Understanding the Local Structure-Property Relationships of Solders in Terrestrial vs. Microgravity Environments Using Electron Microscopy and Nano-mechanical Testing

Siddhartha (Sid) Pathak

Assistant Professor

Chemical and Materials Engineering

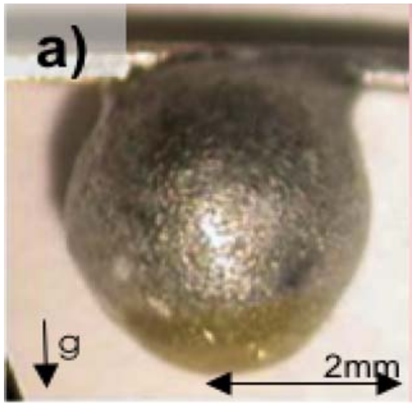
University of Nevada, Reno, NV 89557

https://wolfweb.unr.edu/homepage/spathak/

University of Nevada, Reno

Solders in Terrestrial vs. Microgravity Environments

- In-Space Soldering Investigation (ISSI) experiments performed aboard the International Space Station (ISS) – 2003-2005
- The ISSI data has demonstrated that a ***lack of buoyancy forces*** in microgravity can internally trap the flux created during soldering at interfaces, such as repair joints.
- Hypothesis: such internal porosity can be ***detrimental*** to the desired strength of the joint, as well as its thermal and electrical conductivity
- Results will be instrumental in ***enhancing our fundamental understanding*** of the effects of surface tension driven convection phenomena during solidification processing operations such as brazing, soldering, and welding.
- Furthermore, the microgravity experiments represent a ***lowest gravity boundary condition***. As such, these results could also be useful in predicting solidification behavior on other lower gravity environments (e.g. moon or Mars).



*Photograph of solder drop created **in gravity** hanging from a silver-coated strand of copper wire*

*Photograph of solder drop created on the ISS **in microgravity** with an equilibrium "football" shape.*

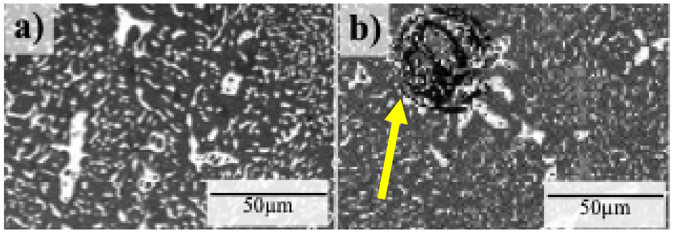
[1] Struk Peter M. , Pettegrew Richard D. . Soldering in Reduced Gravity Experiment. *SDTO 17003-U (SoRGE)* **2017**.
[2] Grugel Richard N., Luz Paul, Smith Guy, Spivey Reggie, Jeter Linda, Gillies Donald, Hua Fay, Anilkumar A. V. Materials research conducted aboard the International Space Station: Facilities overview, operational procedures, and experimental outcomes. *Acta Astronautica* **2008**;62:491-8.
[3] Grugel Richard, Gillies Donald, Murphy Lucinda, Ogle Julie A., Funkhouser Glen, Parris Frank, Anilkumar A.V., Hua Fay. Final Research Report. In-Space Soldering Investigation (ISSI). 2006.

Funding: Nevada NASA EPSCoR.

Collaborator: Dr. Richard Grugel, NASA

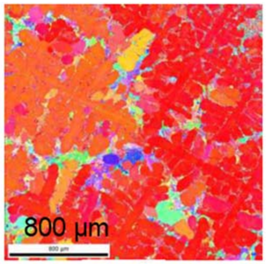
Mechanical Testing Tools at the Micro-to-Nano length scales

- The features of interest (porosity, dendrites etc.) in the solders have very small length scales (µm to mm)
- This requires specialized nano-mechanical tools for testing and characterization.



Terrestrial Gravity Pore in micro-gravity

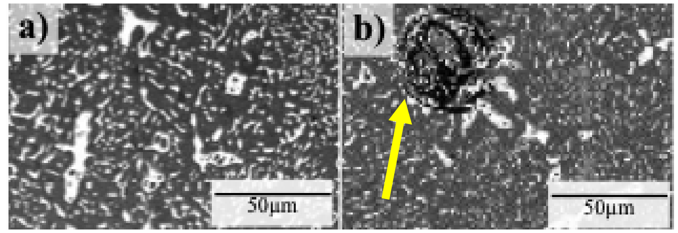
SEM



EBSD

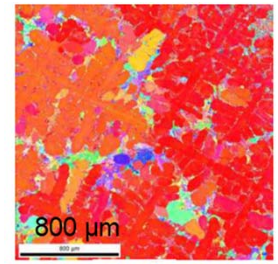
Mechanical Testing Tools at the Micro-to-Nano length scales

- The features of interest (porosity, dendrites etc.) in the solders have very small length scales (μm to mm)
- This requires specialized nano-mechanical tools for testing and characterization.

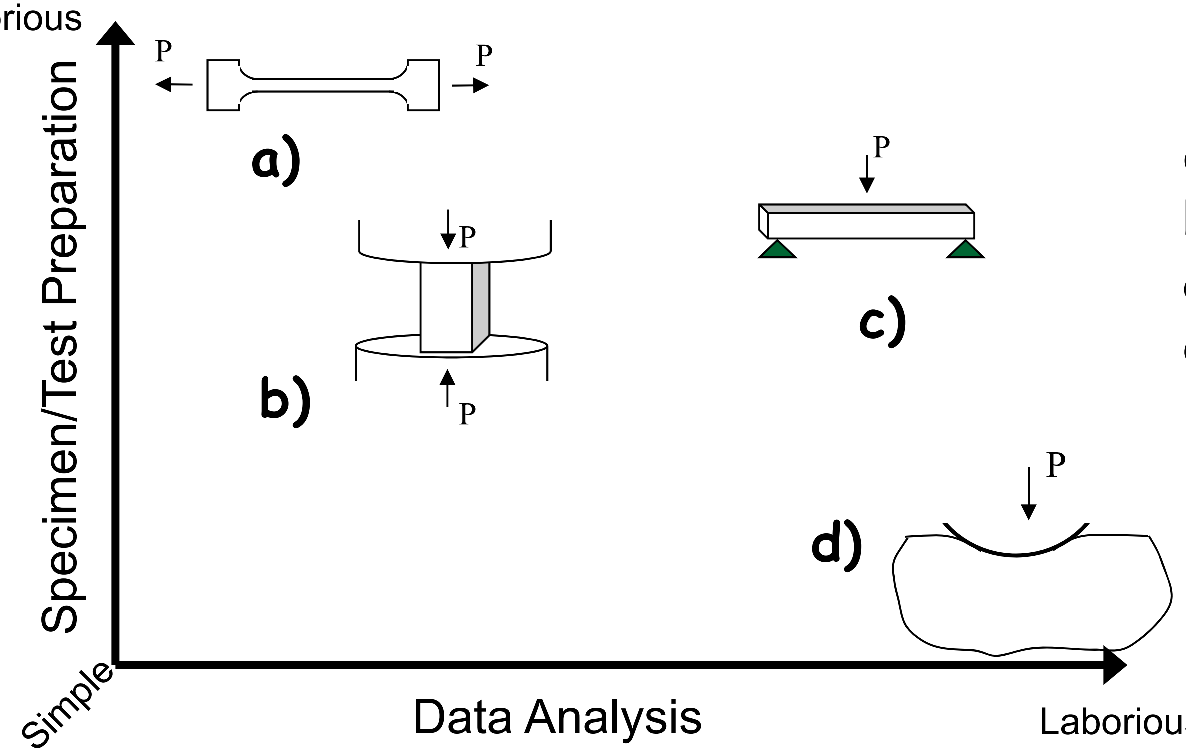


Terrestrial Gravity | Pore in micro-gravity

SEM



EBSD

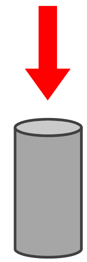


a) Tensile
b) Compression
c) Bending
d) Indentation

Investigating local mechanical response at the micro- and nano-scales: *In-situ* SEM straining capabilities at UNR

Micro-Compression

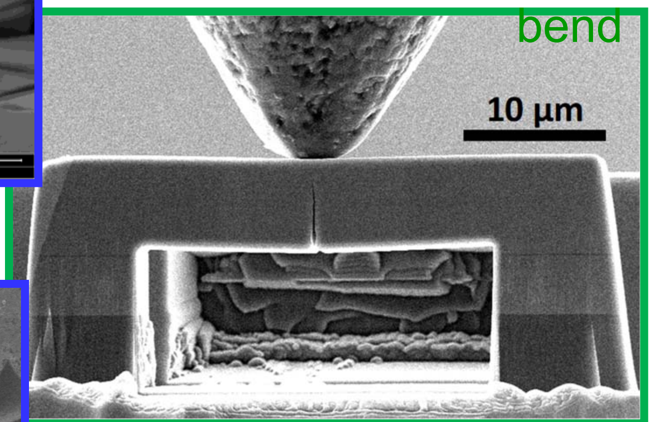
Load Cases | **Samples** | **Properties**

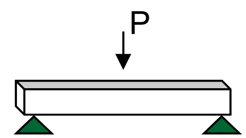


Nanowires, particles MEMS/NEMS, milled structures

E, σ_y, σ_f, n

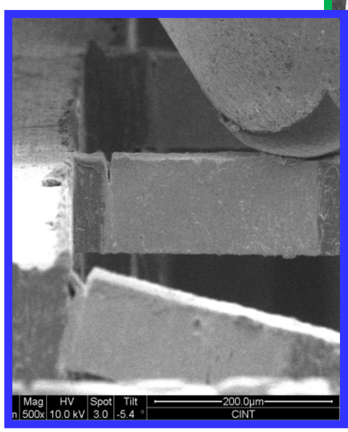
Micro 3-pt bend



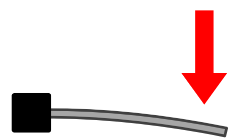


Substrates, thin films, multilayers

E, K_c



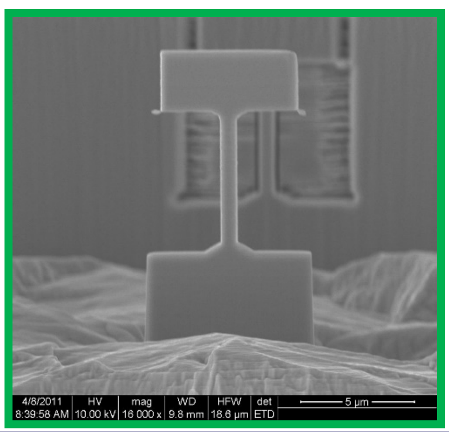
Cantilever Micro-Bending

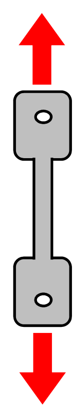


Nanowires, MEMS/NEMS, milled, machined structures

E, σ_y, K_c

Micro-Tension



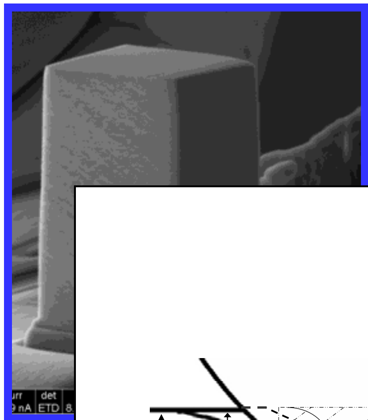


Nanowires, MEMS/NEMS, milled, machined structures

E, σ_y, σ_f, n, K_c

University of Nevada, Reno

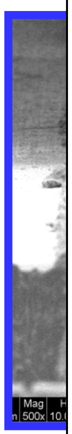
Investigating local mechanical response at the micro- and nano-scales: *In-situ* SEM straining capabilities at UNR

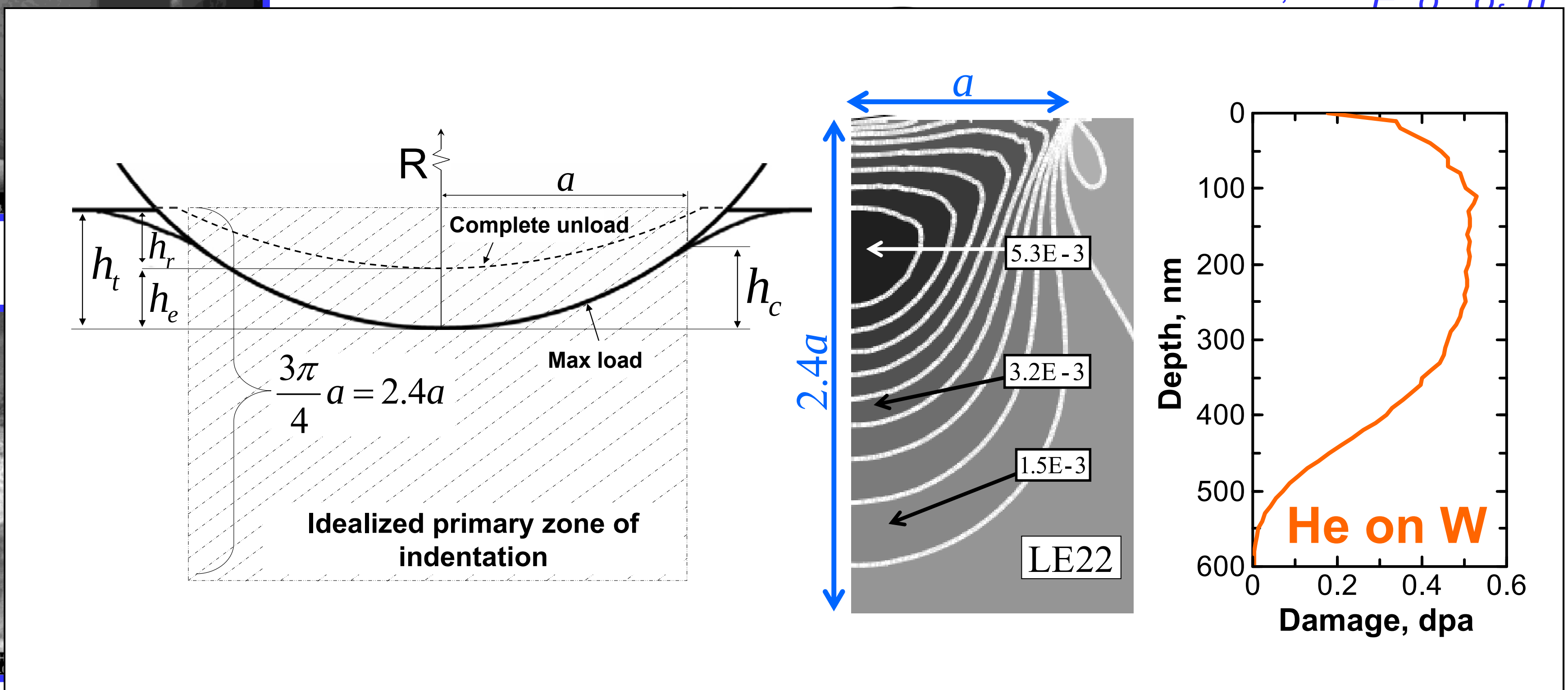


Micro-Compression

Load Cases | **Samples** | **Properties**

Nanowires,



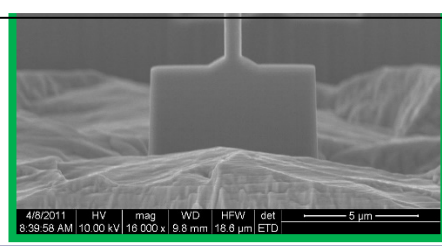


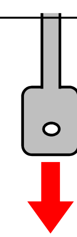
Cantilever Micro-Bending

E, σ_y, σ_f, n, K_c

MEMS/NEMS, milled, machined structures

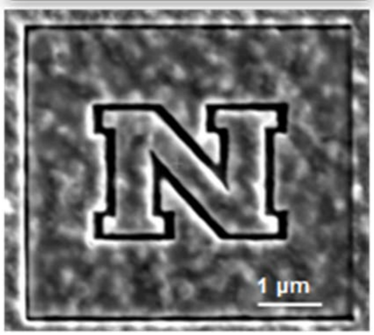
Micro-Tension





University of Nevada, Reno

UNR: Nano-mechanical Testing Facilities



FEI Scios™
Dualbeam FIB/SEM
NSF MRI grant
#1726897.

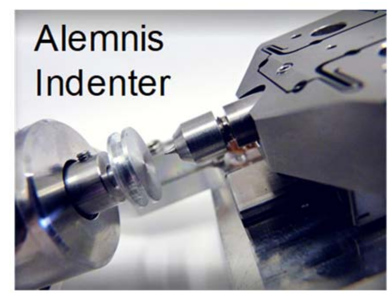
Ex-Situ (in air) indenters

Nanoindenter® XP
LANL Laboratory Education Equipment
Gift (LEEG) Program 2017

Hysitron TI 900 TriboIndenter

In-Situ indenters

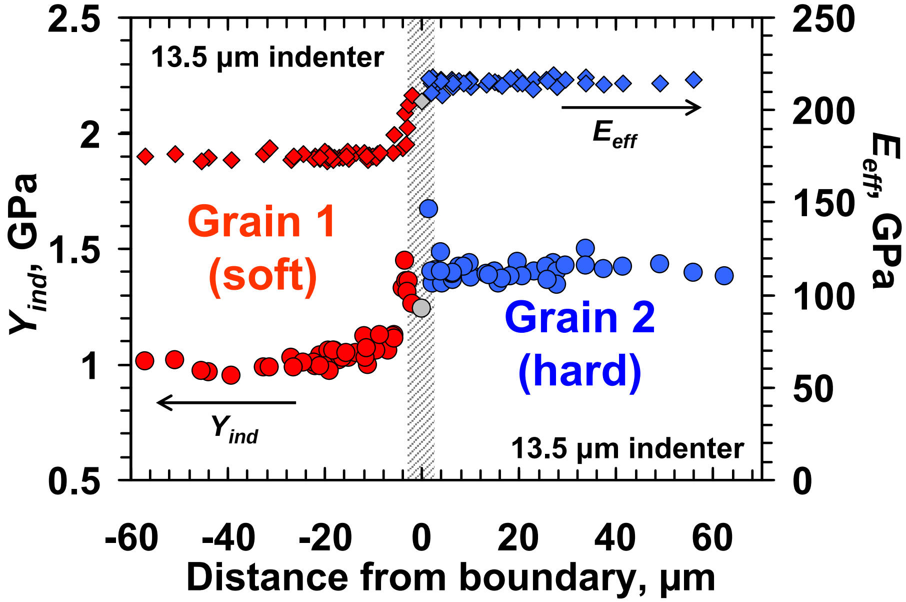
Hysitron PI 85 SEM PicoIndenter

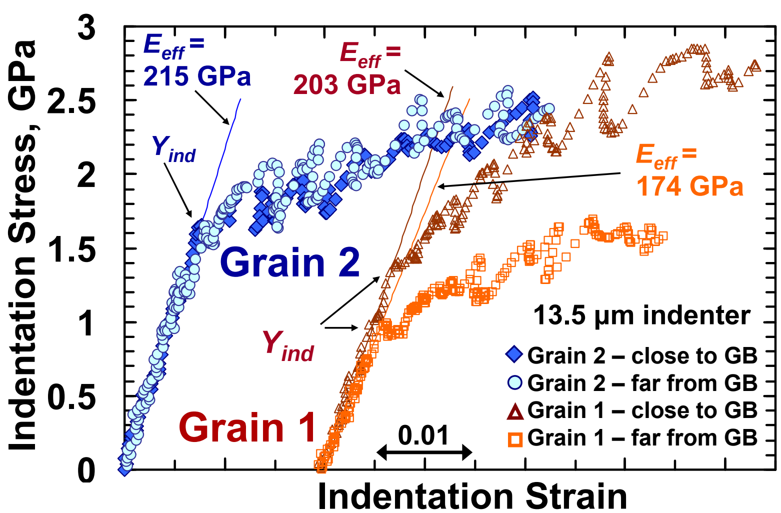


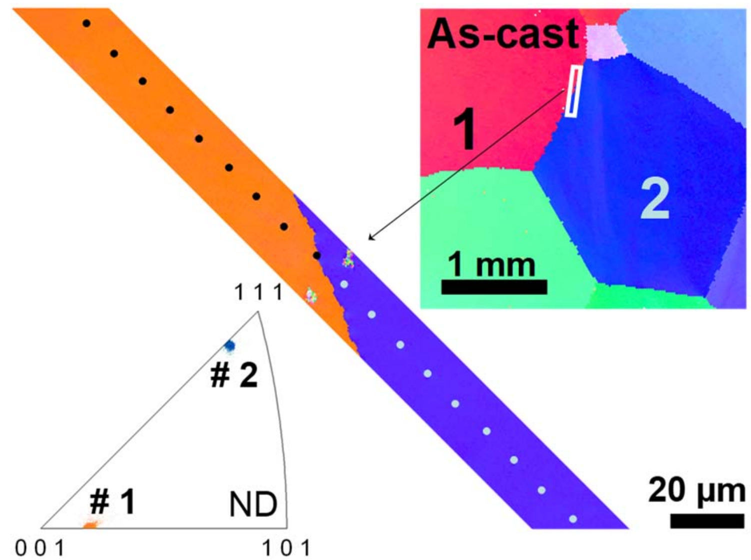
Alemnis Indenter system
DOE FY 2018 Scientific Infrastructure
Support for Consolidated Innovative
Nuclear Research

University of Nevada, Reno

Ex-situ (in air) Indentation across interfaces





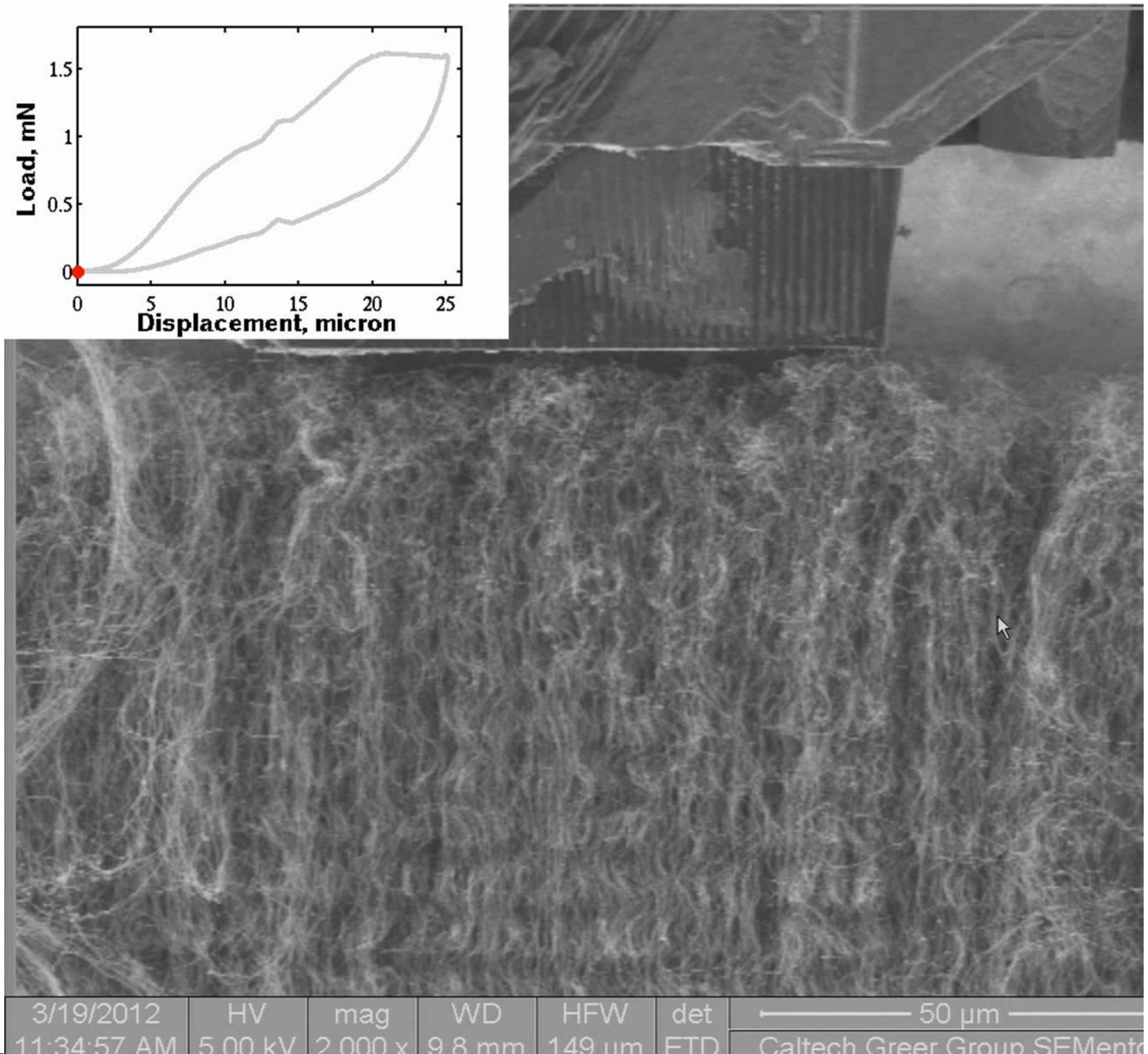


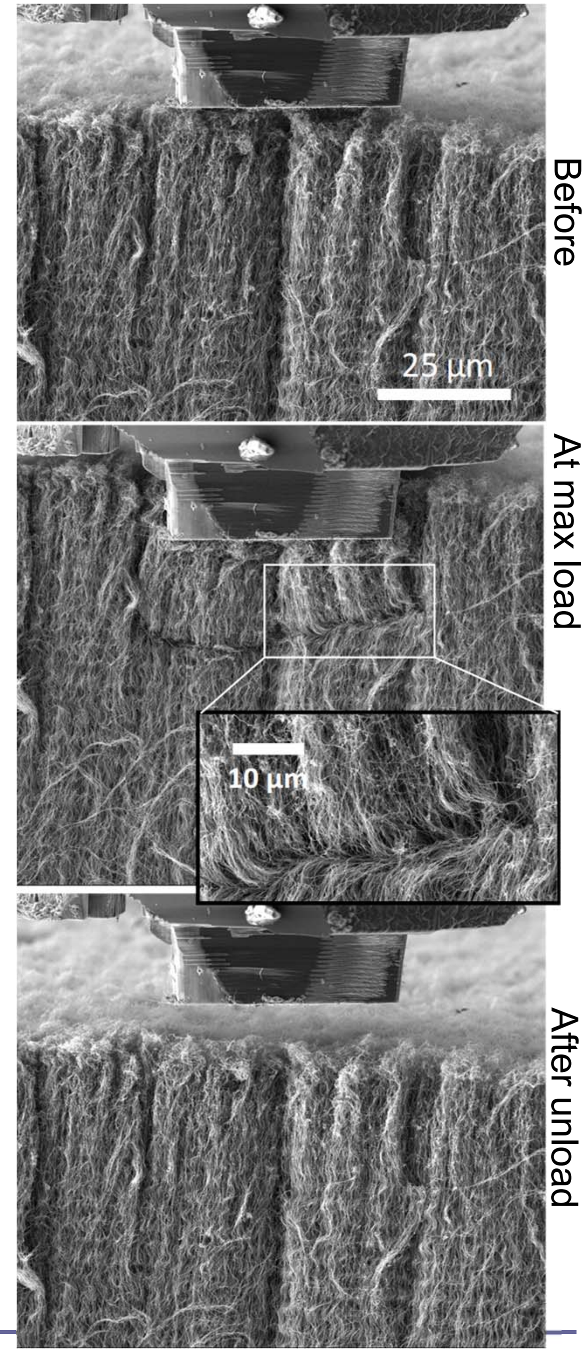
Fe-3%Si steel

University of Nevada, Reno

S Pathak, J Michler, K Wasmer, SR Kalidindi. J. Materials Science (2011)

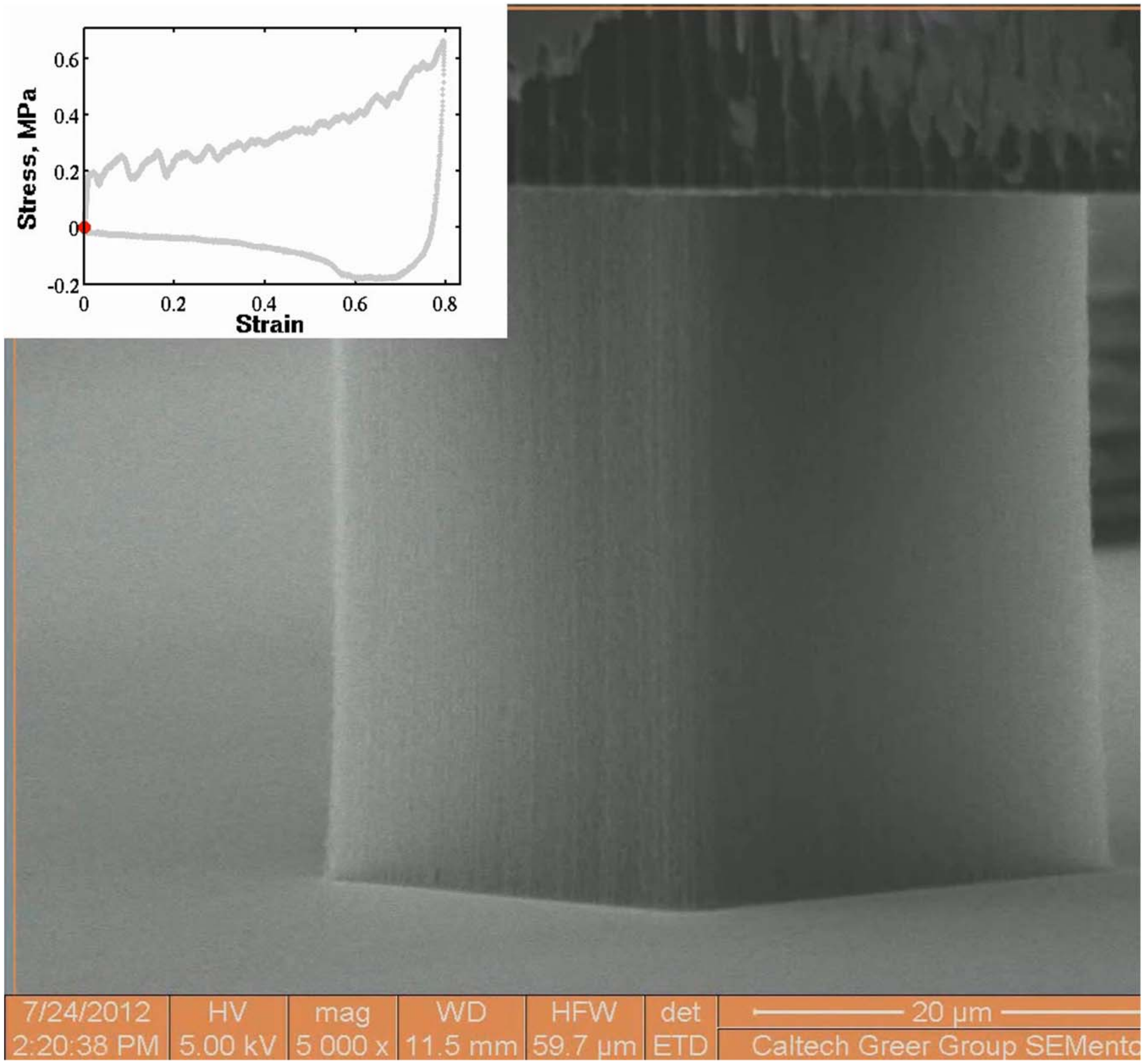
In-situ indentation - deformation of carbon nanotubes



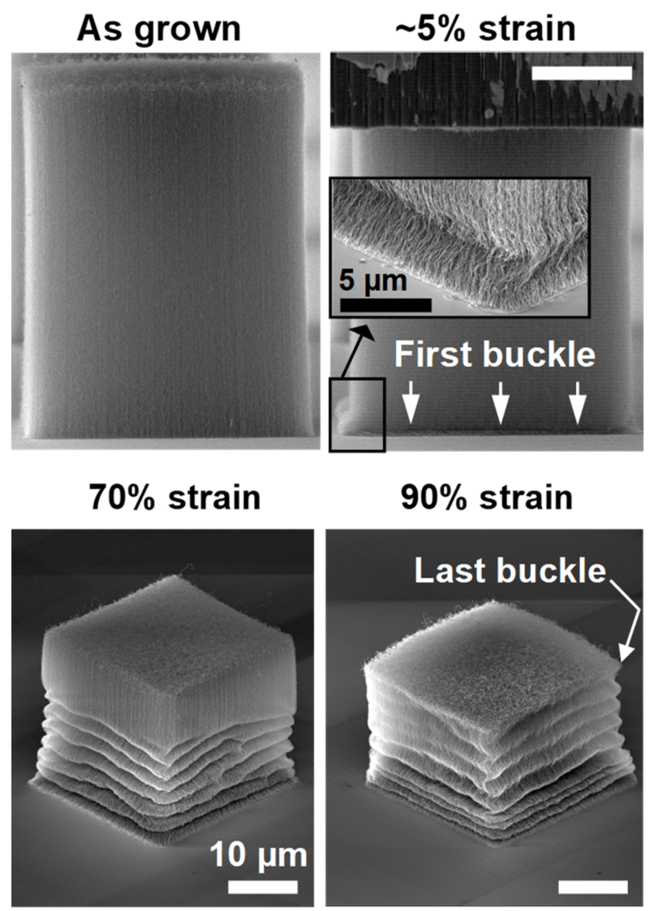


Pathak, Raney, Daraio. Carbon 2013. Effect of morphology on the strain recovery of vertically aligned carbon nanotube arrays- An in situ study

Micro-pillar compression: Deformation of carbon nanotube pillars



Sequence of buckle propagation

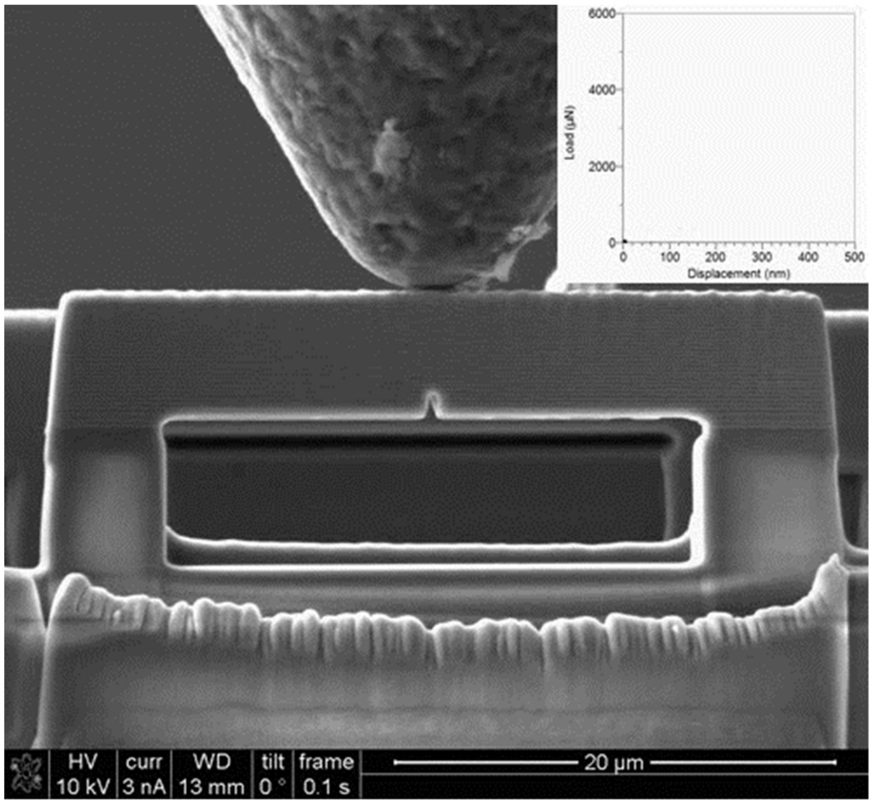


Pathak, Needleman, Hart, Greer. ACS Nano 2013. Local Relative Density Modulates Failure and Strength in Vertically Aligned Carbon Nanotubes

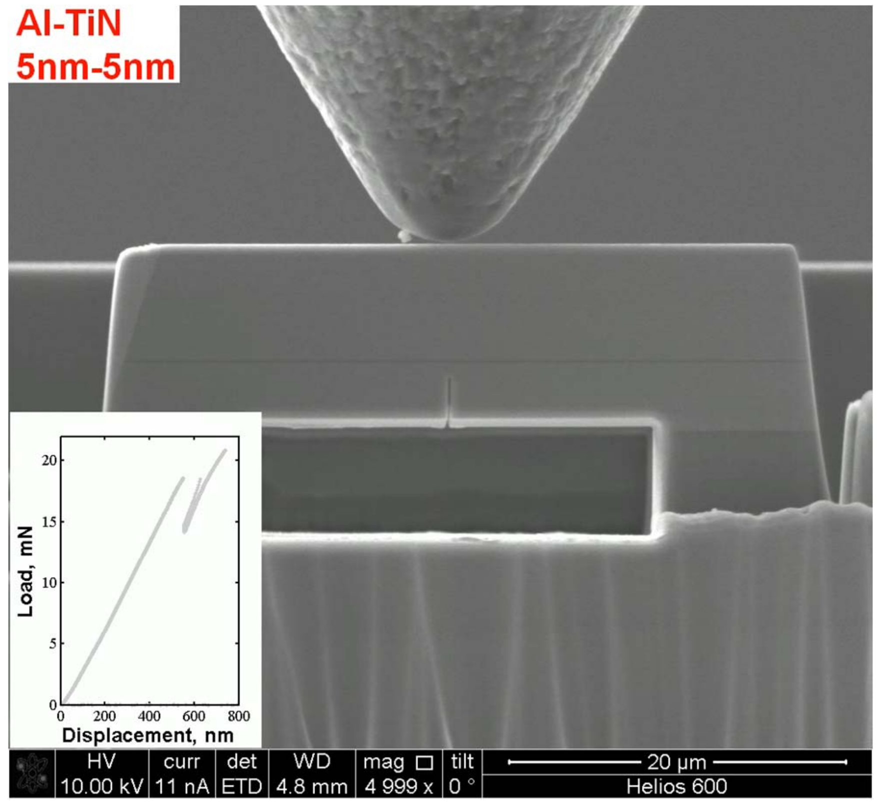
University of Nevada, Reno

Micro 3-point bending
Crack propagation in nanolaminates

Al-TiN 50 nm - 150 nm

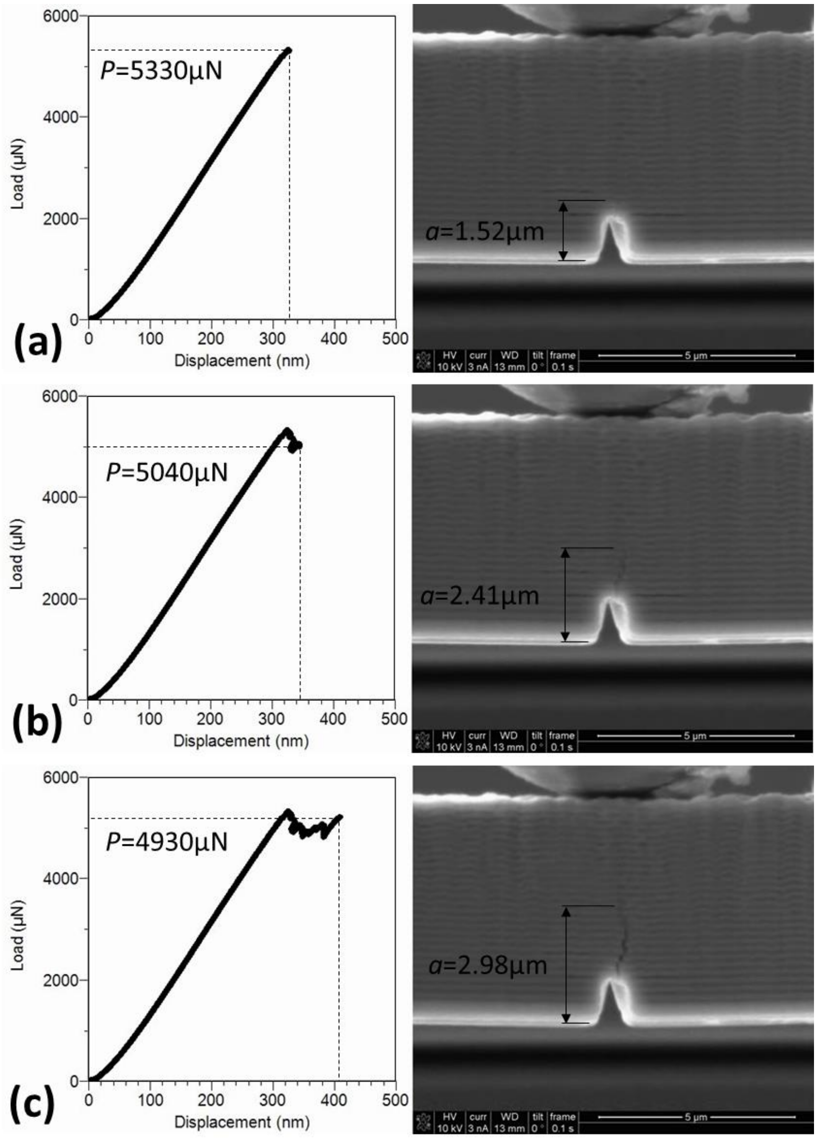


Al-TiN 5 nm - 5 nm

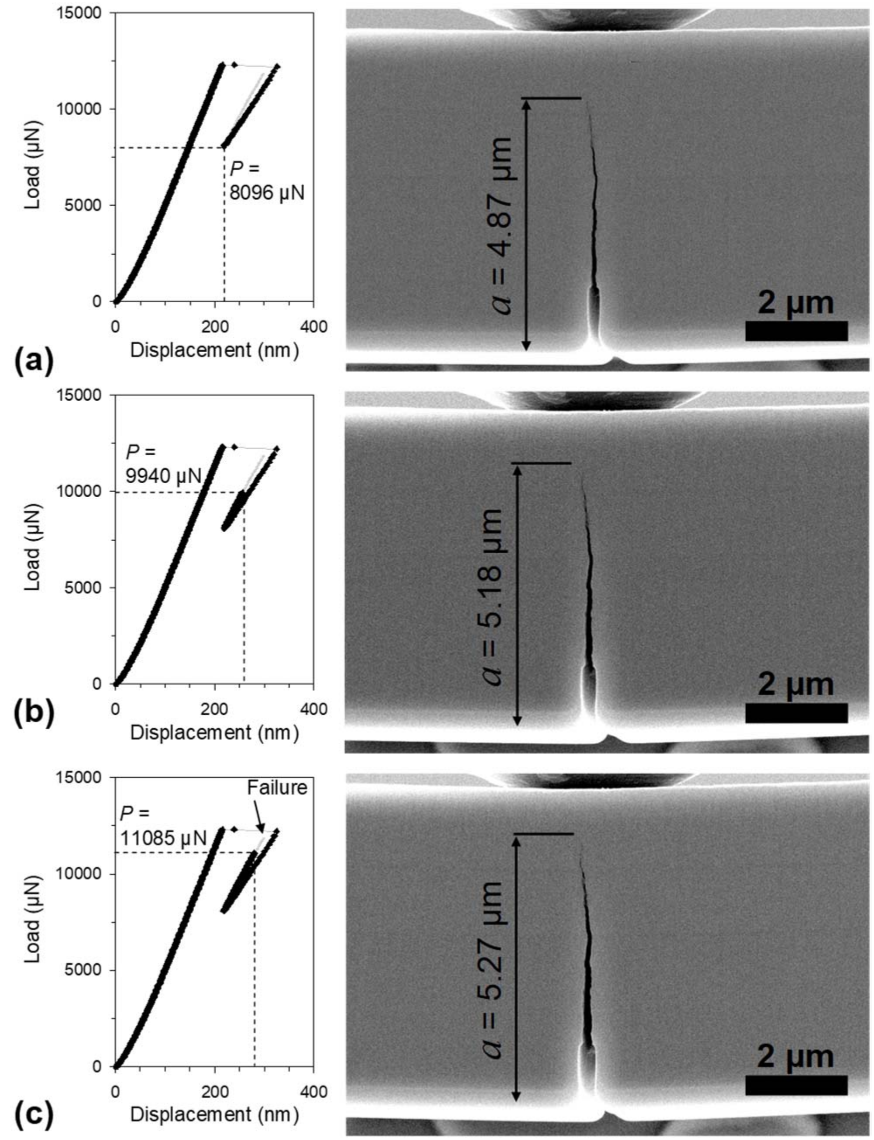


Correlating SEM frames to mechanical data shows stable crack growth under load

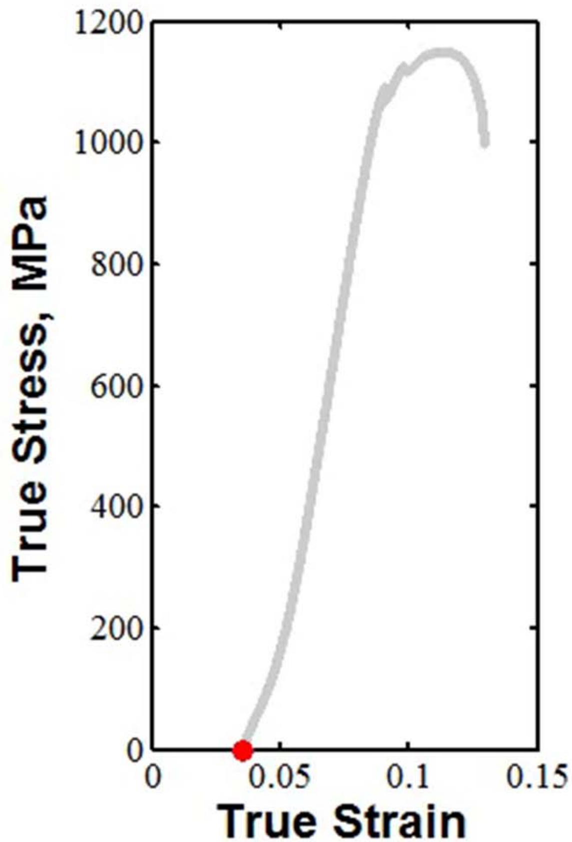
Al-TiN (150nm-50nm)

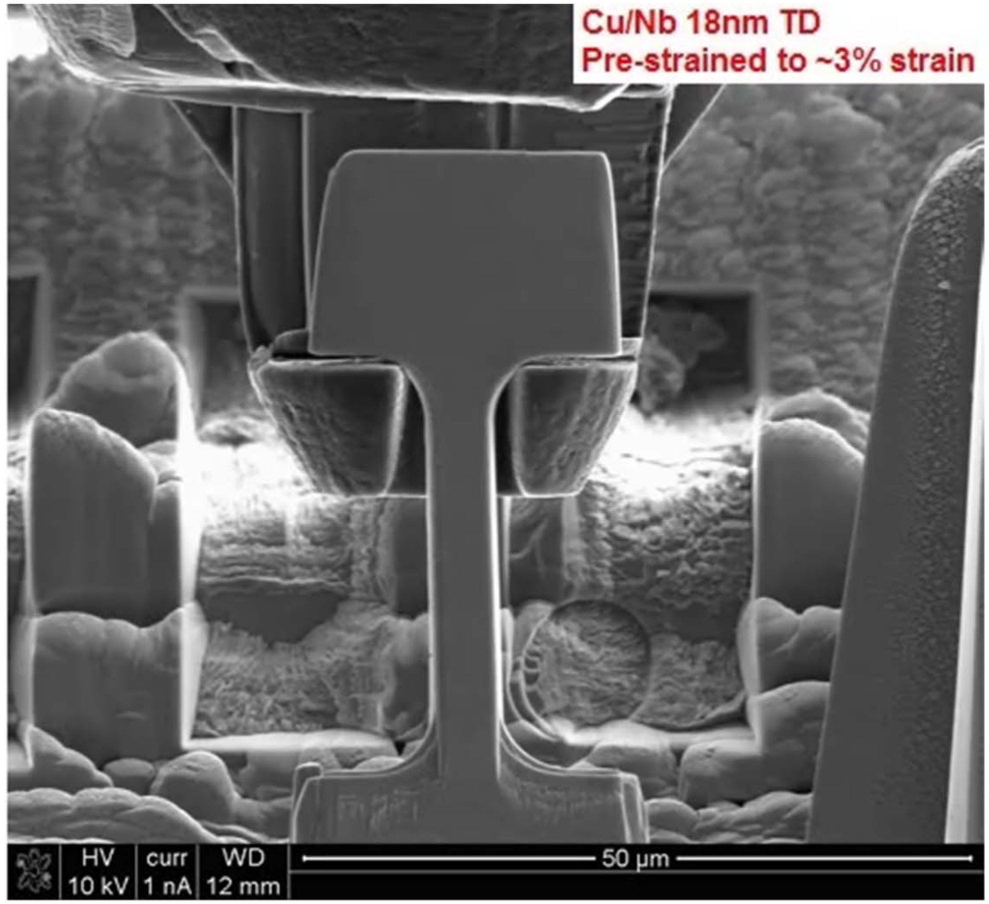


Al-TiN (5nm-5nm)

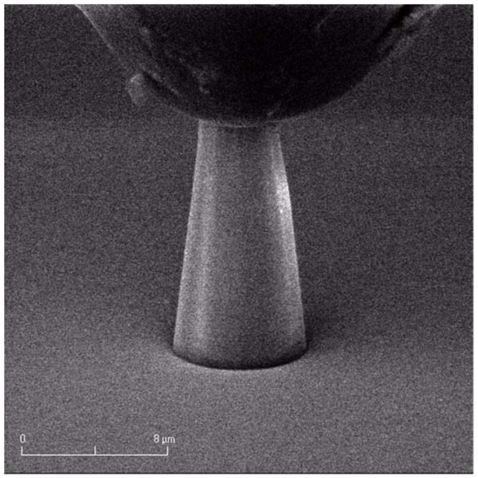


Micro-tensile testing: Cu/Nb multilayers

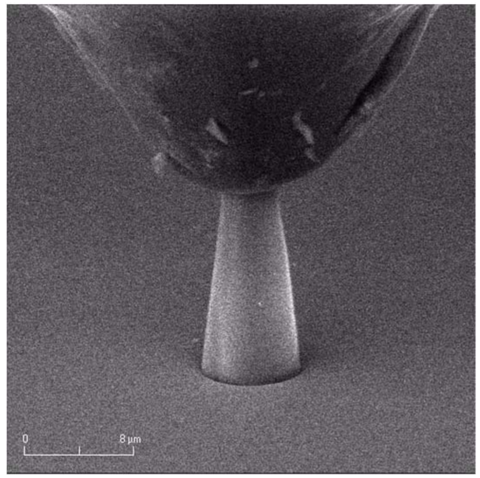




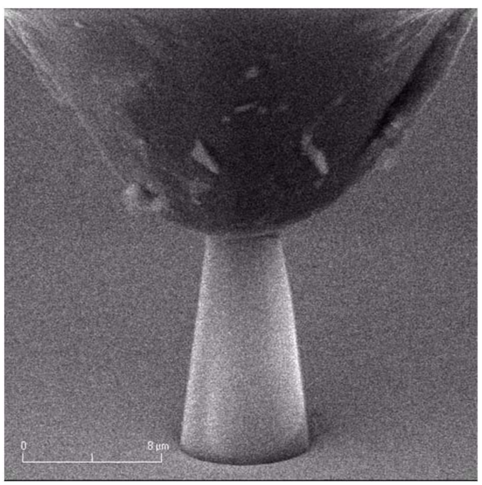
Compression testing of glass pillars at different strain rates



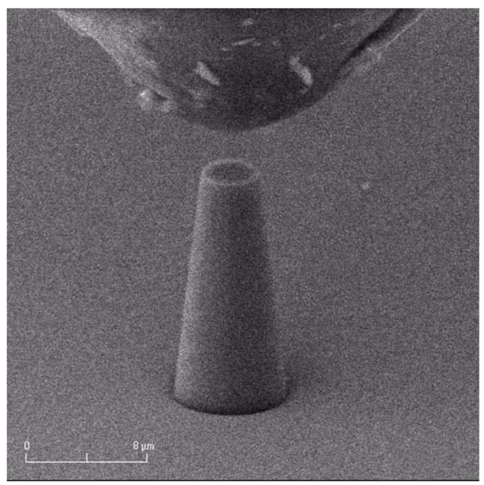
0.08/s



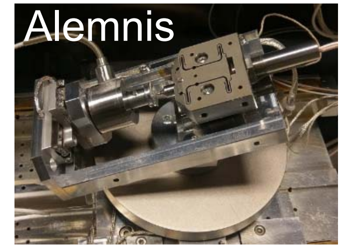
0.6/s



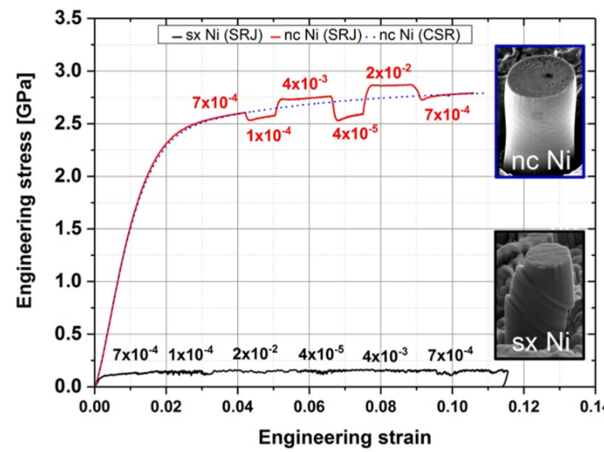
66/s



951/s



- Sample and tip heaters capable of 800°C (1000C currently under development)
- Ability to perform cryo temperature tests at -150°C (under development)

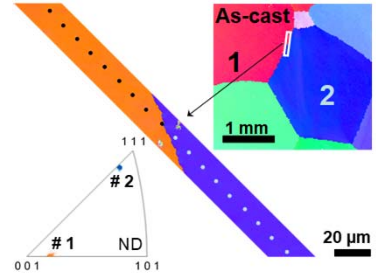


Microcompression strain rate jump tests on nanocrystalline and single crystalline Ni

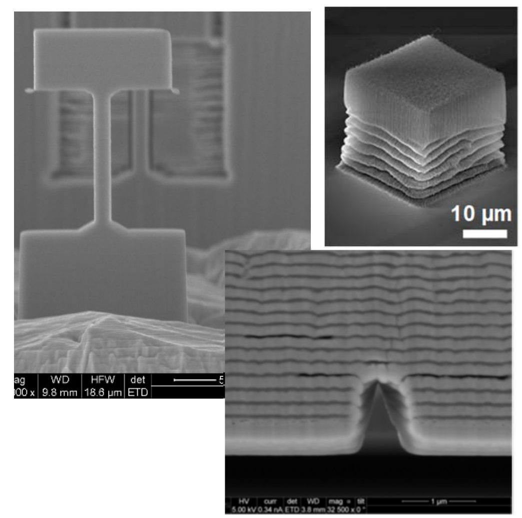
University of Nevada, Reno

Acknowledgment: Alemnis and EMPA, Switzerland

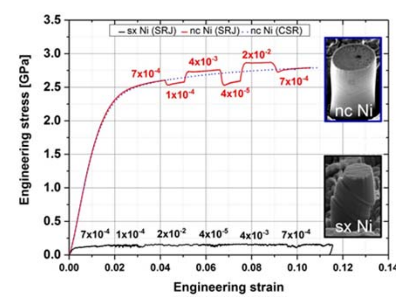
Summary of capabilities



- Spherical Indentation Stress-Strain
- *In-situ* Indentation



- *In-situ* SEM compression
- *In-situ* SEM 3-point bending
- *In-situ* SEM micro-tensile



- *In-situ* high-strain rate testing at elevated temperatures