DOE/NASA/50162-2
NASA TM-100806

Tribological Properties of Alumina-Boria-Silicate Fabric from 25 to 850 °C

Christopher DellaCorte
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

March 1988

Work performed for
U.S. DEPARTMENT OF ENERGY
Conservation and Renewable Energy
Office of Vehicle and Engine R&D
Washington, D.C. 20545
Under Interagency Agreement DE-AI01-86CE50162

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Christopher DellaCorte
National Aeronautics and Space Administration
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E-3986

SUMMARY

Demanding tribological properties are required of the materials used for the sliding seal between the sidewalls and the lower wall of the variable area hypersonic engine. Temperatures range from room temperature and below to operating temperatures of 1000 °C in an environment of air, hydrogen, and water vapor. Candidate sealing materials for this application are an alumina-boria-silicate, ceramic, fabric rope sliding against the engine walls which may be made from copper- or nickel-based alloys. Using a pin-on-disk tribometer, the friction and wear properties of some of these potential materials and possible lubrication methods are evaluated. The ceramic fabric rope displayed unacceptably high friction coefficients (0.6 to 1.3) and, thus, requires lubrication. Sputtered thin films of gold, silver, and CaF_2 reduced the friction by a factor of two. Sprayed coatings of boron nitride did not effectively lubricate the fabric. Static heat treatment tests at 950 °C indicate that the fabric is chemically attacked by large quantities of silver, CaF_2, and boron nitride. Sputtered films or powder impregnation of the fabric with gold may provide adequate lubrication up to 1000 °C without showing any chemical attack.

INTRODUCTION

A critical aspect in the development of the variable cross section area hypersonic engine is the sliding seal between the engine sidewalls and the movable lower engine wall. Figure 1 is an illustration of a proposed sliding seal configuration for this interface. Particularly important to the success of the seal is the material selection and characterization of the sliding components, namely the seal rope fabric and its counterface, the engine walls. These components must have suitable mechanical properties such as strength, creep resistance, and ductility. They must also exhibit excellent thermal stability, chemical stability, and tribological properties over a very wide temperature range which includes cold engine check out and hot engine articulation at approximately 1000 °C.

Several candidate sliding seal and counterface engine materials were selected on the basis of their mechanical properties and suitability as potential high temperature engine seal materials. The candidate seal rope fabric material is made from alumina-boria silicate fibers. It is commercially available under the trade name Nextel 312. It exhibits excellent mechanical properties and is, under most conditions, thermally and chemically stable in air to over 1000 °C. Potential engine wall materials, which will be the seal fabric's sliding counterface, are some nickel base superalloys such as Inconel X750, copper (ref. 1) and a new Ti_3Al-Nb alloy. Copper is not a high temperature alloy but is a material under consideration by the engine manufacturers for this application.

The following study determines the friction and wear properties of some of these potential seal and engine wall materials. The tests are done using a pin-on-disk tribometer. The pin is wrapped with a sample of the seal fabric and slid against a disk made from one of the candidate engine wall materials. The friction and wear properties ascertained are needed to help determine engine wall articulation power requirements, long term stability, and feasibility of having a sliding seal in the hot environment of the engine. Methods to lubricate the materials in the engine environment are also developed and evaluated.

ACKNOWLEDGEMENT

The author wishes to thank Mr. Frank Honecy of the Surface Science Branch at NASA Lewis Research Center, Cleveland, Ohio for preparing and applying the sputtered thin film lubricants to the ceramic fabric samples tested in this study.

EXPERIMENTAL MATERIALS

Seal Fabric

A fabric woven from Alumina-Boria-Silicate fiber was tested in this program. The composition of the fibers, designated AF-40, by weight is, 62 percent Al_2O_3, 14 percent B_2O_3, and 24 percent SiO_2. This fabric has excellent strength and flexibility to temperatures over 1000 °C. The fabric is made from fibers 10 µm in diameter. The weave is a cross weave type. Each weave bundle contains 390 fibers. The fabric is tightly woven and has the general consistency of heavy burlap. Figure 2 is a photograph of the test fabric.

Fabric Lubrication Treatment

Sputtered or sprayed thin films of different solid lubricants were applied over the bare fabric surface prior to sliding. The sputtered lubricants were silver, gold, and calcium fluoride. Silver and gold are potentially good low temperature lubricants because of their low shear strength and calcium fluoride is a good high temperature lubricant because it displays ductile behavior above about 500 °C (ref. 2). The fabric was sputtered clean prior to lubrication deposition. The sputtering parameters are given in Table 1. Boron nitride (BN) was also tested as a potential fabric lubricant. This coating was applied by spraying BN in a methylene chloride slurry onto the fabric then allowing the methylene chloride to evaporate. This procedure produced a relatively thick (~0.1 mm) BN coating on the fabric.

The following lubricant treated fabric specimens were prepared for use in this study:

Fabric Designation	Lubricant Coating
AF-40	1000 Å Ag
AF-40	500 Å CaF_2
AF-40	1000 Å Ag + 500 Å CaF_2
AF-40	1500 Å Au
AF-40	boron nitride

All three of the sputtered lubricants are thermally and chemically stable to at least 950 °C in air or hydrogen, the anticipated engine environment. At elevated temperatures, BN oxidizes in air to form boric oxide which may provide a lubricating effect.

Seal Counterface Material-Test Disks

The counterface materials tested in this study were Inconel X-750 and high purity copper. Inconel X-750 is a nickel-chromium based superalloy hardened to Rockwell C-39. It has a nominal weight percent composition of 70Ni, 16 Cr, 7.5 Fe, 2.5 Ti, 1 Co, 1 Mn and 0.1 C. The surface finish of the Inconel and copper disk specimens is 0.12 and 0.14 μm respectively. The disks are 6.35 cm in diameter and 1.27 cm thick.

APPARATUS AND PROCEDURES

Test Apparatus

A pin-on-disk apparatus was used in this study to evaluate the candidate seal materials and the lubrication methods proposed. Figure 3 is an illustration of the test rig. With this apparatus, a metal pin was covered with the fabric to be tested and loaded against a rotating Inconel or copper disk by means of deadweights. The fabric was fastened to the pin with a wire loop clamp which rested in a machined groove in the outside diameter of the pin (fig. 4). The end of the pin has a 1 in. radius and a 1/8 in. flat spot on its tip. For elevated temperature tests, the specimens were heated by a low frequency induction heating coil located circumferentially around the disk. The surface temperature of the disk was monitored with an infrared pyrometer. The pin generates a 51 mm diameter wear track on the disk.

Test Conditions

The sliding velocity for these tests was 0.27 m/s (100 rpm). The applied load to the pin was 270 g which gave a contact pressure between the fabric and the disk of about 338 kPa. The test atmosphere during these tests was moist air with a relative humidity of 35 percent at 25 °C.

Test Procedure

Prior to each test, the polished disks were cleaned with ethyl alcohol, lightly scrubbed with levigated alumina, rinsed with deionized water, and dried. The specimens were loaded into the test chamber and the test gas was allowed to purge the chamber for 10 min before testing began.

The total test duration was 1 hr. The specimens were slid for the first 20 min at room temperature then slid during a 10 min heating period to 850 °C (700 °C for the copper disks). They were then slid for 10 min at 850 °C (700 °C for the copper disks) and then slid for the final 20 min during cooling to 25 °C. Selected specimens were tested twice to ascertain tribological behavior during repeated test temperature cycles.

TEST RESULTS

The test results are given in table 1 and graphed in figures 5, 6, and 7. Generally, specimen wear was limited to fabric fiber breakage. The disk surface did not significantly wear in any test. Since fiber breakage, compared to an abrasive type of fiber wear, dramatically changes the overall structure of the fabric surface resulting in higher seal leakage rates, it constitutes a catastrophic material failure limiting the useful life of the seal. Thus a measure of a tests success is based upon the extent of fiber breakage and the friction coefficient.

The friction coefficient for the bare AF-40 fabric sliding against the polished Inconel disk was 0.42±0.02 during initial testing at room temperature. Upon heating, the disk began to oxidize and the friction increased. At 850 °C the friction coefficient was very high, 1.25±0.06. As the specimens were cooled, the friction coefficient decreased gradually to about 0.60±0.03 at room temperature. Subsequent heating and cooling cycles produced friction coefficients of 0.60±0.03 at room temperature, which increased, once again, to 1.3±0.1 at 850 °C. Moderate fiber breakage occurred during these tests (fig. 8).

The friction coefficient for the AF-40 fabric was decreased by approximately a factor of two when a solid lubricant film had been applied. Fabric upon which 1000 Å Ag and 500 Å CaF_2 had been sputtered displayed friction coefficients which ranged from 0.30±0.02, at room temperature, to 0.60±0.03 at 850 °C. No fiber breakage was observed after these tests.

When gold was used as the fabric lubricant, the friction behavior was similar to the tests with silver and CaF_2. Friction coefficients ranged from 0.30±0.02, at room temperature, to 0.60±0.03 at 850 °C. No fiber breakage occurred when gold was used as the lubricant.

When the bare AF-40 was slid against a pure copper disk the friction coefficients ranged from 0.25±0.02, at room temperature, to 0.50±0.03 at 700 °C. When heated in air, however, the copper disk surface severely oxidized at temperatures above 450 °C (ref. 4). The oxide film spalled and was therefore not passivating. Thus, without a protective coating, copper is probably not a suitable counterface material. No fiber breakage was observed for these tests.

Boron nitride (BN) is not a good lubricant for this fabric. When slid in air the friction coefficient was 0.65 ±0.03, at room temperature, which increased to 1.4 ±0.1 at 850 °C. The fabric was also chemically attacked by the BN at elevated temperatures becoming very weak and brittle. This was probably because BN at temperatures above 500 °C, oxidizes to B_2O_3 which can act as a fluxing agent to the fabric.

DISCUSSION

When the AF-40, alumina-boria-silicate, fabric was slid against Inconel X750 without any lubrication, the friction was unacceptably high. The friction forces arising from the high friction coefficients lead to excessive stresses in the fabric fibers and ultimately fiber fracture. By sputtering lubricants onto the fabric surface the friction was reduced by providing a low shear strength film between the ceramic fabric and the metal counterface. This lowered the stresses on the fibers and fracture was less likely to occur.

At elevated temperatures, the friction coefficients were twice as high as at room temperature. One factor may be that the metal oxides which formed at elevated temperatures were not very lubricious at room temperature. Another contributing factor may be that the thin sputtered lubricant films diffused into the fabric away from the sliding contact upon heating. Clearly, more lubricants present in the contact area may have further reduced friction. To test this hypothesis, a second lubrication method was investigated.

With this method, the fabric was impregnated with fine lubricant powder by placing a fabric test sample in a jar filled with Ag and CaF_2 powder. The jar was rolled to thoroughly impregnate the fabric with lubricants. The impregnated fabric sample was then removed from the jar and heated in a furnace at 950 °C, in air, to partially sinter the lubricants to achieve some bonding between the lubricants and the fabric fibers prior to sliding. The resulting fabric, however, was severely weakened by this treatment.

Subsequent visual examination of the specimens indicated that a chemical reaction between the lubricant powders and the fabric fibers had occurred. At 950 °C two reactions between boria and Ag and CaF_2 are possible (ref. 3). They are given below:

$$Ag + B_2O_3 \text{ ----- } B_2O_3\text{-}Ag_2O$$

$$CaF_2 + B_2O_3 \text{ ----- } B_2O_3\text{-}CaF_2$$

Since these reactions weaken the fabric, this lubrication method, at least with these lubricants, is unsatisfactory.

Nonetheless, no significant fiber weakening was observed for the fabric that has been sputtered with Ag and CaF_2 even after a 16 hr heat treatment at 950 °C. This was probably because the thin sputtered films constitute a much smaller volume than the impregnated lubricant powders. Therefore much less of the fabric's boria content reacted. Under these conditions this may be a satisfactory lubrication method.

Gold does not react with the fabric yet lubricated as well as silver. Thus gold may provide suitable fabric lubrication to at least 1000 °C even after following the powder lubrication method. These test have not been done.

An SEM analysis of the fabric and the disks after testing indicated that the mode of failure for the fabric is fiber fracture. No fiber abrasion was detected. EDS elemental analysis shows that no lubricants or fibers transferred to the disk surface but there was a transfer of nickel and chromium, or copper depending upon the test, to the fabric surface probably in the form of oxides. These materials may have provided a limited lubricating effect by separating the fabric from the metal counterface (ref. 5).

Clearly, the lubrication of the fabric is necessary to prevent fiber breakage and to reduce friction to acceptable levels. The chosen lubrication method must not only provide an adequate lubricant supply to the sliding contacts for the life of the seal but must also employ lubricants which do not adversely react with the seal fabric and cause its degradation.

CONCLUSIONS

1. The tribological properties of the AF-40 fabric were ascertained. The friction coefficient and the extent of fiber fracture are the significant parameters upon which to measure the success of a test. Counterface disk wear was too small to measure.

2. The friction coefficient was reduced by a factor of two and fiber breakage was prevented by applying a thin sputtered film of Ag and CaF_2 or gold on the fabric surface prior to sliding.

3. Boron nitride did not effectively lubricate the fabric. At elevated temperatures, the BN apparently oxidized and seemed to act as a flux for the ceramic fabric causing severe fabric degradation.

4. When present in large quantities, such as a powdered film, silver, CaF_2, and BN chemically attacked the fabric at elevated temperatures. This effect may be avoided by applying small quantities of the lubricants such as by using a sputtered film.

5. Gold shows promise as being a suitable solid lubricant for the AF-40 fabric. When used as a sputtered film it lowered friction by a factor of two and did not react with the fabric. Powdered lubrication with gold may further reduce the friction coefficient.

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TABLE I. - SPUTTERING PARAMETERS USED TO APPLY SOLID LUBRICANT FILMS TO THE FABRIC SURFACE

Coating material	Thickness Å	Sputtering time, min	Power kW	Argon Flowrate, cc/min	Chamber pressure, mTorr
CaF_2	500	17	1	12	10
Ag	1000	0.5	0.5	12	10
Au	1500	10.0	0.05	≈10	50

TABLE II. - FRICTION AND WEAR DATA SUMMARY FROM FABRIC TRIBOTESS

Fabric coating or lubricant treatment	Disk material	Friction coefficient		Fabric fiber breakage
		25 °C	850 °C	
none	Inconel X750	0.60±0.03	1.30±0.05	Moderate
none	Copper	0.30±0.02	0.50±0.03	None
1000 Å Ag	Inconel X750	0.30±0.02	---------	None
500 Å CaF_2	Inconel X750	---------	0.60±0.03	None
1000 Å Ag + 500 Å CaF_2	Inconel X750	0.30±0.02	0.60±0.03	None
1500 Å Au	Inconel X750	0.30±0.02	0.60±0.03	None
≈0.1 mm BN	Inconel X750	0.05±0.03	1.30±0.05	Moderate

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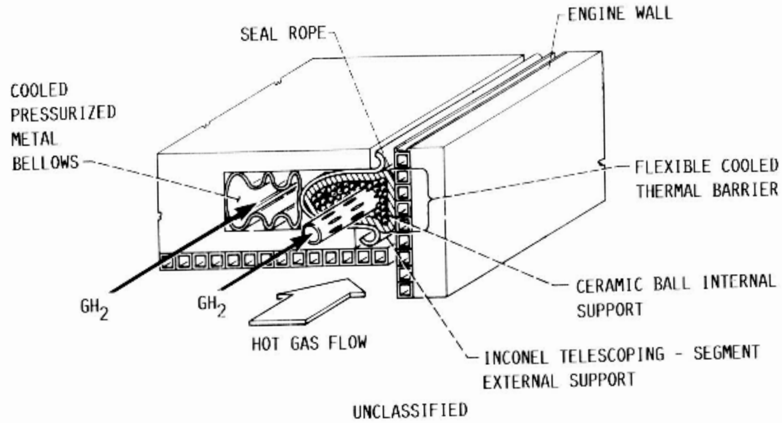

FIGURE 1. - CROSS-SECTION OF ENGINE SEAL.

FIGURE 2. - VIRGIN FABRIC BUNDLE 50x.

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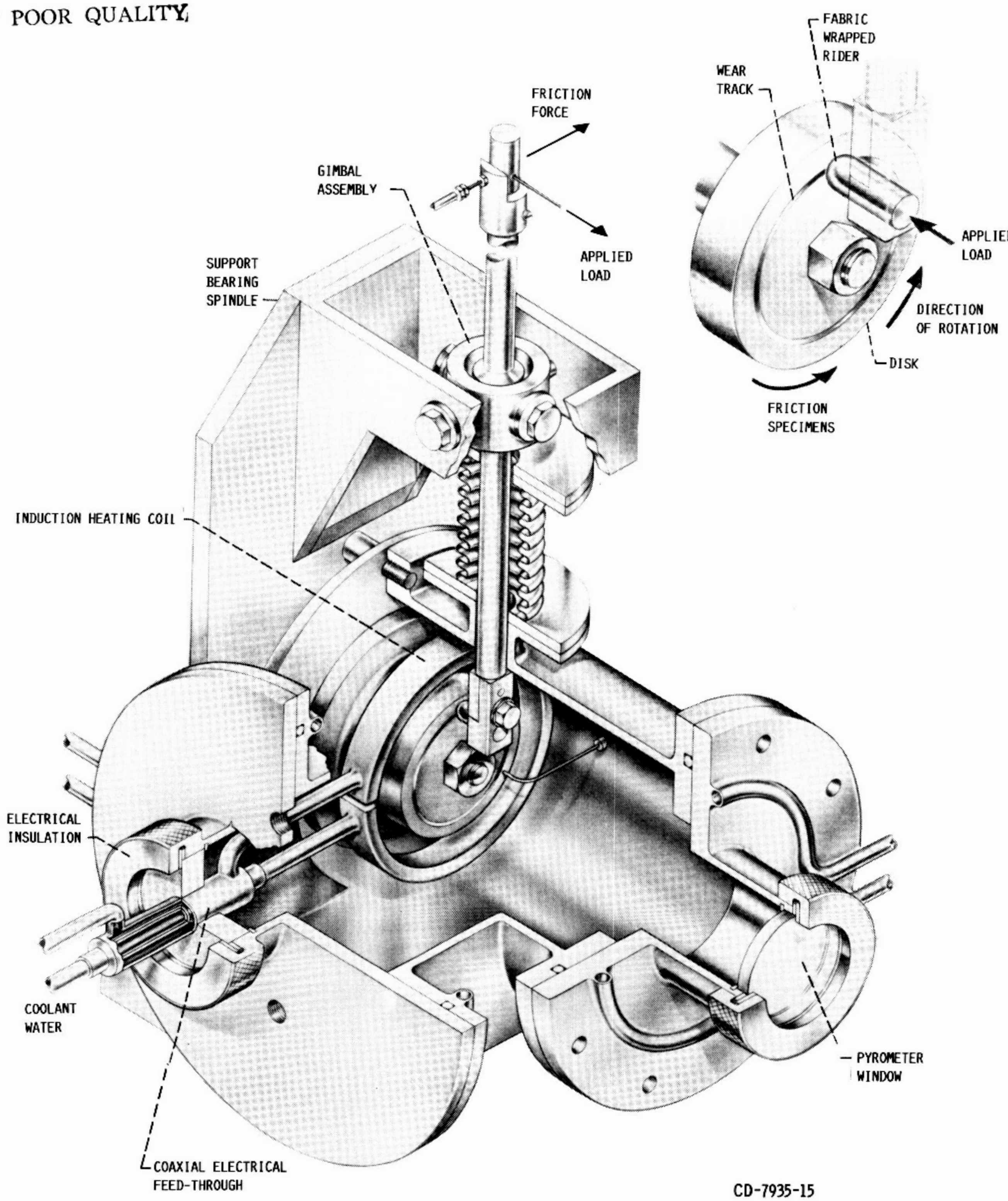

FIGURE 3. - HIGH-TEMPERATURE FRICTION APPARATUS.

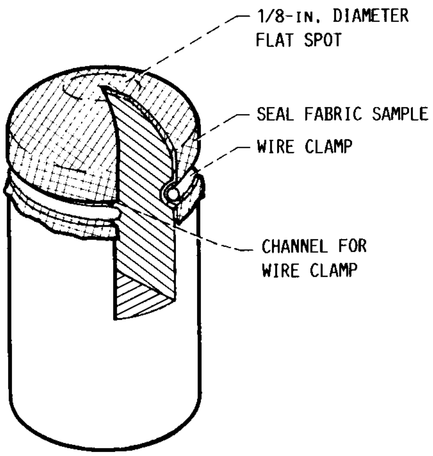

FIGURE 4. - THE PIN TEST SPECIMEN.

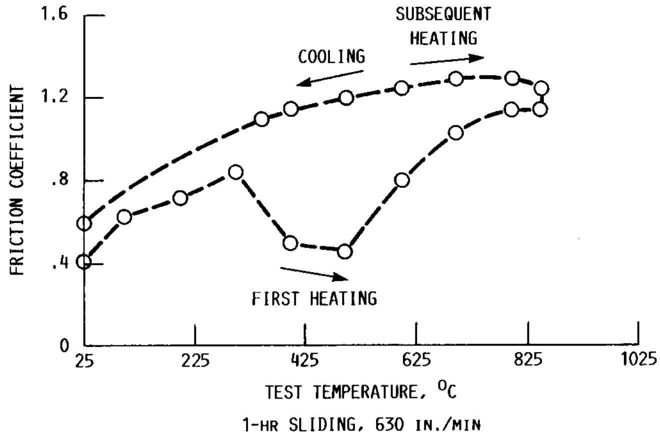

FIGURE 5. - FRICTION COEFFICIENT FOR FABRIC SLIDING AGAINST INCONEL X750 WITH NO LUBRICANT COATINGS.

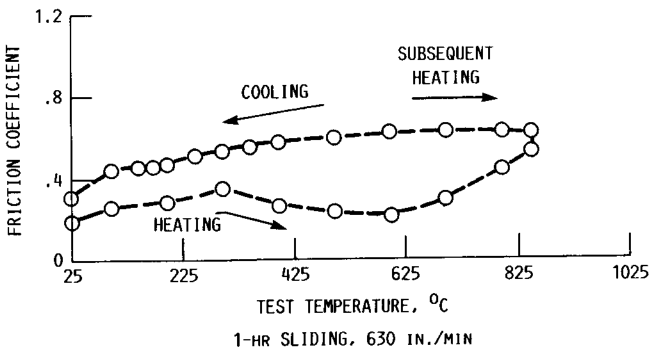

FIGURE 6. - FRICTION COEFFICIENT FOR THE FABRIC WITH Ag AND CaF_2 LUBRICANT COATING SLIDING AGAINST INCONEL X750.

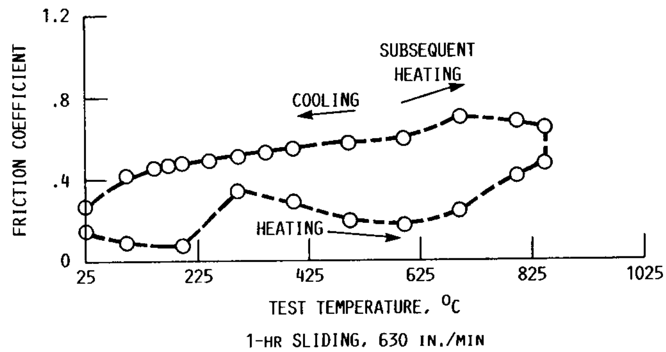

FIGURE 7. - FRICTION COEFFICIENT FOR THE FABRIC WITH GOLD COATING SLIDING AGAINST INCONEL X750.

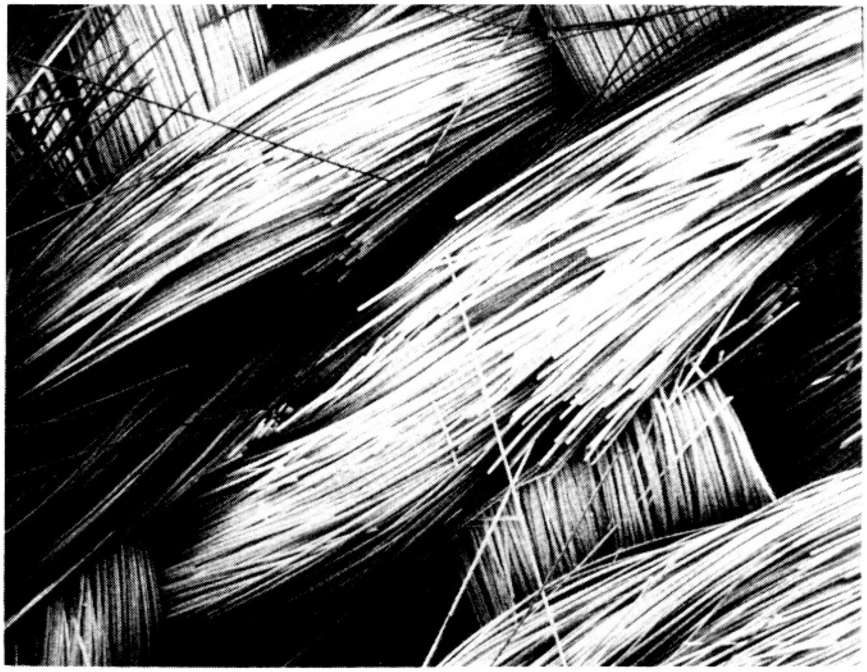

FIGURE 8. - FABRIC FRACTURE AFTER SLIDING AGAINST INCONEL X750. NO LUBRICANT COATINGS APPLIED. (TOP PHOTO 10X, LOWER PHOTO 50X.)

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National Aeronautics and Space Administration

Report Documentation Page

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
NASA TM-100806		

4. Title and Subtitle	5. Report Date
Tribological Properties of Alumina-Boria-Silicate Fabric from 25 to 850 °C	March 1988
	6. Performing Organization Code
7. Author(s)	8. Performing Organization Report No.
Christopher DellaCorte	E-3986
	10. Work Unit No.
	505-63-1A
9. Performing Organization Name and Address	11. Contract or Grant No.
National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135-3191	
	13. Type of Report and Period Covered
12. Sponsoring Agency Name and Address	Technical Memorandum
U.S. Department of Energy Office of Vehicle and Engine R&D Washington, D.C. 20545	14. Sponsoring Agency ~~Code~~Report No. DOE/NASA/50162-2

15. Supplementary Notes

Final Report. Prepared under Interagency Agreement DE-AI01-86CE50162.

16. Abstract

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17. Key Words (Suggested by Author(s))	18. Distribution Statement
Ceramics; Ceramic lubricants; Solid lubricants; Sliding seals; Seal lubricants	Unclassified – Unlimited Subject Category 27 DOE Category UC - 96

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No of pages	22. Price*
Unclassified	Unclassified	11	A02

NASA FORM 1626 OCT 86

*For sale by the National Technical Information Service, Springfield, Virginia 22161