MEMS devices for *In-Situ* Electron Microscopy Testing of Nanostructures

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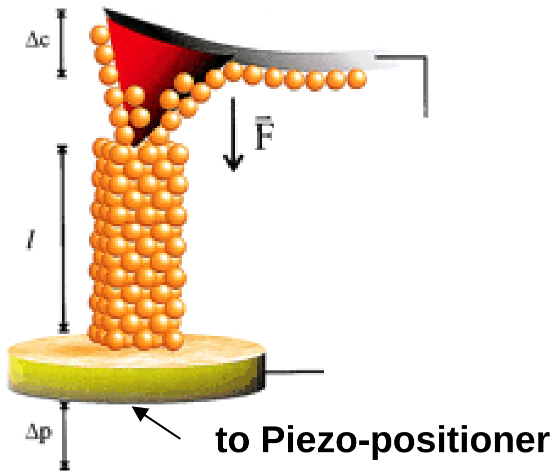
Acknowledgments: NSF-NIRT, NSF-NSEC, FAA, ONR

NEMS Characterization Technique

- Component Constitutive Behavior (CNTs/NWs)
- Electro-Mechanical Characterization at the Device Level

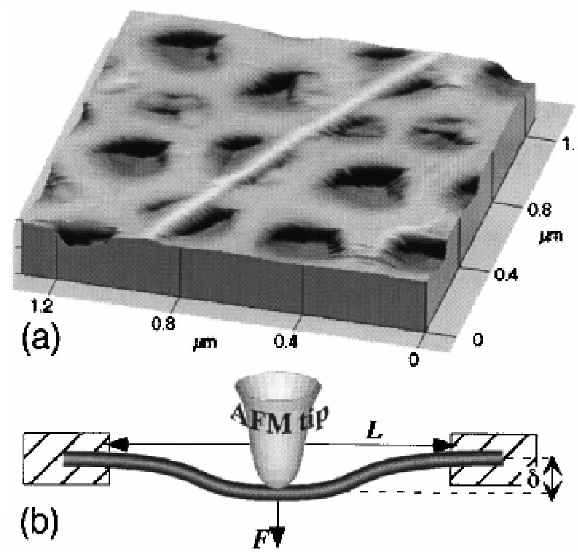
Techniques for Testing Nanostructures

AFM Tension



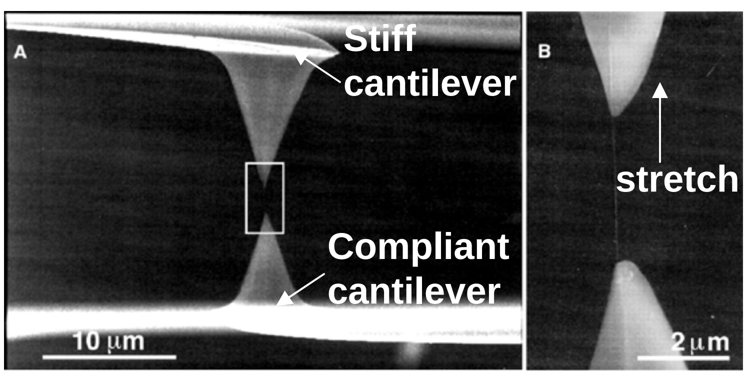
Marszalek et al., *PNAS* (2000)

AFM bending



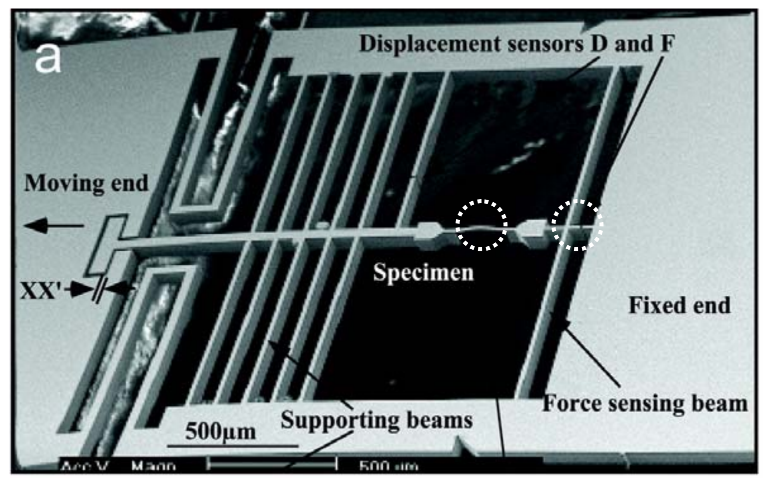
Salvetat et al. *PRL* (1999)

SEM Tension



Yu et al., *Science* (2000)

TEM Tension



Haque and Saif, *Exp. Mechanics* (2002)

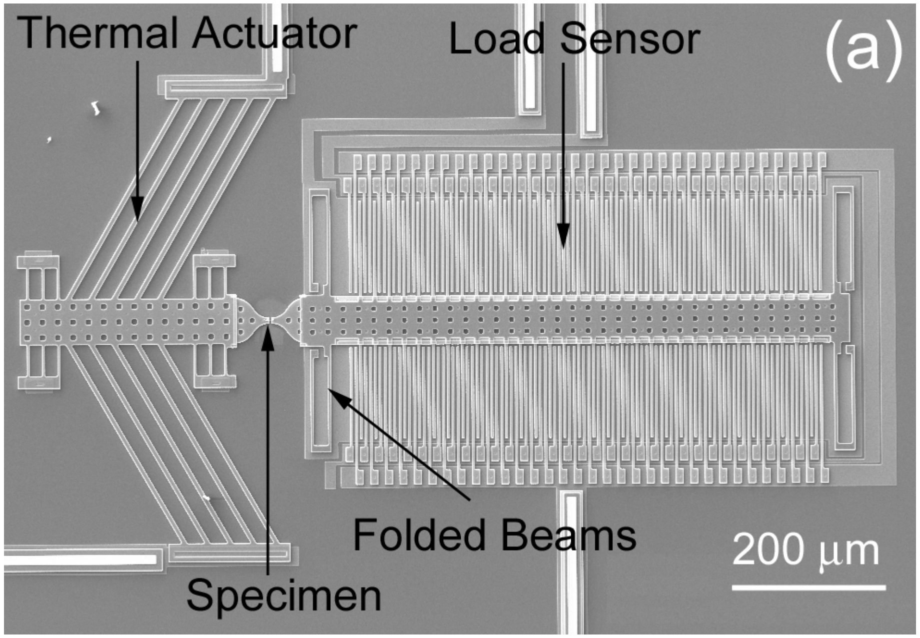
Limitations

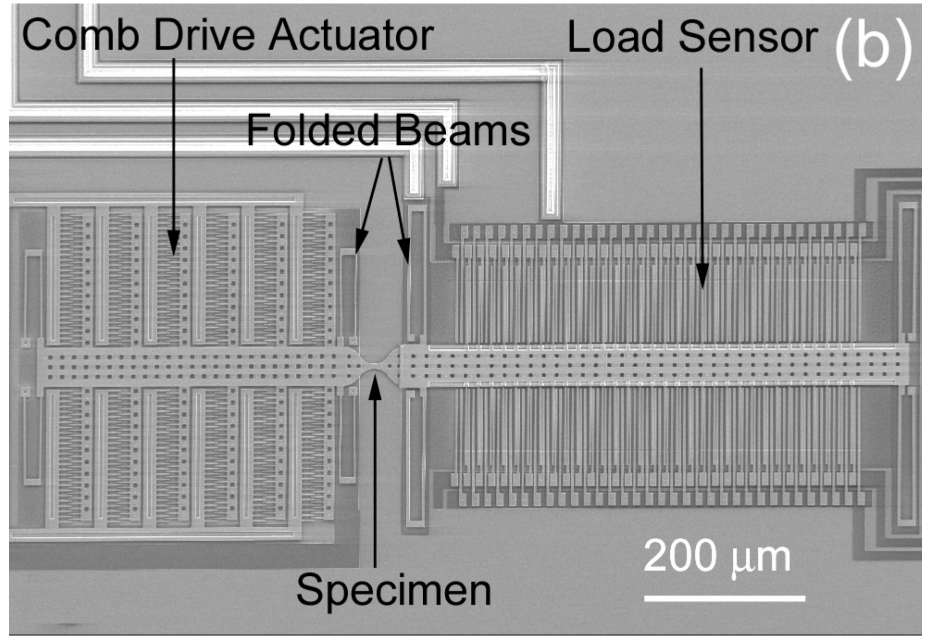
- **Lack *direct* or *simultaneous* real time measurement of load, deformation and imaging of atomic defects.**
- **Imaging is required for both load measurement and atomic defects identification. However, the required magnifications are quite different.**
- **Do not exhibit a single loading condition specially at large deformations.**
- **Are not suitable for *in-situ* TEM (highest atomic resolution).**
- **Cannot measure specimen electronic properties under a well-characterized loading condition.**

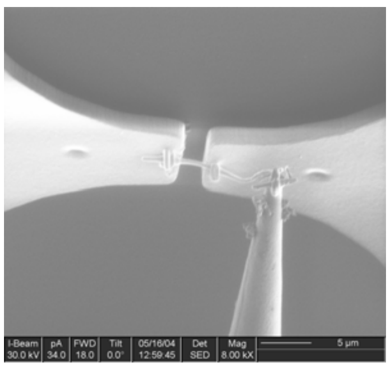
MEMS-based Material Testing System

Displacement Control

Force Control

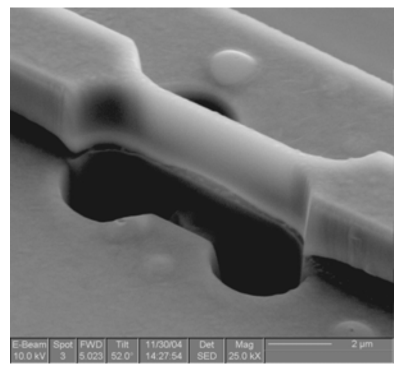






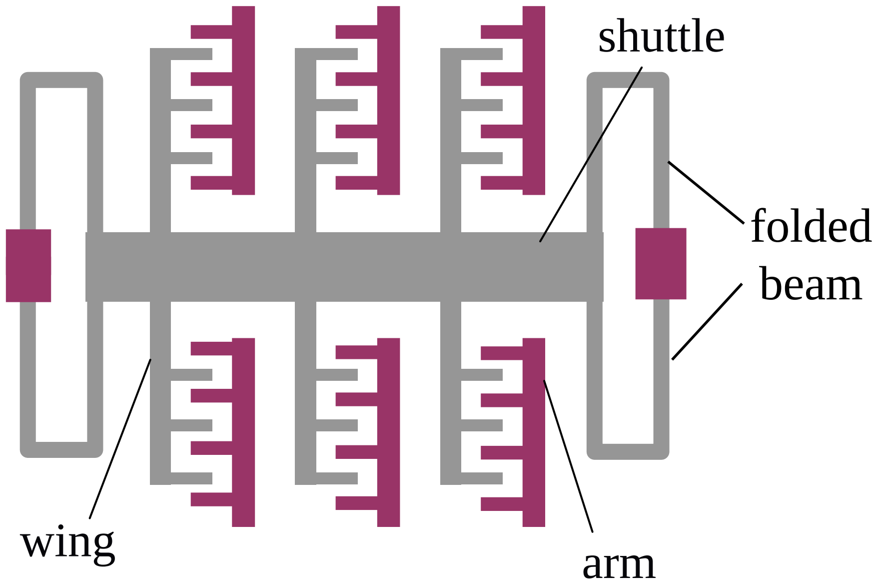
NWs/CNTs

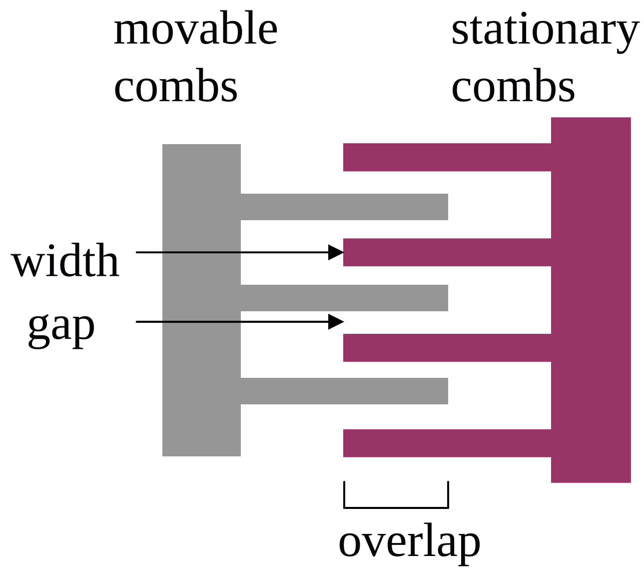
Zhu, Moldovan and Espinosa, *Appl. Phys. Lett.* 86, 013506 (2005)



Thin Films

Electrostatic (Comb Drive) Actuator – Force Control



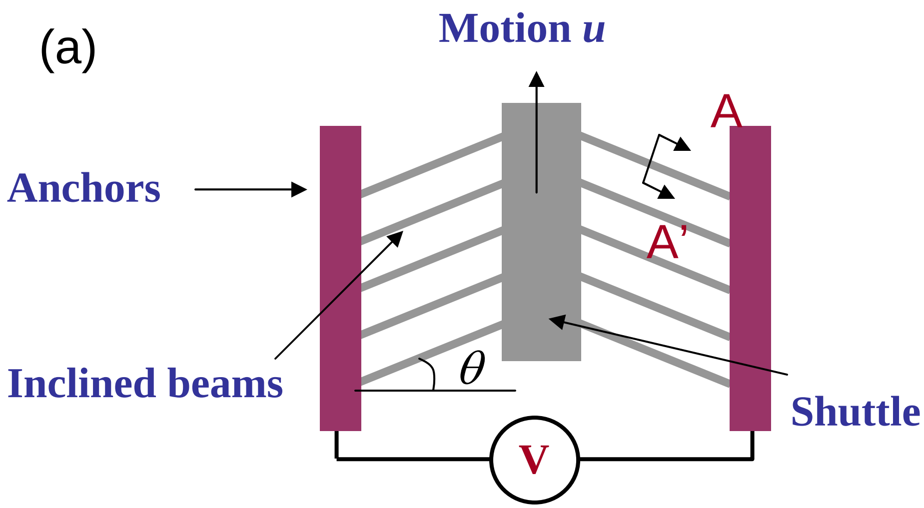


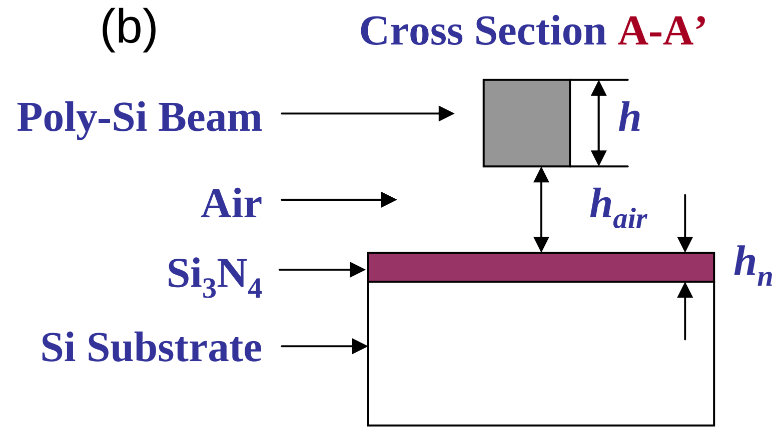
$$F = N\varepsilon_0 V^2 h/d$$

- **Pairs of combs:** **300/1200**
- **Folded beam length:** **125 μm**
- **Folded beam width:** **2.5 μm**
- **Folded beam height:** **3.5 μm**

- **Height (*h*):** **2 μm**
- **Width (*w*):** **2 μm**
- **Gap (*d*):** **2 μm**
- **Overlap (*o*):** **15 μm**
- **Length (*l*):** **30 μm**

Thermal Actuator – Displacement Control





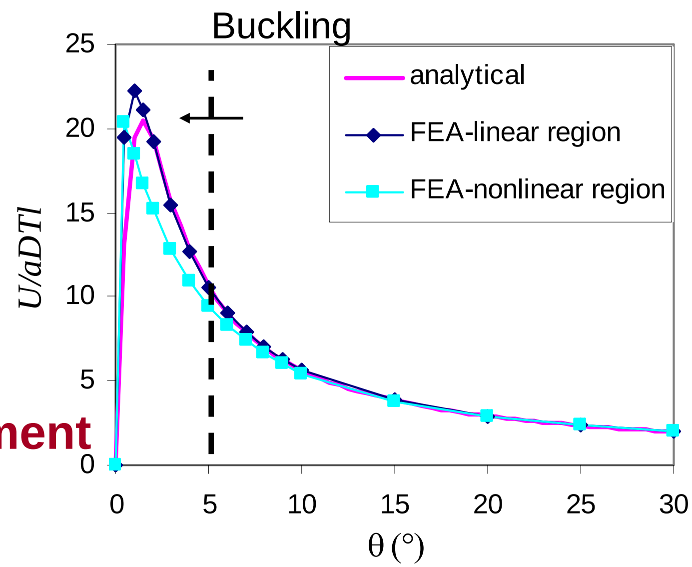
$$U = \frac{\alpha\,\Delta T\, l \sin\theta - \cancel{F_S \frac{l}{2NEA}}}{\left(\psi \sin^2\theta + \cos^2\theta\right)} \;;\; \psi = \frac{Al^2}{12I}$$

ψ : axial over bending stiffness ratio

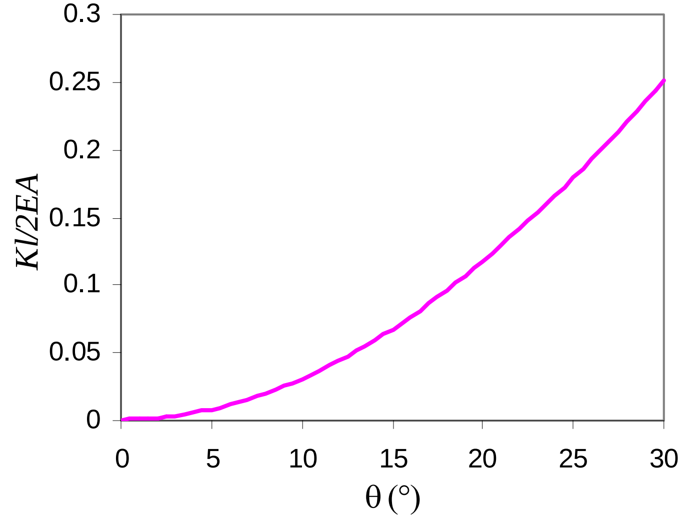
• **Pair of inclined beams:**	**5/10**
• **Beam length:**	**300 μm**
• **Beam width:**	**8 μm**
• **Beam height:**	**3.5 μm**

Prorok, Zhu, Espinosa, Bazant & Yakobson, in *Encyclopedia of Nanoscience and Nanotechnology* (2004), p. 555; Zhu, Corigliano & Espinosa, in press *J. Micromech. Microeng.,* (2006)

Beam Angle Selection - 1



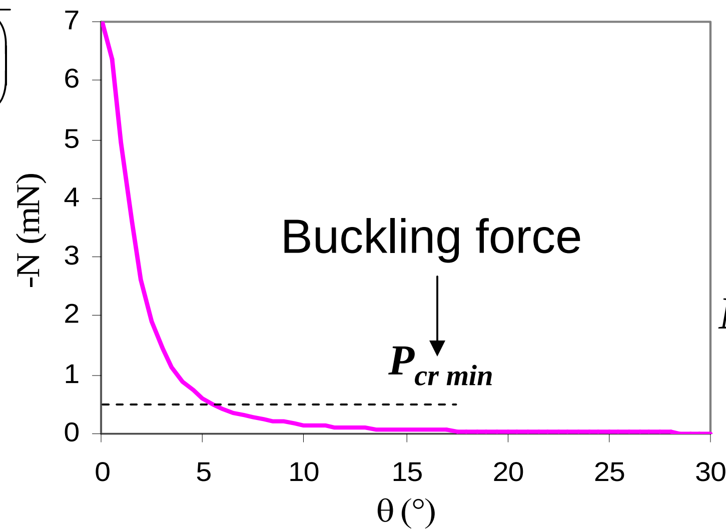
Displacement



Stiffness

$$\frac{U}{\alpha\,\Delta T\,l} = \frac{\sin\theta}{\left(\sin^2\theta + \cos^2\theta\,\frac{12I}{Al^2}\right)}$$

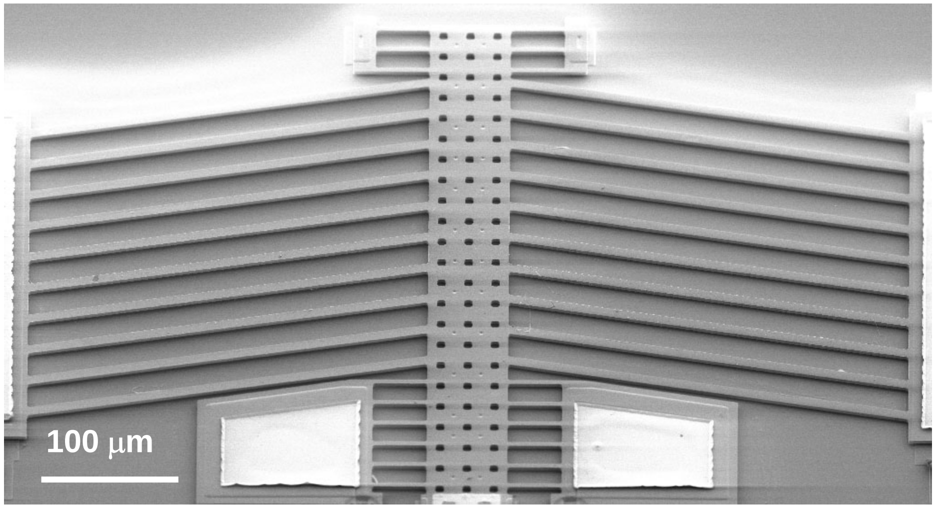
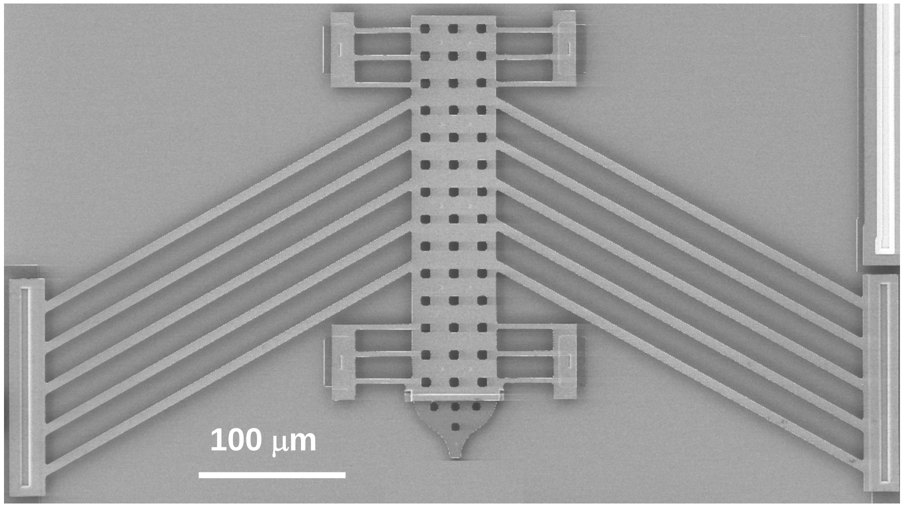
$$\frac{K_{tb}l}{2EA} = \sin^2\theta + \cos^2\theta\,\frac{12I}{Al^2}$$



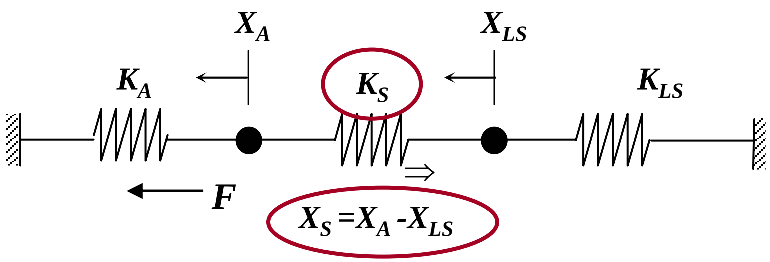
Beam internal force

$$N = \alpha\,\Delta T\,EA\,\frac{\cos^2\theta}{\left(\sin^2\theta\,\frac{Al^2}{12I} + \cos^2\theta\right)}$$

Beam Angle Selection - 2

10^o beam angle	30^o beam angle
	
10 beam pairs	5 beam pairs

Lumped System Model



$$X_{LS} + X_S = X_A$$

$$K_{LS} X_{LS} = K_S X_S \quad \Rightarrow$$

$$K_S X_S + K_A X_A = F = 2N\alpha\Delta TEA\sin\theta$$

To test a particular structure, K_S and X_S are given

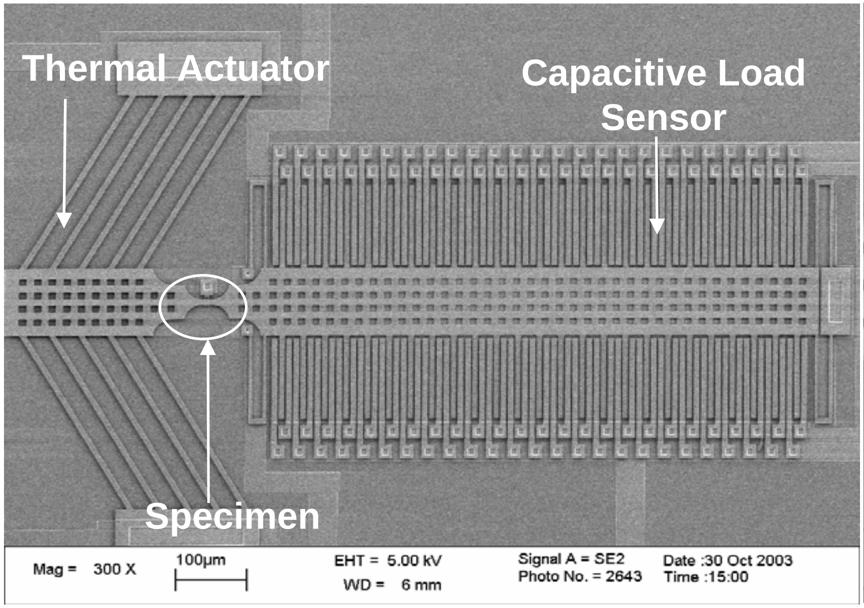
Small K_{lS} $\Rightarrow$ Large X_{LS}

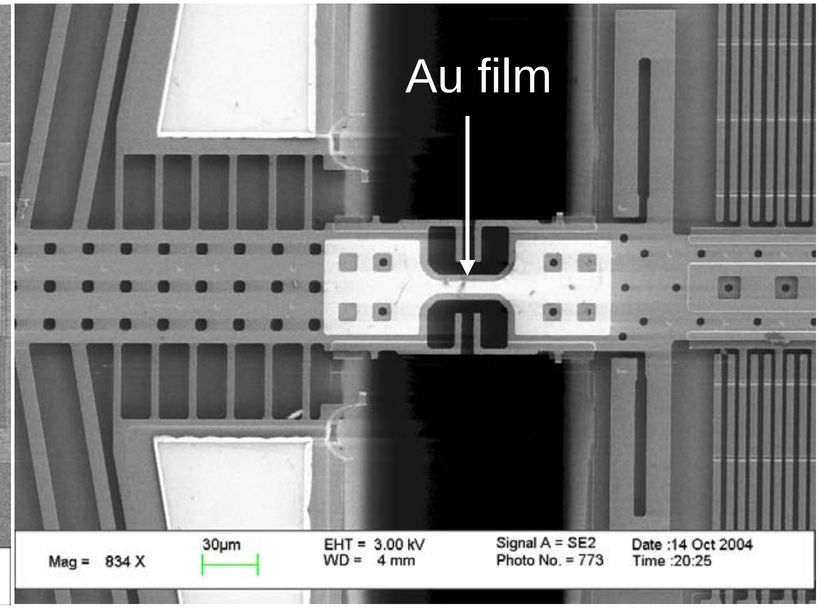
$\Rightarrow$ **high load sensor resolution**

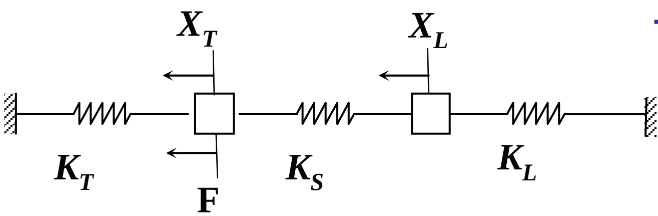
$\Rightarrow$ **large X_A** $\Rightarrow$ **high temperature increase**

$\boxed{K_{LS} \text{ is an important design parameter}}$

Lumped Model – CNT Example







Governing Equations:

$X_S = X_T - X_L$

$K_S X_S = K_L X_L$

$K_T X_T = F - K_S X_S$

$F = NEA\alpha\Delta T\cos\theta$

Thermal actuator stiffness:

$$K_A = 10(\sin^2\theta\frac{Ebh}{l} + \cos^2\theta\frac{Eb^3h}{l^3}) + \frac{12Eb_{sb}^3h}{l_{sb}^3} = 46892\ \text{N/m}$$

MWCNT specimen stiffness:

$$K_S = \frac{E_S A}{l_S} = \frac{E_S \pi D_0 t}{l_S} = 10.7\ \text{N/m}$$

Capacitive load sensor stiffness:

$$K_L = \frac{24EI}{l_L^3} = \frac{2Eb_L^3h_L}{l_L^3} = 18.6\ \text{N/m}$$

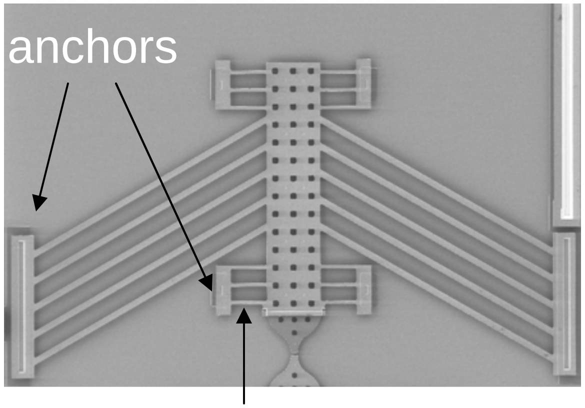
- Pair of inclined beams: 5/10
- Beam length: 300 μm
- Beam width: 8 μm
- Beam height: 3.5 μm

For a MWCNT specimen (l_s = 2 μm; F=1 μN, ε = 4%)

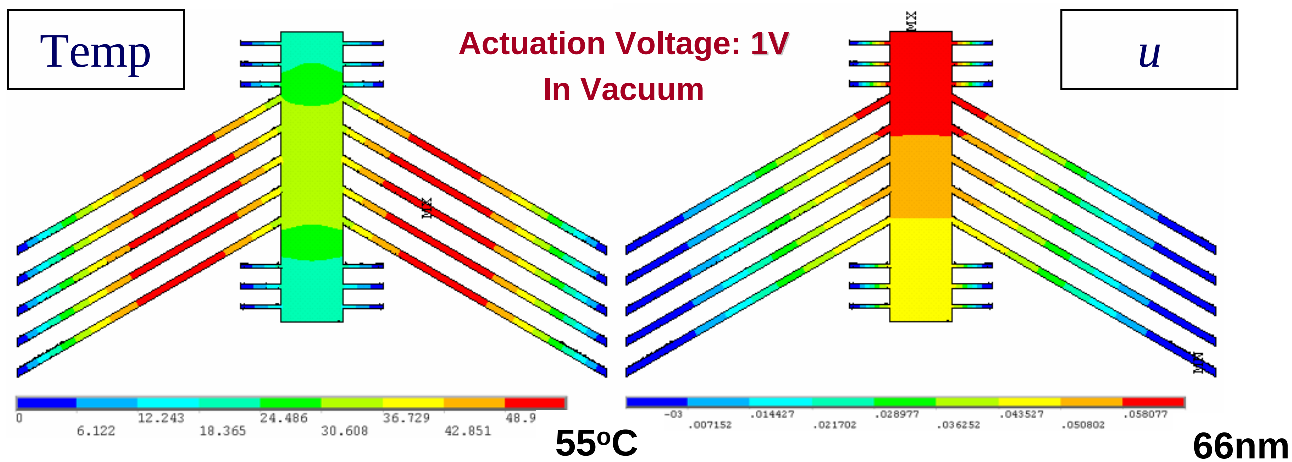
X_T=157.3 nm, X_S=93.5nm and X_L=53.8nm

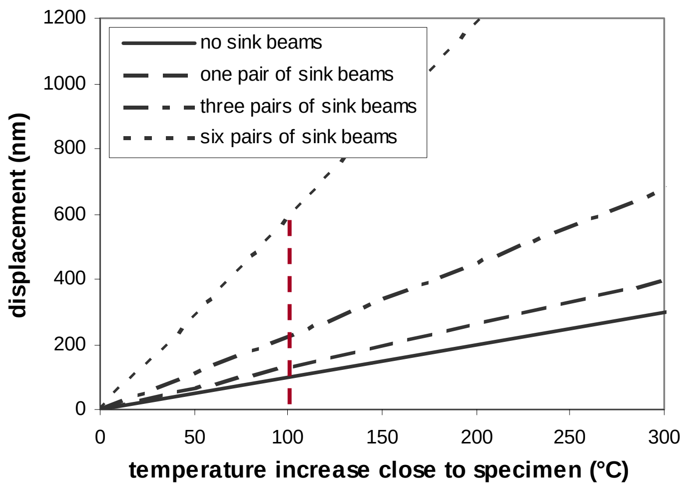
Zhu and Espinosa, *PNAS*, Vol. 102, 2005

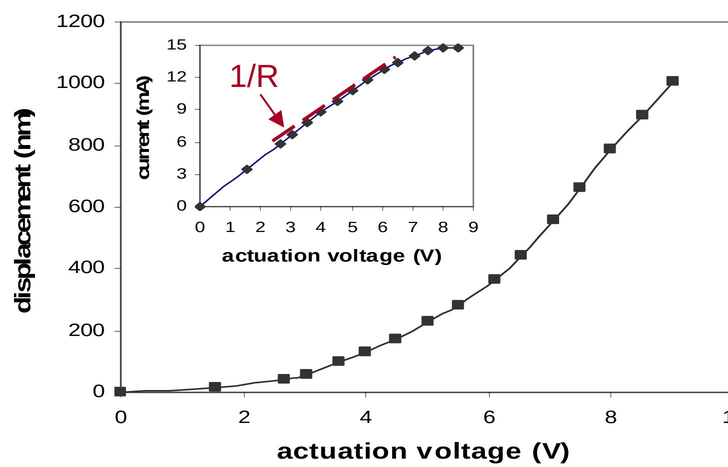
Thermal Actuator – Multiphysics Simulation



Heat sink beams

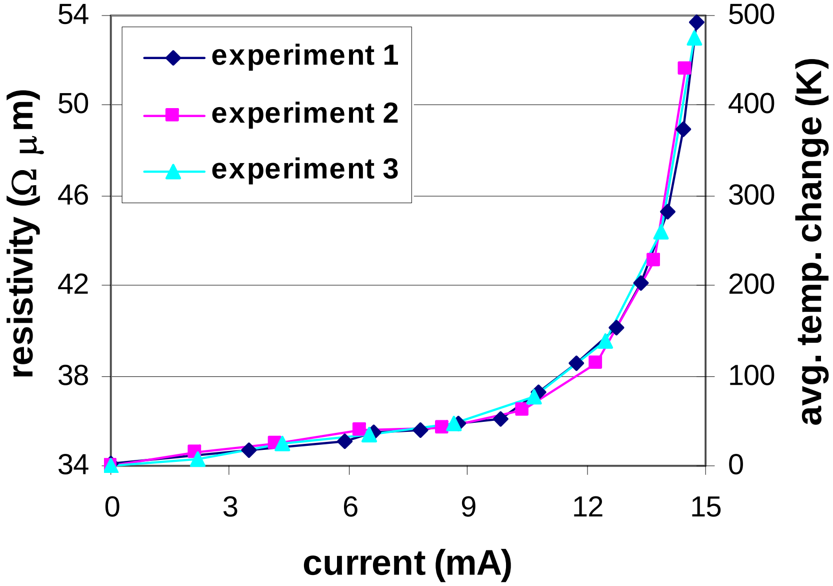


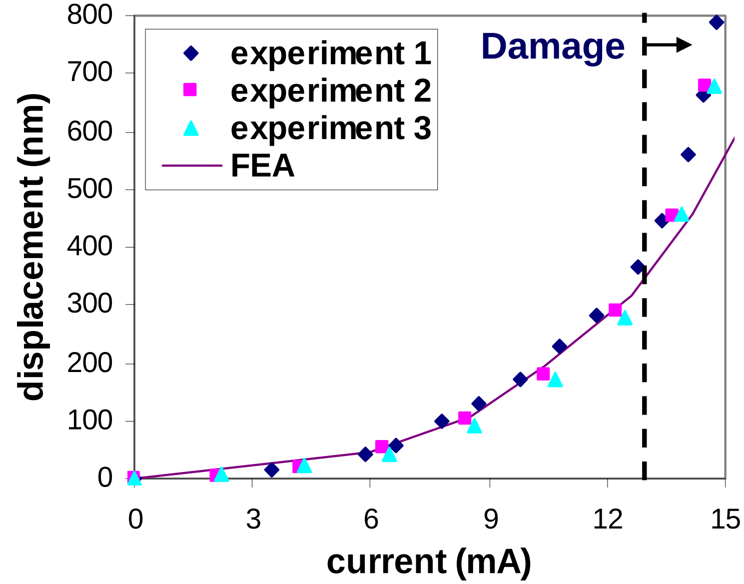


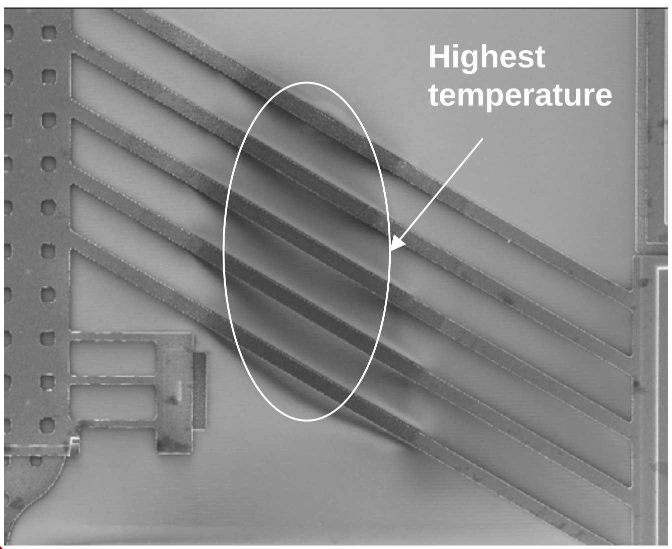


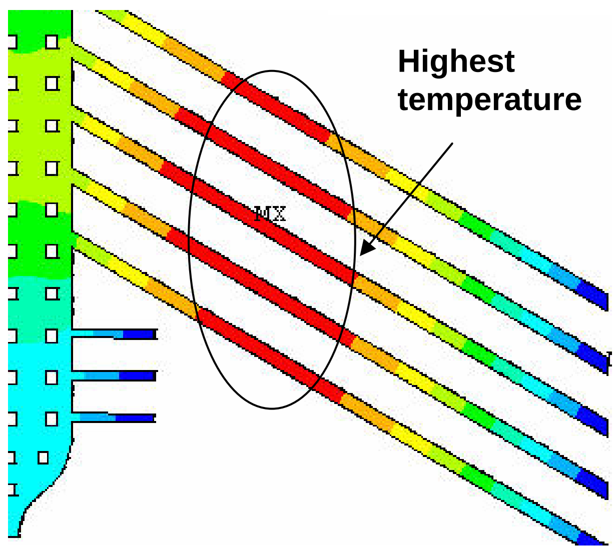
Zhu, Corigliano & Espinosa, in press *J. Micromech. Microeng.*, (2006)

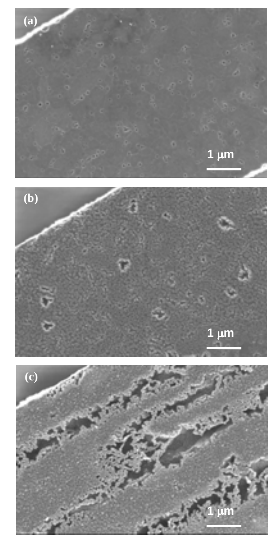
Comparison between Experiments and Simulations



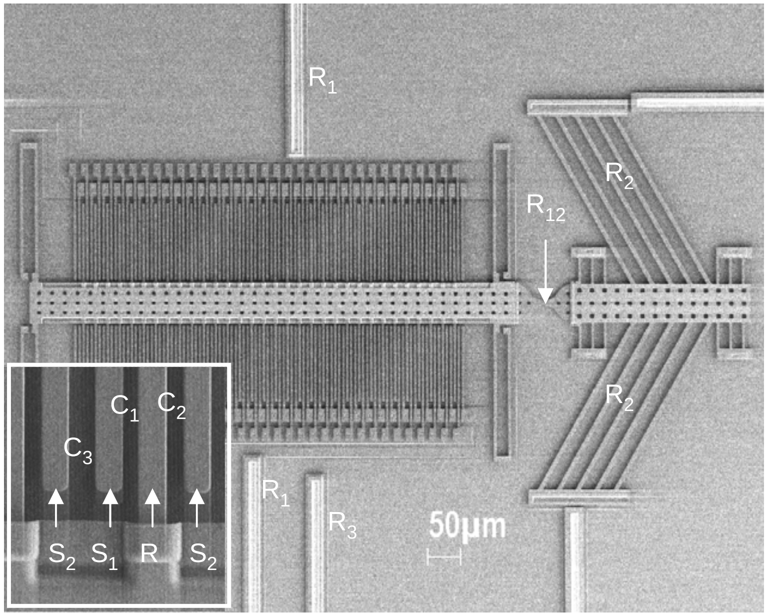


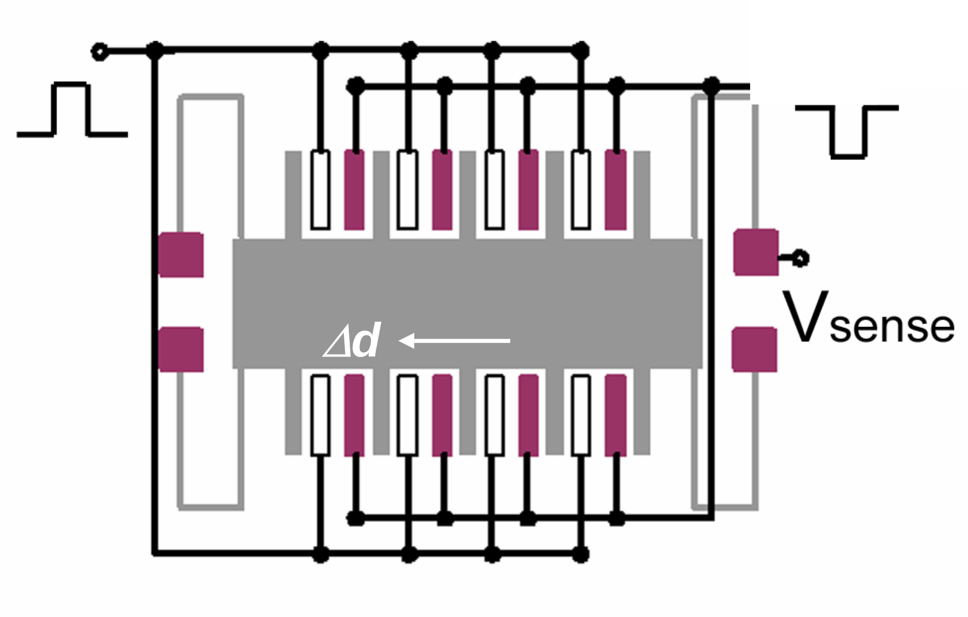






Load Sensor

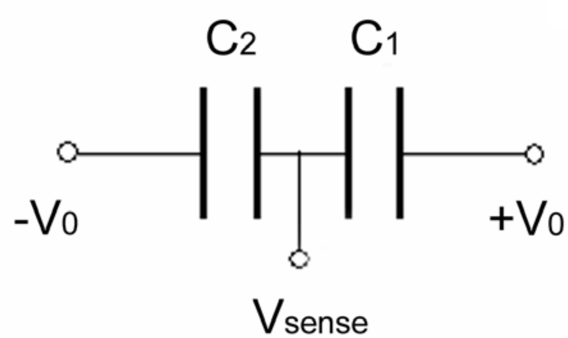




$$\Delta C = C_2 - C_1 = N\varepsilon A\left(\frac{1}{d_0 - \Delta d} - \frac{1}{d_0 + \Delta d}\right)$$

$$\Delta C = \frac{2N\varepsilon A}{d_0^2}\Delta d + o(\Delta d^3)$$

$$\Delta V_{sense} = \frac{V_o}{C_0}\Delta C \sim \frac{2N\varepsilon A V_o}{C_0 d_0^2}\Delta d$$



Zhu, Moldovan and Espinosa, *Appl. Phys. Lett.* 86, 013506 (2005)

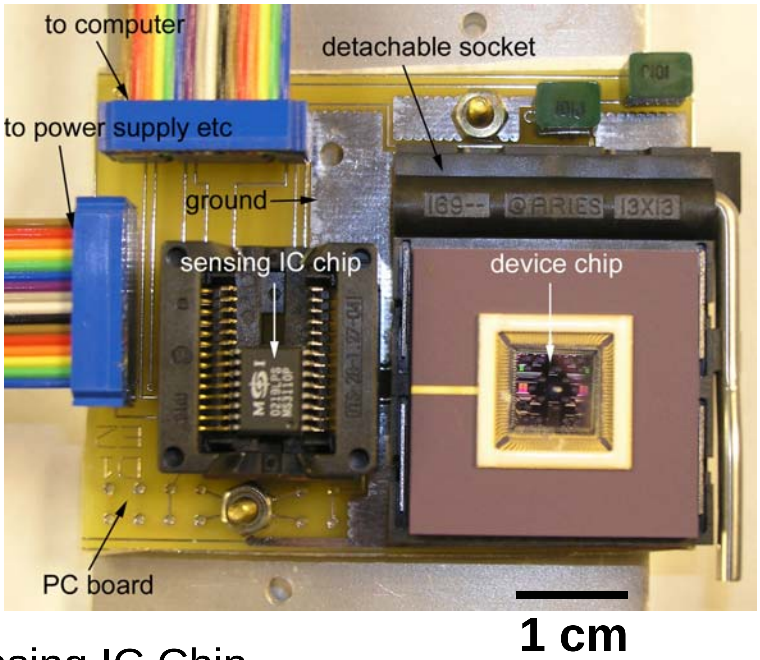
Load Sensor and Signal Conditioning

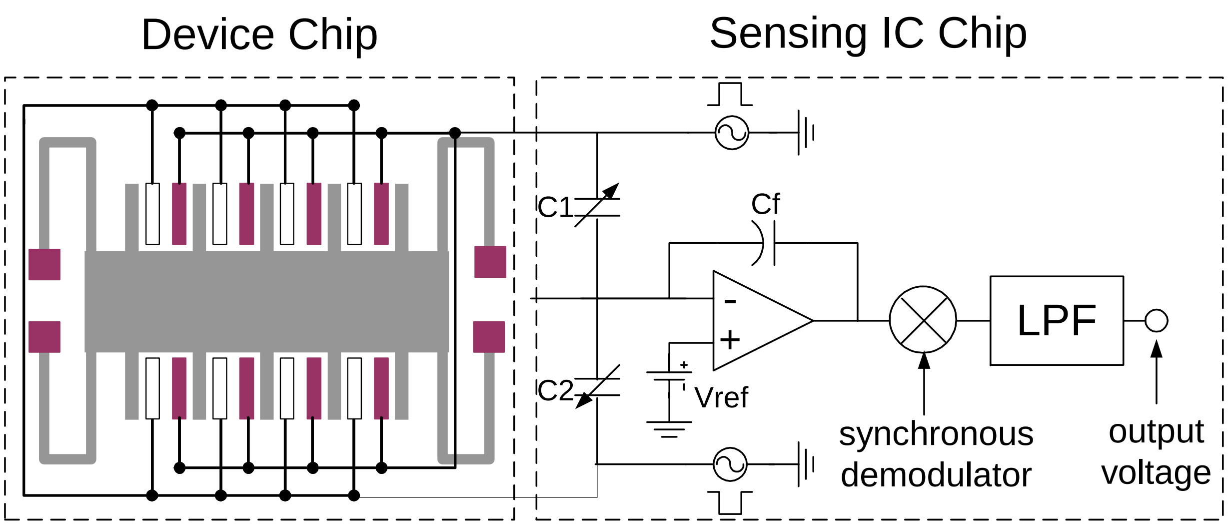
Charge sensing method:

- $\Delta V_{sense} \propto \Delta d$
- Eliminates parasitic capacitances

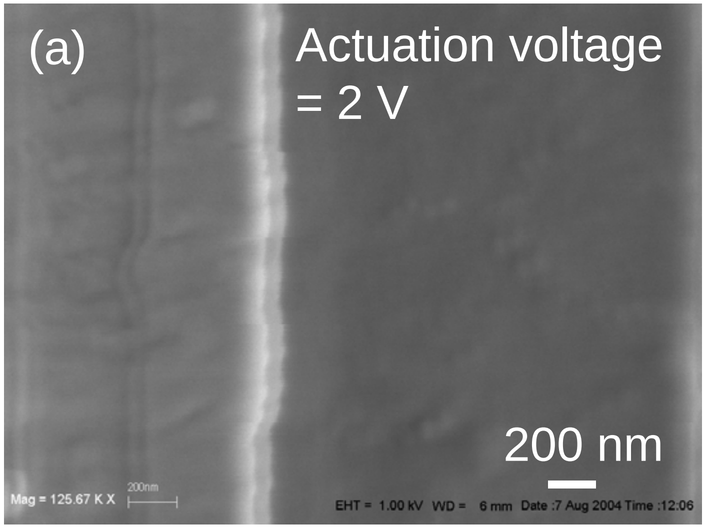
Double chip Architecture

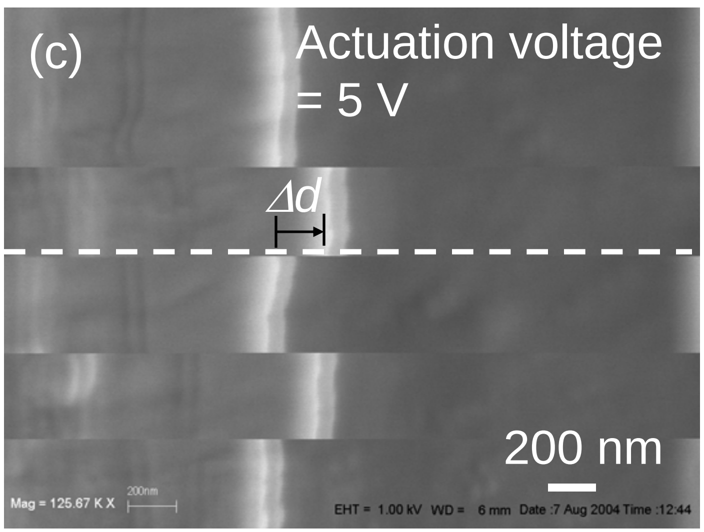
- Both chips on a custom-made PCB;
- Minimizes stray capacitance and electro-magnetic interference.

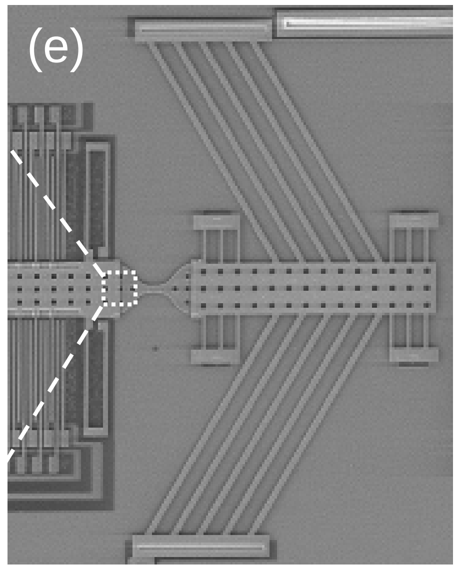


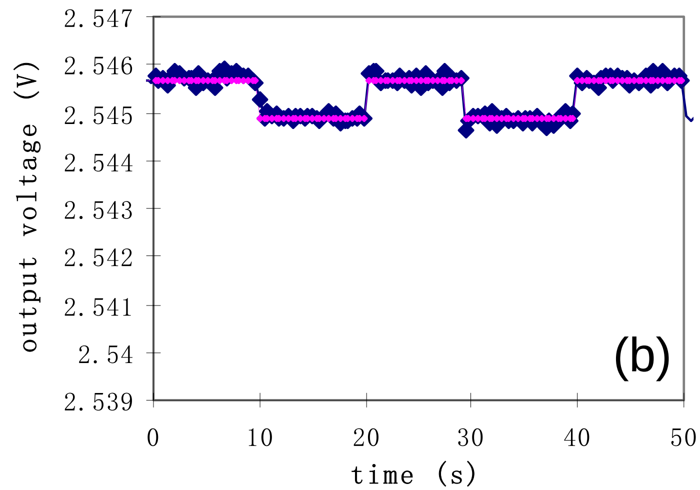


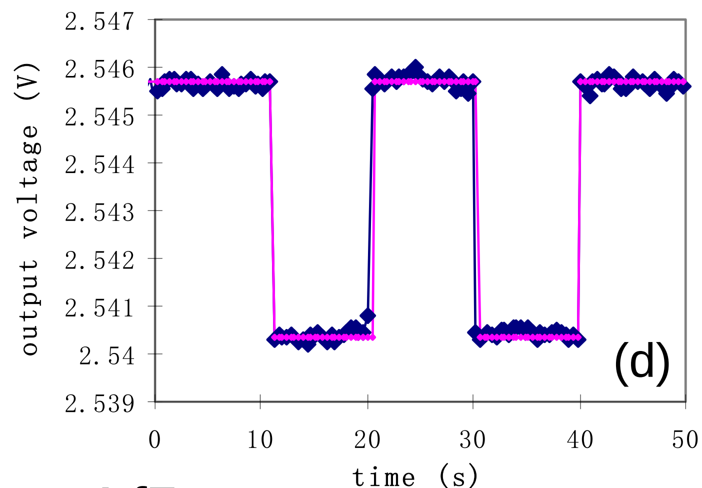
Load Sensor Calibration - 1







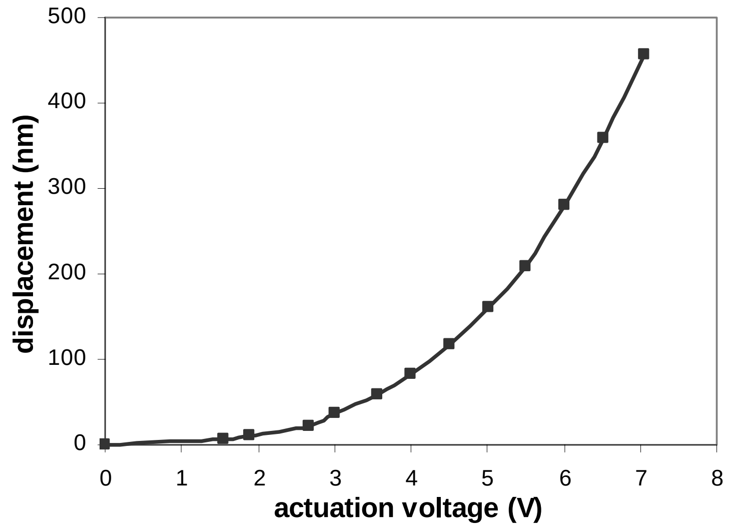


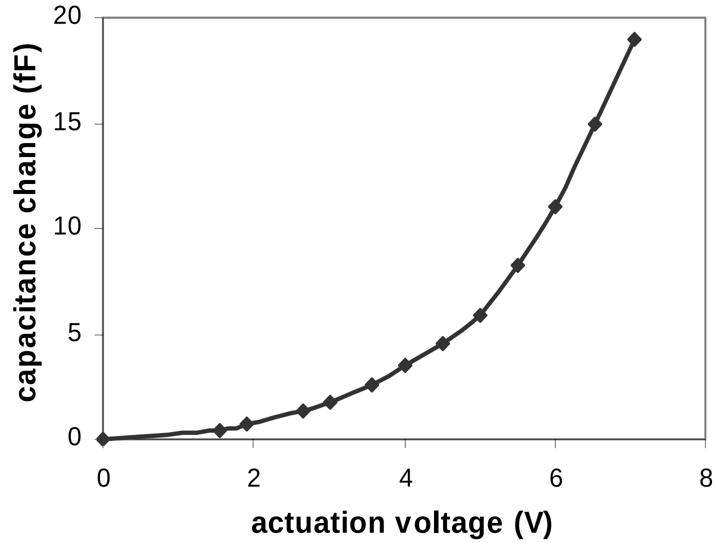


$$\Delta V_{sense} = \frac{V_0}{C_f} \Delta C$$

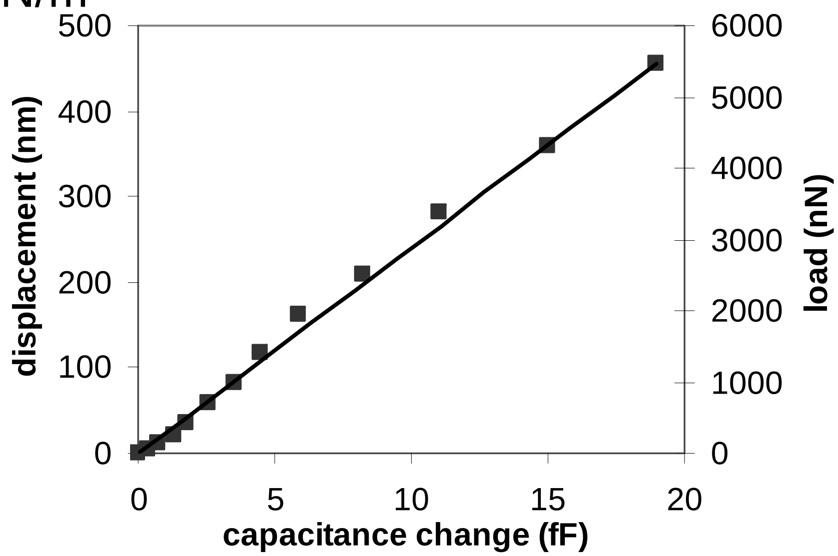
1 mV corresponds to 1 fF

Load Sensor Calibration - 2





Stiffness = 11.8 N/m



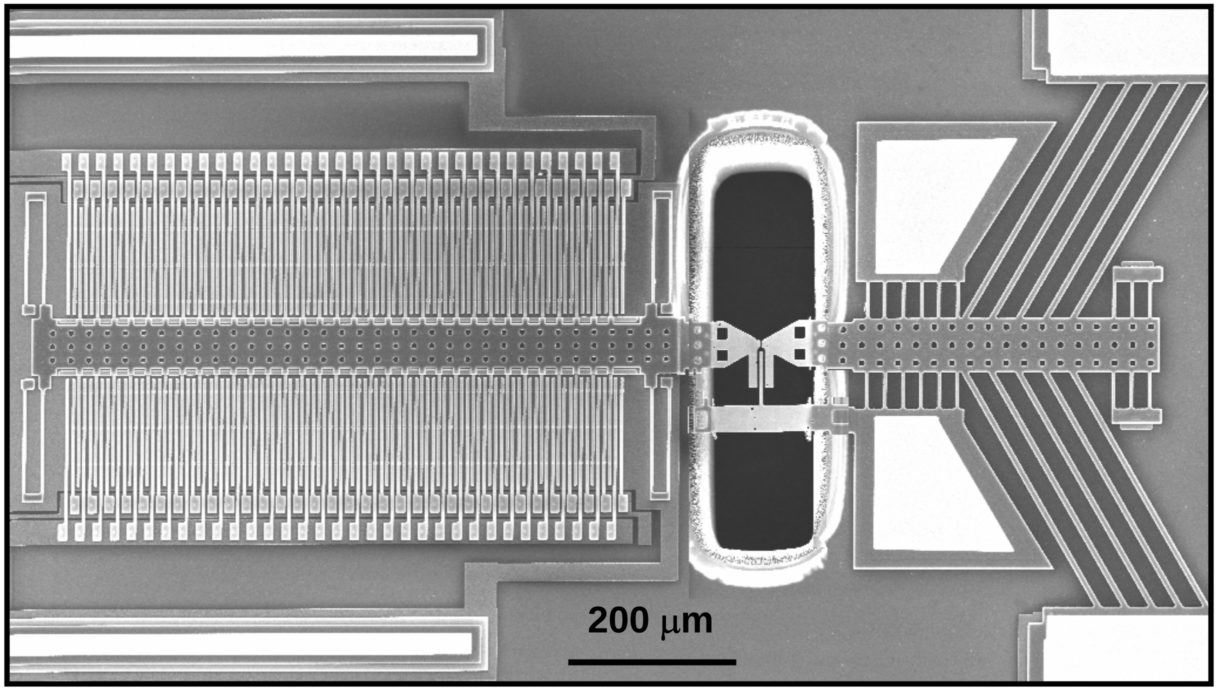
Load = displacement × stiffness

Device Resolution

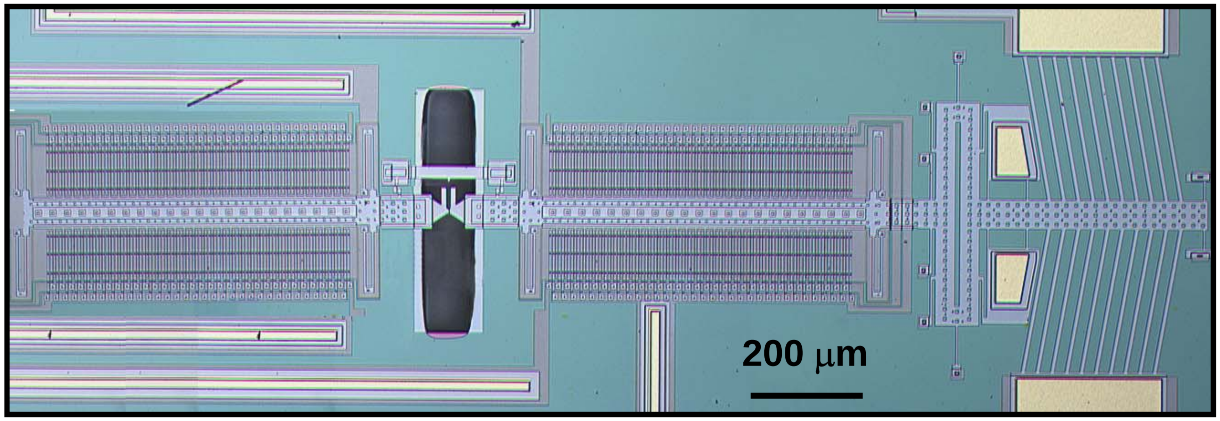
Capacitance change: 0.05 fF
Displacement: 1 nm
Load: 12 nN

Other Configurations

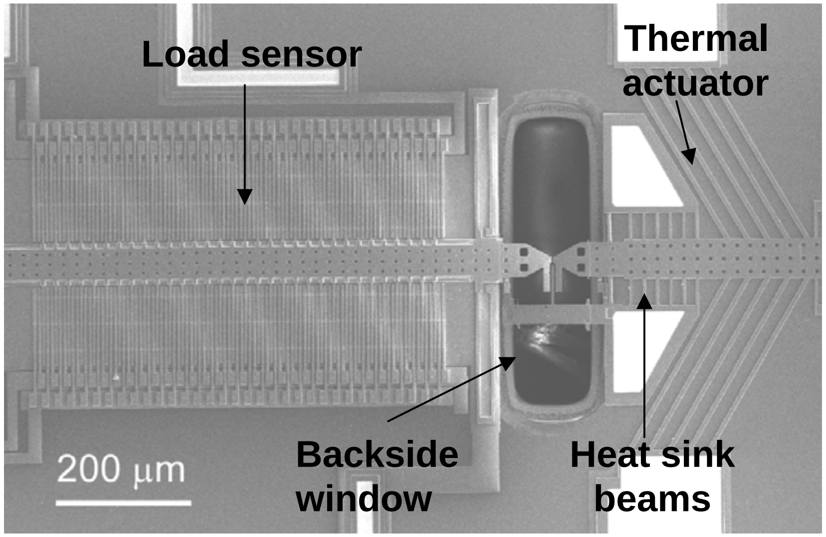
Compression/ Buckling

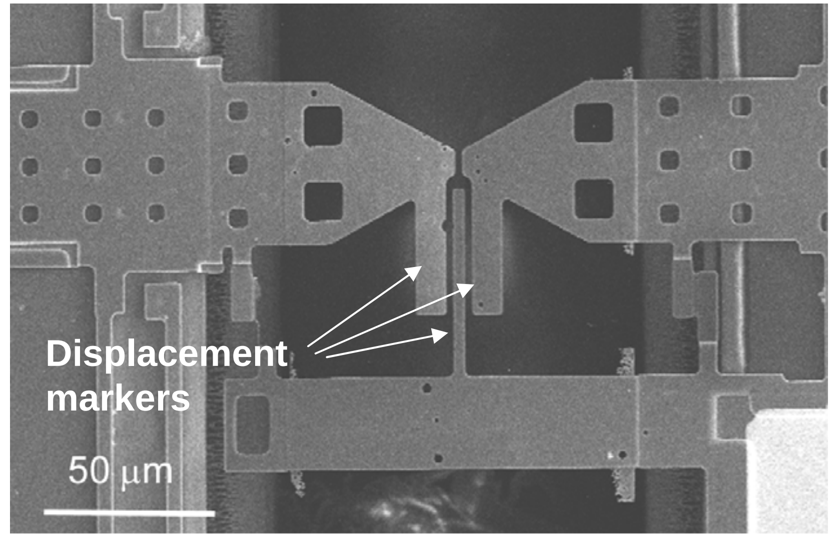


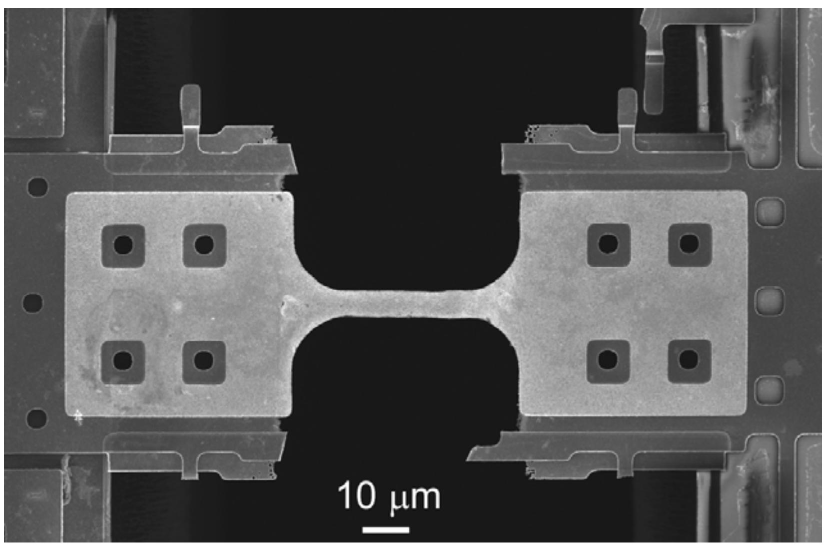
Electronic measurement of load and elongation

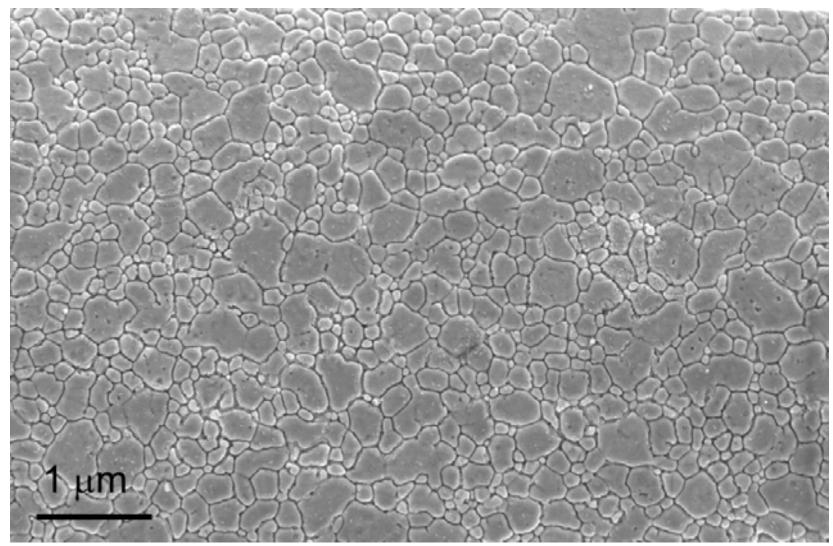


In-situ TEM Devices: Nanostructure & Thin Film

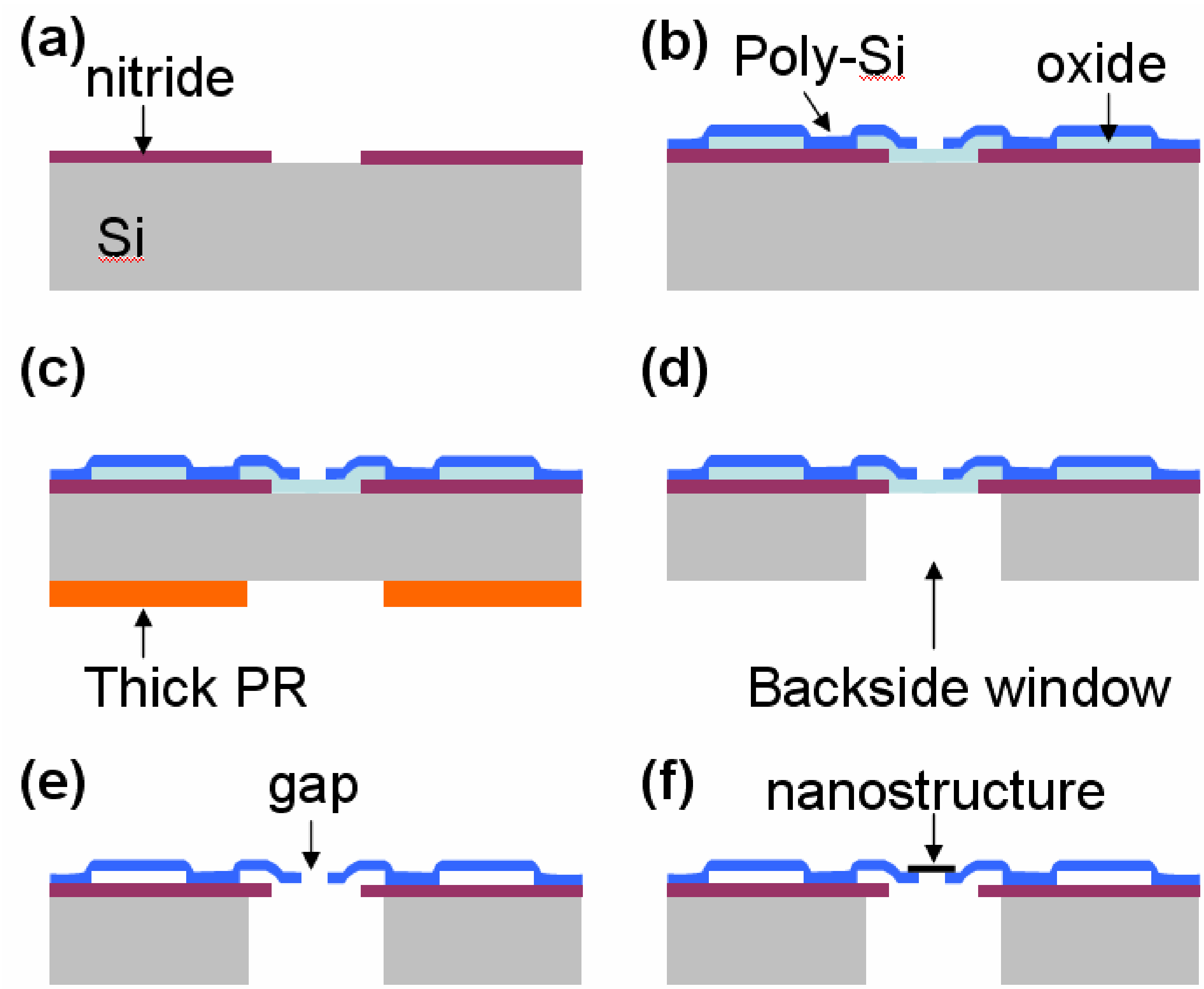






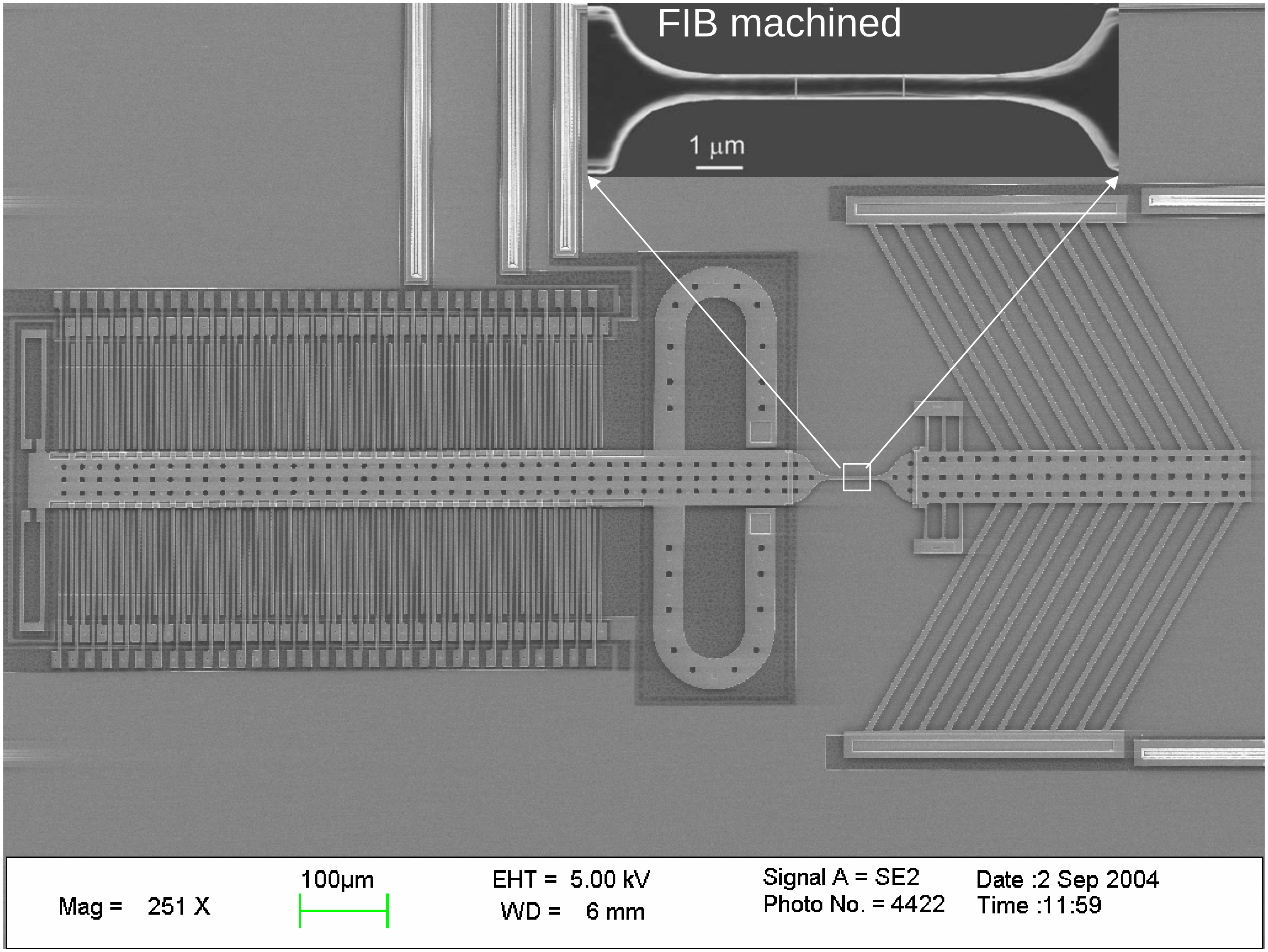


Fabrication Process of *In-situ* TEM Device

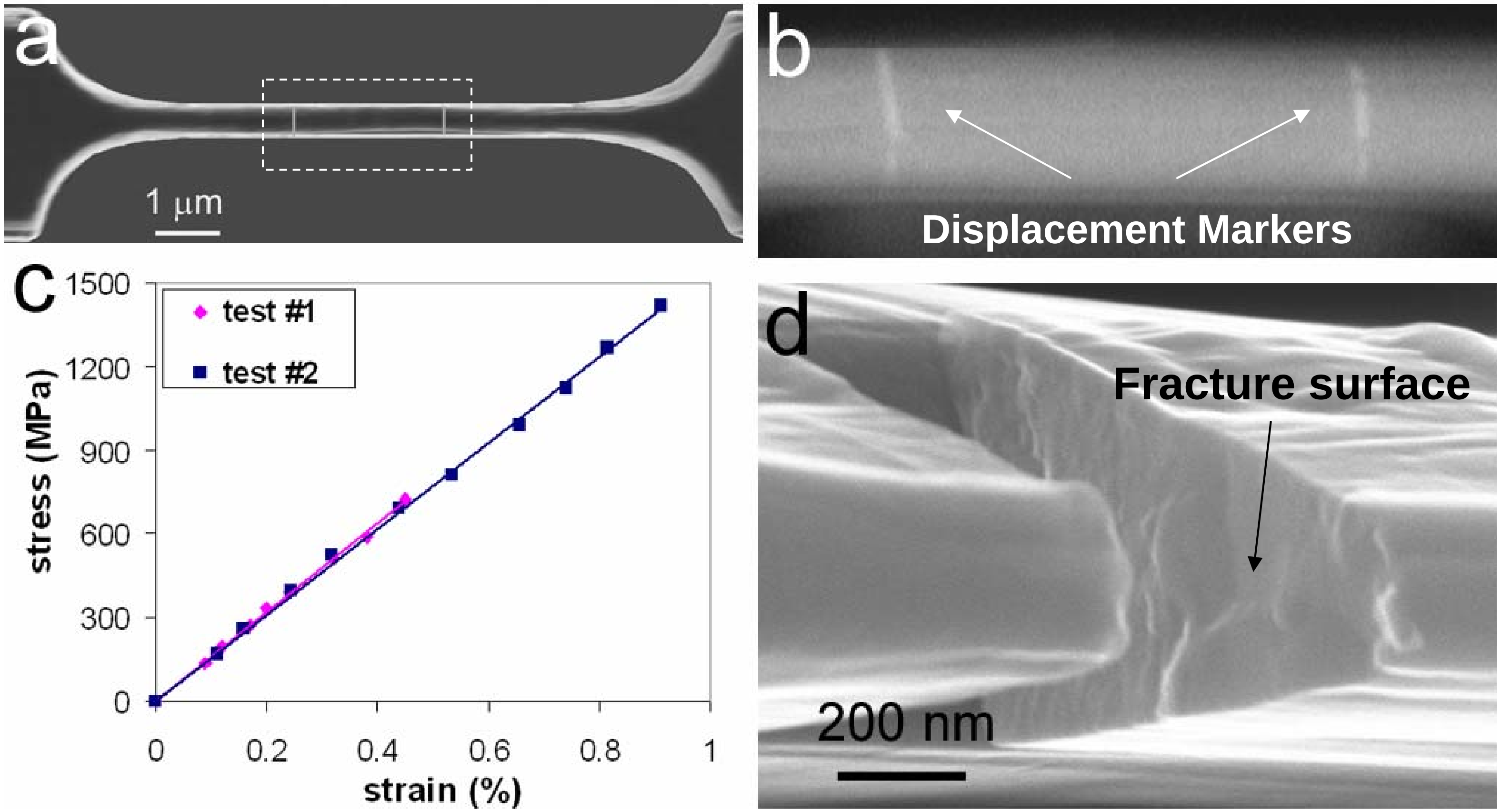


in-situ SEM Experiments

Nanoscale Polysilicon Film - 1



Nanoscale Polysilicon Film - 2



Young's modulus = 155±5 GPa

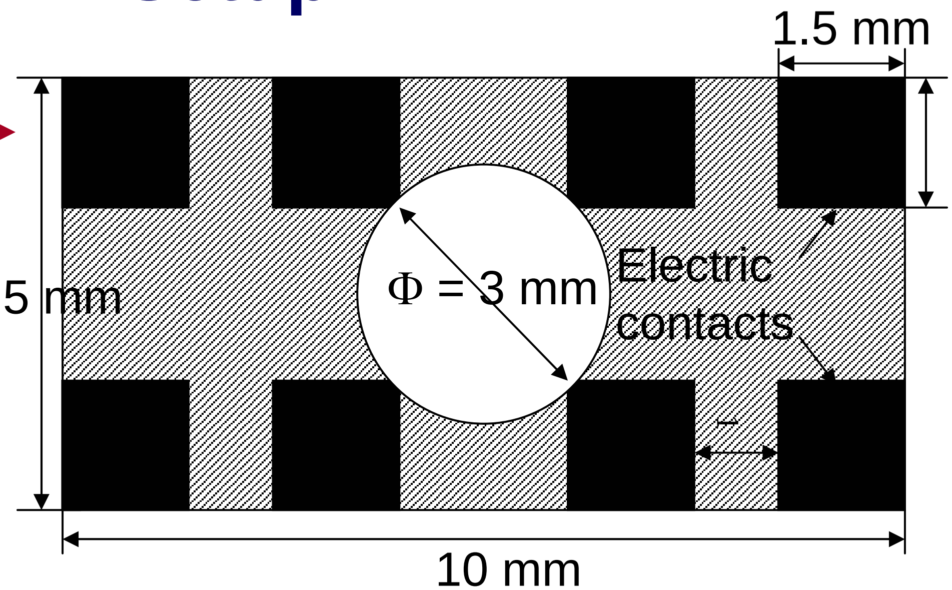
Fracture strength 0.72~1.42 GPa

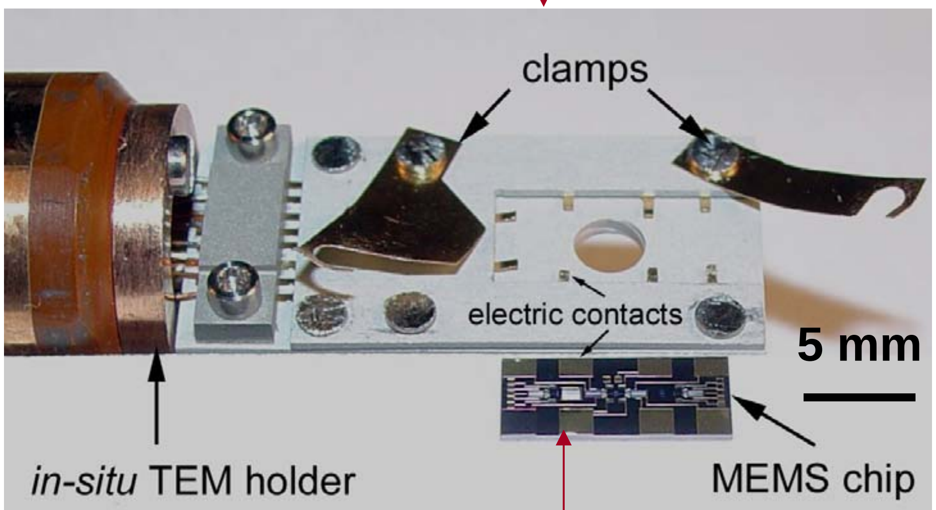
Zhu and Espinosa, *PNAS,* (2005)

in-situ SEM/TEM Experiments of CNTs

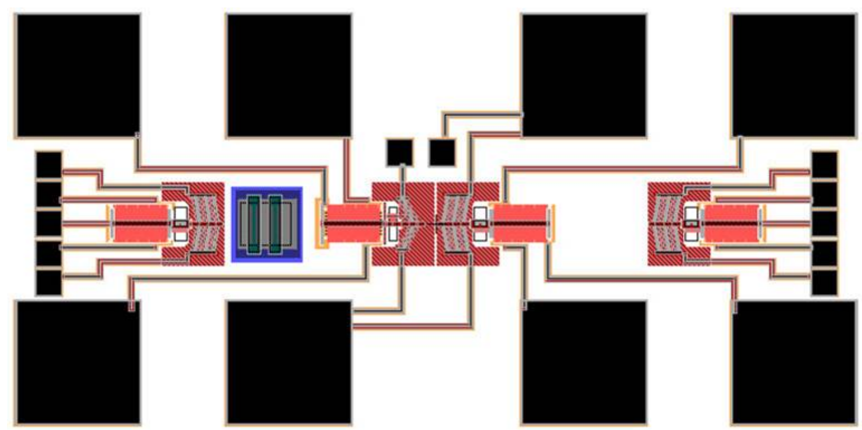
In-situ TEM Setup

TEM holder platform



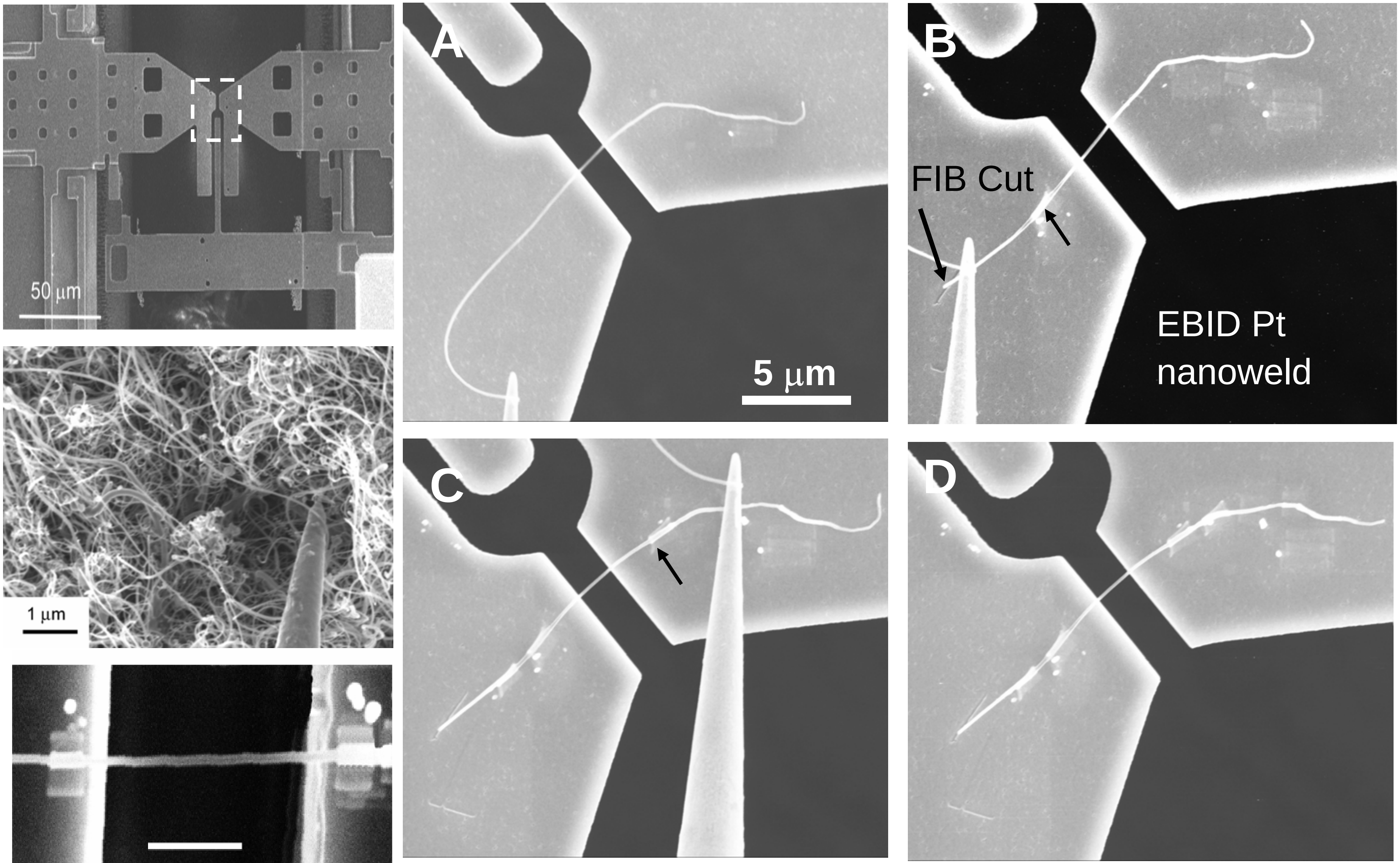


Chip with MEMS devices

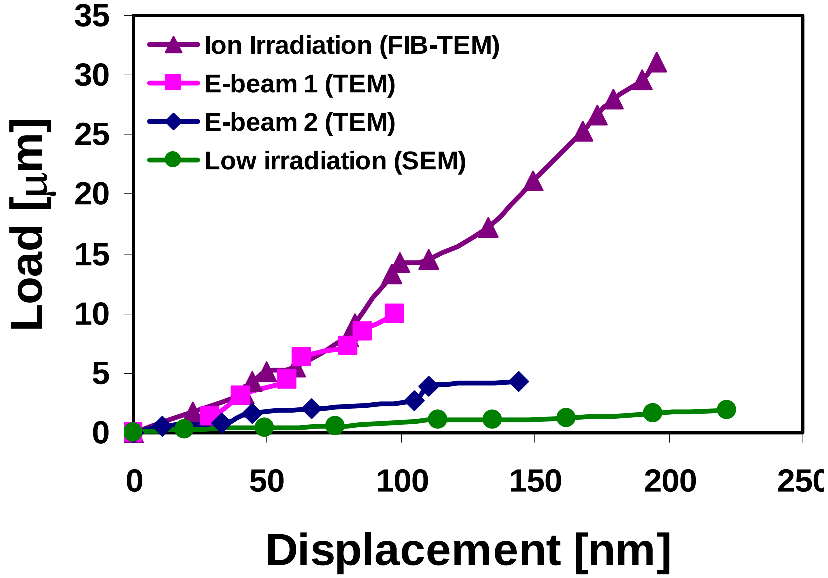


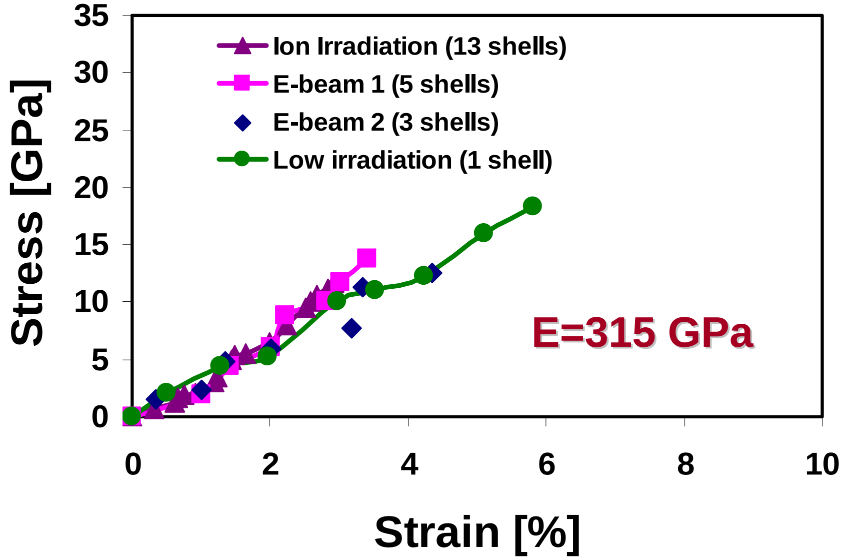
In collaboration with Prof. Ivan Petrov, Center for Microanalysis of Materials, UIUC

Specimen Mounting on MEMS



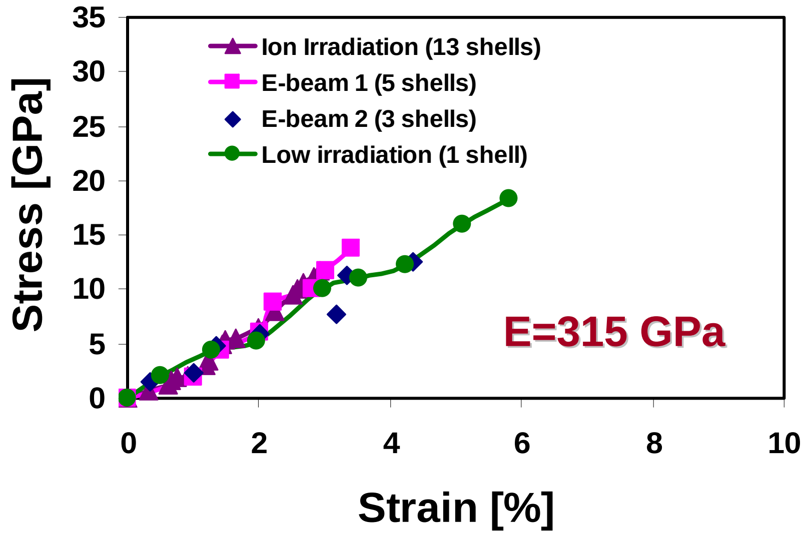
Experimental Data – CVD Grown MWCNTs



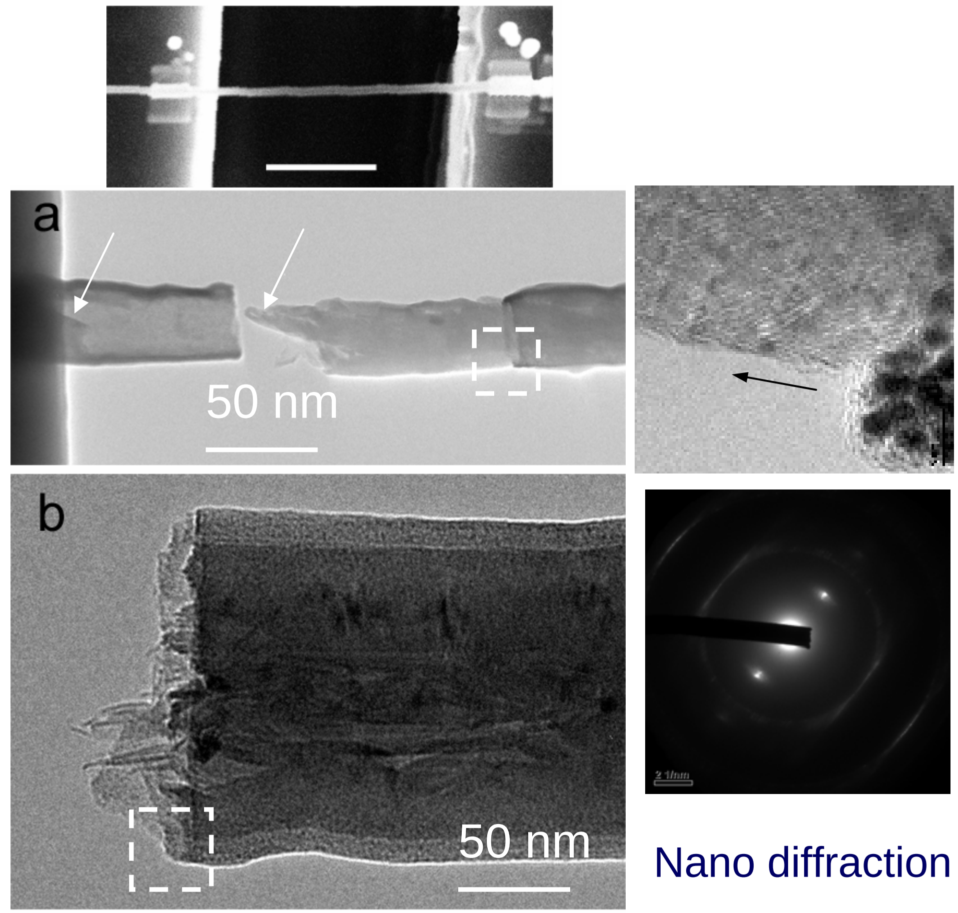


- E-beam 1 and 2 tested in TEM operated at 200 kV (Telescopic failure)
- E-beam 1 (nanowelding very close to shuttle edge, *failure inside observation window*), E-beam 2 (nanowelding ~1.5 μm from edge, *failure just outside observation window*)
- Ion Beam irradiation in FIB with gallium ions (entire cross-sectional failure)
- Low irradiation sample tested in SEM operated at 3-5 kV (Telescopic failure inside observation window)

Constitutive Behavior and Failure Modes



Two Failure Modes



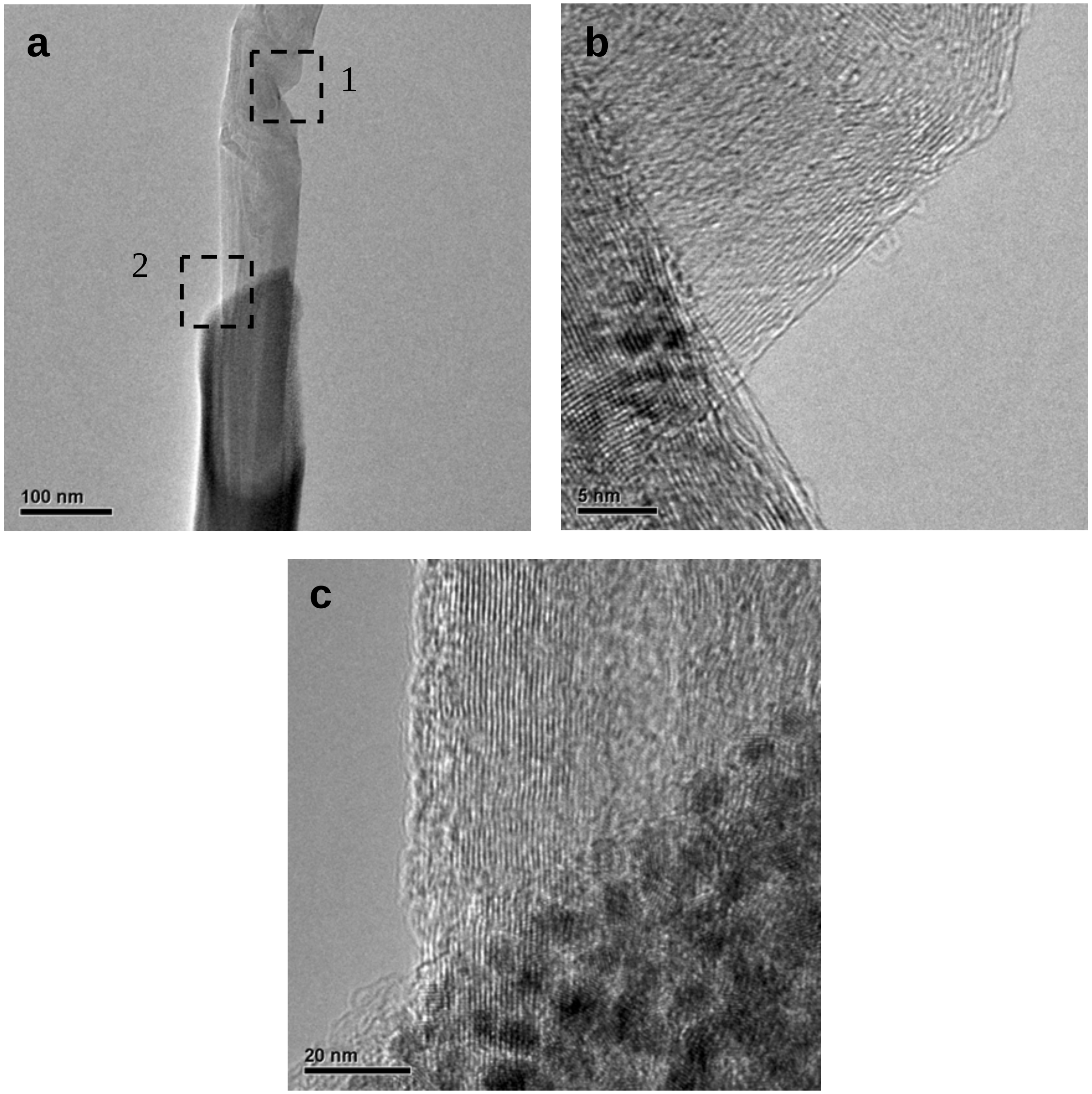
Nano diffraction

CVD Grown MWCNTs

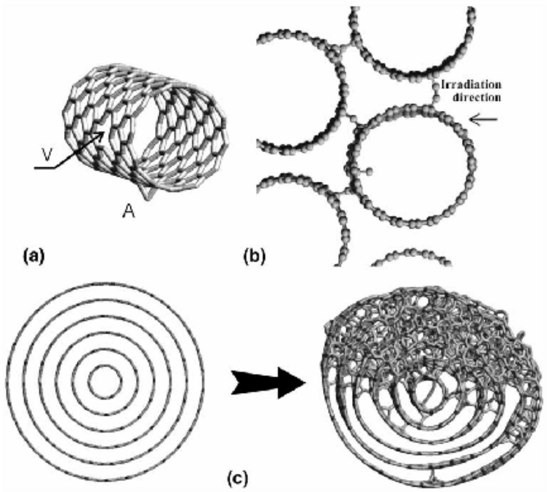
- E-beam 1 and 2 tested in TEM operated at 200 kV (T)
- Ion Beam irradiation in FIB with gallium ions (CS)
- Low irradiation sample tested in SEM operated at 5 kV (T)
- Young's Modulus E=315 GPa

T: telescopic ; CS: entire cross-section

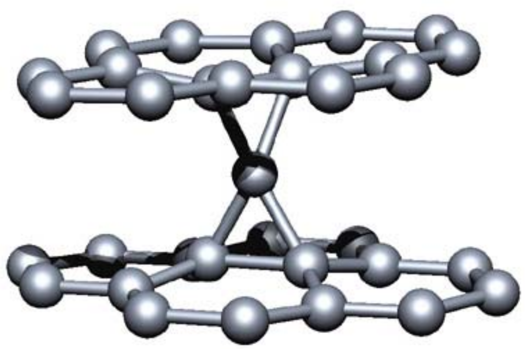
Telescopic Failure



Load Transfer Mechanism- Inter-Shell Bridging

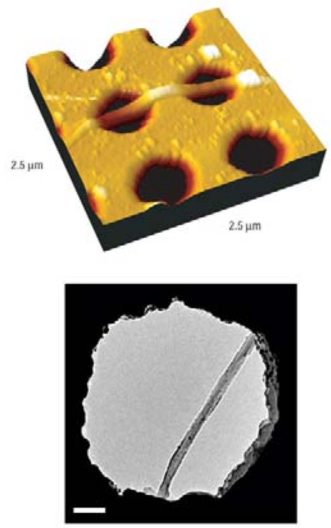


Vacancy or Interstitial threshold Energy is 86 keV

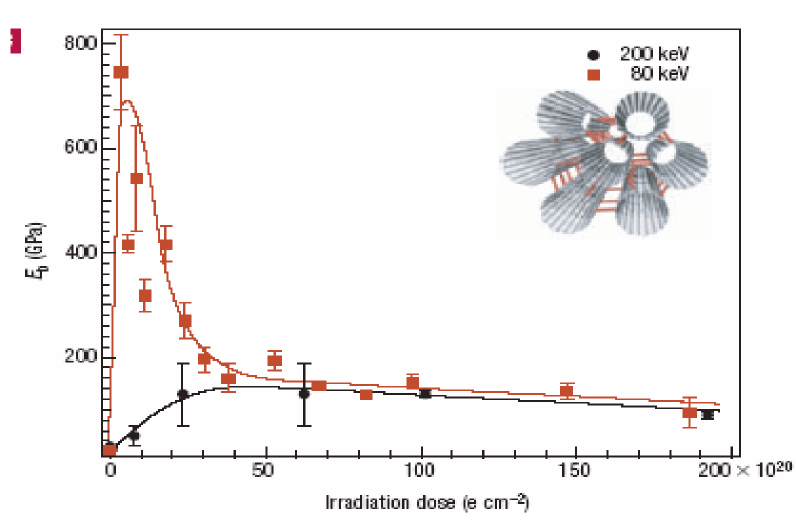


Telling et al., *Nature Mat.* (2003)

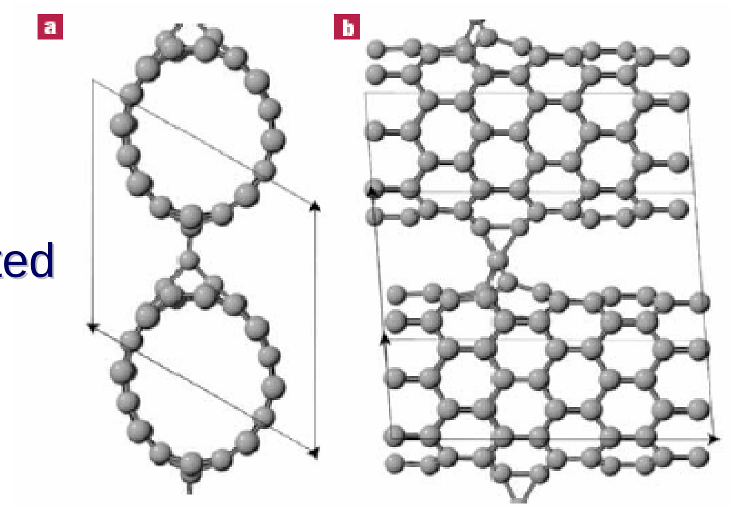
SWCNT bundles



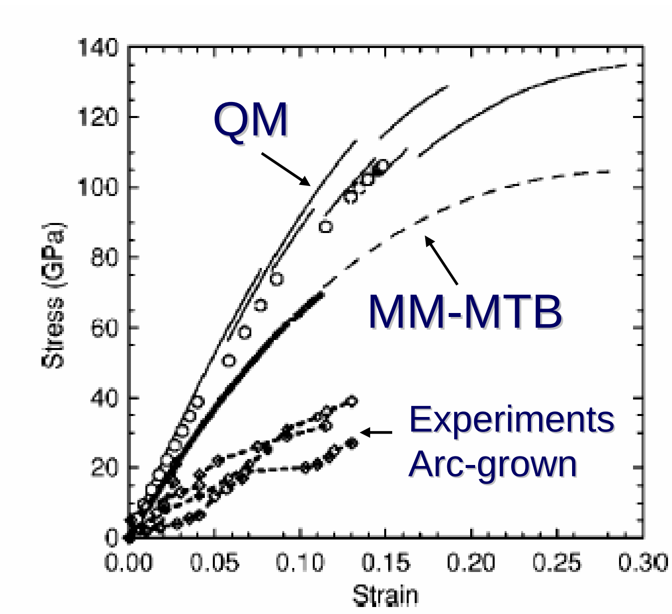
Kis et al., *Nature Mat.* (2004)

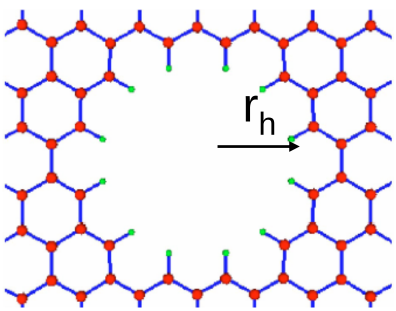


DFT prediction of C atom in a fourfold coordinated interstitial



Discussion



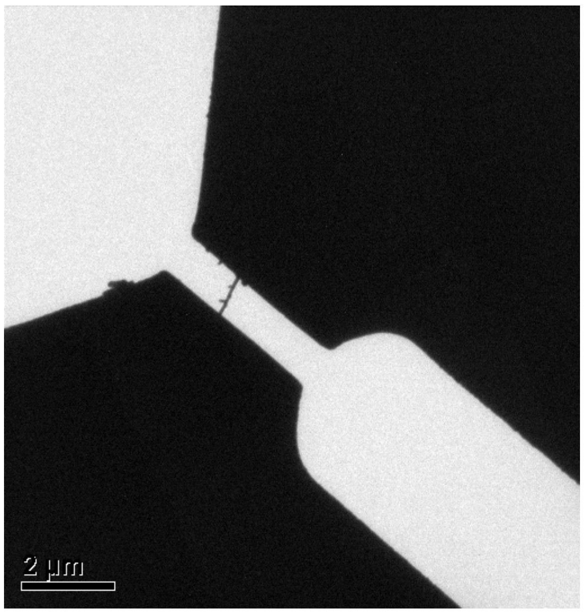


Zhang et al., *PRB*, 71 (2005)

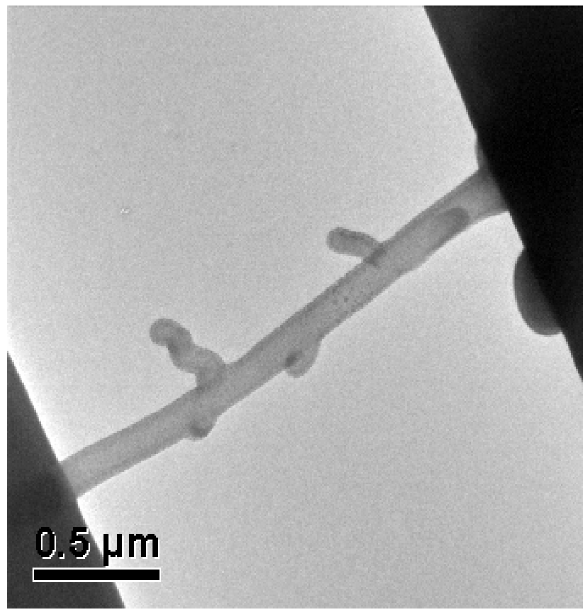
- CVD and arc grown MWCNTs exhibit the same E and comparable failure stresses.
- For the first time, *in-situ* TEM experiments reveal two failure modes with increasing number of failing shells as the irradiation dose and type is varied. The lowest dose was obtained in *in-situ* SEM experiments where single shell failure was observed.
- Failure stress shows statistical behavior consistent with brittle failure and volume scaling.
- Large number of missing atoms, holes with radii of about 3-4 nm are required to explain the low strengths experimentally measured.
- Models based on van der Waals inter-shell interactions are *insufficient* to explain the multi-shell failure observed experimentally. Additional QM studies of mechanical and electronic states are needed.

In-situ TEM Testing of Carbon Nanotubes

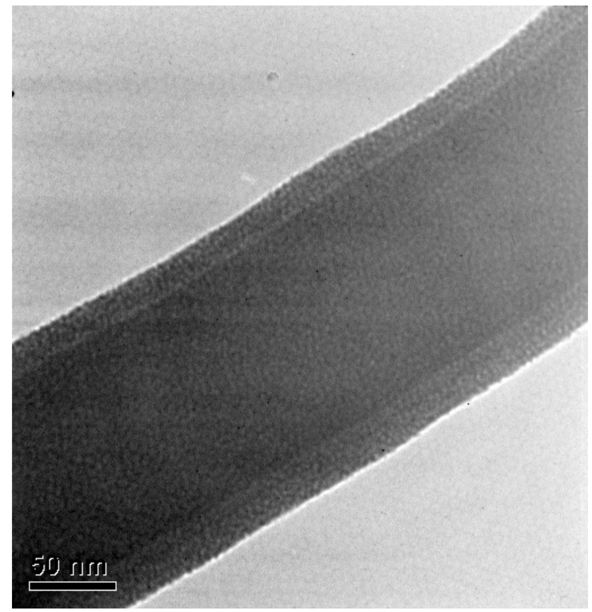
Before Straining



low mag.

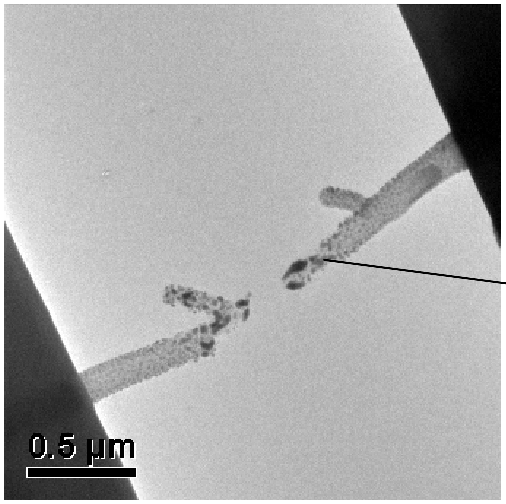


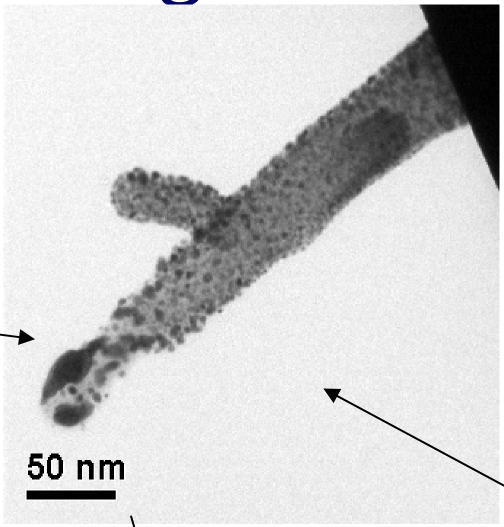
mid. mag.



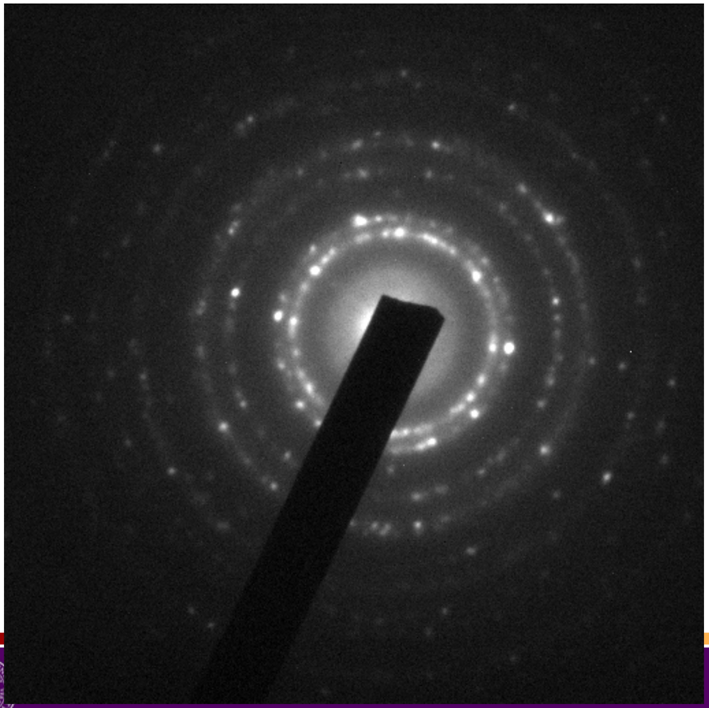
high mag.

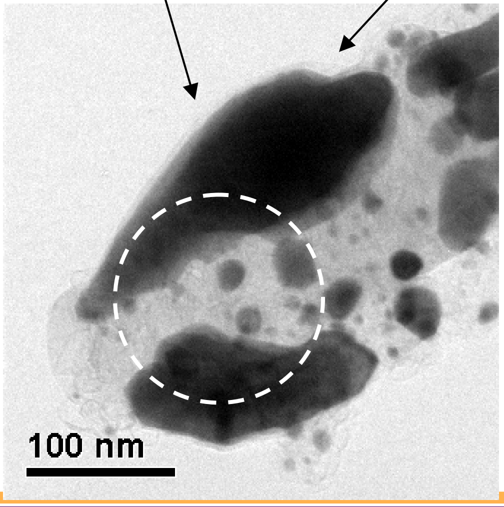
After Straining



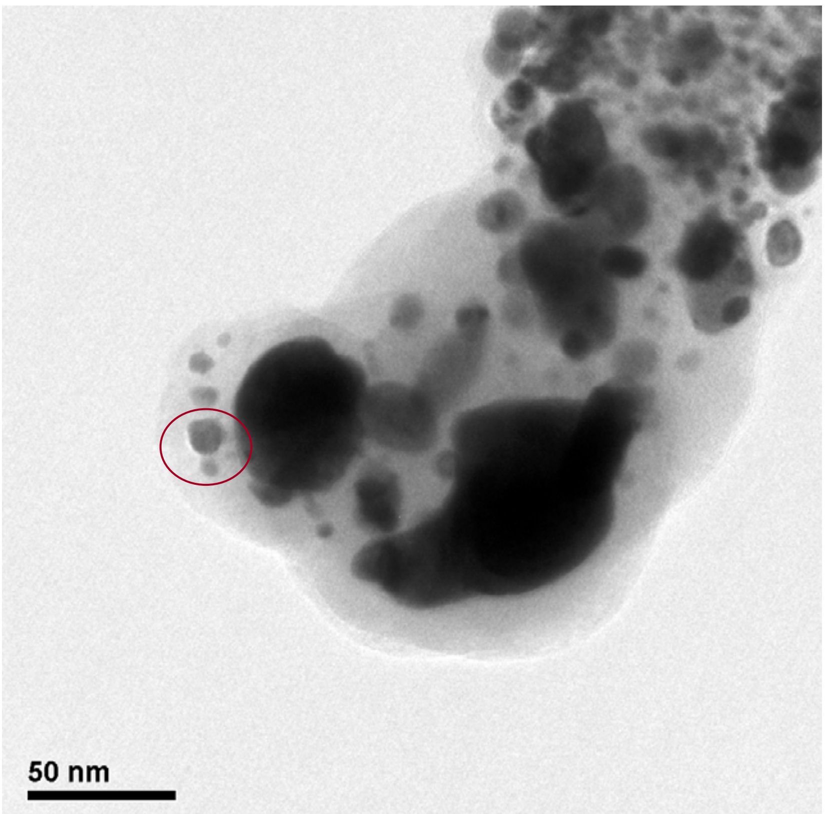


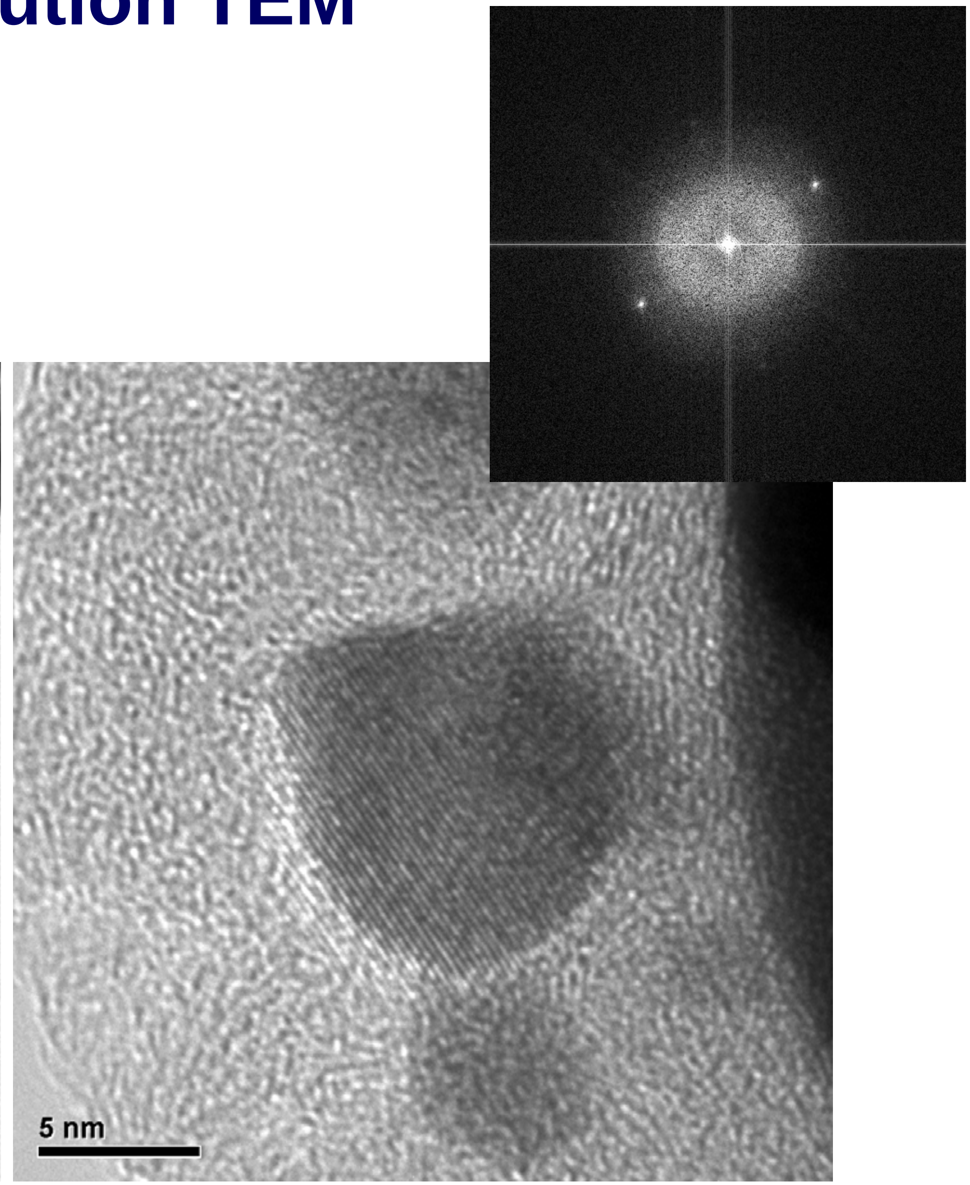
Walls disappeared?



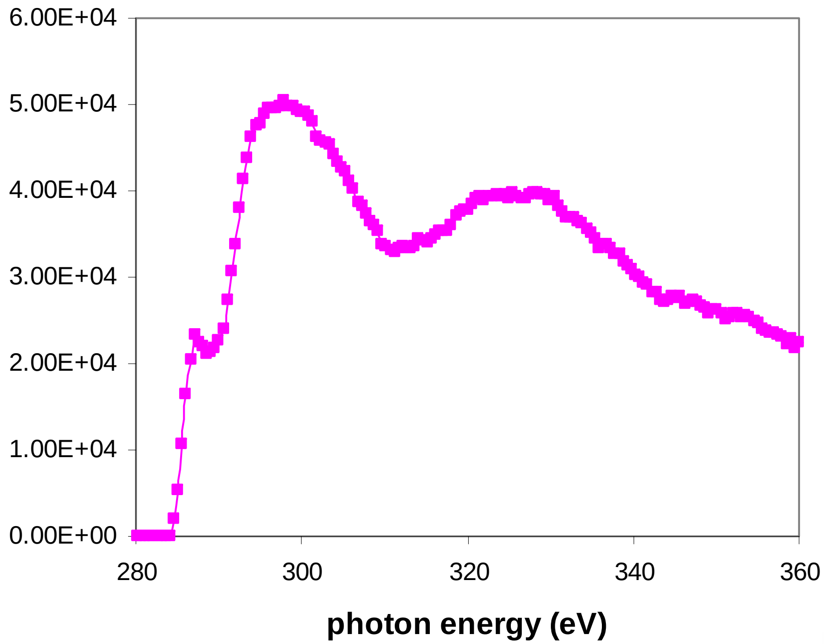


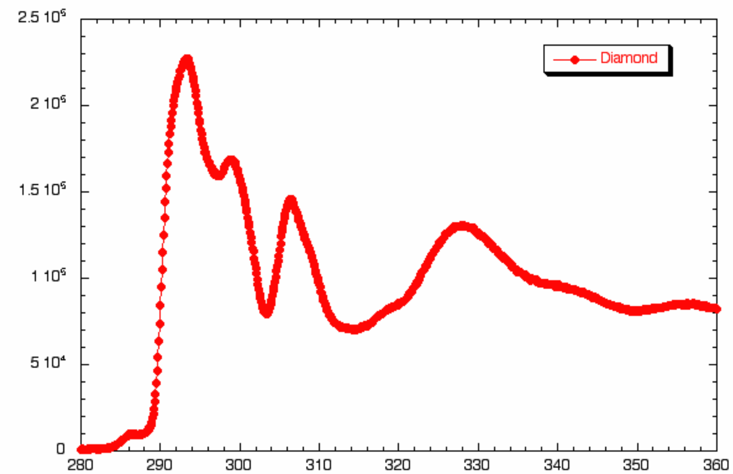
High Resolution TEM

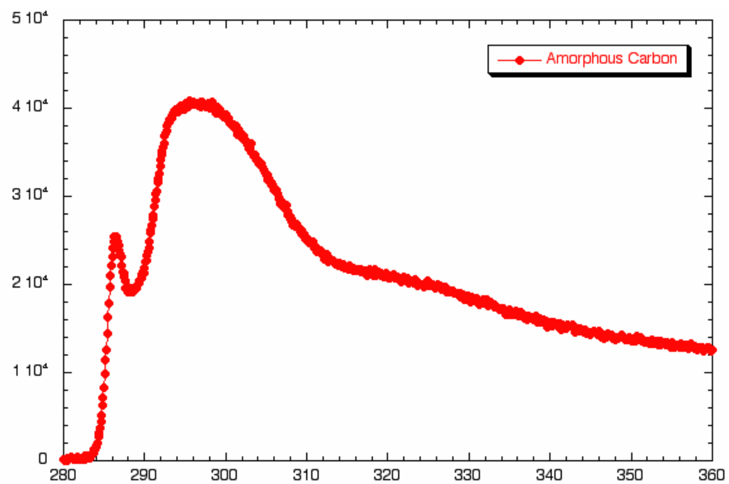


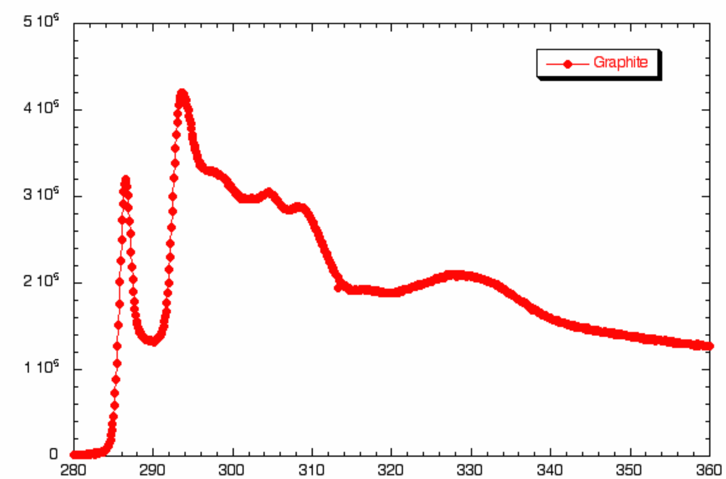


EELS Spectrum

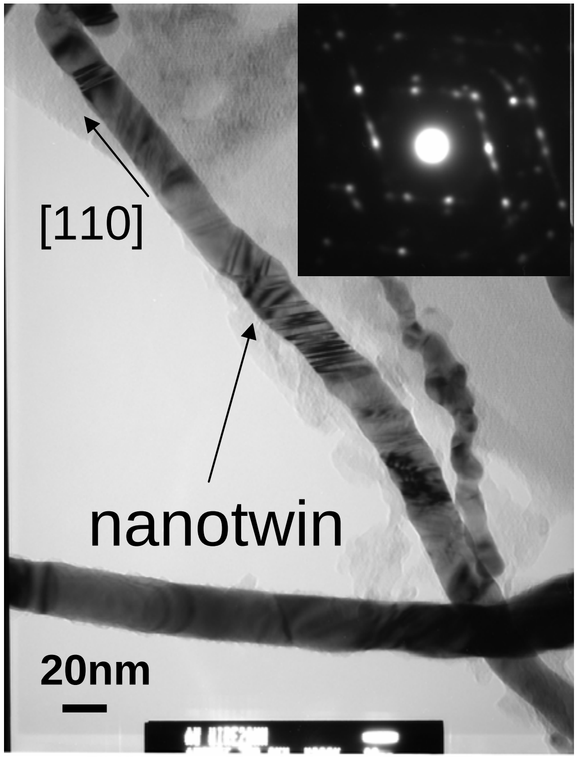


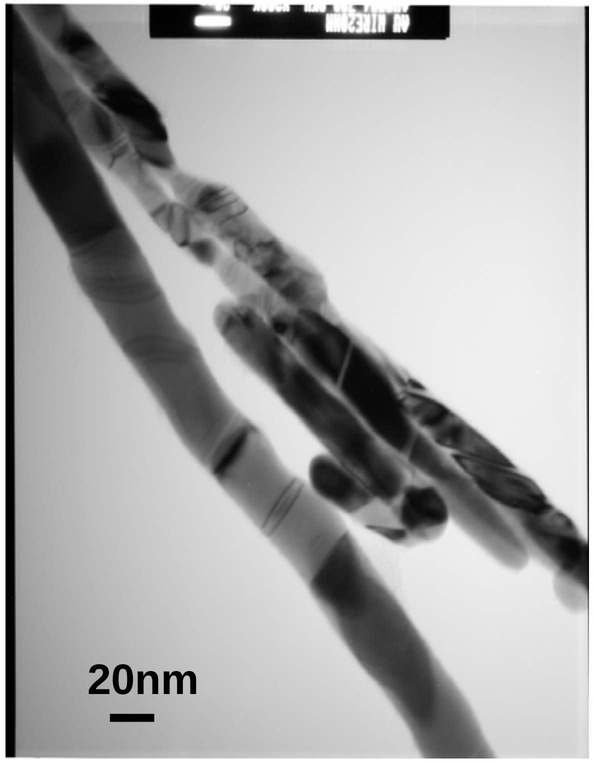




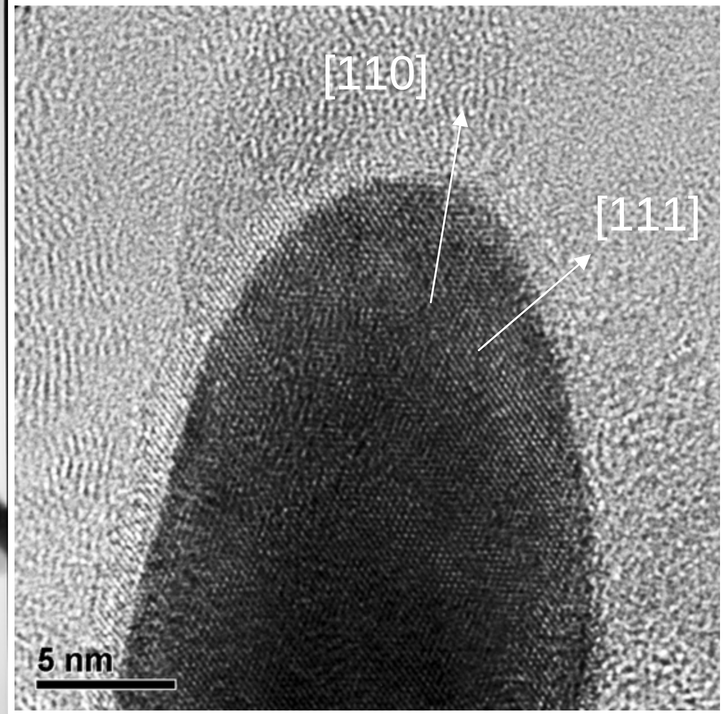


Gold Nanowires





HRTEM

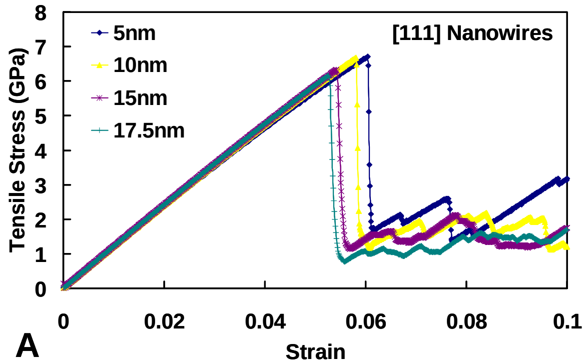


Dr. C. Li

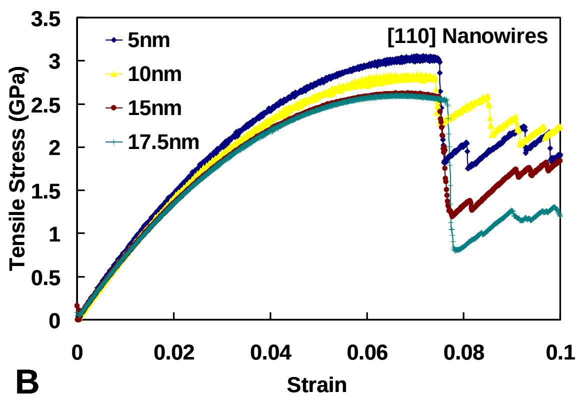
- **[110] growth direction**
- **Average diameter: 20±5 nm**
- **One grain through the thickness**
- **A large number of twins but no dislocations**

In collaboration with Dr. Hsien-Hau Wang, Materials Division, Argonne National Lab

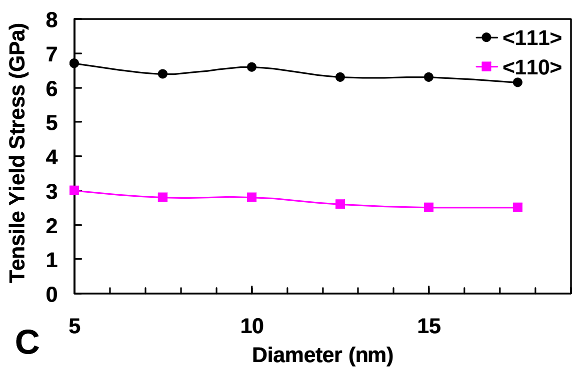
MD Simulations of NWs



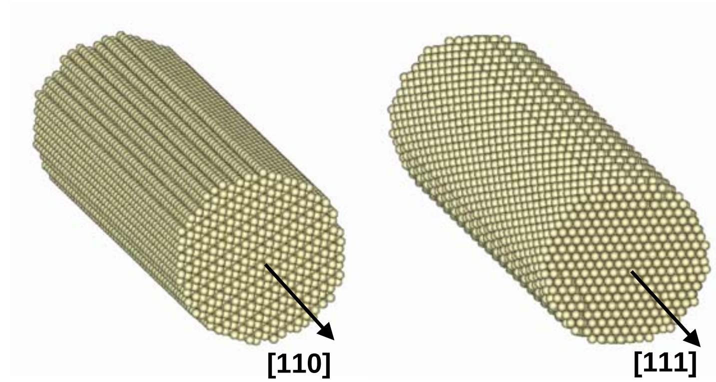
A

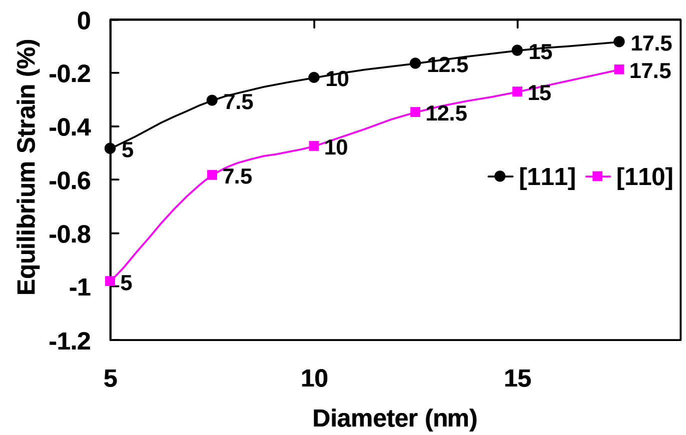


B



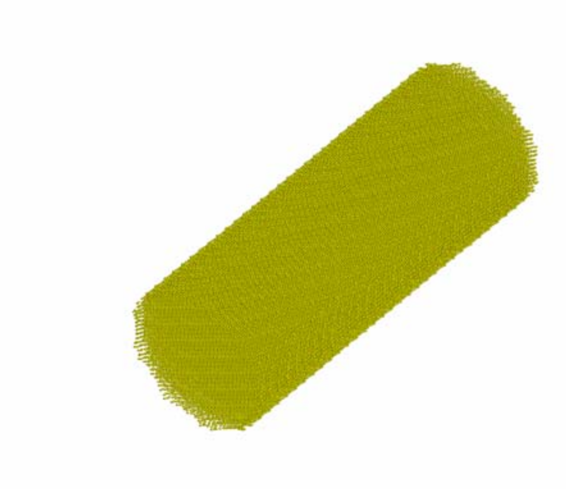
C





(A and B) Tensile stress responses for [111] and [110] axially oriented wires (as seen in Figure 1A and 1B, respectively) with representative radii from 5nm to 17.5nm. (C) Tensile yield stress as a function of diameter for all examined sizes. Both orientations show a drop in tensile yield stress with increasing diameter.

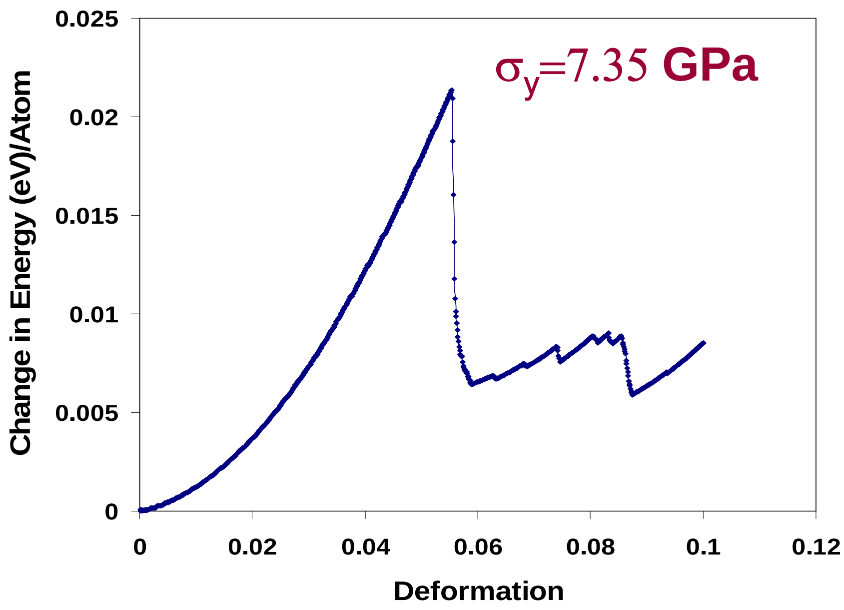
Role of Surface Defects

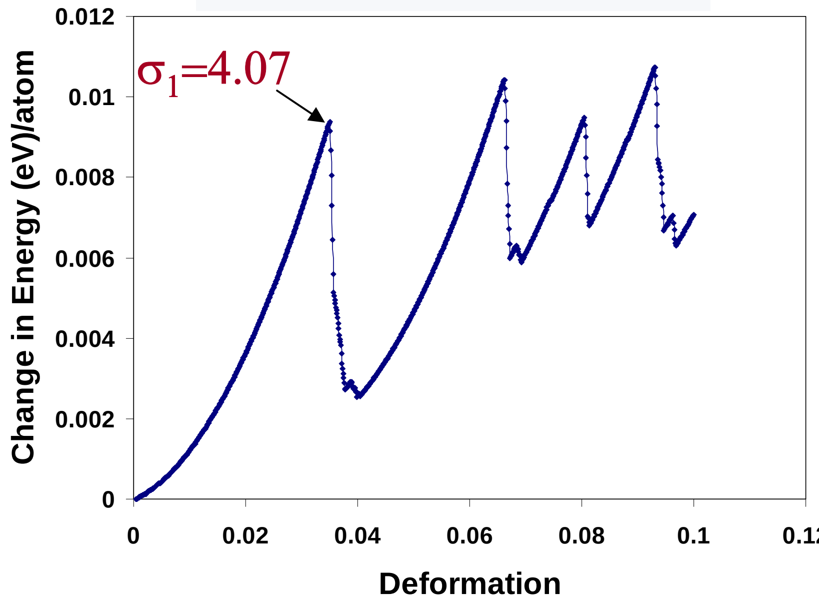


$$\frac{2\Delta E}{V} = E_0\varepsilon^2 + \frac{2}{3}\xi\varepsilon^3$$

Orientation <111>
D=5 nm
Strain Rate = $5\times10^{7}\ s^{-1}$

Step is created by a ½ <110> translation; perfect dislocation shift.





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