Relative Sliding Durability of Two Candidate High-Temperature Oxide Fiber Seal Materials

Christopher DellaCorte* and Bruce M. Steinetz†
NASA Lewis Research Center, Cleveland, Ohio 44135

A test program to determine the relative sliding durability of two candidate ceramic fibers for high-temperature sliding seal applications is described. Pin-on-disk tests were used to evaluate potential seal materials. Friction was measured during the tests, and fiber wear (indicated by the extent of fibers broken in a test bundle or yarn) was measured at the end of a test. In general, friction and wear increase with increased test temperature. The relative fiber durability correlates with tensile strength, indicating that tensile data, which is more readily available than sliding durability data, may be useful in predicting fiber wear behavior under various conditions. A dimensional analysis of the wear data shows that the fiber durability is related to a dimensionless parameter which represents the ratio of the fiber strength to the fiber stresses imposed by sliding.

Introduction

ADVANCED propulsion systems, such as the new generation of launch vehicles and especially hypersonic vehicles such as NASP, will push engine material and system requirements to higher temperatures.[1] New materials, like metal and ceramic matrix composites, are currently under development for these applications.[2] In addition, new technologies and approaches for control surface bearings and seals will be needed.

Several proposed hypersonic vehicles will, for example, require sliding seals to operate in adverse environments encountered in engines and on control surfaces such as flaps and elevons. It has been estimated that linear sliding seals will be needed to seal the airframe structure from the heat of combustion in the engine and the frictional heat of the airstream, and may need to operate at temperatures near 1000°C.[3] Since no current technology can meet these types of challenges, development programs are currently underway to develop suitable seals.

One current approach is to use a braided linear rope seal in sliding contact with a cooled metallic counterface.[4] The rope seal is woven from ceramic fibers and must withstand oxidative and reducing environments, which may contain water vapor, at temperatures to about 1000°C. Figure 1 shows, schematically, one such rope seal design.

For long seal life and low seal actuation forces, friction and wear of the rope must be minimized. Recent research suggests that, in sliding contacts, unlubricated ceramics exhibit high friction and wear.[5] Therefore, the determination and understanding of high-temperature friction and wear properties of candidate seal materials are important issues.

In this article, two candidate ceramic fiber materials based on the Al_2O_3-SiO_2-B_2O_3 system, Nextel 312 and Nextel 440, are compared by sliding them against Inconel 718 counterface disks at temperatures from 25 to 900°C under simulated seal conditions. The goal of this article is twofold. One goal is to
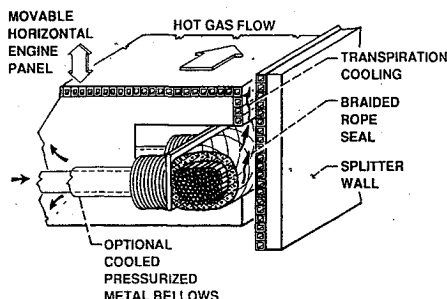
Fig. 1 Cross section of proposed engine seal.

compare the relative durability of these two ceramic candidate materials and assess their potential for seal use. The second goal is to develop a convenient screening technique to evaluate other potential fiber materials.

Apparatus and Procedures

Since many new fibers are available in limited quantities, it is advantageous to have a screening technique that requires a minimum of material and minimal specimen preparation, yet simulates the anticipated seal application. The pin-on-disk test satisfies these needs. To conduct a test, a bundle of the fiber material to be tested, which typically contains ≈6000 fibers, ≈11 μm in diameter, is wrapped over the end of a hemispherically tipped pin, then loaded against a rotating counterface disk surface. Friction is continuously monitored during the test, and fiber wear—which is determined by the number of fibers that break during sliding—is measured after testing.

This testing approach is a variation of the author's work done previously on woven fabric materials.[6] In that work, samples of woven ceramic fabric to be tested were draped over the end of a hemispherically tipped pin and rubbed against metal disks. The approach was successful, but required a substantial amount of woven fabric for each sample.

To accommodate for the available fiber materials, a short bundle of fibers is draped over the end of a specially machined Inconel 718 pin and held in place with loops of circumferentially wound stainless steel wire (Fig. 2). The pin has grooves machined into the tip and shank to accept the bundle and prevent its slipping off during testing. The pin is then slid against a counterface disk in a high-temperature pin on disk tribometer. The disk is 63.5 mm in diameter and 12.7-mm thick. The pin generates a 51-mm wear track on the face of the disk.
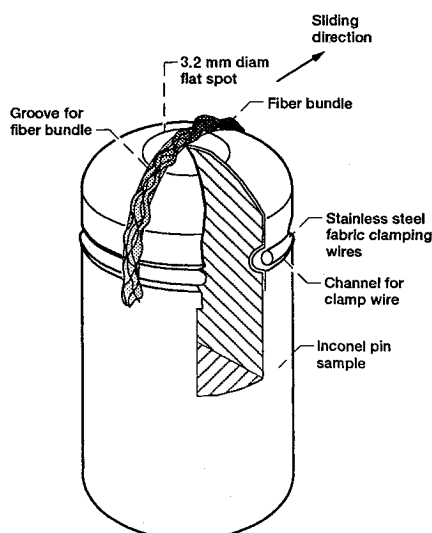
Fig. 2 Pin test specimen.

Fig. 3 SEM photomicrograph of fiber-pin specimen prior to testing. Sliding direction is from left to right.
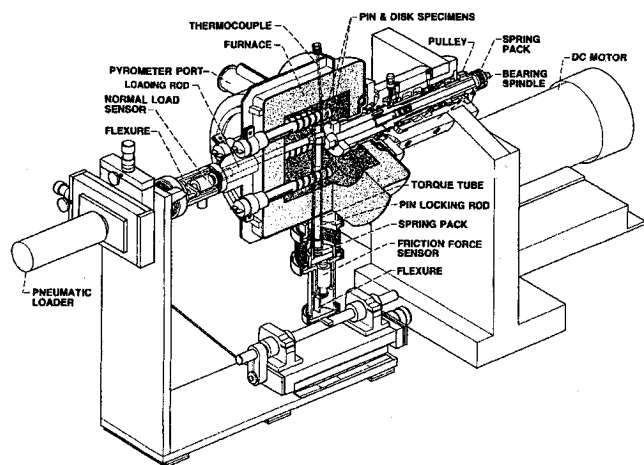

Fig. 4 High-temperature pin on disk tribometer.

The primary objective of these bundle tests is to simulate the contact conditions and wear phenomena which occur during braided seal testing. As in the previous work with the woven fabric,[6] the pin has a small flat spot at its tip, 3.2 mm in diameter, to better support the fiber bundle and provide a uniform sliding area. The bundle is a given a one-half or 180-deg twist across the flat contact spot to help contain the bundle in the sliding contact and to orient the fibers at approximately a 45-deg angle with the sliding direction better simulating proposed braided seal configurations (Fig. 3). Wear

mechanism(s) observed during preliminary bundle tests are very similar to the wear behavior reported on woven, braided rope seals.[3] Therefore, although the bundle test does not provide completely quantified contact conditions (i.e., known real contact area, extent of elastic deformation of the fibers, etc.) it is a suitable simulation of the intended woven seal application and provides a convenient relative measure of fiber wear behavior.

Figure 4 shows a schematic diagram of the tribometer used in this work. The tribometer has been described in detail elsewhere,[7] and its main features will only be briefly described here. The tribometer is capable of sliding pin on disk specimens at loads from 0.1 to 100 kg at sliding speeds from 0.025 to 22 m/s. The specimens can be heated using a SiC glowbar furnace to temperatures of 1200°C. For the tests conducted here, the sliding speed was 0.025 m/s and the load was 0.270 kg, giving a nominal contact stress of 340 kPa. No attempt was made to measure or calculate the real contact area or contact stress. Because the fiber bundle/disk interface is very complex, this type of quantification is beyond the scope of this article. It should be reiterated however, that the twisted bundle/disk interface closely simulates the woven rope seal/counterface wall interface.

The test atmosphere was ambient air with a relative humidity of 60 ± 10% at 25°C. Test temperature was 25, 500, and 900°C. The test duration was 120 min. Friction was continuously measured during the tests. Wear was qualitatively monitored during the tests using a linear variable differential transformer (LVDT) position sensor on the pin specimen, and more exactly quantified after each test. By monitoring pin movement during the test, excessive fiber breakage can be detected and sliding can be suspended prior to the end of the typical 2-h test period. Preliminary tests indicated that fiber wear occurs in a fairly uniform manner during the tests. Fiber wear data, within an uncertainty of about 5%, was determined using SEM micrographs and from post-test visual observations of the worn bundles.

Materials

The materials tested in this program consist of two candidate fiber materials with two different thermal treatments and one disk counterface material. The fibers tested were 3M's Nextel 312 and Nextel 440. The fiber composition and mechanical properties are given in detail in Table 1. The fibers are made from alumina, boria, and silica. Nextel 312 contains more of the glass former, boria, at 14 wt%, than Nextel 440, which contains only 2 wt% boria. Nextel 312, therefore, is more amorphous and glasslike and also has, in general, lower tensile strength than Nextel 440.

Table 1 Composition and physical properties of candidate ceramic seal fiber materials[a]

Property	Nextel 312	Nextel 440
Fiber composition, wt%	62 Al_2O_3	70 Al_2O_3
	24 SiO_2	28 SiO_2
	14 B_2O_3	2 B_2O_3
Fiber diameter, μm	10–12	10–12
Density, g/cc	2.7 to 2.9	3.0
Filament tensile strength (5-mm gauge), MPa	1700	2000
Filament tensile modulus, GPa	150	190
Surface area, m²/g	<0.2	<0.2

[a]Taken from Ref. 10.

Table 2 Composition and hardness of inconel test disk specimens

Property	Valve
Composition, wt %	70 Ni, 16 Cr, 7.5 Fe, 2.5 Ti, 1 Al 1 Co, 1 Mn, 0.1 C and 0.9 other
Hardness, Rockwell C	RC34

The fibers tested were slightly oval in cross section with diameters of about 11 μm. All of the samples were heat cleaned at 500°C in air for 1 h to remove an organic sizing compound used by the manufacturer during processing.[8] Furthermore, some samples which are designated with an HT suffix (e.g., Nextel 440HT) were heat treated in air at 950°C for 12 h to make the fibers more resistant to moisture degradation at temperature. In general, this heat treatment slightly lowers the tensile strength of the fibers.[9]

The test disks were made of Inconel 718, a precipitation-hardened nickel-chromium alloy. Table 2 gives the composition and hardness of the disk material. This material is being considered as a possible candidate for seal applications because of its high-temperature strength and oxidation resistance. Prior to testing, the disk surface is lapped with alumina abrasive to a surface finish of about 0.1 μm rms. After lapping, the specimens are cleaned with freon, ethyl alcohol, scrubbed with a paste of levigated alumina and deionized water, rinsed with deionized water, and air dried.

Results

The friction and wear results for the tests conducted are given in Table 3. Wear is characterized by the durability (the inverse of wear) and is given by cycles to failure (CTF) which is a common durability parameter for fiber materials.[10] For these tests, the CTF is determined by first dividing the number of cycles tested by the number of fibers broken during the test, and then multiplying by the total number of fibers in the bundle tested (in our case there are 6000 fibers). For example, if 50% of the fibers in a bundle (3000 fibers) break after a 2-h test (1200 revolutions or cycles) the CTF is $(1200) \cdot (6000/3000)$ = 2400. That is to say, it would take 2400 disk revolutions or cycles to break through the entire 6000-fiber bundle.

This measurement assumes that the fiber breakage ratio is linear with time, and is therefore, simplistic. However, CTF is a standard comparative measure for fiber durability in the textile industry and proves to be a useful measurement for these tests.

Table 3 Friction and wear (CTF) data summary

Test temperature	Material	μ	CTF, cycles
25°C	Nextel 440	0.49 ± 0.09	7366 ± 2950
	Nextel 440 HT	0.54 ± 0.07	6205 ± 1550
	Nextel 312	0.59 ± 0.10	4000 ± 1455
	Nextel 312 HT	0.53 ± 0.05	3400 ± 750
500°C	Nextel 440	0.65 ± 0.13	8677 ± 3250
	Nextel 440 HT	0.80 ± 0.08	6510 ± 1028
	Nextel 312	0.68 ± 0.09	223 ± 80
	Nextel 312 HT	0.66 ± 0.12	118 ± 60
900°C	Nextel 440	0.66 ± 0.11	3181 ± 800
	Nextel 440 HT	0.64 ± 0.10	281 ± 60
	Nextel 312	0.74 ± 0.13	162 ± 50
	Nextel 312 HT	0.76 ± 0.13	100 ± 10

Note: Test atmosphere, air, R.H. 50–75%; counterface: Inconel 718; test velocity, 0.025m/s; test load, 0.27 kg. HT designates thermal treatment at 950°C for 12 h to improve humidity resistance at high temperatures.
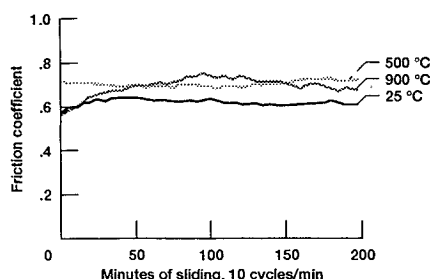

Fig. 5 Friction coefficient vs sliding time for Nextel 440. Test conditions: 0.27 kg (2.65 N) load, Inconel 718 counterface disk, air atmosphere, 2.5 cm/s sliding velocity.

Preliminary sliding tests at short intervals (e.g., 150 cycles) indicated that the fiber wear (breakage rate) rate was approximately linear over the entire life of the bundle. This implies that little or no load sharing occurs between fibers at the sliding interface and those beneath them in the bundle. Therefore, interfiber friction and wear effects are considered small and the wear seems to be dominated by phenomena at the sliding interface.

The friction data for the four test specimen combinations evaluated are given in Table 3, and a typical friction plot is given in Fig. 5. Each data curve in this plot represents the average of at least three experiments. The data scatter values are shown in Table 3. From the data, it can be seen that the friction coefficients at 500 and 900°C are higher than at room temperature. Although there is a slight decrease in friction from 500 to 900°C, this variation is well within data scatter.

Discussion

Considering the CTF data, a ranking of the durability of the fibers can clearly be seen. The most durable fiber is the Nextel 440 followed by the heat-treated 440, Nextel 312, and finally, Nextel 312 in the heat-treated condition. This ranking holds true for all the temperatures tested. To better understand the reason for this ranking, it is instructive to first examine the surface features of the worn fiber specimens and then combine those observations with the fibers' mechanical and physical properties.

Morphological Aspects

Much can be learned about tribological tests by examining the wear surfaces as well as the numerical data values. Figures 6–8 show SEM photomicrographs of fiber bundles after sliding against the counterface disk. One apparent aspect is that the fiber bundles are wearing by brittle fracture and not by abrasion. This finding is consistent with work done previ-

Fig. 6 Nextel 440 fiber-pin specimen after sliding for 24 h at 25°C. Very few fibers are broken, indicating good durability.
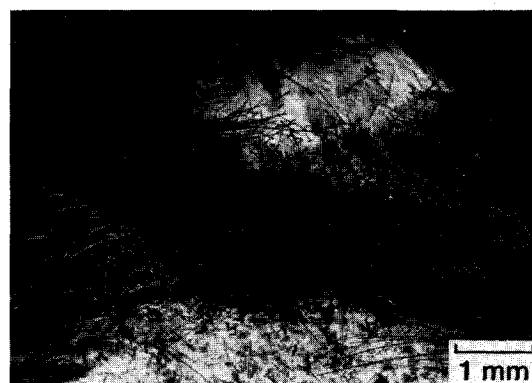

Fig. 7 Nextel 440 HT fiber-pin specimens after testing at 900°C. Virtually all of the fibers were broken during the test, indicating low durability.
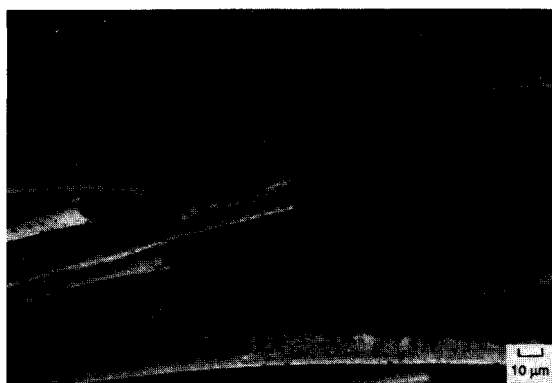
Fig. 8 SEM photomicrograph: Fractured fiber surfaces after sliding at 900°C.

Table 4 Disk surface roughness after oxidation/testing at elevated temperatures

Test temperature	R_A, μm
25°C	0.036 ± 0.003
500°C	0.047 ± 0.04
900°C	0.120 ± 0.02

Table 5 Filament tensile strength

Fiber material	Tensile strength, MPa		
	25°C	500°C	900°C
Nextel 440[a]	2000[a]	1750[a]	1500[a]
Nextel 440HT	1500[a]	1300[b]	1125[b]
Nextel 312[a]	1700[a]	1400[a]	750[a]
Nextel 312HT	1250[a]	1029[b]	551[b]

[a]Data from Ref. 10.
[b]Strength reduction at elevated temperatures estimated from strength reduction of nonheat treated fibers, as recommended by fiber manufacturer.

ously.[6] Also noted, is that little debris, transferred disk material, or reaction products seem to be present. This is coincident with the fact that the Nextel 312 and 440 fibers tested are oxide ceramics which would not normally react in an air environment at these modest test temperatures.

Surface profilometry of the disk surfaces before and after testing indicates that, due to surface oxidation, the disk surface is rougher after elevated temperature runs than after room temperature runs. Although the arithmetic roughness parameter R_a increases threefold (Table 4), the surfaces are still fairly smooth and don't seem to have an effect on the fiber durability. Further tests are required to determine the level of roughness which will cause an increase in fiber breakage.

Table 5 shows the tensile strength as a function of temperature for the four fiber conditions tested. From the data in the Tables 3 and 5, it can be seen that the ranking of the fibers in terms of tensile strength is the same as the ranking of the fibers in terms of durability (CTF). Although the correlation of the rankings of the fiber durability and tensile strength is good, a mathematical or functional relationship between CTF and tensile strength is not immediately apparent. To understand the factors affecting the fiber durability, a dimensional analysis of the fiber durability and the factors affecting the CTF was conducted.

Dimensional Analysis

A power product method of dimensional analysis was utilized to determine potentially meaningful dimensionless parameters and a possible relationship between fiber durability and tensile strength. This technique consists of setting an arbitrary relationship between the desired measurand, the

CTF, and the probable factors affecting it. In our case the durability (CTF) is likely to be affected by fiber tensile strength TS, fiber cross-sectional area A, friction coefficient μ, and load F_n, temperature t, and counterface surface roughness R_a. Since the fiber wear is occurring by brittle fracture, fiber fracture toughness may also be an important factor. However, measurements of this parameter, which is usually based upon tests of macroscopic specimens with known flaw sizes, are unavailable for these microscale fibers. Therefore, for simplicity, fracture toughness and other macroscale factors, such as hardness, are not included in the following simplified analysis.

Mathematically

$$CTF = f(TS, A, \mu \cdot F_n, t, R_a)$$

Since temperature directly affects the fiber tensile strength and the load is constant, we can eliminate temperature and combine the friction coefficient and the load force into one variable, the friction force F_f.

The function f, which is related to the CTF, can be written as a power product relation as follows:

$$f = K \cdot (TS)^a \cdot (A)^b \cdot (F_f)^c \cdot (R_a)^d$$

where K is an arbitrary constant or a function of other variables held constant in these tests (e.g., total number of fibers).

To determine the values of the powers $a-d$, a dimensional analysis is used, equating the units in each parameter to match the nondimensional nature of the CTF. Substituting for the units contained in each parameter and separating for each unit gives

$$\{0\} = 1 \cdot \{Fm^{-2}\}^a \cdot \{m^2\}^b \cdot \{F\}^c \cdot \{m\}^d$$

for force

$$F: 0 = a + c$$

for length

$$m: 0 = -2a + 2b + d$$

Since there are two equations and four unknowns, the results will be somewhat arbitrary, but the assumptions made can be verified by the data and may prove to be useful.

If we let

$$a = 1 \quad \text{then} \quad c = -1$$

Then using the equation for length

$$m: 2 = 2b + d$$

If it is assumed that the surface roughness effect is small for these tests (i.e., $d \approx 0$) then $b = 1$, and the following relation is found:

$$f = K \cdot (TS \cdot A)/(F_f)$$

$$CTF = f[K, (TS \cdot A)/F_f]$$

If we then substitute the fiber diameter squared D^2 for the area, incorporating the $\pi/4$ term in the constant, and expand the friction force back to the product of the friction coefficient and the load, the relation is as follows:

$$CTF = f[K, (TS \cdot D^2)/(\mu \cdot F_N)] = f(K, DR)$$

This relationship gives the fiber durability in terms of a constant K and the durability ratio DR, which incorporates fiber and test condition parameters. The numerator of the

durability ratio is essentially a measure of the fiber breakage strength, and the denominator is a measure of the applied friction force which acts to break the fibers.

To assure the validity of the assumption to neglect the surface roughness, fiber durability tests were conducted at various roughnesses where all other parameters are held constant. Results from these tests are shown in Table 6. Clearly, over the range of surface roughnesses tested, the fiber durability is not greatly affected. This may not be the case for different fibers or slightly more roughened surfaces. More research is underway to determine the limits of the assumption to neglect surface roughness. For the tests discussed here, the assumption to neglect roughness in the CTF relation is justified.

The relationship established by the dimensional analysis and the identification of an important dimensionless parameter helps to understand the data. The usefulness of the analysis and the dimensionless durability ratio can be seen by examining a plot of fiber durability (CTF) vs the durability ratio $(TS \cdot D^2/\mu F_n)$ as shown in Fig. 9. Though the data are not comprehensive, a trend is apparent. This trend indicates that rather than a simple linear function, the CTF seems to be a step function of fiber strength to fiber breakage forces. Many others have recognized and reported the importance of fracture behavior on ceramic wear.[11,12]

The CTF is low for $(TS \cdot D^2/\mu F_n)$ ratios lower than about 0.12. Above this point, the CTF rises dramatically. This implies that a threshold exists around a ratio of 0.12, below which poor fiber durability is exhibited, and above which good fiber durability is observed. Much of the data scatter in Fig. 9 may be the result of uncertainties in the tensile strength data, which in some cases was extrapolated from data at other temperatures and also friction data scatter. Nonetheless, the dimensional analysis helps to delineate the important variables and aids in data interpretation.

For example, the analysis helps us understand why the fibers with higher tensile strength exhibit more durability. Also, the gain in strength must be tempered by a possible increase in friction or load which cause the frictional stresses on the fibers to increase. This indicates that durability can be en-

hanced by reducing the loads or by lubricating the fibers. Both of the options are available to seal designers and have been shown to be effective in previous research with these materials in woven fabric form.[6,8]

Although the data scatter is large, Fig. 9 can be used to determine the approximate value of the threshold separating durability behavior for this materials system. It must be pointed out, however, that this value is only crudely approximate. More data on other fiber materials need to be acquired to ensure better accuracy. Also, this analysis is simplistic and is only based upon mechanics and data taken where physical properties seem to dominate chemical ones. For other material systems, chemical reactivity with the environment and other materials in the environment may have a significant impact on durability.

Conclusions

The relative durability of two fiber materials in two conditions were evaluated using a pin-on-disk tribometer. The developed test technique provided repeatable data in a convenient manner using a minimal amount of test material. The test results and observations were consistent with the brittle behavior of ceramic materials in sliding contact and corroborated research done previously with these ceramic fiber materials in woven fabric form. Based upon these tests the following specific conclusions can be drawn:

1) In terms of fiber durability, Nextel 440, without the heat treatment, is the most durable fiber material.

2) In these tests where the friction coefficients were comparable, the fiber durability ranking correlates well with the tensile strength ranking, indicating that tensile data may provide insight for seal material selection and screening.

3) Over the limited counterface roughness range studied here, surface roughness had no measurable effect on fiber durability.

4) Fiber durability decreased and friction increased with temperature. This may be due to a variety of factors including lower fiber strength and possible changes in the chemistry of the fiber/counterface sliding contact.

5) A simple model developed from a dimensional analysis indicates that fiber durability is related to a durability ratio of the fiber strength to the frictional stresses imposed on the fibers due to sliding.

Acknowledgments

Table 6   Fiber durability of Nextel 440HT as a function of disk surface roughness at constant temperature, 25°C

R_A, μm	CTF
0.036 ± 0.003[a]	6205 ± 1550
0.120 ± 0.020[b]	7000 ± 1550
0.680 ± 0.020[c]	6000 ± 1200

[a]Lapped surface.
[b]Oxidized in air at 900°C for 6 h.
[c]Diamond ground with 150-grit wheel. Data are averaged values from at least three tests. Uncertainties represent data scatter.

Fig. 9   CTF vs durability ratio $TSD^2/\mu F_N$, resembles step function with discontinuity or transition occurring between 0.075 and 0.15.

References

[1]Jaffe, R. I., Perkins, R. A., and Pound, G. M., "Hypersonic Vehicles-Performance," *High-Temperature Oxidation-Resistant Coatings*, National Academy of Sciences, Washington, DC, 1970, pp. 141–158.

[2]Herbell, T. P., and Eckel, A. J., "Ceramic Matrix Composites for Rocket Engine Turbine Applications," American Society for Mechanical Engineers Paper 92-GT-394, June 1992.

[3]Steinetz, B. M., Mutharasan, R., Du, G.-W., Miller, J. H., and Ko, F., "Engine Panel Seals for Hypersonic Engine Applications: High Temperature Leakage Assessments and Flow Modeling," NASA TM-105260, April 1992.

[4]Steinetz, B. M., DellaCorte, C., and Tong, M., "Seal Concept and Material Performance Evaluation for the NASP Engine," *Proceedings of the Seventh NASP Technology Symposium*, Paper 60, Cleveland, OH, Oct. 1989, pp. 39–58.

[5]Yust, C. S., and Allard, L. F., "Wear Characteristics of an Alumina Silicon Carbide Whisker Composite at Temperatures to 800°C in Air," *Tribology Transactions*, Vol. 32, July 1989, pp. 331–338.

[6]DellaCorte, C., "Tribological Properties of Alumina-Boria-Silicate Fabric from 25°C to 850°C," *Tribology Transactions*, Vol. 32, July 1989, pp. 325–330.

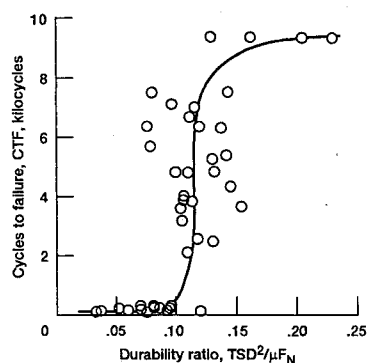[7]Sliney, H. E., and DellaCorte, C., "A New Test Machine for

Measuring Friction and Wear in Controlled Atmospheres to 1200°C," *Lubrication Engineering*, Vol. 47, No. 4, 1991, pp. 314–319.

[8]Holtz, A. R., and Grether, M. F., "High Temperature Properties of Three Nextel Ceramic Fibers," *Advanced Materials Technology 87: Proceedings of the 32nd International SAMPE Symposium and Exhibition*, edited by R. Carson et al., Corvina, CA, 1987, pp. 245–256.

[9]DellaCorte, C., Steinetz, B. M., and Brindley, P. K., "Tribological Properties of Ceramic/Ti₃Al-Nb Sliding Couples for Use as Candidate Seal Materials to 700°C," NASA TM-102401, April 1990.

[10]Miller, B., Friedman, H. L., and Turner, R., "Design and Use of a Cyclic Tensile Abrader for Filaments and Yarns—A Study of Polyester Monofilament Wear," *Textile Research Journal*, Vol. 53, No. 12, 1983, pp. 733–740.

[11]Sliney, H. E., and Deadmore, D. L., "Friction and Wear of Oxide-Ceramic Sliding Against IN-718 Nickel Base Alloy at 25 to 800°C in Atmospheric Air," NASA TM-102291, Aug. 1989.

[12]Kear, B. H., *Tribology of Ceramics*, National Academy Press, 1988, pp. 73–85; see also NASA CR-182506, Washington, DC, June 1988.