Influence of Grain Structure and Doping on the Deformation and Fracture of Polycrystalline Silicon for MEMS/NEMS

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Abstract: The deformation and fracture of polycrystalline silicon (poly-Si) films have important implications for MEMS/NEMS technology. This work focuses on the investigation of the influence of grain structure and doping on the deformation and fracture behavior of poly-Si films grown by low-pressure chemical vapor deposition (LPCVD). The films were deposited on silicon wafers with a variety of grain sizes and doping levels. The deformation behavior was characterized using nano-indentation and tensile testing, while the fracture behavior was studied using SEM and fractography. The results showed that both grain size and doping level had a significant impact on the deformation and fracture properties of the poly-Si films.

Subject Terms: Polycrystalline Silicon, MEMS, Nanomechanics, Fracture, Doping, Low-pressure Chemical Vapor Deposition (LPCVD)
Goals, Accomplishments, On-going Research

Research Objectives

- Investigate the dependence of mode I fracture toughness of polysilicon thin films on grain size and doping.
- Quantify mechanical strength size effects for laminated and columnar grain polysilicon subjected to different doping conditions.

Accomplishments

- Measured the effective mode I critical stress intensity factor for laminated and columnar grain polysilicon films as a function of dopant concentration.
- Quantified the effect of specimen size on fracture strength of polysilicon films via a Weibull analysis to identify the location of failure initiation.
- Employed fusion bonded (Si-Si) chevron notch specimens to study the effect of doping and crystallographic orientation on $K_{IC}$ of Si grain boundaries.
- Characterized the electromechanical behavior of PZT thin films for MEMS.

On-going Research

- Measure the $K_{IC}$ of polysilicon grain boundaries as a function of dopant concentration with fracture experiments on Si-Si wafer bonded specimens with Chevron notches.
- Removal of sidewall defects in columnar grain polysilicon to determine the improvement in mechanical strength.
Polysilicon films were fabricated using a modified Sandia’s SUMMiT V™ microfabrication process.

1 µm thick polysilicon films were micromachined into dog-bone shaped specimens with gage length and width of 1,000 µm and 100 µm respectively.

Both polysilicon types of film were doped with various concentrations of P diffused from a sacrificial phosphosilicate glass (PSG) layer during annealing.
Fabrication of Columnar and Laminated Polysilicon

Columnar Polysilicon

Laminated Polysilicon

1.0 µm

0.5 µm
Polysilicon Films with Different Grain Size and Doping

- Polysilicon film specimens (1 μm thick) were fabricated in a special run at the Sandia National Laboratories.
- Two types of polysilicon films: Large grain and small grain (laminated).
- The laminar structure controlled the grain size.

Mathematically sharp cracks were generated by indentation performed on the substrate near a specimen free edge.

Edge cracks with an average length of 25 μm were introduced to 1 μm thin polysilicon specimens.

Cracks were imaged by SEM and AFM to obtain their geometry and the grain structure at the crack tip.

Crack Tip in Polysilicon Films with Different Grain Structures

- Columnar polySi, 2% PSG

- Laminated polySi, 2% PSG

- The precise location of the crack tip, its geometry and adjacent material were determined using AFM images obtained at different magnifications.

- Experimental setup used to test pre-cracked polysilicon thin films under mode I loading.

**Effect of Grain Structure and Doping on \( K_{IC,\text{eff}} \)**

- \( K_{IC,\text{eff}} \) of columnar polysilicon was higher than that of laminated polysilicon.
- Minimum \( K_{IC} \) for single crystal silicon is 0.84 MPa√m
- Phosphorous atoms diffuse into substitutional sites in Si forming \( \text{P-Si} \) bonds that have higher bond energy: bond energies of Si-Si and P-Si are 326.86 kJmol\(^{-1}\) and 363.6 kJmol\(^{-1}\) respectively.

<table>
<thead>
<tr>
<th>PSG content</th>
<th>Laminated ( K_{IC,\text{eff}} ) (MPa√m)</th>
<th>Columnar ( K_{IC,\text{eff}} ) (MPa√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Undoped</td>
<td>0.99±0.05</td>
<td>0.95±0.08</td>
</tr>
<tr>
<td>0.5% PSG</td>
<td>0.93±0.10</td>
<td>1.02±0.13</td>
</tr>
<tr>
<td>2% PSG</td>
<td>0.97±0.08</td>
<td>1.05±0.14</td>
</tr>
</tbody>
</table>
Sub-Critical Crack Growth in Columnar Polysilicon

- Local crack tip deflection was observed in heavily doped (2%) large grain polysilicon.
- Initial crack propagation was along the most energetically favorable plane, with the crack subsequently arrested at a grain boundary.

![Image showing crack propagation and arrest in columnar polysilicon](image.png)
The fracture strength of laminated polysilicon was 80-150% higher than columnar.

Heavily doped columnar polysilicon film exhibited ~25% lower fracture strength compared to undoped.

This implies that the increase in the upper bound of $K_{IC}$ is due to crack kinking.

### Average strength of polysilicon

<table>
<thead>
<tr>
<th></th>
<th>Undoped (GPa)</th>
<th>2% PSG (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laminated</td>
<td>2.32±0.15</td>
<td>2.46±0.22</td>
</tr>
<tr>
<td>(≈125nm)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Columnar</td>
<td>1.30±0.09</td>
<td>0.95±0.07</td>
</tr>
<tr>
<td>(≈285nm)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Graph:

- **Failure Stress (GPa)**: The graph shows the failure stress for laminated and columnar polysilicon with 0% and 2% PSG content.
- **PSG content**: The x-axis represents the PSG content percentage (0% and 2%).
- **Average Strength of Polysilicon**: The bar chart compares the average strength of undoped and 2% PSG polysilicon for laminated and columnar structures.
Critical Defects on Sidewalls of Columnar Polysilicon

- 100-300 nm deep crevices were found on the side walls of heavily doped columnar polysilicon films.
- Doping had no impact on the surface of laminated polysilicon films.

Columnar polysilicon (undoped)

Grain boundary groove

0.5 µm

Columnar polysilicon (2% PSG)

Grain boundary grooves

1 µm

Crevice

Laminated polysilicon

1 µm
Scaling of Mechanical Strength of Polysilicon

\[ P = 1 - \exp\left(-\left(\frac{\sigma - \sigma_0}{\sigma_c}\right)^m\right) \]

- \( \sigma \) – measured strength
- \( \sigma_c \) – characteristic strength
- \( \sigma_o \) – smallest failure stress
- \( m \) – Weibull modulus

<table>
<thead>
<tr>
<th></th>
<th>0% Columnar</th>
<th>2% Columnar</th>
<th>0% Laminated</th>
<th>2% Laminated</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \sigma_c ) (GPa)</td>
<td>1.34</td>
<td>1.01</td>
<td>2.32</td>
<td>2.51</td>
</tr>
<tr>
<td>( m )</td>
<td>17.63</td>
<td>16.89</td>
<td>10.314</td>
<td>9.09</td>
</tr>
<tr>
<td>( \sigma_c ) (GPa)</td>
<td>1.76</td>
<td>1.48</td>
<td>2.80</td>
<td>2.83</td>
</tr>
<tr>
<td>( m )</td>
<td>12.9</td>
<td>8.7</td>
<td>8.6</td>
<td>13.5</td>
</tr>
</tbody>
</table>
Defects scale with side wall area

\[ \sigma_{c2} = \sigma_{c1} \times \left( \frac{A_1}{A_2} \right)^{\frac{1}{m}} \]

\( A_n \) is side wall area

Defects scale with top surface area

\[ \sigma_{c2} = \sigma_{c1} \times \left( \frac{A_1}{A_2} \right)^{\frac{1}{m}} \]

\( A_n \) is top surface area
Comparison with Existing Polysilicon Processes

The characteristic strength of laminated poly1 improved and matched that of poly3, whereas the strength of columnar poly1 is comparable to that of regular poly1.

Sidewalls of columnar polysilicon were machined using FIB to fabricate defect free specimens.

Currently resolving challenges in gripping the specimens to test them in uniaxial tension using the narrow section.
Traction separation law for the polysilicon grain boundaries obtained from J integral and CTOD measured using DIC.

\[ \sigma(\delta_n) = \frac{\partial J}{\partial \delta_n} \]

Ongoing Work: Cohesive Law for Silicon Grain Boundaries
Effect of Doping on $K_{IC}$ of Si Toughness

Indentation using Vickers tip

For median cracks with $c/a > 2$,

$$K_{IC} = 0.129 \left(\frac{c}{a}\right)^{-3/2} \left(\phi E_{[hkl]} / H\right)^{2/5} \left(Ha^{1/2} / \phi \right)$$

where $E_{[hkl]}$ is the Young’s modulus along [hkl], $H$ is the hardness of Si, $\phi$ is a plastic constraint factor (~3).

$$\frac{1}{E_{[hkl]}} = S_{11} - 2(S_{11} - s_{12} - 1/2S_{44})\gamma_{z\beta\theta}$$

$$\gamma_{z\beta\theta} = \cos^2 \alpha \cos^2 \beta + \cos^2 \theta + \cos^2 \theta \cos^2 \alpha$$

$$\cos \alpha = h / (h^2 + k^2 + l^2)^{1/2}, \cos \beta$$

$$= k / (h^2 + k^2 + l^2)^{1/2}, \cos \theta = l / (h^2 + k^2 + l^2)^{1/2}$$


Indentation will be performed on silicon wafers doped with different concentrations of P.

Effect of doping on $K_{IC}$ of different Si planes is being studied by driving median cracks along [001], [110] and [111] directions.
Fracture toughness of Si – Si interfaces is measured using chevron notch specimens.

\[ K_C = \frac{F_{\text{max}}}{B\sqrt{w}} Y_{\text{min}} \]

Fabrication of Chevron Notch Specimens

1. Silicon wafer
2. Spin coat photoresist
3. Photolithography

Pattern Si wafer using Bosch’s process

Patterned silicon wafer

Direct bonding of Si wafers

Fusion bonded silicon wafers with different concentrations of P are used to study the effect of P doping on fracture toughness of Si-Si interfaces.
**Characterization of Si-Si Bond Interface**

- IR images are used to characterize the quality of the interface.
- Silicon cleaves easily on (111) planes and along [110] direction.
Strength of Si-Si bond interface is dependent on annealing temperature.

Si-Si interface with different concentrations of P, and crystal orientations are being fabricated and tested to understand their effect on $K_{IC}$ of silicon.
PZT thin film bimorph for MAV* wing

- PZT thin films are used in MEMS devices, such as micro-sensors, actuators, and RF-MEMS
- Always fabricated in combination with other films as stacks, they undergo non-standard thermal processing
- Their mechanical and failure properties in PZT stacks are either unknown or widely scattered
- Knowledge of ferroelectric behavior is key in designing reliable devices.
Microscale Measurements

- Specimens were loaded in uniaxial tension
- Strain was calculated by DIC from the speckle pattern on the samples
- Displacement resolution ~25 nm.

• Stress-strain curves were nonlinearly elastic with failure 0.6% strain
• Nonlinearity was attributed to domain switching mechanism in PZT at higher stresses

The modulus of PZT was extracted using simple laminate theory as $84 \pm 3$ GPa and $60.5 \pm 5$ GPa for open and short circuit conditions, respectively.

Electric field induced stress hysteresis loops were measured with applied pre-stress in the range of 0-600 MPa.

At high applied electric fields (> 2MV m\(^{-1}\)), the ferroelectric behavior of PZT is due to electrostriction and domain switching.

The electroactive coefficient, \(e_{31,\text{eff}}\) was used to quantify the ferroelectric behavior of PZT as the ratio of stress to applied electric field:

\[
P = e_{31,\text{eff}} \cdot \frac{V}{t_{\text{PZT}}} \cdot A_{\text{PZT}}
\]
Effect of Applied Pre-Stress on Hysteresis Loops

- Stress induced hysteresis loops became symmetric at high applied stresses.
- The intersection of the hysteresis loops shifted to positive electric fields.

• With no pre-stress, $e_{31,\text{eff}}$ varied nonlinearly with applied electric field between -50 NV$^{-1}$m$^{-1}$ and -30 NV$^{-1}$m$^{-1}$ and was independent of the applied field at high pre-stress amplitudes.

• Pre-stress lowers domain switching activity in PZT and produces more linear actuation force with the applied electric field.

Conclusions

- Evaluated the effective mode I critical stress intensity factor for laminated and columnar grain polysilicon films as a function of dopant concentration to assess the role of grain heterogeneity and local toughness on crack initiation and arrest.
- Quantified the effect of specimen size on fracture strength of polysilicon films via a Weibull analysis to identify the location of failure initiation.
- Failure originates in patterning processes rather than deposition: Investigating the effect of removal of sidewall flaws on strength improvement.
- Investigating the role of doping using bi-crystal experiments using chevron specimens.
- Obtained the electroactive coefficients PZT thin films for MEMS and mechanical properties under open and short circuit conditions: They were the first data of their kind and have drawn interest by industry too.