Tool Development to Elucidate Mechanisms in Complex Environments

Khalid Hattar

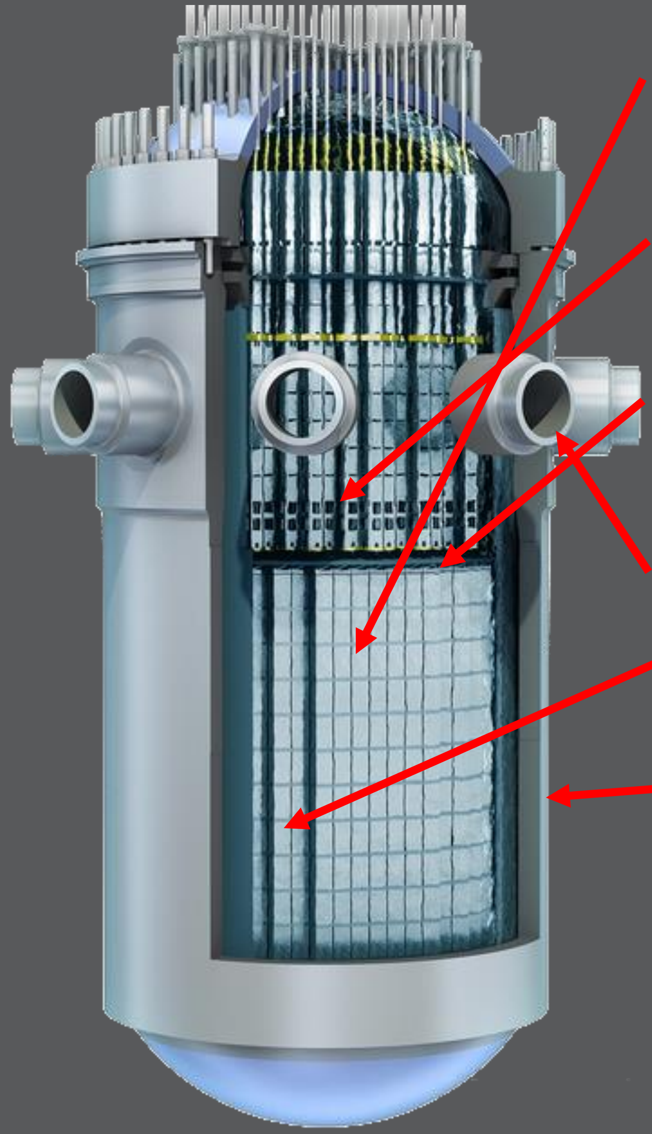
THE UNIVERSITY OF TENNESSEE

Collaborators:

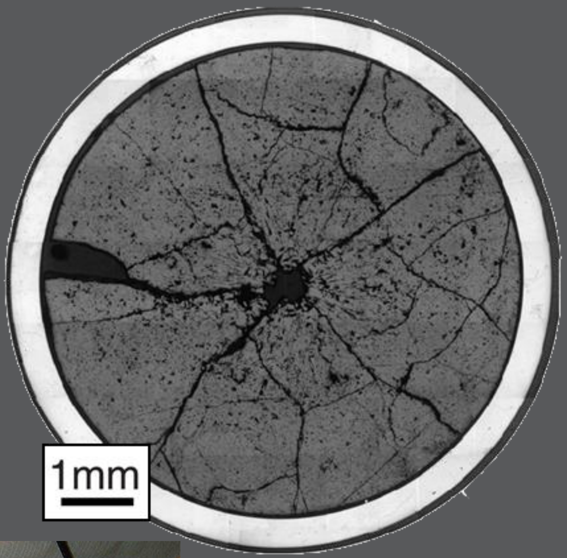
K. Burns, E. Lang, R. Schoell, N. Madden, R. DeMott, K. Small, D.L. Buller, D.C. Bufford, S.H. Pratt, T.J. Boyle, B.A. Hernandez-Sanchez, S.J. Blair, B. Muntifering, C. Chisholm , P. Hosemann, A. Minor, J. A. Hinks, F. Hibberd, A. Ilinov, D. C. Bufford, F. Djurabekova, G. GreavIDES Inces, A. Kuronen, S. E. Donnelly, K. Nordlund, F. Abdeljawad, S.M. Foiles, J. Qu, C. Taylor, J. Sugar, P. Price, C.M. Barr, D. Adams, M. Abere, L. Treadwell, A. Cook, A. Monterrosa,, J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, W. Mook, Hysitron Inc., G.S. Jawaharram, S. Dillon, R.S. Averback, N. Heckman, J. Carroll, S. Briggs, E. Carnes, J. Brinker, D. Sassaki, T. Nenoff, B.G. Clark, P.J. Cappillino, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, L.R. Parent, I. Arslan. & Protochips, Inc.

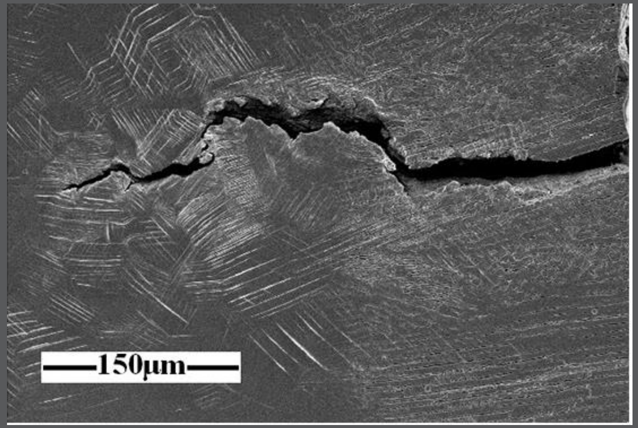
This work was partially funded by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. This work wasperformed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of

Reactor Materials Challenges



- Nuclear Fuels
- Displacement Damage Effects
- Transmutation Effects
- Corrosion
- Creep
- Fatigue



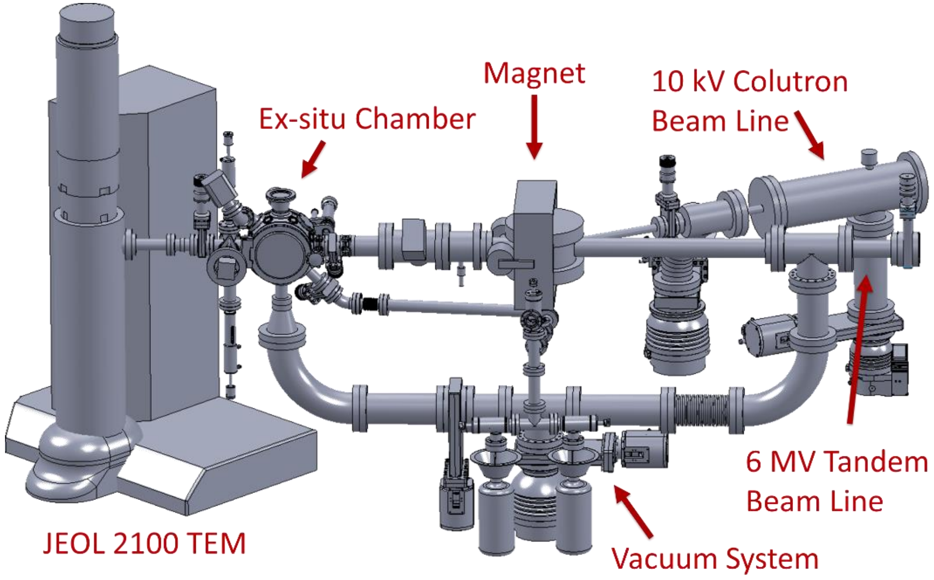


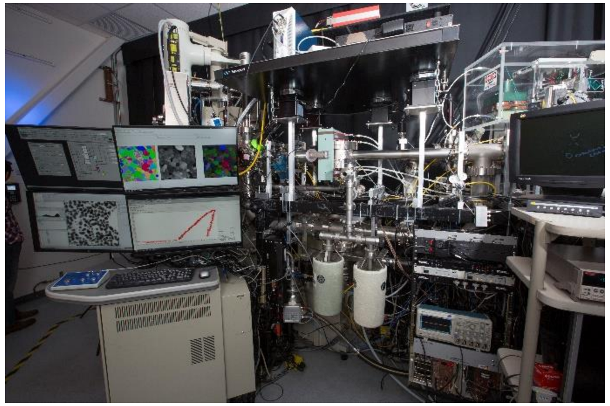
Nuclear reactors pose unique materials challenges due to degradation effects from combined environmental stressors

Concurrent *In situ* Ion Irradiation TEM Facility

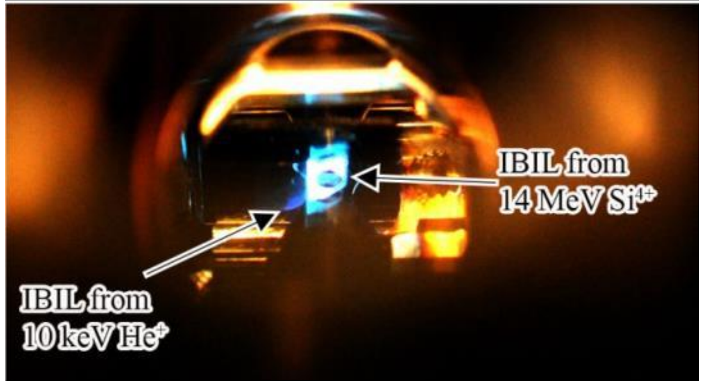
Collaborator: D.L. Buller

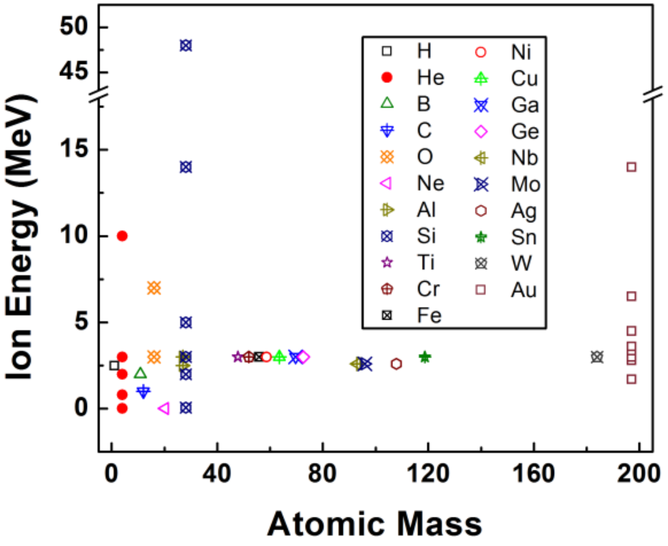
10 kV Colutron - 200 kV TEM - 6 MV Tandem

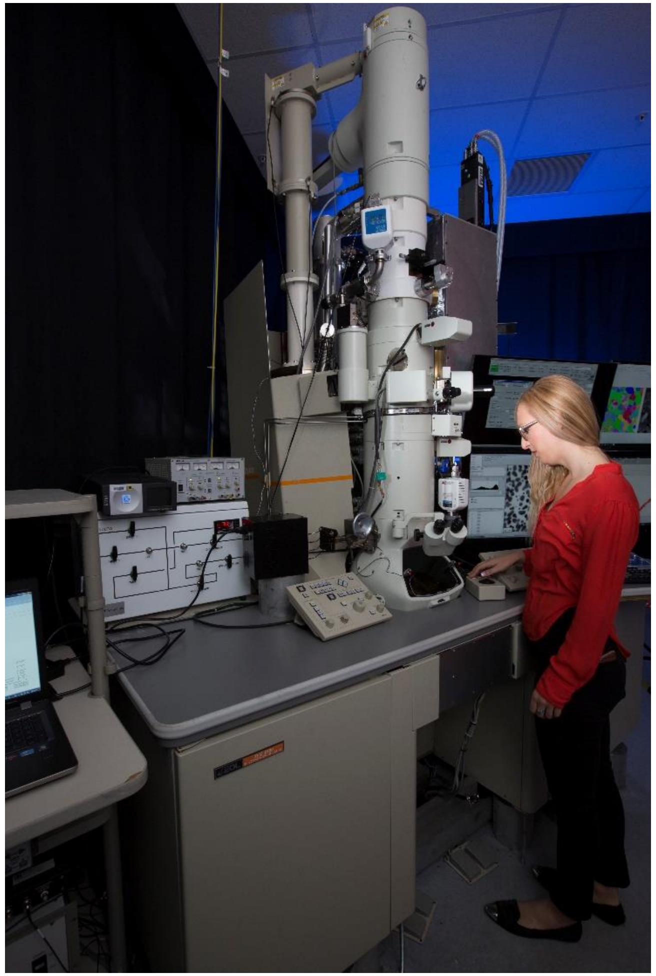




IBIL from a quartz stage inside the TEM







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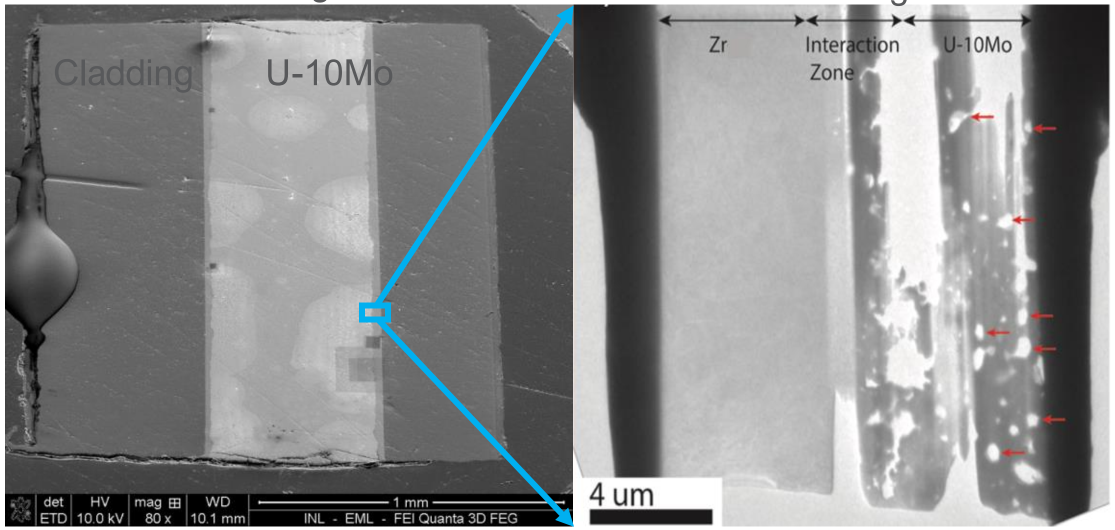
PIE Characterization of U-10Mo/Zr Monolithic Interface

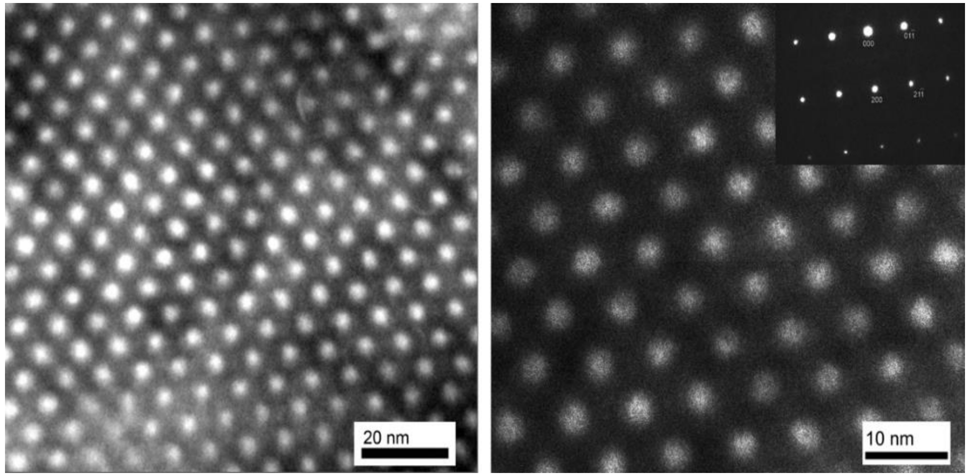
Collaborators: C. Barrr, A. Aitkaliyeva

SEM Overview Image

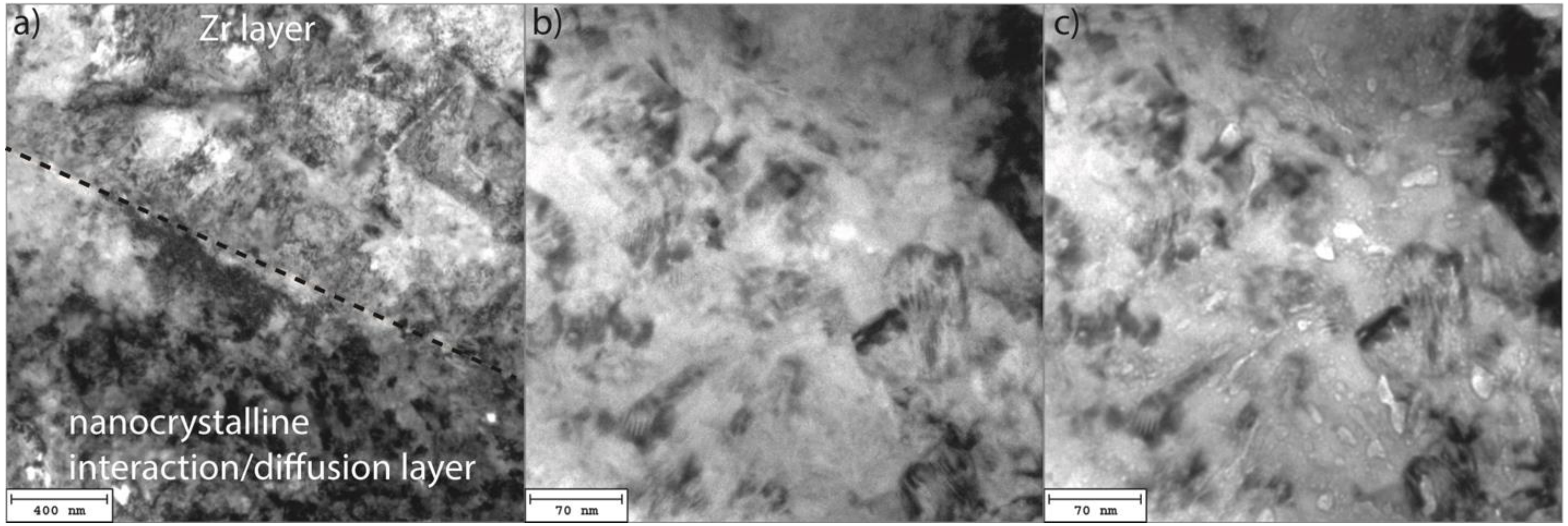
TEM Overview Image



Gas superlattice bubbles ZA [110] in U-10Mo.



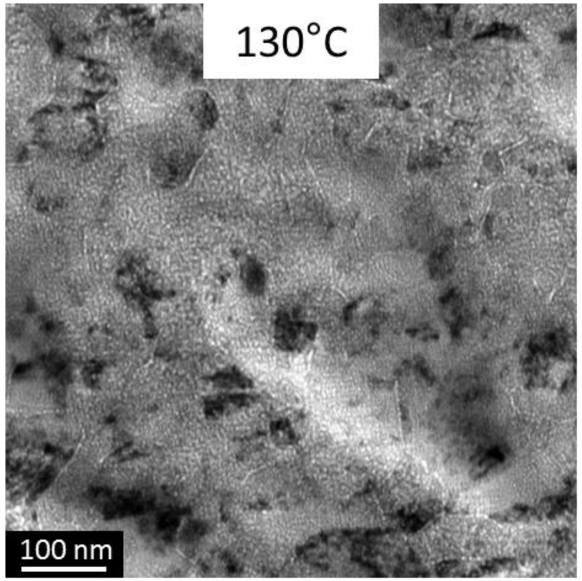
- U-10Mo Monolithic fuel with fission density (fiss/cm^3) = 4.4 x 10^{21}
- Average gas superlattice bubble diameter distribution is 3.5 ± 0.25 nm diameter.

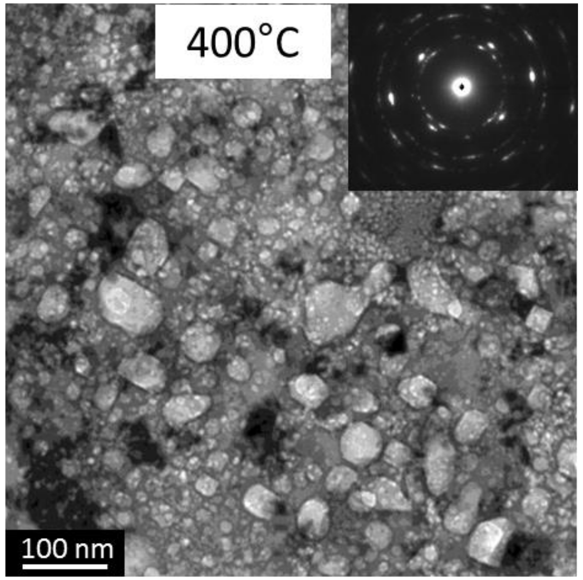


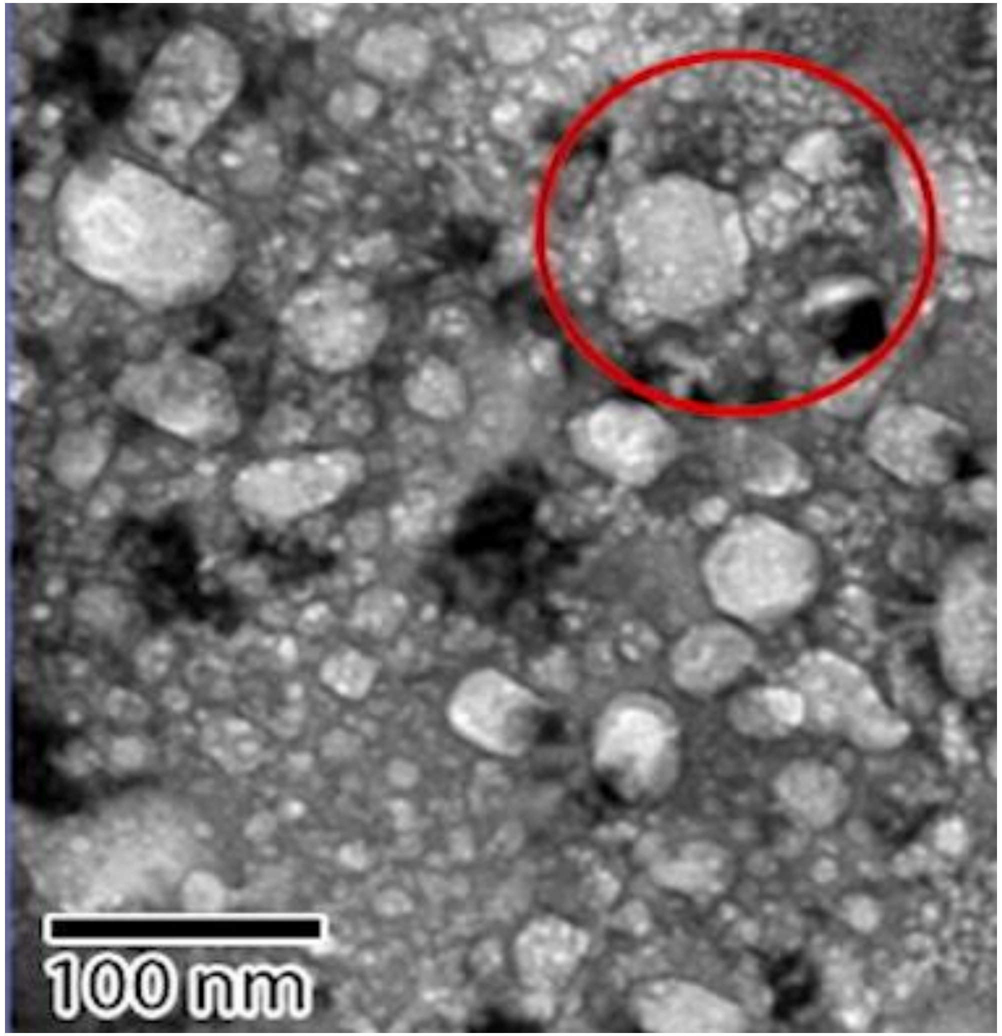
Zr layer and interaction layers where in-focus image of interaction layer (center) and de-focus (-2um) image of interaction layer with high level of porosity apparent

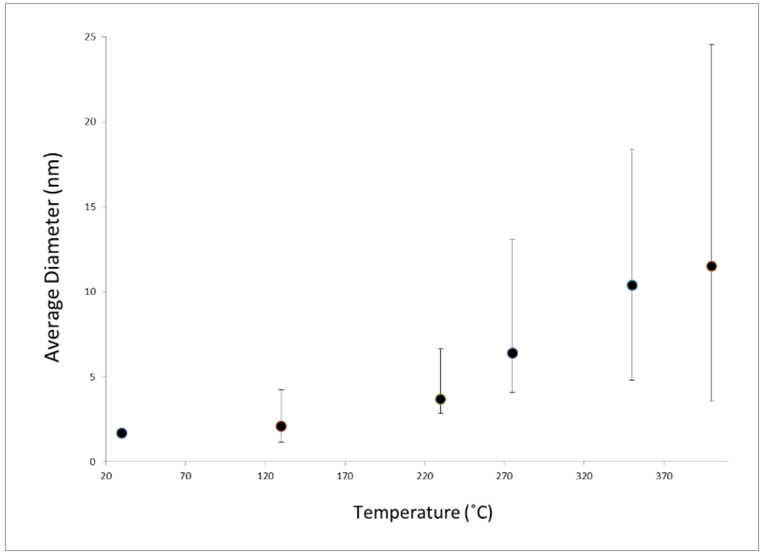
Cavity Growth during In-situ Annealing of 10 keV He+ Implanted and then 3 MeV Irradiated Ni^{3+}

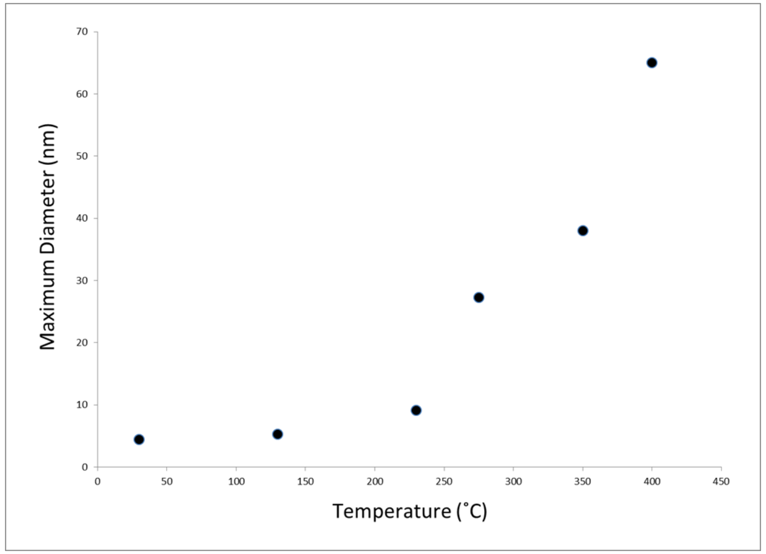
Collaborator: B. Muntifering & J. Qu







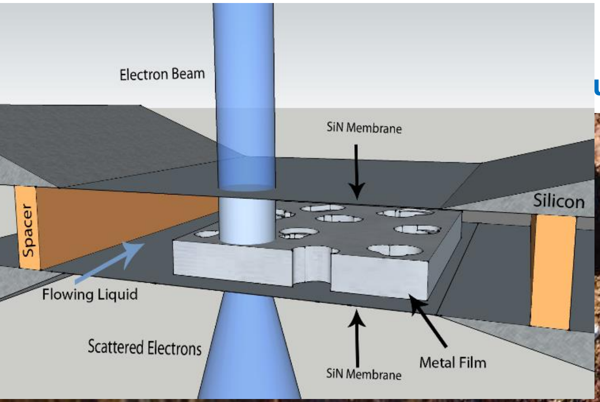




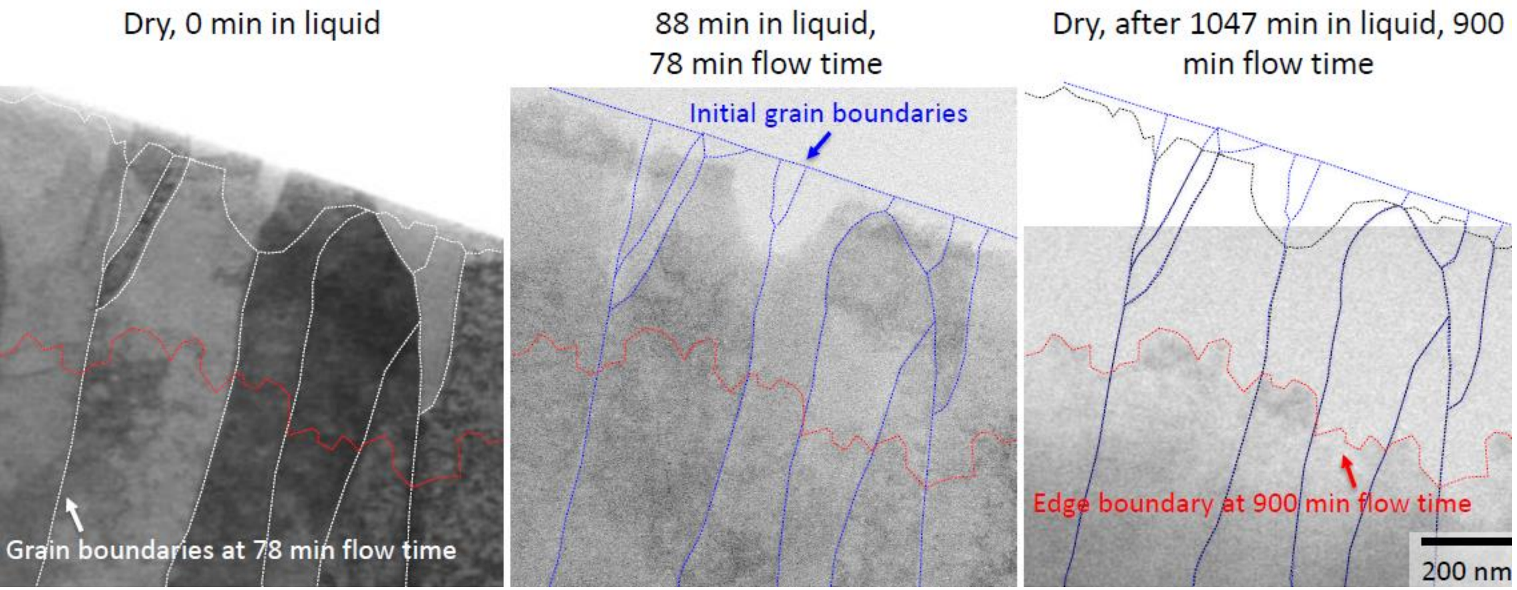
Bubble to cavity transition and cavity evolution can be directly studied

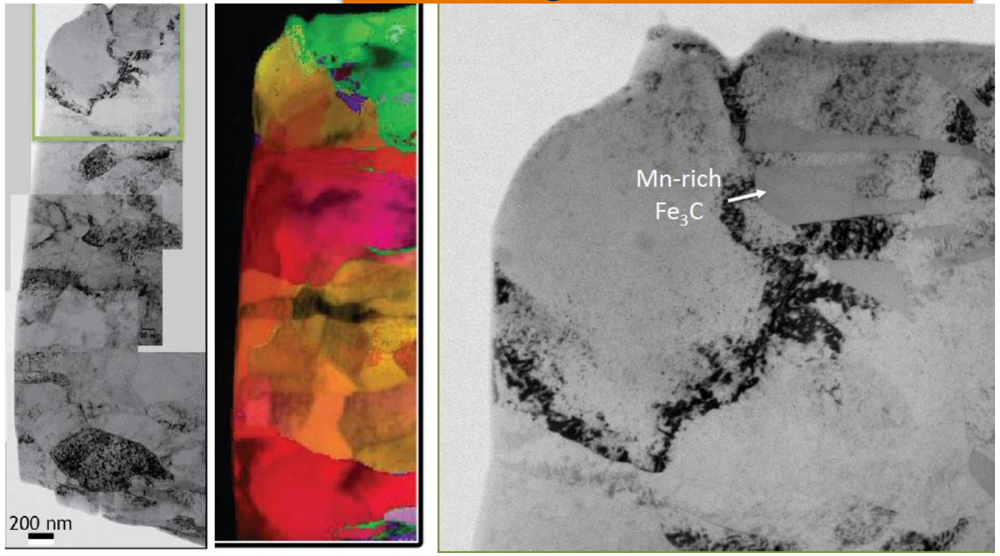
1018 Steel Corrosion Mechanisms

utors: Claire Chisholm, William Mook, Dan Bufford, Katherine Jungjohann & Aramco Services Company



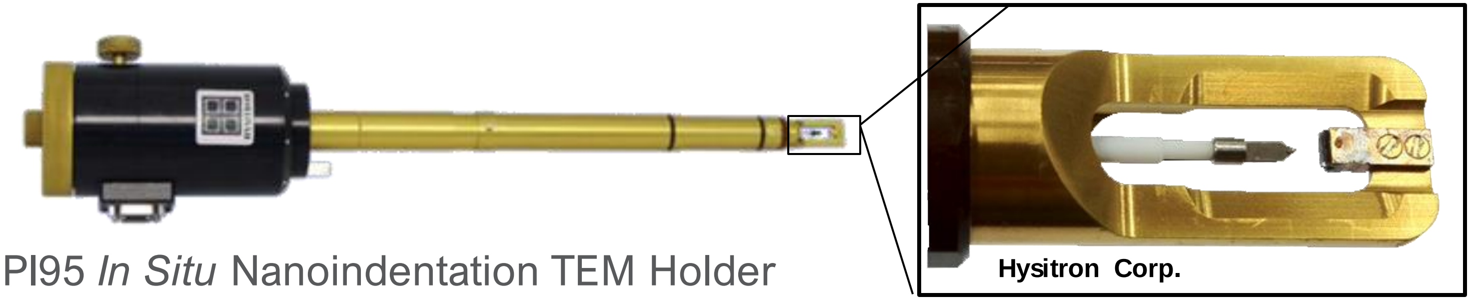
- Ferrite and cementite phases in contact with electrolyte solution will experience charge transfer from the ferrite to the cementite, causing dissolution of the Fe ions in the ferrite grains
- Larger grain size of cementite will result in faster etching of ferrite grain





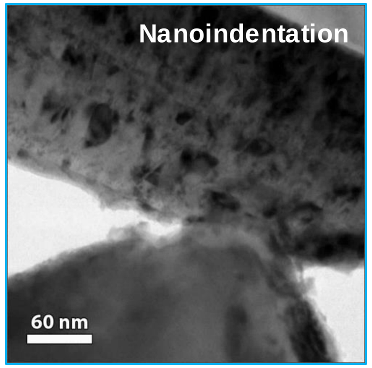
In situ Quantitative Mechanical Testing

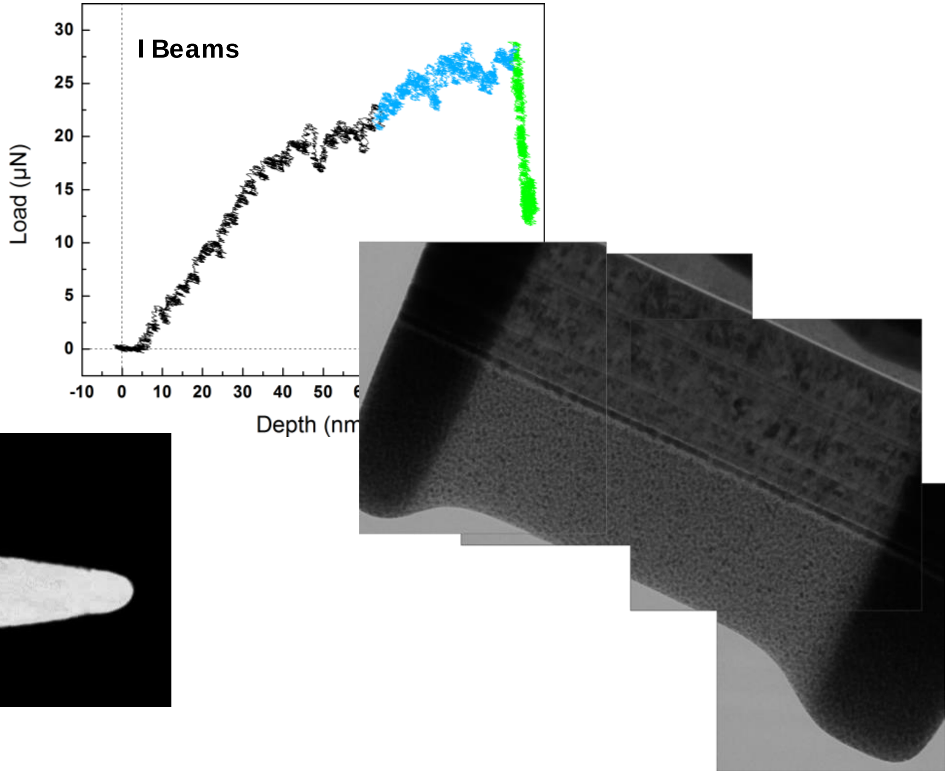
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

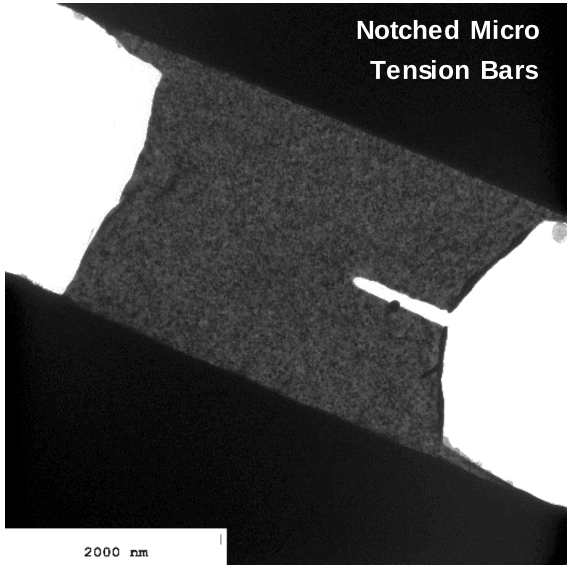


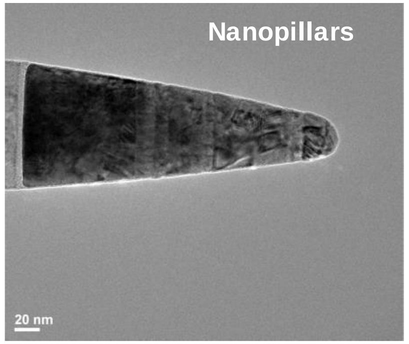
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

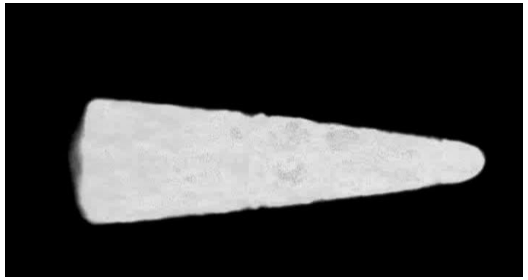
- Sub nanometer displacement resolution
- Quantitative force information with μN resolution
- Concurrent real-time imaging by TEM





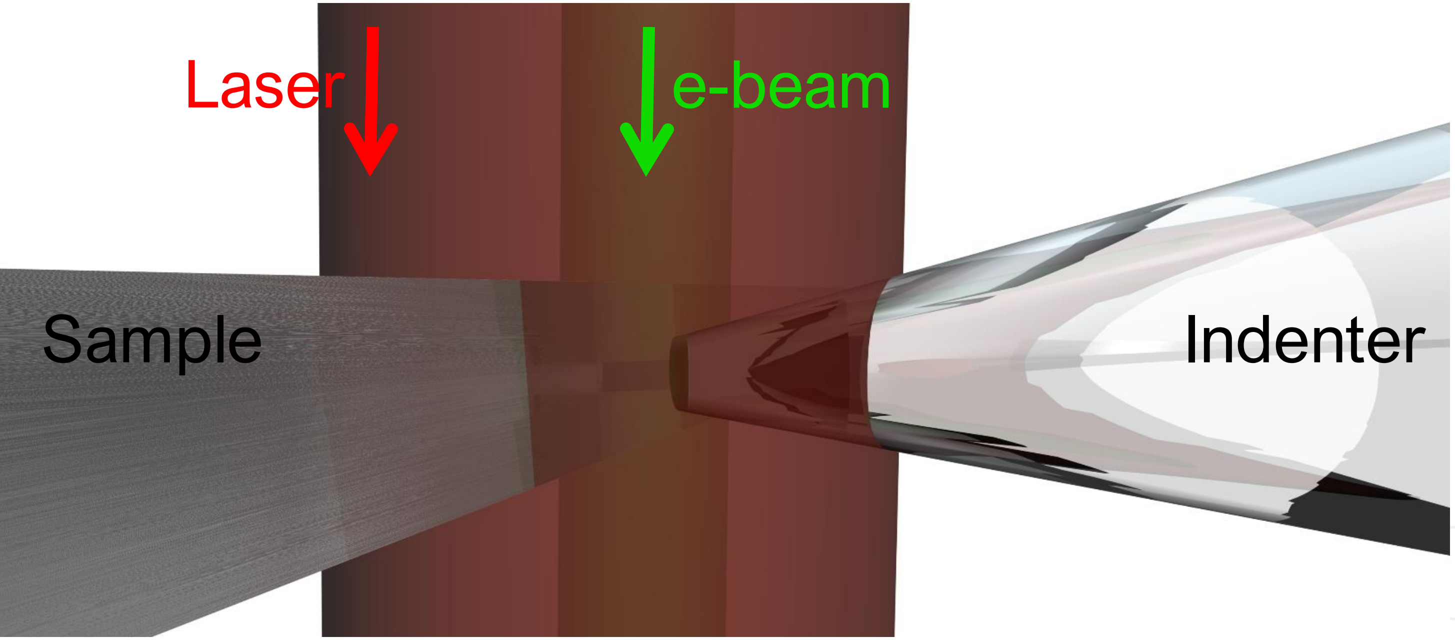




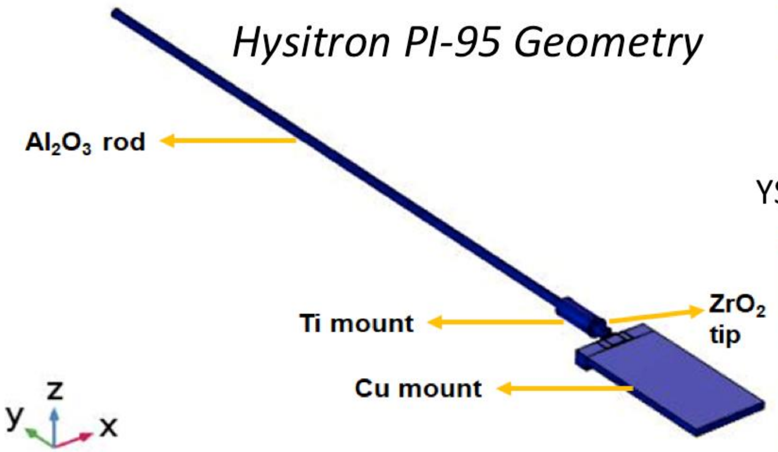


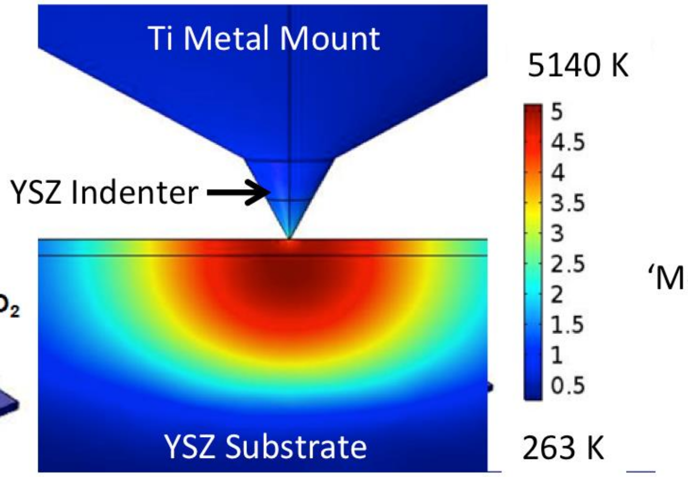
Combing Laser Heating with Mechanical Testing?

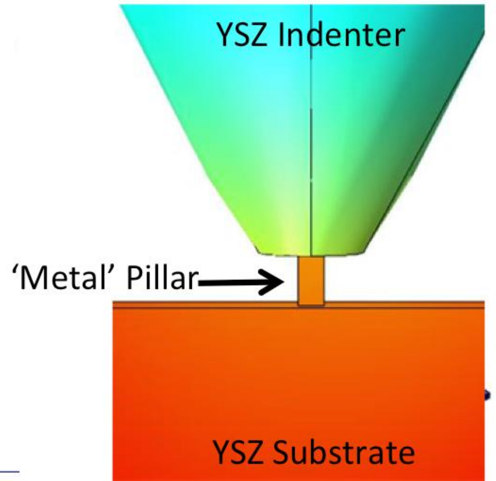
Contributors: R.L. Grosso, E.N.S. Muccillo, D.N.F. Muche, G.S. Jawaharram, C.M. Barr, A.M. Monterrosa, R.H.R. Castro, S.J. Dillon



	Mg	UO_2	Al_2O_3
A'	1	1.5	1.2
f	0.5	0.5	0.5
$d\ (m)$	$1.5x10^{-5}$	$7x10^{-6}$	$1.2x10^{-6}$
$\vec{b}\ (\dot{A})$	6.12	3.87	3.50
$V_a\ (m^3\ mol^{-1})$	1.39×10^{-5}	2.16×10^{-5}	2.60×10^{-5}
$v^*\ \left(\vec{b}^3\right)$ **Average**	5	1.5	2.5
$v^*\ \left(\vec{b}^3\right)$ **T dependence**	$2.72 \times 10^{-14} T^{5.35}$	$5.36 \times 10^{-9} T^{2.63}$	$8.79 \times 10^{-31} T^{9.54}$
$\sigma_c\ (Pa)$	$801,404(T/T_m)^{-5.39}$	$9.21 \times 10^{22} T^{-4.40}$	$2.47 \times 10^{39} T^{-9.69}$
$D_o\ (m^2\ s^{-1})$	1.6	0.34	0.86
$H\ (J\ mol^{-1})$	105,000	380,000	410,000



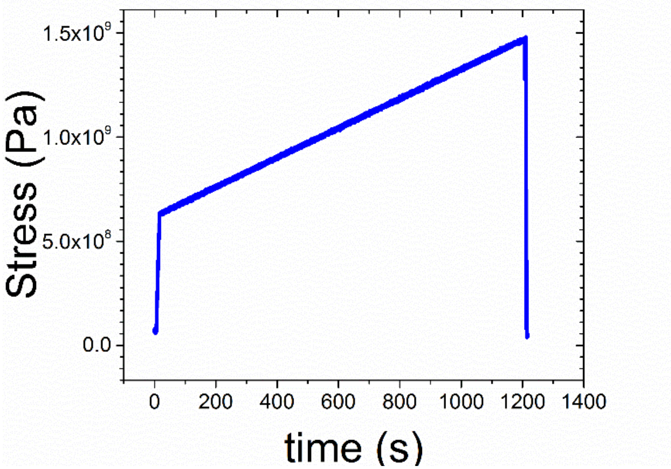




Irradiation Creep (4 MeV Cu^{3+} 10^{-2} DPA/s)

Contributors: G.S. Jawaharram, S. Dillon & R.S. Averback

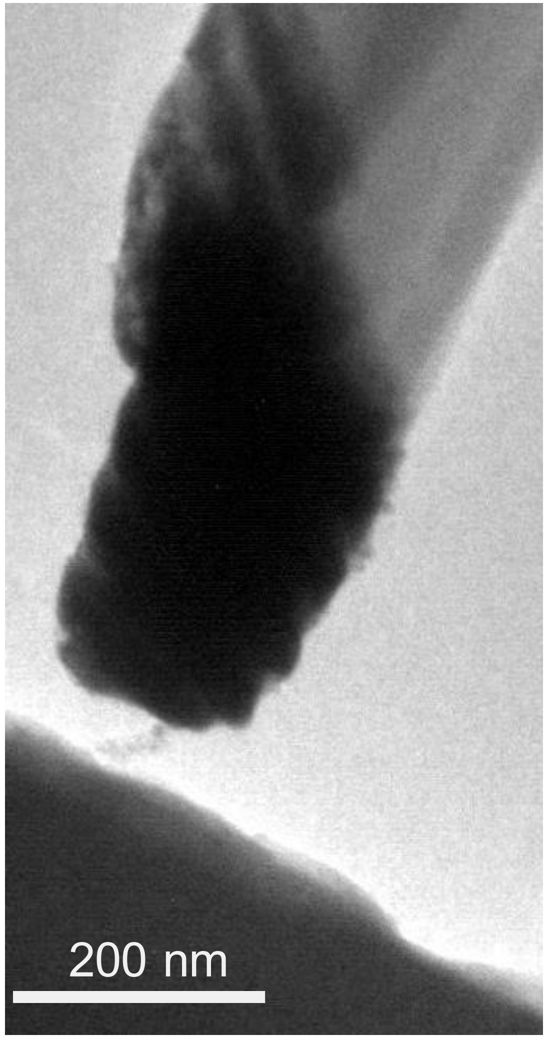
Controlled Loading Rate Experiments



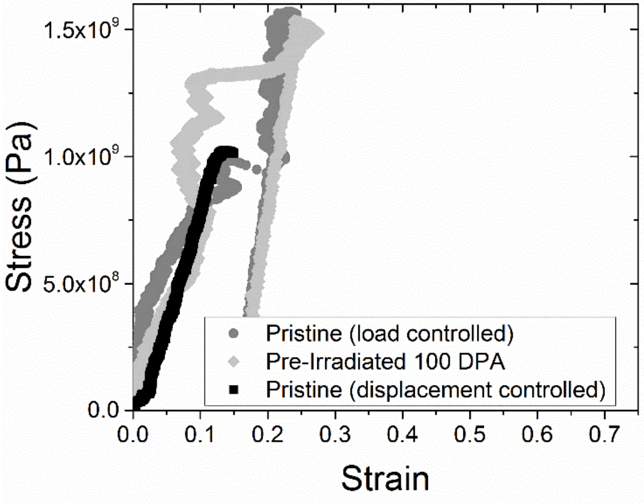
In-situ TEM radiation creep is feasible!

Great nanomechanical properties and irradiation tolerance does NOT mean great irradiation induced creep.

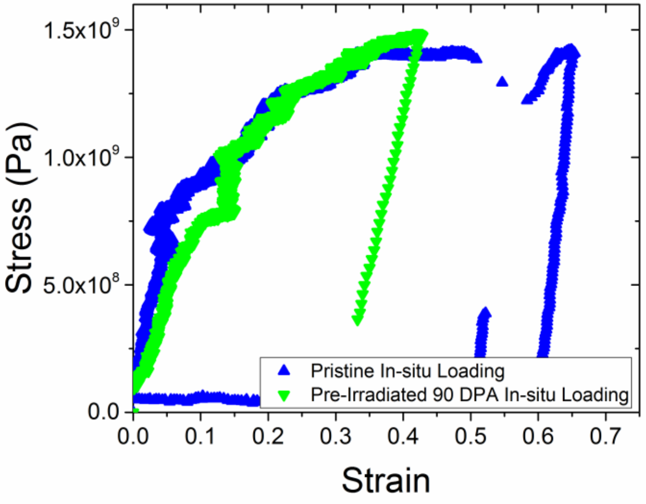
50 nm Cu-W multilayer
20 Min



No Irradiation
(Loading rate 0.6 Mpa s^{-1})

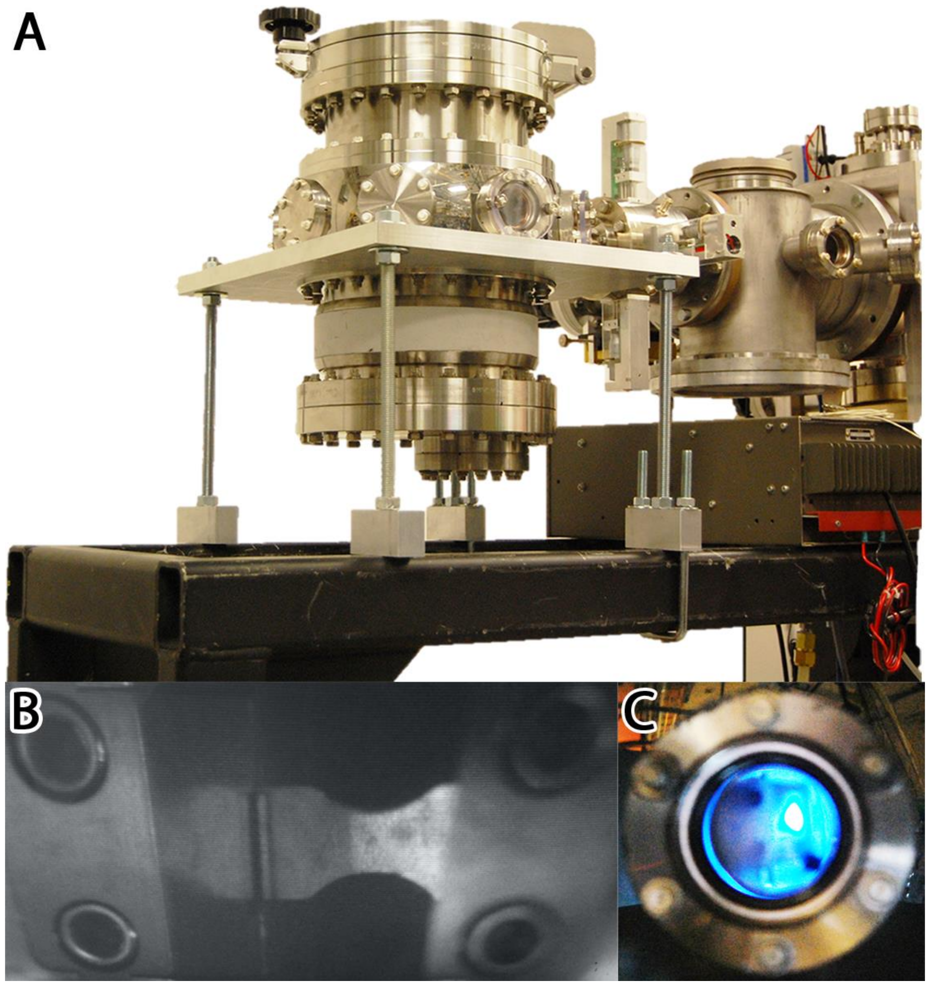


Irradiation Creep
(Loading rate 0.6 Mpa s^{-1})

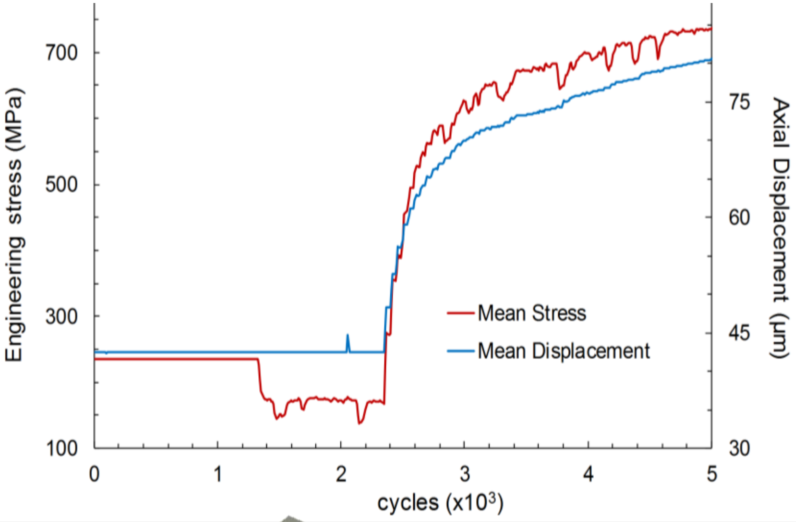


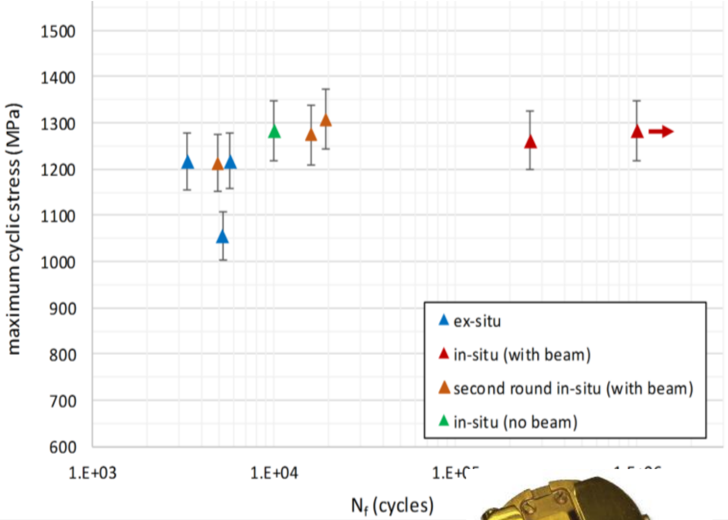
Ex situ Mechanical Testing End Station

Collaborators: D. Buller, B. Boyce, J. Carroll, P. Price, C. Taylor, B. Muntifering, S. Briggs, N. Heckman, J.A. Scott



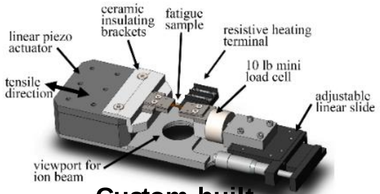
- Combined three individual mechanical testing in tandem beamline end station
- Limited (optical, IR only) imaging capabilities
- Have successfully collected preliminary data using this system





MTI Fullam
Straining Heating

Hysitron PI85
Nanoindenter

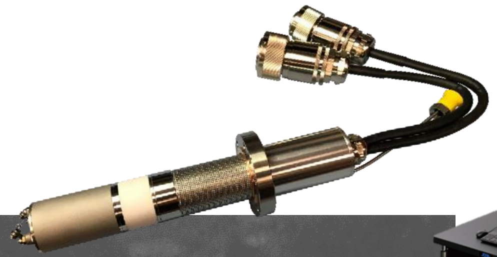


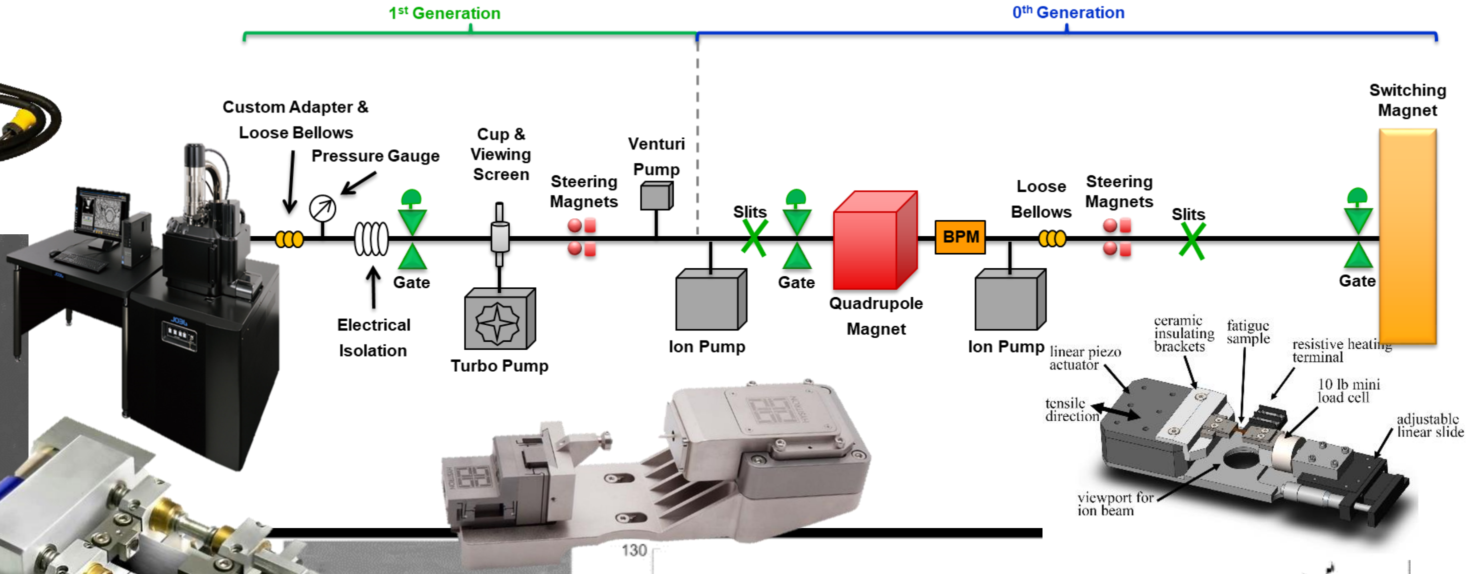
Custom-built
Piezo Fatigue
tester

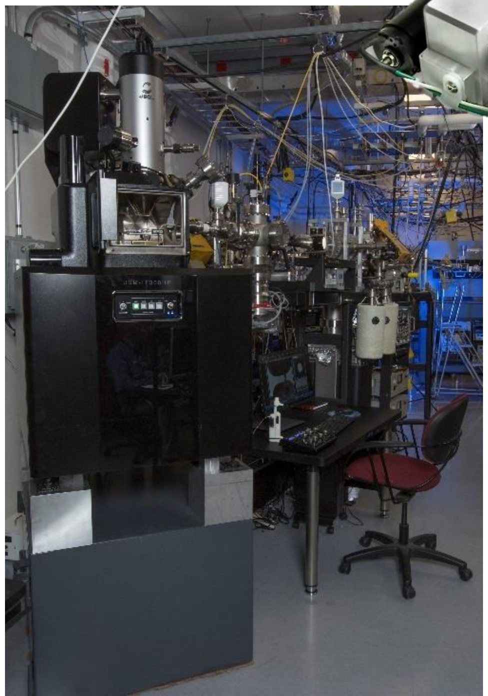
Gatan Cryostage

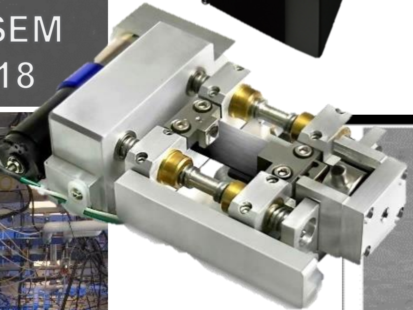
In situ Ion Irradiation SEM (I^{3}SEM)

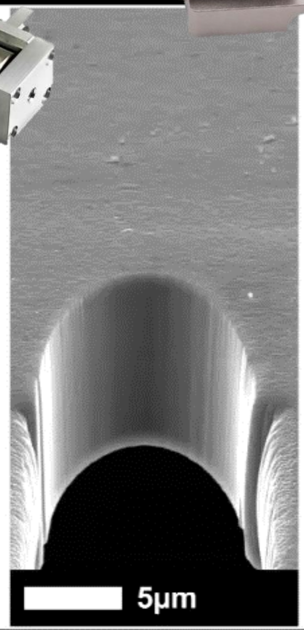
Collaborators: N. Heckman, D. Buller, B. Boyce, J. Carroll, C. Taylor, B. Muntifering, & S. Briggs

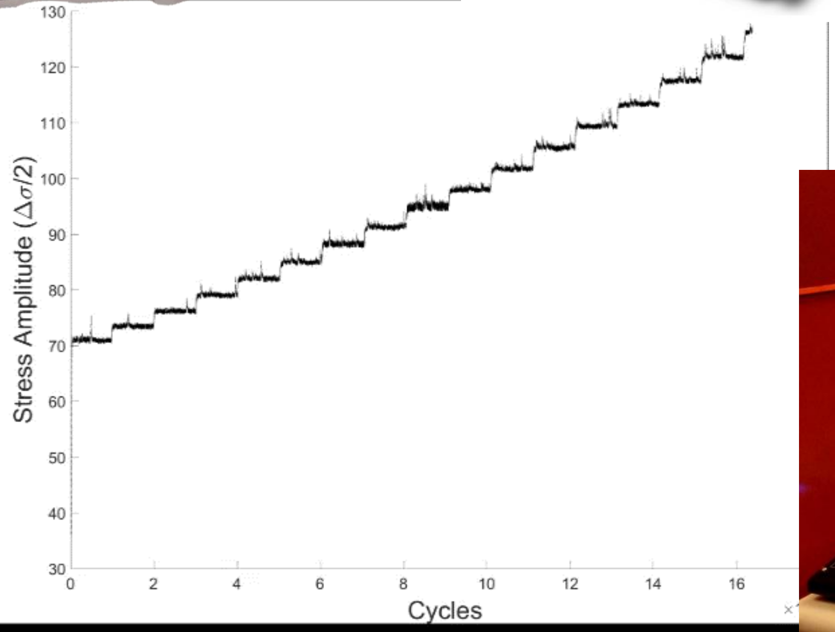


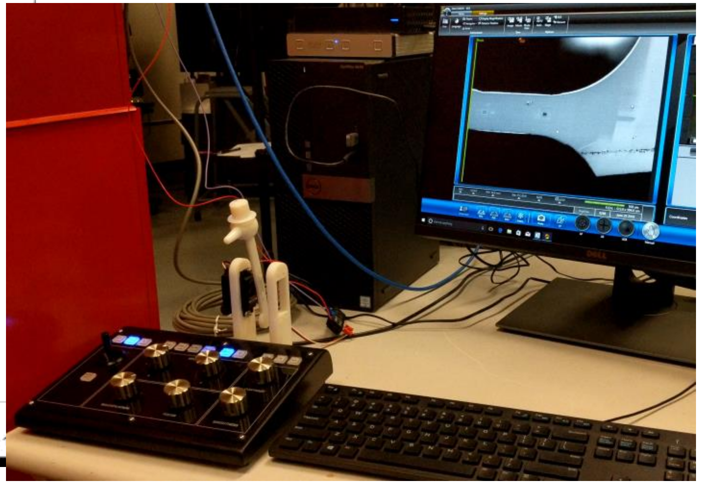












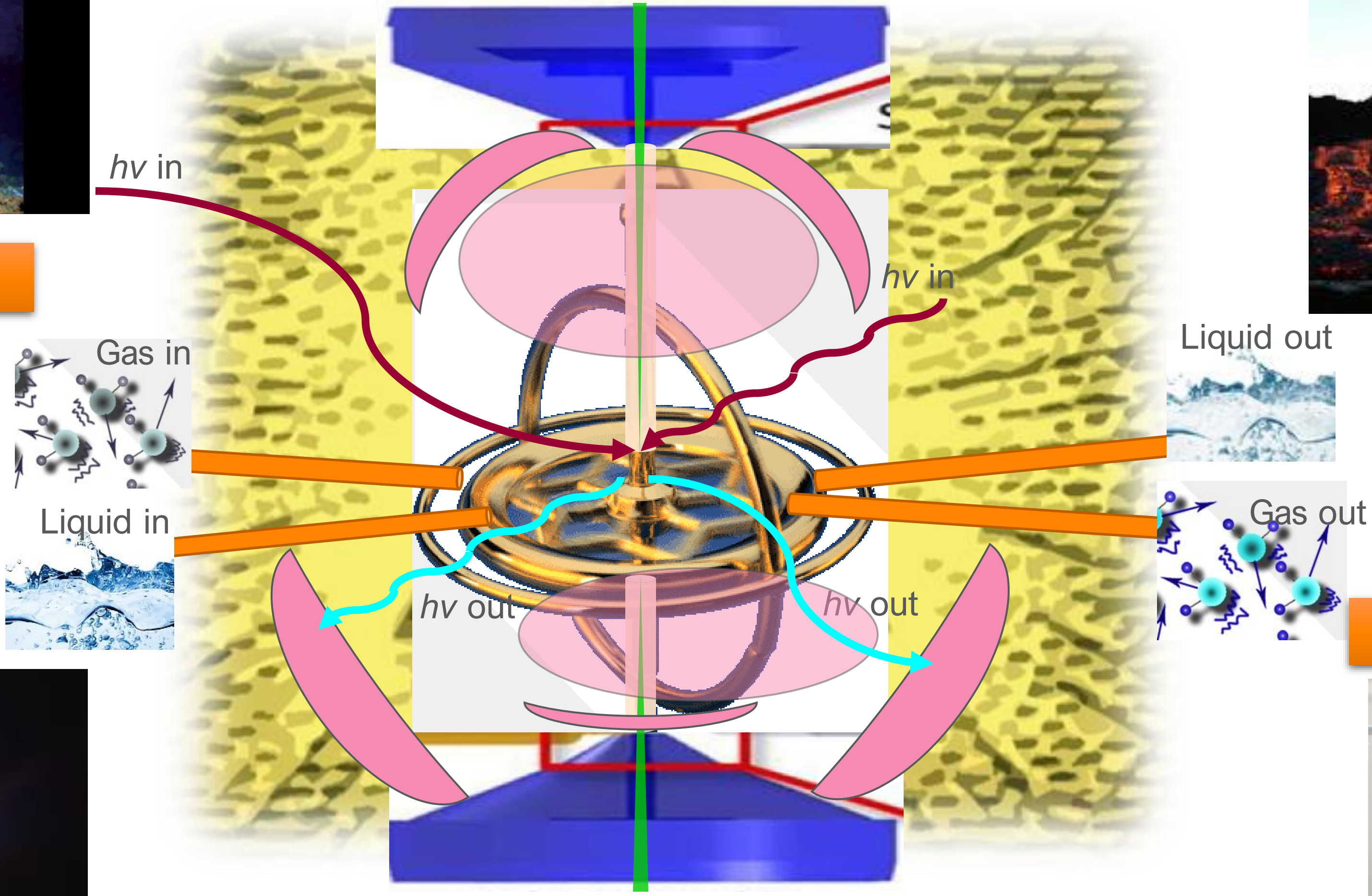
- Nanocrystalline Ni-40Fe, 10-60 nm grain size, 10 μm notch, imaged at 60°
- Cycled at 30 Hz, 4000 cycles between images

The Dream: Testing Greater Extremes in the TEM

Hydrothermal Vents

Advanced Manufacturing

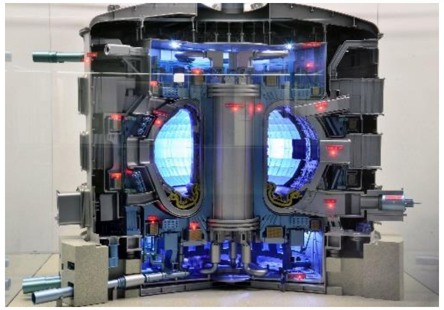
Space Travel



Volcanic Activity

Explosions

Fusion Reactor



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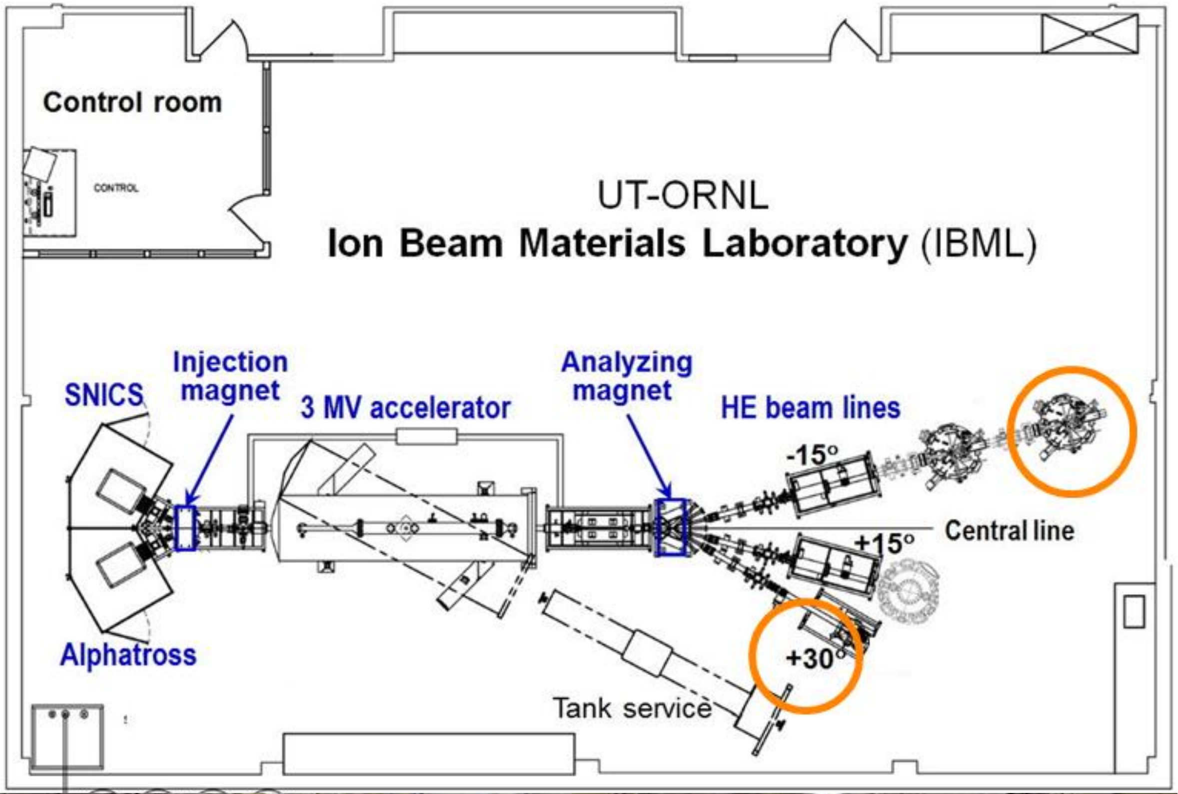
Overview of the Histocial Ion Beam Facility

- ~300 m^2 (45ft x 70ft) – room for growth!
- 3 MV NEC tandem accelerator
- Two ion sources that produce a large variety of ion species between H-Bi ions
  - Source of Negative Ions by Cs Sputtering (SNICS)
  - Alphatross source for helium
- Thermal range: 30 K - 1473 K
- Characterization Techniques:
  - Rutherford Backscattering Spectrometry (RBS), Non-RBS (NRBS), Elastic Recoil Detection (ERD), Time-of-flight (TOF - ERDA), Nuclear Reaction Analysis (NRA)
  - Cathodoluminescence (CL), Ion-beam Induced Luminescence (IBIL), Raman spectroscopy



Zhang, Y. et al. "New Ion Beam Materials Laboratory for Materials Modification and Irradiation Effects Research." NIM:B (2014)

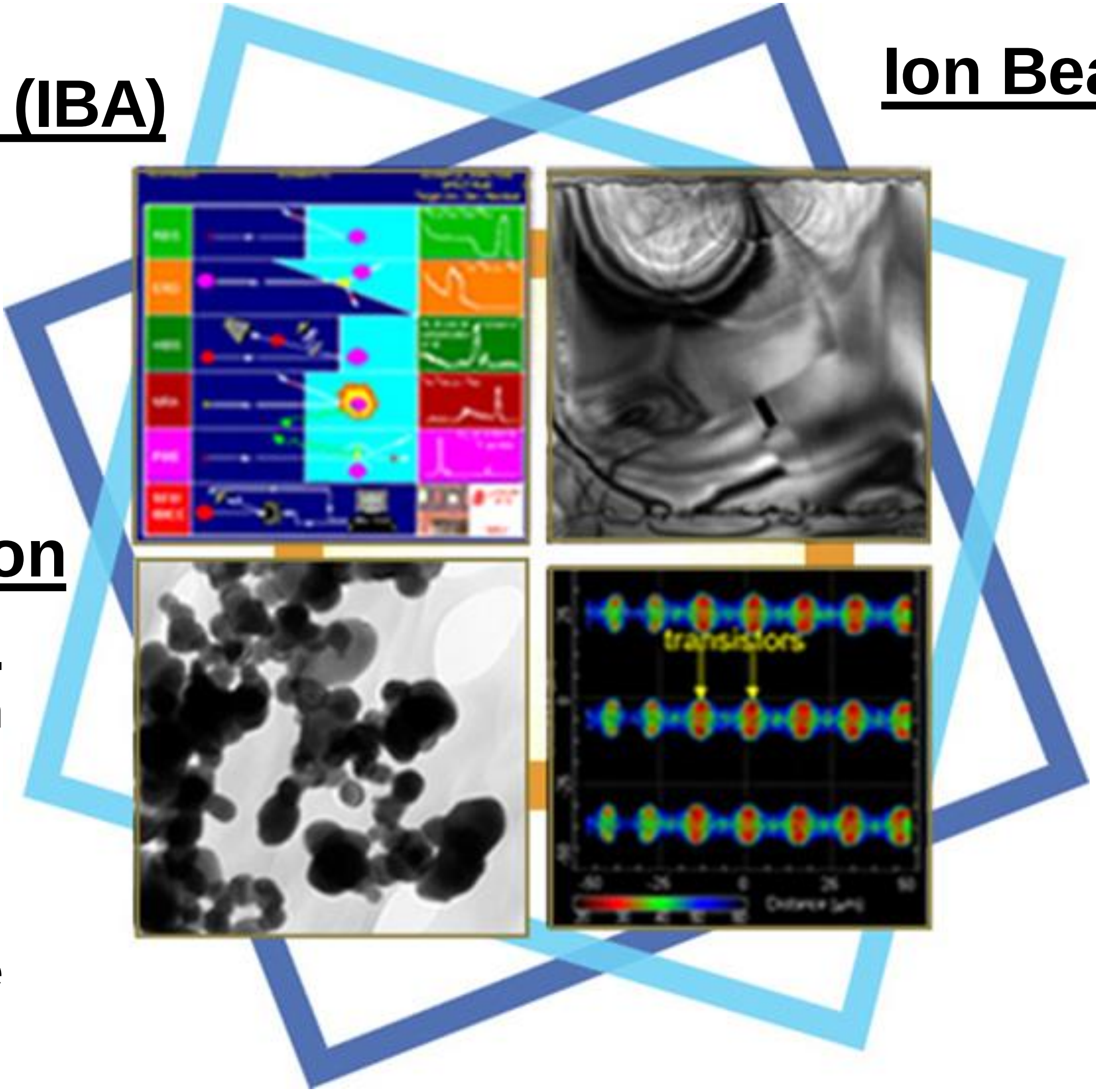
Tennessee Ion Beam Materials Laboratory Vision

Ion Beam Analysis (IBA)

Shooting a charged particle at an unknown material to determine its identity, local chemistry, and structure.

In-situ Ion Irradiation Microscopy (I^3M)

Bombarding samples with various particles and observing the changes in real time to understand how materials will behave in extreme environments.



Ion Beam Modification (IBM)

Changing the optical, mechanical, and chemical properties of materials via ion implantation to meet technological needs

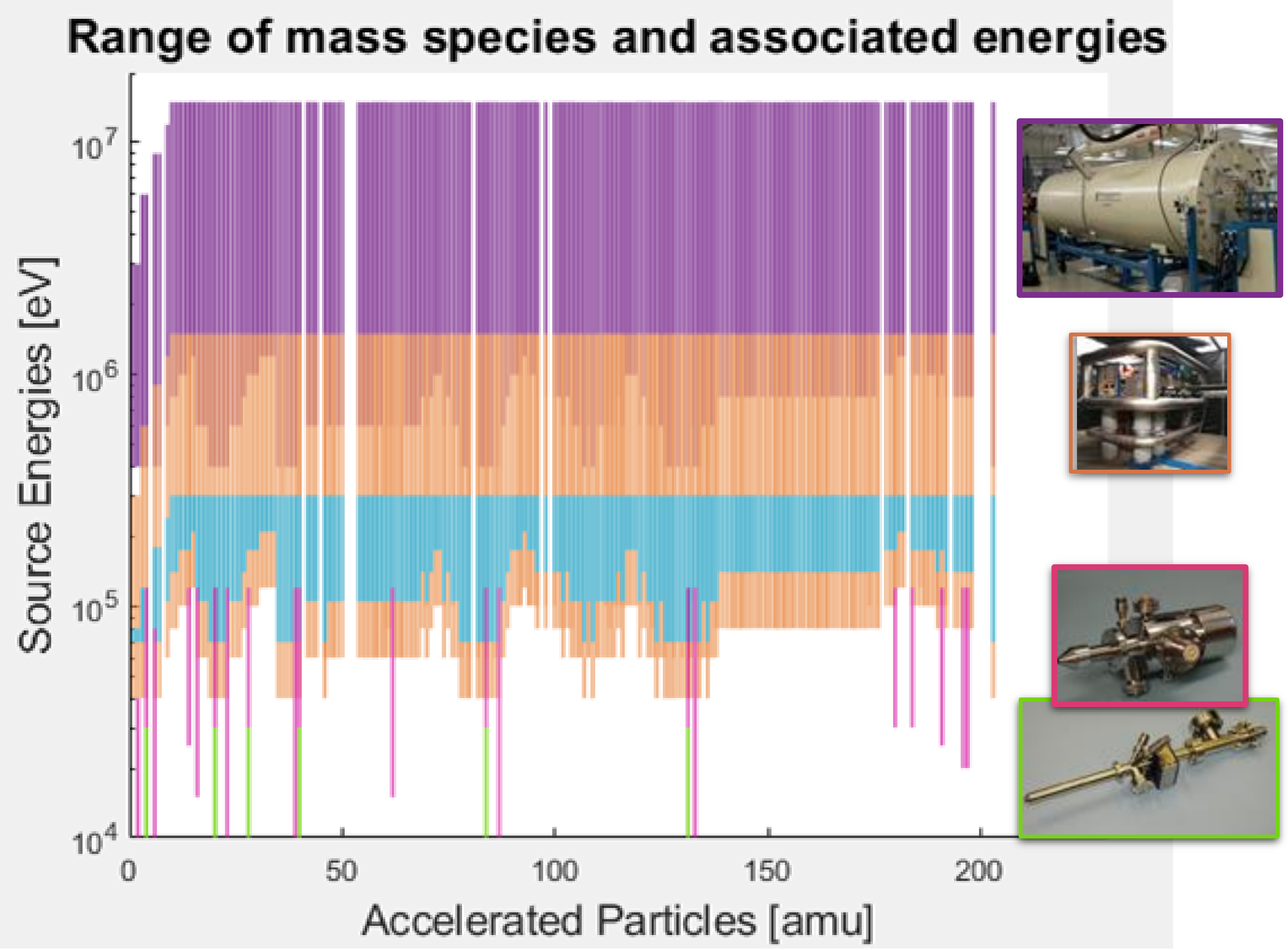
Radiation Effects Microscopy (REM)

Using ion emissions to determine the radiation hardness of microelectronics, identifying potential weaknesses

Collective Capabilities

Ion Sources

- 3 MV NEC Tandem Accelerator
  - H-Bi ions: 400 keV - 15 MeV
- 300 kV NEC ion implanter
  - H-Bi ions: 40 keV - 500 keV
- 48 kV Low Energy Beamline
  - H-Bi ions: 20 keV - 60 keV
- 20kV Ion Gun
  - Inert gases and N_2 : 10 keV - 120 keV
- 5kV Ion Gun
  - Inert gases and N_2 : 20 eV - 30 keV



Electron Sources

- 1-30 kV for electron gun
- 80 kV - 200 kV electrons from TEM

TEM Delivered! - 9/21/2023

JEOL 2100+ Features

- 200 kV, Transmission Electron Microscope (TEM)
- High tilt , ±81°, tilt permitting a range of tomographic techniques
- C0 lens, increases brightness 5x
- Resolution
  - Point: 0.25 nm & Lattice: 0.14 nm
- Gatan Clearview camera
  - Can capture 50 frames per second at 4k resolution (1600 bins +readout)
- Customized: Ports added for ion beam and a high powered laser

JEOL 2100+

2010 – NEC 3 MV Tandem ordered

2012 – First Beam

2014 – First Publication acknowledging I BML

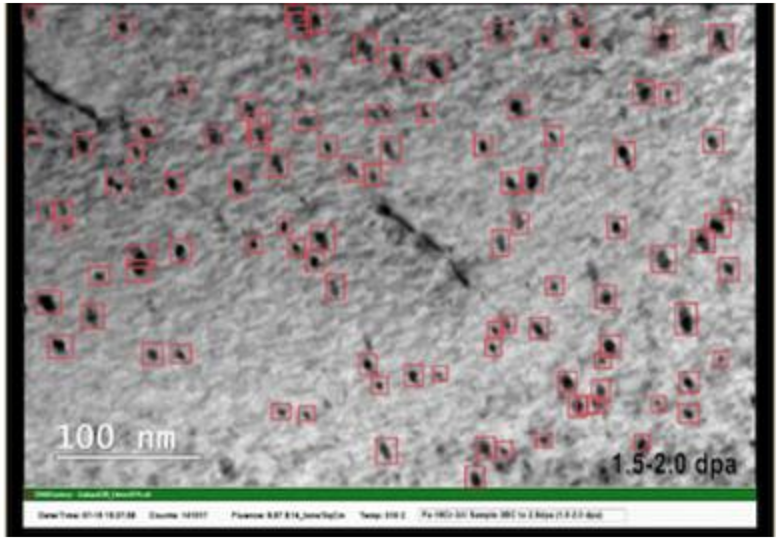
2023 – Dr. Hattar TIBML

Sept 2023

New TEM Capabilities at TIBML

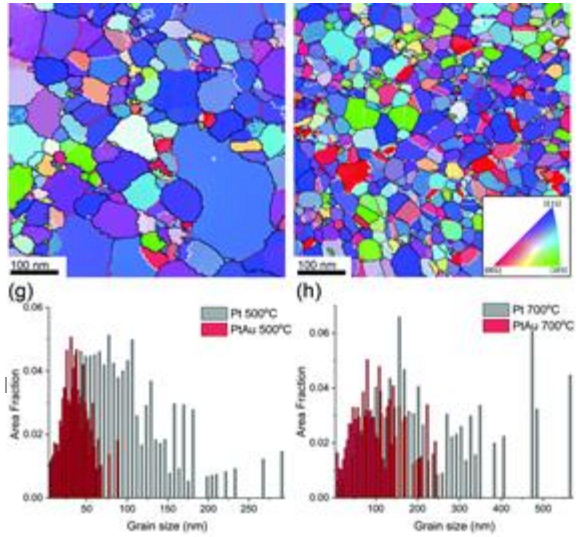
JEOL 2100+ & Theiascientific

- Edge computing system that gives scientists access to ML and computer vision
- In-situ, automated quantitative microscopy image analysis



JEOL 2100+ & ASTAR

- Automatic TEM phase-orientation mapping
- Compares recorded patterns to database of theoretical diffraction patterns to determine relative orientation of crystalline grains, and consequently material texture [1]

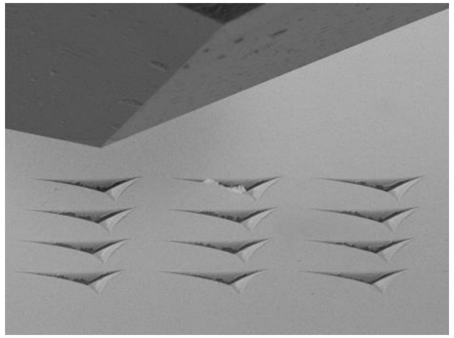


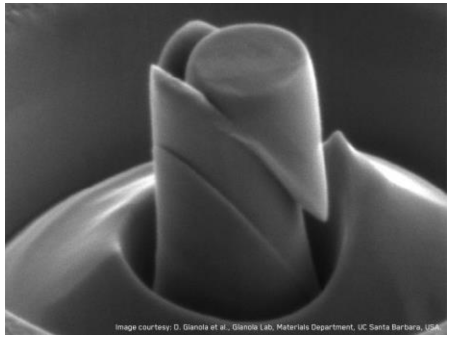
- **2010** — **NEC 3 MV Tandem ordered**
- **2012** — **First Beam**
- **2014** — **First Publication acknowledging IBML**
- **2023** — **Dr. Hattar TIBML**
- **Sept 2023**

[9] Theiascietnific: https://www.theiascientific.com/#
[10] C. Bar. "The role of grain boundary character in solute segregation and thermal stability of nanocrystalline Pt–Au" *Royal Society of Chemistry* (2021)

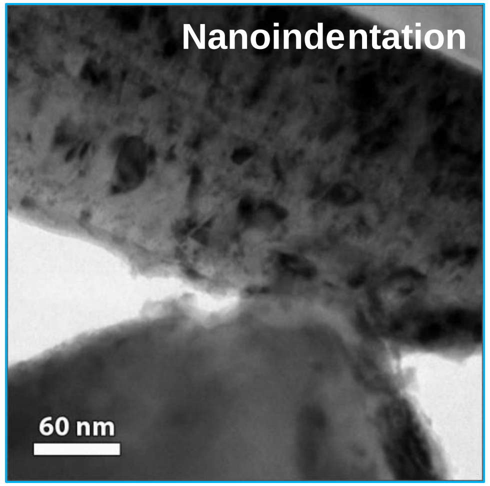
Next TIBML Capabilities: Nanomechanical Testing

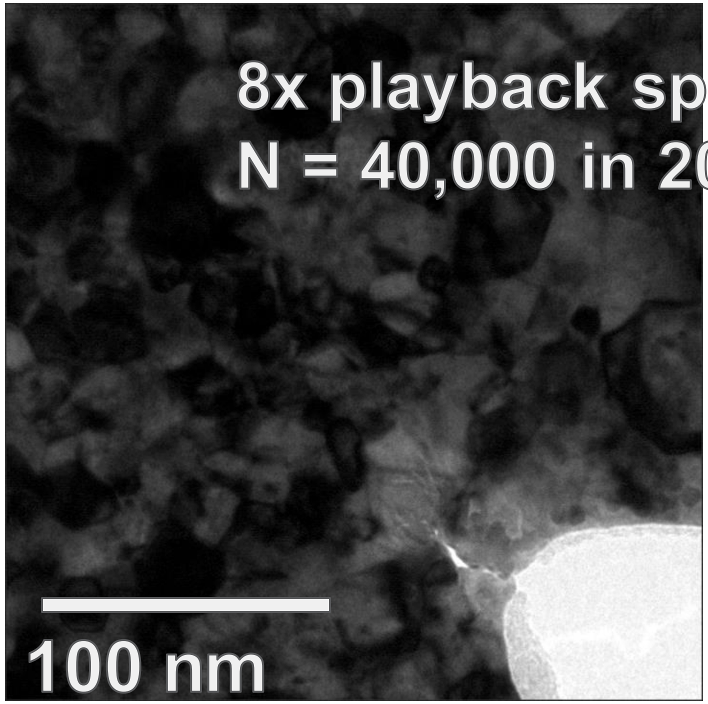
Next Step -Femtotools





Vision Bruker/Hysitron PI-95





SEM/OM length-scale

- CSM, Pillar compression, lock-key tensile, fatigue, etc.
- Force Range 0.5 nN – 200 mN
- Position Sensing 50 pm – 21 mm
- Delivery before 11/1/2024

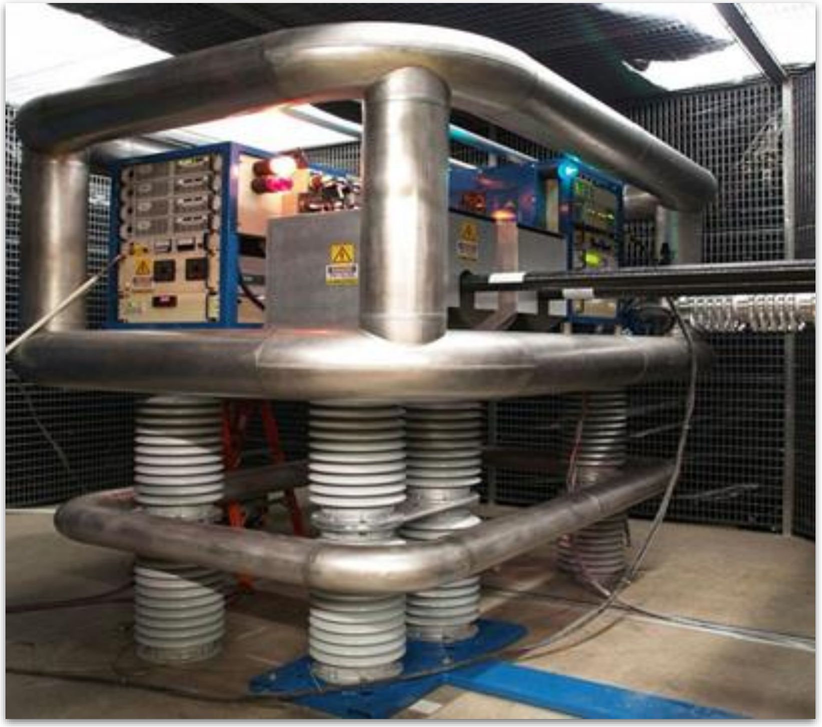
TEM length-scale

- Quantitative force information with µN resolution
- In-situ TEM indentation, pillar compression, lock-key tensile, fatigue, creep, etc.

Upcoming Capabilities: Implanter

<u>300 kV NEC Ion Implanter Features</u>

- 20 - 300 kV energy range
- Vacuum base pressure without beam of 5 x 10^{-8} Torr
- Mass to charge ratio greater than or equal to 200
- Large variety of ions ranging from H - Bi can be produced
- All-metal and ceramic accelerator tube, bakeable to 500°
- Electrostatic quadrupole triplet lens can control beam spot size from 6 mm at 20 keV - 2 cm at 10 keV
- First End station will be dual beam



2010 – NEC 3 MV Tandem ordered

2012 – First Beam

2014 – First Publication acknowledging I BML

2023 – Dr. Hattar TIBML

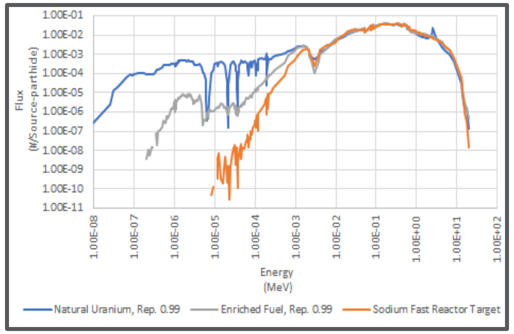
Sept 2023

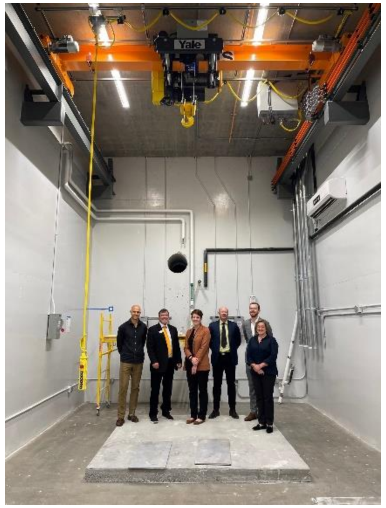
Dec 2023

Dec 2024

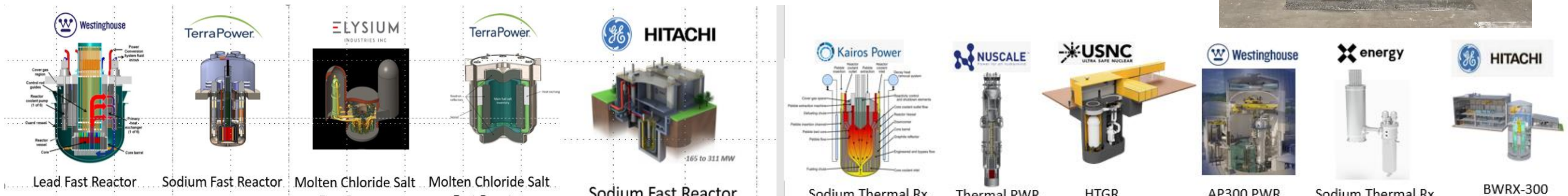
Flexible Neutron Source

- Highly Flexible neutron generator driven **Subcritical** Core designed to replicate the neutron spectrum of any fast reactor (Pb, Na, MSR), thermal reactor, or detonation by using artificial intelligence guided core design.
- Used for
  - Nuclear data uncertainty reduction to support
    - advanced reactor designs
    - nuclear security applications
  - HALEU benchmarking for transportation regulatory support
  - Neutron filter design studies for isotope production
  - Neutron detector validation studies
  - Nuclear criticality safety training



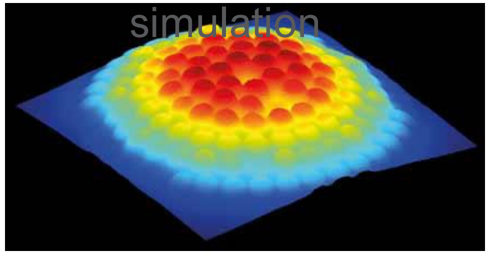


Higher uncertainty in nuclear data results in more conservative designs making plants more expensive and less competitive

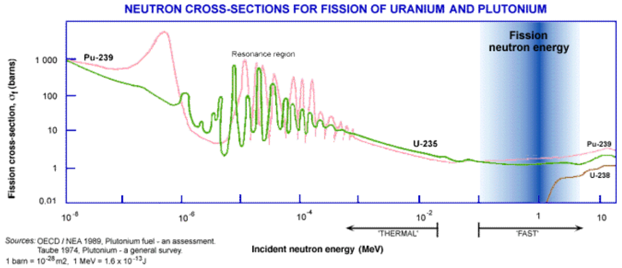


Fast Reactor Cross Section Data Needs

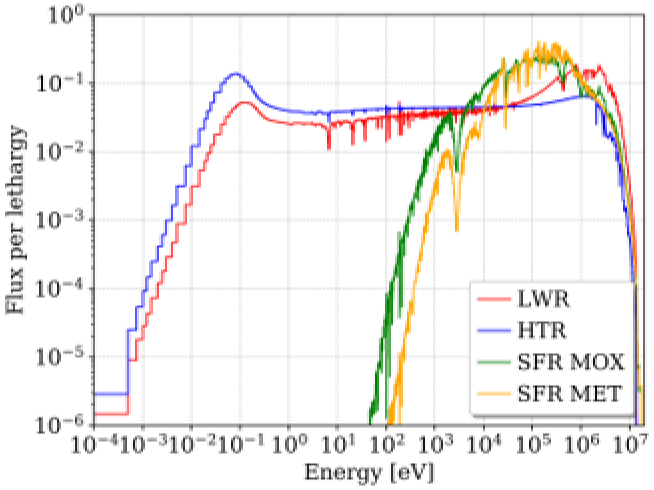
Next generation (fast reactor) reactor design is strongly dependent on modeling & simulation

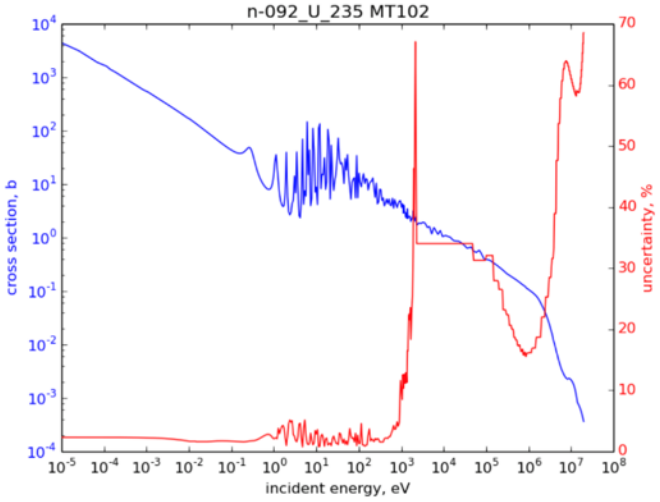


Modeling & simulation depends on nuclear data



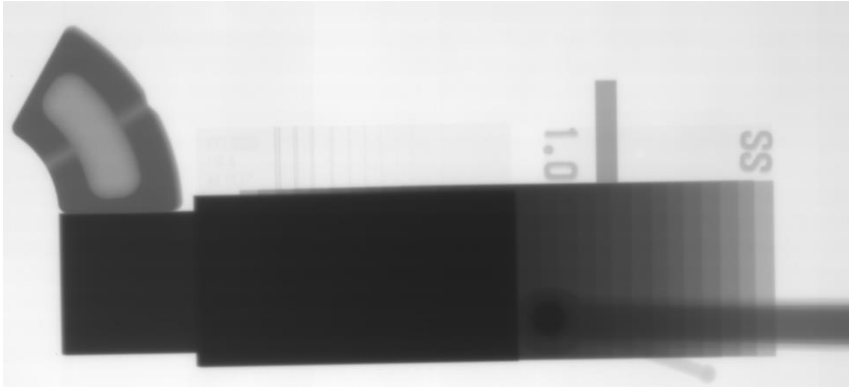
Higher uncertainty in nuclear data will result in more conservative design which will make the plants more expensive and less competitive

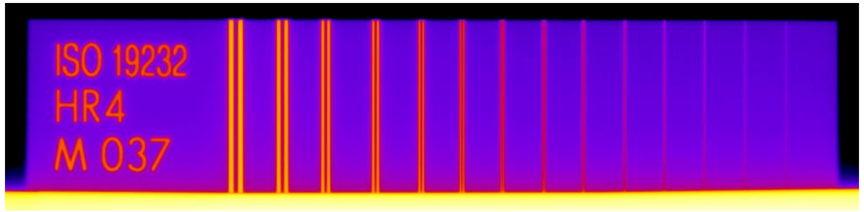


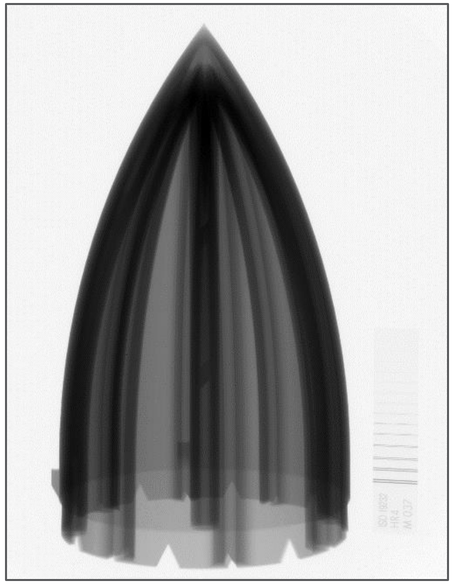


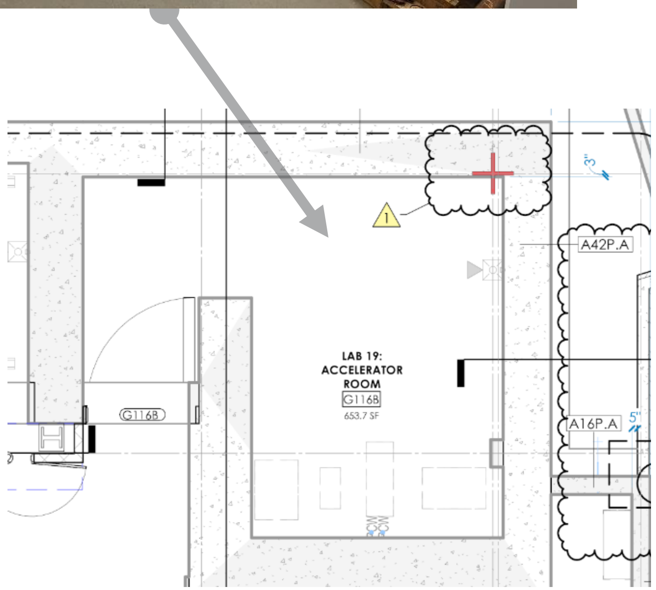
Accelerator Research Center

- Used for general purpose radiography and irradiation.
- Houses: 9MVp Linac and 450kVp Comet mesofocus x-ray sources.



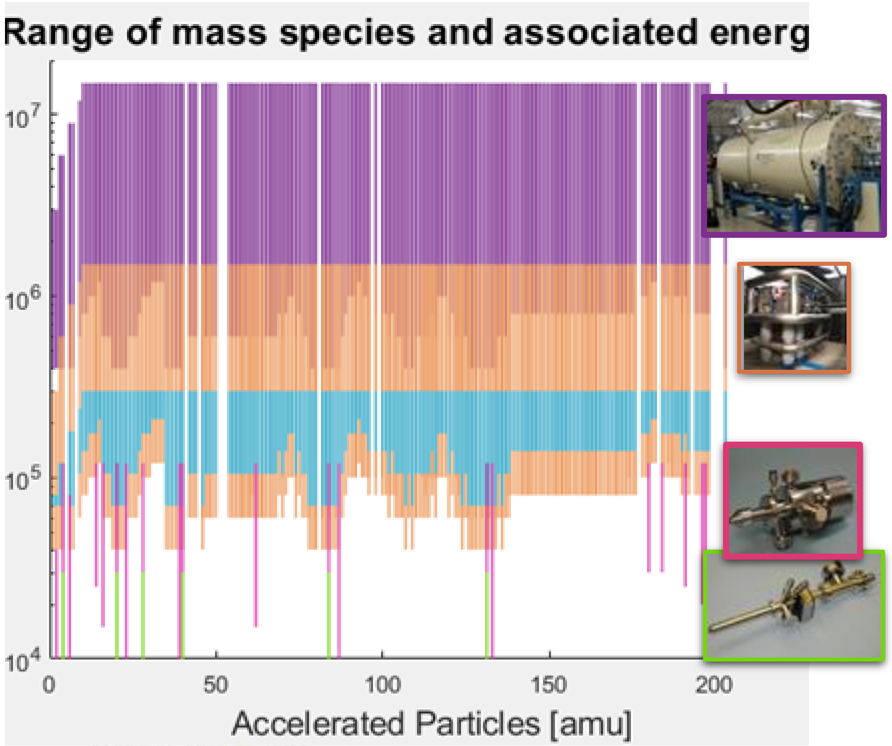






Summary

- TIBML can produce a wide range of ion energy species
- Comprehensive set of IBA characterization techniques
- Planned upcoming capabilities will help create extreme coupled conditions
- Advanced in-situ microscopy and machine learning will permit a deeper understanding of microstructural evolution and subsequent changes in properties



Currently applying the juxtaposed capabilities to various material systems in combined environmental conditions essential for many Nuclear Energy Applications

CINT — Center for Integrated Nanotechnologies

NSUF Nuclear Science User Facilities

TIBML Website

www.TIBML.UTK.edu