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Effect of Control Mode and Test Rate on Fracture Toughness of Advanced Ceramics

Bronson D. Hausmann
Case Western Reserve University, Cleveland, Ohio

Jonathan A. Salem
Glenn Research Center, Cleveland, Ohio

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Case Western Reserve University, Cleveland, Ohio

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Glenn Research Center
Cleveland, Ohio 44135

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Bronson D. Hausmann
Case Western Reserve University
Cleveland, Ohio 44106

Jonathan A. Salem
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Summary

The effects of control mode and test rate on the measured fracture toughness of ceramics were evaluated by using chevron-notched flexure specimens in accordance with ASTM C1421. The use of stroke control gave consistent results with about 2 percent (statistically insignificant) variation in fracture toughness for a very wide range of rates (0.005 to 0.5 mm/min). Use of strain or crack mouth opening displacement (CMOD) control gave ~5 percent (statistically significant) variation over a very wide range of rates (1 to 80 µm/m/s), with the measurements being a function of rate. However, the rate effect was eliminated by use of dry nitrogen, implying a stress corrosion effect rather than a stability effect. With the use of a nitrogen environment during strain-controlled tests, fracture toughness values were within about 1 percent over a wide range of rates (1 to 80 µm/m/s). CMOD or strain control did allow stable crack extension well past maximum force, and thus is preferred for energy calculations. The effort is being used to confirm recommendations for the ASTM test method C1421 on fracture toughness measurement.

Nomenclature

CMOD	crack mouth opening displacement
$K_{Ivb}(A)$	mode I fracture toughness per ASTM C1421
LPD	load point displacement
n	power-law slow crack growth parameter
PID	Proportional-Integral-Derivative feedback loop
P_{max}	maximum force at failure
SEPB	single edge precracked beam

Introduction

Fracture toughness is a critical structural design parameter and an excellent metric for ranking materials. It determines fracture strength in the presence of flaws, both inherent and induced, and defines the endpoint of the slow crack growth curve. For design of aerospace structures, quality measurements are required for exposures to environments ranging from high vacuum and low temperature (e.g., the International Space Station) to high humidity and high temperature (e.g., a Florida launch pad). Although an excellent standard on fracture toughness measurement of ceramics has been developed by ASTM (test method C1421), the range of effects necessary for some applications merit further investigation (Ref. 1). Particularly, measurements on glass-ceramics or glasses are desired. Common concerns include test humidity and test rate (Refs. 2 and 3). A secondary aspect of test rate is the control mode used.

Rate of force application and test environment have a strong effect on the strength (Ref. 4) and thus fracture toughness of ceramics and glasses. This is a result of several factors, including stress corrosion (Ref. 2) and energy stored and released from the test system. Brittle ceramics susceptible to environmental stress corrosion have also been found to exhibit rate-dependent fracture toughness (see Table I and accompanying paper in this volume). Stress corrosion can be mitigated via dry environments and rapid rates, leaving only stability effects as the prominent concern for fracture toughness measurements.

Stored energy release can be controlled by ensuring crack growth stability. Achieving this stability depends on competing variables such as a stiff load train and a sufficiently sensitive force transducer. In some cases, researchers have combined strain-controlled testing with high-capacity (low-resolution) force transducers to produce very stable results (Ref. 5). Because the measurements are small relative to transduce capacity (1 percent of capacity), metrology becomes a concern.

Procedure

In this work, the effects of control mode, test rate, and environment on the fracture toughness of AlliedSignal Ceramic Components AS800 silicon nitride were measured by using chevron-notched flexure specimens in accordance with ASTM test method C1421 (Ref. 1). The effects of humidity and test rate can be confounded, so to distinguish which factors influenced the results, strain control mode tests were employed over a wide range of rates (1 to 80 µm/m/s) in both lab air and nitrogen.

In addition to recording strain, crack mouth opening displacement (CMOD) was recorded using a laser micrometer, which measured the displacement between two parallel silicon carbide pins (see Figure 1) fixed to either side of the notch opening. Back face strain, which is linearly proportional to CMOD in the linear elastic region, was sufficient for confirming stable crack extension well past peak force. This was vital for comparison between lab air and dry nitrogen environment tests because containing the nitrogen prevented use of the laser during testing.

TABLE I.—FRACTURE TOUGHNESS OF AlSiMag 614 ALUMINA

Stroke rate, mm/min	Fracture toughness,[a] $K_{Ivb}(A)$, MPa√m		
	Test environment		
	Water	Air	Silicone oil or dry N_2
0.05	2.75 ± 0.01 (4)	3.19 ± 0.07 (7)	3.37 ± 0.05 (4)
0.01	2.64 ± 0.06 (3)	2.93 ± 0.10 (3)	3.39 ± 0.02 (2)

[a] Mean ± standard deviation (sample size).

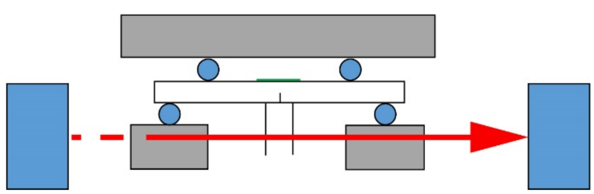


Figure 1.—Crack mouth opening displacement (CMOD) laser micrometer setup. Laser passes over two thin silicon carbide pins perpendicular to crack opening. Detector on the right side of test specimen measures distance between pins, outputting measurement at rate of about 100 Hz.

For ease of comparison, fracture toughness values were normalized to the fracture toughness measured at 0.05 mm/min in stroke control. This is a typical testing condition, making it ideal for comparison in terms of percent deviation between various testing conditions (see Table II and Figure 2) (Ref. 6).

Results

It was found that with a low-capacity force transducer, four-point flexure, and strain control, consistent results occur over a wide range of rates, particularly when environmental effects are minimized (see Table II and Figure 2). When environmental effects are present, the difference is less than 7 percent for an extremely wide range of strain rates. Also noteworthy is that silicon nitrides are sensitive to stress corrosion, as shown in Figure 3, as evidenced by a loss in strength with decreasing stress rate. The effect on the fracture toughness of AS800 is small (<3 percent variation) for reasonable experiment setups. The power-law slow crack growth parameter *n* for some nitrides is similar to that of silicate glass, implying a stress corrosion effect. Use of stroke control, which is less stable, best mitigates the effects occurring in laboratory air (see Table II and Figure 4) and gives very consistent results.

TABLE II.—FRACTURE TOUGHNESS OF AS800 SILICON NITRIDE[a]
[ASTM test method C1421 with chevron-notched beam specimens.]

Testing environment	Testing mode and rate (number of tests)	Fracture toughness,[b] $K_{Ivb}(A)$, MPa$\sqrt{m}$	Deviation from typical test (0.05 mm/min)	Nominal time to peak load, s
Laboratory air	Stroke control			
	0.005 mm/min (3)	7.87±0.05	+0.5%	600
	0.05 mm/min (15)	7.83±0.16	--------	100
	0.2 mm/min (3)	7.89±0.09	+0.7%	20
	0.5 mm/min (4)	8.04±0.04	+2.7%	10
	Strain control			
	0.1 µm/m/s (3)	7.45±0.15	–4.8%	3,300
	1 µm/m/s (10)	7.49±0.28	–4.4%	300
	10.25 µm/m/s (5)	7.60±0.22	–3.0%	50
	40 µm/m/s (3)	7.81±0.10	–0.03%	10
	80 µm/m/s (5)	7.98±0.13	+2.0%	5
	CMOD control[c]			
	0.04 µm/m/s (4)	7.66±0.45	–2.3%	50
Dry nitrogen	Strain control			
	1 µm/m/s (3)	7.81±0.18	–0.03%	300
	40 µm/m/s (3)	7.95±0.12	+1.0%	10
	80 µm/m/s (4)	7.94±0.59	+1.3%	5
All data	----	7.79±0.19	–0.05%	------

[a]AlliedSignal Inc.
[b]Values include standard deviation.
[c]CMOD is crack mouth opening displacement.

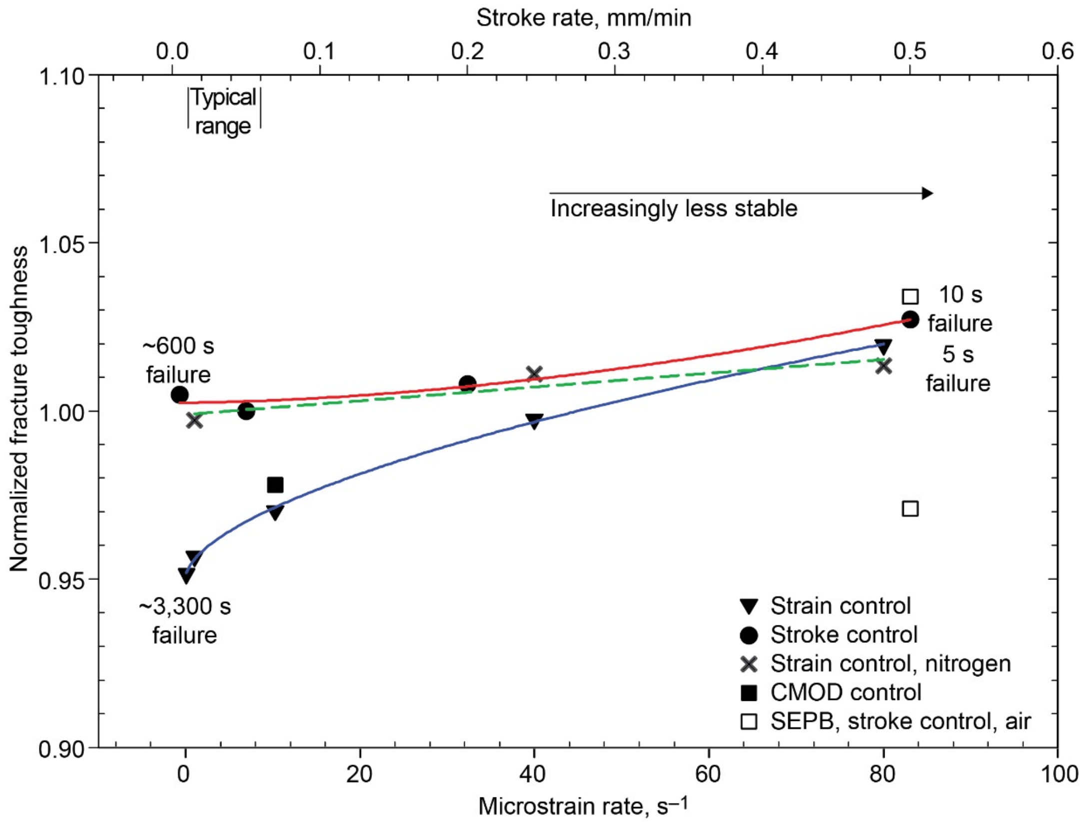
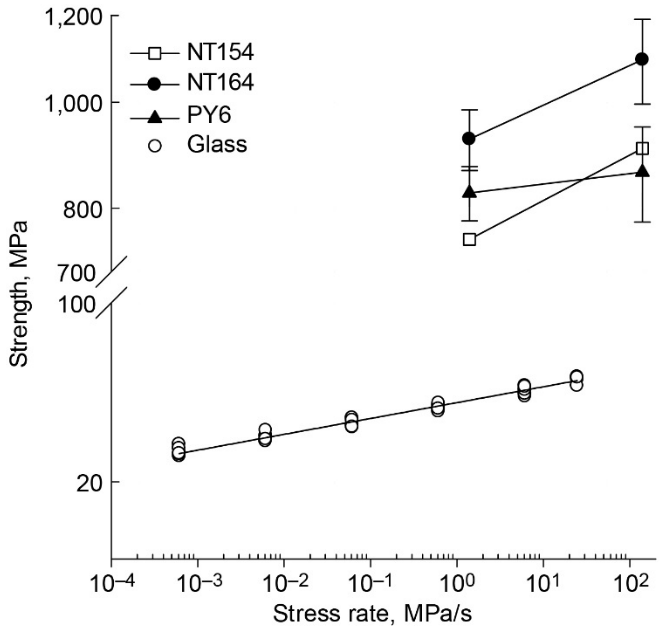


Figure 2.—Rate effects on fracture toughness of AS800 silicon nitride (AlliedSignal Inc.) tests performed in laboratory air at 50 percent relative humidity. Upper x-axis corresponds to stroke control, and lower axis corresponds to strain control. Crack mouth opening displacement (CMOD) control test rate has been converted to microstrain rate for direct comparison (see appendix). Plotted values are normalized against those of average test performed at 0.05 mm/min. Single-edge precracked beam (SEPB) data bounds adapted from References 6 and 7.



Material		Slow crack growth parameter, *n*
Silicon nitride	NT154	23
	NT164	27
	PY6	104
Silicate glass		21

Figure 3.—Constant stress rate curves for several silicon nitrides and glass. Adapted from References 8 and 9. Inset table gives power-law slow crack growth parameter *n* for several commercial silicon nitrides.

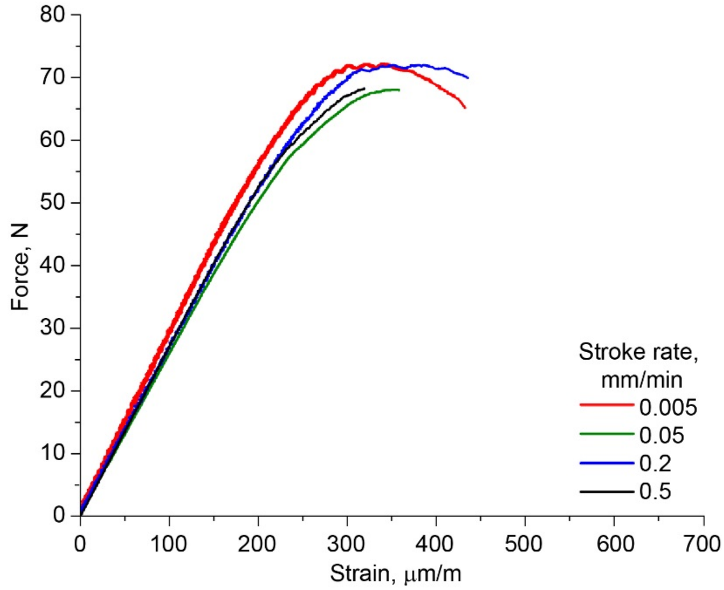


Figure 4.—Stroke rate testing representative curves for ASTM C1421 tests of AS800 silicon nitride (AlliedSignal Inc.) chevron-notched flexure specimens. Force as function of back face strain for stroke control.

Strain control tests in laboratory air show a significant variation in fracture toughness; however, when similar testing is done in dry nitrogen, this variation is significantly reduced (see Figure 5 and Figure 6, respectively). This implies a stress corrosion effect from humidity in the air as opposed to a rate effect from stored energy or other mechanisms. Test repeatability was confirmed by similar force-strain curves as well as sufficient unloading after peak force before failure of the sample. CMOD-controlled testing lacked the repeatability of strain control testing due to loop tuning difficulty (Figure 7). This is seen in the curve's unstable appearance beyond peak load. Further refinement of CMOD testing is left to future work.

Although CMOD control allowed for asymptotic unloading, the unstable appearance of the unloading curve indicates strain energy was wasted on cyclic unloading. The feedback loop had difficulty remaining closed. Similar repeatability issues were seen in high rate tests performed in dry nitrogen by using strain control. Strain control was stable until low force (10 percent of maximum), when it typically became unstable. It is suspected that highly concentrated stress fields on strain gages near failure result in this instability. Although it was difficult to control tests from the CMOD channel, CMOD data were recorded during a number of strain control tests. As long as stable crack growth was achieved, force plotted as a function of CMOD decays asymptotically, allowing for future energy calculations (see Figure 8).

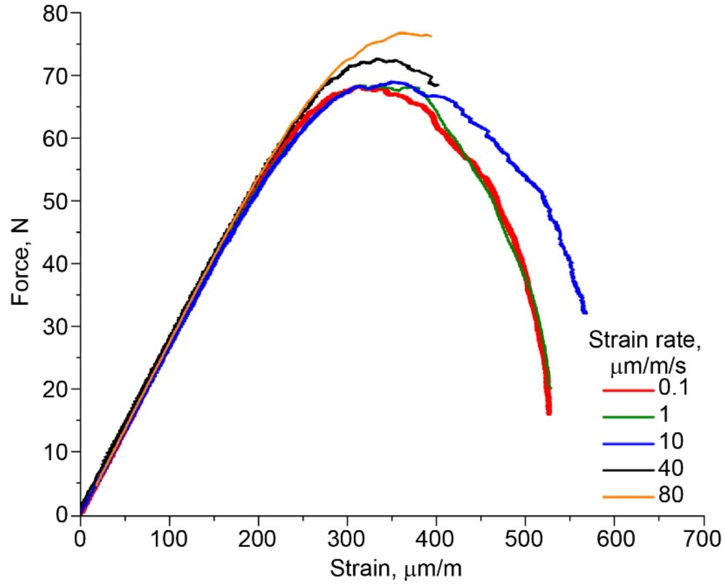


Figure 5.—Strain rate testing representative curves for ASTM C1421 tests of AS800 silicon nitride (AlliedSignal Inc.) chevron-notched flexure specimens. Force as function of back face strain, tested in strain control.

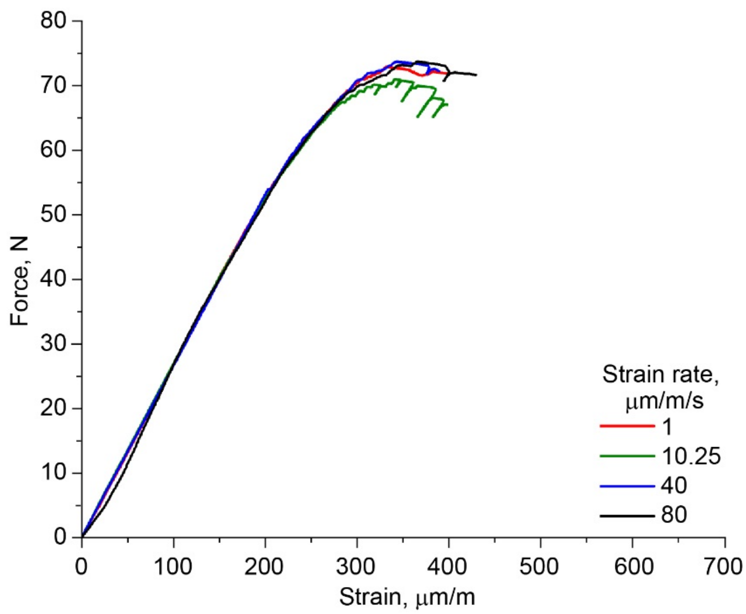


Figure 6.—Strain rate testing in ASTM C1421 tests of AS800 silicon nitride (AlliedSignal Inc.) chevron-notched flexure specimens in dry nitrogen. Force as function of back face strain, tested in strain control.

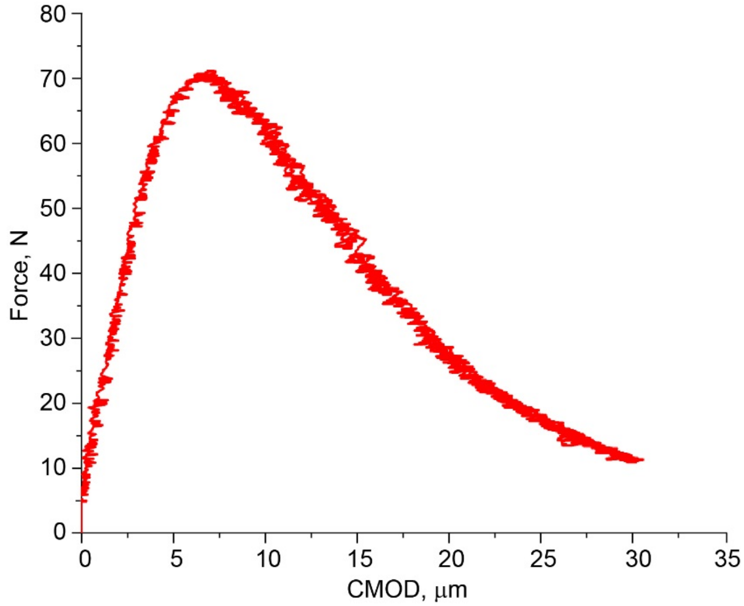


Figure 7.—Crack mouth opening displacement (CMOD) rate testing representative curve for ASTM C1421 tests of AS800 silicon nitride (AlliedSignal Inc.) chevron-notched flexure specimens. Force as function of CMOD, tested in CMOD control.

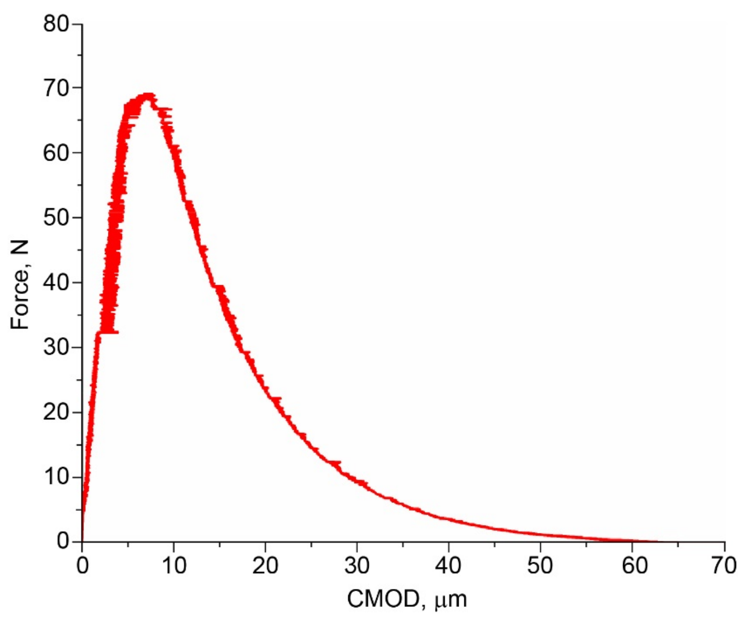


Figure 8.—Force as function of crack mouth opening displacement (CMOD) for strain control in ASTM C1421 tests of AS800 silicon nitride (AlliedSignal Inc.) chevron-notched flexure specimens.

Conclusions

The fracture toughness as measured in stroke control was relatively unaffected by rate variations. Stability beyond maximum force, however, was greatly reduced in this mode, leading to failure within 100 microstrain of peak load. Strain-controlled testing was able to achieve fully asymptotic failure in some cases, often reaching as low as 20 percent of peak force before breaking. Test specimens tested in strain control additionally exhibited stress corrosion behavior in a laboratory air environment, which was confirmed through testing in dry nitrogen. Laboratory air humidity during the test resulted in a fracture toughness reduction of about 5 percent for very slow test rates. For engineering purposes, tests performed within 10 to 15 s result in comparable fracture toughness regardless of test mode. A compromise must be found between test stability and environmental effects.

Future data processing involving calculation of work of fracture and strain energy release rate could be useful to directly compare control modes. Before this is possible, however, a conversion between load point displacement and crack mouth opening displacement must be obtained either experimentally or through finite element analysis. Future work will also include dynamic fatigue testing to confirm that the effect demonstrated in AS800 silicon nitride (AlliedSignal Inc.) is due to slow crack growth.

Appendix—Linear Correlation of CMOD and Back Face Strain

Crack mouth opening displacement (CMOD) and back face strain were linearly correlated using a strain gauge in tandem with a laser micrometer. By matching the strain channel to a predetermined waveform, a fit was generated that provided direct conversion between the two parameters (see Figure 9). Obtaining this data experimentally allowed for a conversion factor tailored to the exact billet of material as well as the exact load frame configuration, all without needing to resort to finite element analysis estimates. Similar methods can be employed to correlate load point displacement to CMOD, and thus to back face strain.

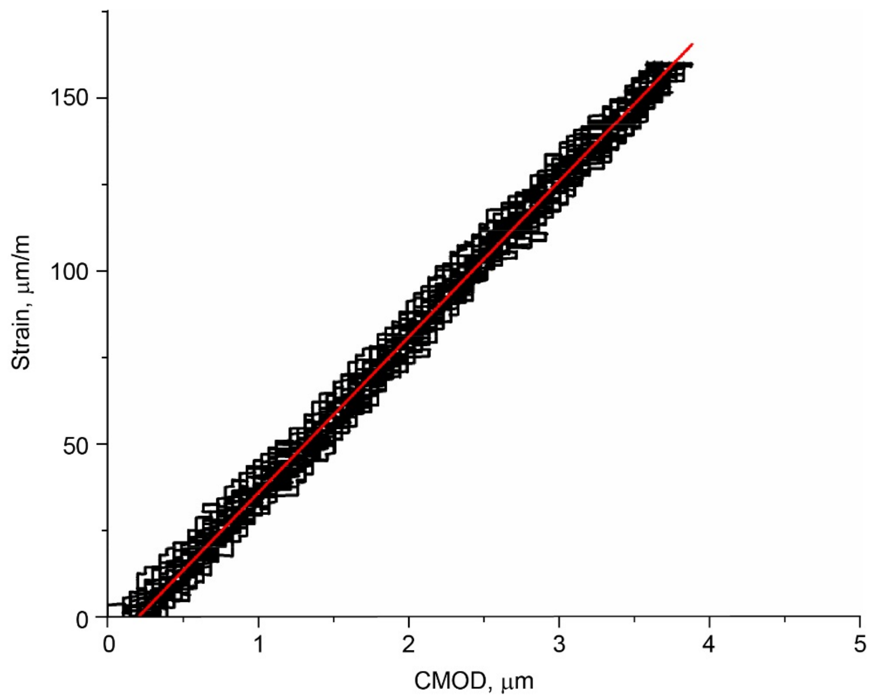


Figure 9.—Linear correlation between crack mouth opening displacement (CMOD) and back face strain for AS800 silicon nitride (AlliedSignal Inc.). The relationship was determined by overlaying several linear loading-unloading cycles and plotting a linear regression for the entire data set.

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