

A new approach to materials design

Mechanical Properties of Materials under Extreme Environments

Julia R. Greer

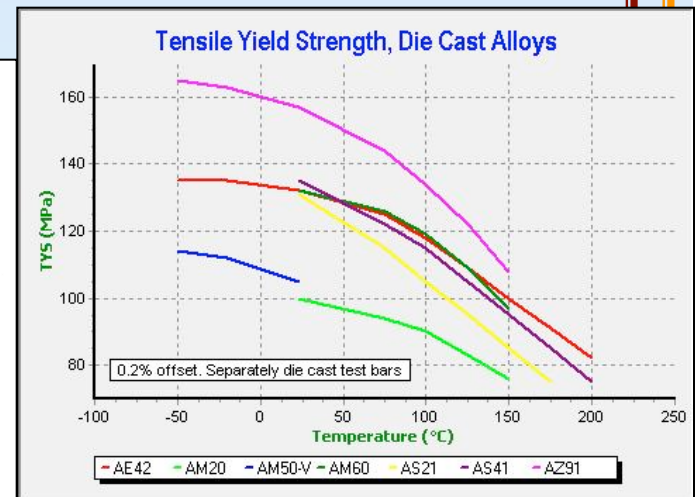
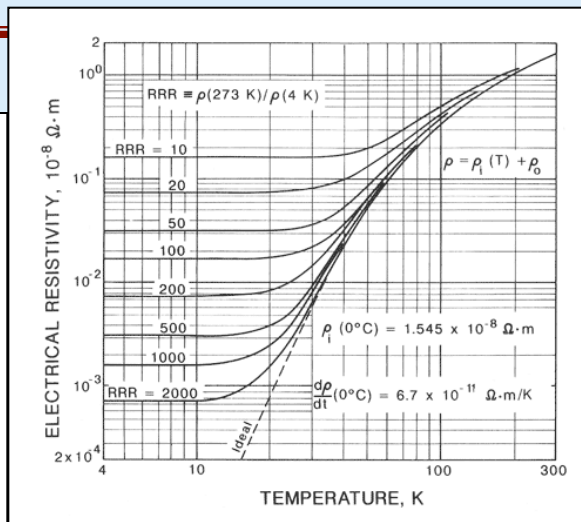
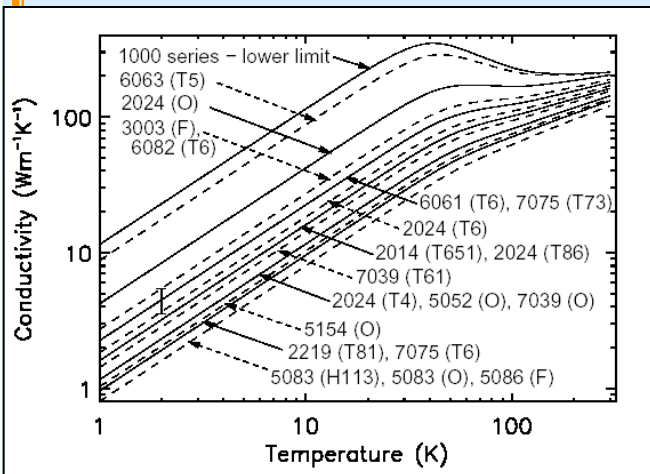
But really it's the work of Andrew T. Jennings,
Ju-Young Kim, Dongchan Jang, Shelby Hutchens, and Mingyuan Huang

**Division of Engineering and Applied Science
California Institute of Technology**



Why do materials matter for Titan?

- Material properties change significantly with temperature
- Variation can be very non linear
- Many materials are unsuitable for low temperature use
- Large amounts of contraction can occur at cryogenic temperatures.
- Successful design requires attention to property variation
- Extensive literature on low-temperature material properties
- **Important:** When it doubt - TEST



Some points to consider:

=> Impact on alignment => Development of interferences between dissimilar materials => Increased strain and possible failure => Impact on wiring



Conventional materials for low-T

Acceptable

- Austenitic stainless steels (304, 316, 321)
- Al alloys (6061, 6063, 1100)
- Cu (OFHC, ETP)
- Brass
- Fiber reinforced plastics (G –10, 11)
- Nb and Ti (used in superconducting RF systems)
- Invar (Ni /Fe alloy) (low α)
- In (used as an O ring material)
- Kapton and Mylar (insulation)
- Quartz (used in windows)

Unacceptable

Martensitic stainless steels -
Undergoes ductile to brittle transition when cooled down.
Carbon steels – also becomes brittle.
Rubber, Teflon and most plastics

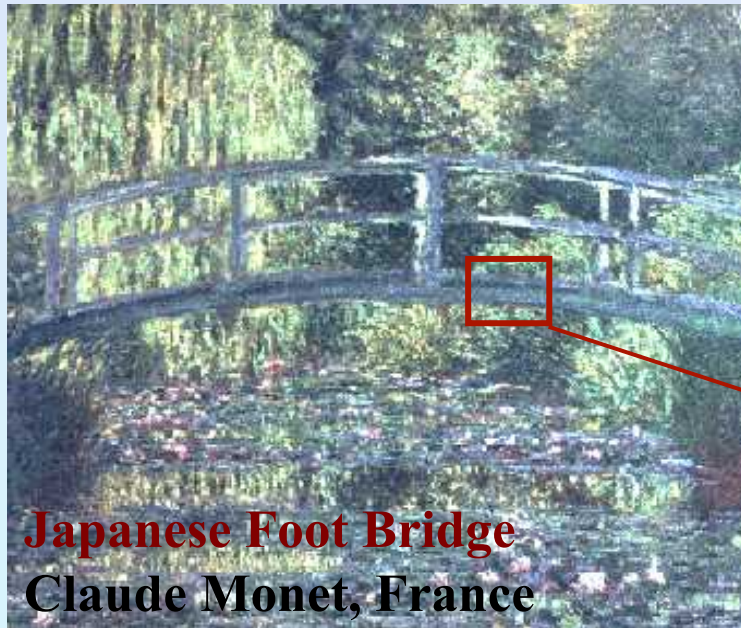
**Conventional bulk materials may pose limitations
need to develop new paradigm of material design =>
META-MATERIALS**



Materials and Length Scales

“We are leaving the age of reductionism and entering the age of emergence...”

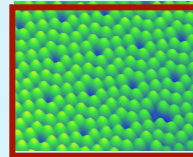
-from “A Different Universe” R. B. Laughlin (Nobel Laureate, Physics 2005)



Japanese Foot Bridge
Claude Monet, France



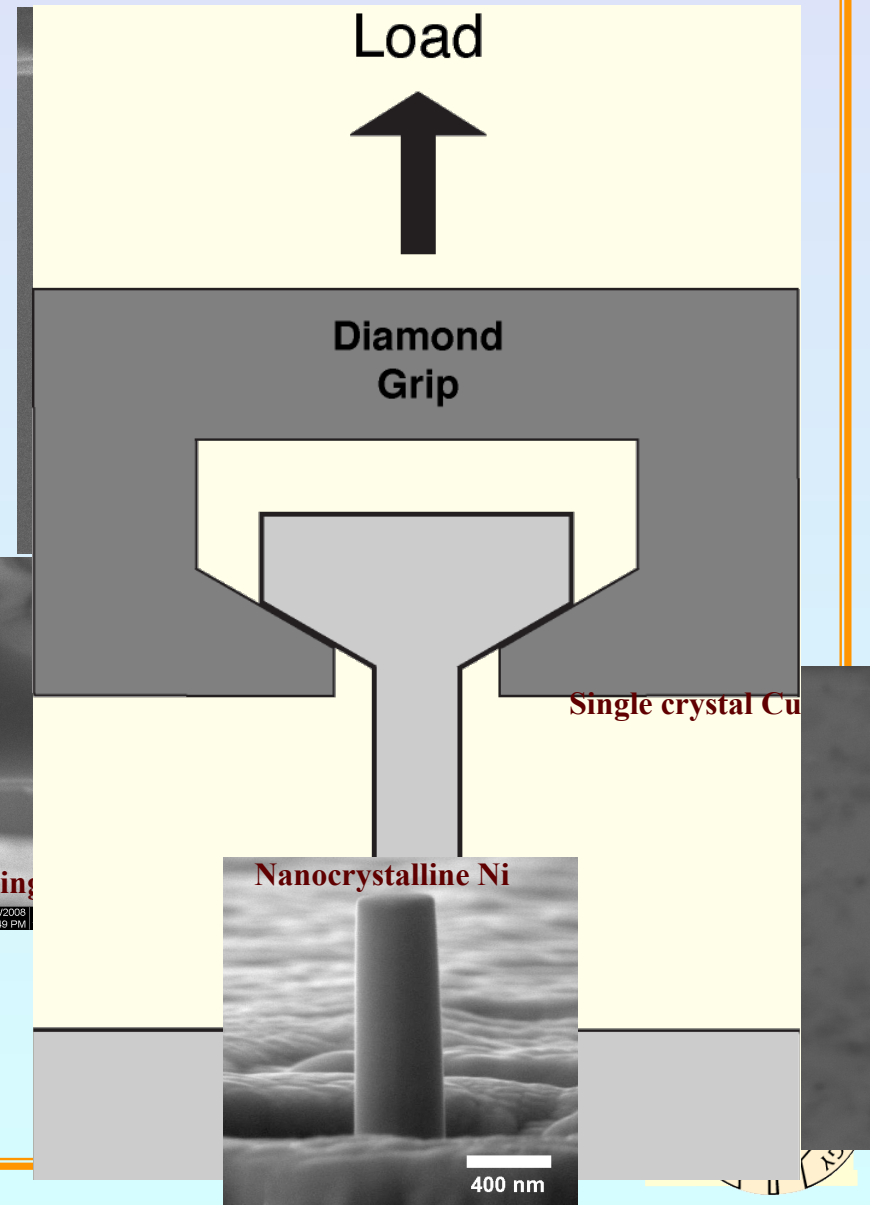
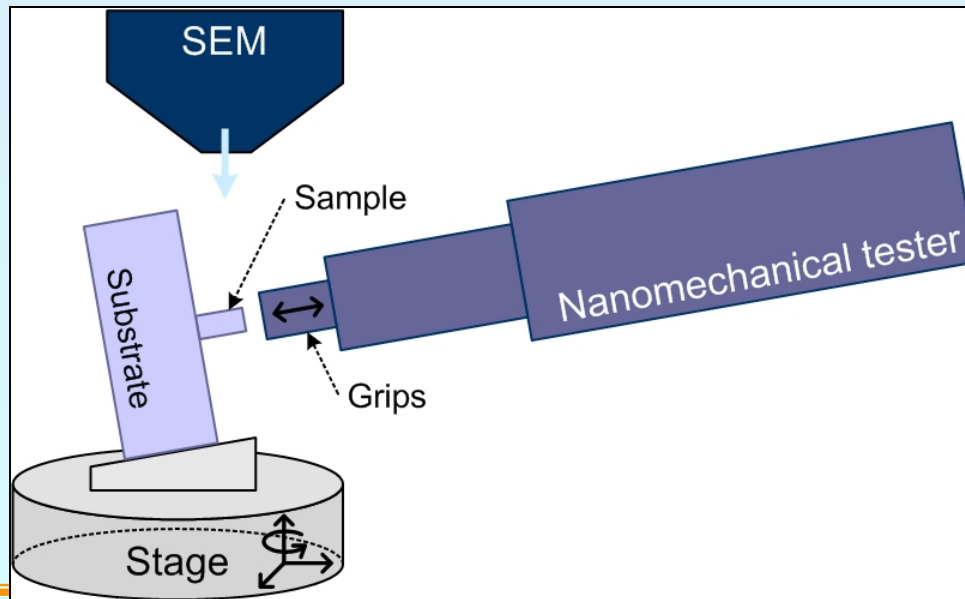
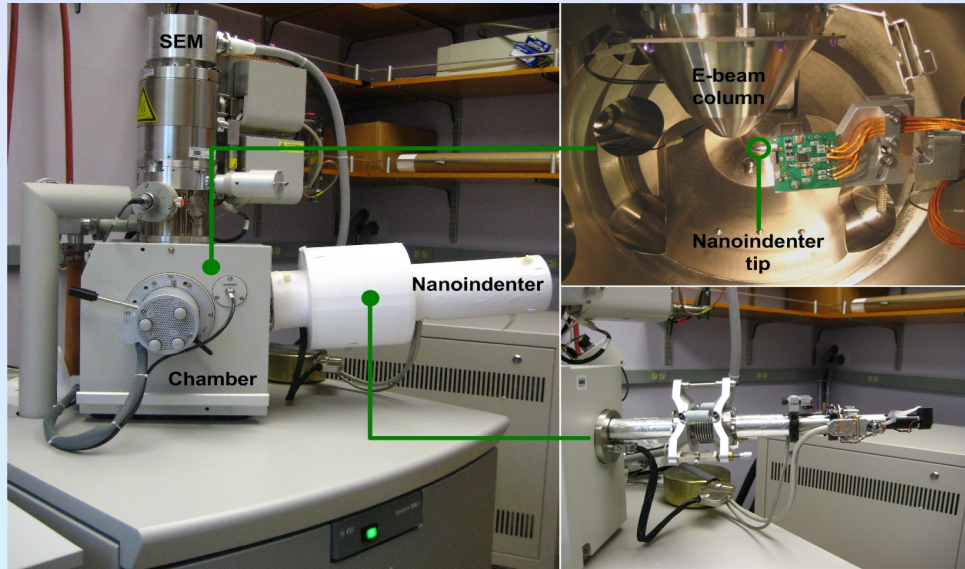
Golden Gate Bridge
San Francisco, CA



In nanocrystals (quantum dots, nanowires, nanotubes, etc.)
size modification tunes a variety of properties: optical, electronic, plasmonic,
thermal, acoustic, etc. which
brings into question **material structural integrity**



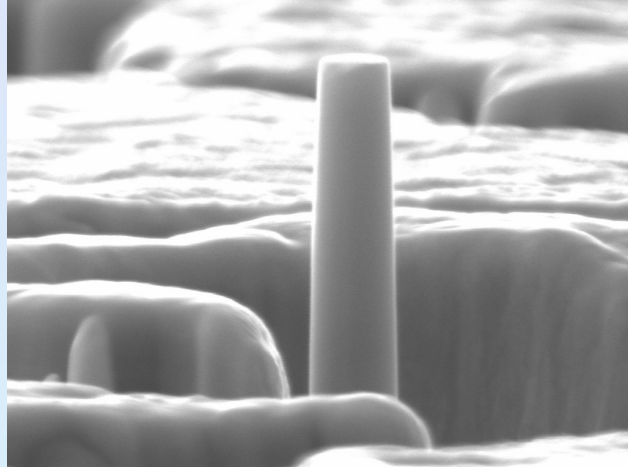
In-situ tension and compression SEMentor: SEM + Nanoindenter



I. Fundamental Mechanical Properties of Materials: Variety of materials, mainly metals...

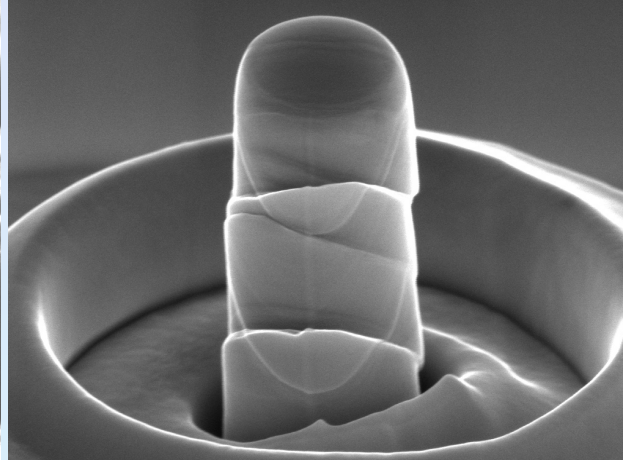
Fabrication Method: Focused Ion Beam (FIB)

Nano-crystalline Ni nano-pillar



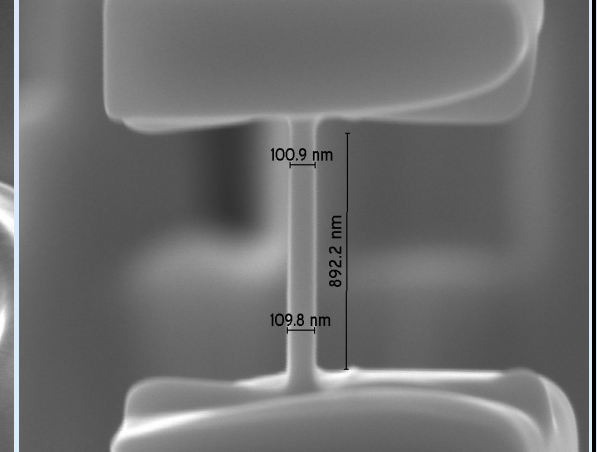
9/25/2008 HV mag WD HFW det 1 μ m
1:15:25 PM 10.00 kV 50 000 x 10.9 mm 2.98 μ m ETD Quanta FEG

Compressed Nb nano-pillar



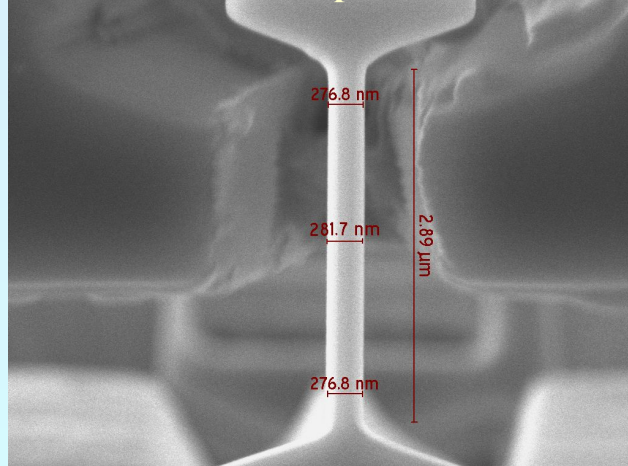
8/29/2008 HV det mag WD 1 μ m
10:40:47 PM 10.00 kV TLD 80 000 x 5.1 mm xt780

Tensile metallic glass sample



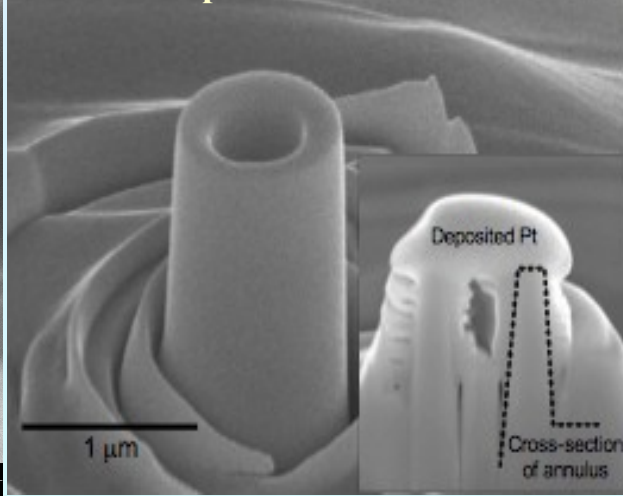
6/11/2009 HV mag WD HFW det 500 nm
7:09:42 PM 10.00 kV 130 000 x 10.1 mm 2.30 μ m ETD

Tension of Au nano-pillar

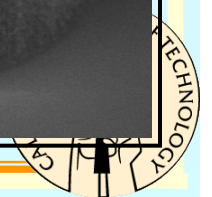
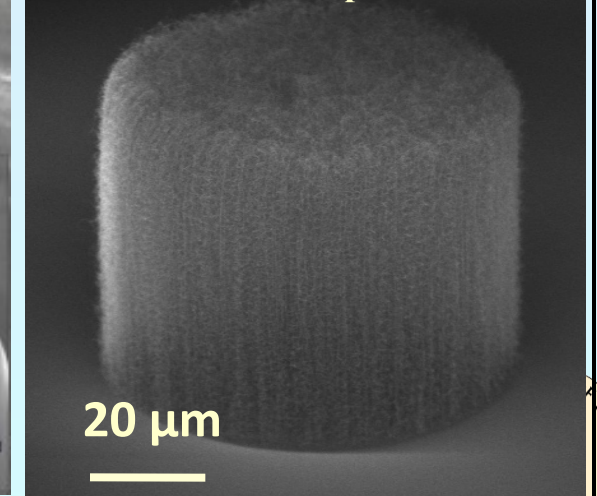


12/23/2008 HV mag WD HFW det 1 μ m
1:46:49 PM 10.00 kV 60 000 x 11.8 mm 4.97 μ m ETD

Mo "anti-pillar"

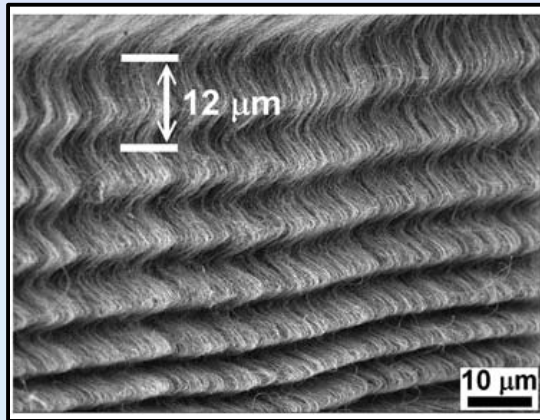


CNT foam micro-pillar



Protective Applications: Carbon Nanotube Foams

Energy Absorbing Protective Layers



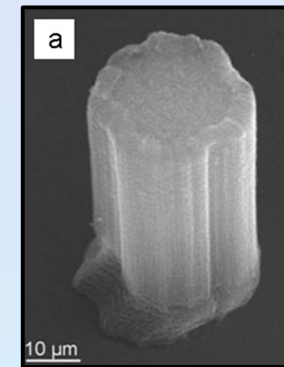
Cao, A., et al. *Science* (2005)
Pathak, S., et al. *Carbon* (2009)

CNT foam characteristics

=> CNTs appear vertically aligned at lower magnification

=> Individual tubes are intertwined at higher magnification

MEMS devices



e.g. contact thermal switch materials

Cho, J., et al. *J Micromech Microeng* (2008)

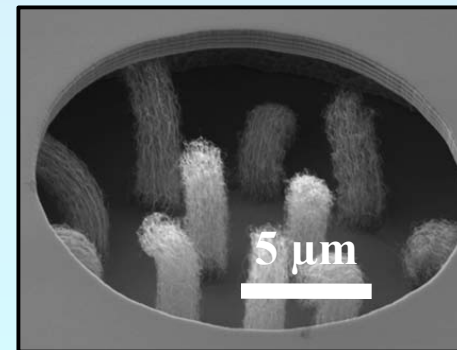
Light Absorbing Coatings

Darkest Man-Made Material, 0.045% reflectance



Yang, Z.-P., et al. *Nano. Lett.* (2008)

Low Vacuum Field Emission Sources

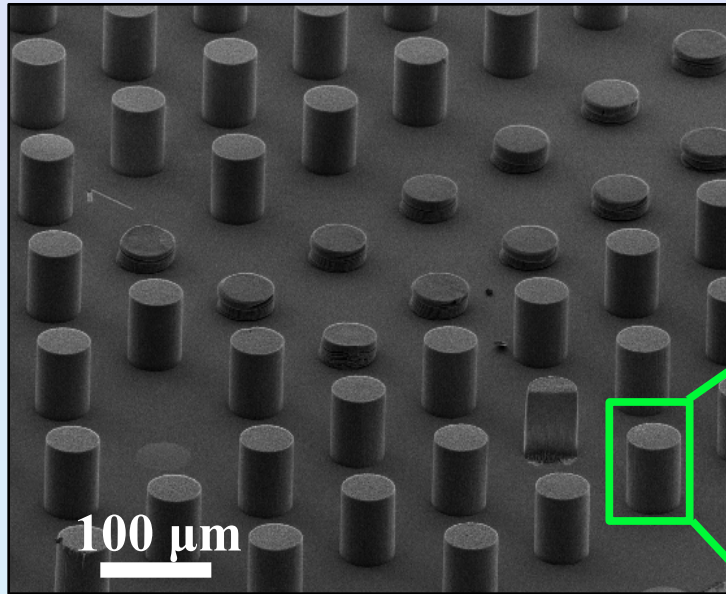


e.g. cold cathode array

Manohara, H. M., et al. *J Infrared Milli TeraHz Waves* (2009)

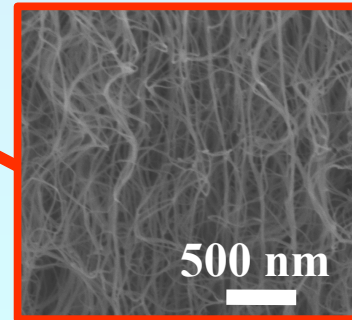
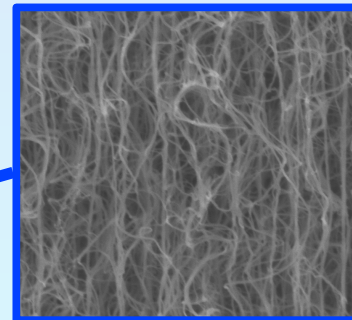
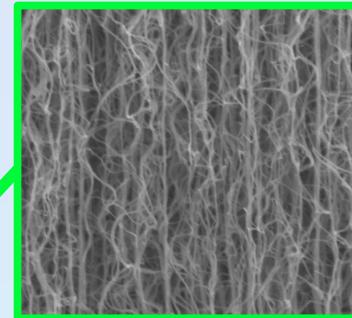
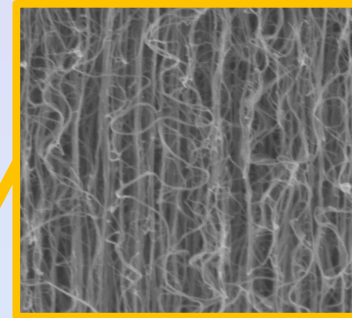
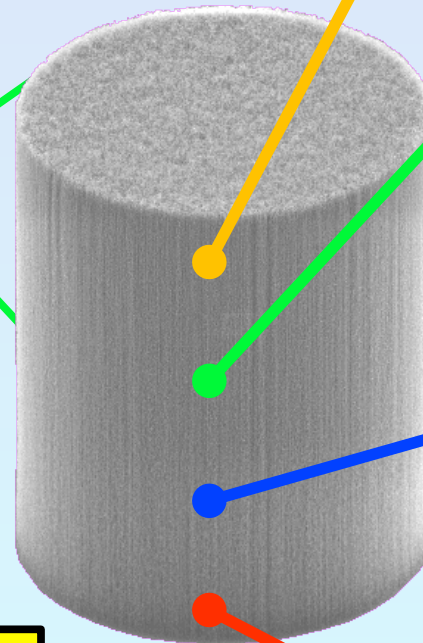


CNT Bundle Morphology



Growth Conditions:
200 Torr 675 °C 15-20 min.
Precursor: Ethylene
Catalyst: Fe

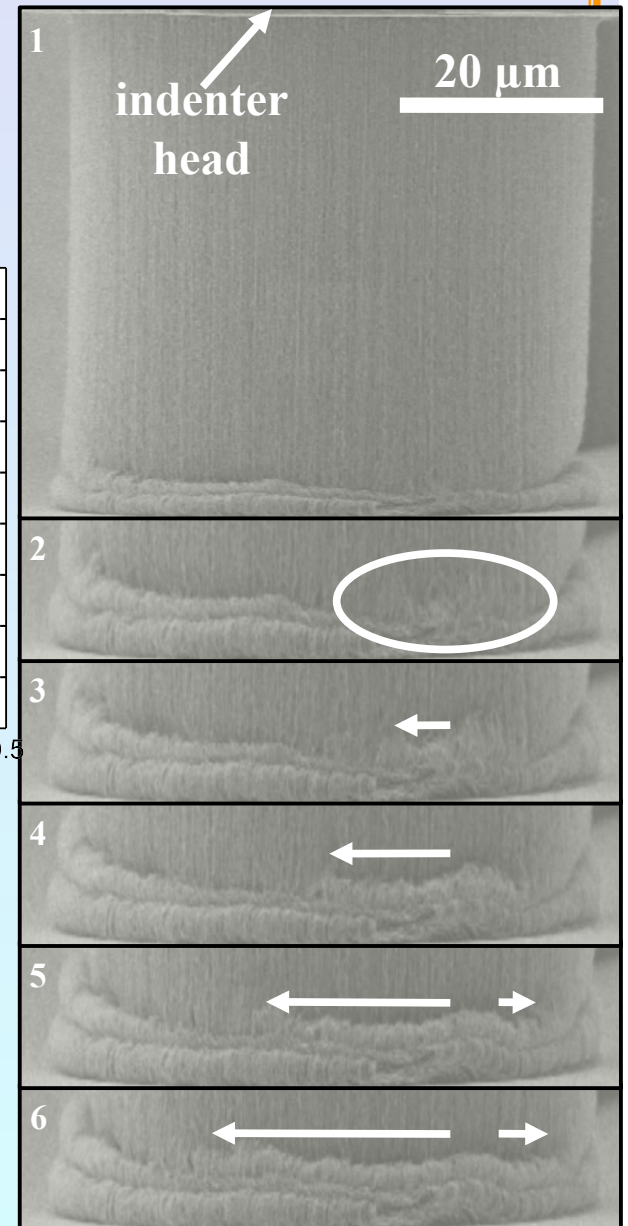
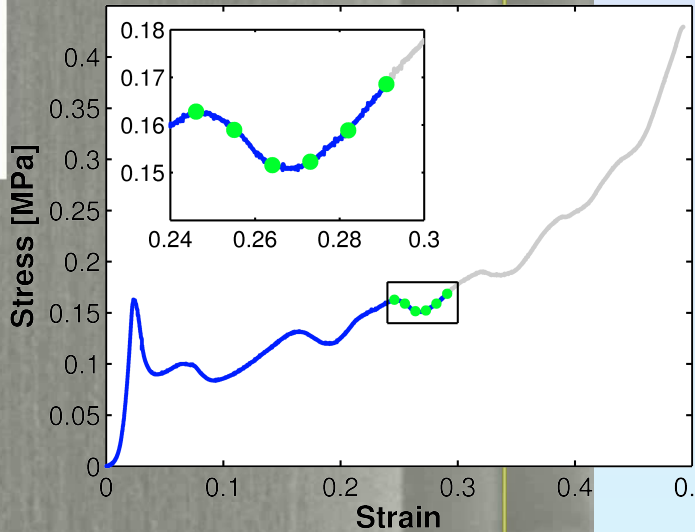
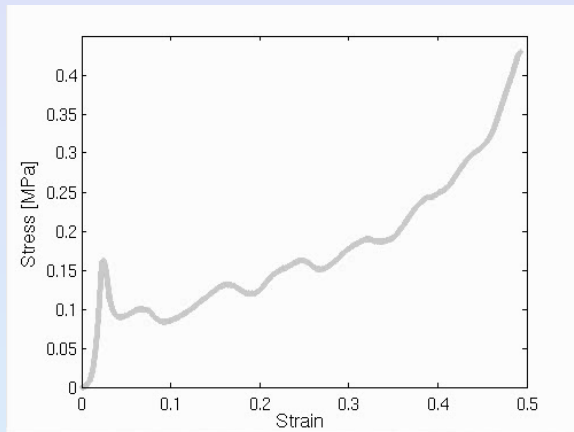
**Unambiguous calculation of
macroscopic stresses and strains
under uniaxial compression**



Increasing Density, Alignment, and Stiffness



In situ Micro-Compression



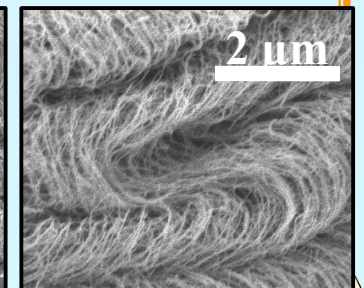
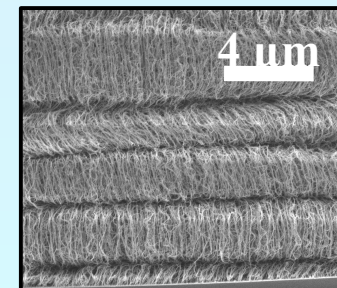
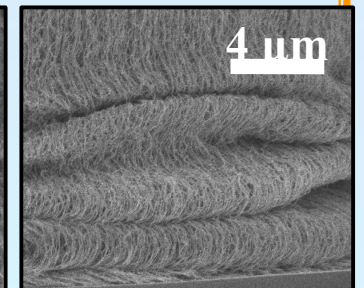
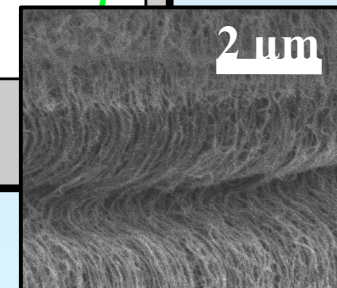
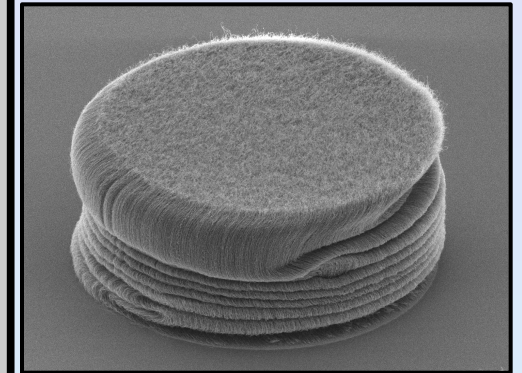
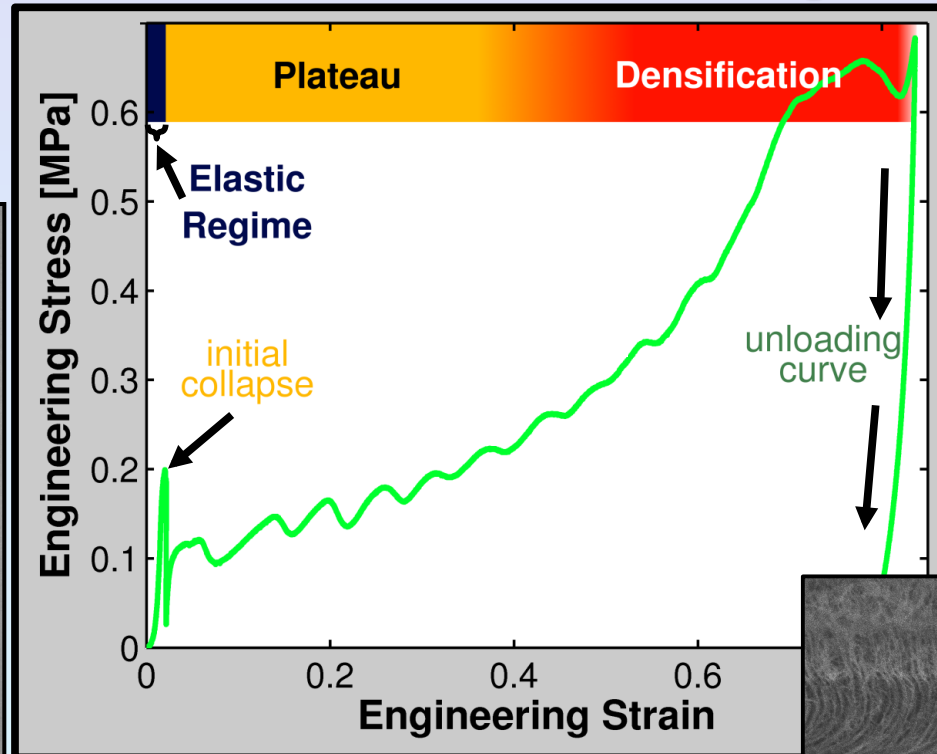
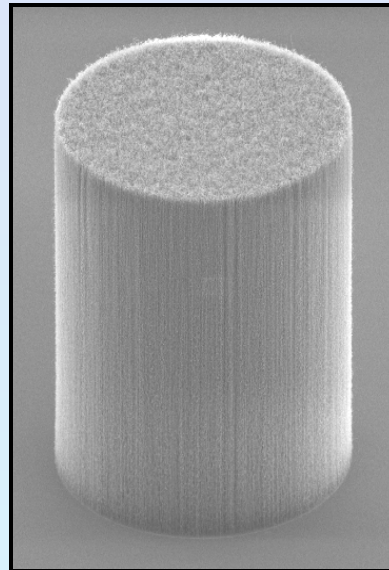
0.001 s^{-1}

6 μm of undeformed pillar collapses to approximately 2 μm of buckle

10/30/2009
0:02:36 AM

- => Buckles at pillar bottom same size as those at top
- => Buckle size is independent of strain rate

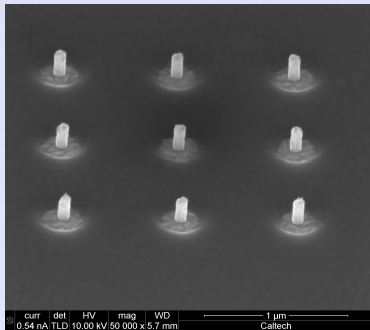
Stress-Strain Response



- => Repeated folding in compressed structures
- => Overall foam-like behavior
- => Plateau undulations indicate collective buckling

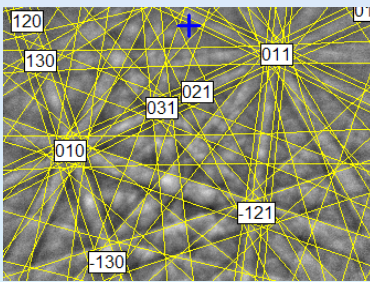
Buckled tubes do not appear to fracture or break, only bend

Metals: Microstructure Control through Novel Synthesis

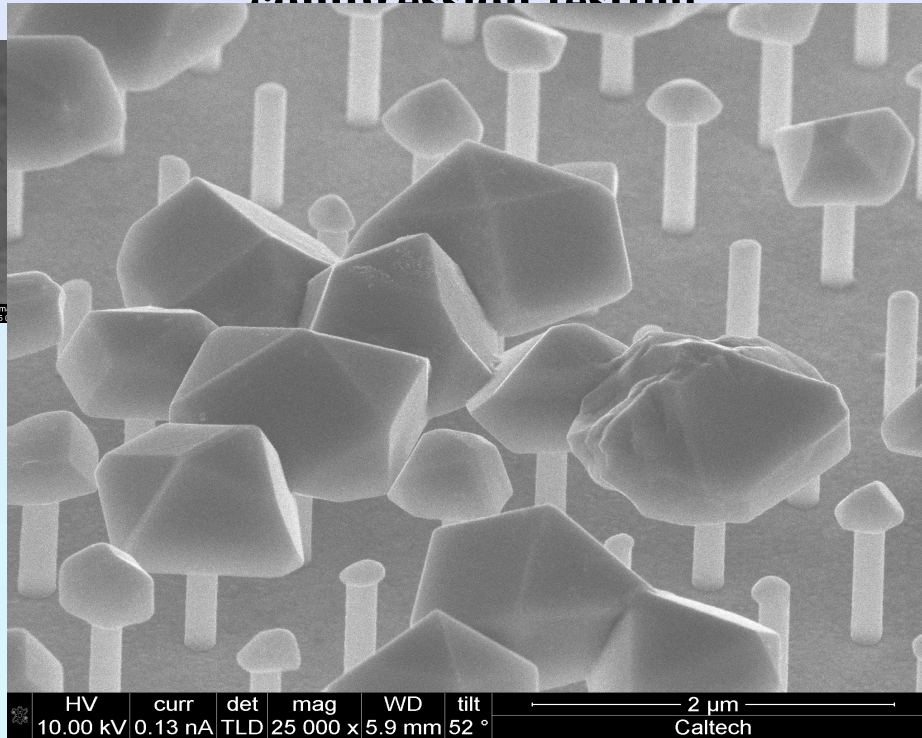


Nano candy canes!

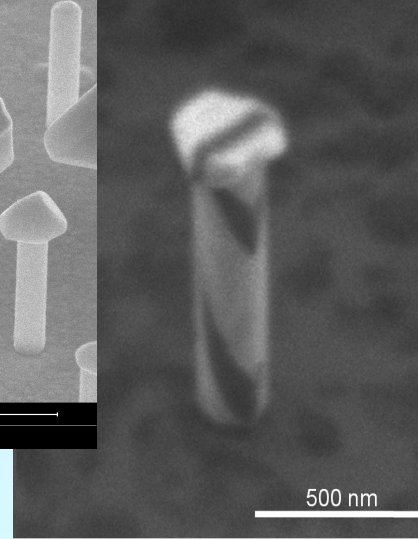
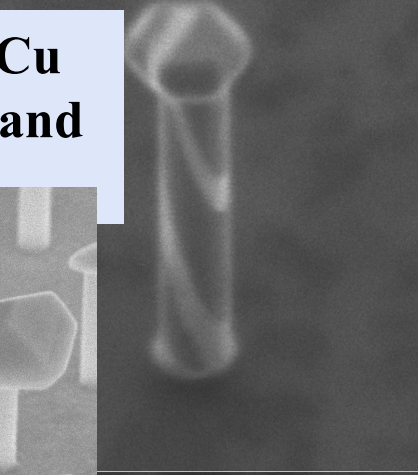
Array of intentionally over-plated Cu single crystalline pillars for tension and compression testing



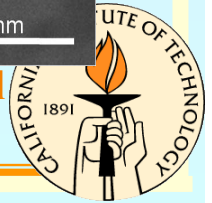
Nanocrystalline Au



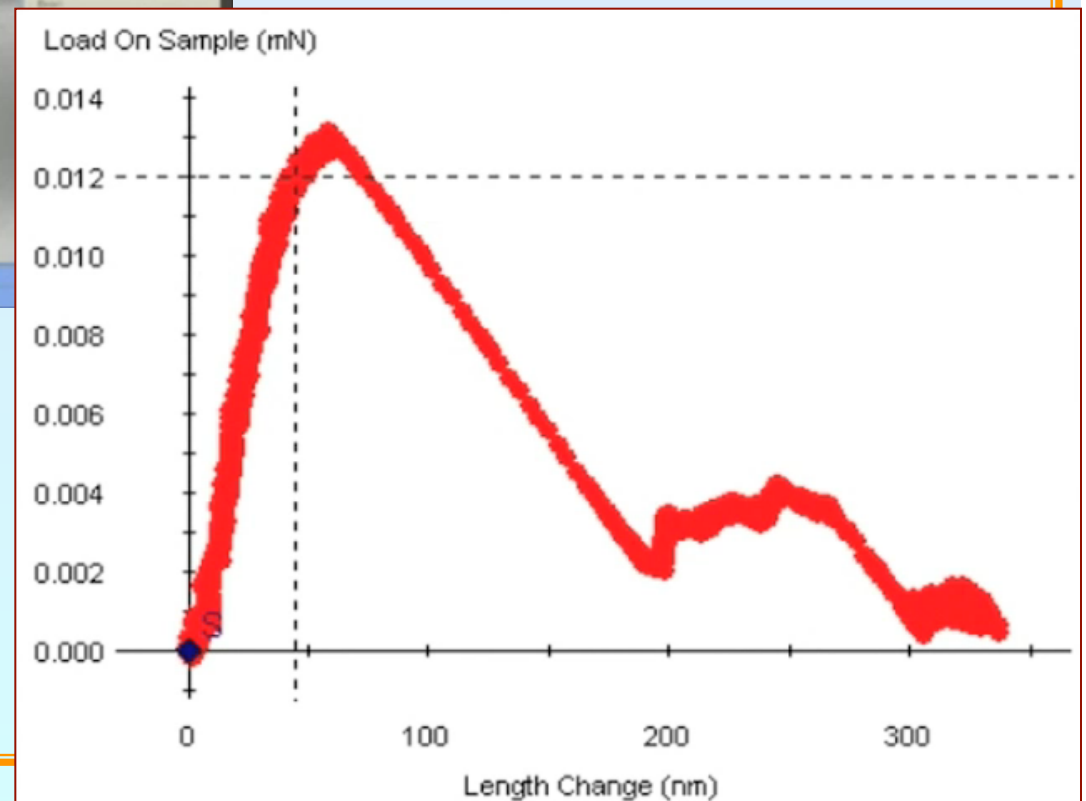
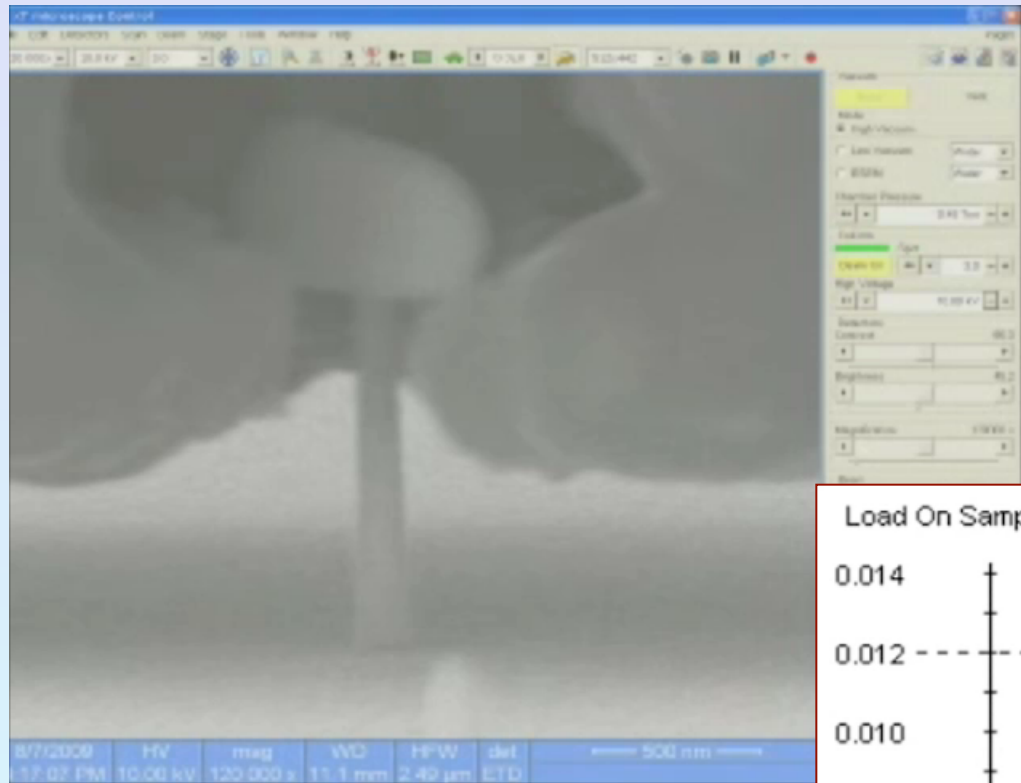
Nanotwinned Cu



Cu with occasional twin boundaries



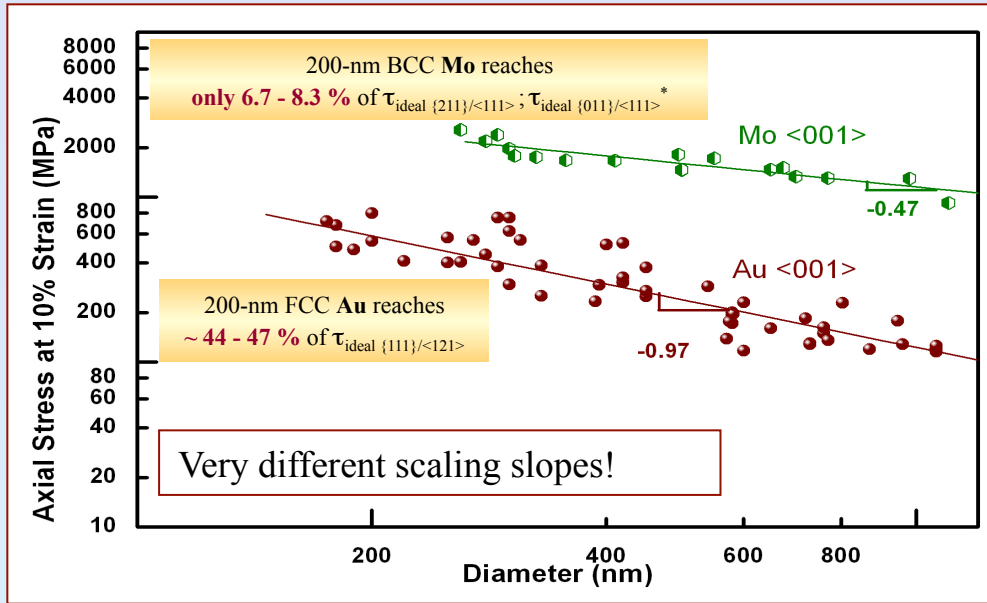
Tensile deformation of Cu at nano-scale



A.T. Jennings and J.R. Greer *Phil Mag* (submitted, 2010)

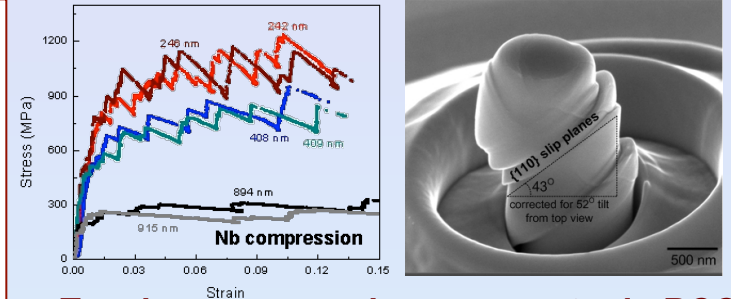
Scaling Laws Single Crystals

*W. Cai (recent calculations)
 *S. Ogata, et al *Phys. Rev. B* 70, 104104 (2004)
 *W. Luo, et al *Phys. Rev. B* 66, 094110 (2002)

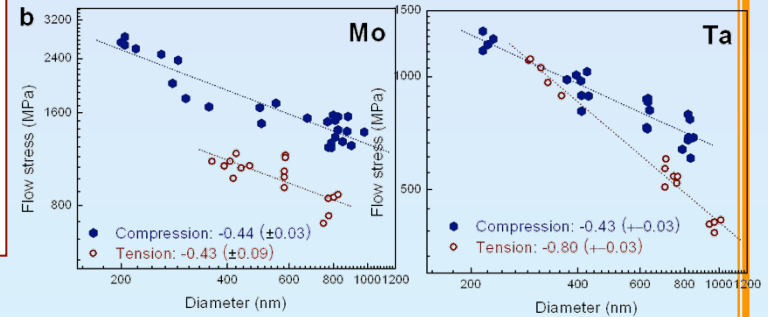


Brinckmann, Kim, Greer, *Phys Rev Lett* (2008)

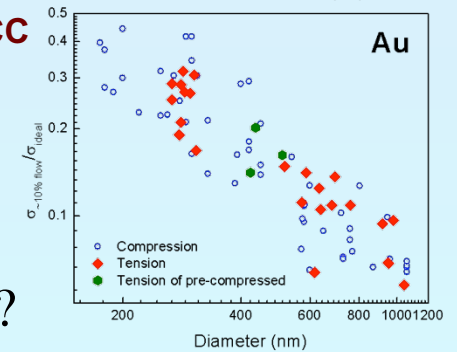
Stochastic discrete strain bursts



Tension-compression asymmetry in BCC



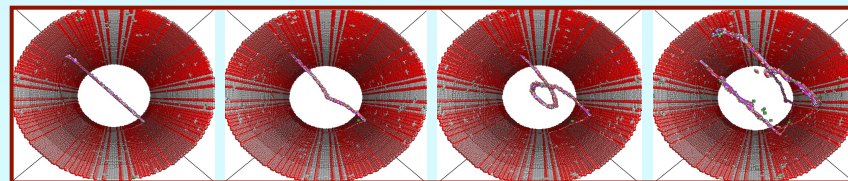
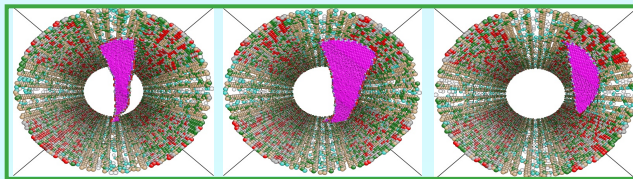
But not in FCC



Smaller is Stronger

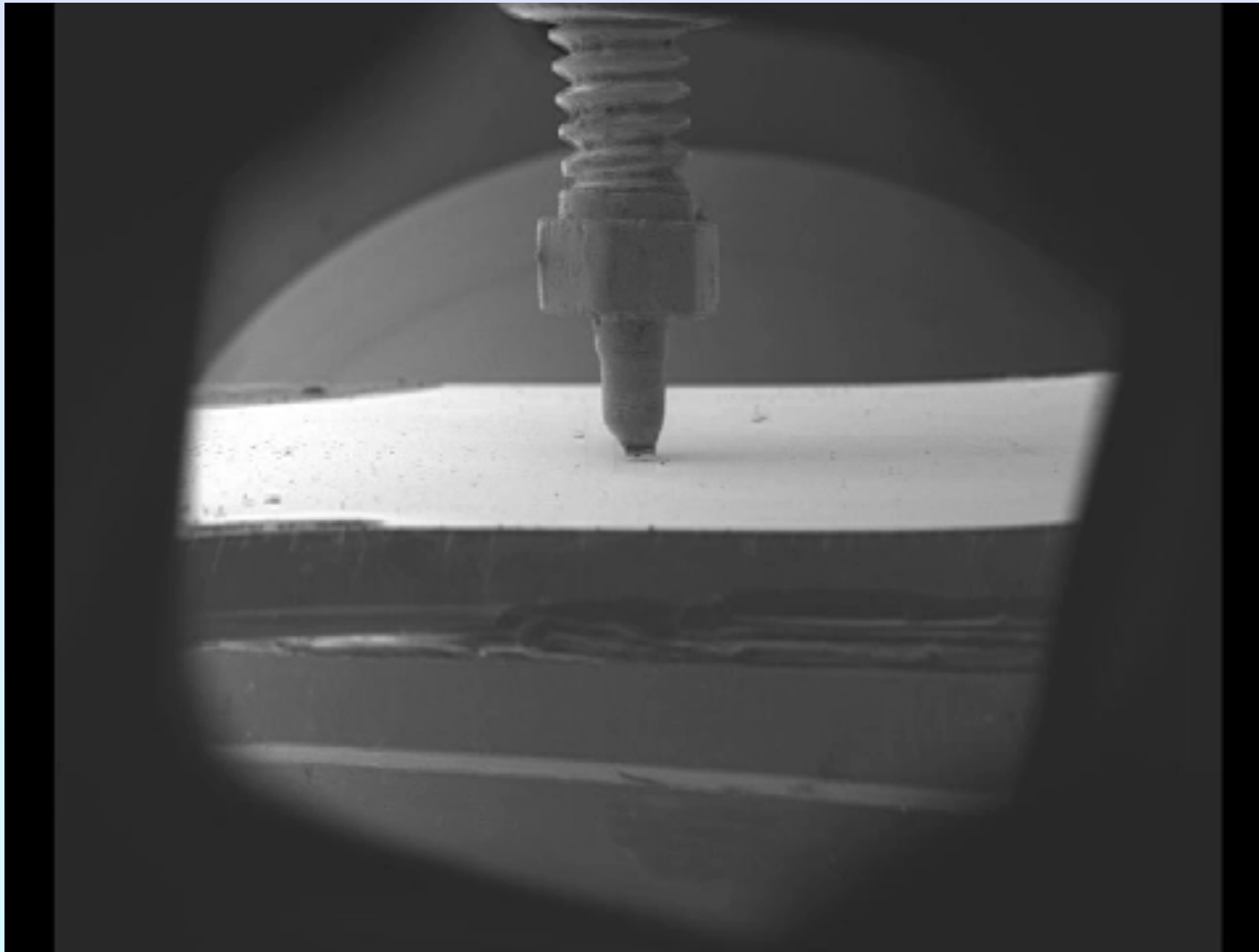
Perhaps fundamentally different plasticity mechanisms:

Dislocation starvation in FCC while Junction formation in BCC?



Insights into deformation mechanisms: TEM

Wait, but how do we TEM the same nano-pillars before and after deformation???

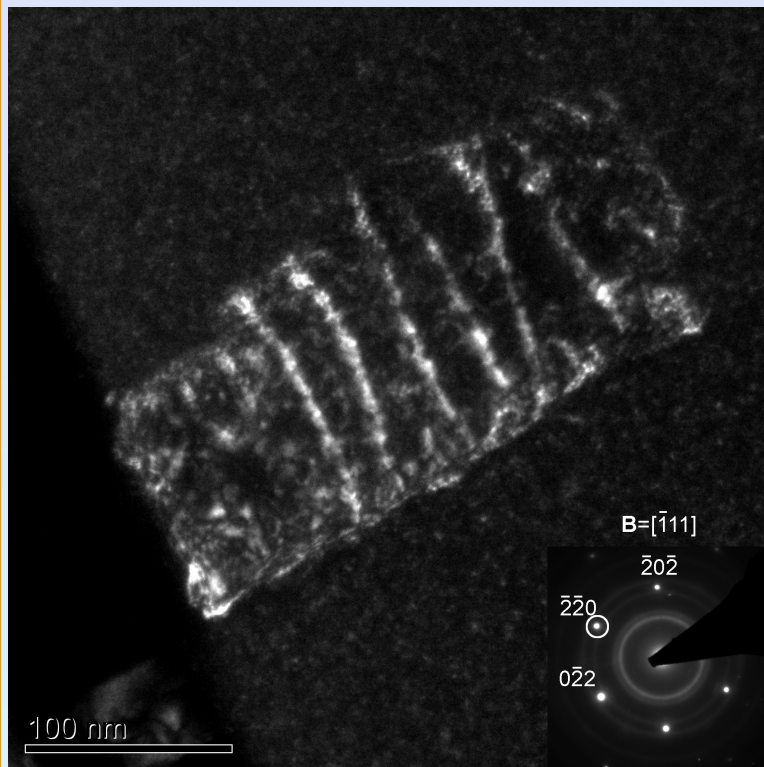


We test them directly on TEM grid!



TEM Analysis of Deformed Pillars

Post-compression Copper

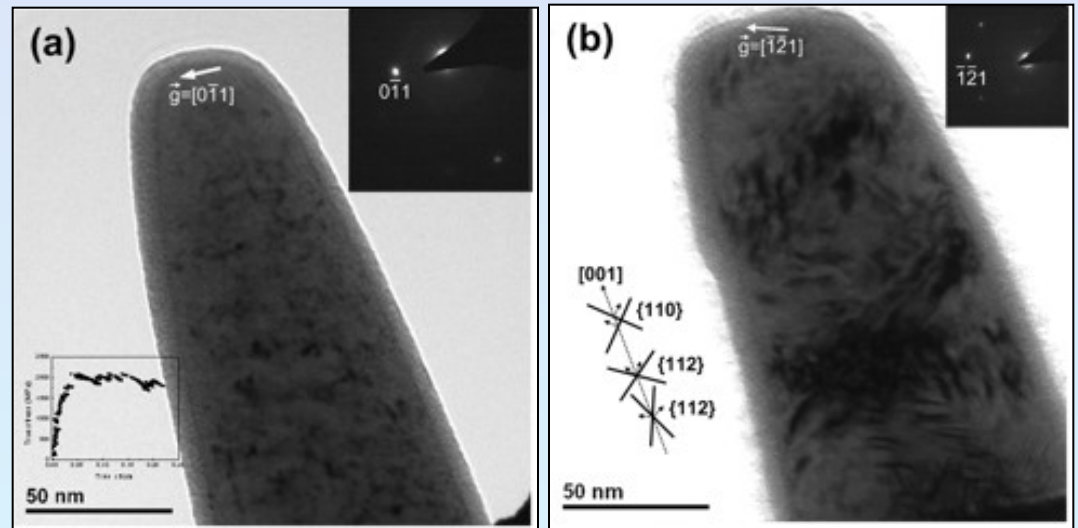


$g \cdot b$ condition =>

Burgers vector = $[1-21]$

Dislocations observed only in planes with no resolved shear stress!

Niobium



Complex dislocation network, junctions, straight parallel segments



II. Fundamental Mechanical Properties of Materials: Amorphous Metallic Glasses

- Basic plasticity units: shear transformation zones (STZs)

- collective atomic rearrangement
- ~ 100 atoms (~ 5 atoms in each direction)

- Macroscopic plastic deformation governed by spatial and temporal distribution of STZs

- uniform distribution → **homogeneous deformation** (high T & low σ)

- densely populated within narrow region → **shear localization, or shear bands** (low T & high σ)

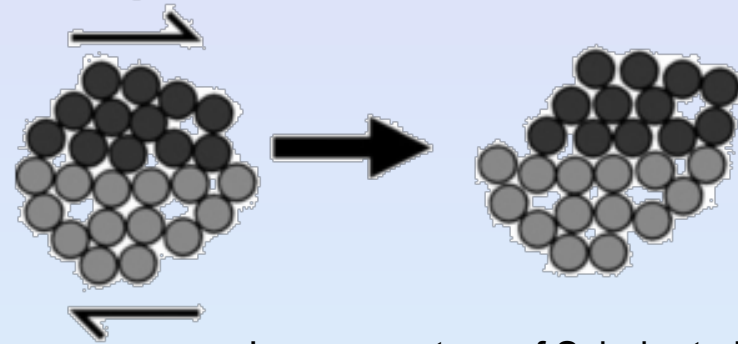
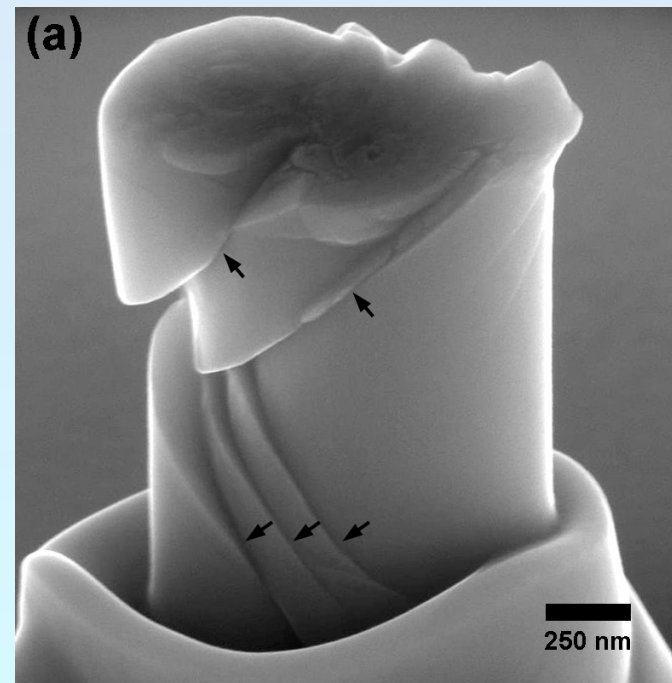


Image courtesy of Schuh et al.

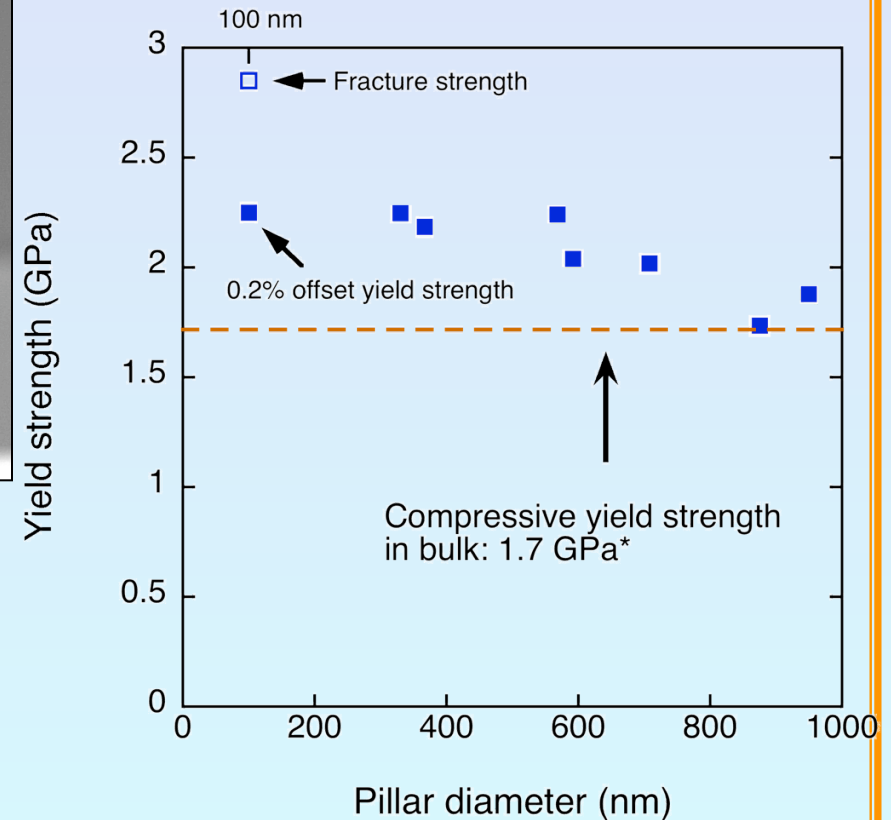
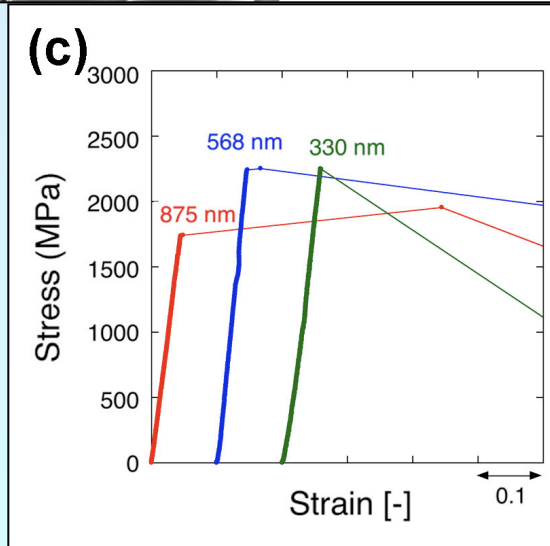
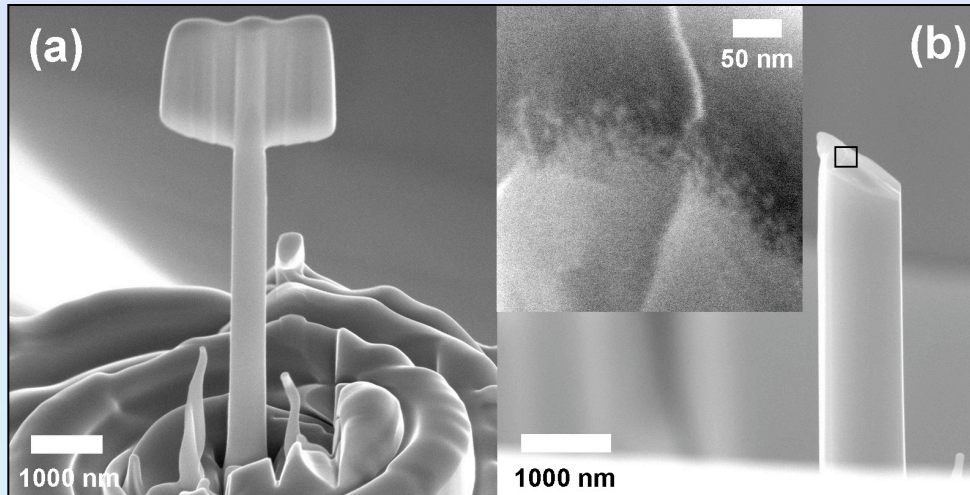


Shear bands formation under load

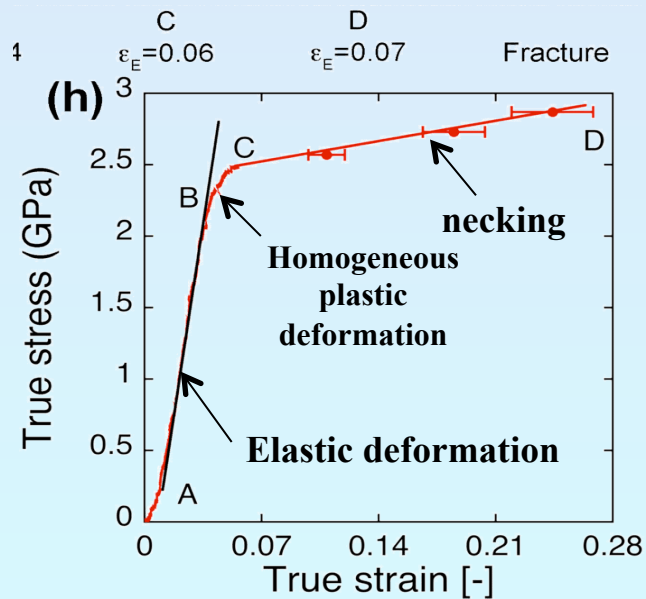
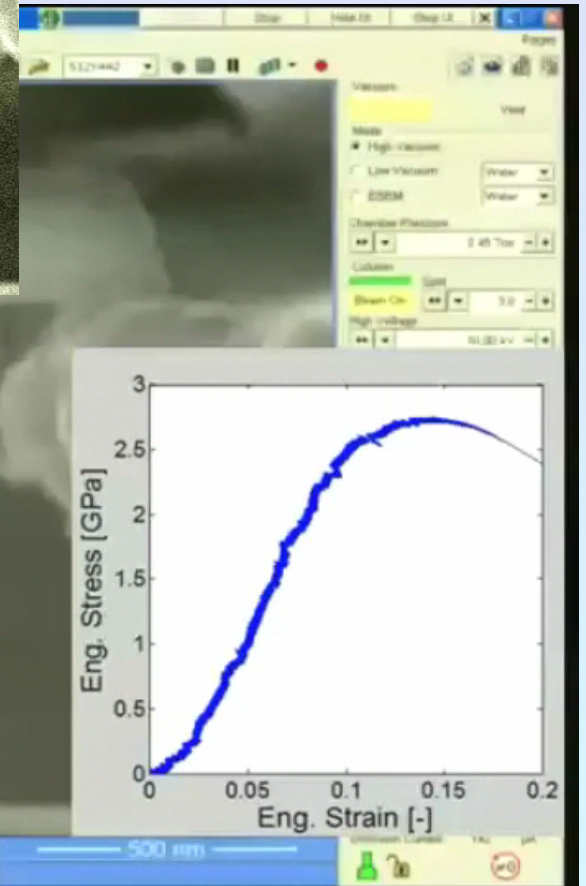
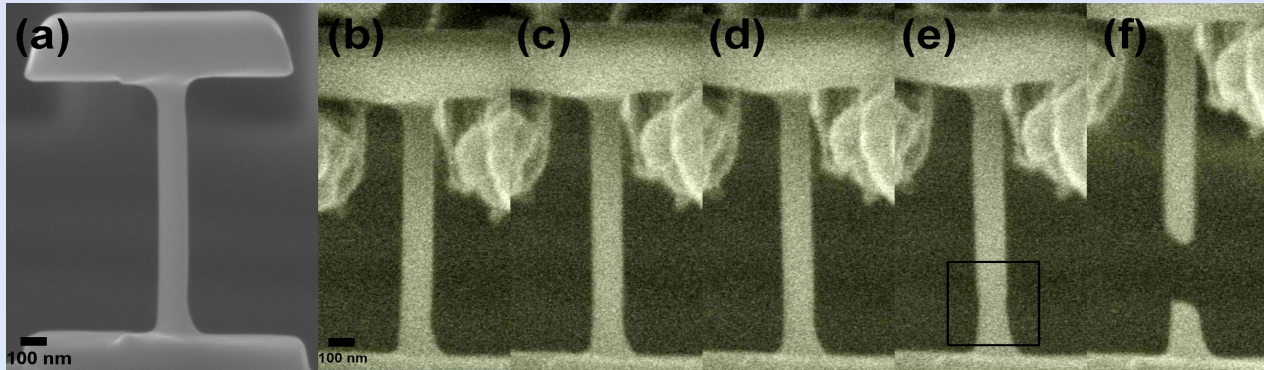


Uniaxial tension of nano-pillars: $D=200\text{nm} +$

Generally fail by catastrophic failure due to shear band formation and propagation



Uniaxial tension at 100nm diameter



- ❖ Homogeneous plastic deformation
- ❖ Necking
- ❖ Work-hardening

Transition to homogeneous deformation mode: stronger and ductile at nano-scale!



Summary and Acknowledgements

- Single metallic crystals at nano-scale exhibit strong size effects in uniaxial compression and tension: **SMALLER IS STRONGER**
- 100nm metallic glasses exhibit enhanced strength and ductility at nano-scale
- Carbon nanotube (CNT) foams can be used in protective application due to their superior energy absorption
- Band gap in graphene might be induced by mechanical perturbation

For details/publications: <http://www.jrgreer.caltech.edu/>



NSF Career Grant
DMR-0748267



JPL: Lee Hall

Columbia U: Jim Hone



Award No: N000140910883



Army Research Office
Institute for Collaborative
Biotechnologies (ICB)



DMR-0520565



Cryogenic module construction

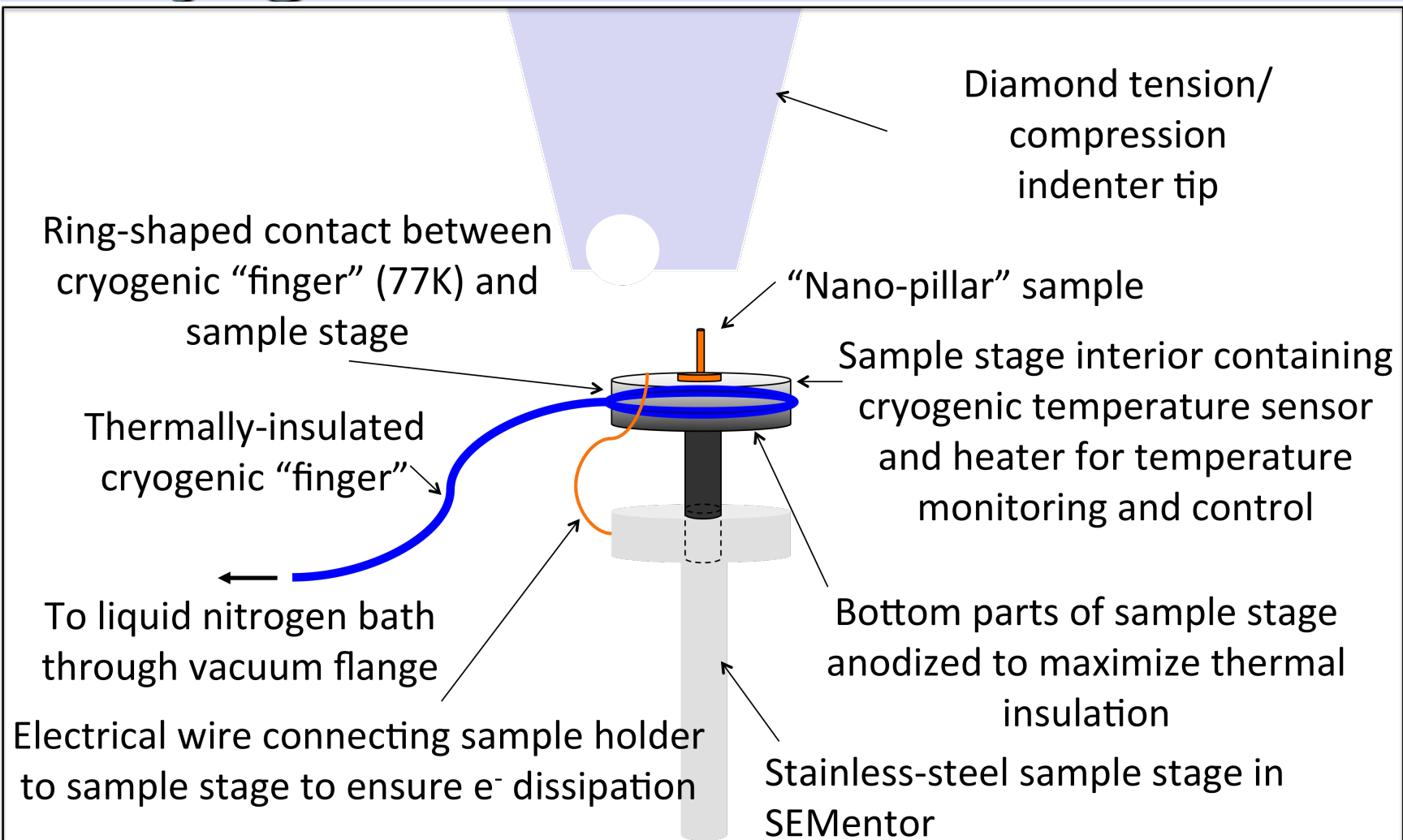
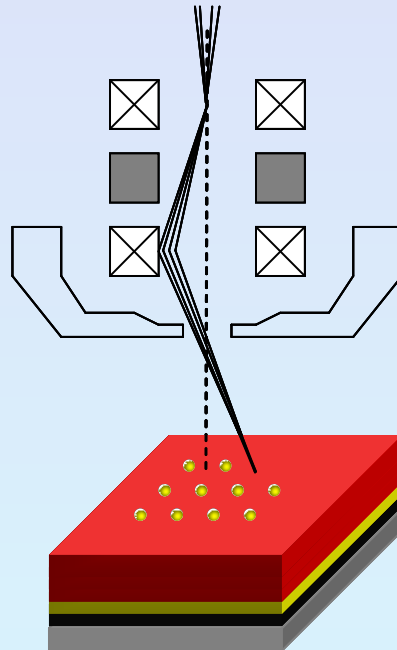


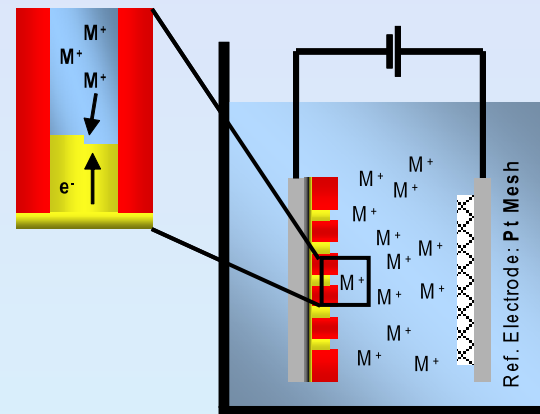
Figure 13. (not to scale) Schematic of cryogenic cooling configuration showing sample stage, sample holder (sample on top), ring-shaped contact between cryogenic finger and sample, conducting wire to dissipate electrons from e-beam, and indenter tip.

FIB-less fabrication: E-beam Litho => Electroplating

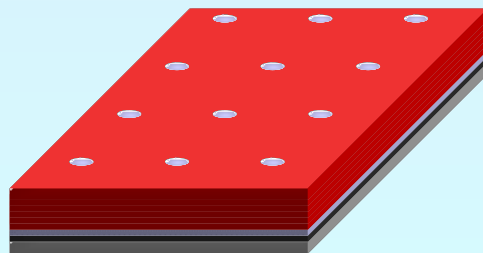
1. E-beam patterning



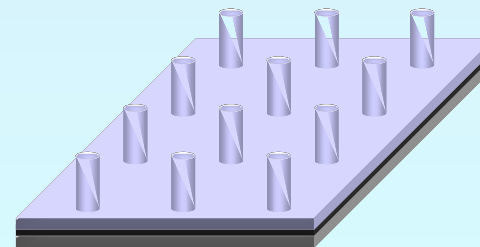
2. Electroplating



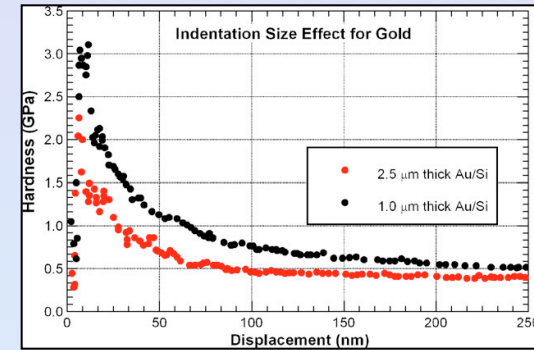
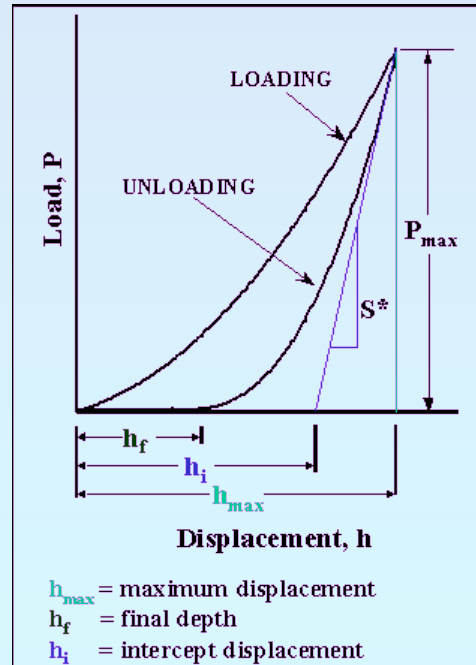
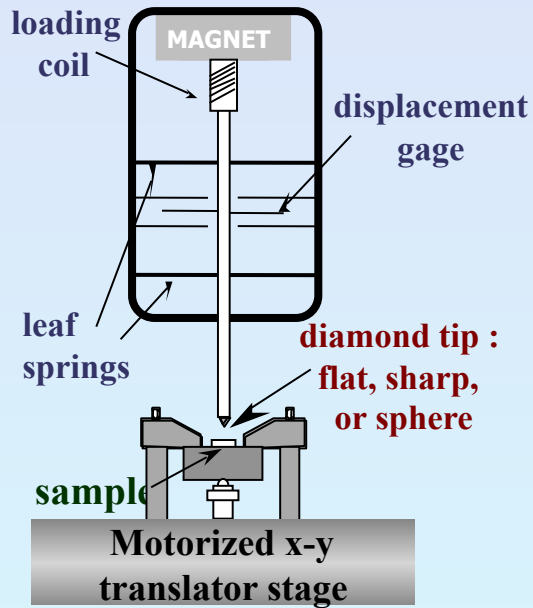
3. Pillars in PMMA Matrix



4. Free-standing pillar array



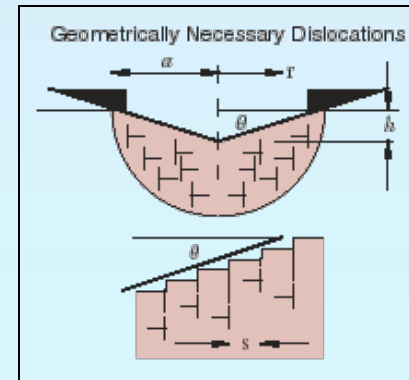
Mechanical Properties via Nanoindentation



Nix, Feng, Greer, et al Thin Solid Films (2007)

$$\left(\frac{H(h)}{H_0} \right)^2 = 1 + h^* \times \frac{1}{h}$$

strain gradients drive hardness increase



$$\rho_{GND} = \frac{3 \tan^2 \theta}{2bh}$$

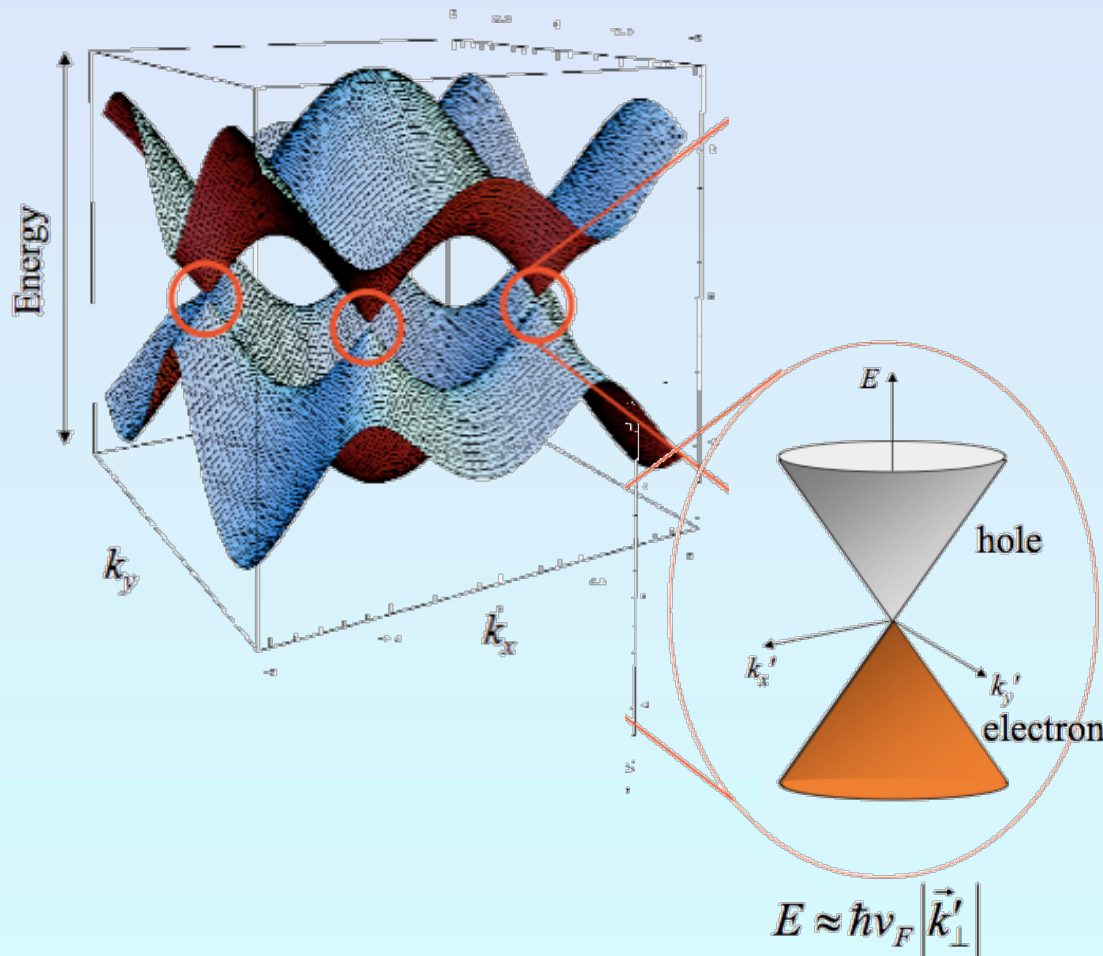
Great need for experimental techniques testing mechanical deformation at **nano-scale** *without* strain gradients!



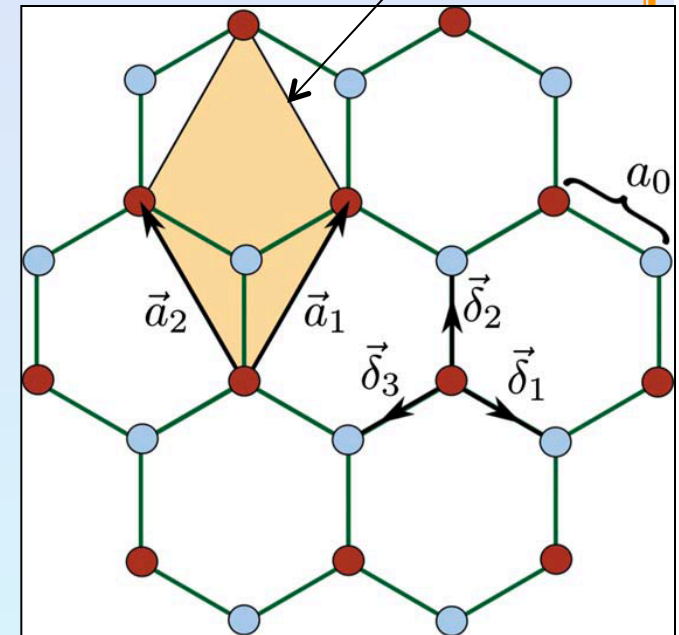
III. Mechanical/Electrical coupling in graphene-based devices

Physical and Electronic Structure

Band structure of graphene (Wallace 1947)



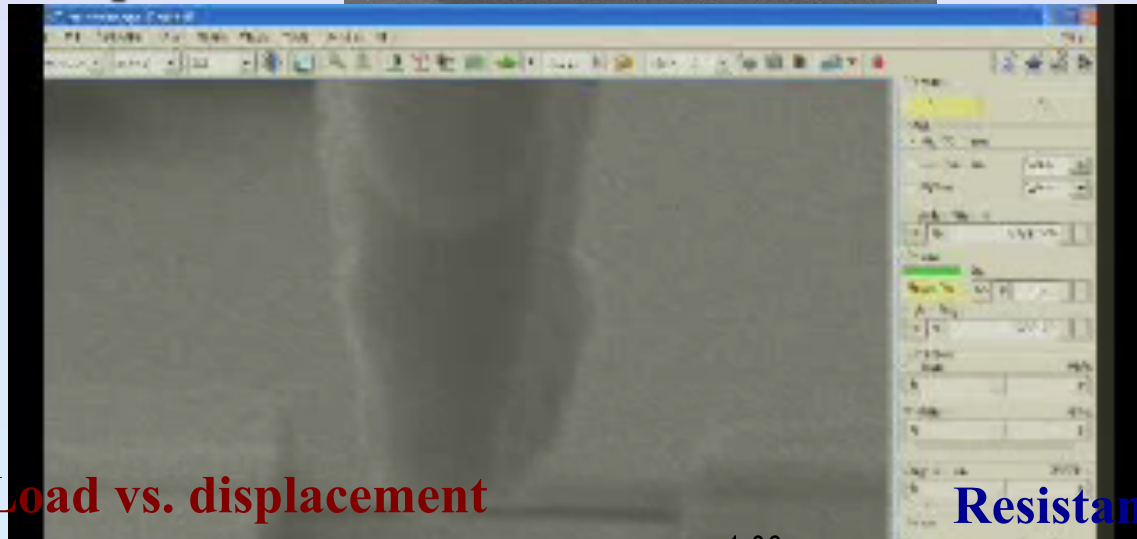
Unit Cell



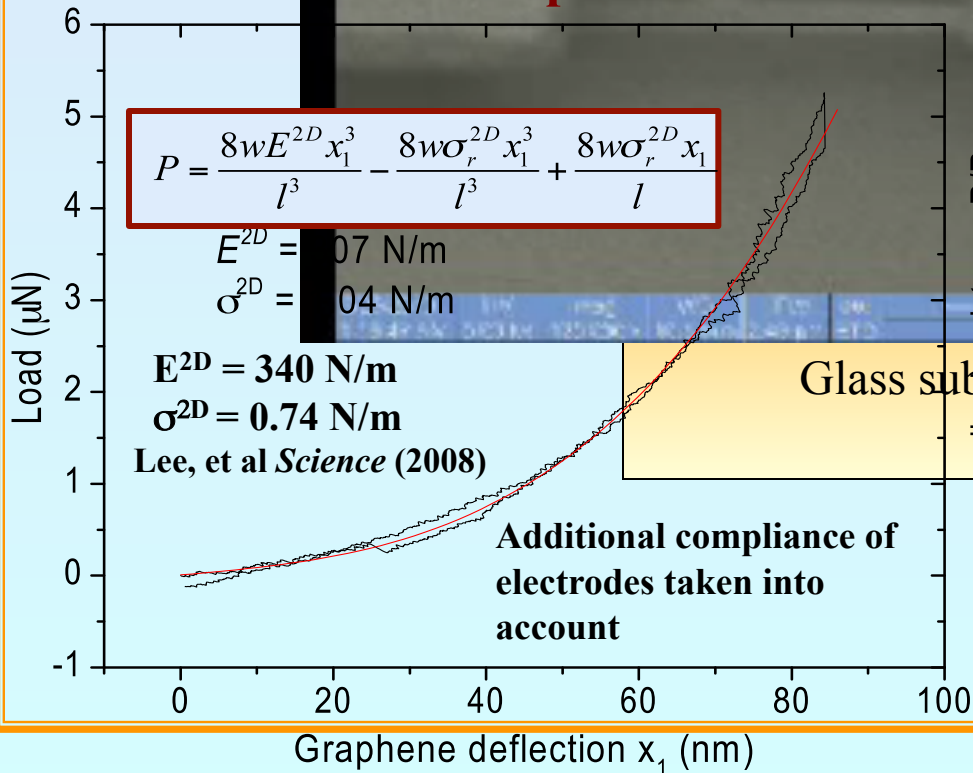
2-D Graphene Lattice

Graphene: Zero band gap “semiconductor”

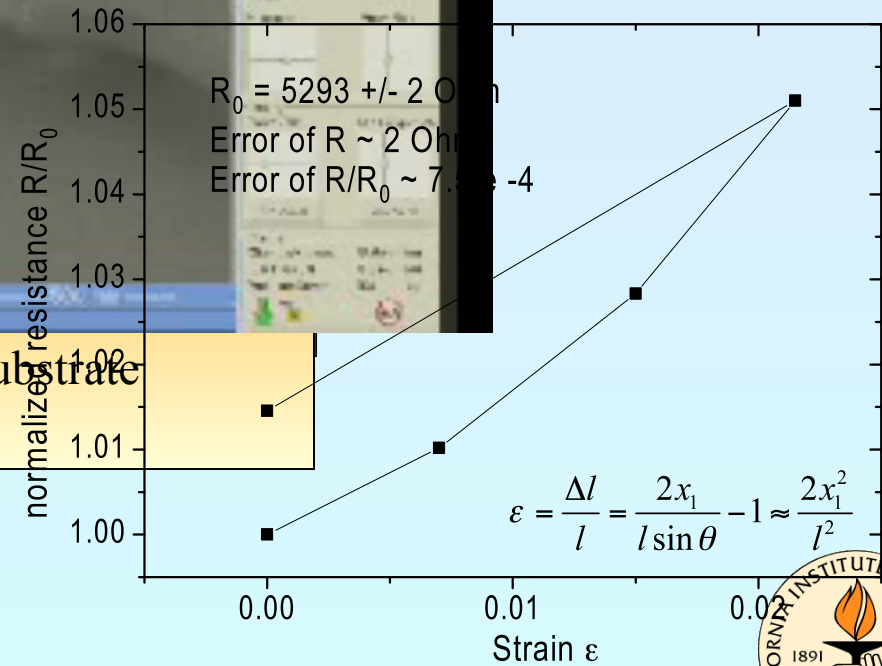
Suspended graphene devices



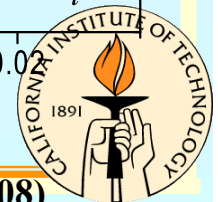
Load vs. displacement



Resistance vs. strain



Herbert, E., (Ph.D. Thesis, 2008)



Mathematical model:

Graphene deflection:

$$P = \frac{8wE^{2D}x_1^3}{l^3} - \frac{8w\sigma_r^{2D}x_1^3}{l^3} + \frac{8w\sigma_r^{2D}x_1}{l} \quad (1)$$

Electrode deflection: $P = 2Kx_2$

Where K is estimated by beam deflection: $K = \frac{4E_g w_g h_g^3}{L_g^3} \approx 1 \times 10^2$

Instrument measures total displ: $x = x_1 + x_2$

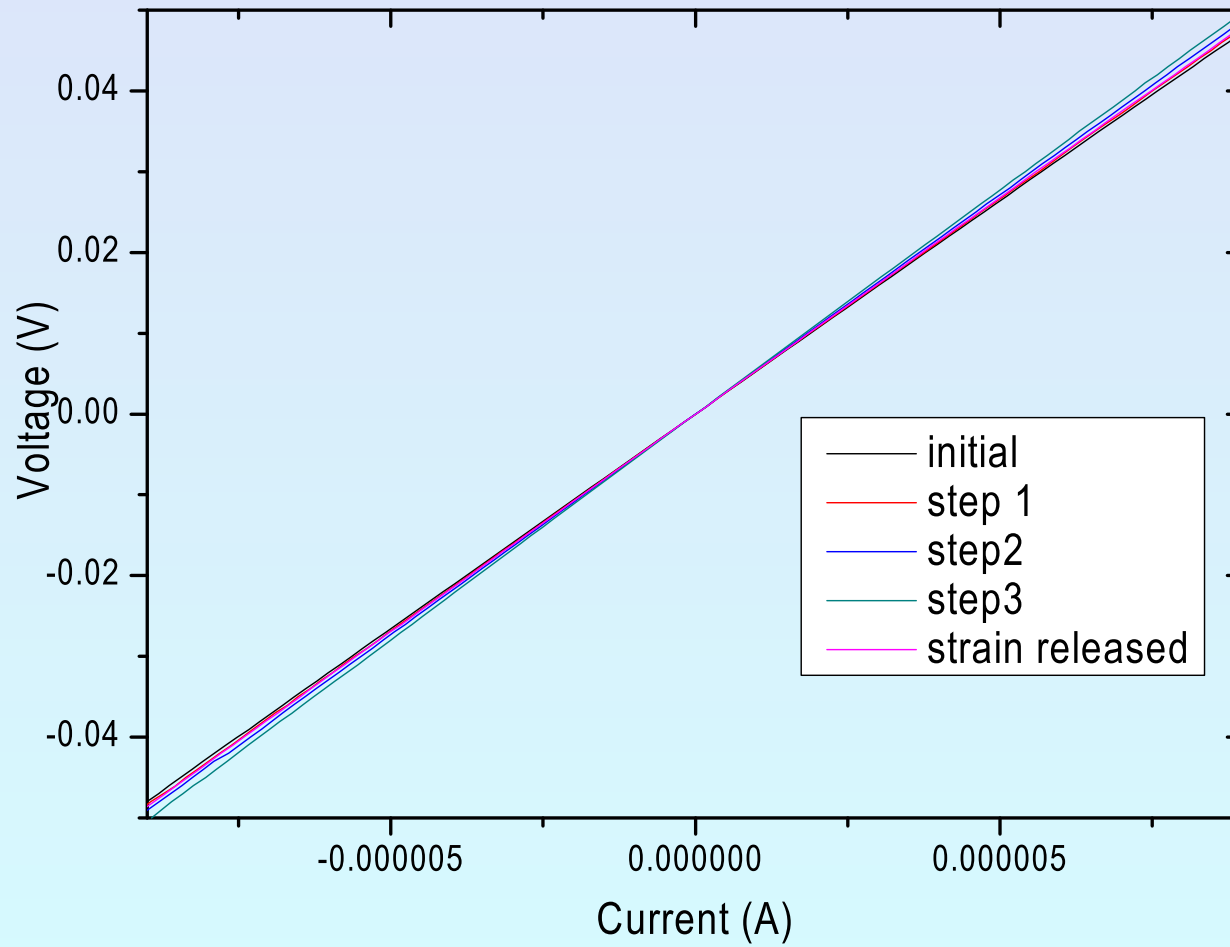
To obtain load vs. displacement, the electrode deflection is subtracted from gross displacement: $x_1 = x - x_2$

Stiffness:

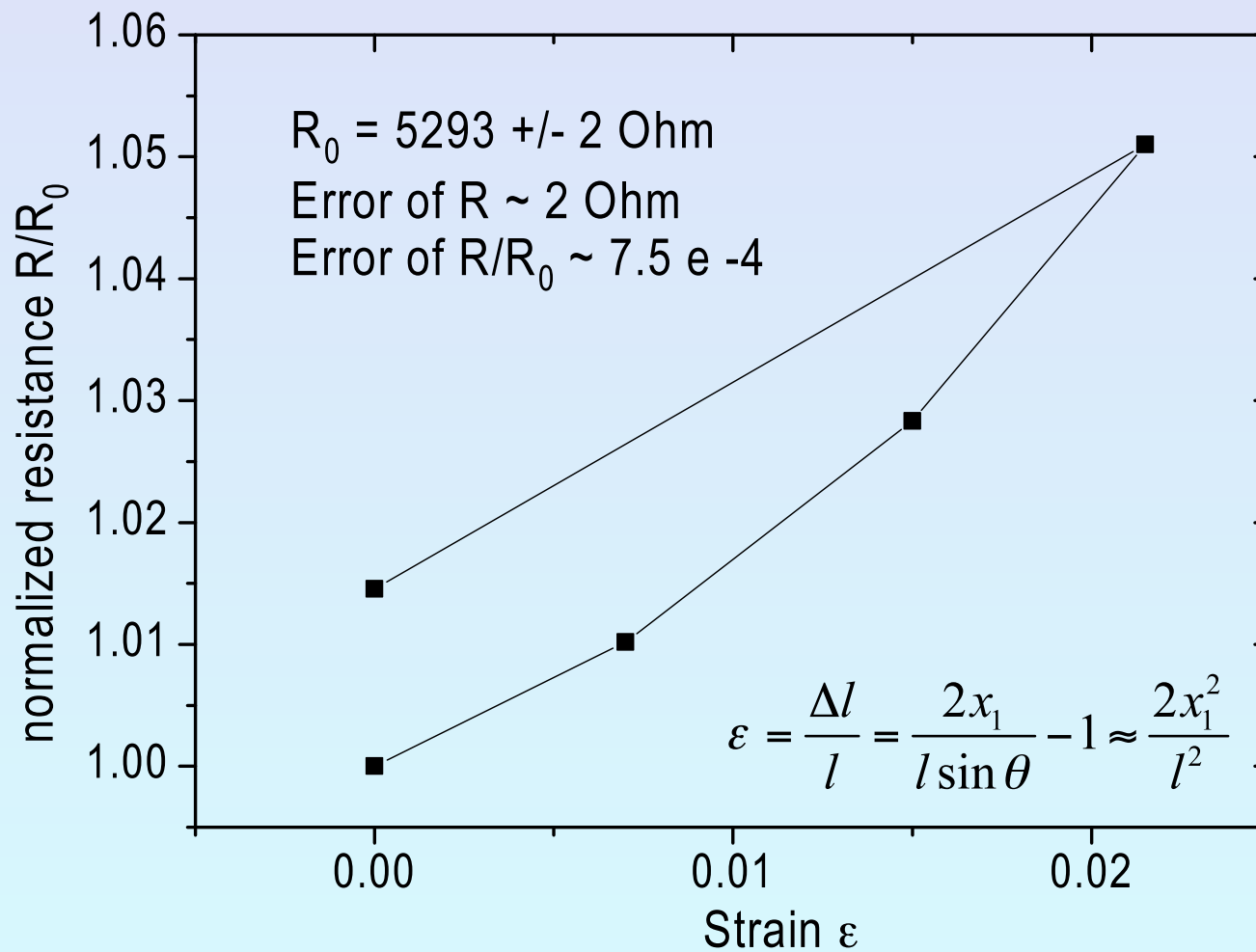
$$\frac{dP}{dx} = \frac{\frac{dP}{dx_1} \frac{dP}{dx_2}}{\frac{dP}{dx_1} + \frac{dP}{dx_2}} = \frac{(Ax_1^2 + B)2K}{2K + Ax_1^2 + B}$$



Electromechanical results: I-V curve



Resistance Change



Strain:

