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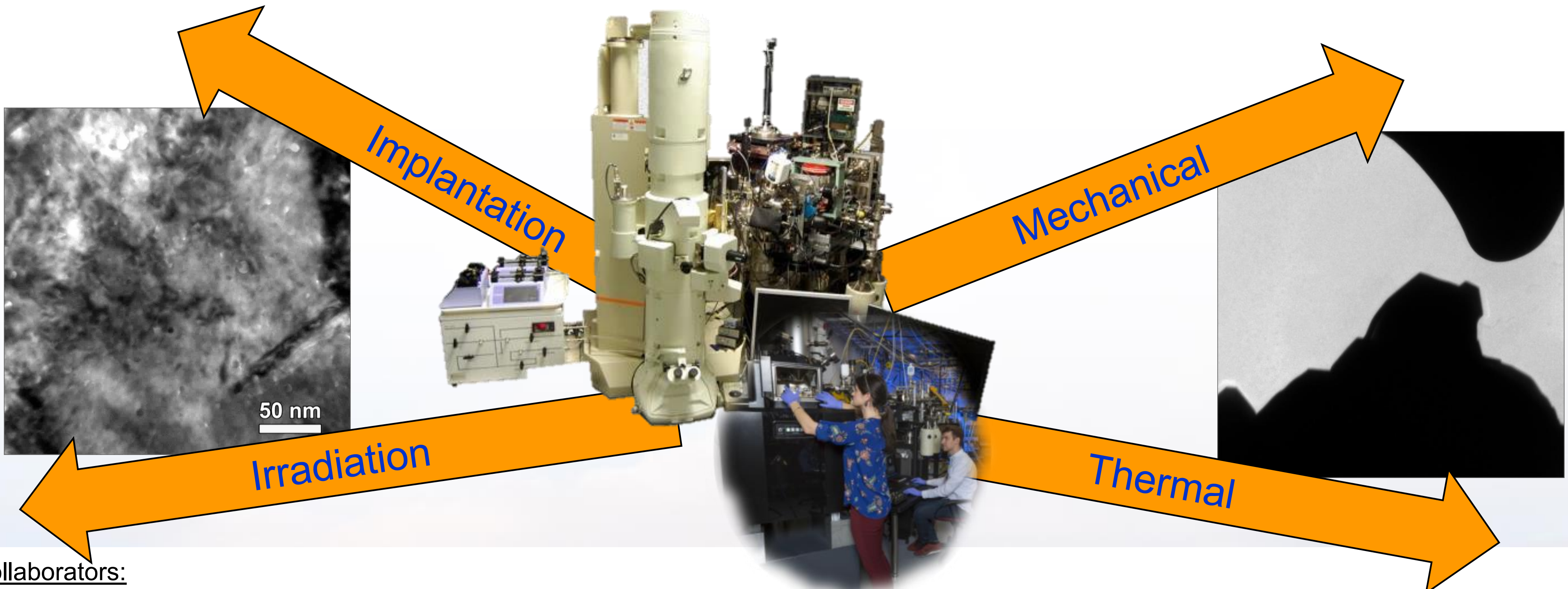
Irradiation Creep and Fatigue Observed via In-situ Electron Microscopy

Khalid Hattar[1], Eric Lang[1], Shen J. Dillon[2,3]

[1]Sandia National Laboratories, Albuquerque, NM 87185, USA

[2]University of Illinois Urbana Champaign, Urbana, IL 61801, USA

[3]University of California, Irvine, CA 92697



Collaborators:

- B.L. Boyce, 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, 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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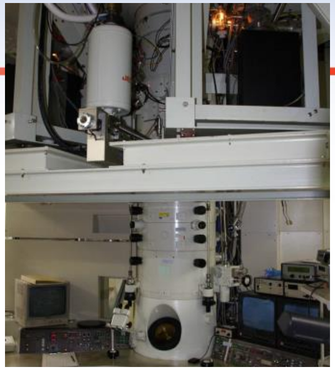
History of *In situ* Ion Irradiation TEM

Courtesy of: J. Hinks



Workshop on Ion Irradiation TEM

Huddersfield, UK (2008)
Albuquerque, USA (2011)
Saporro, Japan (2013)
Paris, France (2016)
Huddersfield, UK (2018)
Ann Arbor, MI, USA (2020)

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

1931
The invention of the TEM

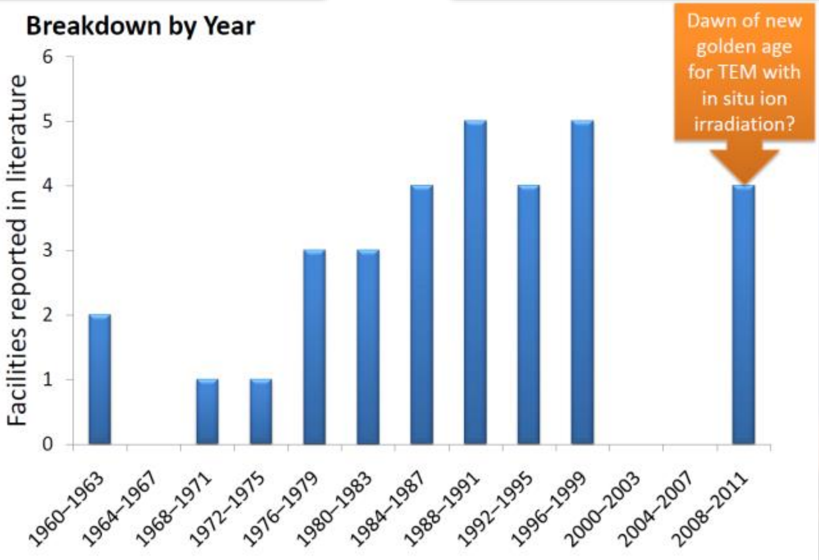
1961
O^- emission reported from a TEM filament by Pashley, Presland, and Meneter at TI Labs, Cambridge, UK

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

1976
First HVEM with ion irradiation at UVA, USA

1978
First in-situ ion irradiation experiments at ANL

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



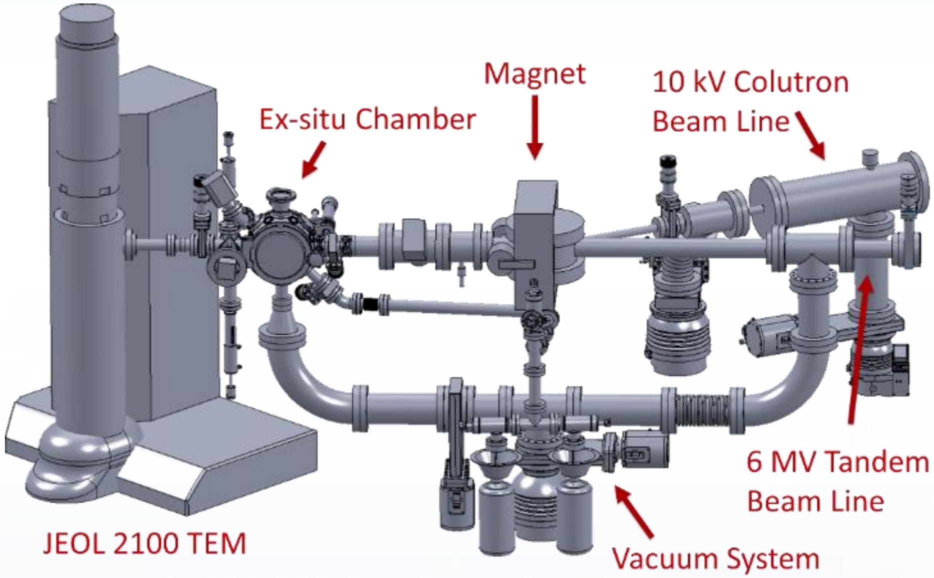
"The direct observation of ion damage in the electron microscope thus represents a powerful means of studying radiation damage"

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

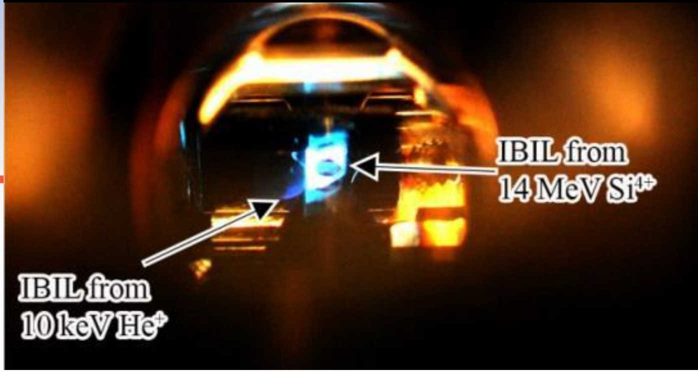
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

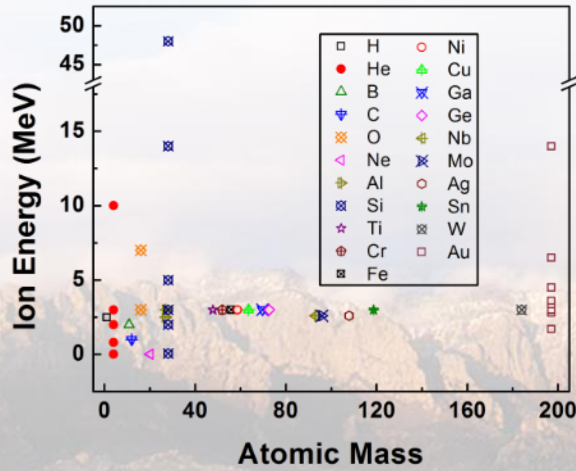
Collaborator: D.L. Buller

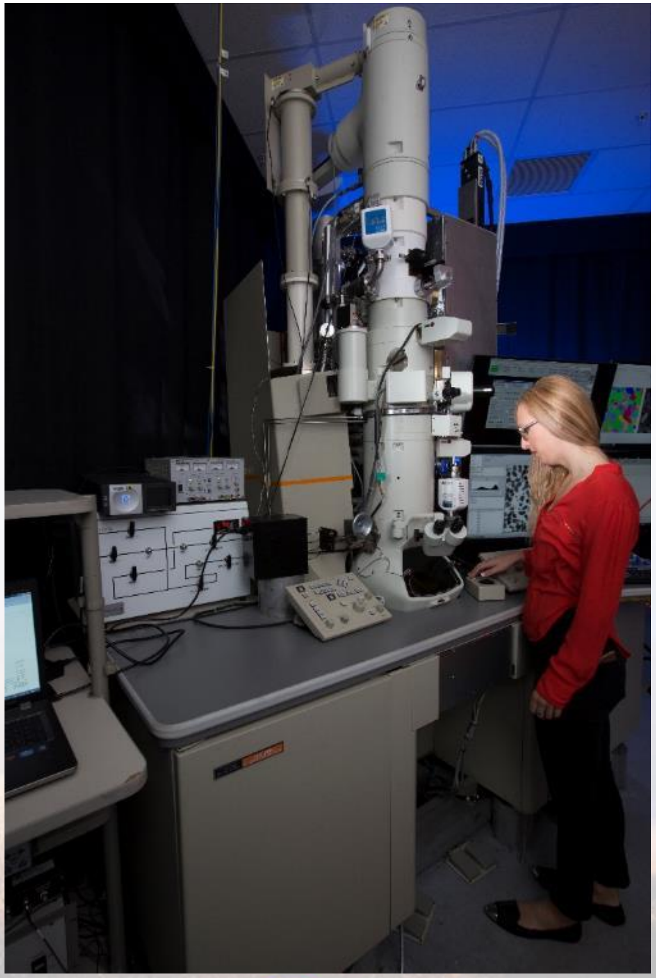
10 kV Colutron - 200 kV TEM - 6 MV Tandem



IBIL from a quartz stage inside the TEM

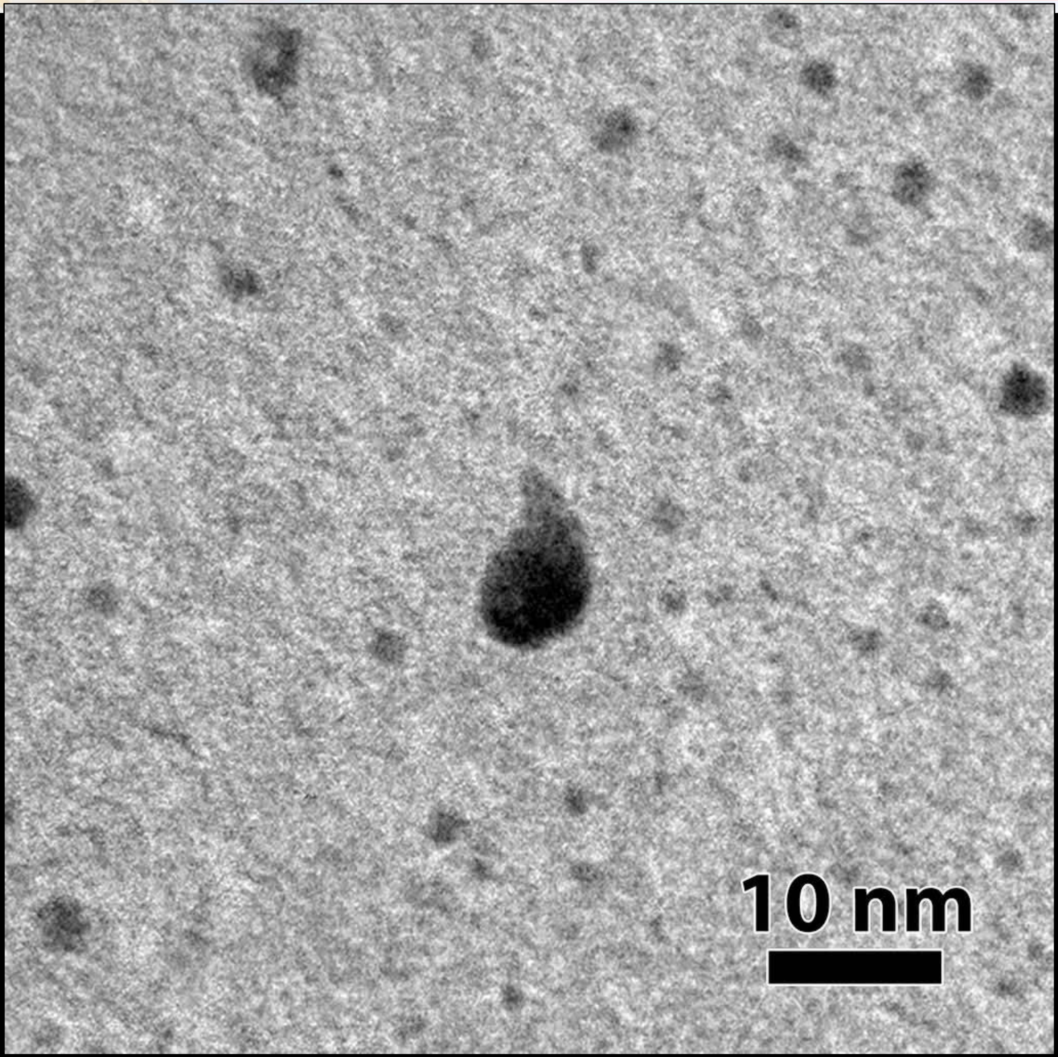


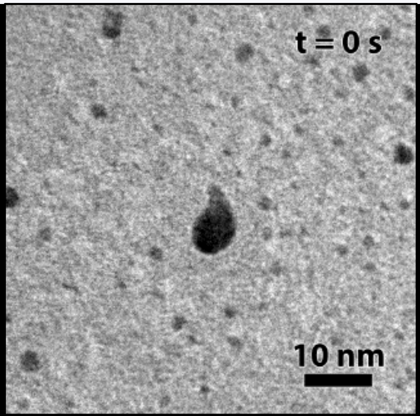


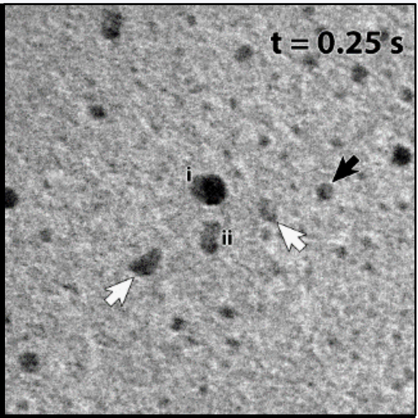


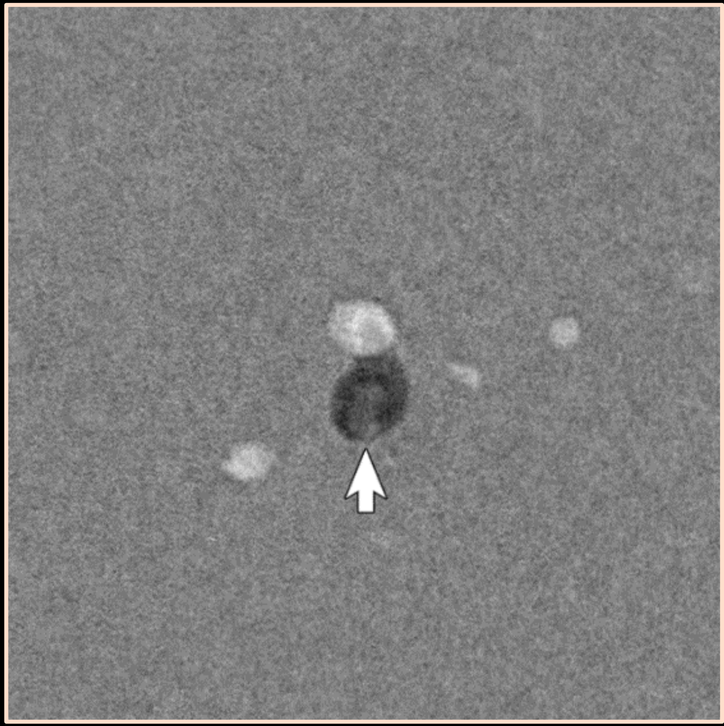
Single Ion Strikes:
46 keV Au^{1-} ions into ~5 nm Au nanoparticles

Collaborator: D.C. Bufford



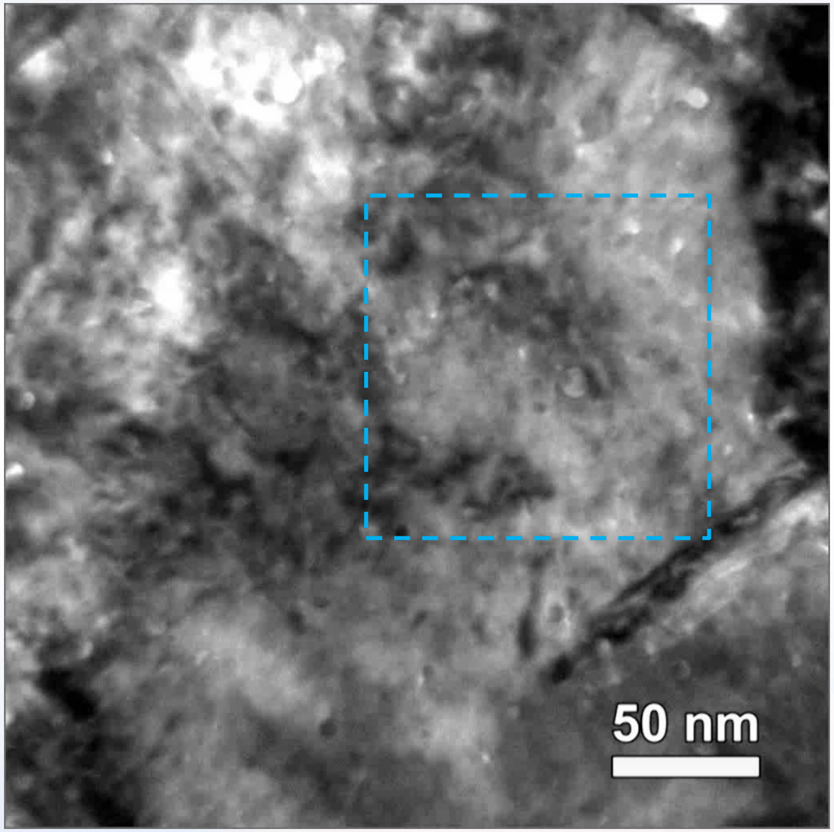


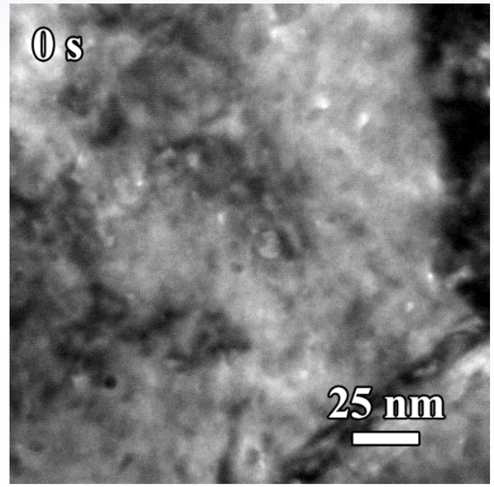


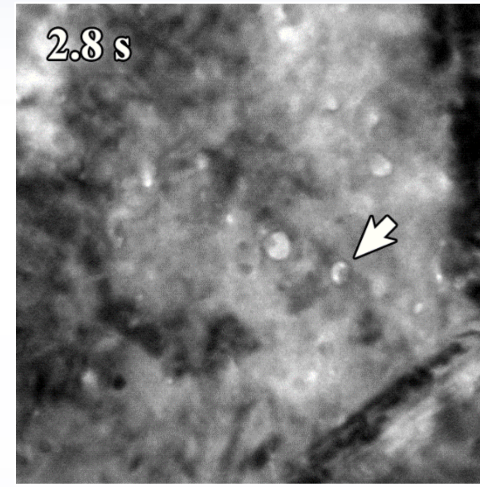


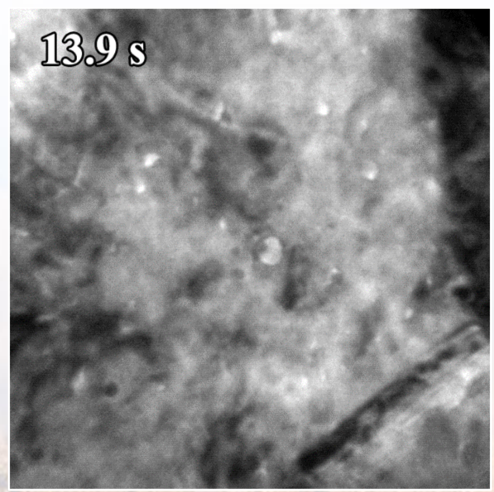
Simultaneous *In situ* TEM Triple Beam: 2.8 MeV Au^{4+} + 10 keV He^{+}/D_2^{+}

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^{3}TEM! Intensive work is still needed to understand the defect structure evolution that has been observed.

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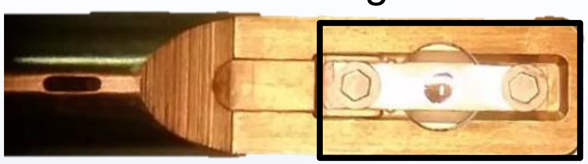
In situ Qualitative Mechanical Testing

Collaborators: C. Barr

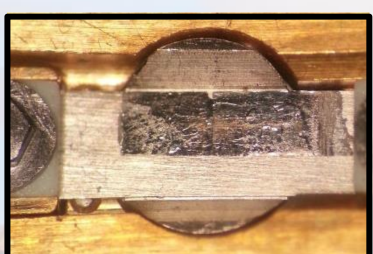
Gatan straining TEM Holder

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

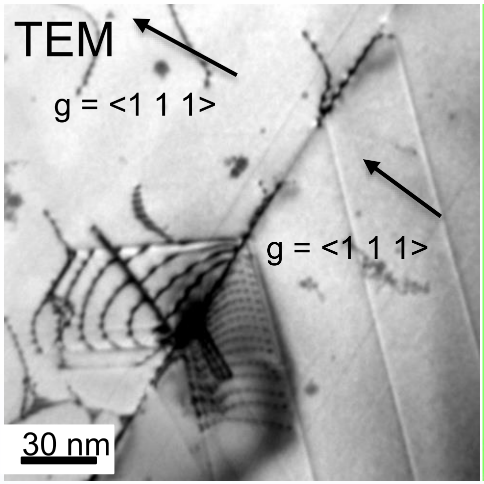
Traditional Gatan Heating and Straining Holder

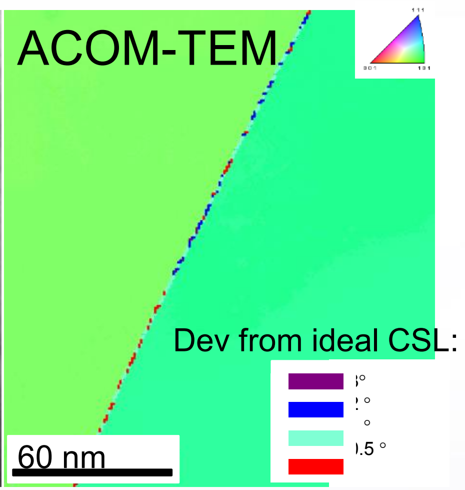


Thin film tension "jig": Jet thinned disk:

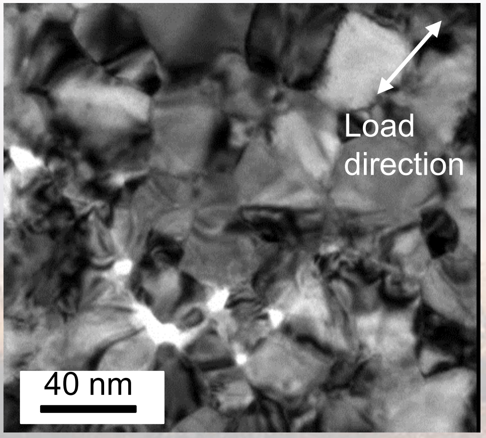


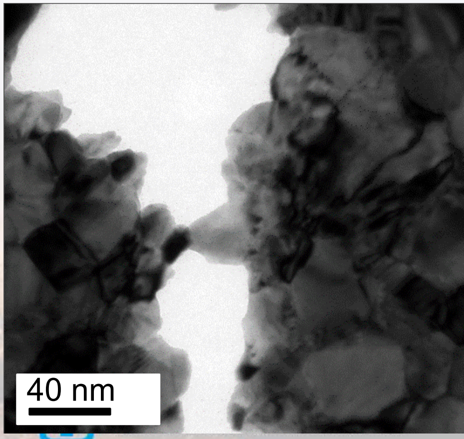
Dislocation interactions as a function of GB character (Σ3 twin GB below):





Observe deformation mechanisms in nanocrystalline metals during tensile straining:



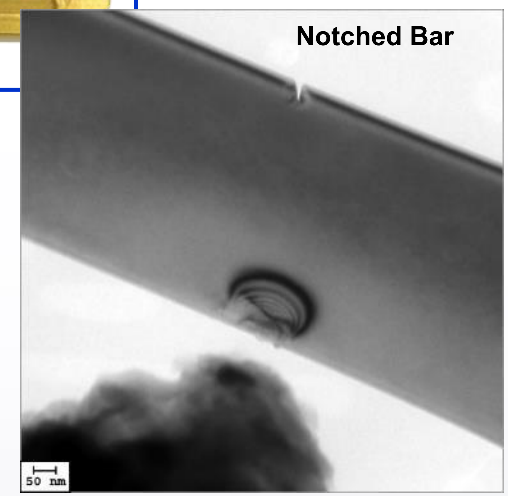


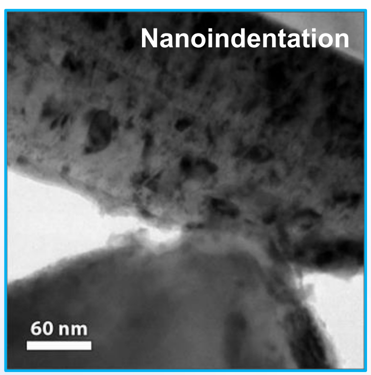
In situ Quantitative Mechanical Testing

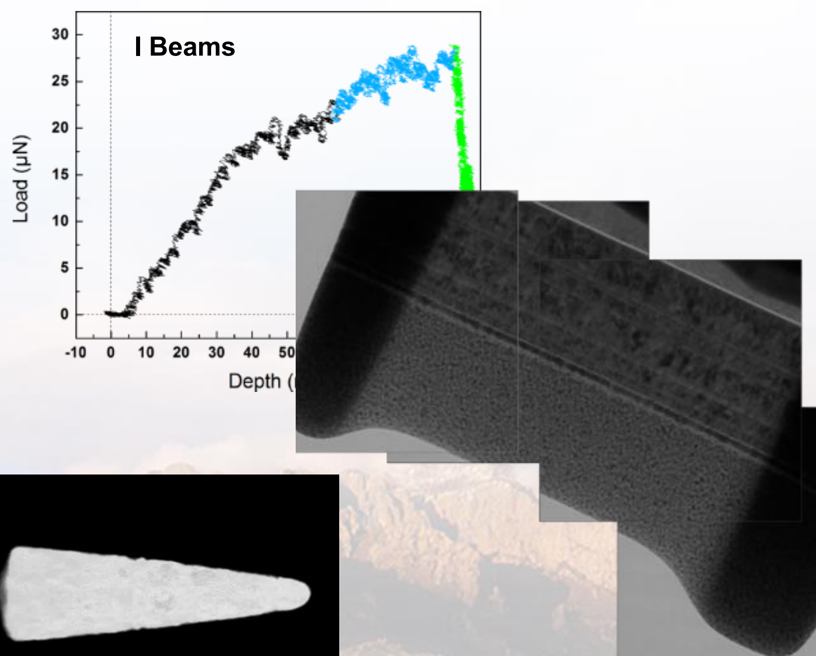
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

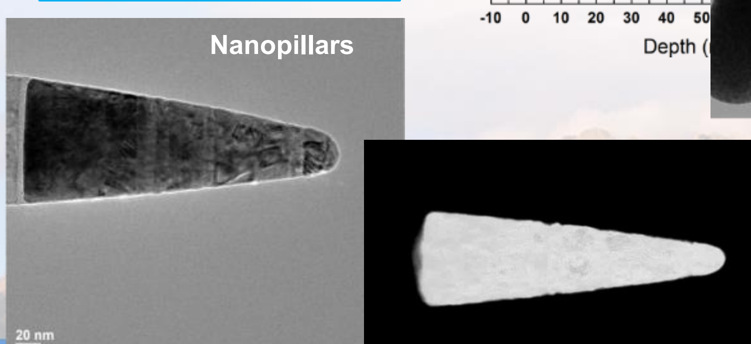
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

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





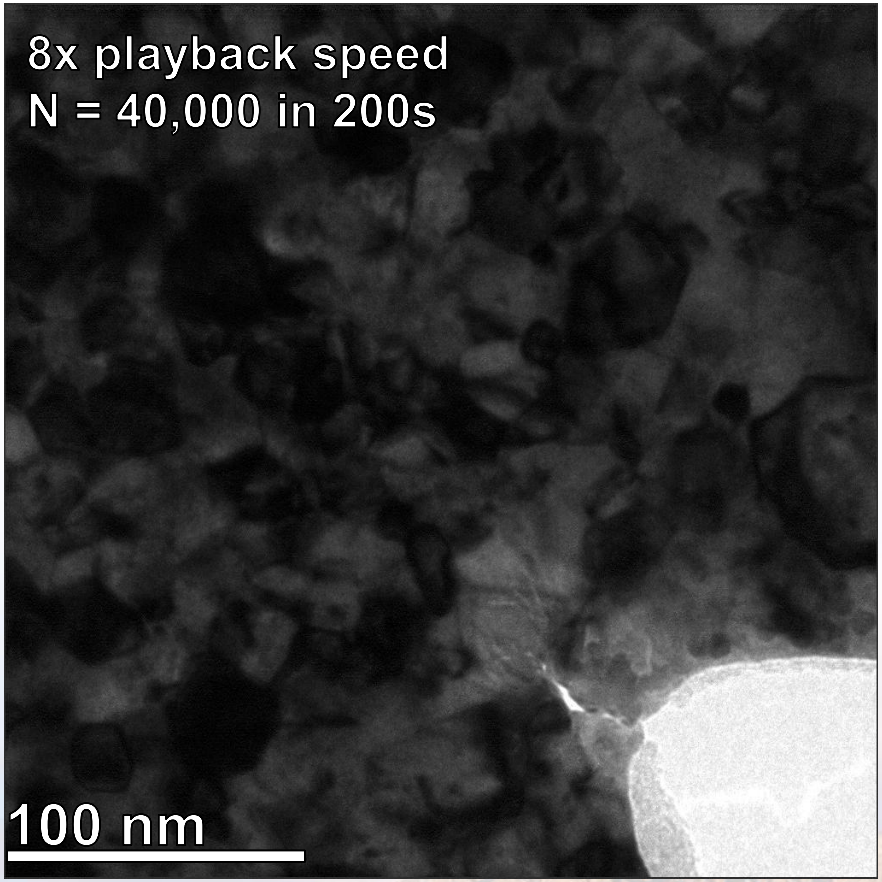




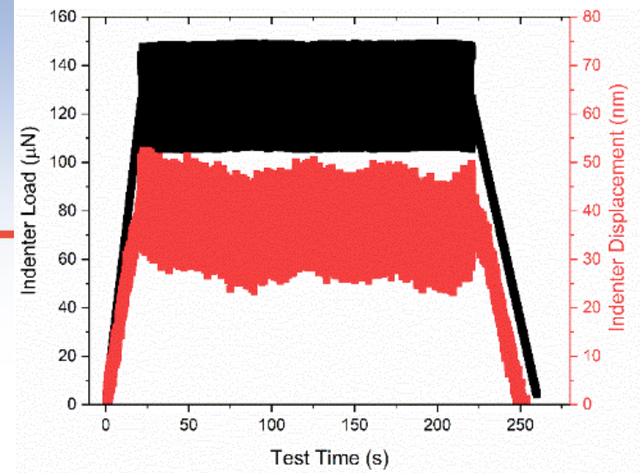
Fatigue Failure in Real Time?

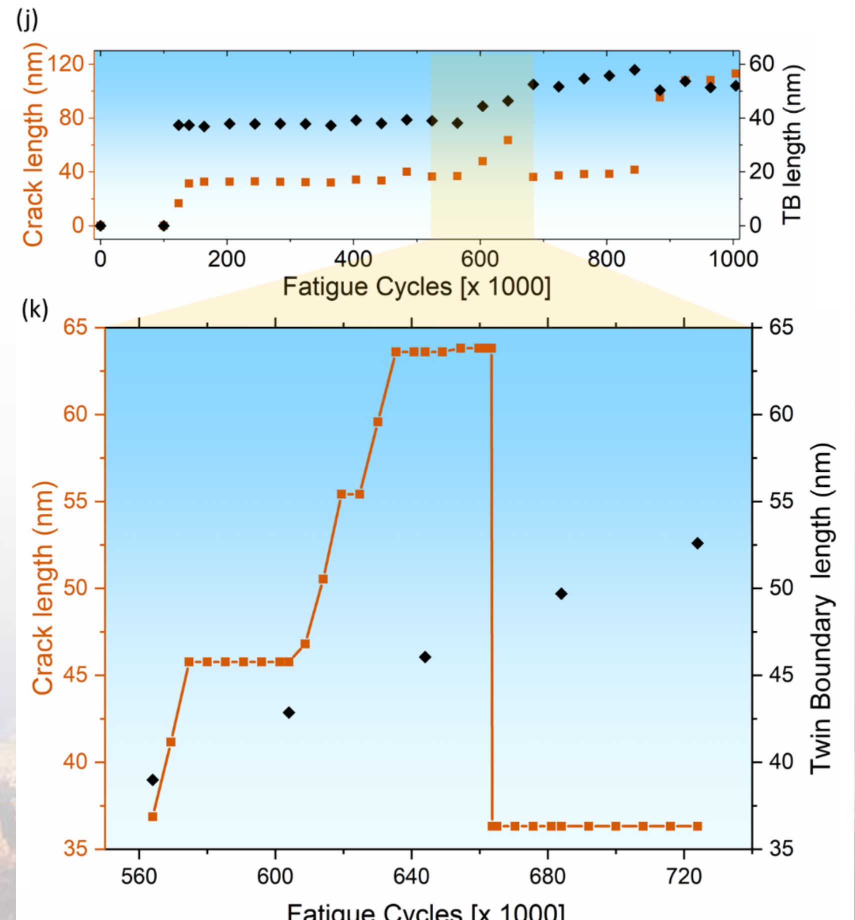
Collaborators: C. Barr, B. Boyce, & W. Mook

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



~ 2 pm/cycle

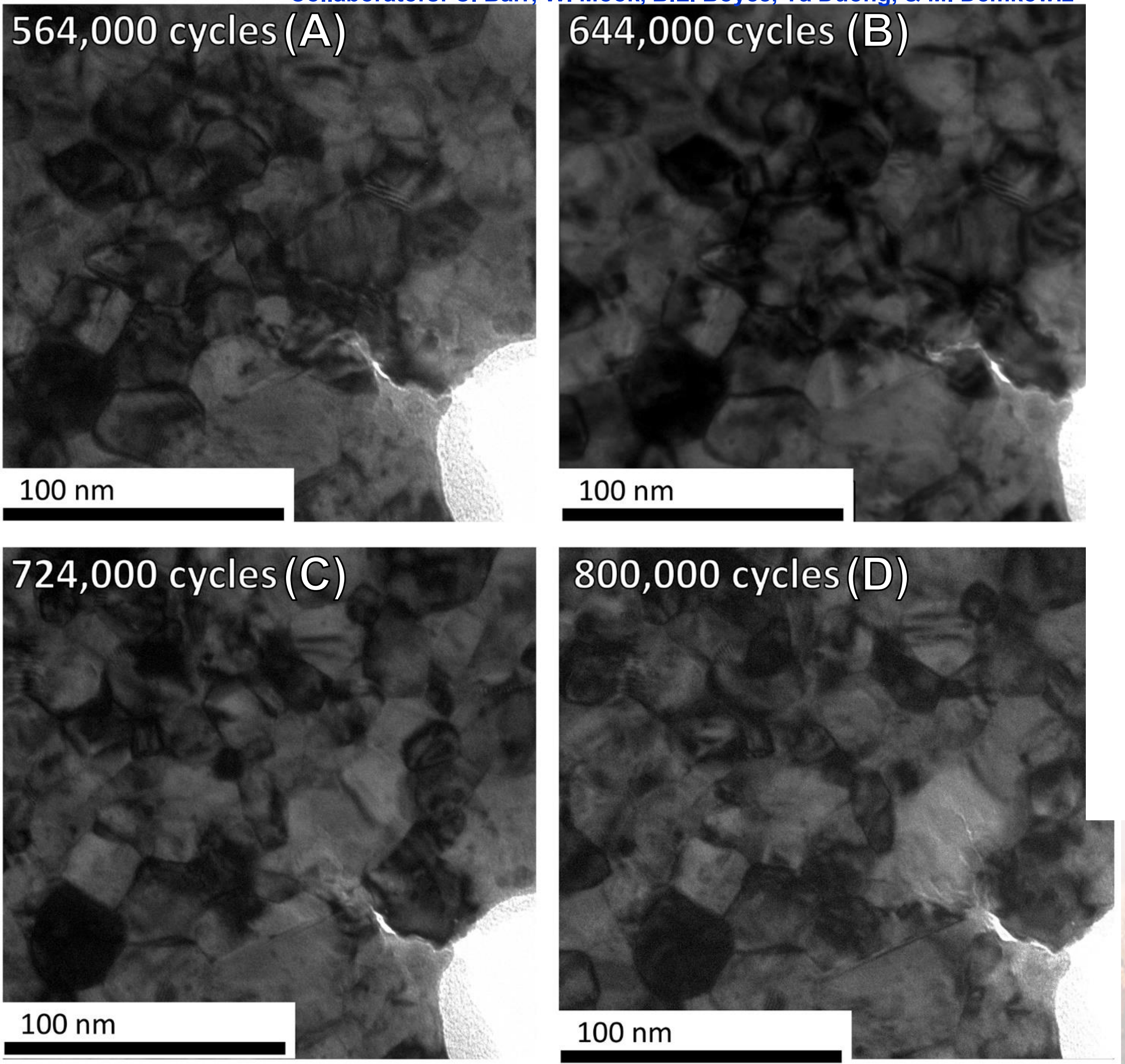


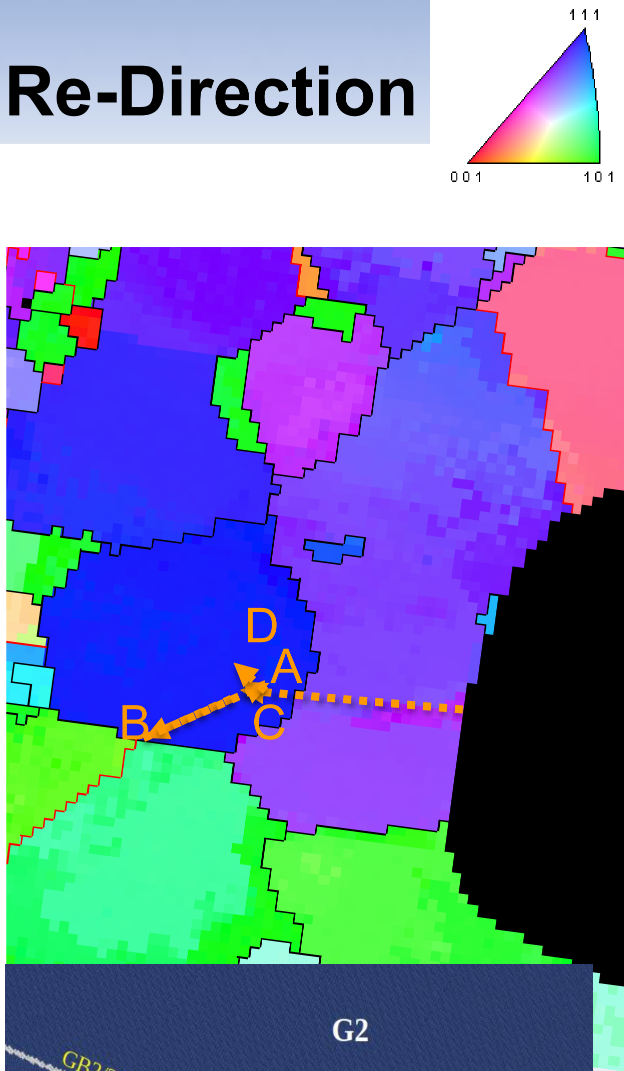


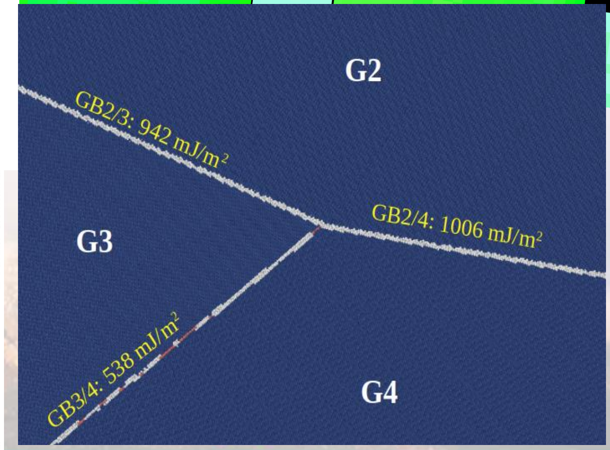
- da/dN = 1.7×10^{12} m/cycle
- Non-linear crack extension rate
- Crack propagation path changes “direction”

Crack Propagation, Closure, and Re-Direction

Collaborators: C. Barr, W. Mook, B.L. Boyce, Ta Duong, & M. Demkowiz

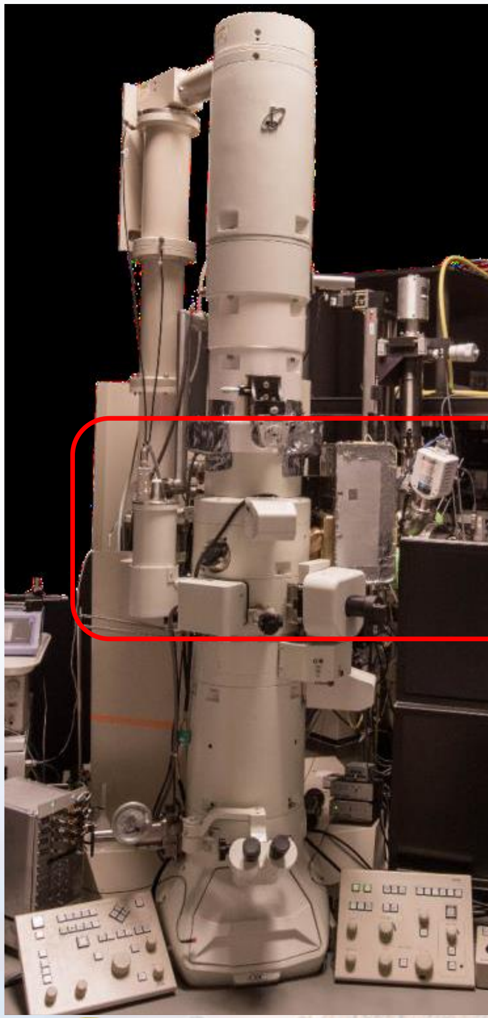


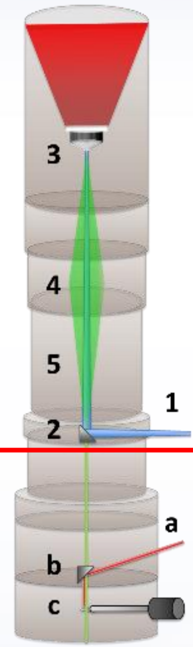




In-situ Specimen Drive Laser System

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





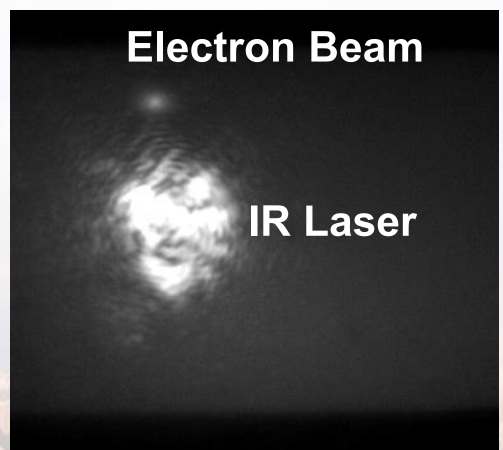
Specimen Drive Laser

a. Adjustable power 1064 nm infrared specimen (IR) drive laser

b. IR laser is reflected directly onto the specimen with metal mirror

c. Heat specimens in *in situ* holders, which otherwise would not be possible

- Laser capabilities:
  - 2-20 Watts
  - Pulsed or continuous operation
  - 50 μm diameter spot size
  - Positioning mirror, which can be used during laser operation



Laser Alignment TEM Holder

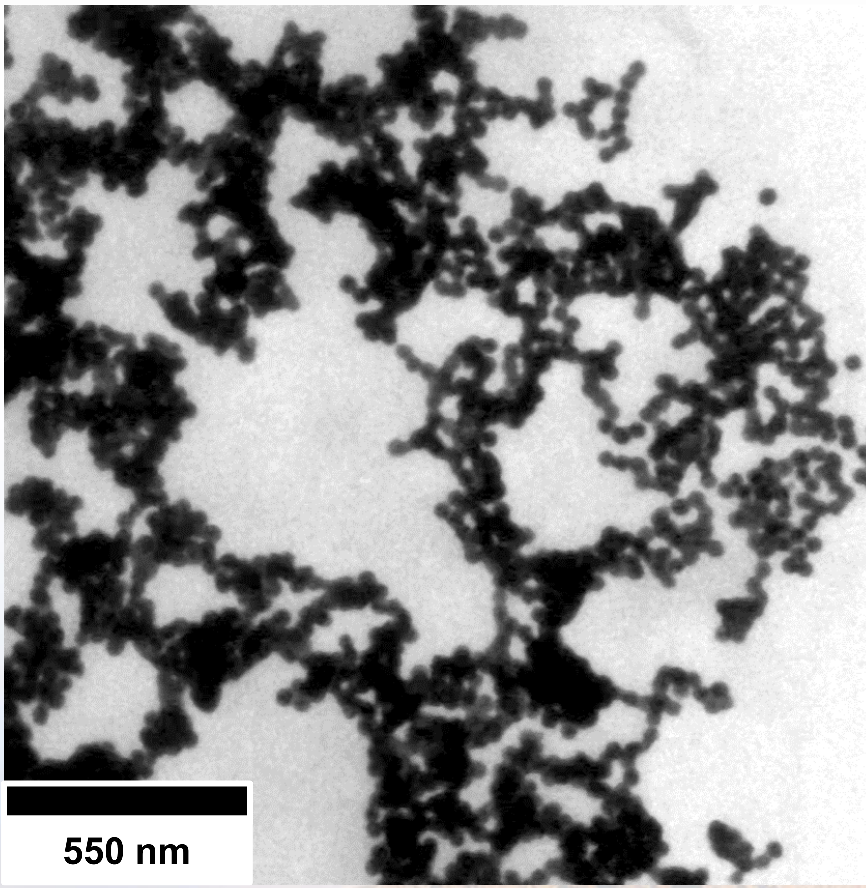
- Phosphor screen
- Borescope
- CCD camera
- Precise alignment of the laser to the electron beam

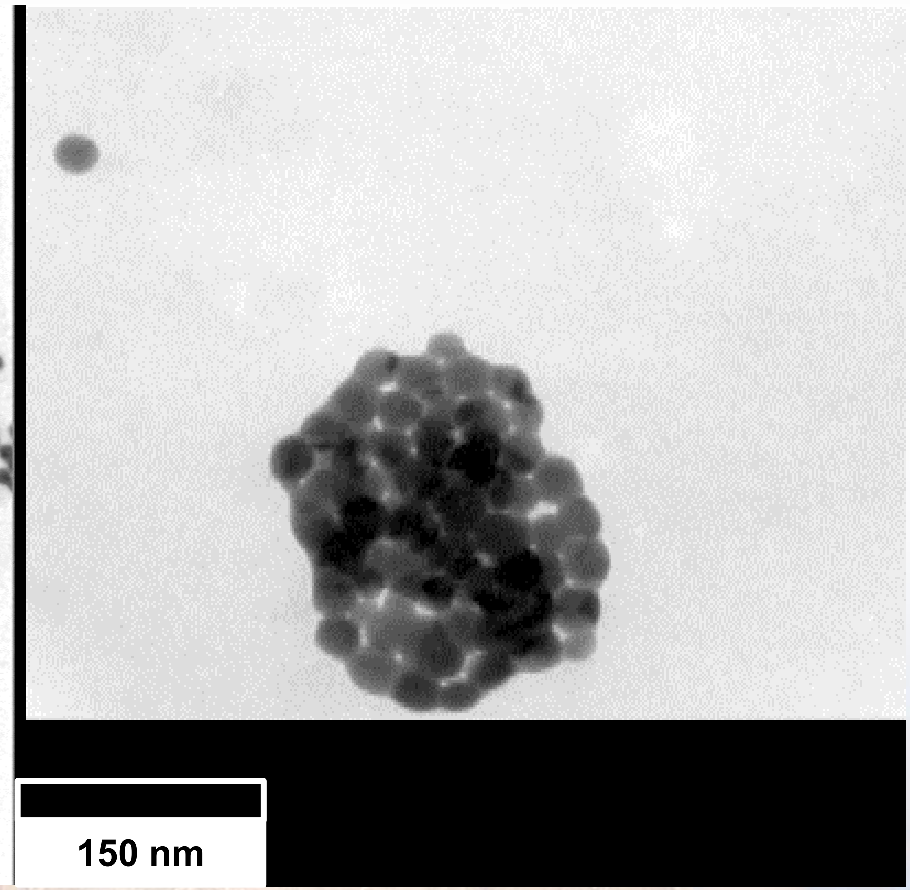
IDES INTEGRATED DYNAMIC ELECTRON SOLUTIONS

Sandia National Laboratories

Complex Interaction Au NPs Exposed to Laser Irradiation

Contributors: P. Price, L. Treadwell, A. Cook, & IDES Inc.



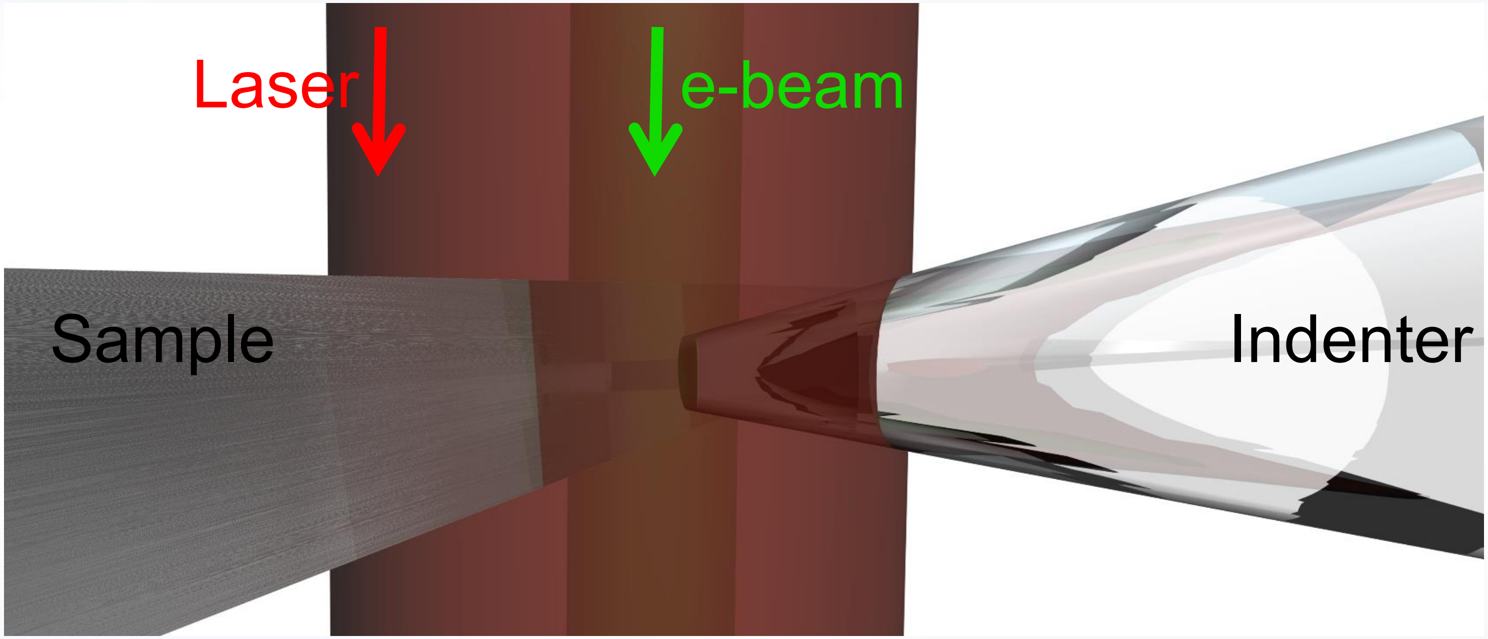


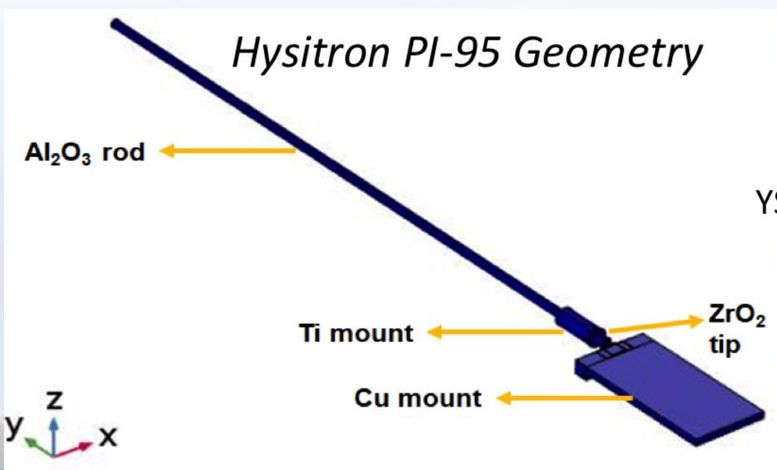
Speed = 2.5x

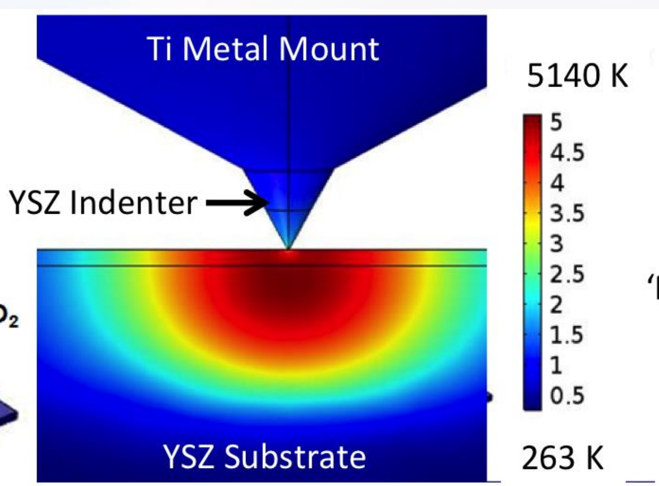
A Complex Combination of Sintering, Reactions, and Ablation Occurs

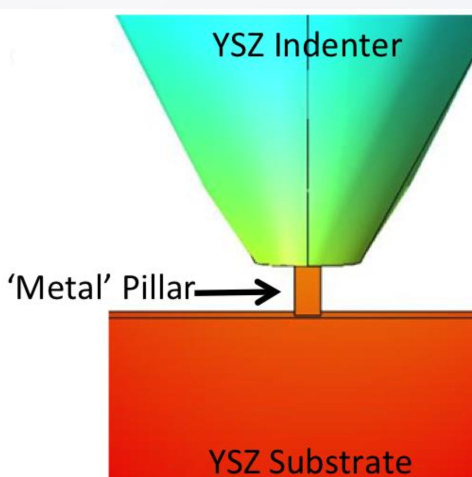
Can we Combine Laser Heating with Mechanical Testing?

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



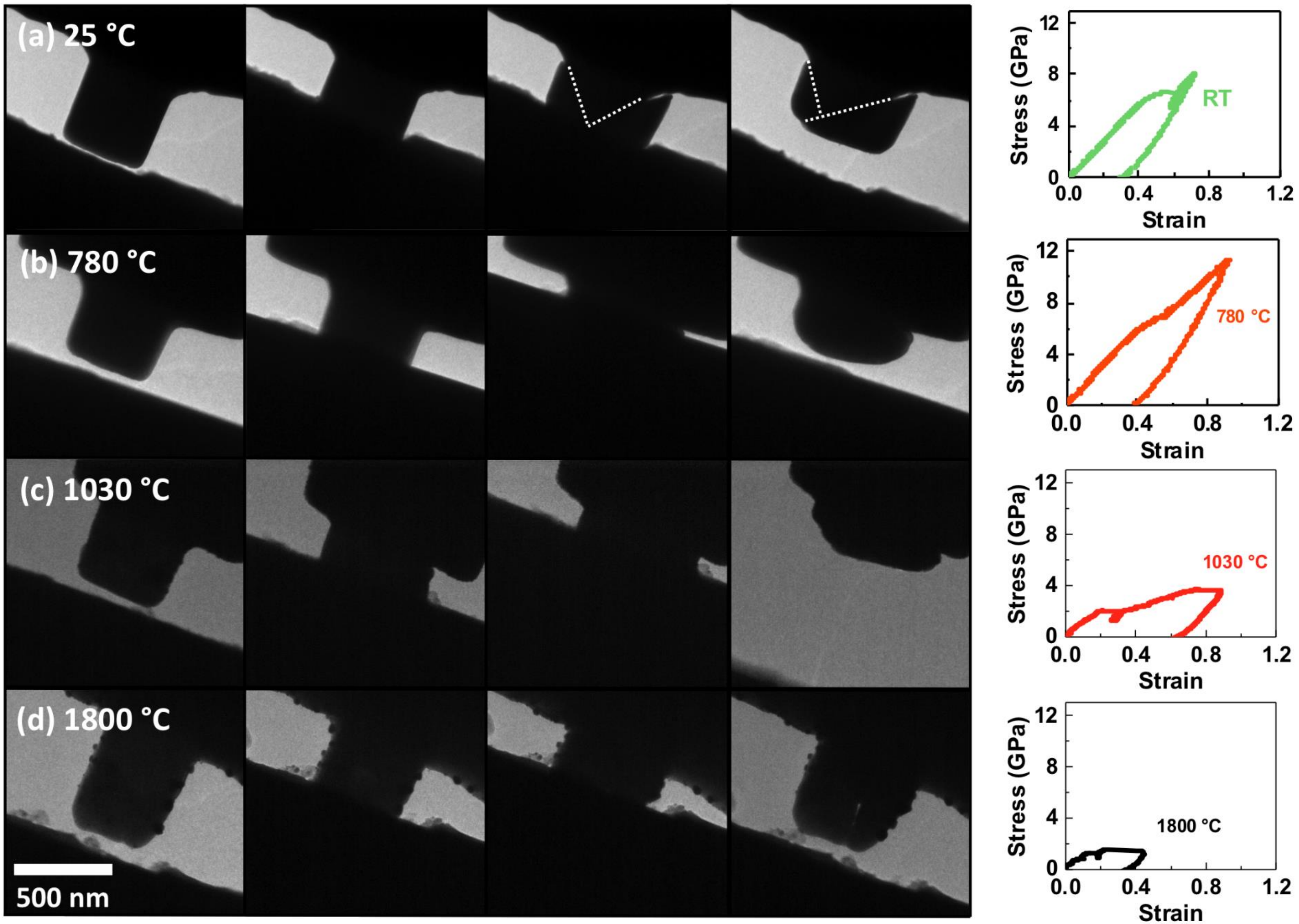






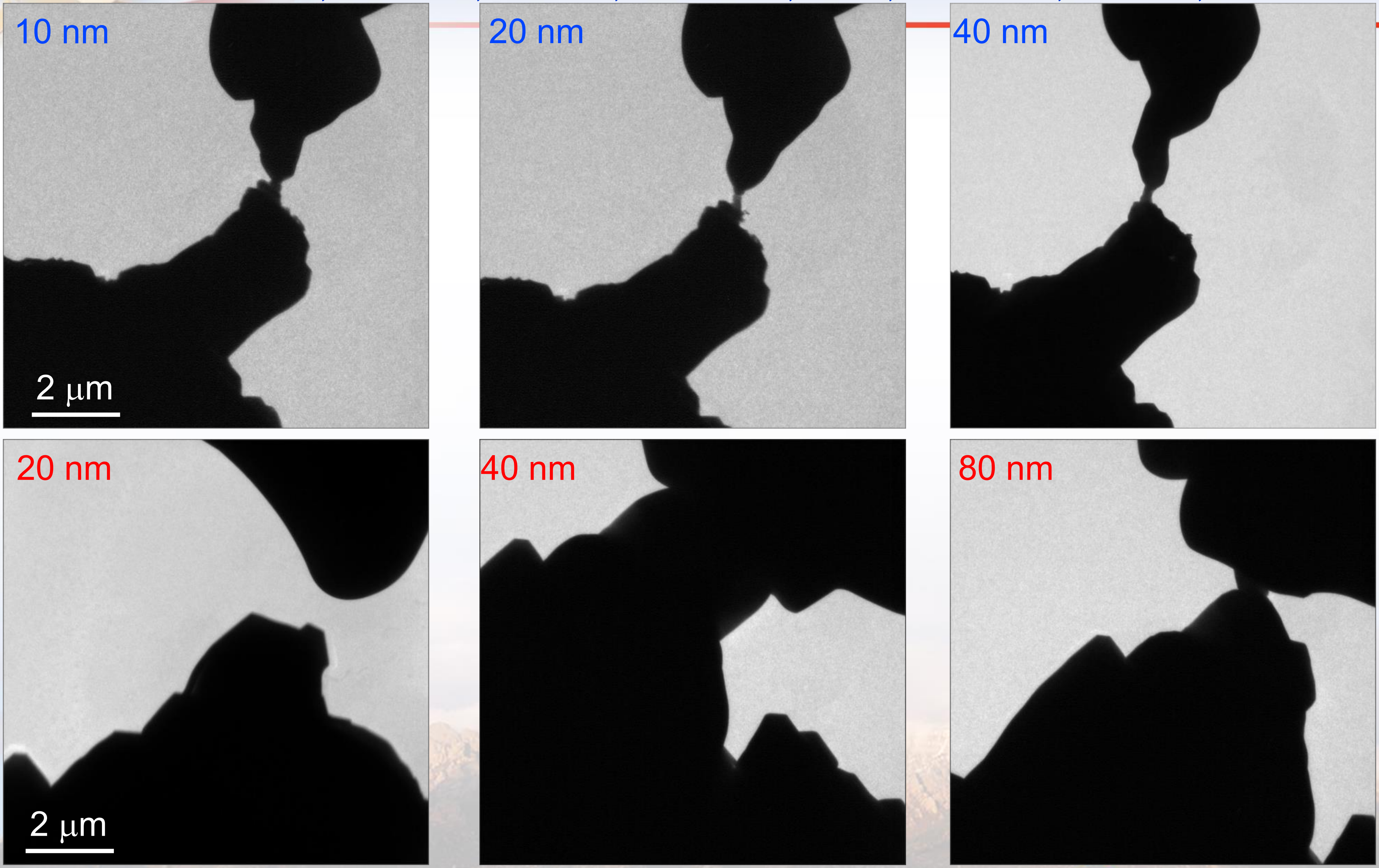
High Temperature Stress-Strain

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

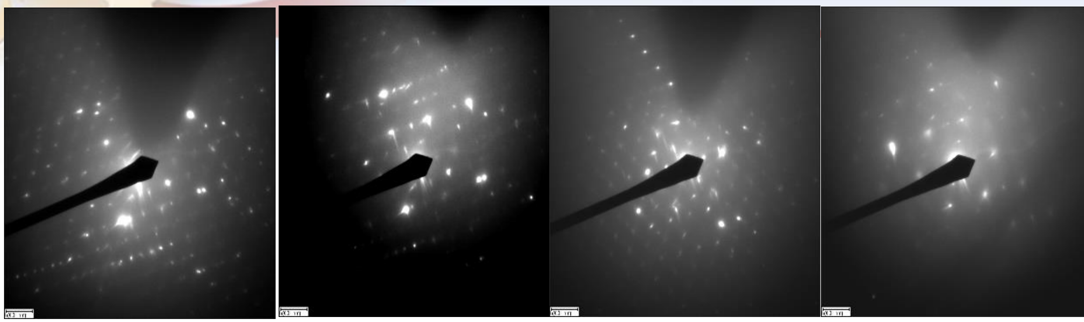


Pushing the Laser Limit – 1604 °C and 2056 °C ScSZ-ScSZ

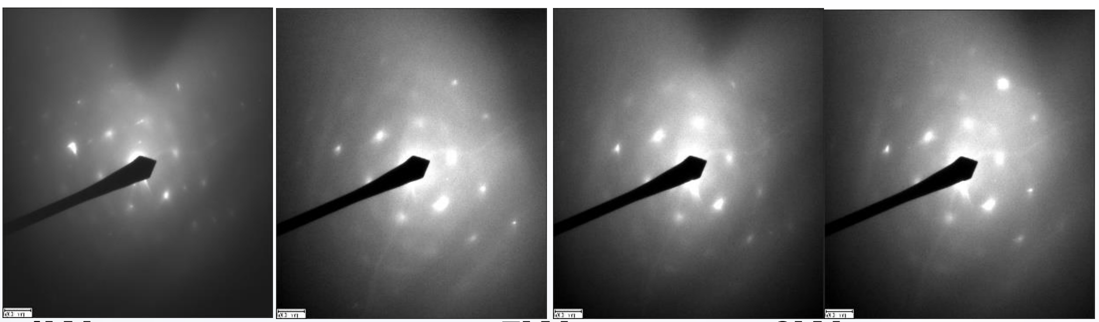
Contributors: R.L. Grosso, E.N.S. Muccillo, D.N.F. Muche, G.S. Jawaharram, C.M. Barr, A.M. Monterrosa, R.H.R. Castro, S.J. Dillon



Diffraction for Temperature Calibration

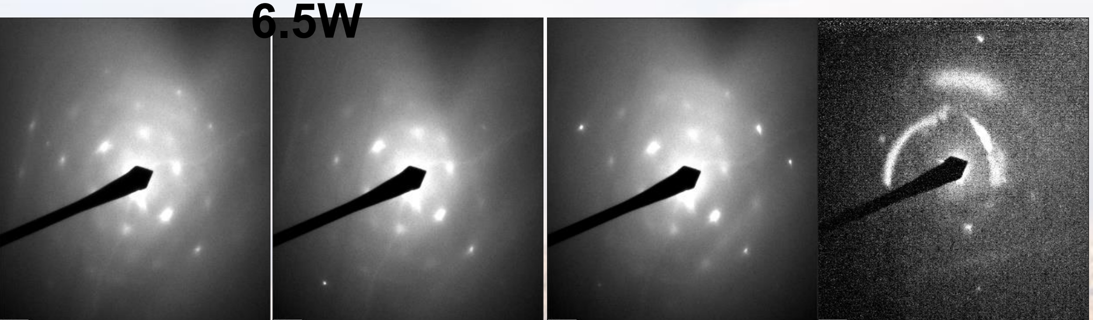


2W (β) 3W heating 3.5W (β)



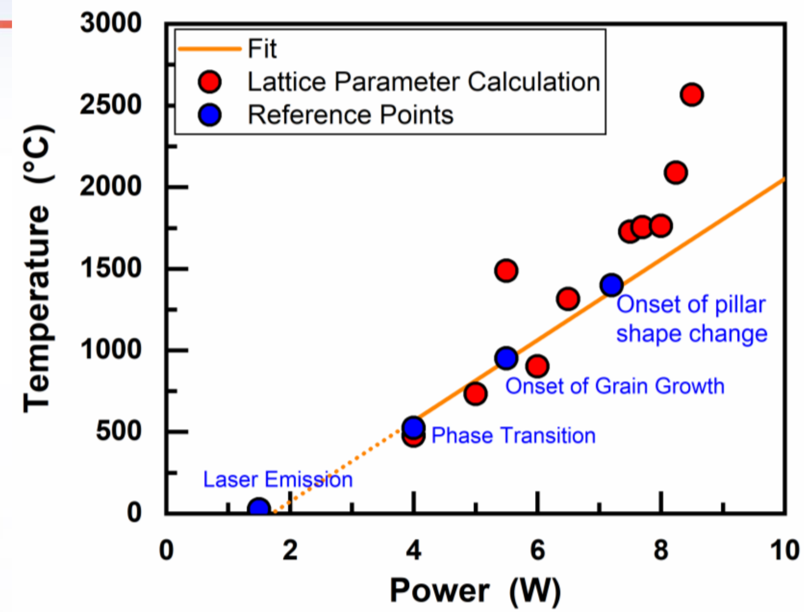
4W 5W 6W

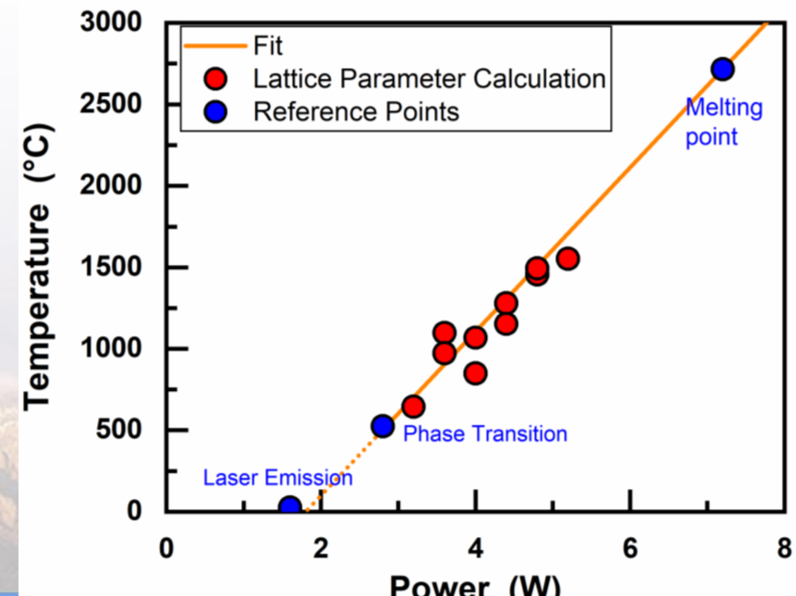
6.5W



7.7W 8W 8.5W

9.5W



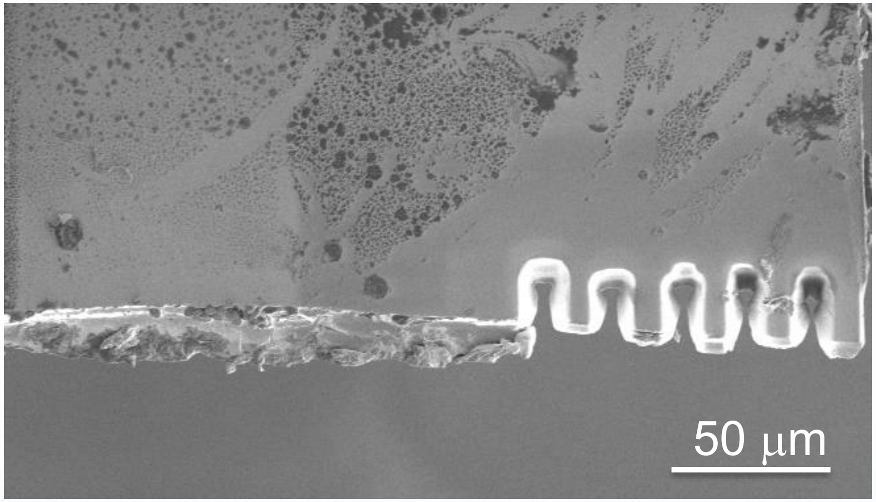


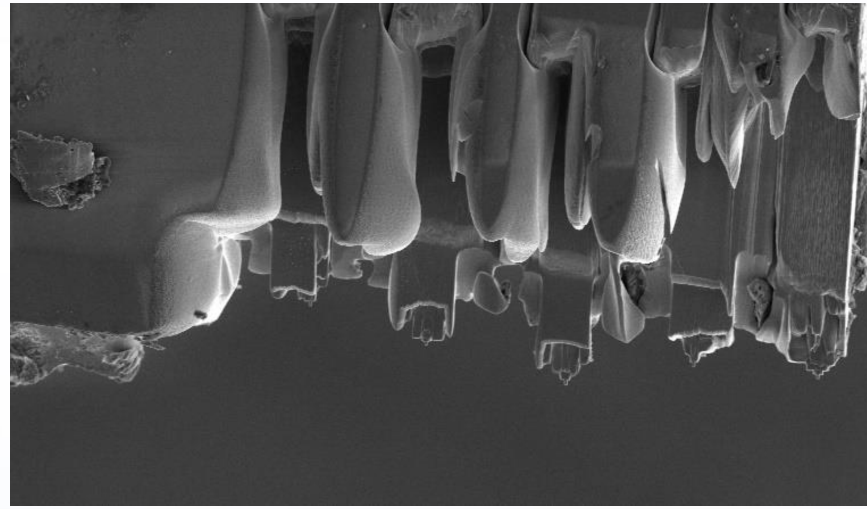
Temperature Upper Bound

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

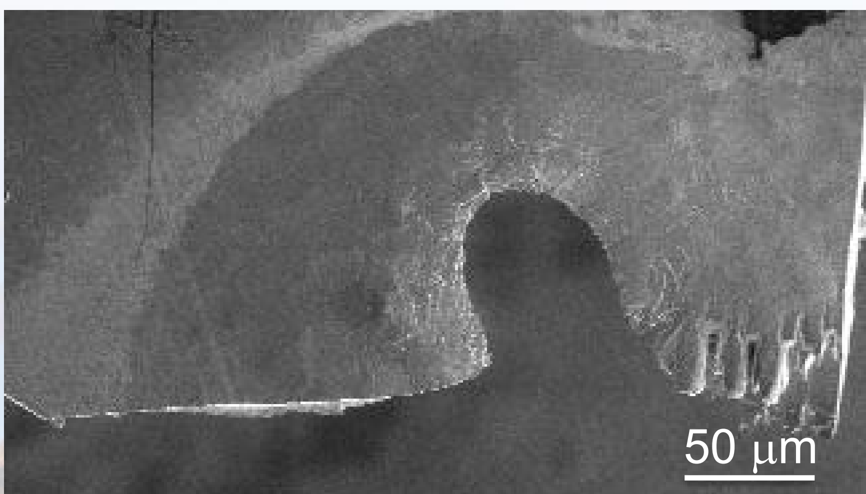
Before

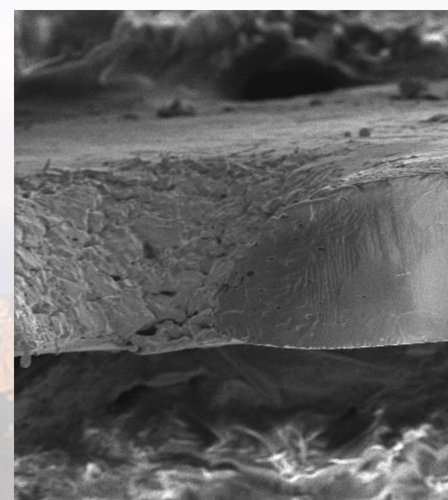
Sc_2O_3 doped ZrO_2

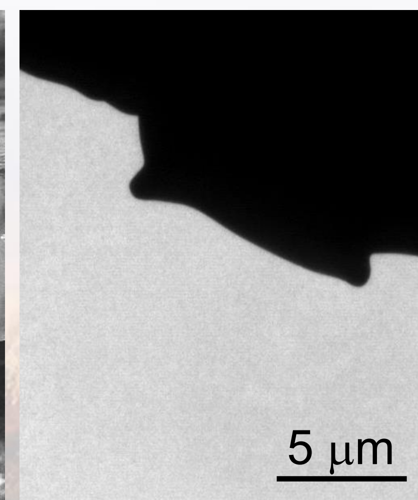




After

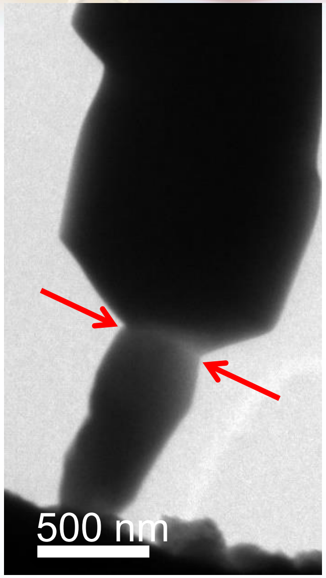


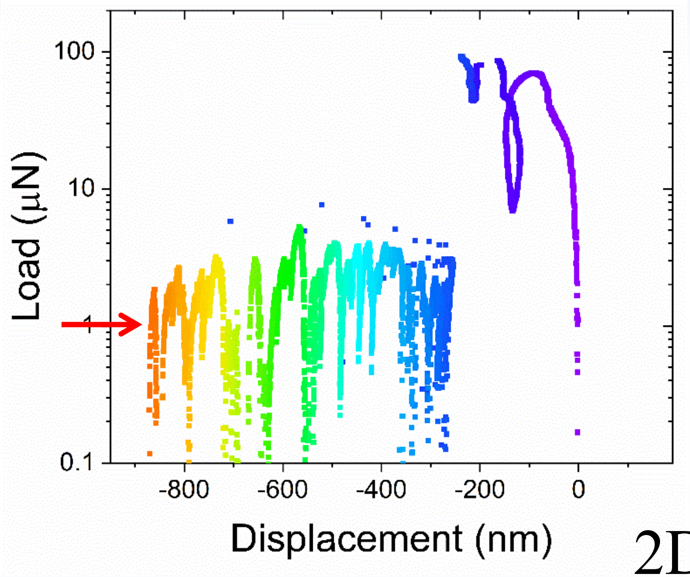


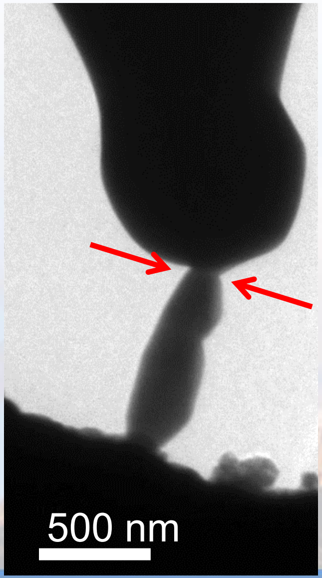


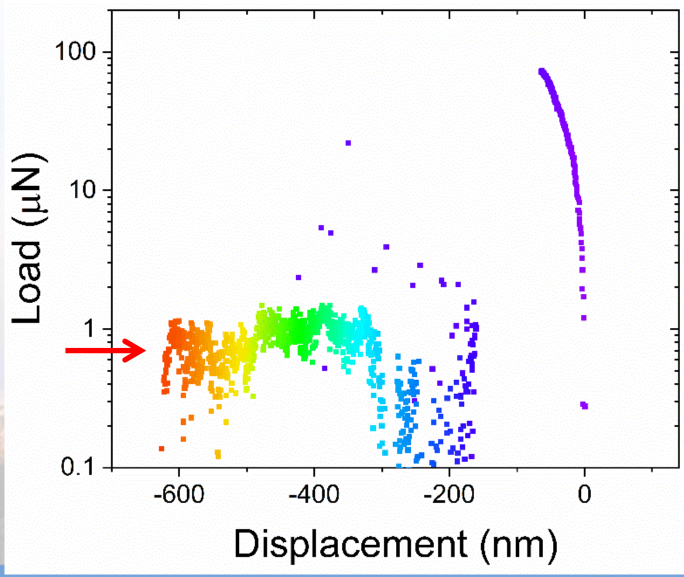
Mechanism for Fiber Growth

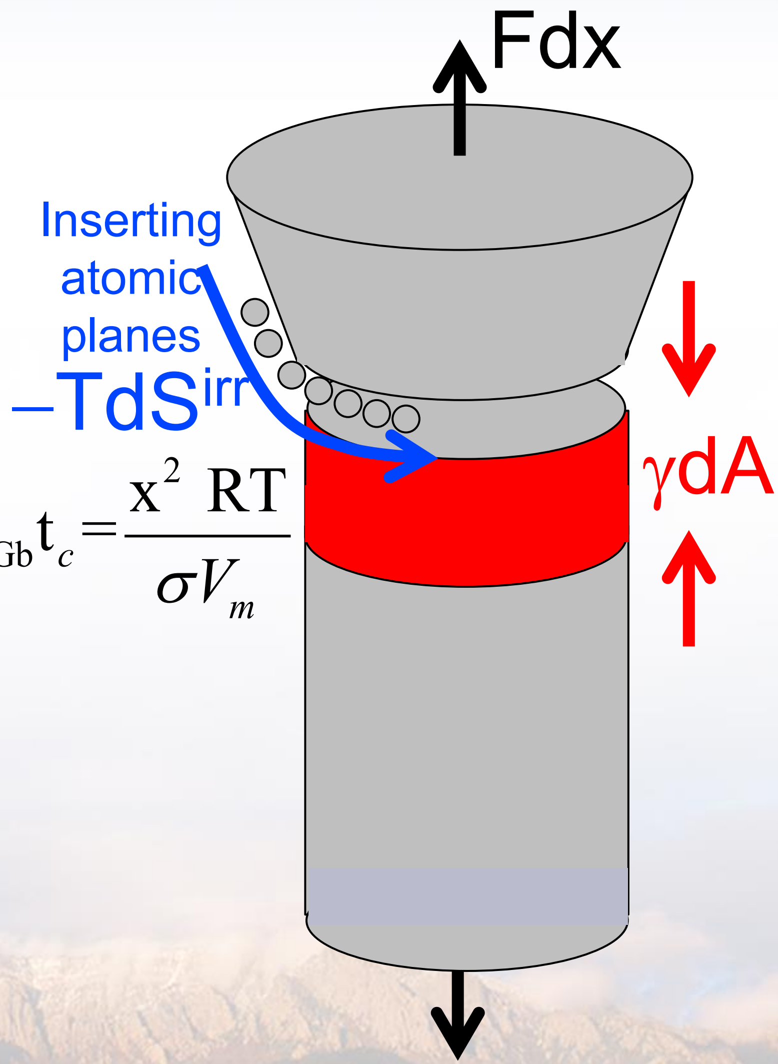
Contributors: R.L. Grosso, E.N.S. Muccillo, D.N.F. Muche, G.S. Jawaharram, C.M. Barr, A.M. Monterrosa, R.H.R. Castro, S.J. Dillon











$$2D_{Gb}t_c = \frac{x^2 RT}{\sigma V_m}$$

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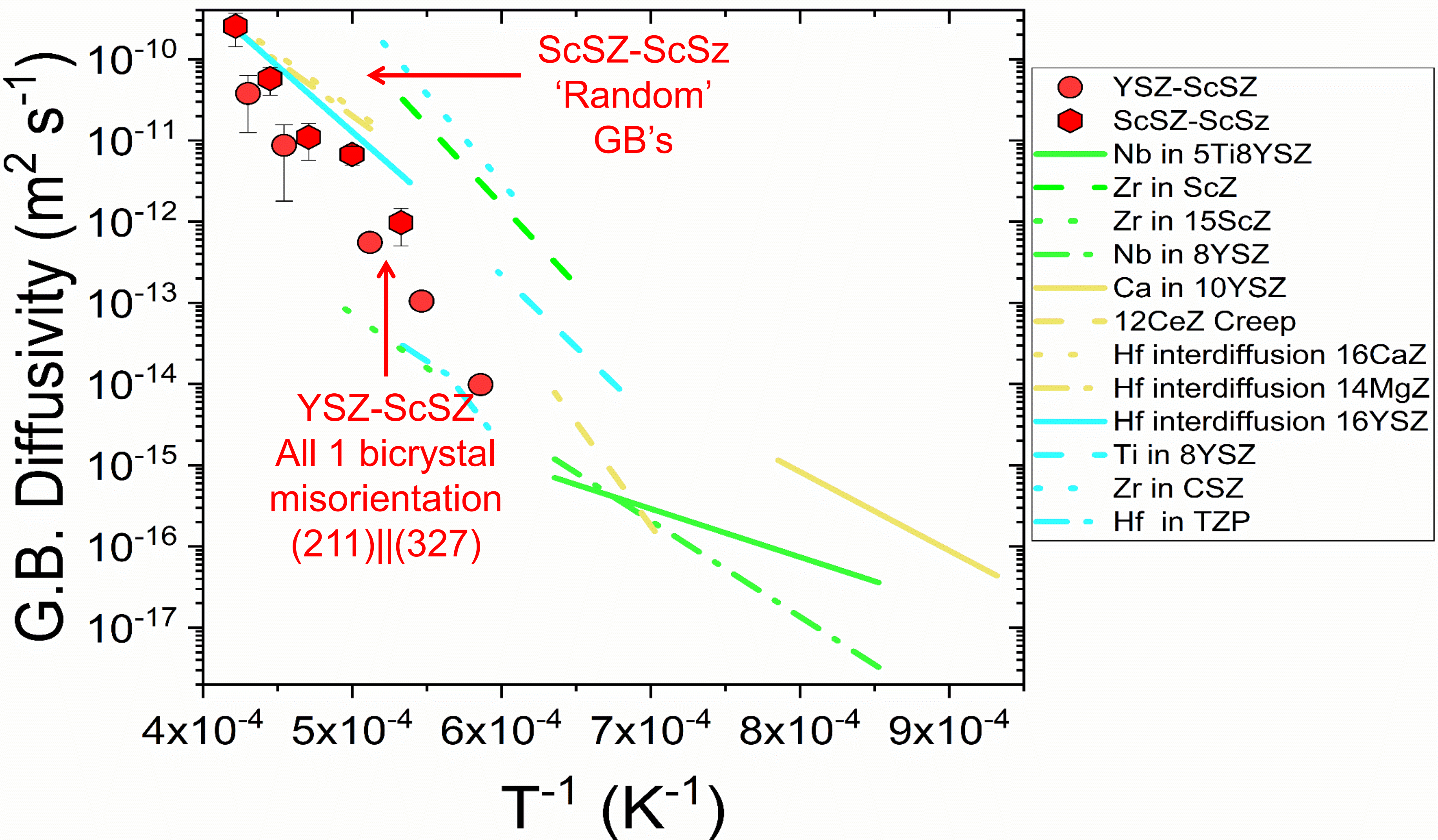
Calculated G.B. Diffusivity Compared to Literature

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

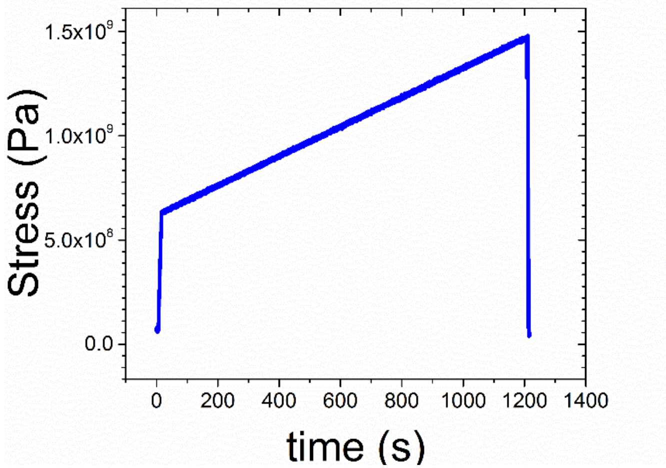


In situ TEM Ion Irradiation + Mechanical Testing = *In situ* TEM Irradiation Creep

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

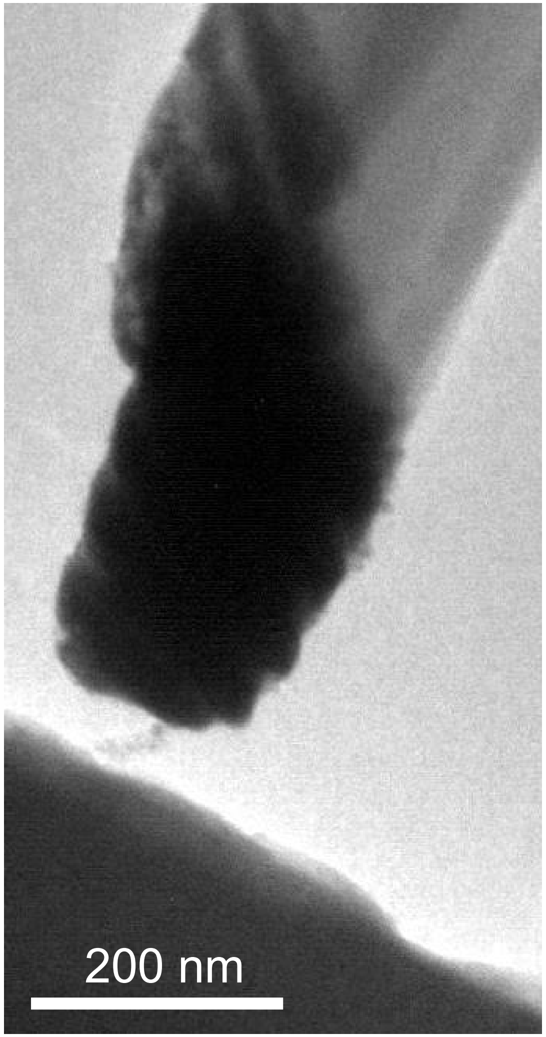
Controlled Loading Rate Experiments

4 MeV Cu^{3+}
10^{-2} DPA/s

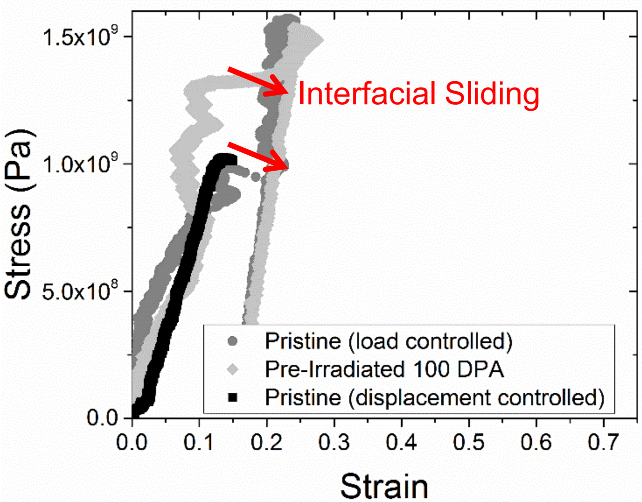


In-situ TEM radiation creep is feasible!

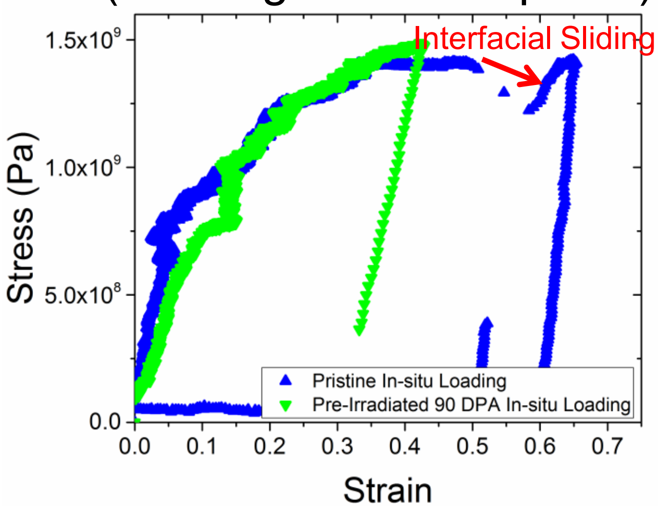
50 nm Cu-W multilayer
20 Min



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

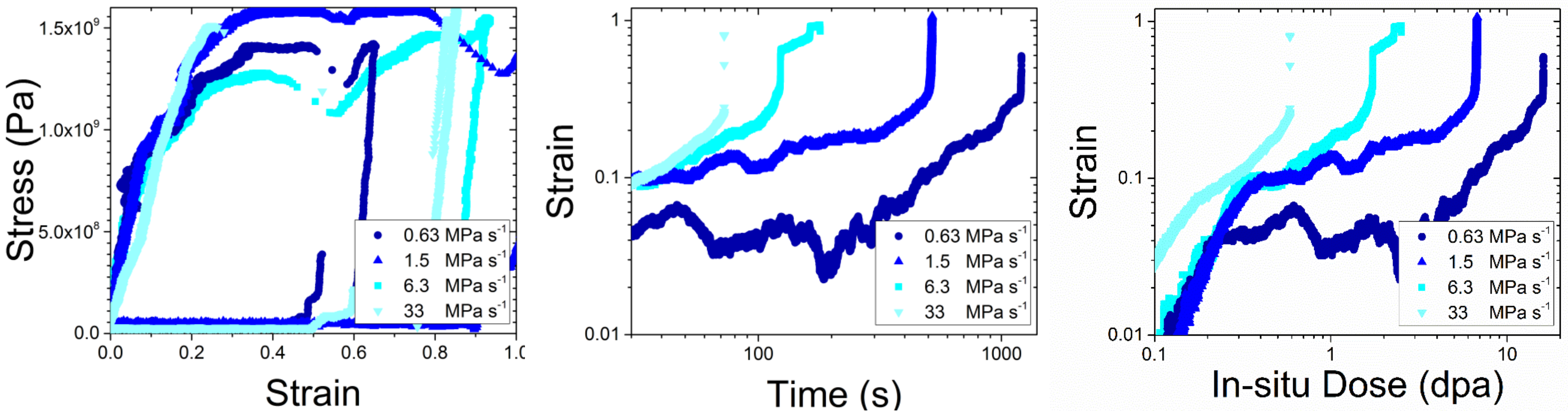


Irradiation Creep
(Loading rate 0.6 Mpa s^{-1})



Creep Response at Different Loading Rates

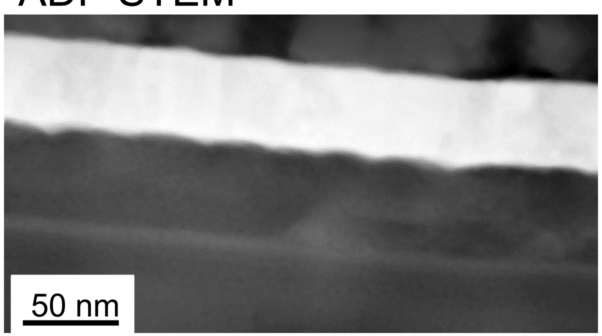
Contributors: G.S. Jawaharram, S. Dillon & R.S. Averback



Significant creep observed at a fraction of the bulk yield strength

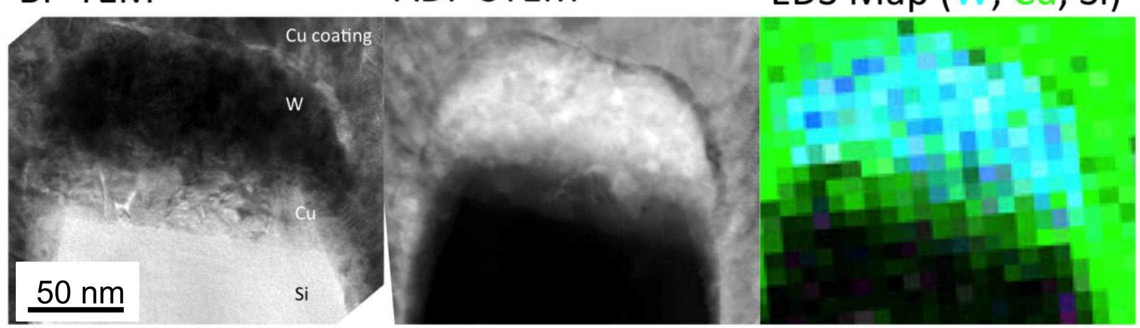
As-deposited Sample

ADF-STEM



Post Creep Characterization

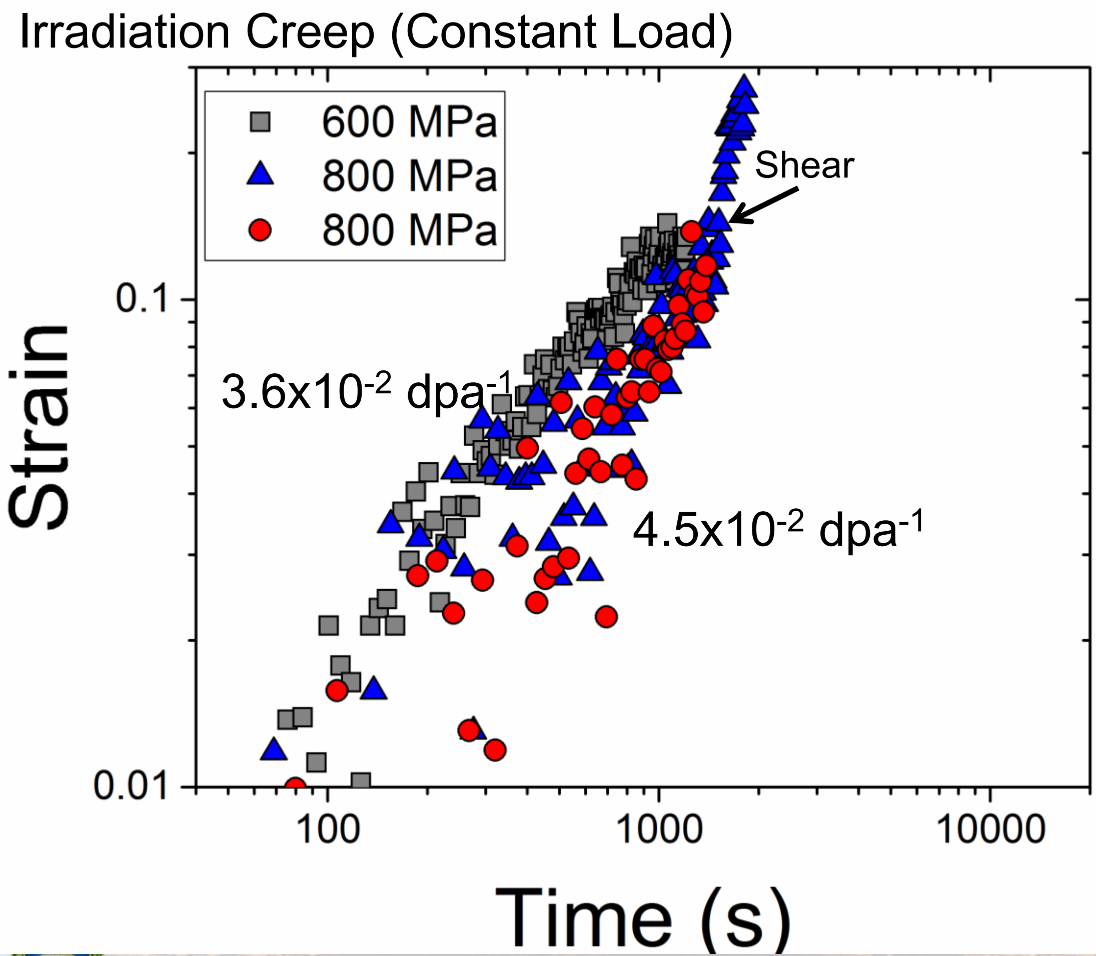
BF-TEM ADF-STEM EDS Map (W, Cu, Si)

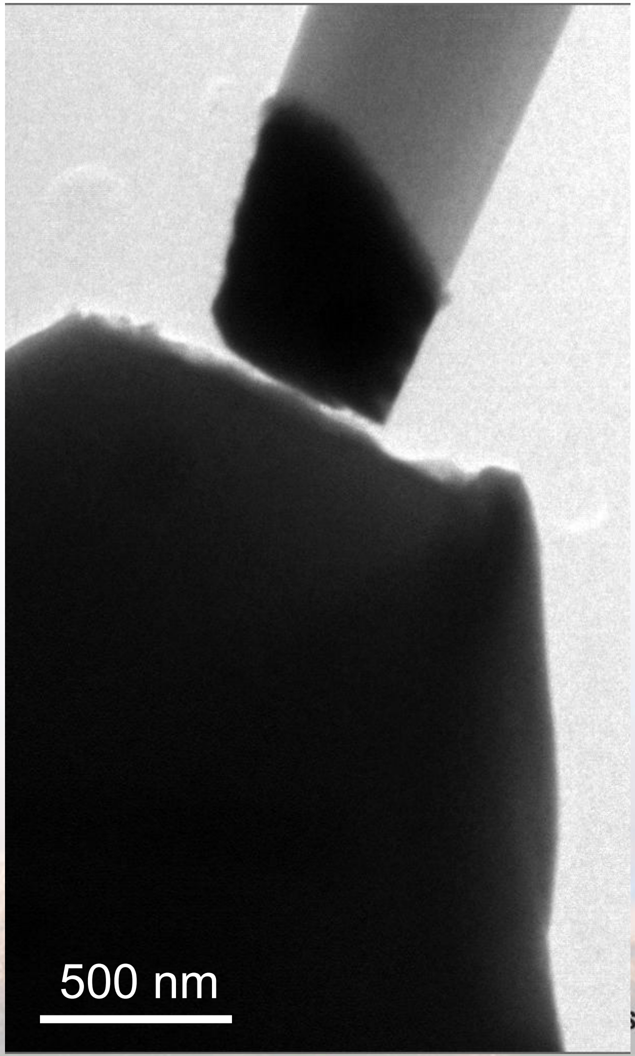


Compression (creep) only observed in Cu layer

Steady-State Irradiation Creep at Constant Load Cu-W

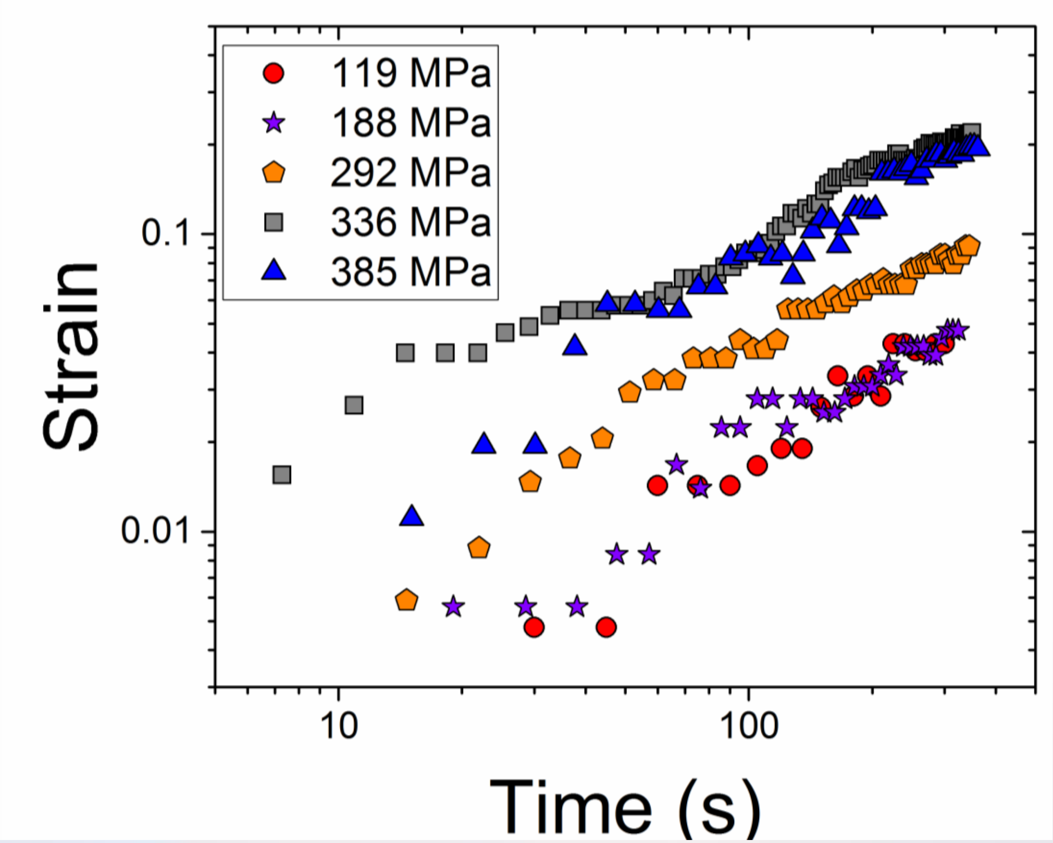
Contributors: G.S. Jawaharram, S. Dillon & R.S. Averback

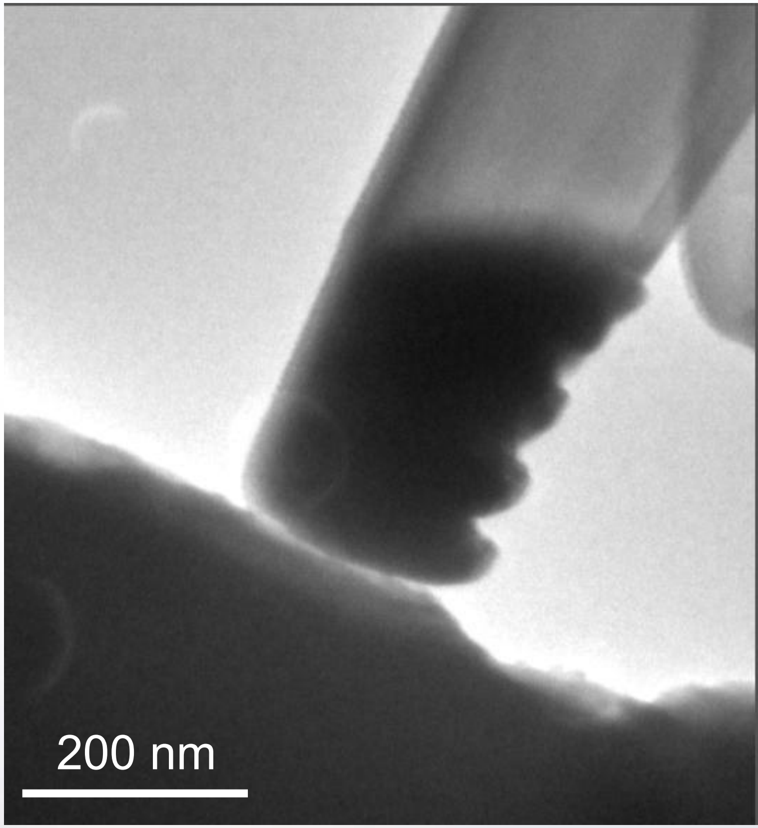




Ni-Ag Multilayer Irradiation Creep at Constant Load

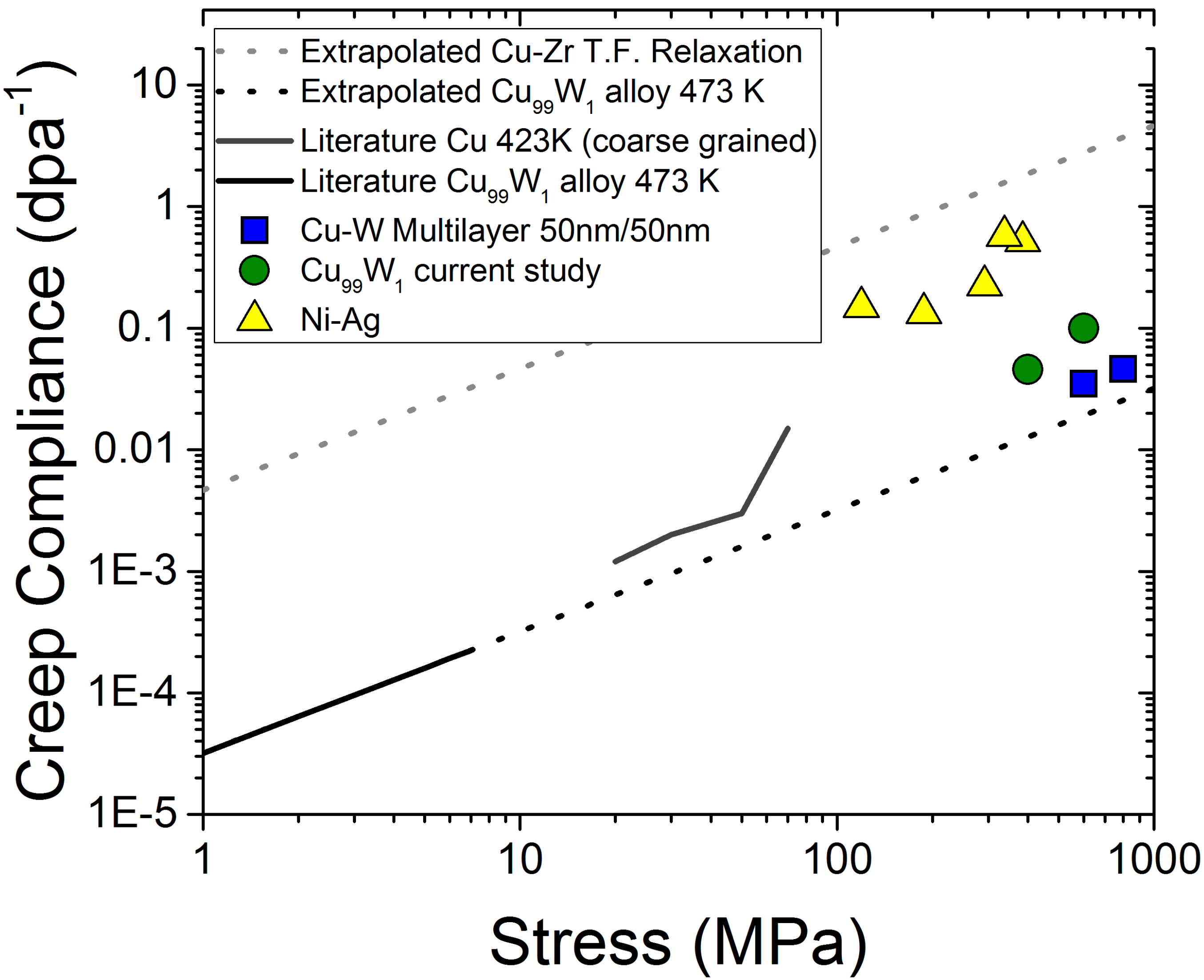
Contributors: G.S. Jawaharram, S. Dillon & R.S. Averback





Comparison of Creep Data

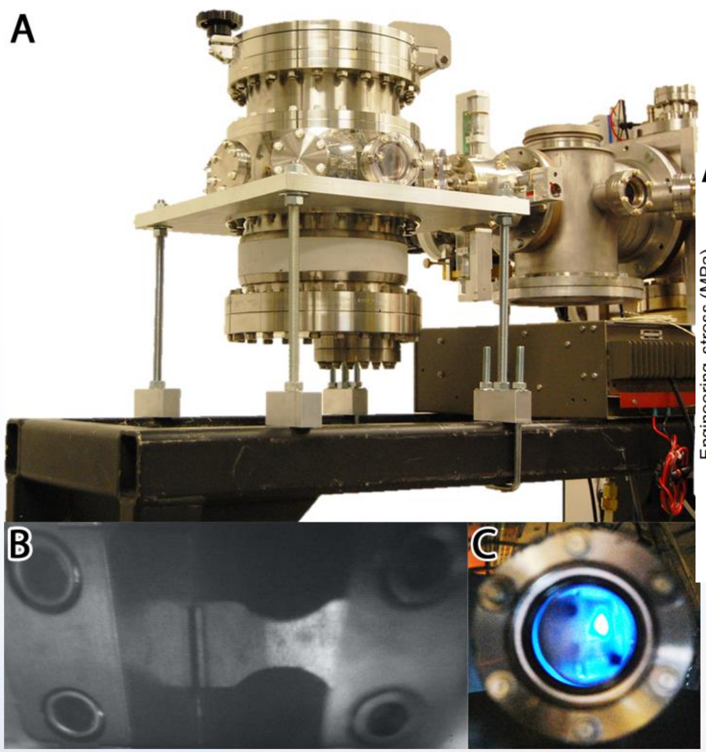
Contributors: G.S. Jawaharram, S. Dillon & R.S. Averback

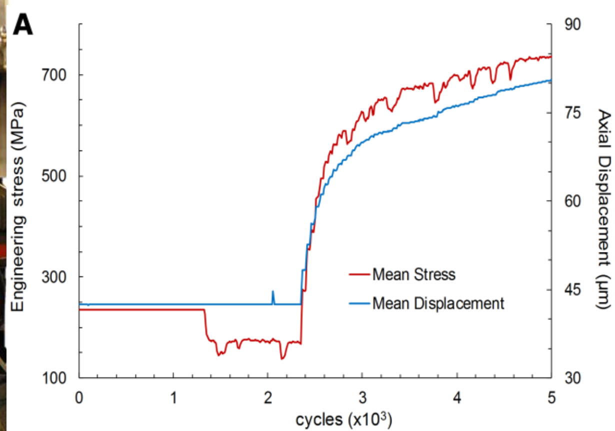


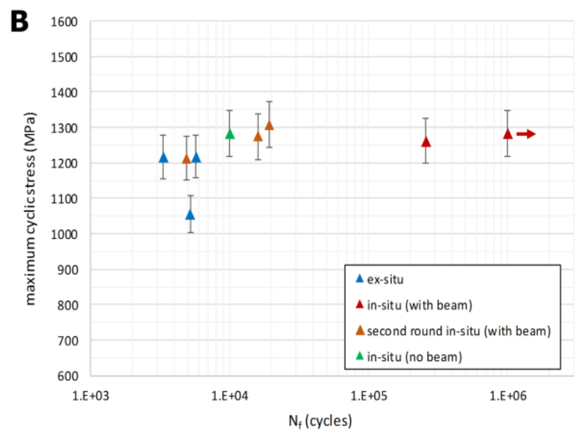
Ex situ Mechanical Testing End Station

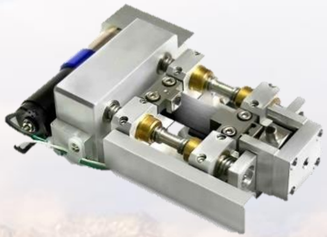
Collaborators: D. Buller, B. Boyce, J. Carroll, P. Price, C. Taylor, B. Muntifering, S. Briggs, N. Heckman, J.A. Scott

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



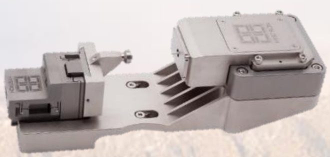


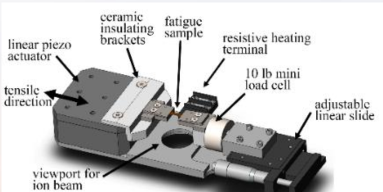




MTI Fullam Straining Heating

Hysitron PI85 Nanoindenter





Custom-built Piezo Fatigue tester

Gatan Cryostage

In situ Ion Irradiation SEM (I^3SEM) Vision

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

In situ SEM by Design

- Field Emission Gun
- Energy 500 eV to 30 keV
- Resolution:
  - 30 kV 1.5 nm
  - 1 kV 4nm
- Mag 5x to 600kx
- Pressure 10-150 Pa
- Sample dimension: 200 mm diameter x 80 mm height
- 90 tilt and 360 rotation
- 12 Ports
- Hysitron PI-85
- MTI/Fullham Heating Straining Stage
- Custom Piezo Fatigue Stage

Proposed Future Capabilities

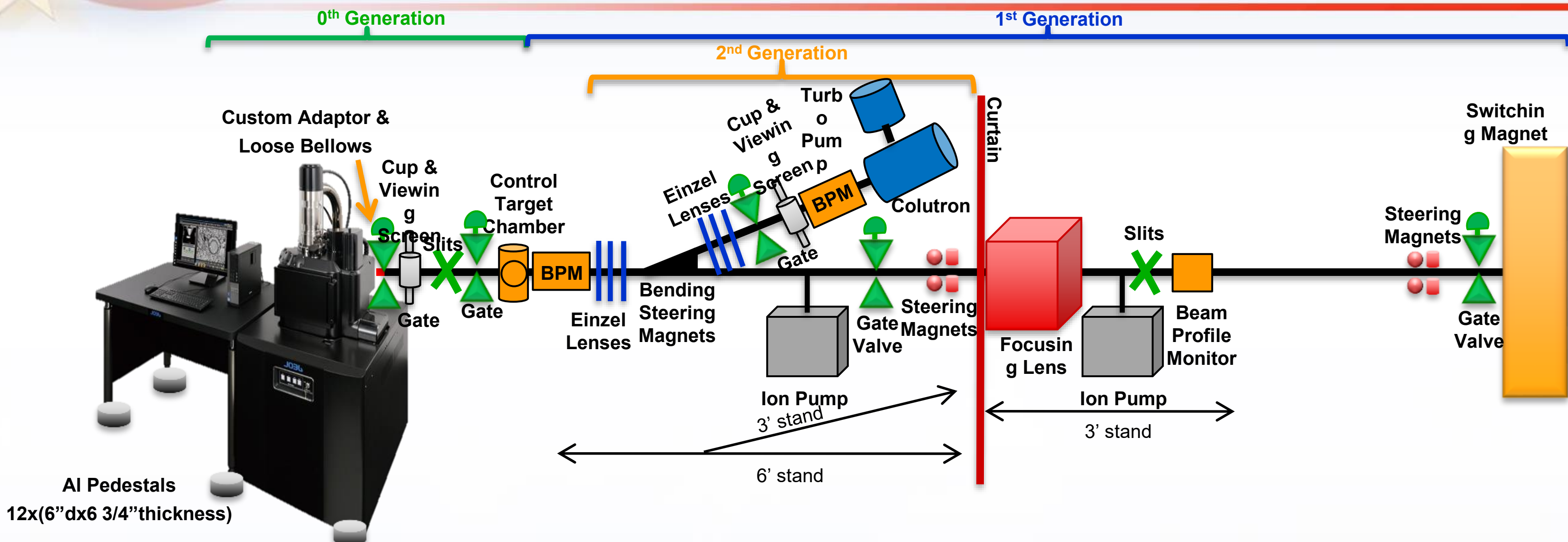
- Low Pressure BSED
- Heating Stage
- Peltier cooled Cryo stage
- High Speed EBSD
- High Speed EDS
- Low energy ion source
- PL/CL/IBIL
- FIB
- Hot/Warm Cell capability
- TKD/STEM detector
- 3D measurement software
- Liquid environments
- Gas injection
- Electron Lithography

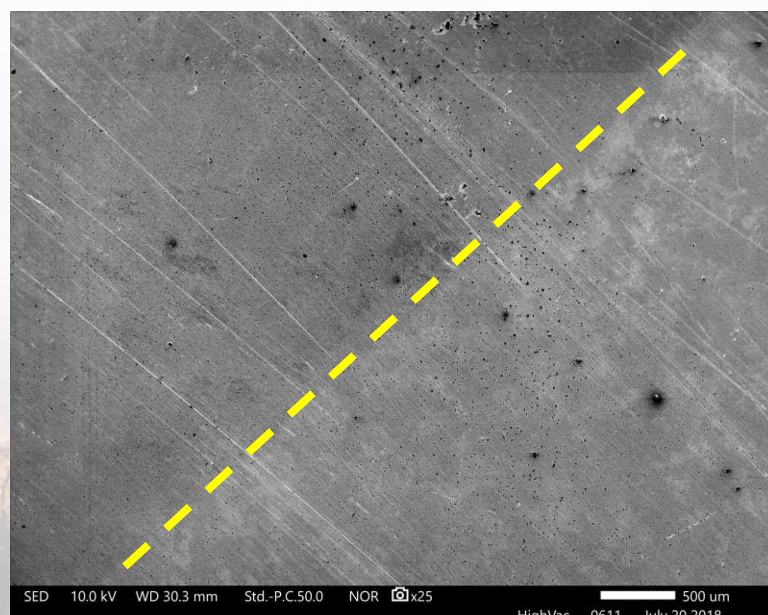
JEOL
JSM-IT300HRLV

We are designing this to be the world's best *in situ* SEM for overlapping extreme environments

Heavy Ion Design and Proof of Concept

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





I^{3}SEM planned for multiphase development. Ultimate plan is for multiple accelerators being attached for dual or triple beam experiments.

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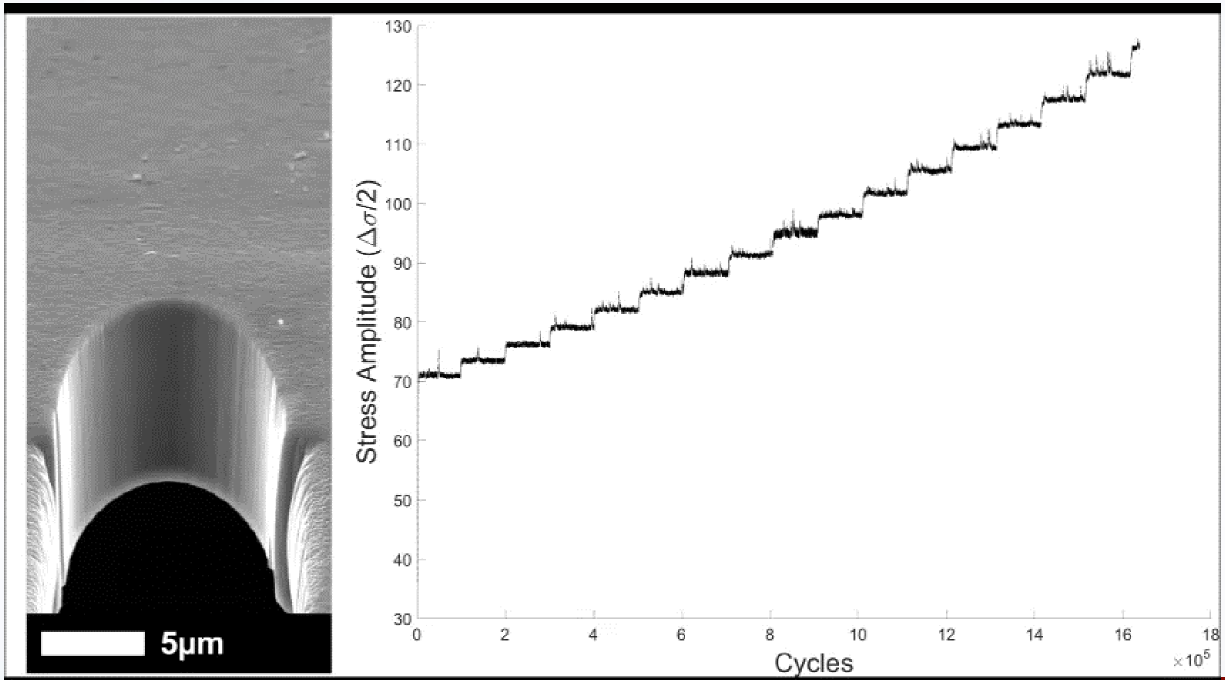
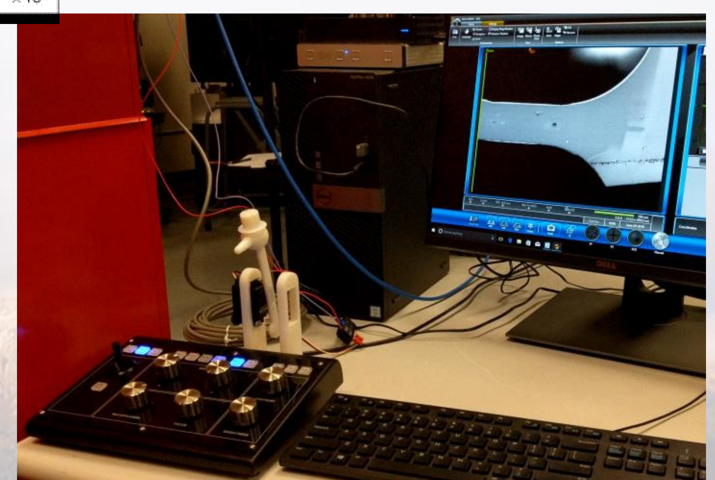
In situ High-cycle Fatigue

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



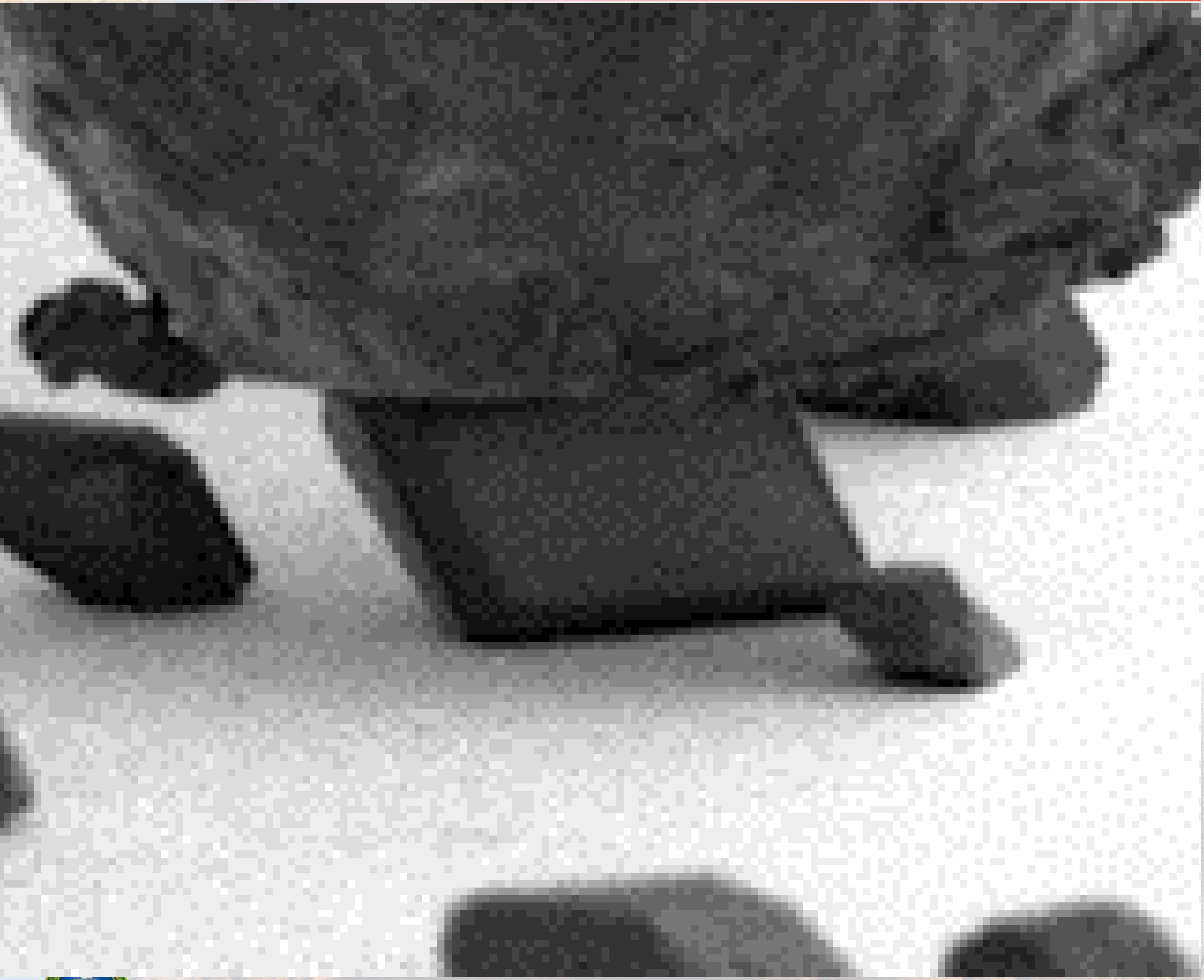
- Nanocrystalline Ni-40Fe, 10-60 nm grain size, 10 μm notch, imaged at 60˚
- Cycled at 30 Hz, 4000 cycles between images

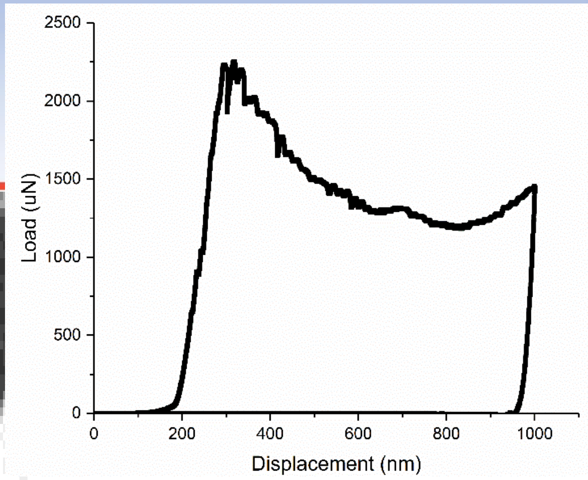
Direct insight into crack propagation and failure during cyclic loading

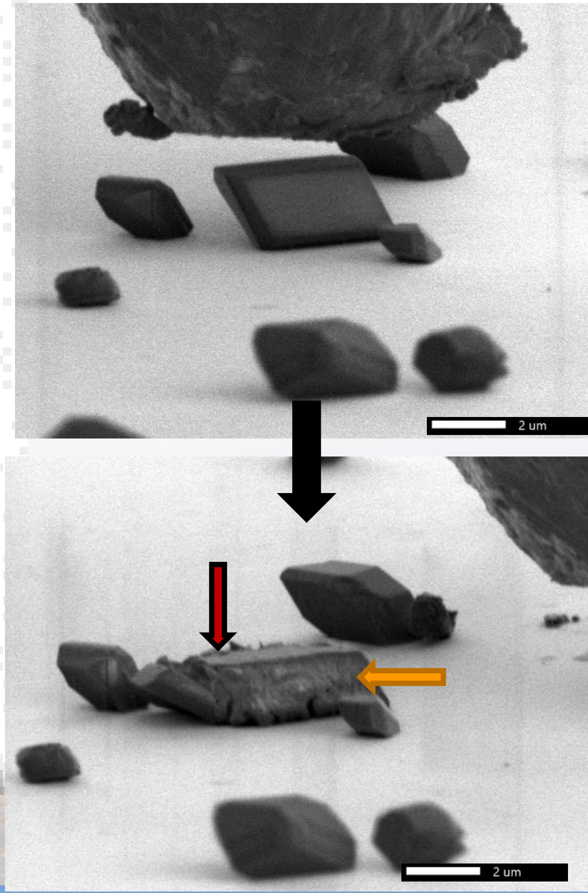


In situ Compression: Molecular Crystals

Collaborators: C.M. Barr, M. Cooper, D.C. Bufford, and J. Lechman



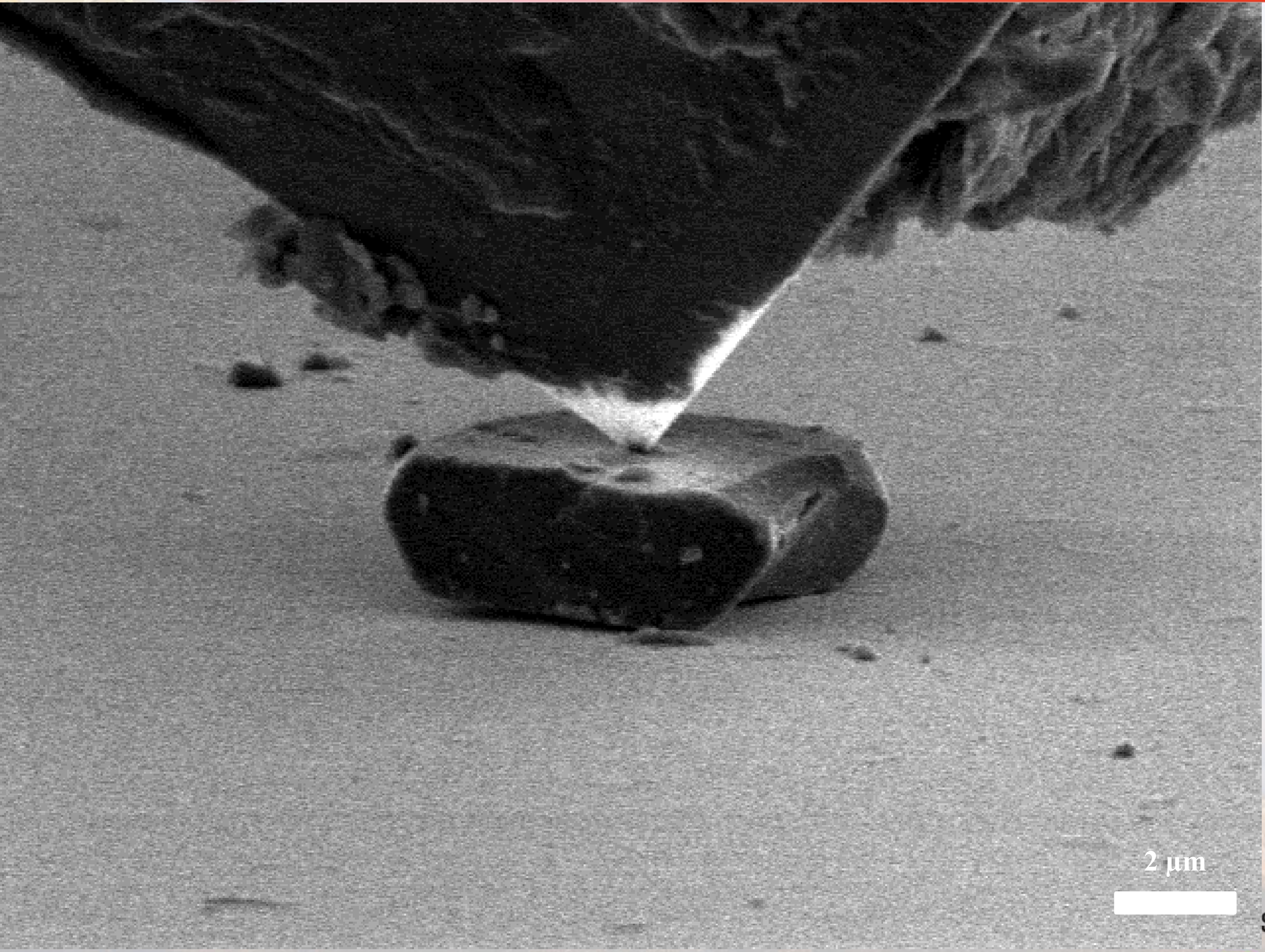




Displacement controlled fracture of molecular crystal

In situ Indentation: Ceramics

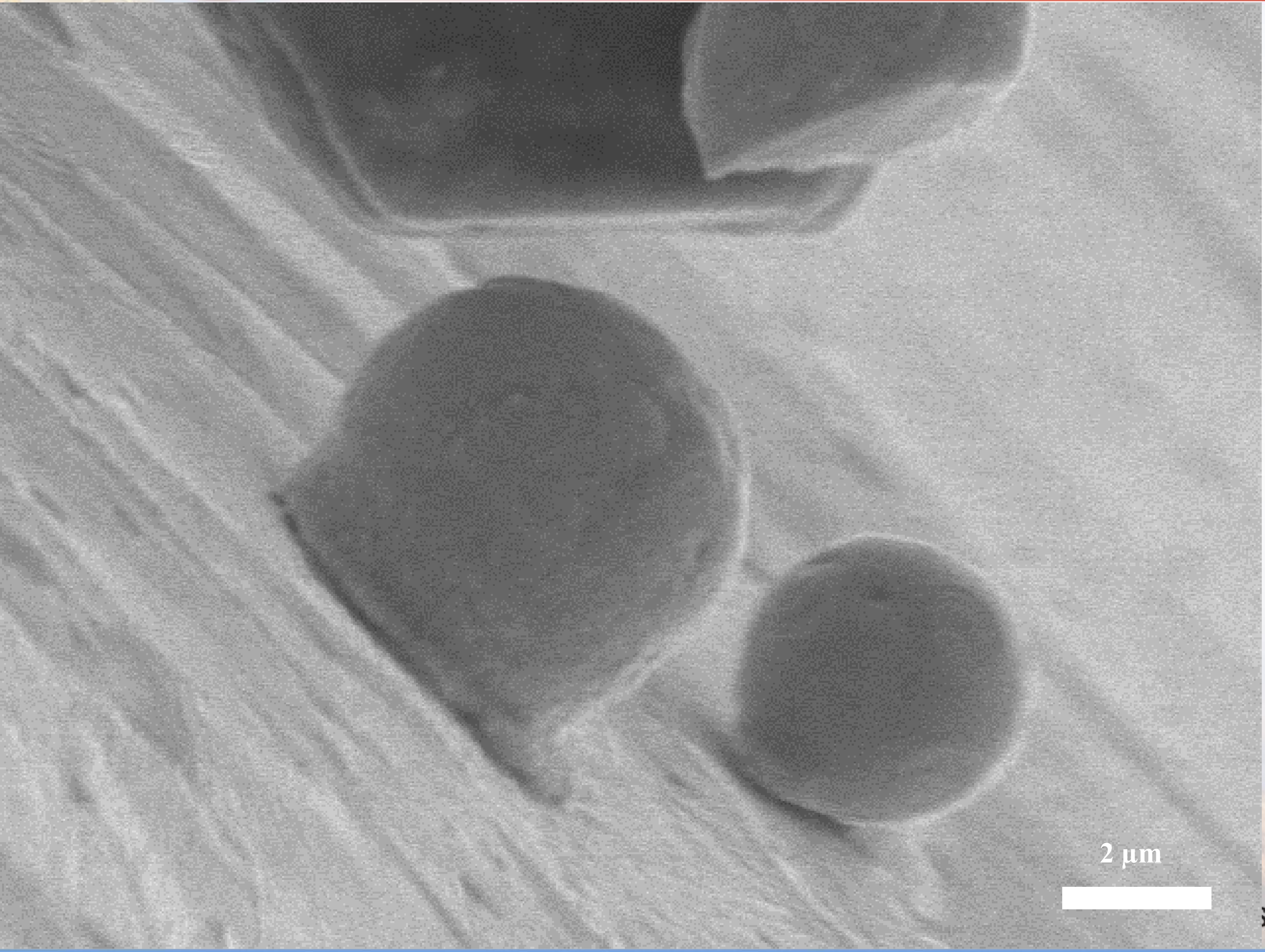
Collaborators: .N. Heckman, B.L. Boyce,



Alumina
30 mN peak load
3 mN/s load rate
1x speed

Angled *In situ* Compression: Steels

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



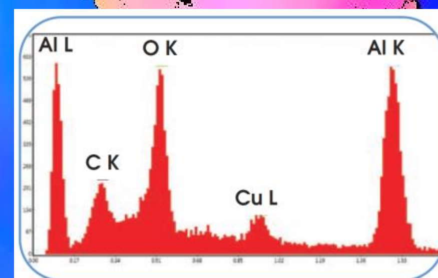
Kovar
5 micron displacement
0.5 micron/s
1x speed

Recently Installed High Speed EBSD and EDS

- EDAX Velocity™ EBSD Camera
  - Capable of fast acquisition (> 3000 indexed points per second)
  - High signal-to-noise ratio, phosphor screen optimized for high speed collection

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

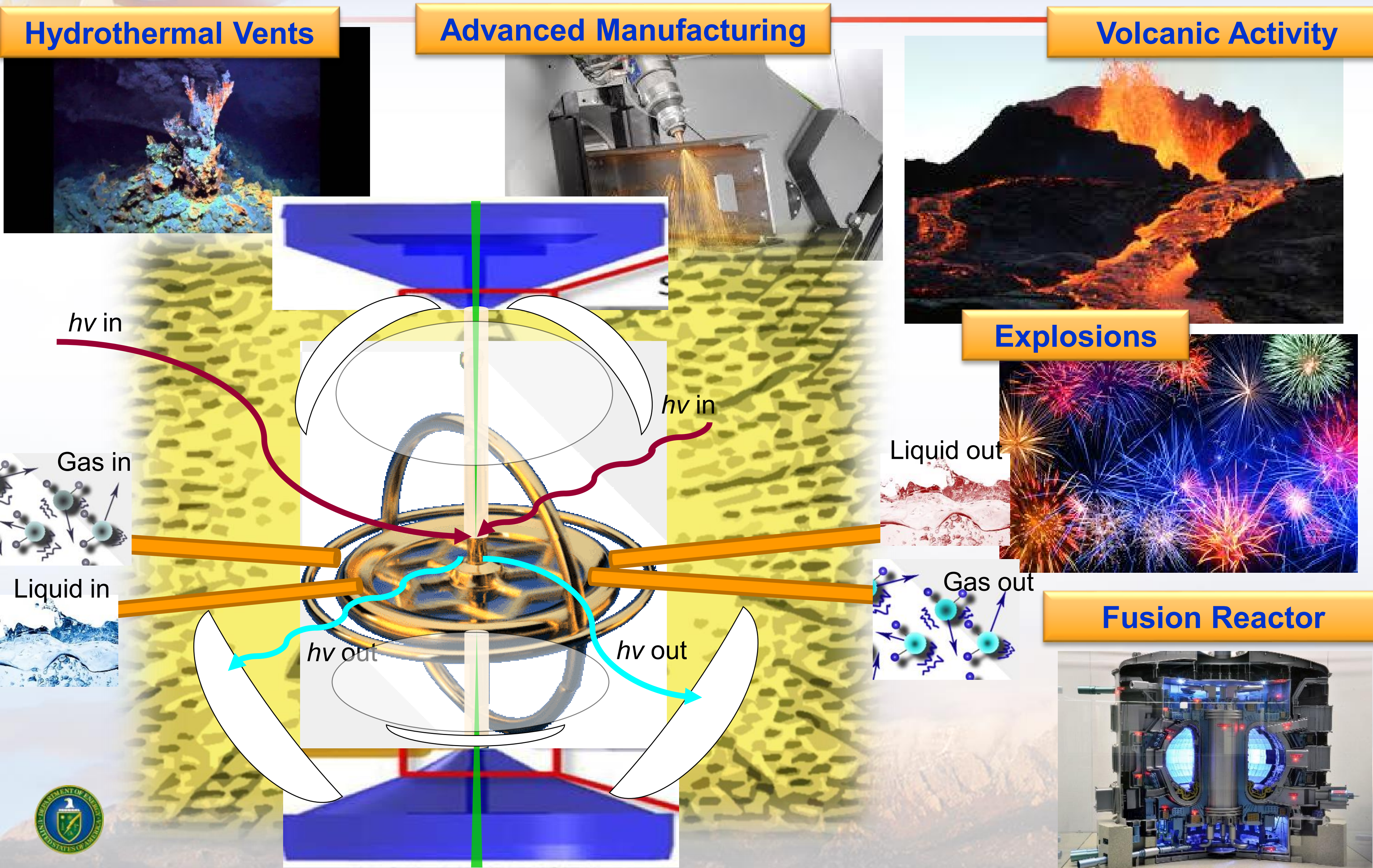
- EDAX Octane Elite EDS System
  - High light element sensitivity
  - High count & throughput rates



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

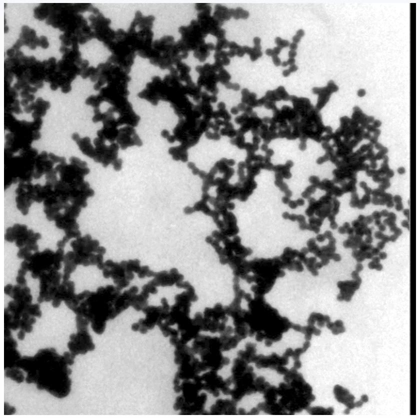
45 um

Future Vision:
Testing Greater Extremes in the TEM

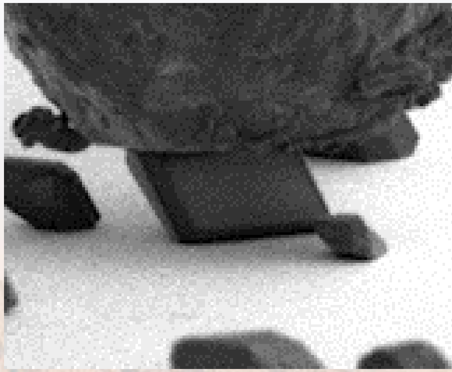


Unconventional *In situ* Microscopy Creates a Wealth of Possibilities

- 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

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Nuclear Science User Facilities

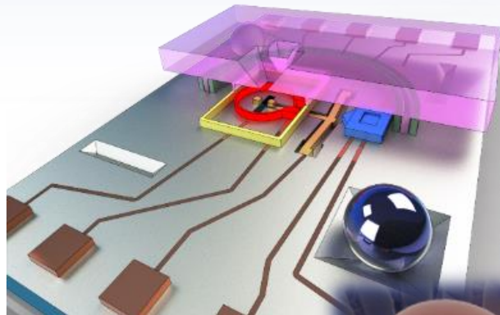
Sandia National Laboratories

Sandia's User and Position <u>Opportunities</u>

D. Hanson, W. Martin, M. Wasiolek

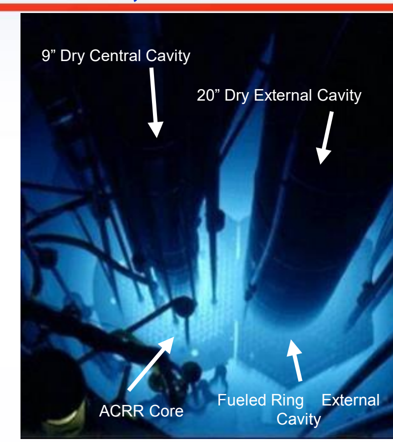
<u>www.cint.lanl.gov</u>

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



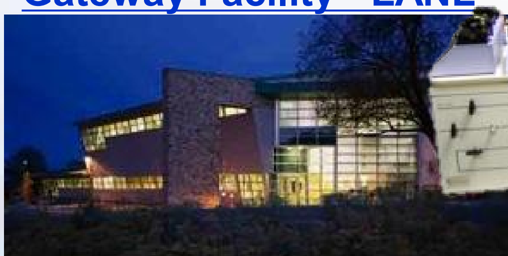
<u>www.nsuf.inl.gov</u>

- Three proposal a year for 9 months



<u>Core Facility - SNL</u>

<u>Gateway Facility - LANL</u>

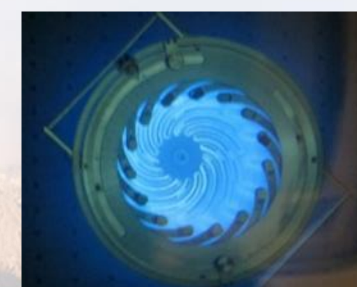


Position Opportunities at:

<u>https://www.sandia.gov/careers/</u>

Staff, Technical Support, Post-docs, and Summer 2022 Inters

khattar@sandia.gov



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.

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