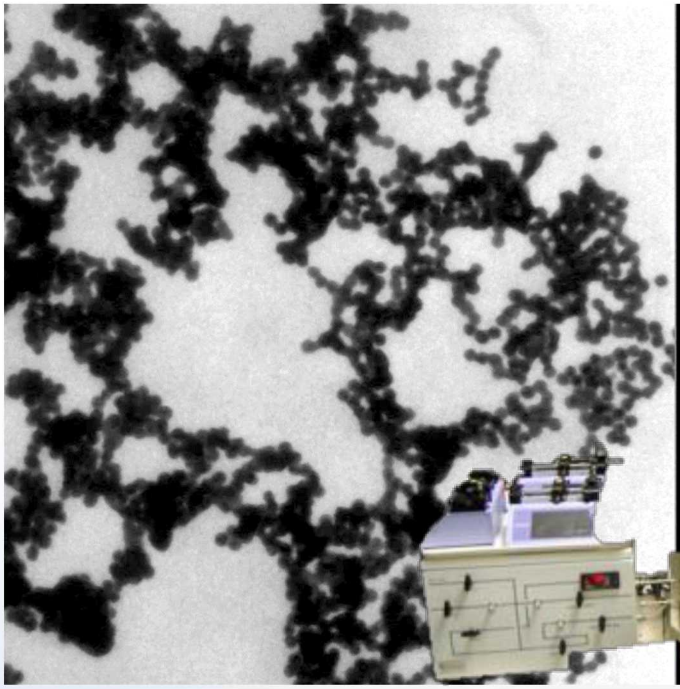
Exploring Extreme Environments with Nanometer Resolution

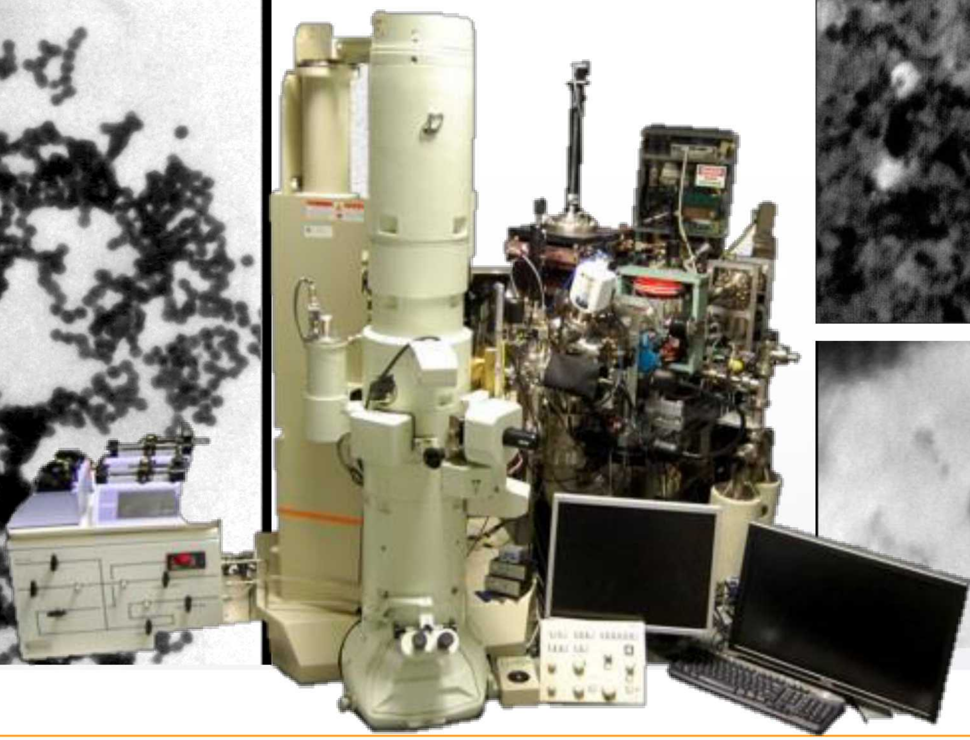
SAND2018-5405PE

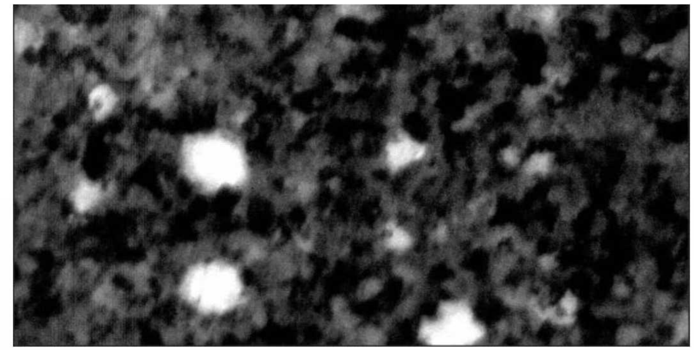
K. Hattar

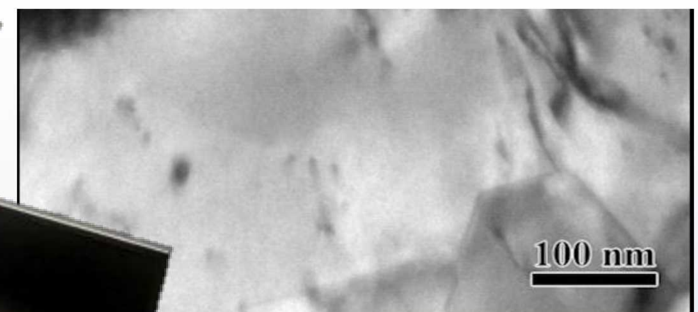
Sandia National Laboratories

March 23rd, 2018









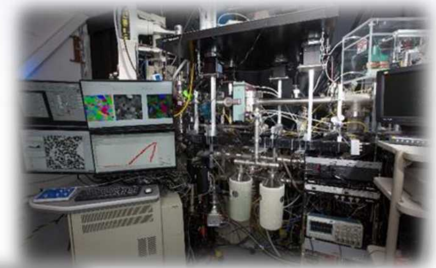
Collaborators:

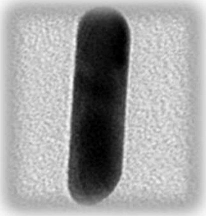
- D.L. Buller, D.C. Bufford, S.H. Pratt, T.J. Boyle, B.A. Hernandez-Sanchez, S.J. Blair, B. Muntifering, C. Chisholm , P. Hosemann, A. Minor, J. A. Hinks, F. Hibberd, A. Ilinov, D. C. Bufford, F. Djurabekova, G. Greaves, A. Kuronen, S. E. Donnelly, K. Nordlund, F. Abdeljawad, S.M. Foiles, J. Qu, C. Taylor, J. Sugar, P. Price, C.M. Barr, D. Adams, M. Abere, L. Treadwell, A. Cook, A. Monterrosa, IDES Inc, J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, W. Mook, Hysitron Inc., G.S. Jawaharram, S. Dillon, R.S. Averback, N. Heckman, J. Carroll, S. Briggs, E. Carnes, J. Brinker, D. Sassaki, T. Nenoff, B.G. Clark, P.J. Cappillino, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, L.R. Parent, I. Arslan. & Protochips, Inc.

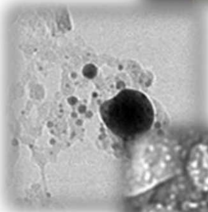
This work was partially funded by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. DOE's National Nuclear Security Administration under contract DE-NA-0003525. The views expressed in the article do not necessarily represent the views of the U.S. DOE or the United States Government.

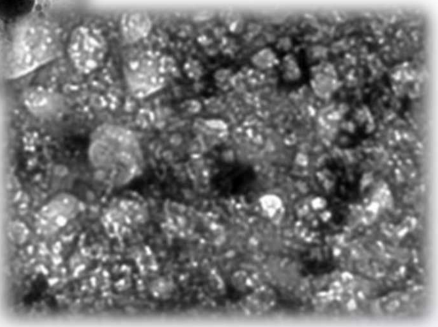
Outline

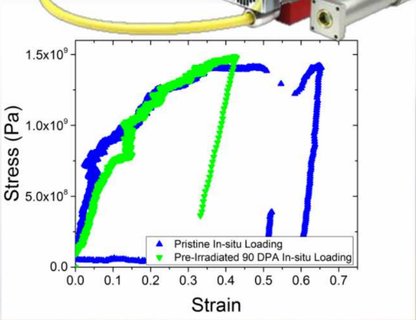
1. Introduction to Sandia National Laboratories and its *In situ* Ion Radiation Transmission Electron Microscope (I^3TEM)
2. Understanding radiation stability in nanoparticles
3. Effect of radiation environments on nanocrystalline metals
4. Lasers in a TEM (why not?)
5. State-of-art in quantitative in situ mechanical testing
6. Other environments (in situ SEM, liquid, and gas)

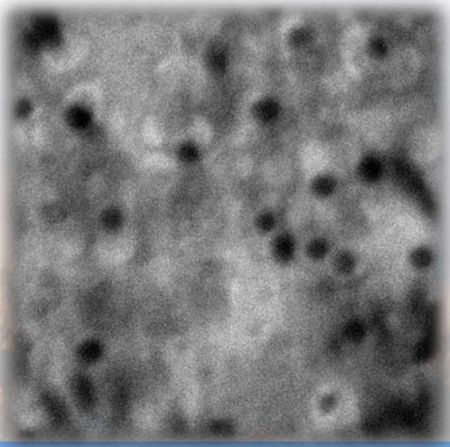










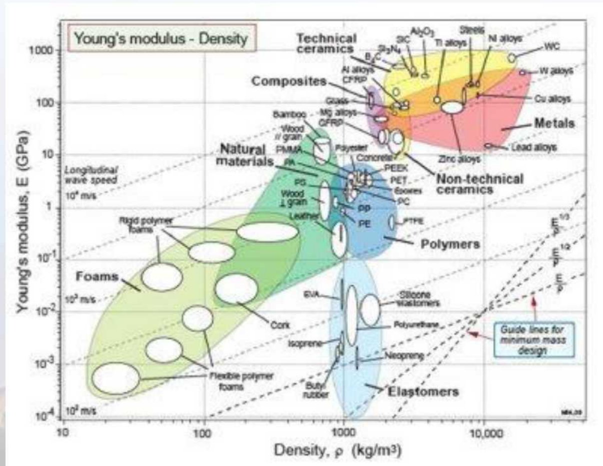


Potential Evolution of System Design

Use the Nearest Stone

to

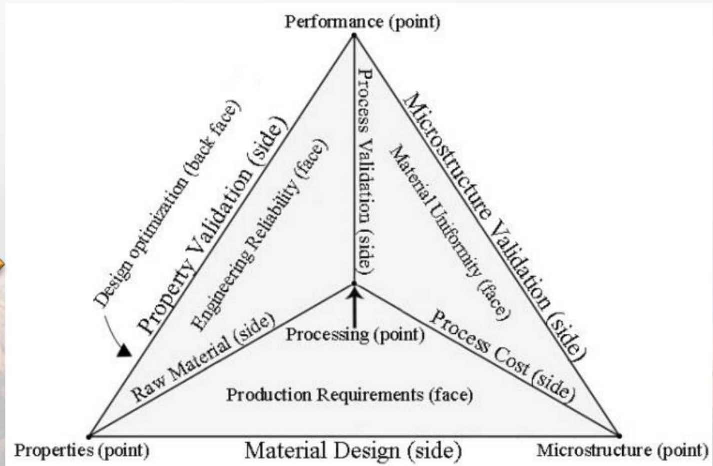
- Radar charts and Ashby plots of current material
- Accelerated and field testing
- **Scientist create a new materials. Engineers find an application.**



Materials by Design

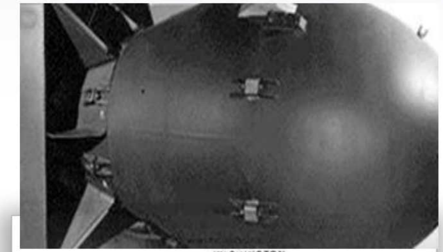
- **Physics-based approach**
- **Requires multiscale modeling**
- **Engineers require given properties, Scientists tailor the chemistry and microstructure to achieve it.**

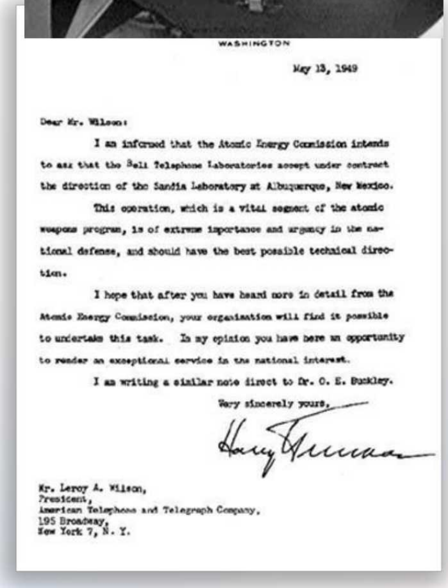
Great vision! We are making strides, but we are not there yet



Sandia National Laboratori<u>es</u>

"Exceptional service in the national interest"





WASHINGTON

May 13, 1949

Dear Mr. Wilson:

I am informed that the Atomic Energy Commission intends to ask that the Bell Telephone Laboratories accept under contract the direction of the Sandia Laboratory at Albuquerque, New Mexico.

This operation, which is a vital segment of the atomic weapons program, is of extreme importance and urgency in the national defense, and should have the best possible technical direction.

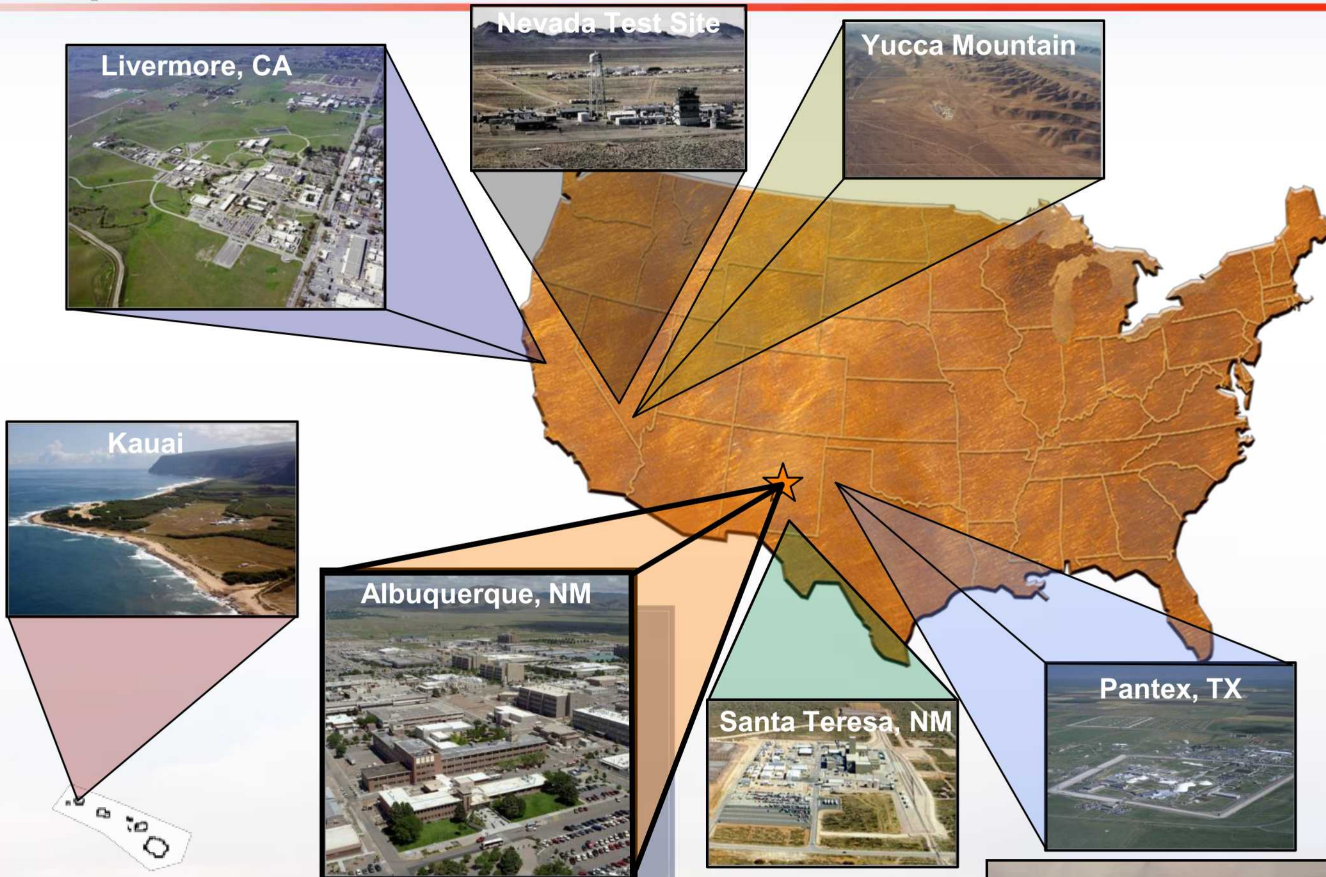
I hope that after you have heard more in detail from the Atomic Energy Commission, your organization will find it possible to undertake this task. In my opinion you have here an opportunity to render an exceptional service in the national interest.

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 develops advanced technologies to ensure global peace." – S. Younger

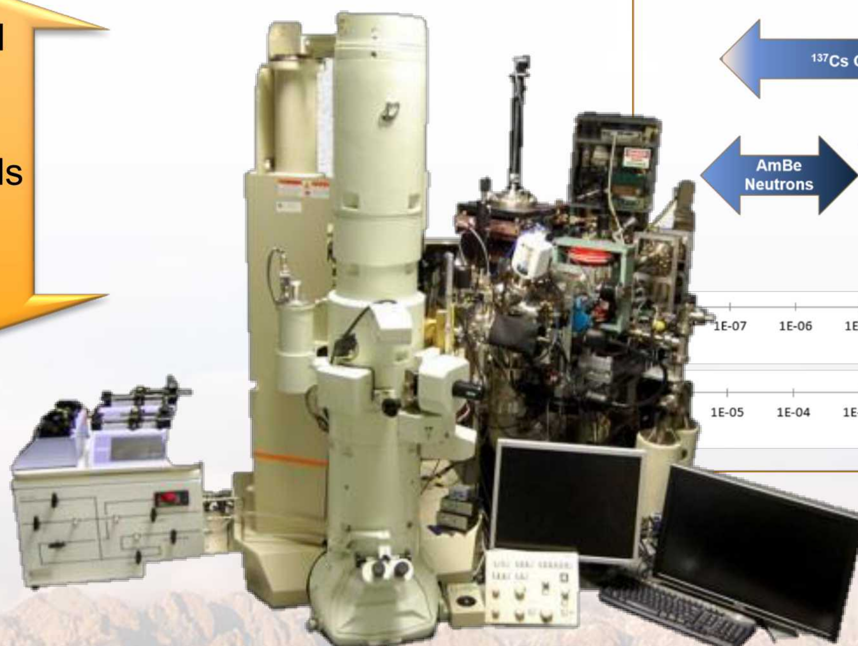
Sandia National Laboratories

Sandia's USER Capabilities

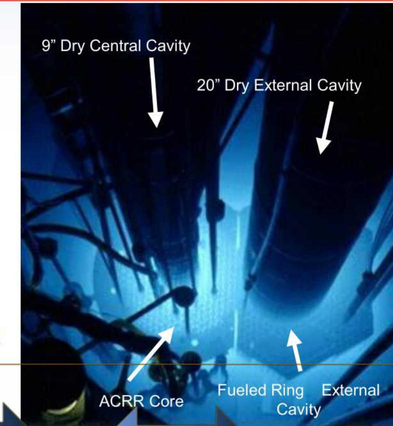
Core Facility - SNL

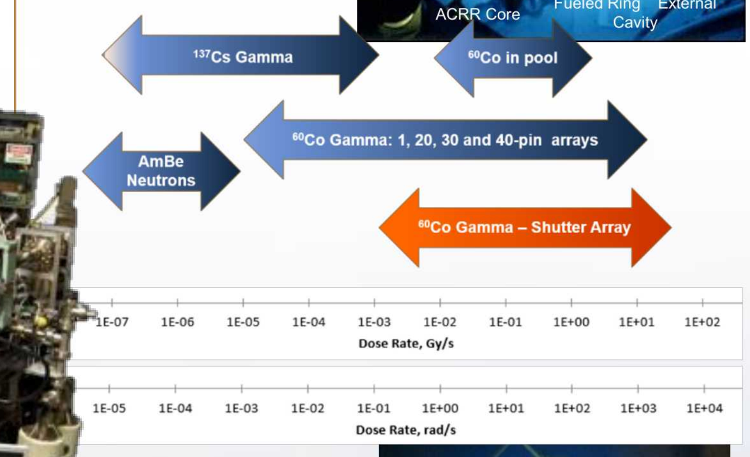
- Nanophotonics & Optical Nanomaterials
- Soft- Biological & Composite Nanomaterials
- Quantum Materials
- In-situ Characterization and Nanomechanics

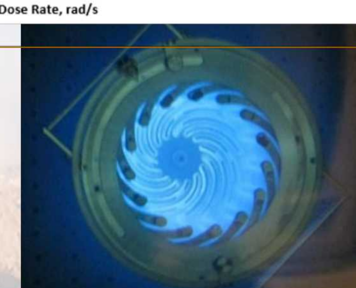
Gateway Facility - LANL



D. Hanson, W. Martin, M. Wasiolek





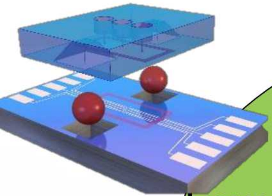


Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

Sandia National Laboratories

In situ Characterization and Nanomechanics

Developing and implementing world-leading capabilities to study the dynamic response of materials and nanosystems to mechanical, electrical, or other stimuli.

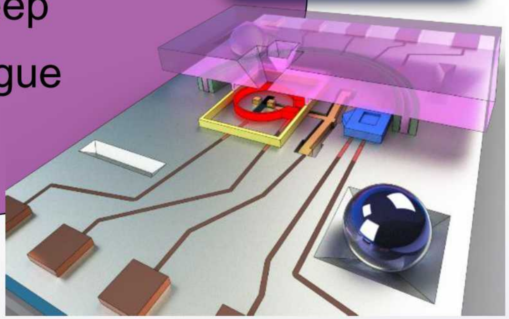


In-Situ Characterization

- Optical Microscopy
- Low-Energy Electron Microscopy
- Scanning Electron Microscopy
- Transmission Electron Microscopy

In-Situ Nanomechanics

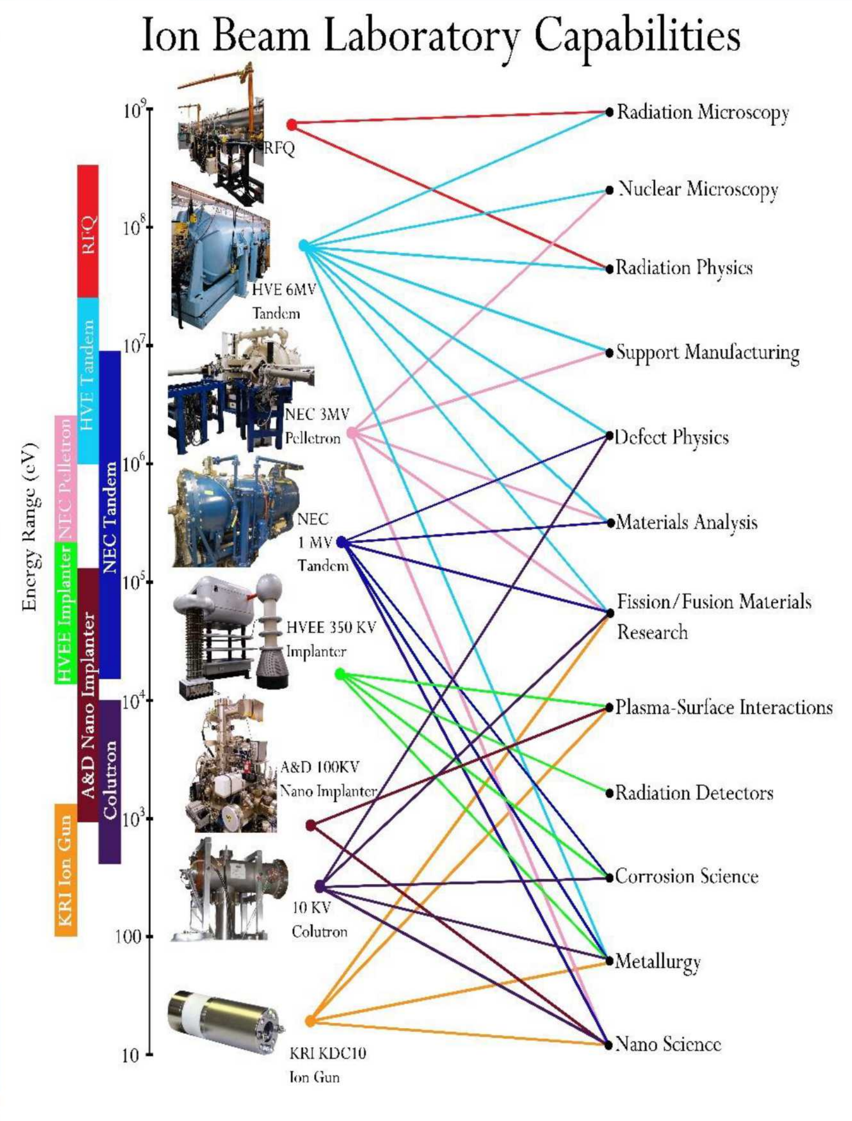
- Indendation
- Monotonic Loading
- Creep
- Fatigue



Scientific challenges

- How do defects and crystal distortions alter the mechanical and other extrinsic properties in nanostructured materials?
- How can we understand and control energy transfer across interfaces and over multiple length and time scales?
- How does the environment change the mechanical response and surface structure of nanoscale materials?

Sandia's Ion Beam Laboratory

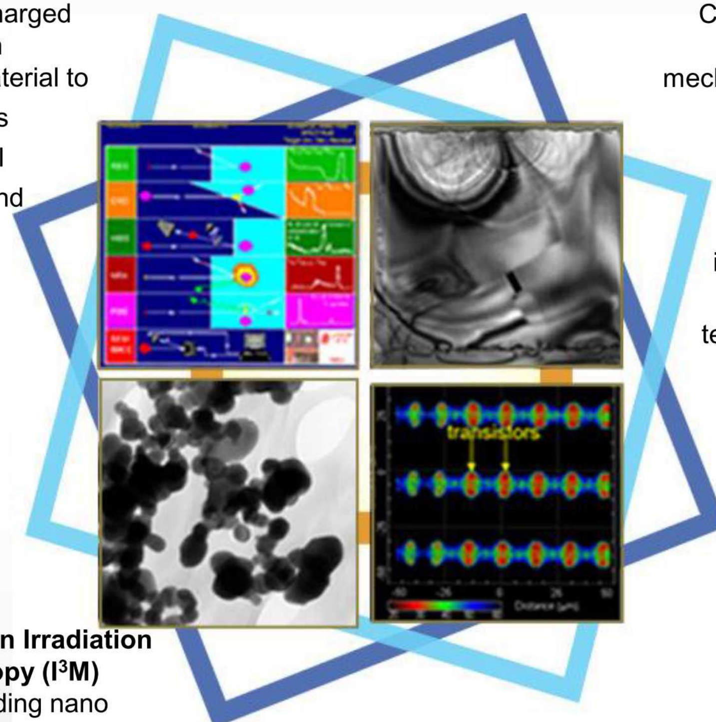


Ion Beam Analysis (IBA)

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 ion beam capability 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

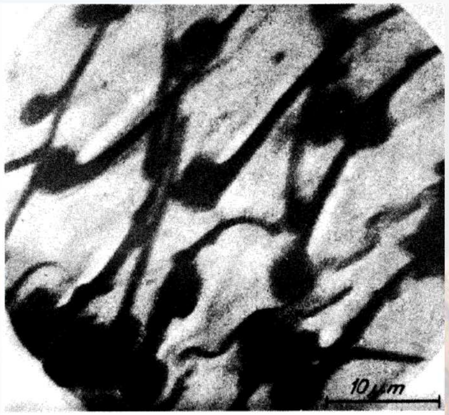


Fig. 6: Wing surface of the house fly.
(First internal photography, U = 60 kV, M_{el} = 2200)
(Driest, E., and Müller, H.O.: Z. Wiss. Mikroskopie 52, 53-57 (1935)

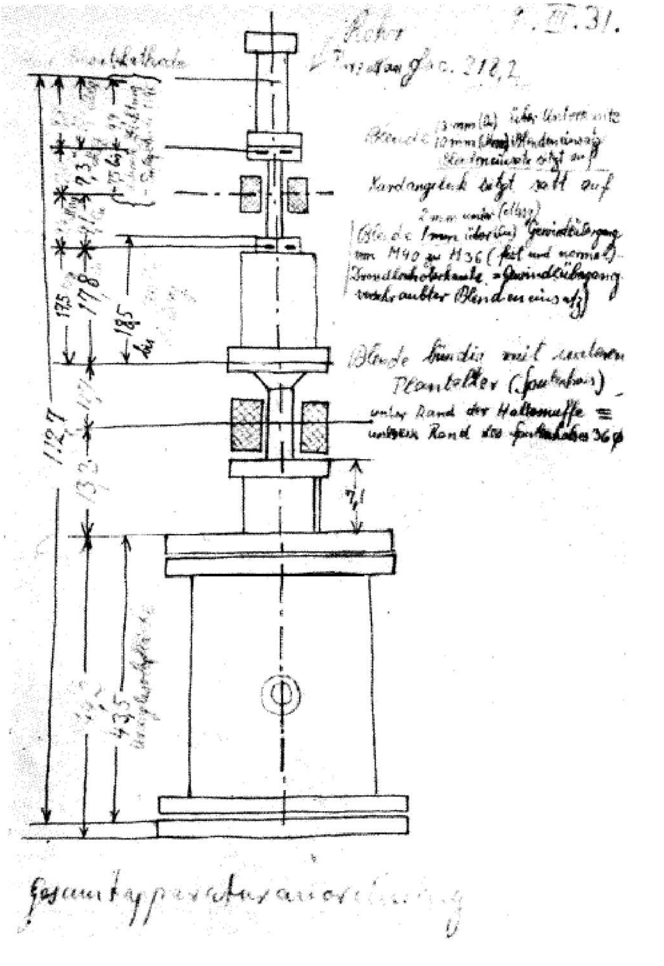


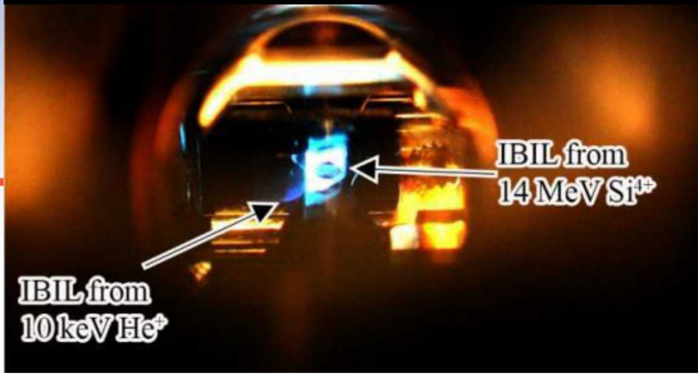
Fig. 2: Sketch by the author (9 March 1931) of the cathode ray tube for testing one-stage and two-stage electron-optical imaging by means of two magnetic electron lenses (electron microscope) [8].

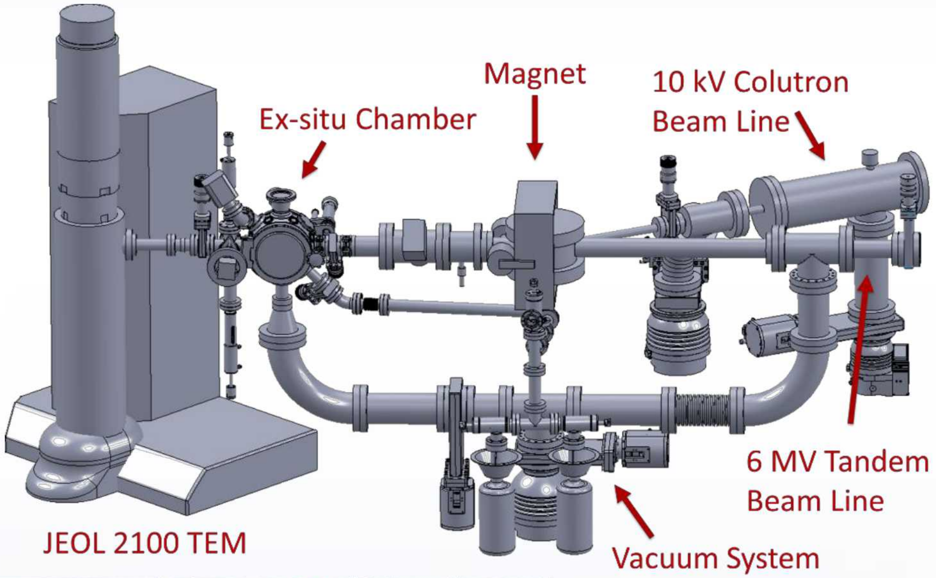
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

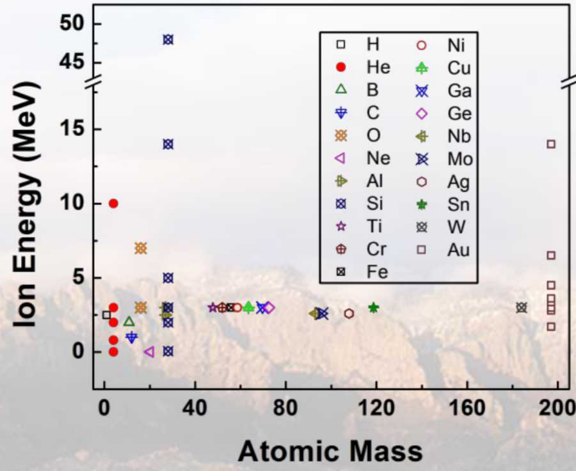
Collaborator: D.L. Buller

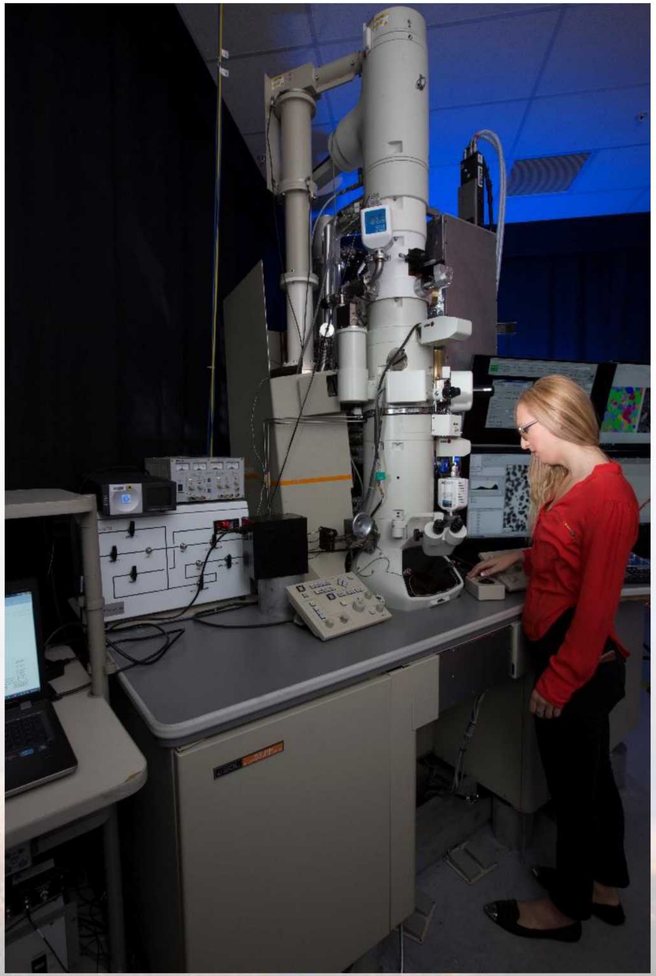
10 kV Colutron - 200 kV TEM - 6 MV Tandem

IBIL from a quartz stage inside the TEM



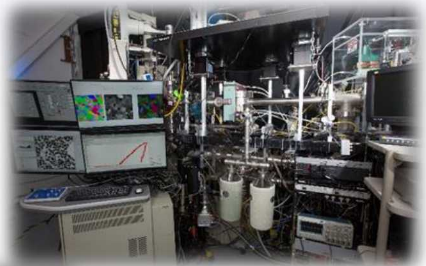


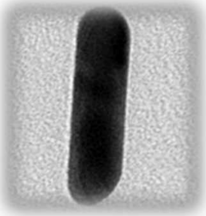


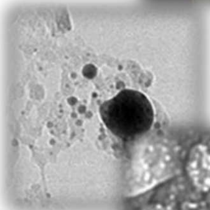


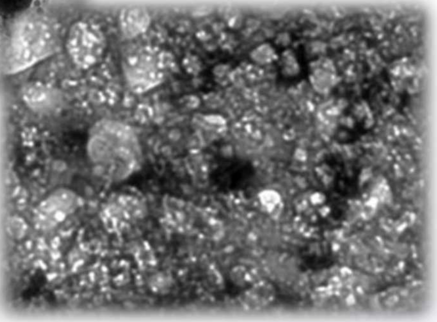
Outline

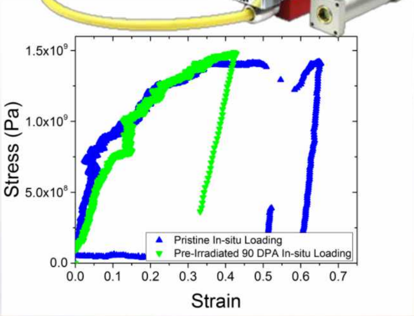
1. Introduction to Sandia National Laboratories and its *In situ* Ion Radiation Transmission Electron Microscope (I^3TEM)
2. Understanding radiation stability in nanoparticles
3. Effect of radiation environments on nanocrystalline metals
4. Lasers in a TEM (why not?)
5. State-of-art in quantitative in situ mechanical testing
6. Other environments
(in situ SEM, liquid, and gas)

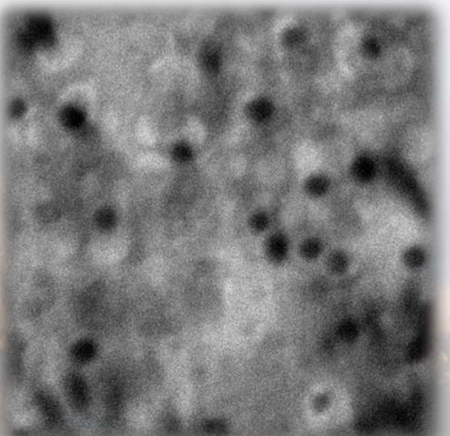






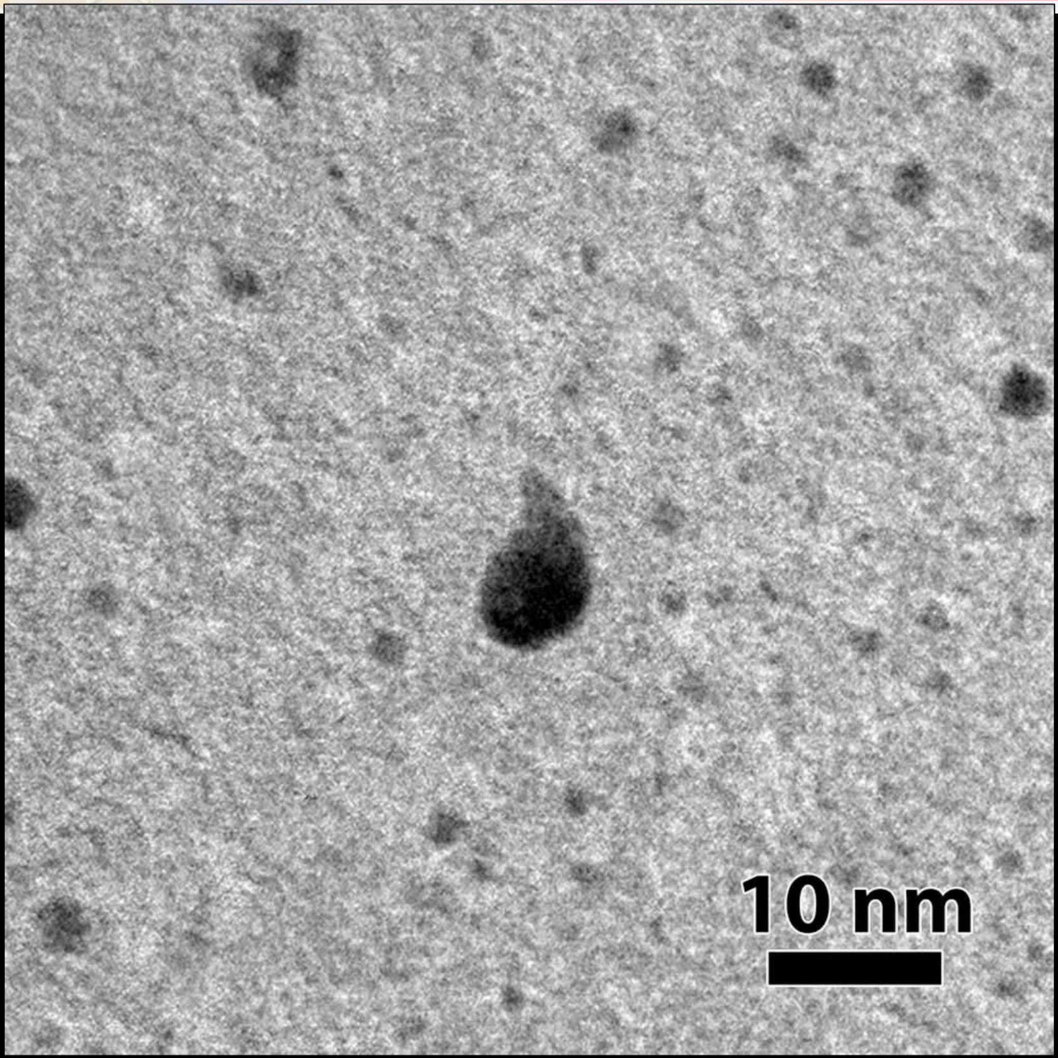


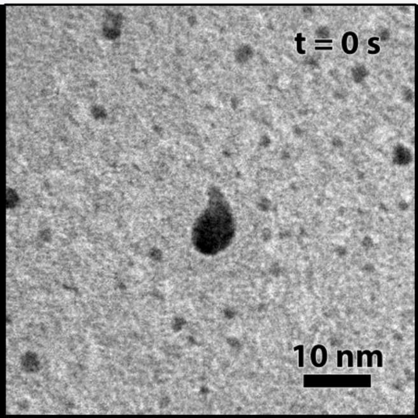


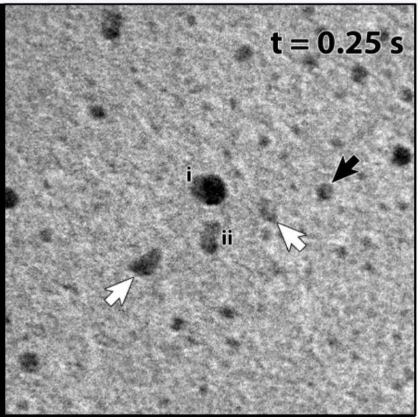


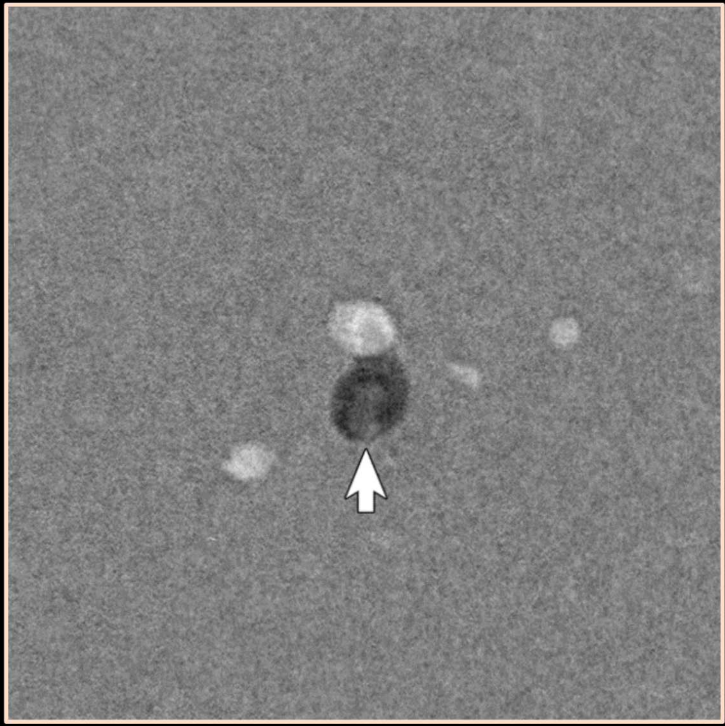
Single Ion Strikes:
46 keV Au^{1-} ions into 5 nm Au nanoparticles

Collaborator: D.C. Bufford



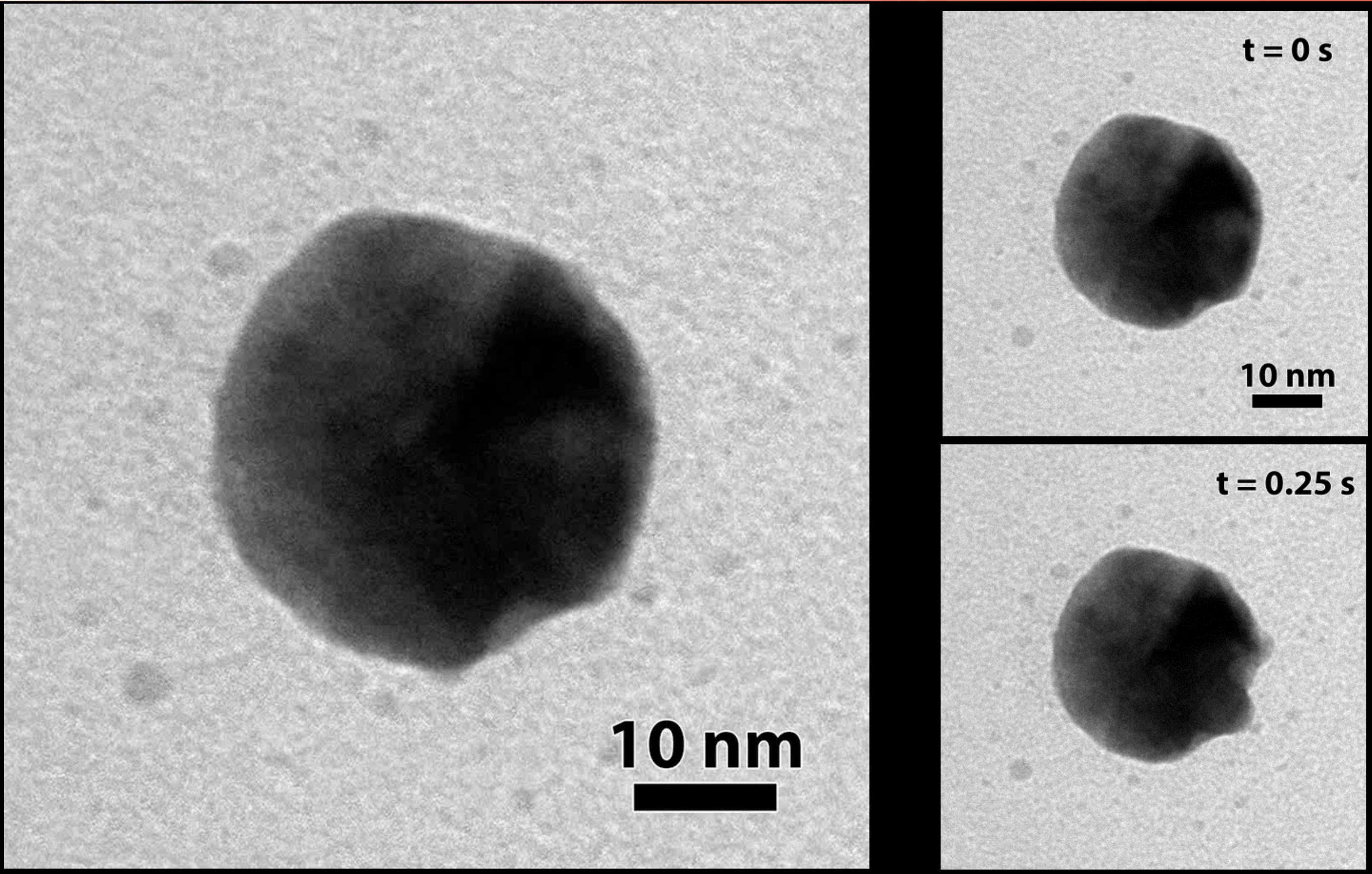






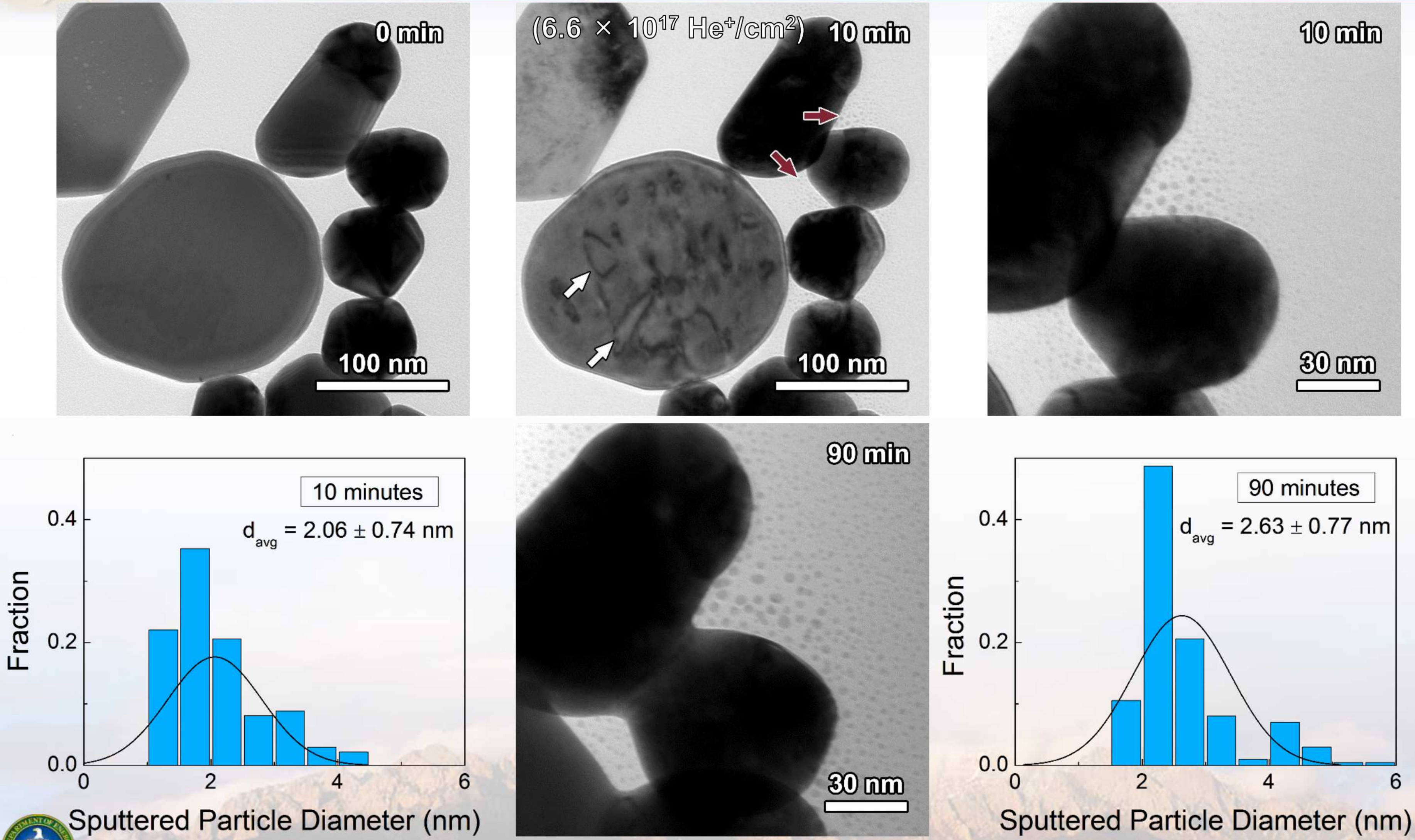
Single Ion Strikes: 46 keV Au^{1-} ions into 20 nm NPs

Collaborator: D.C. Bufford



Formation of Dislocation Loops & Sputtered Particles due to He implantation

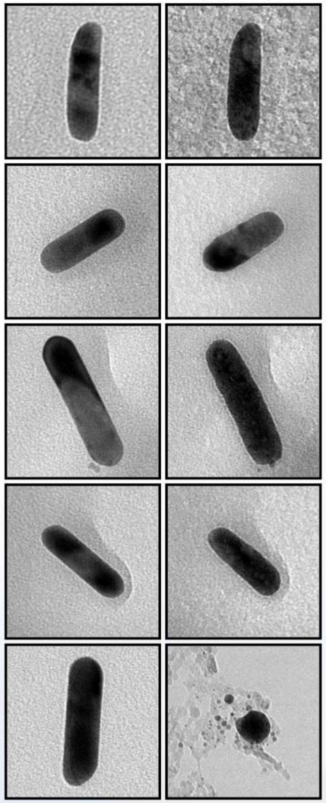
Collaborators: D.C. Bufford, S.H. Pratt & T.J. Boyle

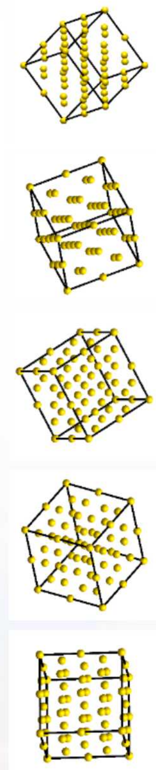


Sandia National Laboratories

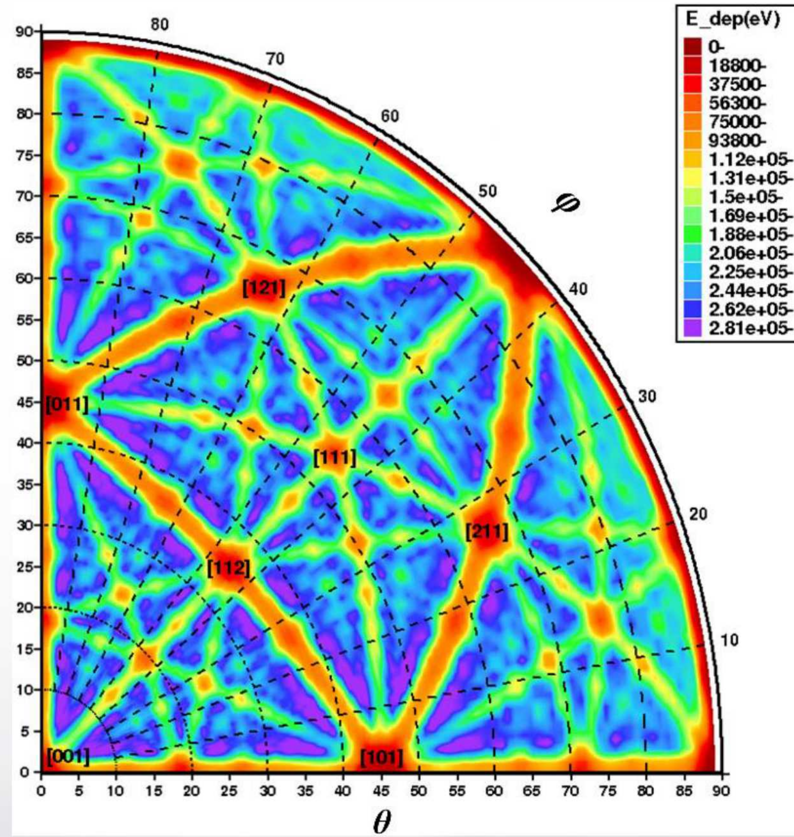
Exploring Radiation effects in Au Nanorods

Collaborators: J. A. Hinks, F. Hibberd, A. Ilinov, D. C. Bufford, F. Djurabekova, G. Greaves, A. Kuronen, S. E. Donnelly & K. Nordlund





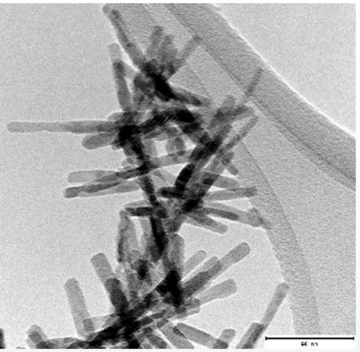
	Fluence (ions.cm^{-2}) 0	Fluence (ions.cm^{-2}) 3.6×10^{15}	Sputtering ΔV (%)	Sputtering Atoms.ion^{-1}	Irradiation orientation Model view	θ	φ
AuNR-1			+8.1%	–2.1		39.1°	21.9°
AuNR-2			+3.5%	–1.0		42.8°	13.7°
AuNR-3			+1.5%	–0.3		25.6°	30.1°
AuNR-4			–1.2%	0.3		59.6°	46.0°
AuNR-5			–89.7%	30.1		18.5°	75.4°



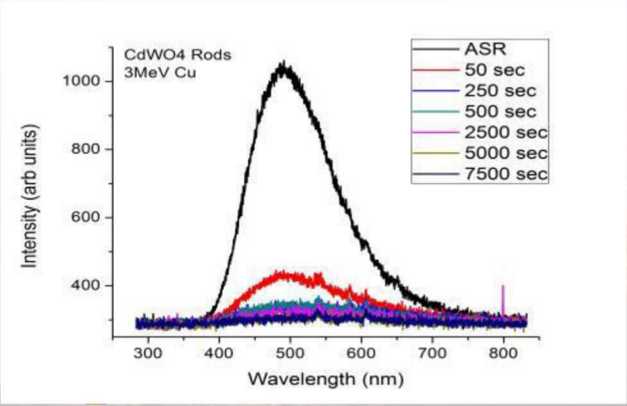
Crystal Orientation Matters!

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

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



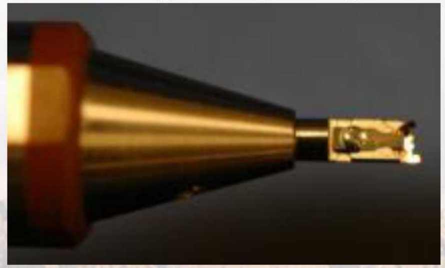
High-Z nanoparticles ($CdWO_4$) are promising, but are radiation sensitive



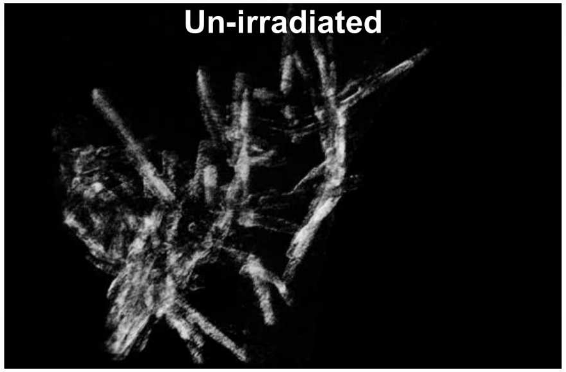
In situ **Ion Irradiation TEM (I^3TEM)**

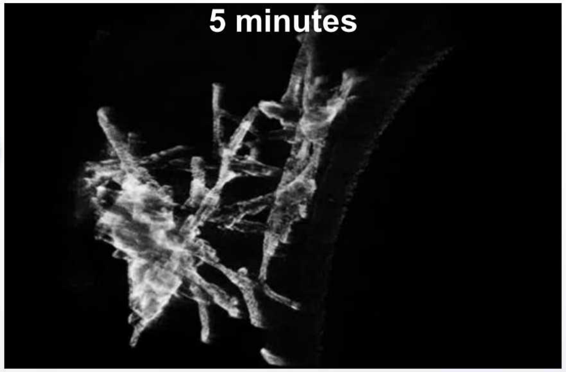
+

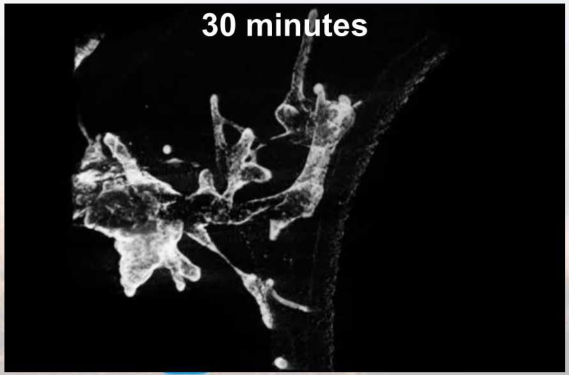
Hummingbird tomography stage



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

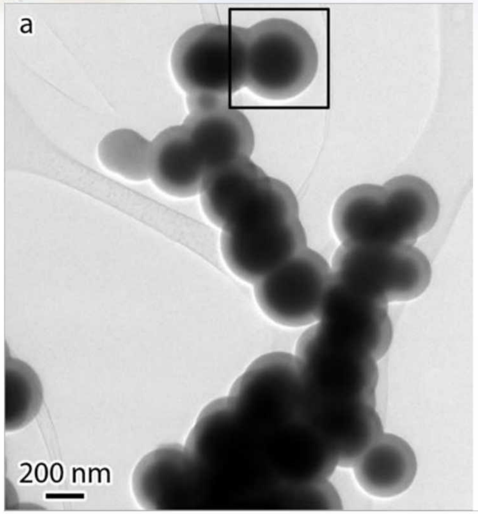


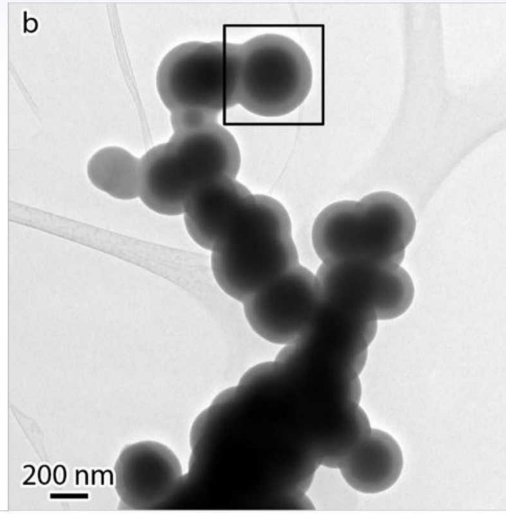


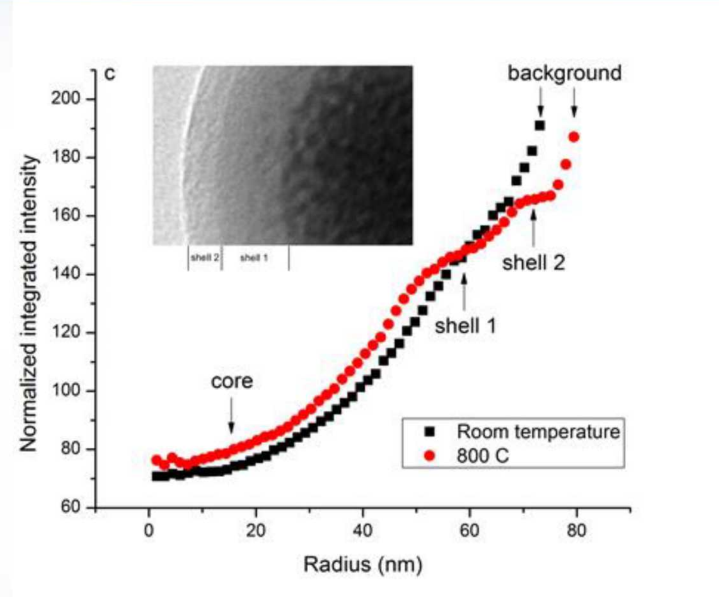


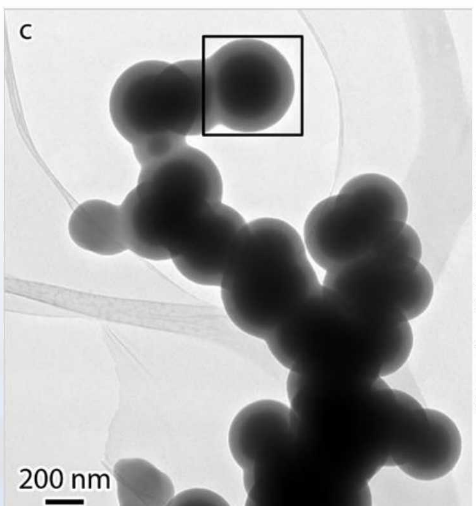
Radiation Stable Nanoparticles!

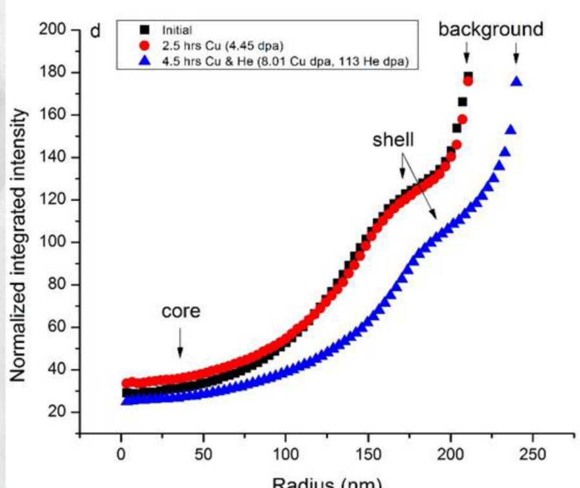
Collaborators: T.J. Boyle, S.J. Blair, B. Muntifering









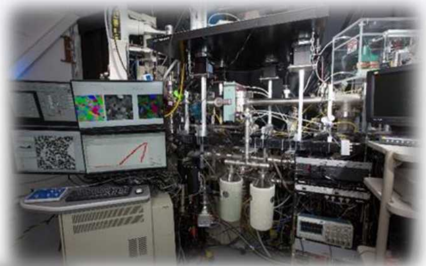


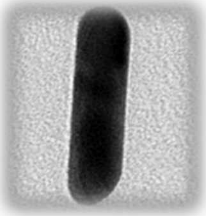
Ion	Energy (keV)	Dose (ions/cm^2) Dose rate (ions/cm^2/s)	Damage (dpa) Damage rate (dpa/s)
Cu	3000	1.2×10^{14} 1.3×10^{10}	4.45 4.9×10^{-4}
He & Cu	10 3000	2.3×10^{18} 1.4×10^{14} 2.2×10^{14} 1.3×10^{10}	113 7.0×10^{-3} 8.01 4.9×10^{-4}

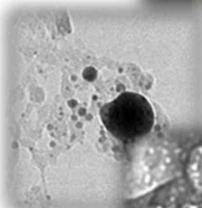
In contrast some NPs appear to be very radiation stabile

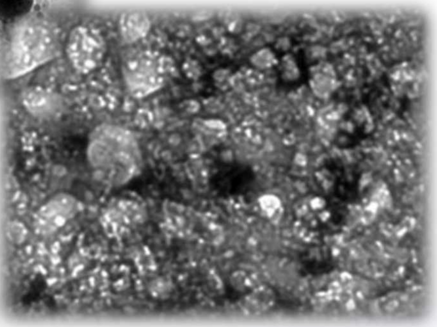
Outline

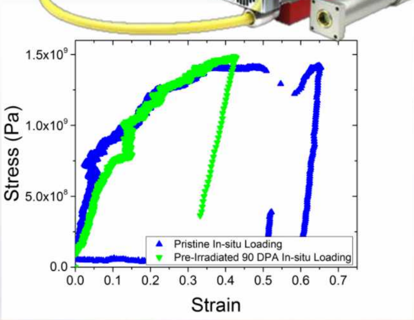
1. Introduction to Sandia National Laboratories and its *In situ* Ion Radiation Transmission Electron Microscope (I^3TEM)
2. Understanding radiation stability in nanoparticles
3. Effect of radiation environments on nanocrystalline metals
4. Lasers in a TEM (why not?)
5. State-of-art in quantitative in situ mechanical testing
6. Other environments (in situ SEM, liquid, and gas)

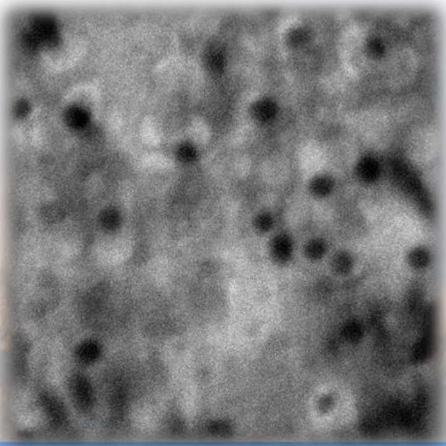








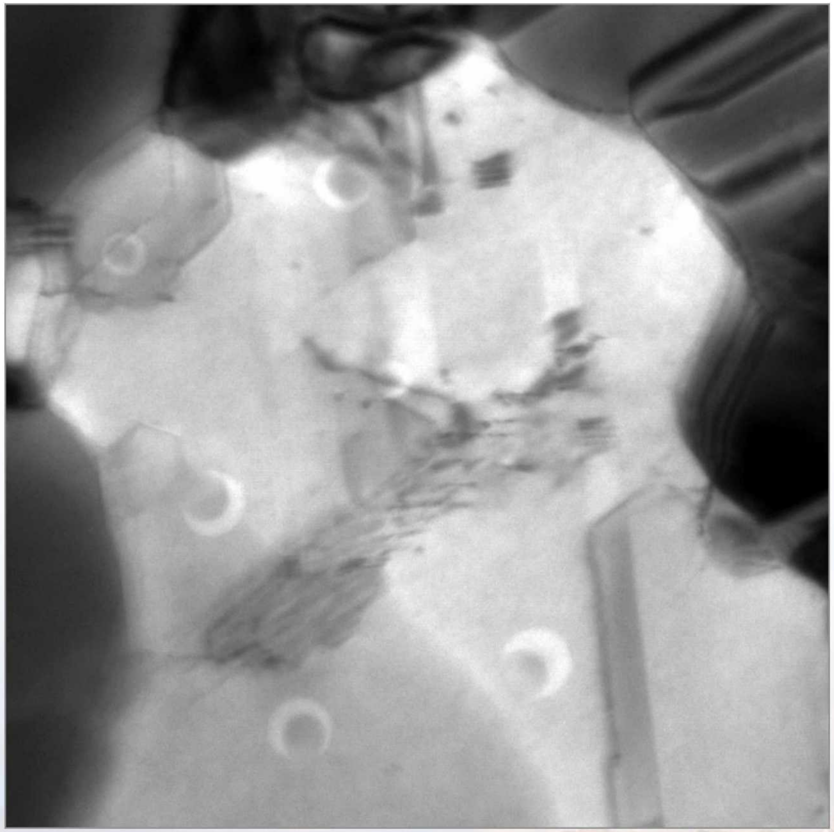




Dose Rate Effects in Nanocrystalline Metals

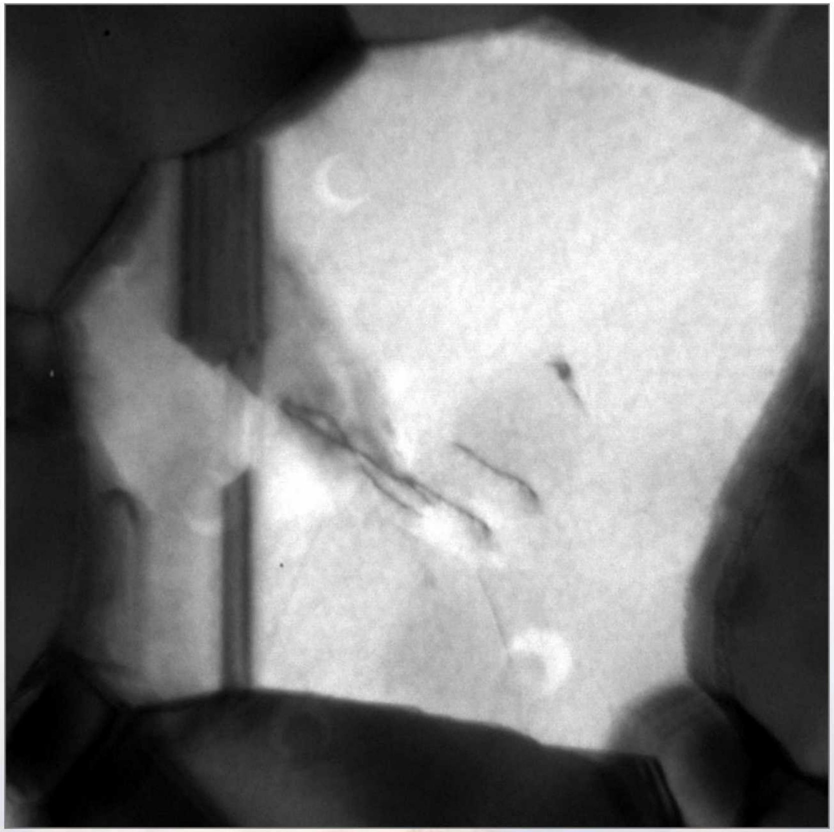
Collaborators: C. Chisholm , P. Hosemann, & A. Minor

7.9×10^9 ions/cm^2/s



vs

6.7×10^7 ions/cm^2/s

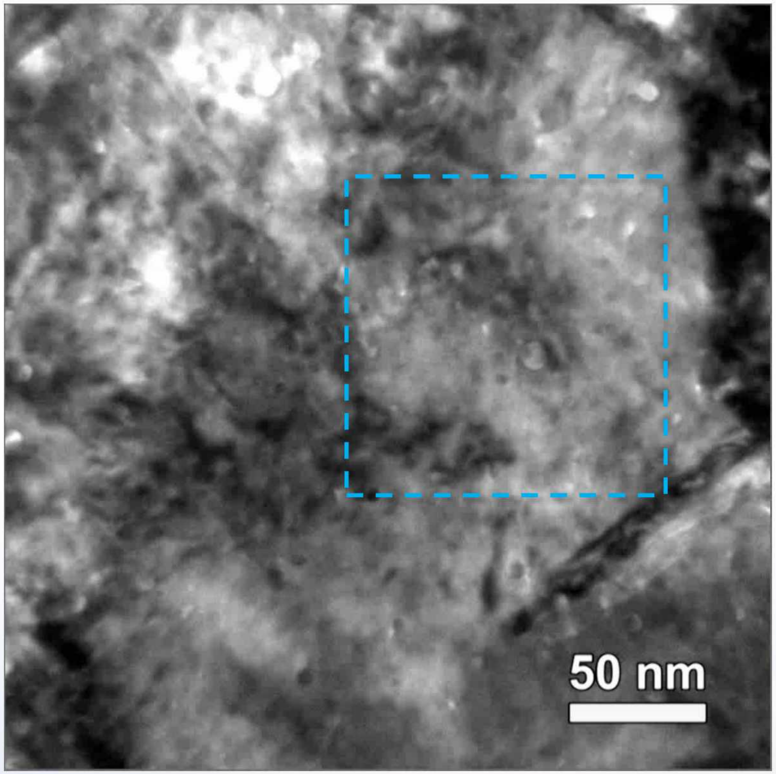


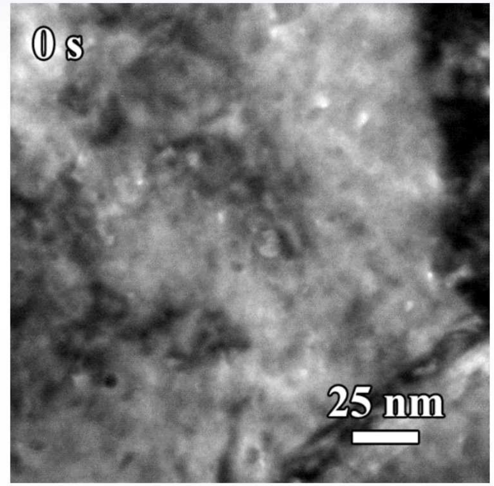
Improved vibrational and ion beam stability permits us to work at 120kx or higher permitting imaging of single cascade events

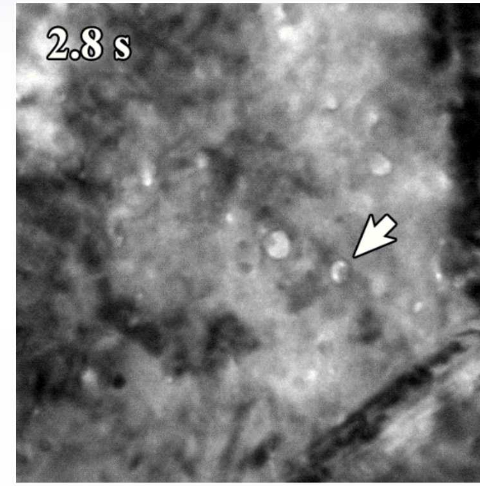
Simultaneous *In situ* TEM Triple Beam: 2.8 MeV Au^{4+} + 10 keV He^{+}/D_2^{+}

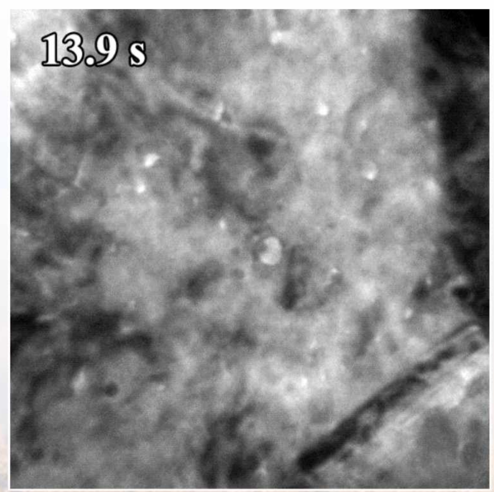
Collaborator: D.C. Bufford

Video playback speed x1.5.







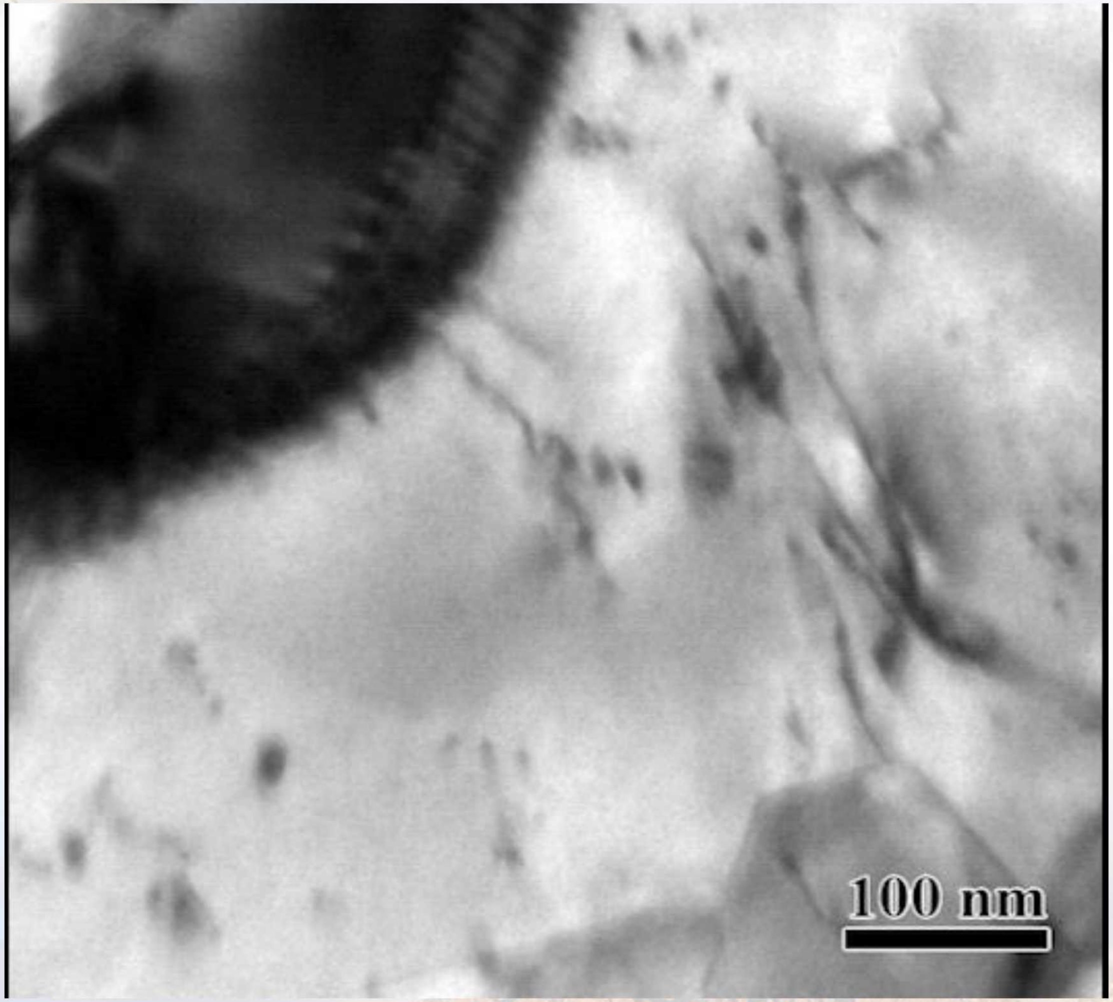


In-situ triple beam He, D_2, and Au beam irradiation has been demonstrated on Sandia's I^3TEM! Intensive work is still needed to understand the defect structure evolution that has been observed.

- **Approximate fluence:**
  - Au 1.2 $\times 10^{13}$ ions/cm^2
  - He 1.3 $\times 10^{15}$ ions/cm^2
  - D 2.2 $\times 10^{15}$ ions/cm^2
- **Cavity nucleation and disappearance**

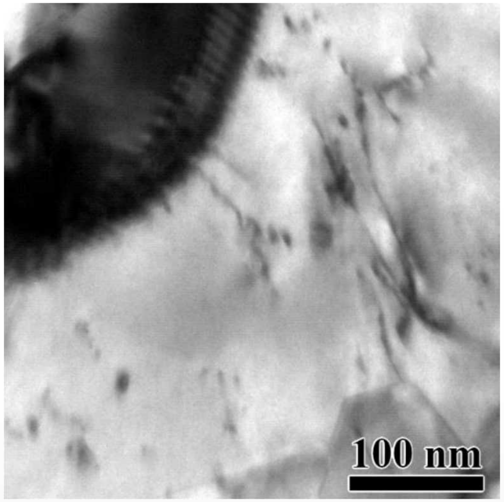
1D Brownian Motion in Real Time

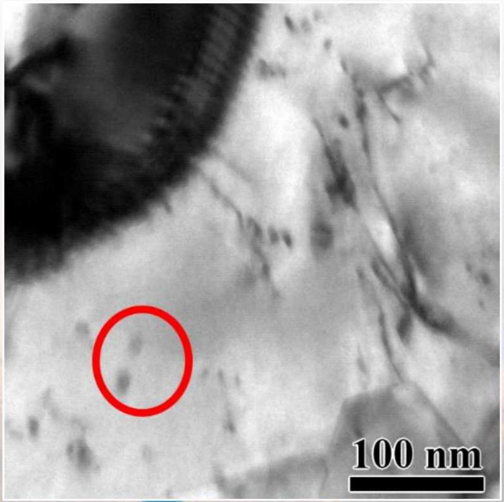
Collaborator: D.C. Bufford



Triple beam condition:

2.8 MeV Au^{4+} + 10 keV $\mathbf{He^{+}/D_2^{+}}$

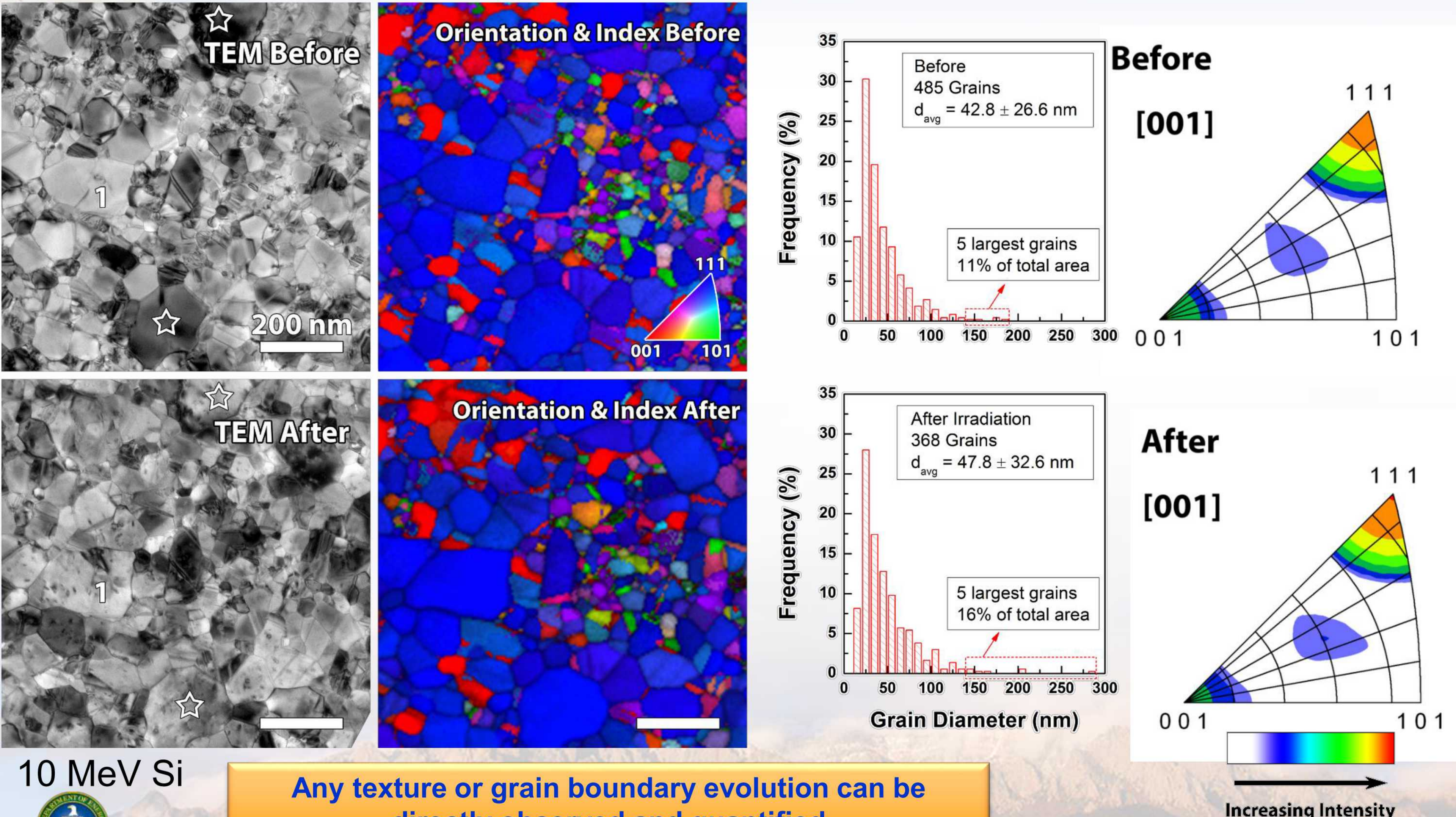




- **Dislocation loop moves between two pinning sites**
  - ~30 nm apart

Quantifying Grain Boundary Radiation Stability of Nanocrystalline Au

Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles



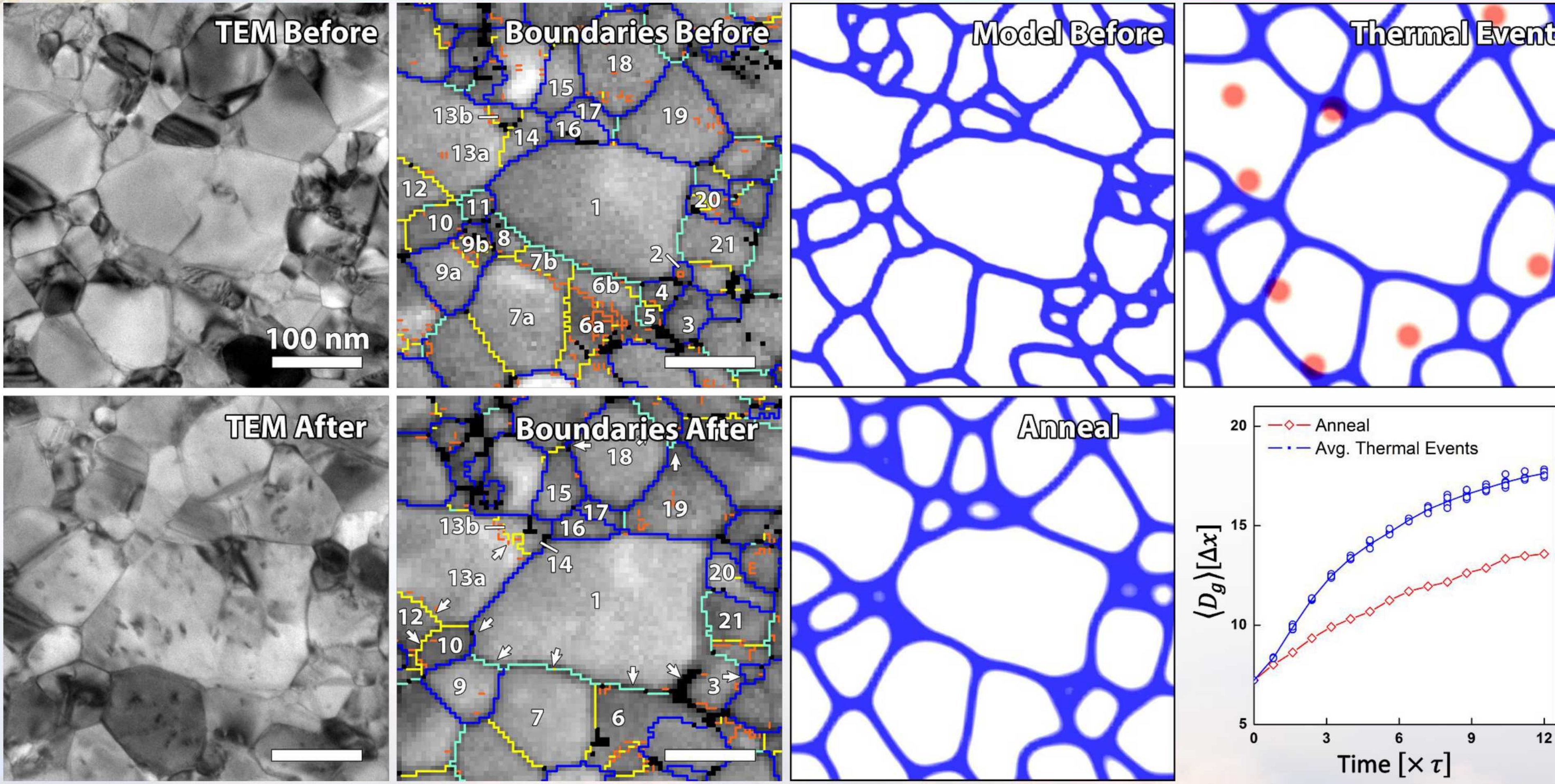
10 MeV Si

Any texture or grain boundary evolution can be directly observed and quantified

Sandia National Laboratories

Direct Comparison to Mesoscale Modeling

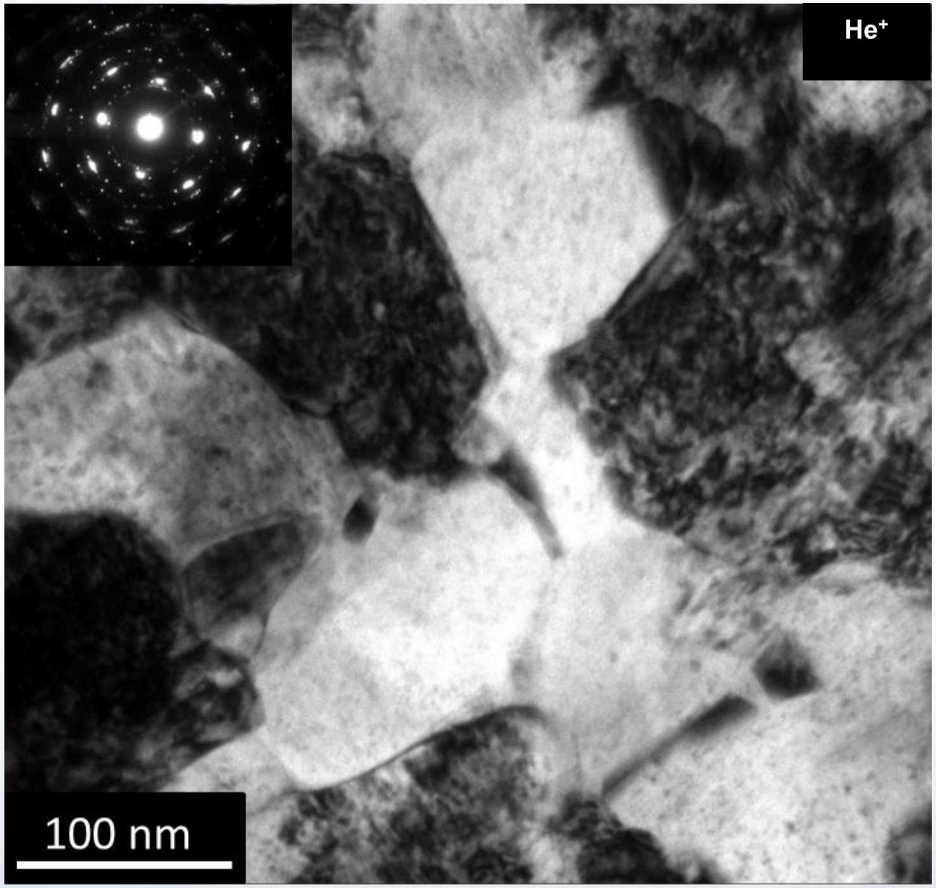
Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles

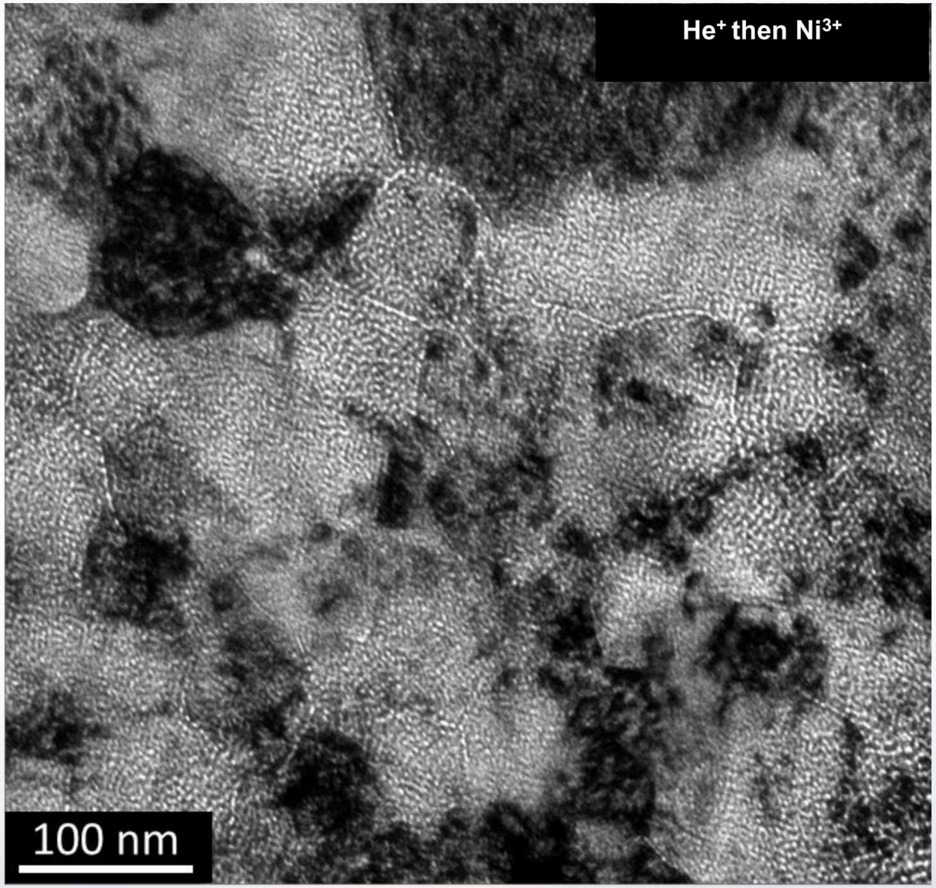


Because of the matching length scale, the initial microstructure can serve as direct input to either MD of mesoscale models & subsequent structural evolution can be directly compared.

Heterogeneous Bubble Formation under Some Radiation Sequences

Collaborator: B. Muntifering & J. Qu





10^{17} He^{+}/cm^{2}

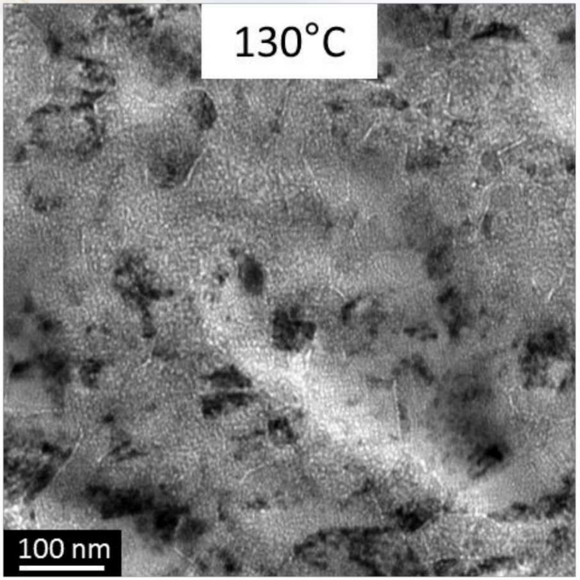
Visible damage to the sample

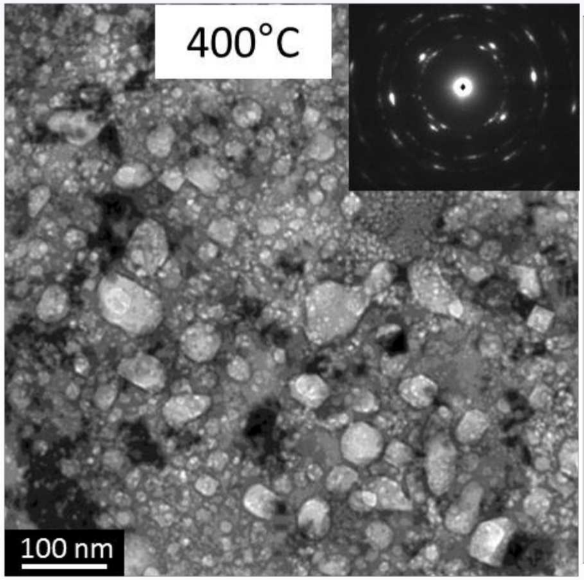
0.7 dpa Ni^{3+} irradiation

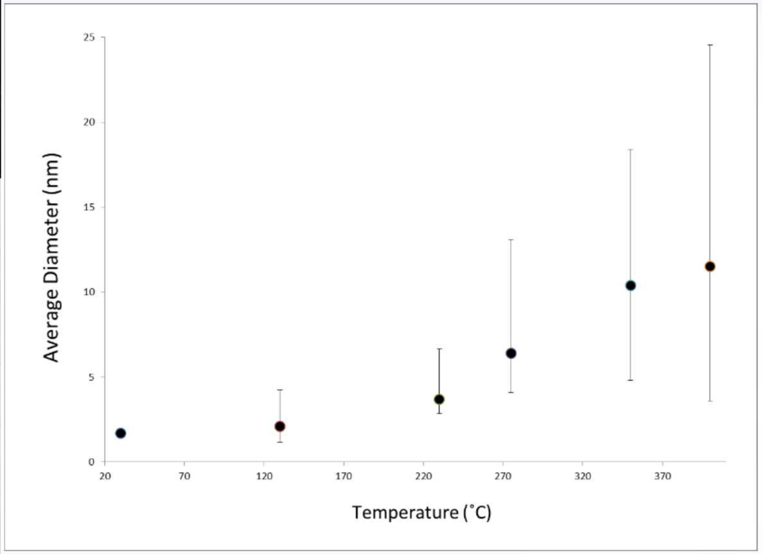
High concentration of cavities along grain boundaries

Cavity Growth during *In situ* Annealing of 10 keV He^{+} Implanted and then 3 MeV Irradiated Ni^{3+}

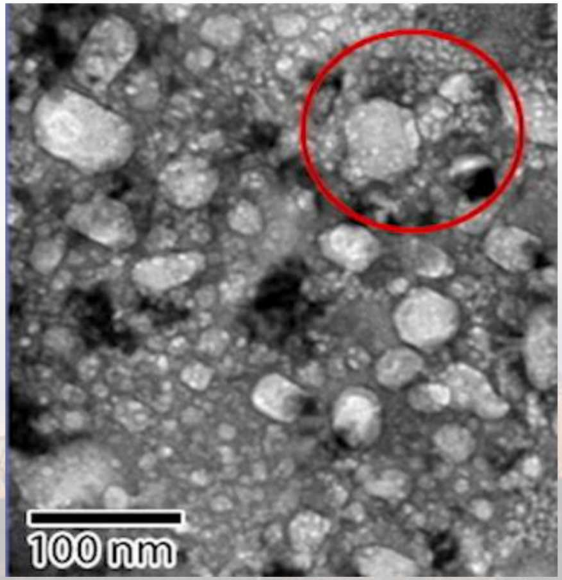
Collaborator: B. Muntifering & J. Qu

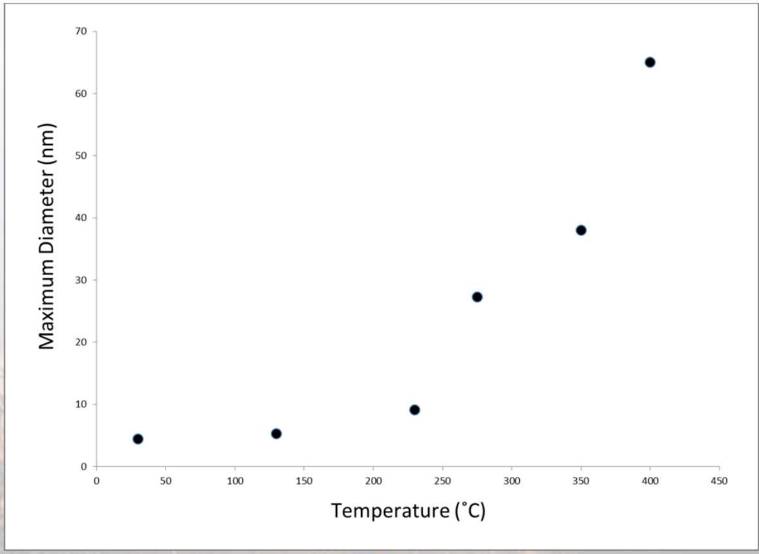






Bubble to cavity transition and cavity evolution can be directly studied



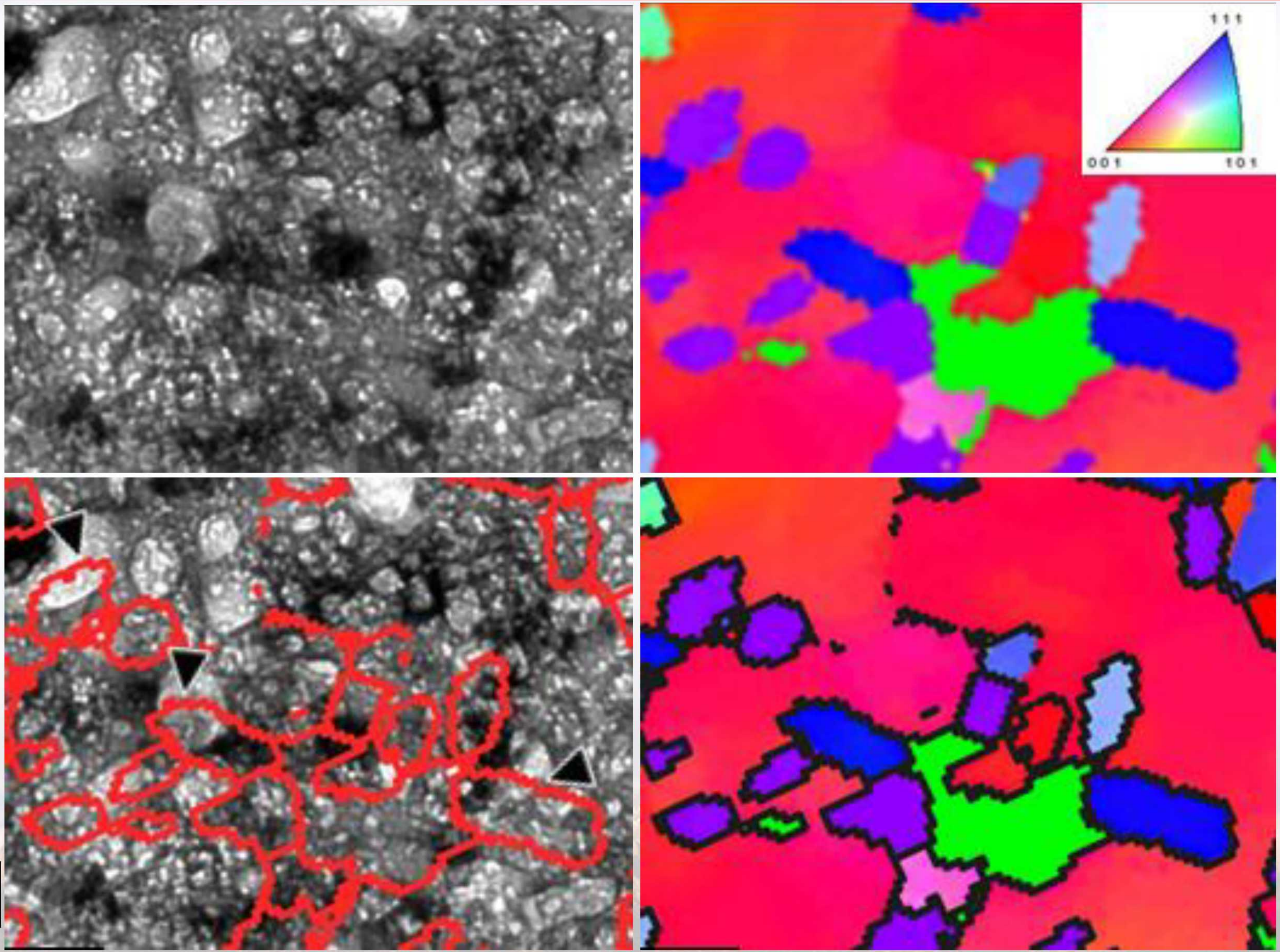


Precession Electron Diffraction Reveals Hidden Grain Structure

Collaborator: B. Muntifering & J. Qu

Cavities in helium implanted, self-ion irradiated,

nc nickel film annealed to 400 ˚C

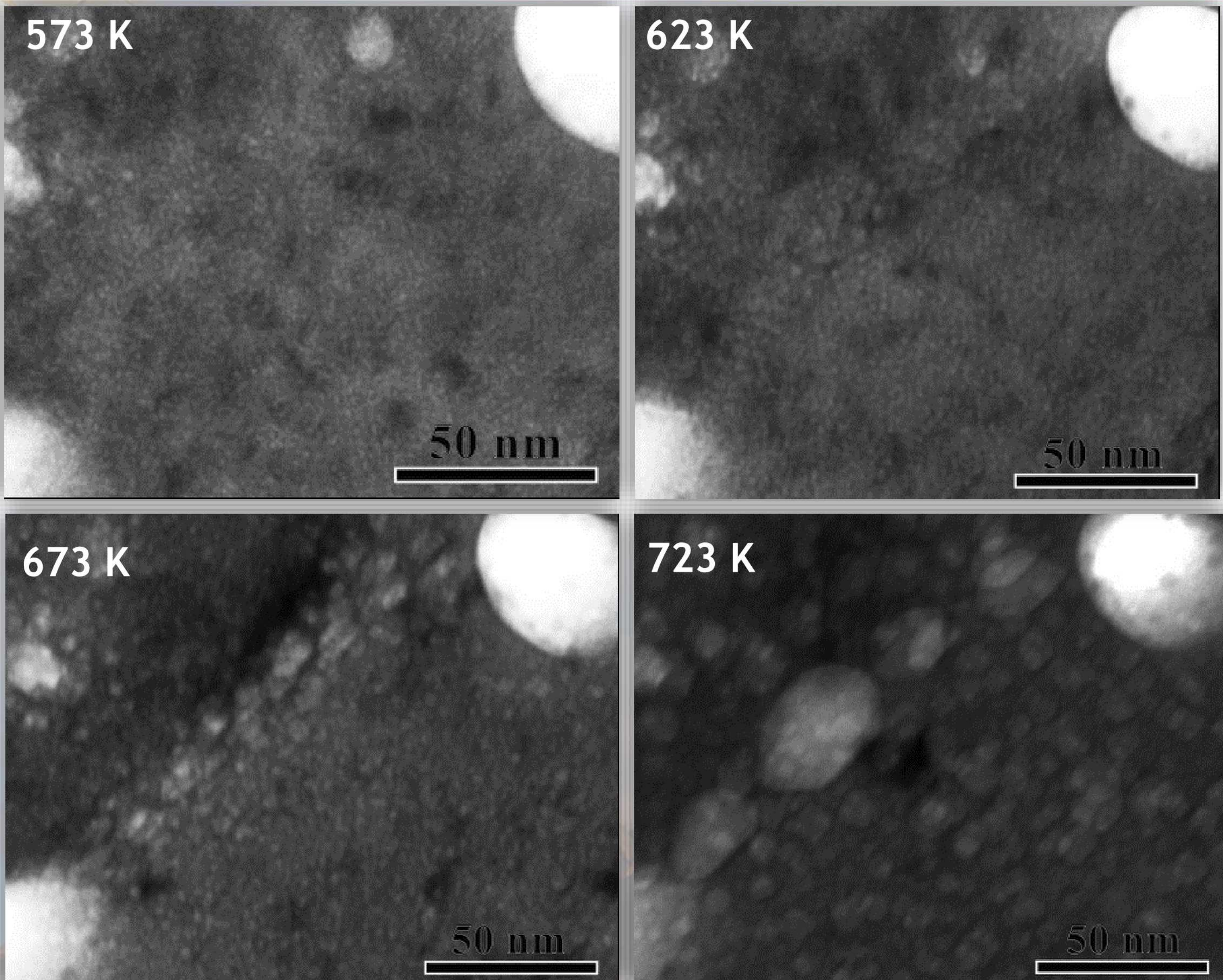


Cavities span multiple grains at identified grain boundaries

100 nm

Bubble Migration and Growth in a Single Grain Boundary

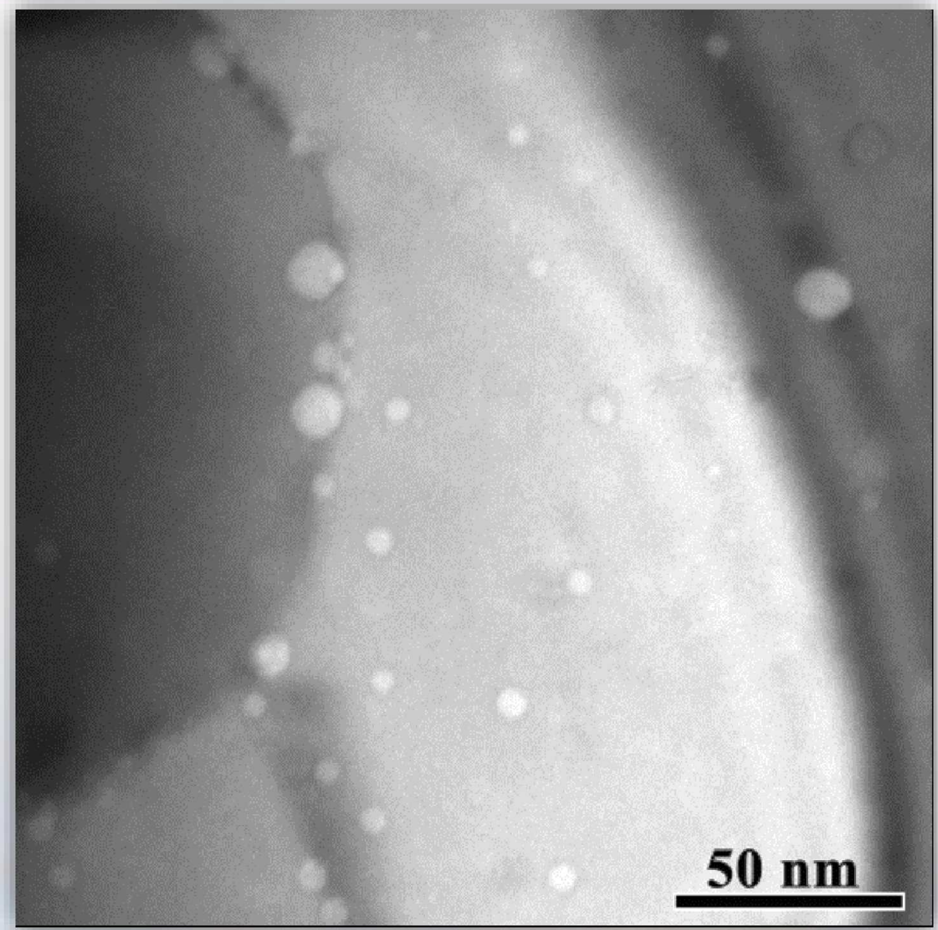
Collaborator: C. Taylor, B. Muntifering, J. Sugar & D. Adams

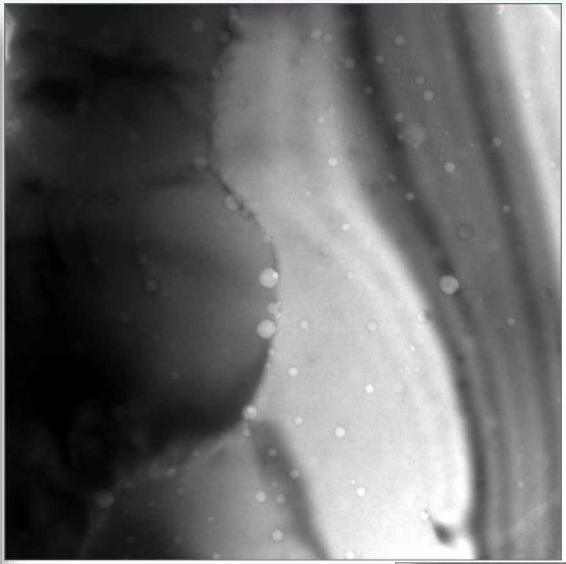


- Growth at GB from diffusion of interstitial He or small He-V clusters
- Growth at GB from diffusion of interstitial He or small He-V clusters
- Bubble migration to GB & cavity growth, intragranular growth from diffusion of interstitial He or small He-V clusters
- Blister formation at boundaries, intragranular faceted cavity growth. Growth by cavity diffusion and coalescence.

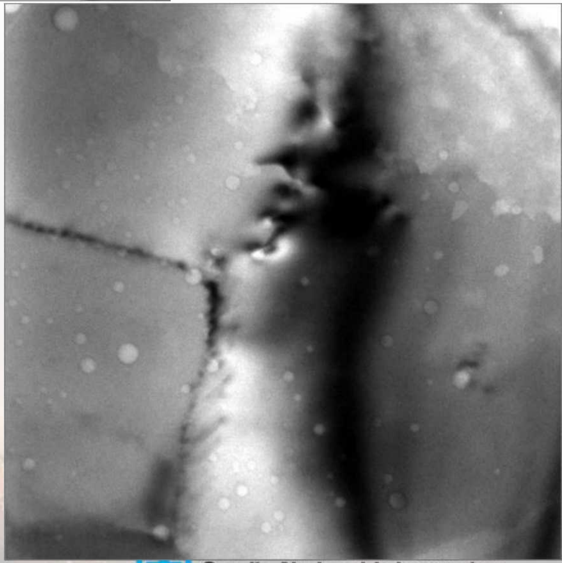
Cavities Role on Grain Boundary Motion

Collaborator: C. Taylor, B. Muntifering, J. Sugar & D. Adams





Cavities in helium implanted, Pd foil during annealing at 700 ˚C

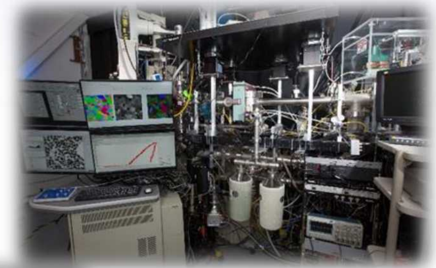
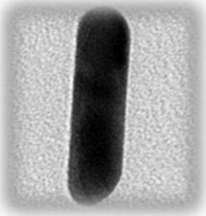
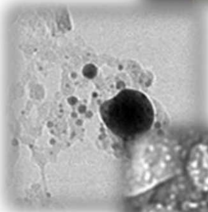
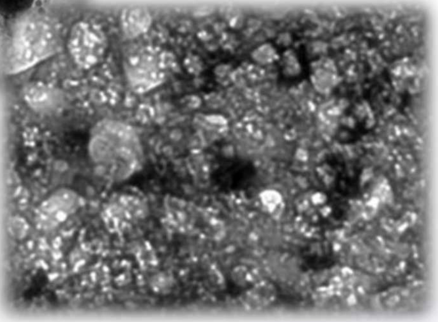


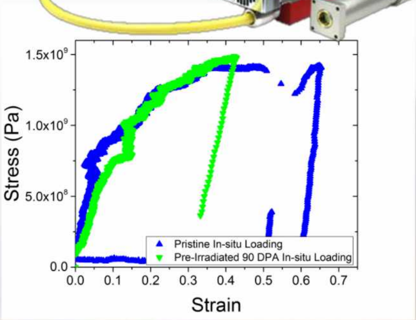
Cavities effect grain boundary mobility, triple junction angle

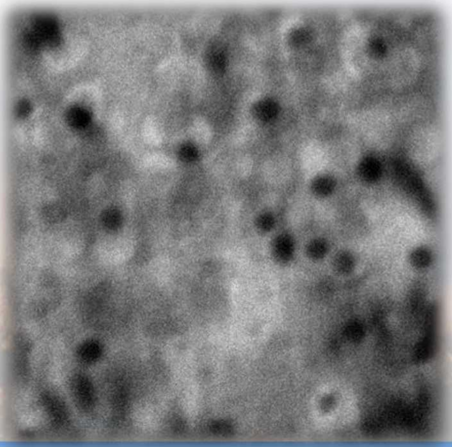
grain boundary motion alters cavity coalescence

Outline

1. Introduction to Sandia National Laboratories and its *In situ* Ion Radiation Transmission Electron Microscope (I^3TEM)
2. Understanding radiation stability in nanoparticles
3. Effect of radiation environments on nanocrystalline metals
4. Lasers in a TEM (why not?)
5. State-of-art in quantitative in situ mechanical testing
6. Other environments
(in situ SEM, liquid, and gas)





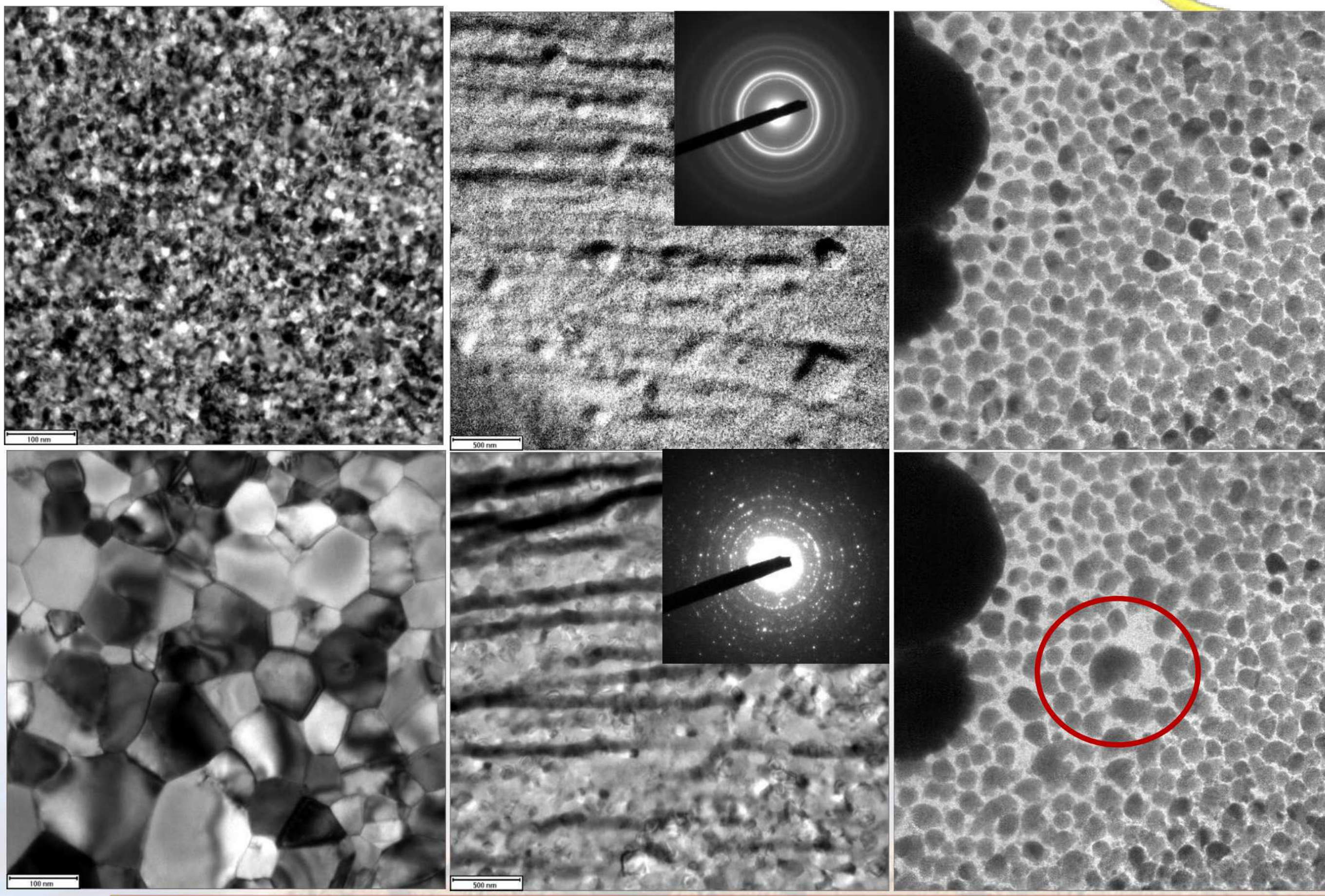
Initial Laser Heating Observations

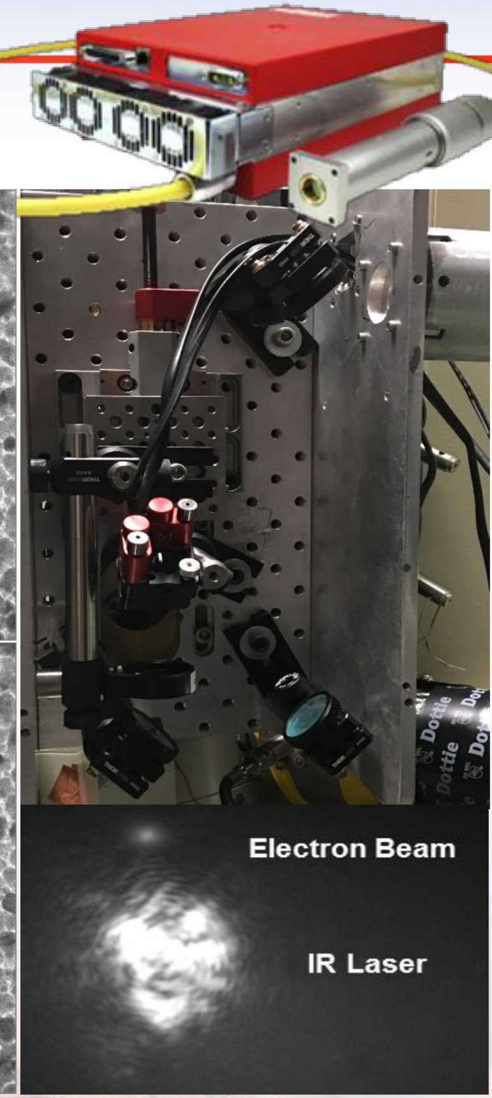
Collaborator: P. Price, C.M. Barr, D. Adams, M. Abere

Pt Grain Growth | **Reactive Multilayer Films** | **Nanoparticle Sintering**

Before

After

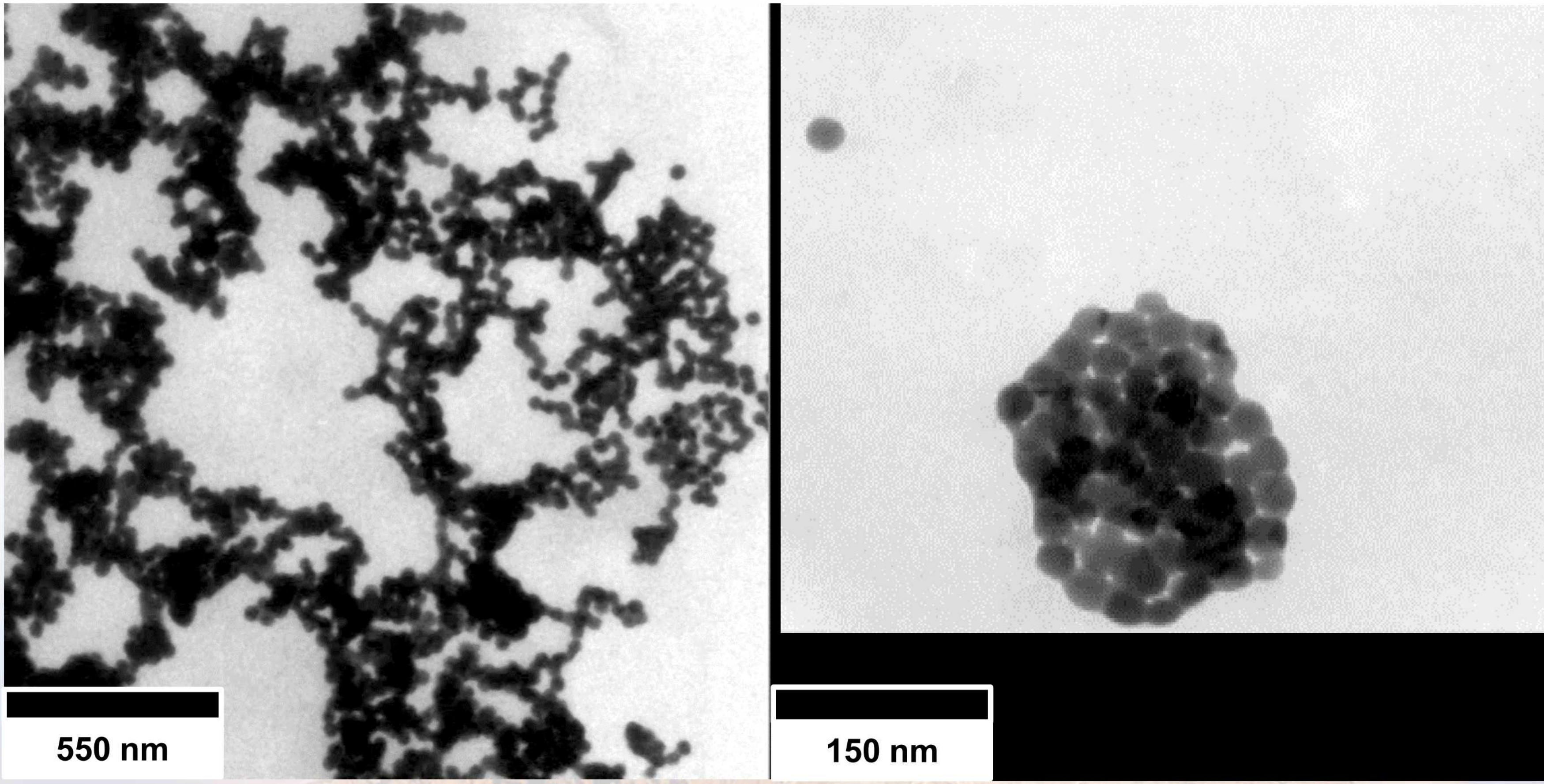




We can now introduce rapid thermal heating with any TEM stage or ion beam conditions

Complex Interaction Au NPs Exposed to Laser Irradiation

Contributors: P. Price, L. Treadwell, A. Cook



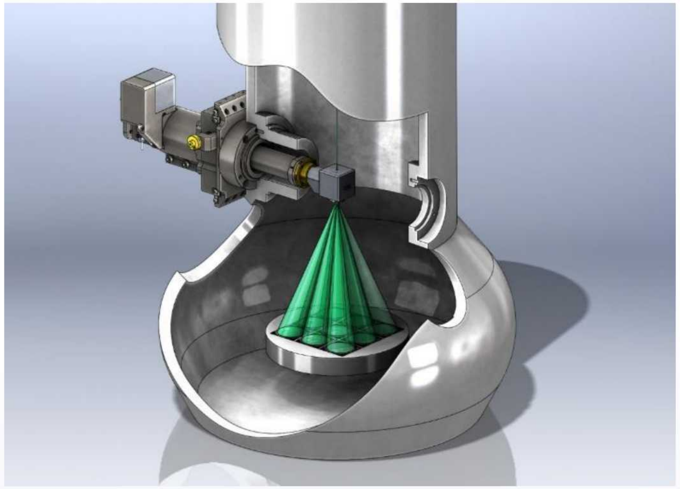
Speed = 2.5x

A Complex Combination of Sintering, Reactions, and Ablation Occurs

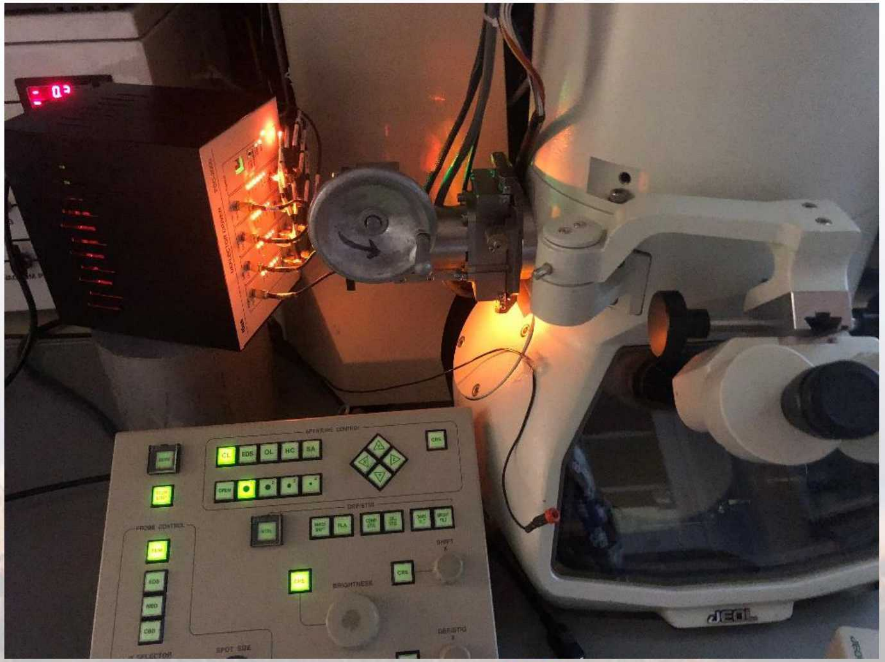
μs Resolution with a Standard Camera

Collaborator: P. Price, A. Monterrosa, D. Adams, M. Abere, & IDES Inc.

fs ps ns **μs** **ms** s

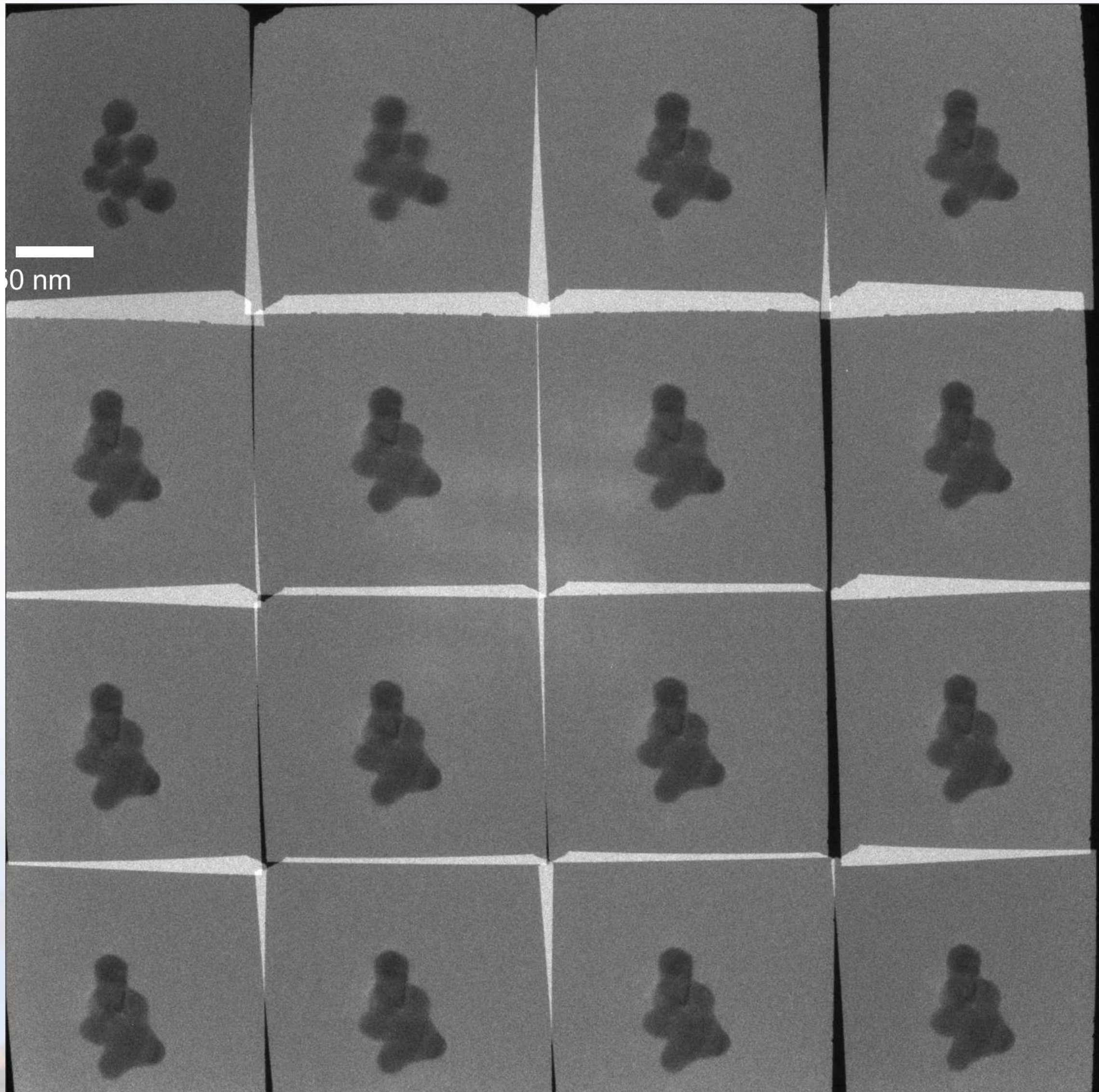


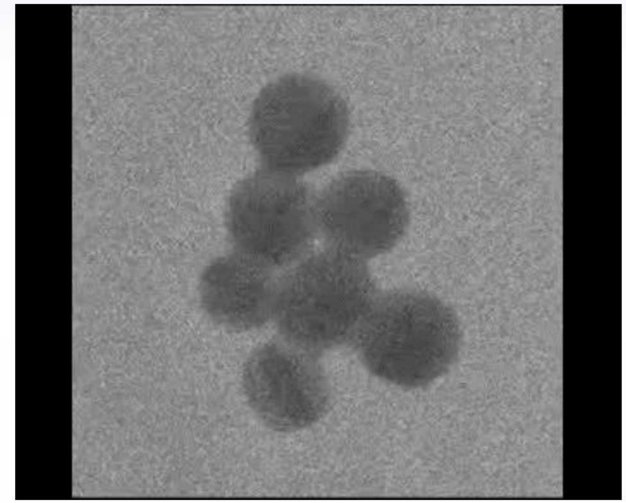
- **Electrostatic deflection of electrons**
- **4, 9, or 16 images per frame, spread over a large camera**
- **Any exposure time up to the limits of the camera**
  - Ultimate limit is beam current/brightness

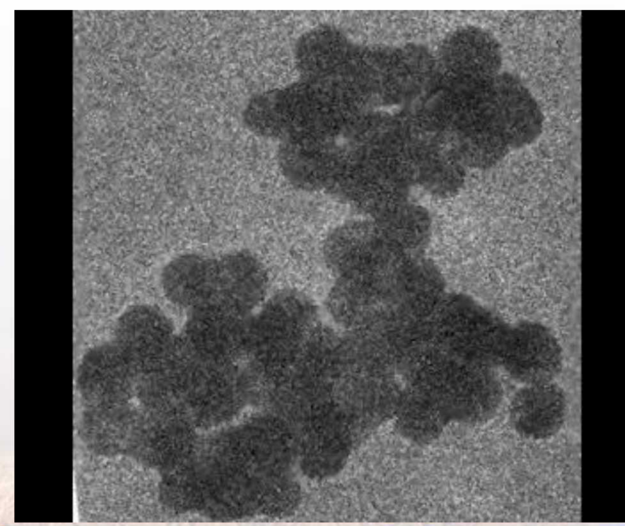


1-to-1 Frame Capture (<5 ms per frame)
Sintering of 20 nm Au Nanoparticles

Collaborator: P. Price, A. Monterrosa, & IDES Inc.



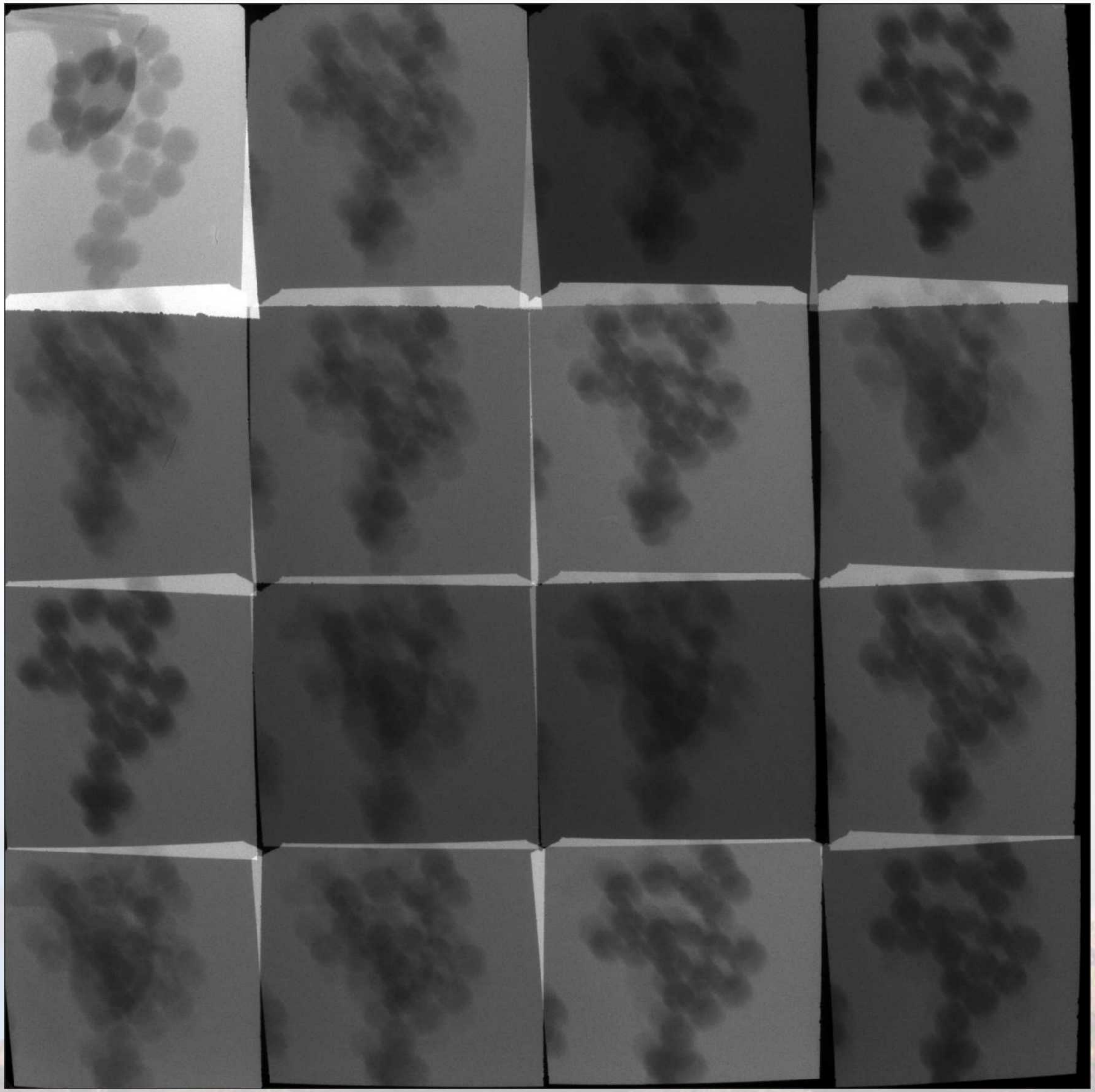




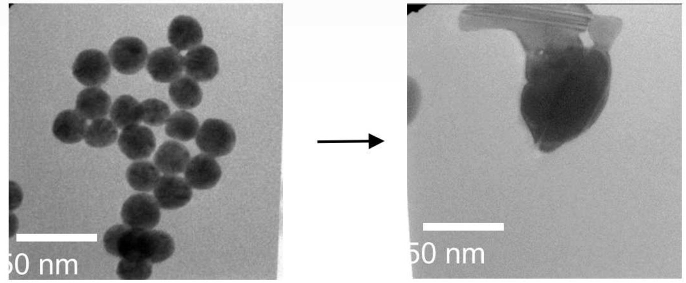
16 frames captured with <5 ms exposure per frame

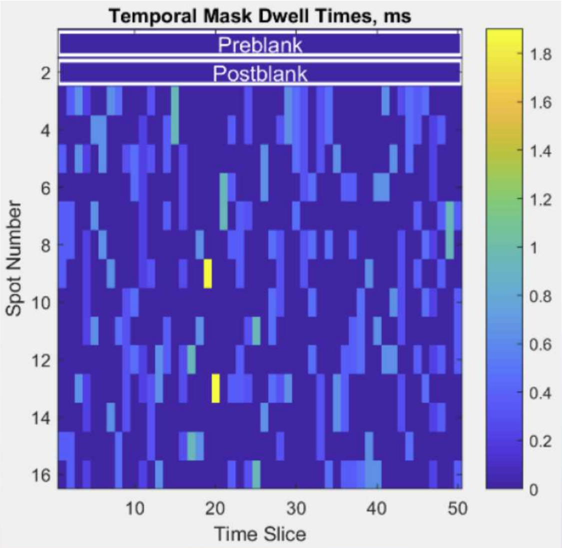
Temporal Compressive Sensing to Improve Temporal Resolution

Collaborator: P. Price, A. Monterrosa, & IDES Inc.



Before → After

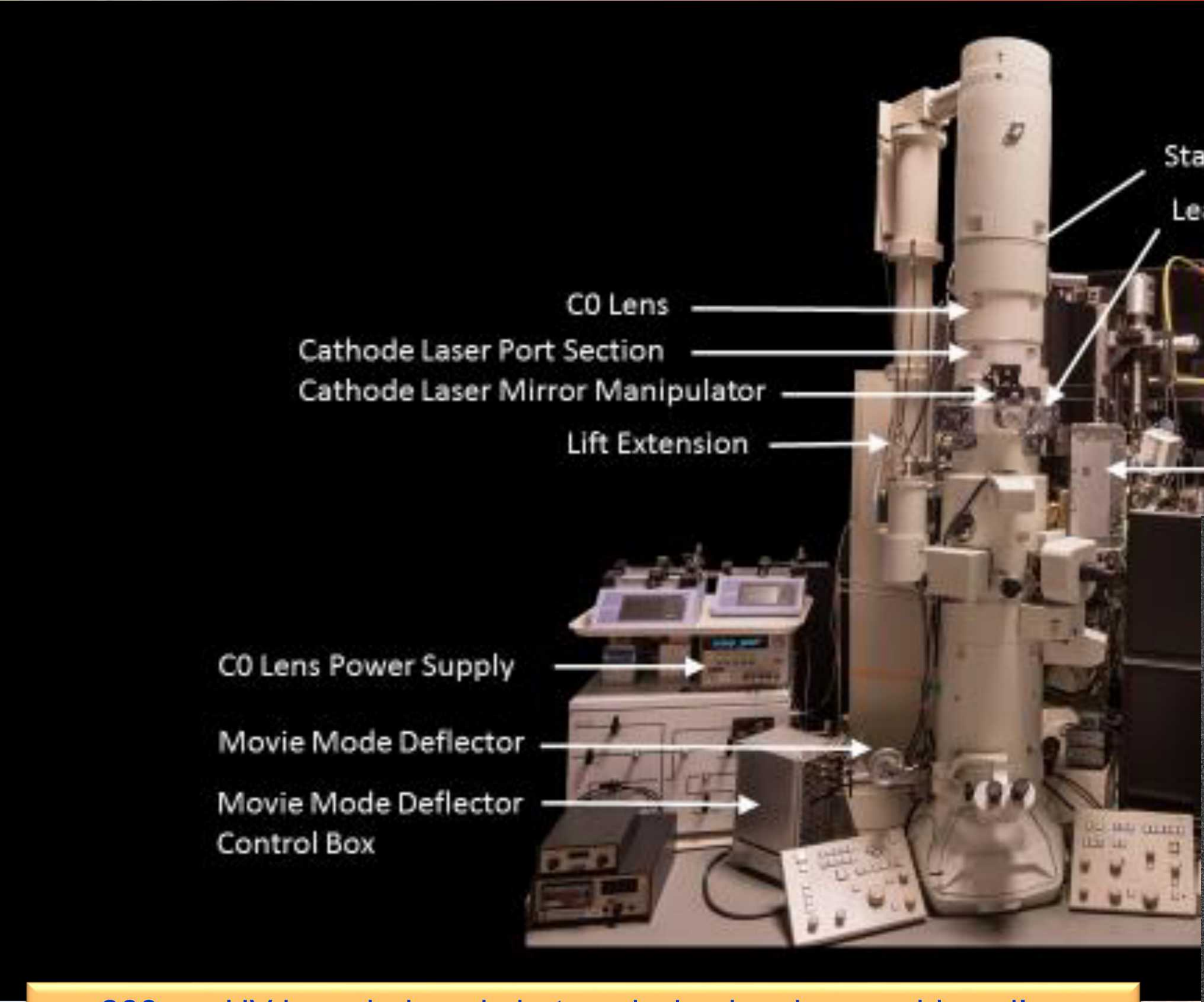


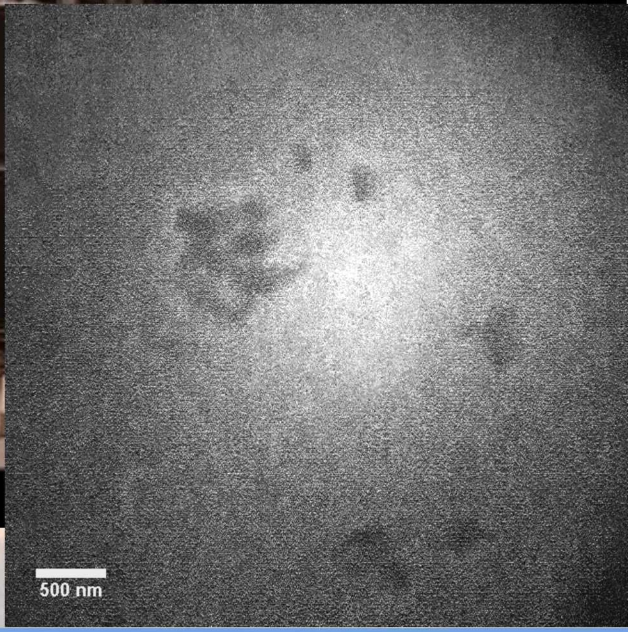


A pseudorandom exposure pattern can produce more than 16 frames within the same exposure time

Current Status of DTEM Conversion

Collaborator: P. Price, A. Monterrosa, C.M. Barr, D. Adams, M. Abere, & IDES Inc.

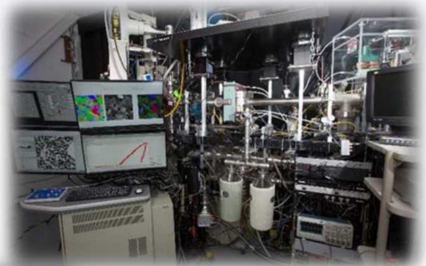


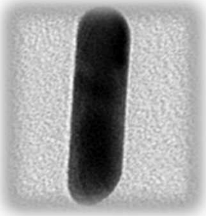


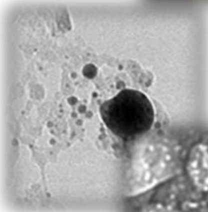
- 266 nm UV laser induced photoemission has been achieved!
- 6 ns single-shot DTEM image of P47

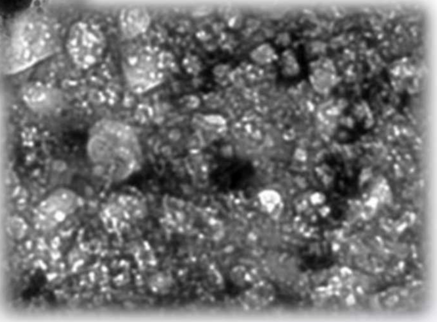
Outline

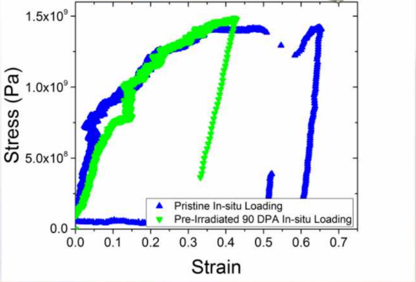
1. Introduction to Sandia National Laboratories and its *In situ* Ion Radiation Transmission Electron Microscope (I^3TEM)
2. Understanding radiation stability in nanoparticles
3. Effect of radiation environments on nanocrystalline metals
4. Lasers in a TEM (why not?)
5. State-of-art in quantitative in situ mechanical testing
6. Other environments (in situ SEM, liquid, and gas)

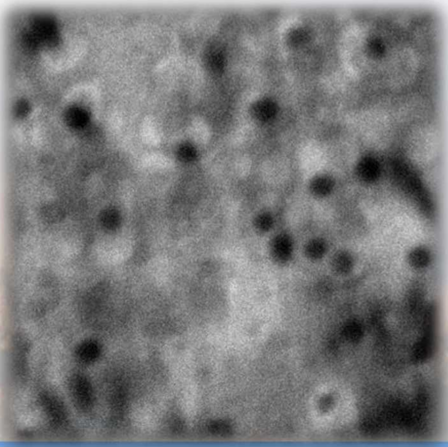












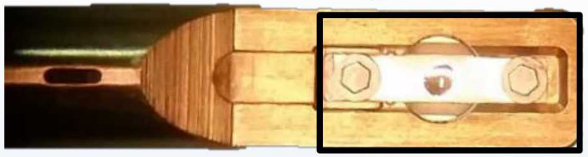
In situ Qualitative Mechanical Testing

Collaborators: C. Barr

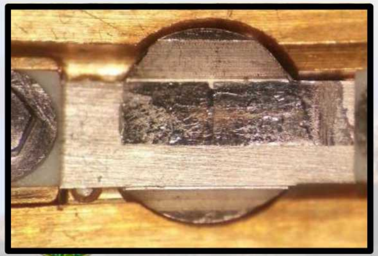
Gatan straining TEM Holder

- Minimal control over displacement and no “out-of-box” force information
- Successful in studies in observing dislocation-GB interactions/mechanisms
- Ideally both grains have kinematic BF 2-beam conditions: challenging in ST holder

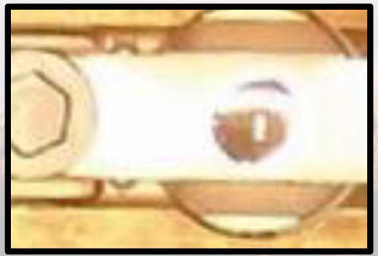
Traditional Gatan Heating and Straining Holder



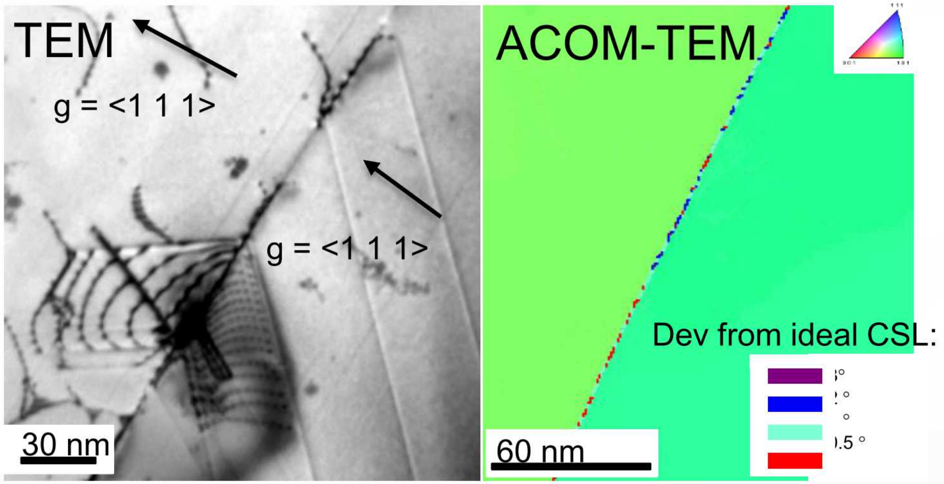
Thin film tension “jig”:



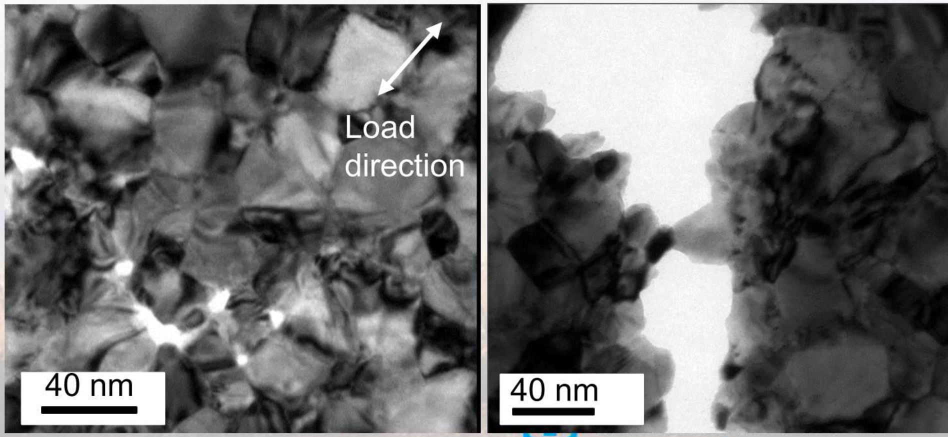
Jet thinned disk:



Dislocation interactions as a function of GB character (Σ3 twin GB below):



Observe deformation mechanisms in nanocrystalline metals during tensile straining:

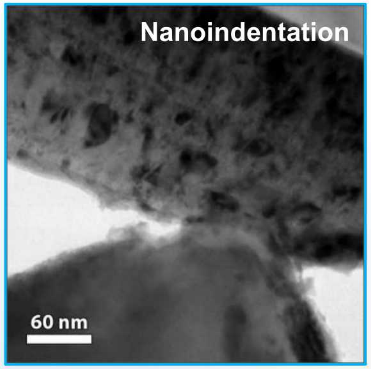


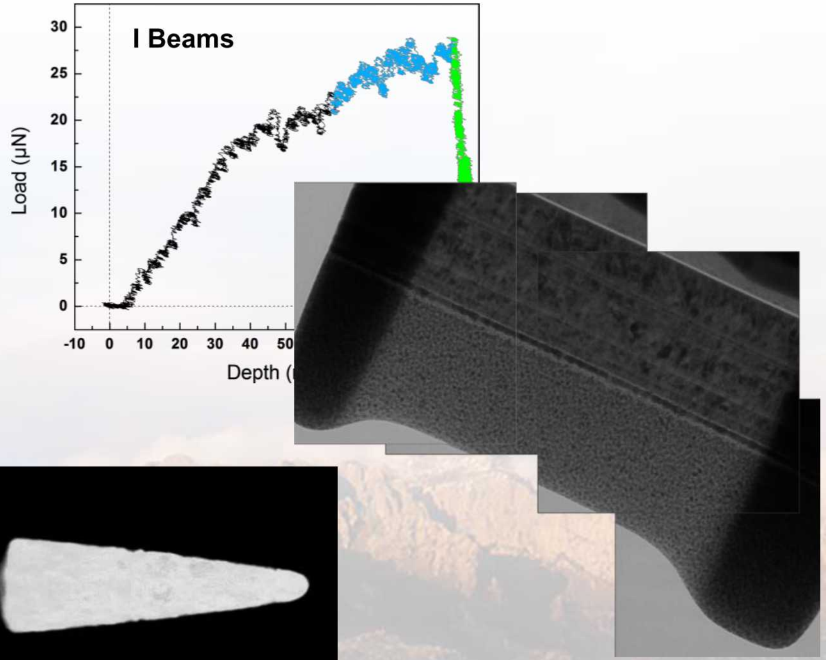
In situ Quantitative Mechanical Testing

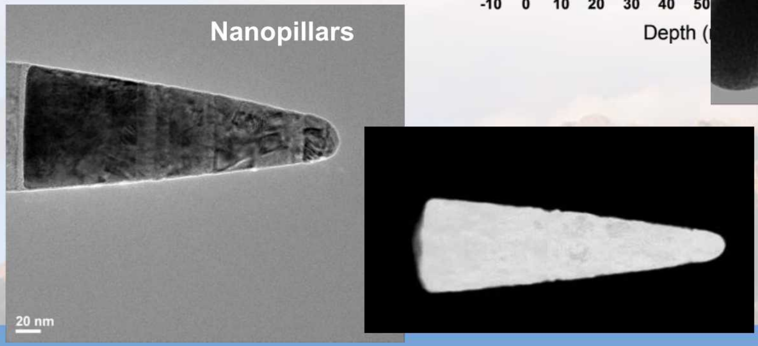
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

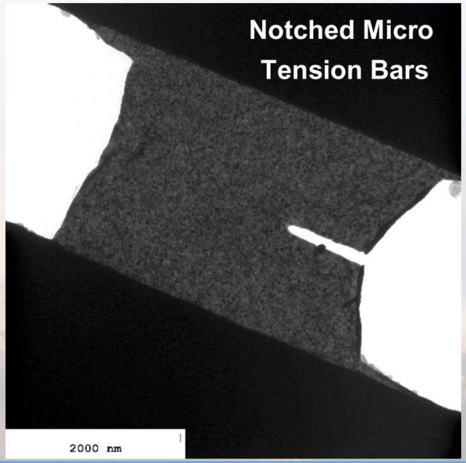
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

- Sub nanometer displacement resolution
- Quantitative force information with μN resolution
- **Concurrent real-time imaging by TEM**



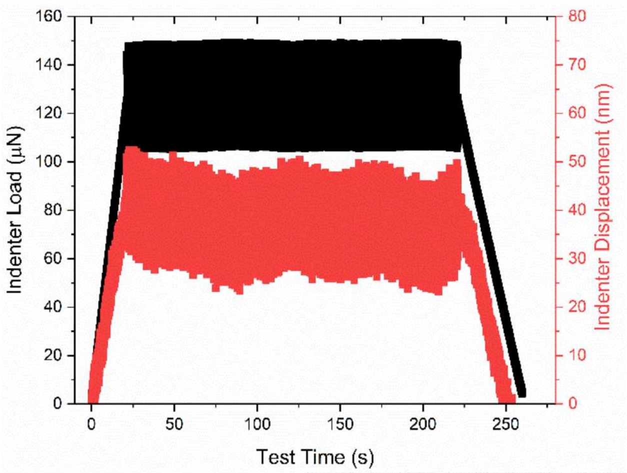






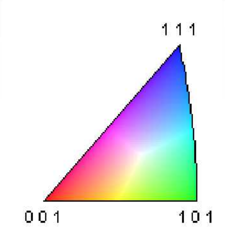
Cyclic Loading Coupled with ACOM

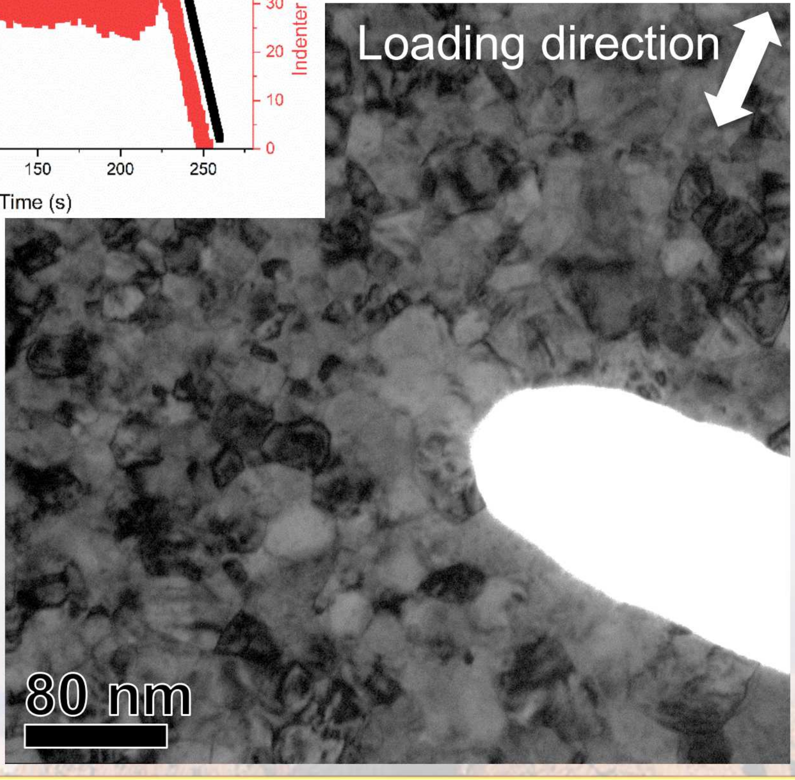
Collaborators: C. Barr & W. Mook

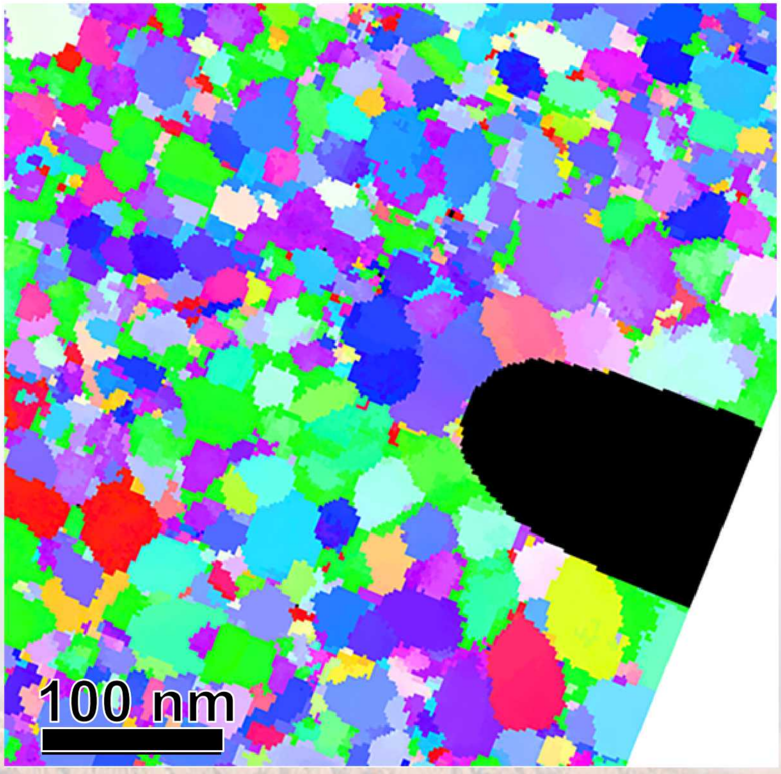


Mean load (P_{mean}) = 135 uN

Amplitude load (P_{amp}) = 35 uN



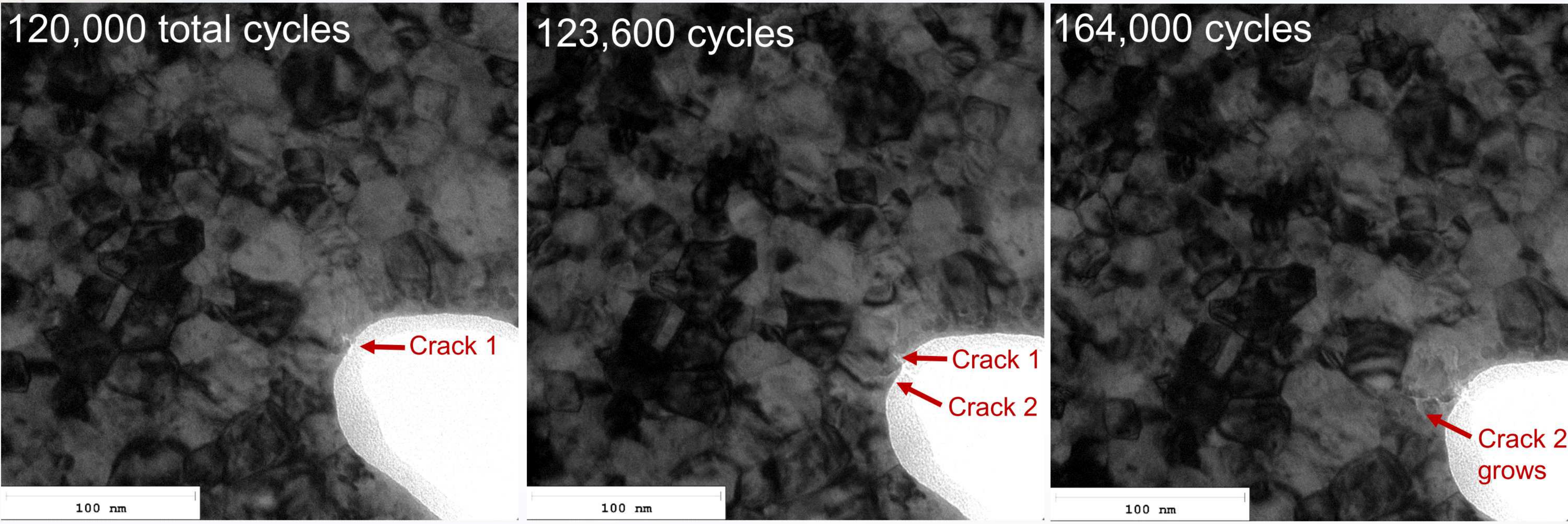




Orientation maps pre-, intermediate, and post- in-situ mechanical test can assist in deconvoluting possible mechanisms during cyclic loading

Crack Initiation at Notch

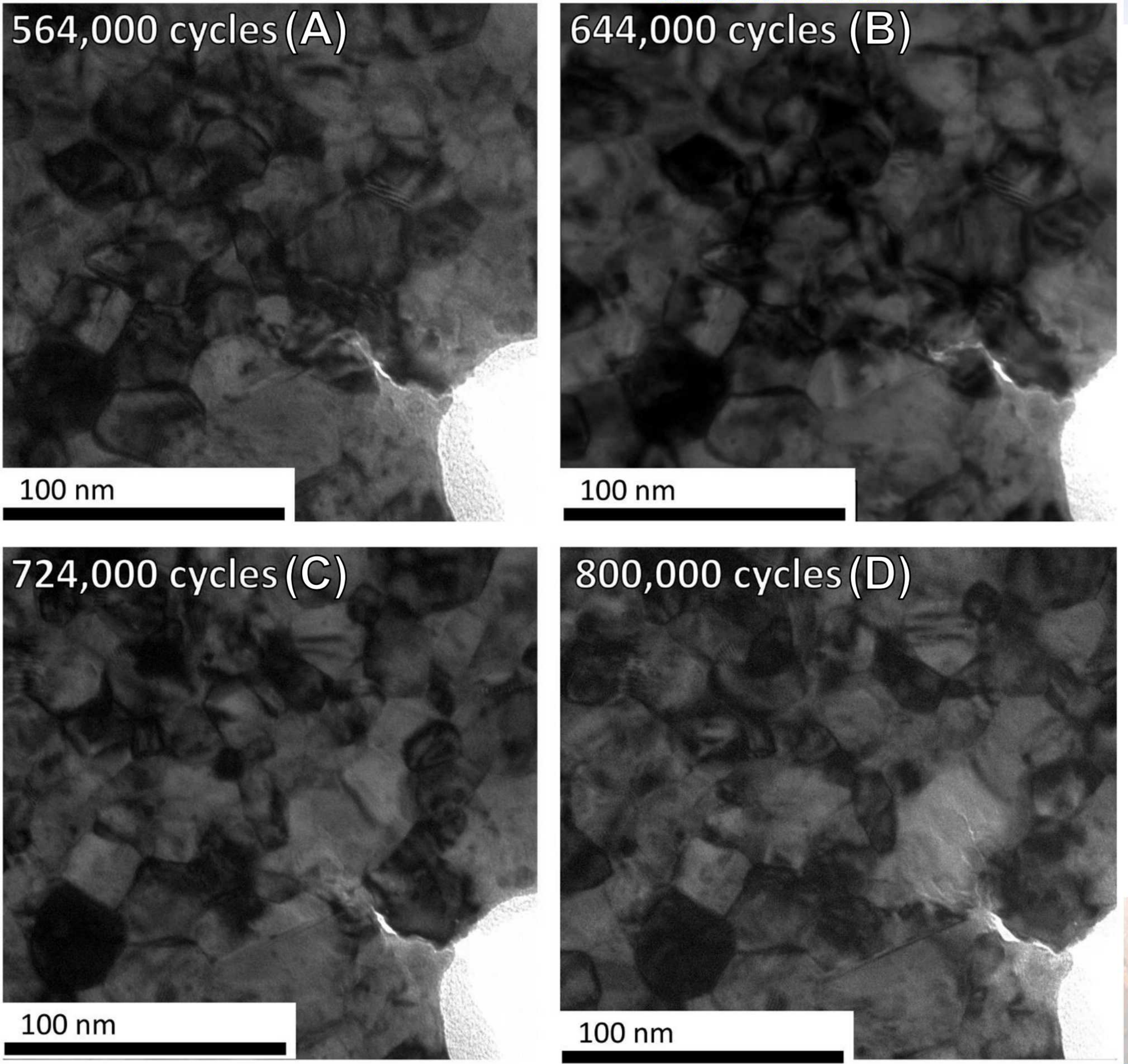
Collaborators: C. Barr & W. Mook

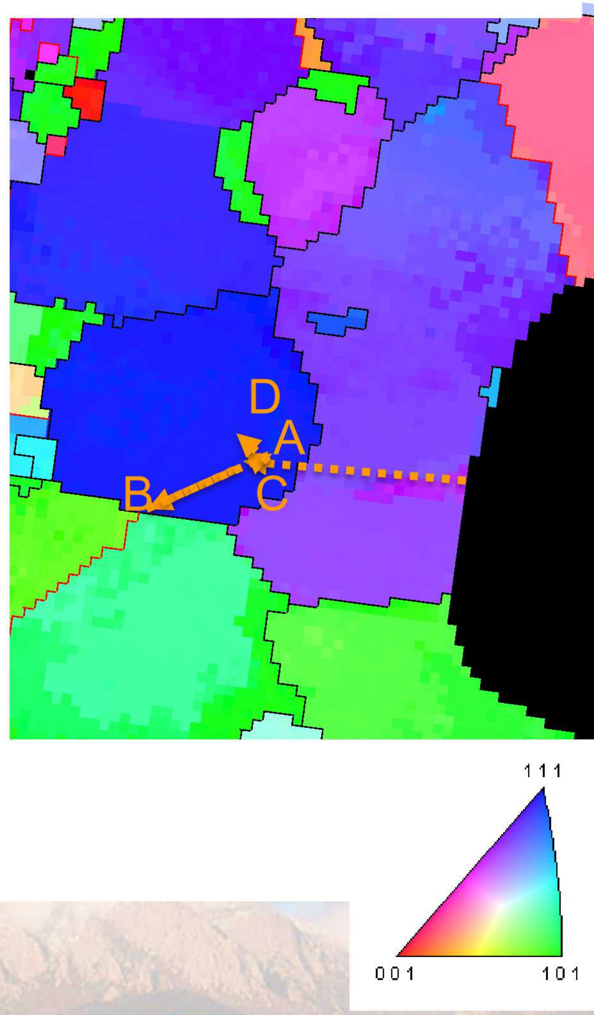


- Crack initiation and initial propagation at notch tip
- Second crack initiates at ~ 90° to first crack, both 45° to notch tip normal
- Intra-granular crack (crack #2) propagates until reaching initial grain boundary and is subsequently arrested

Crack Propagation, Closure, and Re-Direction

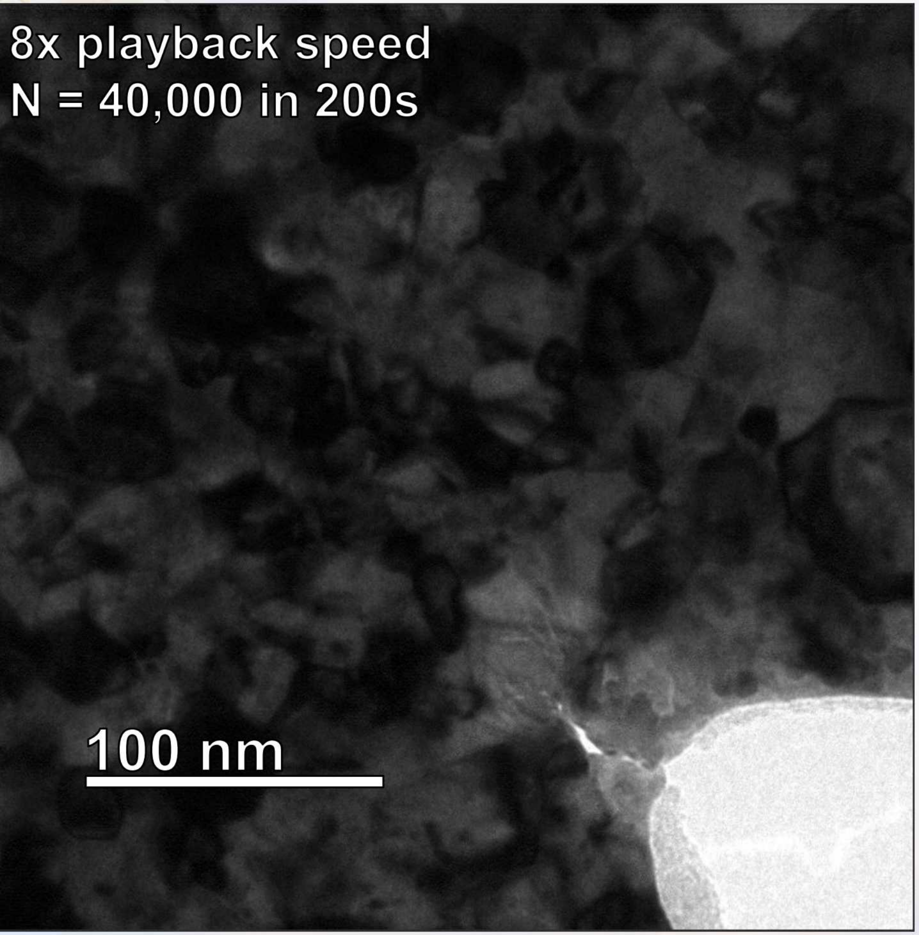
Collaborators: C. Barr & W. Mook

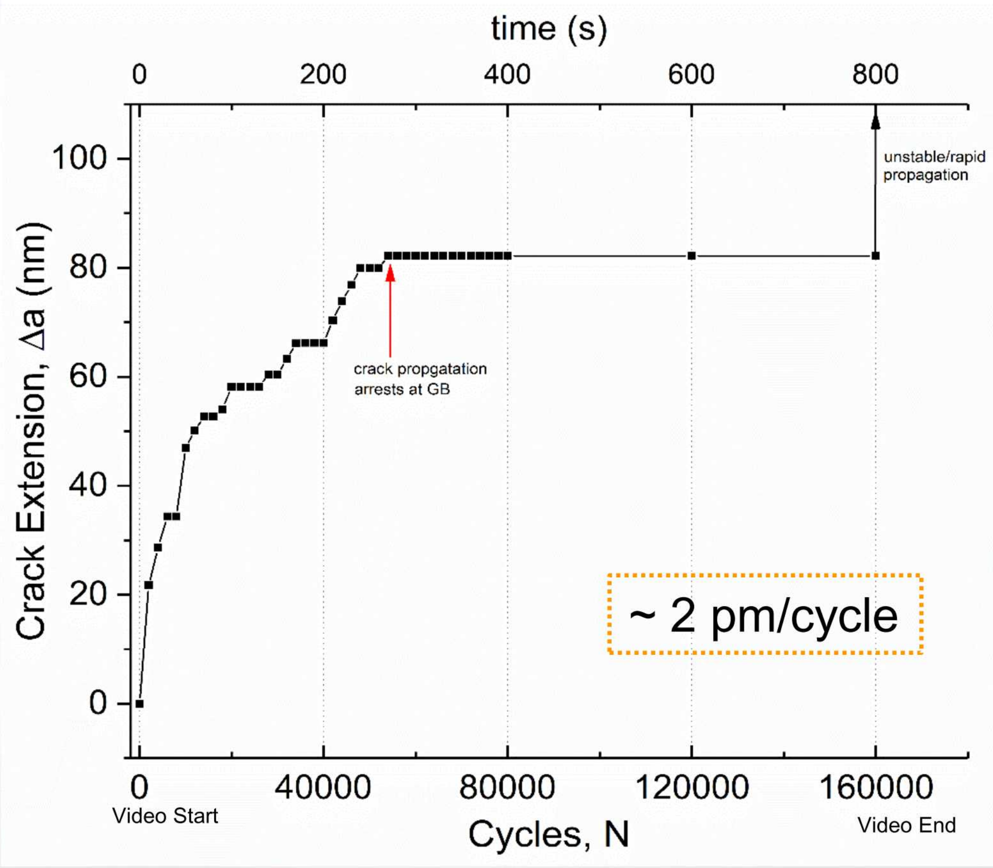




Cyclic Loading: Complex Crack Propagation

Collaborators: C. Barr & W. Mook





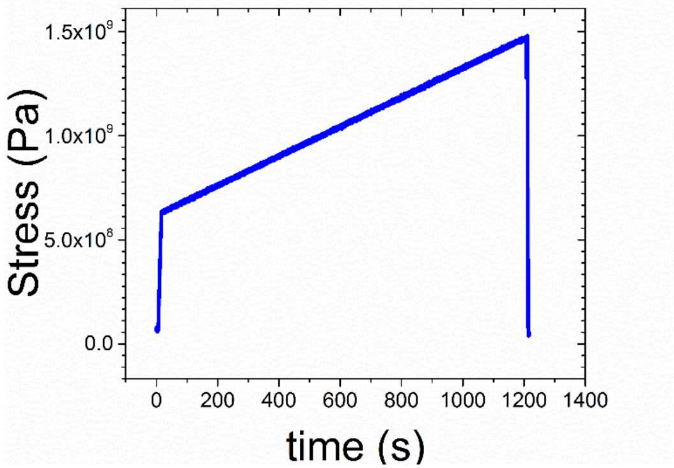
- Mean load: 135 uN; Amplitude load: 35 uN
- 200 Hz, 200s test (15 fps 1k x 1k camera)

- da/dN = 1.7×10^{12} m/cycle
- Non-linear crack extension rate
- Crack propagation path changes “direction”

Irradiation Creep (4 MeV Cu^{3+} 10^{-2} DPA/s)

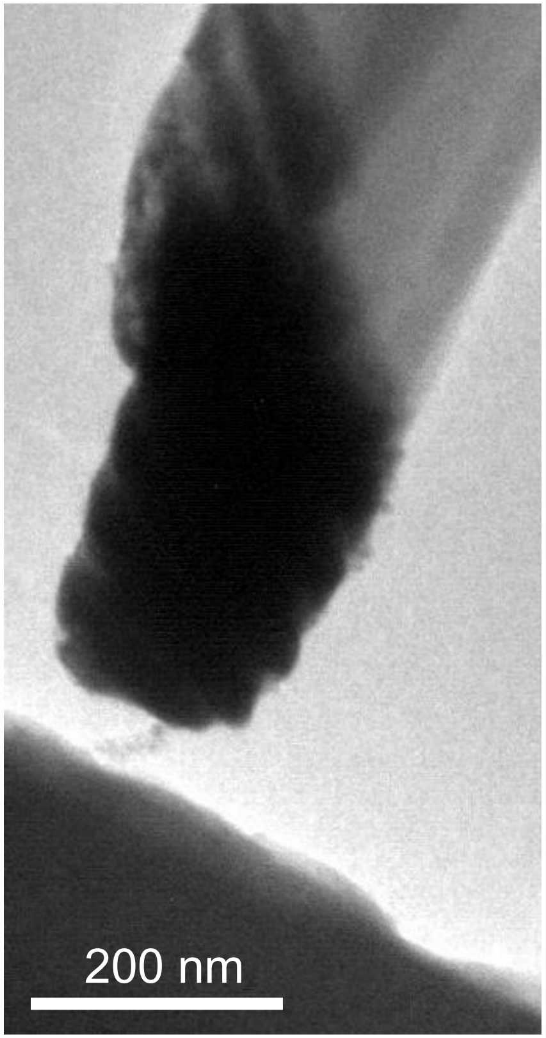
Contributors: G.S. Jawaharram, S. Dillon & R.S. Averback

Controlled Loading Rate Experiments

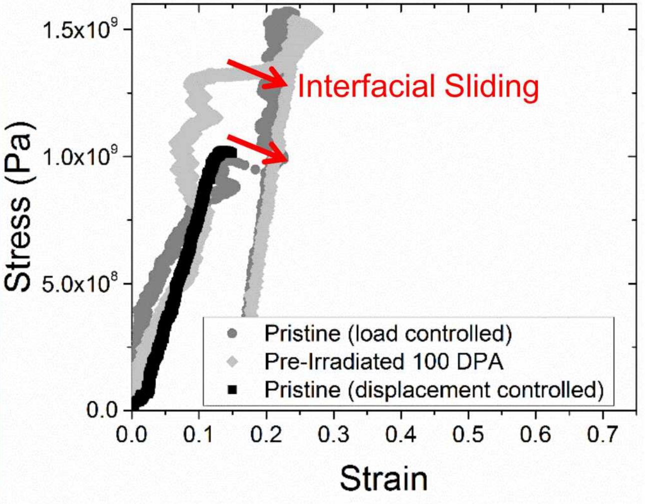


In-situ TEM radiation creep is feasible!

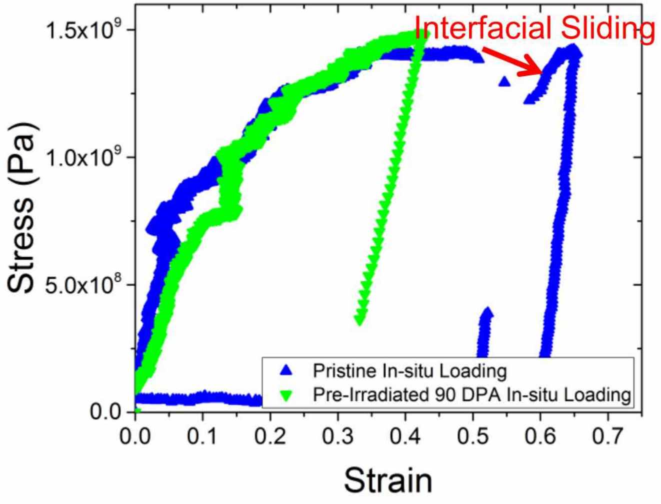
50 nm Cu-W multilayer
20 Min



No Irradiation
(Loading rate 0.6 Mpa s^{-1})

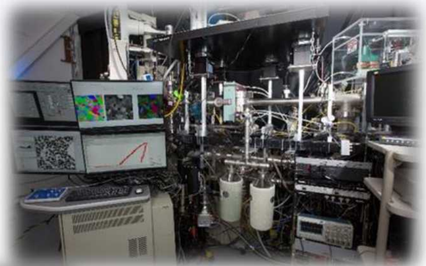


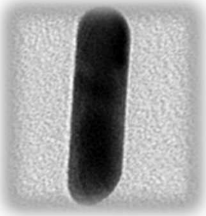
Irradiation Creep
(Loading rate 0.6 Mpa s^{-1})

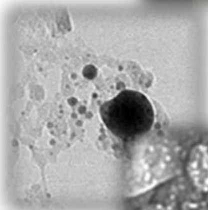


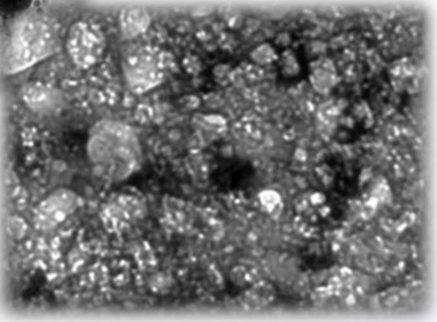
Outline

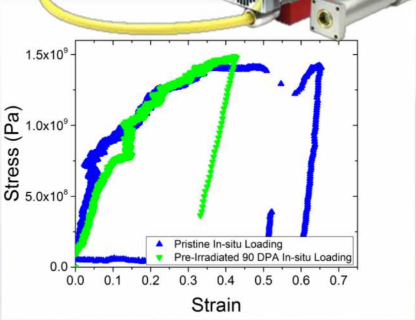
1. Introduction to Sandia National Laboratories and its *In situ* Ion Radiation Transmission Electron Microscope (I^3TEM)
2. Understanding radiation stability in nanoparticles
3. Effect of radiation environments on nanocrystalline metals
4. Lasers in a TEM (why not?)
5. State-of-art in quantitative in situ mechanical testing
6. Other environments
(in situ SEM, liquid, and gas)

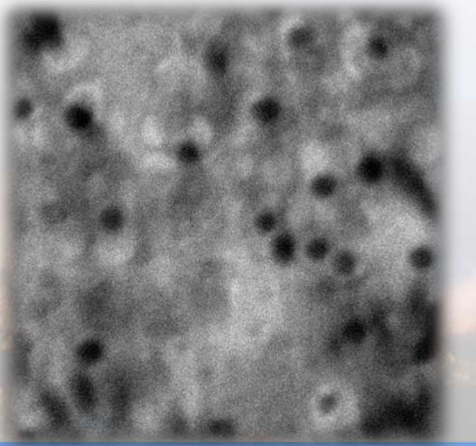






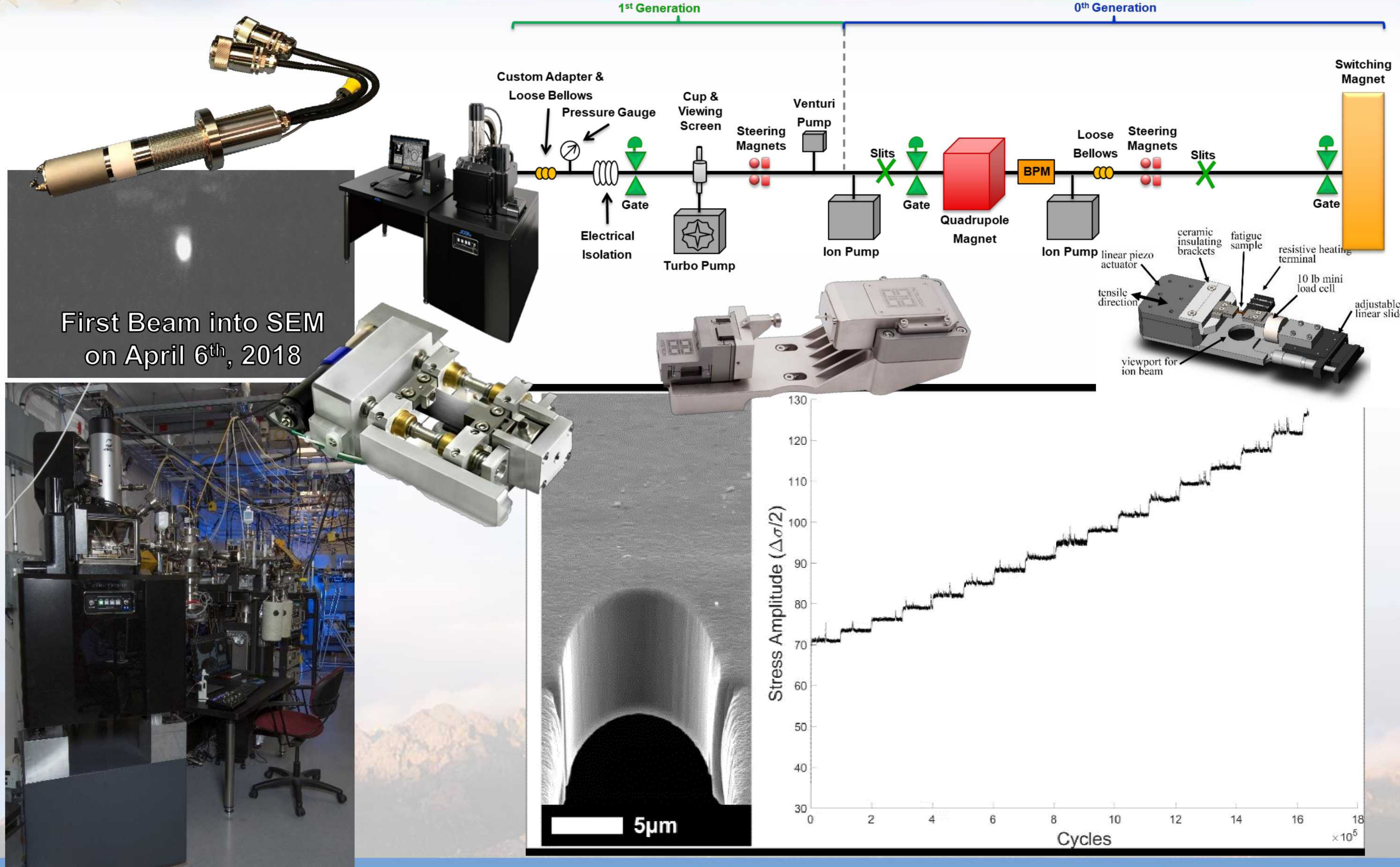






Scaling Back Up:
In situ Ion Irradiation SEM (I^{3}SEM)

Collaborators: N. Heckman, D. Buller, B. Boyce, J. Carroll, C. Taylor, B. Muntifering, & S. Briggs

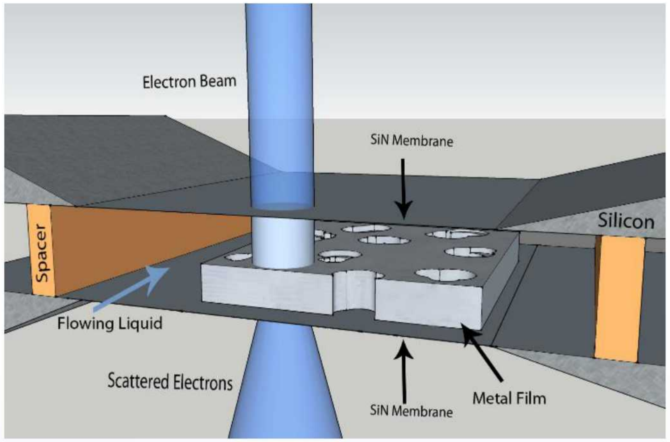


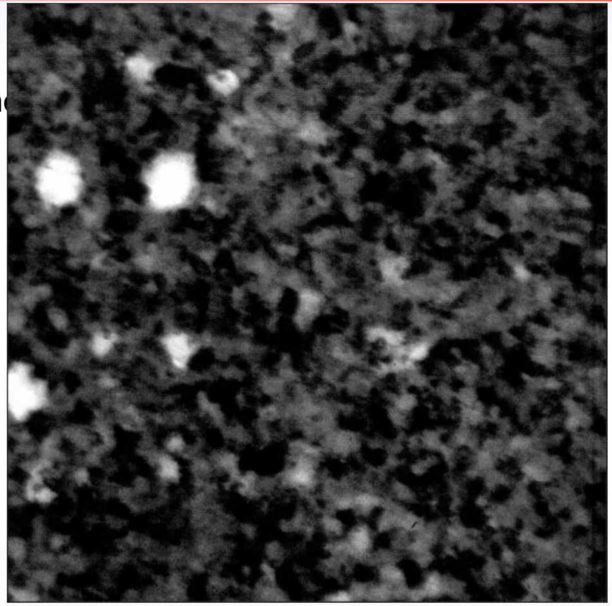
Can We Gain Insight into the Corrosion Process through *In situ* TEM?

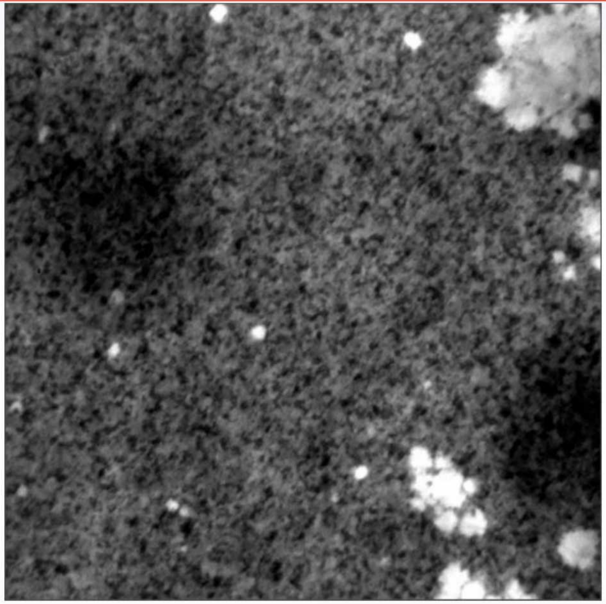
Contributors: D. Gross, J. Kacher, I.M. Robertson & Protochips, Inc.

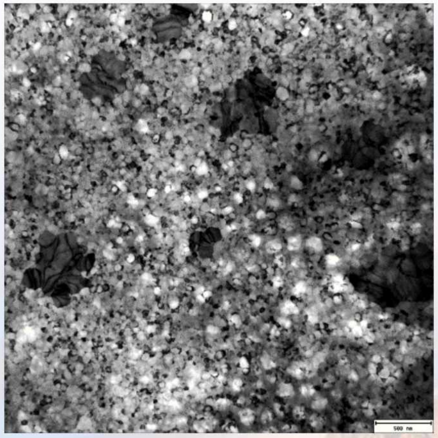
Microfluidic Stage

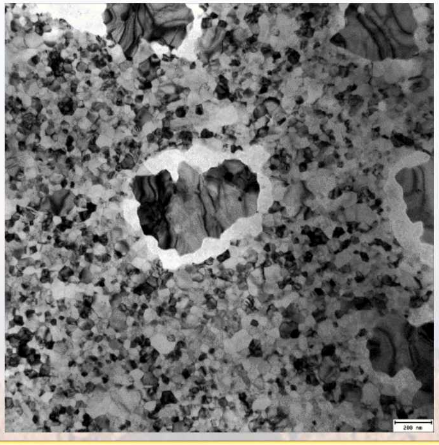
- Mixing of two or more channels
- Continuous observation of the reaction chann

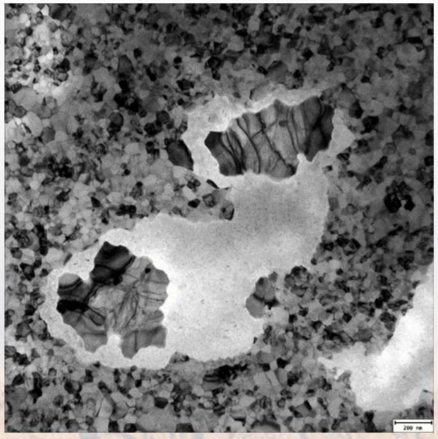


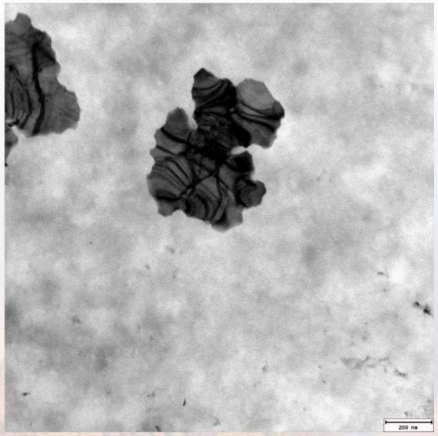












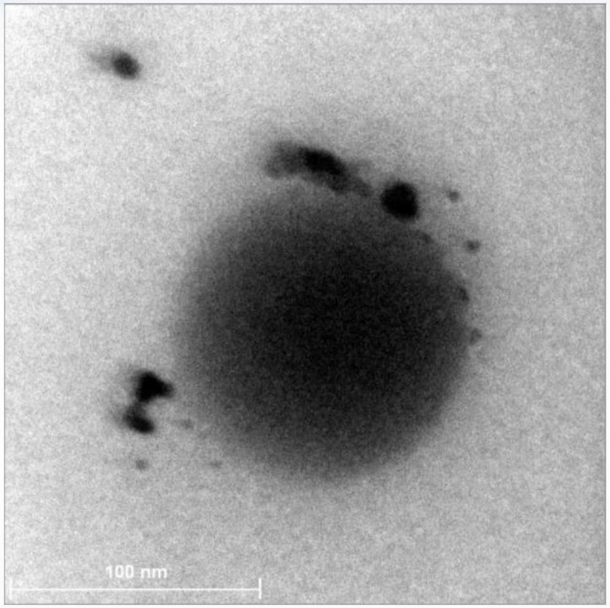
Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.
Large grains resulting from annealing appear more corrosion tolerant

Other Fun Uses of Microfluidic Cell

Protocell Drug Delivery

S. Hoppe,
E. Carnes,
J. Brinker

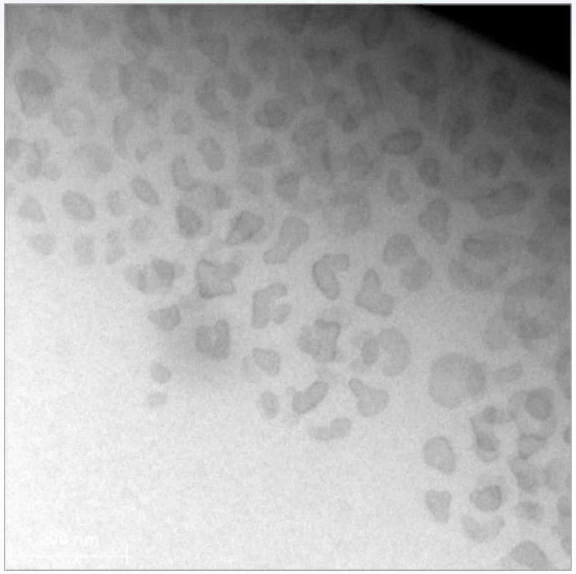
Liposome encapsulated Silica destroyed by the electron beam



BSA Crystallization

S. Hoppe

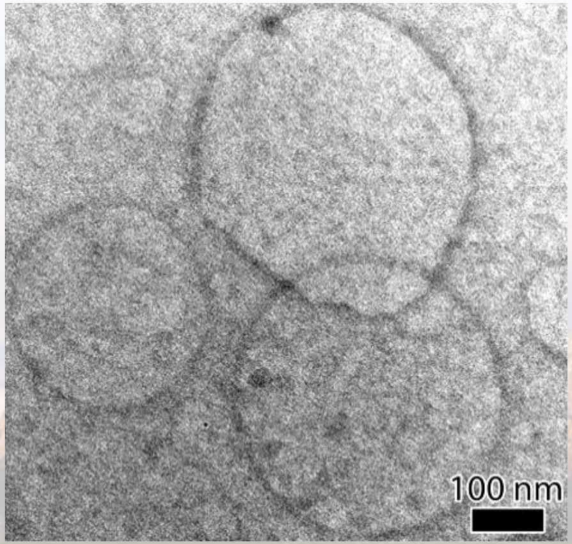
Crystallization of excess Bovine Serum Albumen during flow



Liposomes in Water

S. Hoppe,
D. Sasaki

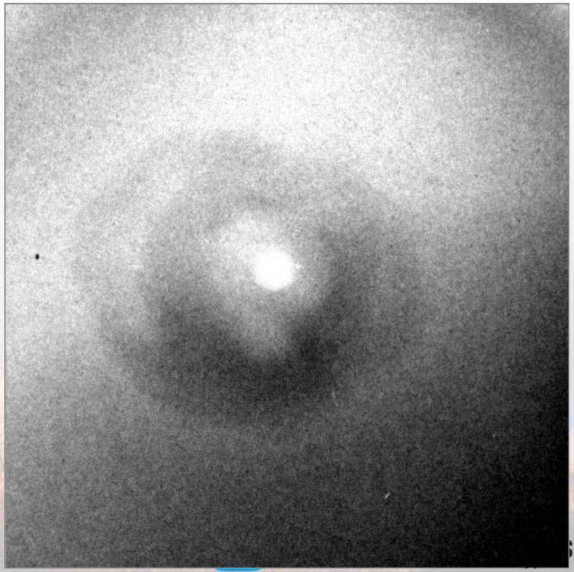
Liposomes imaged in flowing aqueous channel



La Structure Formation

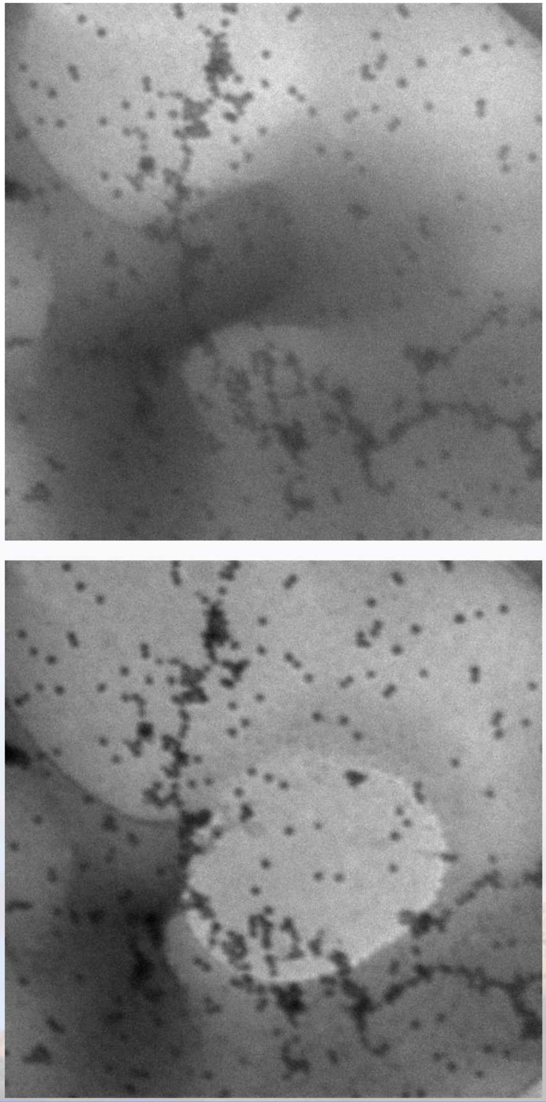
S. Hoppe,
T. Nenoff

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



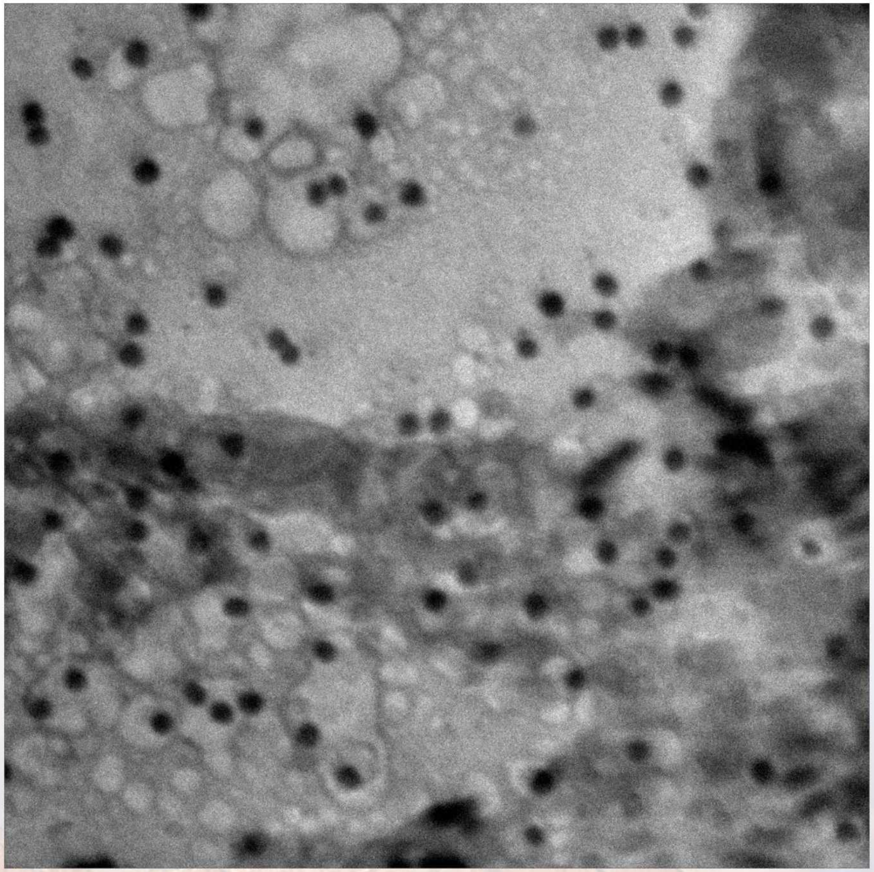
In Situ TEM observation of three-phase mixture

Collaborator: Sarah Pratt



A pair of images before and after radiolysis from focusing the electron beam

Au nanoparticles, aqueous solution, and gas bubbles



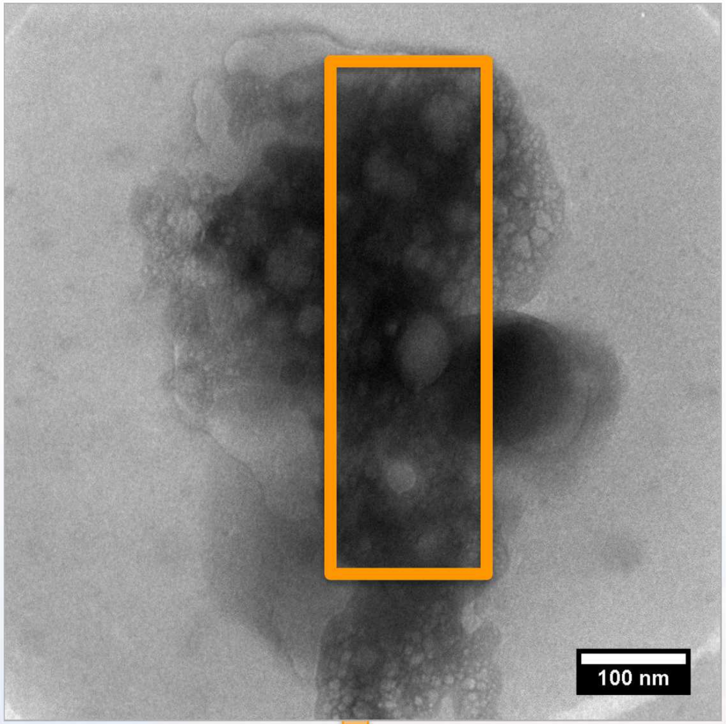
Sandia National Laboratories

PED in Liquid Cell Environment

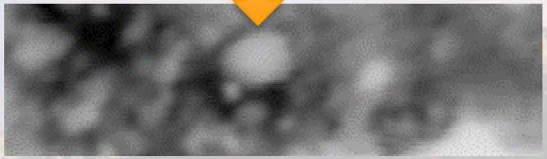
Contributors: C. Taylor, S. Pratt, & T. Nenoff

$LaCl_3 \cdot 7H_2O$: 10 H_2O : PED

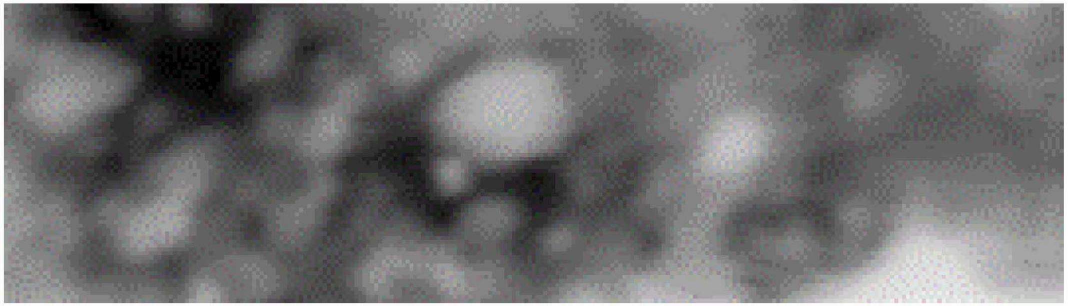
TEM Image



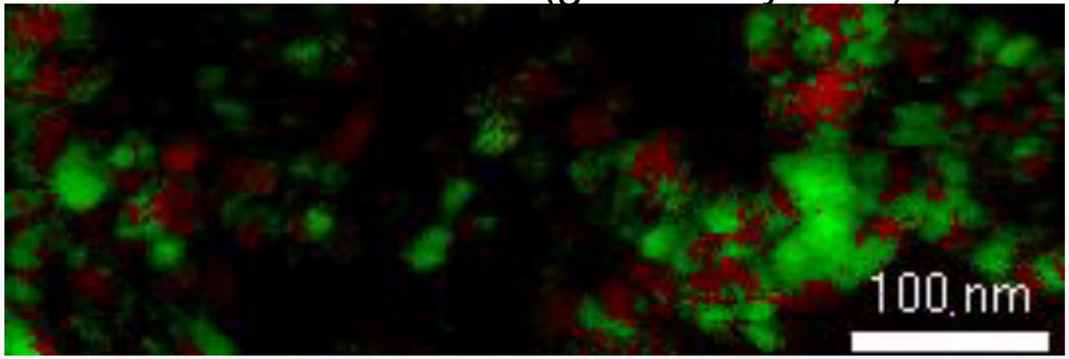
Virtual BF



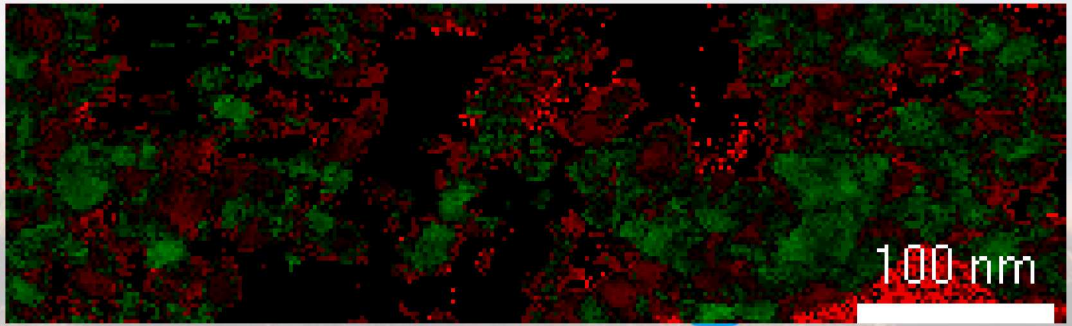
Virtual BF



Index + Phase (green = hydride)

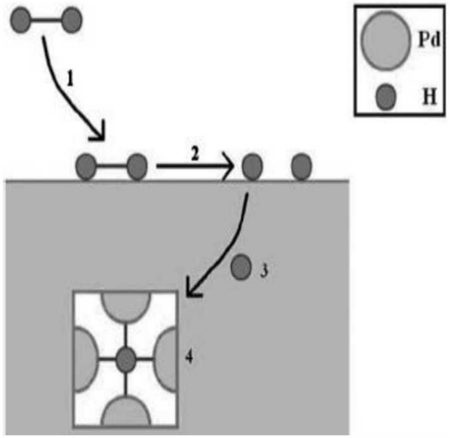


Phase Reliability + Phase

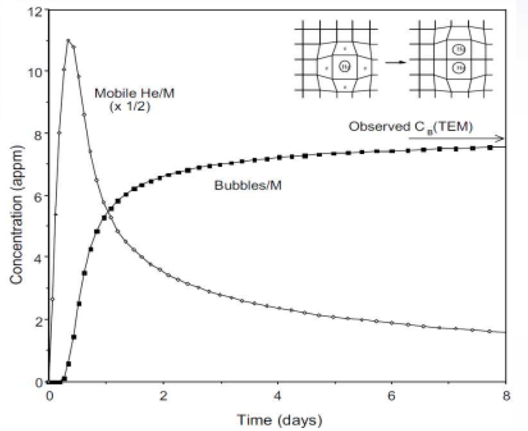


In situ TEM Hydrogen Exposure

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



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

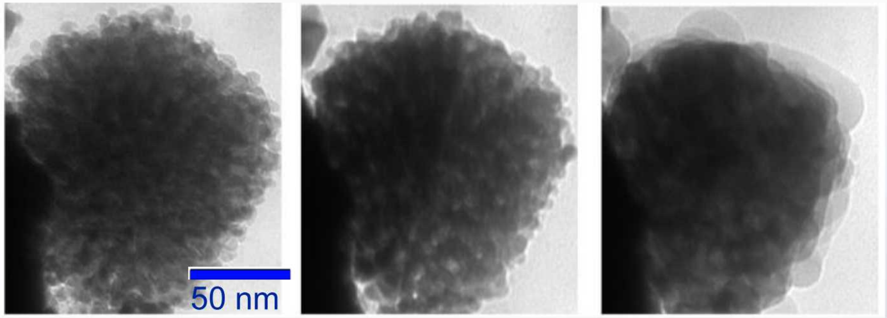


Cowgill, D., Fusion Sci. & Tech., 28 (2005) p. 539
Trinkaus, H. *et al*., JNM (2003) p. 229
Thiebaut, S. *et al*. JNM (2000) p. 217

Vapor-Phase Heating TEM Stage

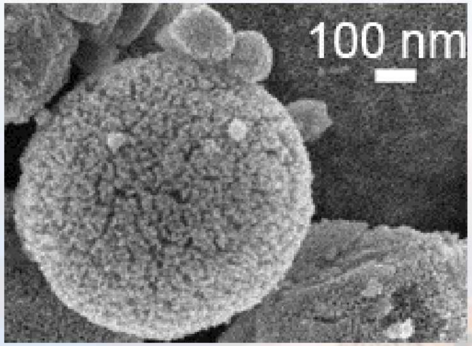
- Compatible with a range of gases
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Compatible with MS and other analytical tools

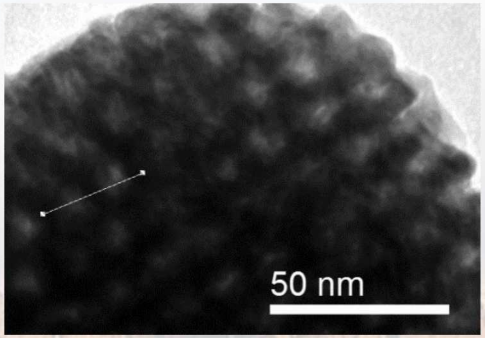
- 1 atm H_2 after several pulses to specified temp.



125° 200° 300°

Harmful effects may be mitigated in nanoporous Pd



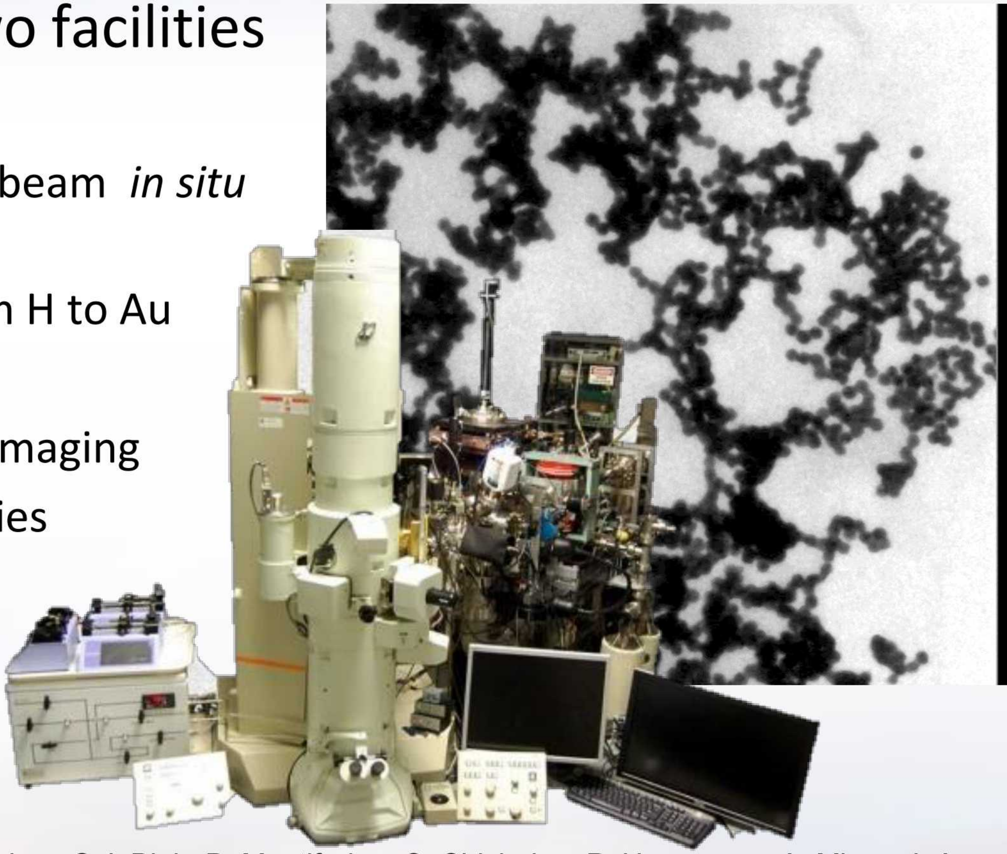


New *in situ* atmospheric heating experiments provide great insight into nanoporous Pd stability

Unconventional *In situ* Microscopy Creates a wealth of Possibilities

Nuclear Science User Facilities

- Sandia's I^{3}TEM is one of only two facilities in the US
  - Only facility in the world with a triple beam *in situ ion irradiation* capabilities
  - *In situ* high energy ion irradiation from H to Au
  - *In situ* gas implantation
  - Dynamic TEM and time compression imaging
  - 11+ TEM stages with various capabilities



Currently applying the I^{3}TEM capabilities to various material systems in combined environmental conditions

Collaborators:

D.L. Buller, D.C. Bufford, S.H. Pratt, T.J. Boyle, B.A. Hernandez-Sanchez, S.J. Blair, B. Muntifering, C. Chisholm , P. Hosemann, A. Minor, J. A. Hinks, F. Hibberd, A. Ilinov, D. C. Bufford, F. Djurabekova, G. Greaves, A. Kuronen, S. E. Donnelly, K. Nordlund, F. Abdeljawad, S.M. Foiles, J. Qu, C. Taylor, J. Sugar, P. Price, C.M. Barr, D. Adams, M. Abere, L. Treadwell, A. Cook, A. Monterrosa, IDES Inc, J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, W. Mook, Hysitron Inc., G.S. Jawaharram, S. Dillon, R.S. Averback, N. Heckman, J. Carroll, S. Briggs, E. Carnes, J. Brinker, D. Sassaki, T. Nenoff, B.G. Clark, P.J. Cappillino, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, L.R. Parent, I. Arslan. & Protochips

This work was partially funded by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. DOE's National Nuclear Security Administration under contract DE-NA-0003525. The views expressed in the article do not necessarily represent the views of the U.S. DOE or the United States Government.

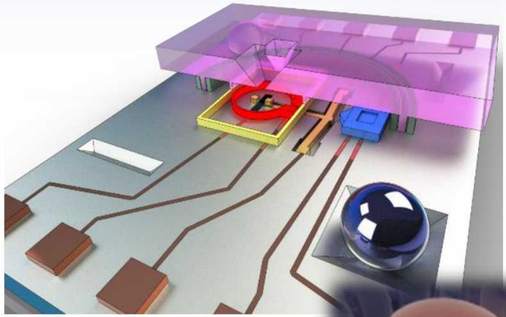
U.S. DEPARTMENT OF ENERGY | Office of Science

Sandia National Laboratories

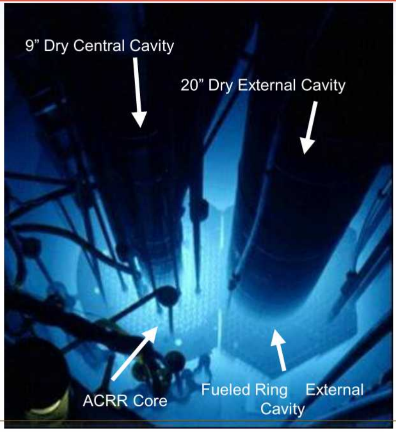
Sandia's USER Capabilities

D. Hanson, W. Martin, M. Wasiolek

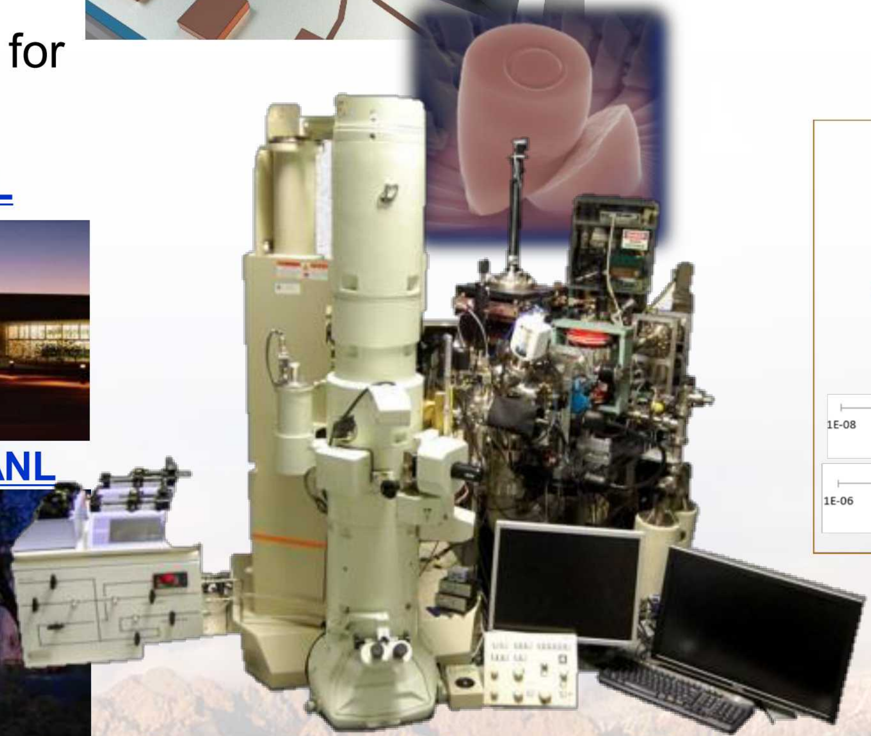
www.cint.lanl.gov
- Spring and Fall proposals for 18 months
- Rapid Access proposal anytime for 3 months

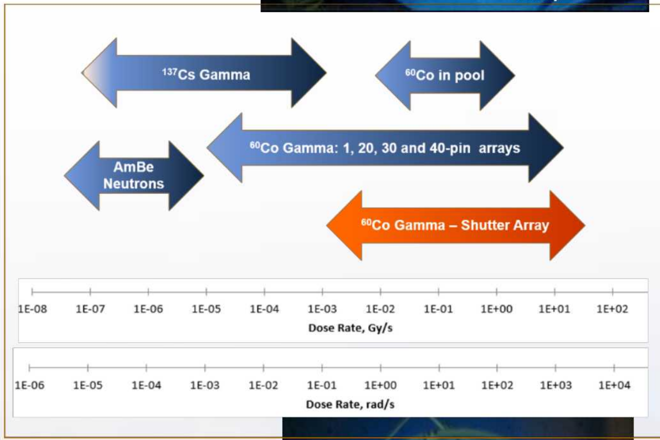


www.nsuf.inl.gov
- Three proposal a year for 9 months

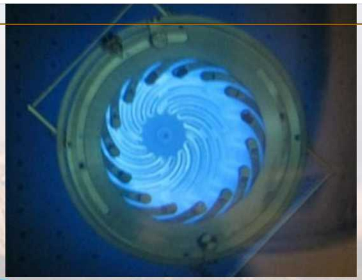


Core Facility - SNL





Gateway Facility - LANL



This work was partially funded by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of EnergyThis work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. DOE's National Nuclear Security Administration under contract DE-NA-0003525. The views expressed in the article do not necessarily represent the views of the U.S. DOE or the United States Government.

Sandia National Laboratories