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Sandia National Laboratories

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AWE Aldermaston, UK

12:10 Thursday 12th Sept. - Session 10b
Manufacturing & Processing Capability

Glass-Ceramics for Improved Strength and Toughness Glass-to-Metal Seals

R. Tandon,

K.G. Ewsuk, A. Eller & C. Newton

U.S. DEPARTMENT OF ENERGY | NNSA Nuclear Security Administration

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Glass-Ceramics Combine Attributes of A Glass (Processability) And A Ceramic (Properties)

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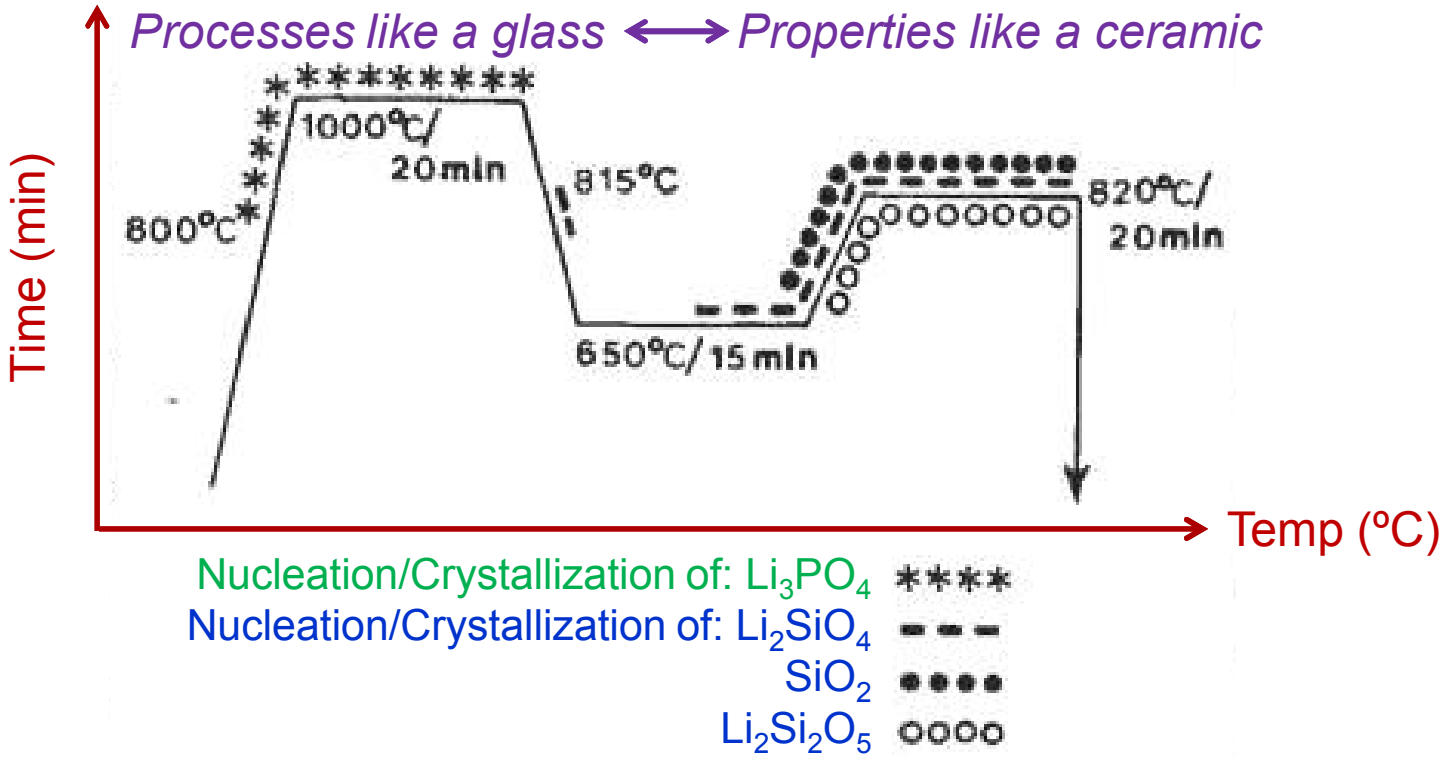
Thermal Processing Determines The Microstructure & Properties Of The Glass-Ceramic



Headley & Loehman, "Crystallization of a Glass-Ceramic by Epitaxial Growth", J Am Ceram Soc, 67 [9] 620-25 (1984).

Objective: Determine Why Glass-Ceramics Are More Crack Tolerant Than Glass

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- **Approach**
  - Characterize Glass-Ceramic Strength & Toughness
    - Modified Li-Silicate Glass Compositions
      - Heat-Treated To Produce Different CTE Glass-Ceramics
- **Characterization Tools**
  - Strength
    - 4-Point Bend Test
  - Fractography
    - Scanning Electron Microscopy (SEM)
  - Toughness
    - Single Edged V-Notched Beam (SEVNB)
    - Indentation Crack Length

4-Point Bend Strength Was Corrected To Account For The Non-Ideal GC Bar Geometry

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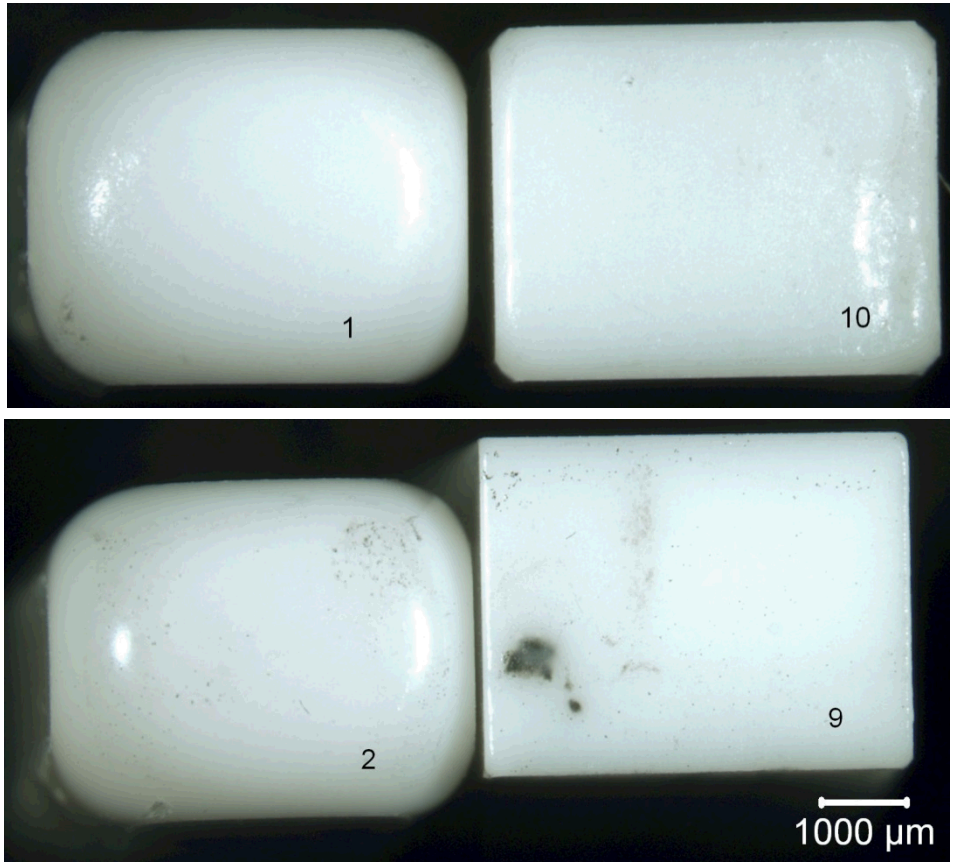
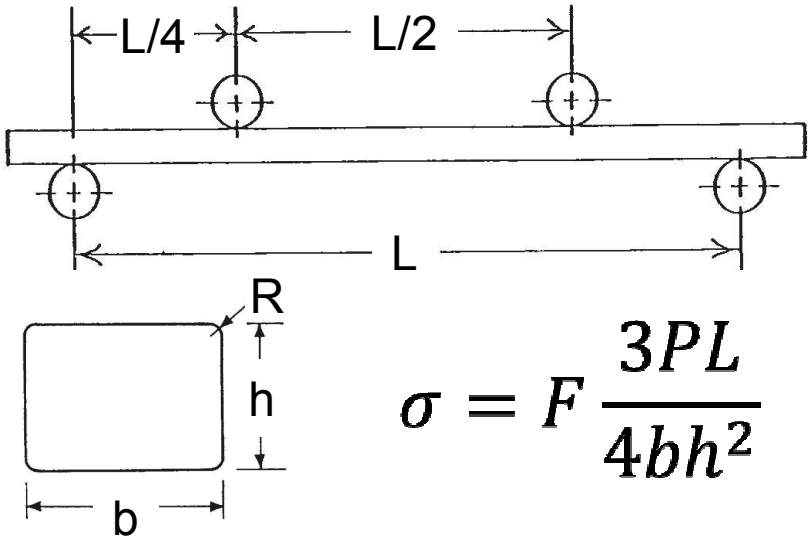
Glass Bars Were Reflowed In Graphite Molds To Produce GC Bend Bars



4-Point Bend Strength Was Measured On The "As-Formed" GC Bars

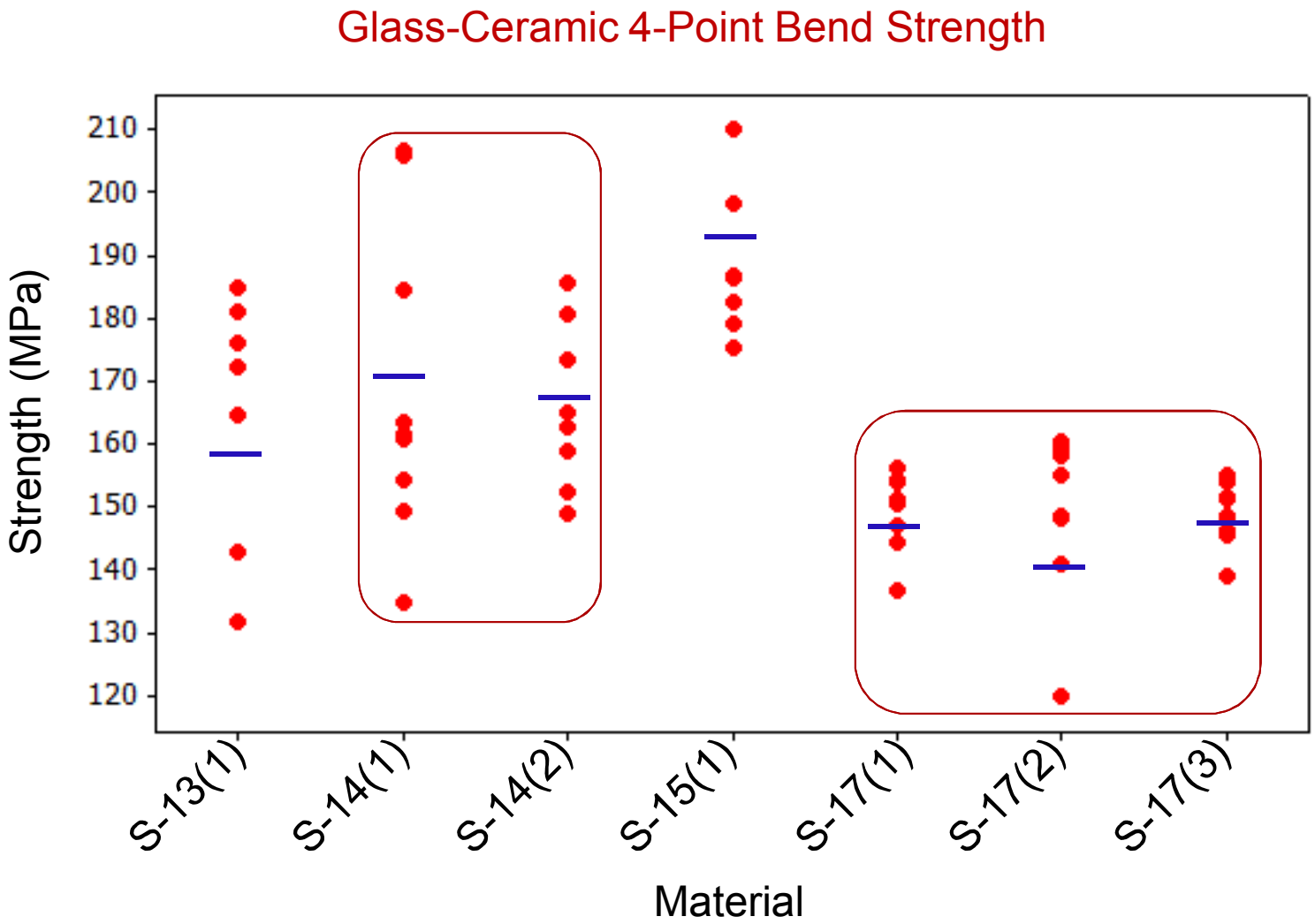


$$\sigma = F\frac{3PL}{4bh^2}$$

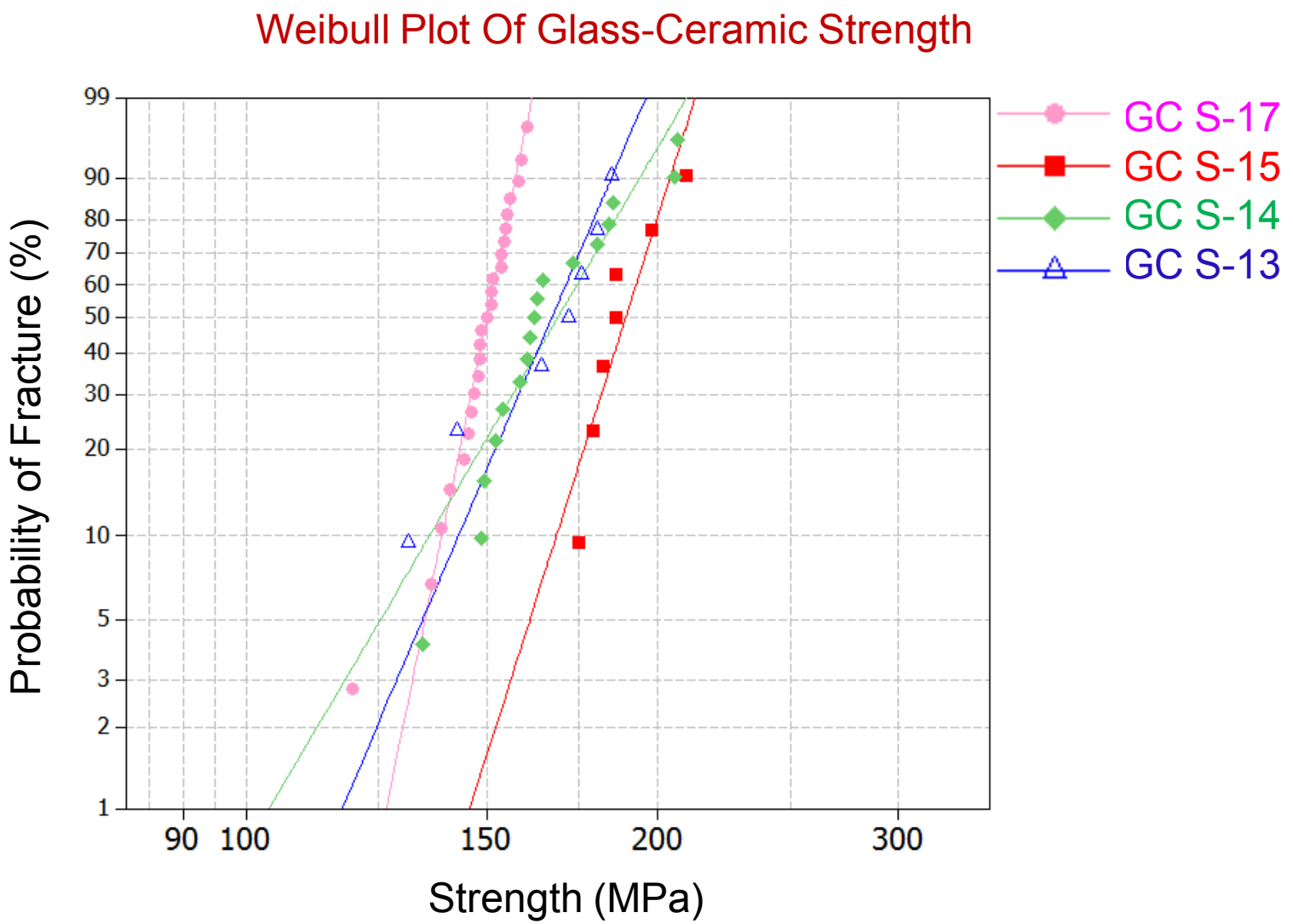
σ = Bend Strength
P = Load
F = Corner R Correction Factor

Flexural Strength of Advanced Ceramics at Ambient Temperature - ASTM C1161 (2008)

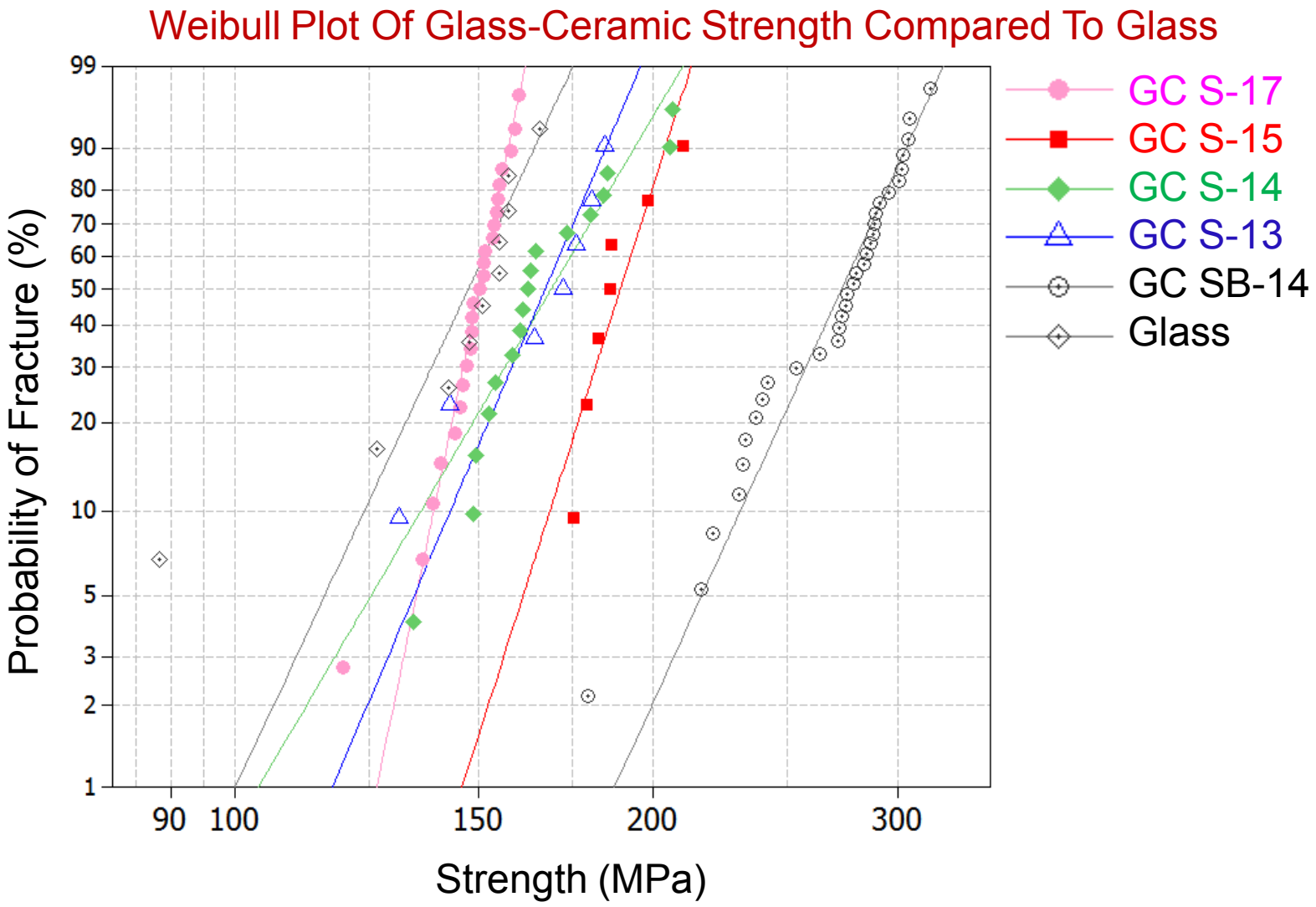
As-Formed Glass-Ceramic Strength Varies With Thermal Processing/CTE



S Glass-Ceramic With A CTE Of 15 Has The Highest Strength



S Glass-Ceramic Strength Is Comparable to The Strength Of Glass



Glass-Ceramic Bend Strength Decreases With Increasing Flaw Size

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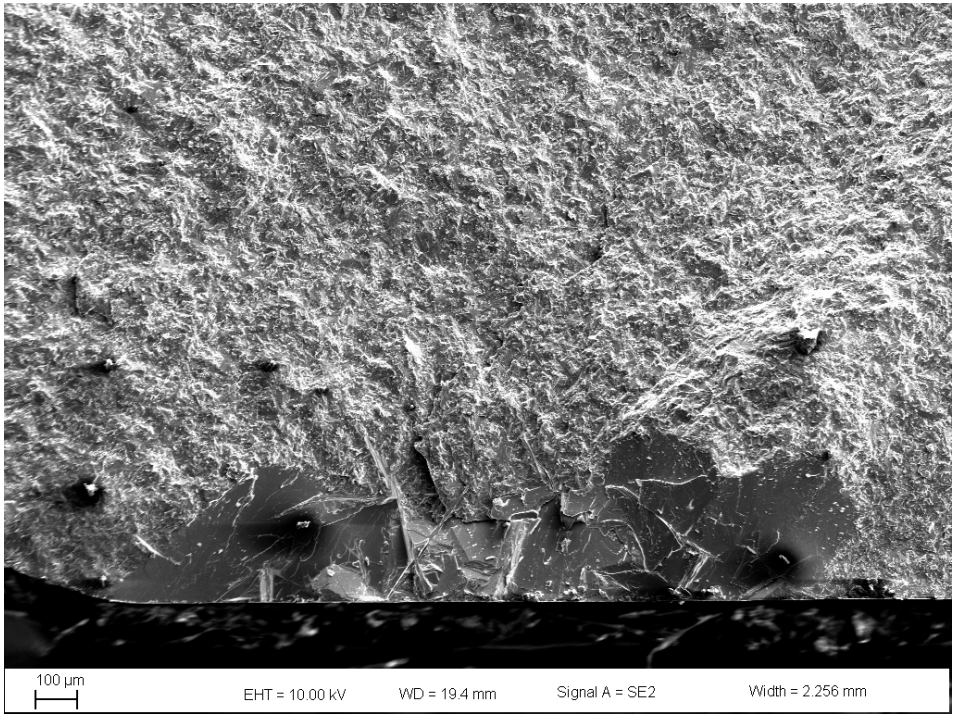
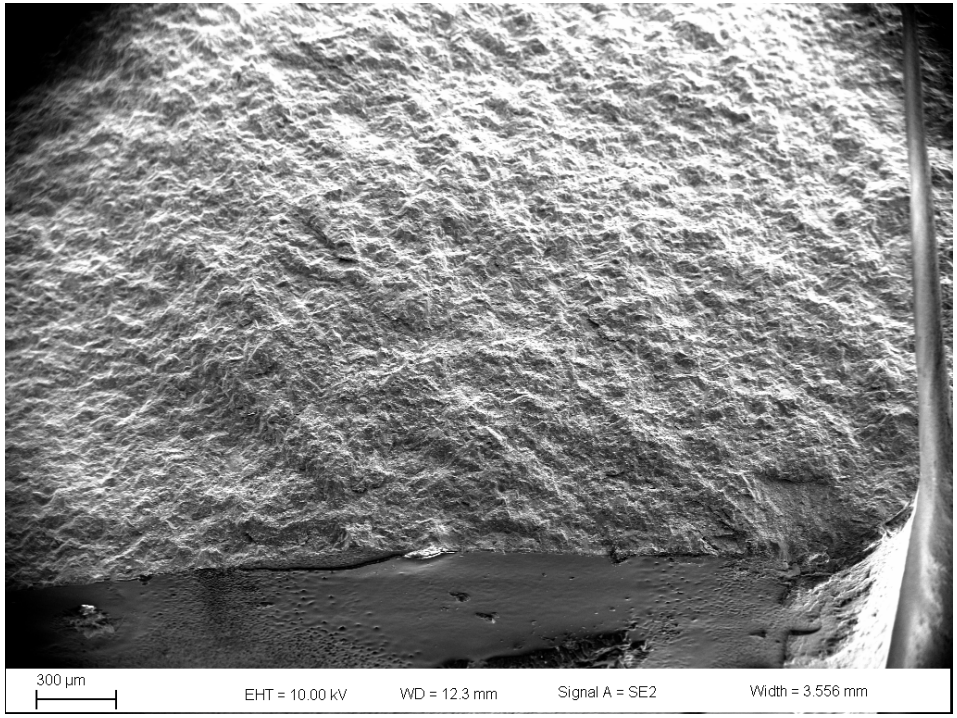
Fractography of GC S-17 Fracture Surfaces



Low Strength



High Strength

Glass-Ceramic Bend Strength Decreases With Increasing Flaw Size

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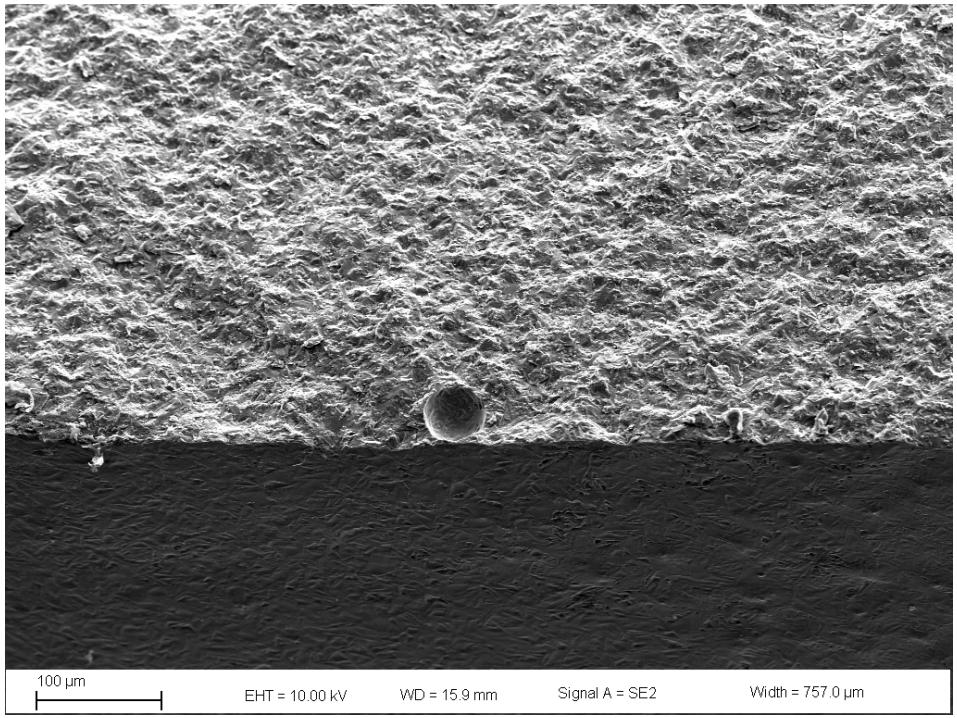
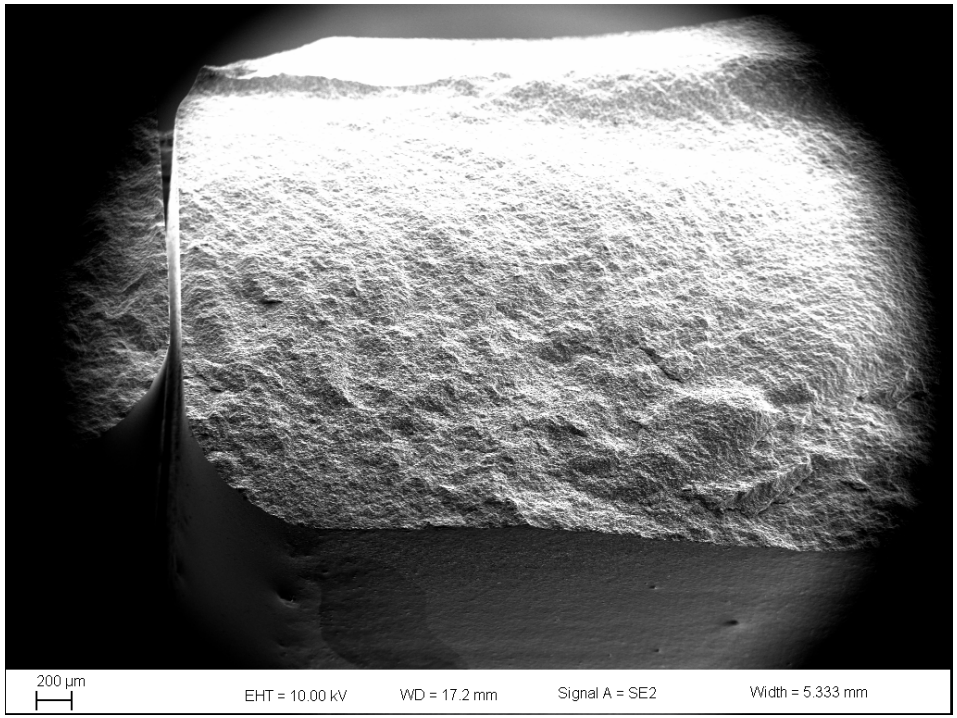
Fractography of GC S-15 Fracture Surfaces



Low Strength



High Strength

Glass-Ceramic Bend Strength Decreases With Increasing Flaw Size

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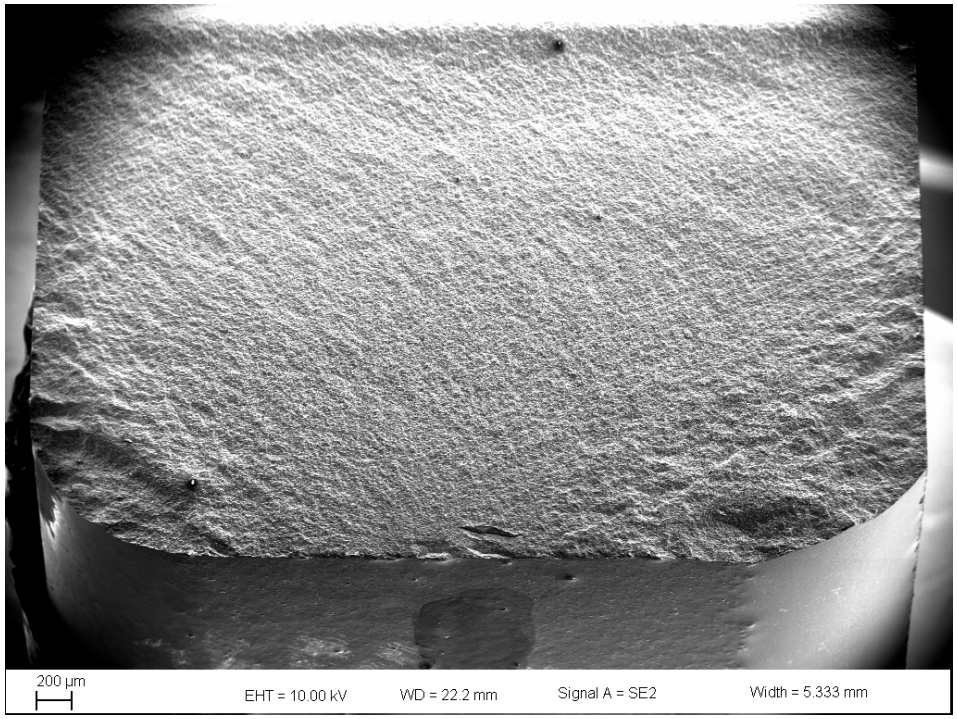
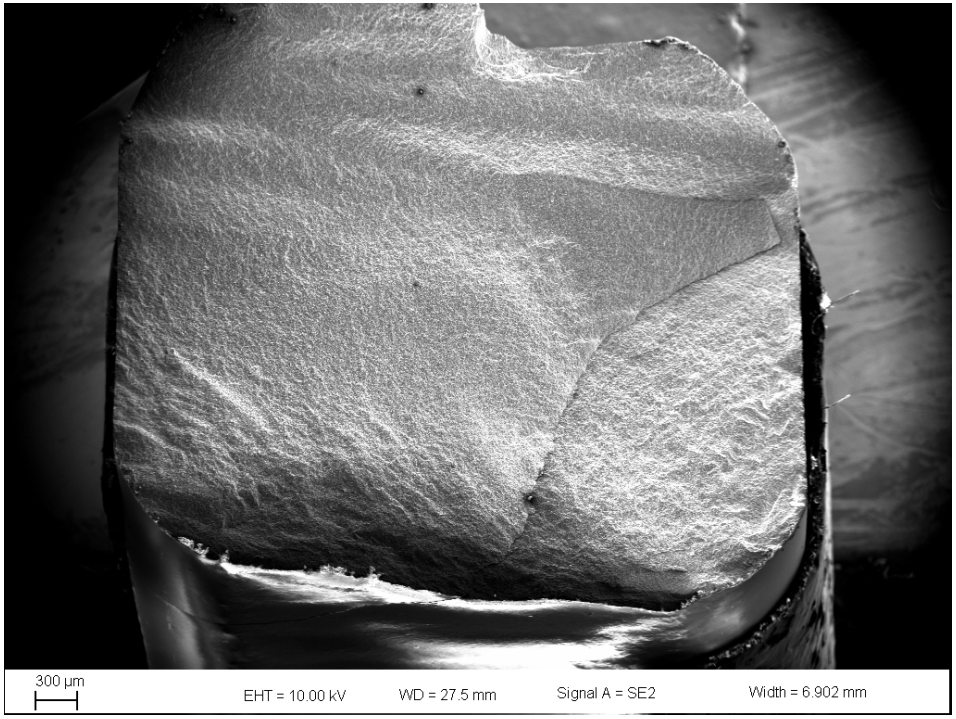
Fractography of GC S-13 Fracture Surfaces



Low Strength



High Strength

GC Fracture Toughness Was Measured Using A Single Edged V-Notched Beam (SEVNB)

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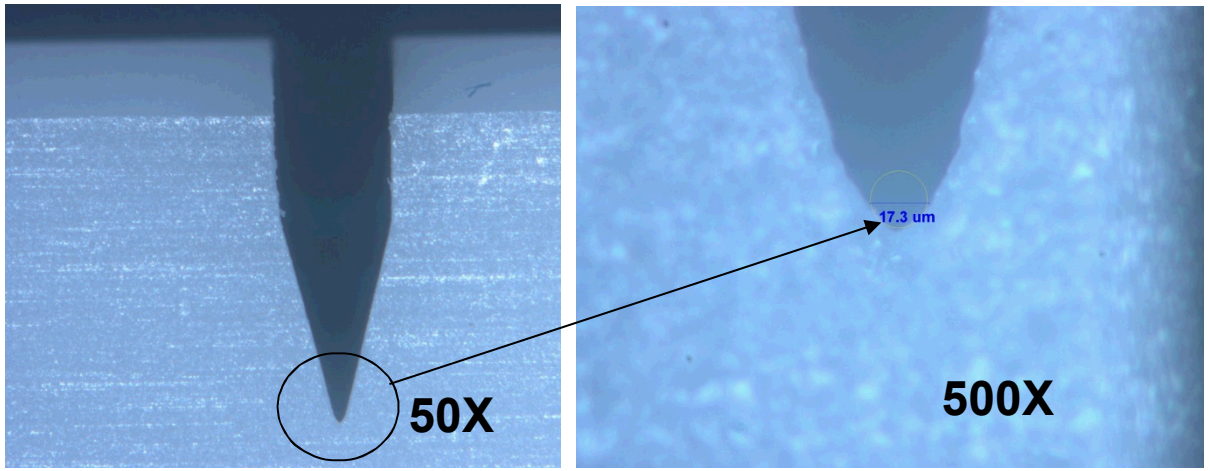
A Sharp Tip Notch (r < 20mm) Was Machined Into A GC Bend Bar

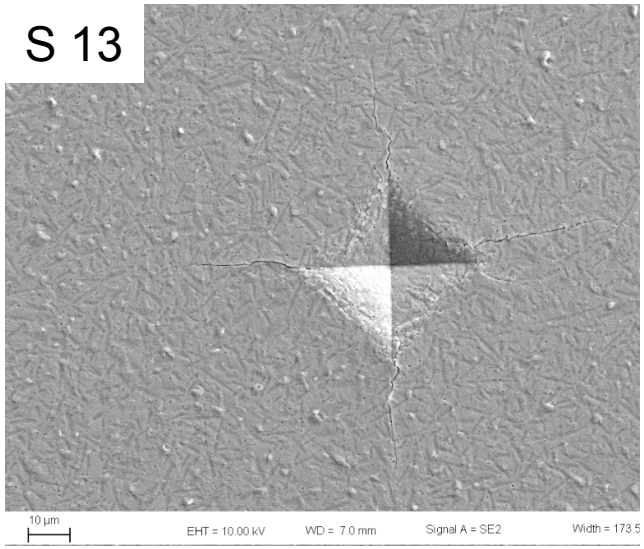


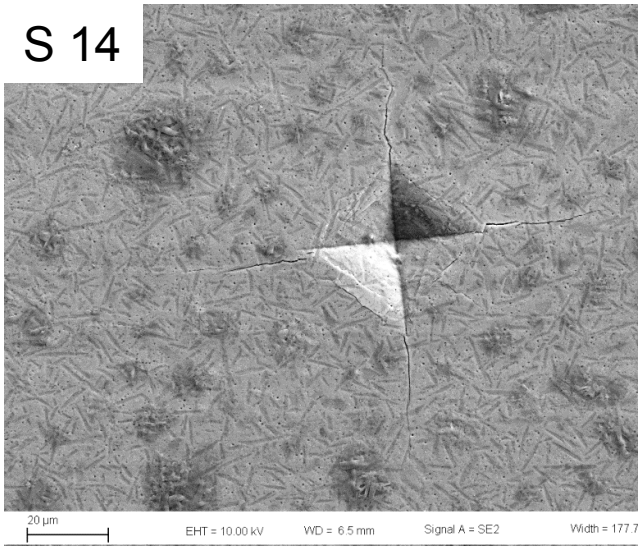
$$K_I = \left[Y' h \frac{\alpha^{1/2}}{(1-\alpha)^{3/2}} \right] \left[\frac{3}{2} P \frac{L/2}{bh^2} \right]$$

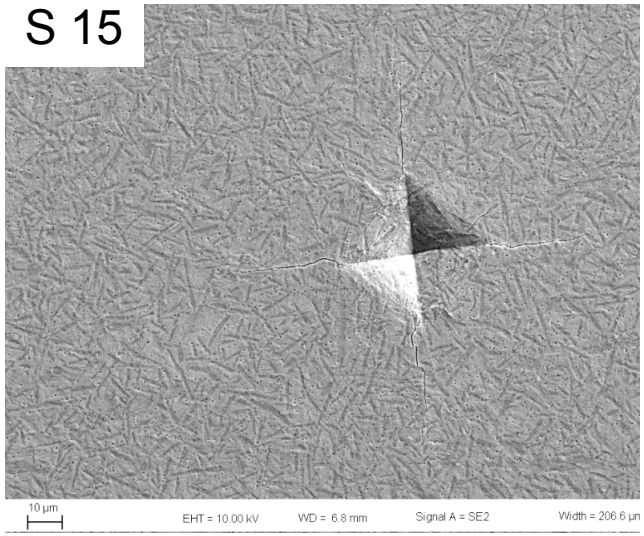
$$Y' = 1.9887 - 1.326\alpha - (3.49 - 0.68\alpha + 1.35\alpha^2)\alpha(1-\alpha)(1+\alpha)^{-2}$$

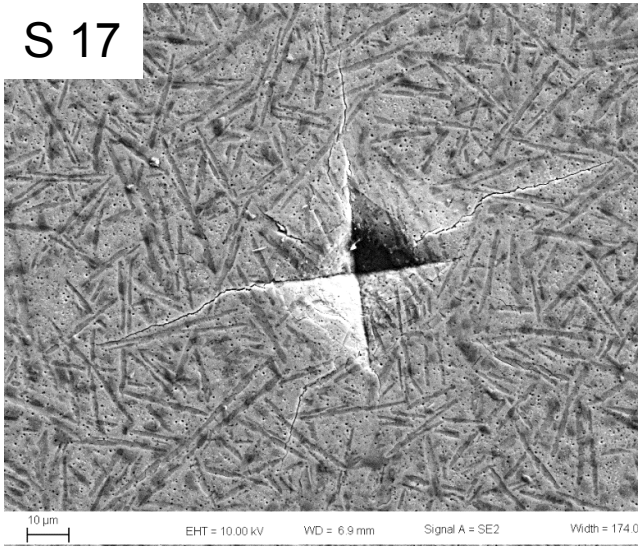
$$\alpha = \frac{length\ of\ notch}{h}$$

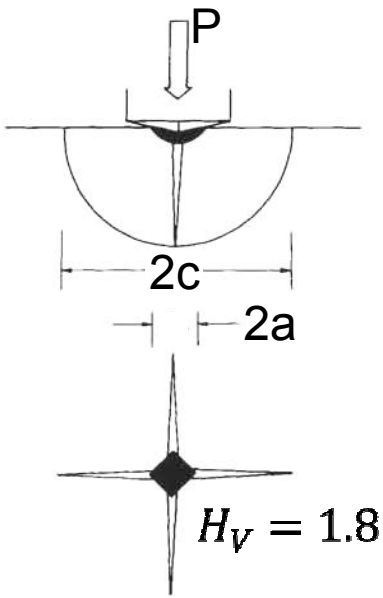
GC Toughness (K_{IC}) & Hardness (H_V) Were Determined From Vickers Indentation











$$H_V = 1.8544 \frac{P}{(2a)^2}$$

$$K_{IC} = 0.016 \frac{P}{C^{3/2}} \left(\frac{E}{H_V}\right)^{1/2}$$

Anstis et al., 'A Critical Evaluation of Indentation Techniques for Measuring Fracture Toughness: I, Direct Crack Measurements," J Am Ceram Soc 64 [9] 633-8 (1981)

Glass-Ceramics Are Tougher Than Glass

Sample	K_{IC} (MPa·m$^{1/2}$)	Vickers Hardness (GPa)	Measurement Method
Glass (typical)	0.7-0.8	6-7	
GC SB-11	1.97 ± 0.05	---	SEVNB
GC SB-14	2.76 ± 0.16	---	SEVNB
GC S-13	1.52 ± 0.26	4.61 ± 0.26	Indentation
GC S-14	1.79 ± 0.32	4.60 ± 0.17	Indentation
GC S-15	1.92 ± 0.22	4.18 ± 0.24	Indentation
GC S-17	1.40 ± 0.26	4.41 ± 0.59	Indentation

The Glass-Ceramic Crystal Structure Gets Coarser With Increasing CTE

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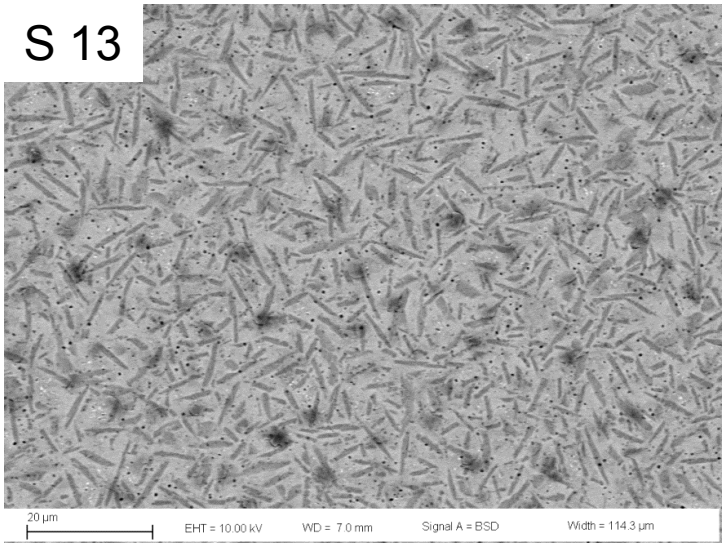
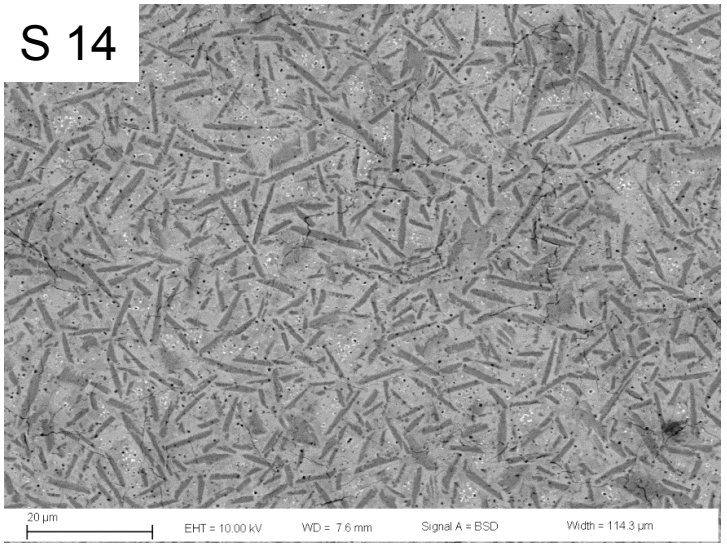
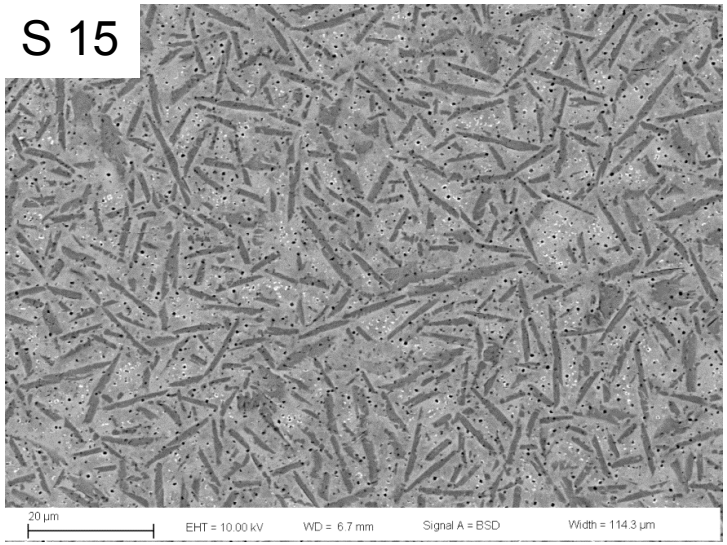
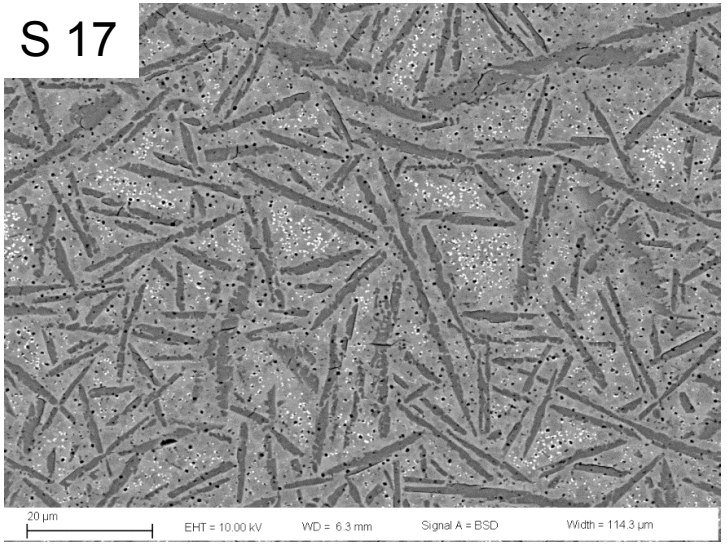
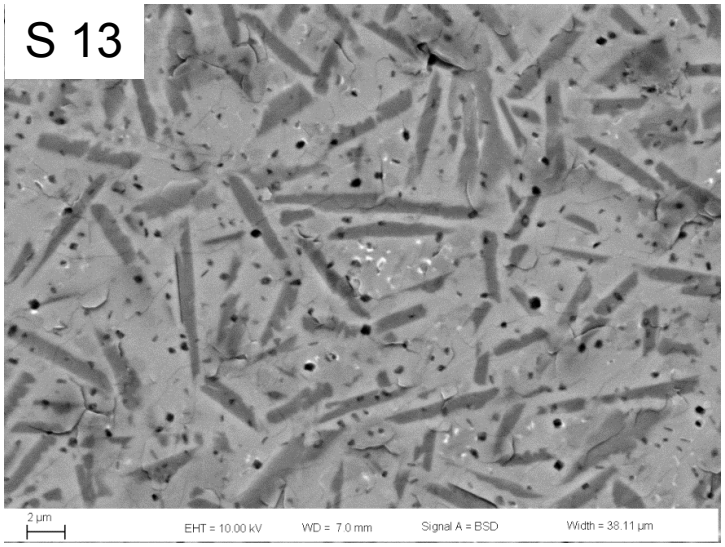


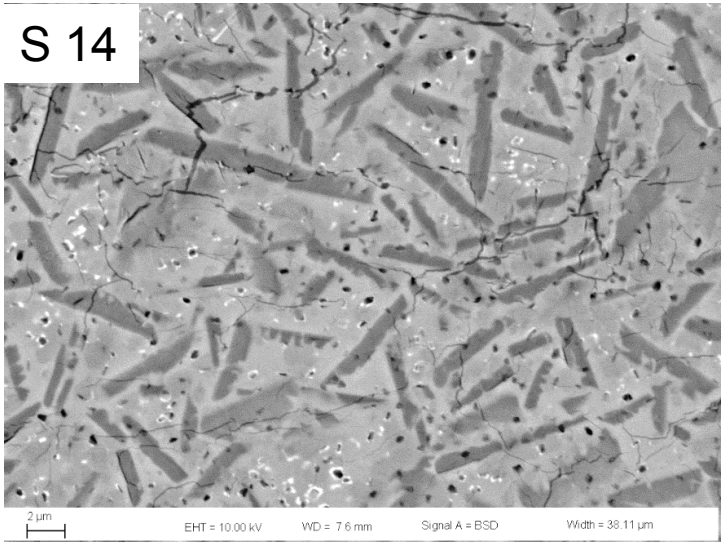


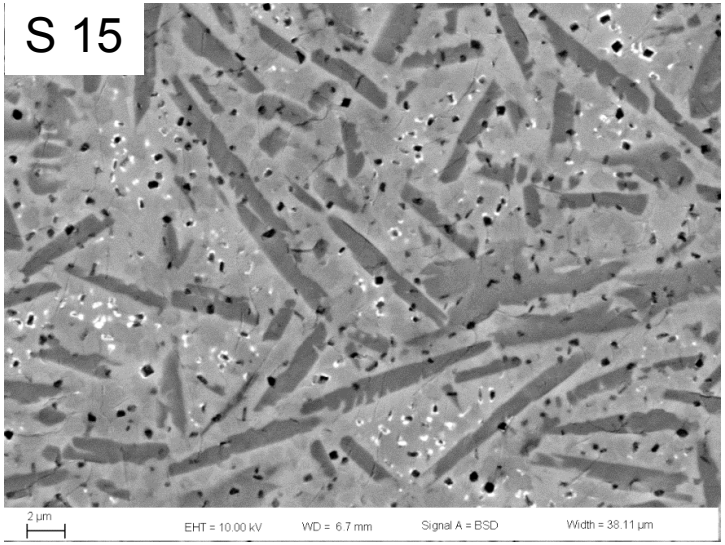


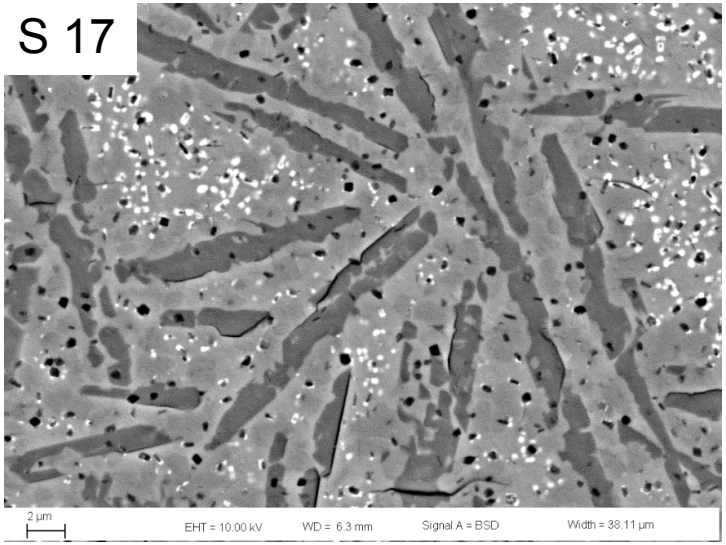


Long Cracks Are Observed Throughout The Glass-Ceramic Microstructure









Glass-Ceramics Are More Crack Tolerant Because Of Their Higher Toughness

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Summary

- **Strength**
  - GC S Is Comparable to Glass
    - Long cracks in microstructure
  - GC SB Is Higher Than Glass
- **Hardness**
  - GC S Is Lower Than Glass
- **Toughness**
  - GC Is 2-3X Glass