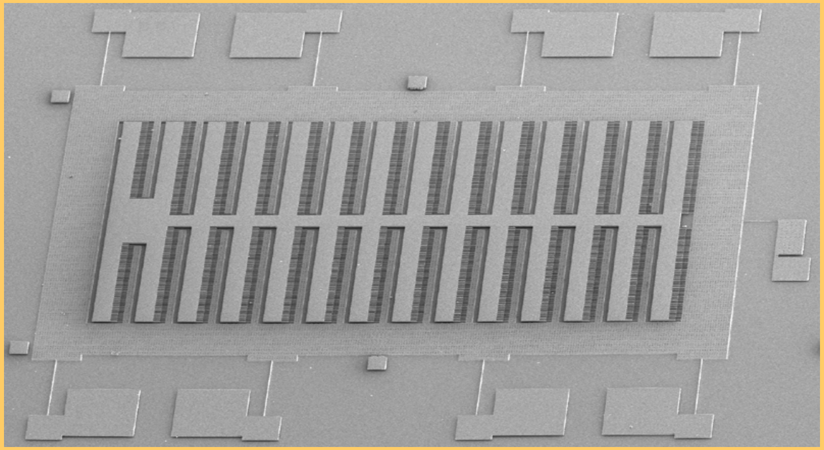
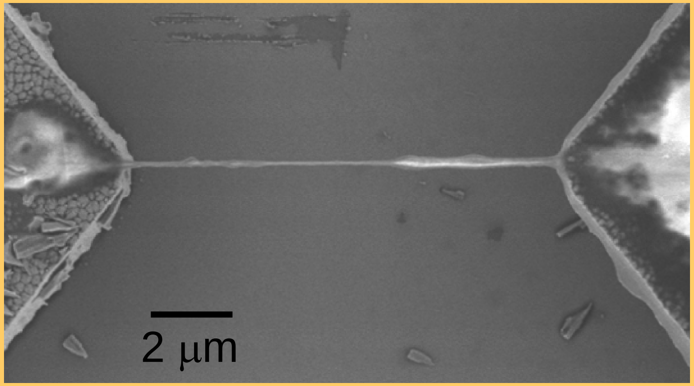
Structural Testing at the Micro and Nano Scales: Breaking Invisible Specimens With Zero Force





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Sponsors:

NSF, NIH, DARPA, ARO, NASA

CCNY
3/19/09

REFERENCES

Experimental work:

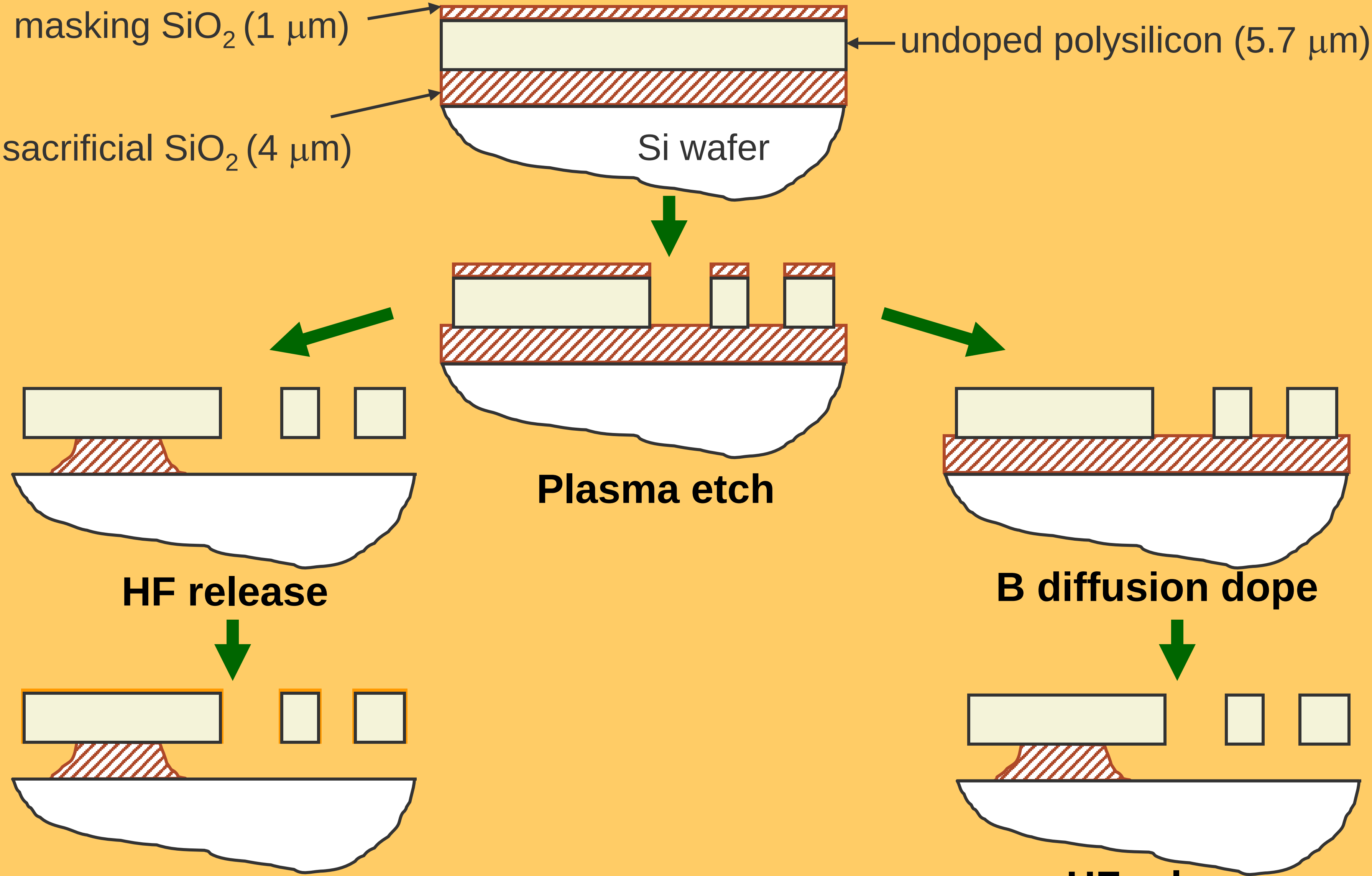
“Fatigue failure in polysilicon: it’s not due to simple stress corrosion cracking,” *Science* (2002).

“Electrostatically actuated failure of microfabricated polysilicon fracture mechanics specimens,” *Proc. R. Soc. Lond.* (1999).

“Mechanical Fatigue of Polysilicon: Effects of Mean Stress and Stress Amplitude,” *Acta Materialia* (2006).

“Nano measurements with micro devices: mechanical properties of hydrated collagen fibrils,” *J. of the R. Soc. Interface* (2006).

Surface Micromachining



masking SiO_2 (1 μm)

undoped polysilicon (5.7 μm)

sacrificial SiO_2 (4 μm)

Si wafer

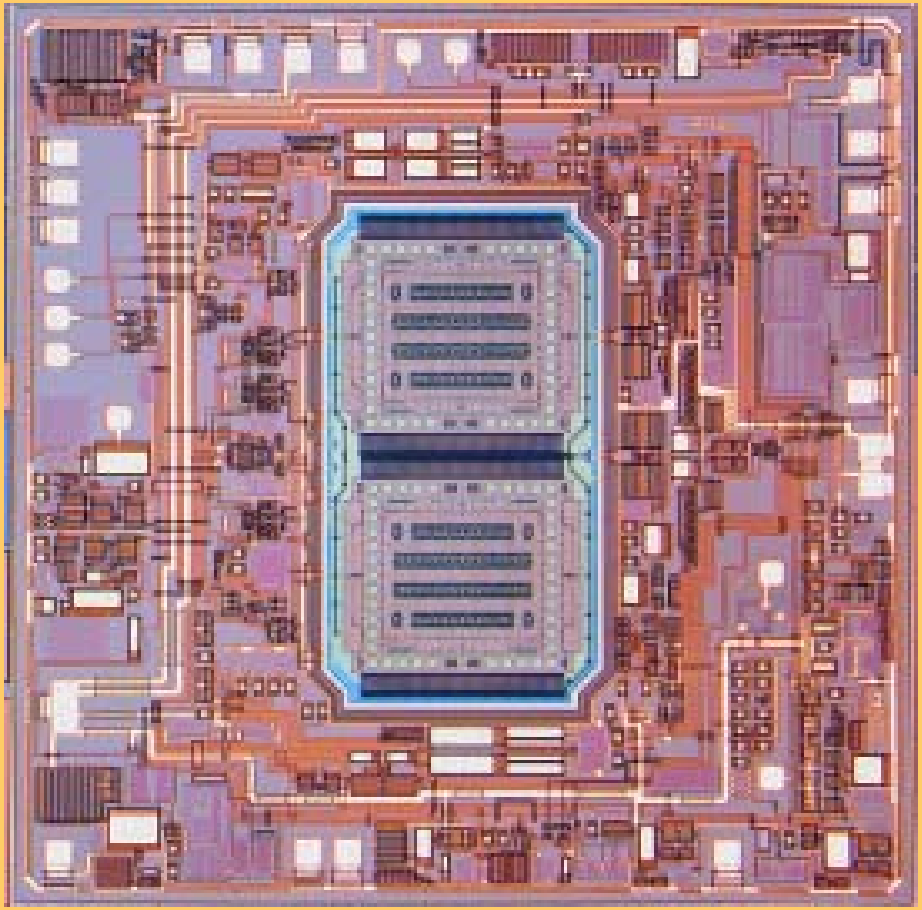
Plasma etch

HF release

B diffusion dope

Pd sputter (17 nm)

HF release



Analog Devices Gyroscope

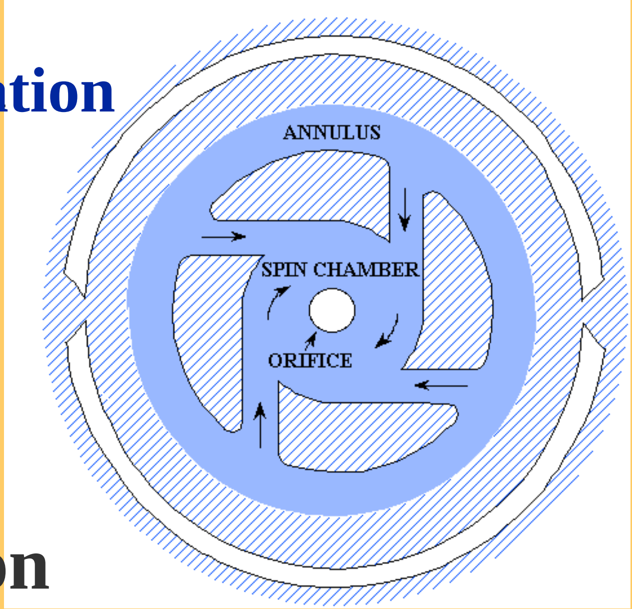
iMEMS Gyro Die Showing the Rate Sensor and Integrated Electronics

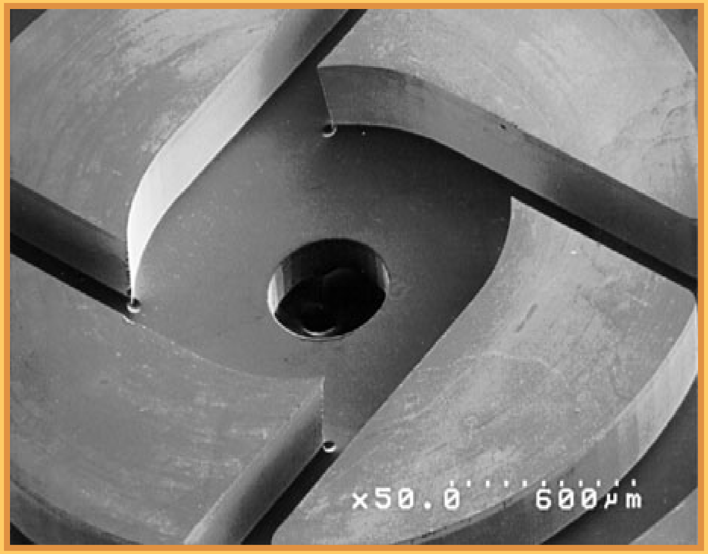
http://www.analog.com/technology/mems/gyroscopes/index.html

MEMS Device-Fuel Atomizer

Motivation

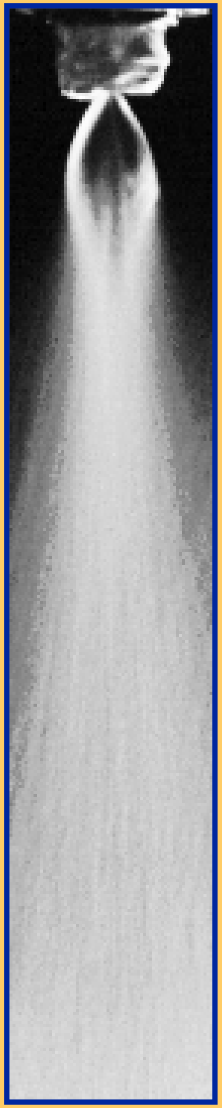
- Reduce cost through batch fabrication
- Achieve desired tolerances using a precise silicon micromachining technology



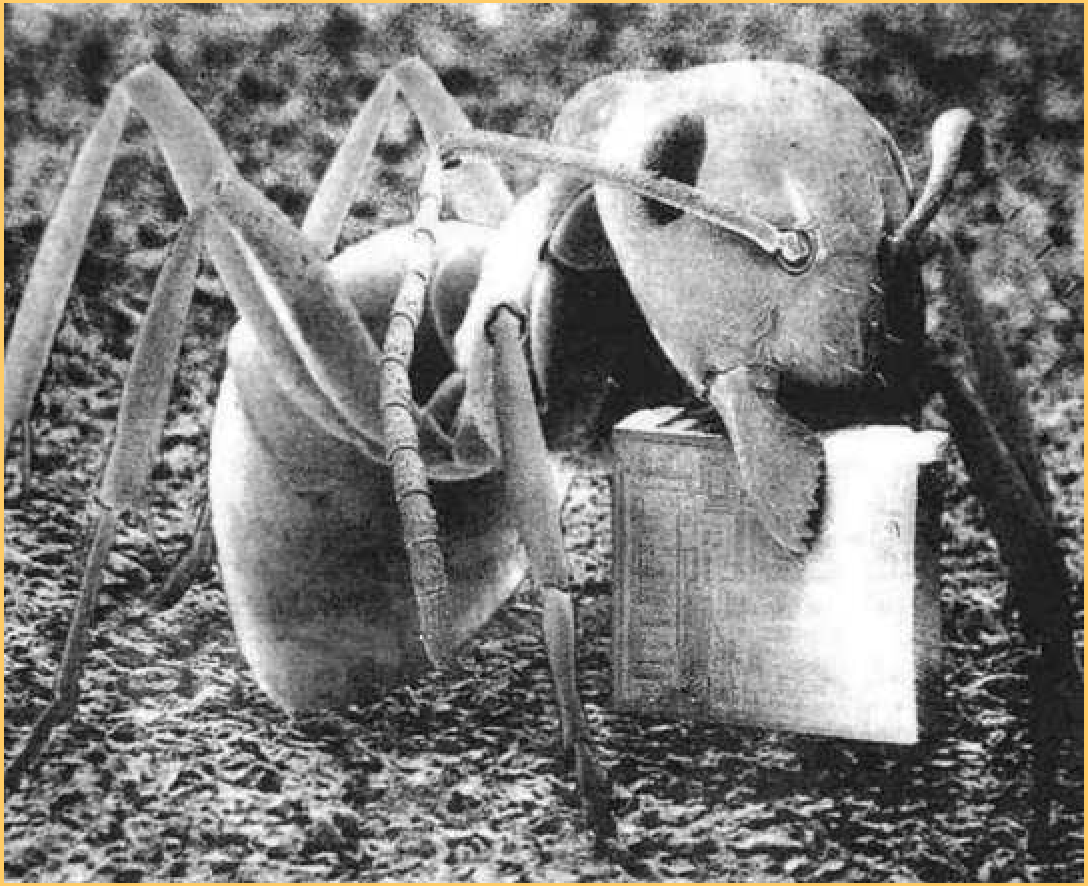


Operation

- Fuel enters the spin chamber through tangential slots
- Fuel swirls in the spin chamber and exits through the orifice in a hollow conical spray
- Swirling produces sprays with wider spray angles as compared to plain orifice atomizers



Ant Carrying a (1000 μm)^2 Microchip



Or is it a Palm Pilot?

ORIGINAL OBJECTIVES

Characterize **strength**, **fracture toughness**, **high cycle fatigue** and **environmentally assisted crack growth** in poly-Si, poly-SiC, and SiC at scales relevant to MEMS devices.

- Develop (micron size) ***on-chip*** specimens.
- Generate data.
- Study mechanisms.
- Formulate predictive models.

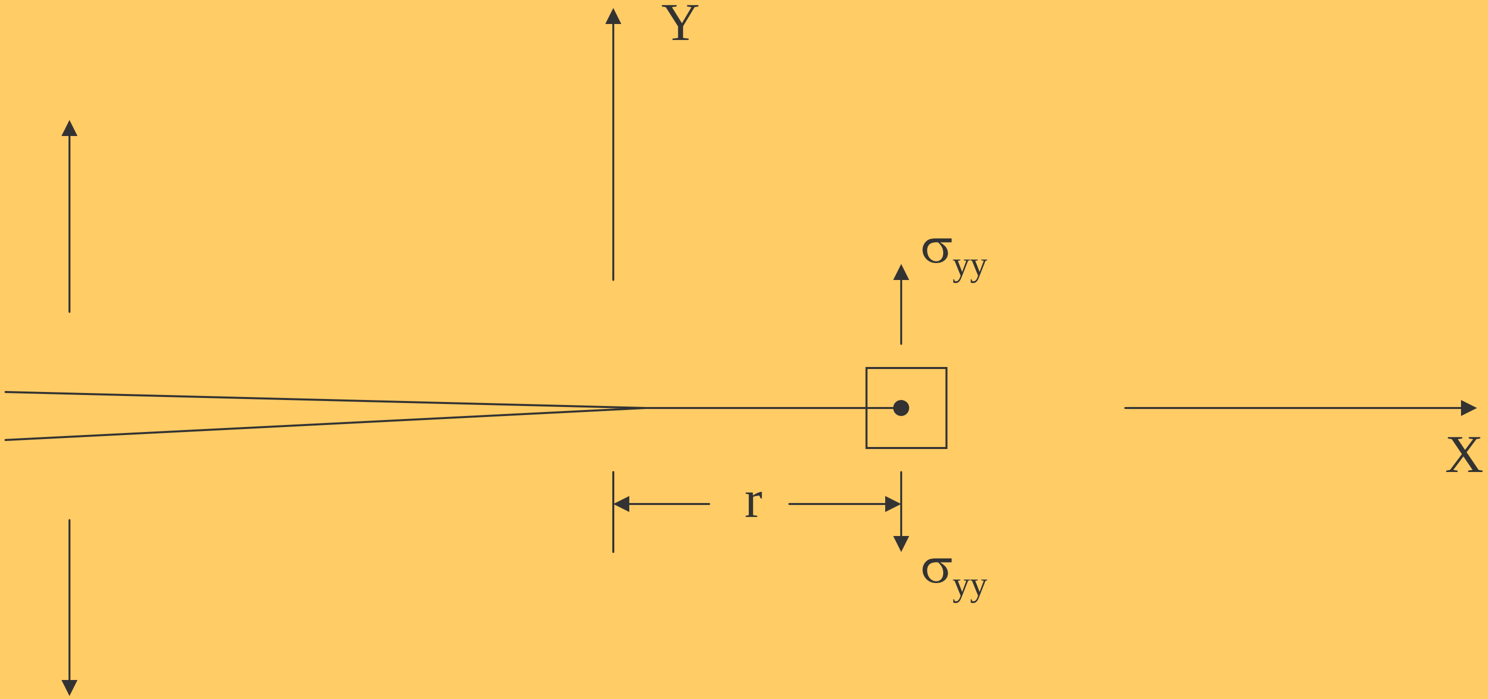
CHALLENGES

- Experiments are difficult to design, execute and interpret.

NEW OBJECTIVES

Use MEMS devices to test nanoscale structures

CRACK TIP PARAMETERS



$$\sigma_{yy} = \frac{K_I}{\sqrt{2\pi r}}$$

K_I is the stress intensity factor

CRACK GROWTH MECHANISMS

Fast fracture

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

High cycle fatigue

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

Stress corrosion

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

Native Oxide
is known to obey this law

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

FOCUS OF THIS TALK

- **Demonstrate that polycrystalline silicon is not susceptible to static fatigue.**

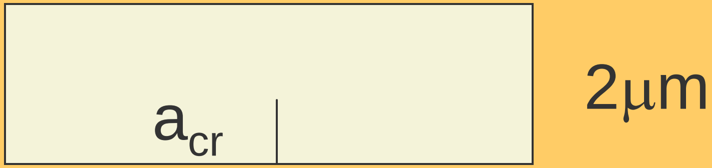
- **Demonstrate that polycrystalline silicon is associated with mechanical fatigue and *strenghtening* mechanisms.**

- **Describe development of nanoscale testing of biological structures.**

Two types of on-chip specimens have been developed:

- Loading through electrostatic actuation
- Loading through fabrication-induced residual stress

Why subcritical crack initiation and growth should be studied in MEMS



Say a_{cr}=1μm

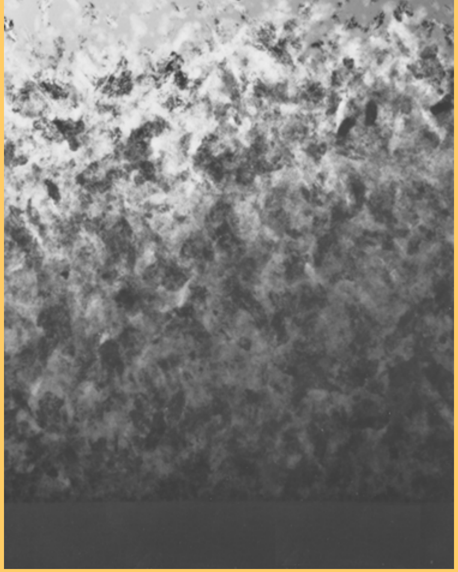
Say t_{life}=10yrs

Then $v_{cr}<10^{-15}$ m/s !!!

CVD Polysilicon - Effects of Deposition Temperature

550°C

580°C

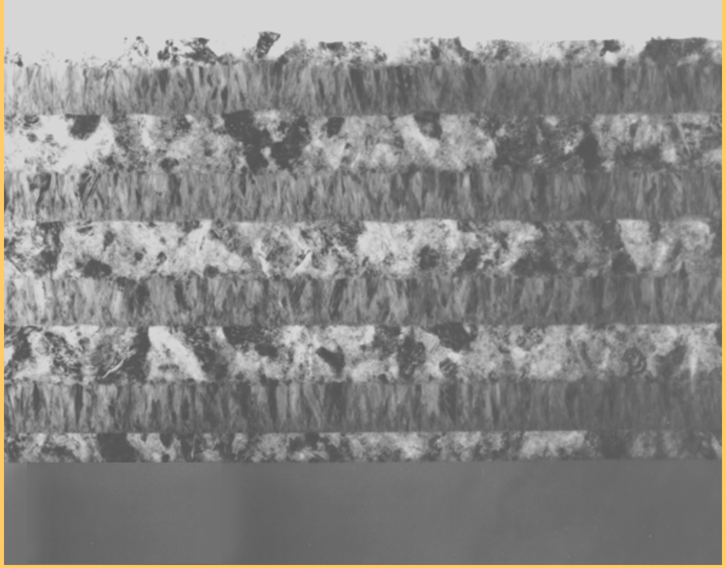


615°C

all films
are ~2-6 µm
thick, and
deposited
on SiO_2

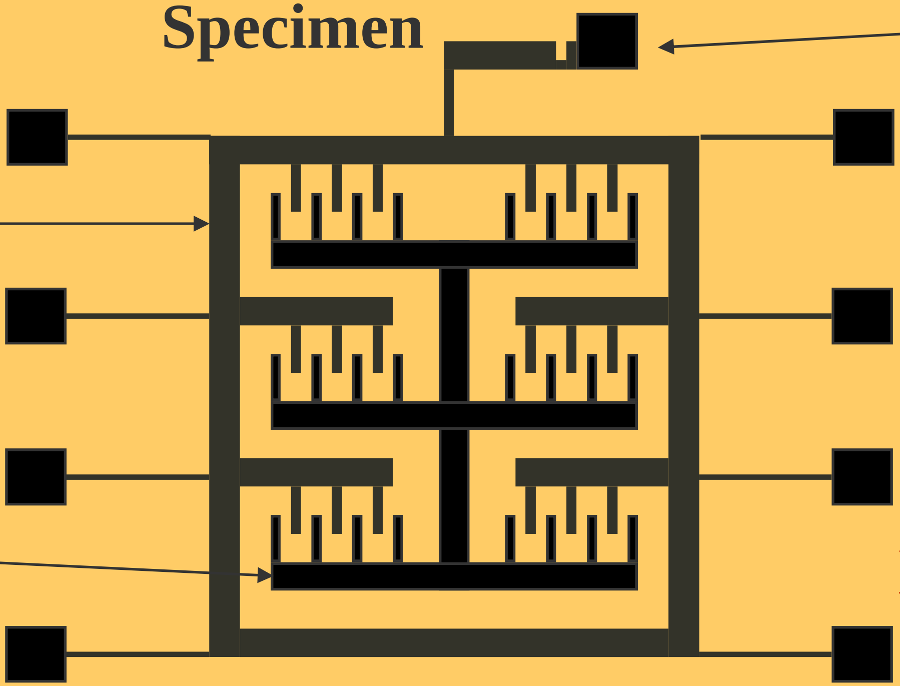
1100°C

570/615°C



MEMS Fracture Mechanics Specimen
integrated with
MEMS Loading Device Actuator

(*Proc. Royal Soc.* A, 455, 3807-3823, 1999)



Fracture Mechanics Specimen

anchor pads

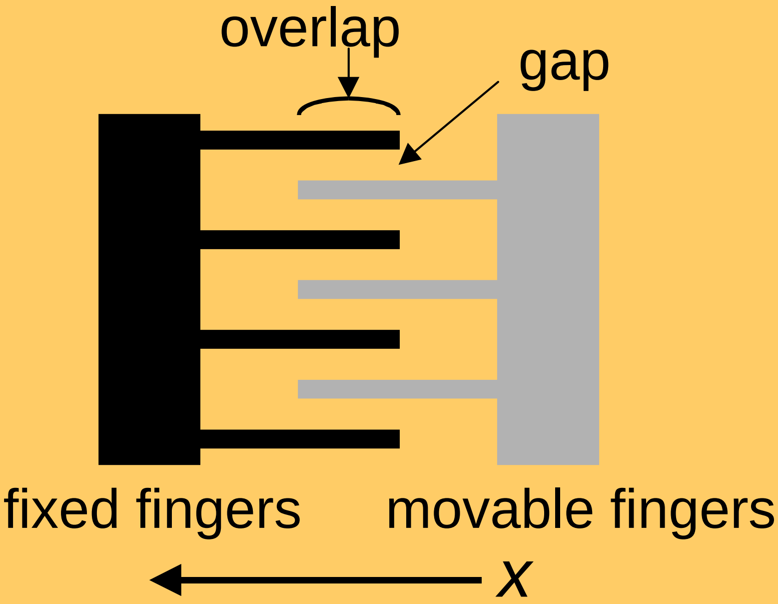
movable comb drive

fixed comb drive

Actuator

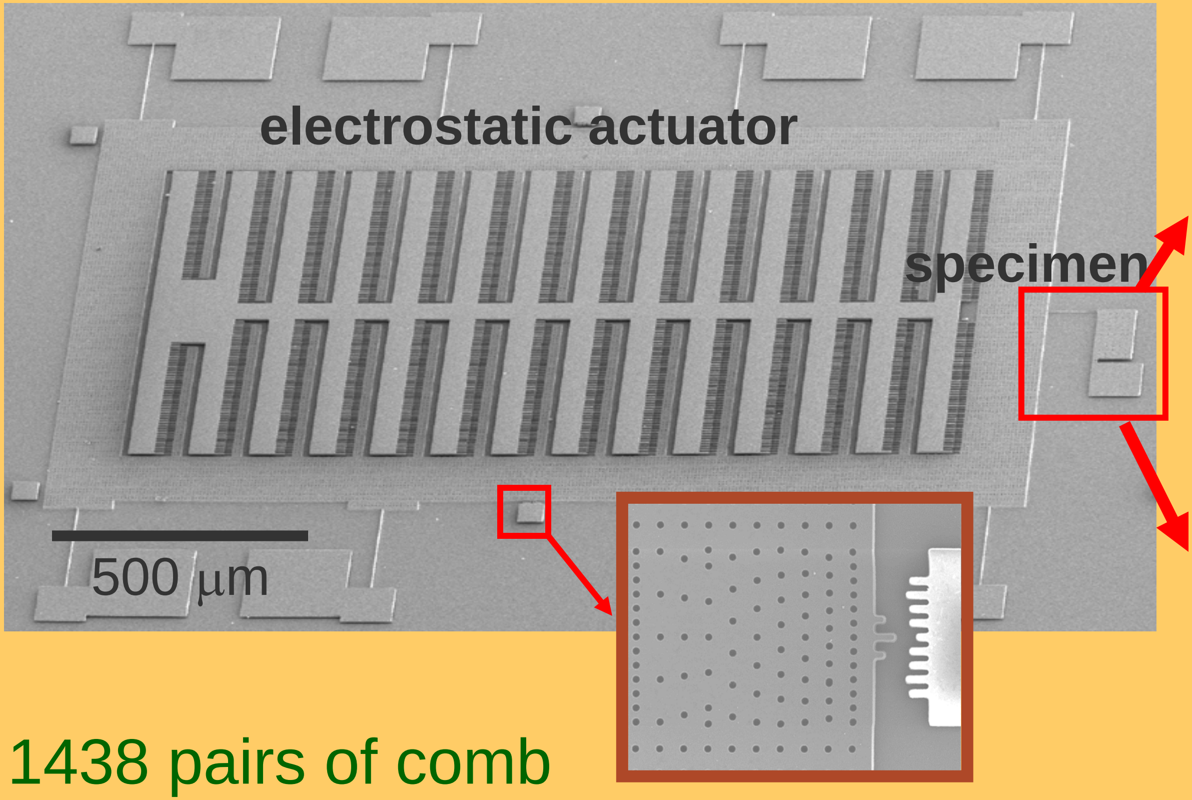
produces ~0.7 mN

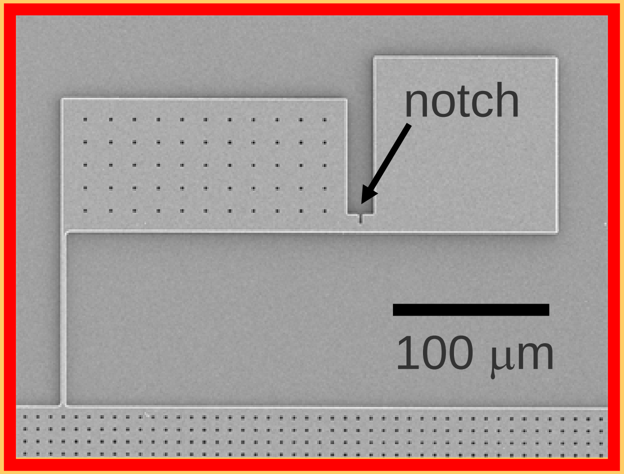
Electrostatic Actuation

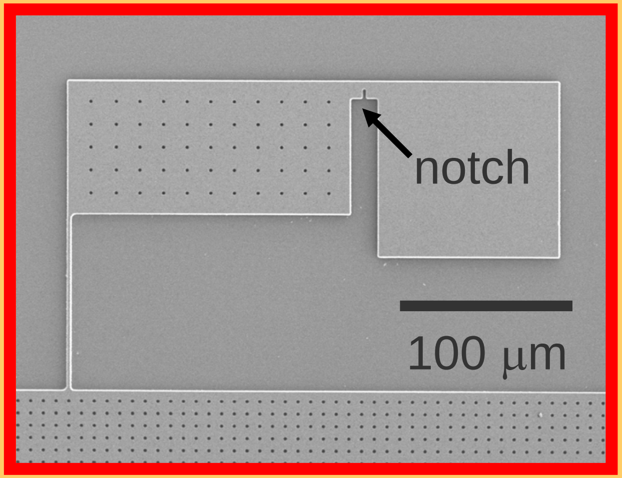


$$F = \frac{1}{2}\frac{\partial C}{\partial x}V^2 = n\varepsilon\frac{h}{g}V^2$$

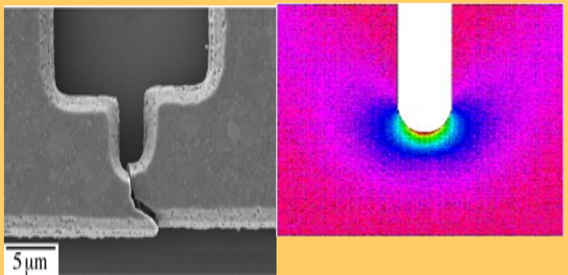
Fatigue Testing





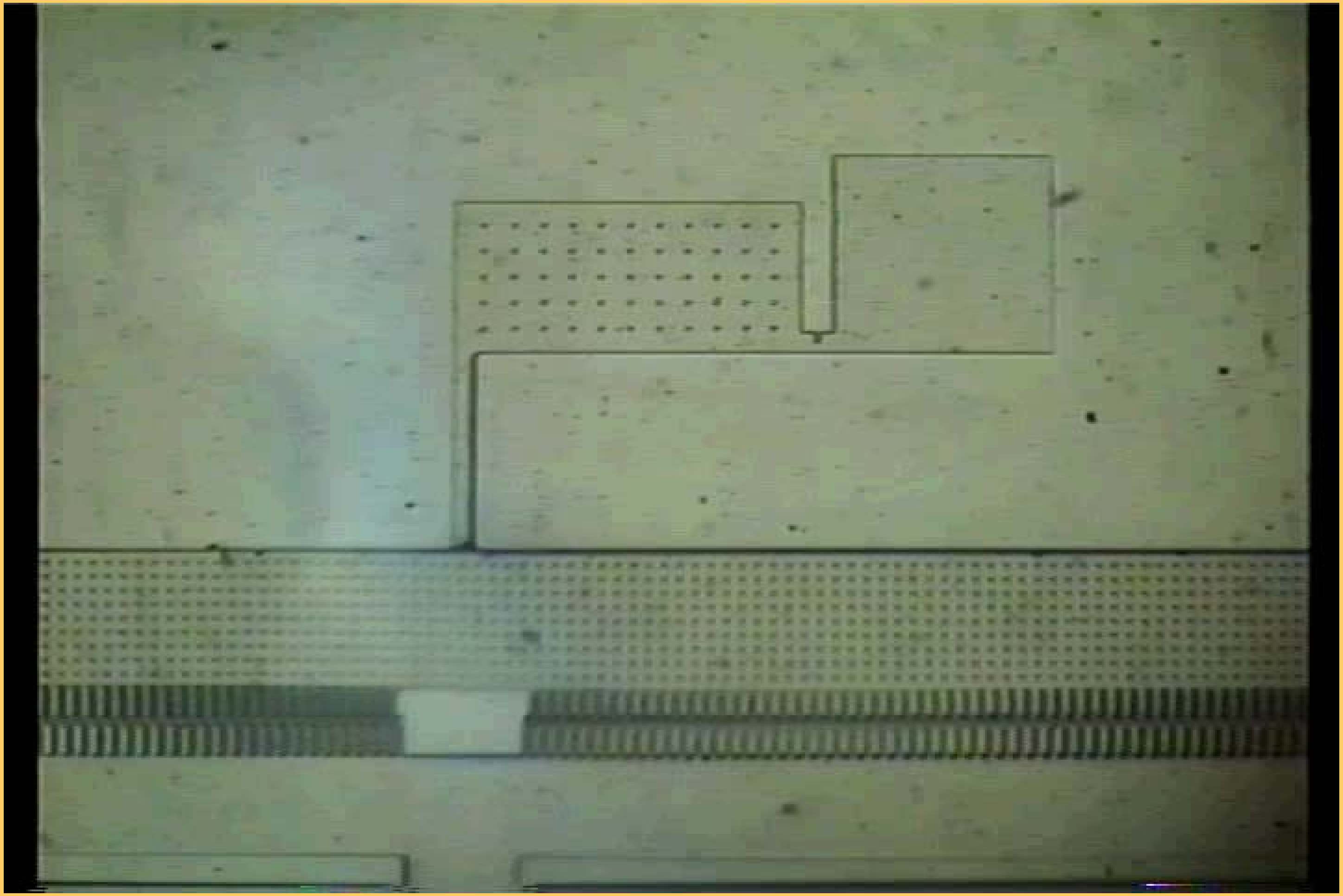


1438 pairs of comb fingers; 0.8 mN at 150 V

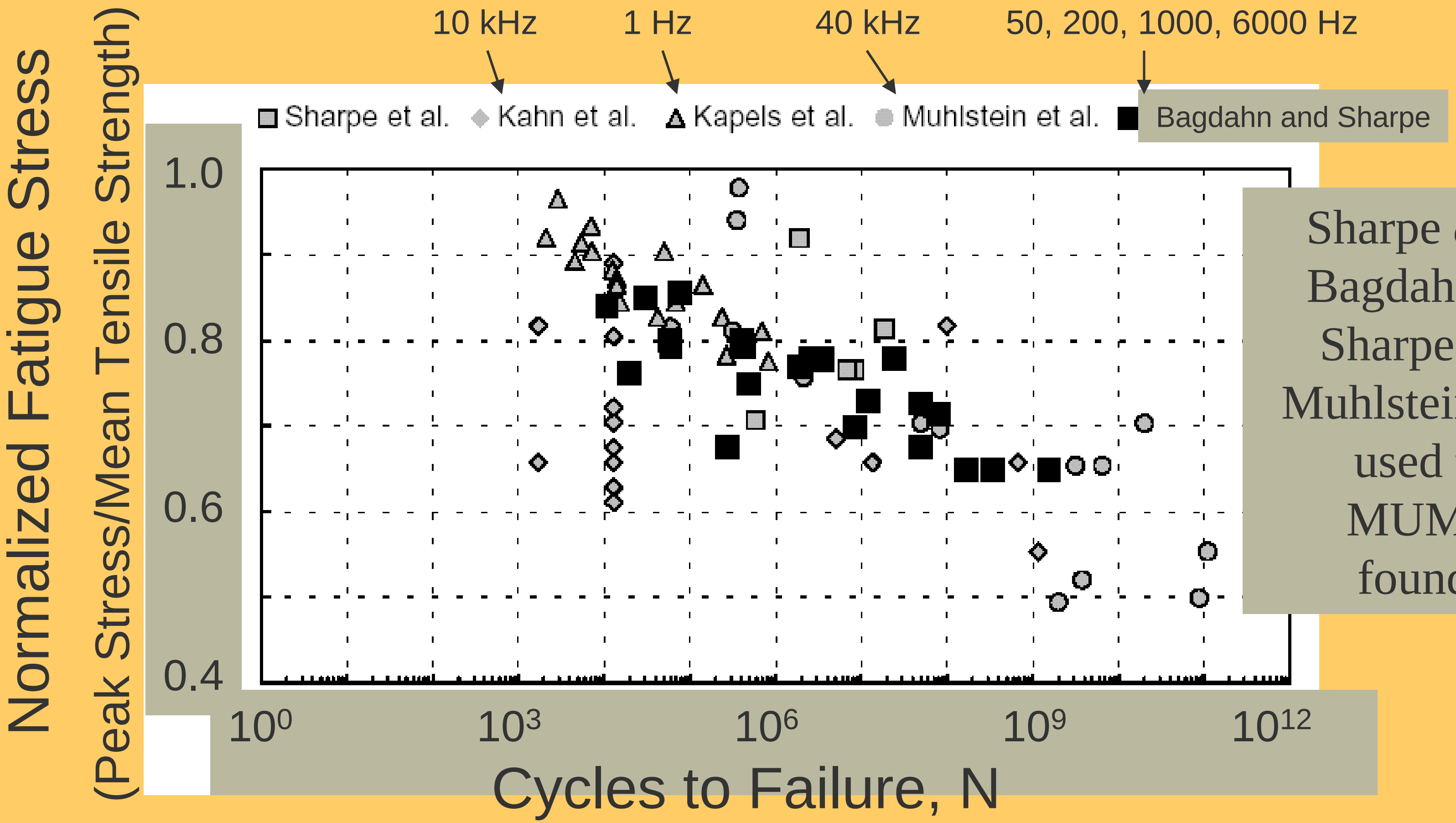


ADVANTAGES OF THIS “ON-CHIP” SPECIMEN

- **No need for external loading device.**
- **Resonance loading can be used to study *very* high cycle fatigue.**
- **Uncracked ligament size of the same order as dimensions of typical MEMS components.**
- **Can adjust mean stress and alternating stress.**



Dynamic Fatigue of Polysilicon



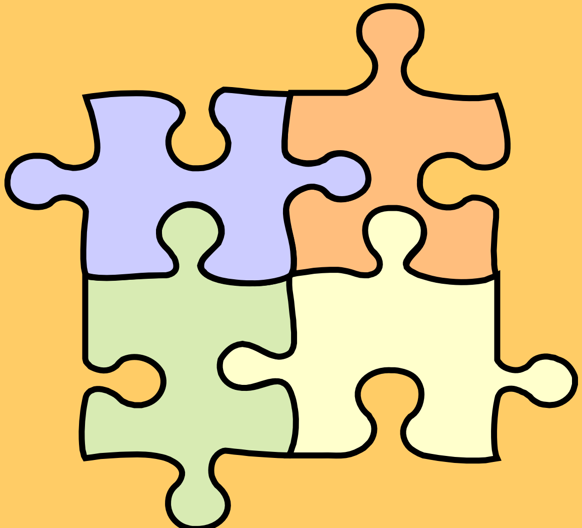
Sharpe *et al.*, Bagdahn and Sharpe, and Muhlstein *et al.* used the MUMPS foundry

Bagdahn and Sharpe, Sensors and Actuators A, 2003

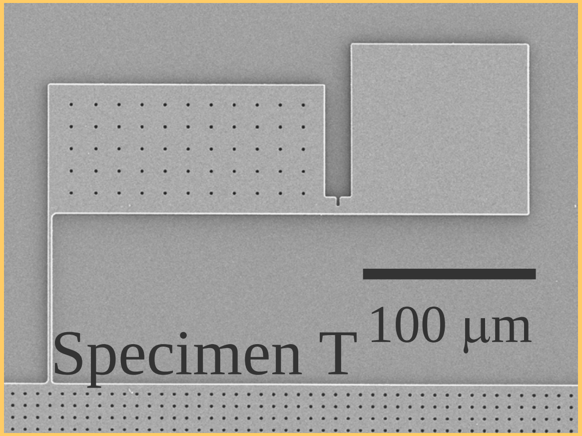
No frequency dependence of fatigue life, only on total number of cycles

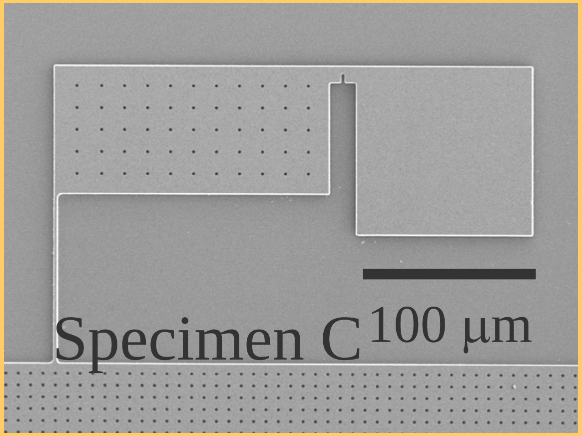
DIFFICULTIES IN DETERMINING ENVIRONMENTAL EFFECTS USING THESE TESTS

- Tests involve cyclic loading, not constant load.
- Tests involve tension and compression.



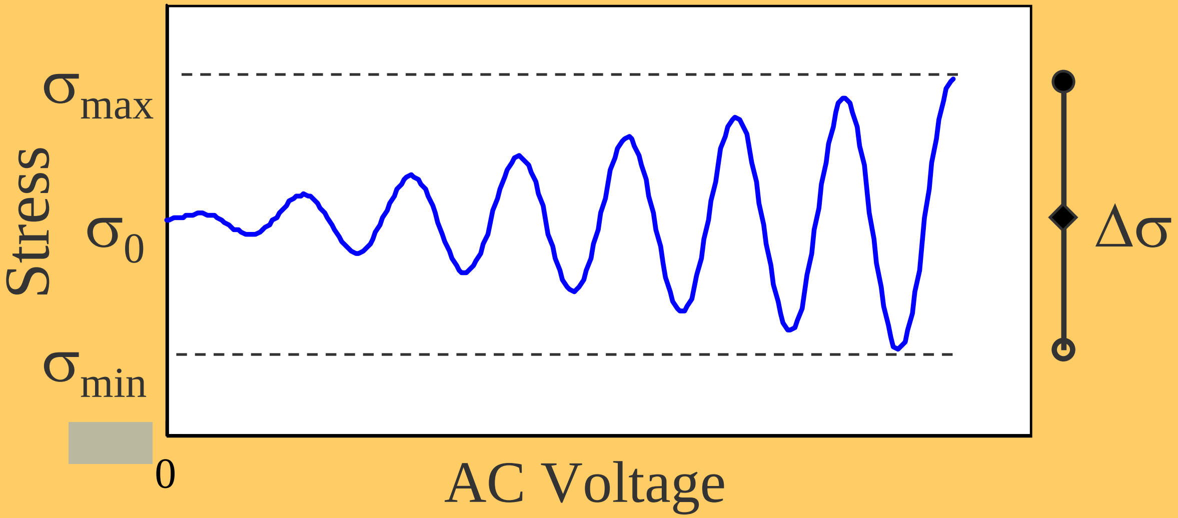
Biased Fatigue Experiment





Load Ratio

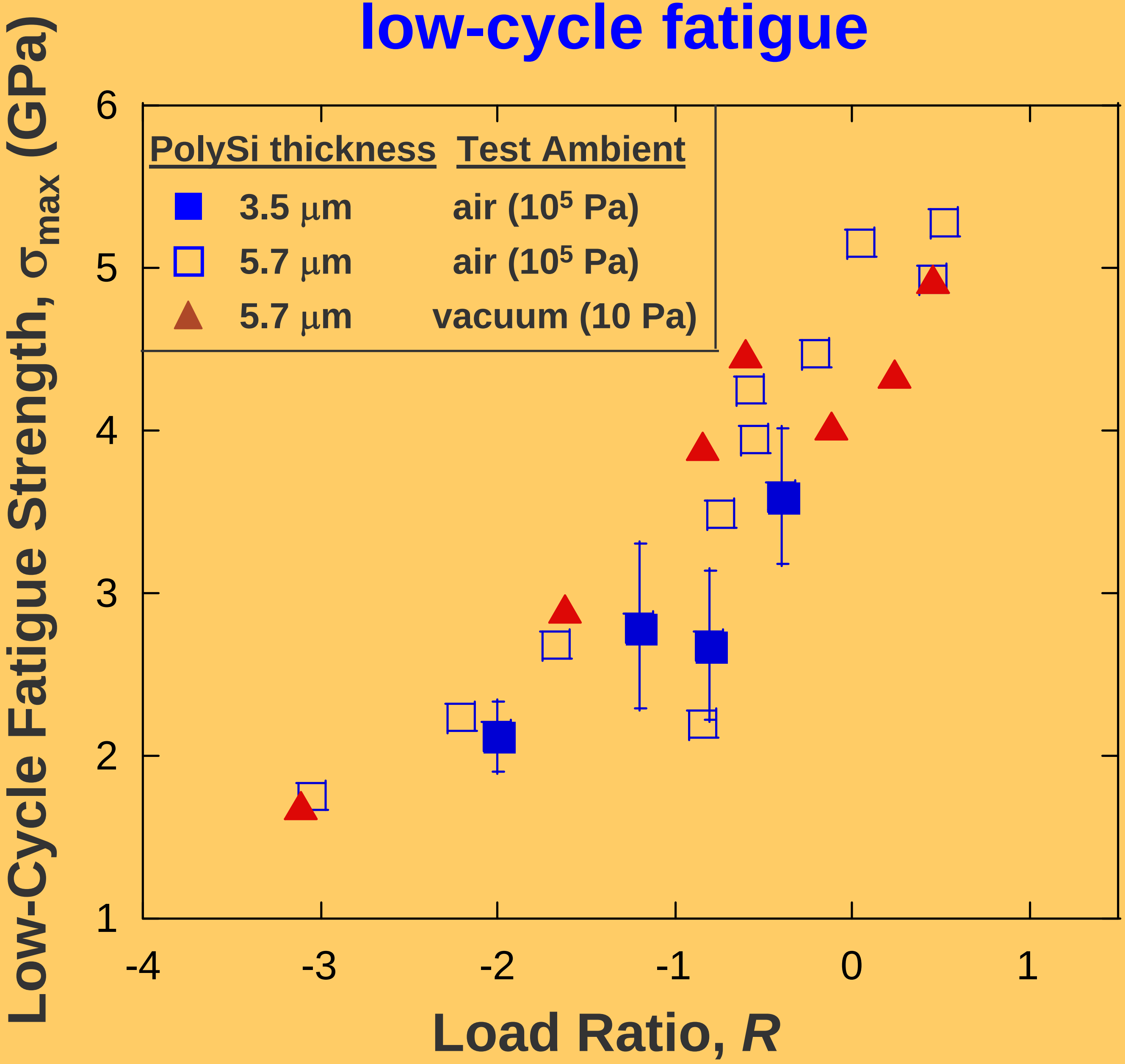
$$R = \frac{\sigma_{min}}{\sigma_{max}}$$



The specimens are given a tensile or compressive bias stress, σ_0, using a DC offset.

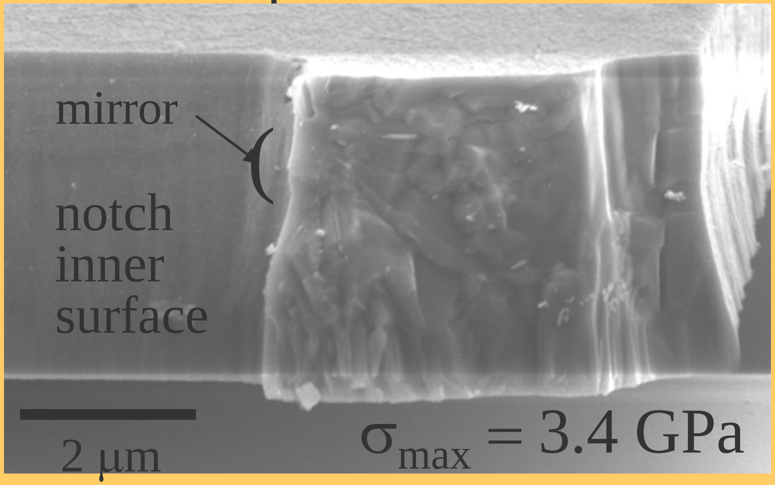
Dynamic Fatigue Results

low-cycle fatigue

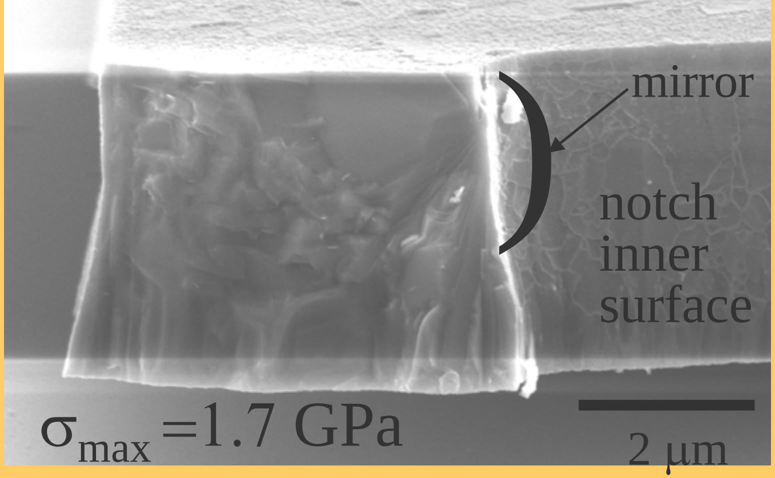


Fractography of Biased Fatigue Specimens

Specimen T



Specimen C



Specimen T was subjected to a high tensile bias stress during resonance and fractured at a σ_{max} of 3.4 GPa.

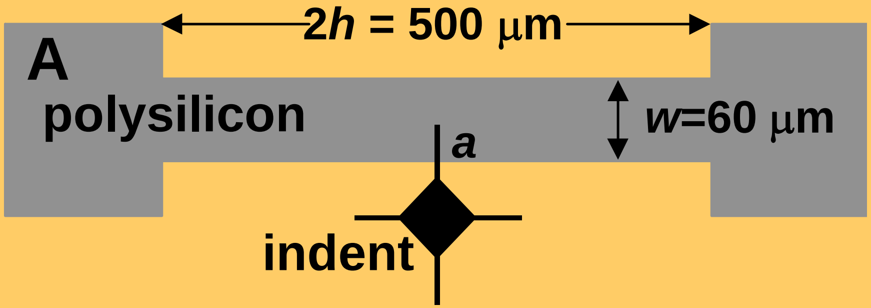
Specimen C was subjected to a high compressive bias stress during resonanc and fractured at a σ_{max} of 1.7 GPa.

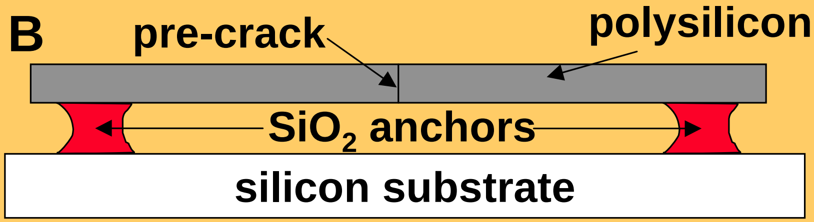
The larger mirror on the fracture surface of Specimen C indicates a larger flaw size at fracture, consistent with the lower σ_{max} and also consistent with K_{crit} = 1.0±0.1 Mpa-m$^{1/2}$.

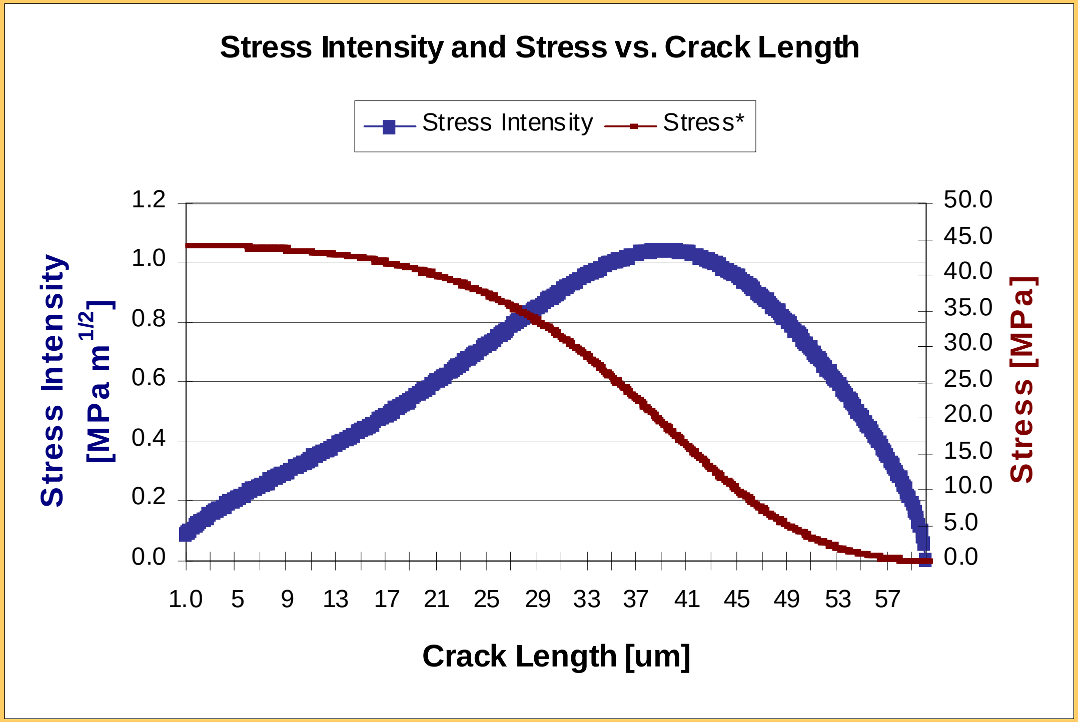
Since the specimens were fabricated from the same polysilicon film, on the same wafer, this is clear evidence of fatigue-induced sub-critical crack growth.

PASSIVE DEVICE ASSOCIATED WITH CONSTANT TENSION

(*Science* 298, 1215-1218, 2002)



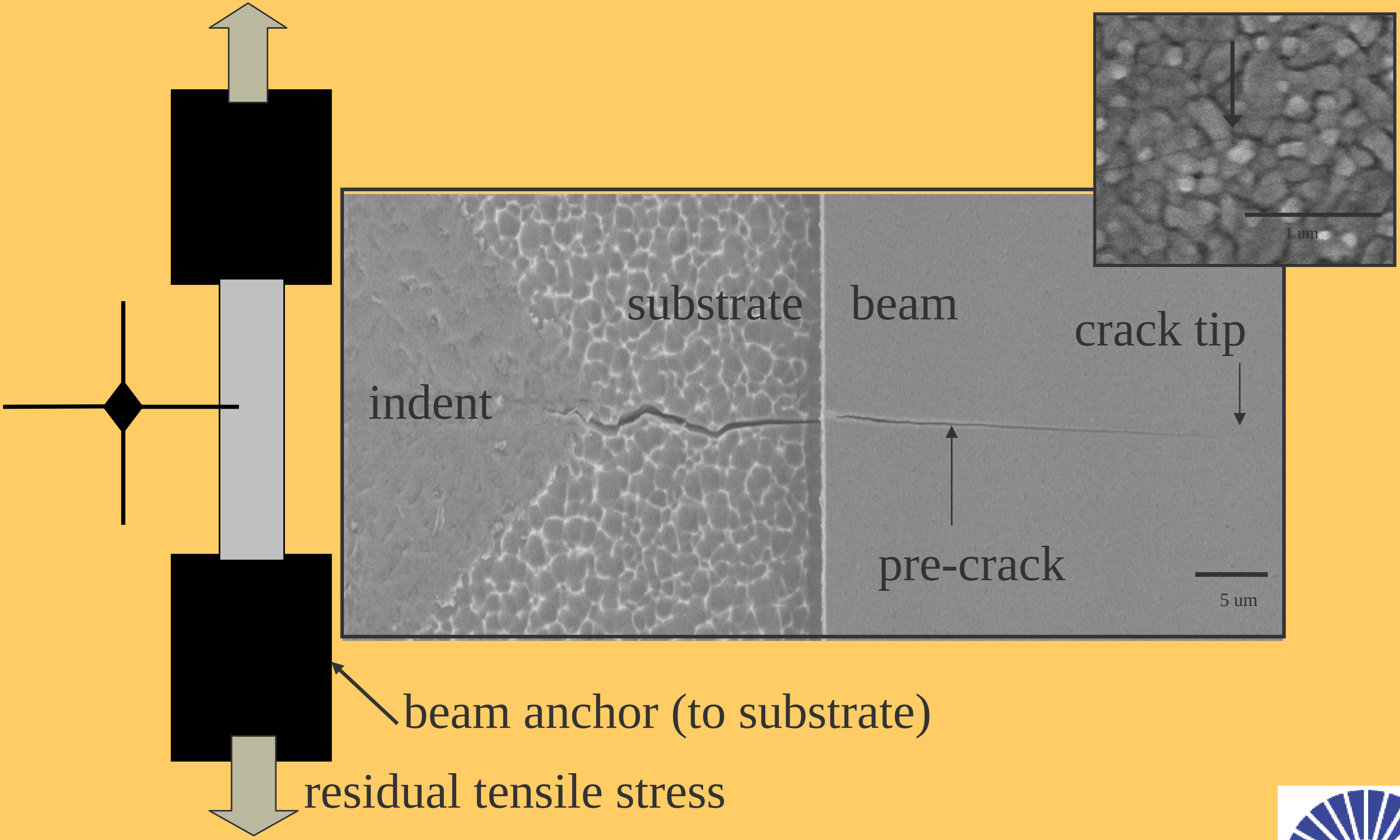




$$K = \sigma^* \sqrt{\pi a}\, F(\alpha)$$

$$\sigma^* = \sigma_{residual}/(1+4aV(\alpha)/2h)$$

INDENTATION CRACK



substrate

beam

crack tip

indent

pre-crack

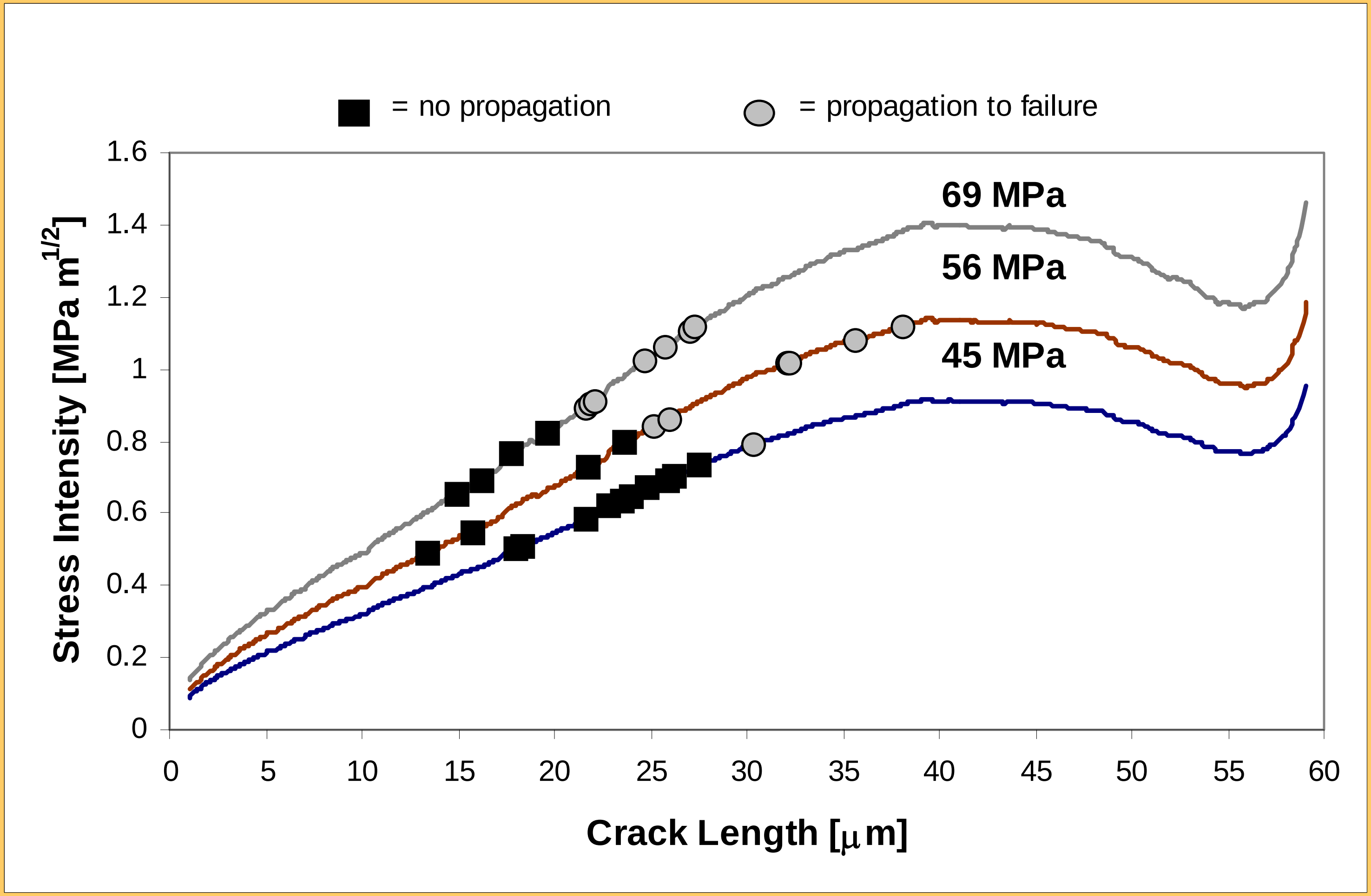
5 um

1 um

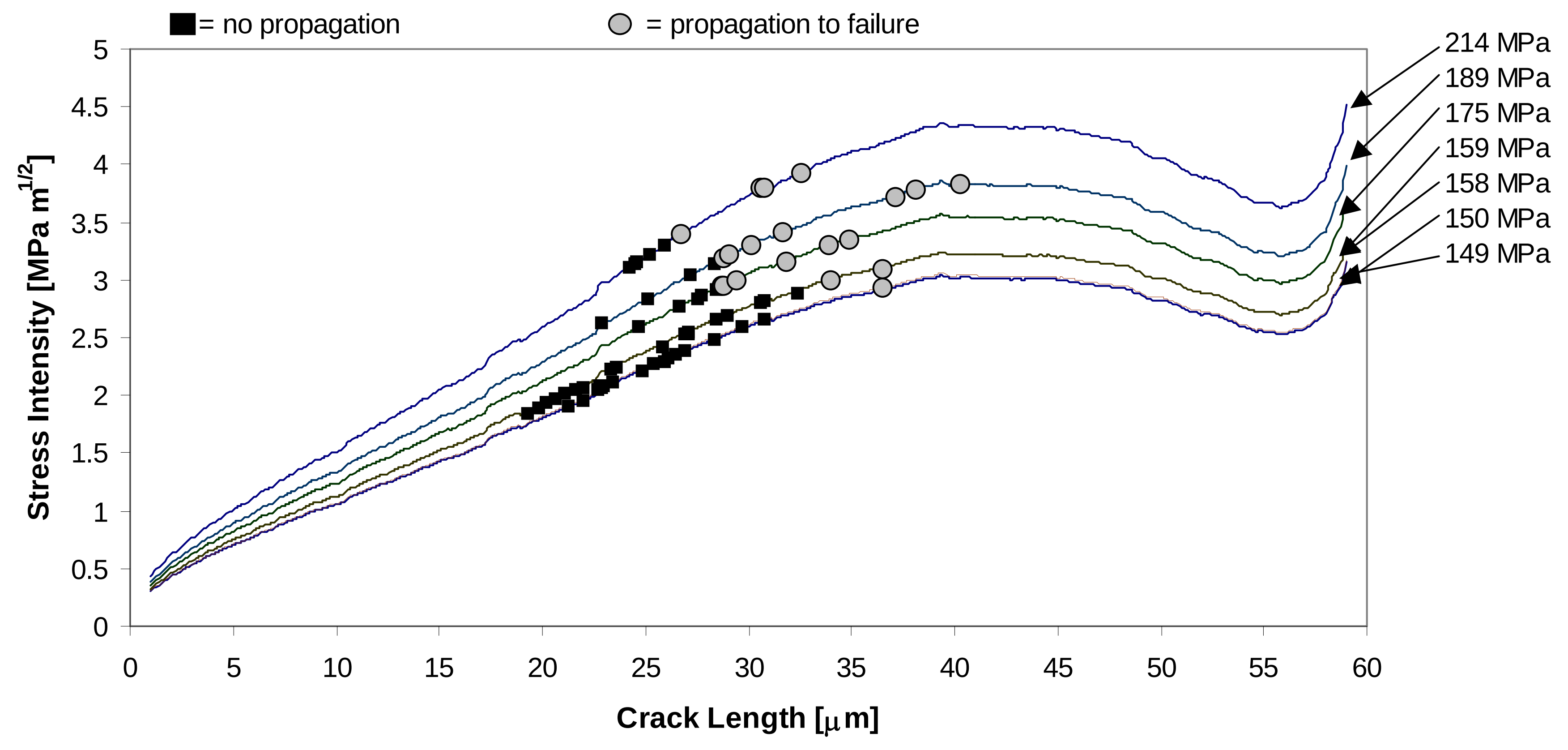
beam anchor (to substrate)

residual tensile stress

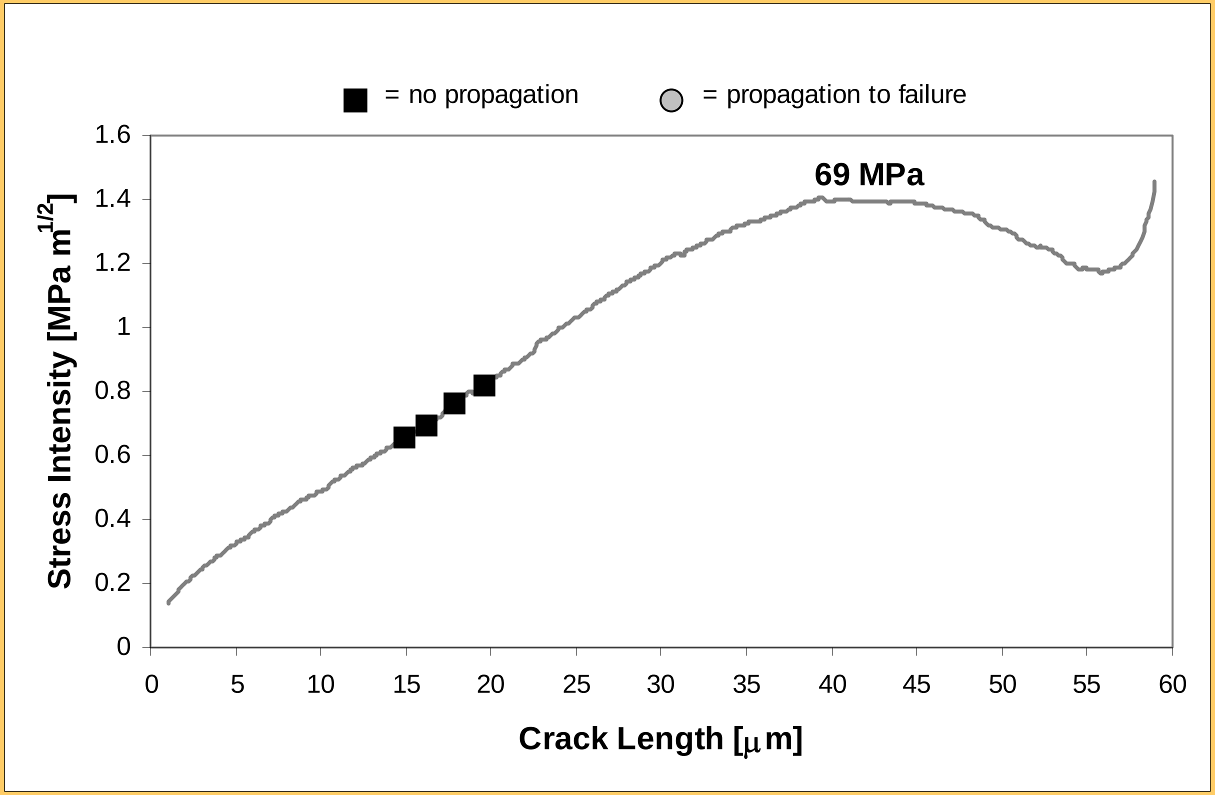
FINE-GRAINED POLYSILICON FRACTURE TOUGHNESS



POLYCRYSTALLINE SILICON CARBIDE FRACTURE TOUGHNESS



FINE-GRAINED SILICON
STATIC FATIGUE STUDY
90% RH



K between 0.62- 0.86 MPa-m$^{1/2}$

No growth in 30 days

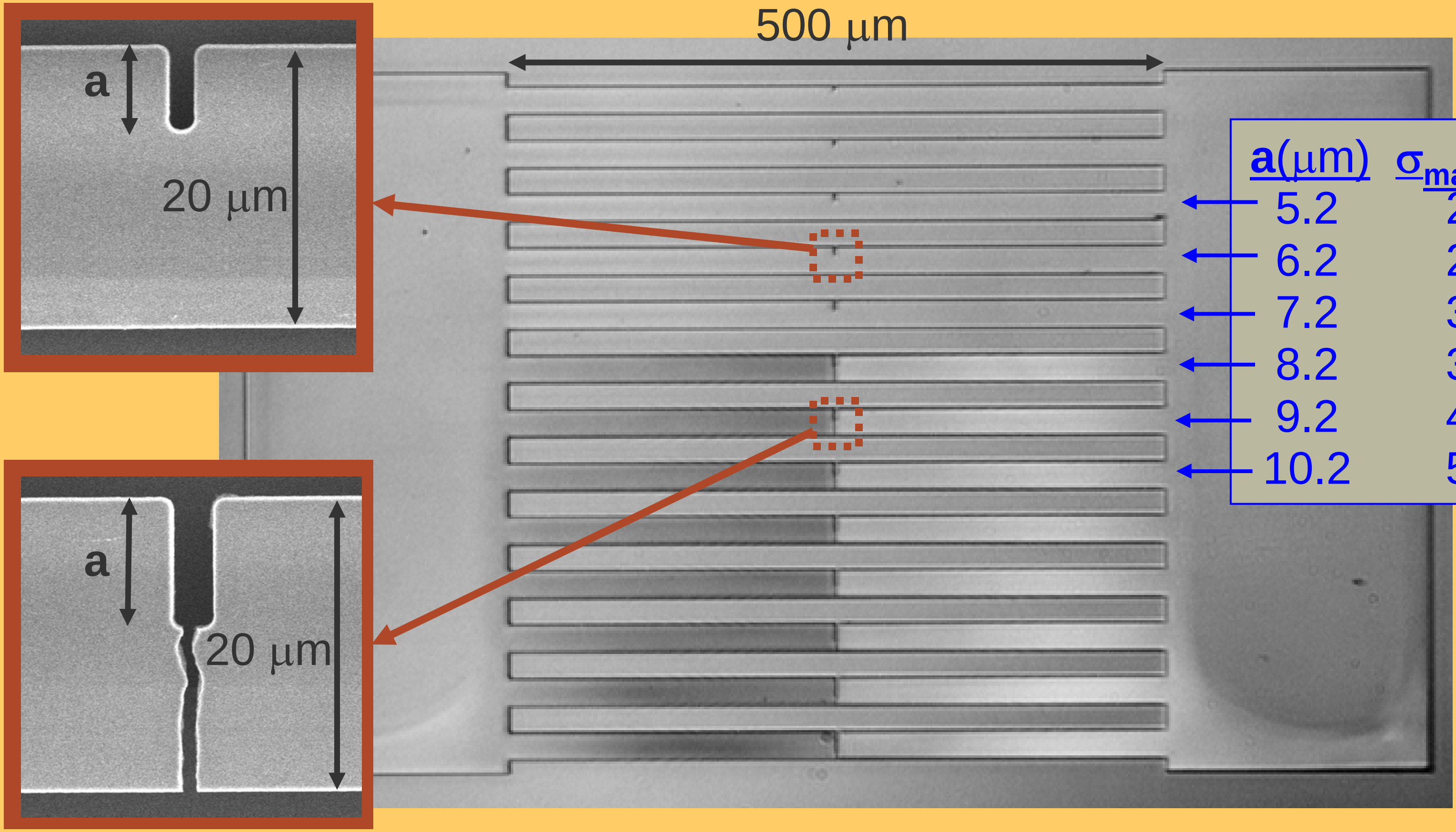
V< 3.9 x10^{-14} m/s

Same results for eight multipoly specimens

Static Fatigue Experiment

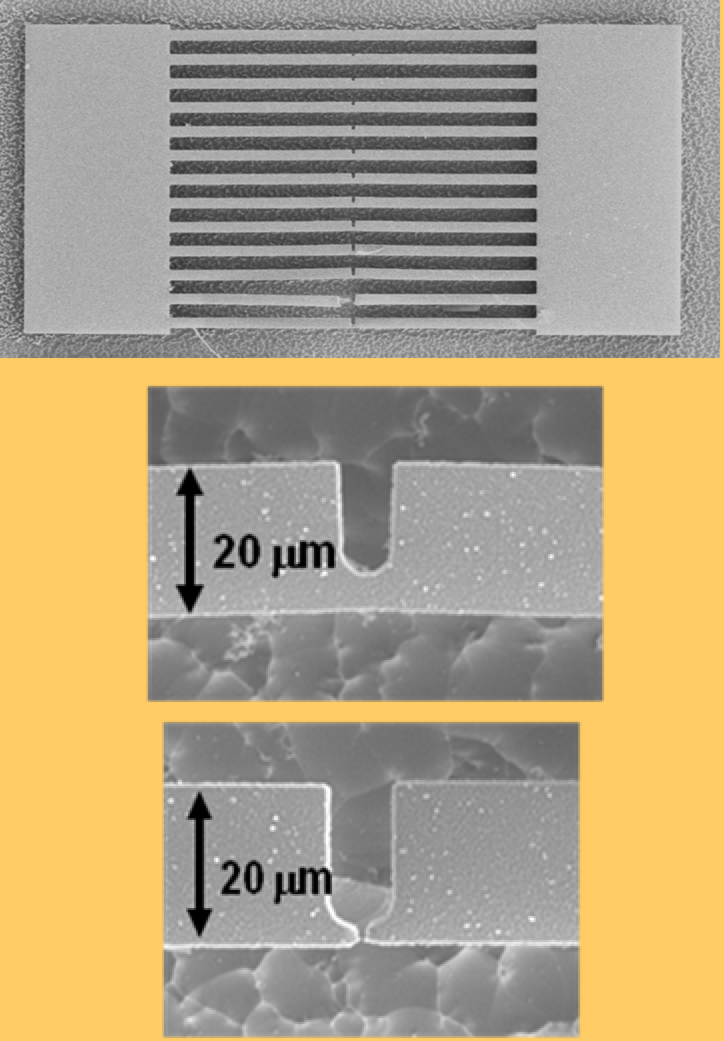
Notched Tensile Beams

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

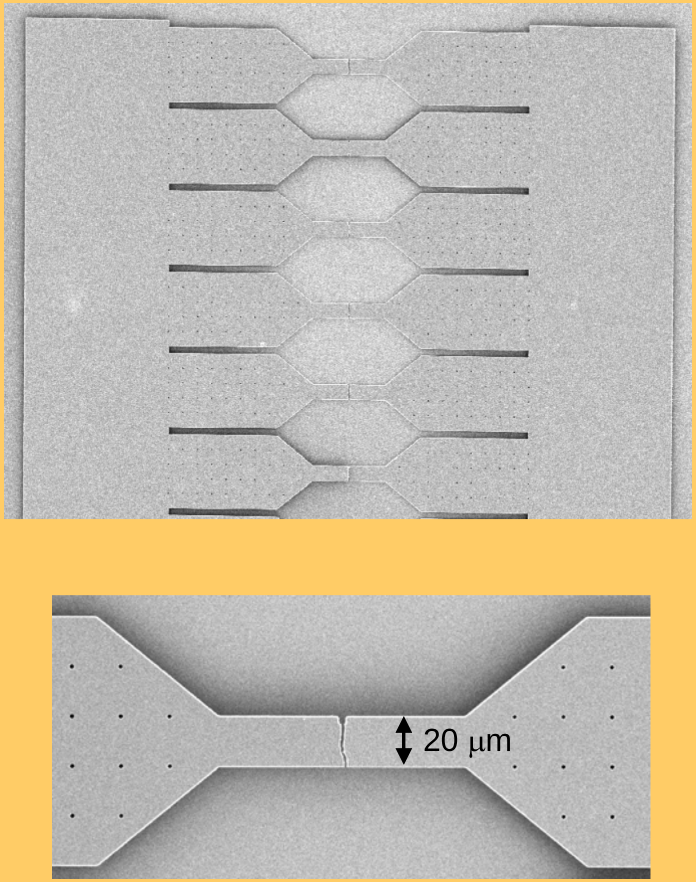


a(μm)	σ_{max}(GPa)	broken
5.2	2.3	0/14
6.2	2.8	0/14
7.2	3.4	0/14
8.2	3.9	11/14
9.2	4.5	14/14
10.2	5.0	14/14

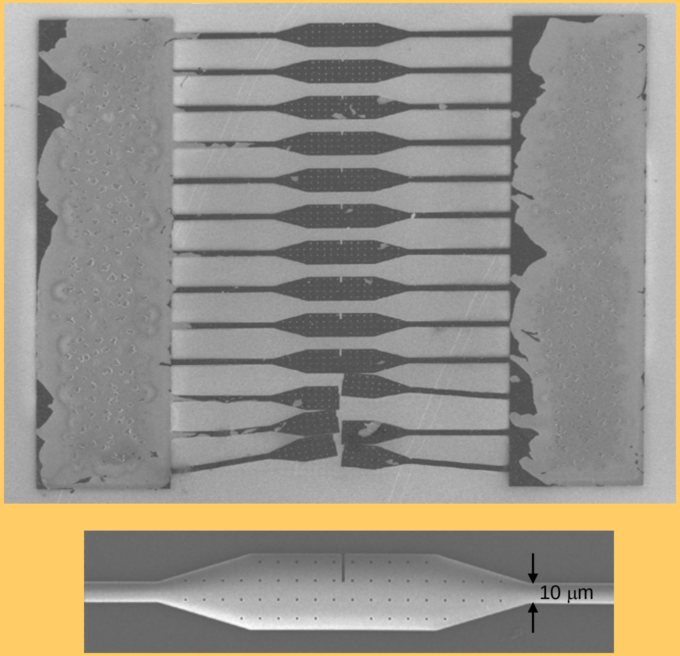
After 200 hrs in 90% humidity → no additional beams broke



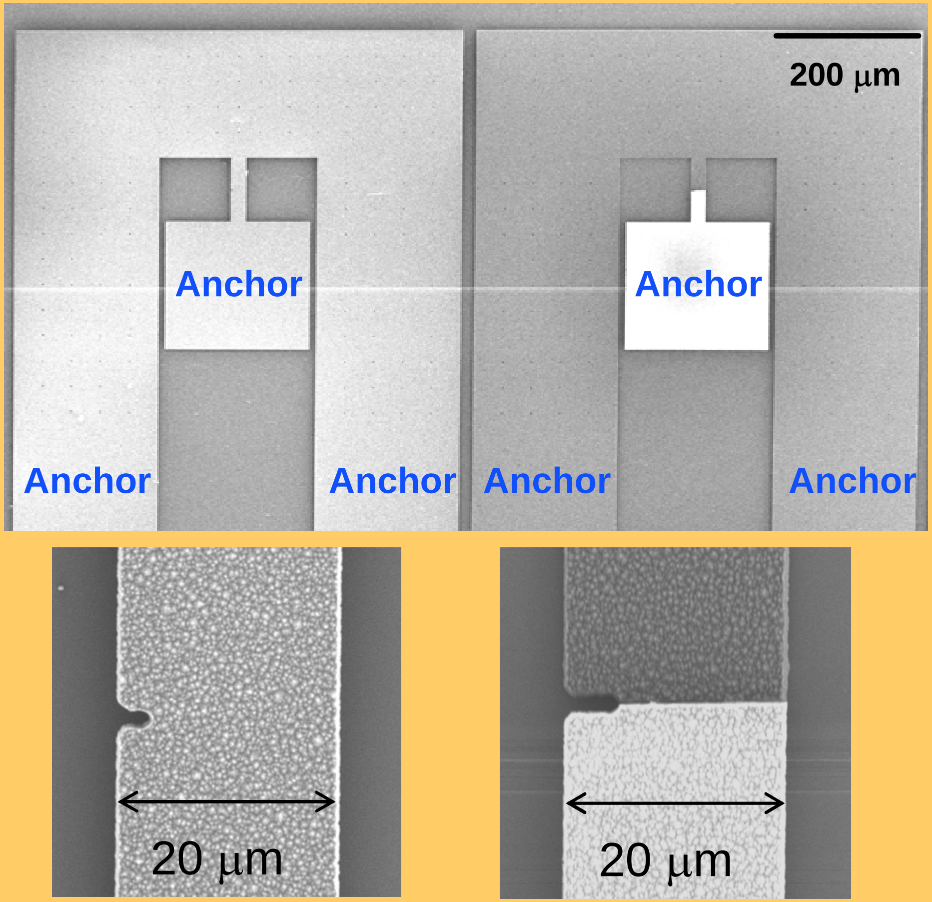
Sputtered Aluminum



Small Residual Tension

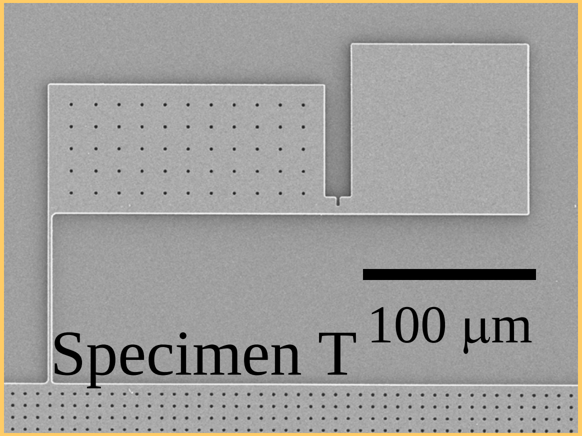


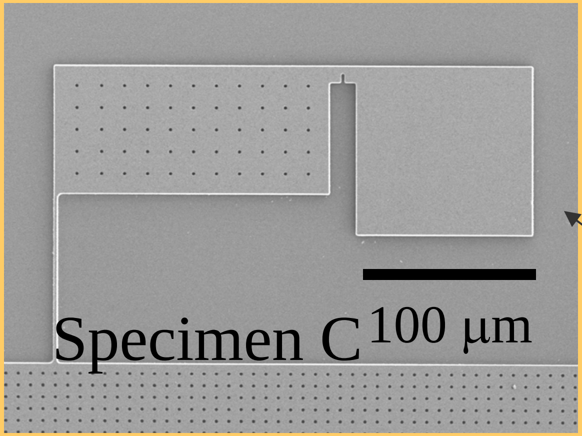
Large Residual Tension
Silicon Nitride



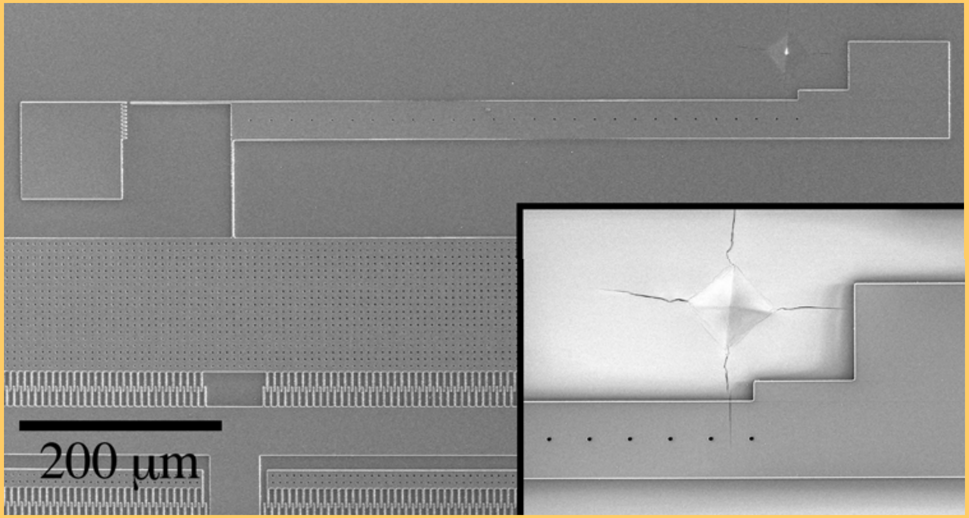
Residual Compression
Columnar Polysilicon

VARIATIONS ON A THEME



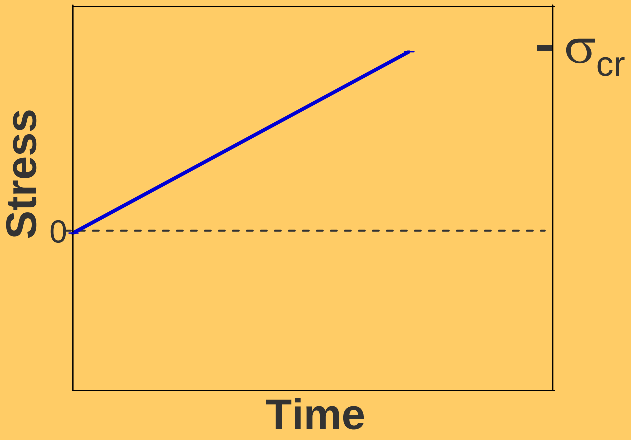


R-ratio
and mean
stress
effects

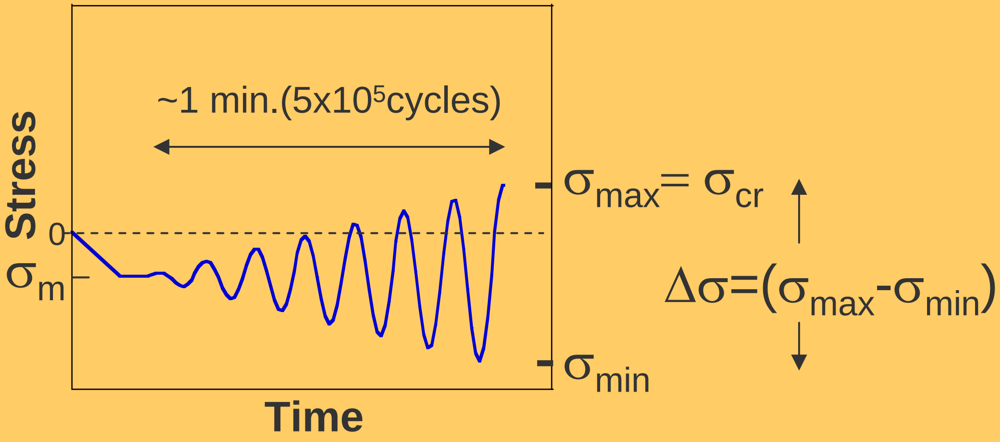


Schematic Bend Strength Tests

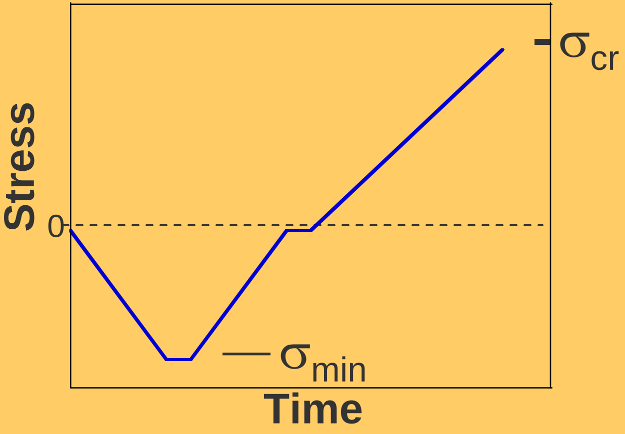
Monotonic



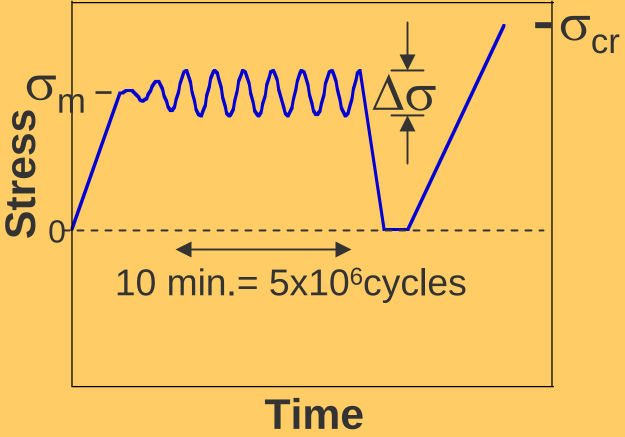
Increasing Amplitude Fatigue



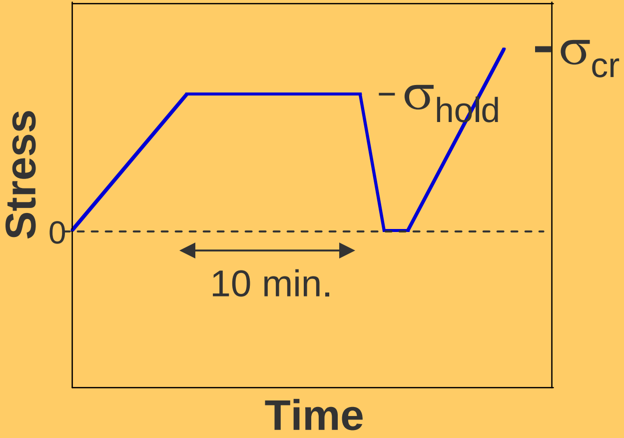
1 Comp/Mono



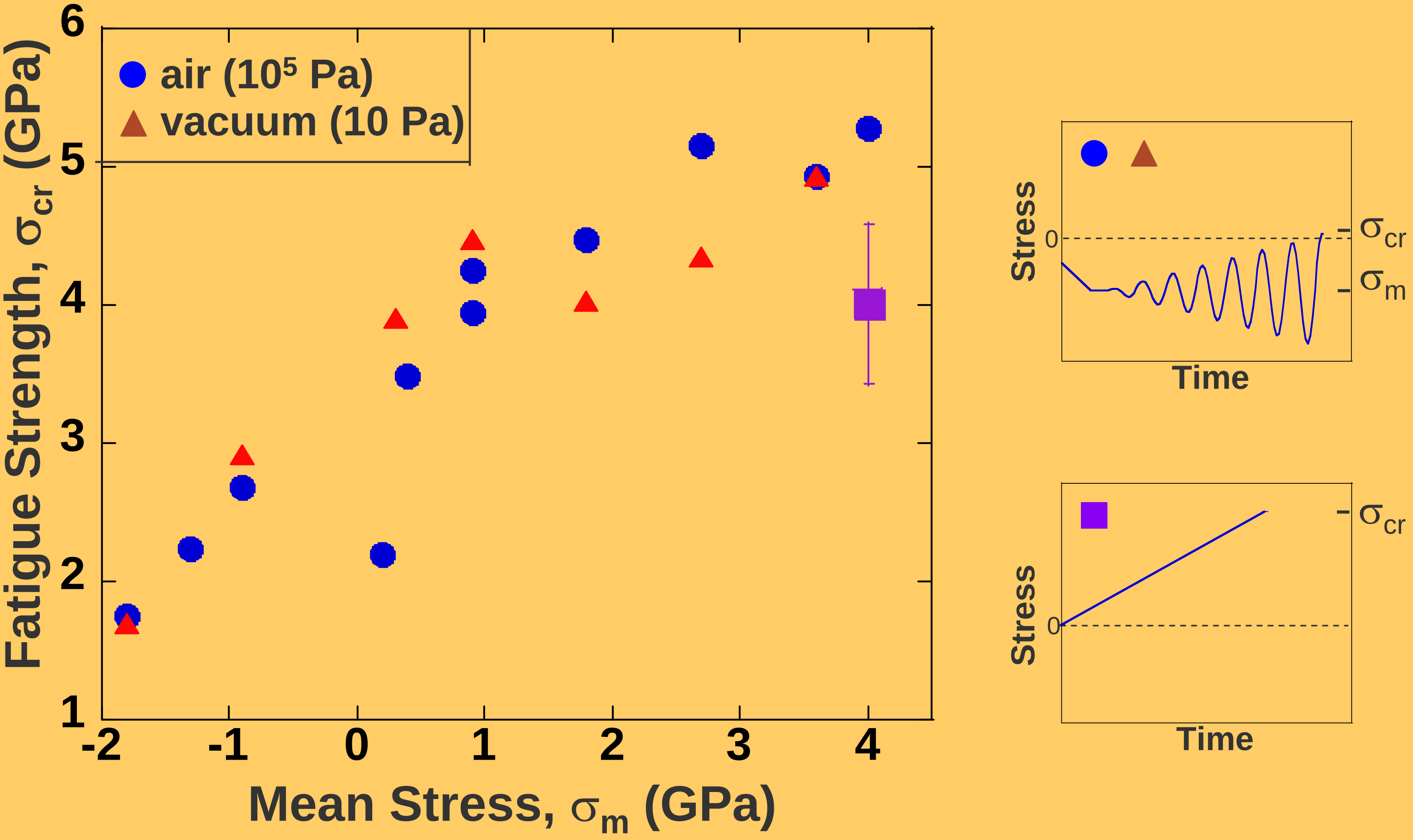
Fixed Δσ Fatigue/Mono



High T Hold/Mono

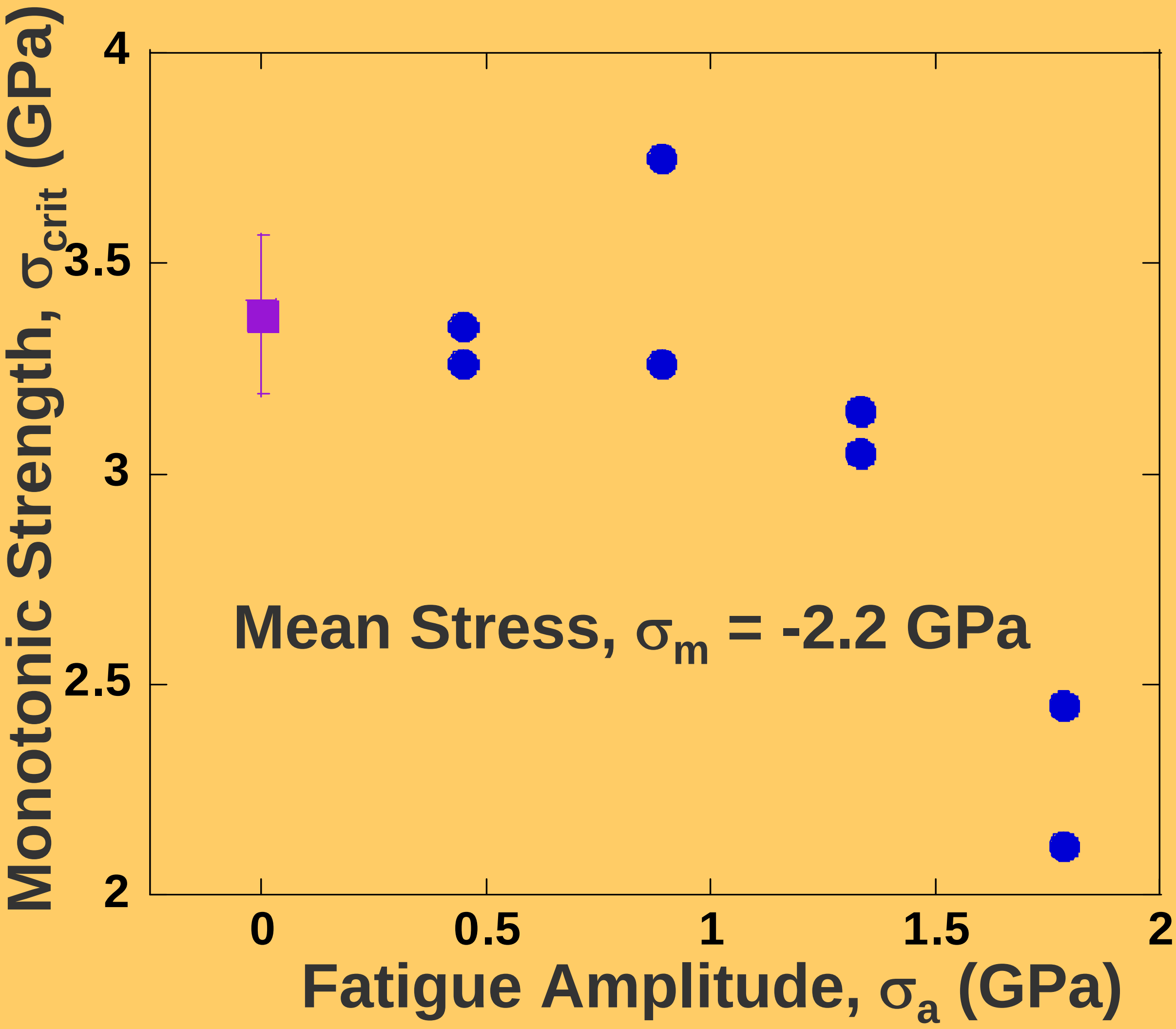


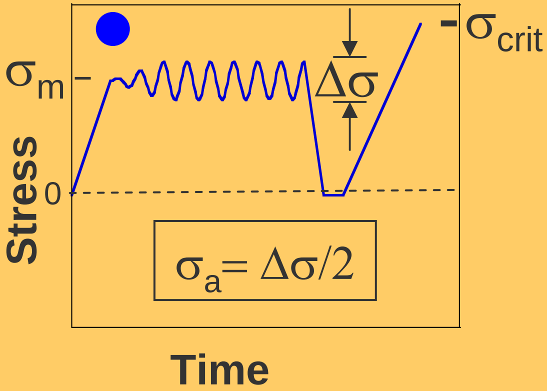
Low-Cycle Fatigue

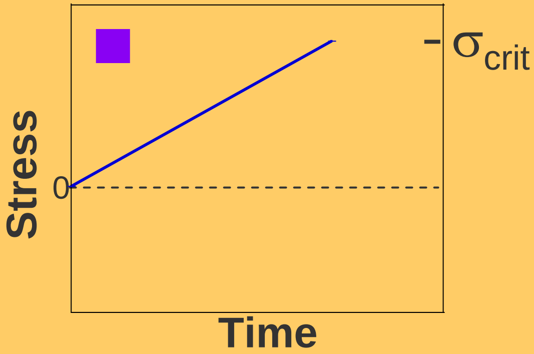


Monotonic Bend Strength

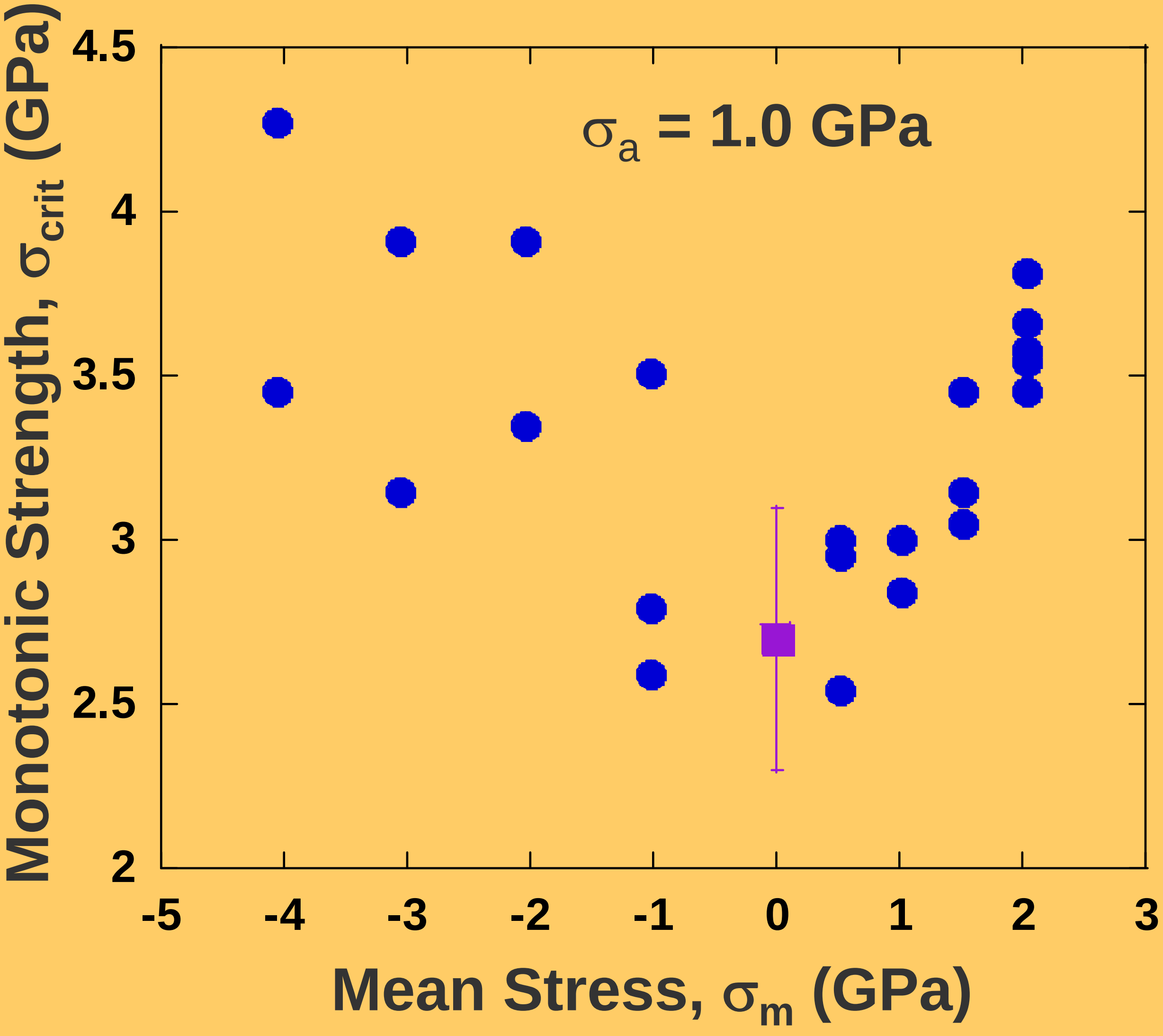
after cycling with a fixed mean stress

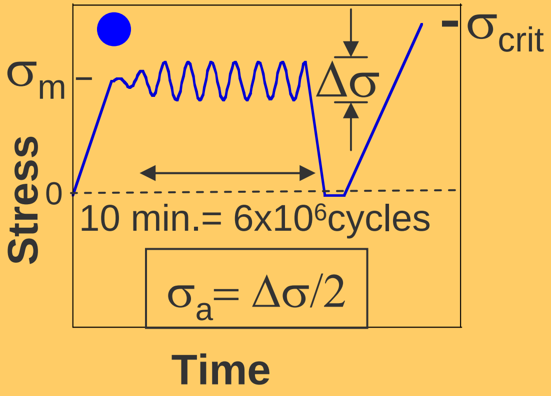


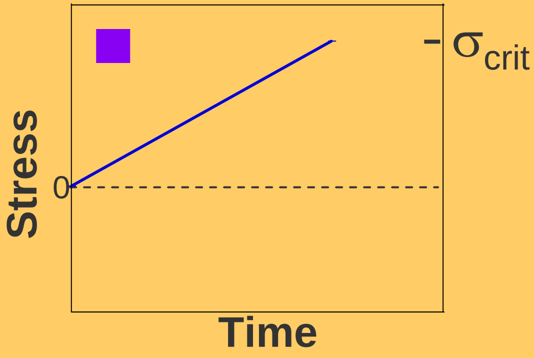




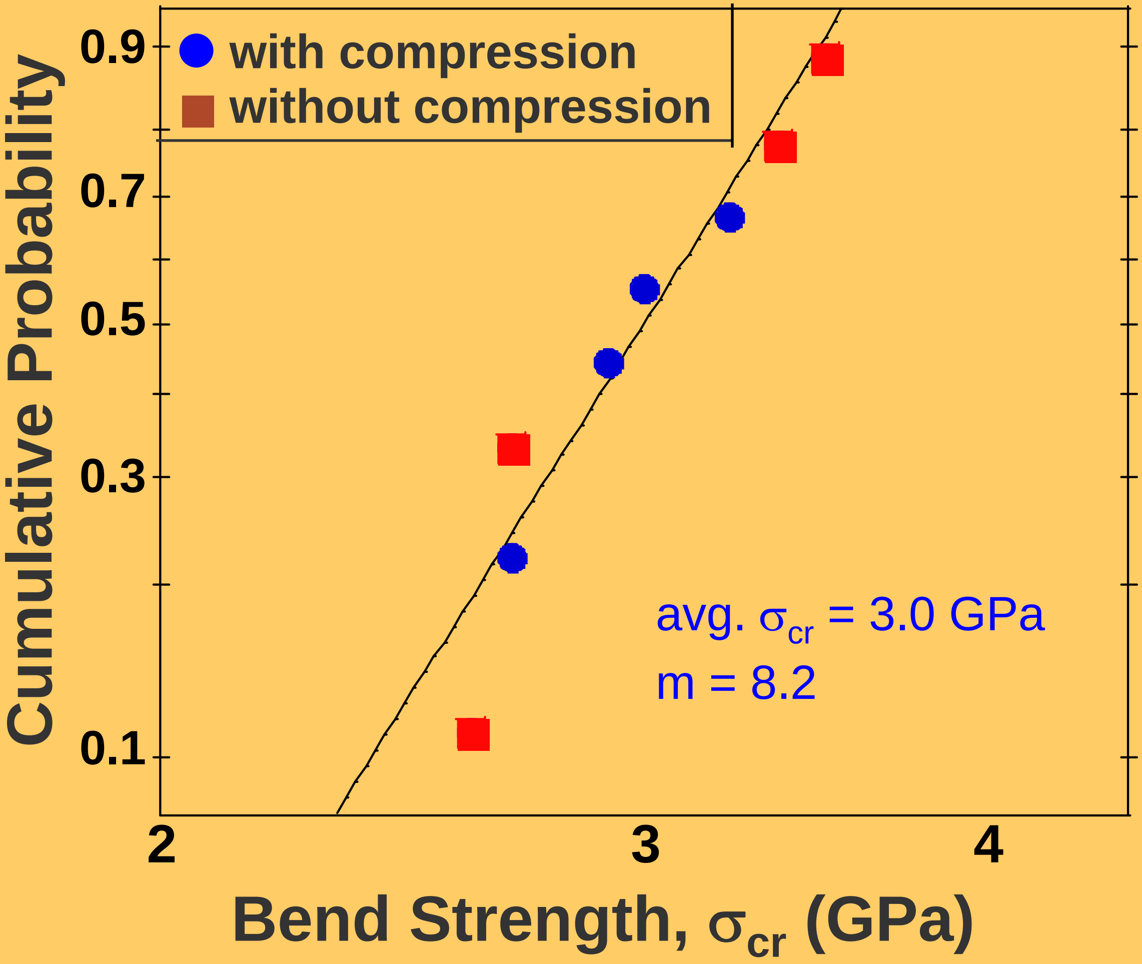
Monotonic Bend Strength
after cycling with a fixed (low) amplitude

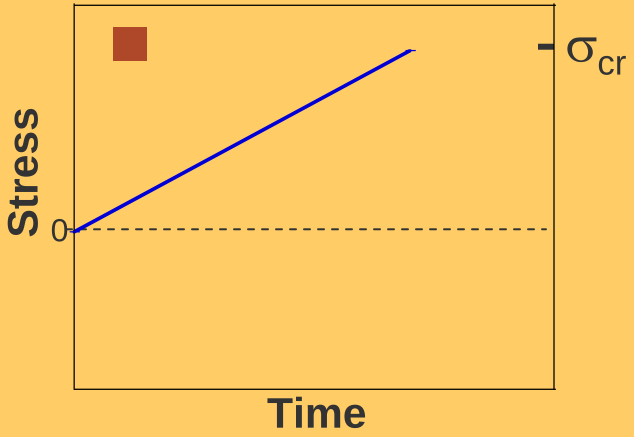


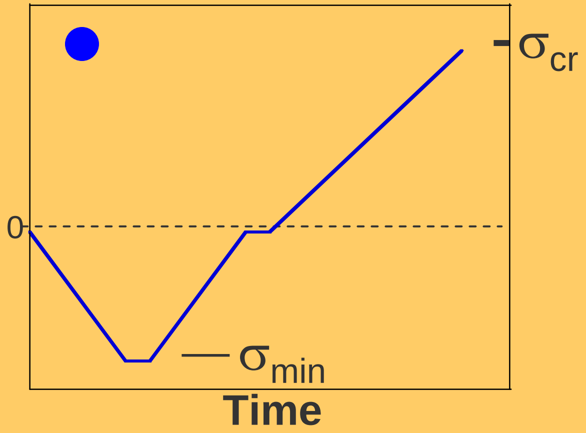




Monotonic Bend Strength
with/without initial compression



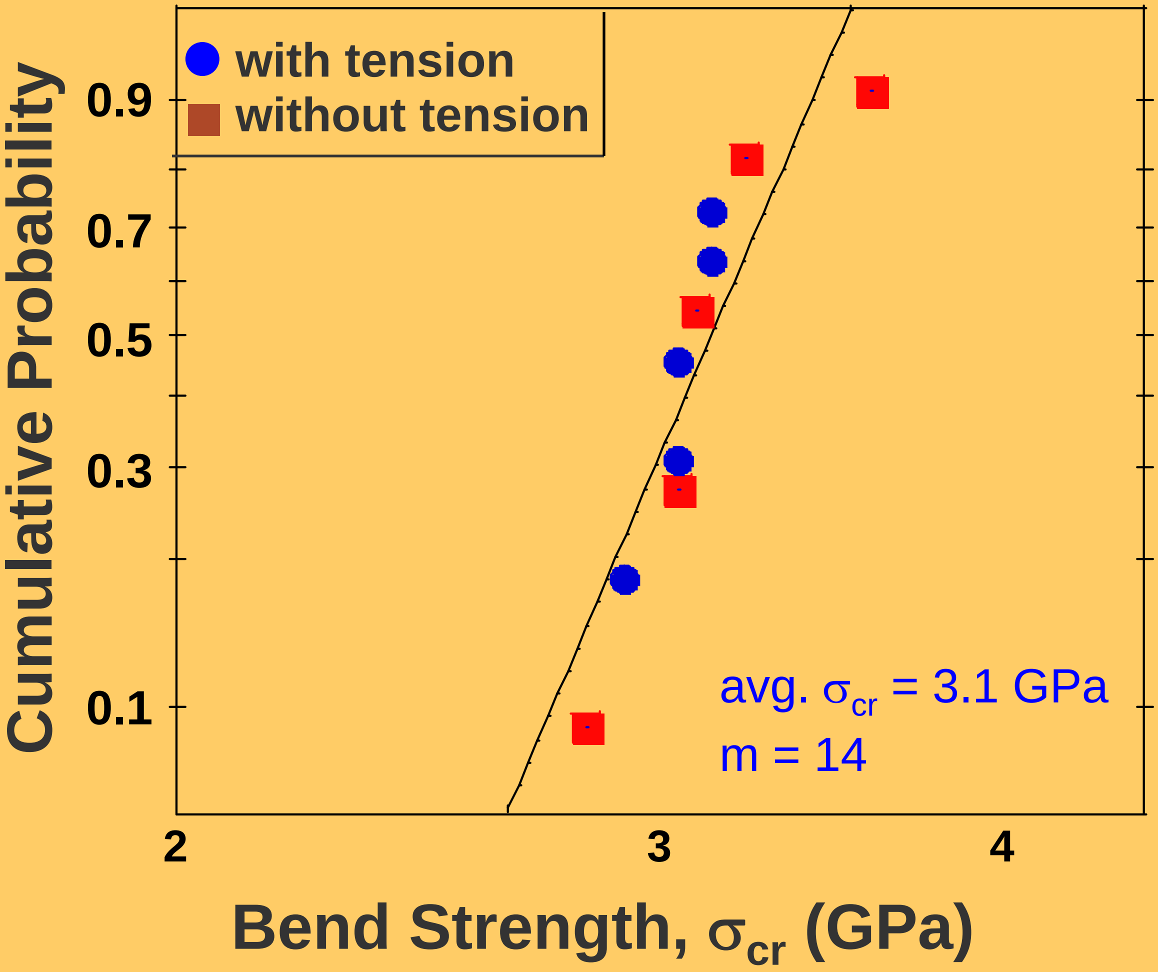


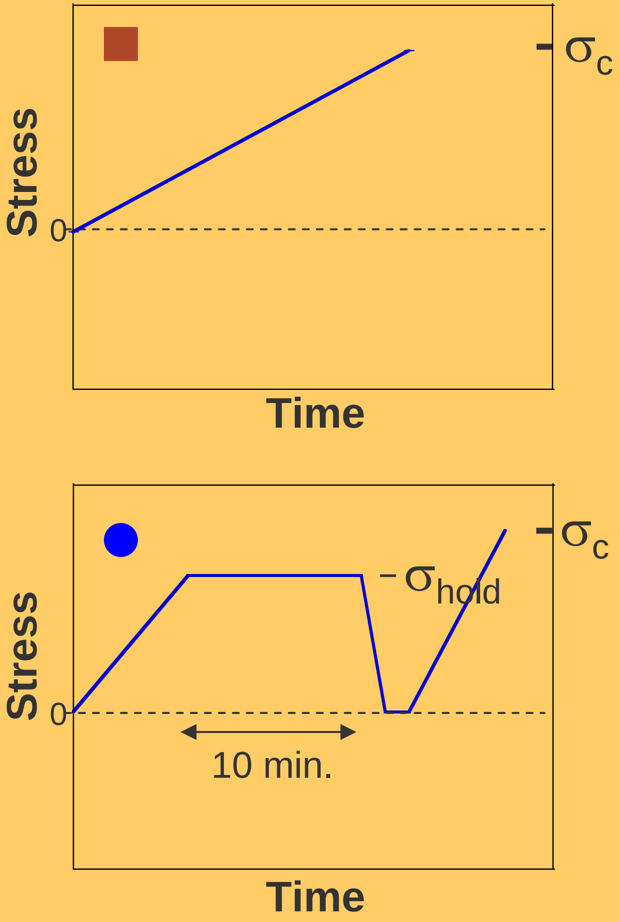


σ_{min} = -4.5 GPa

Monotonic Bend Strength

with/without tensile hold

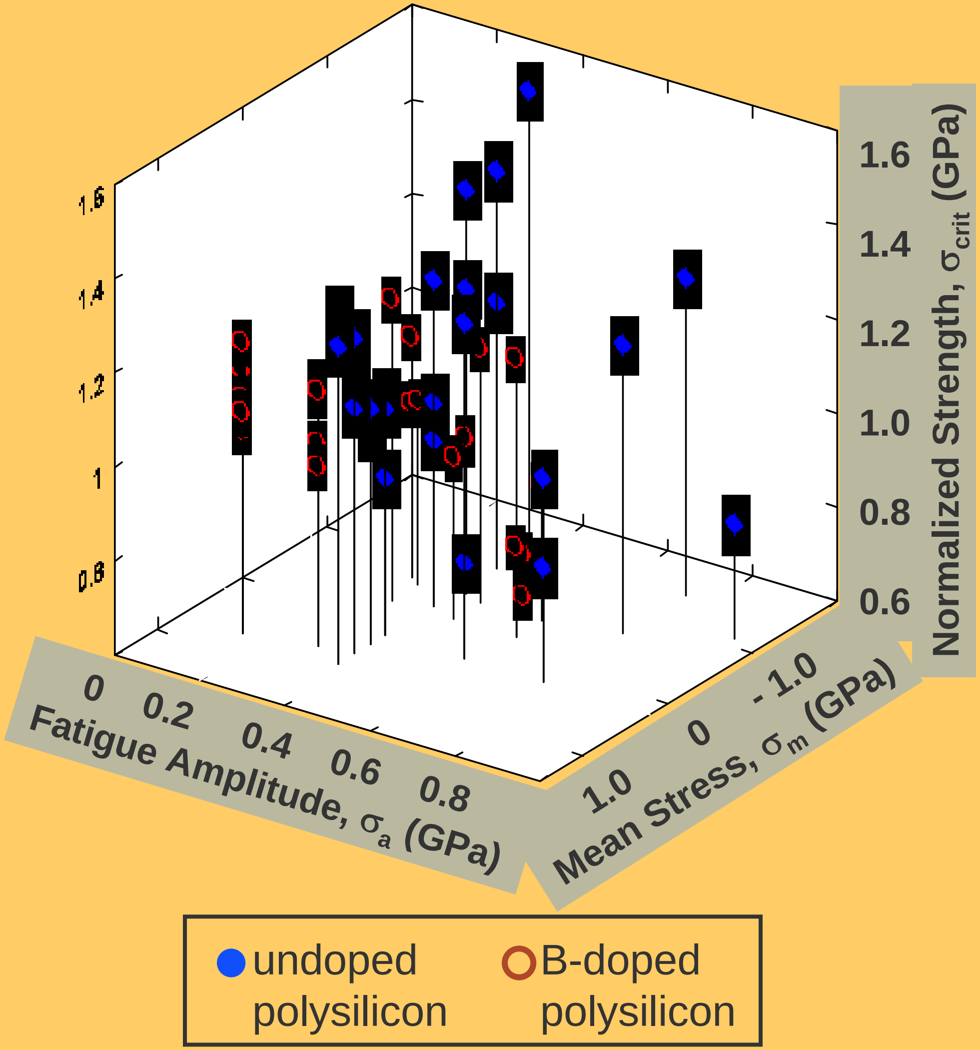




σ_{hold} = 2.7 GPa

Effects on Monotonic Bend Strength

of mean stress σ_m, and fatigue amplitude σ_a



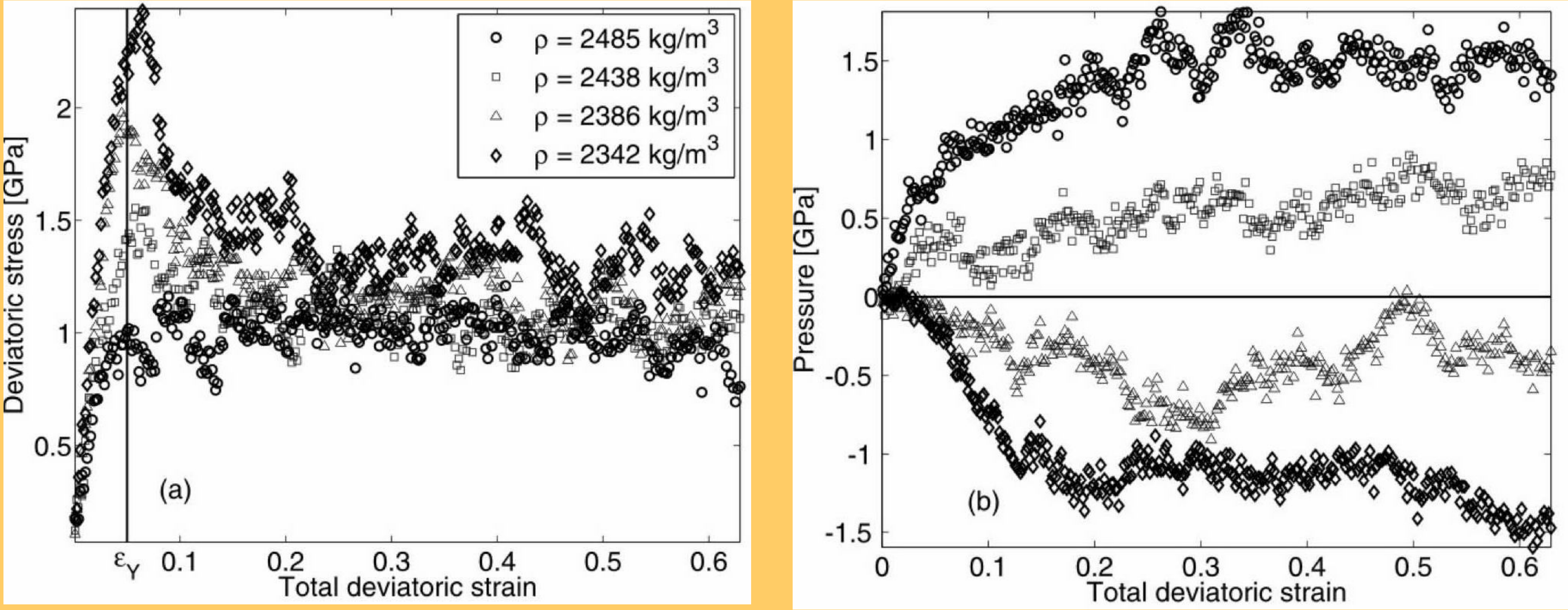
Mean Stress, σ_m (GPa)		low	high
compressive	high	strengthening	weakening
	low	no effect	
tensile	low	no effect	weakening
	high	strengthening	(not measured)

Fatigue Amplitude, σ_a (GPa)

Mechanisms?

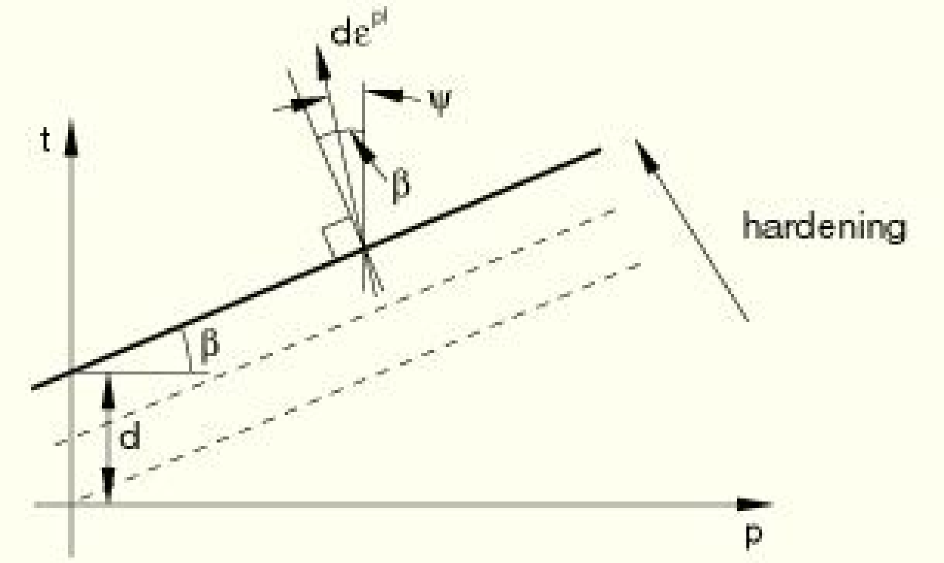
- Phase transformation?
- Microcracking?
- Dislocations?
- Plasticity at grain boundaries?

Plastic flow in amorphous silicon
(M. J. Demkowicz and A. S. Argon)



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

Drucker Prager Model



$$F = t - q\tan\beta - d = 0$$

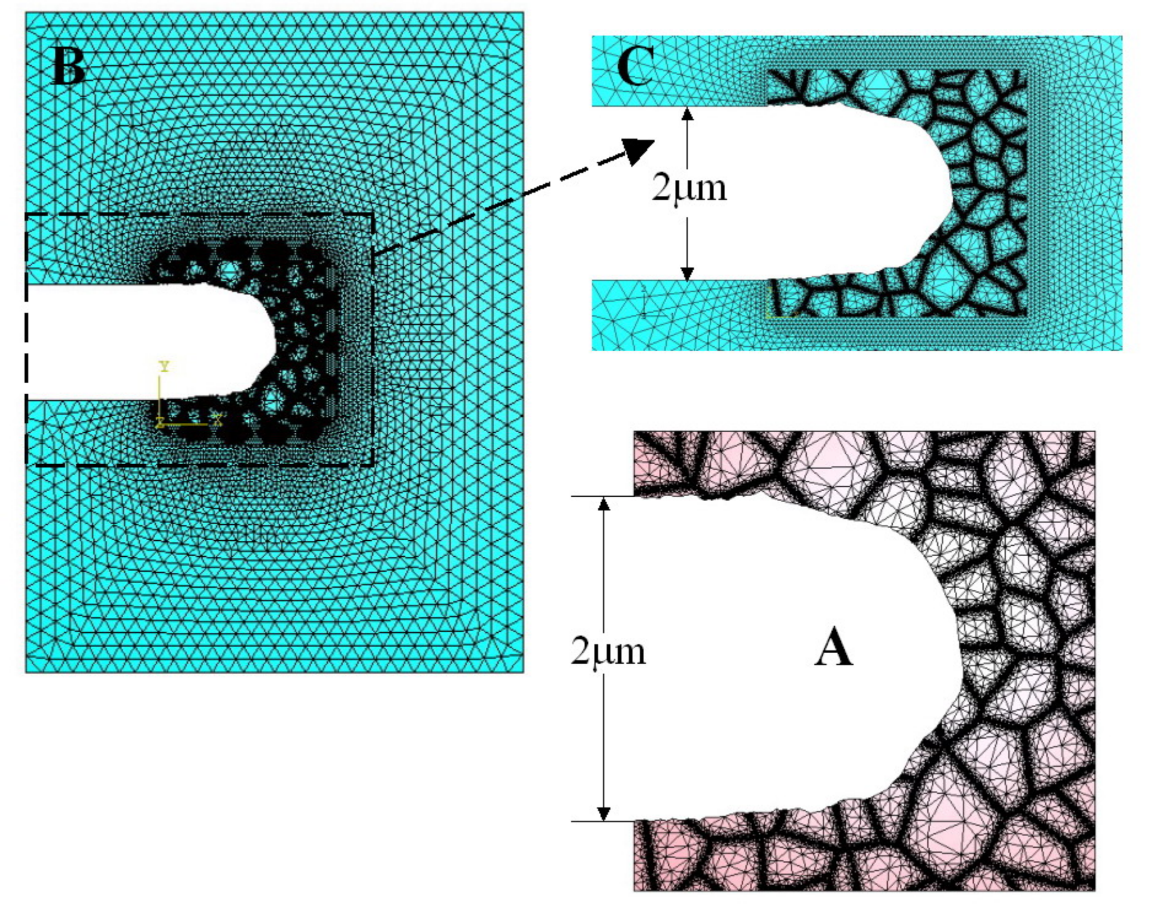
$$G = t - q\tan\psi$$

$$q = \sqrt{\frac{3}{2}(S:S)},\quad S = \sigma + pI$$

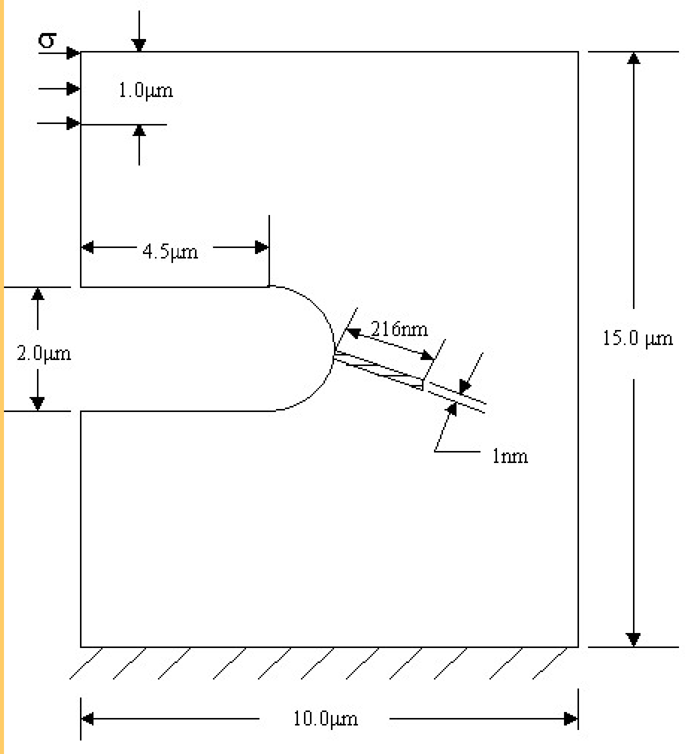
$$d\varepsilon^{p}{}_{ij} = d\lambda\frac{\partial G}{\partial\sigma_{ij}}$$

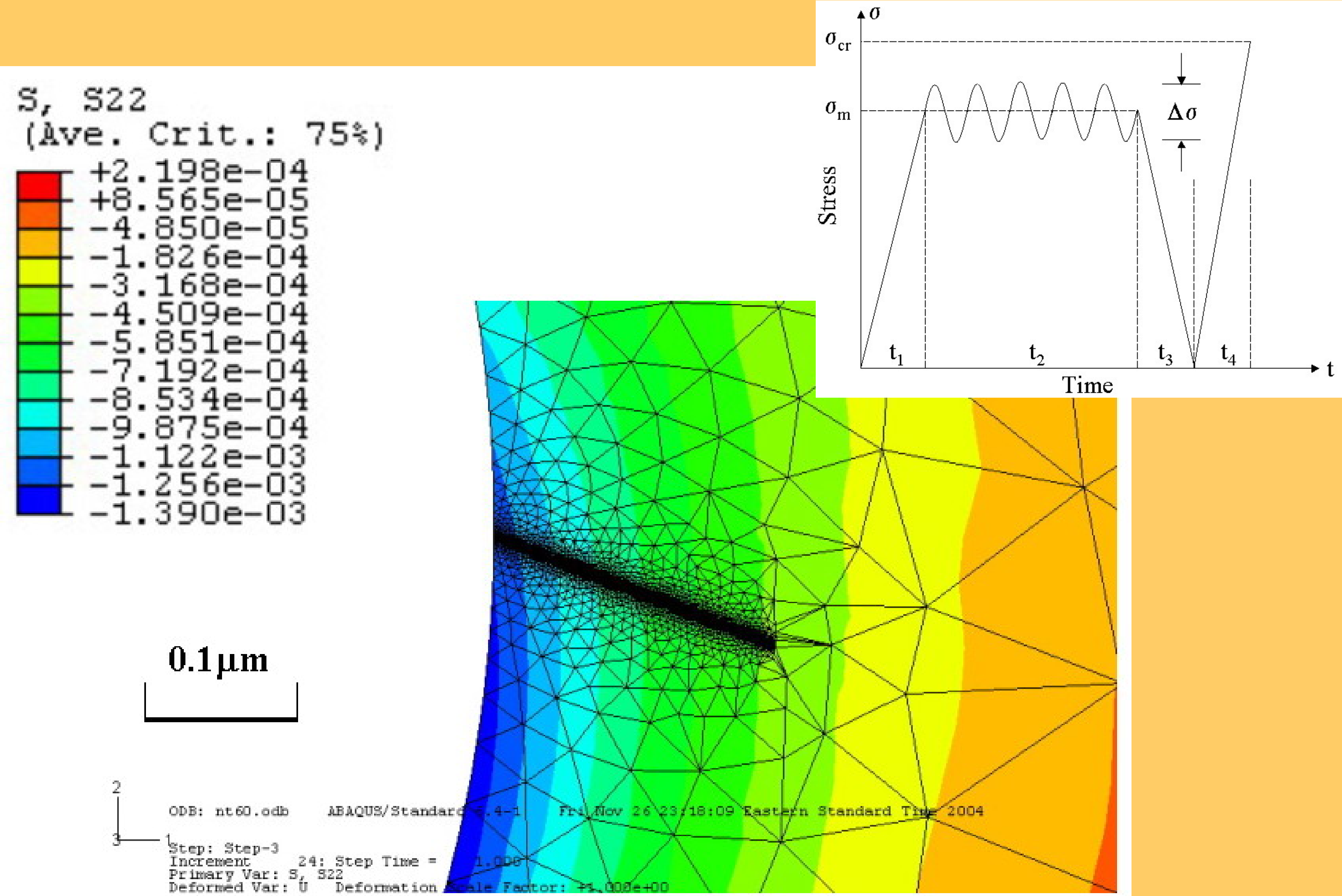
$$d = \sqrt{3}\tau$$

Poisson-Voronoi Local/Global Modeling



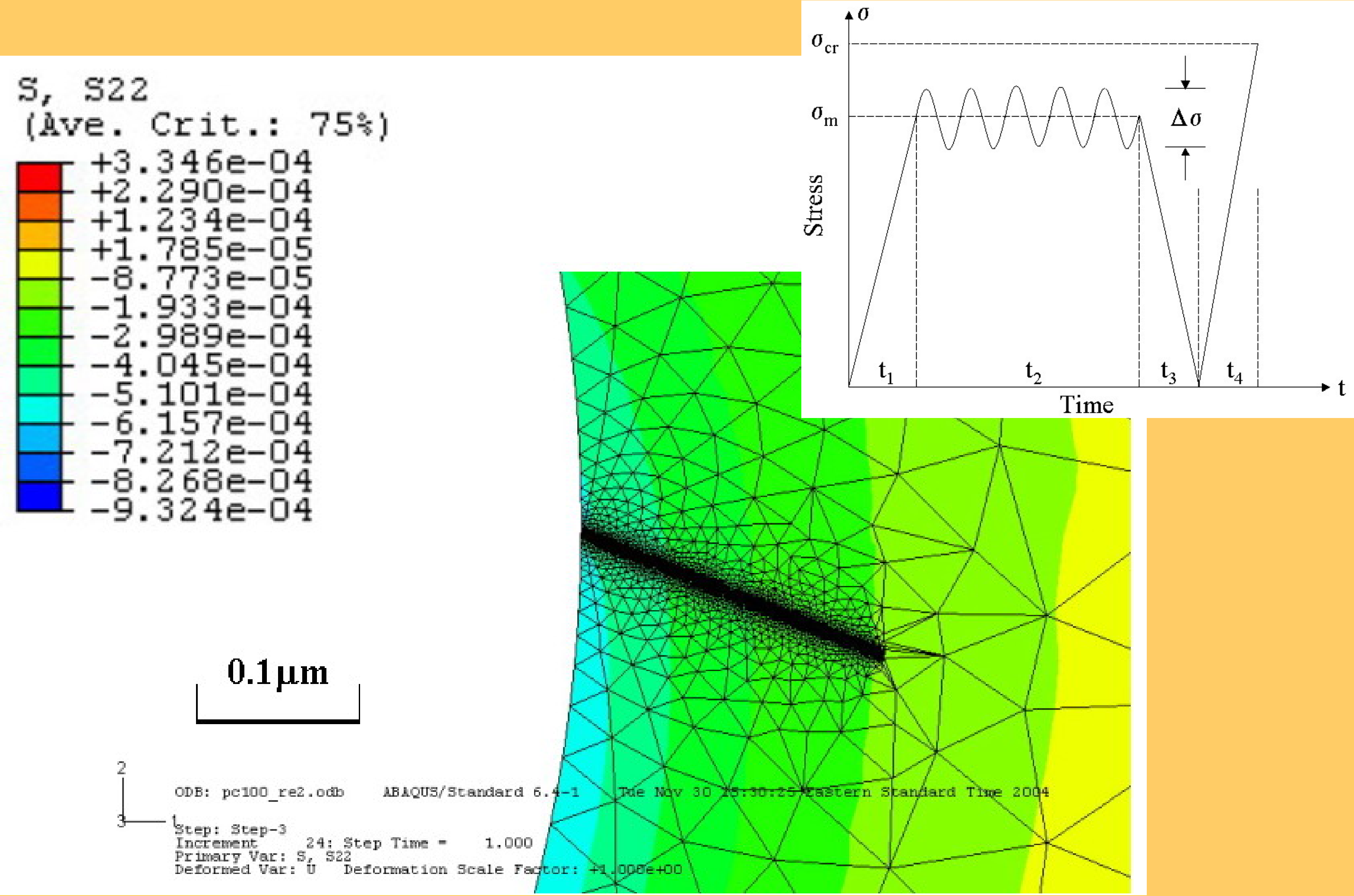
Model for Large Number of Cycles





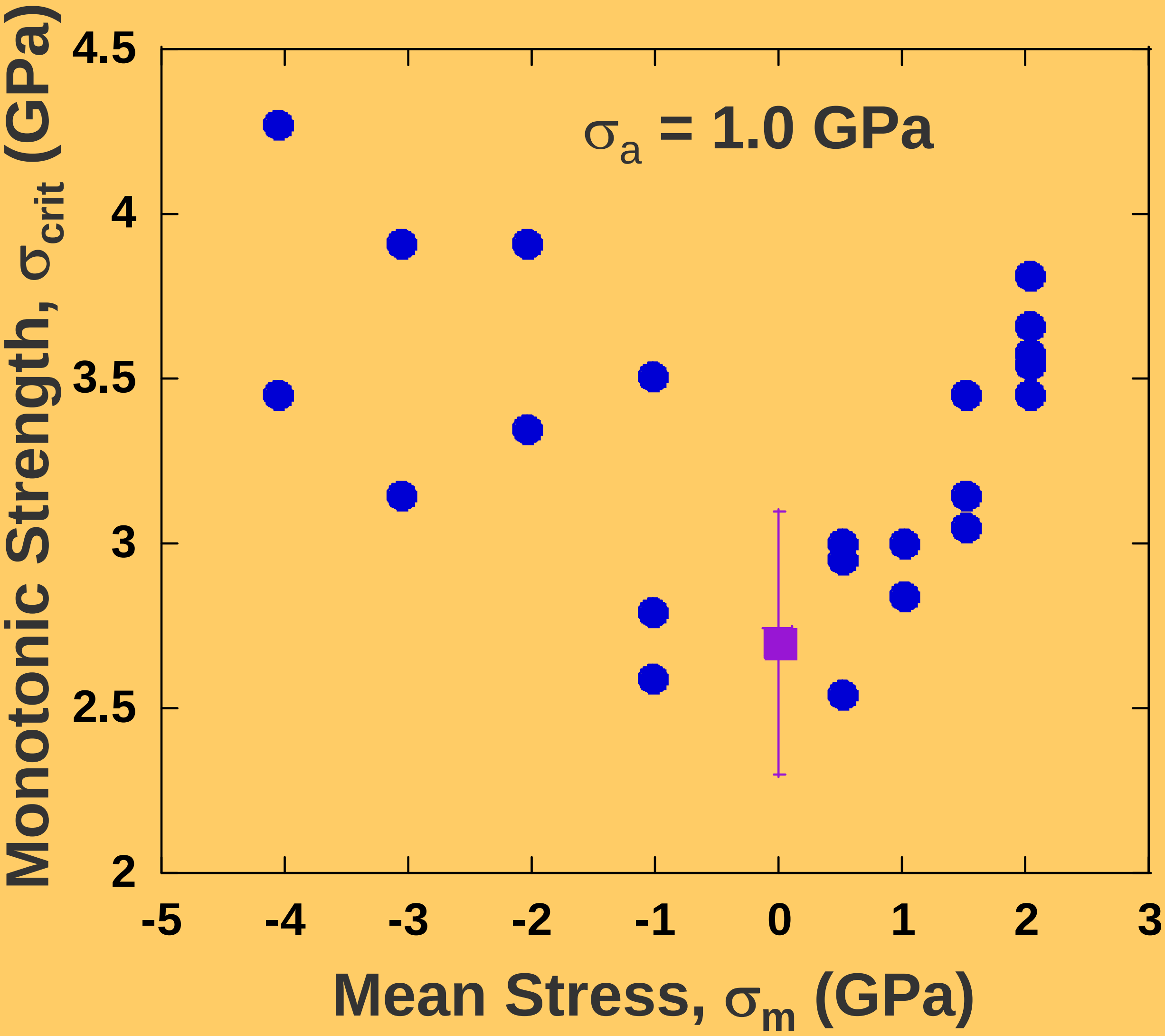
Low Δσ and high σ_m (σ_m = 2.0GPa, Δσ =2.0GPa)

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)

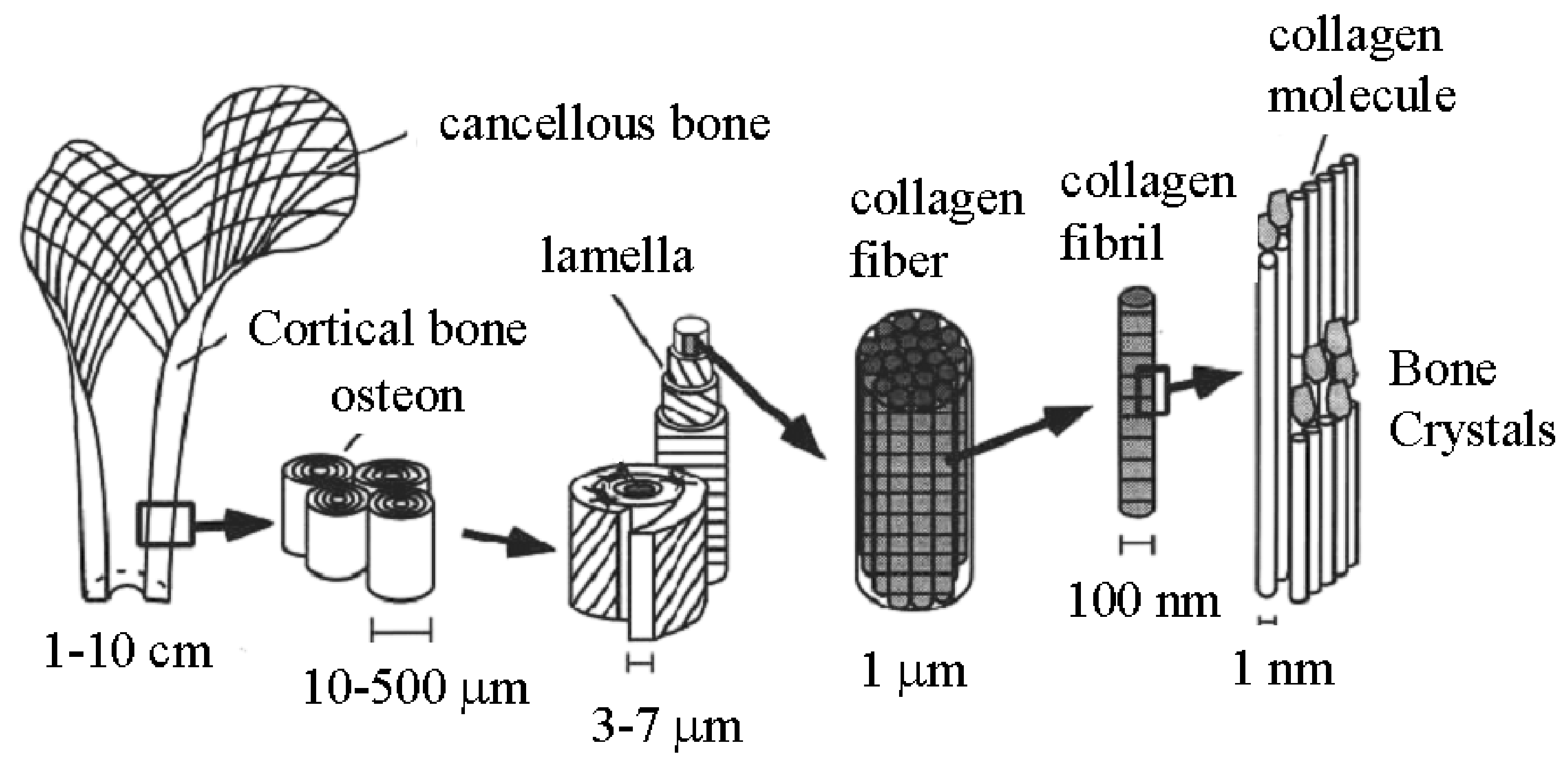
Residual compressive stress ~0.9 GPa after 1000 cycles


σ_a = 1.0 GPa
Monotonic Strength, σ_{crit} (GPa)
4.5
4
3.5
3
2.5
2
-5
-4
-3
-2
-1
0
1
2
3
Mean Stress, σ_m (GPa)

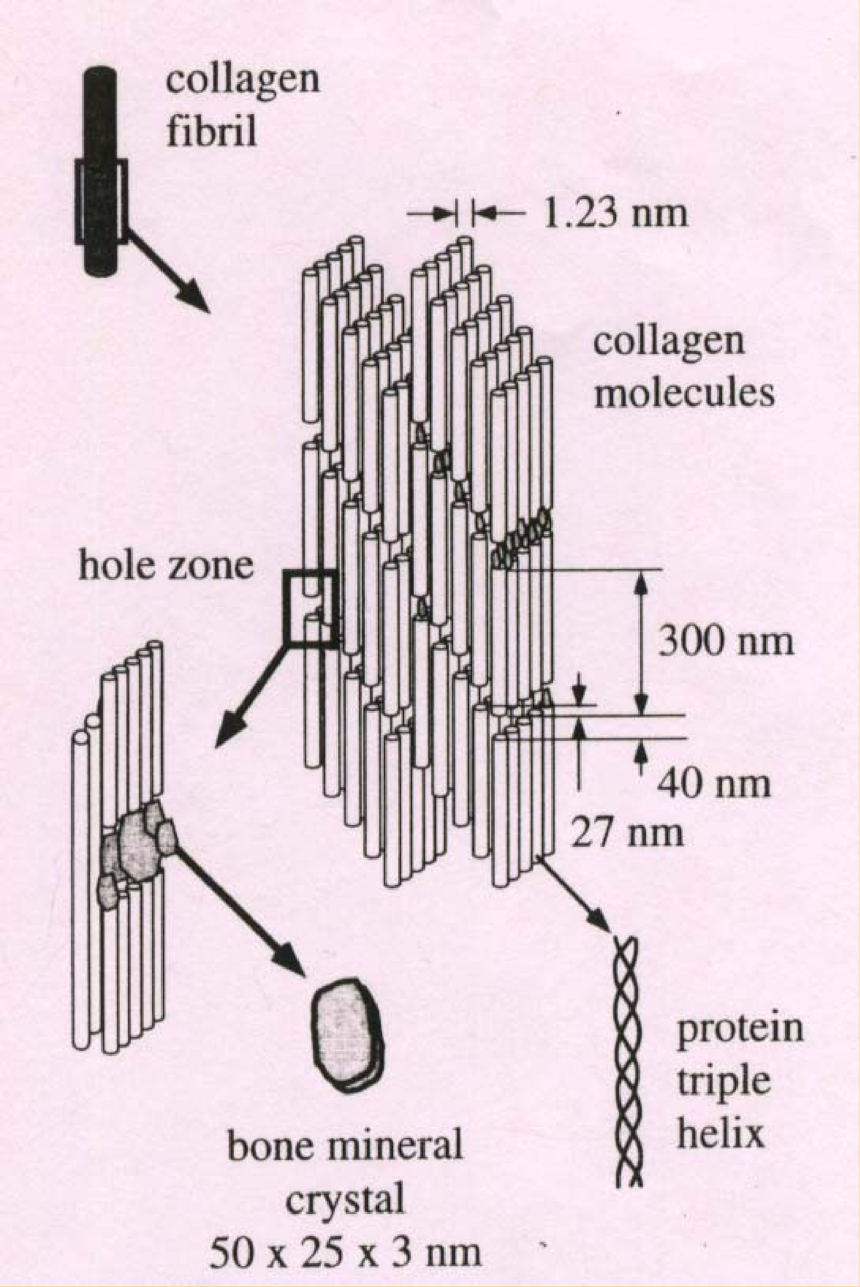
Mechanical Testing of Collagen Fibers (Nanotechnology)

- Most abundant protein in the human body.
- One of the basic components of bone, ligaments, tendons, teeth, skin.
- Collagen monomer:
  - Triple helical structure made of three chains of amino acids.
  - The monomers assemble into fibrils.

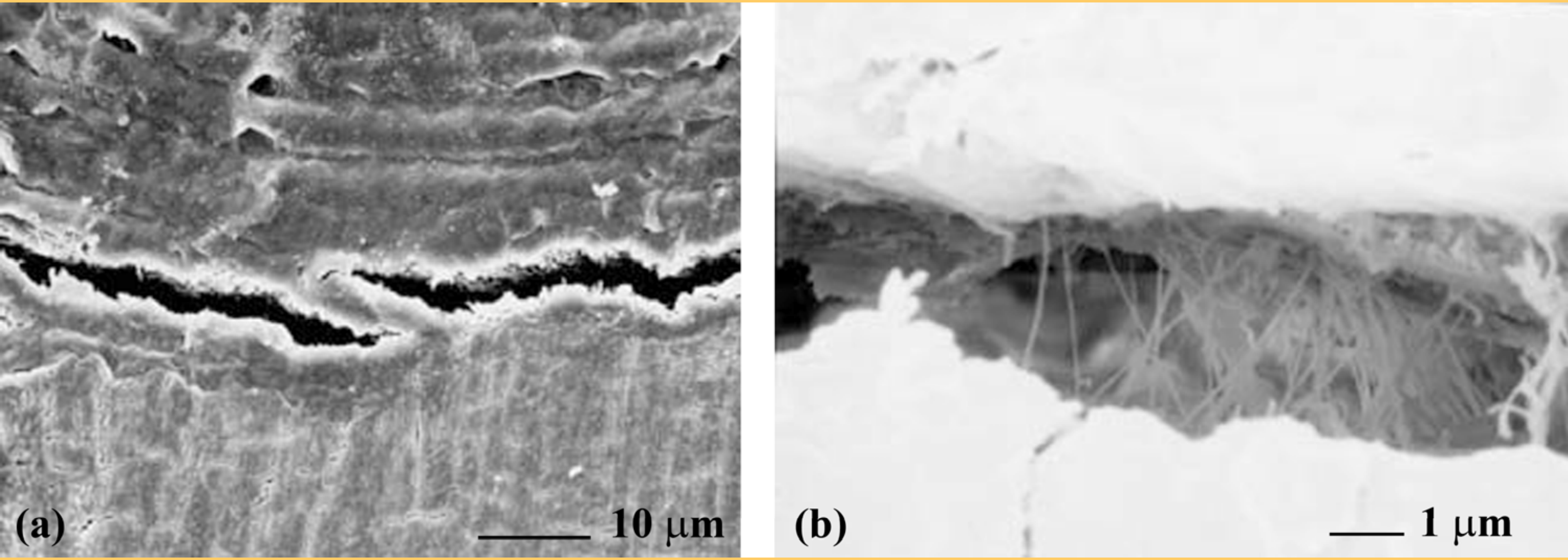
Hierarchical Structure of Bone



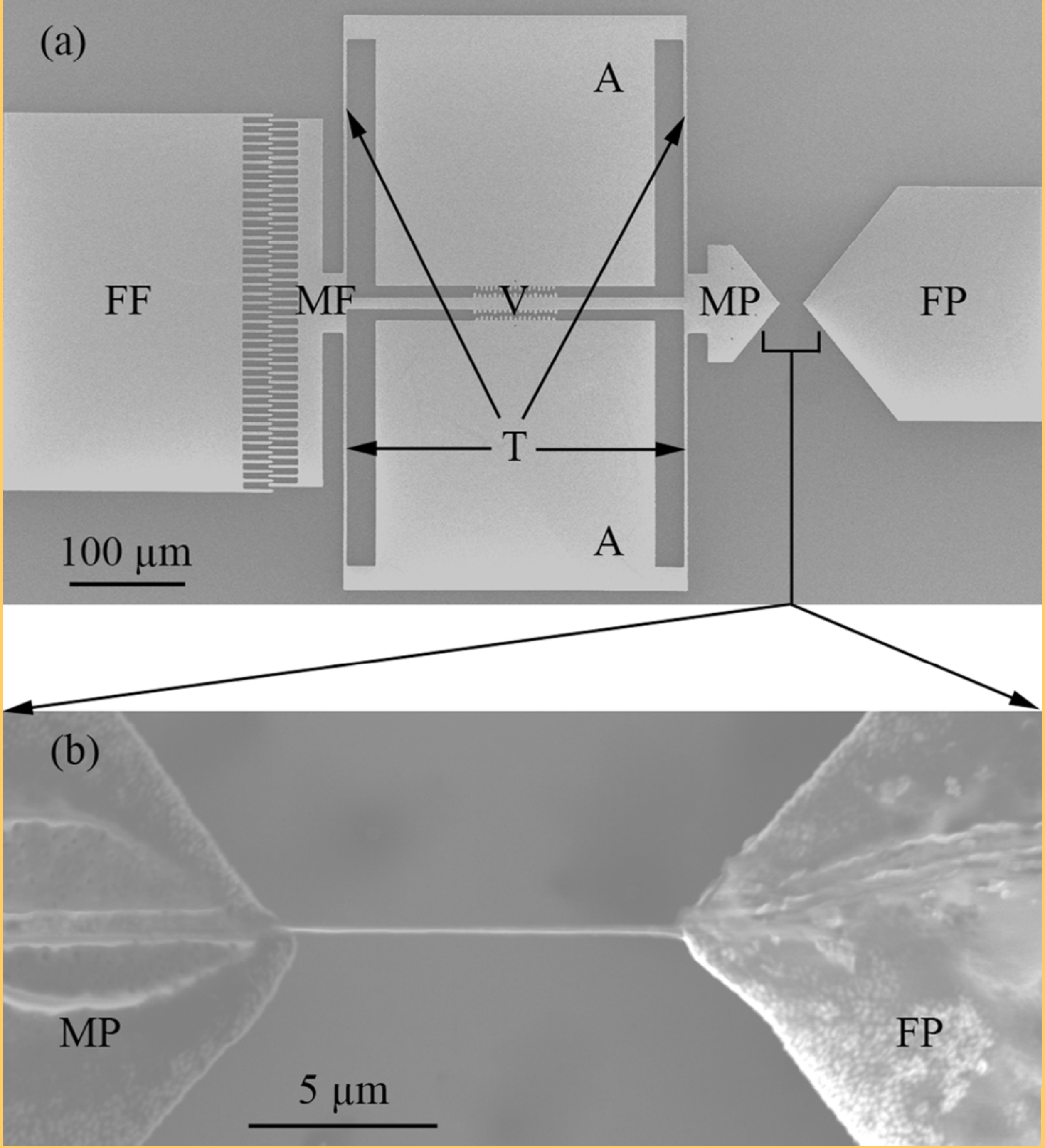
Collagen Fibrils



Rho *et al.*, 1998

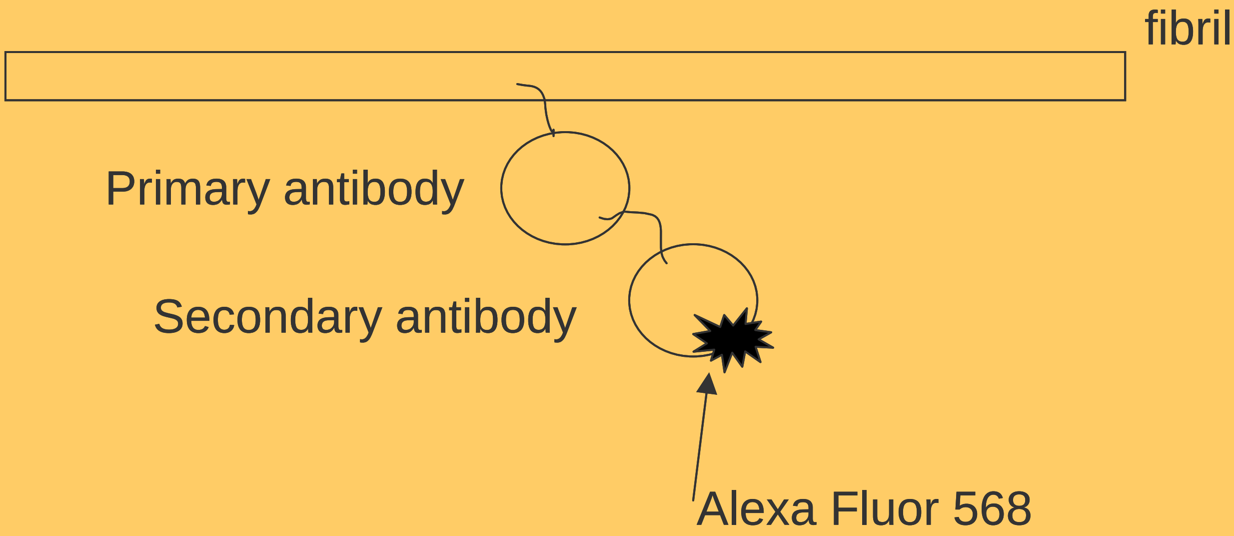


Crack Bridging Mechanisms
(Nalla *et al.* 2005)


(a)
A
FF
MF
V
MP
FP
T
A
100 µm
(b)
MP
FP
5 µm

Labeling fibrils using fluorescent antibodies

1. Imaging using SEM
2. Labeling

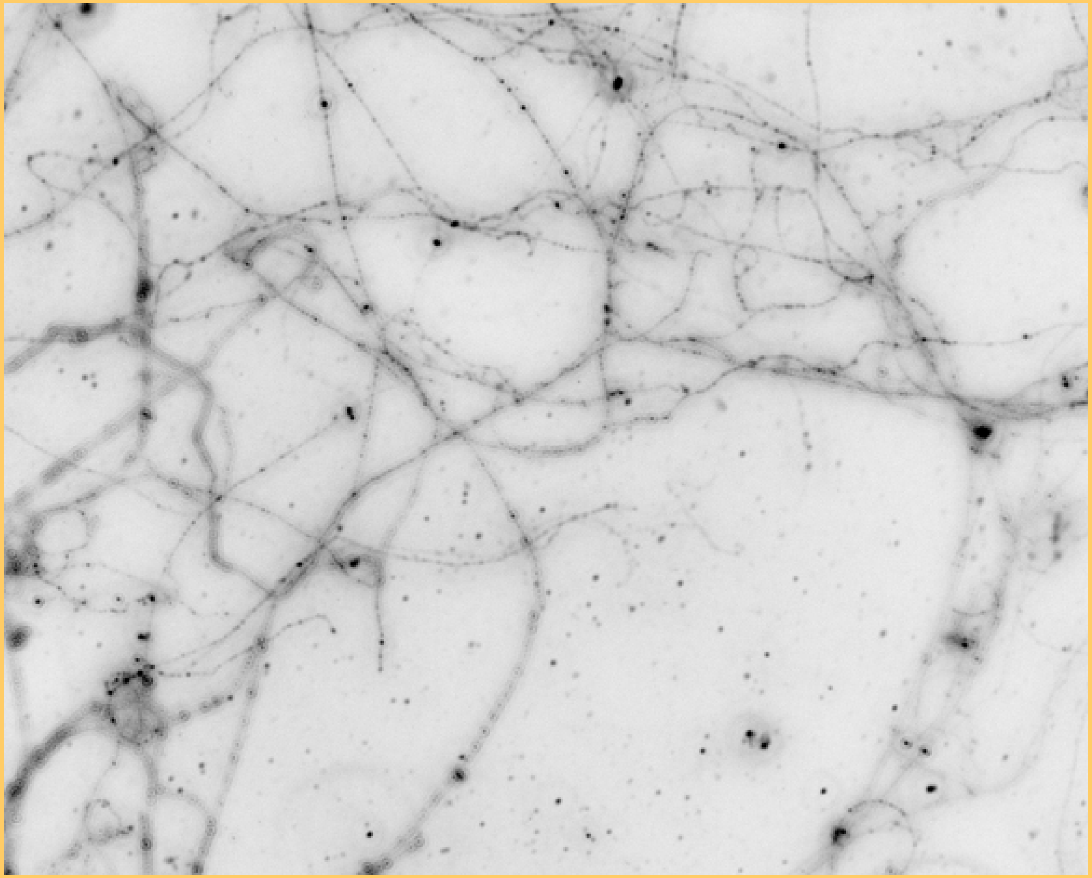


fibril

Primary antibody

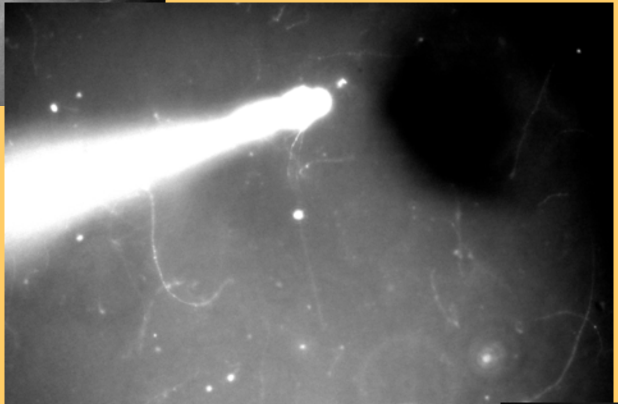
Secondary antibody

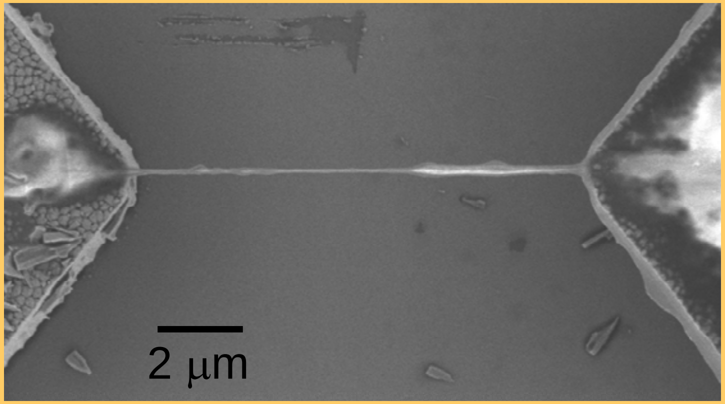
Alexa Fluor 568



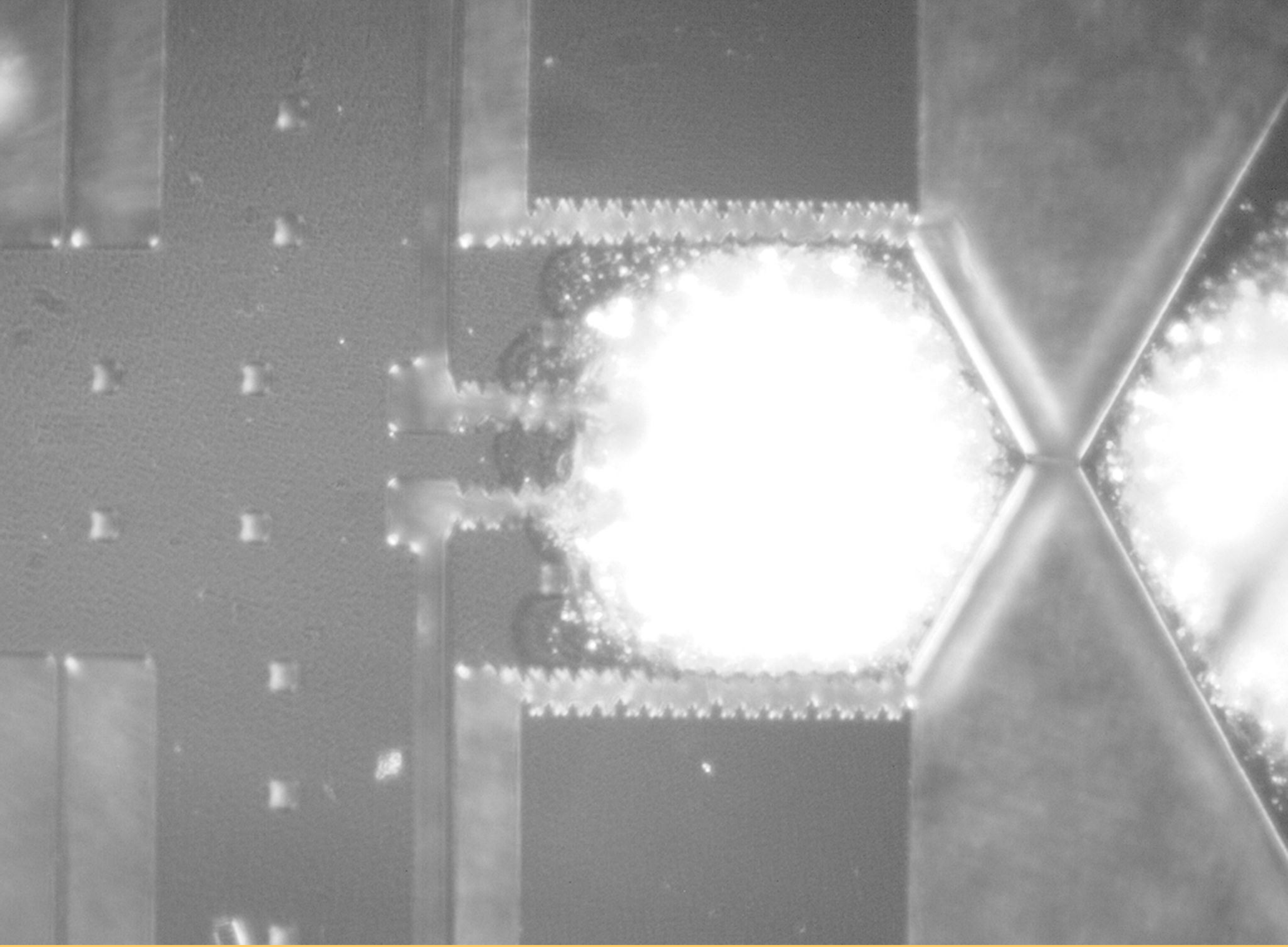
Fluorescently Labeled Collagen Fibers (Negative Image)

Different dilutions of the fibrils were imaged using SEM to determine the appropriate dilution at which individual fibrils were distinguishable. The fibrils were labeled with fluorescent antibodies to achieve contrast and brightness under optical microscope for 5 minutes. Anti-fading agents being tried to allow 30 minutes of manipulation time.





Manipulation using micropipette



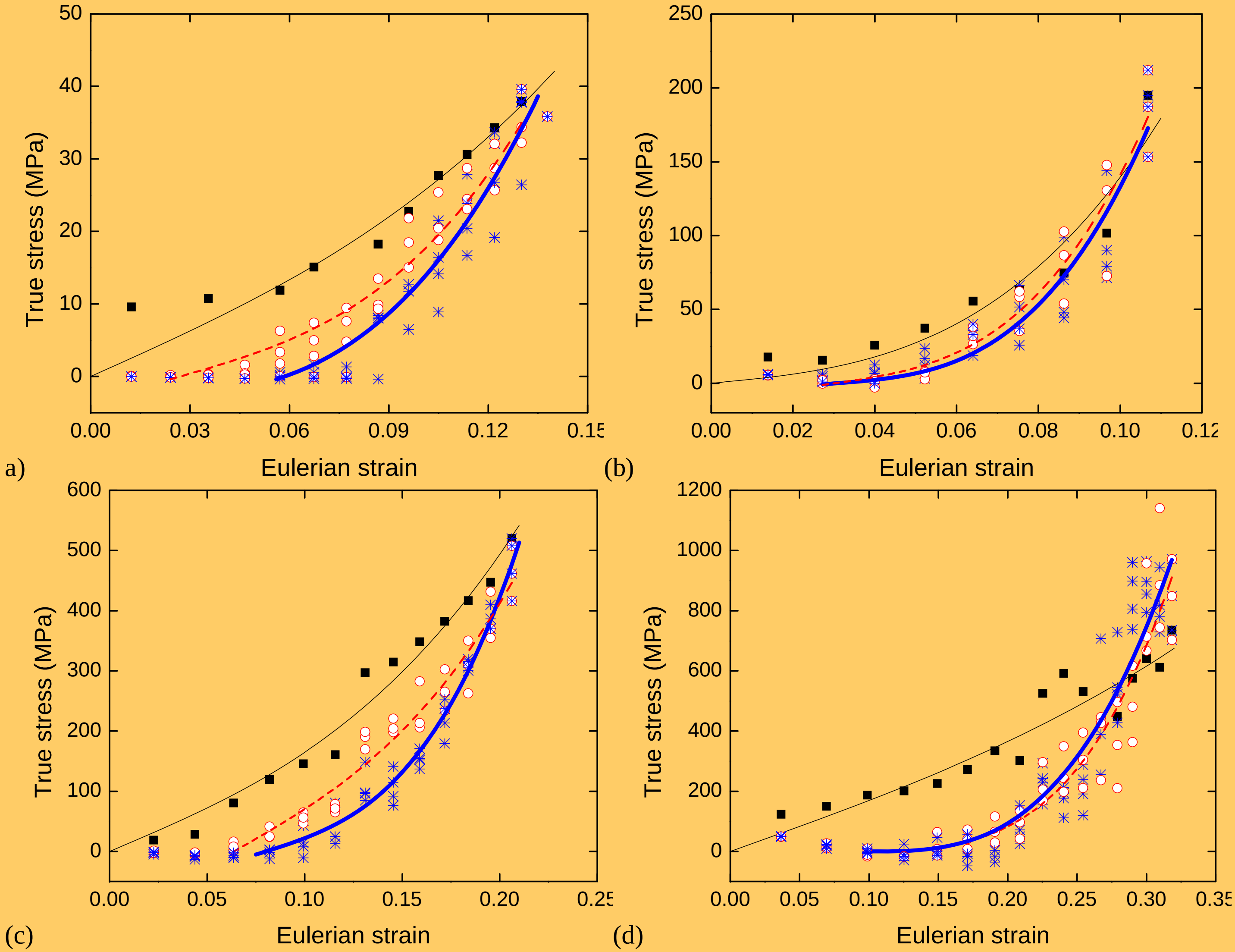
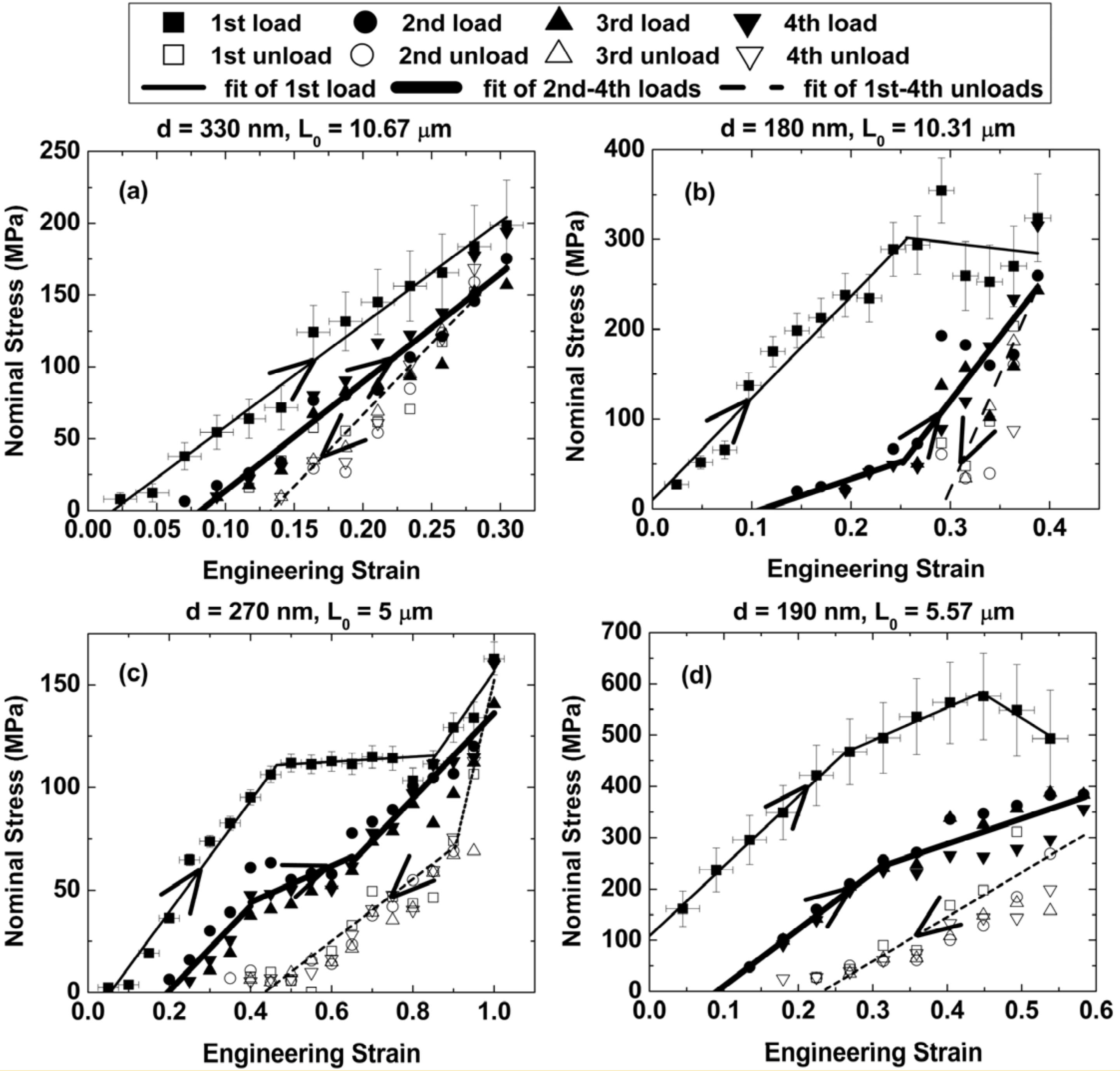


Fig. 2. True stress-Eulerian strain curves showing the data and fits for the first loads (solid squares and thin solid line), first-fourth unloads (stars and thick solid line), and second-fourth loads (open circles and dashed line) for (a) 950 nm diameter, (b) 340 nm diameter, and (c) 240 nm diameter, and (c) 120 nm diameter fibrils. For clarity, the error bars in strain are not included, but would equal about ± 0.005 in (a), ± 0.006 in (b), ± 0.008 in (c), and ± 0.008 in (d).


1st load
2nd load
3rd load
4th load
1st unload
2nd unload
3rd unload
4th unload
fit of 1st load
fit of 2nd-4th loads
fit of 1st-4th unloads
d = 330 nm, L$_0$ = 10.67 μm
(a)
Nominal Stress (MPa)
0
50
100
150
200
250
0.00
0.05
0.10
0.15
0.20
0.25
0.30
Engineering Strain
d = 180 nm, L$_0$ = 10.31 μm
(b)
Nominal Stress (MPa)
0
100
200
300
400
0.0
0.1
0.2
0.3
0.4
Engineering Strain
d = 270 nm, L$_0$ = 5 μm
(c)
Nominal Stress (MPa)
0
50
100
150
0.0
0.2
0.4
0.6
0.8
1.0
Engineering Strain
d = 190 nm, L$_0$ = 5.57 μm
(d)
Nominal Stress (MPa)
0
100
200
300
400
500
600
700
0.0
0.1
0.2
0.3
0.4
0.5
0.6
Engineering Strain