Observations in Fracture Toughness Testing of Glasses and Optical Ceramics

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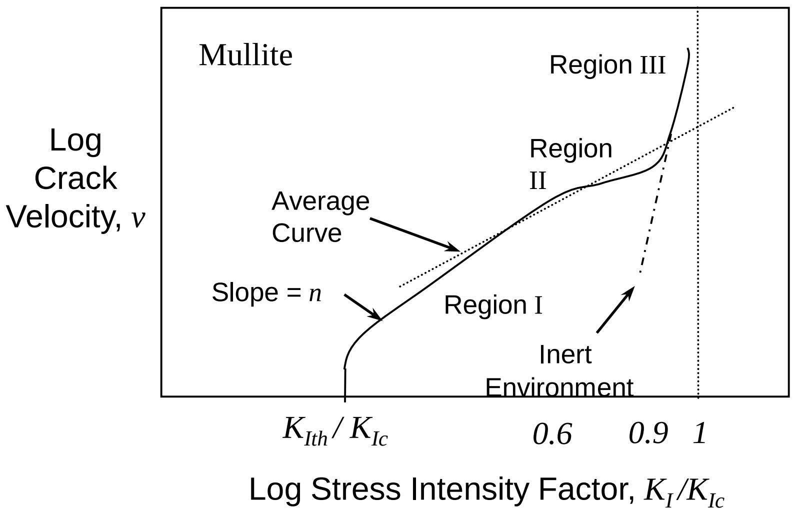
OUTLINE

- 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?

- α SiC:

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

- Greater than 30% difference in literature!!

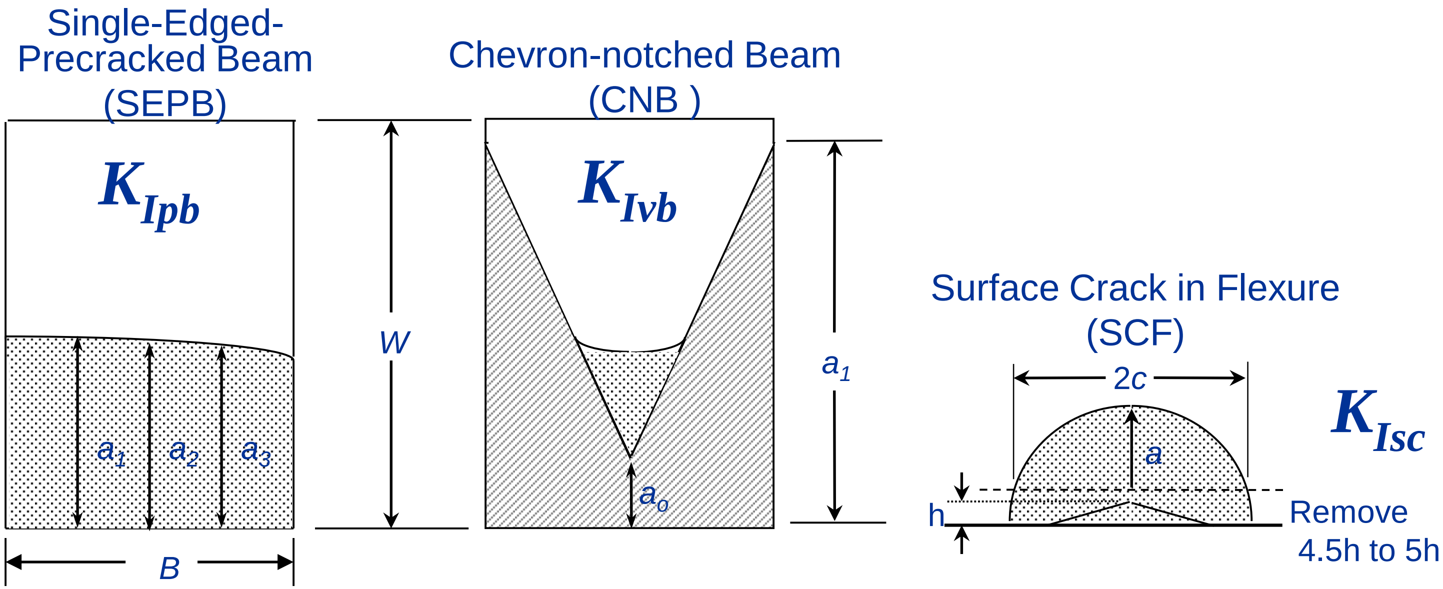
Standard Method C1421

- 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 STANDARDIZED TEST METHODS

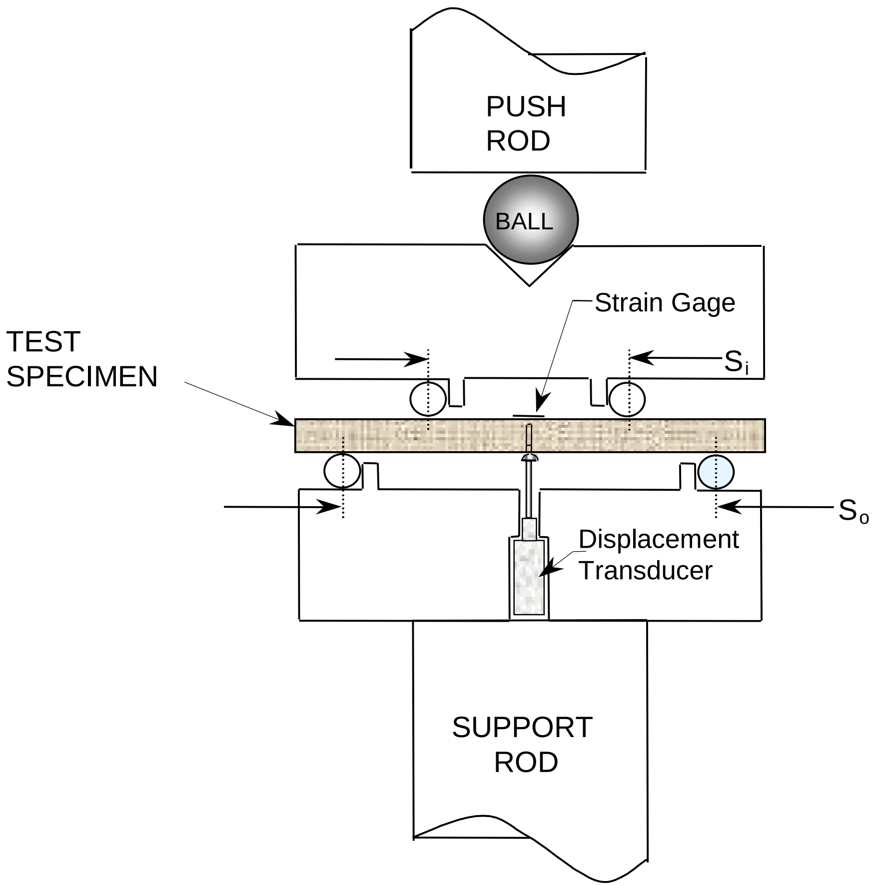
- Flexure test specimens with various cracks:

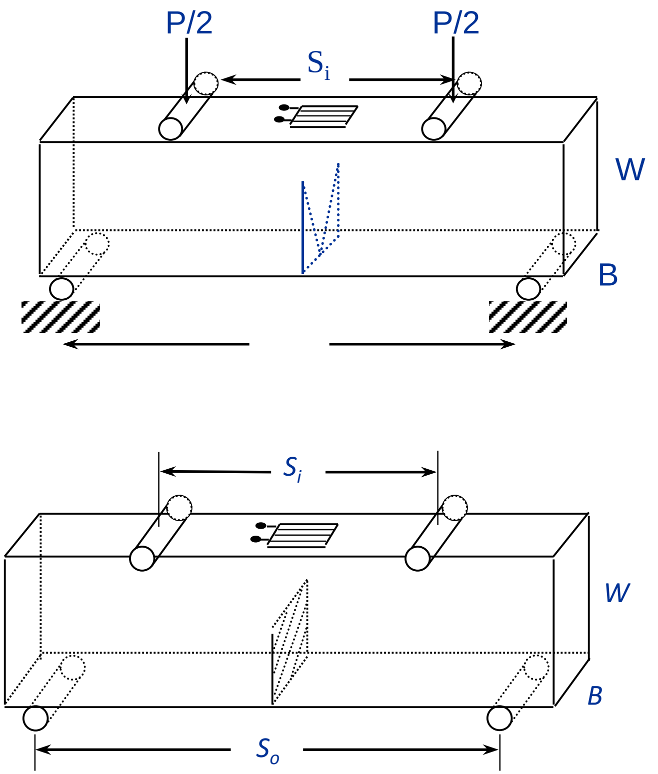


- Different crack size
- Different crack formation history
- Different effort

Similar results for ceramics!

LOADING CONFIGURATION





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

HOW GOOD CAN FRACTURE TOUGHNESS MEASUREMENTS BE WITH C1421?

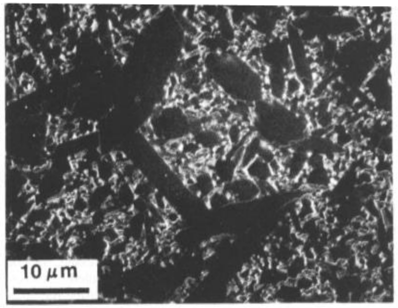
Material	K_{Ivb} MPa√m	K_{Ipb} MPa√m	K_{Isc} MPa√m
α-SiC (JAS)	2.61 ± 0.05	2.58 ± 0.08	2.76 ± 0.08
α-SiC (UW)	2.62 ± 0.06	2.54 ± 0.20	2.69 ± 0.08
ADS96R	3.56 ± 0.03	3.71 ± 0.10	----
ALSIMAG 614	3.19 ± 0.06	3.09 ± 0.17	3.18 ± 0.10
ALSIMAG 614	3.13 ± 0.03	2.98 ± 0.06	----
NC132	4.60 ± 0.13	4.59 ± 0.12	4.55 ± 0.14
NT154	5.18 ± 0.11	5.21 ± 0.02	5.80 ± 0.23
SN260	5.19 ± 0.06	5.13 ± 0.15	----
SiAlON	----	2.45 ± 0.09	2.55 ± 0.05

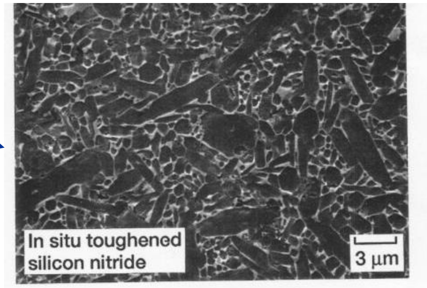
- Three methods are usually within ~3% for “structural” ceramics. 30% => 3%.

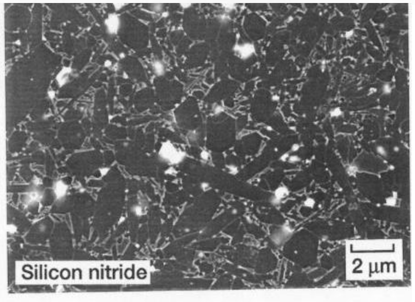
C1421 Captures Grain Size effects on Toughness

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

Increasing grain size



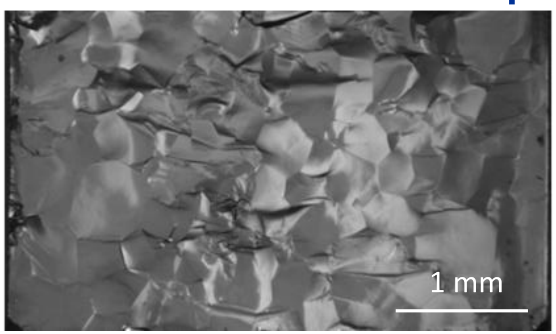


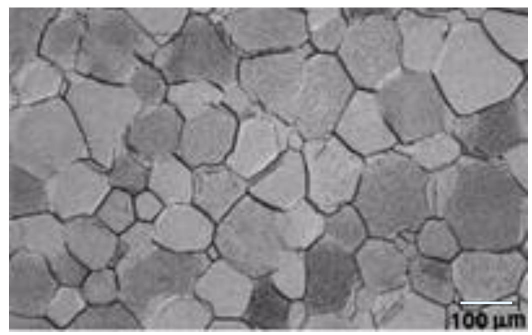


- For ceramics, C1421 provides consistent results with different methods despite different crack geometry.

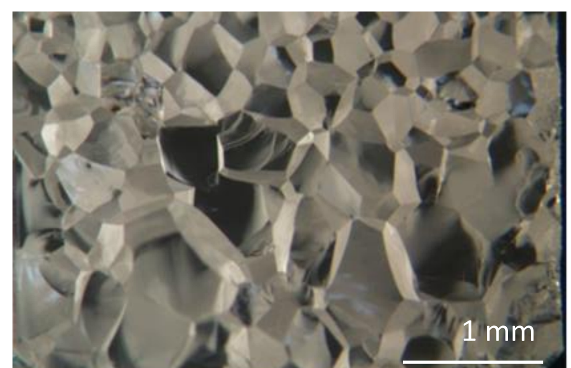
Glass and Optical Materials Tested

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





- Spinel ($MgAlO_4$): GS = 220µm:



Fused Silica

Russian Quartz-Silica

Fracture Toughness (MPa√m)		
Method	Air (75°F, 45% RH)	Dry N_2
SEPB	0.71 ± 0.05	0.73 ± 0.02
VB	0.71 ± 0.04	0.77 ± 0.01

Shuttle Fused Silica (7940)

Fracture Toughness (MPa√m)		
Method	Air (75°F, 45% RH)	Dry N_2
SEPB	0.71 ± 0.04	0.74 ± 0.03
VB	0.73 ± 0.04	0.77 ± 0.02

Literature on 7940 & 7980

Fracture Toughness (MPa√m)				
Method	Vacuum	Vacuum/ Dried	Dry N_2	Source
AMDCB			0.72 ± 0.01	NIST (LB)
DCB	0.74 ± 0.03	0.73 ± 0.02		NIST (SW)
3-Pt	0.75 ± 0.03		0.76	NIST (SW)
DCDC	0.73 to 0.76			Sandia

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

Soda Lime Silicate Glass

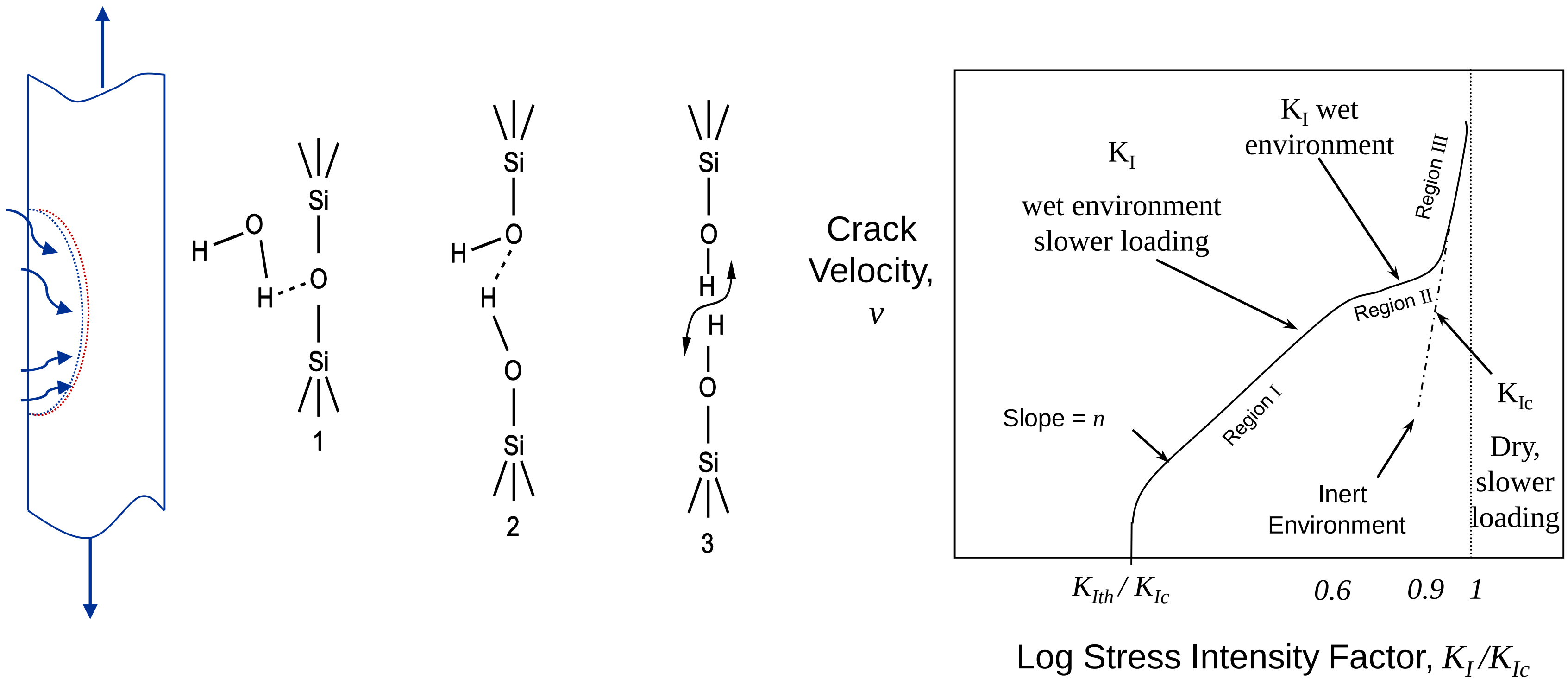
Load Rate (mm/min)	0.005 (54%RH)	0.01 (53%)	0.02 (40%)
K_{Ivb} (MPa√m)	0.65 ± 0.02	0.67 ± 0.03	0.74 ± 0.03

Load Rate (mm/min)	0.005 (LP N_2)	0.02 (LP N_2)	0.005 (HP N_2)	0.02 (HP N_2)
K_{Ivb} (MPa√m)	0.76 ± 0.01	0.75 ± 0.01	0.76 ± 0.01	0.79 ± 0.02

- 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?

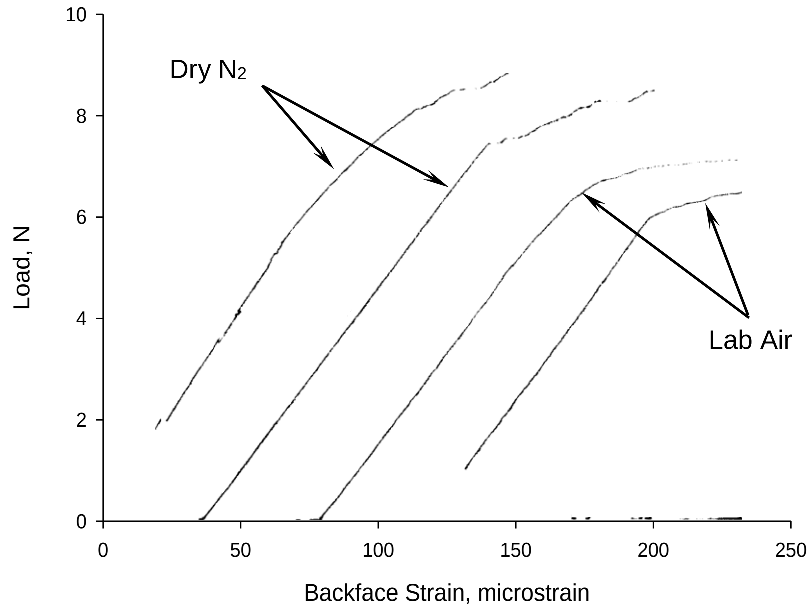
- In humid environments, fracture toughness measurements are points on the slow crack growth curve:

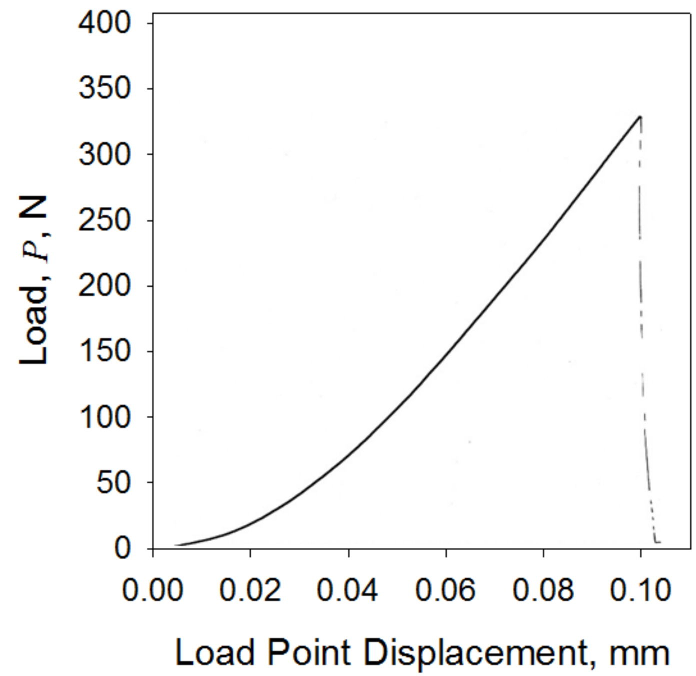


- The unique value, K_{Ic}, corresponds to a dry environment.

Complication: Unstable Fracture

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

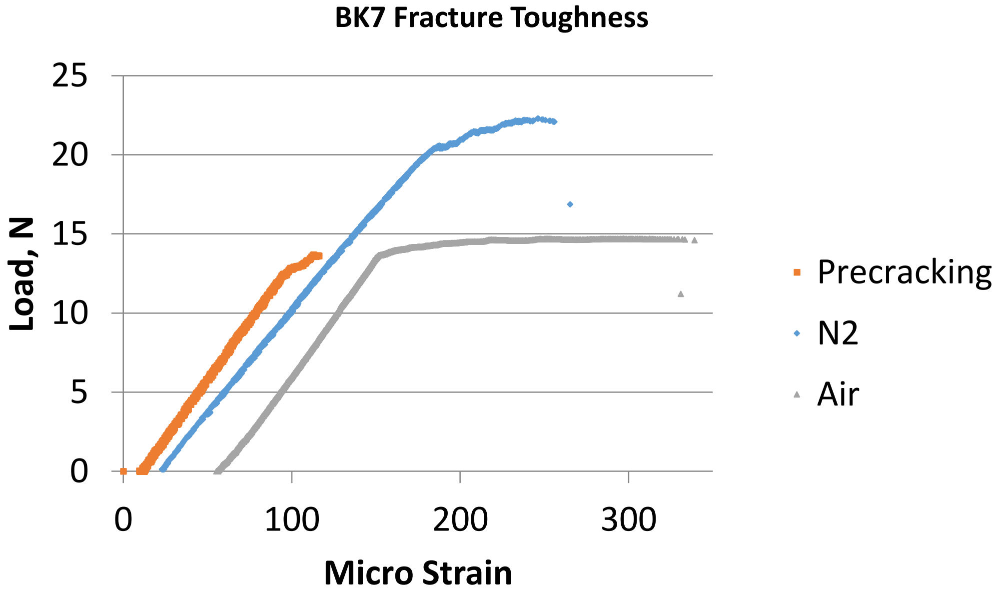


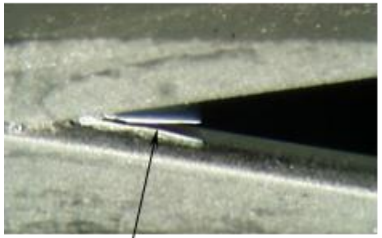


- Required for the chevron-notch. Less of an issue for SEPB and SCF methods.

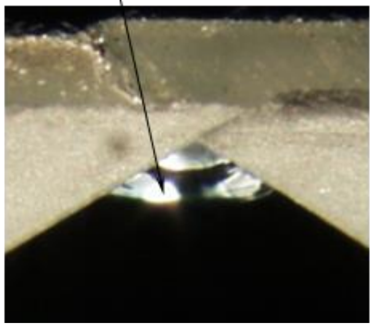
Solutions: precracking & scratching

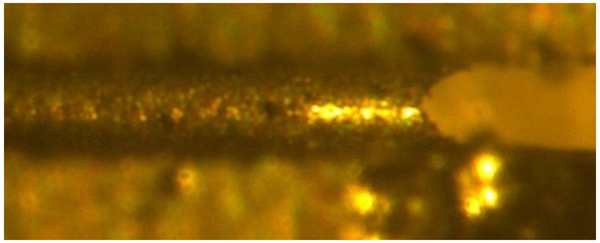
- Precrack in air; fail in N_2:





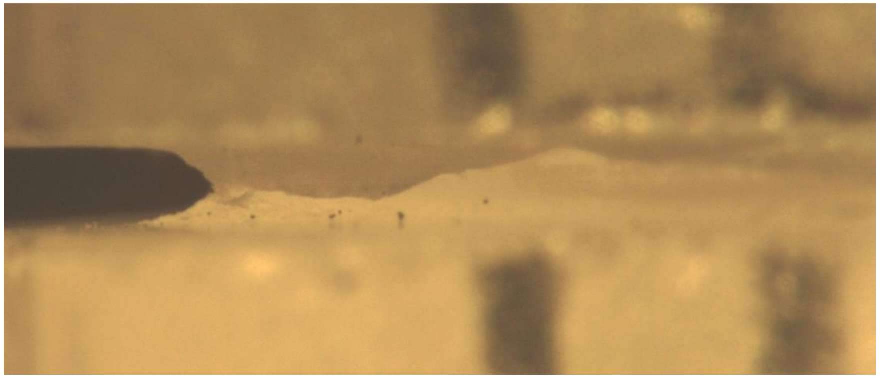
Precrack



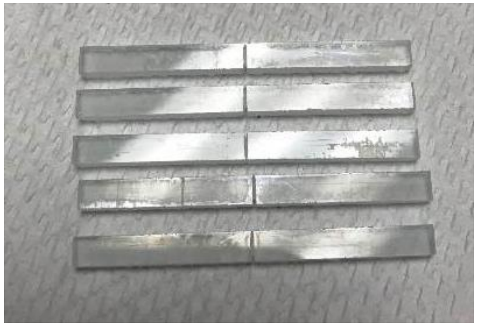


Complication: Cracked Specimens

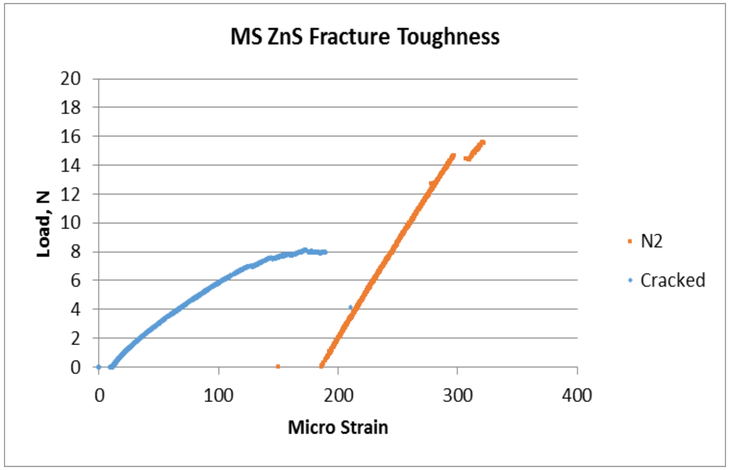
- Some materials are very fragile and can be cracked:



- Bonding agents need to be removed:

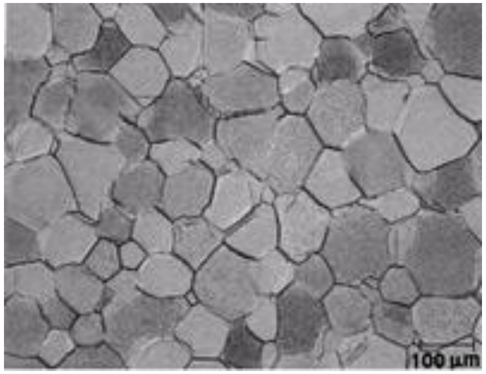


- Results in low compliance:



ALON

$K_{Ivb}(A)$ (MPa√m)		K_{Ipb}	K_{Isc}
~40% RH	N_2	~50% RH	N_2
2.09 ± 0.06	2.18 ± 0.10	2.02 ± 0.04	2.36 ± 0.1



Stress Rate (MPa/s)	0.002 (H_2O)	2 (H_2O)	20 (H_2O)	20 (N_2)
K_{Isc} (MPa√m)	1.80 ± 0.07	1.92 ± 0.06	2.07 ± 0.06	2.36 ± 0.1

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

Spinel

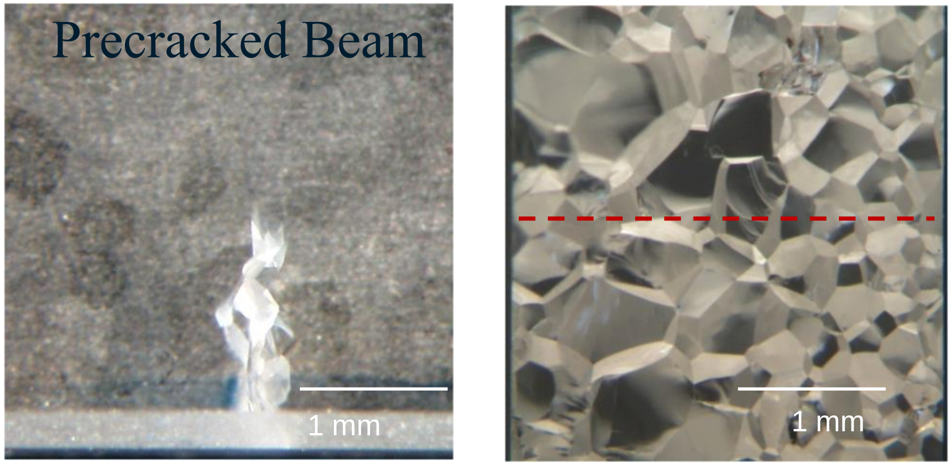
45%–65% RH		N_2 (MPa√m)	
K_{Ipb}	K_{Ivb}	K_{Ipb}	K_{Ivb}
1.32 ± 0.05	1.48 ± 0.14	1.52 ± 0.07	1.58 ± 0.08

Δ = 11% for air; Δ = 4% for N_2

- Similar results for two of the standard methods.
- Numbers converge for nitrogen!

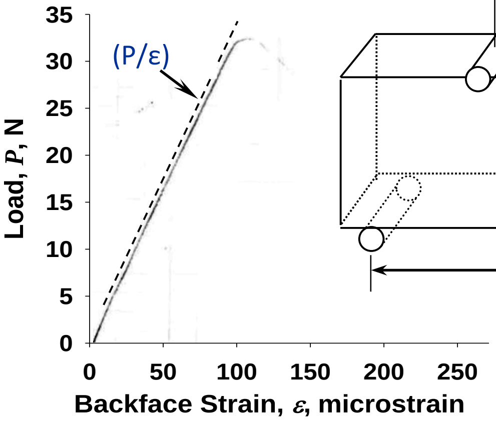
Complication: Crack Length Measurement

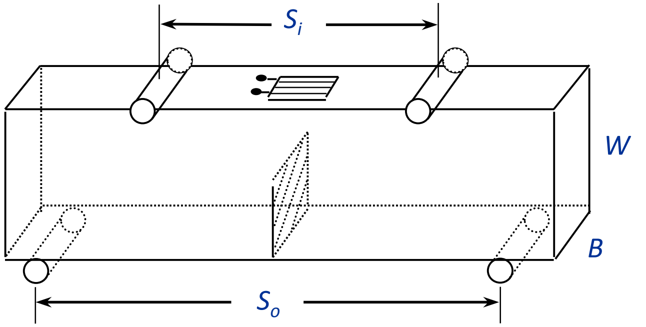
- Crack front apparent…until the specimen is broken:



- Solution is a strain gage……
  a/W α (P/ε).

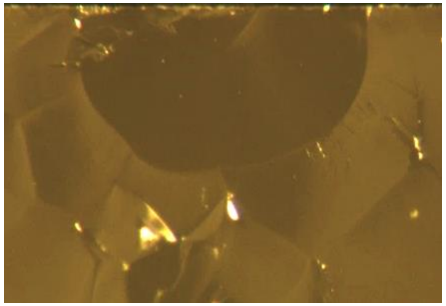
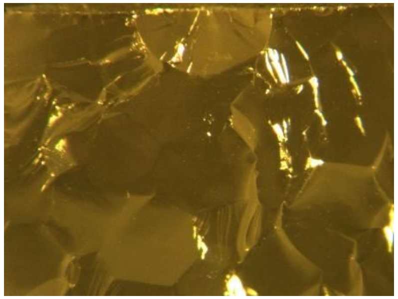
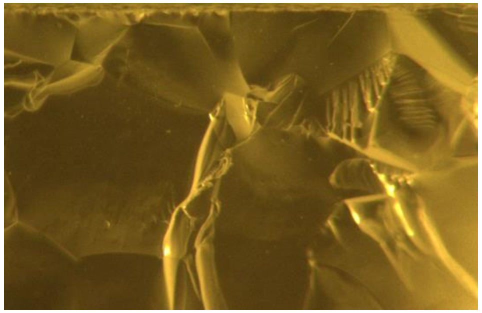
- Or use the chevron, which does not need crack length.



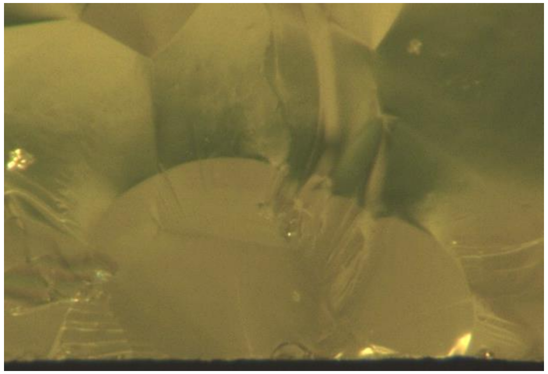
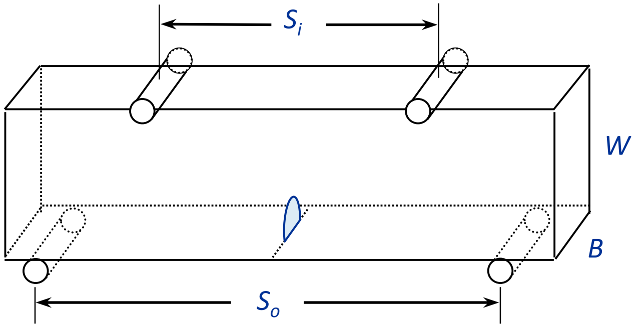


Complication: Crack Length Measurement

- Crack front apparent not always apparent in SCF specimens:

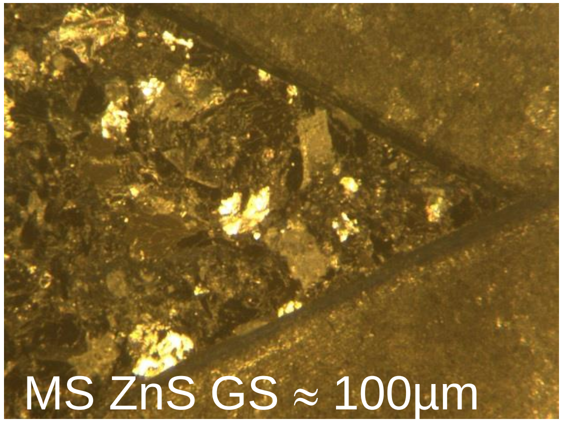




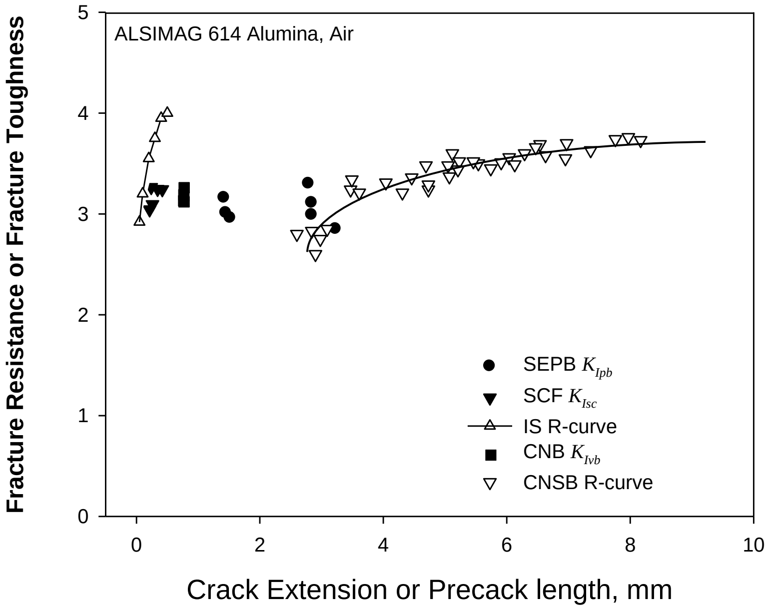
- Less than ideal shapes due to grain structure:

Complication: Crack Growth Resistance

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

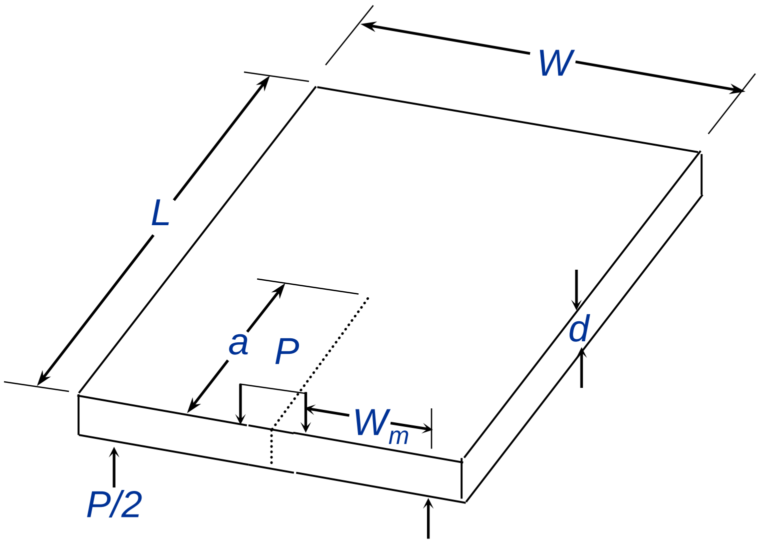


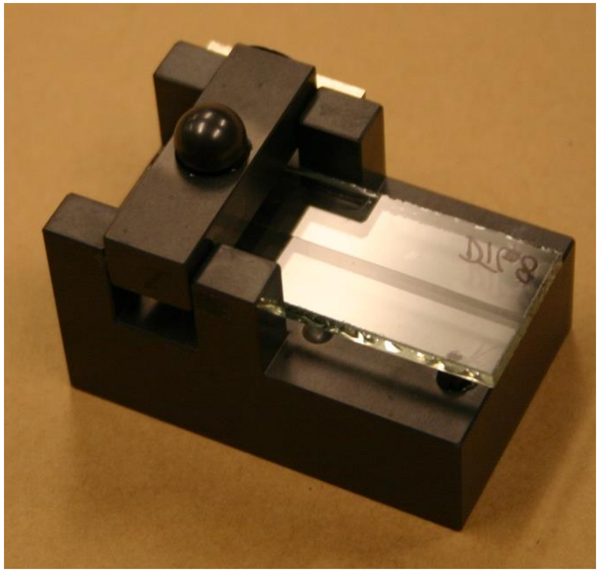


- ASTM C1421 techniques provide relative low values on the R-curve for an alumina.

Complication: Thin Materials

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

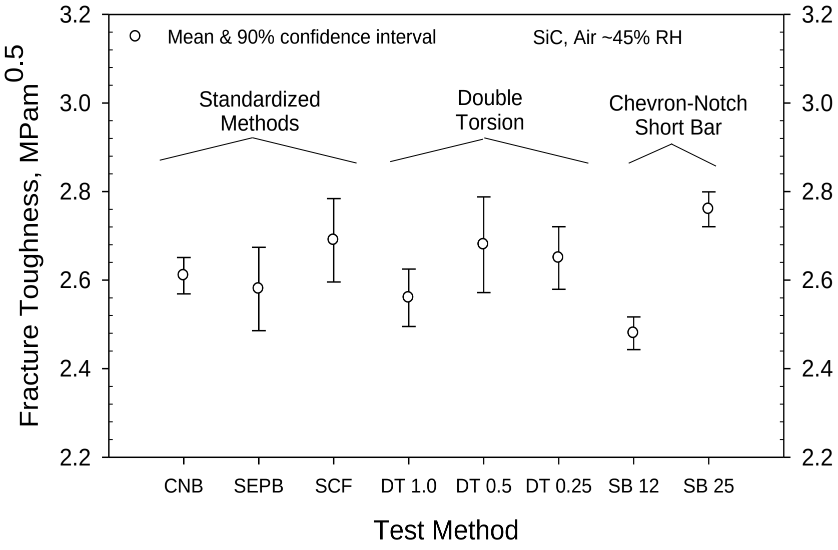


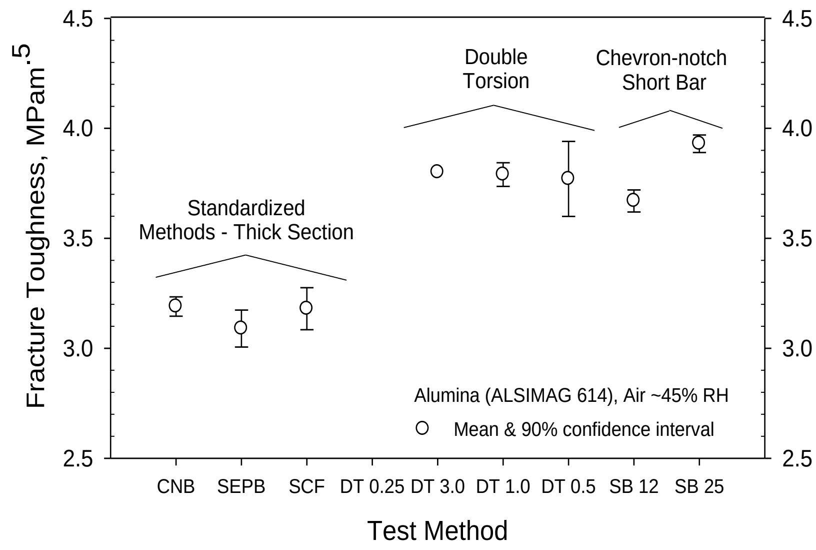


- Comparable results for some materials…………

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

Fracture toughness (MPa√m) of glasses and optical materials via C1421

	Environment		Δ
Glasses	**Air** (%RH/°F)	**Dry N_2**	**%**
MOMA	0.57 ± 0.03 (30/74)	0.69 ± 0.01	-17%
Corning 0120	0.50 ± 0.02 (34/76)	0.67 ± 0.02	-25%
Electro-Glass 2164	0.61 ± 0.05 (32/73)	0.74 ± 0.03	-18%
Schott S8061	0.64 ± 0.01 (23/73)	0.72 ± 0.02	-11%
Schott 8330	0.61 ± 0.04 (60/73)	0.72 ± 0.04	-15%
Soda lime silicate	0.75 ± 0.04 (35/73)	0.80 ± 0.01	-6%
Ba-doped	0.72 ± 0.002 (23/73)	0.76 ± 0.01	-5%
Corning Silica, 7980	0.73 ± 0.04 (45/75)	0.77 ± 0.02	-5%
BK-7	0.87 ± 0.02 (24/73)	0.98 ± 0.04	-11%
Glass Ceramics			
Zerodur	0.89 ± 0.01 (45/73)	0.94 ± 0.01	-5%
Schott S8070	1.57 ± 0.03 (60/73)	1.90 ± 0.03	-17%
Crystalline Optical Ceramics			
ALON	2.09 ± 0.06 (40/70)	2.18 ± 0.10	-4%
Ge	0.67 ± 0.03 (65/74)	No SCG	0%
Sapphire {a}<m>	2.06±0.21	2.31±0.11	-11%
Sapphire {r}<a>	1.96	2.47±0.15	-21%
Sapphire {m}<>	---	---	
Spinel	1.48 ± 0.14 (60/76)	1.58 ± 0.10	-6%
YVO_4 {a}<a>	0.48 ± 0.02 (45/73)	---	
YVO_4 {a}<c>	0.42 ± 0.03 (45/73)	---	
ZnS	0.74 ± 0.03 (60/72)	0.82 ± 0.02	-10%
MS ZnS	0.69 ± 0.03 (58/74)	0.74 ± 0.14	-7%

Glasses < 1

Glass Ceramics ≈ 1 - 2

Crystalline Ceramics σ 0.4 – 2.5

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)

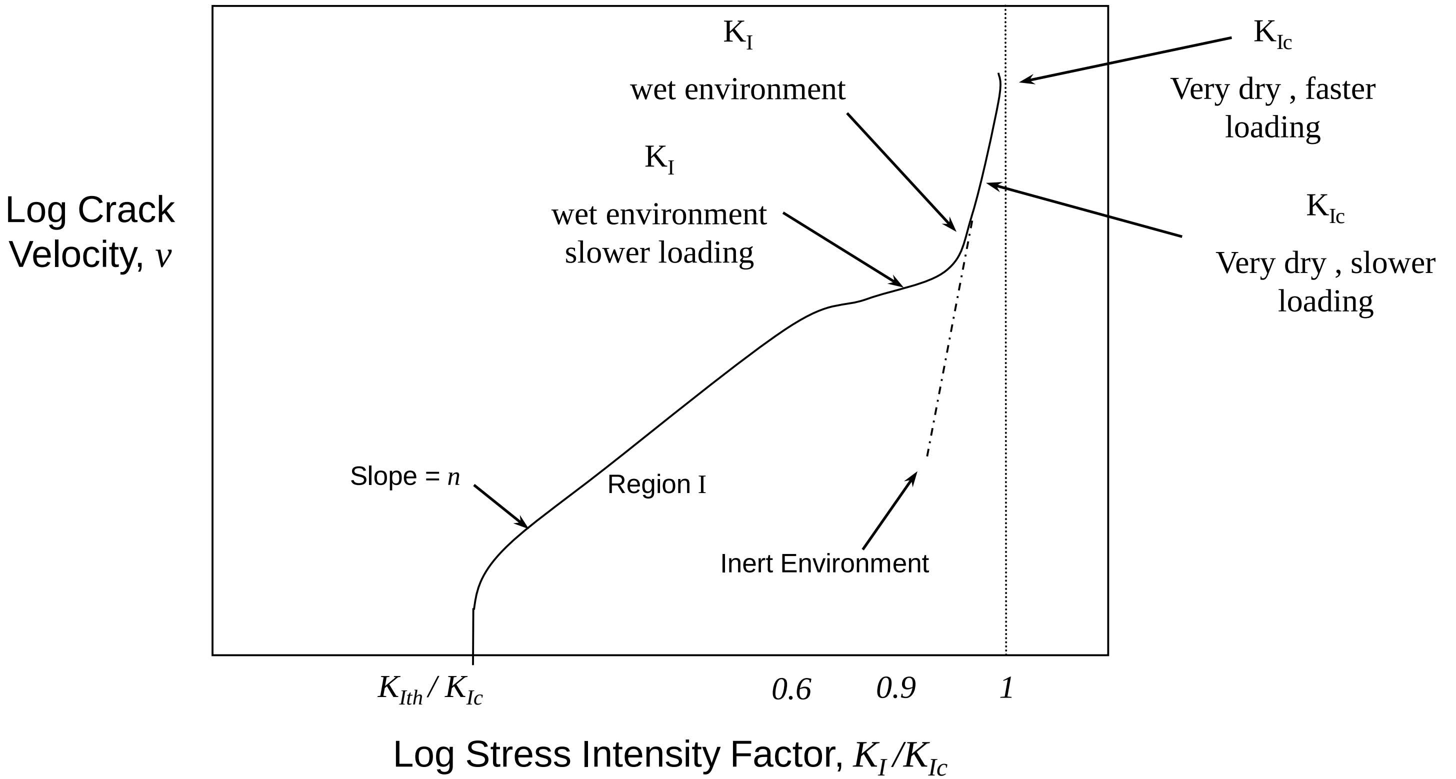
- Common results:

- 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:



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