Structural Design Parameters for Germanium

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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..............
Young’s Modulus - impulse excitation -

- Well oriented germanium….

<table>
<thead>
<tr>
<th>$E_{111}$</th>
<th>$E_{110}$</th>
<th>$E_{100}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>154.8 ± 0.9 GPa</td>
<td>138.3 ± 0.2 GPa</td>
<td>103.1 ± 0.6 GPa</td>
</tr>
</tbody>
</table>

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

Aggregate Constants

<table>
<thead>
<tr>
<th>Formula</th>
<th>$E$ (GPa)</th>
<th>$\nu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Voigt</td>
<td>135</td>
<td>0.20</td>
</tr>
<tr>
<td>Hashin</td>
<td>133</td>
<td>0.21</td>
</tr>
<tr>
<td>Shtrikman</td>
<td>132</td>
<td>0.21</td>
</tr>
<tr>
<td>Reuss</td>
<td>129</td>
<td>0.21</td>
</tr>
</tbody>
</table>
Procedure
- Fracture Toughness -

• Three standard test methods (C1421):

  - Precracked Beam (SEPB)
    \[ \alpha = \frac{a_1 + a_2 + a_3}{3W} \]
  - Chevron Notch Beam (CNB)
    \[ \alpha_1 = \frac{a_1}{W}, \quad \alpha_0 = \frac{a_0}{W} \]
  - Surface Crack Flexure (SCF)
    Remove 4.5h to 5h

• 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.
- $K_{Iscf\{jkl\}} = 0.74 \pm 0.02$ MPa$\sqrt{m}$.
- $K_{Ipb\{100, 110\}} = 0.68 \pm 0.04$ MPa$\sqrt{m}$.
- ~10% difference between SCF and SEPB. Plasticity?
- Practical value of $K_{I\{jkl\}} = 0.68 \pm 0.02$ MPa$\sqrt{m}$.
SCF Fracture Surfaces

- {100} is conchodial 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?
Data of Jaccodine

\[ K_{I\{111\}} \]

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

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

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:

- For a pressurized plate, the stress concentrations at stiff directions are not exhibited. Better test!
• 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, $K_I = 0.77 \pm 0.04$ MPa√m (0.73-0.83).
- For grinding cracks, $K_I = 0.87 \pm 0.04$ MPa√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 $5 \times 10^{-4}$ 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:

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

- Constant stress rate testing:

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

- 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 \( \alpha \)) (Intercept \( \beta \))
Still some scatter.

Medians clarify the trend.

Slope is negative to zero : ∴ $n > 100$, no measurable SCG.
Summary and Conclusions

• Ge exhibits similar fracture toughness of $K_f = 0.68 \pm 0.02 \text{ MPa}^{\sqrt{m}}$ 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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