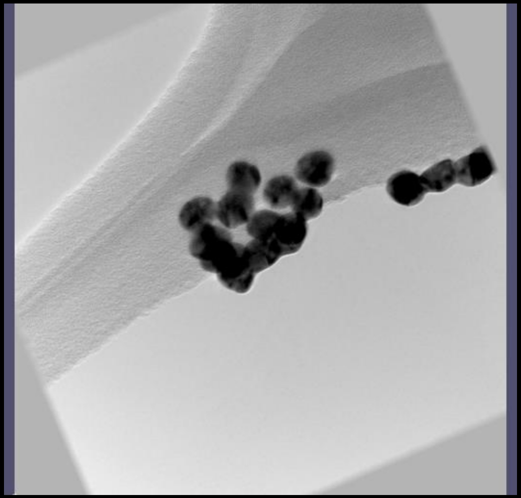
Collaborators: S.M. Hoppe & T.J. Boyle

In situ Ion Irradiation TEM (I³TEM)

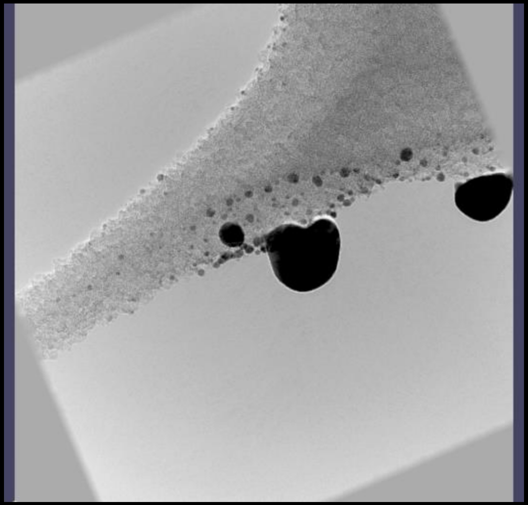
Hummingbird tomography stage



Aligned Au NP tilt series - unirradiated

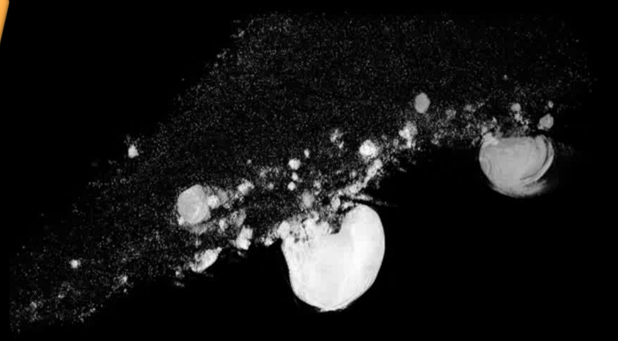


Aligned Au NP tilt series - irradiated



Unirradiated Au NP model

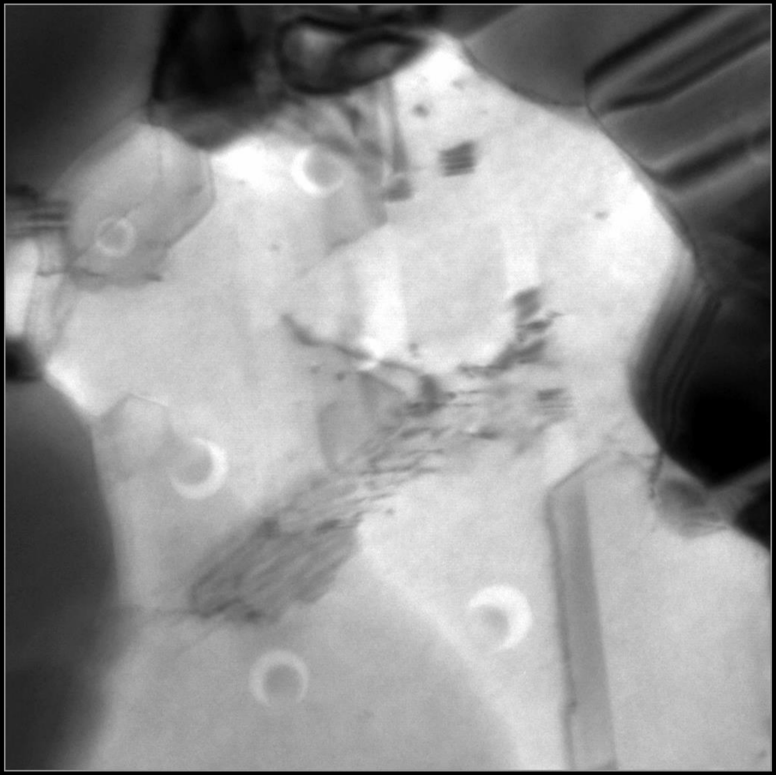
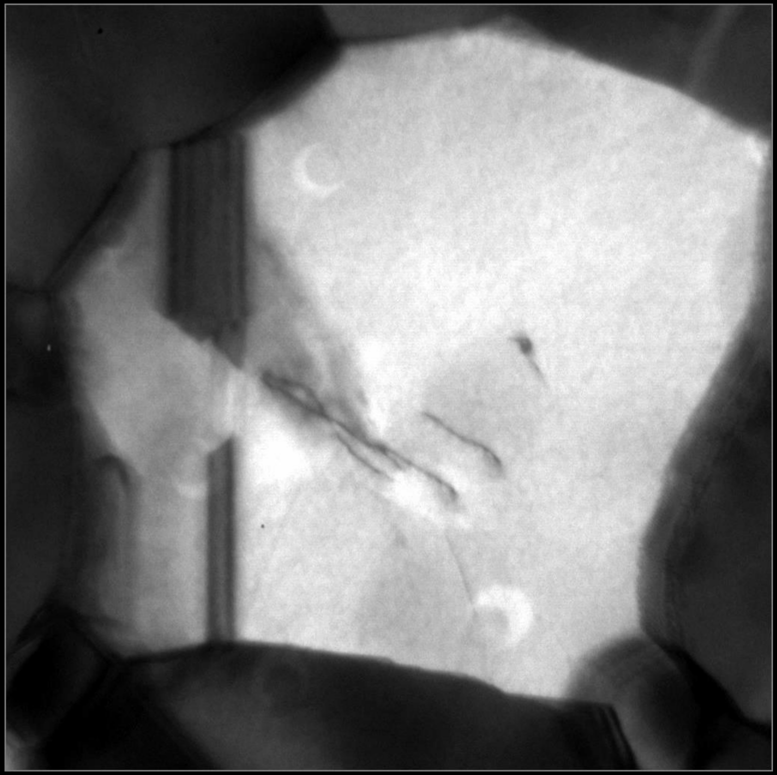
Irradiated Au NP model



The application of advanced microscopy techniques to extreme environments provides exciting new research directions

Single Ion Strikes

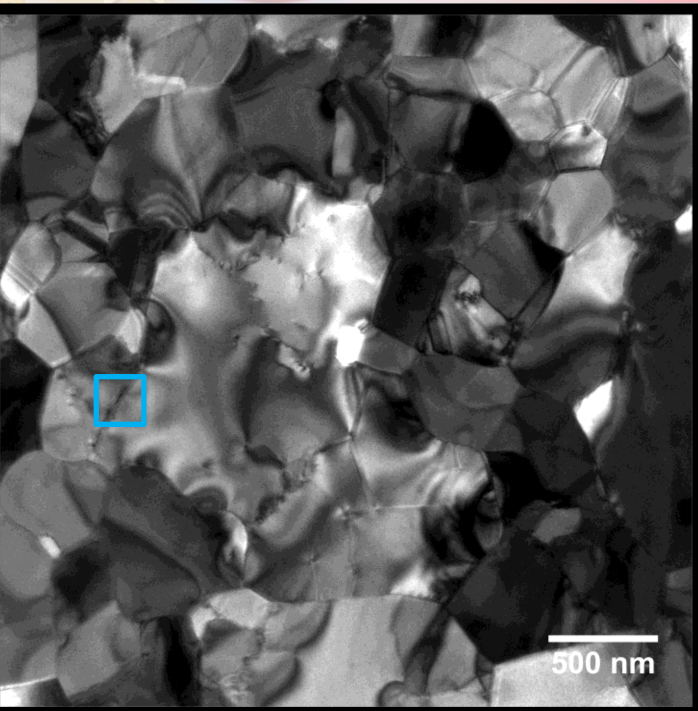
Collaborators: C. Chisholm & A. Minor

7.9 x 10^9 ions/cm^2/s		6.7 x 10^7 ions/cm^2/s
	VS	

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

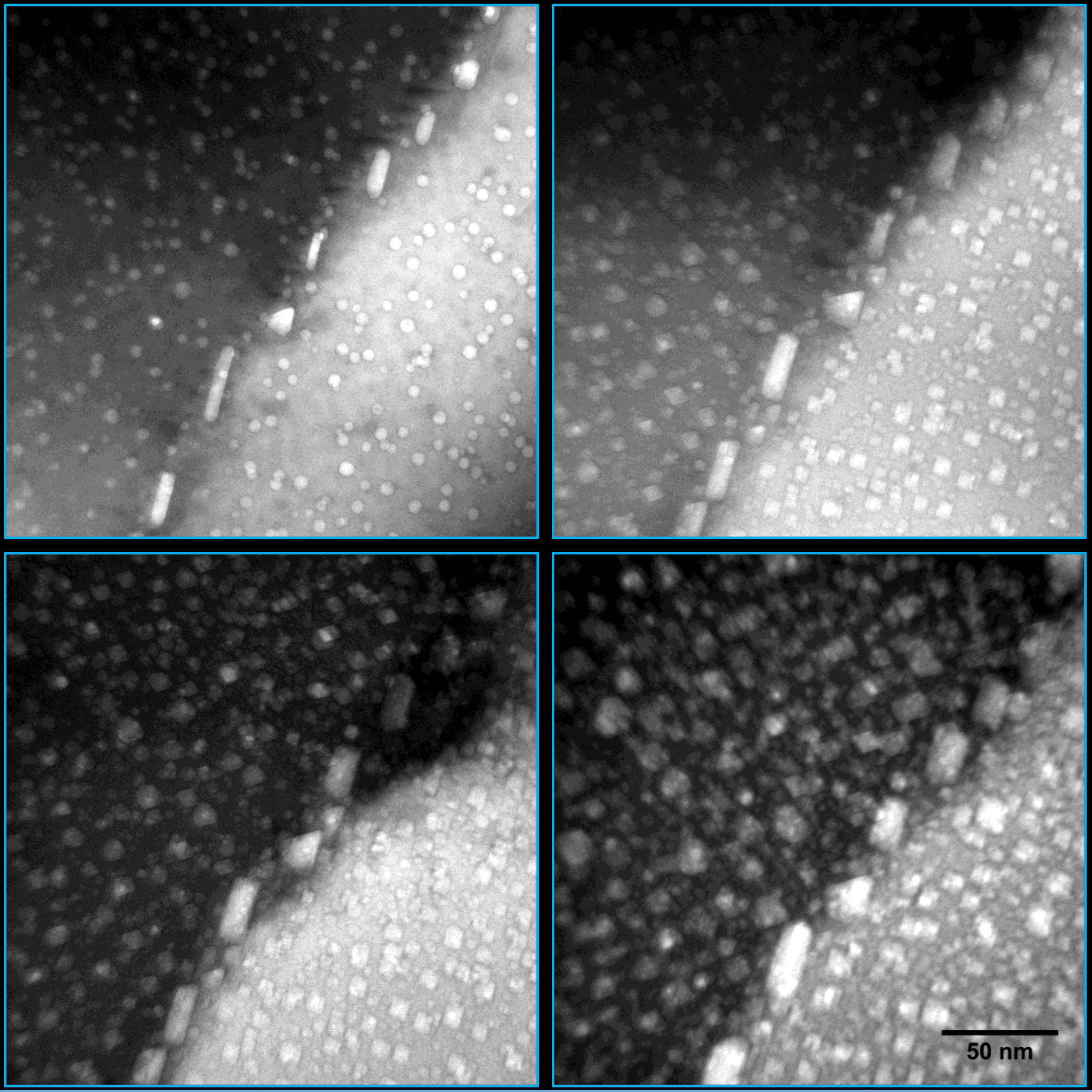
In situ Implantation

Collaborators: C. Chisholm & A. Minor

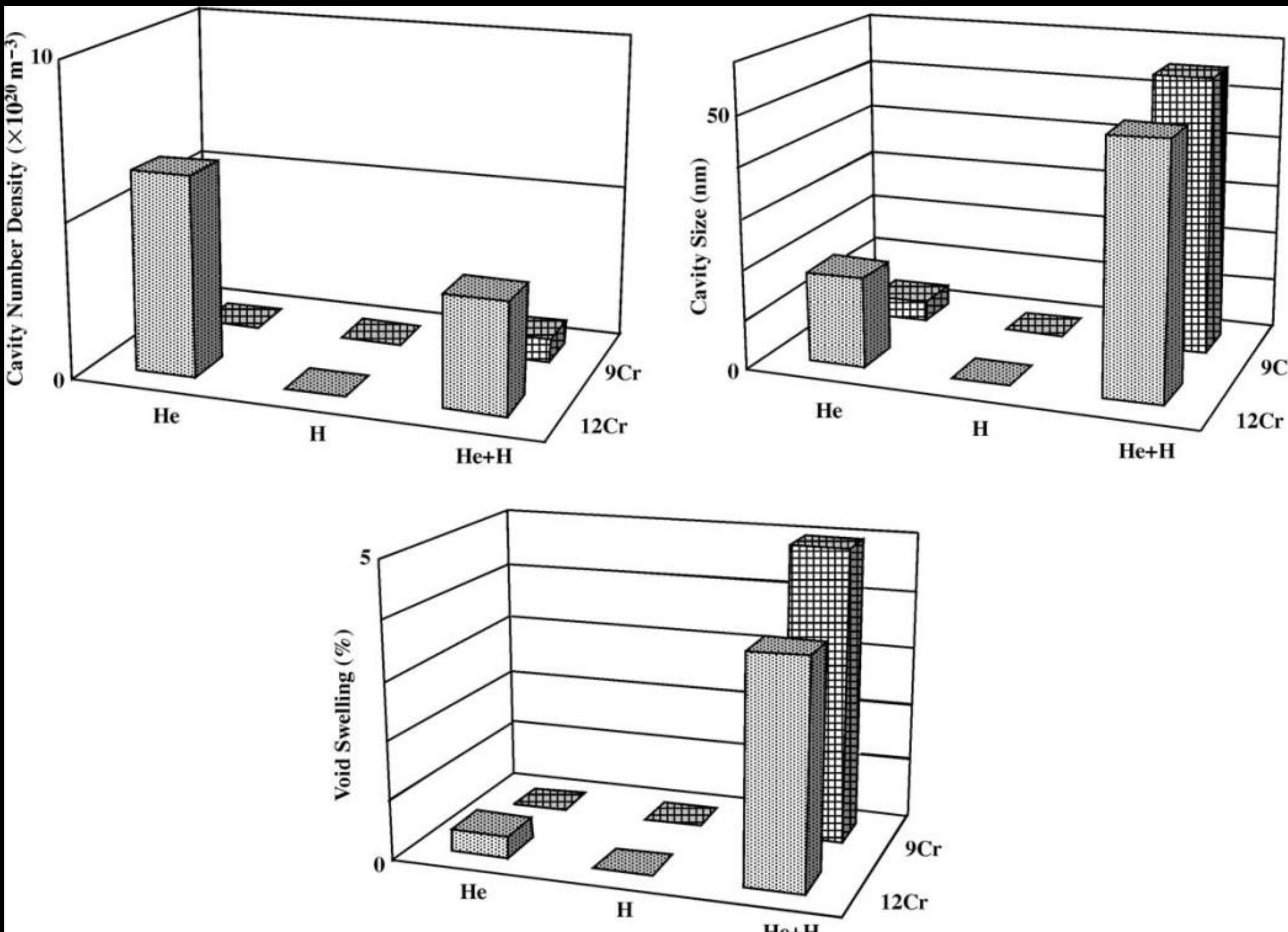


Gold thin-film implanted with 10keV He^{2+}

Result: porous microstructure



H, He, and Displacement Damage Synergy



T. Tanaka et al. "Synergistic effect of helium and hydrogen for deffect evolution under milt-ion irradiation of Fe-Cr ferritic alloys"

J. of Nuclear Materials 329-333 (2004) 294-298

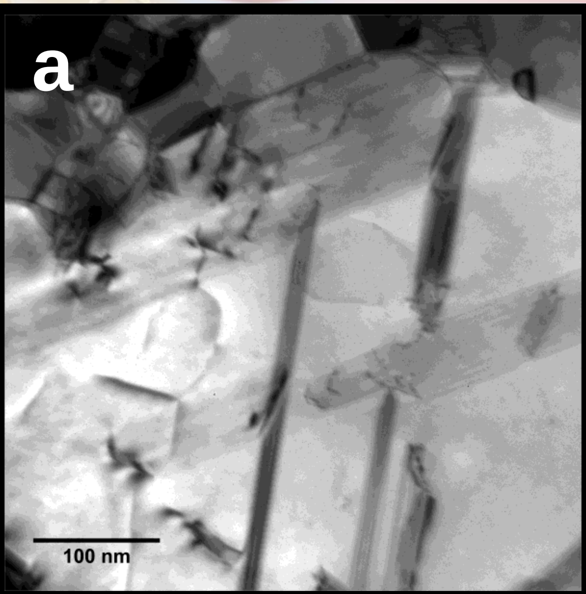
Coupling Effect

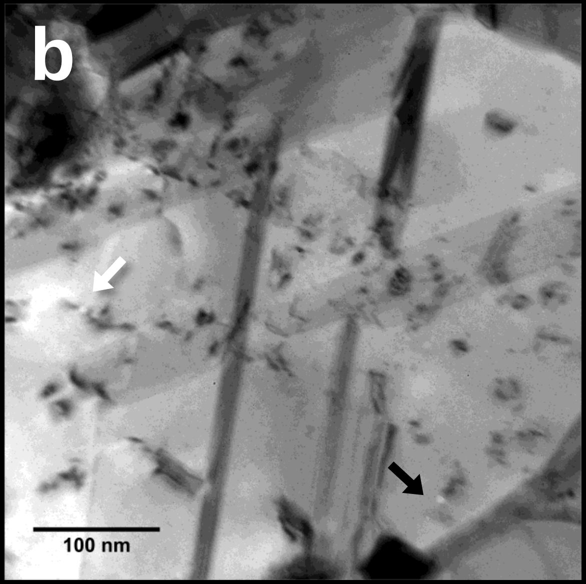
- **H and He are produced as decay products**
- **The relationship between the point defects present, the interstitial hydrogen, and the He bubbles in the system that results in the increased void swelling has only been theorized.**
- **The mechanisms which governs the increased void swelling under the presence of He and H have never been experimental determined**

No capability currently exist for triple beam irradiation in the U.S. and No capability for tripple beam TEM ion irradiation exists in the world

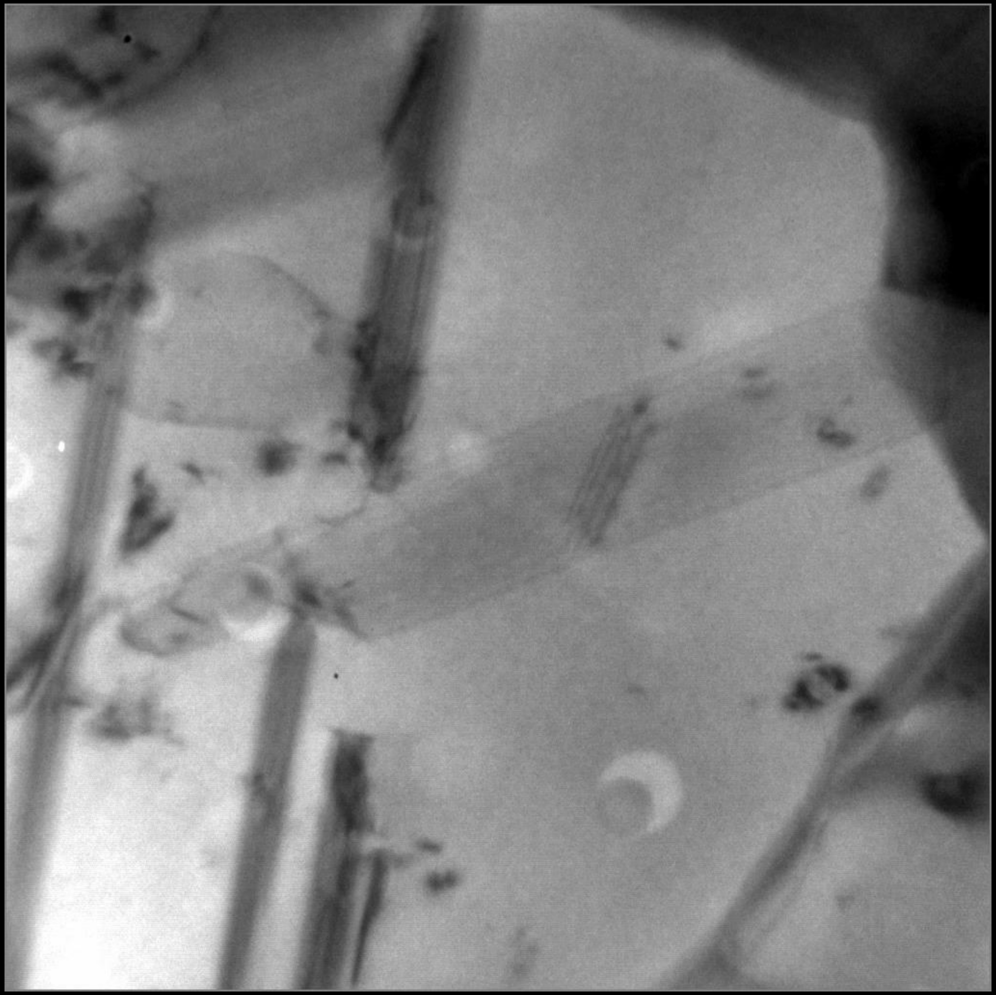
In situ Concurrent Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor



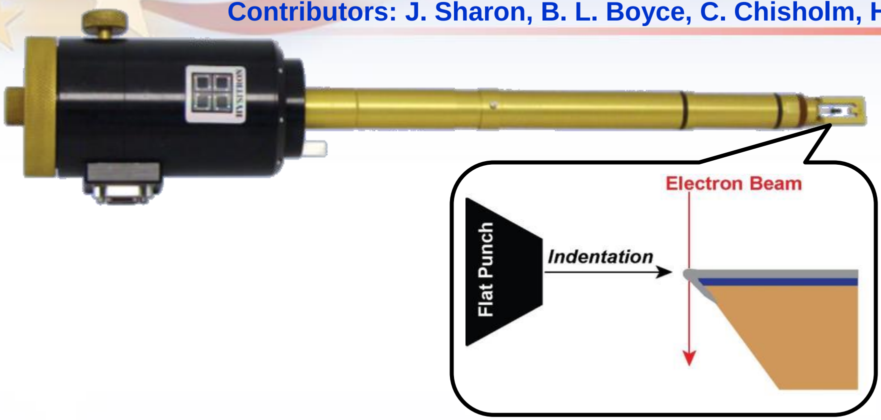


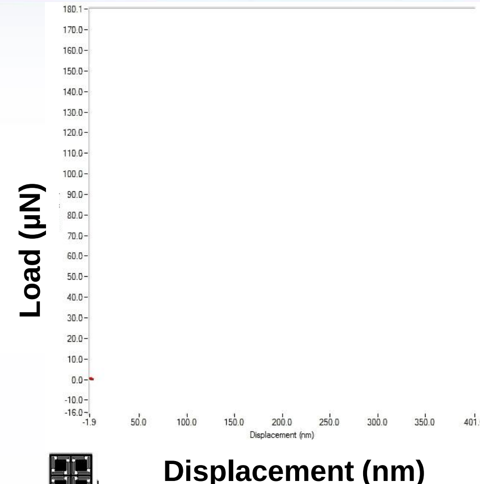
He^{1+} implantation and Au^{4+} irradiation of a gold thin film

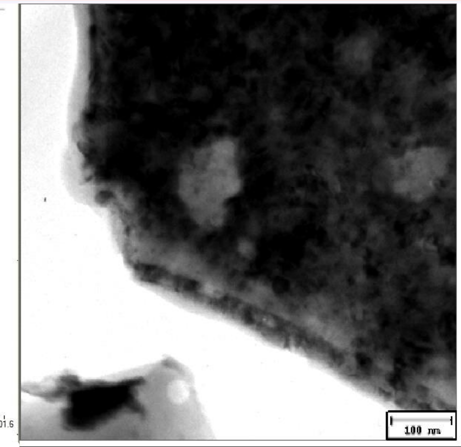


In situ TEM Quantitative Mechanical Testing

Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.





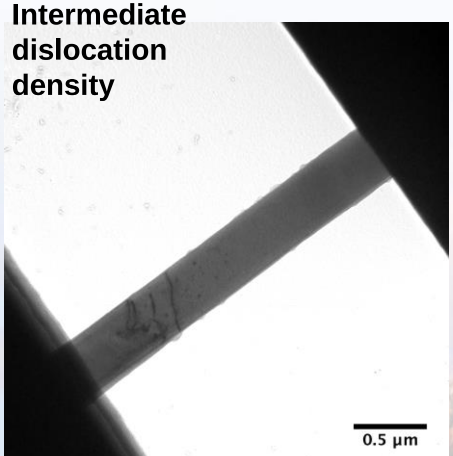


Range of Mechanical Testing Techniques

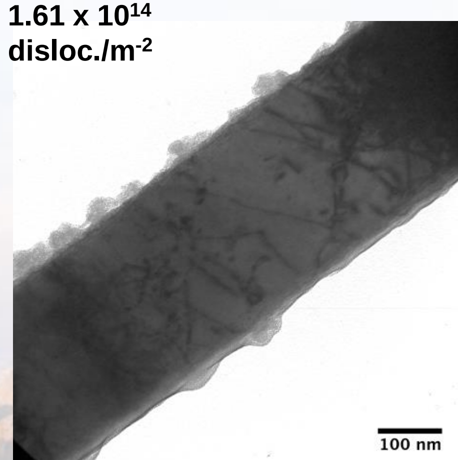
- Indentation
- Compression
- Tension
- Tension
- Wear
- Fatigue
- Creep

Fundamentals of Mechanical Properties

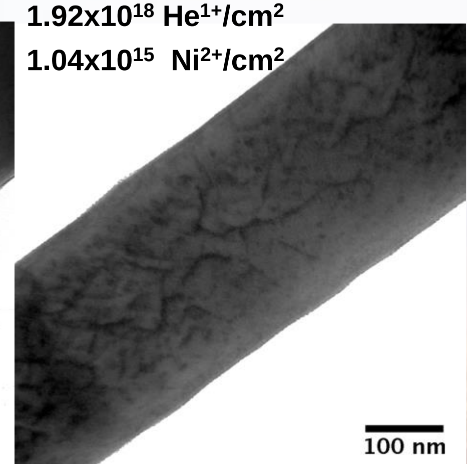
Intermediate dislocation density

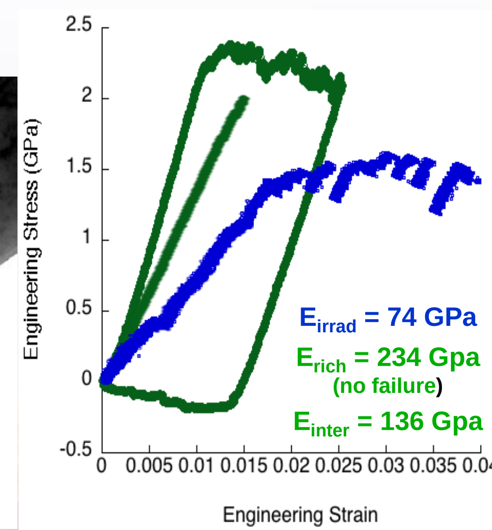


1.61×10^{14} disloc./m^{-2}



1.92×10^{18} He^{1+}/cm^2
1.04×10^{15} Ni^{2+}/cm^2

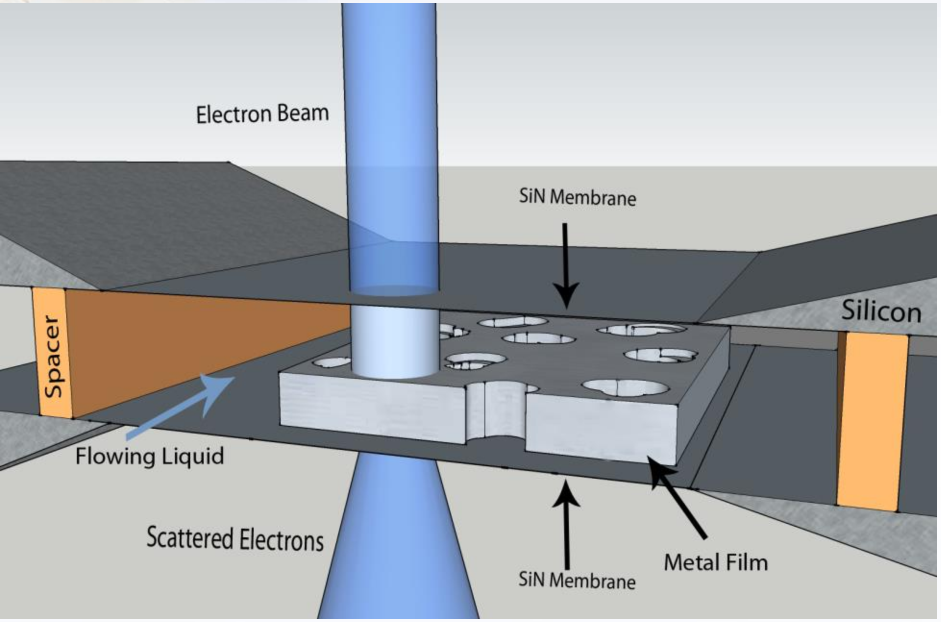




Work has started looking at the quantitative effects of ion irradiation on mechanical properties

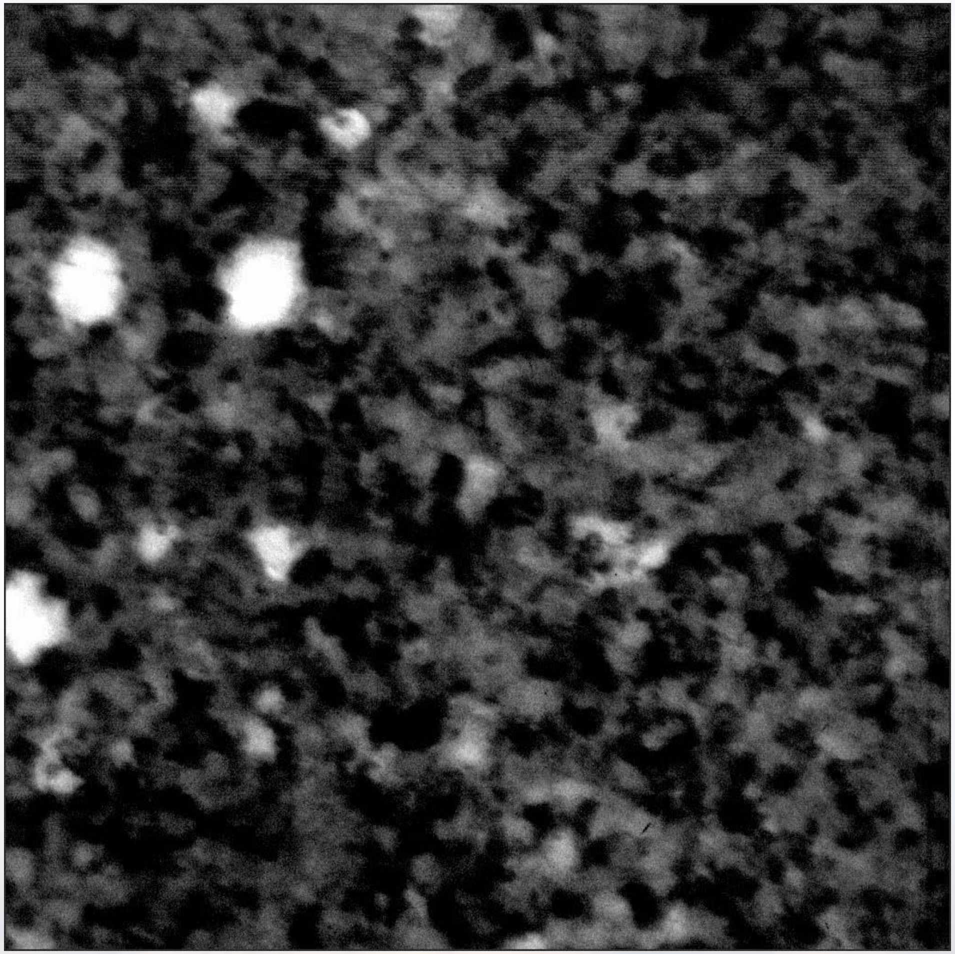
Can We Gain Insight into the Corrosion Process through *In situ* TEM?

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



Microfluidic Stage

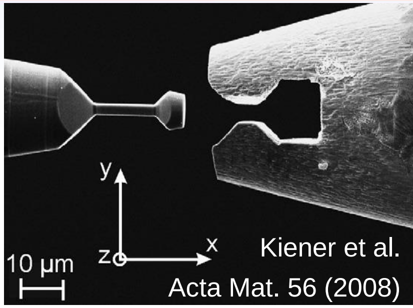
- **Mixing of two or more channels**
- **Continuous observation of the reaction channel**
- **Chamber dimensions are controllable**
- **Films can be directly deposited on the electron transparent SiN membrane**



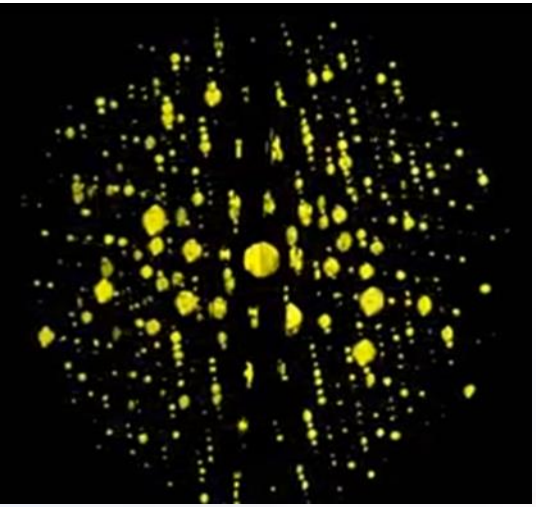
Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.

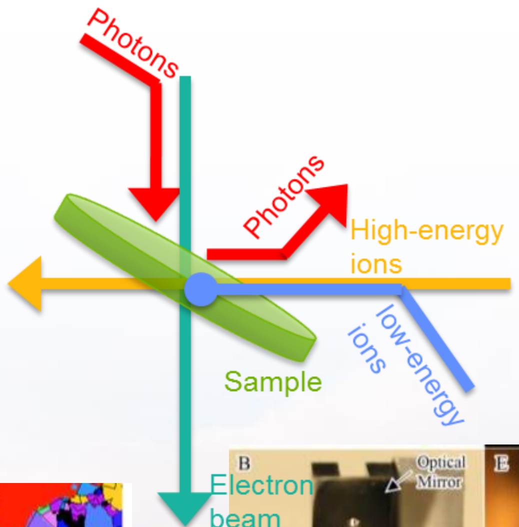
Future Directions Under Pursuit to Consider

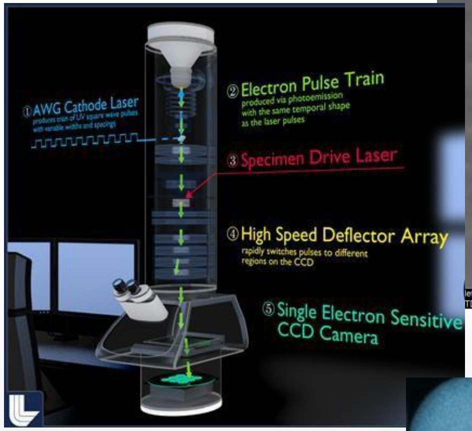
1. In-situ TEM CL, IBIL (currently capable)
2. *In situ* ion irradiation TEM in liquid or gas (currently capable)
3. PED: Local texture characterization (arriving FY15)
4. Quantative in-situ tensile/creep experiments (Sample in development)
5. DTEM: Nanosecond resolution (laser optics needed)

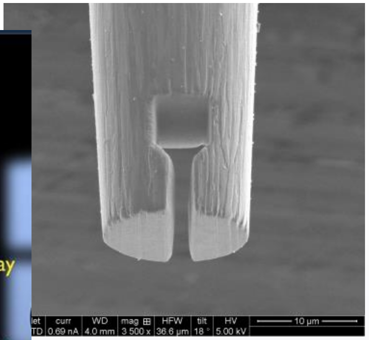


Kiener et al. Acta Mat. 56 (2008)

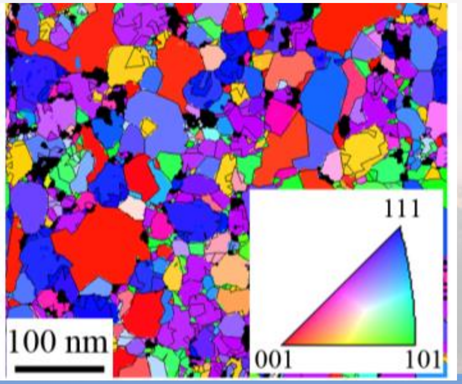


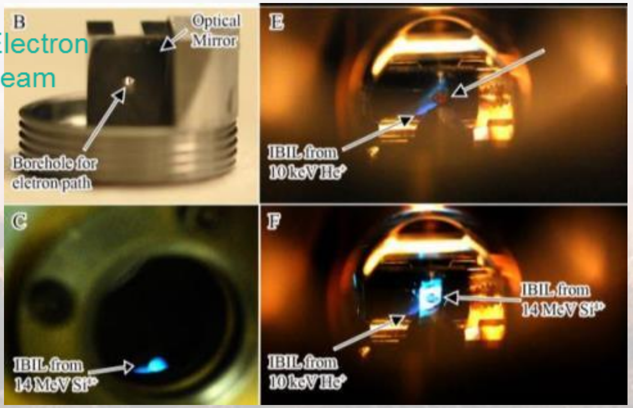


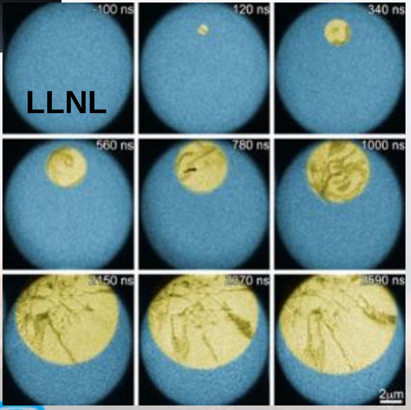




AppFive
NanoMegas







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