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Polyimides Formulated from a Partially Fluorinated Diamine for Aerospace Tribological Applications

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POLYIMIDES FORMULATED FROM A PARTIALLY FLUORINATED DIAMINE
FOR AEROSPACE TRIBOLOGICAL APPLICATIONS

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ABSTRACT

Preliminary tribological studies on new polyimides formulated from the diamine 2,2-bis [4-(4-aminophenoxy)phenyl] hexafluorapane (4-BDAF) indicate that polyimides formulated from this diamine have excellent potential for high temperature tribological applications. The dianhydrides used to make the polyimides were pyromellitic acid (PMDA) and benzophenonetetracarboxylic acid (BTDA). Friction and wear studies at 25° and 200° C indicate that polyimides formulated using 50 mole percent of the PMDA dianhydride and 50 mole percent of the BTDA dianhydride perform better than polyimides formulated solely with the BTDA dianhydride. Graphite fiber reinforced polyimide (GFRPI) composites were formulated with the polyimide made from the BTDA dianhydride, both graphitic and non-graphitic fibers were evaluated. Graphitic fibers produced better tribological results, since thin, flowing, "layer-like" transfer films were produced which did not build-up with long sliding durations. Non-graphitic fibers did not produce this type of transfer.

INTRODUCTION

Considerable research has been conducted on carbon and graphite fiber reinforced polyimides (1-17), and very good tribological results have been reported. Therefore these composites offer outstanding potential for self-lubrication where parts are in sliding contact, especially at elevated temperatures. In the aerospace industry, potential uses include liners for

self-aligning plain spherical bearings, cages and races for ball and roller bearings, and as a seal material for sliding contact seals. Carbon or graphite fibers improve the strength and stiffness of composite while also providing lubrication. Polyimide provides the matrix material for holding the fibers, and under certain conditions (such as at elevated temperatures (18)) can improve the friction and wear properties of the composite.

Polyimide is a generic designation and refers to a class of long-chain polymers which have repeated imide groups in the main chain. By varying the monomeric starting materials, polyimides of different chemical composition and structure can be formulated. Each different polyimide possesses its own physical and chemical properties such as: different elastic modulus, different thermal stability, and different glass transition temperatures (T_g). In the aerospace industry, the desire is to operate at the highest possible temperature because of increased efficiency. Thus, for aerospace tribological applications, the polyimide selected to make composite materials should have the highest possible thermal stability.

In 1975, a polyimide was formulated from partially fluorinated polyimide resins prepared from the diamine 2,2-bis [4-(4-aminophenoxy)phenyl] hexafluoropropane (4-BDAF)(19), which possessed great potential for long term service in highly oxidative environments up to 370° C (20,21). Because of the promise shown in the preliminary physical properties testing, two polyimides were formulated using the 4-BDAF diamine for friction and wear evaluation. The polyimides were molded into hemispherically tipped pins and evaluated on a pin-on-disk tribometer.

GFRPI composites were also formulated from one of the polyimides and from either graphitic or non-graphitic carbon fibers. Friction, wear, wear surface morphology, and transfer film forming characteristics were studied and com-

pared to previous results on a GFRPI composite material, made from a different polyimide and different fiber, which demonstrated excellent tribological properties (both in pin-on-disk testing (15,17) and in spherical bearing testing (11 to 14)).

Robert J. Jones and Glen E. Chang of TRW supplied the polyimides and composites and provided helpful consultations. Edward A. Willis and Phillip R. Meng of NASA Lewis initiated the project.

MATERIALS

Two new polyimides based upon a novel aromatic diamine (2,2-bis [4-(4-aminophenoxy)] hexafluoropropane) (19) were evaluated. The diamine and the dianhydrides used to formulate these polyimides are shown in figure 1. One polyimide was prepared using benzophenonetetracarboxylic acid (BTDA) as the dianhydride, and is designated 4-BDAF/BTDA. The other polyimide was a copolyimide which consisted of 50 mole percent of the previous polyimide and 50 mole percent of a polyimide made from the dianhydride, pyromellitic acid (PMDA). This polyimide is designated 4-BDAF/50BTDA:50PMDA. The preparation of the 4-BDAF diamine and the polyimides made from it is described in references 20 and 21.

In addition to the two polyimide materials, two composites were formulated using the 4-BDAF/BTDA polyimide as the matrix material. One composite was made using a 50 weight percent of a high modulus graphitic carbon fiber (designated type "HG"), and the other was made using 50 weight percent of a high modulus non-graphitic carbon fiber (designated type "HC"). Properties of these fibers are listed in table 1. These composites were compared to a GFRPI composite which was previously evaluated (15,17), and which was made from commercially available polyimide (type "A") and from a low modulus graphitic carbon fiber (designated as fiber type "LG" in table 1).

The polyimide or the polyimide composite was molded into pins 2.0 cm long and 0.95 cm in diameter. A 0.476 cm radius hemisphere was machined on one end. The hemispherically tipped end was slid against a AISI 440C HT (high temperature) stainless steel disk hardened to Rockwell C-60. The disks were lapped and polished to a centerline average (cla) surface finish of 10^{-7} meter (4 μ in). Cleaning with levigated alumina did not change this value.

APPARATUS

A diagram of the high-temperature pin-on-disk tribometer used in this investigation is shown in figure 2. The loads were applied through a lever arm and the same lever arm was also used to transmit the friction force to a strain gage whose output was continuously recorded on a strip-chart recorder (fig. 2). The disks were heated by induction heating. The temperature was monitored by a thermocouple when the disk was not rotating and by an infrared optical pyrometer when it was in motion. The friction specimens were enclosed in a chamber in order to control the atmosphere.

PROCEDURE

Specimen Cleaning

The AISI 440C HT stainless steel disks were cleaned by washing with ethyl alcohol and then by scrubbing with a water paste of levigated alumina. They were then scrubbed with a brush under running distilled water to remove the levigated alumina, and dried with clean compressed air.

The polyimide or polyimide composite pins were scrubbed with a non-abrasive detergent, rinsed with distilled water and dried with clean compressed air.

Experimental Testing

The pin and disk specimens were inserted into the test apparatus and the chamber was then sealed. A controlled moist air test atmosphere (10 000 ppm

H₂O) was purged through the chamber for 15 minutes before each test and continuously throughout the test. After purging, the disk was rotated at either 100 or 1000 rpm. Test temperatures of 25°, 100°, 200°, and 300° C were used in this study. The disk was slowly heated to the desired temperature using induction heating and held for 10 minutes at temperature to allow the temperature to stabilize. The load (a 9.8 N weight) was then gradually applied. The pin slid on a 5.2 cm diameter track on the disk, giving it a 0.27 cm/s sliding speed at 100 rpm or a 2.7 cm/s sliding speed at 1000 rpm.

At various intervals during the experiments, the tests were stopped and the specimens removed. They were examined by optical microscopy and the wear volume was determined by measuring the wear scar on the hemispherically tipped pin and calculating the volume of material worn away. The pin was not removed from the holder and locating pins insured that it was returned to its original position when it was put back into the apparatus.

RESULTS AND DISCUSSION

Unfilled Polyimides

Friction coefficient as a function of sliding distance for 1 kilometer of sliding for the two different unfilled polyimide pins is plotted in figure 3. The sliding speed was 0.27 m/s (100 rpm) and two different temperatures were employed, 25° and 200° C. The friction coefficient is seen to be somewhat dependent on sliding distance and even more so on test temperature. Initially (for about 60 m of sliding), the 4-BDAF/50BTDA:50PMDA polyimide gave a lower friction coefficient than the 4-BDAF/BTDA polyimide at 25° C, but for most of the 1 km test it gave higher friction. A similar situation occurred at 200° C, but the friction remained lower for about 500 m of sliding. Also, it should be noted that the friction coefficient for both polyimides is lower at 200° C than at 25° C.

Polyimide pin wear rates were determined by measuring the wear scar diameter on the hemispherically tipped pins and calculating the volume of material removed. This volume was divided by the sliding distance to give wear rate units of wear volume per unit sliding distance. Wear rates for short sliding durations (0 to 1 km) are given in table 2 for the two polyimides at the two test temperatures of 25° and 200° C. The data indicates that from a wear viewpoint the 4-BDAF/50BTDA:50PMDA polyimide is superior to the 4-BDAF/BTDA polyimide; especially at 200° C, where the wear rate of this polyimide was an order of magnitude less. This result suggests that a polyimide made using 100 percent of the dianhydride PMDA may give additional improved wear results.

Graphite Fiber Reinforced Polyimides

The 4-BDAF/BTDA polyimide was reinforced with 50 weight percent of two different graphite fibers, types "HC" and "HG". Figure 4(a) gives the friction coefficients obtained with these two composites at 25° C as a function of sliding distance. Also shown for comparison is a GFRPI composite material which was evaluated previously but under different conditions (15,17). The polyimide was an additive polyimide (type "A") and the fiber was a low modulus graphitic fiber (type "LG").

The composite made from the type "A" polyimide and the type "LG" fibers gave the lowest friction coefficient after run-in. The friction coefficient of the composites formulated with the 4-BDAF/BTDA polyimide and type "HC" or type "HG" fibers were about the same after "run-in". One exception was a large increase in the friction coefficient for the composite formulated with the type "HC" fiber which occurred between 400 and 500 meters of sliding. A possible reason for this could be that the polyimide covered the fibers and dominated the friction process during this interval.

Wear volume as a function of sliding distance for the three composites evaluated is shown in figure 4(b). After an initial "run-in" (which lasted from 100 to 300 meters of sliding, wear tended to increase at a constant rate for each composite. The wear rates (taken from the slope of the lines in fig. 4(b)) were $2 \times 10^{-15} \text{ m}^3/\text{m}$, $3 \times 10^{-15} \text{ m}^3/\text{m}$, and $4 \times 10^{-15} \text{ m}^3/\text{m}$ for the type "HG" fiber reinforced 4-BDAF/BTDA polyimide, the type "HC" fiber reinforced 4-BDAF/BTDA polyimide, and the type "LG" fiber reinforced type "A" polyimide respectively.

To determine if sliding duration or sliding speed affected the tribological properties, the three composite materials were evaluated for sliding durations up to 90 km for both the 100 and 1000 rpm experiments. Figure 5 presents the data from the 1000 rpm experiments and table 2 summarizes the data from both the 100 and 1000 rpm experiments.

Figure 5 indicates that the composites formulated with type "HC" or type "LG" fibers tended to produce friction coefficients and wear rates that increased on long sliding durations for sliding speeds of 1000 rpm. The composite formulated with type "HG" fibers produced constant friction coefficients and wear rates under these same conditions.

Table 2 indicates that for short sliding durations the friction coefficients obtained at 1000 rpm are lower than those produced at 100 rpm but the wear rates are nearly equal for any given composite. As mentioned previously, for two of the composites at long sliding durations, the friction and wear increased dramatically. For long sliding durations at 100 rpm, however, the friction coefficients and wear rates remained relatively constant. Thus, whether speed or sliding duration had an effect on the friction and wear properties of these composites seemed to depend on the type of fiber used.

The best polyimide composite (4-BDAF/BTDA with type "HG" fibers) was evaluated at the elevated temperatures of 100°, 200°, and 300° C. The sliding speed was 2.7 m/s. The friction coefficient obtained as a function of sliding distance remained relatively constant at these temperatures. Figure 6(a) compares the friction coefficients obtained after 1 kilometer of sliding for the test temperatures of 25°, 100°, 200°, and 300° C. Also shown are friction coefficients from reference 17 for the type "A" polyimide reinforced with type "LG" fibers. The friction coefficient for both polyimide composites tended to increase with increasing temperature, although the 4-BDAF/BTDA composite showed a decrease in friction at 300° C. The type "A" composite tended to produce lower friction coefficients at this sliding distance than did the 4-BDAF/BTDA composites at all temperatures except at 300° C.

Wear rates for the composites at 25°, 100°, 200°, and 300° C are given in figure 6(b) and table 2. The rate tends to increase exponentially with increasing temperature. Considering experimental error and wear rate repeatability, the wear rates of the two composites can be assumed to be equal.

Wear Surface Morphology and Transfer at 25° C

Unfilled polyimides. - Photomicrographs of the 4-BDAF/50BTDA:50PMDA polyimide pin surface after 1 km of sliding at 100 rpm (0.27 m/s) at 25° C is shown in figure 7(a). The wear surface morphology of this polyimide was similar to the 4-BDAF/BTDA polyimide, except that more striations could be seen in the sliding direction and that the wear debris had a tendency to coalesce. In general, both surfaces were very smooth, but there were areas where voids existed. Most likely these were produced during the molding process rather than during the wear process. Very fine, birefringent powdery wear debris was found on both polyimide pin wear scars, indicating the wear particles were anisotropic crystalline.

A photomicrograph of a typical transfer film to the 440C HT stainless steel counterface is shown in figure 7(b). The transfer characteristics of both polyimides were very similar. Flat platelet-like particles tended to fill the low areas or valleys on the metallic surface. If any transfer difference did occur, it appeared that the 4-BDAF/50BTDA:50PMDA polyimide had slightly better flow properties than the 4-BDAF/BTDA polyimide.

Fiber reinforced polyimides. - A photomicrograph of typical wear surface morphology on the 4-BDAF/BTDA polyimide pin which was reinforced with type "HC" fibers is shown in figure 8(a). The polyimide and the fiber were worn to a very smooth finish. There are regions, however, where the polyimide tended to spall from the fibers. This indicates the bond between the fiber and the polyimide is poor.

Figure 8(b) shows a photomicrograph of typical wear surface morphology on the 4-BDAF/BTDA polyimide pin which was reinforced with type "HG" fibers. Considerable back transfer occurred, with the transferred material adhering predominately to the fibers. Some spalling on the pin wear surface was observed to occur, but the polyimide appeared to be more tightly bonded to the "HG" fibers than the "HC" fibers.

For long sliding distances at 1000 rpm with the 4-BDAF/BTDA polyimide reinforced with the type "HC" fibers, increasing friction and wear rates occurred. This was reflected in the surface morphology by cracks in the fibers and large regions spalling from the surface.

Figure 9(a) gives a photomicrograph of typical transfer to a 440C HT stainless steel counterface from the 4-BDAF/BTDA polyimide reinforced with type "HC" fibers for sliding speeds of 100 rpm. They are also typical of short sliding distances at 1000 rpm. The transfer tends to fill low areas (scratches, indentation, etc.), but does not produce plastically flowing films

on the higher regions. For longer sliding distances at 1000 rpm, thicker transfer is produced but not of a plastic nature.

Figure 9(b) gives a photomicrograph of typical transfer to a 440C HT stainless steel counterface from the 4-BDAF/BTDA polyimide reinforced with type "HG" fibers for all sliding speeds and distances at 25° C. Thin plastically flowing transfer occurred that covered the entire wear track surface. The thickness of the transfer was in the order of the wavelength of light (0.4 to 0.8 micrometers), and the transfer remained approximately this thickness for the duration of the experiment (90 km).

The type "A" polyimide reinforced with type "LG" fibers also demonstrated this type of transfer for short sliding distances; but as sliding distance increased, the transfer increased to "local" thickness values of greater than 10 micrometers. In reference 17, it was postulated that this increase in thickness was responsible for increases in adhesion between the surfaces, and thus increases in friction and wear.

Morphology and Transfer at Elevated Temperatures

Unfilled polyimides. - Photomicrographs of typical wear surface morphology of 4-BDAF/BTDA and 4-BDAF/50BTDA:50PMDA pins after 1 kilometer of sliding at 200° C is shown in figure 10. The 4-BDAF/BTDA pin is characterized by a rippled surface in the sliding direction and considerable back transfer (fig. 10(a)). The 4-BDAF/50BTDA:50PMDA pin wear surface is much smoother with only a faint hint of ripples and not a great deal of back transfer.

Transfer from the 4-BDAF/BTDA looks similar to that obtained with this polyimide at 25° C. The wear track and the area outside of the wear track took on a reddish appearance, which indicates that the 440C HT stainless steel has oxidized. This phenomenon occurred at all elevated temperature experiments and may be a reason for the higher friction and wear.

Transfer from the 4-BDAF/50BTDA:50PMDA polyimide was similar to that obtained with the 4-BDAF/BTDA polyimide reinforced with type "HG" fibers. Very thin, plastically flowing transfer films were obtained. It is interesting that at 25° C the wear rates are also very similar (table 2).

Fiber reinforced polyimides. - The 4-BDAF/BTDA polyimide reinforced with type "HG" fibers was evaluated at elevated temperatures. One dominating effect was evident, and that was as testing temperature increased, the polyimide tended to debond from the fibers at a faster rate. Figure 11 shows the wear surface morphology at 200° C which is typical of the elevated temperature experiments. The fibers seem to be relatively unaffected by temperature and as figure 11 indicates, they appear to be solely supporting the load. The type "A" polyimide reinforced with type "LG" fibers showed similar characteristics as the temperature was increased (17).

The transfer at 100° C was more continuous than found at 25° C, but not as thick. As temperature increased the transfer tended to fill only the low spots on the 440C HT stainless steel surface; and as the polyimide debonded from the fibers, the fibers tended to have a polishing effect on the high areas in the wear track with little transfer occurring.

Geometry Considerations

The composite specimens in this study were evaluated as hemispherically tipped pins sliding against 440C HT stainless steel counterfaces. This geometry was chosen since it closely approximates the geometry of a sliding contact seal, which could be a possible end use application of the composite.

It should be indicated that geometry can markedly affect the tribological properties of these composites. For example, in reference 17, the tribological properties of a type "A" polyimide with type "LG" fibers were compared for the case when the pin was made from the composite and slid against a 440C HT

stainless steel disk counterface and for the case when a 440C HT stainless steel pin was slid against a disk made of the composite. In both cases no measureable wear occurred to the metallic specimen, but the friction and wear properties of the composite were completely different. When the pin was made from the composite, friction and composite wear tended to increase with increasing temperature; but when the disk was made of the composite, friction tended to decrease and composite wear was constant with increasing temperature.

These results indicate the importance of carefully choosing the experimental parameters when comparing tribological materials. It also indicates that materials should be evaluated in such a manner that geometry is carefully considered.

REMARKS

This study was of a preliminary nature, designed to look at the potential of making polyimides and GFRPI composites from a new diamine (4-BDAF). Although a finite number of variables were investigated, the study gives some speculation as to the reasons for some phenomena and guidance for future work. For example, the BTDA dianhydride was chosen because it was thought it would produce polyimides with good processibility. The PMDA polyimide was chosen because of its higher thermal stability. The results indicate that the copolyimide made from a 50/50 mole ratio of the BTDA and PMDA dianhydrides, produces surface layers that are more plastic than with BTDA alone. One may speculate that a polyimide made from only PMDA will give a similar result and have higher thermal stability.

Graphite fibers are "sized" by the manufacturer to reduce the electrostatic charges on them and to improve the bonding with the matrix material. Generally, a given manufacturer uses his own polyimide (or other material such as an epoxy) for this purpose. It is reasonable to speculate that this sizing

material may be an important factor in determining the tribological properties of the composite. For instance, a high temperature composite may be stable only up to the stability limit of the sizing material, irregardless of the thermal stability of the polyimide matrix material used. Thus, in formulating polyimide composites, the fiber should be sized with the same polyimide as the matrix material.

Morphology studies of the GFRPI wear surfaces indicated that the surface density of fibers was not the same for the different fibers used. The composites were produced by the same vendor and had the same 1 to 1 ratio by weight of polyimide solids and fiber solids. The probable reason for the surface density differences is that the fibers had different densities. This factor may have affected the tribological properties of the composites. Closer attention to this will be made in the future.

SUMMARY OF RESULTS

Preliminary friction, wear and surface morphological studies of two new polyimides made by the polymerization of a new diamine (4-BDAF) with two different dianhydrides (BTDA and PMDA) and of two different graphite fiber composites made from one of the polyimides (4-BDAF/BTDA) indicate that:

1. A copolyimide formulated using 50 mole percent of each of the dianhydrides BTDA and PMDA exhibited better tribological properties than those formulated with BTDA alone, especially at 200° C.

2. The 4-BDAF/BTDA polyimide reinforced with 50 weight percent graphitic carbon fibers gave better tribological properties than the same polyimide reinforced with 50 weight percent of non-graphitic carbon fibers.

3. Friction coefficients and GFRPI wear rates at 1000 rpm tended to increase with sliding distance for the polyimide reinforced with the non-graphitic carbon fiber but they remained relatively constant for the polyimide

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reinforced with the graphitic carbon fiber. At 100 rpm both fibers produced relatively constant friction and wear rates.

4. The GFRPI wear rates tended to increase exponentially as a function of increasing temperature. Friction coefficient also increased as temperature increased.

5. Wear surface morphology of the GFRPI wear scar indicated that the polyimide tended to debond from the fibers, and the rate appeared to increase as temperature increased.

6. The graphitic carbon fiber produced thin (0.4 to 0.8 μm), plastically flowing transfer films; the non-graphitic carbon fiber did not.

REFERENCES

1. Giltrow, J. P. and Lancaster, J. K., "Properties of Carbon-Fibre-Reinforced Polymers Relevant to Applications in Tribology," International Conference on Carbon Fibres, Their Composites and Applications, Plastics Institute, London (1971) Paper 31.
2. Giltrow, J. P. and Lancaster, J. D., "Friction and Wear of Polymers Reinforced with Carbon Fibers," Nature, 214, 1106-1107 (1967).
3. Lancaster, J. K., "The Effect of Carbon Fibre Reinforcement on the Friction and Wear of Polymers," J. Phys. D., 1, 549-559 (1968).
4. Giltrow, J. P., "A Design Philosophy for Carbon Fibre Reinforced Sliding Components," Tribology, 4 (1), 21-28 (1971).
5. Harris, C. L. and Wyn-Roberts, D., "Wear of Carbon Fibre Reinforced Polymers in a High Vacuum Environment," Nature, 217, 981-982 (1968).
6. Giltrow, J. P. and Lancaster, J. K., "Carbon-Fibre Reinforced Polymers as Self-Lubricating Materials," Tribology Convention, Pithochry, Scotland, May 15-17, 1968, Proceedings, Institution of Mechanical Engineer (1968), pp. 149-159.

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OF POOR QUALITY

7. Giltrow, J. P. and Lancaster, J. K., "The Role of the Counterface in the Friction and Wear of Carbon Fibre Reinforced Thermosetting Resins," Wear, 16 (11), 359-374 (1970).
8. Simon, R. A. and Prosen, S. P., "Graphite Fiber Composites; Shear Strength and Other Properties," Twenty-Third Annual Technical Conference, SPI Reinforced Plastics Composite Division, Proceedings, Society of the Plastics Industry, Inc., Section 16-B (1968), pp. 1-10.
9. Herrick, J. W., "Bearing Materials from Graphite Fiber Composites," Reinforced Plastics - Ever New; Proceedings of the Twenty-Eighth Annual Technical Conference, Society of the Plastics Industry, Inc., (1973), pp. 17-D-1 to 17-D-6.
10. Giltrow, J. P., "The Influence of Temperature on the Wear of Carbon Fiber Reinforced Resins," ASLE Trans., 16 (2), 83-90 (1973).
11. Sliney, H. E. and Johnson, R. L., "Graphite Fiber-Polyimide Composites for Spherical Bearings to 340° C (650° F)," NASA TN D-7078 (1972).
12. Sliney, H. E., Jacobson, T. P., and Munsen, H. E., "Dynamic Load Capacities of Graphite-Fiber - Polyimide Composites in Oscillation Plain Bearings to 340° C (650° F)," NASA TN D-7880 (1975).
13. Sliney, H. E. and Jacobson, T. P., "Performance of Graphite Fiber-Reinforced Polyimide Composites in Self-Aligning Plain Bearings to 315° C," Lubr. Eng., 31 (12), 609-613 (1975).
14. Sliney, H. E. and Jacobson, T. P., "Some Effects of Composition on Friction and Wear of Graphite-Fiber-Reinforced Polyimide Liners in Plain Spherical Bearings," NASA TP-1229 (1978).
15. Fusaro, R. L. and Sliney, H. E., "Friction and Wear Behavior of Graphite Fiber Reinforced Polyimide Composites," ASLE Trans., 21 (4), 337-343 (1978).

ORIGINAL PAGE IS
OF POOR QUALITY

16. Lancaster, J. K., "Geometrical Effects on the Wear of Polymers and Carbons," J. Lubr. Technol., 97 (2), 187-194 (1975).
17. Fusaro, R. L., "Geometrical Aspects of the Tribological Properties of Graphite Fiber Reinforced Polyimide Composites," NASA TM-82757 (1982).
18. Fusaro, R. L., "Polyimide Film Wear - Effect of Temperature and Atmosphere," NASA TN D-8231 (1976).
19. Zakrezewski, G., O'Rell, M. K., Vaughan, R. W., and Jones, R. J., "Development of Autoclave Moldable Addition-Type Polyimide," TRW Systems Group, TRW-26446-6015-RU-00, NASA CR-134900 (1976).
20. Jones, R. J., O'Rell, M. K., and Hom, J. M., "Polyimides Prepared from Perfluoroisopropylidene Diamine," U.S. Patent 4,111,906 (1978).
21. Jones, R. J., O'Rell, M. K., and Hom, J. M. "Fluorinated Aromatic Diamine," U.S. Patent 4,203,922 (1980).

TABLE 1. - TYPICAL FIBER PROPERTIES

Property or characteristic	Graphite fiber Type "LG"(a)	Carbon fiber	
		Type "HC"(b)	Type "HG"(c)
Tensile strength	6.2×10^8 N/m ²	1.6×10^9 N/m ²	1.9×10^9 N/m ²
Elastic modulus	3.0×10^{10} N/m ²	2.6×10^{11} N/m ²	5.2×10^{11} N/m ²
Length	6×10^{-3} m	6×10^{-3} m	6×10^{-3} m
Diameter	8×10^{-6} m	8×10^{-6} m	6×10^{-6} m
Specific gravity	1.4	1.75	1.96
Graphitic	Yes	No	Yes
Carbon assay	99.5 percent	99 percent	99.9 percent

- (a) Low modulus, graphitic
 (b) High modulus, nongraphitic
 (c) High modulus, graphitic

TABLE 2. - SUMMARY OF EXPERIMENTAL RESULTS

Polyimide type	Fiber type	Temperature, °C	Sliding speed, rpm	Average friction coefficient		Average wear rates (m ³ /m x 10 ⁻¹⁵) [Wear volume per unit sliding distance]	
				Short sliding durations	Long sliding durations	Short sliding durations	Long sliding durations
4-BDAF/BTDA	None	25	100	0.53	----	150	----
		200	100	.28	----	550	----
4-BDAF/5OBTDA: 5OPMDA	None	25	100	0.65	----	35	----
		200	100	.40	----	46	----
4-BDAF/BTDA	"HC"	25	100	0.43	0.50	3	4
		25	1000	.28	^a .60	3.5	21
4-BDAF/BTDA	"HG"	25	100	0.38	0.36	2	1.5
		25	1000	.32	.30	2	2
		100	1000	.35	.50	11	27
		200	1000	.50	----	54	----
		300	1000	.35	----	420	----
Type "A"	"LG"	25	100	0.26	0.35	4	1.5
		25	1000	.20	^a .60	3.5	12
		100	1000	.30	.35	18	32
		200	1000	.49	----	68	----
		300	1000	.55	----	190	----

^aFriction coefficient increasing with increasing sliding distance.

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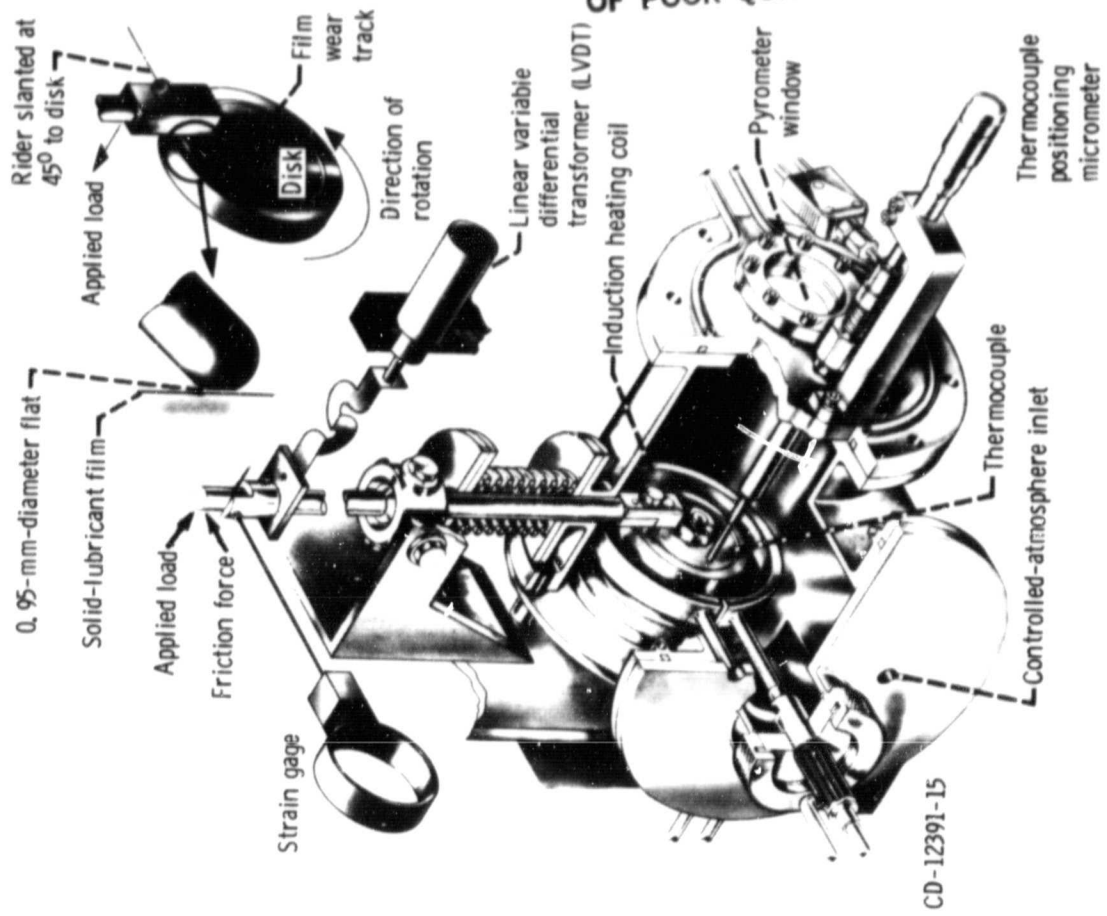


Figure 2. - Pin-on-disk tribometer.

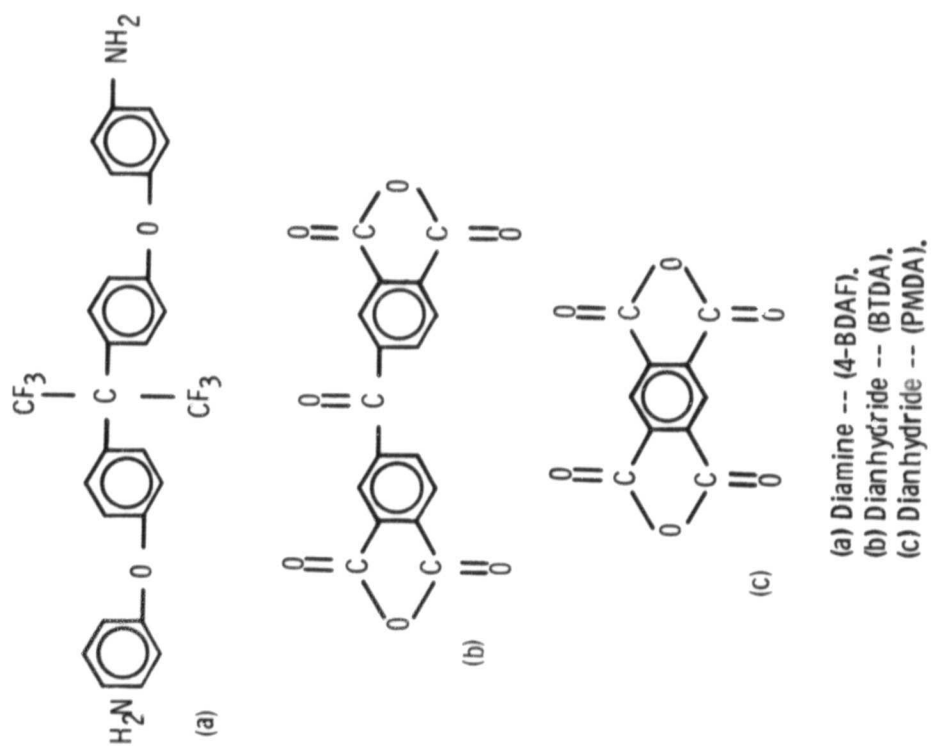


Figure 1. - Monomers used to formulate two new polyimides.

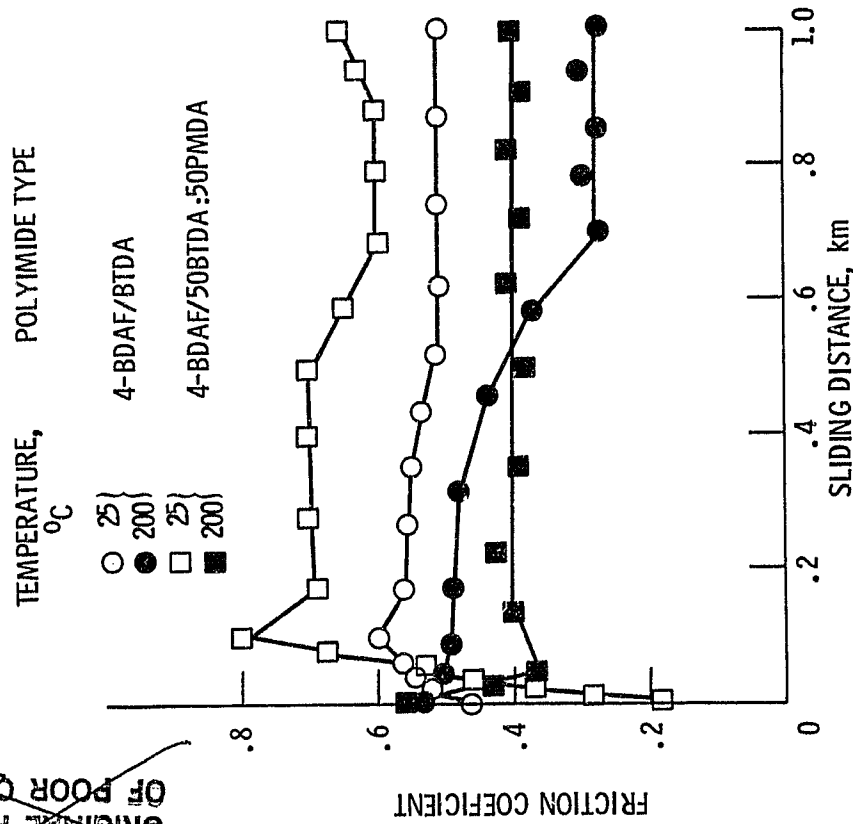


Figure 3. - Friction coefficient as a function of sliding distance for 4-BDAF/BTDA and 4-BDAF/50 BTDA : 50 PMDA unfilled polyimide riders sliding against a 440C HT stainless steel counterface at 0.27 m/s (100 rpm).

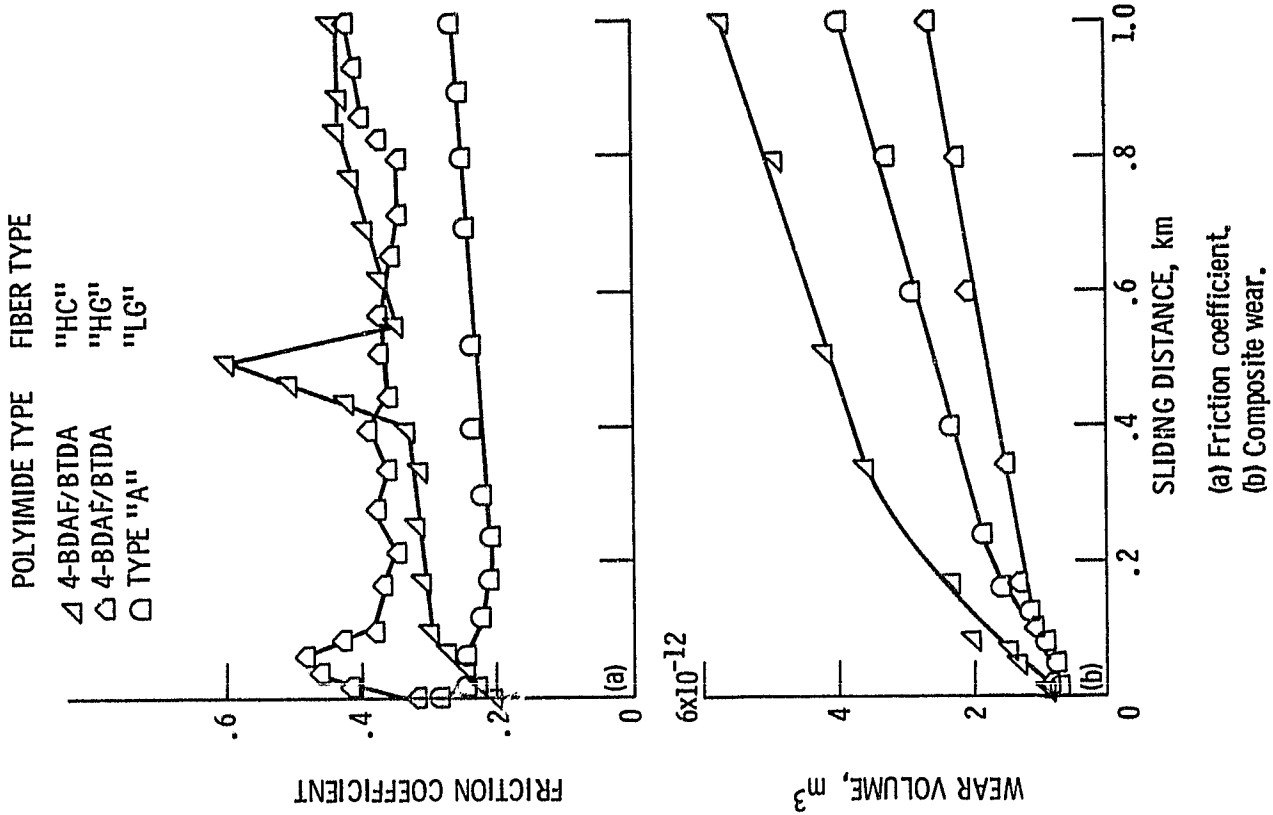


Figure 4. - Friction coefficient and composite wear volume as a function of sliding distance for three fiber reinforced polyimide composite riders sliding against a 440C HT stainless steel counterface at 25°C and 0.27 m/s (100 rpm).

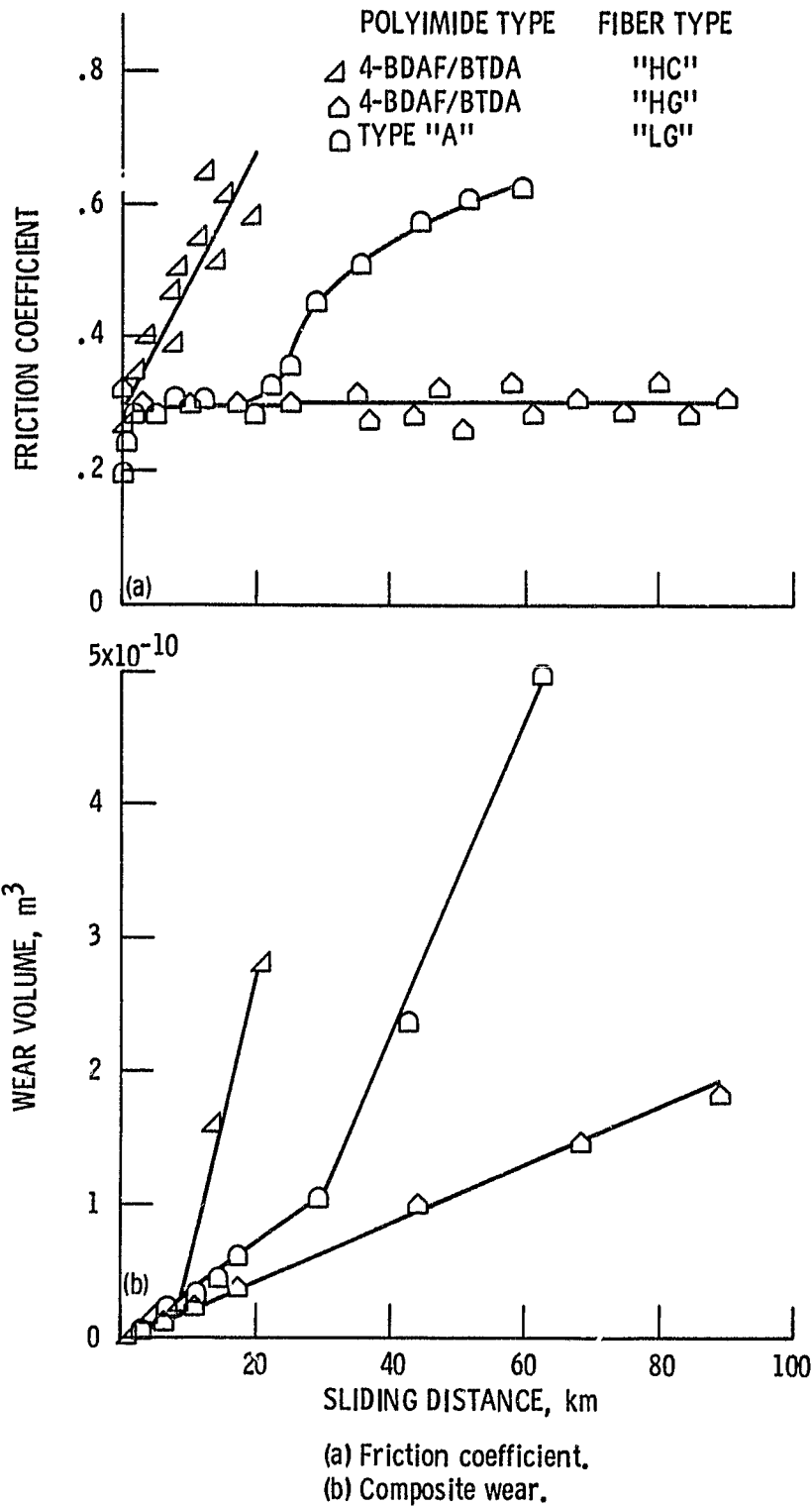


Figure 5. - Friction coefficient and composite wear as a function of long sliding distances for carbon fiber reinforced polyimide composite riders sliding against 440C HT stainless steel counterfaces at 25^o C and 2.7 m/s (1000 rpm).

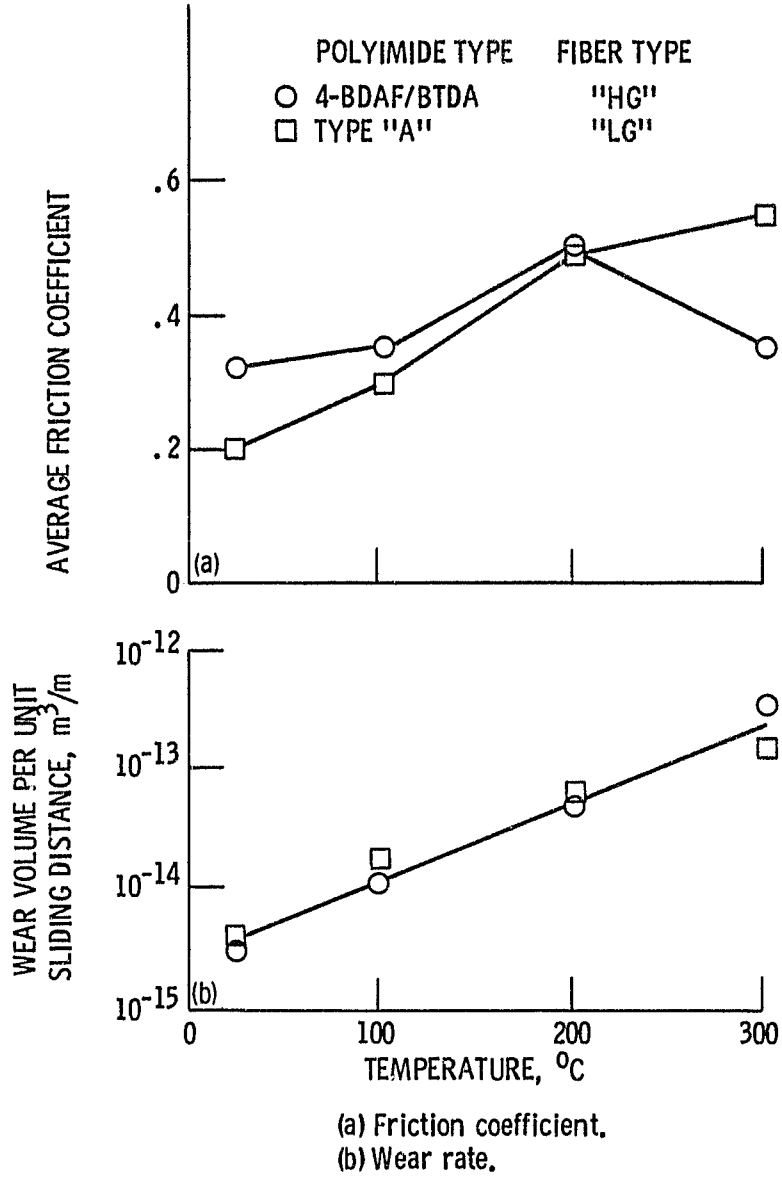
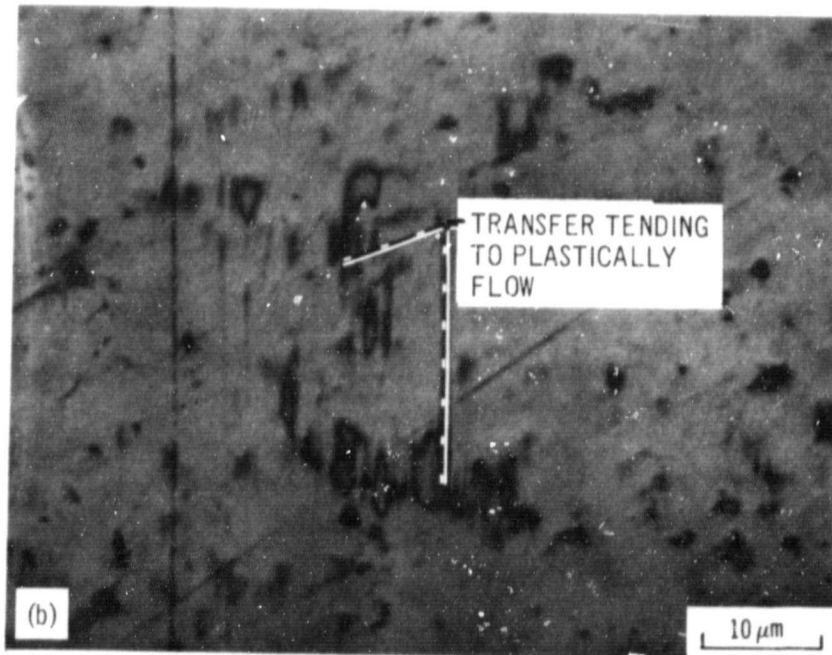
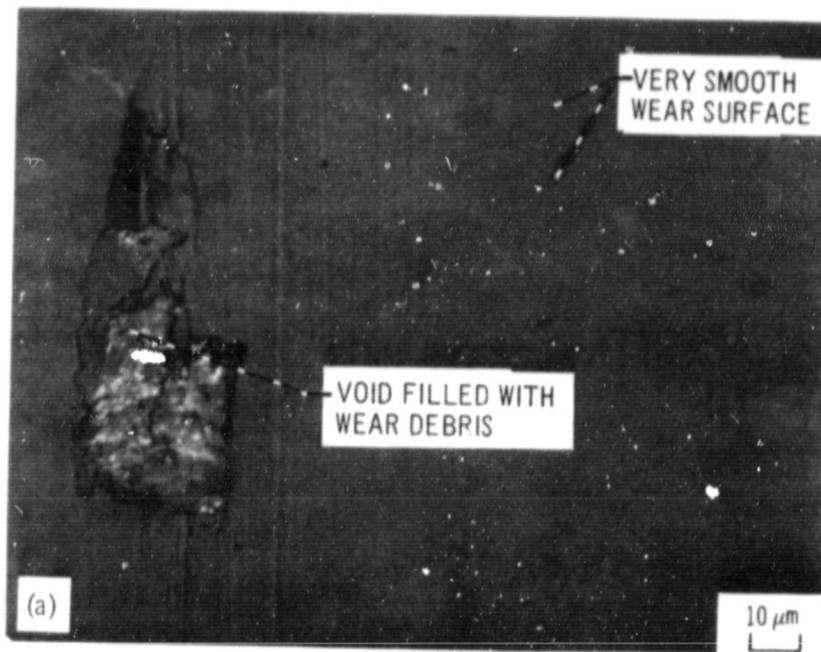


Figure 6. - Effect of temperature on the average friction coefficient and wear rate of carbon fiber reinforced polyimide composite riders for sliding intervals of 0 to 1 kilometer at 2.7 m/s (1000 rpm).

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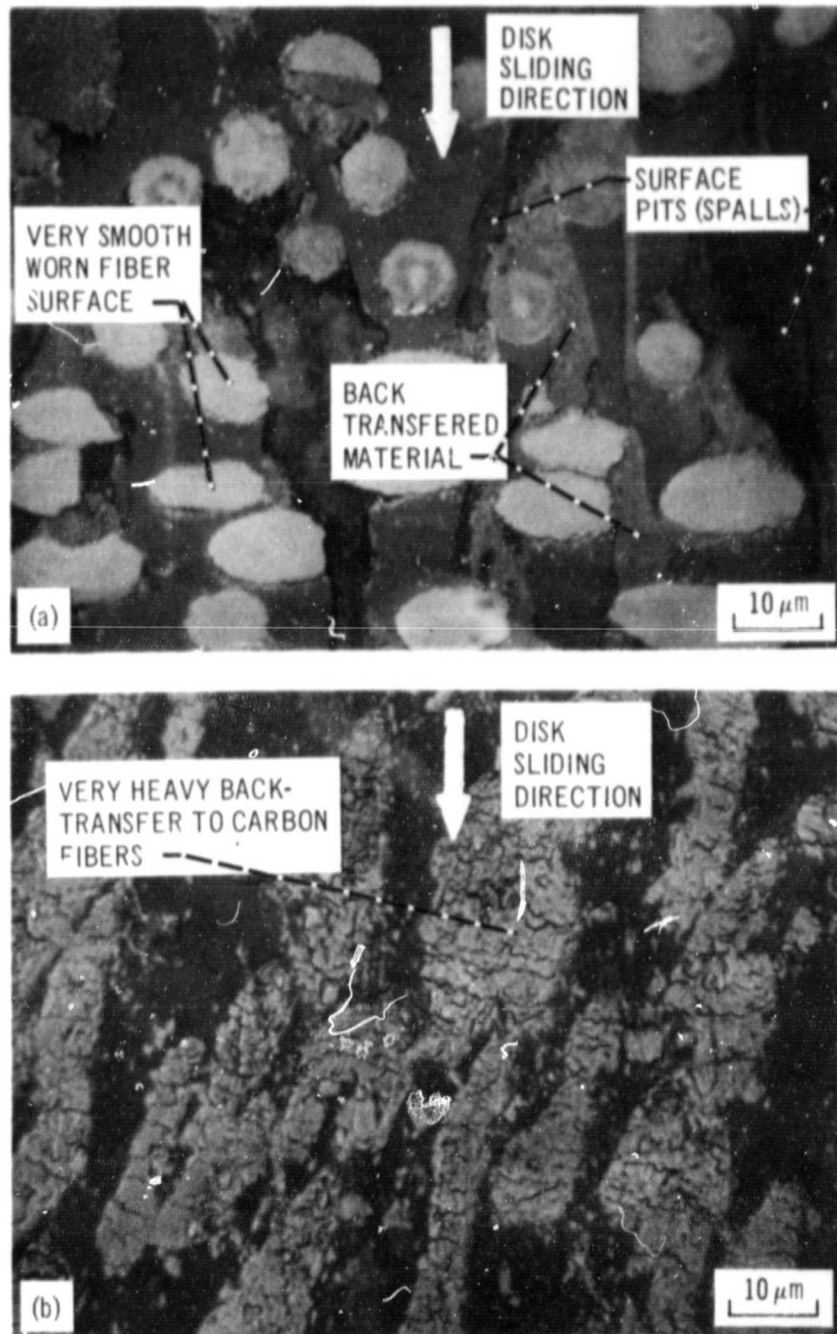


(a) PIN WEAR SURFACE MORPHOLOGY.

(b) TRANSFER TO 440C HT STEEL COUNTERFACE.

Figure 7. - Photomicrographs of the wear surface morphology and transfer films produced from 4-BDAF/50BTDA:50PMDA pins sliding against 440C HT stainless steel counterfaces at 25° C.

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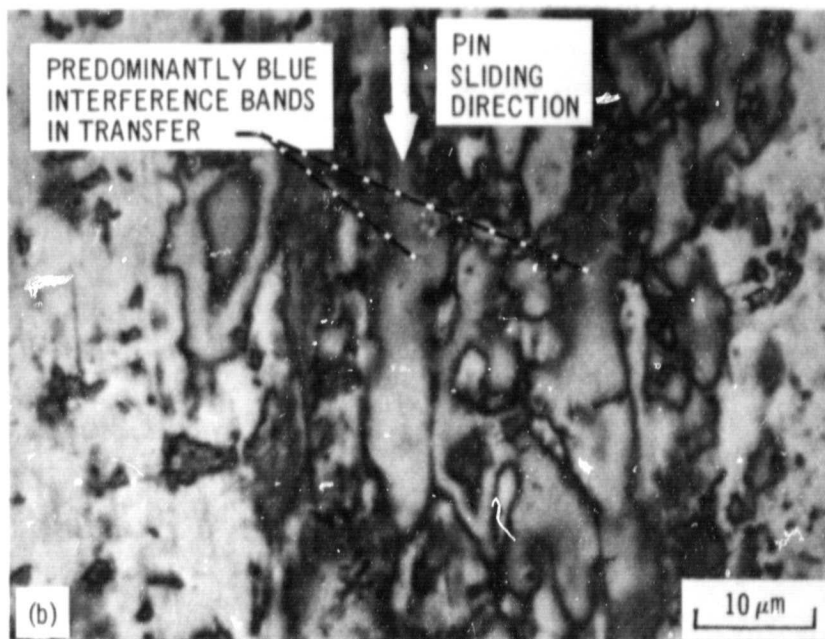
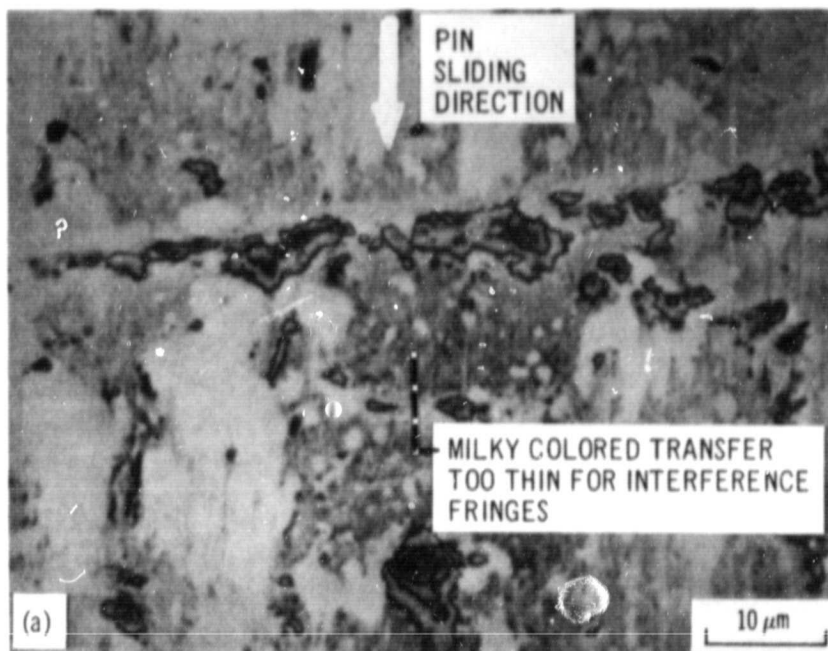


(a) NON-GRAPHITIC CARBON FIBER REINFORCED POLYIMIDE.

(b) GRAPHITIC CARBON FIBER REINFORCED POLYIMIDE.

Figure 8. - Photomicrographs of typical wear surface morphology of non-graphitic carbon fiber (type "HC") and graphitic carbon fiber (type "HG") reinforced polyimide (type 4-BDAF/BTDA) composite pins sliding against 440C HT stainless steel counterfaces at 25° C.

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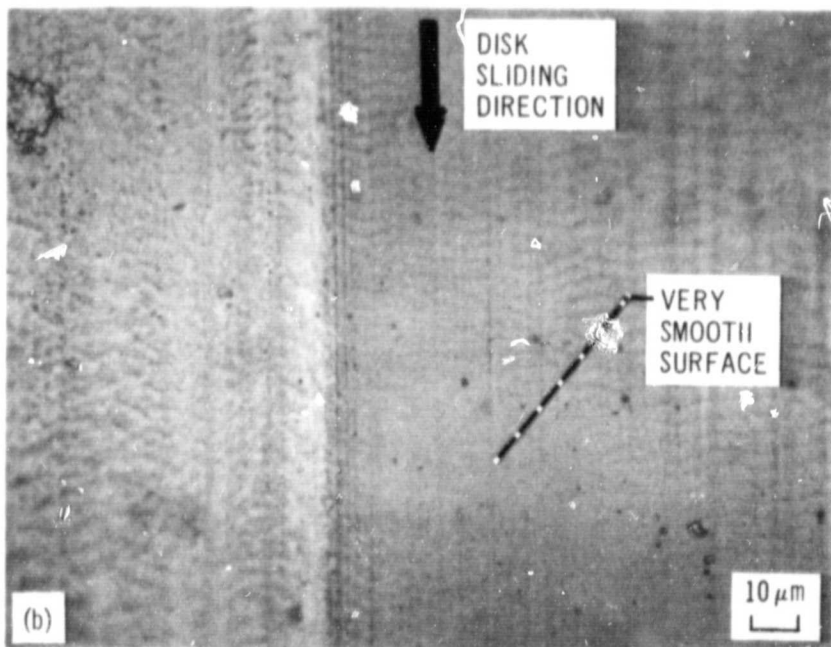
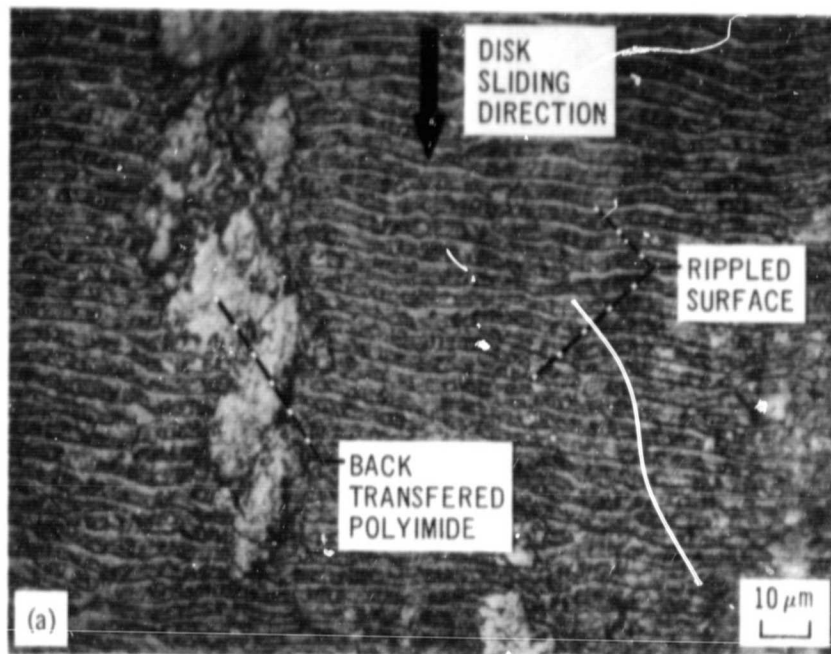


(a) TRANSFER FROM TYPE "HC" COMPOSITES.

(b) TRANSFER FROM TYPE "HG" COMPOSITES.

Figure 9. - Photomicrographs of typical transfer films to 440C HT stainless disks from non-graphitic carbon fiber (type "HC") and graphitic carbon fiber (type "HG") reinforced polyimide (type 4-BDAF/BTDA) pins at 25° C.

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(a) 4-BDAF/BTDA PIN.

(b) 4-BDAF/50BTDA:50PMDA PIN.

Figure 10. - Photomicrographs of the wear surface morphology of 4-BDAF/BTDA and 4-BDAF/50BTDA:50PMDA polyimide pins after 1 kilometer of sliding at 200° C against 440C HT steel counterfaces.

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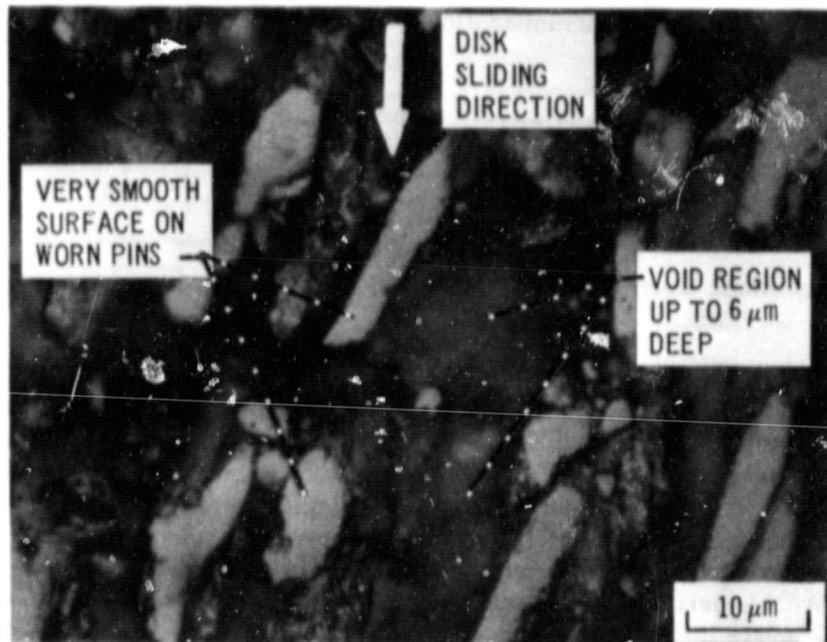


Figure 11. - Photomicrograph of the wear surface morphology of graphitic carbon fiber (type "HG") reinforced polyimide (type 4-BDAF/BTDA) composite pins after 1 km of sliding at 200°C against a counterface of 440C HT stainless steel.