Slow Crack Growth of Germanium

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NASA GRC

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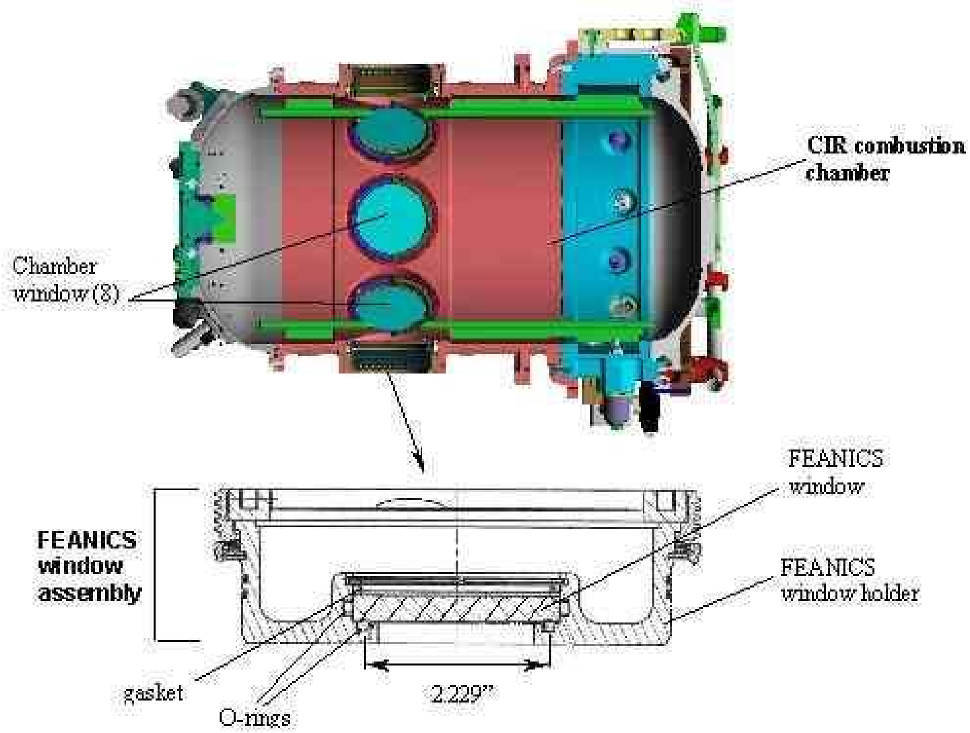
Germanium

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

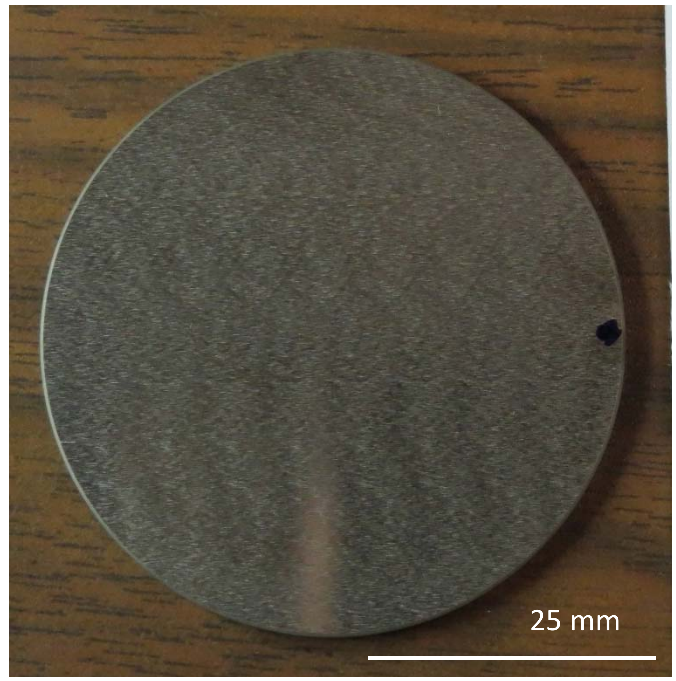


Germanium

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

Material

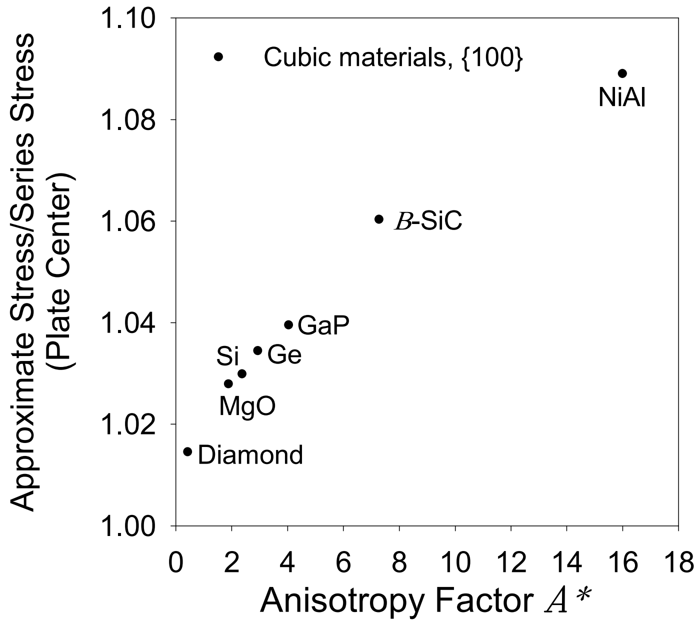
- Single crystal beams
- Coarse gained disks (2” & 5”Φ):

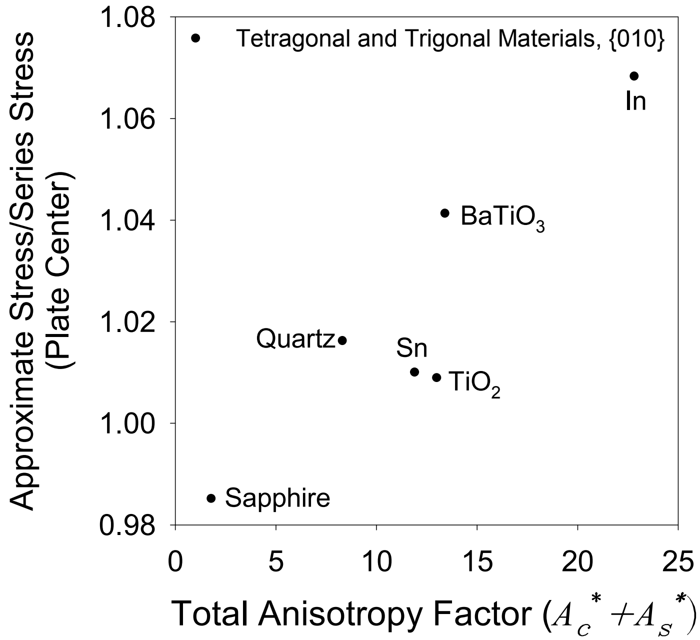


- Variable grain structure – not ideal for testing.

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

Aggregate Constants		
GPa	E	v
Voigt	135	0.20
Hashin	133	0.21
Shtrikman	132	0.21
Reuss	129	0.21

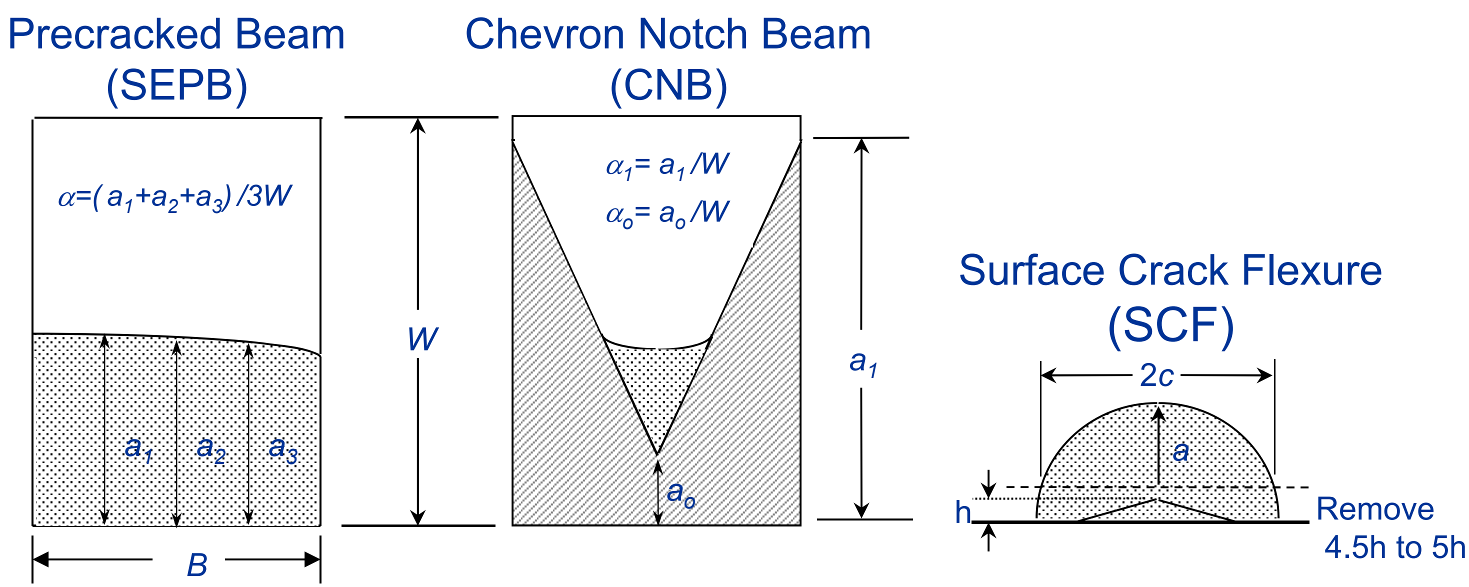
- E_{poly} = 131, v_{poly} = 0.21

Ge	McSkimin	Bogardus	McSkimin	Mason	Average	NASA	% Diff.
Young's Modulus (GPa)							
E<100> =	104. 4	102.0	102.2	103.7	103.1	103.1	0.0%
E<110> =	138.7	136.7	137.0	138.0	137.6	138.3	0.5%
E<111> =	155.8	154.2	154.5	155.1	154.9	154.8	-0.1%

- Well oriented germanium….

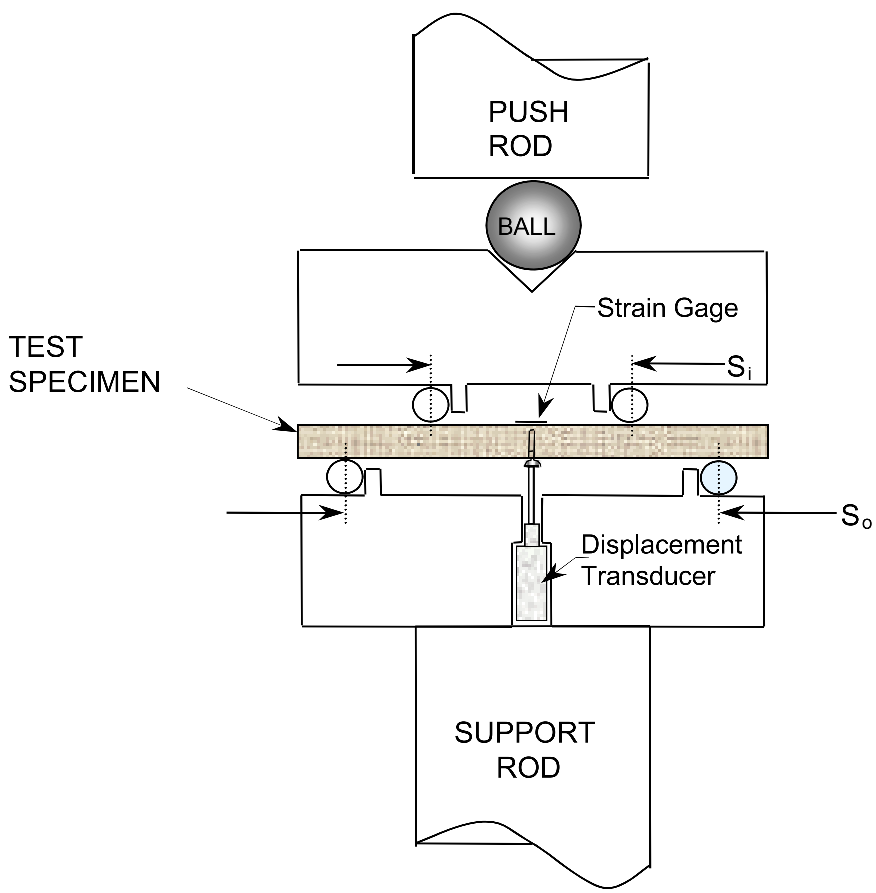
Procedure
- Fracture Toughness -

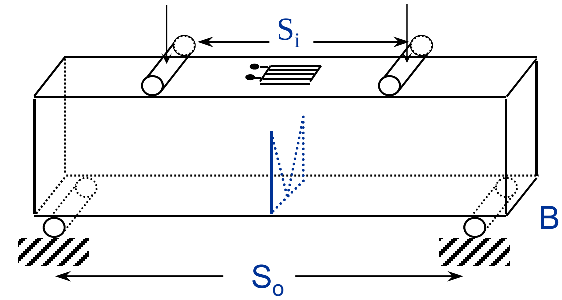
- Three standard test methods (C1421):

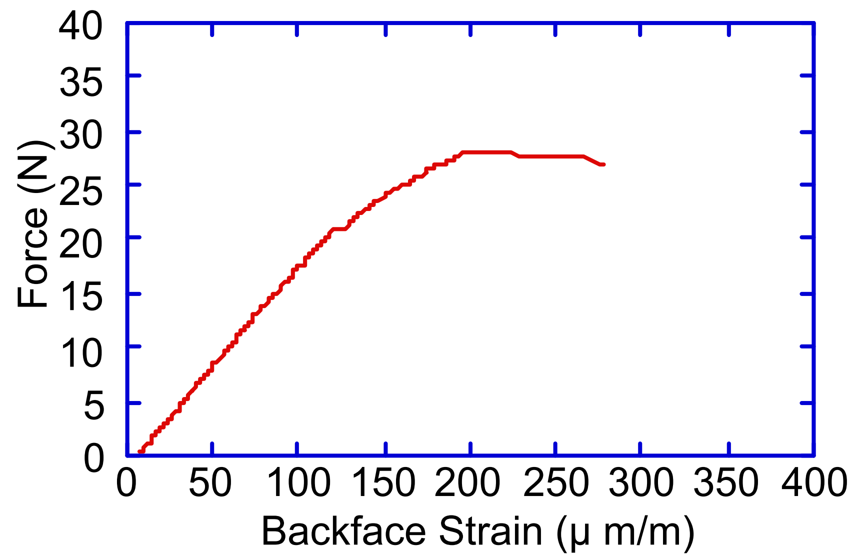


- Different crack size
- Different crack formation history
- Different effort

Loading Configuration
- Fracture Toughness -







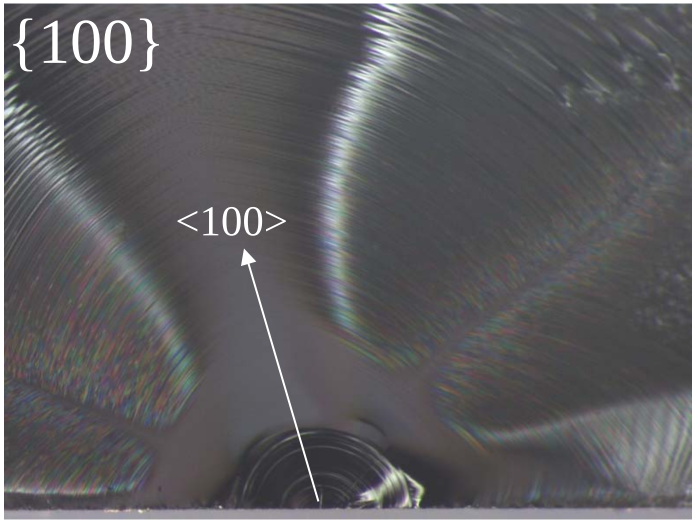
- Relatively simple fixtures: test frame, load cell, recording device.

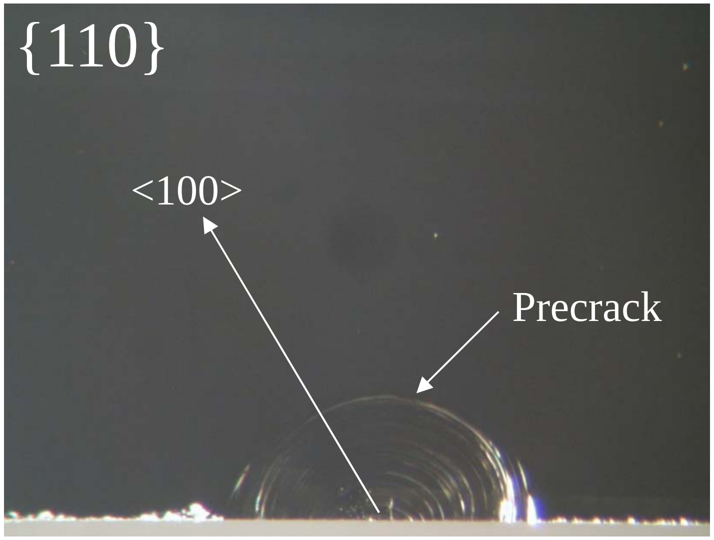
Fracture Toughness

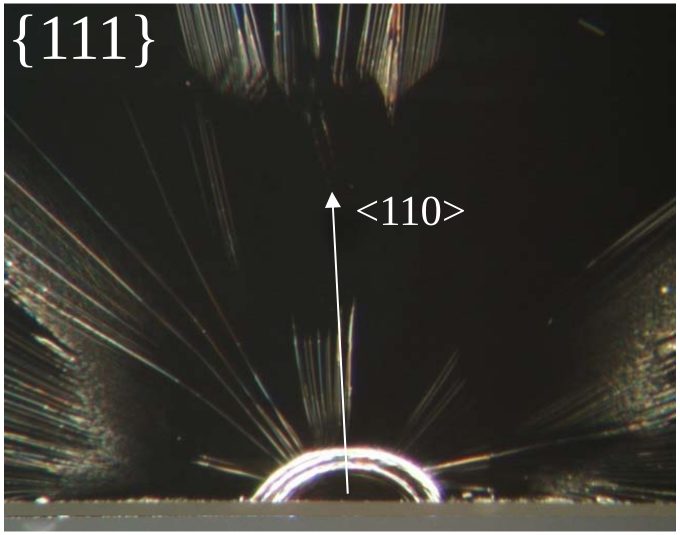
Method	{100}	{110}	{111}
SEPB	0.68 ± 0.04	0.68 ± 0.01	< 0.74
SCF	0.74 ± 0.02	0.74 ± 0.02	0.74 ± 0.02
CNB	In progress	In progress	In progress

- Essentially the same on all planes
- $K_{Iscf\{jkl\}}$ = 0.74 ± 0.02 MPa√m
- $K_{Ipb\{100,\ 110\}}$ = 0.68 ± 0.04 MPa√m
- ~10% difference between SCF and SEPB. Plasticity?
- Practical value of $K_{I\{jkl\}}$ = 0.70 ± 0.02 MPa√m.

SCF Precracks

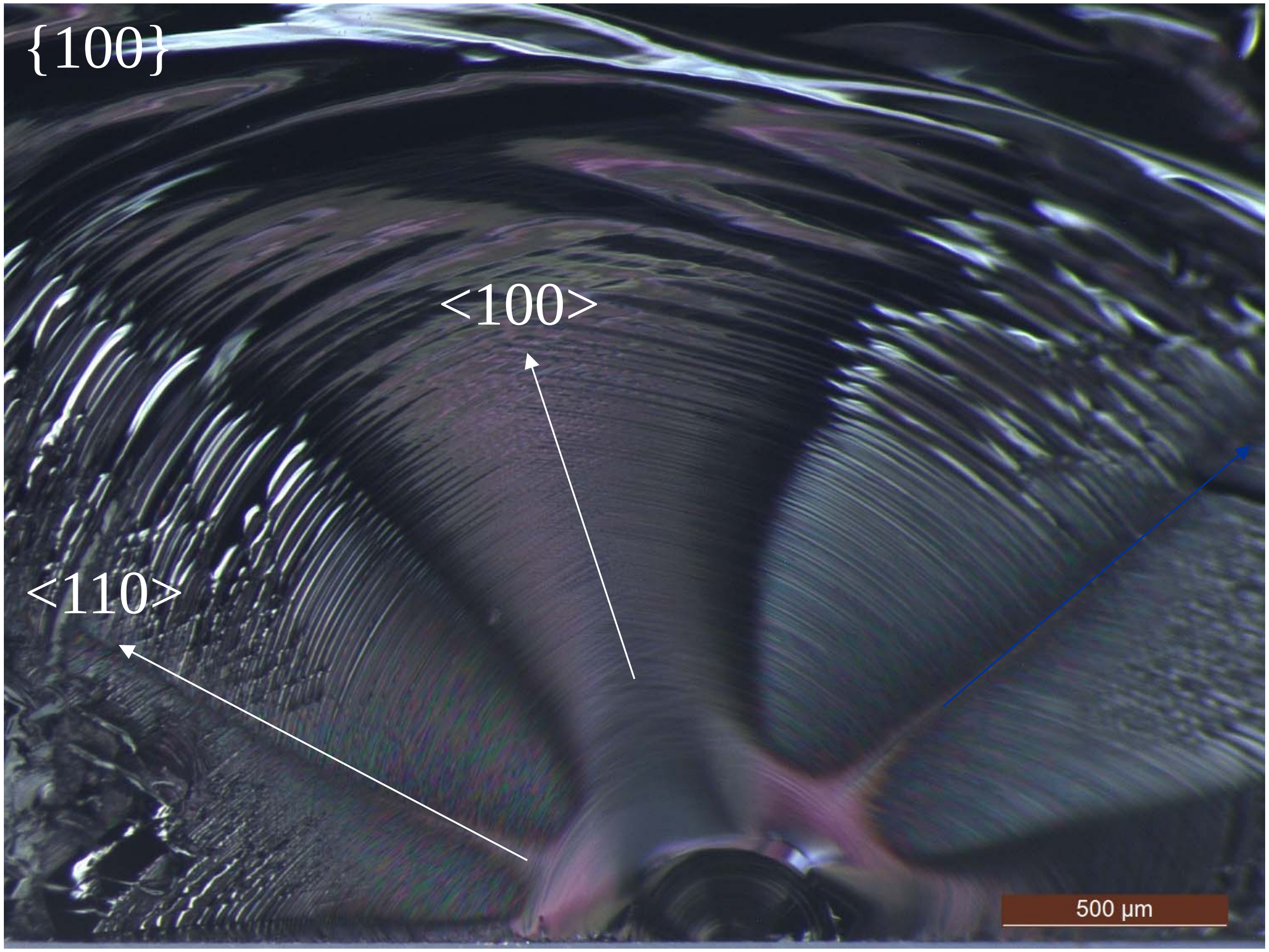






» {100} exhibit cathedral Wallner lines.
» The most planar surface occurs on the {110}.
» {111} tends to exhibit cleavage steps.
» Secondary orientation was not fixed.

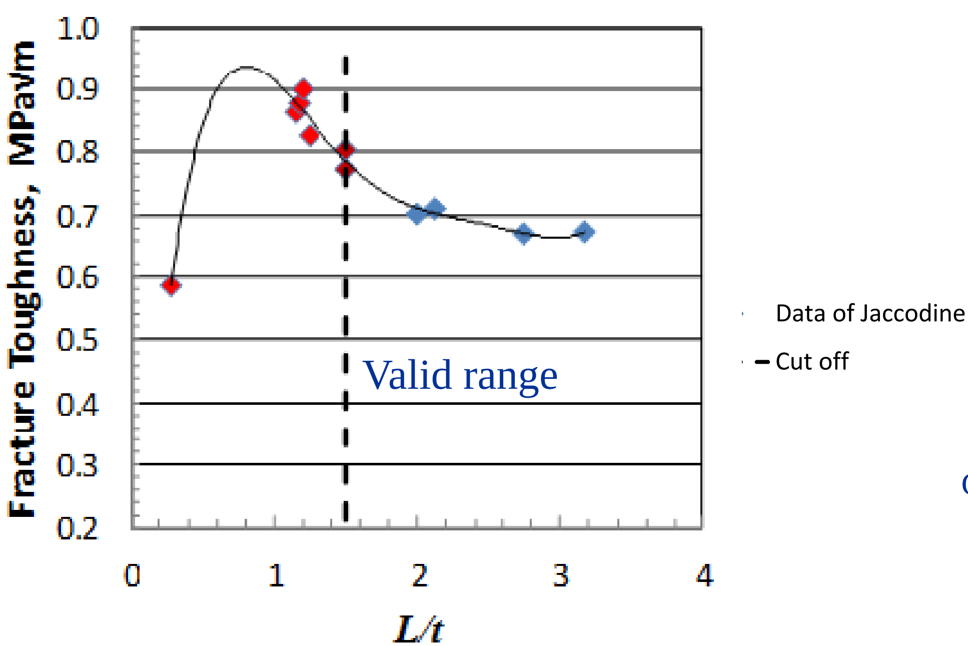
Cathedral Orientation



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

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

- Reported an energy equivalent value of 0.55 MPa√m.
- Used DCB w/ fracture mechanics solution that did not include L/t effects.
- Reanalysis gives $K_{I\{111\}}$ = 0.72 ± 0.05 MPa√m (6) w/ trend toward 0.67 MPa√m:

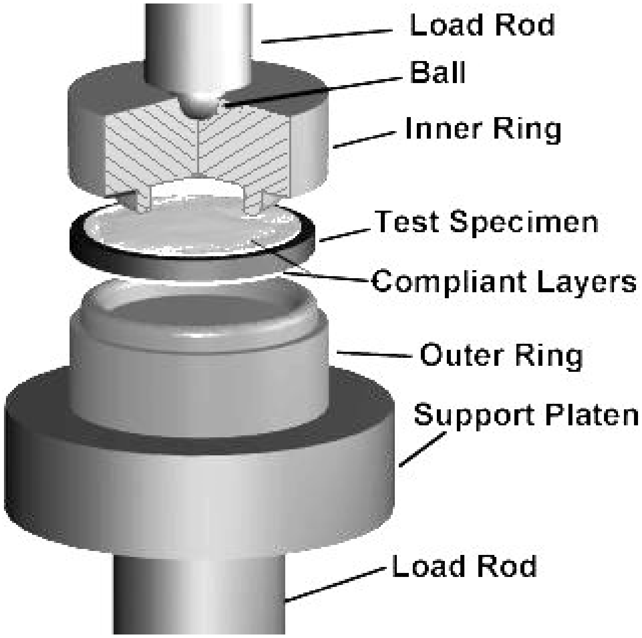


∴ Engineering value ~0.68 MPa√m for low index planes

R.J. Jaccodine, "Surface Energy of Germanium and Silicon," *J. Electrochemical Soc.*, Vol. 110, No. 6, June, 1963, pp. 524-527.

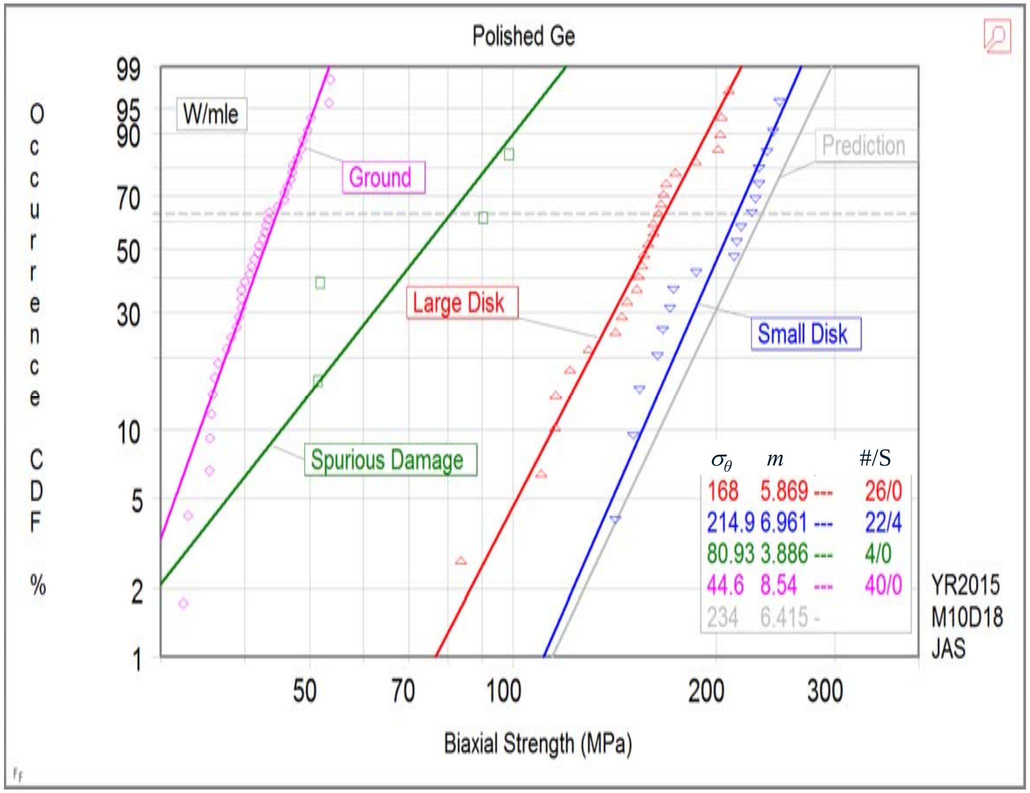
Strength Testing

- 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

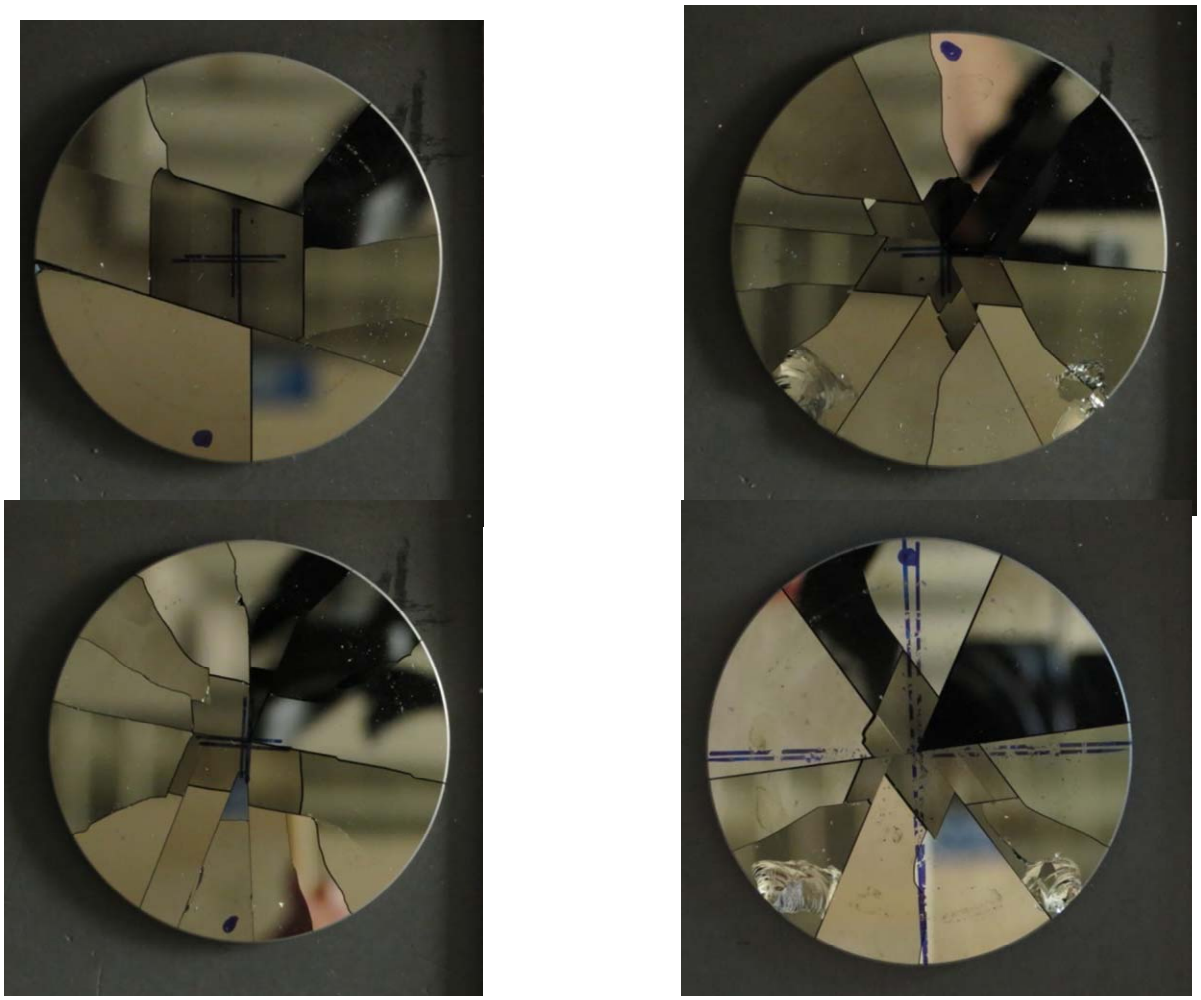
Fracture Strength & Weibull Statistics



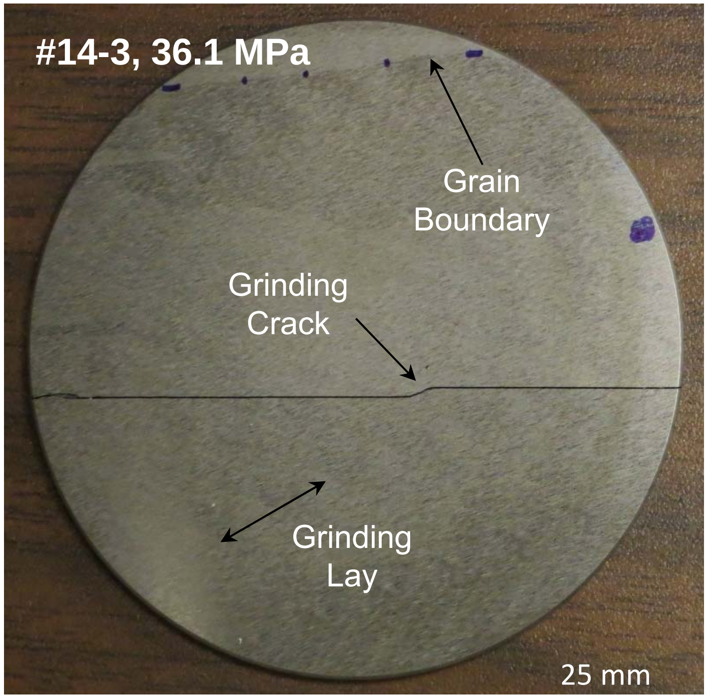
- Polished m = 6; ground m = 9; spurious damage m = 4.
- Scale effect evident: 168 vs 215 MPa.
- Strength of 235 MPa is predicted vs 215 MPa (10%).

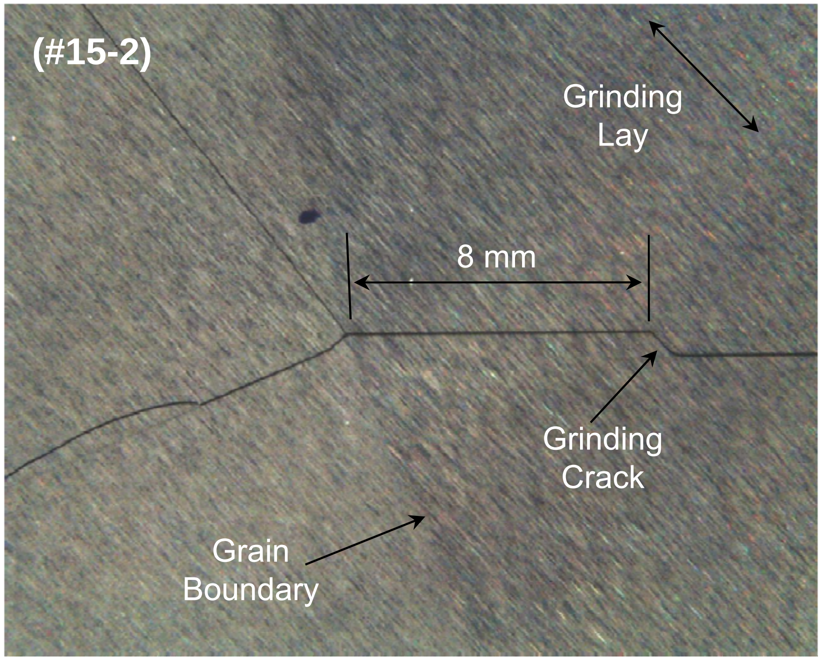
Biaxial Fracture Patterns (polished)

- Repetitive pattern that makes fractography difficult:



Fracture Path
- ground disk -

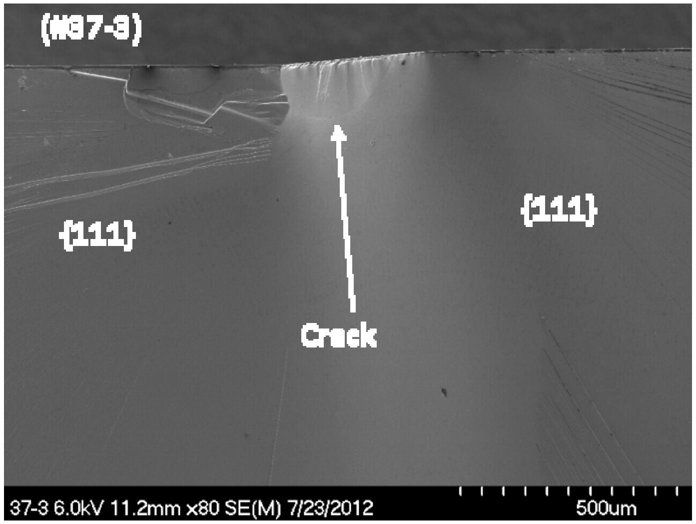


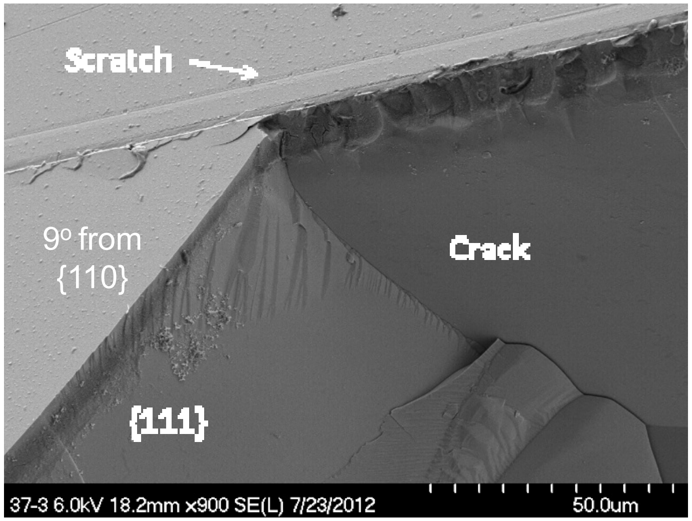


- Crack initiated at a grinding scratch.
- Transited to a low index planes.
- Deflected at a grain boundary.

Fracture Path in a Polished ROR Disk

- Crack initiated from a semi-elliptical crack emanating from a scratch.
- Turned onto the {111} plane:

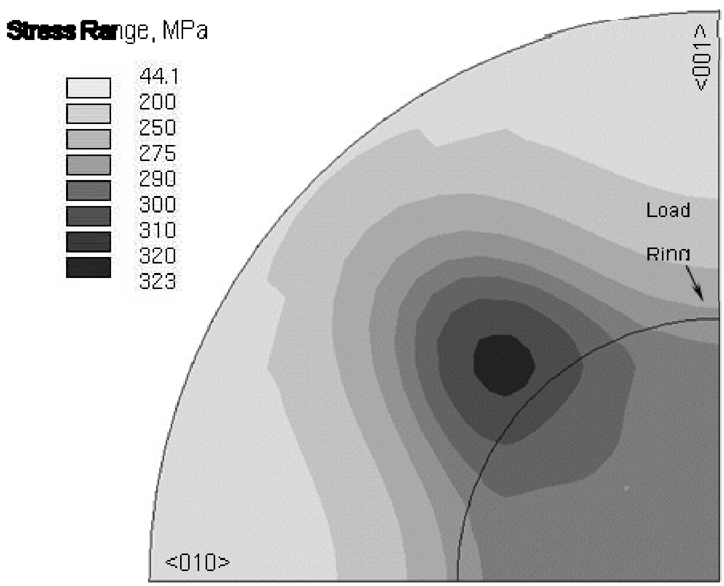




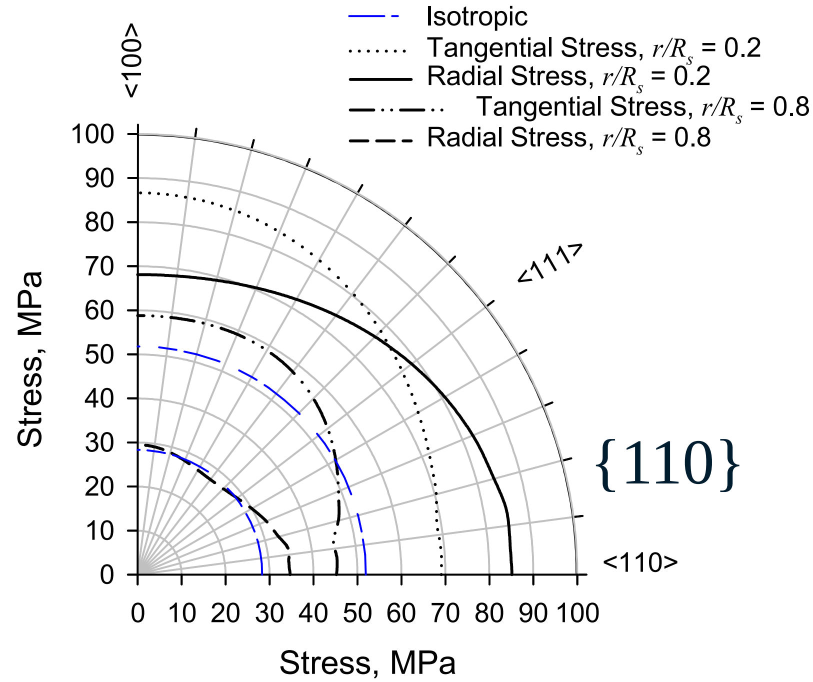
- Opportunity to estimate the fracture toughness!
- $K_{I\{hkl\}}$ = 0.73 MPa√m.
- Why did the crack turn?

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……

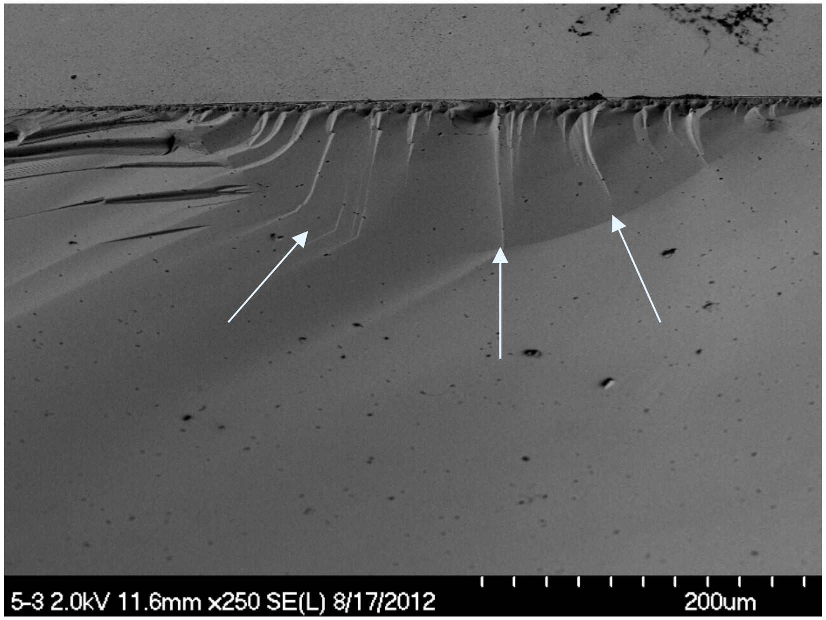


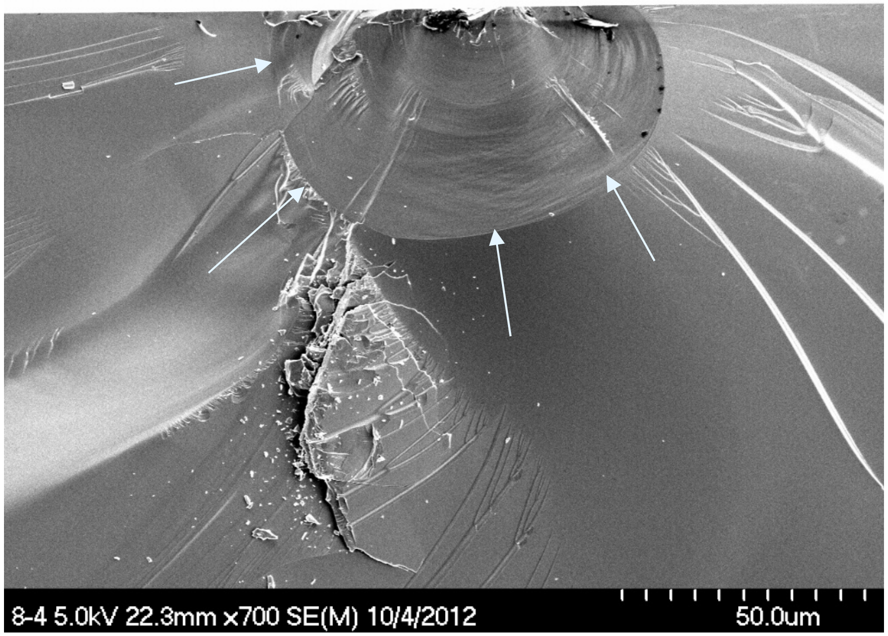
Stress concentration where the load ring intersection the stiff direction.



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 ± 0.04 MPa√m (0.73-0.83).
- For grinding cracks, K_I = 0.87 ± 0.04 MPa√m (0.80 – 0.90).
- Higher due to random orientation and transition to {111}.
- Caveat: local stress not precisely know…..

Slow Crack Growth

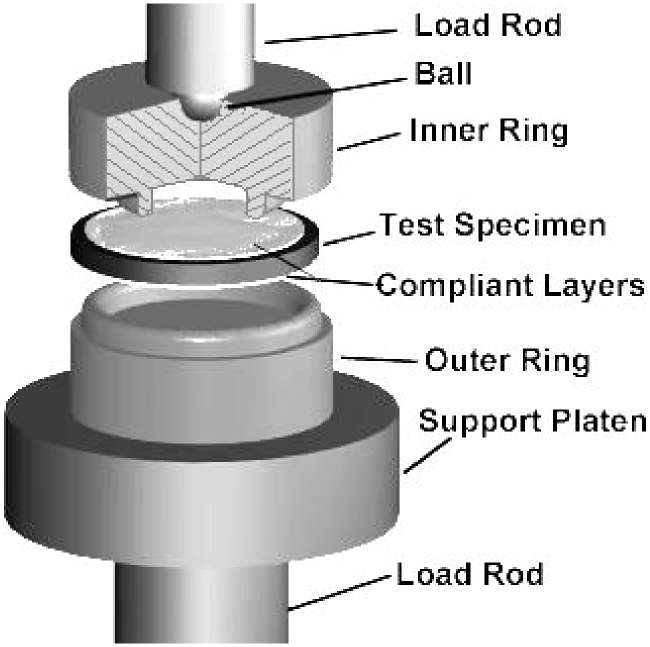
- Experimental Approach -

- 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:

$$v = \frac{da}{dt} = AK_I^n = A^* \left[\frac{K_I}{K_{IC}} \right]^n$$

- Constant stress rate testing:

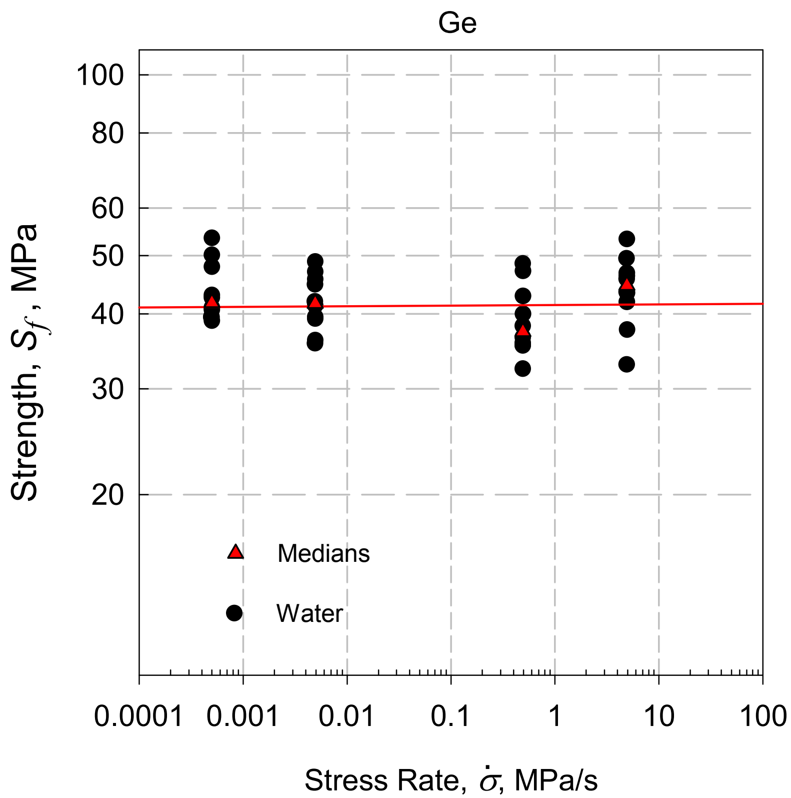
$$\sigma_f = \left[B(n+1)\sigma_i^{n-2}\dot{\sigma} \right]^{1/(n+1)} \qquad B = \frac{2K_{Ic}^{2-n}}{AY^2(n-2)} = \frac{2K_{Ic}^2}{A^* Y^2 (n-2)}$$

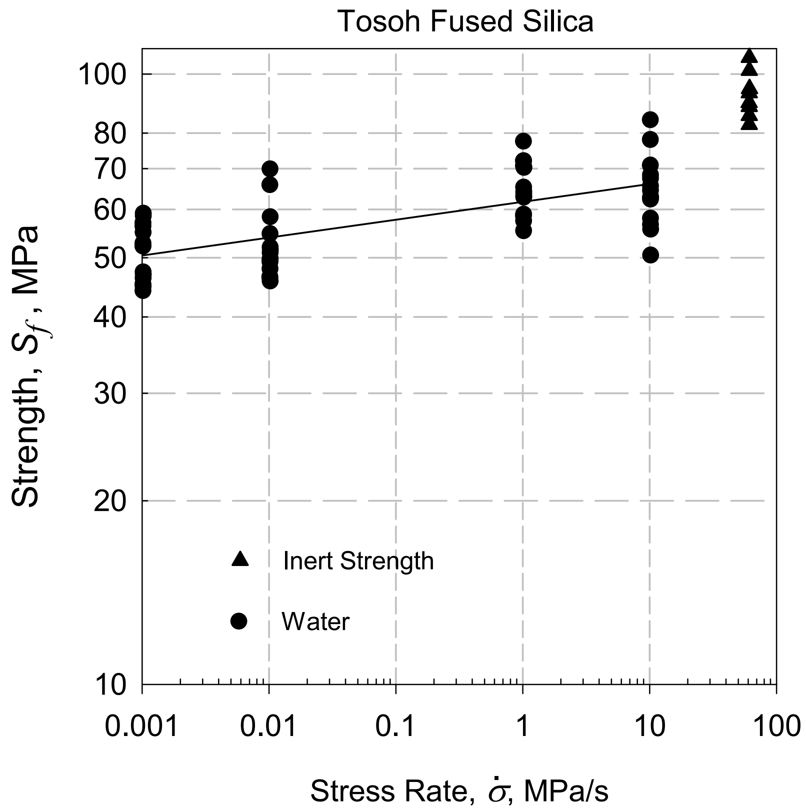
- Parameter extraction via regression:

$$\log_{10} \sigma_f = \frac{1}{n+1} \log_{10} \dot{\sigma} + \log_{10} D \qquad \log_{10} D = \frac{1}{n+1} \log_{10} \left[B(n+1)\sigma_i^{n-2} \right]$$

(Slope α) (Intercept β)

Constant Stress Rate Curve





- 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_I = 0.68 ± 0.02 MPa√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_{poly} = 131 GPa, ν_{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

- Thanks to Terry McCue for SEM fractography.
- Thanks to Rick Rogers for x-ray diffraction.
- Thanks to Penni Dalton for funding & reviews.