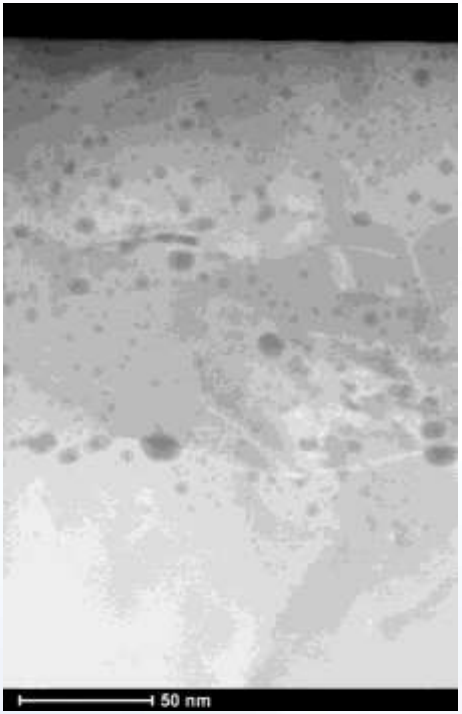
Tailoring Nanostructures via Ion Beam Modificaiton

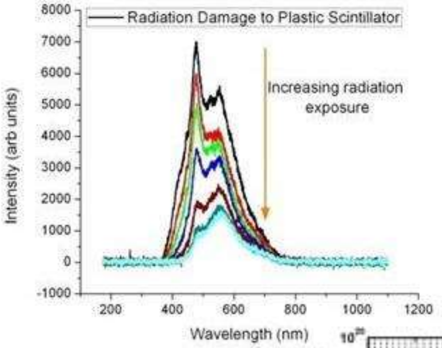
SAND2013-9595C

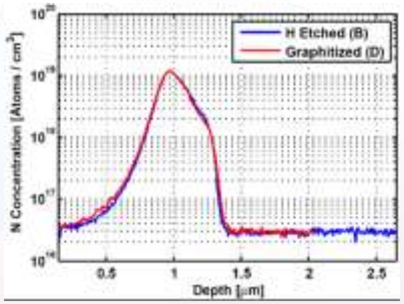
K. Hattar

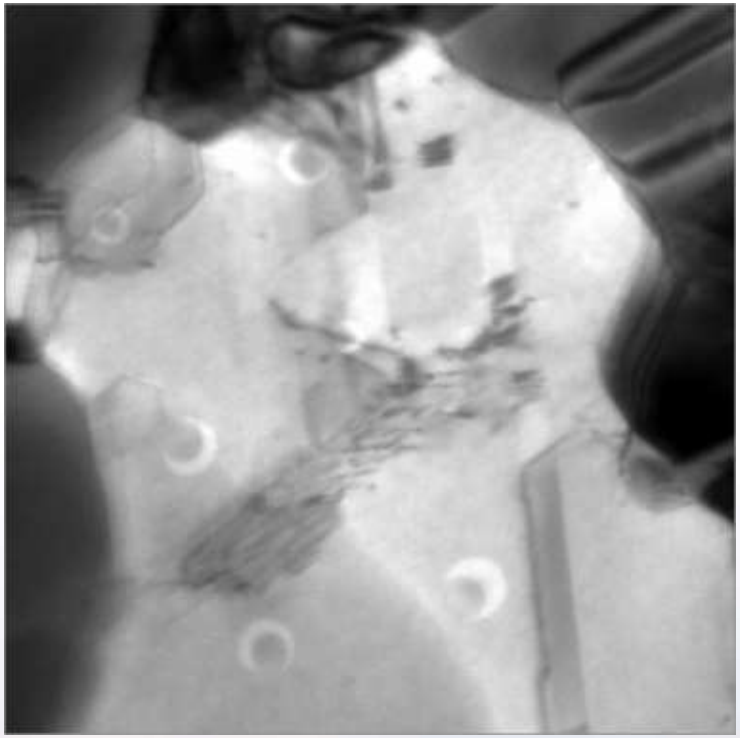
Sandia National Laboratories

November 6, 2013









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.**

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 undertake this task. In my opinion you have here an opportunity to render an exceptional service in the national interest.

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

Very sincerely yours,

Harry Truman

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

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

New Facility

laboratory space

1850 m^2

office space

650 m^2

Old Facility:

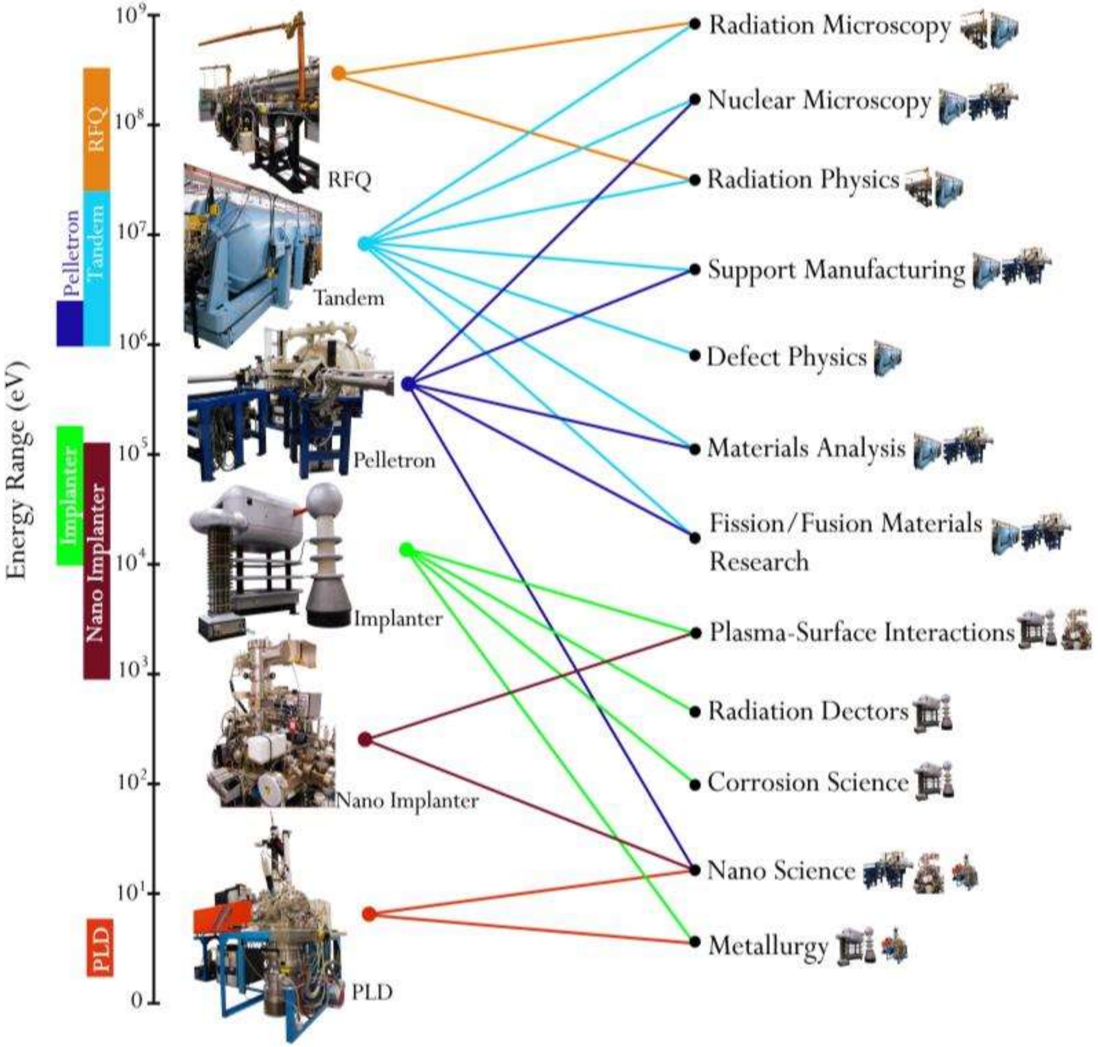
1300 m^2 total

Building: $20M

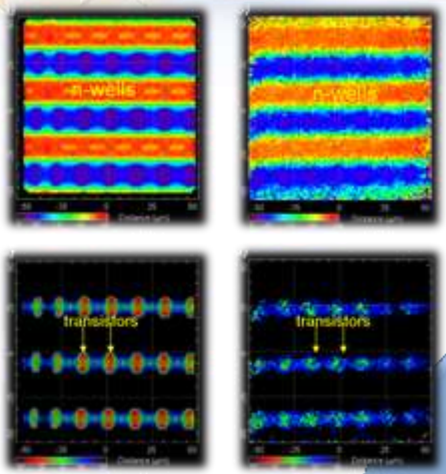
Equipment: $11M

Total: $40M

Ion Beam Laboratory Capabilities



Sandia’s Ion Beam Laboratory



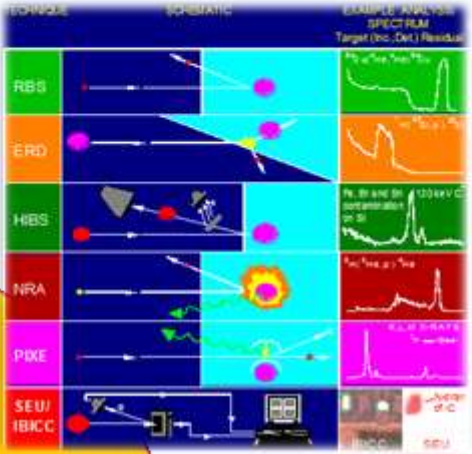
Ion Beam Analysis (IBA)

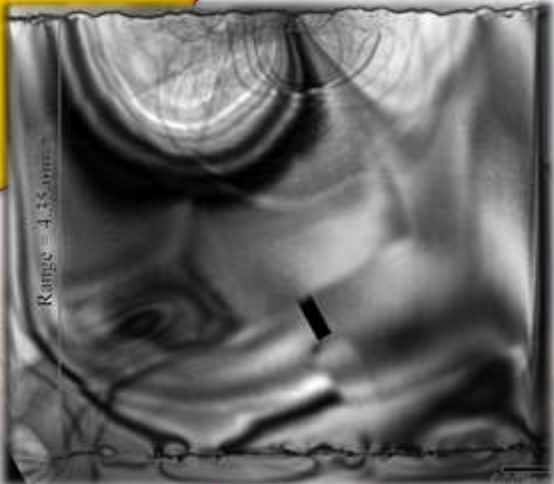
Radiation Effects Microscopy (REM)

Ion Beam Modification (IBM)

In situ Ion Irradiation Transmission Electron Microscopy

(I^3TEM)

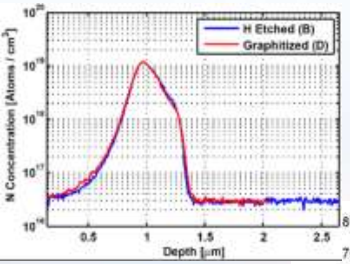


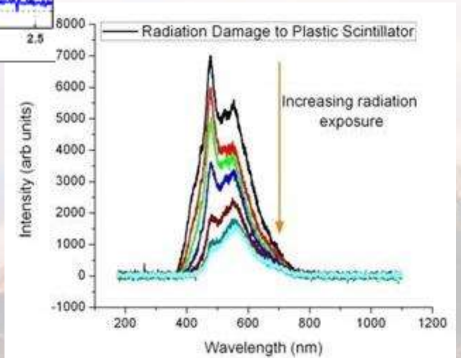


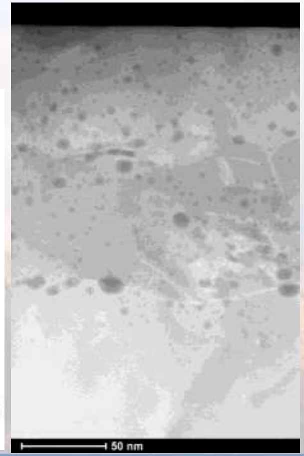
Outline

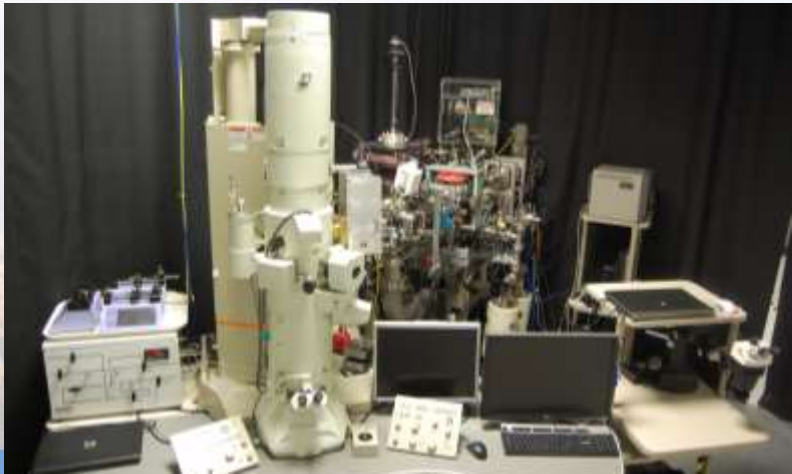
1. **Materials Engineering**
   - Control of Local Chemistry
     - Development of back gates for graphene devices
   - Investigate Radiation Tolerance
     - Various radiation detectors from MOFs to complex oxides
   - Control of Local Structure
     - Improving the wear properties of electrical contacts

2. **Materials Science**
   - Broad Capability of the I³TEM
   - Revisit of the Radiation Detectors
     - In-situ ion irradiation of high-z nanoscintillators
   - Revisit of the Structural Control
     - In-situ study of Au electrode evolution under ion beam modification
     - Initial work on indentation and cyclic compression
   - Fun future directions for real-time nanoscale observation in extreme environments









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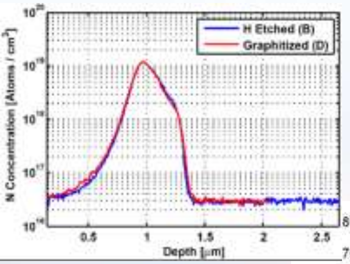
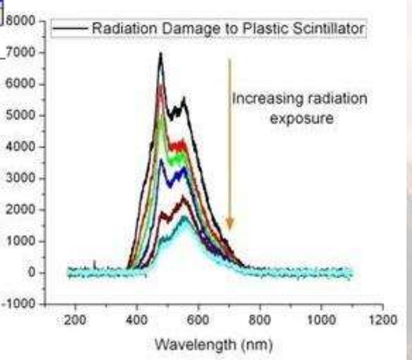
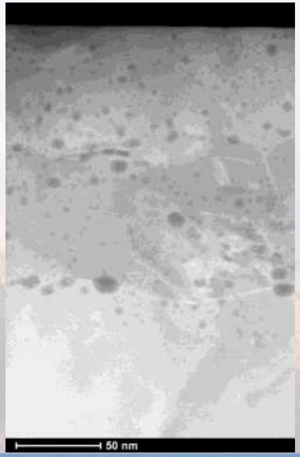
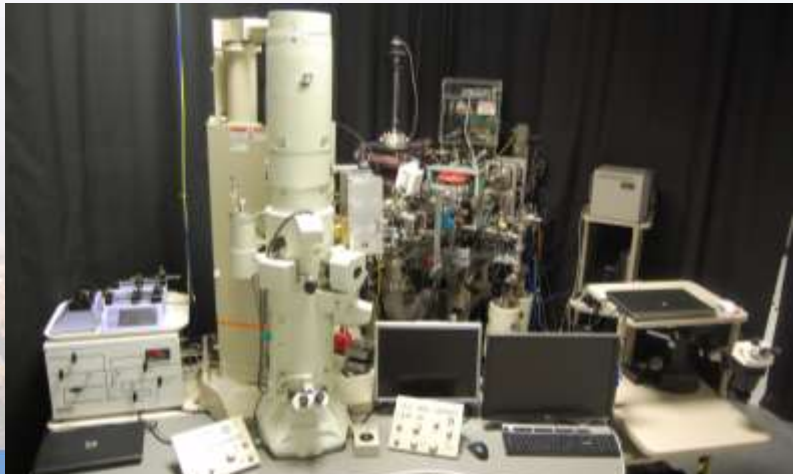
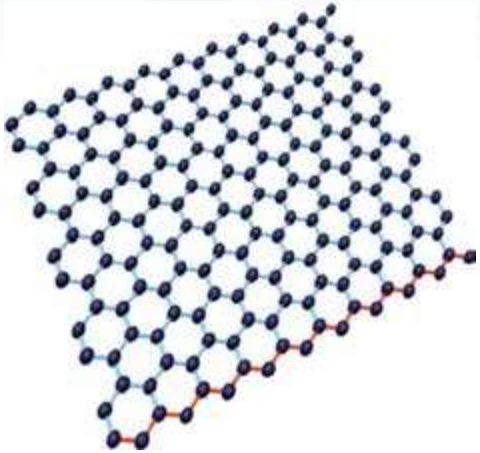




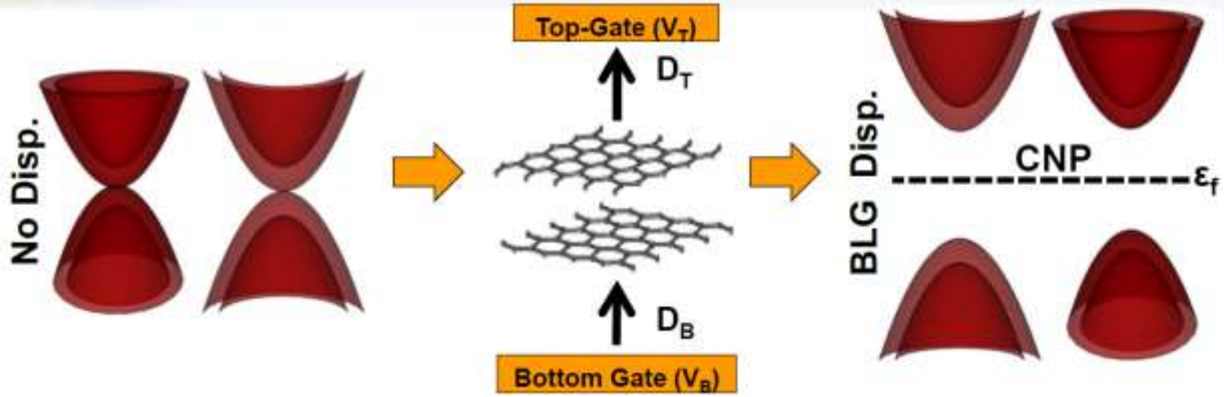


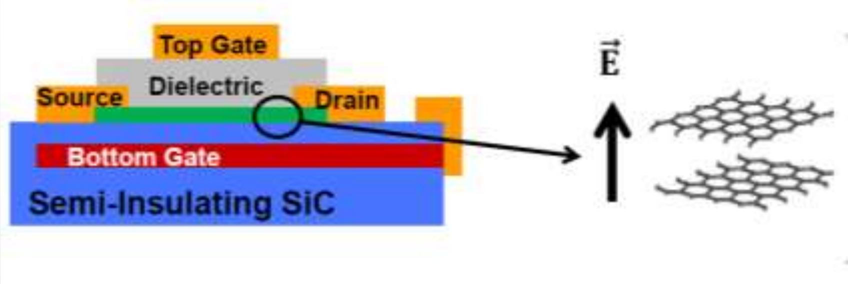


Development of a Dual-Gated Bilayer Graphene Device



2D hexagonal net of sp2 bonded carbon atoms





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

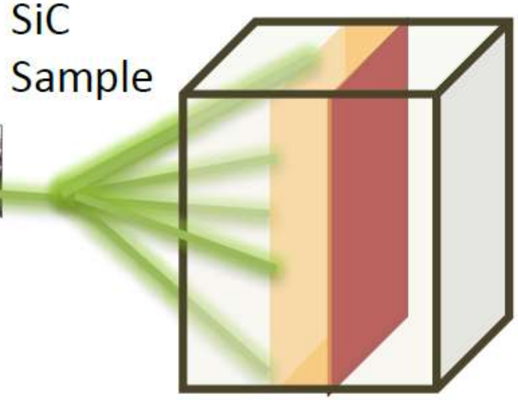
- Induce a bandgap
  - $D_{ave} \neq 0$
- Control Fermi Level (ε_f) to charge neutrality point (CNP)
  - $\delta D = 0$

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

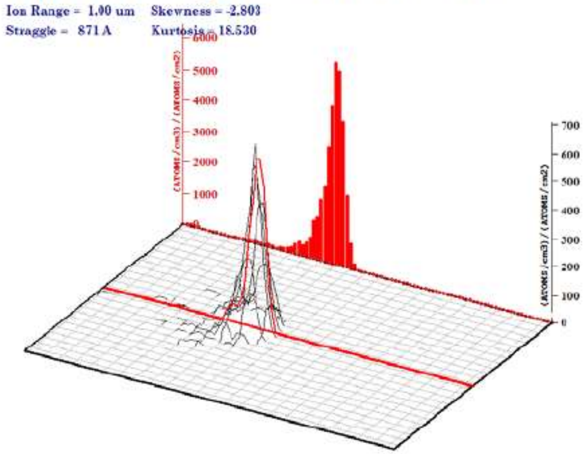
3 MeV Nitrogen Implants to Form a Back-Gate in Semi-Insulating SiC

3 MV NEC
Pelletron

SiC
Sample



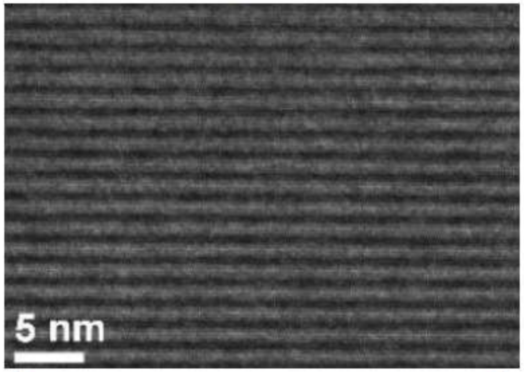
TRIM Simulation of Blanket Implant Depth Distribution



Ion Implantation Results

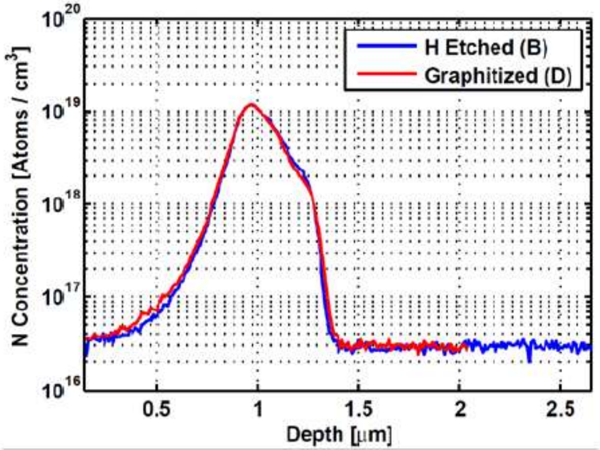
- Implanted N ions (target depth of 1 μm)
- Capacitance measurements indicated an isolated conductive layer at a depth of ~ 700 nm in the SiC

TEM Validation of Microstructure



Tailored ion implantation provides both back gate and side contact structure

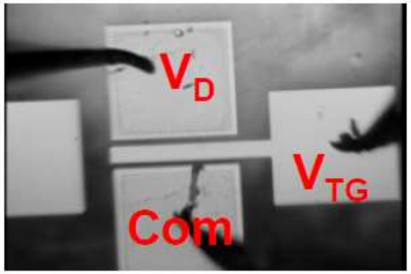
Validation of Implant Depth (SIMS)

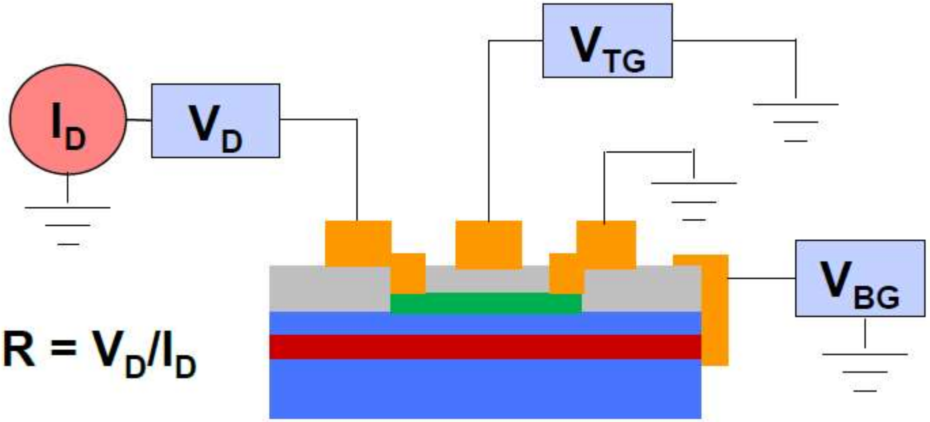


Waldmann *et al.*, Nature Materials, **10**, 357 (2011)

Bandgap Control via Dual Gating

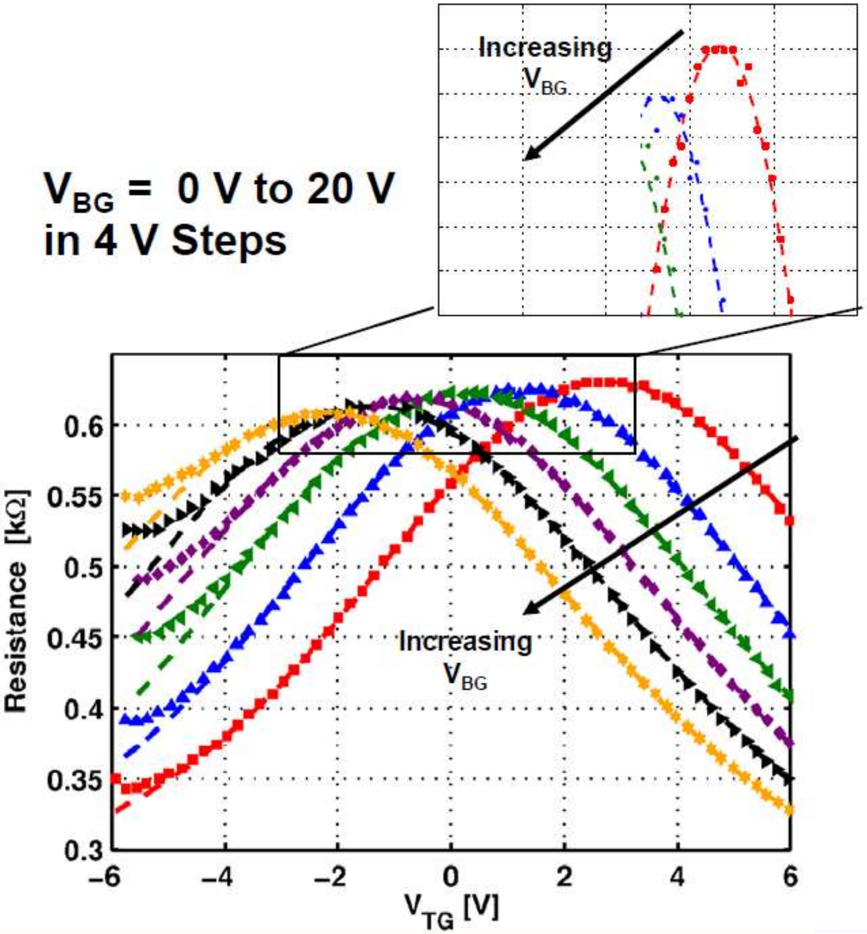
(1st Demonstration of scalable dual-gate BLG FETs)



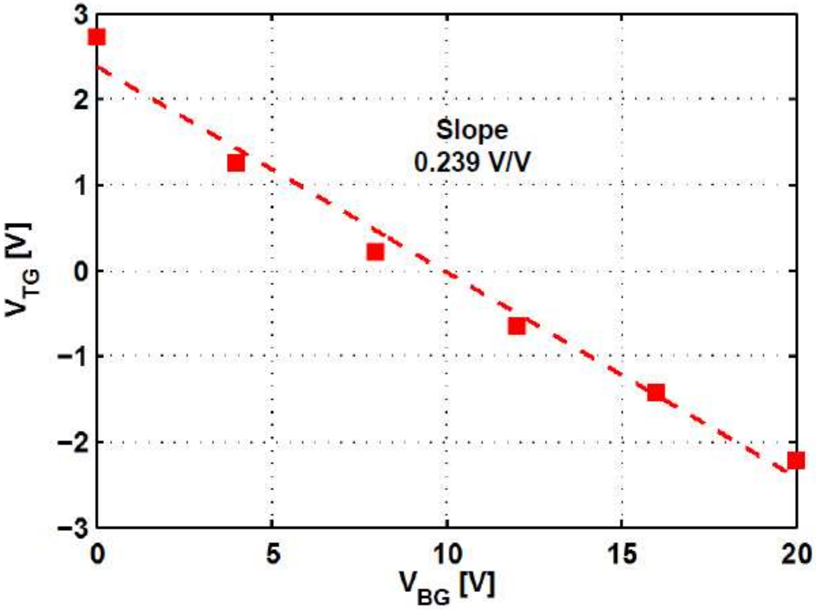


$R = V_D/I_D$

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



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



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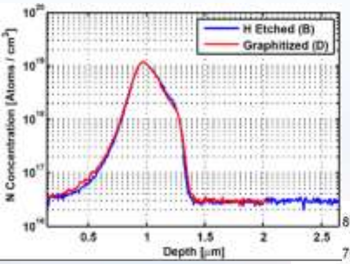
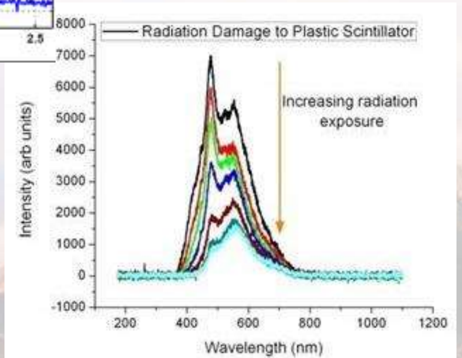
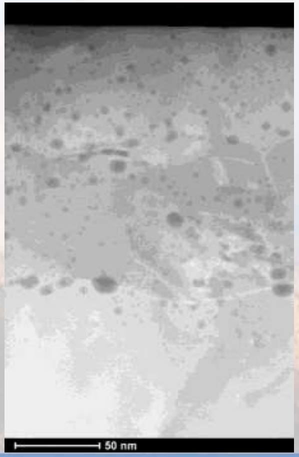
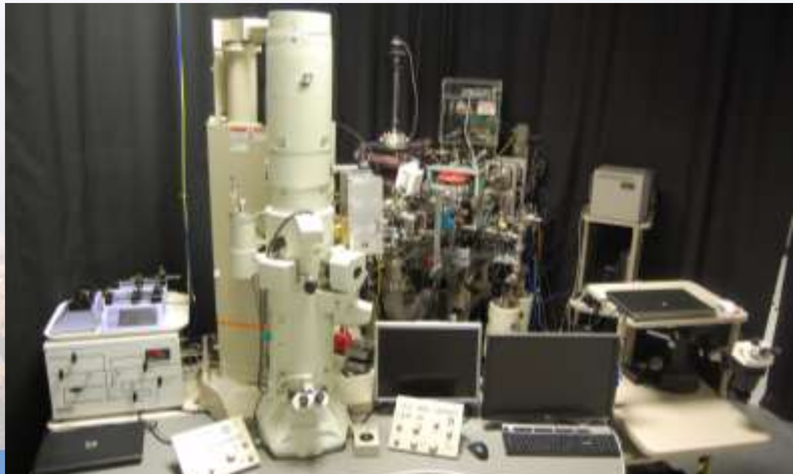


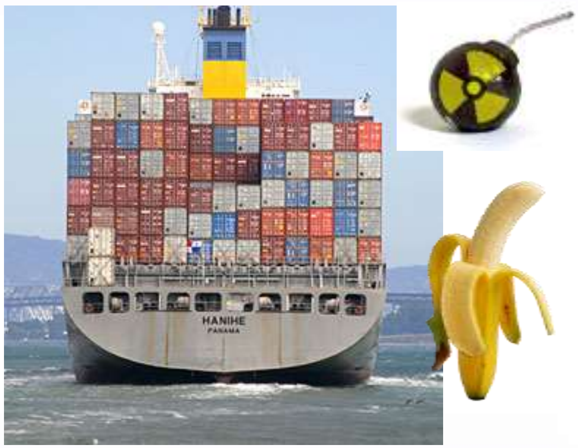




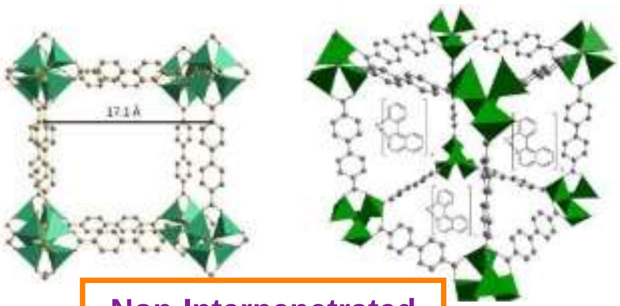


Interest in Scintillators

Crucial to understand radiation-solid interactions on multiple length scales



US Ports: 2 Billion Metric Tons



Non-Interpenetrated IRMOF-10

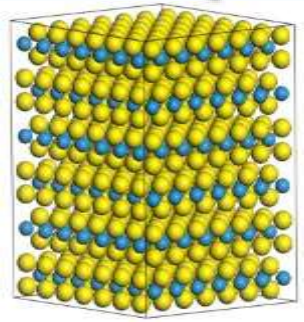
Single Crystal $CdWO_4$

Plastic Scintillators

Nano

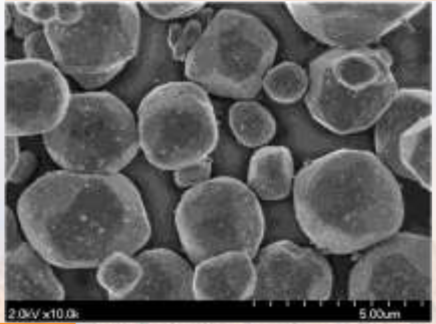
Micro

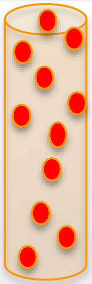
Meso



Tungsten (IV) Sulfide WS_2

Commercial $CaWO_4$ Scintillating Powder





High-Z Nanocomposite

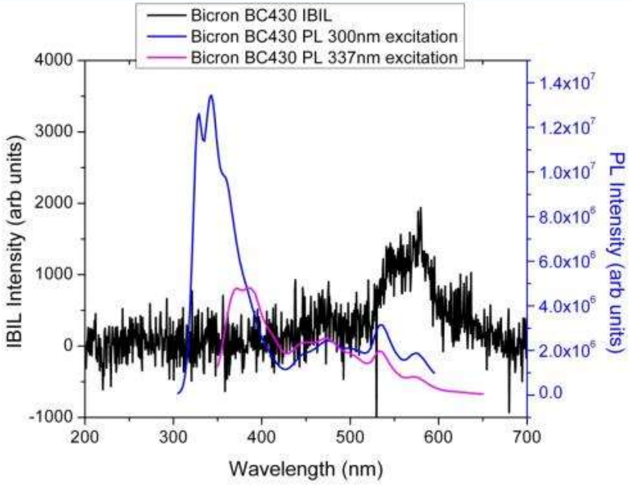
Scintillators with low energy resolution & detection efficiency cannot distinguish radiation type or quantify radiation

High-Z ME_x Nanoscintillator

IBL Capabilities for Luminescence Studies

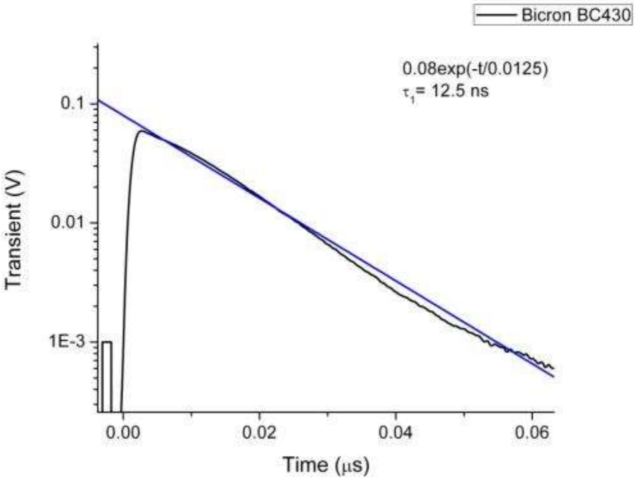
Collaborators: J. Villone & G. Vizkelethy

Spectrometry



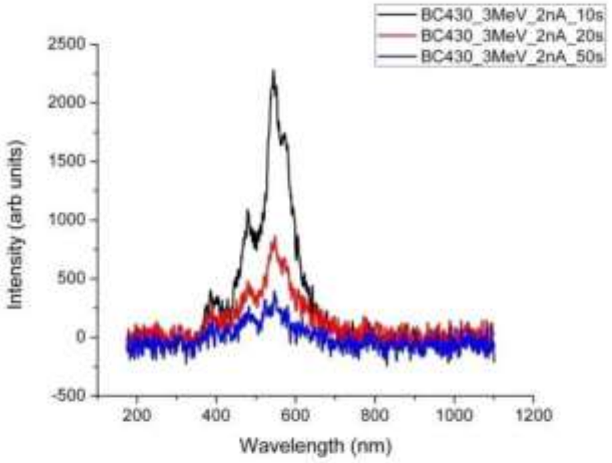
- 3 MeV H^+ beam
- Thin films of samples on PIN diodes
- Hamamatsu PMT run in photon-counting mode
- Light intensity measured as a function of time after ion strike

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

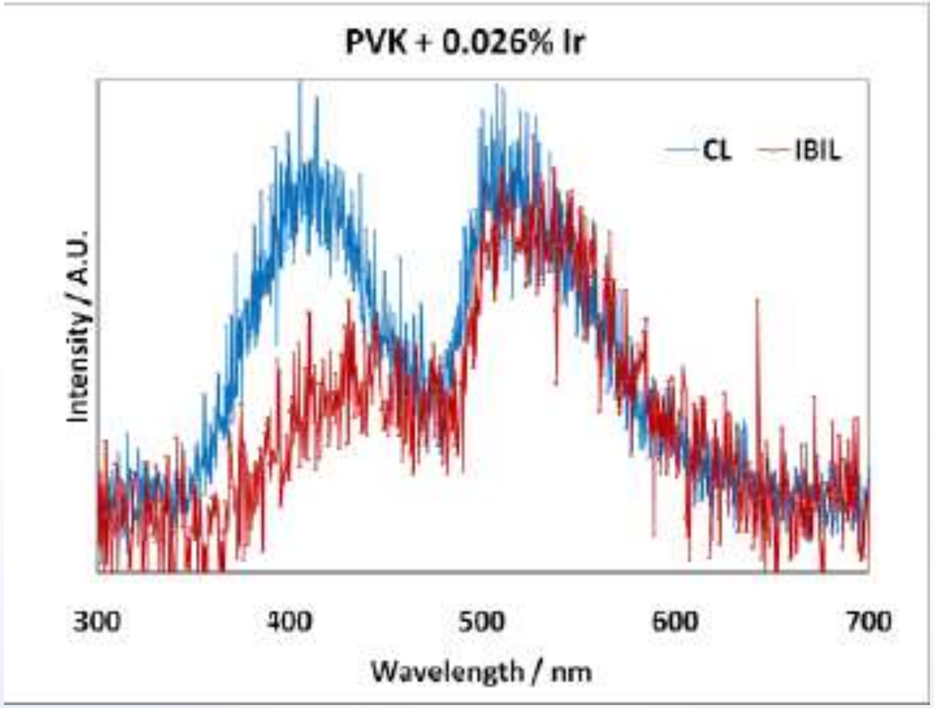


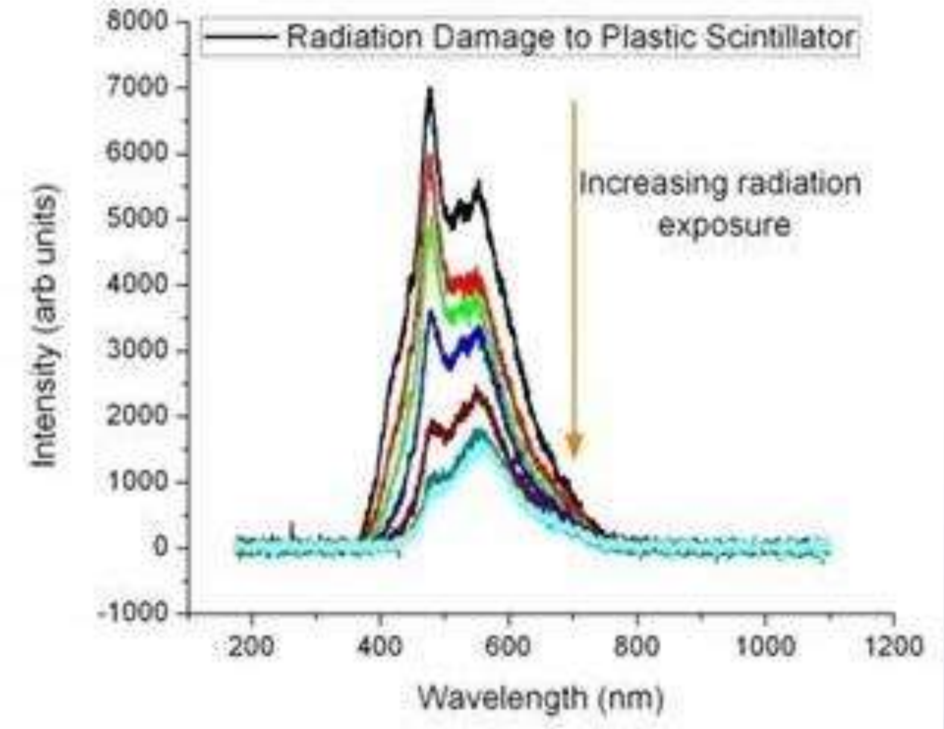
- Radiation hardness experiments performed with 3 MeV H^+ beam from Tandem accelerator
- IBIL spectra measured constantly as sample exposed to beam
- Overall decrease in emitted light observed due to radiation damage

IBIL of MOFs

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

Metal-organic frameworks demonstrate spectral discrimination with IBIL/CL





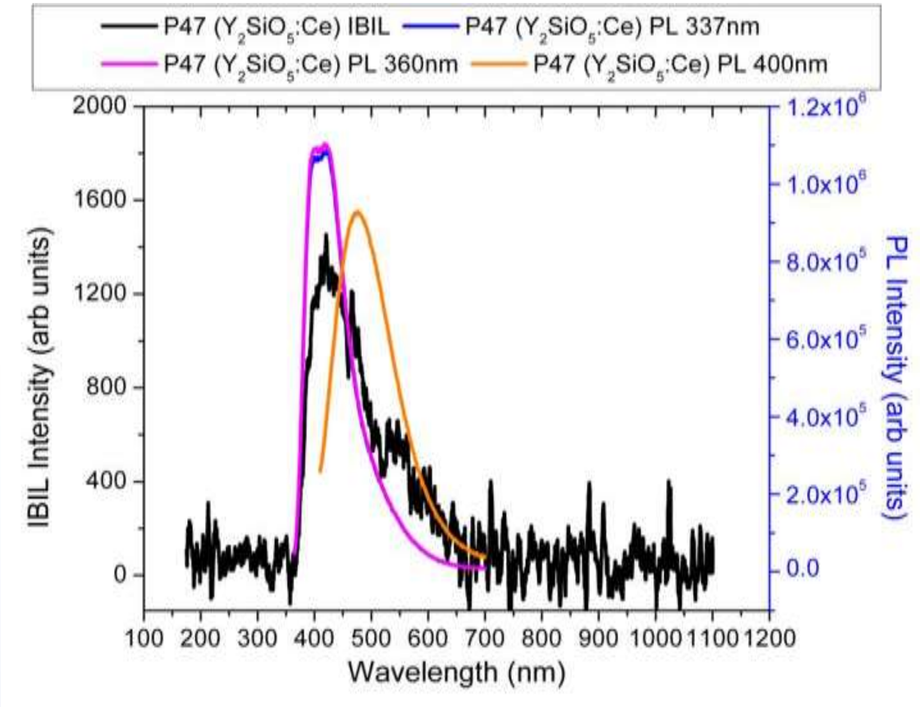
- Spectral discrimination
- CL simulates response to gamma rays
- IBIL simulates response to neutrons

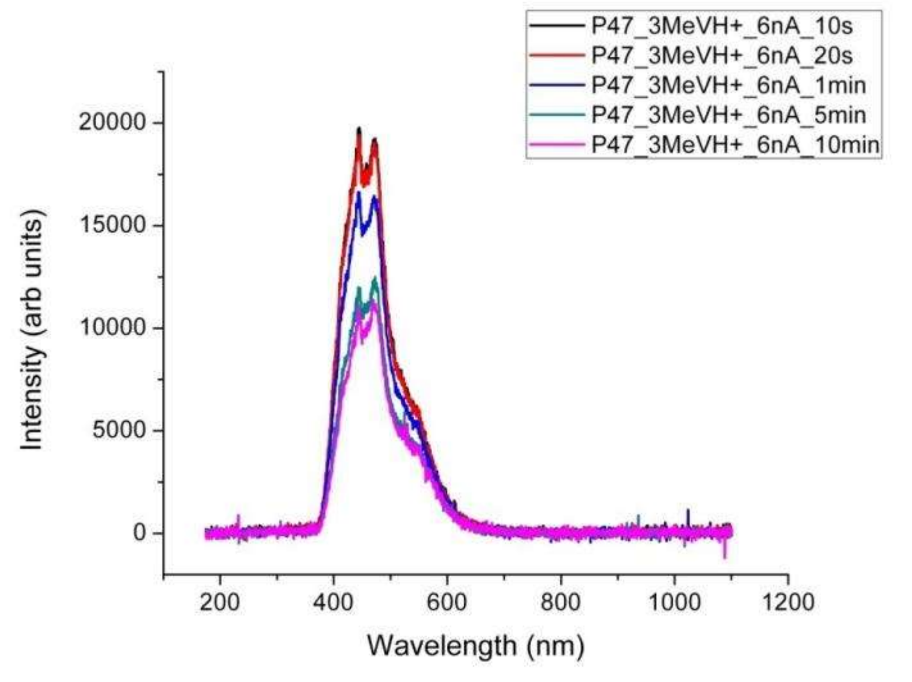
- PL and IBIL of MOF demonstrating spectral discrimination
- IBIL decay of MOFs with irradiation – changes observed in relative peak height

IBIL of Oxides

Collaborators: J. Villone & G. Vizkelethy

P47 phosphor studied for potential in radiation effects microscopy



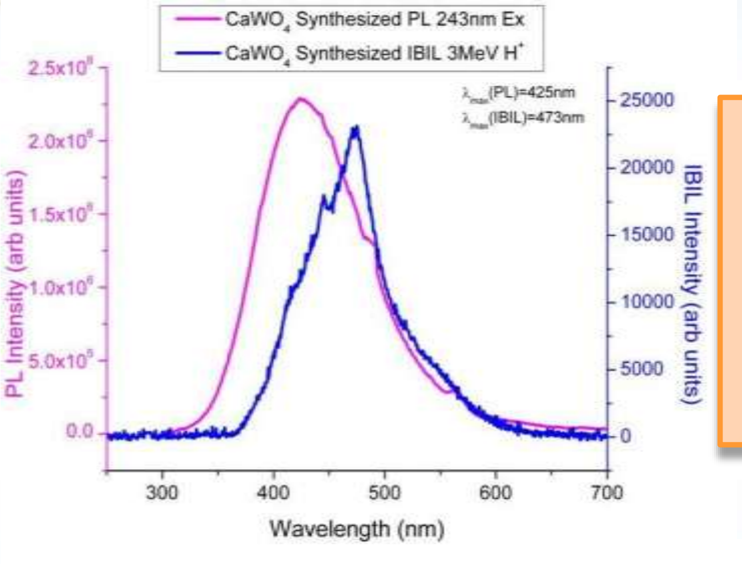


- **P47 is effective phosphor – PL and IBIL similar**
- **Peak emission dependent on excitation wavelength**

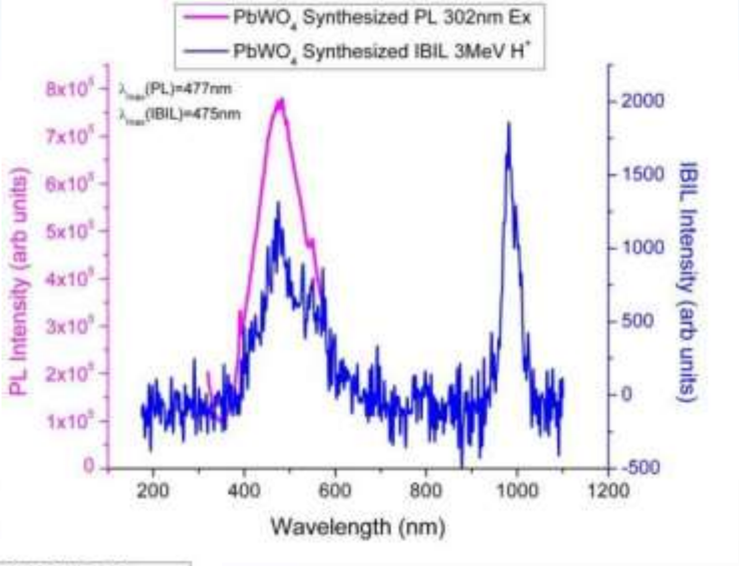
- **Degradation in optical properties also observed in P47**
- **Oxides demonstrate improved radiation tolerance compared to organic scintillators**

IBIL of Nanoscintillators

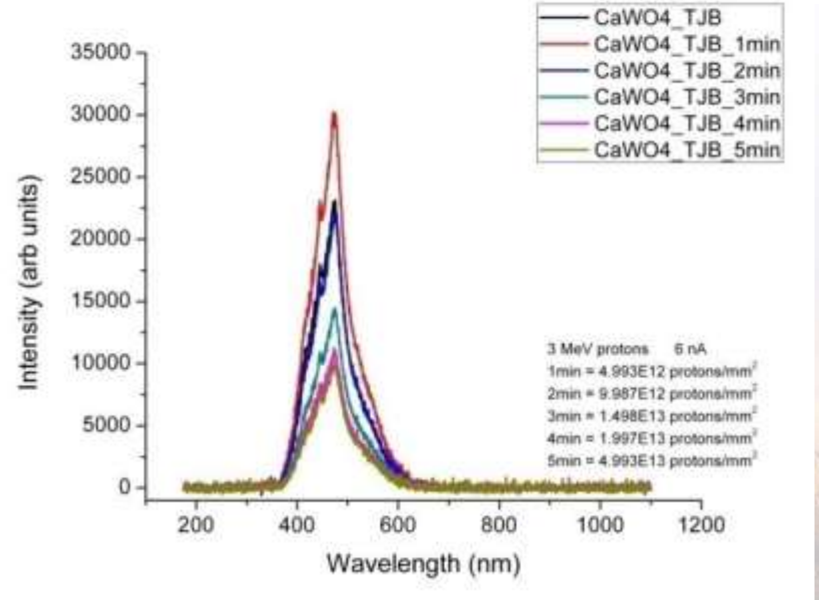
Collaborators: B.A. Hernandez-Sanchez, S.M. Hoppe, T.J. Boyle, J. Villone & P. Yang



Luminescence with proton excitation demonstrates different properties than with UV excitation



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



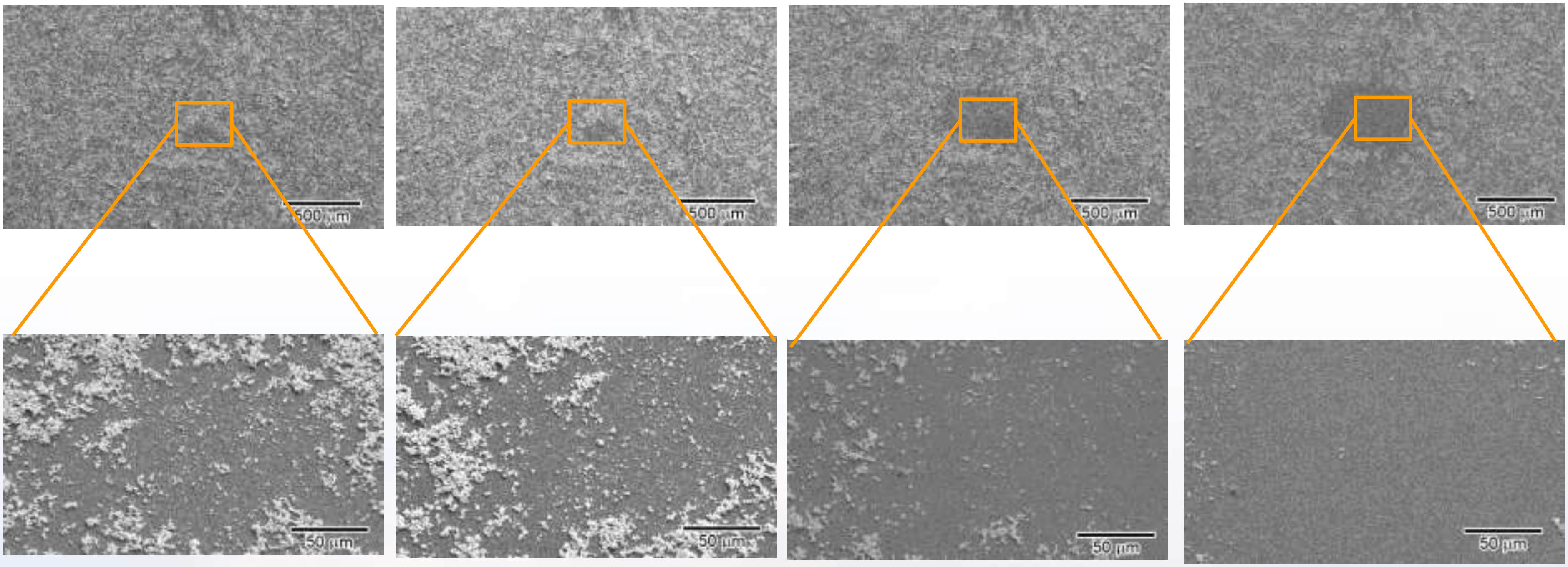
Most materials demonstrate a degradation in optical properties with irradiation – want to understand fundamental mechanism

Sandia National Laboratories

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**

- 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?

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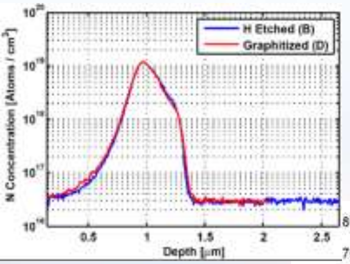
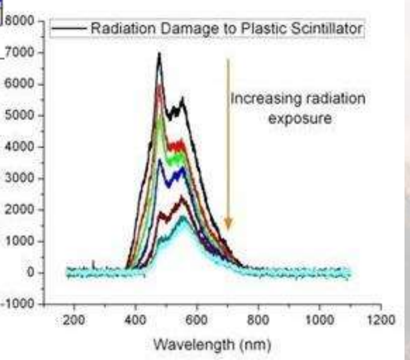
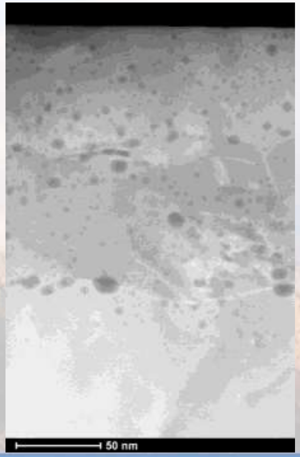
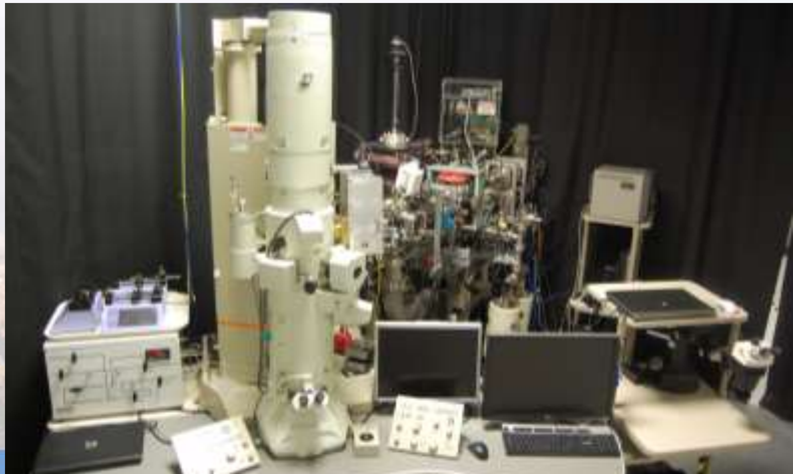
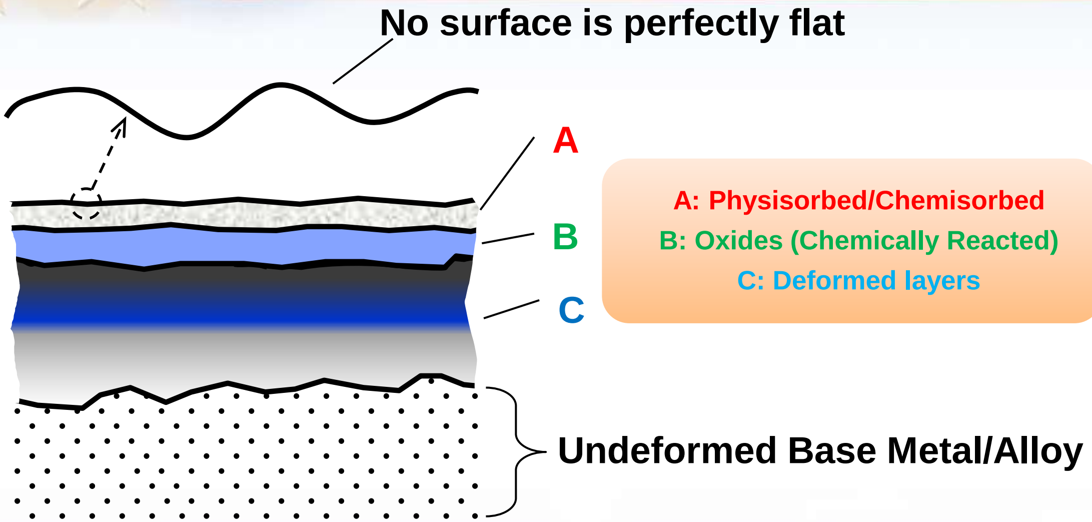








Nature of Metallic Surfaces

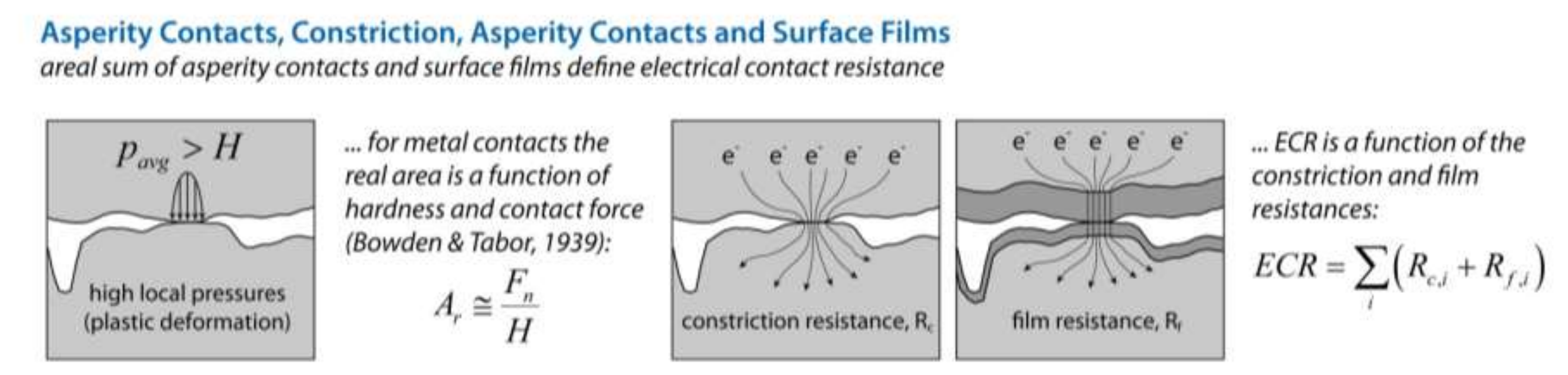


A: Physisorbed/Chemisorbed
B: Oxides (Chemically Reacted)
C: Deformed layers

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

(Low Adhesive Wear)

Or maximized for reduced electrical contact resistance (ECR)



Asperity Contacts, Constriction, Asperity Contacts and Surface Films

areal sum of asperity contacts and surface films define electrical contact resistance

... for metal contacts the real area is a function of hardness and contact force (Bowden & Tabor, 1939):

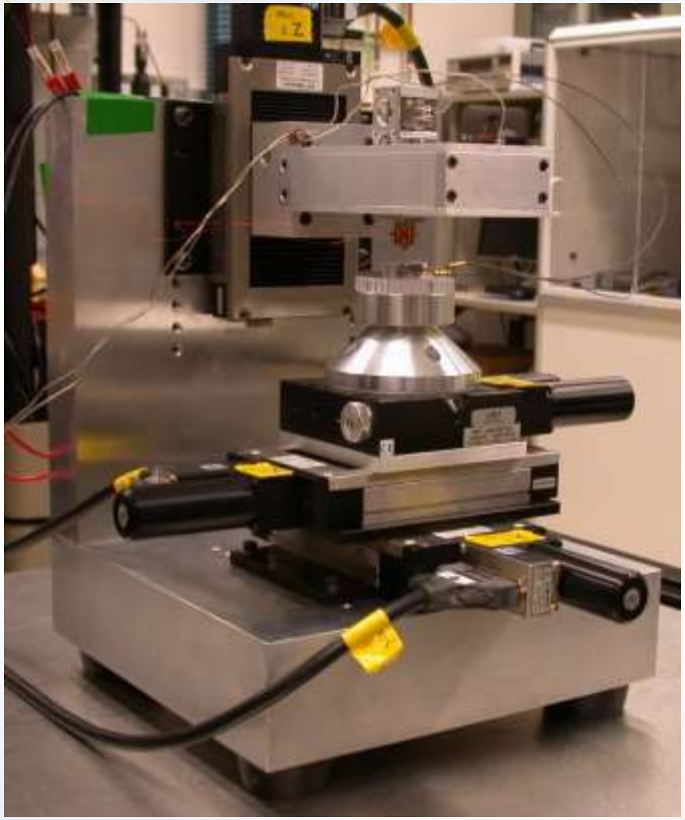
$$A_r \cong \frac{F_n}{H}$$

... ECR is a function of the constriction and film resistances:

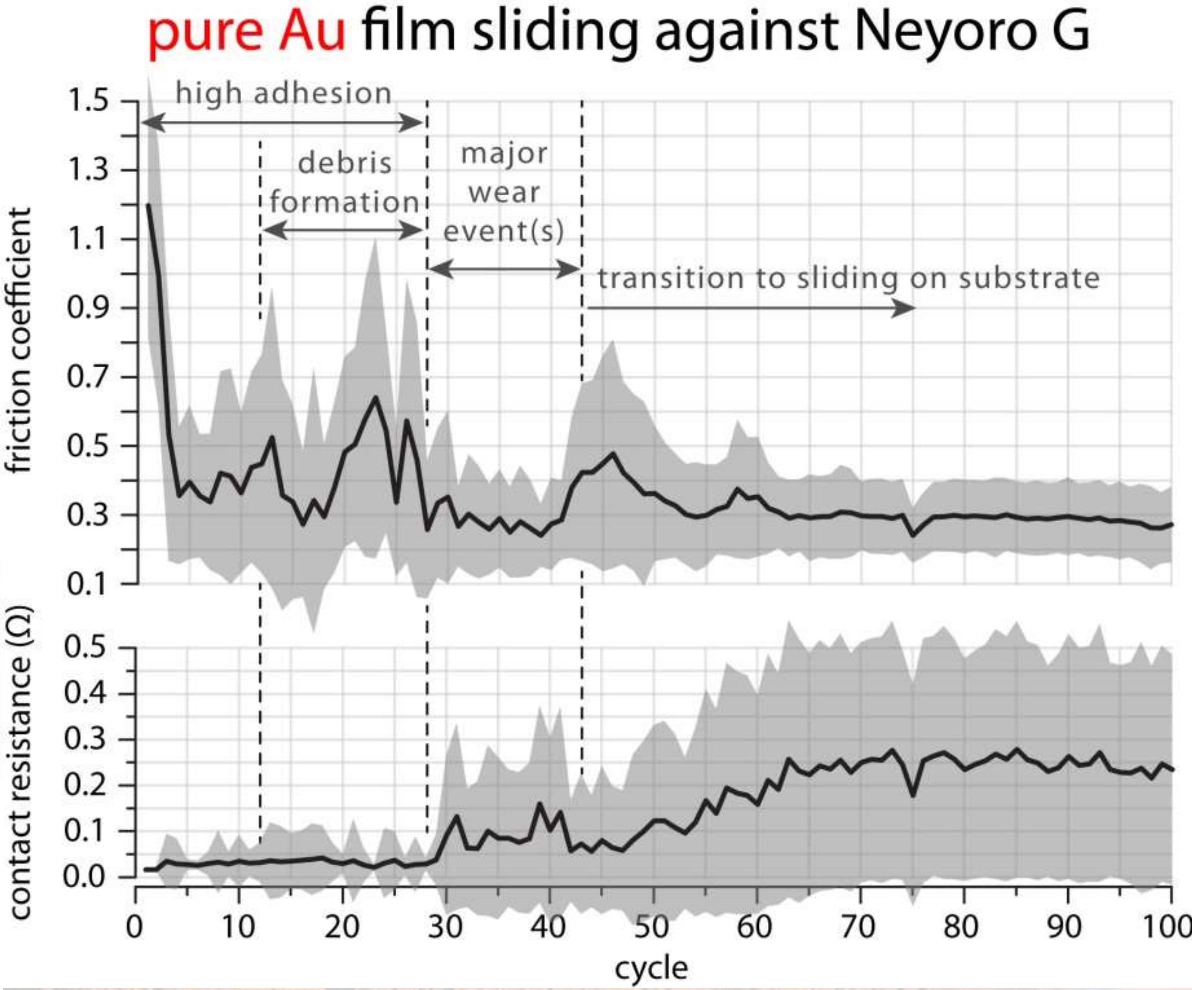
$$ECR = \sum_i \left(R_{c,i} + R_{f,i}\right)$$

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



- **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**



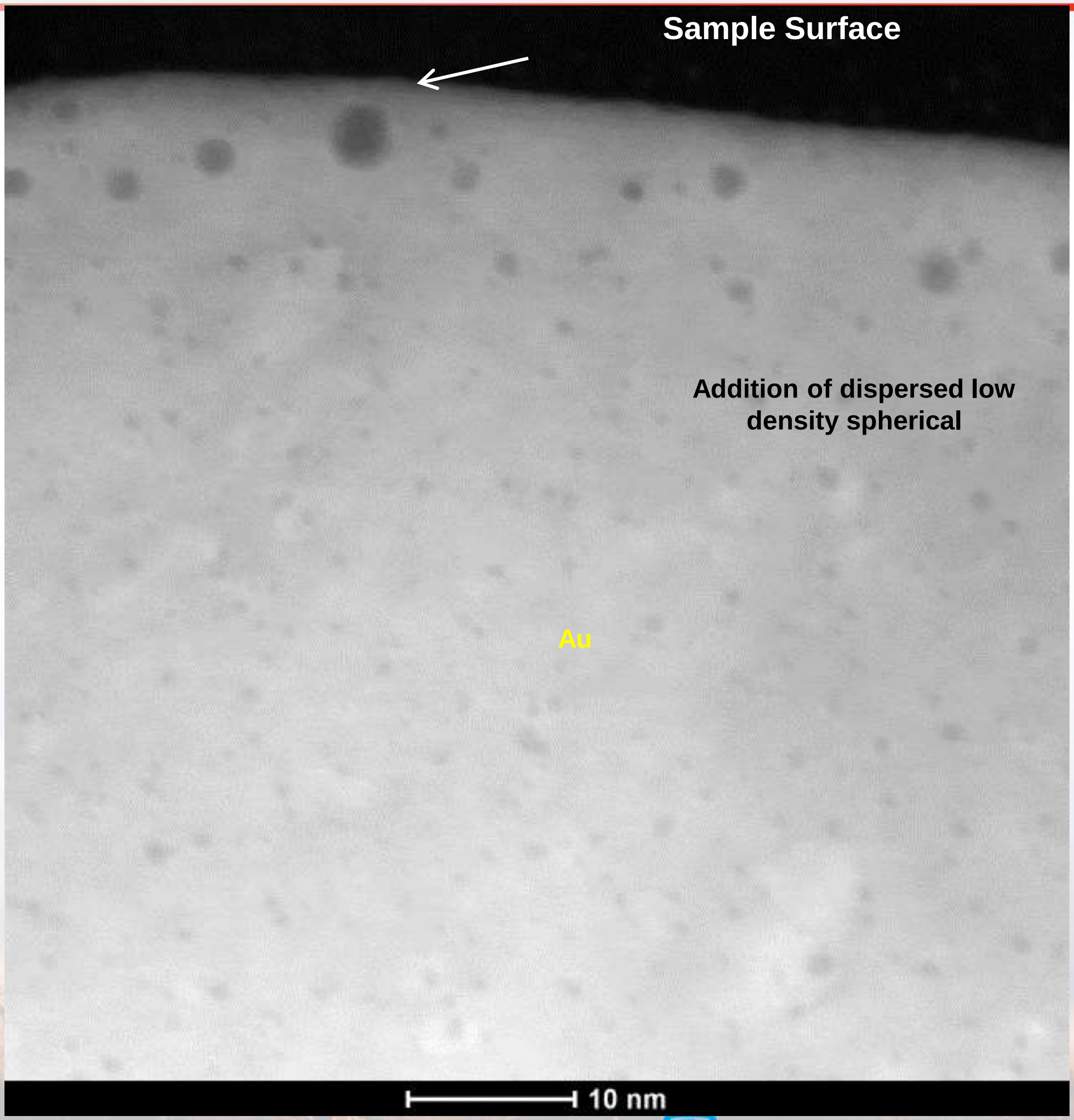
Modeling and STEM of He Implantation

22.5 keV He Ions

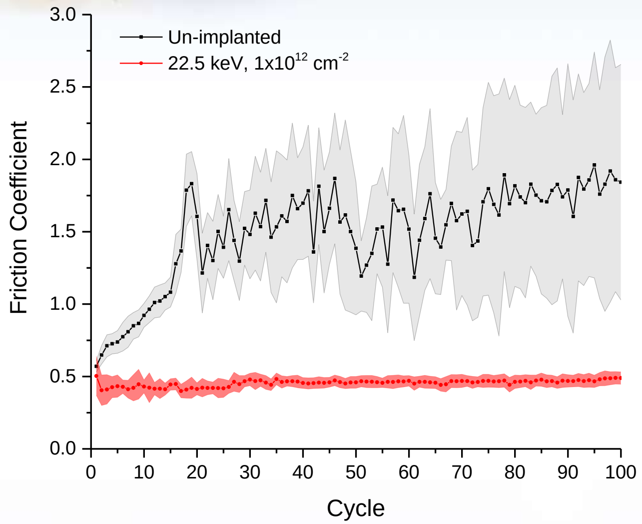
- Simulations: SRIM 2008 (The Stopping and Range of Ions in Matter, J.F. Ziegler, M.D. Ziegler and J.P. Biersack)
  - Monte-Carlo simulation of kinematic interaction based on empirical data fitted functions
  - Input variables of target material include density, AMU, and thickness.
  - Input variables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

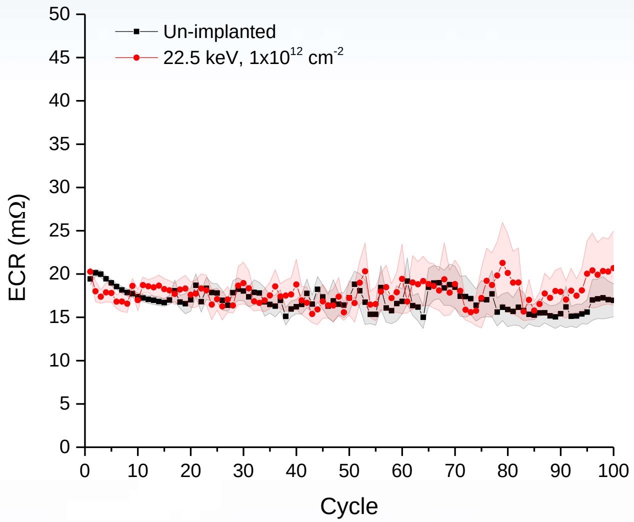
- AC-STEM used to observe the distribution of implanted bubbles
- Bubble locations are in good agreement with SRIM ion range predictions

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



ECR and Wear Measurements

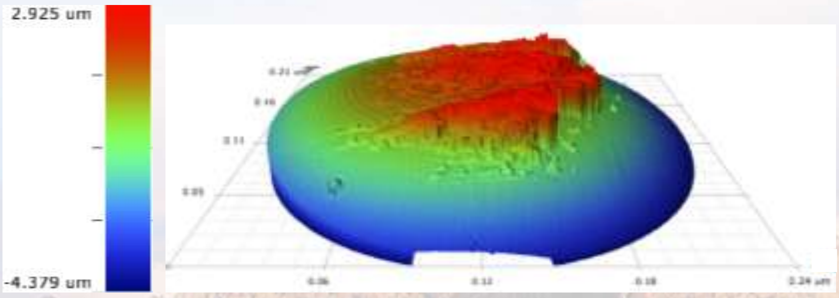




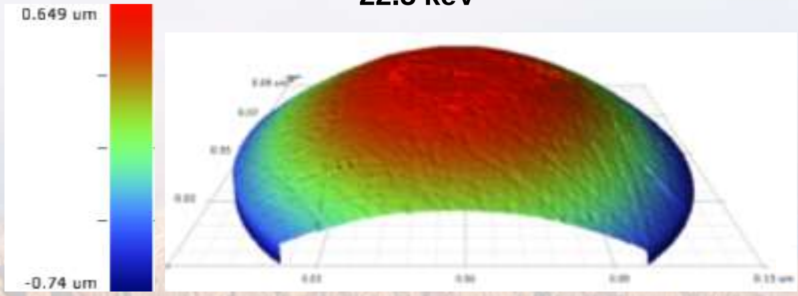
Friction is significantly reduced with ^{3}He implantation while maintaining ECR performance

Scanning white light interferometer topographical construction of riders after 100 cycles

Rider after 100 cycles against Un-implanted Au



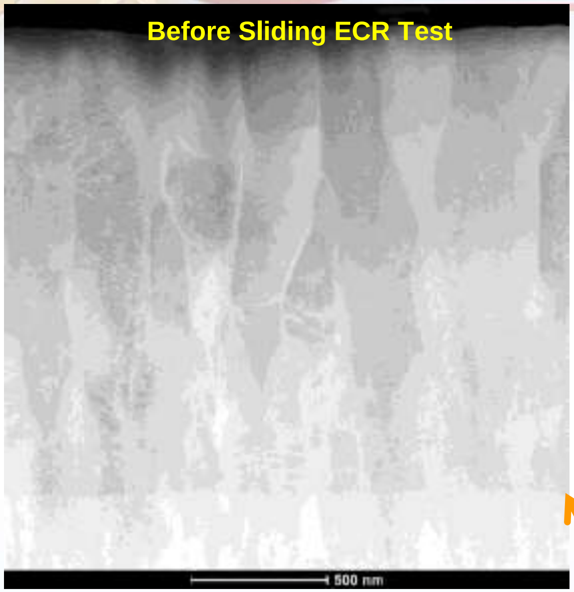
Rider after 100 Cycles against Au implanted to 1E12 cm^{-2} @ 22.5 keV

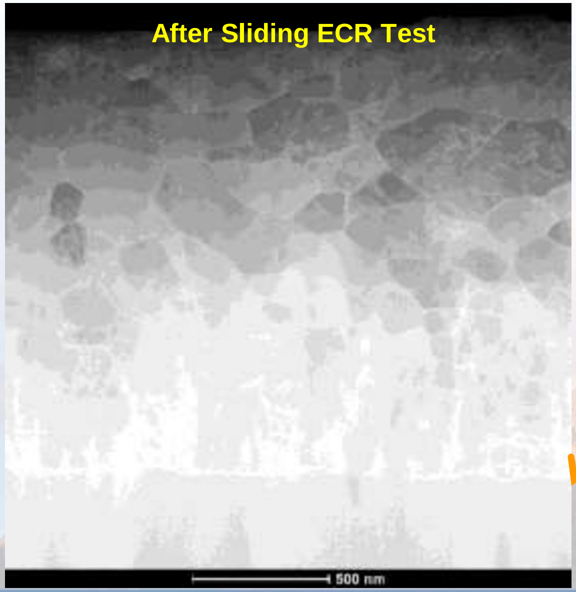


Wear is significantly reduced with minimal effect in ECR

STEM Images of Sub-surfaces

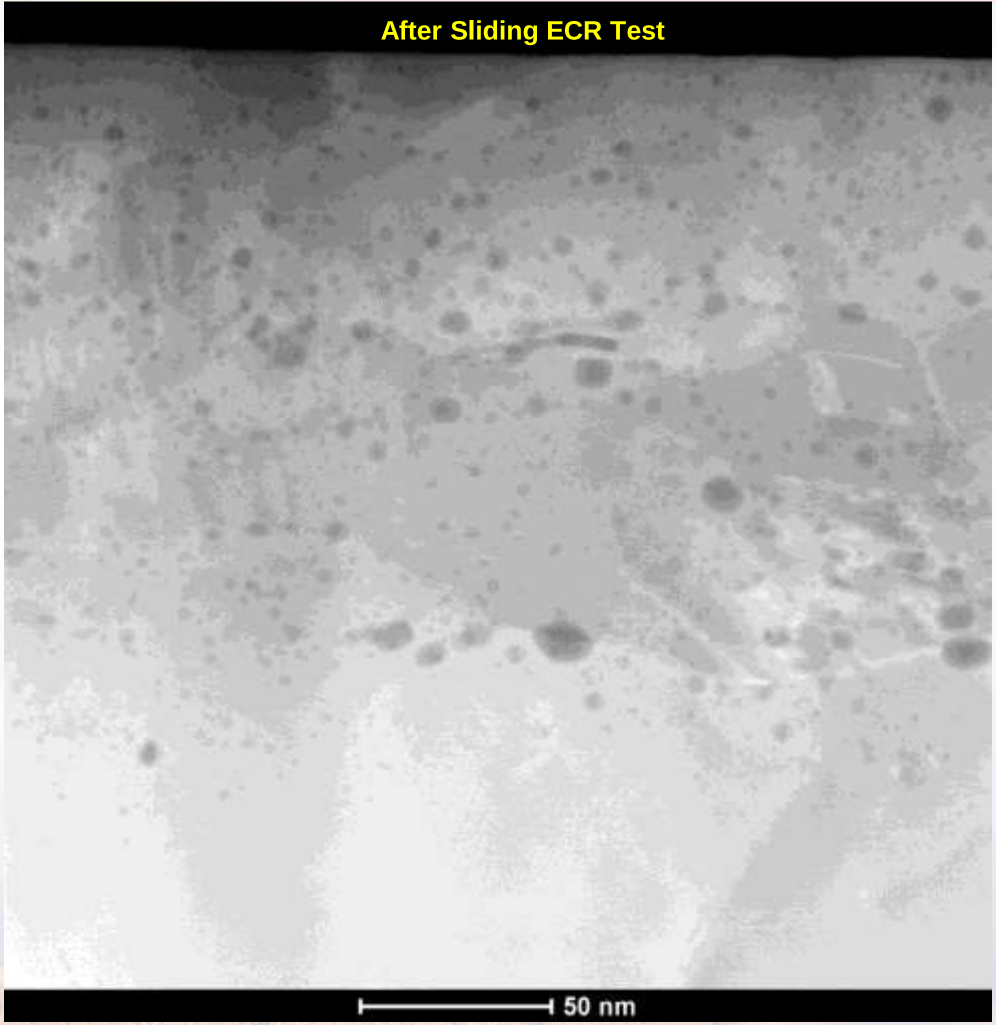
22.5 keV, 1 x 10^{12} cm^{-2}


Before Sliding ECR Test


After Sliding ECR Test

Recrystallization is observable after 100 cycles

Au – Pt Interface

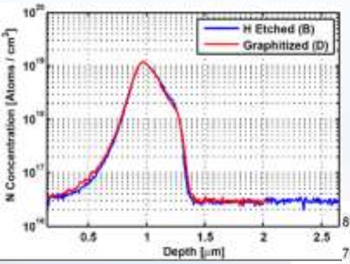

After Sliding ECR Test

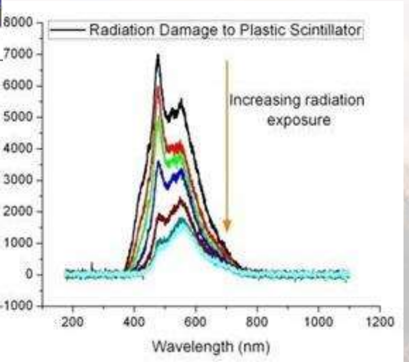
An increase in both observable density and diameter of He bubbles, suggests wear induced He coalescence from interstitial and previously un-observable He

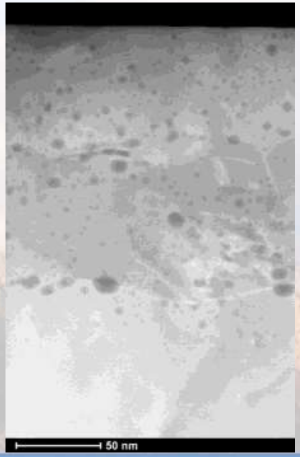
Outline

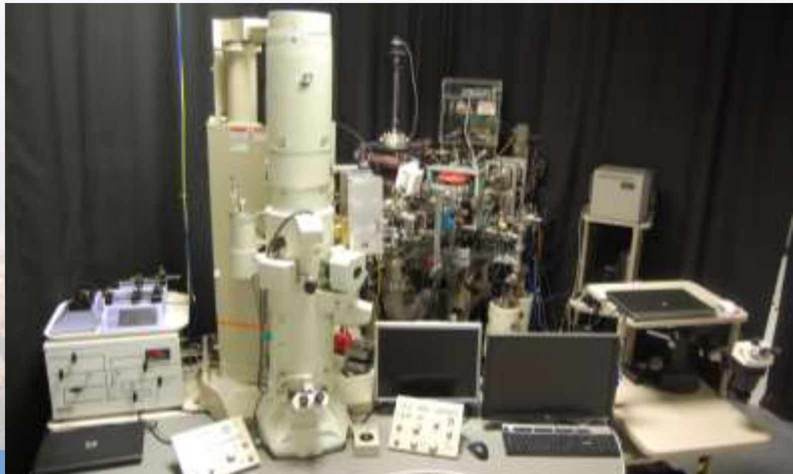
1. **Materials Engineering**
   - Control of Local Chemistry
     - Development of back gates for graphene devices
   - Investigate Radiation Tolerance
     - Various radiation detectors from MOFs to complex oxides
   - Control of Local Structure
     - Improving the wear properties of electrical contacts

2. **Materials Science**
   - Broad Capability of the I^{3}TEM
   - Revisit of the Radiation Detectors
     - In-situ ion irradiation of high-z nanoscintillators
   - Revisit of the Structural Control
     - In-situ study of Au electrode evolution under ion beam modification
     - Initial work on indentation and cyclic compression
   - Fun future directions for real-time nanoscale observation in extreme environments









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

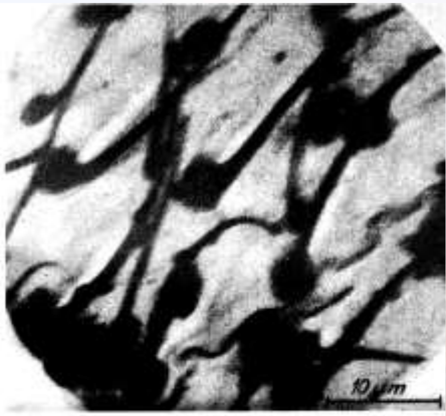


Fig. 6: Wing surface of the house fly
(First internal photographic: U = 80 kV, M = 2200)
(Driest, E., and Müller, H.O.: Z. Wiss. Mikroskopie 52, 53-57 (1935))

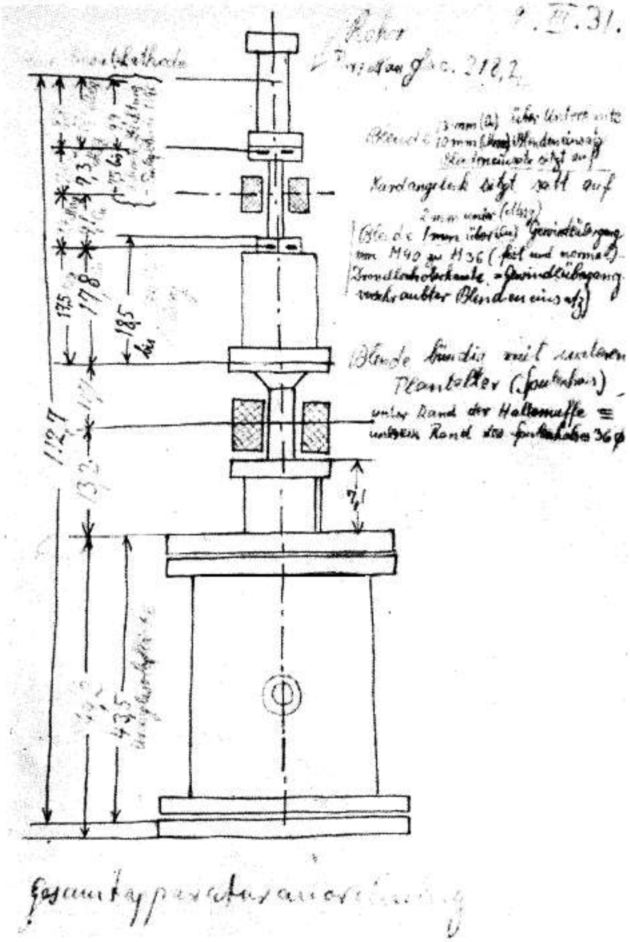
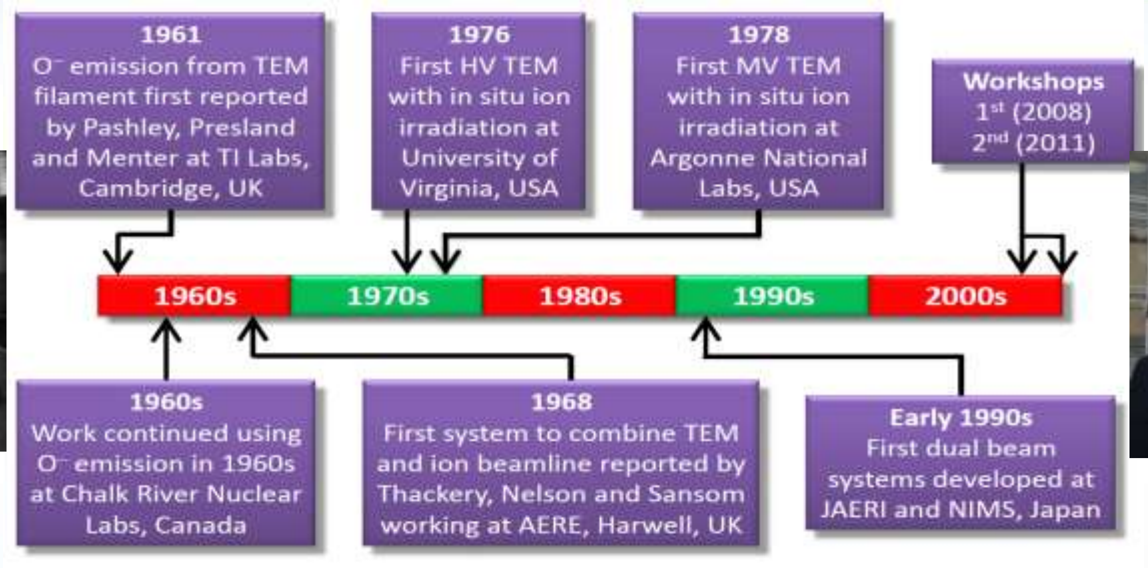


Fig. 2: Sketch by the author (9 March 1931) of the cathode ray tube for testing one-stage and two-stage electron-optical imaging by means of two magnetic electron lenses (electron microscope) [8].

E. Ruska Nobel lecture (1986)

History of *in situ* Irradiation TEM

Courtesy of: J. Hinks



1961
O^- emission from TEM filament first reported by Pashley, Presland and Menter at TI Labs, Cambridge, UK

1976
First HV TEM with in situ ion irradiation at University of Virginia, USA

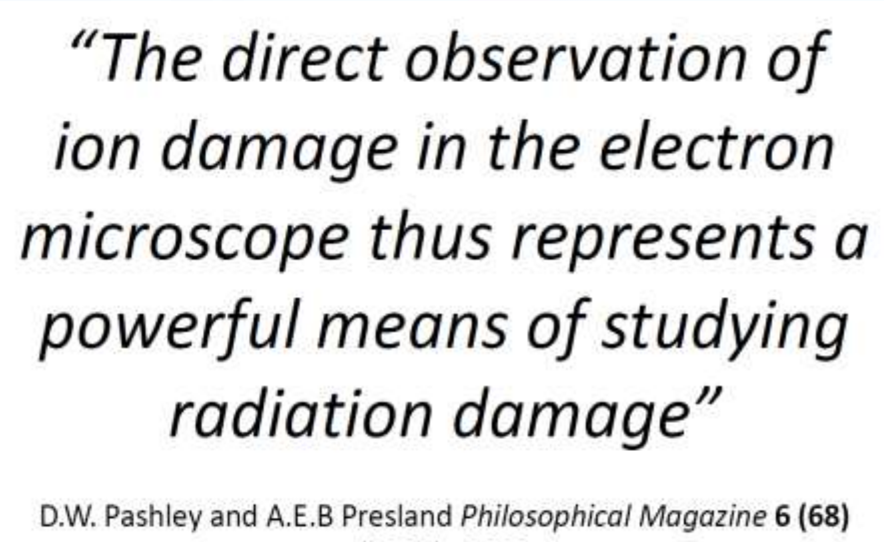
1978
First MV TEM with in situ ion irradiation at Argonne National Labs, USA

Workshops
1st (2008)
2nd (2011)

1960s
Work continued using O^- emission in 1960s at Chalk River Nuclear Labs, Canada

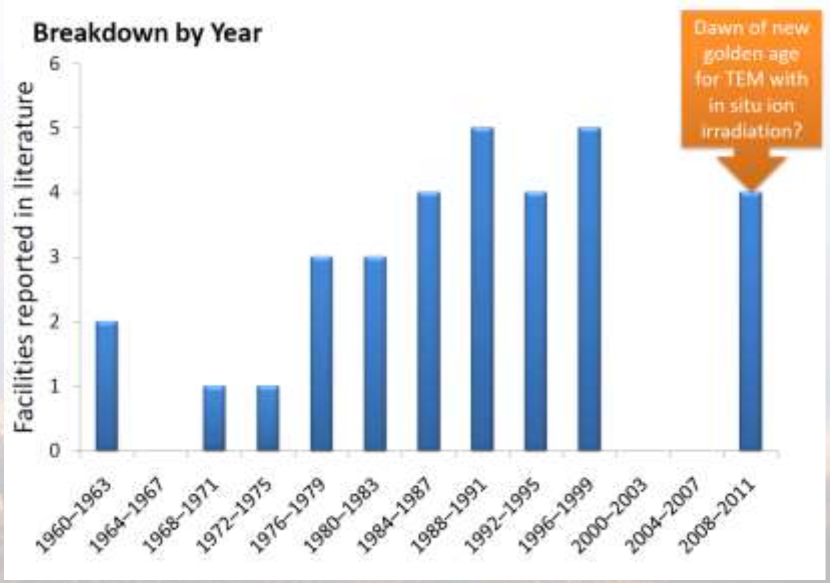
1968
First system to combine TEM and ion beamline reported by Thackery, Nelson and Sansom working at AERE, Harwell, UK

Early 1990s
First dual beam systems developed at JAERI and NIMS, Japan



"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 *Philosophical Magazine* **6 (68)** (1961) p1003

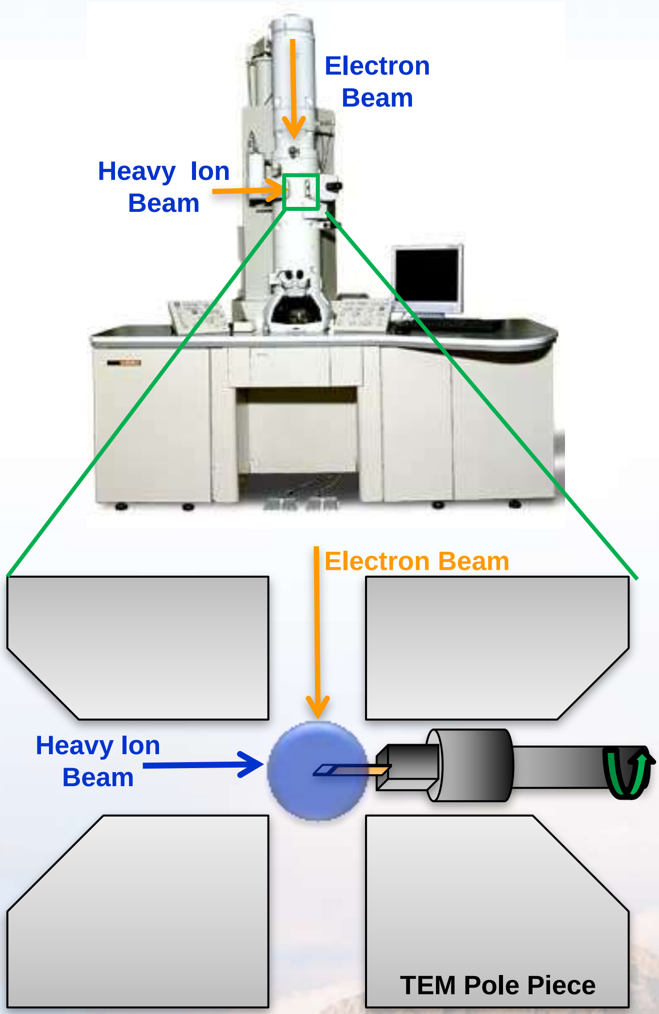


Breakdown by Year

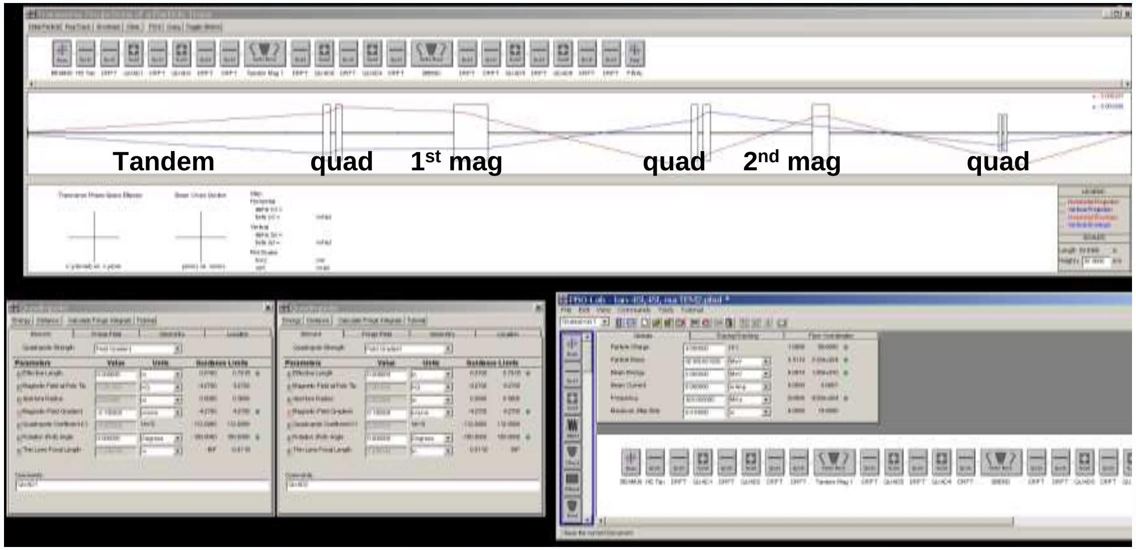
Dawn of new golden age for TEM with in situ ion irradiation?

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

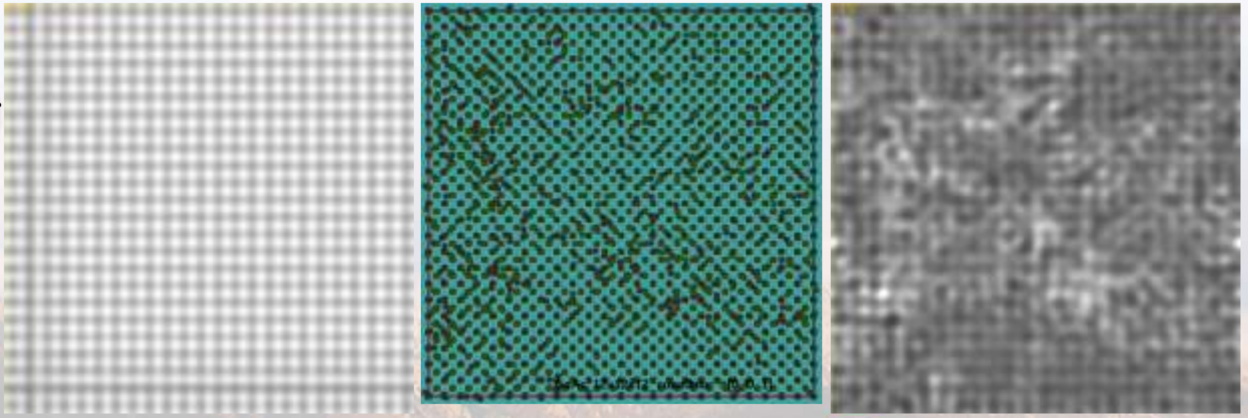
Collaborators: P. Rossi, D.L. Buller, & B.L. Doyle



Ion Optics Feasibility Study

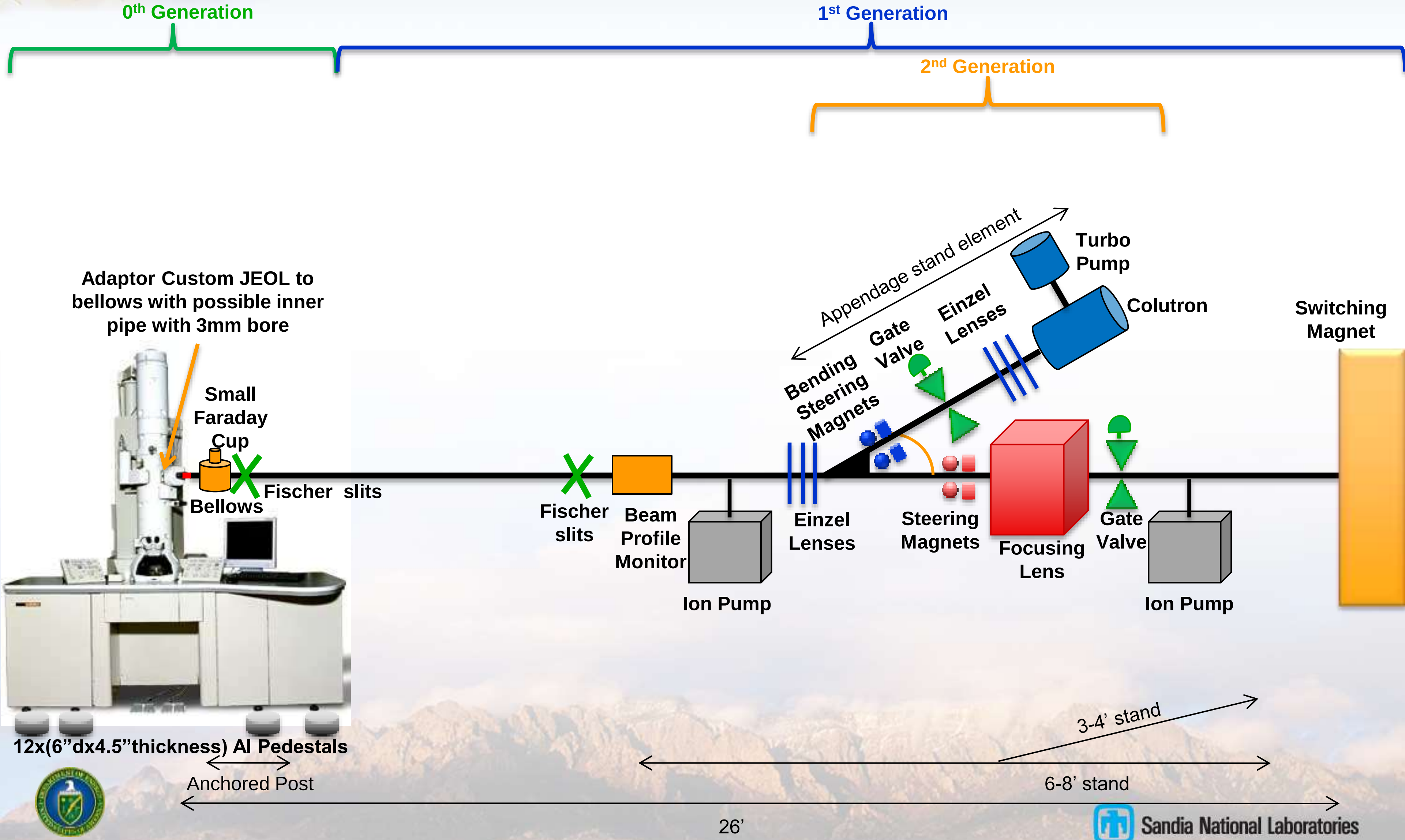


TEM image simulation of radiation damage

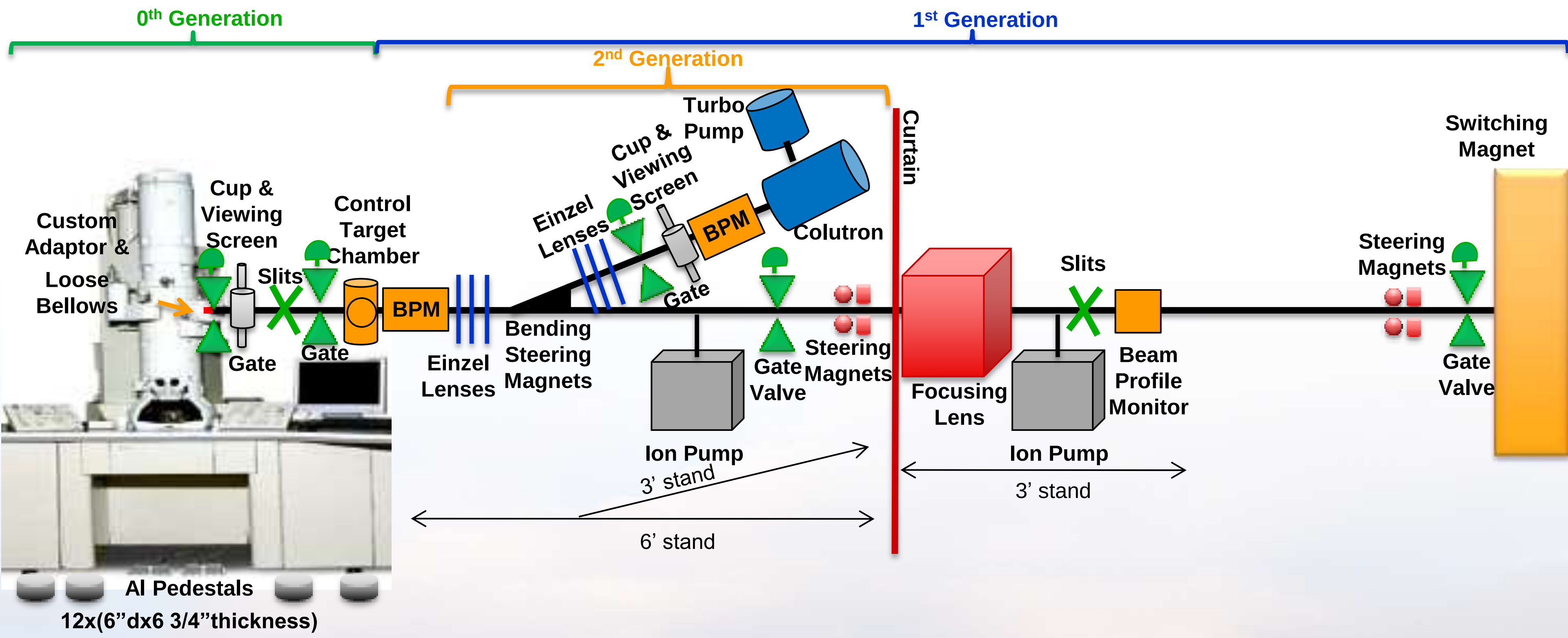

JEMS undamaged | Marlowe | JEMS damaged

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

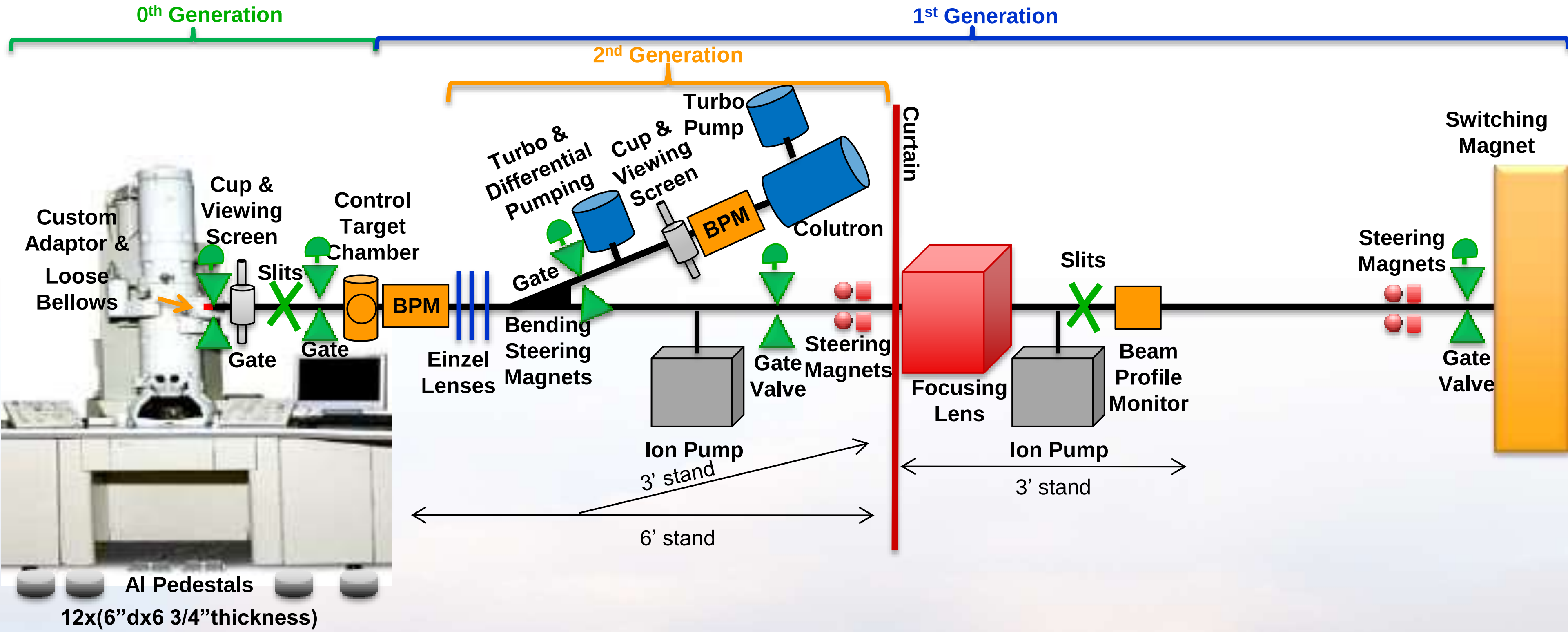
Schematic of the *In situ* TEM Beamline



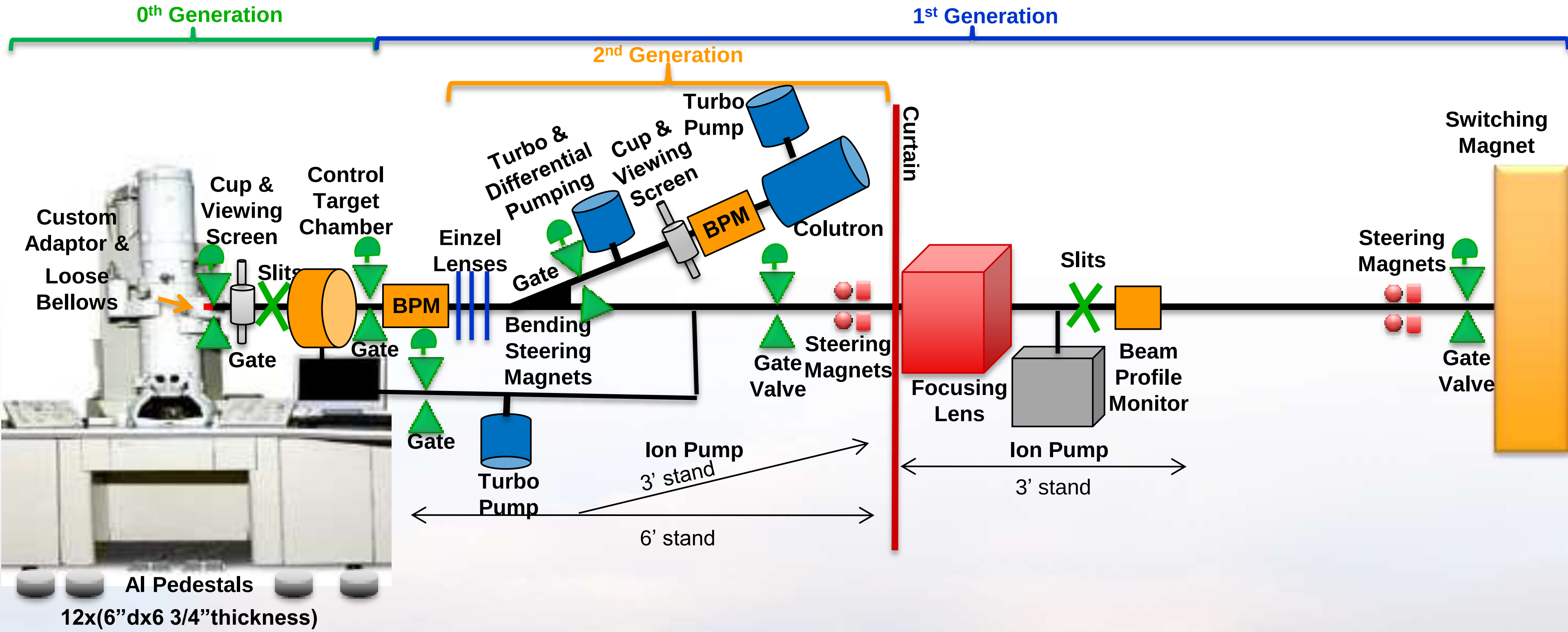
Schematic of the *In situ* TEM Beamline



Schematic of the *In situ* TEM Beamline



Schematic of the *In situ* TEM Beamline



0th Generation

1st Generation

2nd Generation

Custom Adaptor & Loose Bellows

Cup & Viewing Screen

Slits

Gate

Control Target Chamber

Gate

BPM

Einzel Lenses

Gate

Turbo & Differential Pumping

Gate

Bending Steering Magnets

Cup & Viewing Screen

BPM

Turbo Pump

Colutron

Gate

Turbo Pump

Ion Pump

3' stand

6' stand

Gate Valve

Steering Magnets

Curtain

Focusing Lens

Slits

Beam Profile Monitor

Ion Pump

3' stand

Steering Magnets

Gate Valve

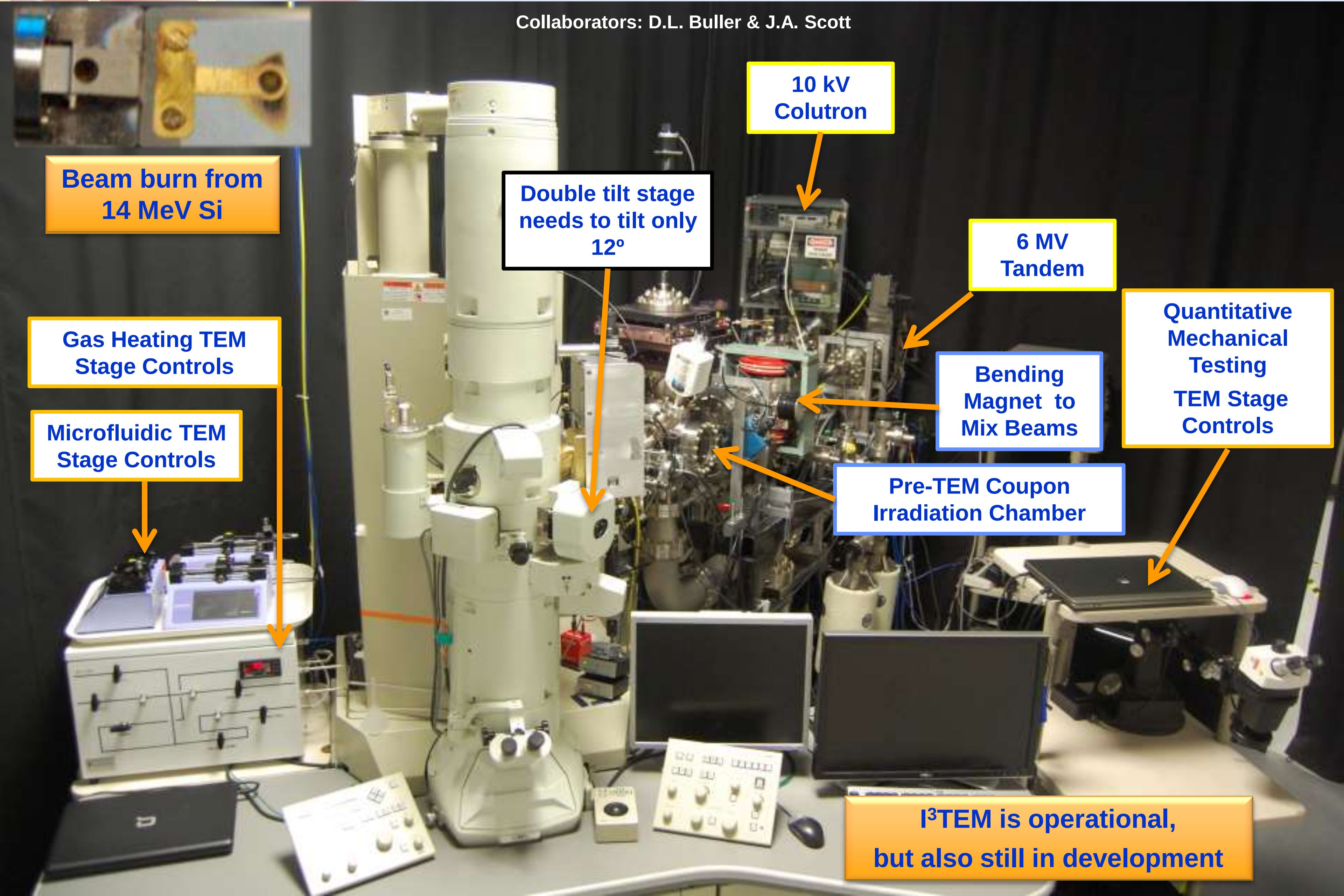
Switching Magnet

Al Pedestals

12x(6"dx6 3/4"thickness)

Current Status of the *In situ* TEM Beamline

Collaborators: D.L. Buller & J.A. Scott



Beam burn from 14 MeV Si

10 kV Colutron

Double tilt stage needs to tilt only 12º

6 MV Tandem

Quantitative Mechanical Testing

TEM Stage Controls

Gas Heating TEM Stage Controls

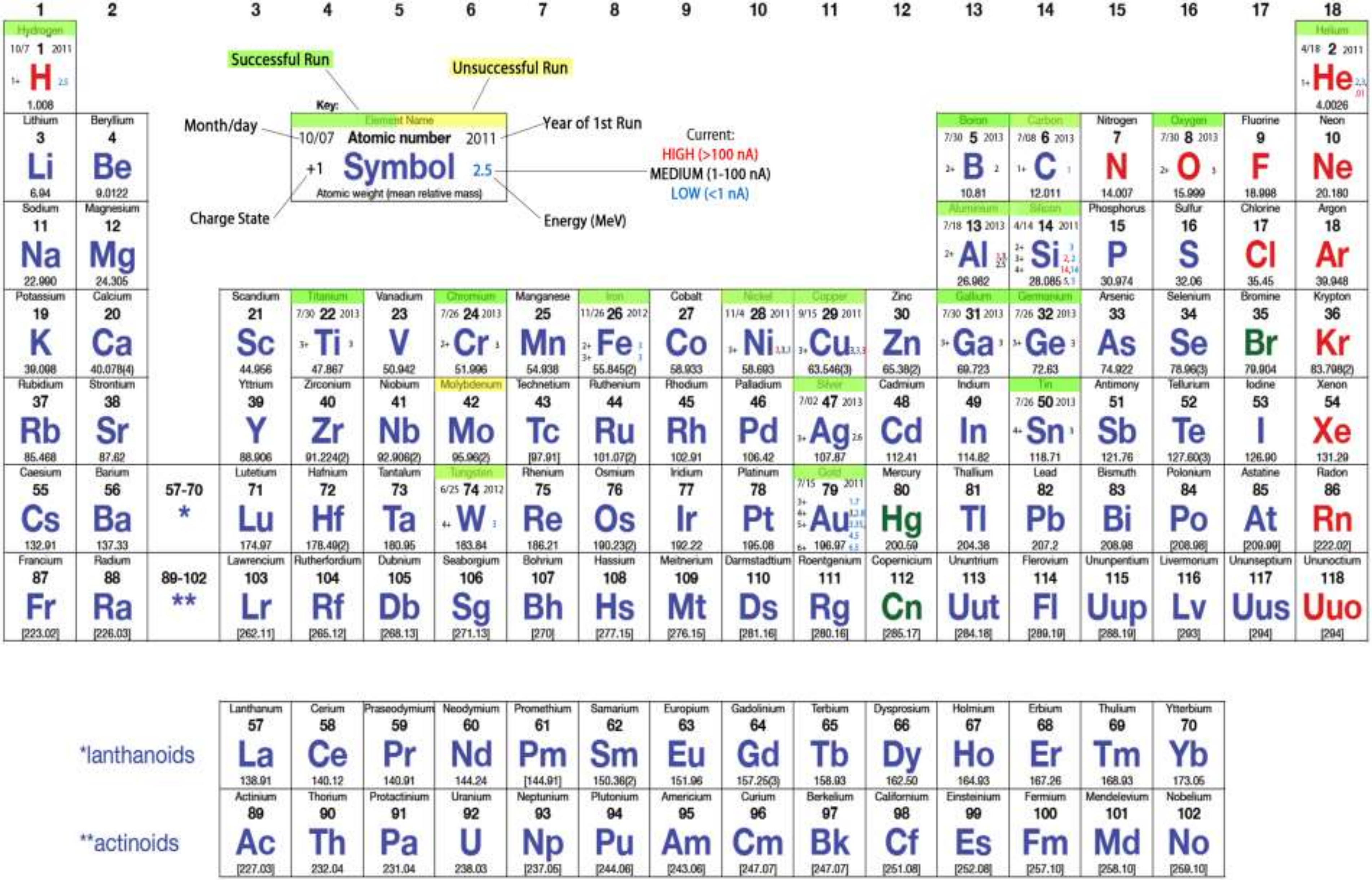
Bending Magnet to Mix Beams

Microfluidic TEM Stage Controls

Pre-TEM Coupon Irradiation Chamber

I^3TEM is operational, but also still in development

Ion Species Attempted as of 7/23/2013



Successful Run

Unsuccessful Run

Key:

Month/day

Element Name

10/07 Atomic number 2011

+1 Symbol 2.5

Atomic weight (mean relative mass)

Year of 1st Run

Charge State

Energy (MeV)

Current:
HIGH (>100 nA)
MEDIUM (1-100 nA)
LOW (<1 nA)

1	2		3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Hydrogen 10/7 1 2011 1+ H 2.5 1.008																		Helium 4/18 2 2011 1+ He 2.3, .01 4.0026
Lithium 3 Li 6.94	Beryllium 4 Be 9.0122												Boron 7/30 5 2013 2+ B 2 10.81	Carbon 7/08 6 2013 1+ C 1 12.011	Nitrogen 7 N 14.007	Oxygen 7/30 8 2013 2+ O 3 15.999	Fluorine 9 F 18.998	Neon 10 Ne 20.180
Sodium 11 Na 22.990	Magnesium 12 Mg 24.305												Aluminium 7/18 13 2013 2+ Al 3.3, 2.5 26.982	Silicon 4/14 14 2011 2+ 3+ 4+ Si 3, 2, 2, 14, 14, 5, 1 28.085	Phosphorus 15 P 30.974	Sulfur 16 S 32.06	Chlorine 17 Cl 35.45	Argon 18 Ar 39.948
Potassium 19 K 39.098	Calcium 20 Ca 40.078(4)		Scandium 21 Sc 44.956	Titanium 7/30 22 2013 3+ Ti 3 47.867	Vanadium 23 V 50.942	Chromium 7/26 24 2013 3+ Cr 3 51.996	Manganese 25 Mn 54.938	Iron 11/26 26 2012 2+ 3+ Fe 3, 3 55.845(2)	Cobalt 27 Co 58.933	Nickel 11/4 28 2011 3+ Ni 3,3,3 58.693	Copper 9/15 29 2011 3+ Cu 3,3,3 63.546(3)	Zinc 30 Zn 65.38(2)	Gallium 7/30 31 2013 3+ Ga 3 69.723	Germanium 7/26 32 2013 3+ Ge 3 72.63	Arsenic 33 As 74.922	Selenium 34 Se 78.96(3)	Bromine 35 Br 79.904	Krypton 36 Kr 83.798(2)
Rubidium 37 Rb 85.468	Strontium 38 Sr 87.62		Yttrium 39 Y 88.906	Zirconium 40 Zr 91.224(2)	Niobium 41 Nb 92.906(2)	Molybdenum 42 Mo 95.96(2)	Technetium 43 Tc [97.91]	Ruthenium 44 Ru 101.07(2)	Rhodium 45 Rh 102.91	Palladium 46 Pd 106.42	Silver 7/02 47 2013 3+ Ag 2.6 107.87	Cadmium 48 Cd 112.41	Indium 49 In 114.82	Tin 7/26 50 2013 4+ Sn 3 118.71	Antimony 51 Sb 121.76	Tellurium 52 Te 127.60(3)	Iodine 53 I 126.90	Xenon 54 Xe 131.29
Caesium 55 Cs 132.91	Barium 56 Ba 137.33	57-70 *	Lutetium 71 Lu 174.97	Hafnium 72 Hf 178.49(2)	Tantalum 73 Ta 180.95	Tungsten 6/25 74 2012 4+ W 3 183.84	Rhenium 75 Re 186.21	Osmium 76 Os 190.23(2)	Iridium 77 Ir 192.22	Platinum 78 Pt 195.08	Gold 7/15 79 2011 3+ 4+ 5+ 6+ Au 1.7, 2.8, 3.15, 4.5, 6.5 196.97	Mercury 80 Hg 200.59	Thallium 81 Tl 204.38	Lead 82 Pb 207.2	Bismuth 83 Bi 208.98	Polonium 84 Po [208.98]	Astatine 85 At [209.99]	Radon 86 Rn [222.02]
Francium 87 Fr [223.02]	Radium 88 Ra [226.03]	89-102 **	Lawrencium 103 Lr [262.11]	Rutherfordium 104 Rf [265.12]	Dubnium 105 Db [268.13]	Seaborgium 106 Sg [271.13]	Bohrium 107 Bh [270]	Hassium 108 Hs [277.15]	Meitnerium 109 Mt [276.15]	Darmstadtium 110 Ds [281.16]	Roentgenium 111 Rg [280.16]	Copernicium 112 Cn [285.17]	Ununtrium 113 Uut [284.18]	Flerovium 114 Fl [289.19]	Ununpentium 115 Uup [288.19]	Livermorium 116 Lv [293]	Ununseptium 117 Uus [294]	Ununoctium 118 Uuo [294]

*lanthanoids	Lanthanum 57 La 138.91	Cerium 58 Ce 140.12	Praseodymium 59 Pr 140.91	Neodymium 60 Nd 144.24	Promethium 61 Pm [144.91]	Samarium 62 Sm 150.36(2)	Europium 63 Eu 151.96	Gadolinium 64 Gd 157.25(3)	Terbium 65 Tb 158.93	Dysprosium 66 Dy 162.50	Holmium 67 Ho 164.93	Erbium 68 Er 167.26	Thulium 69 Tm 168.93	Ytterbium 70 Yb 173.05
**actinoids	Actinium 89 Ac [227.03]	Thorium 90 Th 232.04	Protactinium 91 Pa 231.04	Uranium 92 U 238.03	Neptunium 93 Np [237.05]	Plutonium 94 Pu [244.06]	Americium 95 Am [243.06]	Curium 96 Cm [247.07]	Berkelium 97 Bk [247.07]	Californium 98 Cf [251.08]	Einsteinium 99 Es [252.08]	Fermium 100 Fm [257.10]	Mendelevium 101 Md [258.10]	Nobelium 102 No [259.10]

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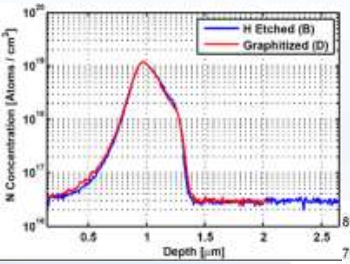
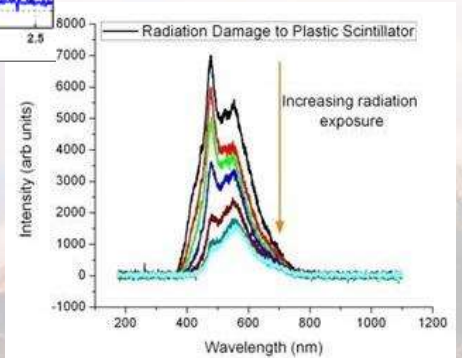
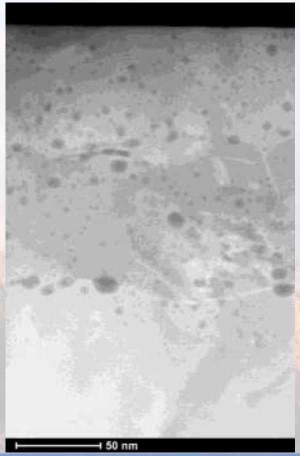
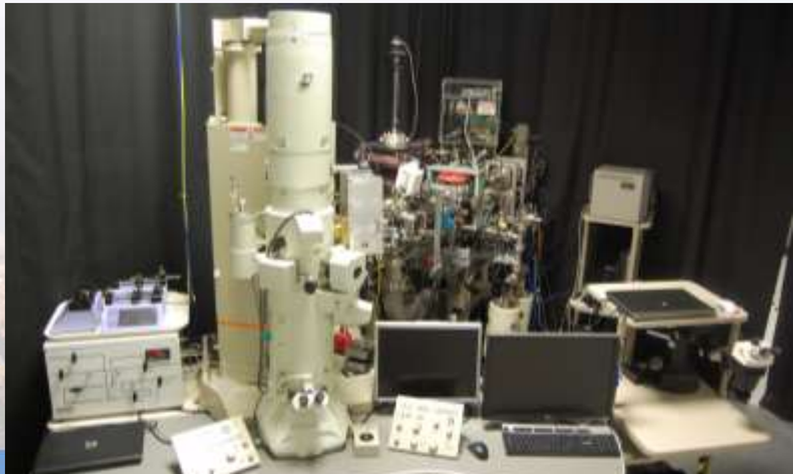


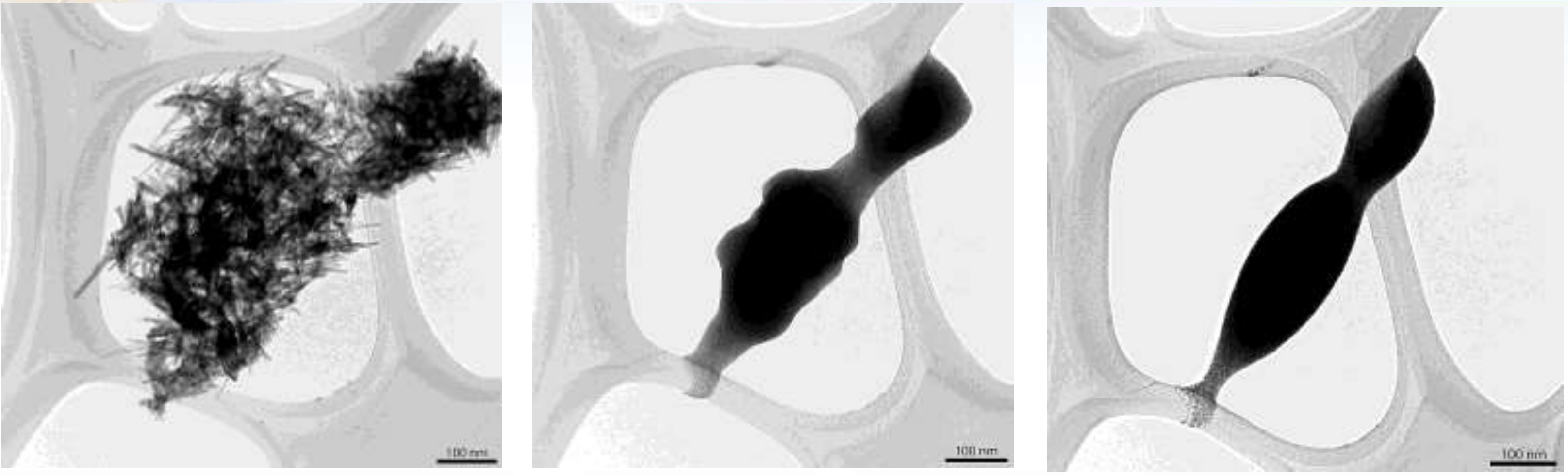


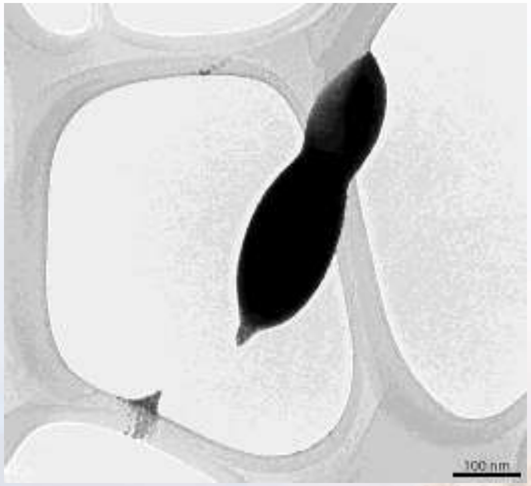


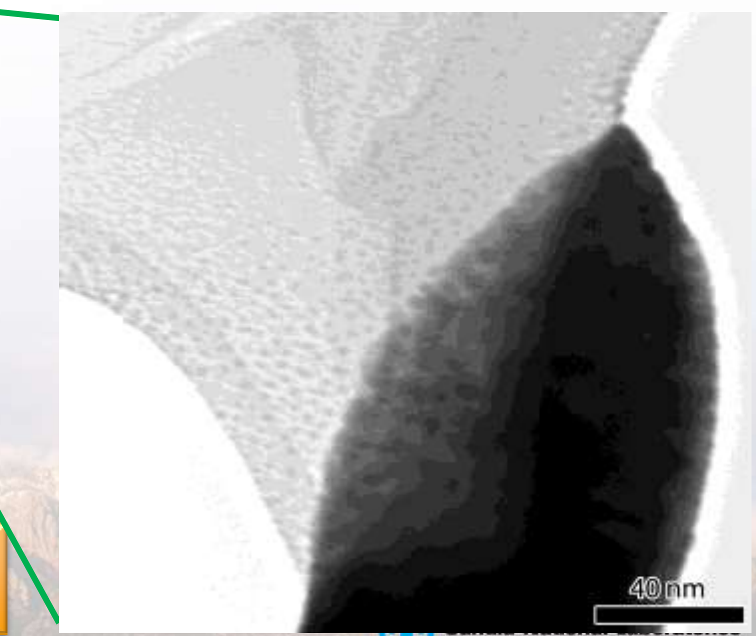


$CdWO_4$ Irradiated with 50 nA of 3 MeV Cu^{3+}

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



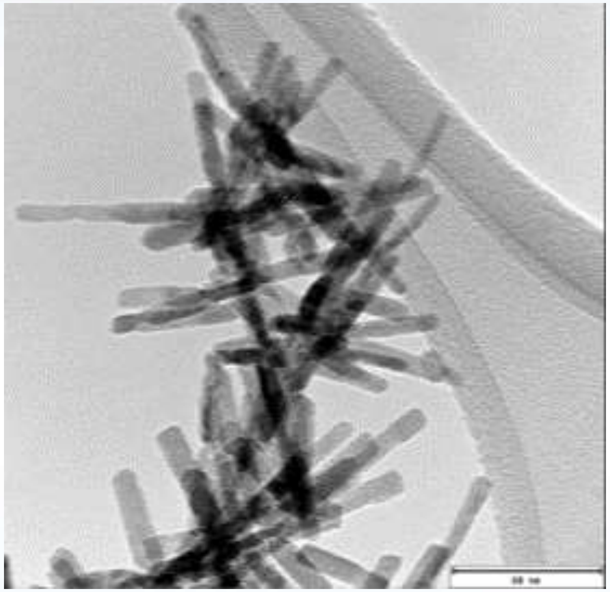




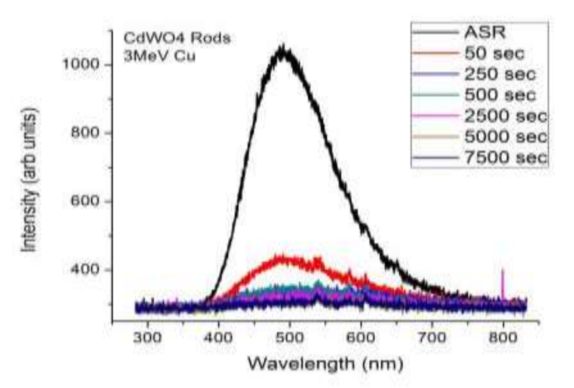
Over 1 hr, nanorods broke into small pieces and sputtered onto nearby lace.

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

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



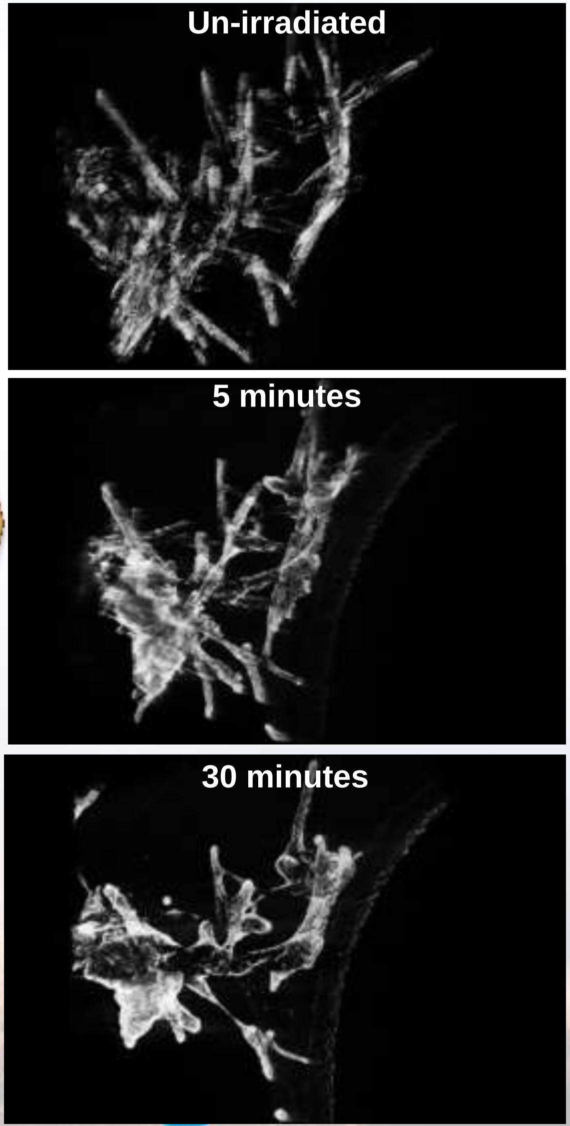
High-Z nanoparticles are promising, but are radiation sensitive



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

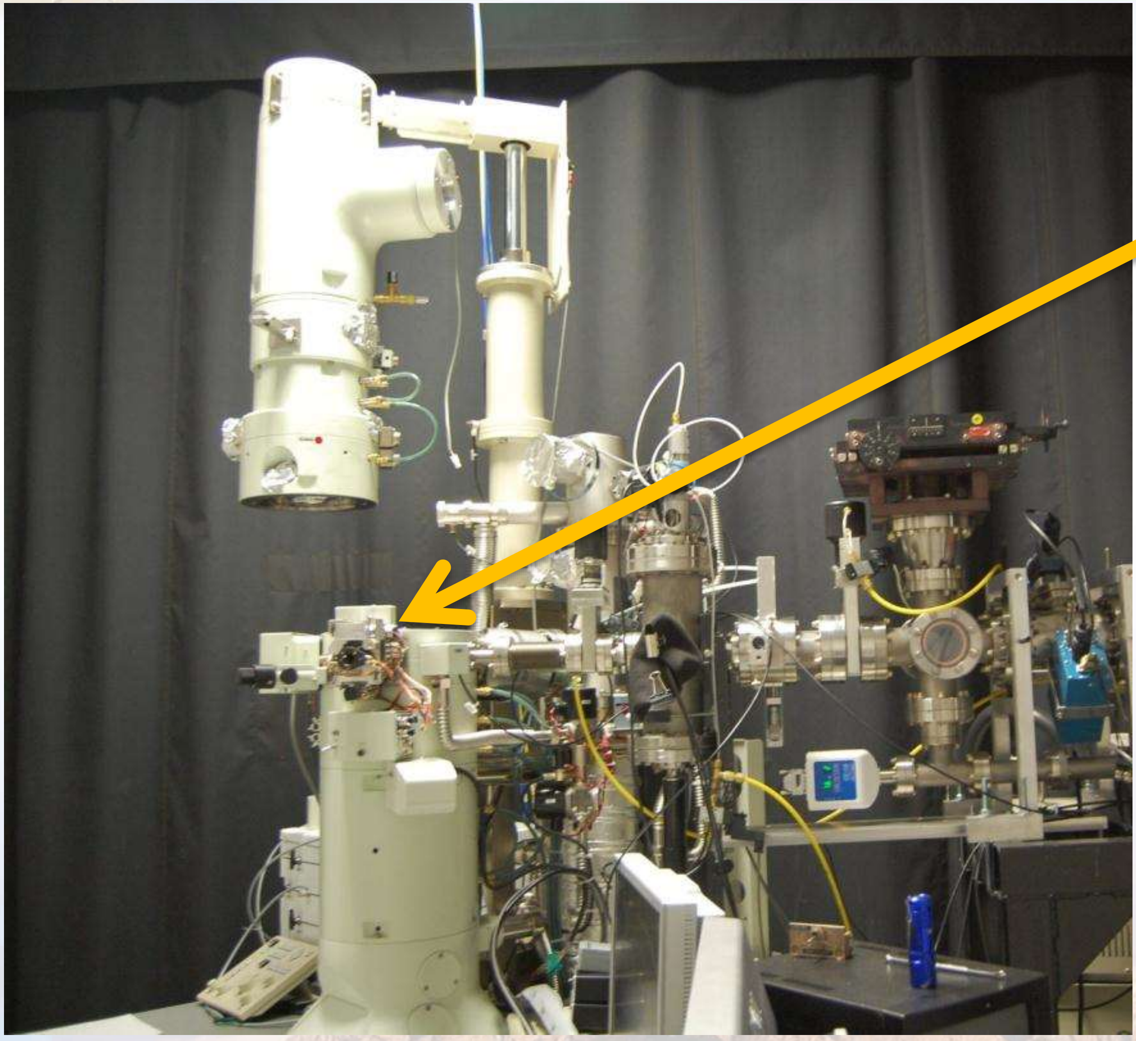
Hummingbird tomography stage

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

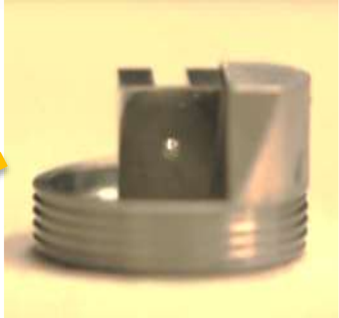


Current work: *In situ* TEM Luminescence

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)

Outline

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   - Control of Local Chemistry
     - Development of back gates for graphene devices
   - Investigate Radiation Tolerance
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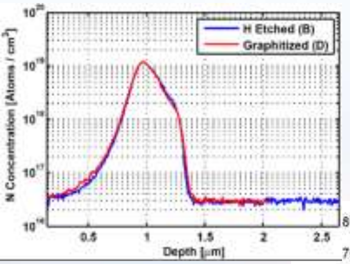
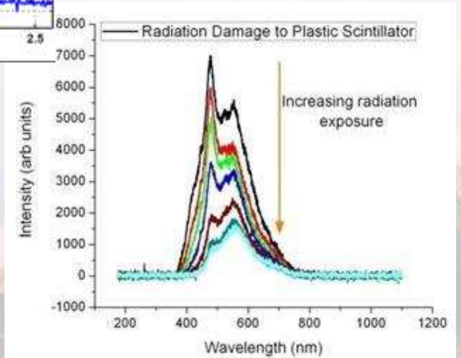
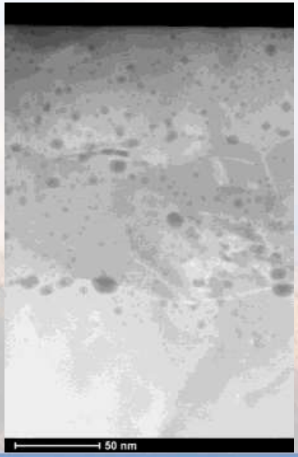
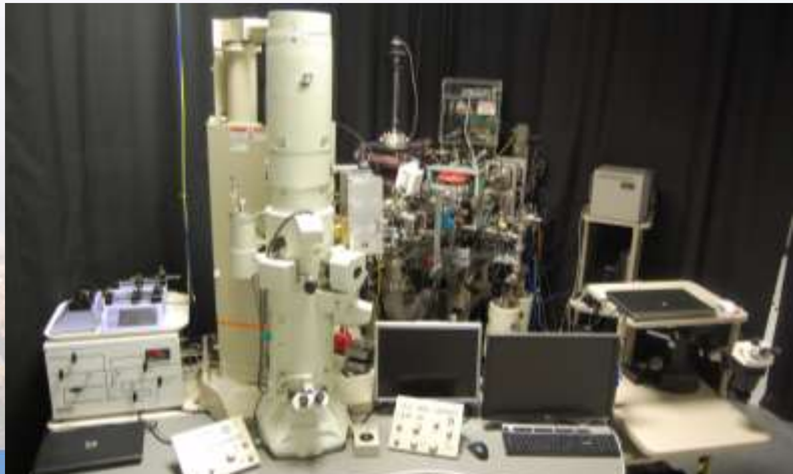
     - Initial work on indentation and cyclic compression
   - Fun future directions for real-time nanoscale observation in extreme environments





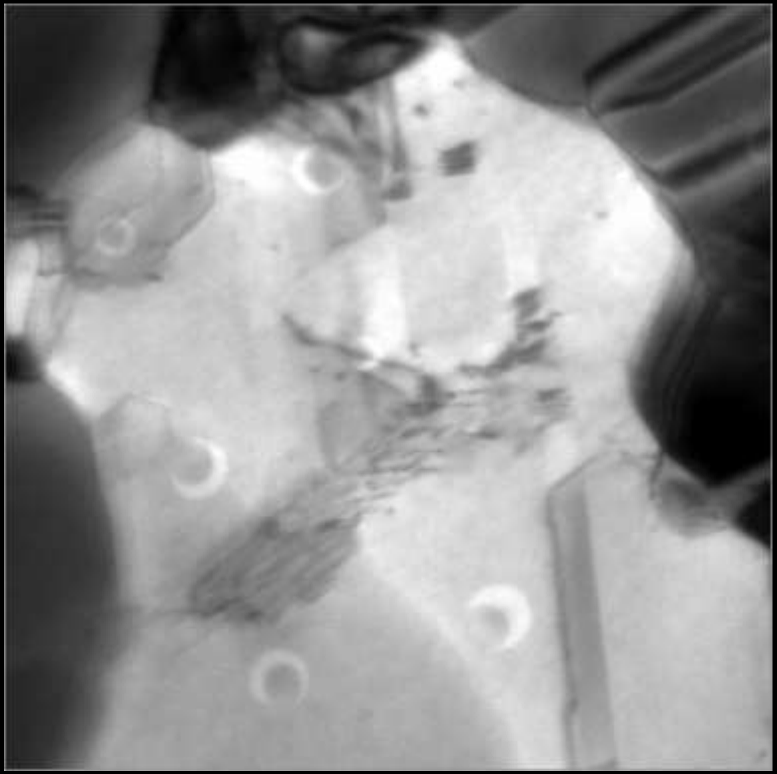




Single Ion Strikes

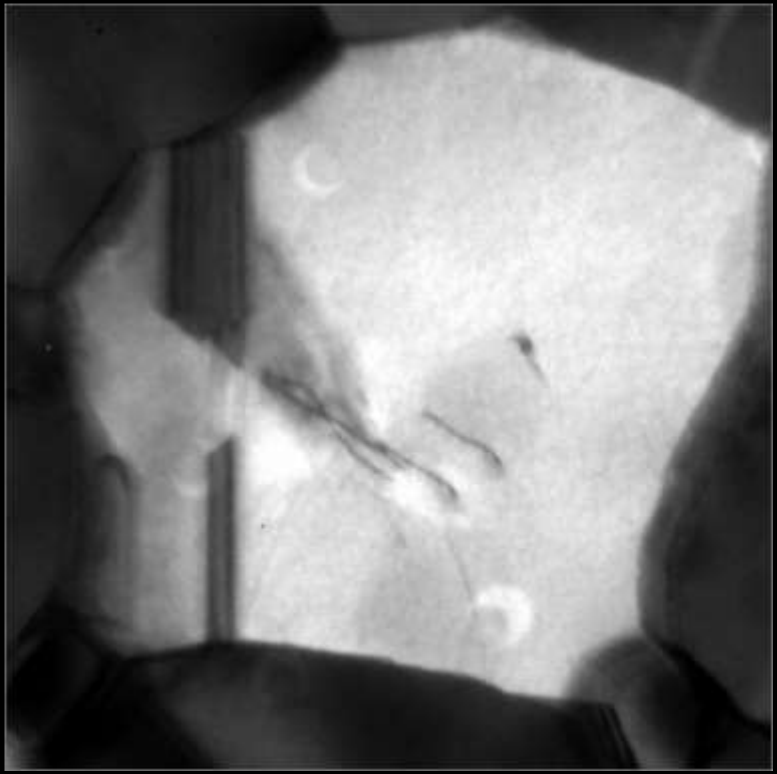
Collaborators: C. Chisholm & A. Minor

7.9×10^9 ions/cm^2/s



VS

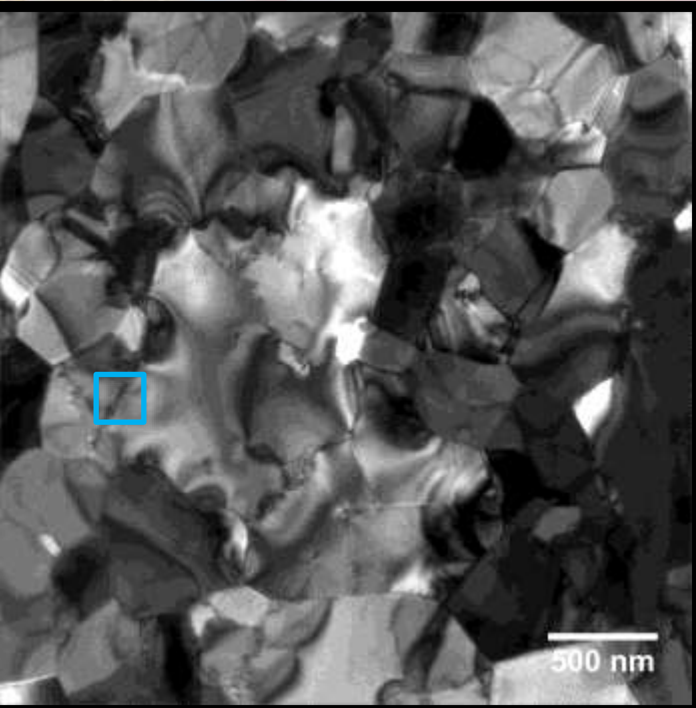
6.7×10^7 ions/cm^2/s



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

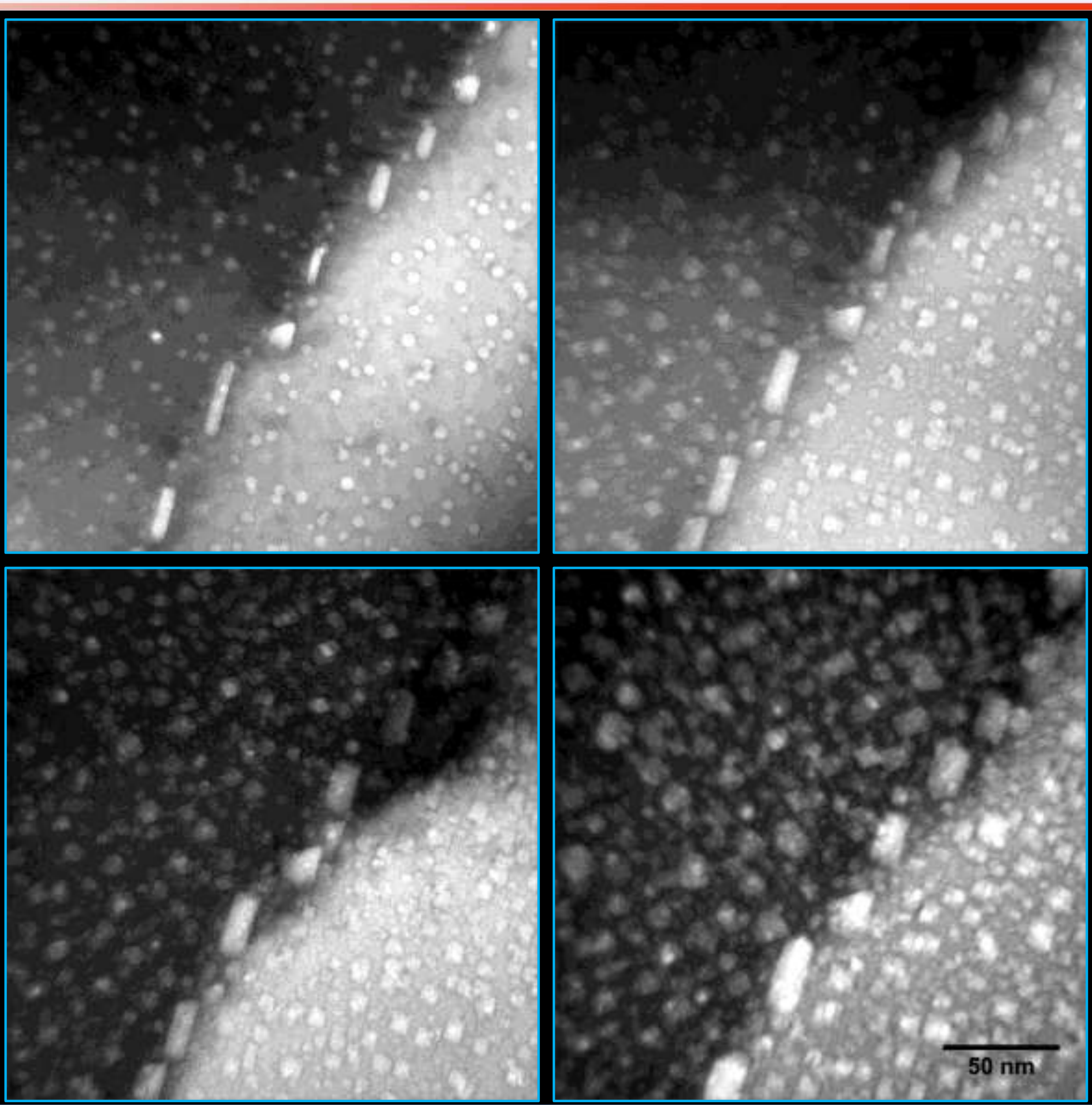
In situ Implantation

Collaborators: C. Chisholm & A. Minor



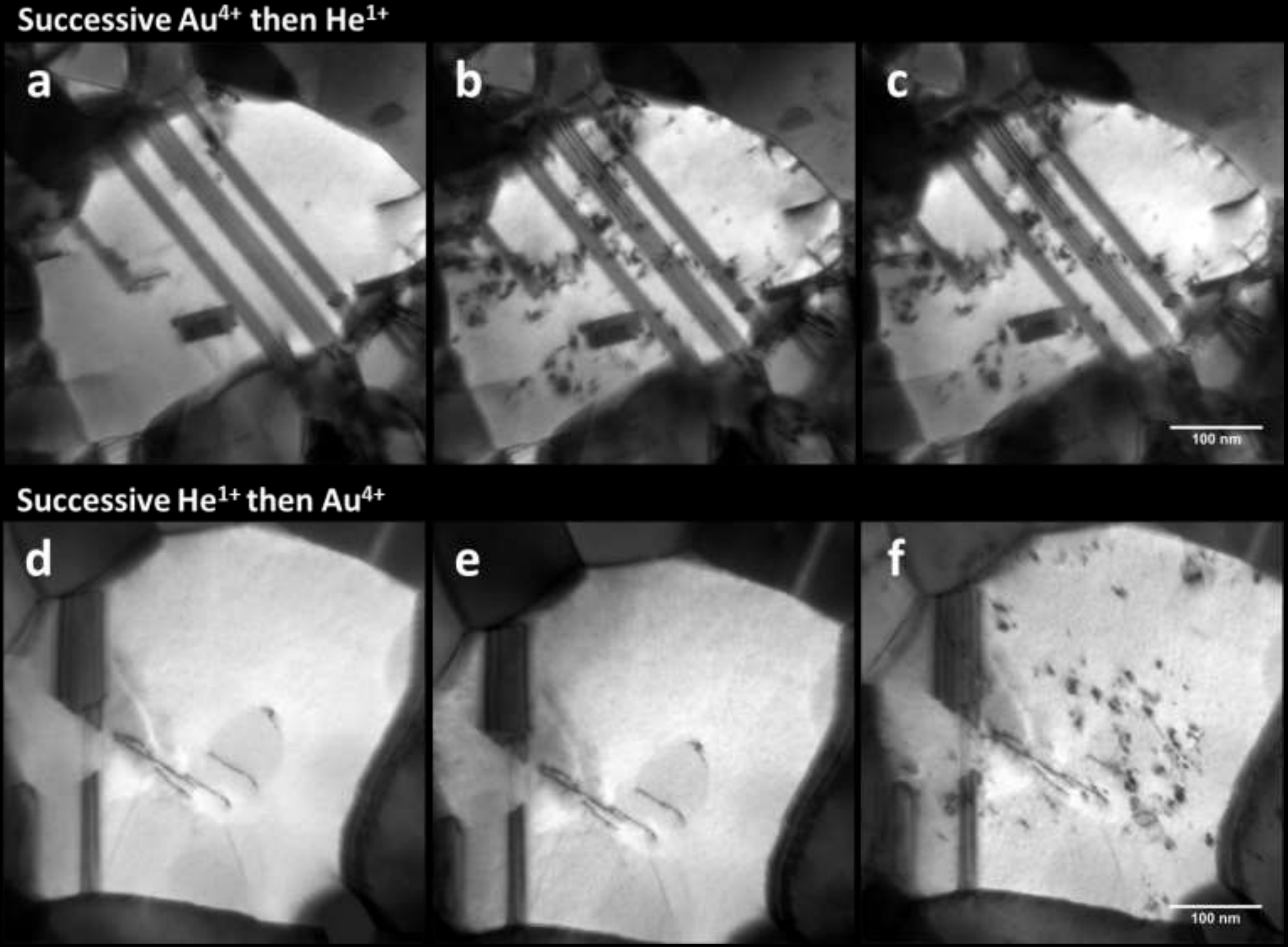
Gold thin-film implanted with 10keV He^{2+}

Result: porous microstructure



in situ Successive Implantation & Irradiation

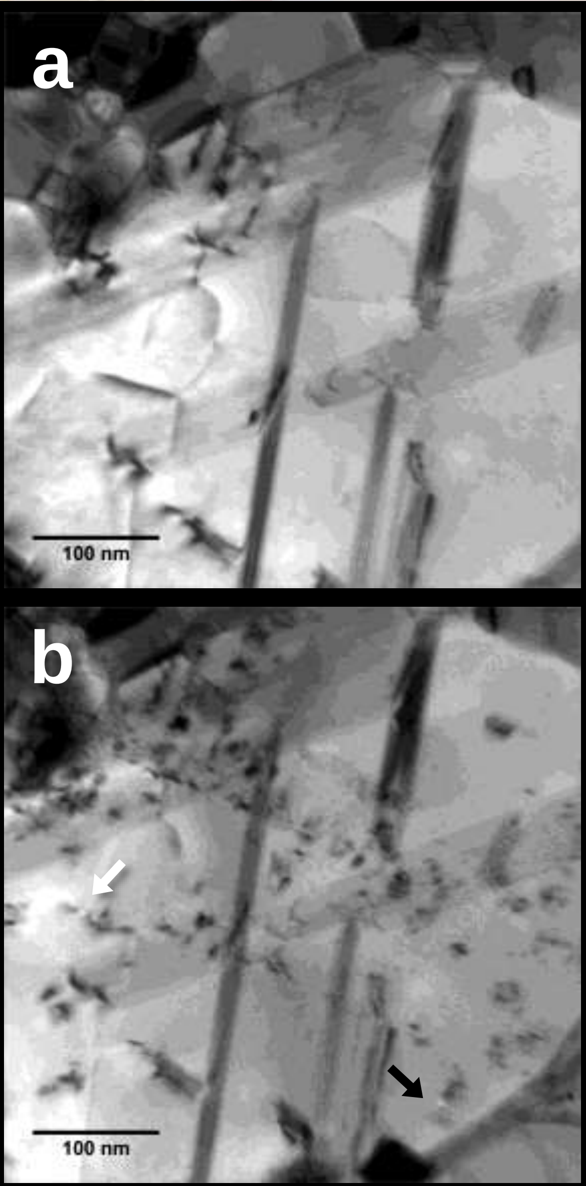
Collaborators: C. Chisholm & A. Minor


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

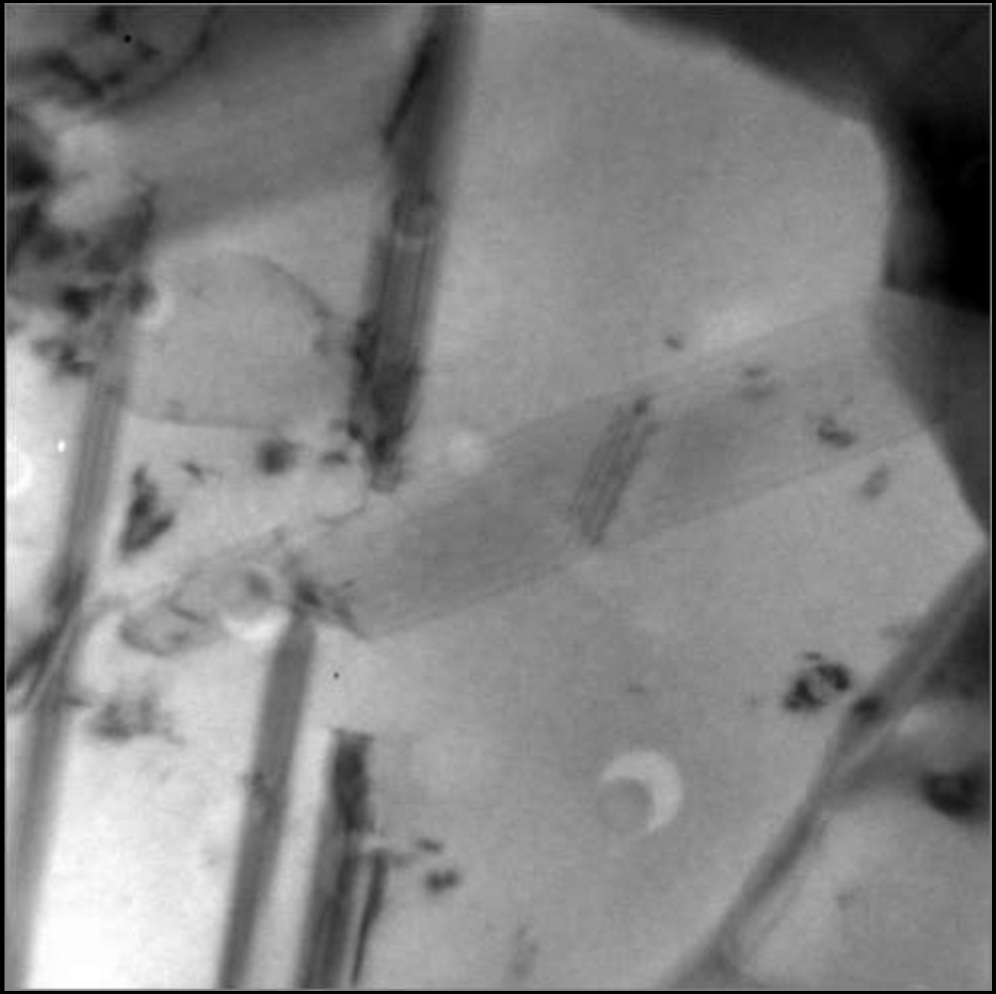
Successive He^{1+} then Au^{4+}

In situ Concurrent Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor



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



Single Ion Strikes During Concurrent Irradiation: Nucleation of Helium Cavities

Collaborators: C. Chisholm & A. Minor

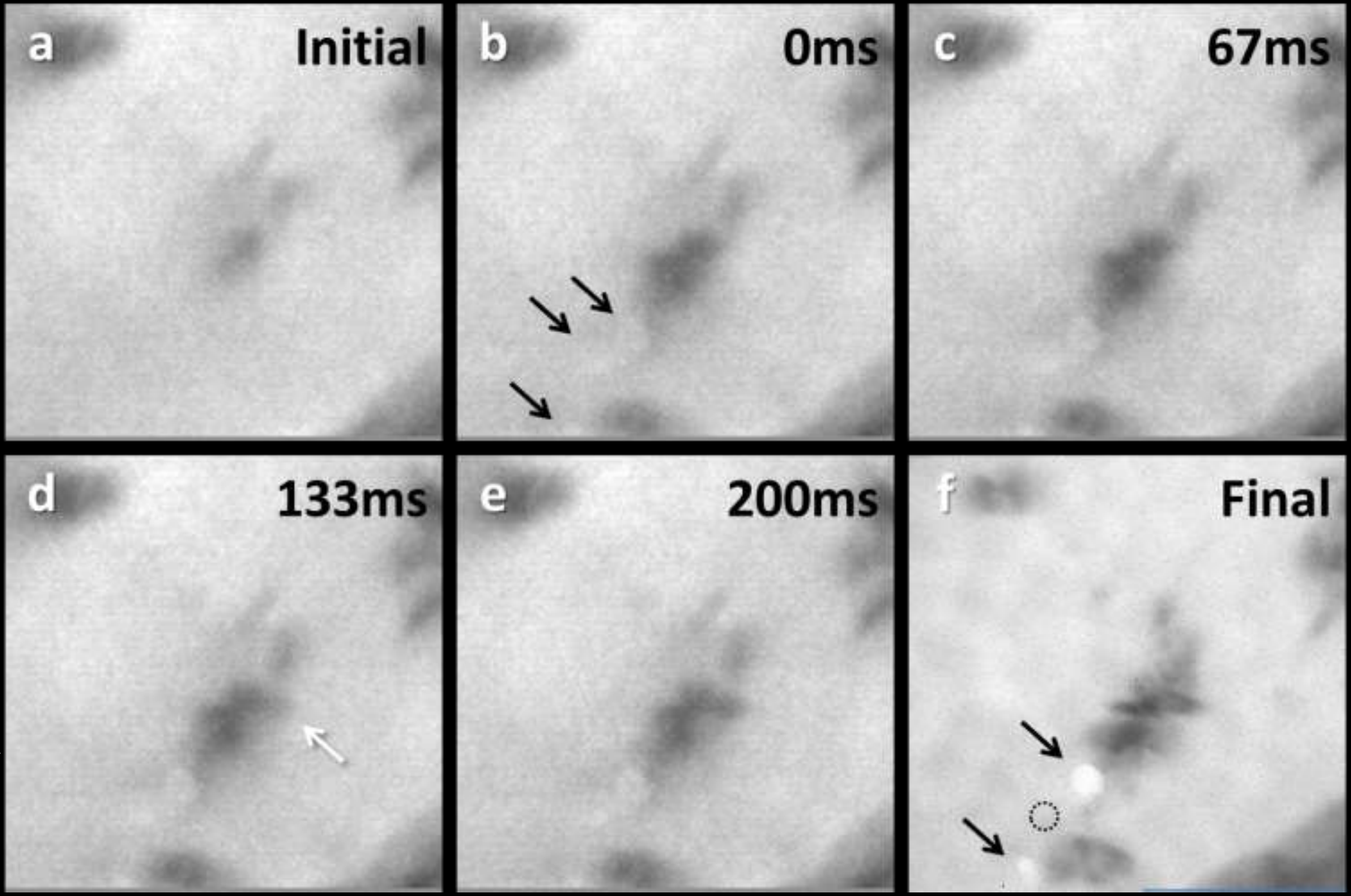
a) Initial microstructure

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

d) Cascade damage still evolving

e) Apparent stability

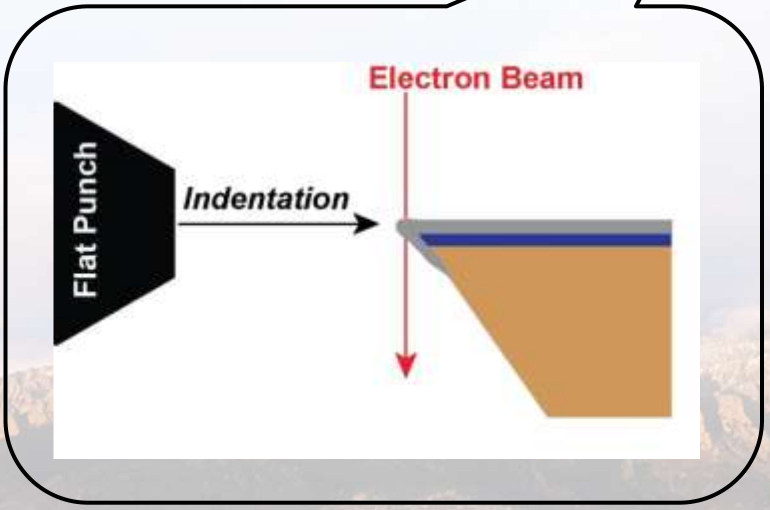
f) Final microstructure: Only two remaining cavities



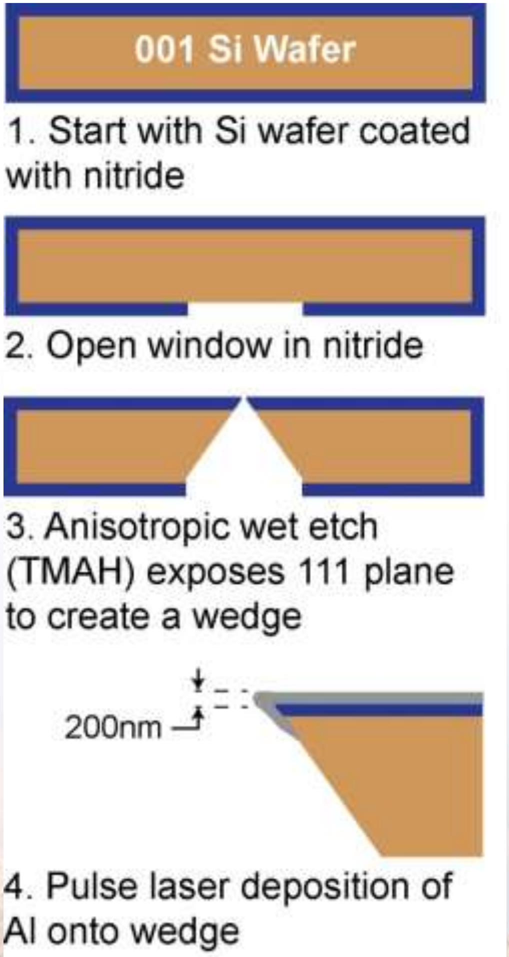
Specimen Synthesis & Testing

In situ TEM Fatigue

- **Microscope: JEOL JEM 2100 $\mathrm{LaB_6}$ TEM**
- **Holder: Hysitron PI-95 PicoIndenter with a 1μm flat tip punch**



Sample Fabrication



1. Start with Si wafer coated with nitride
2. Open window in nitride
3. Anisotropic wet etch (TMAH) exposes 111 plane to create a wedge
4. Pulse laser deposition of Al onto wedge

In situ TEM Quantitative Mechanical Testing

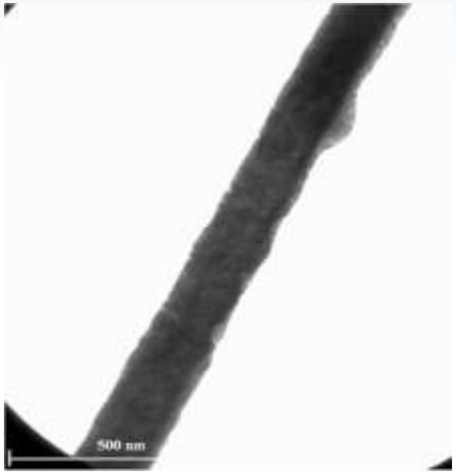
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

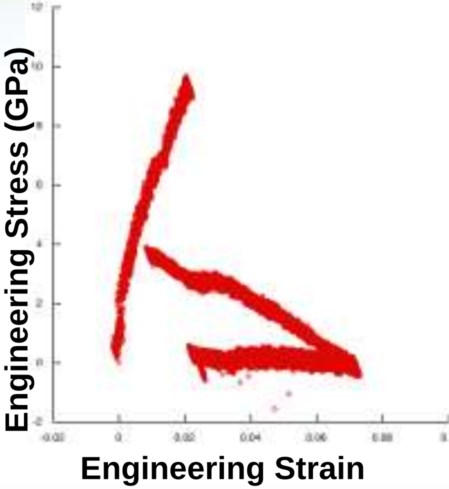
Radiation effect on mechanical properties

- **Direct correlation of dose and defect density with resulting change in strength and ductility**
- **Failure of Mo-wire after 3 MeV Cu irradiation**

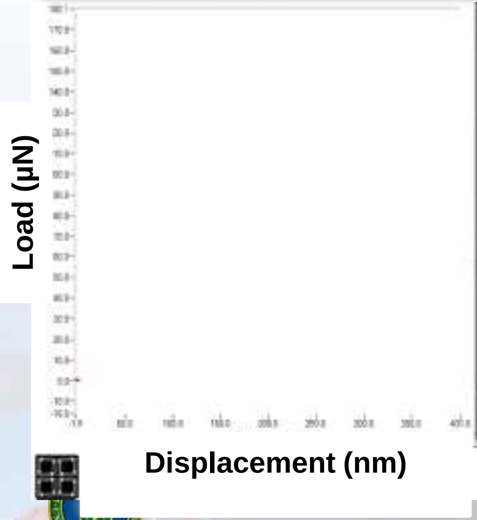
Contact and fatigue effect on structure

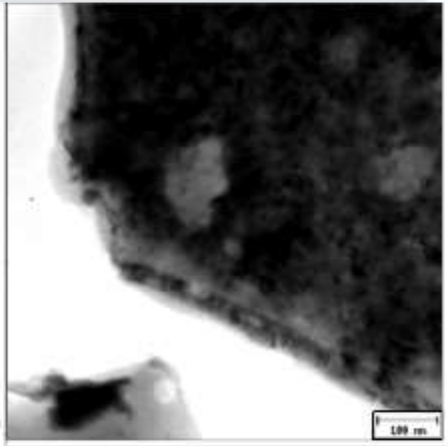
- **Associate change in local hardness and fatigue with corresponding nanostructure**
- **Indent and Fatigue of nanocrystalline Cu film**



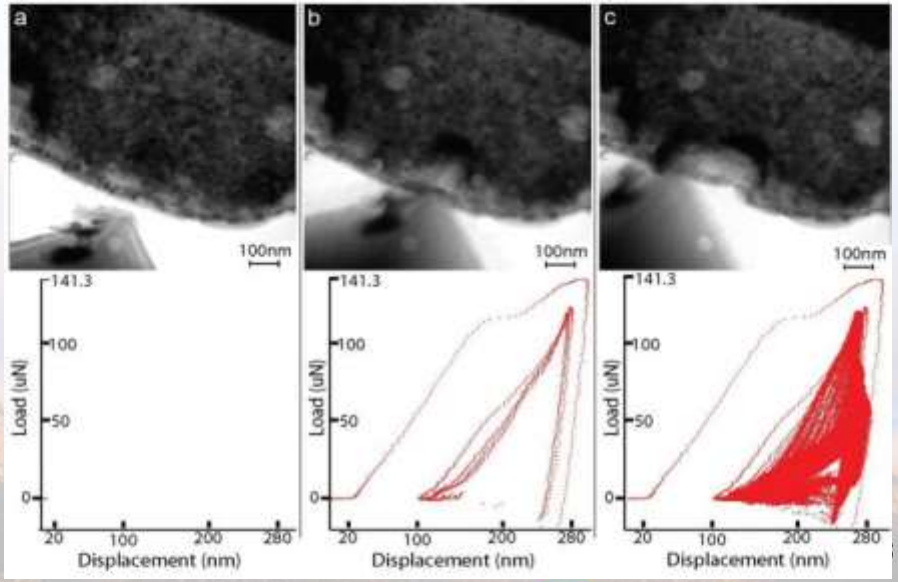


Fundamentals of contact reliability



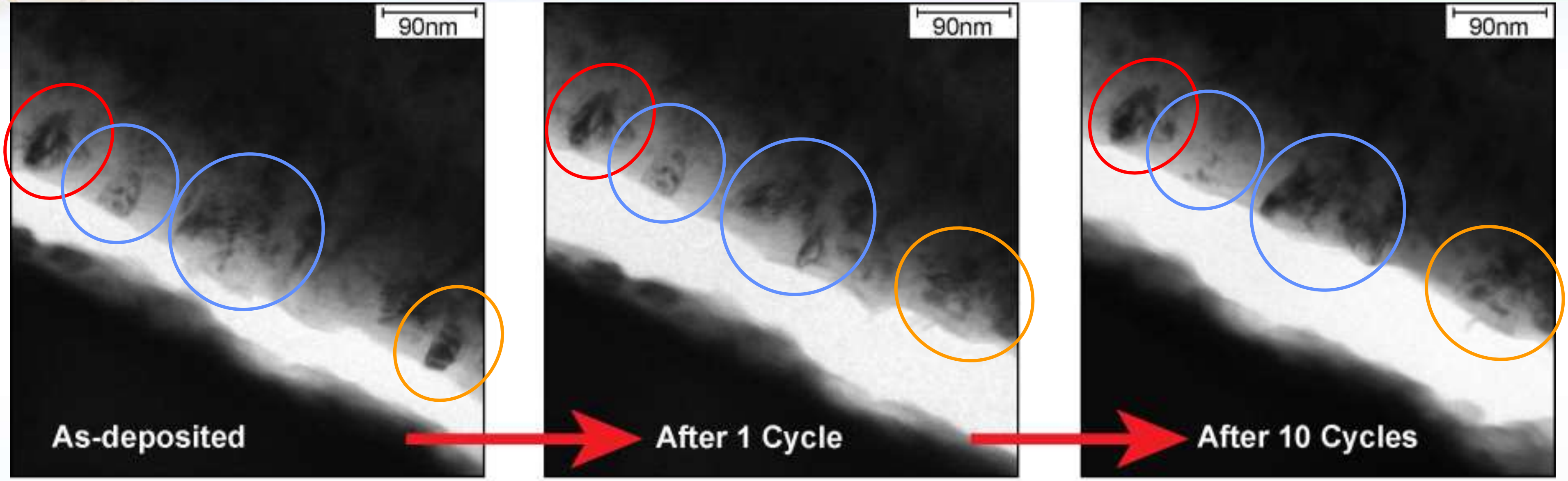


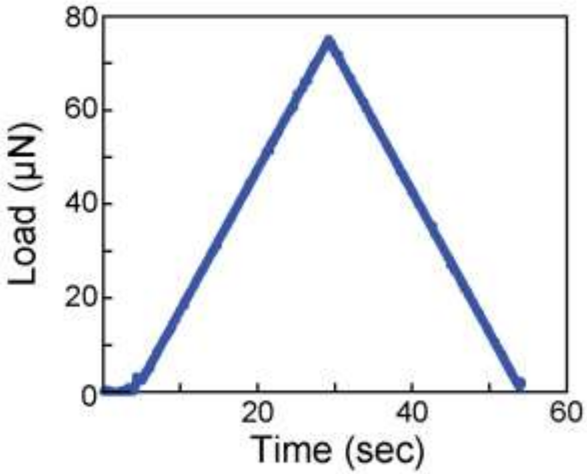
Displacement damage effect on mech. prop.

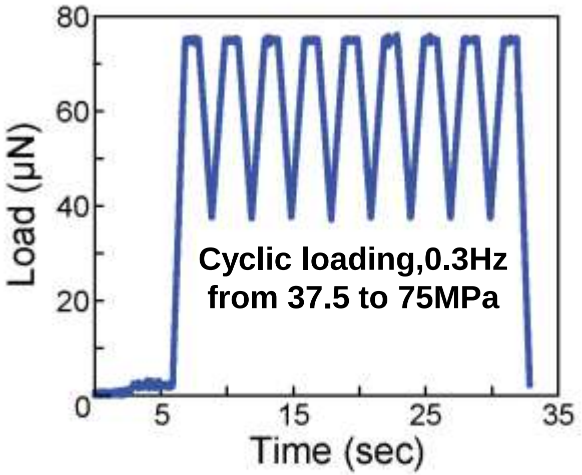


No, Monotonic, or Cyclic Evolution

Contributors: J. Sharon, B. L. Boyce







Cyclic loading,0.3Hz
from 37.5 to 75MPa

375 MPa nominal contact stress

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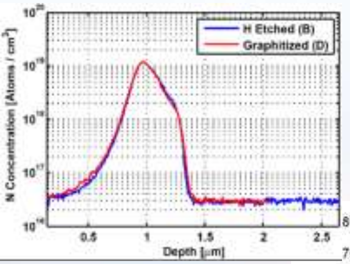
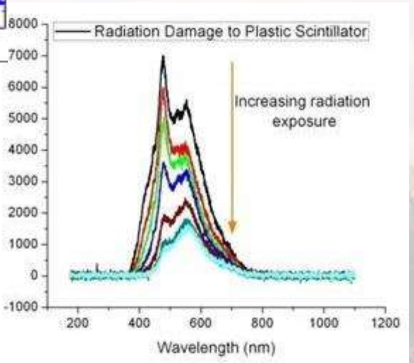
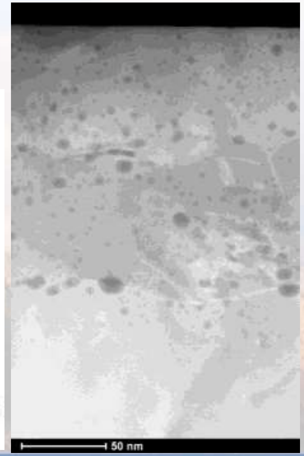
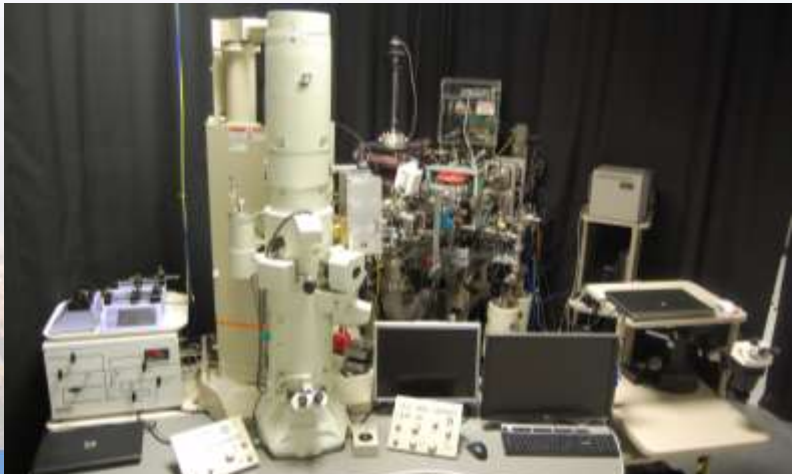


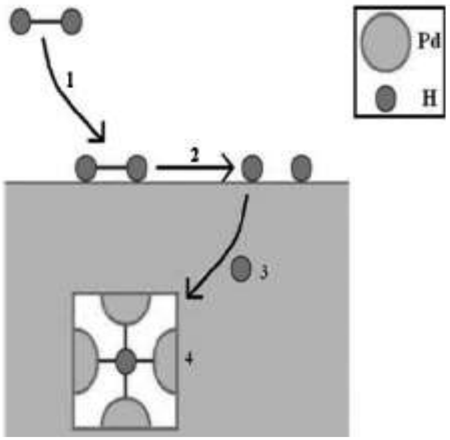




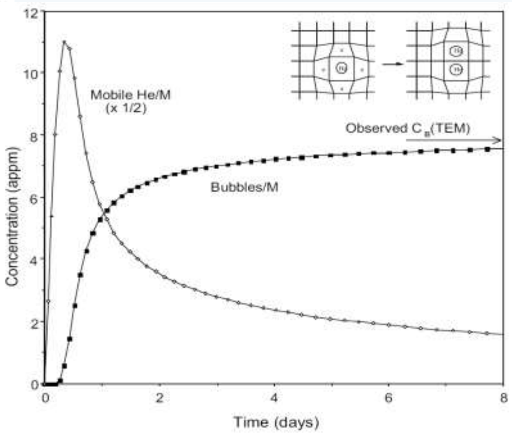


Can *In situ* TEM Address Hydrogen Storage Concerns in Extreme Environments?

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

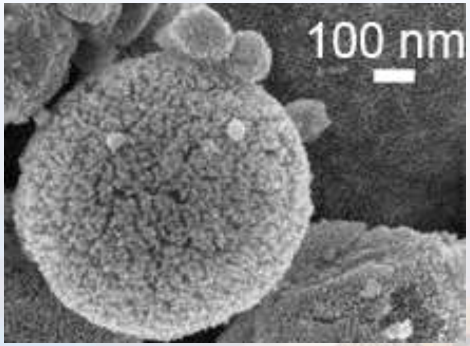


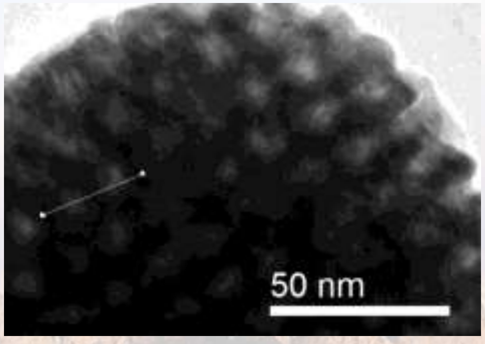
Cowgill, D., Fusion Sci. & Tech., 28 (2005) p. 539

Trinkaus, H. *et al*., JNM (2003) p. 229

Thiebaut, S. *et al*. JNM (2000) p. 217

Harmful effects may be mitigated in nanoporous Pd

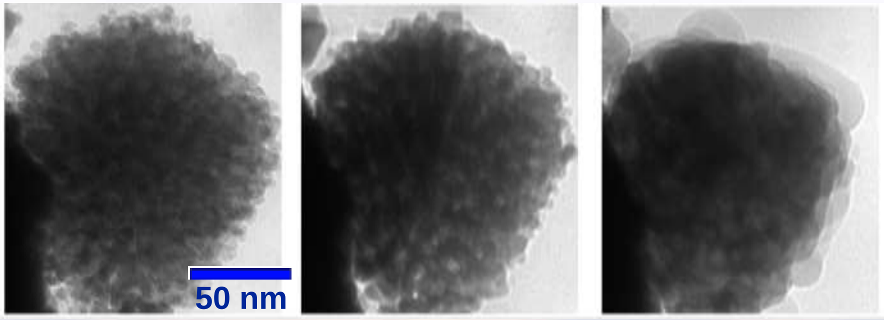




Vapor-Phase Heating TEM Stage

- **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**

- **1 atm H_2 after several pulses to specified temp.**



125° C **200° C** **300° C**

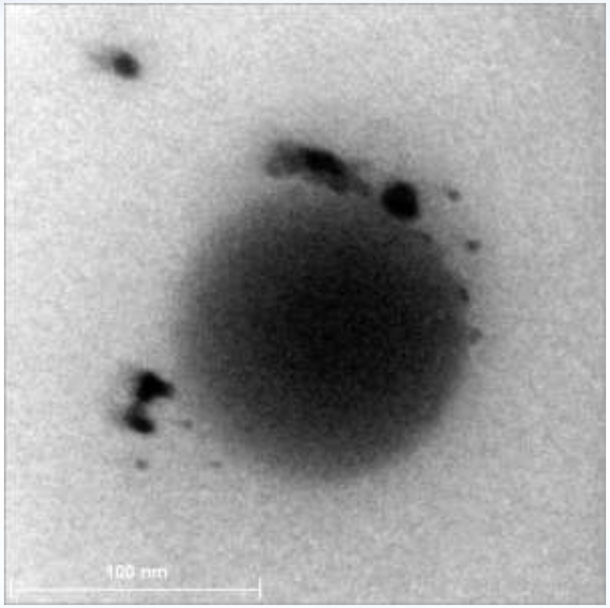
New *in situ* atmospheric heating experiments provide great insight into nanoporous Pd stability

Other Fun Uses of Microfluidic Cell

Protocell Drug Delivery

S. Hoppe,
E. Carnes,
J. Brinker

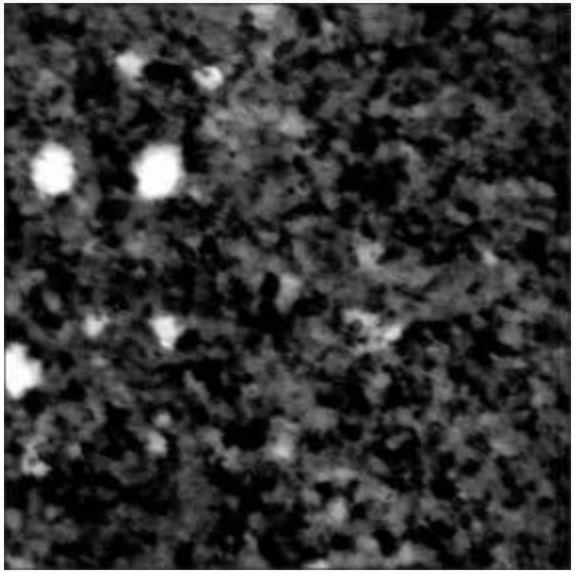
Liposome encapsulated Silica destroyed by the electron beam



Metal Corrosion

D. Gross, J. Kacher,
I.M. Robertson

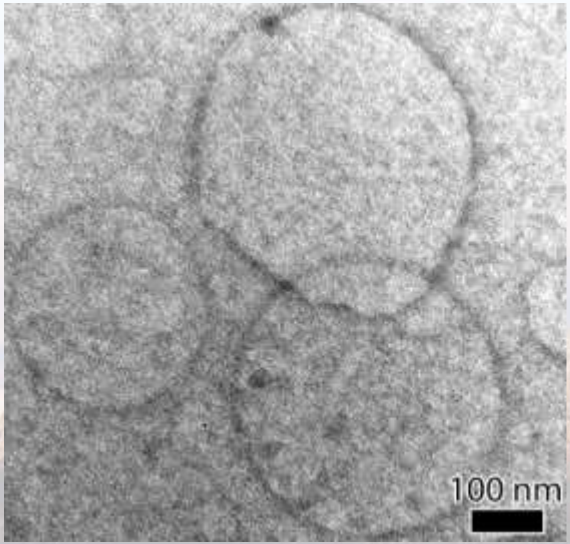
Pitting of nc-Fe due to acetic acid corrosion



Liposomes in Water

S. Hoppe,
D. Sasaki

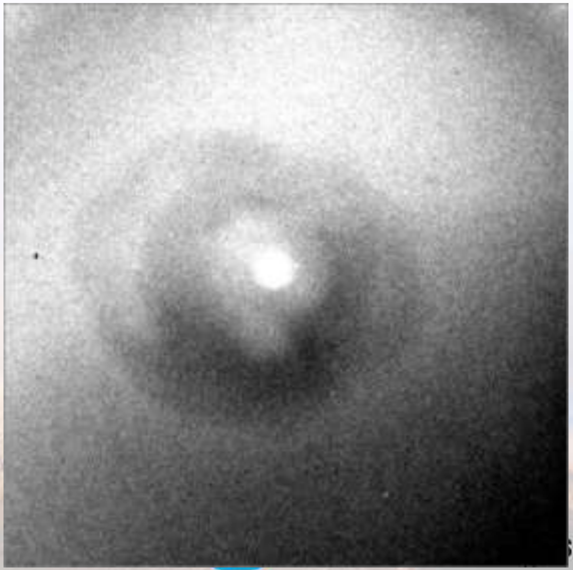
Liposomes imaged in flowing aqueous channel



La Structure Formation

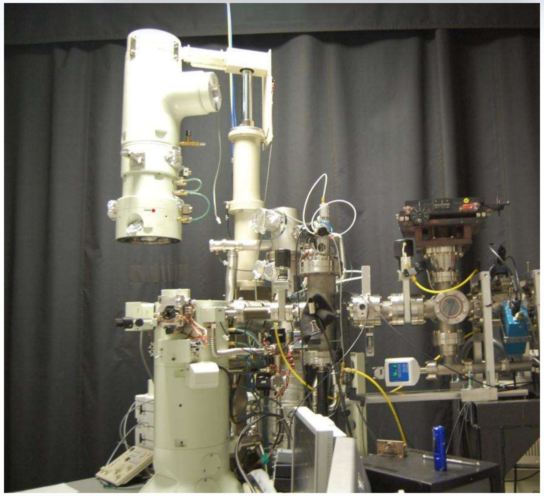
S. Hoppe,
T. Nenoff

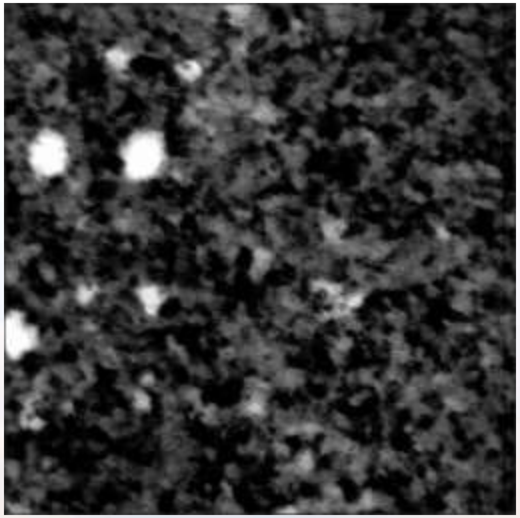
La Nanostructure form from $LaCl_3$ H_2O in wet cell due to beam effects

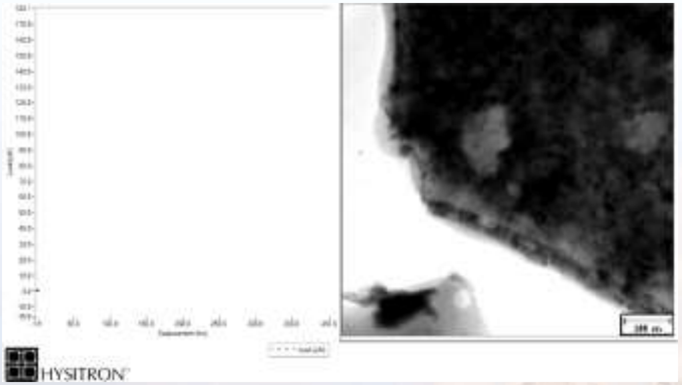


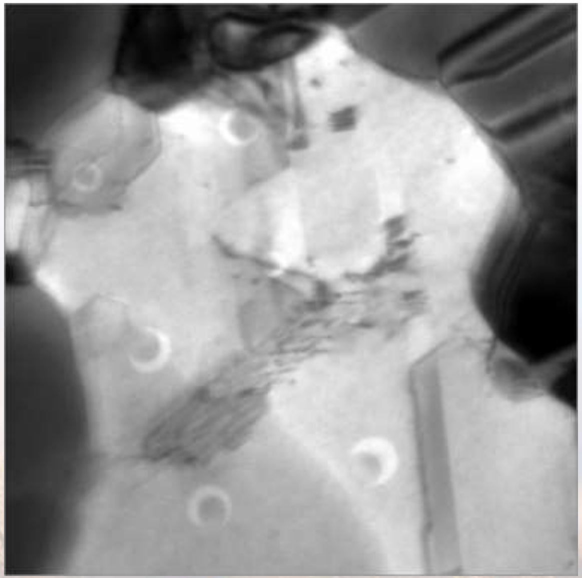
Summary & Future Interests

- 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* capabilities
  - *In situ* high energy ion irradiation from H to Au
  - *In situ* gas implantation
  - Eight 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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