Studies of Radiation Effects on Mechanical Properties using *In situ* SEM and TEM

SAND2018-11498C

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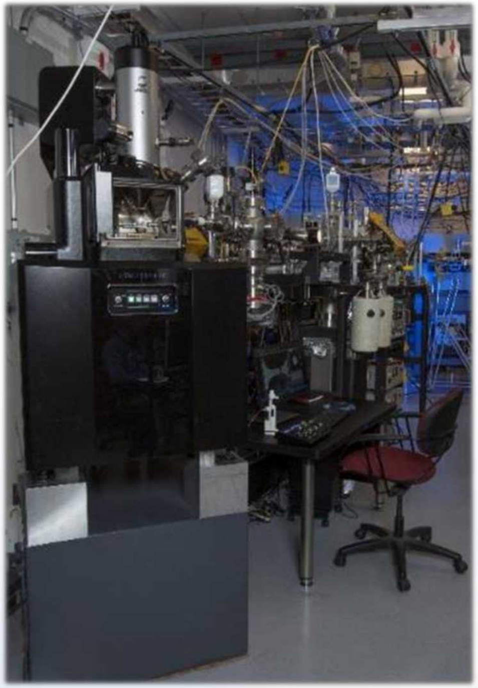
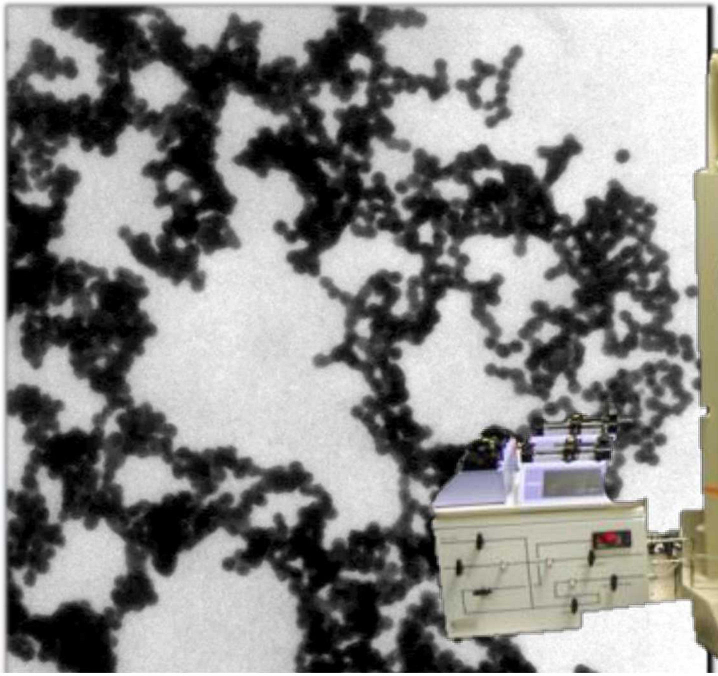
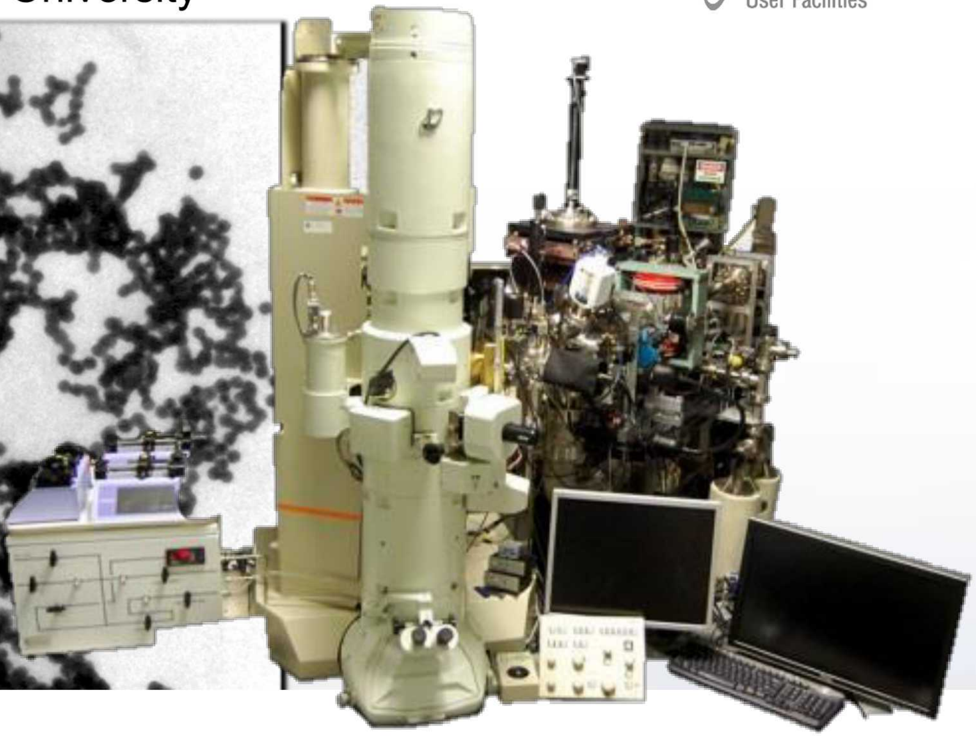






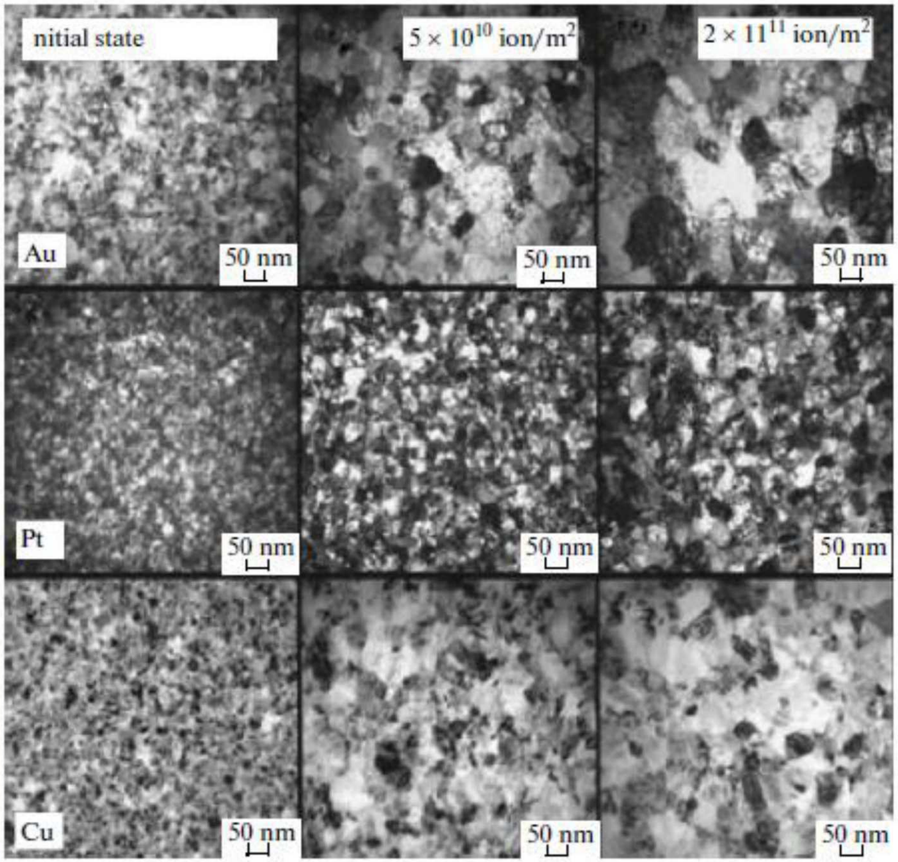
Collaborators:

- 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.

This work was partially funded by the Division of Mate[...]eering, Office of Basic Energy Sciences, U.S. Department of Energy. This work was performed, in part, [...] 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.

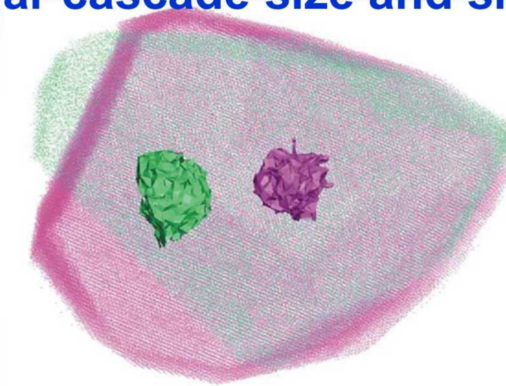
Section Summary: Radiation Tolerance from Nanostructured Metals

Variation in radiation tolerances



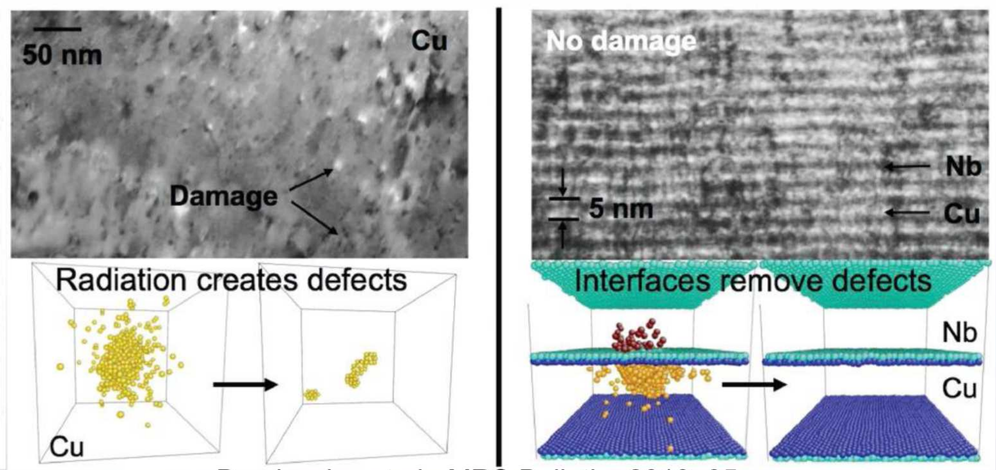
Kaomi et al., JAP: 2008. 104 073525

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

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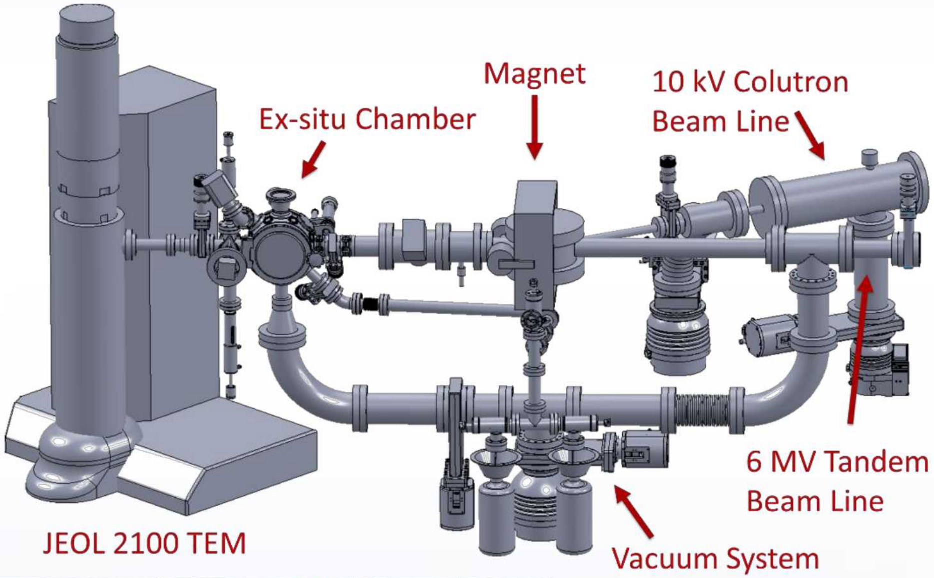
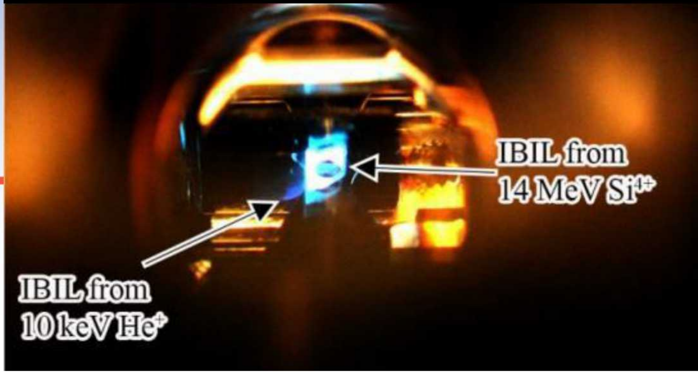
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

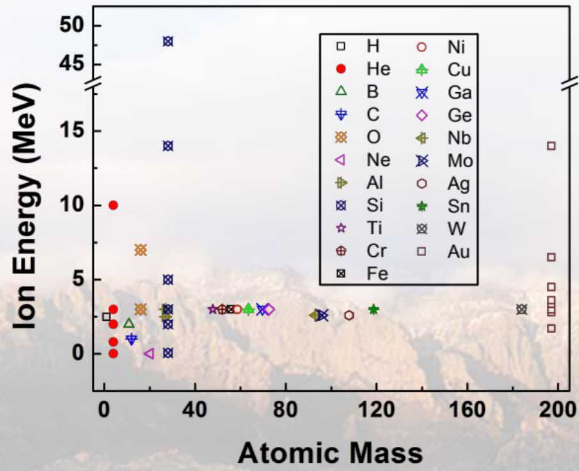
Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem



IBIL from a quartz stage inside the TEM

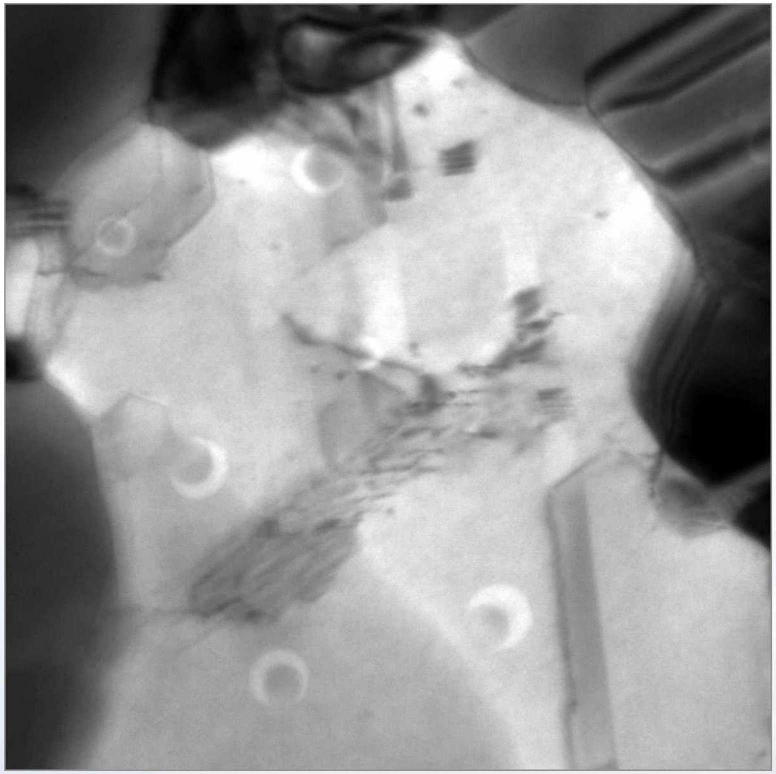




Dose Rate Effects in Nanocrystalline Metals

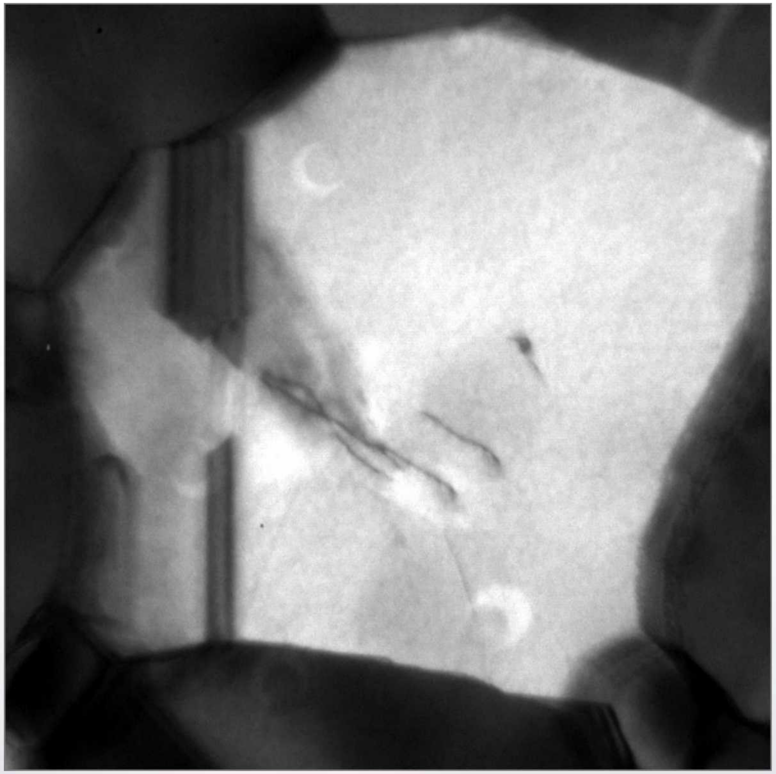
Collaborators: C. Chisholm , P. Hosemann, & A. Minor

7.9 x 10^9 ions/cm^2/s



VS

6.7 x 10^7 ions/cm^2/s

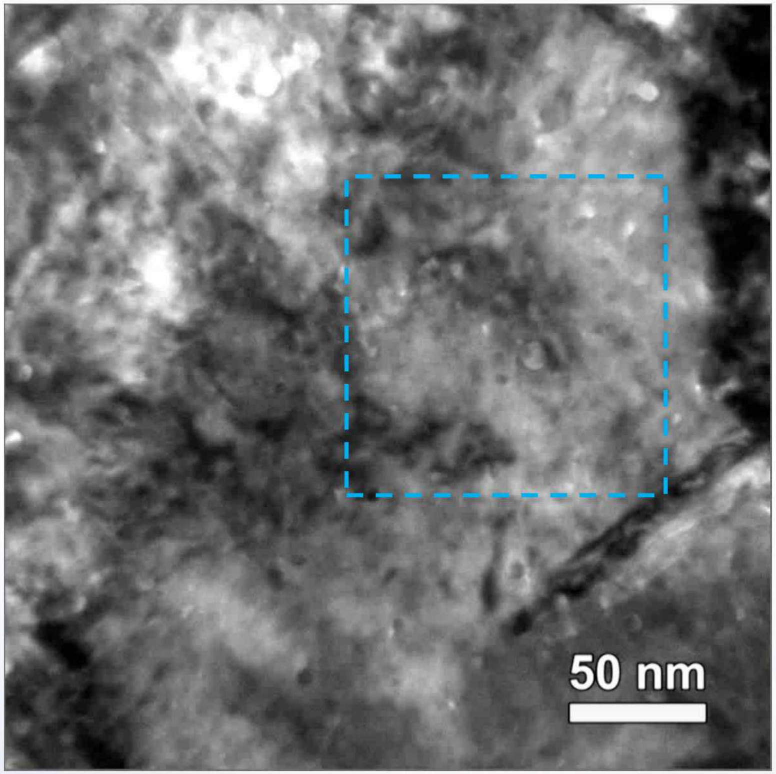


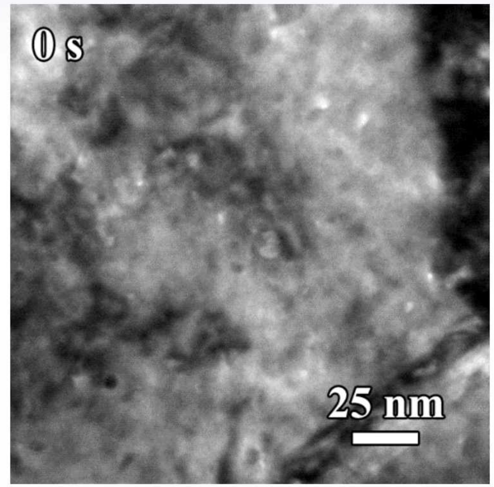
Improved vibrational and ion beam stability permits us to work at 120kx or higher permitting imaging of single cascade events

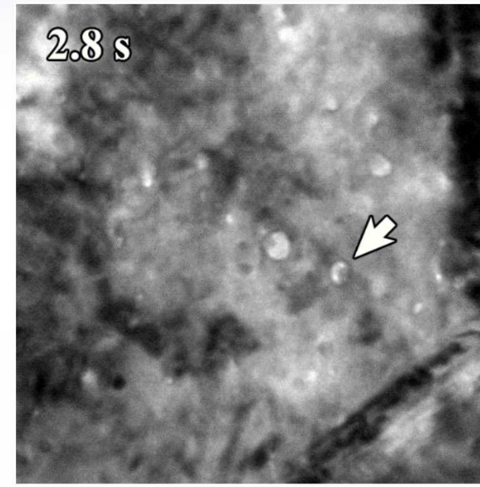
Simultaneous *In situ* TEM Triple Beam: 2.8 MeV Au^{4+} + 10 keV He^{+}/D_2^{+}

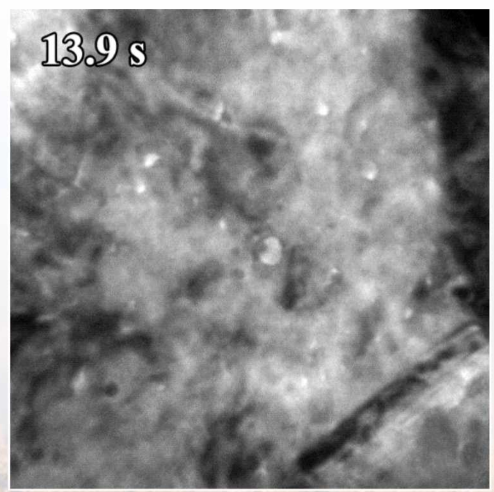
Collaborator: D.C. Bufford

Video playback speed x1.5.







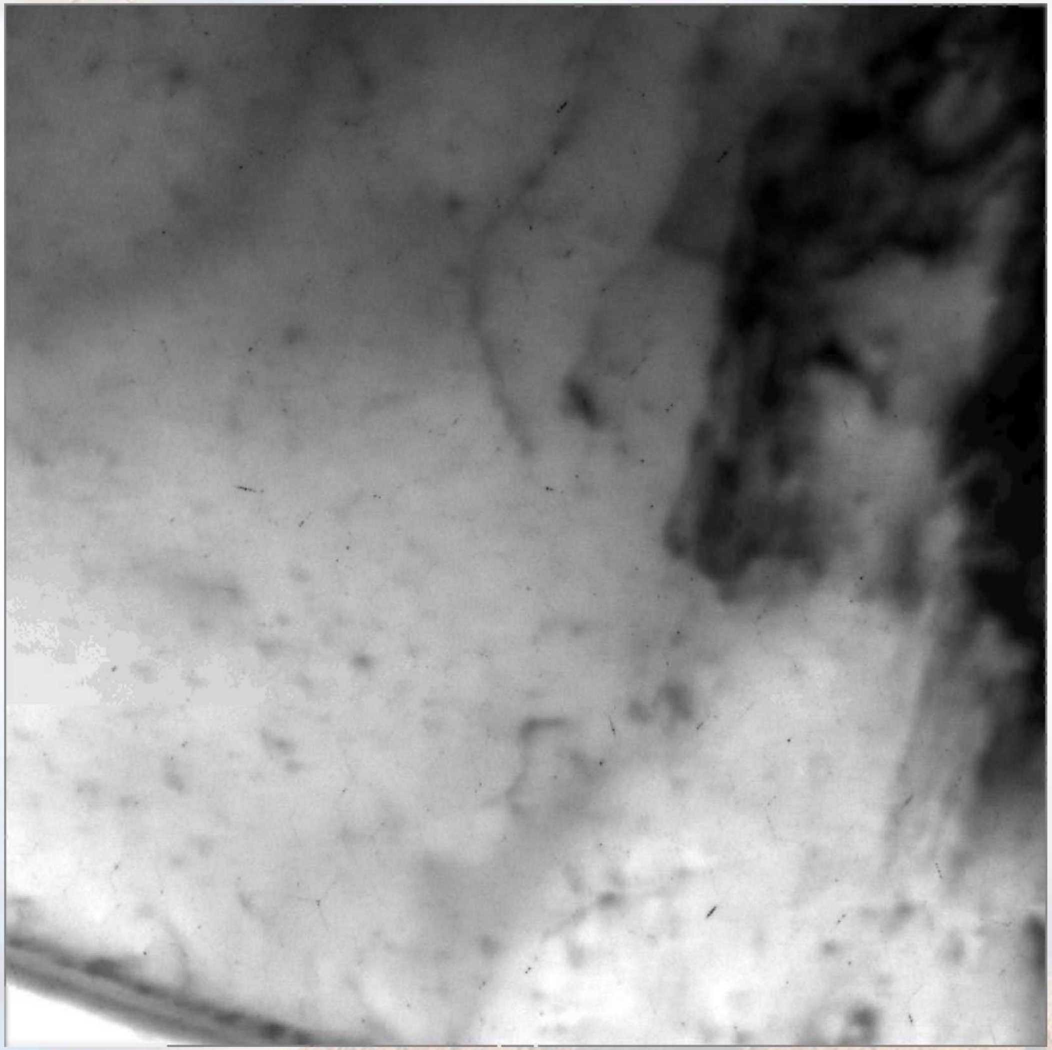


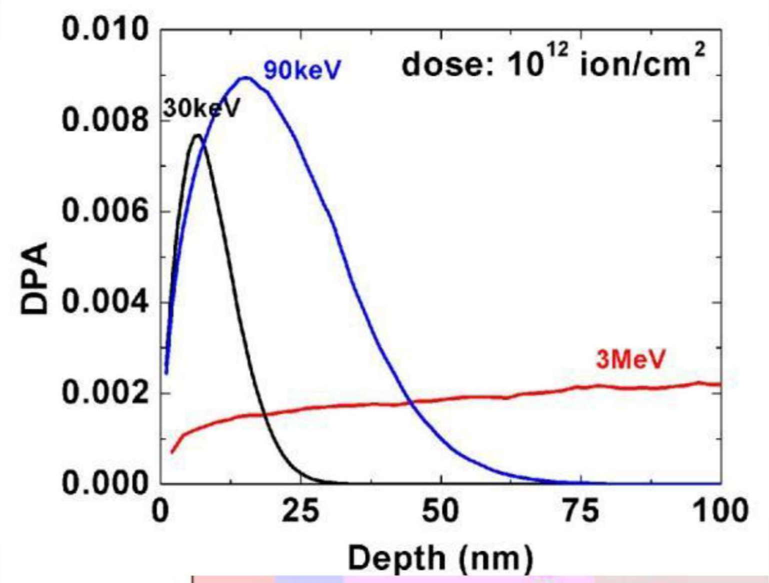
- **Approximate fluence:**
  - Au 1.2 $\times 10^{13}$ ions/cm^2
  - He 1.3 $\times 10^{15}$ ions/cm^2
  - D 2.2 $\times 10^{15}$ ions/cm^2
- **Cavity nucleation and disappearance**

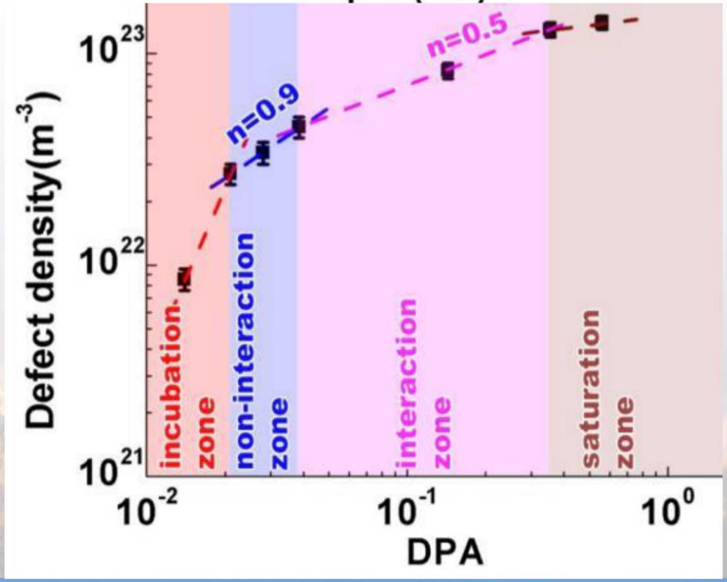
In-situ triple beam He, D_2, and Au beam irradiation has been demonstrated on Sandia's I^3TEM! Intensive work is still needed to understand the defect structure evolution that has been observed.

Quantifying Defect Evolution

Collaborators: N. Li & A. Misra

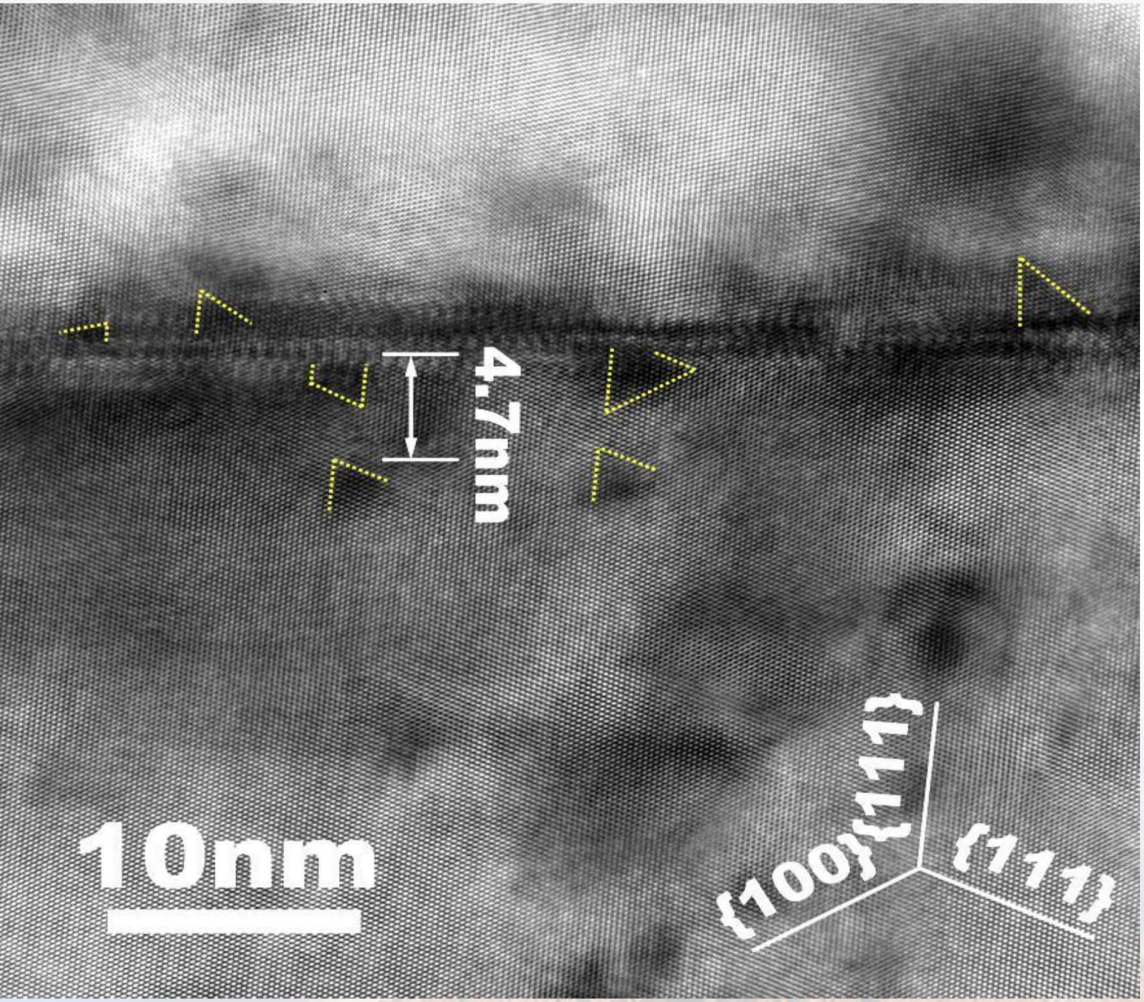


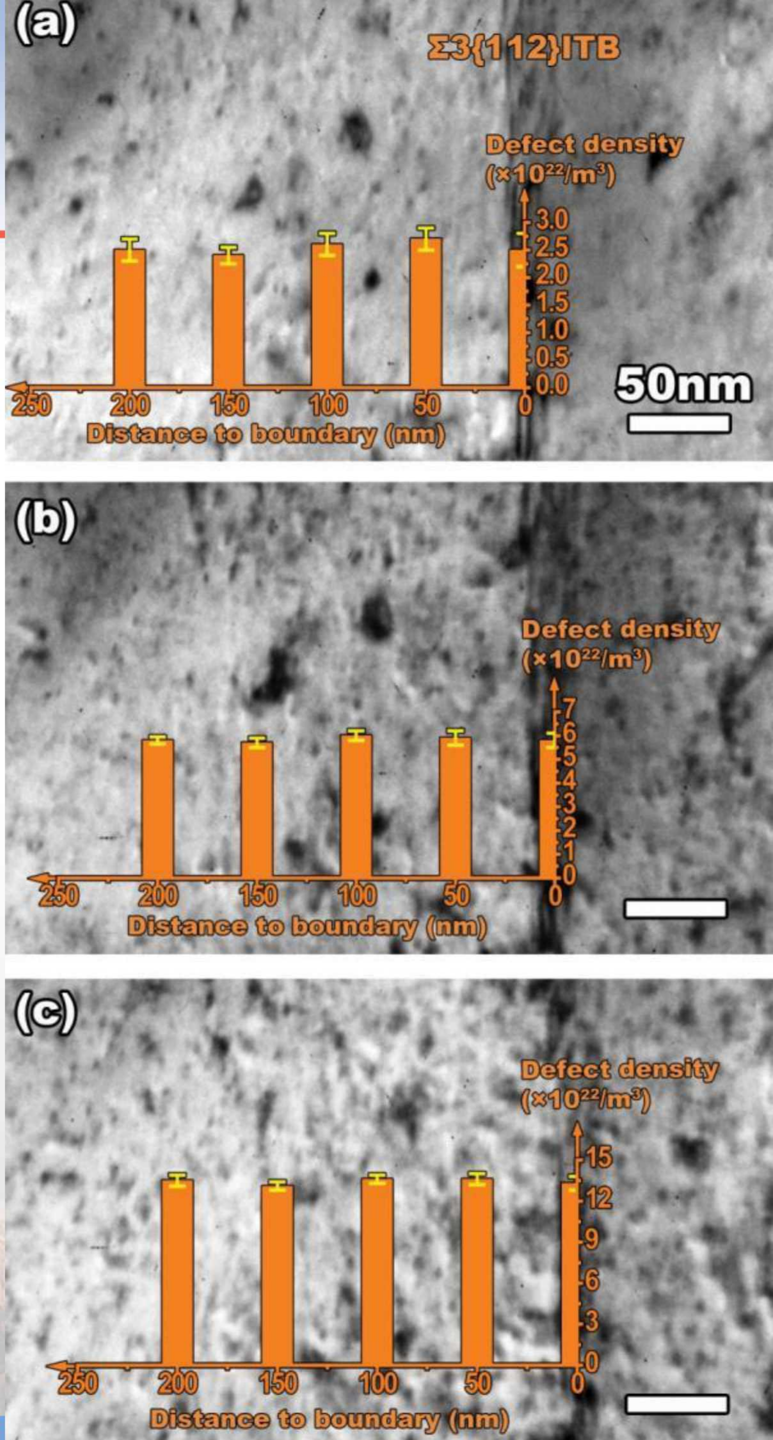




Defects are Altered Little by the Presence of Grain Boundaries

Collaborators: N. Li & A. Misra



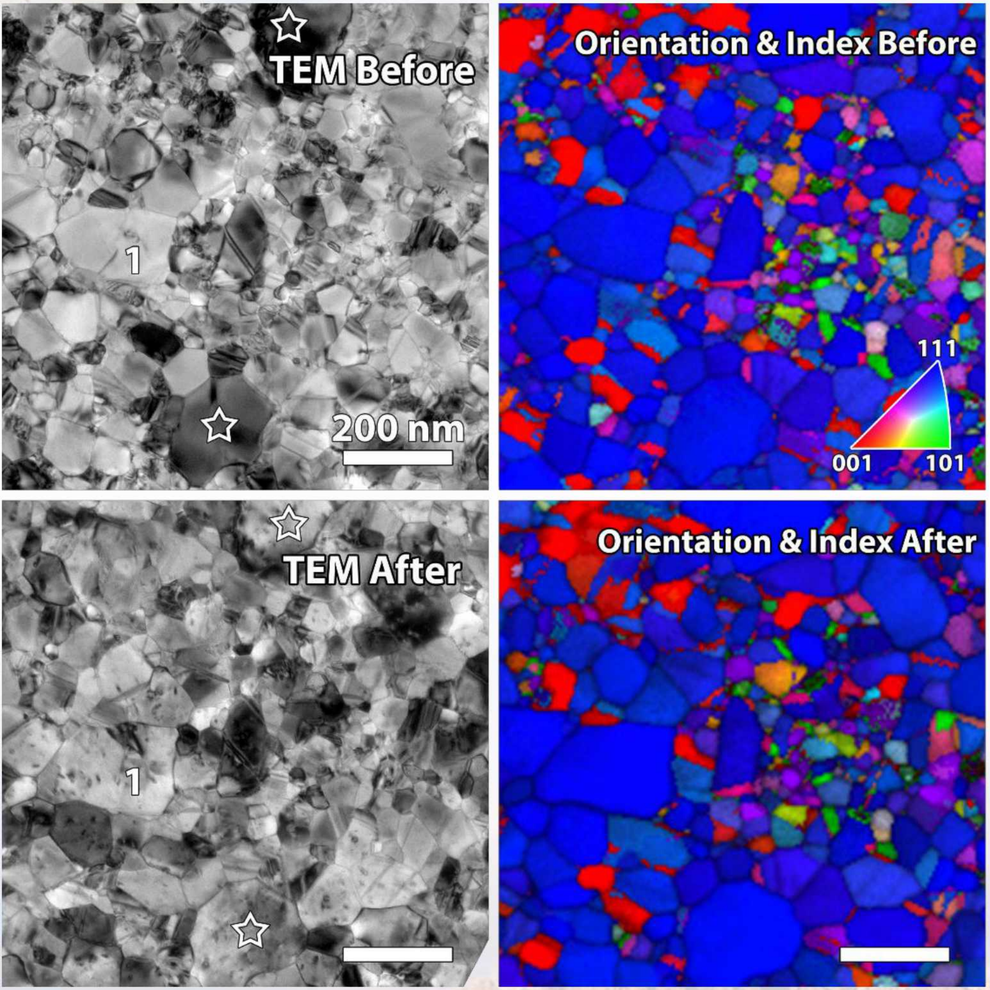


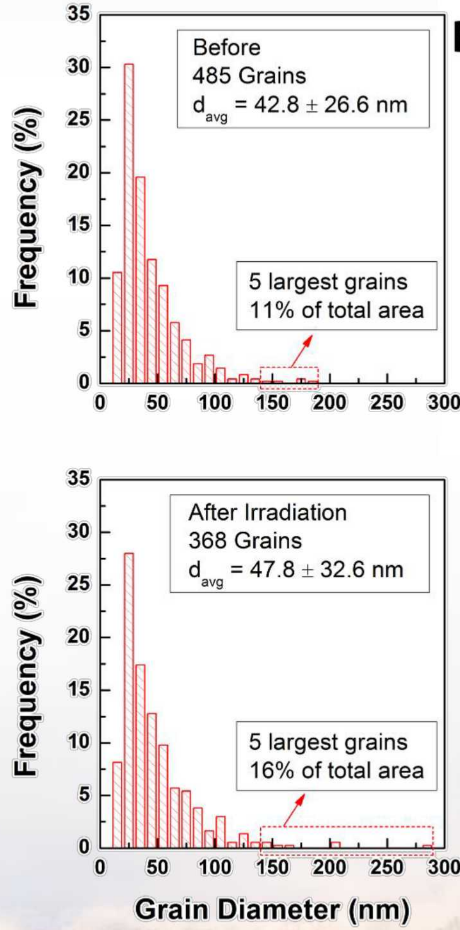
SFT appear to be directly at GB

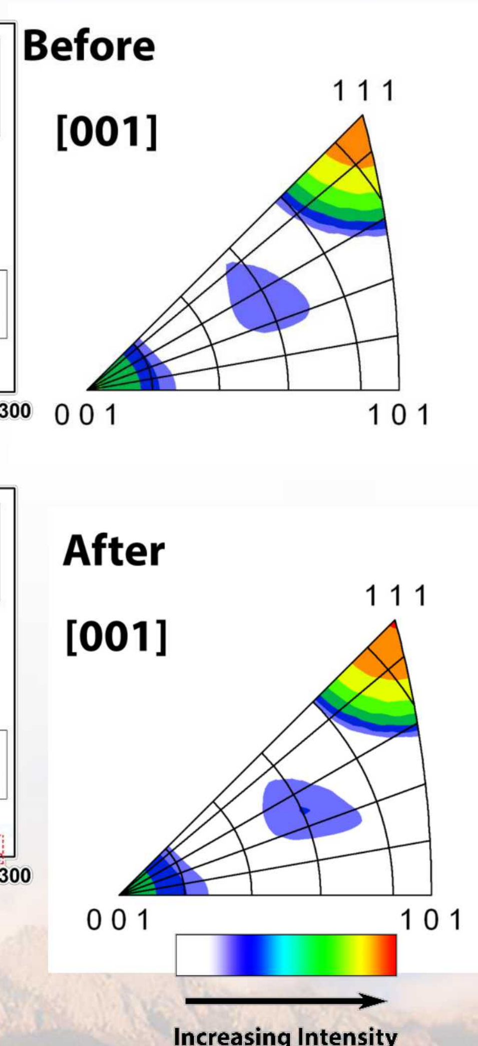
No change in defect density is observed near GB

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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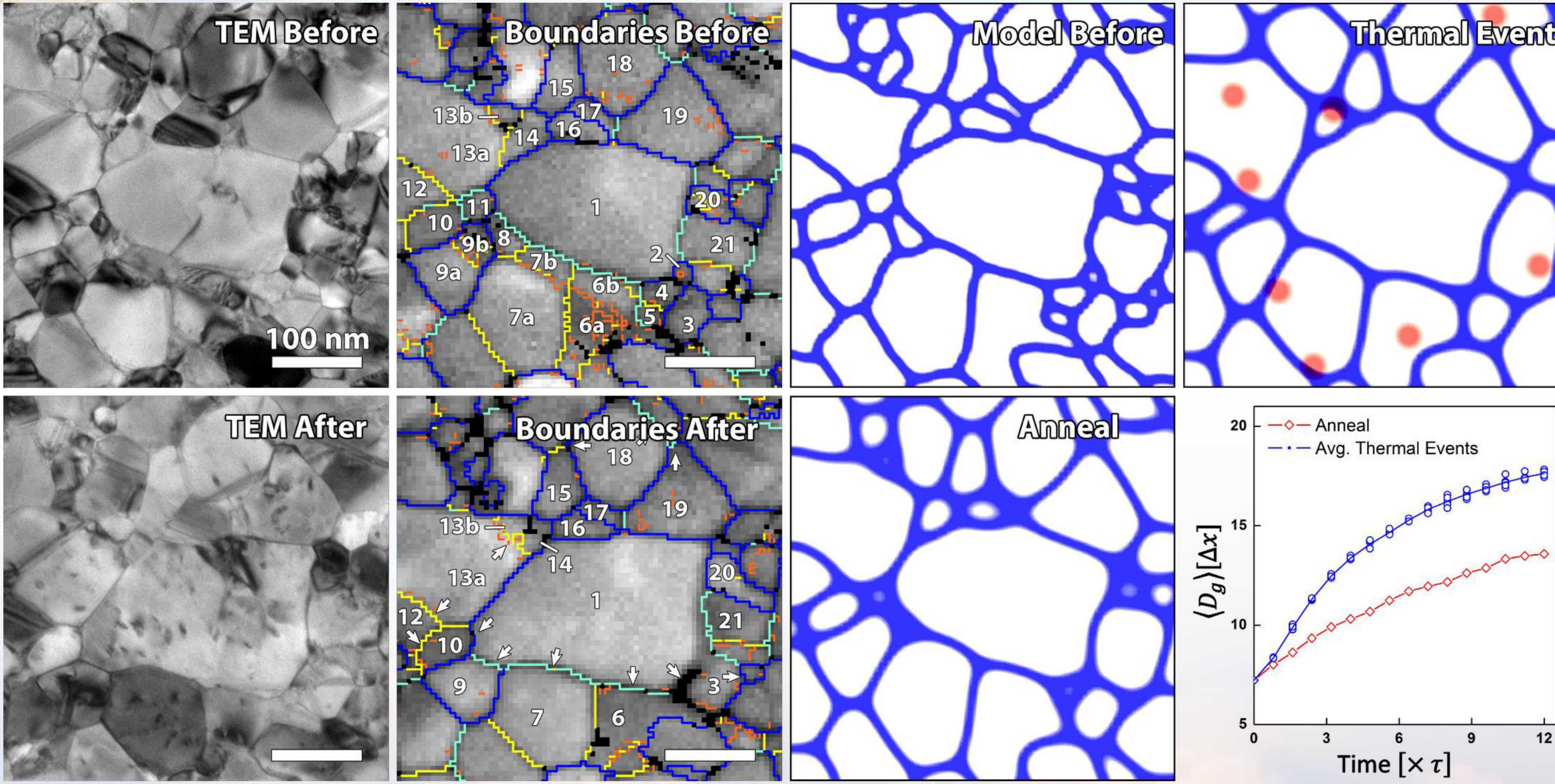
Direct Comparison to Mesoscale Modeling

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.

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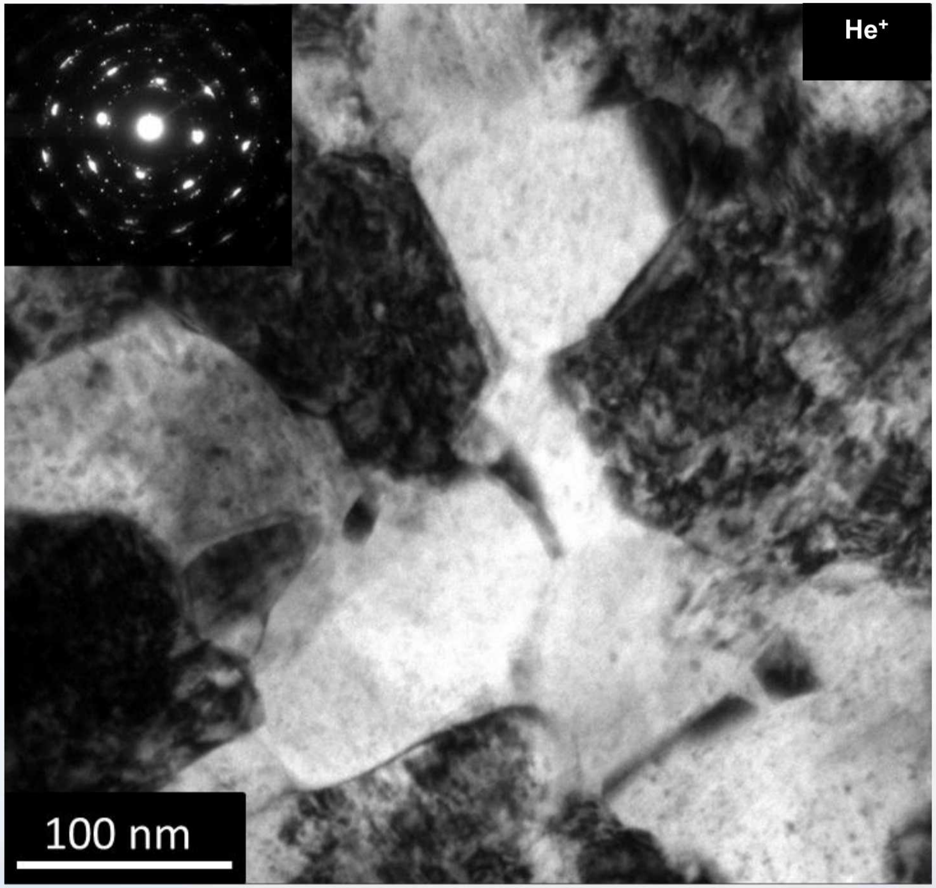
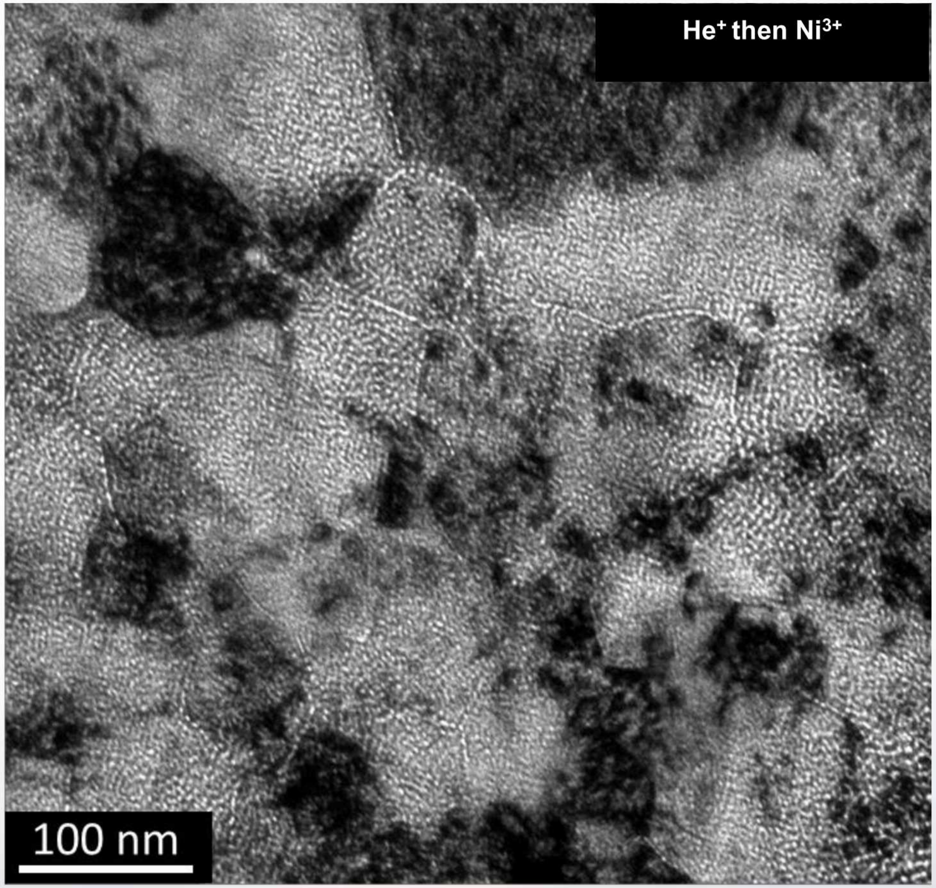
Heterogeneous Bubble Formation under Some Radiation Environments

Collaborator: B. Muntifering & J. Qu





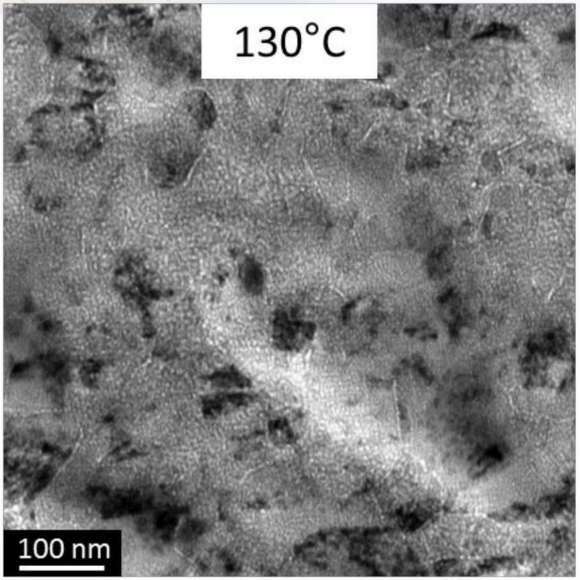
10^{17} He^+/cm^2

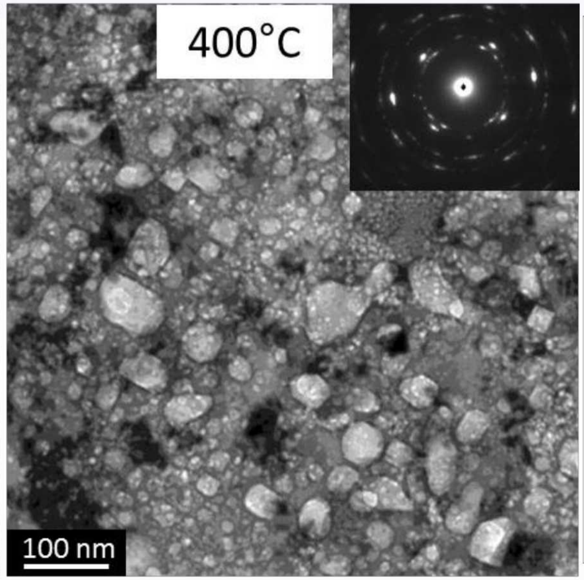
Visible damage to the sample

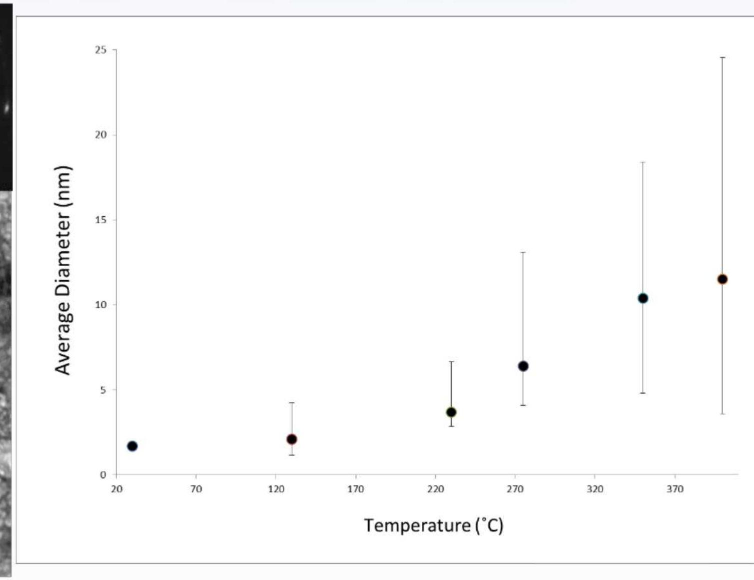
0.7 dpa Ni^{3+} irradiation

High concentration of cavities along grain boundaries

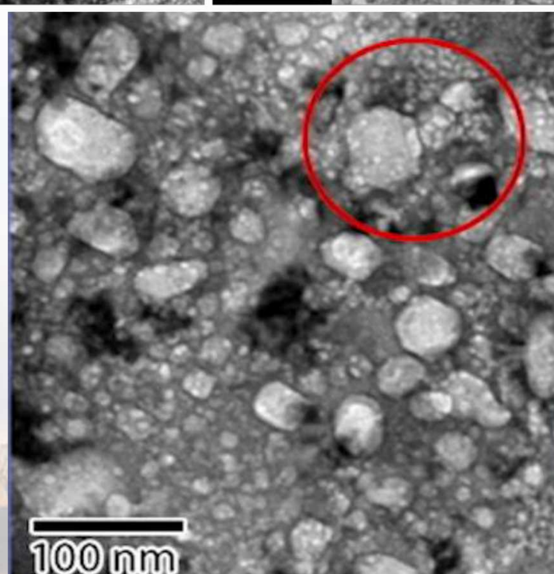
Cavity Growth during In-situ Annealing of 10 keV He^{+} Implanted and then 3 MeV Irradiated Ni^{3+}

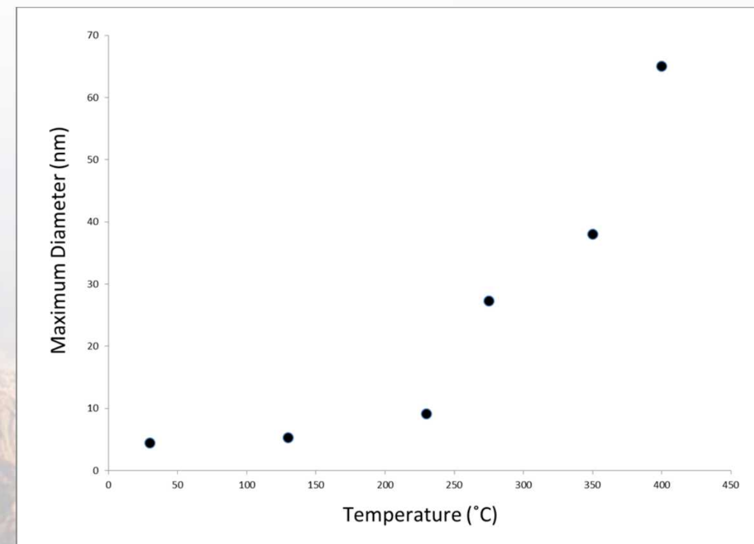






Bubble to cavity transition and cavity evolution can be directly studied

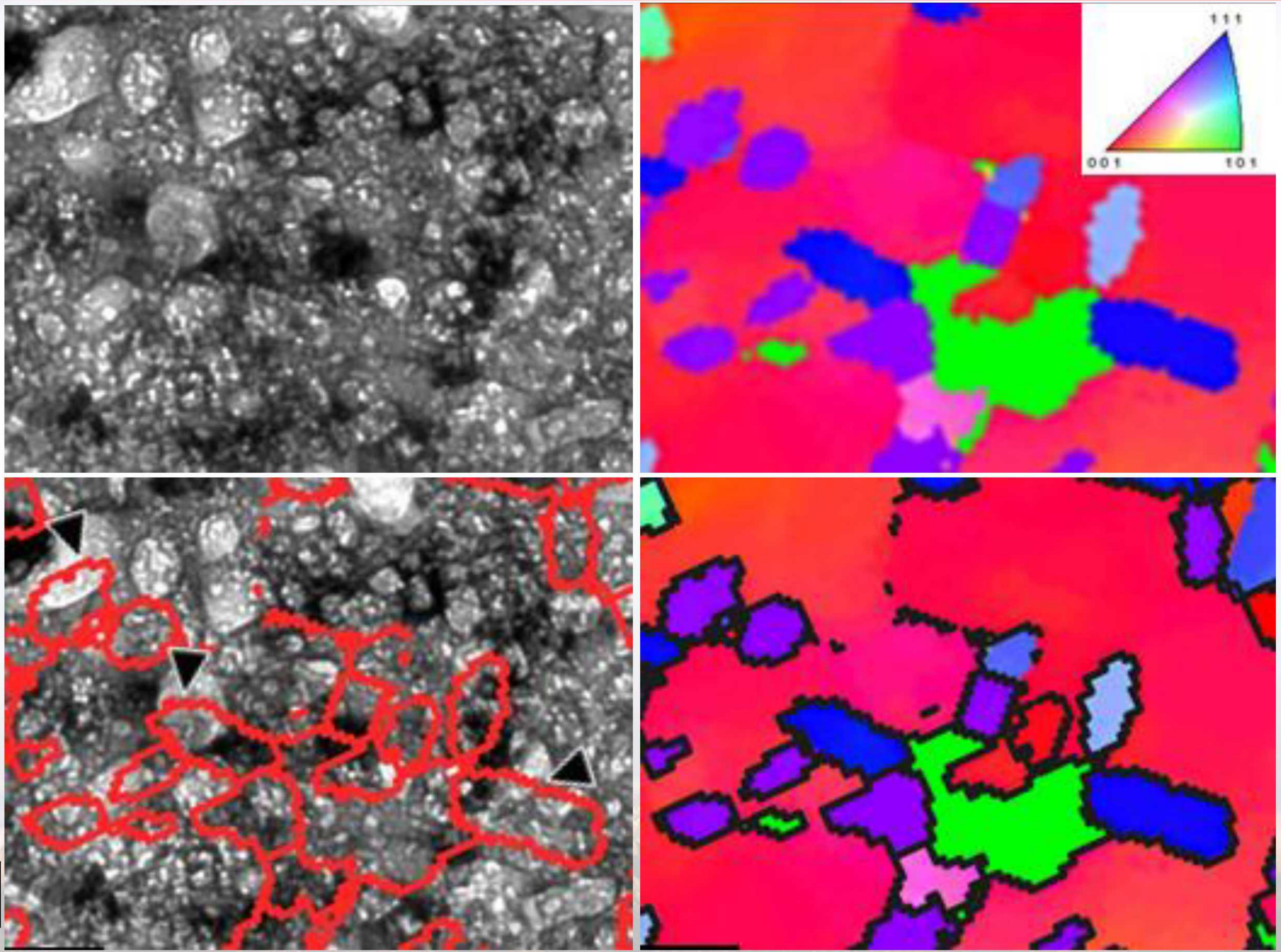




Precession Electron Diffraction Reveals Hidden Grain Structure

Cavities in helium implanted, self-ion irradiated,
nc nickel film annealed to 400 ˚C

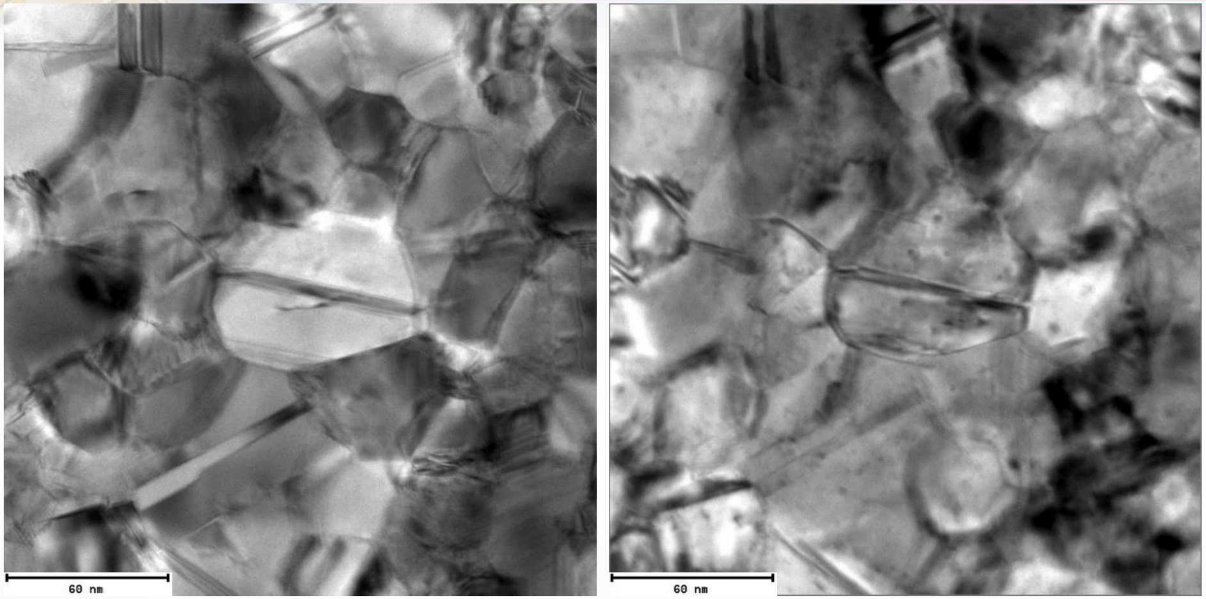
Cavities span multiple grains at identified grain boundaries



100 nm

Nanocrystalline vs. Nanoporous Au results

Collaborators: N. Briot and T.J. Balk

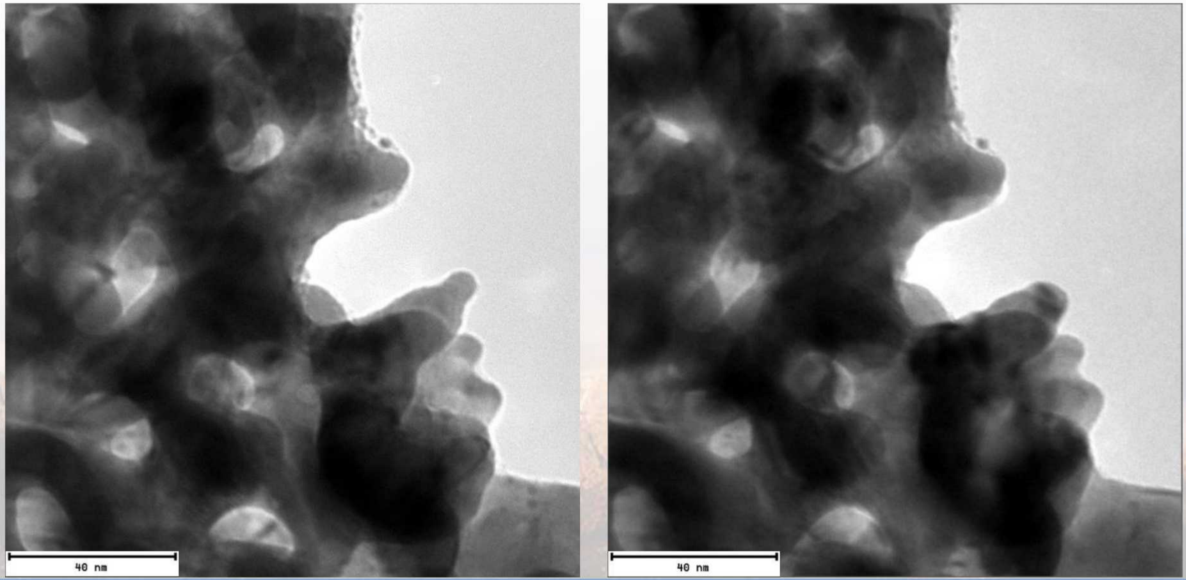


Nanocrystalline Au after ~ 0.5 dpa at 46 keV:

Lots of defects constantly created

Nanoporous Au after ~ 6.6 dpa at 46 keV:

Melting of the ligament

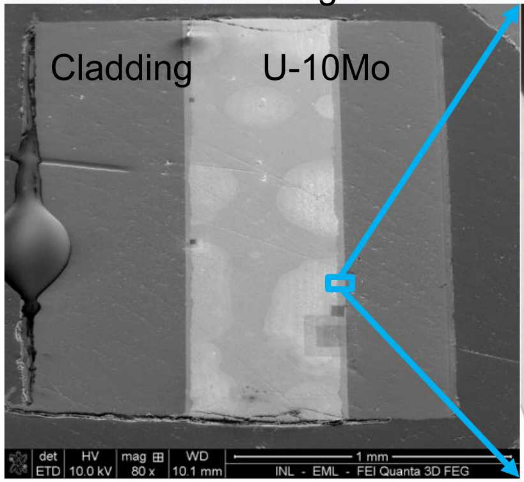


Free surfaces provide the ideal sink, but nanoporous materials undergo surface modification and many other effects under radiation damage

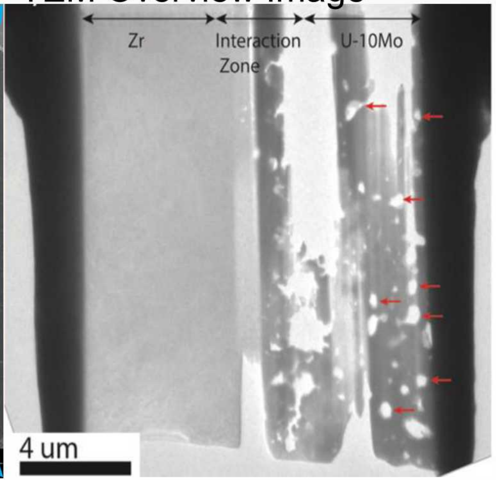
Post Irradiation Characterization of U-10Mo/Zr monolithic interface

Collaborators: C. Barrr, A. Aitkaliyeva

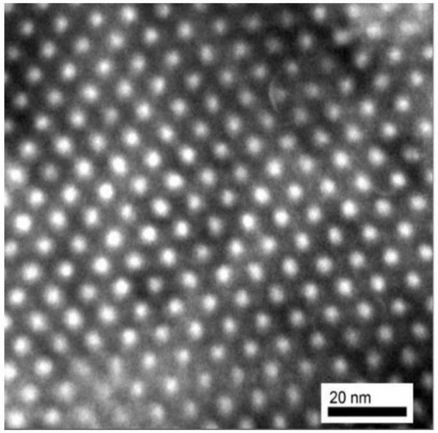
SEM Overview Image

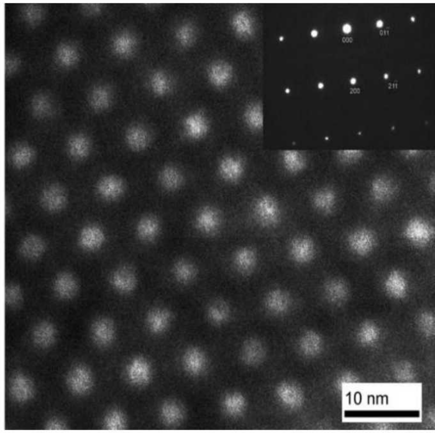


TEM Overview Image

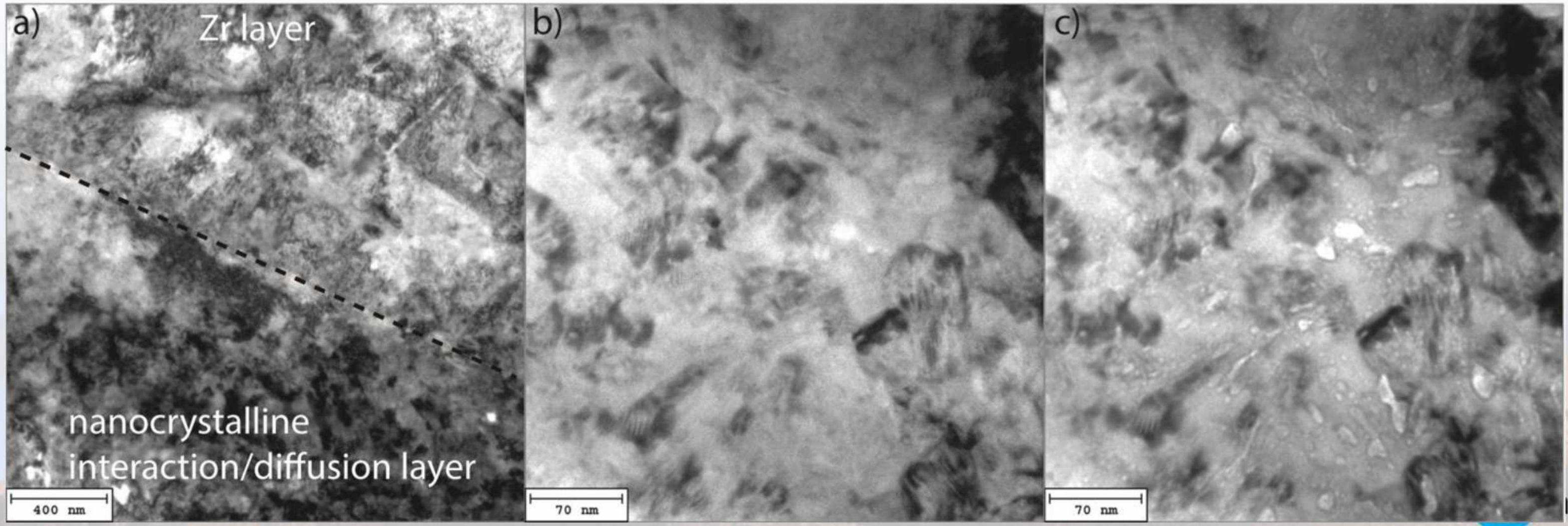


Gas superlattice bubbles ZA [110] in U-10Mo.





- 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

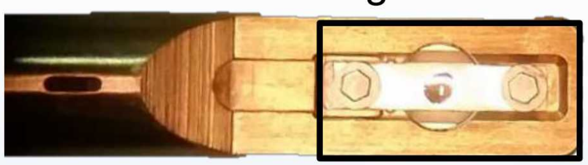
In situ Qualitative Mechanical Testing

Collaborators: C. Barr

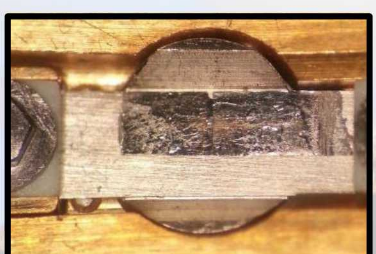
Gatan straining TEM Holder

- 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

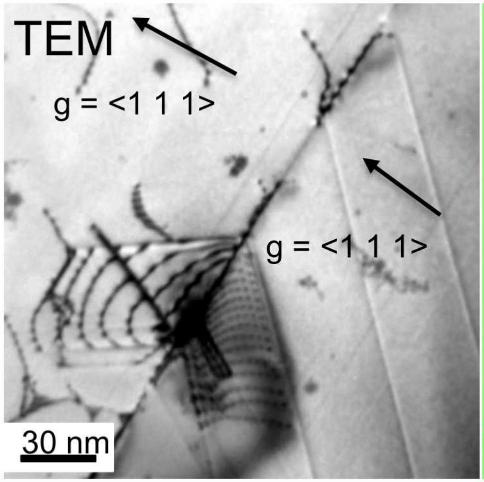


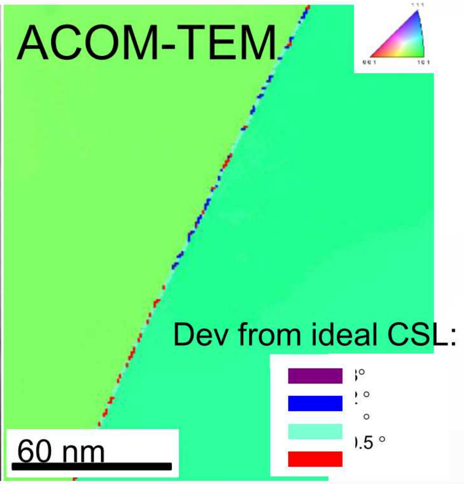
Thin film tension “jig”:



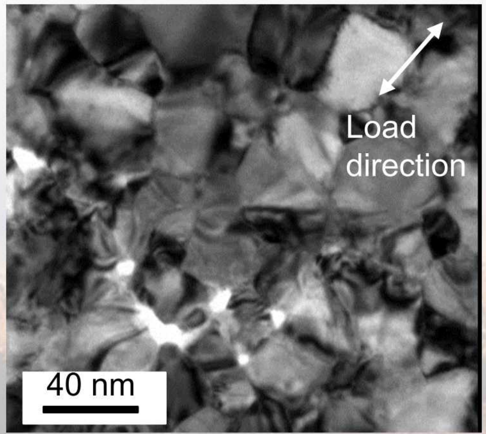
Jet thinned disk:

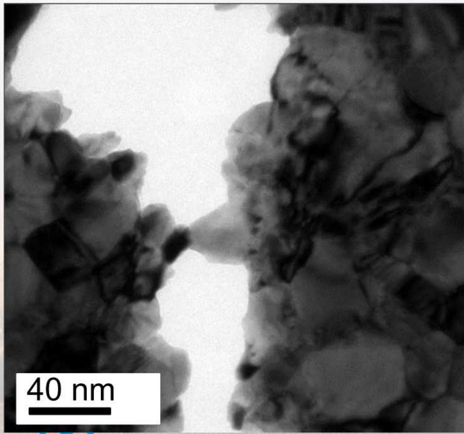
Dislocation interactions as a function of GB character (Σ3 twin GB below):





Observe deformation mechanisms in nanocrystalline metals during tensile straining:



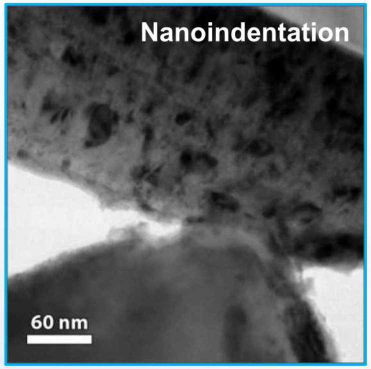


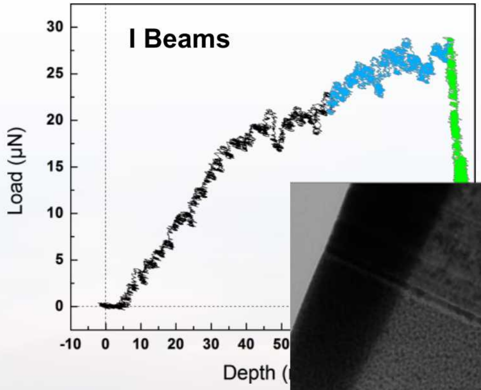
In situ Quantitative Mechanical Testing

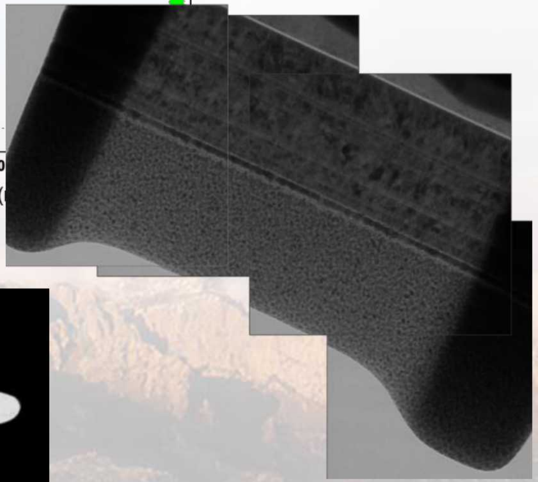
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

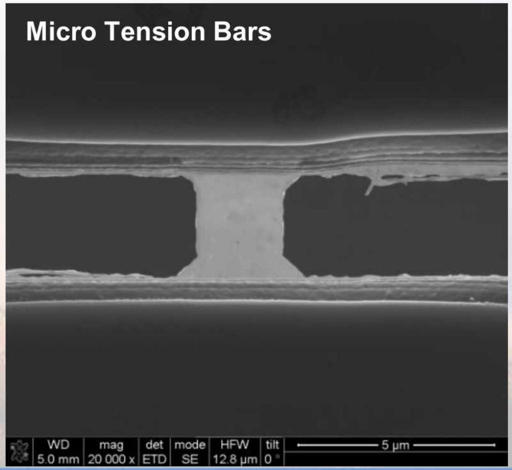
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

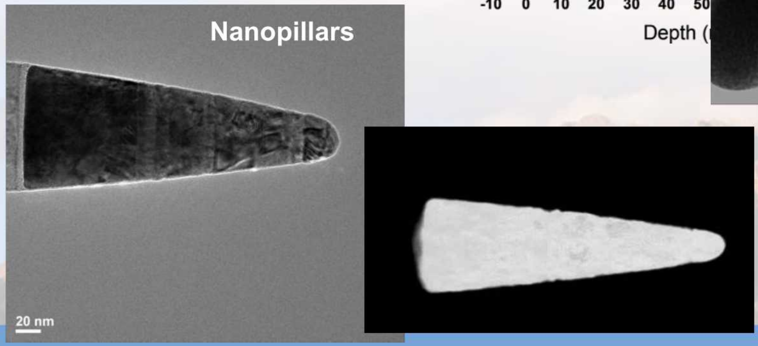
- Sub nanometer displacement resolution
- Quantitative force information with μN resolution
- **Concurrent real-time imaging by TEM**







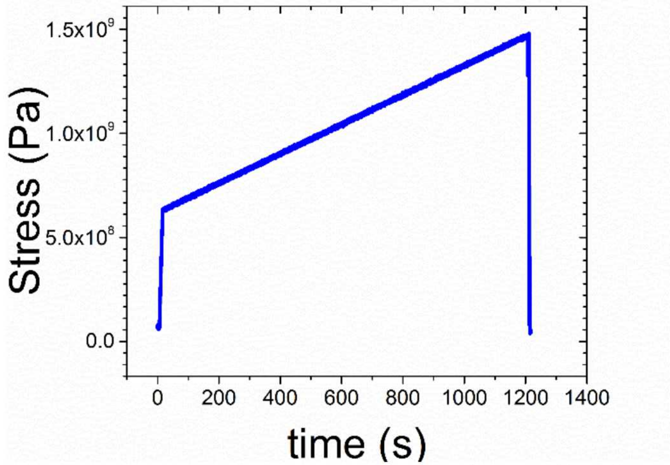




Irradiation Creep (4 MeV Cu^{3+} 10^{-2} DPA/s)

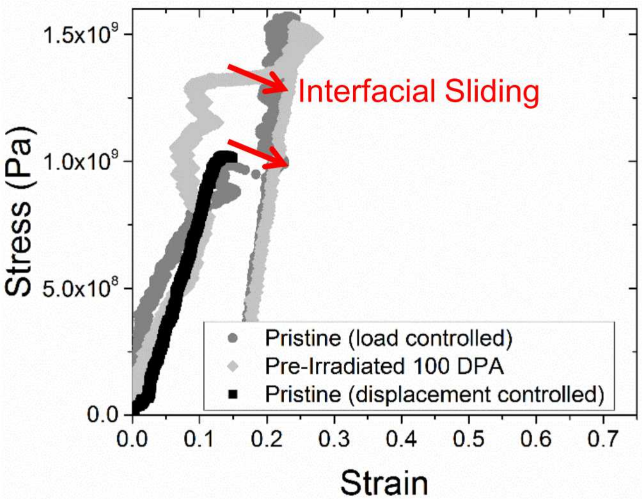
Contributors: S. Dillon & R.S. Averback

Controlled Loading Rate Experiments

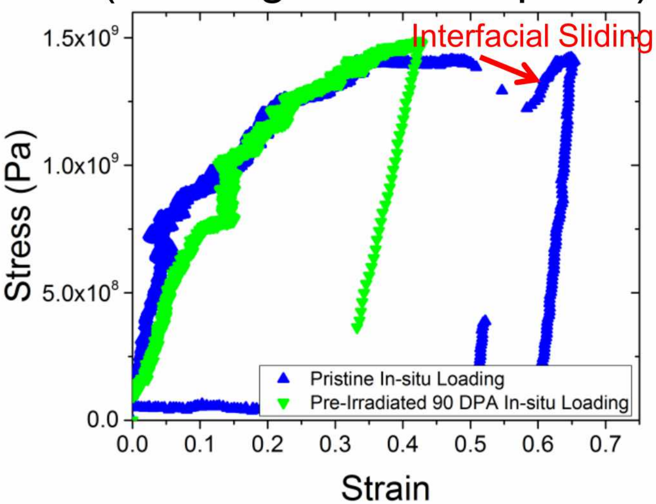


In-situ TEM radiation creep is feasible!

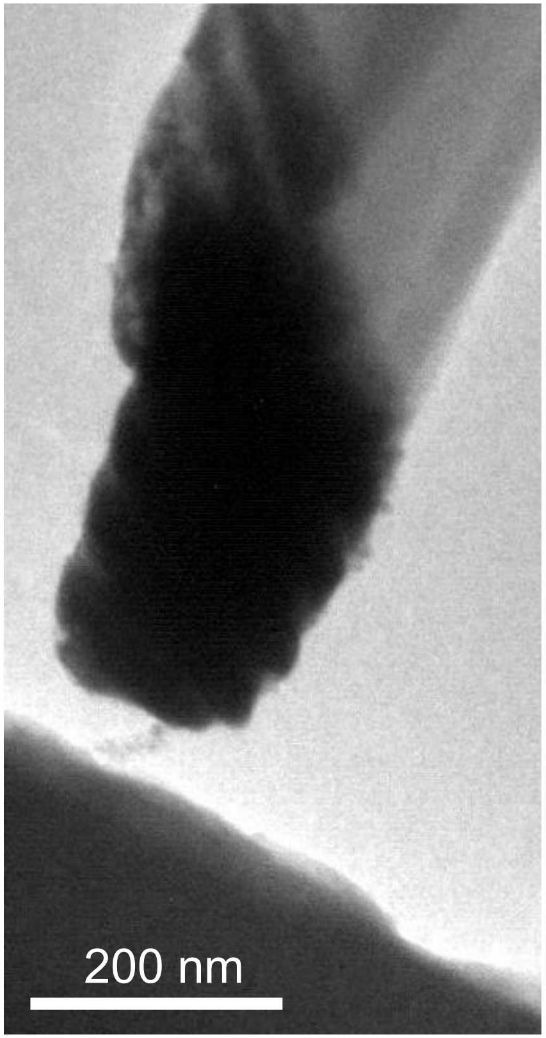
No Irradiation
(Loading rate 0.6 Mpa s^{-1})



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



50 nm Cu-W multilayer
20 Min



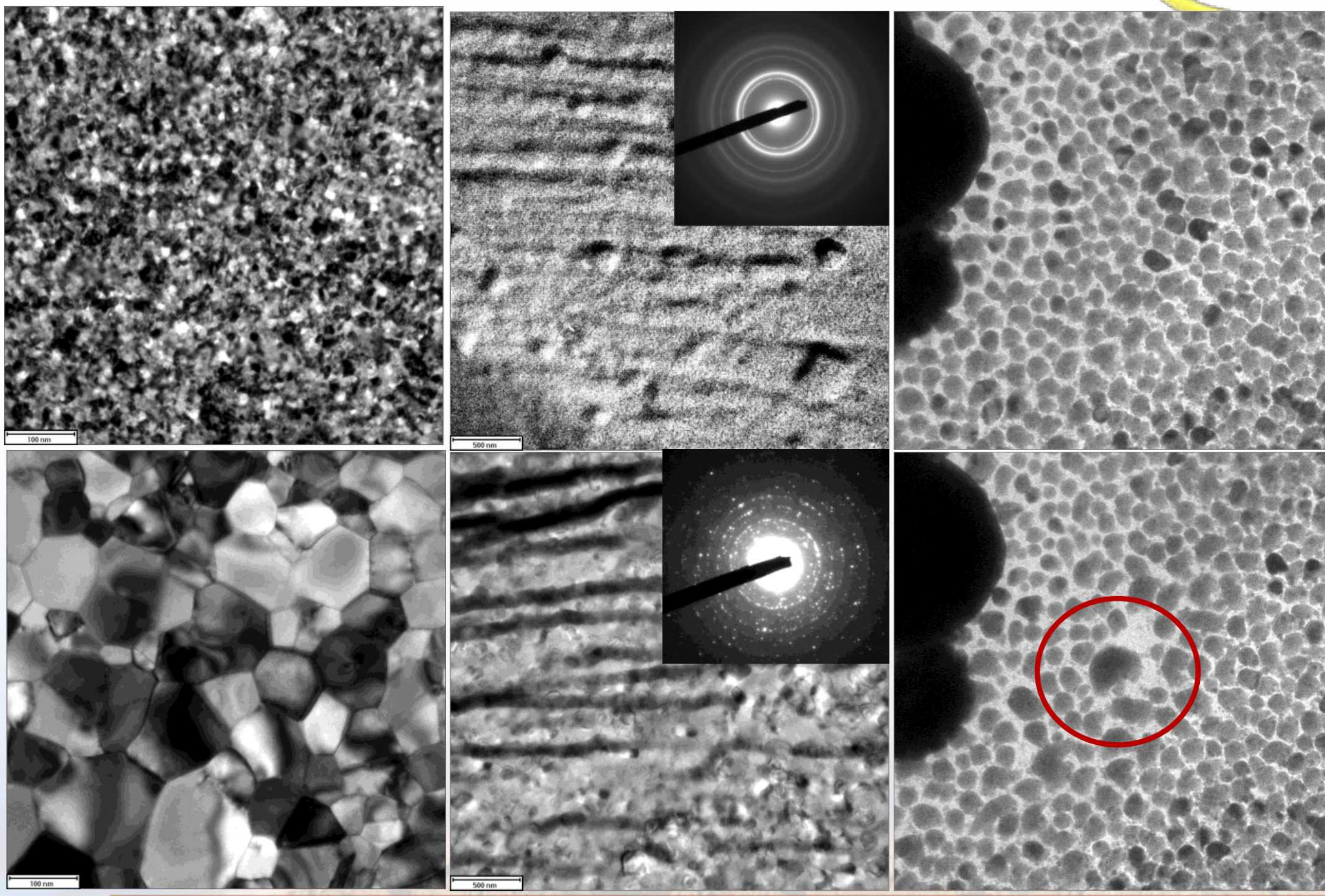
Initial Laser Heating Observations

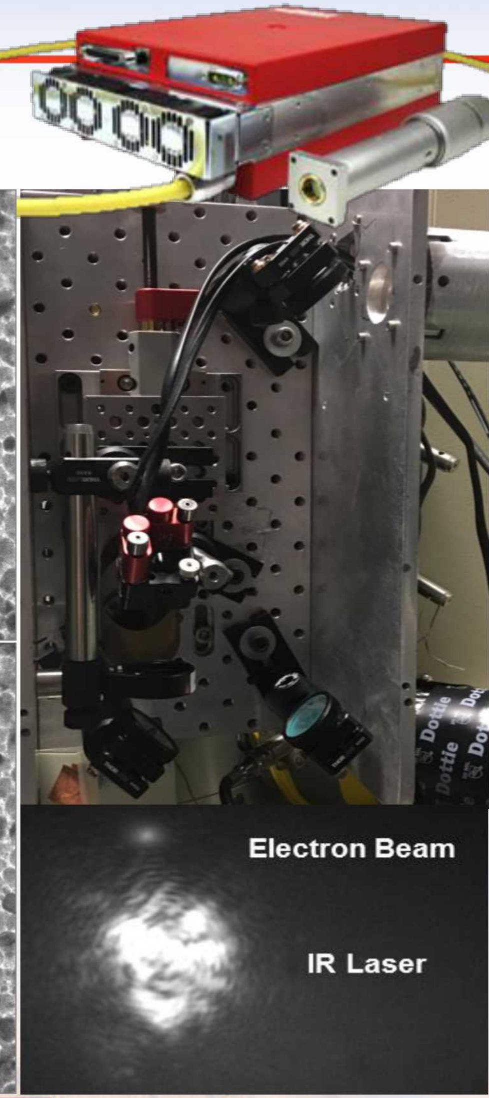
Collaborator: P. Price, C.M. Barr, D. Adams, M. Abere

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

Before

After

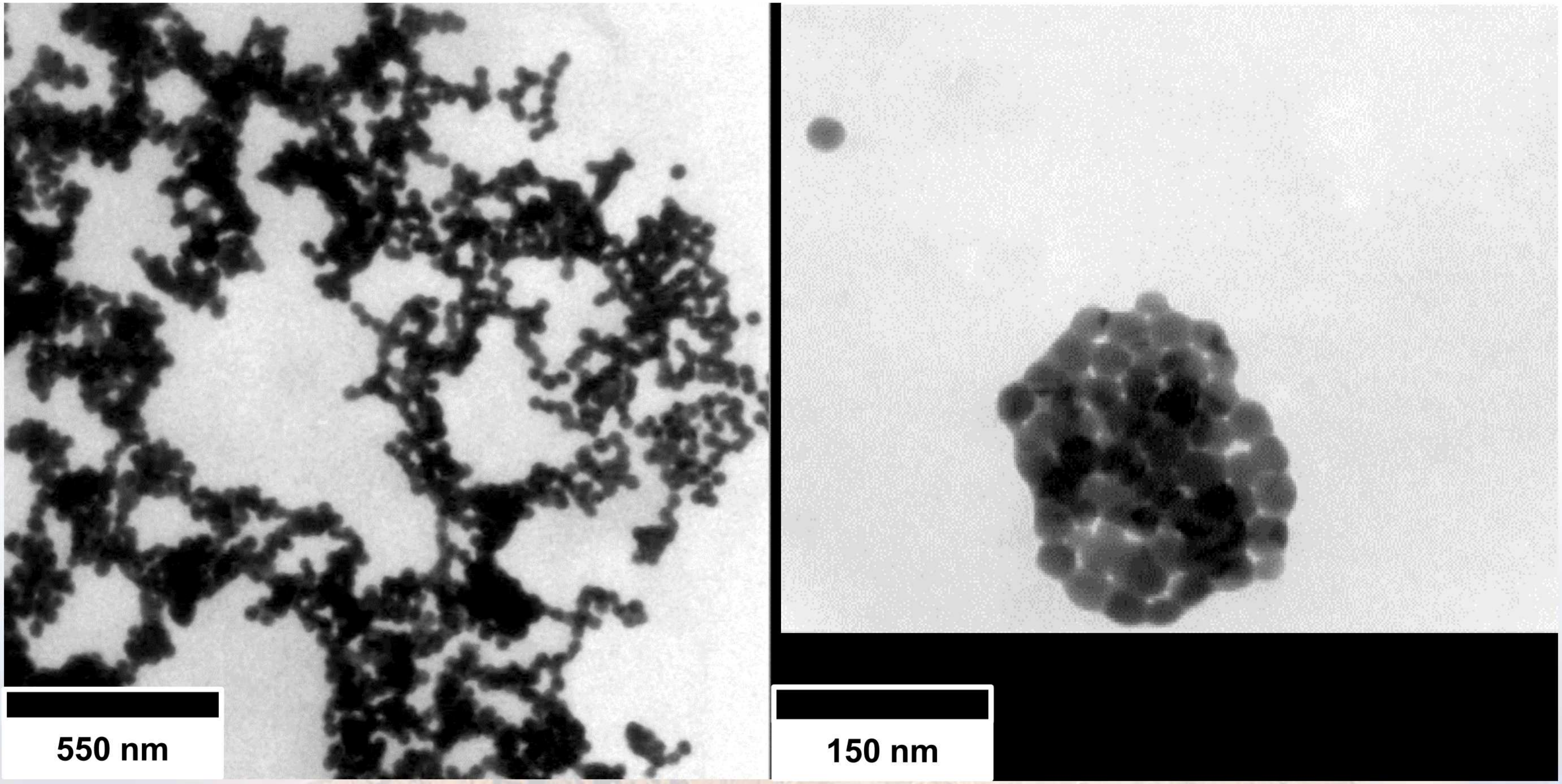




We can now introduce rapid thermal heating with any TEM stage or ion beam conditions

Complex Interaction Au NPs Exposed to Laser Irradiation

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



Speed = 2.5x

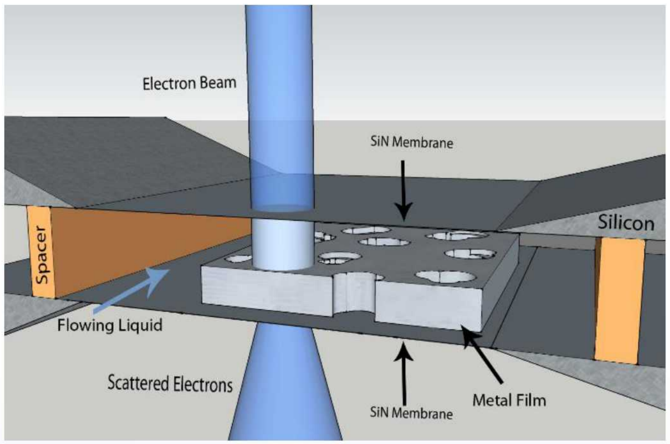
A Complex Combination of Sintering, Reactions, and Ablation Occurs

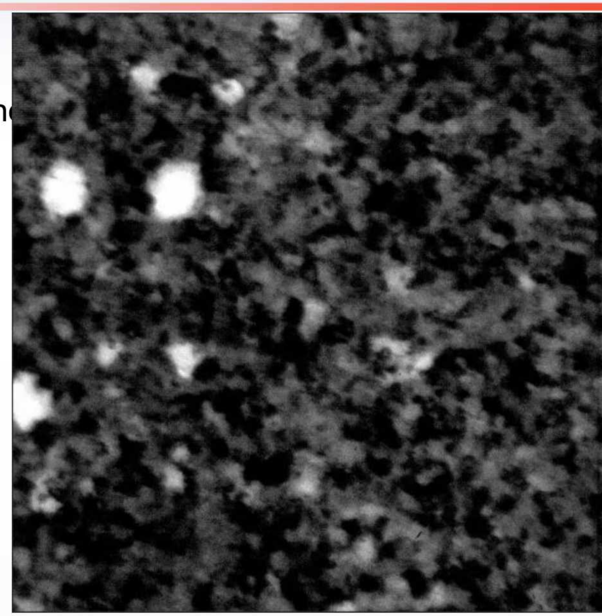
Can We Gain Insight into the Corrosion Process through *In situ* TEM?

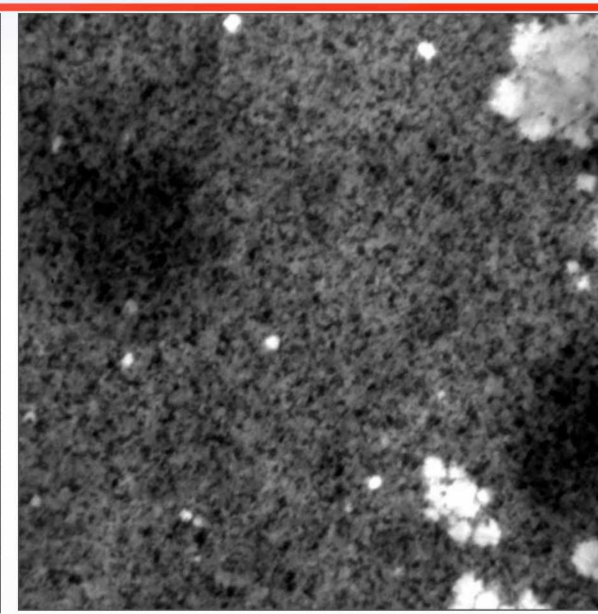
Contributors: D. Gross, J. Kacher, I.M. Robertson & Protochips, Inc.

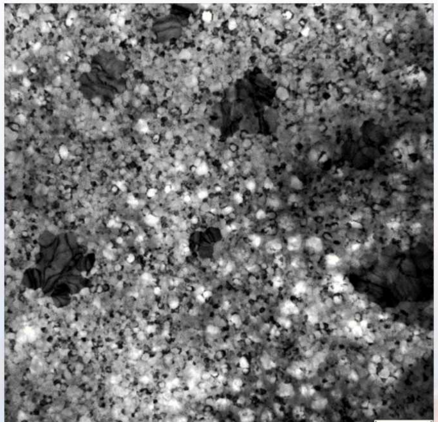
Microfluidic Stage

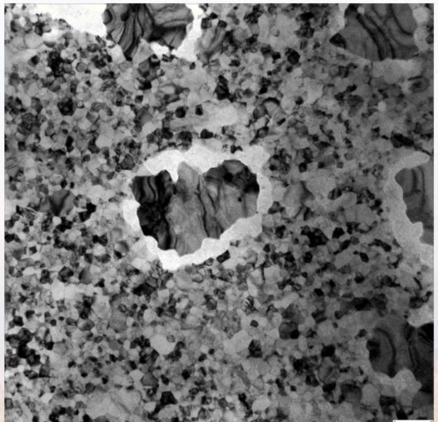
- Mixing of two or more channels
- Continuous observation of the reaction chann

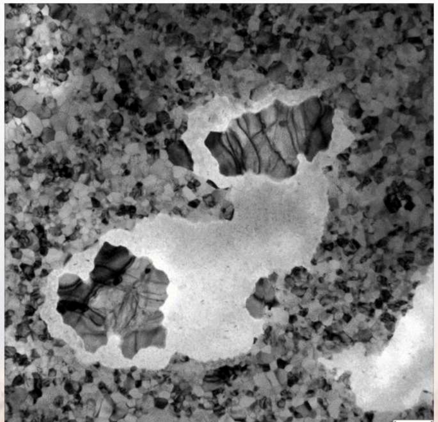


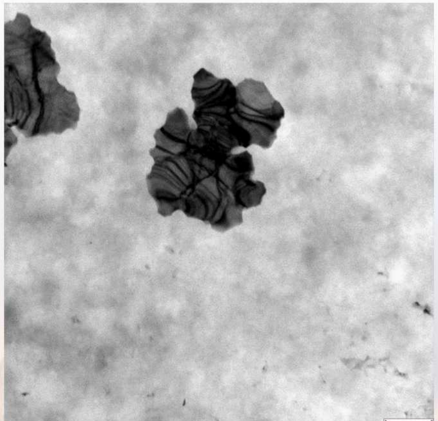












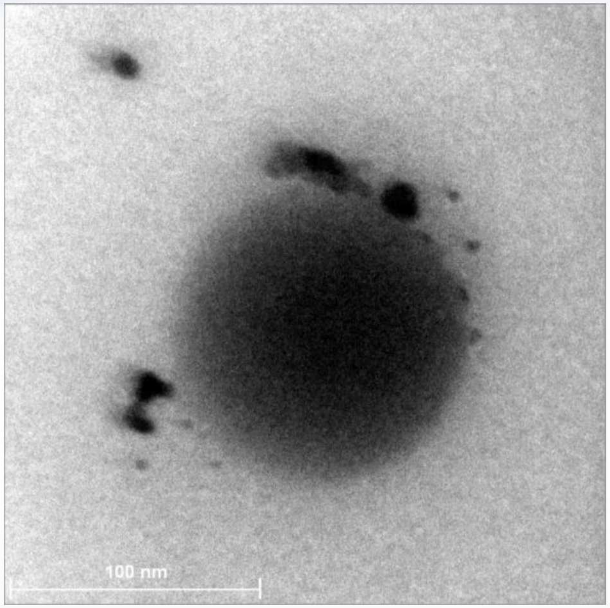
Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.
Large grains resulting from annealing appear more corrosion tolerant

Other Fun Uses of Microfluidic Cell

Protocell Drug Delivery

S. Hoppe,
E. Carnes,
J. Brinker

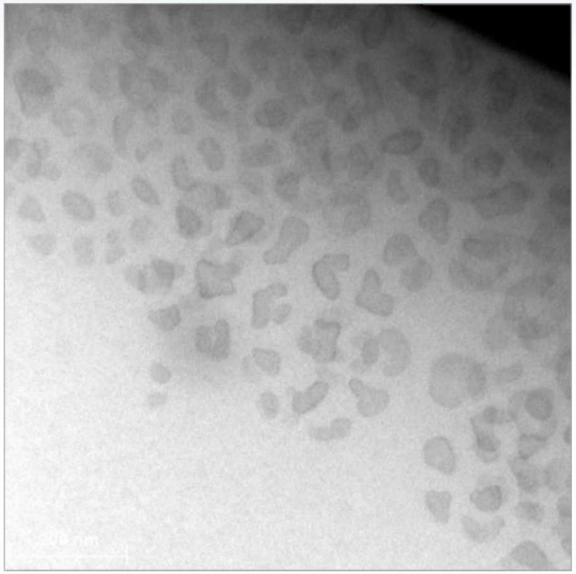
Liposome encapsulated Silica destroyed by the electron beam



BSA Crystallization

S. Hoppe

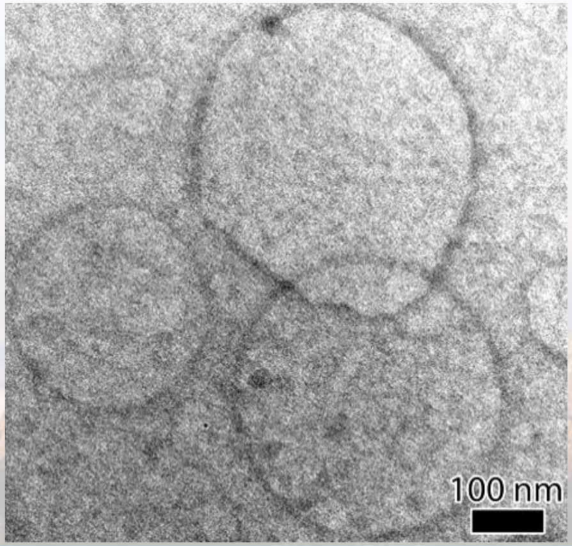
Crystallization of excess Bovine Serum Albumen during flow



Liposomes in Water

S. Hoppe,
D. Sasaki

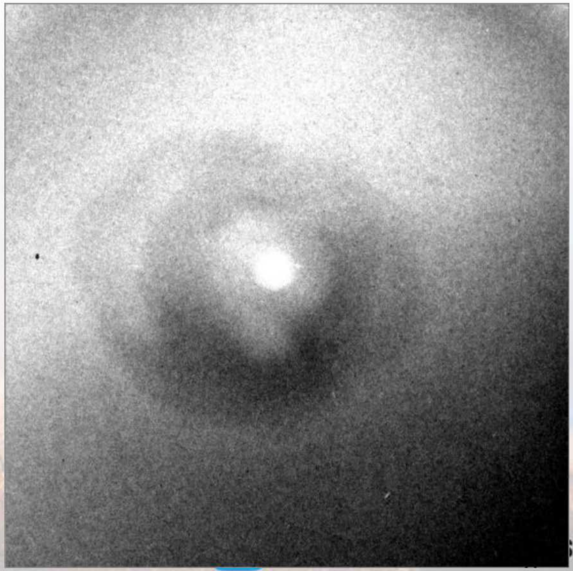
Liposomes imaged in flowing aqueous channel



La Structure Formation

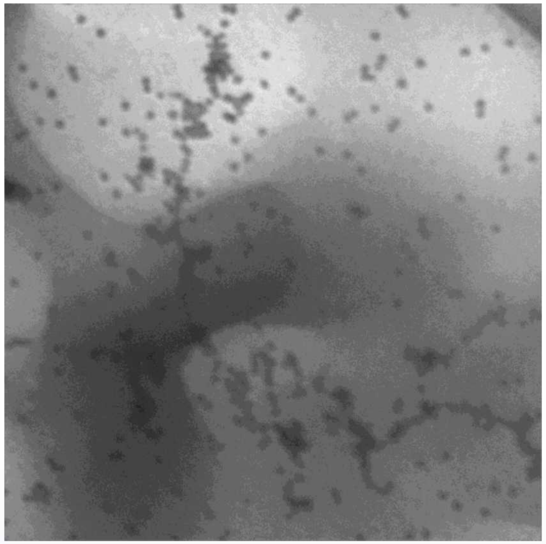
S. Hoppe,
T. Nenoff

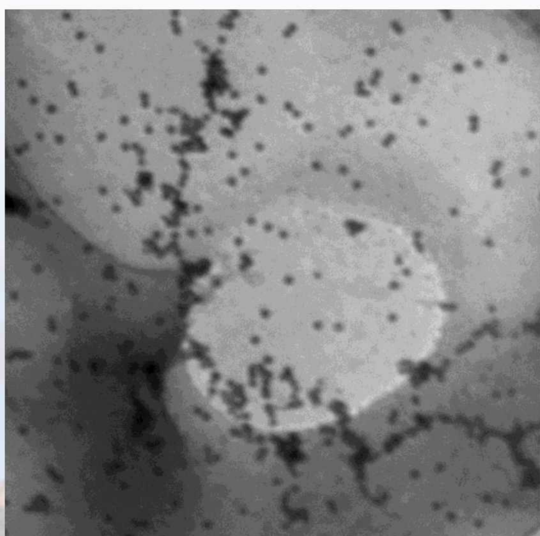
La Nanostructure form from $LaCl_3$ H_2O in wet cell due to beam effects



In Situ TEM observation of three-phase mixture

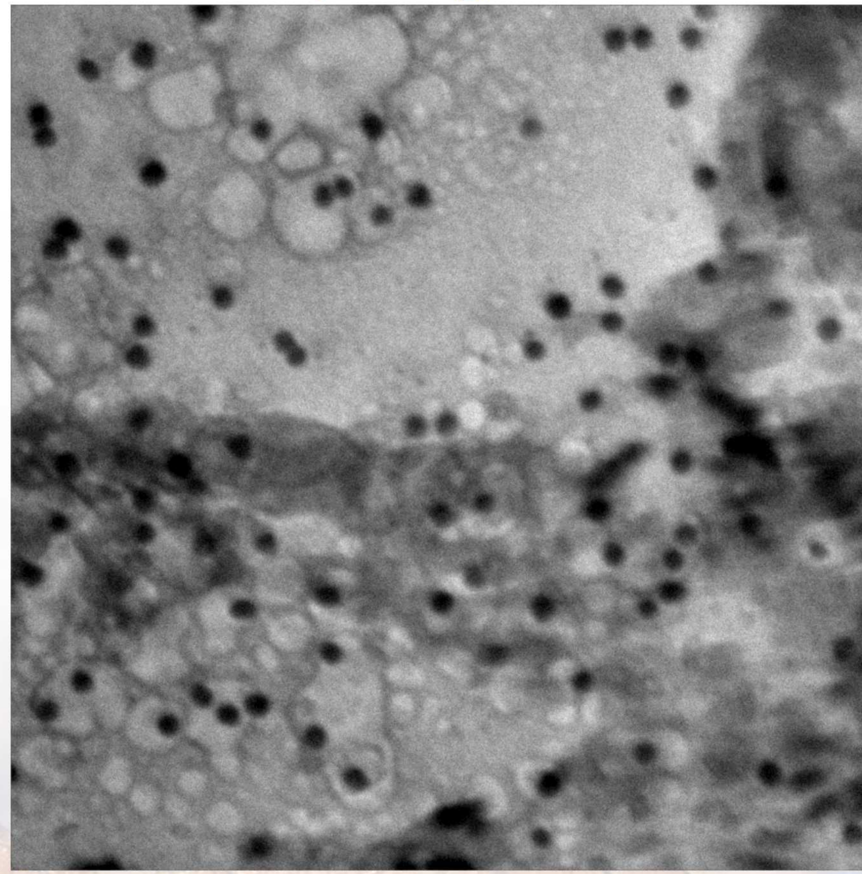
Collaborator: Sarah Hoppe





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

Au nanoparticles, aqueous solution, and gas bubbles

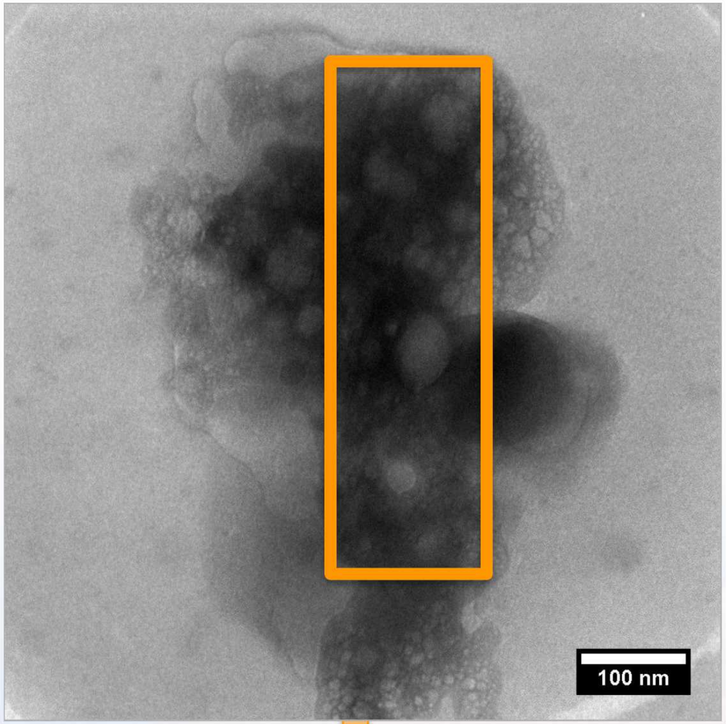


PED in Liquid Cell Environment

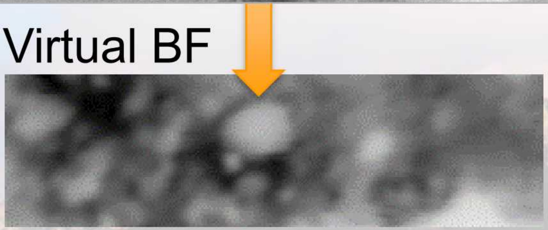
Contributors: C. Taylor, S. Pratt, & T. Nenoff

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

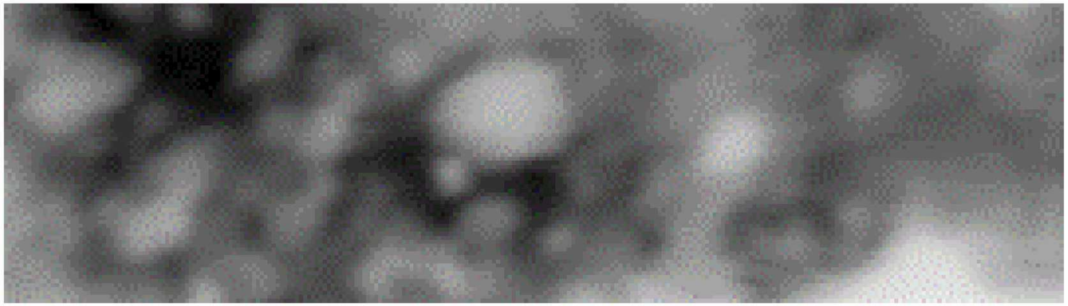
TEM Image



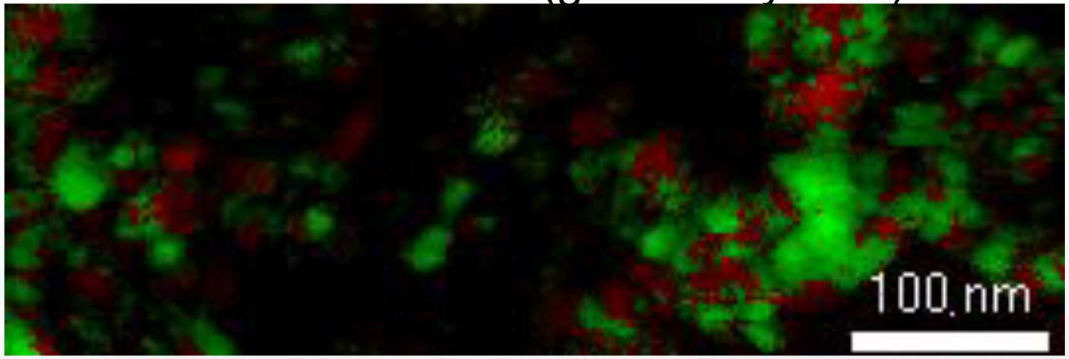
Virtual BF



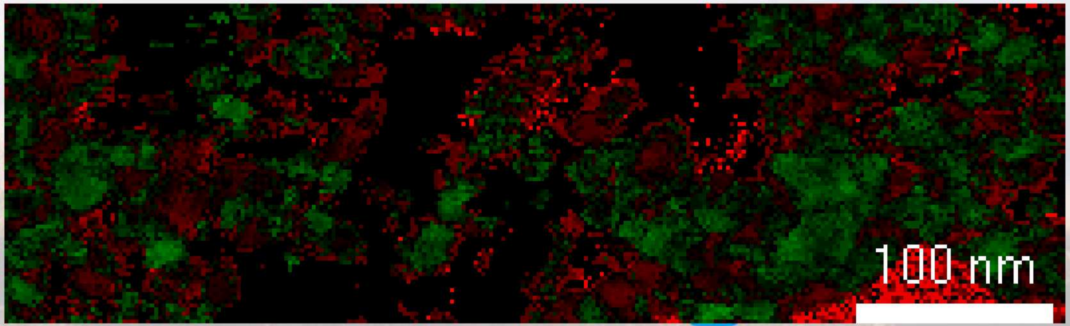
Virtual BF



Index + Phase (green = hydride)

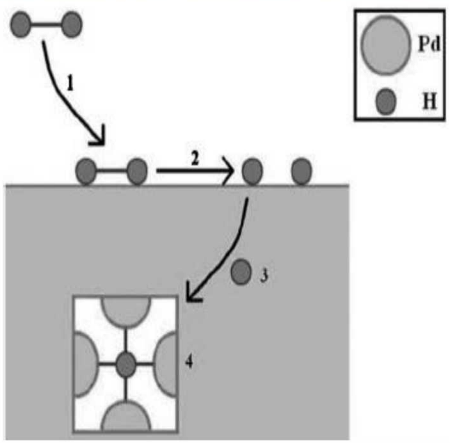


Phase Reliability + Phase

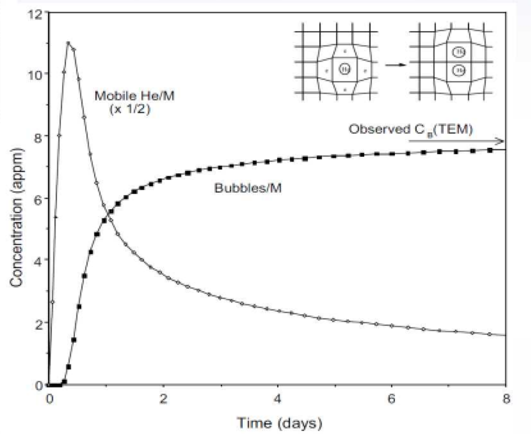


In situ TEM Hydrogen Exposure

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

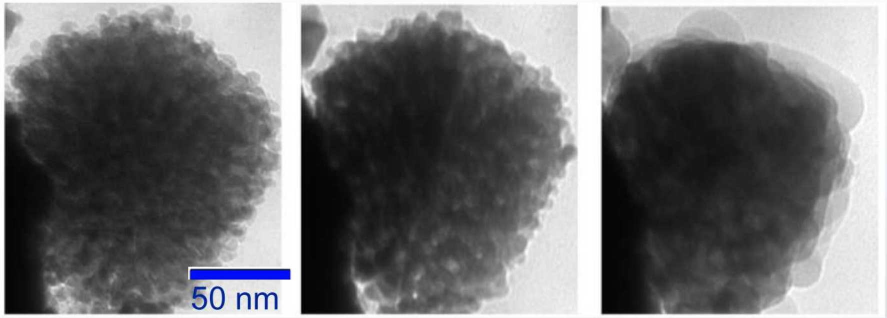


Cowgill, D., Fusion Sci. & Tech., 28 (2005) p. 539
Trinkaus, H. *et al*., JNM (2003) p. 229
Thiebaut, S. *et al*. JNM (2000) p. 217

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

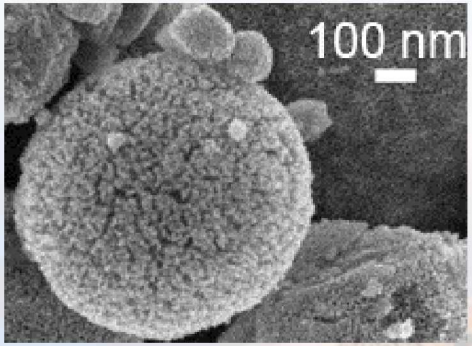
- 1 atm H_2 after several pulses to specified temp.

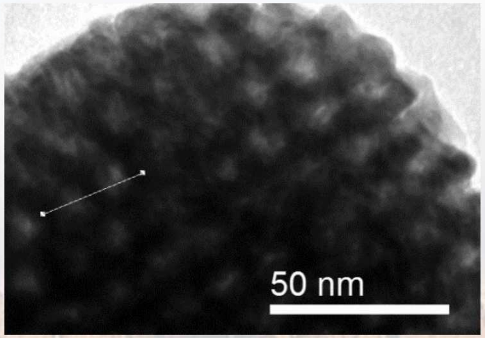


125°   200°   300°

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

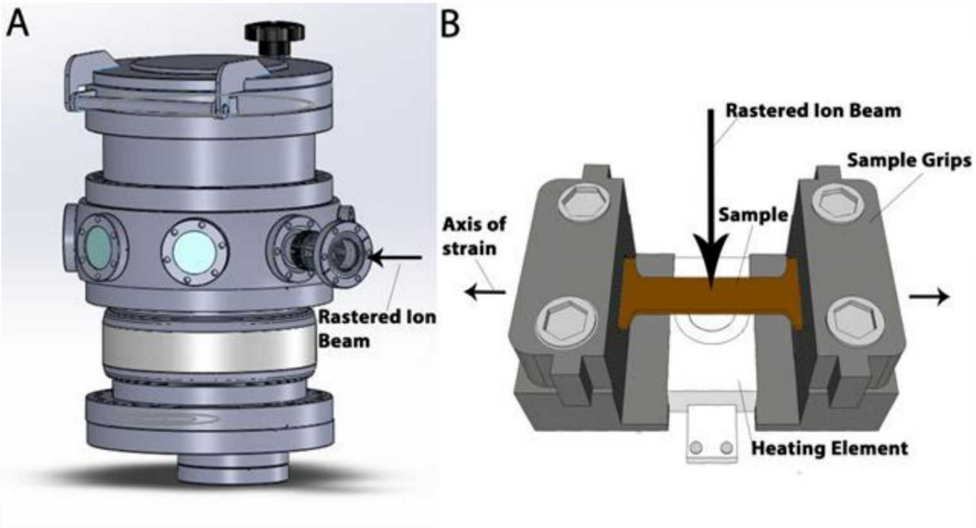
Harmful effects may be mitigated in nanoporous Pd

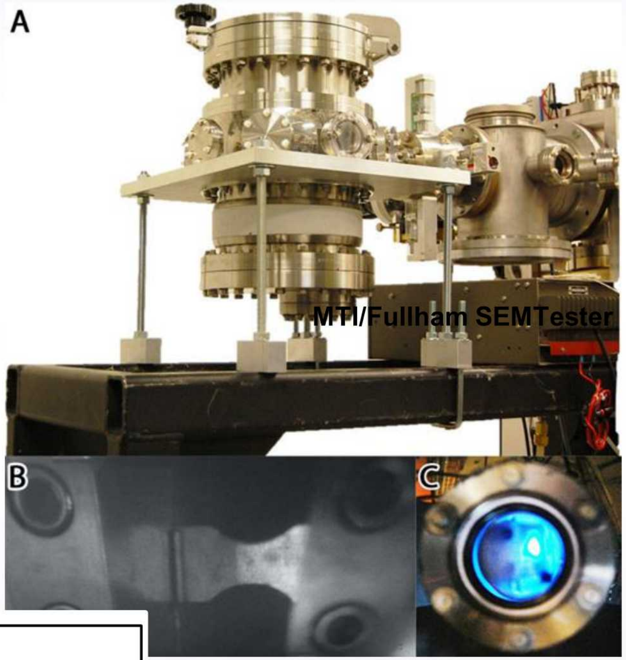


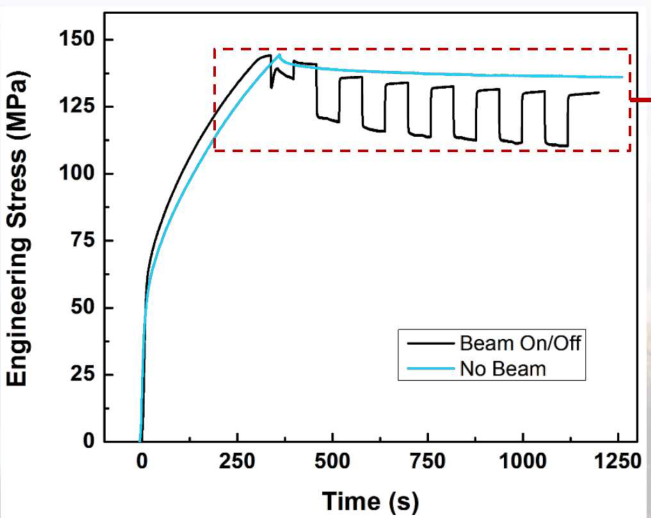


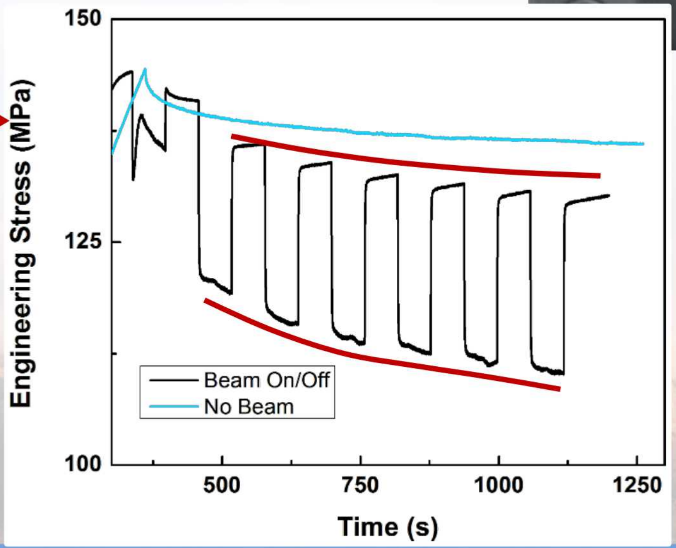
Mechanical Testing End Station

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

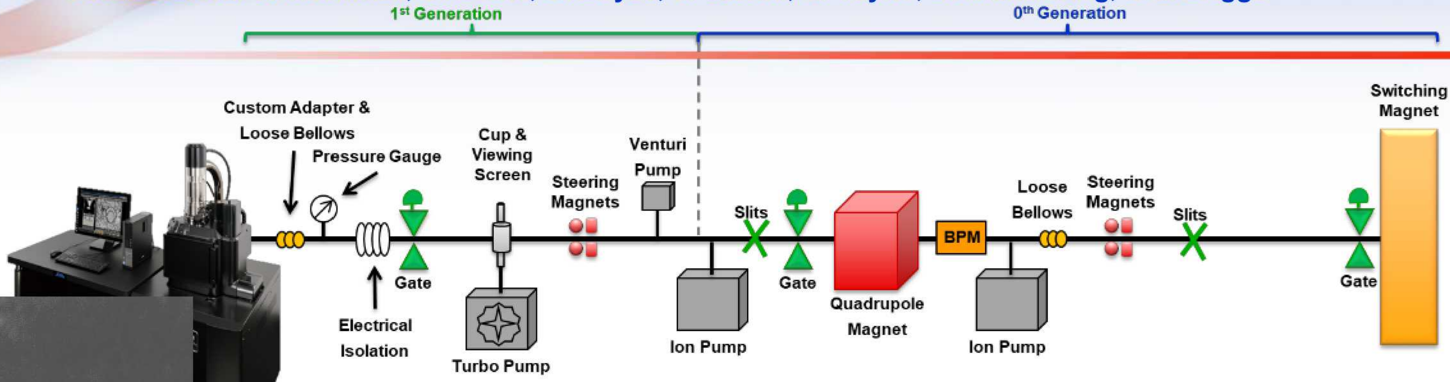
Beam cycled on/off at 60 s intervals

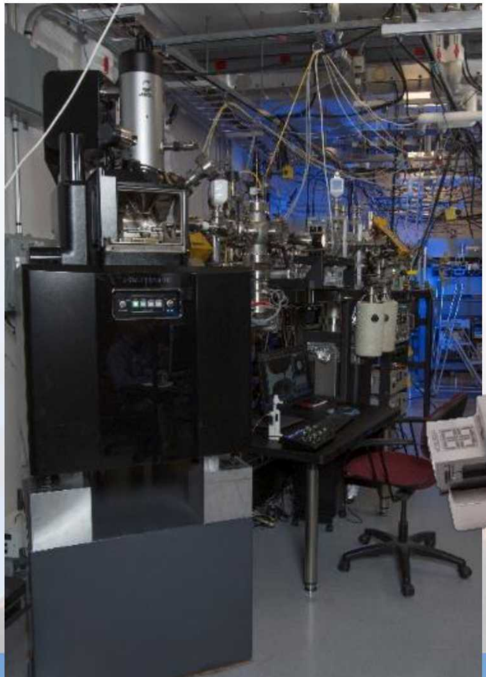
Beam conditions: 4.5 MeV H^+: 2.1×10^{11} ions $cm^{-2}s^{-1}$

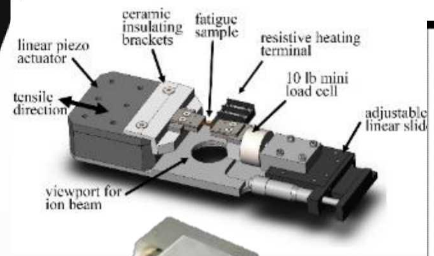
Sandia National Laboratories

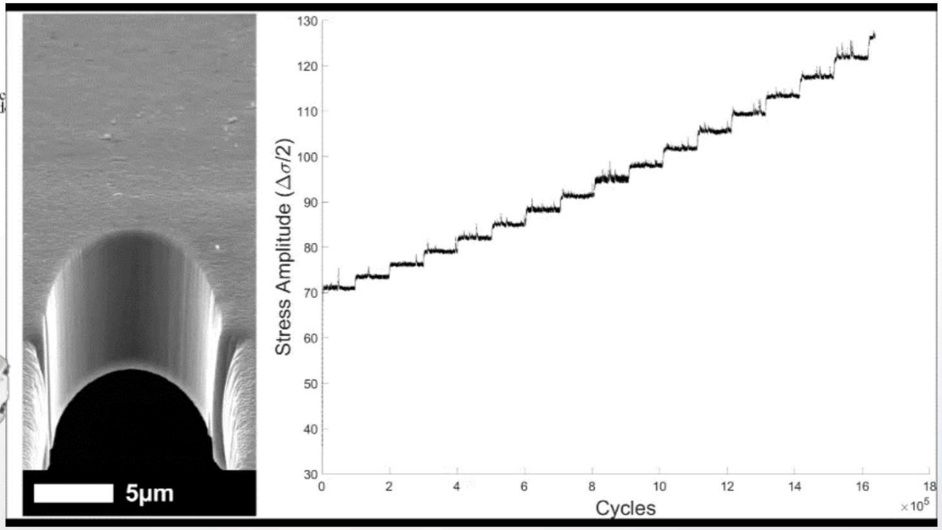
In situ Ion Irradiation SEM (I³SEM)

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









S.A. Briggs

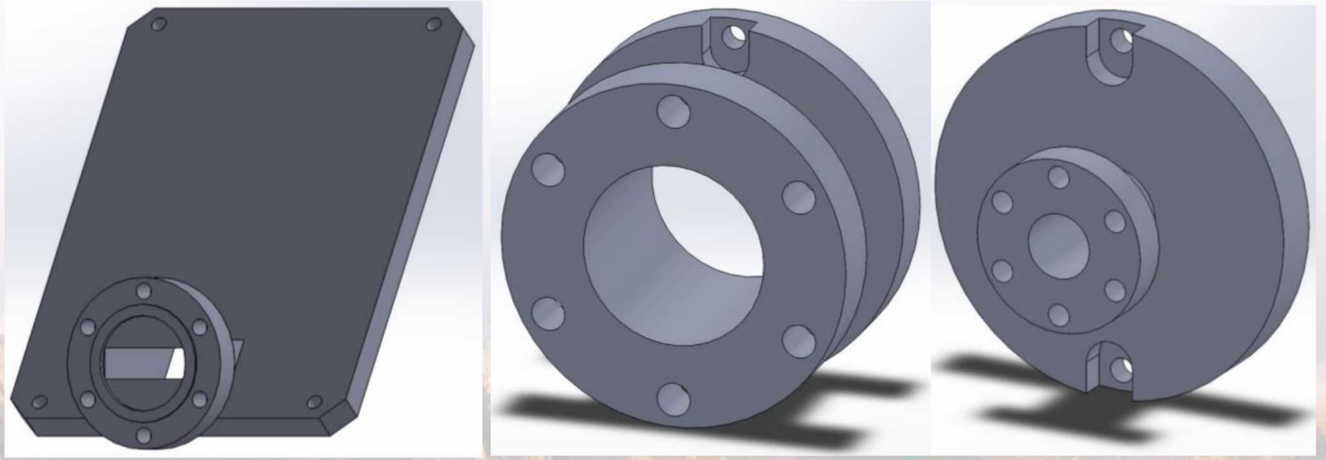
Coupling a 6 MV Tandem and Ion Gun to a SEM Tuesday

August 14, 4:45 PM in Room Ft. Worth 6-7

Customization of the *In situ* SEM Beamline

Collaborators: D.L. Buller & S. Briggs

- Couple beamline with 2.75” CF connection on low end of WDS port
- Custom rectangular piping allows for broad x-direction rastering
- Additional JEOL-to-CF adapters made for instrumentation and cable feed throughs

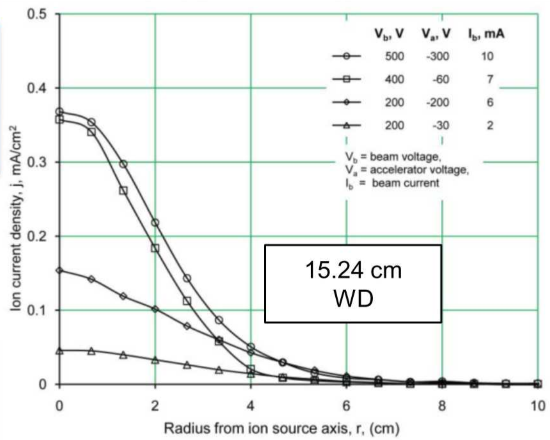


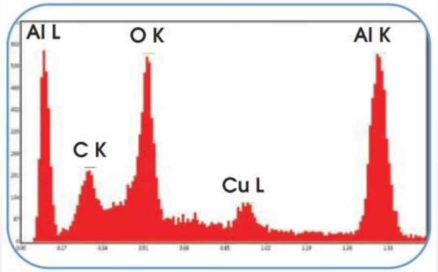
Future Dierection of the *In situ* SEM

Collaborators: D.L. Buller & S. Briggs

- Mate with Kaufman & Robinson KDC10 Plasma Source
  - 10 mA gas-fed source, energies from 100 eV to 1.2 keV
  - Capable of running inert gases, some reactive gases (H, O_2), and mixtures

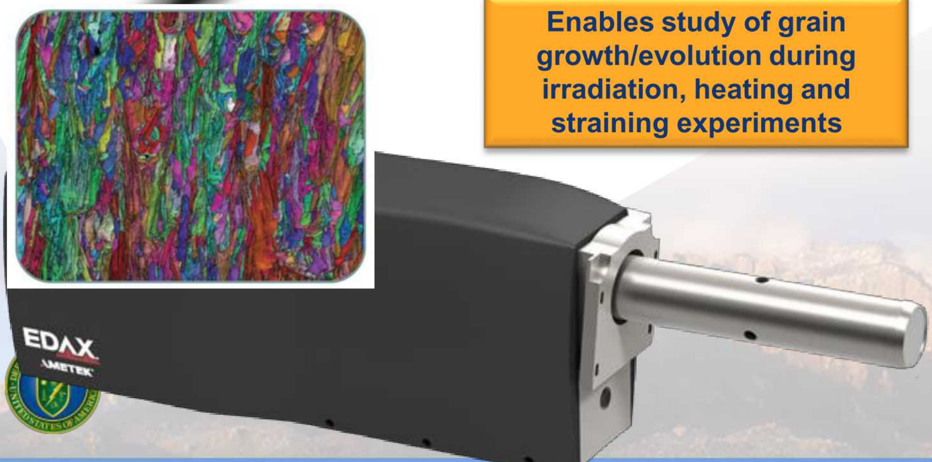
Adds low-E He implantation and dual beam capabilities to SEM





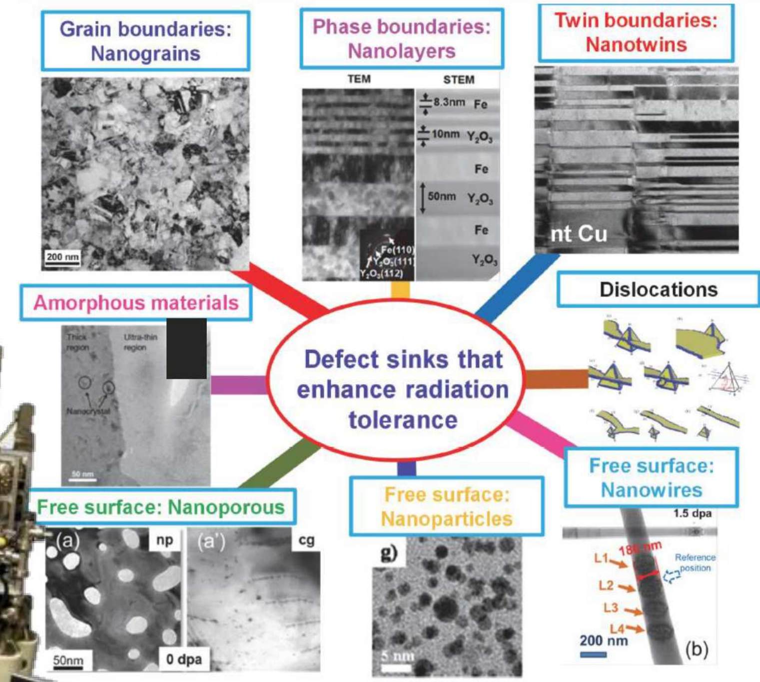
Allows for analysis of precipitates, solute segregation, and phase ID

Enables study of grain growth/evolution during irradiation, heating and straining experiments

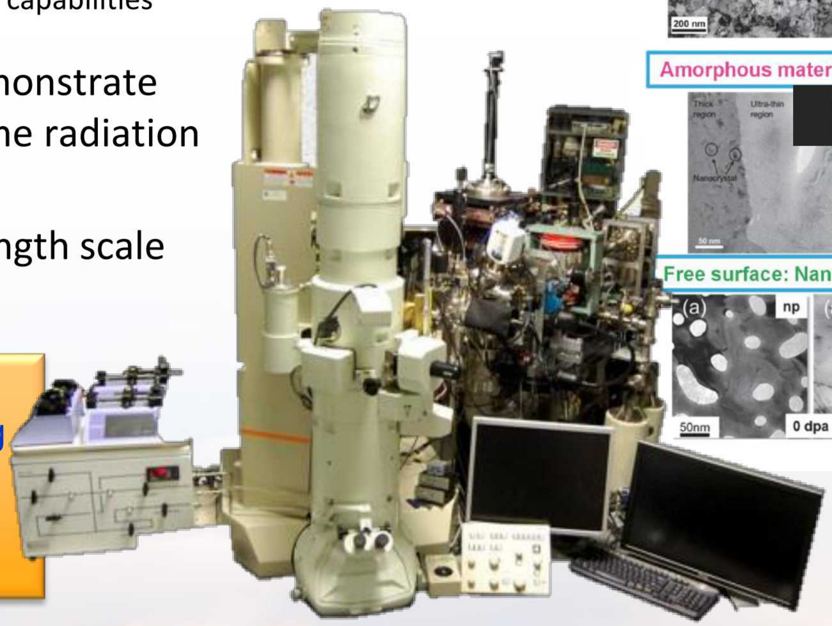


Summary

- Sandia's I^3TEM 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 I^3TEM 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.

NSUF Nuclear Science User Facilities

U.S. DEPARTMENT OF ENERGY Office of Science

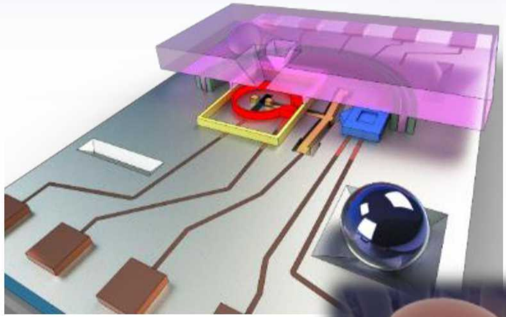
Sandia National Laboratories

Sandia's USER Capabilities

D. Hanson, W. Martin, M. Wasiolek

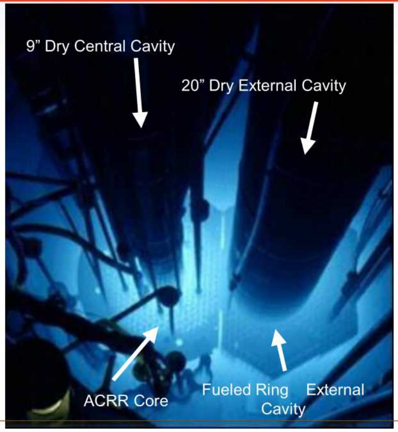
www.cint.lanl.gov

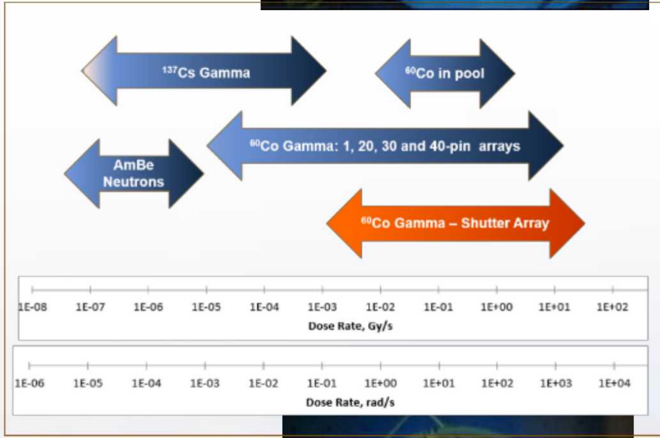
- Spring and Fall proposals for 18 months
- Rapid Access proposal anytime for 3 months



www.nsuf.inl.gov

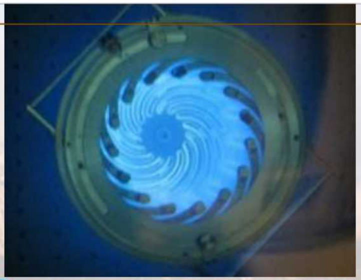
- Three proposal a year for 9 months





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

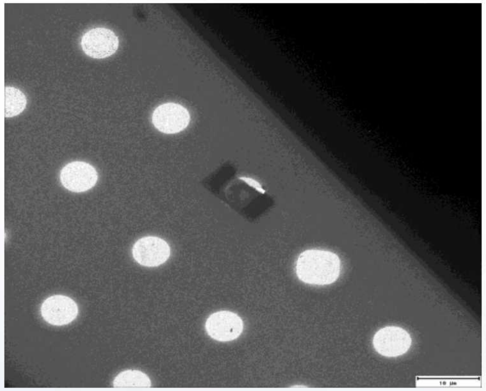
Back-up Slides

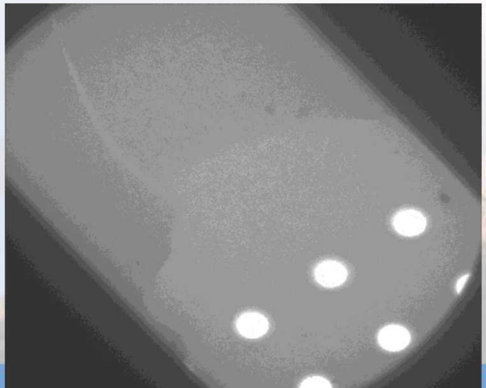
Feasibility of Studying Zircaloy 2 at Nominally 1 atm

Collaborators: S. Rajasekhara and B.G. Clark

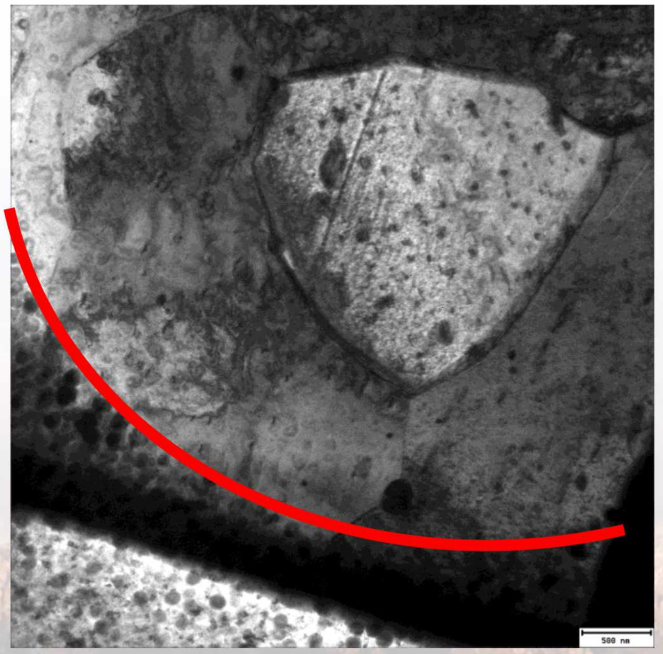
Vapor-Phase Heating TEM Stage

- 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

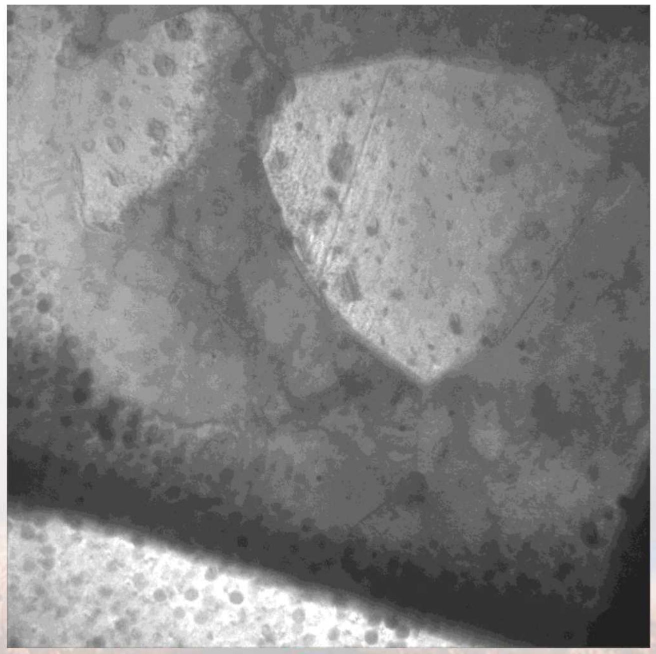




Vacuum & Single Window



Nominally 1 atm H_2 & Two Windows

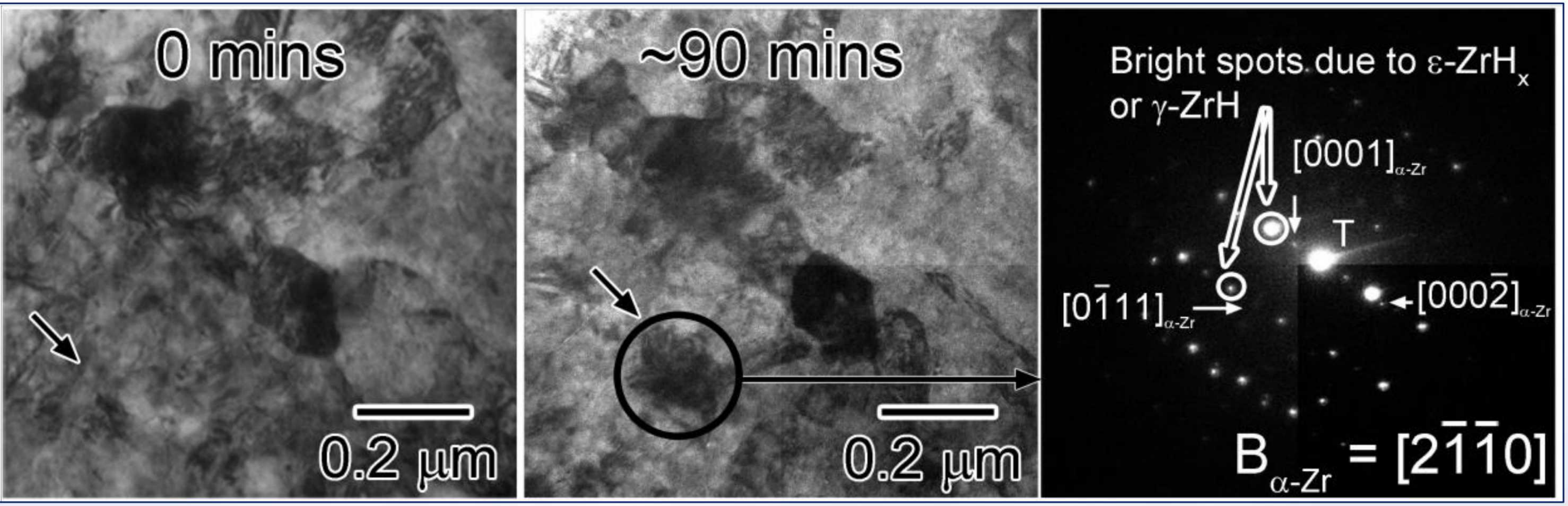


Most features are observed in both despite the decreased resolution resulting from the additional SiN window and 5 µm of air

In situ Observation of Hydride Formation in Zirlo

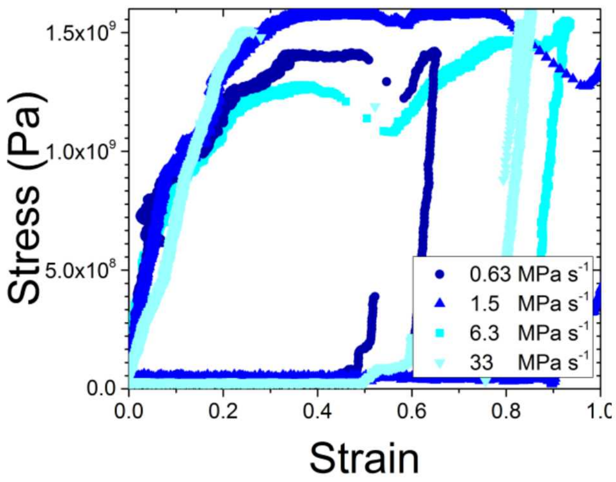
Collaborators: S. Rajasekhara and B.G. Clark

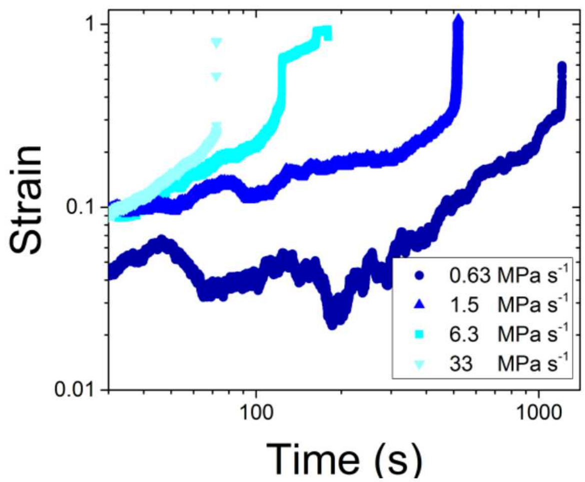
Absolute hydrogen pressure: 327 torr (~ 0.5 atm),
Ramp rate: 1 °C/s, Final temperature: ~ 400 C, Dwell time: ~ 90 mins

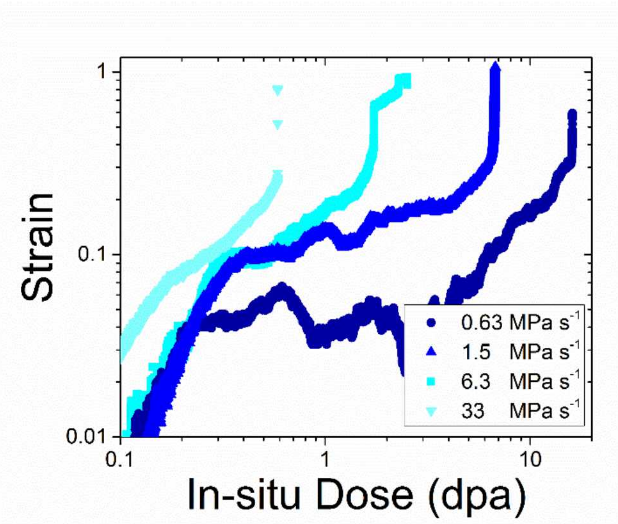


Hydride formation shown, for the first time by use of a novel TEM gas-cell stage, at elevated temperature and hydrogen pressure

Creep Response at Different Loading Rates



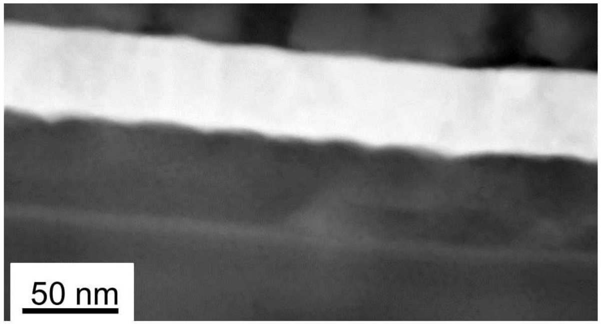




Significant creep observed at a fraction of the bulk yield strength

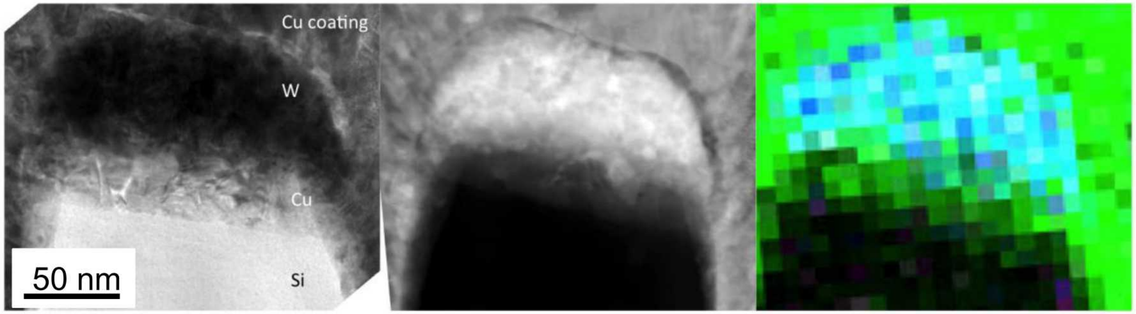
As-deposited Sample

ADF-STEM



Post Creep Characterization

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



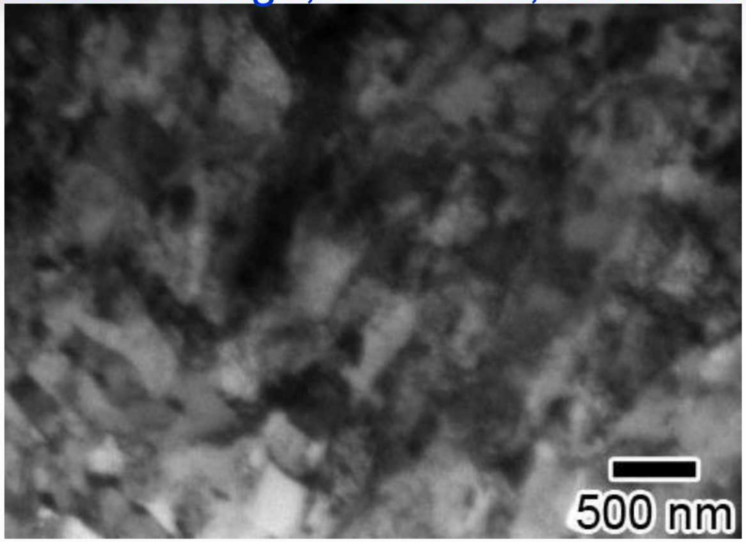
Compression (creep) only observed in Cu layer

In situ TEM Ion Irradiation

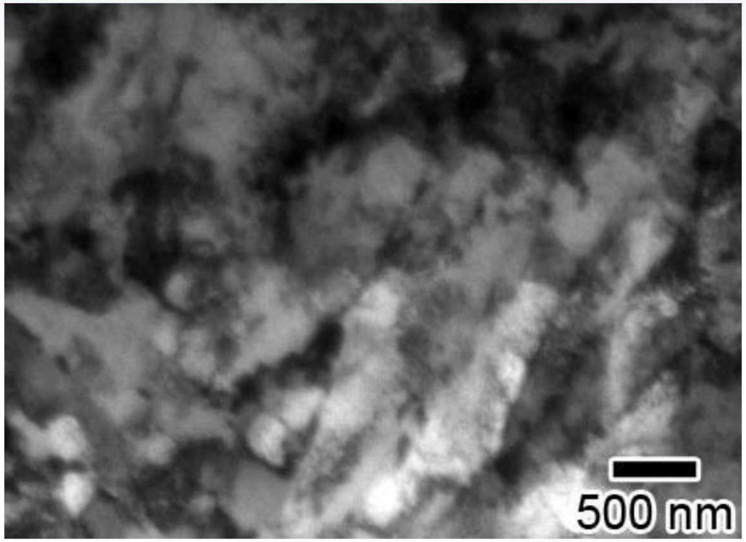
HT9 3 MeV Cu^{3+} at ~10 nA RT

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

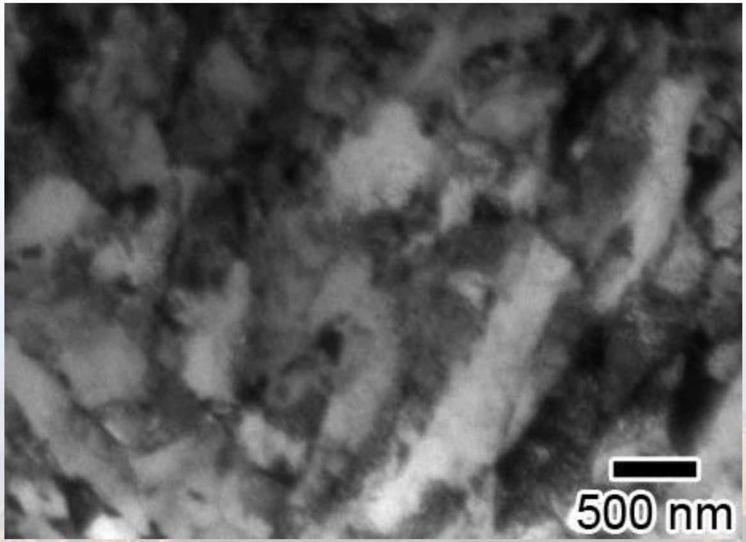
Initial



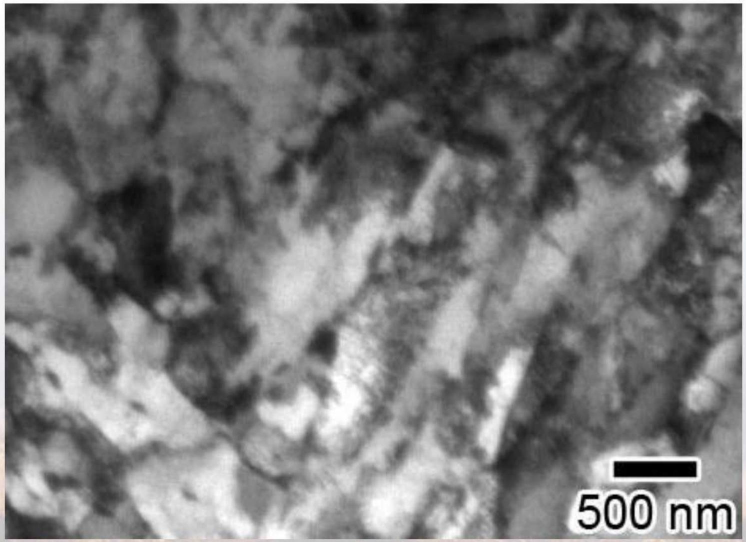
2 hrs



3 hrs

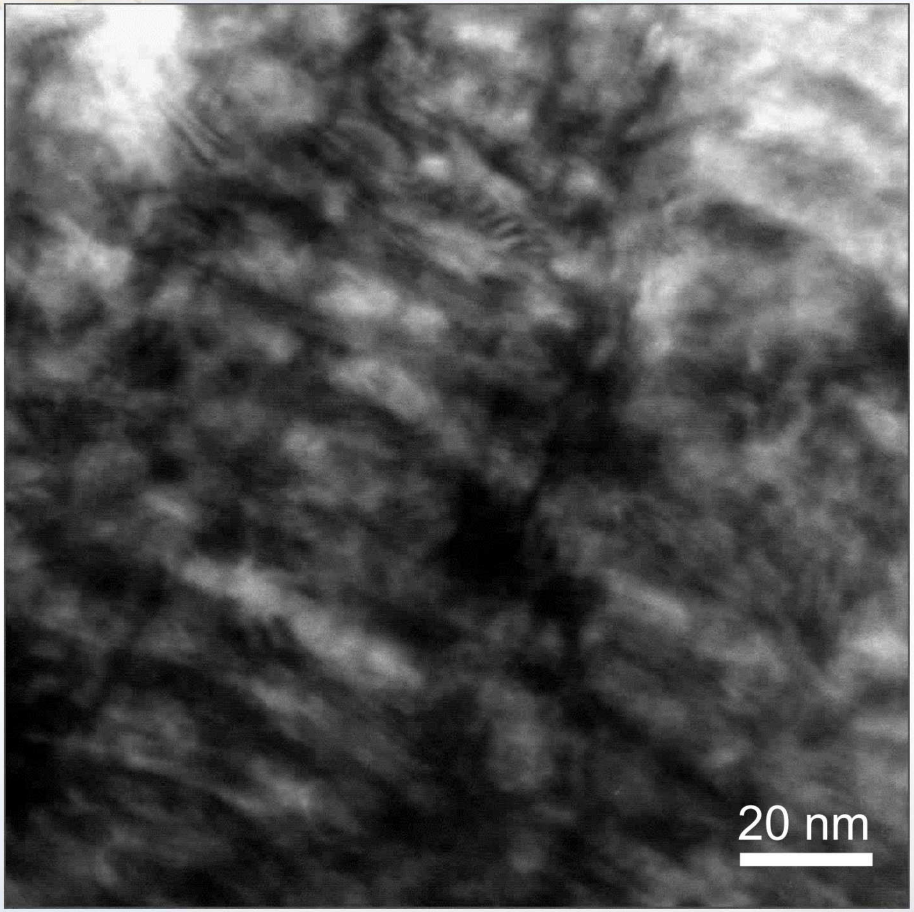


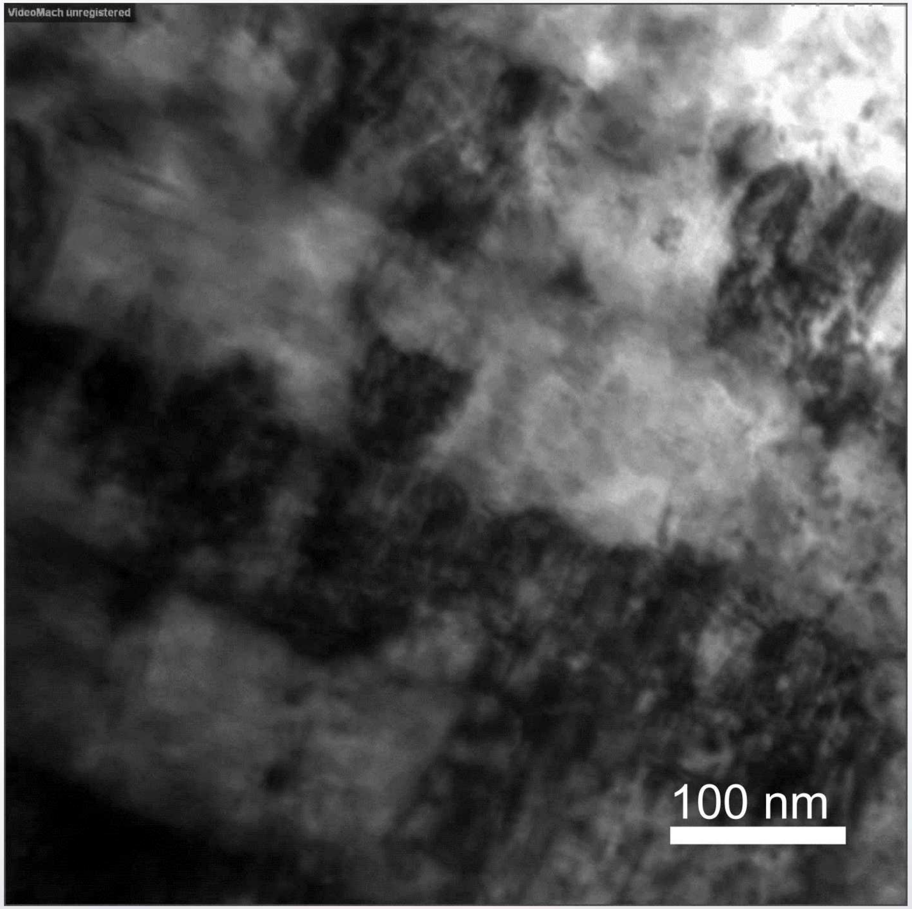
6 hrs



Layer Deposition to Provide Defect Sinks

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



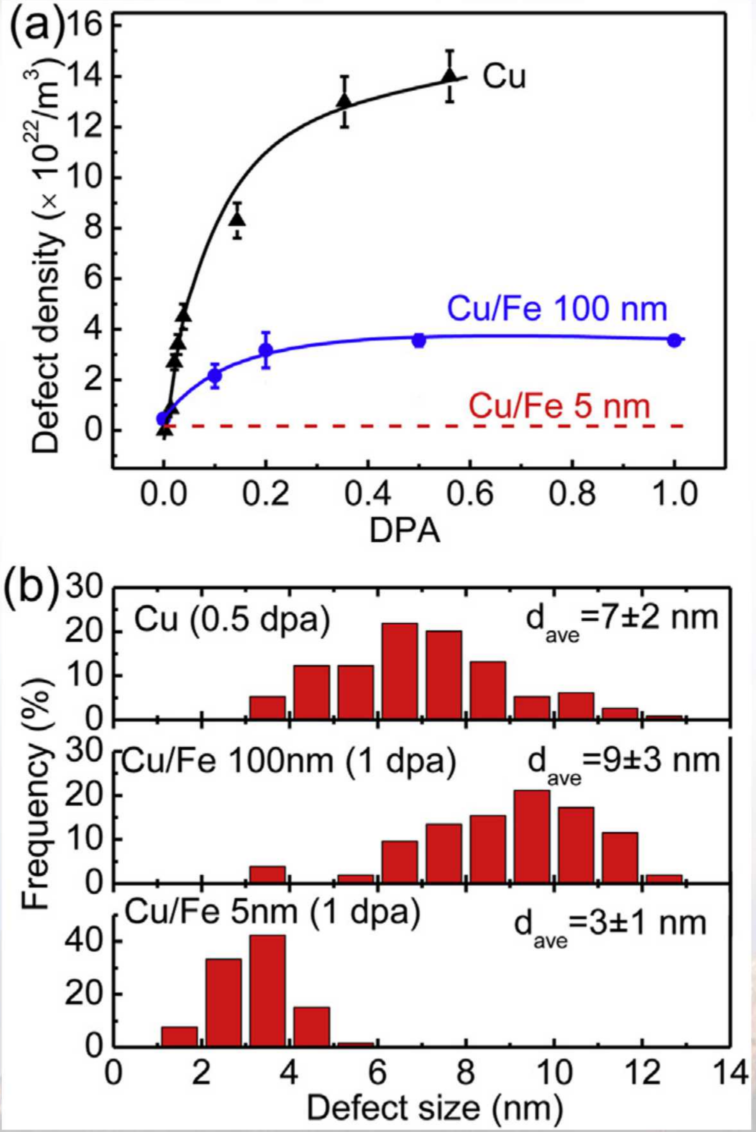


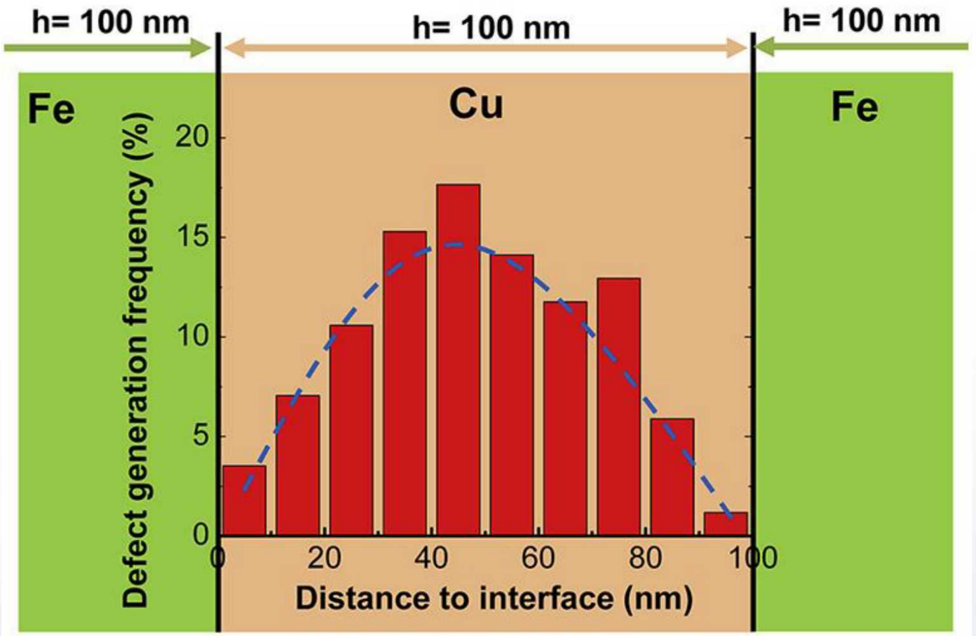
***In situ* TEM self-ion irradiation of Cu/Fe 100 nm and 5 nm multilayers with 3 MeV Cu**

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

Layer Deposition to Provide Defect Sinks

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

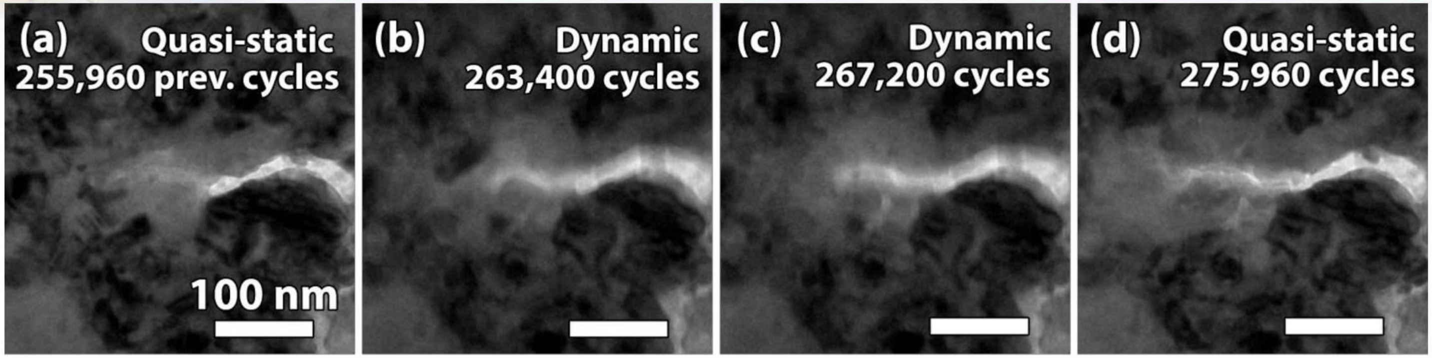


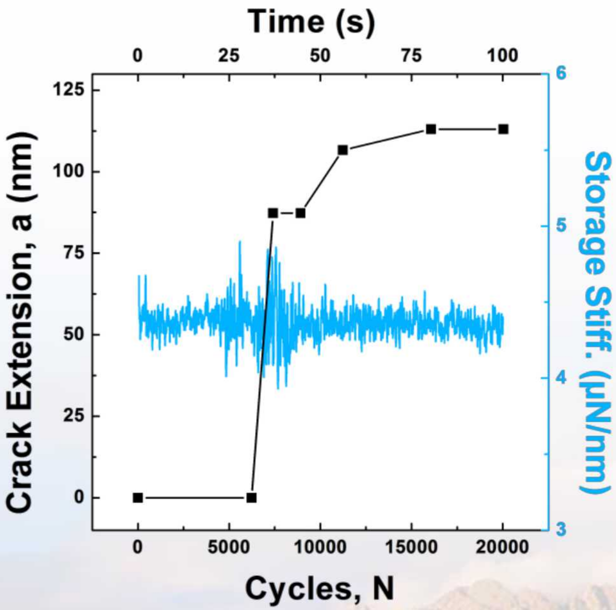


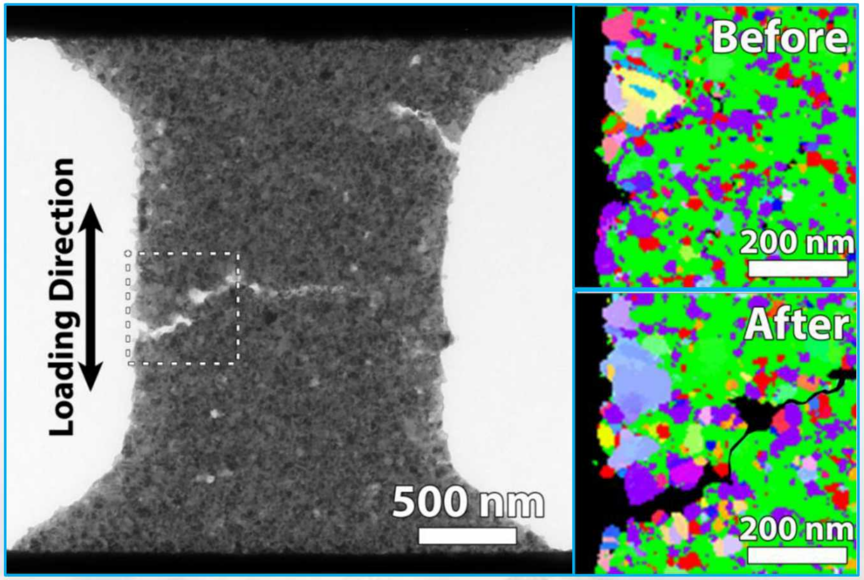
- **Layered structure provides significant improvement over either pure system.**
- **The radiation tolerance improves with decreasing layer thickness.**

High Cycle Fatigue: Crack Growth Quantified

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





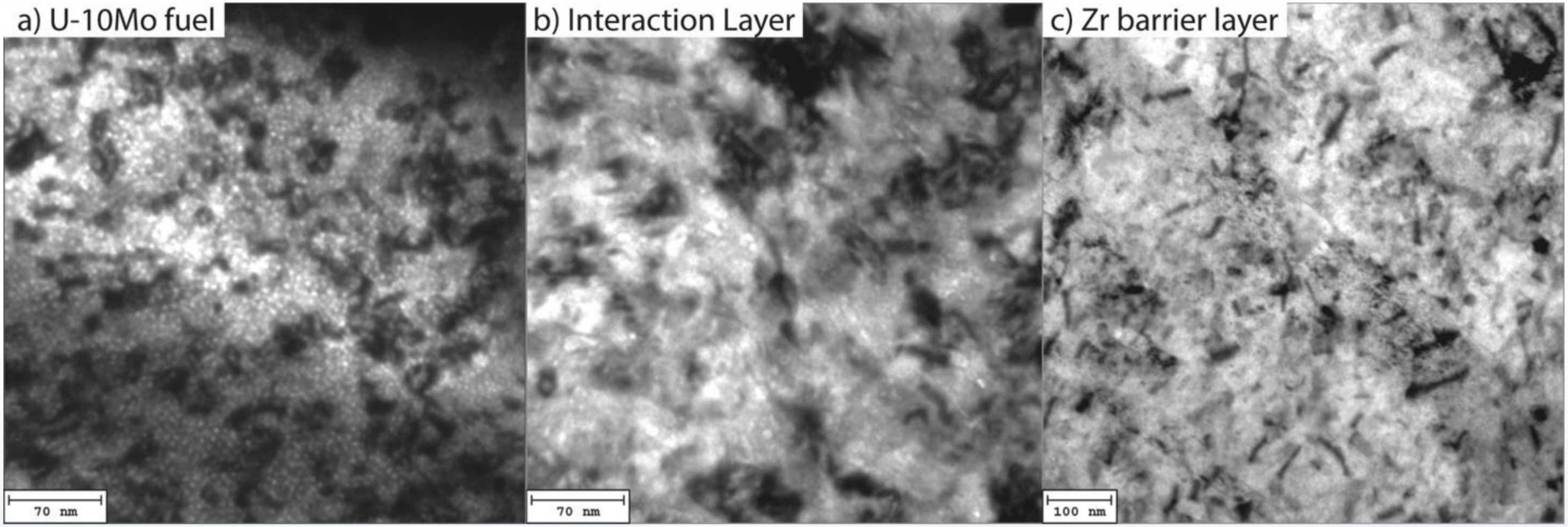


- Picometer-per-cycle measured crack growth rate
- Evidence of fatigue-induced grain growth.

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

Collaborators: C. Barrr, A. Aitkaliyeva

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

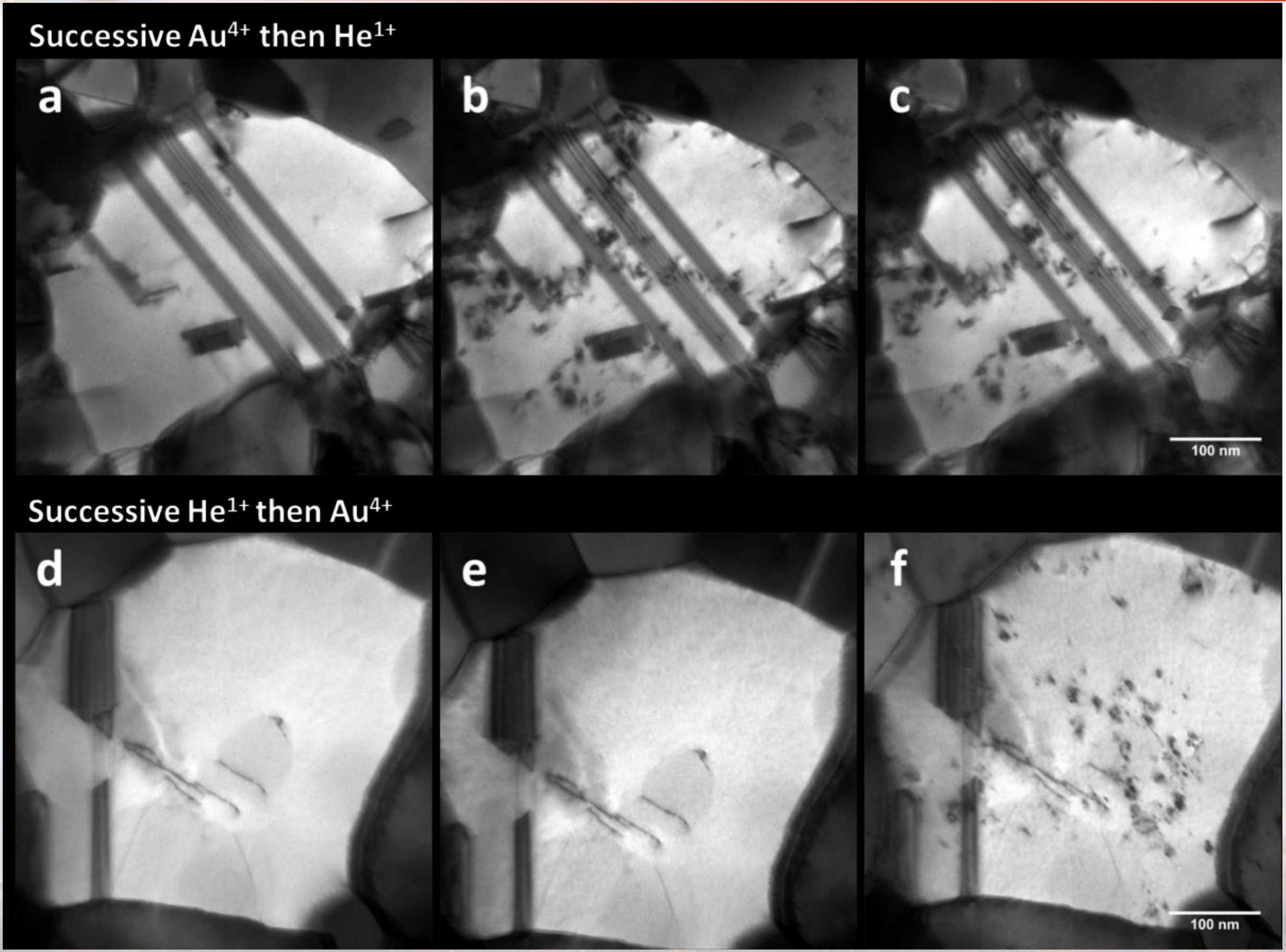


- Continued stability of the periodic gas bubble superlattice in the U10Mo fuel
  - Apparent surface oxide (likely UO_2) post in-situ TEM irradiation

- Growth of dislocation loops (arrowed in red) in Zr barrier layer apparent and indications of increased coalescence of porosity in interaction layer

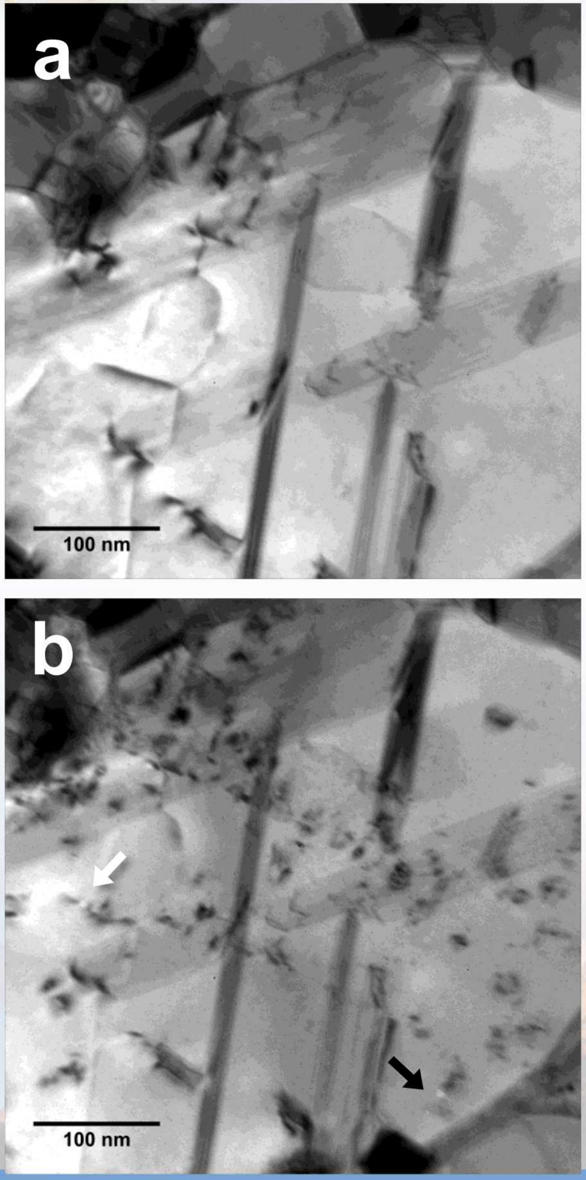
In situ Successive Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor



In situ Concurrent Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor



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