Slow Crack Growth of Germanium

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Germanium

• Good electromagnetic transmission in 2-15 µm range. Specialty window material:
Germanium

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

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

- 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.

- Relatively low. Running mechanical test on off-axis planes is problematic if the anisotropy is large.
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>GPa</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):
  - Precracked Beam (SEPB)
    \[ \alpha = \frac{a_1 + a_2 + a_3}{3W} \]
  - Chevron Notch Beam (CNB)
    \[ \alpha_t = \frac{a_t}{W}, \quad \alpha_o = \frac{a_o}{W} \]
  - Surface Crack Flexure (SCF)
    Remove 4.5h to 5h

• Different crack size
• Different crack formation history
• Different effort
• Relatively simple fixtures: test frame, load cell, recording device.
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>0.68 ± 0.04</td>
<td>0.68 ± 0.01</td>
<td>&lt; 0.74</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>
<tr>
<td>CNB</td>
<td>In progress</td>
<td>In progress</td>
<td>In progress</td>
</tr>
</tbody>
</table>

- Essentially the same on all planes
- $K_{Iscf_{jkl}} = 0.74 \pm 0.02 \text{ MPa}\sqrt{\text{m}}$
- $K_{Ipb_{\{100}, \{110\}}} = 0.68 \pm 0.04 \text{ MPa}\sqrt{\text{m}}$
- ~10% difference between SCF and SEPB. Plasticity?
- Practical value of $K_{I_{jkl}} = 0.70 \pm 0.02 \text{ MPa}\sqrt{\text{m}}$. 
SCF Precracks

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

- Peak of cathedral corresponds to the <100> {100}. 


$K_{I\{111\}}$ Data of Jaccodine

- 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 $K_{I\{111\}} = 0.72 \pm 0.05$ MPa$\sqrt{m}$ (6) w/ trend toward 0.67 MPa$\sqrt{m}$:

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

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 strain controlled situations……

Stress concentration where the load ring intersection the stiff direction.

But, for a pressurized plate, the stress concentrations are not exhibited.
Fracture Toughness
– semi-elliptical cracks on high index planes -

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

\[ \nu = \frac{d\alpha}{dt} = AK_i^n = A \left( \frac{K_I}{K_{IC}} \right)^n \]

- Constant stress rate testing:

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

- 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 $K_f = 0.68 \pm 0.02 \text{ MPa}\sqrt{\text{m}}$ 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 Yong’s modulus and Poisson’s ratio are $E_{poly} = 131$ GPa, $\nu_{poly} = 0.21$.

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

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

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

• POR maybe a better test method, but more effort.
Acknowledgements

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• Thanks to Rick Rogers for x-ray diffraction.
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