



This paper describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government.

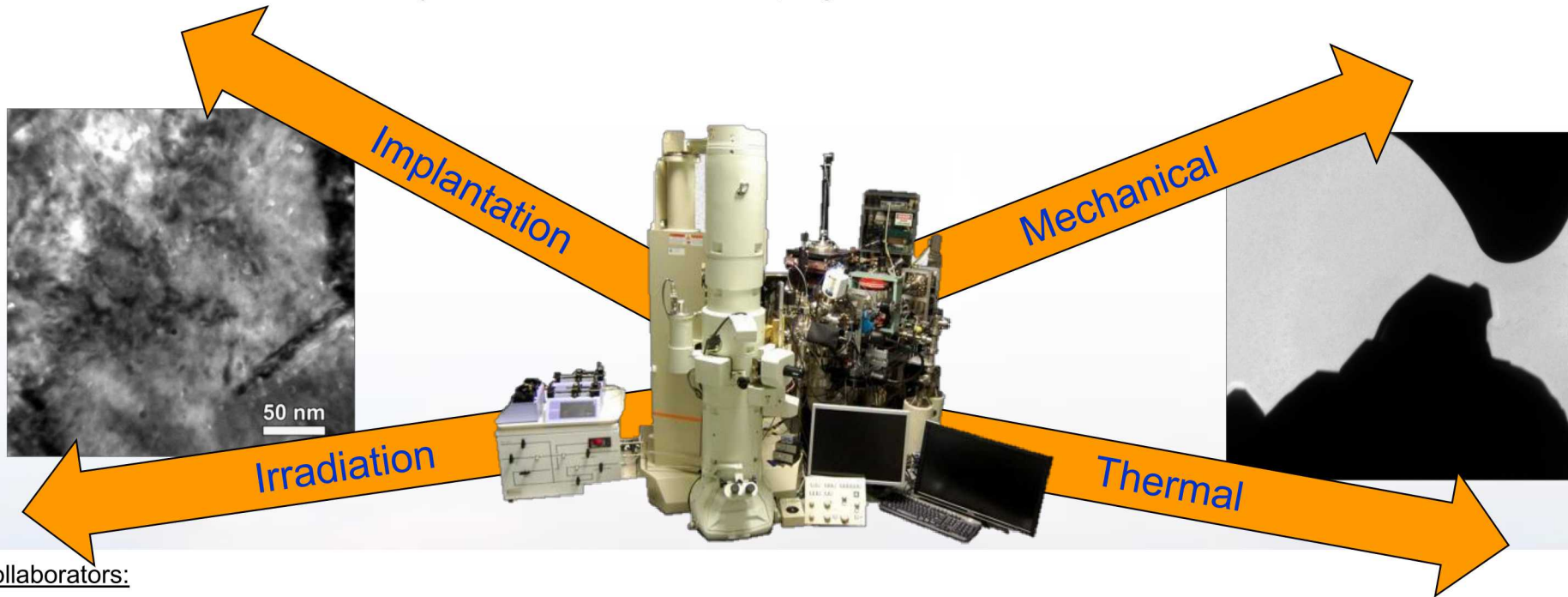
Exploring the Extremes of Mechanical Behavior through In-situ Electron Microscopy

SAND2020-2771C
Nuclear Science
User Facilities

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²University of Illinois Urbana Champaign, Urbana, IL 61801, 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. Averbach, 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 Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned sic Energy Sciences, U.S. Department of Energy. This work was | subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. 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.

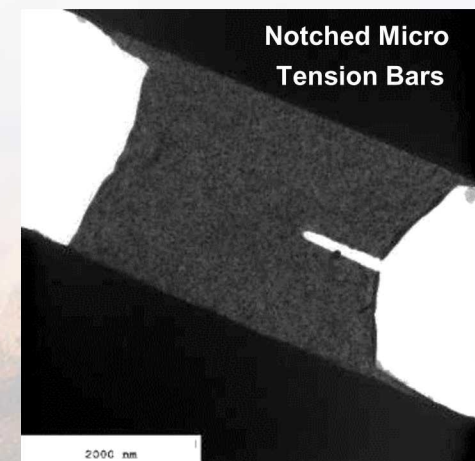
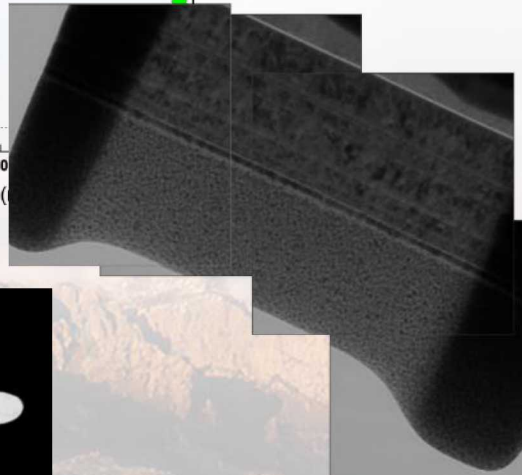
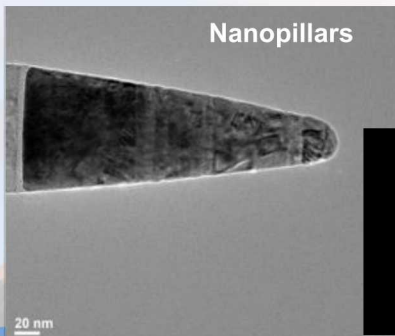
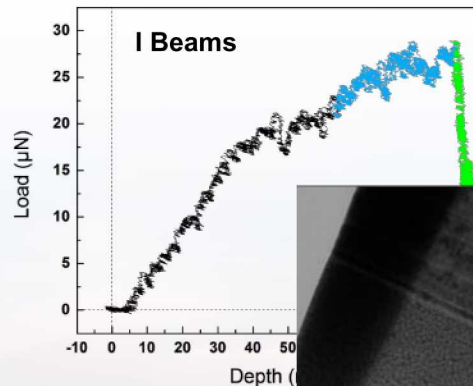
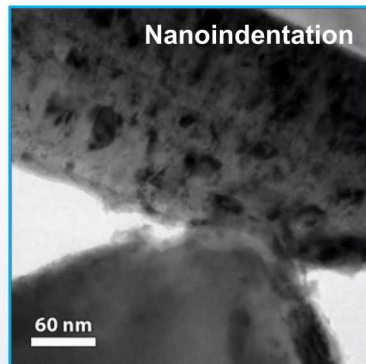
In situ Quantitative Mechanical Testing

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**

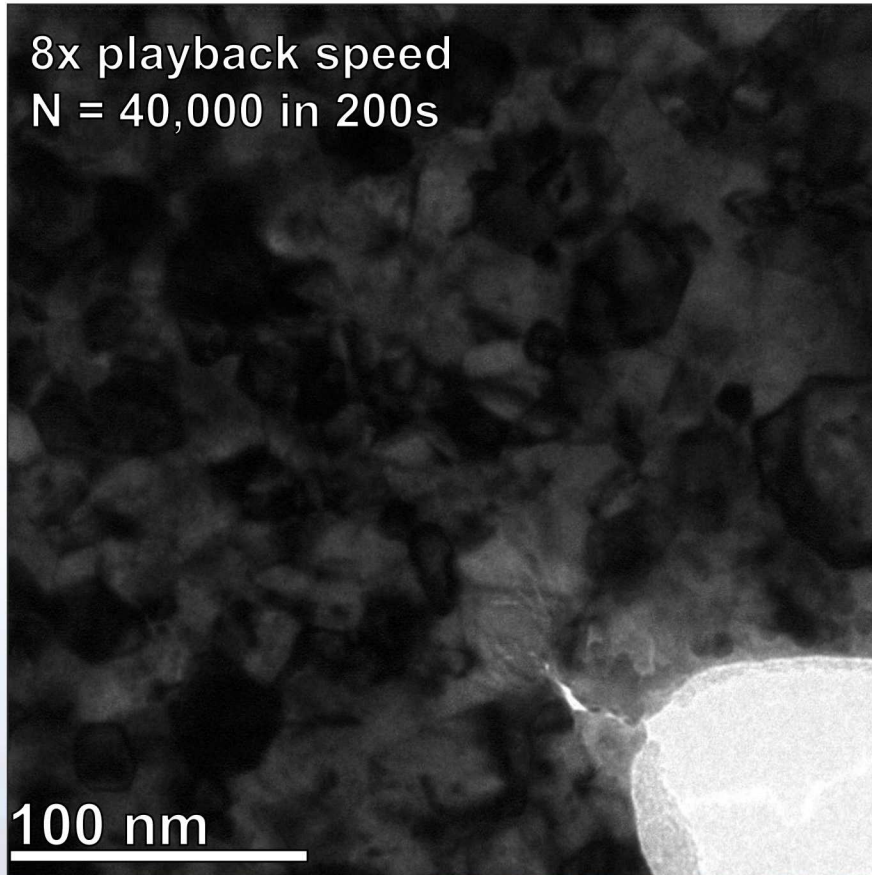


Fatigue Failure in Real Time?

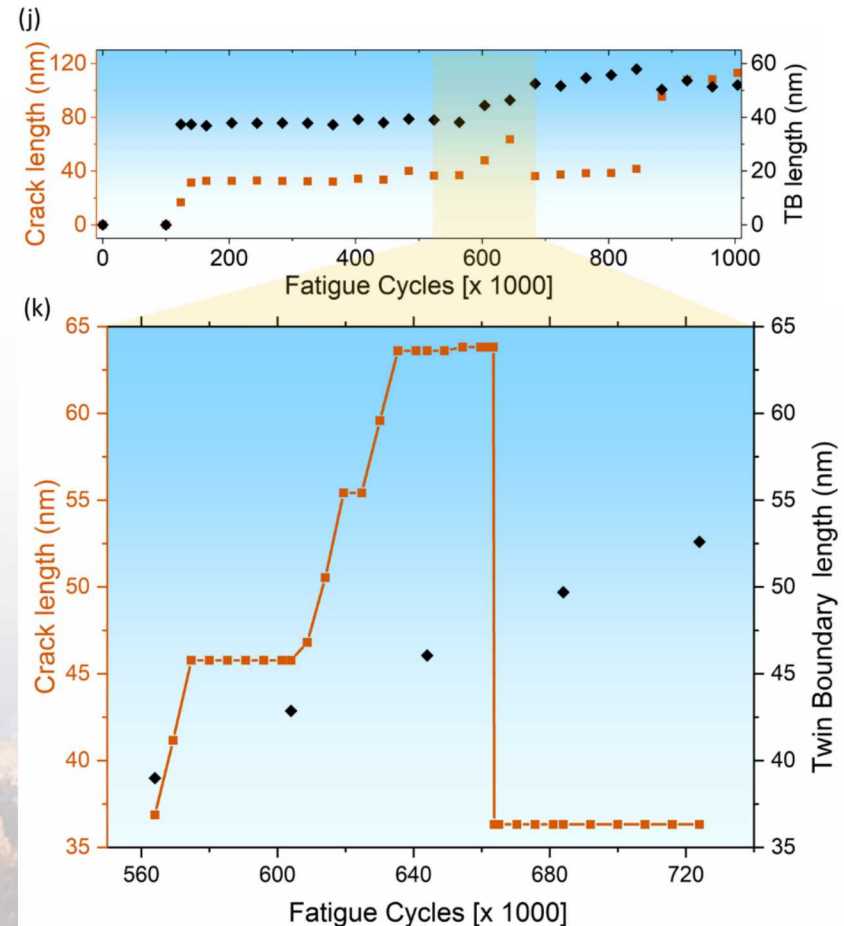
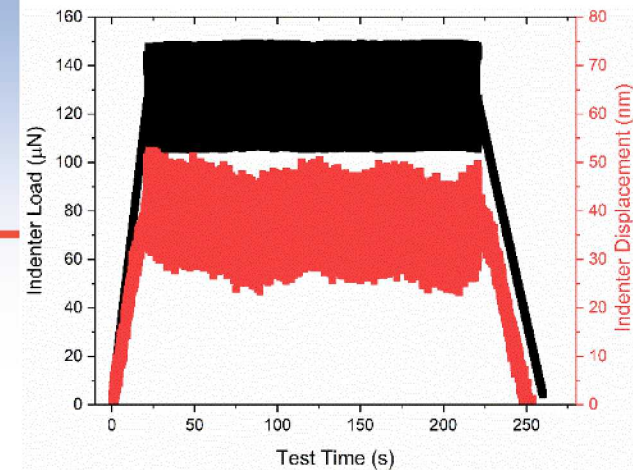
Collaborators: C. Barr, B. Boyce, & W. Mook

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

8x playback speed
N = 40,000 in 200s



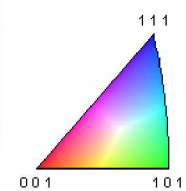
$\sim 2 \text{ pm/cycle}$



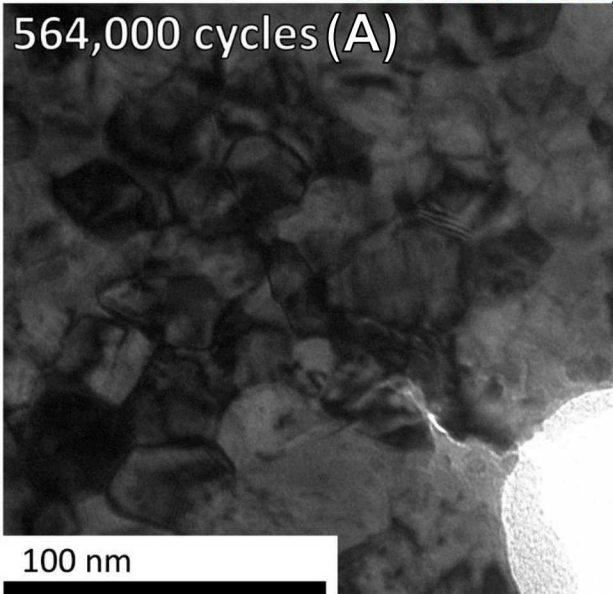
- $da/dN = 1.7 \times 10^{12} \text{ m/cycle}$
- Non-linear crack extension rate
- Crack propagation path changes "direction"

Crack Propagation, Closure, and Re-Direction

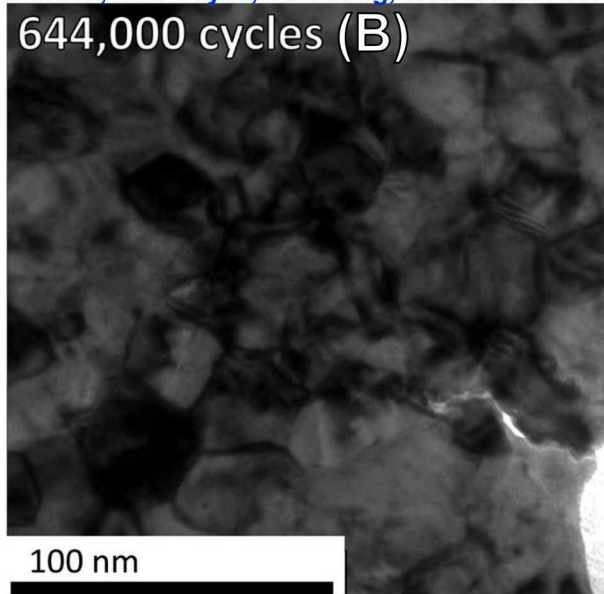
Collaborators: C. Barr, W. Mook, B.L. Boyce, Ta Duong, & M. Demkowicz



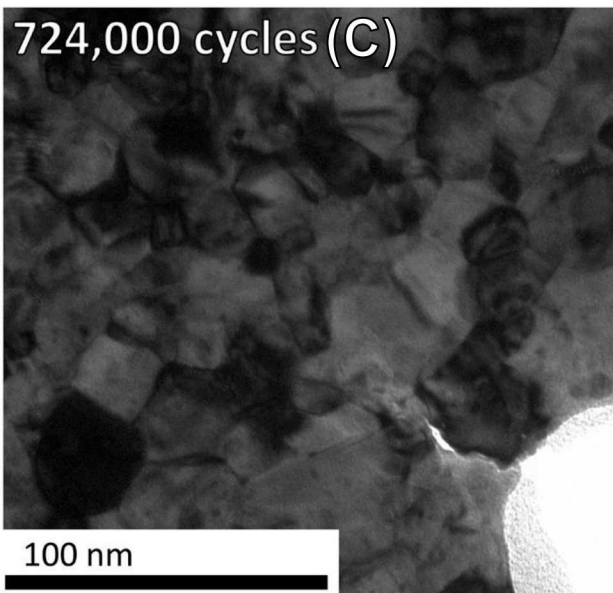
564,000 cycles (A)



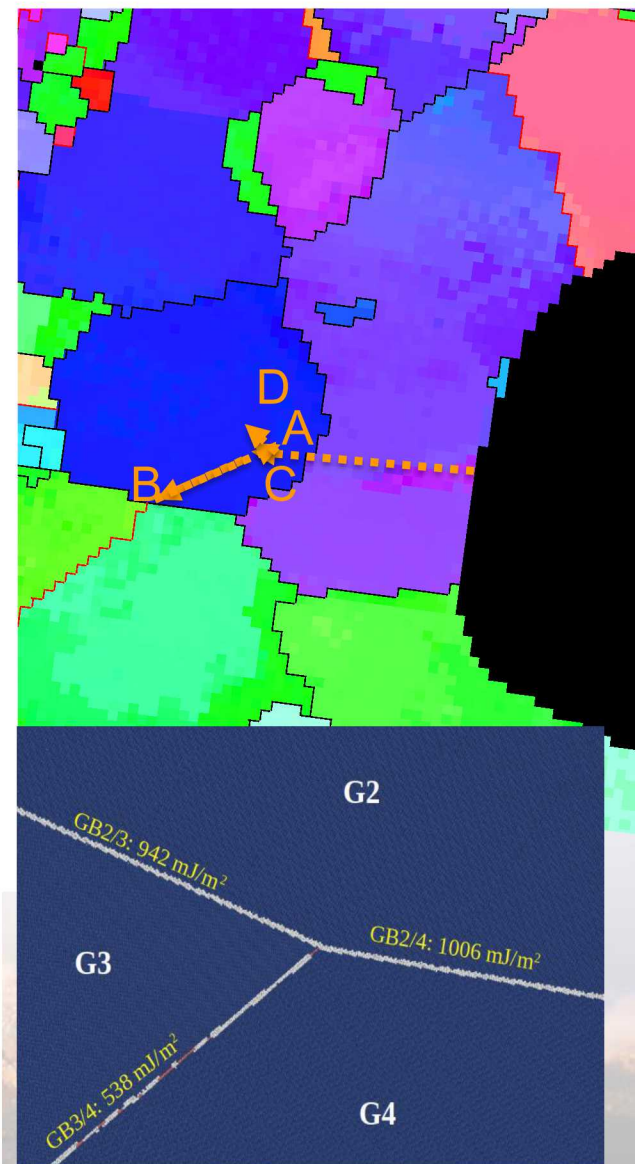
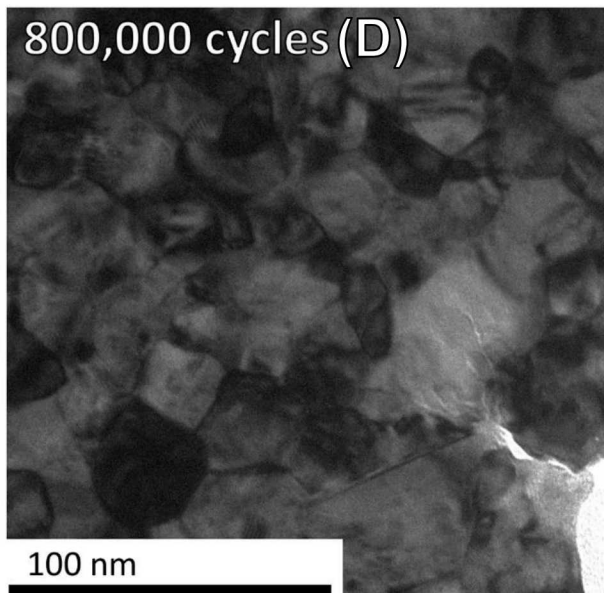
644,000 cycles (B)



724,000 cycles (C)



800,000 cycles (D)

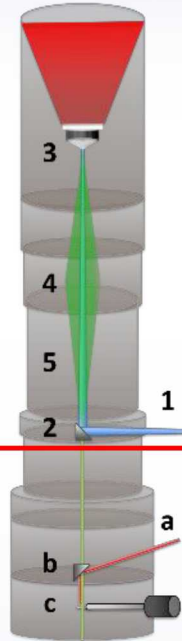


In-situ Specimen Drive Laser System

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



Electron Beam

IR Laser

Laser Alignment TEM Holder

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

IDES INTEGRATED
DYNAMIC
ELECTRON
SOLUTIONS

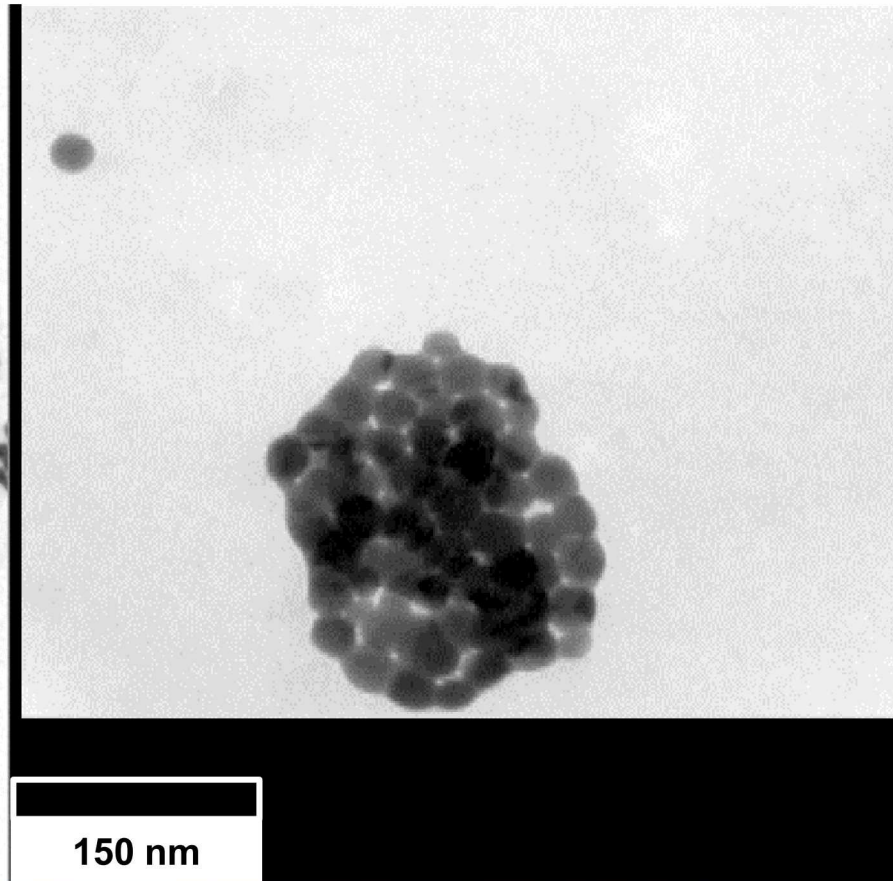
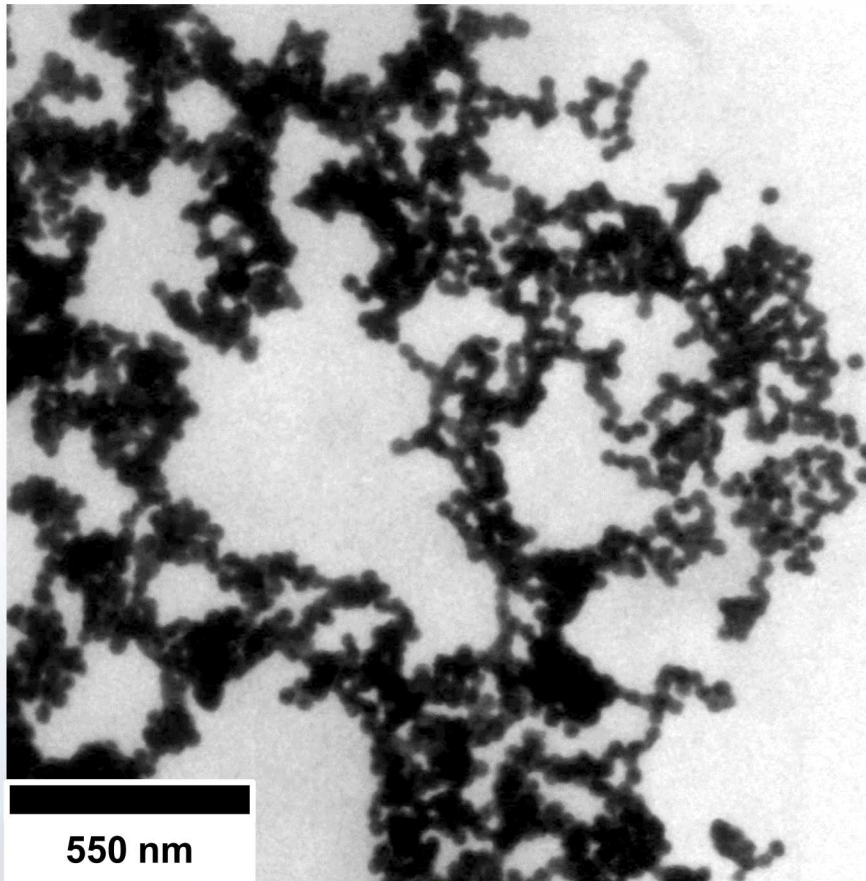


Sandia National Laboratories



Complex Interaction Au NPs Exposed to Laser Irradiation

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



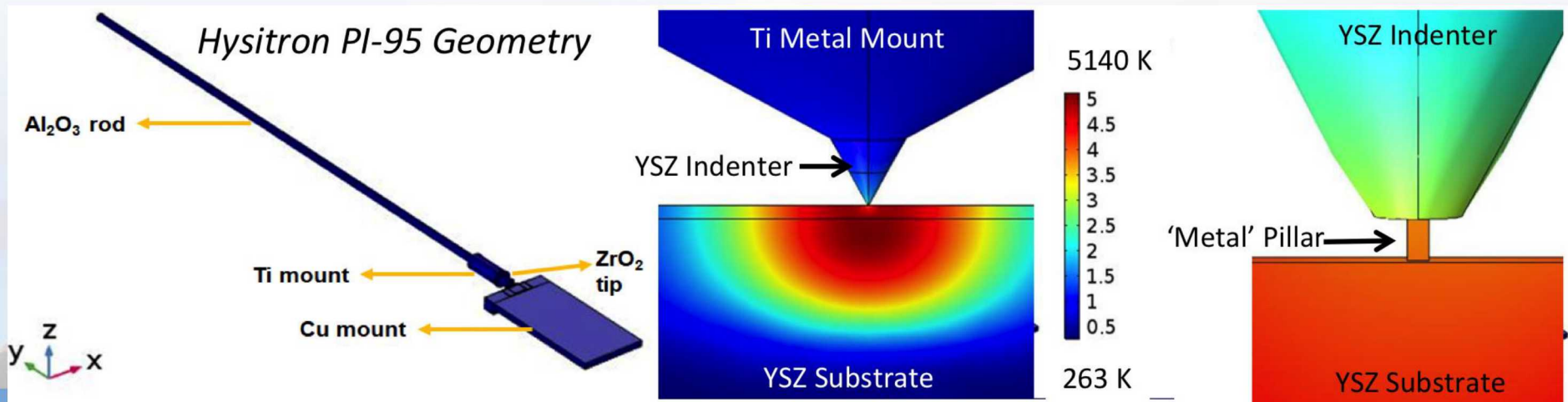
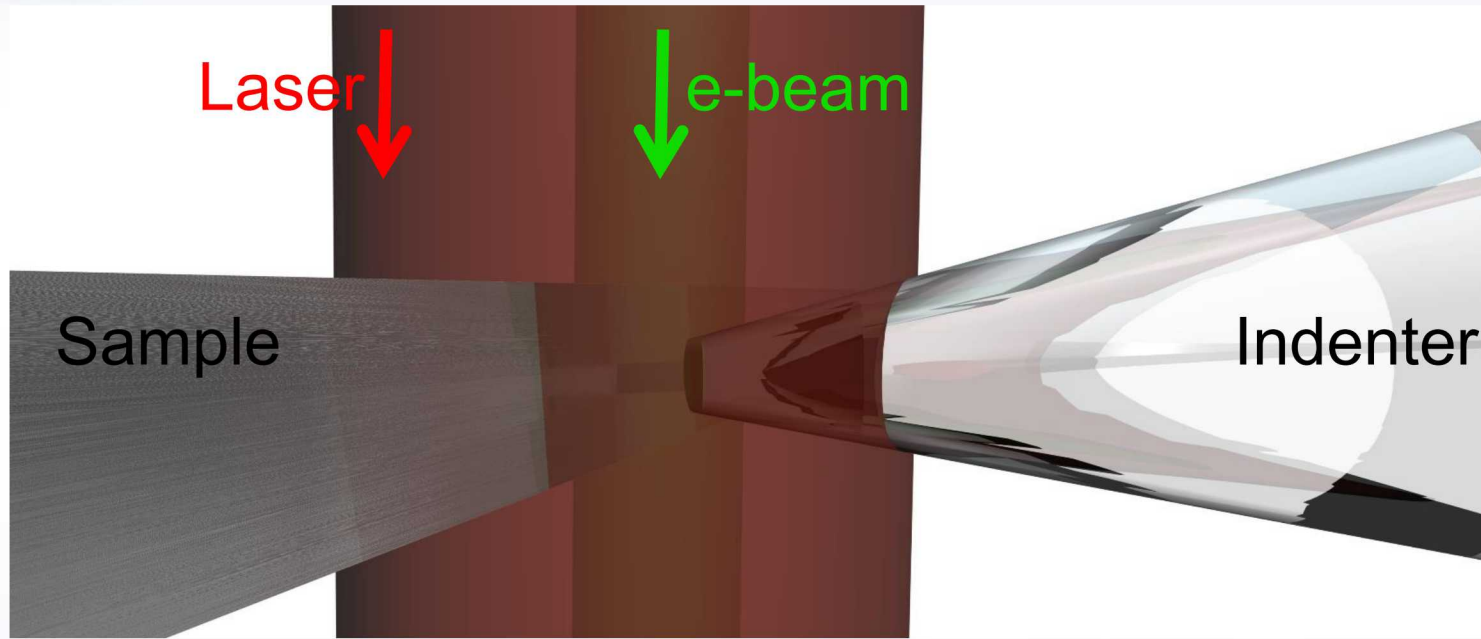
Speed = 2.5x



A Complex Combination of Sintering, Reactions, and Ablation Occurs

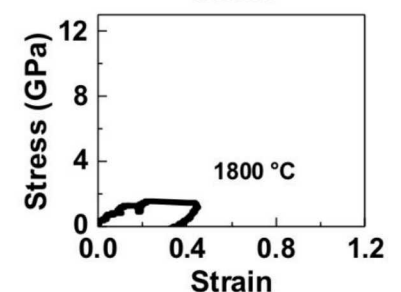
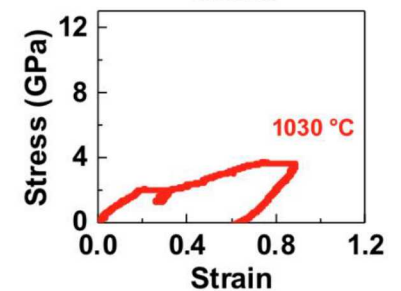
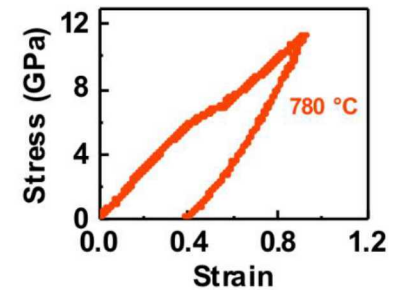
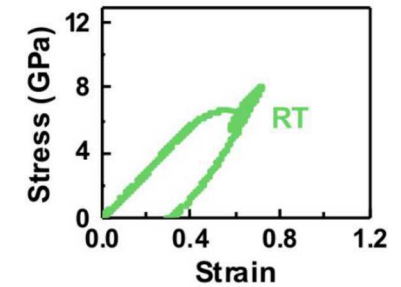
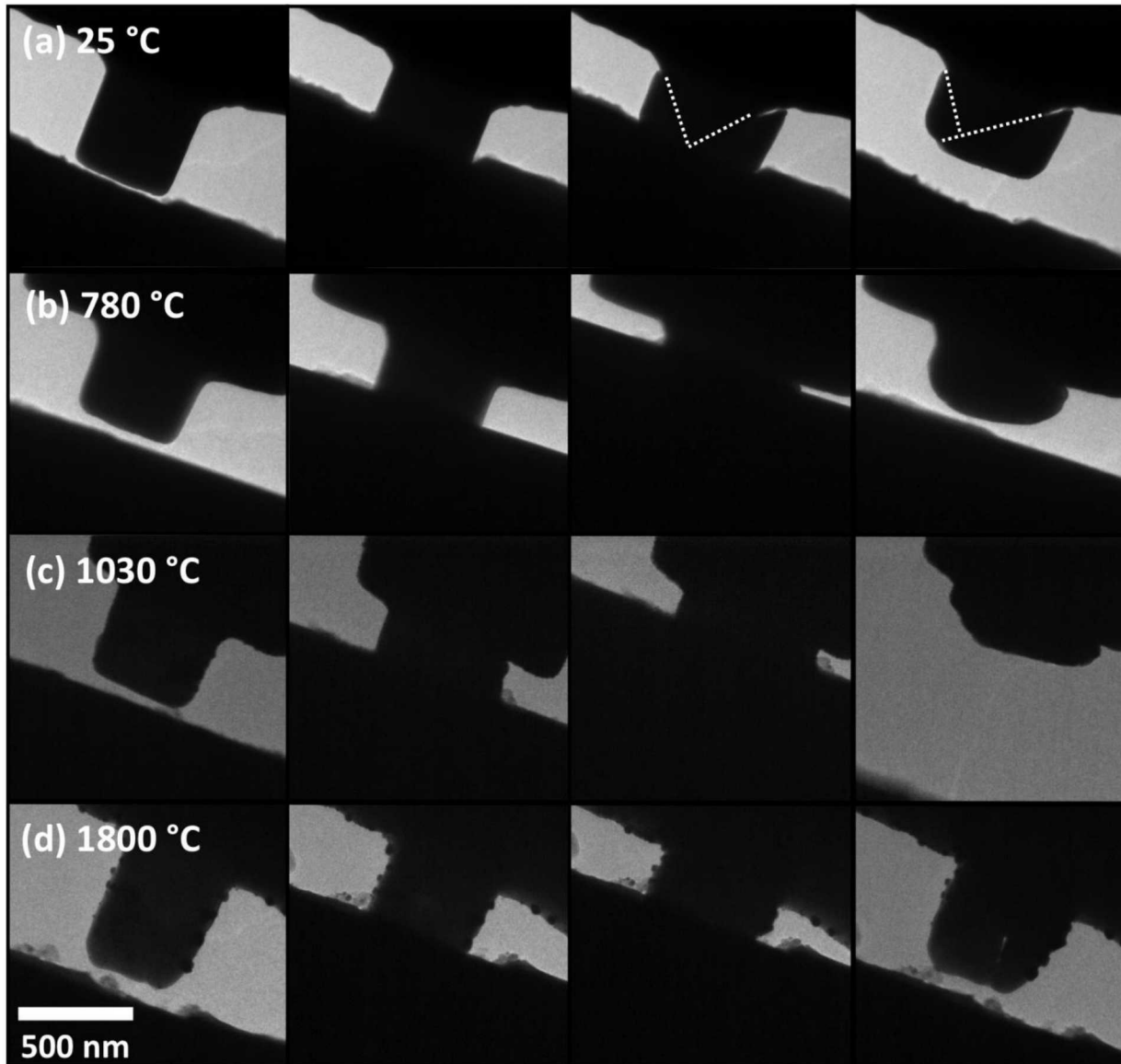
Can we Combine Laser Heating with Mechanical Testing?

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



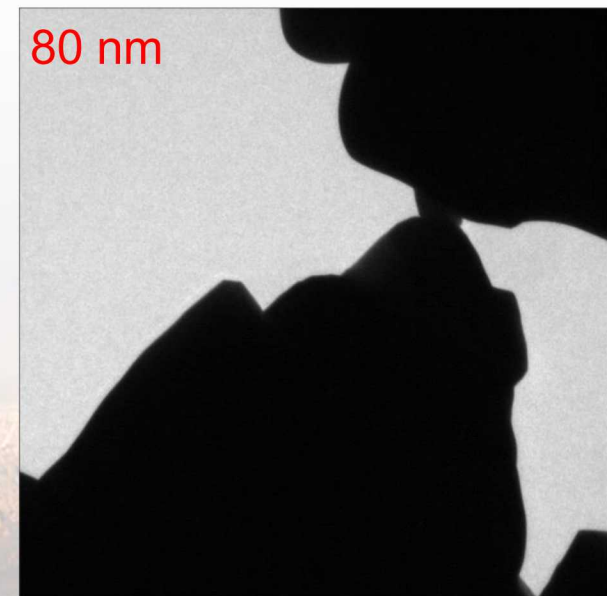
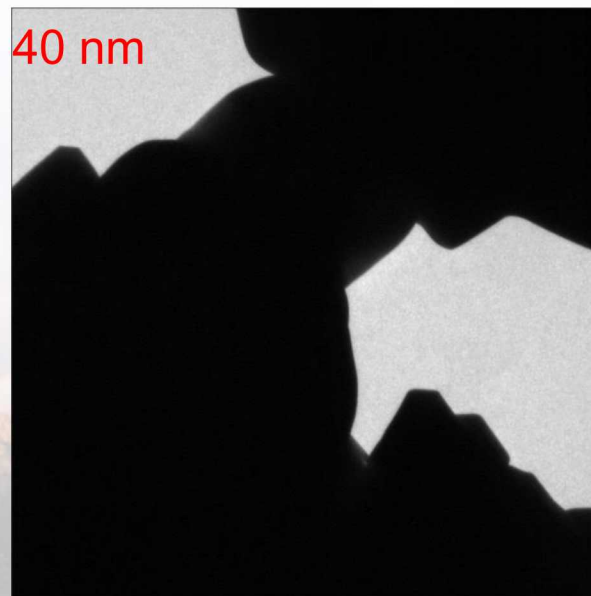
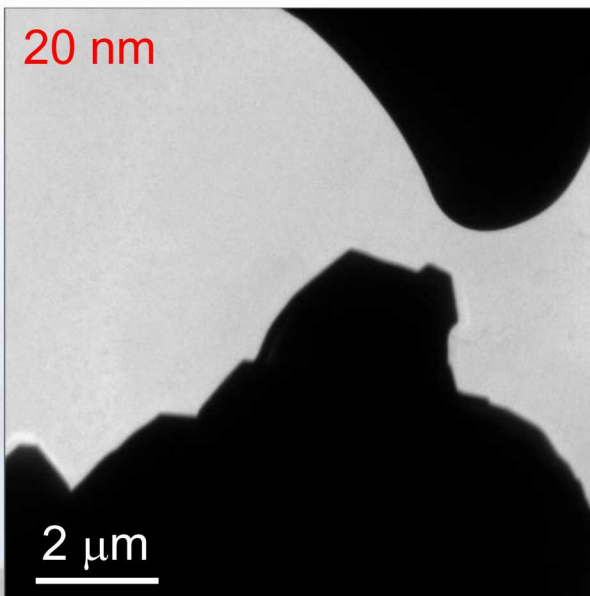
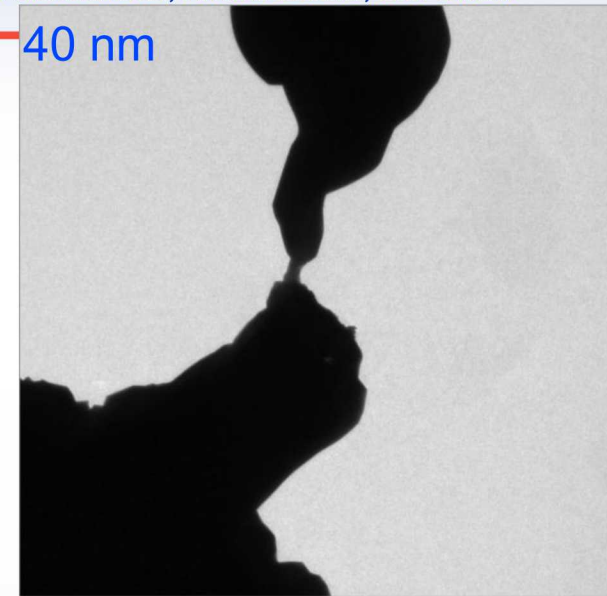
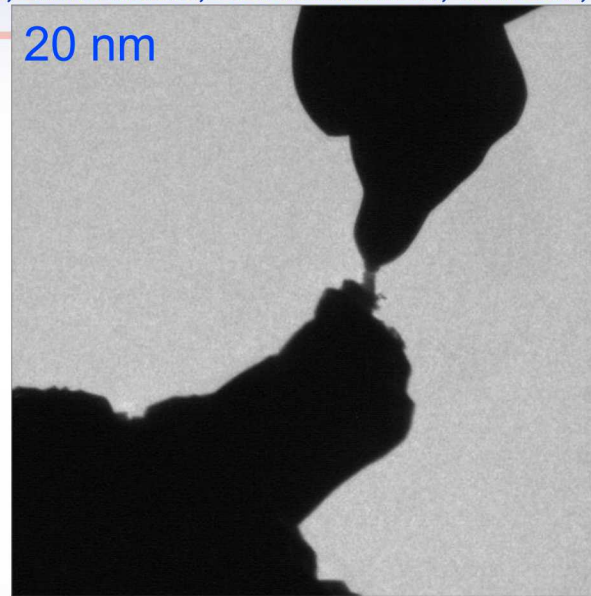
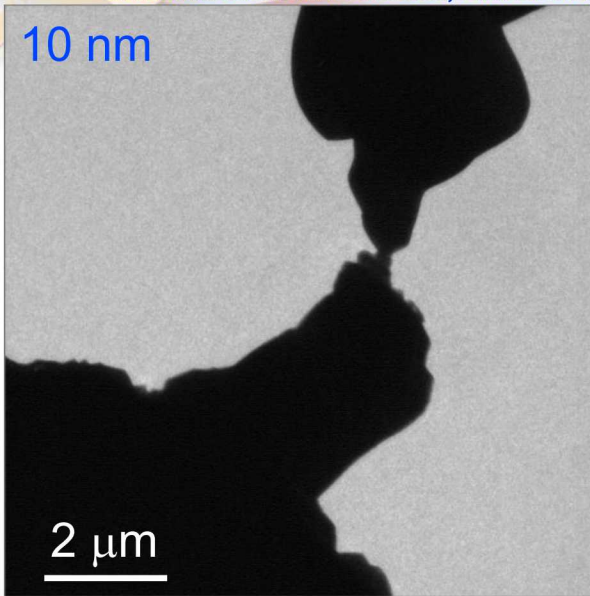
High Temperature Stress-Strain

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



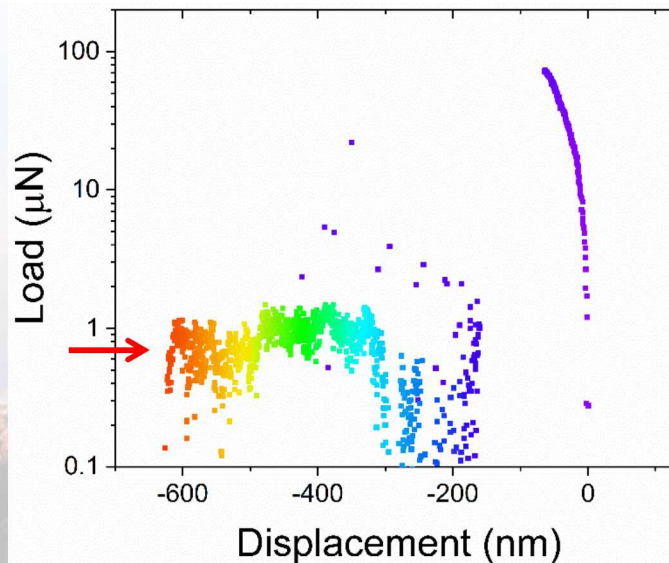
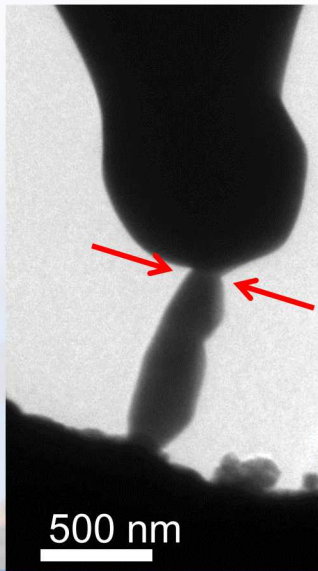
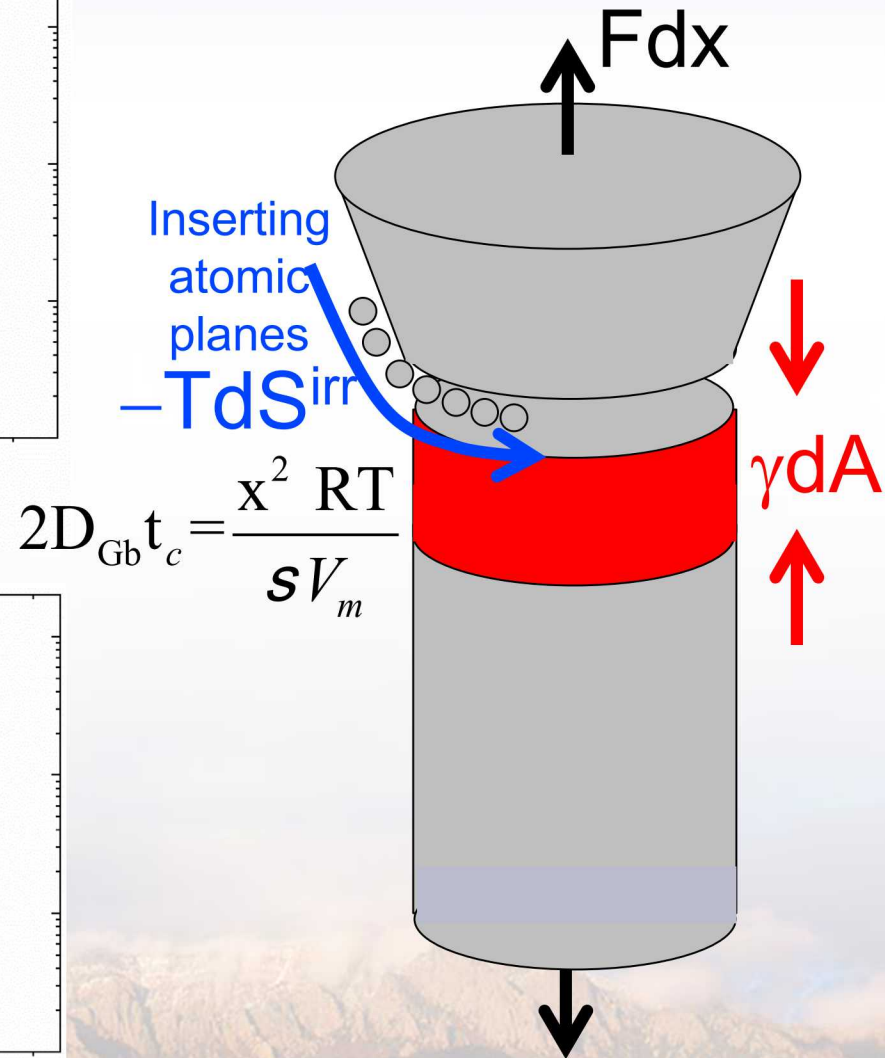
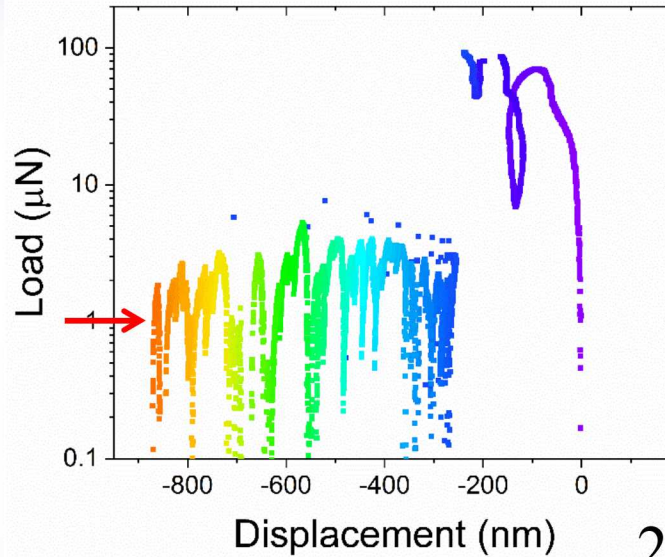
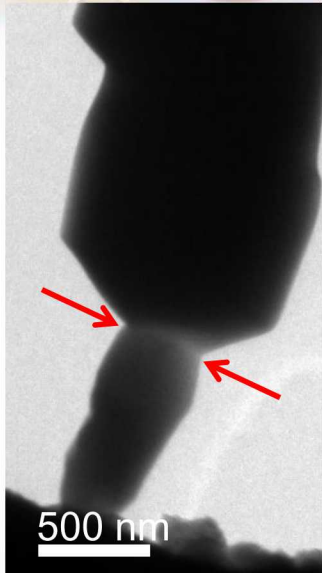
Pushing the Laser Limit - 1604 °C and 2056 °C ScSZ-ScSZ

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



Mechanism for Fiber Growth

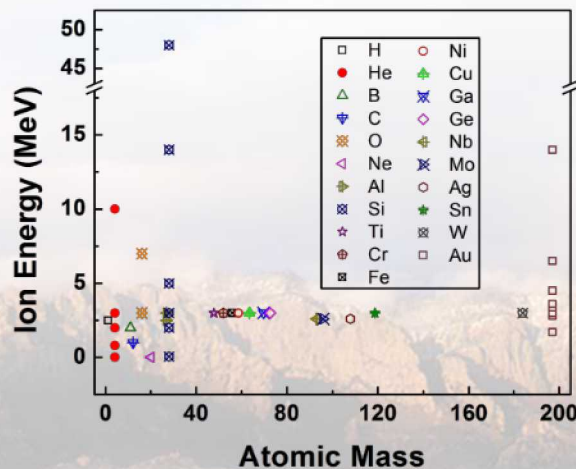
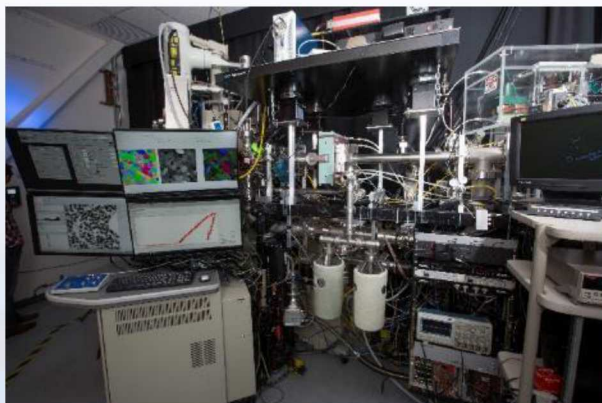
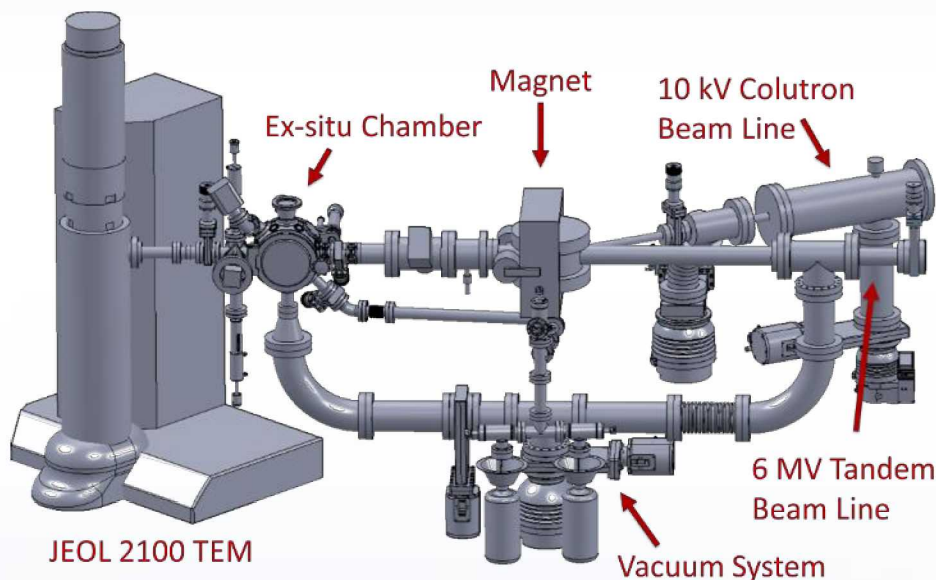
Contributors: R.L. Grosso, E.N.S. Muccillo, D.N.F. Muche, G.S. Jawaharram, C.M. Barr, A.M. Monterrosa, R.H.R. Castro, S.J. Dillon



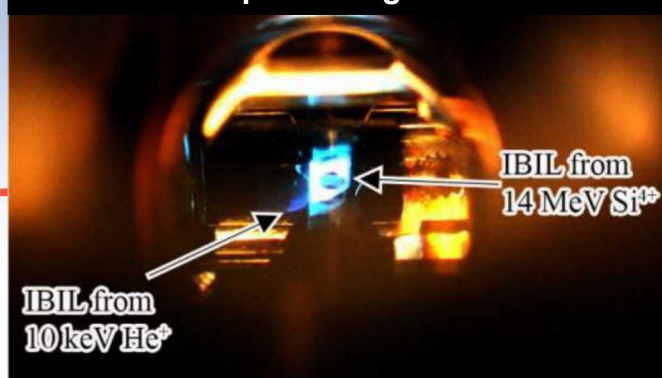
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem



IBIL from a quartz stage inside the TEM

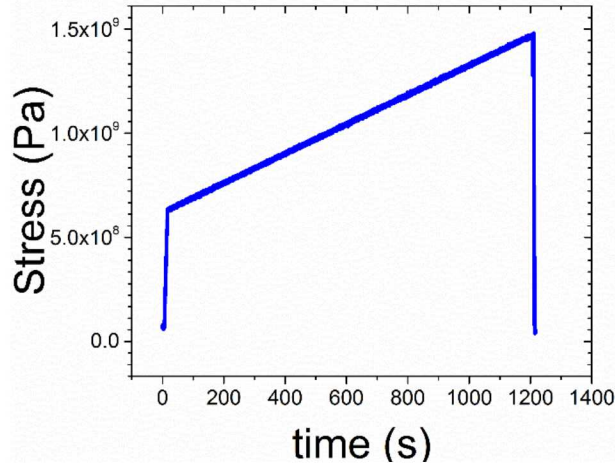


In situ TEM Ion Irradiation + Mechanical Testing = *In situ* TEM Irradiation Creep

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

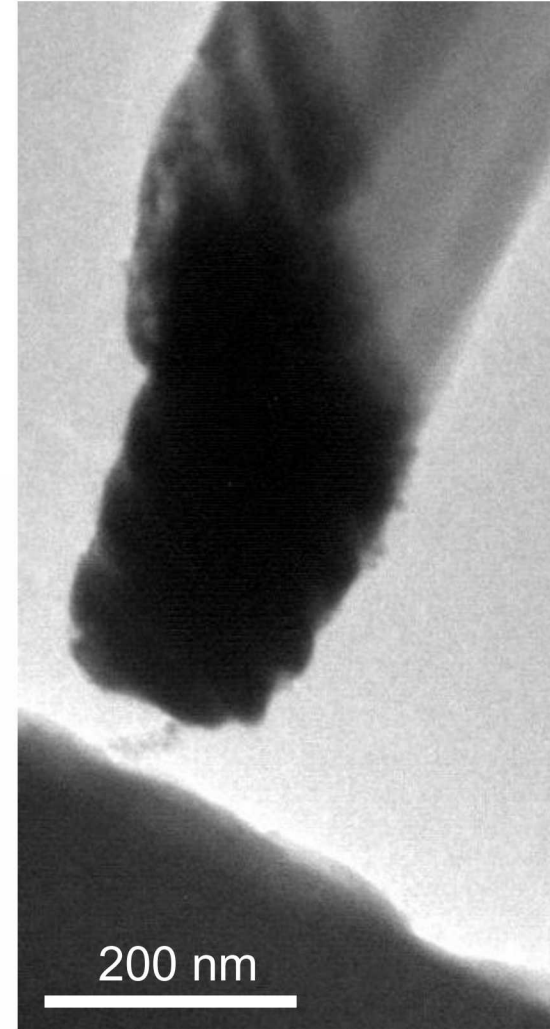
Controlled Loading Rate Experiments

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

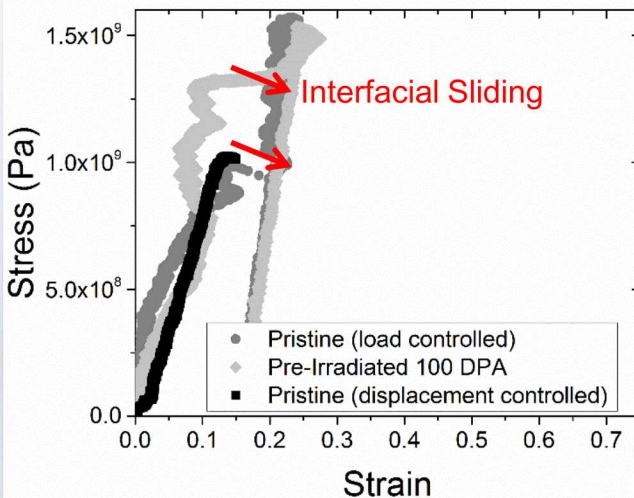


**In-situ TEM
radiation
creep is
feasible!**

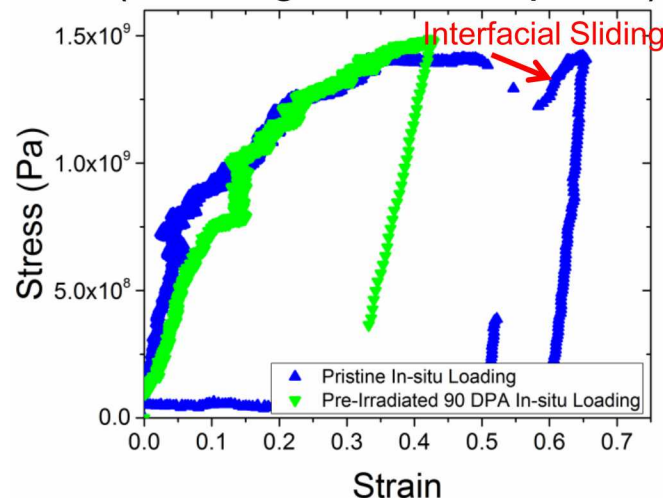
50 nm Cu-W multilayer
20 Min



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



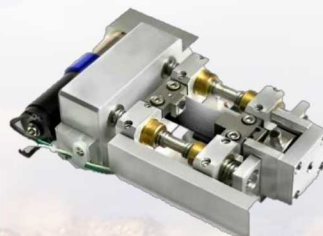
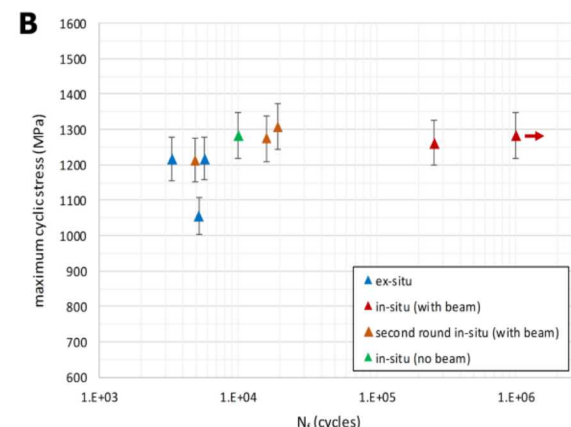
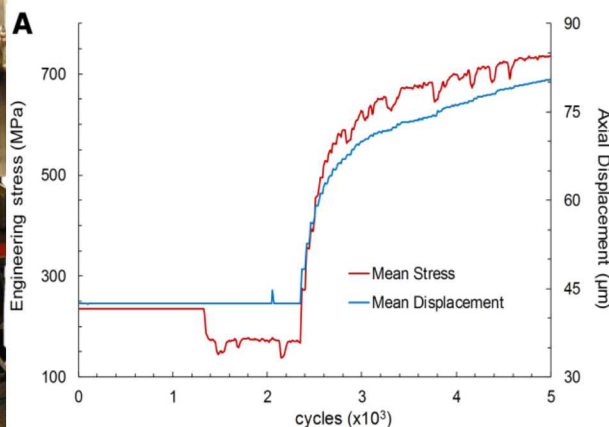
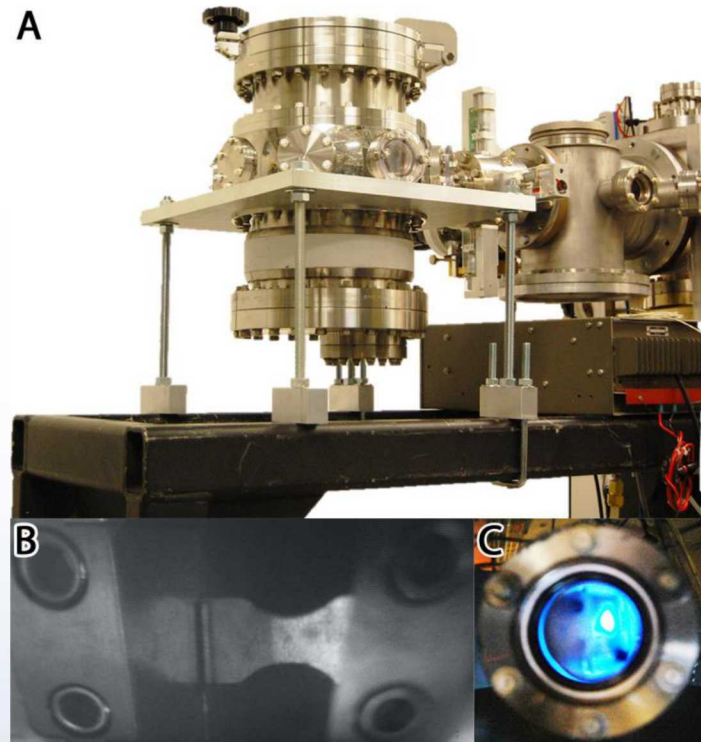
Irradiation Creep (Loading rate 0.6 Mpa s^{-1})



Ex situ Mechanical Testing End Station

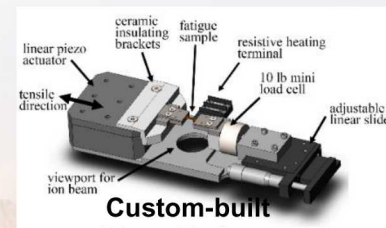
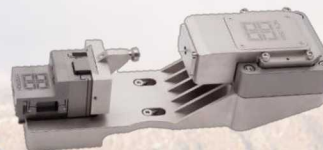
Collaborators: D. Buller, B. Boyce, J. Carroll, P. Price, C. Taylor, B. Muntifer, S. Briggs, N. Heckman, J.A. Scott

- 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

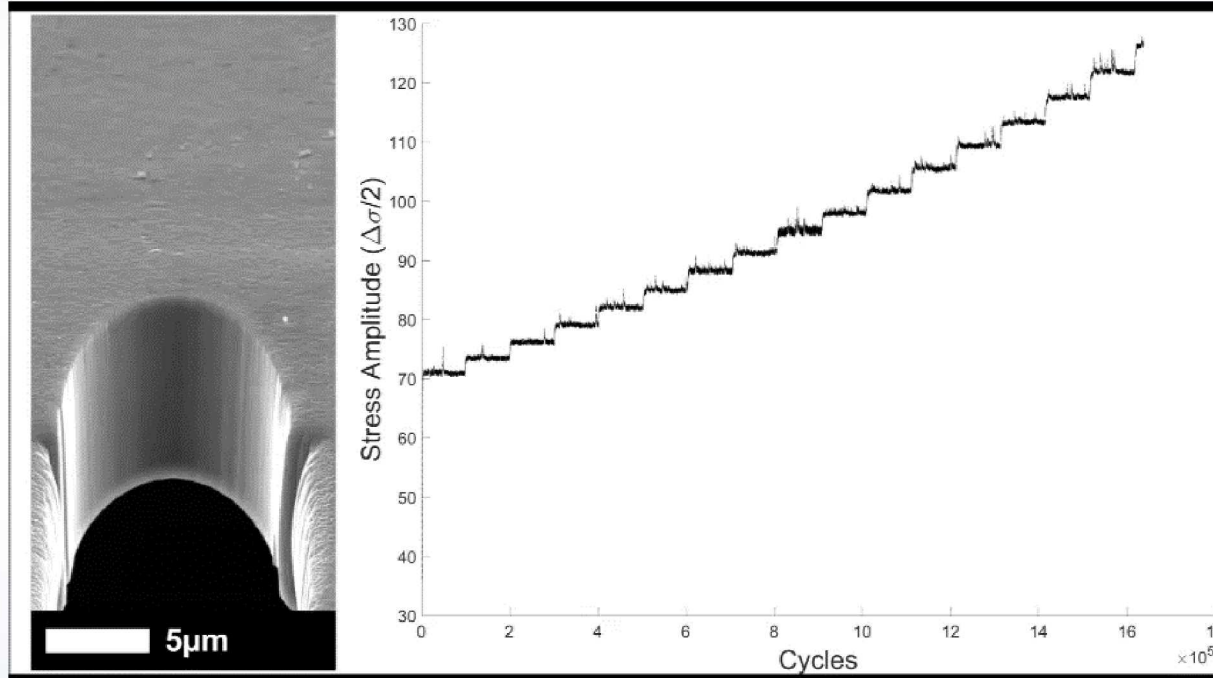


Gatan Cryostage



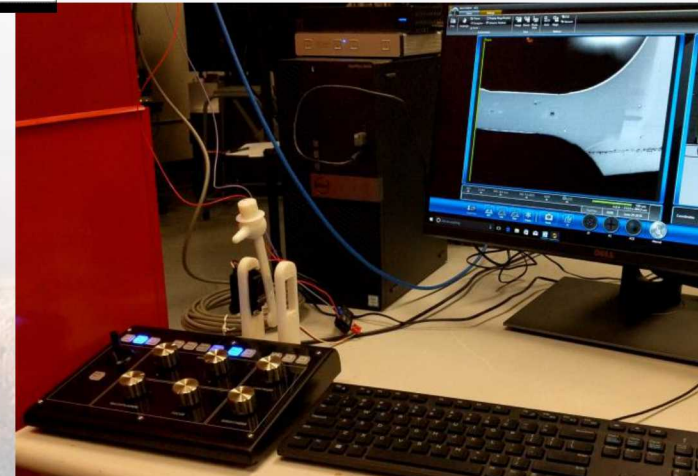
In situ High-cycle Fatigue

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



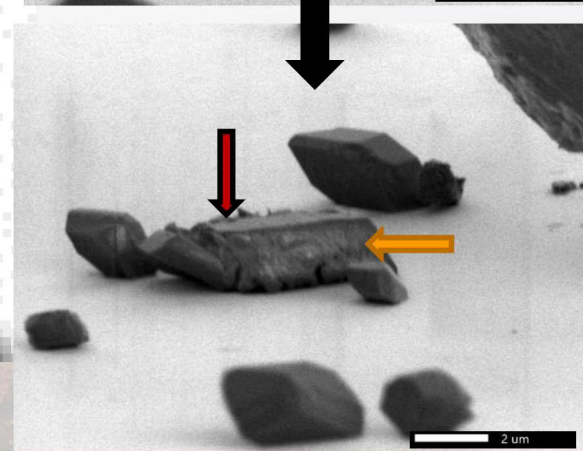
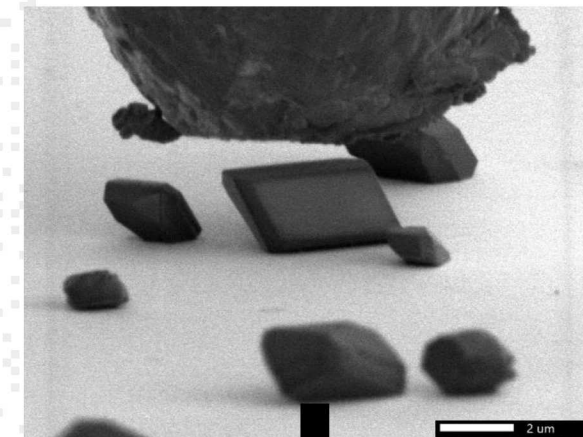
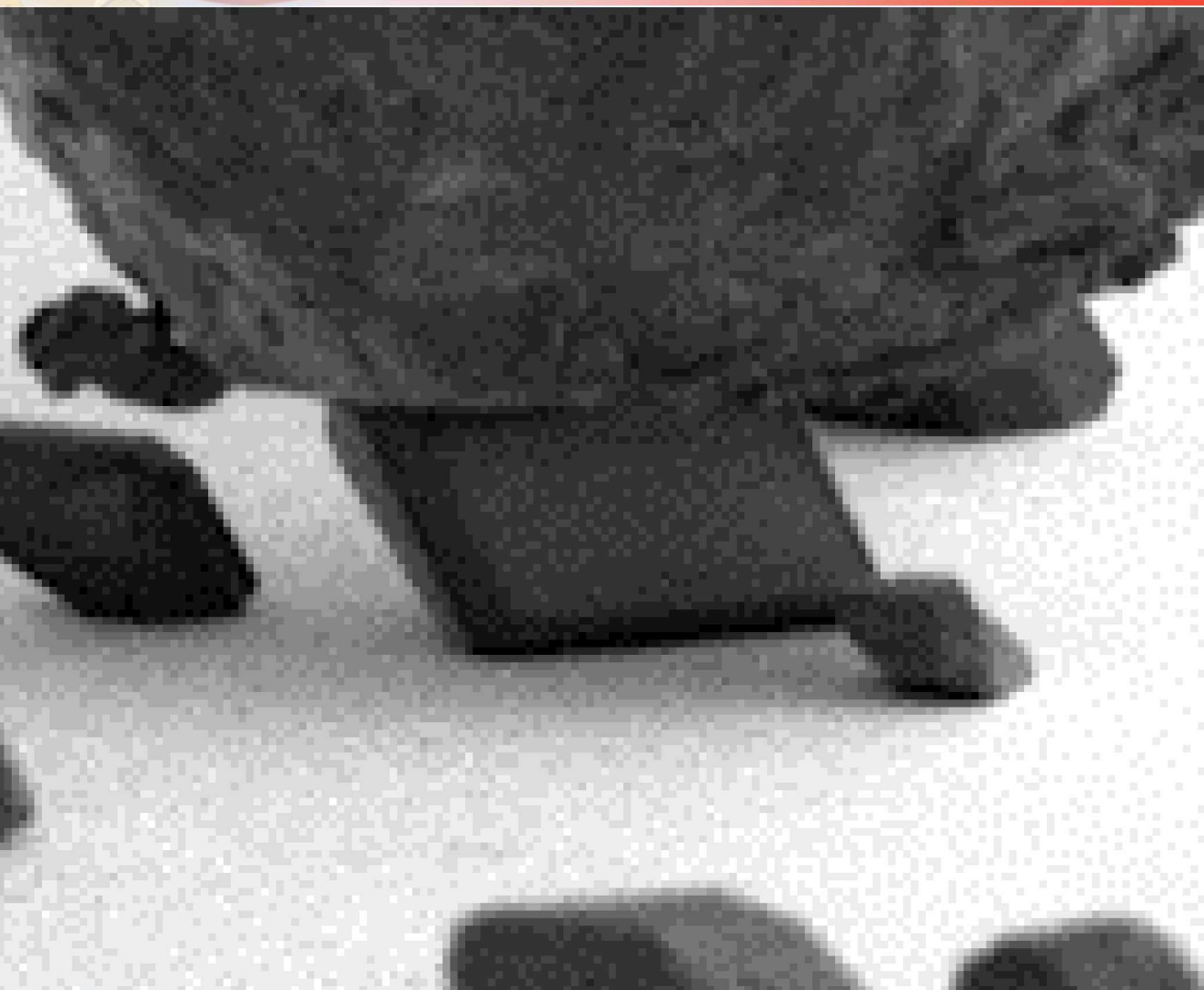
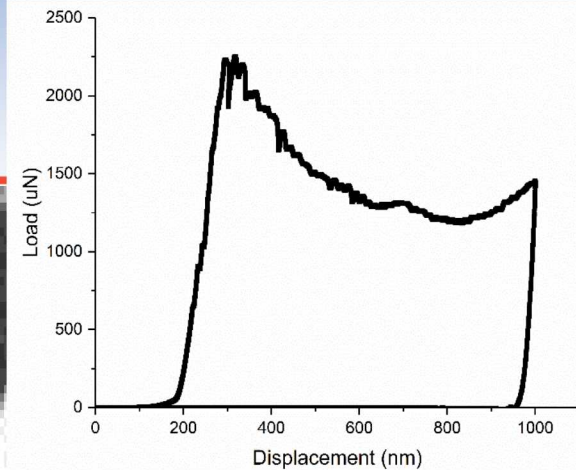
- Nanocrystalline Ni-40Fe, 10-60 nm grain size, 10 μm notch, imaged at 60°
- Cycled at 30 Hz, 4000 cycles between images

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

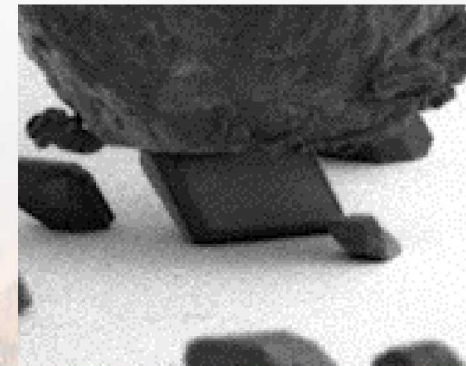
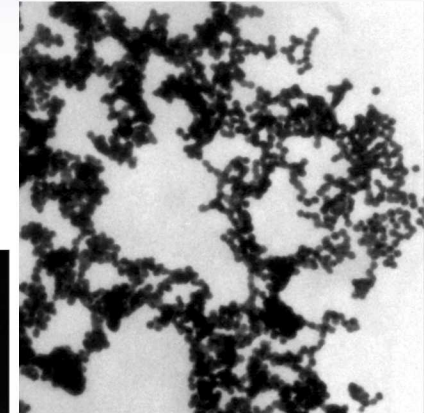
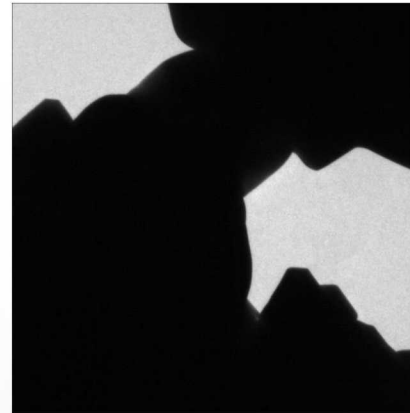


Unconventional *In situ* Microscopy Creates a Wealth of Possibilities



- 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 I³SEM 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. Averbach, 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.



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Sandia's User and Position Opportunities



D. Hanson, W. Martin, M. Wasiolek

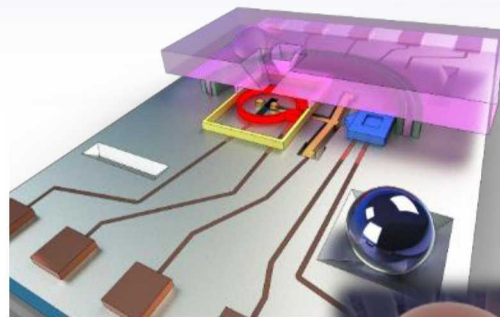
www.cint.lanl.gov

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

Core Facility - SNL

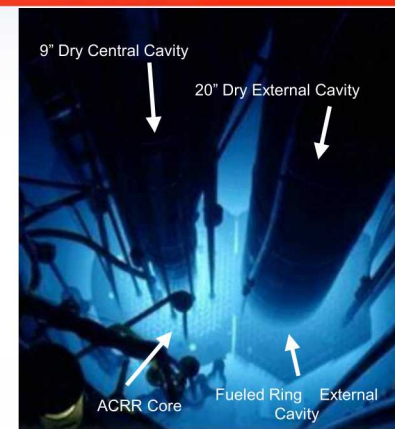


Gateway Facility - LANL



www.nsunf.inl.gov

- Three proposal a year for 9 months



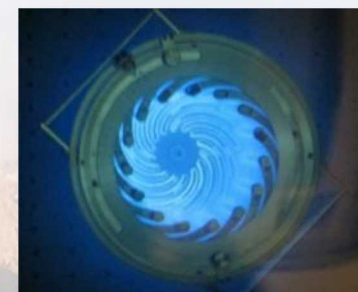
Position Opportunities at:

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

Post-doc = 671121

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