In-situ Ion Beam Microscopy Capabilities at the Sandia Ion Beam Laboratory

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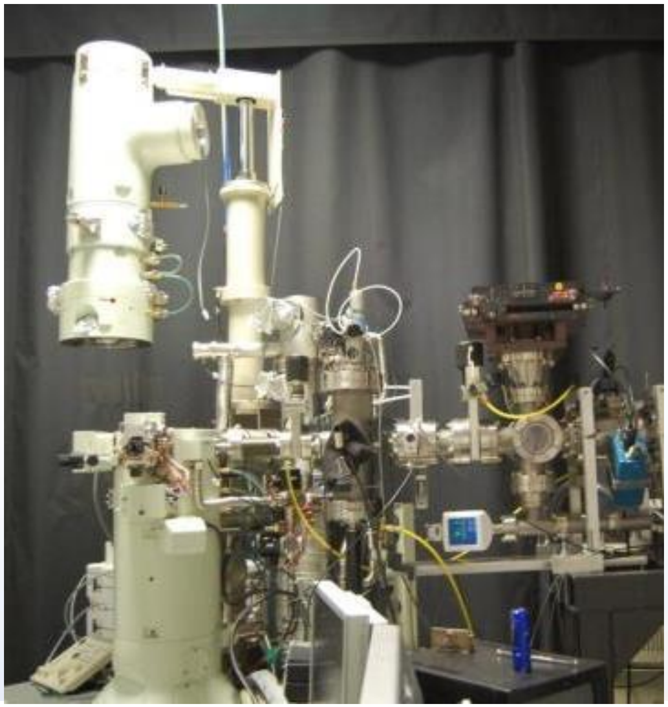
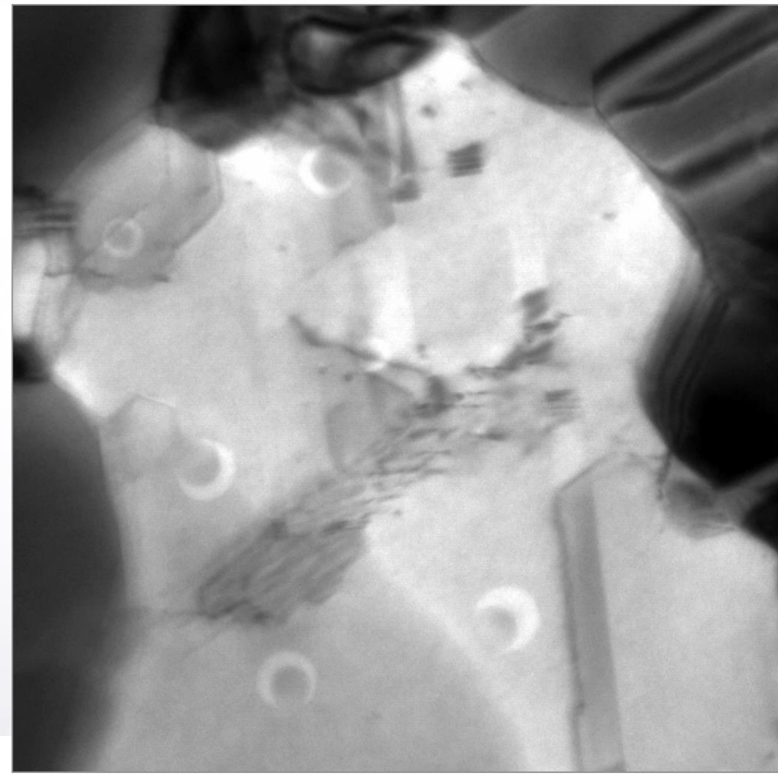
S.A. Briggs, C.M. Barr, P.M. Price, C.A. Taylor, B.R. Muntifering, D.C. Bufford, K. Hattar

Sandia National Laboratories

November 29th, 2017



In situ TEM microscopy has recently undergone significant growth allowing for dynamic observation of structural evolution that occurs due to exposure to various extreme environments relevant to nuclear applications



Collaborators:

- Sandia: M. Abere, B. Boyce, T.J. Boyle, D. Buller, C. Chisholm, B.G. Clark, R. Dingreville, R.F. Hess, A.C. Kilgo, B.E. Klamm, M.T. Marshall, W.M. Mook, S.H. Pratt, J.D. Puskar, J.A. Scott, & J.A. Sharon
- External: A. Aitkaliyeva, H. Bei, P.J. Ferreira, K.J. Ganesh, E.P. George, D. Gross, P. Hosemann, J. Kacher, S. Maloy, A. Minor, J. Qu, S. Rajesekhara, I.M. Robertson, D. Stauffer, & Hysitron Inc.

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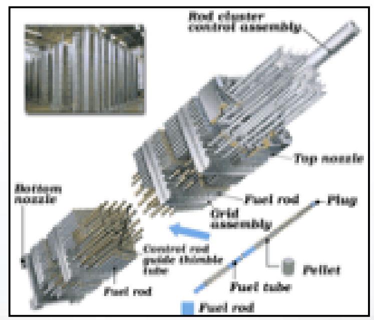
Investigating the nm Scale to Understand the km Scale

1 nm 1 μm 1 mm 1 m 1 km 10^7 m



In situ Ion Irradiation TEM (I^3TEM)

Ion Beam Lab (IBL)

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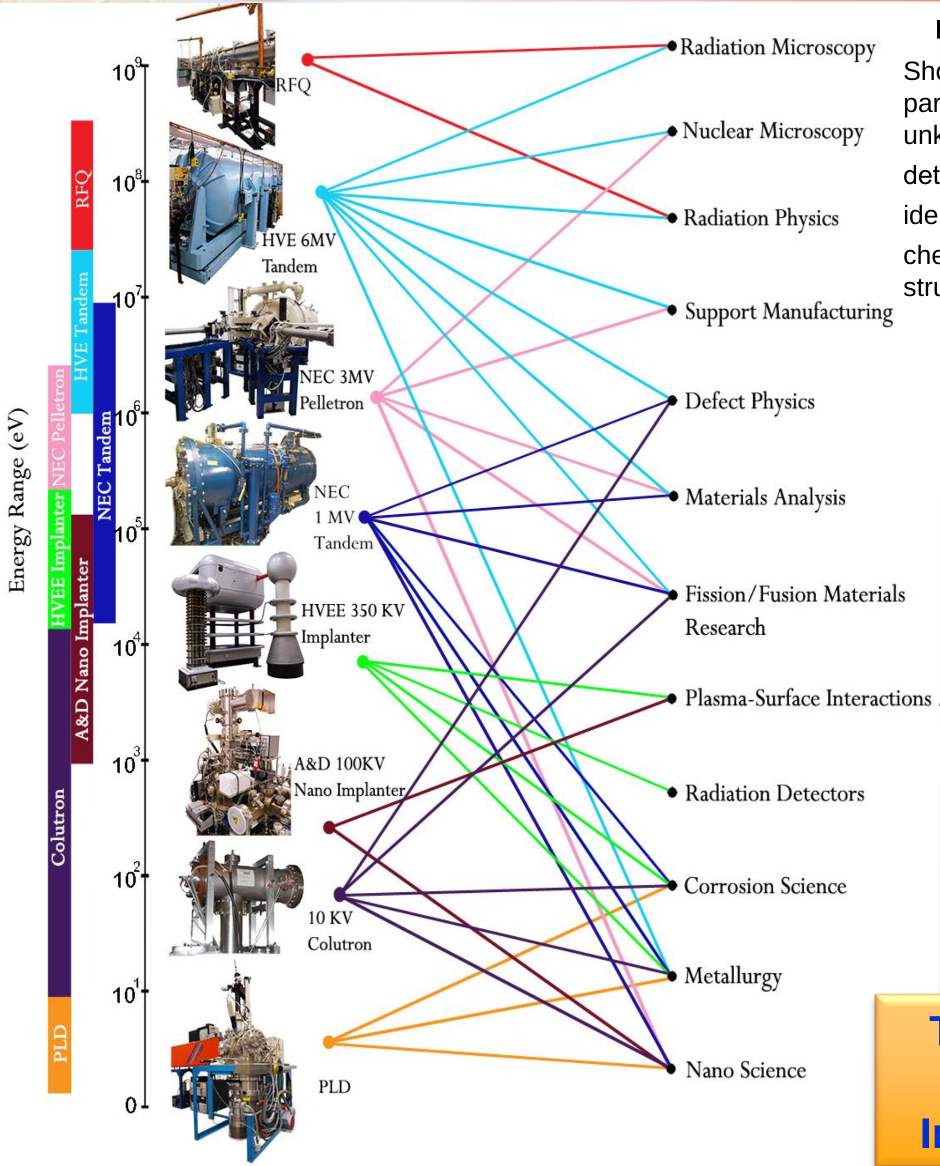
Sandia's Ion Beam Laboratory

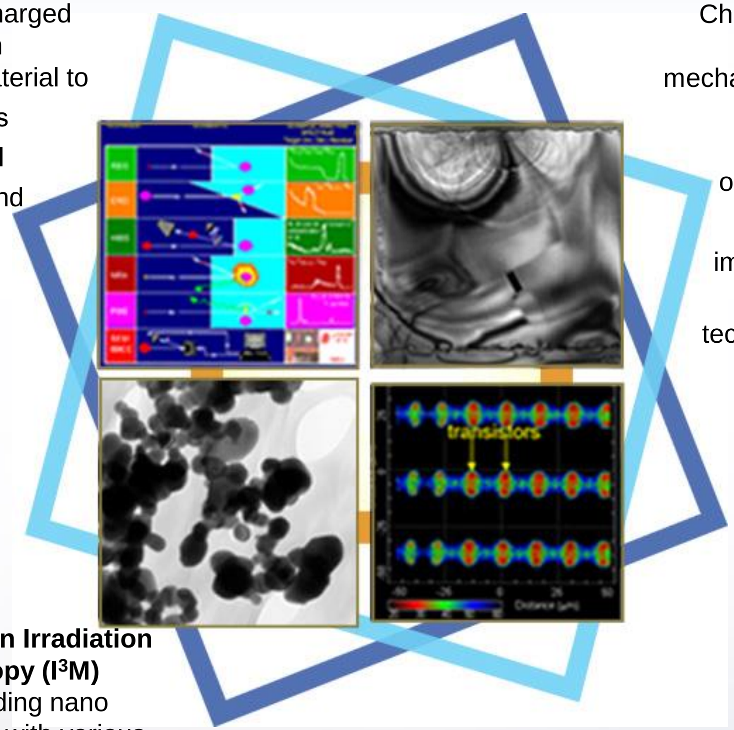


Ion Beam Analysis (IBA)

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

Ion Beam Modification (IBM)

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



***In Situ* Ion Irradiation Microscopy (I³M)**

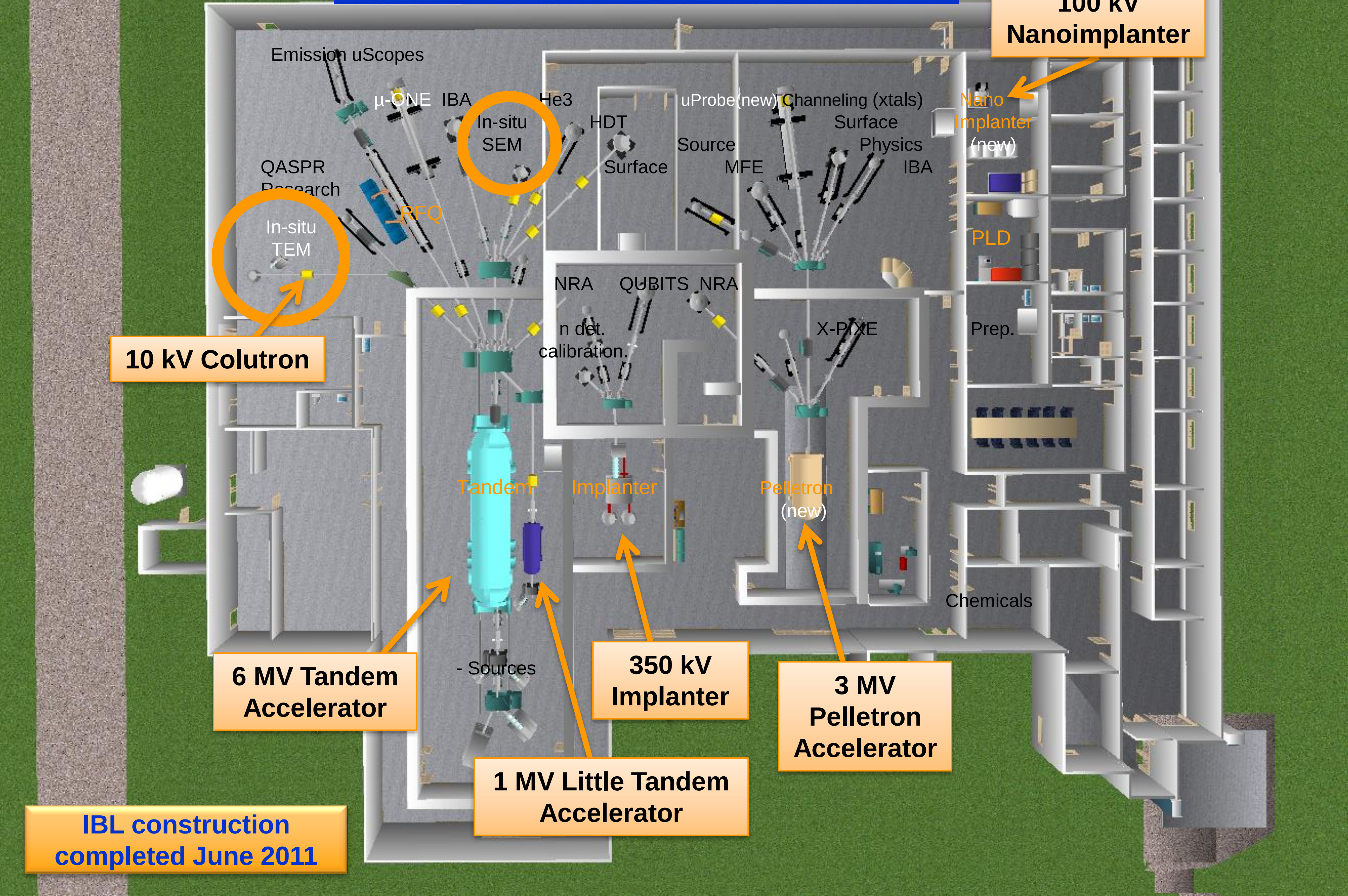
Bombarding nano samples with various particles and observing the changes in real time to understand how materials will behave in extreme environments.

Radiation Effects Microscopy (REM)

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

The IBL has a unique and comprehensive ion beam capability set including and *In situ* Ion Irradiation Transmission Electron Microscopy.

New Ion Beam Laboratory Content



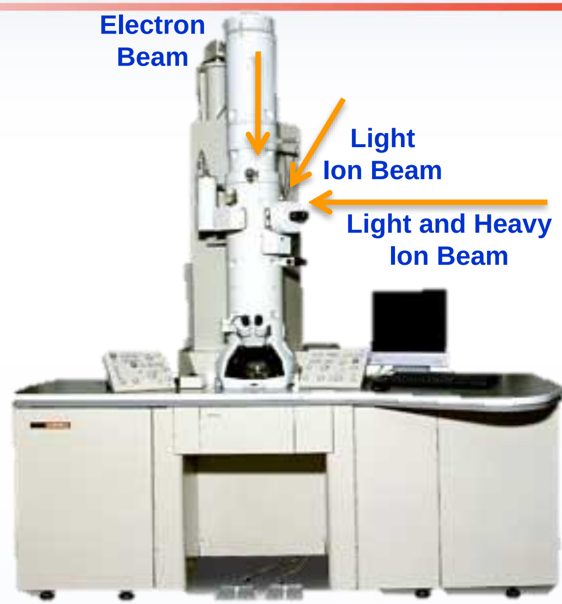
In situ Ion Irradiation TEM Facility

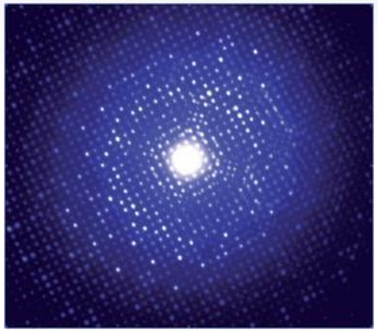
Proposed Capabilities

- 200 kV LaB_6 TEM
- Ion beams considered:
  - Range of Sputtered Ions
  - 10 keV D^{2+}
  - 10 keV He^{+}
- All beams hit same location

- Nanosecond time resolution (DTEM)
- Procession scanning (EBSD in TEM)
- *In situ* PL, CL, and IBIL

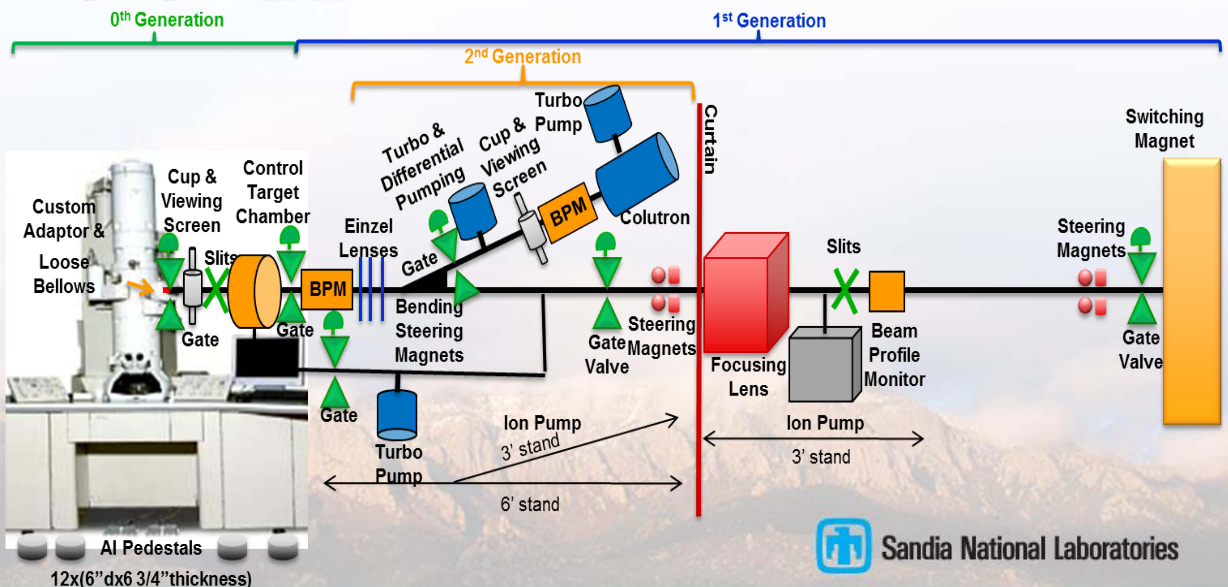
- *In situ* vapor phase stage
- *In situ* liquid mixing stage
- *In situ* heating
- Tomography stage (2x)
- *In situ* cooling stage
- *In situ* electrical bias stage
- *In situ* straining stage (3x)





TVIPS

Hummingbird



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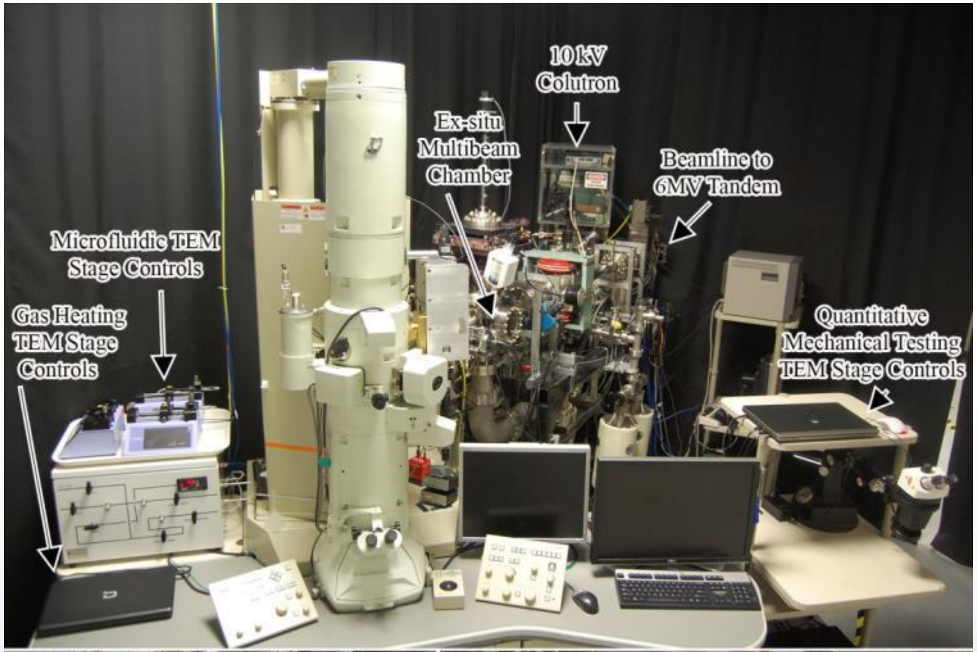
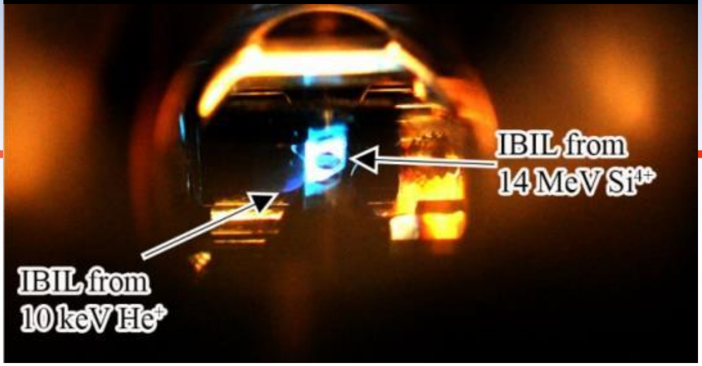
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem

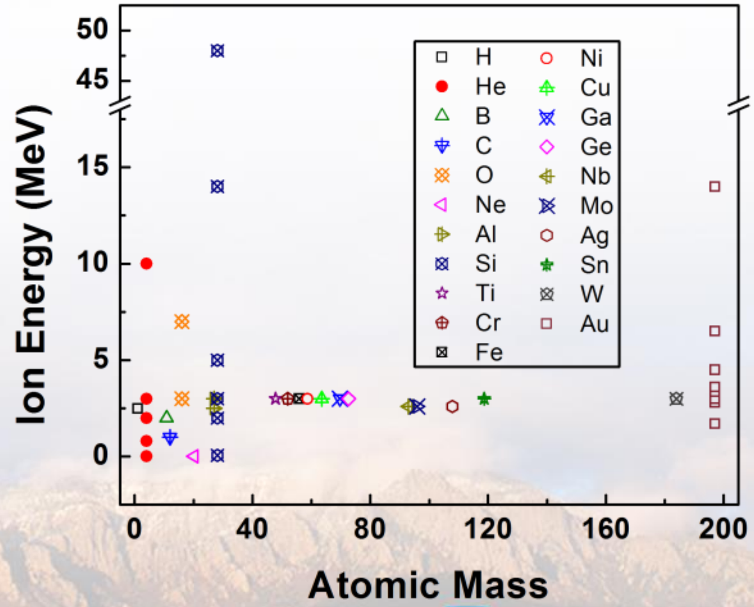


IBIL from a quartz stage inside the TEM



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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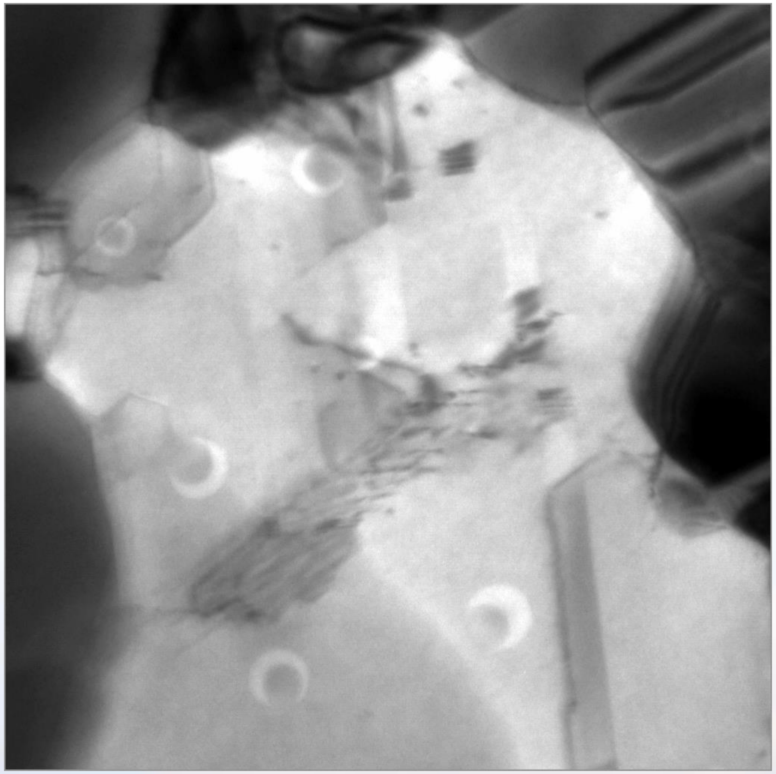
Wide Range of Damage Rates

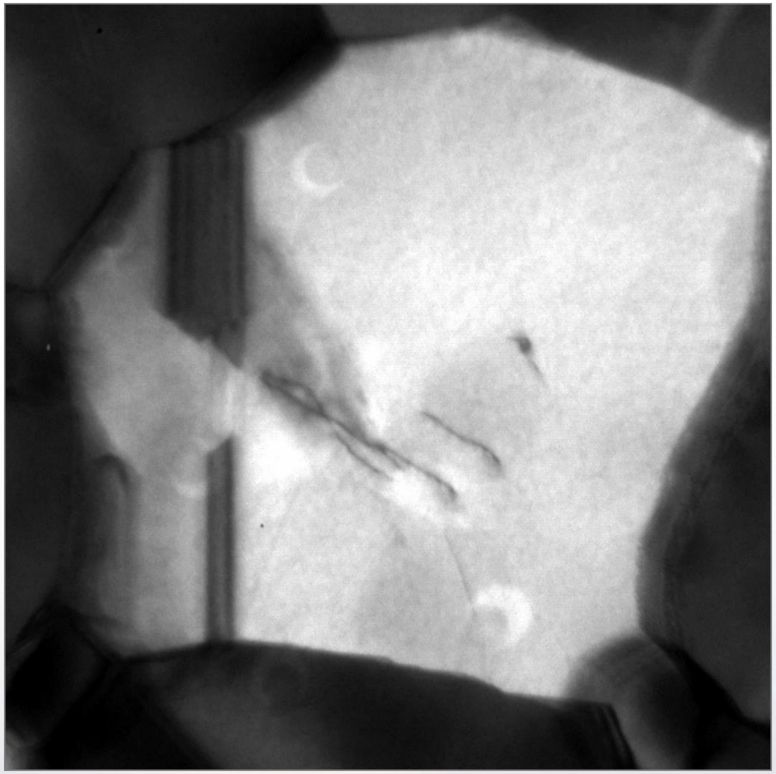
Collaborators: C. Chisholm & A. Minor

7.9 x 10^9 ions/cm^2/s



VS

6.7 x 10^7 ions/cm^2/s



Improved vibrational and ion beam stability permits us to work at 120kx or higher permitting imaging over a wide range of damage rates

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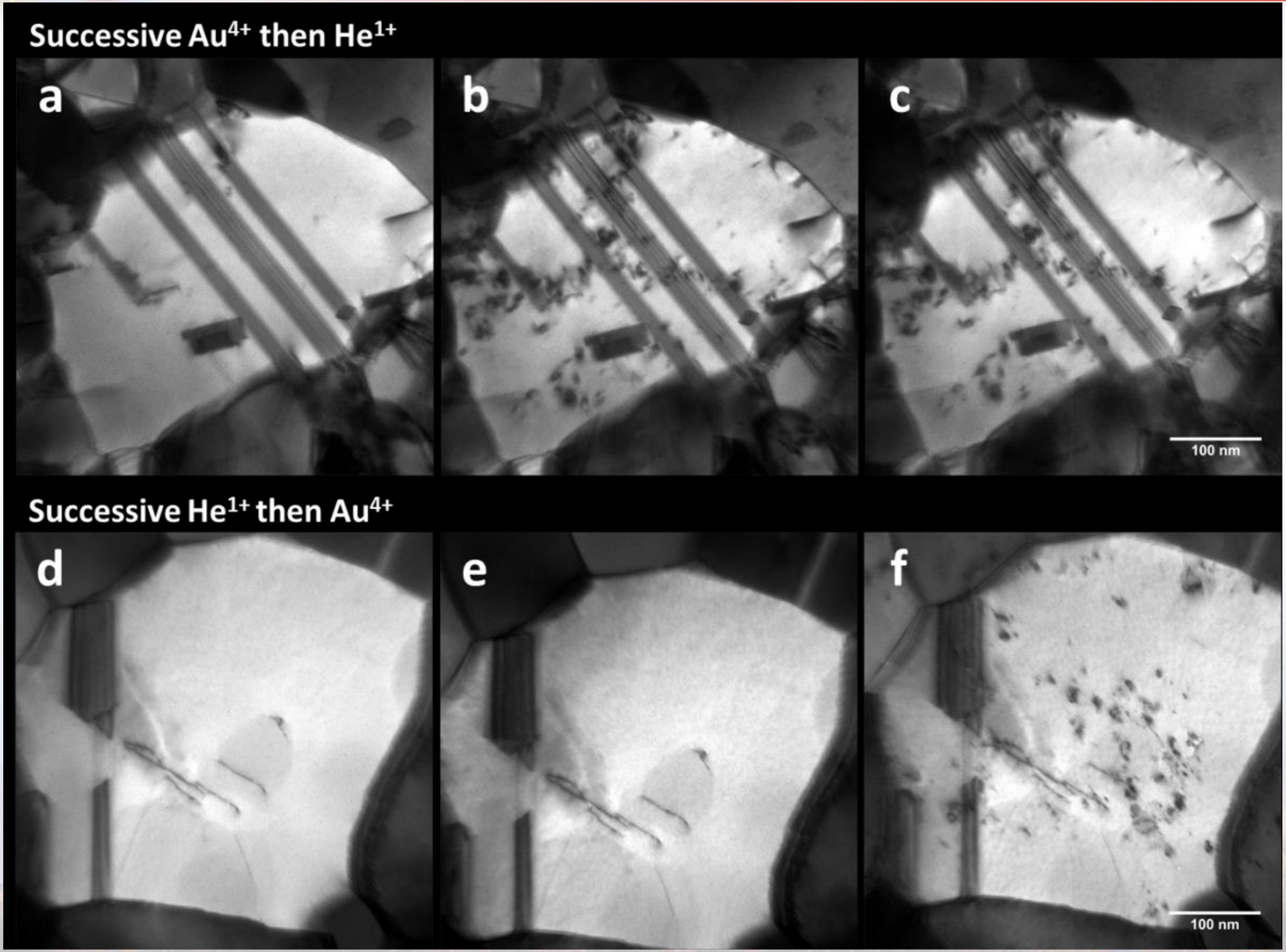
In situ Successive Implantation & Irradiation

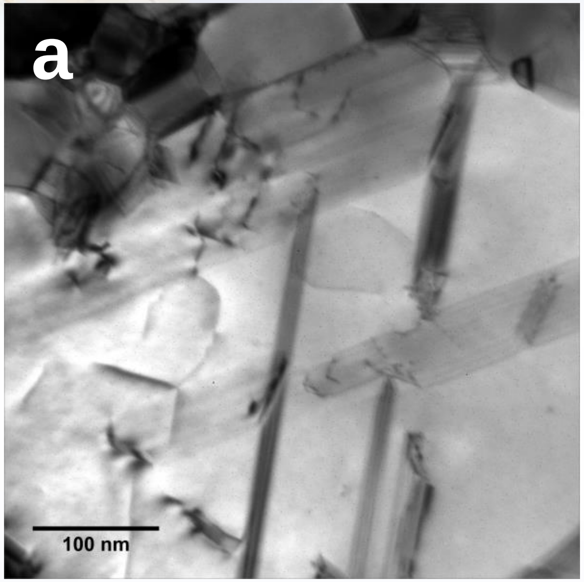
Collaborators: C. Chisholm & A. Minor

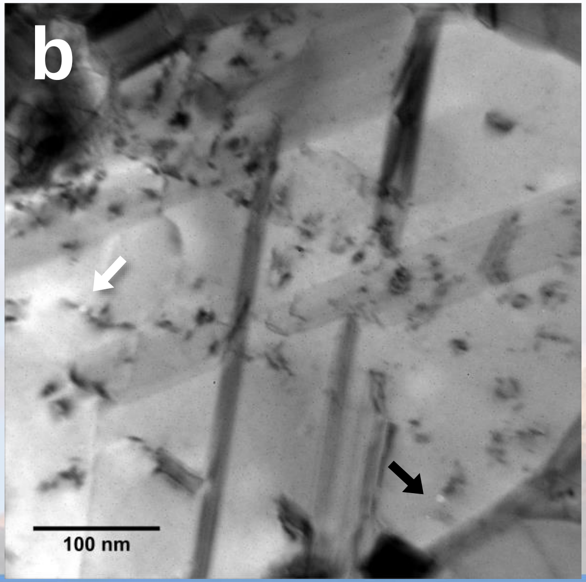


Laboratories

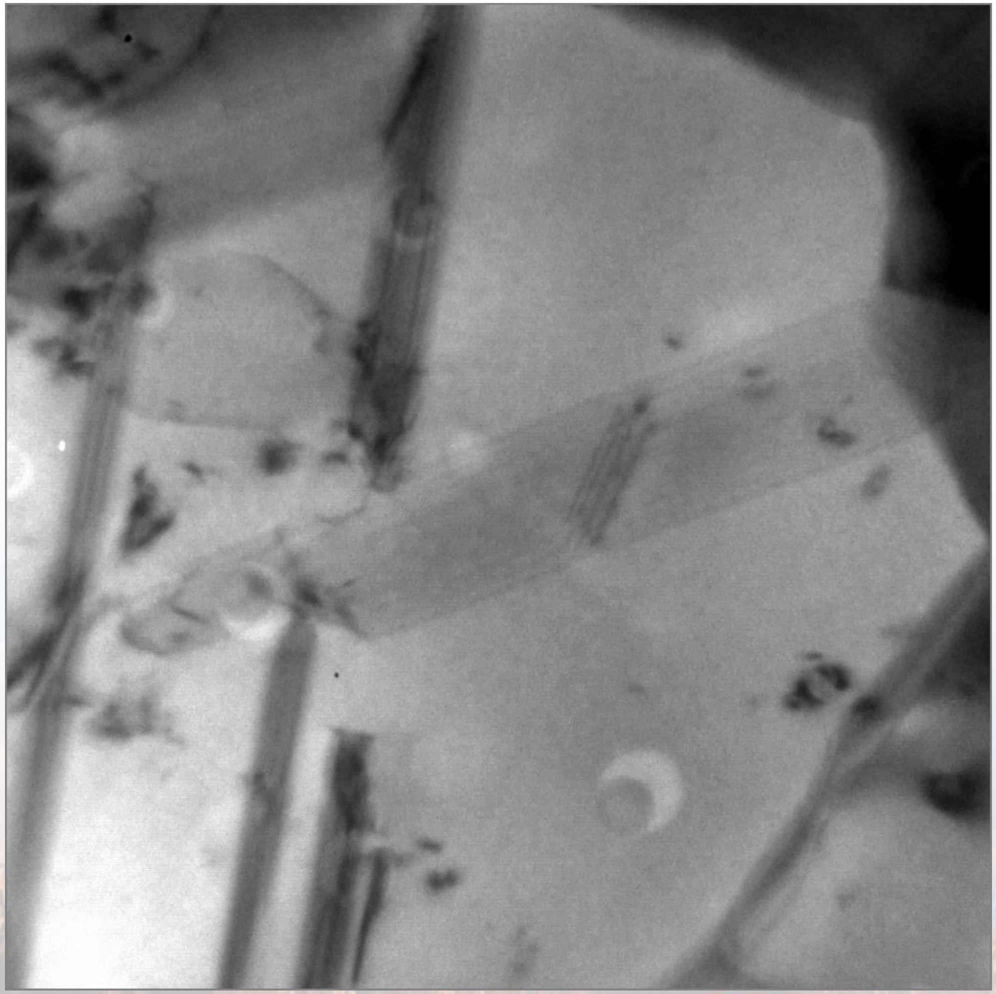
In situ Concurrent Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor





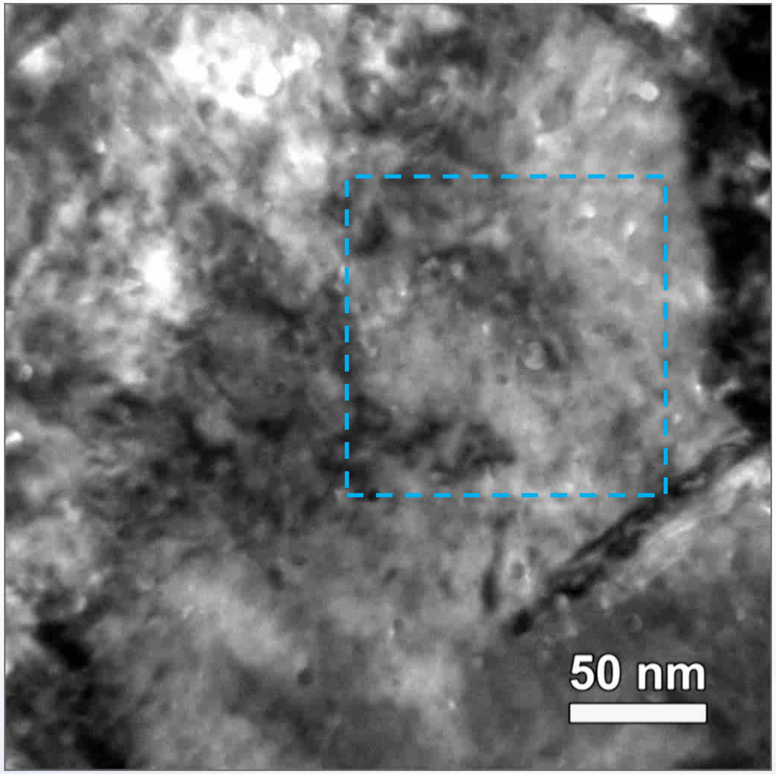
He^{1+} implantation and Au^{4+} irradiation of a gold film

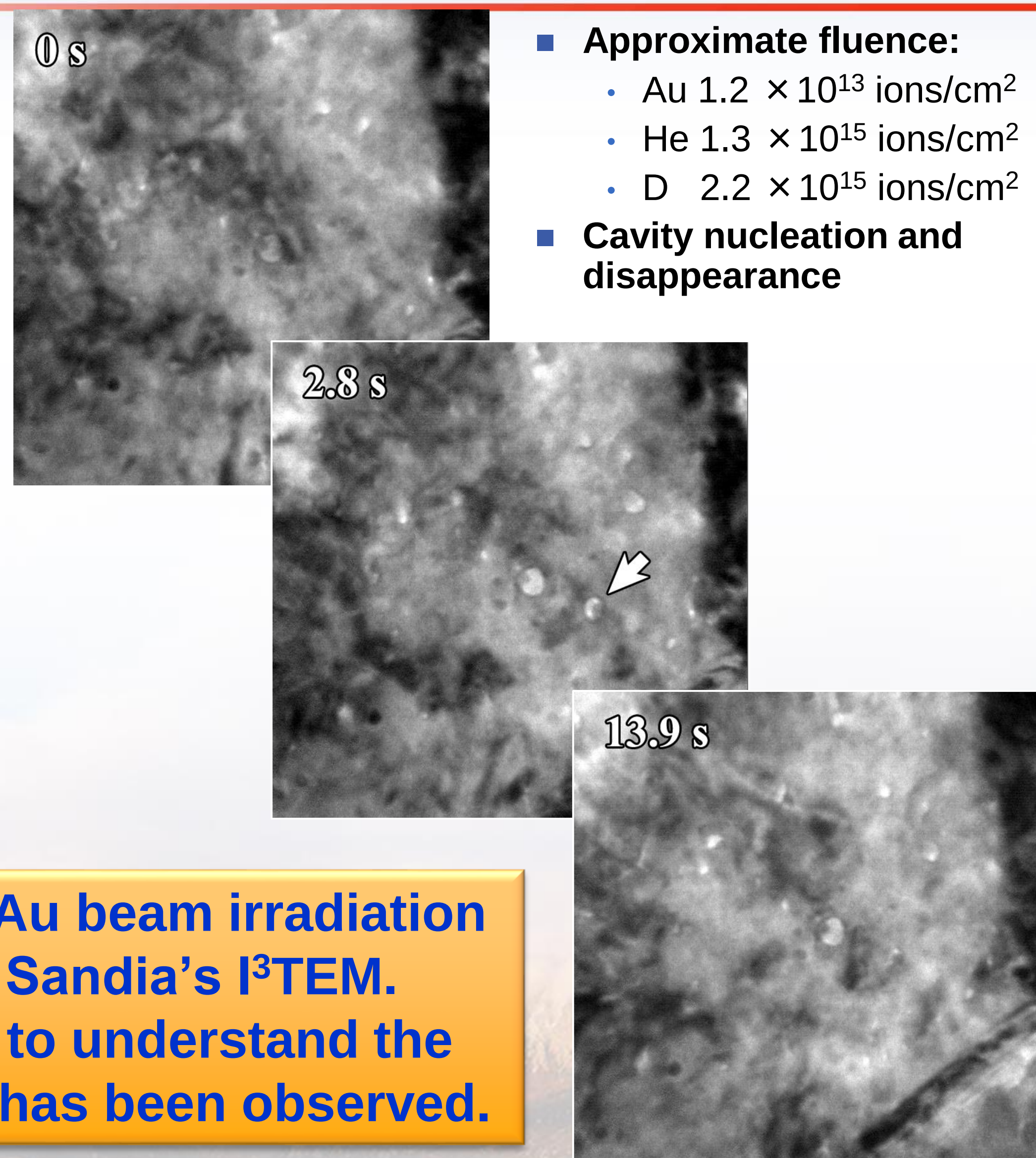


2.8 MeV Au^{4+} + 10 keV He^{+}/D_2^{+}

Collaborators: D.C. Bufford

Video playback speed x1.5.



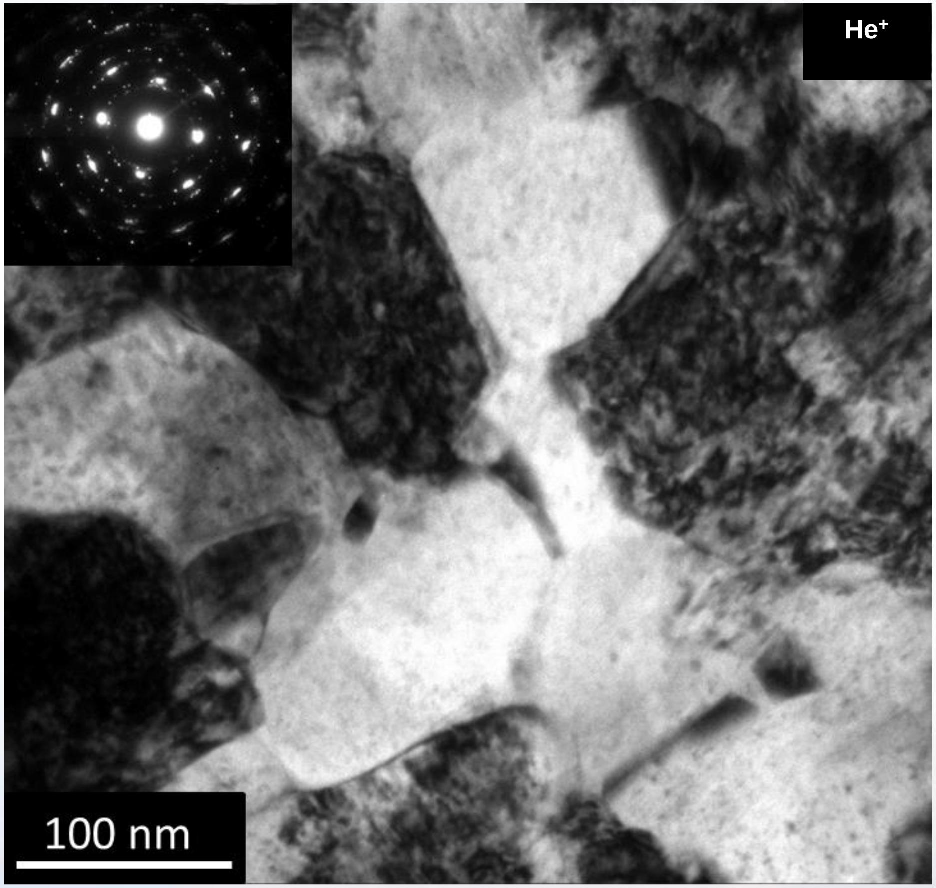


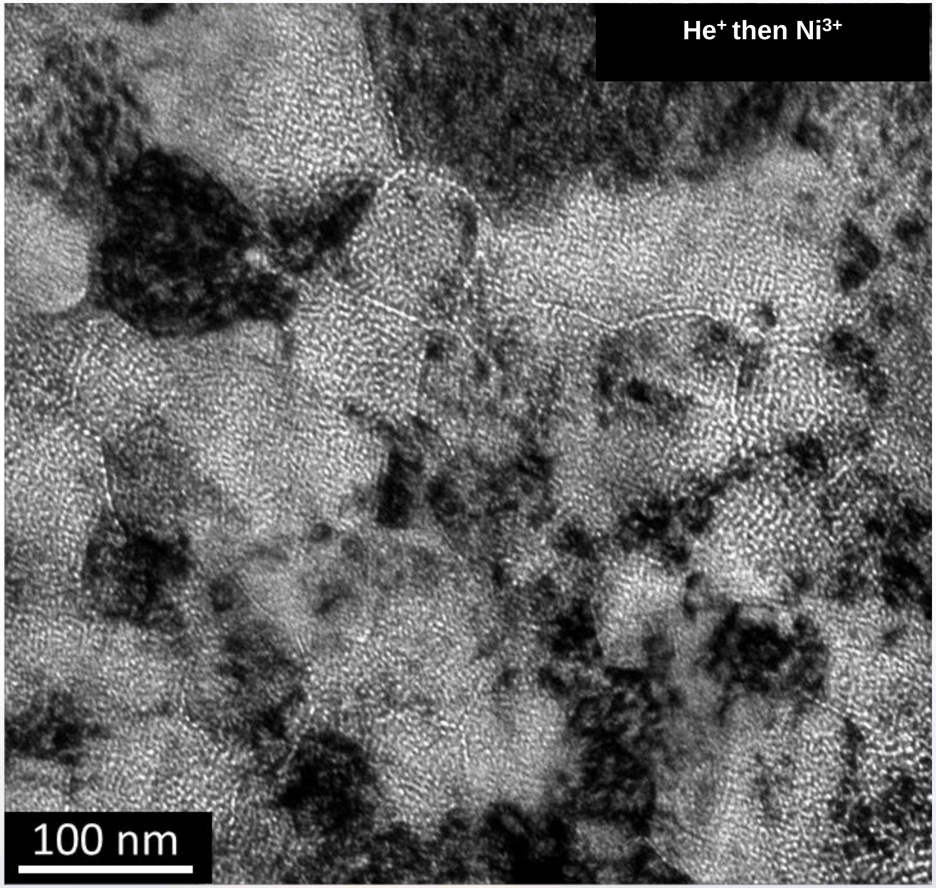
- **Approximate fluence:**
  - Au 1.2 $\times 10^{13}$ ions/cm^2
  - He 1.3 $\times 10^{15}$ ions/cm^2
  - D 2.2 $\times 10^{15}$ ions/cm^2
- **Cavity nucleation and disappearance**

In-situ triple beam He, D_2, and Au beam irradiation has been demonstrated on Sandia's I^{3}TEM. Intensive work is still needed to understand the defect structure evolution that has been observed.

Heterogeneous Bubble Formation under Some Radiation Environments

Collaborator: B. Muntifering & J. Qu





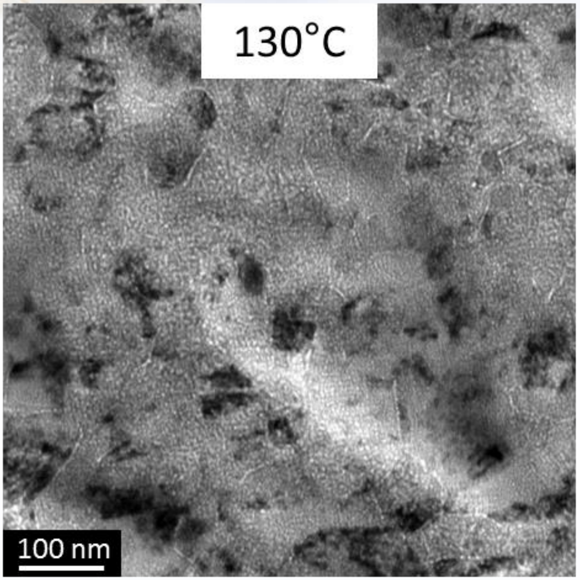
10^{17} He^+/cm^2

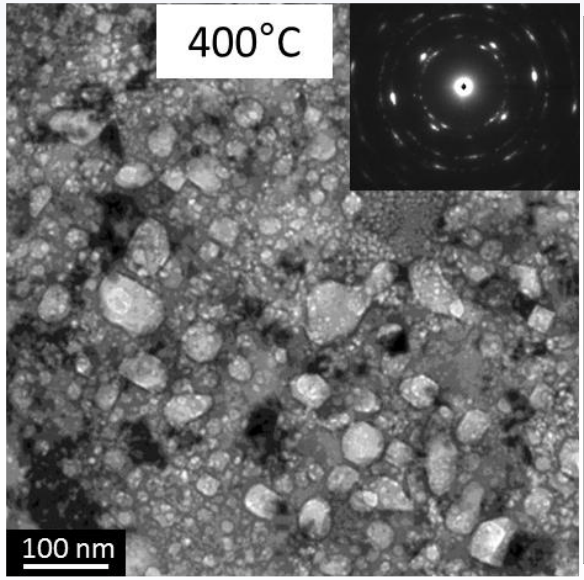
Visible damage to the sample

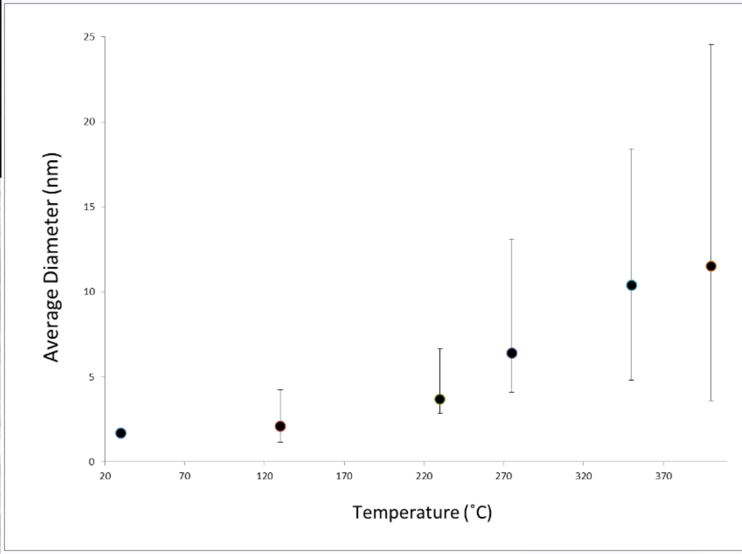
0.7 dpa Ni^{3+} irradiation

High concentration of cavities along grain boundaries

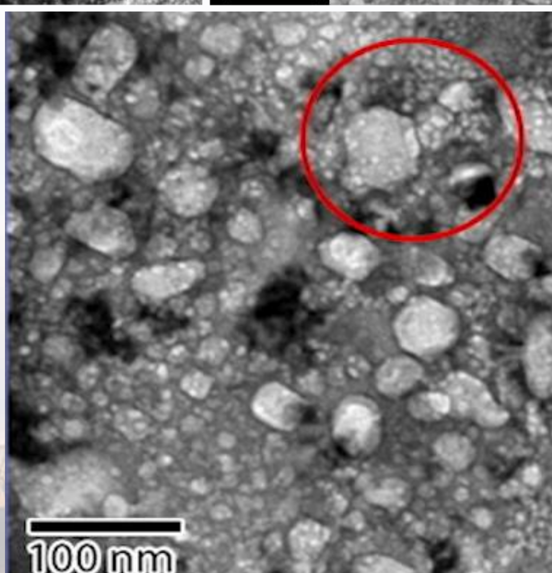
Cavity Growth during In-situ Annealing of 10 keV He^{+} Implanted and then 3 MeV Irradiated Ni^{3+}

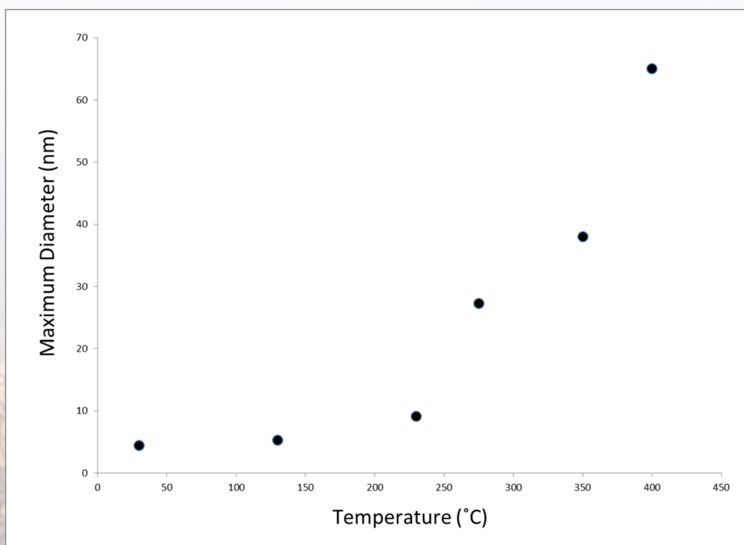






Bubble to cavity transition and cavity evolution can be directly studied

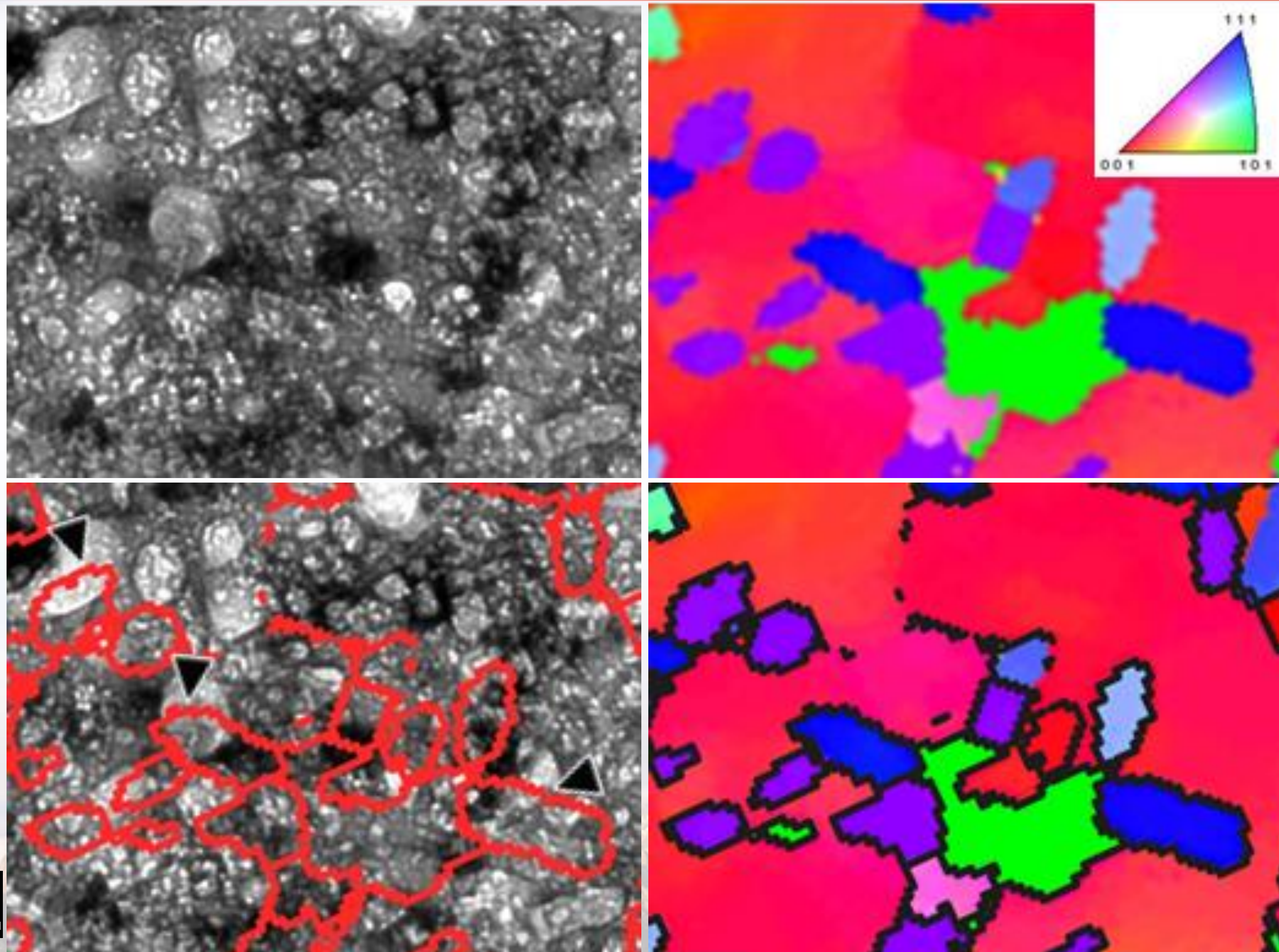




Precession Electron Diffraction Reveals Hidden Grain Structure

Cavities in helium implanted, self-ion irradiated,

nc nickel film annealed to 400 ˚C

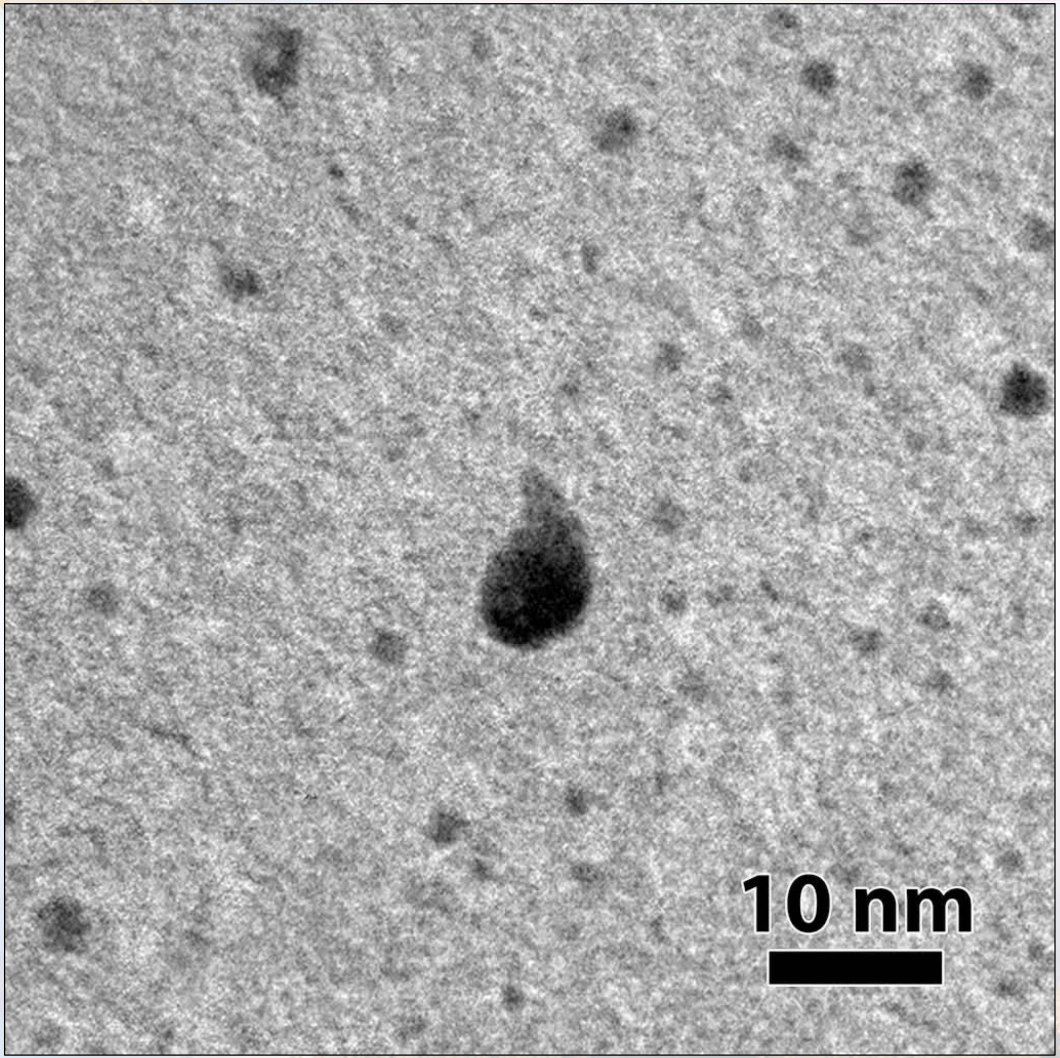


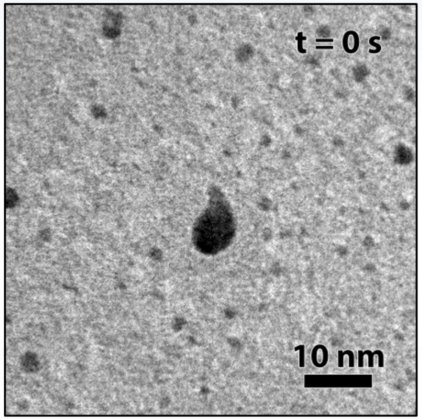
Cavities span multiple grains at identified grain boundaries

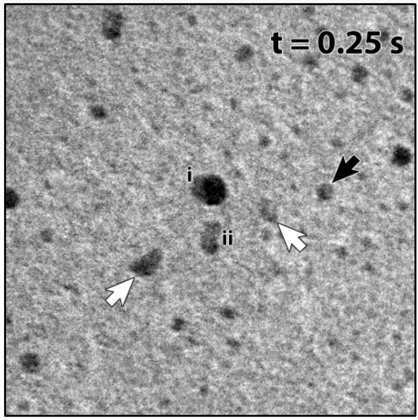
100 nm

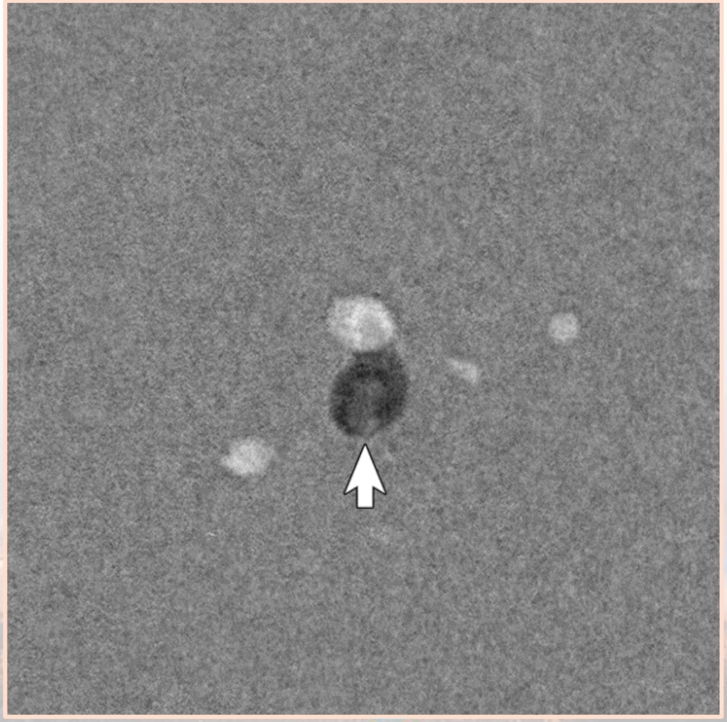
Single Ion Strikes:
46 keV Au^{1-} ions into 5 nm Au nanoparticles

Collaborator: D.C. Bufford



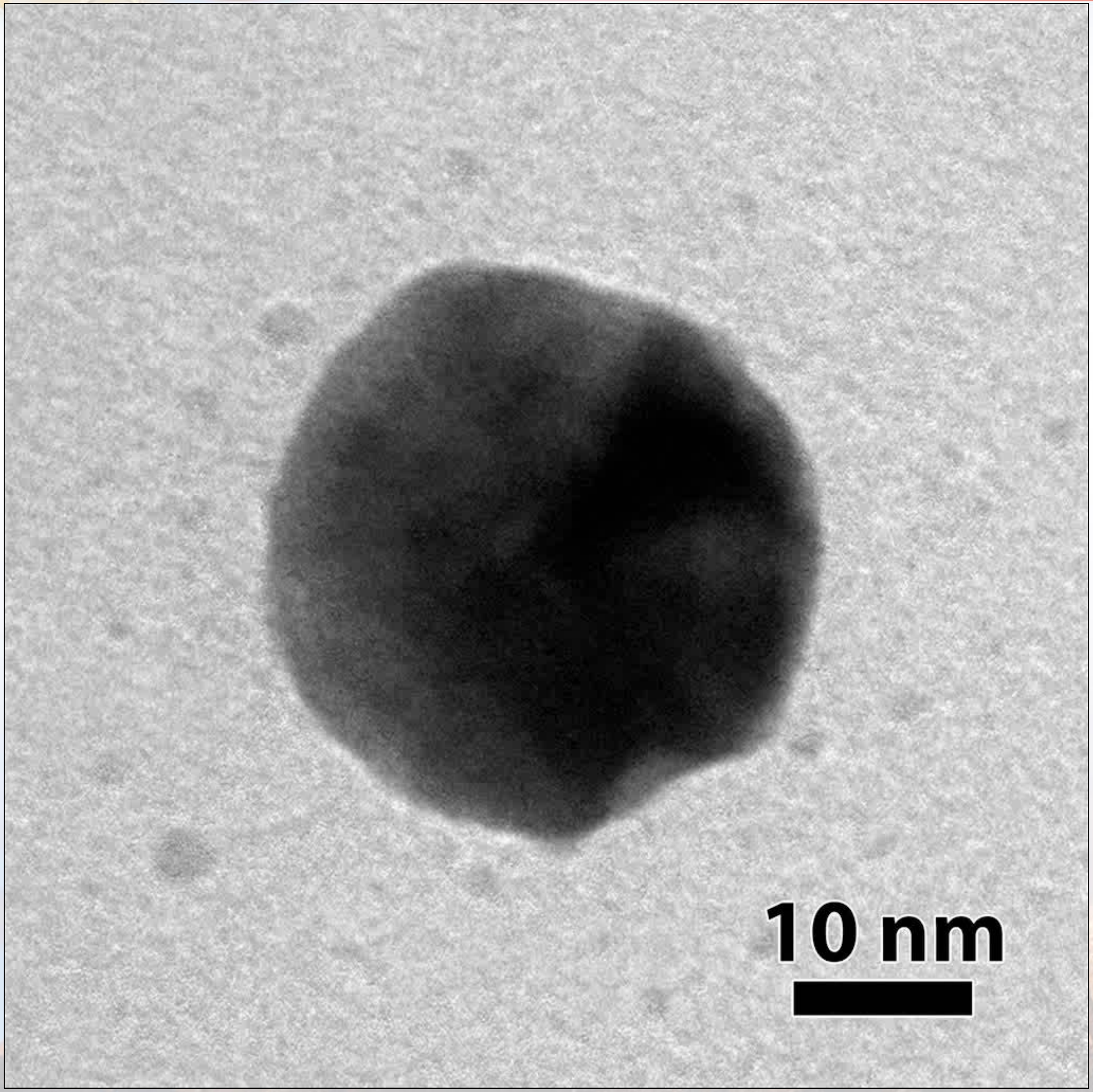


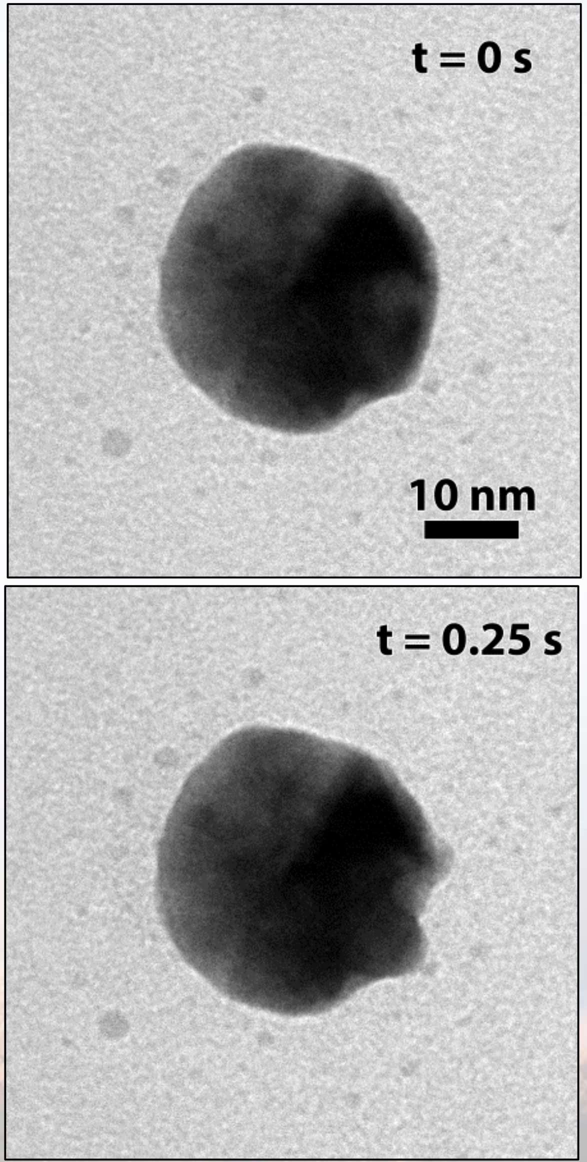




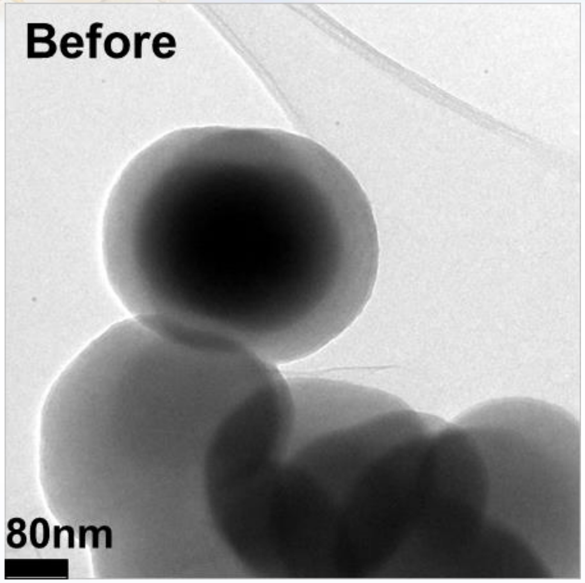
Single Ion Effects with 46 keV Au^{1-} ions: 20 nm

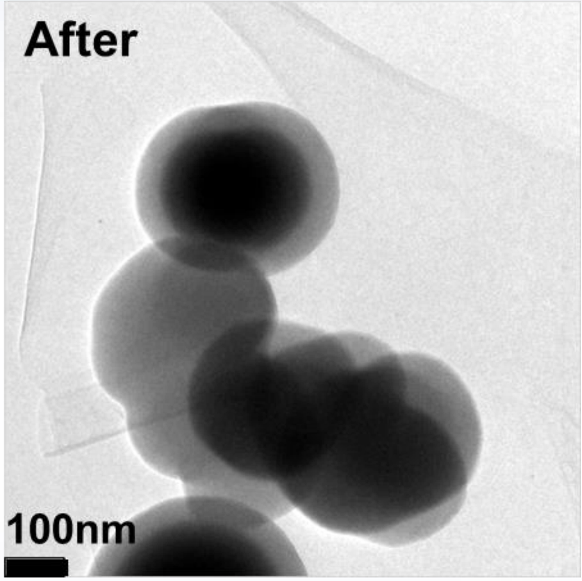
Collaborator: D.C. Bufford

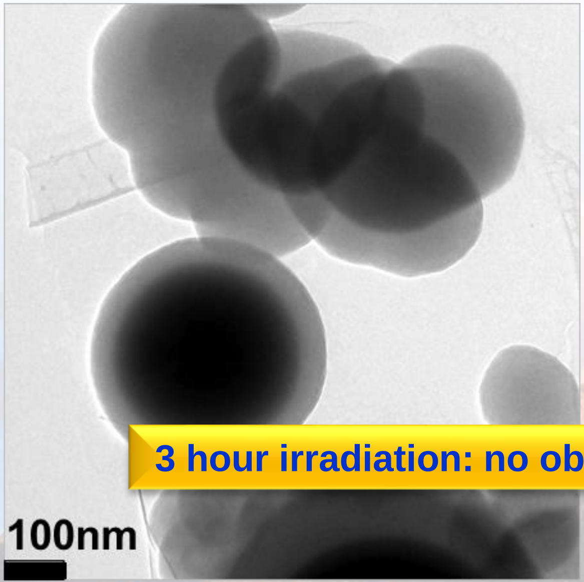


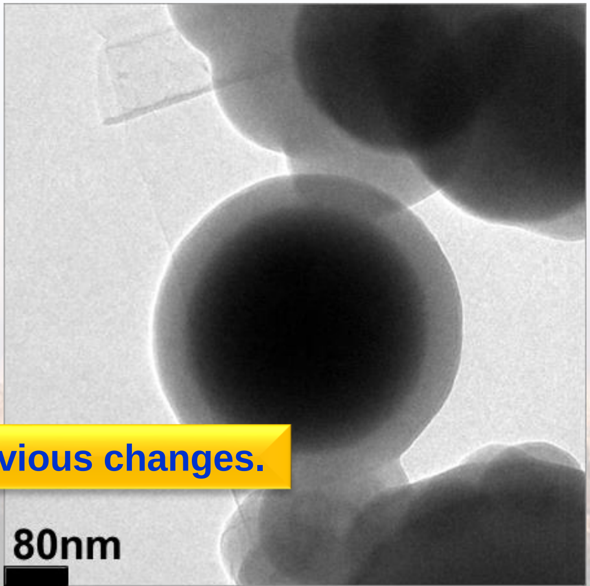


Irradiation of Amorphous Hf Oxide Nanoparticles with 10 nA of 3 MeV Cu Resulted in No Obvious Changes

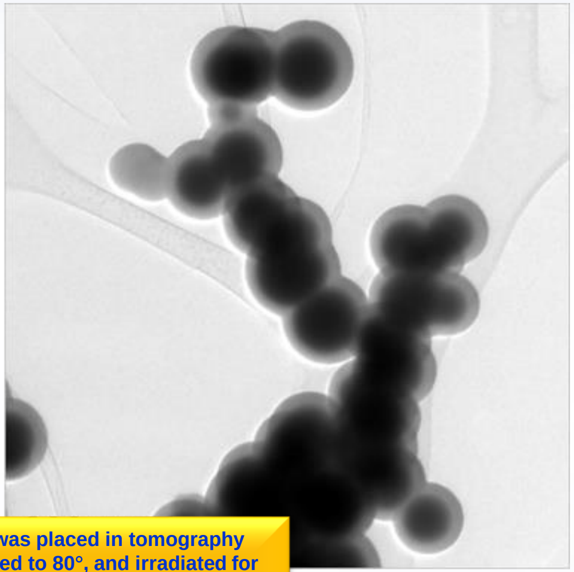




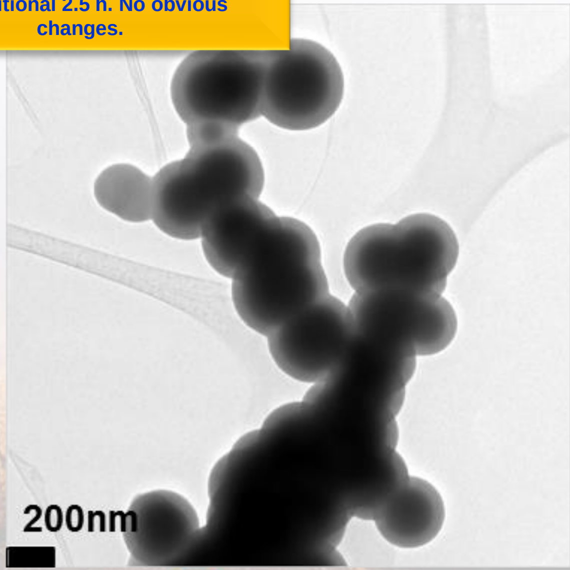




3 hour irradiation: no obvious changes.

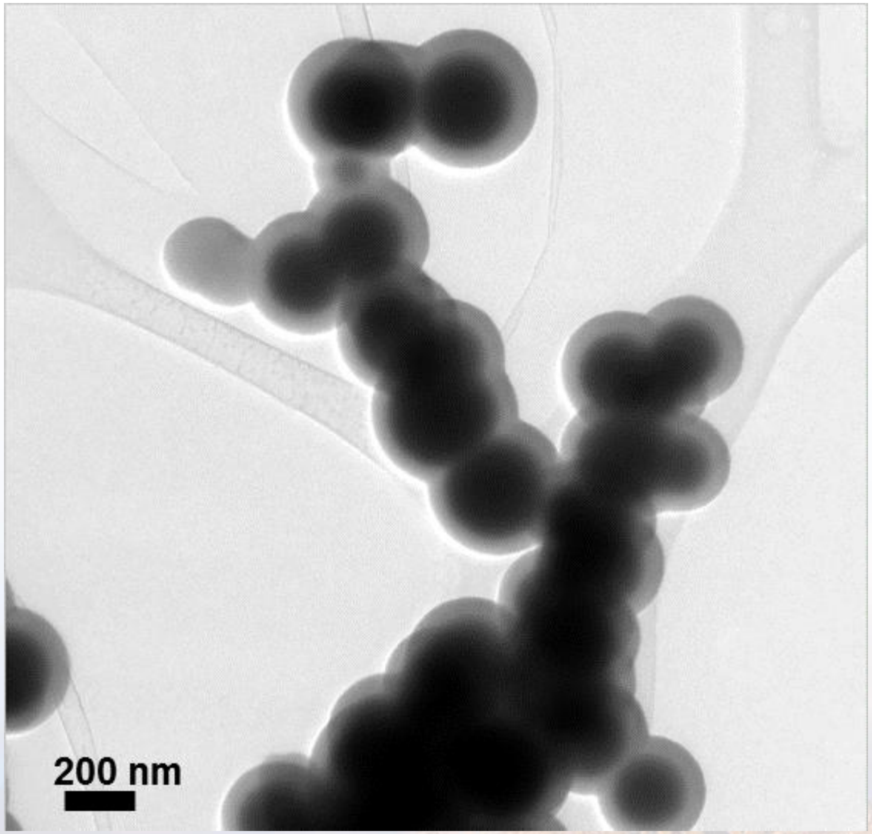


Sample was placed in tomography holder, tilted to 80°, and irradiated for an additional 2.5 h. No obvious changes.

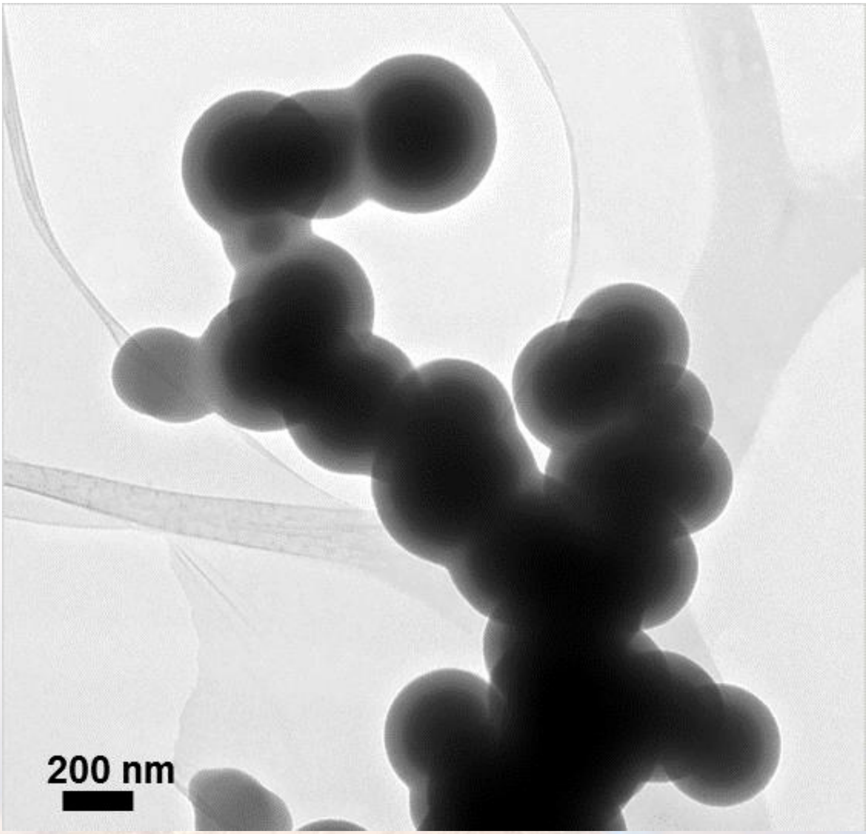


Additional Concurrent 3 MeV Cu/ 10 keV He Irradiation for 4.5 h Caused Swelling of the Hf Oxide Nanoparticles

Nanoparticles after 5.5 h of 3 MeV Cu Irradiation



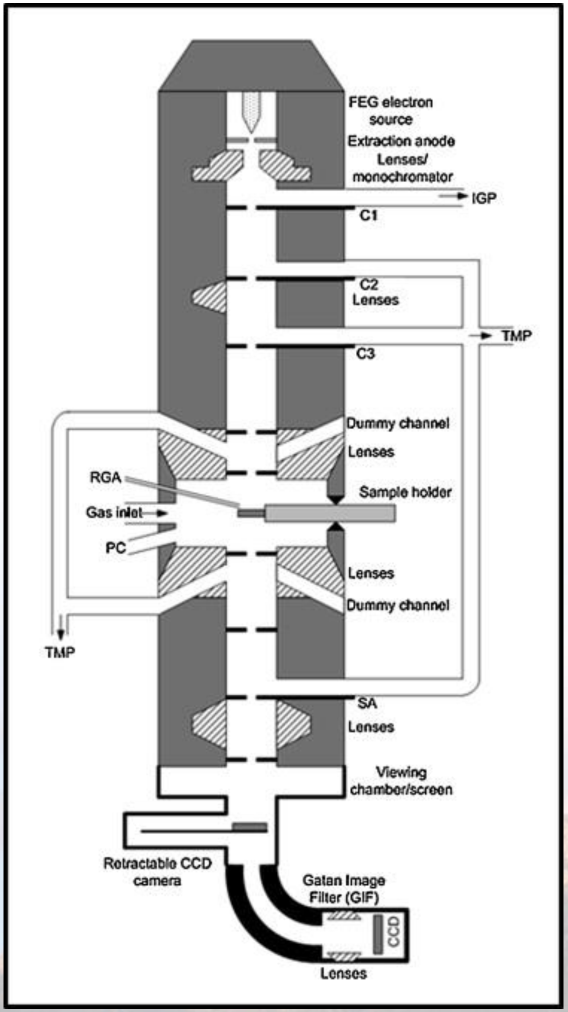
Nanoparticles after He + Cu Dual Beam Irradiation

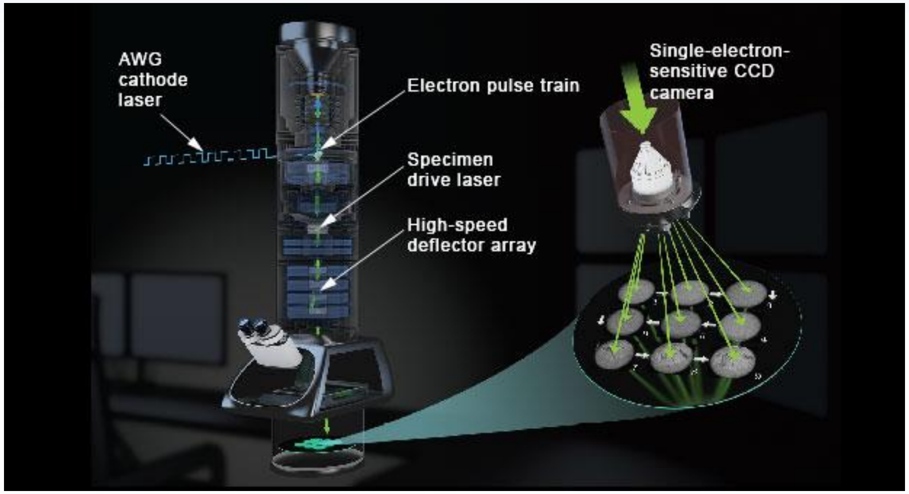


Future I^{3}TEM Modifications and Upgrades

Environmental TEM

Observe chemical reactions and environmental degradation in low vacuum





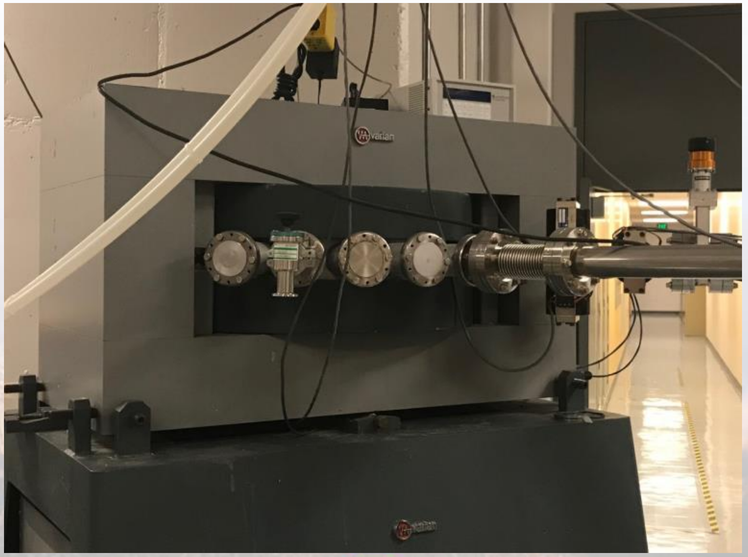
Dynamic TEM

Investigate rapid and irreversible material transformations on the ms to ns timescale

Bending Magnet Upgrade

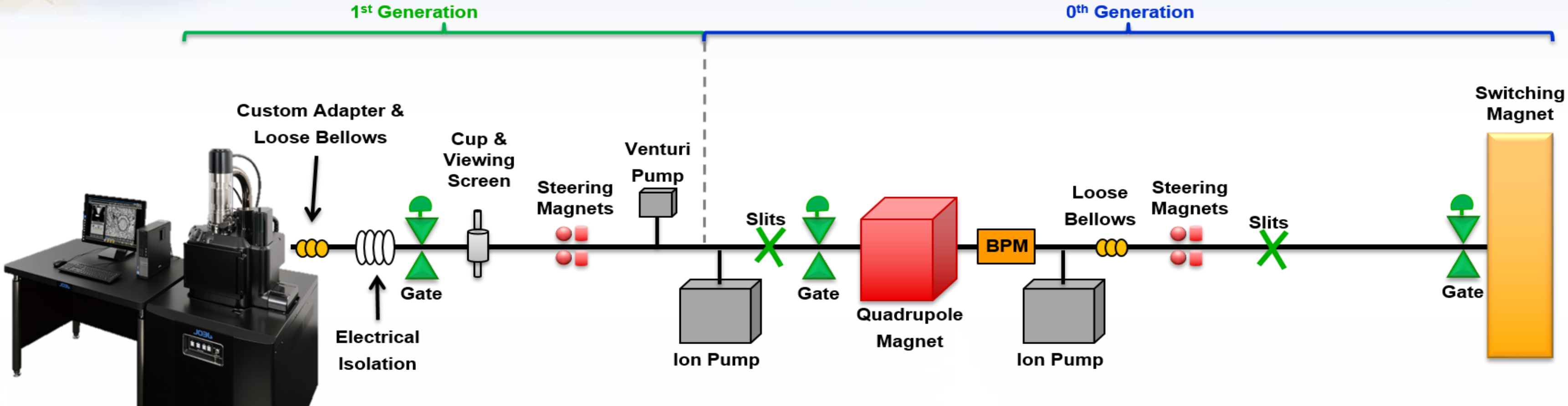
Will allow for very high energy (100+ MeV) ions to be directed into the I^{3}TEM column

We are continuing to developed advanced in-situ microscopy capabilities in order to complement our suite of in-situ irradiation facilities and sample holders

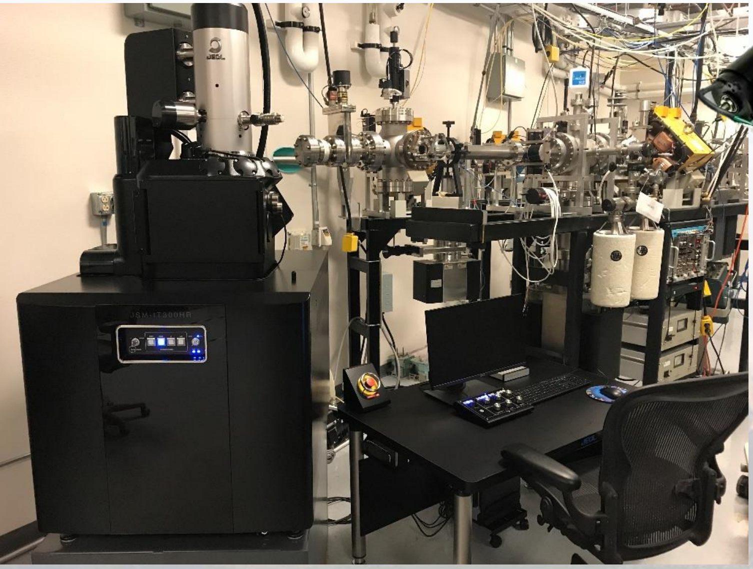


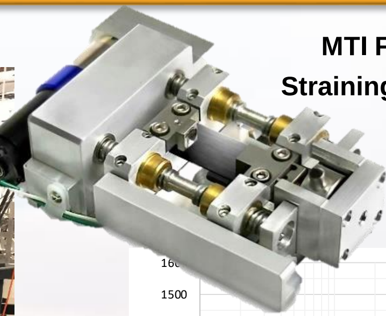
Schematic of the *In situ* SEM Beamline

Collaborators: D.L. Buller & S. Briggs



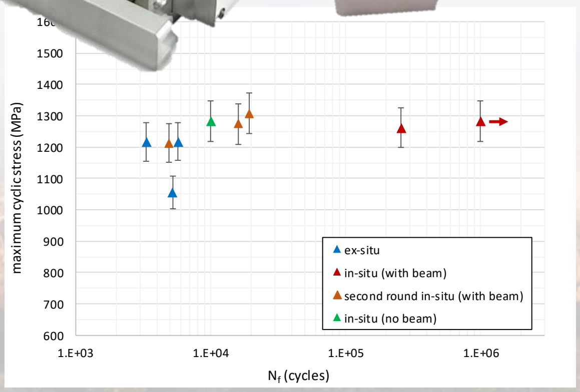
Beam Line planned for the *in situ* SEM will be developed in phases. Ultimate plan is for multiple accelerators being attached for dual or triple beam experiments.



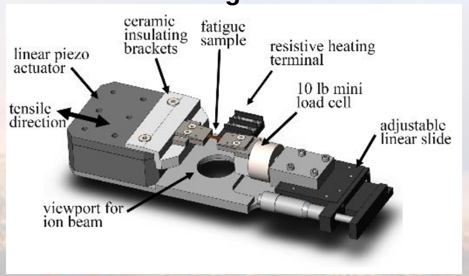


MTI Fullam Straining Heating

Hysitron PI85 Nanoindenter



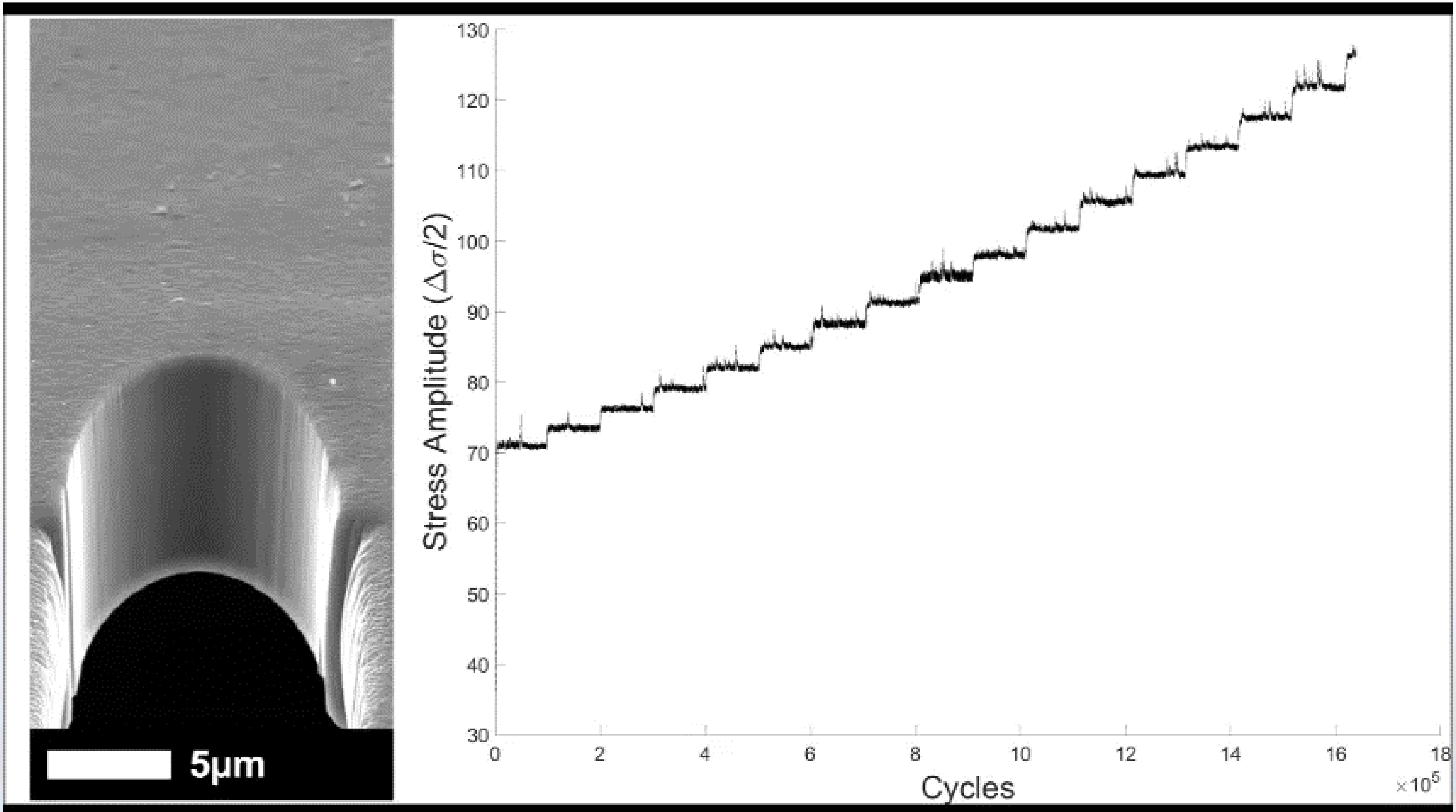
Custom-built Piezo Fatigue tester



Crack Propagation during Fatigue Testing of Nanocrystalline Ni-40Fe

Collaborators: N. Heckman & B. Boyce

10-60 nm grains, 10 µm notch milled in specimen. 30 Hz load frequency, 4000 cycles/image



SEM is operational and in-situ mechanical testing is in progress! Mating to accelerator beam line will allow for advanced studies of radiation creep and other radiation-induced phenomena.

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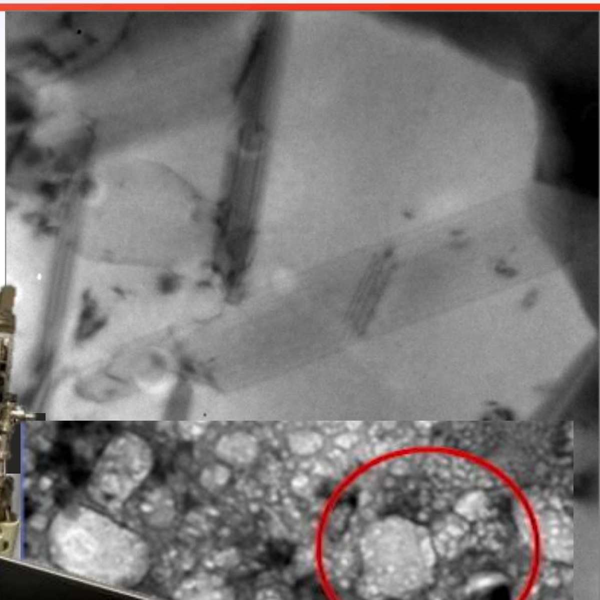
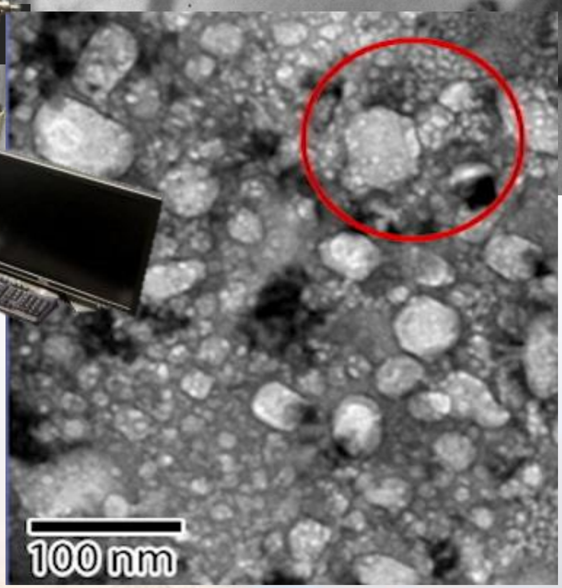
Summary

- **Sandia's I³TEM is one of only two facilities in the US**
  - **Only facility in the world with a wealth of dual *in situ ion irradiation* capabilities**
  - ***In situ* high energy ion irradiation from H to Au**
  - ***In situ* gas implantation**
  - **11 TEM stages with various capabilities**
- **Currently applying the current I³TEM capabilities to various material systems in combined environmental conditions and expand the capabilities**





Sandia's I³TEM although still under development is providing a wealth of interesting initial observations

<u>Collaborators:</u>

- **IBL: C.M. Barr, S.A. Briggs, D.C. Bufford, D. Buller, C. Chisholm, B.G. Clark, M.T. Marshall, B. Muntifering, S.H. Pratt, & P. Price**
- **Sandia: M. Abere, B. Boyce, T.J. Boyle, R. Dingreville, R.F. Hess, A.C. Kilgo, B.E. Klamm, W.M. Mook, J.D. Puskar, J.A. Scott, & J.A. Sharon**
- **External: A. Aitkaliyeva, H. Bei, P.J. Ferreira, K.J. Ganesh, E.P. George, D. Gross, P. Hosemann, J. Kacher, S. Maloy, A. Minor, J. Qu, S. Rajesekhara, I.M. Robertson, D. Stauffer, & Hysitron Inc.**

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Thank you for your attention.

Access to the I^3TEM and associated facilities is now available through both the Nuclear Science User Facilities (NSUF) and the Center for Integrated Nanotechnologies (CINT).

Nuclear Science User Facilities

https://nsuf.inl.gov

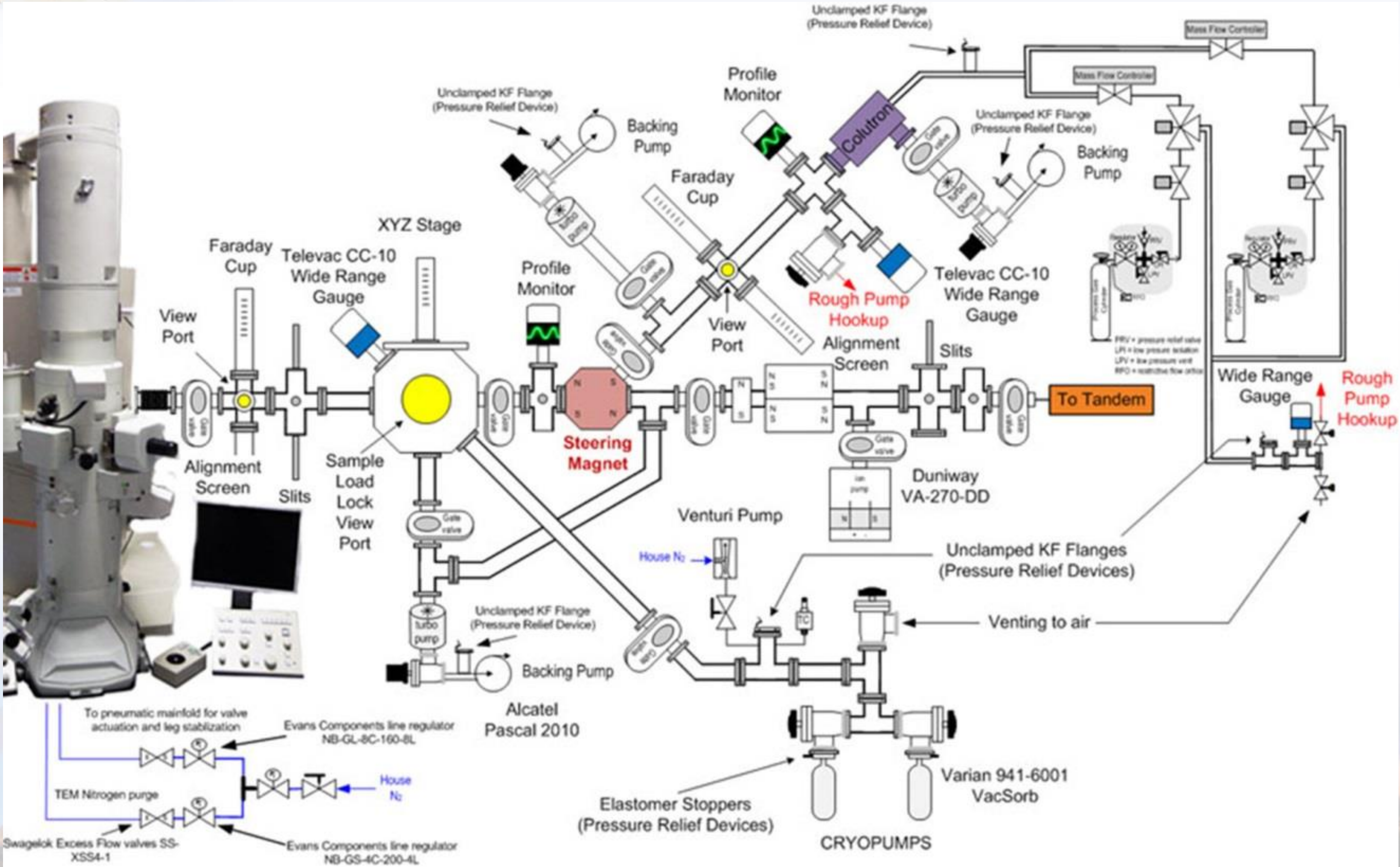
Center for Integrated Nanotechnologies

http://cint.lanl.gov

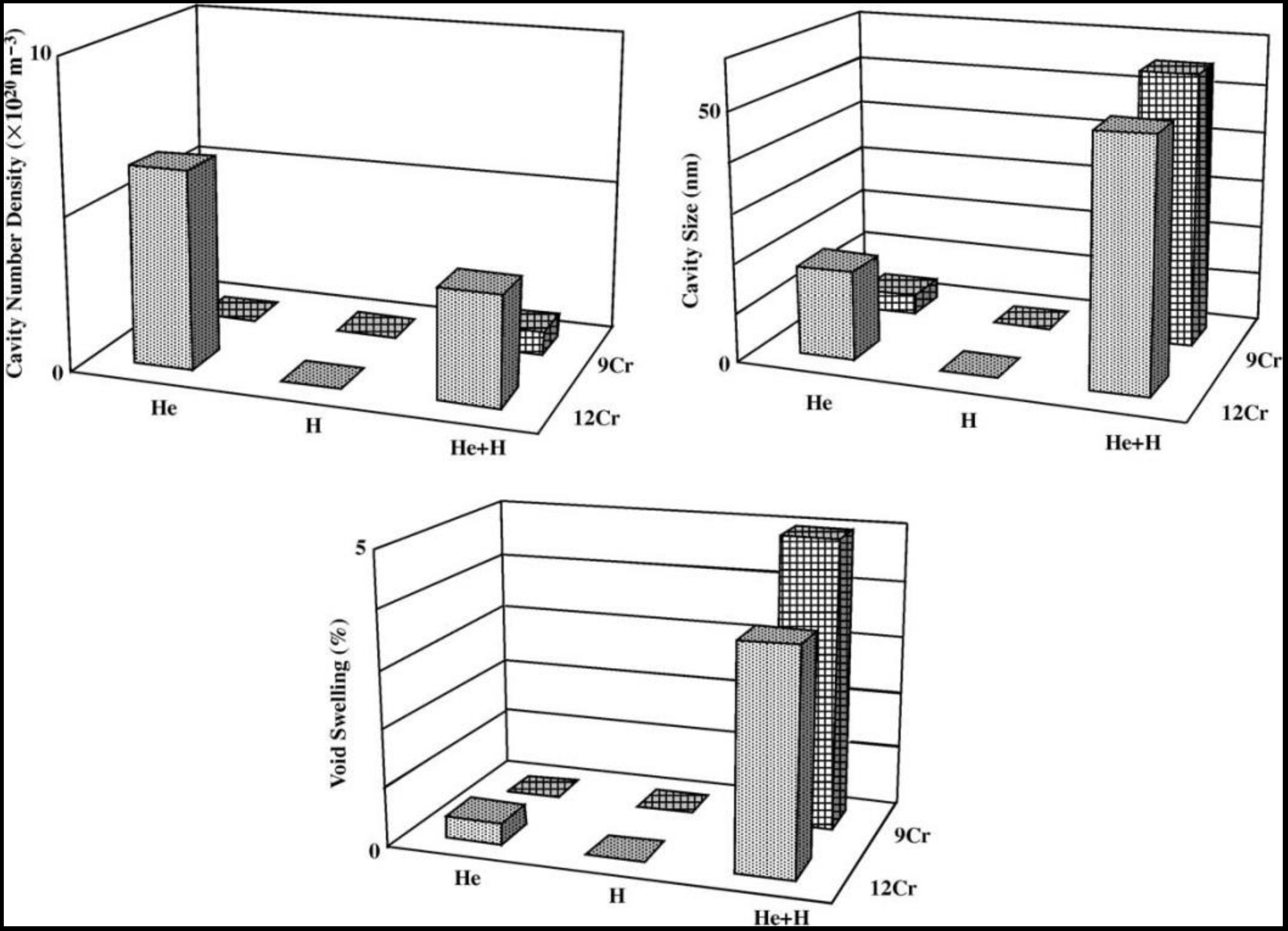
Extra Slides

Schematic of the *In situ* TEM Beamline

Collaborators: M.T. Marshall J.A. Scott, & D.L. Buller



H, He, and Displacement Damage Synergy



T. Tanaka et al. "Synergistic effect of helium and hydrogen for defect 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**

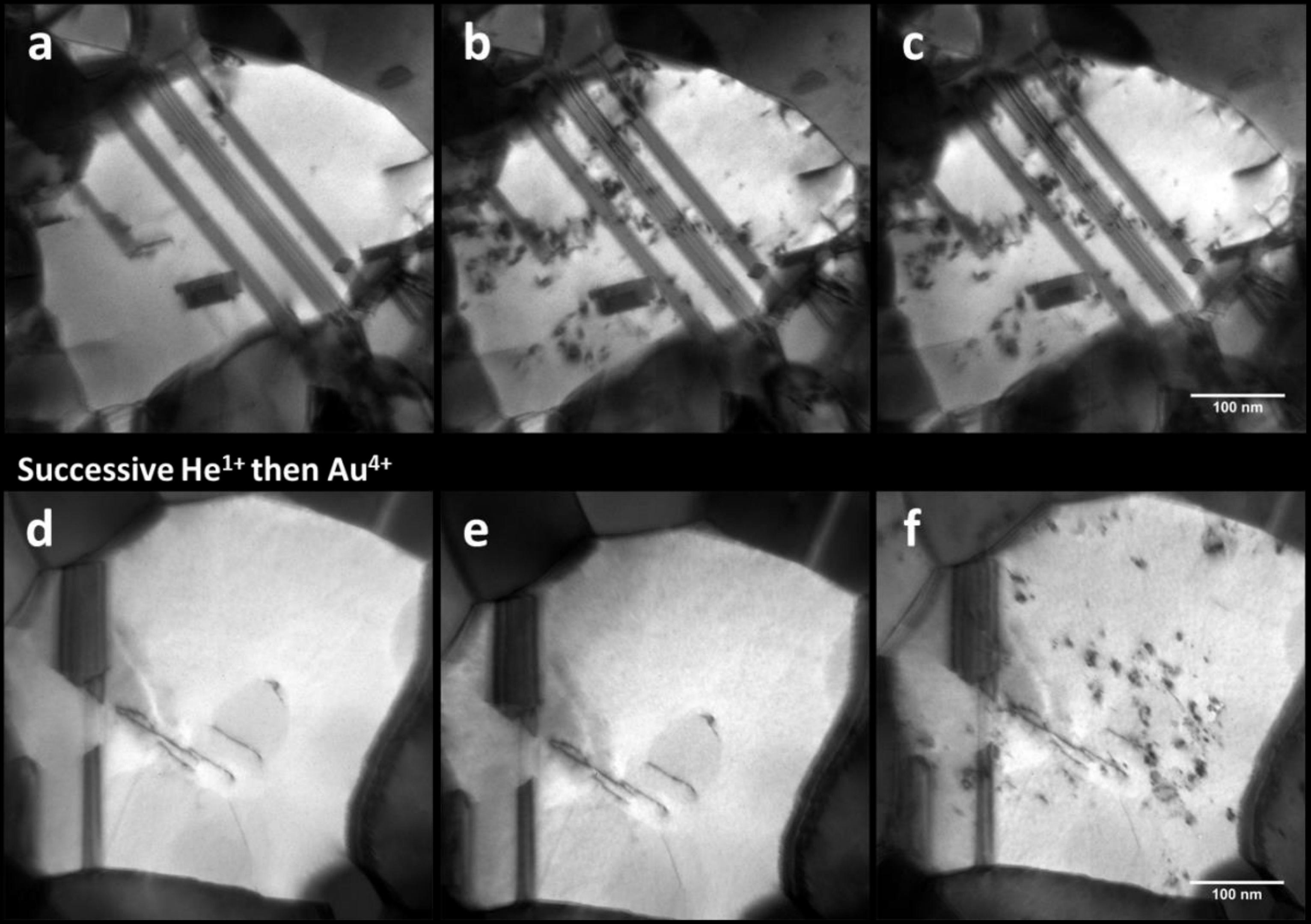
Difficulty of performing triple-beam irradiation has resulted in a limited number of facilities world wide

In situ Successive Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor

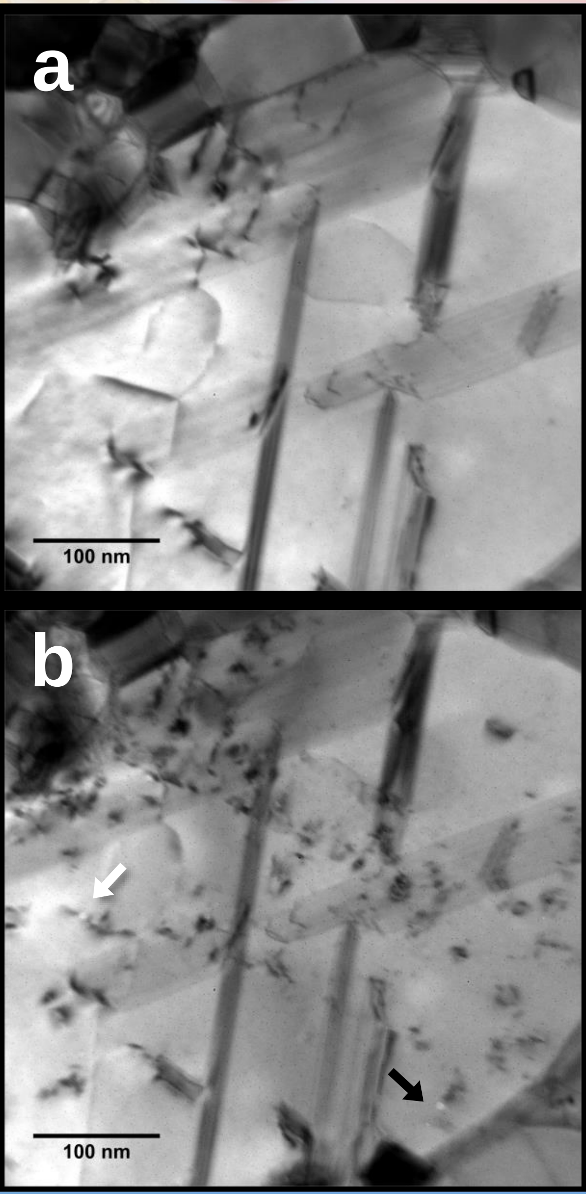
Successive Au^{4+} then He^{1+}

Successive He^{1+} then Au^{4+}

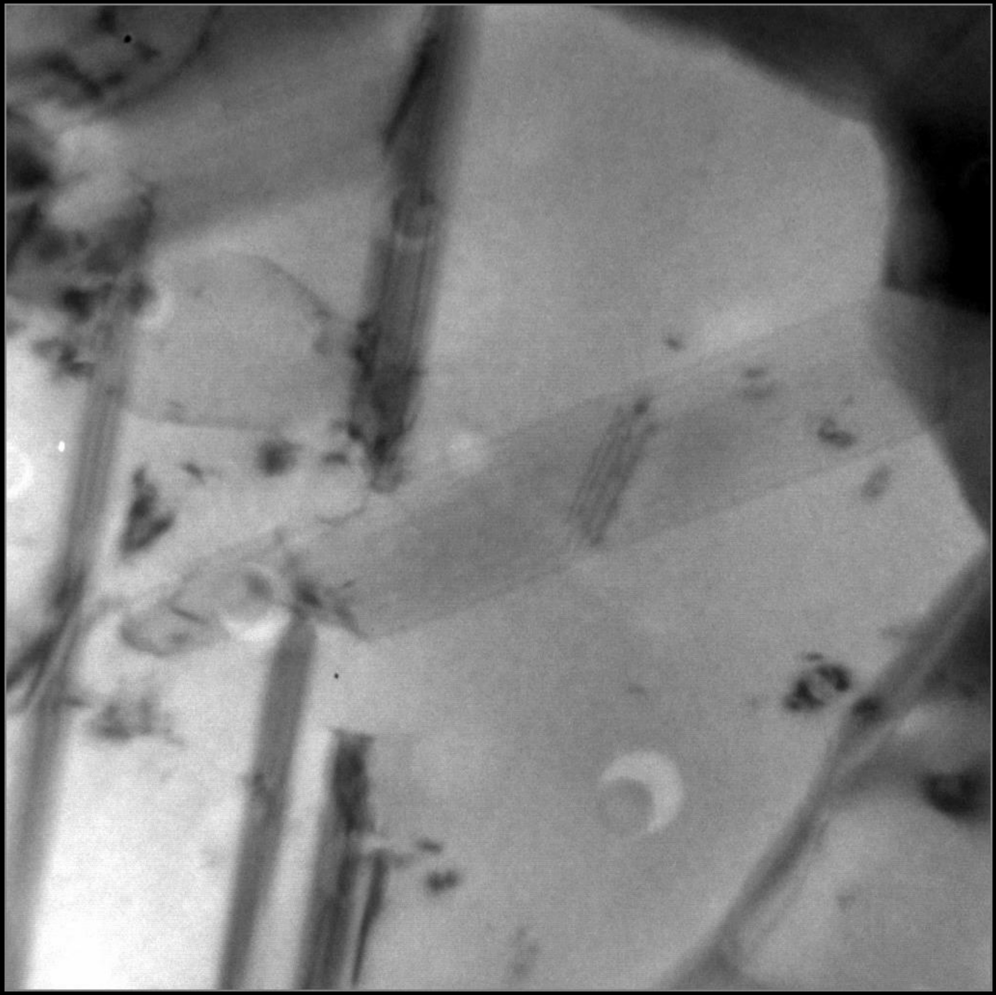


In situ Concurrent Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor

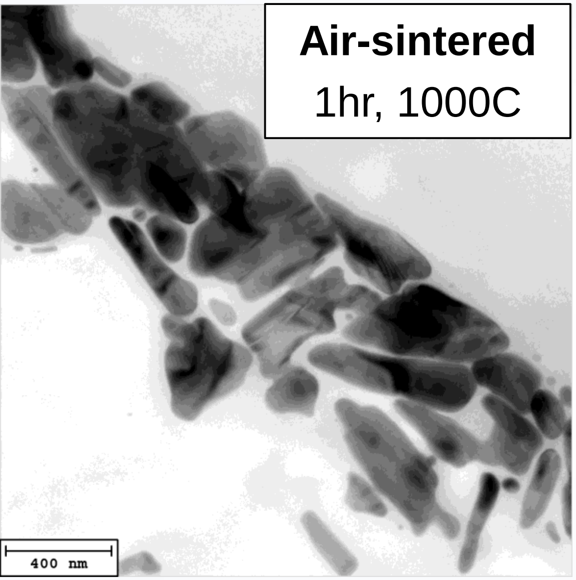


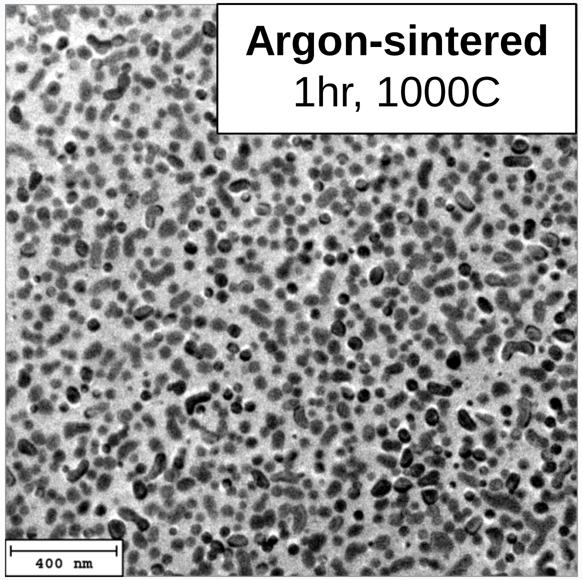
He^{1+} implantation and Au^{4+} irradiation of a gold thin film

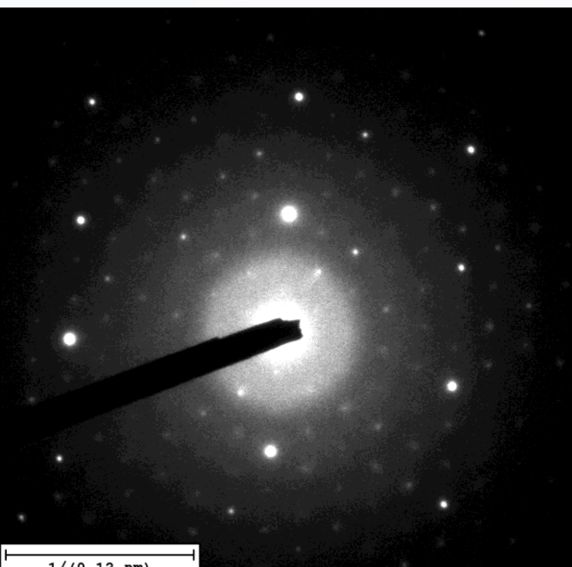


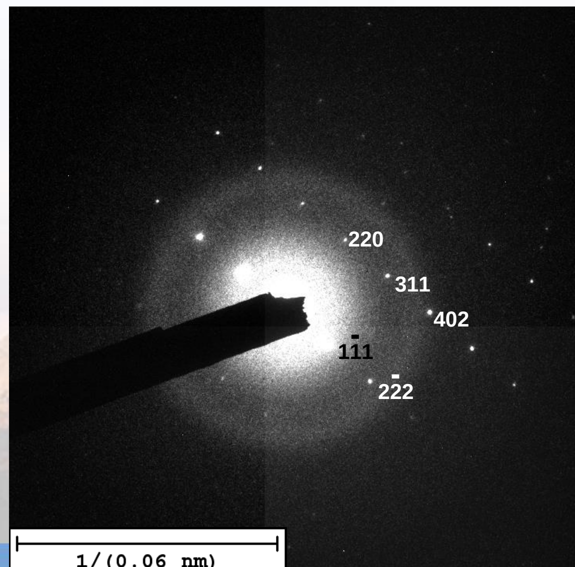
Uranium Oxide Formation in Different Sintering Environments

Collaborators: S.A. Briggs, R. Hess, and B. Klamm









Nanoparticles prepared from solution of $UO_2(NO_3)_2$, PEI, and EDTA

Ar-sintered specimen phase diffraction patterns map to fluorite/FCC structure characteristic of UO_2

- Larger lattice parameter suggests hypostoichiometric uranium-dioxide phase (UO_{2-x})

Air-sintered specimens do not map to fluorite/FCC

- Likely a hyperstoichiometric uranium-oxide phase (U_3O_8, U_4O_9)

Characterization of ion-irradiation response is work-in-progress

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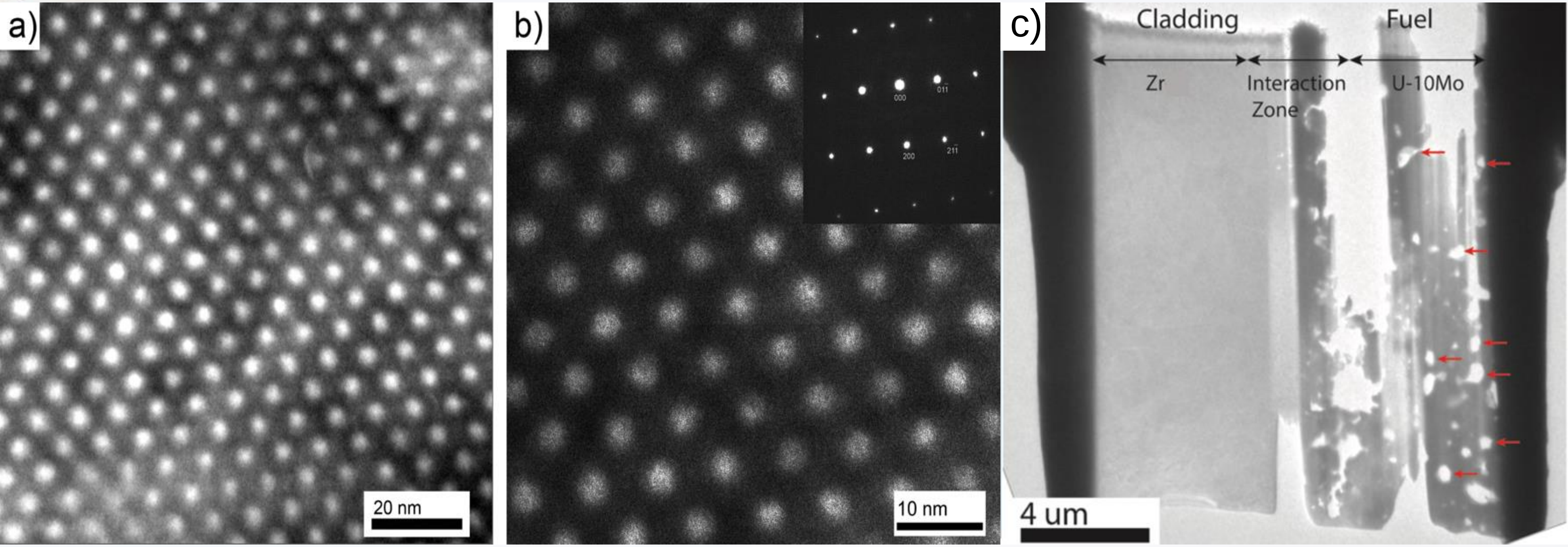
U-10Mo Depleted Samples from INL

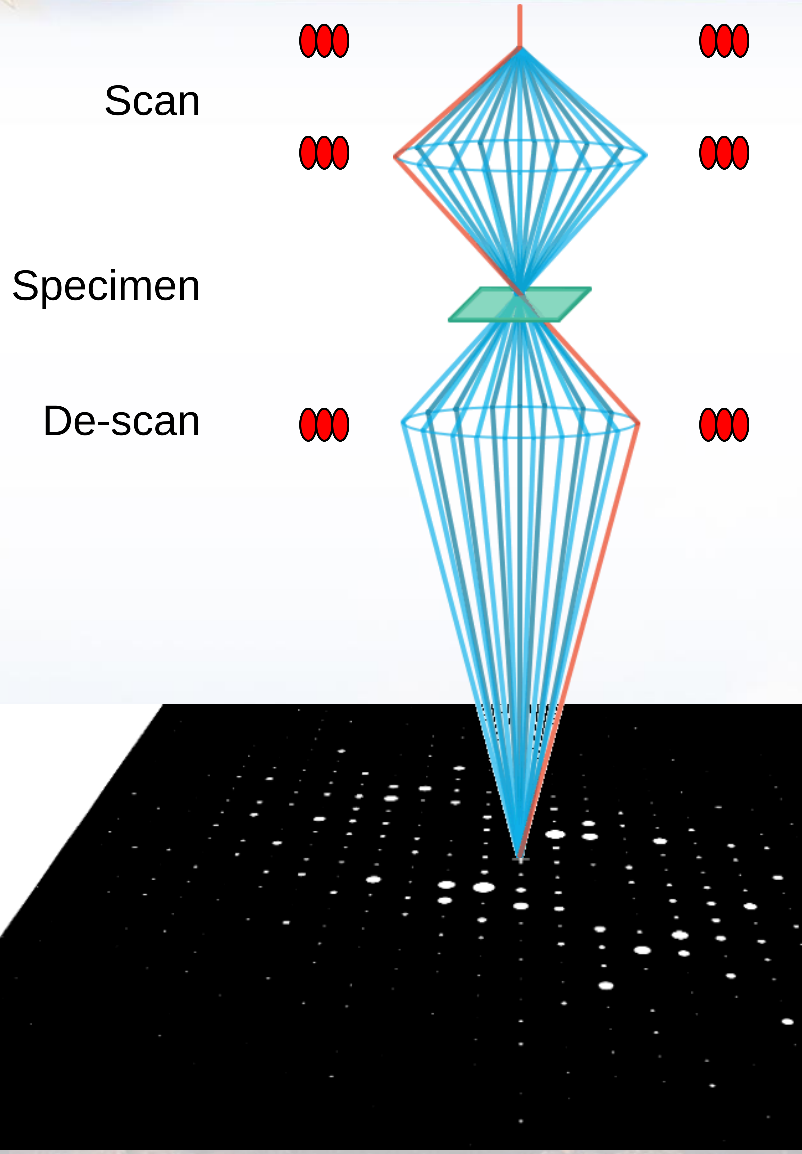
Collaborators: C.M. Barr, A. Aitkaliyeva, R. Dingreville

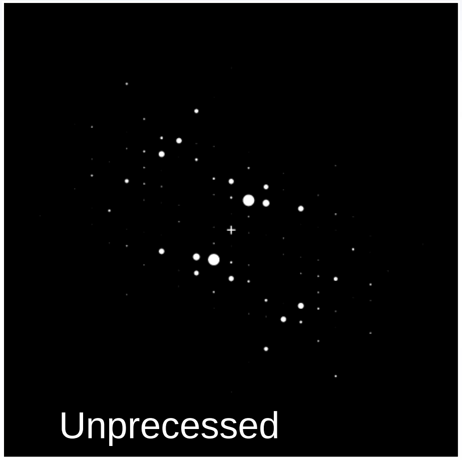


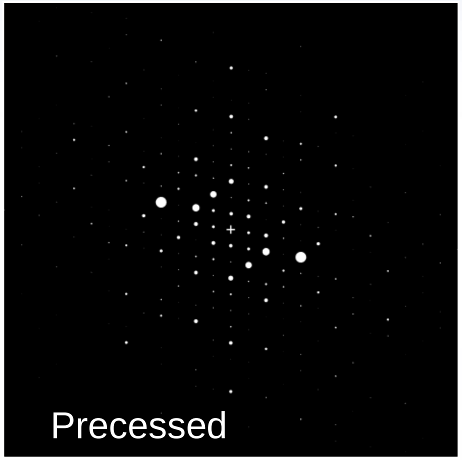
- Local average fission density: 4.42 x 10^{21} (fiss/cm^3)
- Samples from AFIP-6MKII Reduced Enrichment for Research and Test Reactors (RERTR) experiments at Idaho National Laboratory (INL)
- The average gas superlattice bubble diameter distribution: 3.5 ± 0.25 nm diameter

Precession Electron Diffraction (PED) Microscopy

Collaborators: K.J. Ganesh, S. Rajasekhara, & P.J. Ferreira







<u>Advantages</u>

- **< 10 nm spatial resolution**
- **Near kinematical electron diffraction**
- **Symmetry ambiguities are resolved**
- **Fast and automated acquisition**
  - **~200 grains in 15 min.**

Chris Own, PhD Dissertation, Northwestern University, 2004

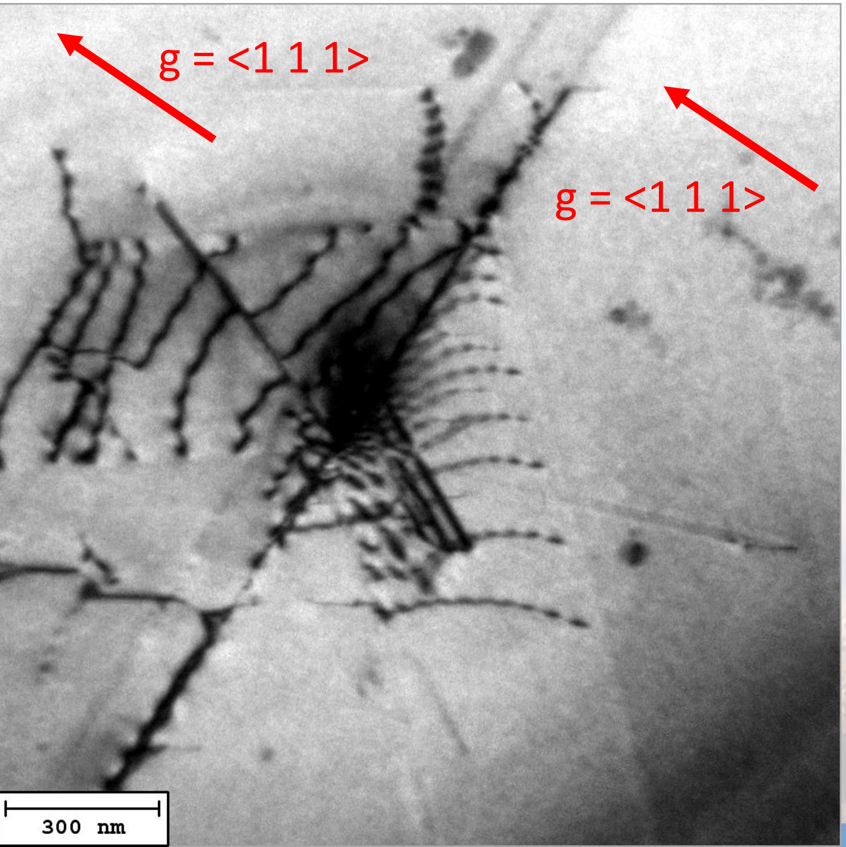
Slide courtesy: K. J. Ganesh, K. Hattar

In-situ TEM straining observation of dislocations – coherent twin boundary in 304SS

Contributors: C.M. Barr

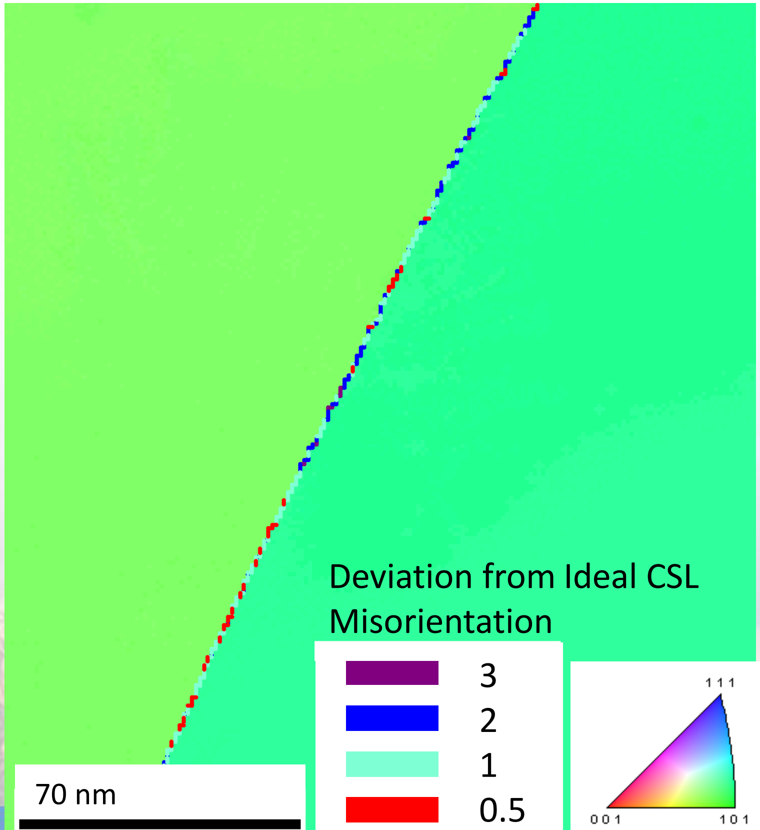
- Precession enhanced TEM orientation map (left) used to examine change in the twin boundary misorientation after straining observed in kinematic BF-TEM (right) during in-situ TEM experiment.
- Effort is to link in-situ dislocation-GB interactions during straining with changes in local grain boundary structure

BF-TEM Image:



Same area PED map after straining

Precession enhanced TEM orientation map:

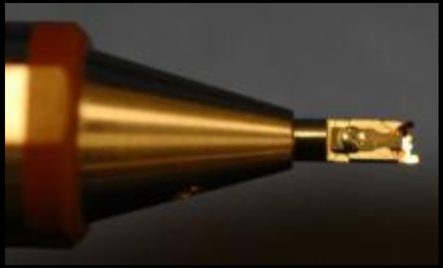


Electron Tomography Provides 3D Insight

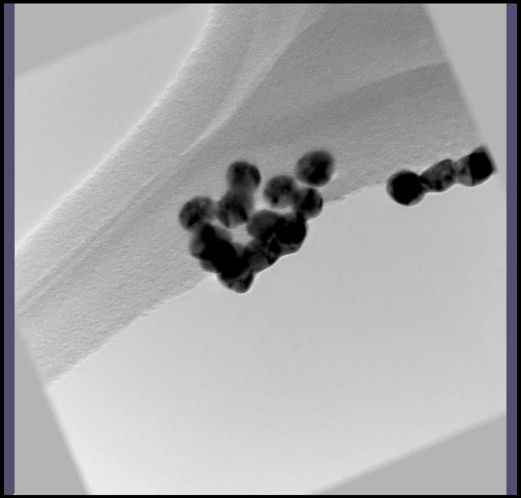
Collaborators: S.H. Pratt & T.J. Boyle

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

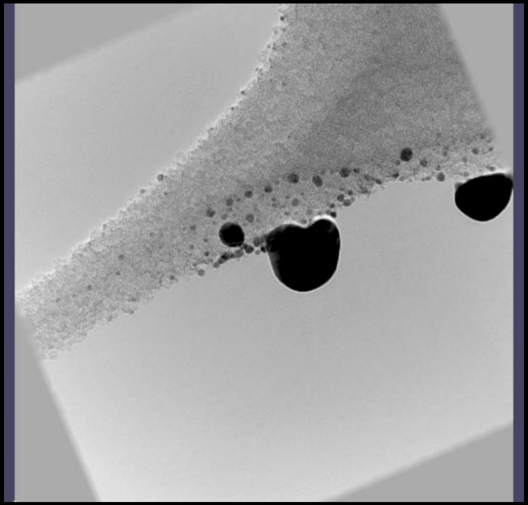
Hummingbird tomography stage



Aligned Au NP tilt series - unirradiated

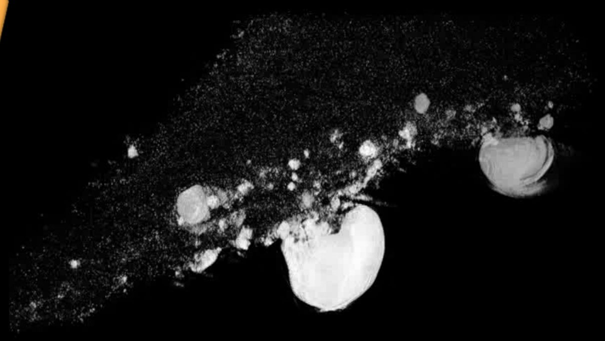


Aligned Au NP tilt series - irradiated



Unirradiated Au NP model

Irradiated Au NP model



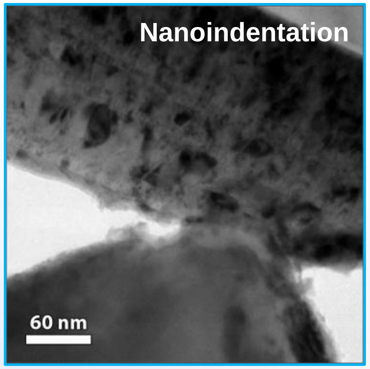
The application of advanced microscopy techniques to extreme environments provides exciting new research directions

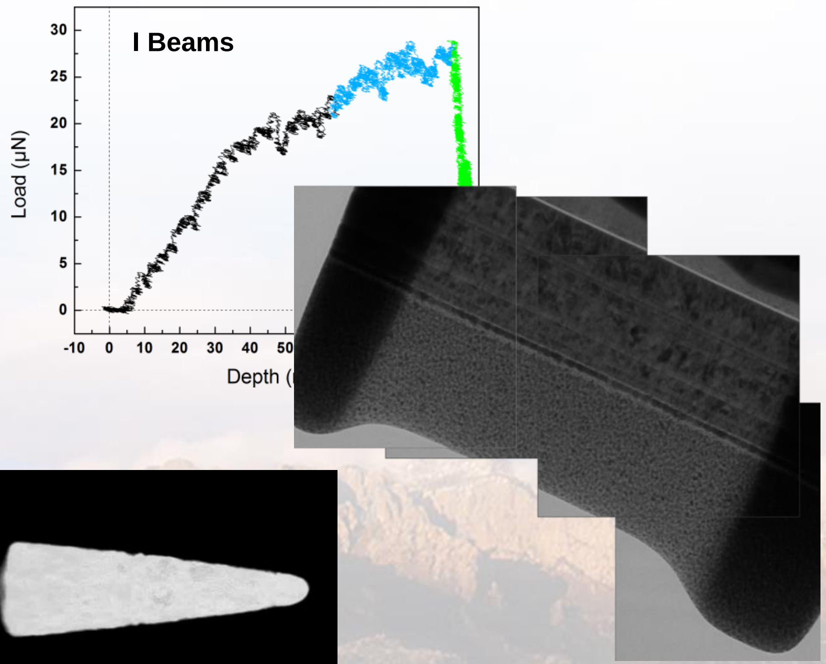
In situ Quantitative Mechanical Testing

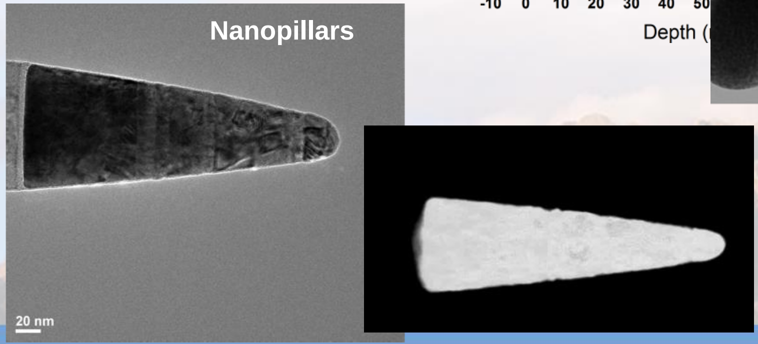
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

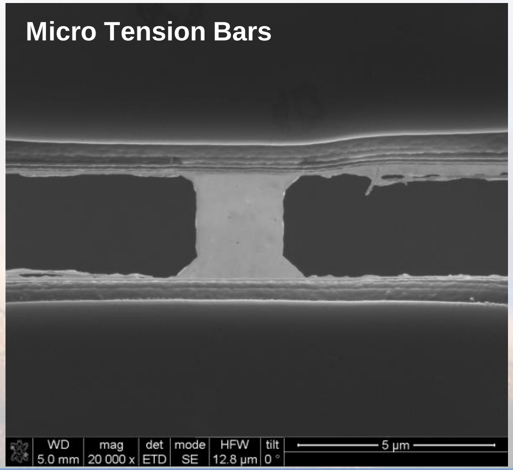
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

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



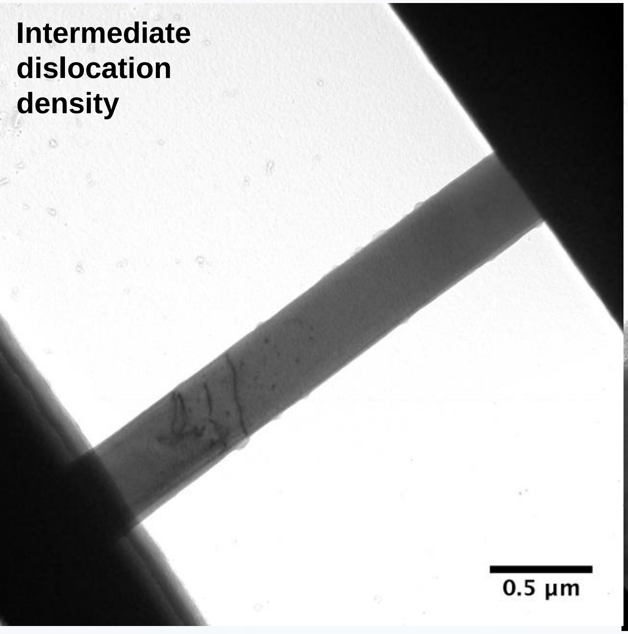


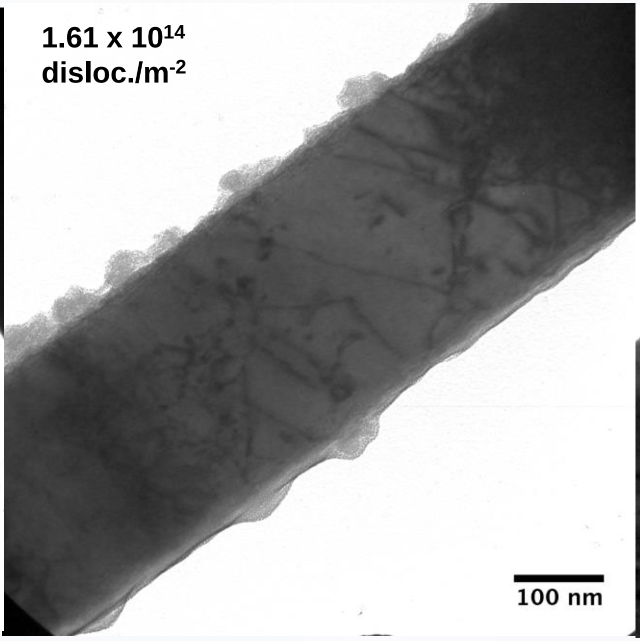


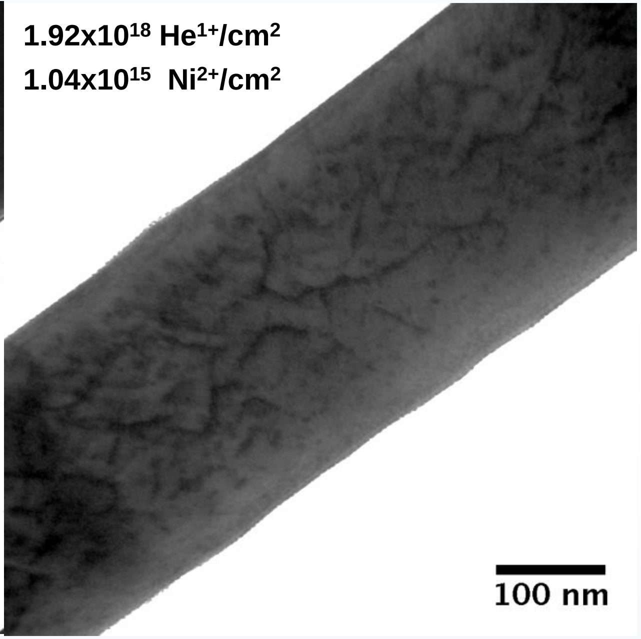


Next Steps: *In situ* TEM Quantitative Mechanical Testing

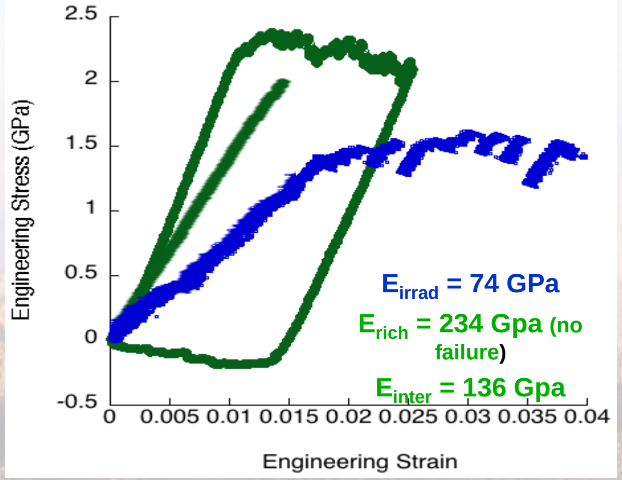
Contributors: C. Chisholm, H. Bei, E.P. George, P. Hosemann, & A.M. Minor







Work has started by looking sequentially at the quantitative effects of ion irradiation on mechanical properties utilizing in-situ ion irradiation TEM and in-situ TEM straining.



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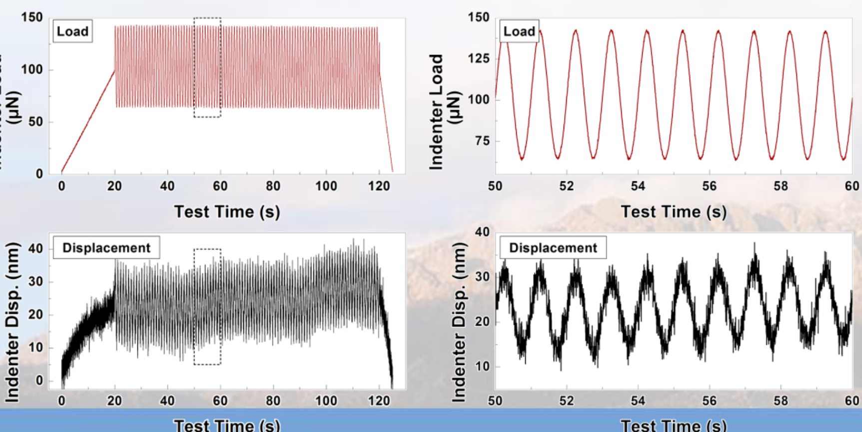
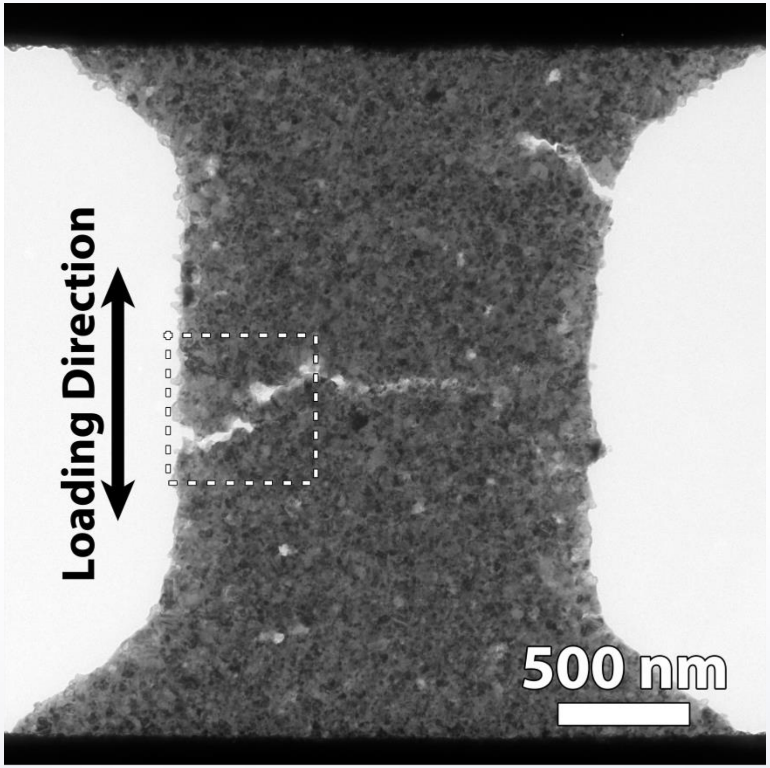
In situ TEM Quantitative Fatigue Testing

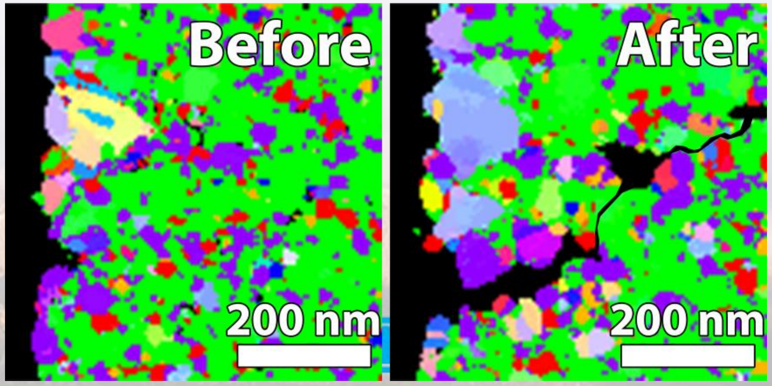
Contributors: D.C. Bufford, D. Stauffer, W. Mook

3× playback speed



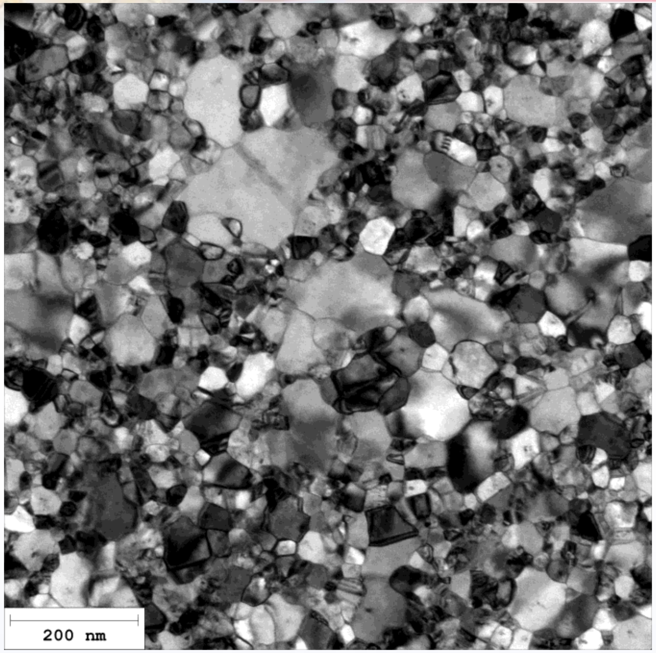
High cycle fatigue in real time with nanometer resolution

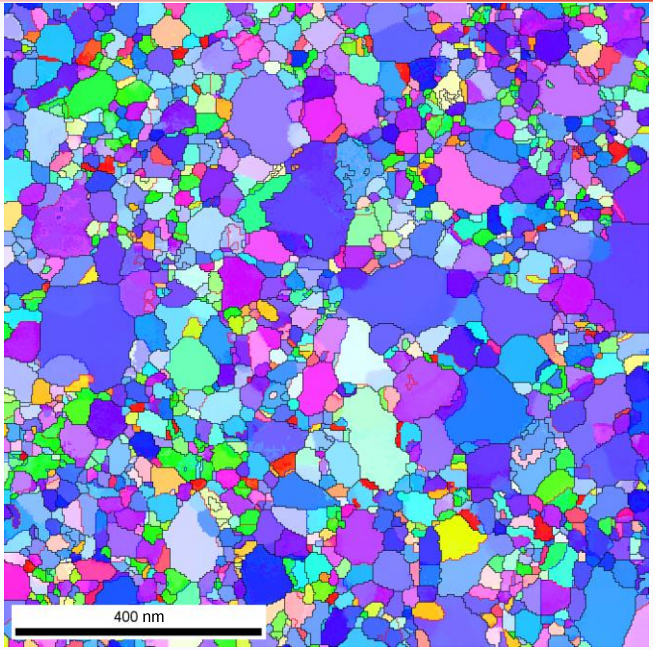


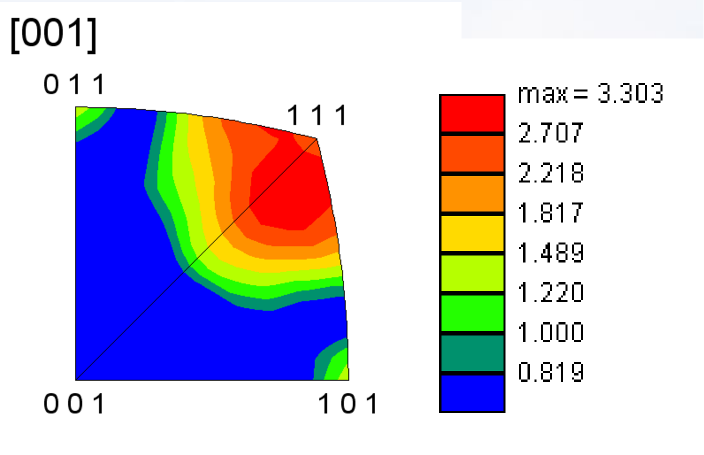


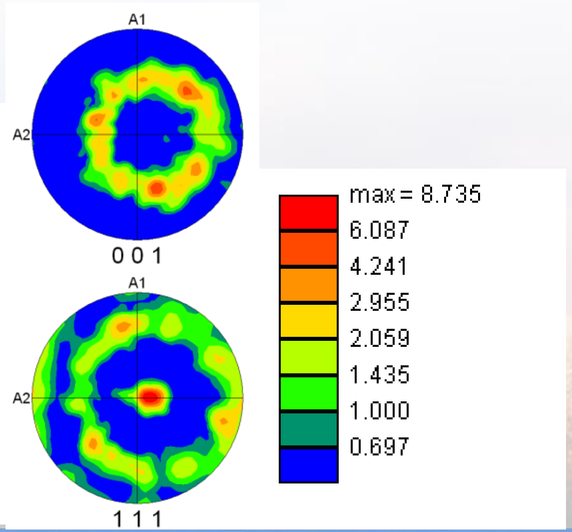
FCC Pt after laser heating

Contributors: C.M. Barr, P. Price, D. Adams, M. Abere









- PED Coupled with BF-TEM after annealing
- Pt after in-situ TEM laser heating
- Orientation texture indicates a strong, deviated {111} fiber texture