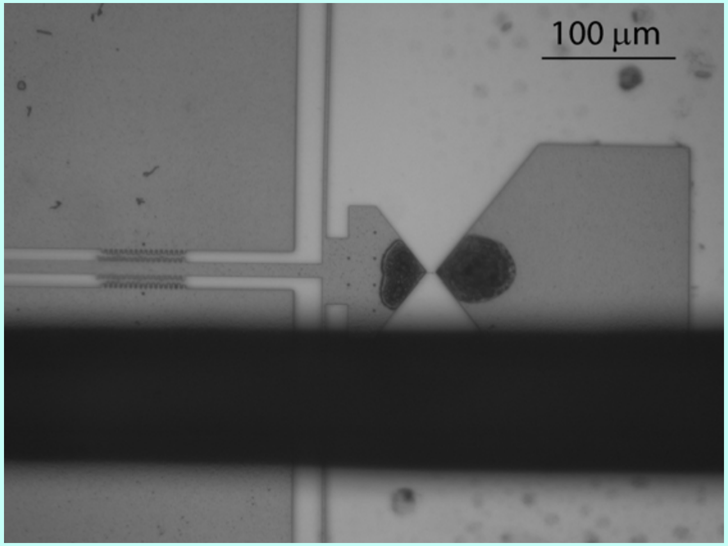
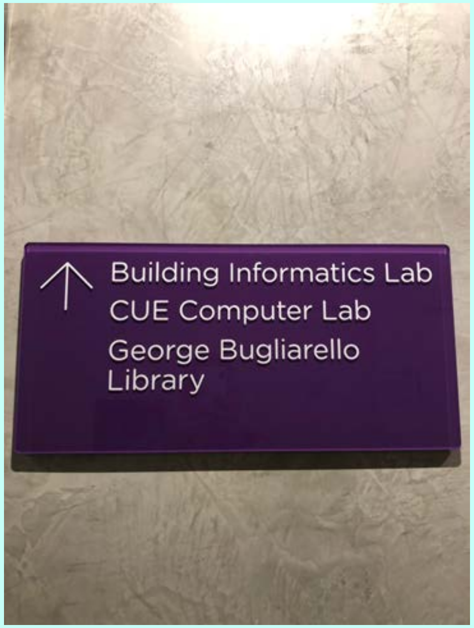


Structural Testing at the Micro and Nano Scales: Breaking Invisible Specimens with Zero Force

Roberto Ballarini
Thomas and Laura Hsu Professor
University of Houston

Support:

NSF
NASA
DARPA
NIH



NYU Tandon School of Engineering
12/3/18

I am an "experiment-inspired modeler"

$$\int_a^\infty \frac{2b_1(t)}{x-t}dt + \int_a^\infty K_{11}(x,t)b_1(t)dt + \int_w^a K_{12}(x,t)b_2(t)dt = 0 \qquad x > a$$

$$\int_w^a \frac{2b_2(t)}{x-t}dt + \int_w^a K_{21}(x,t)b_2(t)dt + \int_a^\infty K_{22}(x,t)b_1(t)dt = 0 \qquad w < x < a$$

$$\int_{-1}^1 \frac{\bar{b}_i(\eta)}{\xi-\eta}d\eta \approx \sum_{k=1}^N W_k \frac{\bar{g}_i(\eta_k)}{\xi_j-\eta_k} \qquad \int_{-1}^1 K_{mn}(\eta,\xi)\bar{b}_i(\eta)d\eta \approx \sum_{k=1}^N W_k K_{mn}(\xi_j,\eta_k)\bar{g}_i(\eta_k)$$

$$W_k = -\frac{2N+s_1+s_2+2}{(N+1)!(N+s_1+s_2+1)}\frac{\Gamma(N+s_1+1)\Gamma(N+s_2+1)}{\Gamma(N+s_1+s_2+1)} \times \frac{2^{s_1+s_2}}{P_N^{\prime(s_1,s_2)}(\eta_k)P_{N+1}^{(s_1,s_2)}(\eta_k)}$$

$$\Gamma = \frac{D}{Dt}\int_{D_t-S_t}\rho\eta\, dV + \int_{\partial D_t}\frac{q_i n_i}{\theta}\, dA - \int_{D_t-S_t}\frac{\rho\dot{r}}{\theta}\, dV \geq 0,$$

$$-\int_{\partial D_0}\dot{\phi}W_e\, dA_0 - \int_{S_0}\|\dot{\phi}D_J\|N_J\, dA_0 = \int_{\partial D_0}\left(\frac{\partial\phi}{\partial t}+v_k\frac{\partial\phi}{\partial x_k}\right)D_J N_J\, dA_0 - \int_{S_0}\left\|\left(\frac{\partial\phi}{\partial t}+v_k\frac{\partial\phi}{\partial x_k}\right)D_J\right\|N_J\, dA_0$$

$$i\hbar\frac{\partial}{\partial t}\Psi(\mathbf{r},t) = \hat{H}\Psi = \left(-\frac{\hbar^2}{2m}\nabla^2 + V(\mathbf{r})\right)\Psi(\mathbf{r},t) = -\frac{\hbar^2}{2m}\nabla^2\Psi(\mathbf{r},t) + V(\mathbf{r})\Psi(\mathbf{r},t)$$

$$J = \sum_{\alpha\in S_0}\sum_{i,j}^{2}\left[\left\{W^\alpha\delta_{1j} - P_{ij}^\alpha\frac{\partial u_i(\mathbf{X}_\alpha)}{\partial X_1}\right\}\frac{\partial q(\mathbf{X}_\alpha)}{\partial X_j}\right]S_0^\alpha,$$

$$\sigma_{rr} + i\tau_{r\theta} = \Phi_i(z) + \overline{\Phi_i(z)} - \frac{\bar{z}}{z}[z\overline{\Phi_i'(z)} + \overline{\Psi_i(z)}]$$

$$\sigma_{\theta\theta} + i\tau_{r\theta} = \Phi_i(z) + \overline{\Phi_i(z)} + \frac{z}{\bar{z}}[\bar{z}\Phi_i'(z) + \Psi_i(z)]$$

$$\frac{\partial}{\partial\theta}(u_x + iu_y) = \frac{iz}{2\mu_i}\left\{\kappa_i\Phi_i(z) - \overline{\Phi_i(z)} + \frac{\bar{z}}{z}[z\overline{\Phi_i'(z)} + \overline{\Psi_i(z)}]\right\}$$

Outline: A glimpse of experiments and modeling for itty-bitty structures

Experiments at small scales

Micro:
***Active* and *passive* devices for fatigue, fracture and strength of MEMS materials**

Nano:
Collagen fibrils, carbon nanotubes,
carbon nanotube/polymer interfaces

Modeling tools

Molecular dynamics-informed continuum micromechanics

Discrete element method for mesoscopic modeling of carbon nanotubes

Coupled and uncoupled multiscale-multiphysics simulations

Good old continuum mechanics

Students/Post-docs
Faculty/industry colleagues

Collaborators

Yuye Tang, Lucas Hale
Bill Gerberich, Traian Dumitrica
University of Minnesota

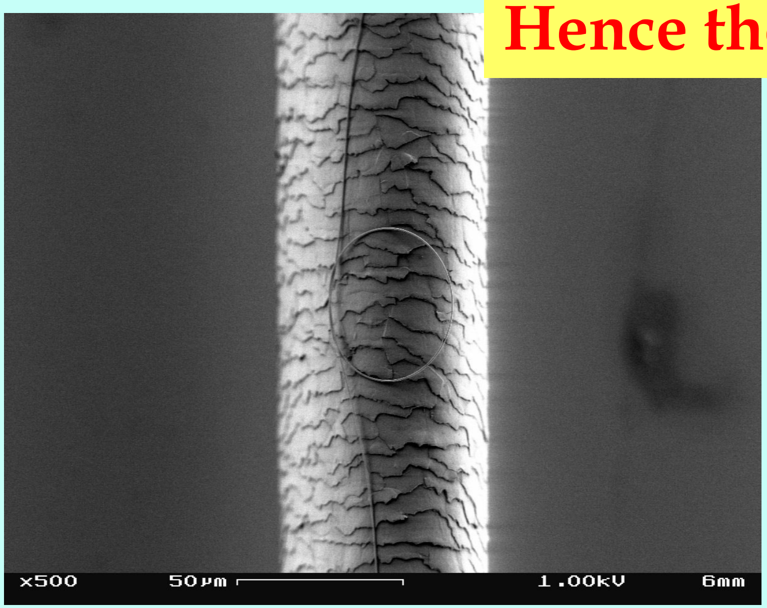
Brandy Smith, Julie Liu, Hal Kahn,
Steve Eppell, Arthur Heuer
Case Western Reserve University

Yogee Ganesan, Jun Lou, Boris Yakobson
Rice University

Brad Boyce, Michael Dugger, Maarten de Boer
Sandia National Laboratories

Itasca Consulting Group, Inc.

Ted Belytschko (R.I.P) and Markus Buehler
Northwestern University and M.I.T.

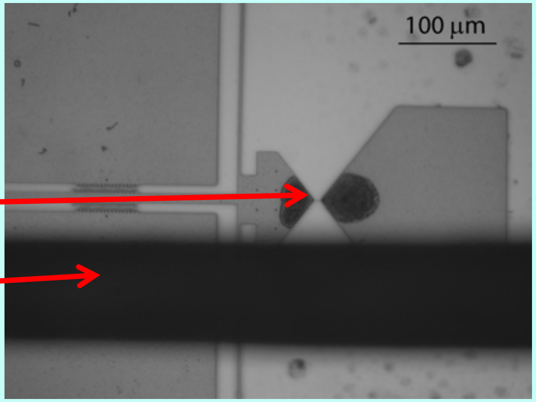


Hence the title

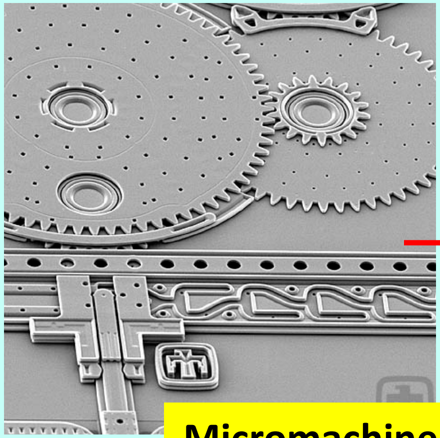
What is a Newton force?
It is ~100 grams of Chinese sausage.
A µN is simply not enough sausage.

What is a micron (µm)?
It is ~1/100 the thickness of hair.

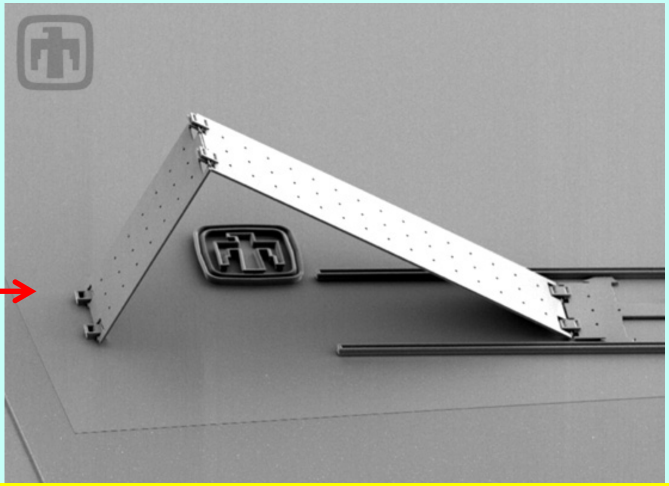
So, for example, a collagen fibril is 1000 thinner than the hair.



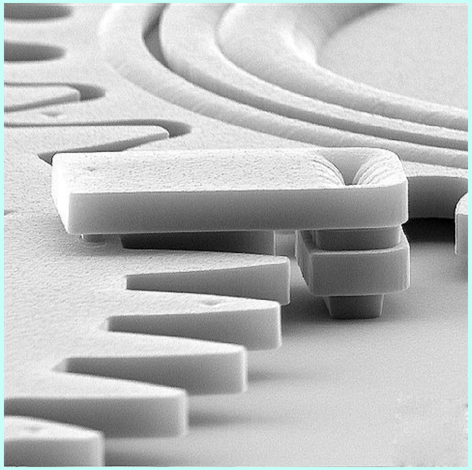
MEMS applications



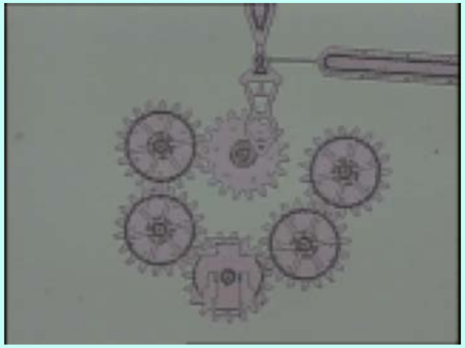
Micromachine

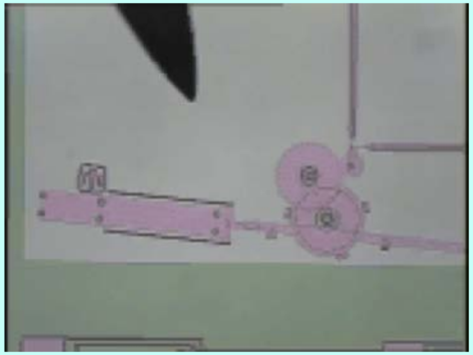


Micromachine-driven foldable mirror



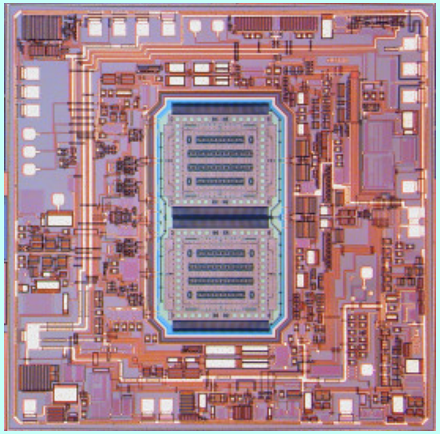
Details



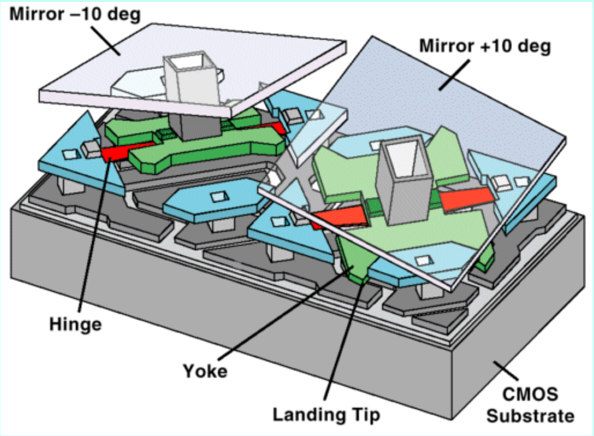


Sandia National Laboratory

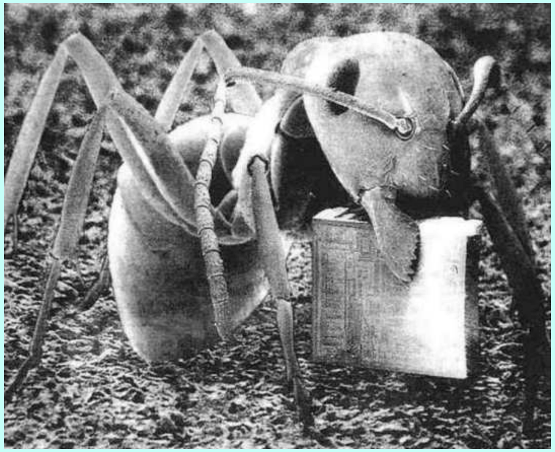
Analog Devices gyroscope

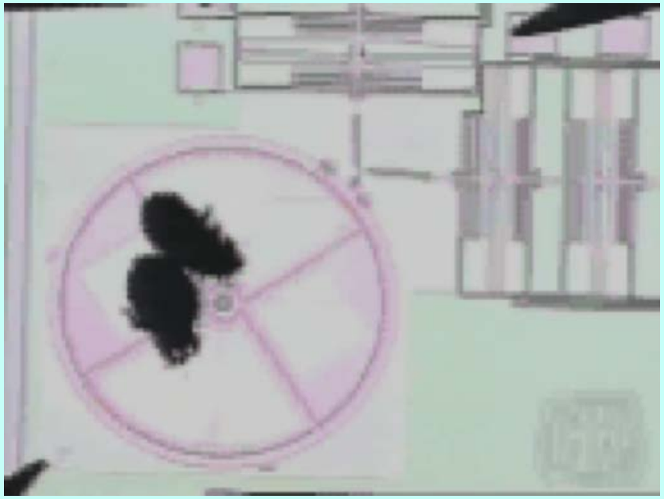


Texas Instruments DLP



The real markets





MEMS materials and structures

"Electrostatically Actuated Failure of Microfabricated Polysilicon Fracture Mechanics Specimens," *Proc. Roy. Society of London,* **1999.**

"Fatigue Failure in Polysilicon Not Due to Simple Stress Corrosion," *Science,* **2002.**

"Mechanical Fatigue of Polysilicon: Effects of Mean Stress and Stress Amplitude," *Acta Materialia,* **2006.**

"Fracture Toughness of LPCVD Polycrystalline Silicon Carbide Thin Films," *J. of Applied Physics,* **2006.**

"Using Microfabricated Devices to Determine the Fracture Strength of Materials," *Int. J. Mat. Research,* **2010.**

"Modeling of Probabilistic Failure of Polycrystalline Silicon MEMS Structures," *J. of the American Ceramic Society,* **2015.**

"Temperature Dependent Fracture Initiation in Microscale Silicon," *Scripta Materialia,* **2017.**

Nanoscale fiber-like structures

"Deformation mechanisms of collagen fibrils under uniaxial tension," ***J. Royal Society Interface,*** **2010.**

"Nanomeasurements with microdevices: mechanical properties of hydrated collagen fibrils," ***J. Royal Society Interface,*** **2006.**

"Interface Toughness of Carbon Nanotube Reinforced Epoxy Composites," ***J. American Chemical Society: Applied Materials and Interfaces,*** **2011.**

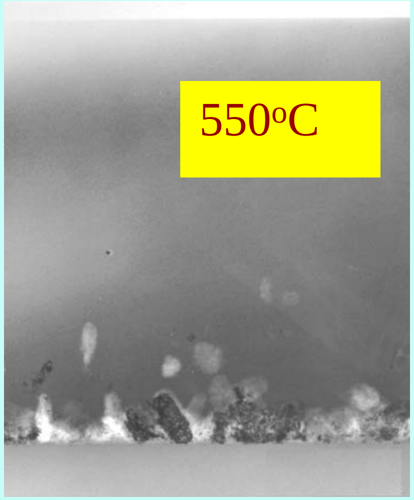
"Stress-strain experiments on individual collagen fibrils," ***Biophysical Journal,*** **2008.**

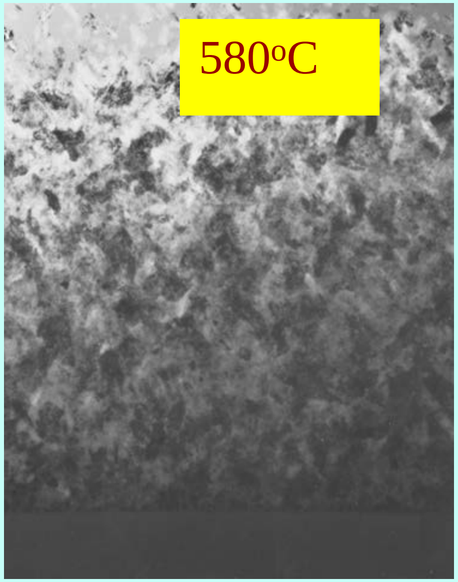
"*In Vitro* fracture testing of submicron diameter collagen fibril specimens," ***Biophysical Journal,*** **2010.**

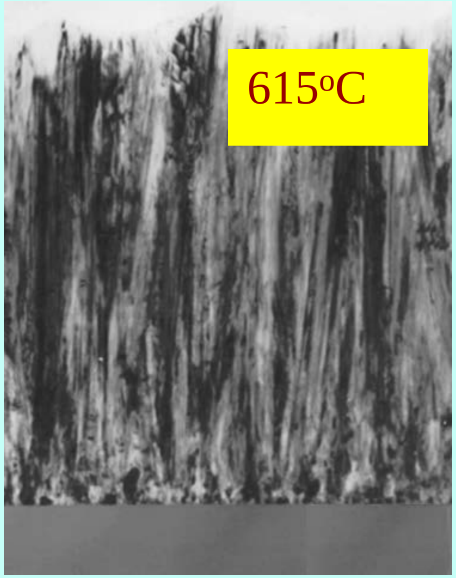
"Development and Application of a Novel Micro-fabricated Device for *In Situ* Tensile Testing of 1-D Nanomaterials," ***J. of MEMS*****, 2010.**

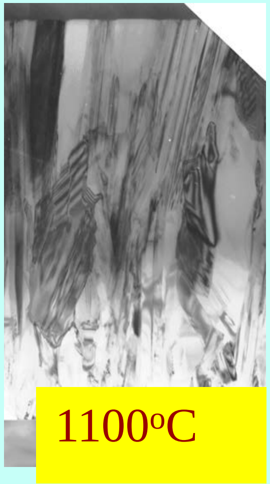
"Tension testing of mammalian collagen: A first report," ***J. Royal Society Interface,*** **2016.**

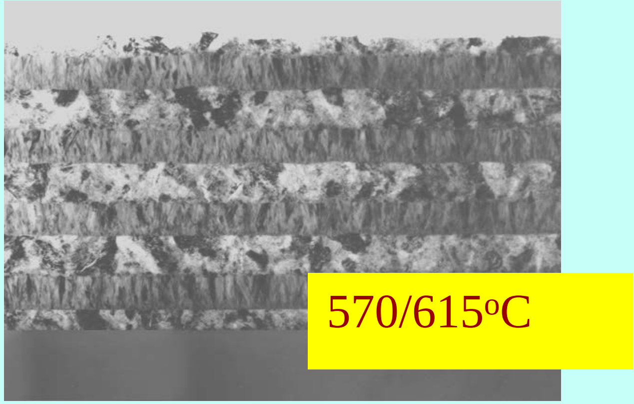
Mechanical properties depend on fabrication procedures



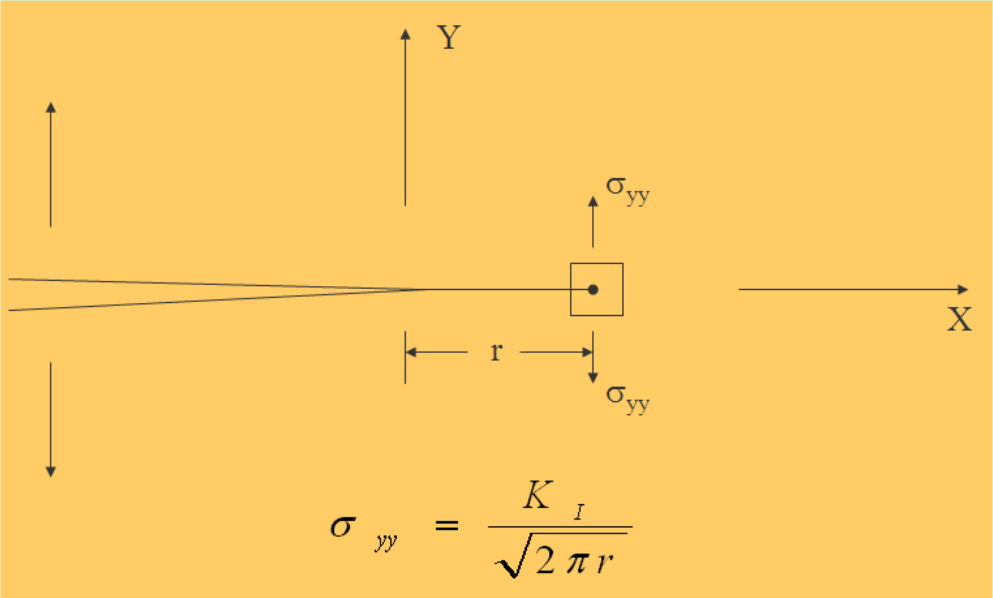








Fundamental Studies: Possible crack growth mechanisms



Fast fracture

$$K_I = F(a/b)\sigma\sqrt{\pi a} = K_I^{cr}$$

High cycle fatigue

$$\frac{da}{dN} = C\left(\Delta K_I\right)^m \quad ???$$

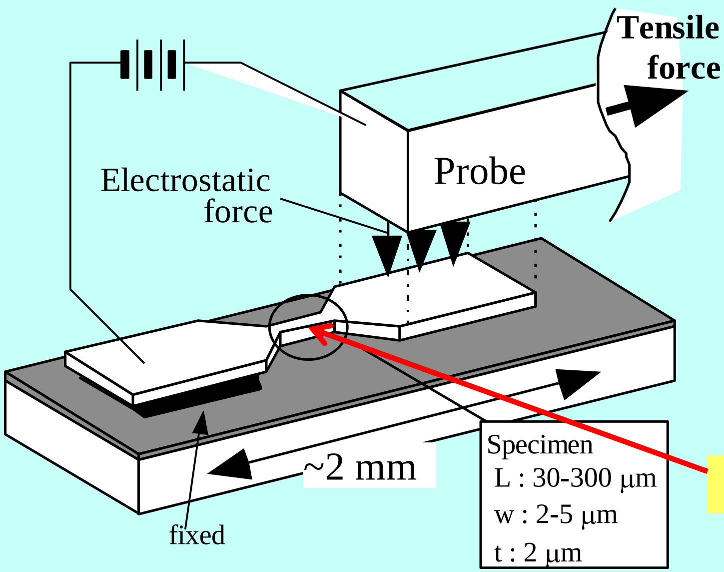
Stress corrosion

$$\frac{da}{dt} = DK_I^n \quad ???$$

If applicable, how sensitive are the parameters to processing procedures?

Reliability Issues

Example: Weibull approach to design of brittle MEMS

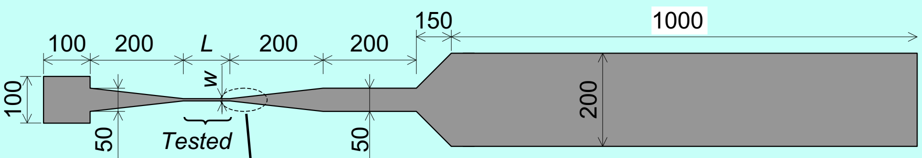


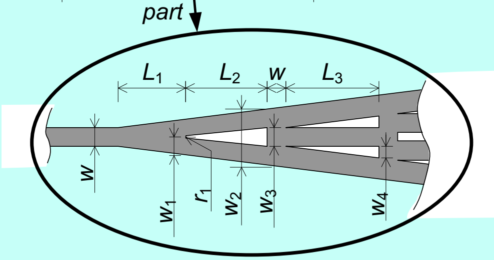
Gauge region smaller than RVE

$$P_f = 1 - \exp\left[-\frac{\sigma^m}{\alpha} V_E\right] = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_\theta}\right)^m\right]$$

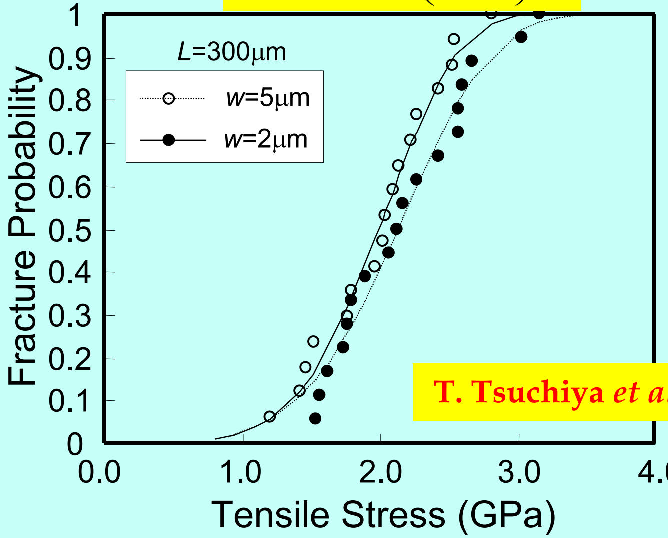
$$\frac{\mu}{\sigma_\theta} = \Gamma\left(\frac{1+m}{m}\right)$$

$$\frac{\sigma^{allow}}{\mu} = \frac{\left[-\ln(1-P_f)\right]^{1/m}}{\Gamma\left(\frac{1+m}{m}\right)}$$





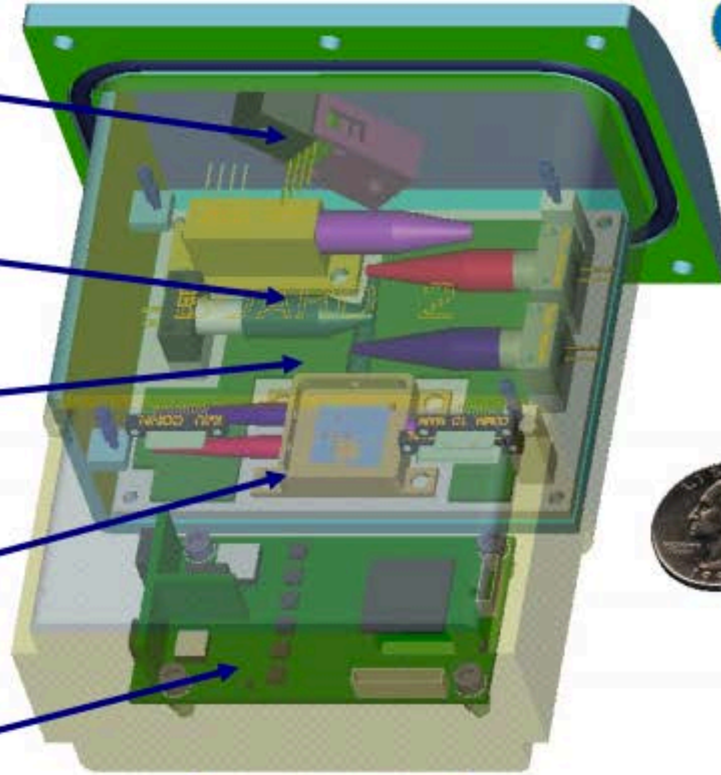
w (μm)	2	5
L_1 (μm)	6	10
L_2 (μm)	10	30
L_3 (μm)	10	23
w_1 (μm)	1.5	3.5
w_2 (μm)	6	15
w_3 (μm)	2	5
w_4 (μm)	1.25	2.9
r_1 (μm)	0.5	0.5



T. Tsuchiya *et al.*, 1998

Torpedo S&A Technology
- CCAT S&A EDM-1 Design

- Integrated MEMS based COTS pressure sensor for water flow sensing
- Optical Charging System
- S&A Electronics - Processor and logic Circuit
- MEMS Safe-Arm chip package <1 cu inch
- Low Cost & High Accuracy MEMS based IMU
- Integrated initiator Fire-set, slapper, lead

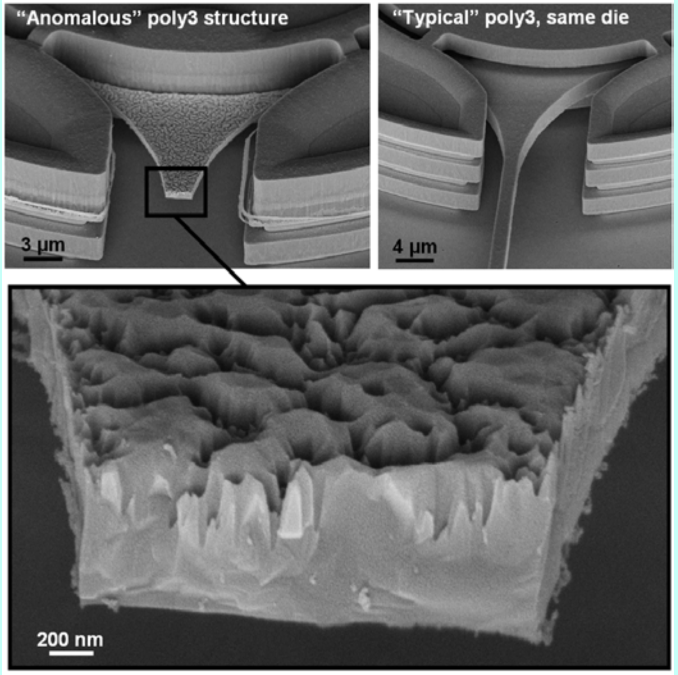


MEMS Safe-Arm Chip Provides Interruption and Arming Energy Control

To confidently design for very small probabilities of failure it is *necessary* to understand the fundamental mechanisms of failure (what are the “correct” distributions), and the coupling between fabrication, structural shape, and these mechanisms. It is not clear whether this will be *sufficient*. But it is worth trying.

If not …

An argument for proof testing?

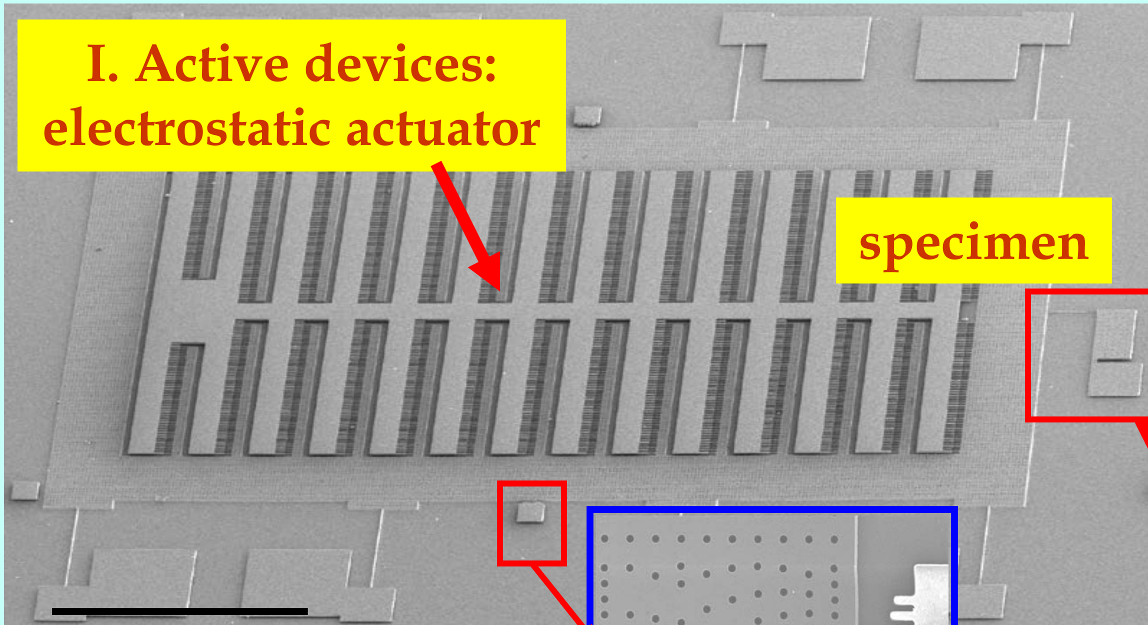


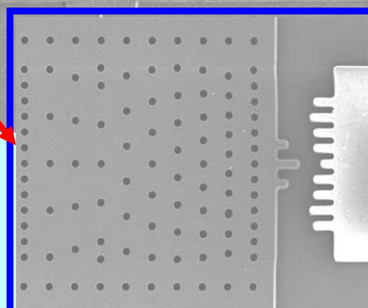
Anomalous defect morphology found n Sandia's SUMMiT V polysilicon MEMS poly3 layer. Fracture strength <0.05 Gpa, whereas the characteristic strength from a large collection of tensile tests was 2.35 GPa!!!!!

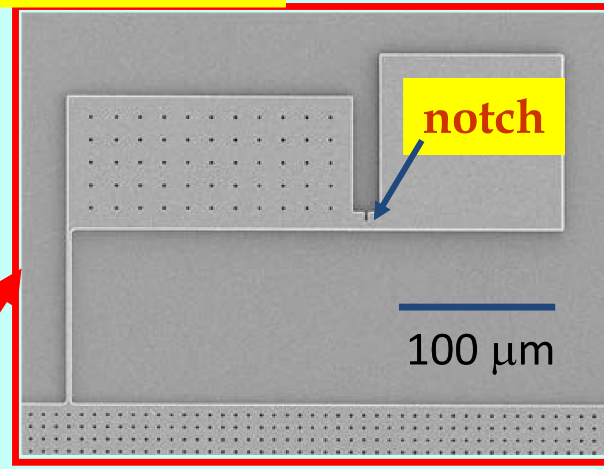
***Boyce, Ballarini, Chasiotis, Journal of Micromechanics and Microengineering*, 2010**

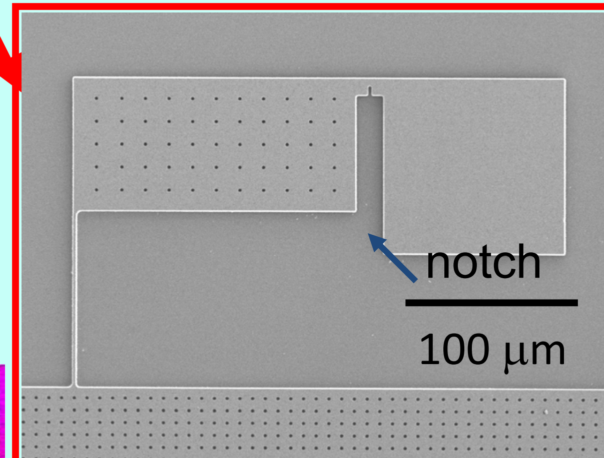
Our initial "on-chip" paradigm for studying mechanisms of fracture, fatigue and strength of MEMS

I. Active devices: electrostatic actuator



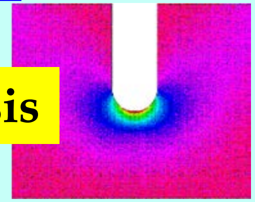






$$F = \frac{1}{2}\frac{\partial C}{\partial x}V^2 = n\varepsilon\frac{h}{g}V^2$$

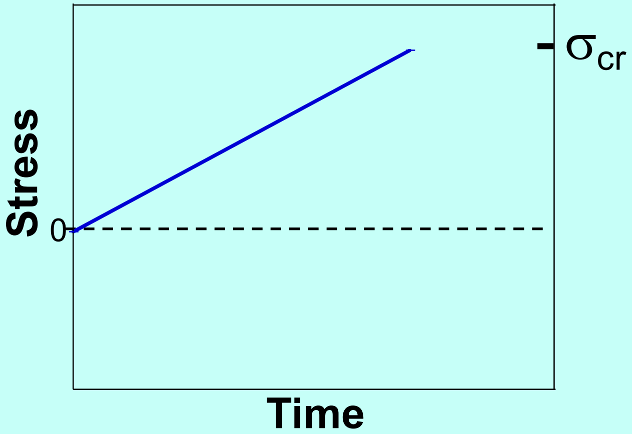
1438 pairs of comb fingers; 0.8 mN at 150 V



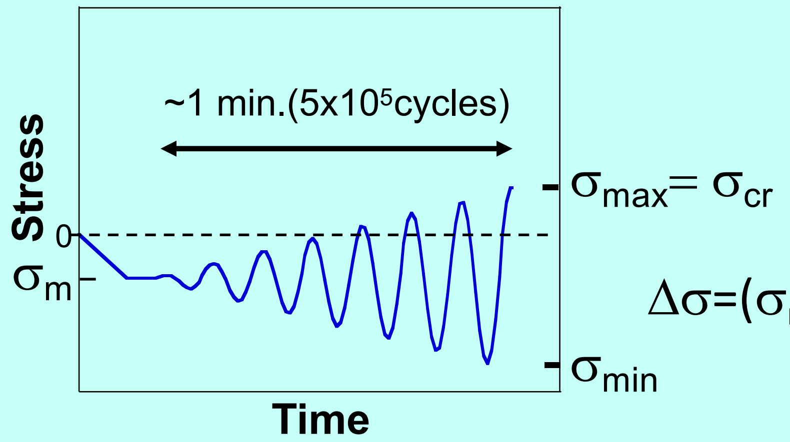
stress analysis

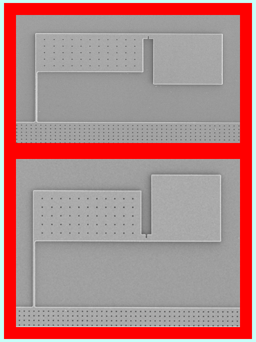
Proc. Roy. Society of London, 1999
Science, 2001

Monotonic



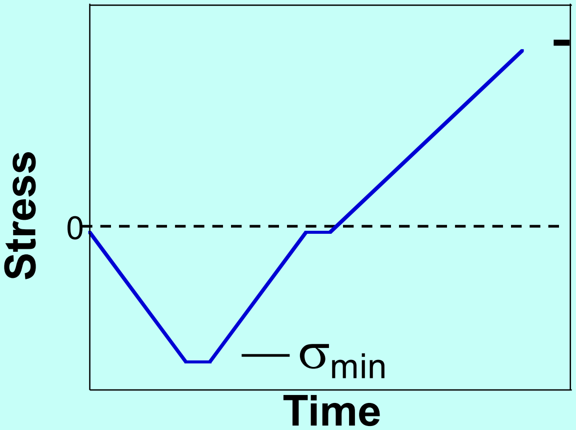
Increasing Amplitude Fatigue



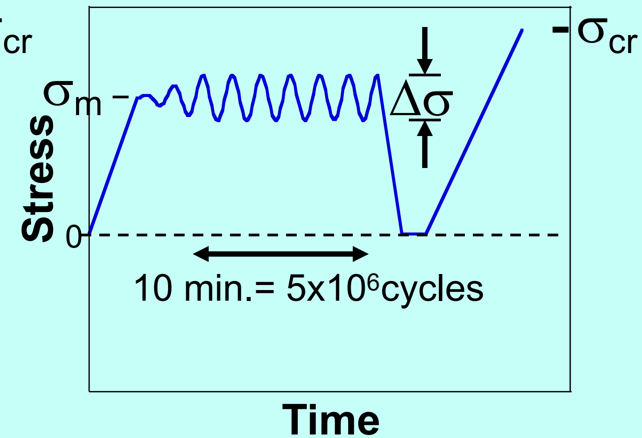


$\Delta\sigma=(\sigma_{max}-\sigma_{min})$

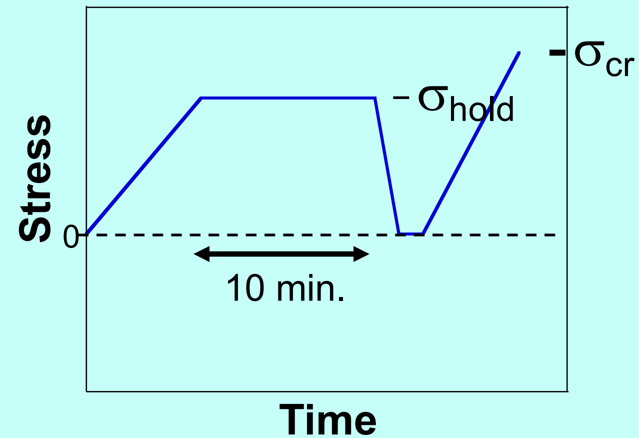
1 Comp/Mono



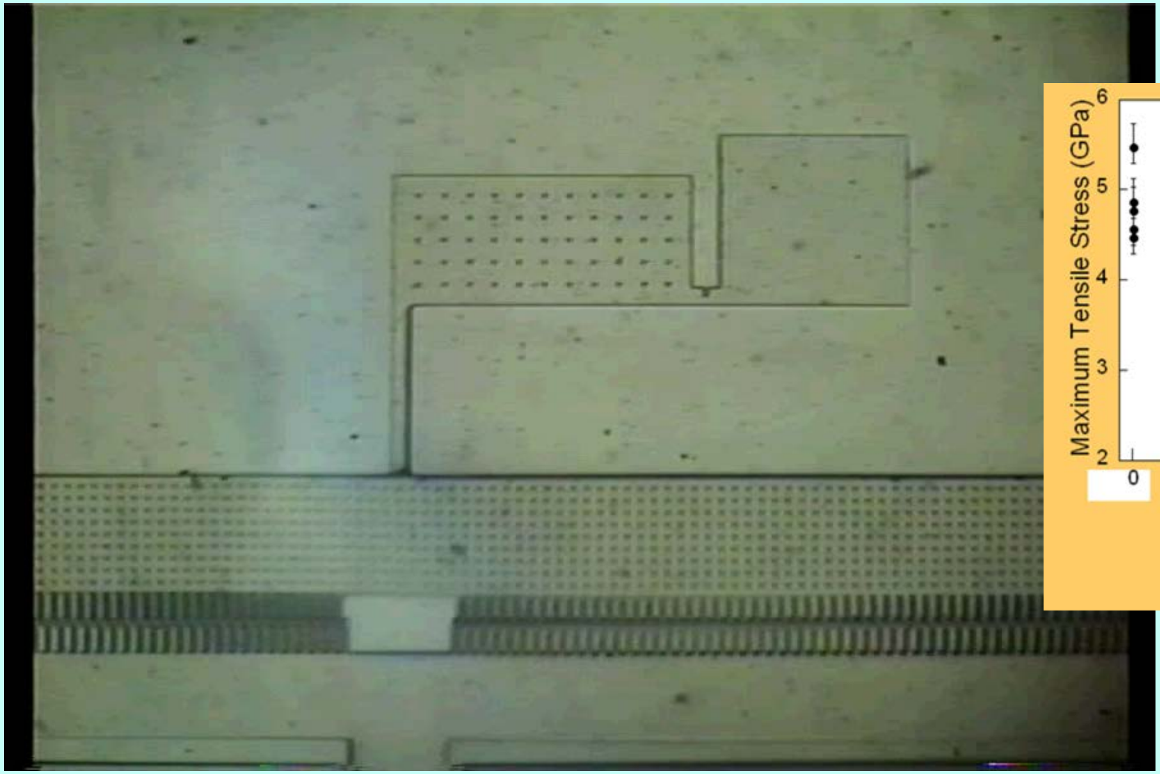
Fixed Δσ Fatigue/Mono

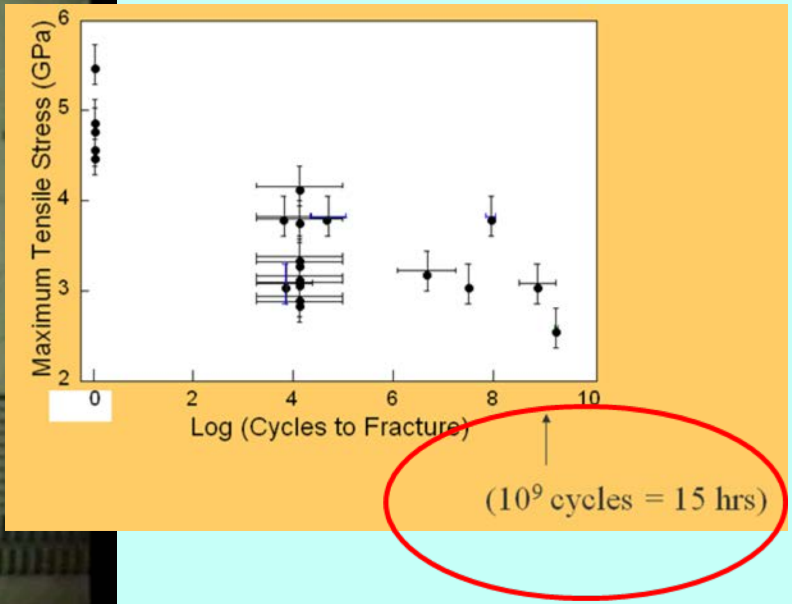


High T Hold/Mono

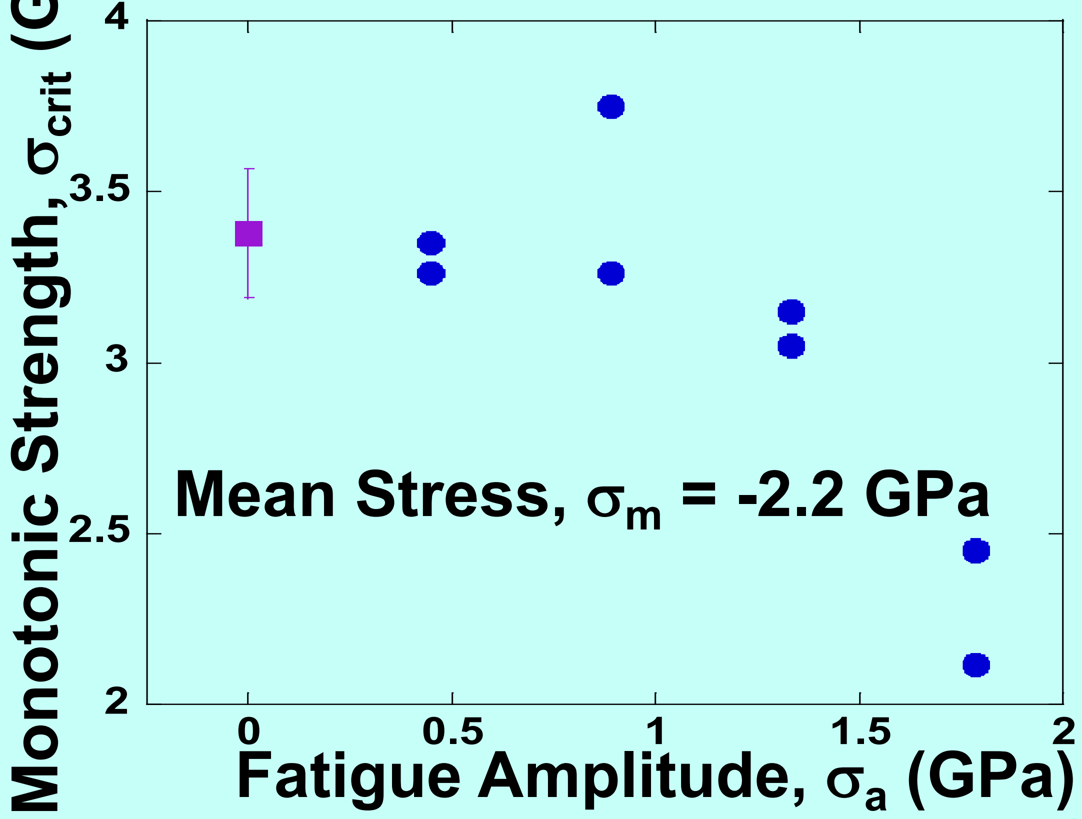


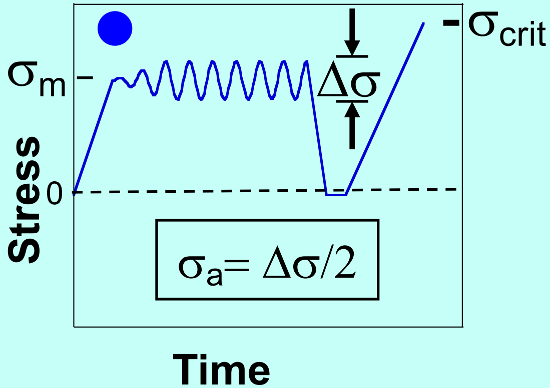
Fatigue of notched polysilicon: applications include MEMS resonators (that have been developed to replace quartz)

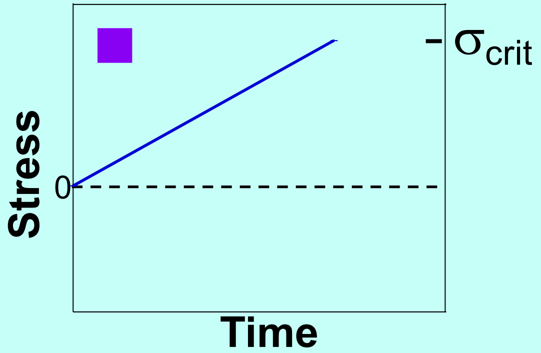




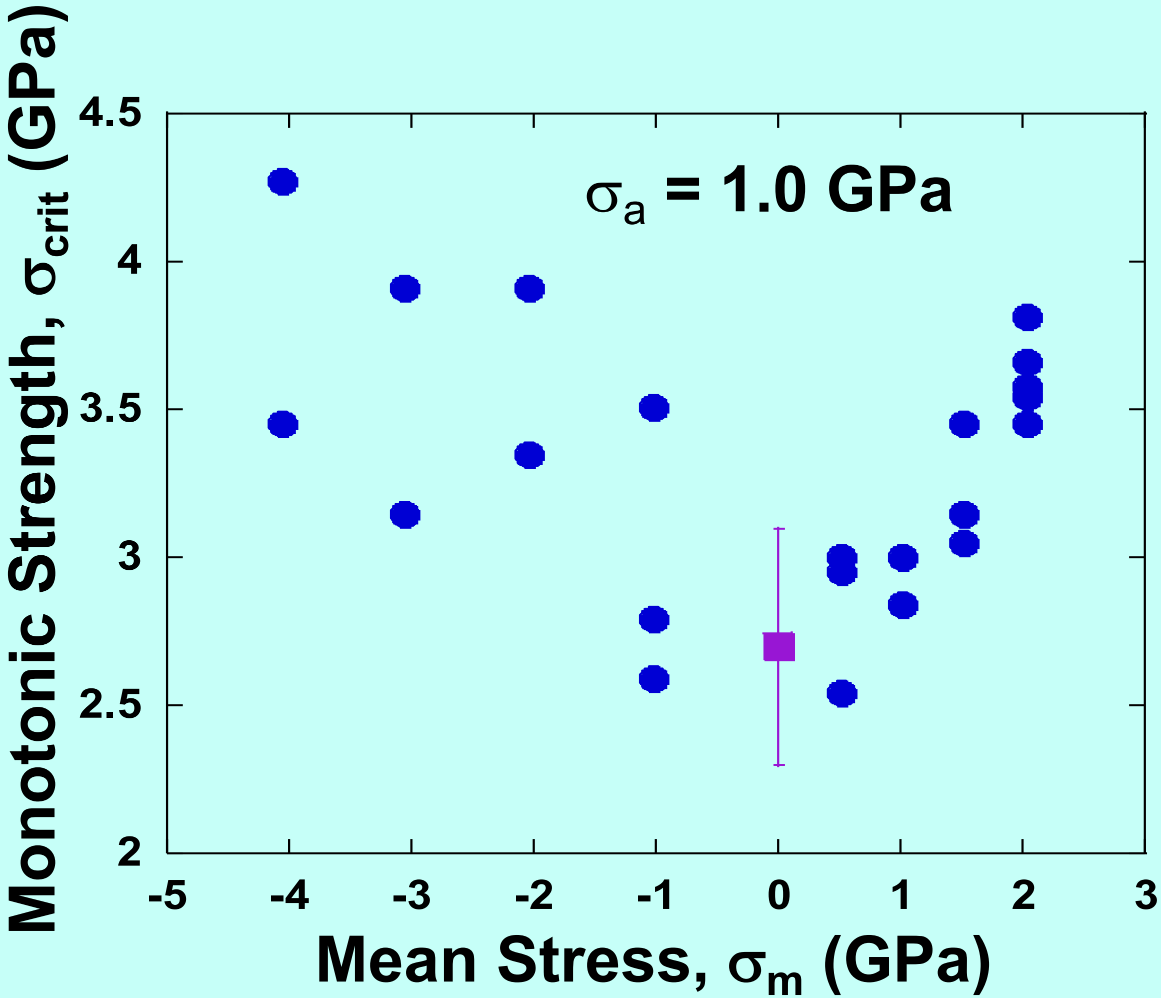
High amplitude: weakening

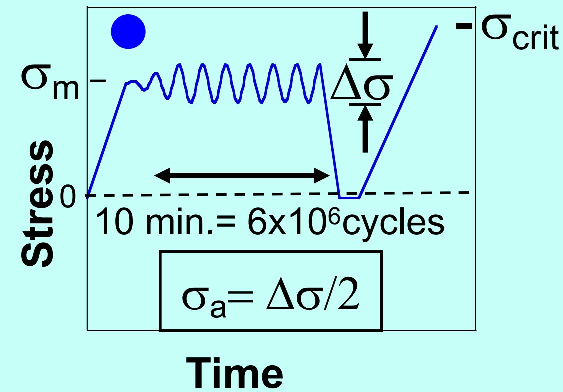


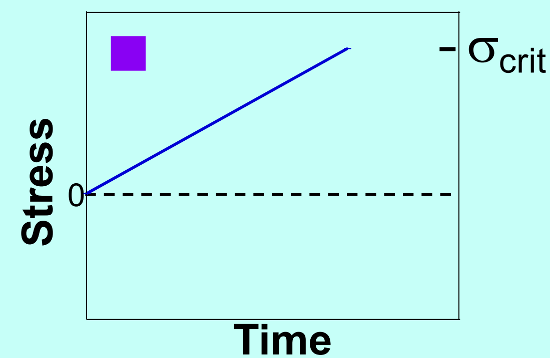




Low amplitude: increased strength







Experimental evidence of silicon's ability to deform plastically at room temperature

Crack

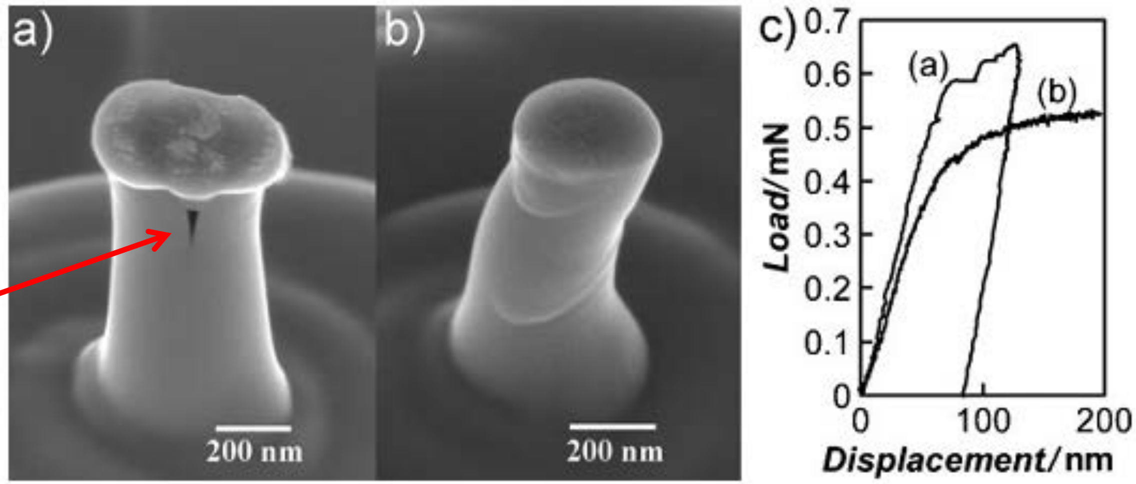
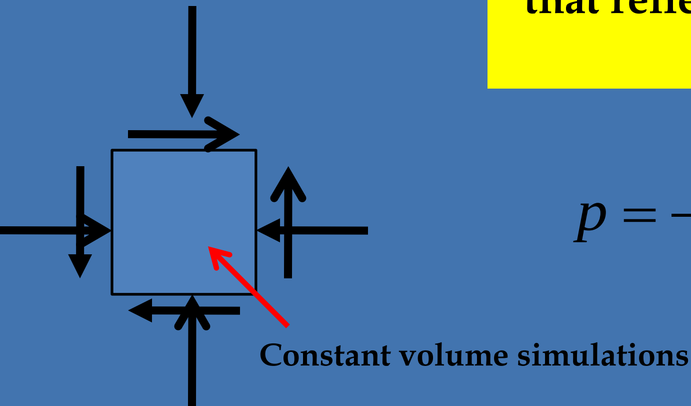


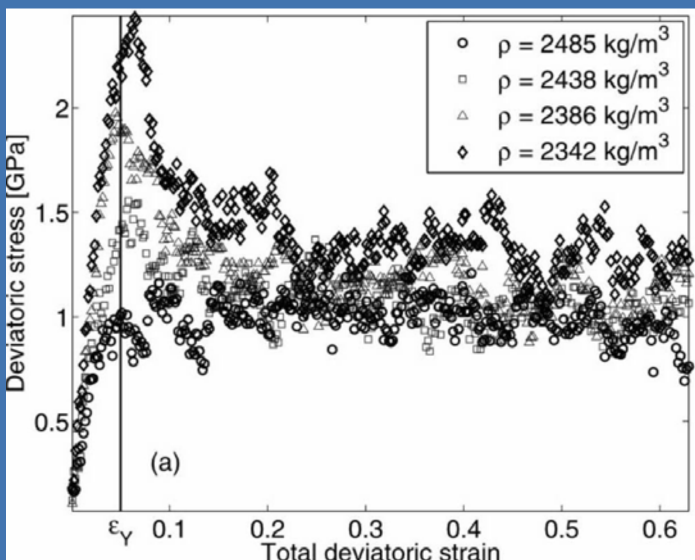
Figure 3. (a) and (b) shows HR-SEM images of two compressed silicon pillars at 45° tilt. The diameter of the pillars were 400 and 310 nm respectively. (a) was compressed with the MTS and (b) with the in situ setup. (c) shows load curves for the compression tests.

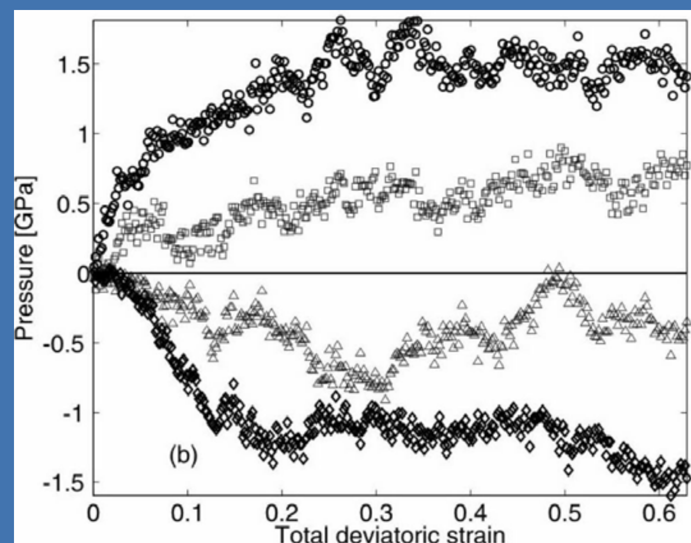
Advanced Functional Materials, 2009

Weakening still not fully understood.
But we have a plausible model for strengthening that relies on MD results for amorphous silicon that predict plasticity.



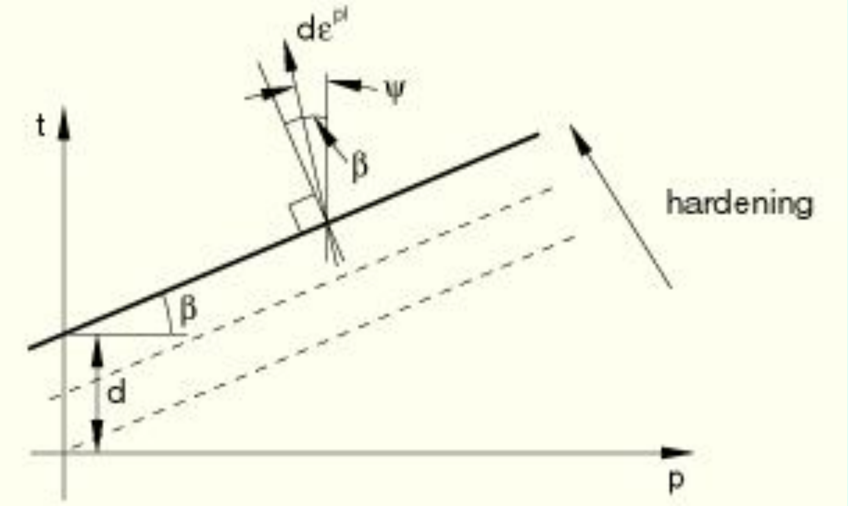
$$p = -\frac{1}{3}tr(\sigma), \quad s = \left| \sigma - \frac{1}{3}tr(\sigma)I \right|$$





M. J. Demkowicz and A. S. Argon

Uncoupled multiscale modeling



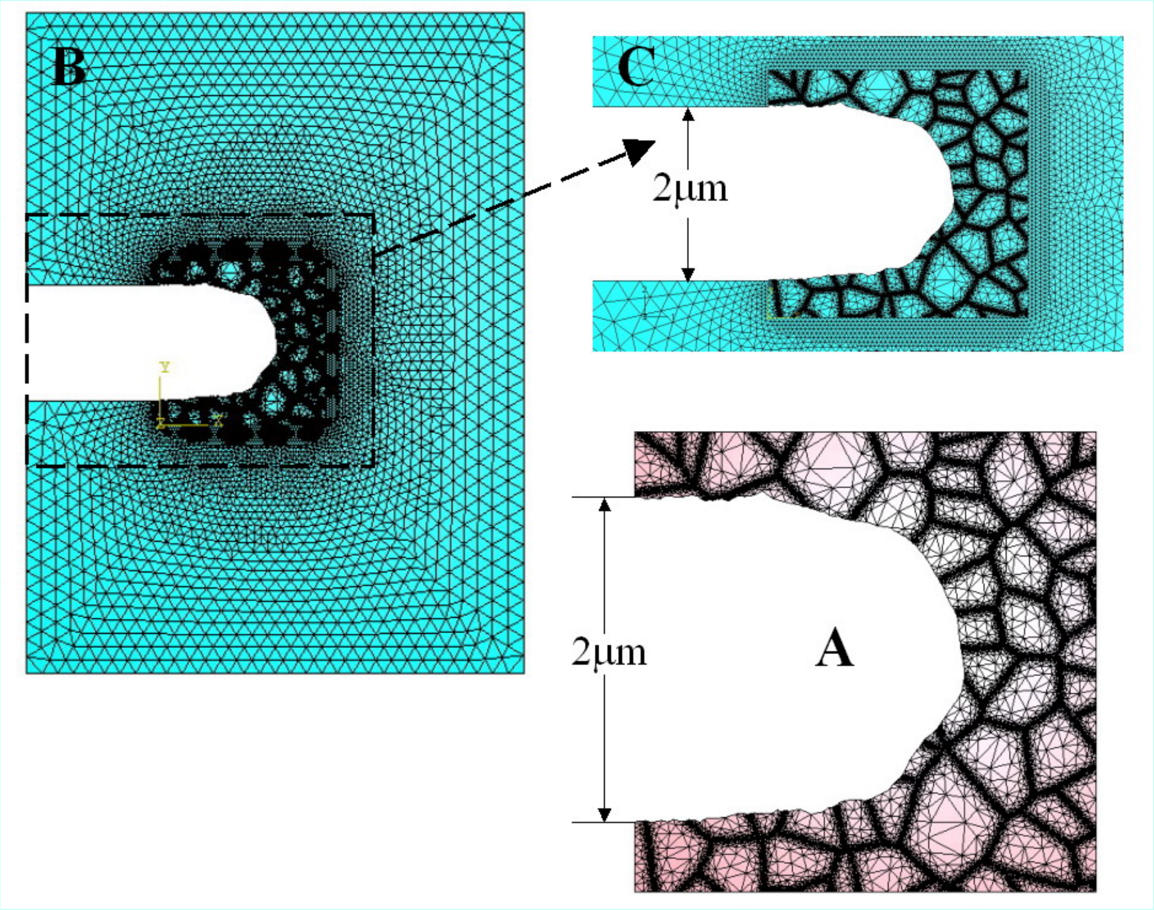
Drucker-Prager plasticity model

$$F = t - \sqrt{\frac{3}{2} s : s}\tan\beta - \sqrt{3}\tau = 0$$

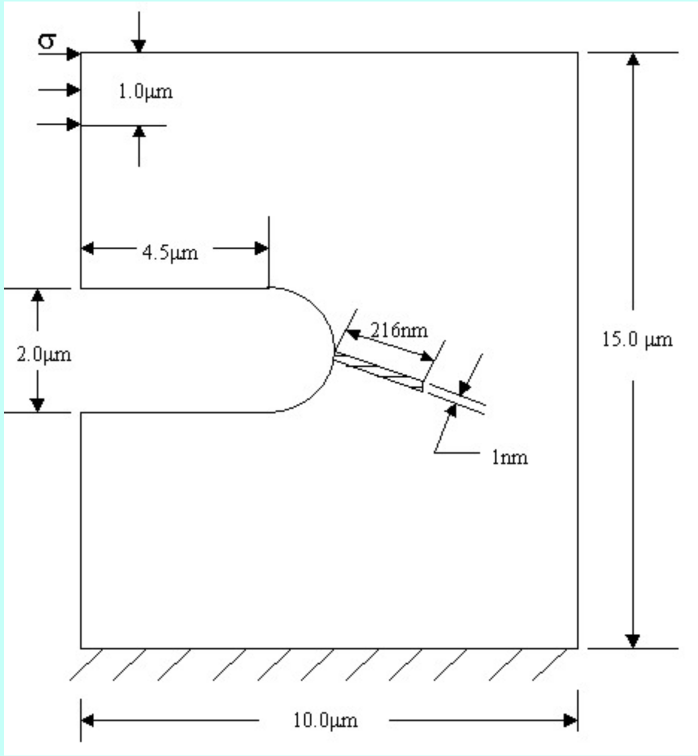
$$G = t - q\tan\psi$$

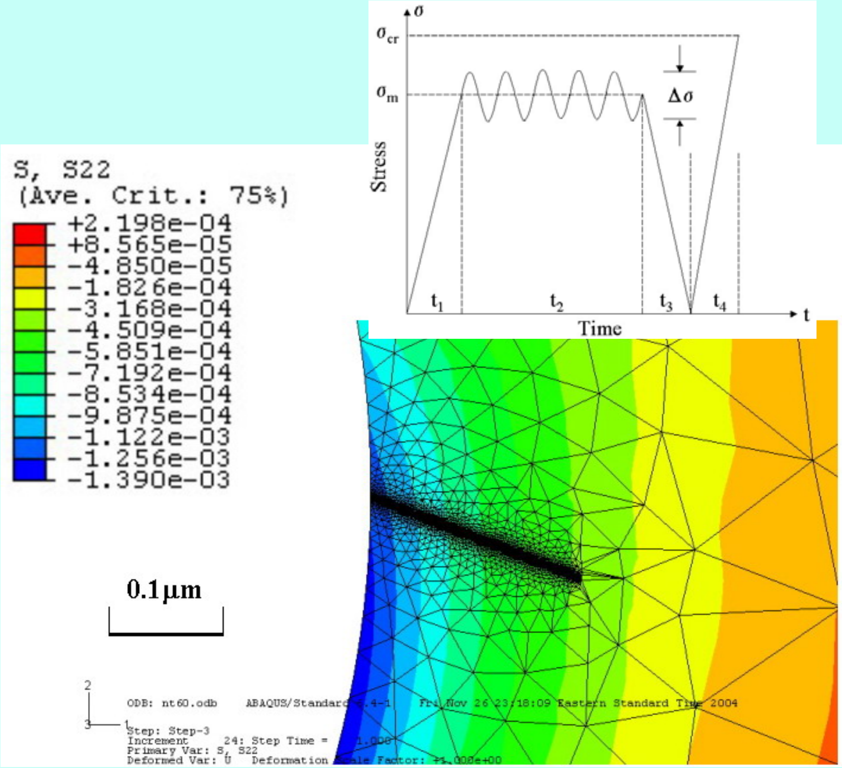
$$d\varepsilon^{p}{}_{ij} = d\lambda \frac{\partial G}{\partial \sigma_{ij}}$$

Poisson-Voronoi Local/Global Modeling



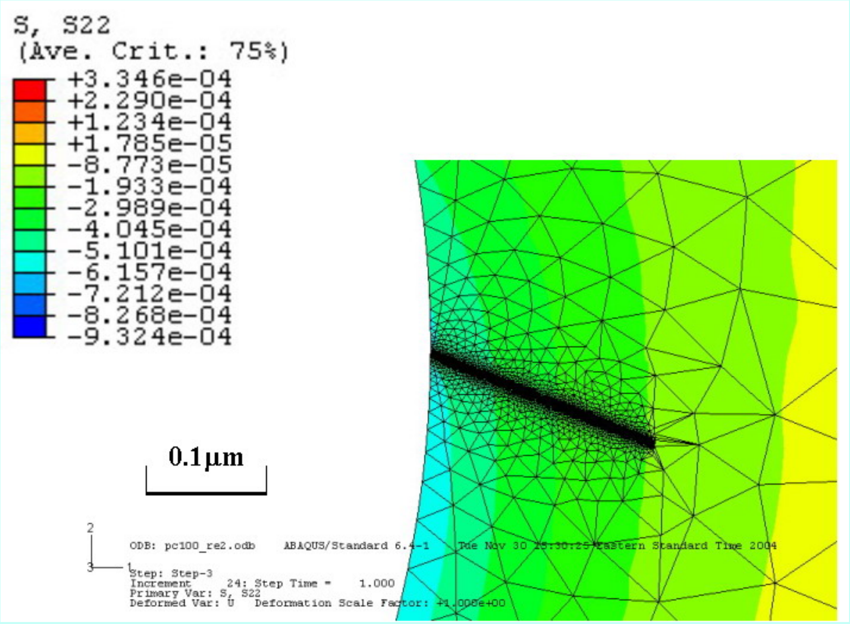
Model for large number of cycles subsequent to initiation





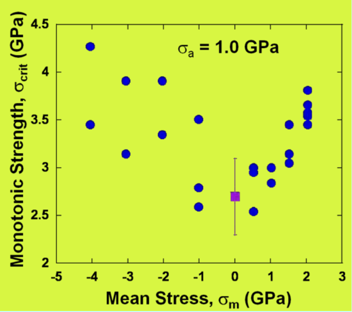
Low $\Delta\sigma$ and high σ_m (σ_m = 2.0GPa, $\Delta\sigma$ =2.0GPa)

Residual compressive stress ~1.4 GPa after 1000 cycles



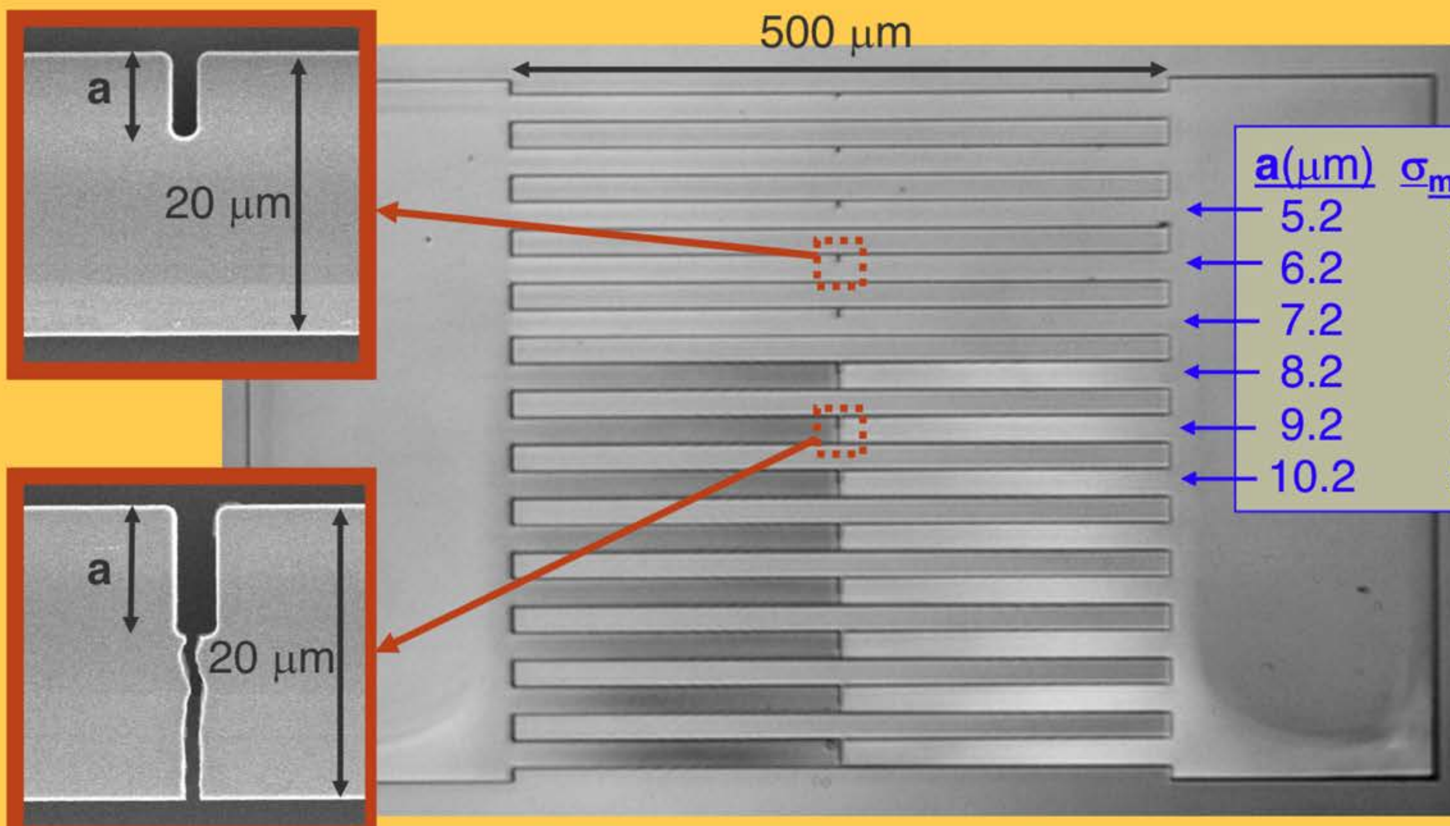
Low $\Delta\sigma$ and high σ_m (σ_m = -3.5 GPa, $\Delta\sigma$ =2.0GPa)

Residual compressive stress ~0.9 GPa after 1000 cycles



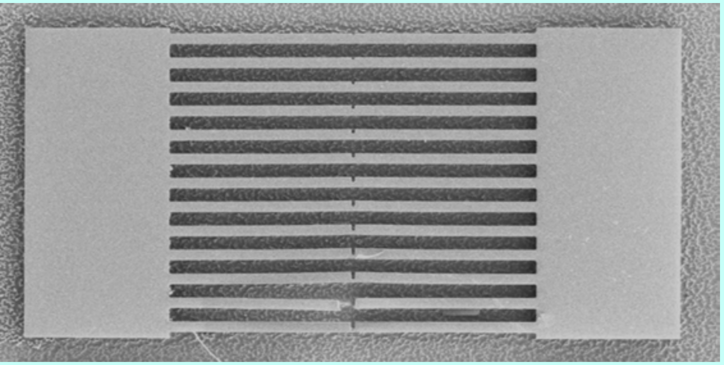
2. Passive devices for strength, fracture toughness, and static fatigue studies (go-no go tests).

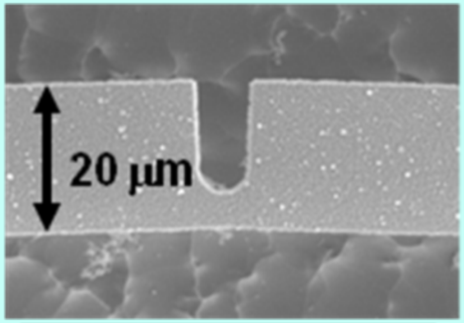
Undoped LPCVD Polysilicon
deposited at 570°C, annealed at 615°C → 318 MPa (Tensile)

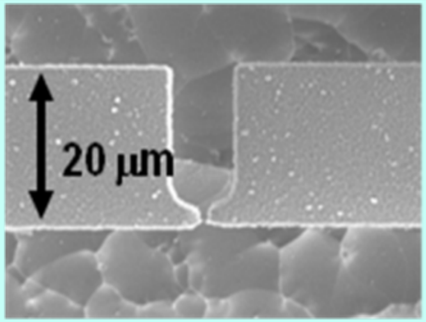


a(µm)	σ_{max}(GPa)	broken
5.2	2.3	0/14
6.2	2.8	0/14
7.2	3.4	0/14
8.2	3.9	11/14
9.2	4.5	14/14
10.2	5.0	14/14

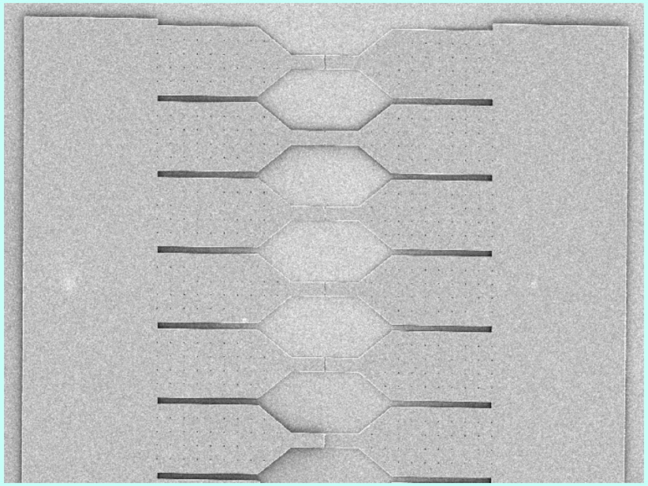
After 200 hrs in 90% humidity → no additional beams broke

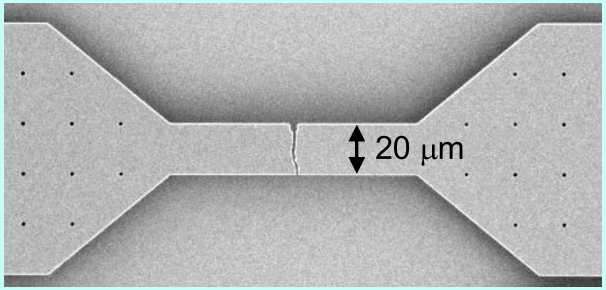




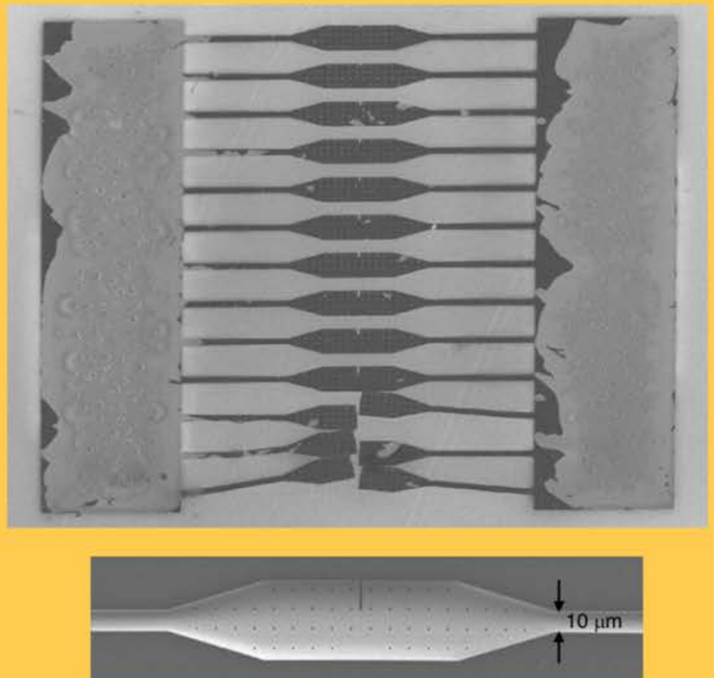


Sputtered Aluminum

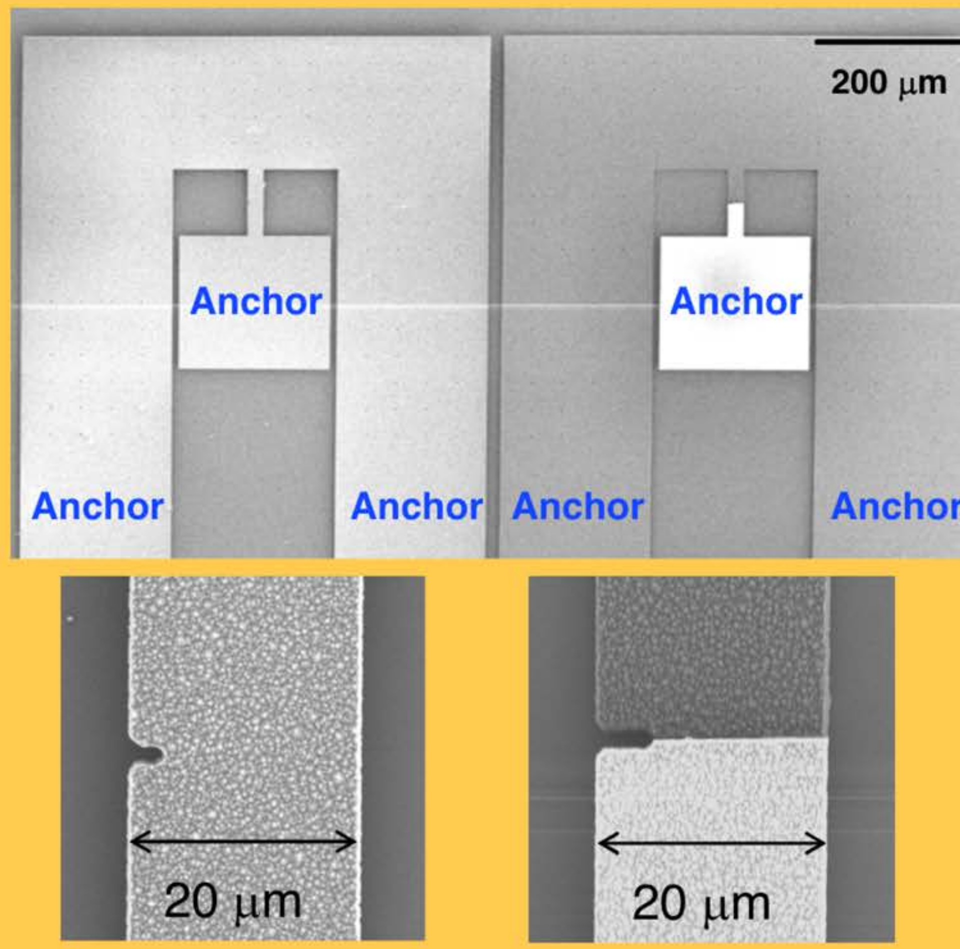




PolySi annealed to a small residual tension



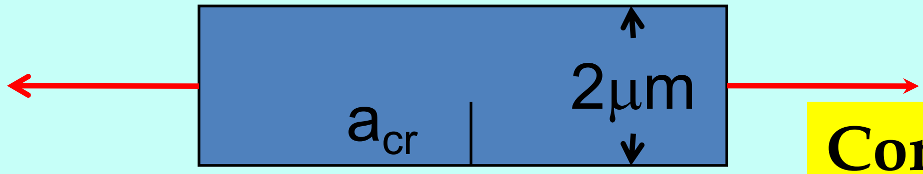
Large Residual Tension
Silicon Nitride



Residual Compression
Columnar Polysilicon

Static fatigue testing of polycrystalline Si and SiC

Why study environmentally assisted cracking in MEMS



Constant force

Say a_{cr}=1μm

Say t_{life}=10yrs

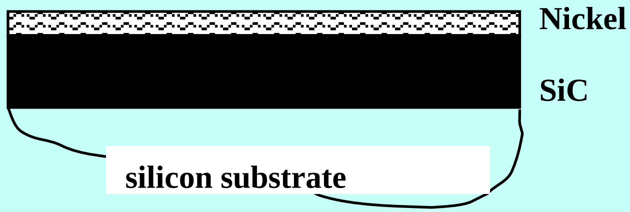
Then v_{crack} should be <10^{-15} m/s !!!

???

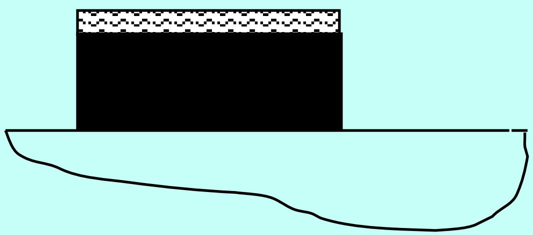
How can we run this test on MEMS?

Static fatigue testing of polycrystalline Si and SiC

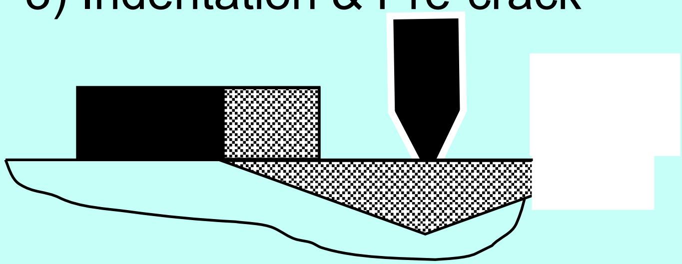
1) APCVD Deposition (1050 C)



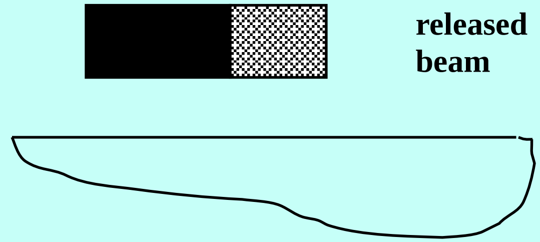
2) Reactive Ion Etch

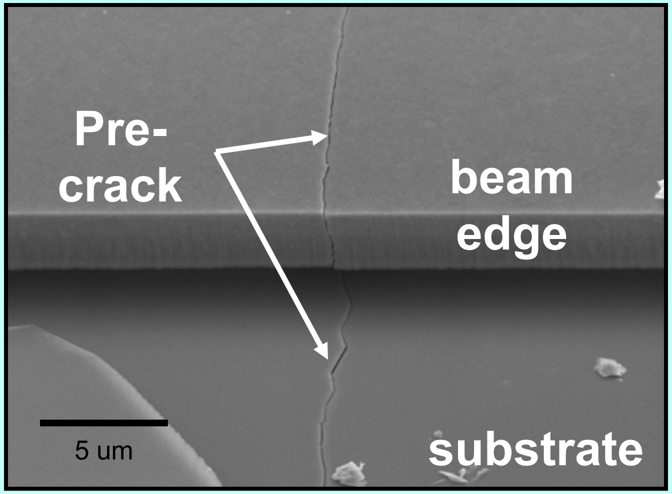


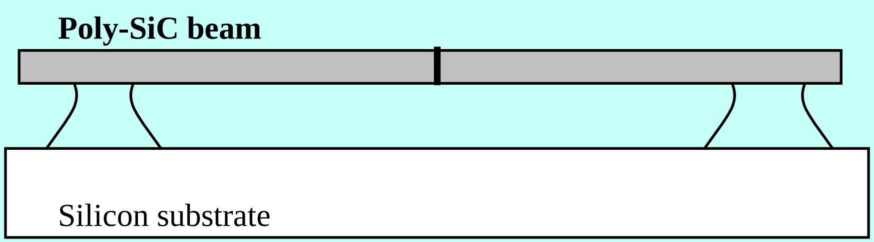
3) Indentation & Pre-crack

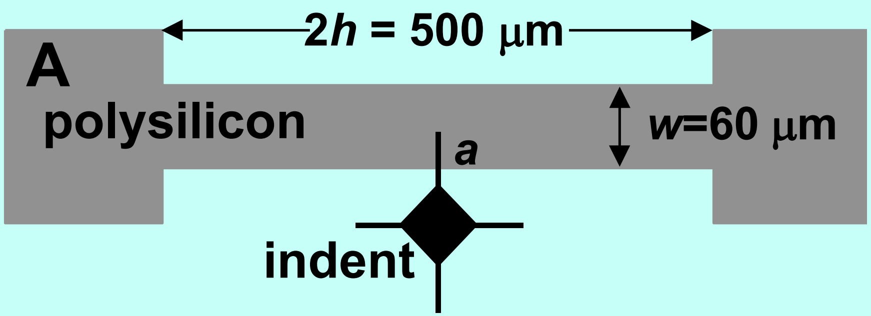


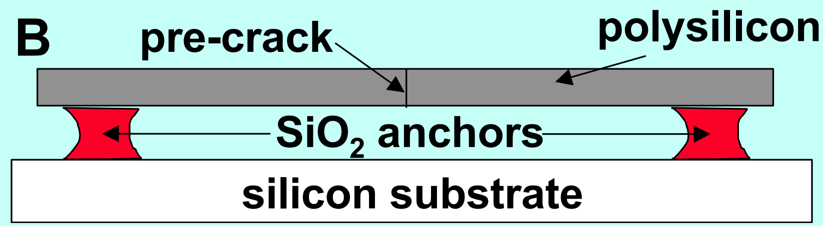
4) Device Release (substrate as release layer)

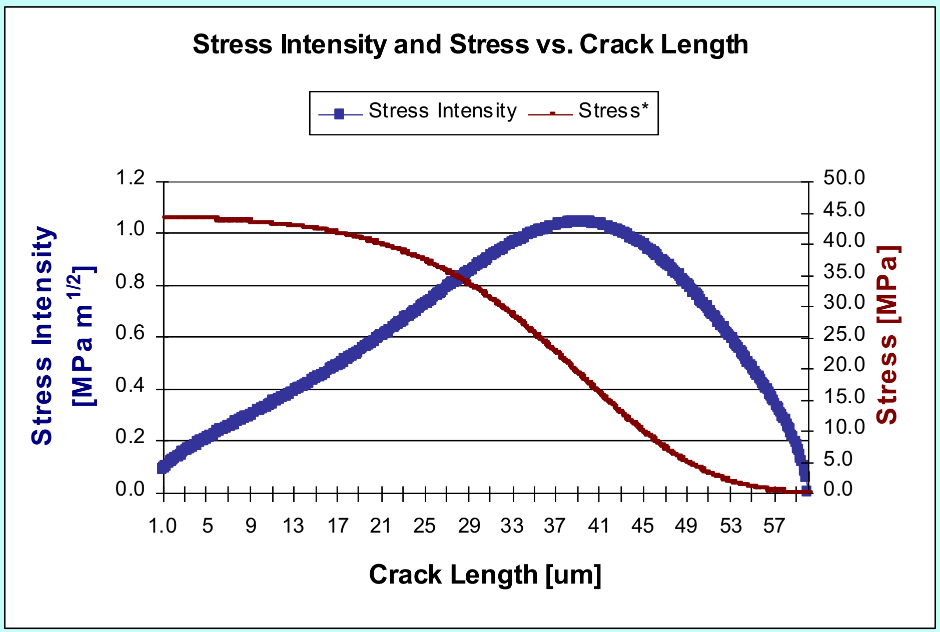


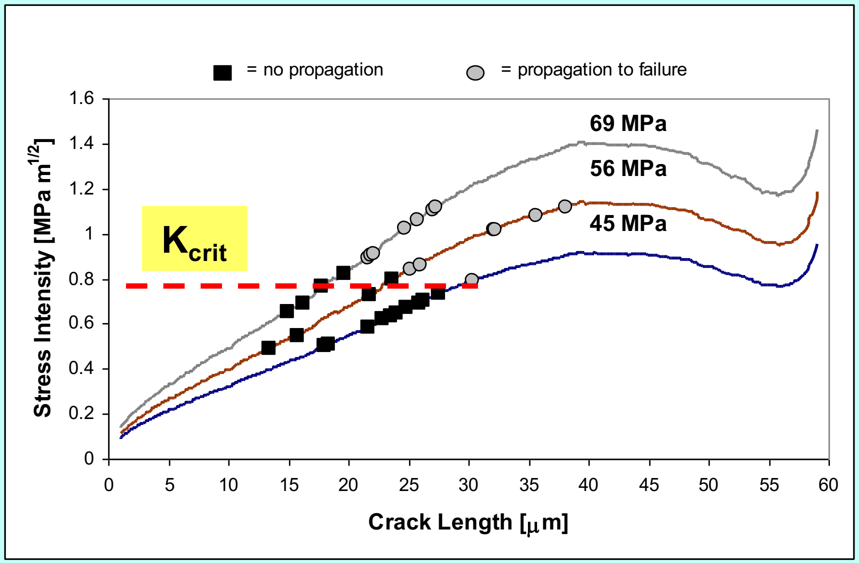




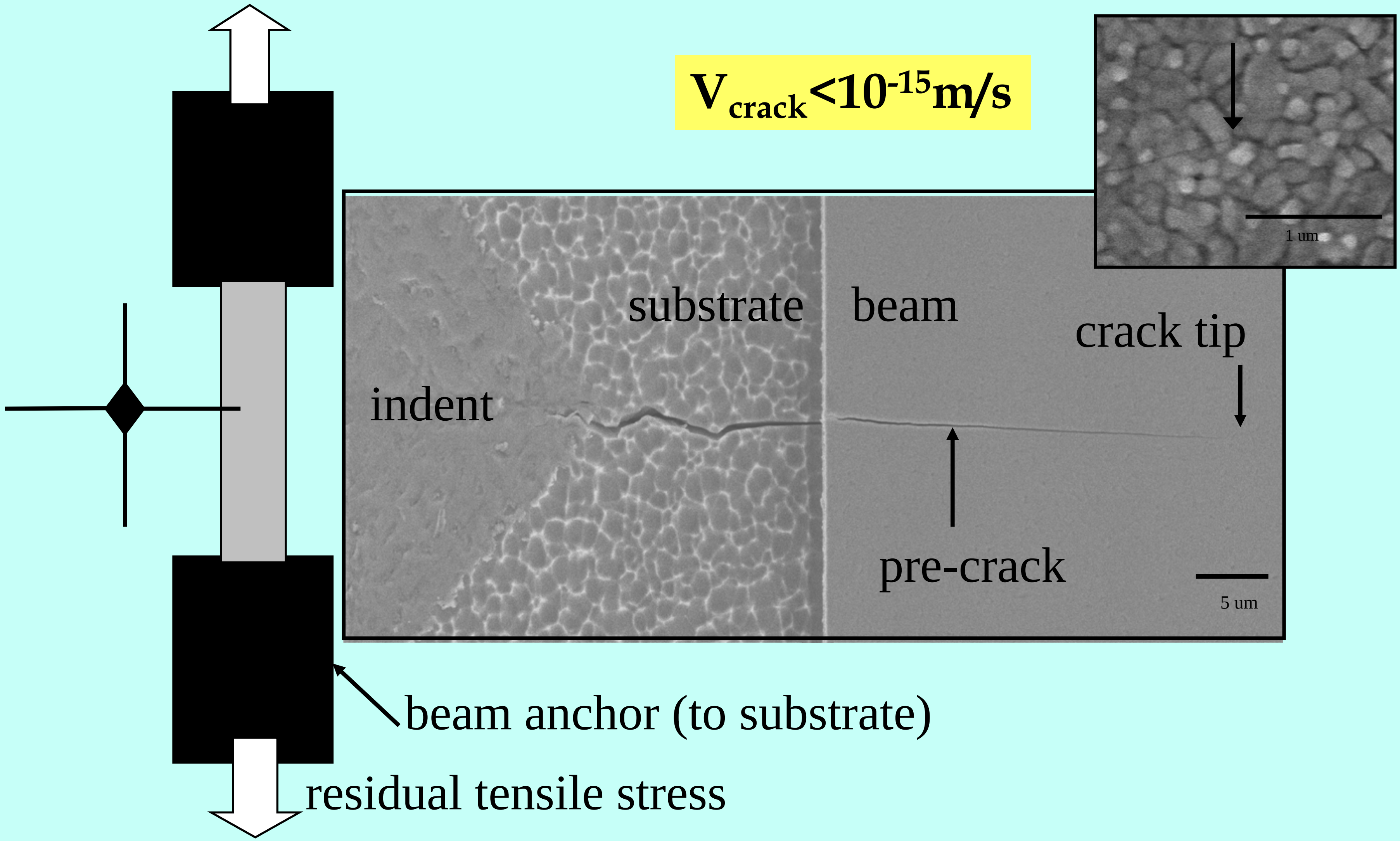








***Science*, 2002**


$V_{crack} < 10^{-15} m/s$
1 um
substrate
beam
crack tip
indent
pre-crack
5 um
beam anchor (to substrate)
residual tensile stress

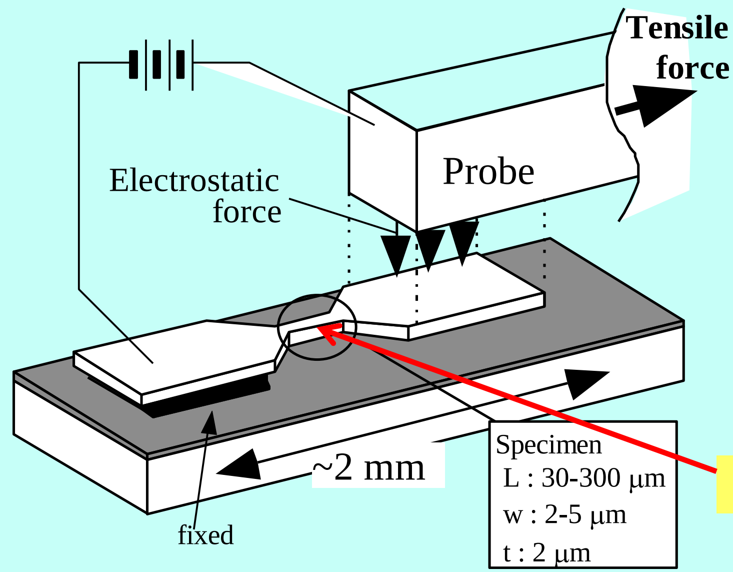
Reliability Issues

Example: Weibull approach to design of brittle MEMS

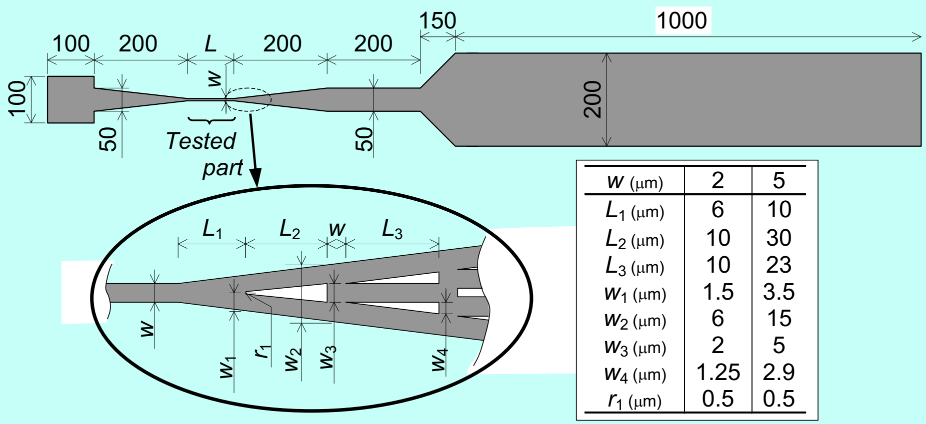
$$P_f = 1 - \exp\left[-\frac{\sigma^m}{\alpha} V_E \right] = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_\theta} \right)^m \right]$$

$$\frac{\mu}{\sigma_\theta} = \Gamma\left(\frac{1+m}{m} \right)$$

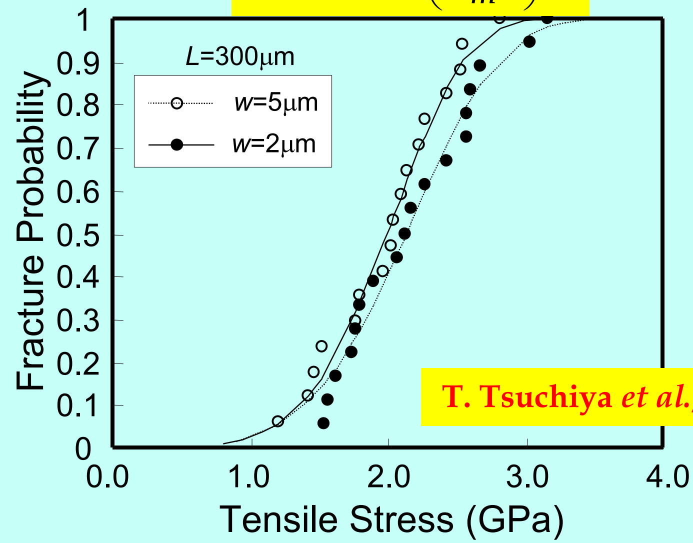
$$\frac{\sigma^{allow}}{\mu} = \frac{\left[-\ln(1-P_f) \right]^{1/m}}{\Gamma\left(\frac{1+m}{m} \right)}$$



Gauge region smaller than RVE

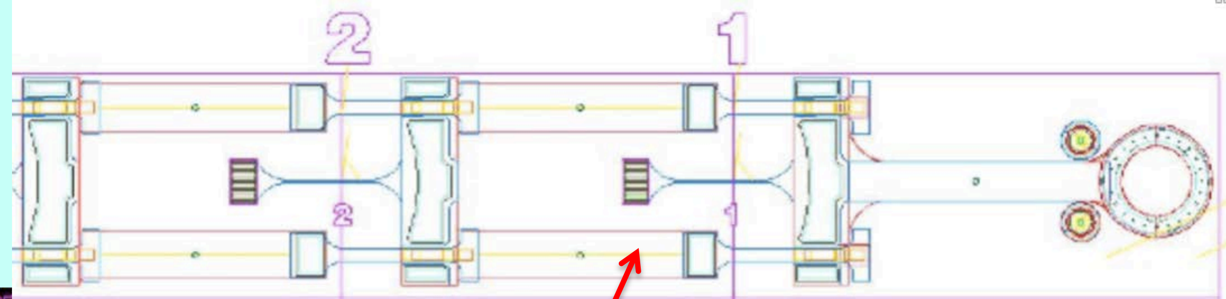


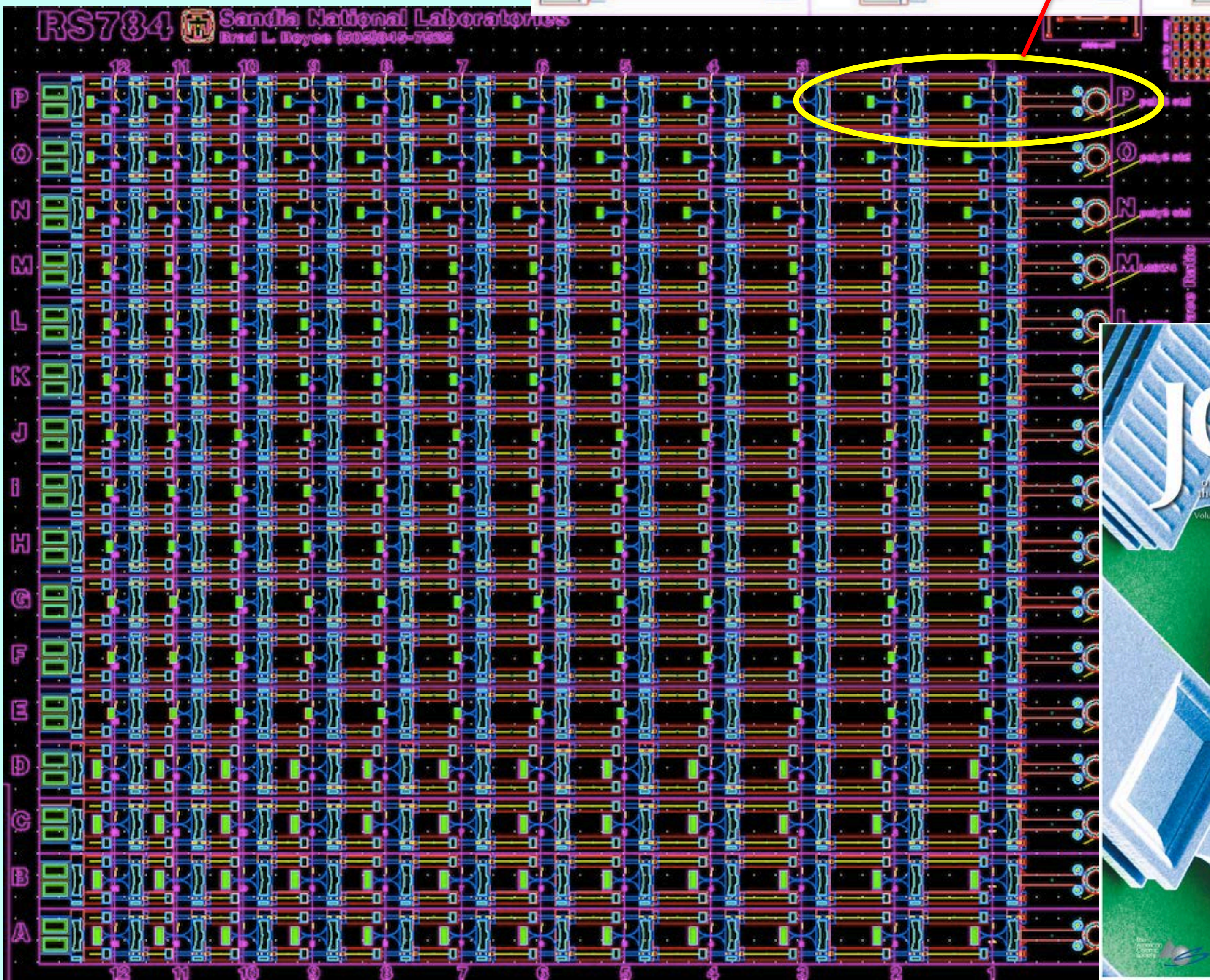
w (μm)	2	5
L_1 (μm)	6	10
L_2 (μm)	10	30
L_3 (μm)	10	23
w_1 (μm)	1.5	3.5
w_2 (μm)	6	15
w_3 (μm)	2	5
w_4 (μm)	1.25	2.9
r_1 (μm)	0.5	0.5

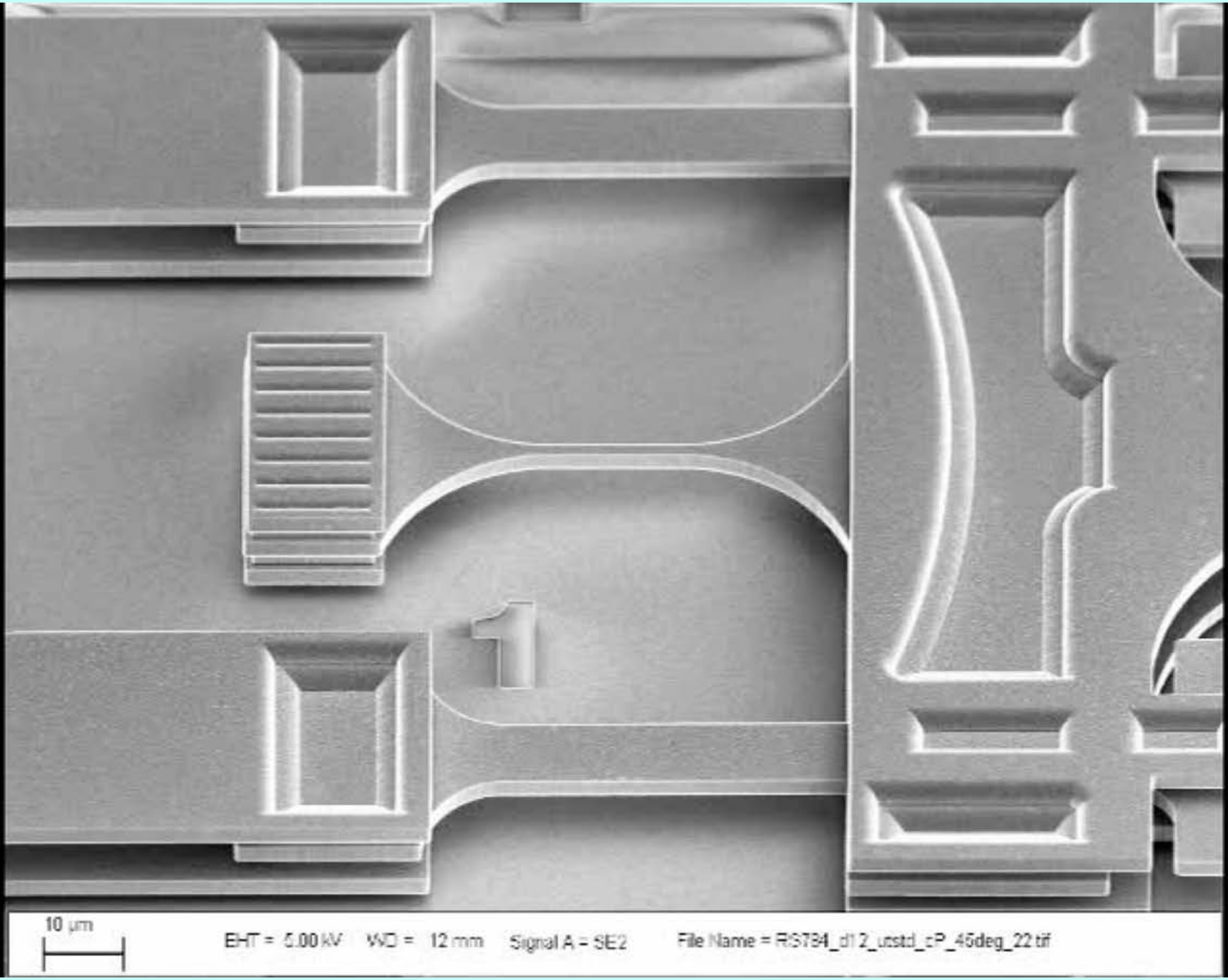


T. Tsuchiya *et al.*, 1998

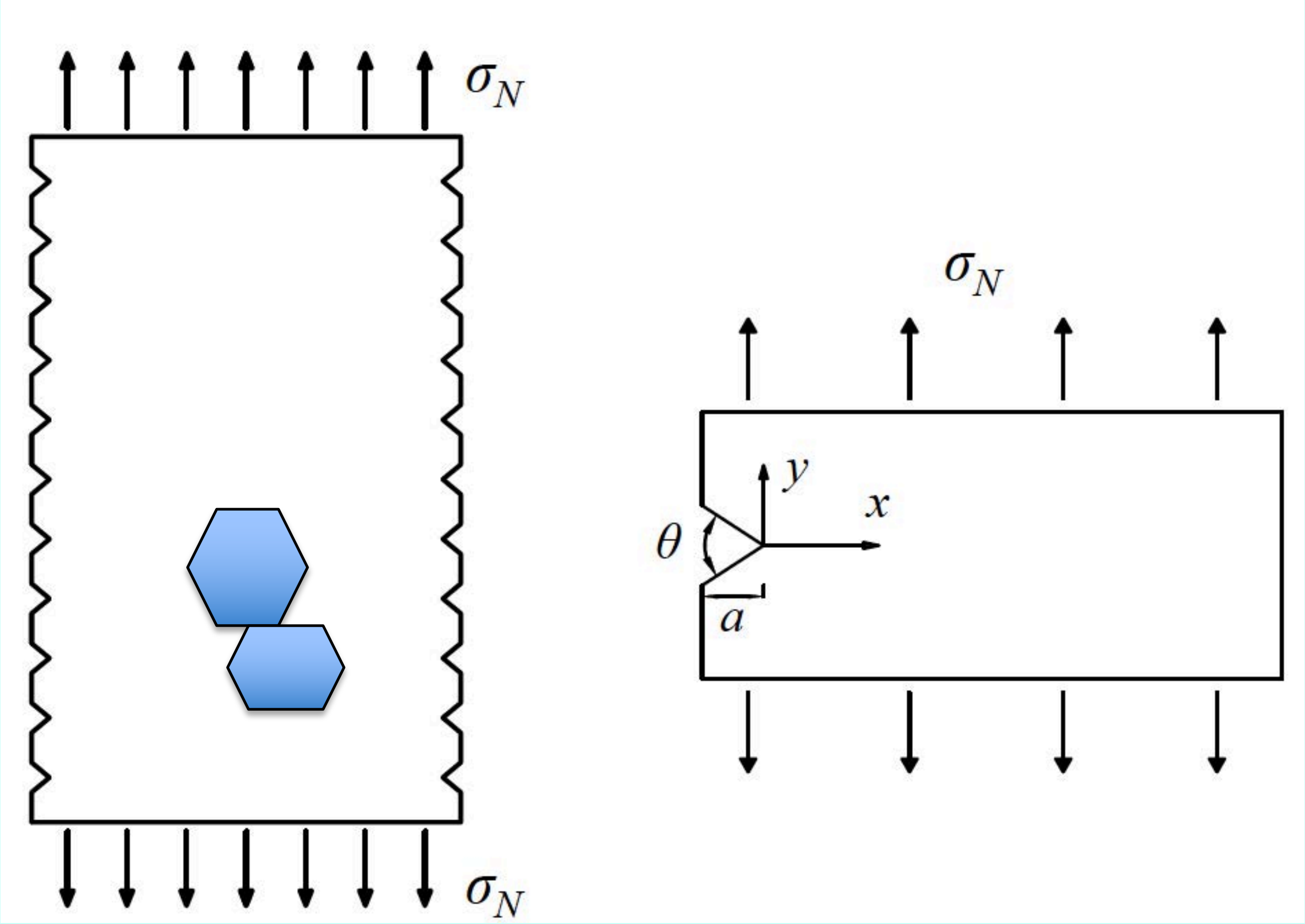
**Slack chain tester:
A way of obtaining lots of strength data**






1
10 µm
EHT = 5.00 kV WD = 12 mm Signal A = SE2 File Name = RS794_d12_utstd_cP_45deg_22.tif

Finite Weakest Link Model
(inherent size effect on distributions)



Feature Article

***J. American Ceramic Society*, June 2015**

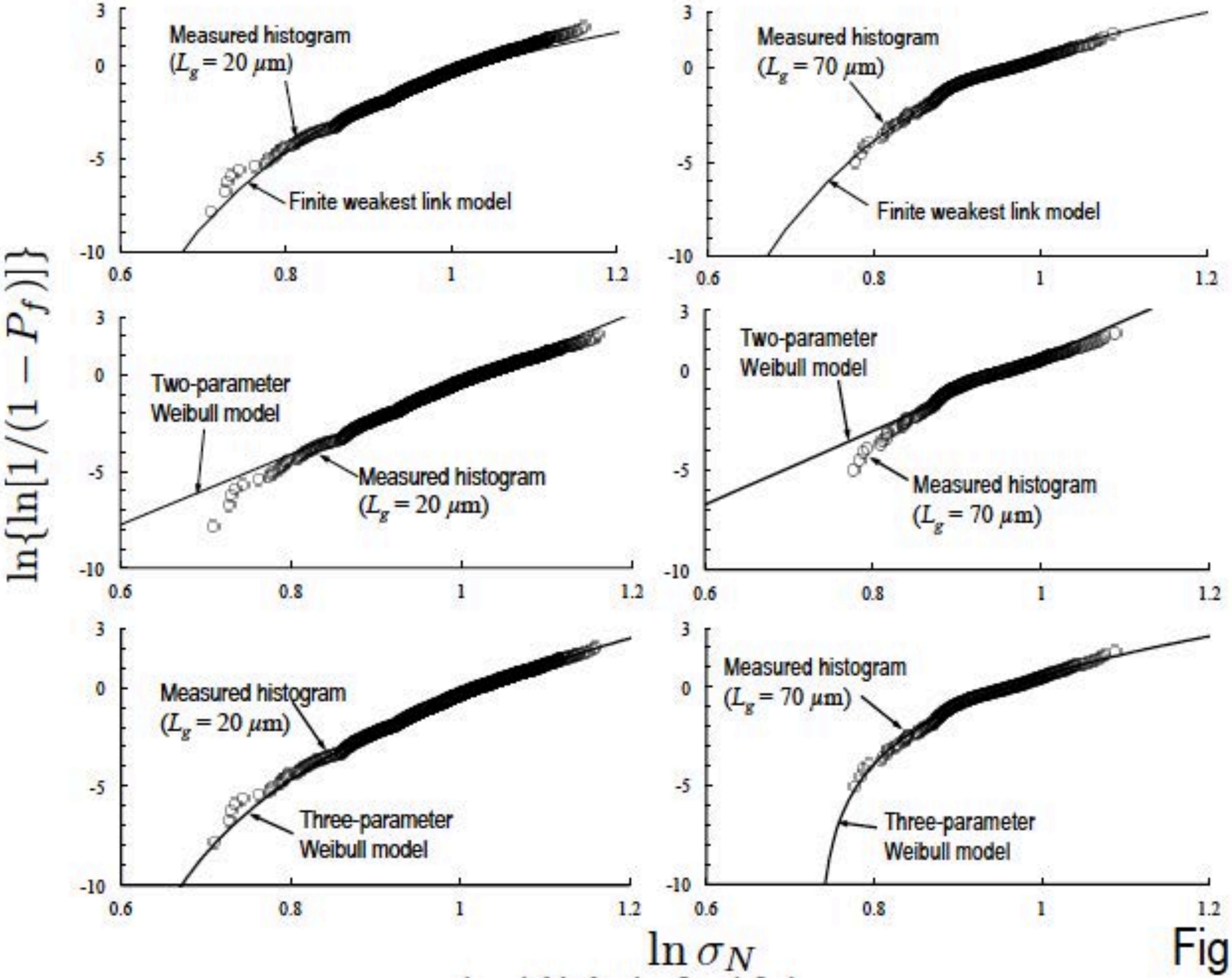
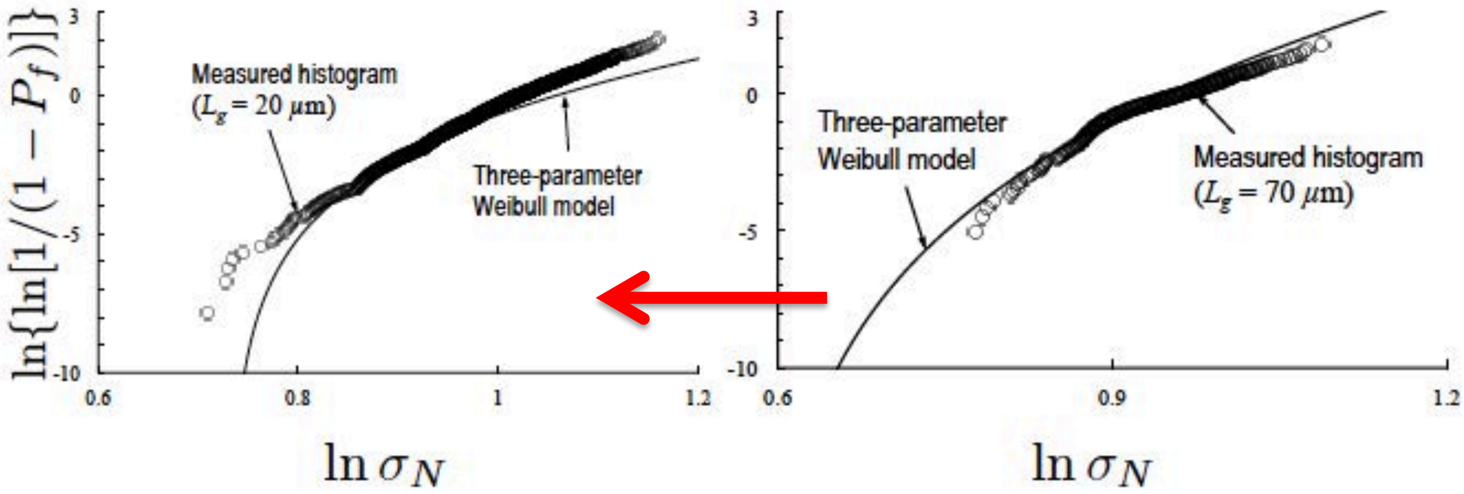
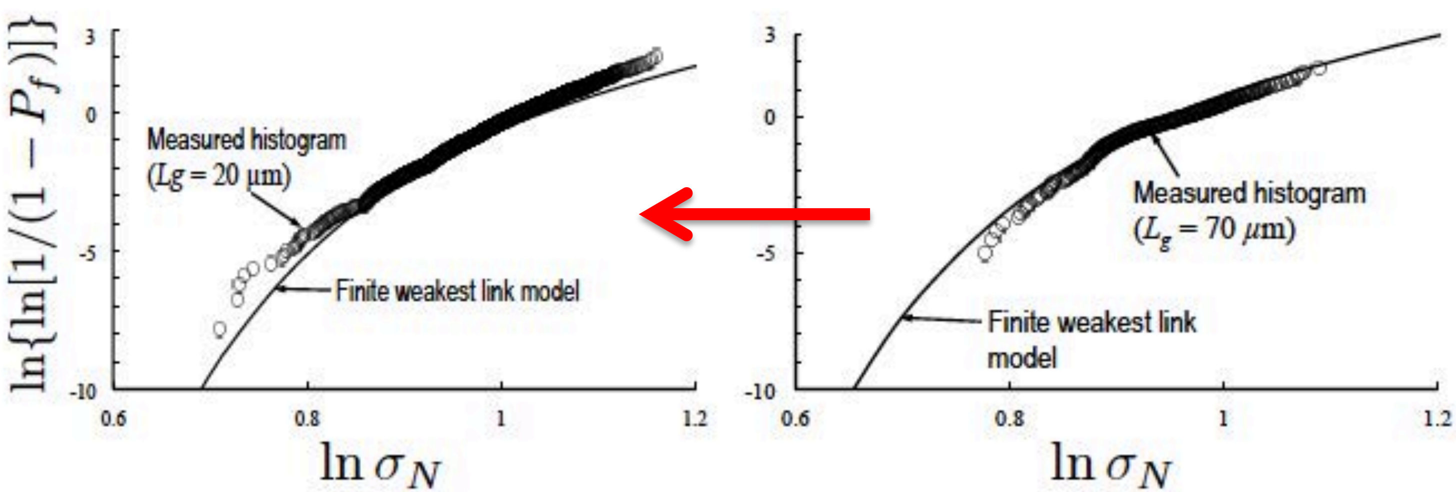


Fig. 5

Optimum Fits of Weibull Models and Finite Weakest Link Model
Data courtesy of Brad Boyce (Sandia National Labs



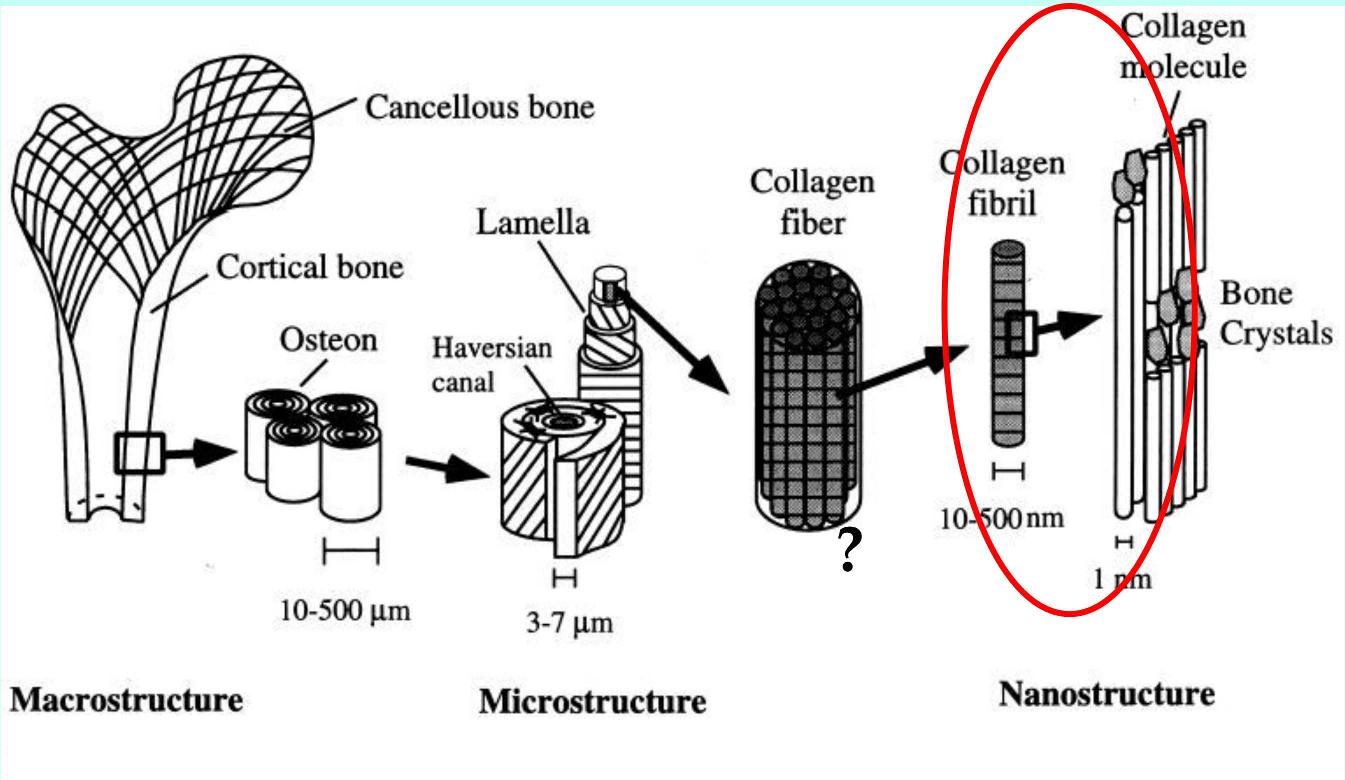
Calibrate with one set and predict other



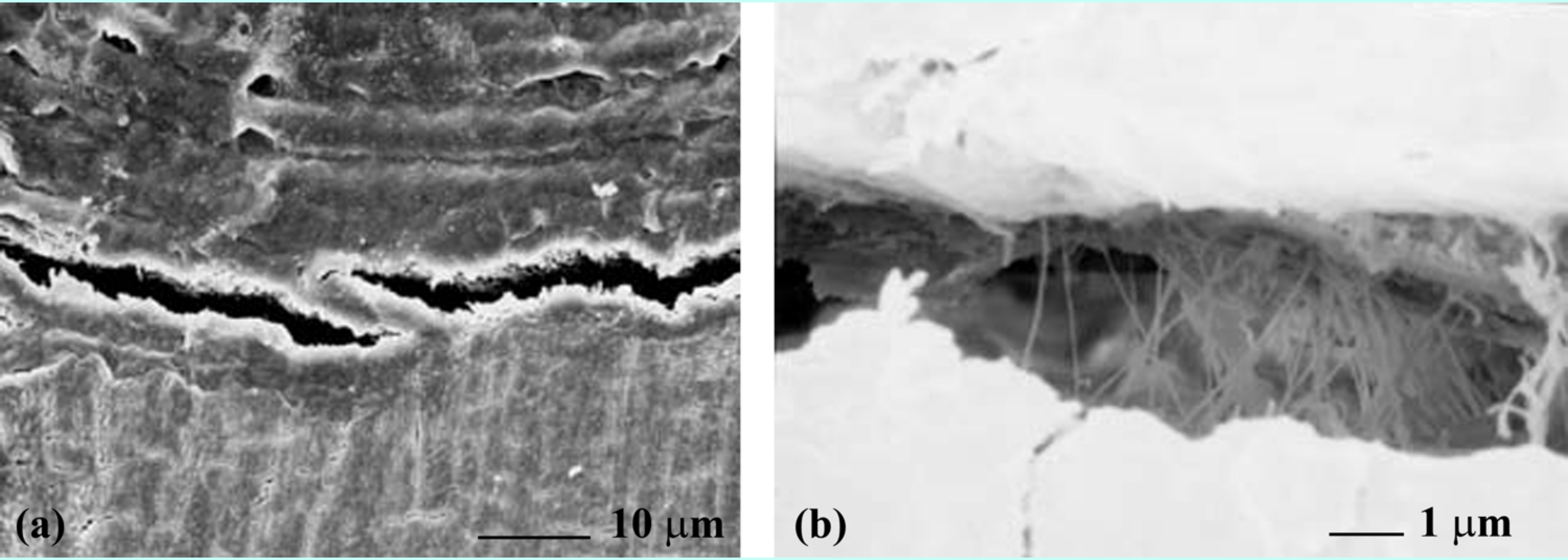
MEMS devices to test nanoscale structures

Collagen fibrils
Carbon nanotubes

Why?
1. What are the origins of the toughness of bone and other tissues?
2. Development of multiscale models



J.Y. Rho, et al., 1998



Crack bridging mechanisms
(Nalla *et al.* 2005)

Available material

Type-I collagen fibrils isolated from sea cucumber - tens of μm long with diameters ~ 100-300 nm

Estimate

Elastic modulus ~ 1 GPa

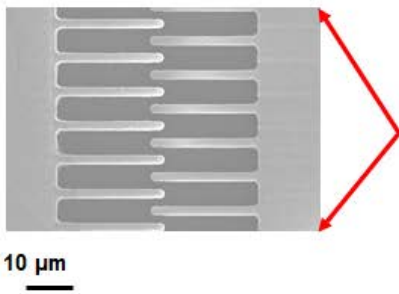
Strain to failure ~ 100%

Actuator

Force ~ 10s μN

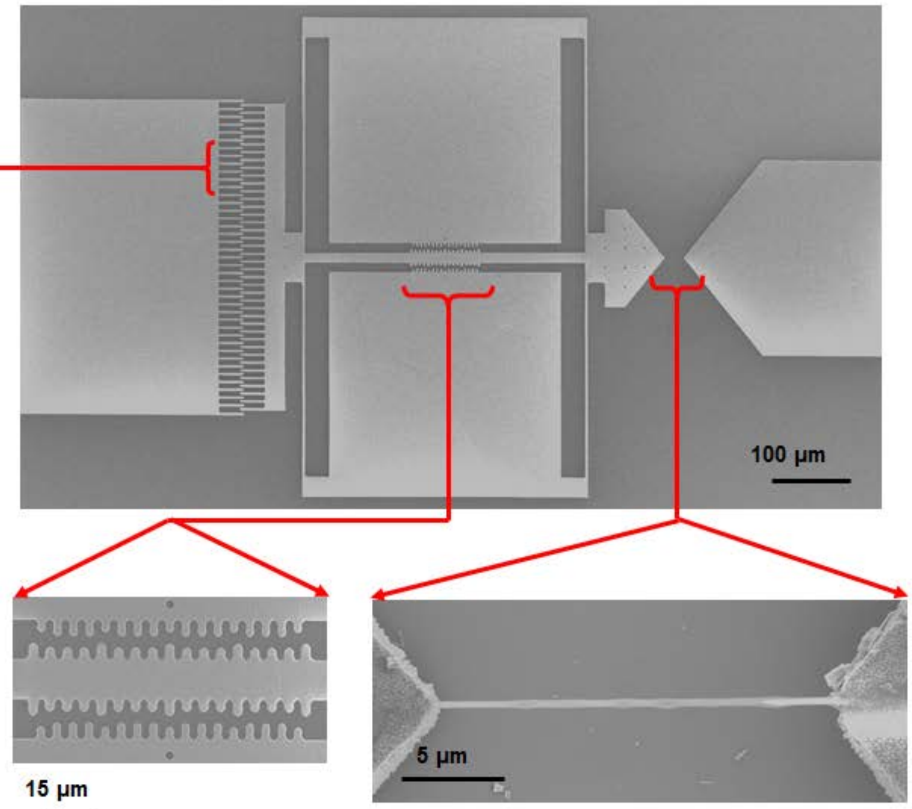
Stroke ~ 10 μm

First generation device (electrostatic actuation/Vernier scale)

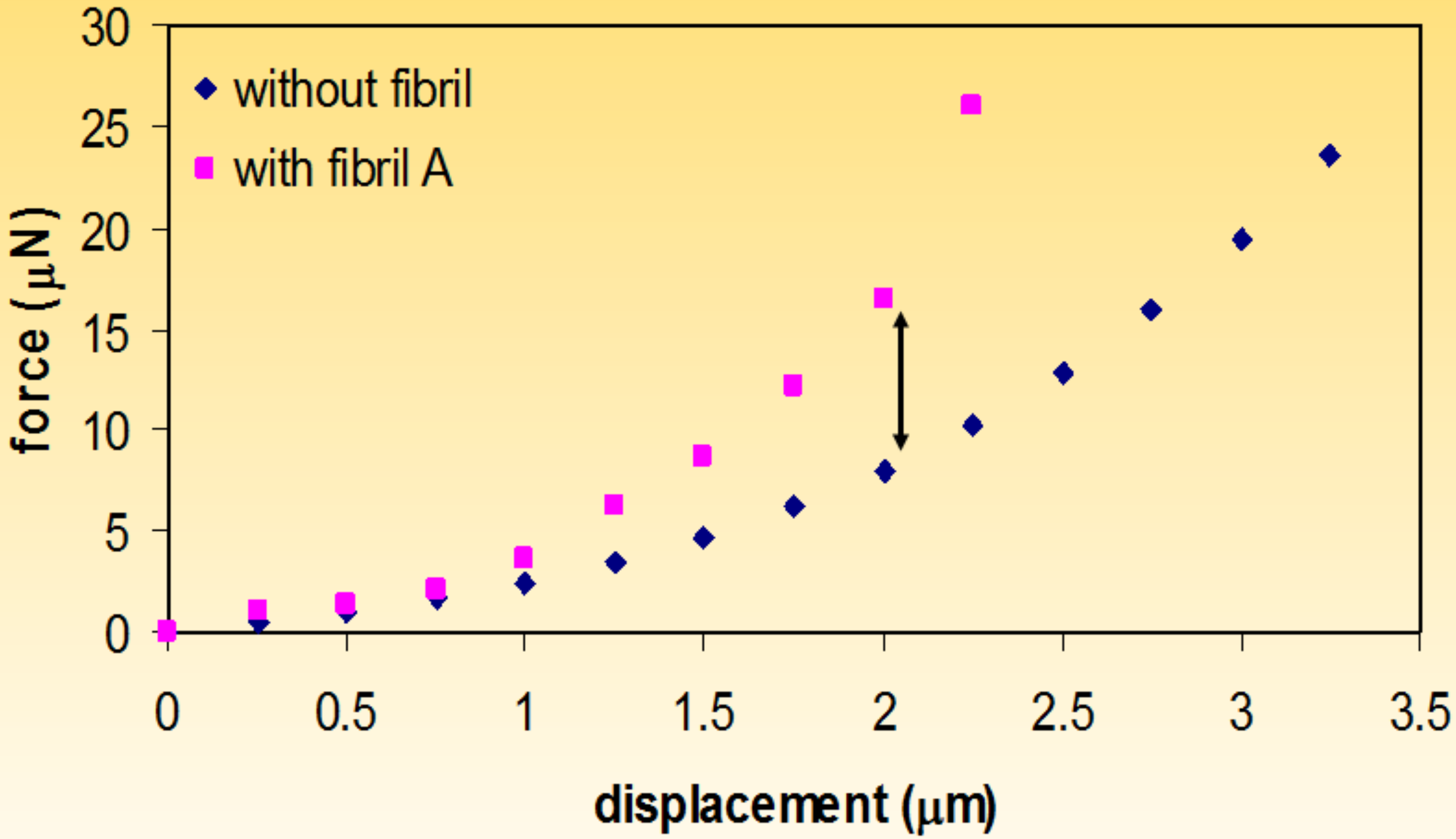


$$F = \frac{1}{2}\frac{\partial C}{\partial x}V^2 = n\varepsilon \frac{h}{g}V^2$$

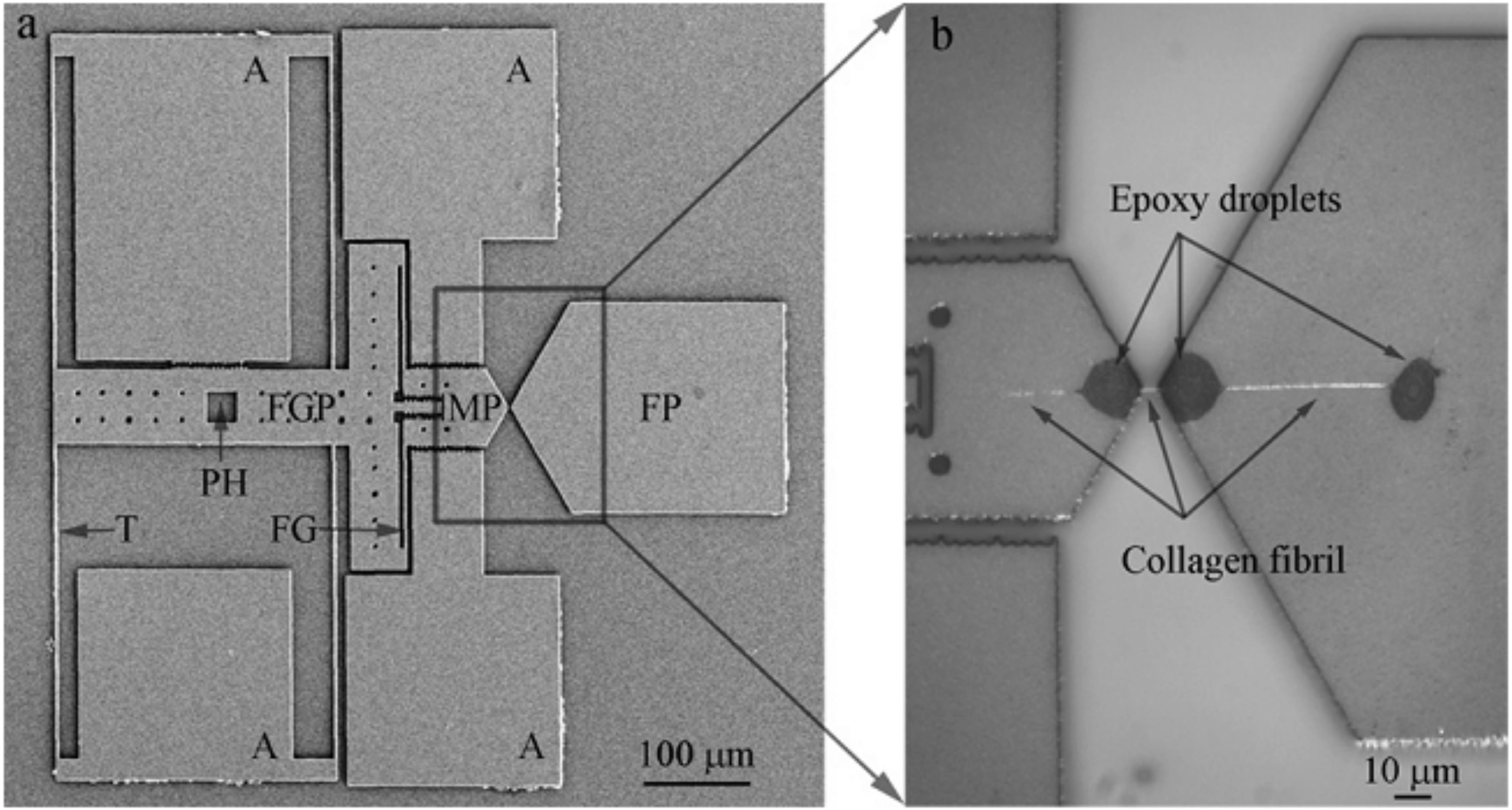
F: force
C: capacitance
x: distance
V: voltage
n: number of finger
ε: permittivity
h: height of the finger
g: gap between the fingers



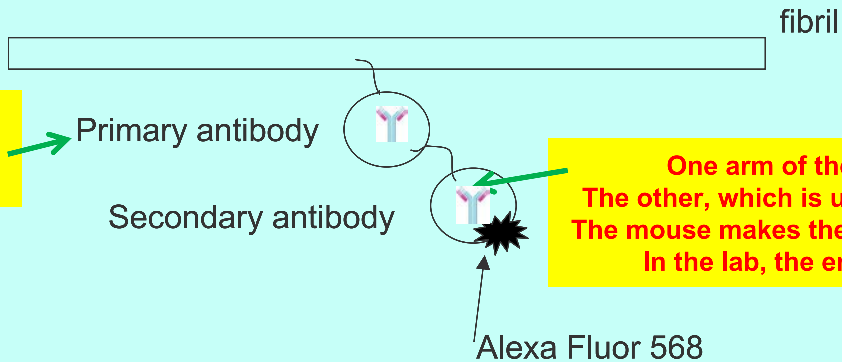
Results
Force-Displacement Curve



Second generation device: piezo-driven and DIC (54nm displacement, 50nN force sensitivities)

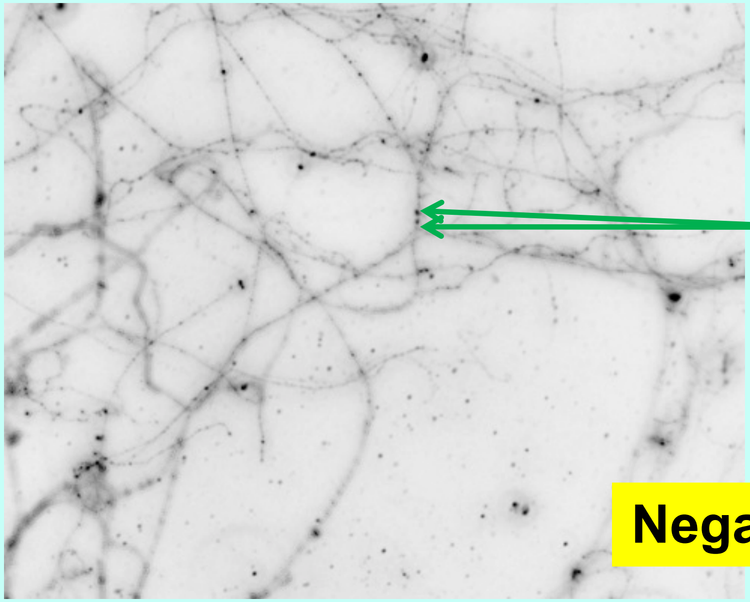


Current effort: fluorescently tagged antibodies in solution will be used as markers for imaging deformation



Collagen fibrils put in blood of a goat. Goat makes primary antibodies, which are in turn retrieved from blood.

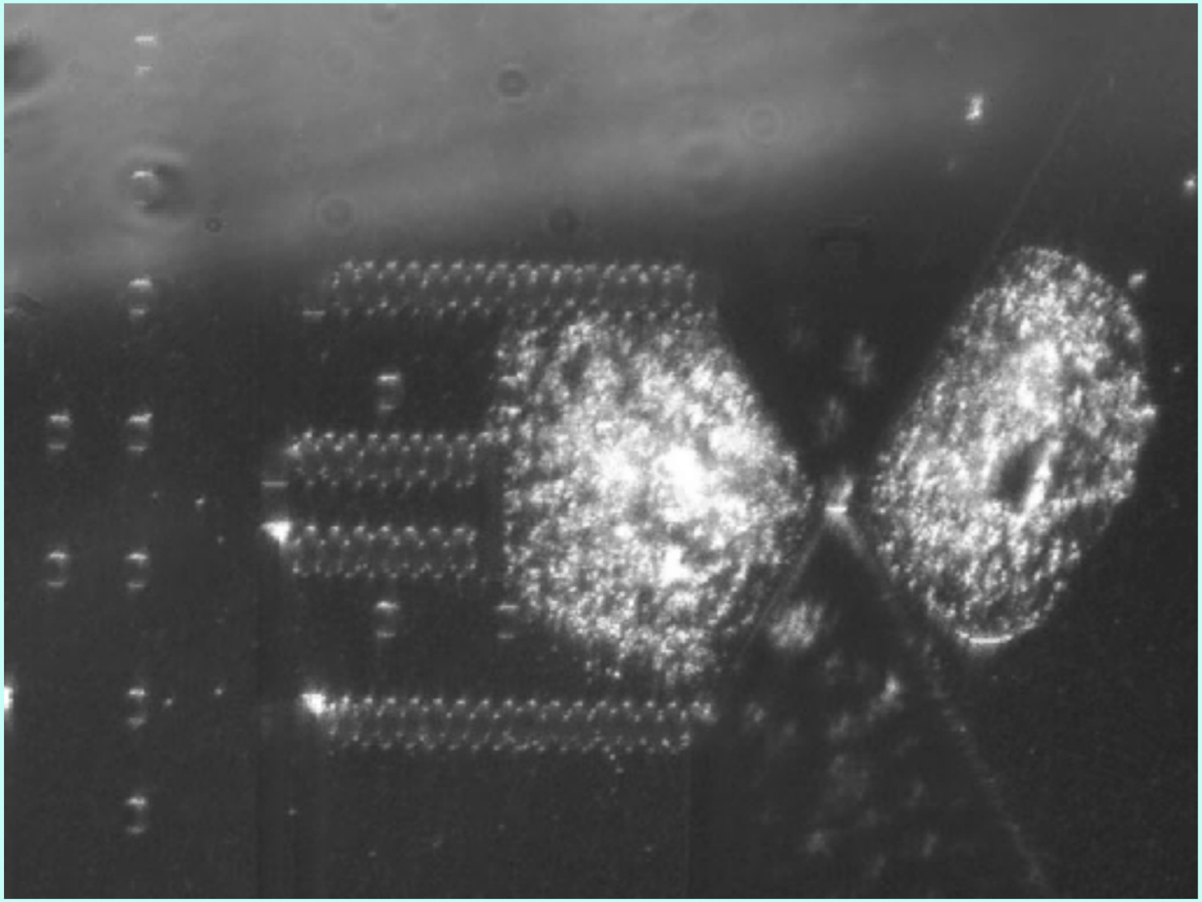
One arm of the primary's Y goes after the fibril. The other, which is universal, is cleaved and put in a mouse. The mouse makes the "mouse-anti-goat" secondary antibody. In the lab, the emitter is attached to the secondary.

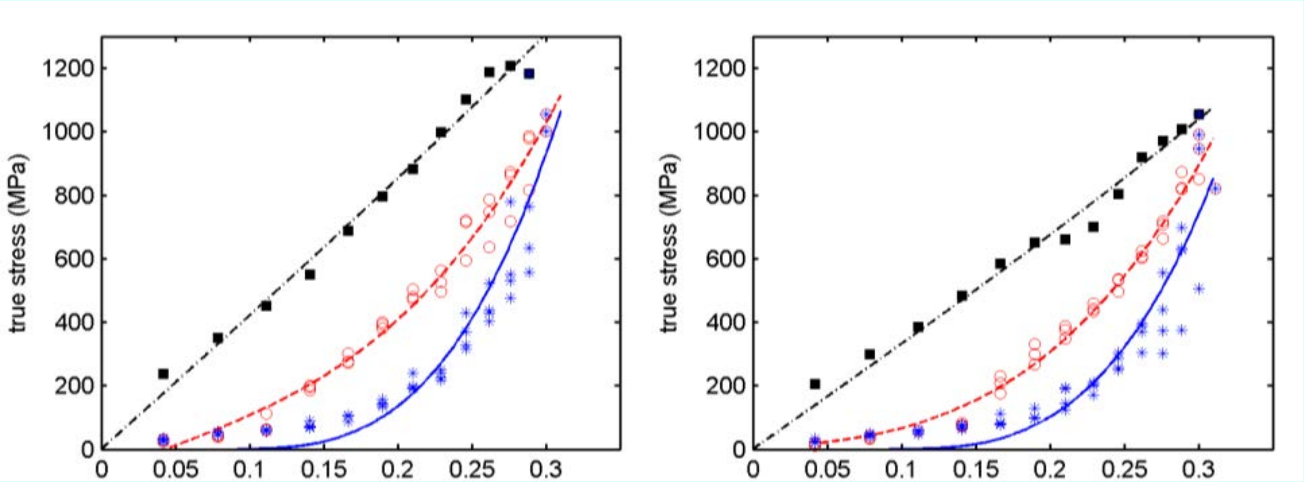


These will be used with DIC to measure strains.

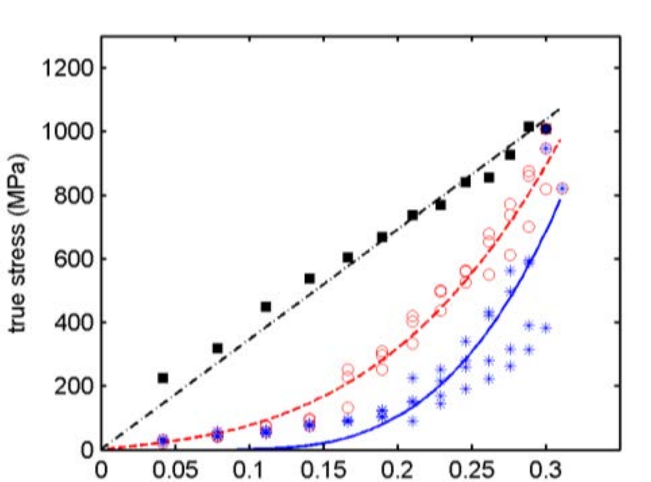
Negative image

Fibril breaking in air

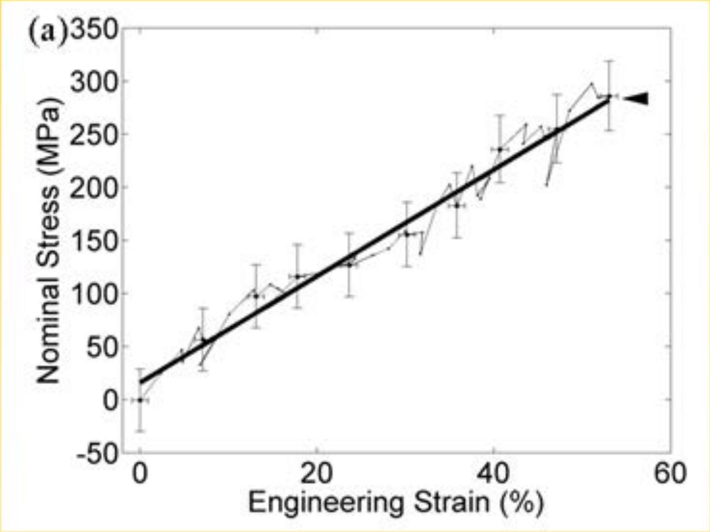


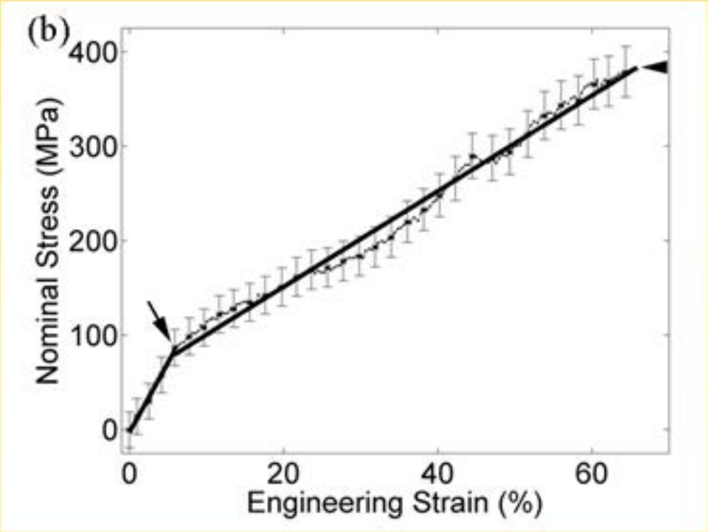


Recovery when placed in high humidity for 100, 200 minutes



True stress-Eulerian strain curves showing the data and fits for the first loads (solid squares and dash-dot line), first-fourth unloads (stars and solid line), and second-fourth loads (open circles and dashed line) for (a) test 1, (b) test 2, and (c) test 3.

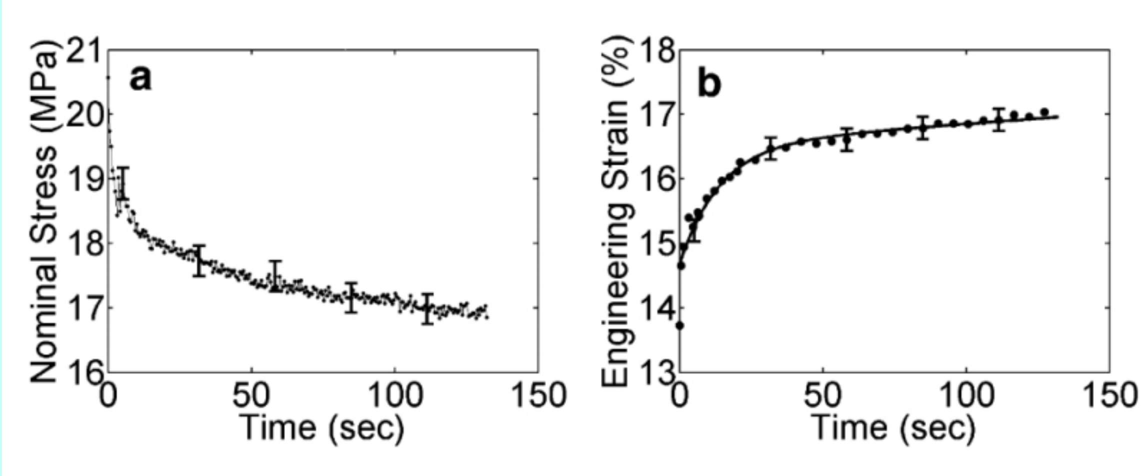


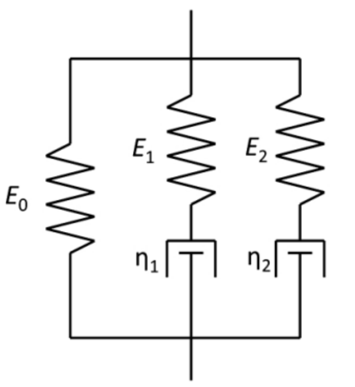


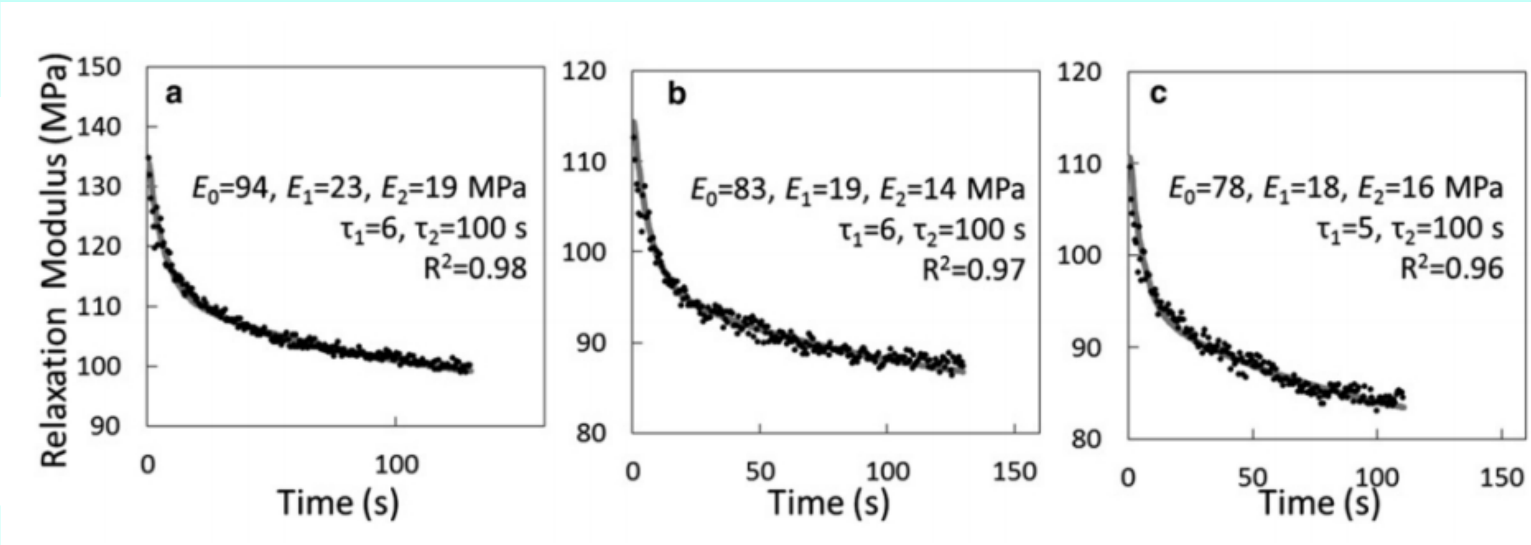
First ever experiments to failure!
Strain to failure as high as 1.0
Strength a better part of a GPa

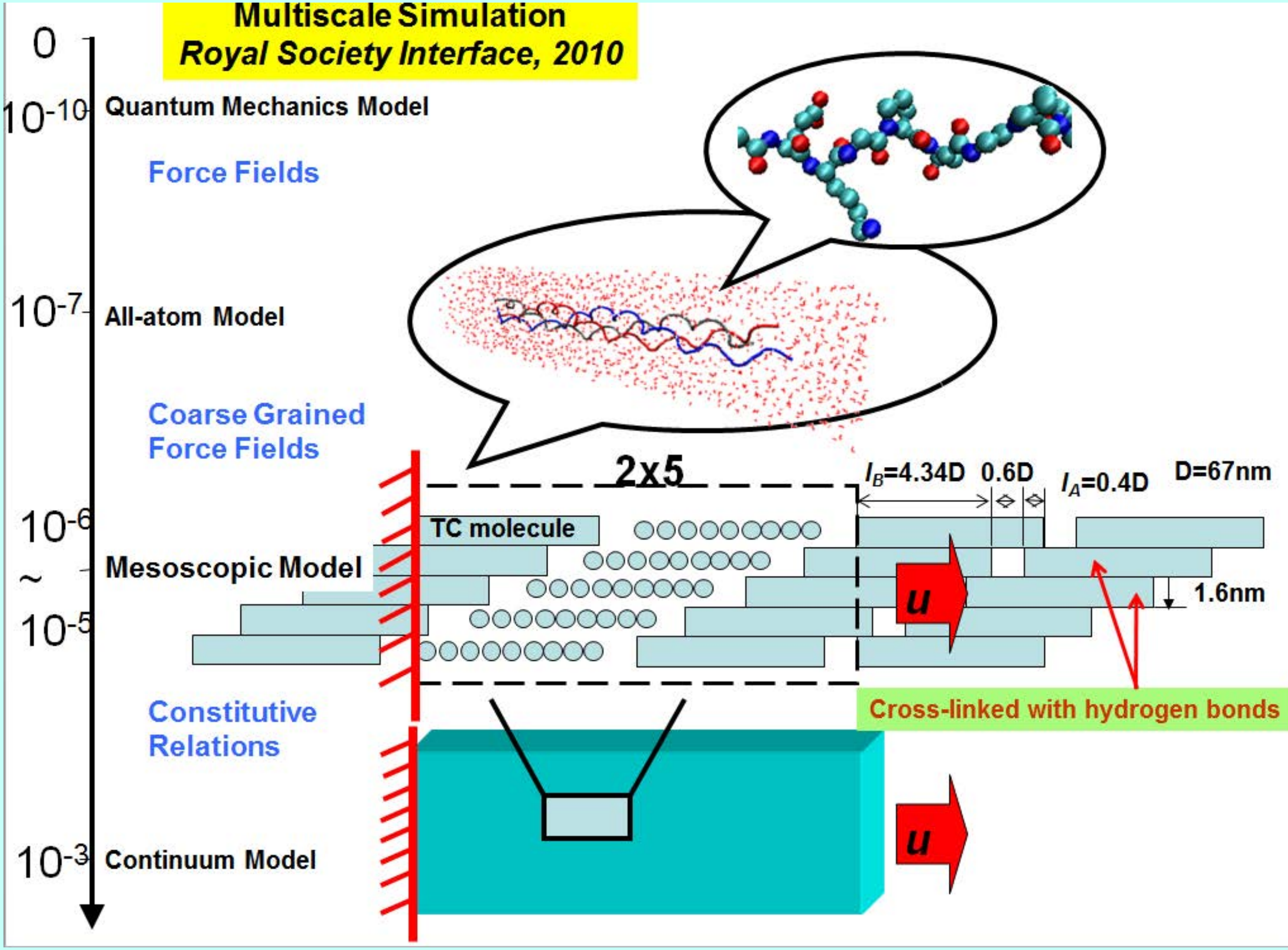
Question: Why do they fail catastrophically upon peak load brittle?

Viscoelastic behavior



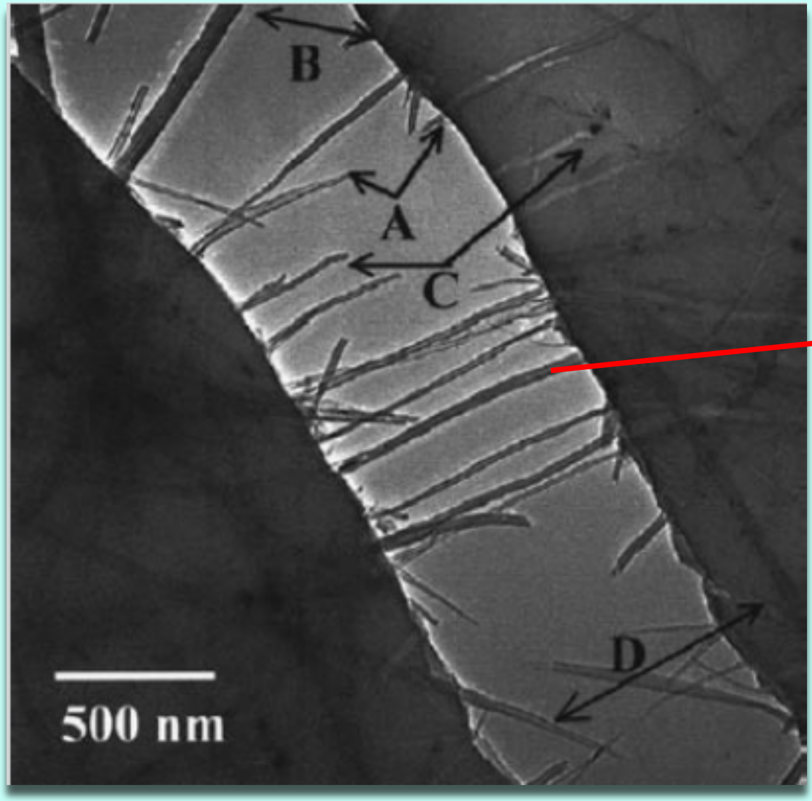


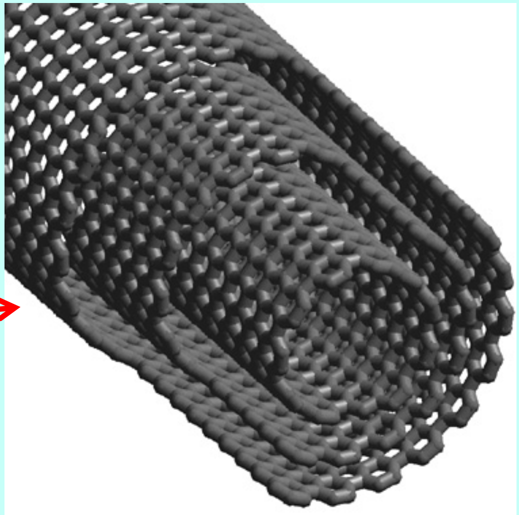




Multiscale Simulation
Royal Society Interface, 2010
0
10-10 Quantum Mechanics Model
Force Fields
10-7 All-atom Model
Coarse Grained
Force Fields
10-6
Mesoscopic Model
~
10-5
Constitutive
Relations
10-3 Continuum Model
2x5
TC molecule
lB=4.34D 0.6D lA=0.4D D=67nm
1.6nm
u
Cross-linked with hydrogen bonds
u

CNT Reinforced Nanocomposites





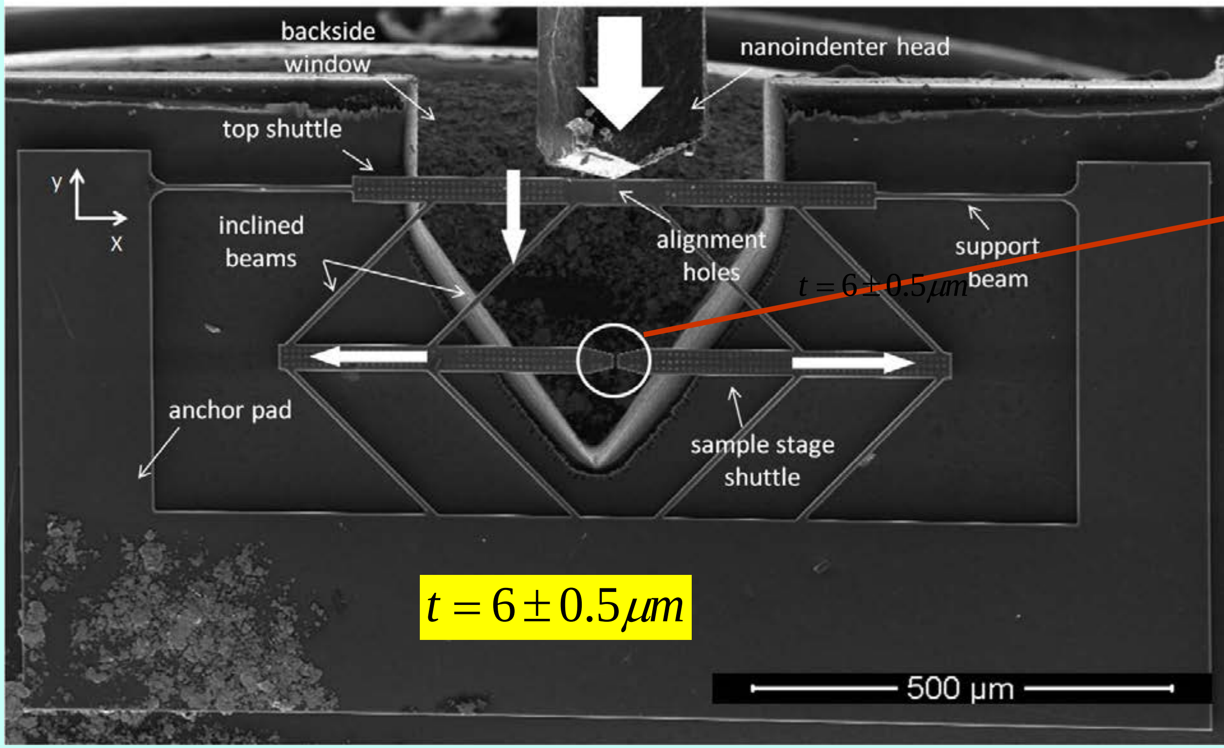
Crack bridging observed in MWNT/Polystyrene film (D. Qian *et al.*, 2000)

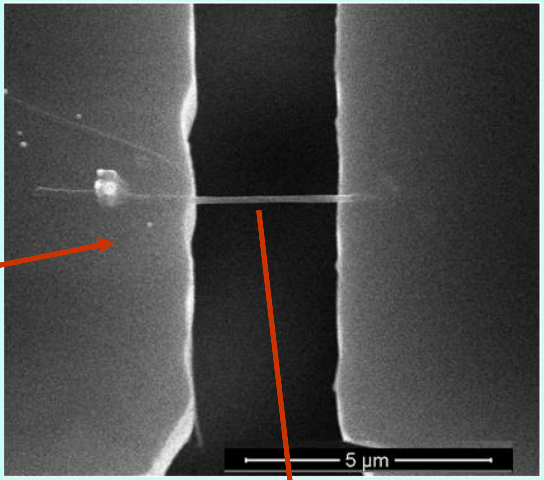
Questions:
How tough is the interface?
What can be done to increase toughness if it is too low?

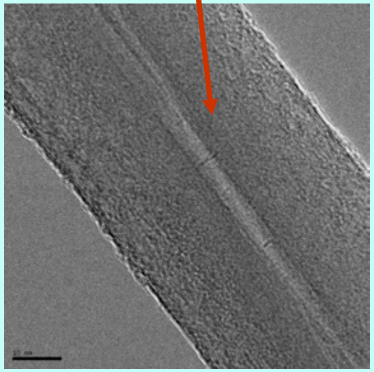
Multi-wall Carbon Nanotube/Epoxy Interface Toughness

Inside SEM chamber

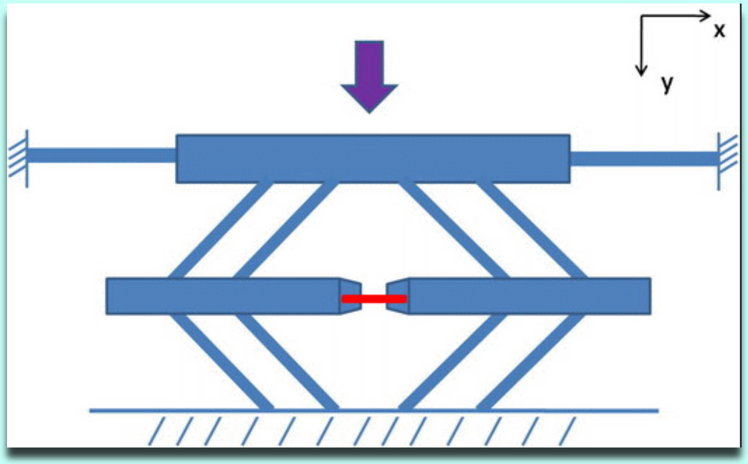
Nanoindenter (70nN, 0.9nm res.)

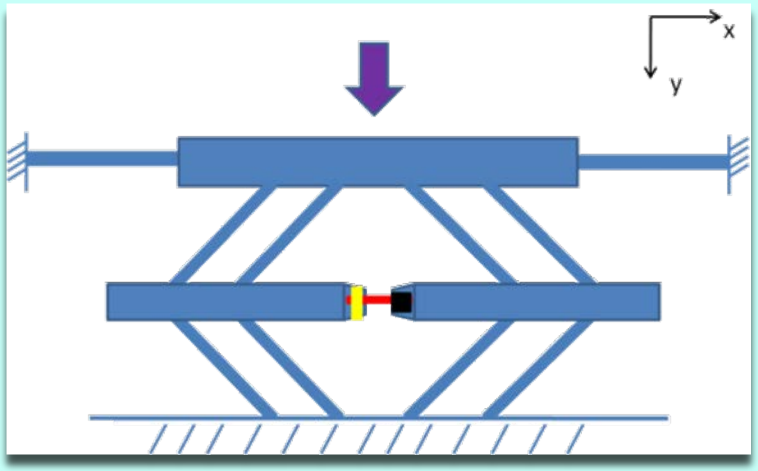






(Epon 828 epoxy)

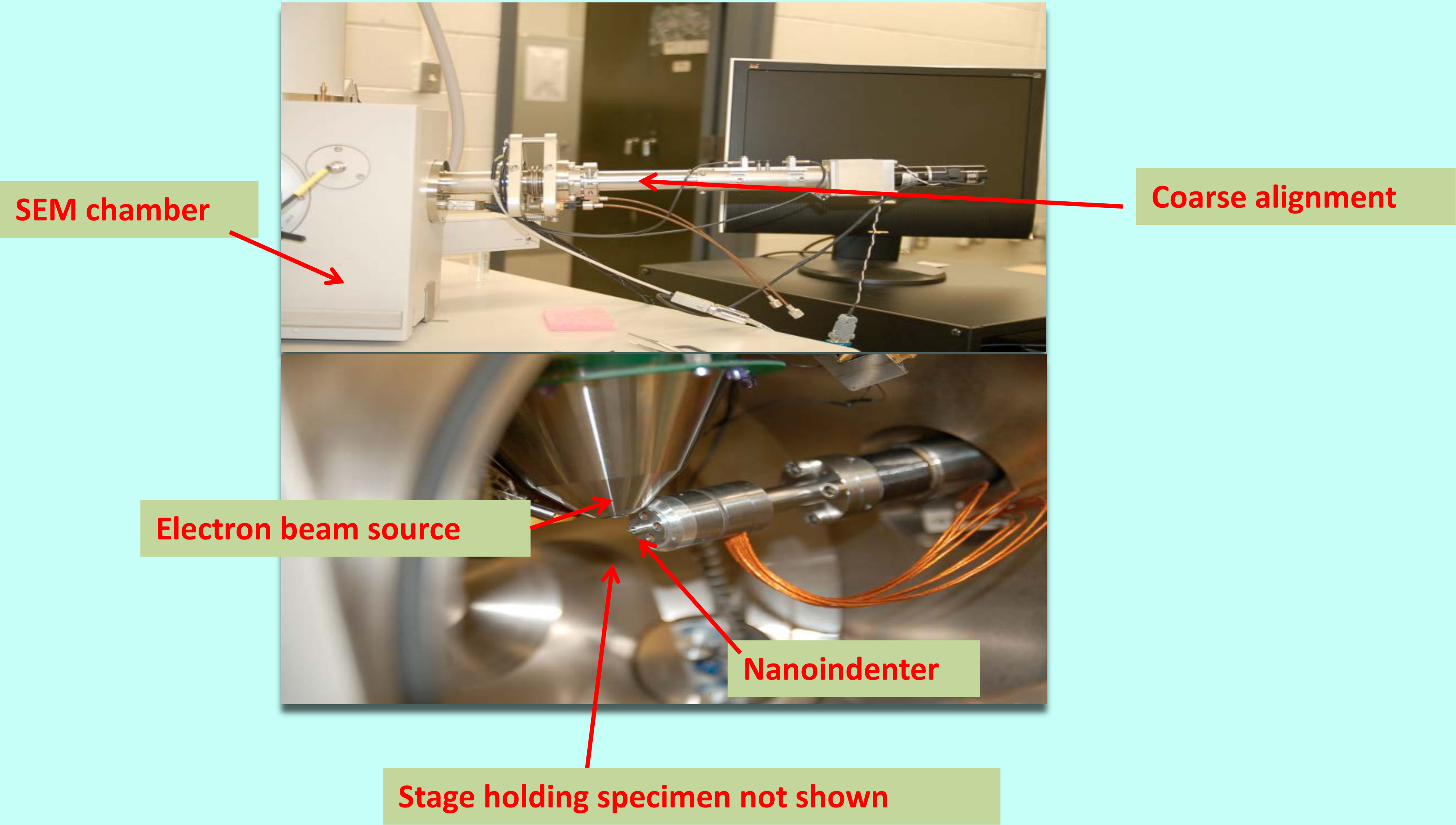




Testing of nano-fibers

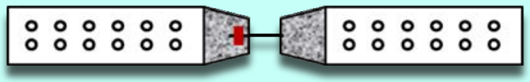
Pull-out tests

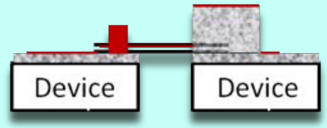
In-Situ SEM Experiments

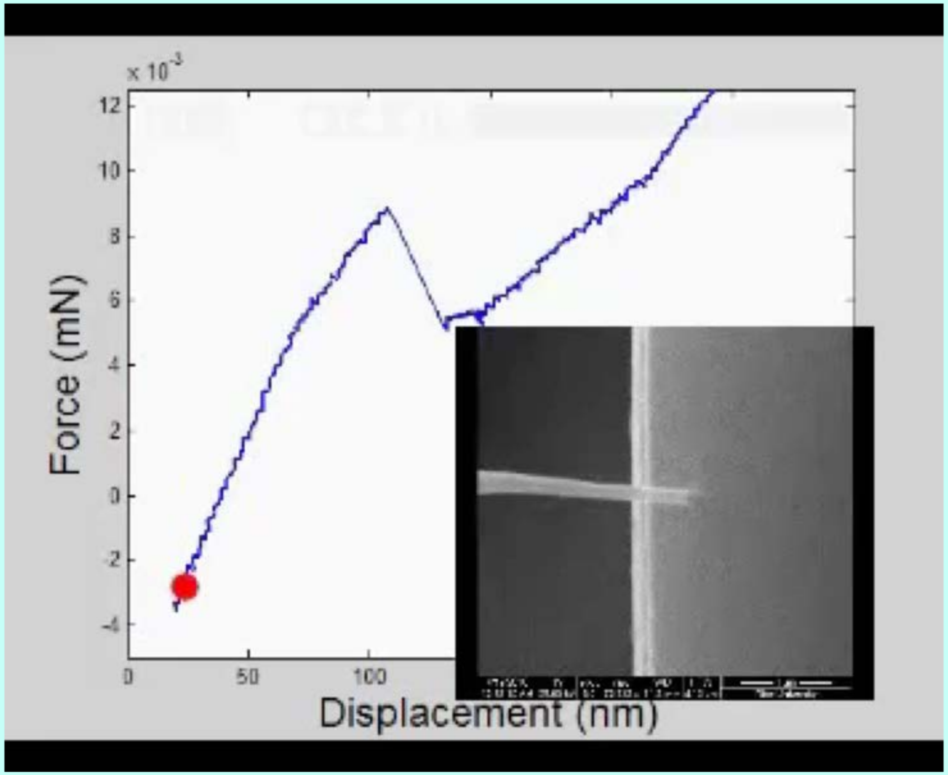


InSEM^{R} system allows usage of nanoindenter (Agilent^{TM} G200) within FEI Quanta FEG SEM

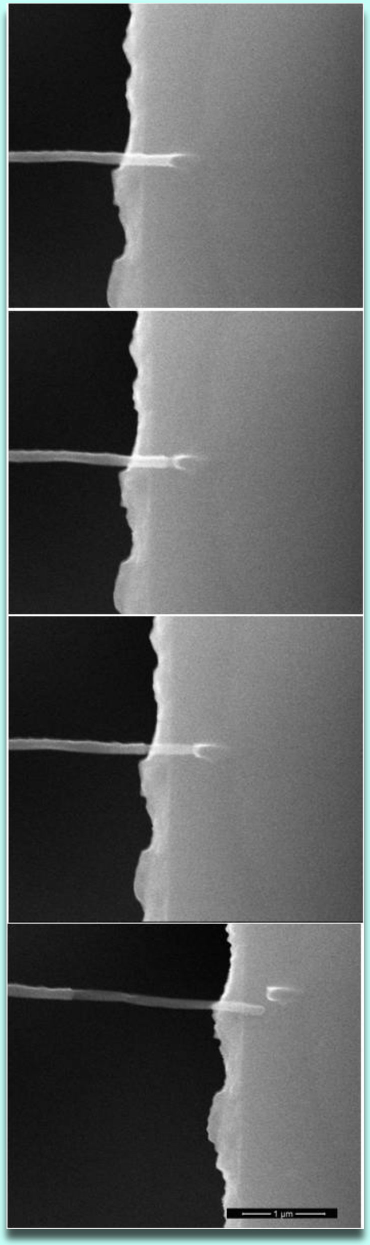
Single MWNT Pullout Experiments



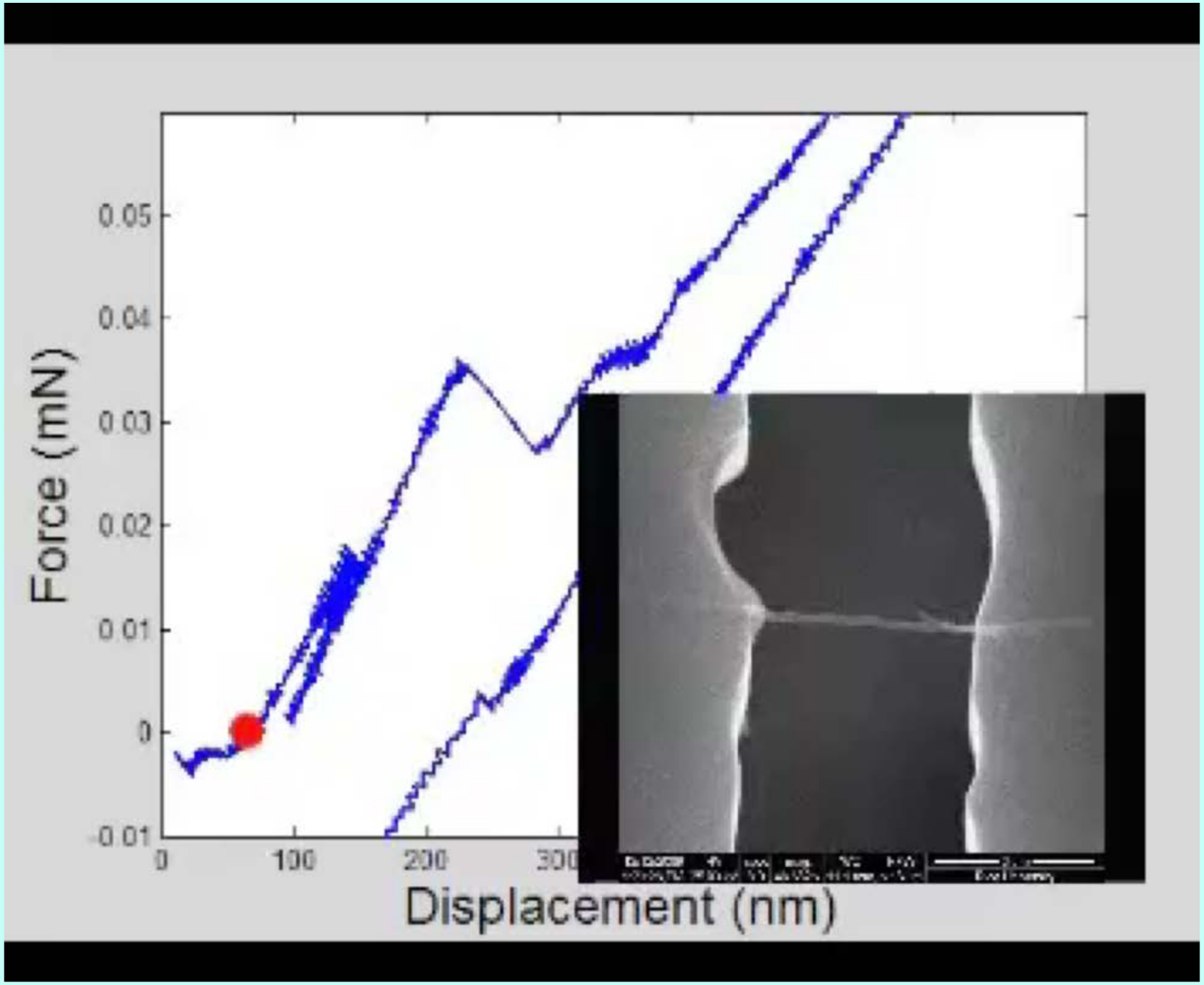




MWNT pullout video



Single MWNT pullout at t=0, t=19, t=70 and t=300 seconds respectively



Force (mN)
Displacement (nm)
0.05
0.04
0.03
0.02
0.01
0
-0.01
0
100
200
300

Temperature Dependant Fracture Initiation in Silicon

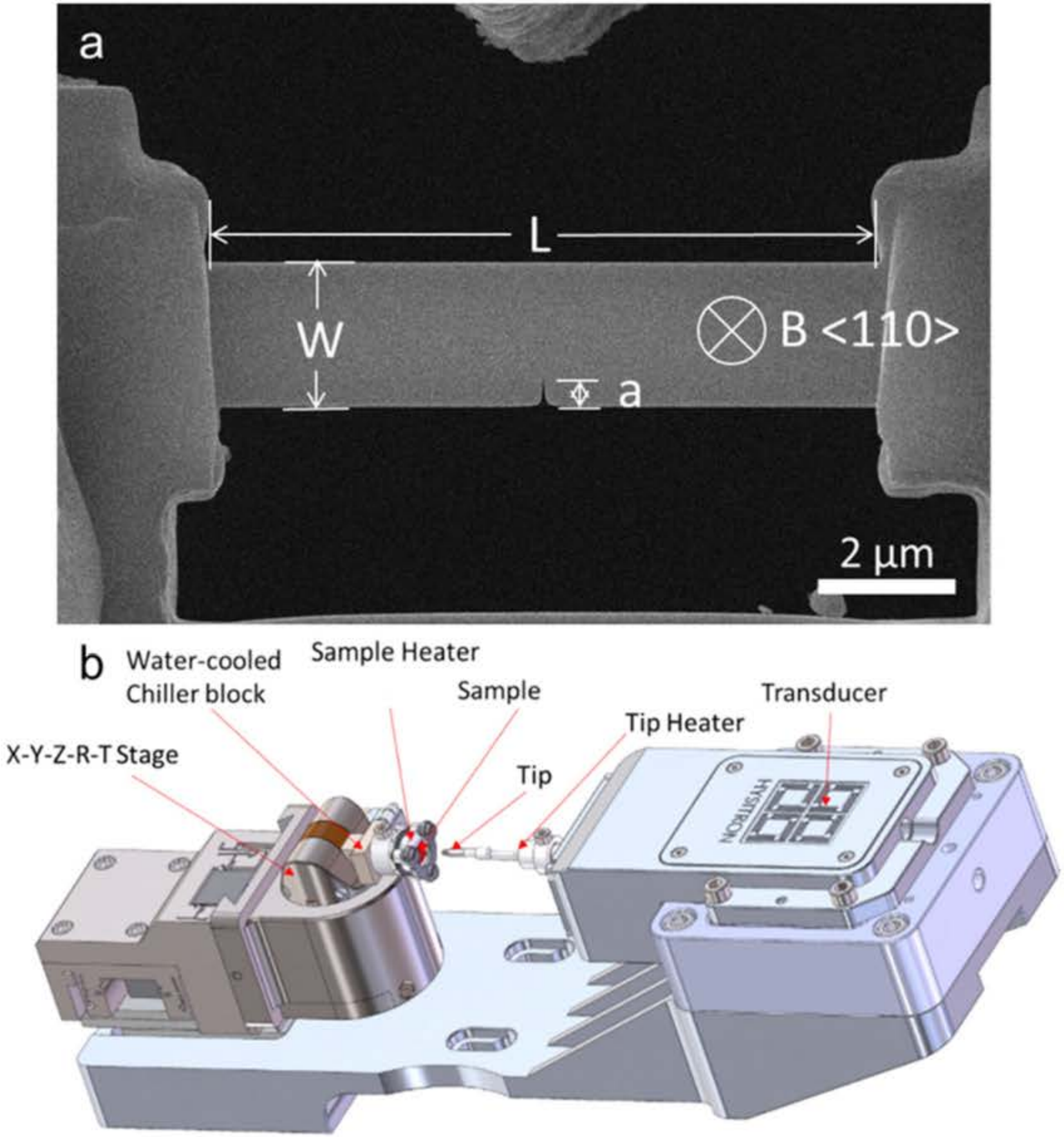
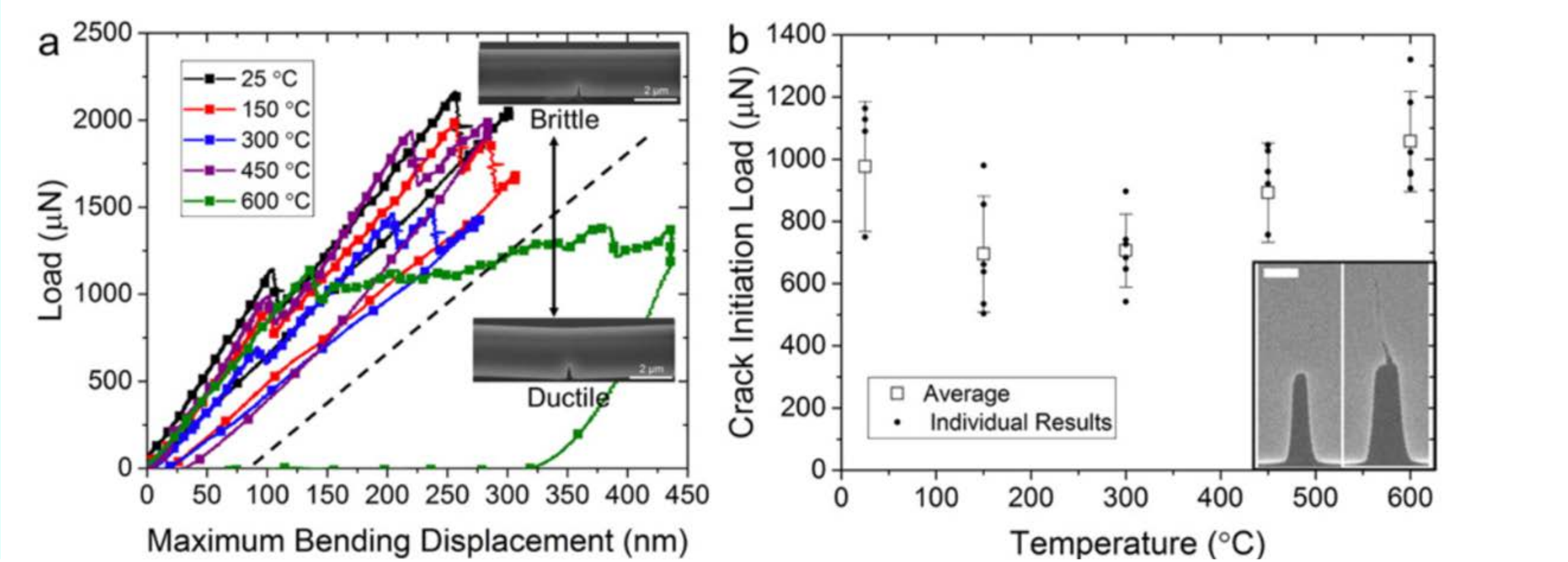
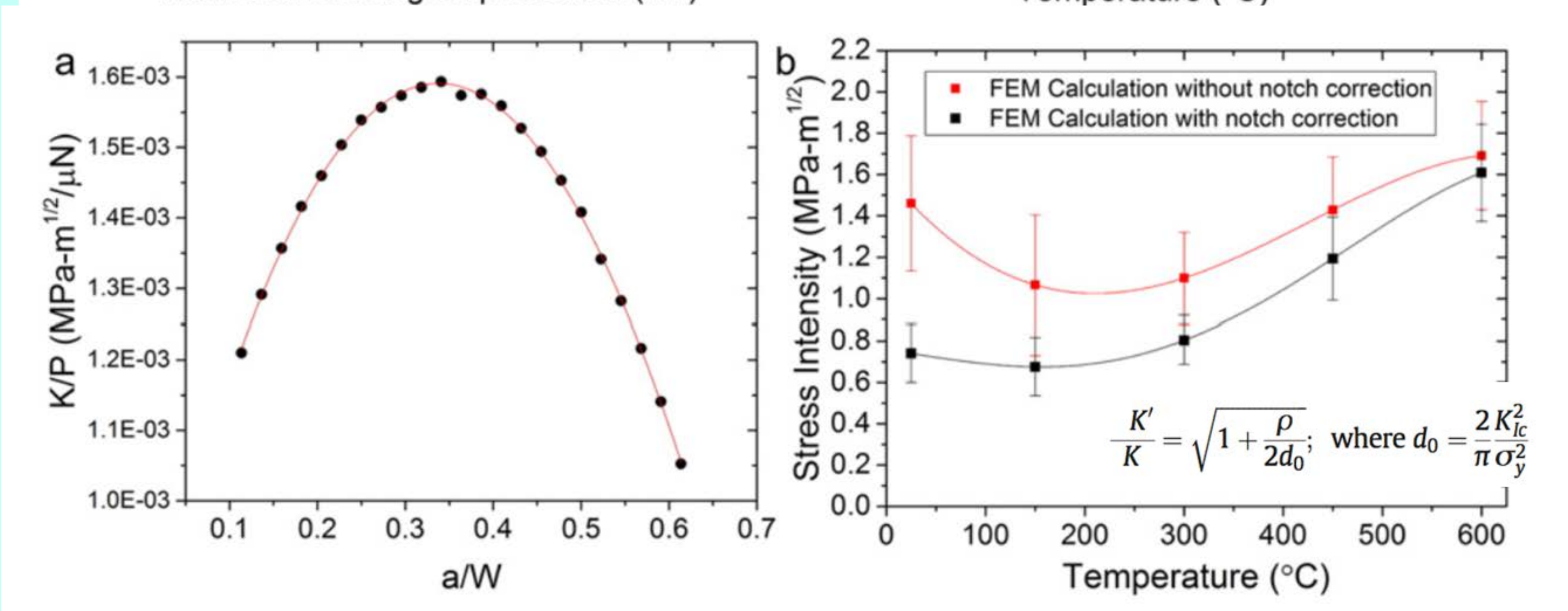
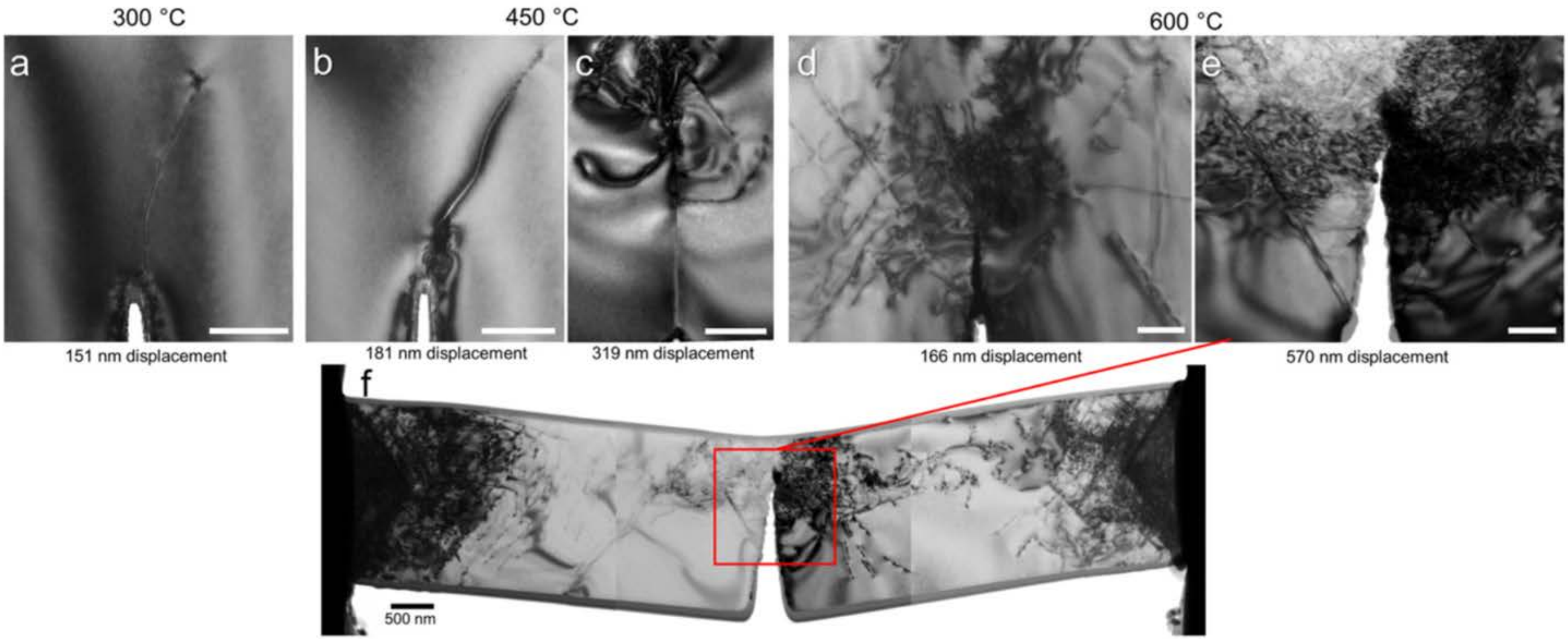


Fig. 1. a) SEM micrograph of as fabricated pre-notched microscale Si bending beam, with important quantities marked: L (length), W (width), B (thickness), a (notch length). Note this is an earlier specimen with a/W = 0.2 compared to a/W = 0.33 for the majority of the work which produced no significant difference in results. b) Schematic of Hysitron PI-87 SEM indenter system with integrated tip and sample heaters.


a
25 °C
150 °C
300 °C
450 °C
600 °C
Brittle
Ductile
2 µm
2 µm
Load (µN)
Maximum Bending Displacement (nm)
b
Crack Initiation Load (µN)
Average
Individual Results
Temperature (°C)


a
K/P (MPa-m^1/2/µN)
a/W
b
FEM Calculation without notch correction
FEM Calculation with notch correction
Stress Intensity (MPa-m^1/2)
Temperature (°C)
$\frac{K'}{K} = \sqrt{1 + \frac{\rho}{2d_0}}$; where $d_0 = \frac{2}{\pi}\frac{K_{Ic}^2}{\sigma_y^2}$

Post-Mortem TEM Showing Dislocations



"We have not succeeded in answering all of our problems. The answers we have found only serve to raise a whole set of new questions. In some ways we feel we are as confused as ever, but we believe we are confused on a higher level and about more important things."

(Posted outside mathematics reading room at Tromso University)