Structural Design Parameters for Germanium

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NASA GRC

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Germanium

- Good electromagnetic transmission in 2-15 μm range. Used for specialty windows; solar cells; substrates.

- Space Act Agreement with an industrial partner to determine the transient reliability of a proprietary, thermally and mechanically loaded, Ge window, along with the input design properties.
Germanium

- Brittle transition metal.
- Relatively soft.
- Behaves like a soft, brittle ceramic.
- Stress corrosion cracking?
- What is the fracture toughness?

Objective

- Measure mechanical properties
- Perform transient reliability analysis.
Material

- Single crystal beams
- Polycrystalline disks (2” & 5” Φ):

- Coarse, variable grain structure – not ideal for testing.
Anisotropy

- Anisotropy factor $A^*$ measures relative magnitude of elastic anisotropy exhibited by a crystal. $A^* = 0$ for isotropic materials, $A^* = 0$ to 1 for many single crystals.

- Running mechanical test on off-axis planes can be problematic if the anisotropy is large.
- Relatively low $A^*$ - proceed

![Graph showing anisotropy factors and approximate stress/series stress for different materials.]

**Cubic materials, \{100\}**
- NiAl
- B-SiC
- GaP
- Si
- Ge
- MgO
- Diamond

**Tetragonal and Trigonal Materials, \{010\}**
- In
- BaTiO$_3$
- Sn
- TiO$_2$
- Sapphire

**Approximate Stress/Series Stress (Plate Center)**

<table>
<thead>
<tr>
<th>Approximate Stress/Series Stress (Plate Center)</th>
<th>Anisotropy Factor $A^*$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.00</td>
<td>0</td>
</tr>
<tr>
<td>1.02</td>
<td>2</td>
</tr>
<tr>
<td>1.04</td>
<td>4</td>
</tr>
<tr>
<td>1.06</td>
<td>6</td>
</tr>
<tr>
<td>1.08</td>
<td>8</td>
</tr>
</tbody>
</table>

**Total Anisotropy Factor ($A_c^* + A_s^*$)**

<table>
<thead>
<tr>
<th>Total Anisotropy Factor ($A_c^* + A_s^*$)</th>
<th>Approximate Stress/Series Stress (Plate Center)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.00</td>
<td>0.98</td>
</tr>
<tr>
<td>1.02</td>
<td>1.00</td>
</tr>
<tr>
<td>1.04</td>
<td>1.02</td>
</tr>
<tr>
<td>1.06</td>
<td>1.04</td>
</tr>
<tr>
<td>1.08</td>
<td>1.06</td>
</tr>
</tbody>
</table>
Young’s Modulus
- impulse excitation -

• $E_{<111>}$ = 154.8 ± 0.9 GPa
• $E_{<110>}$ = 138.3 ± 0.2
• $E_{<100>}$ = 103.1 ± 0.6

• $E_{\text{poly}}$ = 131, $\nu_{\text{poly}}$ = 0.21

<table>
<thead>
<tr>
<th>Aggregate Constants</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formula</td>
</tr>
<tr>
<td>Voigt</td>
</tr>
<tr>
<td>Hashin</td>
</tr>
<tr>
<td>Shtrikman</td>
</tr>
<tr>
<td>Reuss</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>$Ge$</th>
<th>McSkimin</th>
<th>Bogardus</th>
<th>McSkimin</th>
<th>Mason</th>
<th>Average</th>
<th>NASA</th>
<th>% Diff.</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_{&lt;100&gt;}$ =</td>
<td>104.4</td>
<td>102.0</td>
<td>102.2</td>
<td>103.7</td>
<td>103.1</td>
<td>103.1</td>
<td>0.0%</td>
</tr>
<tr>
<td>$E_{&lt;110&gt;}$ =</td>
<td>138.7</td>
<td>136.7</td>
<td>137.0</td>
<td>138.0</td>
<td>137.6</td>
<td>138.3</td>
<td>0.5%</td>
</tr>
<tr>
<td>$E_{&lt;111&gt;}$ =</td>
<td>155.8</td>
<td>154.2</td>
<td>154.5</td>
<td>155.1</td>
<td>154.9</td>
<td>154.8</td>
<td>-0.1%</td>
</tr>
</tbody>
</table>

• Well oriented germanium….
Procedure
- Fracture Toughness -

- Three standard test methods (C1421):

- Different crack size and crack formation history.
- Different effort.
- Some methods don’t work well on some materials.
Fracture Toughness

<table>
<thead>
<tr>
<th>Method</th>
<th>{100}</th>
<th>{110}</th>
<th>{111}</th>
</tr>
</thead>
<tbody>
<tr>
<td>SEPB</td>
<td>0.67 ± 0.04</td>
<td>0.68 ± 0.01</td>
<td>0.72 ± 0.02</td>
</tr>
<tr>
<td>CNB</td>
<td>0.67 ± 0.03</td>
<td>0.69 ± 0.02</td>
<td>0.75 ± 0.03</td>
</tr>
<tr>
<td>SCF</td>
<td>0.74 ± 0.02</td>
<td>0.74 ± 0.02</td>
<td>0.74 ± 0.02</td>
</tr>
</tbody>
</table>

- Essentially similar on all planes.
- $K_{I\text{scf}\{jkl\}} = 0.74 \pm 0.02 \text{ MPa} \sqrt{\text{m}}$.
- $K_{I\text{pb}\{100, 110\}} = 0.68 \pm 0.04 \text{ MPa} \sqrt{\text{m}}$.
- ~10% difference between SCF and SEPB. Plasticity?
- Practical value of $K_{I\{jkl\}} = 0.68 \pm 0.02 \text{ MPa} \sqrt{\text{m}}$. 


SCF Fracture Surfaces

- {100} is conchoidal and exhibits cathedral Wallner lines.
- The most planar surface occurs on the {110}.
- {111} is planar but tends to exhibit cleavage steps.
- Secondary orientation was not fixed.
Cathedral Orientation

- Peak of cathedral corresponds to the $<100> \{100\}$. 
CNB Fracture Surfaces

- Ambient lighting:

\{100\} Smooth, Rounded - Conchoidal
\{110\} Smooth, Flat - Cleavage
\{111\} Stepped, Flat - Cleavage
CNB Fracture Surfaces

- **Oblique lighting:**
  - {100} Smooth, dimples, Rounded
  - {110} Fine Wallner lines Flat
  - {111} Stepped Flat

Pores or inclusions?
$K_{I_{\{111\}}}$ Data of Jaccodine

- Reported an energy equivalent value of 0.55 MPa$\sqrt{\text{m}}$.
- Used DCB w/ fracture mechanics solution that did not include $L/t$ effects.
- Reanalysis gives $K_{I_{\{111\}}} = 0.69 \pm 0.02$ MPa$\sqrt{\text{m}}$ (4):

\[\therefore \text{Engineering value } \sim 0.68 \pm 0.02 \text{ MPa}$\sqrt{\text{m}}$ for low index planes

Strength Testing

- Constant Stress Rate Tests (5 MPa/s)
- Biaxial Flexure ring-on-ring (ROR)
- ~400 grit as-ground surfaces in distilled, deionized water
- ~Polished surface in lab air

ASTM C1499
Fracture Strength & Weibull Statistics

- Polished $m = 6$; ground $m = 9$; spurious damage $m = 4$.
- Scale effect evident: 168 vs 215 MPa.
- Strength of 235 MPa is predicted vs 215 MPa (10%).
Biaxial Fracture Patterns (polished)

• Repetitive pattern that makes fractography difficult:
Fracture Path
- ground disk -

- Crack initiated at a grinding scratch.
- Transited to a low index planes.
- Deflected at a grain boundary.
Fracture Path in a Polished ROR Disk

- Crack initiated from a semi-elliptical crack emanating from a scratch.
- Turned onto the \{111\} plane:
  - Opportunity to estimate the fracture toughness!
  - \( K_{I\{hkl\}} = 0.73 \text{ MPa}\sqrt{\text{m}}. \)
  - Why did the crack turn?
Preferred Fracture Plane

- The fracture toughness on low index planes is similar, so why is the \{111\} the preferred propagation plane?
- The \{111\} is the stiffest direction, and stiff directions exhibit high stresses under displacement controlled situations (NiAl):

  • Stress concentration where the load ring intersects the stiff direction! Anisotropy changes the stress distribution.
Pressurized Plate

- Applying pressure avoids contacts:

![Diagram of pressurized plate]

- For a pressurized plate, the stress concentrations at stiff directions are not exhibited. Better test!
Pressurized Plate (POR)

- Measured strength is ~20% greater than expected from the ROR data because the stress concentration has been removed. ROR is conservative.
Fracture Toughness
– semi-elliptical cracks on high index planes -

- For polished specimens, $K_I = 0.77 \pm 0.04 \text{ MPa}\sqrt{\text{m}}$ (0.73-0.83).
- For grinding cracks, $K_I = 0.87 \pm 0.04 \text{ MPa}\sqrt{\text{m}}$ (0.80 – 0.90).
- Higher due to random orientation and transition to \{111\}.
- Caveat: local stress not precisely known…..
Slow Crack Growth
- Experimental Approach -

• Constant Stress Rate Testing “dynamic fatigue”
  - ASTM C1368

• Strength based approach with advantages & disadvantages:
  - rapid test; simple geometry
  - samples the inherent, small flaws
  - statistical scatter (many specimens needed)
  - averaging of fatigue regions
Experimental Procedure

• Constant Stress Rate Tests (5 to 5 x 10^-4 MPa/s)
• Biaxial Flexure (Ring-on-ring)
• Distilled, deionized water
• ~400 grit as-ground surfaces
• ~10 tests per stress rate
• ~40 tests
Slow Crack Growth Analysis

• Crack growth function:

\[ v = \frac{da}{dt} = AK_i^n = A \left( \frac{K_I}{K_{IC}} \right)^n \]

• Constant stress rate testing:

\[ \sigma_f = \left[ B(n+1)\sigma_i^{n-2} \dot{\sigma} \right]^{1/(n+1)} \]

\[ B = \frac{2K^{2-n}_{IC}}{AY^2(n-2)} = \frac{2K^2_{IC}}{AY^2(n-2)} \]

• Parameter extraction via regression:

\[ \log_{10} \sigma_f = \frac{1}{n+1} \log_{10} \dot{\sigma} + \log_{10} D \quad \log_{10} D = \frac{1}{n+1} \log_{10} \left[ B(n+1)\sigma_i^{n-2} \right] \]

(Slope \( \alpha \)) \quad (Intercept \( \beta \))
- Still some scatter.
- Medians clarify the trend.
- Slope is negative to zero $\therefore n > 100$, no measurable SCG.
Summary and Conclusions

• Ge exhibits similar fracture toughness of $K_I = 0.68 \pm 0.02 \text{ MPa} \sqrt{\text{m}}$ on low index planes. Lower than Si!

• Randomly oriented cracks exhibit higher apparent toughness, but turn and propagate on the stiff \{111\} directions due to higher stresses (?)…..FEA.

• Natural cleavage plane appears to be the \{110\}.

• Weibull modulus varies from $m = 4$ (spurious) to $m = 9$ (ground).

• Strength varies from $S_f = 40 \text{ MPa}$ (ground) to 160 MPa (polished).

• Ge exhibits a Weibull scale effect, but does not exhibit measurable SCG.
Summary and Conclusions

• Aggregate, polycrystalline Young’s modulus and Poisson’s ratio are $E_{\text{poly}} = 131$ GPa, $\nu_{\text{poly}} = 0.21$.

• ROR loading results in stress concentrations at the stiff directions of single crystals.

• From a stress state point-of-view, a lower strength measurement is expected………

• However, from an effective area perspective, a high strength should be measured.

• Pressure loading (POR) is a better test method for single crystals, because it avoids stress concentrations, but it is more effort……
Potential Future Work

- Cyclic fatigue testing
- Finite element analysis of ROR specimens
- Testing of more pressure-on-ring specimens
- Further SCF testing
- SCG testing in other environments
Acknowledgements

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