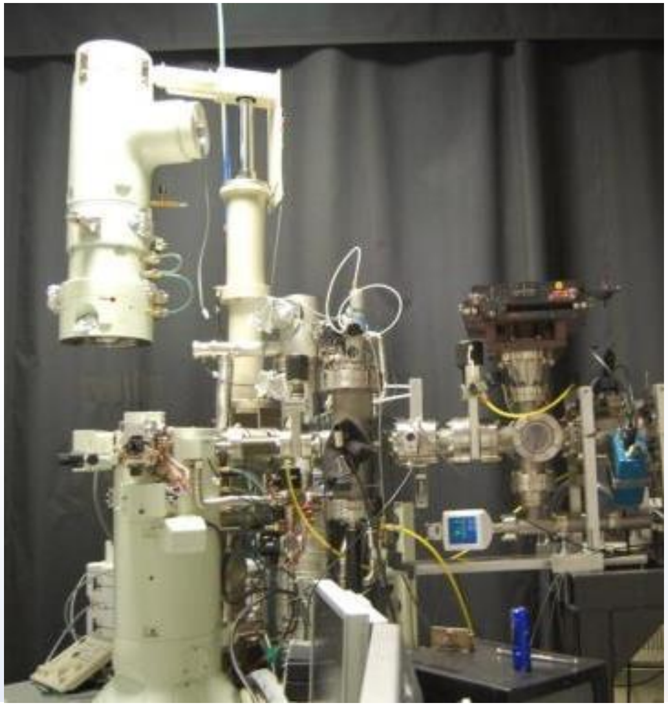
Sandia's *In-situ* Ion Irradiation TEM Facility

SAND2014-19057PE

K. Hattar

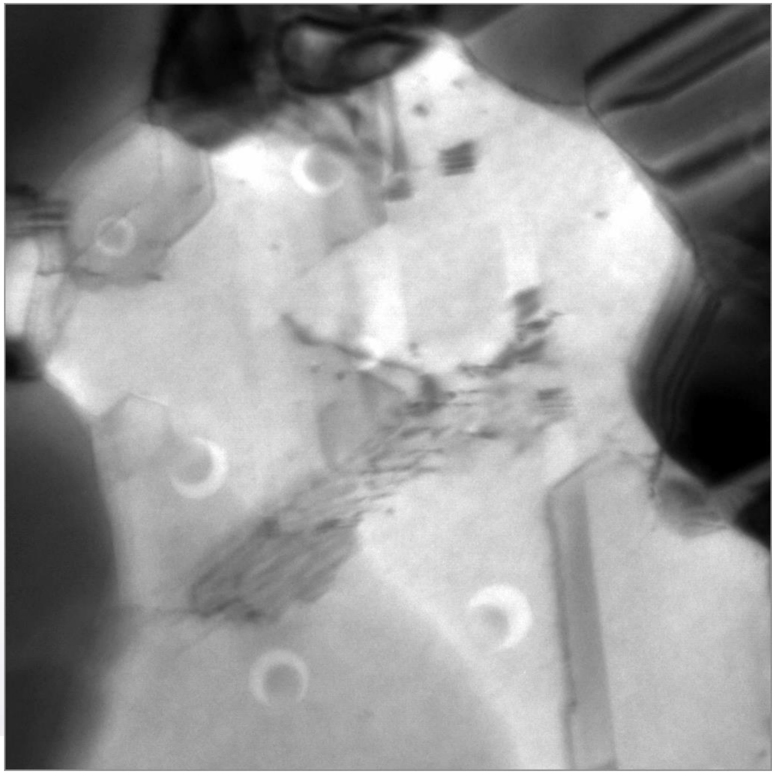
Ion Beam Lab at Sandia National Laboratories

November 21, 2014



***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, J. Villone, G. Vizkelethy, 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, P. Feng, F.P. Doty, B.A. Hernandez-Sanchez, P. Yang, J-E Mogonye, S.V. Prasad , P. Kotula, S. Howell, T. Ohta, & T. Beechem**
- **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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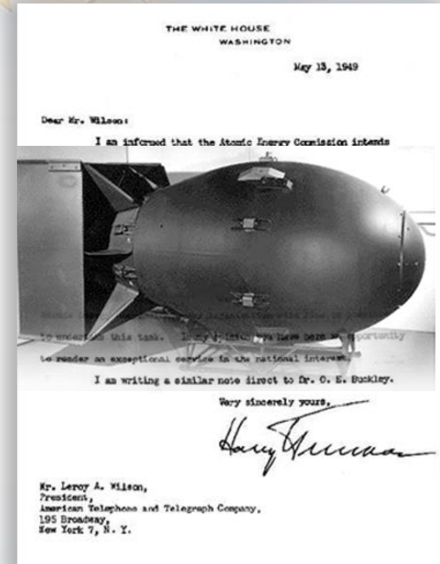
Sandia National Laboratories

"Exceptional service in the national interest"



THE WHITE HOUSE
WASHINGTON

May 13, 1949

Dear Mr. Wilson:

I am informed that the Atomic Energy Commission intends

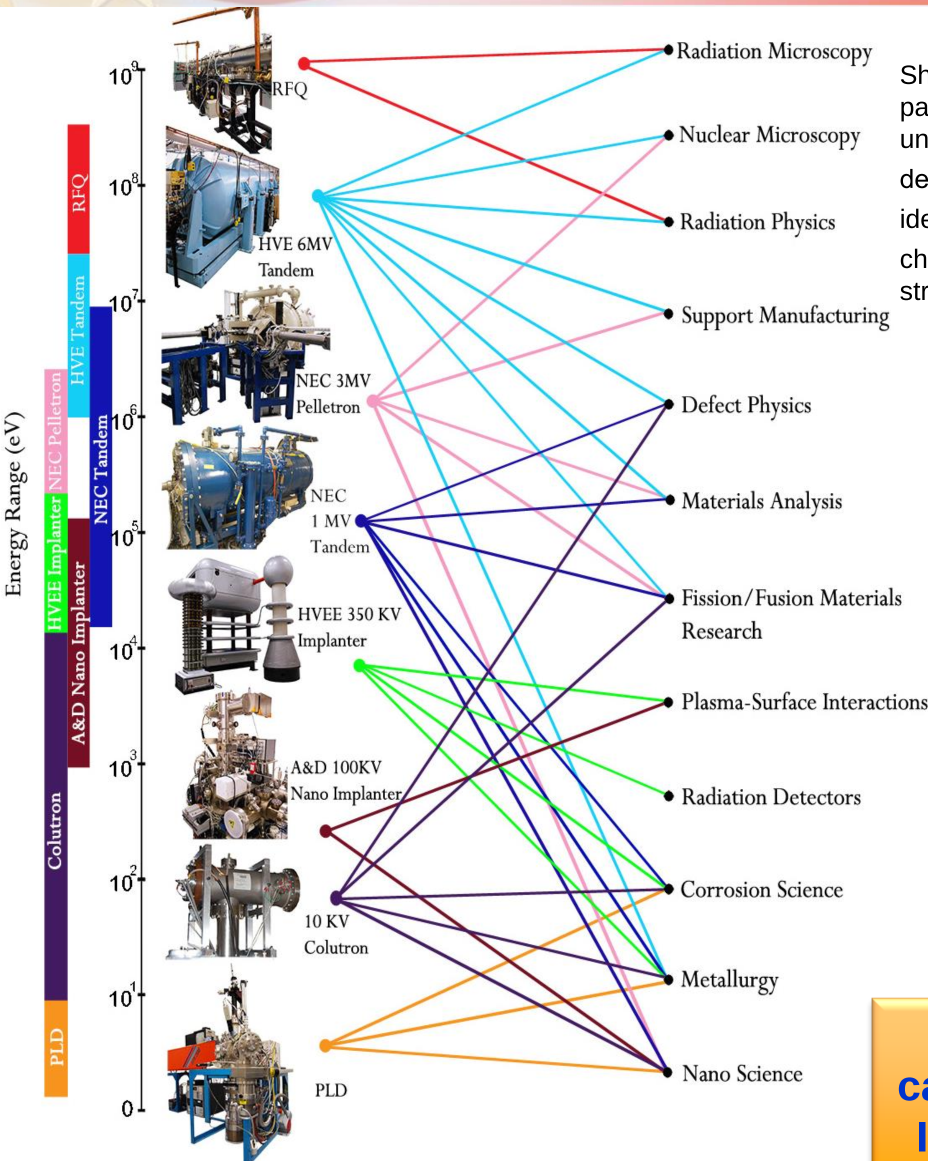
to render an exceptional service in the national interest.

I am writing a similar note direct to Dr. O. E. Buckley.

Very sincerely yours,

Mr. Leroy A. Wilson,
President,
American Telephone and Telegraph Company,
195 Broadway,
New York 7, N. Y.

Sandia's Ion Beam Laboratory

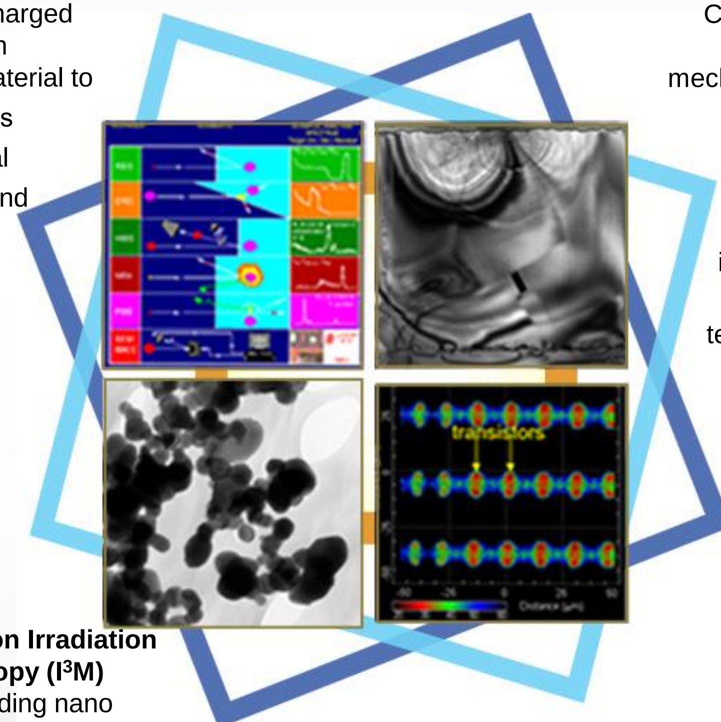


Ion Beam Analysis (IBA)

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 capability ion beam set including and *In situ* Ion Irradiation Transmission Electron Microscopy.

Potential Evolution of System Design

Use the Nearest Stone

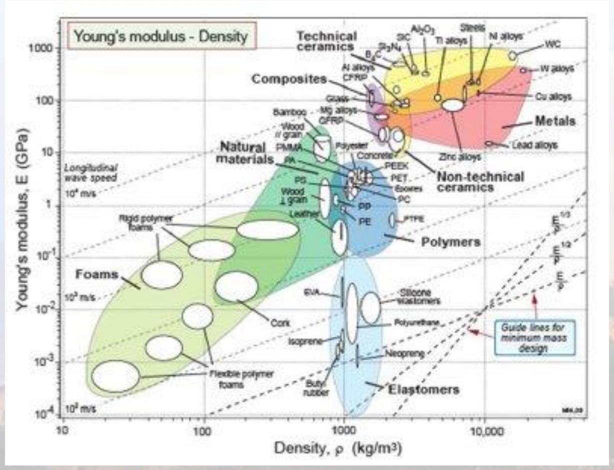
to

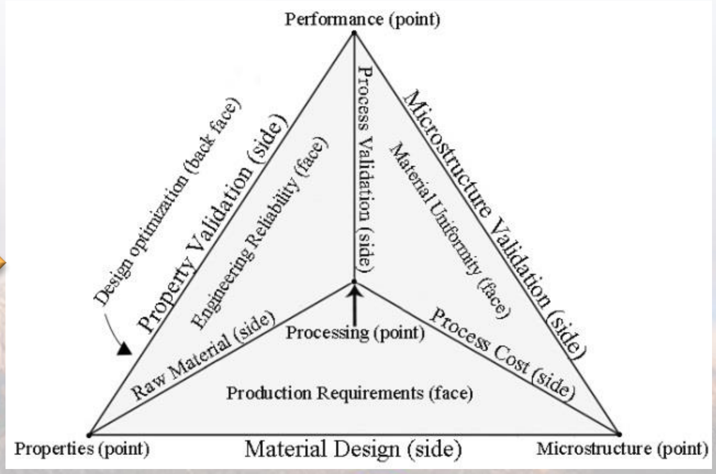
- Radar charts and Ashby plots of current material
- Accelerated and field testing
- **Scientist create a new materials. Engineers find an application.**

Materials by Design

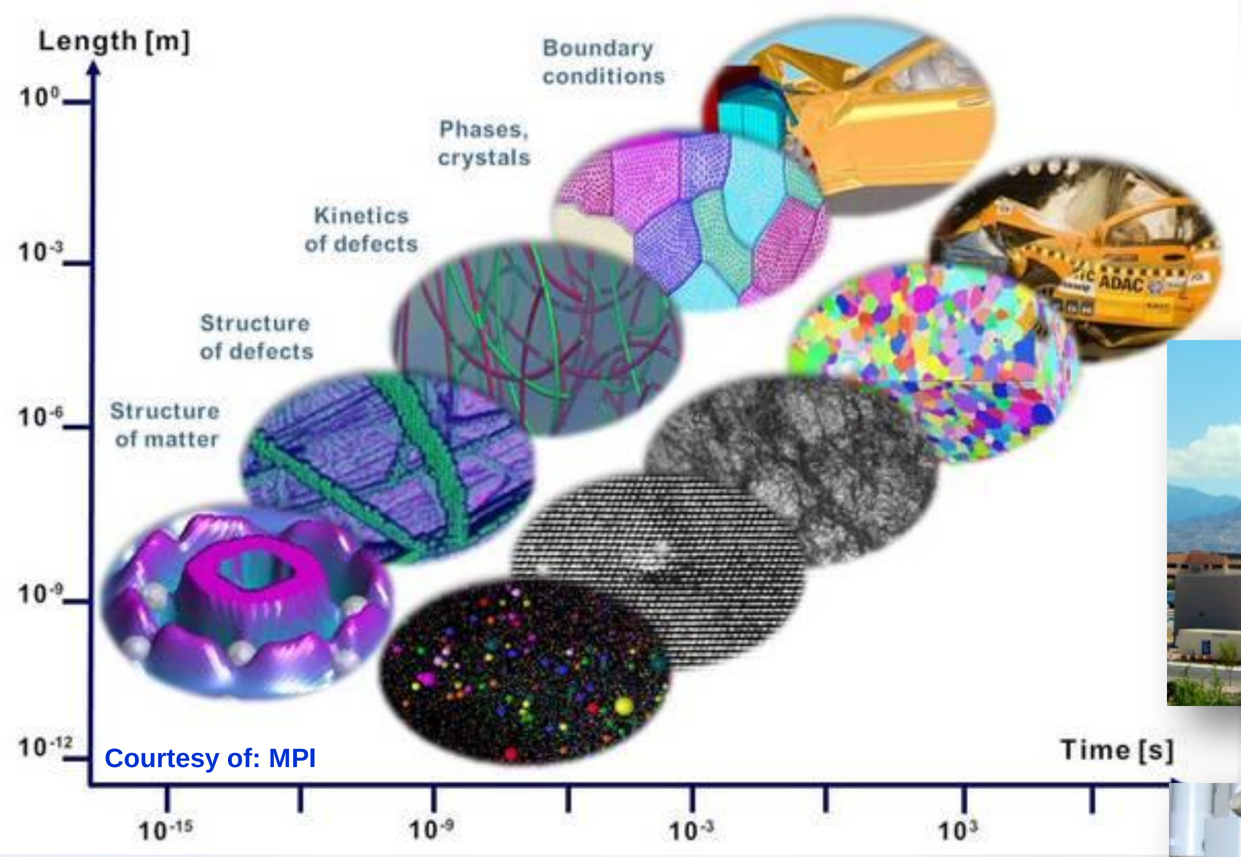
- **Physics-based approach**
- **Requires multiscale modeling**
- **Engineers require given properties, Scientists tailor the chemistry and microstructure to achieve it.**

Great vision! We are making strides, but we are not there yet



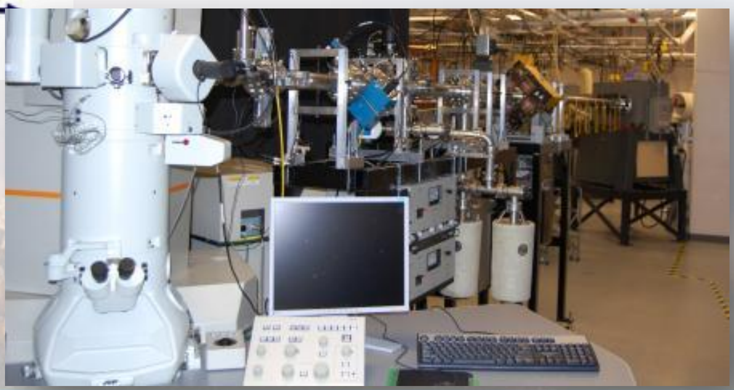


Investigating the nm Scale to Understand the km Scale to Understand Materials Response in the Extremes



Ion Beam Lab (IBL)

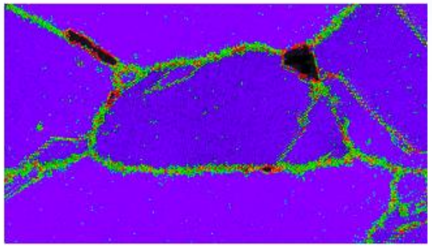
In situ Ion Irradiation TEM (I^3TEM)

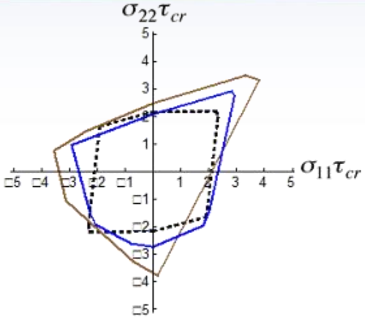


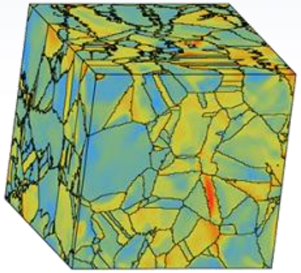
To develop predictive physics-based models, a fundamental understanding of the structure of mater, defects, an the kinetics of structural evolution in the environments of interest are needed

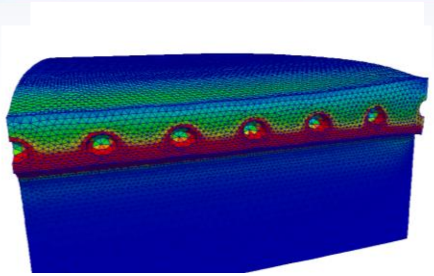
Multiscale Simulation & Experiments to Understand and Predict the Sources of Material Variability

simulations

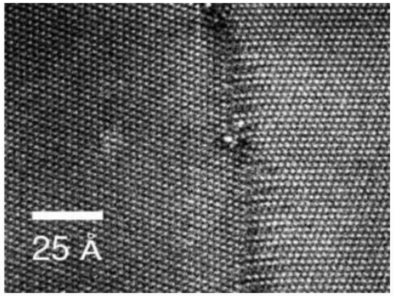


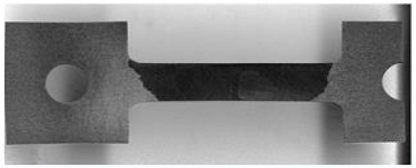


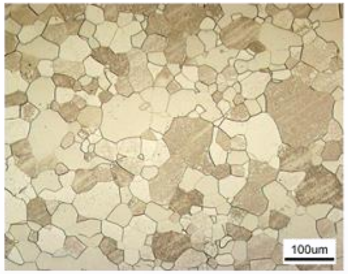


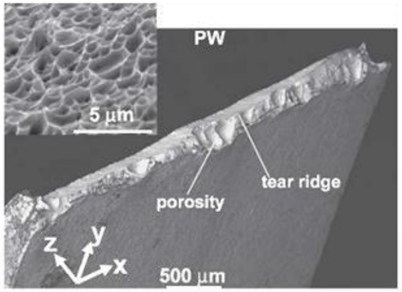


experiments









Atomic scale phenomena
10^{-9} m 10^{-9} s

Single crystal behavior
10^{-6} m 10^{0} s

Microstructural effects
10^{-3} m 10^{3} s

Material performance
10^{0} m 10^{6} s

Atoms-up: Develop physics-based models to provide scientific insight

Continuum-down: Augment engineering-scale models to provide improved fidelity

Where have Ion Beam Modified Materials been Utilized?

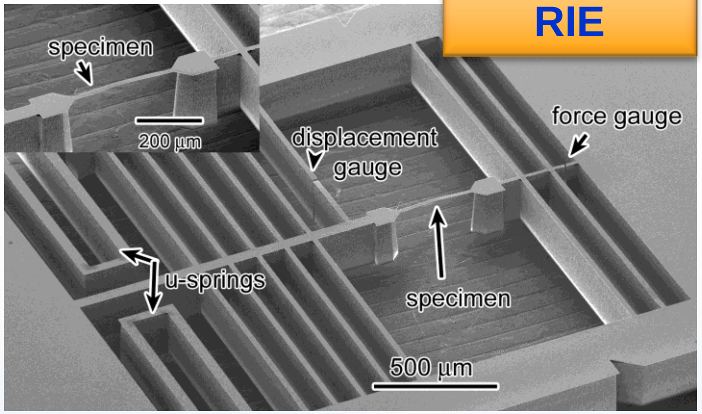
Ion Beam Modification (IBM)

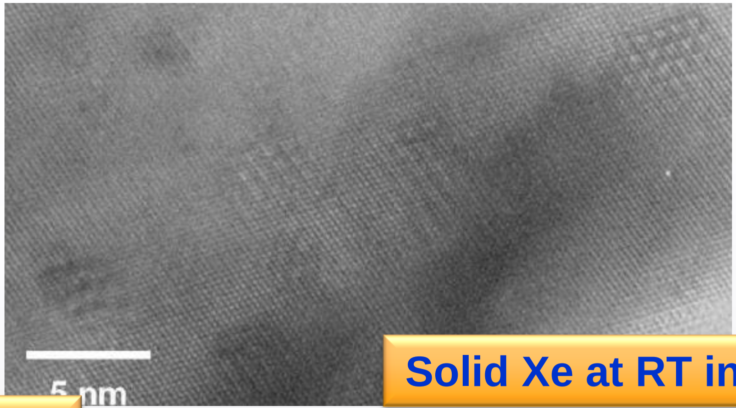
Changing the optical, mechanical, and chemical properties of materials via ion implantation to meet technological needs

Dopants in Si found in every microelectronic device

Thompson et al. Science 2007

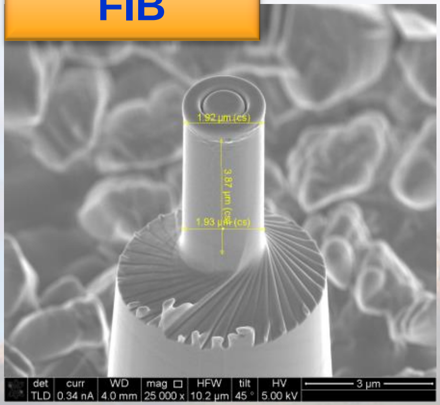
RIE



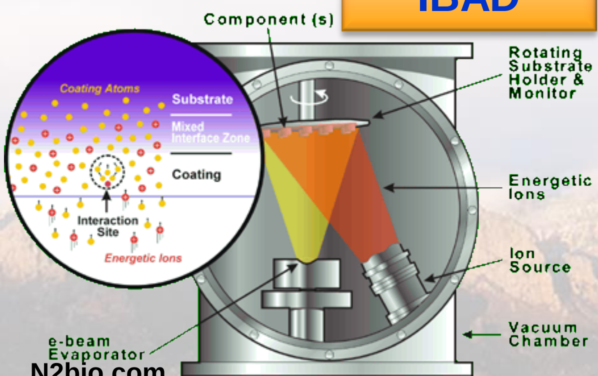


Solid Xe at RT in Al

FIB



IBAD



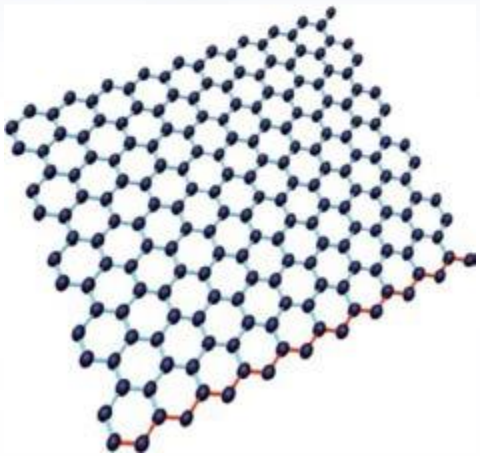
N2bio.com

Proton Cancer Therapy

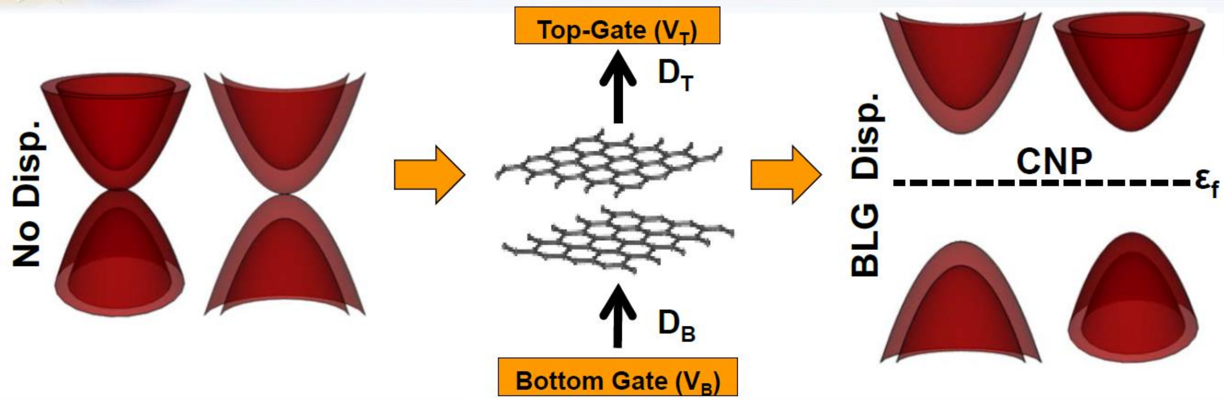
Ion Beam Applications iba-worldwide.com

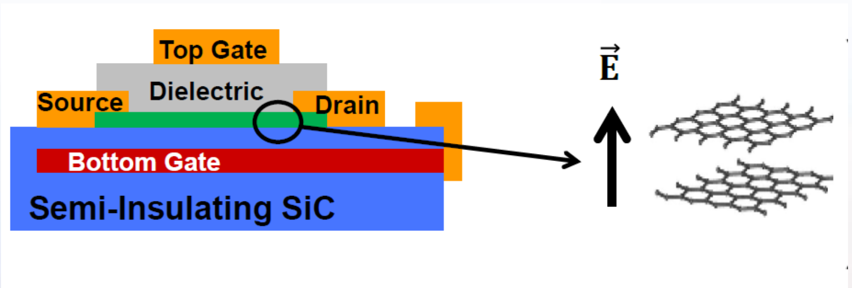
Development of a Dual-Gated Bilayer Graphene Device

Collaborators: S. Howell, T. Ohta, & T. Beechem



2D hexagonal net of sp2 bonded carbon atoms





A combination of displacements (top & bottom gates) are needed to:

- Induce a bandgap
  - $D_{ave} \neq 0$
- Control Fermi Level (ε_f) to charge neutrality point (CNP)
  - $\delta D = 0$

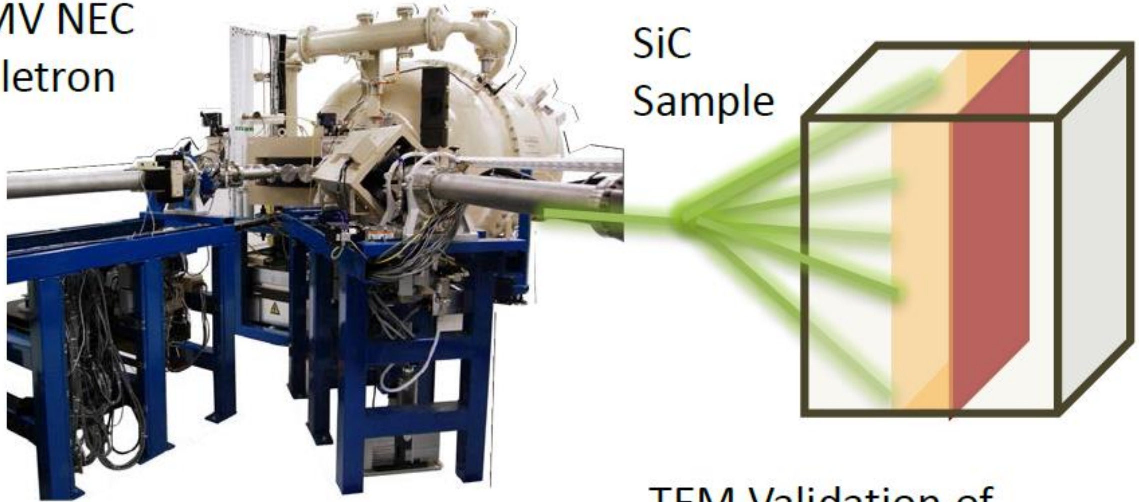
Zhang *et al.*, *Nature*, 459, 820 (2009)

3 MeV Nitrogen Implants to Form a Back-Gate in Semi-Insulating SiC

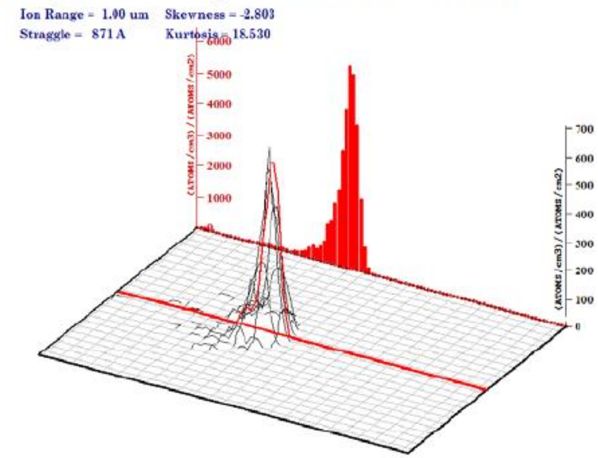
Collaborators: S. Howell, T. Ohta, & T. Beechem

3 MV NEC Pelletron

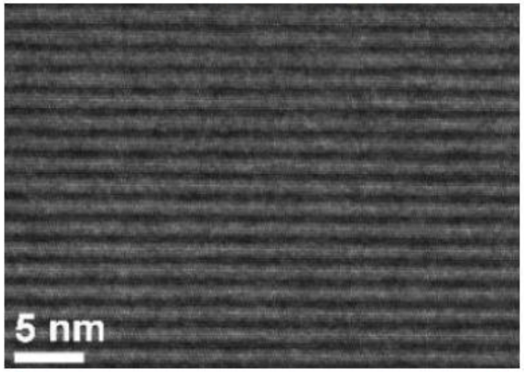
SiC Sample



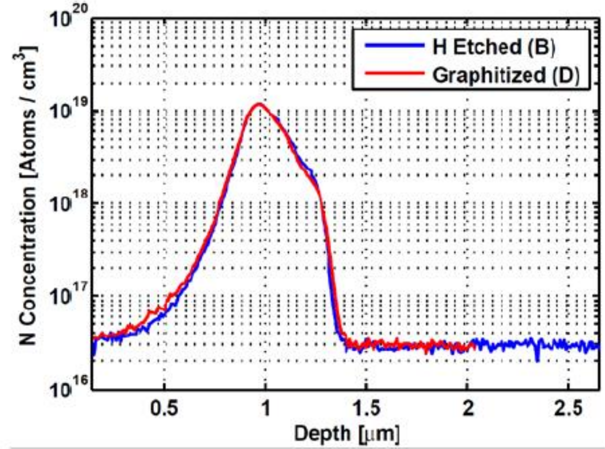
TRIM Simulation of Blanket Implant Depth Distribution



TEM Validation of Microstructure



Validation of Implant Depth (SIMS)



Ion Implantation Results

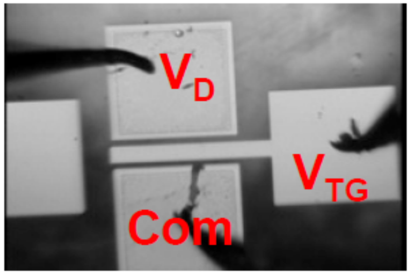
- Implanted N ions (target depth of 1 μm)
- Capacitance measurements indicated an isolated conductive layer at a depth of ~ 700 nm in the SiC

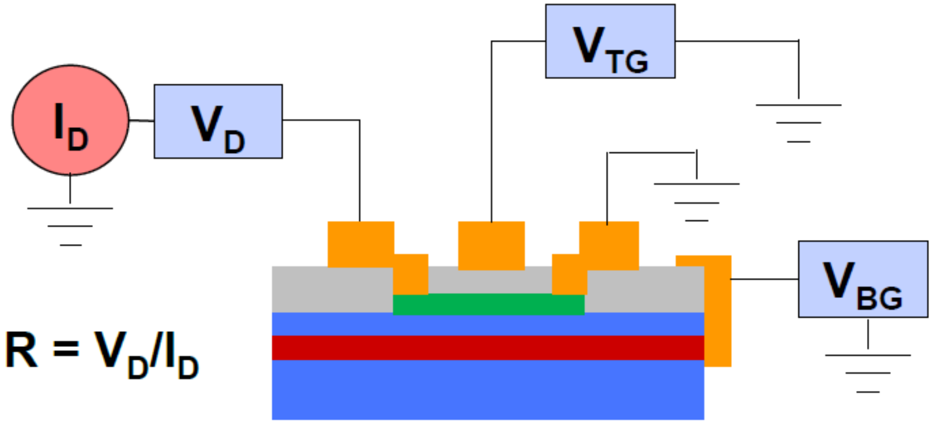
Tailored ion implantation provides both back gate and side contact structure

Waldmann *et al*., Nature Materials, **10**, 357 (2011)

Bandgap Control via Dual Gating
(1st Demonstration of scalable dual-gate BLG FETs)

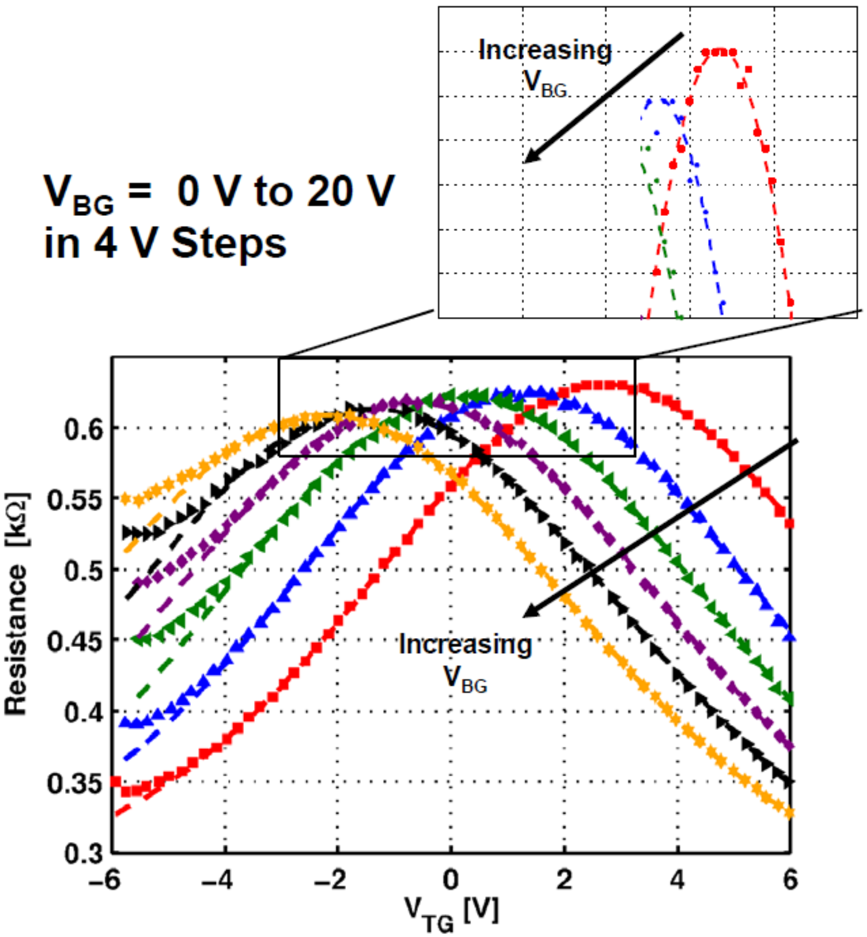
Collaborators: S. Howell, T. Ohta, & T. Beechem



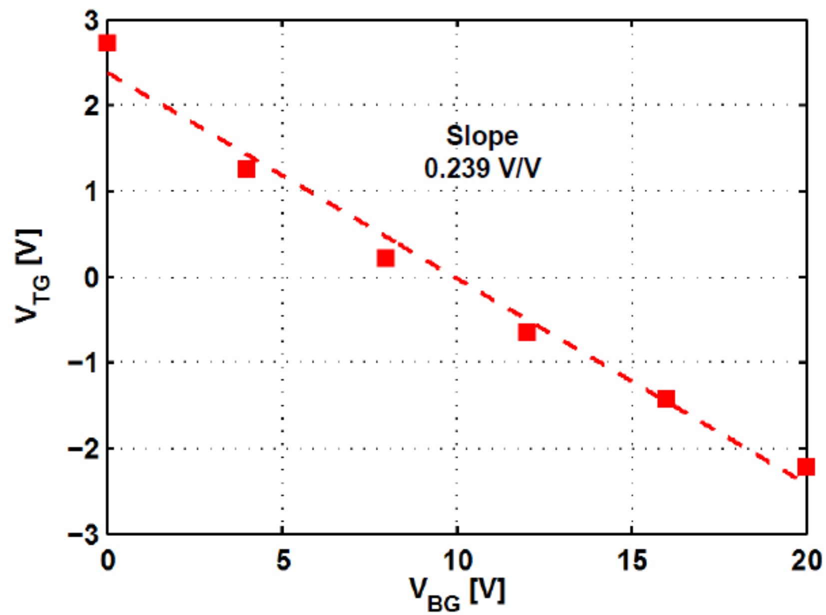


$R = V_D/I_D$

V_{BG} = 0 V to 20 V in 4 V Steps



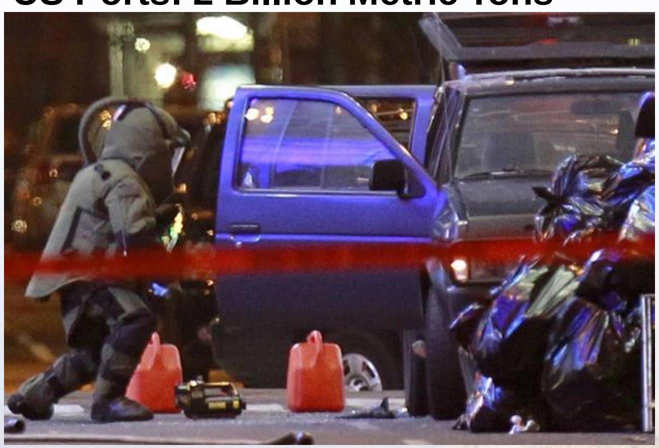
$$Slope = -(\varepsilon_b d_t)/(\varepsilon_t d_b)$$



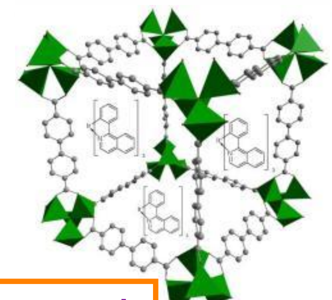
Interest in Scintillators

Crucial to understand radiation-solid interactions on multiple length scales

US Ports: 2 Billion Metric Tons



Scintillators with low energy resolution & detection efficiency cannot distinguish radiation type or quantify radiation

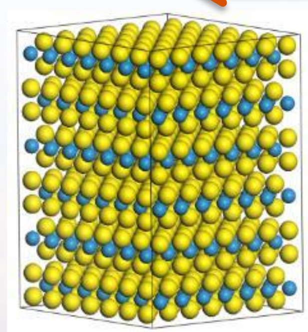


Non-Interpenetrated IRMOF-10

Single Crystal $CdWO_4$

Plastic Scintillators

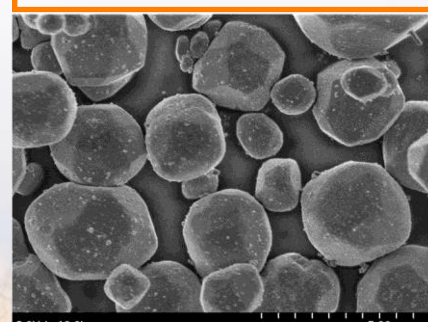
Nano — **Micro** — **Meso**



Tungsten (IV) Sulfide WS_2

High-Z ME_x Nanoscintillator

Commercial $CaWO_4$ Scintillating Powder

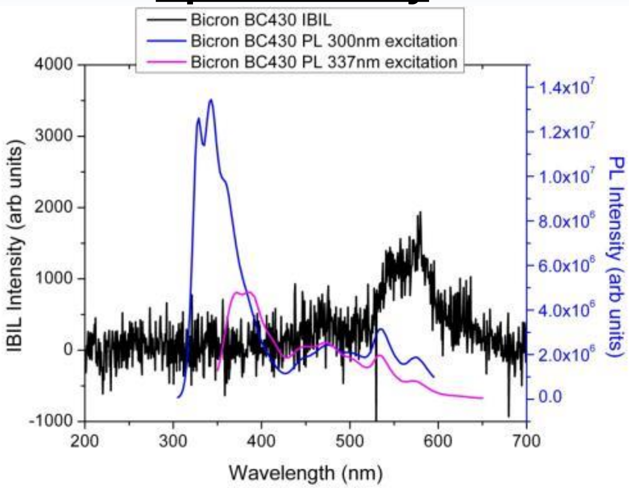


High-Z Nanocomposite

IBL Capabilities for Luminescence Studies

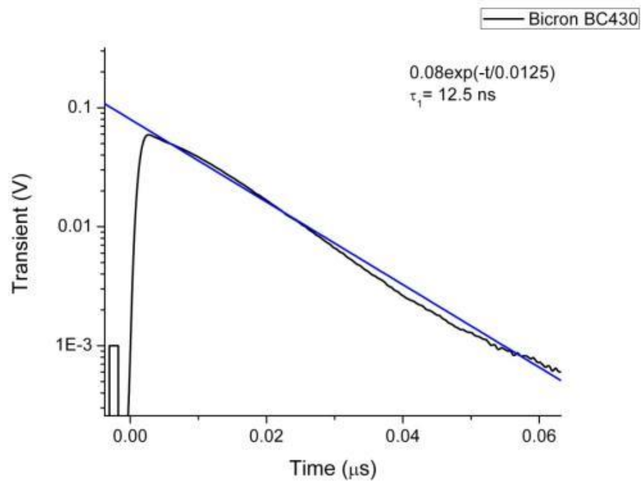
Collaborators: J. Villone & G. Vizkelethy

Spectrometry



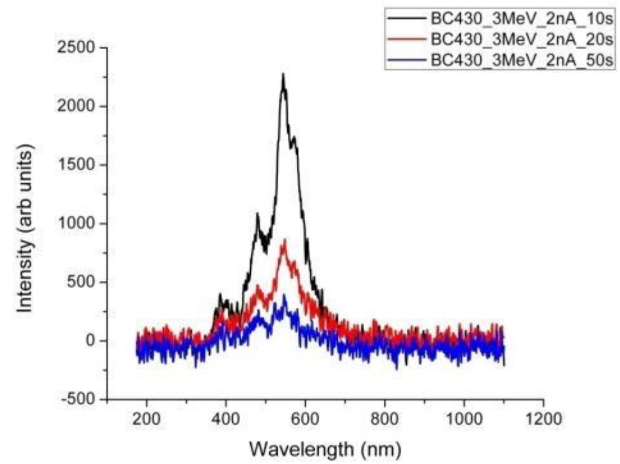
- 3 MeV H^+ beam
- Thin films of samples on PIN diodes
- Hamamatsu PMT run in photon-counting mode
- Light intensity measured as a function of time after ion strike

Decay Time



- 3 MeV H^+ beam used as excitation
- Scintillation light collected as ion beam excites sample
- Light collected with OM-40 microscope or fiber optic mounted close to sample
- Avantes AvaSpec 2048 spectrometer

Radiation Hardness



- Radiation hardness experiments performed with 3 MeV H^+ beam from Tandem accelerator
- IBIL spectra measured constantly as sample exposed to beam
- Overall decrease in emitted light observed due to radiation damage

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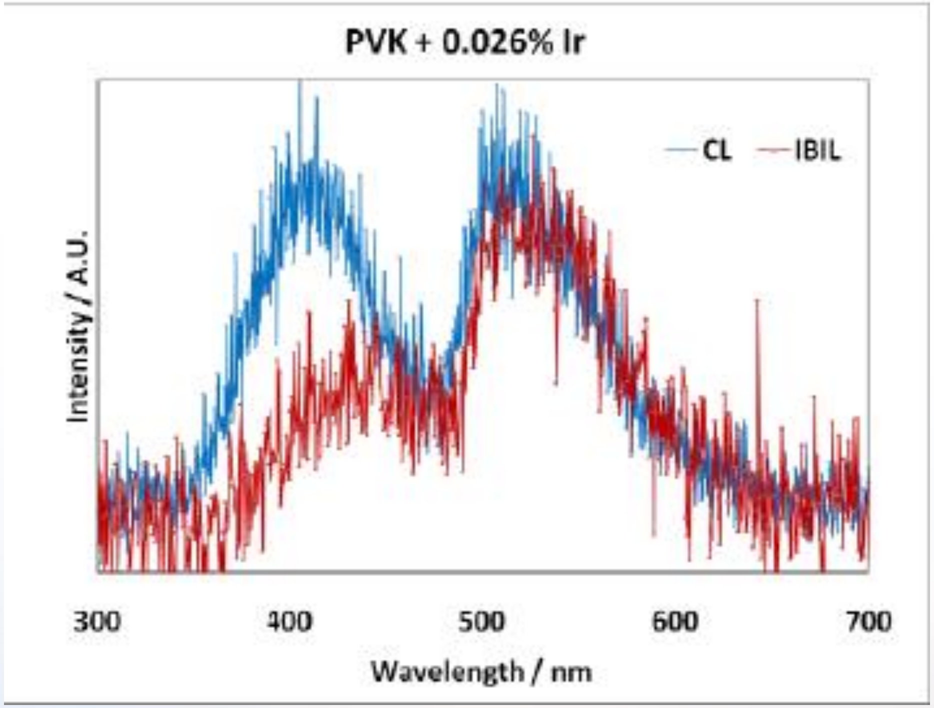
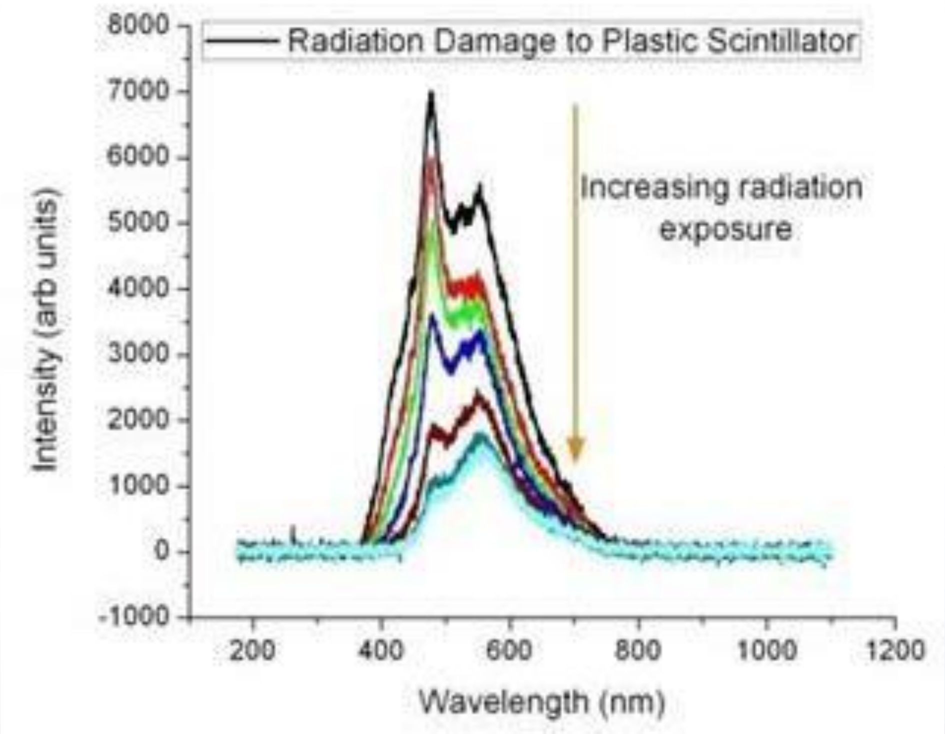
IBIL of MOFs

Collaborators: P. Feng, F.P. Doty, & J. Villone

Metal-organic frameworks demonstrate spectral discrimination with IBIL/CL





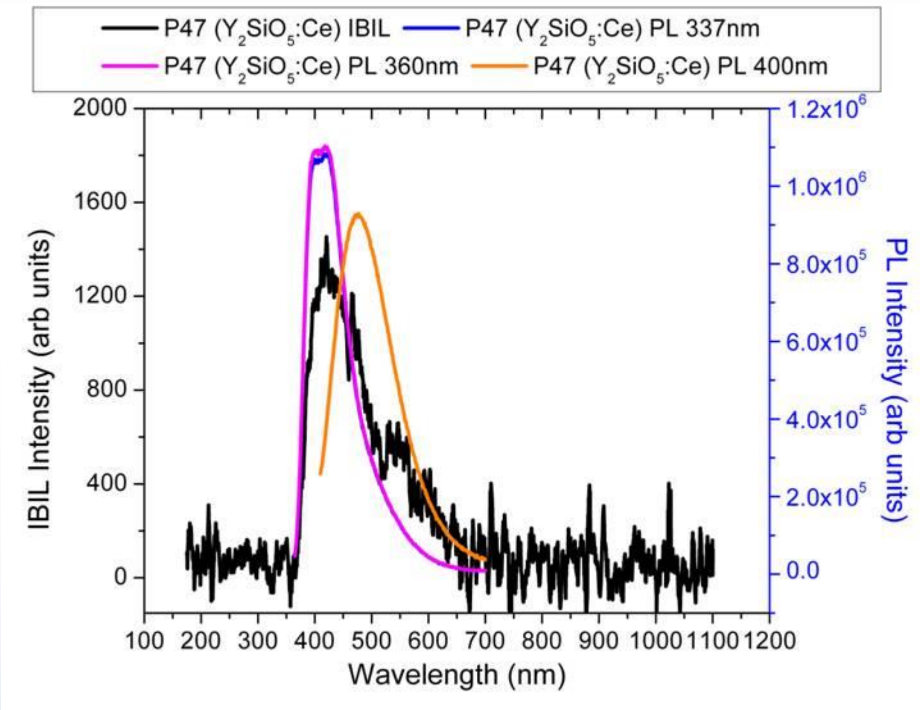
- Spectral discrimination
- CL simulates response to gamma rays
- IBIL simulates response to neutrons

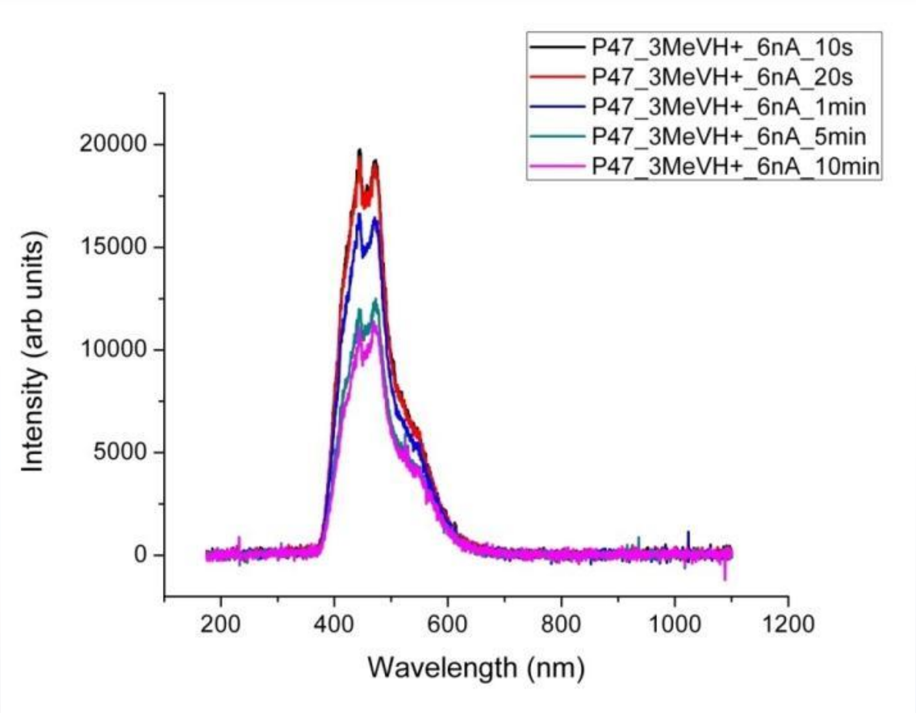
- PL and IBIL of MOF demonstrating spectral discrimination
- IBIL decay of MOFs with irradiation – changes observed in relative peak height

IBIL of Oxides

Collaborators: J. Villone & G. Vizkelethy

P47 phosphor studied for potential in radiation effects microscopy





- P47 is effective phosphor – PL and IBIL similar
- Peak emission dependent on excitation wavelength

- Degradation in optical properties also observed in P47
- Oxides demonstrate improved radiation tolerance compared to organic scintillators

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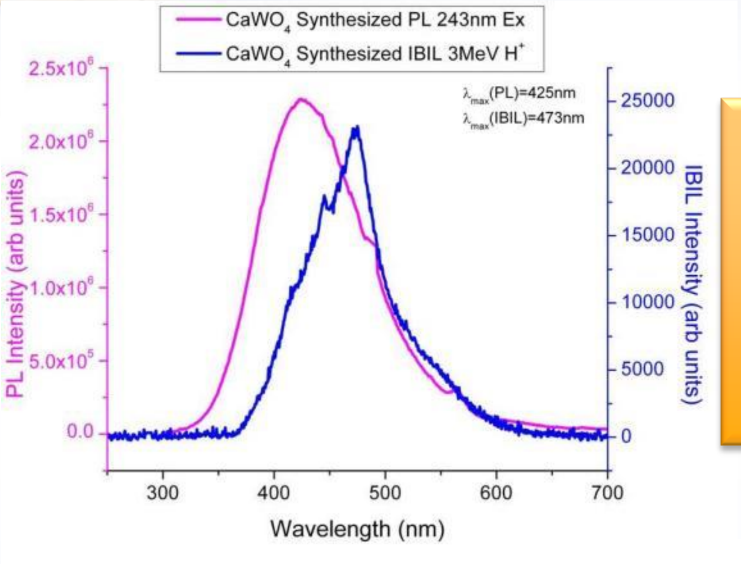
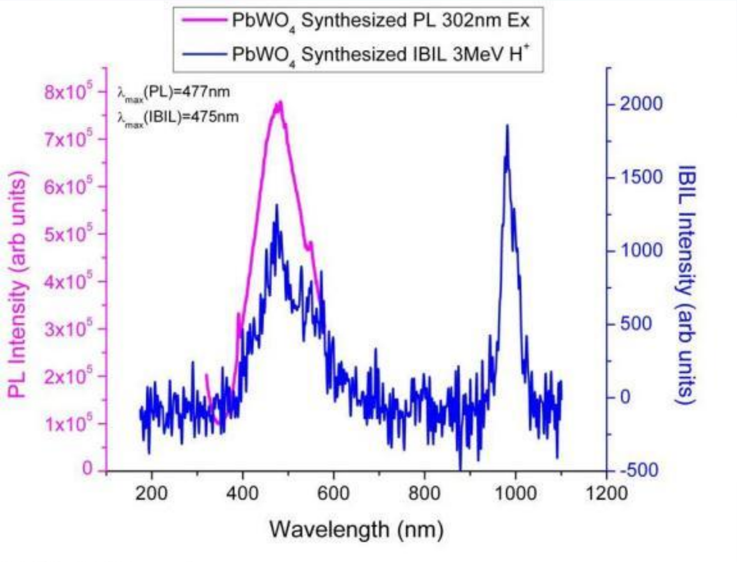
IBIL of Nanoscintillators

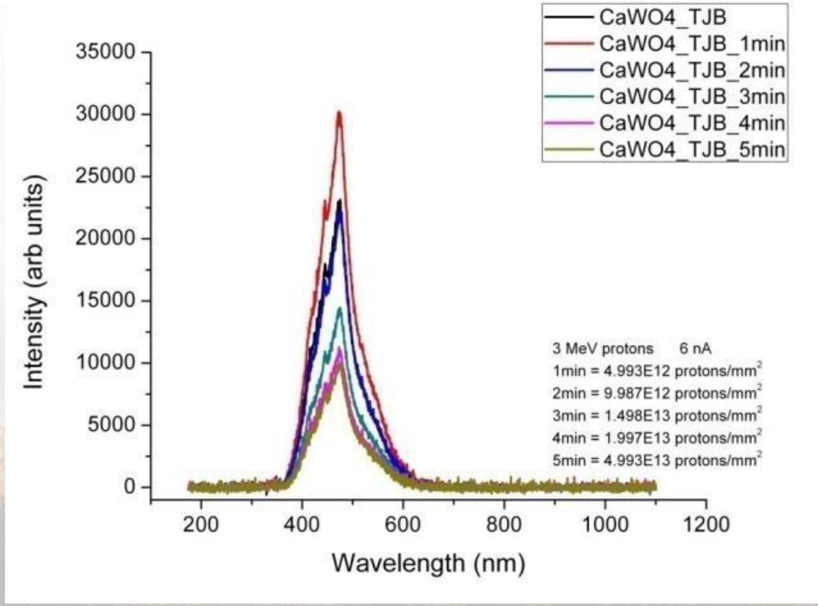
Collaborators: B.A. Hernandez-Sanchez, S.M. Hoppe, T.J. Boyle, J. Villone & P. Yang



Luminescence with proton excitation demonstrates different properties than with UV excitation



Crucial to study materials with various excitation mechanisms to fully understand luminescent properties



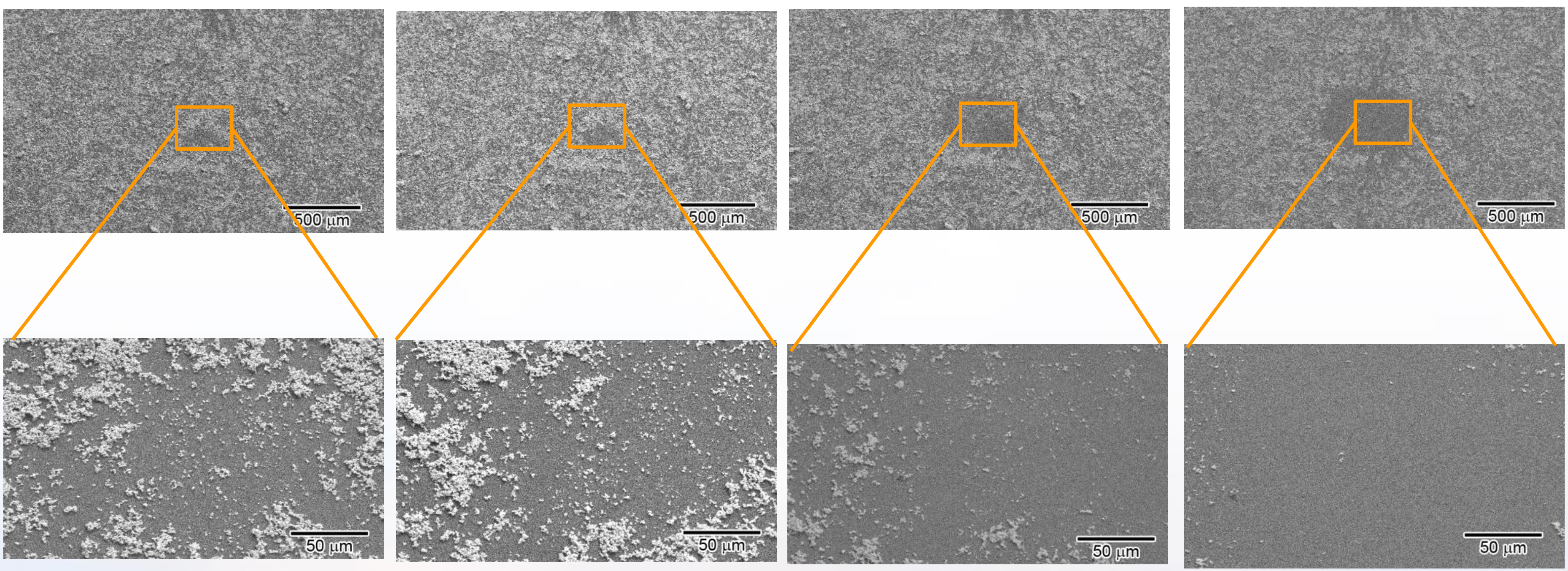
Most materials demonstrate a degradation in optical properties with irradiation – want to understand fundamental mechanism

Nearly *in situ* SEM of Nanoscintillators

Collaborators: B.A. Hernandez-Sanchez, S.M. Hoppe, T.J. Boyle, J. Villone & P. Yang

Nearly In-situ SEM Ion Irradiation of Nanoscintillators

As deposited Nanoparticles | **3 MeV H^+ 7 nA 1 sec** | **3 MeV H^+ 7 nA 5 sec** | **3 MeV H^+ 7 nA 30 sec**



- **Drop cast films of $PbWO_4$ nanoscintillators irradiated with 3 MeV H^+ beam, then imaged with SEM**
- **Material being ablated off of the surface – need better technique to study microstructural changes**

Can we understand how the microstructure is affected by irradiation?
How does the influences effect optical properties?

Benefits & Limitations of *in situ* TEM

Benefits

1. **Real-time nanoscale resolution observations of microstructural dynamics**

Limitations

1. **Predominantly limited to microstructural characterization**
   - Some work in thermal, optical, and mechanical properties
2. **Limited to electron transparent films**
   - Can often prefer surface mechanisms to bulk mechanisms
   - Local stresses state in the sample is difficult to predict
3. **Electron beam effects**
   - Radiolysis and Knock-on Damage
4. **Vacuum conditions**
   - 10^{-7} Torr limits gas and liquid experiments feasibility
5. **Local probing**
   - Portions of the world study is small

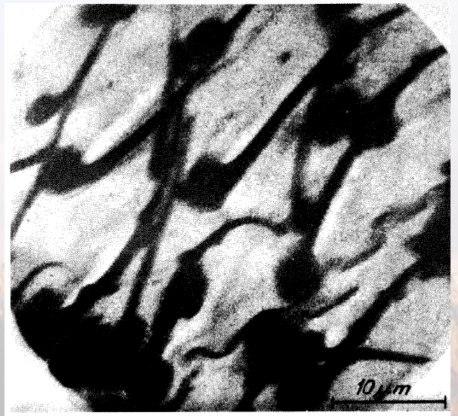


Fig. 6: Wing surface of the house fly.
(First internal photography, U = 60 kV, M_o = 2200)
(Driest, E., and Müller, H.O.: Z. Wiss. Mikroskopie 52, 53-57 (1935)

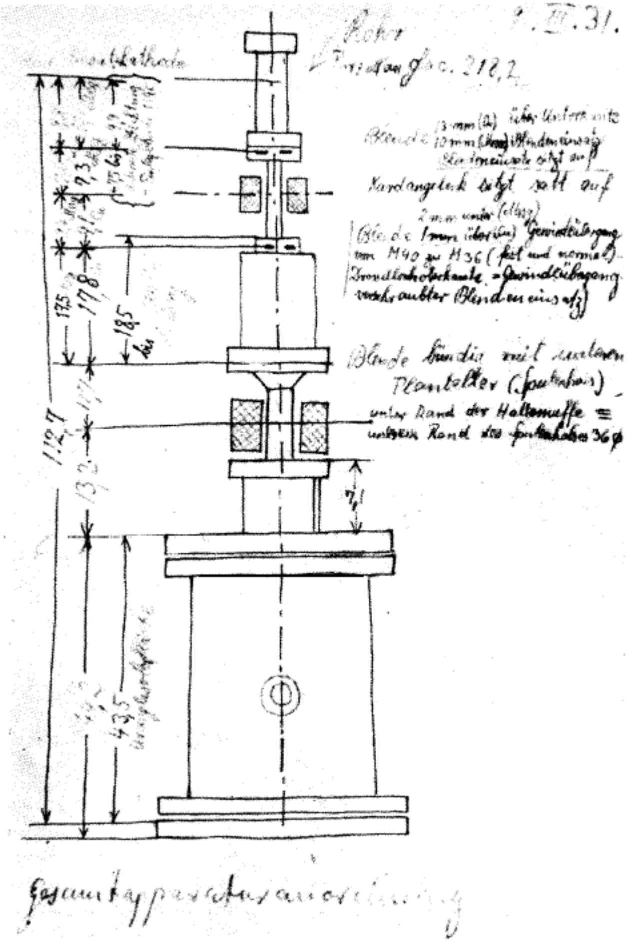
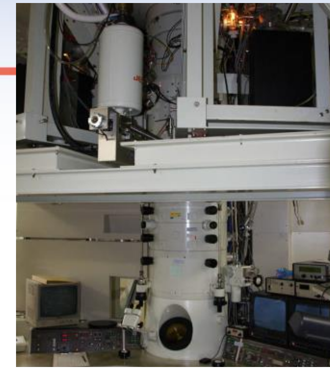


Fig. 2: Sketch by the author (9 March 1931) of the cathode ray tube for testing one-stage and two-stage electron-optical imaging by means of two magnetic electron lenses (electron microscope) [8].

E. Ruska Nobel lecture (1986)

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

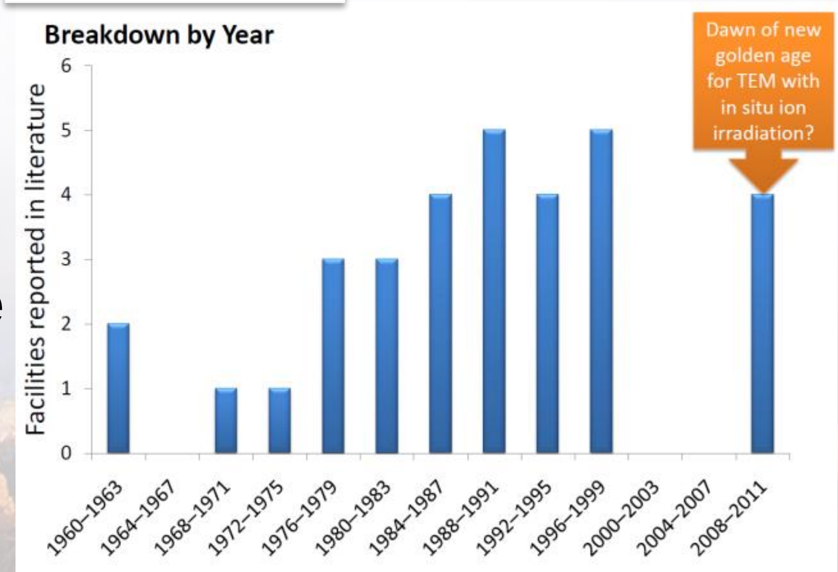
1968
First TEM beamline combination by Thackery, Nelson, and Sansom at AERE Harwell, UK

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

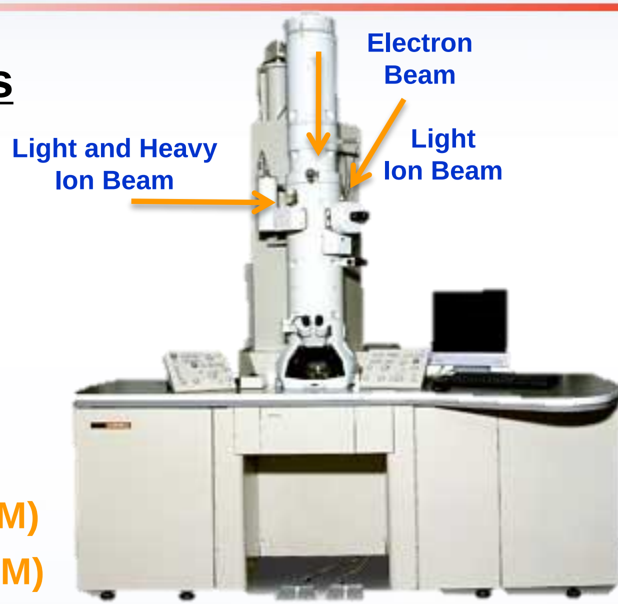
In situ Ion Irradiation TEM Facility

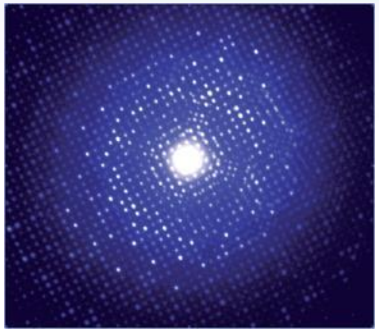
Proposed Capabilities

- 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

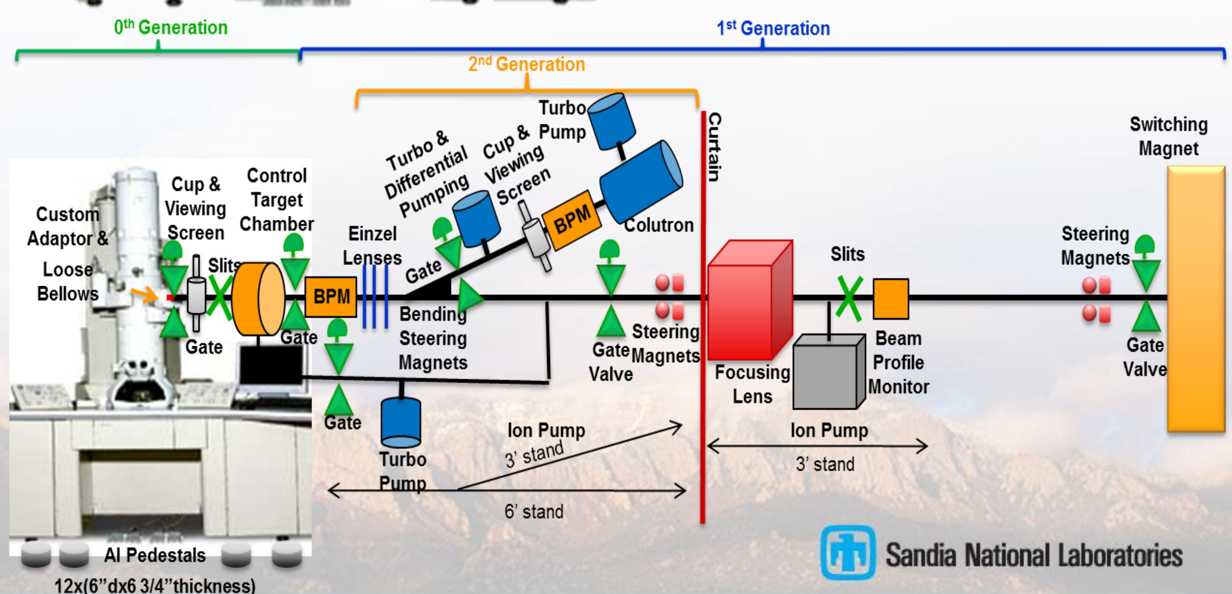
- *In situ* vapor phase stage
- *In situ* liquid mixing stage
- *In situ* heating
- Tomography stage (2x)
- *In situ* cooling stage
- *In situ* electrical bias stage
- *In situ* straining stage





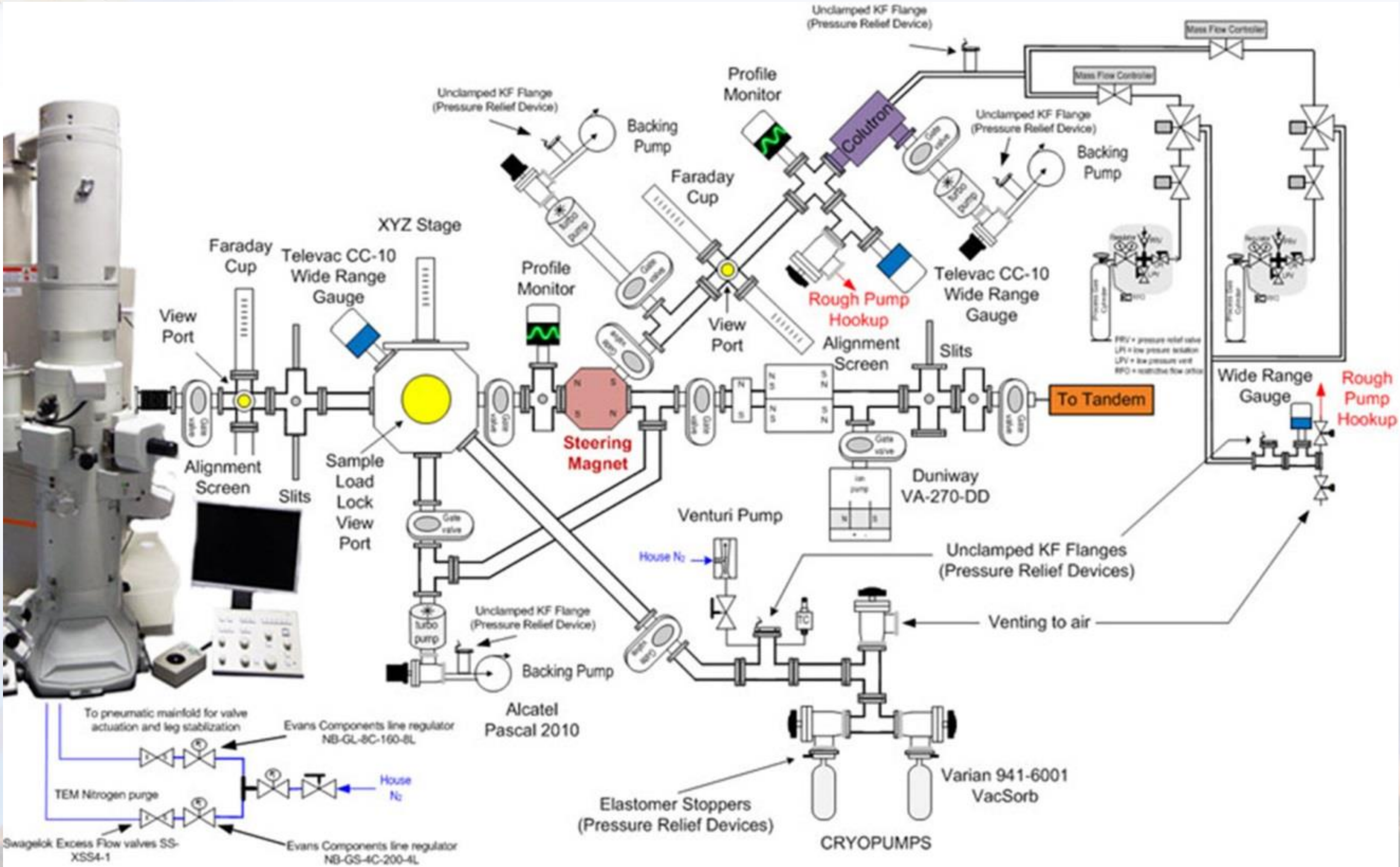
TVIPS

Hummingbird



Schematic of the *In situ* TEM Beamline

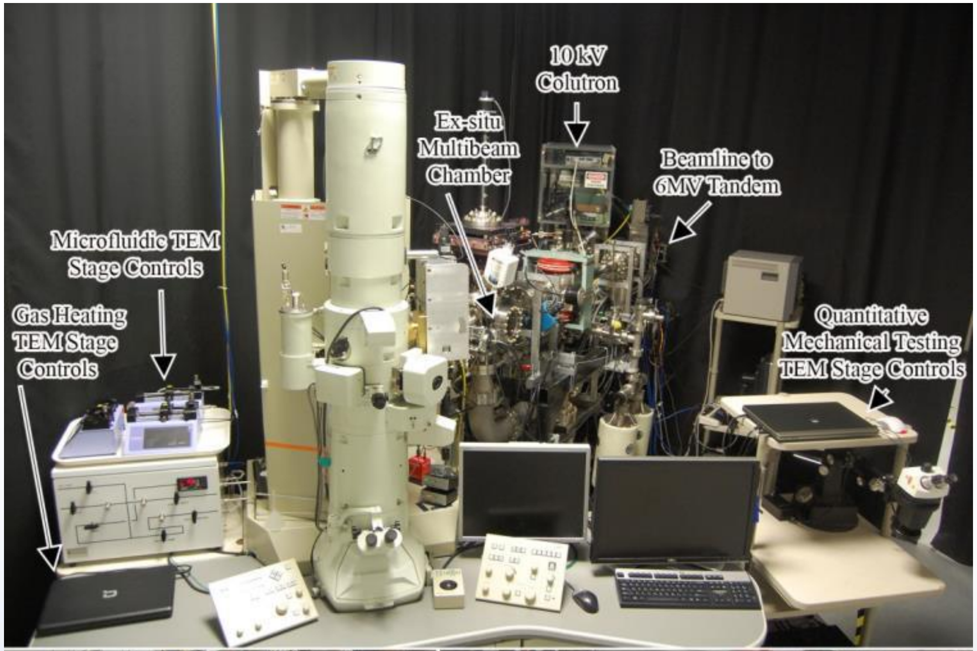
Collaborators: M.T. Marshall J.A. Scott, & D.L. Buller



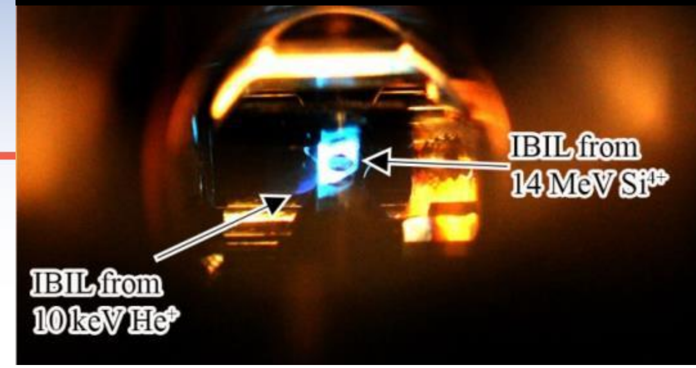
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem

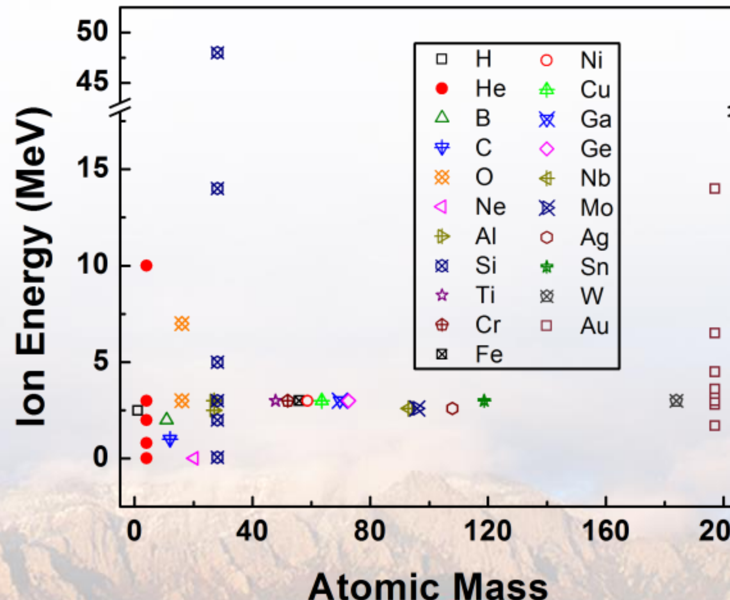


IBIL from a quartz stage inside the TEM



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

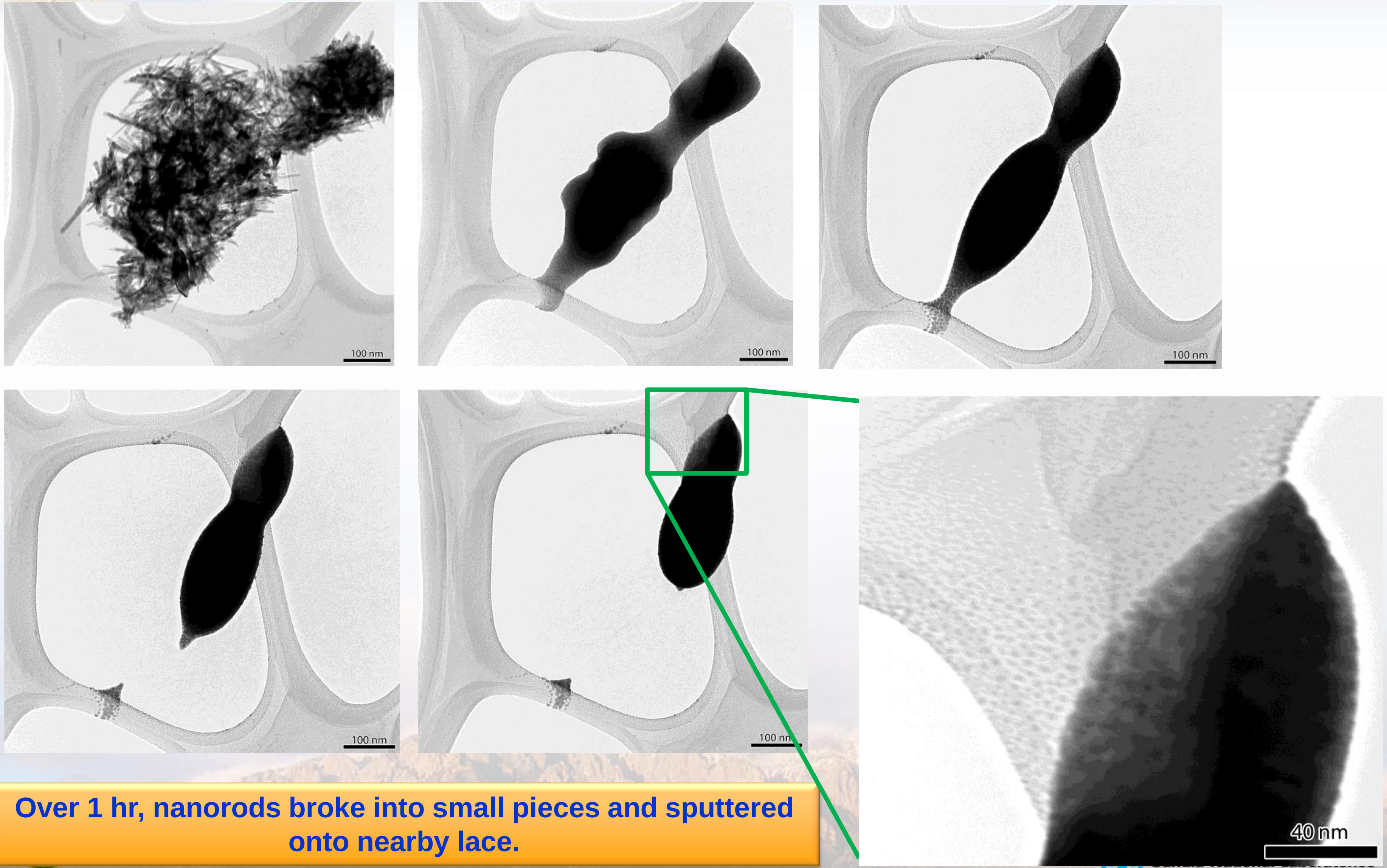
Ion species & energy introduced into the TEM



Sandia National Laboratories

$CdWO_4$ Irradiated with 50 nA of 3 MeV Cu^{3+}

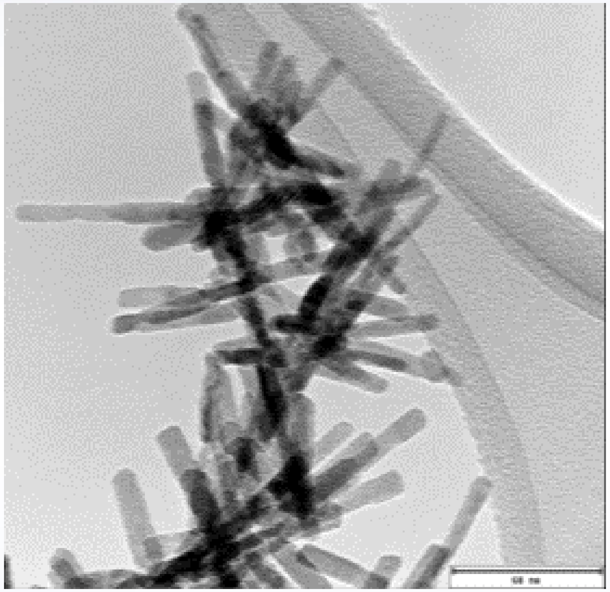
Collaborators: S.M. Hoppe & B.A. Hernandez-Sanchez



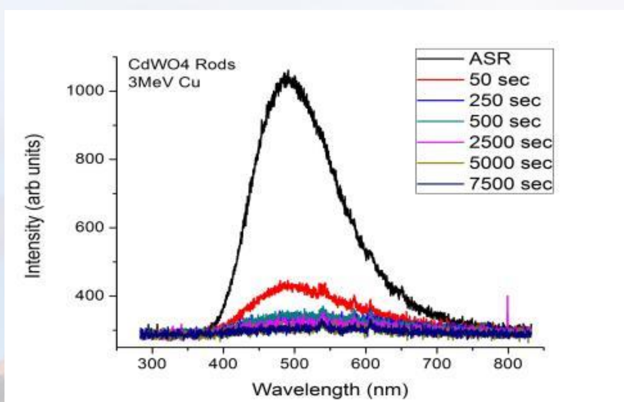
Over 1 hr, nanorods broke into small pieces and sputtered onto nearby lace.

Radiation Tolerance is Needed in Advanced Scintillators for Non-proliferation Applications

Contributors: S.M. Hoppe, B.A. Hernandez-Sanchez, T. Boyle



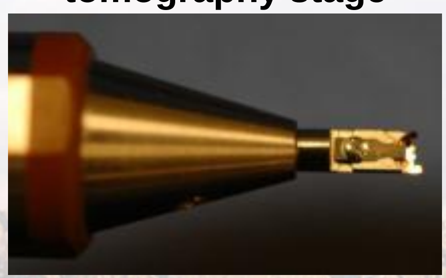
High-Z nanoparticles are promising, but are radiation sensitive



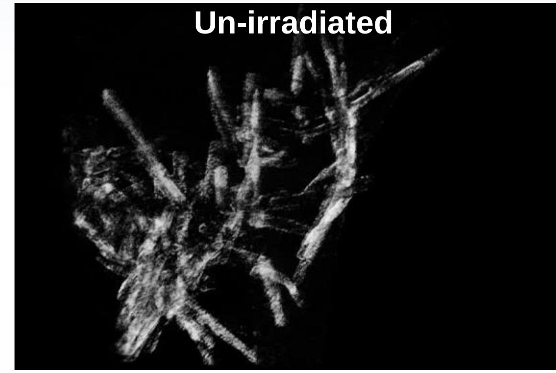
In situ Ion Irradiation TEM (I[3]TEM)

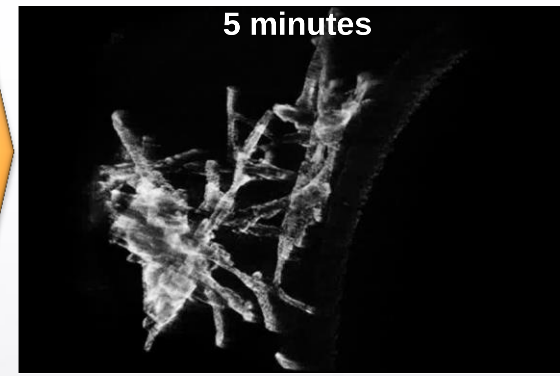
+

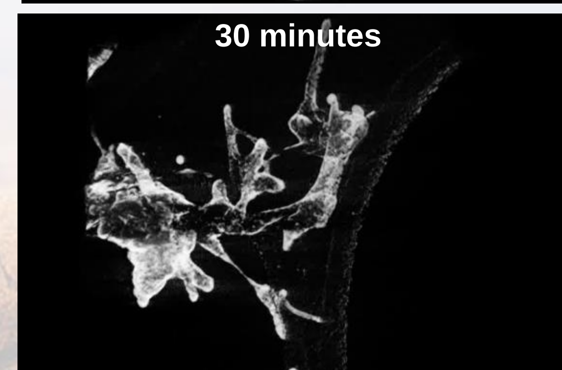
Hummingbird tomography stage



Tomography of Irradiated $CdWO_4$:
3 MeV Cu^{3+} at ~30 nA







Cumulative Effects of Ion Irradiation as a Function of Ion Energy and Au Particle Size

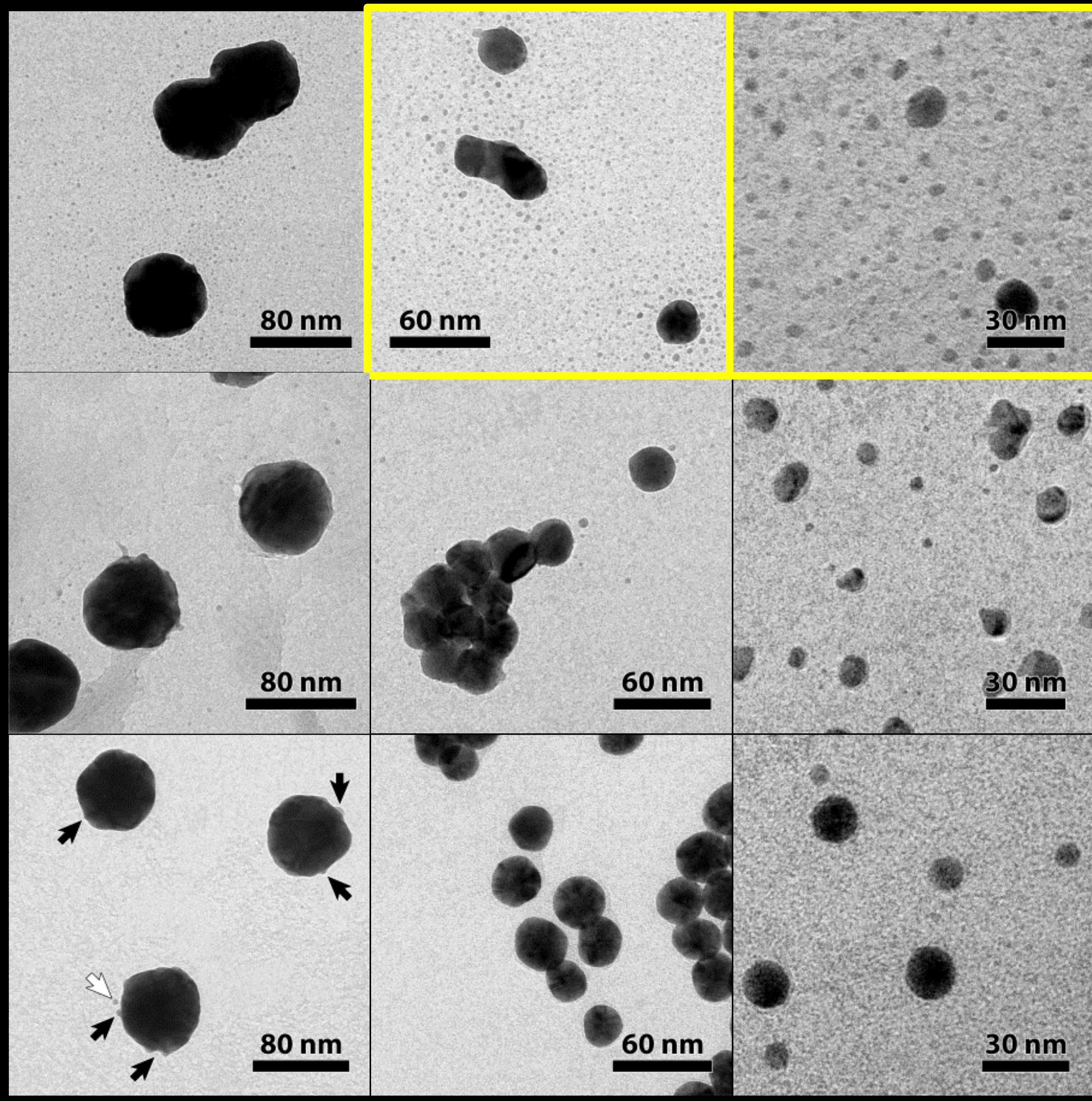
Collaborator: D.C. Bufford

60 nm | 20 nm | 5 nm

46 keV Au^{1-}
3.4×10^{14} /cm^2

2.8 MeV Au^{4+}
4×10^{13} /cm^2

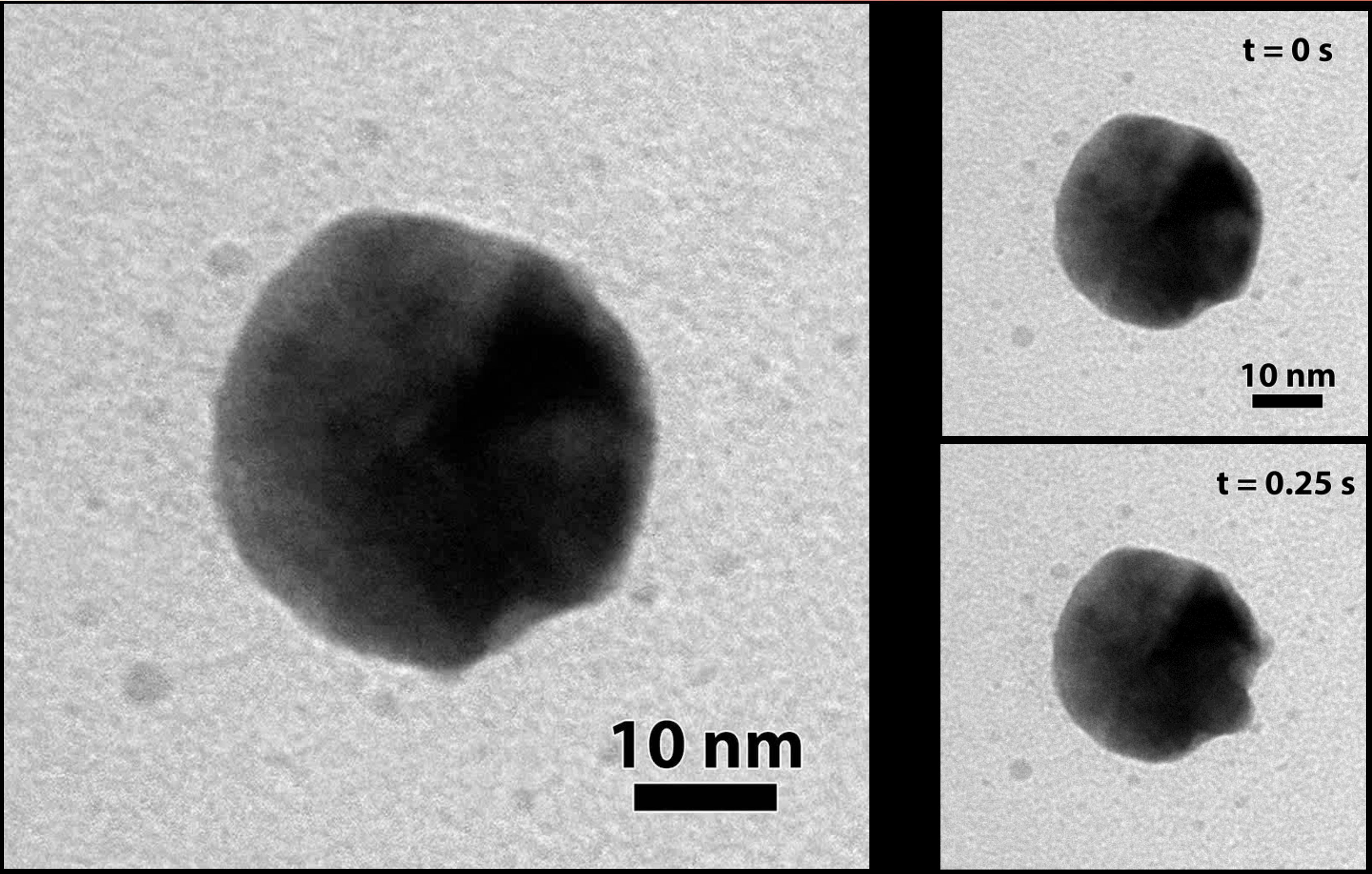
10 MeV Au^{8+}
1.3×10^{12} /cm^2



Particle and ion energy dictate the ratio of sputtering, particle motion, particle agglomeration, and other active mechanisms

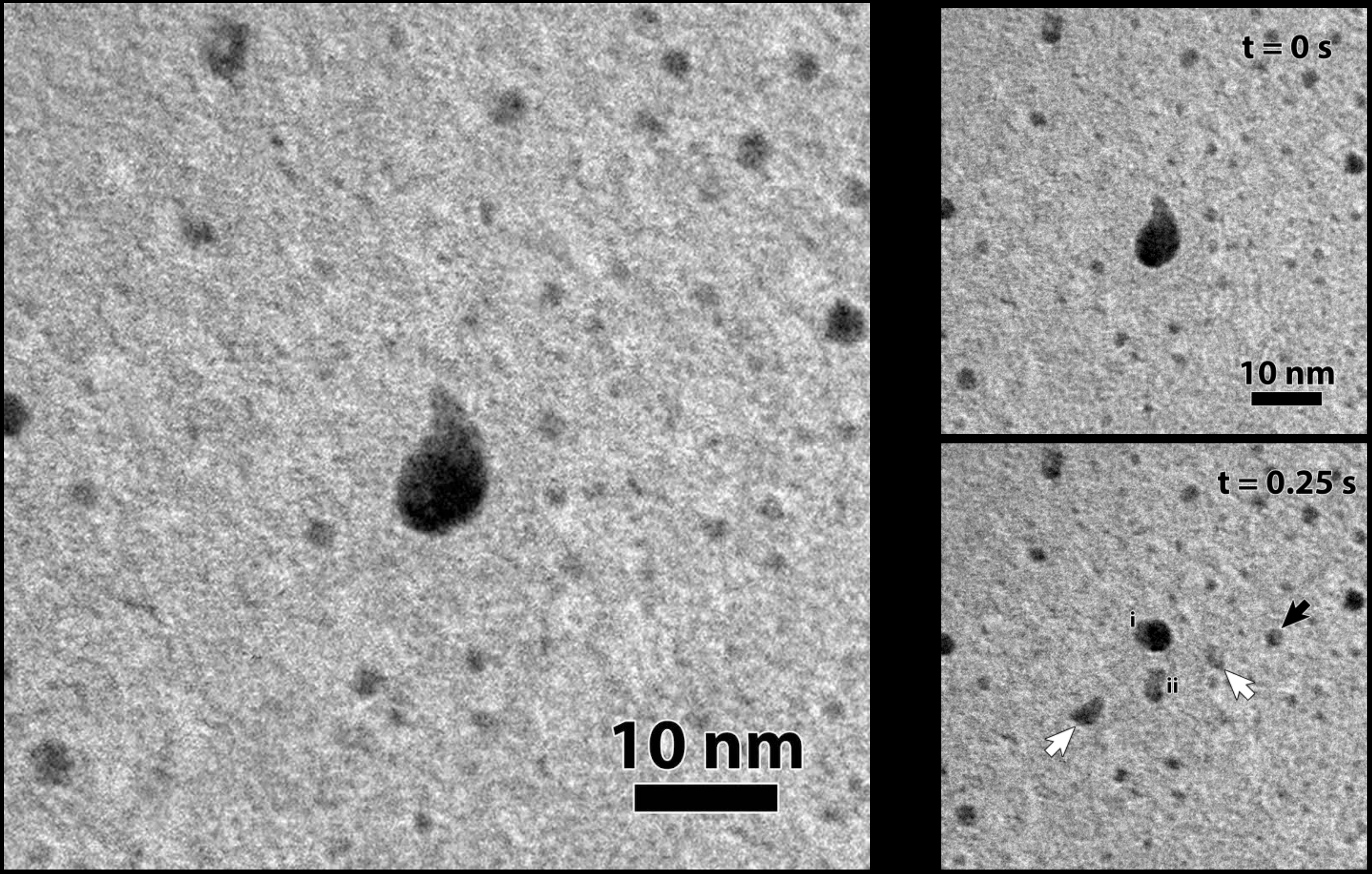
Single Ion Effects with 46 keV Au^{1-} ions: 20 nm

Collaborator: D.C. Bufford



Single Ion Effects with 46 keV Au^{1-} ions: 5 nm

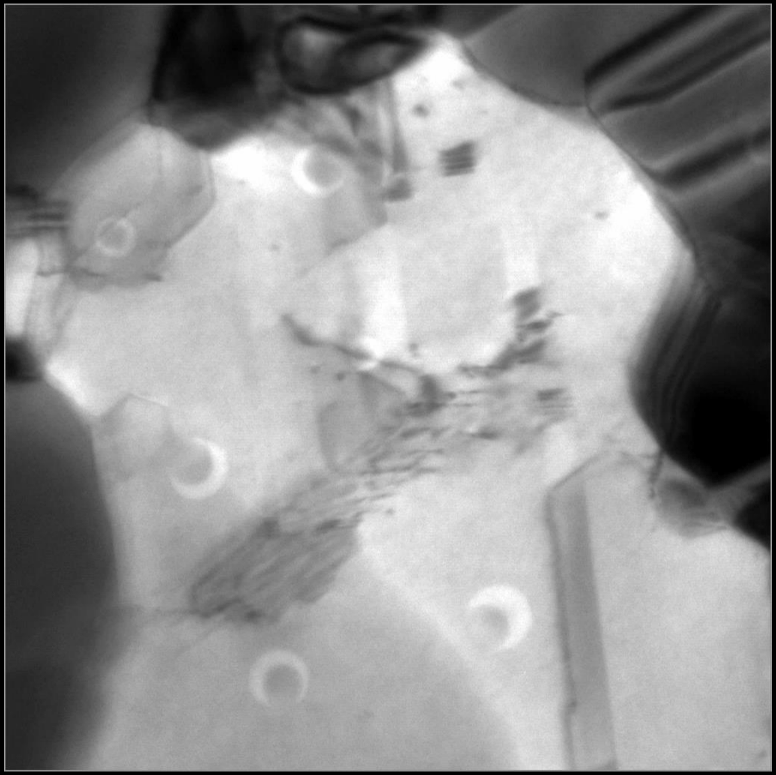
Collaborator: D.C. Bufford



Single Ion Strikes

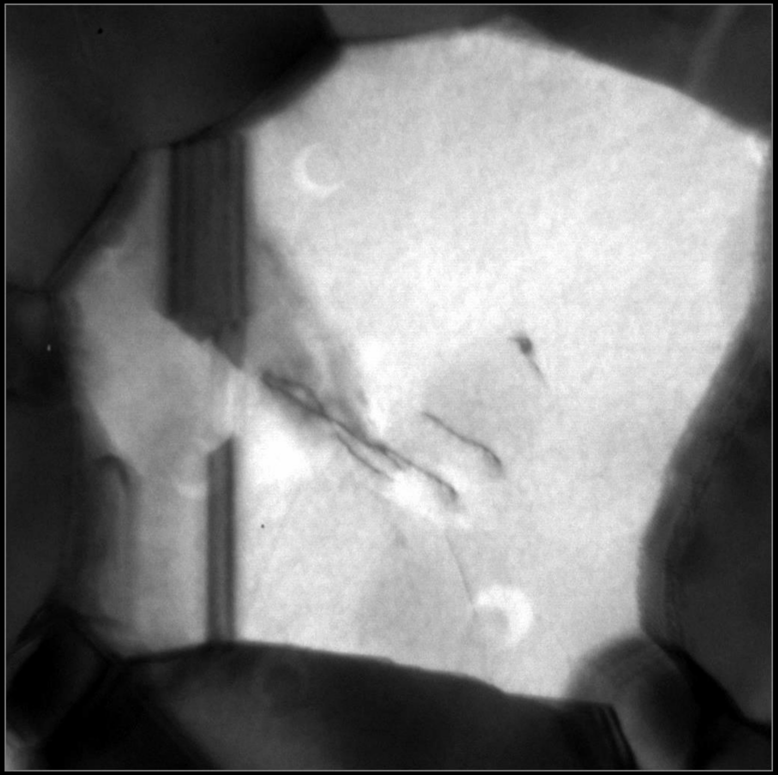
Collaborators: C. Chisholm & A. Minor

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



VS

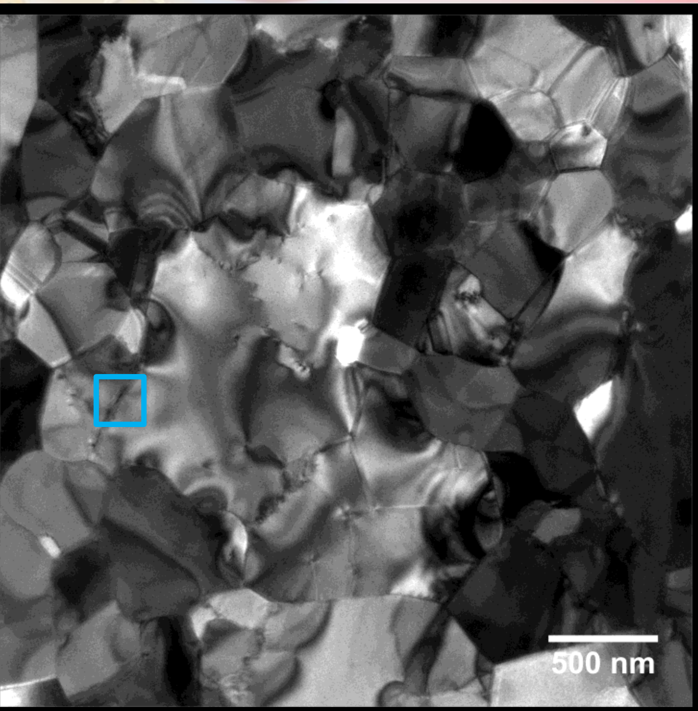
6.7 x 10^7 ions/cm^2/s



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

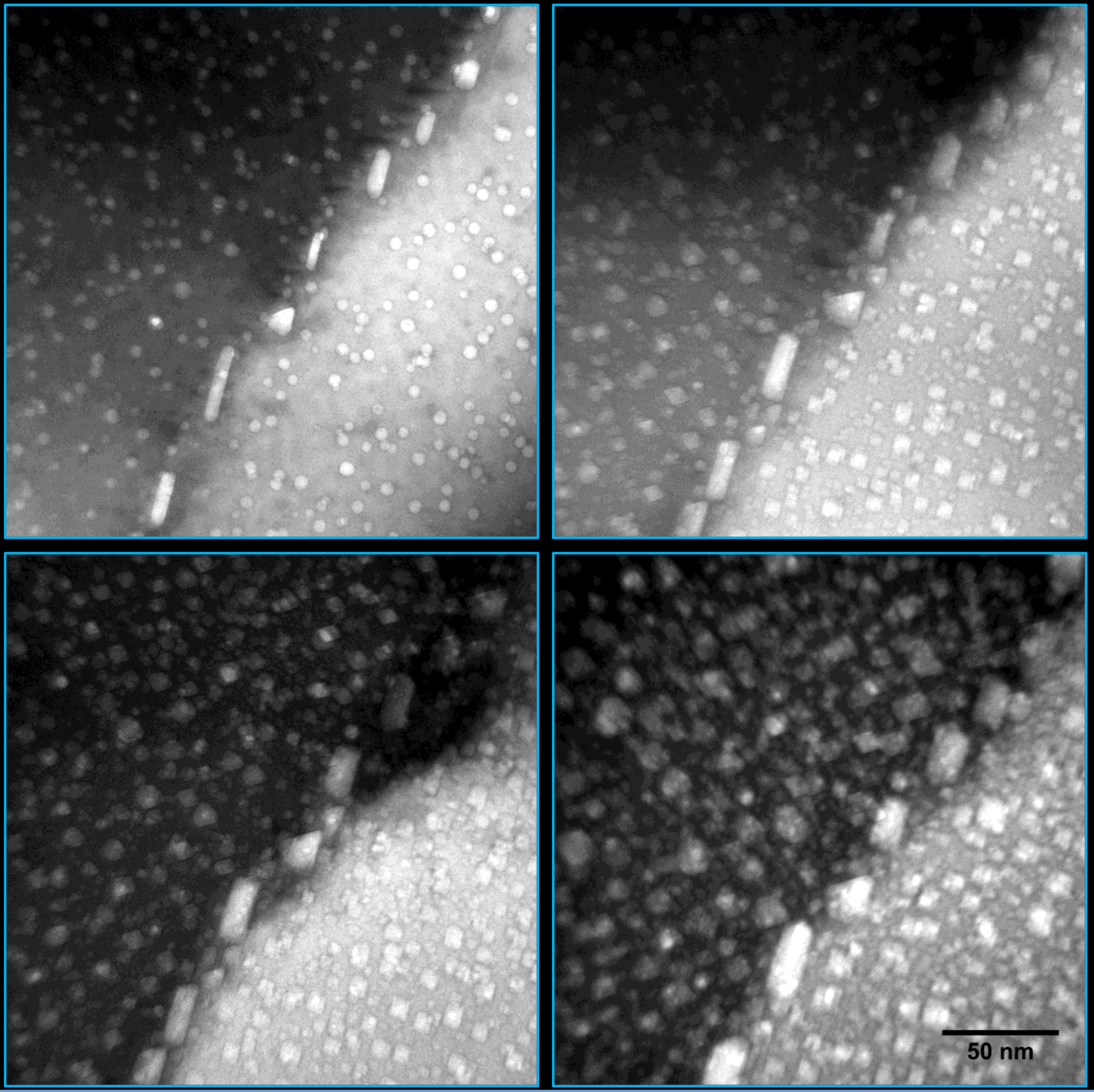
In situ Implantation

Collaborators: C. Chisholm & A. Minor

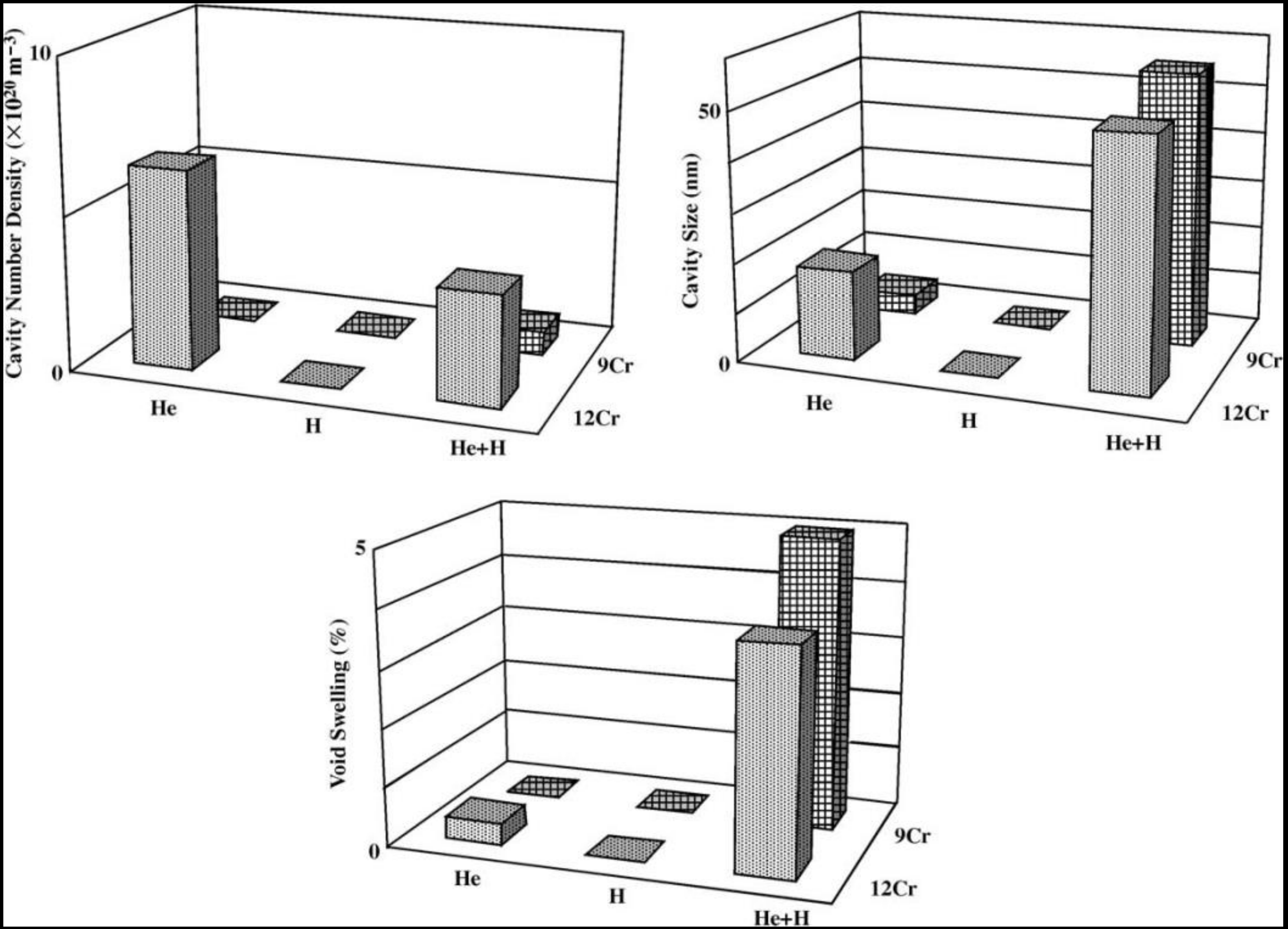


Gold thin-film implanted with 10keV He^{2+}

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

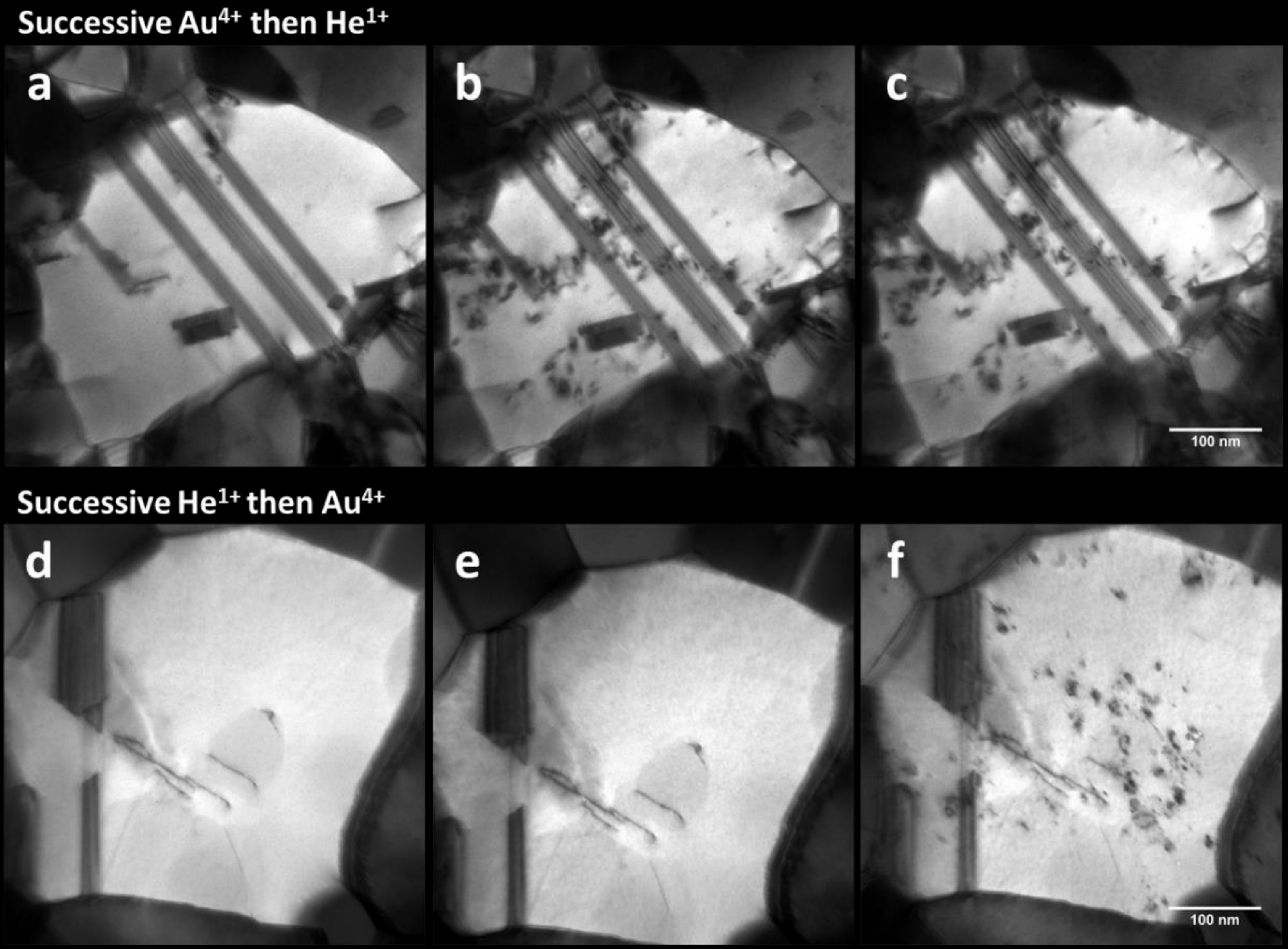
Coupling Effect

- 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

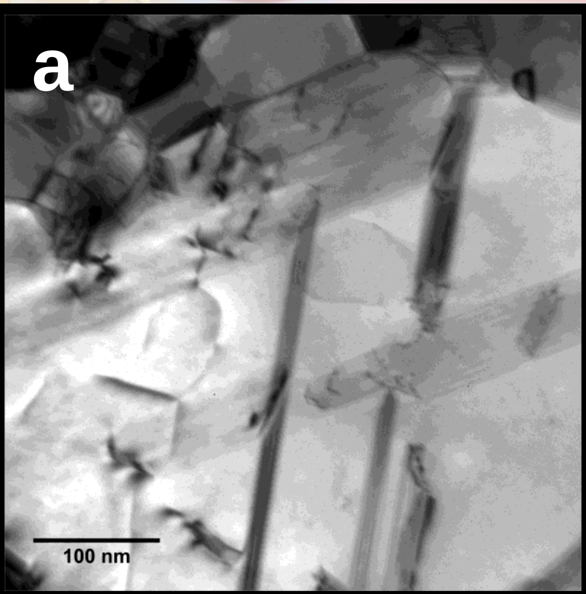
In situ Successive Implantation & Irradiation

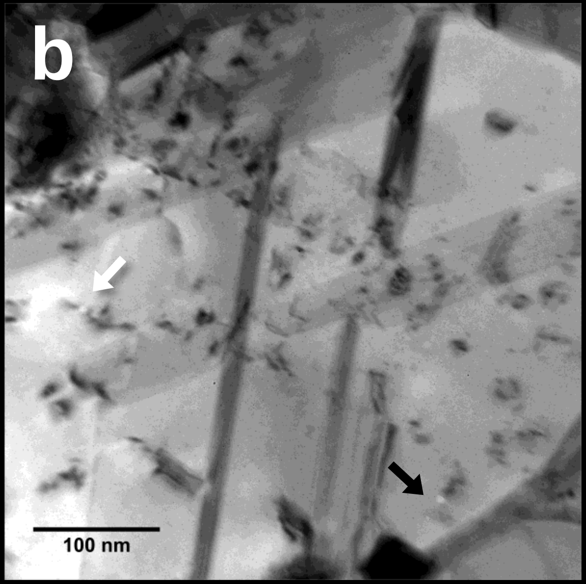
Collaborators: C. Chisholm & A. Minor



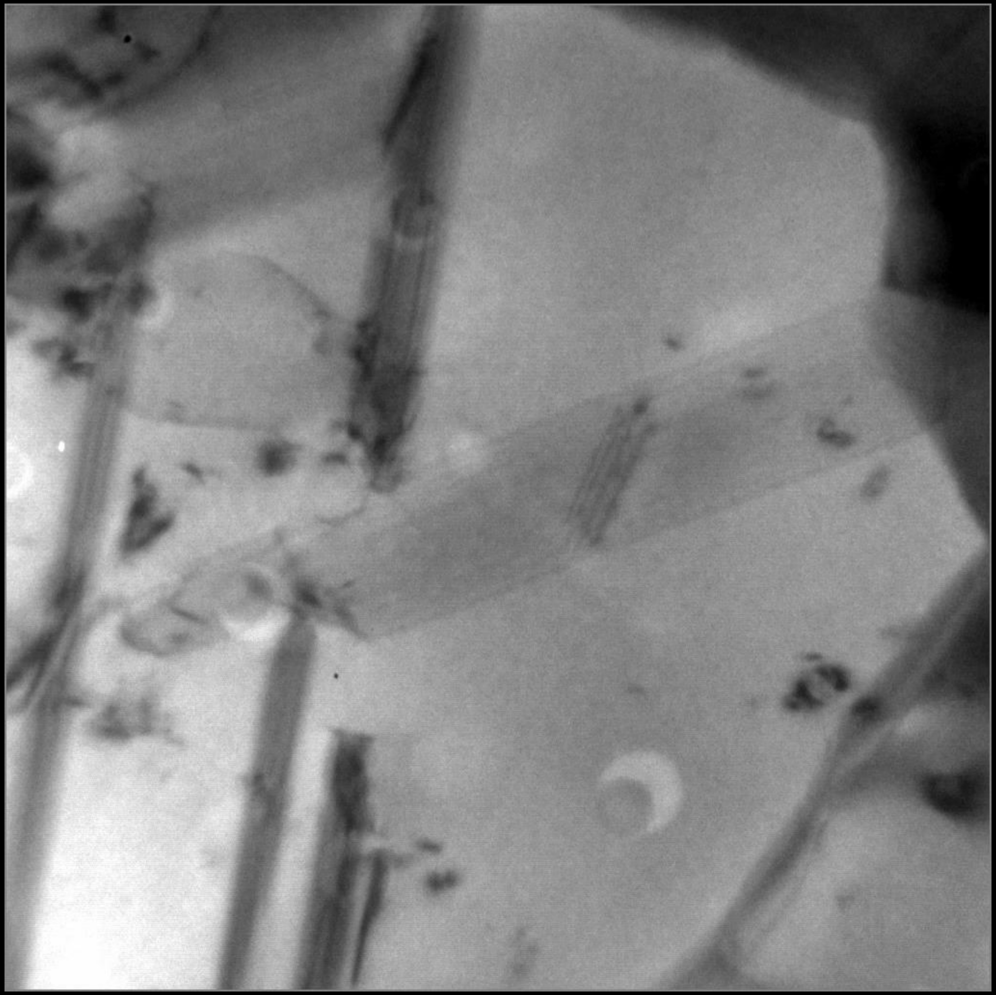
In situ Concurrent Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor





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



Single Ion Strikes During Concurrent Irradiation: Nucleation of Helium Cavities

Collaborators: C. Chisholm & A. Minor

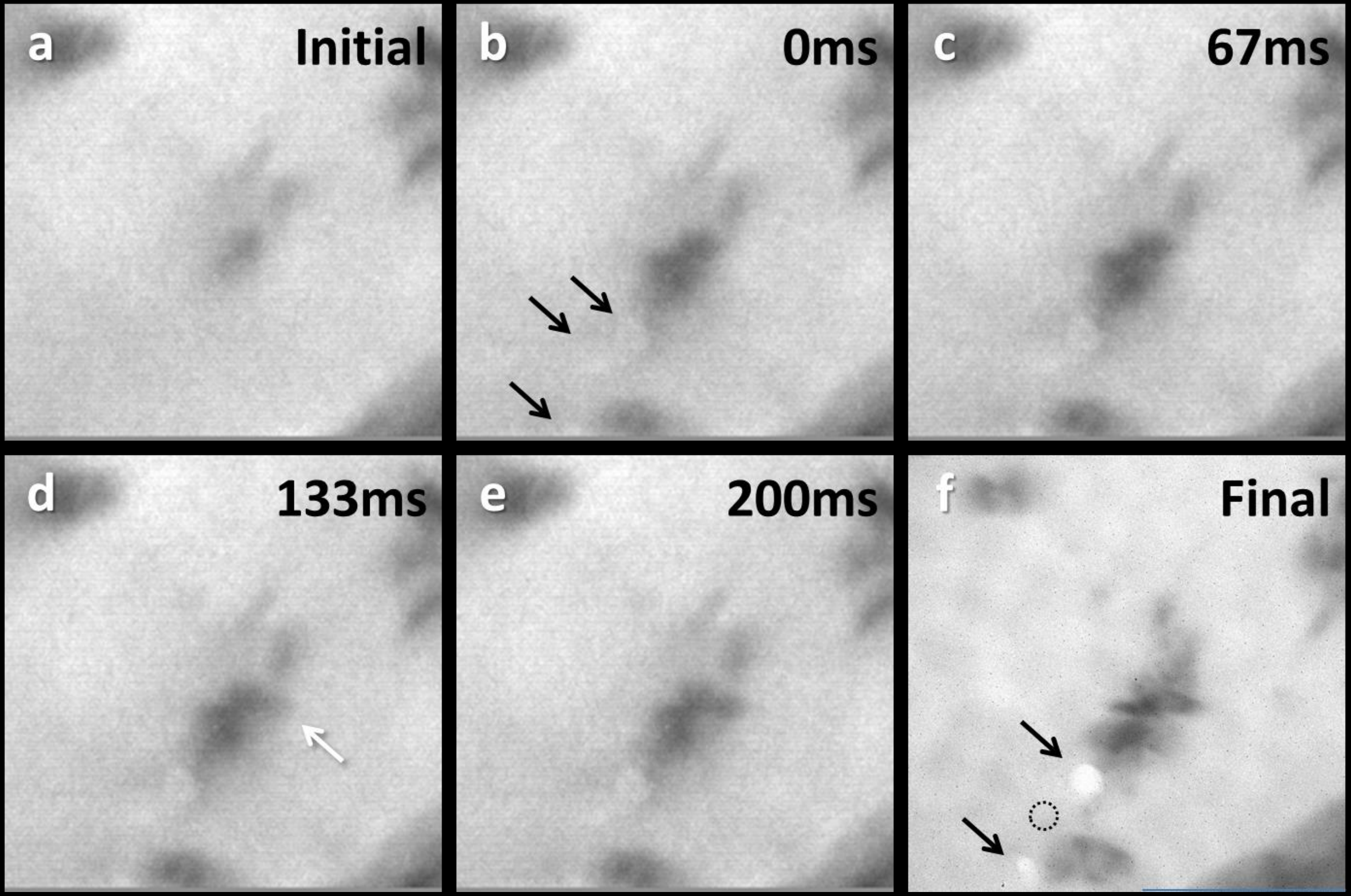
a) Initial microstructure

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

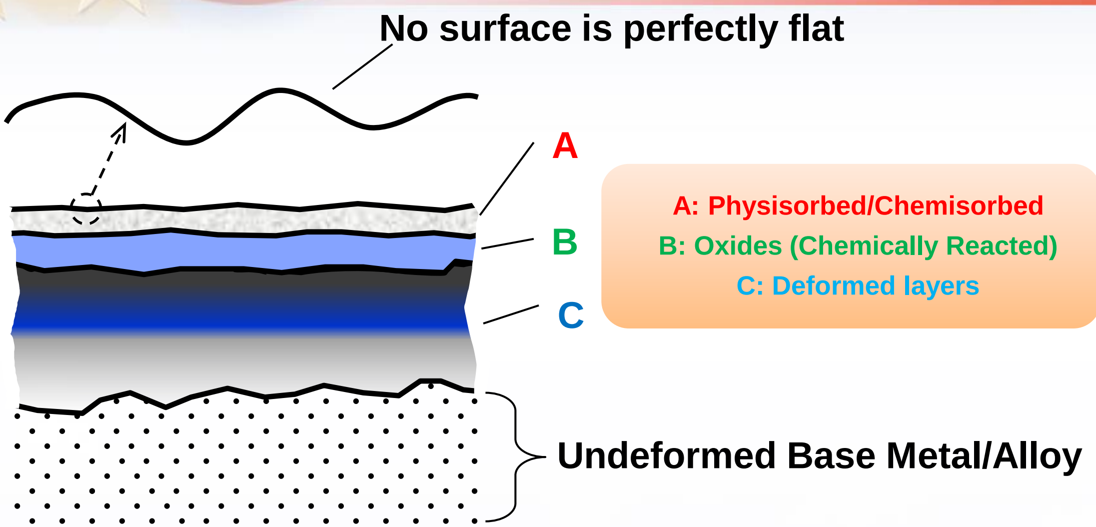
d) Cascade damage still evolving

e) Apparent stability

f) Final microstructure: Only two remaining cavities



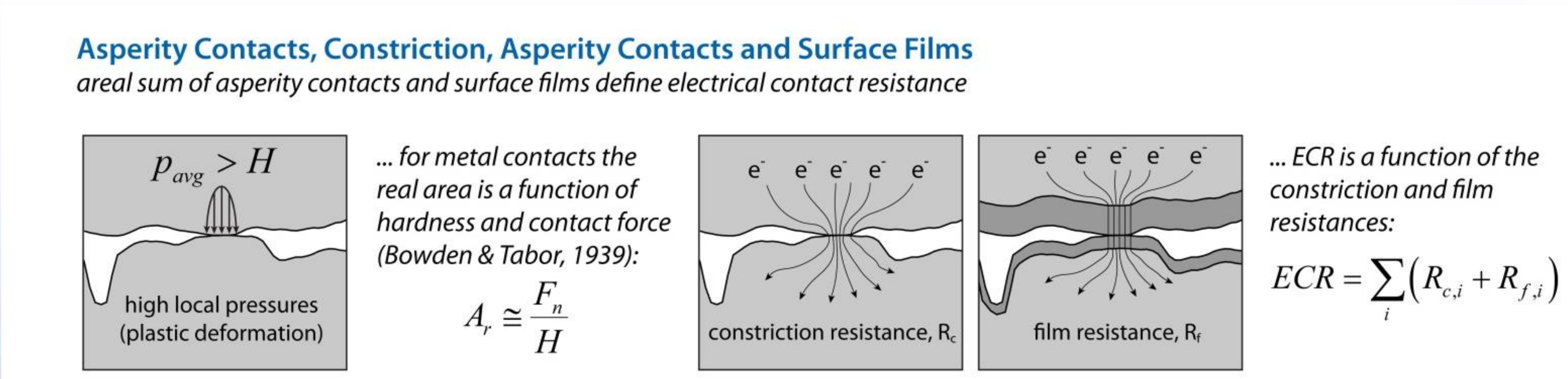
Nature of Metallic Surfaces



Real area of contact (A_r) to be minimized for low adhesion

(Low Adhesive Wear)

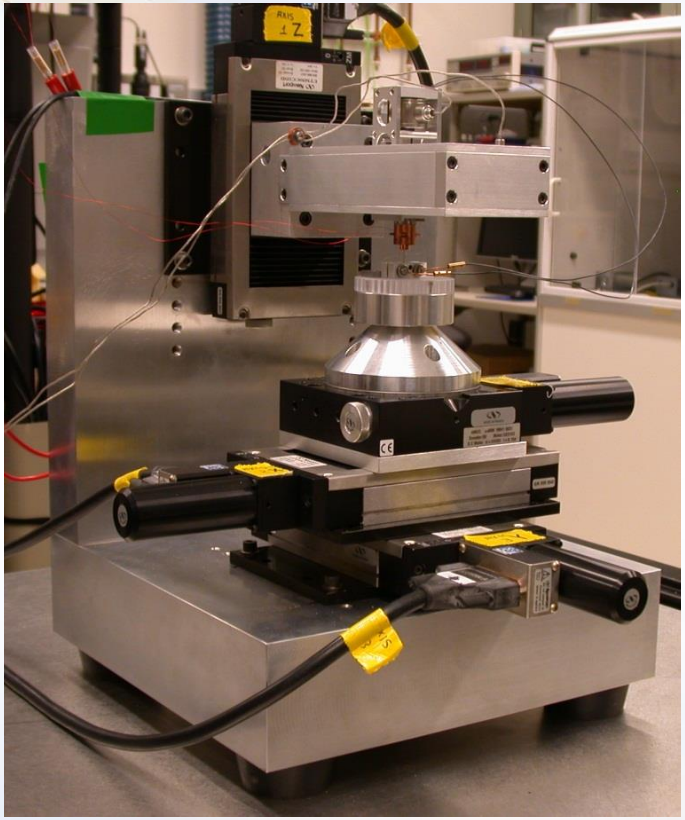
Or maximized for reduced electrical contact resistance (ECR)

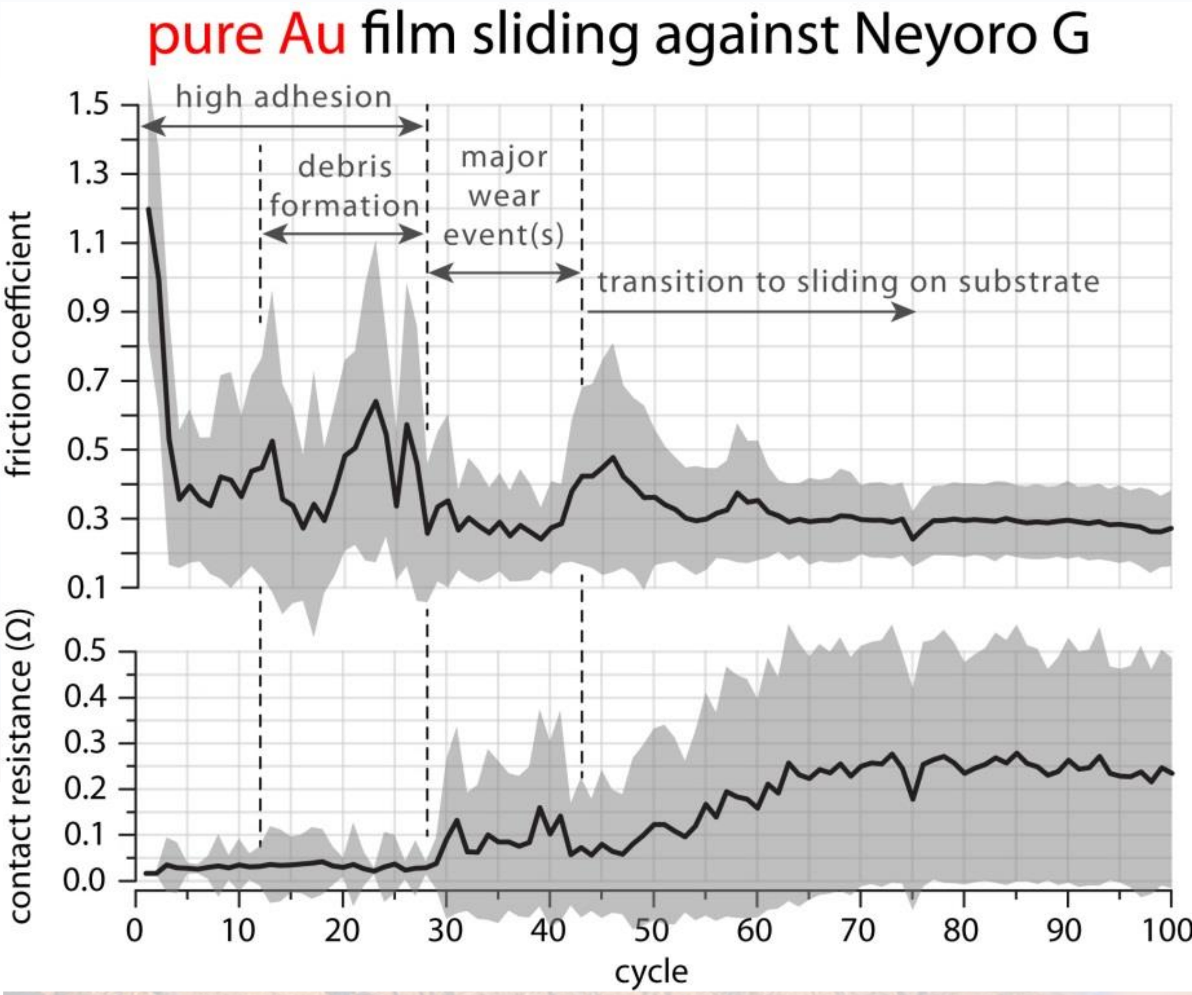


Archard, Journal of Applied Physics (1953) 24:981
R. Holm, Electrical Contacts Handbook (1958) Berlin: Springer-Verlag
Greenwood & Williamson, Proc. Royal Society (1966) A295:300
T.W. Scharf & S.V. Prasad, Journal of Material Science (2013) 48:511-531

ECR-Friction Behavior of Pure Au

Collaborators: J-E Mogonye & S.V. Prasad





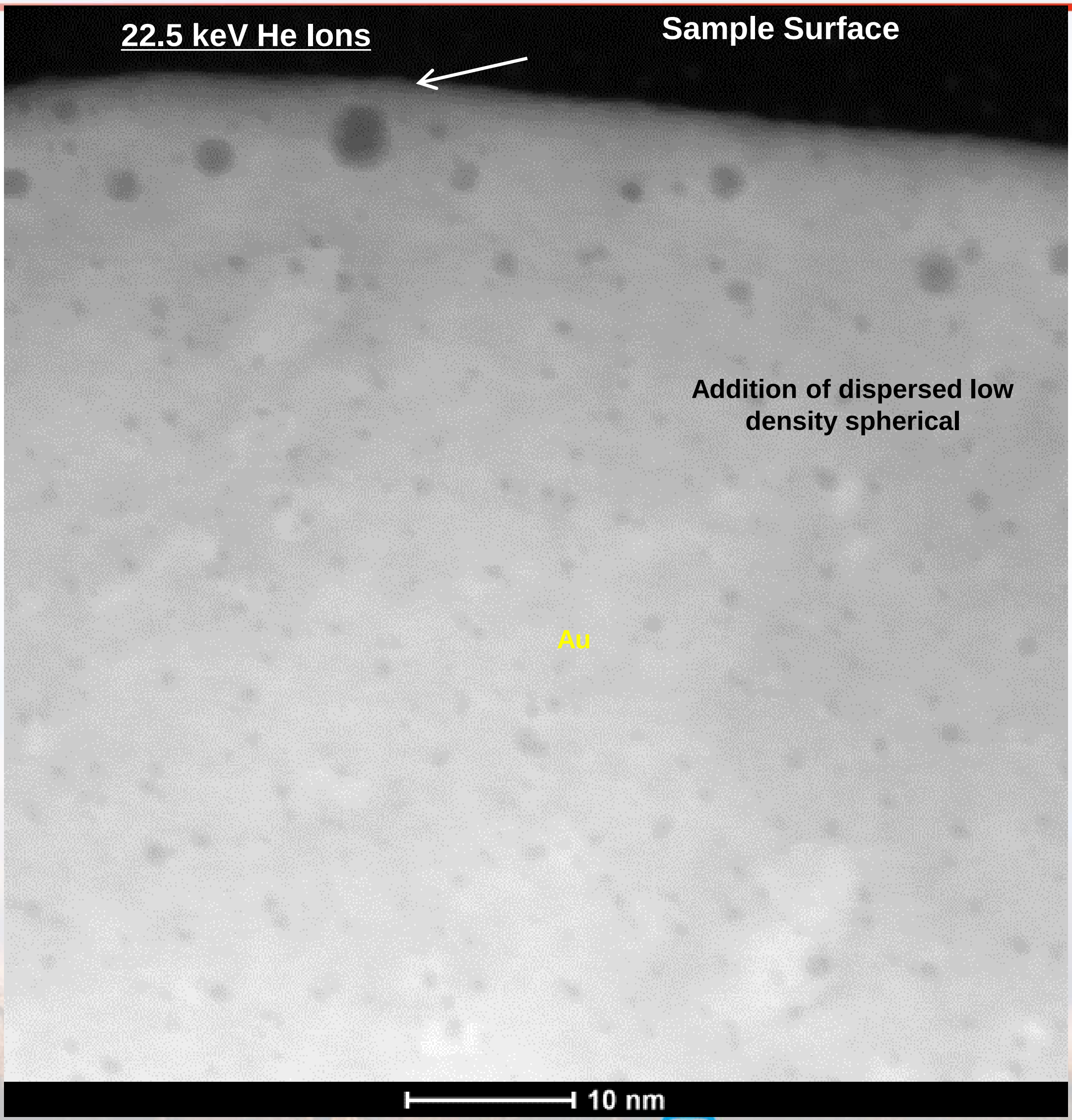
- Neyoro G (Au-Cu), $\frac{1}{16}$ in. radius hemispherical tip rider
- F_n = 100 mN (≈290 MPa contact stress)
- 100 Cycles @ v = 1 mm/s
- 1 – 2 mV bias to achieve approximately 100 mA
- Lab air environment at room temperature

Modeling and STEM of He Implantation

Collaborators: P. Kotula, J-E Mogonye & S.V. Prasad

- Simulations: SRIM 2008 (The Stopping and Range of Ions in Matter, J.F. Ziegler, M.D. Ziegler and J.P. Biersack)
  - Monte-Carlo simulation of kinematic interaction based on empirical data fitted functions
    - Input variables of target material include density, AMU, and thickness.
  - Input variables of ions include AMU, energy, and angle of incidence.
  - Assumes isotropic material, thus no consideration for channeling effects

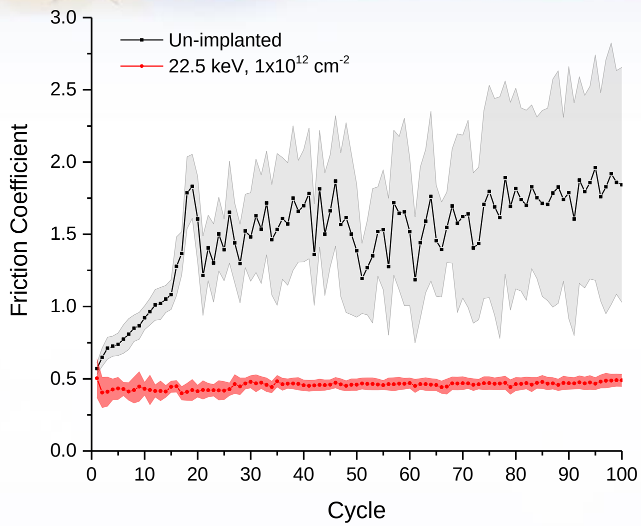
- AC-STEM used to observe the distribution of implanted bubbles
  - Bubble locations are in good agreement with SRIM ion range predictions

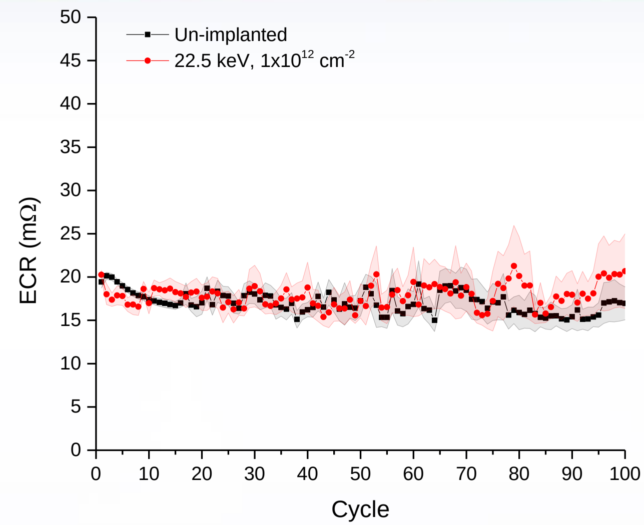


He implantation result in small dispersed spherical structures assumed to be He bubbles. Dispersion and depth can be tailored

ECR and Wear Measurements

Collaborators: J-E Mogonye & S.V. Prasad

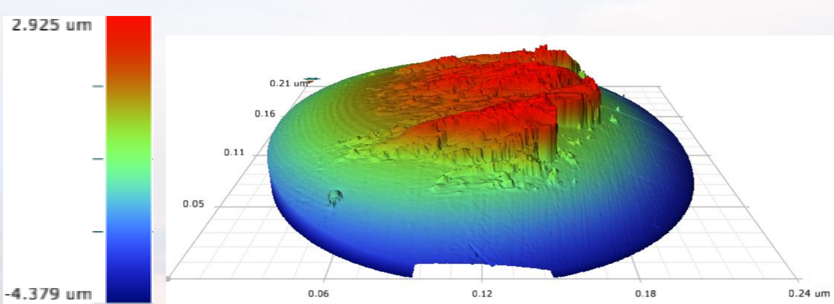




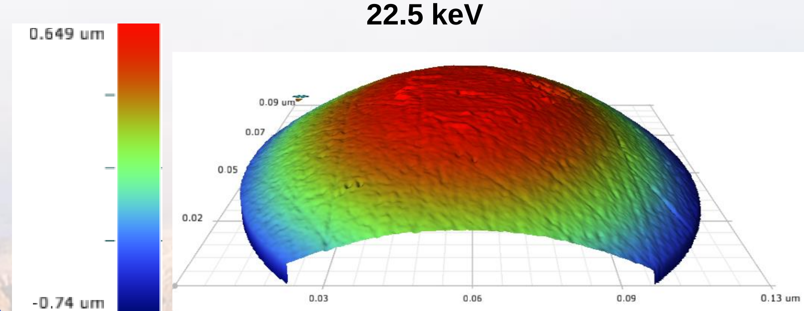
Friction is significantly reduced with ^{3}He implantation while maintaining ECR performance

Scanning white light interferometer topographical construction of riders after 100 cycles

Rider after 100 cycles against Un-implanted Au



Rider after 100 Cycles against Au implanted to 1E12 cm^{-2} @ 22.5 keV

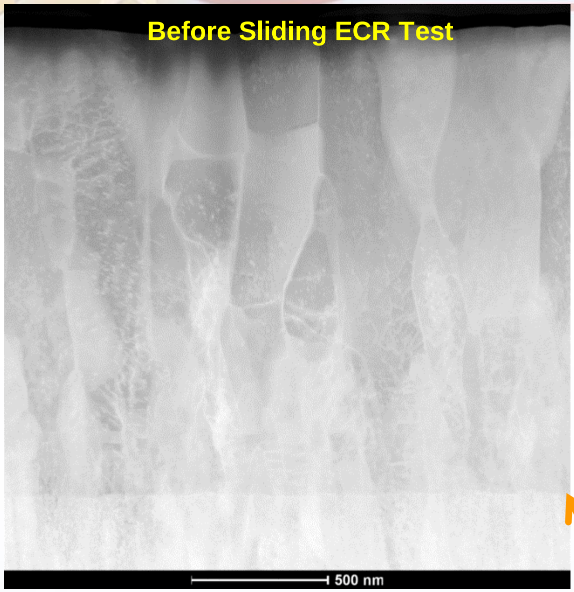


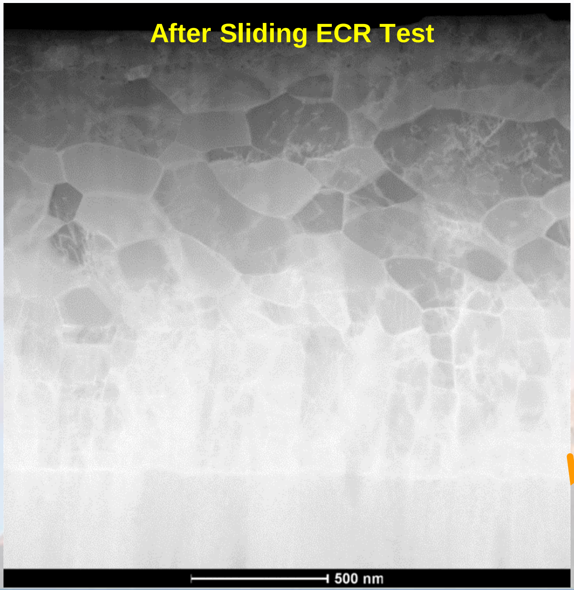
Wear is significantly reduced with minimal effect in ECR

Sandia National Laboratories

STEM Images of Sub-surfaces

Collaborators: J-E Mogonye & S.V. Prasad



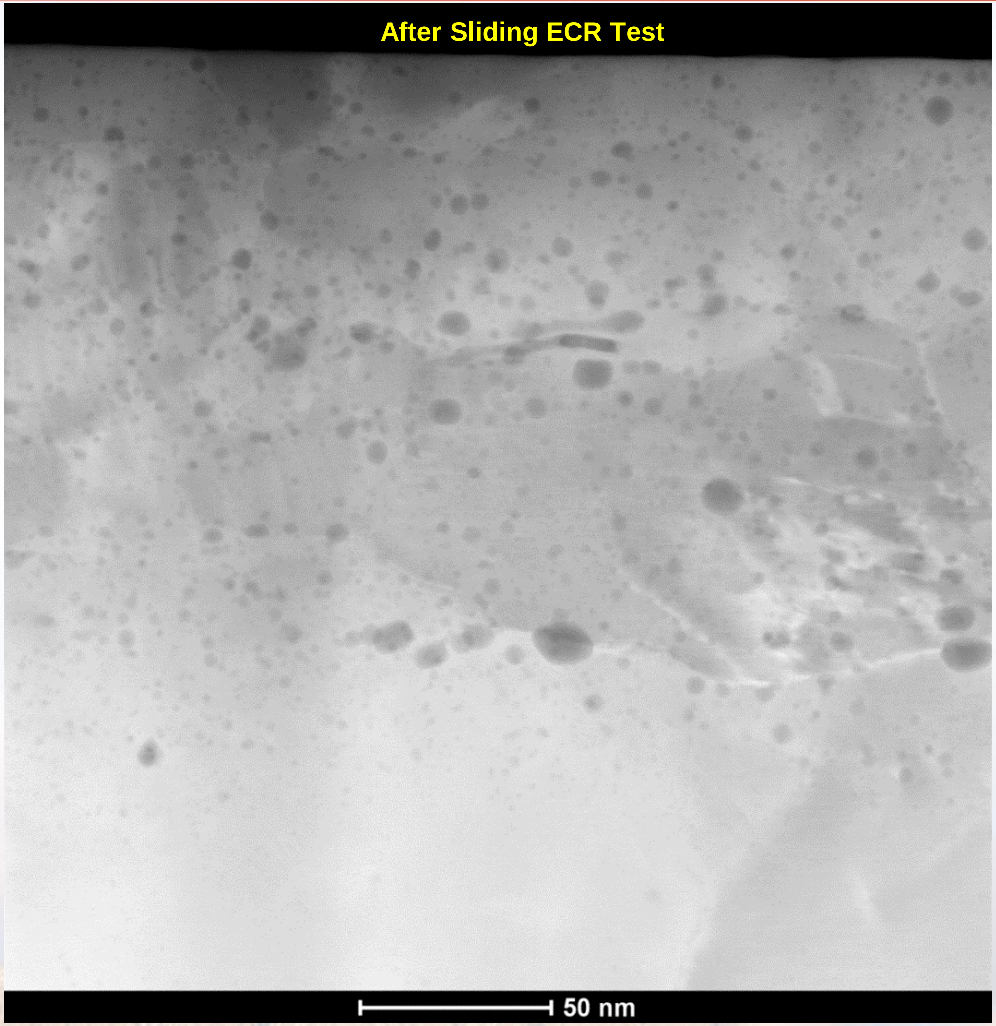


22.5 keV

1×10^{12} cm^{-2}

Recrystallization is observable after 100 cycles

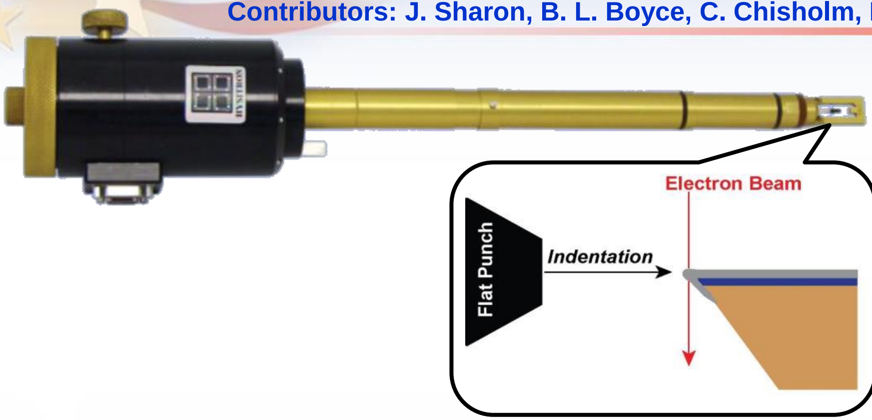
Au – Pt Interface

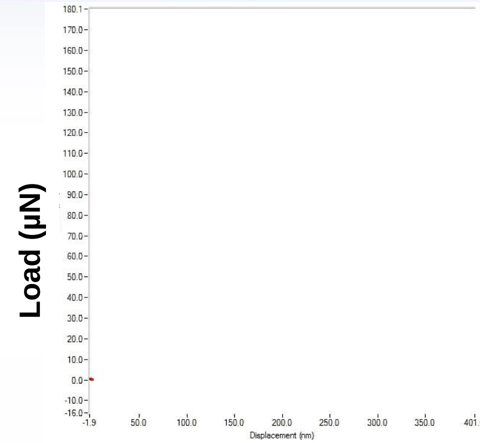


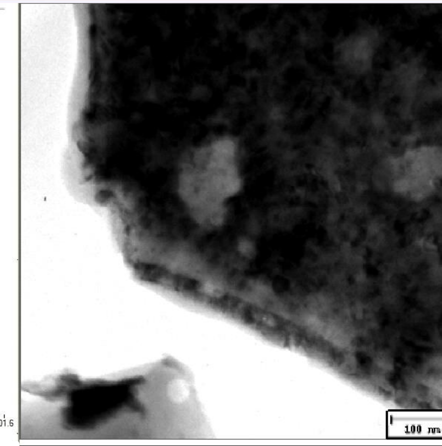
An increase in both observable density and diameter of He bubbles, suggests wear induced He coalescence from interstitial and previously un-observable He

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.





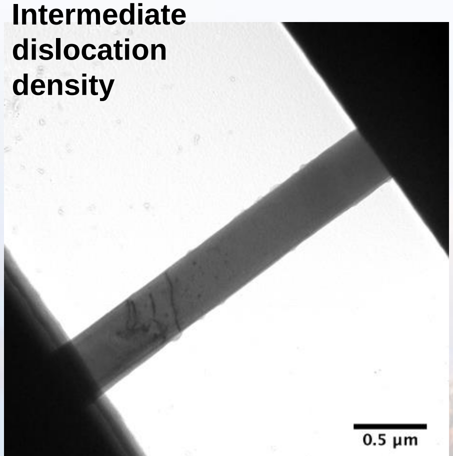


Range of Mechanical Testing Techniques

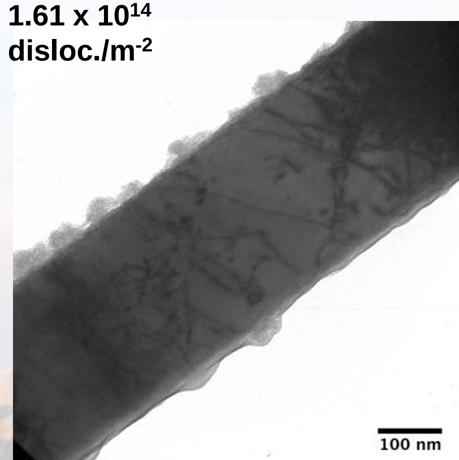
- Indentation
- Compression
- Tension
- Tension
- Wear
- Fatigue
- Creep

Fundamentals of Mechanical Properties

Intermediate dislocation density

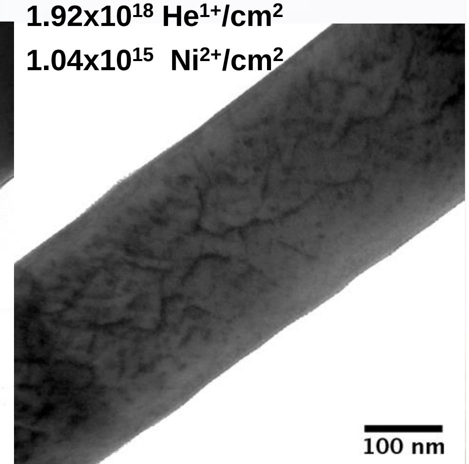


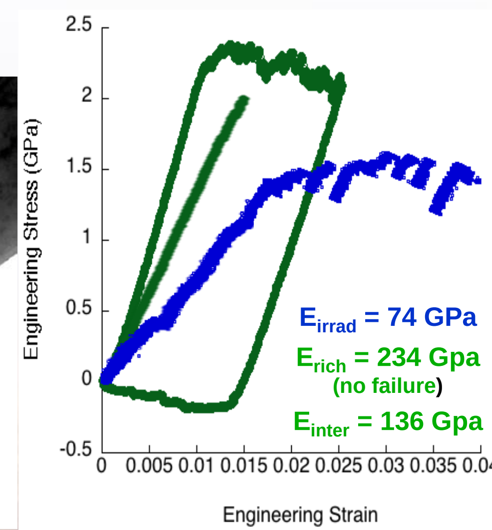
1.61×10^{14} disloc./m^{-2}



1.92×10^{18} He^{1+}/cm^2

1.04×10^{15} Ni^{2+}/cm^2

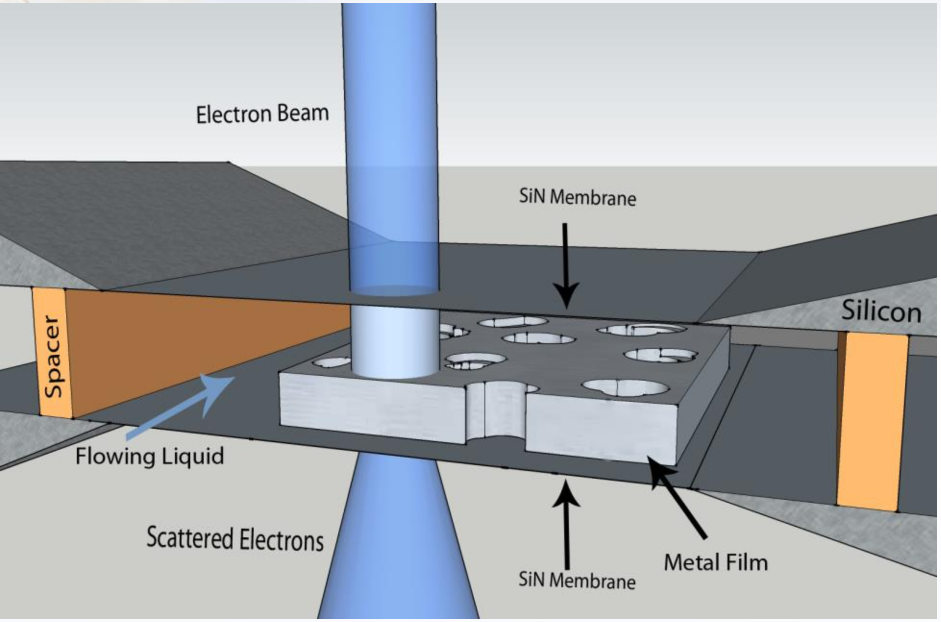




Work has started looking at the quantitative effects of ion irradiation on mechanical properties

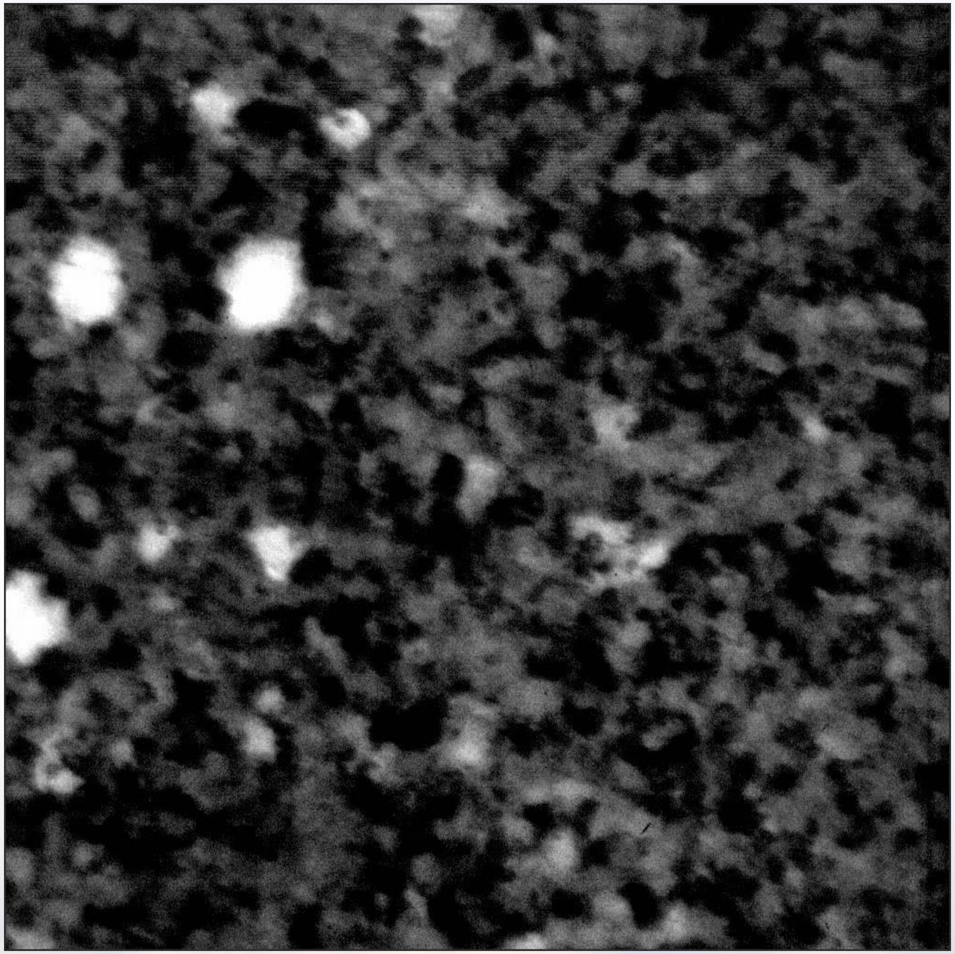
In situ TEM Corrosion

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



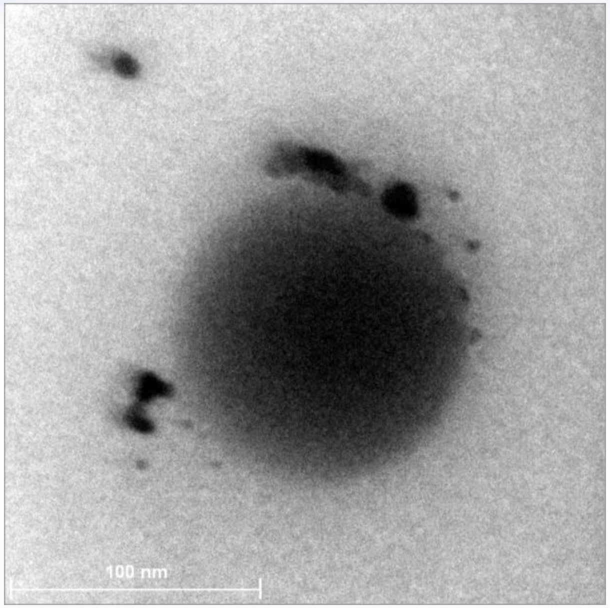
Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.

Other Fun Uses of Microfluidic Cell

Protocell Drug Delivery

S.H. Pratt,
E. Carnes,
J. Brinker

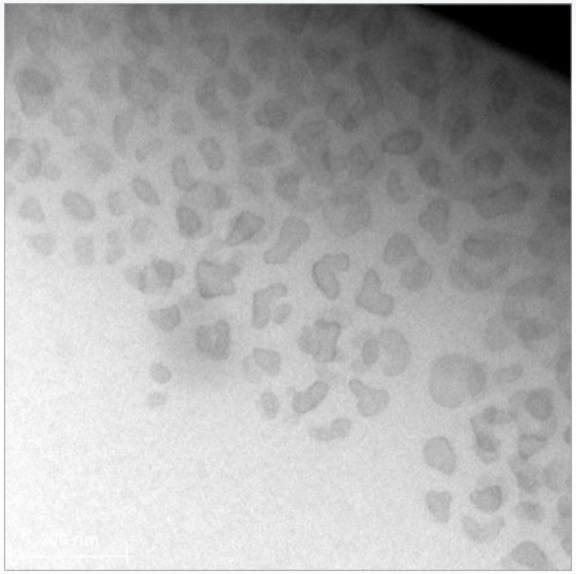
Liposome encapsulated Silica destroyed by the electron beam



BSA Crystallization

S.H. Pratt

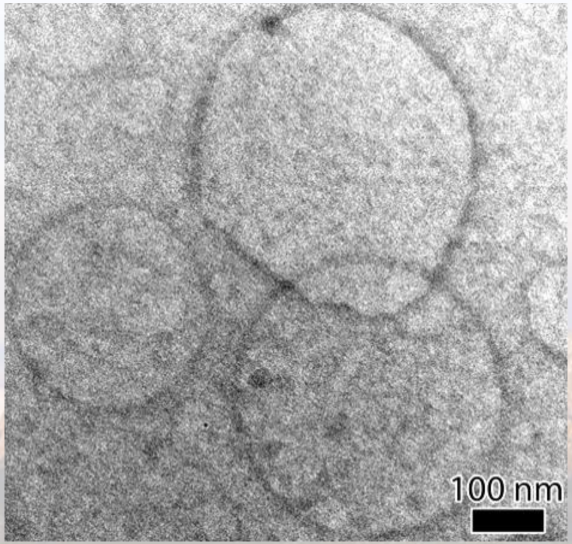
Crystallization of excess Bovine Serum Albumen during flow



Liposomes in Water

S.H. Pratt,
D. Sasaki

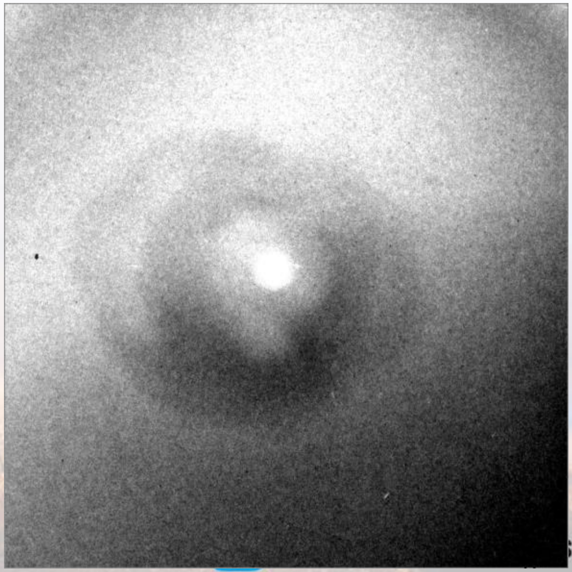
Liposomes imaged in flowing aqueous channel



La Structure Formation

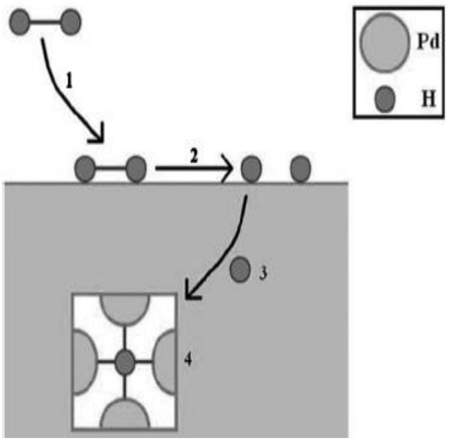
S.H. Pratt,
T. Nenoff

La Nanostructure form from $LaCl_3$ H_2O in wet cell due to beam effects

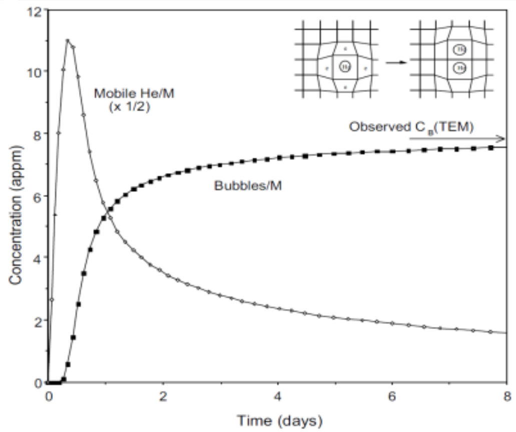


Can *In situ* TEM Address Hydrogen Storage Concerns in Extreme Environments?

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



R. Delmelle, J., Phys. Chem. Chem. Phys. (2011) p.11412

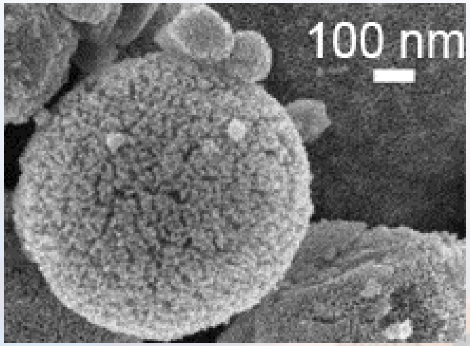


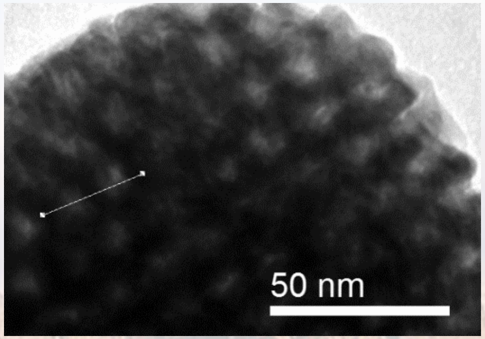
Cowgill, D., Fusion Sci. & Tech., 28 (2005) p. 539
Trinkaus, H. *et al*., JNM (2003) p. 229
Thiebaut, S. *et al*. JNM (2000) p. 217

Vapor-Phase Heating TEM Stage

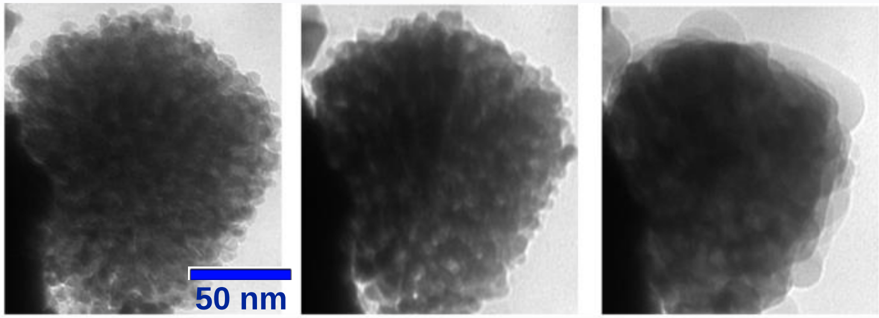
- Compatible with a range of gases
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Compatible with MS and other analytical tools

Harmful effects may be mitigated in nanoporous Pd





- 1 atm H_2 after several pulses to specified temp.

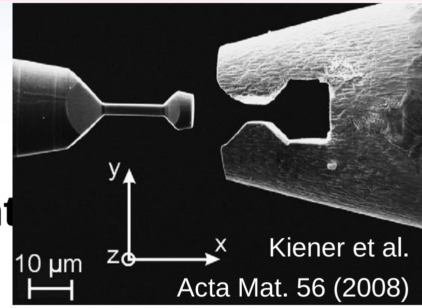


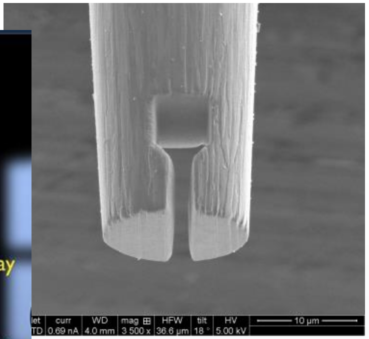
125° C   200° C   300° C

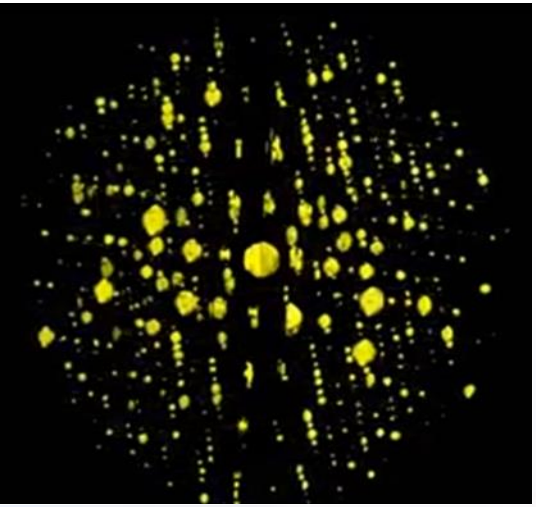
New *in situ* atmospheric heating experiments provide great insight into nanoporous Pd stability

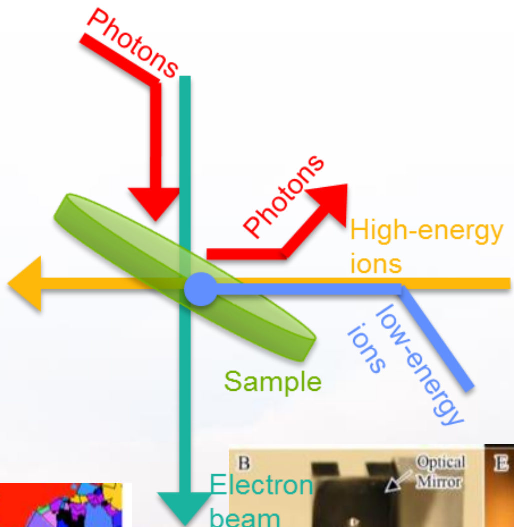
Future Directions Under Pursuit

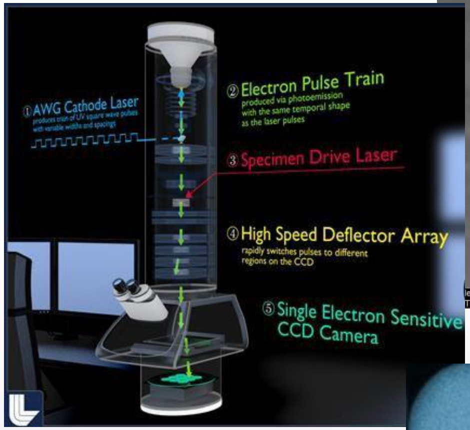
1. In-situ TEM CL, IBIL (currently capable)
2. *In situ* ion irradiation TEM in liquid or gas (currently capable)
3. PED: Local texture characterization (arrived & waiting install)
4. Quantitative in-situ tensile/creep experiments (Sample in development
5. DTEM: Nanosecond resolution (laser optics needed)

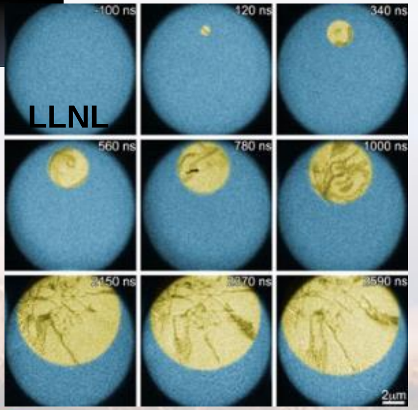




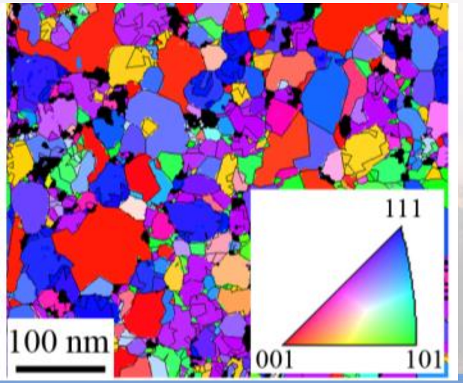


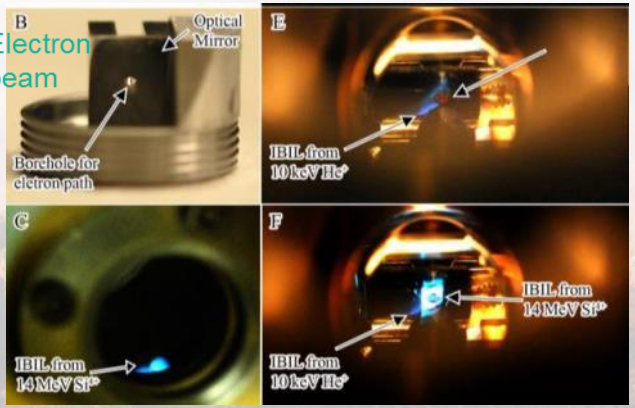






AppFive
NanoMegas





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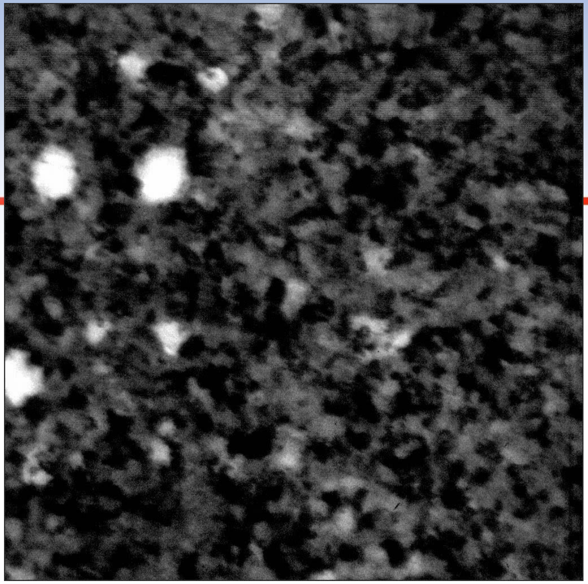
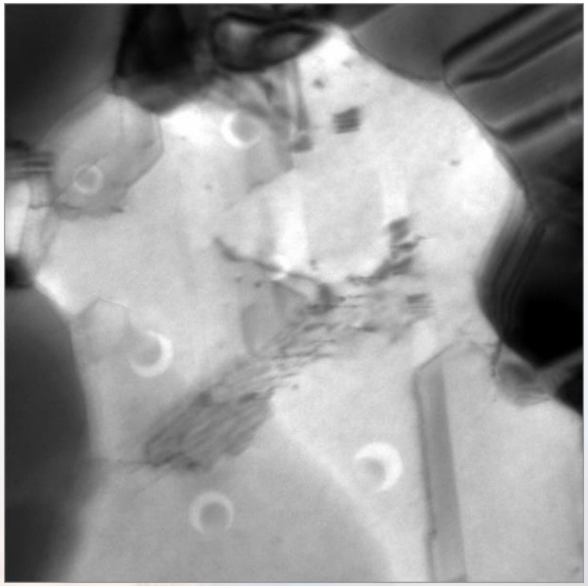
Summary





- Sandia's I^{3}TEM 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 (two beta-testing)
- Apply the current I^{3}TEM capabilities to various material systems in combined environmental conditions
- Expand the I^{3}TEM capability to include
  - Nano to microsecond imaging
  - Laser heating and/or shock to the sample
  - Addition of precession electron diffraction

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

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