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”\(\Phi\)):

  ![Image of coarse gained disks]

- 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 -

- $E_{111} = 154.8 \pm 0.9$ GPa
- $E_{110} = 138.3 \pm 0.2$
- $E_{100} = 103.1 \pm 0.6$

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

Aggregate Constants

<table>
<thead>
<tr>
<th>Aggregate Constants</th>
<th>GPa</th>
<th>$E$</th>
<th>$\nu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Voigt</td>
<td>135</td>
<td>0.20</td>
<td></td>
</tr>
<tr>
<td>Hashin</td>
<td>133</td>
<td>0.21</td>
<td></td>
</tr>
<tr>
<td>Shtrikman</td>
<td>132</td>
<td>0.21</td>
<td></td>
</tr>
<tr>
<td>Reuss</td>
<td>129</td>
<td>0.21</td>
<td></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>$E_{100}$</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>$E_{110}$</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>$E_{111}$</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)
  - Chevron Notch Beam (CNB)
  - Surface Crack Flexure (SCF)

\[ \alpha = \frac{a_1 + a_2 + a_3}{3W} \]

\[ \alpha_0 = \frac{a_0}{W} \]

\[ \alpha_1 = \frac{a_1}{W} \]

- Different crack size
- Different crack formation history
- Different effort
Loading Configuration
- Fracture Toughness -

- 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
- $K_{Iscf\{jkl\}} = 0.74 \pm 0.02$ MPa√m
- $K_{Ipb\{100, 110\}} = 0.68 \pm 0.04$ MPa√m
- ~10% difference between SCF and SEPB. Plasticity?
- Practical value of $K_{I\{jkl\}} = 0.70 \pm 0.02$ MPa√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.
• 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\(\sqrt{m}\):

$$\therefore$$ Engineering value

\~0.68 MPa\(\sqrt{m}\) 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 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, $K_I = 0.77 \pm 0.04 \text{ MPa} \sqrt{\text{m}} (0.73-0.83)$.
• 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:
  \[ \sigma_f = \left[ B(n + 1)\sigma_i^{n-2} \dot{\sigma} \right]^{1/(n+1)} \]
  \[ B = \frac{2K_{Ic}^{2-n}}{AY^2(n-2)} = \frac{2K_{Ic}^2}{AY^2(n-2)} \]

- Parameter extraction via regression:
  \[ \log_{10} \sigma_f = \frac{1}{n + 1} \log_{10} \dot{\sigma} + \log_{10} D \]
  \[ \log_{10} D = \frac{1}{n + 1} \log_{10} \left[ B(n + 1)\sigma_i^{n-2} \right] \]
  (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 $K_f = 0.68 \pm 0.02$ 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 $S_f = 40$ 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 $E_{\text{poly}} = 131$ 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 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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