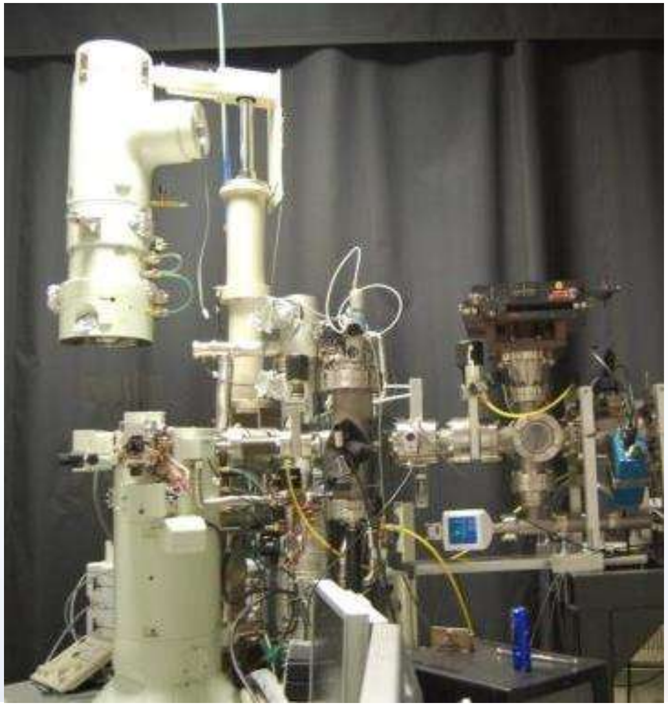
Attempting to Understand Materials in the Extreme from the Nanoscale Up

SAND2015-20761C

K. Hattar

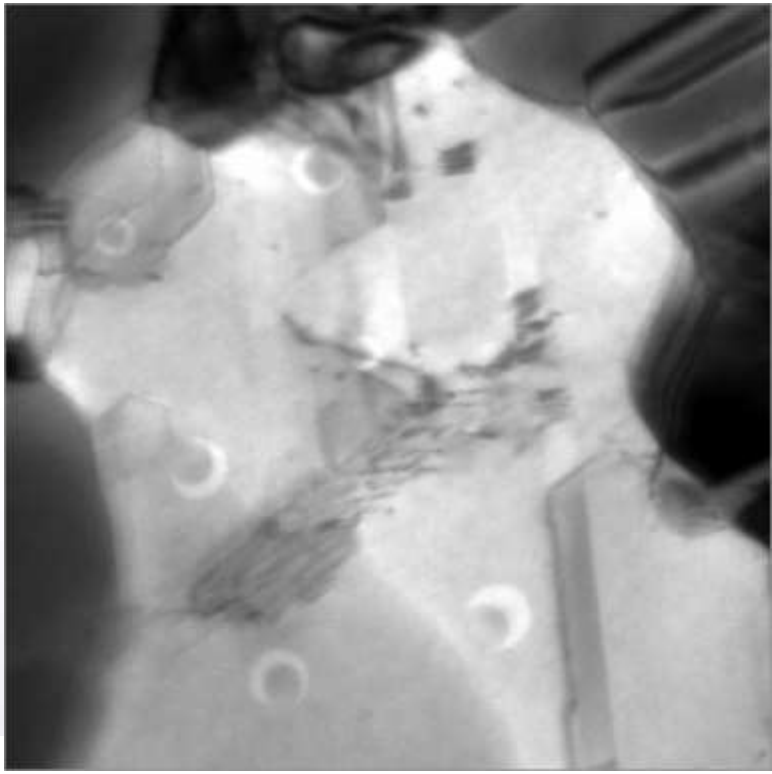
Ion Beam Lab at Sandia National Laboratories

January 15, 2015



***In situ* TEM microscopy**

has recently undergone significant growth providing capabilities to investigate the structural evolution that occurs due to various extreme environments and combinations thereof



Collaborators:

- **IBL: D.C. Bufford, D. Buller, C. Chisholm, B.G. Clark, J. Villone, G. Vizkelethy, B.L. Doyle, S. H. Pratt, & M.T. Marshall**
- **Sandia: B. Boyce, T.J. Boyle, P.J. Cappillino, J.A. Scott, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, E. Carnes, J. Brinker, D. Sasaki, J.A. Sharon, T. Nenoff, W.M. Mook, P. Feng, F.P. Doty, B.A. Hernandez-Sanchez, P. Yang, J-E Mogonye, S.V. Prasad , P. Kotula, S. Howell, T. Ohta, & T. Beechem**
- **External: A. Minor, L.R. Parent, I. Arslan, H. Bei, E.P. George, P. Hosemann, D. Gross, J. Kacher, & I.M. Robertson**

This work was supported by the US Department of Energy, Office of Basic Energy Sciences.

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My Research Experience

Sandia National Laboratories

"Exceptional service in the national interest"


THE WHITE HOUSE
WASHINGTON

May 13, 1949

Dear Mr. Wilson:

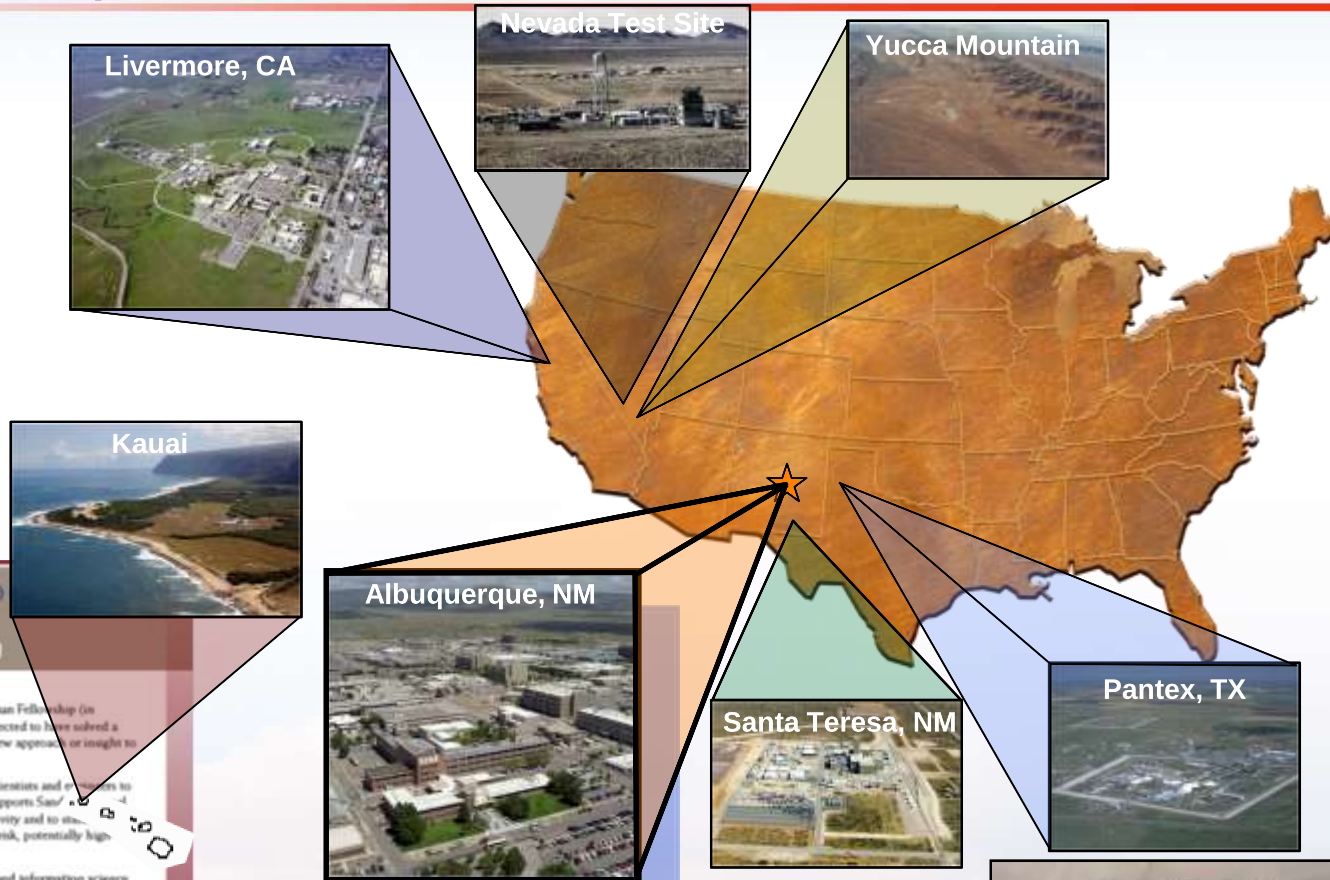
I am informed that the Atomic Energy Commission intends

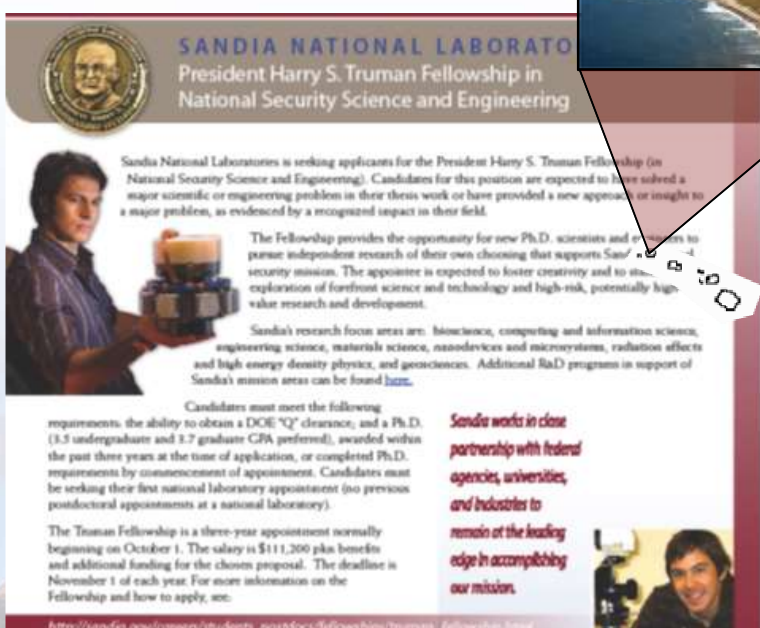
to render an exceptional service in the national interest.

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

Very sincerely yours,

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




SANDIA NATIONAL LABORATO

President Harry S. Truman Fellowship in National Security Science and Engineering

Sandia National Laboratories is seeking applicants for the President Harry S. Truman Fellowship (in National Security Science and Engineering). Candidates for this position are expected to have solved a major scientific or engineering problem in their thesis work or have provided a new approach or insight to a major problem, as evidenced by a recognized impact in their field.

The Fellowship provides the opportunity for new Ph.D. scientists and engineers to pursue independent research of their own choosing that supports Sandia's national security mission. The appointee is expected to foster creativity and to stimulate exploration of forefront science and technology and high-risk, potentially high-value research and development.

Sandia's research focus areas are: bioscience, computing and information science, engineering science, materials science, nanodevices and microsystems, radiation effects and high energy density physics, and geosciences. Additional R&D programs in support of Sandia's mission areas can be found here.

Candidates must meet the following requirements: the ability to obtain a DOE "Q" clearance, and a Ph.D. (3.5 undergraduate and 3.7 graduate GPA preferred), awarded within the past three years at the time of application, or completed Ph.D. requirements by commencement of appointment. Candidates must be seeking their first national laboratory appointment (no previous postdoctoral appointments at a national laboratory).

The Truman Fellowship is a three-year appointment normally beginning on October 1. The salary is $111,200 plus benefits and additional funding for the chosen proposal. The deadline is November 1 of each year. For more information on the Fellowship and how to apply, see:

Sandia works in close partnership with federal agencies, universities, and industries to remain at the leading edge in accomplishing our mission.

http://sandia.gov/careers/students_postdocs/fellowships/truman_fellowship.html

Sandia National Laboratories

Potential Evolution of System Design

Use the Nearest Stone

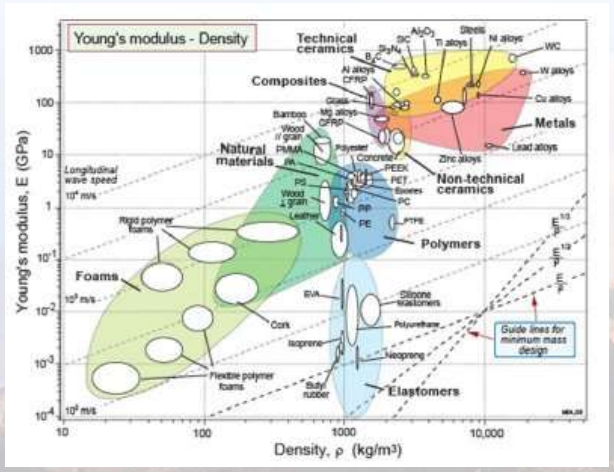
to

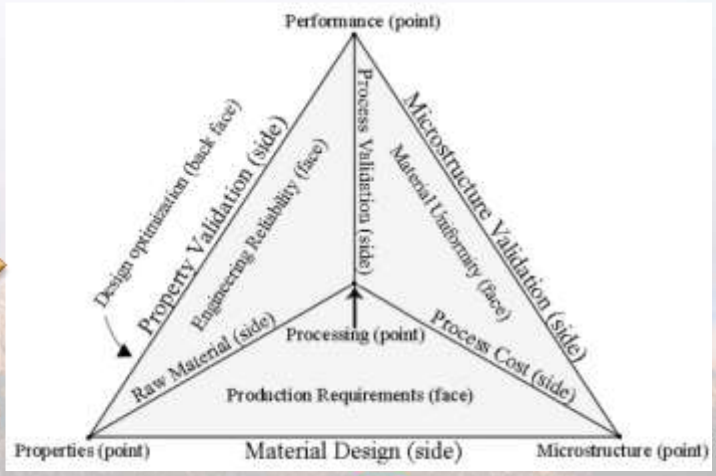
- Radar charts and Ashby plots of current material
- Accelerated and field testing
- **Scientist create a new materials. Engineers find an application.**

Materials by Design

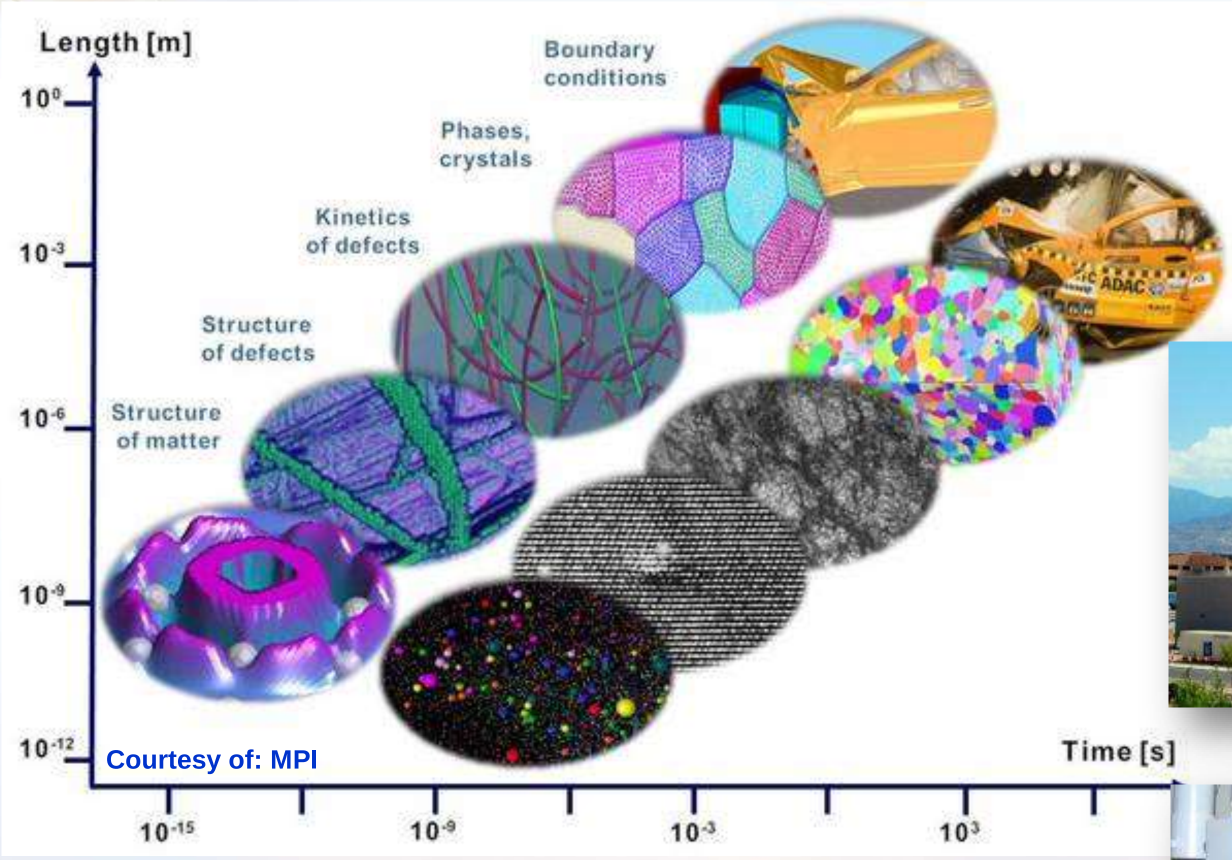
- **Physics-based approach**
- **Requires multiscale modeling**
- **Engineers require given properties, Scientists tailor the chemistry and microstructure to achieve it.**

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



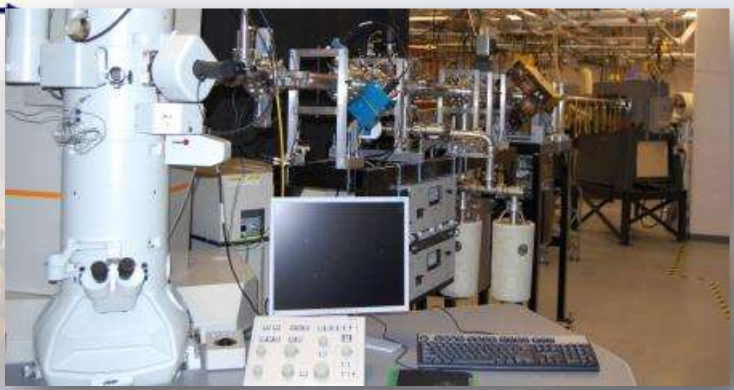


Investigating the nm Scale to Understand the km Scale to Understand Materials Response in the Extremes



Ion Beam Lab (IBL)

In situ Ion Irradiation TEM (I^3TEM)



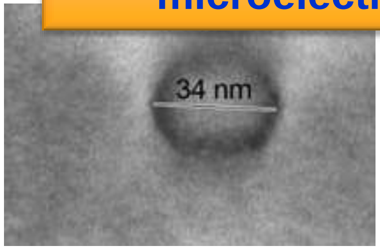
To develop predictive physics-based models, a fundamental understanding of the structure of mater, defects, an the kinetics of structural evolution in the environments of interest are needed

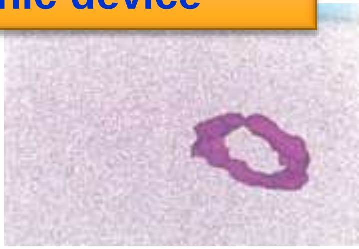
Where have Ion Beam Modified Materials been Utilized?

Ion Beam Modification (IBM)

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

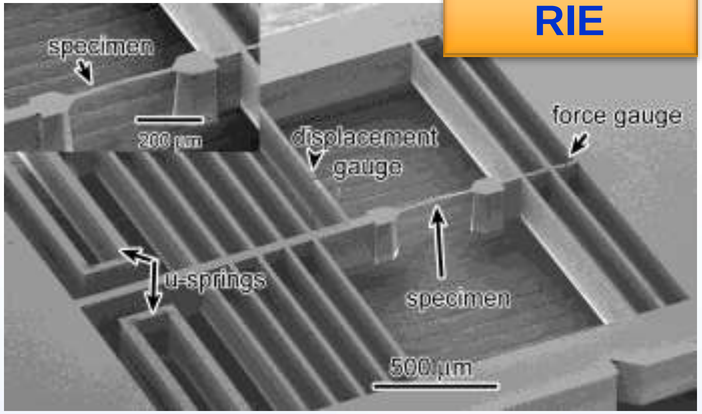
Dopants in Si found in every microelectronic device

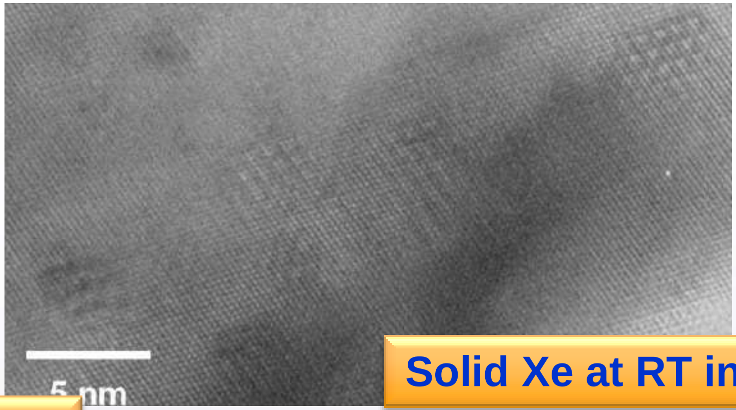




Thompson et al.
Science 2007

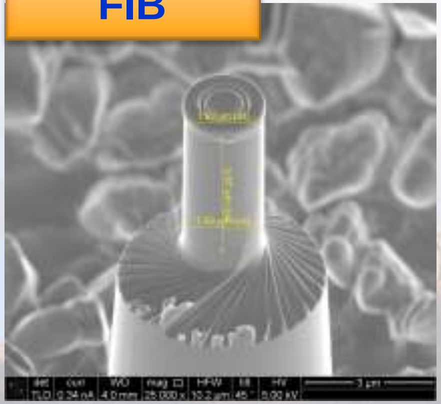
RIE



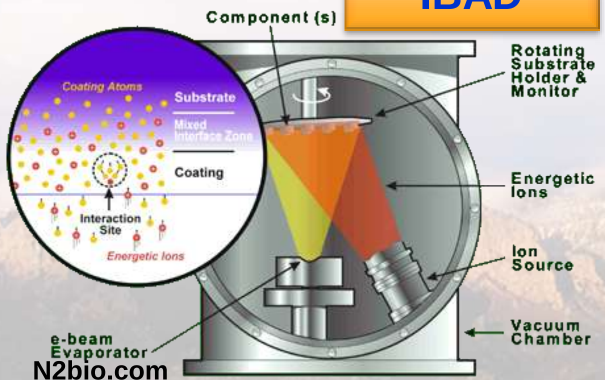


Solid Xe at RT in Al

FIB



IBAD

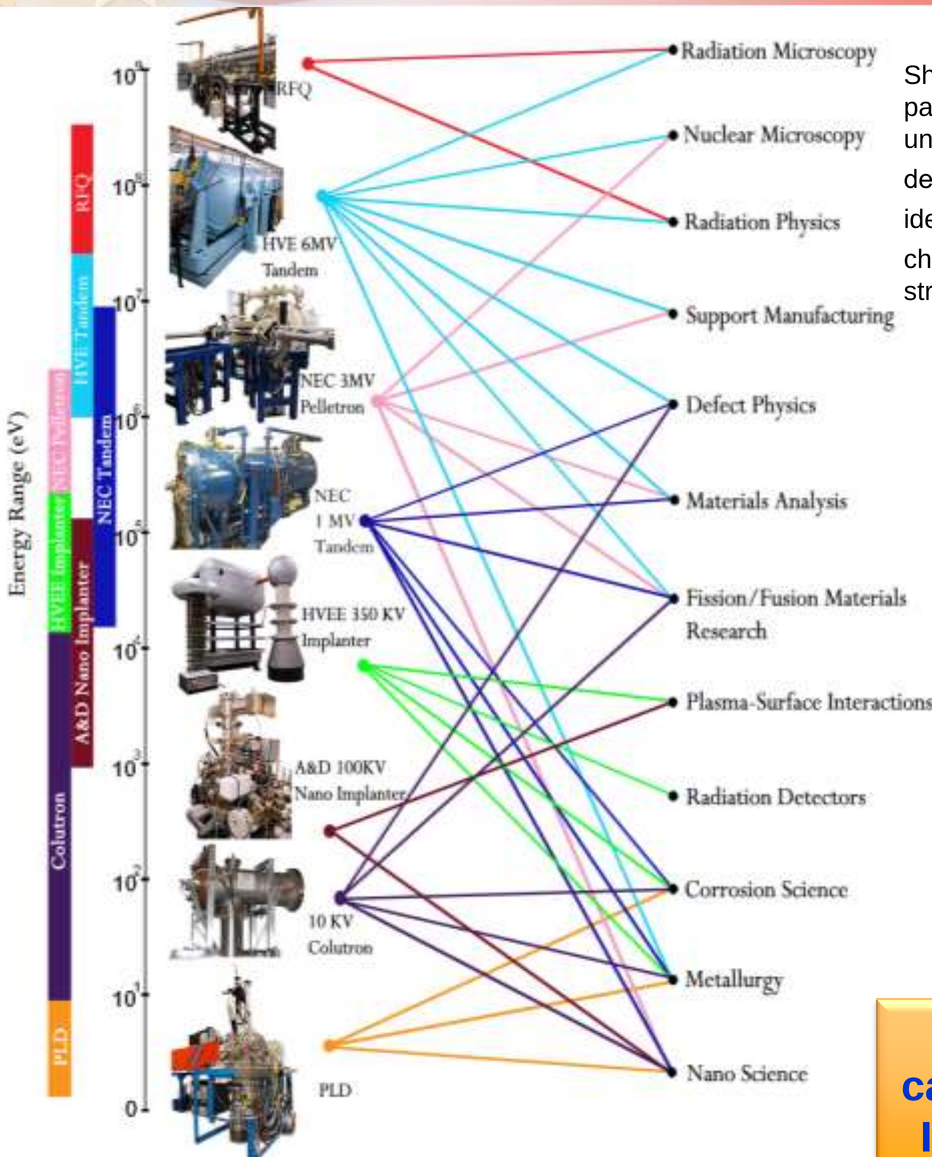


N2bio.com

Proton Cancer Therapy

Ion Beam Applications
iba-worldwide.com

Sandia's Ion Beam Laboratory

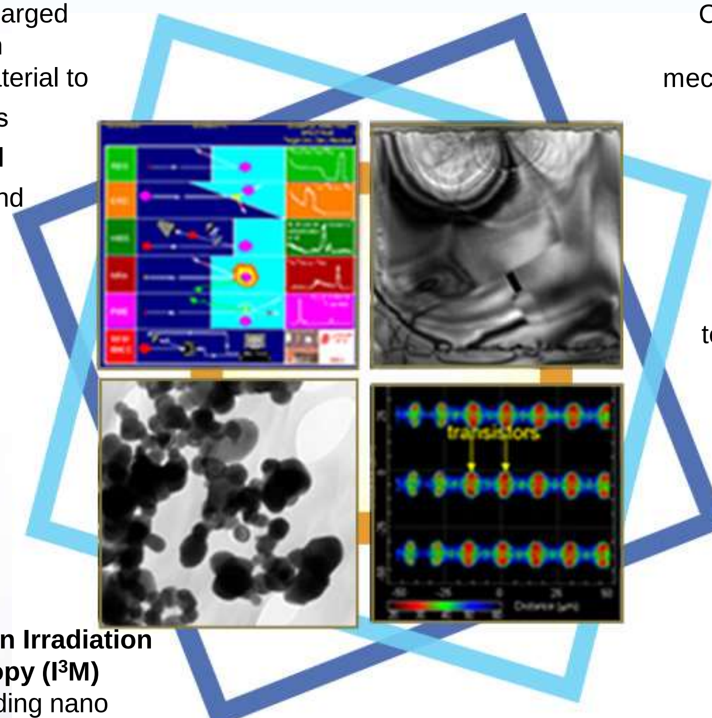


Ion Beam Analysis (IBA)

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

Ion Beam Modification (IBM)

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



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

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

Radiation Effects Microscopy (REM)

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

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

Benefits & Limitations of *in situ* TEM

Benefits

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

Limitations

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

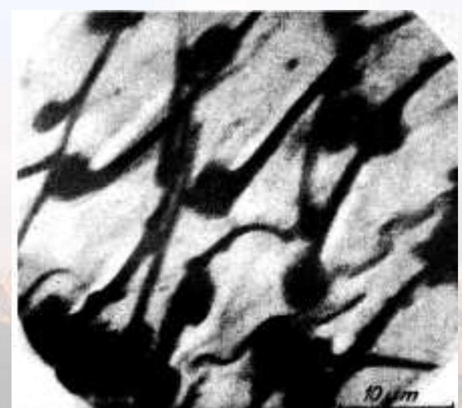


Fig. 6: Wing surface of the house fly
(First internal photography; U = 60 kV, M = 2200)
(Driest, E., and Müller, H.O.: Z. Wiss. Mikroskopie 52, 53-57 (1935))

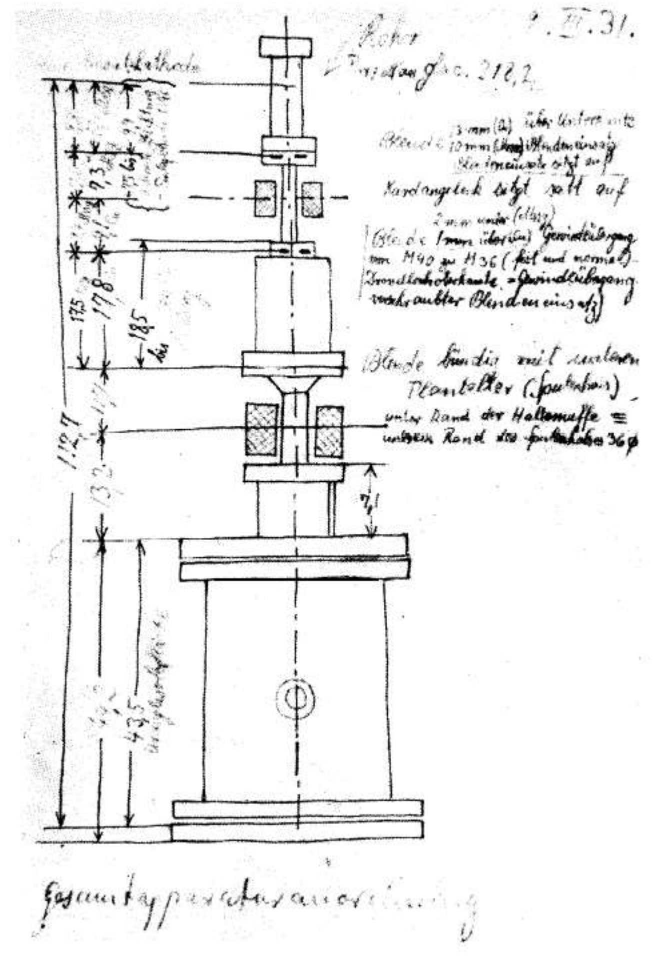


Fig. 2: Sketch by the author (9 March 1931) of the cathode ray tube for testing one-stage and two-stage electron-optical imaging by means of two magnetic electron lenses (electron microscope) [8].

History of *In situ* Ion Irradiation TEM

Courtesy of: J. Hinks

1930s… | 1960s | 1970s | 1980s | 1990s | 2000s | 2010s

1931
The invention of the TEM

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

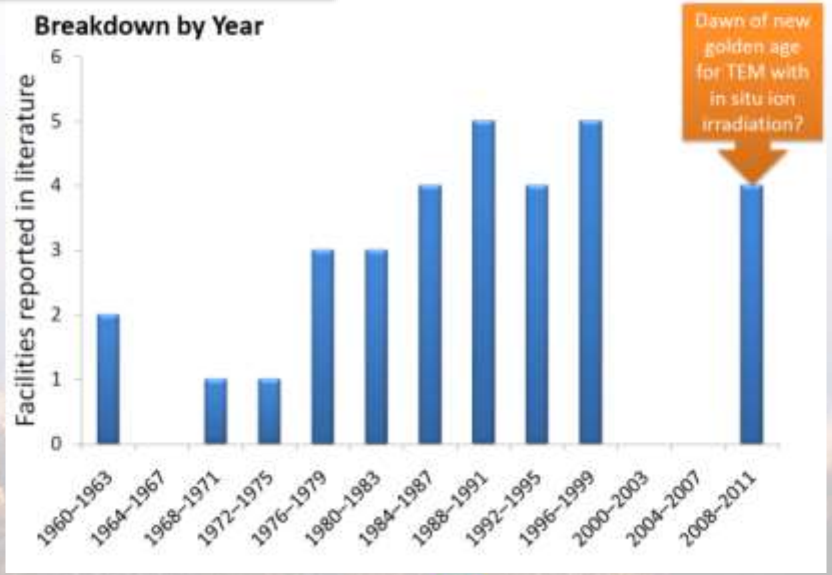
1968
First TEM beamline combination by Thackery, Nelson, and Sansom at AERE Harwell, UK

1976
First HVEM with ion irradiation at UVA, USA

1978
First in-situ ion irradiation experiments at ANL

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

Workshop on Ion Irradiation TEM
Huddersfield, UK (2008)
Albuquerque, USA (2011)
Saporro, Japan (2013)



"The direct observation of ion damage in the electron microscope thus represents a powerful means of studying radiation damage"

D.W. Pashley and A.E.B. Presland Phil Mag. 6(68) 1961 p. 1003

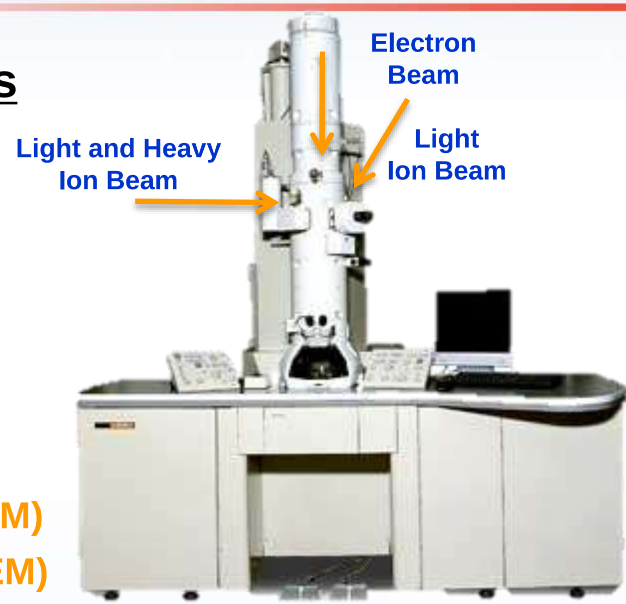
In situ Ion Irradiation TEM Facility

Proposed Capabilities

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

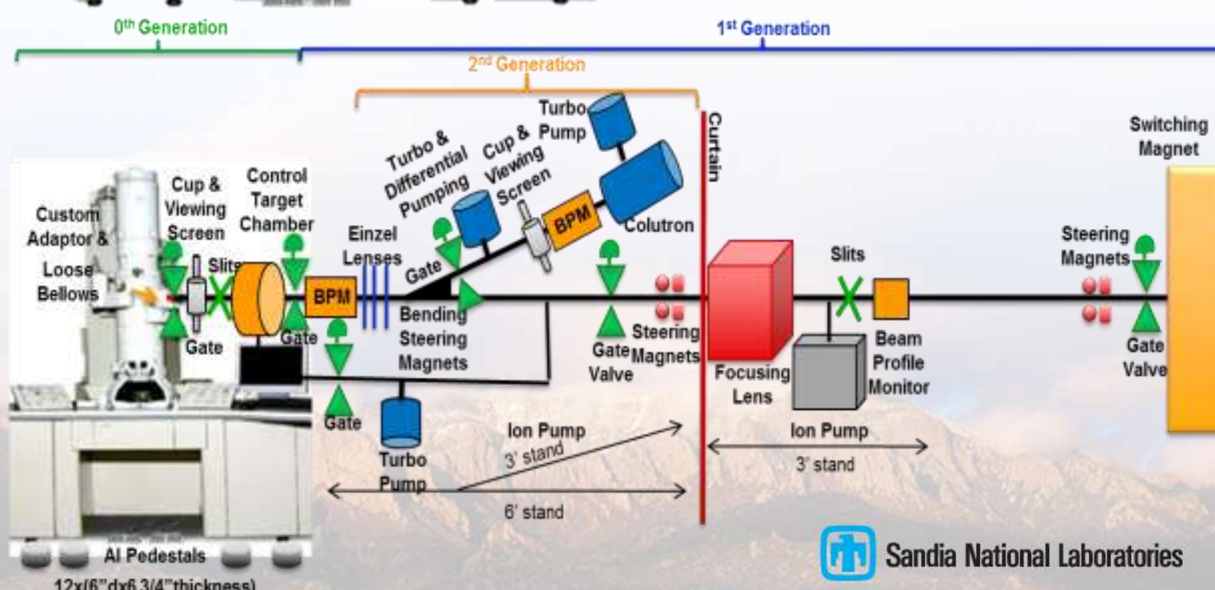
- Nanosecond time resolution (DTEM)
- Procession scanning (EBSD in TEM)
- *In situ* PL, CL, and IBIL

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



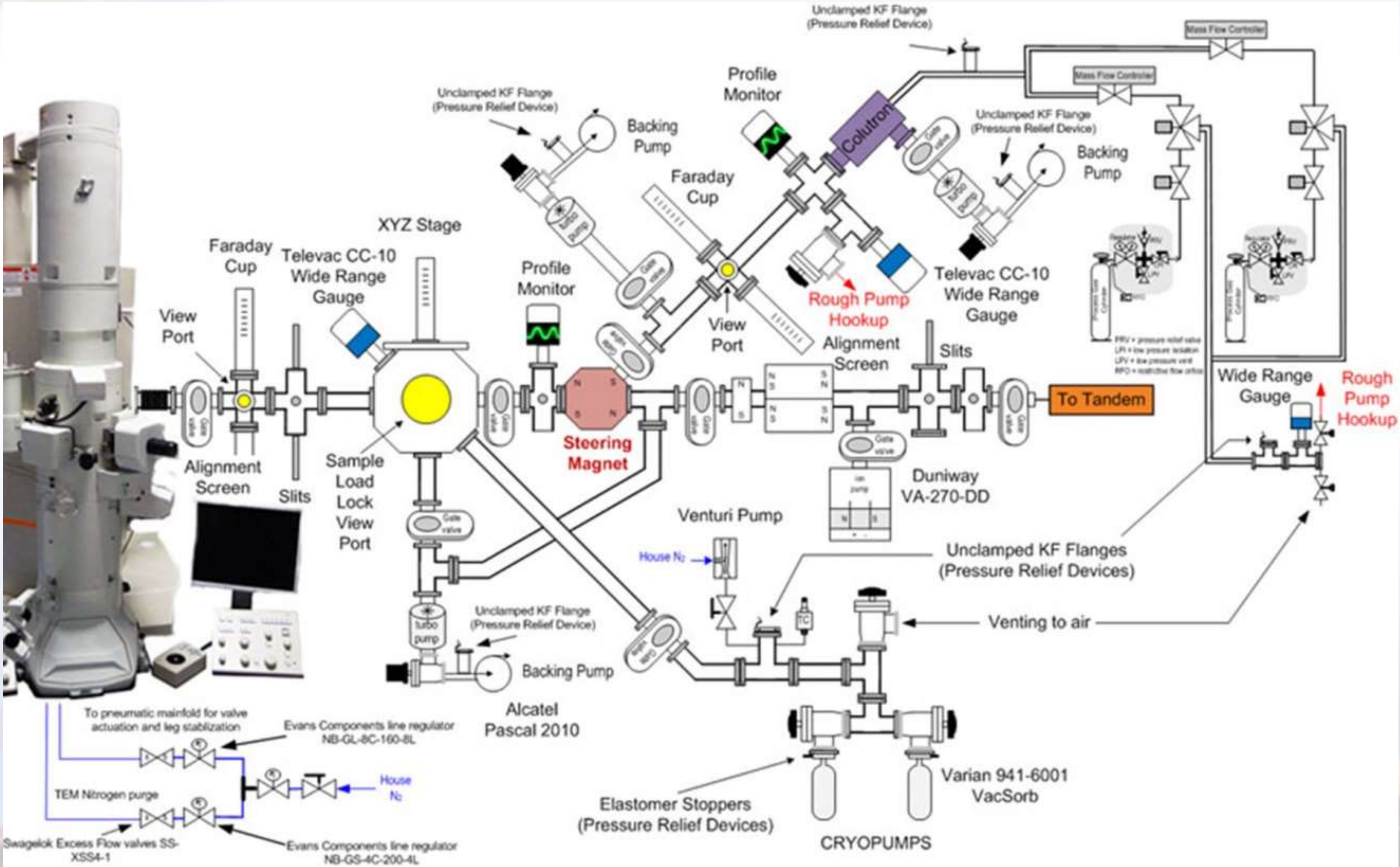
TVIPS

Hummingbird



Schematic of the *In situ* TEM Beamline

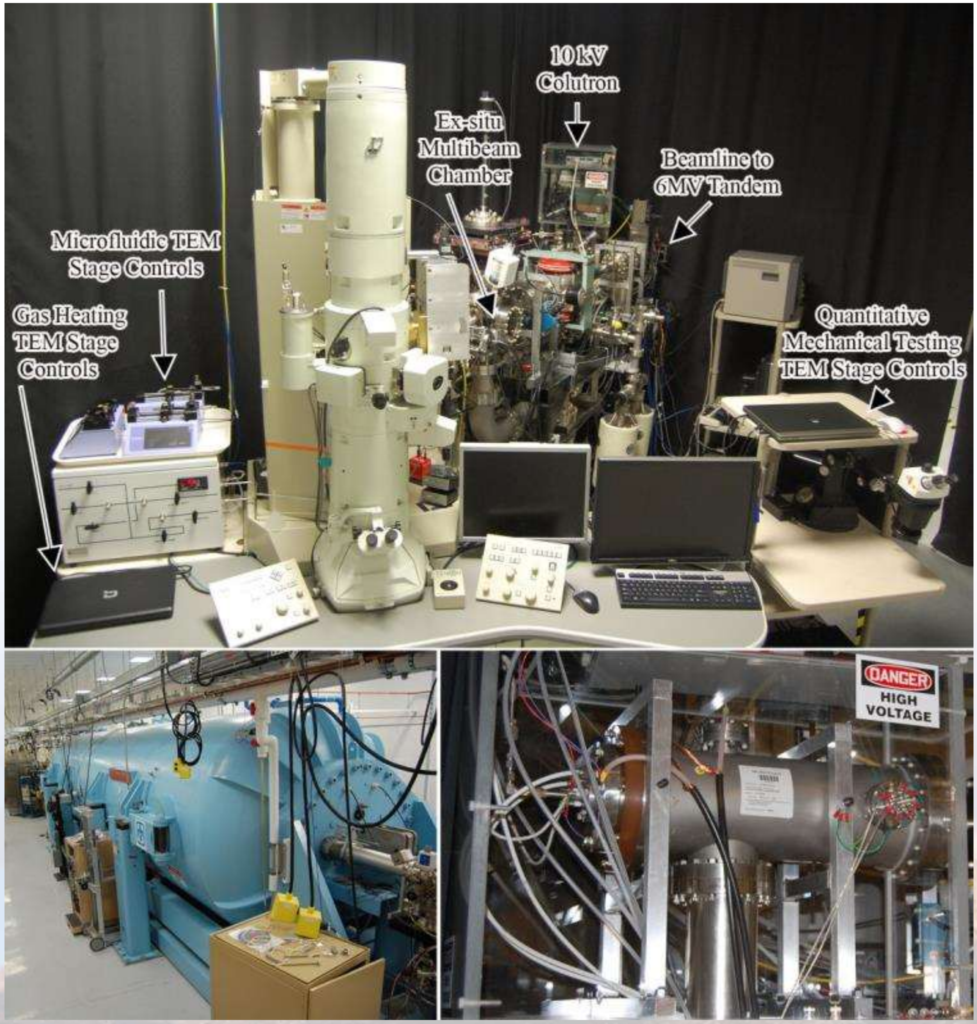
Collaborators: M.T. Marshall J.A. Scott, & D.L. Buller



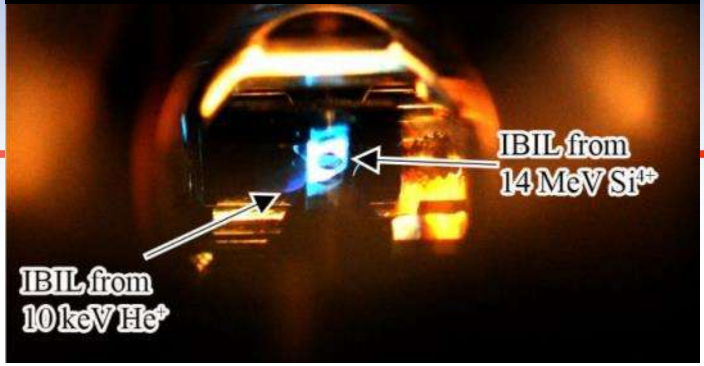
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem

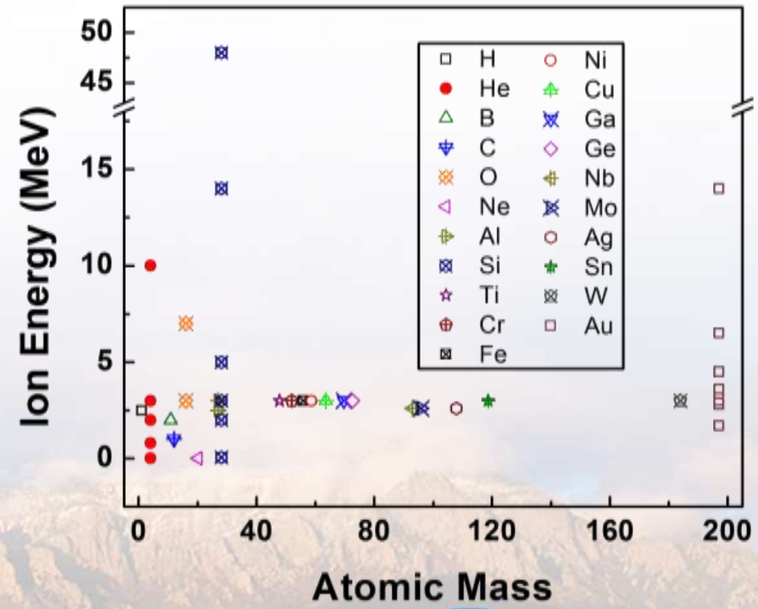


IBIL from a quartz stage inside the TEM



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

Ion species & energy introduced into the TEM



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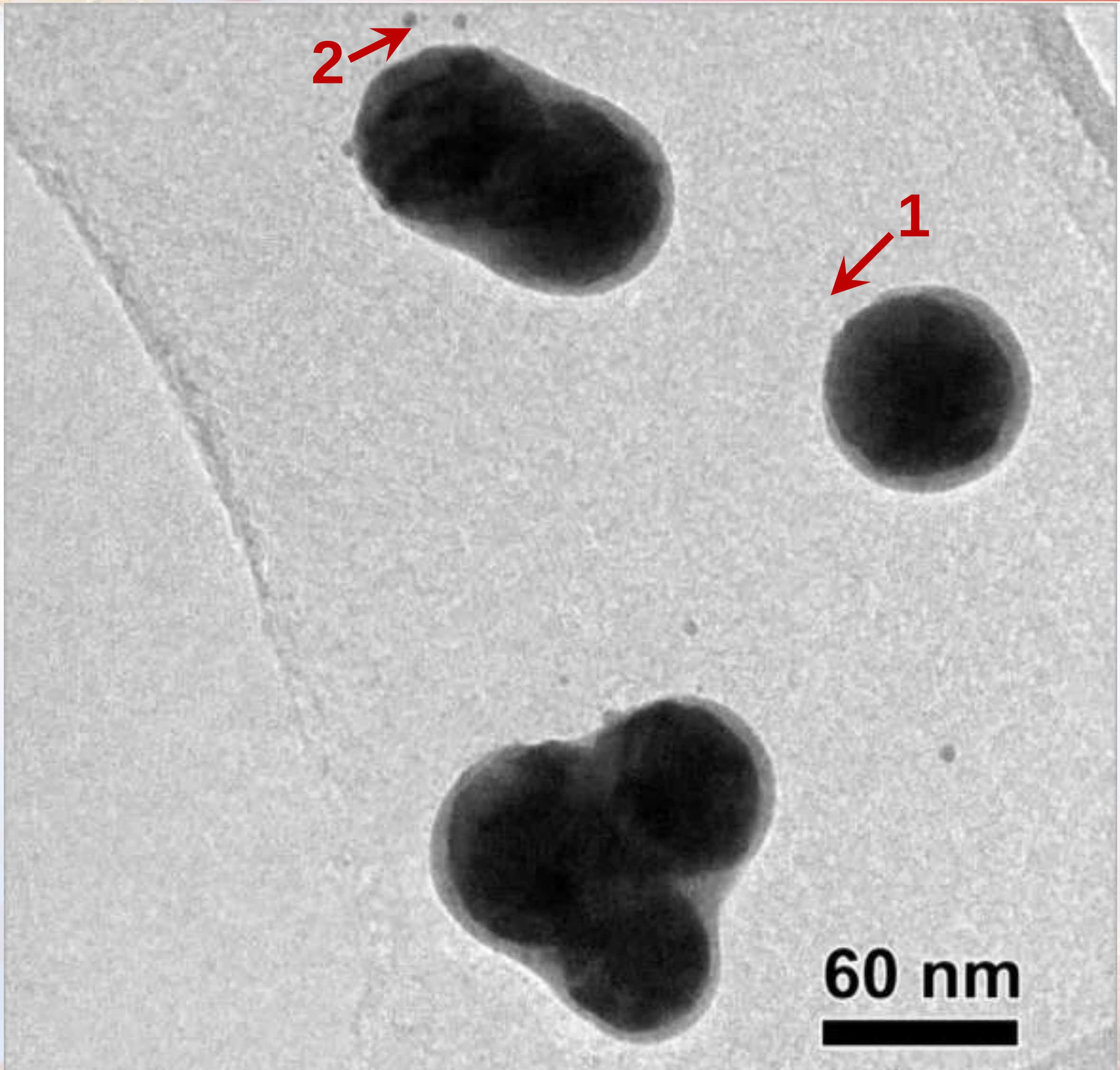
Single Ion Strikes

Collaborator: D.C. Bufford



Video playback at 2x real time.

- **2.8 MeV Au^{4+} ions into 60 nm diameter Au nanoparticles**
- **100 kx magnification**
- **Nanoscale filaments created by individual ions**

The permanent and transient structures resulting from single ion strikes can be directly observed

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

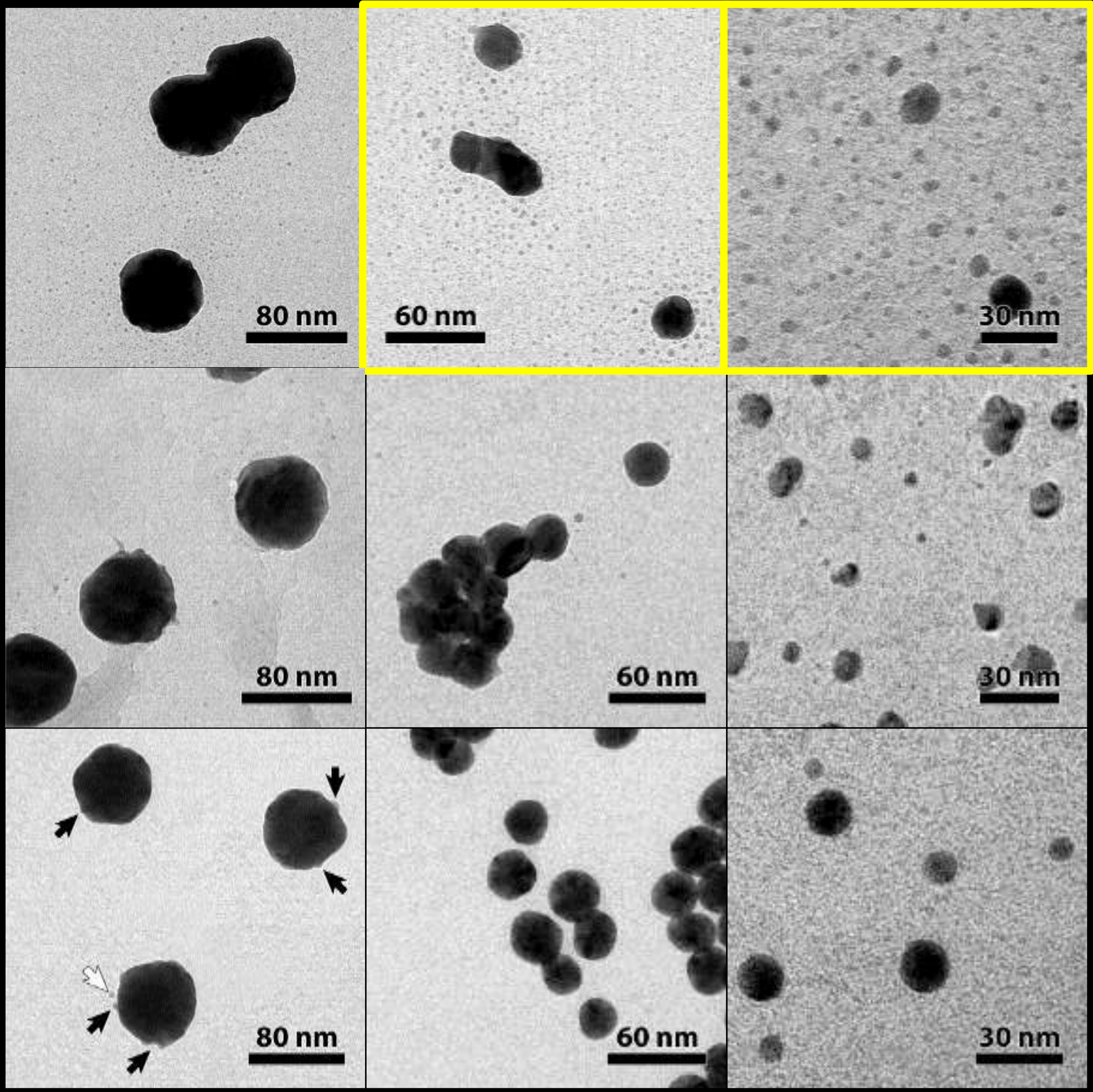
Collaborator: D.C. Bufford

60 nm | 20 nm | 5 nm

46 keV Au^{1-}
3.4×10^{14} /cm^2

2.8 MeV Au^{4+}
4×10^{13} /cm^2

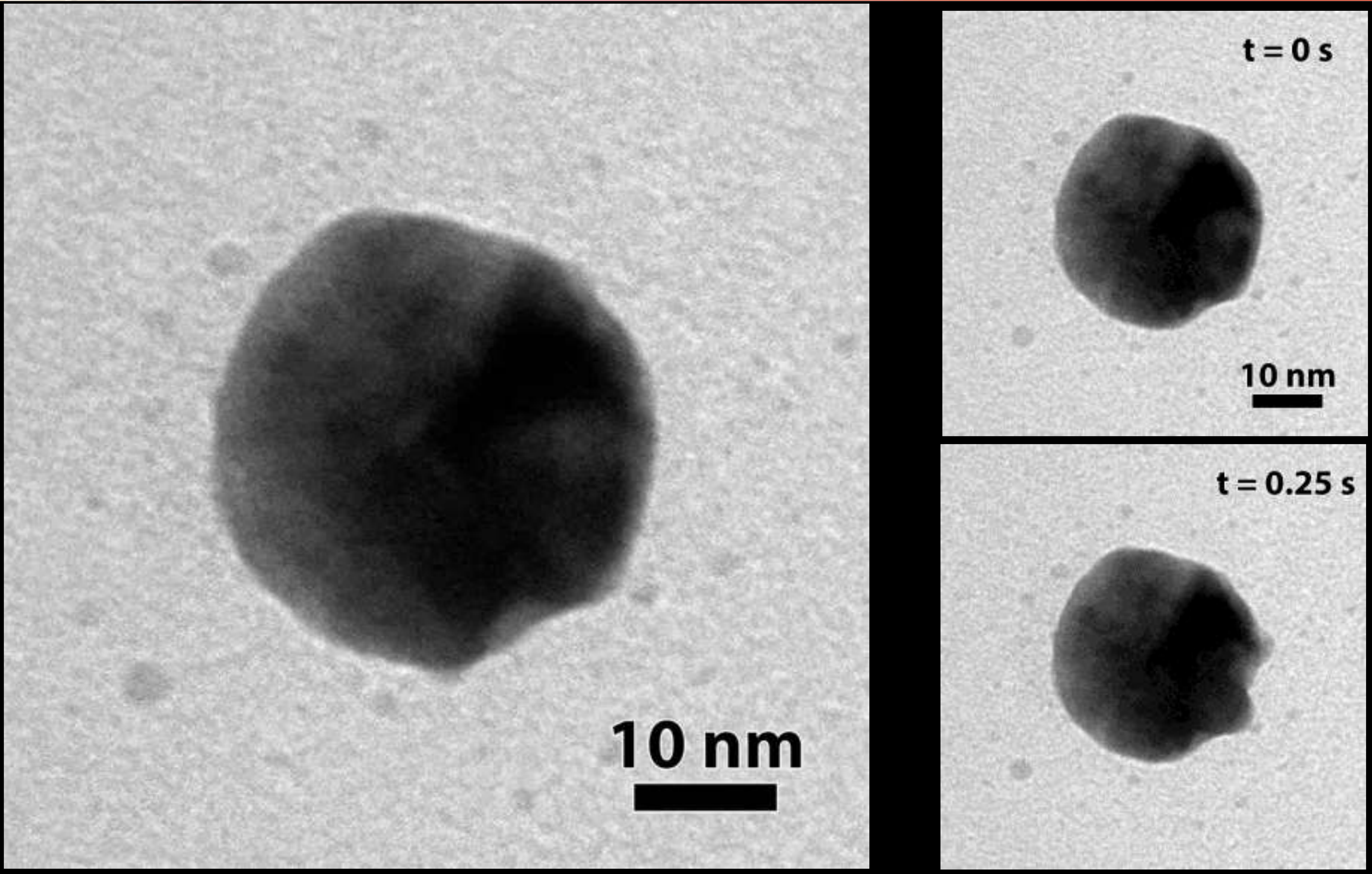
10 MeV Au^{8+}
1.3×10^{12} /cm^2



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

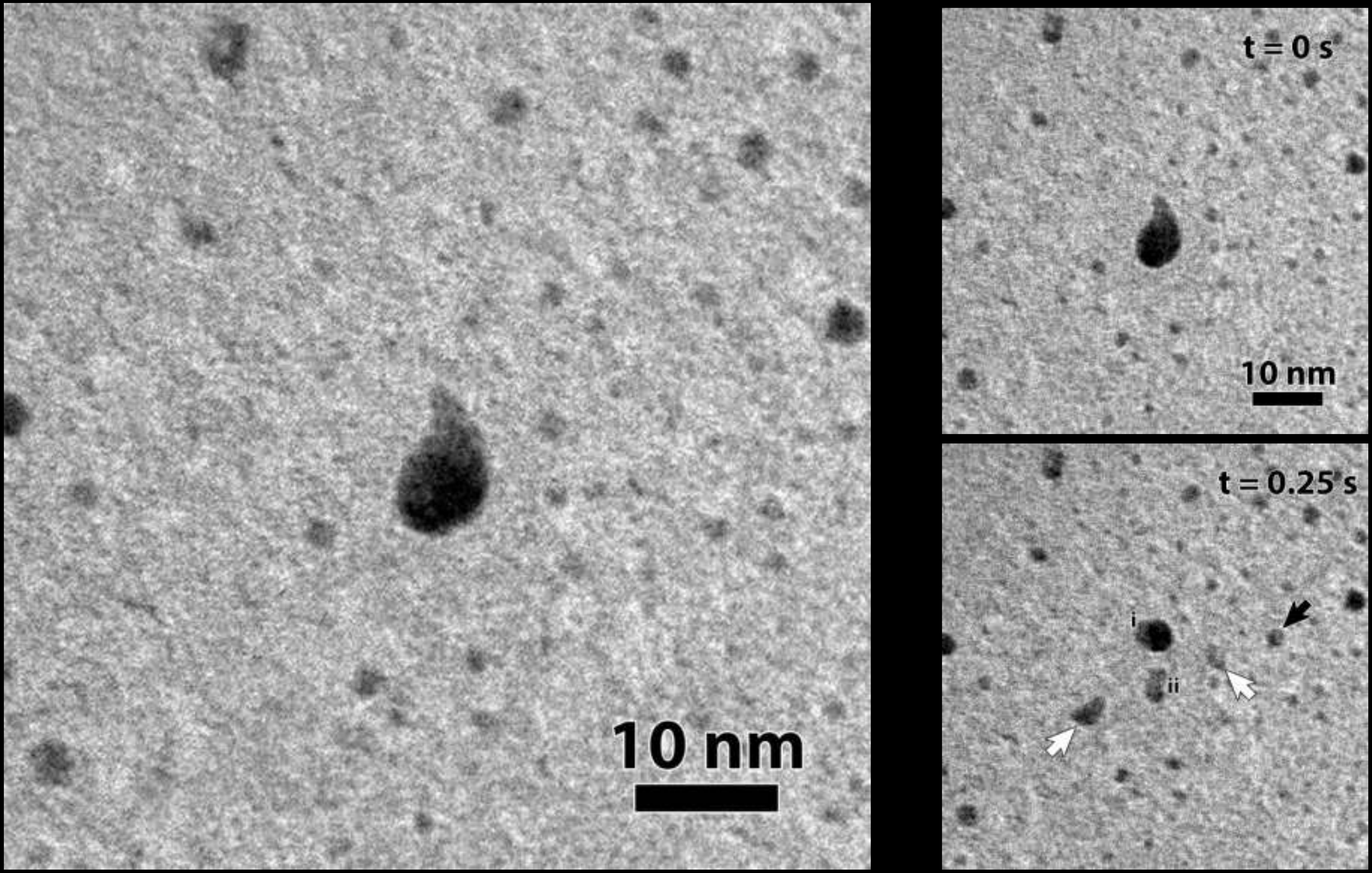
Single Ion Effects with 46 keV Au^{1-} ions: 20 nm

Collaborator: D.C. Bufford



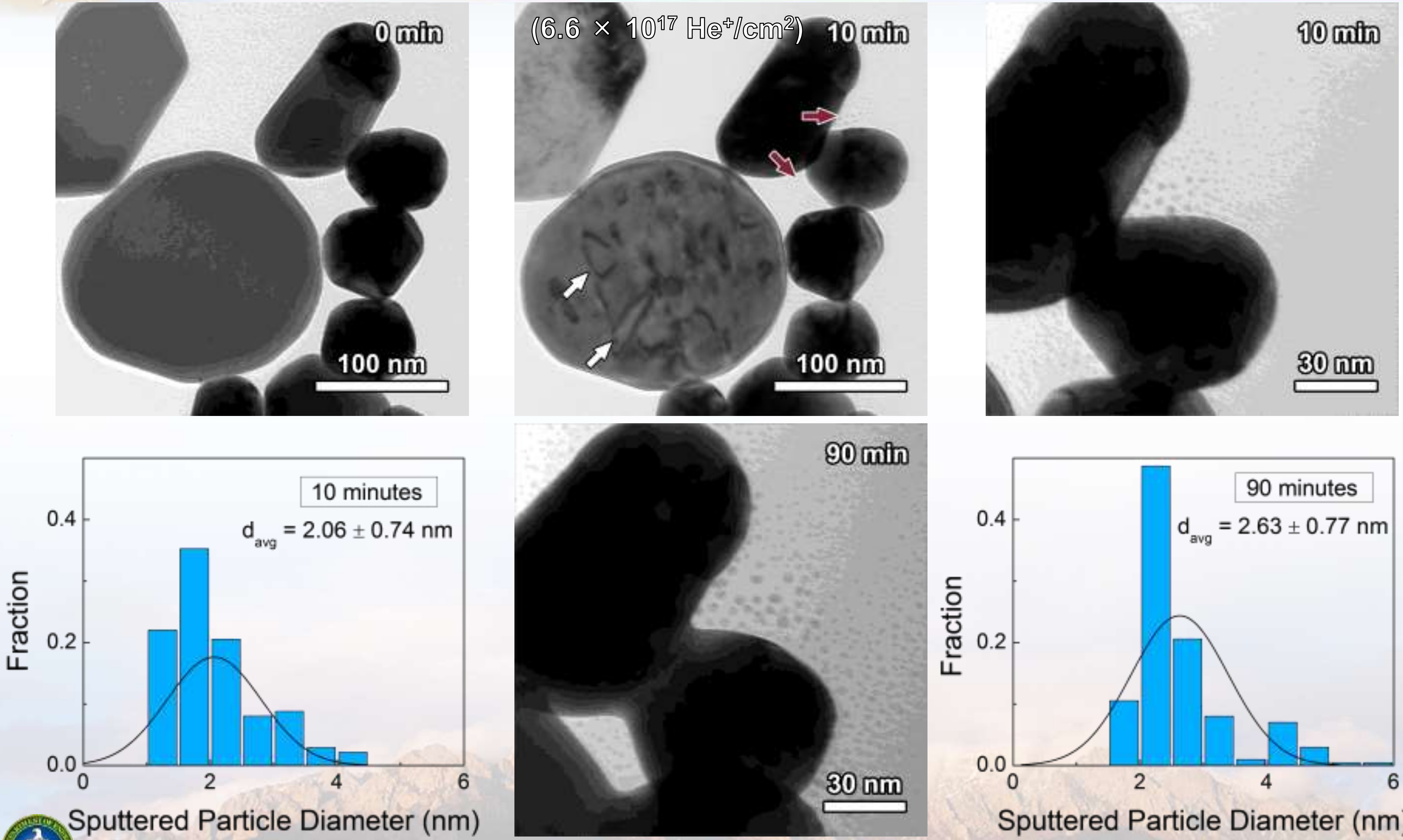
Single Ion Effects with 46 keV Au^{1-} ions: 5 nm

Collaborator: D.C. Bufford



Formation of Dislocation Loops & Sputtered Particles due to He implantation

Collaborators: D.C. Bufford, S.H. Pratt & T.J. Boyle



Advanced Microscopy Techniques Applied to Nanoparticles in Radiation Environments

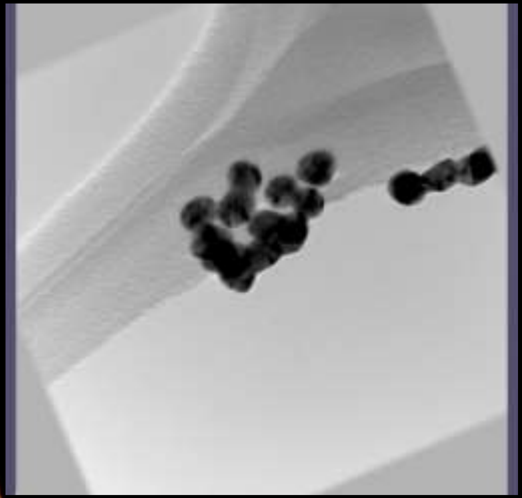
Collaborators: S.H. Pratt & T.J. Boyle

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

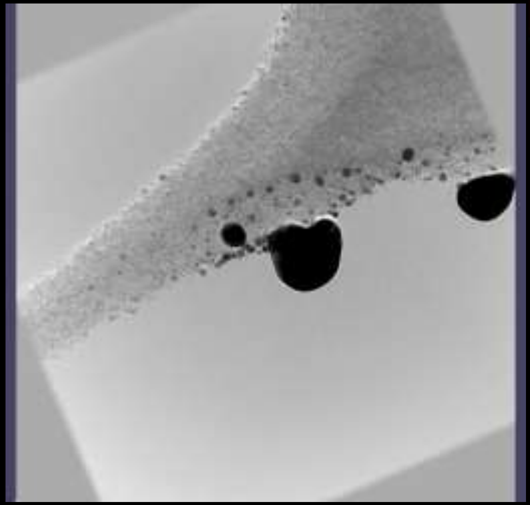
+

Hummingbird tomography stage

Aligned Au NP tilt series - unirradiated

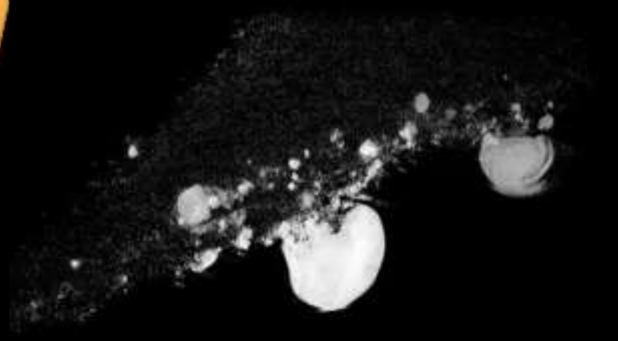


Aligned Au NP tilt series - irradiated



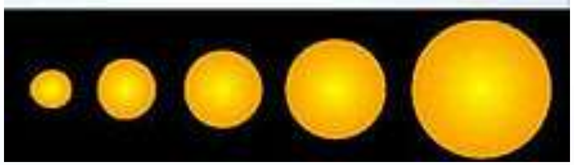
Unirradiated Au NP model

Irradiated Au NP model



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

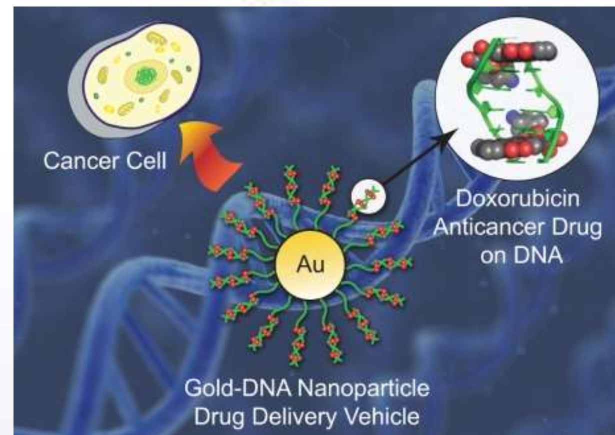
Nanoparticles in Extreme Environments

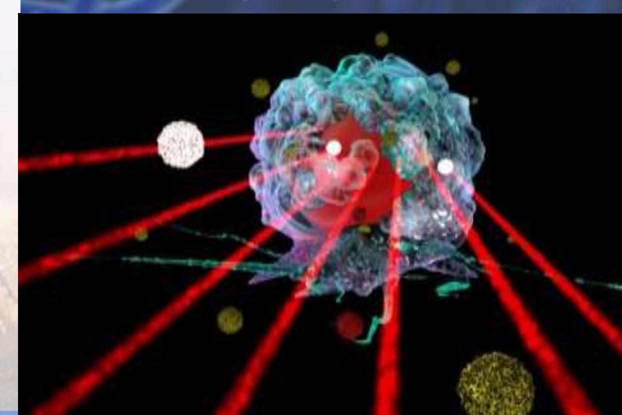


Nenoff et. al. *SNL News Release* (2007).

Au nanoparticles (Au NP) are of interest due to their unique optical, electronic, molecular-recognition, and catalytic properties

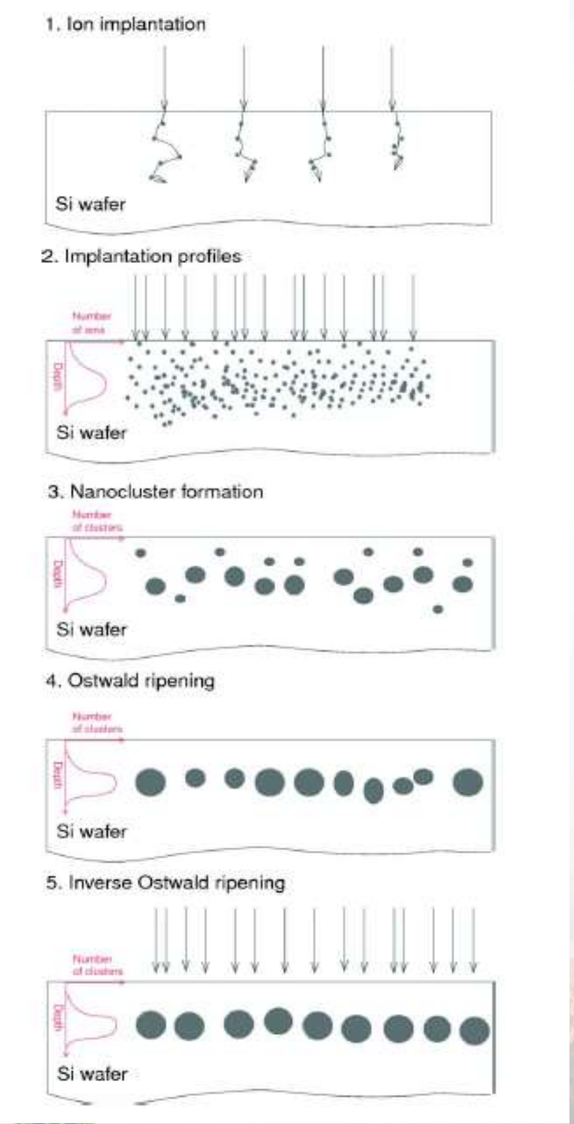
- **Photothermal and laser ablation cancer therapies – concentrate IR into heat**
- **Tumor targeting and drug carriers – enhance dose delivery**
- **Catalysts for air-pollution control**



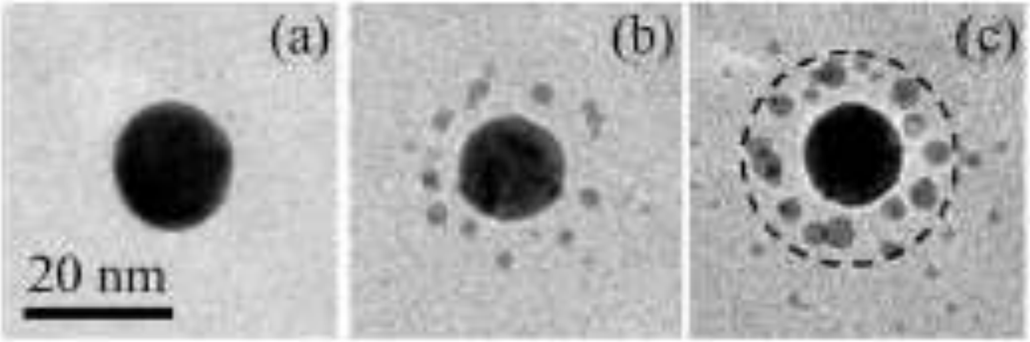


Do the unique properties of the NP withstand extreme radiation?

Previous Studies Investigated Embedded Particles



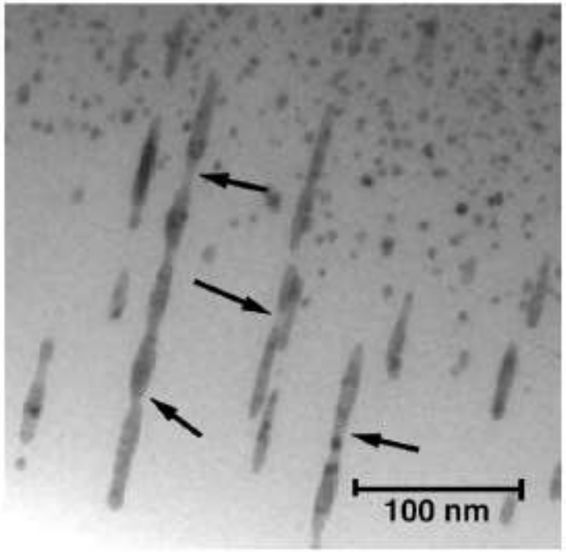
A.V. Krasheninnikov and K. Nordlund. *J. Appl. Phys.* 107 (2012) 071301.



G. Rizza et. al. *Phys. Rev. B 76* (2007) 245414.

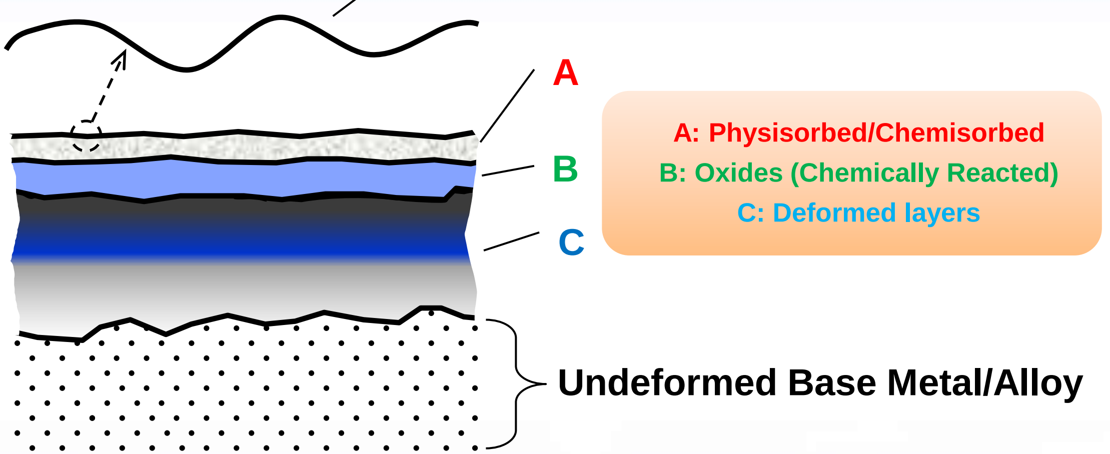
Au NCs in SiO_2 matrix irradiated at increasing fluences with 4 MeV Au ions at room temperature. Two generations of satellites observed.

Ion tracks can be formed in SiO_2 matrices and nanoparticle elongation and nanowire formation has been observed. Pt NP are shown, irradiated with 185 MeV Au ions. Arrows point to nanorod fragments.



M.C. Ridgway et. al. *NIM B 267* (2009) 931.

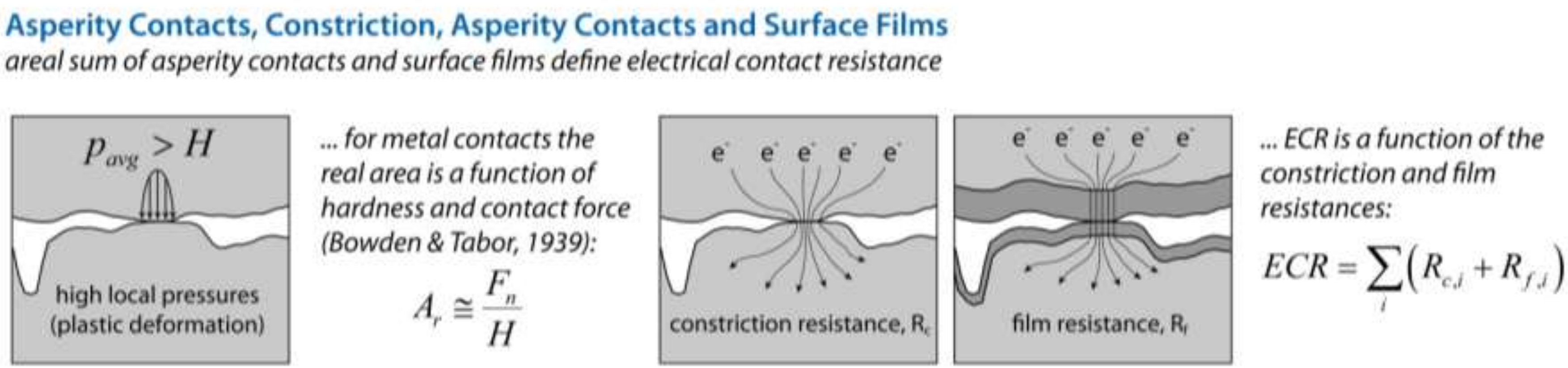
Nature of Metallic Surfaces



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

(Low Adhesive Wear)

Or maximized for reduced electrical contact resistance (ECR)



Archard, Journal of Applied Physics (1953) 24:981

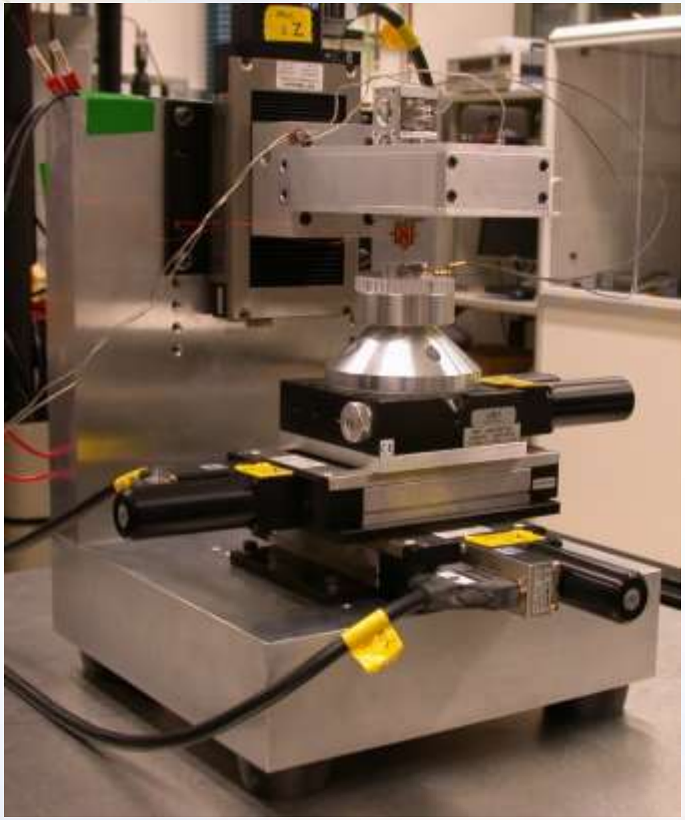
R. Holm, Electrical Contacts Handbook (1958) Berlin: Springer-Verlag

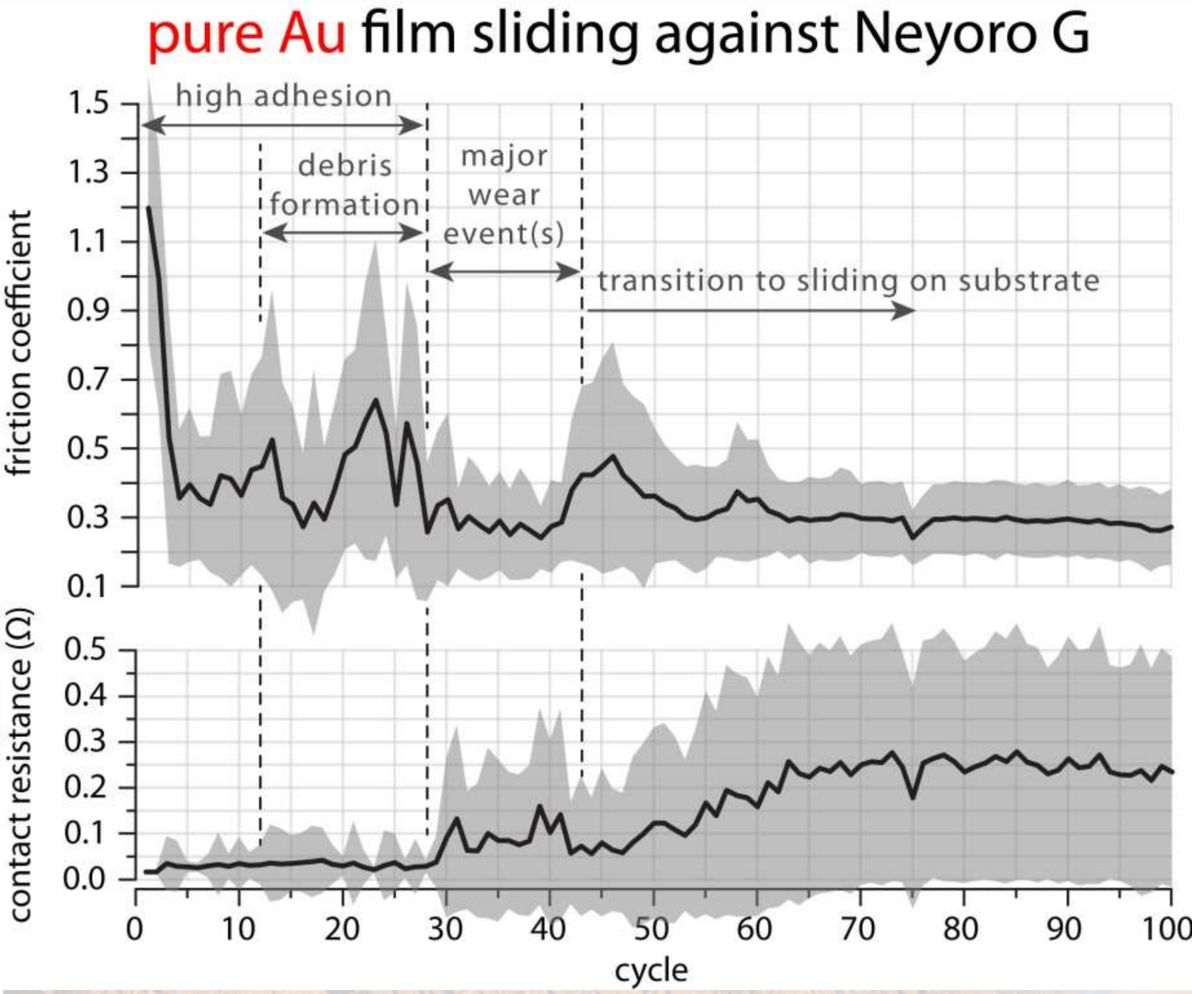
Greenwood & Williamson, Proc. Royal Society (1966) A295:300

T.W. Scharf & S.V. Prasad, Journal of Material Science (2013) 48:511-531

ECR-Friction Behavior of Pure Au

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





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

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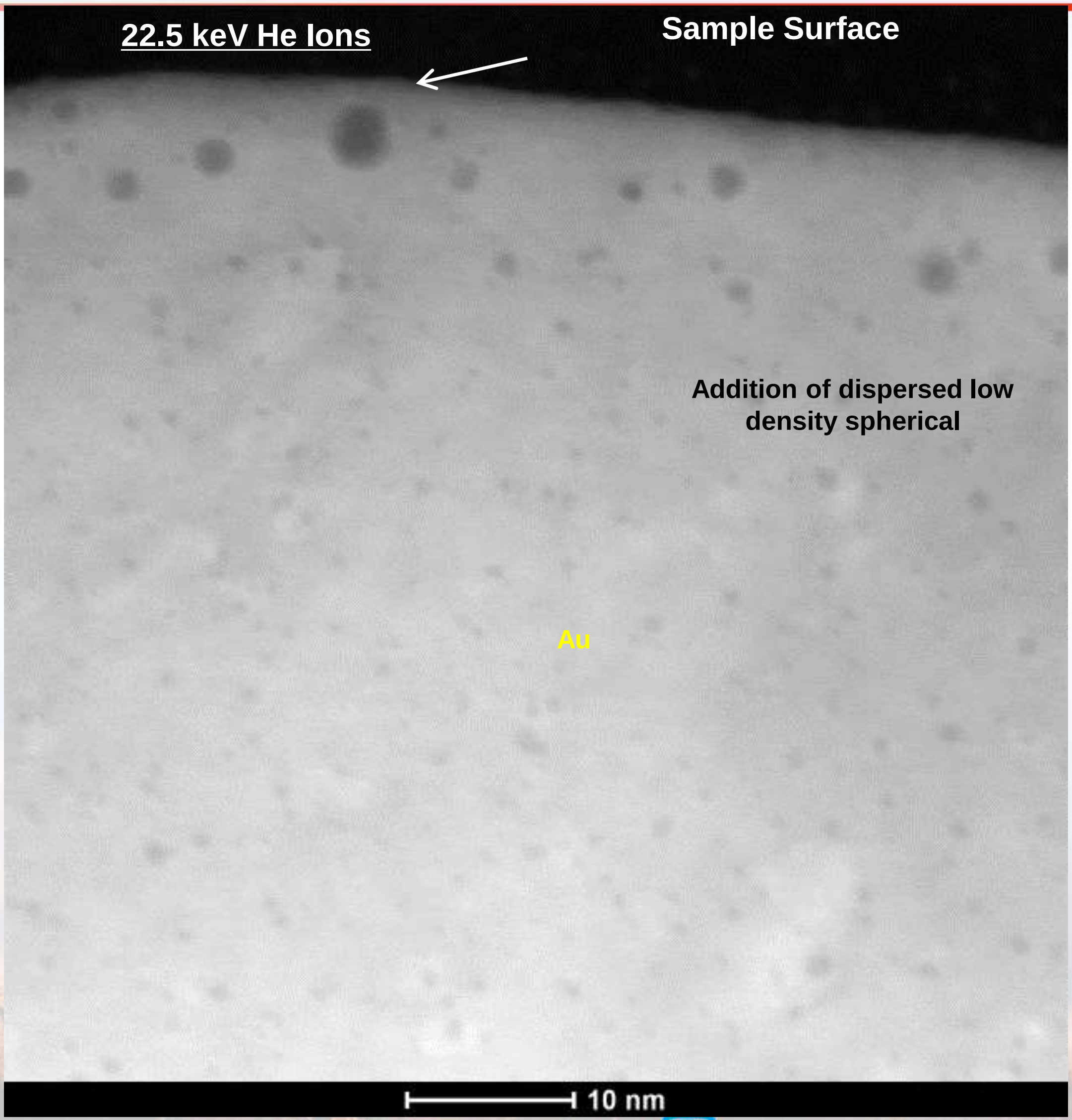
Modeling and STEM of He Implantation

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

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

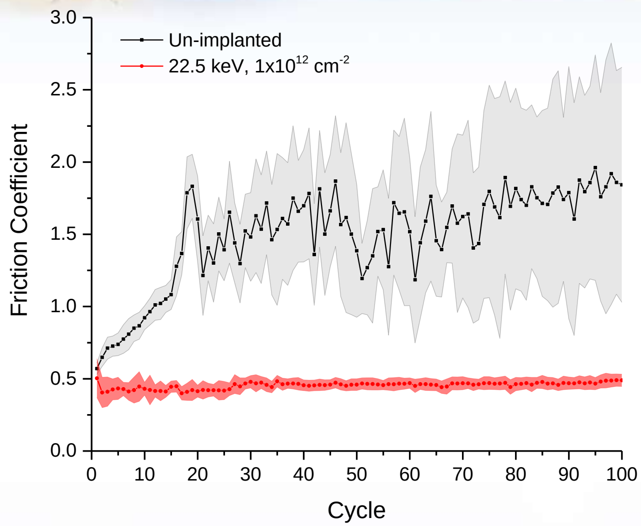
- AC-STEM used to observe the distribution of implanted bubbles
- Bubble locations are in good agreement with SRIM ion range predictions

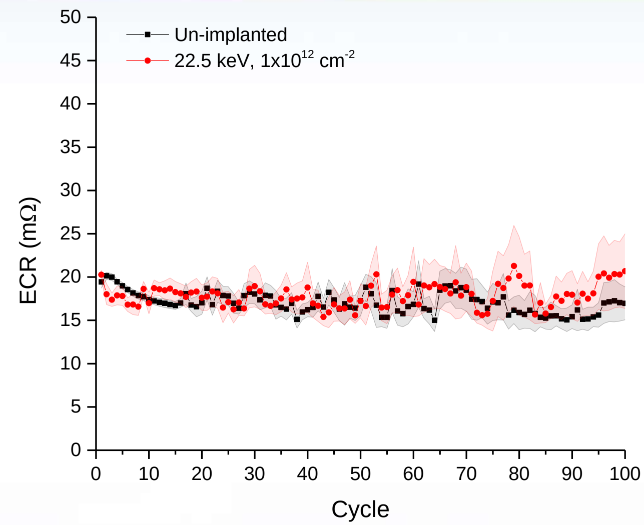
He implantation result in small dispersed spherical structures assumed to be He bubbles. Dispersion and depth can be tailored



ECR and Wear Measurements

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

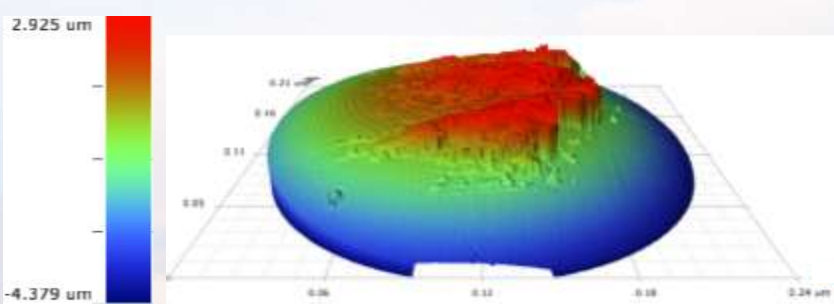




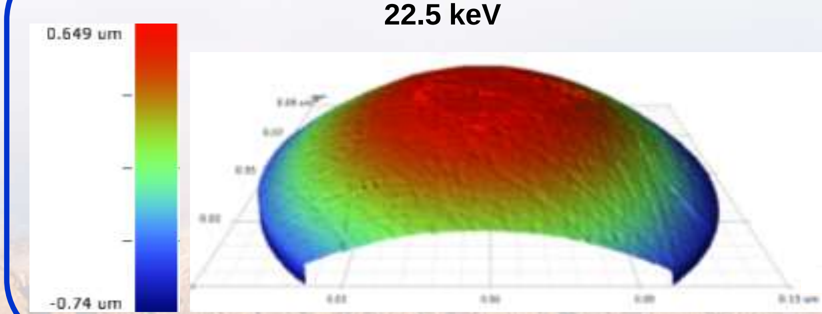
Friction is significantly reduced with ^{3}He implantation while maintaining ECR performance

Scanning white light interferometer topographical construction of riders after 100 cycles

Rider after 100 cycles against Un-implanted Au



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



Wear is significantly reduced with minimal effect in ECR

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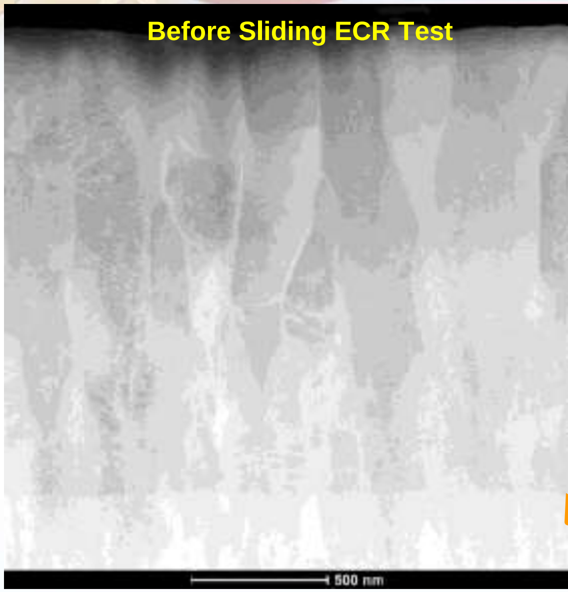
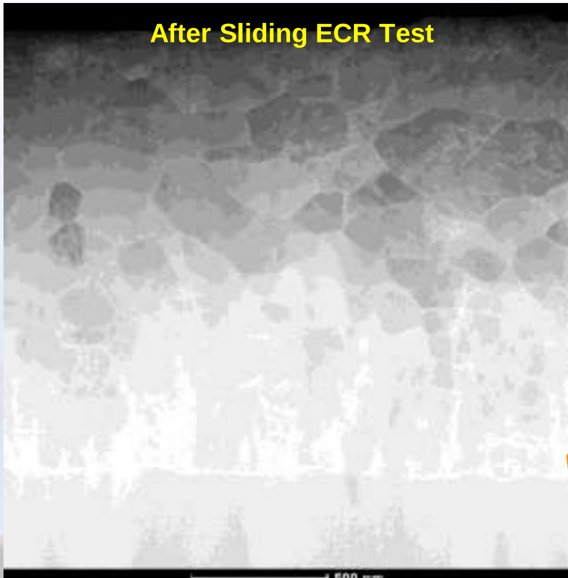
STEM Images of Sub-surfaces

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



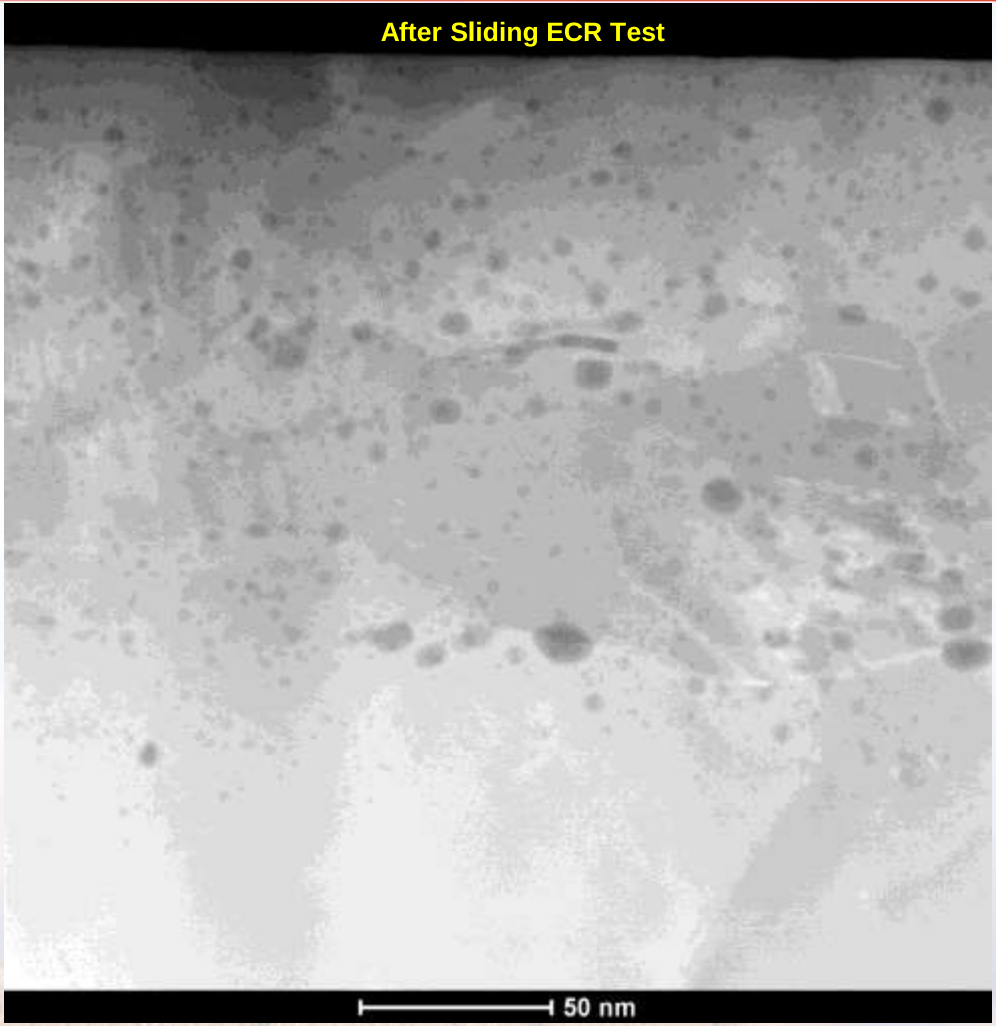


22.5 keV

1×10^{12} cm^{-2}

Recrystallization is observable after 100 cycles

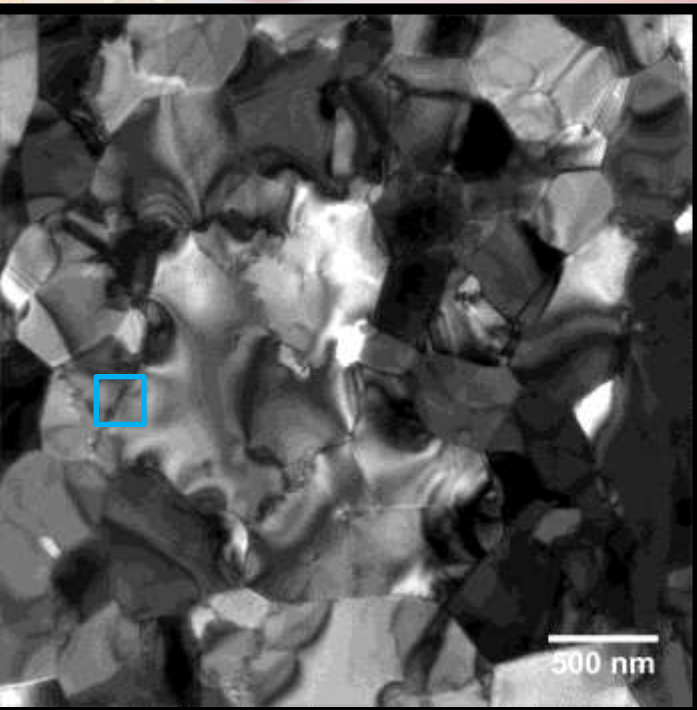
Au – Pt Interface



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

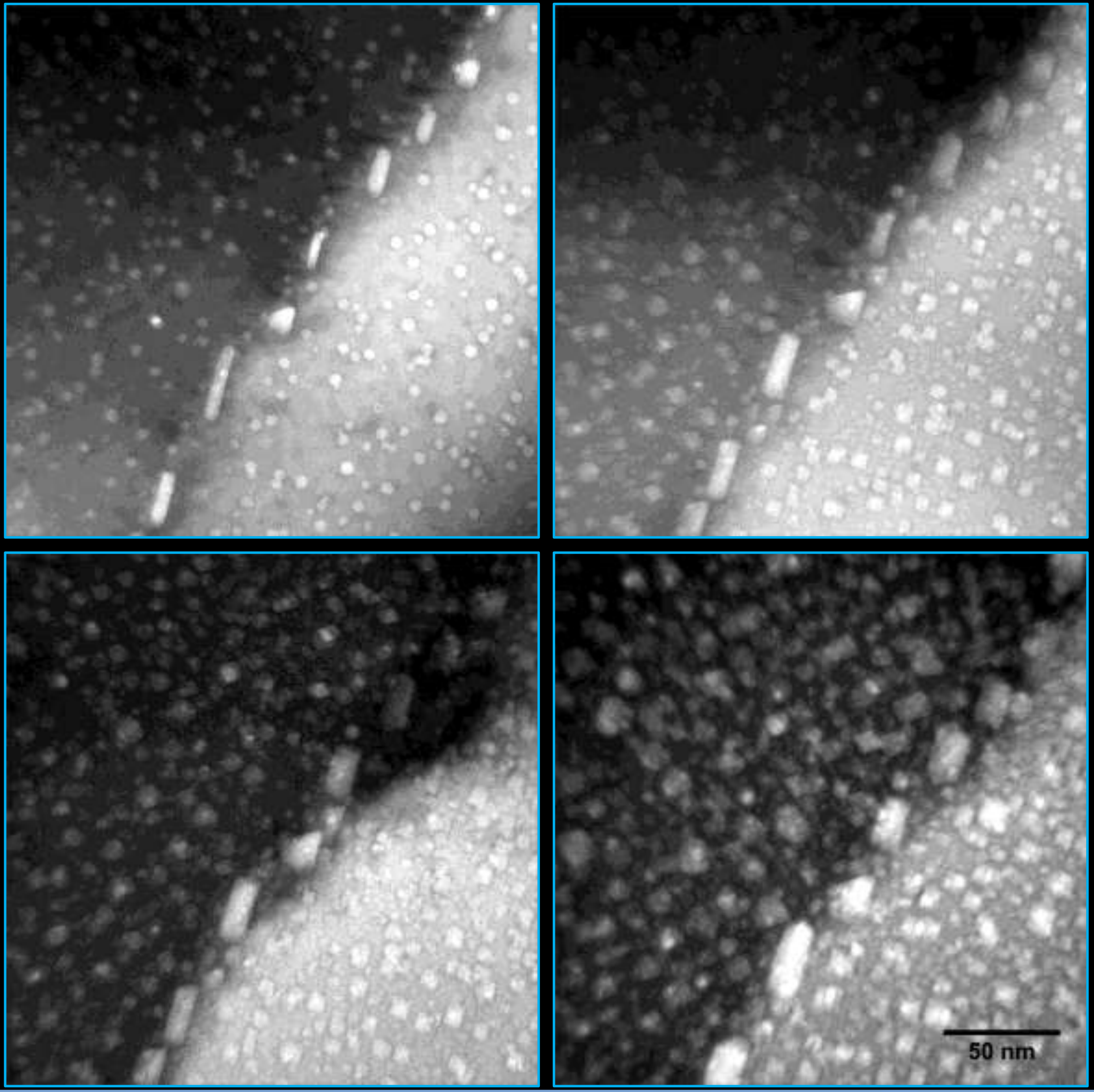
In situ Implantation

Collaborators: C. Chisholm, P. Hosemann, & A. Minor

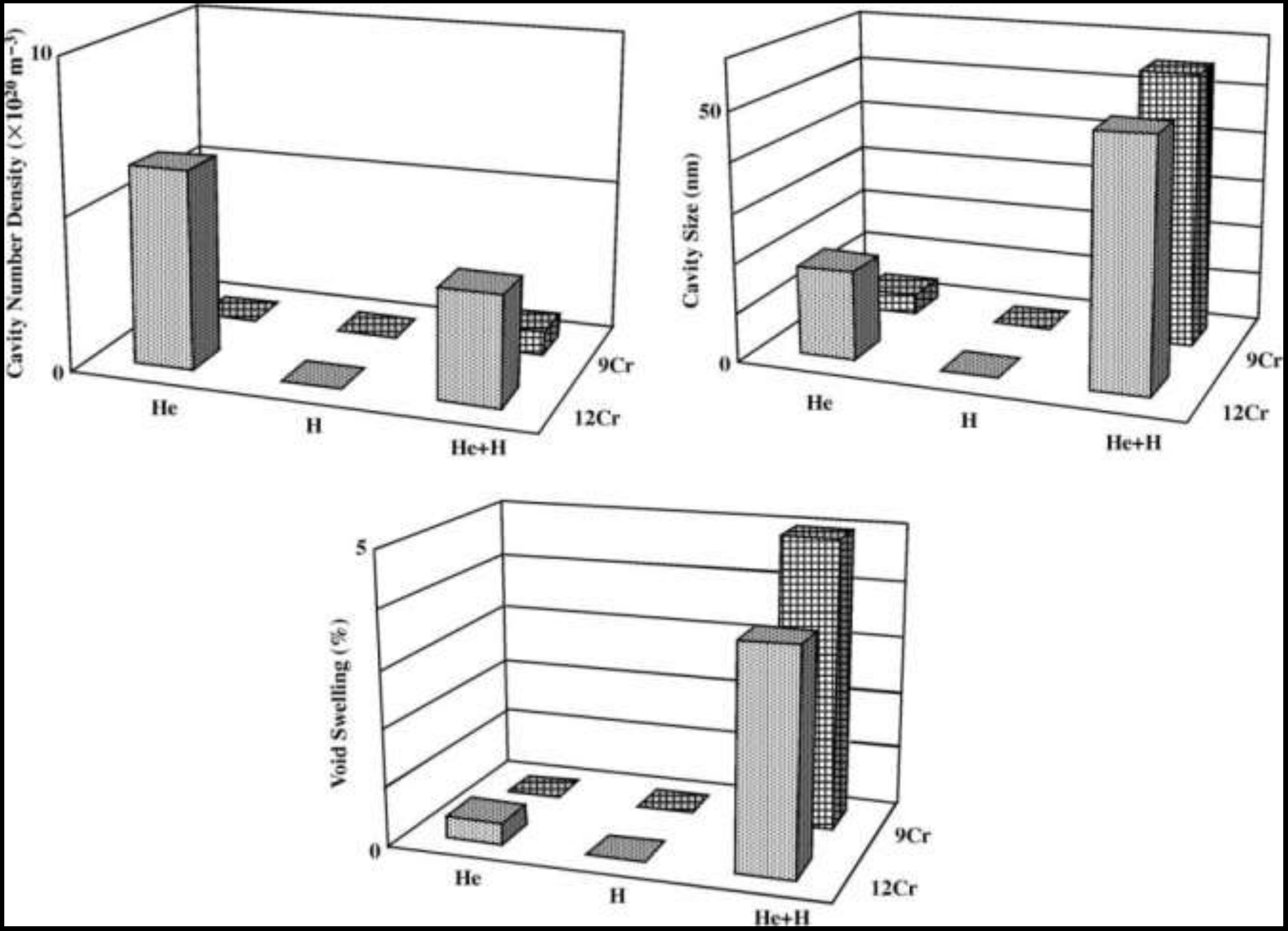


Gold thin-film implanted with 10keV He^{2+}

Result: porous microstructure



H, He, and Displacement Damage Synergy



T. Tanaka et al. "Synergistic effect of helium and hydrogen for defect evolution under milt-ion irradiation of Fe-Cr ferritic alloys"
J. of Nuclear Materials 329-333 (2004) 294-298

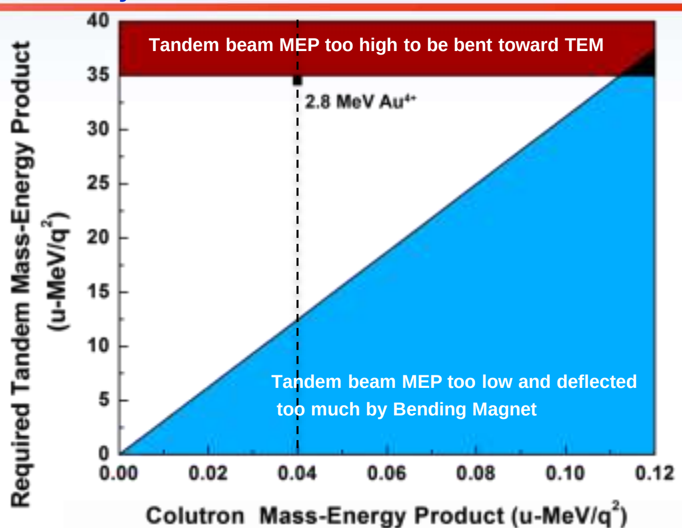
Coupling Effect

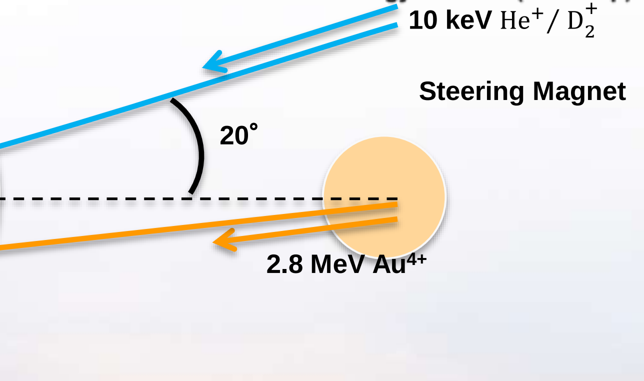
- H and He are produced as decay products
- The relationship between the point defects present, the interstitial hydrogen, and the He bubbles in the system that results in the increased void swelling has only been theorized.
- The mechanisms which governs the increased void swelling under the presence of He and H have never been experimental determined

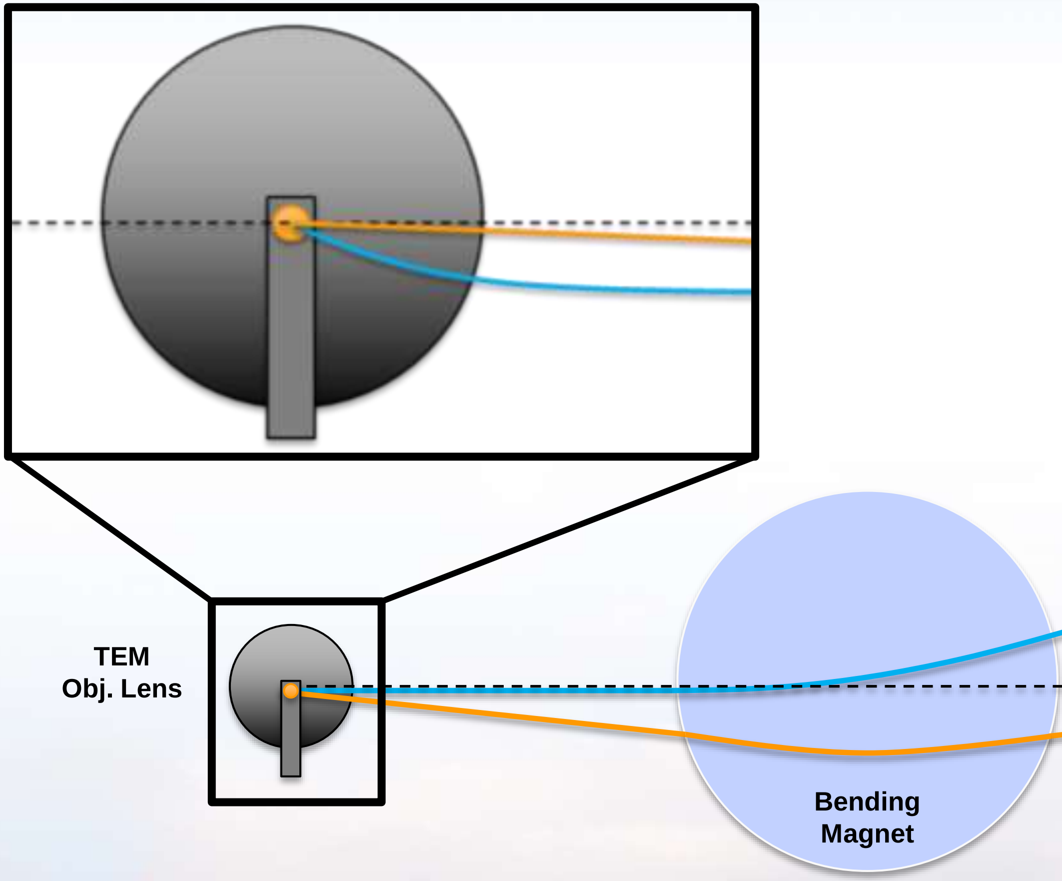
Difficulty of performing triple-beam irradiation has resulted in a limited number of facilities world wide

Modeling Beam Mixing and Deflection

Collaborators: M. Steckbeck, D.C. Bufford, & B.L. Doyle







- Must compensate for deflection of Tandem beam by bending magnet
  Colutron beams deflected by the TEM objective lens
- Insignificant deflection of Tandem beams
- With 10 keV He/D_2 we can use Tandem beams ≳13 MeV/q^2
- Au, He, and D_2 ions all reach the sample concurrently

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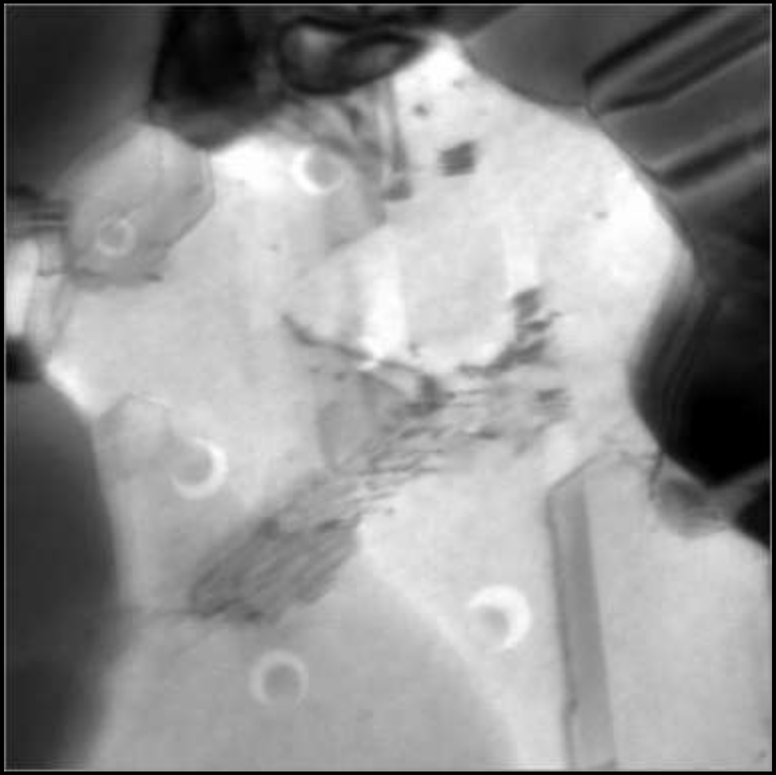
Single Ion Strikes

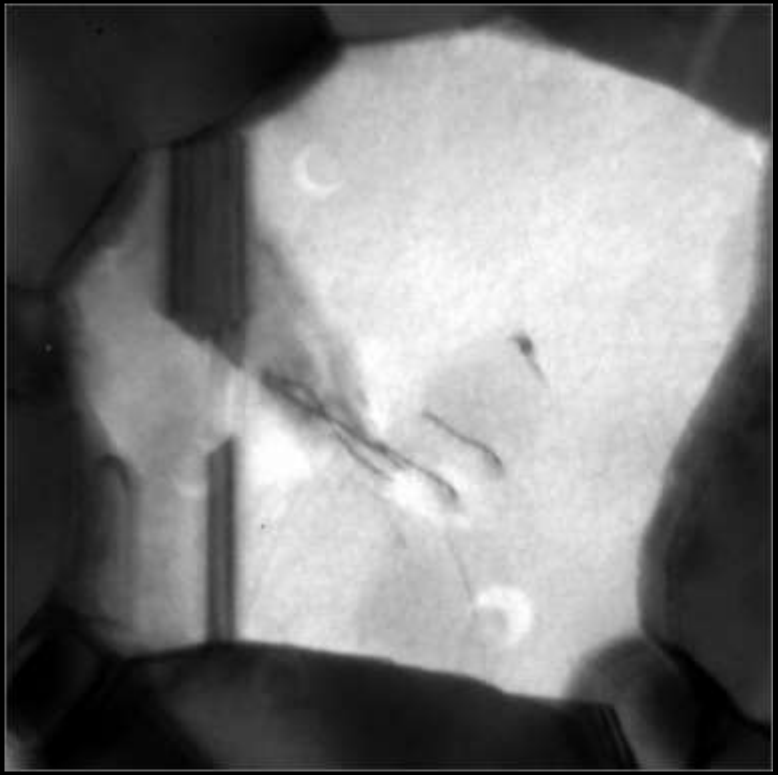
Collaborators: C. Chisholm , P. Hosemann, & A. Minor

7.9×10^9 ions/cm^2/s



VS

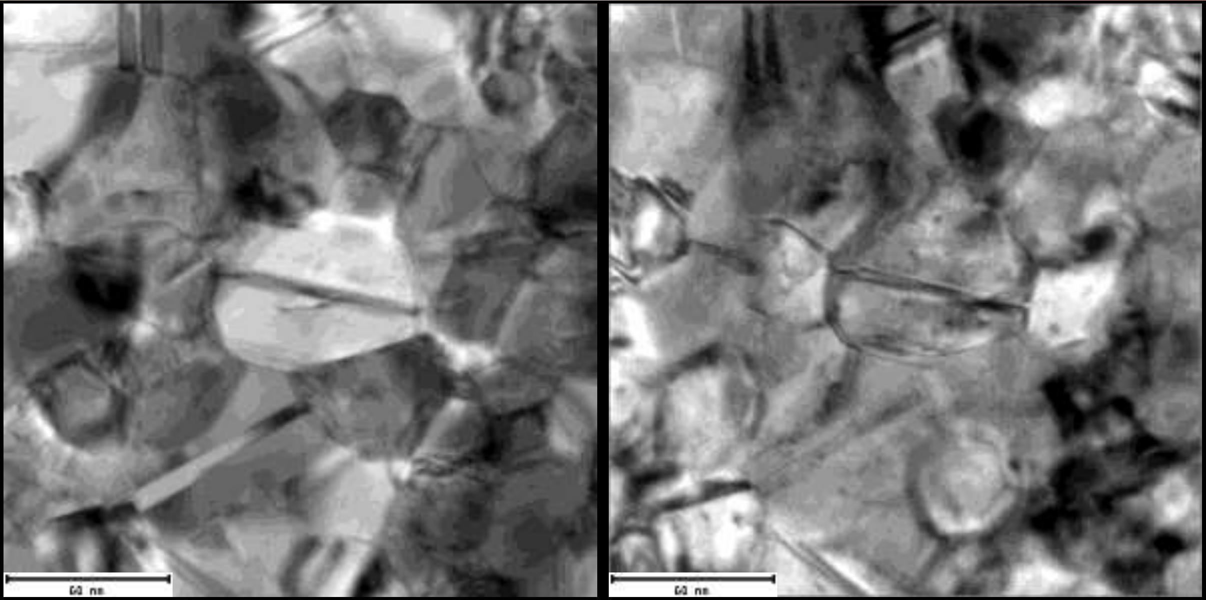
6.7×10^7 ions/cm^2/s



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

Nanocrystalline vs. Nanoporous Au results

Collaborators: N. Briot and T.J. Balk

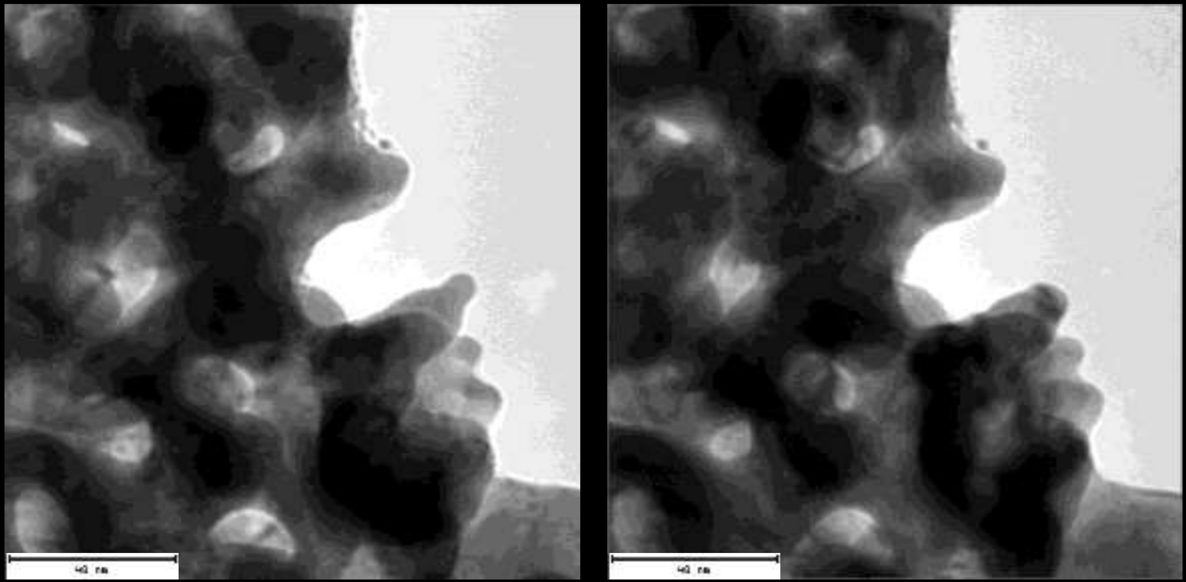


Nanoporous Au after ~ 6.6 dpa at 46 keV:

Melting of the ligament

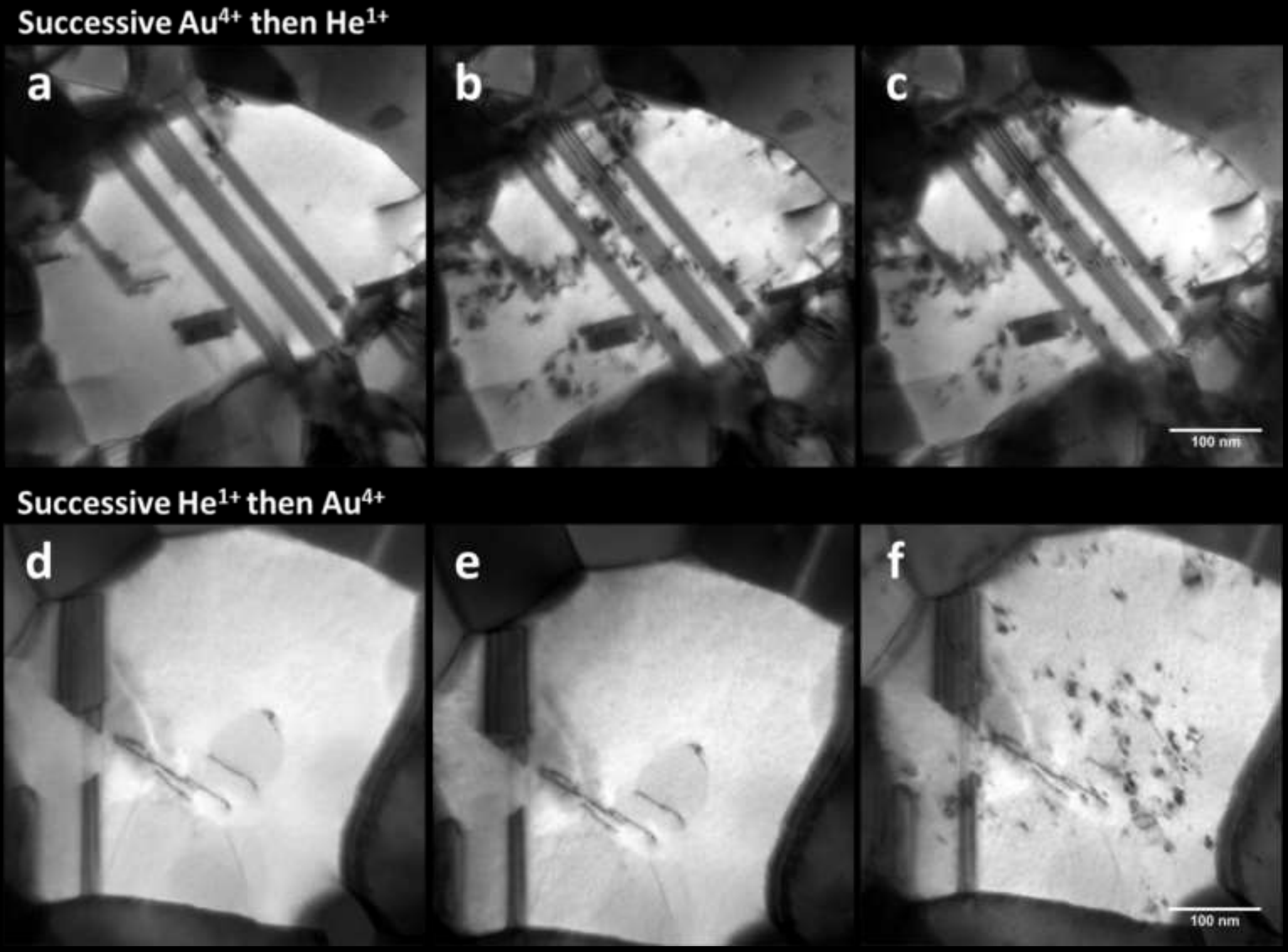
Nanocrystalline Au after ~ 0.5 dpa at 46 keV:

Lots of defects constantly created



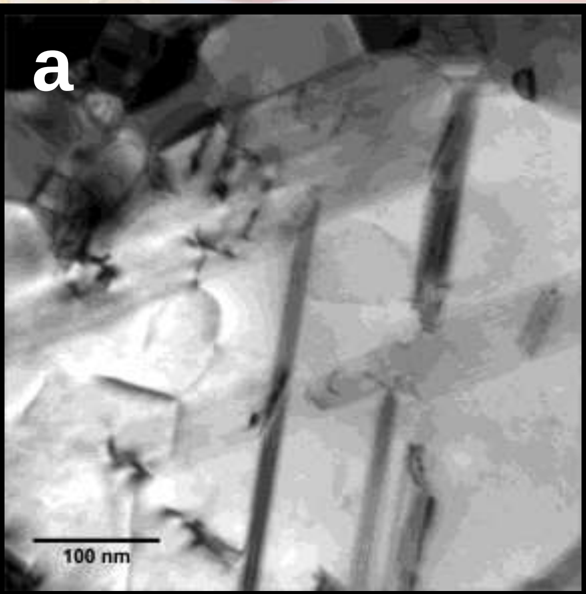
In situ Successive Implantation & Irradiation

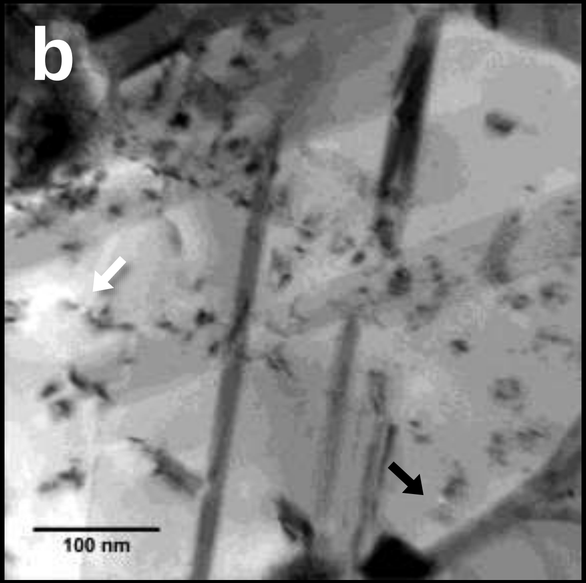
Collaborators: C. Chisholm , P. Hosemann, & A. Minor



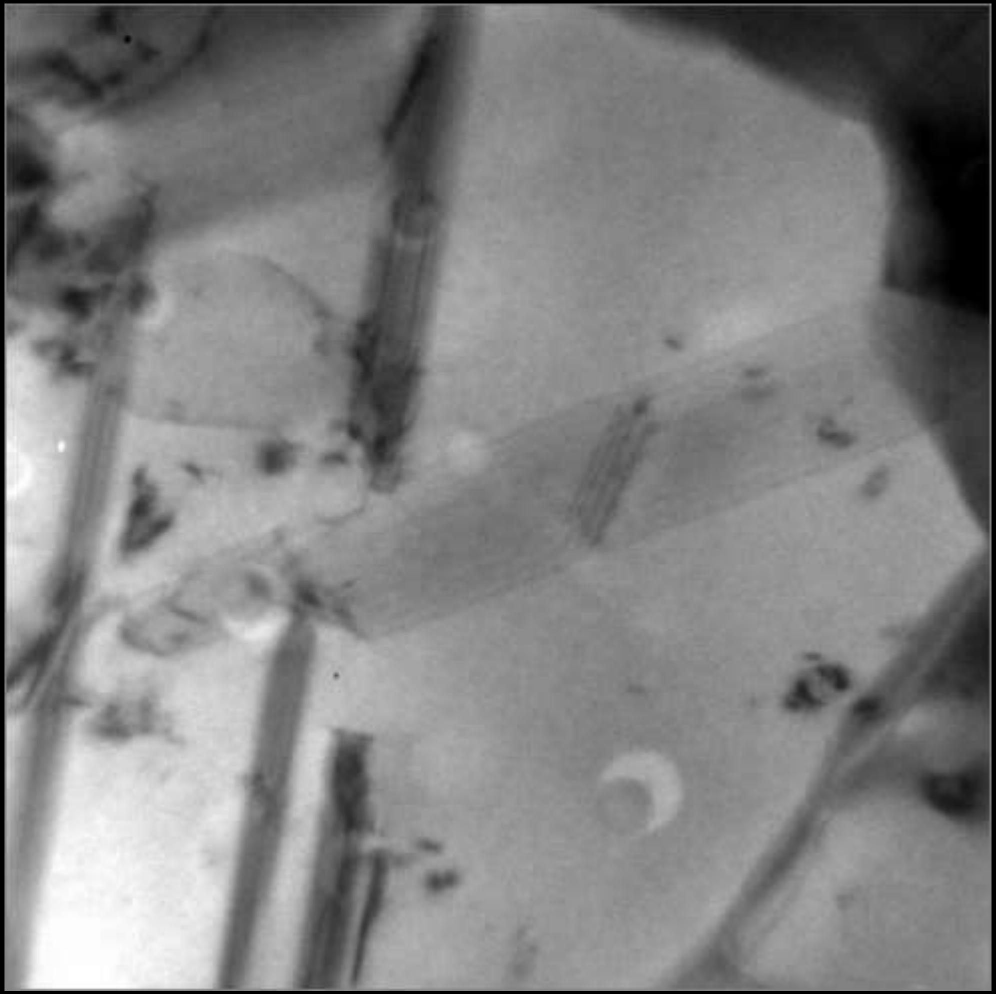
In situ Concurrent Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor





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



Single Ion Strikes During Concurrent Irradiation: Nucleation of Helium Cavities

Collaborators: C. Chisholm, P. Hosemann, & A. Minor

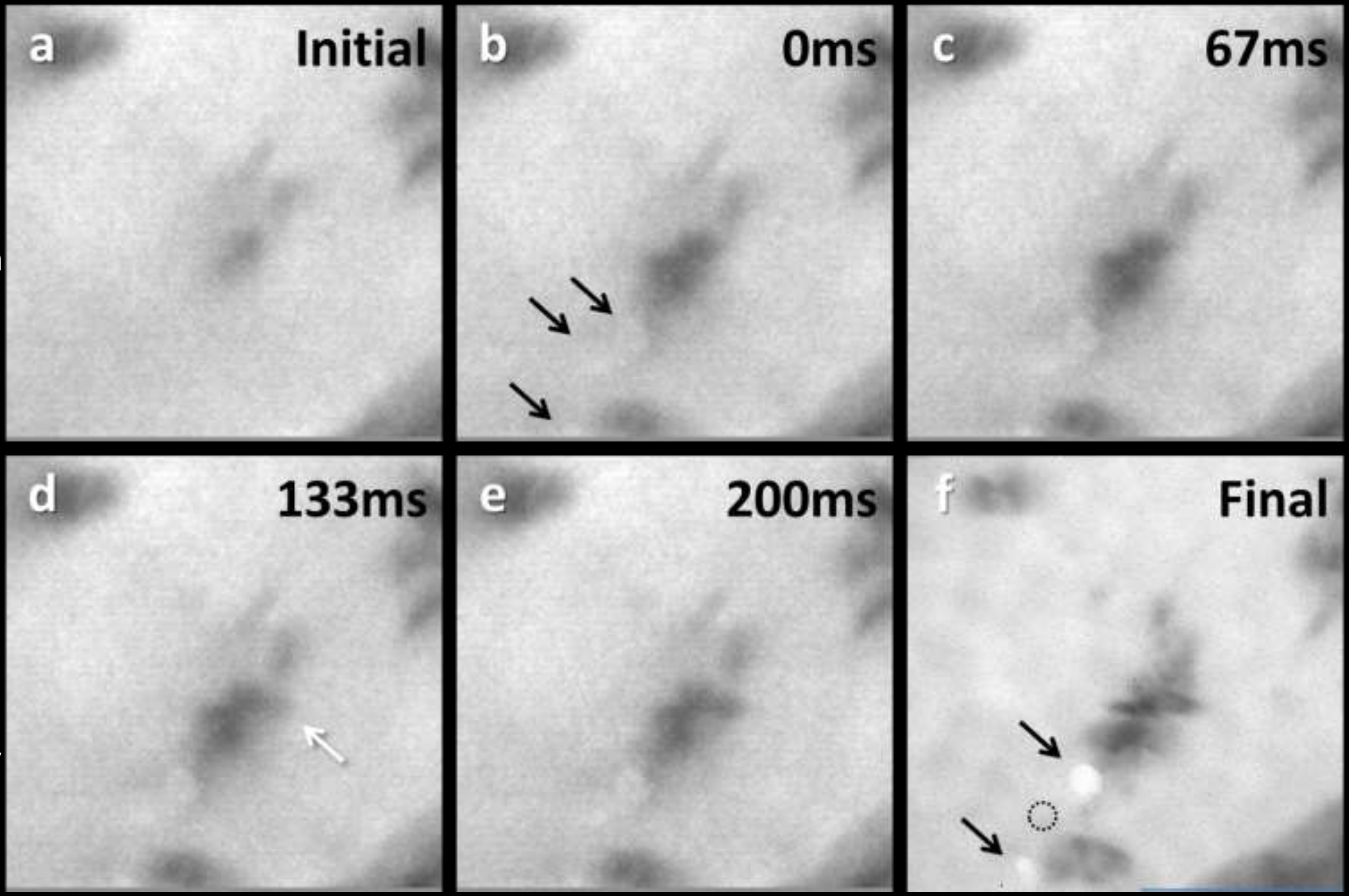
a) Initial microstructure

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

d) Cascade damage still evolving

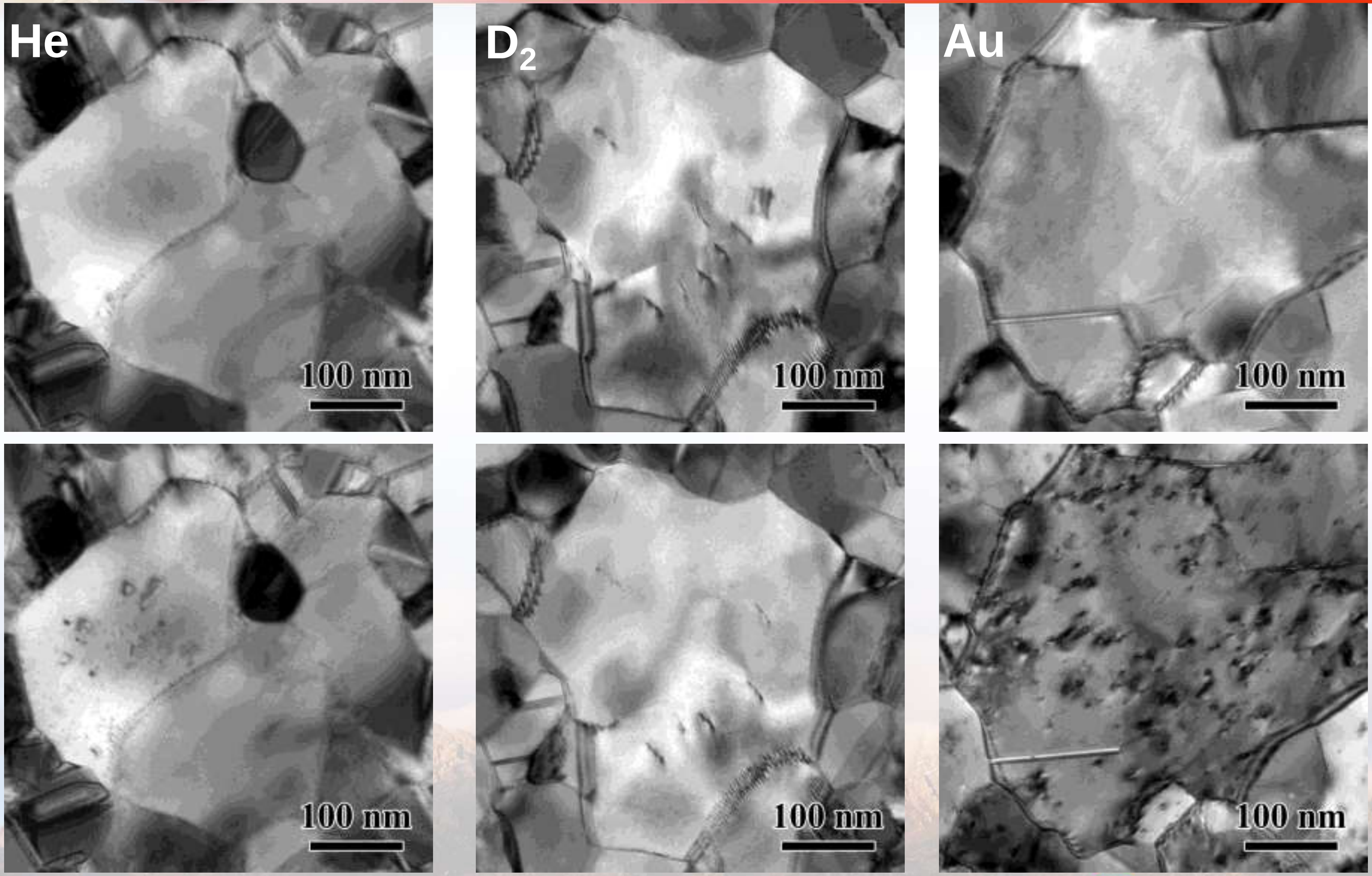
e) Apparent stability

f) Final microstructure: Only two remaining cavities



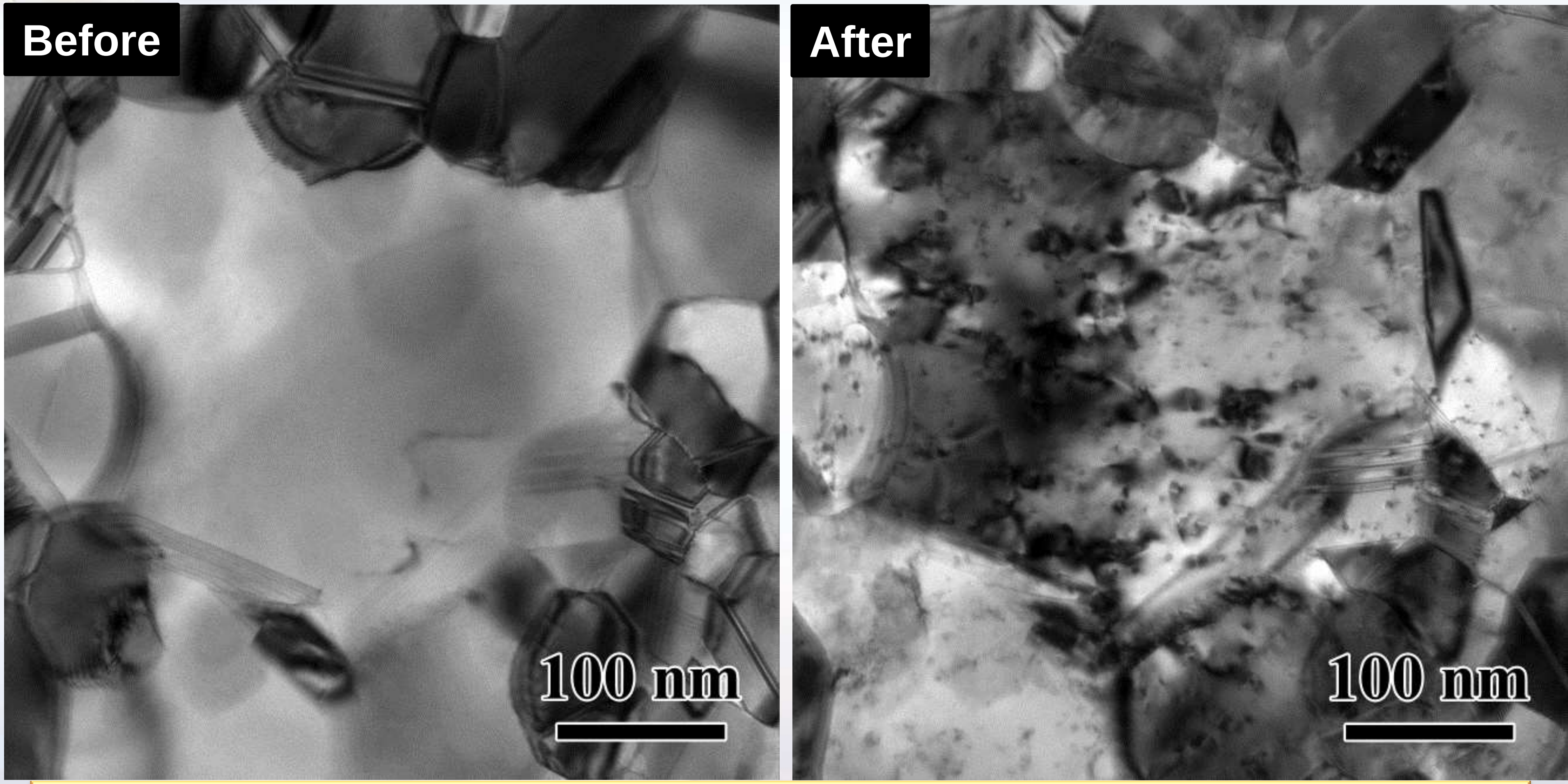
Aligned Individual Colutron and Tandem Beams

Collaborators: D.C. Bufford



Concurrent 10 keV He, 10 keV D_2, and 3 MeV Au

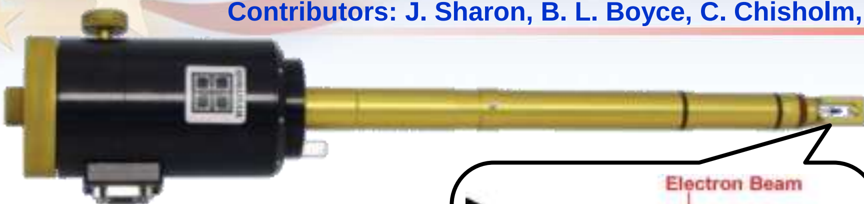
Collaborators: D.C. Bufford

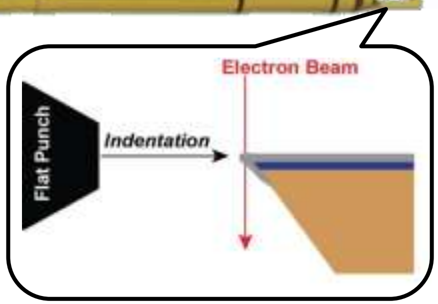


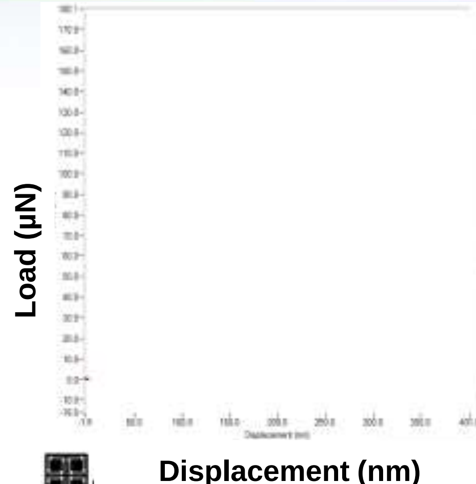
In-situ triple beam He, D_2, and Au beam irradiation has been demonstrated on Sandia's I^3TEM! Intensive work is still needed to understand the defect structure evolution that has been observed.

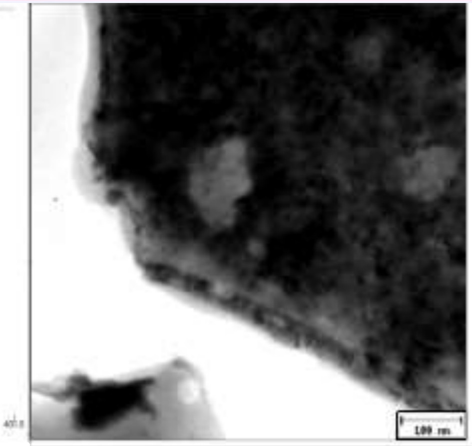
Next Steps: *In situ* TEM Quantitative Mechanical Testing

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





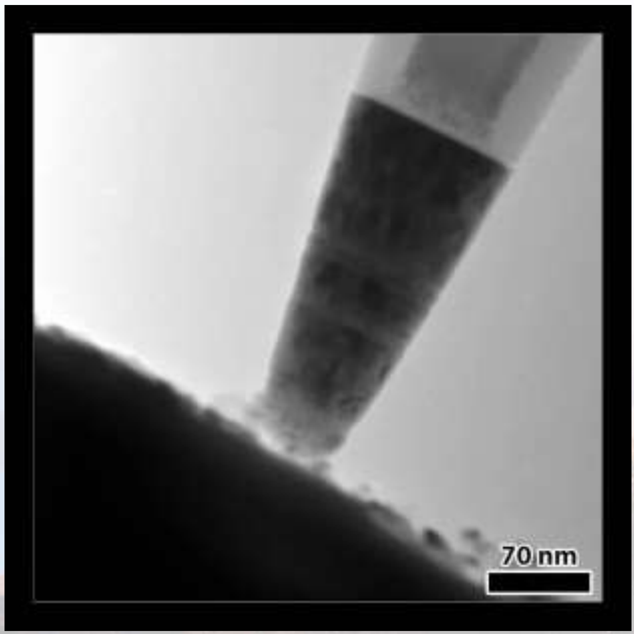


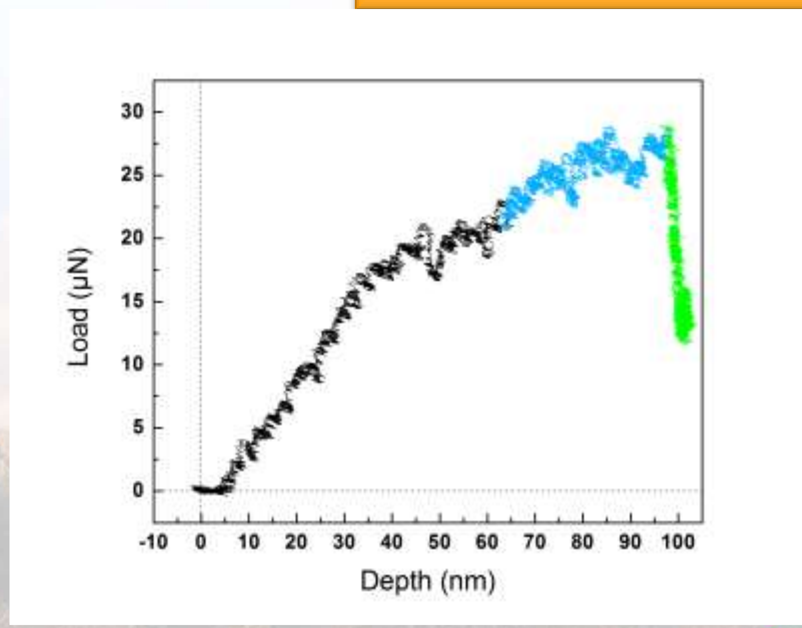


Range of Mechanical Testing Techniques

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

Fundamentals of Mechanical Properties

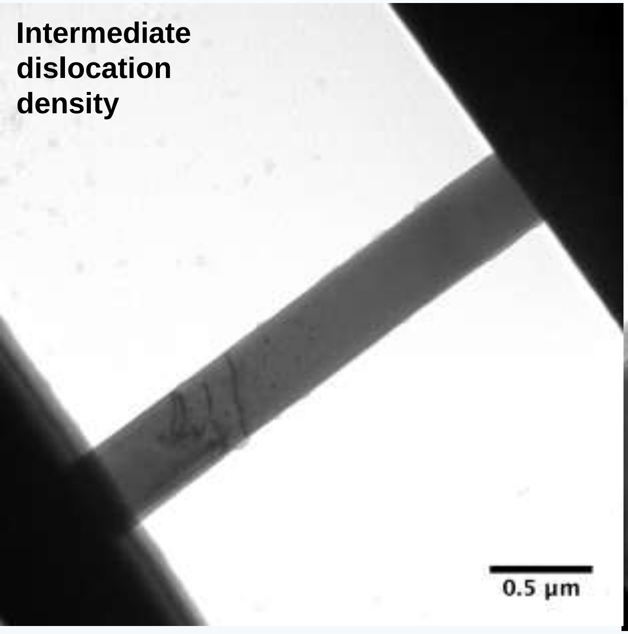


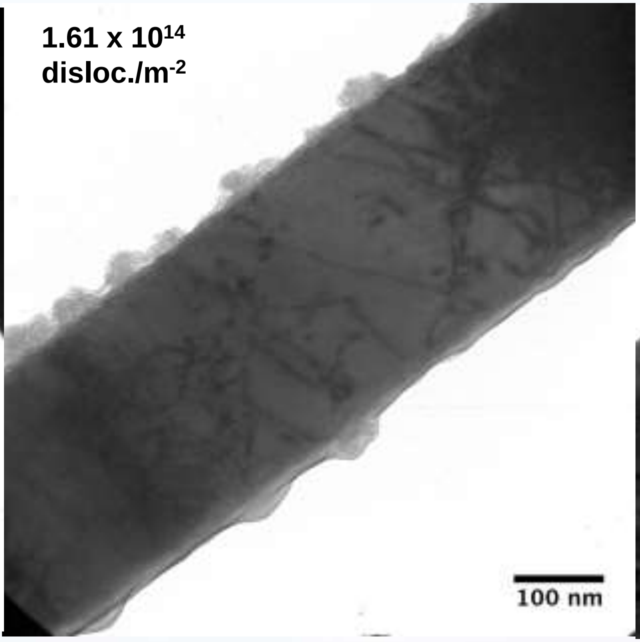


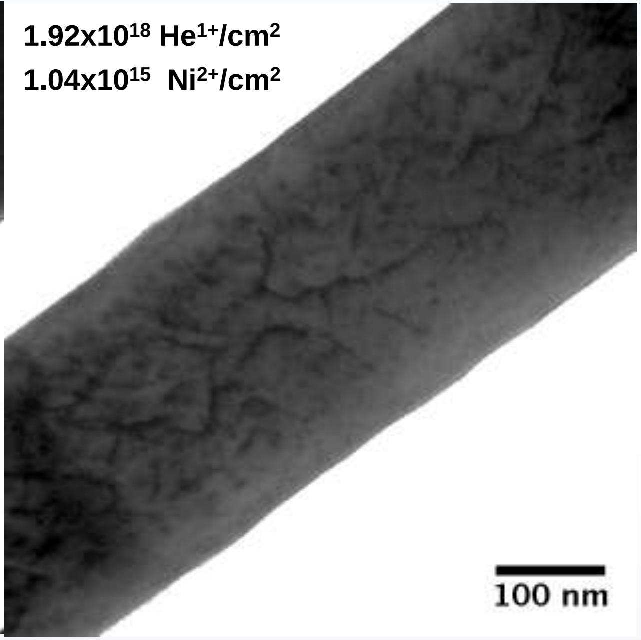
- 0.5 nm/s loading rate
- Trapezoid load function
- 60s load/60s hold/60s unload

Next Steps: *In situ* TEM Quantitative Mechanical Testing

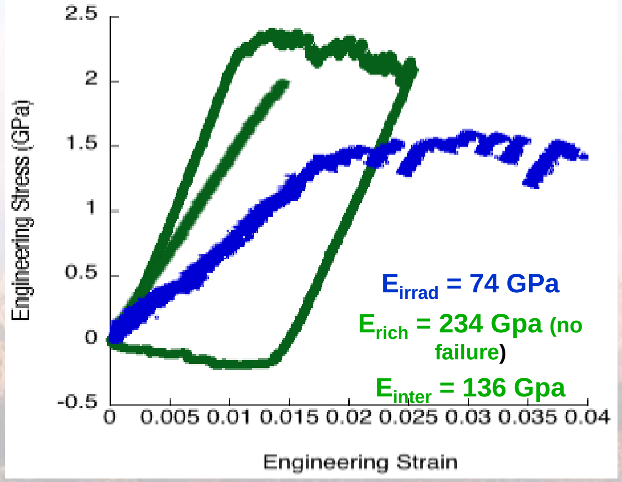
Contributors: C. Chisholm, H. Bei, E.P. George, P. Hosemann, & A.M. Minor





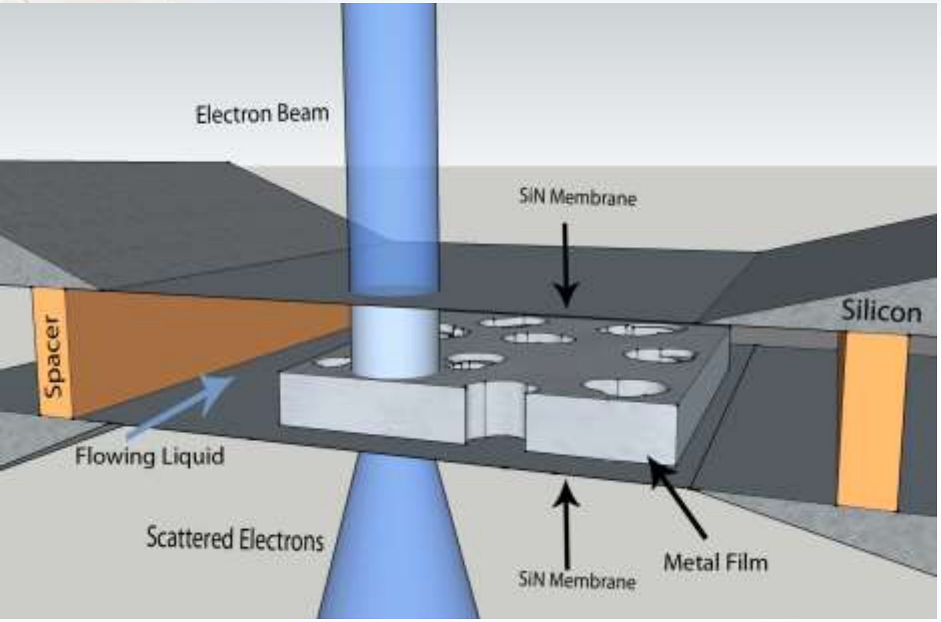


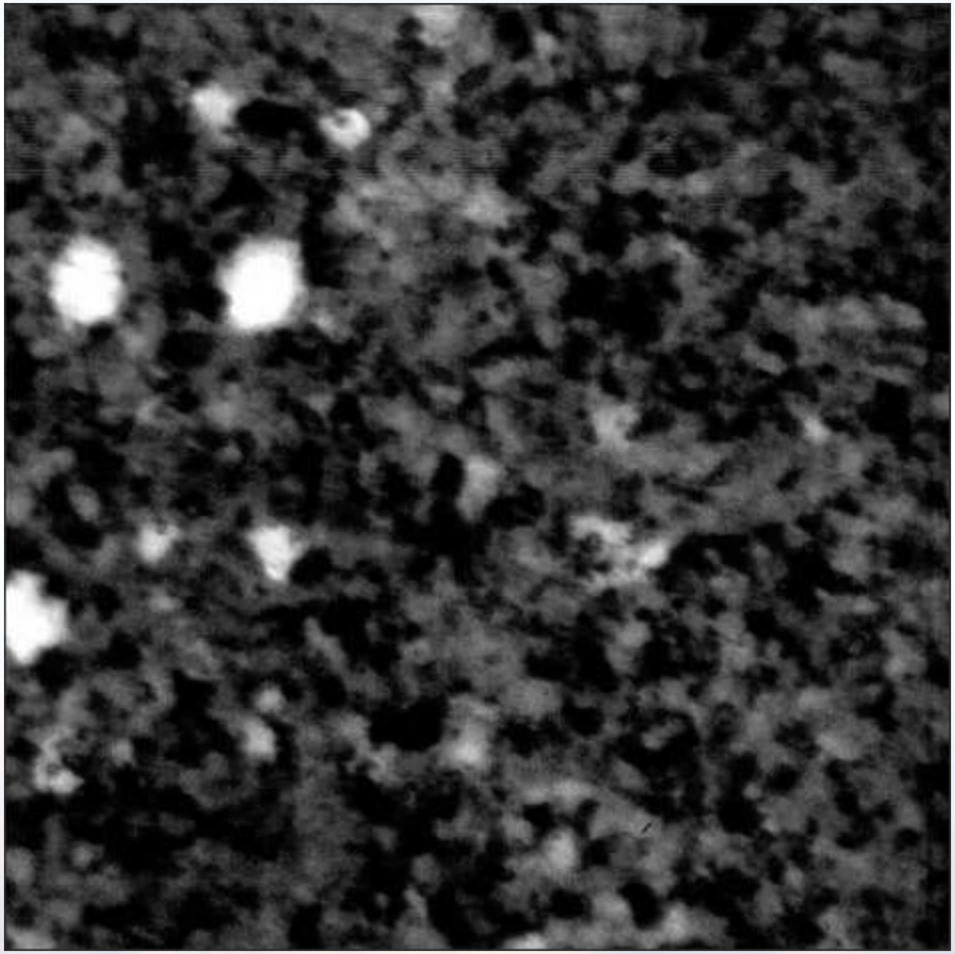
Work has started by looking sequentially at the quantitative effects of ion irradiation on mechanical properties utilizing in-situ ion irradiation TEM and in-situ TEM straining.



In situ TEM Corrosion

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





Microfluidic Stage

- Mixing of two or more channels
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Films can be directly deposited on the electron transparent SiN membrane

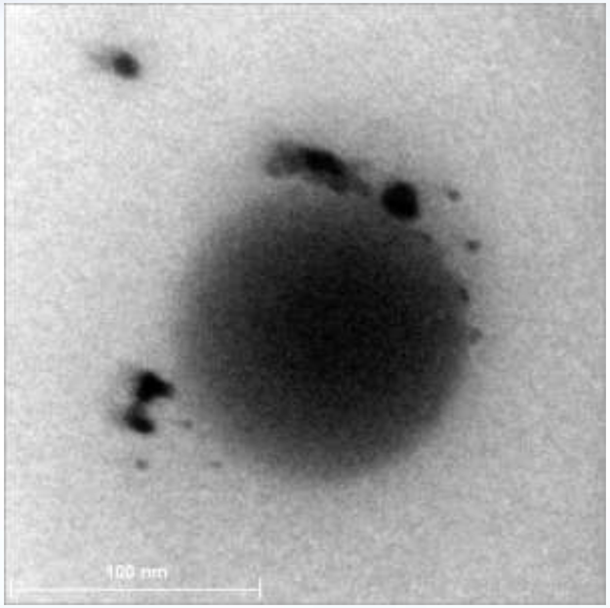
Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.

Other Fun Uses of Microfluidic Cell

Protocell Drug Delivery

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

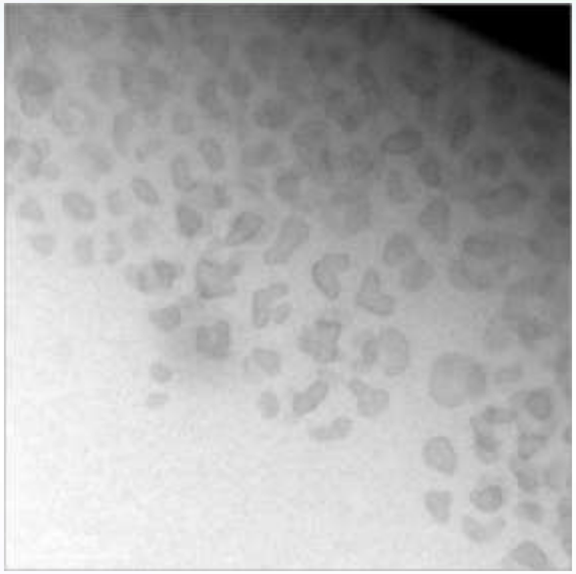
Liposome encapsulated Silica destroyed by the electron beam



BSA Crystallization

S.H. Pratt

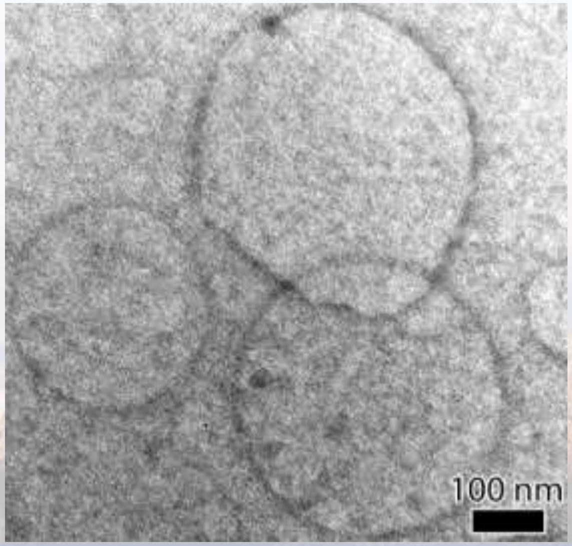
Crystallization of excess Bovine Serum Albumen during flow



Liposomes in Water

S.H. Pratt,
D. Sasaki

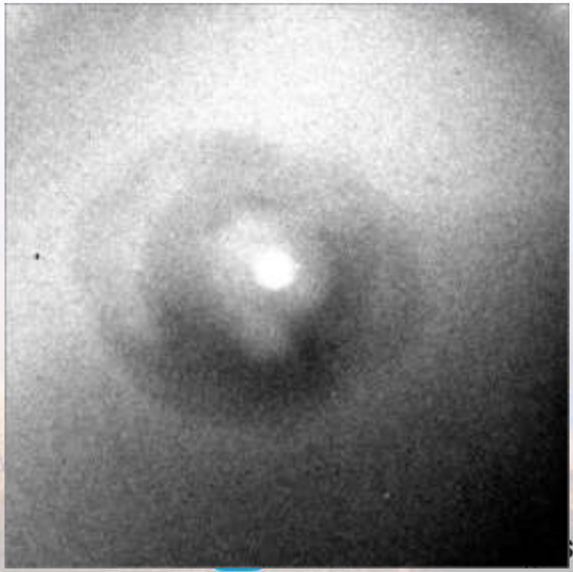
Liposomes imaged in flowing aqueous channel



La Structure Formation

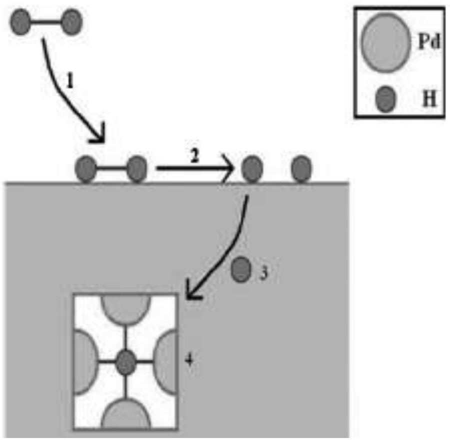
S.H. Pratt,
T. Nenoff

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

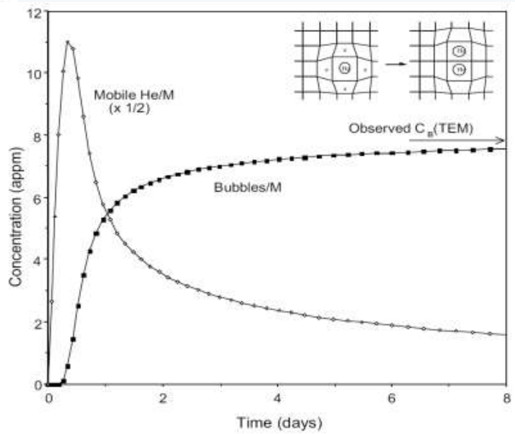


Can *In situ* TEM Address Hydrogen Storage Concerns in Extreme Environments?

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



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

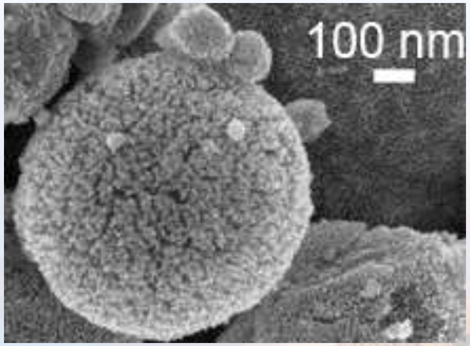


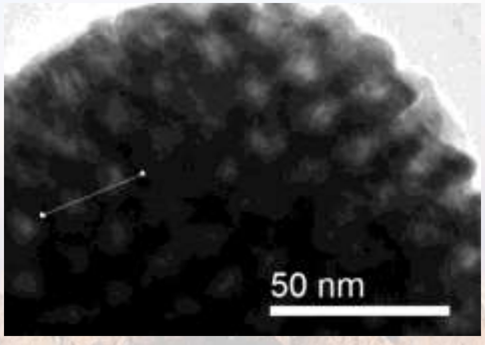
Cowgill, D., Fusion Sci. & Tech., 28 (2005) p. 539

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

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

Harmful effects may be mitigated in nanoporous Pd

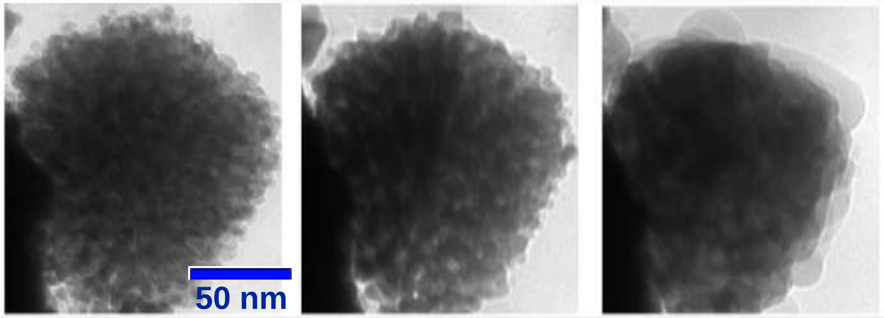




Vapor-Phase Heating TEM Stage

- 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.

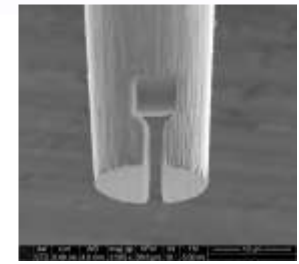


125° C 200° C 300° C

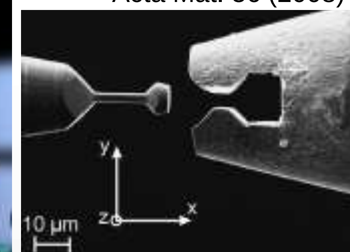
New *in situ* atmospheric heating experiments provide great insight into nanoporous Pd stability

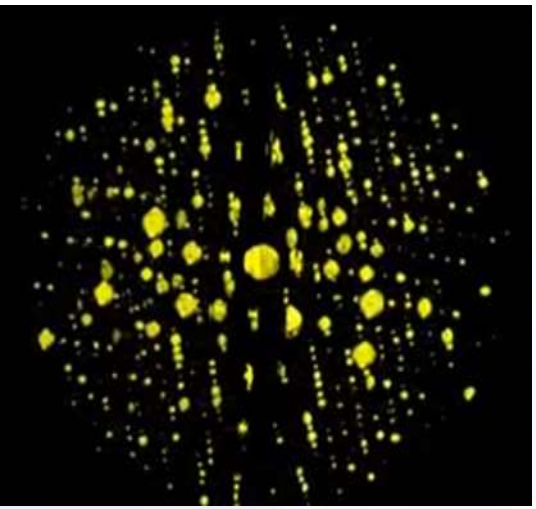
Future Directions Under Pursuit

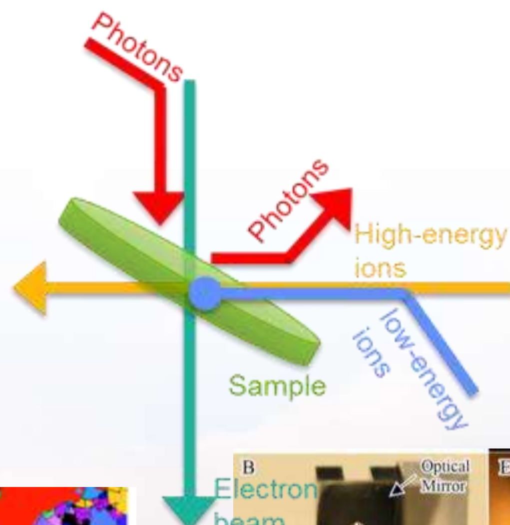
1. **Quantitative in-situ optical tensile/creep experiments (First experiments completed)**
2. **In-situ TEM CL, IBIL (currently capable)**
3. ***In situ* ion irradiation TEM in liquid or gas (currently capable)**
4. **PED: Local texture characterization (Install waiting on training)**
5. **DTEM: Nanosecond resolution (laser optics needed)**



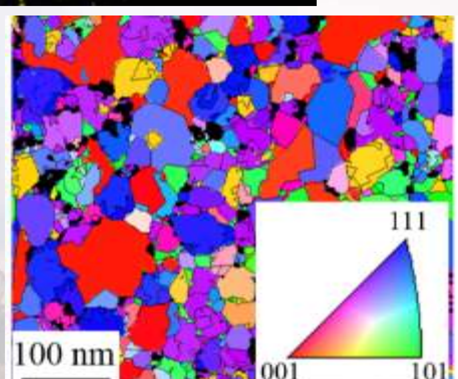
Kiener et al.
Acta Mat. 56 (2008)

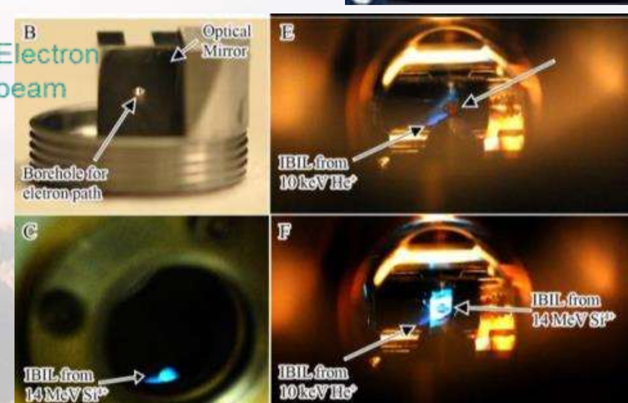




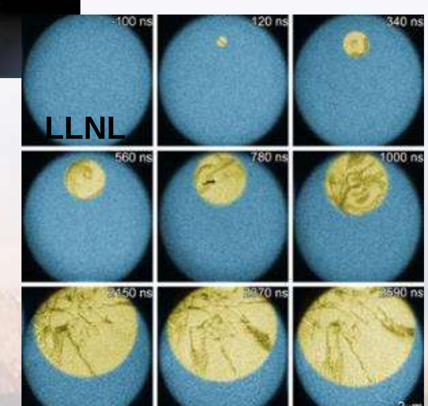


AppFive NanoMegas





LLNL



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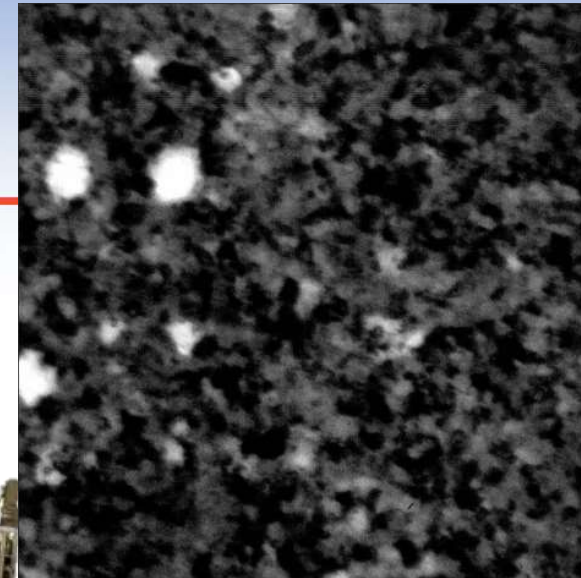
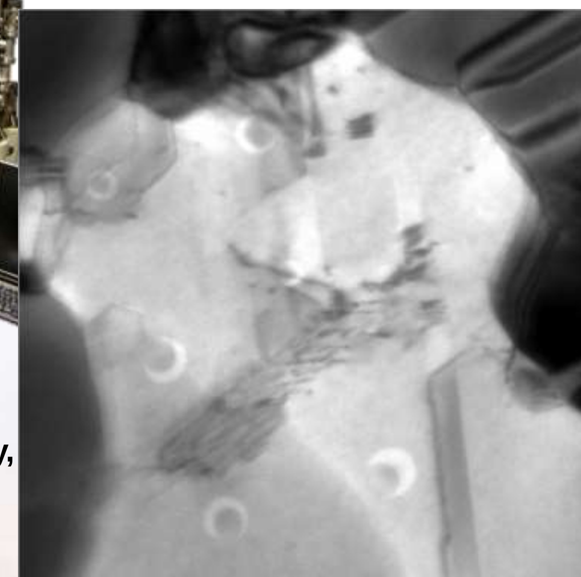
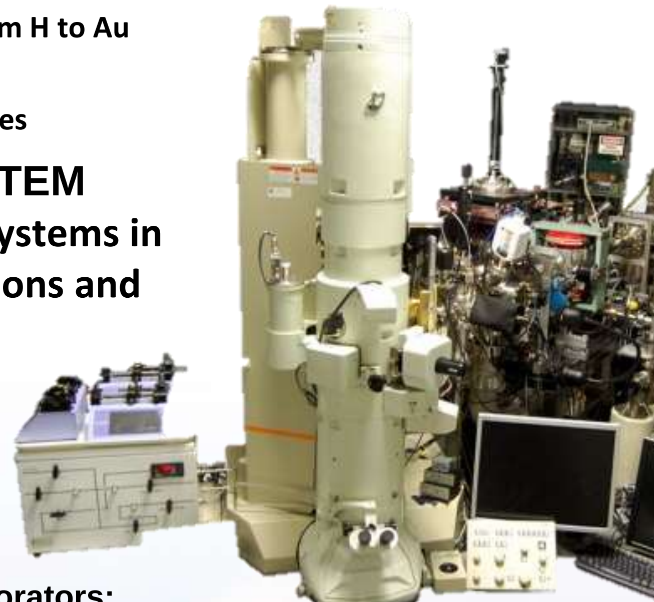
Summary





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



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

Collaborators:

- **IBL: D.C. Bufford, D. Buller, C. Chisholm, B.G. Clark, J. Villone, G. Vizkelethy, B.L. Doyle, S. H. Pratt, & M.T. Marshall**
- **Sandia: B. Boyce, T.J. Boyle, P.J. Cappillino, J.A. Scott, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, E. Carnes, J. Brinker, D. Sasaki, J.A. Sharon, T. Nenoff, W.M. Mook, P. Feng, F.P. Doty, B.A. Hernandez-Sanchez, P. Yang, J-E Mogonye, S.V. Prasad , P. Kotula, S. Howell, T. Ohta, & T. Beechem**
- **External: A. Minor, L.R. Parent, I. Arslan, H. Bei, E.P. George, P. Hosemann, D. Gross, J. Kacher, & I.M. Robertson**

U.S. DEPARTMENT OF ENERGY | Office of Science

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