SAND2022-12055C

Exploring Phase and Microstructural Stability through *In situ* SEM and TEM

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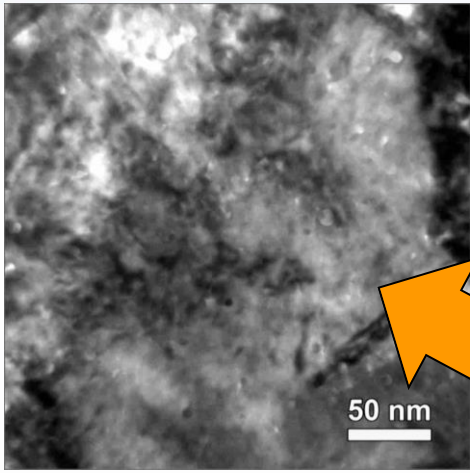
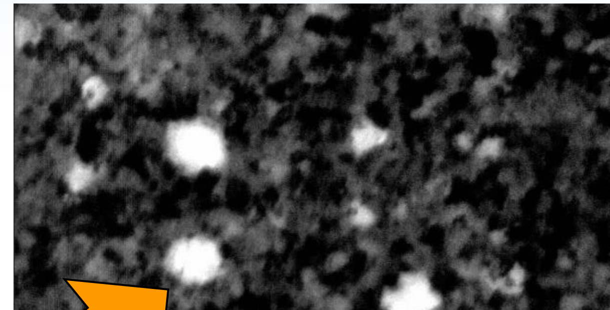
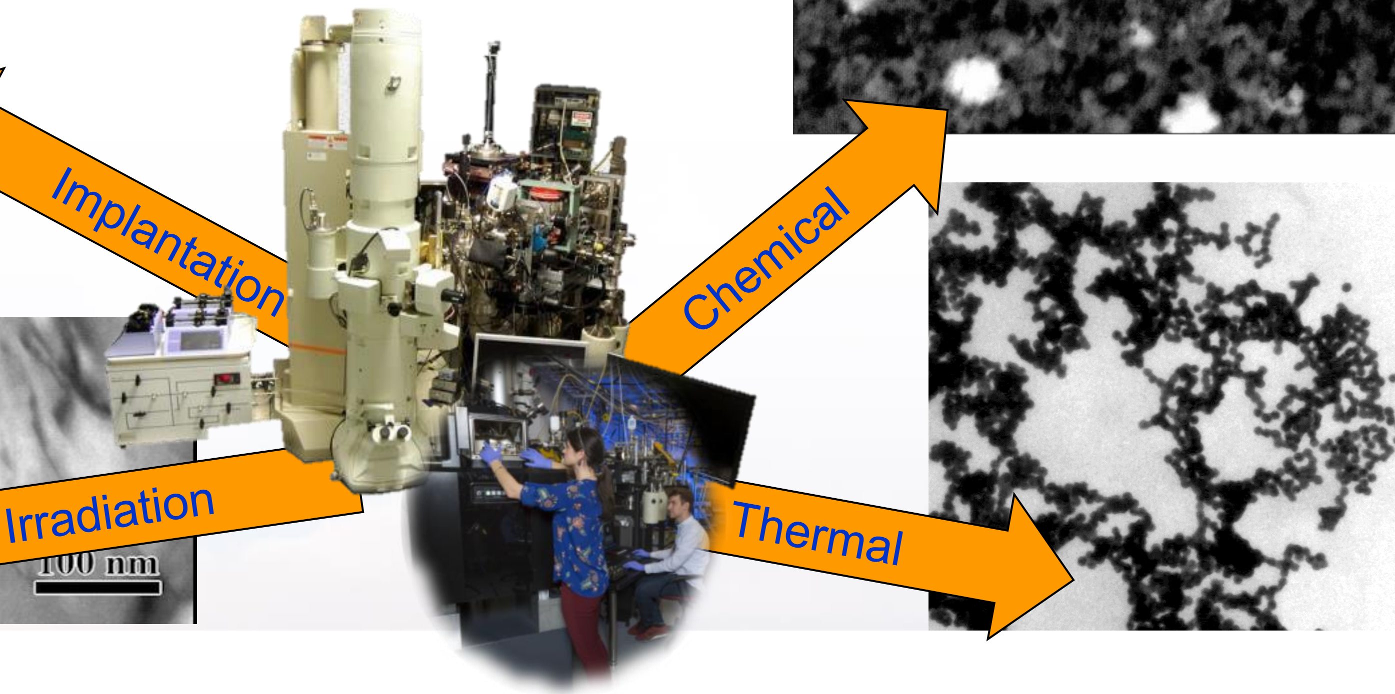
Sandia National Laboratories
University of New Mexico







Collaborators:

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

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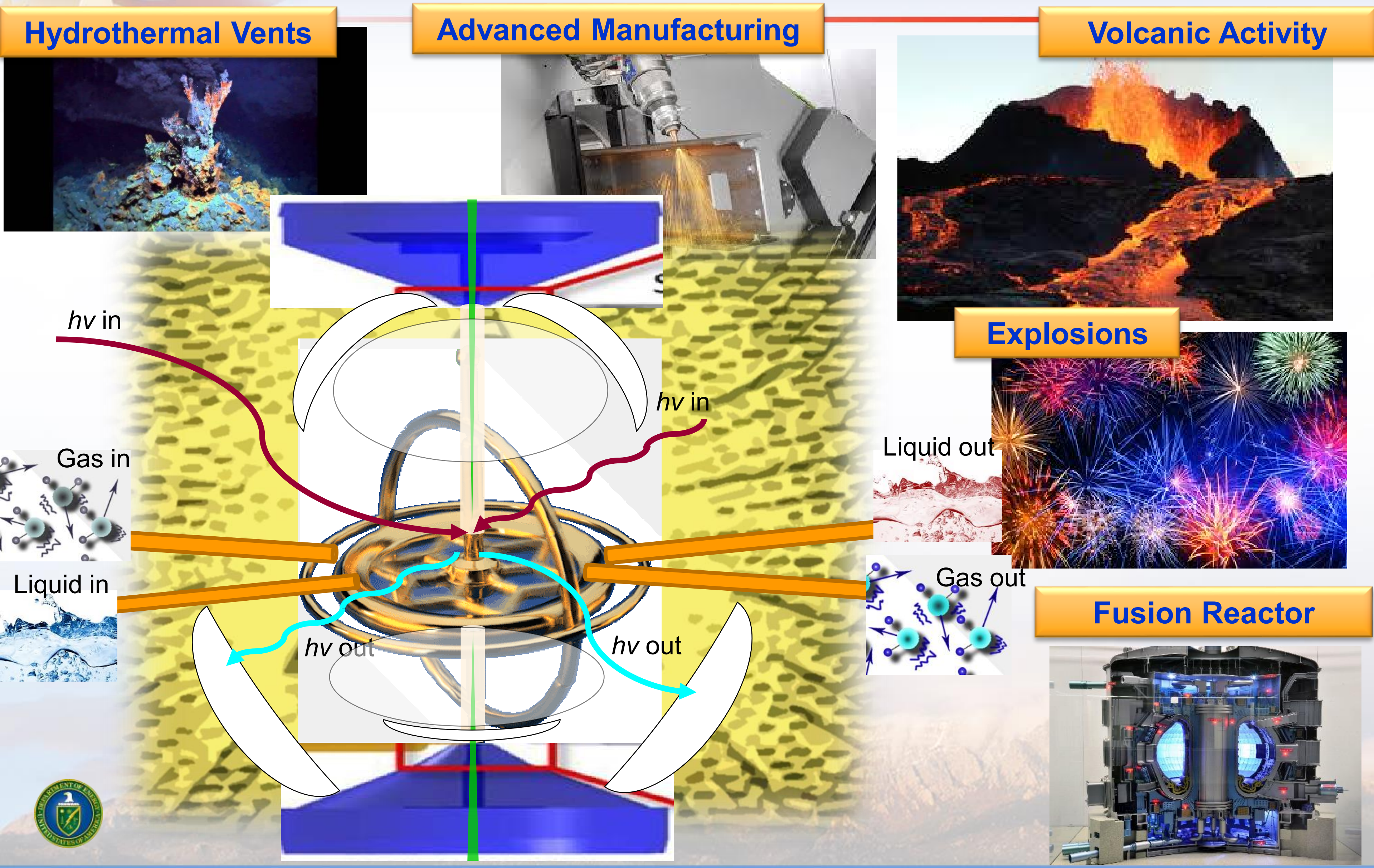
Testing Greater Extremes in the TEM



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)

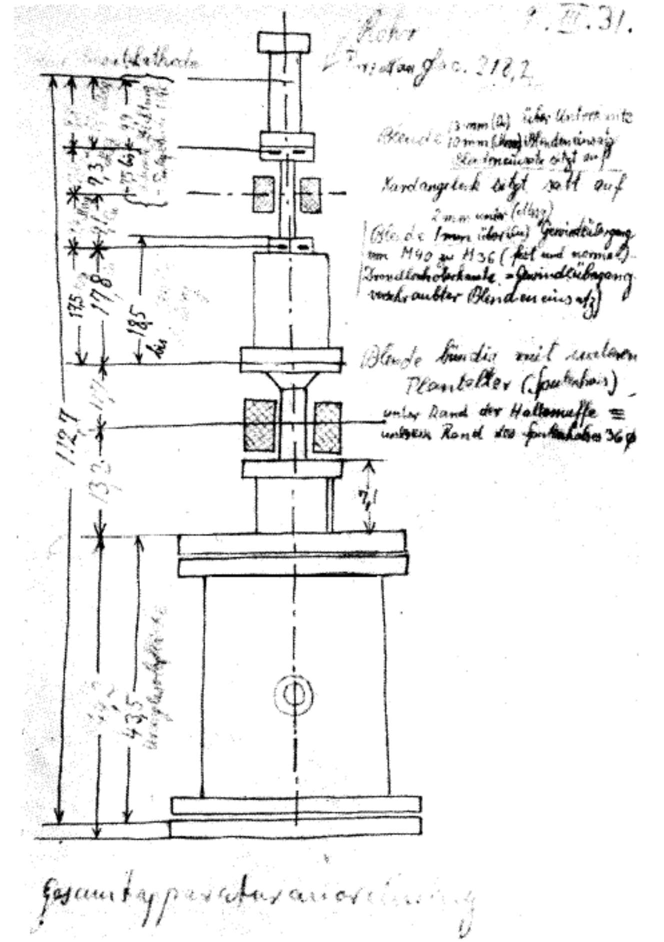
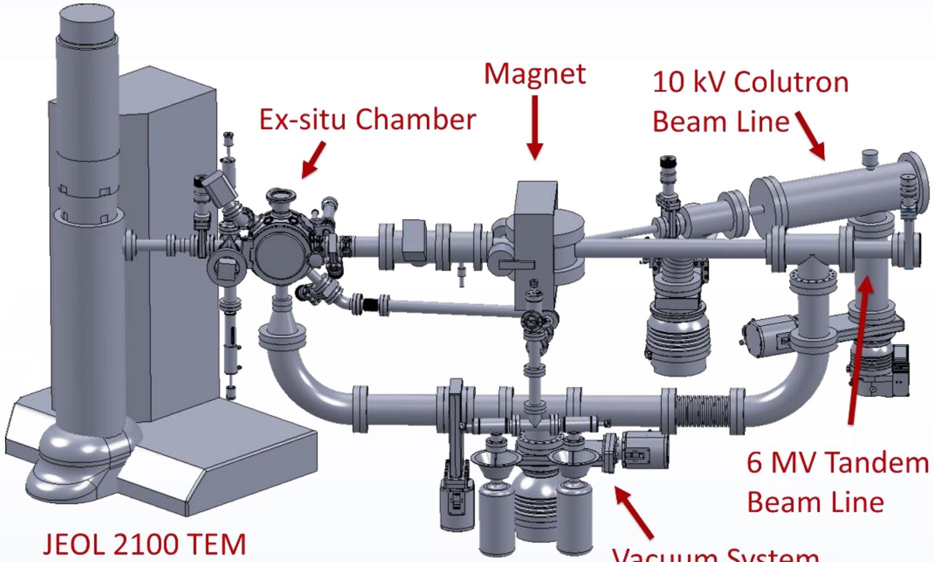


Fig. 2: Sketch by the author (9 March 1931) of the cathode ray tube for testing one-stage and two-stage electron-optical imaging by means of two magnetic electron lenses (electron microscope) [8].

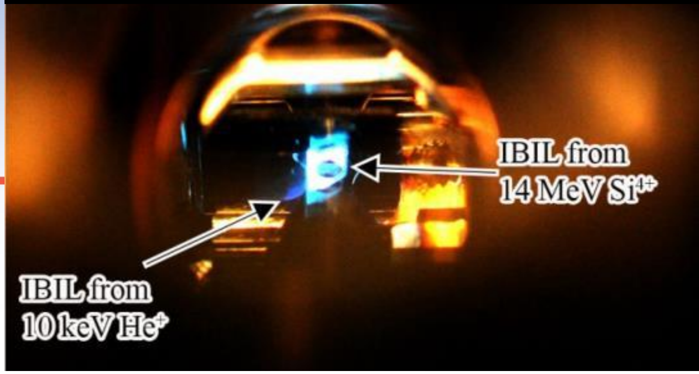
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

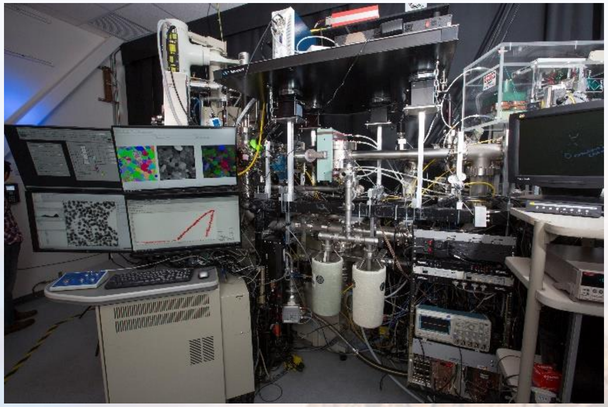
Collaborator: D.L. Buller

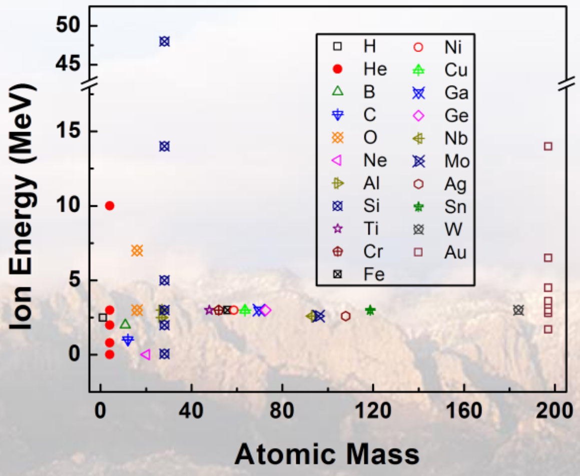
10 kV Colutron - 200 kV TEM - 6 MV Tandem

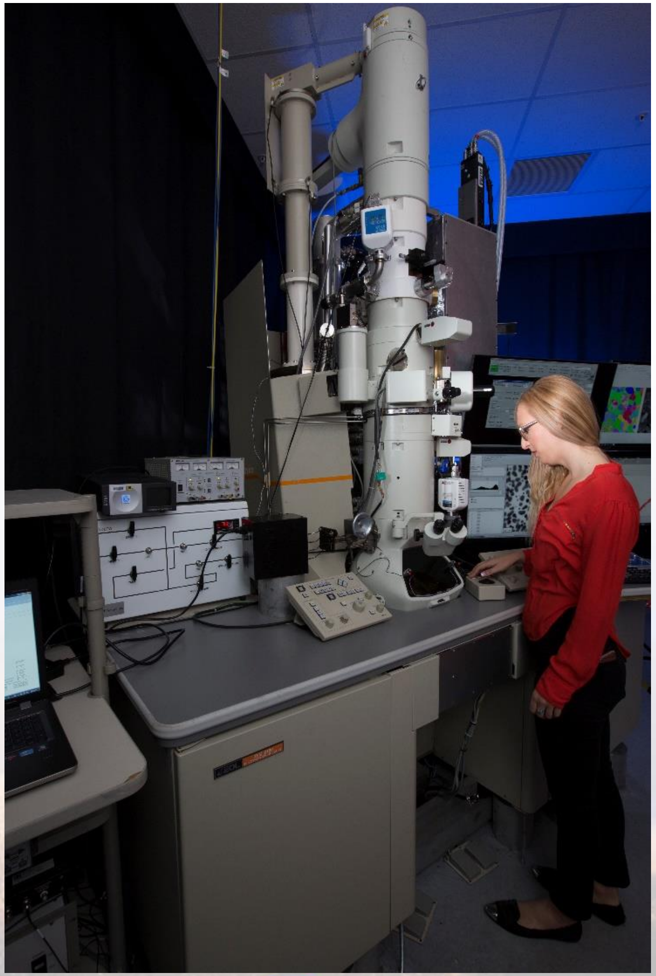


IBIL from a quartz stage inside the TEM



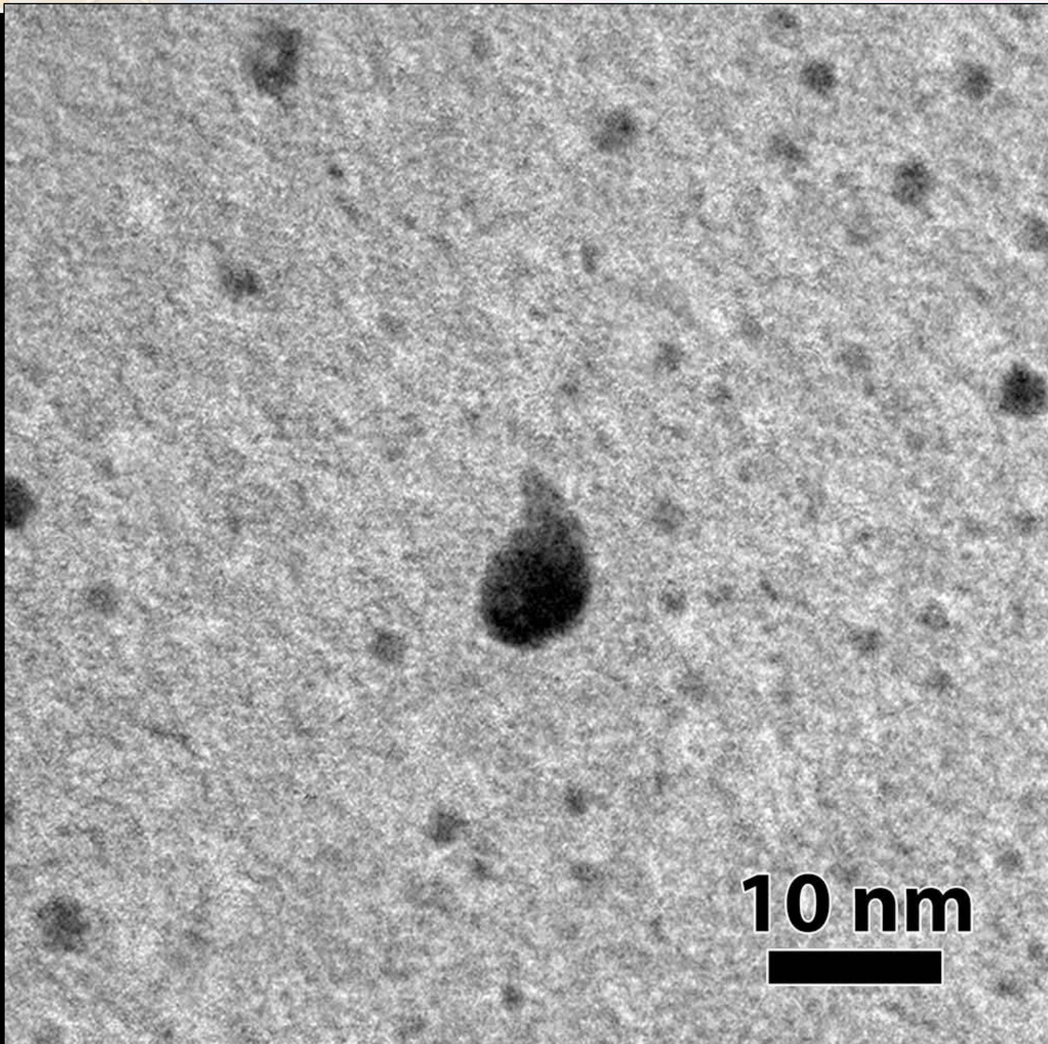


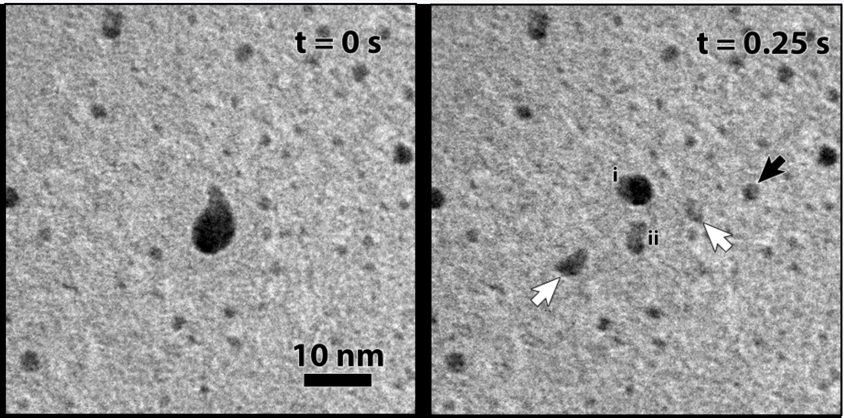


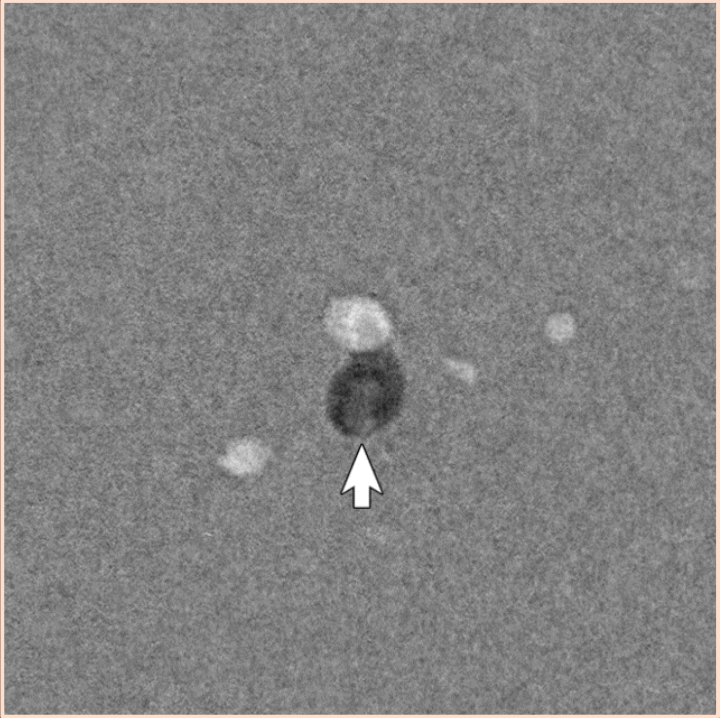


Single Ion Strikes:
46 keV Au^{1-} ions into 5 nm Au nanoparticles

Collaborator: D.C. Bufford



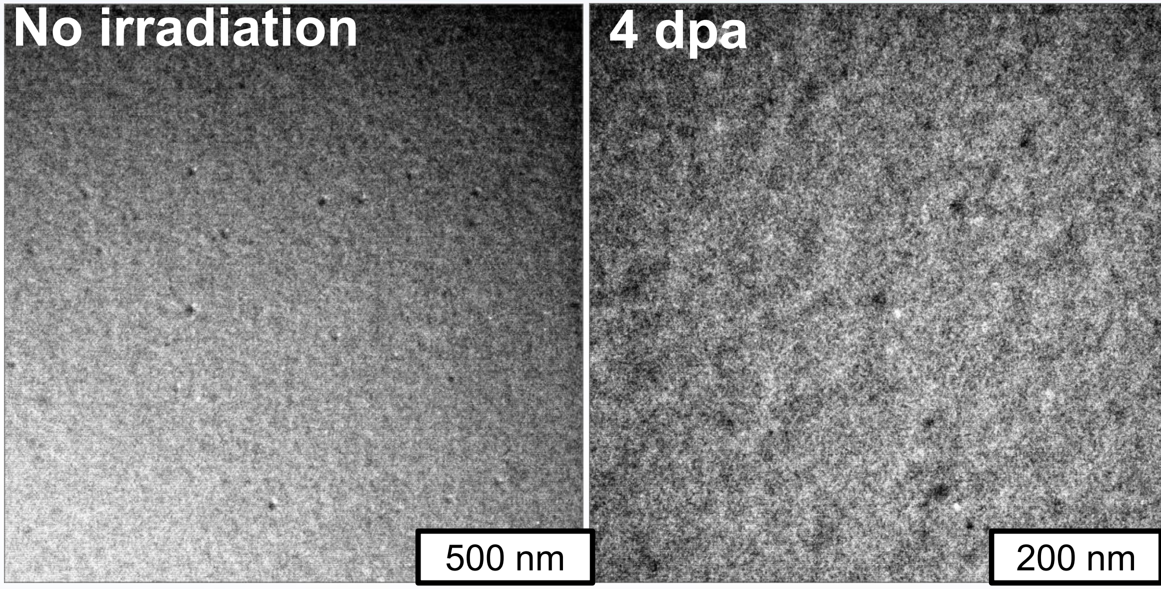




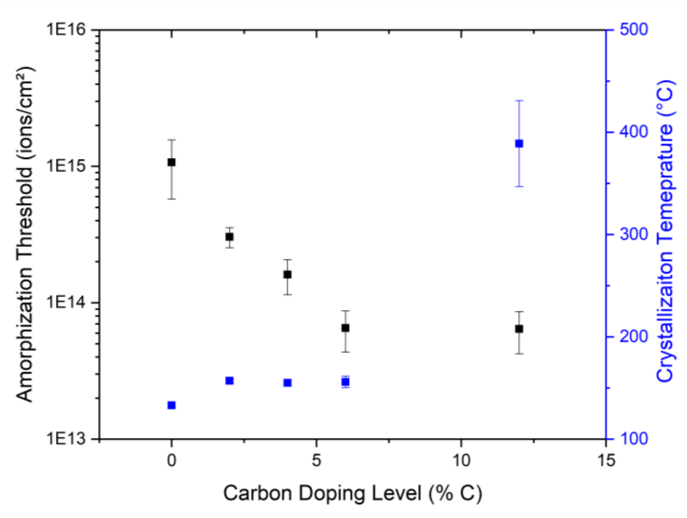
Radiation Tolerance in Phase Change Memory

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

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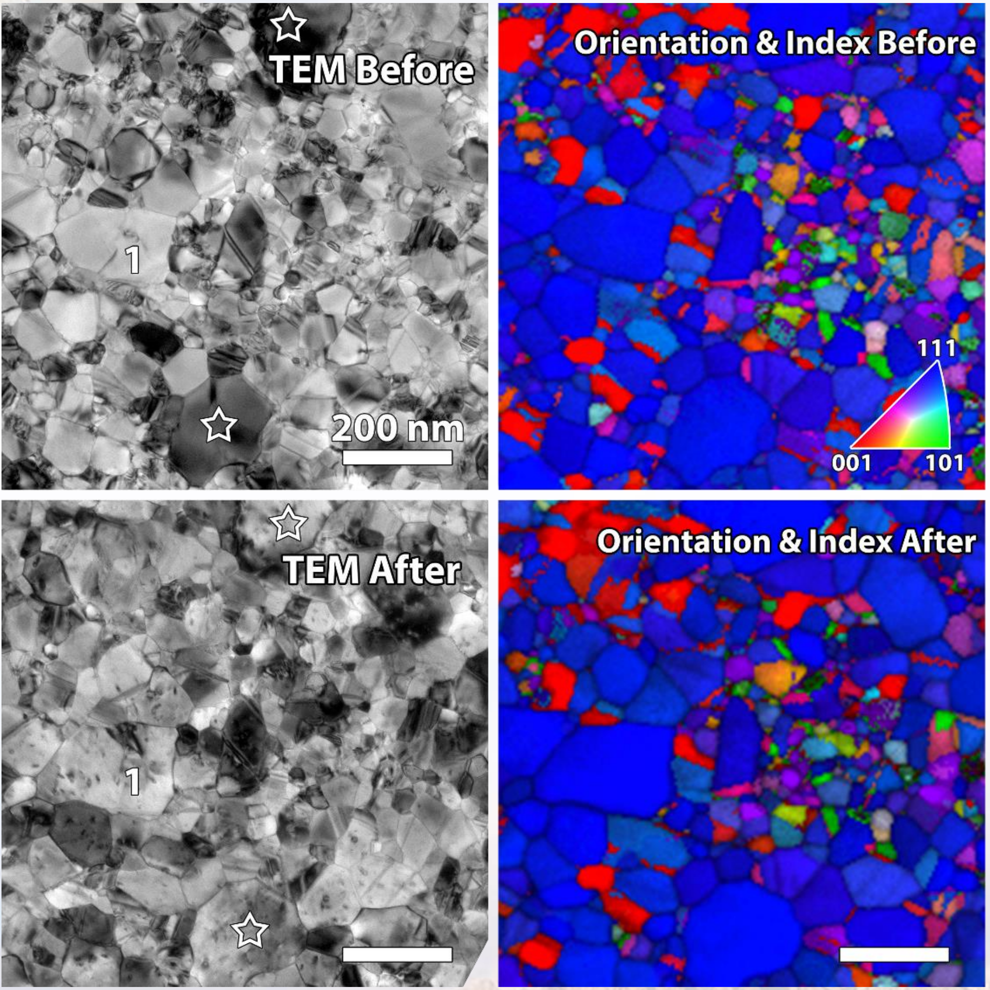
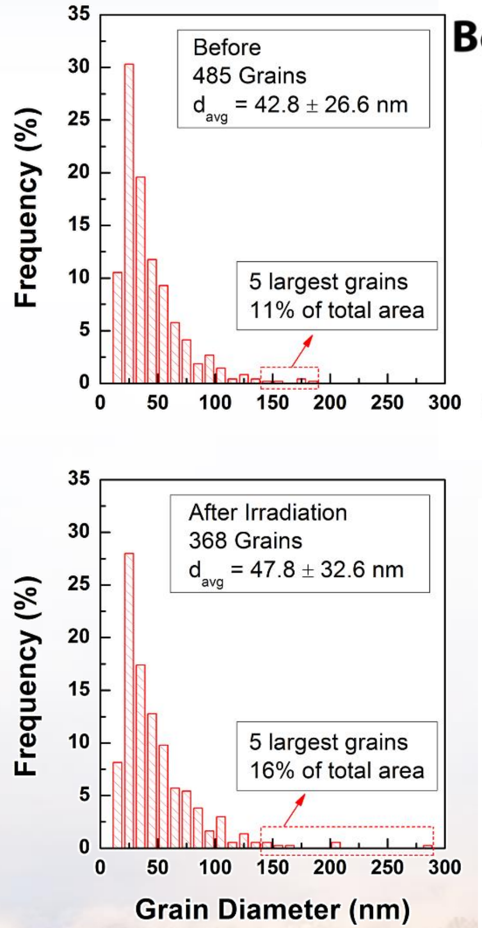
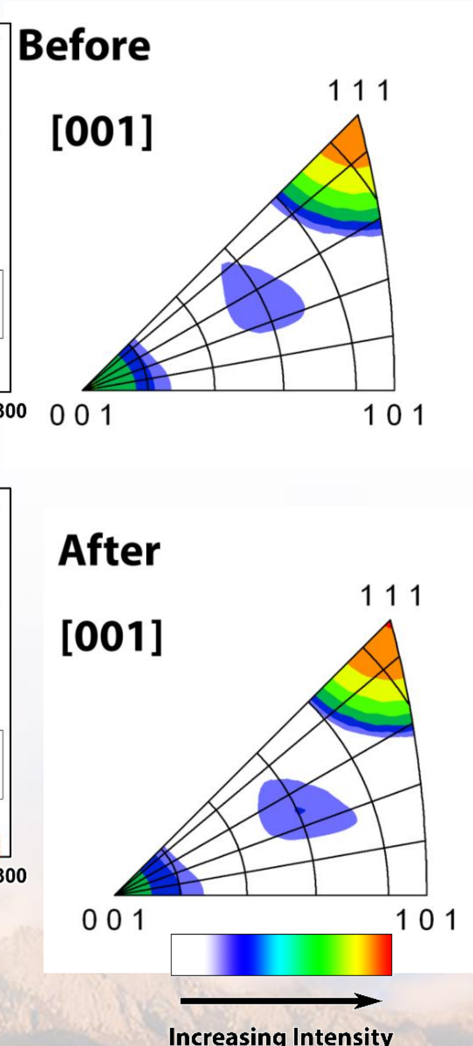
Quantifying Grain Boundary Radiation Stability of Nanocrystalline Au

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





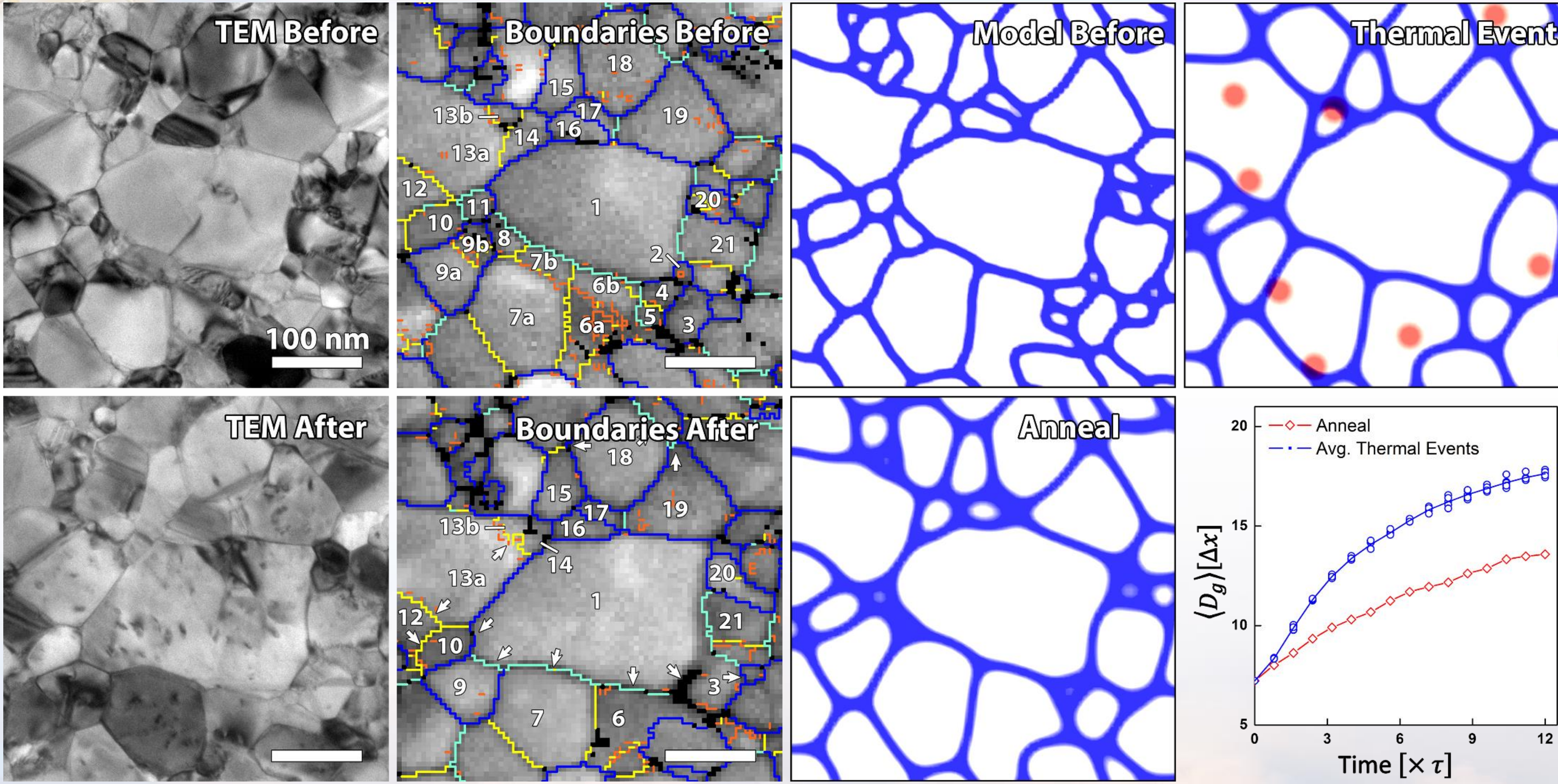


10 MeV Si

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

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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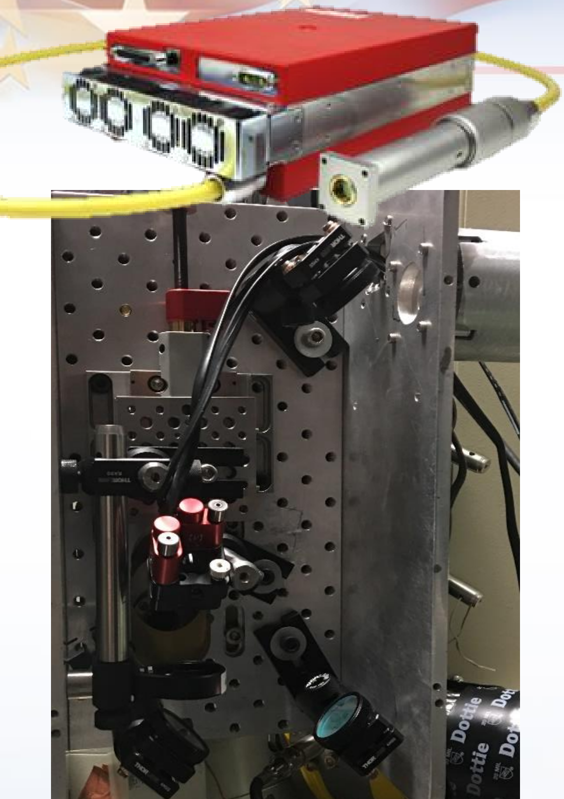
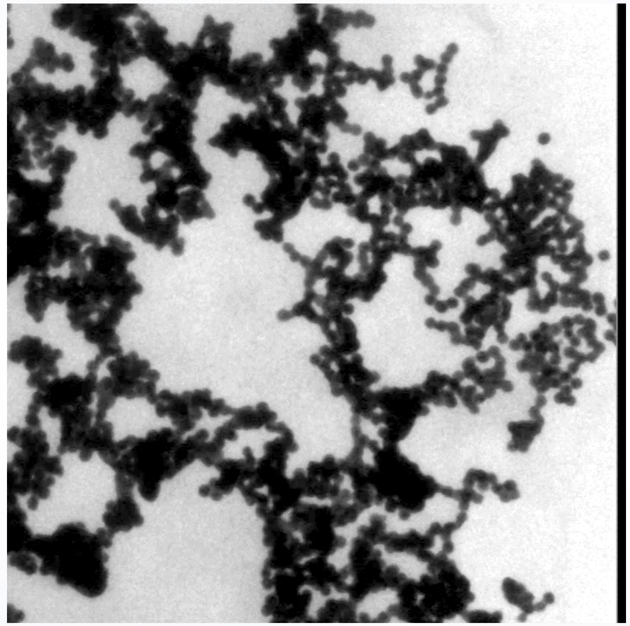
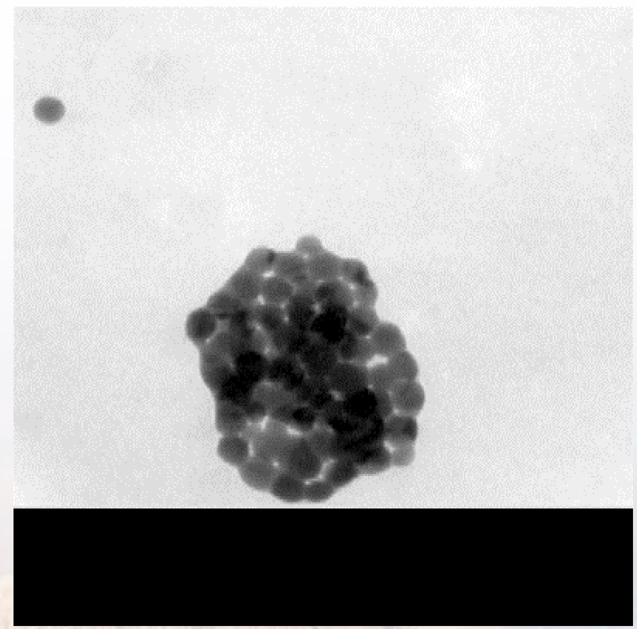
Initial Laser Heating Observations

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





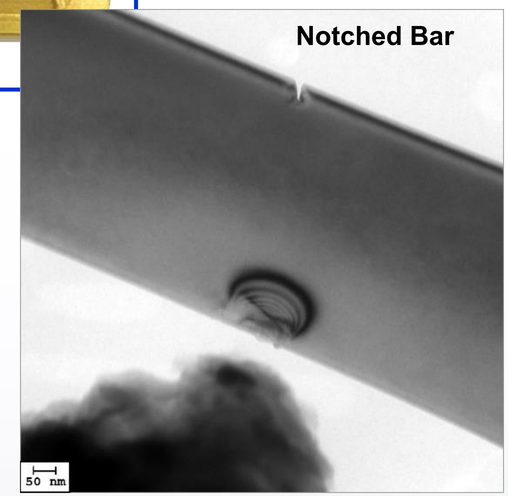
A Complex Combination of Sintering, Reactions, and Ablation Occurs



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

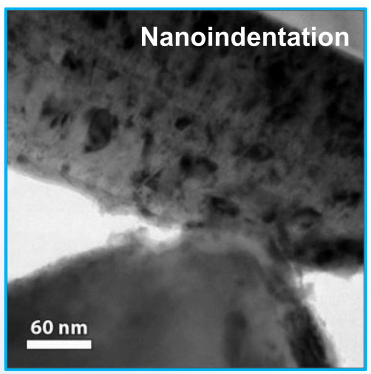
In situ Quantitative Mechanical Testing

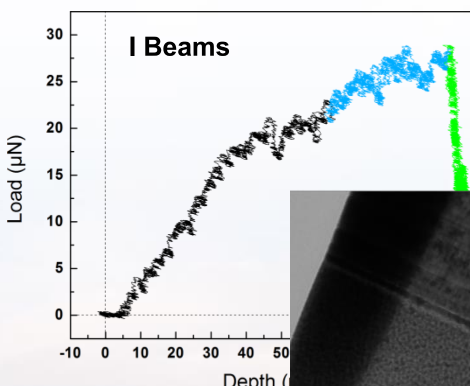
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

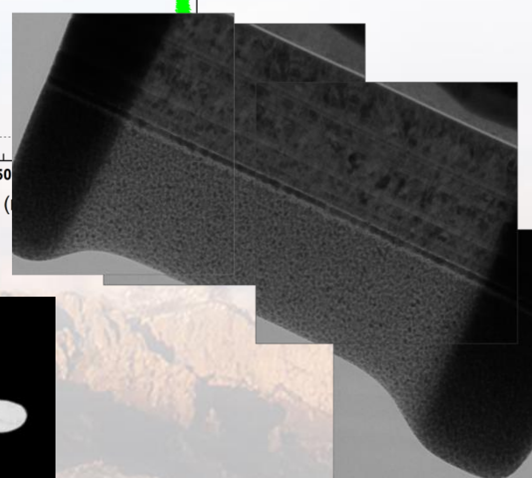


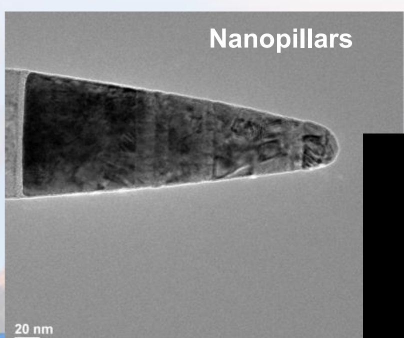
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

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



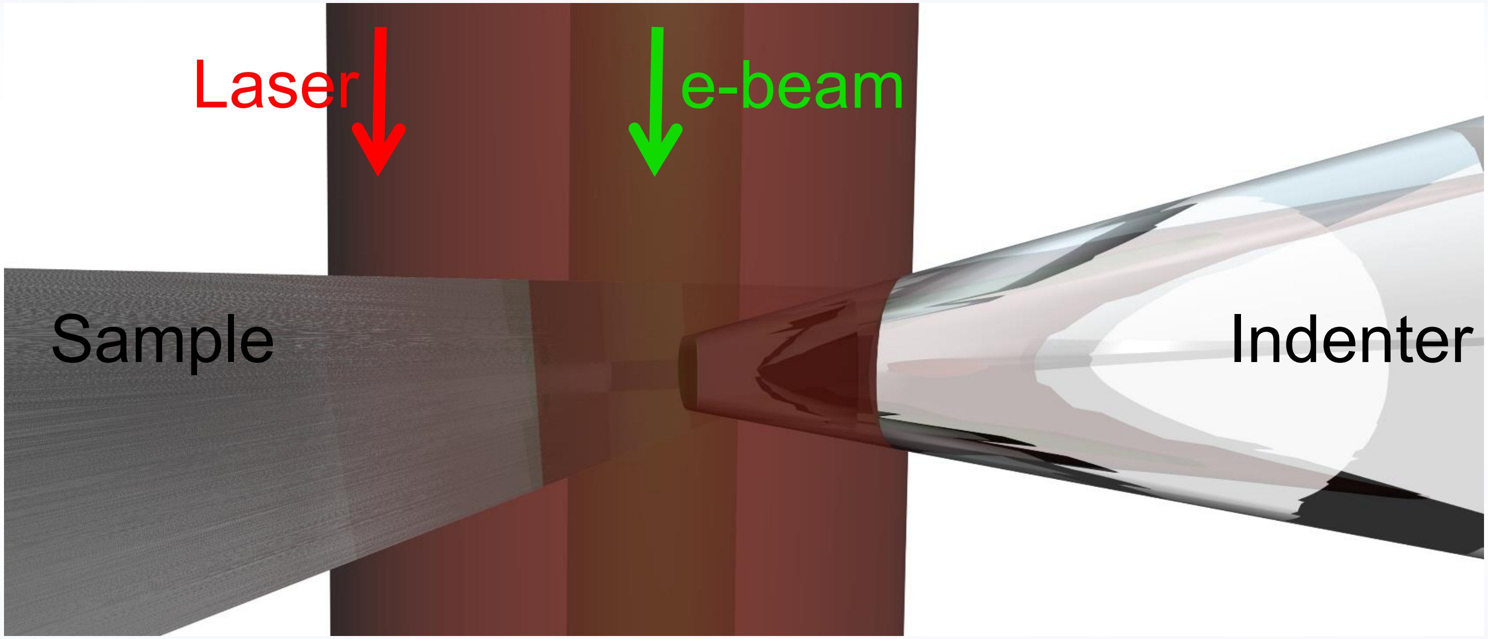


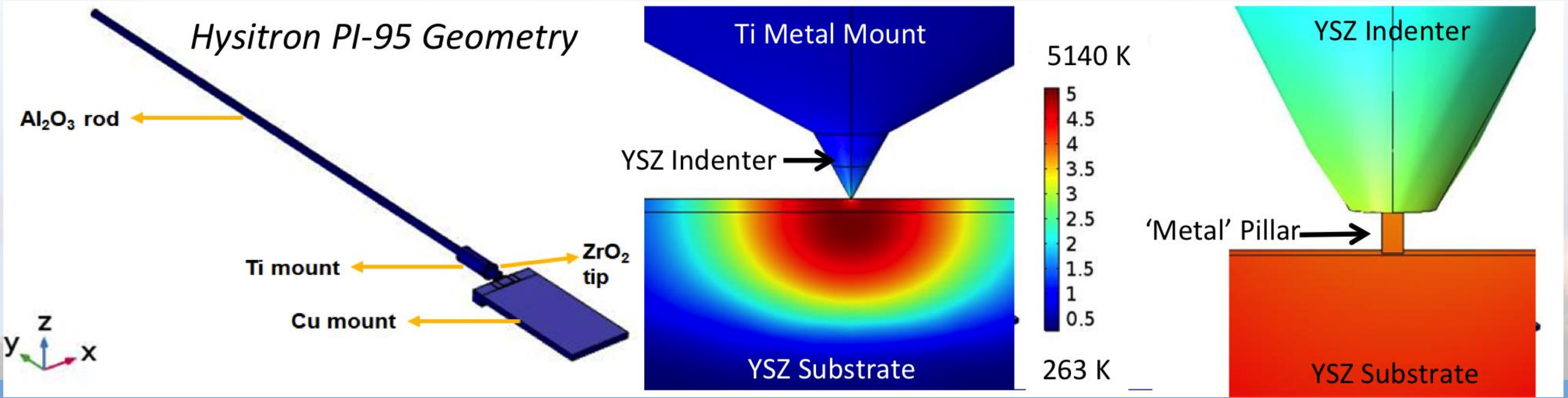




Can we Combine Laser Heating with Mechanical Testing?

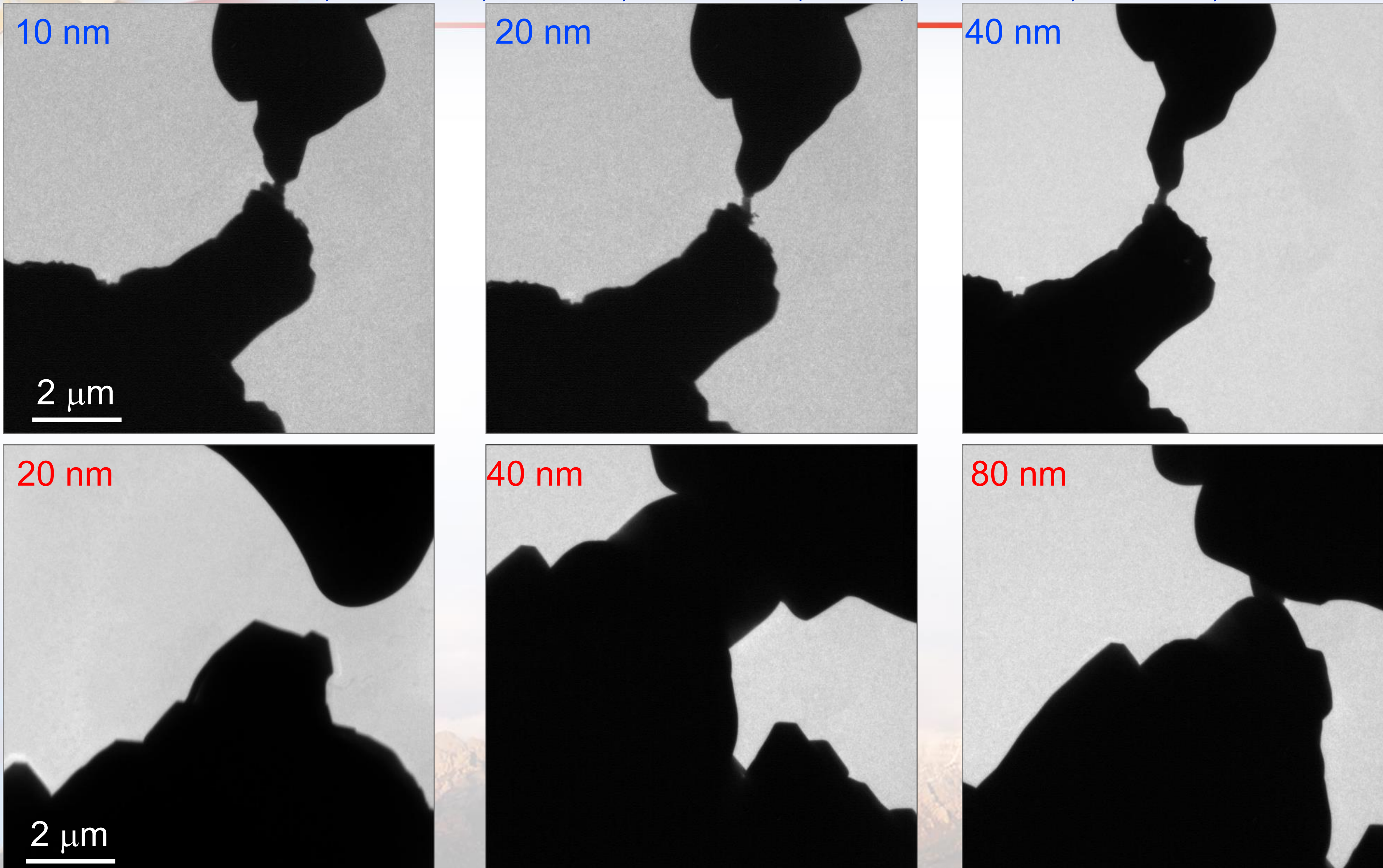
Contributors: R.L. Grosso, E.N.S. Muccillo, D.N.F. Muche, G.S. Jawaharram, C.M. Barr, A.M. Monterrosa, R.H.R. Castro, S.J. Dillon





Pushing the Laser Limit - 1604 ^{o}C and 2056 ^{o}C ScSZ-ScSZ

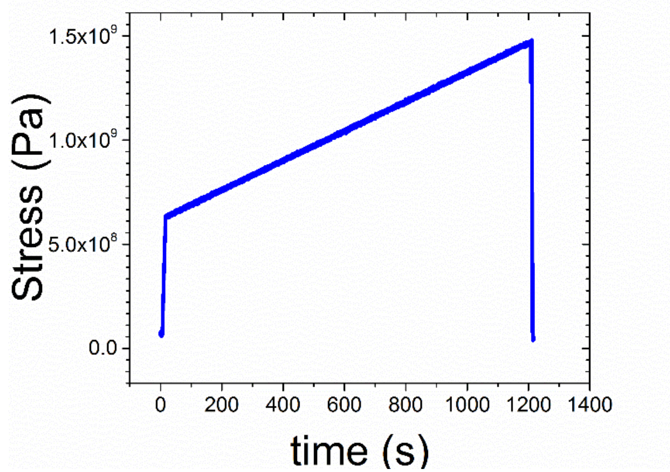
Contributors: R.L. Grosso, E.N.S. Muccillo, D.N.F. Muche, G.S. Jawaharram, C.M. Barr, A.M. Monterrosa, R.H.R. Castro, S.J. Dillon



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

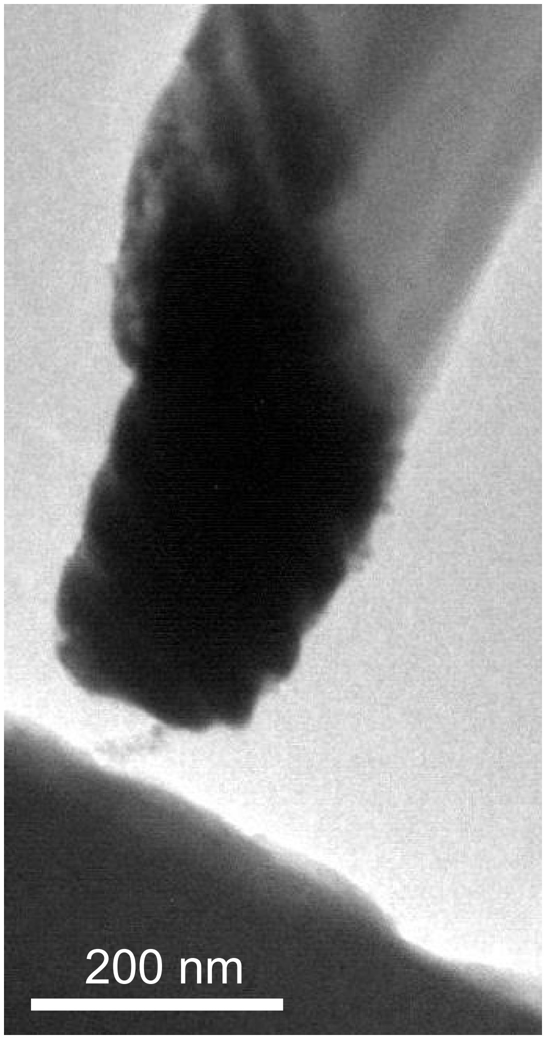
Contributors: G.S. Jawaharram, S. Dillon & R.S. Averback

Controlled Loading Rate Experiments

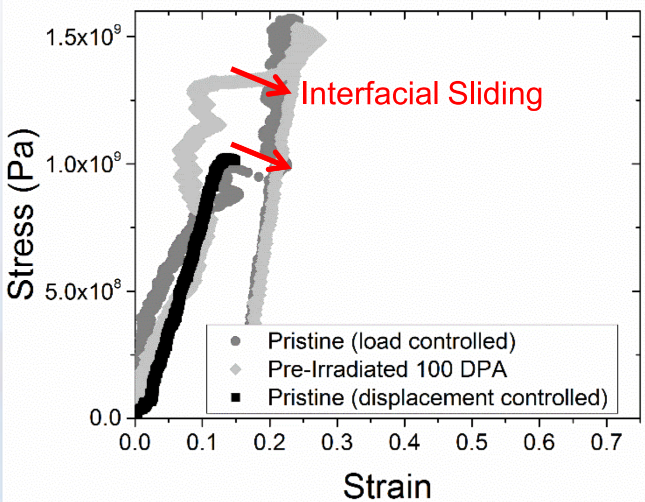


In-situ TEM radiation creep is feasible!

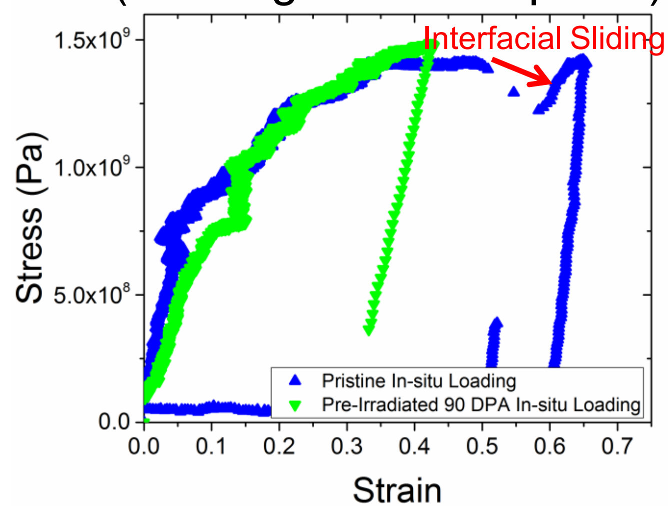
50 nm Cu-W multilayer
20 Min



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



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

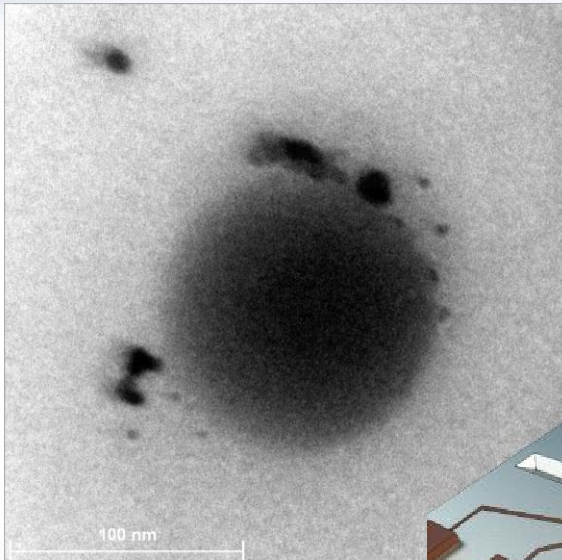


Phase Stability in Microfluidic Environment

Protocell Drug Delivery

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

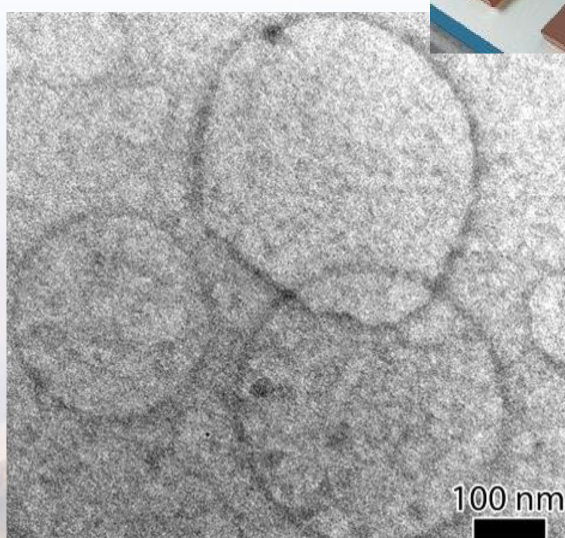
Liposome encapsulated Silica destroyed by the electron beam

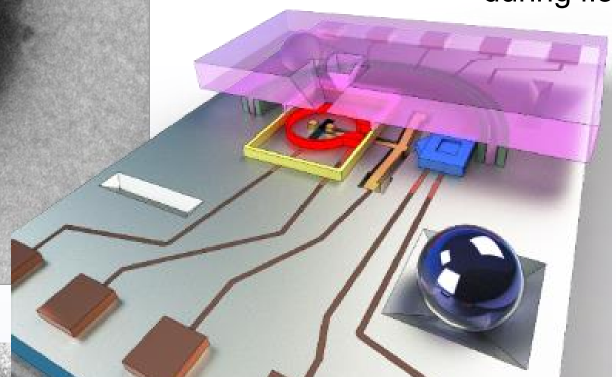


Liposomes in Water

S.H. Pratt,
D. Sasaki

Liposomes imaged in flowing aqueous channel

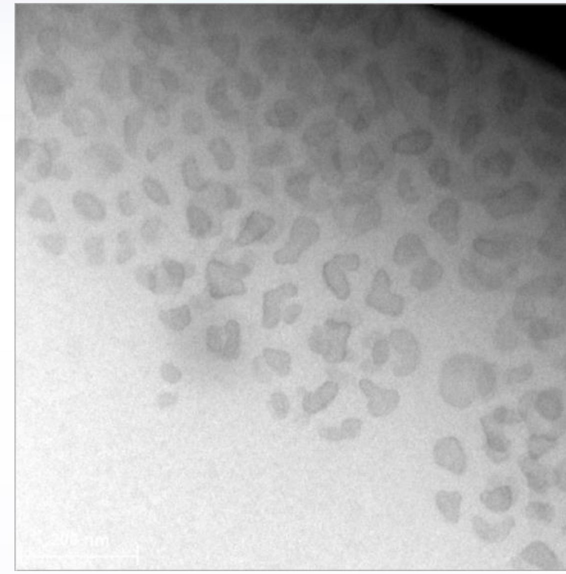




BSA Crystallization

S.H. Pratt

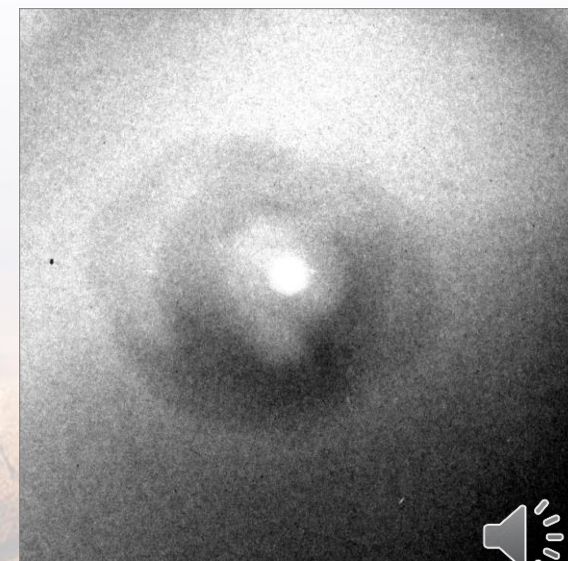
Crystallization of excess Bovine Serum Albumen during flow



La Structure Formation

S.H. Pratt,
T. Nenoff

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

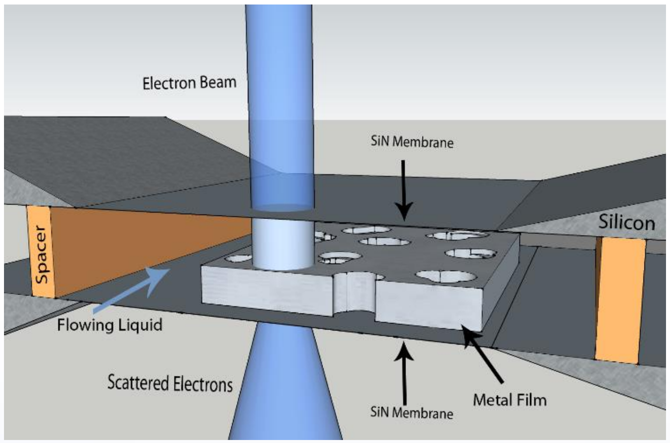


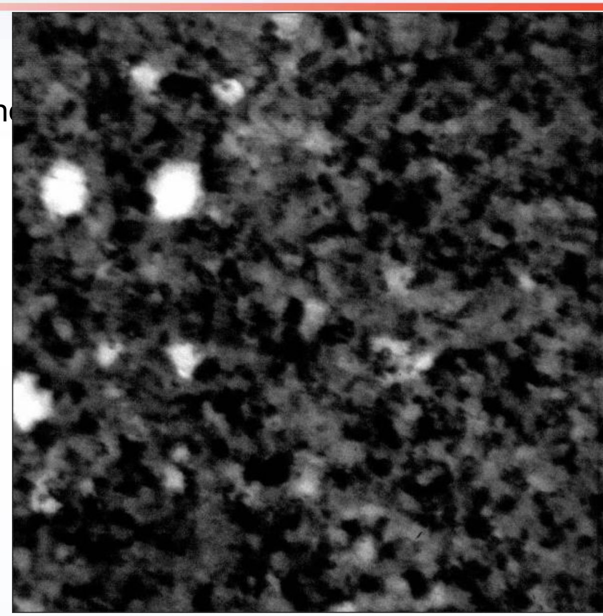
Can We Gain Insight into the Corrosion Process through *In situ* TEM?

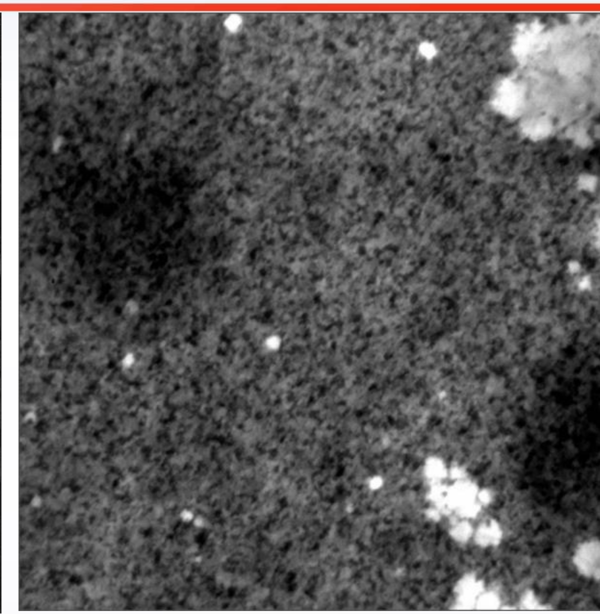
Contributors: D. Gross, J. Kacher, I.M. Robertson & Protochips, Inc.

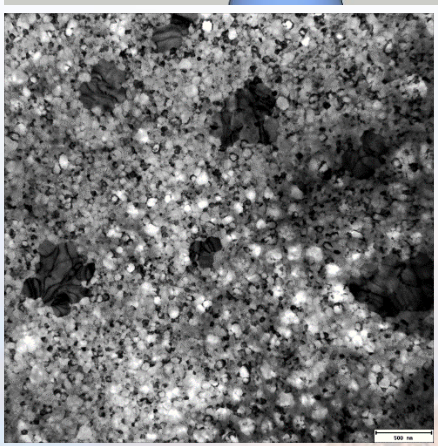
Microfluidic Stage

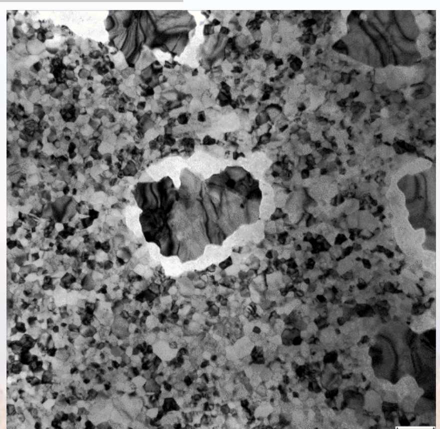
- Mixing of two or more channels
- Continuous observation of the reaction chann

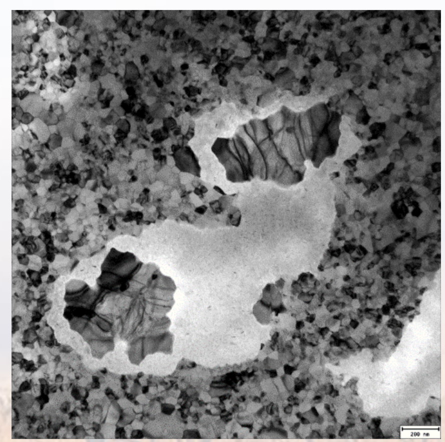


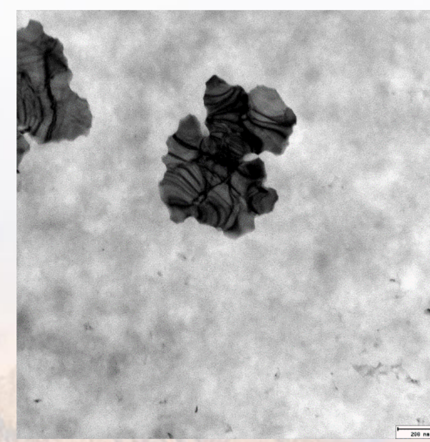












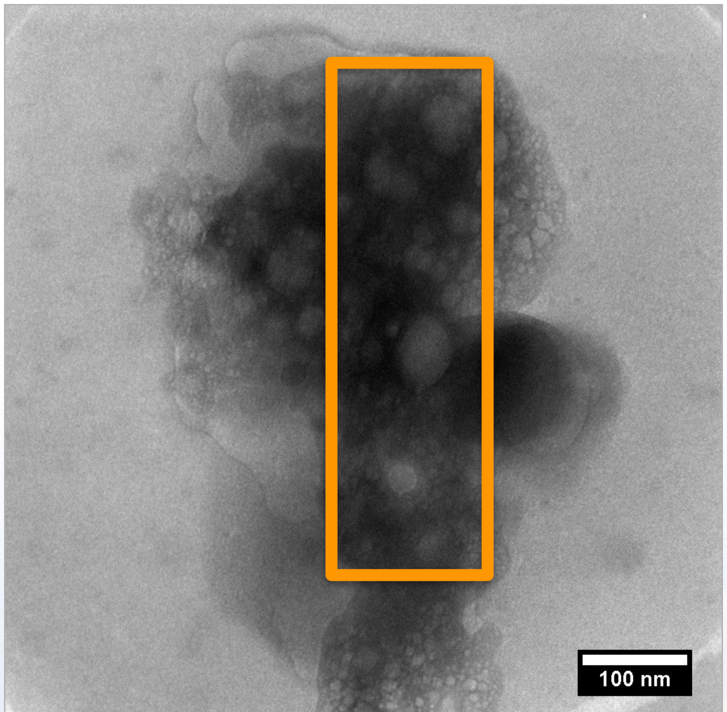
Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.
Large grains resulting from annealing appear more corrosion tolerant

PED in Liquid Cell Environment

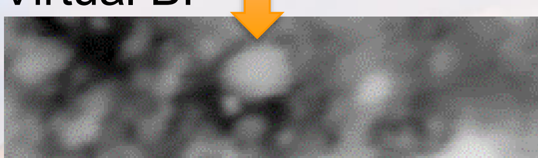
Contributors: C. Taylor, S. Pratt, & T. Nenoff

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

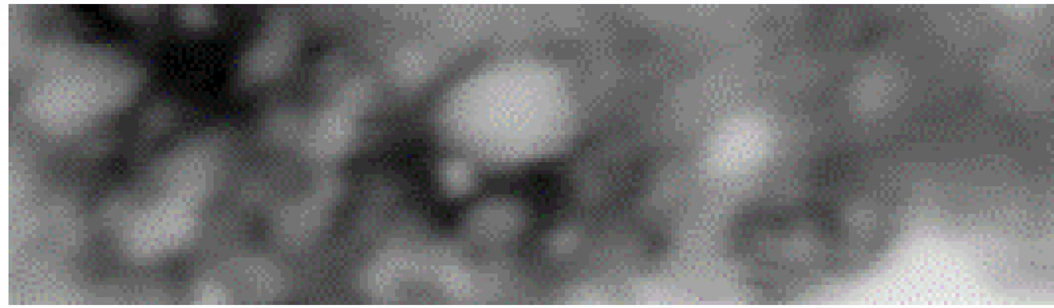
TEM Image



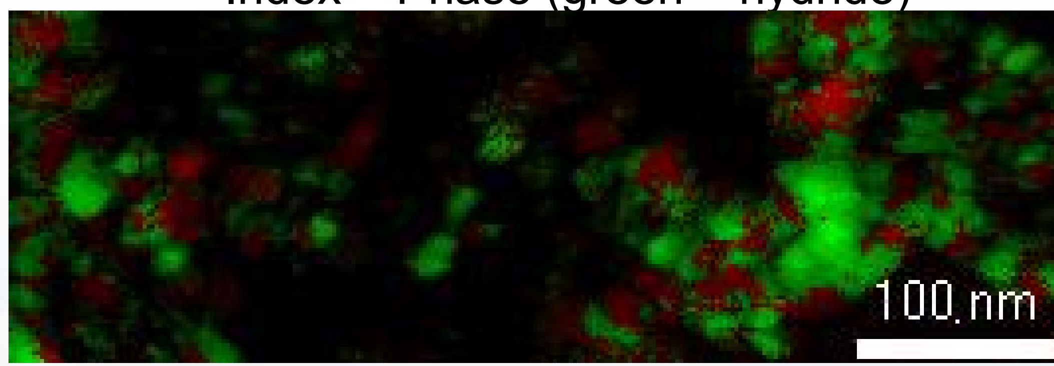
Virtual BF



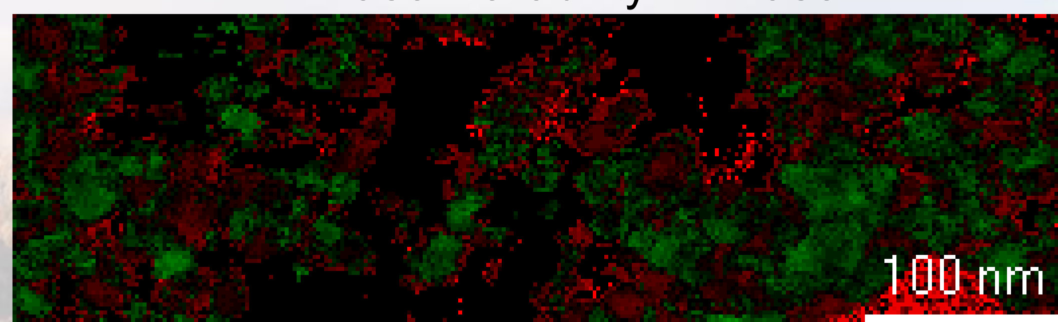
Virtual BF



Index + Phase (green = hydride)



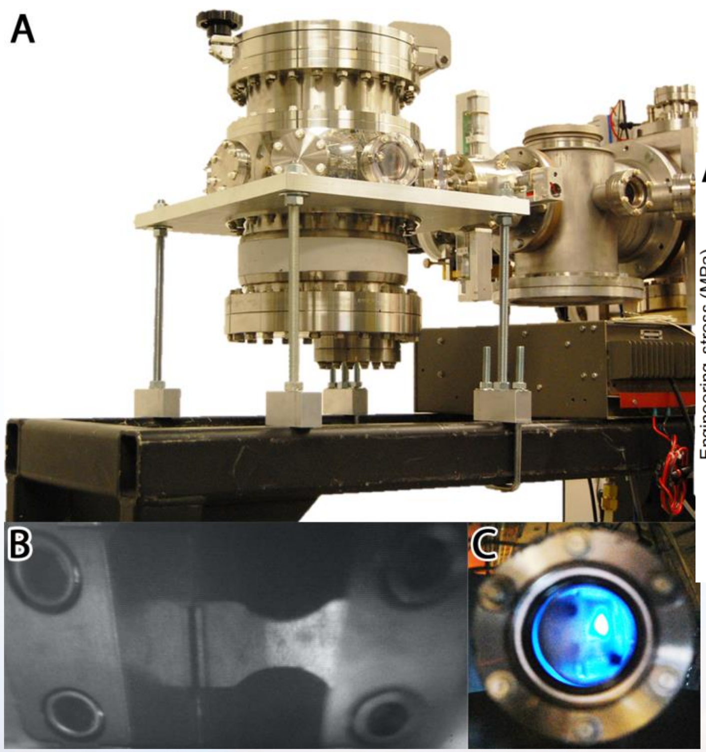
Phase Reliability + Phase

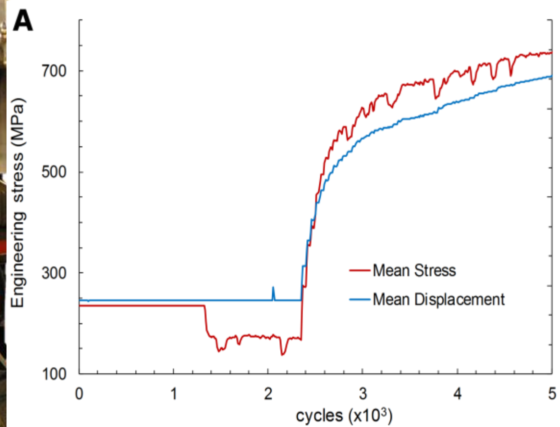


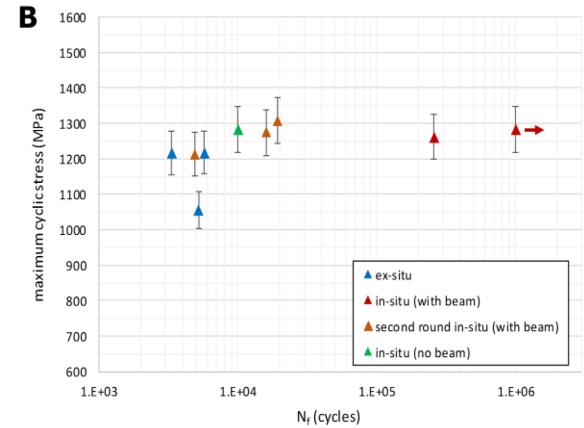
Ex situ Mechanical Testing End Station

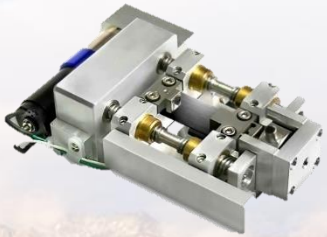
Collaborators: D. Buller, B. Boyce, J. Carroll, P. Price, C. Taylor, B. Muntifering, S. Briggs, N. Heckman, J.A. Scott

- Combined three individual mechanical testing in tandem beamline end station
- Limited (optical, IR only) imaging capabilities
- Have successfully collected preliminary data using this system



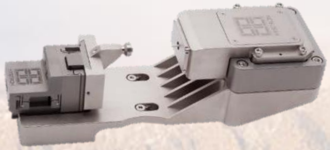


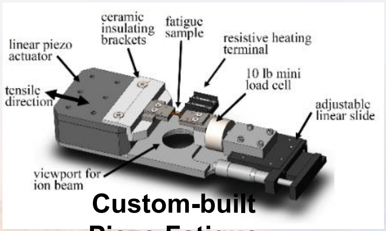




MTI Fullam Straining Heating

Hysitron PI85 Nanoindenter



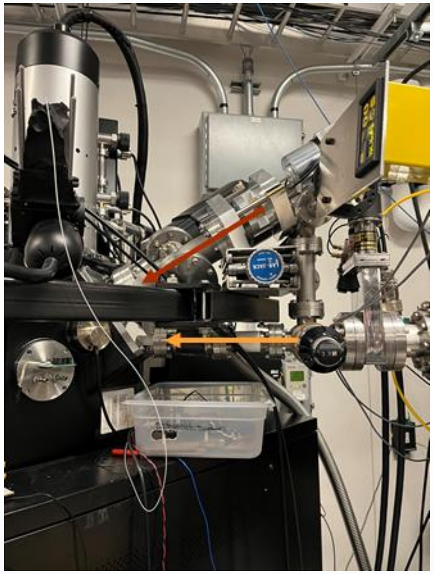


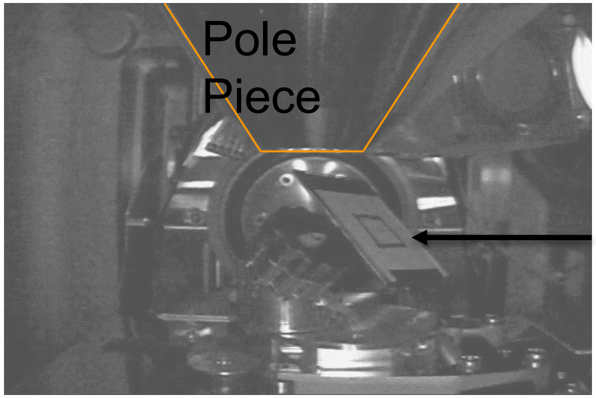
Custom-built Piezo Fatigue tester

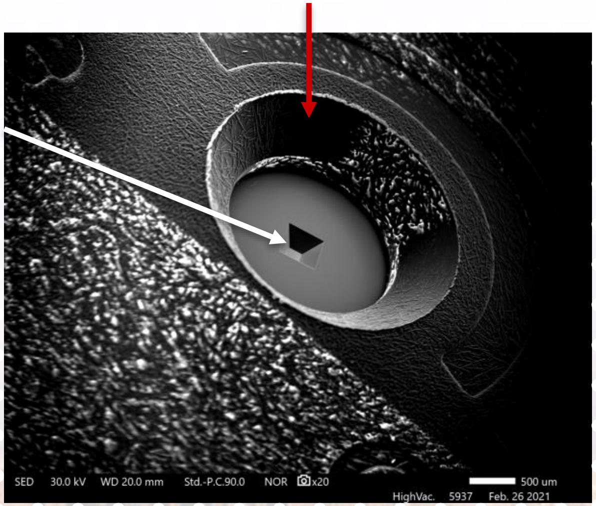
Gatan Cryostage

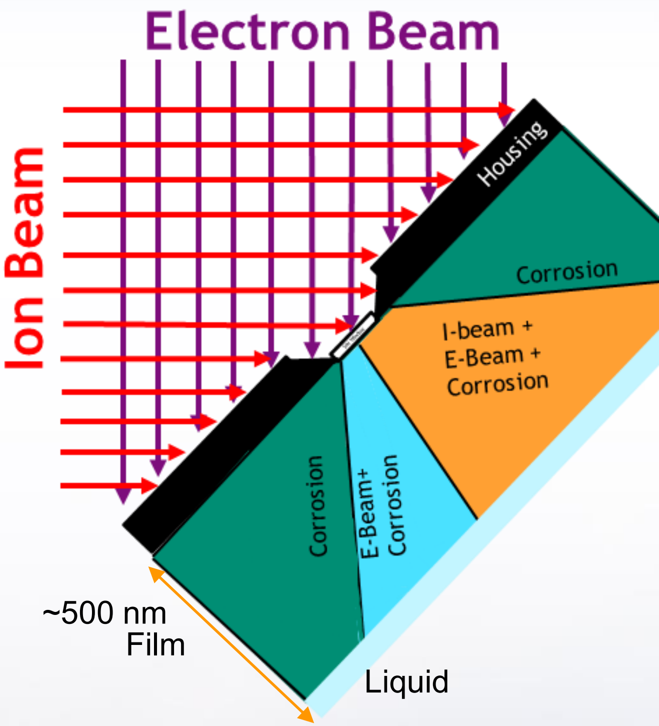
In situ Extreme SEM

Collaborators: .N. Heckman, B.L. Boyce, B. Derby, N. Li

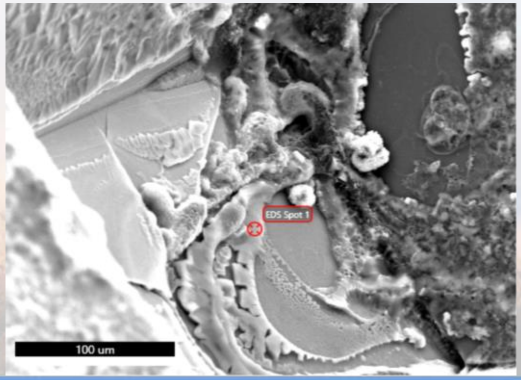






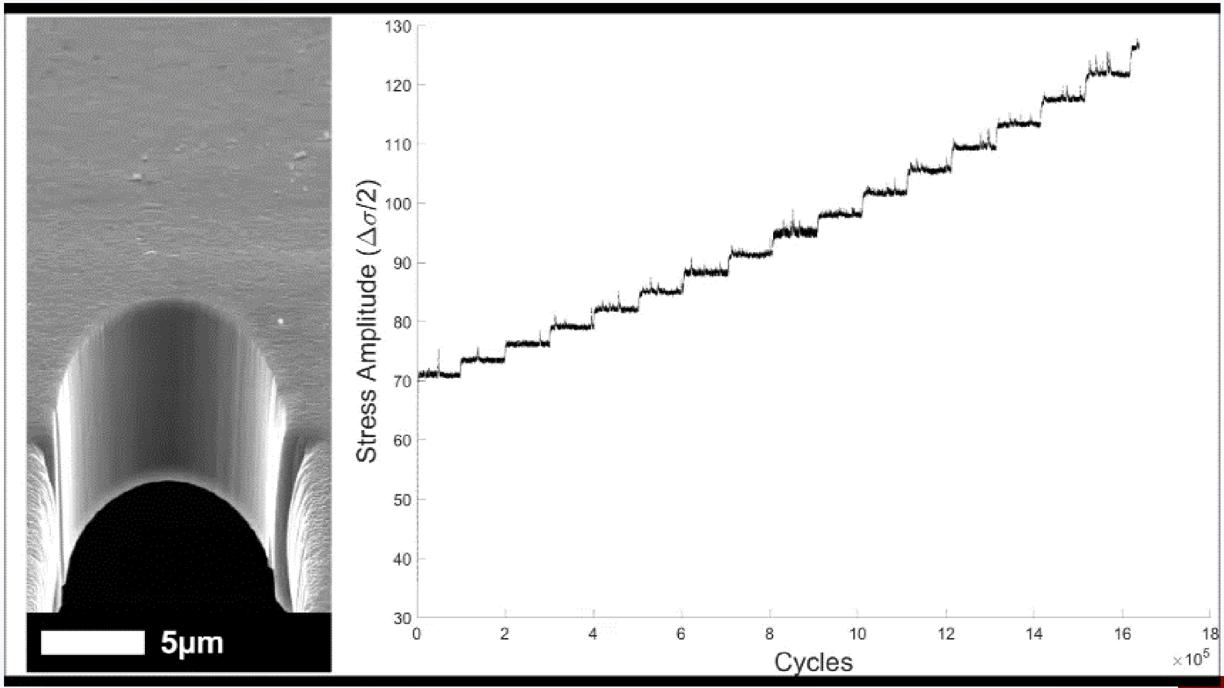


We can now perform in-situ SEM heavy ion irradiation, ion implantation, corrosion, creep, and fatigue.



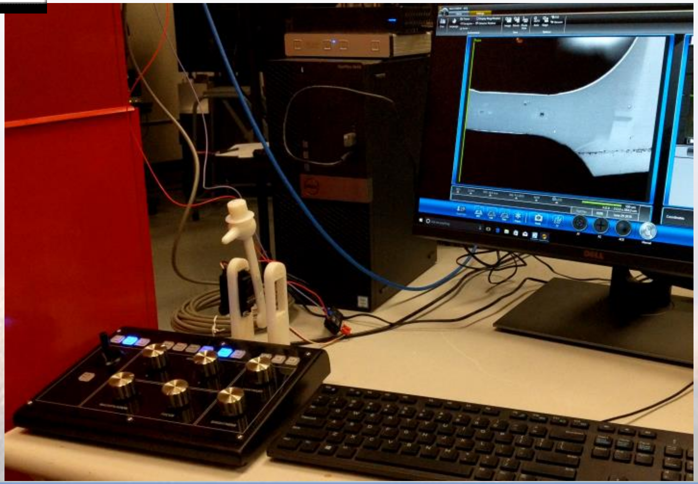
In situ High-cycle Fatigue

Collaborators: .N. Heckman, B.L. Boyce,



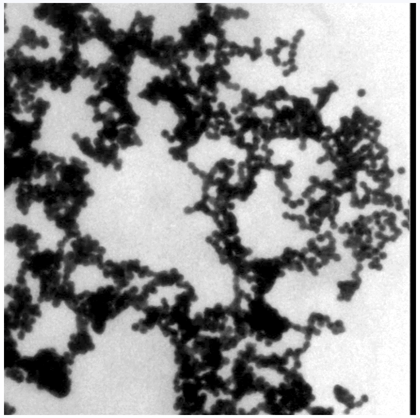
- Nanocrystalline Ni-40Fe, 10-60 nm grain size, 10 µm notch, imaged at 60°
- Cycled at 30 Hz, 4000 cycles between images

Direct insight into crack propagation and failure during cyclic loading

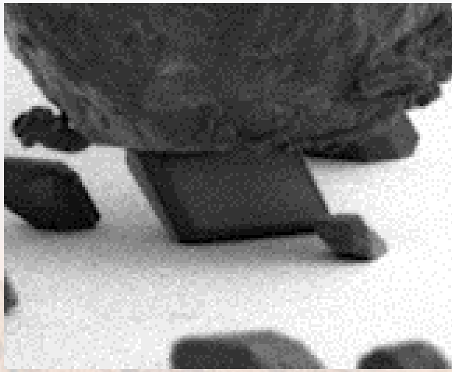


Unconventional *In situ* Microscopy Creates a Wealth of Possibilities

- Plethora of extreme environments that have not been fully explored.
- Utilizing the TEM as an experimental chamber provides a range of nanoscale extreme environments.
- Combining extreme conditions opens up the ability more complex real world applications.
- ACOM and other analytical techniques coupled with in situ observations provides a nice bridge to MD and mesoscale modeling.
- If you would like to hear about the I^3SEM let me know



The future is bright and fastly approaching for coupled *in situ* TEM



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

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

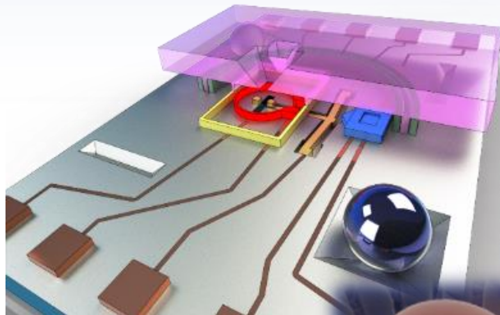
Sandia National Laboratories

Sandia's User and Position Opportunities

D. Hanson, W. Martin, M. Wasiolek

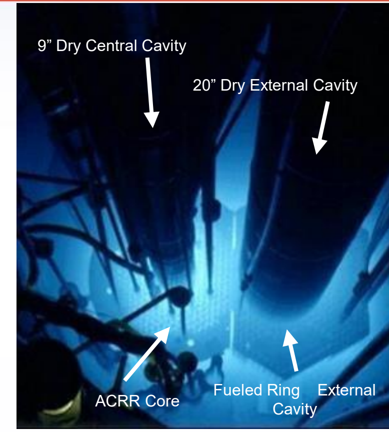
www.cint.lanl.gov

- 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

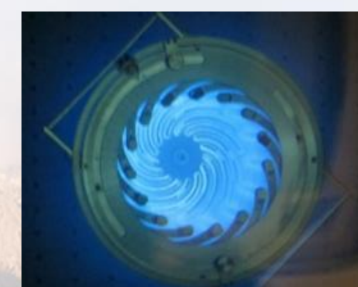
Position Oppurtunities at:

https://www.sandia.gov/careers/

Post-doc = 671121

Grad Student = 670865

Undergrad Student = 670864



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.

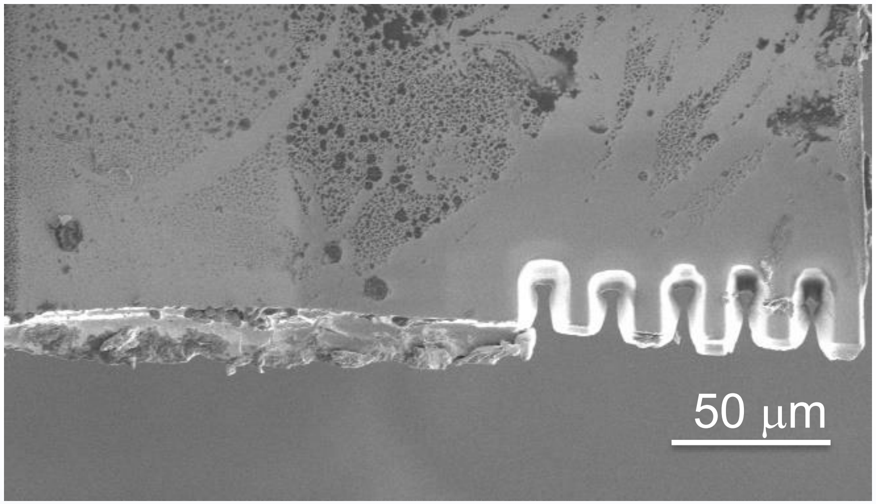
Back-up

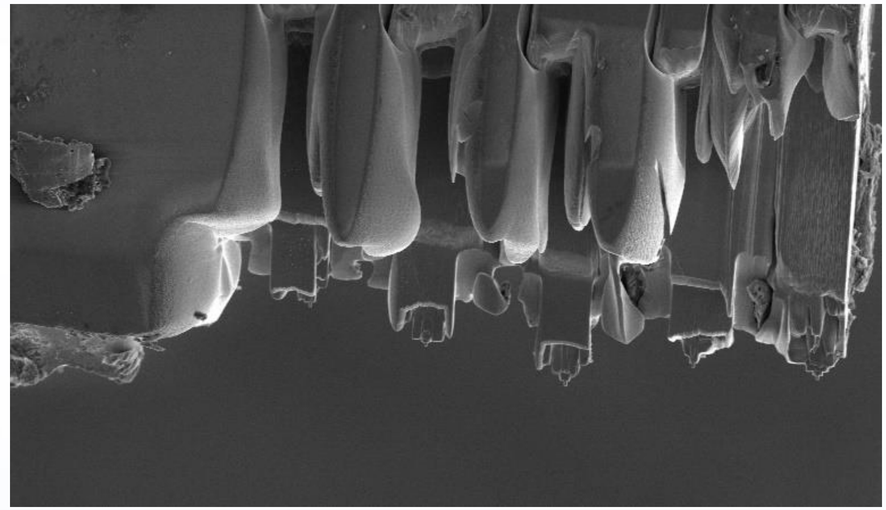
Temperature Upper Bound

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

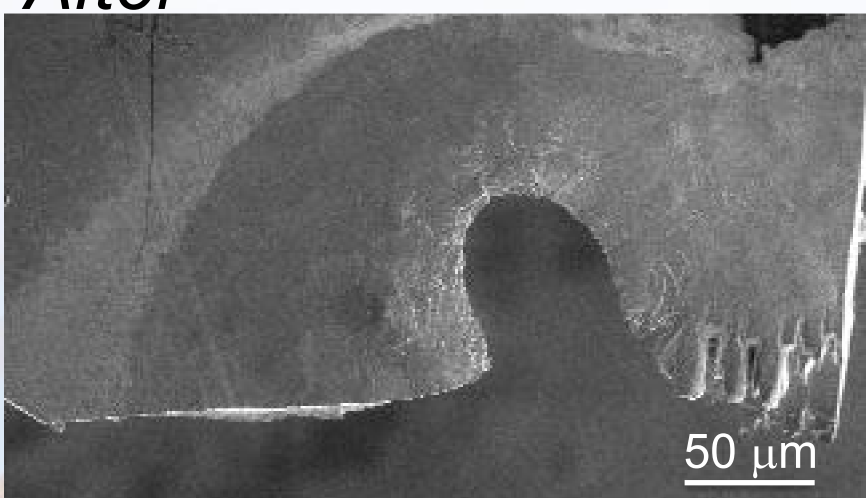
Before

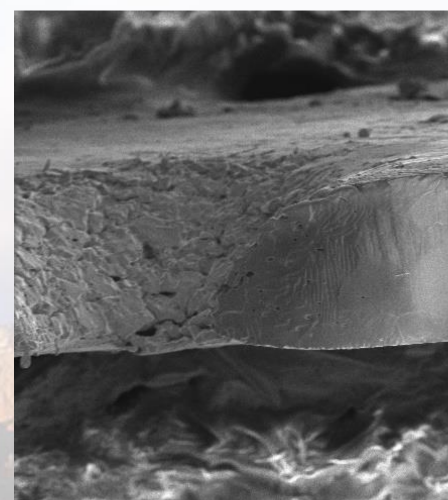
Sc_2O_3 doped ZrO_2

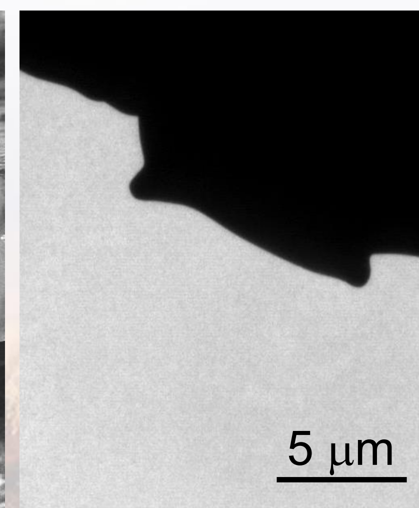




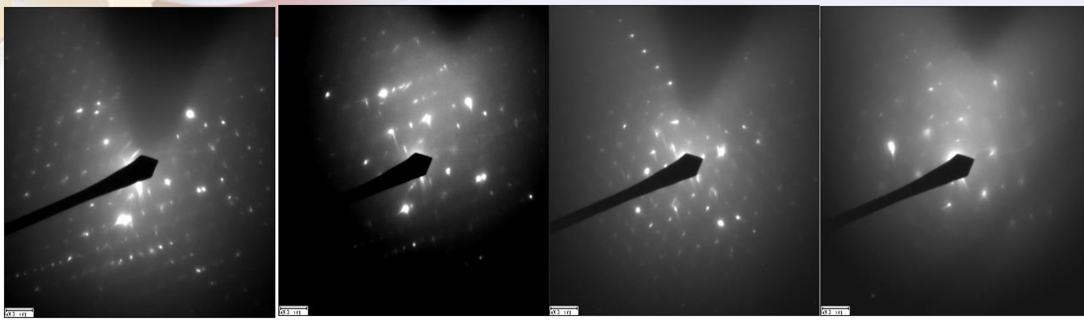
After



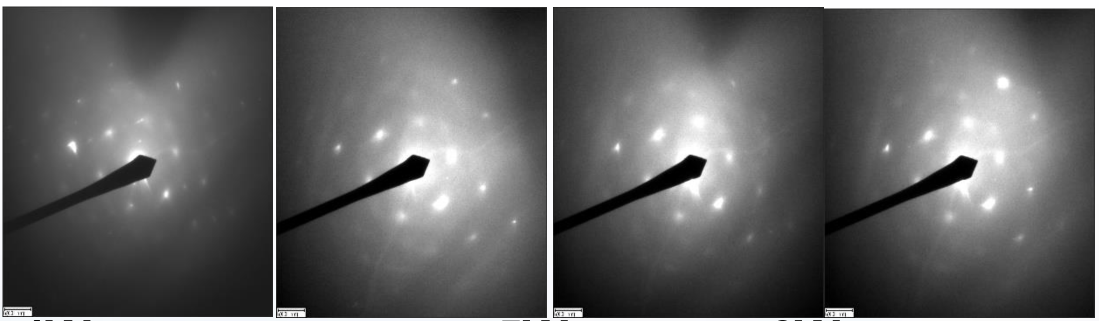




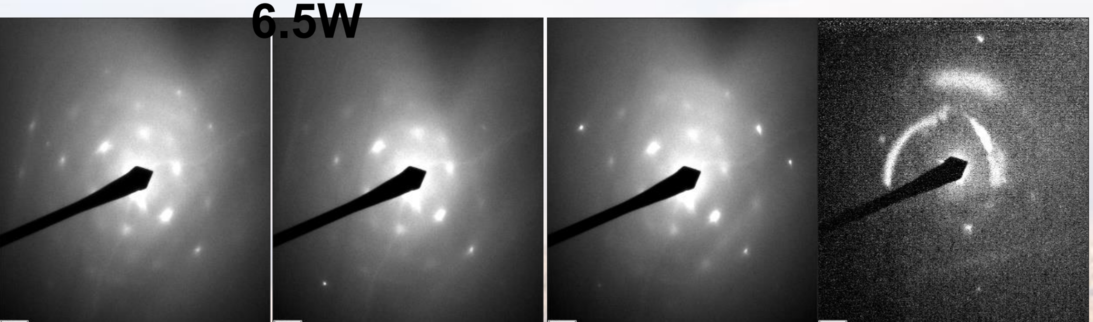
Diffraction for Temperature Calibration



2W (β) 3W heating 3.5W (β)



4W 5W 6W 6.5W



7.7W 8W 8.5W 9.5W