Failure of Silicon: Crack Formation and Propagation

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MEMS, Microsystems and Micromachines

- Microturbine
- Micron-scale moveable mirrors
- Series of gears
- Microhinge

R. Conant, 1999

MCNC/Cronos
Outline

- Mechanical properties of silicon
- Brittle fracture of silicon
- Strength vs. fracture toughness
- Delayed failure of thin-film silicon
- Role of the native oxide layer
- Suppression/prediction of fracture
• crystal structure
  - diamond cubic structure (face-centered cubic)

• brittle-to-ductile transition (DBTT at ~500°C)
  - below the DBTT (or at high strain rates), Si is completely brittle
    - dislocations not mobile, Si fractures by cleavage on {111} planes
    - fracture strengths ~ 1 to 20 GPa in single-crystal silicon
    - fracture strengths ~ 3 to 5 GPa in polycrystalline silicon
  - above the DBTT, silicon becomes gradually ductile
    - glide motion of \((a/2)<110>\) dislocations on \(\{111\}\) planes
    - dissociation into \((a/6)<112>\) Shockley partials with 4-6 nm stacking faults
    - heterogeneous dislocation nucleation in “dislocation-free” crystals
e.g., at surfaces or due to deformation-induced amorphous Si
    - solid-solution hardening by impurity solutes, e.g., oxygen, nitrogen

a = 0.534 nm
Mobile Dislocations in Silicon at 25°C

- Indent went to a peak depth of 216 nm
- No phase transformations
- Large plastic extrusions of the diamond cubic phase
- Dislocation nucleation easier than phase transformation


Wall & Dahmen, 1997
Modes of Failure in Silicon

- Brittle (catastrophic) fracture
  - catastrophic transgranular cleavage fracture on \{111\} planes
  - evidence for \{110\} cleavage for “low energy/velocity” fractures

- Sustained-load cracking (delayed fracture)
  - no evidence for delayed fracture from subcritical crack growth, e.g.,
    due to stress-corrosion cracking, in bulk silicon below the DBTT
    (<500°C)
  - evidence for moisture-induced cracking in thin film silicon

- Cyclic fatigue failure (delayed fracture)
  - no evidence for delayed fracture from fatigue cracking under
    alternating loads in bulk silicon below the DBTT
  - strong evidence of premature fatigue failure of thin film silicon
What affects resistance to brittle fracture in silicon?

- **Intrinsic factors**
  - bond rupture
  - plasticity, i.e., mobile dislocations
  - defect (crack) population

- **Toughening mechanisms**
  - intrinsic mechanisms (ahead of crack tip)
    - microstructure, e.g., second phases
  - extrinsic (crack-tip shielding) mechanisms (behind crack tip)
    - crack bridging (intergranular cracking)
    - microcrack toughening (from dilation and reduced stiffness)
    - residual stresses (compressive for toughening)
Brittle Fracture of Silicon

{111} cleavage

transgranular cleavage fracture

{110} cleavage

inner surface of notch

mirror

fracture surface

0.5 μm

Muhlstein, Brown, Ritchie, Sensors & Actuators, 2001

Ballarini et al., ASTM STP 1413, 2001
Brittle Fracture of Silicon

- **elastic modulus**
  - $E \sim 160$ GPa

- **high fracture strengths**
  - 1 to 20 GPa in single-crystal silicon
  - 3 to 5 GPa in polycrystalline silicon
  - dependent on defect size, loading mode, specimen size, orientation, test method
  - probability of fracture dependent on “weakest-link” (Weibull) statistics

- **low fracture toughness**
  - $K_c \sim 1$ MPa√m in polysilicon thin films
  - $K_c \sim 0.7$-1.3 MPa√m in single-crystal films
  - dependent on specimen type, orientation and investigator
  - independent of microstructure

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Sharpe et al., ASTM STP 1413, 2001

Johnson et al., ASTM STP 1413, 2001
• Brittle fracture of silicon governed solely by the rupture of Si-Si bonds at the crack tip
  - $K_c$ is independent of microstructure

• Except variations due to orientation (in single-crystal Si) and experimental error, fracture strength depends on the defect population

• The probability of failure, $P_F$, can thus best be described in terms of "weakest-link" statistics

$$P_F(\sigma) = 1 - \exp \left[ -\int_0^V dV \left( \frac{\sigma - \sigma_u}{\sigma_o} \right)^m N \right]$$

  - where $\sigma_u$ is the lower bound fracture strength, $\sigma_o$ is the "scale parameter", $m$ is the Weibull modulus, and $V$ is the volume of the sample

LaVan et al., ASTM STP 1413, 2001
Strength vs. Fracture Toughness

- fracture strength/strain subject to extreme variability – not a material property
- more fundamental parameter is the fracture toughness - \( K_c \) or \( G_c \)
  - where \( K_c \) is the critical value of the stress intensity \( K \) to cause fracture
    \[
    K_c = Q \sigma_F (\pi a_c)^{\frac{1}{2}}
    \]
    \( \sigma_F \) is the fracture strength
    \( a_c \) is the critical crack size
    \( Q \) is a geometry factor (~unity)
  - and \( G_c \) is the strain energy release rate
    \[
    G_c = (K_c)^2/E
    \]
    \( E \) is Young’s modulus
- \( K_c = 1 \text{ MPa}\sqrt{\text{m}} \) in Si and is independent of microstructure and dopant
Measurement of Fracture Toughness

\[ K_c = Q \sigma_F (\pi a_c)^{1/2} \]

- Measurement of the fracture toughness of thin-film silicon using MEMS

Ballerini et al., ASTM STP 1413, 2001
Fracture Mechanics Approach

- low fracture toughness $K_c$ in silicon
  - 0.7 to 1.3 MPa√m in single-crystal Si
  - 1 MPa√m in polysilicon thin films
- compare with $K_c$ values of:
  - ~0.6 MPa√m in (soda-lime) glass
  - 2 to 3 MPa√m in human teeth (dentin)
  - 3 to 8 MPa√m in alumina ceramics
  - 20 to 200 MPa√m in steels
- from this microstructure-independent $K_c$ value in Si, can:
  - determine the fracture strength, $\sigma_F$, as a function of the largest defect size, $a_c$
    \[ K_c = Q \sigma_F (\pi a_c)^{1/2} \]

• **Probability of brittle fracture depends on defect (crack) population**
  - use fracture strength approach with weakest-link statistics to determine probability of fracture
  - characterize defect population at sub-micron resolution (actually tens of nanometers)
    - X-ray tomography (e.g., Xradia, Concord, CA)
    - GHz acoustic microscopy

\[
K_c \sim Q \sigma_F (\pi a_c)^{1/2} \sim 1 \text{ MPa}\sqrt{\text{m}}
\]
Modes of Failure in Silicon

- Brittle (catastrophic) fracture
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- Sustained-load cracking (delayed fracture)
  - no evidence for delayed fracture from subcritical crack growth, e.g., due to stress-corrosion cracking, in bulk silicon below the DBTT (<500°C)
  - evidence for moisture-induced cracking in thin film silicon

- Cyclic fatigue failure (delayed fracture)
  - no evidence for delayed fracture from fatigue cracking under alternating loads in bulk silicon below the DBTT
  - strong evidence of premature fatigue failure of thin film silicon
• micron-scale silicon films display some evidence of time-delayed failure under sustained (non-cyclic) loading

• lives for thin-film silicon are somewhat shorter in water

• no evidence of such time-delayed failure in bulk silicon
• Brittle (catastrophic) fracture
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• **composition**

  MUMPs process - LPCVD reactor*
  n-type – P doped
  deposited Si and PSG layers
  thermally annealed at ~900°C

• **microstructure**

  nominal grain size ~100 nm
  low residual stresses ~ -9 MPa

• **mechanical properties**

  $E \sim 163$ GPa, $\nu \sim 0.22$
  bending strength, $\sigma_F \sim 3 - 5$ GPa
  fracture toughness $K_c \sim 1$ MPa$\sqrt{m}$

  *MCNC/JDS Uniphase/Cronos/MEMSCAP

  **Contaminants**
  
  $1 \times 10^{19}$ atoms cm$^{-3}$ P
  $2 \times 10^{18}$ atoms/cm$^{-3}$ H
  $1 \times 10^{18}$ atoms/cm$^{-3}$ O
  $6 \times 10^{17}$ atoms/cm$^{-3}$ C
defects in the polysilicon films

- stacking faults
- Lomer-Cottrell locks
- microtwins

1 MeV HVTEM images

Electrostatically-Actuated Resonant Fatigue Testing

- Notched cantilever beam attached to ~300 μm square perforated plate (resonant mass)
- "Comb drives" on one side are electrostatically forced to resonate at ~ 40 kHz, with $R = -1$
- Other side provides for capacitive sensing of motion, calibrated with machine vision system (Freeman, MIT)
- Stress amplitudes determined by finite-element analysis (ANSYS)
- Smallest notch root radius (1 – 1.5 μm) achieved by photolithographic masking

Brown, Van Arsdell, Muhlstein et al.
Micron-scale \( p \)-type (110) single crystal Si films can fail after \( 10^9 \) cycles at (maximum principal) stresses (on 110 plane) of one half the (single cycle) fracture strength.

{110} crack paths suggest mechanisms other than {111} cleavage.

\[
\begin{align*}
\text{Stress Amplitude (GPa)} & \\
\text{Fatigue Life (Cycles)} &
\end{align*}
\]

Micron-scale polycrystalline $n$-type Si is susceptible to fatigue failure.

Films can fail after $10^9$ cycles at stresses of one half the (single cycle) fracture strength.

Fatigue Life, $N_f$ (Cycles)

- Slivers and debris on fractures consistent with some degree of microcracking.

Muhlstein, Brown, Ritchie, Sensors & Actuators, 2001
Fatigue of Single Crystal and Polycrystalline Silicon Thin Films

- Micron-scale silicon films display delayed failure under high-cycle fatigue loading
- No such delayed fatigue failure is seen in bulk silicon
Transgranular Cleavage Fracture

- transgranular cleavage cracking from notch under sustained loads
- some evidence of secondary cracking and multiple microcracking

Traditional Fatigue Mechanisms

Bulk ductile materials

Extrinsic Processes

- Metals
  - asperity, oxide wedge
  - striations
  - Crack Advance: $K_{\max}$ Controlled
  - Levels of Closure: $K_{\max}$ Controlled

Intrinsic Processes

- Crack Advance: $\Delta K$ Controlled

Ceramics

- Bridging grains
- crack advance by static modes
- Bridging Degradation: $\Delta K$ Controlled

Bulk brittle materials

- Crack Advance: $K_{\max}$ Controlled
- $\Delta K$ Controlled

Graphs:

1. 2024-T4 Smooth Bar Rotating Beam Fatigue
   Templin, et al. (1950)

2. $\text{Al}_2\text{O}_3$, Tension-tension fatigue
   Lathabai, et al. (1990)
Proposed Mechanisms of Silicon Fatigue

- Dislocation activity in thin films
- Stress-induced phase transformations (e.g., amorphous Si)
- Impurity effects (e.g., precipitates)
- Suppression of crack-tip shielding
- Surface effects (native oxide layer)
Notch Root Oxide Thickening

- native oxide thickness ~30 nm
- *in fatigue*, oxide thickness at notch root seen to thicken three-fold to ~100 nm
- *in sustained loading*, no such thickening is seen

\[ \sigma_a = 2.26 \text{ GPa}, \quad N_f = 3.56 \times 10^9 \text{ cycles} \]

Thermal vs. Mechanical Oxide Thickening

- temperature measured in situ at various stresses using a high-resolution IR camera
- IR camera capable of detecting $\Delta T$ to within mK with lateral positioning within microns
- small changes in $\Delta T$ of the resonant mass due to friction with the air
- notch region shows no change (<1 K) in $\Delta T$ during the fatigue test
- the observed 3-fold thickening of the oxide film in the notch region is promoted by mechanical rather than thermal factors

Crack Initiation in Notch Root Oxide

- Crack initiation in oxide scale during interrupted fatigue test
- Evidence of several cracks ~40 – 50 nm in length
- Length of cracks consistent with change in resonant frequency
- Strongly suggests subcritical cracking in the oxide layer, consistent with proposed model for fatigue

Interrupted after $3.56 \times 10^9$ cycles at $\sigma_a = 2.51$ GPa

Progressive time/cycle dependent fatigue mechanism could involve an alternating process of oxide formation and oxide cracking. However, the fracture toughnesses of Si and SiO₂ are comparable:

- Si: \( K_c \sim 1 \text{ MPa}\sqrt{m} \)
- SiO₂: \( K_c \sim 0.8 - 1 \text{ MPa}\sqrt{m} \)

In contrast, the susceptibility of Si and SiO₂ to environmentally-assisted cracking in the presence of moisture are quite different, with silica glass being much more prone to stress-corrosion cracking:

- Si: \( K_{isc} \sim 1 \text{ MPa}\sqrt{m} \) (in moisture)
- SiO₂: \( K_{isc} \sim 0.25 \text{ MPa}\sqrt{m} \)

Thus, fatigue mechanism is postulated as a sequential process of:

- mechanically-induced surface oxide thickening
- environmentally-assisted oxide cracking
- final brittle fracture of silicon
Silicon Fatigue Mechanism - Reaction-Layer Fatigue -

(a) Notch Root

(b) Reaction-Layer Thickening

(c) Reaction-Layer Crack Initiation

(d) Subcritical Crack Growth

(e) Unstable Crack Growth

• measured change in natural frequency used to compute specimen compliance and hence crack length throughout the test

• for $\sigma_a = 2 - 5$ GPa, crack lengths at onset of specimen failure remain less than $\sim 50$ nm

$K_c = Q \sigma_F (\pi a_c)^{1/2}$

- this suggests that the entire fatigue process, i.e.,
  - crack initiation
  - subcritical crack growth
  - onset of final failure

occurs within the native oxide layer

Why is Only Thin-Film Silicon Susceptible to Reaction-Layer Fatigue?

- mechanism is active for thin-film and bulk silicon in moist air
- due to low surface-to-volume ratio of bulk materials, the effect is insignificant
- critical crack size for failure can be reached in the oxide layer only for thin-film silicon, i.e., where \( a_c < h \)

Muhlstein, Ritchie, 2002
Interfacial Crack Solutions: Crack Inside Layer, Normal to Interface

• Beuth (1992)
  – extension of Civilek (1985) and Suo and Hutchinson (1989, 1990)
  – dislocation-based fracture mechanics solution
• Ye, Suo, and Evans (1992)

\[ \alpha = \frac{E_1 - E_2}{E_1 + E_2} \]

\[ \beta = \frac{\mu_1(1-2\nu_2) - \mu_2(1-2\nu_1)}{2\mu_1(1-\nu_2) + 2\mu_2(1-\nu_1)} \]

\[ \text{SiO}_2/\text{Si} \]
\[ \alpha = -0.5 \]
\[ \beta = -0.2 \]
Crack-Growth Rates and Final Failure

- estimated cracking rates display decreasing growth-rate behavior, consistent with:
  - small-crack effects
  - displacement-control conditions
  - residual stresses in film
  - growth toward SiO₂/Si interface

Muhlstein, Stach, Ritchie, Acta Mat., 2002
Solution for Crack in Native Oxide of Si

- Interfacial solutions for a compliant (cracked) SiO$_2$ layer on a stiff silicon substrate
- Crack-driving force $K_{I}$ is $f(a,h)$
- Maximum $K$ is found at $a_c/h \sim 0.8$

$K_{I,0}$ is the interfacial $K$ where $a/h = 0.05$; $h = 100$ nm

Muhlstein and Ritchie, *Int. J. Fract.*, 2003
Interfacial Crack-Driving Force

- Maximum $K$ at $(a/h) \sim 0.8$

- In range of fatigue failure, where $\sigma_{\text{app}} \sim 2$ to 5 GPa, cyclic-induced oxidation required for reaction-layer fatigue

- Oxide thickness $\geq 46$ nm for failure at $\sigma_{\text{app}} < 5$ GPa

- Oxide thickness $\geq 2.9$ nm for crack initiation at $\sigma_{\text{app}} < 5$ GPa

Muhlstein and Ritchie, *Int. J. Fract.*, 2003
Bounds for Reaction-Layer Fatigue

- behavior dependent on reaction-layer thickness
- bounds set by $K_{\text{iscc}}$ and $K_c$ of the oxide
- regimes consist of:
  - no crack initiation in oxide ($K < K_{\text{iscc}}$)
  - cracking in oxide but no failure ($K_{\text{iscc}} < K < K_c$)
  - reaction-layer fatigue ($K > K_c$)

Reaction-layer fatigue provides a mechanism for delayed failure in thin films of materials that are ostensibly immune to stress corrosion and fatigue in their bulk form
Alkene-Based Self-Assembled Monolayer Coatings

- fatigue testing in the absence of oxide formation achieved through the application of alkene-based monolayer coatings

- Si chip is dipped in HF and then coated with alkene-based monolayer coating – 1-octadecene
- alkene-based coating bonds directly to the H-terminated silicon surface
- coating is a few nm thick, hydrophobic, and stable up to 400°C; providing a surface barrier to moisture and oxygen

Muhlstein, Ashurst, Maboudian, Ritchie, 2001
Suppression of Reaction-Layer Fatigue

- SAM-coated Si samples display far reduced susceptibility to cyclic fatigue
- Absence of oxide formation acts to prevent premature fatigue in Si-films

- Alkene-based SAM coatings, however, do lower the fracture strength
- Oxidation during release smooths out surface; with coatings, sharp surface features remain

• Below a ductile-brittle transition temperature of ~500°C, Si displays a high fracture strength (1 - 20 GPa in mono- and 3 - 5 GPa in poly-crystalline Si)

• However, Si is intrinsically brittle with a fracture toughness of ~1 MPa $\sqrt{m}$ (approximately twice that of window pane glass!). This value is independent of microstructure and dopant type

• Evaluation of probability of fracture can be made using weakest-link statistics and/or nanoscale crack detection

• Thin film (micron-scale) Si is susceptible to delayed fracture under sustained and particularly high-cycle fatigue loading - prematurely failure can occur in room air at ~50% of the fracture strength

• Mechanism of cyclic fatigue is associated with mechanically-induced thickening and moisture-induced cracking of the native oxide (SiO$_2$) layer

• Mechanism significant in thin-film (and not bulk) Si as the critical crack sizes for device failure are less than native oxide thickness, i.e., $a_c < h_{\text{oxide}}$

• Suppression of oxide formation at the notch root, using alkene-based SAM coatings, markedly reduces the susceptibility of thin-film silicon to fatigue.
**Brittle Fracture**
- Si-Si bond rupture
- defect (crack) population
- residual stresses

*Probability of fracture depends on defect (crack) population*
- smooth surfaces, round-off edges, etch out cracks
- use weakest-link statistics
- detect microcracks on the scale of tens of nanometers

**Delayed Fracture**
- cracking in native oxide layer (thin film silicon)

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**Bottom line:**

What affects fracture in silicon?

- **Fracture strength**, $\sigma_F$ (GPa)
- **Critical crack size**, $a_c$ (nm)
- **Probability of fracture**, $P_f$

\[ K_c \sim Q \sigma_F (\pi a_c)^{1/2} \approx 1 \text{ MPa} \sqrt{\text{m}} \]

- safe fractures
- weakest-link statistics