Effects of Texture and Silica Content on Crack Growth in Boron Nitrides

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Outline

- Background
- Materials and properties of interest
- Past work
  - chemistry, microstructure, texture, moisture absorption
  - strength, modulus, CTE, conductivity
- Slow crack growth
  - procedure
  - failure sources
- Fracture toughness
- Summary

Background

- Crucibles for melting and casting glasses and metals, insulators for furnaces, molds for hot pressing, and neutron absorbers are subjected to harsh conditions:
  - High temperatures
  - Thermal shock and gradients
  - Structural loads
- Historically, material selection has primarily been driven by cost and capability.

➢ ***Hot pressed hexagonal boron nitride***

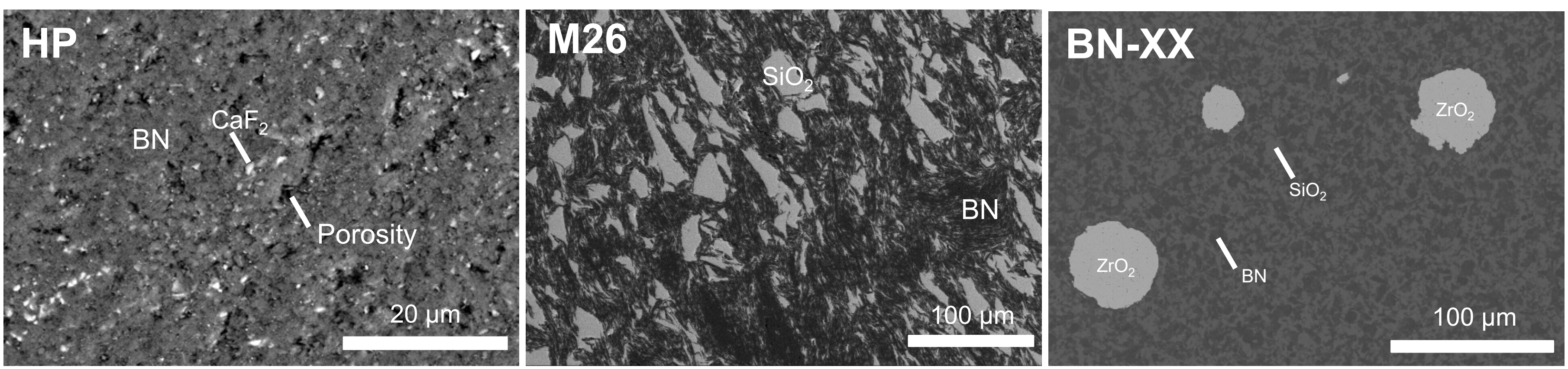
Grades & Microstructure of BN’s

- Similar costs w/exception of Tokuyama Hi-M
- Very different microstructure.
- Texture is not apparent in microstructure via SEM.
- Porosity is apparent in HP grade, less in other grades.
- Large silica content is a concern for crack growth.

Powder XRD Rietveld Refinement (wt%)

Grade	BN	CaF_2	ZrO_2	AlN	Amorp.	Rel. Cost
HP	98	2	0	0	0	1.0
M26	68	0	0	0	32	1.1
BN-XX	56	0	1	0	43	1.0
M	41	0	0	0	59	1.0
Hi-M	27	0	0	72	0	4.7

*Amorphous content is likely SiO_2, confirmed with EDS.

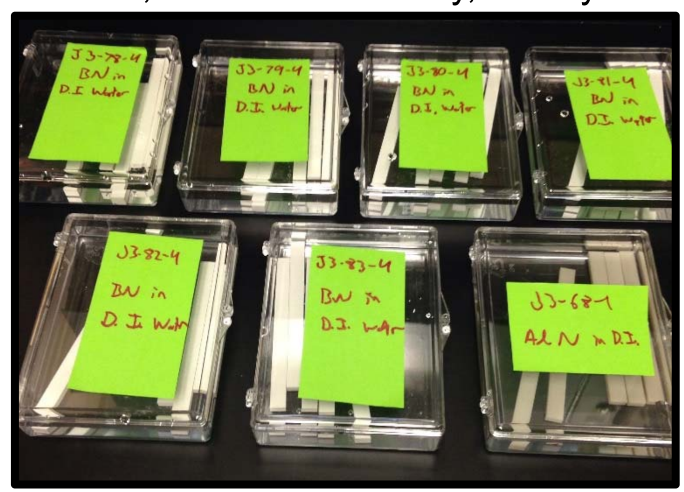


Past Work on Moisture Absorption

- Samples were subjected to one of three moisture levels for >20 days while tracking mass change:
  - 100C, w/<5% rel. humidity.
  - 50C, w/90% rel. humidity.
  - 25C, 100% rel. humidity.
  - Investigated for two hot pressed orientations w/ high aspect ratio samples.

Drying Oven
100C, <5% rel. humidity, 50 days

Submerged in Water
25C, 100% rel. humidity, 90 days



Moisture Absorption (cont.)

- Mass change tracks with open pore porosity (high, medium, low).
- HP hot press orientation has influence on the transfer of moisture (high, low).
- Submerged HP samples produced $CaB_6O_9(OH)_2(H_2O)_3$ surface salt.

Sample	Porosity (%)	Dry Oven, 100C Mass Loss (%)	90% Chamber, 50C Mass Gain (%)	Submerged, 25C Mass Gain (%)
HP ∥	<14	1.1 ± 0.5	0.97 ± 0.07	4.6 ± 0.3
HP ⊥	<14	0.12 ± 0.01	0.33 ± 0.05	3.7 ± 0.5
M26 ∥	<4.7	0.025 ± 0.003	0.020 ± 0.005	2.7 ± 0.3
M26 ⊥	<4.7	0.035 ± 0.004	0.019 ± 0.008	3.2 ± 0.8
M ∥	<3.0	0.026 ± 0.005	0.018 ± 0.005	1.8 ± 0.1
M ⊥	<3.0	0.036 ± 0.003	0.005 ± 0.003	1.7 ± 0.1

% OP

BN wt%

- Mass change is inversely correlated to silica content….
- Absorption effect rather than silica related effect……

Moisture Absorption on Strength

- Samples from moisture absorption study were tested for flexural strength and elastic modulus after soak.
- HP ∥, HP ⊥, and M ∥, all have significant changes in strength and elastic modulus properties with moisture exposure ($P<0.05$).

	<5% Rel. Humidity	~60% Rel. Humidity	90% Rel. Humidity		
Sample	**Dry Oven Strength (MPa)**	**As-machined Strength (MPa)**	**90% Chamber Strength (MPa)**	**P-Value [Oven vs Chamber]**	
HP ∥	52	43	28	0.000005	46%Δ
HP ⊥	80	76	70	0.005	13%Δ
M26 ∥	60	62	58	0.3	3%Δ
M26 ⊥	43	50	40	0.2	7%Δ
M ∥	24	25	22	0.01	8%Δ
M ⊥	60	62	60	0.3	0%Δ

Slow Crack Growth (SCG)

- Ceramics and glass exhibit loss of strength over time under static loads. The phenomenon is know as “slow crack growth” or “static fatigue” and is a form of stress corrosion.
- No data in the literature on BN.
- We need to know the SCG parameters for BN to see if stress corrosion should be a design consideration.
- Usual model: $$v = \frac{da}{dt} = AK_I^n = A^* \left[\frac{K_I}{K_{IC}} \right]^n$$

<u>Sensitivity Parameter n</u>

< 20 high sensitivity (glasses)

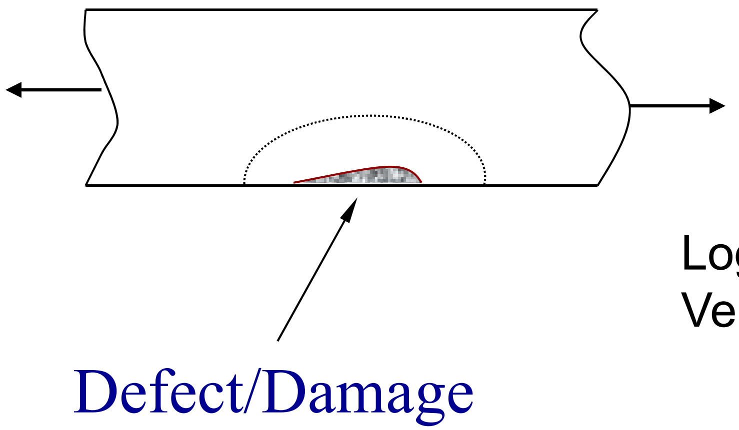
< 40 intermediate (alumina's)

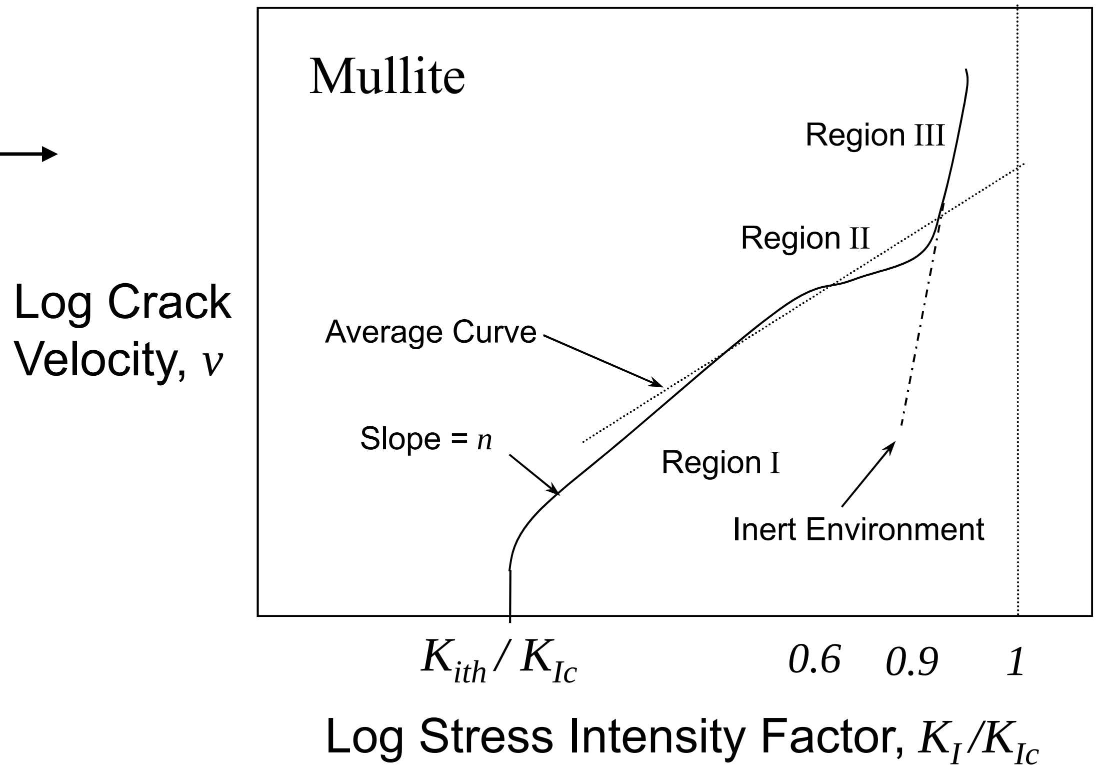
> 50 low (Si_3N_4)

> 100 ~ insensitive (Ge, Si, α SiC)

- v – crack velocity
- σ is Stress
- n & A are SCG parameters
- K_{Ic} is Fracture toughness

SCG Observations and Formulations





- Region I crack growth Function:

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

Rapid Technique for SCG Parameters

- Constant Stress Rate Testing or "Dynamic fatigue":
  - well defined time-to-failure
  - can be rapid
  - simple test

- Strength based approach with advantages & disadvantages:
  - samples the inherent, small flaws (length scale)
  - statistical scatter (many specimens needed)
  - averaging of regions of the SCG curve
  - shorter time scale than the application

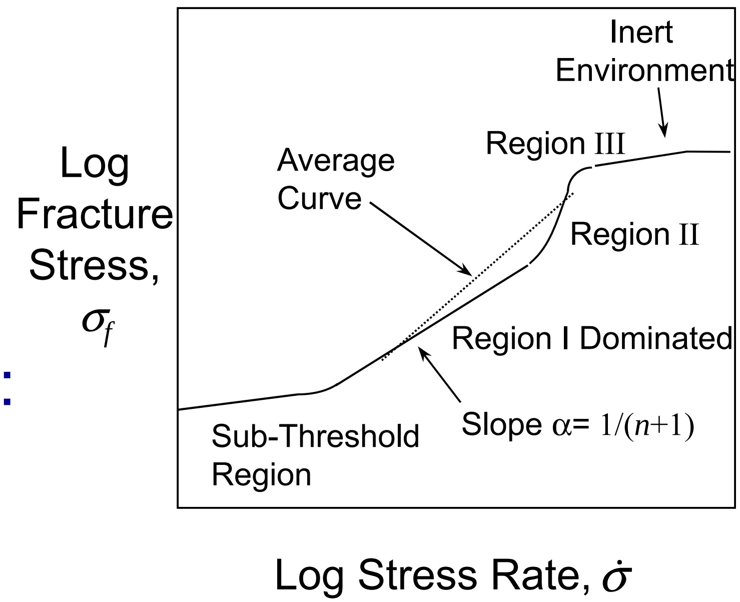
SCG Analysis

- Crack growth Function:

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

- Constant Stress Rate Testing:

$$S_f = \left[B(n+1)\sigma_i^{n-2}\dot{\sigma} \right]^{1/(n+1)}$$



- 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)

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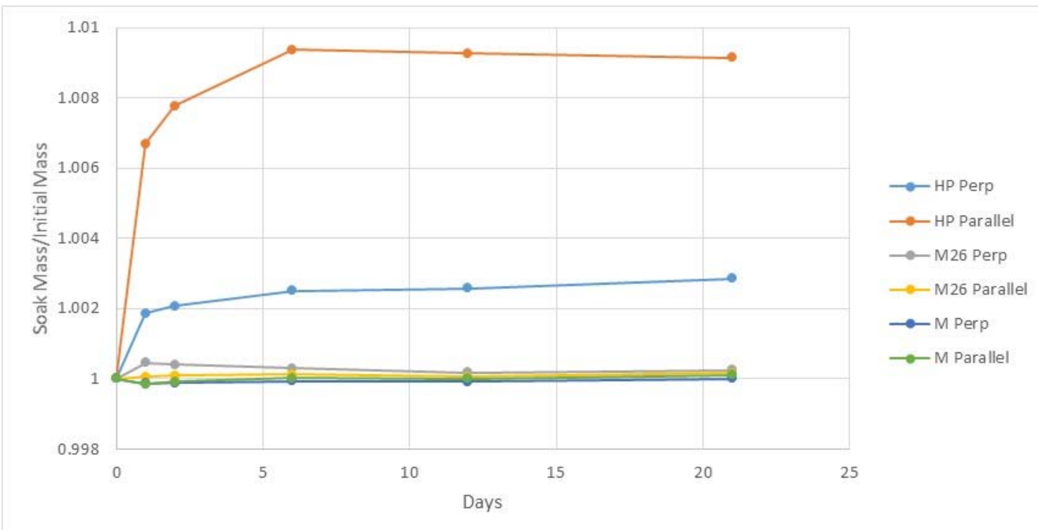
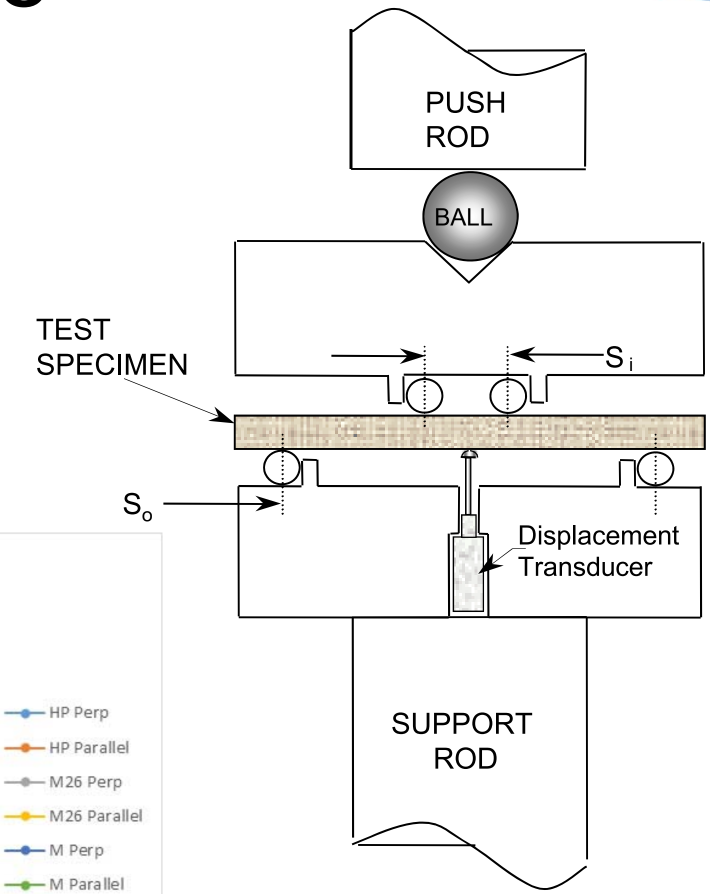
Experimental Procedure

- Constant Stress Rate Tests
  (10 to 0.001 MPa/s)
- Uniaxial Flexure (4-point)
- As-machined surfaces

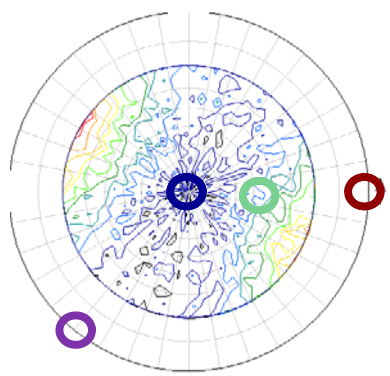




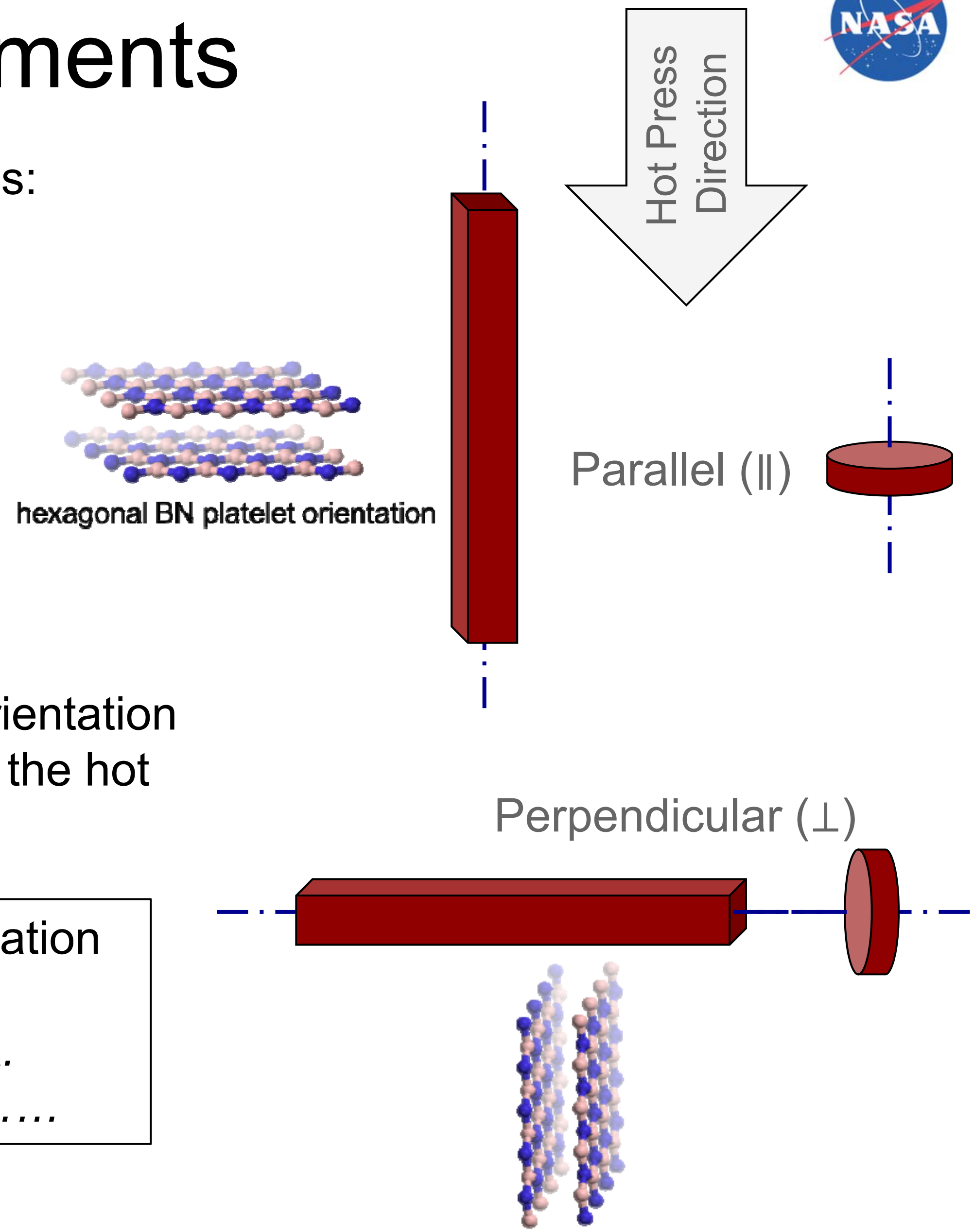
➢ Test in air at 60% RH after conditioning for >5 days.

Property Measurements

- Building dataset to compare grades:
  - Collecting data from 25 to 900°C .

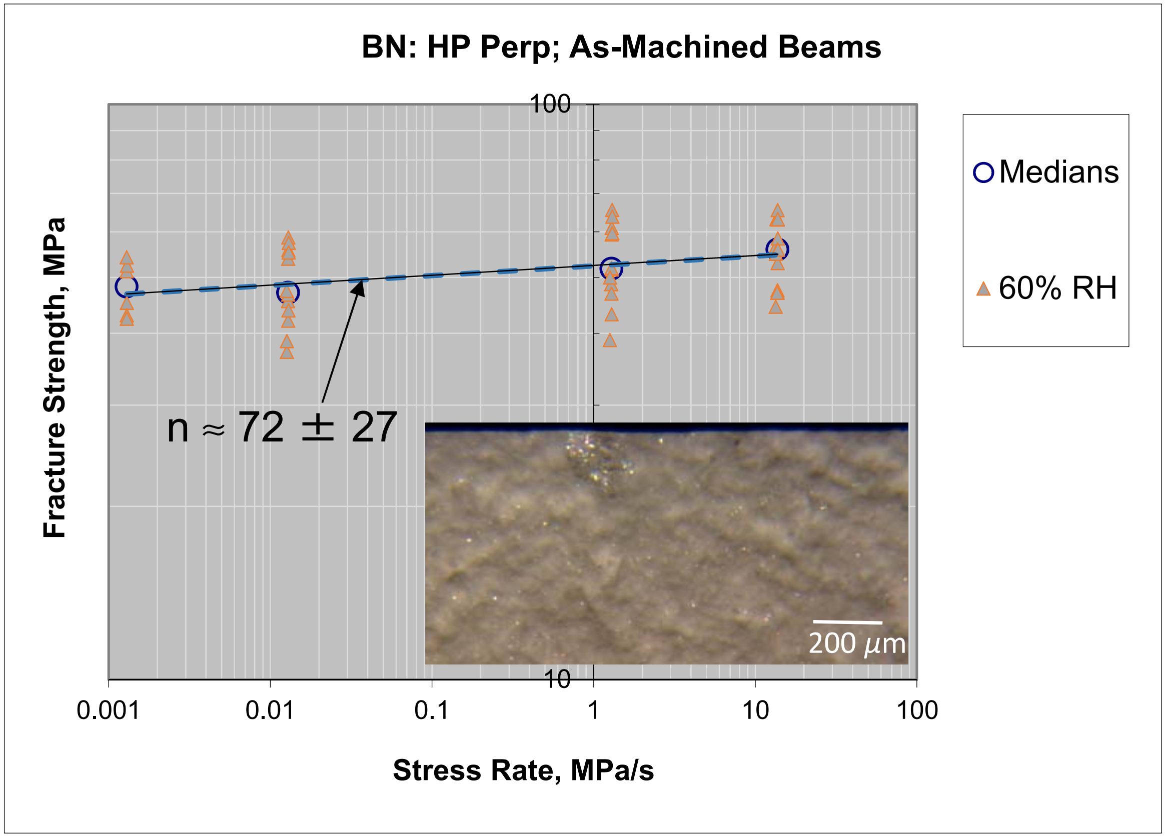


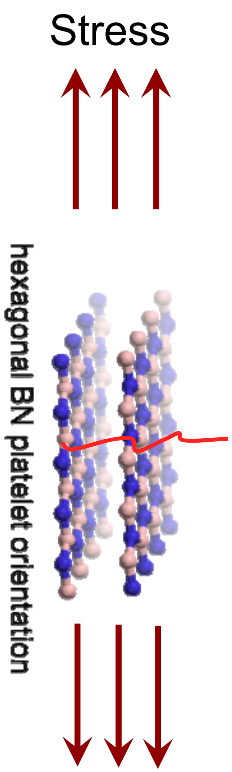
- Collecting data on samples with orientation "Parallel ∥" or "Perpendicular ⊥" to the hot press direction.



NASA/TM—2018-219949 "Evaluation of Boron Nitride Materials"
Jonathan A. Mackey, Jonathan A. Salem, and Hani Kamhawi…………

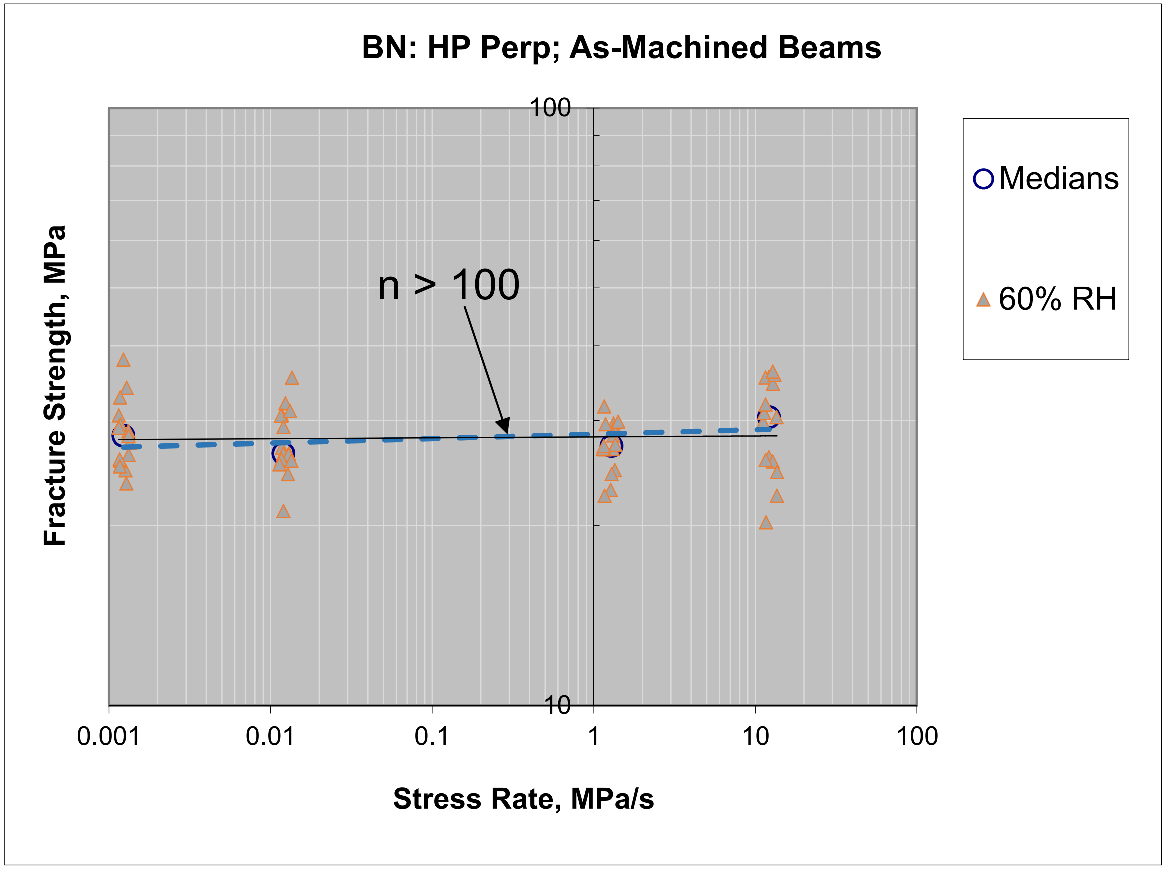
Results for HP BN Perpendicular

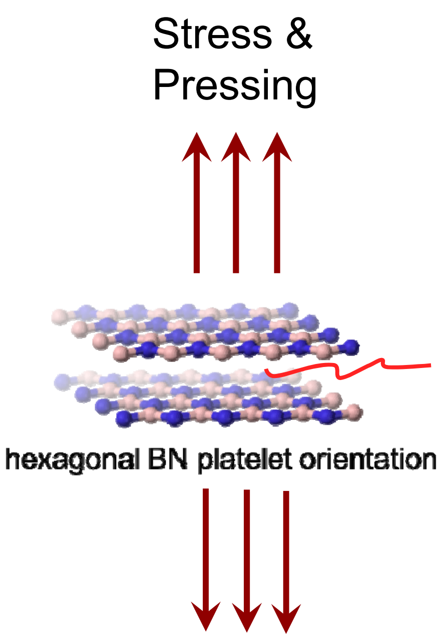




- SCG parameter n = ~72 ± 27
- Good corrosion resistance to SCG across platelets.

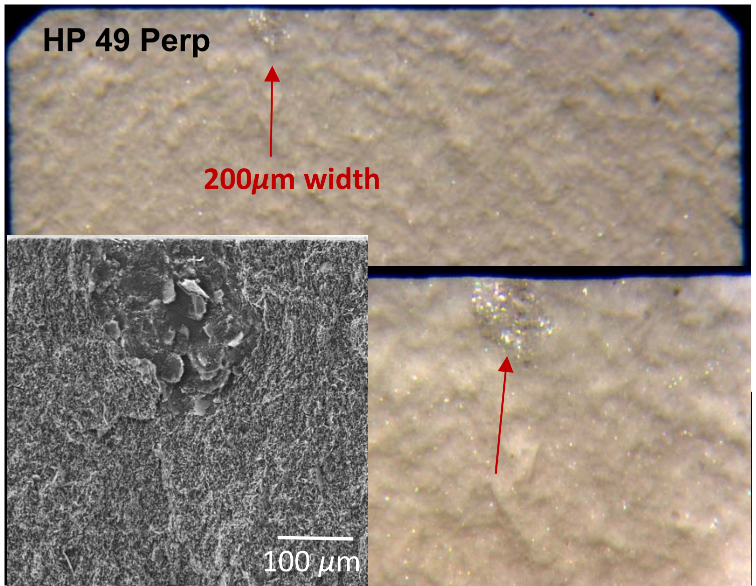
Results for HP BN Parallel

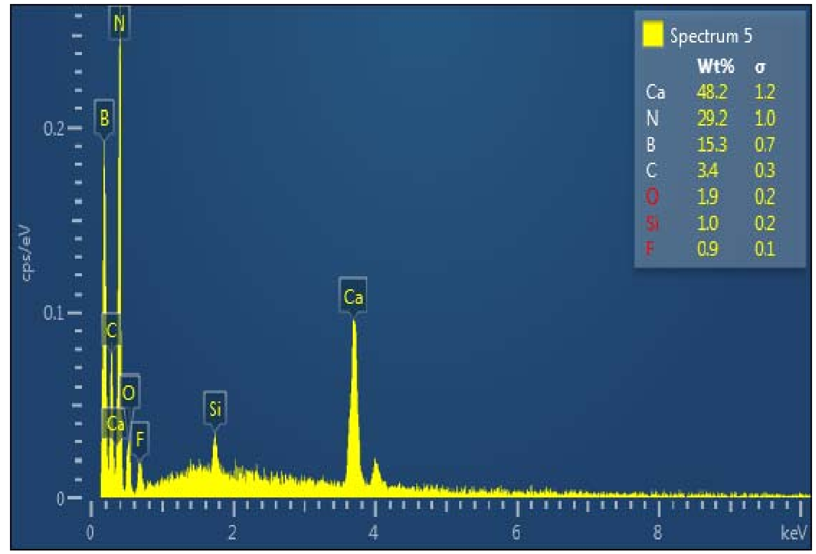


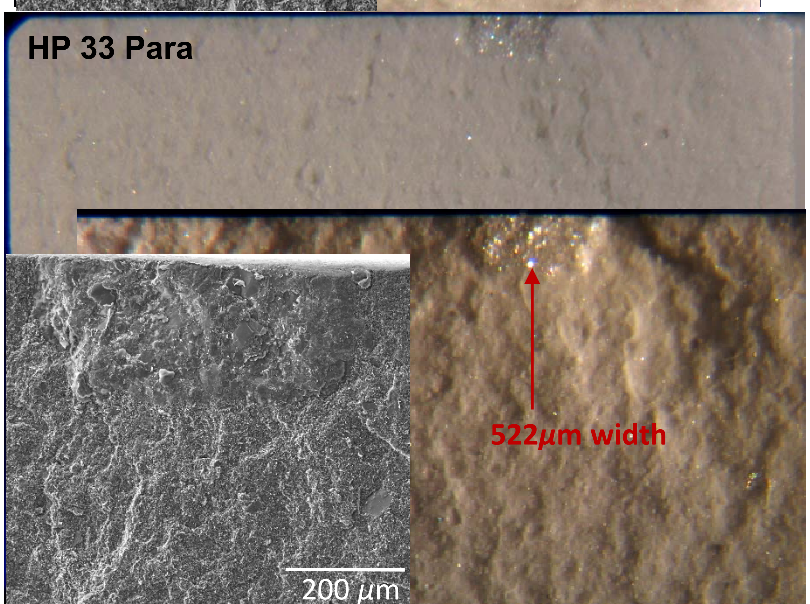


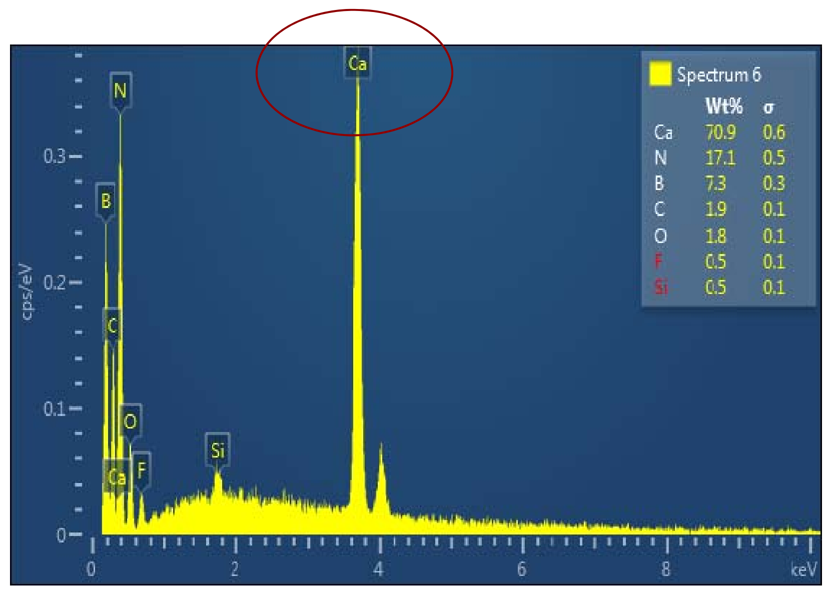
- SCG parameter *n* >100 is very high.
- Very good corrosion resistance between platelets.

HP Failure Source: Ca rich agglomerates

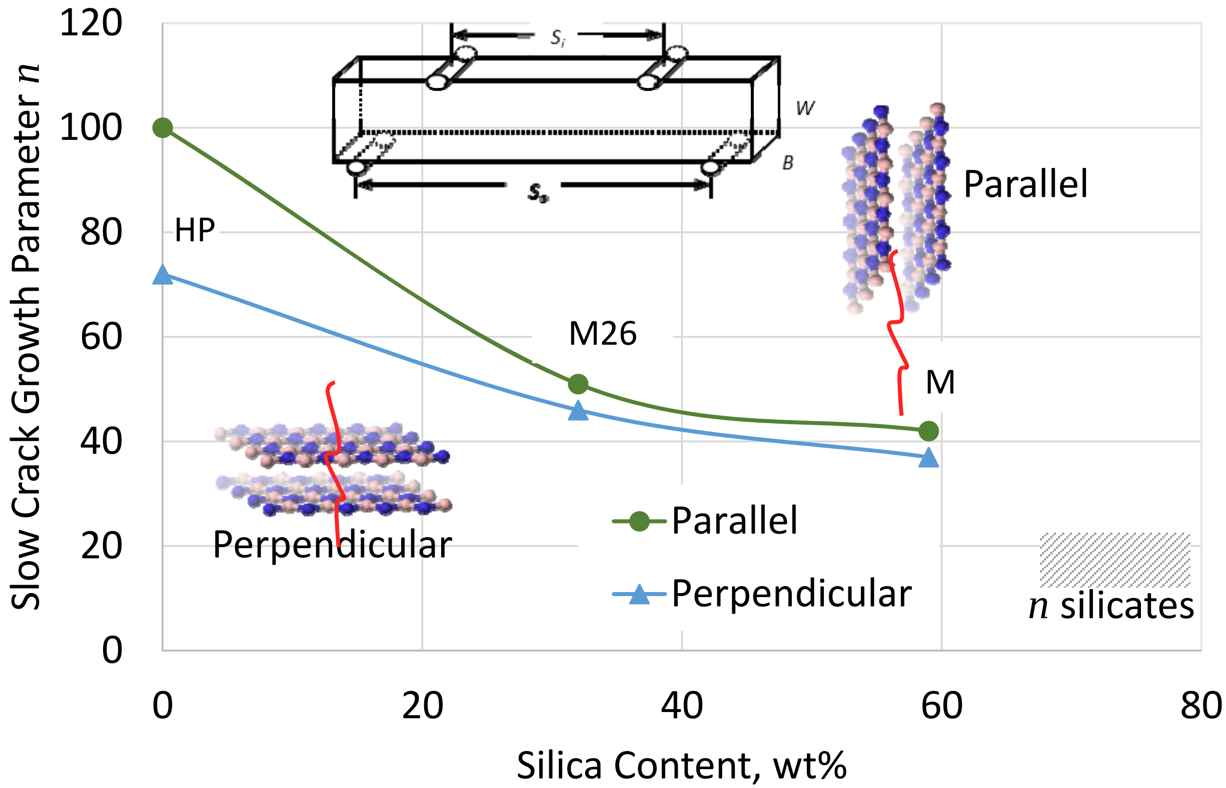








Slow Crack Growth Results



Material	SiO_2 wt. %	*n*
HP ⊥	0	72
HP ‖	0	>100
M26 ⊥	32	46
M26 ‖	32	51
M ⊥	59	37
M ‖	59	42
Glass	73	20
SiO_2	100	40

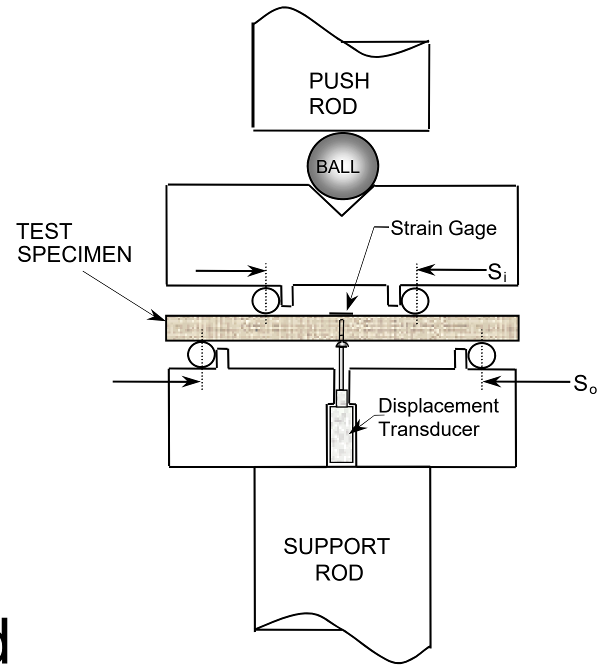
- Silica additions increases slow crack growth susceptibility.
- Better than glass. Easier to corrode across rather than between the platelets.
- Van der Waals bonds are resistant to corrosion…….
- Strength loss observed in moisture study is likely an absorption effect rather than a slow crack growth effect (large n for HP //).

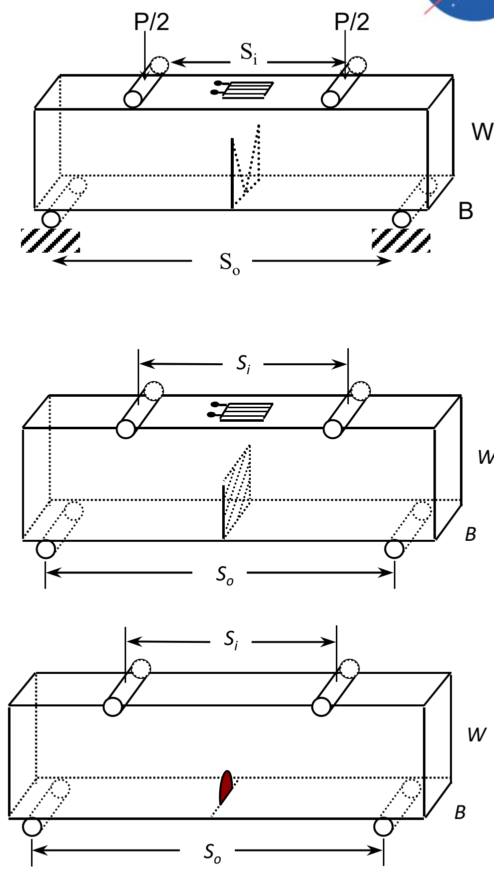
Fracture Toughness

- Three standard methods are available.
- Relatively simple setup: fixture, test frame, load cell, recording device.

x Indentation caused crushing w/o cracking.

x That made SCF and SEPB difficult.

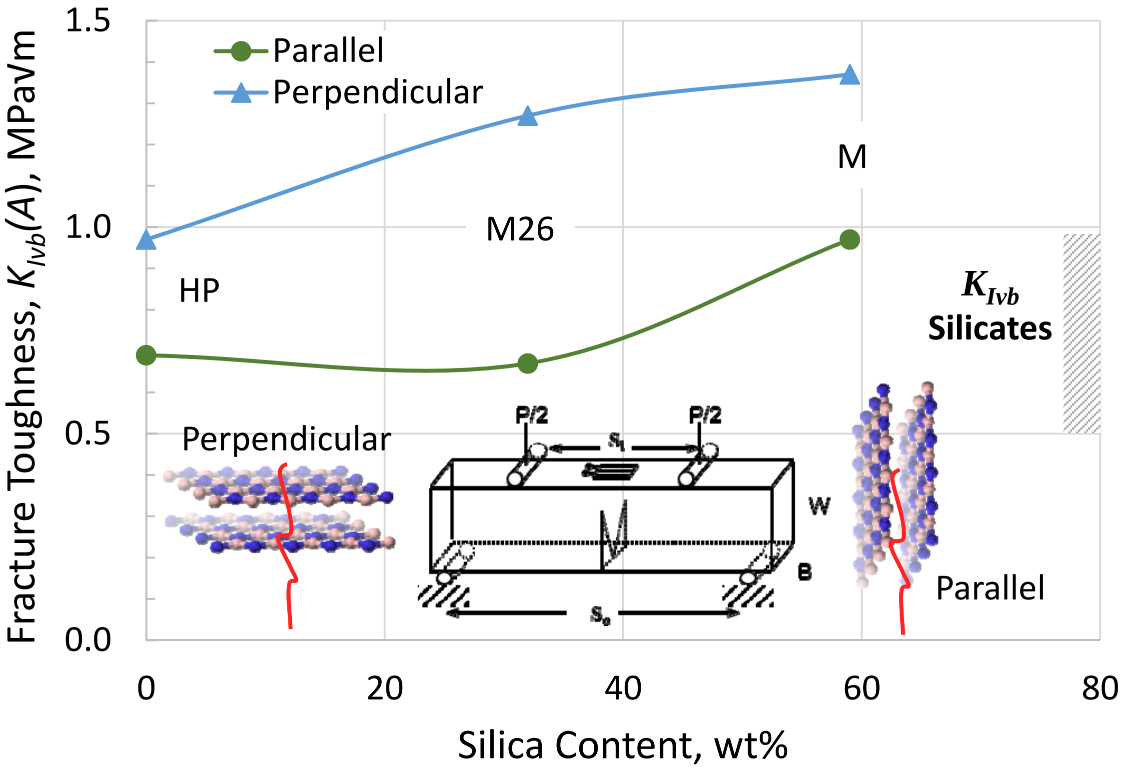




"Measuring the Real Fracture Toughness of Ceramics: ASTM C1421," J.A. Salem, G.D. Quinn, M.G. Jenkins, pp. 531-553 in Fracture Mechanics of Ceramics: Active Materials, Nanoscale Materials, Composites, Glass, and Fundamentals, Springer, (2005).

➢ Used the chevron notch.

Fracture Toughness Results

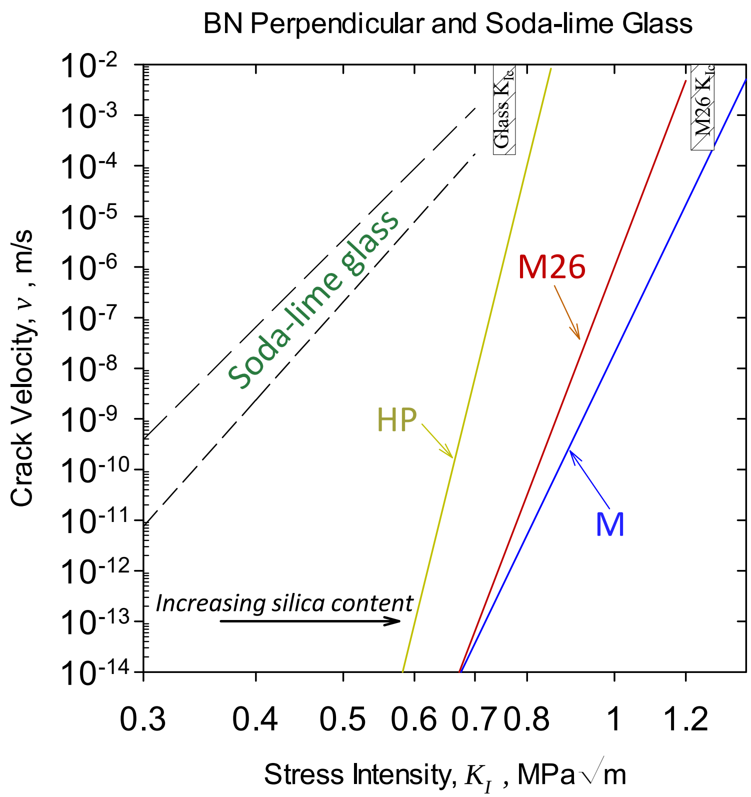


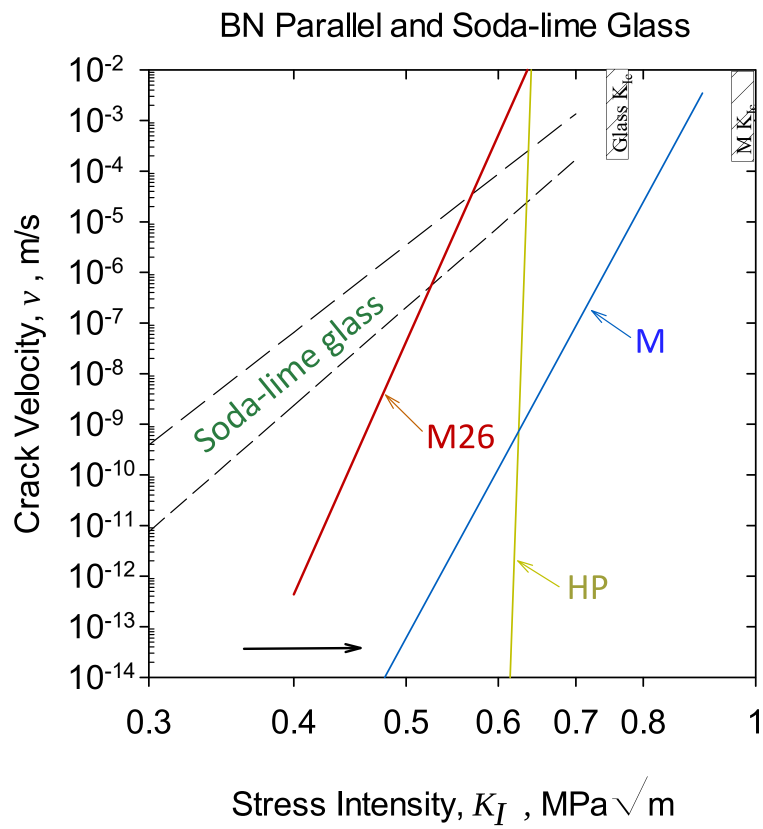
Material	Silica Content wt.%	K_{Ivb} (N_2) MPa√m
HP ⊥	0	0.97
HP ‖	0	0.69
M26 ⊥	32	1.27
M26 ‖	32	0.67
M ⊥	59	1.37
M ‖	59	0.97
Glass	73	0.78
SiO_2	100	0.75
Si_3N_4	0	4+
Steel	0	>20

- Silica additions increases toughness.
- Similar to glass. Easier to separate than split the platelets.
- This contrasts with the SCG behavior…..Bond type…….

Crack Velocity Curves BN vs Glass

- Glasses readily exhibit SCG and thus are a good comparison.





- As compared to glass, BN's are less sensitive to changes in K_I, but M26|| and HP|| have lower K_{Ic}.

Summary

- Fracture toughness is low, around that of glass, and is a function of orientation (bond type). ☹
- It is easier to fracture van der Waal bonds between BN platelets than the covalent bonds within platelets.
- Increasing silica content in BN's increases toughness.☺
- BN's exhibits limited slow crack growth in humidity. ☺
- Increasing silica content in BN's increases SCG. ☹
- SCG parameters are a function of test orientation due crystallographic texture & bond type.
- Van der Waal bonds between platelet are insensitive to water.
- Covalent bonds within platelets are sensitive to water.

Summary (Cont.)

- Crack growth is due to SCG as driven by residual absorption stresses or externally applied stresses.

Future work:

- Fractography to identify origins.
- Reliability analysis of BN components.

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NASA