Observations in Fracture Toughness Testing of Glasses and Optical Ceramics

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Jon Salem
NASA GRC
OUTLINE

• Background
• Test methods employed
• Results for ALON, spinel, fused silica, glass
• Interferences (complications) encountered:
  - Stress corrosion and rate effects
  - Stability
  - Crack length measurement (grain size)
  - Damaged specimens
  - Crack growth resistance

• Summary
Importance of Fracture Toughness

- Governs the fracture strength by way of the flaw distribution.
- Determines the end of the slow crack growth curve:
  - Needed to determine design parameter from data.
  - Thus a critical structural design parameter.
  - And an excellent metric to rank materials.
Fracture Toughness Definitions

- **Fracture toughness**—a generic term for measures of resistance of extension of a crack. *(E399, E1823)*
- ASTM E399: crack extension resistance at the onset of crack extension under specific operational conditions (stable or unstable).
- C1421: *fracture toughness* $K_{Ivb}$ - the measured stress intensity factor corresponding to the extension resistance of a stably-extending crack in a chevron-notched test specimen.
- For engineering purposes, generally boils down to a procedure specific value (real, measurable crack; defined configuration).
How Variable were Fracture Toughness Measurements prior to Standardization?

- $\alpha$ SiC:

<table>
<thead>
<tr>
<th></th>
<th>Precracked beam (SEPB)</th>
<th>Surface crack in flexure (SCF)</th>
<th>Chevron-notch (CNB)</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2.41 ± 0.14</td>
<td>3.01 ± 0.35</td>
<td>2.91 ± 0.31</td>
<td>A</td>
</tr>
<tr>
<td></td>
<td>3.01 ± 0.06</td>
<td>3.01 ± 0.06</td>
<td>2.71</td>
<td>B</td>
</tr>
<tr>
<td></td>
<td>3.45 ± 0.15</td>
<td></td>
<td></td>
<td>C</td>
</tr>
<tr>
<td></td>
<td>3.31 ± 0.19</td>
<td></td>
<td></td>
<td>D</td>
</tr>
<tr>
<td></td>
<td>3.11 ± 0.26</td>
<td></td>
<td></td>
<td>E</td>
</tr>
<tr>
<td></td>
<td>3.00 ± 0.04</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>3.04 ± 0.24</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.82 ± 0.31</td>
<td></td>
<td></td>
<td>F</td>
</tr>
<tr>
<td></td>
<td>3.10</td>
<td></td>
<td></td>
<td>G</td>
</tr>
</tbody>
</table>

- Greater than 30% difference in literature!!
Standard Method C1421

- ASTM C28 developed standard C1421 for fracture toughness testing of ceramics.
- First draft written in 1998; finished in 1999. My goal was to standardize multiple methods and keep individual errors below 2%.
- The standard covers three methods.
- Very similar results from all three methods.
- Emphasis was on structural ceramics for heat engine applications.

- Is C1421 applicable to glasses and optical ceramics? What issues need address?
C1421 STANDARIZED TEST METHODS

- Flexure test specimens with various cracks:
  - Different crack size
  - Different crack formation history
  - Different effort

\[
K_{Ipb} \quad K_{Ivb} \quad K_{Isc}
\]

- Similar results for ceramics!

Remove 4.5h to 5h
Relatively simple fixtures: test frame, load cell, recording device.
**HOW GOOD CAN FRACTURE TOUGHNESS MEASUREMENTS BE WITH C1421?**

<table>
<thead>
<tr>
<th>Material</th>
<th>$K_{fvb}$</th>
<th>$K_{Ipb}$</th>
<th>$K_{Isc}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-SiC (JAS)</td>
<td>2.61 ± 0.05</td>
<td>2.58 ± 0.08</td>
<td>2.76 ± 0.08</td>
</tr>
<tr>
<td>α-SiC (UW)</td>
<td>2.62 ± 0.06</td>
<td>2.54 ± 0.20</td>
<td>2.69 ± 0.08</td>
</tr>
<tr>
<td>ADS96R</td>
<td>3.56 ± 0.03</td>
<td>3.71 ± 0.10</td>
<td>----</td>
</tr>
<tr>
<td>ALSIMAG 614</td>
<td>3.19 ± 0.06</td>
<td>3.09 ± 0.17</td>
<td>3.18 ± 0.10</td>
</tr>
<tr>
<td>ALSIMAG 614</td>
<td>3.13 ± 0.03</td>
<td>2.98 ± 0.06</td>
<td>----</td>
</tr>
<tr>
<td>NC132</td>
<td>4.60 ± 0.13</td>
<td>4.59 ± 0.12</td>
<td>4.55 ± 0.14</td>
</tr>
<tr>
<td>NT154</td>
<td>5.18 ± 0.11</td>
<td>5.21 ± 0.02</td>
<td>5.80 ± 0.23</td>
</tr>
<tr>
<td>SN260</td>
<td>5.19 ± 0.06</td>
<td>5.13 ± 0.15</td>
<td>----</td>
</tr>
<tr>
<td>SiAlON</td>
<td>----</td>
<td>2.45 ± 0.09</td>
<td>2.55 ± 0.05</td>
</tr>
</tbody>
</table>

- Three methods are usually within ~3% for “structural” ceramics. 30% => 3%.
# C1421 Captures Grain Size effects on Toughness

<table>
<thead>
<tr>
<th>Silicon Nitride</th>
<th>( K_{Ivb} (A) ) MPa(\sqrt{m} )</th>
<th>( K_{Ipb} ) MPa(\sqrt{m} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>NKK</td>
<td>10.4 ± 0.5</td>
<td>10.3 ± 0.6</td>
</tr>
<tr>
<td>AS440</td>
<td>7.2 ± 0.3</td>
<td>7.3 ± 0.3</td>
</tr>
<tr>
<td>AS800 (light)</td>
<td>7.40 (1)</td>
<td>7.6 ± 0.2</td>
</tr>
<tr>
<td>AS800 (dark)</td>
<td>6.9 ± 0.1</td>
<td>7.2 ± 0.2</td>
</tr>
<tr>
<td>PY6</td>
<td>6.3 ± 0.1</td>
<td>6.2 ± 0.1</td>
</tr>
<tr>
<td>SRBSN 147-31N</td>
<td>5.3 ± 0.2</td>
<td>5.6 ± 0.2</td>
</tr>
</tbody>
</table>

Increasing grain size

- For ceramics, C1421 provides consistent results with different methods despite different crack geometry.
Glass and Optical Materials Tested

- Fused Silica
- Soda-Lime Silicate Glass
- ALON: GS = 235µm

- Spinel (MgAlO₄): GS = 220µm:
Fused Silica

Russian Quartz-Silica

<table>
<thead>
<tr>
<th>Fracture Toughness (MPa√m)</th>
<th>Method</th>
<th>Air (75°F, 45% RH)</th>
<th>Dry N₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>SEPB</td>
<td>0.71 ± 0.05</td>
<td>0.73 ± 0.02</td>
<td></td>
</tr>
<tr>
<td>VB</td>
<td>0.71 ± 0.04</td>
<td>0.77 ± 0.01</td>
<td></td>
</tr>
</tbody>
</table>

Shuttle Fused Silica (7940)

<table>
<thead>
<tr>
<th>Fracture Toughness (MPa√m)</th>
<th>Method</th>
<th>Air (75°F, 45% RH)</th>
<th>Dry N₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>SEPB</td>
<td>0.71 ± 0.04</td>
<td>0.74 ± 0.03</td>
<td></td>
</tr>
<tr>
<td>VB</td>
<td>0.73 ± 0.04</td>
<td>0.77 ± 0.02</td>
<td></td>
</tr>
</tbody>
</table>

Literature on 7940 & 7980

<table>
<thead>
<tr>
<th>Fracture Toughness (MPa√m)</th>
<th>Method</th>
<th>Vacuum</th>
<th>Vacuum/Dried</th>
<th>Dry N₂</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>AMDCB</td>
<td></td>
<td></td>
<td></td>
<td>0.72 ± 0.01</td>
<td>NIST (LB)</td>
</tr>
<tr>
<td>DCB</td>
<td>0.74 ± 0.03</td>
<td></td>
<td>0.73 ± 0.02</td>
<td></td>
<td>NIST (SW)</td>
</tr>
<tr>
<td>3-Pt</td>
<td>0.75 ± 0.03</td>
<td></td>
<td></td>
<td>0.76</td>
<td>NIST (SW)</td>
</tr>
<tr>
<td>DCDC</td>
<td>0.73 to 0.76</td>
<td></td>
<td></td>
<td></td>
<td>Sandia</td>
</tr>
</tbody>
</table>

- Conclusion: very similar results by all. Metrology is OK!
- Toughness in air is less than in nitrogen.
Soda Lime Silicate Glass

<table>
<thead>
<tr>
<th>Load Rate (mm/min)</th>
<th>0.005 (54%RH)</th>
<th>0.01 (53%)</th>
<th>0.02 (40%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K_{Ivb}$ (MPa√m)</td>
<td>0.65 ± 0.02</td>
<td>0.67 ± 0.03</td>
<td>0.74 ± 0.03</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Load Rate (mm/min)</th>
<th>0.005 (LP N$_2$)</th>
<th>0.02 (LP N$_2$)</th>
<th>0.005 (HP N$_2$)</th>
<th>0.02 (HP N$_2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K_{Ivb}$ (MPa√m)</td>
<td>0.76 ± 0.01</td>
<td>0.75 ± 0.01</td>
<td>0.76 ± 0.01</td>
<td>0.79 ± 0.02</td>
</tr>
</tbody>
</table>

- Systematic increase with test speed and decrease of humidity.
- Differences are associated w/ rate and environment: SCG!! Go dry!
So, what is Fracture Toughness of a Glass?

- In humid environments, fracture toughness measurements are points on the slow crack growth curve:

- The unique value, $K_{ic}$, corresponds to a dry environment.

Log Stress Intensity Factor, $K/I/K_{ic}$
Complication: Unstable Fracture

- In dry environments, stable crack extension can be difficult to obtain:

- Required for the chevron-notch. Less of an issue for SEPB and SCF methods.
Solutions: precracking & scratching

- Precrack in air; fail in N\textsubscript{2}:

![Graph showing BK7 Fracture Toughness](image)

- Precracking
- N\textsubscript{2}
- Air
Complication: Cracked Specimens

- Some materials are very fragile and can be cracked:
- Results in low compliance:
- Bonding agents need to be removed:
ALON

<table>
<thead>
<tr>
<th>$K_{Ivb}(A)$ (MPa√m)</th>
<th>$K_{Ipb}$</th>
<th>$K_{Isc}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>~40% RH</td>
<td>$N_2$</td>
<td>~50% RH</td>
</tr>
<tr>
<td>2.09 ± 0.06</td>
<td>2.18 ± 0.10</td>
<td>2.02 ± 0.04</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Stress Rate (MPa/s)</th>
<th>0.002 (H$_2$O)</th>
<th>2 (H$_2$O)</th>
<th>20 (H$_2$O)</th>
<th>20 ($N_2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K_{Isc}$ (MPa√m)</td>
<td>1.80 ± 0.07</td>
<td>1.92 ± 0.06</td>
<td>2.07 ± 0.06</td>
<td>2.36 ± 0.1</td>
</tr>
</tbody>
</table>

- Similar results for the three standard methods.
- Again, differences are associated w/rate and environment: SCG!! Go dry!
Spinel

<table>
<thead>
<tr>
<th>45%–65% RH</th>
<th>N₂ (MPa√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K_{lpb}$</td>
<td>$K_{Ivb}$</td>
</tr>
<tr>
<td>1.32 ± 0.05</td>
<td>1.48 ± 0.14</td>
</tr>
</tbody>
</table>

$\Delta = 11\%$ for air; $\Delta = 4\%$ for N₂

- Similar results for two of the standard methods.
- Numbers converge for nitrogen!
Complication: Crack Length Measurement

- Crack front apparent...until the specimen is broken:

- Solution is a strain gage......

a/W \propto (P/\varepsilon).

- Or use the chevron, which does not need crack length.
Complication: Crack Length Measurement

- Crack front apparent not always apparent in SCF specimens:

- Less than ideal shapes due to grain structure:
Complication: Crack Growth Resistance

• Some optical materials have a coarse grain structure. This can cause an R-curve:

![Image of MS ZnS GS ≈ 100µm]

• ASTM C1421 techniques provide relative low values on the R-curve for an alumina.
Complication: Thin Materials

- Double torsion specimen (Non-Standard). Thin plate loaded on one edge:

- Comparable results for some materials………..
Non-Standard Option: Double torsion

- Results compare well for fine grained materials:

- For coarse grained materials, crack growth resistance causes elevated values relative to short crack methods.
Interferences (complications) to Address when Testing Glasses and Optical Ceramics

• Stress corrosion (humidity and rate)
• Stable crack extension (CNB)
• Crack length measurement (SEPB & SCF)
• Specimen damage (CNB)
• Crack growth resistance
## Data Ranges

<table>
<thead>
<tr>
<th>Glasses</th>
<th>Glass Ceramics</th>
<th>Crystalline Optical Ceramics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fracture toughness (MPa√m) of glasses and optical materials via C1421</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Glasses < 1
- **Glasses**
  - MOMA: 0.57 ± 0.03 (30/74) → 0.69 ± 0.01 (30/74)
  - Corning 0120: 0.50 ± 0.02 (34/76) → 0.67 ± 0.02 (34/76)
  - Electro-Glass 2164: 0.61 ± 0.05 (32/73) → 0.74 ± 0.03 (32/73)
  - Schott S8061: 0.64 ± 0.01 (23/73) → 0.72 ± 0.02 (23/73)
  - Schott 8330: 0.61 ± 0.04 (60/73) → 0.72 ± 0.04 (60/73)
  - Soda lime silicate: 0.75 ± 0.04 (35/73) → 0.80 ± 0.01 (35/73)
  - Ba-doped: 0.72 ± 0.002 (23/73) → 0.76 ± 0.01 (23/73)
  - Corning Silica, 7980: 0.73 ± 0.04 (45/75) → 0.77 ± 0.02 (45/75)
  - BK-7: 0.87 ± 0.02 (24/73) → 0.98 ± 0.04 (24/73)

### Glass Ceramics
- **Glass Ceramics**
  - Zerodur: 0.89 ± 0.01 (45/73) → 0.94 ± 0.01 (45/73)
  - Schott S8070: 1.57 ± 0.03 (60/73) → 1.90 ± 0.03 (60/73)

### Crystalline Optical Ceramics
- **Crystalline Optical Ceramics**
  - ALON: 2.09 ± 0.06 (40/70) → 2.18 ± 0.10 (40/70)
  - Ge: 0.67 ± 0.03 (65/74) → No SCG (65/74)
  - Sapphire (a) <m>: 2.06±0.21 → 2.31±0.11 (65/74)
  - Sapphire (r) <a>: 1.96 → 2.47±0.15 (65/74)
  - Sapphire (m) <>: --- → --- (65/74)
  - Spinel: 1.48 ± 0.14 (60/76) → 1.58 ± 0.10 (60/76)
  - YVO₄ (a) <a>: 0.48 ± 0.02 (45/73) → --- (45/73)
  - YVO₄ (a) <c>: 0.42 ± 0.03 (45/73) → --- (45/73)
  - ZnS: 0.74 ± 0.03 (60/72) → 0.82 ± 0.02 (60/72)
  - MS ZnS: 0.69 ± 0.03 (58/74) → 0.74 ± 0.14 (58/74)
SUMMARY

• Fracture toughness measurements of glasses are points on the SCG curve.
• Thus rate and environment effects occur.
• These effects can be minimized and consistent results obtained by using faster rates or dryer environments.
• The limiting value occurs in a very dry environment.
• ASTM C1421 is generally applicable with appropriate considerations.
Suggested Changes to C1421

• Recommend testing in a dry environment
• For air, ensure a narrower range of rates
• Notes on precracking for dry testing (CNB)
• Guidance on precrack measurements (SEPB, SCF)
Air vs Nitrogen (or Vacuum)

• Common results:

• Due to SCG:

• Need dryer environment or faster rates to approach the limiting value.
So, what is Fracture Toughness of a Glass?

- In humid environments, fracture toughness measurements are points on the slow crack growth curve:

  ![Fracture Toughness Diagram]

  - The unique value, $K_{Ic}$, corresponds to a dry environment.

So, what is Fracture Toughness of a Glass?