Probing Materials Response to Extreme Environments

SAND2015-10087C

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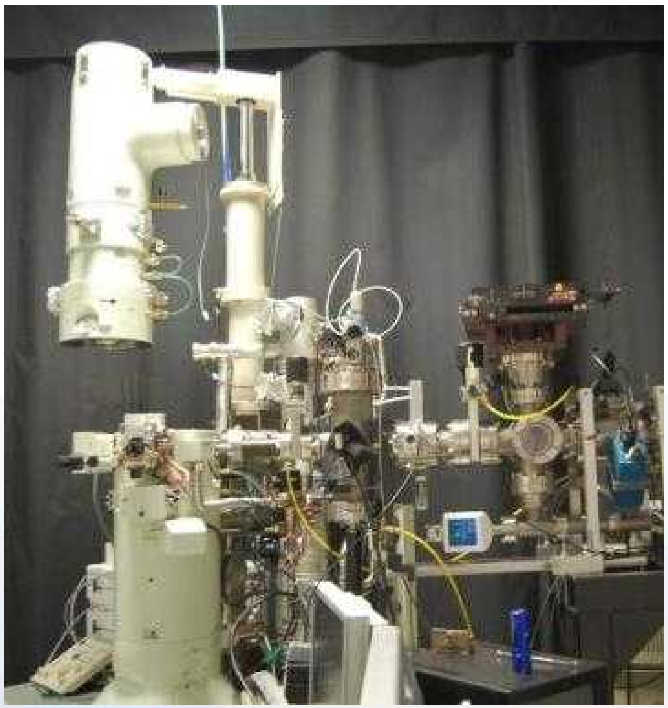
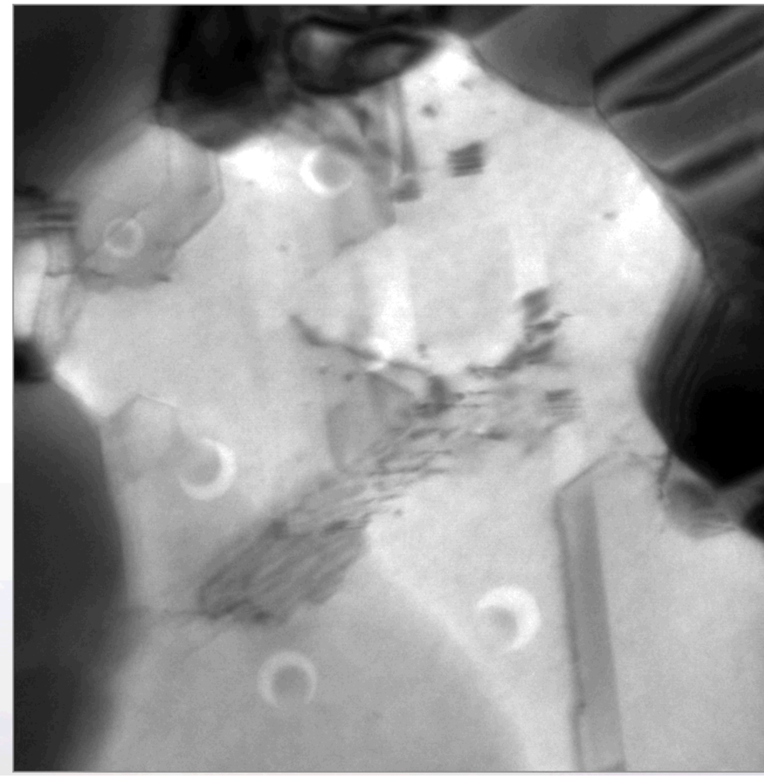
Ion Beam Lab at Sandia National Laboratories

Nov. 10, 2015



In situ TEM microscopy

Has recently undergone significant growth providing 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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Sandia's Extreme Environment History

- **50+ years history probing radiation-solid interactions**
  - Fundamental ion-solid understanding
  - Electronic system response to intense pulsed n/γ environments
  - Tritium containing materials
  - Fission/fusion reactor materials

RADIATION EFFECTS IN SEMICONDUCTORS

Proceedings of the Santa Fe Conference on Radiation Effects in Semiconductors, held October 3-5, 1967

Edited by F. L. Vook

Sandia Laboratories, Albuquerque, New Mexico

The field of radiation effects in semiconductors has rapidly advanced in recent years and substantial amounts of applicable evidence have been amassed.

VOLUME 16, NUMBER 3 APPLIED PHYSICS LETTERS 1 FEBRUARY 1970

ION IMPLANTATION DEPTH DISTRIBUTIONS: ENERGY DEPOSITION INTO ATOMIC PROCESSES AND ION LOCATIONS*

David K. Brice

Sandia Laboratories, Albuquerque, New Mexico 87115

(Received 20 October 1969; in final form 11 December 1969)

IEEE TRANSACTIONS ON NUCLEAR SCIENCE, VOL. 53, NO. 6, DECEMBER 2006

Damage Equivalence of Heavy Ions in Silicon Bipolar Junction Transistors

E. Bielejec, G. Vizkelethy, N. R. Kolb, D. B. King, and B. L. Doyle

SATURATION AND ISOTOPIC REPLACEMENT OF DEUTERIUM IN LOW-Z MATERIALS*

B.L. DOYLE, W.R. WAMPLER, D.K. BRICE and S.T. PICRAUX

Sandia National Laboratories†, Albuquerque, New Mexico 87185, USA

J. Nucl. Materials 93&94, 551 (1980)

Other SNL talks by Brandom Aguirre (this session) and Jose Pacheco (Friday session)

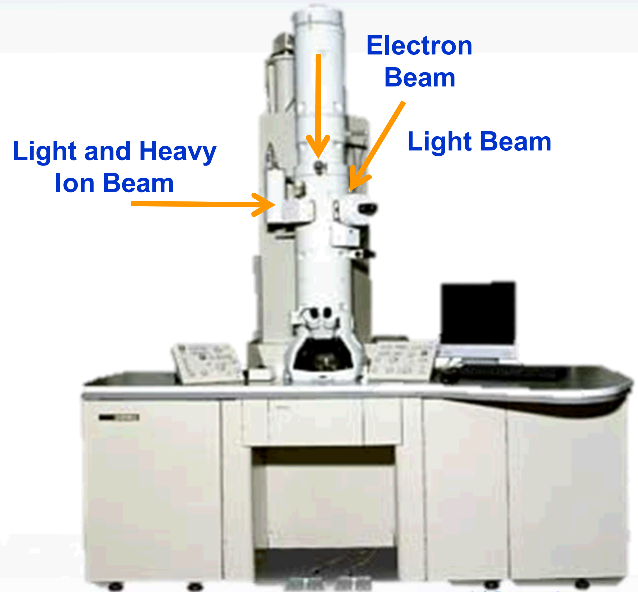
New Capabilities for *In Situ* Extreme Environments

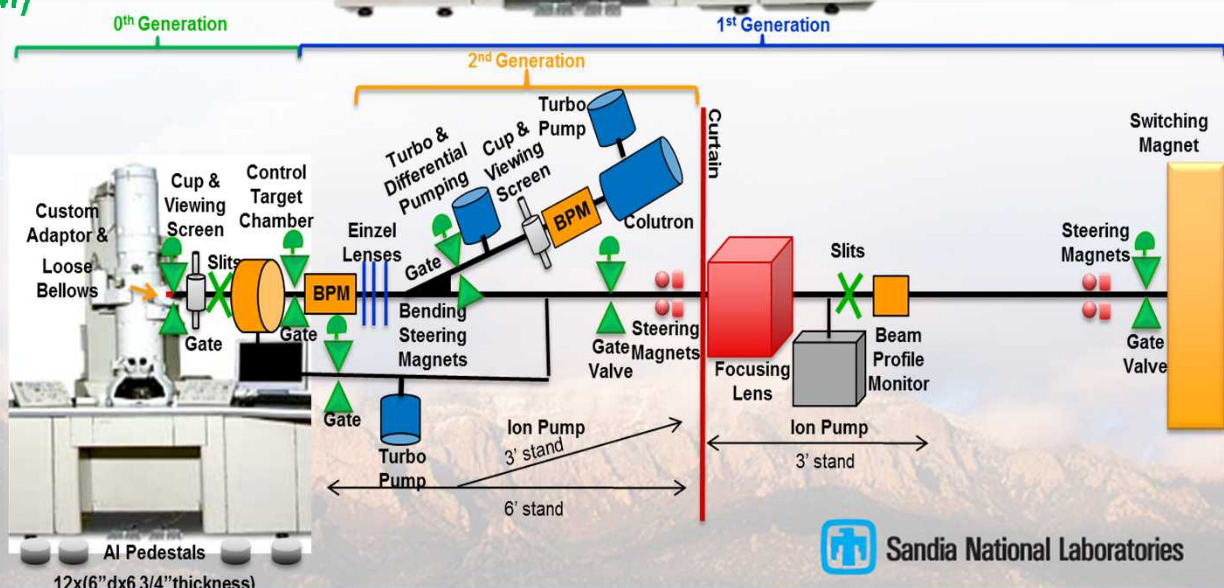
- **TEM with dual, co-linear ion beams**
  - Dual tilt, Heating, Tomography
  - Gas and Liquid Cells
  - Hysitron mechanical test stage
  - Precession Diffraction
- **Meso-Scale Mechanical Test with Ion Irradiation**
  - High Temperature (800C)
  - ASTM-standard sample geometry
  - 4.5MeV protons penetrate through 50um Cu samples

In situ Ion Irradiation TEM Facility

Capabilities:

- 200 kV LaB_6 TEM
- Ion beams used:
  - Range of Sputtered Ions from Tandem
  - 10 keV D_2^+
  - 10 keV He^+
  - (Simultaneous D_2+He)
- All beams hit same location
- Nanosecond time resolution (DTEM)
- Procession scanning (EBSD in TEM)
- In situ CL, and IBIL, and PL
- *In situ* vapor phase stage
- *In situ* liquid mixing stage
- *In situ* heating
- Tomography stage (2 axis)
- *In situ* straining stage
- *In situ* cooling stage
- *In situ* electrical bias stage





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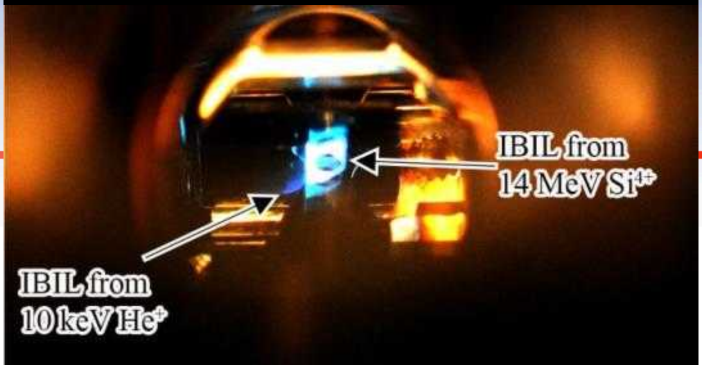
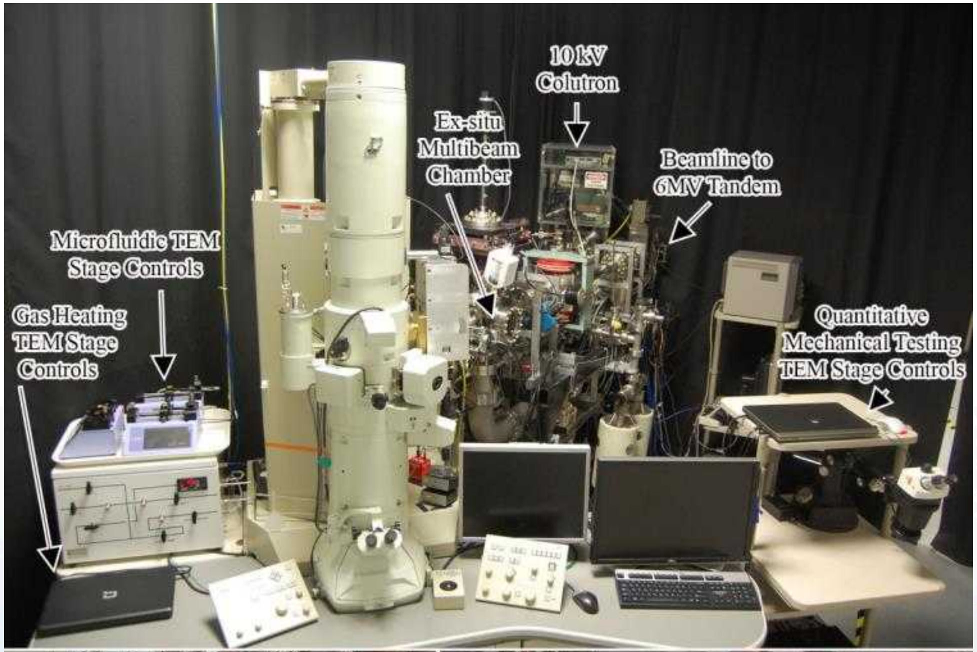
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

6 MV Tandem - 10 kV Colutron - 200 kV TEM

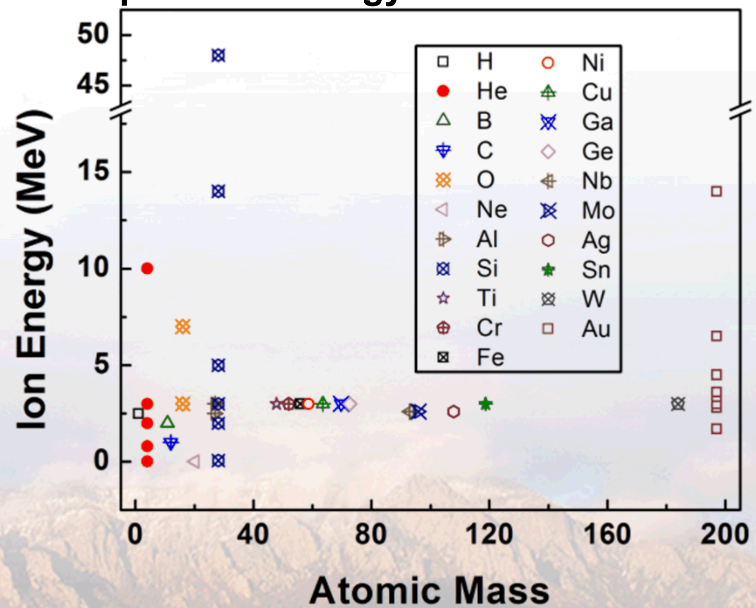
IBIL from a quartz stage inside the TEM





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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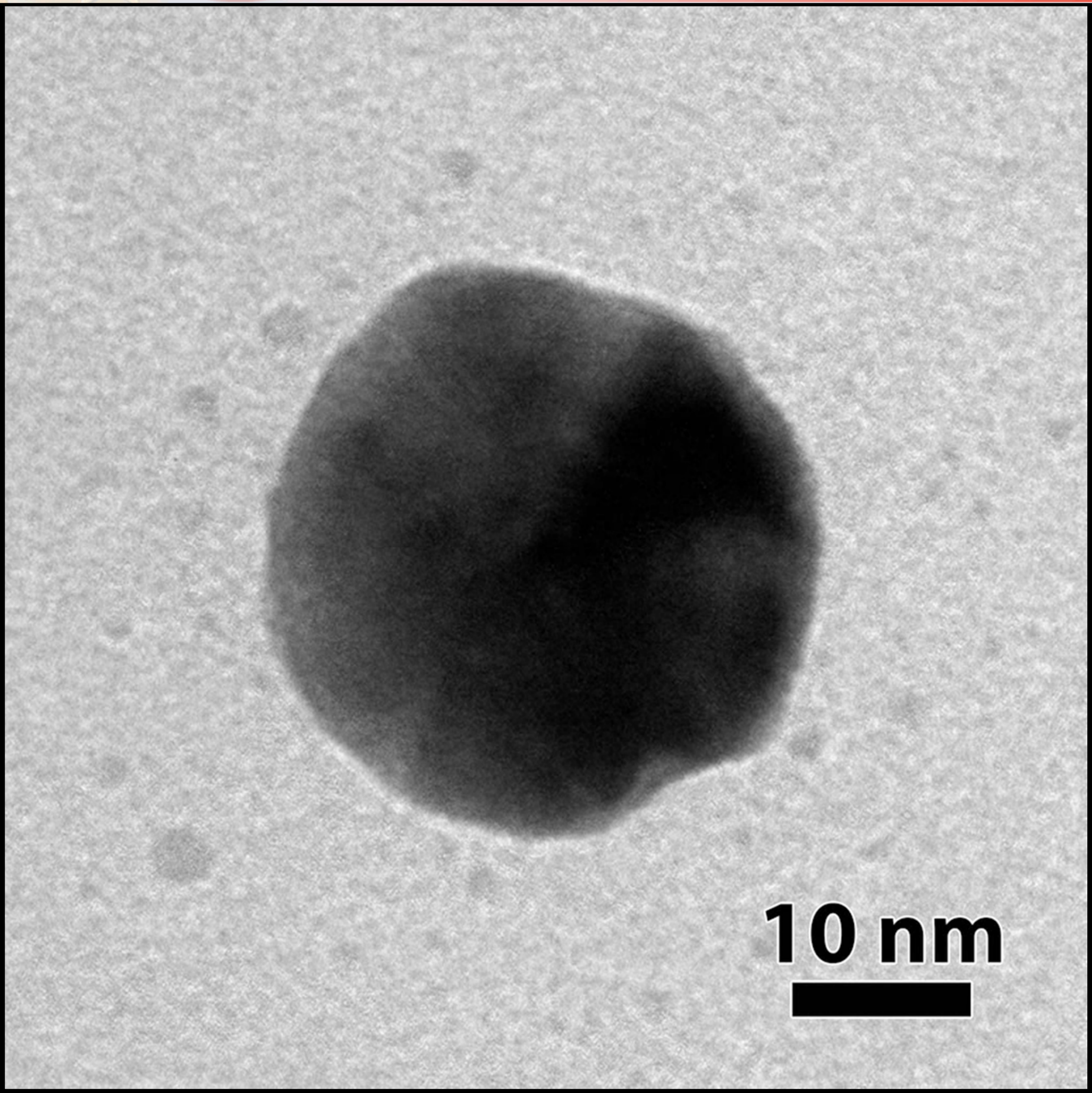
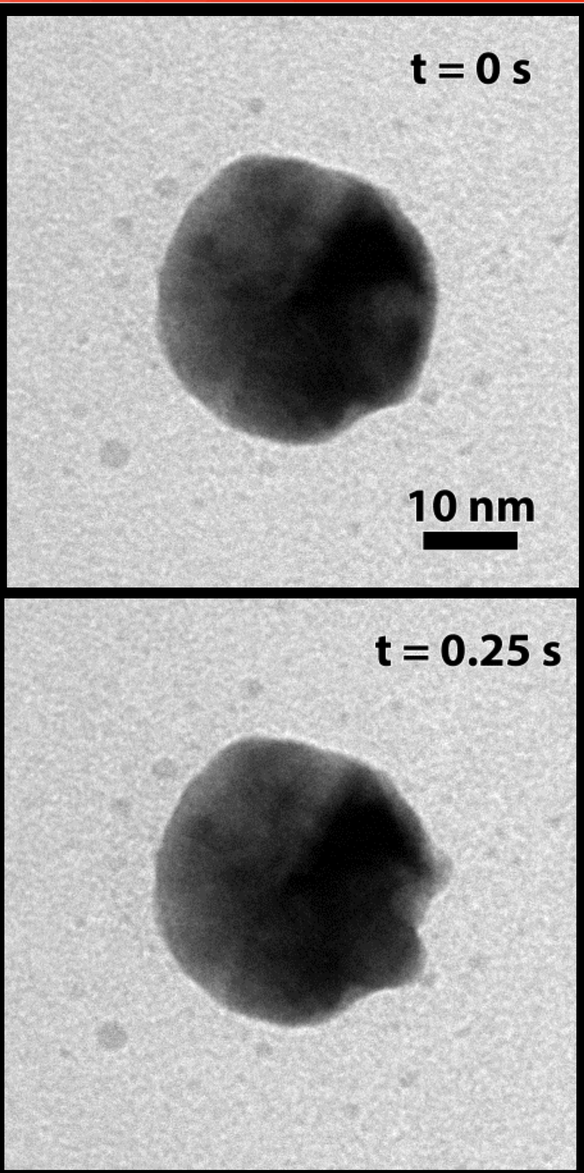
Single Ion Effects in 20 nm Au nanoparticle: 46keV Au ions (range ~7nm)

Collaborator: D.C. Bufford

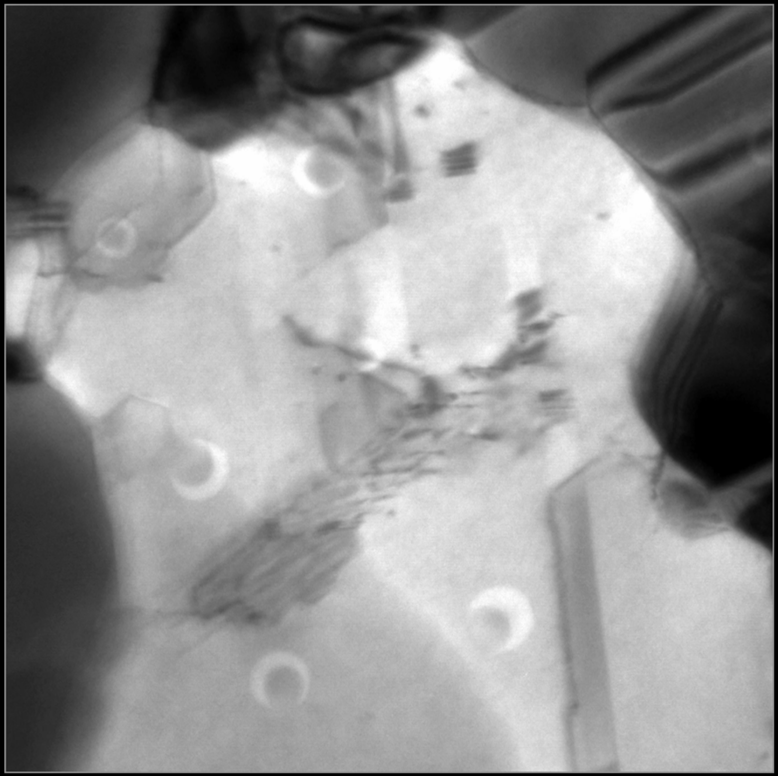




Single Ion Strikes – 2.8MeV Au^{4+} into/through Au film

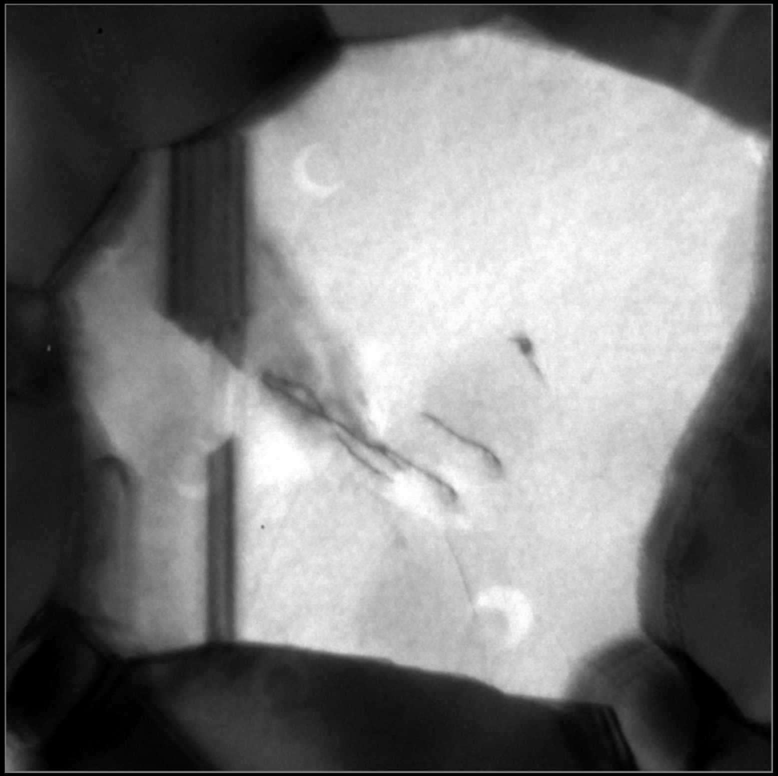
Collaborators: C. Chisholm & A. Minor

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



VS

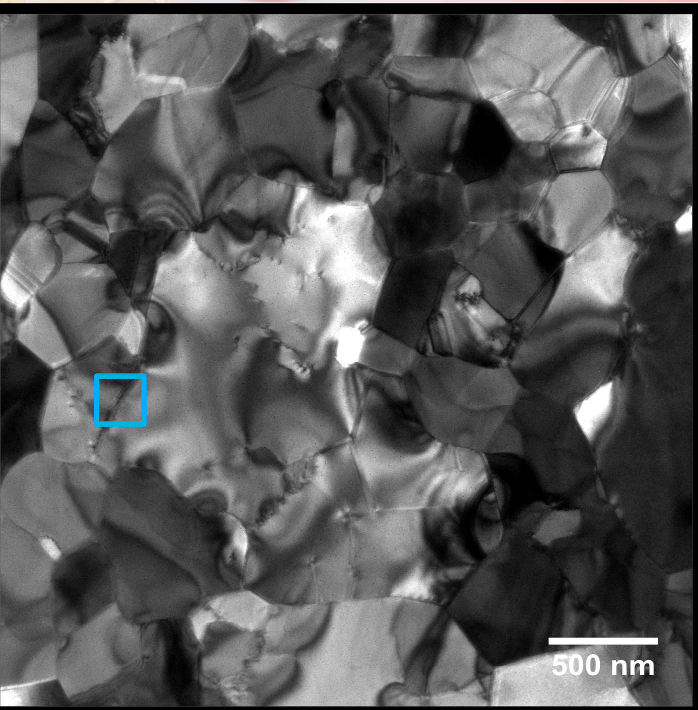
6.7 x 10^7 ions/cm^2/s

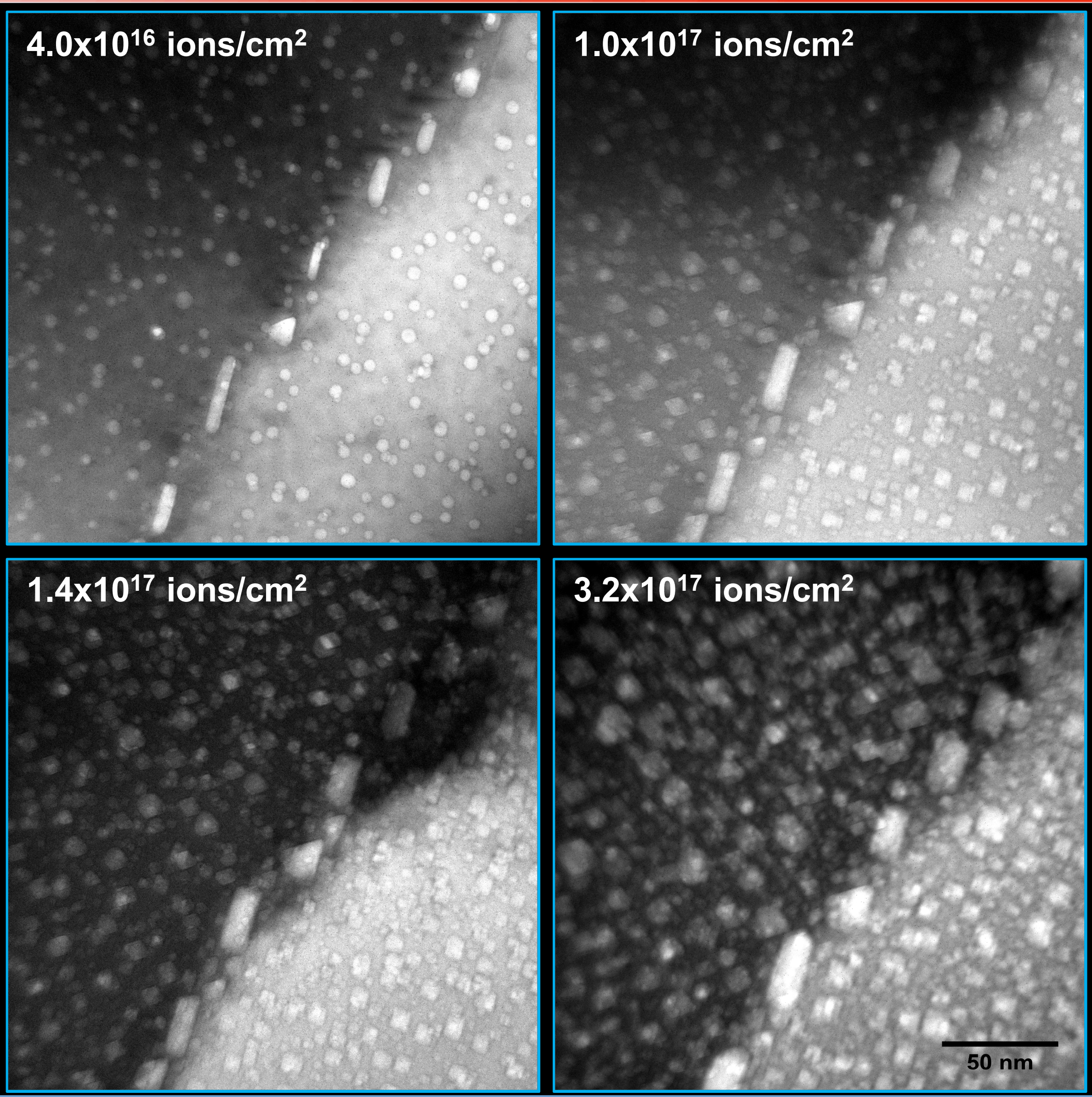


Improved vibrational and ion beam stability permits us to work at 120kx or higher permitting imaging of single cascade events

In situ Implantation

Collaborators: C. Chisholm & A. Minor



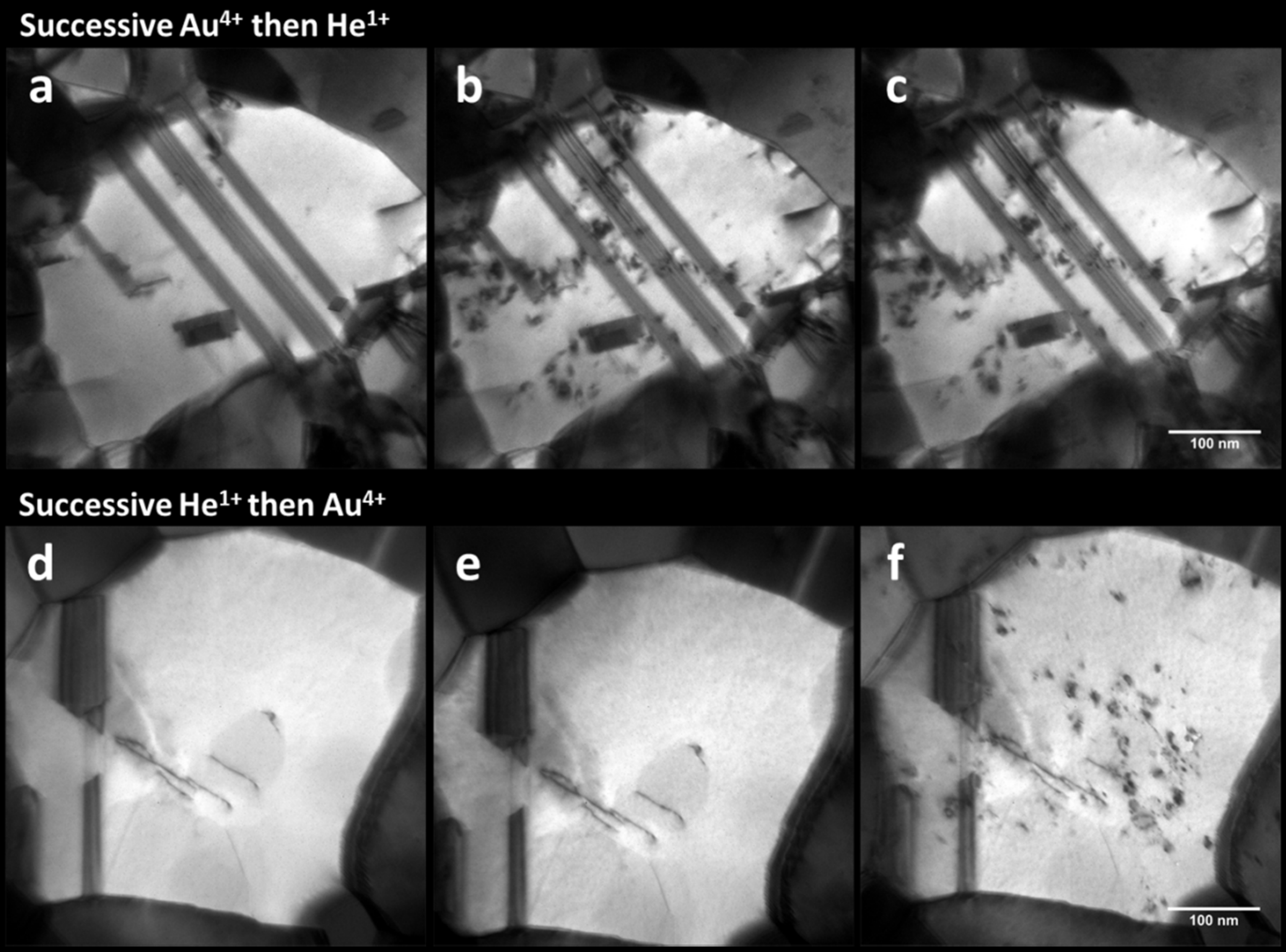


Au implanted with 10keV He^+

He bubble formation on grain boundary and in the bulk

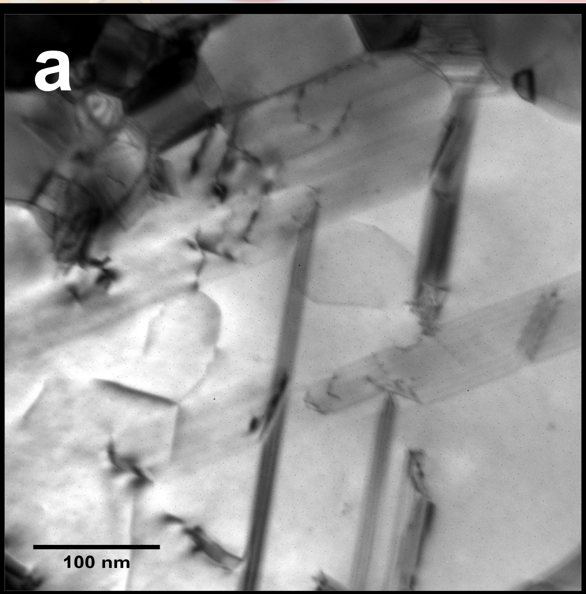
In situ Successive Implantation then Irradiation ($2.8MeV\ Au^{4+}$, $10keV\ He^{+}$)

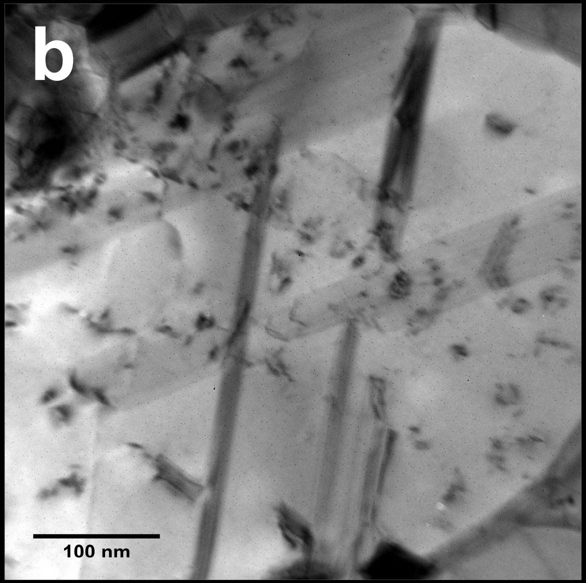
Collaborators: C. Chisholm & A. Minor



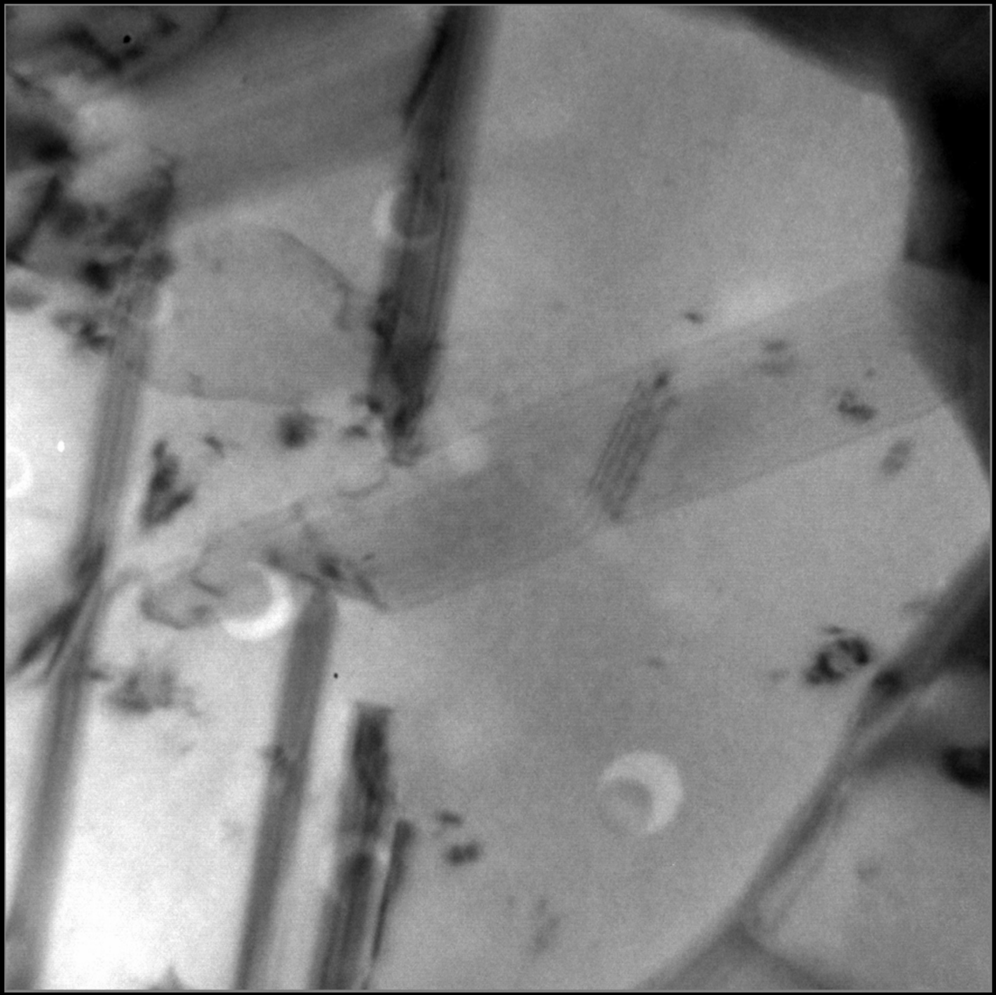
In situ Concurrent Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor

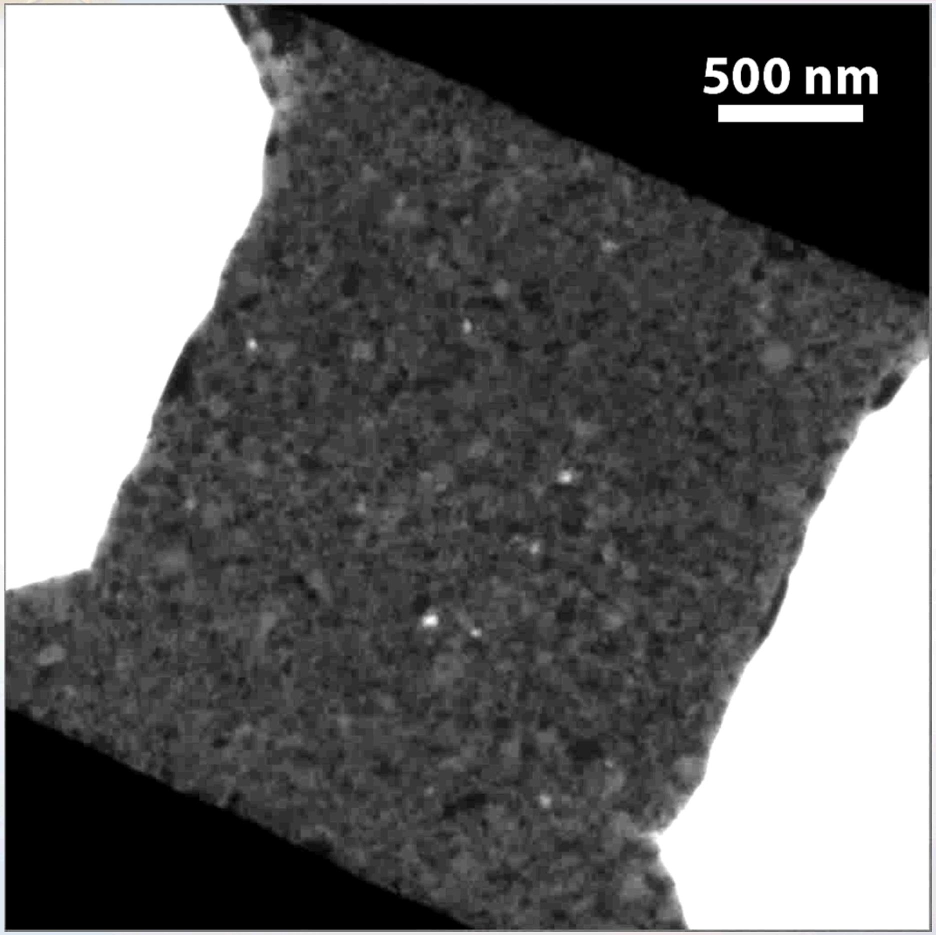




He^+ implantation and concurrent Au^{4+} irradiation: Transient bubble formation and dissolution

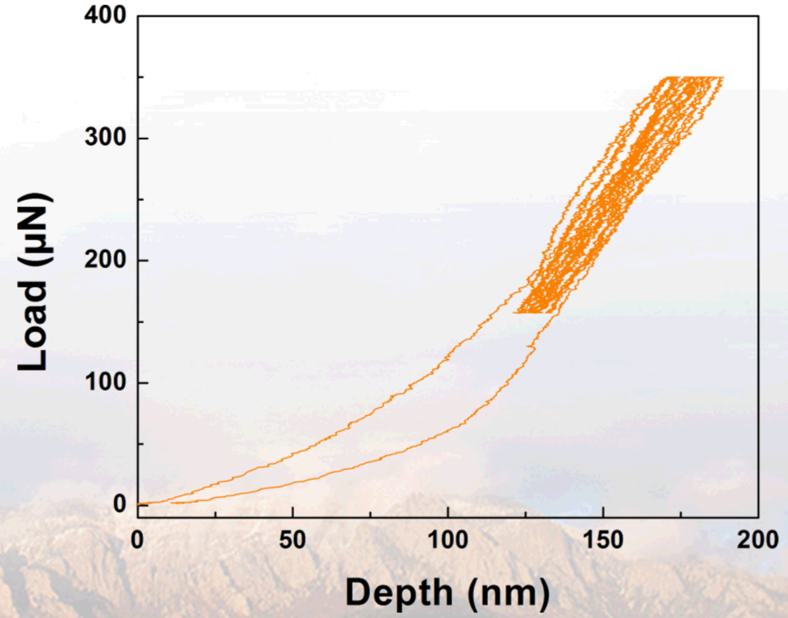


In Situ Tensile Testing



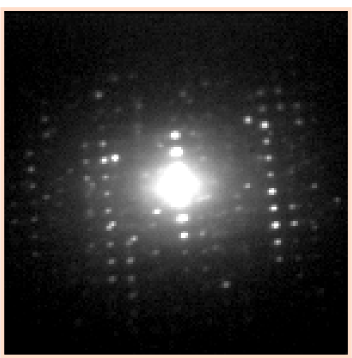
- Slow crack propagation
- Evidence of grain growth

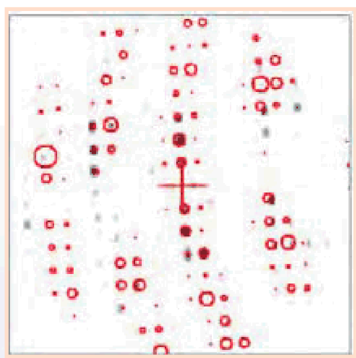
- **Cyclic loading:**
  - Crack initiated in previous monotonic test
  - 9 cycles to ~87.5% of that load
  - 50 % unloading
  - Slow crack propagation

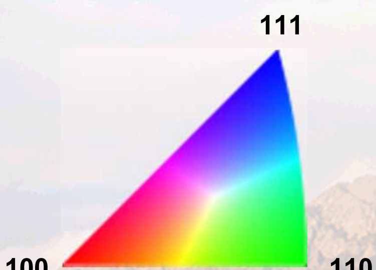


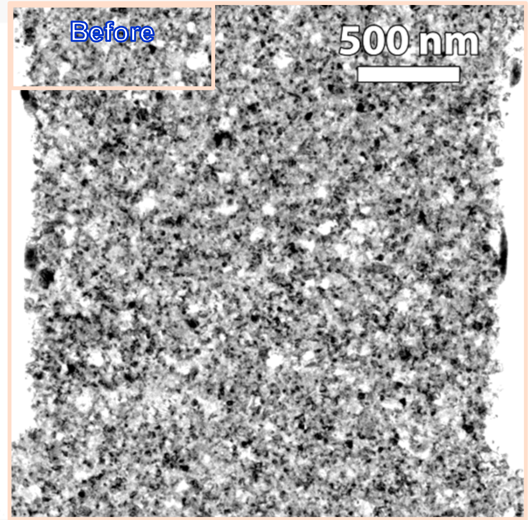
Precession Diffraction: Quantifying Microstructural Change

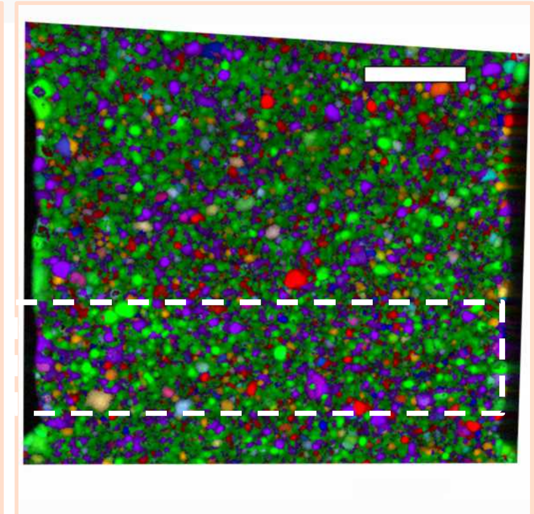
- **Combining orientation mapping with deformation**
- **EBSD-like capability in the TEM**
  - Powerful analytical tools available

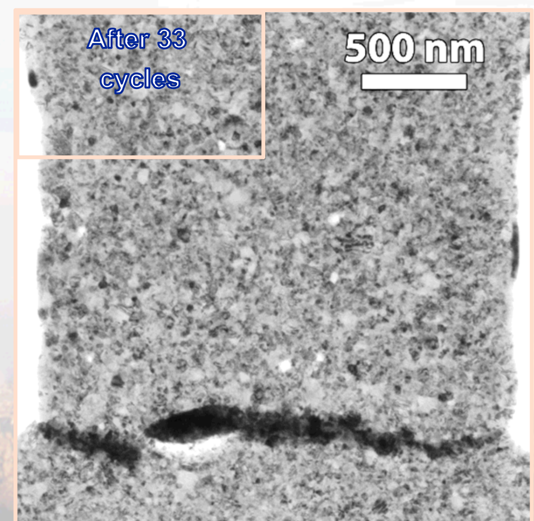


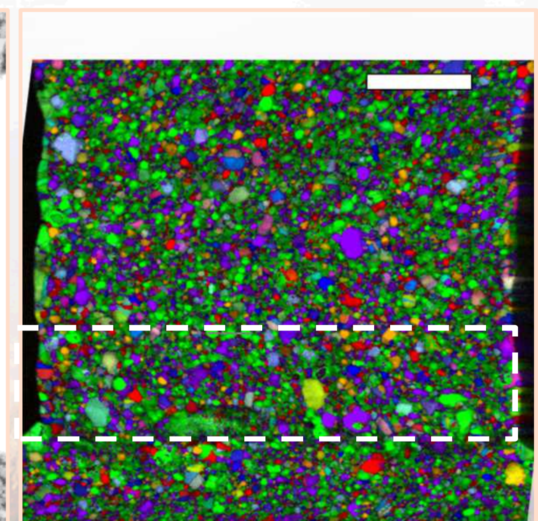








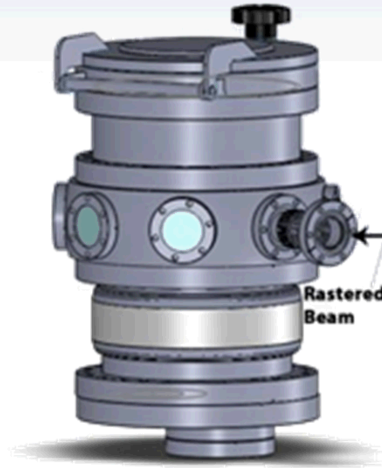


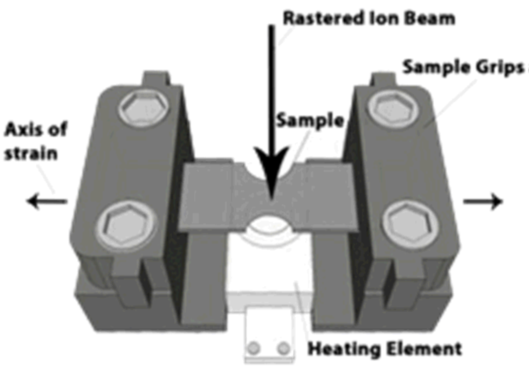


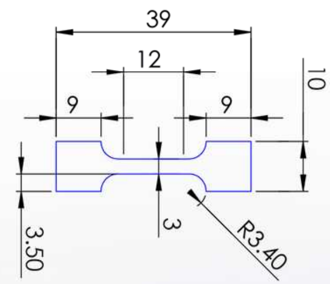
Extreme Environments at the Meso Scale

- **Meso-scale mechanical test**
  - Straining + Heating stage
  - Ions from the 6MV Tandem
  - Heating to 800C

- **30 GPa stress, 58% strain**
  - Geometry based on ASTM-E8
  - 50 – 100 μm sample thickness
  - Limited to 4.5MeV protons by radiation levels (neutron production by SS)

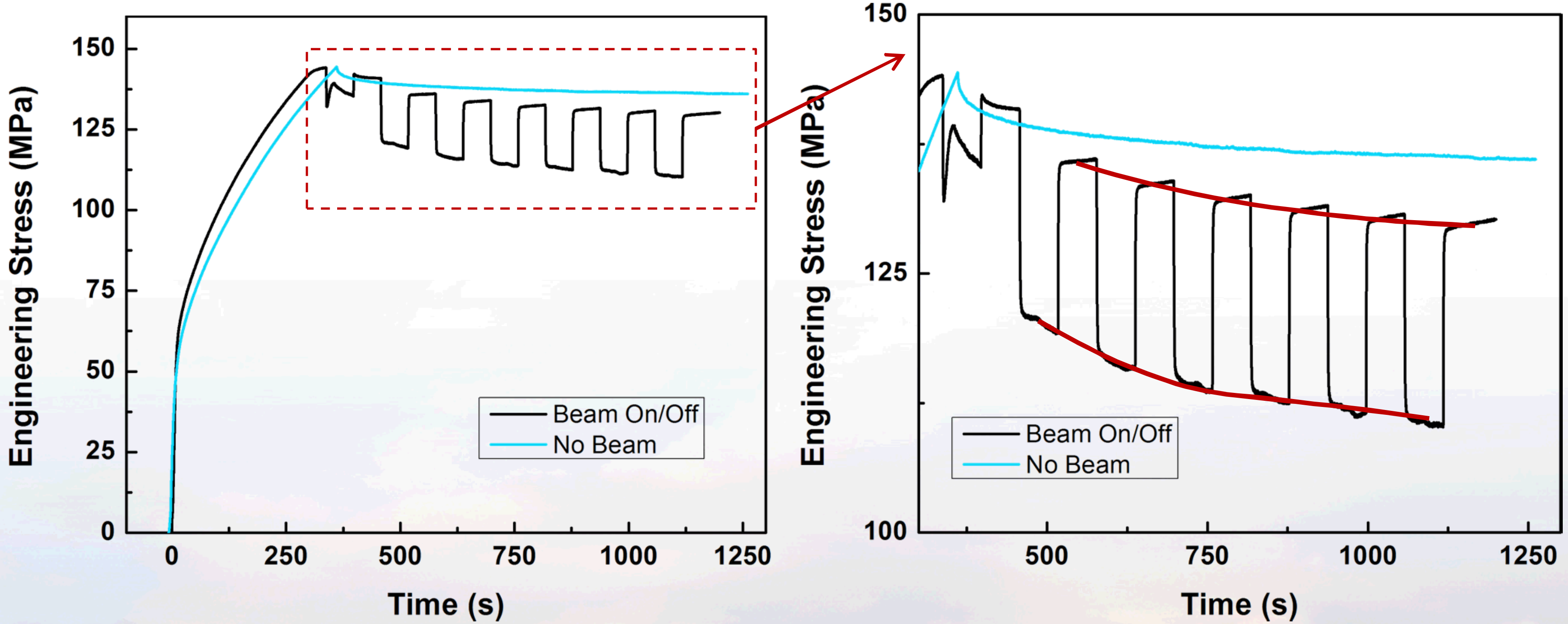






Copper Stress Relaxation with/without Irradiation

- **Commercial OHFC Copper, 50μm thick**
  - Similar 0.25mm/min elongation rate to 22.5 N load
  - Approximately 75% of typical ultimate tensile load
  - 4.5 MeV protons have projected range of 65μm



- **Preliminary data – still optimizing configuration**
- **Different relaxation rates with beam on and off**

Summary

- **Sandia has developed unique in-situ experimental capability**

- **I3TEM – *In situ* experimental workhorse**
  - Dual, colinear ion beams (keV to MeV)
  - Broad array of stages to tailor environment
  - Detailed microstructure characterization
  - Mechanical characterization

- **Meso-scale mechanical properties**
  - Straining + heating stage
  - MeV ion beam for defect generation or implantation