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EFFECT OF THERMAL AGING ON THE TRIBOLOGICAL PROPERTIES OF POLYIMIDE FILMS AND POLYIMIDE-BONDED GRAPHITE FLUORIDE FILMS

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ABSTRACT

The effect of thermal aging on the weight loss, adherence, friction and wear of polyimide films and polyimide-bonded graphite fluoride films applied to 440C-HT stainless steel disks and to 304 stainless steel thin foils was studied. The films were exposed at temperatures of 315°, 345°, 370°, or 400° C for 100 hours or more and then evaluated at temperatures of 25°, 315°, or 345° C in atmospheres of dry or moist air. Polyimide films were found to be brittle after thermal exposure; but polyimide-bonded graphite fluoride films possessed good adherence and gave low friction and wear results. Thus, polyimide-bonded graphite fluoride films appear to be good candidates for solid lubrication applications where long thermal soaks are prevalent.

INTRODUCTION

In general, solid lubricants are employed in situations where oils and greases are not functional. One of the newest applications for solid lubricants is in gas bearings. One type of gas bearing is a compliant-surface bearing, which employs flexible members such as foils (1-5). Rubbing contact can occur in these bearings at startup, at shutdown, or when dynamic loading occurs. The function of the solid lubricant is to minimize the wear during these intervals.

In applications related to aircraft (1), these bearings operate at relatively high temperatures. One material currently used as a solid lubricant in these bearings is a polytetrafluoroethylene-based film that has an upper temperature limit of about 250° C. For a number of reasons, including efficiency, there would be an advantage to operating these bearings at temperatures higher than the polytetrafluoroethylene-based film would allow. For this reason, polyimide films and polyimide-bonded graphite fluoride films were evaluated to determine if they might be considered as candidates for foil bearing lubricants. Previous tests on these films had disclosed that they possess very good friction and wear properties in air up to 400° C (6-10).

The conditions under which the solid lubricant films must operate in foil bearings are fairly stringent. While the loads are relatively light, the films must be able to withstand long soaks at elevated temperatures and they must be able to adhere to flexible, compliant bearing surfaces. Thus, an experimental program was devised to determine the effect of thermal aging on the weight loss, adherence, friction coefficient, and wear properties of the films.

The films were exposed at temperatures of 315°, 345°, 370°, or 400° C for 100 hours or more and the rate of film weight loss was determined. The effect of surface finish on the weight loss rate and adherence of the films was also investigated. The friction and wear tests were conducted on the thermally aged films at temperatures of 25°, 315°, and 345° C and the results were compared to similar tests on non-aged films. The tests were stopped at intervals of 15, 60, and 250 kilocycles of sliding and the sliding surfaces were examined by optical microscopy and photographed. Surface profiles of the film wear tracks were also taken. The experimental conditions used were a load of 1 kilogram, a linear sliding speed of 2.6 meters per second (1000 rpm), and controlled atmospheres of either moist air (10 000-ppm H_2O) (approx. 50 percent relative humidity at ambient temperatures) or dry air (<20-ppm H_2O).

MATERIALS

Pyralin polyimide (PI-4701) was used in this study. The polyimide was obtained as a thick precursor solution. To provide a sprayable mixture, a thinner consisting of N-methyl-pyrrolidone and xylene was added to it.

The polyimide-bonded graphite fluoride films were prepared by mixing equal parts by weight of polyimide solids with graphite fluoride powder. The graphite fluoride used had a fluorine-carbon ratio that varied from 1.0 to 0.85.

The films were applied to 304 stainless steel foils (6.3 by 5.1 by 0.015 cm) that had a hardness of Rockwell B-87 and to 440C-HT steel disks (1.2 cm thick by 6.3 cm in diam) that had a hardness of Rockwell C-58. The riders used in the friction and wear tests were also made from the 440C-HT steel with a hardness of Rockwell C-58.

FRICTION APPARATUS

A hemisphere-on-flat type of sliding friction apparatus was used to study the friction and wear characteristics of the solid lubricant films. A schematic arrangement of the friction specimens is given in figure 1. The riders were hemispherically tipped pins with a radius of 0.476 centimeter. They were loaded with a 1-kilogram mass against a flat, 6.3-centimeter-diameter disk, which was rotated at 1000 rpm. The rider slid on a 5-centimeter-diameter track on the disk and obtained a linear sliding speed of 2.6 meters per second.

The friction specimens were enclosed in a chamber in order that the atmosphere could be controlled. The disk was heated by a high-frequency induction unit. The temperature of the disk was monitored by a thermocouple when the disk was not rotating and by an infrared pyrometer when it was in motion.

PROCEDURE

Surface Preparation and Cleaning

Three different surface preparations were used in this study. The first was to rub the surfaces with a water paste of levigated alumina. The roughness (centerline average (cla)) of this surface after cleaning was 0.10 to 0.20 micrometer. The second was to roughen the surfaces by sanding with number 150 wet sandpaper to a cla roughness of 0.25 to 0.36 micrometer. The third was to roughen the surfaces by sandblasting to a cla roughness of 0.90 to 1.2 micrometers.

After surface roughening, the disks were scrubbed with a brush under running tap water to ensure that no abrasive particles remained. A water paste of levigated alumina was next rubbed by hand over the surface with a polishing cloth. This was followed by a second scrubbing under running tap water. The disks were rinsed in distilled water and clean, dry compressed air was then used to quickly dry the surfaces. The disks were stored in a desiccator until they were coated with the solid lubricant.

The riders were washed with ethyl alcohol and then scrubbed with a water paste of levigated alumina. The riders were next rinsed in distilled water and dried with compressed air. Lubricant was not applied to the riders.

Film Application

An artist's airbrush was used to apply the polyimide and polyimide-bonded graphite fluoride films to the disks and foils. The films did not dry rapidly; thus, only a thin layer was applied at one time in order to prevent "running." Each thin layer was cured completely before the next layer was applied. The cure was to heat the films at 100° C for 1 hour and then at 300° C for 2 hours. It typically took two to three thin-layer applications to achieve the desired thickness of 15 to 20 micrometers.

Thermal Aging Tests

The thermal aging tests were conducted on polyimide films and polyimide-bonded graphite fluoride films applied to both 440C-HT steel disks and 304 stainless steel foils. The films were exposed at temperatures of 315°, 345°, 370°, and 400° C. At various intervals they were taken out of the oven, let cool, and weighed to obtain a weight loss. Cooling time was kept to a minimum, so such variables as water absorption could be kept to a minimum. The disks were aged for only 100 hours or less; the foils were aged for as long as 400 hours.

Adherence Tests

The adherence tests were performed only on the films applied to the foils before and after thermal aging. They consisted of simple bending and flexing tests of the foils. The foils with applied films were repeatedly flexed and bent by hand to various degrees and then observed under optical microscopy for cracking. In a few instances thermal exposure alone caused the films to spall.

Friction and Wear Tests

The procedure for conducting the friction and wear tests was as follows: A rider and disk (with applied solid lubricant film) were inserted into the friction apparatus and the test chamber was sealed. Dry air (<20-ppm H_2O) or moist air (10 000-ppm H_2O) was purged through the chamber for 15 minutes. The flow rate was 1500 cubic centimeters per minute and the volume of the chamber was 2000 cubic centimeters. The disk was then set into rotation at 1000 rpm, and a lowering device gradually applied a load of 1-kilogram mass to the specimens. When the films were evaluated at elevated temperatures, the disk with the applied film was held at that temperature for at least 10 minutes before applying the load to ensure that the temperature had stabilized.

Each test was stopped after 15 minutes (15 kc of sliding). The rider and disk were removed from the friction apparatus and the contact areas were photographed. Several surface profiles of the wear track on the solid lubricant film were also made. The rider and disk were then placed back into the apparatus and the test procedure was repeated. The rider was not removed from the holder when it was photographed, and locating pins in the apparatus ensured that it was returned to its original position. The same was true for the disk. Each test was stopped and the procedure repeated at intervals of 60 and 250 kilocycles of sliding.

RESULTS AND DISCUSSION

Effect of Thermal Aging

Polyimide films. - Polyimide films with no solid lubricant additives were evaluated first. The conventional technique for preparing a surface for good film adherence has been to sandblast the surface. In this study, when the thin foils (0.015 cm thick) were sandblasted, they became distorted. Thus, two other surface preparation techniques were evaluated: wet sanding of the surface, and cleaning with levigated alumina only. These techniques did not distort the foils.

Films of polyimide were applied to all three pretreated surfaces and were thermally exposed to 345° C for as long as 200 hours. The adherence of the films was evaluated by bending tests before thermal exposure and after exposure to 345° C for 100 hours. No cracking or spalling of the films was observed. After 125 hours of exposure, however, the film applied to the foil surface that was cleaned only with levigated alumina started to spall in one of the corners. No spalling of the film applied to the sanded surface was observed for exposure times to 200 hours. Because of this and because sandblasting distorted the foils, the surface preparation technique chosen in this study was sanding.

In addition to adherence, the effect of metallic surface pretreatment and bulk metallic mass on the percentage of weight lost by polyimide films exposed at 345° C was also investigated (both thin foil and thick disk substrates were used). Table I presents those results, which show that there is essentially no effect. The weight lost by the film applied to the smooth foil surface after an exposure of 125 hours was slightly more than for the others because the film spalled.

The effect of temperature on the weight lost by polyimide films applied to sanded surfaces is shown in figure 2. Three test temperatures were used; 345°, 370°, and 400° C. The weight loss rate was constant at any particular temperature and increased with temperature. Table II gives the rates as determined from the slope of the curves in figure 2.

As mentioned previously, the film exposed at 345° C for 200 hours adhered well. The film aged at 370° C initially adhered well, but after 78 hours blistering and spalling occurred. The film aged at 400° C showed the same type of spalling after only 6 hours, and it was completely gone from the surface after 24 hours.

The thermal aging tests suggest that two processes take place in the polyimide films as a result of thermal exposure to high temperatures. The first is that the film evaporates from the surface resulting in a weight loss, and the second is that the film becomes brittle possibly from crosslinking or oxidation of the molecules.

Polyimide-bonded graphite fluoride films. - Polyimide-bonded graphite fluoride films were applied to foils and disks and thermally exposed to elevated temperatures, similarly to the polyimide films. One additional aging temperature, 315° C, was added to the program. Figure 3 gives the results of those aging experiments. As with the polyimide films, the polyimide-bonded graphite fluoride films lost weight at a constant rate at each exposure temperature, and the rate increased with increasing temperature.

Table II compares the weight loss rates calculated from the curves in figure 3 with those obtained on films of polyimide alone. At temperatures of 315° to 370° C the weight loss rates were slightly higher for the polyimide-bonded graphite fluoride films than for the polyimide films. The most probable reason for this was that the surface of the polyimide-bonded graphite fluoride films was considerably rougher than the surface of the polyimide films and thus the exposure area was greater. But at

400° C the weight loss rate was less for the polyimide-bonded $(CF_x)_n$ than for the polyimide alone, probably because $(CF_x)_n$ has a higher thermal stability than polyimide at this temperature.

Regardless of how long or at what temperature the polyimide-bonded graphite fluoride films were aged, no cracking or spalling of the films from the foils or disks was observed. The main detrimental effect of elevated temperature and exposure time was the complete removal of the polyimide in certain areas of the film. Most likely, this was due to the facts that polyimide decomposes at a faster rate than does graphite fluoride (at least at the temperatures evaluated in this study) and that using an airbrush to apply these films does not result in an even distribution of the two constituents. These sparse areas of polyimide did not seem to adversely affect the friction and wear results, since graphite fluoride is an excellent lubricant without a binder.

The foils with applied films were repeatedly flexed and bent by hand to various degrees (before and after thermal aging), and then observed under optical microscopy. Extremely good adherence was obtained and no cracking was observed.

Friction and Wear

Polyimide films. - Polyimide films applied to sanded 440C-HT steel disks were also thermally exposed to 345° C along with the foils, but only for 100 hours. After aging these films were evaluated on a pin-on-disk type of friction and wear apparatus, and the data were compared to similar results from films that were not thermally aged. The test conditions were a moist air atmosphere (10 000-ppm H_2O), a 1-kilogram load, a 1000-rpm rotational speed (2.6-m/sec linear speed), and a 25° C test temperature.

Previous work (9, 10) on nonaged films showed that under these conditions a typical friction coefficient would be 0.11 ± 0.03 and that the wear process is one of gradually wearing through the film. With films of the thickness used in this study, it would take approximately 60 kilocycles of sliding to wear through the films.

The friction tests were run for 15 kilocycles of sliding and then stopped so that the surfaces could be evaluated. Figure 4 shows friction traces, for that interval, for a film aged for 100 hours at 345° C and for a film not aged. The figure illustrates the large difference in friction that was obtained with the two films.

The reasons for this difference became evident when the films were examined using optical microscopy and surface profilometry. Figures 5 and 6 show what happened. Thermal exposure to 345° C for 100 hours has apparently embrittled the polyimide film to the extent that, when the load was applied to the film under sliding conditions, it underwent brittle fracture in the wear track area. Figure 5 gives a cross-sectional view of the wear track area where the film has spalled. Figure 6 shows photomicrographs of an area of the wear track where part of the film has spalled from the surface and also of a region that is in the process of spalling.

Even though the film spalled, it did not make a clean break from the surface. The insert in figure 6 shows that a very thin layer of polyimide remained behind (less than a micrometer). This layer, combined with the debris from the spalled polyimide, formed a very thin secondary film on the disk surface, which provided lubrication. This is an analogous situation to that described in references 9 and 10, except that in those studies the secondary film formed only after the original film had been worn through.

When lubrication by the secondary film mechanism occurs, the friction coefficient is usually higher because a certain amount of metallic contact occurs (see insert in fig. 6). Also as sliding continues, the amount of polyimide material in the contact area is gradually depleted by flowing out the sides of the wear track. Thus, friction usually increases with sliding time because of the increasing metallic contact.

Polyimide-bonded graphite fluoride films. - Friction and wear experiments were conducted on the thermally aged polyimide-bonded graphite fluoride films at 25° C in atmospheres of either moist air (10 000-ppm H_2O) or dry air (<20-ppm H_2O). Representative friction traces are shown for these experiments in figure 7. The effect of test atmosphere on the friction coefficient of the films was considerable. In general, the friction coefficient rose more rapidly and to a higher value in moist air than in dry air. The traces are also smoother in dry air, which indicates better lubrication.

The effect of thermal aging on the friction coefficient of the films appears to be minimal. For a specific atmosphere, the individual traces look somewhat different, but the variation is over the same range. Thermal exposure at the higher temperatures did seem to reduce the useful life of the films, however. For example, the friction trace for the film aged at 370° C became rougher after only 190 kilocycles of sliding and the average value increased (fig. 7(a)).

Representative surface profiles of the film wear tracks in a moist air atmosphere are shown in figure 8 for sliding intervals of 15, 60, and 250 kilocycles. The figure illustrates that wear was not a process of gradual attrition but a process of quickly wearing through the film and then sliding on a very thin film (secondary film) at the surface of the metal disk. The process is somewhat analogous to the process described for the polyimide films, but brittle fracture did not occur in this case. The films were all approximately the same thickness before thermal aging; thus, figure 8 also illustrates the reduction of film thickness due to thermal aging.

Not all the experiments conducted in moist air were duplicated in dry air. Surface profiles of the film wear tracks that were duplicated show that considerably less wear of the films takes place in dry air. Thus, as the friction traces of figure 7 illustrated, moisture in air is detrimental to the friction and wear process of these films.

In addition to surface profiles of the films, the surfaces were also studied by optical microscopy and photographed. Figures 9 to 13 are photomicrographs of rider contact areas and film wear tracks for representative tests.

Figure 9 compares the contact areas for nonaged films and for films aged for 100 hours at 315° C that have slid for 15 kilocycles in a moist air atmosphere at 25° C. The rider that slid on the nonaged film shows no wear and only a transfer film; in contrast, the rider that slid on the film aged at 315° C has a discernible circular wear scar. The film wear track for the nonaged film is very smooth and the metal substrate cannot yet be seen; the metallic substrate of the disk can be seen in the wear track of the film aged at 315° C. Thus, the most probable reason for the rider wear on the film aged at 315° C was that a certain amount of metallic contact occurred.

After 60 kilocycles of sliding, a circular wear scar appeared on the rider that slid on film which was not thermally aged and the metallic substrate of the disk could be seen in the film wear track area. The rider wear scar and film wear track for the 315° C thermally aged film looked much the same as they did at 15 kilocycles, except that they had become slightly larger.

Figure 10 shows the contact areas after 250 kilocycles of sliding. The photomicrographs illustrate the greater wear of the rider that slid on film thermally aged at 315° C; they also illustrate that the polyimide-bonded graphite fluoride film is being depleted in the wear track area. The metal-film ratio is higher after 250 kilocycles than after 15 kilocycles. The protrusions seen on either side of the circular rider wear scars in figure 10 are transferred films from the sides of the wear track on the disk and are not wear.

Figure 11 shows high magnification photomicrographs of the wear track (after sliding intervals of (a) 15 kc and (b) 250 kc in a moist air atmosphere) for a polyimide-bonded graphite fluoride film not aged prior to testing. After 15 kilocycles of sliding, striations can be seen in the wear track film at the side of the track and in the center the film has coalesced into a thin, very smooth, transparent film. This transparency was not noticeable at low magnifications.

As mentioned previously, the film gradually thinned on continued sliding. Figure 11(b) shows the thinning of the film after 250 kilocycles of sliding. The polyimide-bonded graphite fluoride filled up the scratches on the sanded disk and tended to flow across the flat plateaus between the scratches. Interference bands can be seen in these flowing films indicating the thickness is of the order of the wavelength of light (0.4 to 0.8 μm).

Figure 12 shows similar high magnification photomicrographs of the wear track on a polyimide-bonded graphite fluoride film which was thermally aged at 315° C for 10 hours before testing at 25° C in a moist air atmosphere. After 15 kilocycles of sliding, it is seen that the film in the center of the wear track (fig. 12(a)) is thinner than the nonaged film (fig. 11(a)), and it is also not as continuous. An edge can be seen in figure 12(a) which is a demarcation between two different thickness areas of the film.

Comparing the aged and nonaged films after 250 kilocycles of sliding, it is seen that the major difference is that the wear track on the aged film has become more depleted (fig. 12(b)) than the nonaged film (fig. 11(b)). More metallic areas are seen in the aged film wear track and also the scratches are tending to become depleted of polyimide bonded graphite fluoride material. Other than more rapid depletion, no other differences in the lubricating mechanism of the thin secondary film could be detected.

Photomicrographs were also taken of the specimens tested in a dry air atmosphere. Figure 13 shows photomicrographs of the contact areas after 15 and 250 kilocycles of sliding on a film aged for 100 hours at 315° C. The test specimens from experiments on nonaged films look almost exactly the same; thus, they are not shown. When compared to the photomicrographs of figures 9 and 10 the photomicrographs of figure 13 demonstrate that moisture has an adverse effect on these films. This agrees with the friction traces of figure 7.

A quantitative comparison of rider wear is given in table III. Wear rate is given for the three sliding intervals: 0 to 15 kilocycles, 15 to 60 kilocycles, and 60 to 250 kilocycles. In general, the wear rates obtained in moist air increased as a function of increasing thermal exposure temperature. In dry air, this was not the case; for those films evaluated, the same rates were obtained for nonexposed films as for thermally exposed films.

Lubrication at elevated temperatures. - The effect of thermal aging on the friction and wear properties of polyimide-bonded graphite fluoride films at elevated temperatures was also investigated. These experiments were conducted for 60 kilocycles of sliding in moist air (10 000-ppm H_2O) at temperatures of either 315° or 345° C. Three different films were evaluated: the nonaged film, the film aged at 315° C, and the film aged at 345° C.

The friction results are given in figure 14. The traces indicate that thermal aging did not have much, if any, effect on the friction characteristics of the films at elevated temperatures. The friction coefficients of the films tested at 315° C began to increase between 50 and 60 kilocycles of sliding (figs. 14(a) to (c)), which indicated that film failure was imminent. The film tested at 345° C failed after 30 kilocycles of sliding (fig. 14(d)). Although a shorter wear life occurred at elevated temperatures than at 25° C, the friction coefficients obtained were less. After a short "run-in" at elevated temperatures, the friction coefficient dropped to a value less than 0.02 (fig. 14). The lowest value obtained at 25° C was the initial value of 0.05 (fig. 7).

The rider wear rates obtained are shown in table III(b) and (c). The rates at elevated temperatures did not depend upon whether the films were aged or not, and the values obtained were very similar to those found for films aged at 345° C or higher and tested at 25° C. When compared to those films aged at 315° C and tested at 25° C; however, the rider wear rates at elevated temperatures were about 10 times as great. This indicates that either too high an aging temperature or too high a test temperature can reduce the lubricating efficiency of the film.

Photomicrographs were taken of the sliding surfaces after 60 kilocycles of sliding in a moist air atmosphere at 315° C. Rider wear scars and film wear tracks are shown for films not thermally aged (fig. 15(a)) and for films aged for 100 hours at 315° C (fig. 15(b)). As the figure indicates, thermal aging did not produce any detectable differences in wear scar or film appearance between tests at 315° C.

When compared to tests at 25° C, some differences are seen. The transfer films and wear track films at 315° C are thicker, not as continuous, not transparent, and there is a considerable amount of extremely fine, black powdery debris present. Figure 16 shows a high magnification photomicrograph of a secondary film on the wear track of a film aged at 315° C and tested at 315° C, which is typical of the tests conducted at elevated temperatures. The differences in film appearance between the 25° C (fig. 12) and 315° C (fig. 16) may be due to good versus poor lubrication rather than a temperature effect; however, since the photomicrographs taken of the surfaces at 25° C were taken when good lubrication was occurring, while the photomicrographs taken at a test temperature of 315 were taken when failure was imminent.

Wear mechanisms. - Two wear mechanisms appear to be operating when metal riders slide against polyimide or polyimide-bonded graphite fluoride films. The first occurs when the film is strong enough to support the load or is thick enough to enable plastic deformation (or wear) to increase the contact area of the film so that the film will still support the load without the rider making metallic contact with the disk. The second mechanism occurs when the film will not support the load.

In the first mechanism (film supports load), no wear is observed to the rider, just transferred film material. The wear process is one of gradual wear through the film until the metallic substrate is reached.

In the second mechanism (film does not support the load), the rider quickly plows through the film or brittlely fractures it away. Film wear debris and material from the sides of the film wear track then combine to form a very thin secondary film at the metallic surface. In the process, some metallic contact occurs and a flat is worn on the rider. The secondary film is very dynamic in nature, it is constantly flowing and being reformed from solid lubricant wear debris and from material from the sides of the wear track. Wear to the rider continually increases because a small amount of metallic contact takes places intermittently. As sliding continues, the lubricant supply is gradually depleted (flows out the sides of the wear track) and friction and rider wear gradually increase.

In some instances a combination of the two mechanisms occurs during the wear life of a film. The rider will gradually wear through the film until the metallic substrate is reached and then a secondary film will form that will extend the life for a period of time.

SUMMARY OF RESULTS

A preliminary investigation of the effect of thermal aging on the adherence, weight loss, friction, and wear of polyimide and polyimide-bonded graphite fluoride films gave the following results:

1. The presence of 50 weight percent graphite fluoride in polyimide films improved the ability of the polyimide films to withstand thermal aging and to provide better lubrication.

2. Friction and wear results were not adversely affected by thermal aging when graphite fluoride was present.

3. Weight loss due to thermal aging was low for both films for exposure temperatures to 315° C.

4. Friction and wear results were sensitive to environmental conditions; that is, better results were obtained in dry air than in moist air.

5. Pretreating the surfaces by sanding is an effective means of promoting good adhesion of the films.

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TABLE I. - EFFECT OF METALLIC SURFACE PRETREATMENT AND BULK MASS ON WEIGHT LOSS OF POLYIMIDE FILMS EXPOSED AT 345° C FOR VARIOUS TIMES

Exposure time, hr	Smooth foil surface	Sanded foil surface	Sandblasted foil surface	Sanded disk surface
	Weight of steel, g			
	3.9183	3.9315	3.8782	301.5010
	Weight of film, g			
	0.1919	0.1010	0.1335	0.1350
	Weight loss of film, percent of total film weight			
20	9	6	7	5
27	11	--	9	--
34	12	8	10	--
51	13	9	11	--
75	14	13	14	--
82	15	--	16	--
100	19	17	18	20
125	24	19	20	--

TABLE II. - EFFECT OF THERMAL EXPOSURE ON RATE OF WEIGHT LOSS OF POLYIMIDE AND POLYIMIDE-BONDED GRAPHITE FLUORIDE FILMS

Exposure temperature, °C	Polyimide	Polyimide-bonded graphite fluoride
	Rate of weight loss, percent/hr	
315	----	0.04
345	0.17	.25
370	.41	.71
400	4.0	1.6

TABLE III. - WEAR RATE OF RIDERS THAT SLID ON POLYIMIDE-BONDED GRAPHITE FLUORIDE FILMS THERMALLY EXPOSED AT VARIOUS TEMPERATURES FOR 100 HOURS

Sliding interval, kilocycles	Exposure temperature, ^{o}C									
	None		315		345		370		400	
	Air environment									
	Moist	Dry	Moist	Dry	Moist	Dry	Moist	Dry	Moist	Dry
	Rider wear rates, m^3/m									
(a) Test temperature, 25^{o} C										
0-15	(a)	(a)	0.47×10^{-15}	(a)	0.55×10^{-15}	(a)	0.62×10^{-15}	---	0.80×10^{-15}	---
15-60	0.047×10^{-15}	0.017×10^{-15}	$.049\times10^{-15}$	0.014×10^{-15}	$.29\times10^{-15}$	0.016×10^{-15}	$.29\times10^{-15}$	---	$.50\times10^{-15}$	---
60-250	$.034\times10^{-15}$	$.034\times10^{-15}$	$.14\times10^{-15}$	$.031\times10^{-15}$	$.39\times10^{-15}$	------------	$.86\times10^{-15}$	---	------------	---
(b) Test temperature, 315^{o} C										
0-60	0.33×10^{-15}	------------	0.33×10^{-15}	------------	0.38×10^{-15}	------------	------------	---	------------	---
(c) Test temperature, 345^{o} C										
0-30	------------	------------	------------	------------	[b]0.39×10^{-15}	------------	------------	---	------------	---

[a]Transfer only.

[b]Film failed after 30 kilocycles.

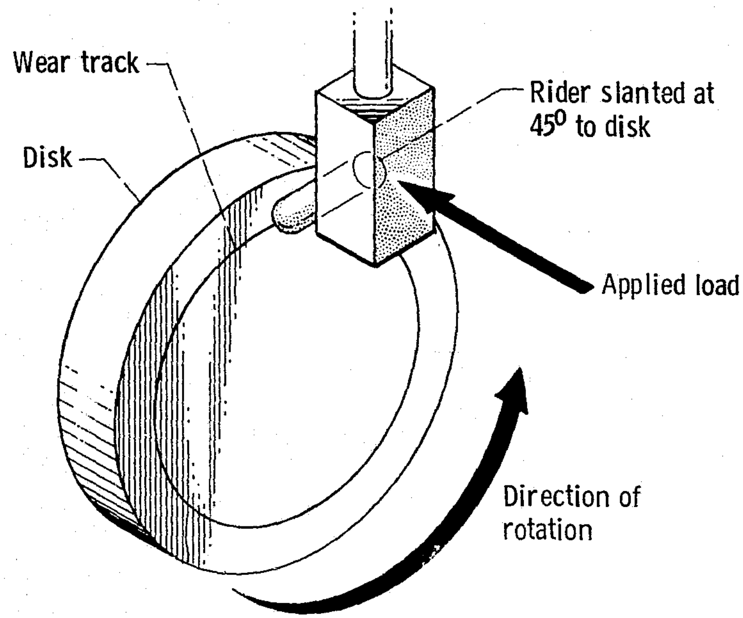

Fig. 1 - Schematic arrangement of friction specimens.

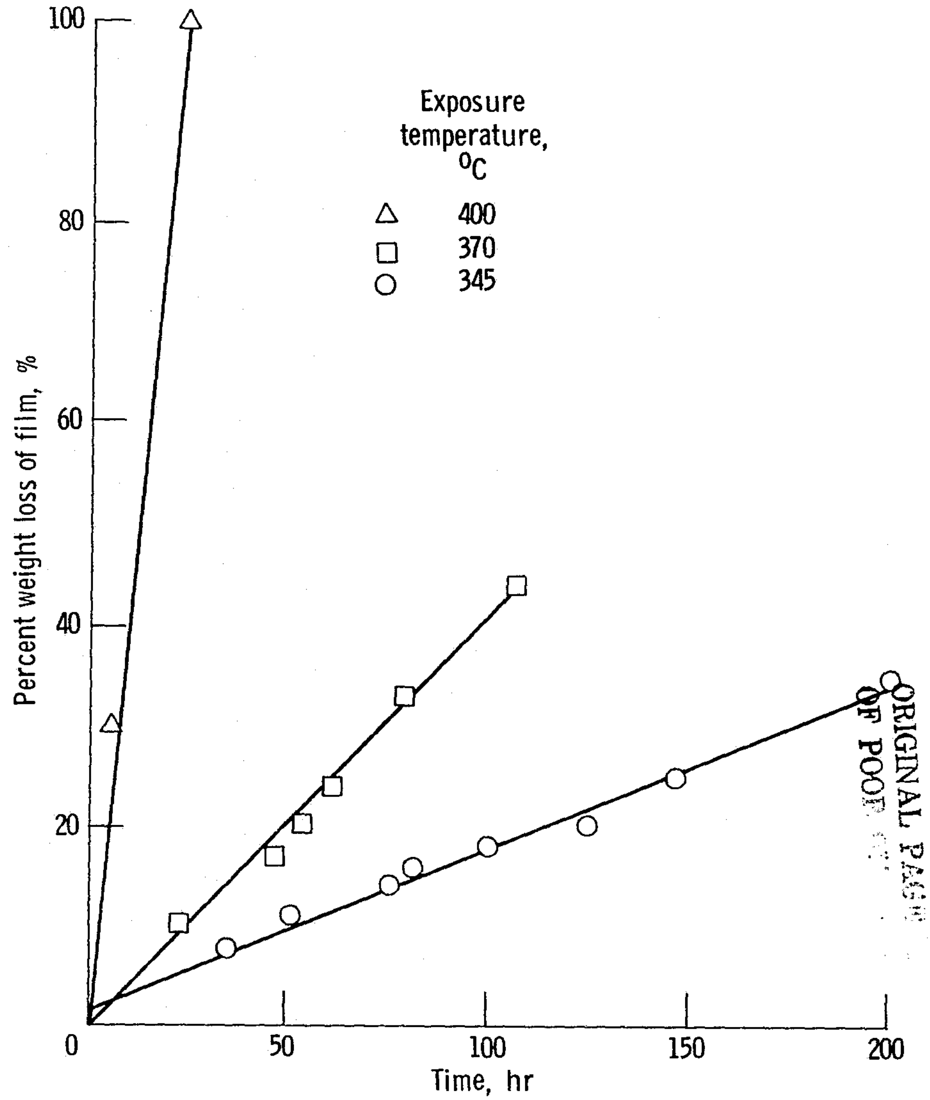

Fig. 2 - Rate of polyimide film weight loss as function of exposure temperature.

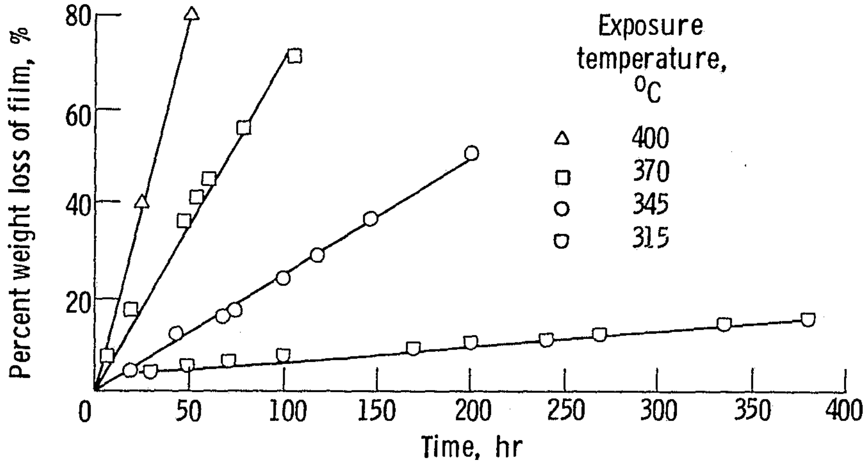

Fig. 3 - Rate of polyimide-bonded graphite fluoride film weight loss as function of exposure temperature.

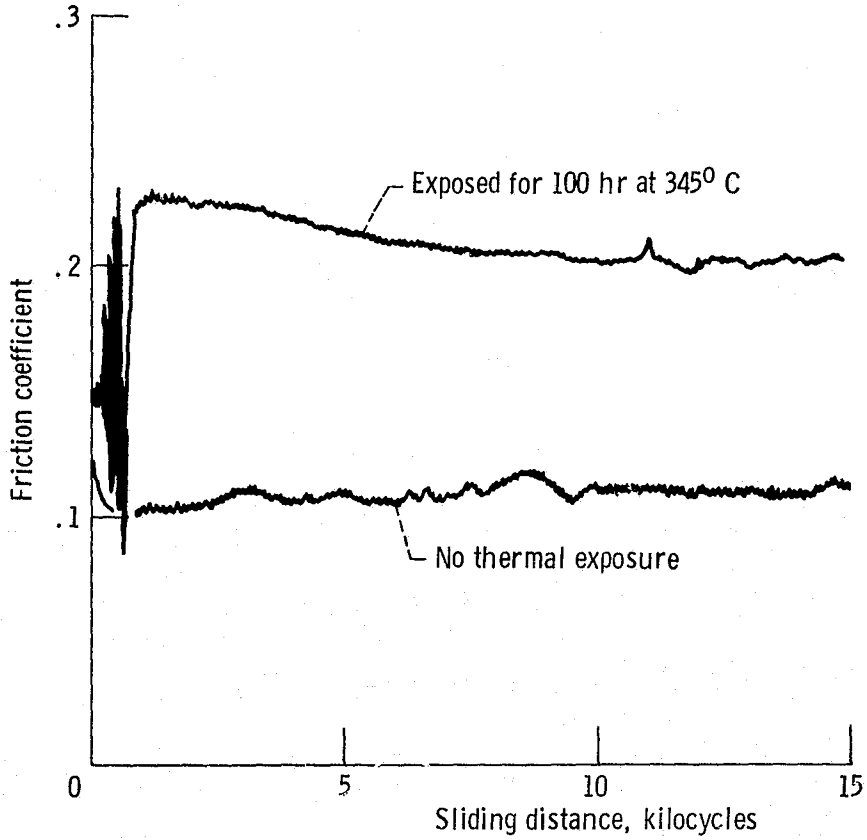

Fig. 4 - Friction coefficient of polyimide films at 25° C in a moist air atmosphere (10 000 ppm H_2O) as a function of sliding distance for non-thermally aged films and for films aged at 345° C for 100 hours.

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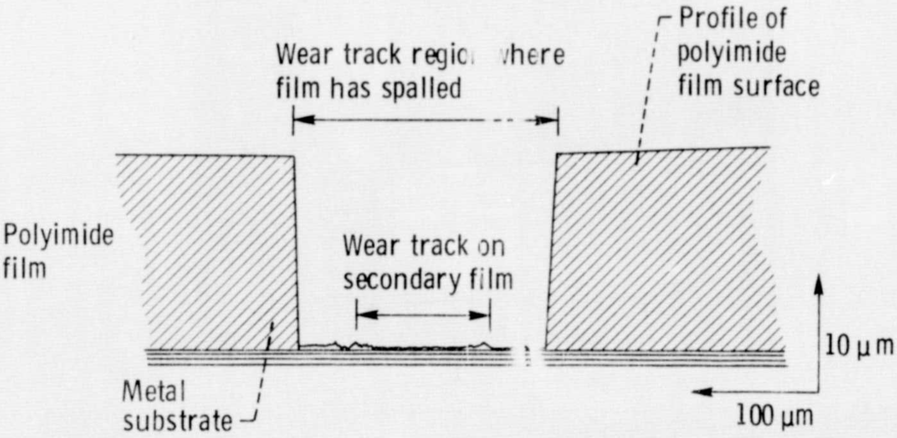

Fig. 5 - Cross sectional view of a polyimide film wear track (thermally exposed at 345^{0} C for 100 hr) after 15 kc of sliding in moist air at 25^{0} C, showing the spallation of the film and the formation of a very thin secondary film on the metallic substrate.

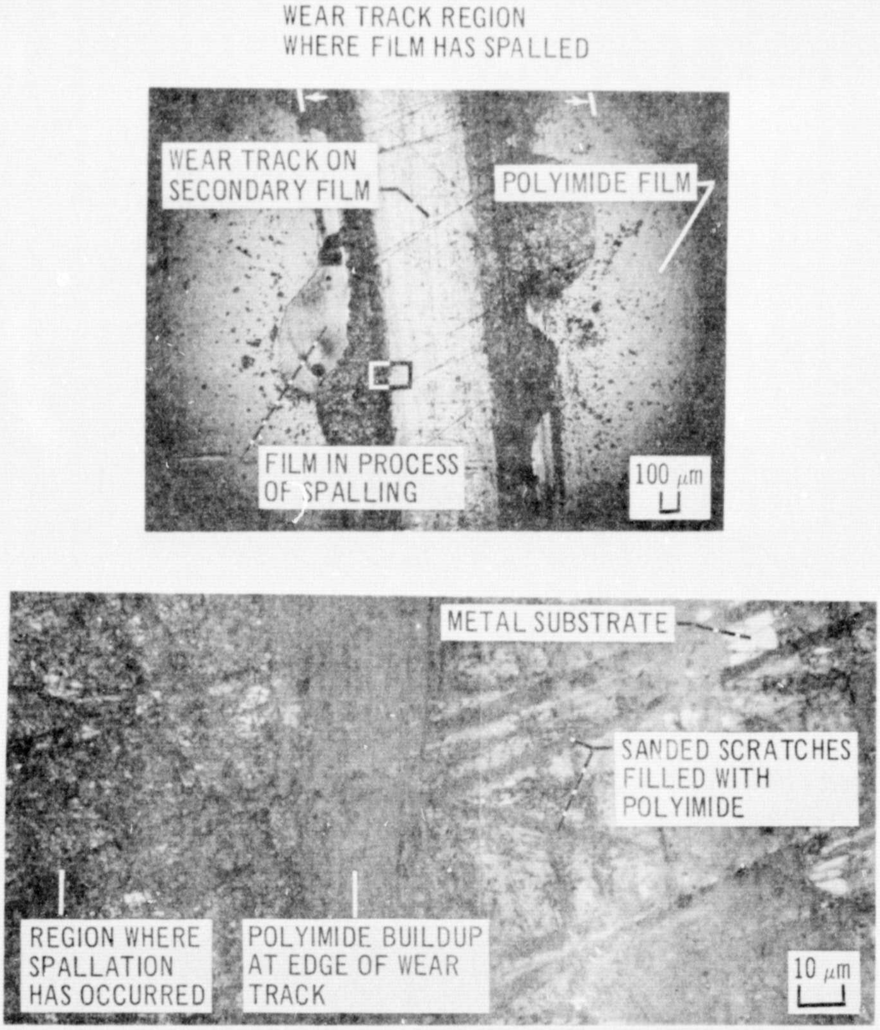

Fig. 6 - Photomicrographs of the wear track after 15 kilocycles of sliding at 25 C in moist air on a polyimide film which was thermally aged for 100 hours prior to testing, showing spallation of the film and the formation of a very thin secondary film on the metallic substrate.

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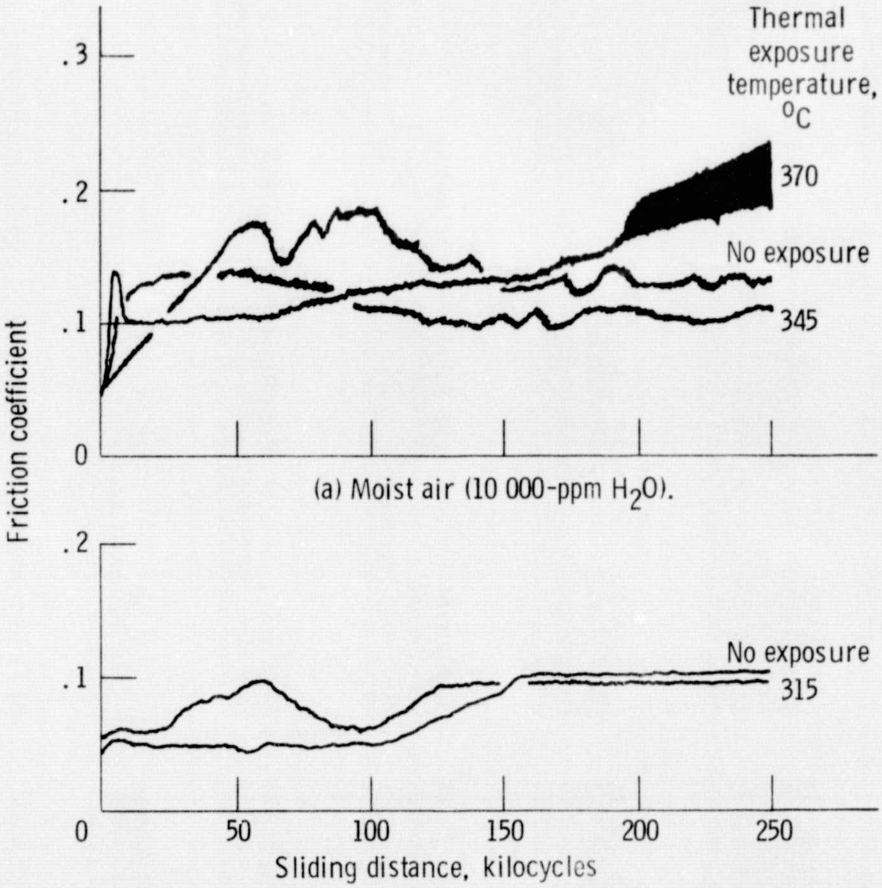


(b) Dry air (20 ppm H_2O).

Fig. 7 - Friction coefficient of thermally aged polyimide-bonded graphite fluoride films as a function of sliding distance in a (a) moist air and in a (b) dry air atmosphere at 25^{0} C.

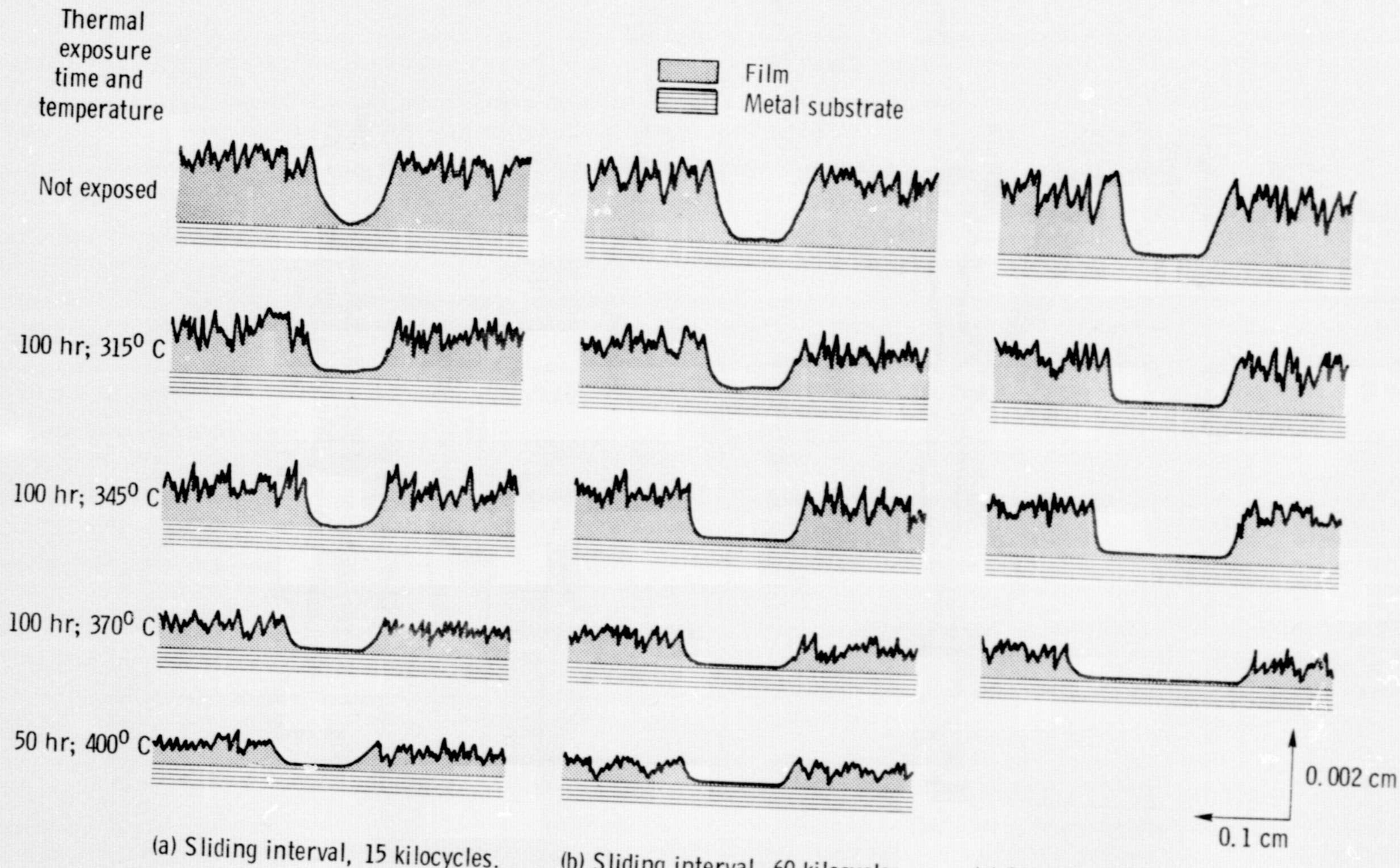
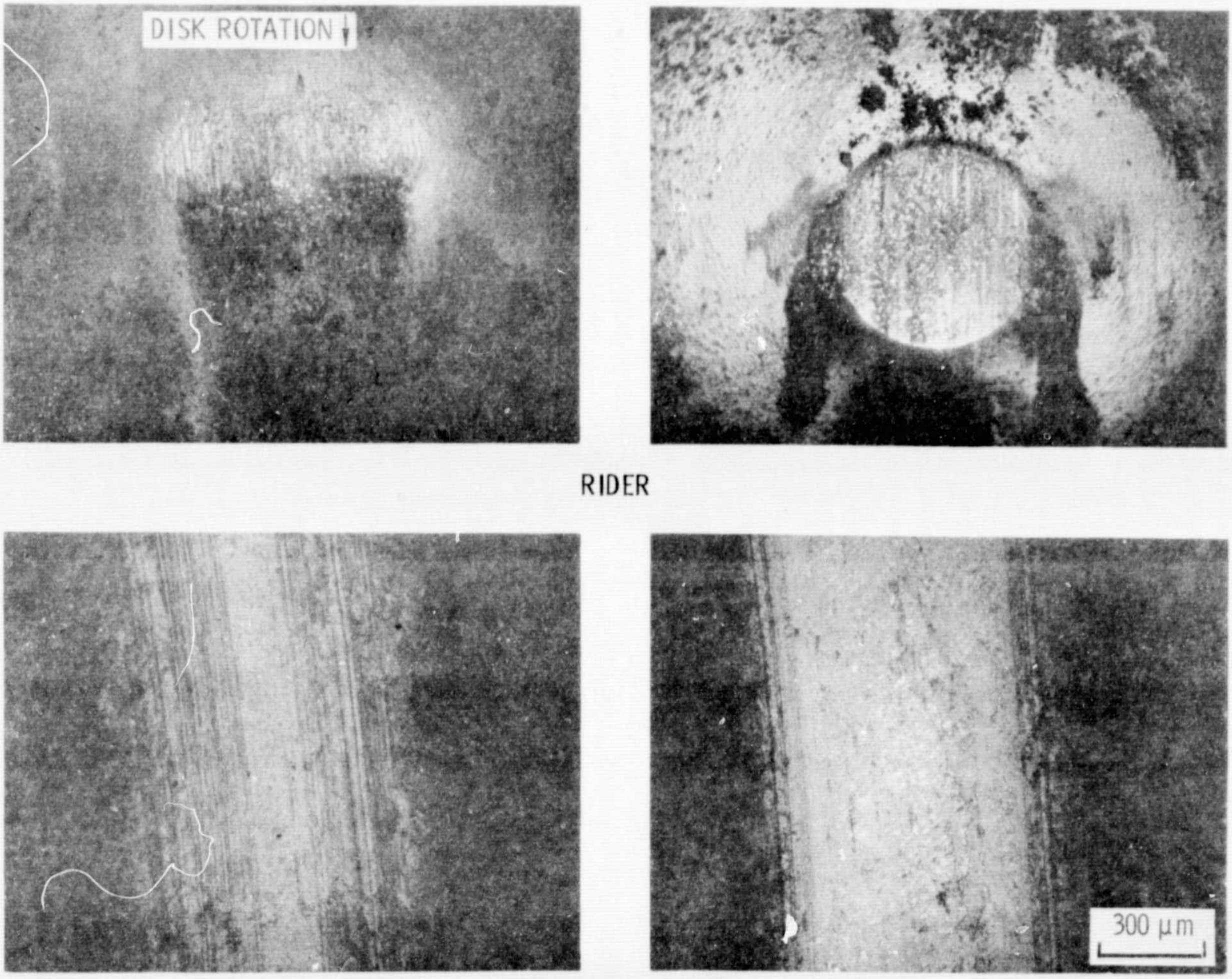


Fig. 8 - Wear at 25° C of polyimide-bonded graphite fluoride films (thermally aged at various temperatures) in a moist air atmosphere (10 000-ppm H_2O) after sliding for intervals of 15, 60, and 250 kilocycles.



POLYMIDE-BONDED $(CF_x)_n$ FILM

(a) NOT THERMALLY AGED. (b) AGED AT 315^{0} C FOR 100 HOURS.

Fig. 9 - Rider wear scars and polymide-bonded graphite fluoride film wear tracks after 15 kilocycles of sliding at 25^{0} C in a moist air atmosphere (10 000 ppm H_2O) for films that were (a) Not thermally aged, and for films (b) Aged at 315^{0} C for 100 hours.

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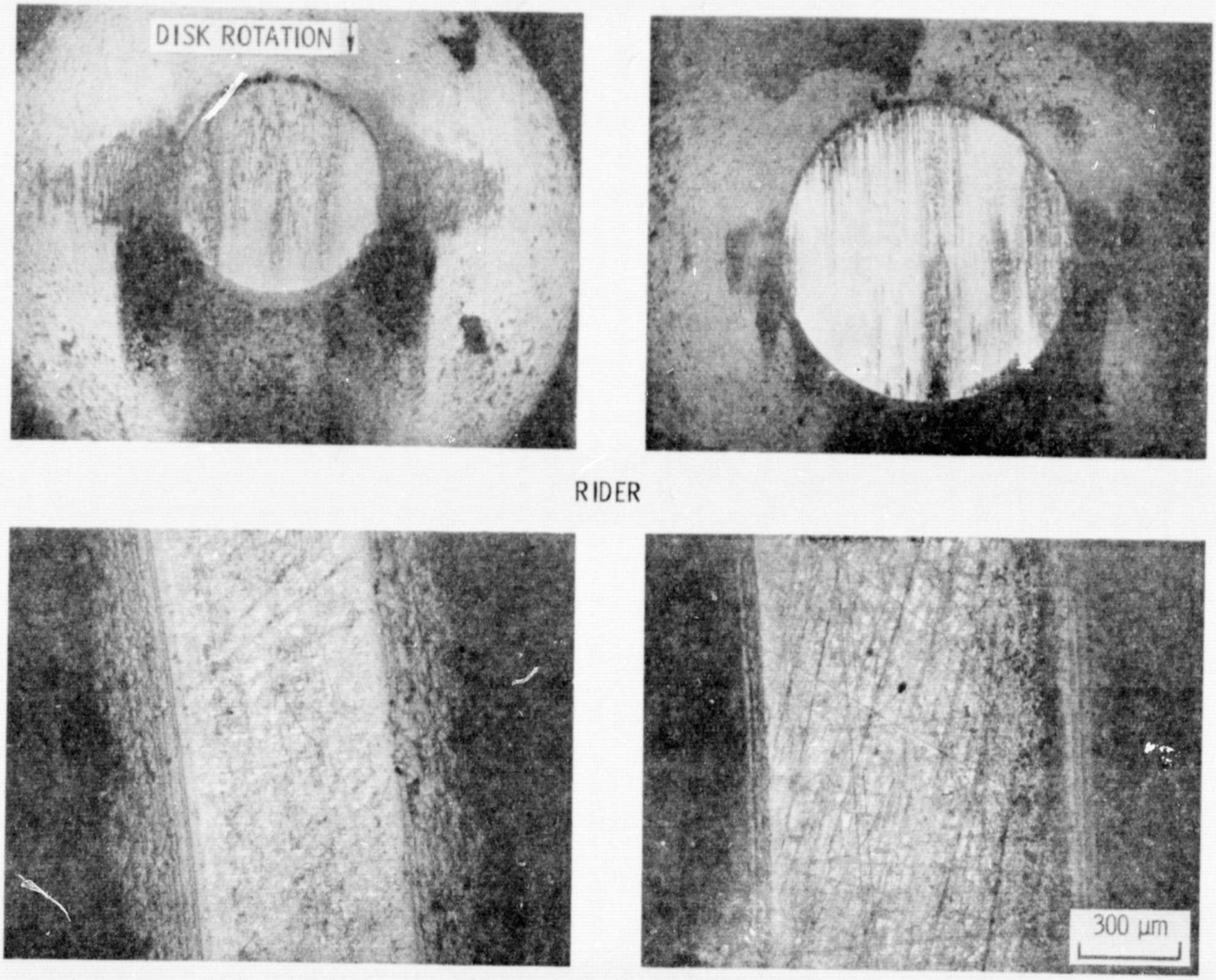
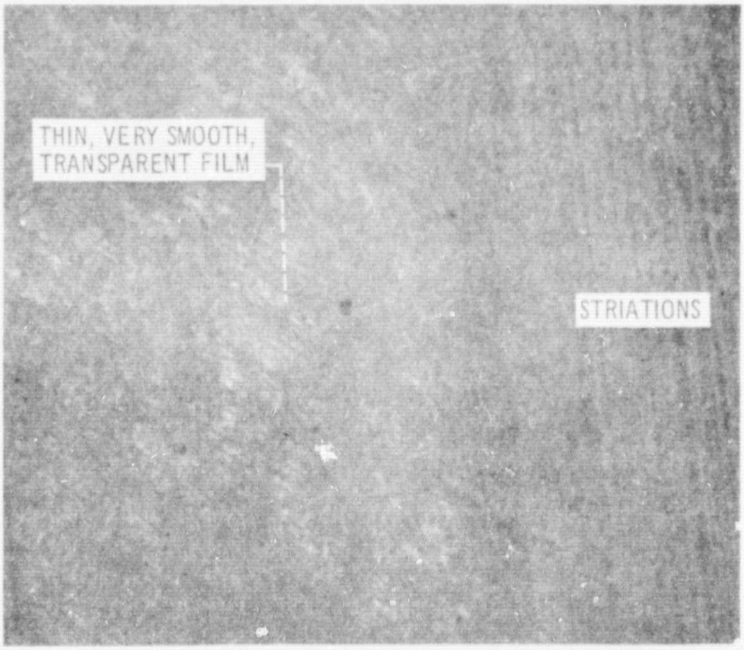


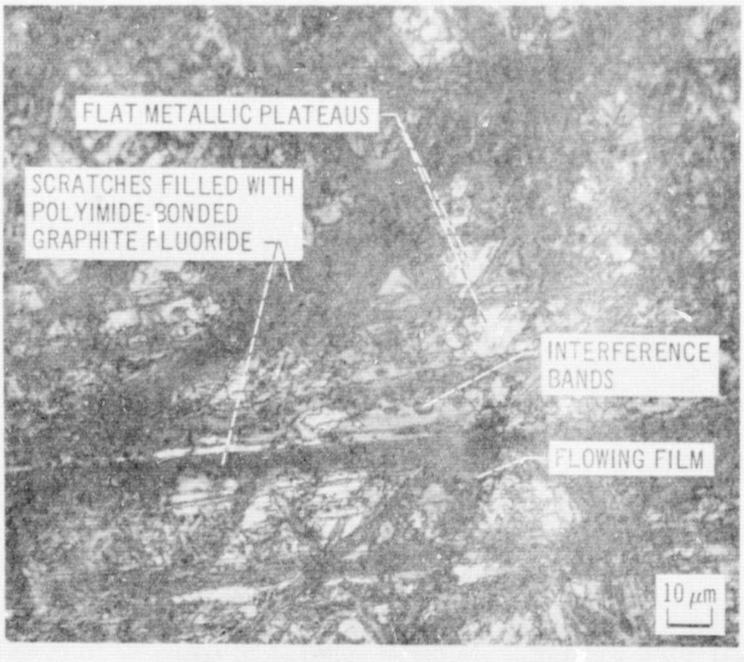
POLYMIDE-BONDED $(CF_x)_n$ FILM

(a) NOT THERMALLY AGED. (b) AGED AT 315° C FOR 100 HOURS.

Fig. 10 - Rider wear scars and polymide-bonded graphite fluoride film wear tracks after 250 kilocycles of sliding at 25° C in a moist air atmosphere (10 000 ppm H_2O) for films that were (a) Not thermally aged, and for films (b) Aged at 315° C for 100 hours.

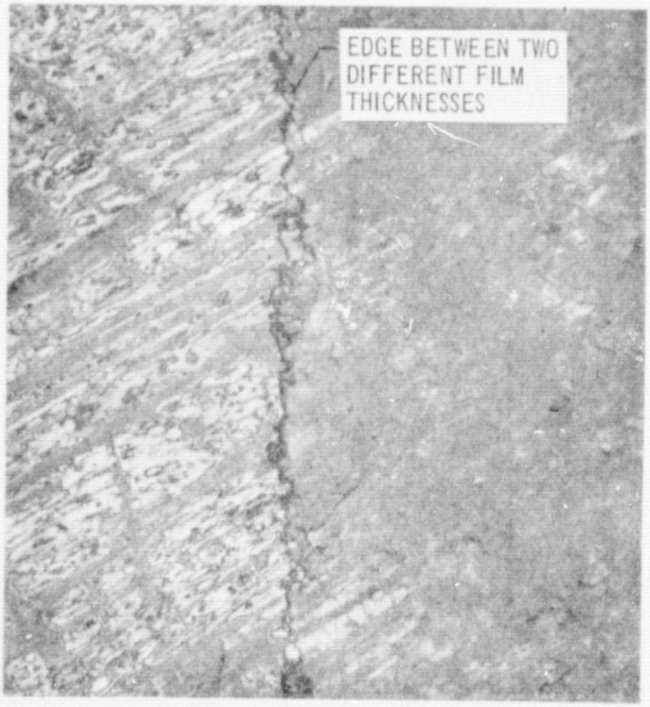


(a) SLIDING INTERVAL, 15 KILOCYCLES.

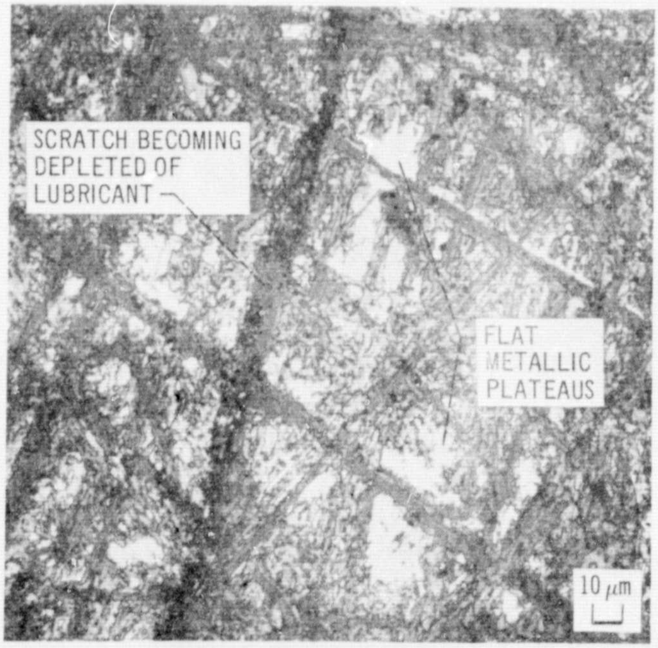


(b) SLIDING INTERVAL, 250 KILOCYCLES.

Fig. 11 - High magnification photomicrographs of the wear tracks on polyimide-bonded graphite fluoride films which were not thermally exposed prior to testing at 25 C in moist air (10 000 ppm H_2O).



(a) SLIDING INTERVAL, 15 KILOCYCLES.



(b) SLIDING INTERVAL, 250 KILOCYCLES.

Fig. 12 - High magnification photomicrographs of the wear tracks on polyimide-bonded graphite fluoride films which were thermally aged at 315 C for 100 hours prior to testing at 25 C in moist air (10 000 ppm H_2O).

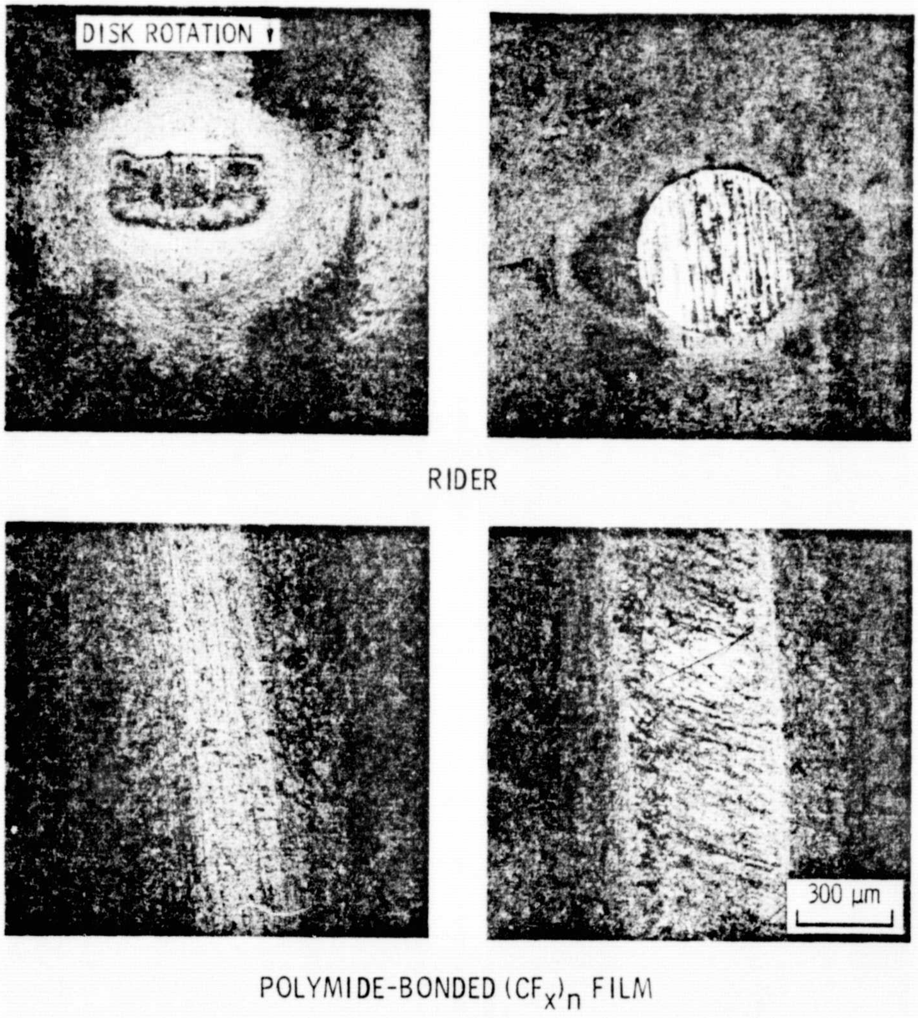


Fig. 13 - Rider wear scars and film wear tracks for tests conducted in dry air at 25° C on polymide-bonded graphite fluoride films thermally aged at 315° C for 100 hours and then slid for (a) 15 kilocycles and (b) 250 kilocycles.

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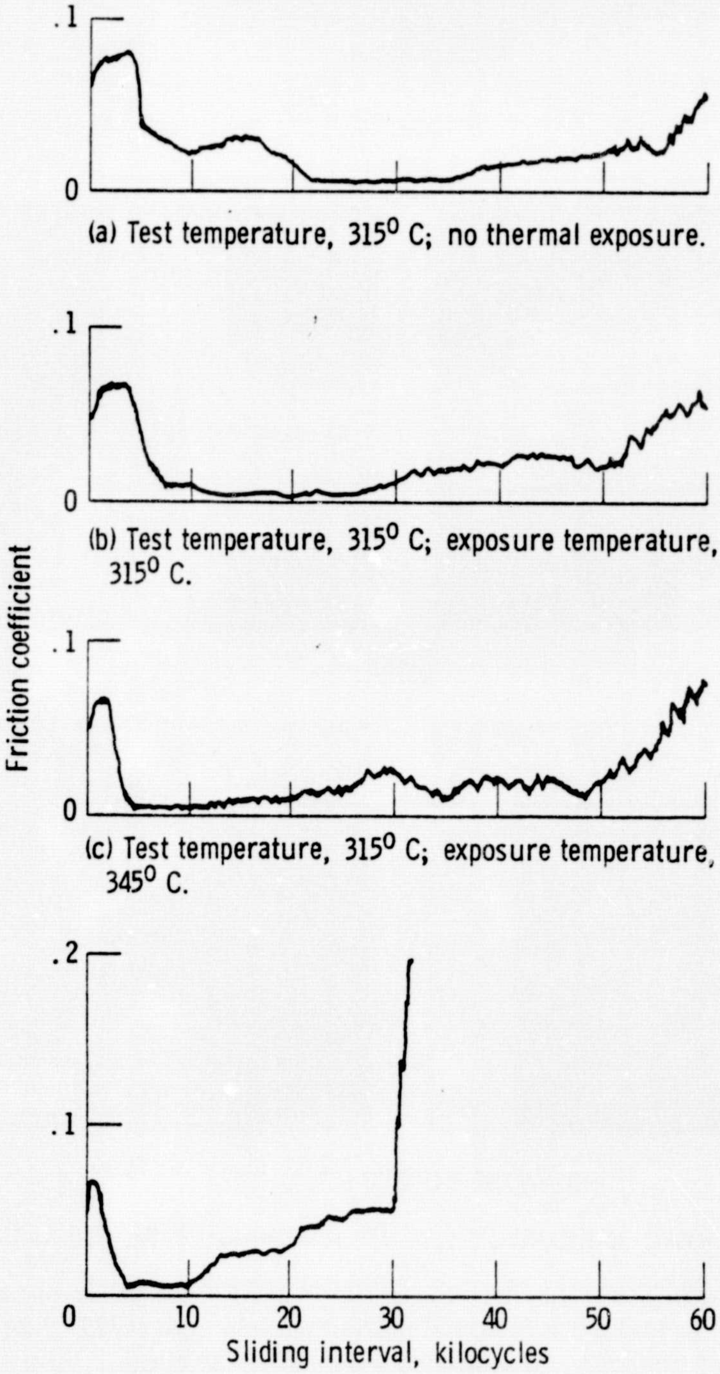


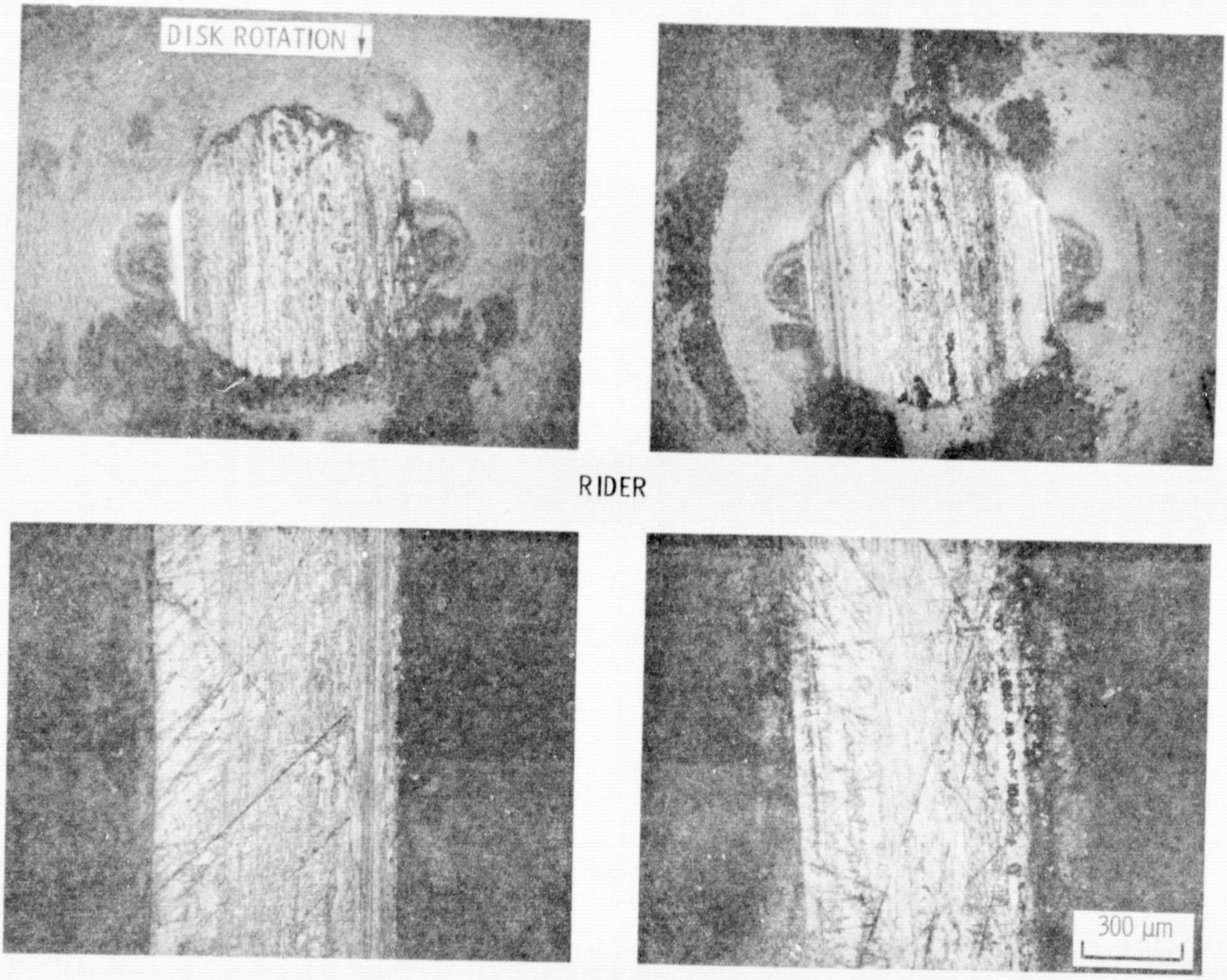
(a) Test temperature, 315° C; no thermal exposure.

(b) Test temperature, 315° C; exposure temperature, 315° C.

(c) Test temperature, 315° C; exposure temperature, 345° C.

(d) Test temperature, 345° C; exposure temperature, 345° C.

Fig. 14 - Friction traces taken at elevated temperatures for polyimide-bonded graphite fluoride films exposed to various temperatures for 100 hours and then tested in a moist air atmosphere (10 000-ppm H_2O).



POLYMIDE-BONDED $(CF_x)_n$ FILM

(a) NOT THERMALLY AGED.

(b) AGED AT 315° C FOR 100 HOURS.

Fig. 15 - Rider wear scars and film wear tracks, after 60 kilocycles of sliding at 315° C in a moist air atmosphere (10 000 ppm H_2O), on polymide-bonded graphite fluoride films which were (a) Not thermally aged and which were (b) Aged at 315° C for 100 hours.

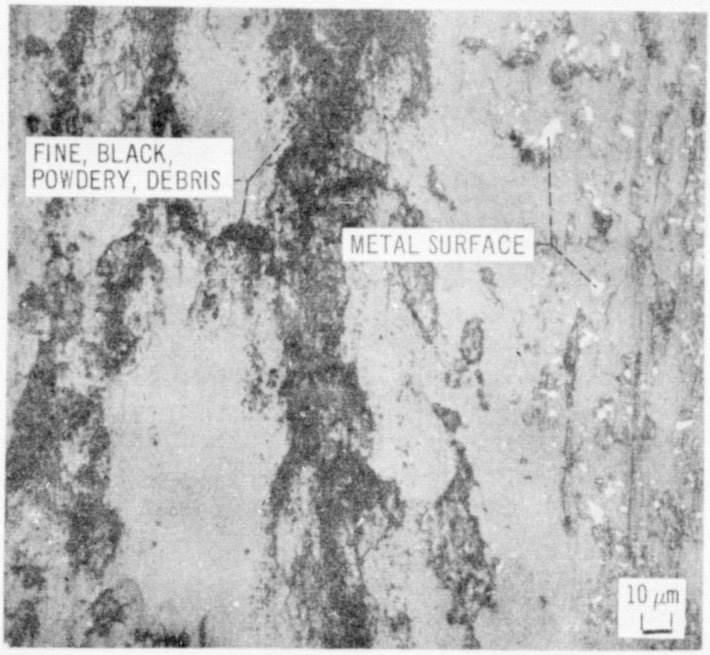


Fig. 16 - High magnification photograph of the wear track after 60 kilocycles of sliding at 315 C in moist air (10 000 ppm H_2O) on a polyimide-bonded graphite fluoride film which was thermally aged for 100 hours prior to testing at 315 C.