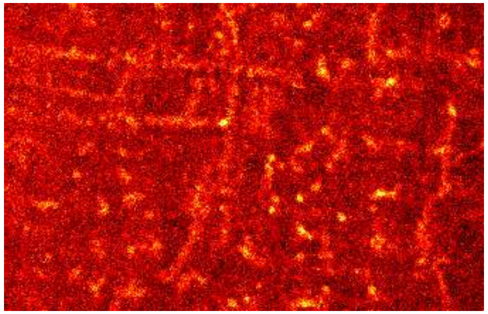
Investigating Materials for Nuclear Related Industries at the Nanoscale

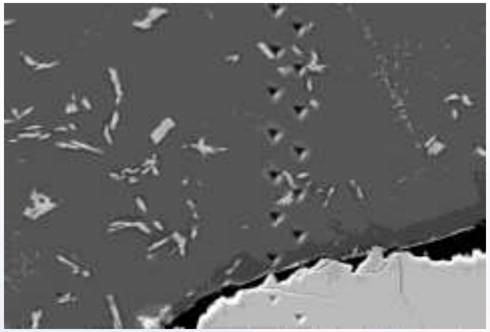
SAND2012-7536C

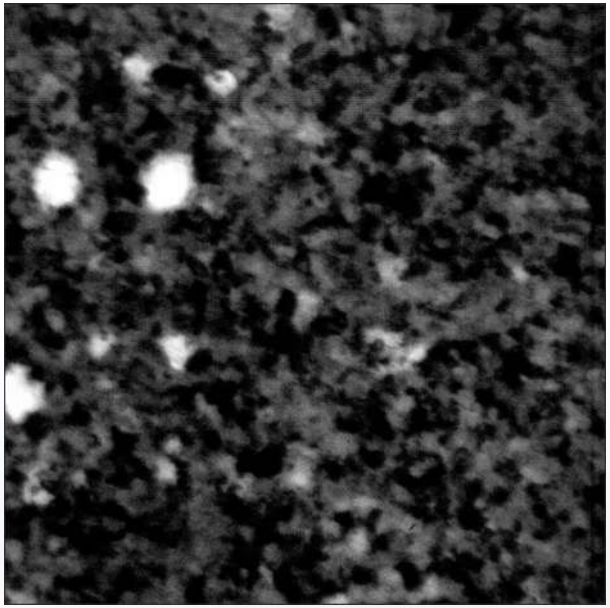
K. Hattar

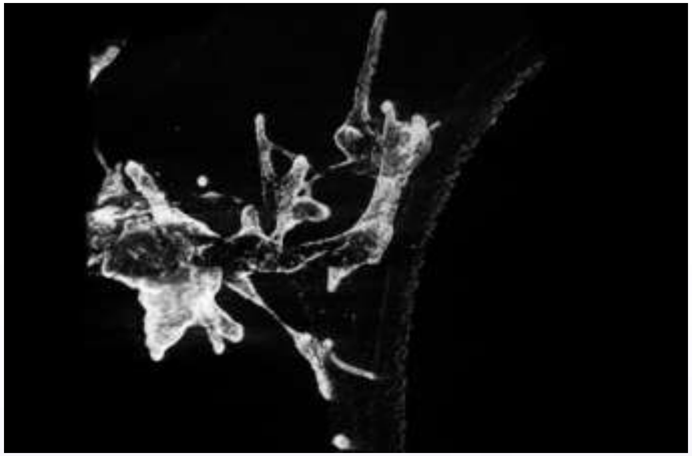
Sandia National Laboratories

September 19, 2012









Collaborators:

- **IBL:** D. Buller, S.B. Van Deusen, B.G. Clark, B.L. Doyle, S.M. Hoppe, S. Rajasekhara, J. Villone, G. Vizkelethy
- **Sandia:** T.E. Buchheit, B. Boyce, T.J. Boyle, F.P. Doty, P. Feng, S. Goods, B.A. Hernandez-Sanchez, A.C. Kilgo, P.G. Kotula, J. Puskar, M.J. Rye, J.A. Scott, P. Yang
- **External:** L.N. Brewer, S. Maloy, A. McGinnis, P. Rossi, D. Masiel, D. Gross, J. Kacher, I.M. Robertson, & Protochips Inc.

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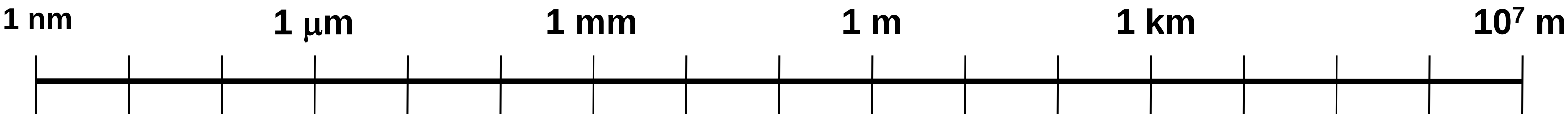
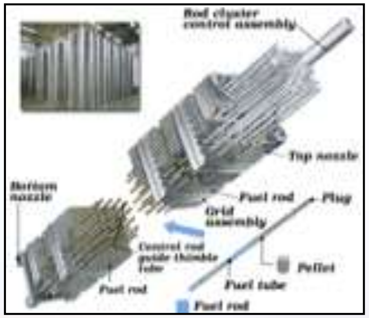
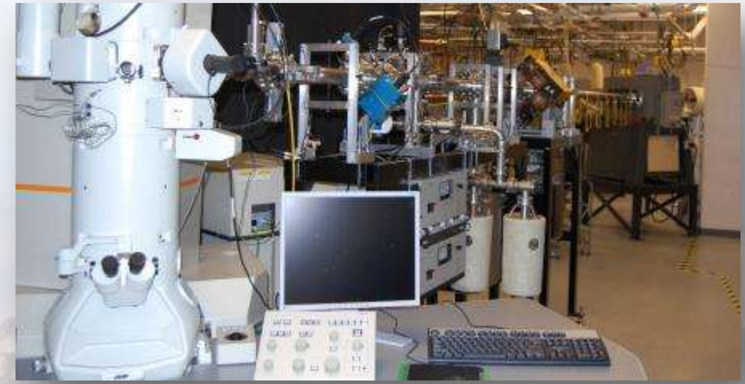
Investigating the nm Scale to Understand the km Scale







In situ Ion Irradiation TEM (I^3TEM)

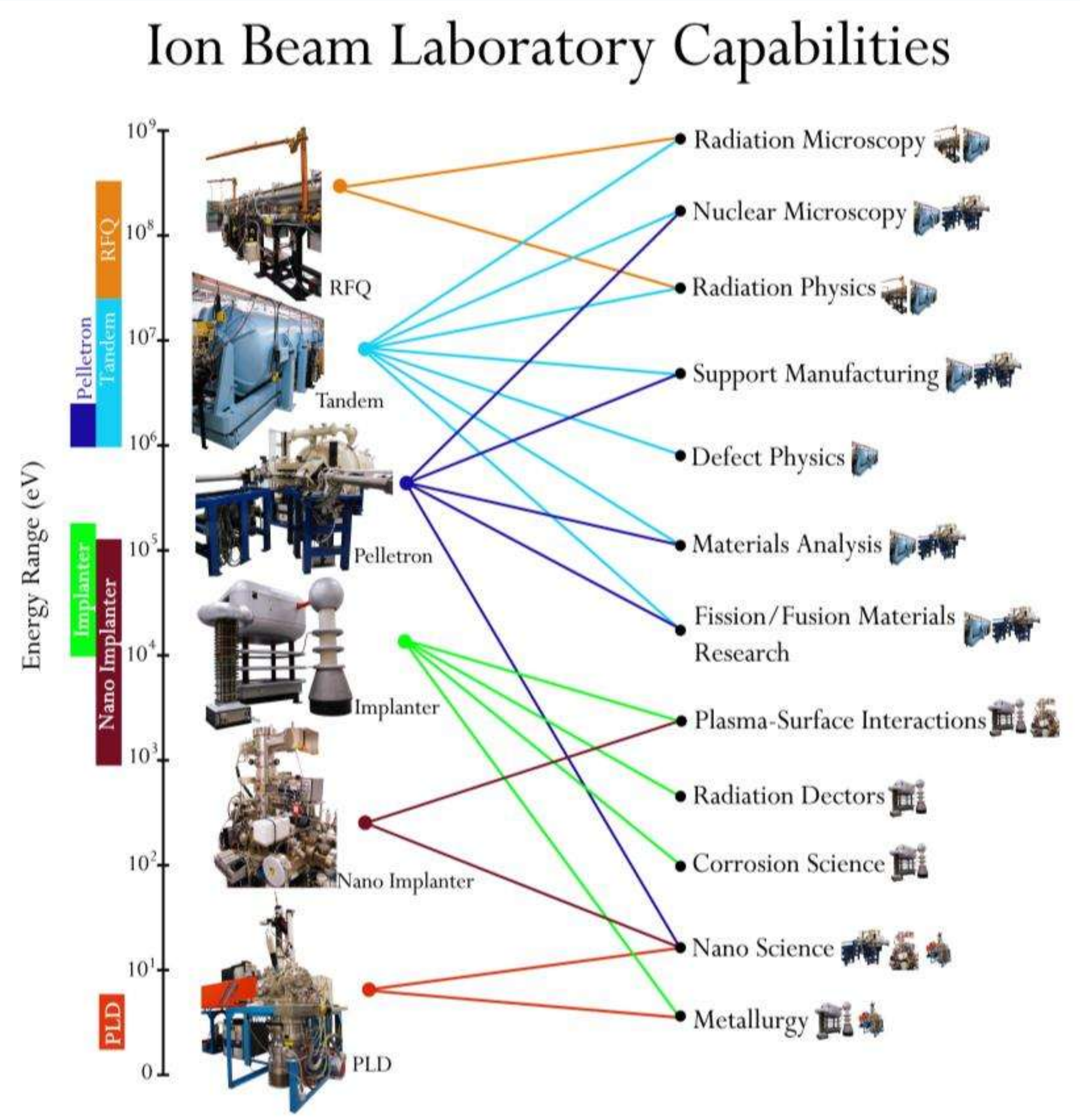
Ion Beam Lab (IBL)

Sandia's New Ion Beam Lab

A special 72 wheeled vehicle with independent steering for each pair of wheels was used to move the Tandem accelerator

<u>New Facility</u>
laboratory space
1850 m^2
office space
650 m^2
<u>Old Facility:</u>
1300 m^2 total

Building: $20M
<u>Equipment: $11M</u>
Total: $40M



Ion Beam Laboratory Capabilities

Sandia's Approach to Rapid Material Validation for Advanced Materials Necessary for New Reactors

- New materials are needed for extreme environments
- Several theories exist for the desired microstructure
- Current neutron fluxes require decades for testing

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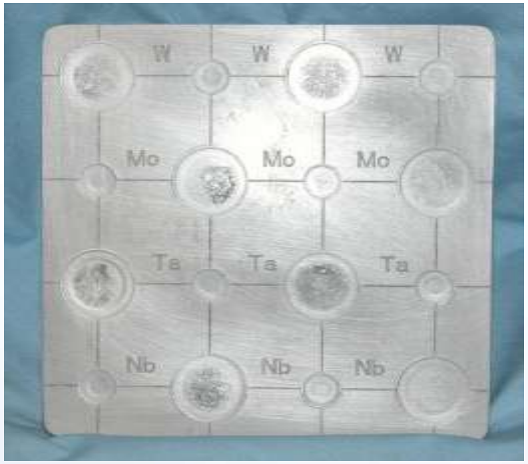
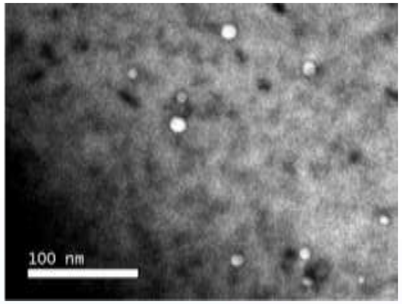
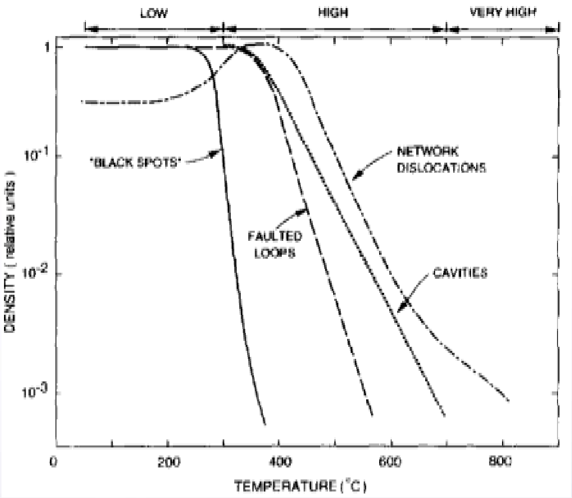
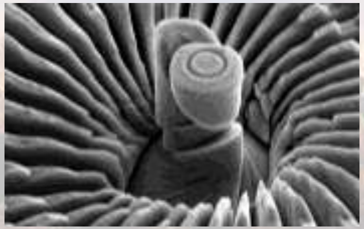






Microstructural Characterization (XTEM)

Mechanical Properties (small-scale testing)



Local Composition (Diffusion Couples)
+
Local Microstructural Control (Ion Irradiation)

Validating Comparison to Neutron Irradiation Experiments + Investigation into new materials

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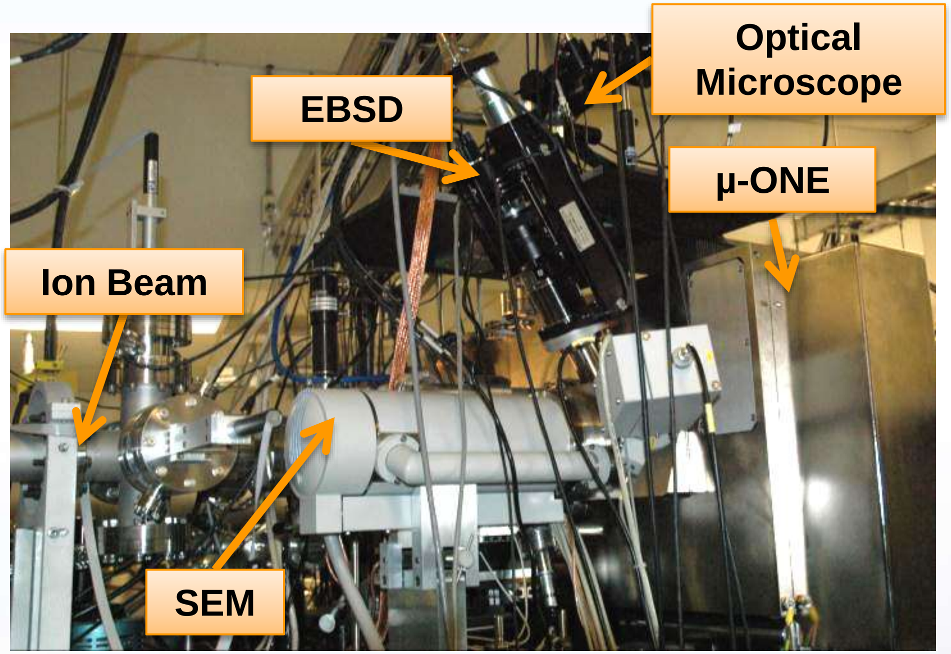
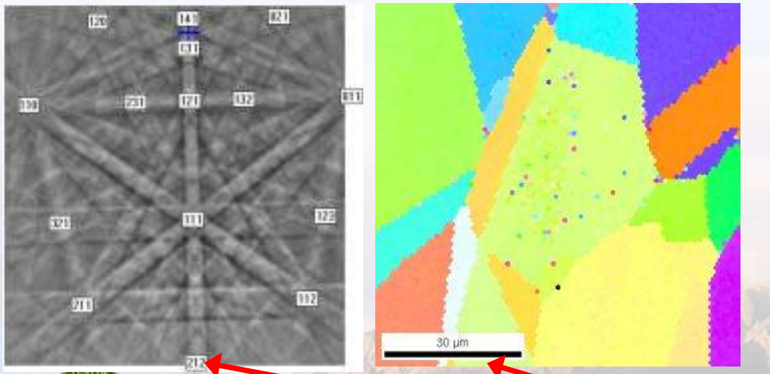
Micro-ONE Capabilities

Micro-ONE = Micrometer resolution Optical, Nuclear, and Electron Microscope

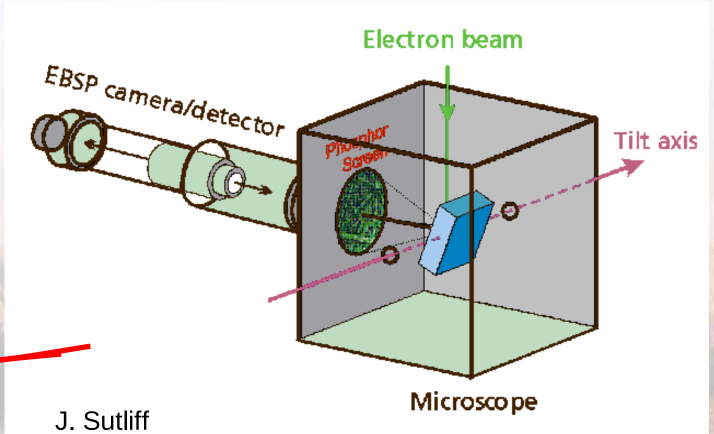
Micro-ONE capabilities

- **Beam size: < 1μm (~ 0.5 μm)**
- **Current: single ions to 10,000 ions/s**
- **Ions: H, He, heavy ions**
- **Energy: (q+1)*6 MV for heavy ions**
- **Scan size: ~ 100x100 μm^2**
- **Stage position with 50 nm resolution**
- **Fast blanking capabilities**
- **Navigation based on GDS II files**
- **IBIC and TRIBIC capabilities**
- **EBSD mapping**





First EBSD Pattern and Map obtained with this system



J. Sutliff

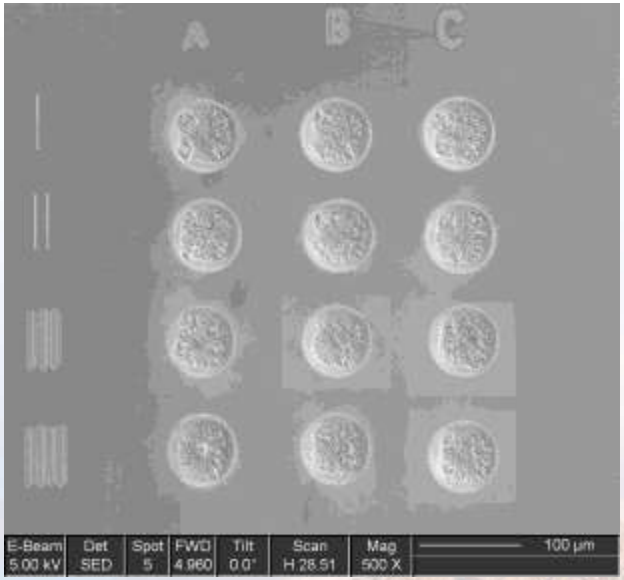
Allows parallel imaging of changes in microstructure: grain size & phase transformations.

Micropillar Compression Experiments

Collaborators: S.B. Van Deusen, D.L. Buller, M.J. Rye, L.N. Brewer, & B. Boyce

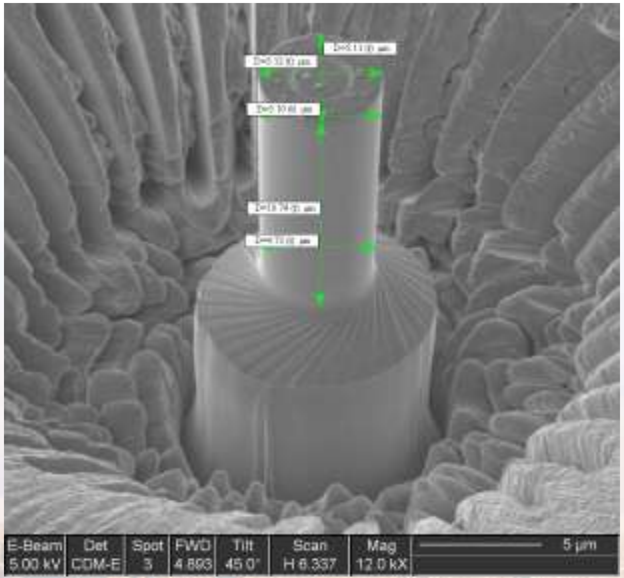
Sample Preparation:

- Copper single crystals (FCC)
- Different crystallographic orientations: (100), (110), and (111)
- Self-ion Implants at 30 MeV to 0 (control), 50 dpa, and 100 dpa.



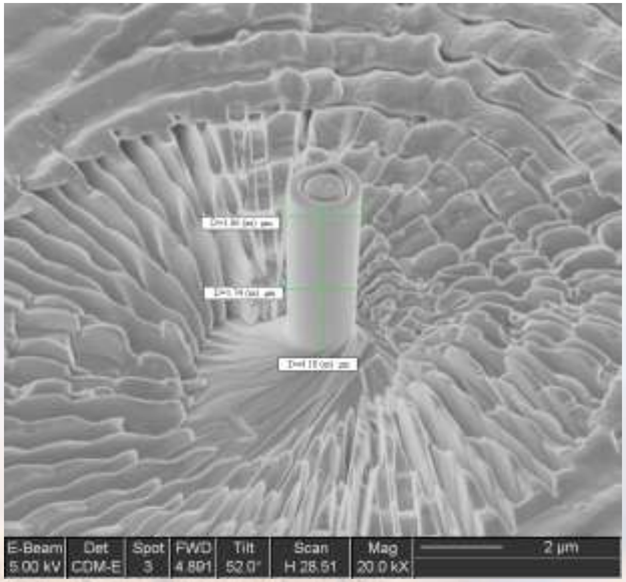
Pillar Manufacturing:

- We employed Uchic's FIB lathe machining process for straight-walled cylinders.
- Array of at least 9 nominally identical pillars tested per condition to assess statistical variability.
- Height varies from 4 µm to 10 µm



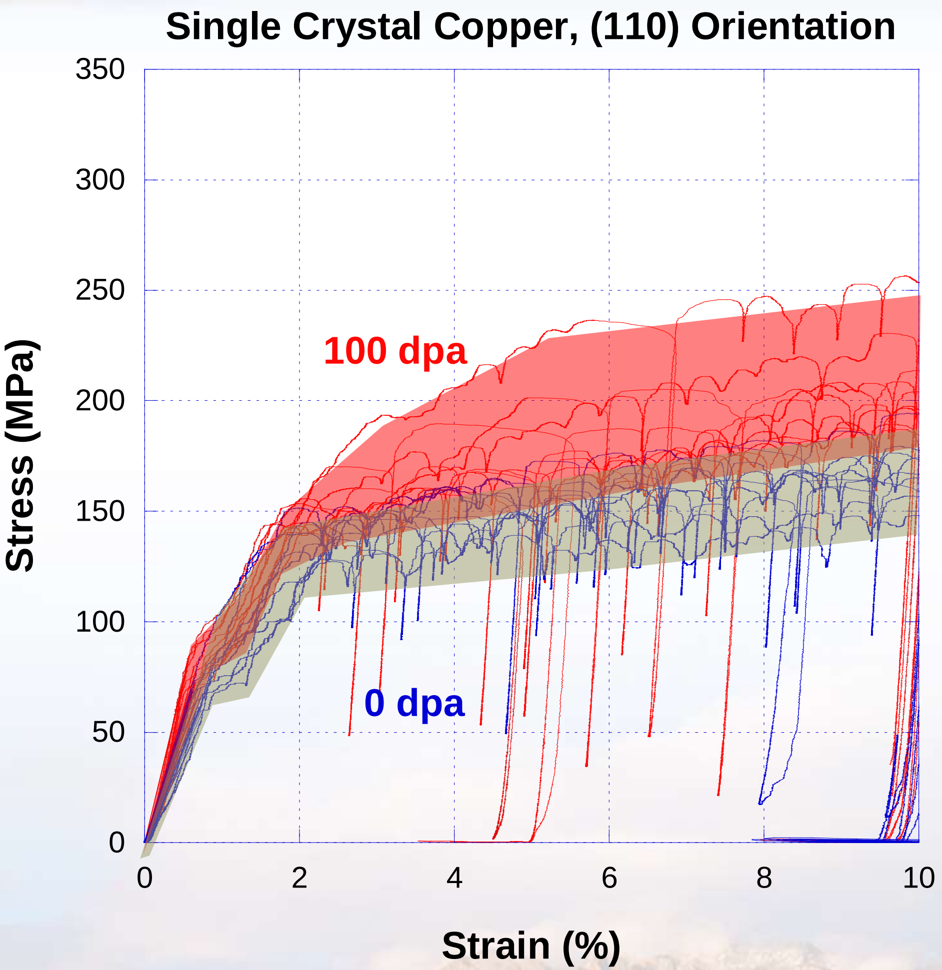
Compression Testing:

- Hysitron Performech Nanoindenter permits <1 nm and <1 µN resolution.
- 25 µm flat ended cone indenter in feedback displacement control, rather than typical force control.
- Pillars compressed 10% strain at a strain rate of 0.025 s^{-1}.

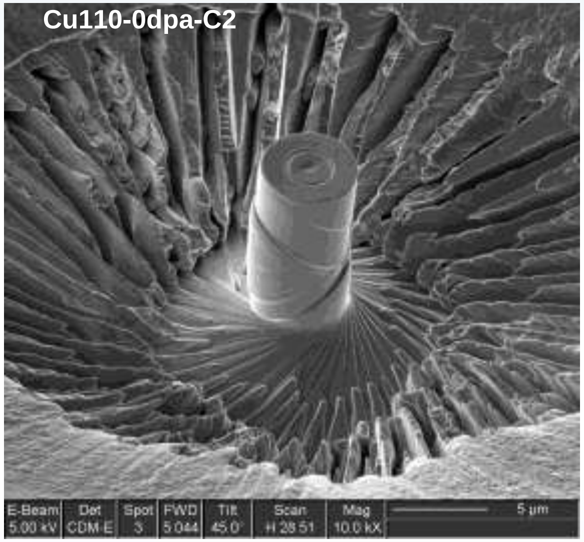


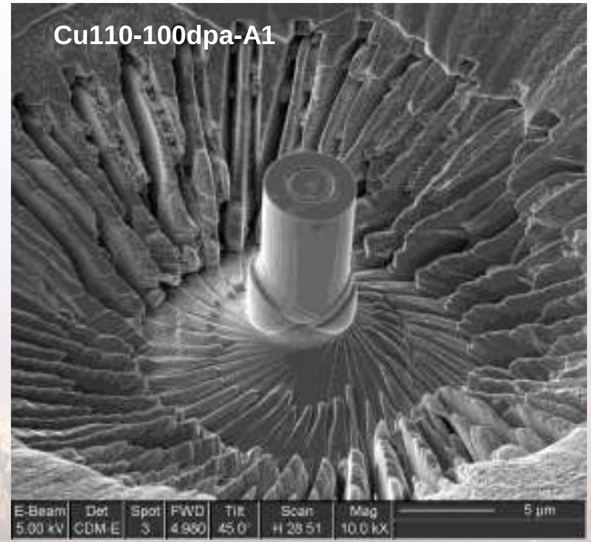
Large Micropillar Compression

Collaborators: S.B. Van Deusen, D.L. Buller, M.J. Rye, L.N. Brewer, & B. Boyce



Single Crystal Copper, (110) Orientation

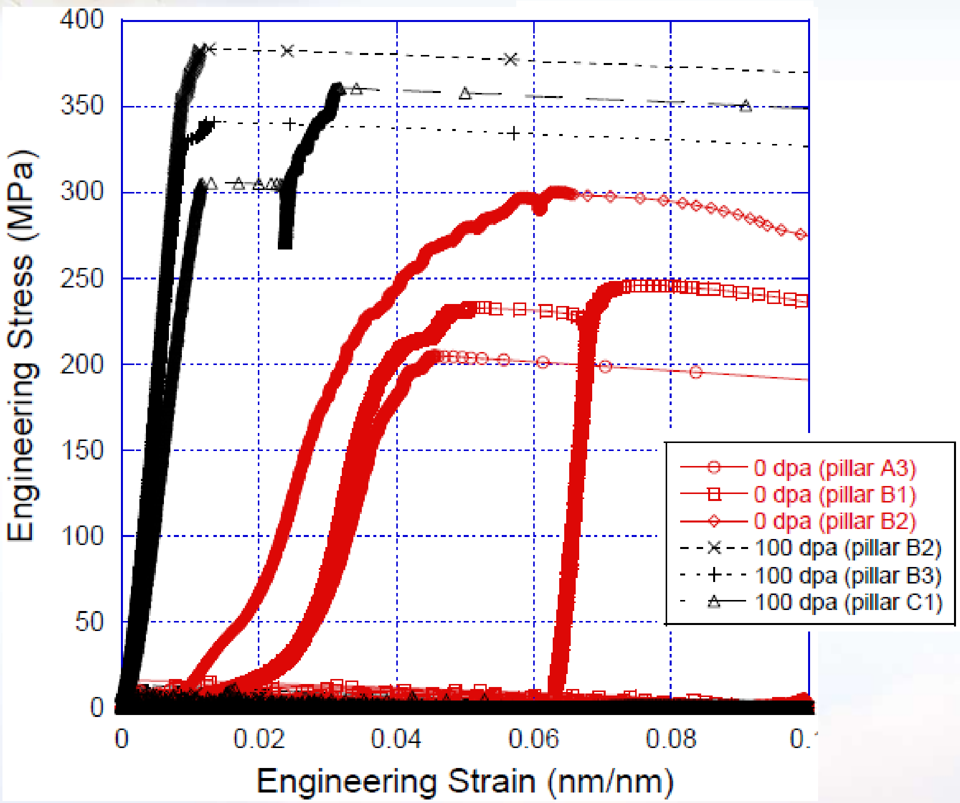




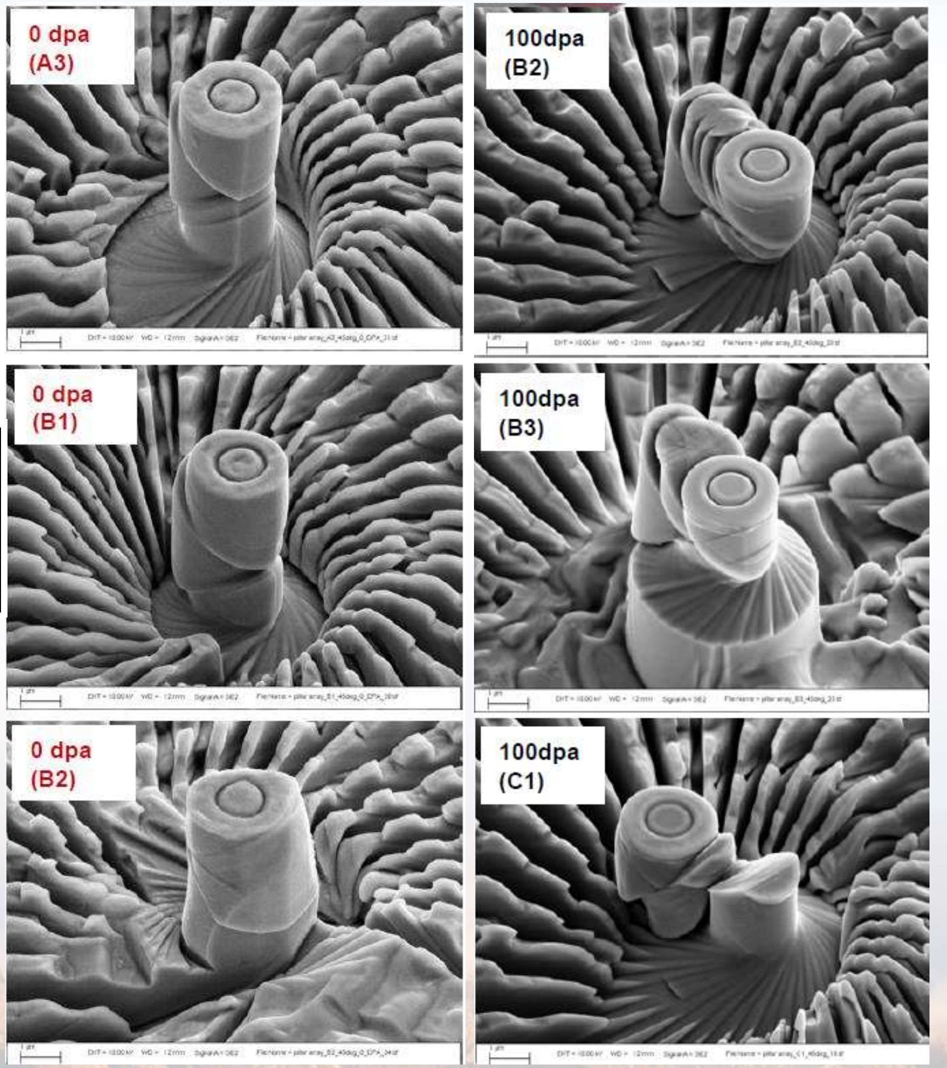
Minimal difference between the control and irradiated 10 µm-tall pillars. Slip occurred in the bottom fraction of the pillars.

Intermediate Micropillar Compression

Collaborators: S.B. Van Deusen, D.L. Buller, M.J. Rye, L.N. Brewer, & B. Boyce

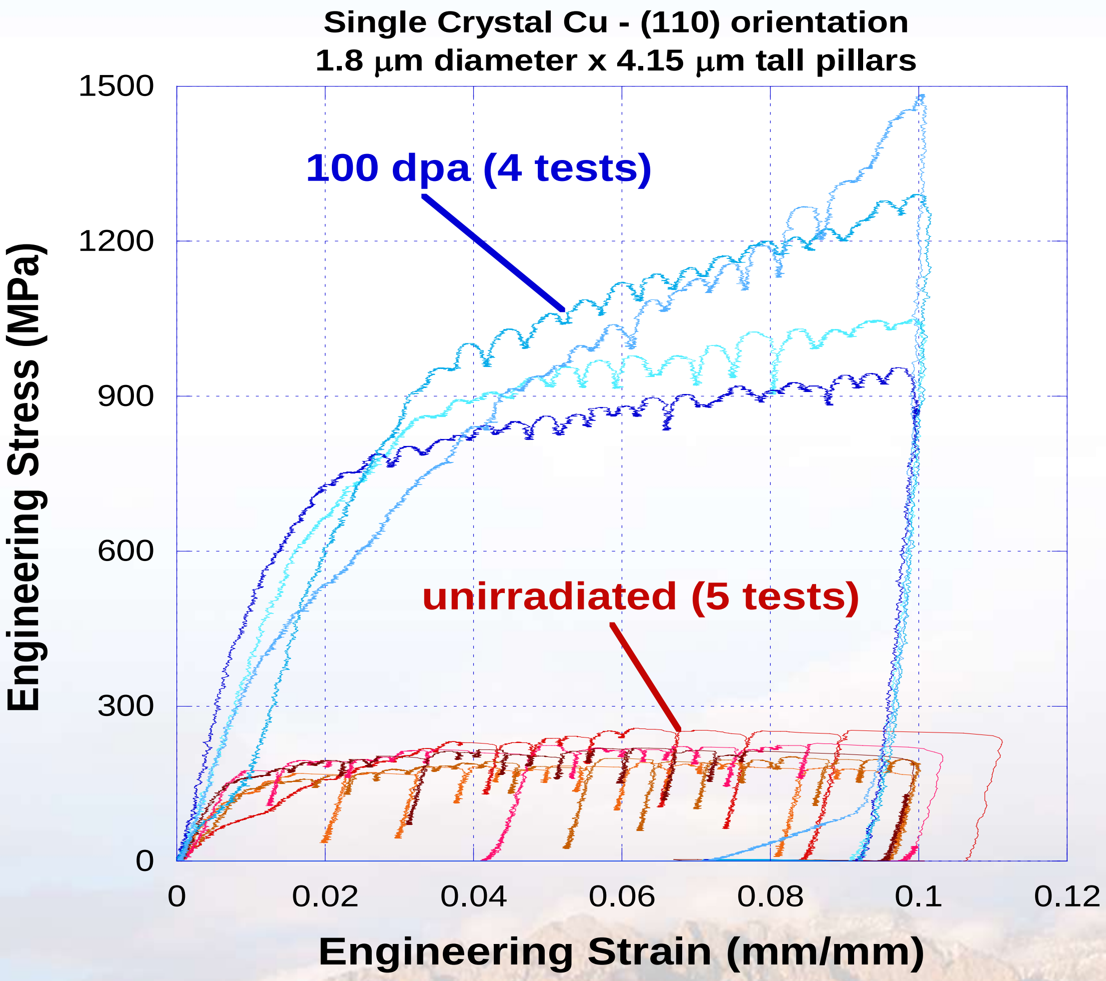


5 µm-tall pillars show greater distinction with catastrophic failure

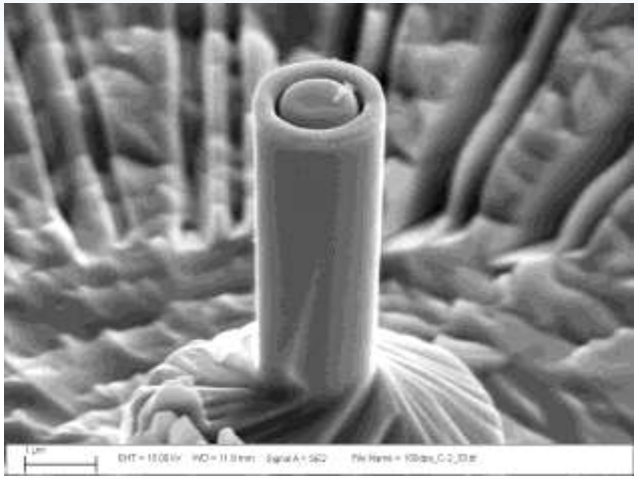


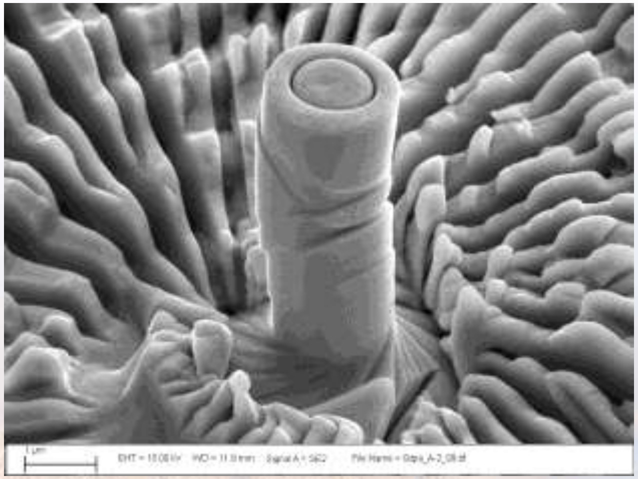
Small Micropillar Compression

Collaborators: S.B. Van Deusen, D.L. Buller, M.J. Rye, L.N. Brewer, & B. Boyce



Single Crystal Cu - (110) orientation
1.8 μm diameter x 4.15 μm tall pillars





Initial tests indicate that the 4 μm-tall pillars are 5 times stronger and show no signs of slip band formation

Testing of Irradiated Stainless Steels

Collaborators: S.B. Van Deusen, D.L. Buller, T.E. Buchheit, A.C. Kilgo, & L.N. Brewer

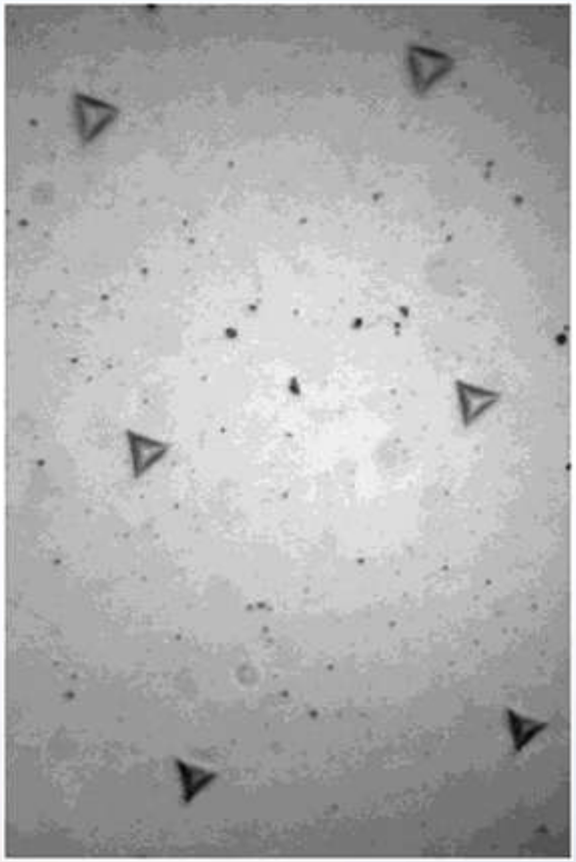
- Micropillar is difficult for many polycrystalline materials
  - Due to the dependence of FIB milling rate on orientation

To validate the approach:

1. Metals previously tested by Neutron Irradiation must be tested
2. The effect of temperature and various ion characteristics must be considered

Thus, we irradiated

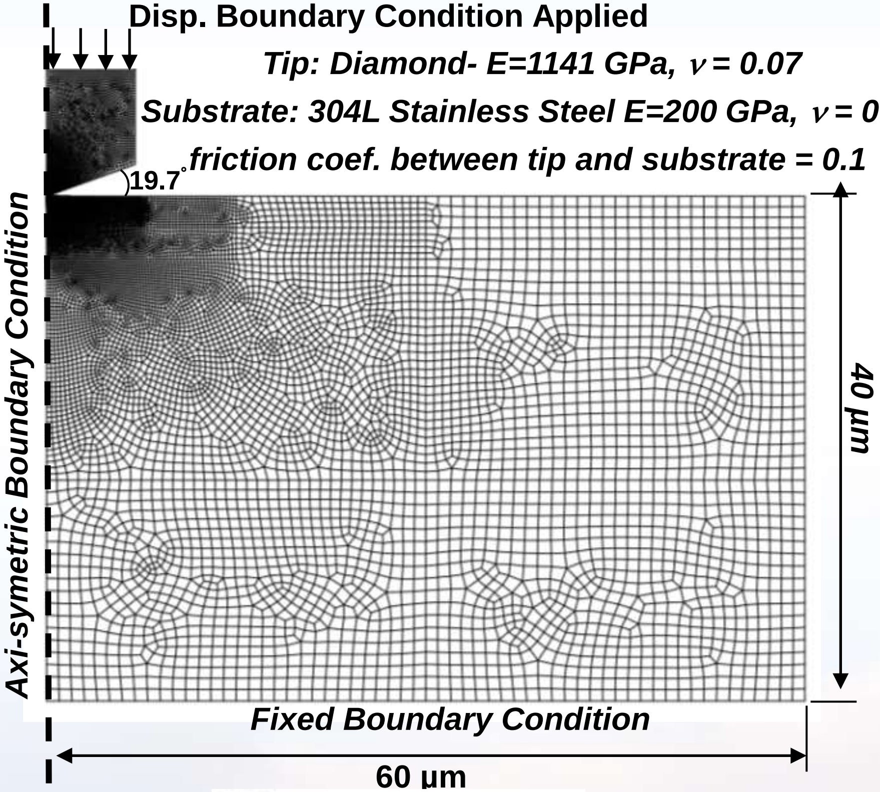
- 420, 409, and 316L SS
- Approximately 10 dpa, 40 dpa, and 100 dpa
- Temperatures of 400 ºC, 500 ºC, and 600 ºC



Three steel compositions were irradiated under various conditions. Nanoindentation was selected as the optimal small scale testing method.

Finite Element Simulations of Indentations into Ion Irradiated Steels

Collaborators: S.B. Van Deusen, D.L. Buller, T.E. Buchheit, A.C. Kilgo, & L.N. Brewer



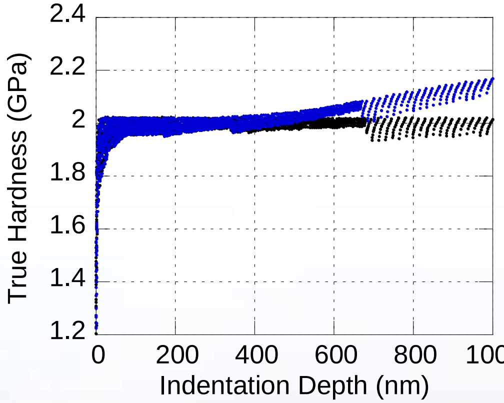
Disp. Boundary Condition Applied

Tip: Diamond- E=1141 GPa, $\nu = 0.07$

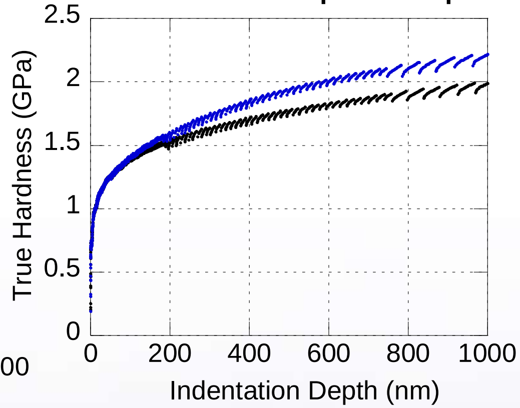
Substrate: 304L Stainless Steel E=200 GPa, $\nu = 0.07$

friction coef. between tip and substrate = 0.1

Hardness vs. Depth
Simulated Indentation Experiments
304SS - Conical Tip Geometry

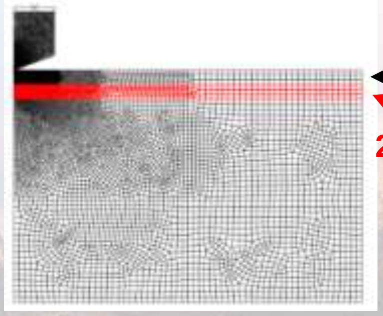


Hardness vs. Depth
Simulated Indentation Experiments
304SS - 10 um spherical tip



Without hardened subsurface layer

With hardened subsurface layer



σ_y=350 MPa

σ_y=700 MPa

2.5 µm top layer

2.5 µm hardened layer

Deviations due to ion irradiation are expected from both spherical and conical indentations

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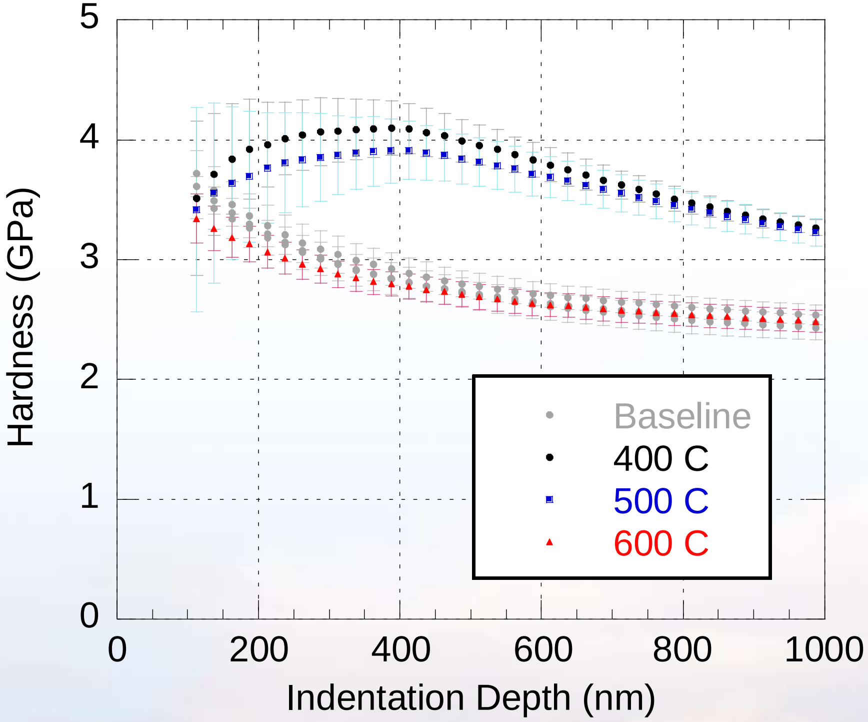
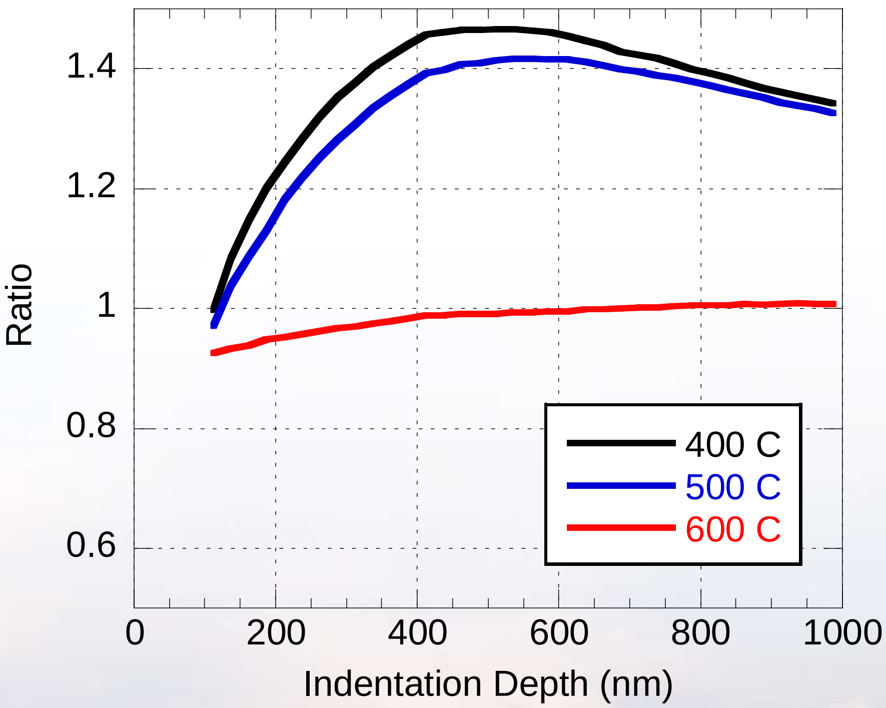
Berkovich Indentation of 100 dpa Irradiated Samples

Collaborators: S.B. Van Deusen, D.L. Buller, T.E. Buchheit, A.C. Kilgo, & L.N. Brewer

Hardness vs. Indentation Depth Comparison of 100 dpa measurements



Baseline to Implanted Region Hardness Ratio vs. Indentation Depth - 100 dpa experiments



At 100 dpa, the hardness difference between 400 ºC and 500 ºC sample and the control microstructure has increased.

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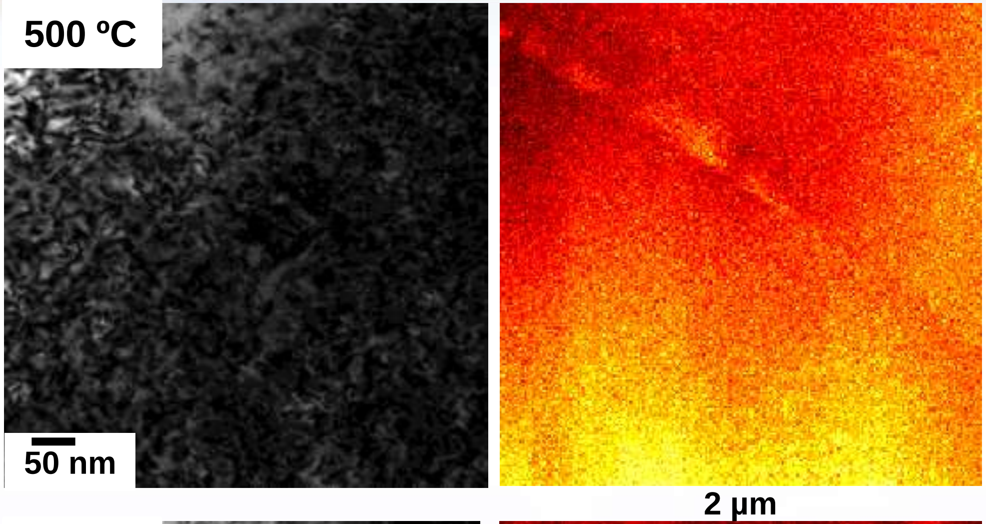
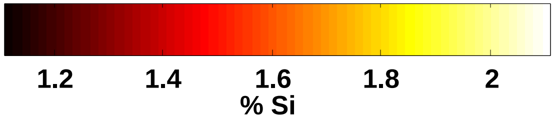
Microstructural Evolution between 500 ºC and 600 ºC

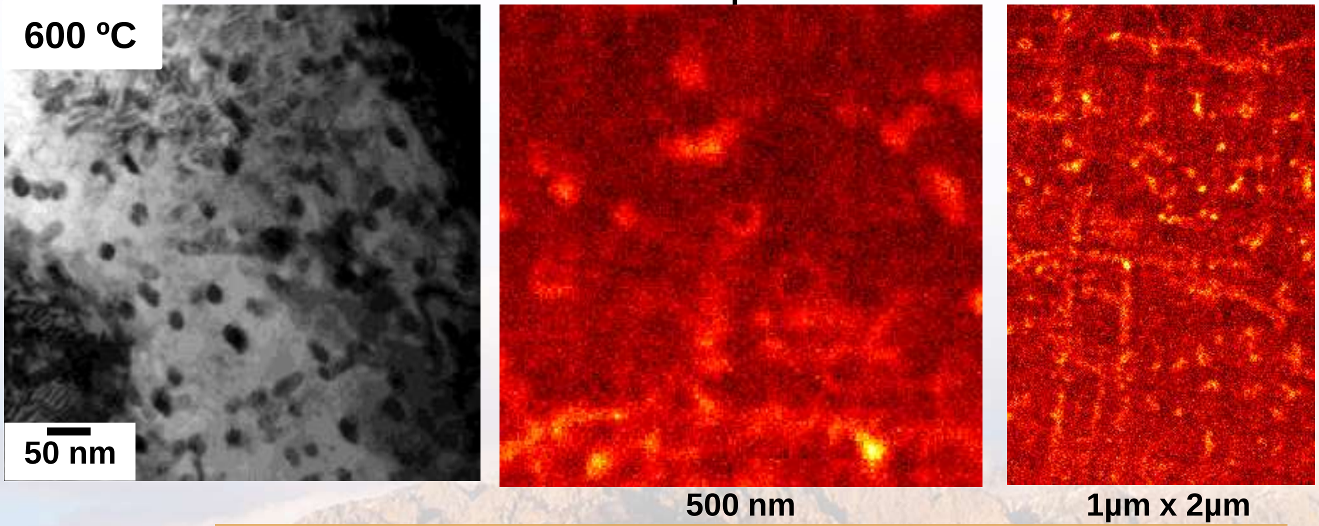
316L Stainless Steel: 100 dpa, 20 MeV Nickel Ions

Collaborators: P.G. Kotula, S.B. Van Deusen, D.L. Buller, T.E. Buchheit, A.C. Kilgo, & L.N. Brewer

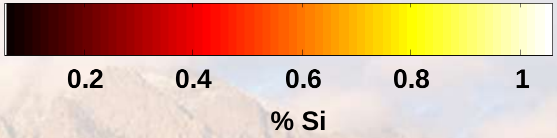


- Large number of small defects present in the irradiated region
- No significant segregation of either the Ni or Si constituents





- Voids are formed and are self-ordered
- Significant segregation of either the Ni or Si constituents



Ni and Si rich regions appear to self-organize and sometimes surround voids at 600 °C, but not 500 °C

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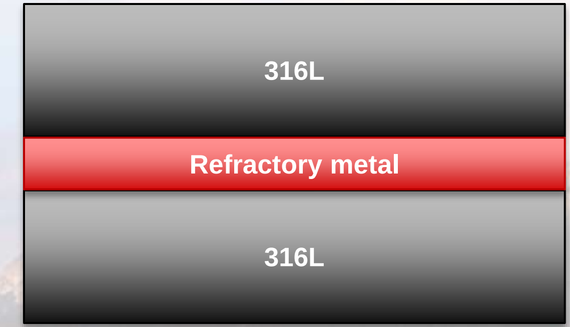
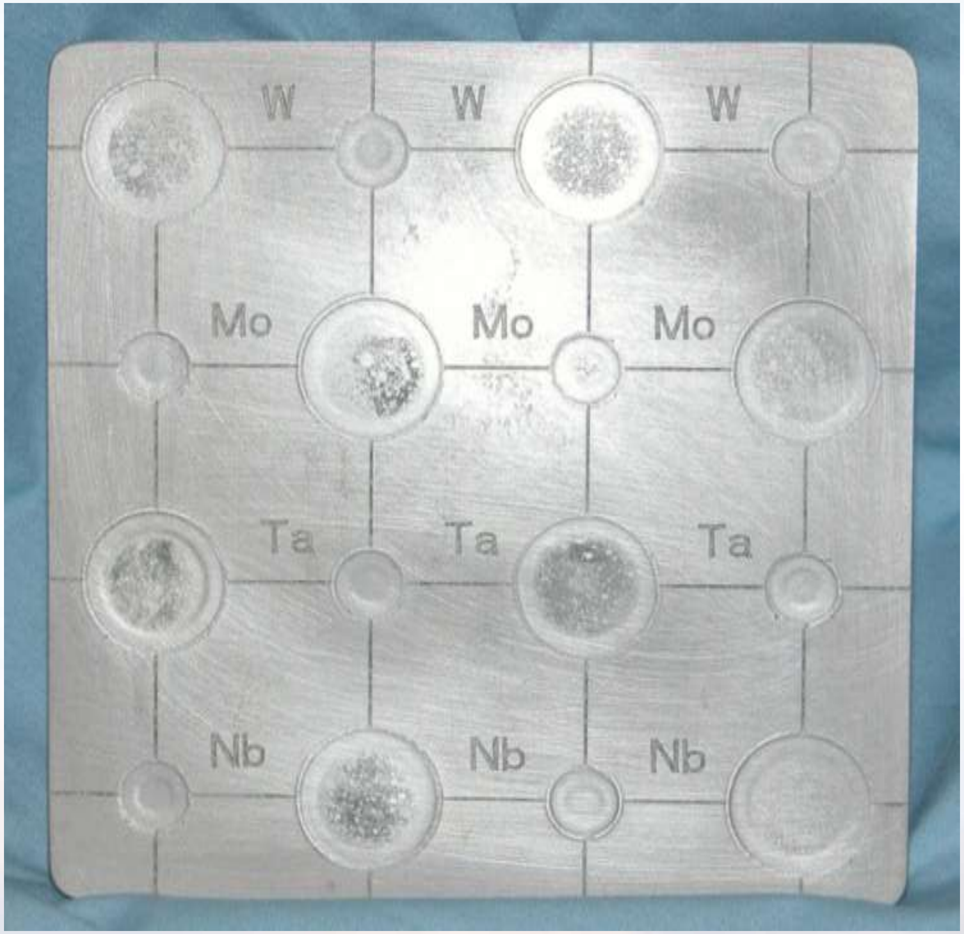
Diffusion Couple Production

Collaborators: S.B. Van Deusen, D.L. Buller, A. McGinnis, L.N. Brewer, A.C. Kilgo, J. Puskar, & S. Goods

- Initial work was started on single crystal Cu, 316L, 409, and 420
- Two sets of binary couples are being produced based on
  - 316L and HT9 steels
- Coupled with:
  - Refractory metals-Ta, W, Nb, Mo
- Coupled by HIP
- Vacuum annealed for 400 hrs. at 1100 °C
- EDM cut for ion irradiation, microstructural analysis, small-scale mechanical property testing.





Diffusion multiples provide a method to rapidly test many alloy compositions

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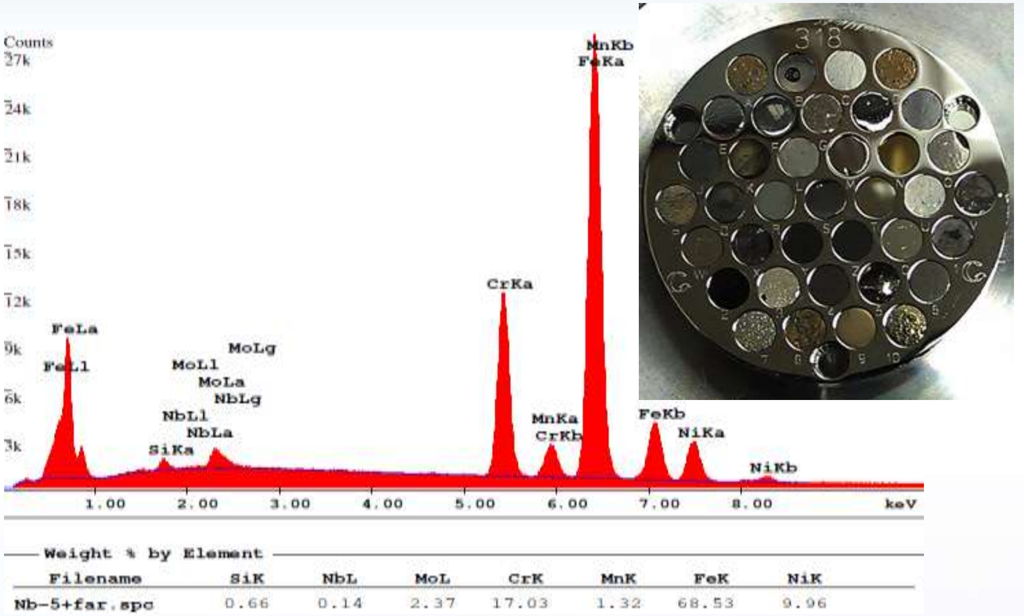
Combinatorial Nanoindentation and EDS

Collaborators: S.B. Van Deusen, DL. Buller, A. McGinnis, L.N. Brewer, A.C. Kilgo, J. Puskar, & S. Goods



Weight % by Element

Filename	SiK	NbL	MoL	CrK	MnK	FeK	NiK
Nb-5+far.spc	0.66	0.14	2.37	17.03	1.32	68.53	9.96

Nanoindentation:

- Continuous Stiffness Measurement (CSM)
- Indentation Depth: ≤ 200 nm
- Indentation Spacing: 5 µm
- Drift Rate: ≤ 0.02 nm/s
- 3-wide array of indentation away from interface.
- Modulus and hardness values are the average of these 3 indents.

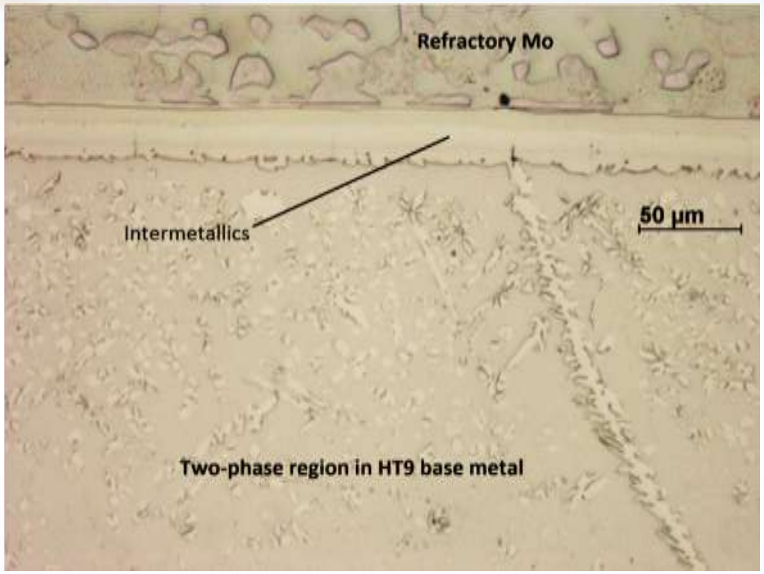
Pure Standards Analyzed:

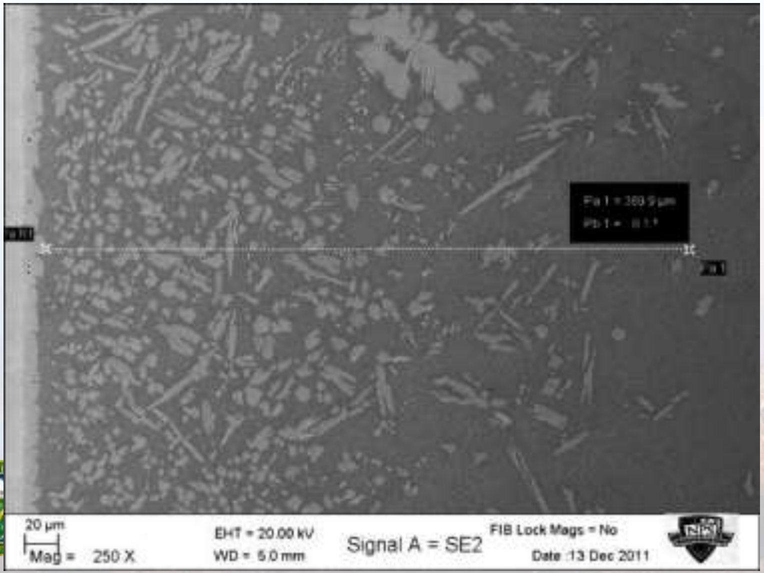
Fe (FeS_2), Cr (Cr_2O_3), Mn, Mo, Ni, Nb (Nb_2O_5), Si (SiO_2),Ta, V (V_2O_5), W

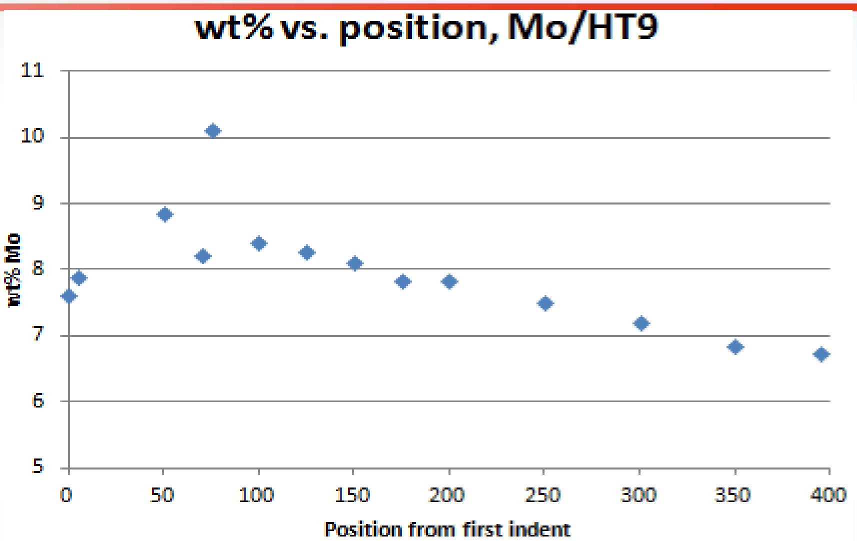
Nanoindentation and EDS provide rapid characterization of variations within the diffusion multiples

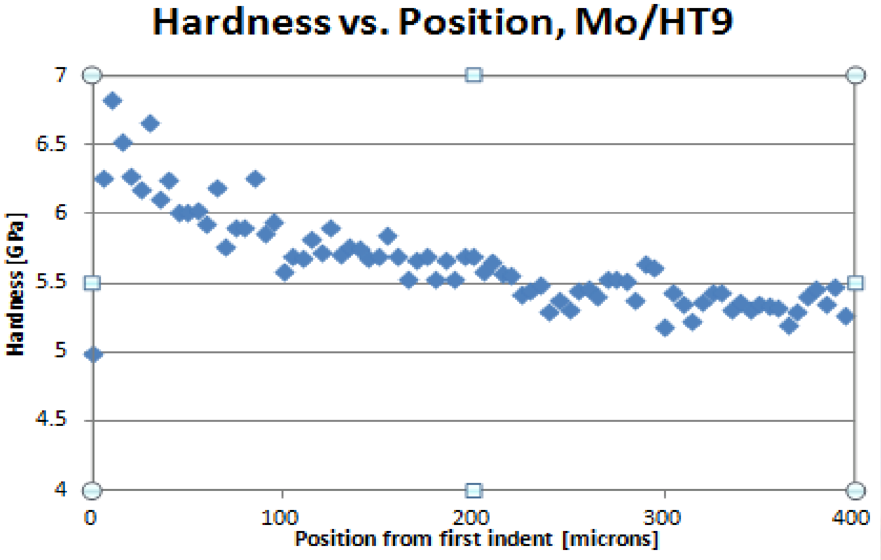
Composition and Hardness for Mo/HT9 Diffusion Couple

Collaborators: S.B. Van Deusen, DL. Buller, A. McGinnis, L.N. Brewer, A.C. Kilgo, J. Puskar, & S. Goods





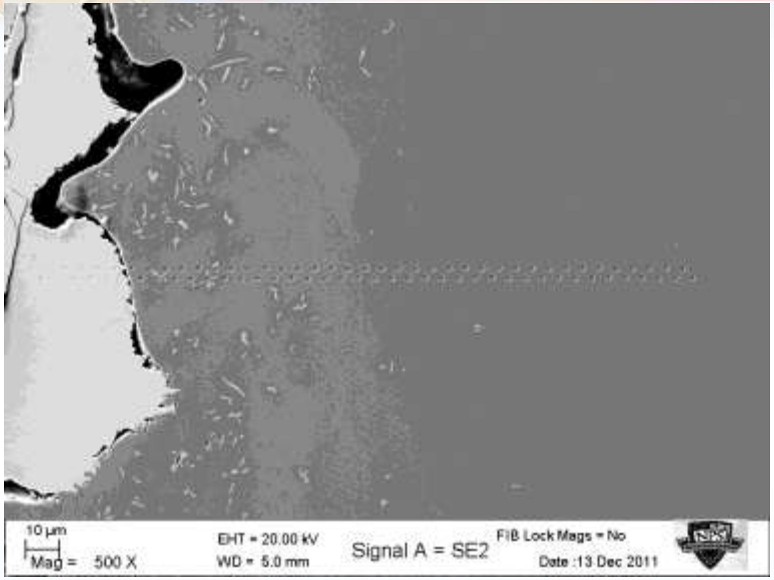

wt% vs. position, Mo/HT9

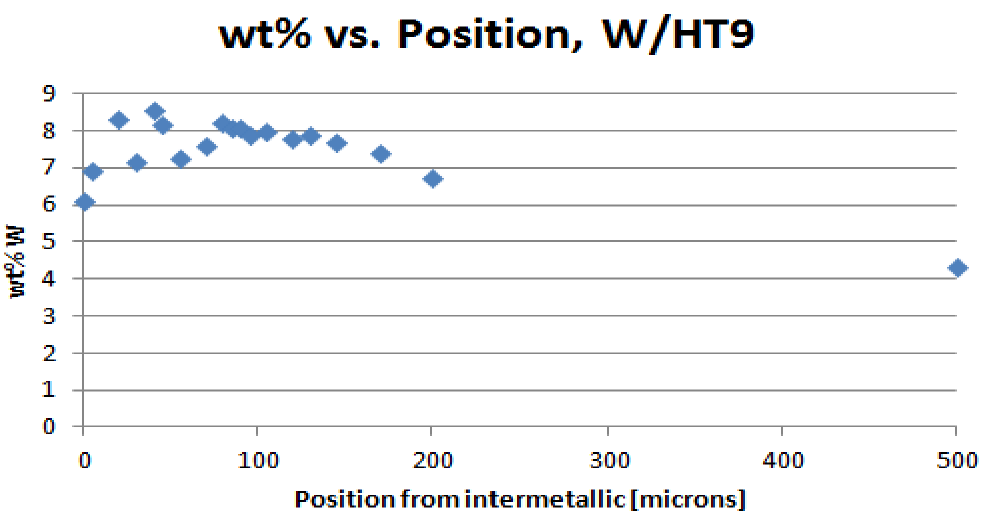

Hardness vs. Position, Mo/HT9

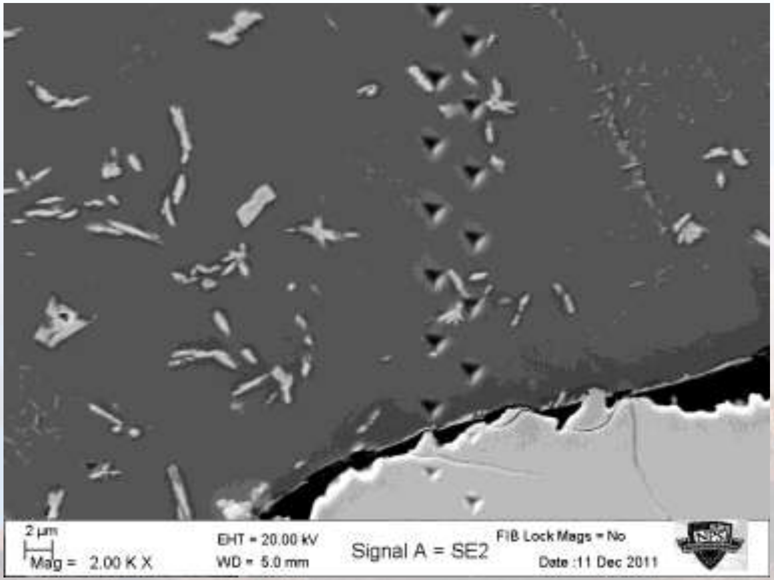
Rapid characterization of many compositions

Composition and Hardness for W/HT9 Diffusion Couple

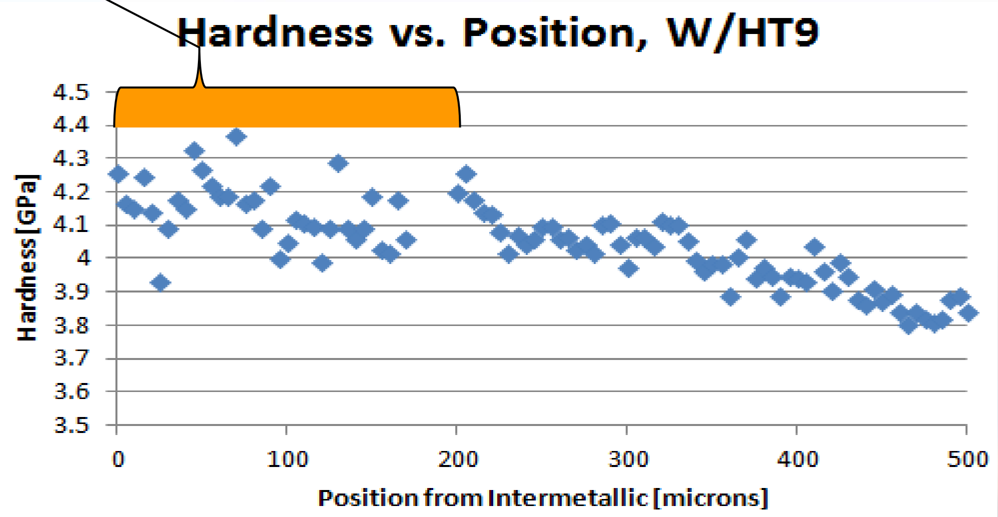
Collaborators: S.B. Van Deusen, DL. Buller, A. McGinnis, L.N. Brewer, A.C. Kilgo, J. Puskar, & S. Goods







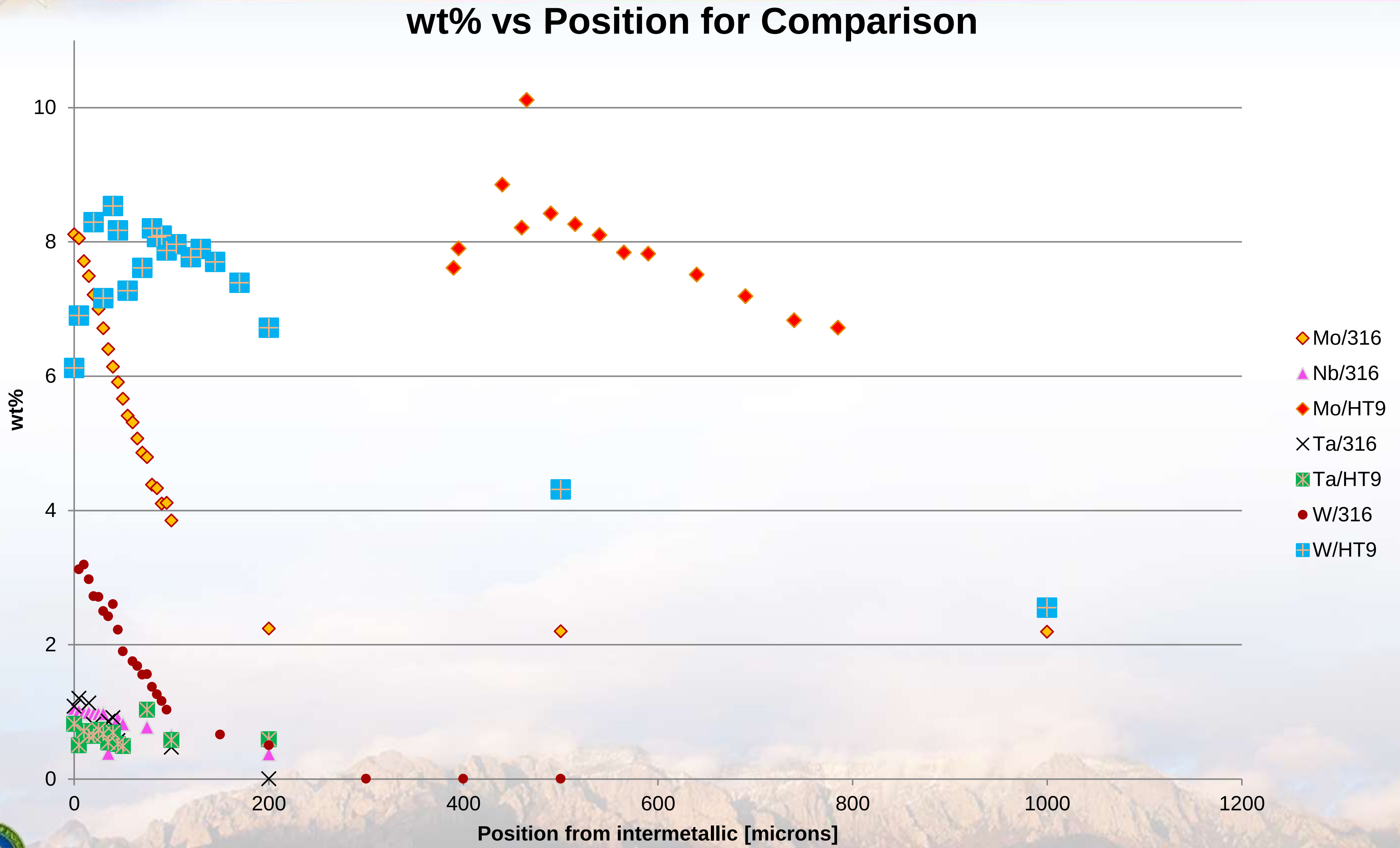
Multi-Phase Region



Multi-phase region has nearly uniform hardness

Composition Comparison for All Diffusion Couples

Collaborators: S.B. Van Deusen, D.L. Buller, A. McGinnis, L.N. Brewer, A.C. Kilgo, J. Puskar, & S. Goods

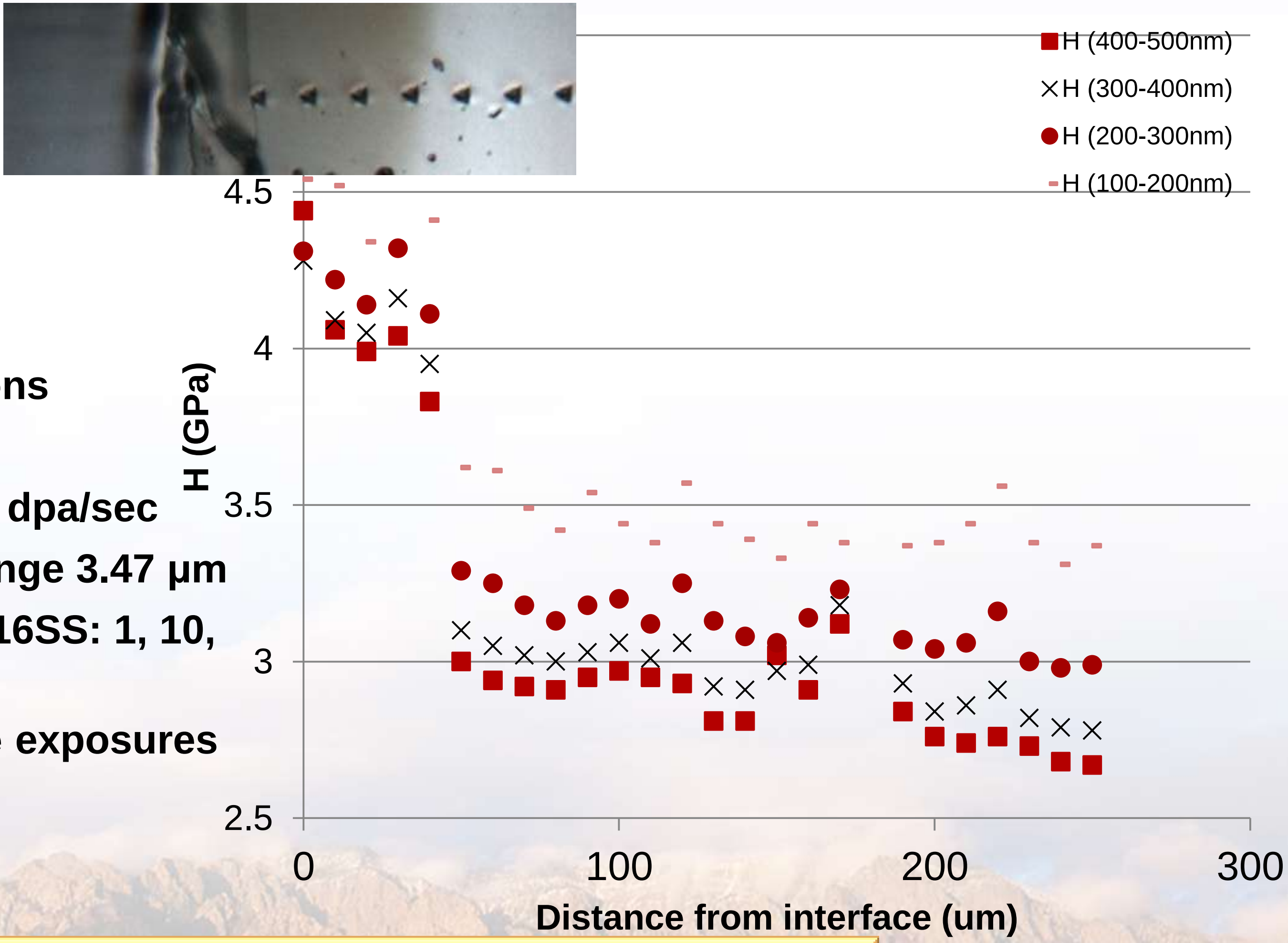


Diffusion profiles vary significantly between different couple sets

Hardness in Nickel Irradiation Zone of Mo/316 Couple

Collaborators: S.B. Van Deusen, DL. Buller, A. McGinnis, L.N. Brewer, A.J. Kilgo, J. Puskar, & S. Goods

Hardness taken from different depths

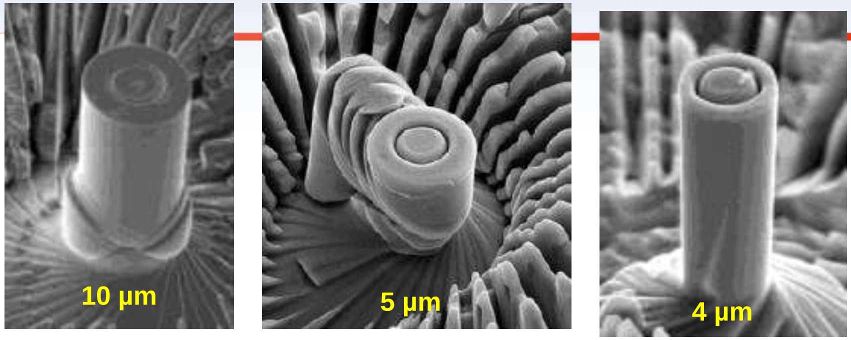


- Nickel ion beam conditions
  - 20 MeV Ni
  - Damage rate ~0.003 dpa/sec
  - Estimated end of range 3.47 µm
  - Exposures on Mo-316SS: 1, 10, and 30 dpa
  - "room" temperature exposures

Initial results indicate that heavy ion irradiation alters the toughness as a function of depth

Sandia National Laboratories

Small Scale Mechanical Testing and TEM Results

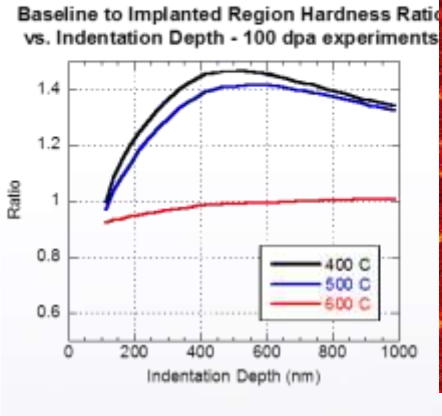


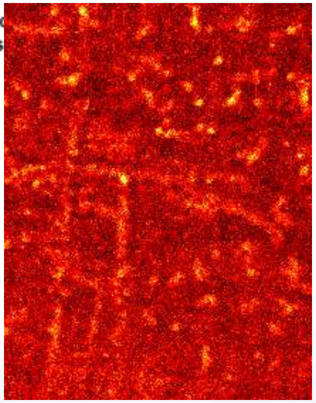
Proof of Concept:

- (100), (110), and (111) single crystal Cu
- 20 MeV Cu to approximately 100 dpa
- Micropillar compression of 162 pillars of three sizes

Validation Experiments:

- 420F, 409, and 316L SS
- 20 MeV Ni irradiation to approximately 10 dpa, 40 dpa, and 100 dpa at 400 ºC, 500 ºC, and 600 ºC



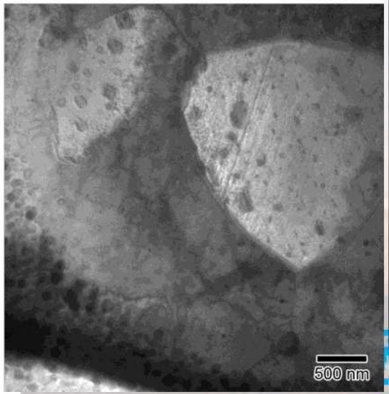


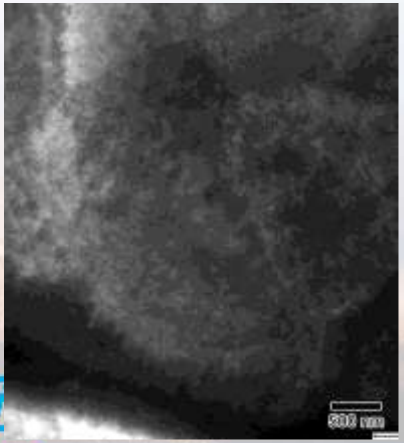
Diffusion Couple Experiments:

- W, Mo, Ta, and Nb HIP with 316L then diffusion annealed
- Select 20 MeV Ni irradiation to approximately 10 dpa, 40 dpa, and 100 dpa and temperatures of 400 ºC, 500 ºC, and 600 ºC
- W, Mo, and Ta HIP with HT9 then diffusion annealed

***In situ* TEM investigations:**

- *In situ* ion irradiation facility is operating
- *In situ* oxidation at temperature up to nominally 1200 ºC



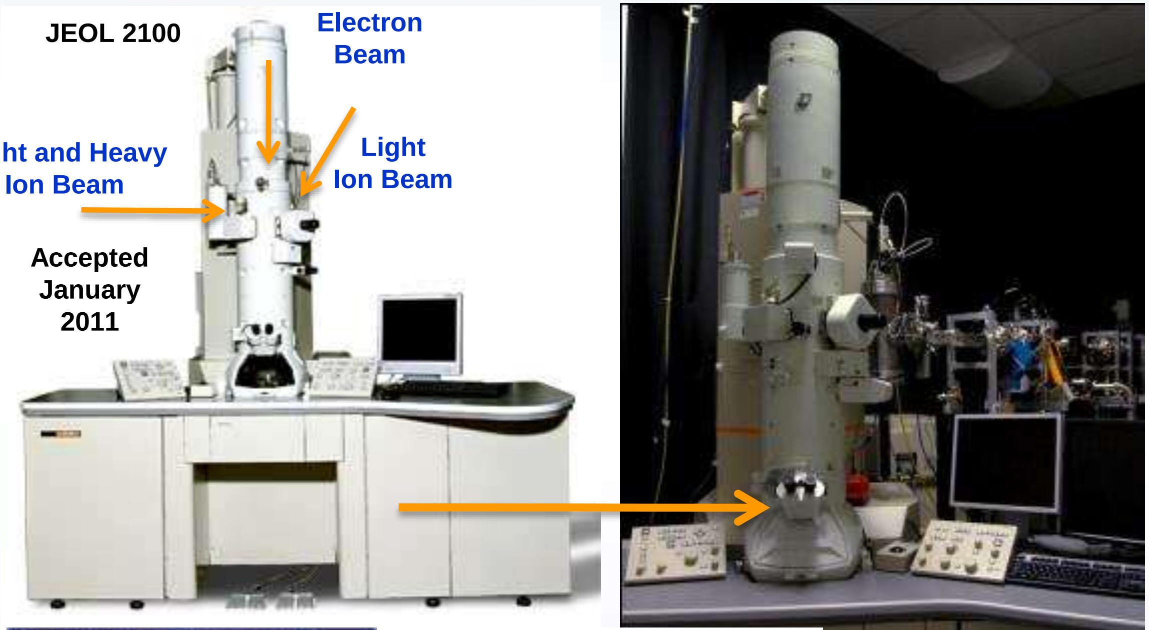


In situ Ion Irradiation TEM (I^3TEM)

Collaborators: D. Buller, B.G. Clark, & B.L. Doyle

Proposed Capabilities

- 200 kV LaB_6 TEM
- Ion beams considered:
  - 3 MeV He^{1+} Si^{3+} Cu^{3+} Au^{3+}
  - 14 MeV Si^{3+}
  - 10 keV D^{2+}
  - 10 keV He^{+}
  - All beams will hit same location
- Electron tomography
- Nanosecond time resolution (DTEM)
- Precession scanning (EBSD in TEM)
- *In situ* PL, CL, and IL
    - Optical pathway purchased
- *In situ* heating and cooling stage
- *In situ* electrical measurement stage
- *In situ* straining stage
- *In situ* vapor phase stage
- *In situ* liquid mixing stage



JEOL 2100

Electron Beam

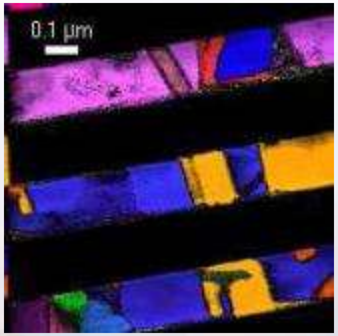
Light and Heavy Ion Beam

Light Ion Beam

Accepted January 2011

TVIPS

Hummingbird

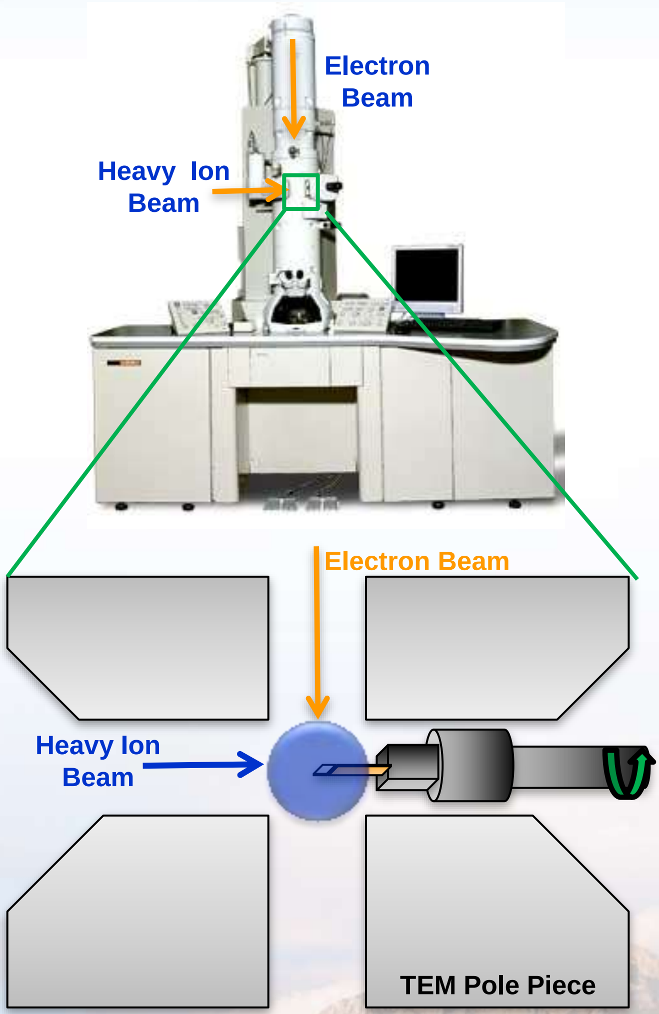


Nanomegas

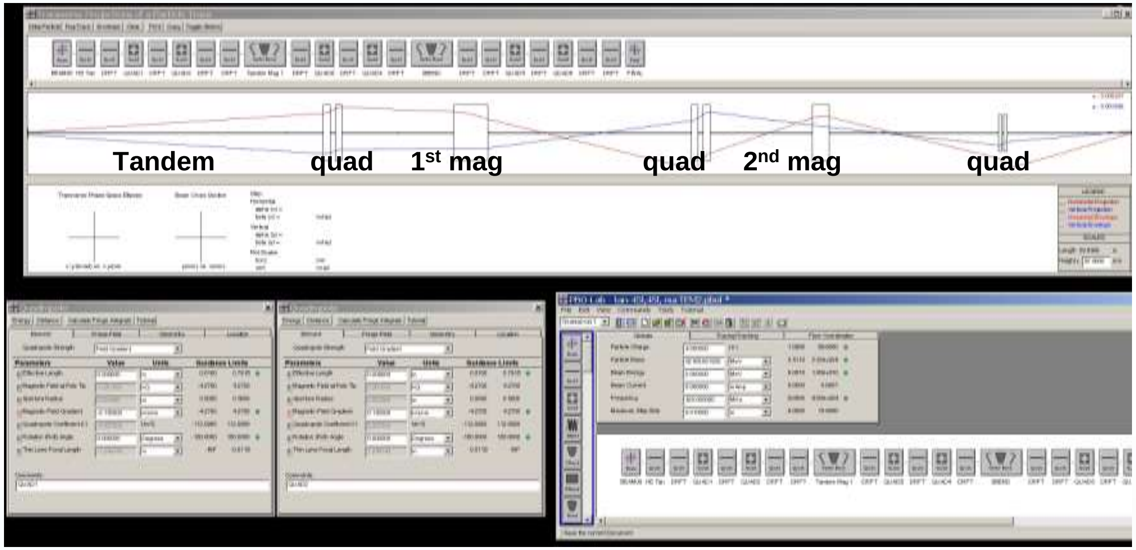
We have completed the 1st generation (Tandem Accelerator) and have initiated building the 2nd generation (Colutron Accelerator). Many potential additions are being considered

Beam Optic Models and Image Simulation of the *In situ* Ion Irradiation TEM

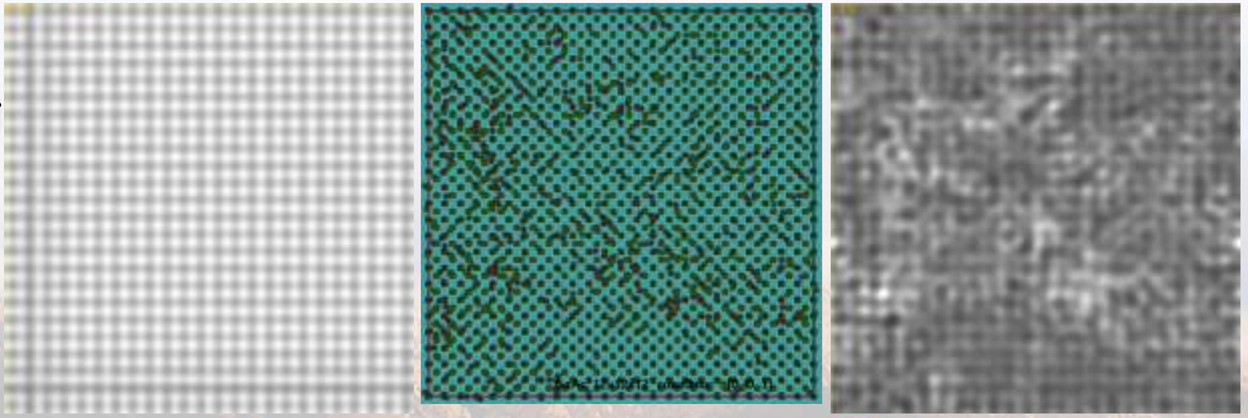
Collaborators: P. Rossi, D.L. Buller, & B.L. Doyle



Ion Optics Feasibility Study



TEM image simulation of radiation damage



JEMS undamaged

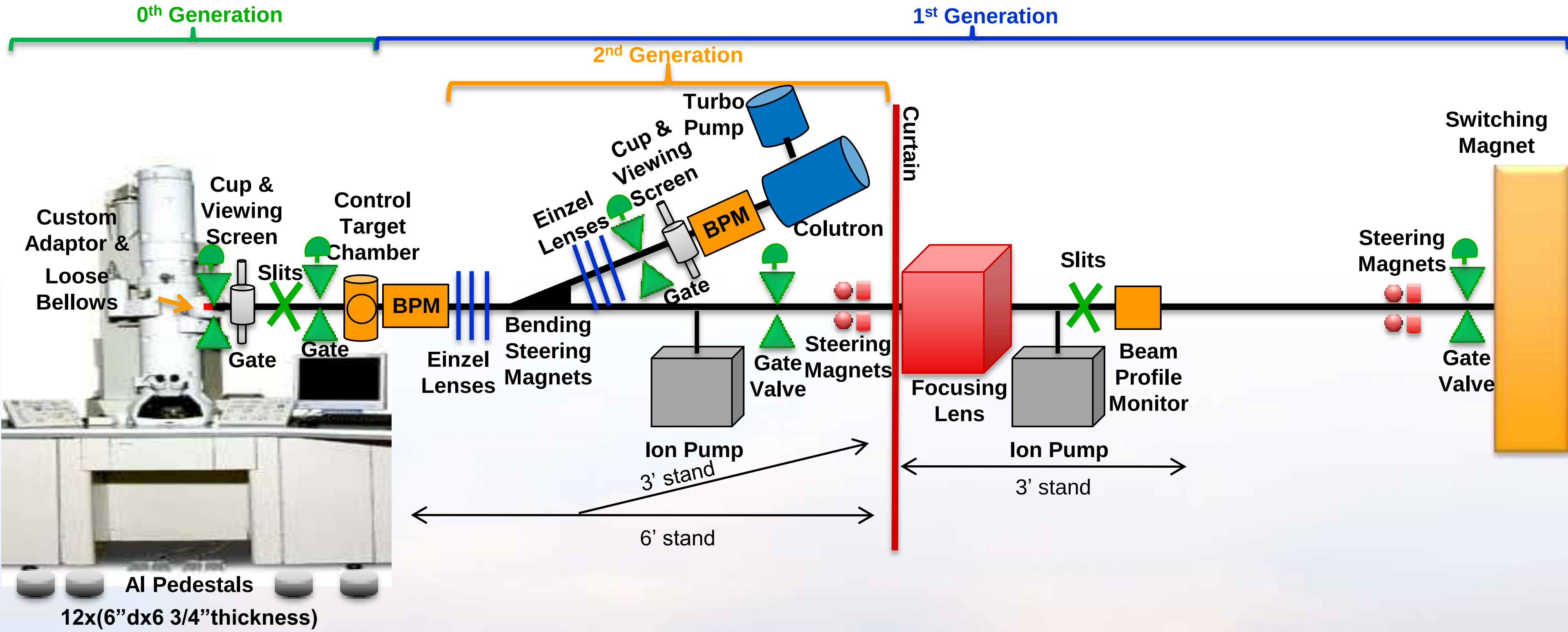
Marlowe

JEMS damaged

Simulation (Marlowe+JEMS) shows the project is viable (30 MeV Cu -> GaAs)

Schematic of the *In situ* TEM Beamline

Collaborators: D. Buller & B.L. Doyle



0th Generation

1st Generation

2nd Generation

Custom Adaptor & Loose Bellows

Cup & Viewing Screen

Slits

Gate

Gate

Control Target Chamber

BPM

Einzel Lenses

Bending Steering Magnets

Einzel Lenses

Cup & Viewing Screen

BPM

Gate

Turbo Pump

Colutron

Ion Pump

Gate Valve

Steering Magnets

Curtain

Focusing Lens

Slits

Beam Profile Monitor

Ion Pump

Steering Magnets

Gate Valve

Switching Magnet

3’ stand

6’ stand

3’ stand

Al Pedestals

12x(6”dx6 3/4”thickness)

Planned Development of the I^3TEM

Collaborators: D. Buller, B.G. Clark, & B.L. Doyle

0th Generation

- 200 kV LaB_6 TEM
- *In situ* vapor phase stage
- *In situ* liquid mixing stage
- Simple tomography holder

Delivery October 29, 2010 &
Accepted January 31, 2011

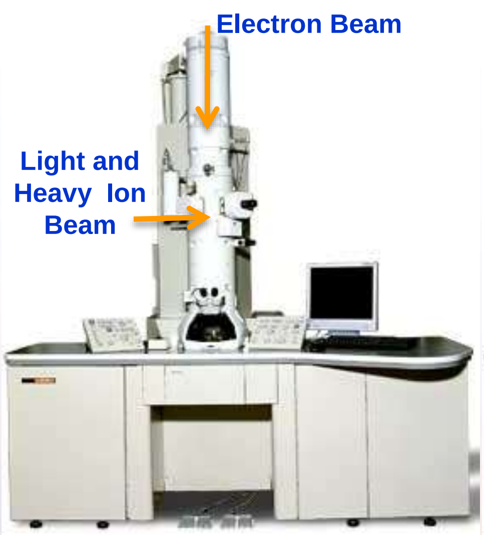
1st Generation

- Insertion of collimated Tandem ion beam at 0-9º
- Vacuum, electrical, and mechanical Isolation
- Adequate dosimetry

First beam into TEM April 19, 2011

2nd Generation

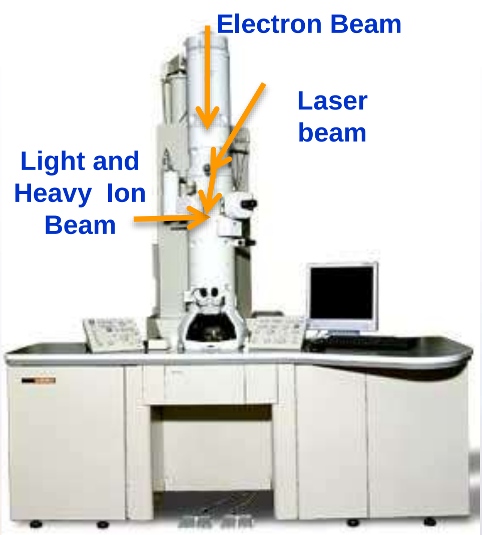
- Concurrent heavy and light ion implantation
- A Colutron ion beam will be bent in line with the Tandem beam.



Construction nearly complete

3rd Generation

- Plan-view laser exposure and DTEM capabilities
- Provide ion and laser excitation sources with nanosecond timing

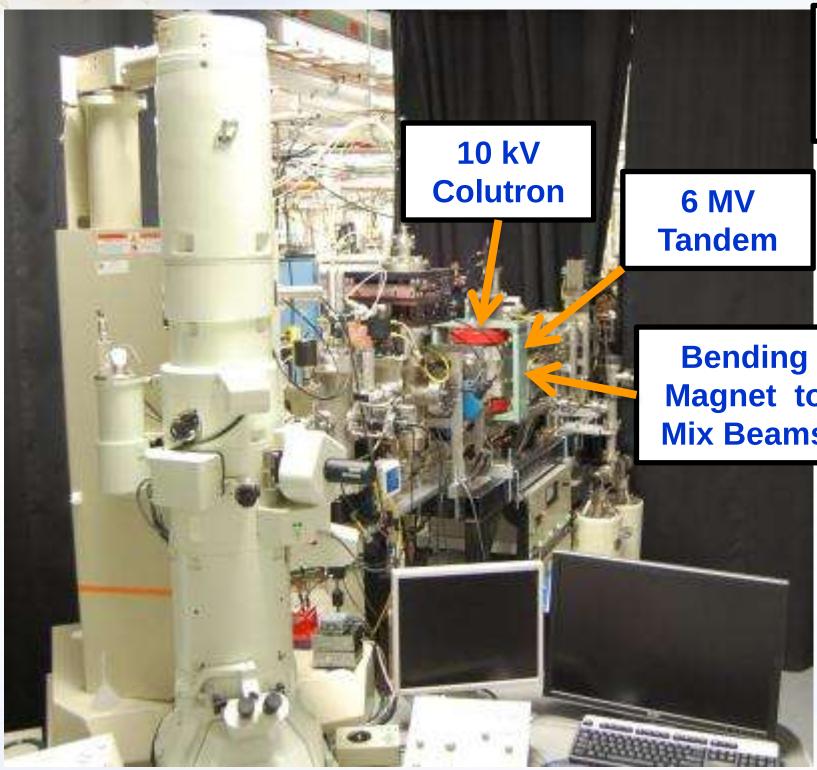


Construction design determined

Each facility addition will expand potential capabilities

Current Status of the *In situ* TEM Beamline

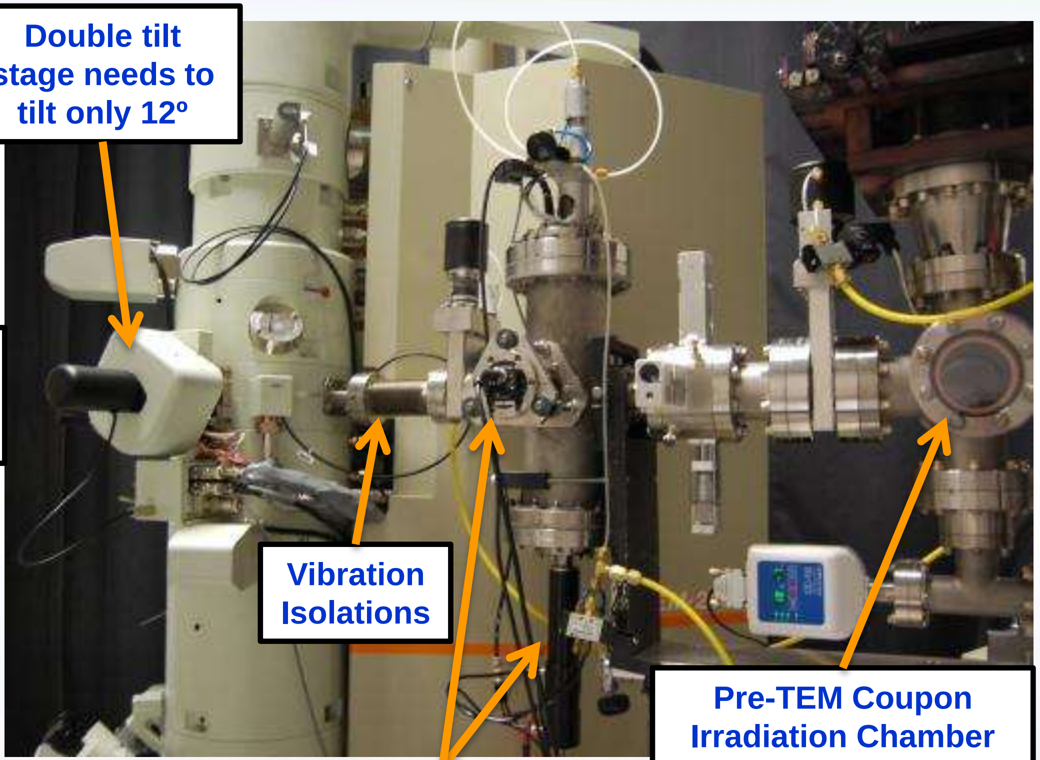
Collaborators: D. Buller & J.A. Scott



10 kV Colutron

6 MV Tandem

Bending Magnet to Mix Beams



Double tilt stage needs to tilt only 12º

Vibration Isolations

Faraday Cup and Viewing Screen

Pre-TEM Coupon Irradiation Chamber

Beam burn from 14 MeV Si

0th and 1st Generation are completed and operated regularly

2nd Generation was assembled and placed under vacuum on 12/23/2011

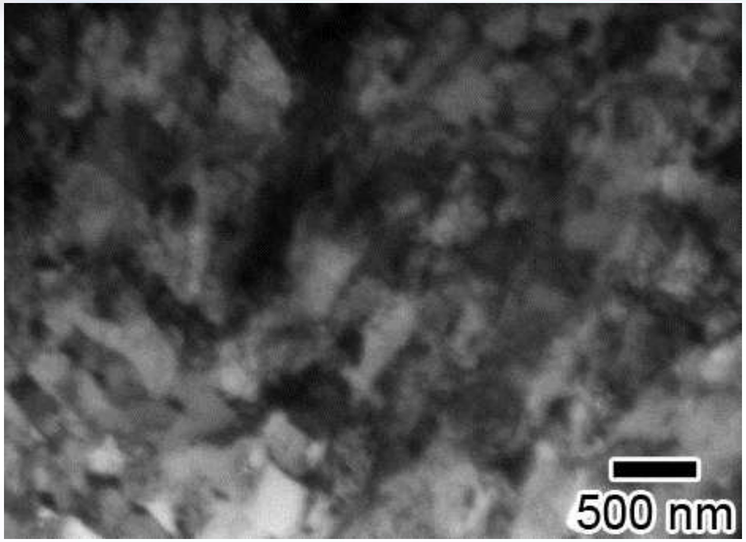
We hope to have concurrent heavy and light ion irradiation facility operational in 2012

In situ TEM Ion Irradiation

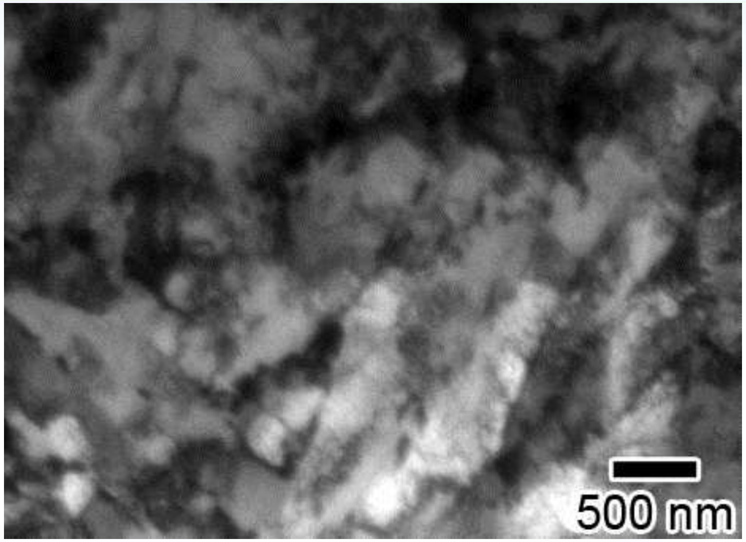
HT9   3 MeV Cu^{3+} at ~10 nA   RT

Collaborators: A.C. Kilgo, J. Puskar, & S. Maloy

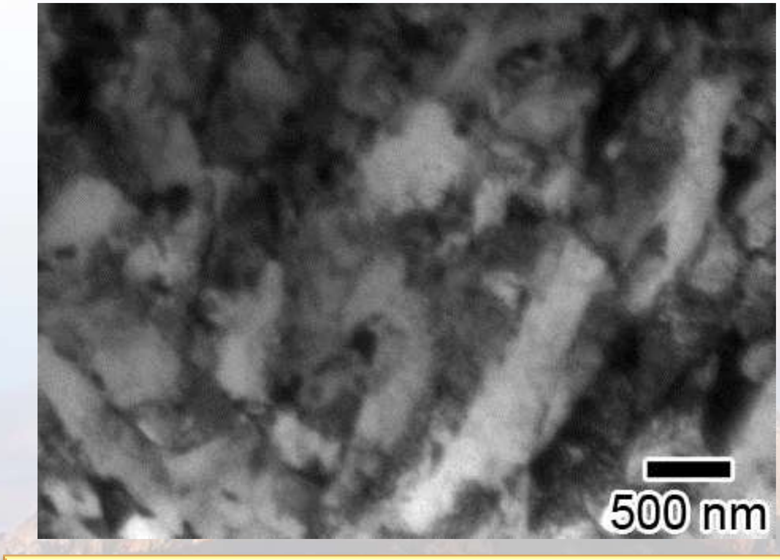
Initial



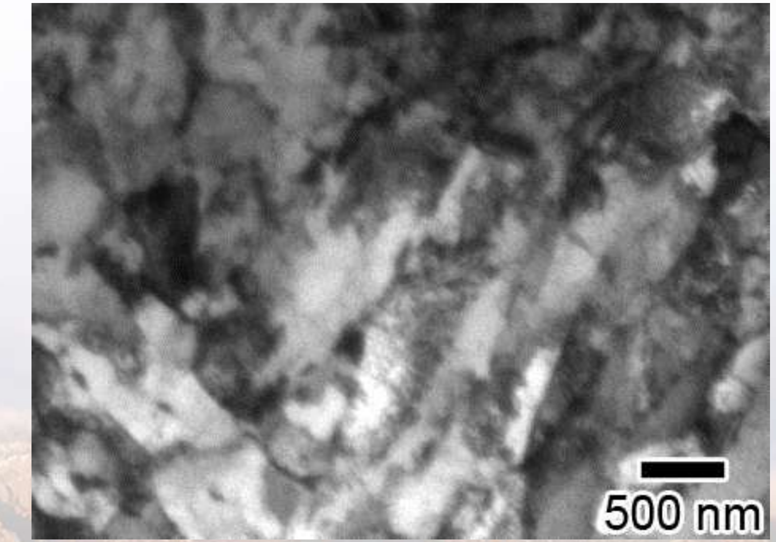
2 hrs



3 hrs



6 hrs



These initial studies show the I^3TEM facility is operational

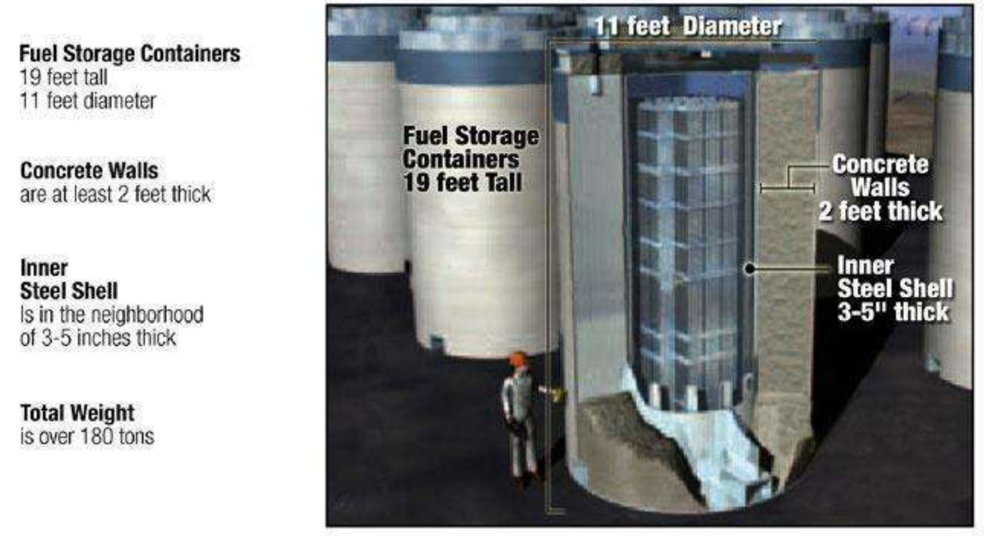
Without Yucca Mountain, What State Will Fuels and Cladding be in After ~100 Years?

- Protocol for interim storage of spent nuclear fuels (SNF) is dry storage
- Currently there are 121 storage sites (at both active and decommissioned reactors), and 2,000 MT/yr of new waste
- With no permanent solution for storage, SNF must remain in ‘retrievable’ condition <u>indefinitely</u>
- During storage, degradation of materials could lead to loss of material integrity
- Trend toward high burn-up fuels complicates the issue

What will be the materials state hundreds of years from now?

Dry storage casks at Diablo Canyon, SLO county

Dry storage of fuels, schematic



Fuel Storage Containers
19 feet tall
11 feet diameter

Concrete Walls
are at least 2 feet thick

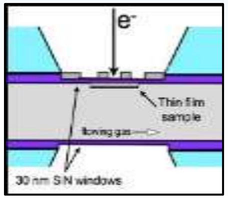
Inner Steel Shell
Is in the neighborhood of 3-5 inches thick

Total Weight
is over 180 tons

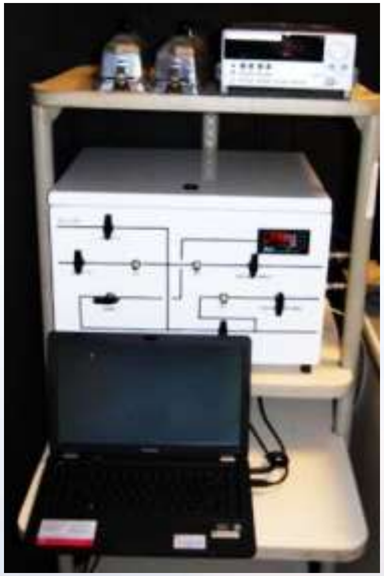
Dry Storage of Used Nuclear Fuels: Fundamental Studies of Cladding

Collaborators: B.G. Clark & Protochips Inc.

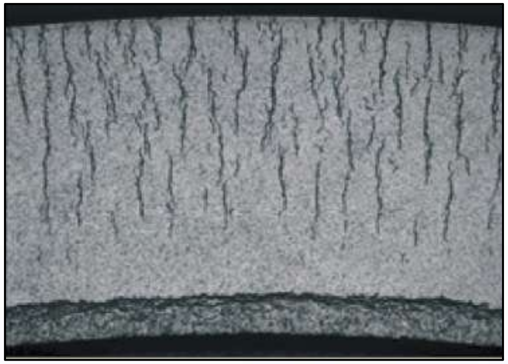
Evaluate electrochemical properties of hydrides and mechanisms of enhanced corrosion



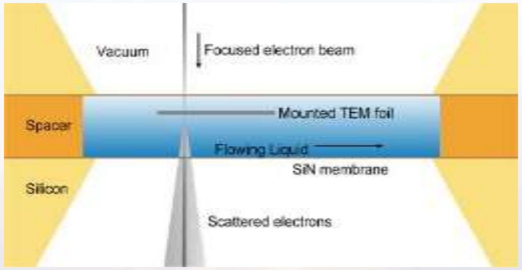
Quantify effect of irradiation on solubility of H and evaluate pick up mechanism



Analyze variables influencing hydride reorientation mechanism



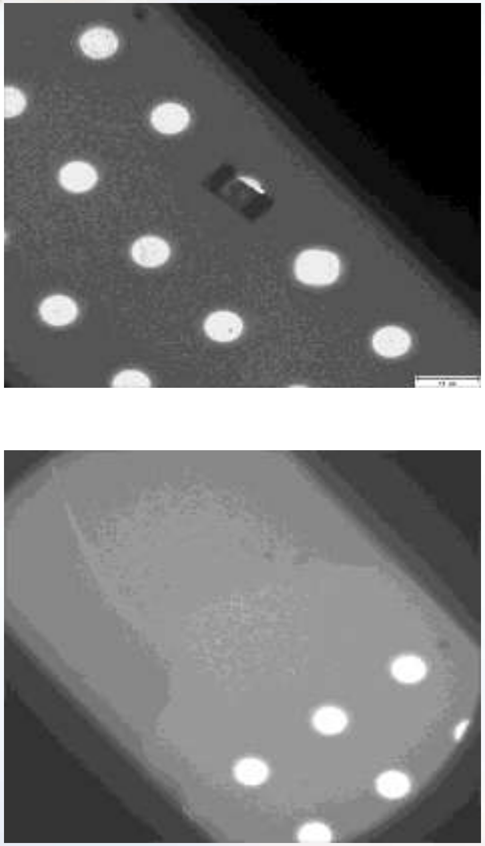
Analyze microstructures as function of alloy and burn-up to create simulated microstructures for advanced testing



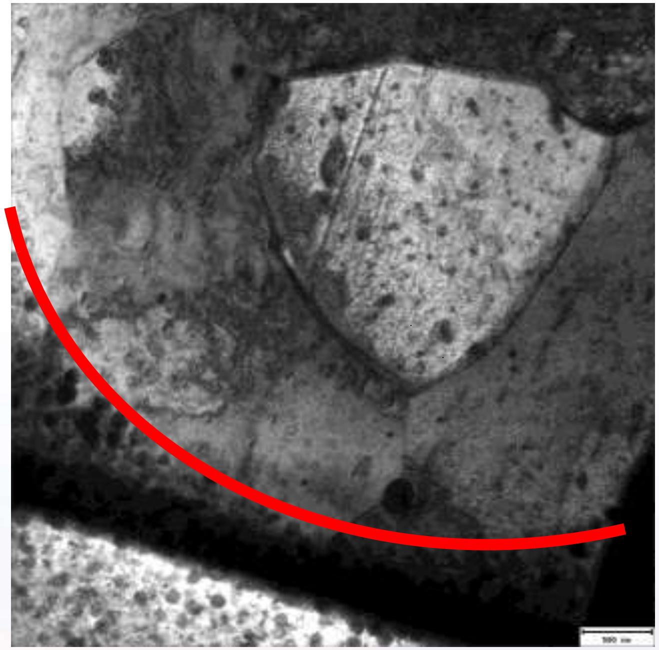
Understanding fundamental mechanisms will directly impact the design and use of dry storage methods

Feasibility of Studying Zircaloy 2 at Nominally 1 atm

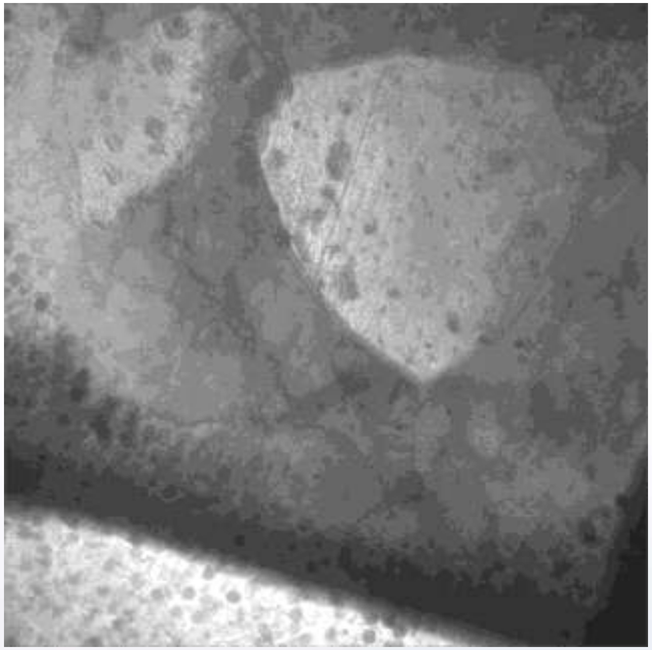
Collaborators: S. Rajasekhara & B.G. Clark



Vacuum &
Single Window



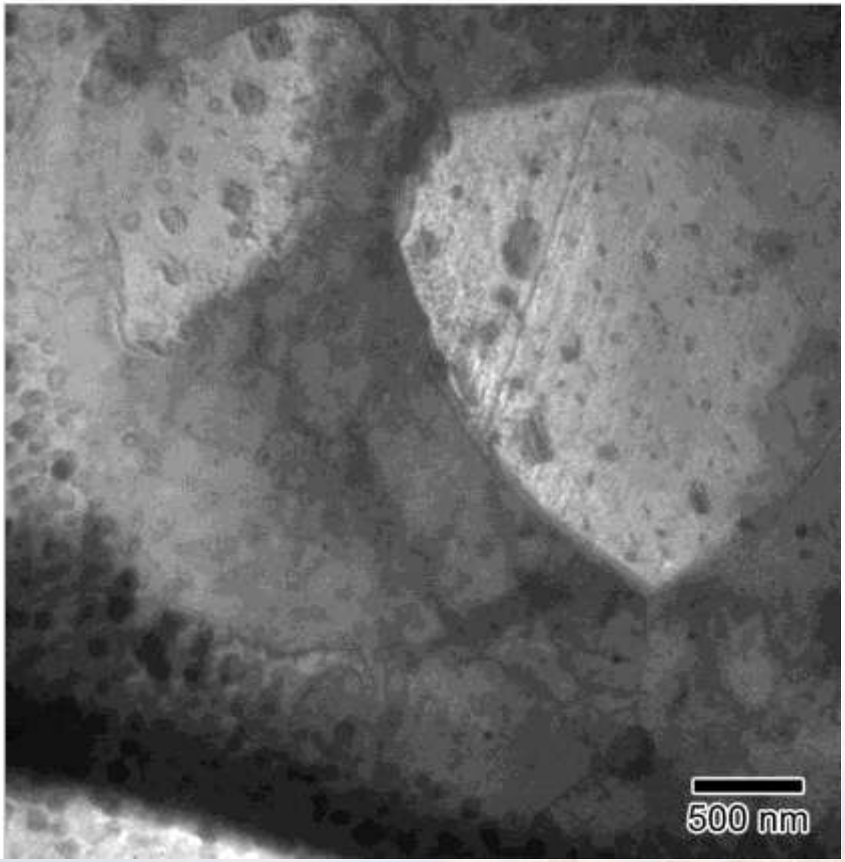
Nominally 1 atm &
Two Windows

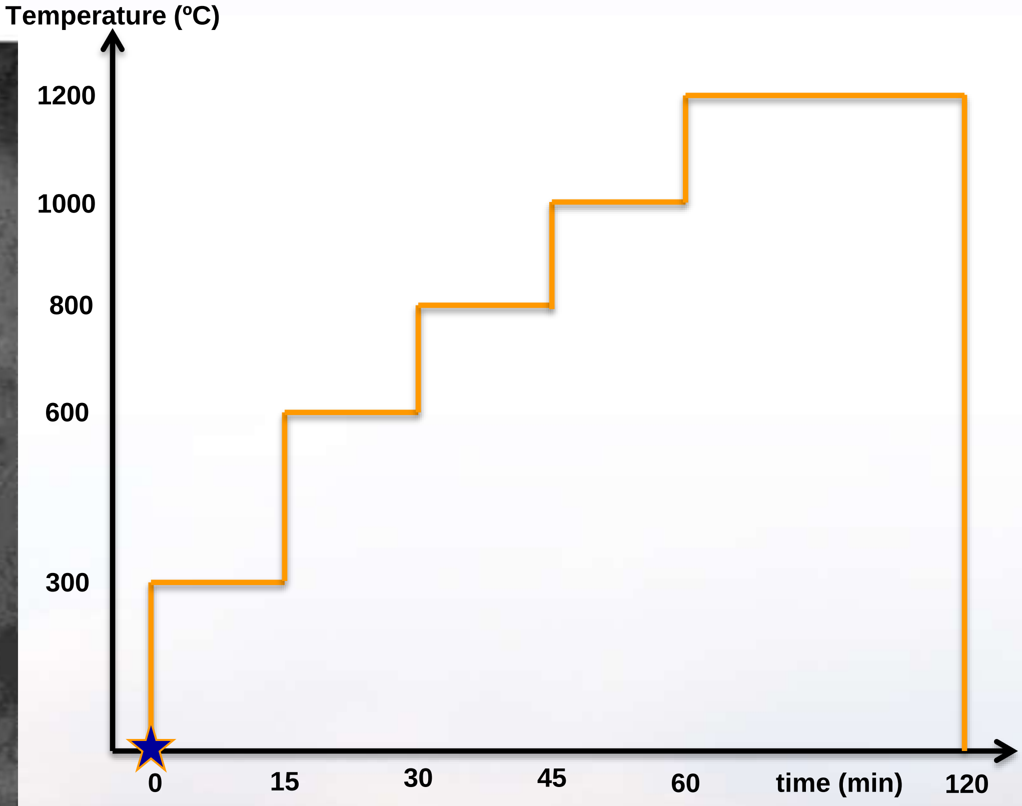


Most features are observed in both despite the decreased resolution resulting from the additional SiN window and 5 µm of air

In situ TEM Heating of Zircaloy

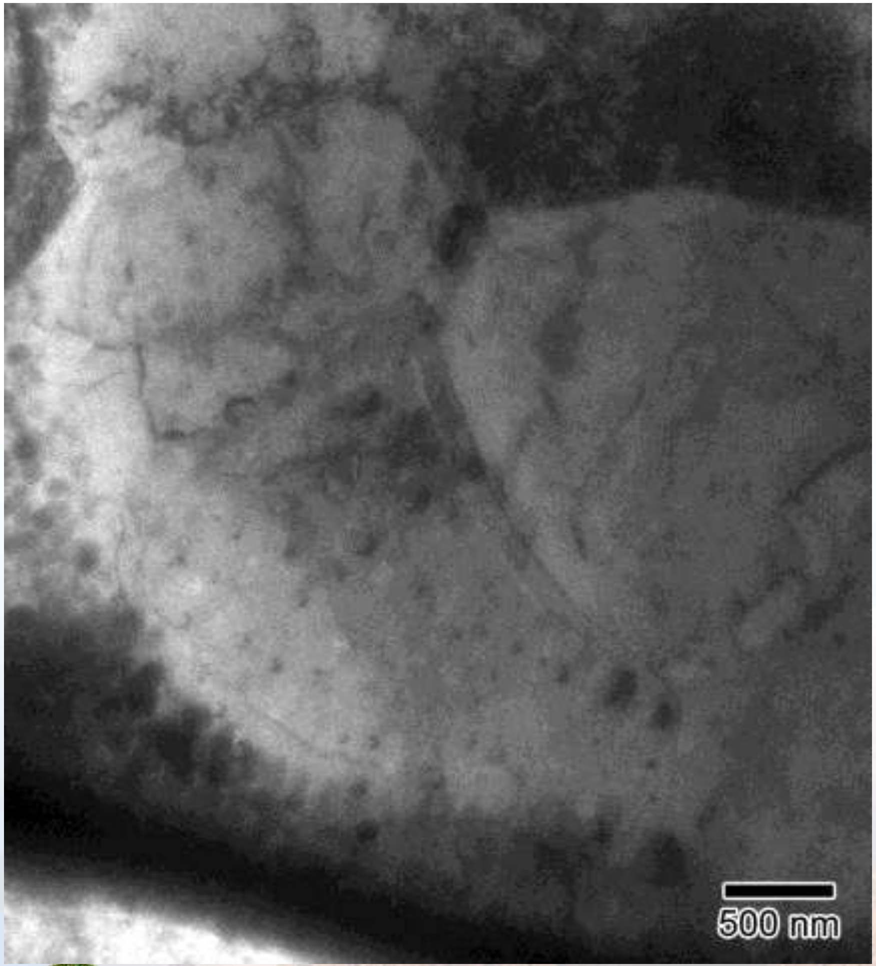
Collaborators: S. Rajasekhara & B.G. Clark

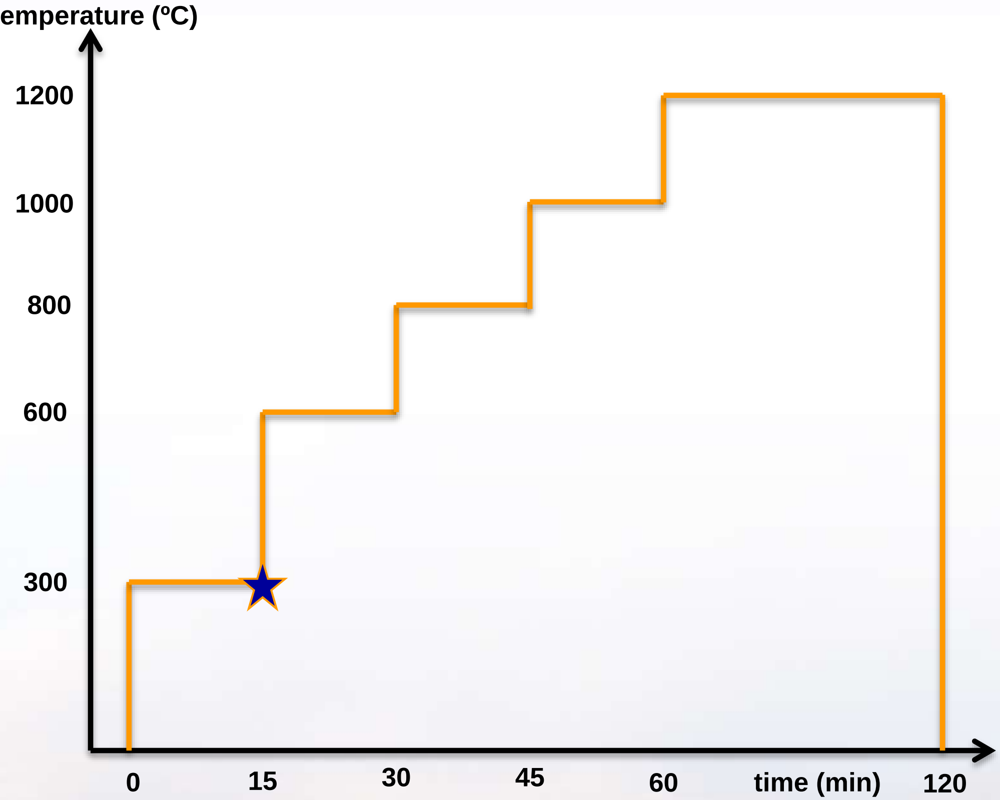




In situ TEM Heating of Zircaloy

Collaborators: S. Rajasekhara & B.G. Clark

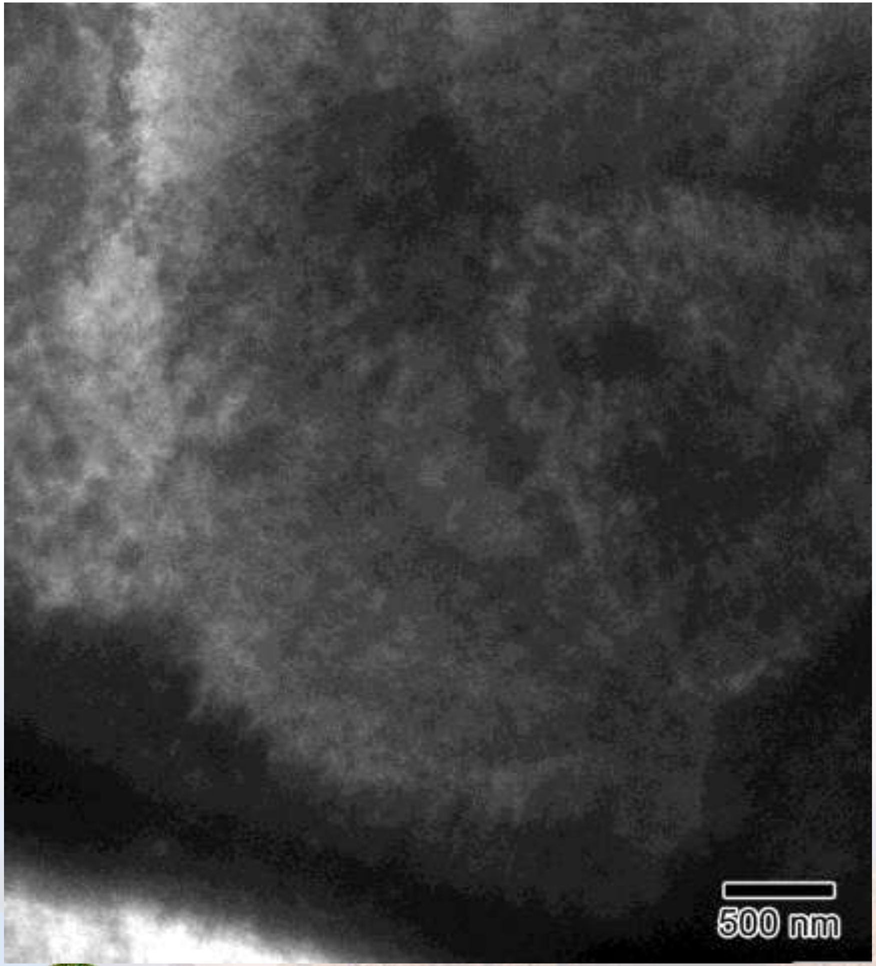


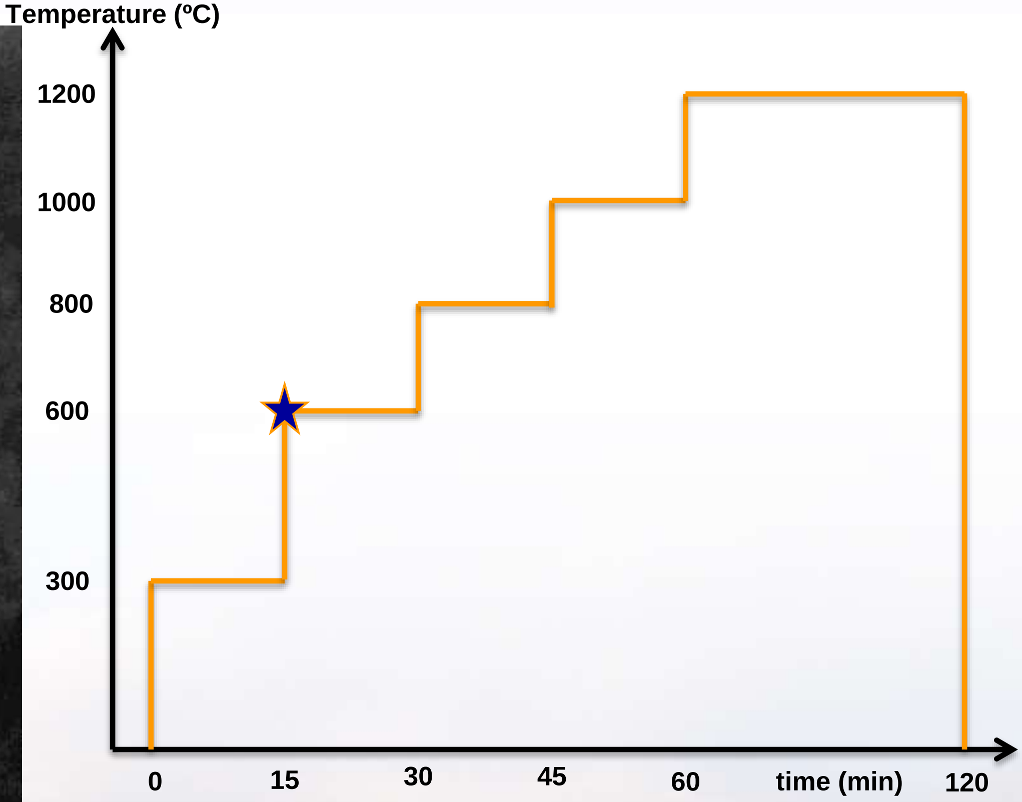


Some of the contamination appears to lessen

In situ TEM Heating of Zircaloy

Collaborators: S. Rajasekhara & B.G. Clark

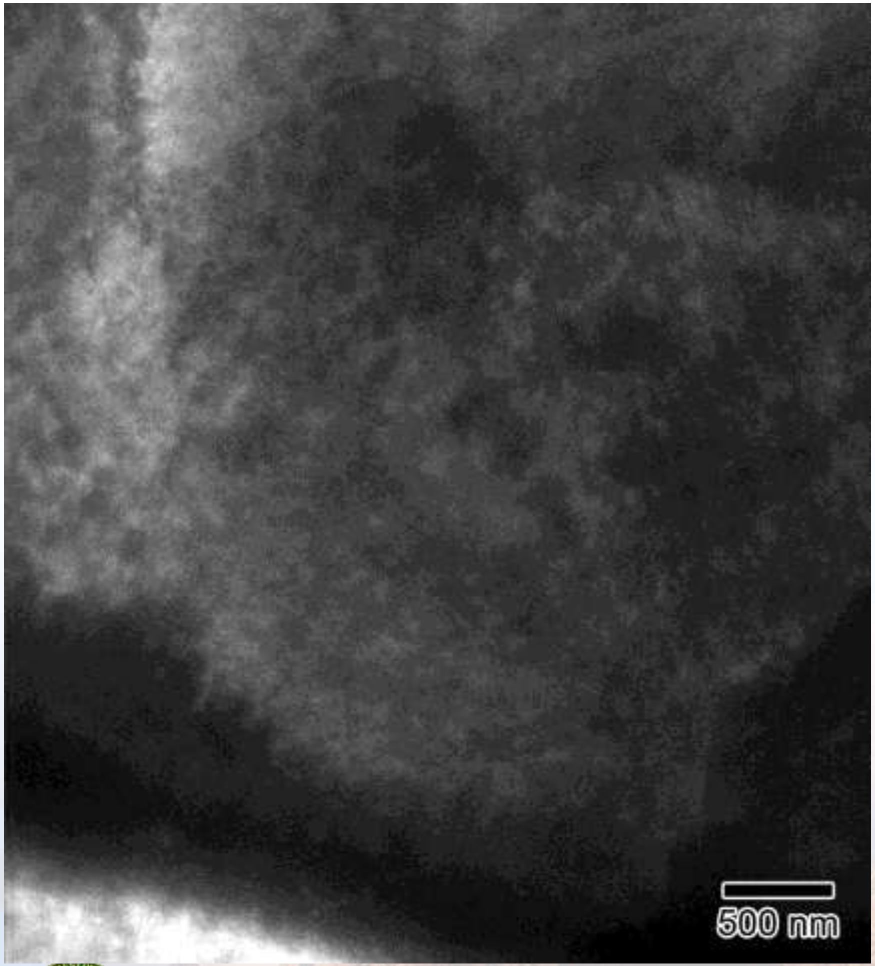


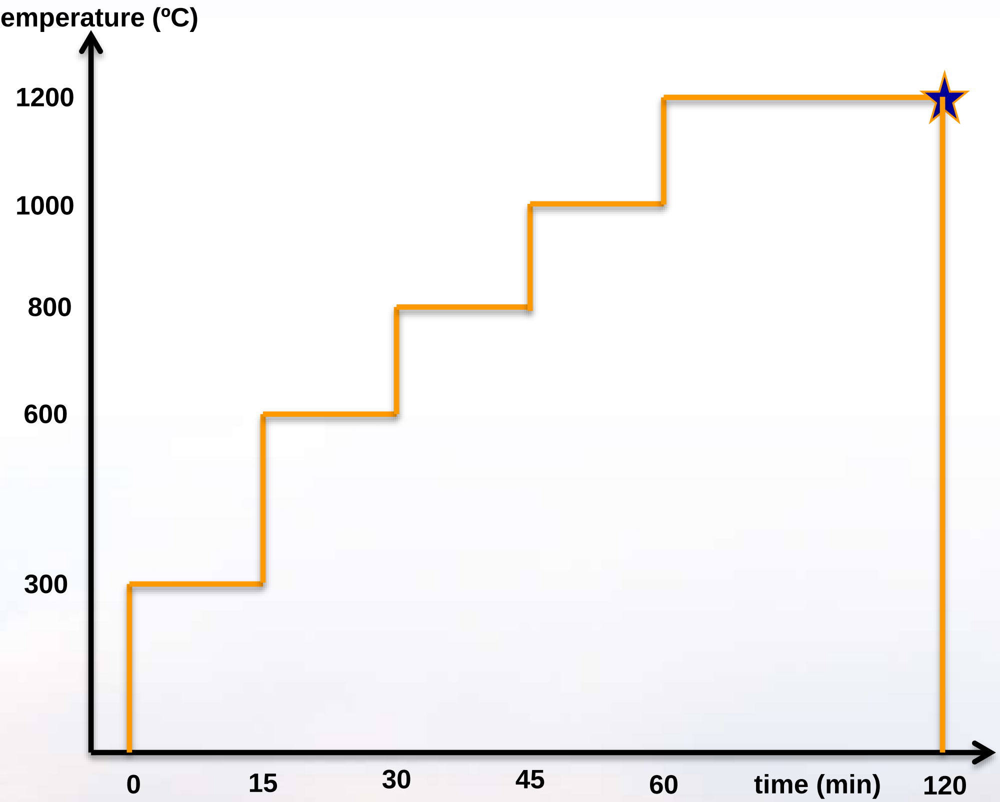


Drastic microstructural evolution due to exponential oxidation

In situ TEM Heating of Zircaloy

Collaborators: S. Rajasekhara & B.G. Clark

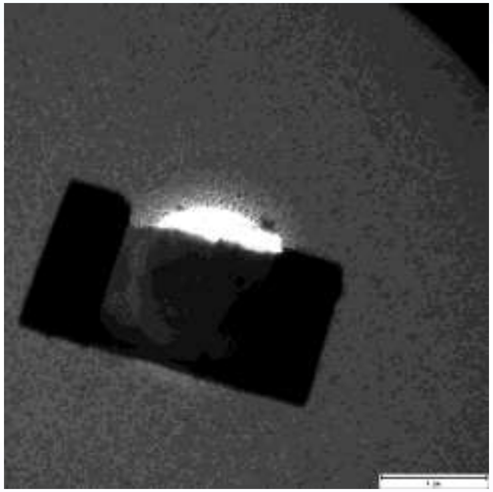




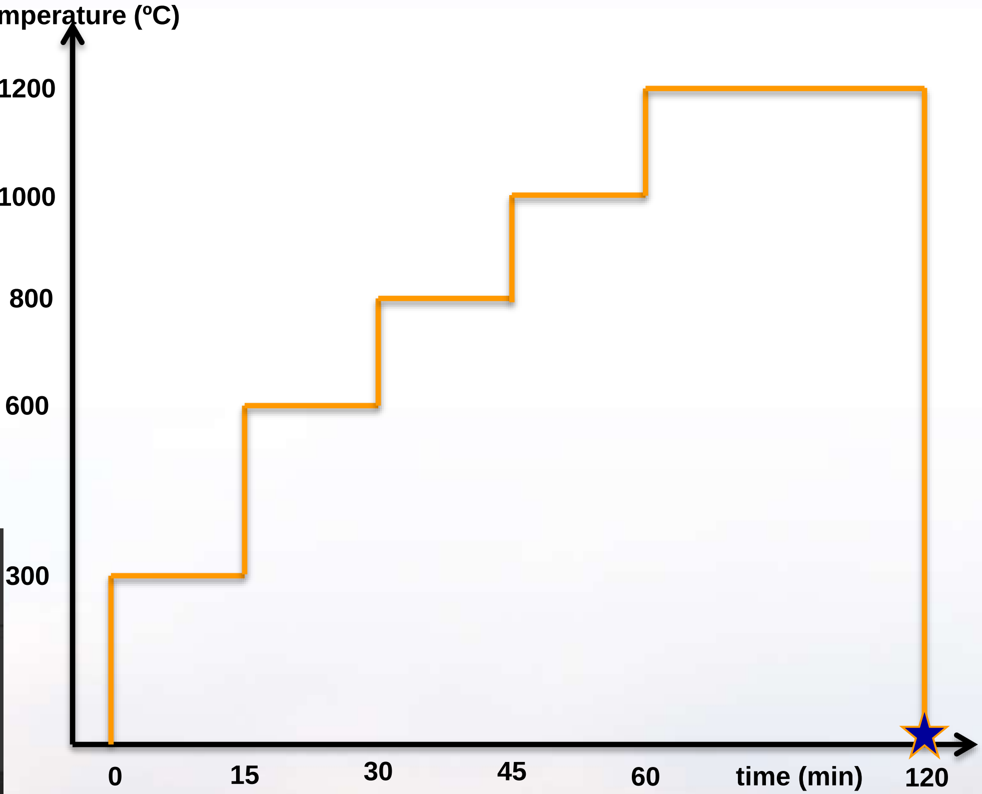
Both stage and FIB foil are stable for 1 hour at 1200 ºC

Nominal Temperature and Pressure

Collaborators: S. Rajasekhara & B.G. Clark



Temperature based on Protochips calibration was not independently verified



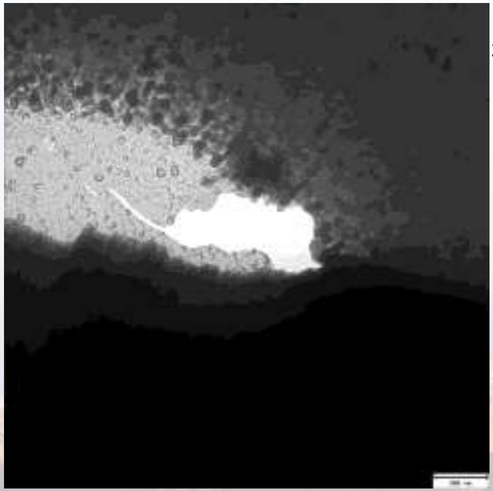
Pressure is between 84,000 Pa

(ABQ atmospheric pressure)

and

3.2 x 10^{-4} Pa

(Poor TEM column Pressure)

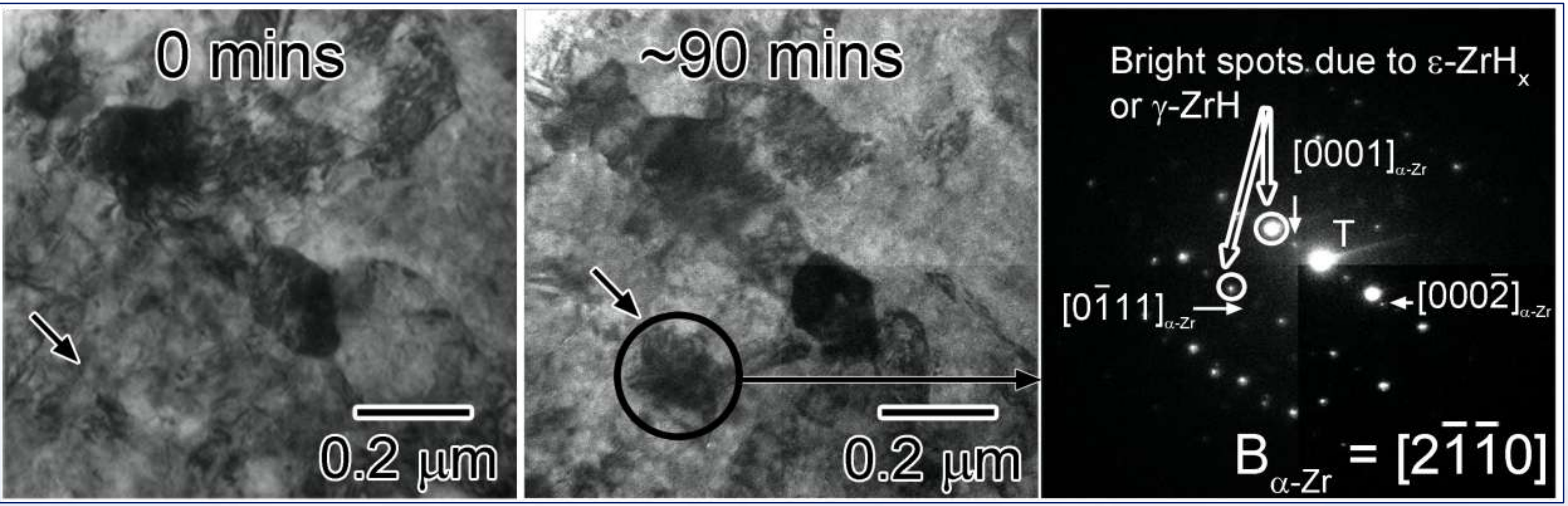


Temperature and Pressure reported have yet to be calibrated

In situ Observation of Hydride Formation in Zirlo

Collaborators: S. Rajasekhara & B.G. Clark

Absolute hydrogen pressure: 327 torr (~ 0.5 atm),
Ramp rate: 1 ^{o}C/s, Final temperature: ~ 400 C, Dwell time: ~ 90 mins



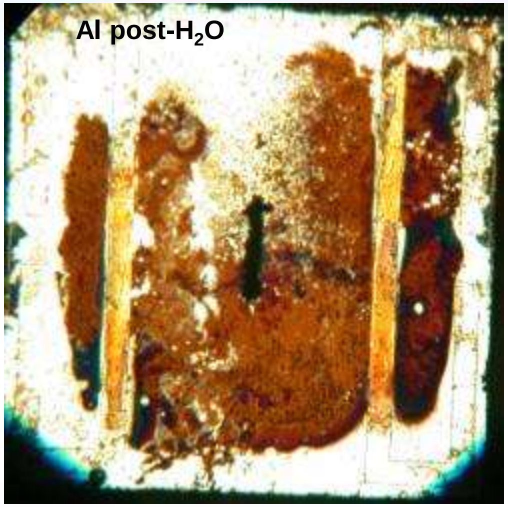
Hydride formation shown, for the first time by use of a novel TEM gas-cell stage, at elevated temperature and hydrogen pressure

Aluminum Appears to Wash off of Si Chips

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

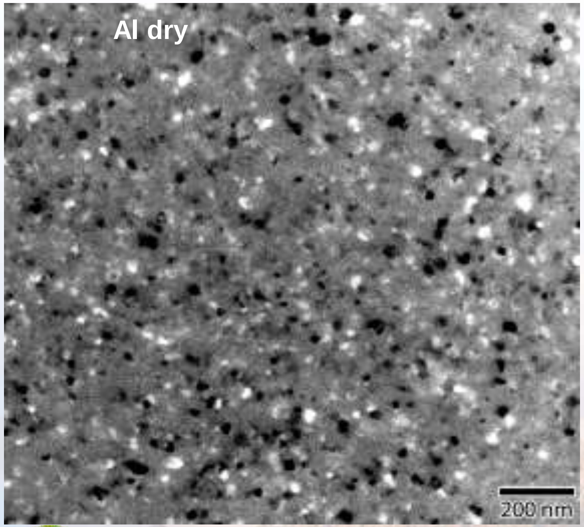
Little change seen in TEM when H_2O flowed over Al, but significant change to E-Chip surface

Change seen in TEM when saturated NaCl solution flowed with almost no Al visible on E-Chip afterwards

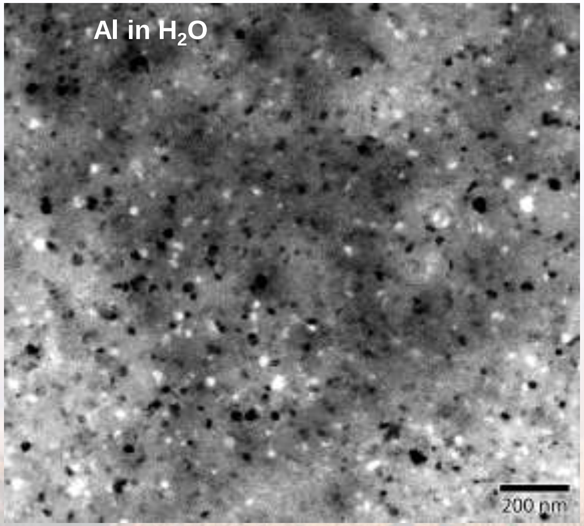


Al post-H_2O

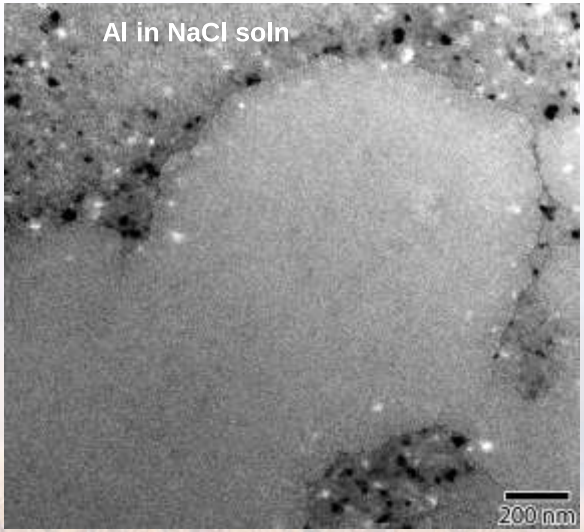
Al post-NaCl



Al dry



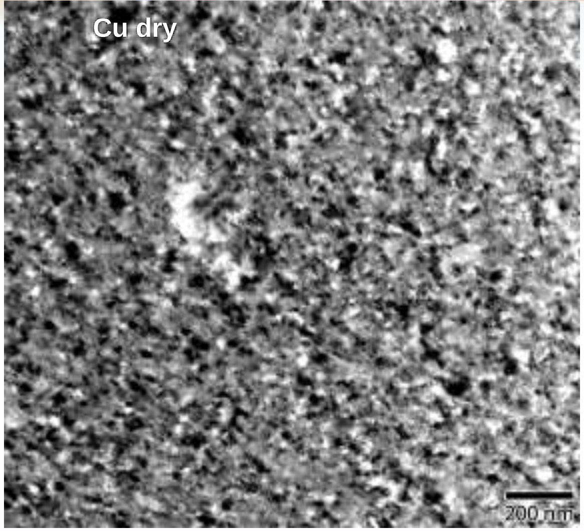
Al in H_2O

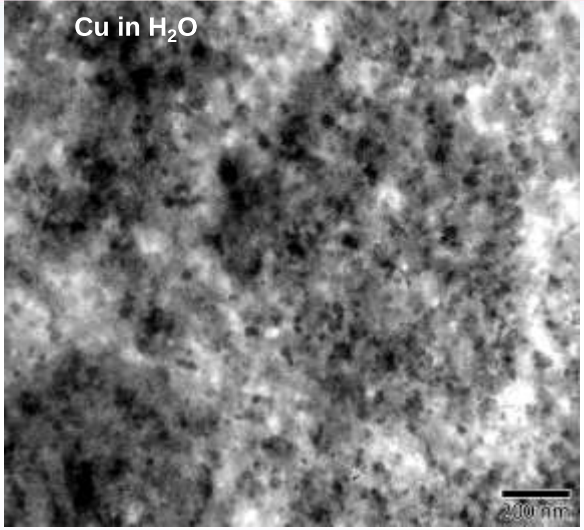


Al in NaCl soln

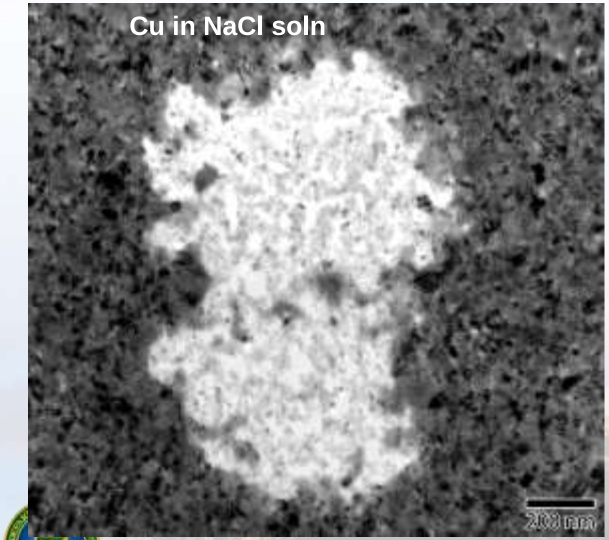
Copper Appears to Oxidize and Pit

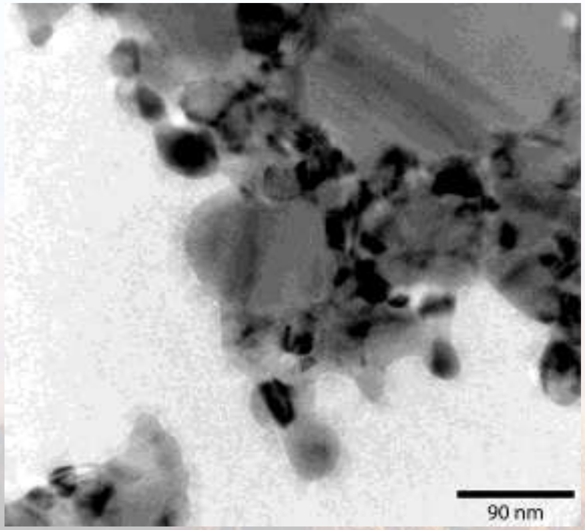
Collaborators: S.M. Hoppe & B.A. Hernandez-Sanchez


Cu dry


Cu in H_2O


Cu post-H_2O

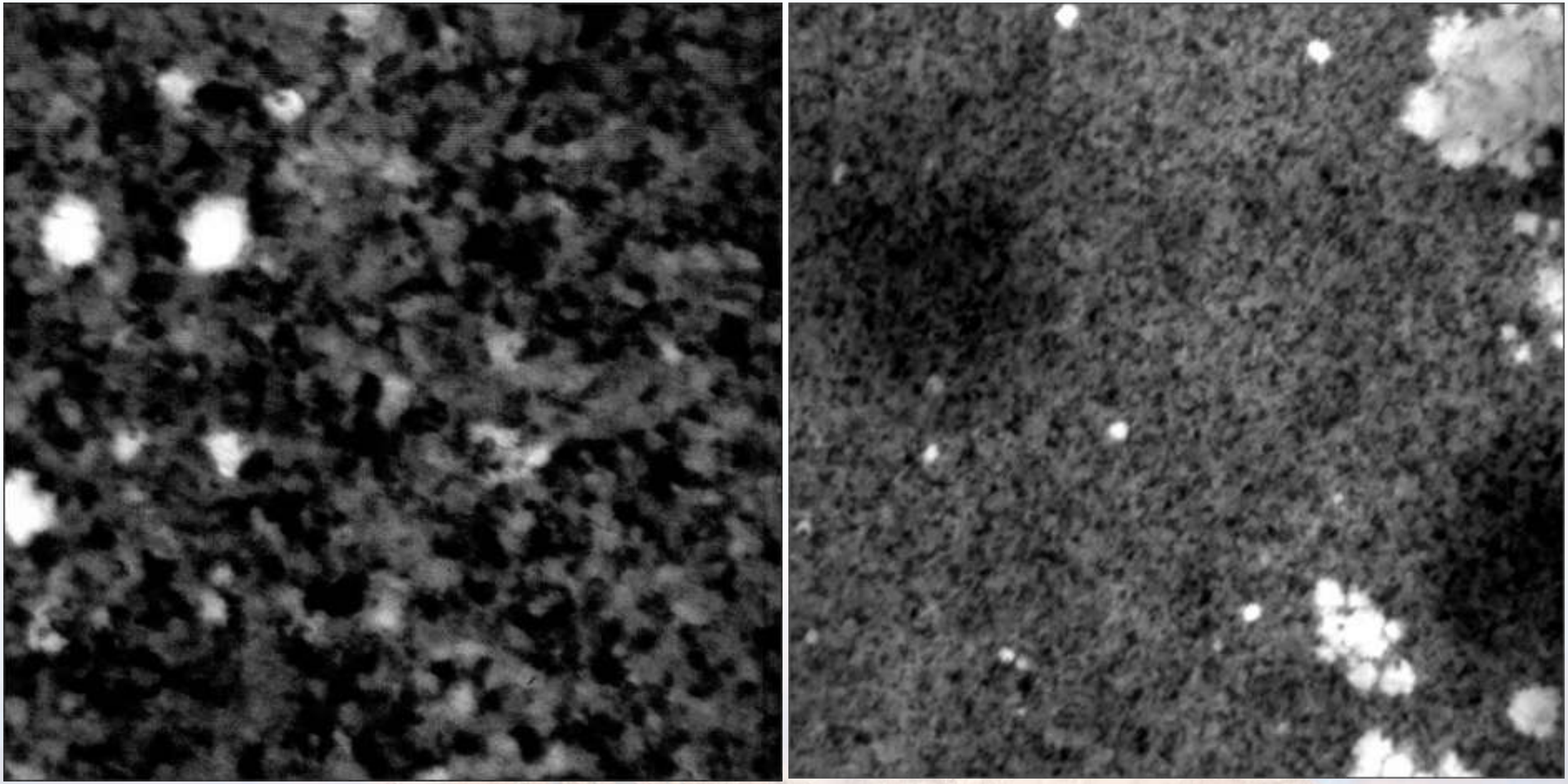

Cu in NaCl soln




Cu post-NaCl

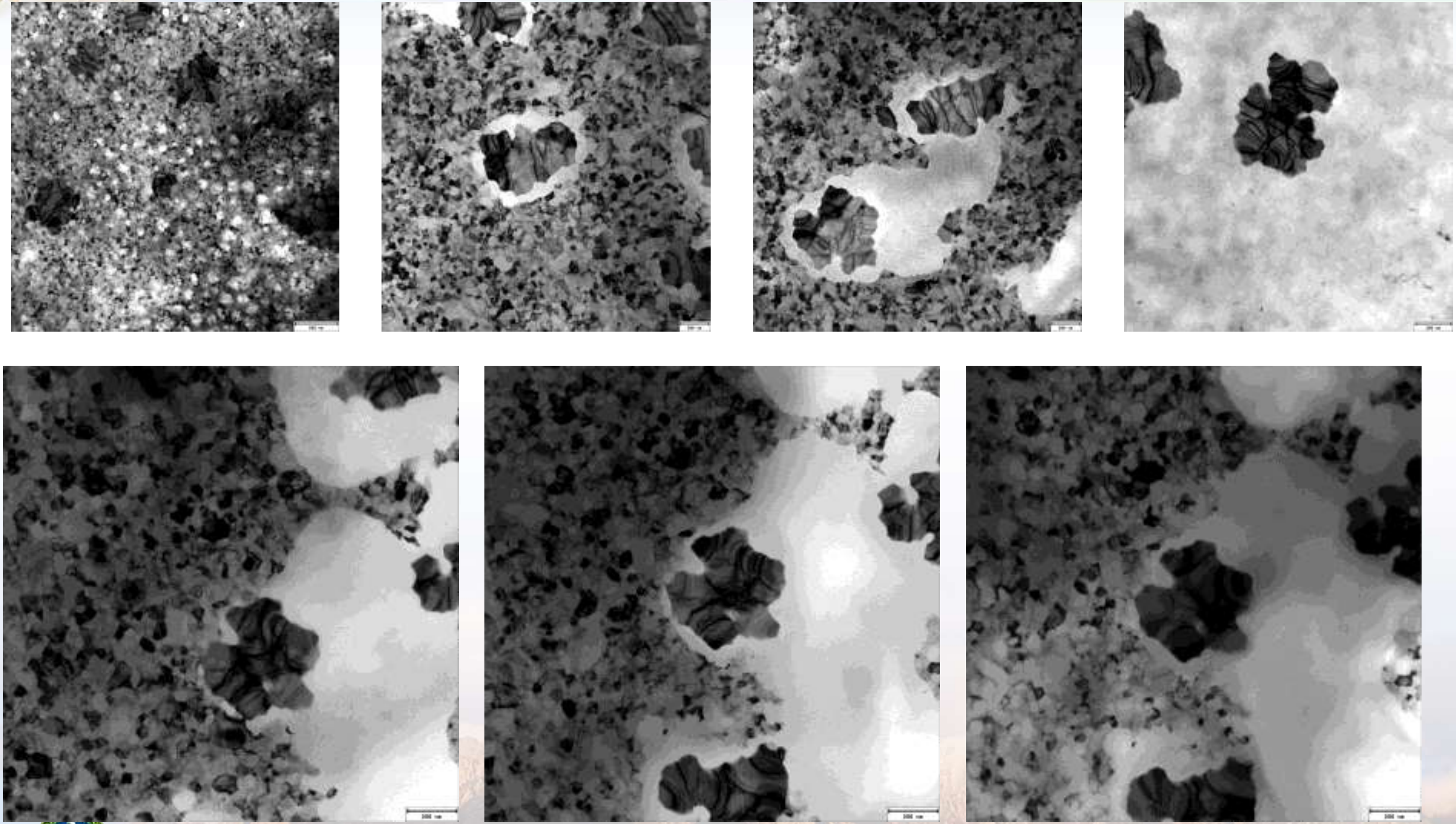
Acetic Acid Corroding Nanograined Iron

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



Acetic Acid Corroding in Annealed Nanograined Iron

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



Advanced Applications of Scintillators

Existing single crystal γ-ray scintillators suffer from performance and reliability issues

Current Issues

1) Long decay times
2) Crystal anisotropy
3) Low energy resolution (low luminosity & poor linearity)
4) Complicated synthesis (single crystal growth)
5) Chemical instability (hygroscopic)

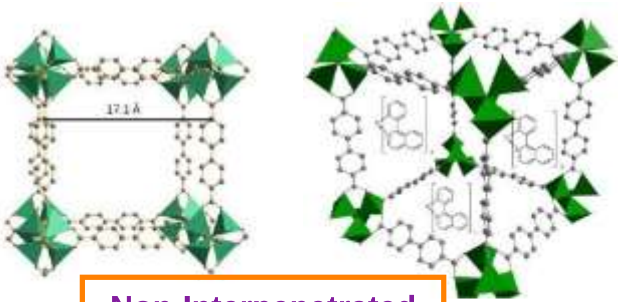
US Ports:

2 Billion Metric Tons

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

Interest in Scintillators

Crucial to understand radiation-solid interactions on multiple length scales



Non-Interpenetrated IRMOF-10

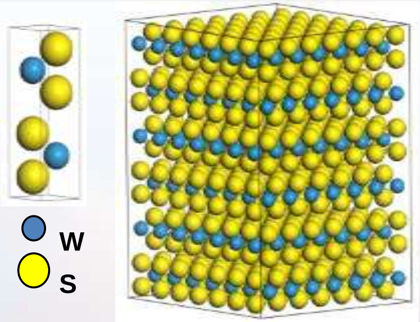
Single Crystal $CdWO_4$

Plastic Scintillators

Nano

Micro

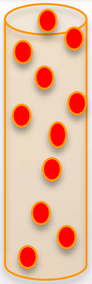
Meso



Tungsten (IV) Sulfide WS_2

Structure of High-Z ME_x Nanoscintillator

Commercial $CaWO_4$ Scintillating Powder

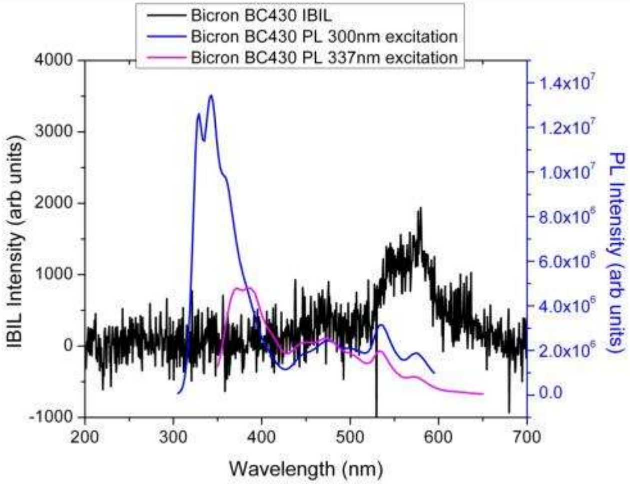


High-Z Nanocomposite

IBL Capabilities for Luminescence Studies

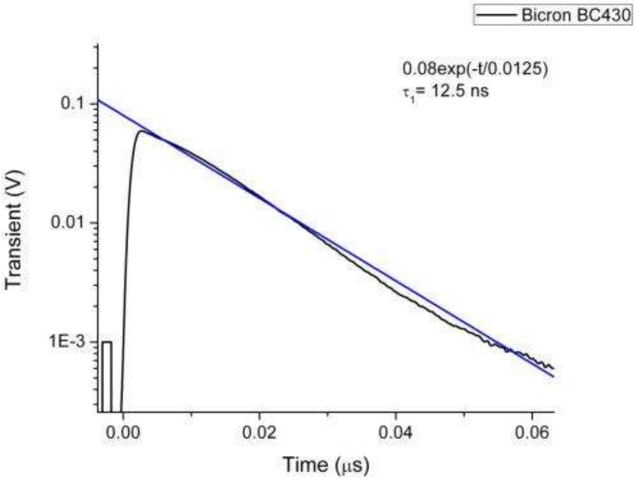
Collaborators: J. Villone & G. Vizkelethy

Spectrometry



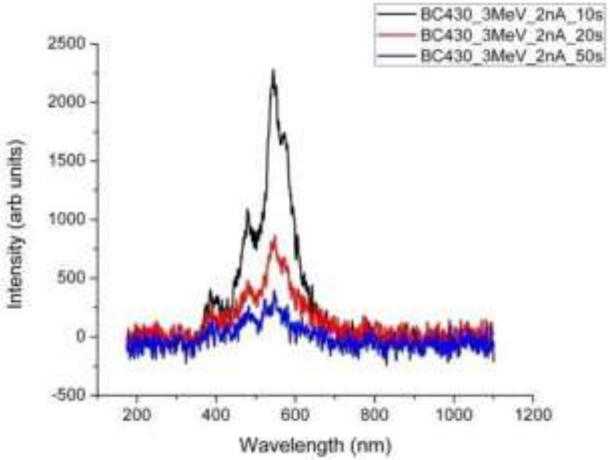
- 3 MeV H^+ beam
- Thin films of samples on PIN diodes
- Hamamatsu PMT run in photon-counting mode
- Light intensity measured as a function of time after ion strike

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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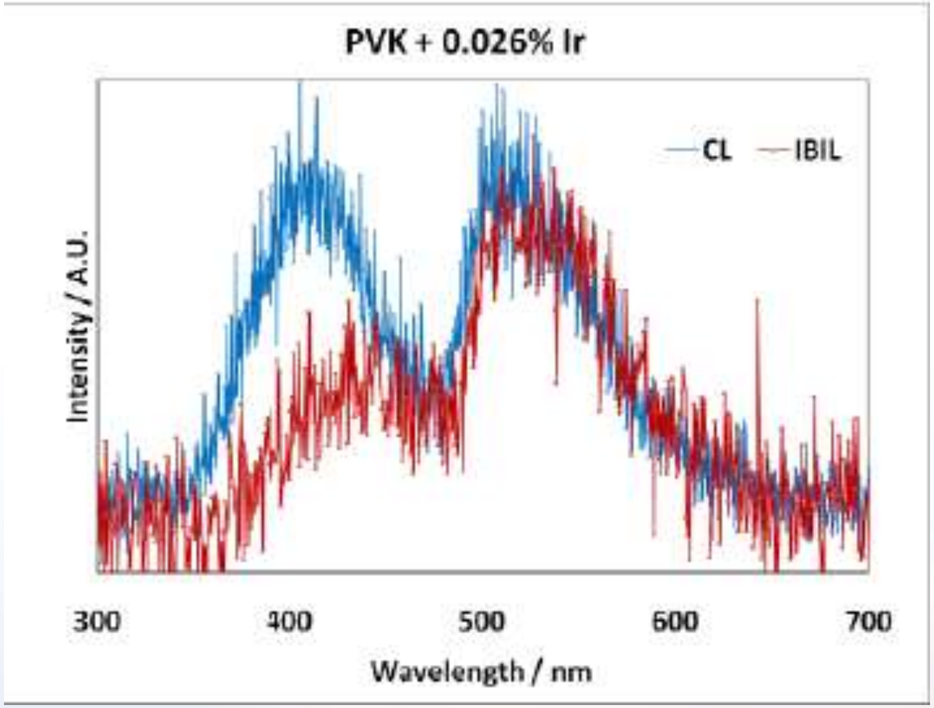
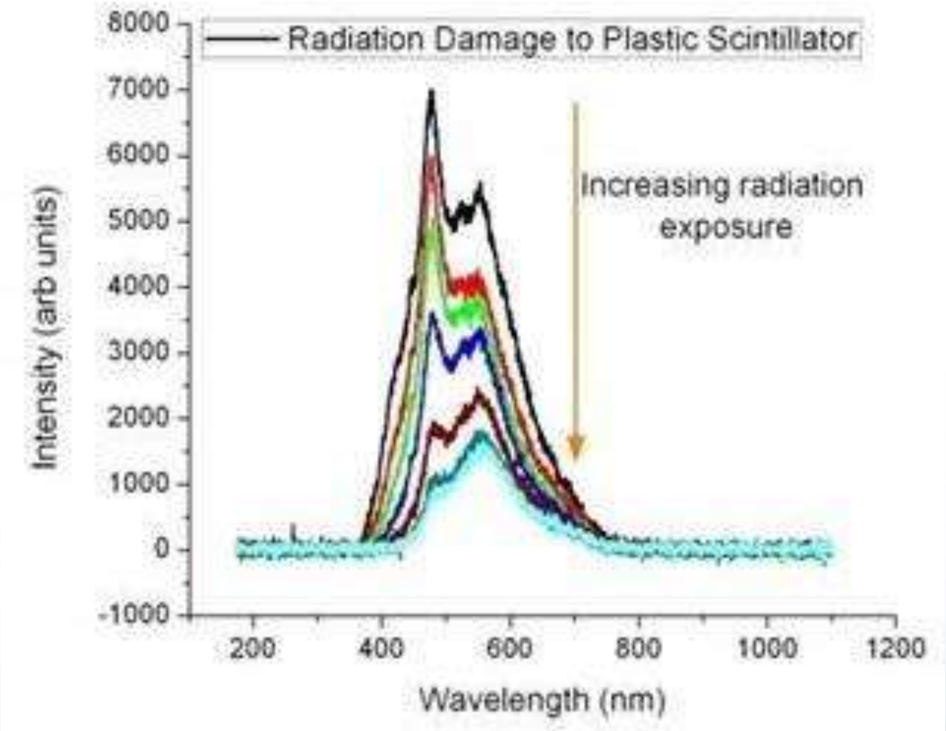
IBIL of MOFs

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

Metal-organic frameworks demonstrate spectral discrimination with IBIL/CL





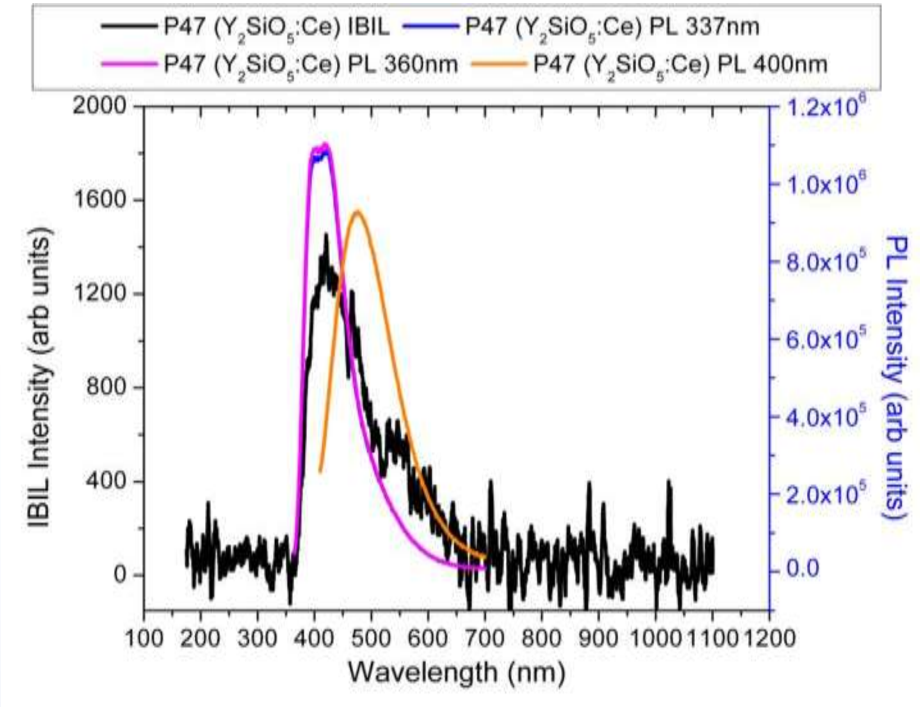
- **Spectral discrimination**
- **CL simulates response to gamma rays**
- **IBIL simulates response to neutrons**

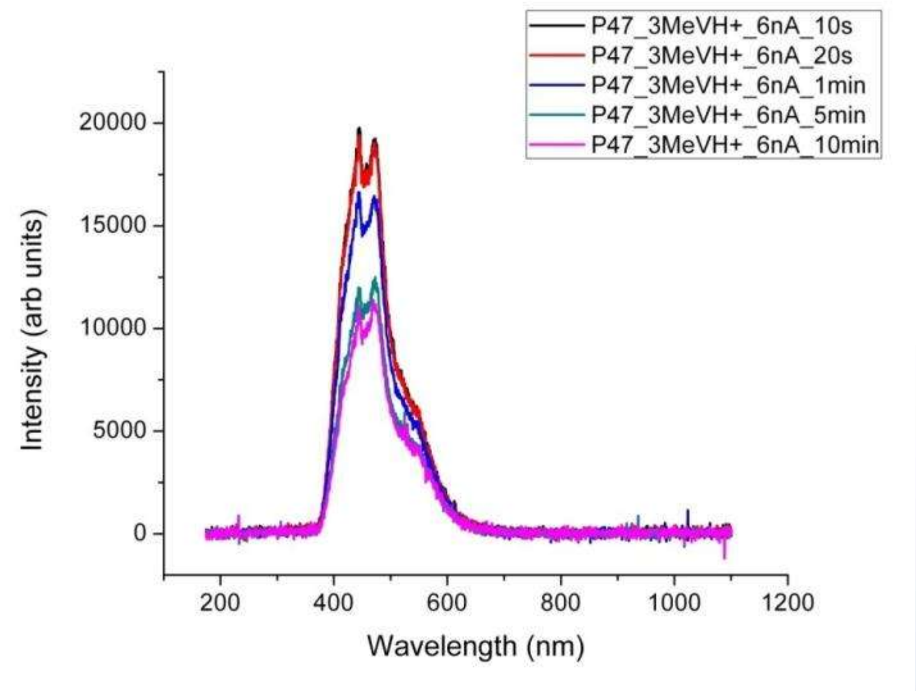
- **PL and IBIL of MOF demonstrating spectral discrimination**
- **IBIL decay of MOFs with irradiation – changes observed in relative peak height**

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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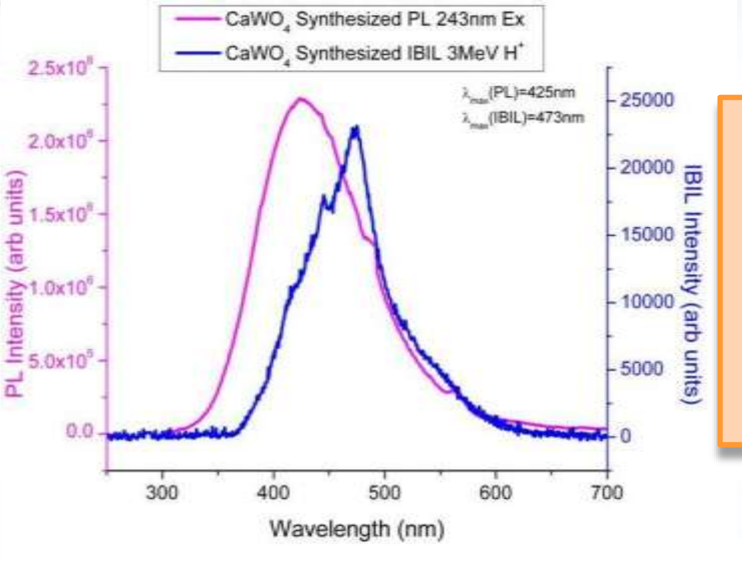
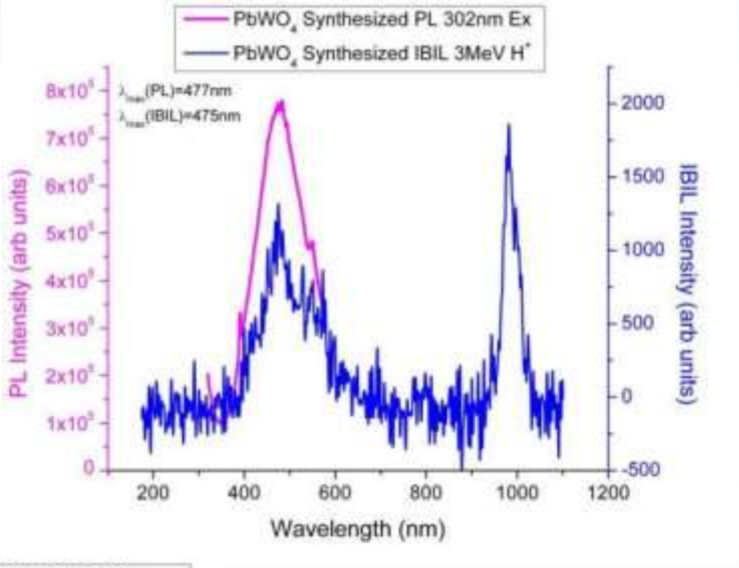
IBIL of Nanoscintillators

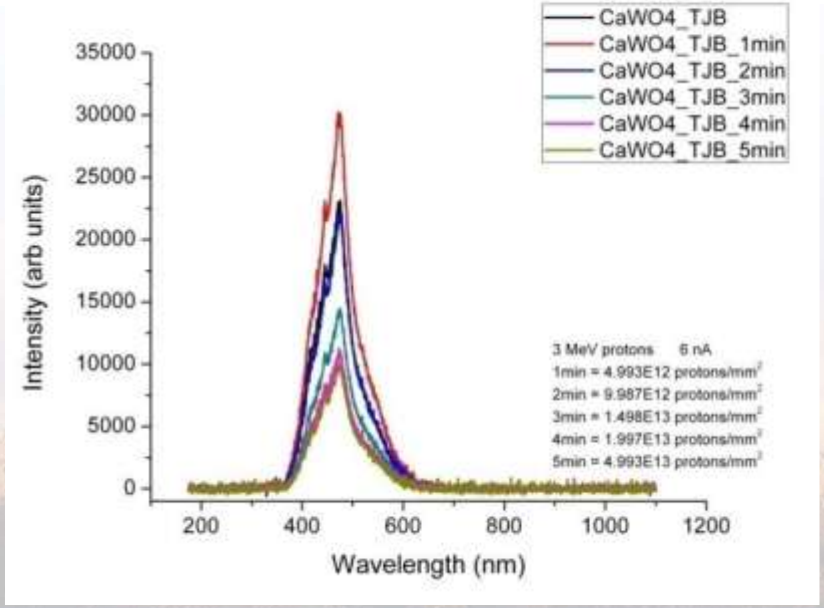
Collaborators: B.A. Hernandez-Sanchez, S.M. Hoppe, T.J. Boyle, J. Villone & P. Yang



Luminescence with proton excitation demonstrates different properties than with UV excitation



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



3 MeV protons 6 nA
1min = 4.993E12 protons/mm^2
2min = 9.987E12 protons/mm^2
3min = 1.498E13 protons/mm^2
4min = 1.997E13 protons/mm^2
5min = 4.993E13 protons/mm^2

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

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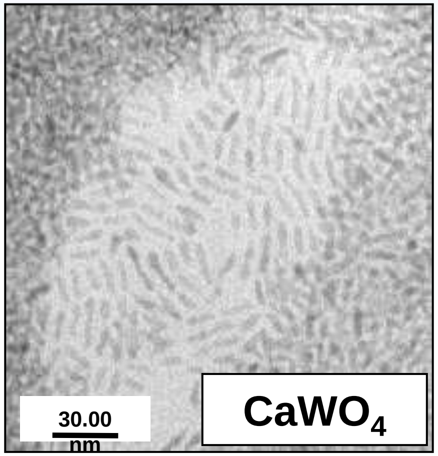
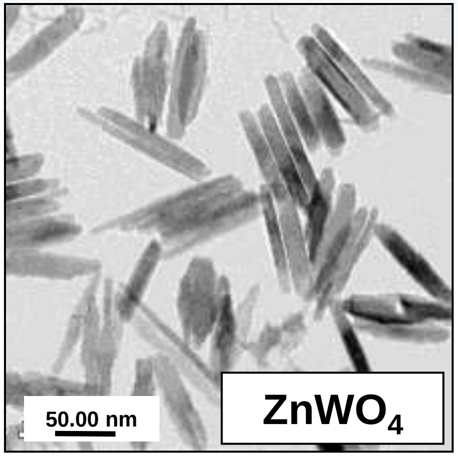
Synthesis Controlled Nanostructured Tungstates

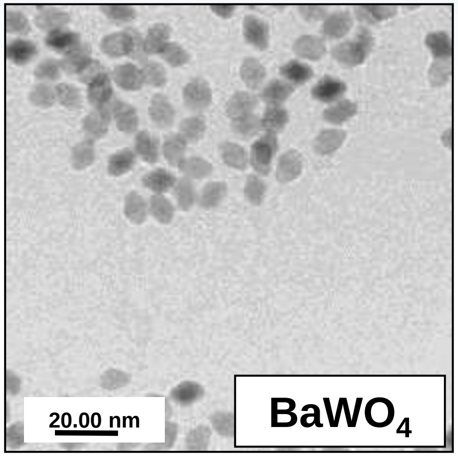
Collaborators: B.A. Hernandez-Sanchez, S.M. Hoppe, T.J. Boyle, J. Villone, & P. Yang



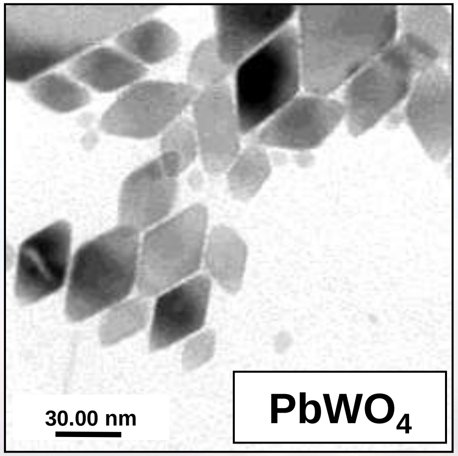
$CaWO_4$



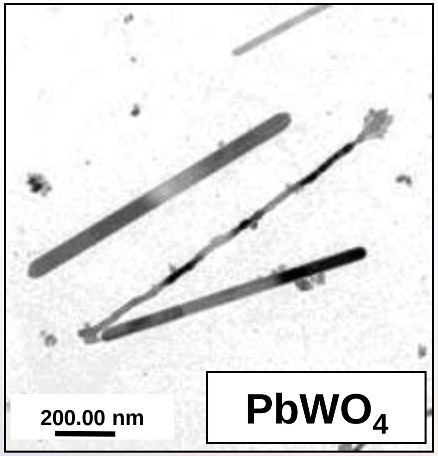
$ZnWO_4$



$BaWO_4$



$PbWO_4$



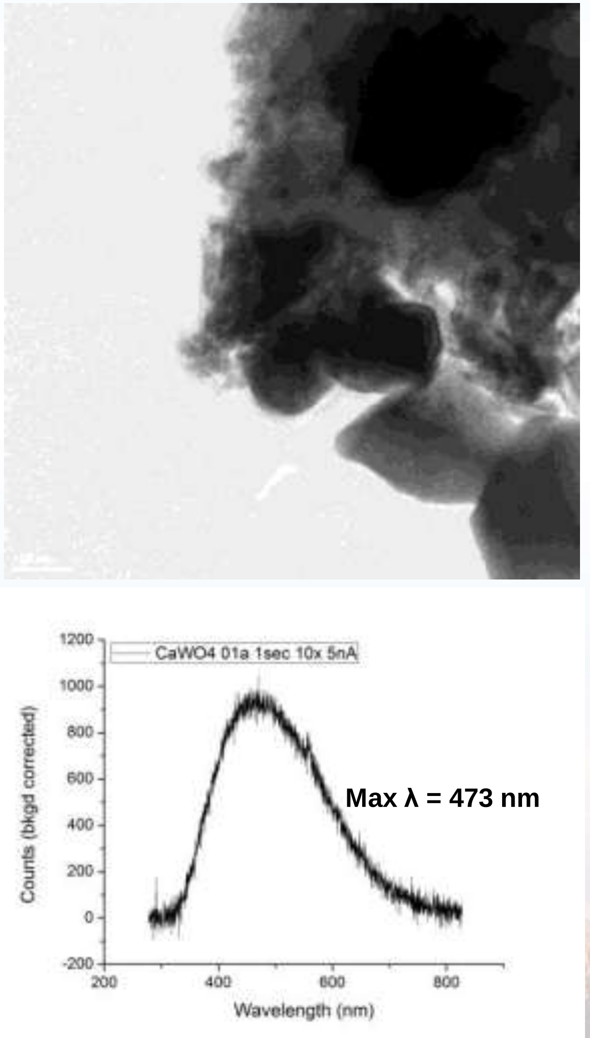
$PbWO_4$

- Entire family of MWO_4 materials synthesized:
  M = Ca, Cd, Pb, Sr, Ba, Zn, Na
- Varying composition and synthesis methods result in a range of interesting morphologies

Luminescent Properties are Dependent on Processing Route

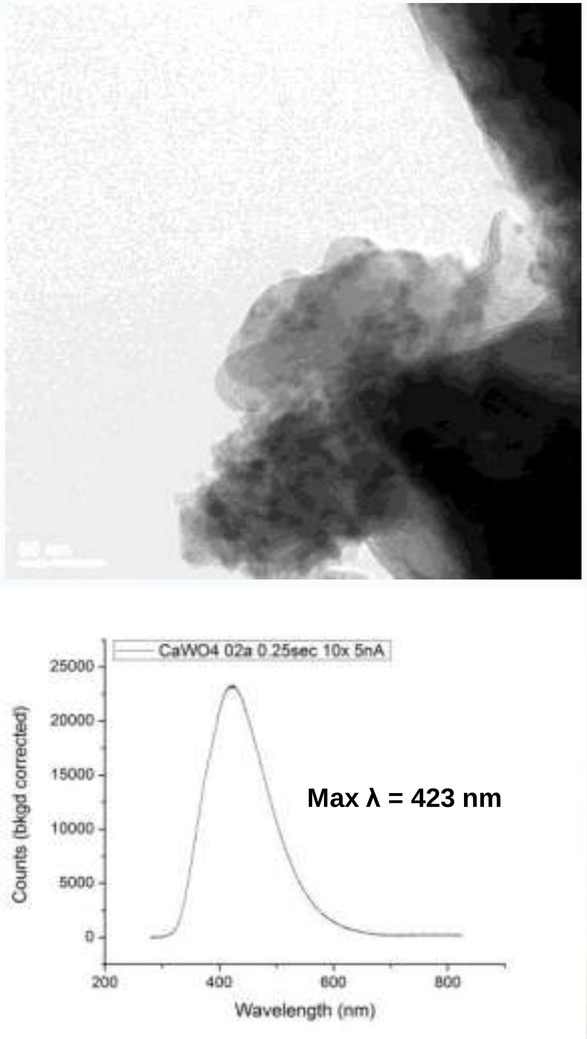
Collaborators: B.A. Hernandez-Sanchez, S.M. Hoppe, T.J. Boyle, J. Villone, & P. Yang

CaWO4 py solvo LS



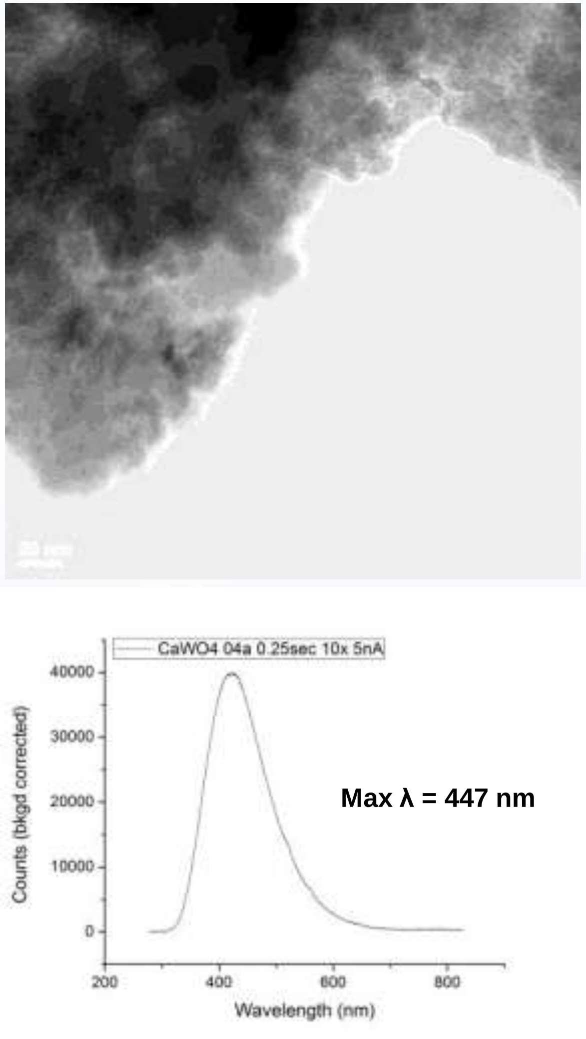
Max λ = 473 nm

CaWO4 OA/OL solvo LS



Max λ = 423 nm

CaWO4 OA/OL SPPT LS



Max λ = 447 nm

Intensity decreases and peak shifts based on processing route

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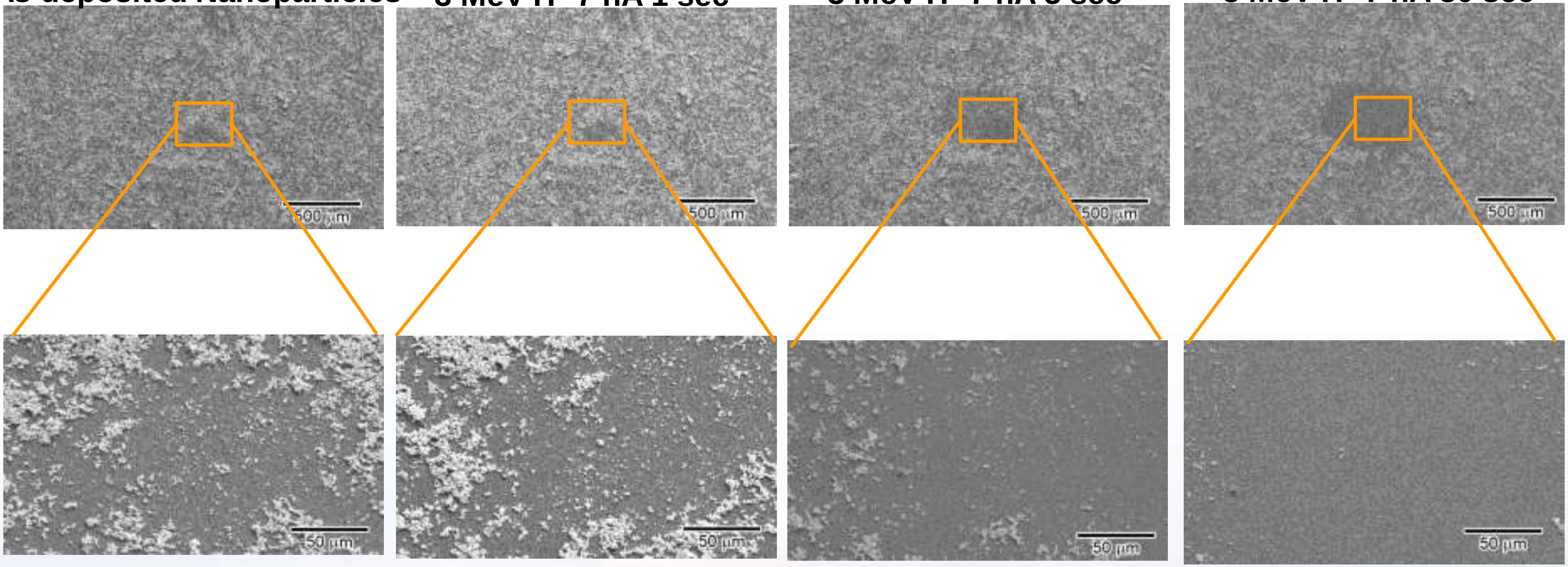
Results - Nanoscintillators

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

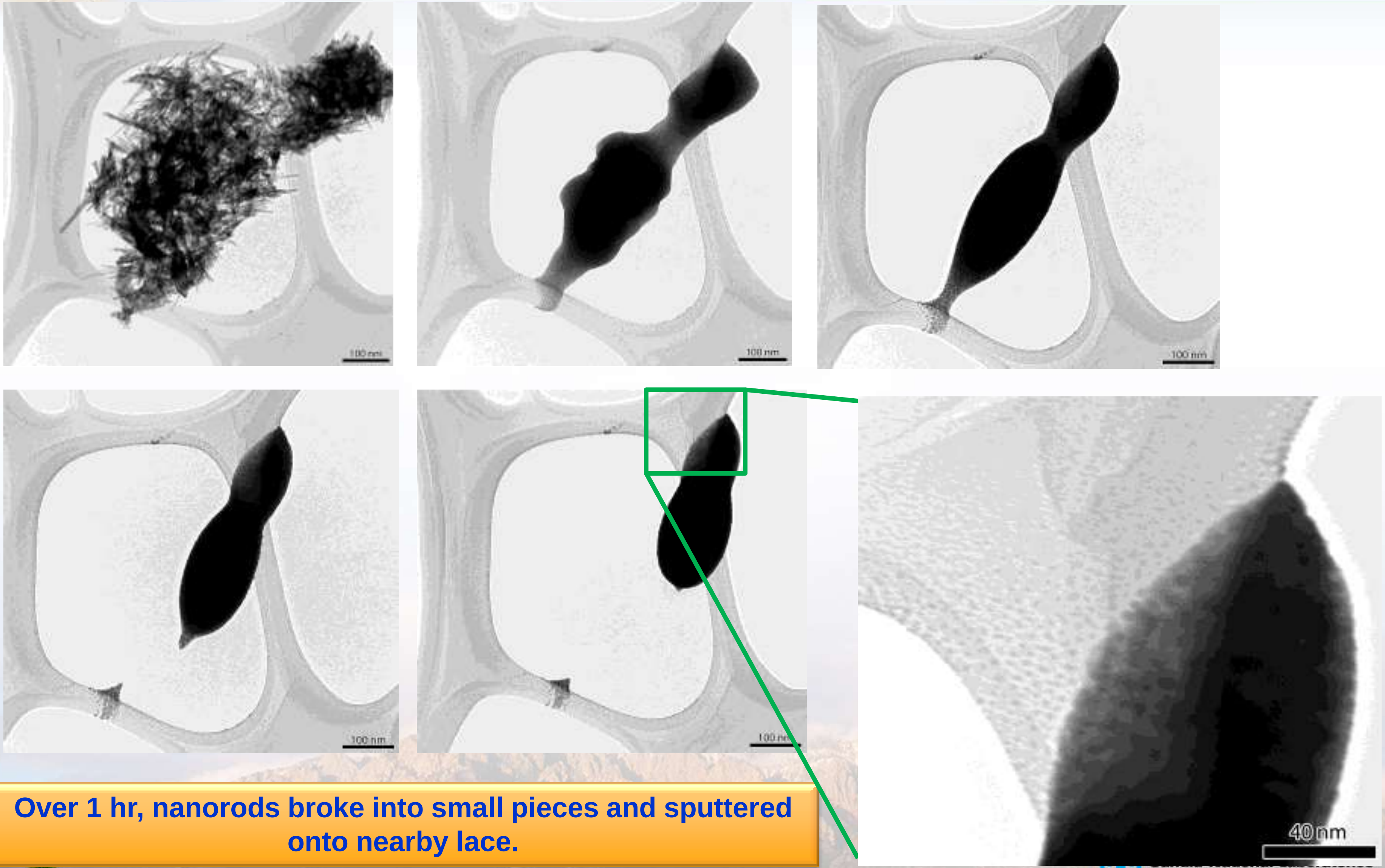
3 MeV H$^+$ 7 nA 5 sec

3 MeV H$^+$ 7 nA 30 sec

- **Want to understand if microstructure is affected by irradiation and how that influences optical properties**
- **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**

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

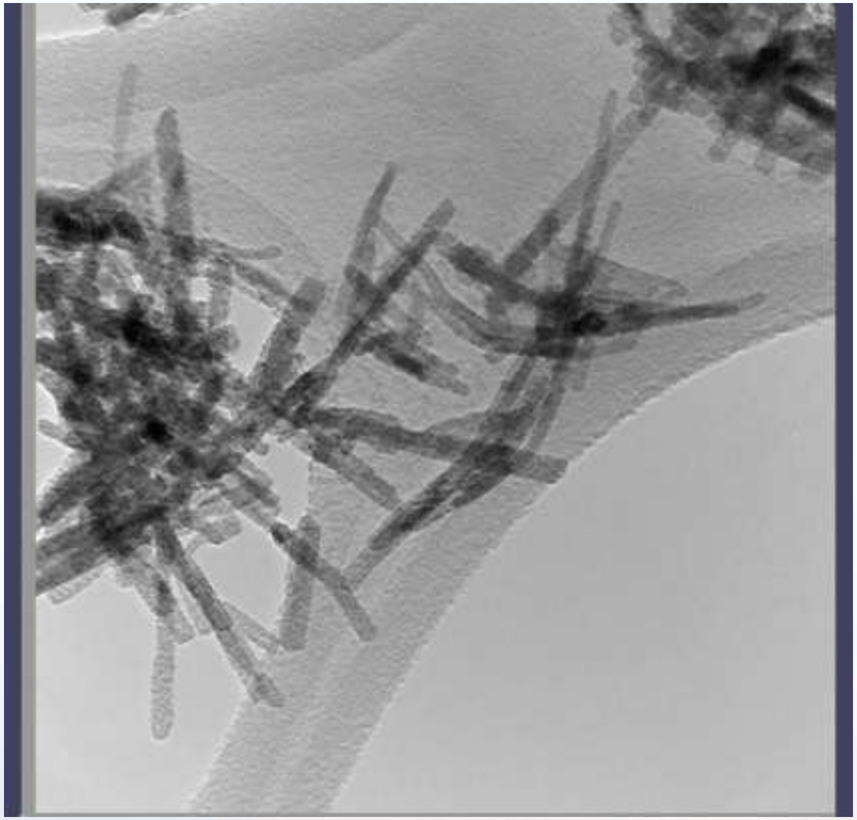
Collaborators: S.M. Hoppe & B.A. Hernandez-Sanchez

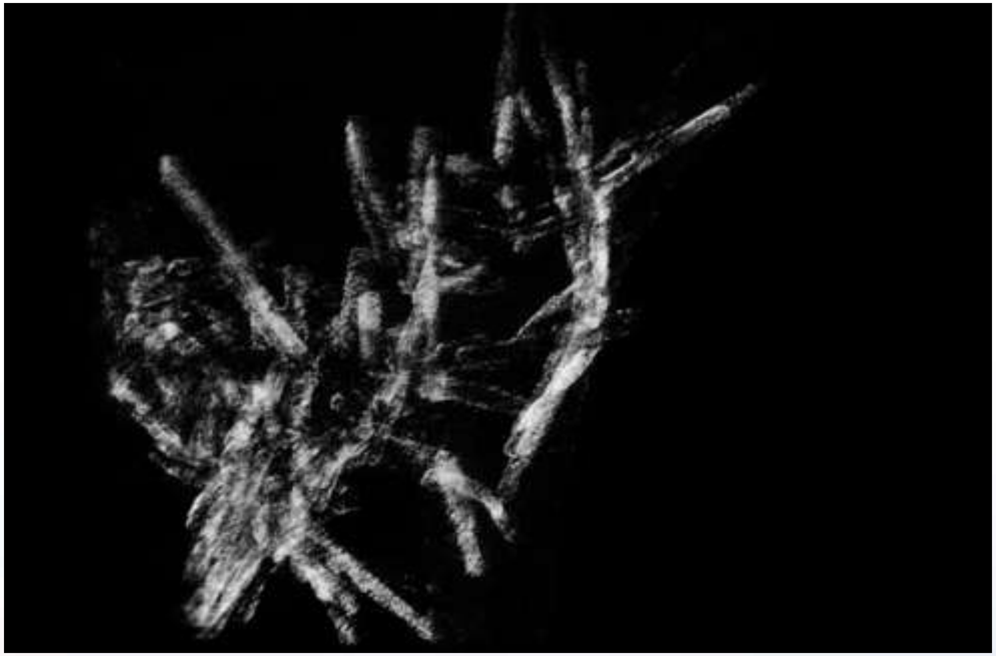


Over 1 hr, nanorods broke into small pieces and sputtered onto nearby lace.

Tomographic Reconstruction of $CdWO_4$

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



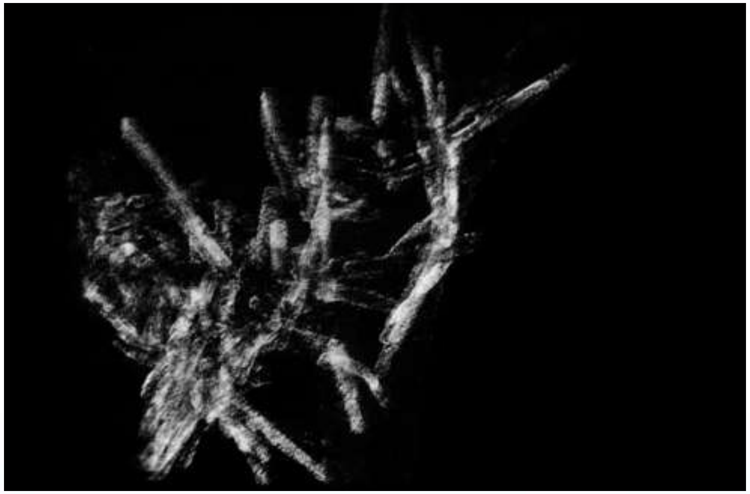


Recent advancements in TEM control and reconstruction software permit collection and production of 3D model of the “transmission” micrograph.

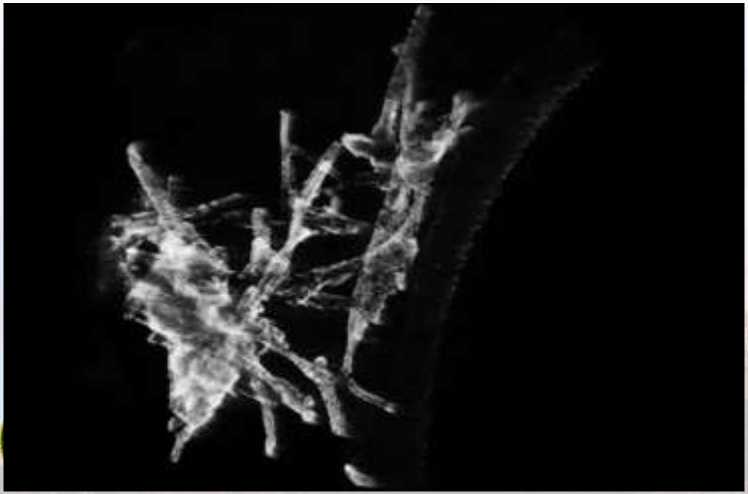
Tomography of $CdWO_4$ Irradiated with 30 nA of 3 MeV Cu^{3+}

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

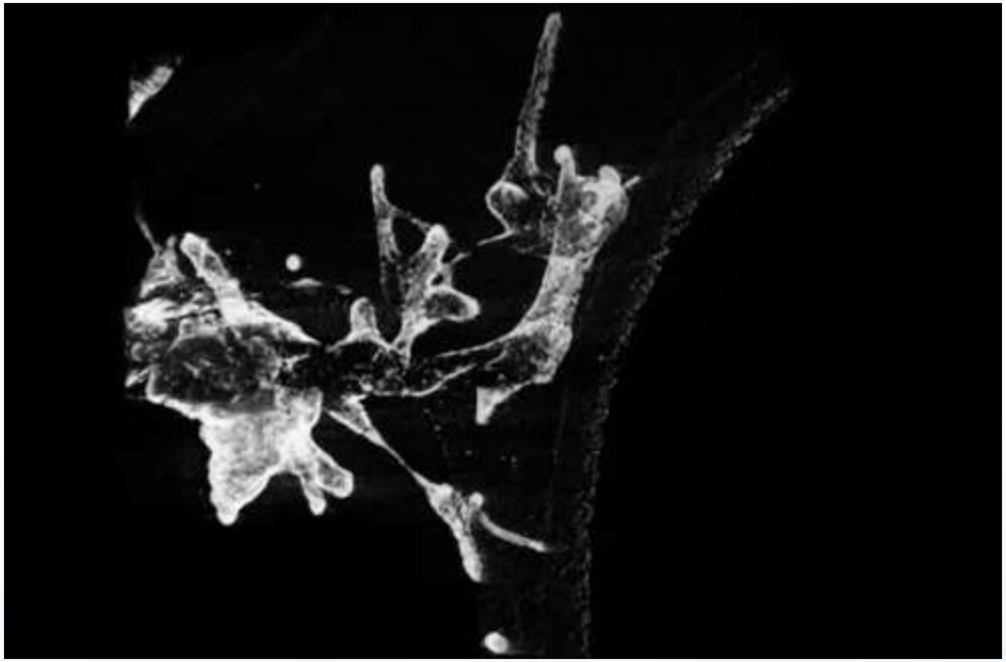
Unirradiated



5 minutes



30 minutes



Tilt series were collected after each dose of irradiation resulting in 4D tomography with 3D reconstructions showing radiation damage over time.

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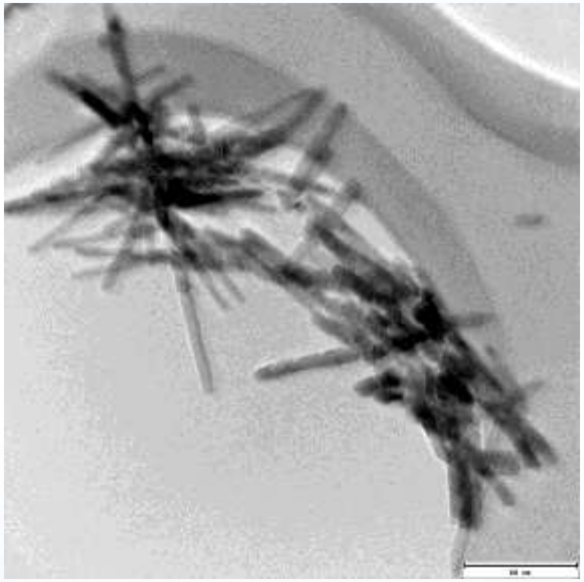
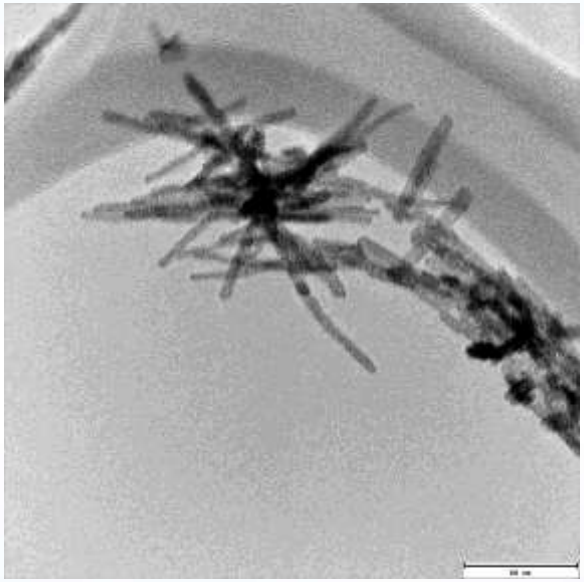
Current work: *In situ* Proton Irradiation as First Order Simulation of Neutrons

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

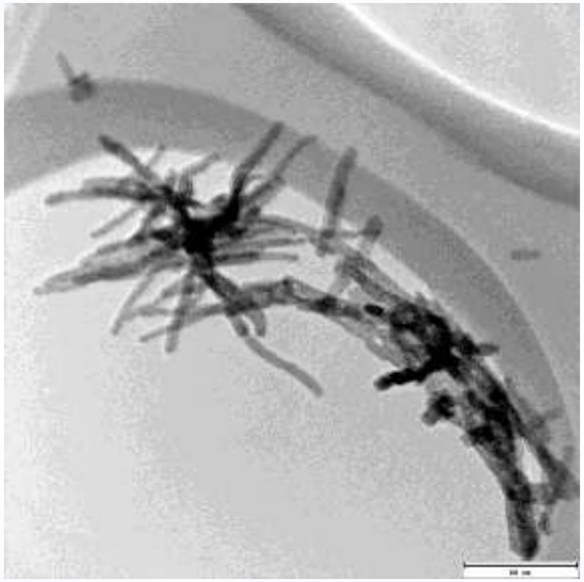
Unirradiated

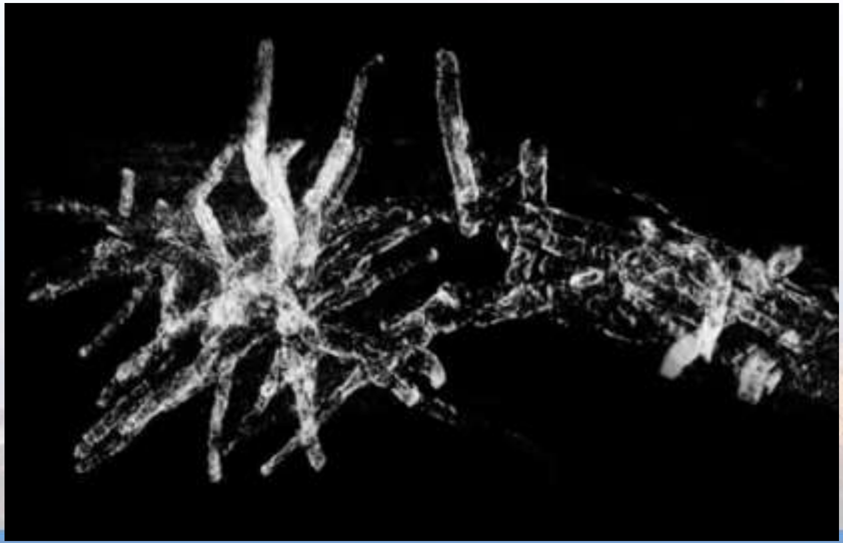


15 minutes



60 minutes





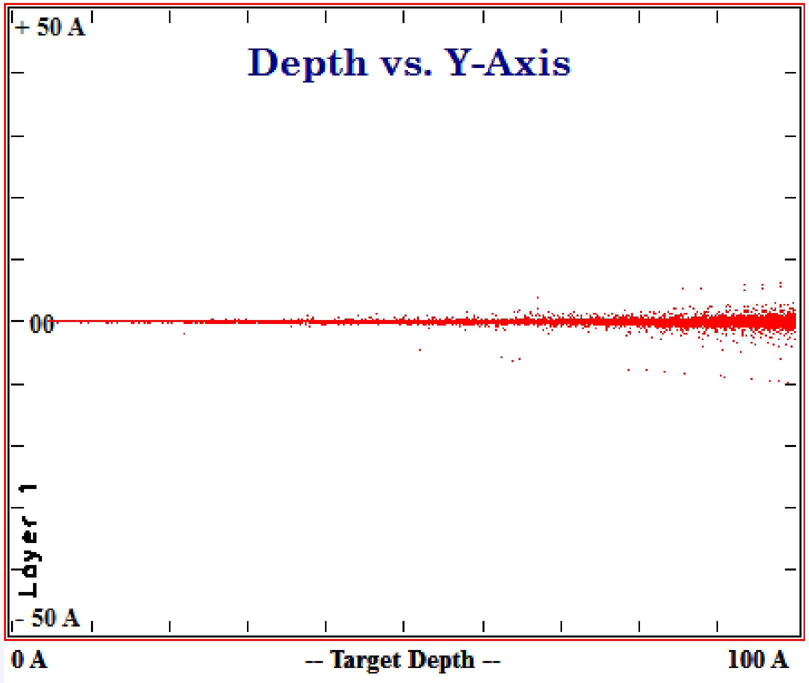
160 nA of 2.5 MeV H^+ used to simulate neutron radiation shows less change. Results suggest good radiation hardness for tungstate nanorod-composite scintillators.

Comparison of Proton and Copper Irradiation

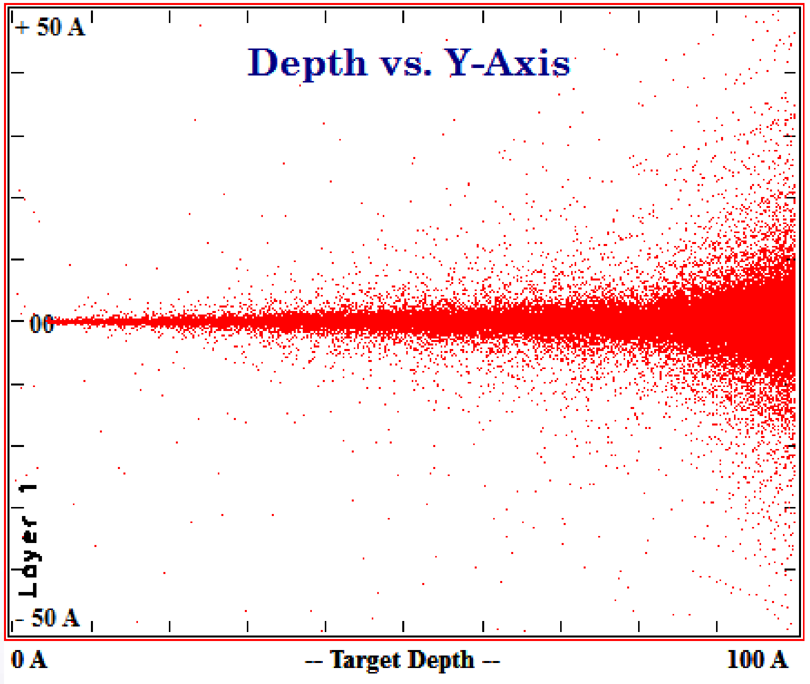
Theoretical Comparison

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

Displacement Damage from 2.5 MeV H



Displacement Damage from 3 MeV Cu



SRIM simulation of the Frenkel pairs created by the ion irradiation conditions used in 10 nm thick $CdWO_4$ noparticle.

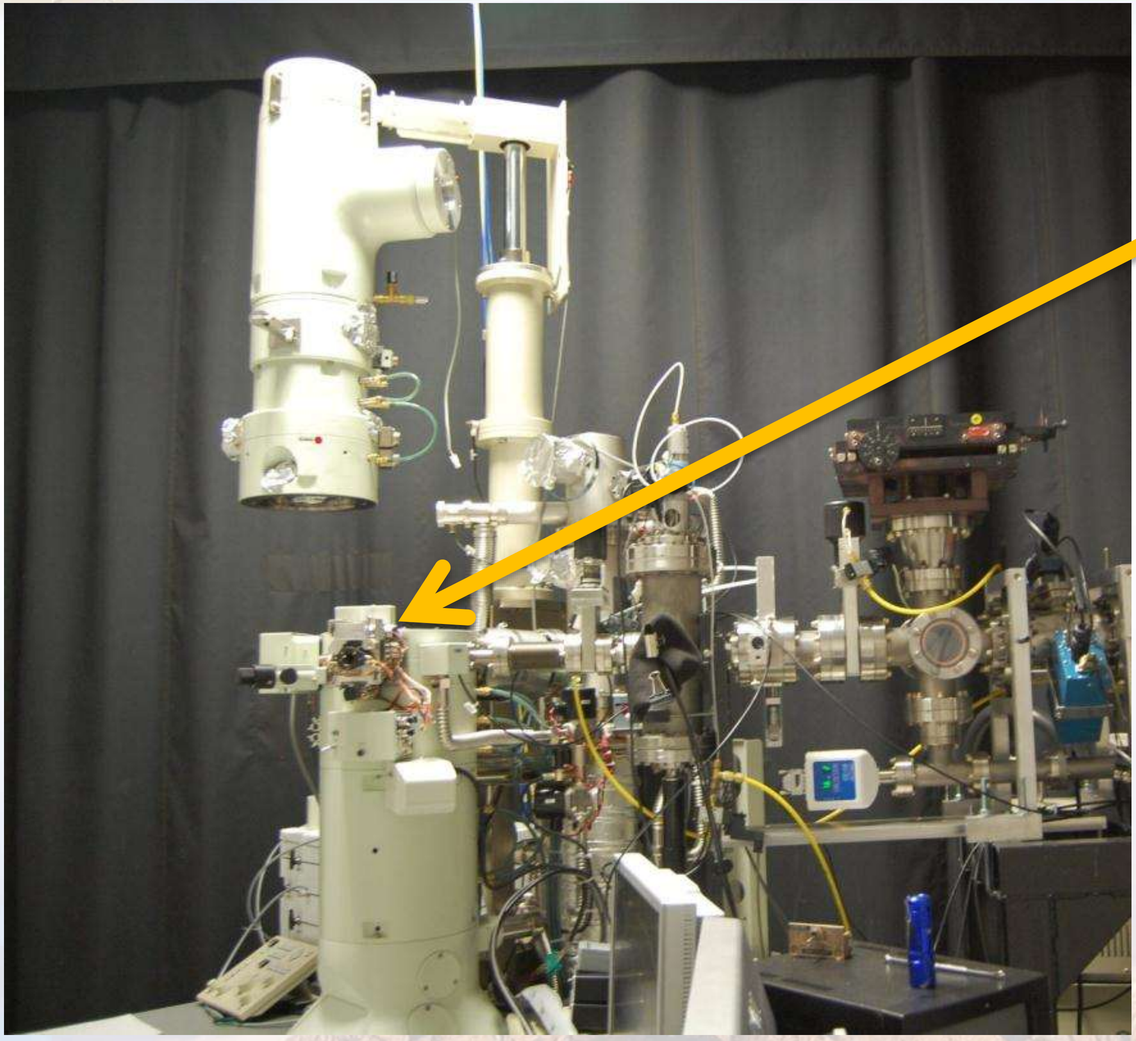
Note the large number of pairs over the same simulation set, as well as the two order magnitude dE/dx elec. and four dE/dx Nuc.

Sample	Density (g/cm3)	Species	Energy (MeV)	Current (nA)	**dE/dx Elec.**	**dE/dx Nuc**	Proj. Range (um)	Long. Straggle (um)	Lat. Struggle (um)
CdWO4	7.9	H	2.5	~100-200	**5.97E-02**	**3.80E-05**	33.62	2.2	3.59
PbWO4	8.235	H	2.5	~100-200	**5.18E-02**	**3.39E-05**	37.22	2.8	4.63
CdWO4	7.9	Cu	3	~10-30	**2.19E+00**	**5.31E-01**	1.25	0.4581	0.4096
PbWO4	8.235	Cu	3	~10-30	**2.67E+00**	**6.11E-01**	1.16	0.3632	0.3328

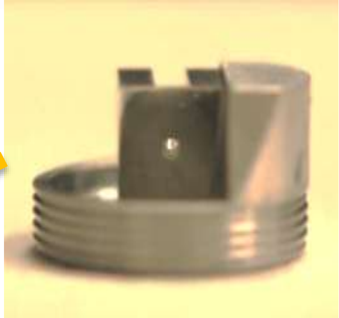
The Stopping and Range of Ions in Matter (SRIM) is a Monte Carlo-based simulation of the ion beam interaction with an amorphous material.

Current work: *In situ* TEM Luminescence

Collaborators: D. Masiel



Optical Mirror in TEM



First IBIL in TEM

Optical Pathway in an I^{3}TEM

- **Angled mirror with bore hole for the electron path was installed.**
- **Mirror is located on top of the objective polepiece “heart of the TEM”**
- **Port is being constructed with thick leaded glass to permit light through, but not x-rays created by ion or electron beams.**
- **Should permit *in situ* IBIL and CL.**

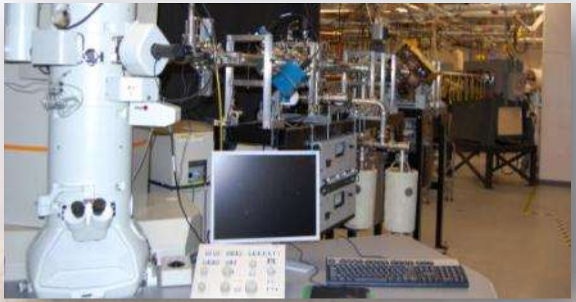
An optical port is currently being added to the I^{3}TEM, which, if successful, will permit *in situ* TEM luminescence studies (CL and IBIL)

Summary and Future Work

- **The new ion beam lab at Sandia is operational**
  - 5 accelerators ranging from 1 keV to 100s of MeV
- **Developed a combinatorial approach to rapidly test the radiation damage for nuclear energy applications**
  - Ion Implanted to high dpa using heavy ion irradiation
  - A variety of small-scale mechanical property testing
    - Berkovich and spherical indentations
    - Micropillar compressions
- ***In situ* ion irradiation and gas exposure provides insight into the microstrucutral evolution under various extreme conditions**
- **Can investigate the structure of samples during IBIL**

Future Work

- **Attempt concurrent *in situ* ion irradiation**
- **Determine feasibility of *in situ* fluidic ion irradiation TEM**
- **Obtain *in situ* TEM IBIL of electron transparent samples**



Sandia is developing a suite of *in situ* TEM tools for studies applicable to nuclear materials