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Sliding Durability of Two Carbide-Oxide Candidate High Temperature Fiber Seal Materials in Air to 900 °C

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SLIDING DURABILITY OF TWO CARBIDE-OXIDE CANDIDATE HIGH TEMPERATURE

FIBER SEAL MATERIALS IN AIR TO 900 °C

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SUMMARY

A test program to determine the friction and wear properties of two complex carbide-oxide ceramic fibers for high temperature sliding seal applications is described. The fibers are based on Si, C, O and Ti or Si, C, N and O ceramic systems. Pin-on-disk tests using ceramic fiber covered pins and Inconel 718 disks, were conducted in air from 25 to 900 °C to evaluate potential seal materials. This testing procedure was used in a previous study of oxide ceramic fibers which were found to exhibit wear behavior based predominantly upon their mechanical properties.

Like the oxide fibers tested previously, these carbide-oxide ceramic fibers, show an increase in friction and wear with increased test temperature. At room temperature, the wear behavior seems to be based upon mechanical properties, namely tensile strength. At 500 and especially 900 °C, the fibers wear by both mechanical fracture and by oxidative type wear. Based upon post test microscopic and x-ray analyses, interaction between the fiber constituents and elements transferred from the counterface, namely nickel and chromium, may have occurred enhancing the tribochemical wear process. These results indicate that these carbide-oxide fibers may not be suitable materials for high temperature sliding seals which encounter oxygen containing environments.

INTRODUCTION

New technology has recently emerged to address high temperature sliding seal needs for advanced propulsion systems. Some examples are engine sidewall and airframe seals for hypersonic vehicles such as the National AeroSpace Plane (NASP) (ref. 1 and fig. 1). These new seal designs are based upon the ability to use compliant ceramic monoliths or woven ceramic fiber ropes in sliding against counterfaces such as cooled copper, superalloys and other ceramics. In these applications, the seal materials may have to endure sliding contact over a wide temperature range. Therefore the tribology, or friction and wear behavior, of these materials is a critical research area.

The authors have studied the friction and wear of ceramic fiber candidate seal materials previously. The studies included sliding tests of woven fabric materials (refs. 2 and 3) and individual fiber strands or bundles using a pin-on-disk tribometer (ref. 4). The results of reference 4 suggest that the tribology of oxide ceramic fibers in an air environment is dominated by fiber physical properties such as tensile strength and tribological test parameters such as the applied load during sliding. With oxide fibers, chemistry only played a role when the ceramic fibers were in direct contact with reactive lubricant materials at elevated temperatures (ref. 2).

The present study considers two complex oxide-carbide ceramic fibers tested in an air environment at temperatures to 900 °C. Under static conditions the fibers are protected from the atmosphere by passivating layers of SiO₂ (ref. 5). Because sliding may remove this passivating layer allowing atmospheric attack, namely oxidation, to occur, the sliding durability of these carbide-oxide ceramic fibers must be ascertained under simulated seal conditions to determine their suitability as candidate seal materials.

The following test program uses pin-on-disk tests to determine the friction and wear properties of two silicon based fibers, Tyranno (ref. 6), a Si, C, Ti, O ceramic fiber and HPZ (ref. 7), a Si, C, N, O ceramic fiber. Both of these fibers exhibit excellent high temperature strength properties and are promising candidates for seal applications. Following the sliding tests, SEM analyses were conducted to determine the wear mode(s) of the fibers. The test procedure and apparatus have been completely described in previous papers (refs. 4 and 8) and will only be briefly described in this paper.

Material Description and Preparation

The materials tested in this test program consist of two candidate fiber materials and one disk counterface material. The fibers tested were Dow Corning's HPZ and Textron/UBE Industry's Tyranno ceramic fibers. The fiber compositions and mechanical properties are given in table I.

The ceramic fiber HPZ is an amorphous combination of Si, N, C and O. HPZ is a textile grade inorganic silicon-carbonitride type fiber prepared from hydropolysilazane polymer by a pyrolysis process. The HPZ fibers have a slightly oval cross section with an average diameter of about 12 μm (ref. 6).

The Tyranno fibers are an amorphous combination of Si, C, O and Ti. They are prepared by pyrolysis of the polymer precursor polytitanocarbosilane (PTC) in the temperature range of 700 to 1500 $^{\circ}\text{C}$. Heat treating the cured Tyranno fiber above 1400 $^{\circ}\text{C}$ can produce micro-crystalline (crystallite sizes ≈ 1 nm) phases of β -SiC (ref. 7). Since the work presented here limits the temperature exposure to 900 $^{\circ}\text{C}$ the fibers remain amorphous. The Tyranno fibers have a round cross section and are 11 μm in diameter.

Prior to testing, the Tyranno and the HPZ fibers, were heat cleaned at 500 $^{\circ}\text{C}$, in air for 1 hr to remove an organic sizing compound (PolyVinylAlcohol, PVA) used by the manufacturers during processing (refs. 6 and 7).

The test disks were made of Inconel 718, a nickel-chromium superalloy precipitation hardened to 34 on the Rockwell C scale. Table II gives the composition and some representative properties 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 levitated alumina and deionized water, rinsed with deionized water and air dried.

Apparatus and Procedures

A pin-on-disk test configuration is used to determine the friction and wear properties of the fibers. To conduct a test, a bundle of the fiber material to be tested is wrapped over the tip of a hemispherically tipped pin then loaded against a rotating counterface disk surface. The bundle resembles a piece of string or yarn about 2 mm in diameter and typically contains ≈ 6000 , 10 to 12 μm diameter fibers. 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 technique uses a minimum amount of fiber material to assess durability under simulated seal conditions of sliding speed, atmosphere and contact pressures.

To prepare a fiber specimen, a bundle of fibers is draped over the tip 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 fiber strand and prevent its slipping off during testing. 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 given a one half or 180° twist across the flat contact spot to help contain the bundle in the sliding contact and to orient the fibers at approximately a 45° angle with the sliding direction to better simulate proposed braided seal configurations (fig. 3).

To test a fiber candidate, the pin is 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 disk.

For the tests conducted here, the sliding speed was 0.025 m/s and the load was 0.270 Kg. The test atmosphere was ambient air with a relative humidity ranging from 50 to 75 percent at 25 °C. Test temperature was 25, 500, and 900 °C. The test duration was 120 min. At least three repeat tests were performed for each test condition and material combination.

Fiber wear data, within an uncertainty of about 5 percent, was determined using SEM micrographs and from visual observations. In addition, SEM analyses coupled with Energy Dispersive X-Ray Spectroscopy (EDS) is used to characterize and understand the fiber wear behavior. Disk wear, in general, was not measured because previous experience with testing of this type indicated that it is too small to quantify. Also, for the seal applications anticipated, wear of the fibers is much more critical than the mild counterface wear which occurs during sliding. Therefore, only fiber wear and friction will be considered.

RESULTS

Tribotesting

The friction and wear results for the tests conducted are given in table III. Wear is given by cycles to failure, CTF which is a common wear parameter for fiber materials (ref. 10). For these tests, the CTF is determined by first multiplying the number of cycles tested by the total numbers of fibers in the bundle tested (in our case 6000 fibers) and then dividing by the number of fibers broken during the test. For example, if 6000 fibers are tested for 1200 cycles and 50 percent of the fibers in the bundle (3000 fibers) break after the test, then 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 rate is linear with time and is therefore, simplistic. However, CTF is a standard measure for fiber durability in the textile industry and proves to be a useful measurement for these tests.

The friction behavior of the two fibers at the three test temperatures is plotted in figure 4 and the fiber wear data, the CTF, is plotted in figure 5. Each data point represents the average of at least three repeat tests with new specimens used for each test. The error bars shown in figure 4 represent data scatter. For HPZ, the friction coefficient is relatively constant over the entire test temperature range and the CTF is also fairly constant. For the Tyranno, however, the friction and wear increase dramatically with test temperature.

Wear specimen analysis. - The wear surfaces, namely the fiber bundles, were analyzed after testing by optical microscopy and scanning electron microscopy coupled with energy dispersive x-ray analysis (SEM-EDX). Specimens tested at room temperature exhibited wear features typical of brittle fracture

such as fractured fiber surfaces and no evidence of gentle abrasive or polishing wear (fig. 6). This is consistent with tests of other ceramic fibers (refs. 2 and 3). In previous work (ref. 4) with oxide ceramic fibers, brittle wear behavior was modelled using dimensional analysis and the same type of approach would be appropriate to apply to the room temperature tests conducted with the HPZ and Tyranno fibers.

At elevated temperatures the wear behavior, as observed by microscopy, is not by simple brittle fracture. The apparent wear mode is by wearing of the fiber surface layer (presumably a passivating oxide) which is generated by oxidation of the fiber at elevated temperatures (figs. 7 and 8). Since these fibers are not fully oxidized fibers, when they are tested in air at elevated temperatures an oxide layer grows on the fiber surface. During sliding the layer is worn off and fresh unoxidized surface is exposed which is then oxidized. This causes fiber wear in an oxidative-abrasive mode and eventually leads to fiber breakage. This behavior can be seen in figures 7 and 8 which show large amounts of wear debris at the sliding interface and severe fiber degradation at the breakage surface (fig. 9). Further evidence for an oxidative rather than a mechanical fracture wear mode is the lack of sharp fracture surfaces which accompany mechanical wear. In these elevated temperature tests the wear mode follows an oxide generation and removal path rather than simple brittle fracture which occurs at room temperature.

EDS analyses indicate that the debris is a combination of predominantly the fiber constituents as well as small amounts of wear debris from the Inconel disk as evidenced by the presence of nickel, chromium and iron (figs. 10 and 11). Because this wear mode is not predominantly based upon mechanical factors, as brittle fracture is, mechanical explanations of the wear behavior are inappropriate.

DISCUSSION

Considering the CTF data, shown in figure 5, it is apparent that the two fibers have markedly different tribological characteristics. At 25 °C, the Tyranno fiber has lower friction and better wear properties than the HPZ. At 900 °C, the performance of the Tyranno is significantly degraded and that of the HPZ remains about the same. The surface features of the worn specimens, combined with the fibers' mechanical and physical properties help explain why these tribological differences exist.

Relation of wear to physical properties. - The fiber tensile strength as a function of temperature is given in table IV. From the values it can be seen that the Tyranno is about twice as strong as the HPZ at room temperature but loses about half of its strength so that it is only about as strong as HPZ at 900 °C. This dramatic strength loss for Tyranno may be one factor in causing the dramatic drop in the CTF as the temperature is increased.

The tensile strength for HPZ, on the other hand, is relatively constant over the temperature range studied. Between 25 and 900 °C, HPZ only experiences about a 10 percent strength reduction. This may be one reason why the CTF for the HPZ is approximately constant for these tests regardless of temperature.

In a previous study on fully oxidized ceramic fibers (ref. 4), it was determined through the use of a dimensional analysis, that the wear behavior, or CTF, of the fibers is a function of the ratio of fiber strength in tension (tensile strength·fiber area) to the friction force(friction coefficient·applied load) assuming that mechanical effects dominate the wear process. Mathematically, the relation is as follows:

$$CTF = f((TS \cdot D^2)/\mu \cdot F_N)$$

where

CTF sliding cycles to failure
 f "is a function of"
TS fiber tensile strength
D fiber diameter
 μ friction coefficient
 F_N applied load

For the carbide-oxide fibers studied here this condition is satisfied only during room temperature testing. The wear mechanism for tests conducted at elevated temperature indicate that oxidative effects play a more significant role in the wear behavior than mechanical effects. This is especially apparent by the lack of brittle fracture surfaces in the wear area. Nonetheless, the previously derived mechanical model can be applied to the room temperature data gathered here where oxidative wear is not important.

Figure 12 shows the data curve generated from the data collected in reference 4 for oxide fibers. The data for the HPZ and the Tyranno fibers at room temperature falls on the curve indicating that mechanical effects do dominate the low temperature wear process. At elevated temperatures, the data deviates from the predicted mechanical behavior curve indicating that oxidation may indeed play an important role in the wear process.

These results highlight the importance of chemical stability for materials which are candidates for high temperature applications. The tests conducted here were of limited duration yet significant oxidative degradation of the fibers occurred. This was more so the case for the Tyranno fibers which displayed low wear resistance at high temperatures in addition to fiber oxidation. These results suggest that, under sliding conditions, more stable fibers based on oxides (e.g., $Al_2O_3-SiO_2$) be considered for seal applications for high temperature environments containing air.

Recently, Misra et al. (ref. 11) has conducted thermodynamic calculations on the stability of selected ceramics in both air and hydrogen with and without water vapor present. His research suggests that even nonoxide fibers like SiC are susceptible to degradation in reducing environments as well. This evidence further illustrates the potential problems of the use of fibers such as HPZ and Tyranno. Misra's work indicates that alumina based fibers are the best candidates for high temperature service in both oxidizing and reducing environments and could be considered for the application studied here.

SUMMARY AND CONCLUSIONS

The relative durability of two carbide-oxide fiber materials was evaluated using a pin-on-disk tribometer. The test results and observations from room temperature tests were consistent with the brittle behavior of ceramic materials in sliding contact and corroborated research done previously with oxide ceramic fiber materials. At elevated temperatures, chemical effects on friction and wear, namely fiber oxidative wear phenomena, seemed to play an important role degrading the tribological performance of the fibers.

Based upon these tests the following specific conclusions can be drawn:

1. The HPZ fiber displayed a remarkably constant wear rate with temperature which may have been due, in part, to its stable mechanical properties.

2. The fiber durability at room temperature appears to be related to the fiber mechanical properties and tribological parameters as observed in previous studies.

3. The room temperature behavior was in general agreement with a mechanical model developed for oxide fibers tested previously. At elevated temperatures, however, the mechanical model does not adequately predict fiber performance further indicating that chemical and oxidative as well as mechanical properties affect tribological performance of the fibers.

4. Based upon the evidence of chemical-oxidative wear the use of these carbide-oxide fibers for seal applications which include high temperature air exposure is not recommended.

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TABLE I. - COMPOSITION AND PHYSICAL PROPERTIES OF CANDIDATE CERAMIC SEAL MATERIALS^a

Property	Tyranno	HPZ
Fiber composition, wt%	50 Si 30 C 17 O 3 Ti	57 Si 28 N 10 C 5 O
Fiber diameter, μm	11	12.6
Density, g/cc	2.4	2.4
Filament tensile strength @ 25 °C, MPa	3000	1625
Filament tensile modulus, GPa	180	176

^aTaken from manufacturer's data.

TABLE II. - NOMINAL COMPOSITION AND HARDNESS OF INCONEL TEST DISK SPECIMENS

Property	Value
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	RC 34

TABLE III. - FRICTION AND WEAR DATA SUMMARY^a

Test temperature, °C	Material	μ	CTF, cycles
25	Tyranno HPZ	0.17±0.02	17 700±5000
		0.69±0.05	2 070±400
500	Tyranno HPZ	0.34±0.05	3 100±500
		0.59±0.05	2 140±600
900	Tyranno HPZ	0.61±0.10	1 250±500
		0.63±0.04	2 000±350

^aUncertainties represent one standard deviation for the friction coefficient and data scatter band for the CTF data.

**TABLE IV. - FILAMENT TENSILE STRENGTH
VERSUS TEMPERATURE^a**

Fiber material	Tensile strength, mPa		
	25 °C	500 °C	900 °C
Tyranno	3000	2200	1500
HPZ	1625	1523	1456

^aData taken from manufacturer data sheet.

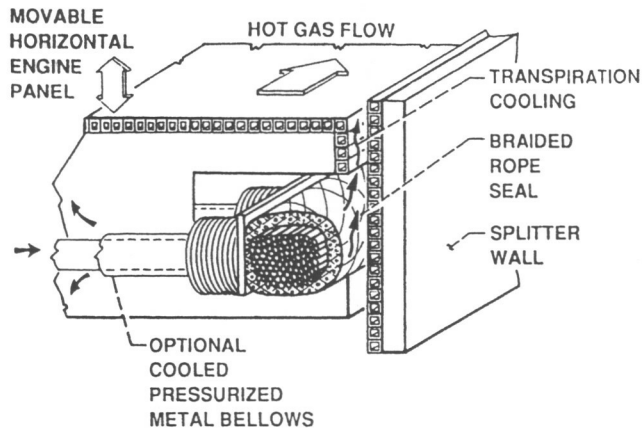


Figure 1.—Cross section of proposed engine seal.

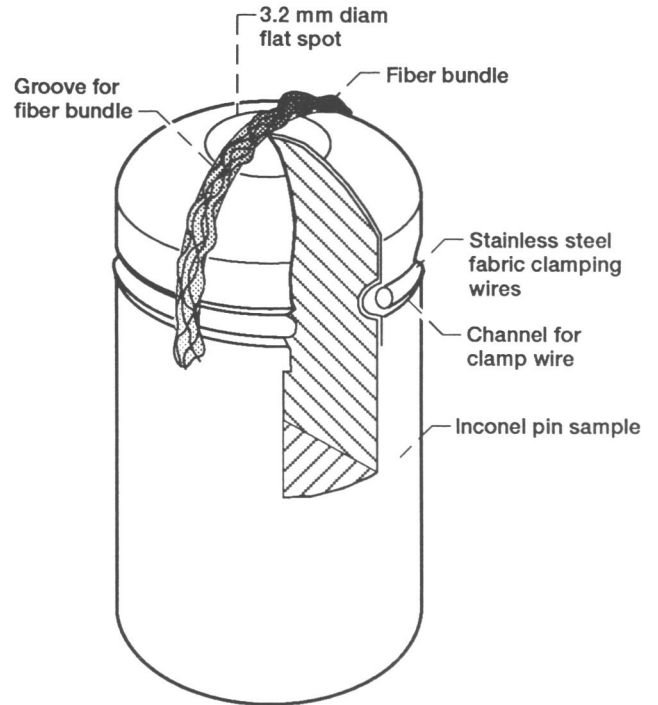


Figure 2.—Pin test specimen.

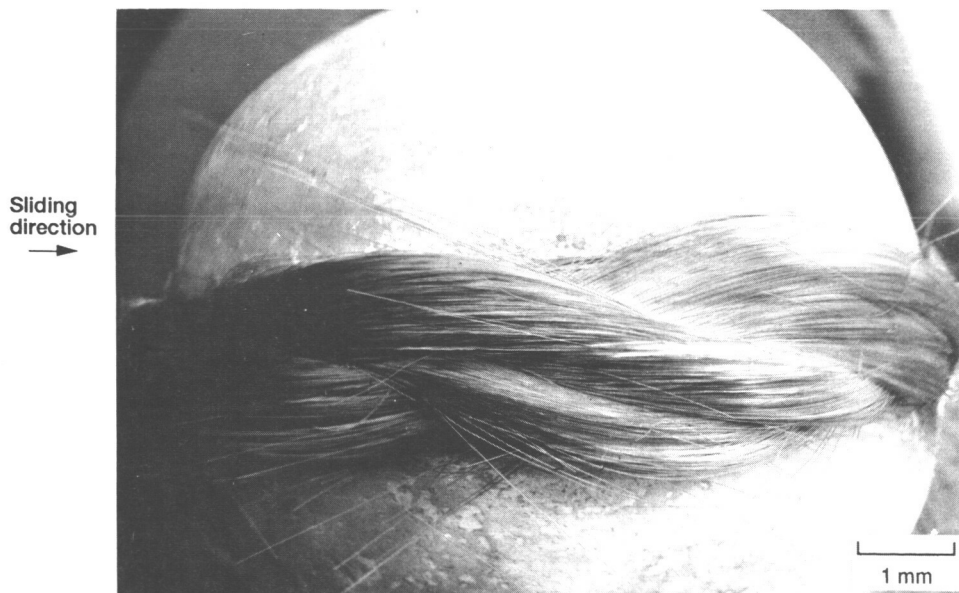


Figure 3.—SEM photomicrograph of fiber-pin specimen prior to testing. Sliding direction is from left to right.

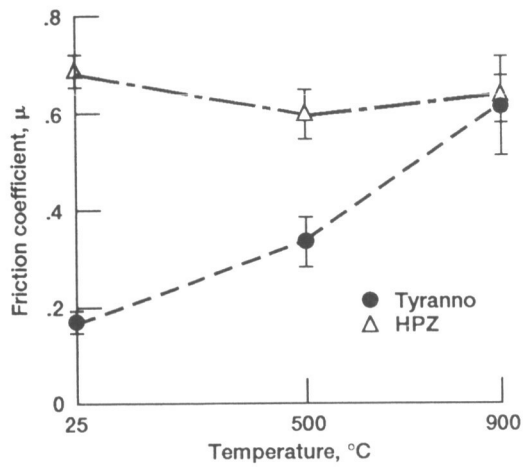


Figure 4.—Friction coefficient vs. temperature for HPZ and Tyranno fiber in sliding against Inconel 718 at 0.025 m/s, 4.9N load in air. Error bars represent one standard deviation of data.

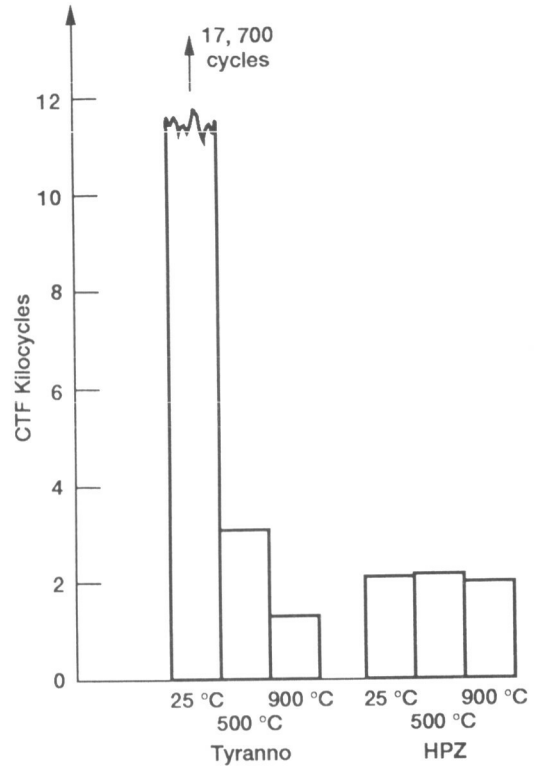


Figure 5.—Cycles to failures (CTF) data for the Tyranno and HPZ fibers after sliding against Inconel 718 at 0.025 m/s, 4.9N load in air.

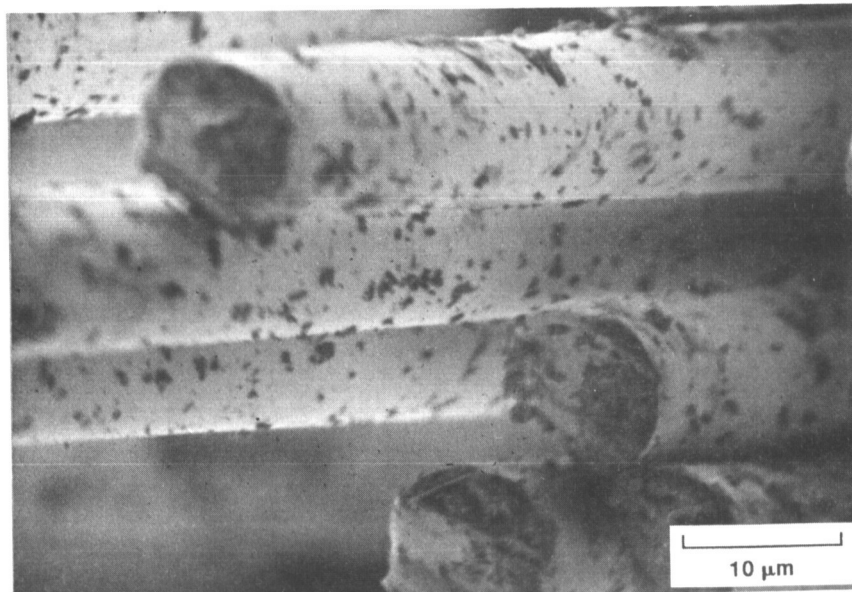


Figure 6.—Tyranno fibers after sliding at room temperature. Brittle behavior, as seen in sharp, faceted failure surface, characterizes the wear behavior.

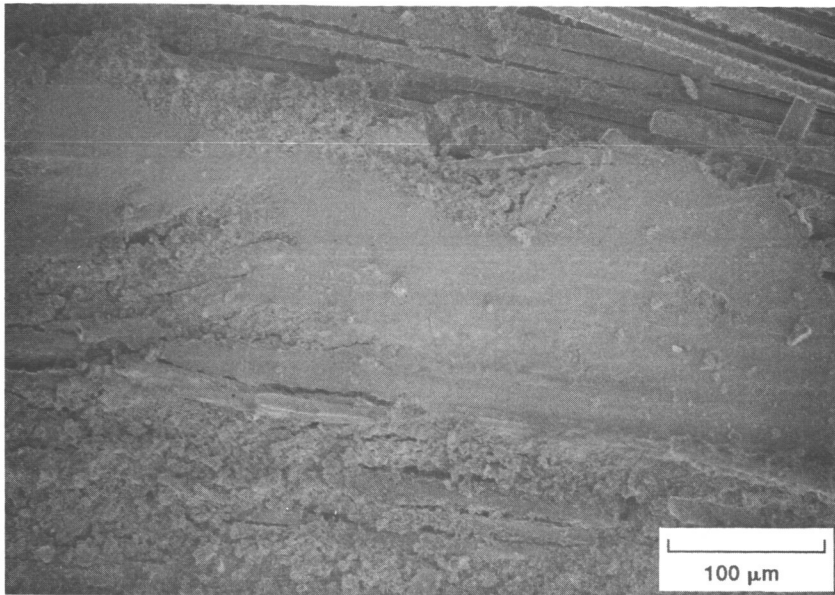


Figure 7.—Wear debris deposit on fiber surface of Tyranno specimen after sliding at 900 °C.

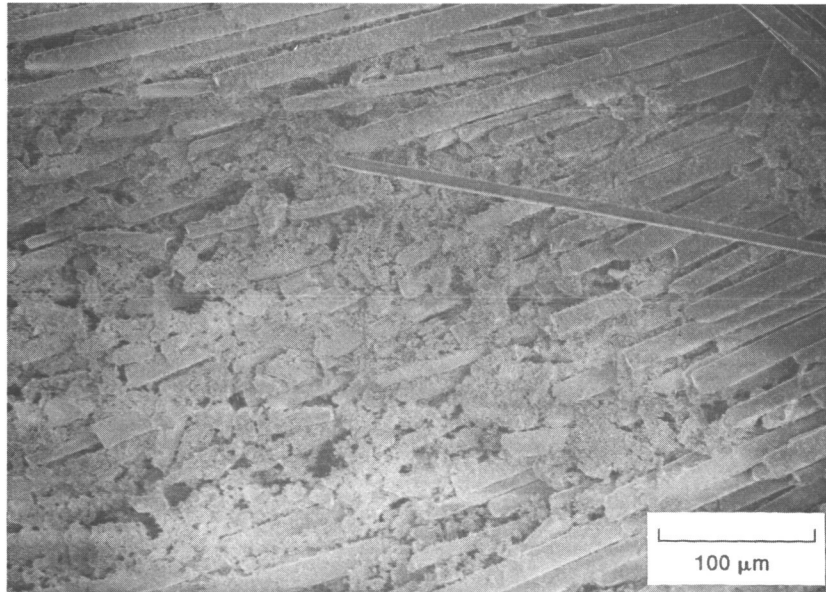


Figure 8.—Wear debris deposit on surface of HPZ fibers after sliding at 900 °C.

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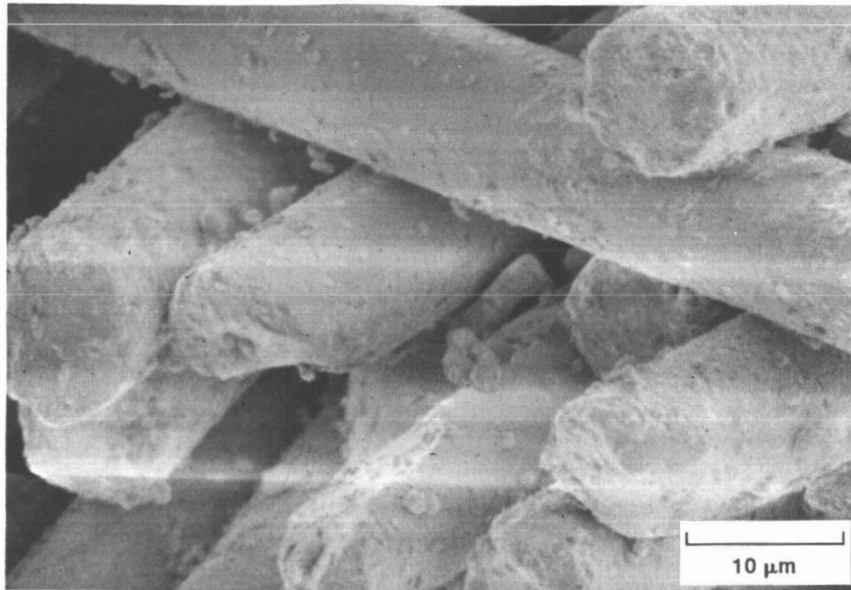


Figure 9.—Tyranno fiber wear surface after 900 °C testing. Environmental degradation (oxidation) is evidenced by the uneven failure surface.

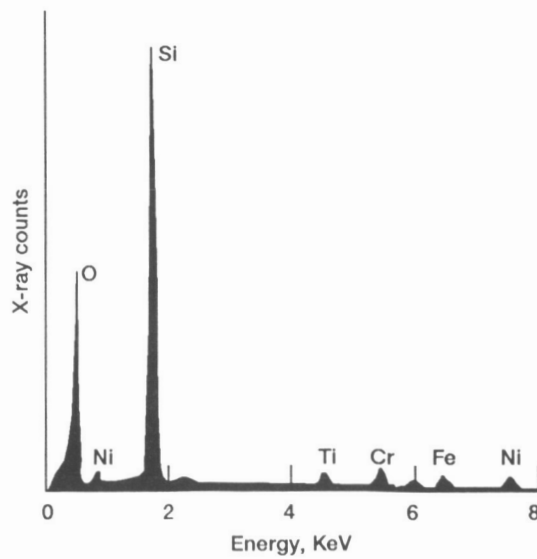


Figure 10.—EDS X-ray spectrum analysis of wear debris on Tyranno fibers after testing at 900 °C. Spectrum shows transfer of Ni, Cr and Fe from Inconel 718 disk.

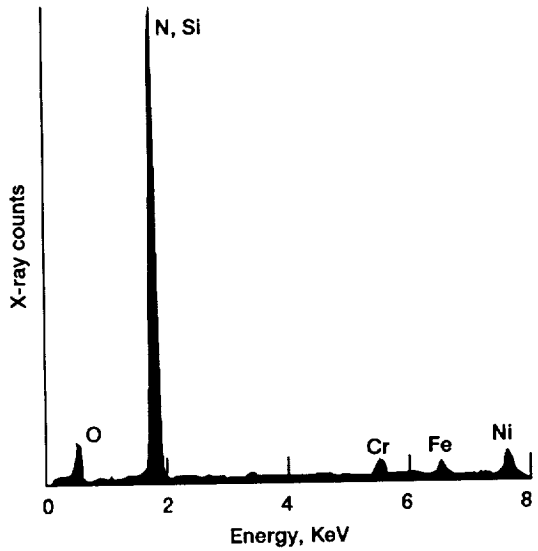


Figure 11.—EDS X-ray analysis of wear debris on HPZ fibers after testing at 900 °C. Spectrum shows transfer of Ni, Cr and Fe from Inconel 718 disk.

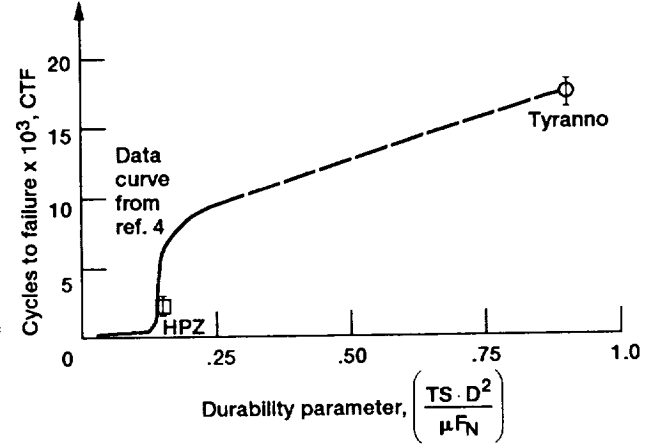


Figure 12.—Fiber durability (CTF) vs. non-dimensional durability parameter which represents fiber strength in tension to sliding friction forces. Data for HPZ and Tyranno shown from tests at 25 °C. Error bars represent data scatter from repeat tests. Curve (solid portion) generated in ref. 4. Results show a relation between fiber wear and mechanical properties.

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