Effect of Control Mode and Load Rate on Fracture Toughness of Brittle Ceramics

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Background – Fracture Toughness

- Key design parameter for brittle materials
- Endpoint of the slow crack growth (SCG) curve
- Materials need to be investigated in varied environments for aerospace applications (high vacuum to humid launch pad)

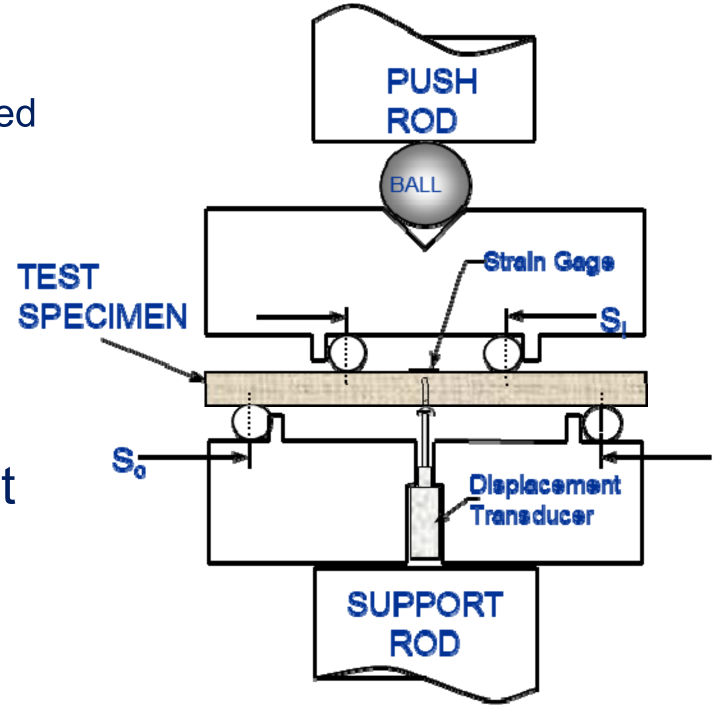
Background – Stress Corrosion

- Materials under stress exhibit slow crack growth in corrosive environments
- For some materials (silica glasses, silicon nitrides) sensitivity extends to humid air
- Stress corrosion susceptibility implies test rate and control mode sensitivity
  - New surface creation (crack growth) rate is negatively correlated with strength for stress corrosion-sensitive materials

S. W. Freiman, S. M. Wiederhorn, and J. Mecholsky John J., “Environmentally Enhanced Fracture of Glass: A Historical Perspective,” *Journal of the American Ceramic Society*, vol. 92, no. 7, pp. 1371–1382, Jul. 2009.

Procedure

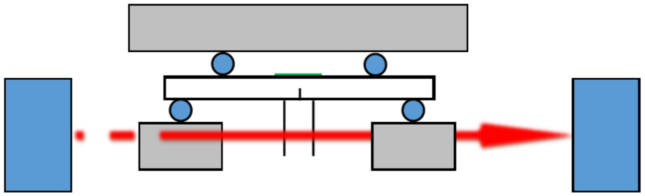
- Testing in accordance with ASTM 1421
  - Standard Test Method for Fracture Toughness of Advanced Ceramics at Ambient Temperatures
  - Chevron-notched beam, four-point bending
- Testing controlled by PID (proportion-integral-derivative) feedback loop
- Stroke control testing via system displacement transducer
- Strain control via back face strain gage
- CMOD control via laser micrometer



ASTM C1421-15, Standard Test Methods for Determination of Fracture Toughness of Advanced Ceramics at Ambient Temperature, ASTM International, West Conshohocken, PA, 2015, www.astm.org.

Approach – Control Mode

- Crack growth stability beyond peak loads is necessary for energy calculations.
  - Want to avoid catastrophic/instantaneous crack extension
- Stability is achieved through stiff testing equipment (load cell and actuator)
- Testing control mode affects stability

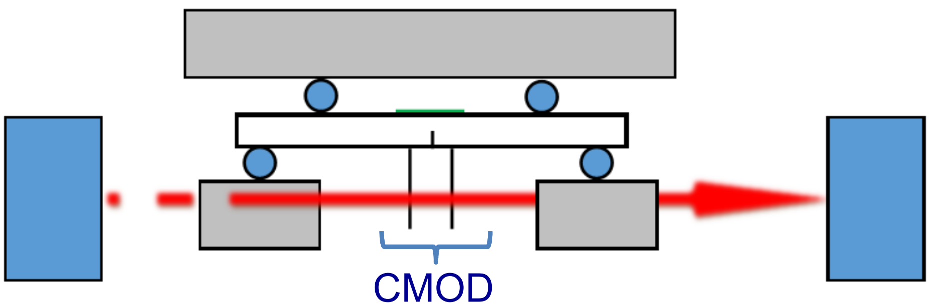


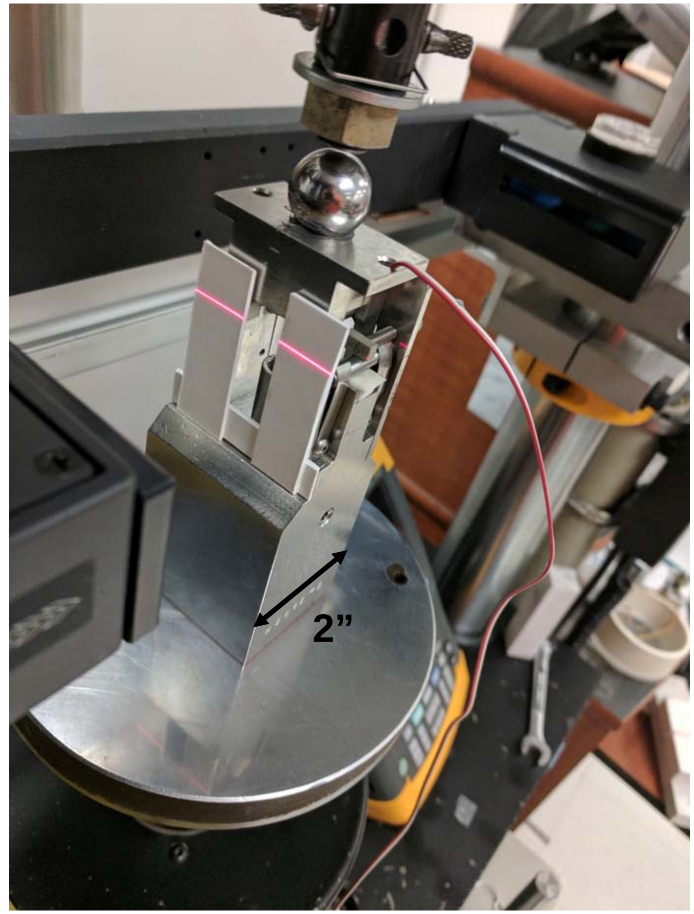
  - CMOD (**crack mouth opening displacement**) control >= Strain Control > Stroke Control
  - CMOD testing difficult to achieve in brittle, low fracture toughness materials due to a low signal to noise ratio. Stiff (high capacity) load cells can be used to test low fracture toughness brittle materials.

Garcia-Prieto, A., Hernandez, J., Lopez, M., and Baudin, C., "Controlled fracture test for brittle ceramics," Journal of Strain Analysis, vol. 46, 2011, pp. 27–32.

Procedure – CMOD Measurement

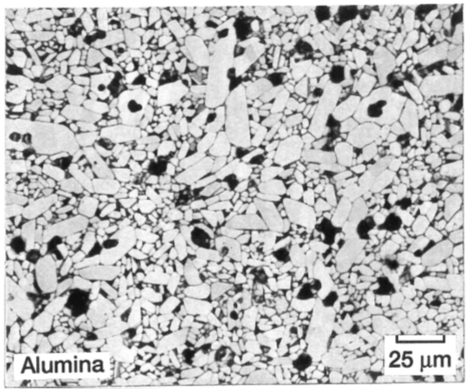
- CMOD testing via laser micrometer measurement
  - Proved difficult to maintain feedback loop
  - Can easily measure CMOD, difficult to test at a set CMOD rate
  - Linearly related to back face strain

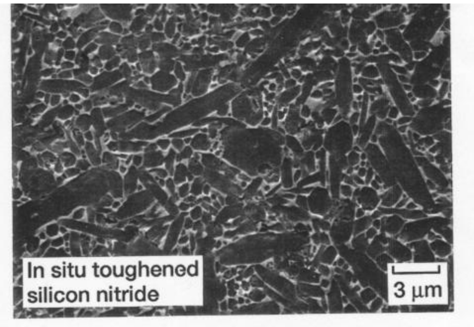


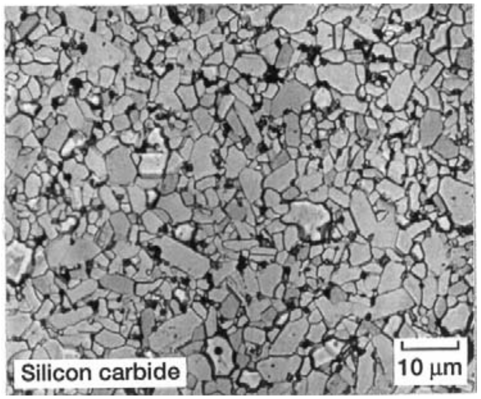


Procedure – Tested Materials

- ALSIMAG Alumina
  - Moderate stress corrosion expected
- α-Silicon Carbide
  - Little to no stress corrosion expected
  - Have observed no response to changes in humidity
  - Rate effect left to future work, suspected to be negligible
- AS800 Silicon Nitride
  - Slight stress corrosion expected







Tested Materials – ALSIMAG Alumina

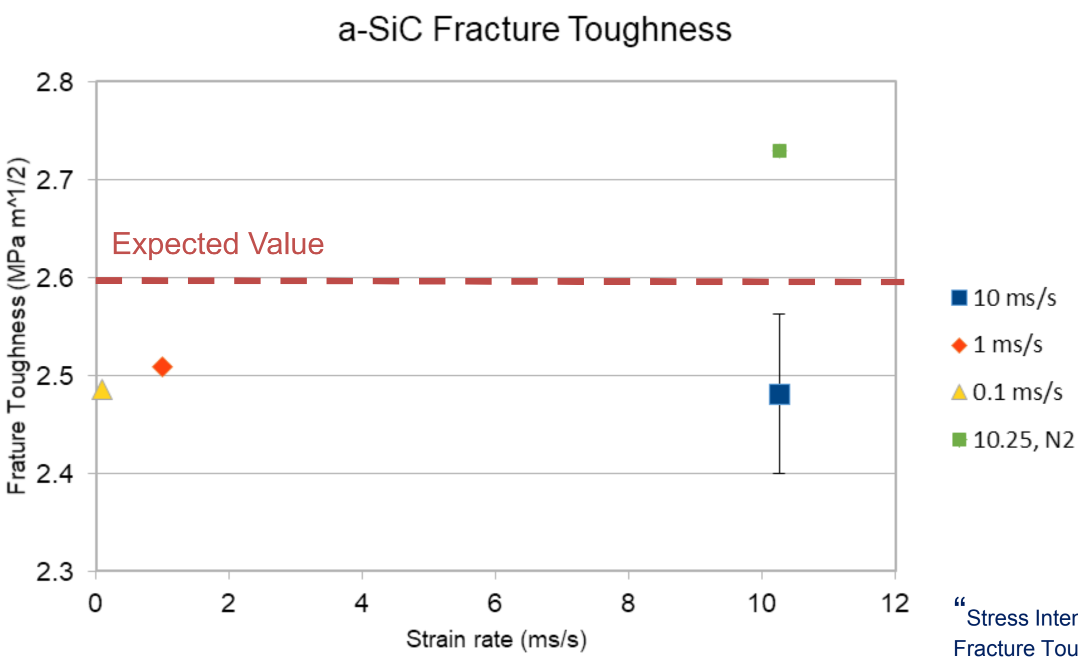
- Both rate effect and environmental stress corrosion observed:

Test Specimen K_{Ivb} *(A)* MPa√m	Test Environment		
	Water	Air	Silicone Oil or Dry N_2
0.05 mm/min	2.75 ± 0.01 (4)	3.19 ± 0.07 (7)	3.37 ± 0.05 (4)
0.01 mm/min	2.64 ± 0.06 (3)	2.93 ± 0.10 (3)	3.39 ± 0.02 (2)

- Difference of about 20% between water and N_2 testing. Testing rate affected results by 4~8%, with a consistent trend
- Decrease in fracture toughness with test rate implies corrosion effect; most of the effect was eliminated with N_2
- Future testing planned

Tested Materials – α SiC in Strain Control

- Possible environmental stress corrosion observed, negligible rate effect
- Preliminary data, too low of sample size for meaningful conclusion



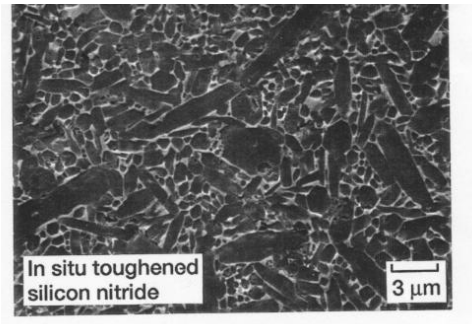
Material	K_{Ivb} MPa√m	K_{Ipb} MPa√m	K_{Isc} MPa√m
α-SiC (JAS)	2.61 ± 0.05	2.58 ± 0.08	2.76 ± 0.08
α-SiC (UW)	2.62 ± 0.06	2.54 ± 0.20	2.69 ± 0.08
ADS96R	3.56 ± 0.03	3.71 ± 0.10	----
ALSIMAG 614	3.19 ± 0.06	3.09 ± 0.17	3.18 ± 0.10
ALSIMAG 614	3.13 ± 0.03	2.98 ± 0.06	----
NC132	4.60 ± 0.13	4.59 ± 0.12	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

"Stress Intensity Factor Coefficients For Chevron-Notched Flexure Specimens and A Comparison Fracture Toughness Methods," J.A. Salem, L. Ghosn, M.G. Jenkins and G.D. Quinn, *Ceramic Engineering and Science Proceedings*, Vol. 20, No. 3, pp. 503-512 (1999).

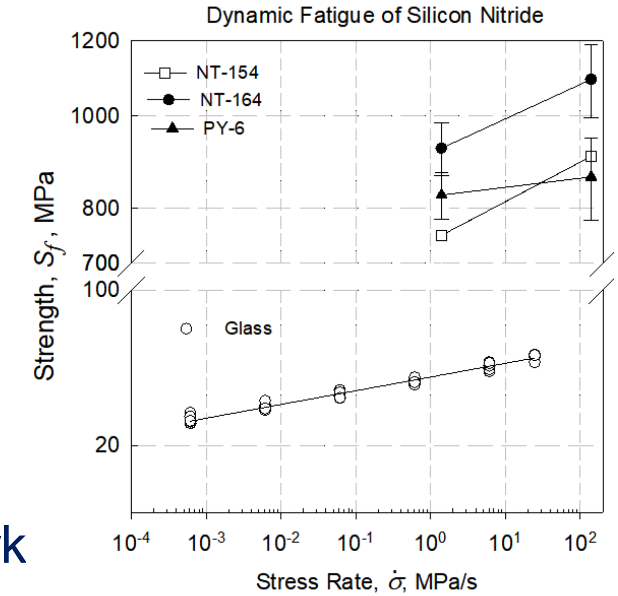
Tested Materials – AS800 Silicon Nitride

- Two of three nitrides have very low slow crack growth parameter n; Very limited data, but implication is that some nitrides exhibit SCG like glass.
- Suspect intergranular grain boundary phase as the cause.

Material	SCG Parameter *n*
NT154	23
NT164	27
PY-6	104
Glass	21

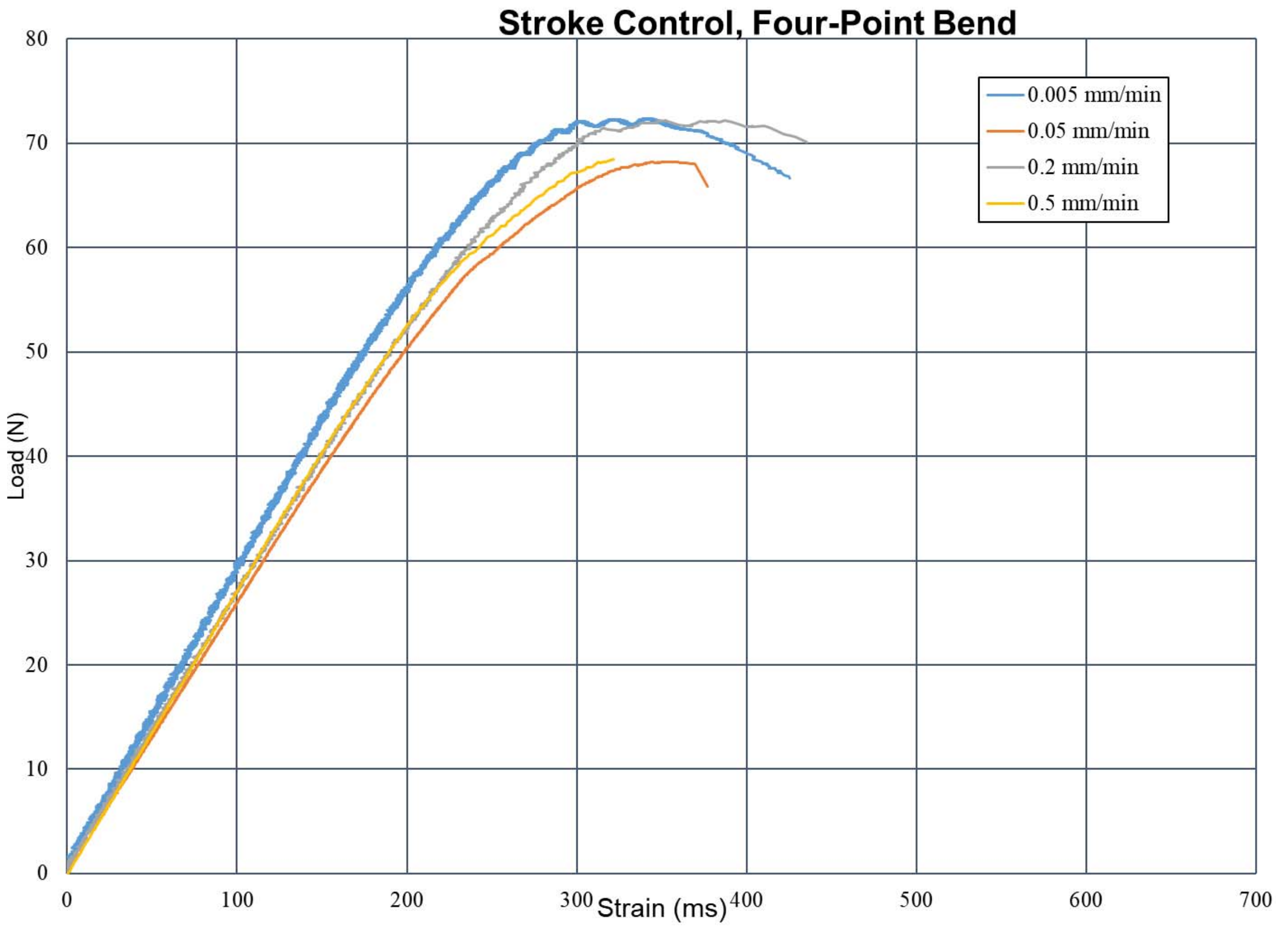


$$v = \frac{da}{dt} = A_I K_I^n = A_I^* [\frac{K_I}{K_{IC}}]^n$$



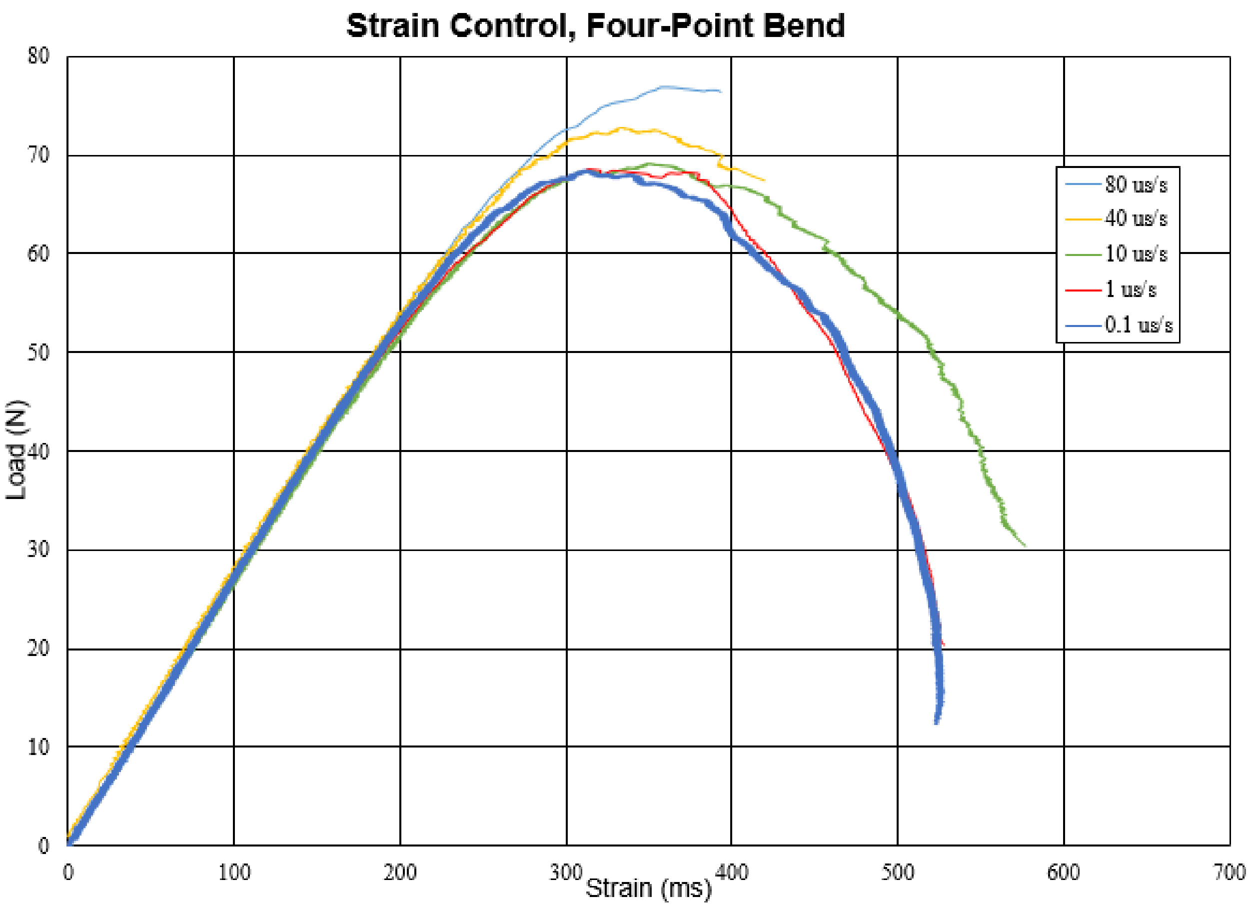
- Dynamic Fatigue Testing of AS800 left to future work

Results – AS800 Stroke Control



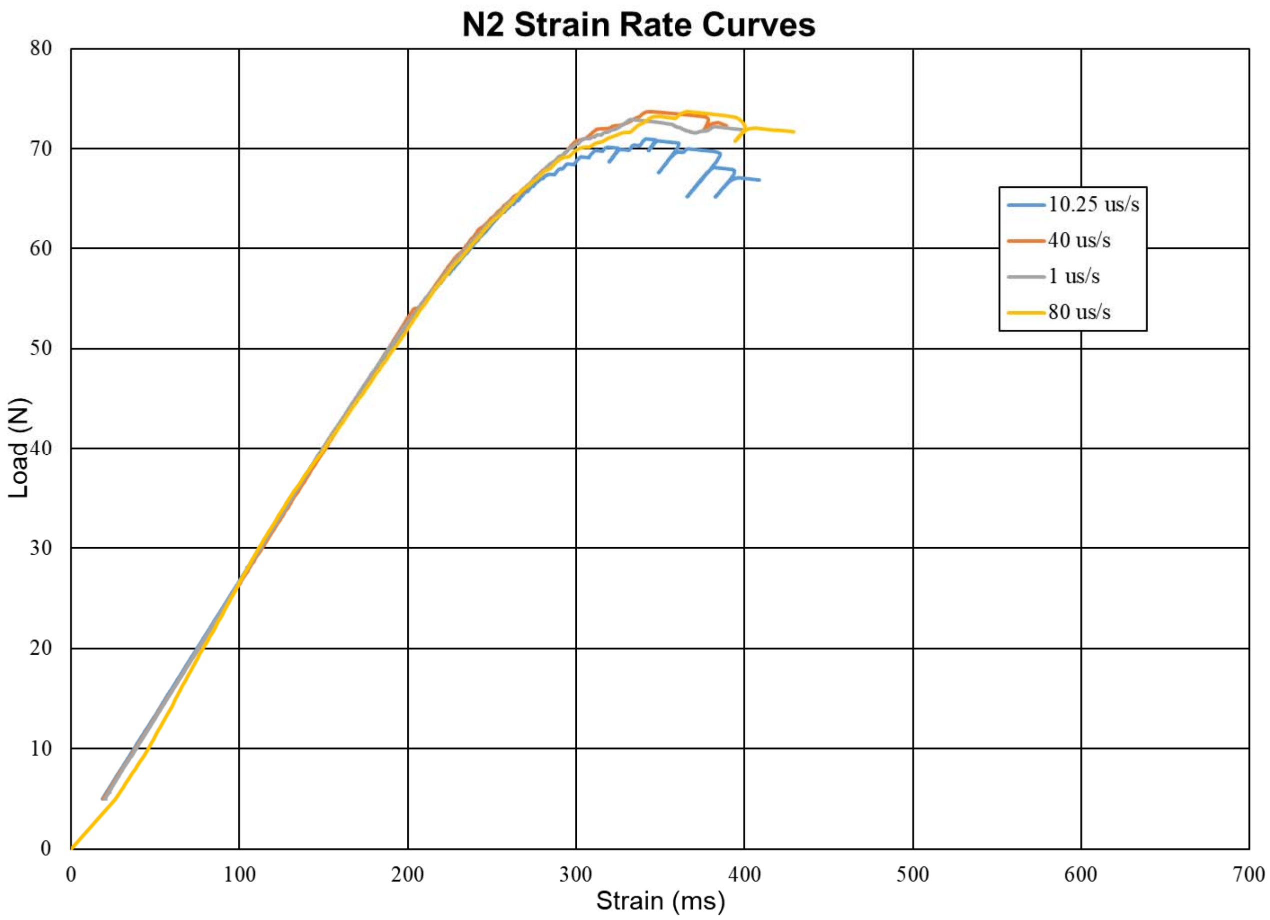
- Stable through max load at all rates except for 0.5 mm/min, which typically failed prematurely
- No well-defined rate sensitivity

Results – AS800 Strain Control – Lab Air



- Stability up to 80 ms/s
- Small but significant rate sensitivity

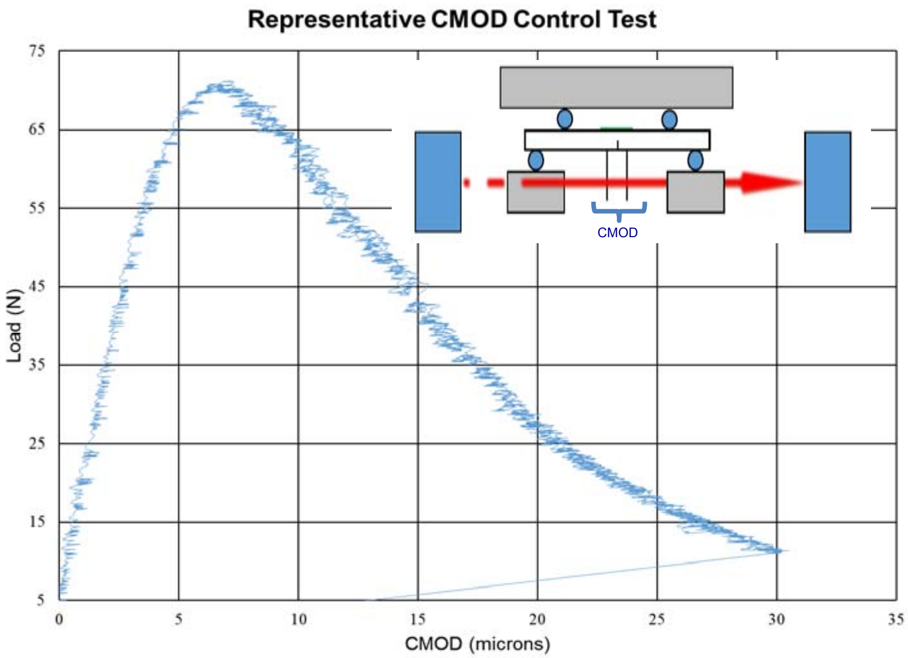
Results – AS800 Strain Control – Dry N_2



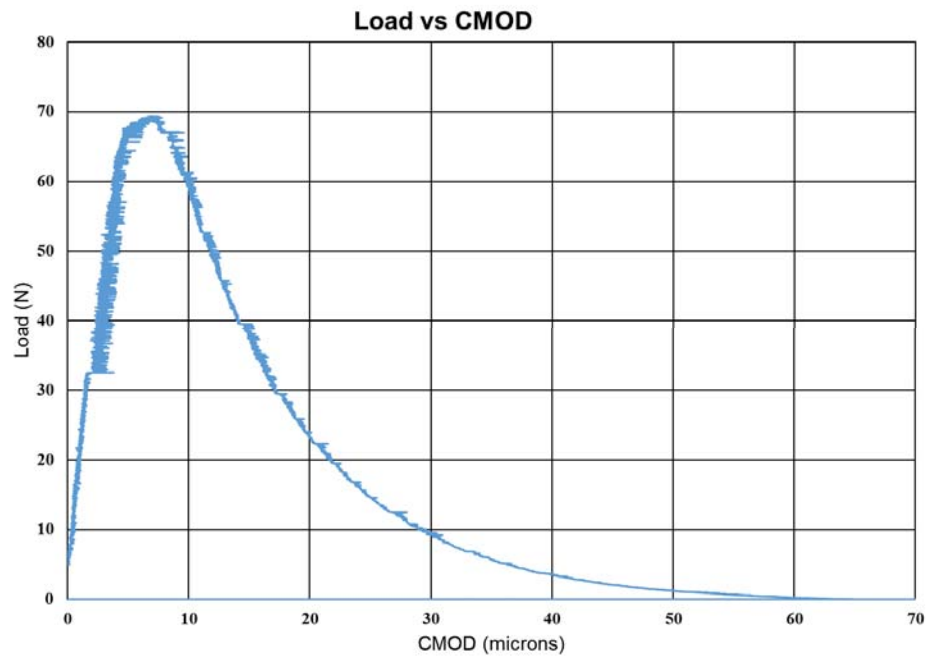
- Much more consistent result – N_2 removes rate effect, implying humidity was the cause.

Results – AS800 CMOD Control

CMOD Control



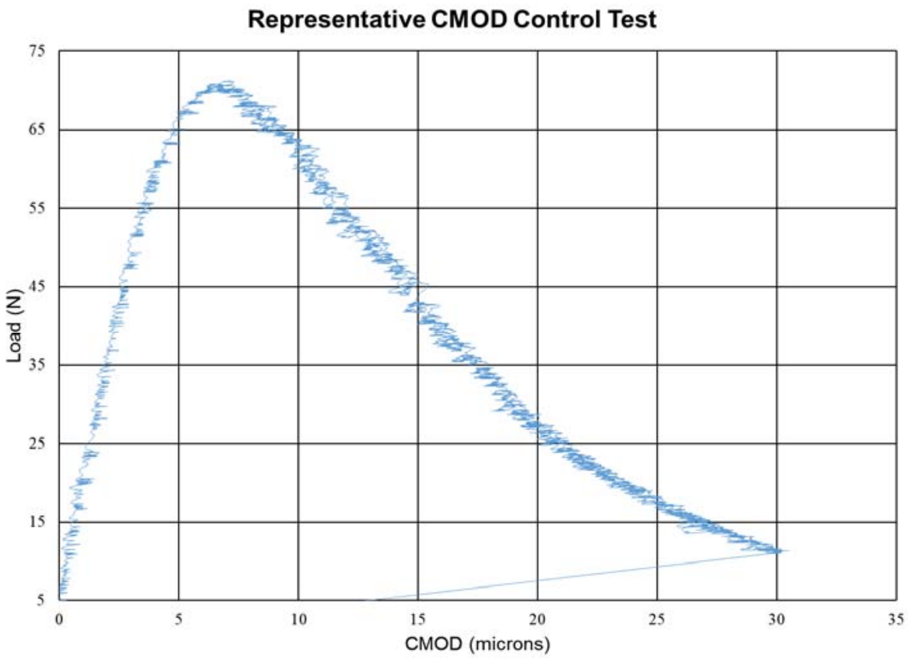
Back Face Strain Control



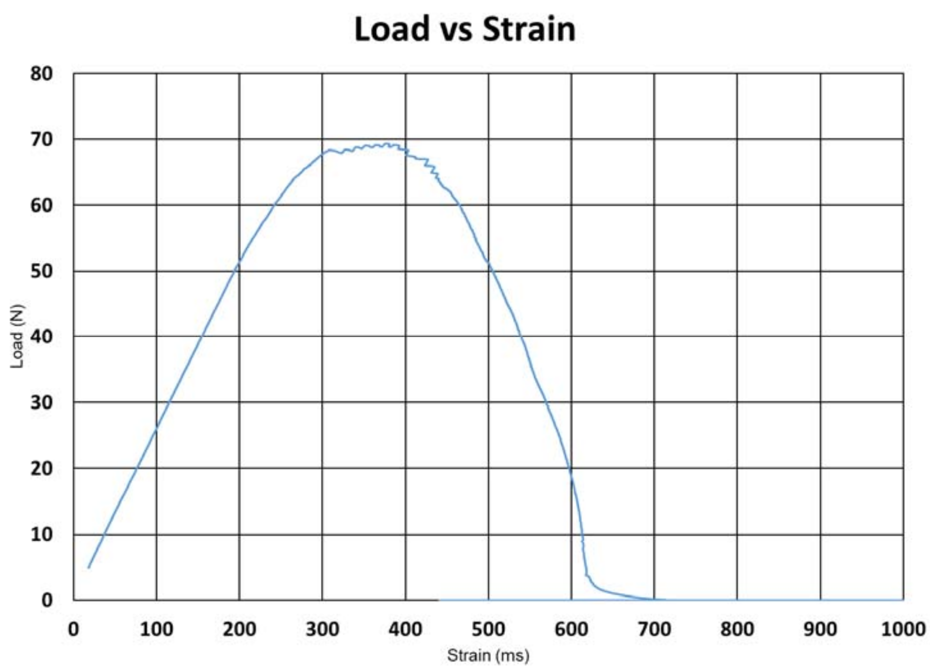
Asymptotic drop in load can be observed as a function of CMOD, with constant CMOD rate; more intuitive curve than BF strain produces

Results – AS800 CMOD Control

CMOD Control



Back Face Strain Control



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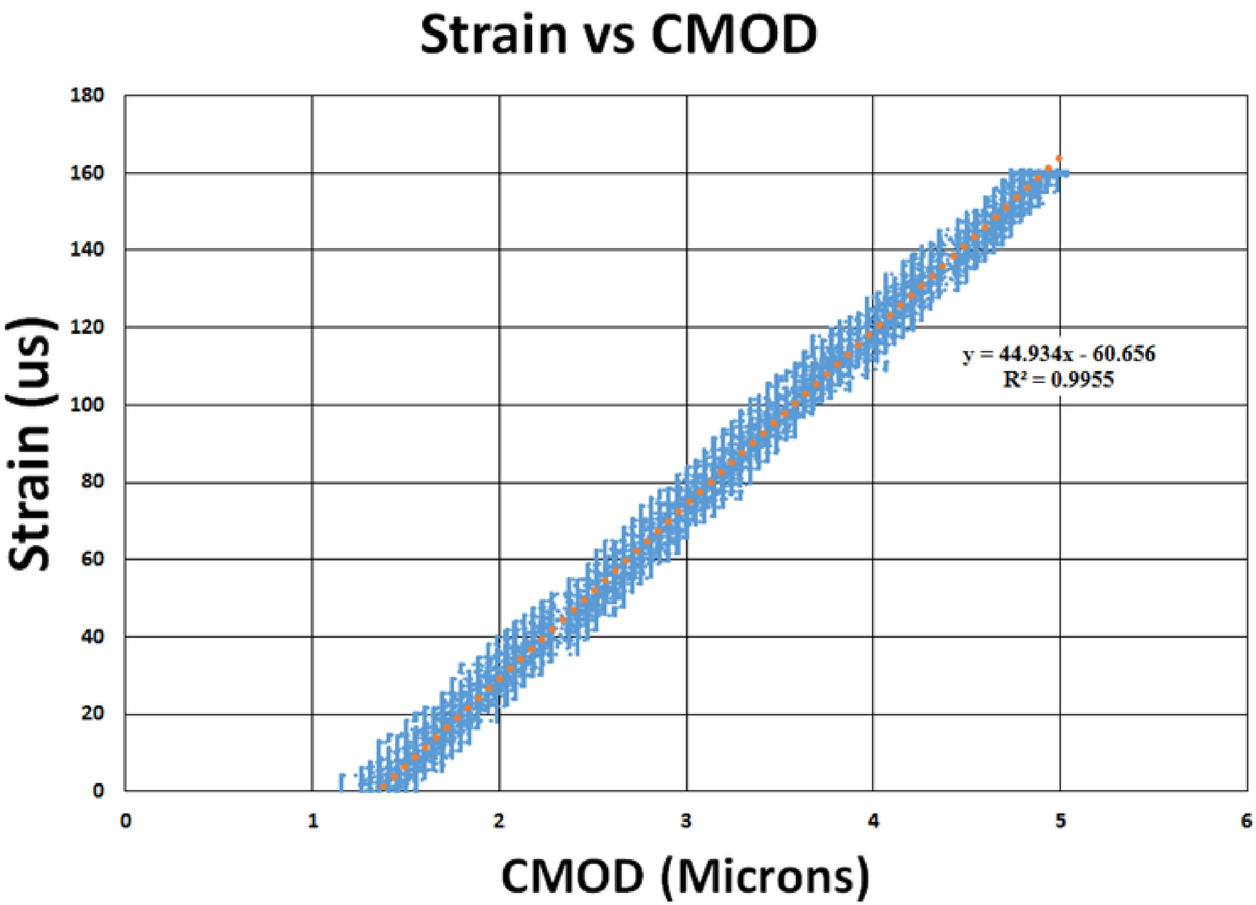
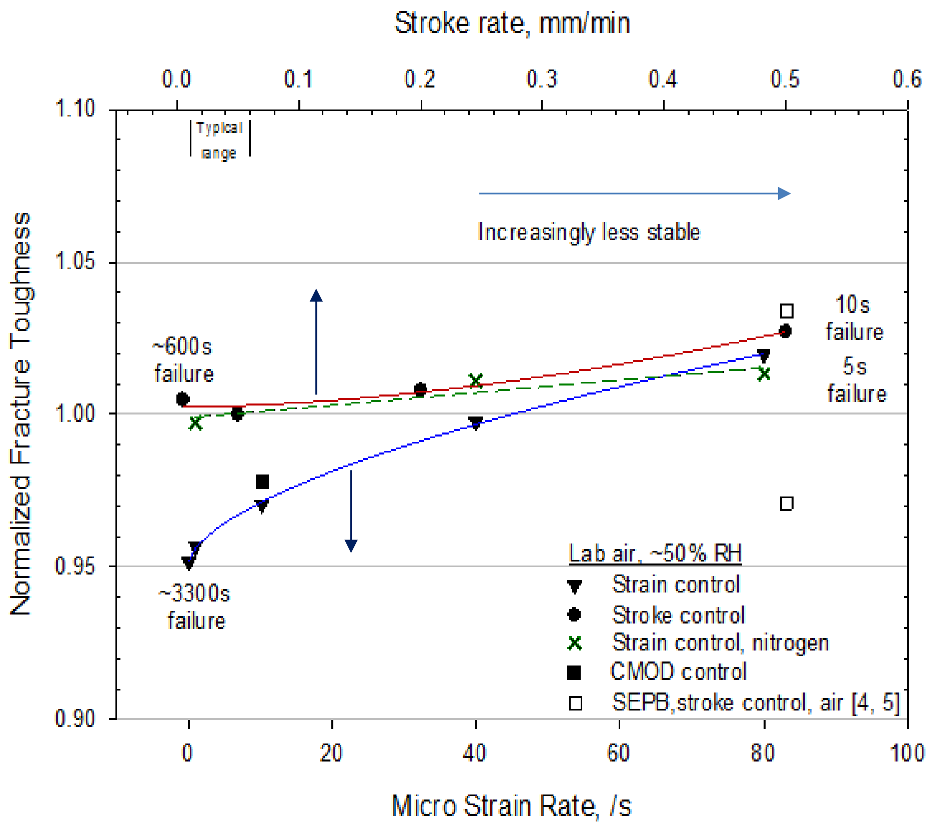
Results – AS800 CMOD Control



- Difficult to govern CMOD rate using direct measurement, can convert from BF strain however
- Linear relationship dependent on the material

Results – AS800 Stroke/Strain Comparison



- Stroke and strain control shows less than 5% difference over a very wide range of rates
- Stroke control produces less than 2% variation over a wide range
- Strain control results in lower fracture toughness, exhibits small but systemic increase over an extreme range.
- Testing in nitrogen eliminates strain rate sensitivity (<2%Δ), implying a corrosion-related effect.

Salem, Jonathan & Ghosn, Louis & Jenkins, Michael & Quinn, George. (2008). Stress Intensity Factor Coefficients for Chevron-Notched Flexure Specimens and a Comparison Fracture Toughness Methods. Ceram. Eng. Sci. Proc. 20. 503 - 512. 10.1002/9780470294567. ch58.

Choi, S.R. & Pereira, M.J. et al. "Foreign Object Damage Behavior of Two Gas-turbine Grade Silicon Nitrides by Steel Ball Projectiles at Ambient Temperature," NASA TM 20020082954, August 2002.

AS800 – Summary of Values

Testing Environment	Testing Mode and Rate (number of tests)	Fracture Toughness, (Mpa$\sqrt{m}$)	Deviation from Typical (0.05 mm/min) Test	Nominal time to peak load (s)
Lab Air	**Stroke Control**			
	0.005 mm/min	7.87±0.05	+0.48%	600
	0.05 mm/min	7.83±0.16	---	100
	0.2 mm/min	7.89±0.09	+0.7%	20
	0.5 mm/min	8.04±0.05	+2.7%	10
	Strain Control			
	0.1 με/s	7.45±0.15	-4.8%	3300
	1 με/s	7.49±0.28	-4.4%	300
	10.25 με/s	7.60±0.22	-3.0%	50
	40 με/s	7.81±0.10	-0.03%	10
	80 με/s	7.98±0.13	+1.96%	5
	CMOD Control			
	0.04 μm/s	7.66±0.45	-2.3%	50
Dry Nitrogen	**Strain Control**			
	1 με/s	7.81±0.18	-0.03%	300
	40 με/s	7.95±0.12	+1.0%	10
	80 με/s	7.94±0.59	+1.3%	5
All Data	---	7.79±0.19	-0.05%	---

- Least difference occurs for stroke control
- Most difference for strain control
- Nitrogen eliminates the effect

Conclusions

- Regardless of mode and environment, data converges for 5-10 second failure time
- 5% deviation in fracture toughness of AS800 with test rate, caused by humidity during testing; Confirmed with testing in N_2
- Similar trend found in ALSIMAG Alumina. SiC in progress, does not yet seem to exhibit stress corrosion
- CMOD can be correlated to back face strain linearly, allowing for constant CMOD rate in strain control
- CMOD as test metric may provide a more useful view of stable crack growth for energy calculations
- For general purposes, stroke control worked very well when testing silicon nitride with the chevron notch beam

Future Work

- Comprehensive testing of SiC and soda lime glass to examine highly sensitive and insensitive materials
- Dynamic fatigue testing of materials to quantify stress corrosion behavior in various environments
- N_2 testing of AS800 chevrons in stroke control to confirm increased stress corrosion from strain control
- Improvement of CMOD control capabilities

Acknowledgements

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