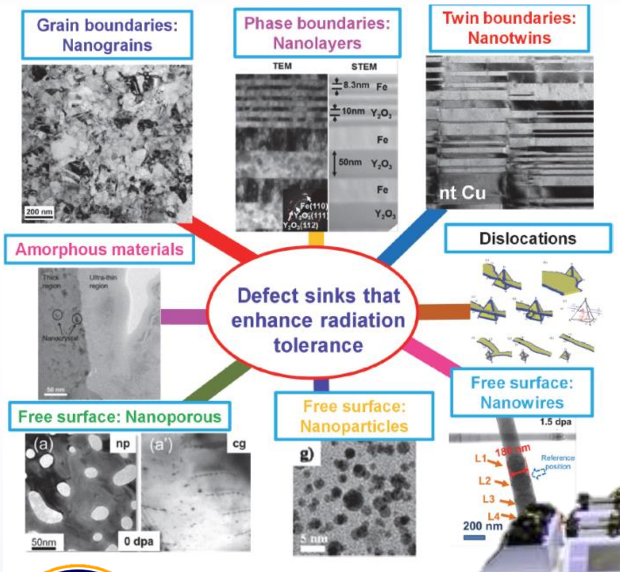
Understanding and Tailoring Nanomaterials for Extreme Environments

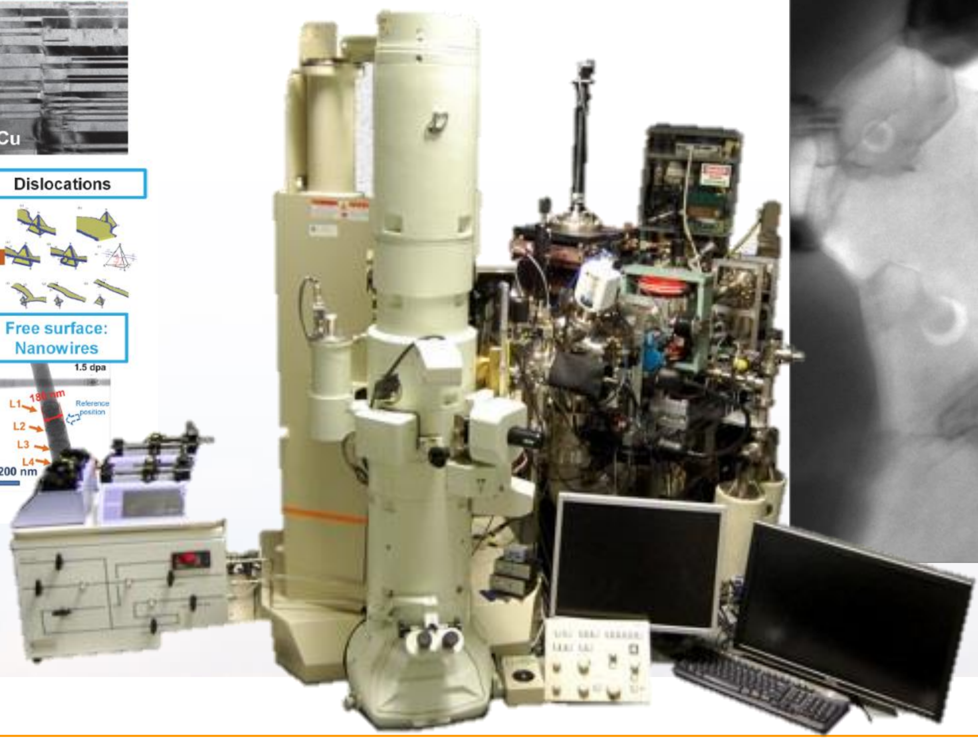
SAND2018-2313PE

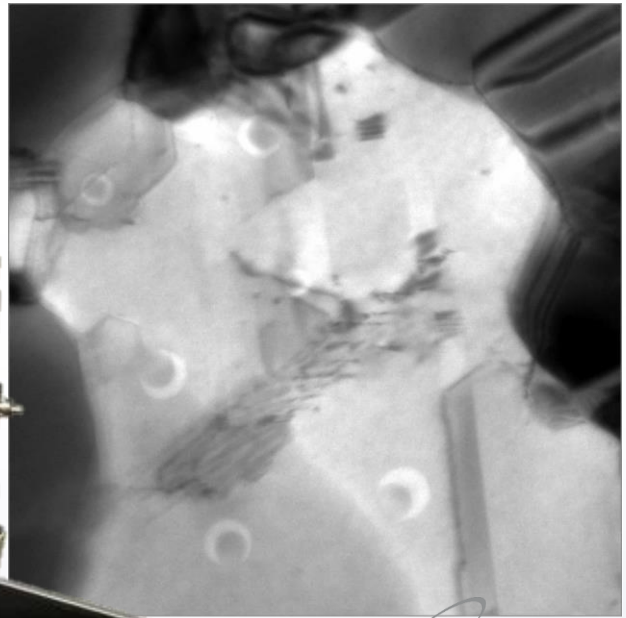
K. Hattar

Ion Beam Lab at Sandia National Laboratories

March 5th, 2018







Collaborators:

- IBL: C.Taylor, C.M. Barr, S.A. Briggs, D.C. Bufford, D. Buller, C. Chisholm, B.G. Clark, M.T. Marshall, B. Muntifering, S.H. Pratt, & P. Price
- Sandia: M. Abere, B. Boyce, T.J. Boyle, R. Dingreville, R.F. Hess, A.C. Kilgo, B.E. Klamm, W.M. Mook, J.D. Puskar, J.A. Scott, & J.A. Sharon
- External: A. Aitkaliyeva, H. Bei, P.J. Ferreira, K.J. Ganesh, E.P. George, D. Gross, P. Hosemann, J. Kacher, S. Maloy, A. Minor, J. Qu, S. Rajesekhara, I.M. Robertson, D. Stauffer, & Hysitron Inc.

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Potential Evolution of System Design

Use the Nearest Stone

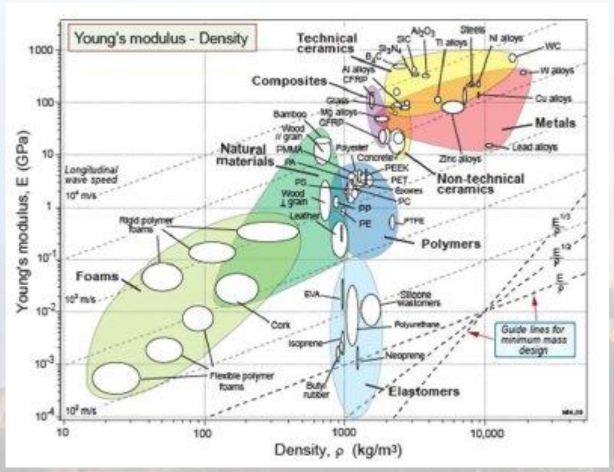
to

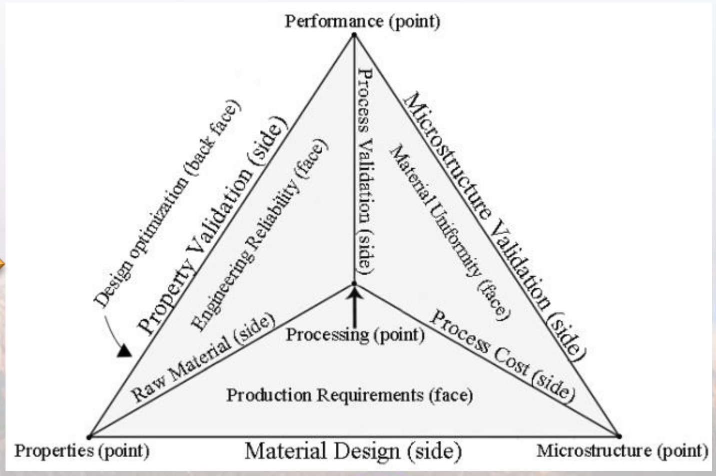
- Radar charts and Ashby plots of current material
- Accelerated and field testing
- **Scientist create a new materials. Engineers find an application.**

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





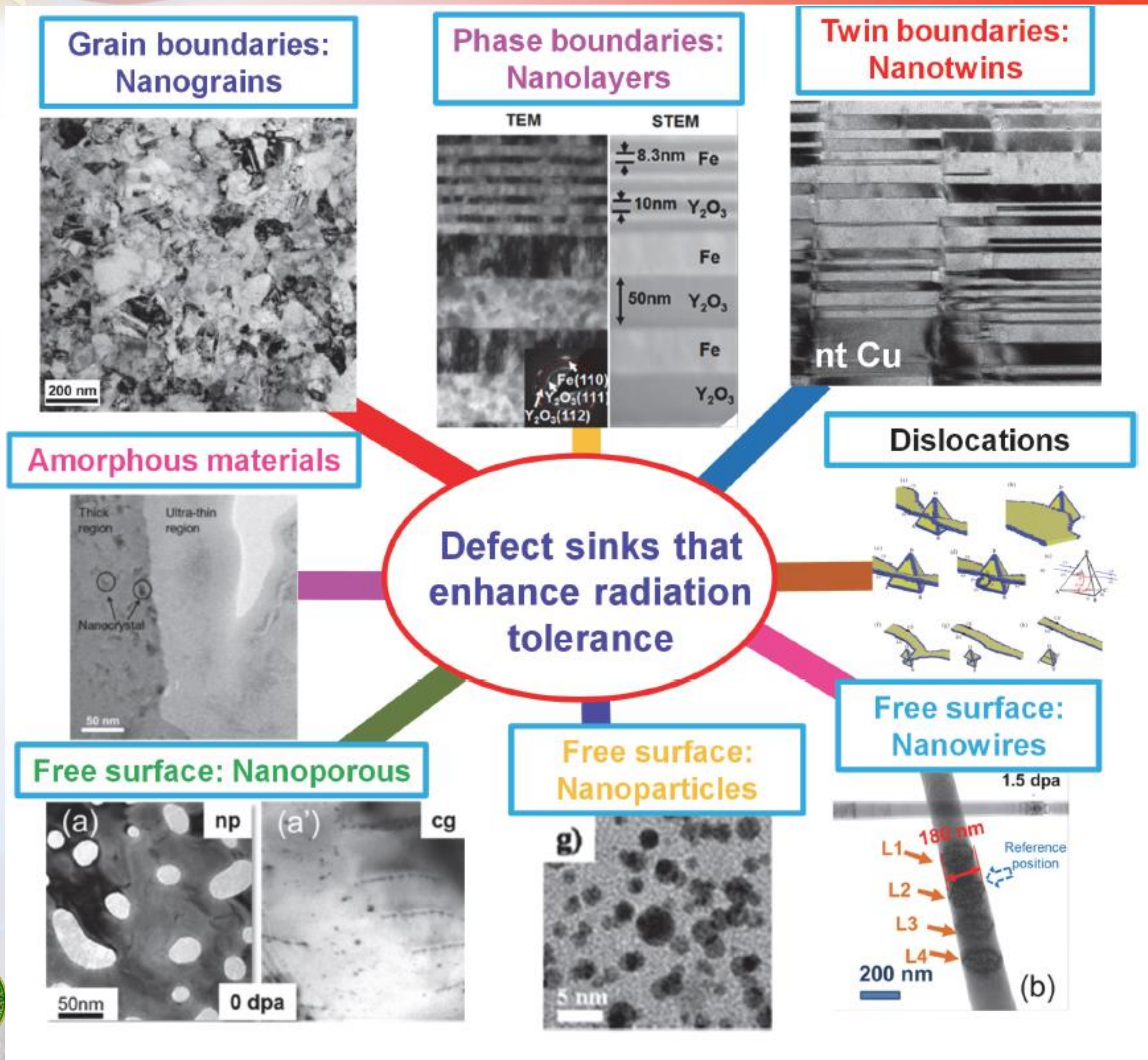
Outline

1. How Does Nanostructuring Effect a Materials Radiation Tolerances?

2. Can We Tailor Boundaries/Interfaces to Control Radiation Tolerances?

3. Can We Explore Other Aspects of the Complex Environment Present in Modern and Future Reactors Using *In situ* Techniques?

Nanomaterials Proposed for Improving Radiation Tolerance

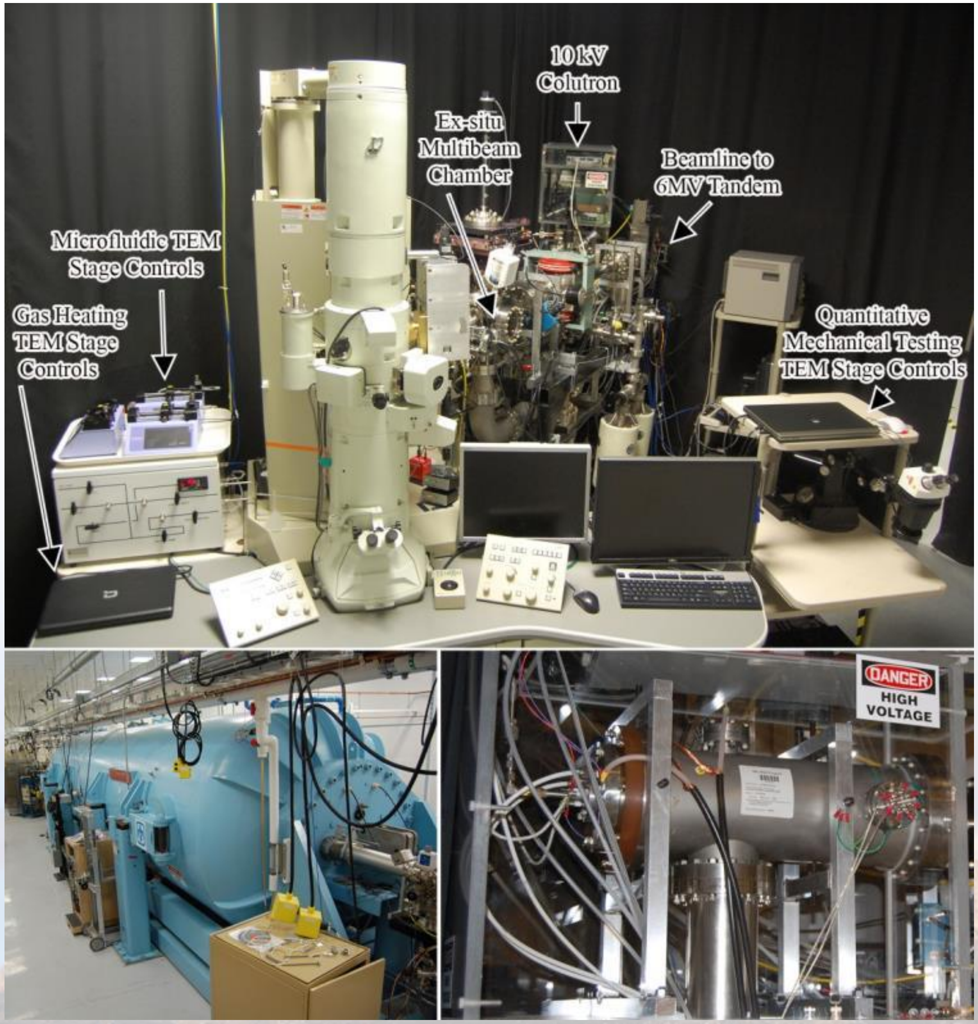


[X. Zhang et al., submitted 2017]

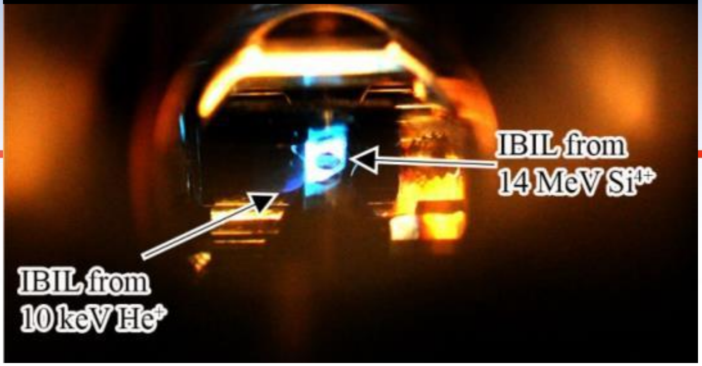
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem

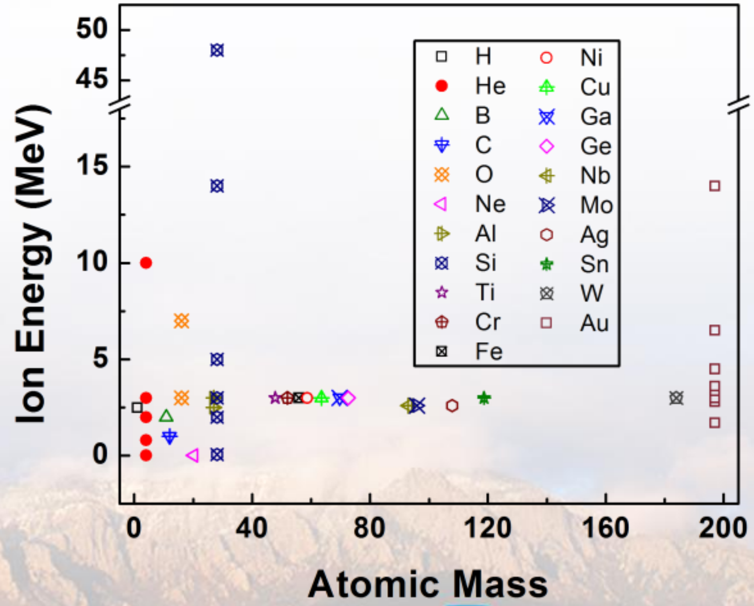


IBIL from a quartz stage inside the TEM



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

Ion species & energy introduced into the TEM



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

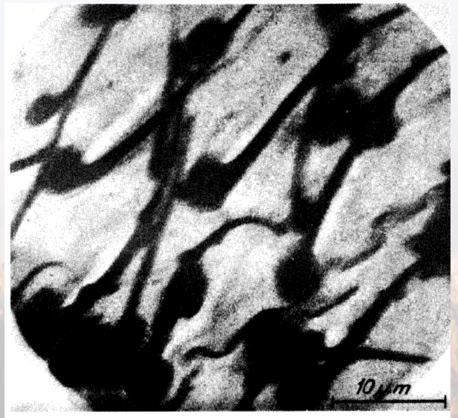


Fig. 6: Wing surface of the house fly.
(First internal photography, U = 60 kV, M_{el} = 2200)
(Driest, E., and Müller, H.O.: Z. Wiss. Mikroskopie 52, 53-57 (1935)

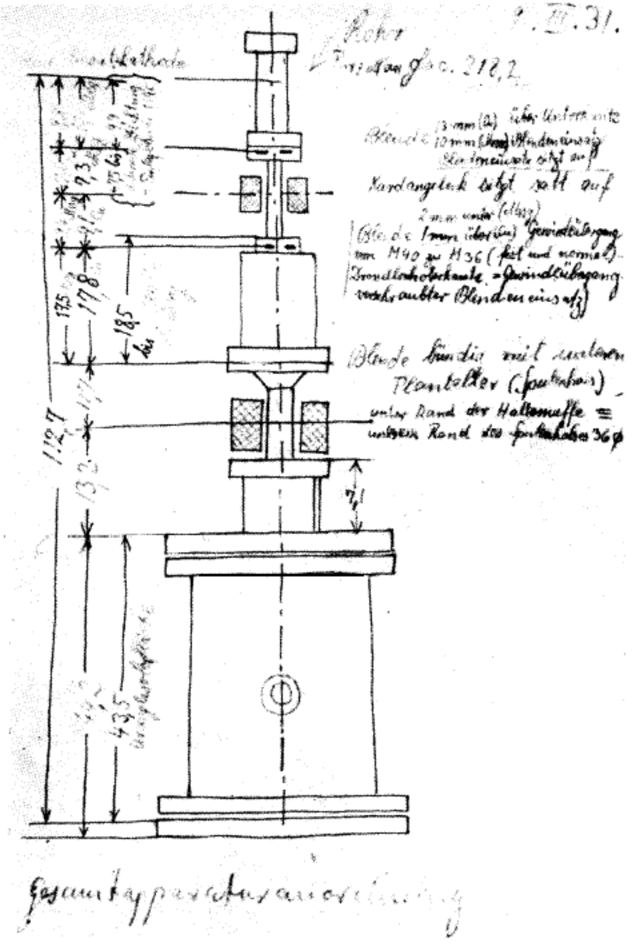
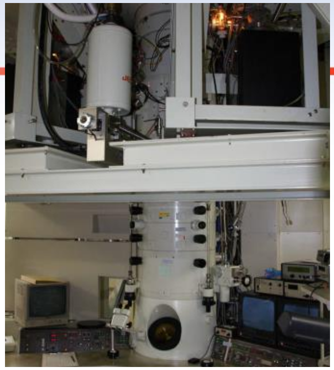


Fig. 2: Sketch by the author (9 March 1931) of the cathode ray tube for testing one-stage and two-stage electron-optical imaging by means of two magnetic electron lenses (electron microscope) [8].

E. Ruska Nobel lecture (1986)

History of *In situ* Ion Irradiation TEM

Courtesy of: J. Hinks



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

1931
The invention of the TEM

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

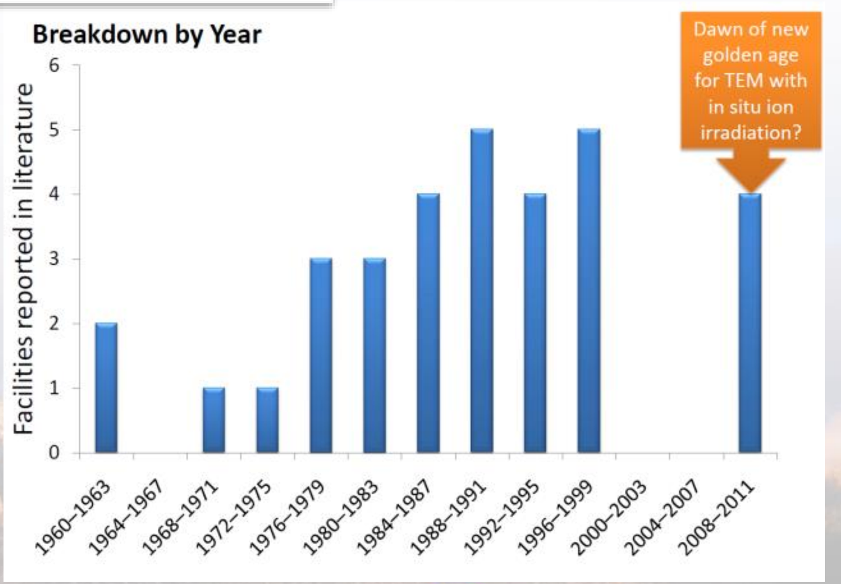
1968
First TEM beamline combination by Thackery, Nelson, and Sansom at AERE Harwell, UK

1976
First HVEM with ion irradiation at UVA, USA

1978
First in-situ ion irradiation experiments at ANL

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

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



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

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

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

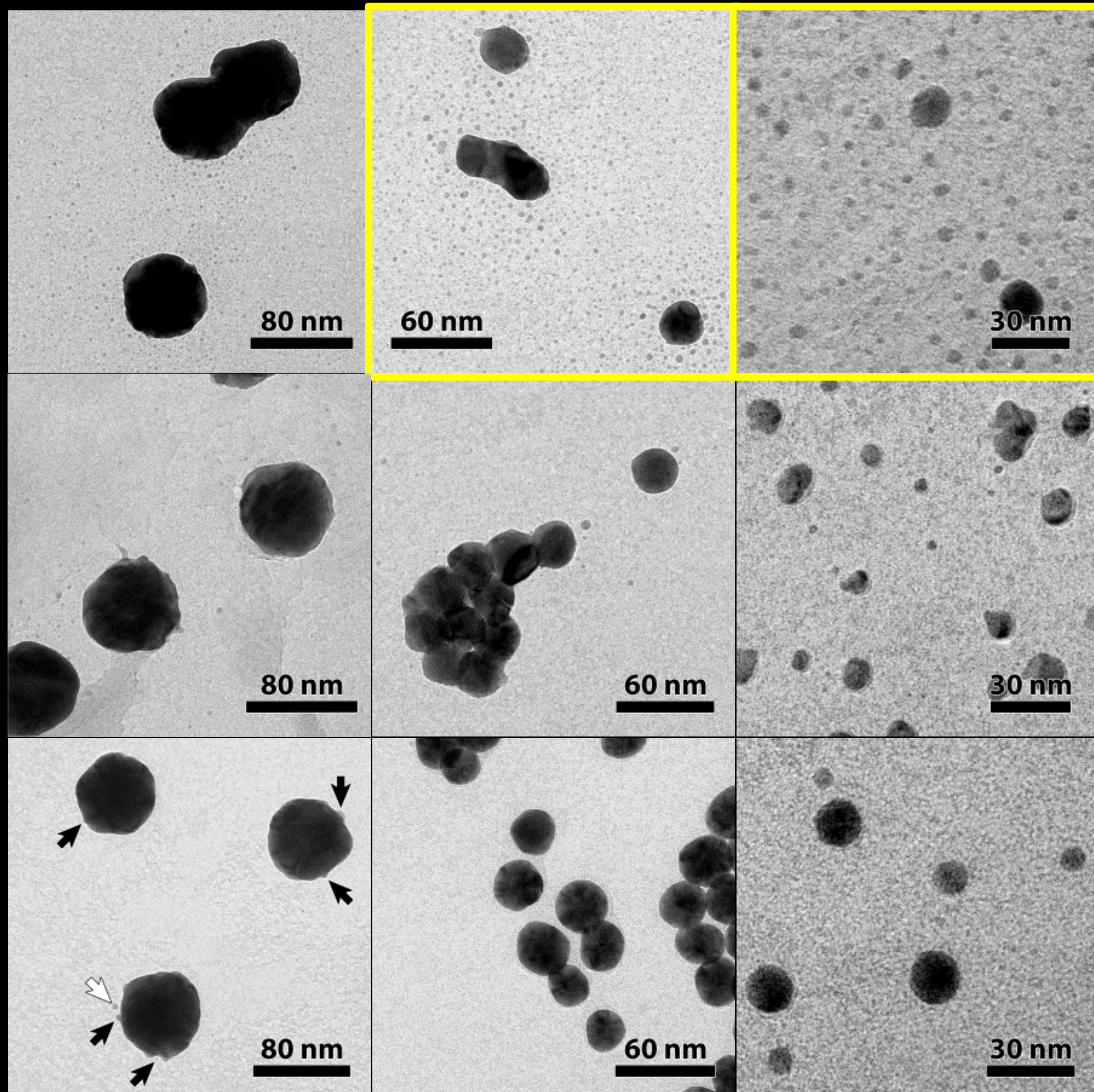
Collaborator: D.C. Bufford

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

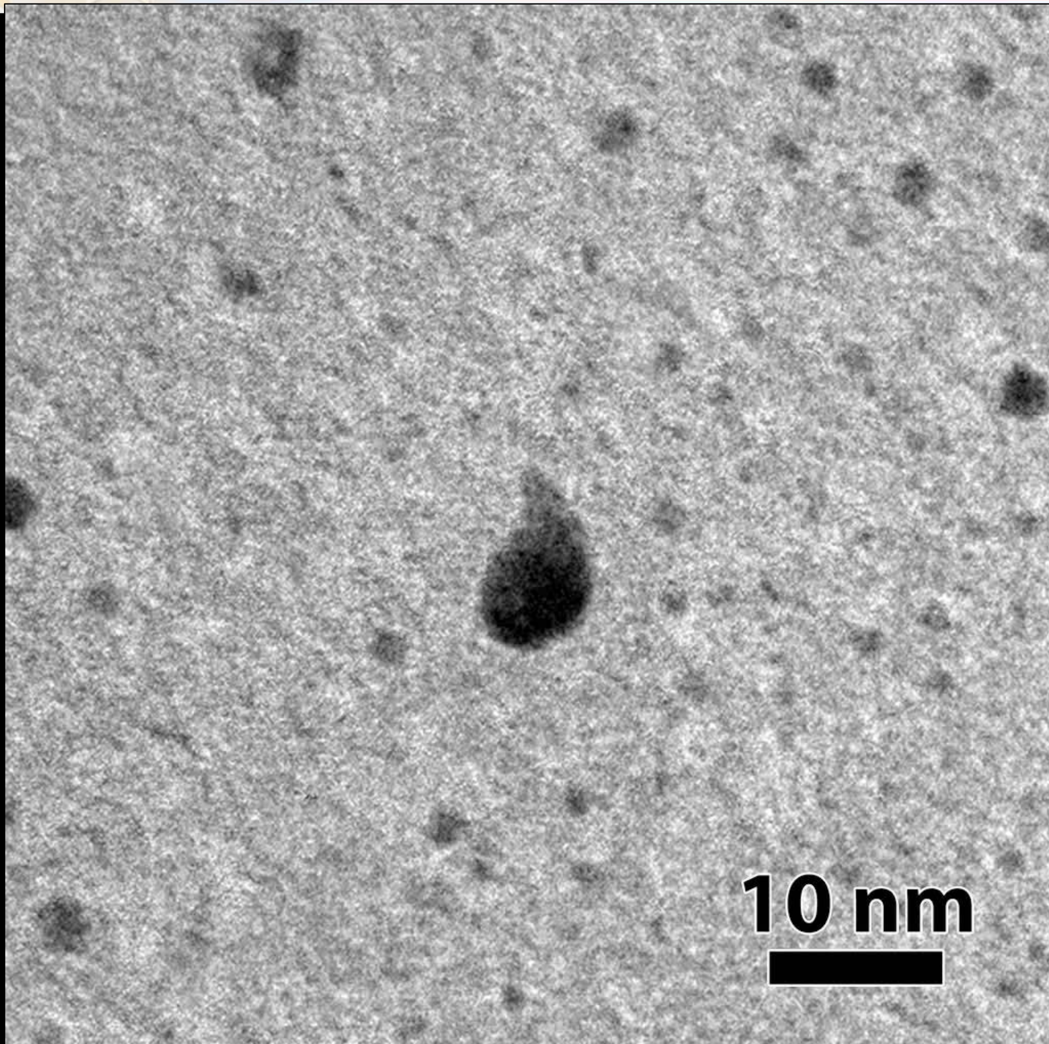
10 MeV Au^{8+}
1.3×10^{12} /cm^2

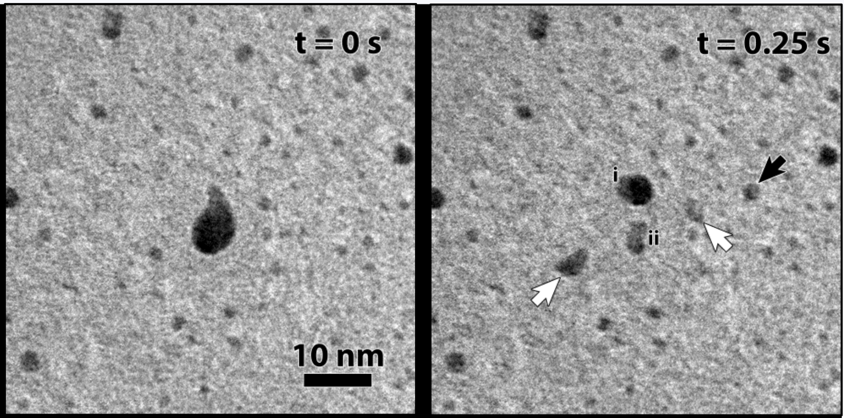


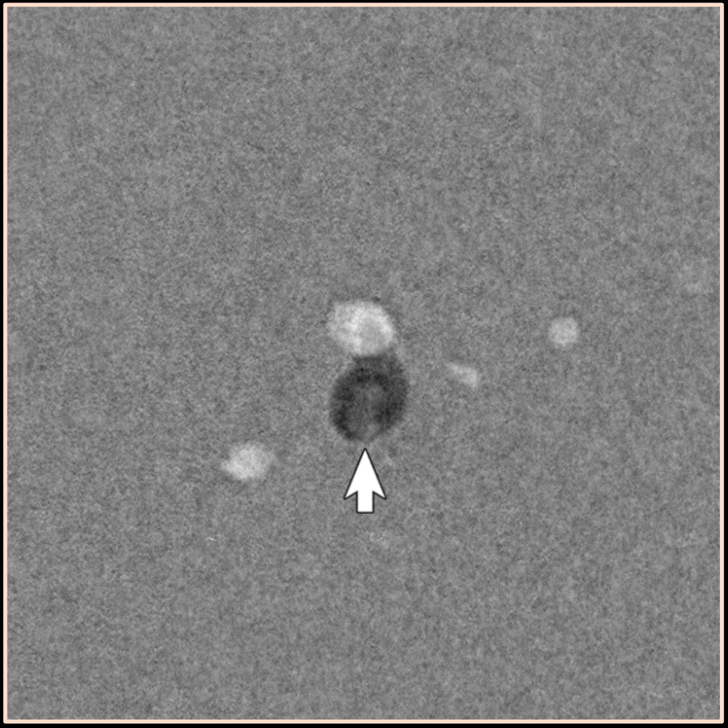
Particle and ion energy dictate the ratio of sputtering, particle motion, particle agglomeration, and <u>other active mechanisms</u>

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

Collaborator: D.C. Bufford

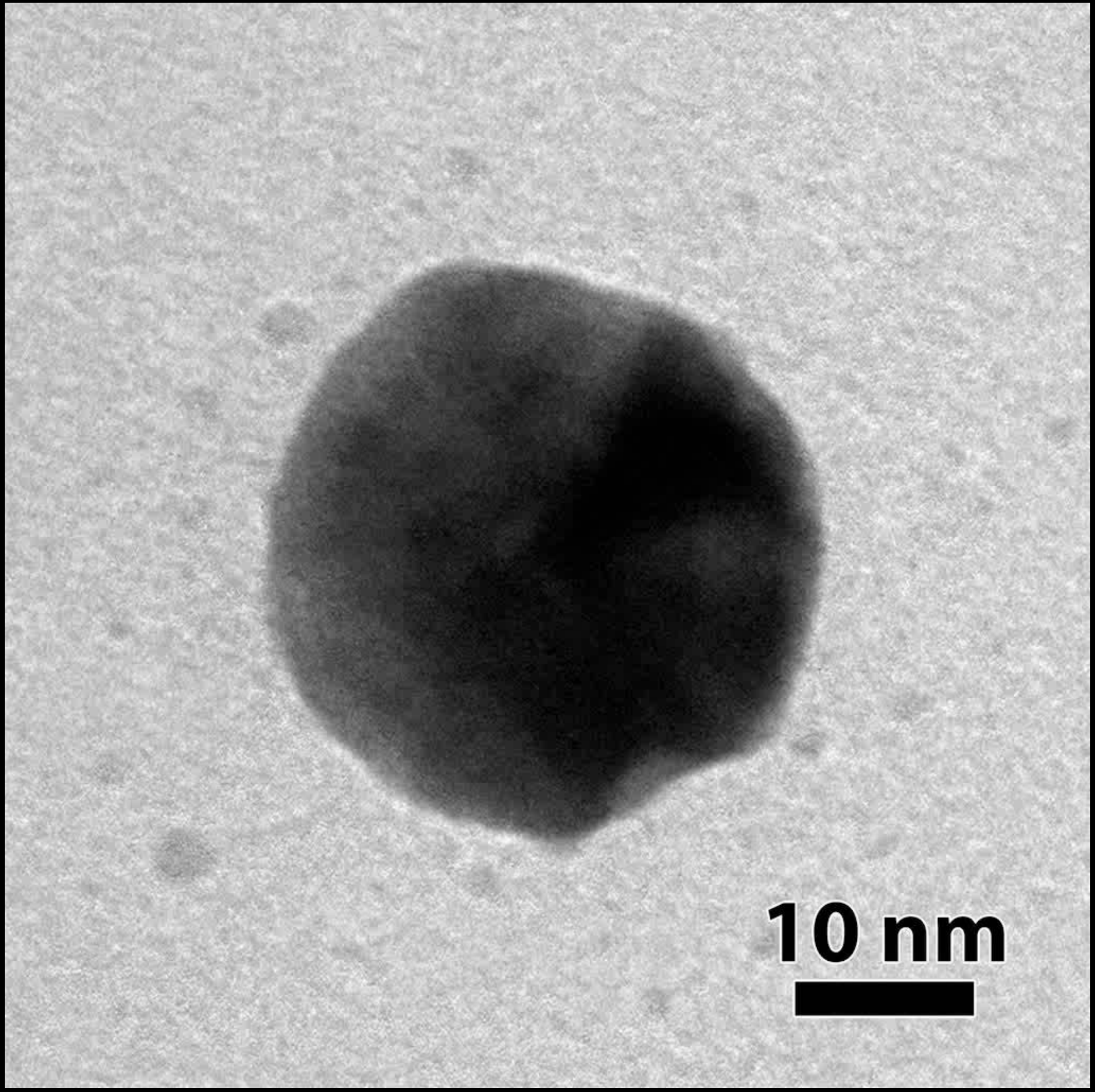


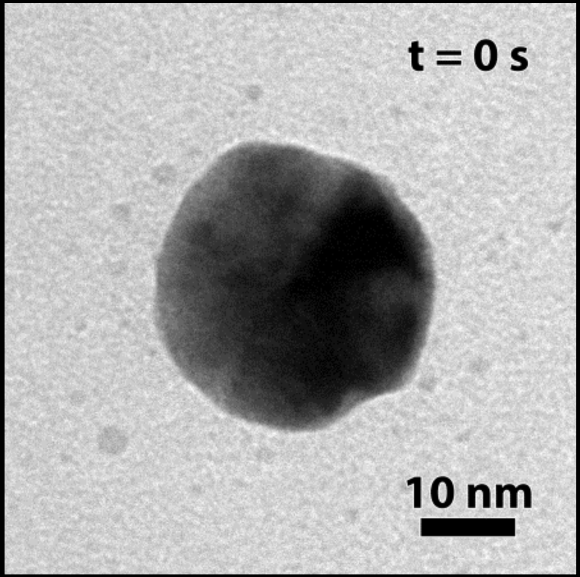


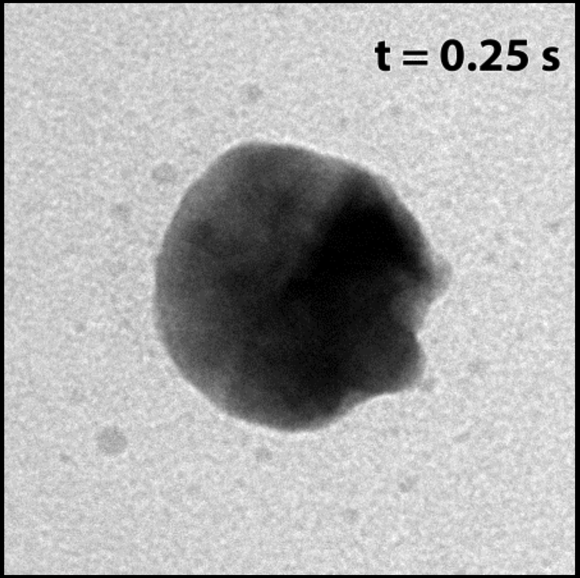


Single Ion Effects with 46 keV Au^{1-} ions: 20 nm

Collaborator: D.C. Bufford

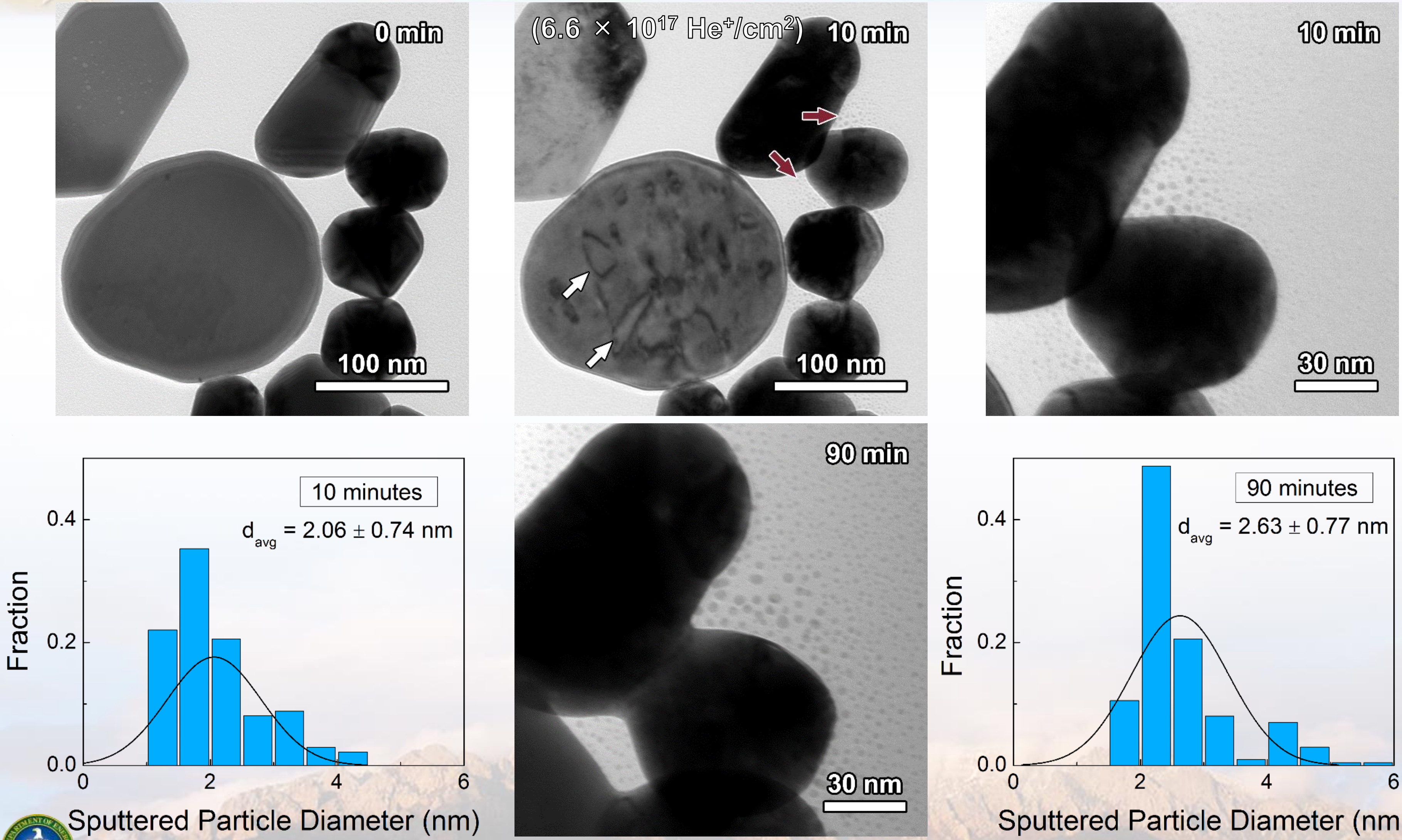






Formation of Dislocation Loops & Sputtered Particles due to He implantation

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



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Electron Tomography Provides 3D Insight

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

***In situ* Ion Irradiation TEM (I³TEM)**

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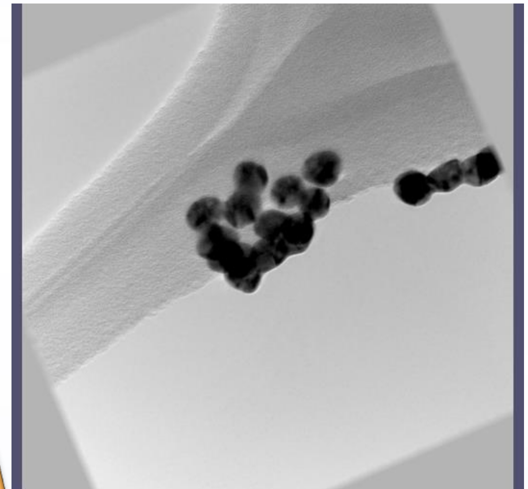
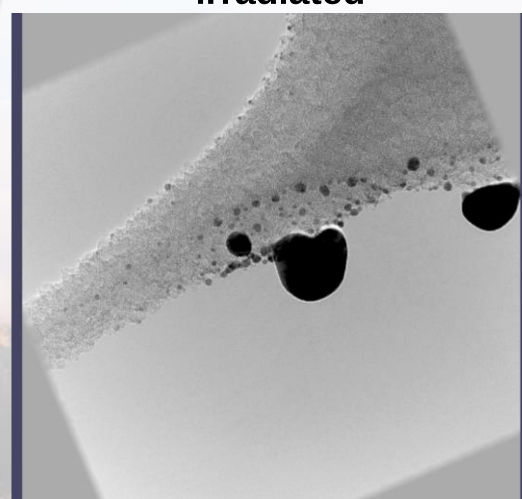
Hummingbird tomography stage

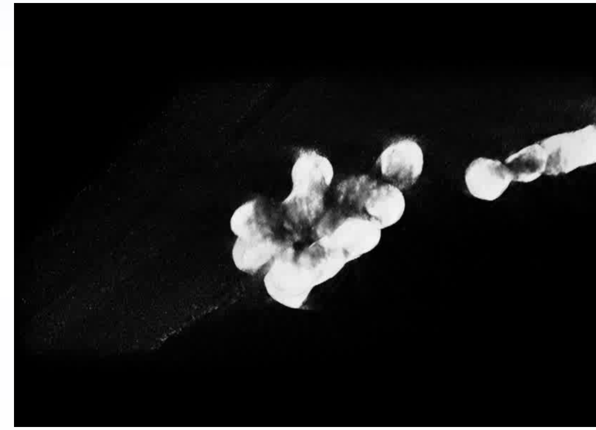
Aligned Au NP tilt series - unirradiated



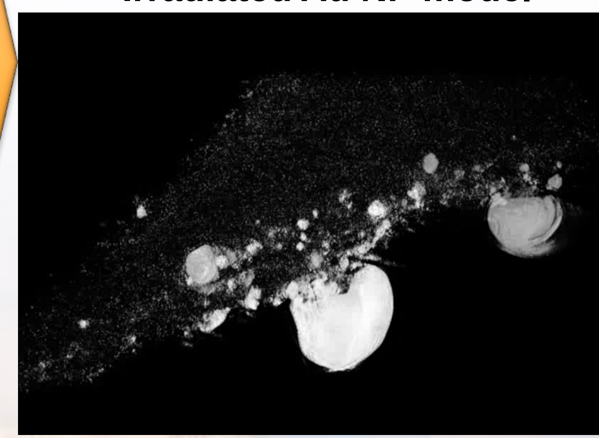
Aligned Au NP tilt series - irradiated



Unirradiated Au NP model



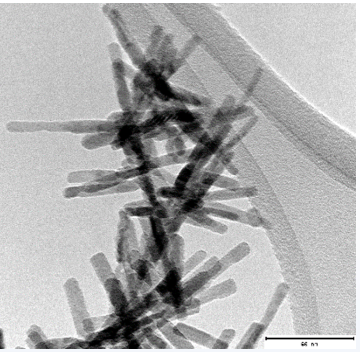
Irradiated Au NP model



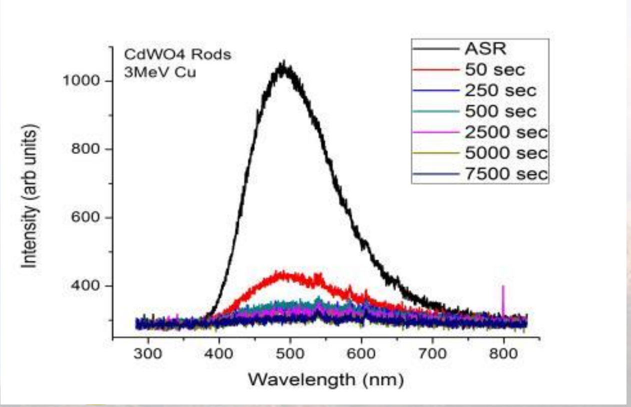
The application of advanced microscopy techniques to extreme environments provides exciting new research directions

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

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



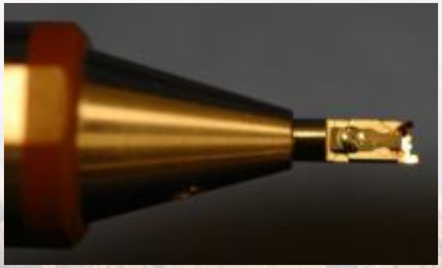
High-Z nanoparticles ($CdWO_4$) are promising, but are radiation sensitive



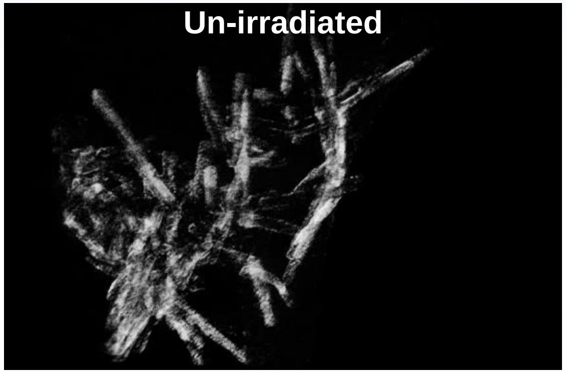
In situ Ion Irradiation TEM (I^3TEM)

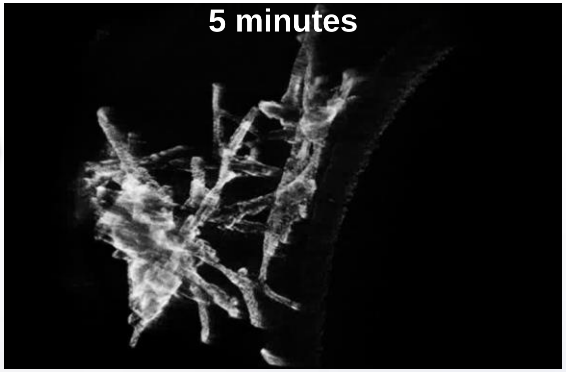
+

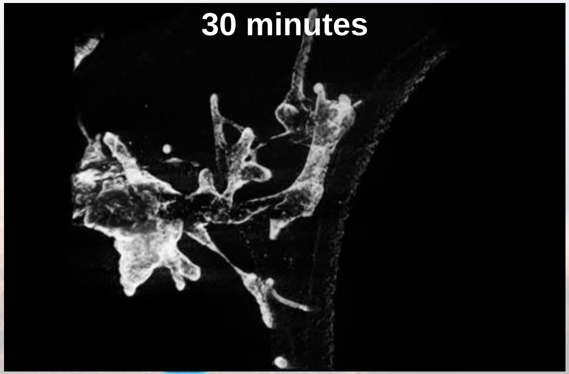
Hummingbird tomography stage



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

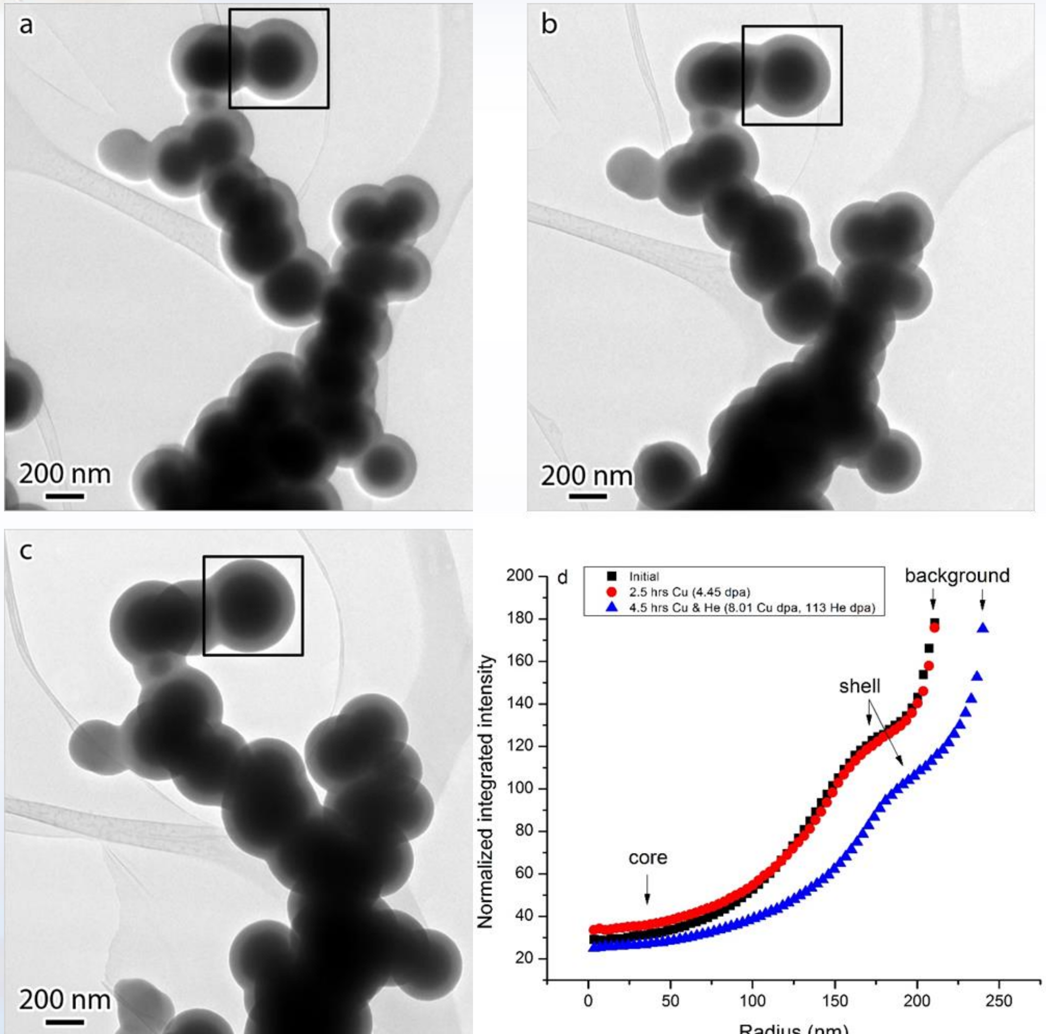


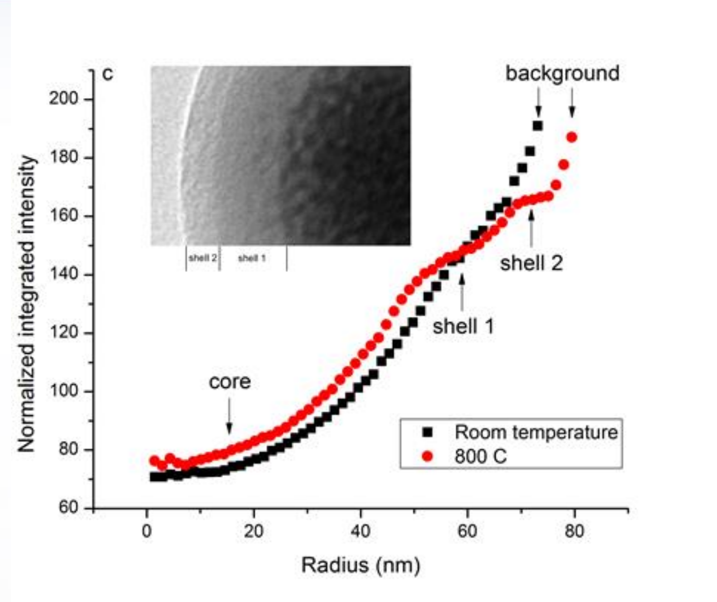




Radiation Stable Nanoparticles

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 x 10^{14} 1.3 x 10^{10}	4.45 4.9 x 10^{-4}
He & Cu	10 3000	2.3 x 10^{18} 1.4 x 10^{14} 2.2 x 10^{14} 1.3 x 10^{10}	113 7.0 x 10^{-3} 8.01 4.9 x 10^{-4}

In contrast some NPs appear to be very radiation stabile

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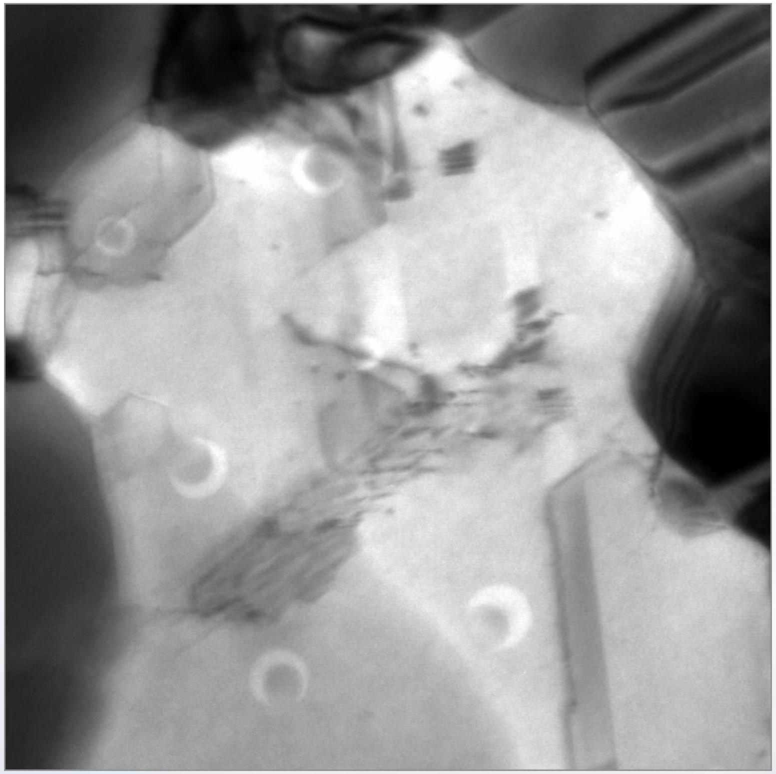
Dose Rate Effects in Nanocrystalline Metals

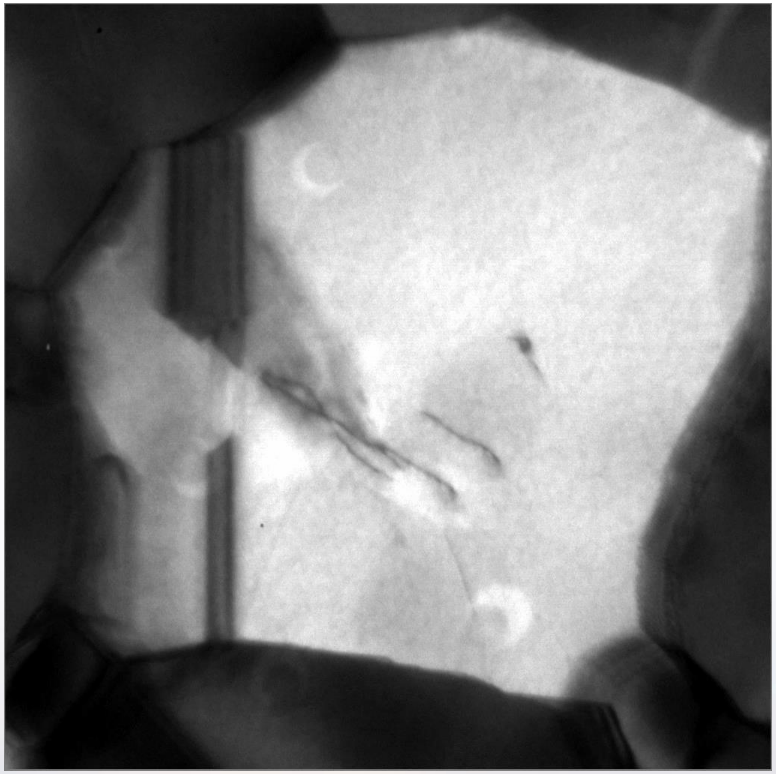
Collaborators: C. Chisholm , P. Hosemann, & A. Minor

7.9×10^9 ions/cm²/s



VS

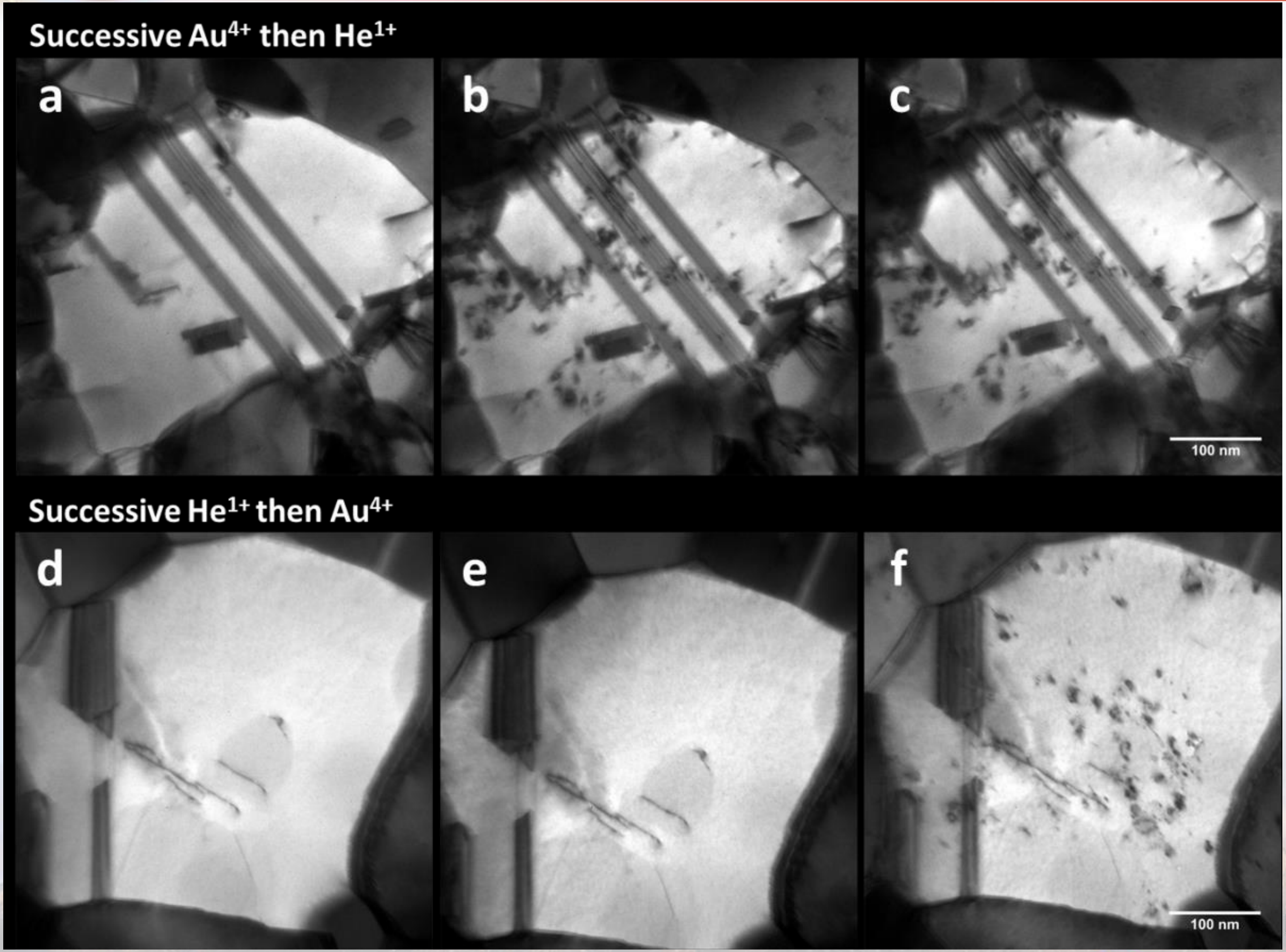
6.7×10^7 ions/cm²/s



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

In situ Successive Implantation & Irradiation

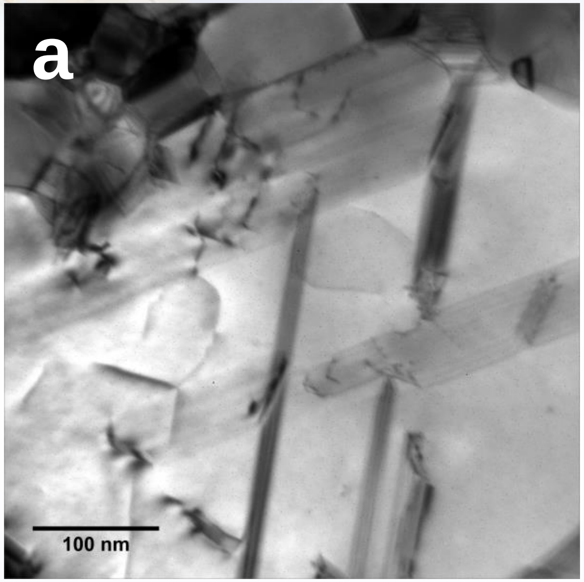
Collaborators: C. Chisholm & A. Minor

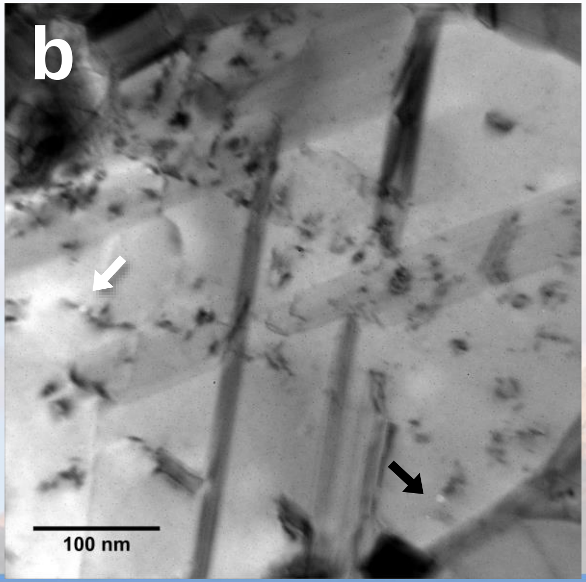


Laboratories

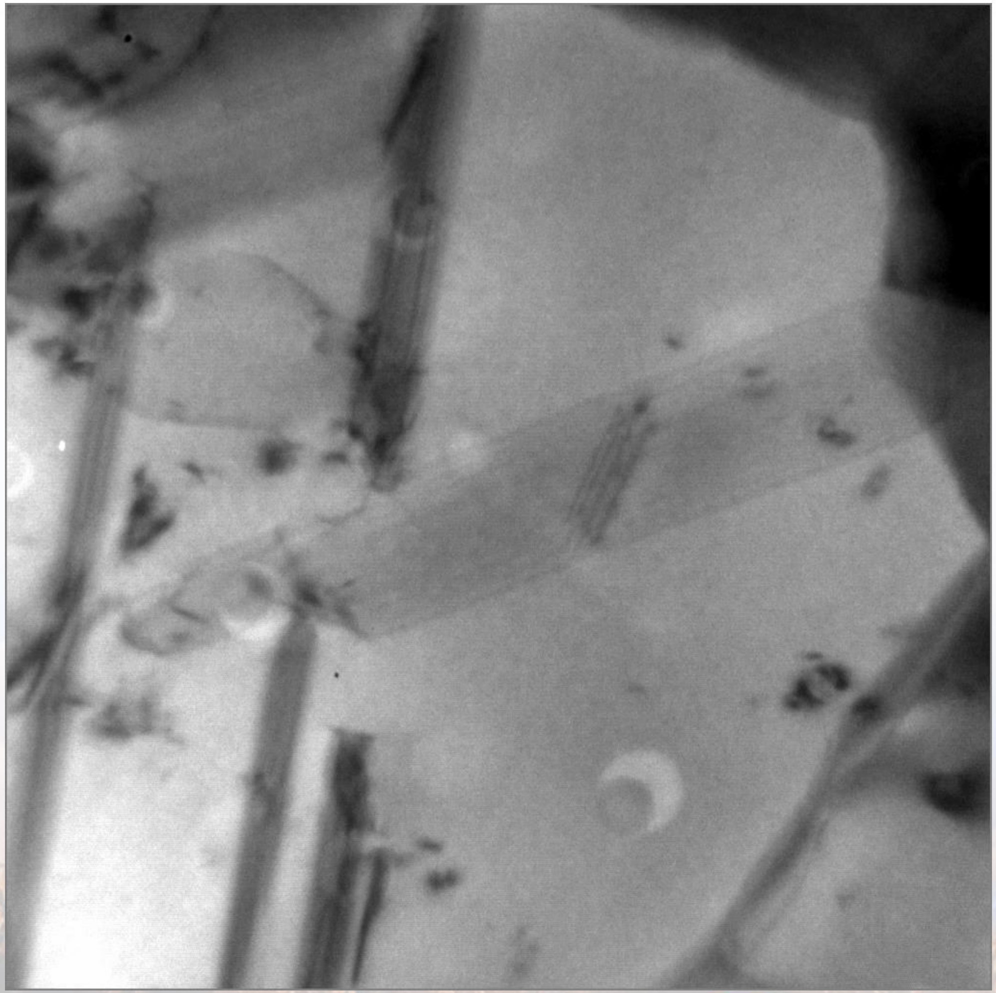
In situ Concurrent Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor





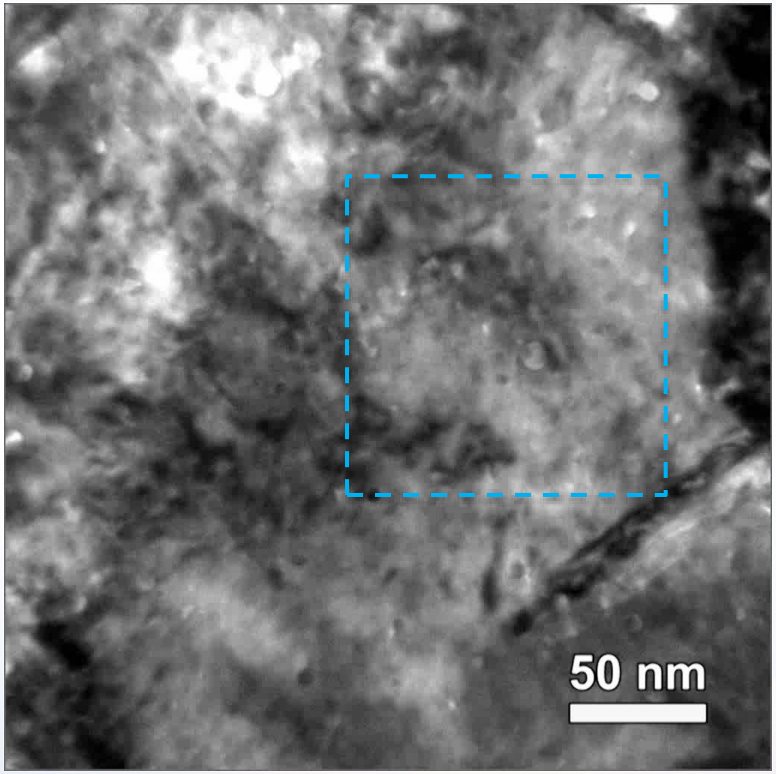
He^{1+} implantation and Au^{4+} irradiation of a gold film

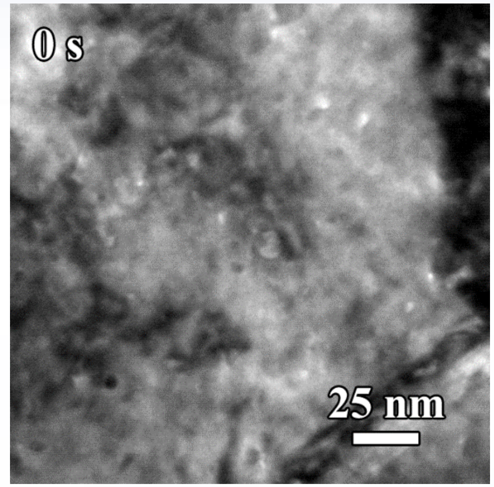


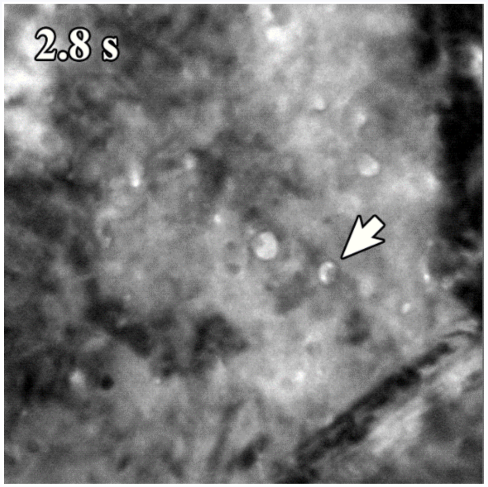
Simultaneous *In situ* TEM Triple Beam: 2.8 MeV Au^{4+} + 10 keV He^{+}/D_2^{+}

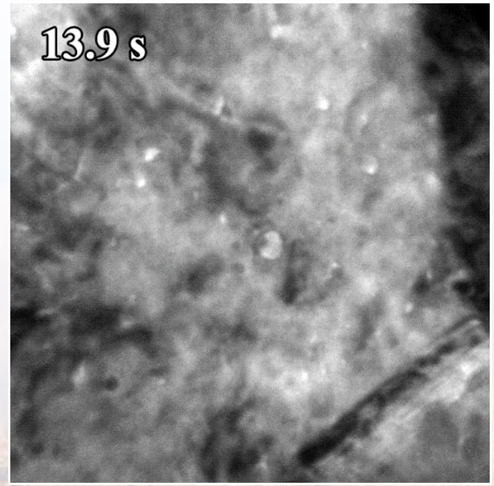
Collaborator: D.C. Bufford

Video playback speed x1.5.







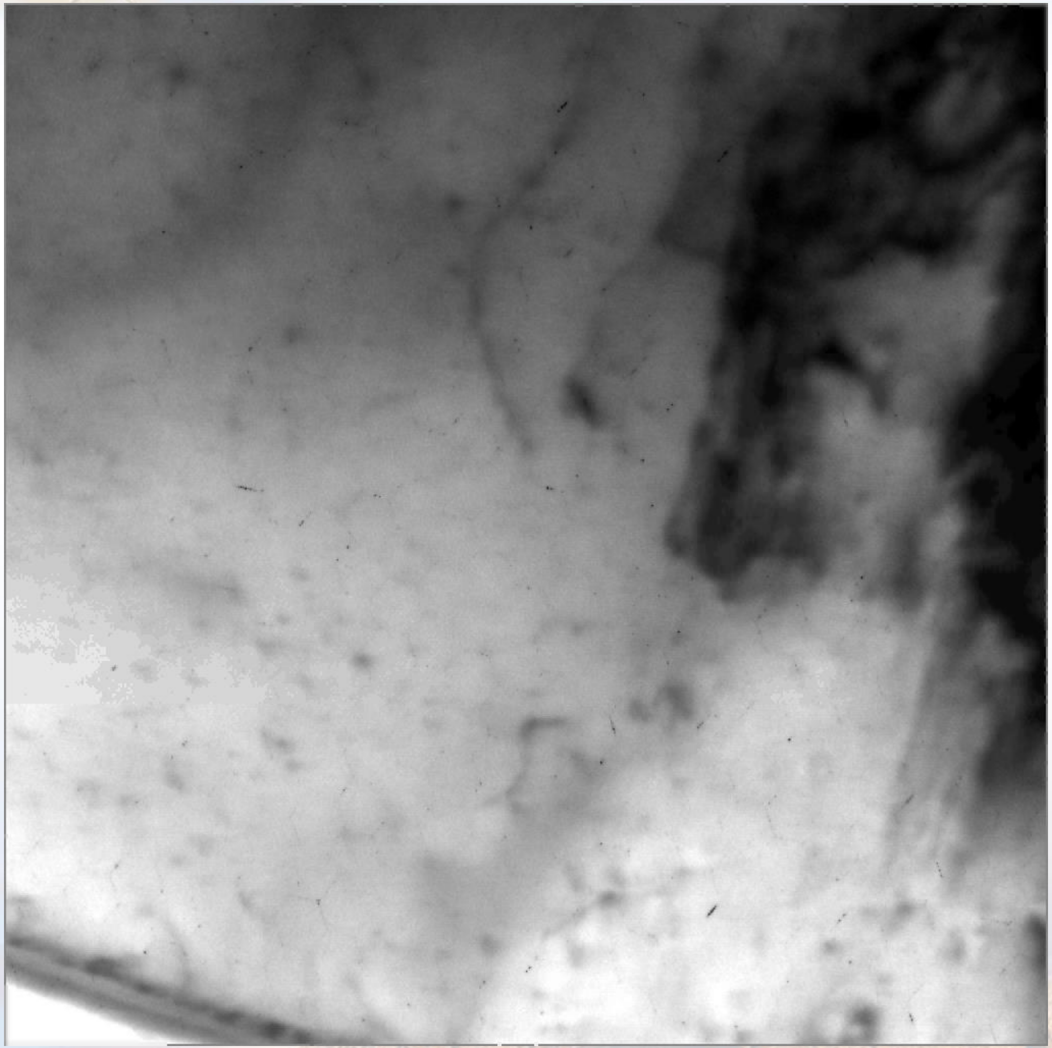


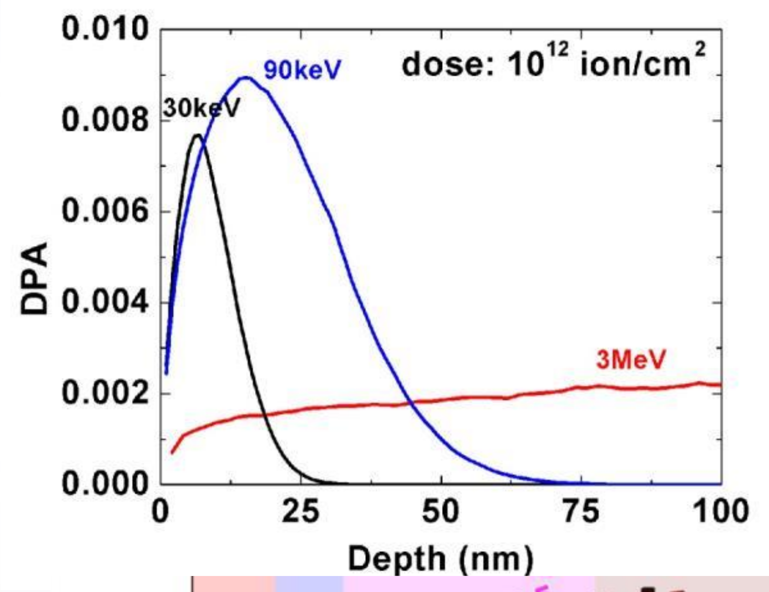
In-situ triple beam He, D_2, and Au beam irradiation has been demonstrated on Sandia's I^3TEM! Intensive work is still needed to understand the defect structure evolution that has been observed.

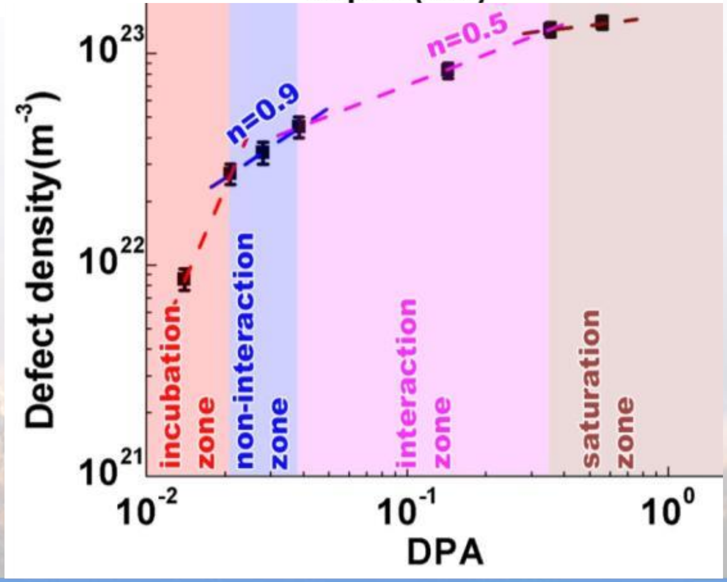
- **Approximate fluence:**
  - Au 1.2 $\times 10^{13}$ ions/cm^2
  - He 1.3 $\times 10^{15}$ ions/cm^2
  - D 2.2 $\times 10^{15}$ ions/cm^2
- **Cavity nucleation and disappearance**

Quantifying Defect Evolution

Collaborators: N. Li & A. Misra

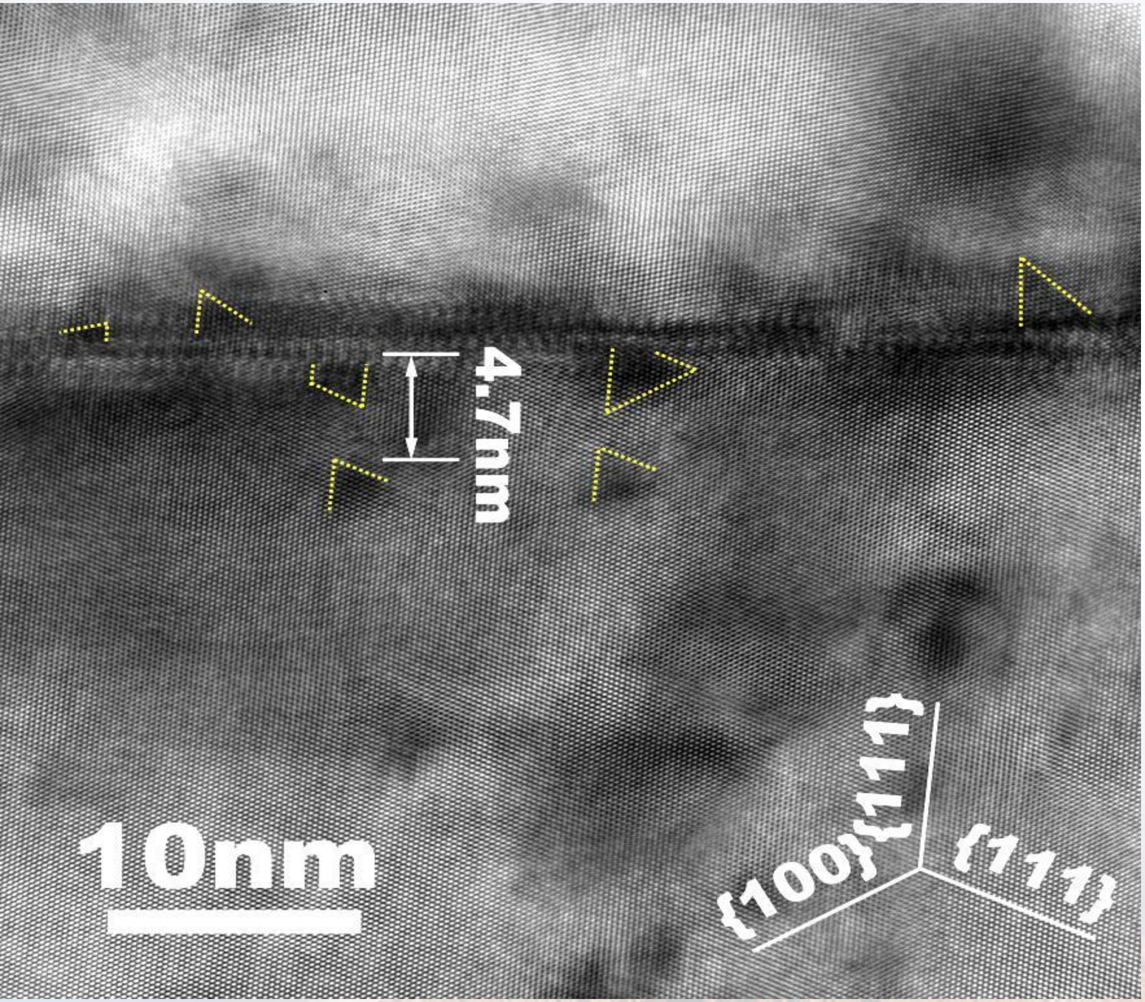


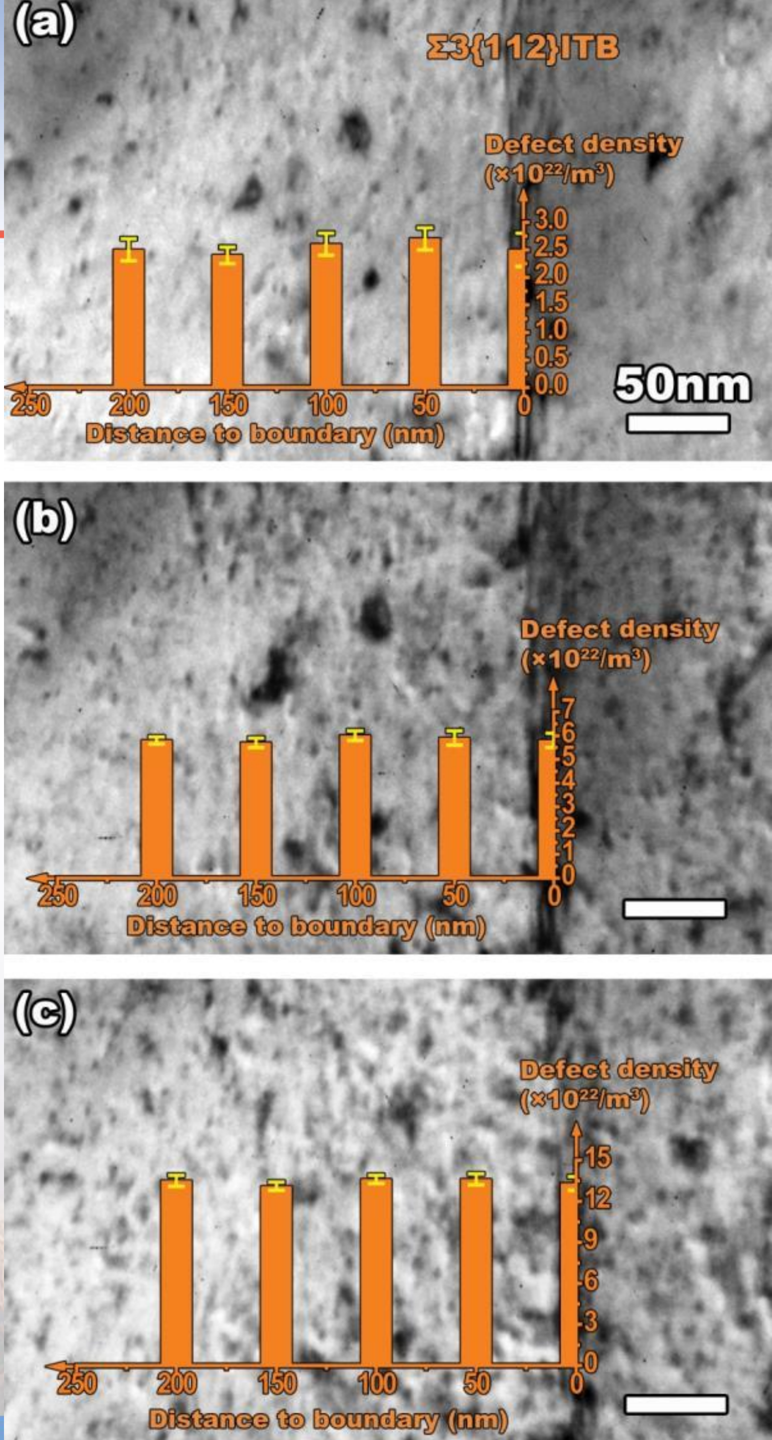




Defects are Altered Little by the Presence of Grain Boundaries

Collaborators: N. Li & A. Misra



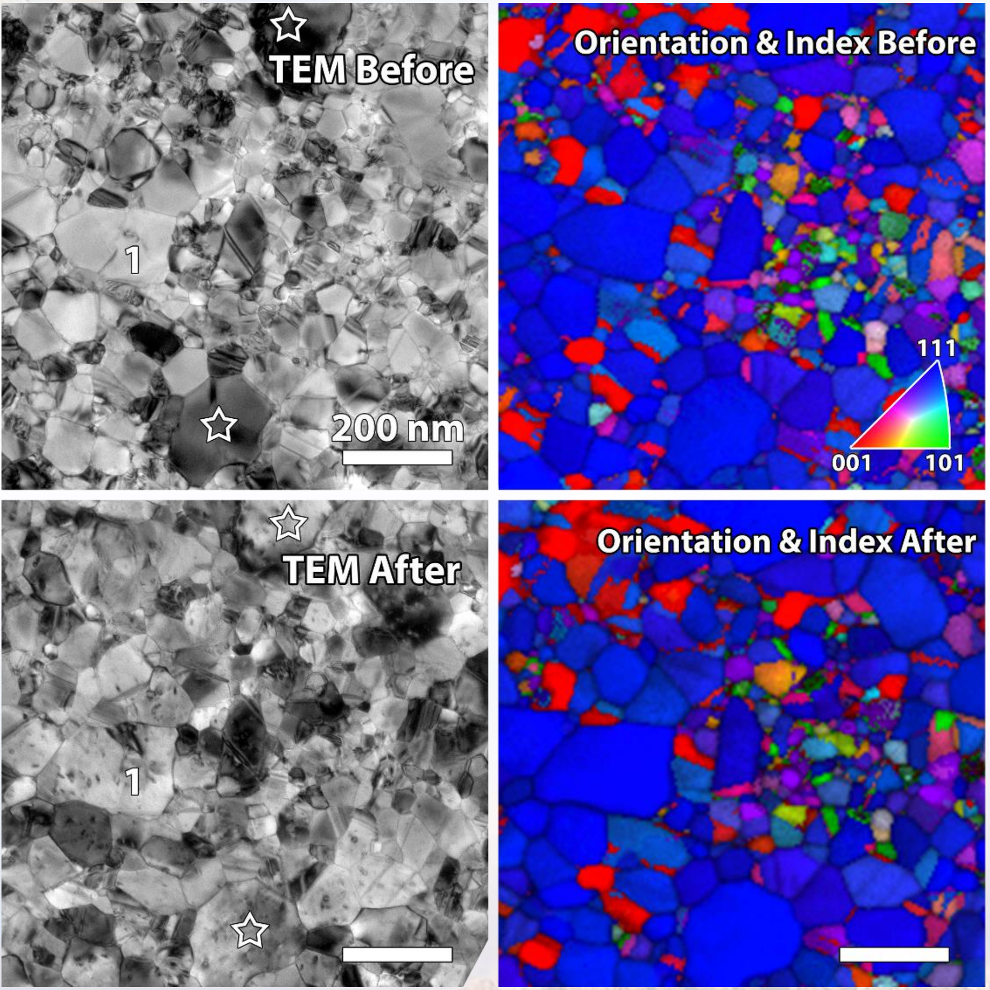


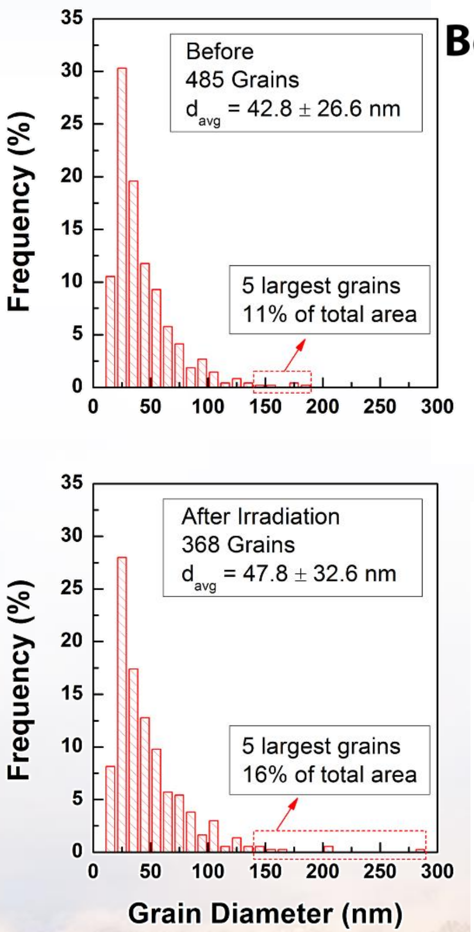
SFT appear to be directly at GB

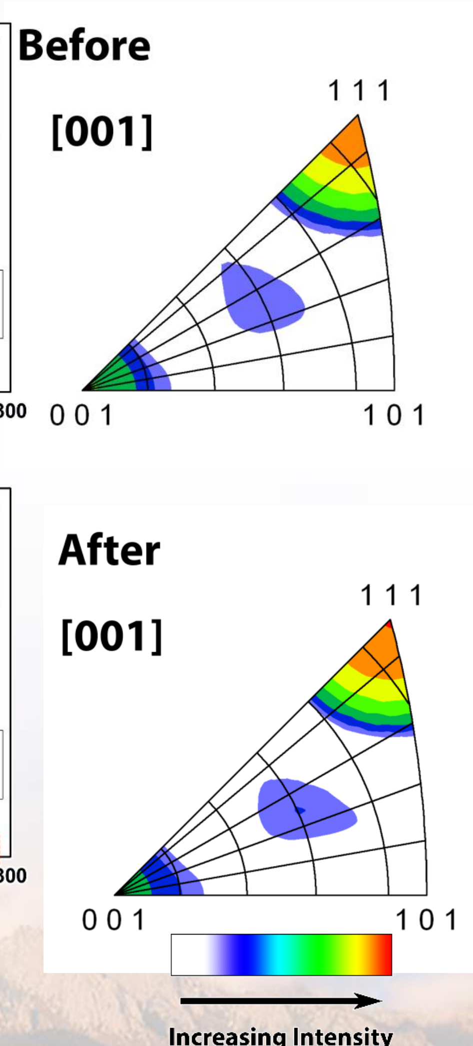
No change in defect density is observed near GB

Quantifying Stability of Nanocrystalline Au during 10 MeV Si Ion Irradiation

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







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

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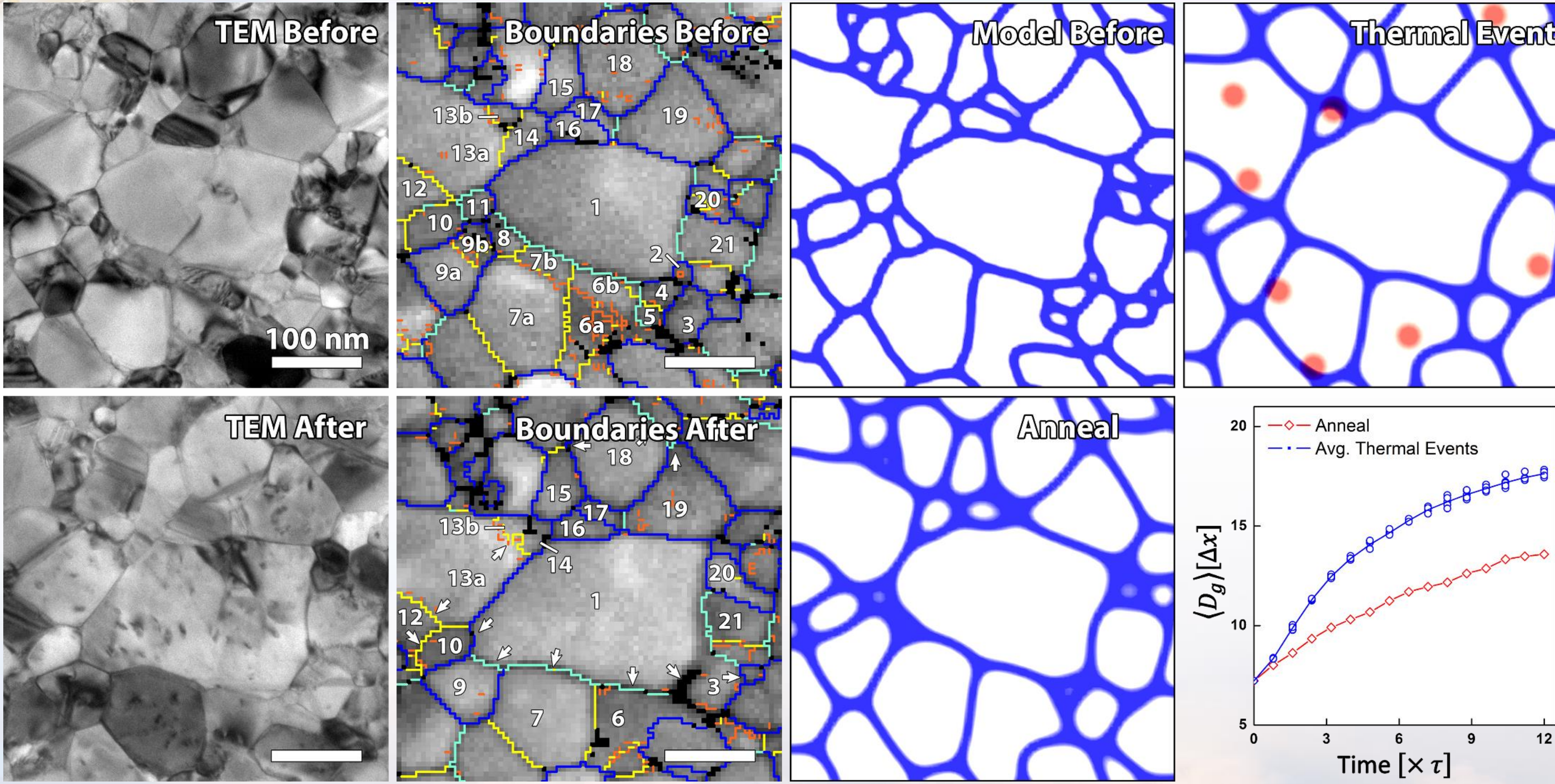
Direct Comparison to Mesoscale Modeling

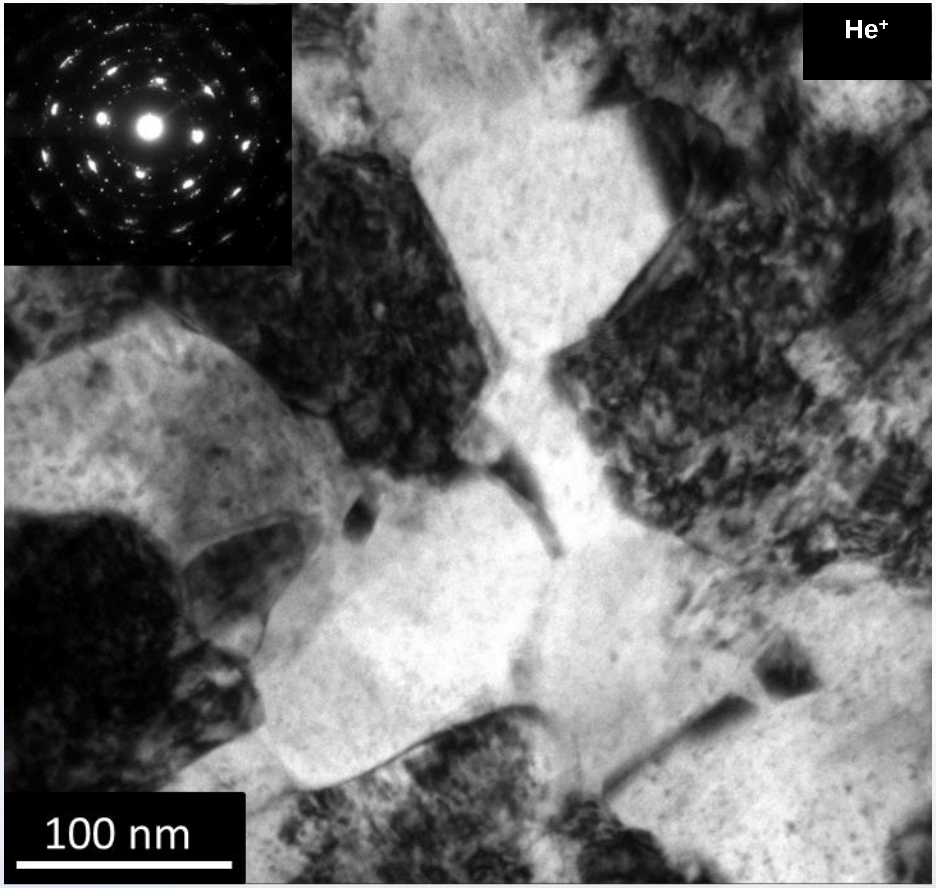
Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles

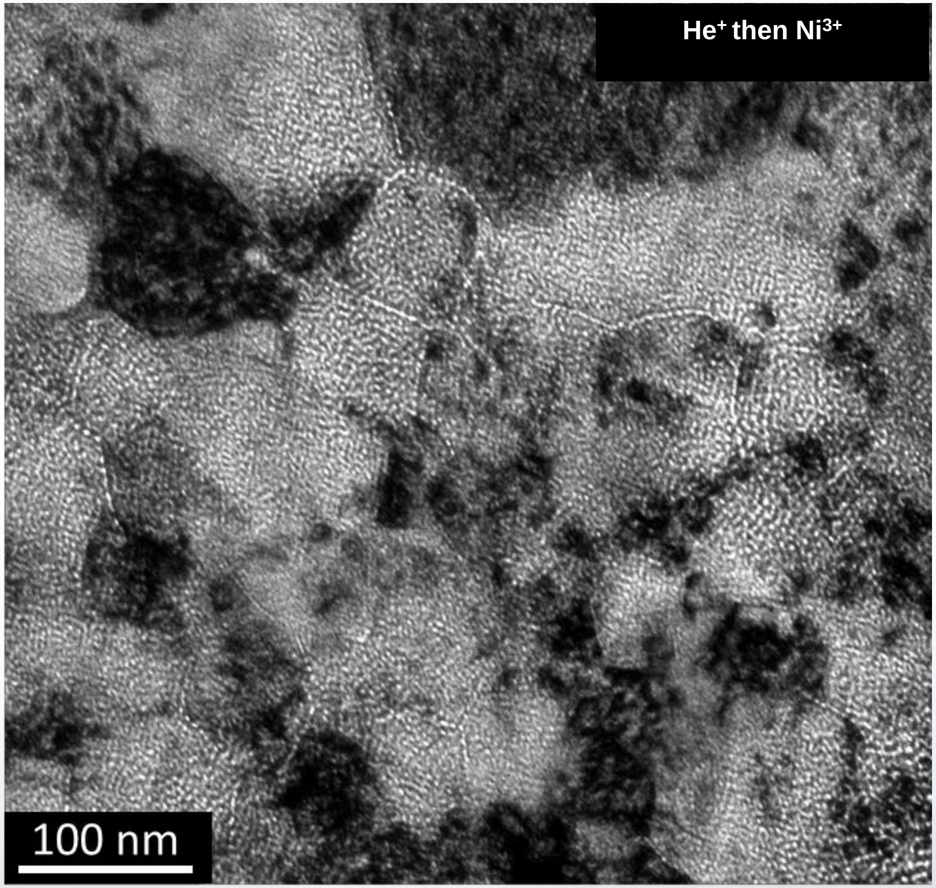


Because of the matching length scale, the initial microstructure can serve as direct input to either MD of mesoscale models & subsequent structural evolution can be directly compared.

Heterogeneous Bubble Formation under Some Radiation Environments

Collaborator: B. Muntifering & J. Qu





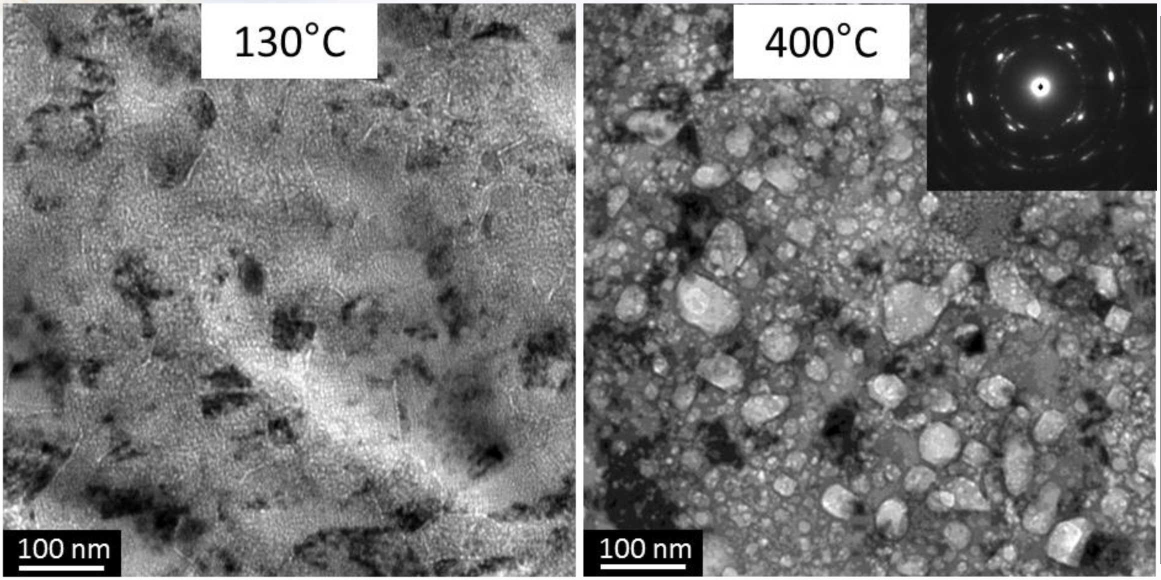
10^{17} He^+/cm^2

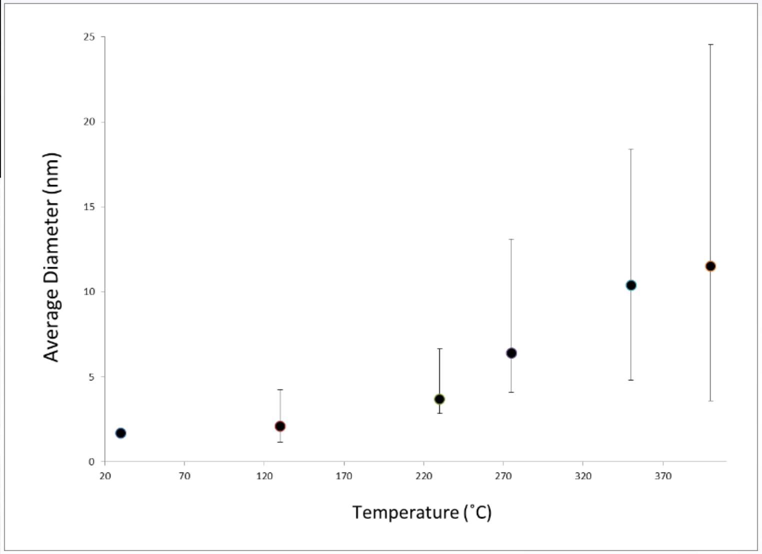
Visible damage to the sample

0.7 dpa Ni^{3+} irradiation

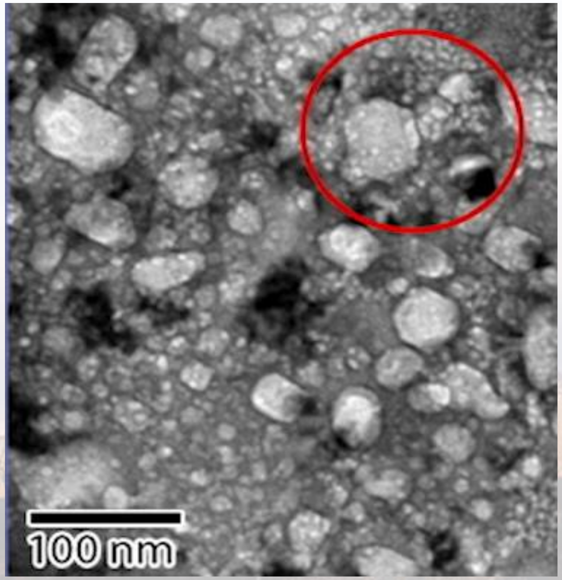
High concentration of cavities along grain boundaries

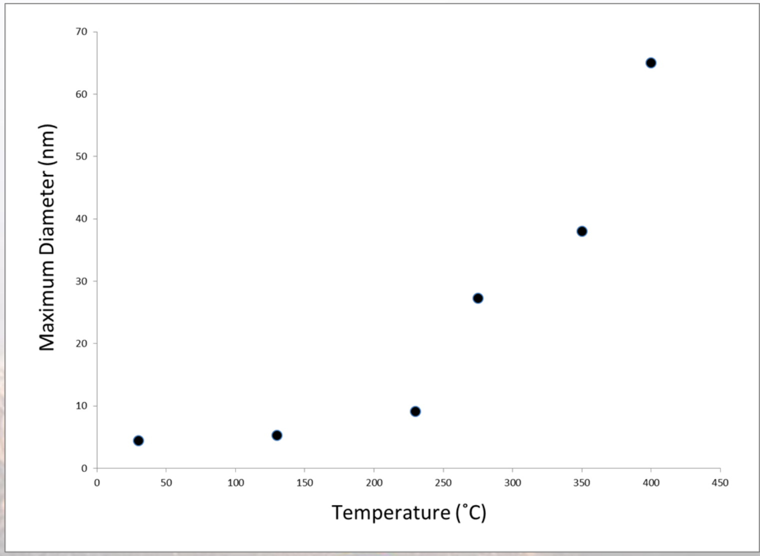
Cavity Growth during In-situ Annealing of 10 keV He^+ Implanted and then 3 MeV Irradiated Ni^{3+}





Bubble to cavity transition and cavity evolution can be directly studied

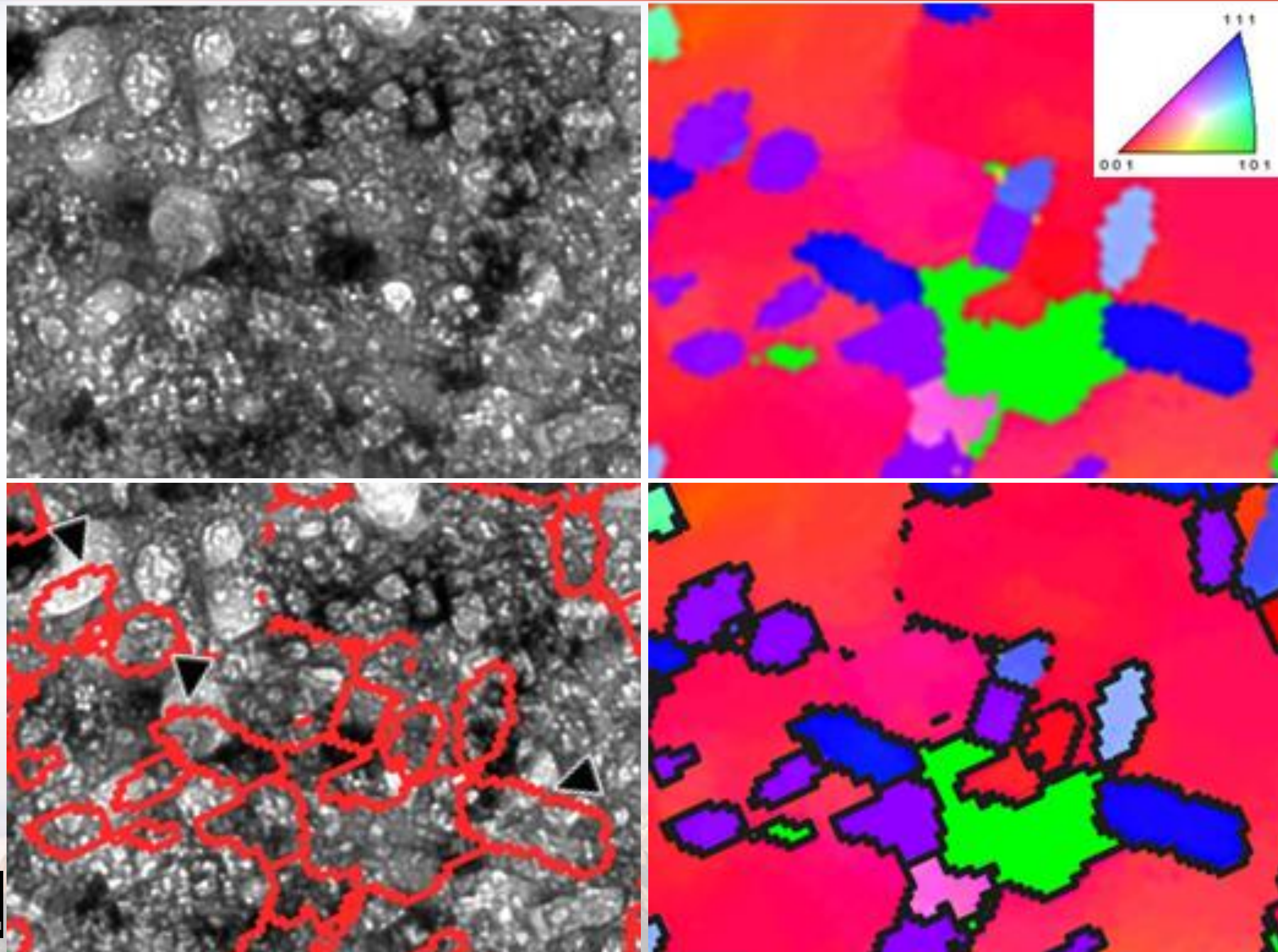




Precession Electron Diffraction Reveals Hidden Grain Structure

Cavities in helium implanted, self-ion irradiated,

nc nickel film annealed to 400 ˚C

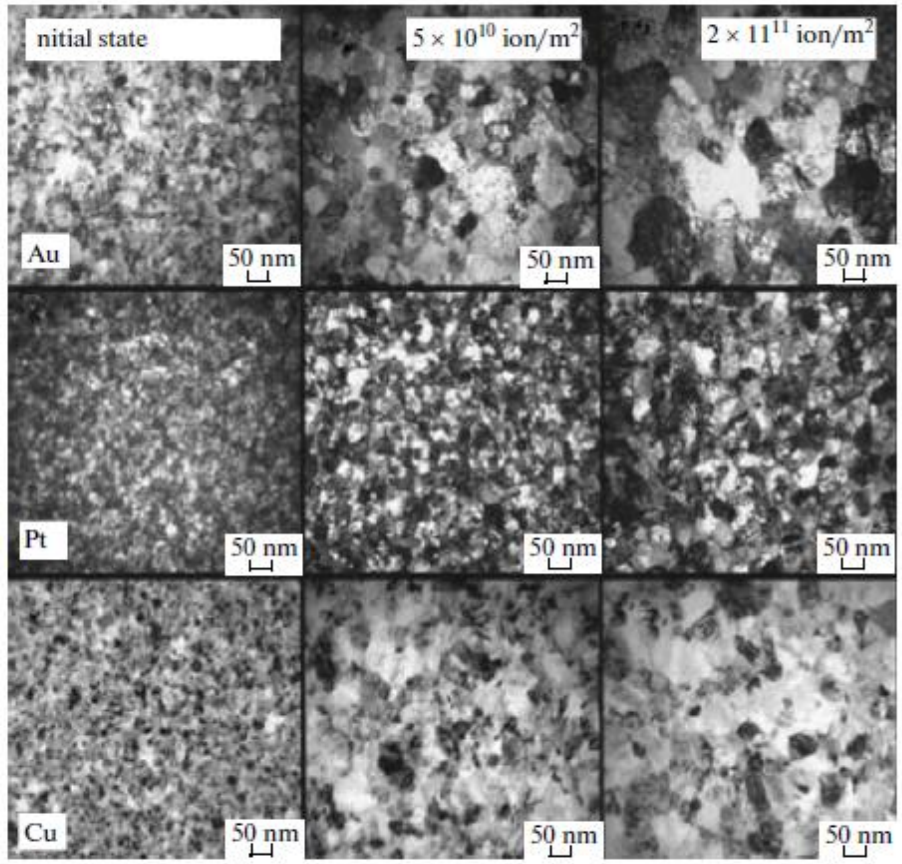


Cavities span multiple grains at identified grain boundaries

100 nm

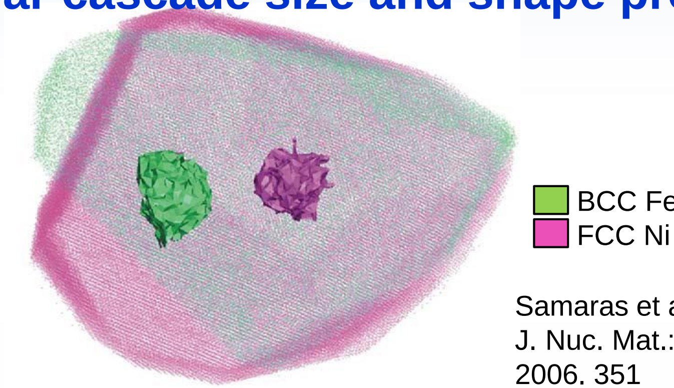
Section Summary:
Radiation Tolerance from Nanostructured Metals

Variation in radiation tolerances



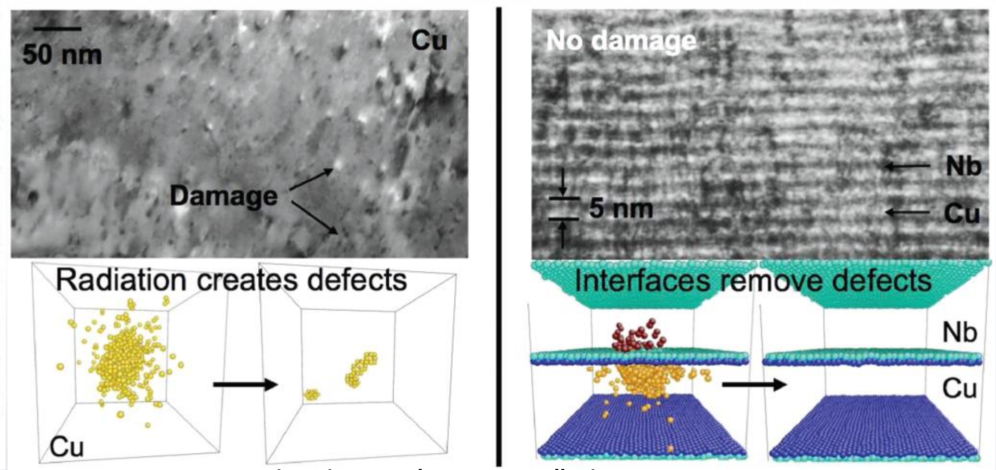
Kaomi et al., JAP: 2008. 104 073525

Similar cascade size and shape predicted



Samaras et al., J. Nuc. Mat.: 2006. 351

Nanolamellars are radiation tolerant



Demkowicz et al., MRS Bulletin: 2010. 35

To a first order mean grain size comparison, these reports appear conflicting.
Not necessarily the case if initial microstructural details and associated properties are considered

This research was partially funded by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering and by the Laboratory Directed Research and Development (LDRD) program at Sandia National Laboratories. Sandia National Laboratories is a multimission 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-NA-0003525. SAND2017-4611 A

Sandia National Laboratories

Outline

1. How Does Nanostructuring Effect a Materials Radiation Tolerances?

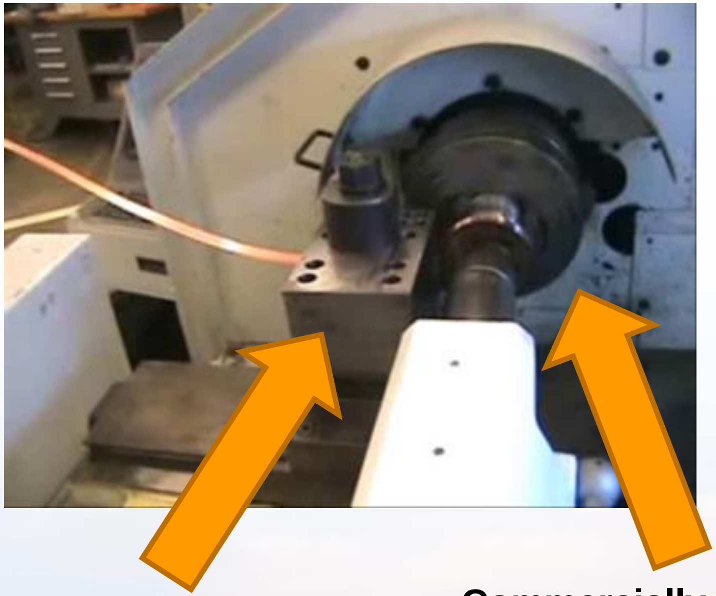
2. Can We Tailor Boundaries/Interfaces to Control Radiation Tolerances?

3. Can We Explore Other Aspects of the Complex Environment Present in Modern and Future Reactors Using *In situ* Techniques?

Large Scale Production of Ultrafine Grained Tungsten

Collaborators: O. El-Atwani, M. Efe, J.P. Allain

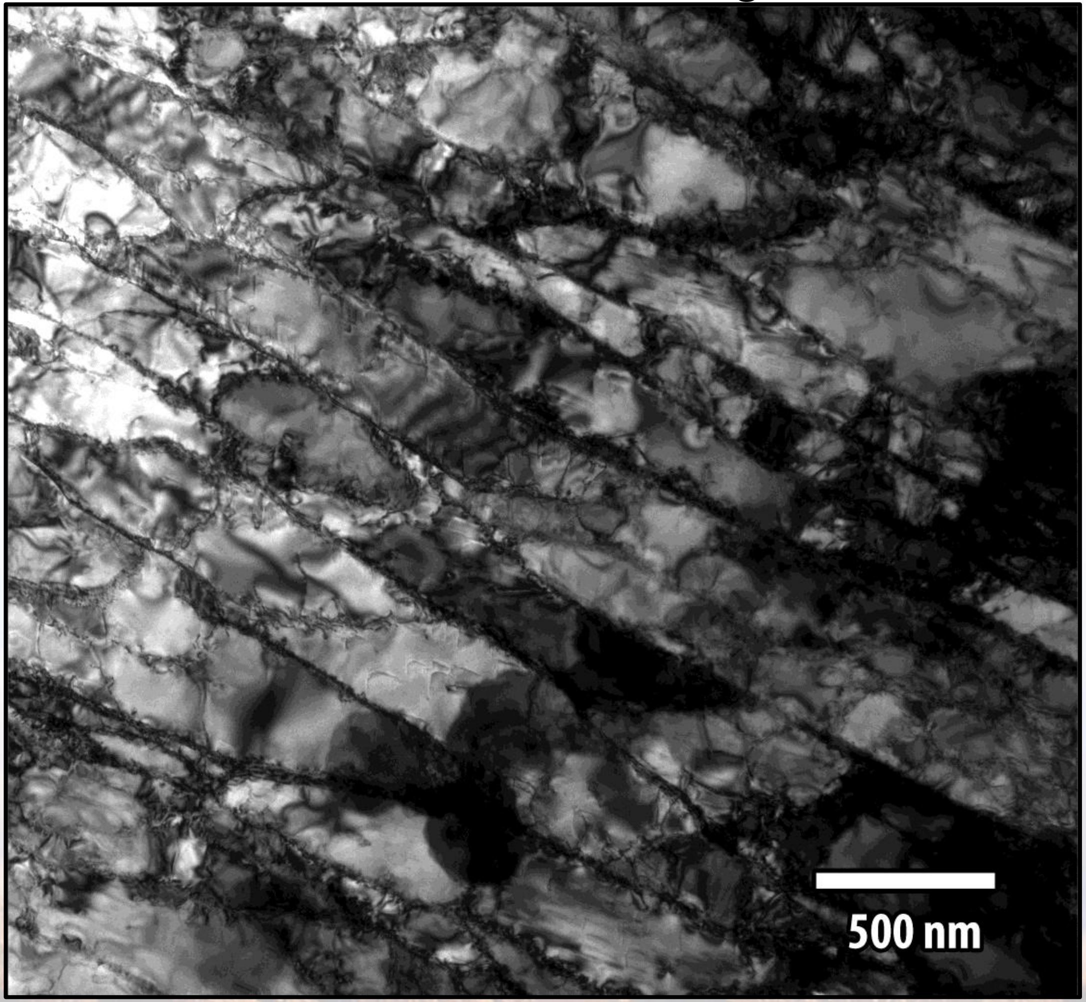
Snapshot of the Process



Extrusion Machining tooling

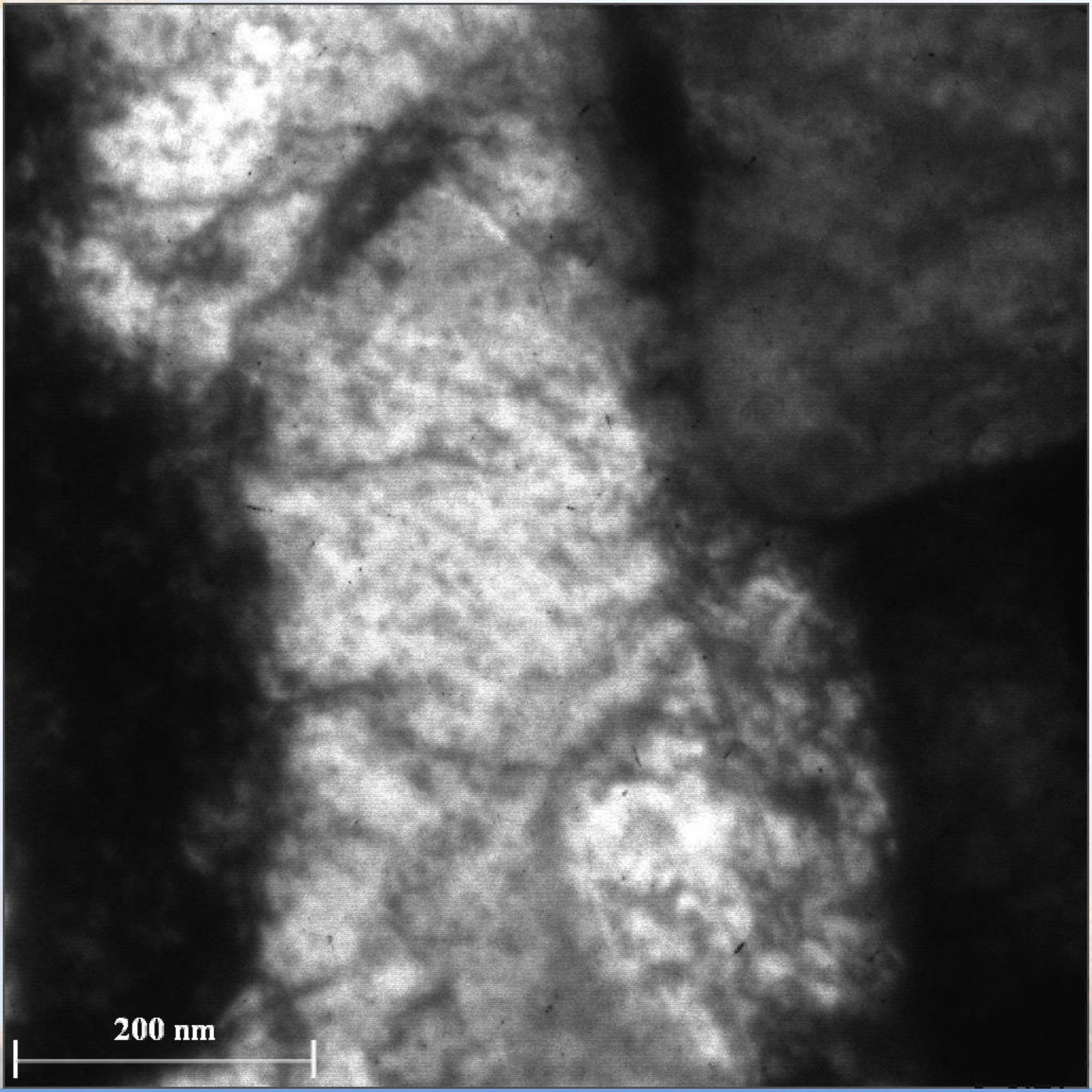
Commercially available lathe

Ultrafine Grained Tungsten



Displacement Damage Evolution at Nanometer Resolution

Collaborators: O. El-Atwani, M. Efe, J.P. Allain



In situ **TEM self-ion irradiation at 3 MeV of a samples from the same SPD tungsten lot used for the He implantation.**

I^{3}TEM can providing insight into:

1) **Loop formation**
2) **Loop stability & migration**
3) **Rad & structural defect interactions**

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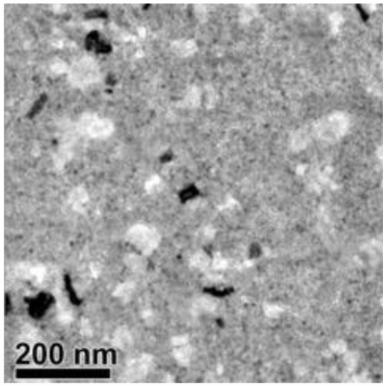
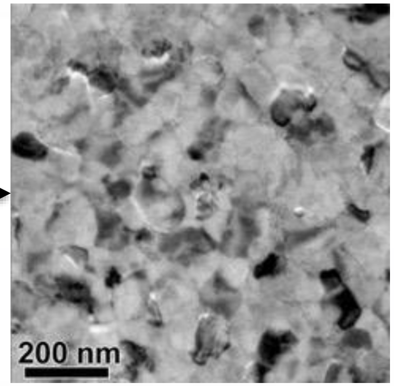
Scaling down to Nanocrystalline Tungsten Alloys

Collaborators: O.K. Donaldson, T. Kaub, G. Thompson, and J. Trelewicz

Nanocrystalline W

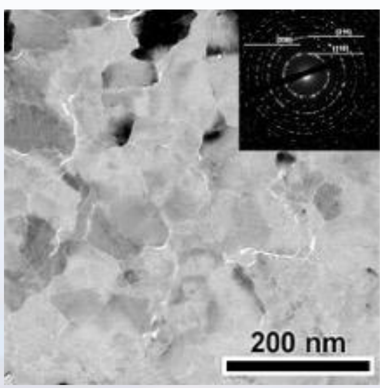


650° C

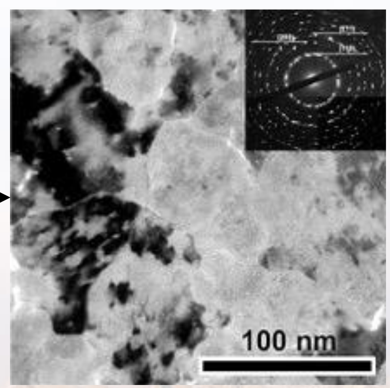


d_{avg} = 30 nm

d_{avg} = 86 nm

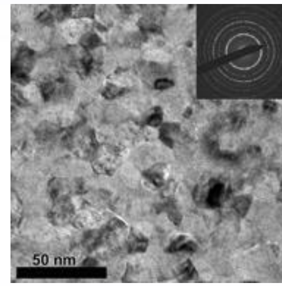


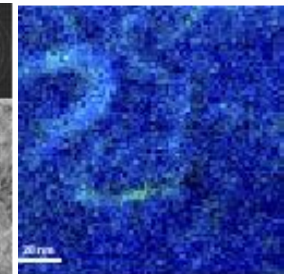
8.5 dpa



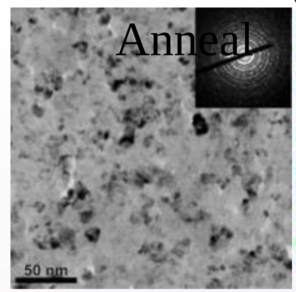
$\rho_{defect} = 2.5 \times 10^{22}\ m^{-3}$

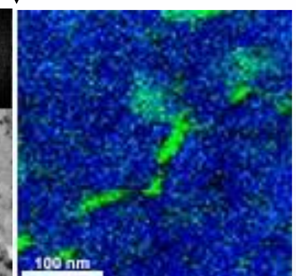
Nanocrystalline W-20at.%Ti





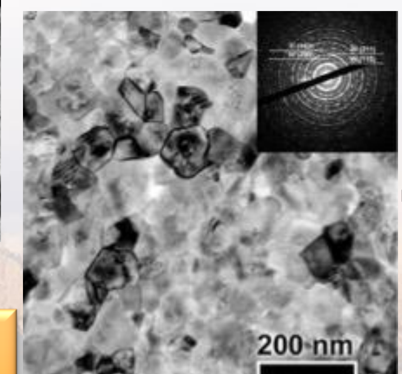
1000°C Anneal



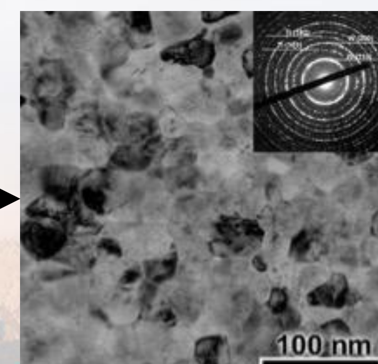


Grain growth is hampered by the addition of Ti

Ti solute is heterogeneously distributed after annealing



6.3 dpa

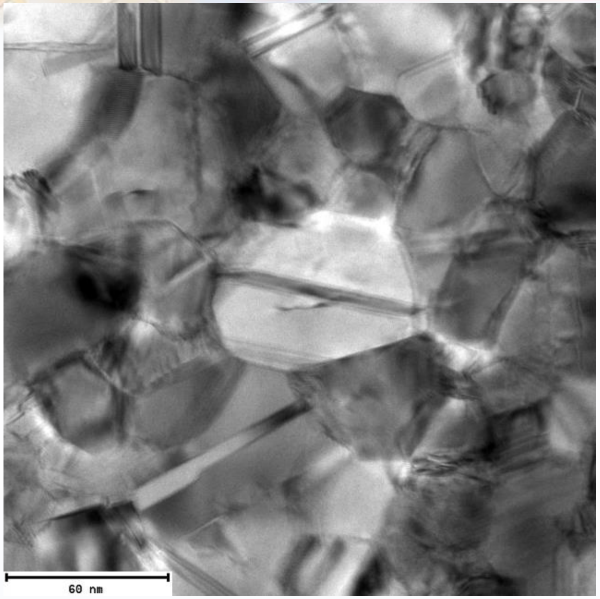


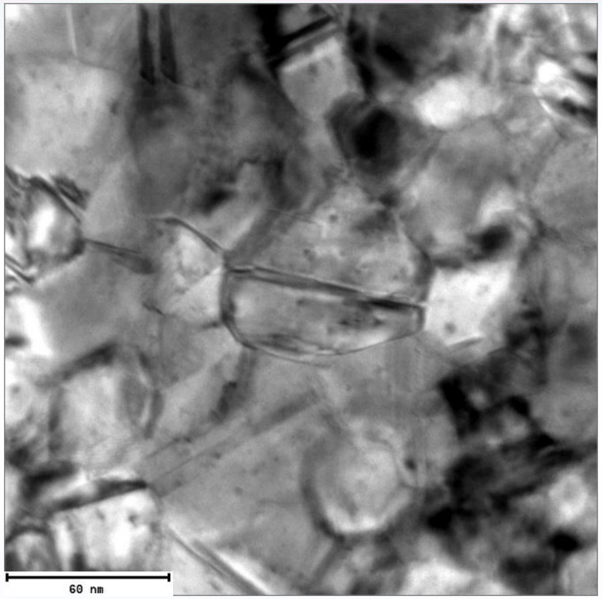
$\rho_{defect} = 2.8 \times 10^{22}\ m^{-3}$

Alloying does not negatively effect radiation tolerance, while improving thermal and mechanical properties

Nanocrystalline vs. Nanoporous Au results

Collaborators: N. Briot and T.J. Balk



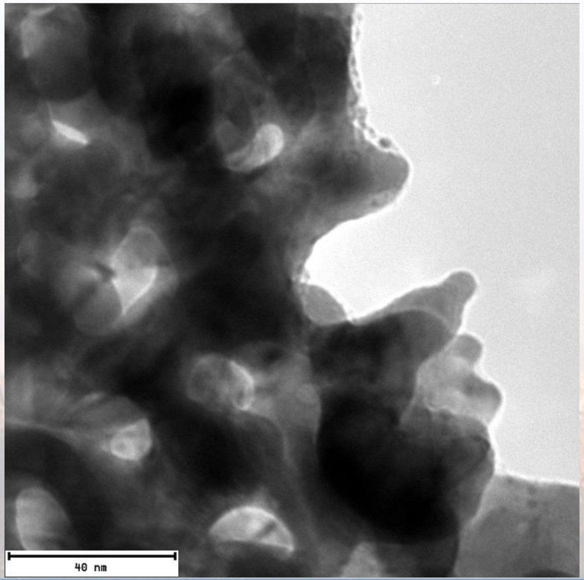


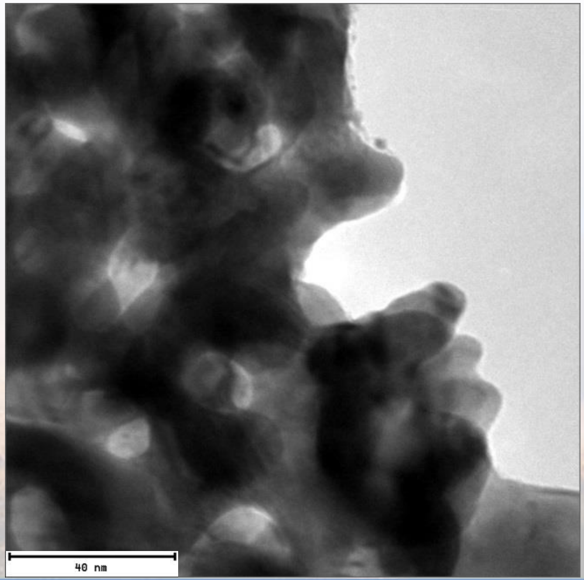
Nanoporous Au after ~ 6.6 dpa at 46 keV:

Melting of the ligament

Nanocrystalline Au after ~ 0.5 dpa at 46 keV:

Lots of defects constantly created





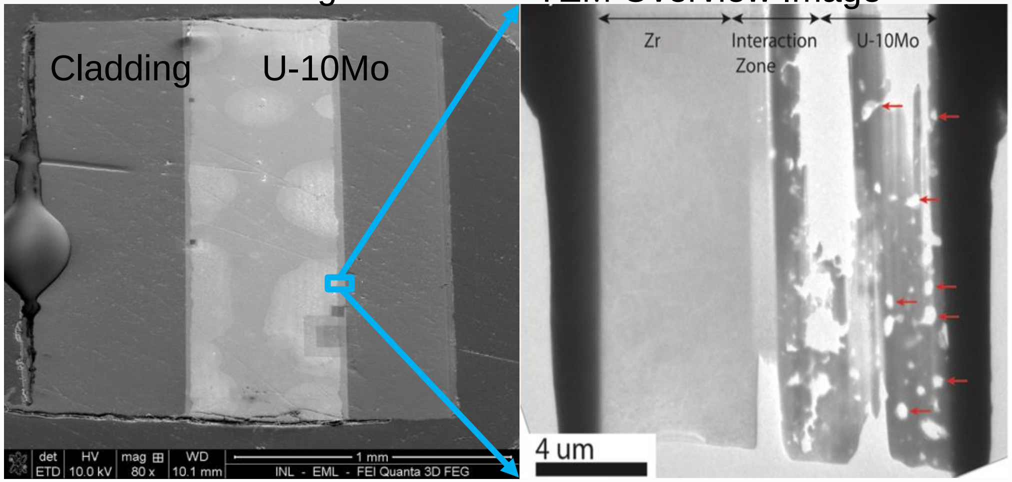
Free surfaces provide the ideal sink, but nanoporous materials undergo surface modification and many other effects under radiation damage

Post Irradiation Characterization of U-10Mo/Zr monolithic interface

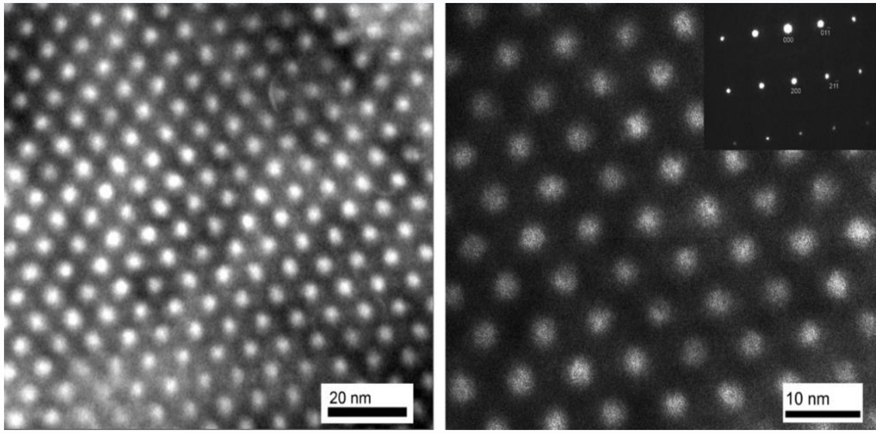
Collaborators: C. Barrr, A. Aitkaliyeva

SEM Overview Image

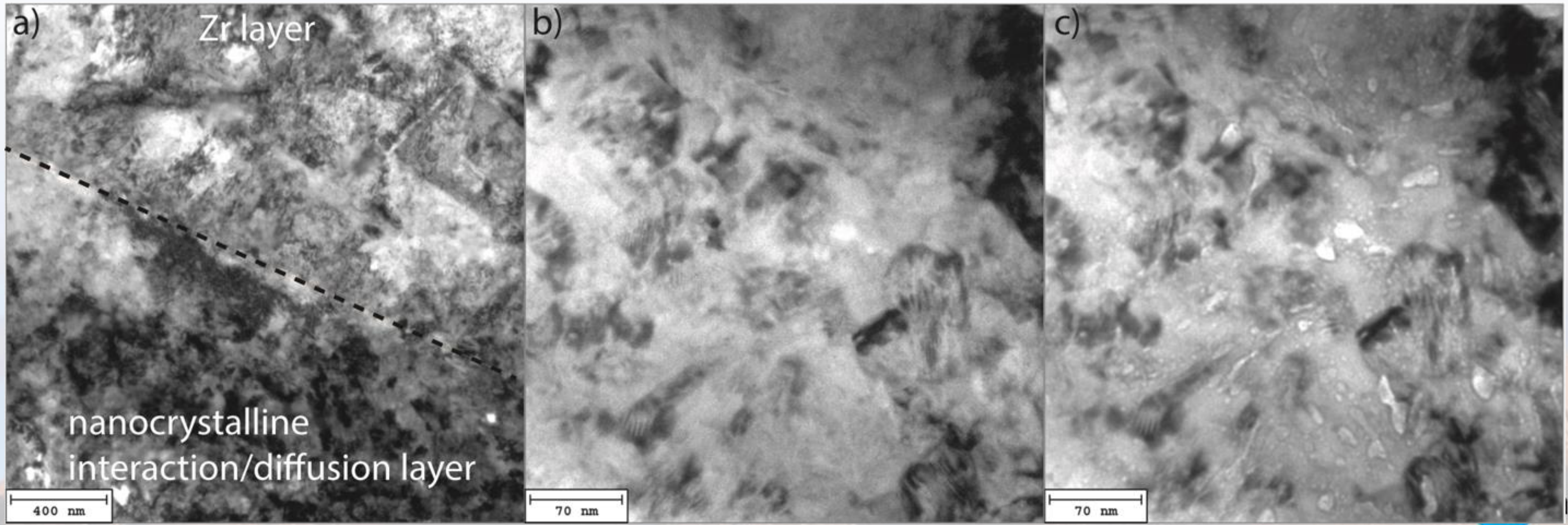
TEM Overview Image



Gas superlattice bubbles ZA [110] in U-10Mo.



- U-10Mo Monolithic fuel with fission density (fiss/cm^3) = 4.4 x 10^{21}
- Average gas superlattice bubble diameter distribution is 3.5 ± 0.25 nm diameter.

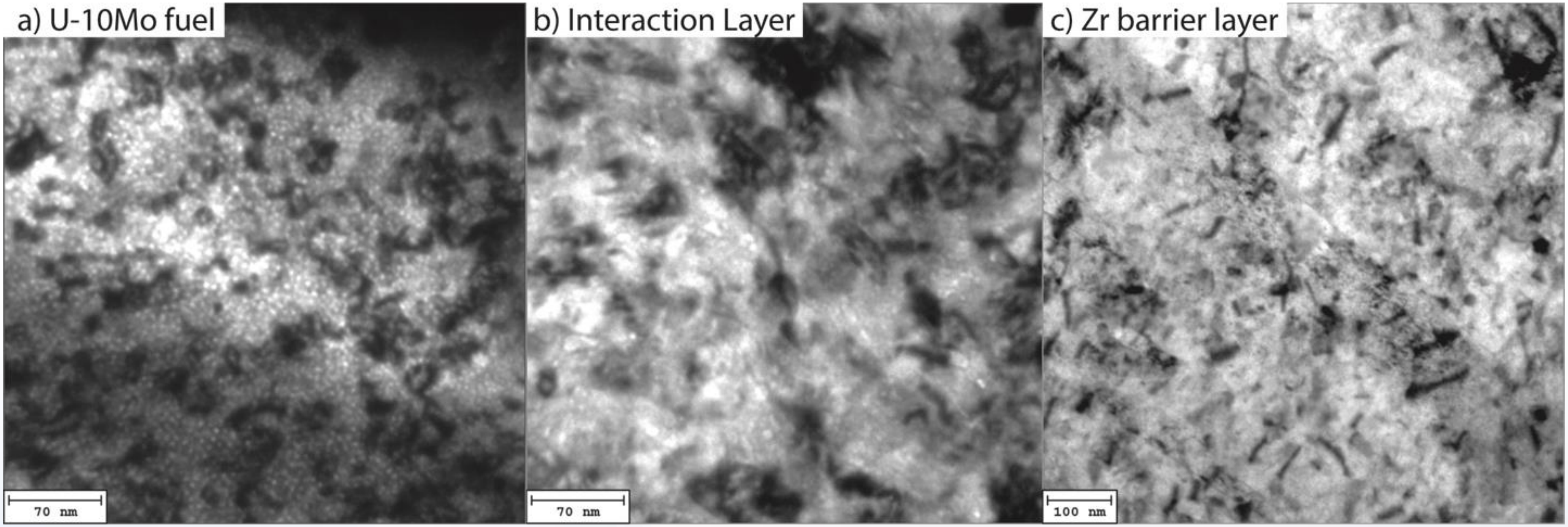


Zr layer and interaction layers where in-focus image of interaction layer (center) and de-focus (-2um) image of interaction layer with high level of porosity apparent

In-situ TEM ion irradiations of PIE U-Mo/Zr interface

Collaborators: C. Barrr, A. Aitkaliyeva

***Irradiation Conditions*:** Fluence = 1.1 x 10^{16} ions/cm^2; 3MeV^{2+} Zr ions at RT



- Continued stability of the periodic gas bubble superlattice in the U10Mo fuel
  - Apparent surface oxide (likely UO_2) post in-situ TEM irradiation
- Growth of dislocation loops (arrowed in red) in Zr barrier layer apparent and indications of increased coalescence of porosity in interaction layer

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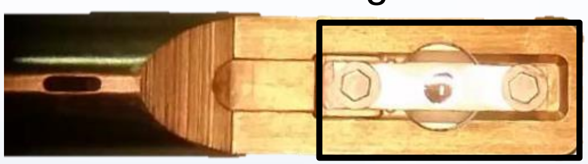
In situ Qualitative Mechanical Testing

Collaborators: C. Barr

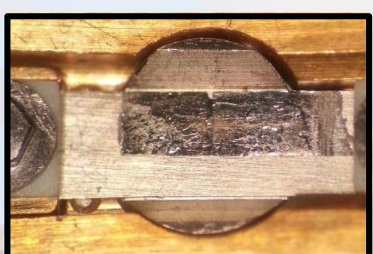
Gatan straining TEM Holder

- 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

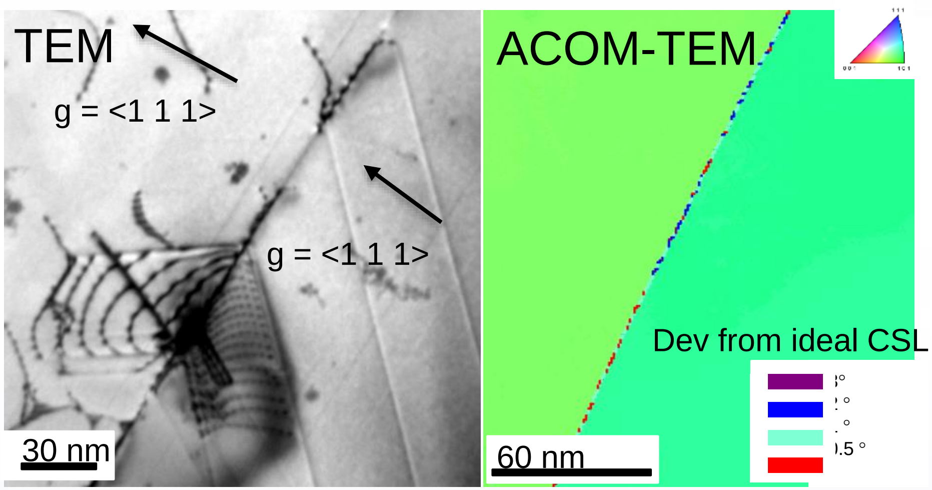


Thin film tension “jig”:

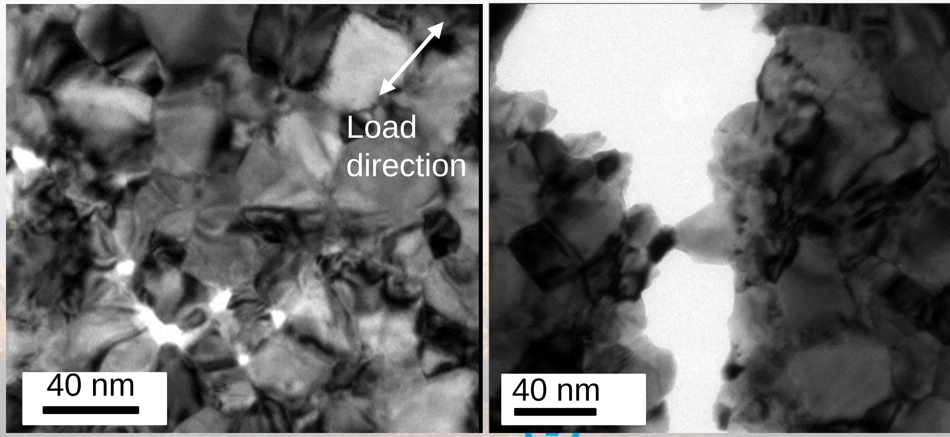


Jet thinned disk:

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



Observe deformation mechanisms in nanocrystalline metals during tensile straining:

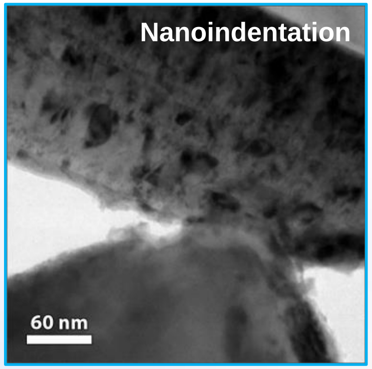


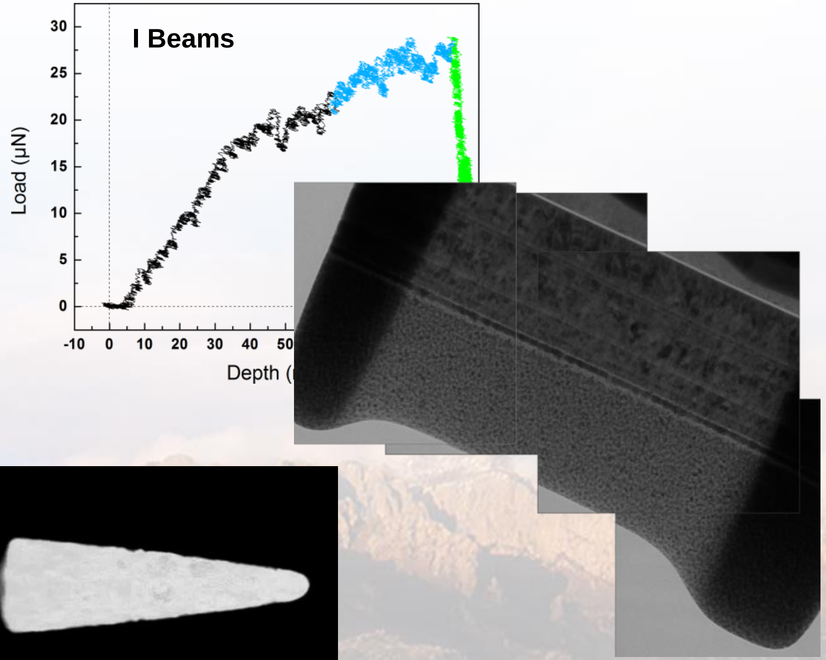
In situ Quantitative Mechanical Testing

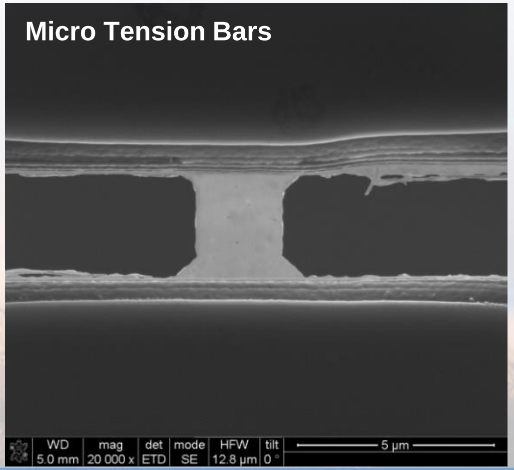
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

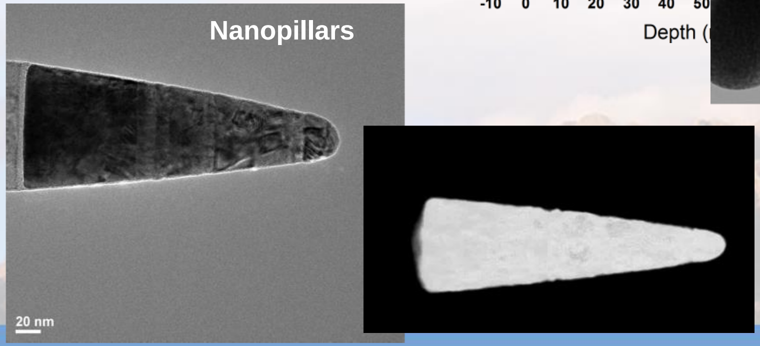
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

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



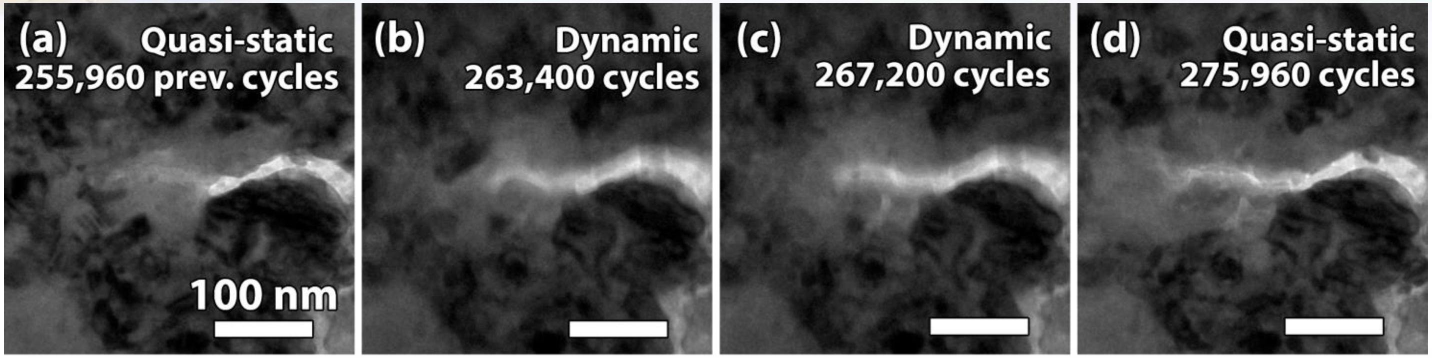


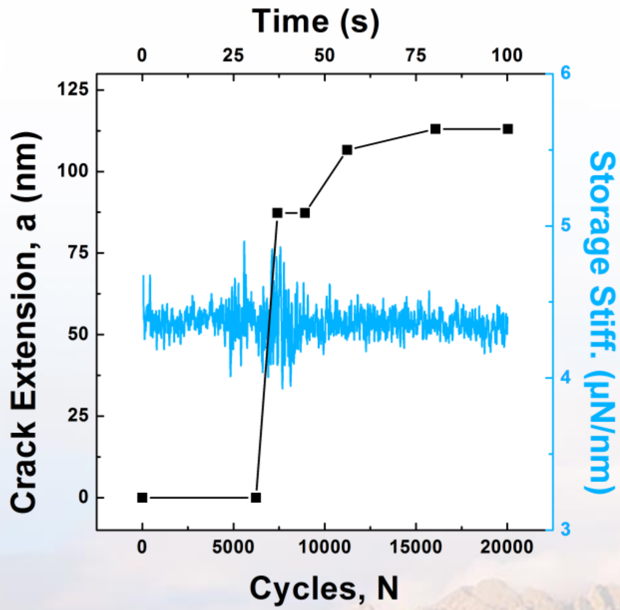


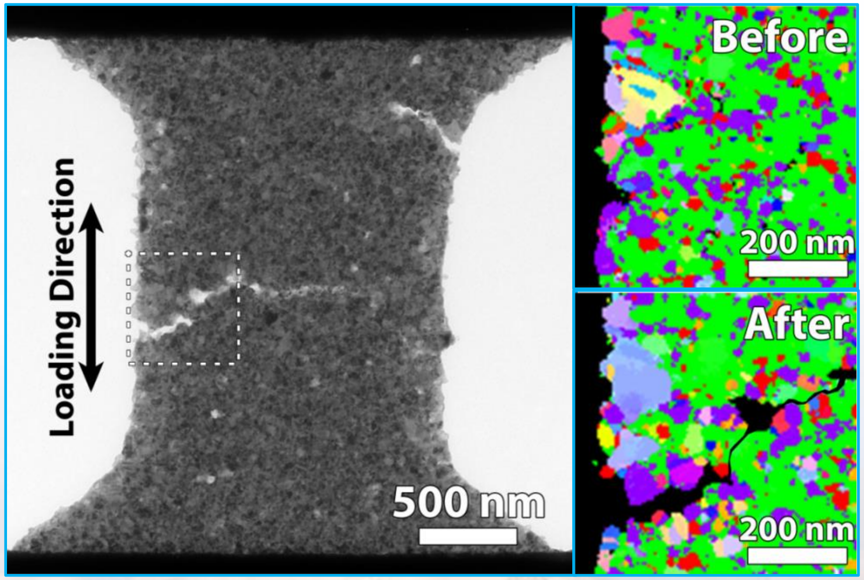


High Cycle Fatigue: Crack Growth Quantified

Contributors: D.C. Buller, B. L. Boyce, W. Mook, D. Stauffer & Hysitron Inc.





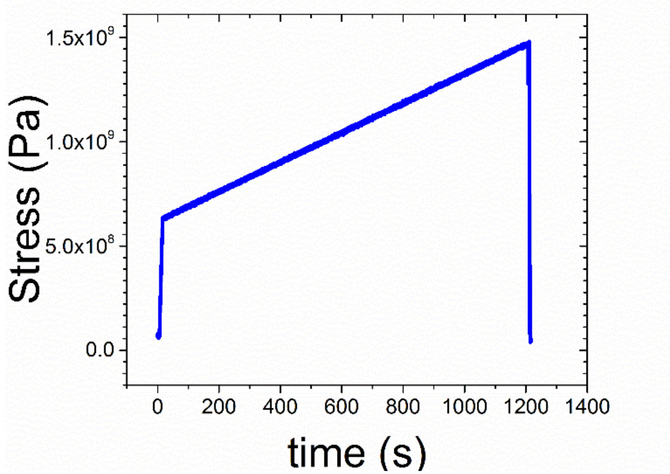


- Picometer-per-cycle measured crack growth rate
- Evidence of fatigue-induced grain growth.

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

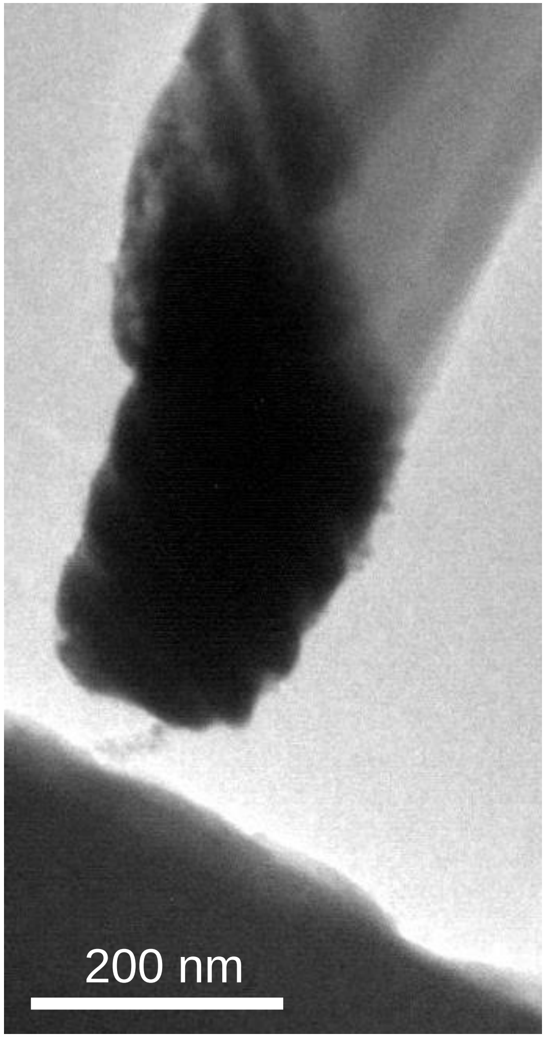
Contributors: S. Dillon & R.S. Averback

Controlled Loading Rate Experiments

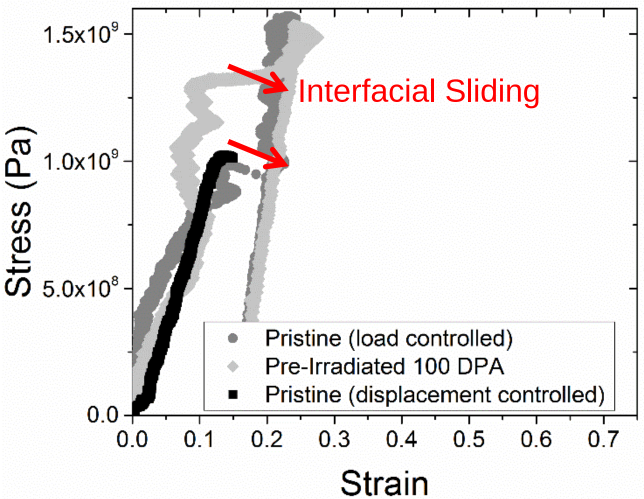


In-situ TEM radiation creep is feasible!

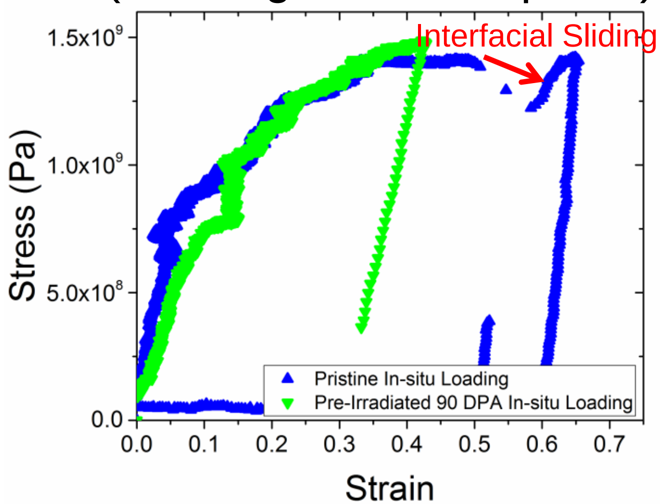
50 nm Cu-W multilayer
20 Min



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



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



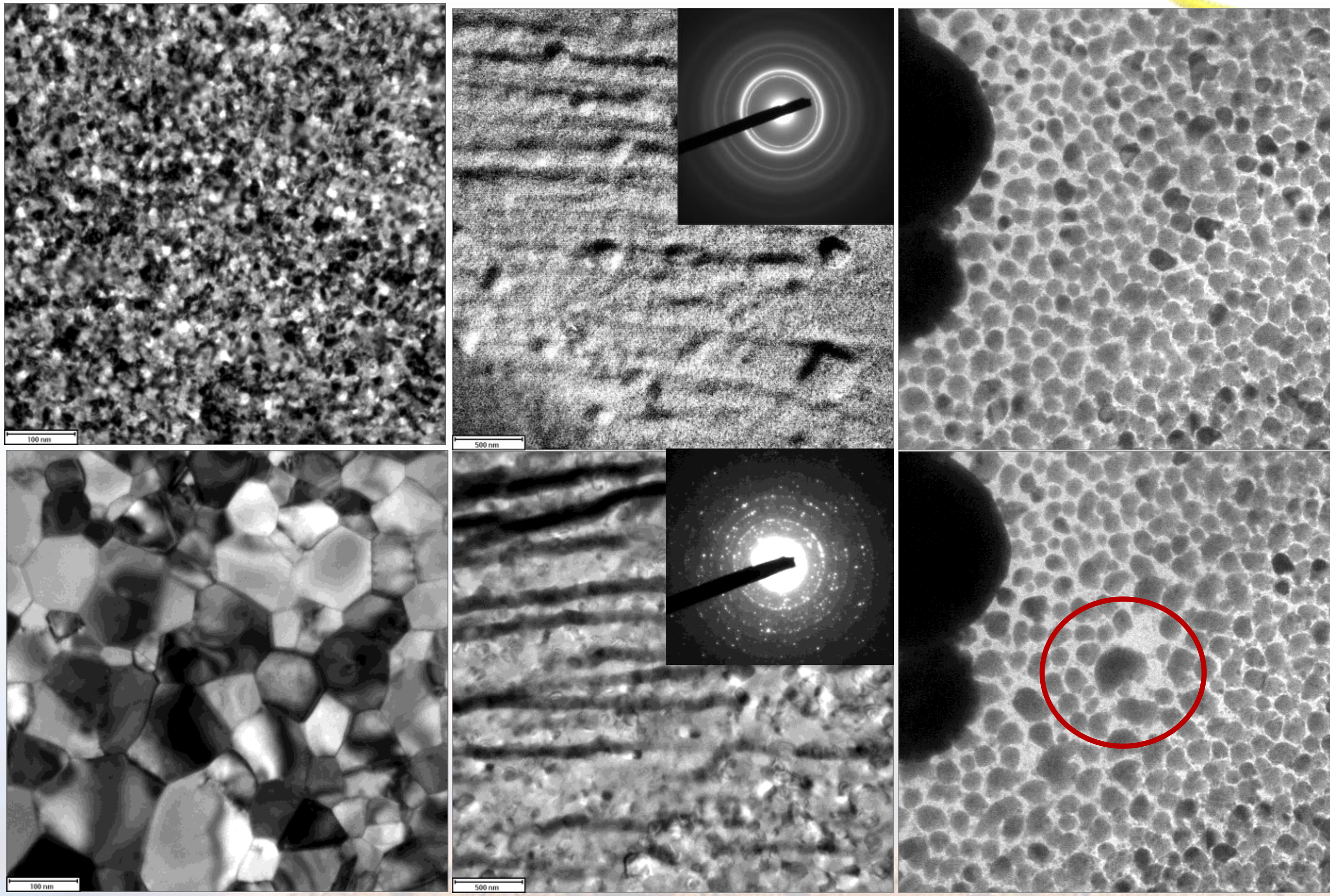
Initial Laser Heating Observations

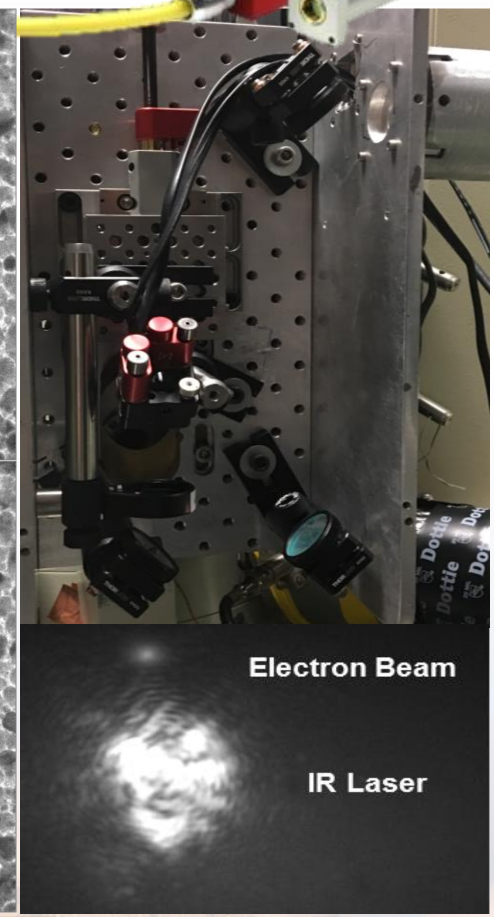
Collaborator: P. Price, C.M. Barr, D. Adams, M. Abere

Pt Grain Growth | Reactive Multilayer Films | Nanoparticle Sintering

Before

After

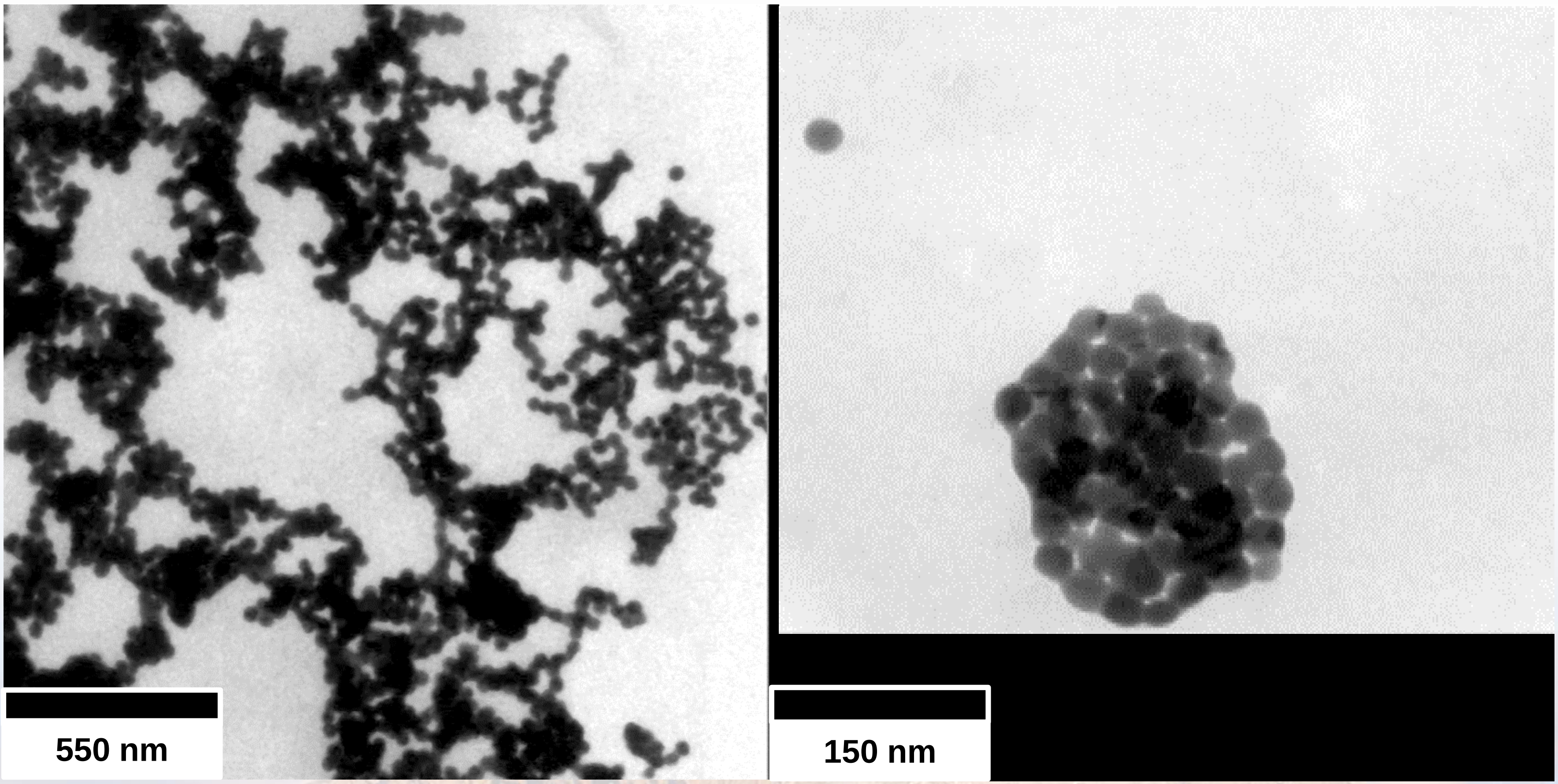




We can now introduce rapid thermal heating with any TEM stage or ion beam conditions

Complex Interaction Au NPs Exposed to Laser Irradiation

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



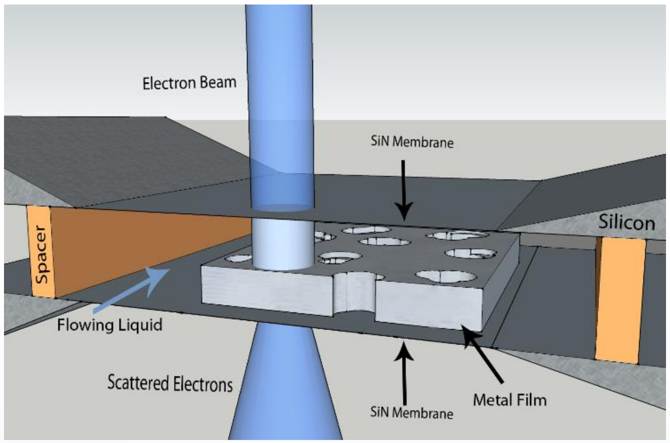
Speed = 2.5x

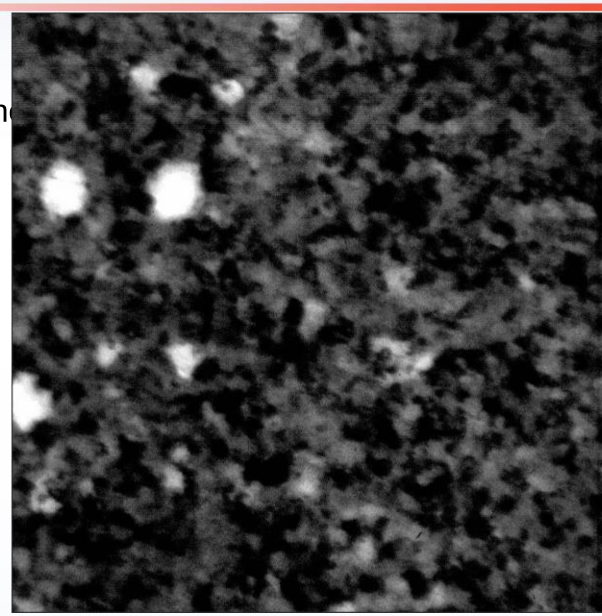
A Complex Combination of Sintering, Reactions, and Ablation Occurs

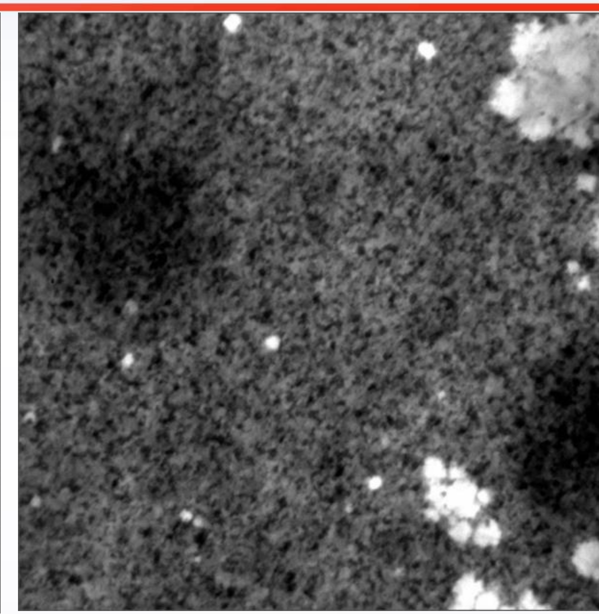
Can We Gain Insight into the Corrosion Process through *In situ* TEM?

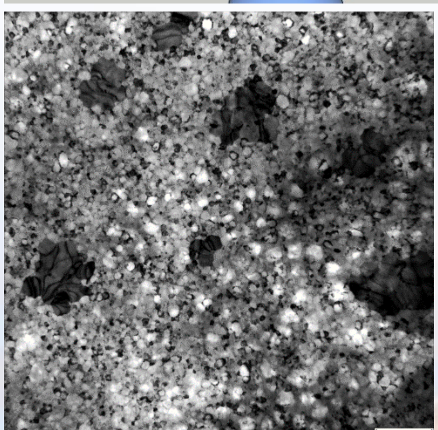
Contributors: D. Gross, J. Kacher, I.M. Robertson & Protochips, Inc.

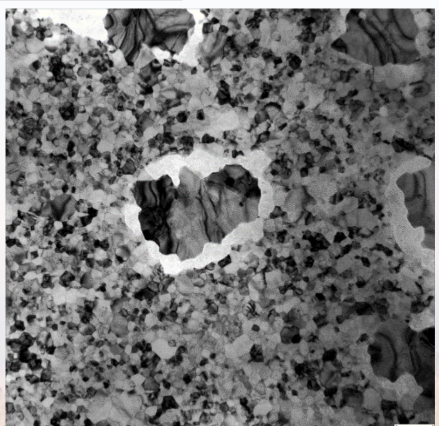
Microfluidic Stage
- Mixing of two or more channels
- Continuous observation of the reaction chann

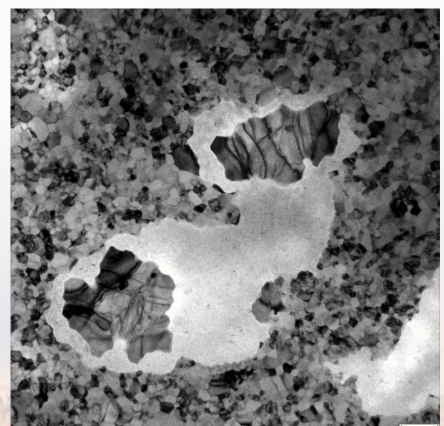


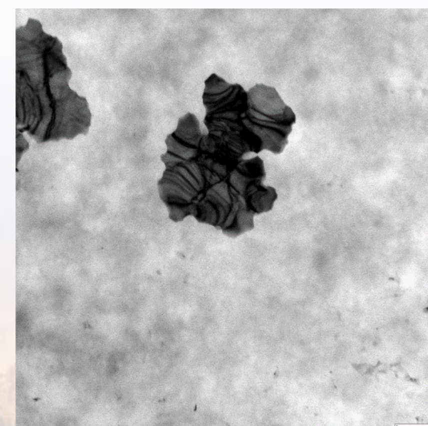












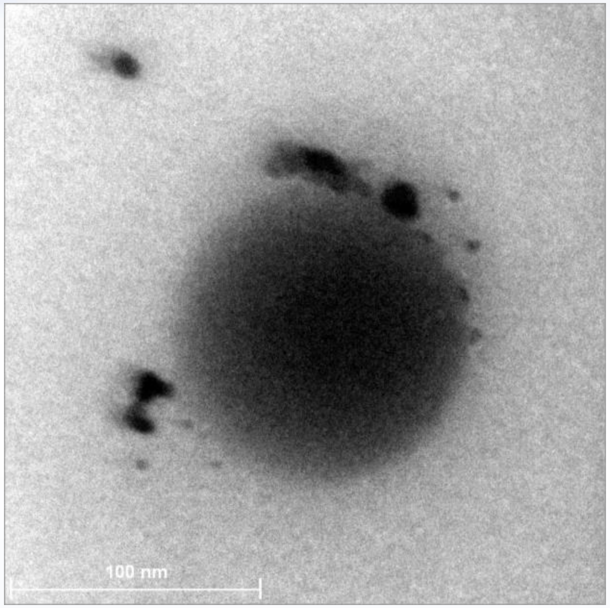
Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.
Large grains resulting from annealing appear more corrosion tolerant

Other Fun Uses of Microfluidic Cell

Protocell Drug Delivery

S. Hoppe,
E. Carnes,
J. Brinker

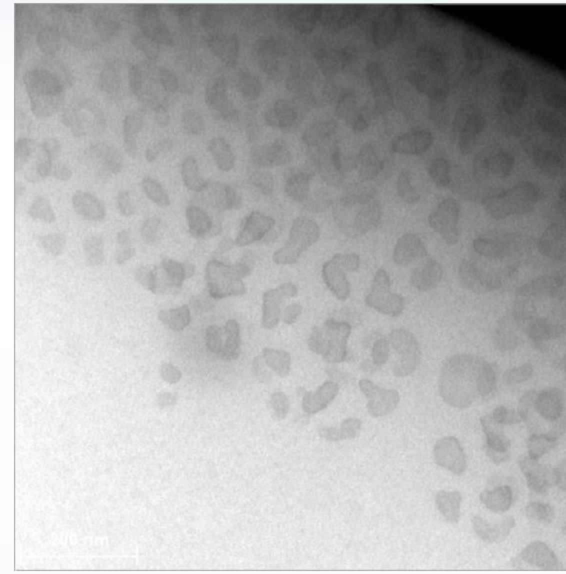
Liposome encapsulated Silica destroyed by the electron beam



BSA Crystallization

S. Hoppe

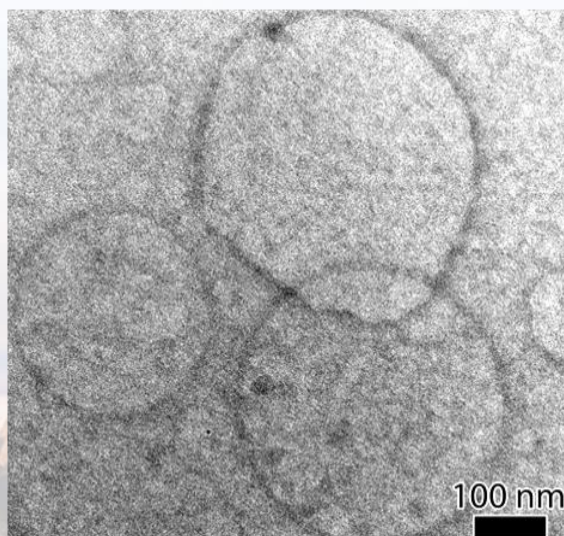
Crystallization of excess Bovine Serum Albumen during flow



Liposomes in Water

S. Hoppe,
D. Sasaki

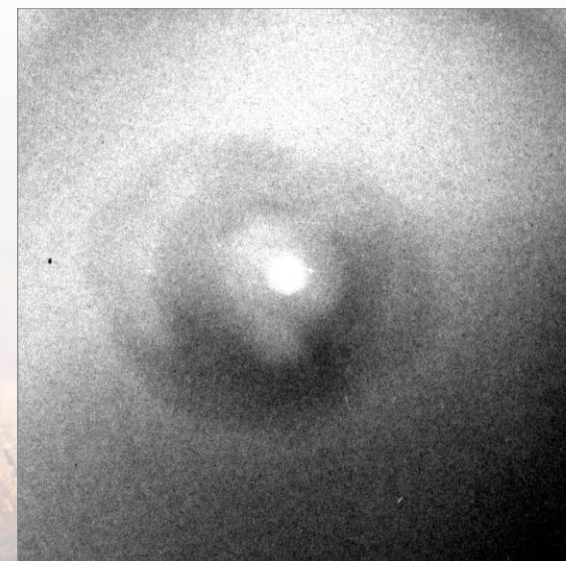
Liposomes imaged in flowing aqueous channel



La Structure Formation

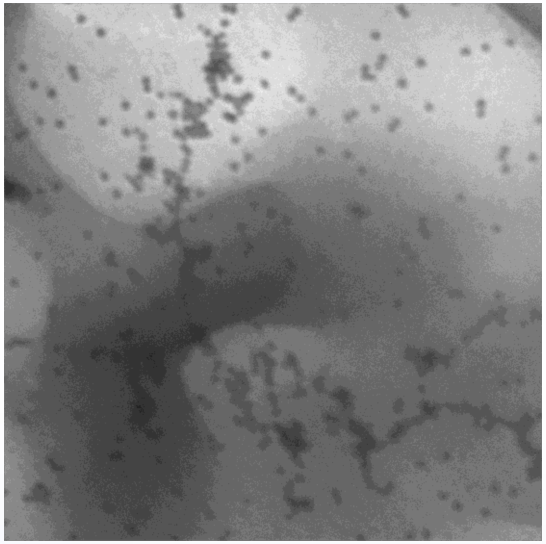
S. Hoppe,
T. Nenoff

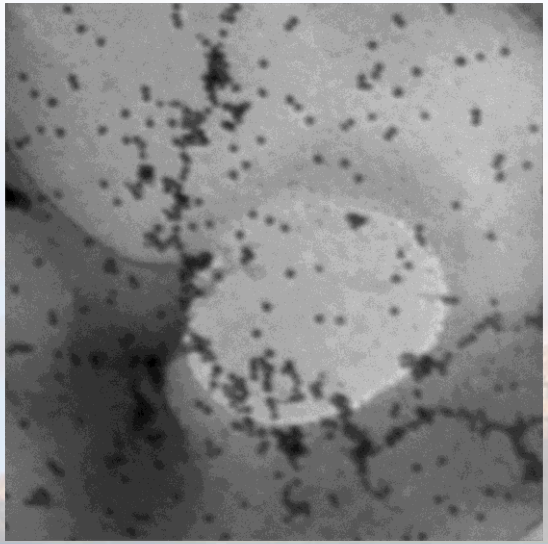
La Nanostructure form from $LaCl_3$ H_2O in wet cell due to beam effects



In Situ TEM observation of three-phase mixture

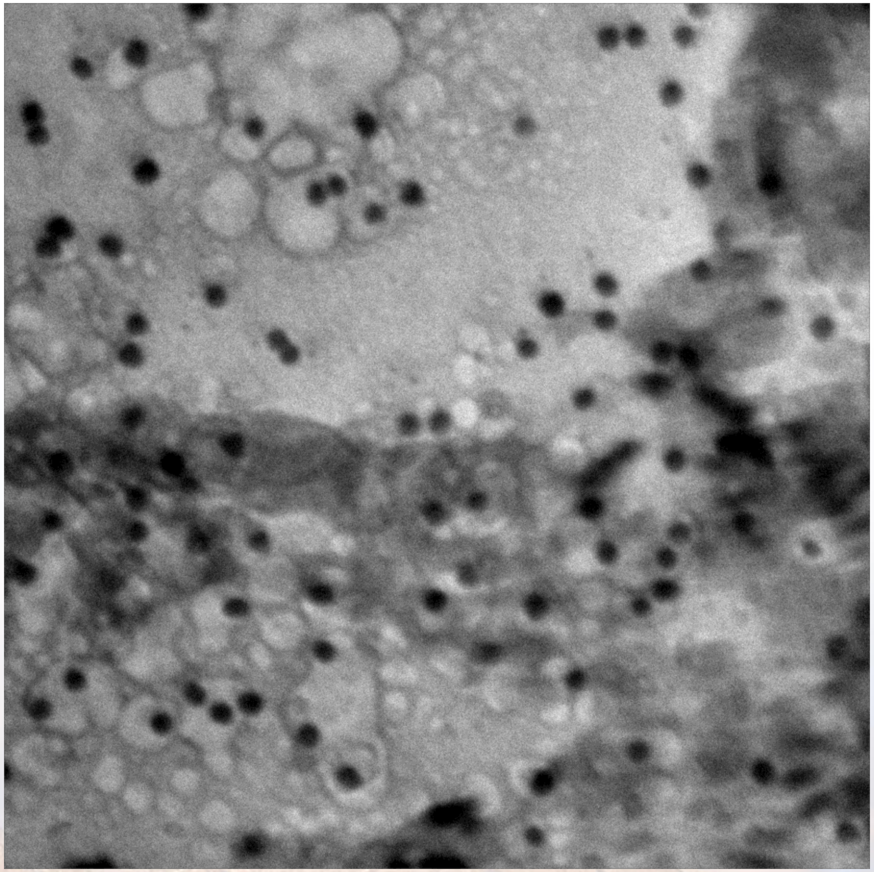
Collaborator: Sarah Hoppe





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

Au nanoparticles, aqueous solution, and gas bubbles

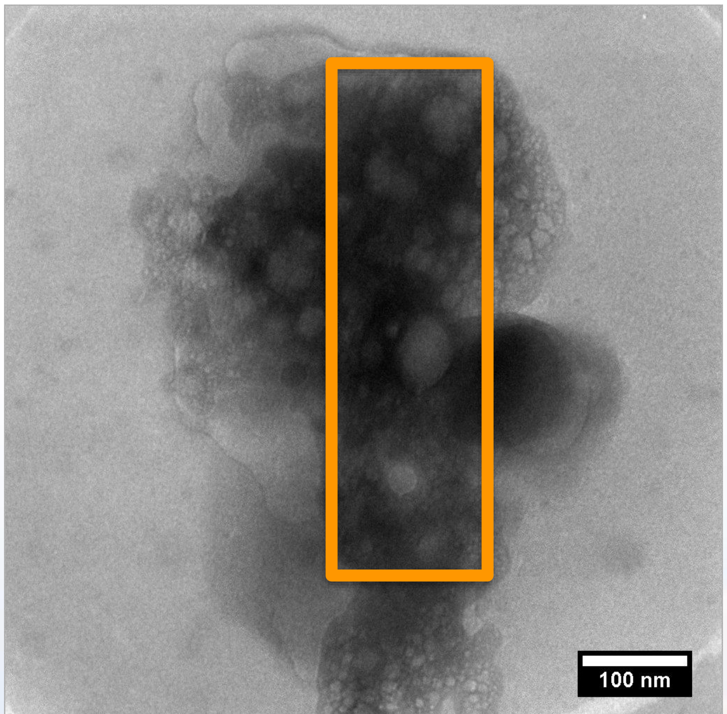


PED in Liquid Cell Environment

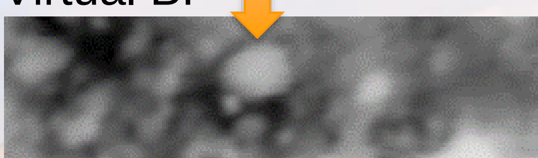
Contributors: C. Taylor, S. Pratt, & T. Nenoff

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

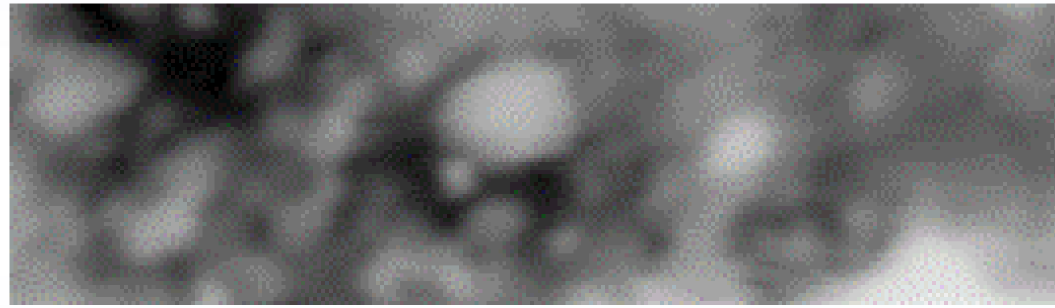
TEM Image



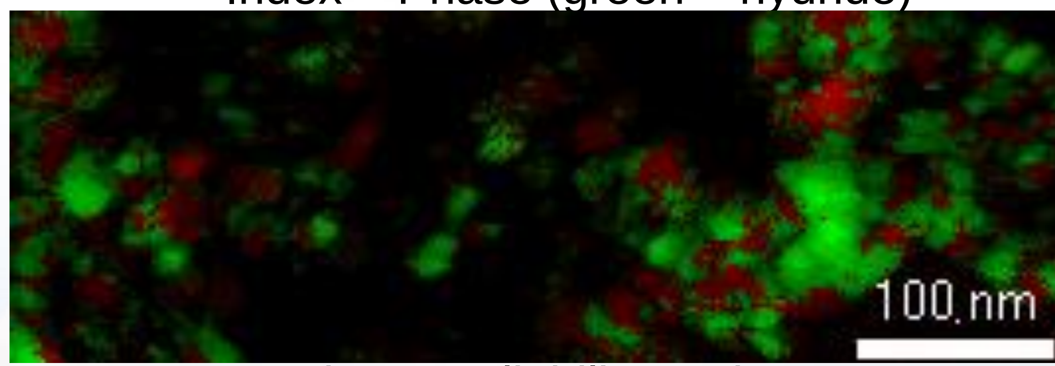
Virtual BF



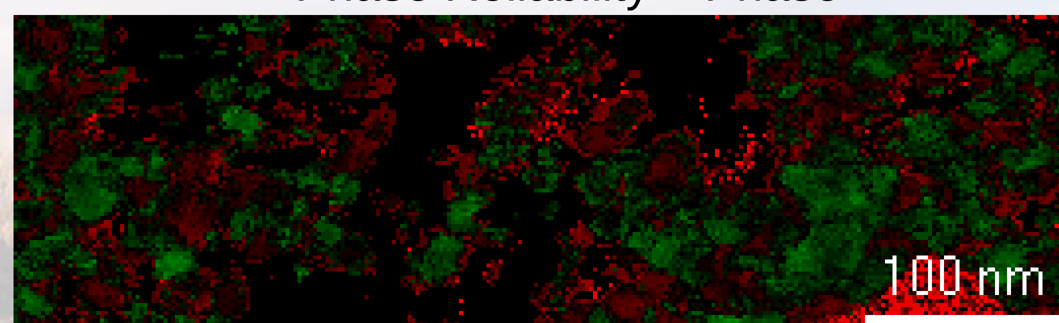
Virtual BF



Index + Phase (green = hydride)

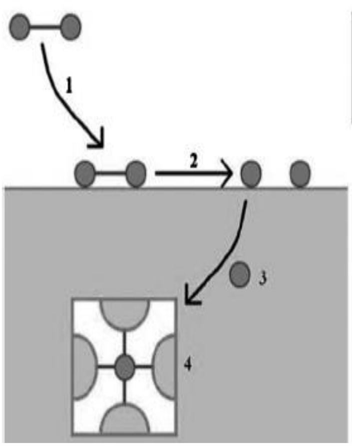


Phase Reliability + Phase

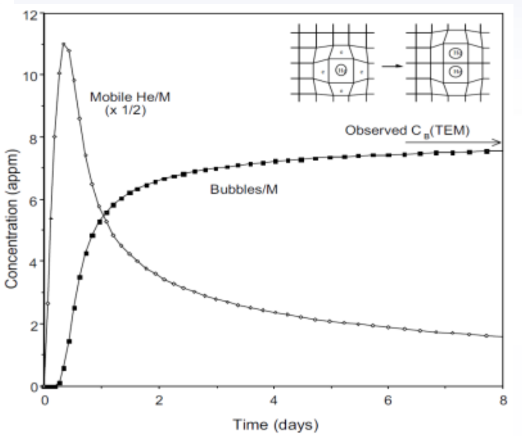


In situ TEM Hydrogen Exposure

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

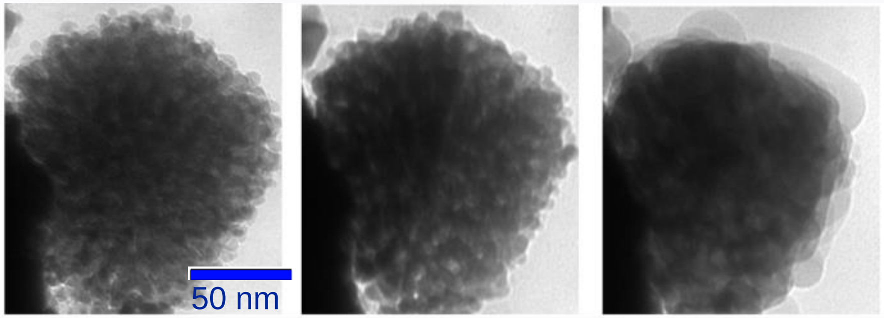


Cowgill, D., Fusion Sci. & Tech., 28 (2005) p. 539
Trinkaus, H. *et al*., JNM (2003) p. 229
Thiebaut, S. *et al*. JNM (2000) p. 217

Vapor-Phase Heating TEM Stage

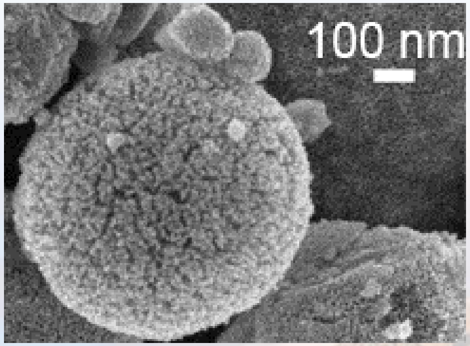
- Compatible with a range of gases
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Compatible with MS and other analytical tools

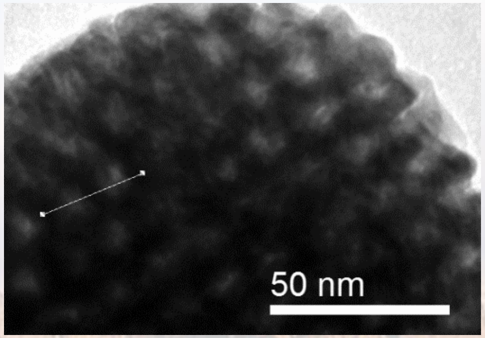
- 1 atm H_2 after several pulses to specified temp.



125° 200° 300°

Harmful effects may be mitigated in nanoporous Pd



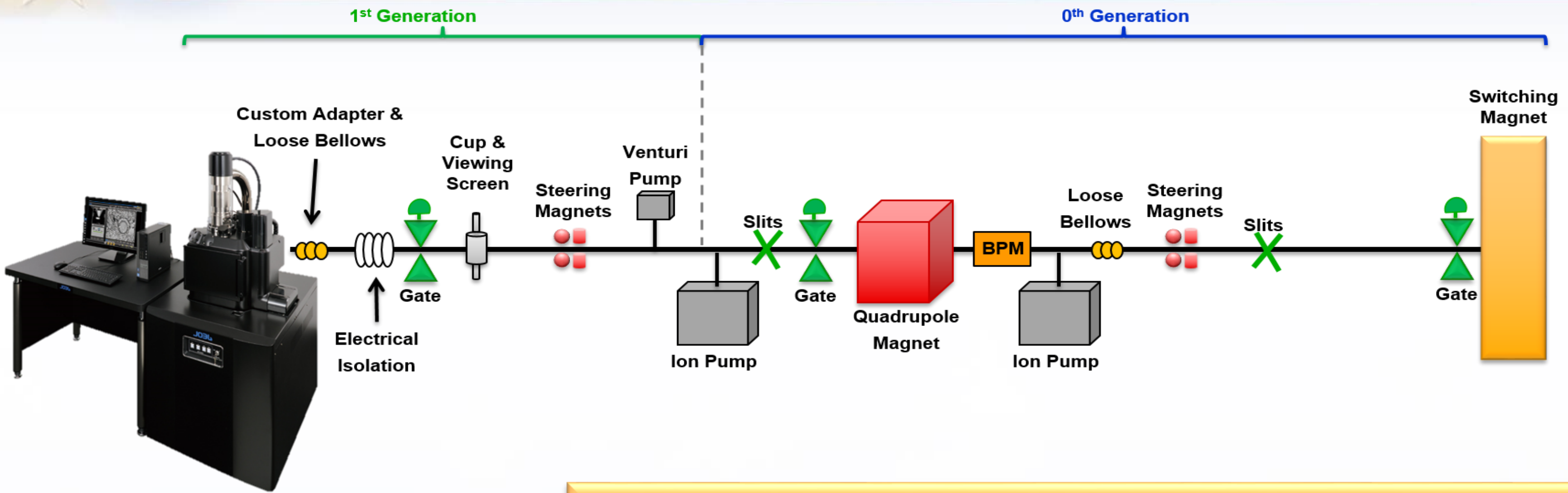


New *in situ* atmospheric heating experiments provide great insight into nanoporous Pd stability

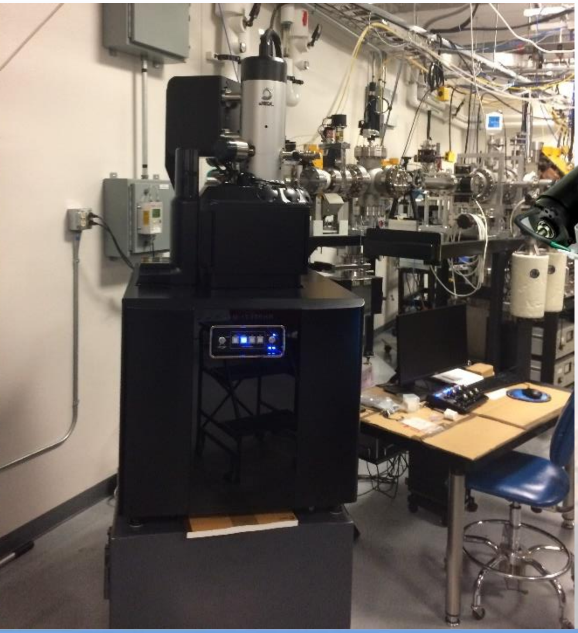
Schematic of the *In situ* SEM Beamline

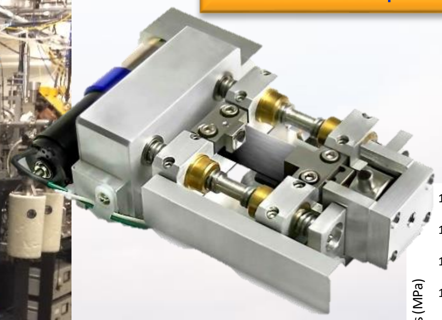
Collaborators: D.L. Buller & S. Briggs

8/24/2017

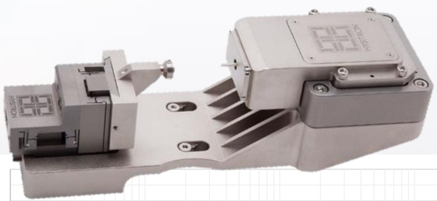


Beam Line planned for the *in situ* SEM will be developed in phases. Ultimate plan is for multiple accelerators being attached for dual or triple beam experiments.

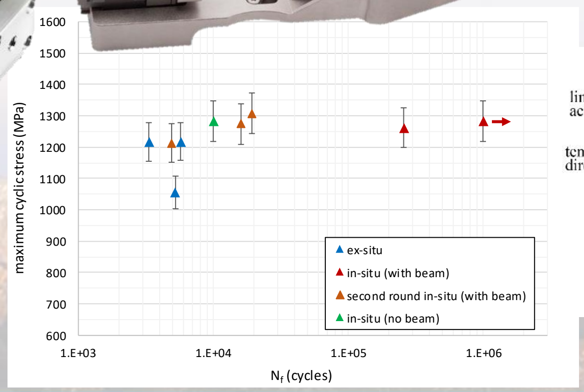




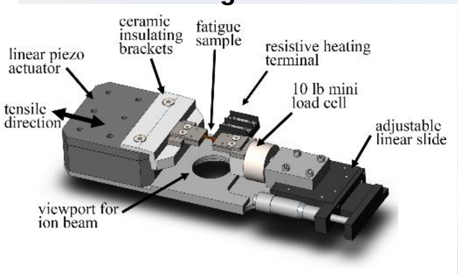
MTI Fullam Straining Heating



Hysitron PI85 Nanoindenter

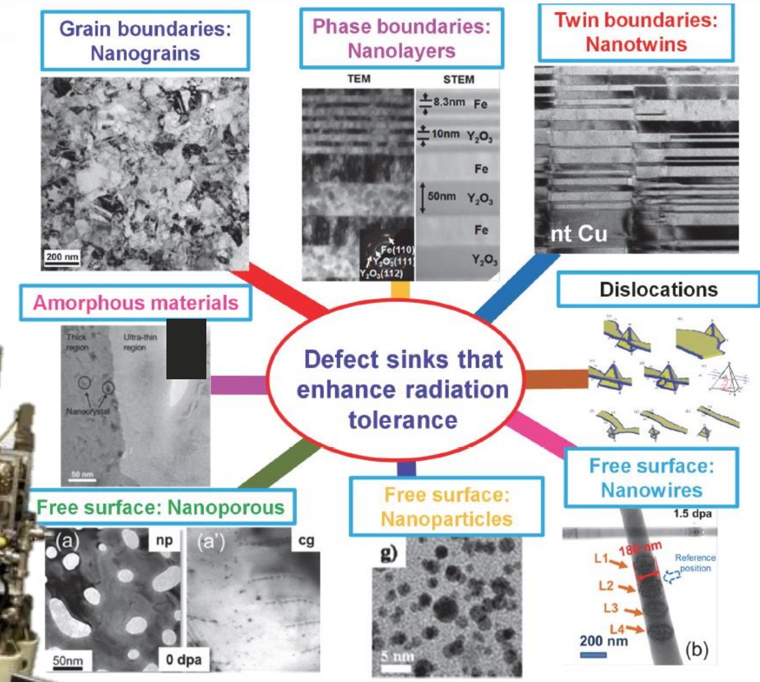


Custom-built Piezo Fatigue tester

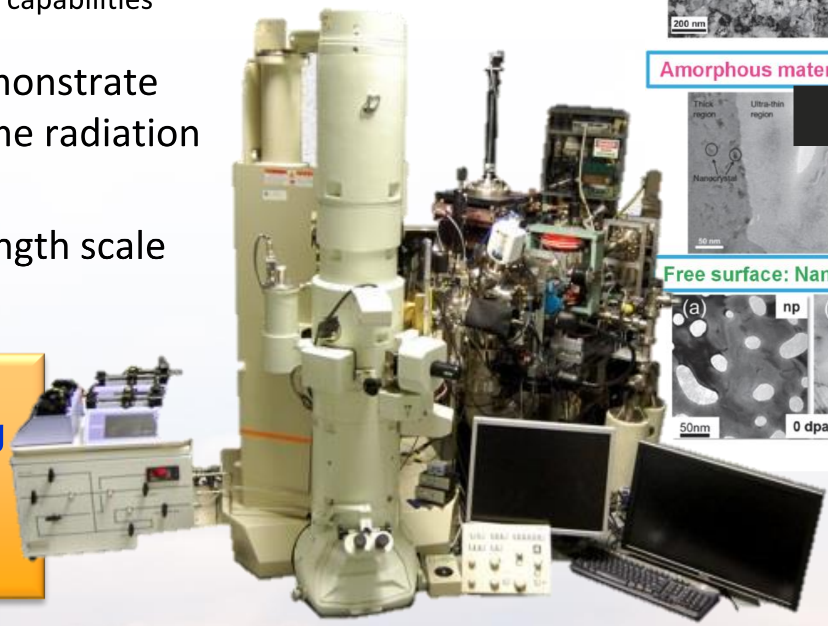


Summary

- Sandia's I³TEM is a unique facility for understanding mechanisms at the nanoscale
  - Only facility in the world with a wealth of overlapping *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



- Some nanostructures demonstrate improved response in some radiation environments, but **not** all
- More than just a single length scale maters



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

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 US Department of Energy (DOE) Office of Science by Los Alamos National Laboratory [Contract DE-AC52-06NA25396] and Sandia National Laboratories [Contract DEAC04-94AL85000]. 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.

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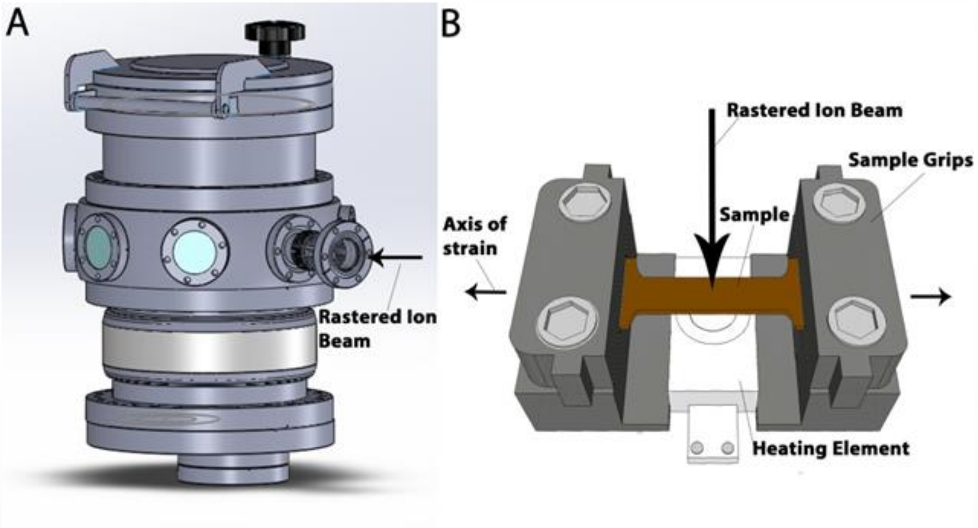
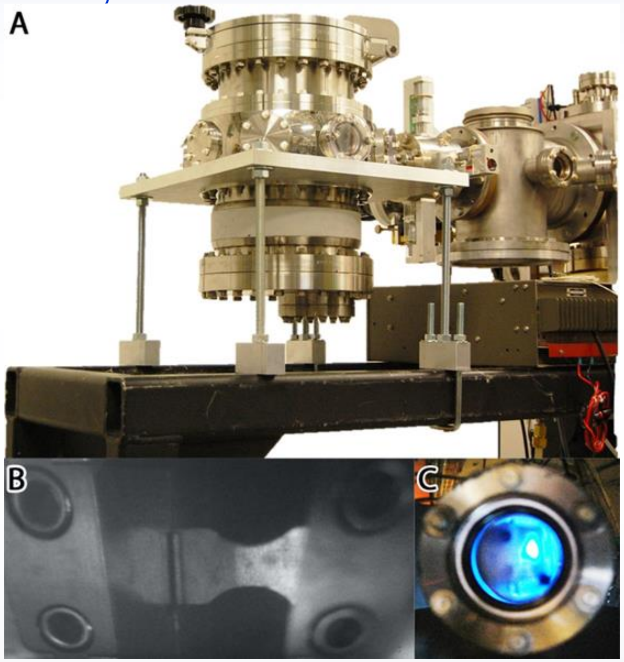
Back-up Slides

Mechanical Testing End Station

Collaborator: M. Steckbeck, B. Boyce, T. Furnish, D. Bufford, D. Buller, C. Barr

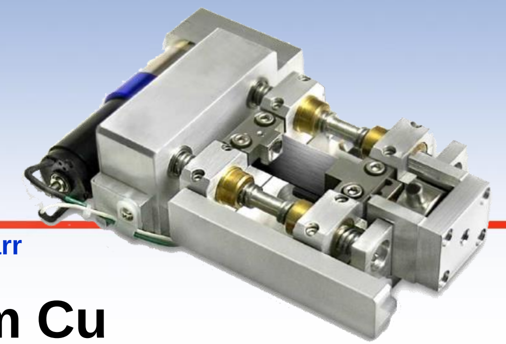




End-station developed consists of a two possible micro-mechanical test frames situated to receive a variety of ion species at energies up to 88 MeV from the 6 MV Van de Graff Tandem accelerator:

1. Commercial MTI/Fullham Multi-use Mechanical Stage (4000 N max): tensile, three point bend, creep, stress relaxation, others
2. Fatigue (custom design) for thin (5 to 10 micron samples)

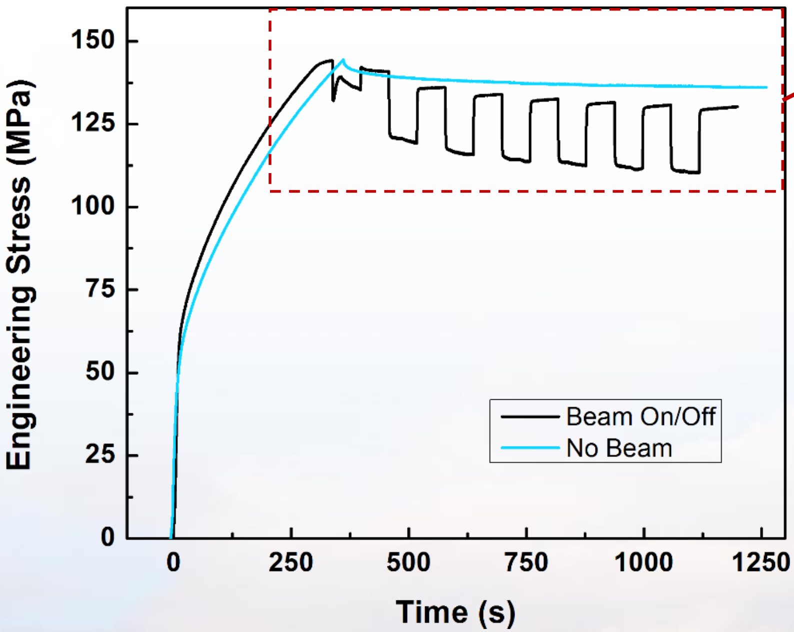
Irradiation and Stress Relaxation

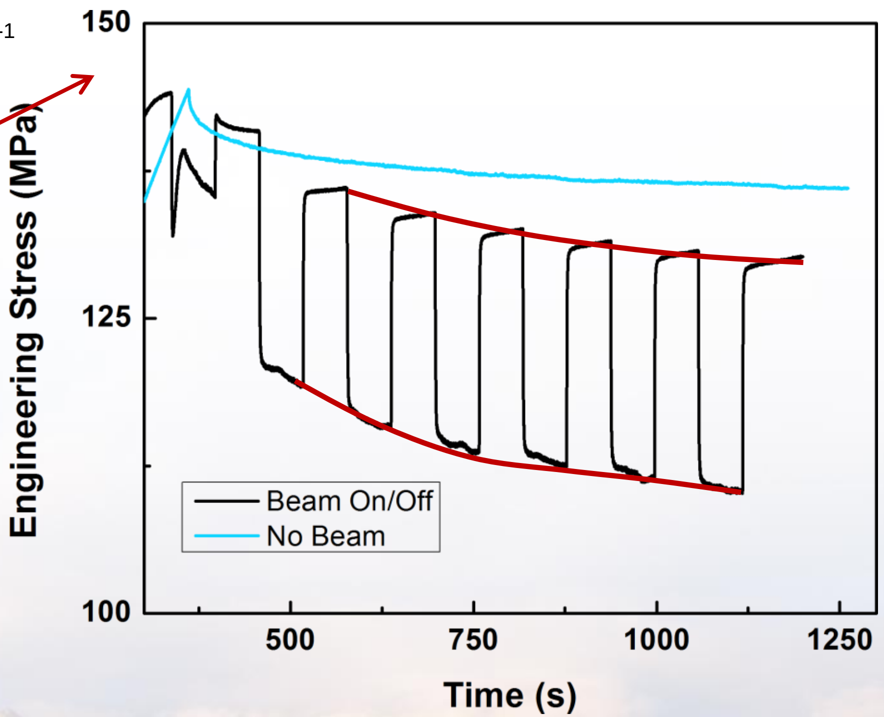


MTI/Fullham SEMTester

Collaborator: M. Steckbeck, B. Boyce, T. Furnish, D. Bufford, D. Buller, C. Barr

- **0.25mm/min elongation rate to 22.5 N load in 50 um Cu**
  - Approximately 75% of typical ultimate tensile load
  - 900 s hold at constant position
  - Beam cycled on/off at 60 s intervals
  - Beam conditions: 4.5 MeV H^+: 2.1 × 10^{11} ions $cm^{-2}s^{-1}$





- Offset likely an artifact
- Different relaxation rates with beam on and off

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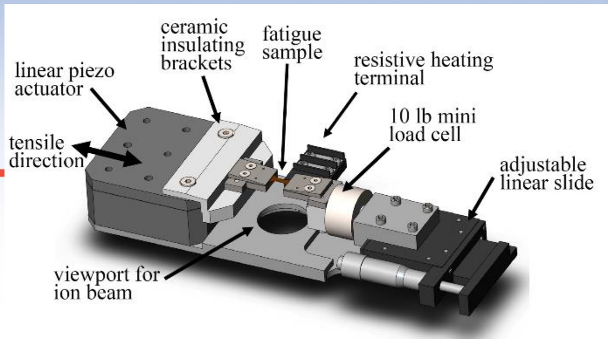
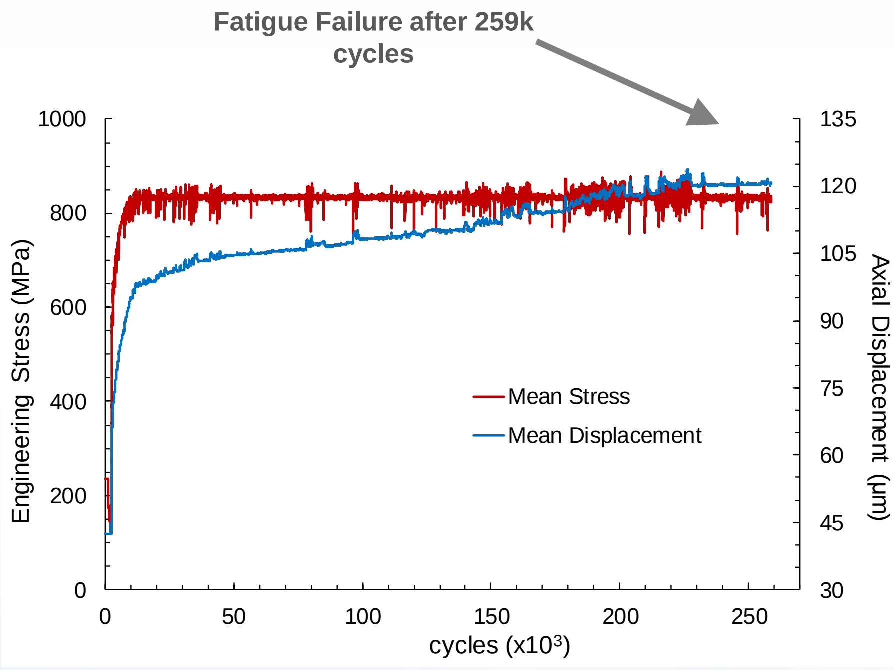
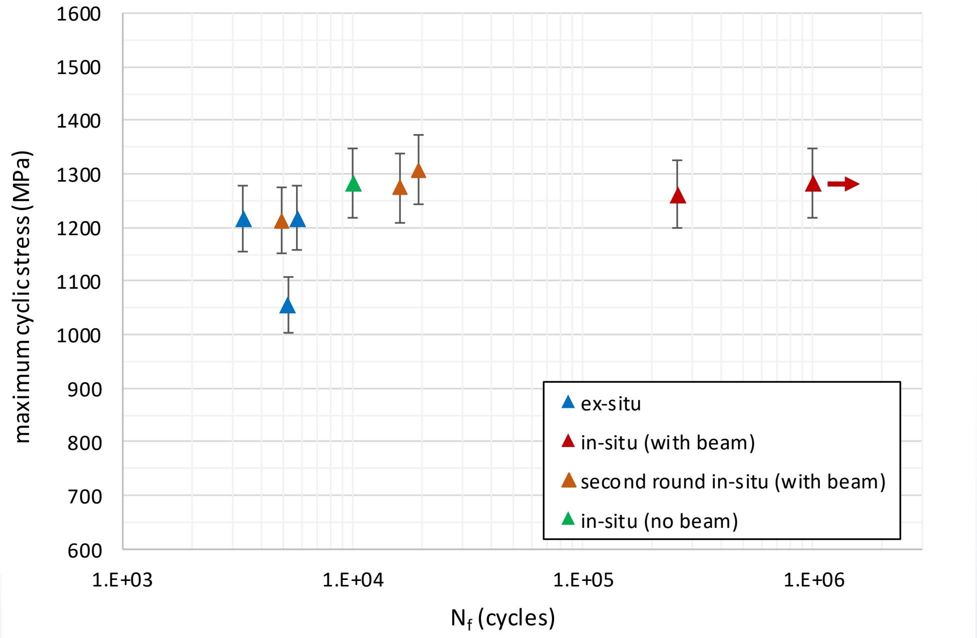
Irradiation and Fatigue



Collaborator: B. Boyce, T. Furnish, D. Buller





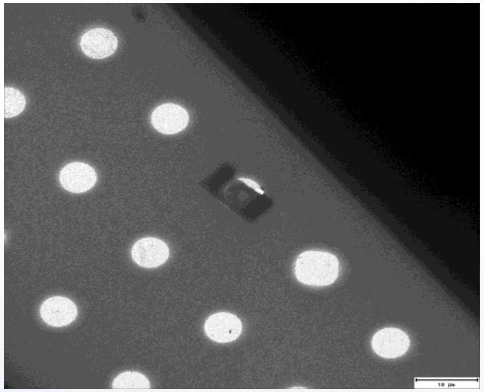
- Preliminary fatigue results in Ni-Fe alloy with both irradiation beam on sample (10 MeV He^+) and beam off sample.
- Custom mechanical stage designed for thin samples (5 to10 um thickness: suitable for allowing entire thickness to be irradiated with raster or defocused beam)

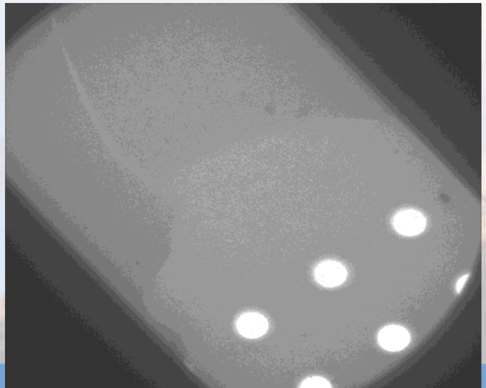
Feasibility of Studying Zircaloy 2 at Nominally 1 atm

Collaborators: S. Rajasekhara and B.G. Clark

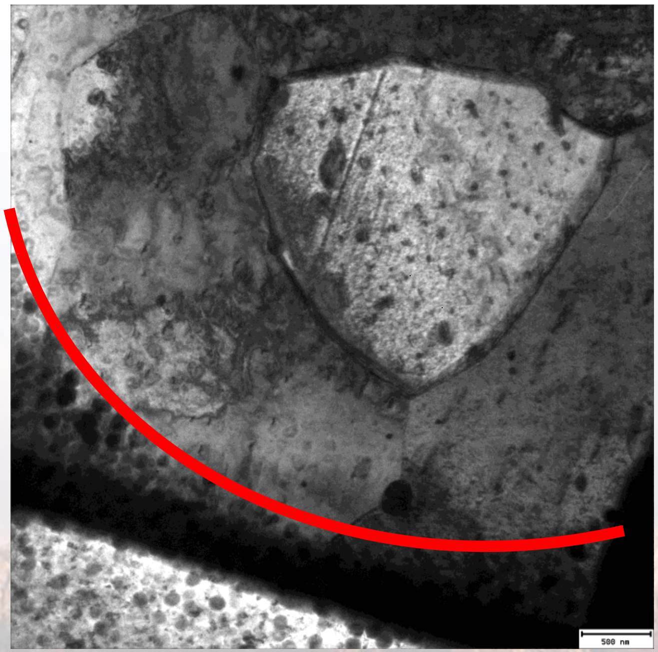
Vapor-Phase Heating TEM Stage

- 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

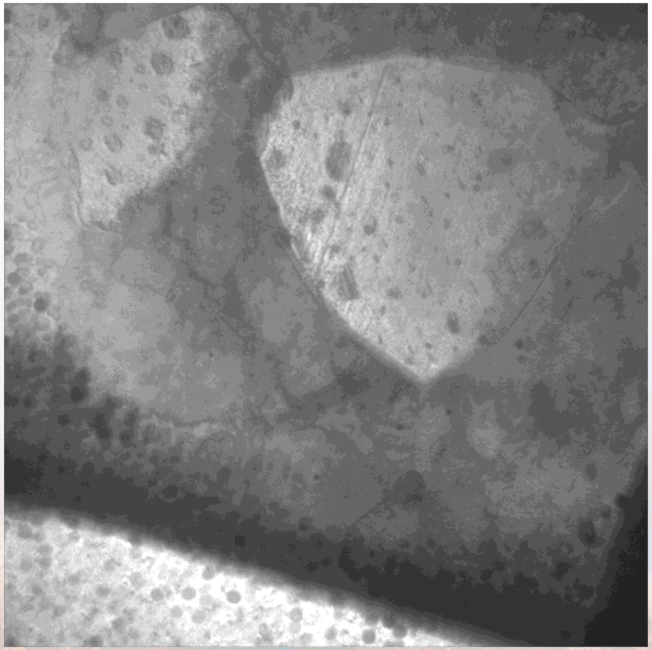




Vacuum & Single Window



Nominally 1 atm H_2 & Two Windows



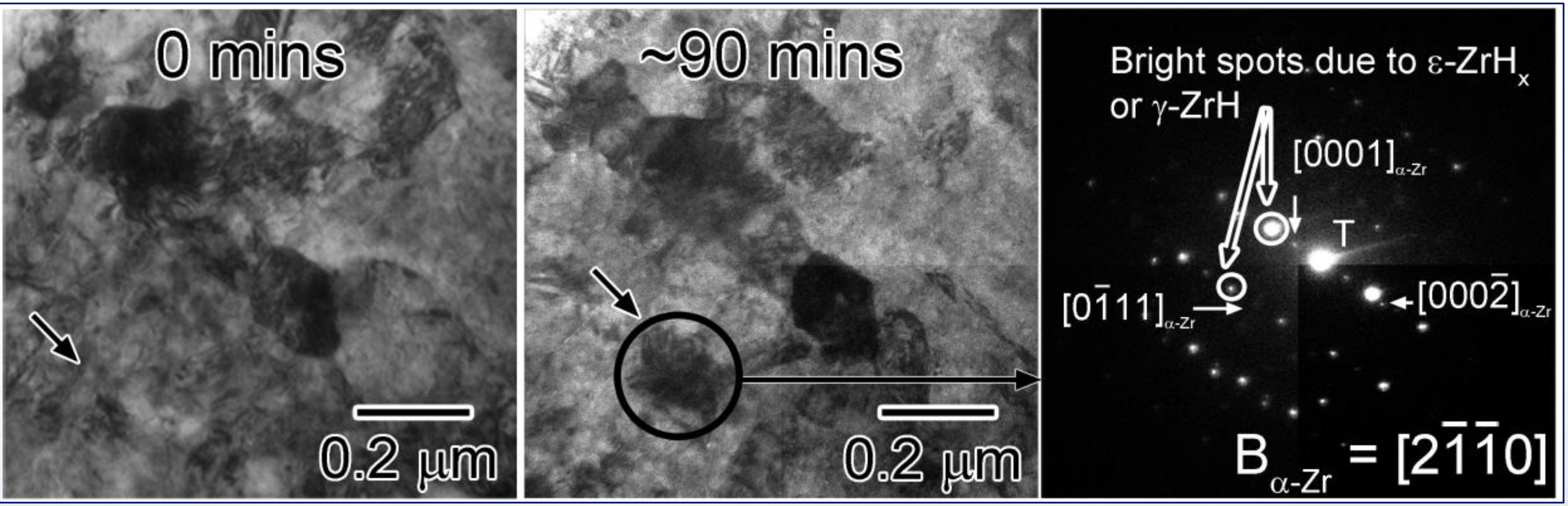
Most features are observed in both despite the decreased resolution resulting from the additional SiN window and 5 μm of air

In situ Observation of Hydride Formation in Zirlo

Collaborators: S. Rajasekhara and B.G. Clark

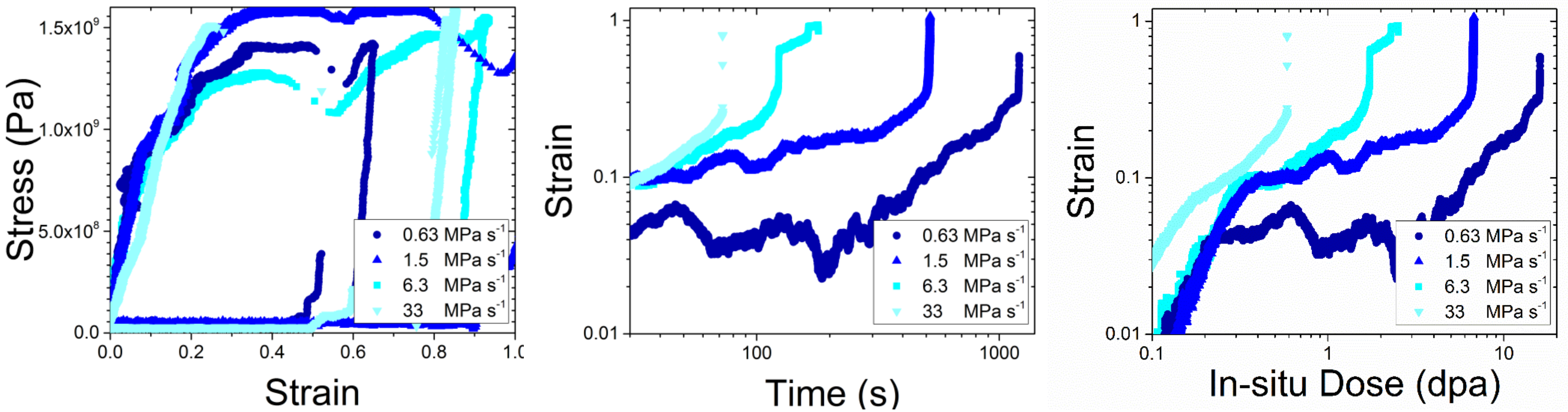
Absolute hydrogen pressure: 327 torr (~ 0.5 atm),

Ramp rate: 1 ^{o}C/s, Final temperature: ~ 400 C, Dwell time: ~ 90 mins



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

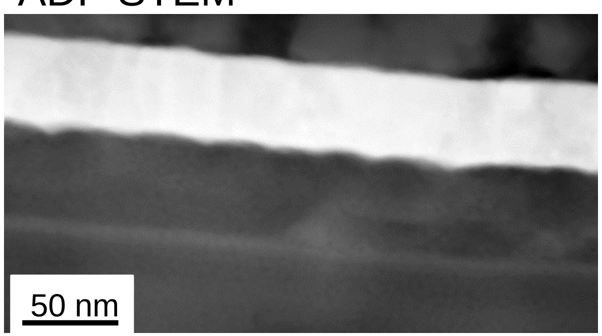
Creep Response at Different Loading Rates



Significant creep observed at a fraction of the bulk yield strength

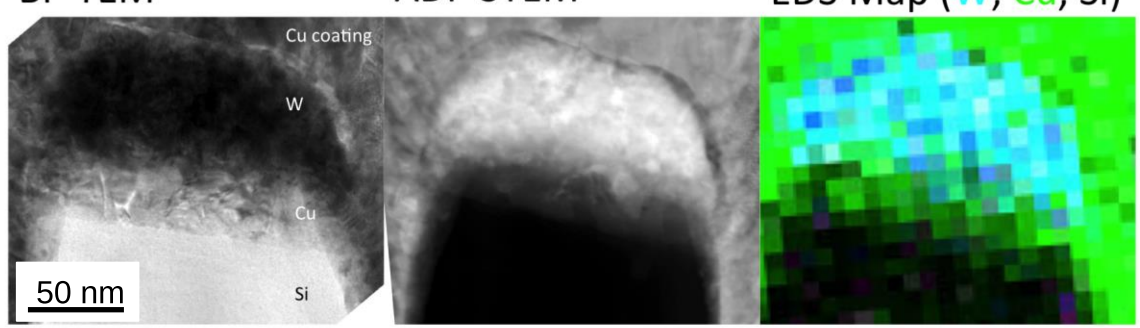
As-deposited Sample

ADF-STEM



Post Creep Characterization

BF-TEM ADF-STEM EDS Map (W, Cu, Si)



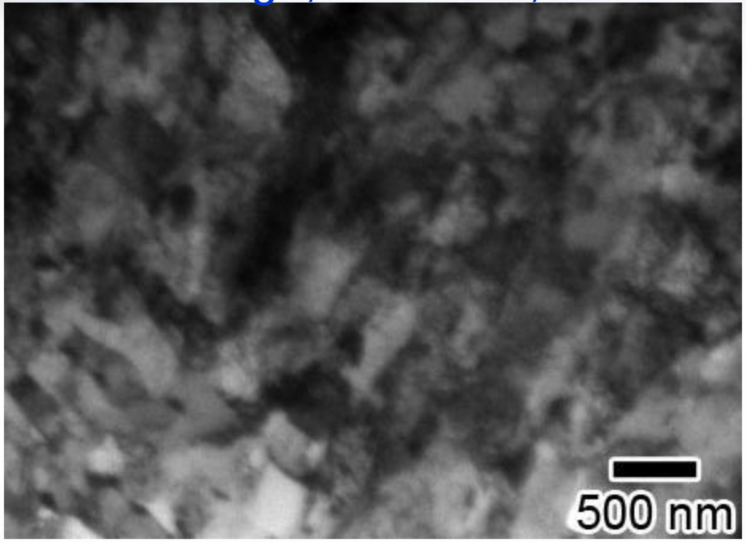
Compression (creep) only observed in Cu layer

In situ TEM Ion Irradiation

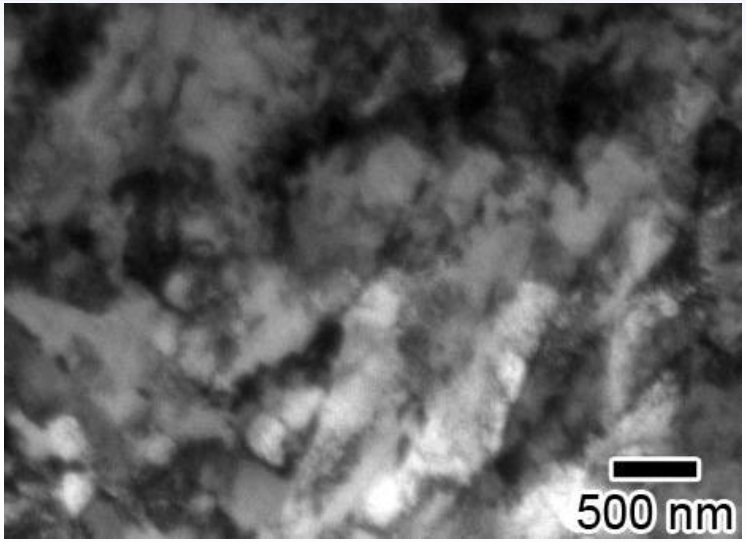
HT9   3 MeV Cu^{3+} at ~10 nA   RT

Collaborators: A. Kilgo, J. Puskar, and S. Maloy

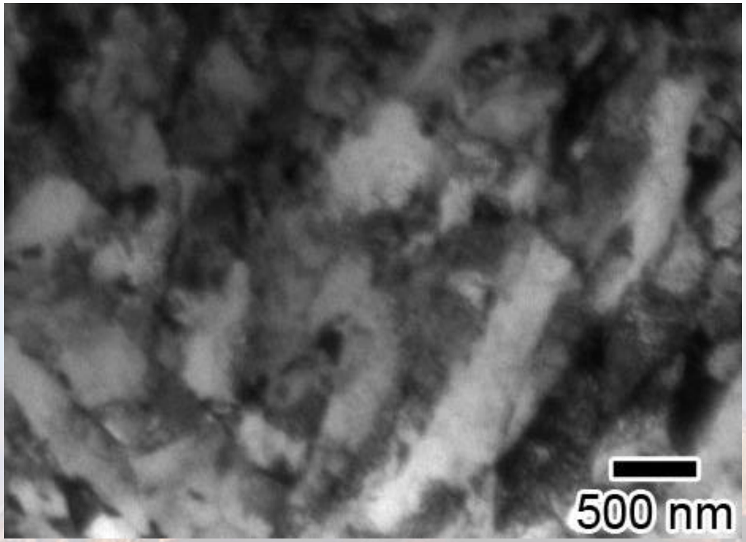
Initial



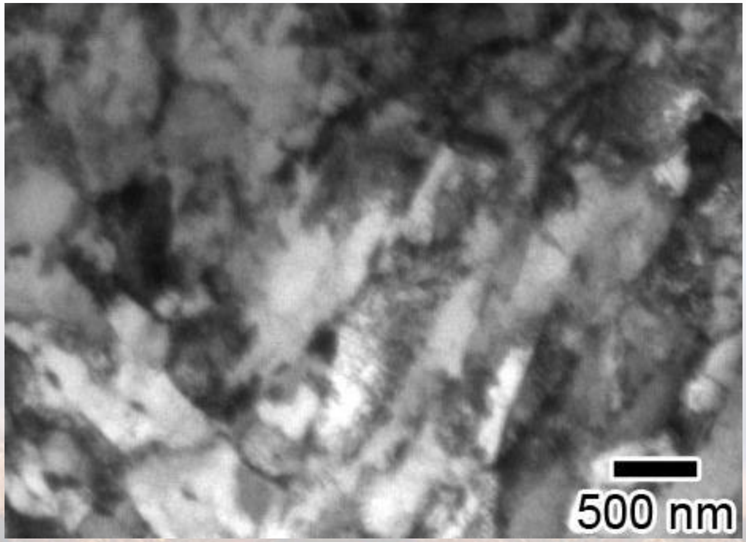
2 hrs



3 hrs



6 hrs



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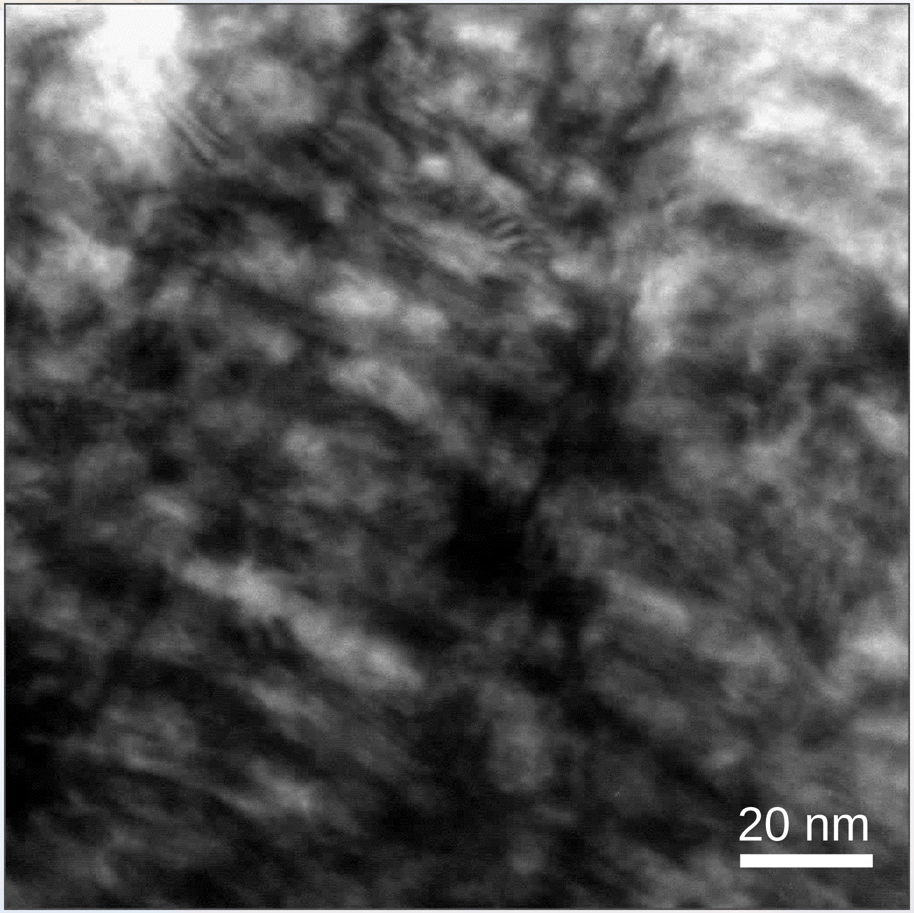
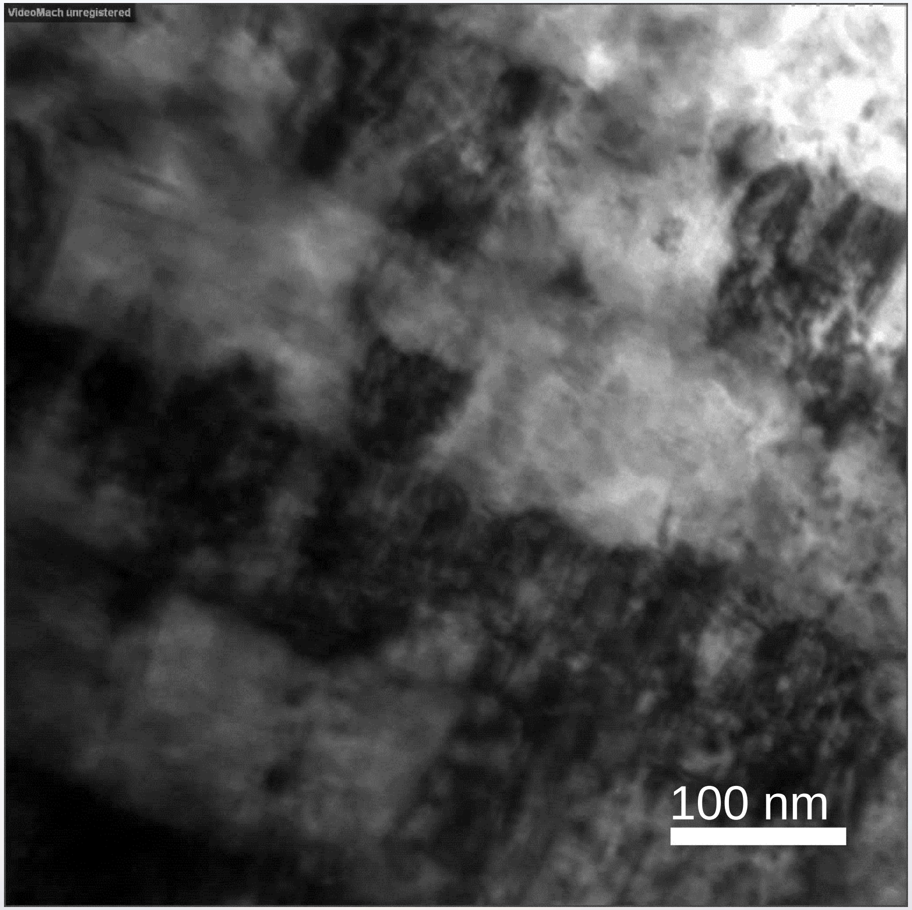
Layer Deposition to Provide Defect Sinks

Collaborators: Y. Chen, N. Li, D. Bufford, X. Zhang





In situ **TEM self-ion irradiation of Cu/Fe 100 nm and 5 nm multilayers with 3 MeV Cu**

Film deposition of layered structures can also provide an opportunity for radiation defect sinks

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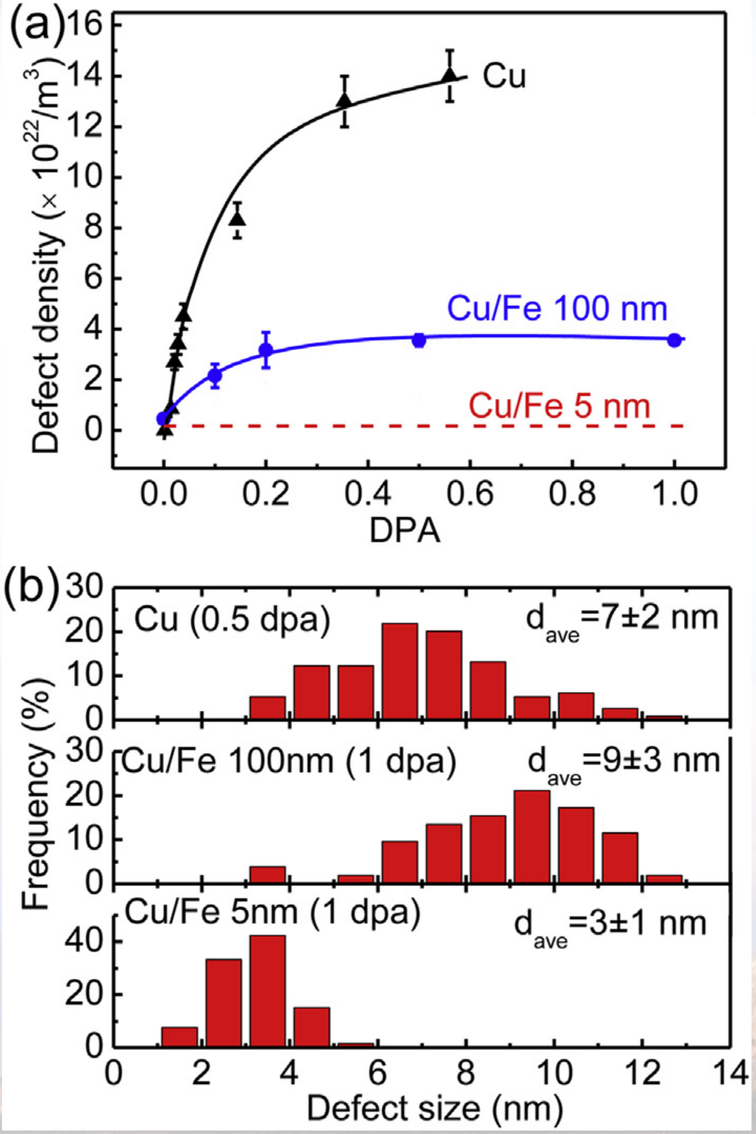
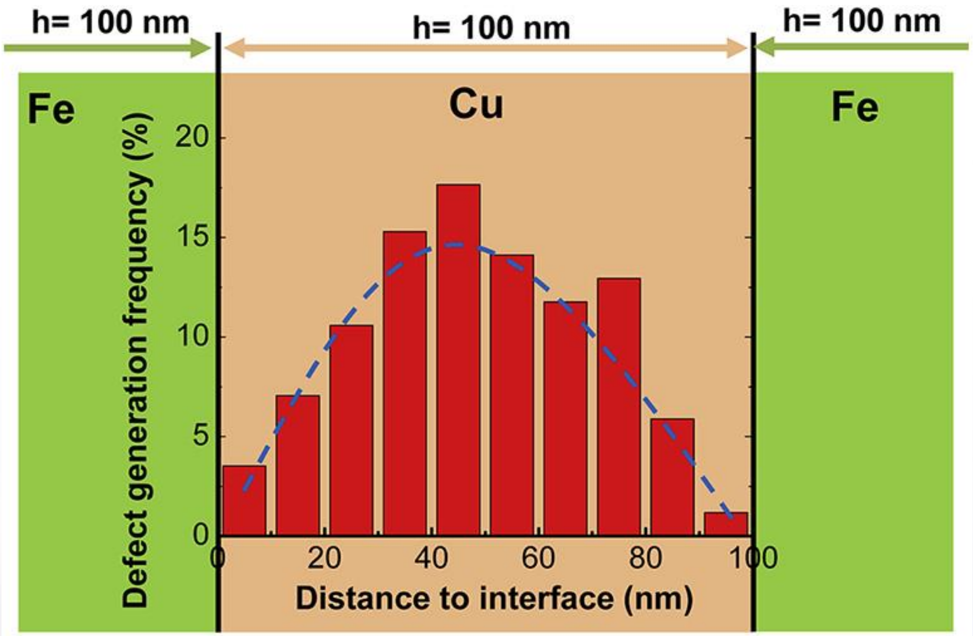
Layer Deposition to Provide Defect Sinks

Collaborators: Y. Chen, N. Li, D. Bufford, X. Zhang





- **Layered structure provides significant improvement over either pure system.**
- **The radiation tolerance improves with decreasing layer thickness.**