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

Roberto Ballarini
University of Minnesota

Collaborators:
Hal Kahn, Arthur Heuer, Steve Eppell

Sponsors:
NSF, NIH, DARPA, ARO, NASA

CCNY
3/19/09
REFERENCES

Experimental work:


Surface Micromachining

Si wafer

masking SiO\(_2\) (1\,\mu m)

sacrificial SiO\(_2\) (4\,\mu m)

undoped polysilicon (5.7\,\mu m)

Plasma etch

HF release

Pd sputter (17 nm)

B diffusion dope

HF release
Analog Devices Gyroscope

iMEMS Gyro Die Showing the Rate Sensor and Integrated Electronics

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 \((1000 \, \mu m)^2\) 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

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

High cycle fatigue

\[ \frac{da}{dN} = C(\Delta K_I)^m \]

Stress corrosion

\[ \frac{da}{dt} = DK^n_I \]

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

Native Oxide is known to obey this law
FOCUS OF THIS TALK

• Demonstrate that polycrystalline silicon is not susceptible to static fatigue.

  • Demonstrate that polycrystalline silicon is associated with mechanical fatigue and strengthening 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 $a_{cr} = 1 \mu m$

Say $t_{life} = 10$ yrs

Then $v_{cr} < 10^{-15}$ m/s !!!
CVD Polysilicon - Effects of Deposition Temperature

550°C 580°C 615°C

570/615°C 1100°C

all films are ~2-6 µm thick, and deposited on SiO2
MEMS Fracture Mechanics Specimen
*integrated with*
MEMS Loading Device Actuator


Fracture Mechanics Specimen

- movable comb drive
- fixed comb drive

Actuator
produces ~0.7 mN

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


Sharpe et al., Bagdahn and Sharpe, and Muhlstein et al. used the MUMPS foundry.
DIFFICULTIES IN DETERMINING ENVIRONMENTAL EFFECTS USING THESE TESTS

• Tests involve cyclic loading, not constant load.

• Tests involve tension and compression.
The specimens are given a tensile or compressive bias stress, $\sigma_0$, using a DC offset.

**Load Ratio**

$$R = \frac{\sigma_{\text{min}}}{\sigma_{\text{max}}}$$
Dynamic Fatigue Results

low-cycle fatigue

<table>
<thead>
<tr>
<th>PolySi thickness</th>
<th>Test Ambient</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.5 μm</td>
<td>air (10^5 Pa)</td>
</tr>
<tr>
<td>5.7 μm</td>
<td>air (10^5 Pa)</td>
</tr>
<tr>
<td>5.7 μm</td>
<td>vacuum (10 Pa)</td>
</tr>
</tbody>
</table>

Load Ratio, $R$

Low-Cycle Fatigue Strength, $\sigma_{max}$ (GPa)
Fractography of Biased Fatigue Specimens

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

Specimen C was subjected to a high compressive bias stress during resonance and fractured at a $\sigma_{\text{max}}$ 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 $\sigma_{\text{max}}$ and also consistent with $K_{\text{crit}} = 1.0 \pm 0.1 \text{ Mpa-m}^{1/2}$.

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)

\[ F(a) = \alpha \pi \sigma \]

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

\[ K = \sigma^* \sqrt{\pi a} F(\alpha) \]
INDENTATION CRACK

- substrate
- beam
- pre-crack
- crack tip
- beam anchor (to substrate)
- residual tensile stress

CWRU
FINE-GRAINED POLYSILICON FRACTURE TOUGHNESS

\[ \text{Crack Length [\(\mu\text{m}\)]} \]

\[ \text{Stress Intensity [MPa m}^{1/2}\text{]} \]

- = no propagation
- = propagation to failure

45 MPa

56 MPa

69 MPa

FINE-GRAINED POLYSILICON FRACTURE TOUGHNESS
POLYCRYSTALLINE SILICON CARBIDE
FRACTURE TOUGHNESS

![Graph showing stress intensity vs. crack length for different stress levels.]

- Crack Length [μm]
- Stress Intensity [MPa m$^{1/2}$]

- Black square = no propagation
- Grey circle = propagation to failure

Stress Levels:
- 214 MPa
- 189 MPa
- 175 MPa
- 159 MPa
- 158 MPa
- 150 MPa
- 149 MPa

= no propagation = propagation to failure
FINE-GRAINED SILICON
STATIC FATIGUE STUDY
90% RH

K between 0.62 - 0.86 MPa-m$^{1/2}$
No growth in 30 days
$V < 3.9 \times 10^{-14}$ 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)

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

Specimen T 100 μm

Specimen C 100 μm

R-ratio and mean stress effects
Schematic Bend Strength Tests

Monotonic

Increasing Amplitude Fatigue

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

$\sigma_{\text{max}} = \sigma_{\text{cr}}$

$1 \text{ Comp/Mono}$

$\sigma_{\text{cr}}$

$\sigma_{\text{min}}$

$\sigma_{m}$

$\sigma_{\text{hold}}$

$10 \text{ min.} = 5 \times 10^6 \text{cycles}$

$10 \text{ min.}$
Low-Cycle Fatigue

Mean Stress, $\sigma_m$ (GPa)

Fatigue Strength, $\sigma_{cr}$ (GPa)

- air ($10^5$ Pa)
- vacuum ($10$ Pa)
Monotonic Bend Strength after cycling with a fixed mean stress.

Mean Stress, $\sigma_m = -2.2$ GPa

Fatigue Amplitude, $\sigma_a$ (GPa)

Monotonic Strength, $\sigma_{crit}$ (GPa)
Monotonic Bend Strength after cycling with a fixed (low) amplitude

\[ \sigma_a = 1.0 \text{ GPa} \]

Mean Stress, \( \sigma_m \) (GPa)

Monotonic Strength, \( \sigma_{\text{crit}} \) (GPa)

\[ \sigma_a = \frac{\Delta \sigma}{2} \]

10 min. = 6x10^6 cycles
Monotonic Bend Strength
with/without initial compression

avg. $\sigma_{cr} = 3.0$ GPa
$m = 8.2$

$\sigma_{min} = -4.5$ GPa
Monotonic Bend Strength
with/without tensile hold

Bend Strength, $\sigma_{cr}$ (GPa)

Cumulative Probability

avg. $\sigma_{cr} = 3.1$ GPa

$\sigma_{hold} = 2.7$ GPa

$\sigma_{c}$

Time

Stress

$\sigma_{c}$

Time

$\sigma_{hold}$

10 min.

with tension

without tension
Effects on Monotonic Bend Strength of mean stress $\sigma_m$, and fatigue amplitude $\sigma_a$
Mechanisms?

- Phase transformation?
- Microcracking?
- Dislocations?
- Plasticity at grain boundaries?
Plastic flow in amorphous silicon
(M. J. Demkowicz and A. S. Argon)

\[ p = -\frac{1}{3} tr(\sigma), \quad \sigma_{\text{dev}} = \left| \sigma - \frac{1}{3} tr(\sigma) I \right| \]
Drucker Prager Model

\[ F = t - q \tan \beta - d = 0 \]
\[ G = t - q \tan \psi \]
\[ q = \sqrt{\frac{3}{2}} (S : S), \quad S = \sigma + pI \]
\[ d = \sqrt{3} \tau \]

\[ d \varepsilon^{*}_{ij} = d\lambda \frac{\partial G}{\partial \sigma \varepsilon_{ij}} \]
Poisson-Voronoi Local/Global Modeling

Model for Large Number of Cycles
Low $\Delta \sigma$ and high $\sigma_m$ ($\sigma_m = 2.0\text{GPa}$, $\Delta \sigma = 2.0\text{GPa}$)

Residual compressive stress $\sim 1.4\ \text{GPa}$ after 1000 cycles
Low $\Delta \sigma$ and high $\sigma_m$ ($\sigma_m = -3.5 \text{ GPa}, \Delta \sigma = 2.0 \text{ GPa}$)

Residual compressive stress $\sim 0.9 \text{ GPa}$ after 1000 cycles
$\sigma_a = 1.0 \text{ GPa}$
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

- Cancellous bone
- Lamella
- Cortical bone
- Osteon
- Collagen fiber
- Collagen fibril
- Bone Crystals

1-10 cm
10-500 μm
3-7 μm
1 μm
100 nm
1 nm
Collagen Fibrils

Rho et al., 1998
Crack Bridging Mechanisms
(Nalla et al. 2005)
Labeling fibrils using fluorescent antibodies

1. Imaging using SEM
2. Labeling

- Primary antibody
- Secondary antibody
- Alexa Fluor 568
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).
The graphs show the nominal stress (MPa) versus engineering strain for different load and unload cycles. The dimensions d and L₀ are provided for each set of data.

- **Figure (a)**: d = 330 nm, L₀ = 0.1067 μm
- **Figure (b)**: d = 180 nm, L₀ = 0.1031 μm
- **Figure (c)**: d = 270 nm, L₀ = 0.05 μm
- **Figure (d)**: d = 190 nm, L₀ = 0.0557 μm