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EFFECT OF ATMOSPHERE AND TEMPERATURE ON WEAR, FRICTION, AND TRANSFER OF POLYIMIDE FILMS

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ABSTRACT

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INTRODUCTION

The use of polymers for lubrication applications is continually increasing. Polymers are needed which have improved friction and wear properties as well as thermal stability at higher temperatures. One class of stable organic polymers, which has demonstrated increased capabilities in these areas, is polyimide (1)-(17).

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basic polyimide structure (18)-(20). But, before polyimides can be synthesized with improved lubrication properties, a more basic understanding of the friction and wear processes of this polymer is necessary.

Previous studies by this investigator on pyralin polyimide films have shown that the friction and wear lives (endurance lives) of these films were temperature and atmosphere dependent (21). It was shown in Ref. 21 that a transition from high friction to low friction and from short wear lives to long wear lives occurred in dry argon or in dry air (<20 ppm H_2O) somewhere between the temperature of 25 C and 100 C.

A more detailed study of the friction transition was reported in Ref. 22, and the transition was found to occur in dry argon at 40±10 C. It was postulated in Ref. 22 that the transition was due to a reordering of the surface into a texture conducive to easy shear. Such a texture could be produced by an extended chain molecular structure with the chains parallel to the sliding direction. For texturing to occur, the molecules seemed to need a certain degree of freedom, which was supplied by thermal energy.

The object of this investigation was to determine if any correlation exists between the friction transition and the wear of pyralin polyimide films which were evaluated in atmospheres of dry argon (<20 ppm H_2O), dry air (<20 ppm H_2O), and moist air (10 000 ppm H_2O). To accomplish this, a photomicrographic and surface profile study was made of the wear of polyimide films. Photomicrographs of rider transfer films and polyimide film wear tracks were taken after various sliding intervals and compared. Surface profiles of the polyimide film wear tracks were also taken at these same intervals and compared.

APPARATUS DESCRIPTION

A hemisphere-on-flat sliding friction apparatus was used to study the polyimide friction and wear transition. The friction specimens (Fig. 1) consisted of a flat (6.3 cm diam) disk in sliding contact with a stationary (0.476 cm radius) hemispherically tipped rider. The wear track diameter on the disk was varied by changing the position of the rider. Thus, several tests could be performed on each disk on diameters that ranged from 3.8 to 5.8 centimeters. The apparatus was equipped with a variable-speed motor and gear reduction system so that rotational speed could be controlled. Induction heating was used to heat the disk, and a strain gage sensed the frictional force, which was continuously recorded on a strip-chart recorder.

DISK SURFACE PREPARATION AND CLEANING PROCEDURE

The riders and disks were made of 440C stainless steel with a Rockwell hardness of C-60. In order to ensure good adherence of the polyimide film to the disks, the surfaces were roughened by sandblasting to an rms of 0.90×10^{-6} meter.

After surface roughening, the disks were scrubbed with a brush under running water to ensure that no abrasive particles remained. A water paste of levigated alumina was next rubbed over the surface with a polishing cloth. This was followed by a second scrubbing under running water. The disks were than rinsed in distilled water and stored in a desiccator until they were coated with the polyimide.

The riders were first scrubbed with alcohol. Then a water paste of levigated alumina was applied with a polishing cloth. The riders were then rinsed in distilled water and stored in a desiccator. Polyimide films were not applied to the riders.

FILM APPLICATION

A thinner consisting of N-methyl-pyrrolidone and xylene was added to the polyimide precursor solution to obtain a sprayable mixture. The polyimide solution was sprayed onto each disk by using an artist's airbrush. The film did not dry rapidly. Thus, to keep the film from running, only a thin film was applied at one time. The film was heated at 100 C for 1 hour and then another thin film was applied, etc. When the desired thickness of 25±2 micrometers was obtained, the film was cured. The curing procedure was to heat the film at 100 C for 1 hour and then heat it for 2 hours at 300 C.

EXPERIMENTAL PROCEDURE

The procedure for conducting the wear tests was as follows: a rider and a disk (with applied polyimide film) were inserted into the friction apparatus. The test chamber was sealed, and dry argon (<20 ppm H_2O), dry air (<20 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.

When the purge was completed, the temperature of the disk was slowly raised to the desired temperature by induction heating. The temperature was held for 10 minutes to allow it to stabilize. The disk was then rotated at 1000 rpm and a 1-kilogram load was applied.

Each test was stopped after 1 kilocycle (1 min.) of sliding. The rider and disk were removed from the friction apparatus and the contact areas were photographed. A surface profile of the wear track on the polyimide film was also taken. The rider and disk were then placed back into the apparatus and the previous test procedure was repeated. The rider was not removed from the holder when it was photographed, and locating pins in the apparatus insured that it was returned to its original position. The same was true for the disk.

Each test was stopped and the previous procedure repeated at intervals of 1, 5, 15, 30, and 60 minutes, respectively. For those tests which were run longer than 60 minutes, random times were chosen to stop the tests and examine the specimens.

RESULTS AND DISCUSSION

Friction Coefficient

In a previous study (21), a transition from high friction to low friction and from short endurance lives to long endurance lives was found to occur in either dry argon (<20 ppm H_2O) or dry air (<20 ppm H_2O) somewhere between 25 C and 100 C. The same study revealed that when water vapor was present in the air (10 000 ppm H_2O) the transition was shifted upward to somewhere between 100 C and 200 C.

To ascertain if a wear transition accompanied the friction transition, an experimental program was conducted in each of the previously mentioned atmospheres at test temperatures of 25 C, 100 C, and 200 C. Figures 2 to 4 present the friction results of that study. The figures depict representative friction traces (for the first 60 kilocycles of sliding) which occurred at each test temperature and in each test atmosphere. The gaps in the traces represent the intervals when the tests were stopped so that wear measurements could be made.

Figure 2 illustrates the effect of atmosphere on the friction coefficient at 25 C. The highest value for the friction coefficient was obtained in dry air, while the lowest was obtained in moist air. The most likely reason for the reduction in friction in moist air is that absorbed H_2O reduced the adhesion occurring between the sliding surfaces. Further evidence of this is seen in the dry air and dry argon tests. On startup, the friction coefficient always started out at a lower value than its resultant average value. Possibly this reduction was due to H_2O which was absorbed during the interval when wear measurements were made. This will be discussed in a later section in terms of wear.

Figure 3 gives representative friction traces for the tests conducted at 100 C. Comparing these results to those at 25 C shows that the effect caused by the atmosphere has changed. Instead of moist air giving the lowest value of friction coefficient, it now gives the highest, and the friction trace is very erratic. Also, at 100 C, the films seemed to exhibit some sort of "run-in" phenomena, whereas at 25 C they did not. "Run-in" is a process whereby the friction coefficient starts out high and then drops to some low value; this was postulated in Ref. 22 to be due to orientation taking place at the sliding interface. An exception to this was in moist air at 100 C. On subsequent start-ups, after stopping the tests to measure wear, no "run-on" was observed.

At 200 C, the friction tests were not stopped until 60 kilocycles of sliding were completed. Figure 4 gives those results. The average values for the friction coefficients obtained in dry argon and dry air were very similar to their respective values obtained at 100 C. In moist air however, the average value obtained at 200 C was much lower than that obtained at 100 C. This indicates that between 100 C and 200 C, a friction transition similar to that which occurred in dry air (at a lower temperature) is taking place.

Even though the average friction coefficient was lower in moist air at 200 C than at 100 C, the friction trace was still somewhat erratic. After various intervals of sliding, a sudden increase in the friction coefficient occurred. This only lasted a few seconds and then the friction coefficient usually would drop to a value lower than its previous steady-state value. Apparently the sliding surfaces briefly lost their ordered configuration either because of the wear of the film itself or because of the disruption of the transfer film on the rider. The effect was that the friction suddenly increased until reordering took place.

Table 1 summarizes the average friction coefficients which occurred during various sliding intervals over the lives of the tests. Also shown in the table is the variation from the average which occurred in each interval.

Wear

Polyimide film wear was studied by stopping the tests (after 1, 5, 15, 30, and 60 kilocycles of sliding) and taking surface profiles of the film wear track. Figure 5 gives representative surface profiles for experiments conducted at 25 C in each of the three test atmospheres. The figure illustrates that atmosphere does not seem to greatly affect the rate at which the film wears at 25 C. The shape of the cross-sectional area varies slightly for each atmosphere, but the difference is no more than one would expect from variations from test to test or from variations around the circumference of the track. Wear to the polyimide film at 25 C consisted of a gradual process of wearing the film away until metallic contact occurred.

Similar wear tests were conducted at 100 C and 200 C. Figure 6 compares the wear at each test temperature and in each test atmosphere after 60 kilocycles of sliding. At 100 C and 200 C, the wear in dry air and dry argon were reduced considerably compared to the wear at 25 C. In fact, in dry argon, the wear was so small that it was undetectable in the first 60 kilocycles of sliding. It is thus apparent that, in dry argon and in dry air a wear transition of some sort accompanied the friction transition which was found to occur at 40±10 C. When moisture was present in the air it was found that the friction transition was translated to a higher temperature, somewhere between 100 C and 200 C. The surface profiles of Fig. 6 indicate that a similar effect has occurred to the wear transition in moist air.

In order to quantify the wear process, film wear was calculated by measuring the cross sectional area of the film wear track (from the surface profiles) after each sliding interval. These values for the 25 C tests are plotted in Fig. 7 as a function of the number of sliding revolutions (expresses in kilocycles). The general trend of the polyimide film wear at 25 C is that it increases in a linear manner (from zero) as a function of the number of sliding revolutions. By fitting the best straight line to the points and taking its slope, the wear rate was found to be 4×10^{-6} cm^2/kilocycle. Wear rate is expressed in terms of cross-sectional-area and kilocycles of sliding rather than in terms of wear volume and sliding distance because it is felt these parameters are more representative of the wear process involved in this test configuration.

In order to quantify the wear at 100 C and 200 C, sliding was continued for an extended period of time. At various intervals the tests were stopped and surface profiles of the wear tracks were taken. Figures 8 and 9 present the test results of those tests. The figures indicate that, in general, for any particular set of experimental conditions the wear rate is constant. The one exception existed at 200 C in a dry argon atmosphere between 600 and 900 kilocycles of sliding; here the wear rate changed from 0.02×10^{-6} to 0.2×10^{-6} cm^2/kilocycle. Since this change was found to be very reproducible, the wear process must have changed.

Figure 10 sums the results on the effect of temperature and atmosphere on the wear rate and friction coefficient of polyimide films. The friction properties and wear properties of the films are shown to correlate very well. The main difference is at 25 C, where the wear rate is relatively insensitive to atmosphere but the friction coefficient is very dependent on atmosphere. The figure illustrates that, in dry air and dry argon, a transition from high friction to low friction and from high wear to low wear takes place between 25 C and 100 C; while in moist air, this transition has been translated to between 100 C and 200 C. The figure also exemplifies the large variations in wear and friction coefficient that can occur just by varying the experimental atmosphere.

Surface Appearance

During the intervals when the tests were stopped for wear measurements, photomicrographs of the rider transfer films and polyimide film wear tracks were taken. Figures 11 and 12 illustrate the difference in surface appearance for tests conducted at temperatures above and below the transition in a dry argon atmosphere. The photomicrographs shown were taken after 30 kilocycles of sliding at the test temperatures of 25 C (Fig. 11) and 100 C (Fig. 12).

The difference in wear to the polyimide film wear track is very apparent. At 25 C, severe wear has taken place and considerable powdered debris is seen on both sides of the wear track. At 100 C, hardly any wear is apparent and the only indication of a wear track are striations which run parallel to the sliding direction.

Transfer to the rider is also very different above and below the transition. At 25 C the transfer film has a ridged appearance and looks in the photomicrograph of Fig. 11(a) to be more like metallic wear than a transfer film;

however, on examinating the specimen at higher magnification, it appeared to be transfer. The dark material at the leading edge of the ridges is powdered material which builds up, and this material gets compressed into the ridges. Thus the transfer at 25 C seems to be characterized by large patches of powdery material which eventually gets compressed into thick layers or ridges.

At 100 C, the transfer is very different. The ridged appearance is absent and the material is drawn out into thin plate-like sheets (although it is not obvious in Fig. 12(a)). Thus, transfer is present both above and below the transition, but the important factor seems to be the type of transfer that takes place.

At 25 C, the transfer film on the rider and the wear track on the polyimide film in dry air and dry argon look very similar. At 100 C, however, transfer and wear are somewhat different; Fig. 13 illustrates the difference. In dry air at 100 C, the wear track on the polyimide film is considerably more prominent and rougher looking than in dry argon at 100 C, but not nearly as severely worn as at 25 C. The transfer to the rider appears to be in some intermediate stage between the type of transfer obtained at 25 C and that obtained in dry argon at 100 C.

A variation in transfer was found to occur in moist air at 25 C. Figure 14 illustrates that effect. After 30 kilocycles of sliding the wear track on the polyimide film looked much like that obtained in dry argon at 25 C, but the transfer to the rider was minimal. The polyimide wear debris seemed to have flowed through the contact zone and deposited itself in the exit area. This possibly could be the reason for the lower friction coefficient obtained in moist air as compared to either dry air or dry argon at 25 C. This will be commented on later in the paper.

Another interesting phenomenon which occurred in moist air at 25 C was that, after the original polyimide film had worn away, a thin film of polyimide formed on the metallic disk surface and provided lubrication. Most likely this film formed from polyimide wear debris. When this occurred, a transfer film formed on the rider which corresponded to the newly formed film on the metallic disk surface. Lubrication by this mechanism could continue for an extended period of time. In the case of Fig. 14, the test was stopped after 400 kilocycles of sliding and failure did not appear imminent. Generally, when lubrication occurred by this mechanism, friction increased gradually with time (table 1).

CONCLUDING REMARKS

In a previous study (Ref. 22), it was found that a transition from high friction to low friction (0.23 to 0.02) took place in a dry argon atmosphere (<20 ppm H_2O) at 40±10 C. Since that time, it has been determined that a friction transition (0.35 to 0.11) also took place in a dry air atmosphere (<20 ppm H_2O) at the same temperature. The results of Ref. 21 and this study indicate that a friction transition also takes place in moist air (10 000 ppm H_2O); however for this to occur, a higher temperature must be obtained (somewhere between 100 C and 200 C).

In this study, it was apparent that in all atmospheres a wear transition (from high wear to low wear) accompanied the friction transition. The magnitude of the reduction of wear was very dependent upon the type of atmosphere in which the tests were conducted. By far the largest reduction occurred in dry argon (>600 times). In dry air the reduction was only about 40 times.

The results in the moist air test atmosphere were quite different from those obtained in either dry argon or dry air. It is well known that many polymers including polyimide tend to absorb H_2O. It was found by torsional braid analysis (22) that, when even a very small quantity of H_2O was present in the testing atmosphere, a new mechanical relaxation peak occurred. Bernier and Kline (23) believe that a peak such as this is caused by water molecules which become hydrogen bonded to the carbonyl oxygen present in the polyimide structure.

In Ref. 22 it was postulated that the transition occurred because the molecules on the surface were reordered into a structure conducive to low friction by the mechanical stresses that occurred during the sliding process. At temperatures below the transition, this reordering cannot occur since the molecules do not possess the degree of freedom necessary for reordering. It is postulated that the presence of H_2O molecules, which are hydrogen bonded to the polyimide chains, put a further constraint on the molecules ability to reorder. Thus, only when a sufficiently high temperature is reached, so as to remove the H_2O molecules (or at least make their presence negligible), can the friction and also the wear be reduced.

The photomicrographs of the riders indicate that transfer takes place, both above and below the transition; however these preliminary studies indicate that the type of transfer is very different. Below the transition, the transfer is heavy and looks much like compressed polyimide powder. At temperatures above the transition (especially in dry argon), the transfer is very thin and appears to be polyimide material drawn out into a thin lamellar-like type of structure. It is possible that the transition may be due to an ordered transfer film forming on the rider and may have nothing to do with the properties of the polyimide film itself. Preliminary work however, indicates that this is not the case and that order is also taking place on the polyimide film. As seen in Figs. 11(b) and 12(b), a considerable change in appearance has taken place in the polyimide film wear tracks at temperatures above and below the transition.

The friction coefficient and wear, which occurred to the polyimide film in a moist air atmosphere at 25 C, do not seem to be in strictest agreement. When compared to tests in dry air or in dry argon at 25 C, H_2O was found to reduce the friction coefficient but not to affect wear. From observations of transfer film formation during the first 60 cycles of sliding, it is apparent that a strongly bonded transfer film did not form. A dynamic situation seemed to occur where the polyimide debris flowed through the contact area and deposited itself in the exit area. After certain intervals of sliding, this material would break off and new material would form. While wear to the polyimide film at 25 C was relatively unaffected by transfer to the rider, it appears that friction was not. The formation of a coarse, heavy transfer film appears to be an undesirable quality as far as friction is concerned.

SUMMARY OF RESULTS

Friction and wear experiments conducted on pyralin polyimide films in dry argon, dry air, and moist air indicate the following:

1. A wear transition (from high wear to low wear) was found to accompany the friction transition (from high friction to low friction) in all three test atmospheres.

2. The transition in dry argon or dry air was previously found to occur at 40±10 C. In this study, it was found that when H_2O was present in air (10 000 ppm H_2O), the transition was shifted to a temperature somewhere between 100 C and 200 C.

3. It is postulated that, in moist air, the H_2O molecules hydrogen bond to the polyimide molecular chains and constrain their mobility so that orientation does not take place until a sufficiently high temperature is obtained.

4. Transfer to the riders was quite different above and below the transition. Below the transition, transfer was coarse and heavy; but, above the transition, transfer was thin, drawn out, and lamellar.

5. At temperatures below the transition, the wear rate did not markedly depend upon the type of atmosphere in which the experiments were conducted; however at temperatures above the transition, wear rate was strongly dependent upon the atmosphere.

6. In general, for any particular set of experimental conditions, the wear rates were found to be relatively constant for the duration of the tests.

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TABLE 1. - AVERAGE FRICTION COEFFICIENT FOR POLYIMIDE FILMS (AFTER "RUN-IN")

[Moist air (10 000 ppm H_2O), dry air and dry argon (<20 ppm H_2O).]

Average friction coefficient (deviation from average)

Sliding interval (kc)	25° C			100° C				200° C	
	Moist air	Dry air	Dry argon	Moist air	Dry air	Dry argon	Moist air	Dry air	Dry argon
0-60	0.11 (±0.03)	0.32 (±0.07)	0.22 (±0.03)	0.21 (±0.08)	0.11 (±0.02)	0.02 (±0.005)	$0.05\ ^{+0.15}_{-0.01}$	0.11 (±0.02)	0.03 (±0.01)
60-100	0.15 (±0.05)	*	*	*	0.10 (±0.02)	0.02 (±0.005)	$0.06\ ^{+0.10}_{-0.01}$	0.11 (±0.02)	0.04 (±0.01)
100-200	0.21 (±0.03)	*	*	*	0.10 (±0.02)	0.02 (±0.005)	$0.07\ ^{+0.07}_{-0.02}$	0.11 (±0.02)	0.04 (±0.01)
200-400	0.21 (±0.03)	*	*	*	0.10 (±0.02)	0.02 (±0.005)	$0.13\ ^{+0.02}_{-0.08}$	0.11 (±0.04)	0.04 (±0.01)
400-600	*	*	*	*	0.10 (±0.02)	0.02 (±0.005)	*	*	0.04 (±0.01)
600-1 200	*	*	*	*	0.10 (±0.02)	0.02 (±0.005)	*	*	0.06 (±0.02)
1 200-10 000	*	*	*	*	*	0.03 (±0.01)	*	*	*

*Test either discontinued or film worn away.

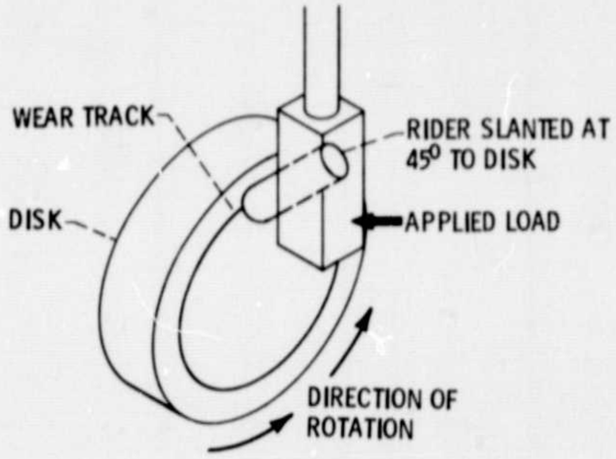


Figure 1. - Schematic diagram of friction apparatus.

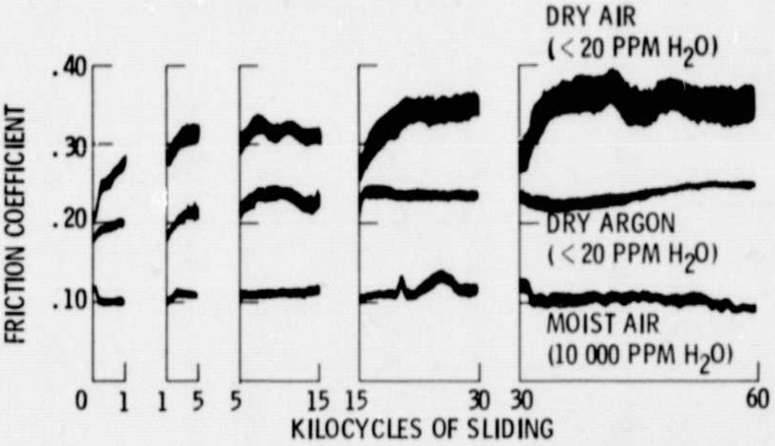


Figure 2. - Effect of atmosphere on the friction coefficient of polyimide films at 25^0 C as a function of kilocycles of sliding.

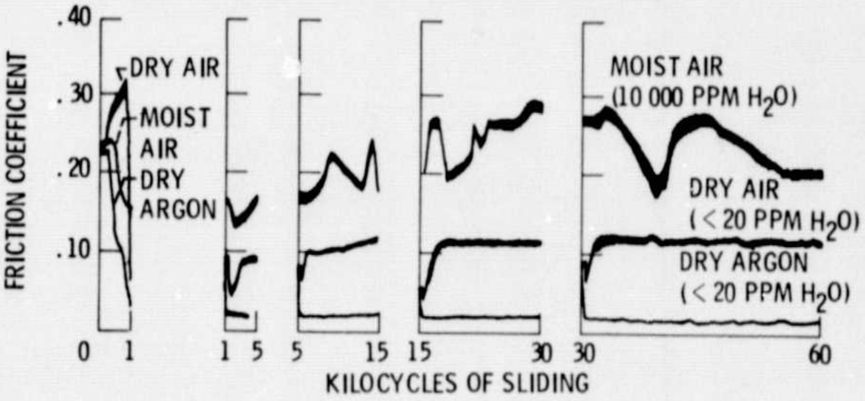


Figure 3. - Effect of atmosphere on the friction coefficient of polyimide films at 100^0 C as a function of kilocycles of sliding.

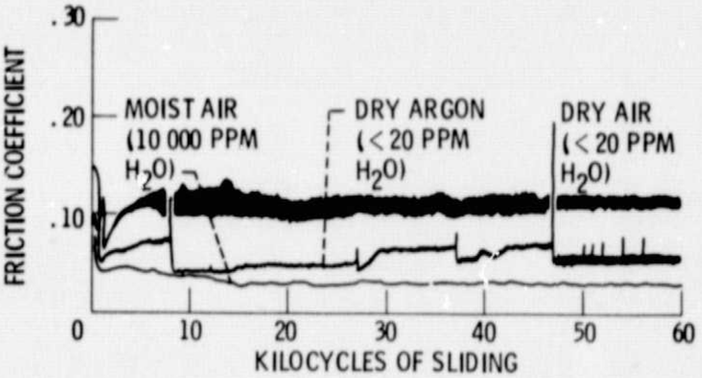


Figure 4. - Effect of atmosphere on the friction coefficient of polyimide films at 200° C as a function of kilocycles of sliding.

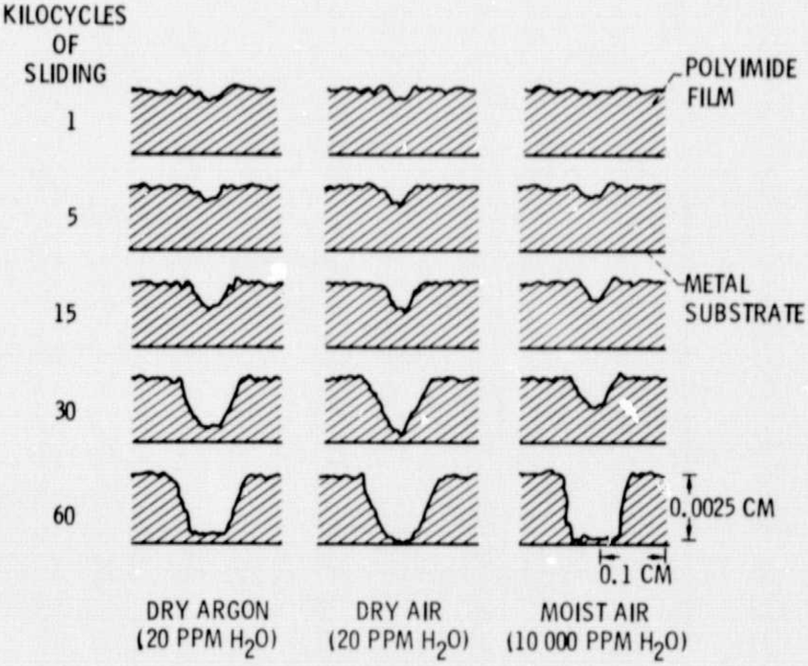


Figure 5. - Effect of atmosphere on the wear of polyimide films at 25° C.

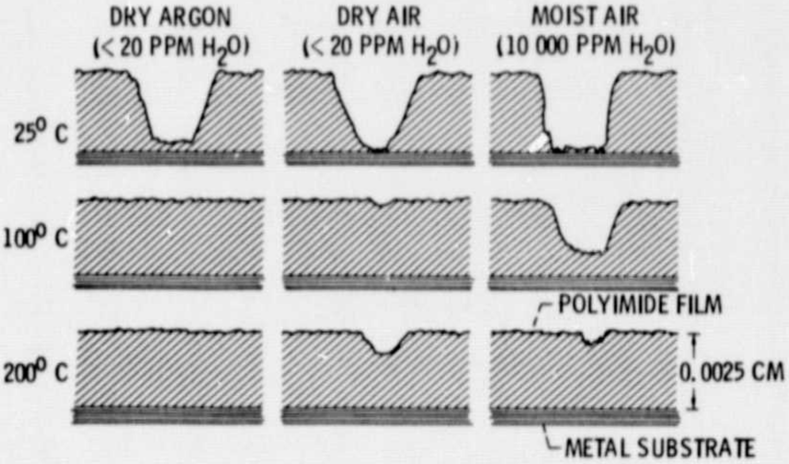


Figure 6. - Surface profiles of polyimide film wear tracks after 60 kilocycles of sliding.

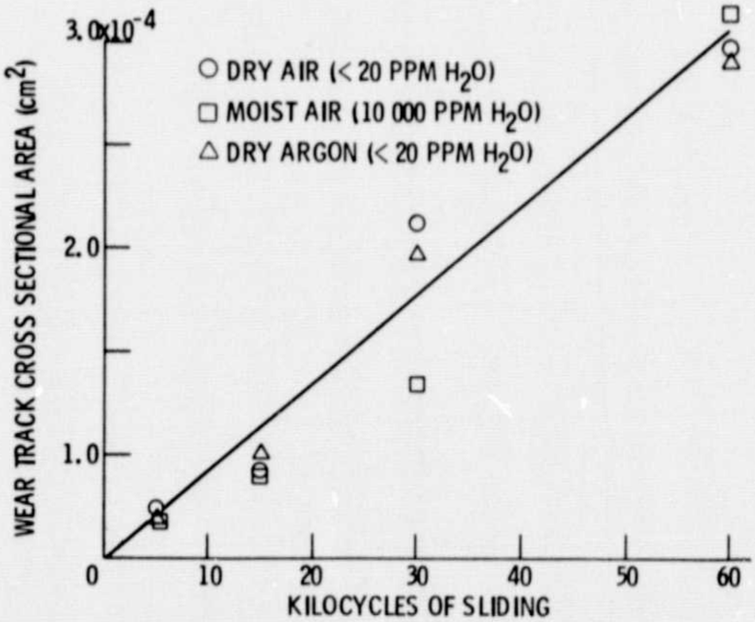


Figure 7. - Polyimide film wear at 25° C as a function of kilocycles of sliding.

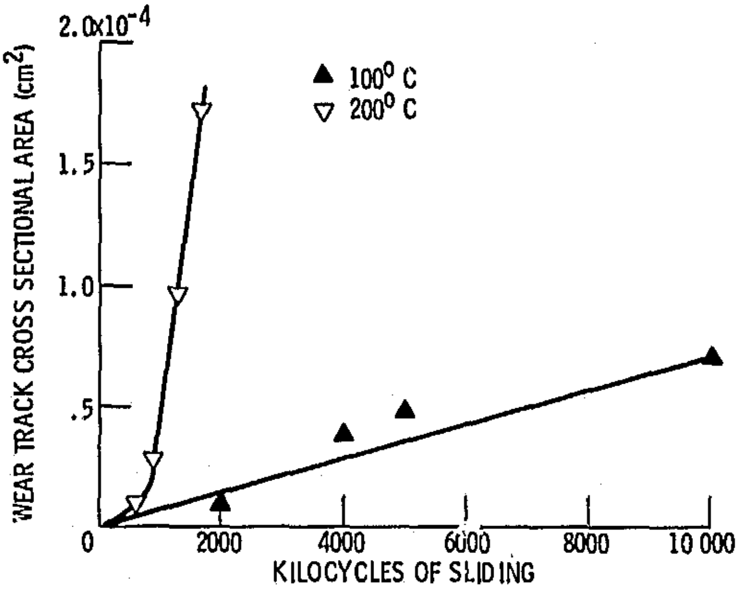


Figure 8. - Effect of temperature on the wear rate of polyimide films evaluated in dry argon.

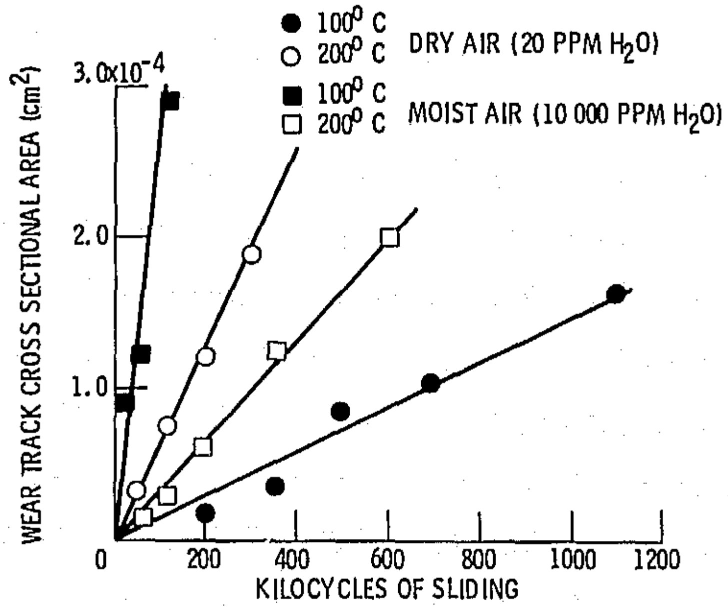
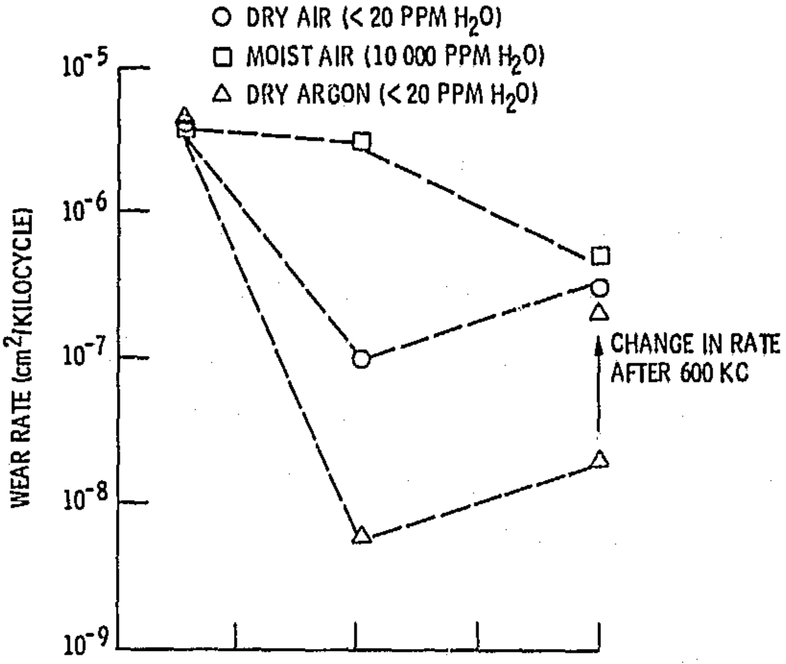
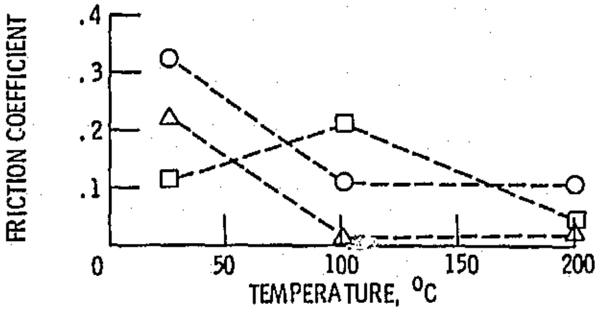


Figure 9. - Effect of temperature and moisture content of air on the wear rate of polyimide films.

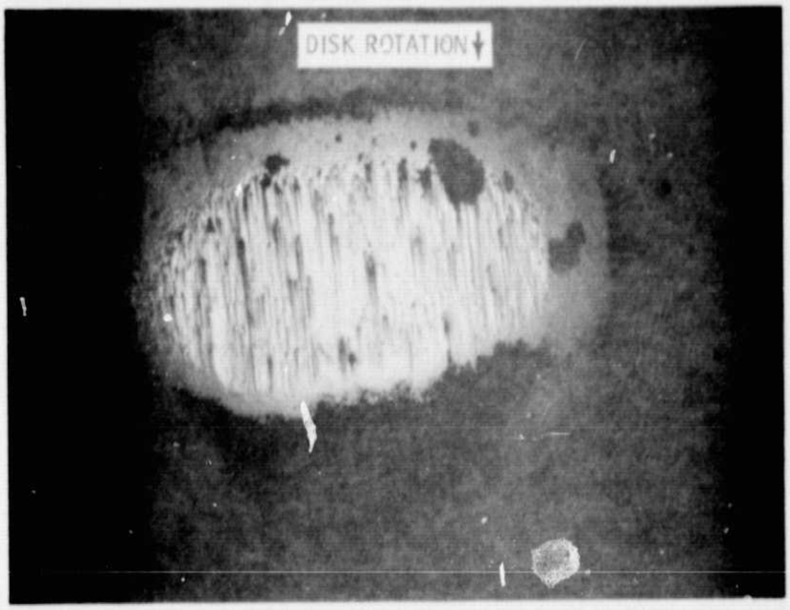


(a) WEAR RATE.

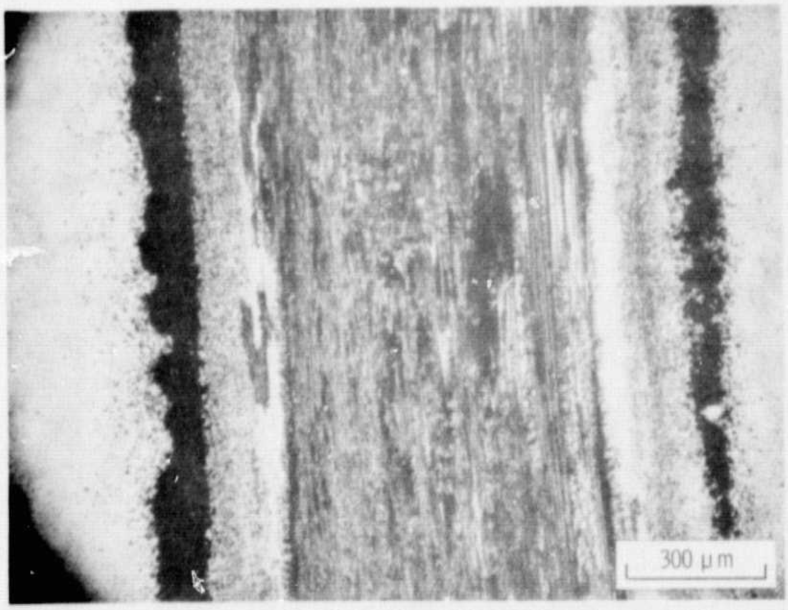


(b) FRICTION COEFFICIENT

Figure 10. - Effect of temperature and atmosphere on the wear rate and average friction coefficient of polyimide films.



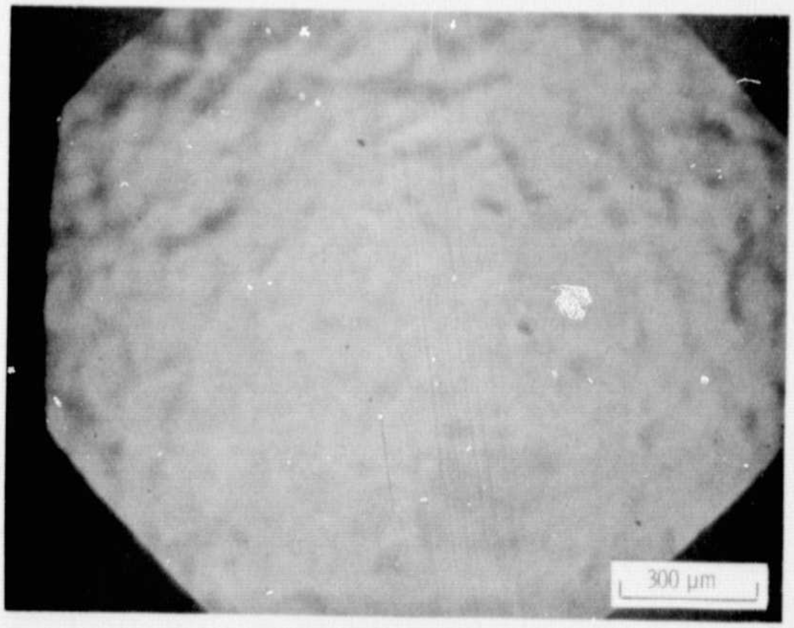
(a) RIDER TRANSFER FILM.



(b) POLYMIDE FILM WEAR TRACK.

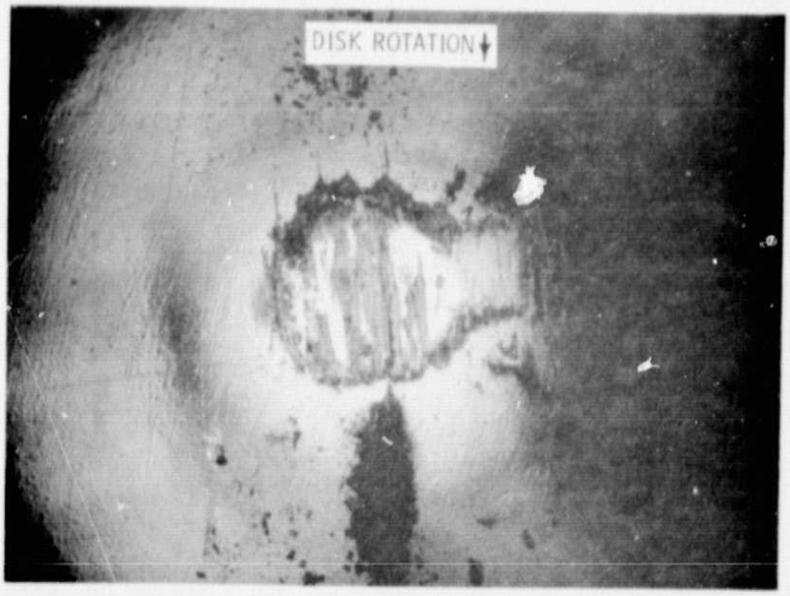
Figure 11. - Photomicrographs taken after 30 kilocycles of sliding at 25° C in a dry argon atmosphere (<20 ppm H_2O).

(a) RIDER TRANSFER FILM.

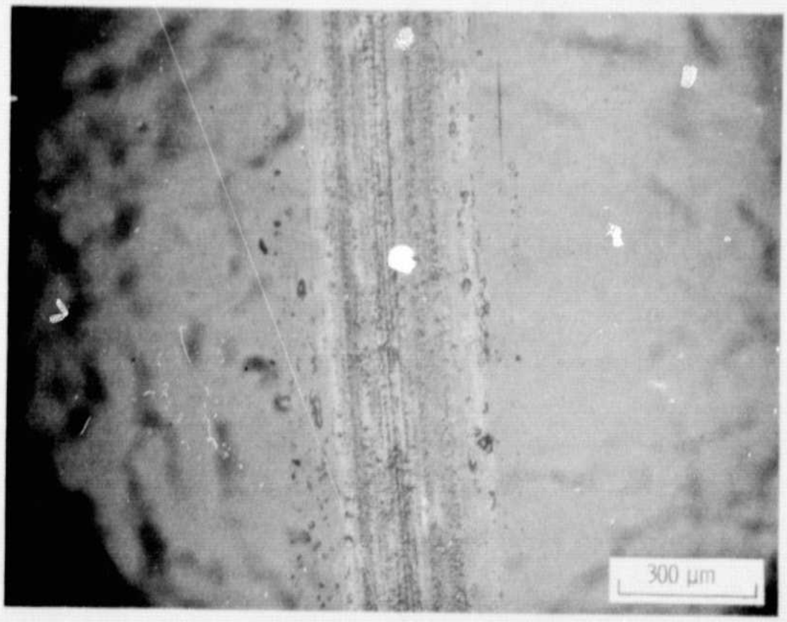


(b) POLYMIDE FILM WEAR TRACK.

Figure 12. - Photomicrographs taken after 30 kilocycles of sliding at 100° C in a dry argon atmosphere (<20 ppm H_2O).

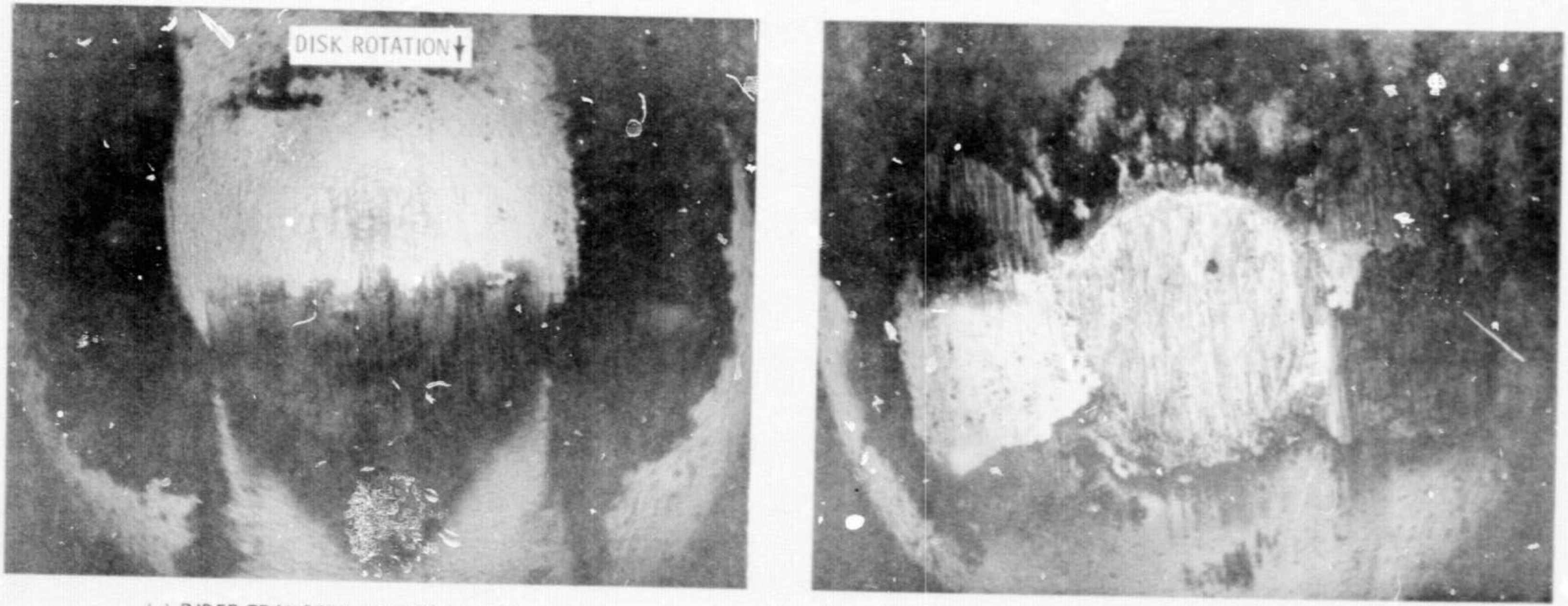


(a) RIDER TRANSFER FILM.



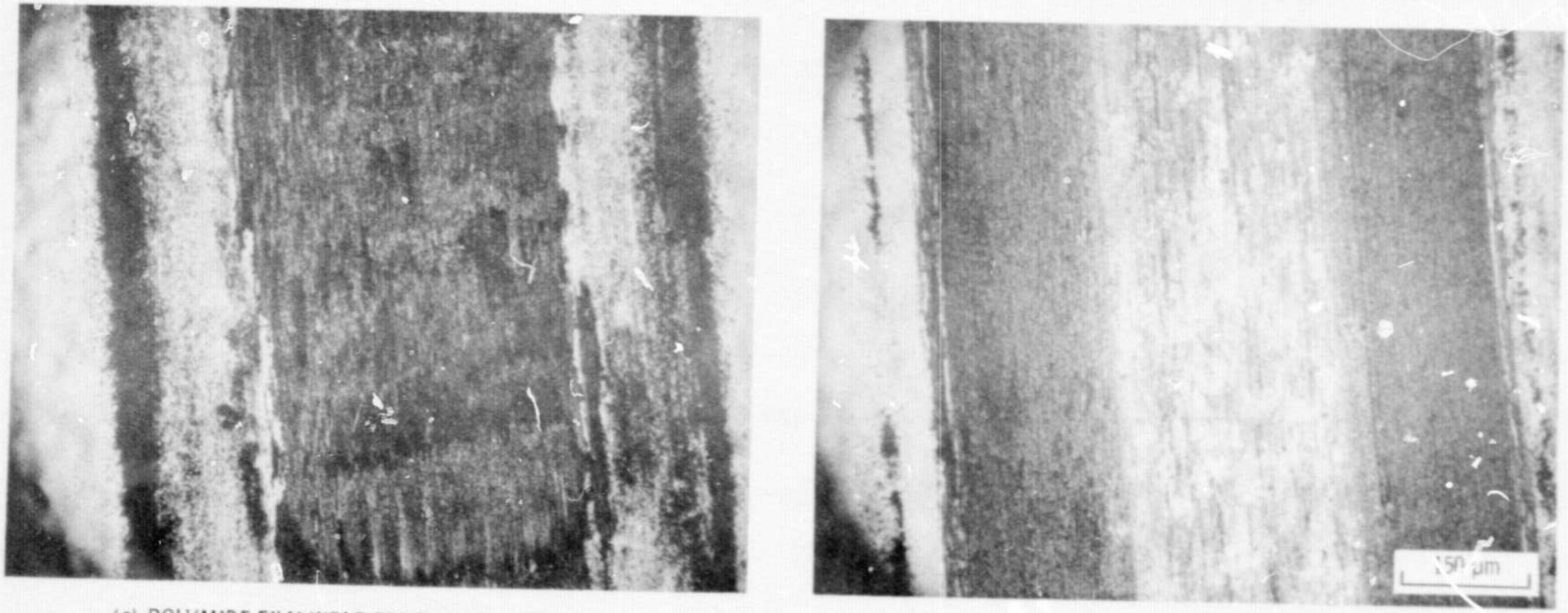
(b) POLYMIDE FILM WEAR TRACK.

Figure 13. - Photomicrographs taken after 30 kilocycles of sliding at 100° C in a dry air atmosphere (<20 ppm H_2O).



(a) RIDER TRANSFER FILM (30 kc OF SLIDING).

(b) RIDER TRANSFER FILM (400 kc OF SLIDING).



(c) POLYMIDE FILM WEAR TRACK (30 kc OF SLIDING).

(d) POLYMIDE FILM WEAR TRACK (400 kc OF SLIDING).

Figure 14. - Photomicrographs taken after 30 and 400 kilocycles of sliding in moist air, at 25° C.