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Evaluation and Characterization of Ceramic Bearing Materials

by Paul J. Huang, Clifford W. Hubbard,
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Abstract

This report discusses the findings from one phase of our ongoing work to evaluate materials for Army bearing systems. The objective of this phase is to determine the response and longevity of various silicon nitride (Si_3N_4) materials to rolling contact fatigue (RCF) using hybrid and all-ceramic systems. Tests were conducted under regular lubrication and lubrication-starved conditions for extended periods. A correlation between RCF life and the hardness, strength, and microstructure of each silicon nitride is made. The various silicon nitride materials evaluated in these RCF tests were selected on the basis of providing a varied response to the RCF parameters and conditions used.

Acknowledgments

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1. Introduction

The pursuit to improve tribological performance of bearings has taken us to examine the use of hybrid and all-ceramic systems. The effort to improve lifetimes of bearing components is propelled by (1) the requirement for drive-train components to survive higher loads, temperatures, and speeds, necessitated in advanced emerging Army systems, and (2) the need to reduce surface degradation of current system components from environmental effects.

Current ceramic bearing materials being considered today fail in the same noncatastrophic mode as steel elements, which is an important consideration for their acceptability (Katz 1995). Other reasons why silicon nitride (Si_3N_4) is being considered for replacing steel elements are high hardness, low density, corrosion resistance, high operating temperatures, and high bend strength. Hardness is important for wear and abrasion resistance. Lower density allows for higher rotational speeds, and the other desired properties need no further elaboration (Katz 1993).

Silicon nitride has been intensely studied for more than 20 yr as an alternative for many metallic structural applications at room and elevated temperatures. Many of these applications have centered on high-temperature materials for engines. Other applications have included cutting tools, electronic packaging, bearings, low-density structural materials, and wear components.

Silicon nitride components are difficult to fabricate. Typically, parts are densified from silicon nitride starting powders; although, for a few applications (such as electronic), silicon nitride is applied by chemical vapor deposition. Because silicon nitride is a covalently bonded material and has a low self-diffusion coefficient, it takes a large amount of energy to promote densification through diffusion. This can only be accomplished at extremely high temperatures and pressures. Because of this difficulty, densification aids are added to promote sintering at low temperatures. These densification aids react with the silica inherently present on the surface of each silicon nitride particle to form a liquid phase. This liquid phase allows some densification through particle rearrangement. More significantly, it allows for the silicon nitride to be sintered through a solution reprecipitation mechanism. Because of the presence of these densification aids, it is best to think of silicon nitride as an alloy, since the choice of densification aids greatly affects the final properties of the material. In most cases, the densification aids react with the silica to form a second phase that can be either crystalline or amorphous and is usually located at the grain boundaries. SiAlONs are a special case where alumina is added along with other densification aids. The alumina goes into solid solution with the silicon nitride, with the aluminum and oxygen substituting for the silicon and nitrogen, respectively.

Because of the many different denisifcation aids that can be used, different silicon nitride alloys can be developed to maximize materials property for specific applications. Densif-ication aids can be chosen to allow sintering of silicon nitride at temperatures below its decomposition temperature without the aid of pressure, The selections of densification aids determines the processing technique (i.e., gas-pressure sintered [S], hot isostatically pressed [HIPed], or hot pressed), which, in turn, determines the microstructural features. HIPed silicon nitrides tend to have finer grain sizes and higher strengths, whereas, in sintered materials, a duplex microstructure can be developed, which can lead to a higher toughness.

2. Materials and Experimental Procedure

2.1 Materials. Various silicon nitride materials (Table 1) were selected for evaluation in rolling contact fatigue (RCF) tests using hybrid and all-ceramic systems. The materials were selected on the basis of providing a varied response to the RCF parameters and conditions used, not solely for a comparison of bearing quality. Most, if not all, of the materials have since been replaced by their manufacturers with upgraded or modified versions. One material, Allied-Signal GN10 was not developed for bearing material applications but rather as a high-temperature structural ceramic.

2.2 RCF. All RCF testing for the present effort was performed on a ball/rod rig as seen in Figure 1 (developed by Federal-Mogul and now produced by NTN) under the conditions listed in Table 2.

Table 1. Materials Information

Supplier	Densification Method	Additive	Density (g/cm^3)	Knoop Hardness at 1,000 g (GPa)	Phase Content
Norton Advanced Ceramics (Cerbec)	HIP	MgO	3.23	15.65	25% a 75% β
ESK-EK9980 HIP	HIP	MgO	3.17	14.51	β phase
ESK-EK9980 S	S	La_2O_3/Al_2O_3	3.26	13.77	β phase
Allied-Signal GN10	HIP	Y_2O_3/SrO	3.31	—	>95% β

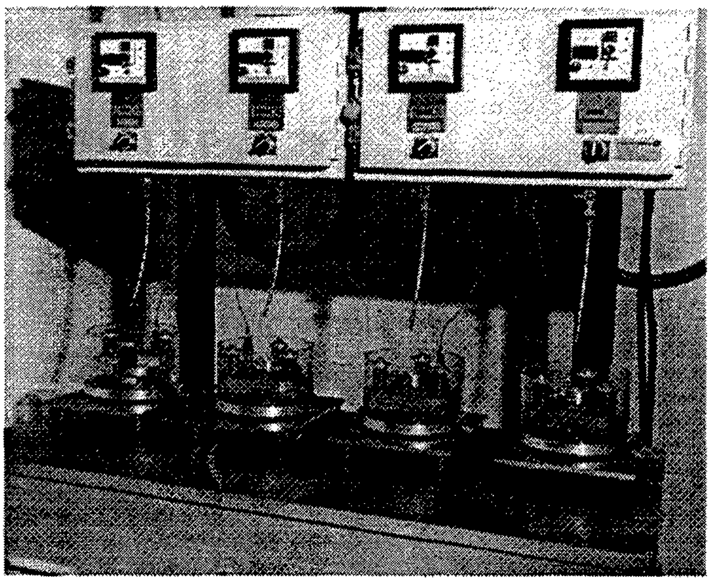


Figure 1. RCF Test Rig.

The RCF operates under the basic principle (as illustrated in Figure 2) and consists of a rotating cylindrical test specimen alternately stressed by rolling contact with three radially loaded balls. The three balls, separated by a retainer, are radially loaded against the test specimen by two tapered bearing cups thrust-loaded by three compression springs (Glover 1982).

Replacing the balls, as necessary, during RCF testing with hybrid systems provides further information on whether or not spalling or wear might be life limiting for a silicon nitride bearing material.

Table 2. Conditions for RCF Testing

Hertzian Stress	6.07 GPa (865 ksi) for condition 1[a] 6.40 GPa (911 ksi) for condition 2[b]
Rotational Speed	3,600 rpm
Lubrication Supply	8-10 drops/min
Lubrication Type	MIL-PBF-23699[c]
Specimen Length	76.2 mm +0.025/–0.000 in
Specimen Diameter	9.52 mm +0.0000/–0.00005 in
Surface Finish	0.05 to 0.10 μm AA
Temperature	20–25° C

[a] Ceramic rod with steel balls.
[b] Ceramic rod with ceramic balls.
[c] U.S. Department of Defense (1997).

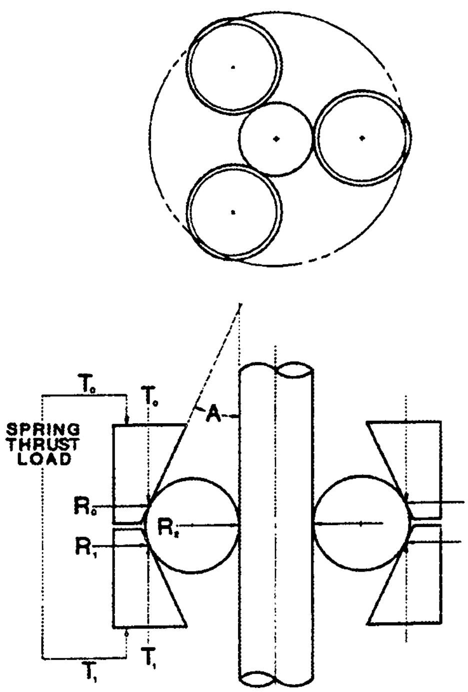


Figure 2. **Schematic of Tester.**

All four stations of the RCF tester were operated simultaneously to speed up acquisition of the RCF data. At least three wear tracks and associated fatigue spalls were obtained for each specimen condition, and the specimens were alternated among the test stations to minimize any systematic experimental error. During the ceramic-on-steel tests, the balls were 52,100 steel balls and the rods were the silicon nitride materials. In the ceramic-on-ceramic tests, the ceramic balls were NBD 100 grade 5 silicon nitride, while the rods were the silicon nitride materials indicated in the specific tests. On the ah-ceramic system test, lubrication (ML-PRP-23699 [U.S. Department of Defense 1997]) was provided for the fiist 24 hr and then discontinued for the remainder of the test.

2.3 **Characterization.** **The** room-temperature tensile strength of each material was determined by diametrally compressing a right circular cylinder between two flat platens. Tests were conducted in air using a crosshead speed of 0.5 mm/min. A single piece of a manila file folder was placed between the platen and the specimen at each loading point to provide appropriate stress distribution. The specimens had a diameter-to-thickness ratio of 4 to 1, with a nominal diameter of 9.5 mm and a thickness of 2.4 mm. The diameter was the same as that of the RCF specimens. All specimens were machined from a single RCF rod of each silicon nitride. No additional machining was done to the circumference of any cylinders, but both flat surfaces were machined to a 20.3-μm RMS finish or better. The tensile strength was calculated using equation (1):

$$\sigma_T = 2P/\pi dt, \tag{1}$$

where

σ_T = tensile strength (MPa),

P = applied load (N),

d = specimen diameter (mm), and

t = specimen thickness (mm).

Hardness was determined using a Knoop diamond indenter with a 1,000-g load.

Samples were prepared for microstructural characterization by sectioning RCF rods with a diamond saw and mounting the sections in acrylic The samples were then rough-ground with silicon carbide abrasives and ground for 12 hr with 9-µm diamond media on lead platens using kerosene as a lubricant. The samples were given a final polish using 0.05-µm silica with a nylon cloth on a vibratory polisher.

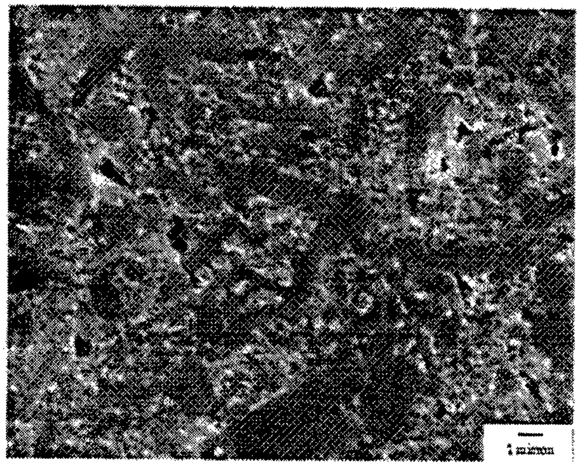
Optical microscopy was conducted to examine the distribution of the phases and the homogeneity of the material. Samples were etched with a boiling 40% I-IF solution for 10 min and then coated with 4 nm of a gold/palladium alloy. Scanning electron microscopy (SEM) was also used to examine the microstructure and fracture surfaces as seen in Figures 3 and 4. X-ray diffraction (XRD) was performed for phase analysis.

3. Results

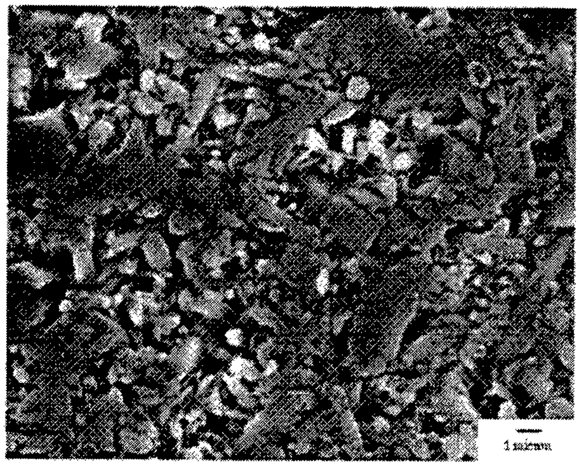
3.1 Ceramic vs. Steel. In the hybrid tests (i.e., silicon nitride rods and steel balls), the steel balls failed before the ceramics. When the steel balls failed, they were replaced and the test was continued until the ceramic rods failed.

As can be seen in Table 3, of the four ceramics that were run to failure, the ESK sintered material had a substantially longer fatigue life than the other materials tested. ESK HIPed had the second longest lifetime. Both of these materials greatly exceeded the lifetimes of the Cerbec and GN10 materials.

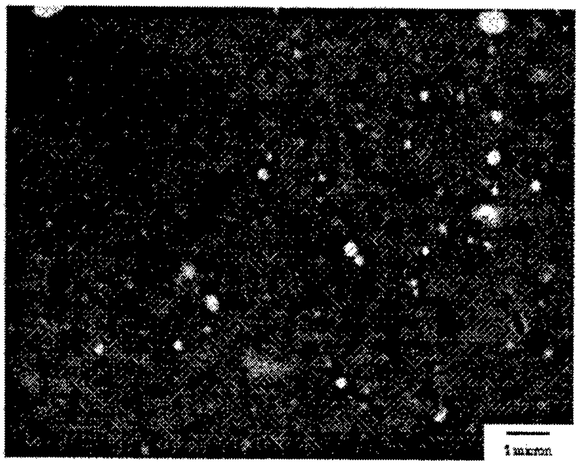
3.2 **Ceramic vs. Ceramic.** Tests were done on Cerbec and GN10 specimens, where, after 24 hr, the lubrication feed was stopped with the idea of accelerating the test in a more severe condition. It was observed that the lubrication-starved condition had a higher temperature than the lubricated condition. Retained lubrication was observed when testing was concluded, which prevented the steel raceway from seizing during the tests.



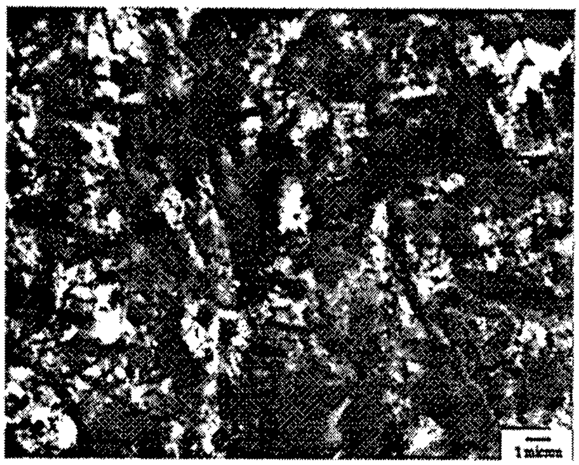
(a) ESK Sintered at 5,000×.



(b) ESK HIPed at 5,000×.



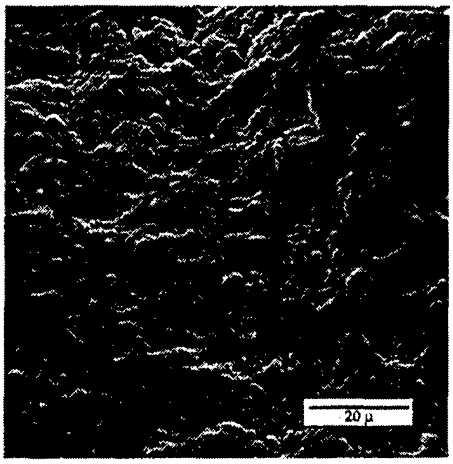
(c) **Cerbec at 9,000×.**



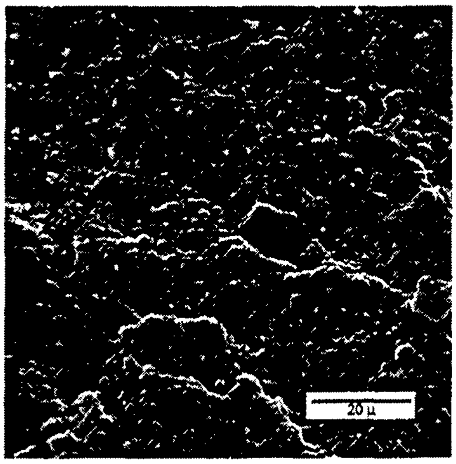
(d) GN10 at 5,000×.

Figure 3. Micrographs of Si_3N_4 Specimens Using SEM.

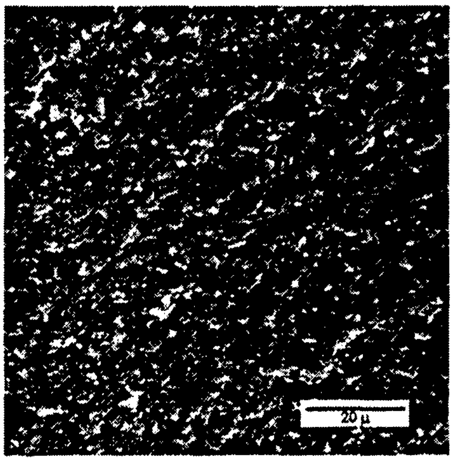
The Cerbec material had a runout at 586.6 and 1,179.1 hr, while the GN10 specimens produced failures at 3 1.1 and 87.9 hr and a runout at 473.4 hr. Typical spallation occurred in all the materials except for GN10. Spallation is when material chips/spa& off the specimen in a fashion similar to metallic bearing materials. While failure was not the result of chipping or spallation for GN10, a smooth elliptical depression, which acted like a spall, was formed in the wear track, thereby



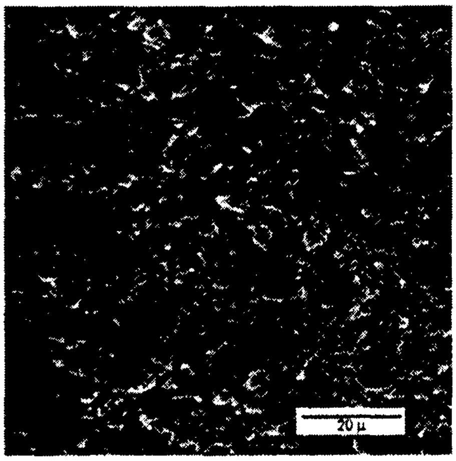
(a) ESK Sintered at 2,000×.



(b) ESK HIPed at 2,000×.



(c) Cerbec at 2,000×.



(d) GN10 at 2,000×.

Figure 4. Fractography Micrographs of Diametral Compression Specimens Using SEM.

terminating the test. It was confirmed by the preliminary profilometry data that the surface was smooth at the point of failure, whereas traditional failures have a very rough surface profile. This indicates that GN10 is not a bearing-grade material.

3.3 Diametral Compression. The results of the diametral compression testing can be seen, in Table 4.

Table 3. Lifetime Data - Tests Conducted With Ceramic Rod on Steel Balk

Ceramics (Si_3N_4): With 52,100 Balls	B10($\times10^6$)	B50($\times10^6$)	Slope
ESK-EK9980 S (with 4 pts.)	264.21	404.49	4.42
ESK-EK9980 HIP (with 6 pts.)	108.71	366.86	1.55
Cerbec (with 9 pts.)	14.47	63.02	1.28
Allied-Signal GN10 (with 9 pts.)	6.56	30.38	1.23
M50 Steel Baseline (Middleton et al. 1992)	2.74	7.91	2.93

Table 4. Diametral Compression Test Data

Material	Mean σ_T (MPa)	No. of Specimens Tested	Standard Deviation
Allied-Steel GN10	772	11	55
ESK-EK9980 S	709	12	91
ESK-EK9980 HIP	708	12	92
Cerbec	589	12	134

4. Discussion

4.1 RCF Test, The results of the RCF test are shown in Tables 3 and 5. It can be seen by comparing Tables 1 and 3 that there is a trend between having low hardness and longer RCF lifetime for the three materials designed specifically as bearing materials. Lower hardness materials distribute the load over a greater area and reduce the stress on the material. Comparison of hybrid tests and all-ceramic tests showed a significant improvement over steel systems (Middleton et al. 1991). In the all-ceramic systems tested here under lubrication-starved conditions, the ceramic-on-ceramic systems showed that they could continue to perform when lubrication was discontinued. The runouts of these tests were discontinued because the length of time of the test did not justify the continuation of the test until the ceramic rod failed. Runout refers to the ability of the material to not fail in a reasonable time.

Table 5. Lifetime Data - Tests Conducted With Ceramic Rod on Ceramic Balls

Ceramics (Si_3N_4): With Ceramic (Si_3N_4) Balls	B10($\times 10^6$)	B50($\times 10^6$)	Slope
GN10 (With 14 pts.) With NBD 100 Balls	0.95	82.03	0.42
Cerbec With NBD 100 Balls	no data, all runouts[a]	—	—

[a] Runout tests that are discontinued before failure of the ceramic rod occurred.

4.2 **Diametral Compression Test, The** diametral compression test was used to determine the tensile strength of these materials because of the similarity in the specimen geometry between this technique and the RCF test. This technique has been previously used to determine the tensile strength of ceramics, having been first used to test concrete in the early 1950s (Cameiro and Barcellos 1953) and since then for advanced monolithic ceramics (e.g., Si_3N_4 and Al_2O_3) (Rudnick, Hunter, and Holden 1963; Marion and Johnstone 1977; Ovri and Davies 1987; 1988). Failure of ceramic bearings typically occurs due to spallation that results from the development, growth, and coalescence of microcracks at or very near the surface. Table 4 summarizes the tensile strength of each material. There does not appear to be a correlation between strength and RCF lifetime. GN10 had the highest strength and the lowest standard deviation, yet had the shortest RCF lifetime. This is not surprising since GN10 was developed for structural and not bearing applications. There was essentially no difference in strengths between the two ESK materials and yet the sintered material had a substantially greater lifetime in RCF.

4.3 **Microscopy.**

4.3.1 ***Optical Microscopy. Optical*** microscopy showed that the two ESK silicon nitrides had a more uniform distribution of a second phase and a more homogeneous microstructure than either the Cerbec or GN10 materials. In the Cerbec and GN10 materials, there were large pockets of second phase, while, in both ESK materials, the second phase was uniformly distributed. There also appeared to be preferential polishing of the second phase in the ESK materials, indicating that this second phase was not as hard as the silicon nitride.

4.3.2 Electron Microscopy. The scanning electron micrographs taken of the polished and etched samples (Figure 3) show the microstructure and distribution of grain boundary phase. The ESK materials are marked by having larger acicular grains (with a high length-to-diameter [L/D] ratio) surrounded by smaller equiaxed grains. The grain boundary phase is distributed evenly along the grains. There are no large pockets of it. This is in contrast to the GN10 material, where there are large pockets of the grain boundary phase. This microstructure is marked by having a more uniform grain size, and the large grains that are forming do not have as large an L/D ratio as the ESK materials. The Cerbec material has a fine equiaxed grain size. This is to be expected, given that it was processed at temperatures low enough to keep some of the alpha-phase silicon nitride from reacting to form the beta-phase silicon nitride. The fine grain size and alpha phase are what give the Cerbec silicon nitride its high hardness. Pockets of gram boundary phase can be seen to be nonuniformly distributed within this material.

The two ESK materials had similar microstructures that appear to be advantageous in RCF. Although fracture toughness was not measured here, it is believed that the microstructures of the ESK materials would give higher fracture toughness than the GN10 or Cerbec material. This could result in longer RCF lifetimes. Clearly, the grain boundary phase in the ESK materials was more evenly distributed. Under high Hertzian loads, these large pockets of grain boundary phase could act as flaws. When the grain boundary phase is more evenly distributed, the loads are carried by the stronger silicon nitride phase. Although this explains why the two ESK materials performed better than the Cerbec and GN10 materials, it does not explain the significant differences between the ESK sintered and ESK HIPed. The ESK HIPed had a density of 3.17 g/cm^3, which is lower than the theoretical density of silicon nitride, which is 3.22 g/ cm^3. Residual porosity was not removed during the HIPing process, which could explain the difference between the ESK materials. The scanning electron micrographs of the fracture surface clearly show that the ESK HIPed material has more porosity than the sintered material. More work is needed to positively determine the amount of porosity in the ESK HIPed material. The longer lifetimes of the ESK materials appear to be due, in part, to the presence of a softer, more uniformly distributed second phase, which allows for greater stress distribution of the Hertzian stresses. The lower porosity of the ESK sintered compared to the ESK HIPed may account for the different lifetimes of these similar silicon nitrides. In fact, all the HIPed materials seem to have a higher degree of porosity than the sintered material.

The all-ceramic systems exhibited greater RCF endurance than the hybrid systems, and extraordinary RCF life was observed for lubrication-starved all-ceramic systems. The Cerbec material performed better than the GN10 material in the all-ceramic system. Not surprisingly, the Cerbec material also performed better than the GN10 in the hybrid bearing systems.

For the hybrid bearing systems the ESK sintered material was substantially better than any of the other materials tested.

More work is needed to determine whether a large-grained duplex microstructure, fine-grained duplex microstructure, or intermediate-grained microstructure is best. Work is under way at this time to determine which is the best microstructure and to determine the influence of fracture toughness.

As expected, the nature and distribution of the grain boundary phase have an important effect on the RCF lifetimes. More work to understand the nature of the grain boundary phase is also under way. Transmission electron microscopy (TEM) is being carried out to determine the chemistry and crystallinity of the grain boundary phase. Use of a nanoindentor to determine the hardness of the grain boundary phase is being explored. It was noted that the grain boundary phase of the ESK sintered was much more resistant to the hydrofluoride (HF) etch than the ESK HIPed, which, given the same etch conditions, was overetched as compared to the sintered material. This made it hard to evaluate the porosity of the ESK HIPed material and compare it to the other materials. Additional work is needed to determine the porosity of the different materials tested.

Materials to be used for bearing should have the minimum amount of porosity possible. Even a small amount of fine porosity greatly affects the RCF lifetimes.

Sintering may be a better way to densify bearing materials than HIPing. During the HIP cycle, residual porosity is squeezed until the pressure in the pore equals the HIP pressure; then, there is no more pore removal. Sintering is usually a slower process and uses a greater amount of liquid phase. This can result in more complete pore removal. This work is part of an ongoing effort to evaluate bearing materials and to understand the attributes that make them good so that better bearing materials can be designed. Future work will include evaluation of different silicon nitrides, as well as other materials.

5. Conclusion

A duplex microstructure consisting of large acicular grains with a high L/D ratio surrounded by smaller grams gives the best RCF lifetime.

A homogeneous fine distribution of the grain boundary phase with no large pockets of grain boundary phase gives the best RCF performance.

Low hardness materials seem to perform better than high hardness materials.

Small amounts of porosity degrade RCF performance without affecting strength and hardness.

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This report discusses the findings from one phase of our ongoing work to evaluate materials for Army bearing systems. The objective of this phase is to determine the response and longevity of various silicon nitride Si_3N_4 materials to rolling contact fatigue (RCF) using hybrid and all-ceramic systems. Tests were conducted under regular lubrication and lubrication-starved conditions for extended periods. A correlation between RCF life and the hardness, strength, and microstructure of each silicon nitride is made. The various silicon nitride materials evaluated in these RCF tests were selected on the basis of providing a varied response to the RCF parameters and conditions used.

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