SAND2022-6440C

What is the Physical Limit of Coupled *In situ* Microscopy Experiments?

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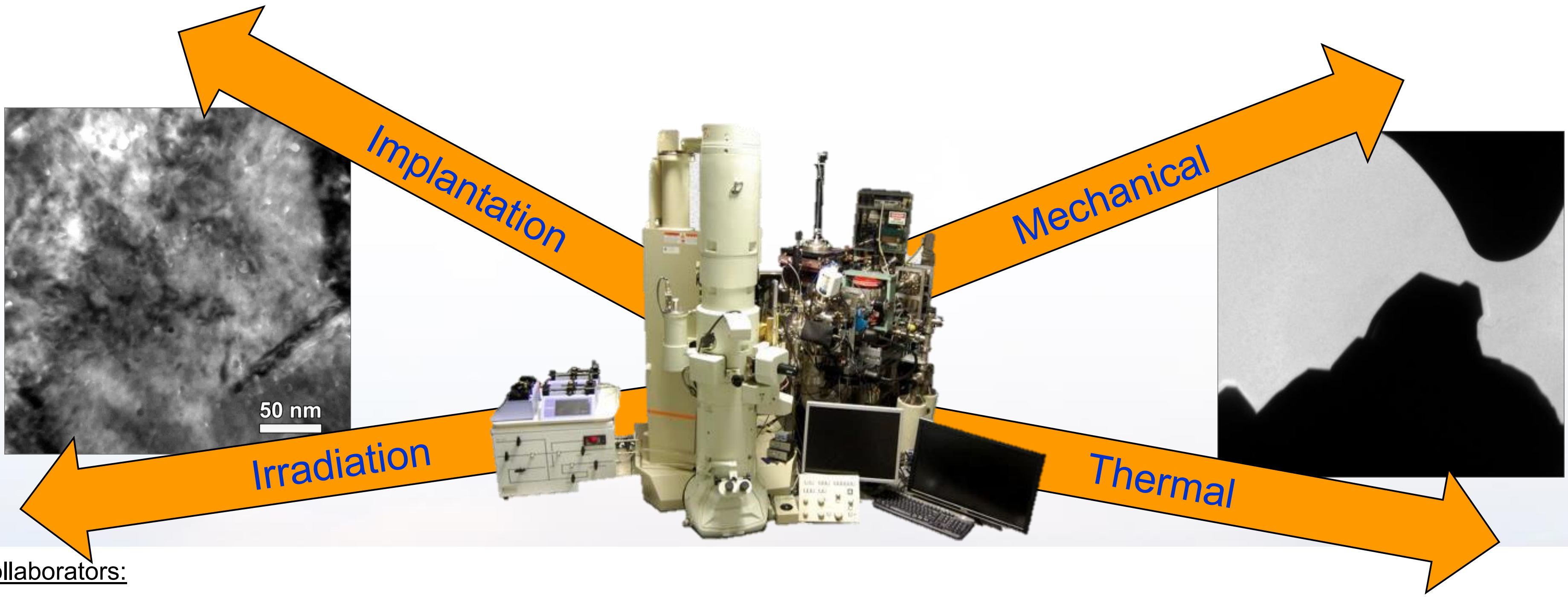

Collaborators:

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

This work was partially funded by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. This work was ... Office of Science. Sandia National Laboratories is a multi-mission laboratory ... , 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.

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Benefits & Limitations of *in situ* TEM

Benefits

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

Limitations

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

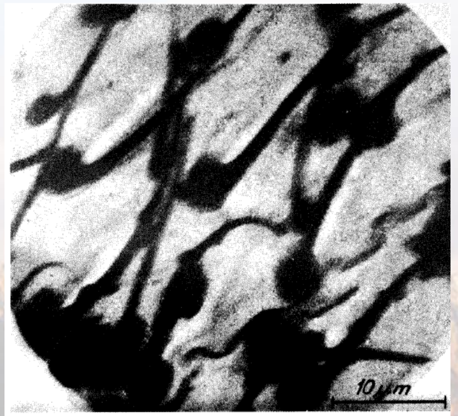


Fig. 6: Wing surface of the house fly.
(First internal photography, U = 60 kV, M_{el} = 2200)
(Driest, E., and Müller, H.O.: Z. Wiss. Mikroskopie 52, 53-57 (1935)

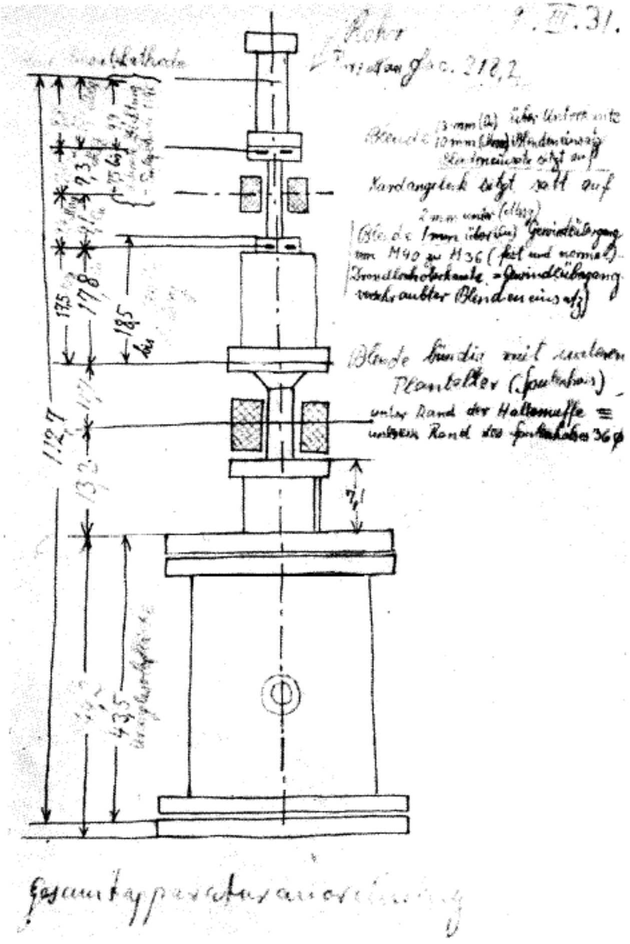
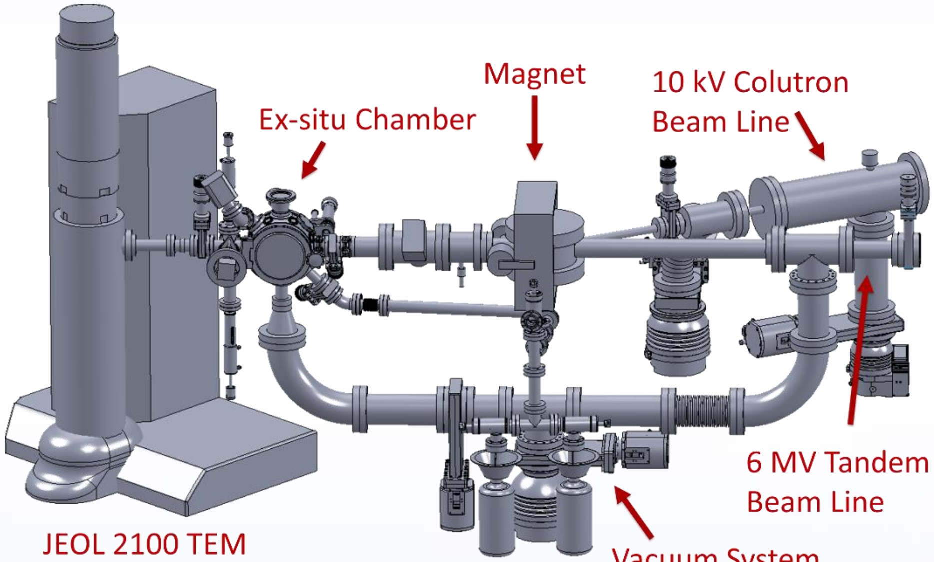


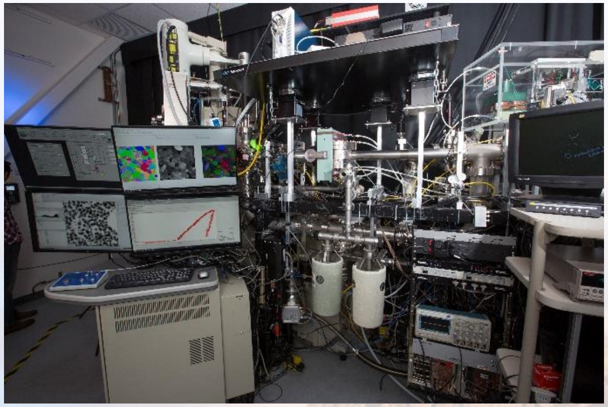
Fig. 2: Sketch by the author (9 March 1931) of the cathode ray tube for testing one-stage and two-stage electron-optical imaging by means of two magnetic electron lenses (electron microscope) [8].

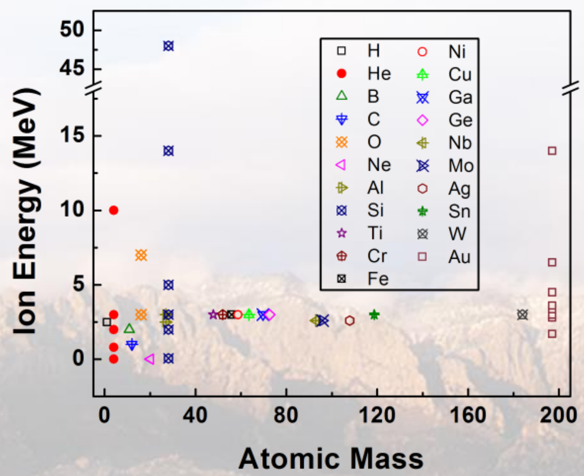
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

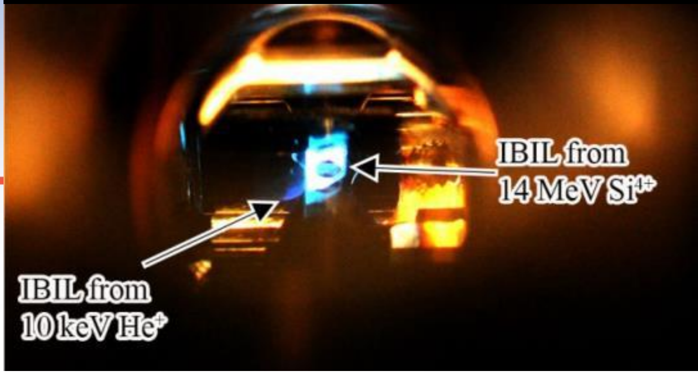
10 kV Colutron - 200 kV TEM - 6 MV Tandem

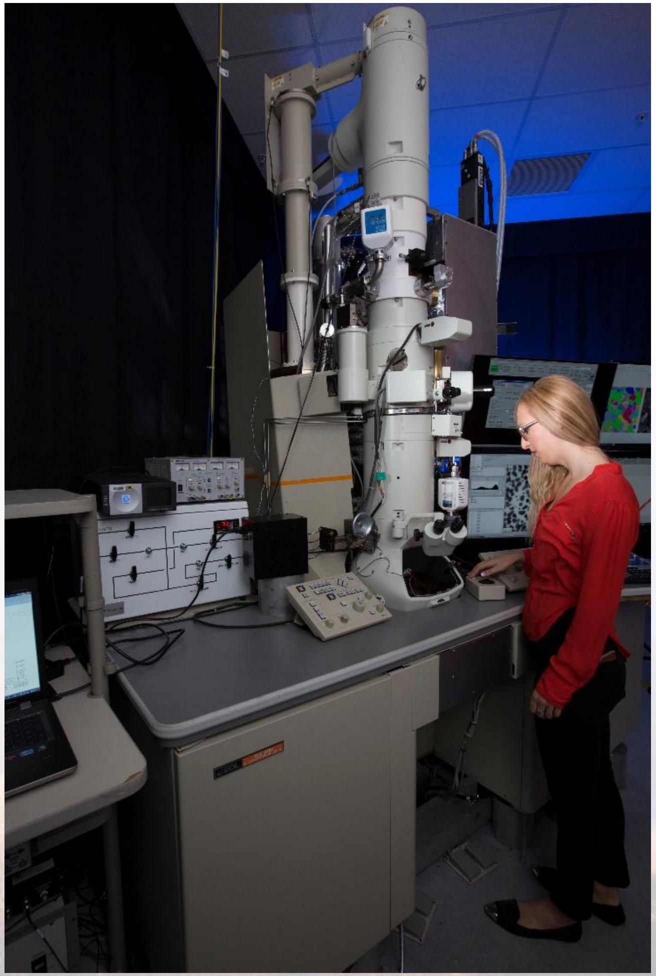






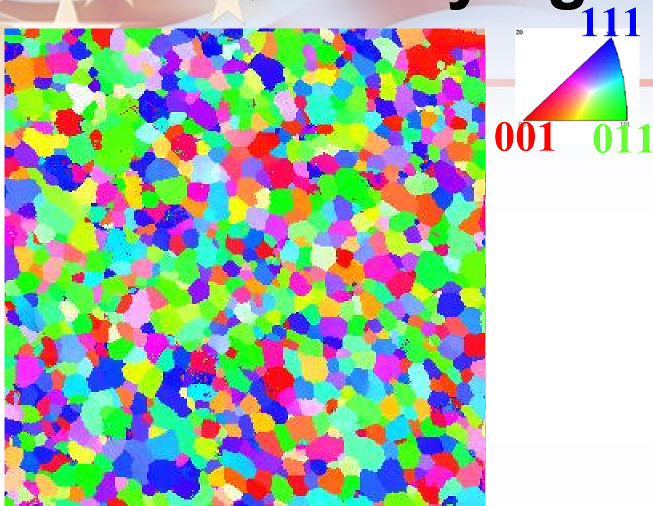
IBIL from a quartz stage inside the TEM



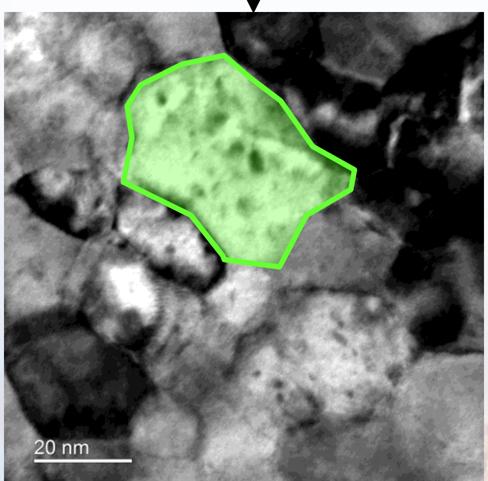


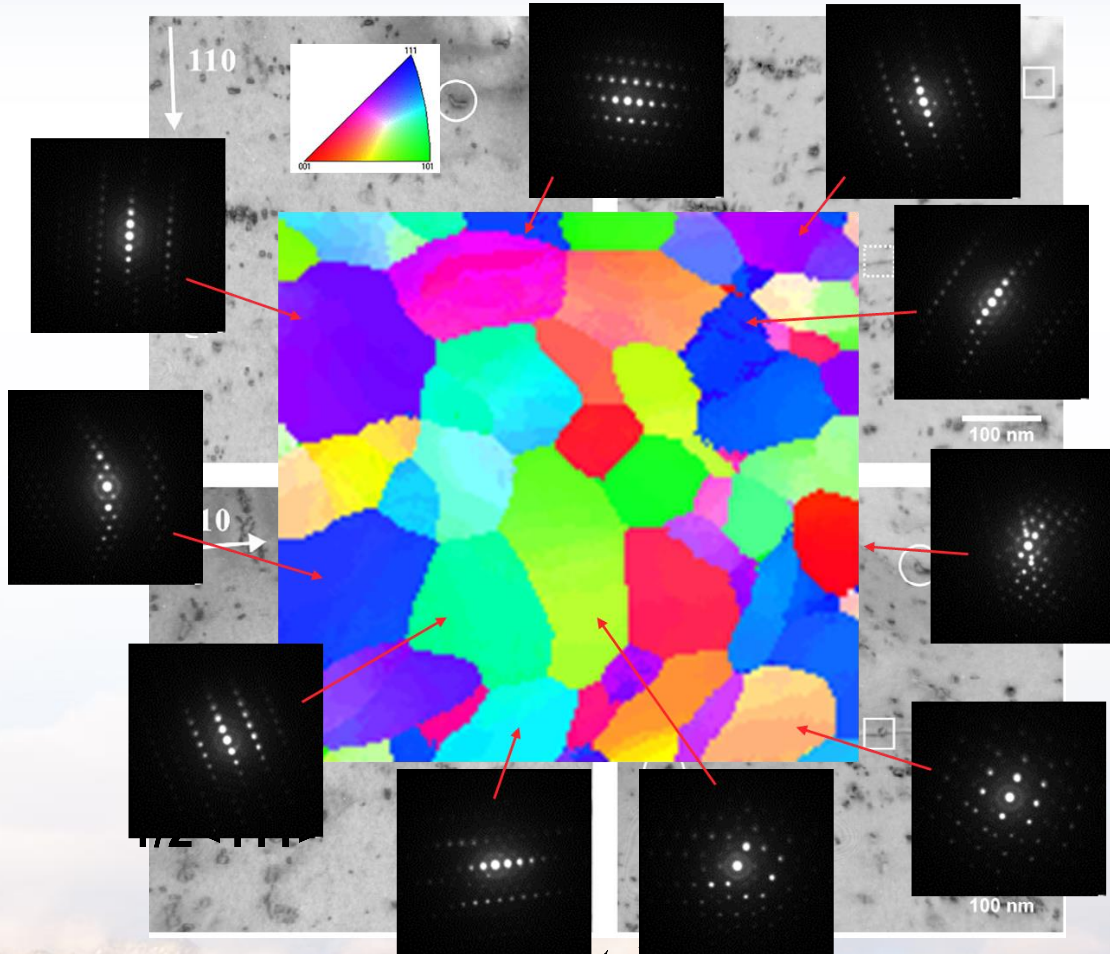
Quantifying Damage in Nanocrystalline W

Collaborator: W.S. Cinningham and J. Trelewicz

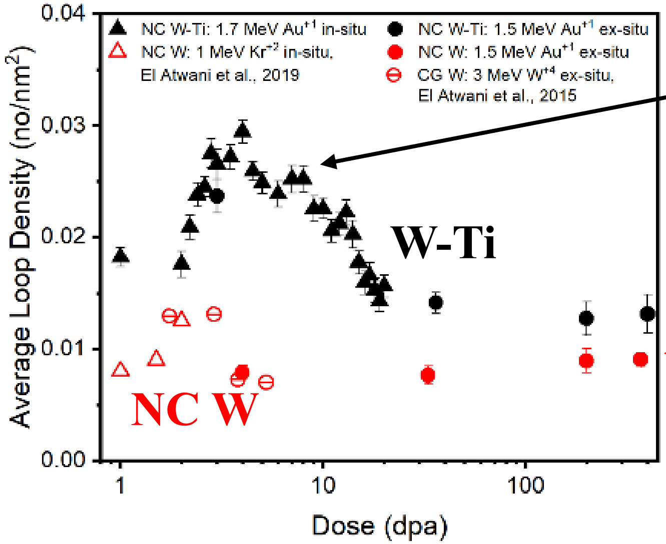


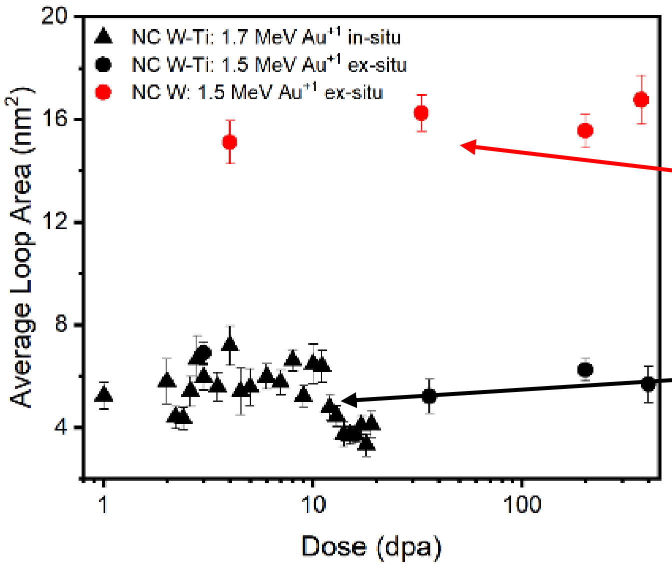
1.7 MeV Au+ ions to 10 dpa





Damage Evolution in NC W and W-Ti Films



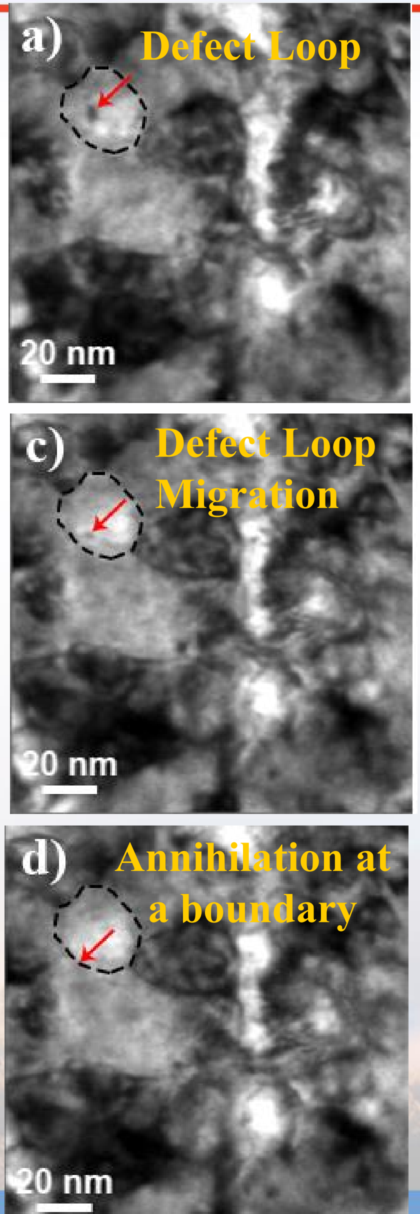


Damage peak followed by reduction in loop density

Steady-state loop density slightly greater than NC W

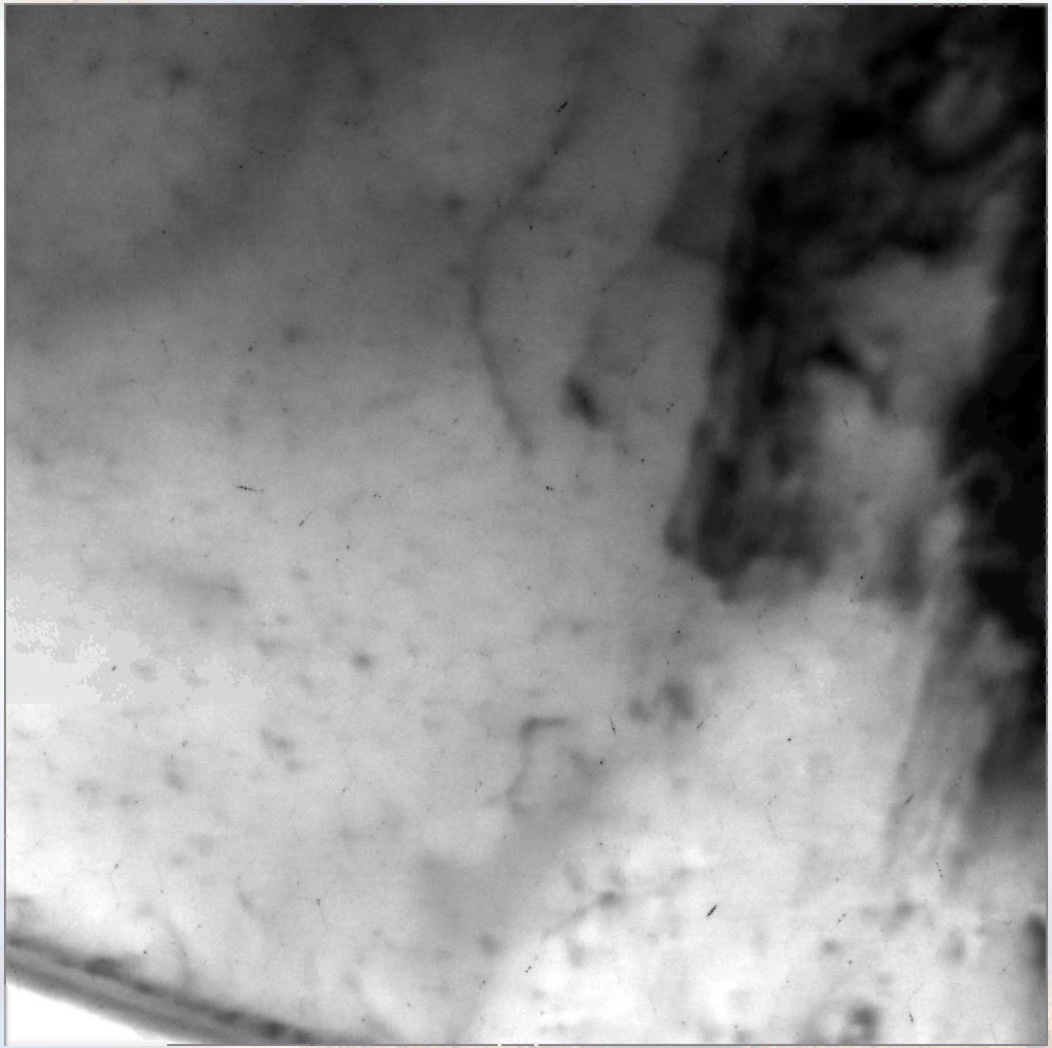
Considerably larger loops in NC W relative to W-Ti

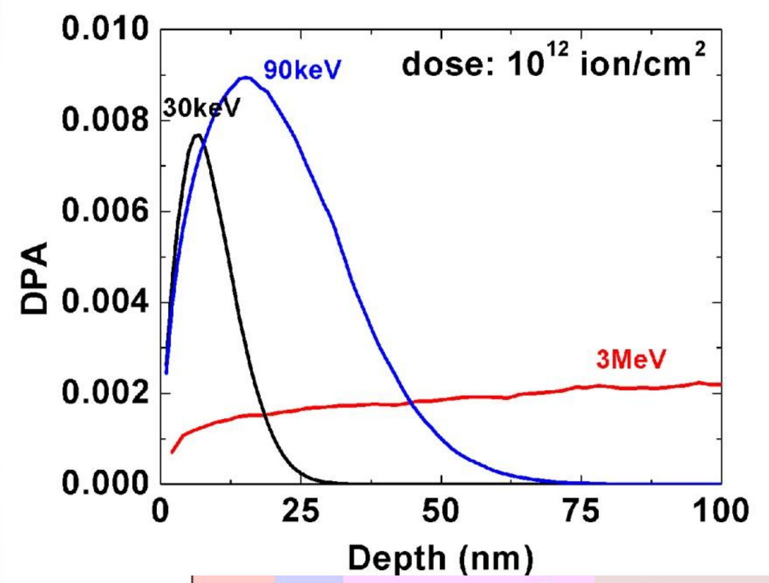
Only subtle variations in the loop area

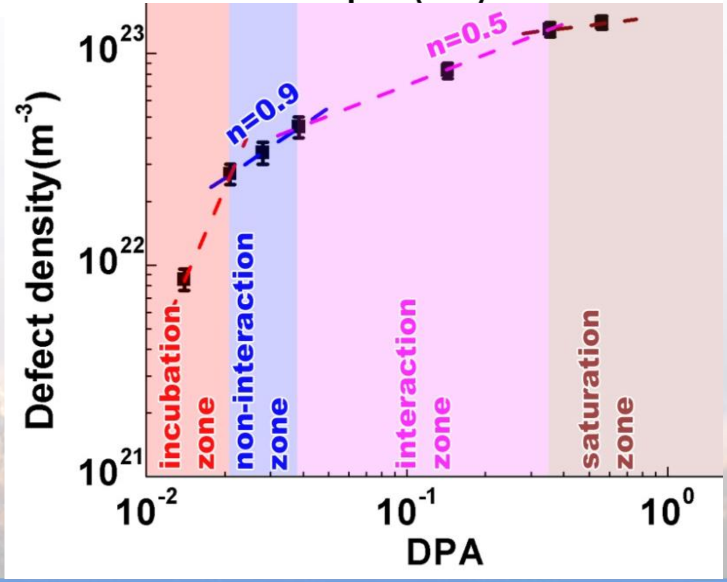


Evolution of Radiation Defects in Cu TEM Foil

Collaborators: N. Li, A. Misra



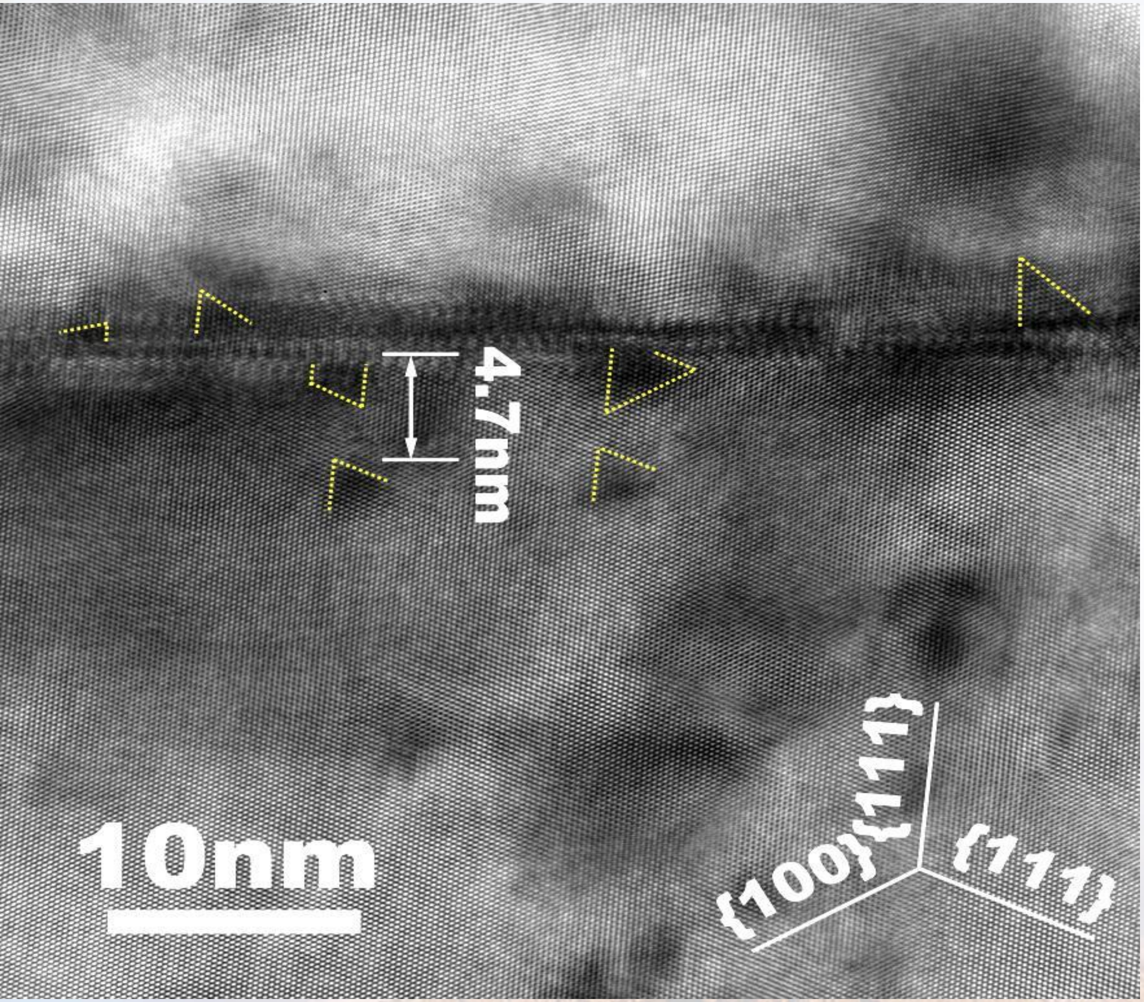


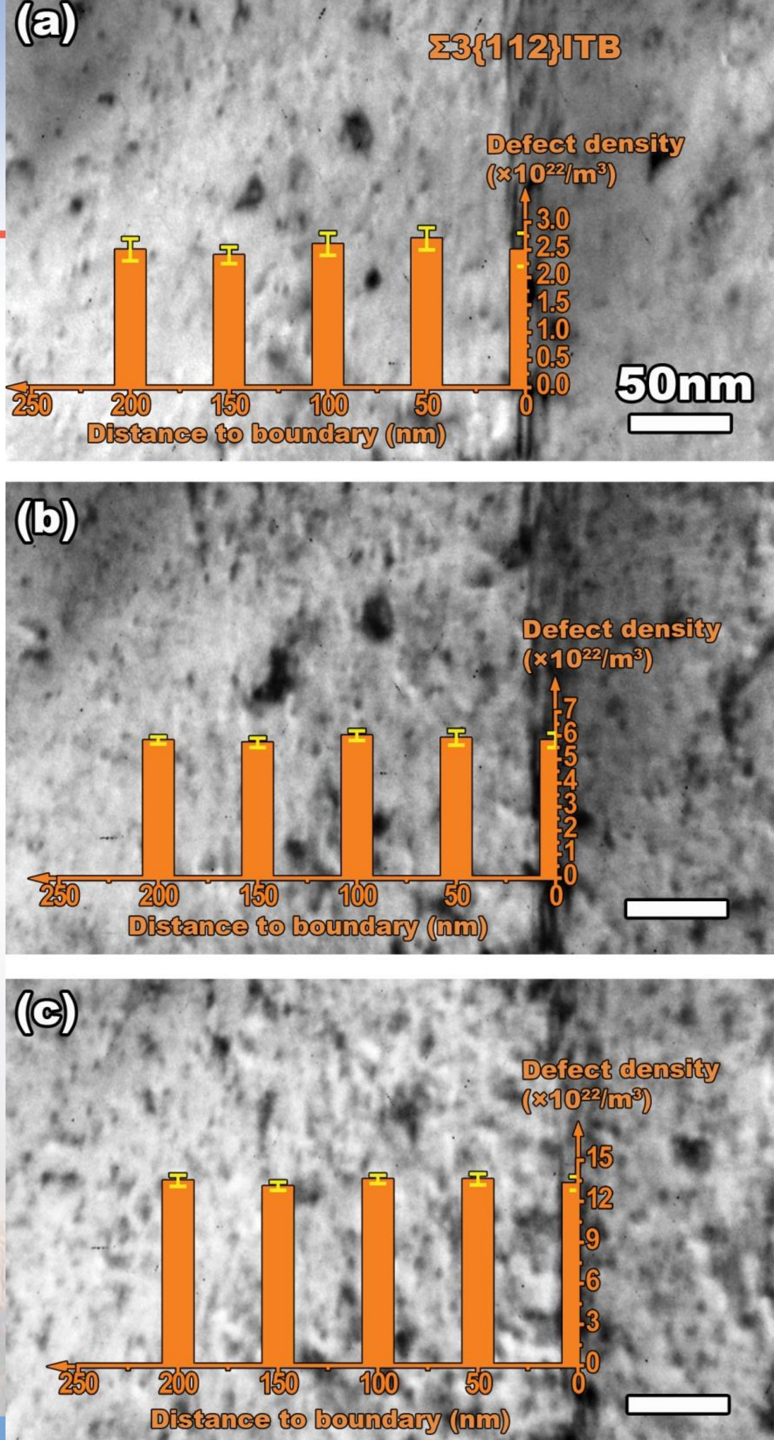


- FIB processed sputter deposited high purity Cu foils
- Tailored to have high density of Σ3 boundaries

Defect are Altered Little by the Presence of Grain Boundaries

Collaborators: N. Li, A. Misra



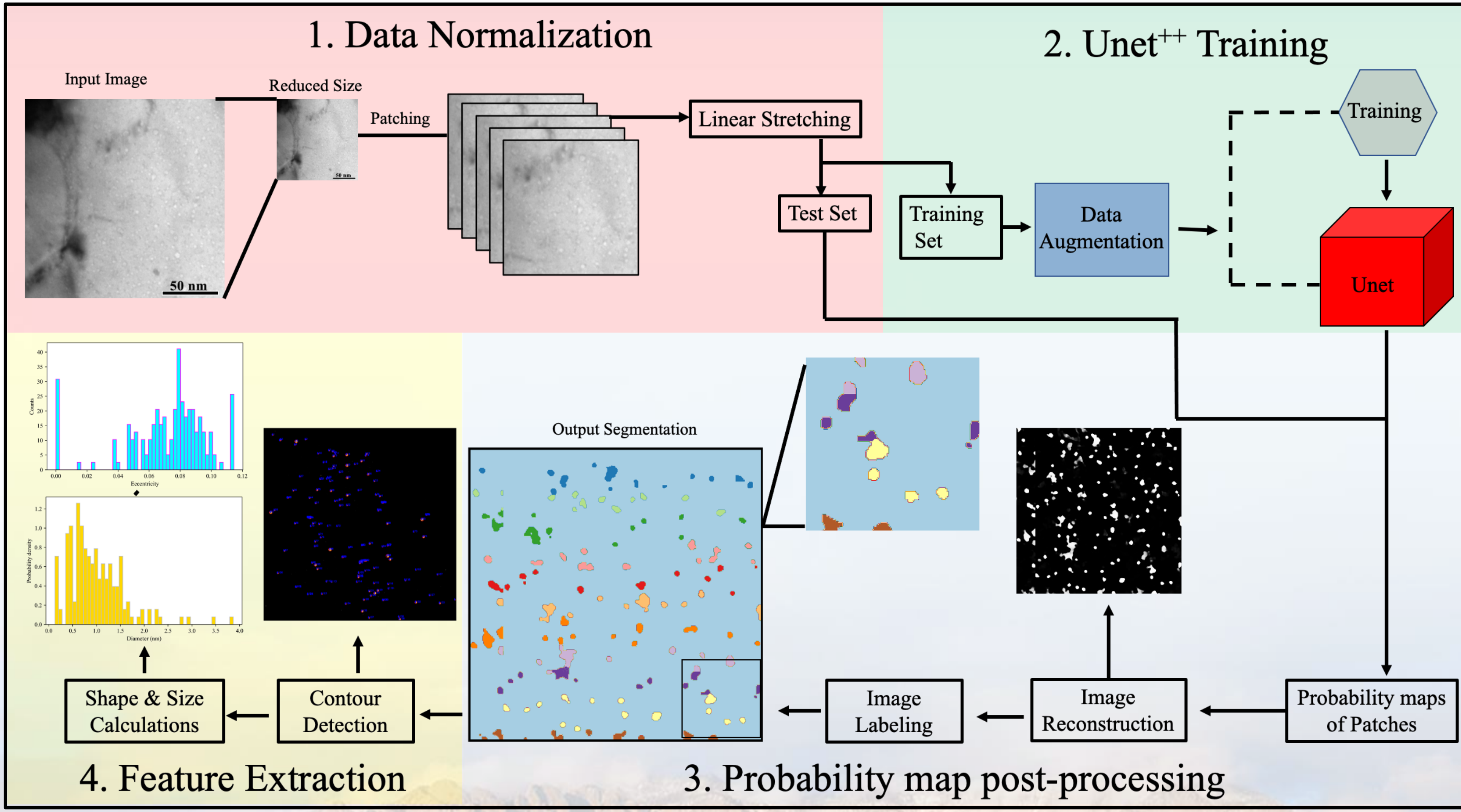


SFT appear to be directly at GB

No change in defect density is observed near GB

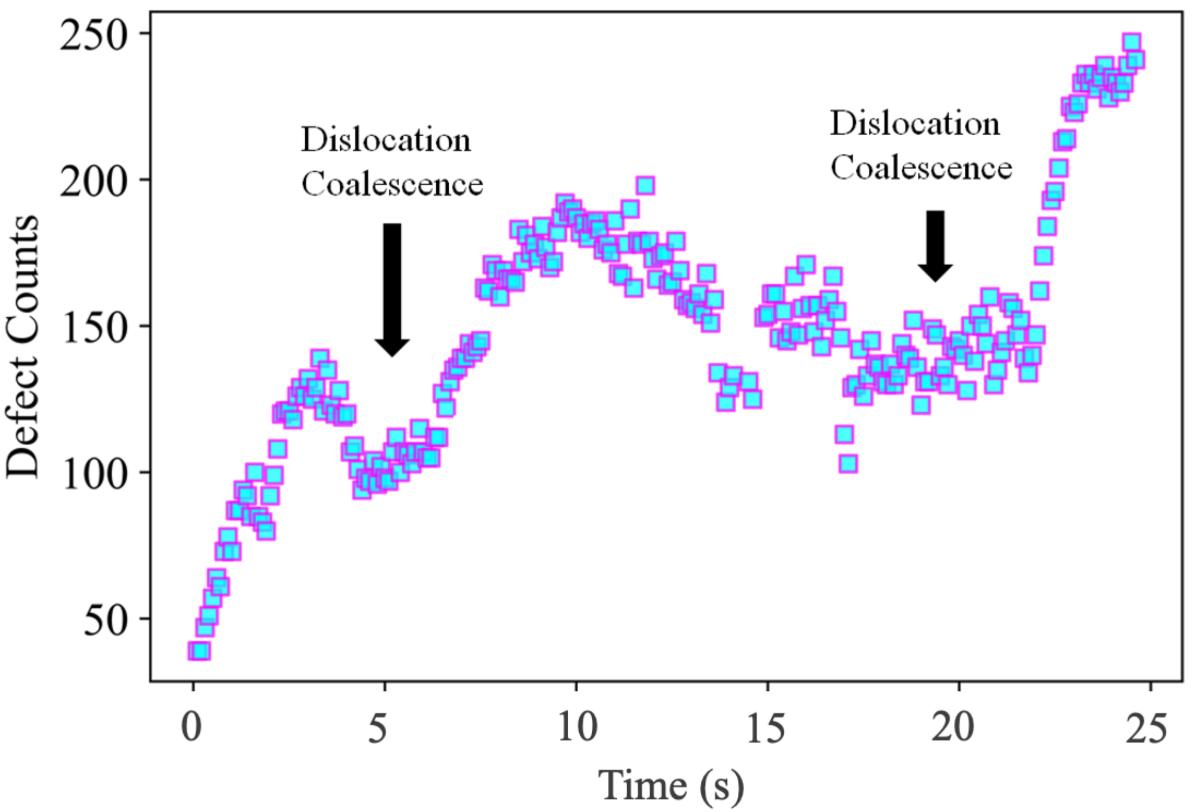
Applying Machine Learning to I^{3}TEM Data

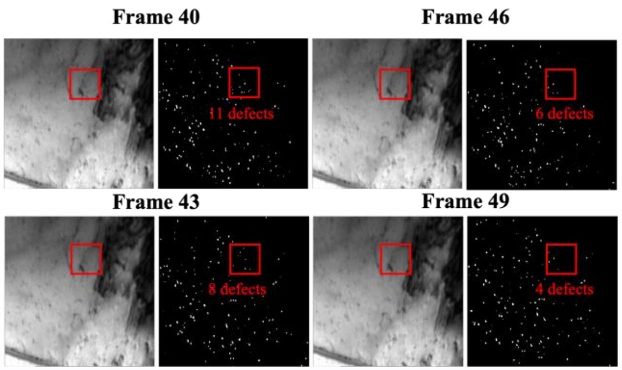
Collaborators: K. Bruns, M.C. Scott, A. Minor

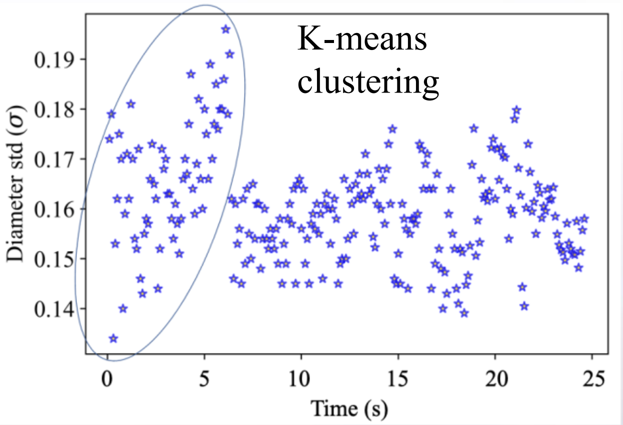


A 2nd Look at Self-ion Irradiation of Cu Metal

Collaborators: K. Bruns, M.C. Scott, A. Minor

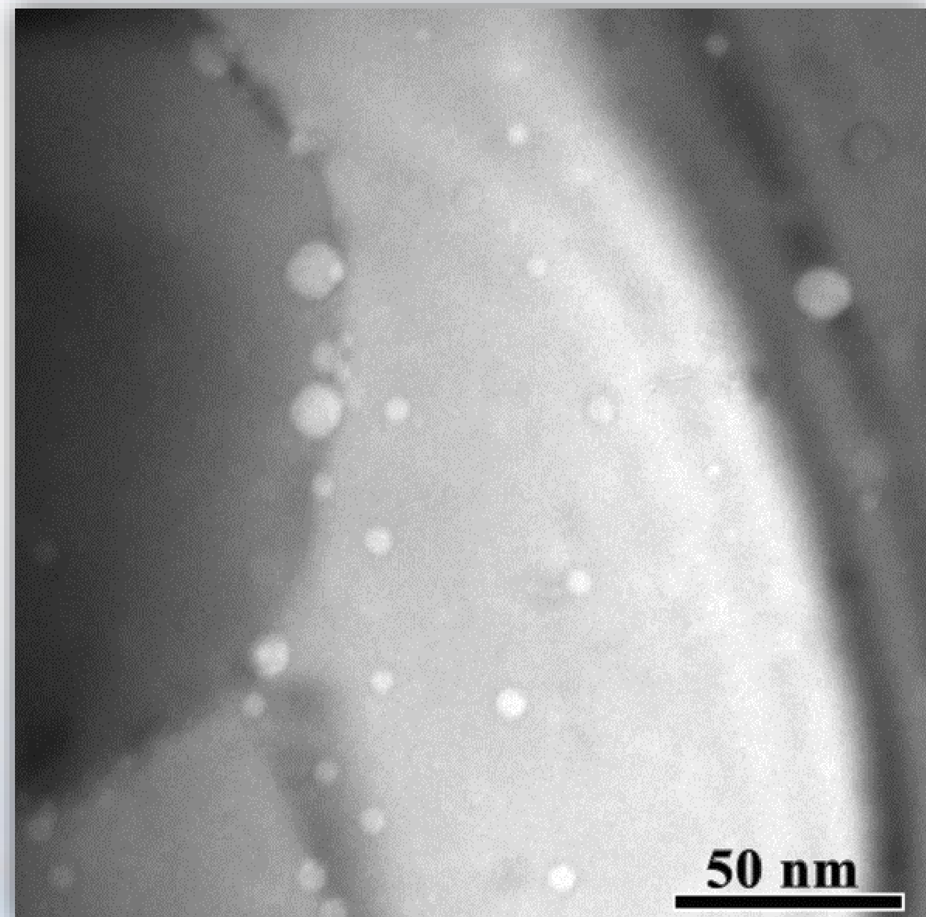


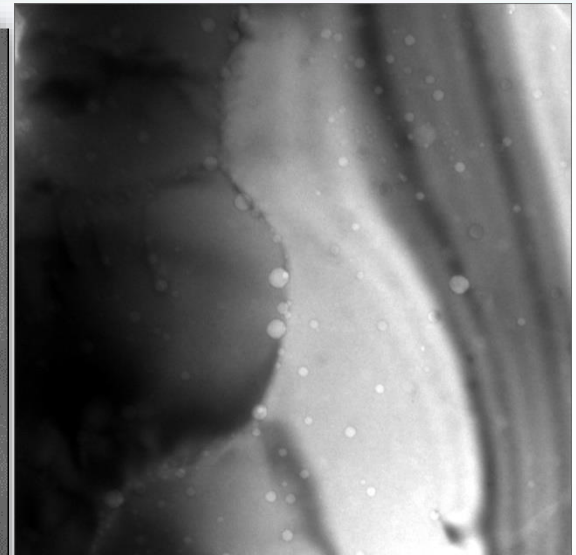




Cavities Role on Grain Boundary Motion

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





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

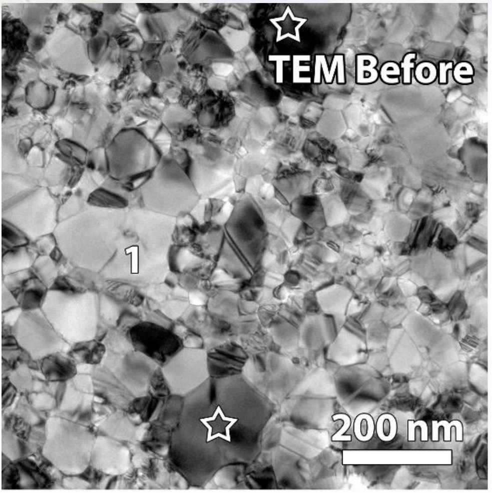
Cavities effect grain boundary mobility, triple junction angle

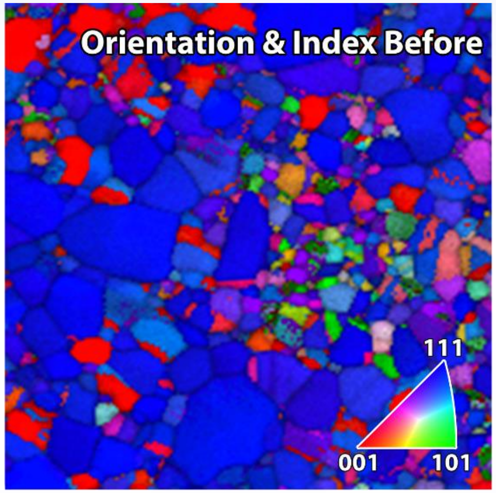
↔

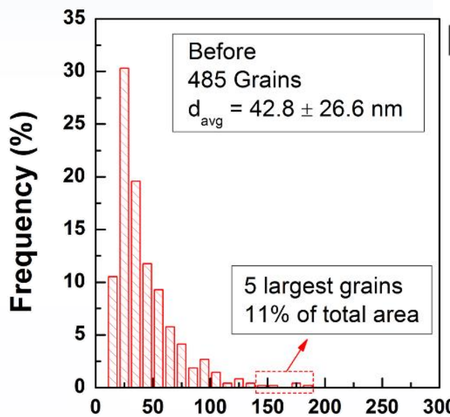
grain boundary motion alters cavity coalescence

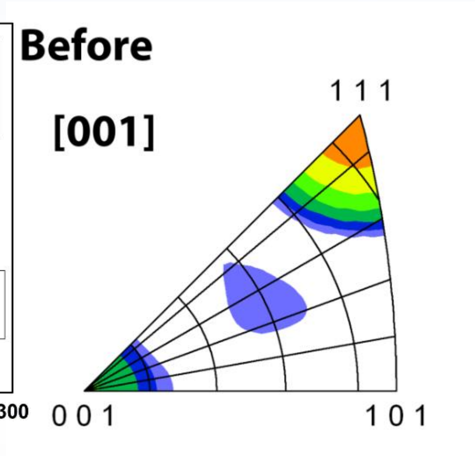
Quantifying Grain Boundary Radiation Stability of Nanocrystalline Au

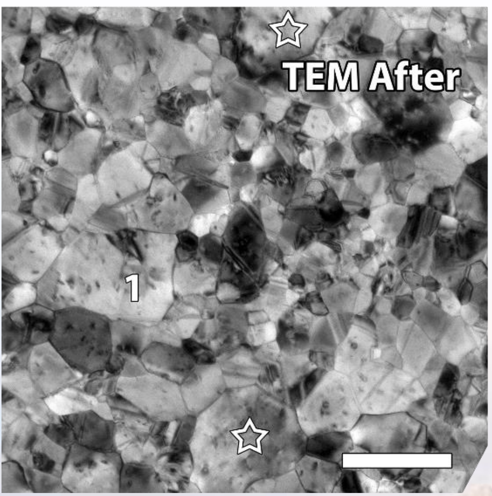
Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles

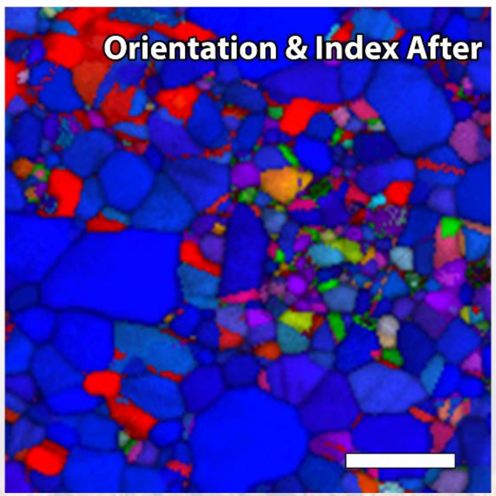


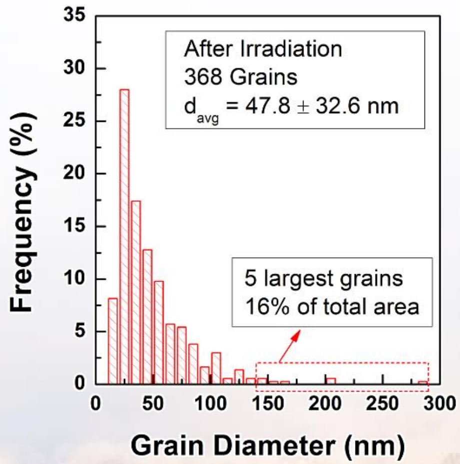


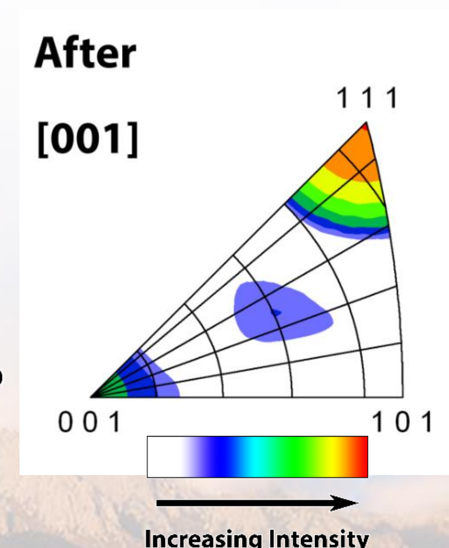












10 MeV Si

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

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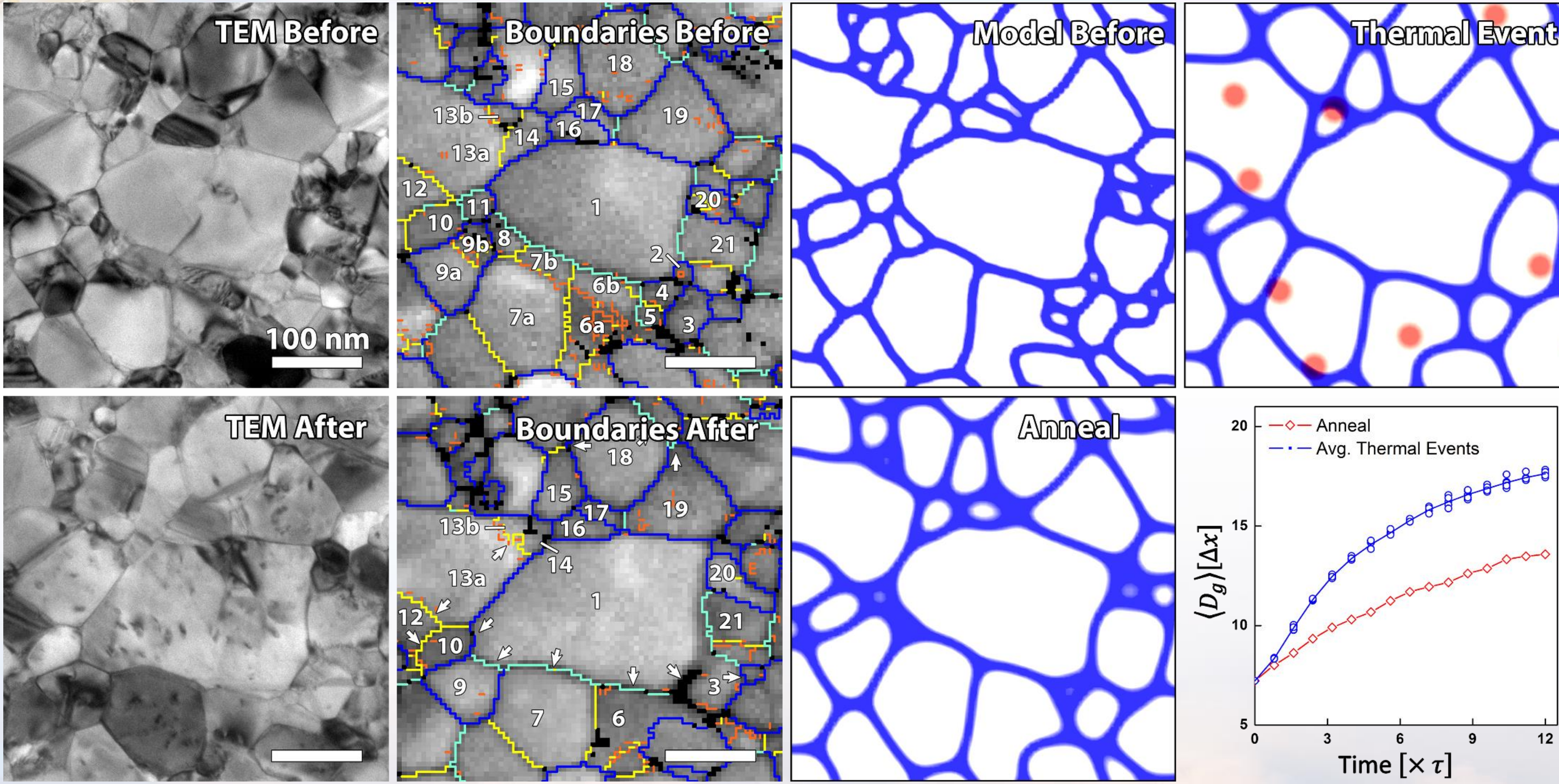
Direct Comparison to Mesoscale Modeling

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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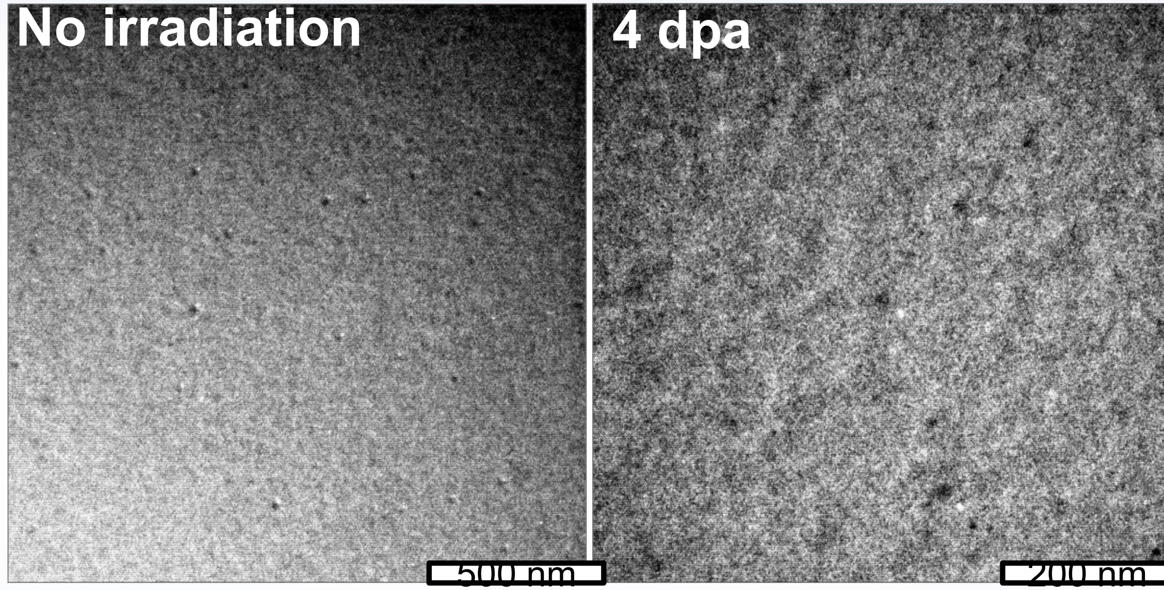
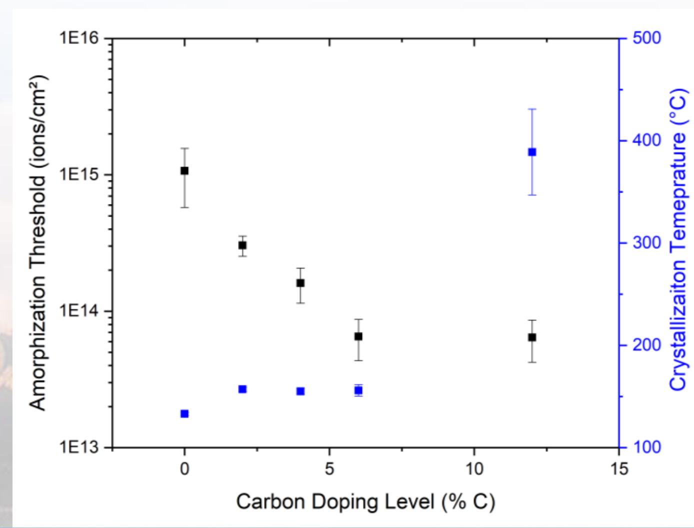
Radiation Tolerance in Phase Change Memory

Contributors: Trevor Clark, Eric Lang, Ethan Scott, and David Adams

- **90 nm-thick GST with 0-20 %C**
- **Plan View:**
  - Anneal (5 ˚C/min)
  - RT irradiation (4dpa)
    - Anneal (5 ˚C/min)
  - 100 ˚C hold & irradiation (4dpa)
- **Cross section FIB lift-outs:**
  - RT Irradiation
  - 200 C & 300 C Hold
- **Irradiation Conditions**
  - 2.8 MeV Au^{4+}
  - Up to 4 dpa



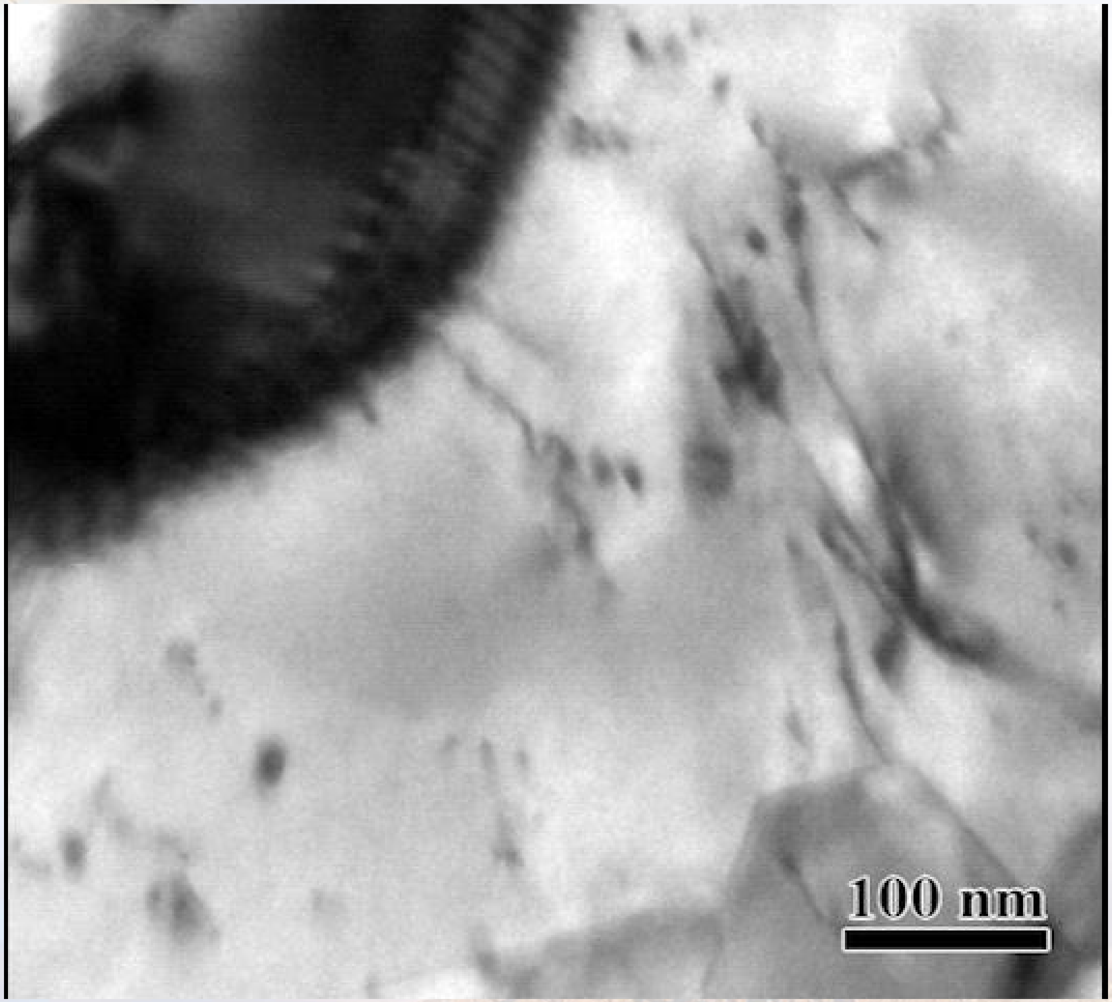
GST 0 %C Anneal 5°C/min from100-150 °C 15x speed



Amorphization and crystallization temperature are carbon dependent

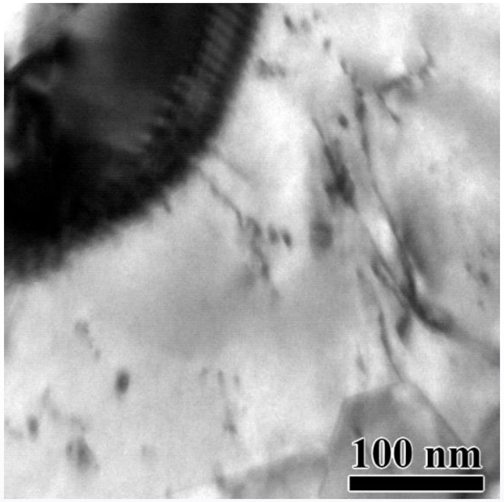
1D Brownian Motion in Real Time

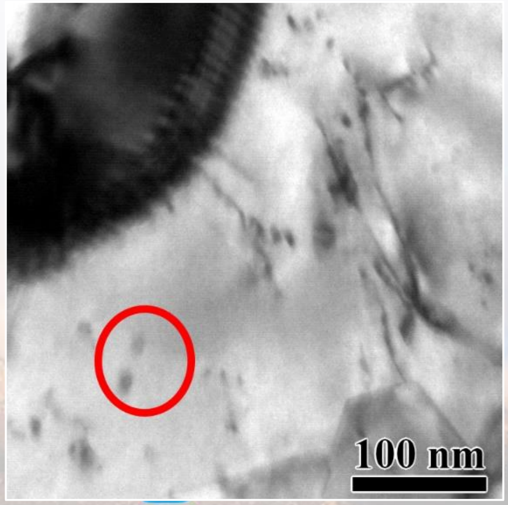
Collaborator: D.C. Bufford



Triple beam condition:

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

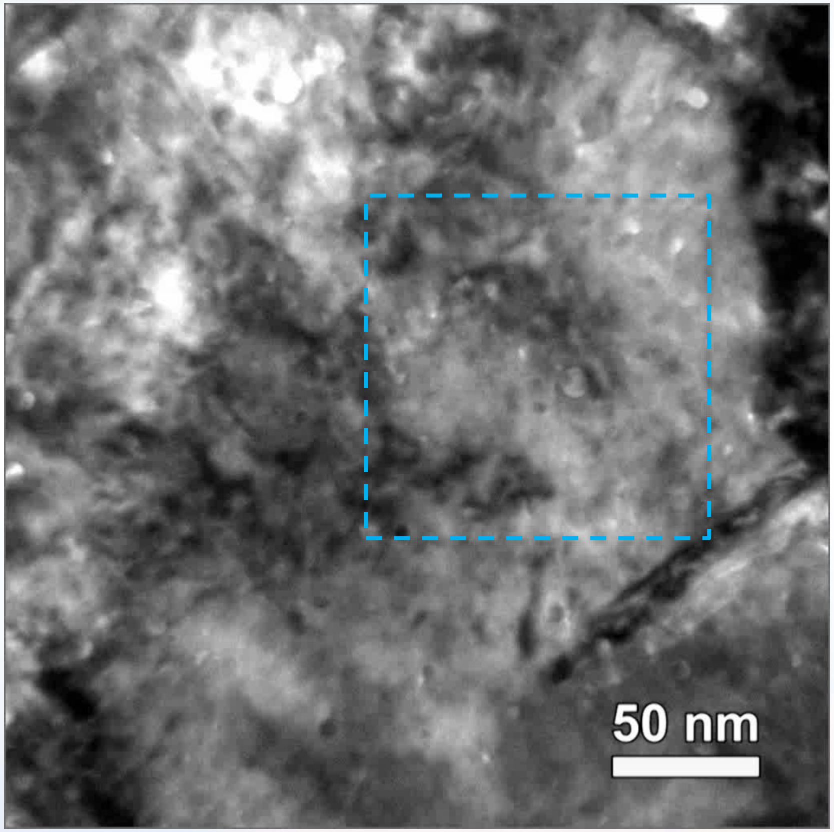


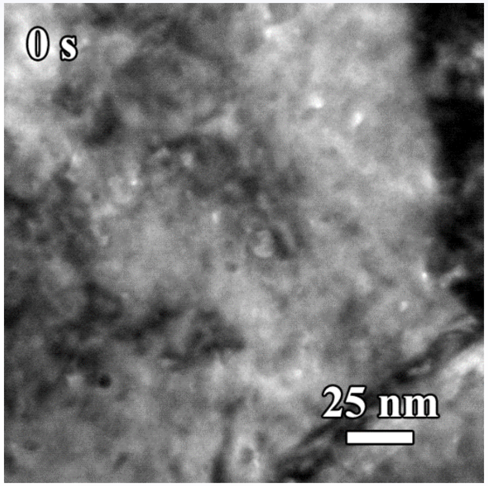


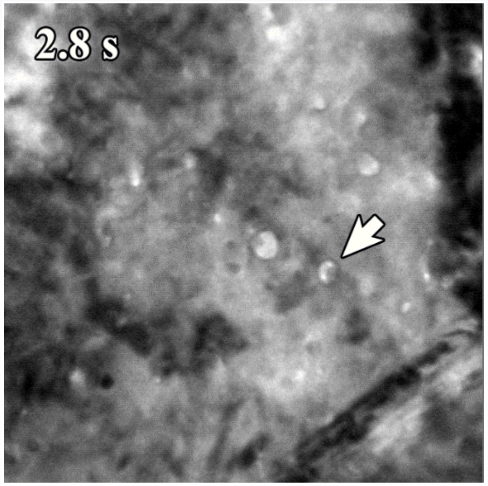
- **Dislocation loop moves between two pinning sites**
  - ~30 nm apart

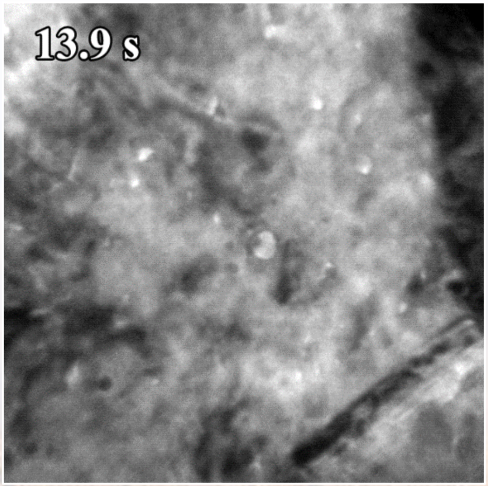
Simultaneous *In situ* TEM Triple Beam: 2.8 MeV Au^{4+} + 10 keV He^{+}/D_2^{+}

Collaborator: D.C. Bufford









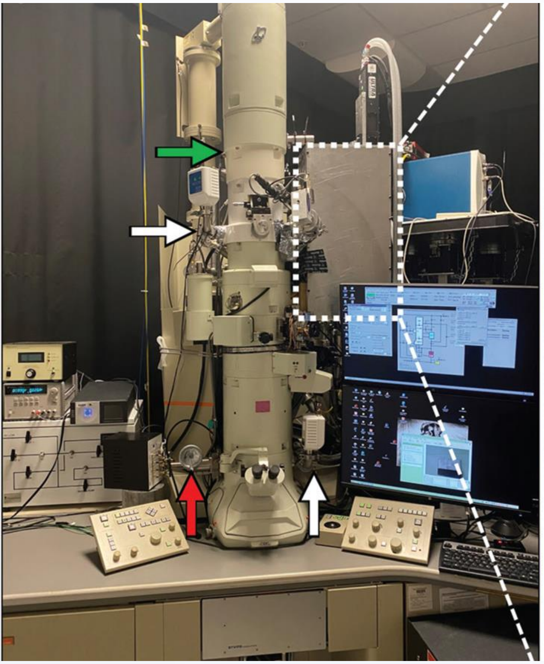
Speed x1.5

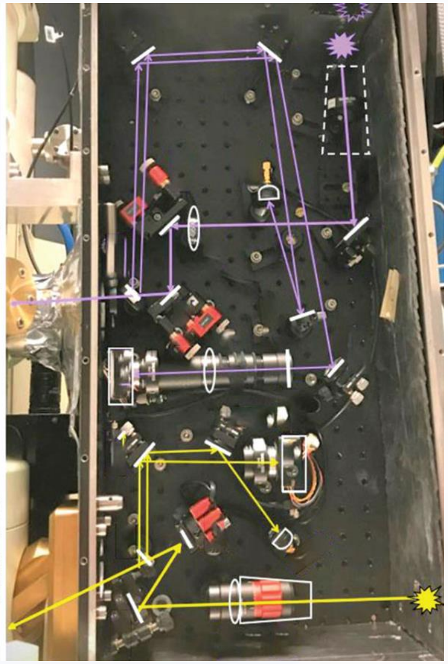
- **Approximate fluence:**
  - Au 1.2 $\times 10^{13}$ ions/cm^2
  - He 1.3 $\times 10^{15}$ ions/cm^2
  - D 2.2 $\times 10^{15}$ ions/cm^2
- **Cavity nucleation and disappearance**

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.

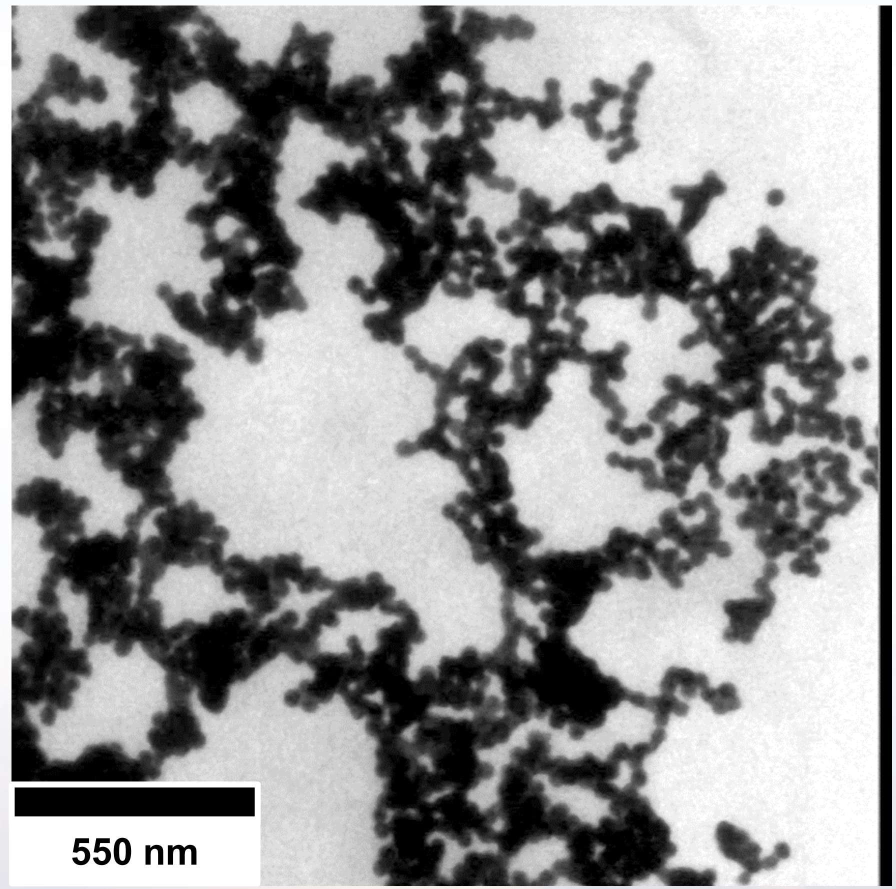
Laser Irradiation in a TEM

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





A Complex Combination of Sintering, Reactions, and Ablation Occurs



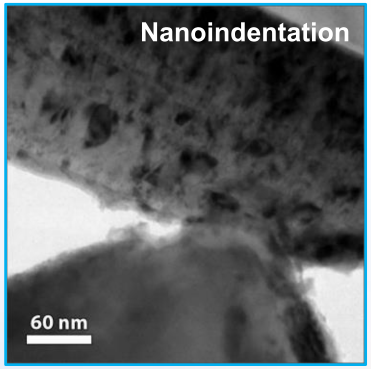
Speed = 2.5x

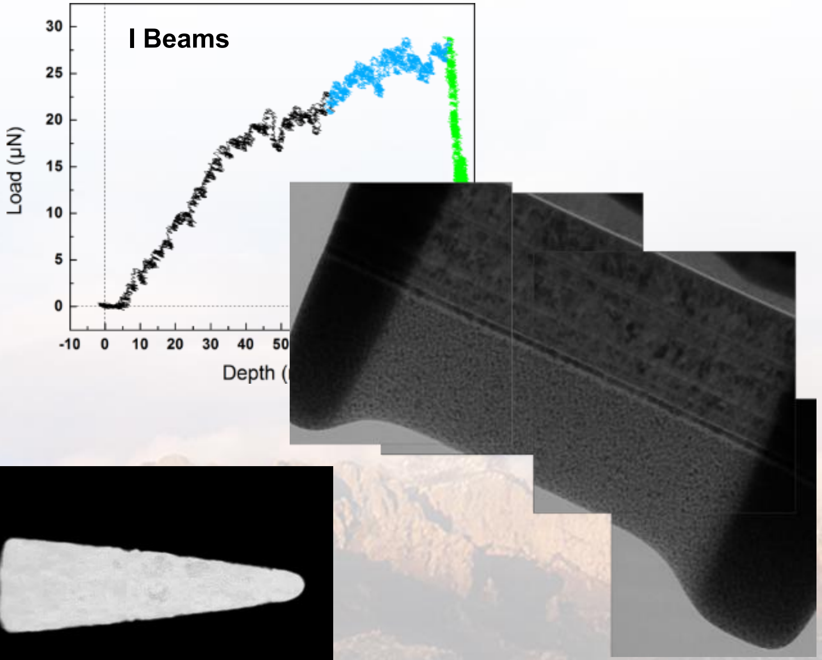
In situ Quantitative Mechanical Testing

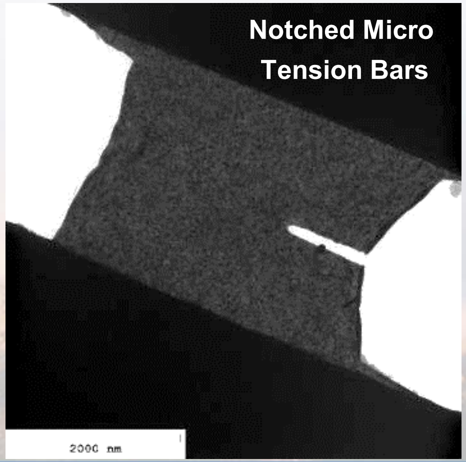
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

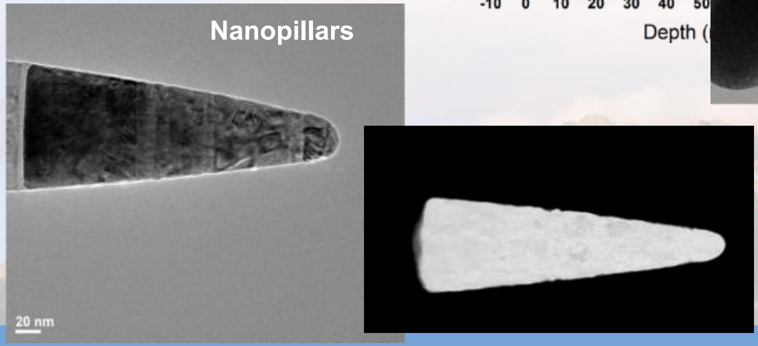
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

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



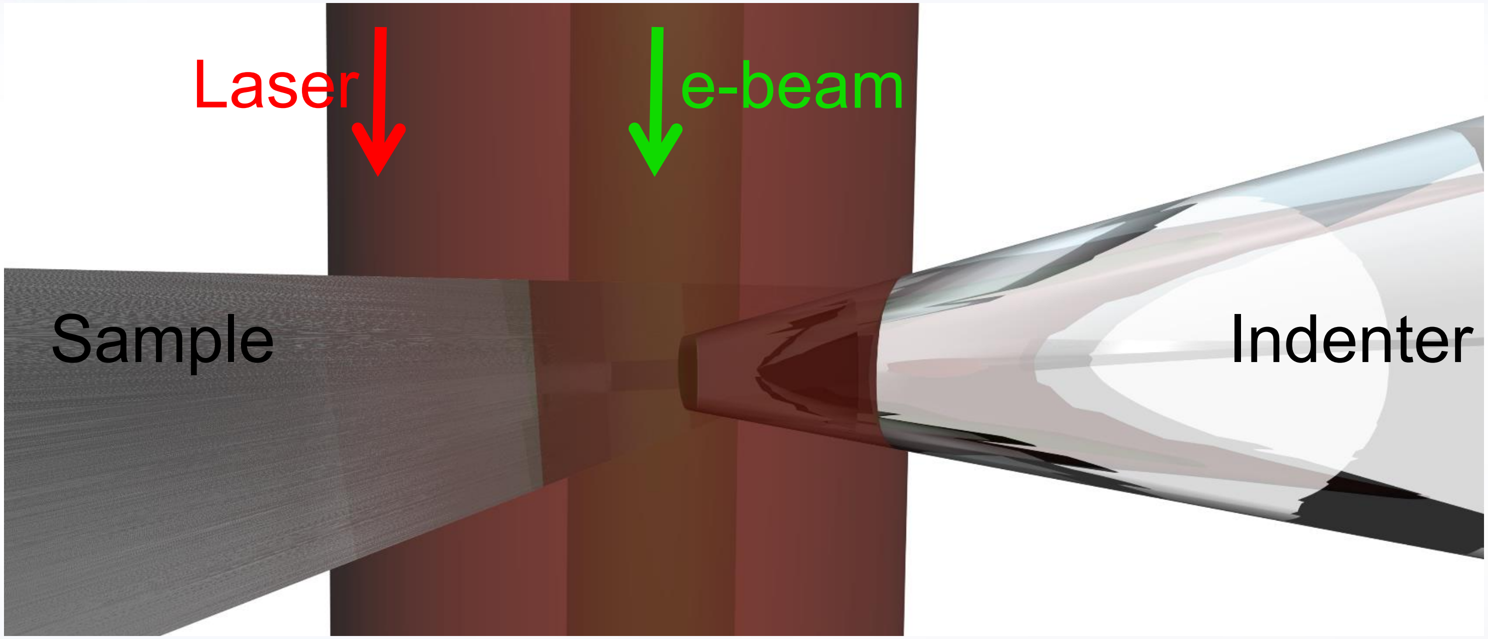


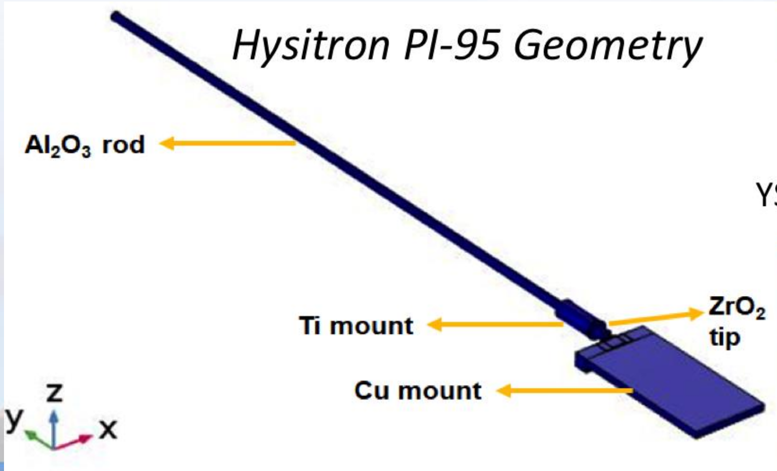


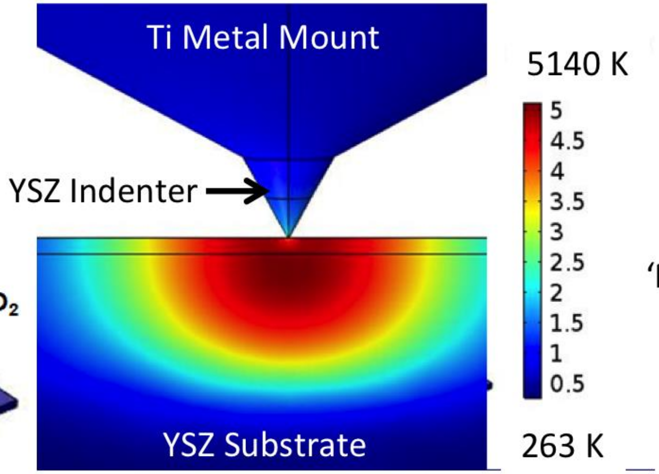


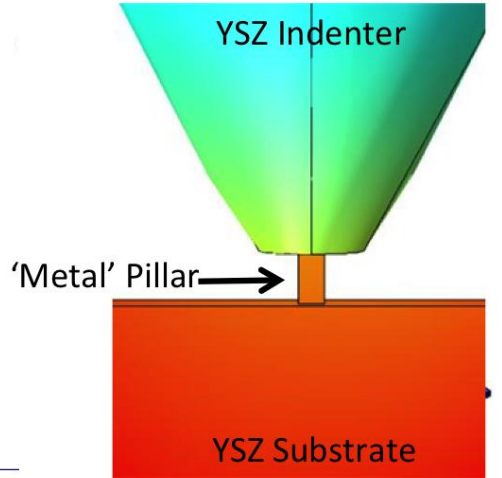
Can we Combine Laser Heating with Mechanical Testing?

Contributors: R.L. Grosso, E.N.S. Muccillo, D.N.F. Muche, G.S. Jawaharram, C.M. Barr, A.M. Monterrosa, R.H.R. Castro, S.J. Dillon





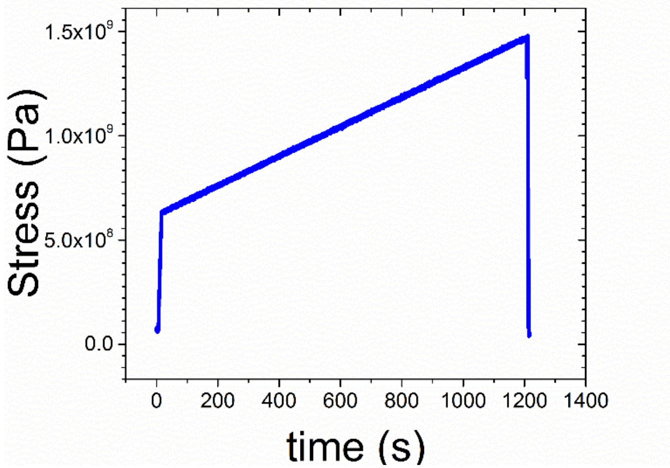




Irradiation Creep (4 MeV Cu^{3+} 10^{-2} DPA/s)

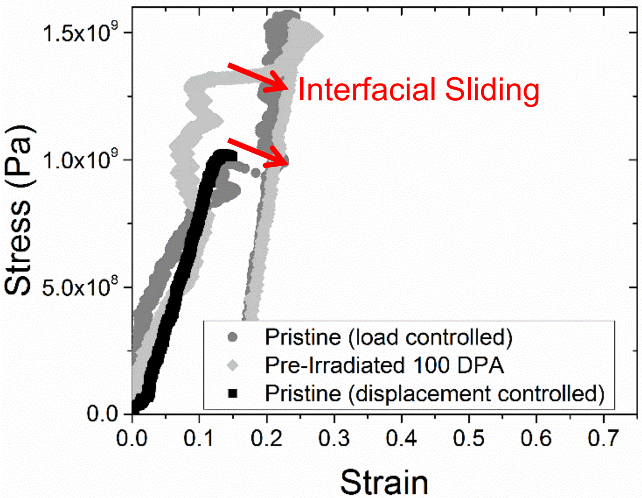
Contributors: G.S. Jawaharram, S. Dillon & R.S. Averback

Controlled Loading Rate Experiments

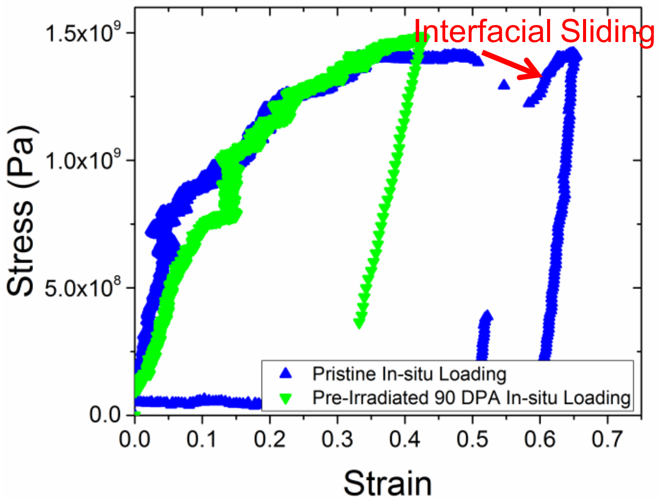


In-situ TEM radiation creep is feasible!

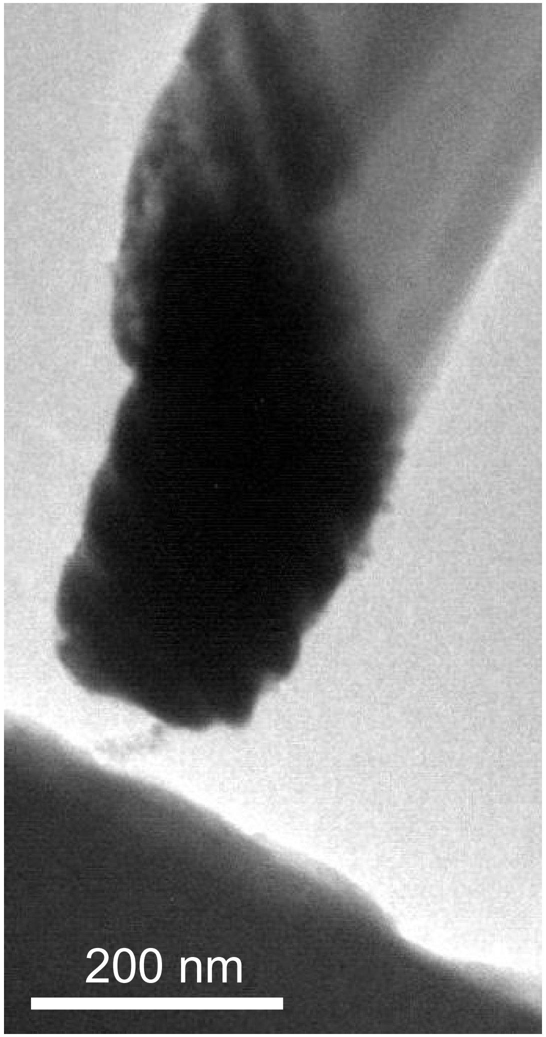
No Irradiation
(Loading rate 0.6 Mpa s^{-1})



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



50 nm Cu-W multilayer
20 Min

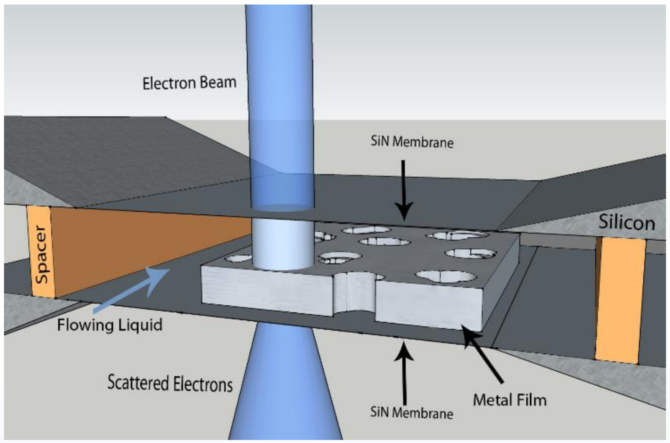


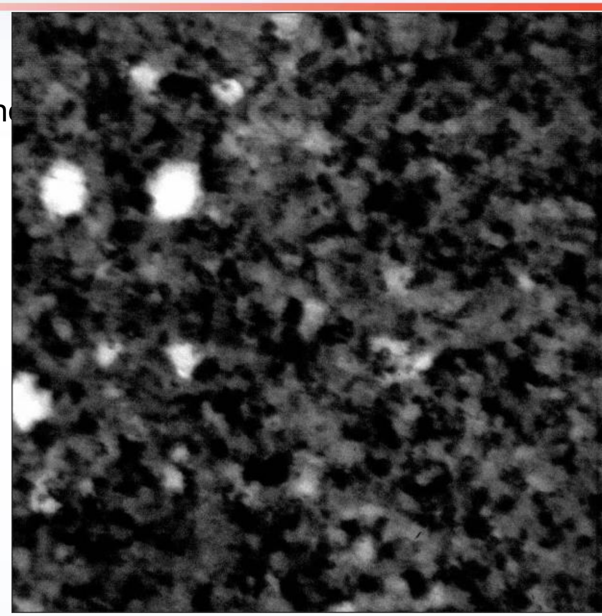
Can We Gain Insight into the Corrosion Process through *In situ* TEM?

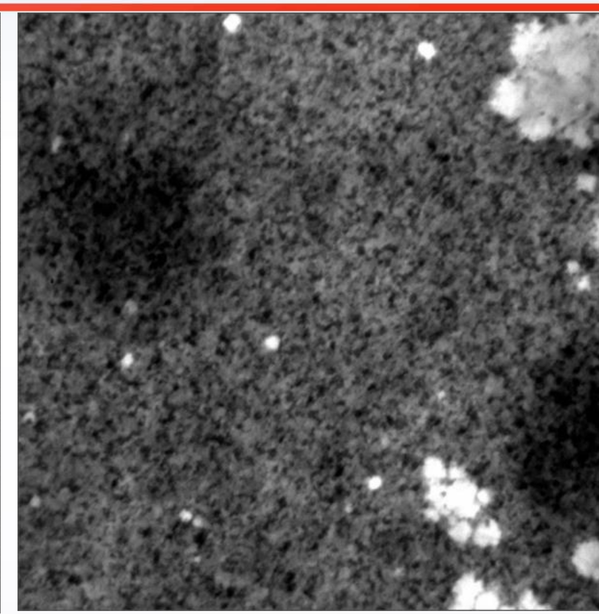
Contributors: D. Gross, J. Kacher, I.M. Robertson & Protochips, Inc.

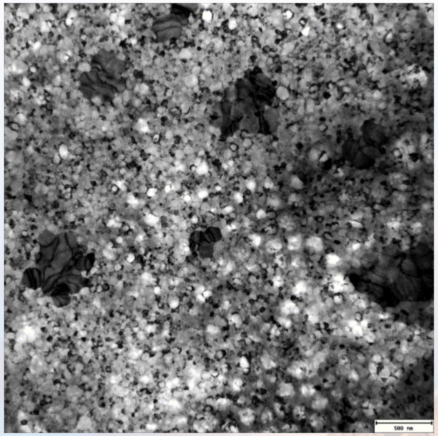
<u>Microfluidic Stage</u>

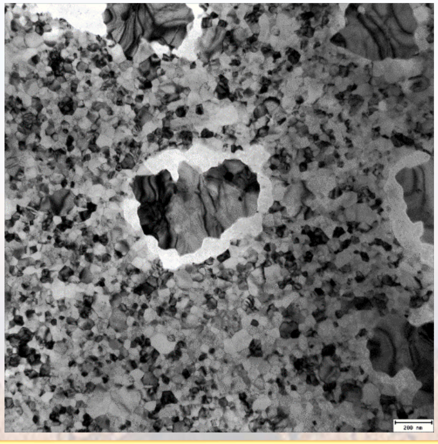
- Mixing of two or more channels
- Continuous observation of the reaction chann

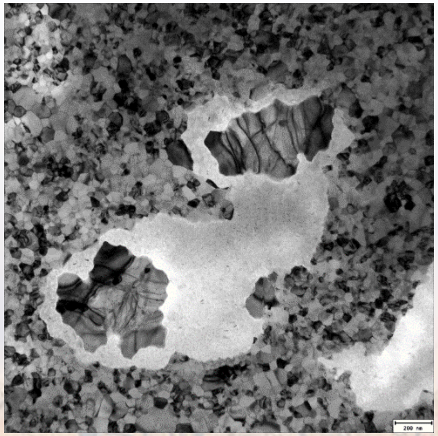


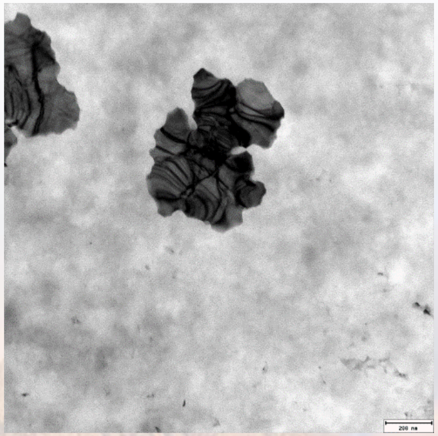












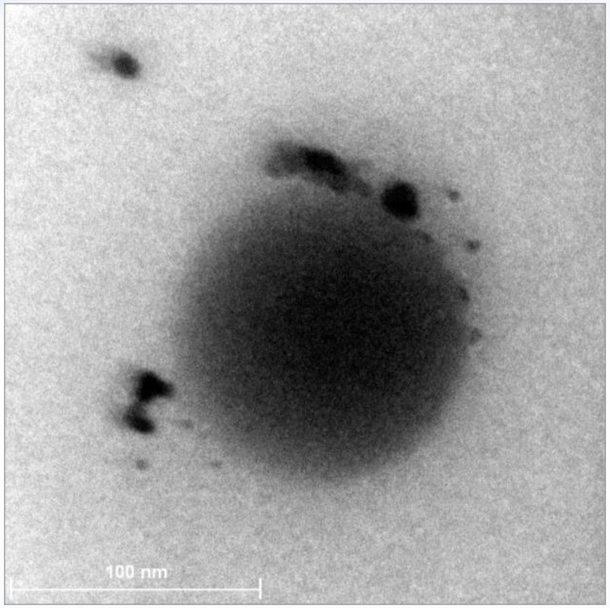
Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.
Large grains resulting from annealing appear more corrosion tolerant

Other Fun Uses of Microfluidic Cell

Protocell Drug Delivery

S. Hoppe,
E. Carnes,
J. Brinker

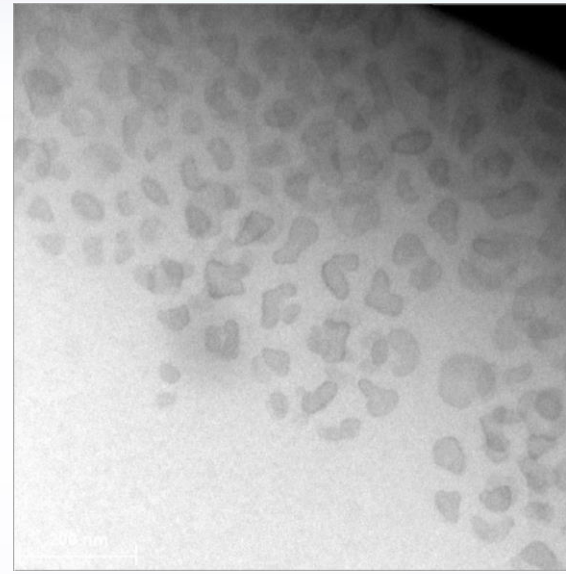
Liposome encapsulated Silica destroyed by the electron beam



BSA Crystallization

S. Hoppe

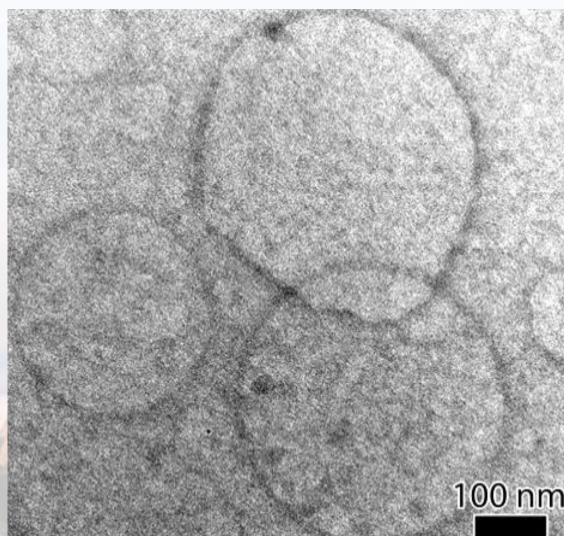
Crystallization of excess Bovine Serum Albumen during flow



Liposomes in Water

S. Hoppe,
D. Sasaki

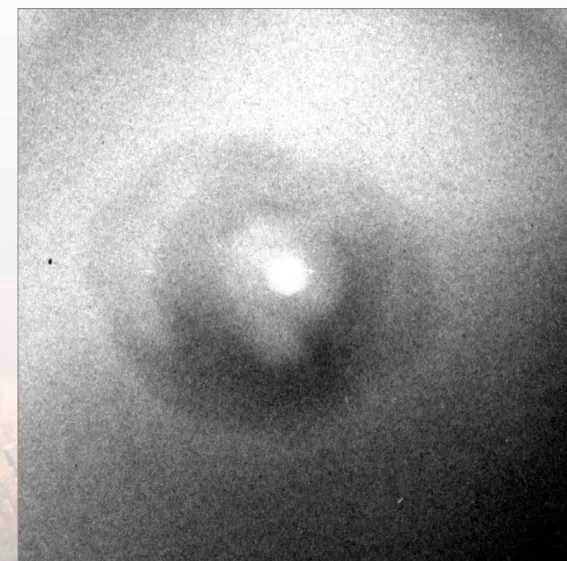
Liposomes imaged in flowing aqueous channel



La Structure Formation

S. Hoppe,
T. Nenoff

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

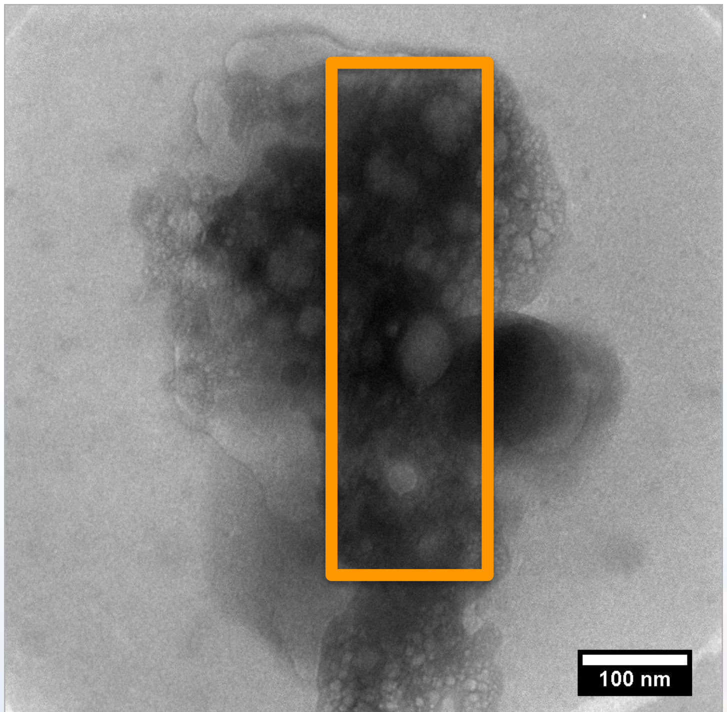


ACOM in Liquid Cell Environment

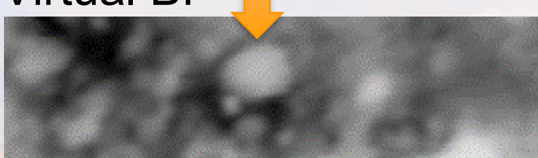
Contributors: C. Taylor, S. Pratt, & T. Nenoff

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

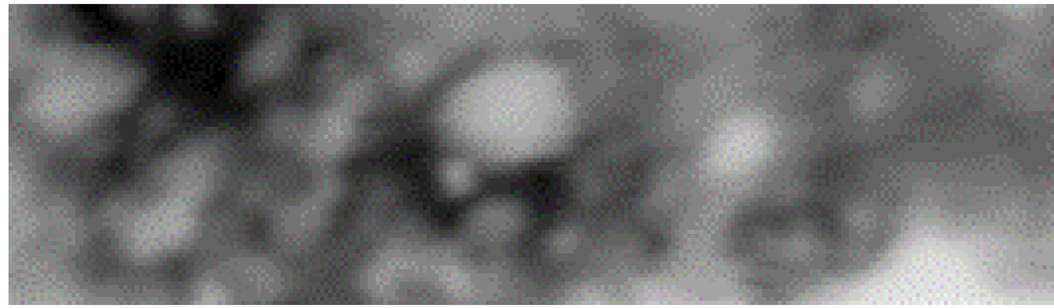
TEM Image



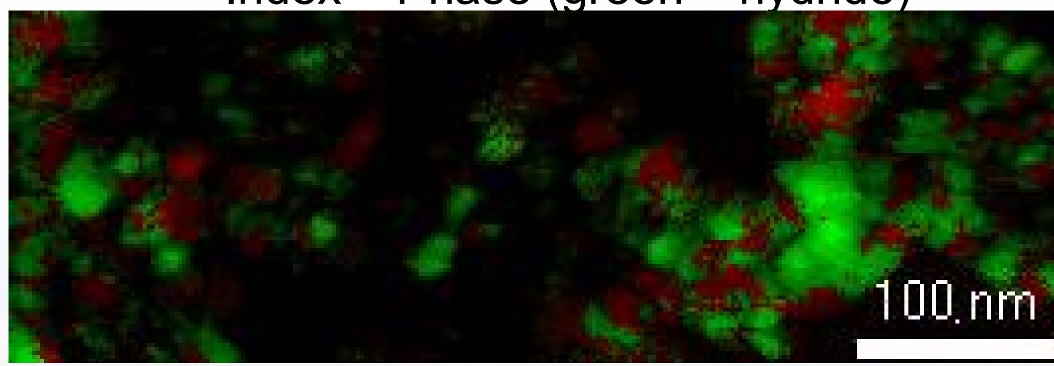
Virtual BF



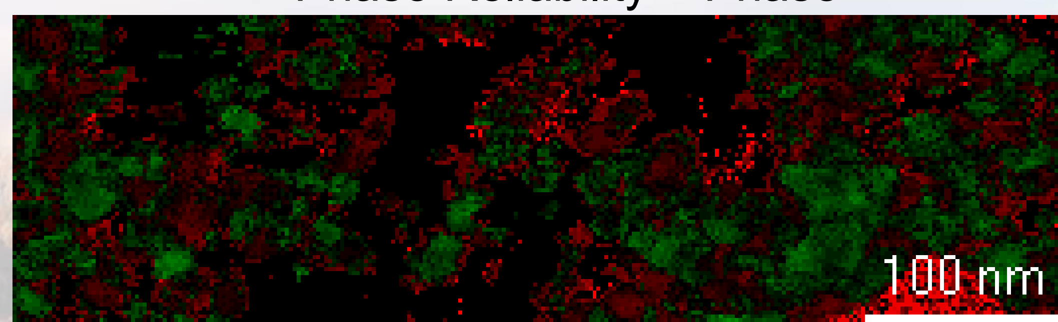
Virtual BF



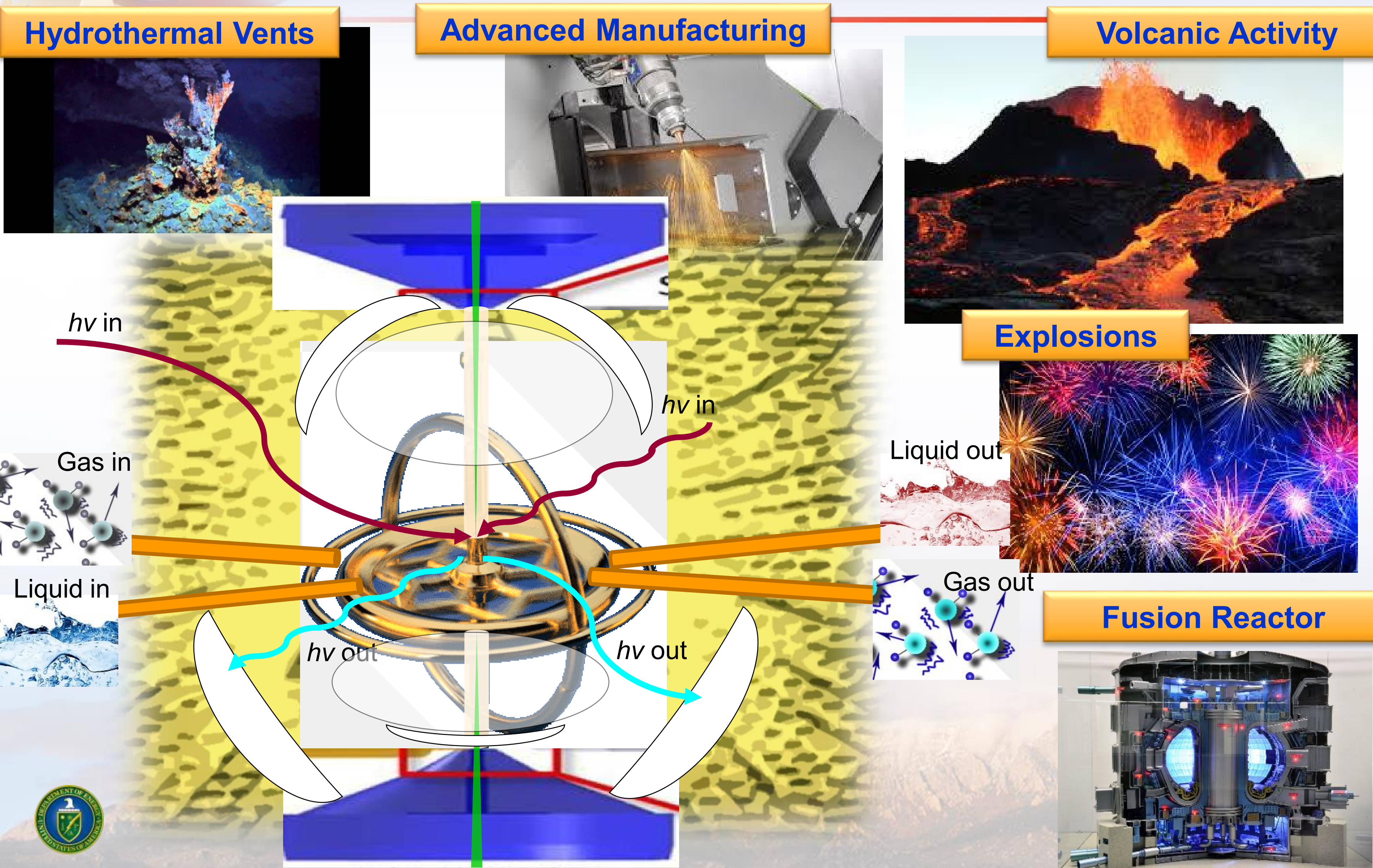
Index + Phase (green = hydride)



Phase Reliability + Phase

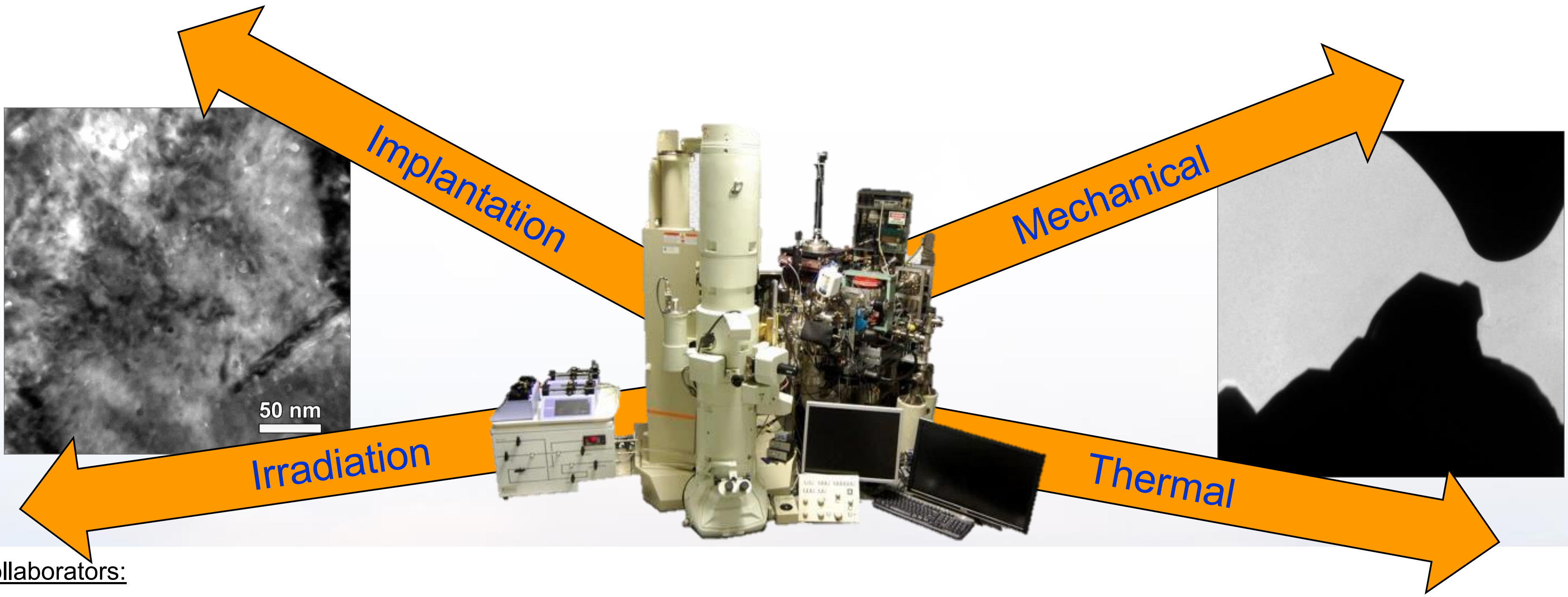


The Dream:
Testing Greater Extremes in the TEM



What is the Physical Limit of Coupled *In situ* Microscopy Experiments?

Khalid Hattar
Sandia National Laboratories, Albuquerque, NM 87185, USA



Collaborators:

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

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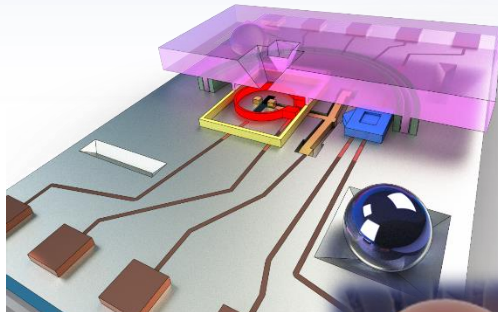
Sandia's USER Capabilities

D. Hanson, W. Martin, M. Wasiolek

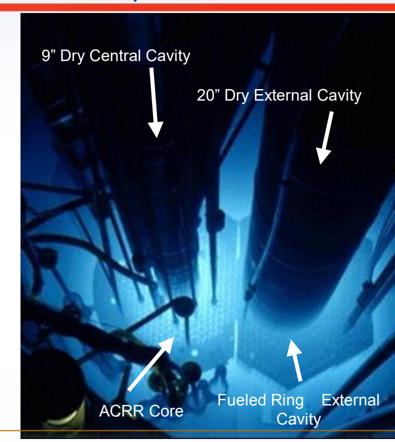
www.cint.lanl.gov

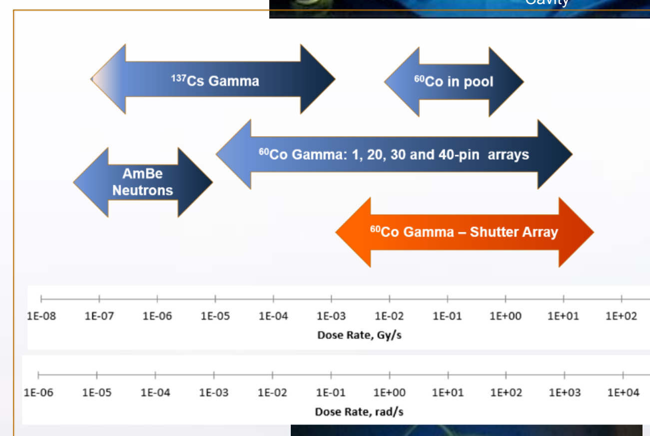
- Spring and Fall proposals for 18 months
- Rapid Access proposal anytime for 3 months



www.nsuf.inl.gov

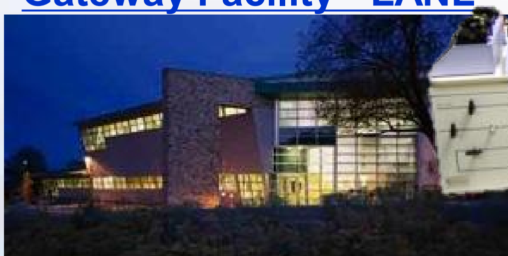
- Three proposal a year for 9 months

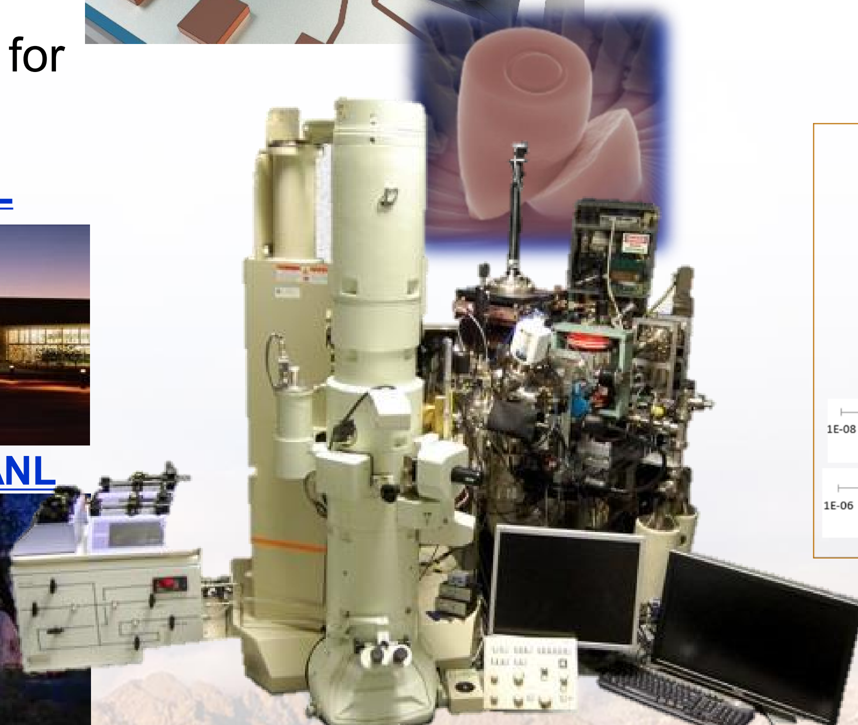


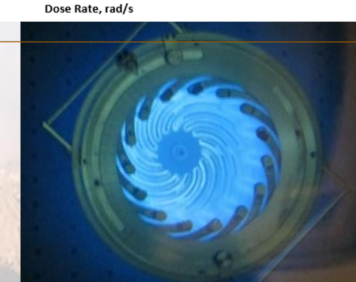


Core Facility - SNL

Gateway Facility - LANL







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