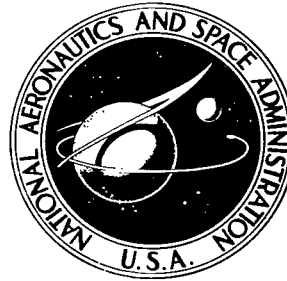


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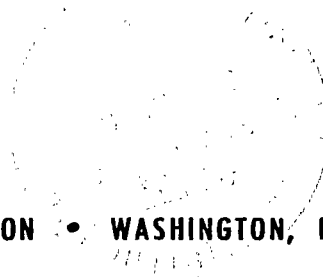
POLYIMIDE FILM WEAR -
EFFECT OF TEMPERATURE
AND ATMOSPHERE

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16. Abstract Friction and wear experiments conducted on polyimide films bonded to 440C stainless steel disks indicated that a wear transition (from high wear to low wear) accompanied a friction transition (from high friction to low friction). In dry argon or dry air (<20 ppm H ₂ O), the transition was found to occur at 40 ^o ±10 ^o C; when H ₂ O was present in air (10 000 ppm H ₂ O), the transition was found to be elevated to a temperature between 100 ^o and 200 ^o C. Wear rate calculations made at 25 ^o , 100 ^o , and 200 ^o C indicated that film wear was relatively constant for particular experimental conditions; however, the rate was strongly dependent on temperature and atmosphere.			
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POLYIMIDE FILM WEAR - EFFECT OF TEMPERATURE AND ATMOSPHERE

by Robert L. Fusaro

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SUMMARY

A pin-on-disk type of friction apparatus was used to investigate the wear properties of polyimide films ($25 \pm 2 \mu\text{m}$ thick) bonded to 440C stainless steel disks. Experiments were conducted in dry argon ($<20 \text{ ppm H}_2\text{O}$), dry air ($<20 \text{ ppm H}_2\text{O}$), and moist air ($10\,000 \text{ ppm H}_2\text{O}$) at a speed of 1000 rpm (2.6 m/sec), a load of 1 kilogram, and temperatures of 25° , 100° , and 200° C .

A wear transition (from high wear to low wear) was found to accompany a friction transition (from high friction to low friction). The transition occurred in dry argon or in dry air at $40^\circ \pm 10^\circ \text{ C}$; when moisture was present in air, the transition was elevated to a temperature between 100° and 200° C . It was postulated that H_2O molecules hydrogen bonded to the polyimide chains and constrained molecular orientation until a sufficiently high temperature was reached so as to remove the H_2O molecules or at least make their presence negligible.

Wear rate calculations indicated that polyimide film wear was relatively constant for any particular set of experimental conditions. At temperatures below the transition, the wear rate was not markedly affected by the atmosphere in which the film was evaluated; however, at temperatures above the transition, the wear rate was strongly dependent on atmosphere. By far the best results were obtained in a dry argon atmosphere where the wear to the film at 100° C was reduced about 600 times the value obtained at 25° C . In dry air the reduction was only about 40 times.

The study also indicated that rider wear was minimal, if not nonexistent, until the films were worn away and the metal substrate exposed. Flat looking areas on the riders consisted of transferred polyimide material.

It was also observed that after the original polyimide film was worn away, a secondary film could form on the surface (from polyimide wear debris) and provide lubrication for an extended period of time.

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 (refs. 1 to 17).

Polyimide refers to a general class of long chain polymers which have recurring imide groups as an integral part of the main chain. The mechanical properties can be altered while still maintaining the basic polyimide structure by varying the monomeric starting materials and by controlling cure procedure (refs. 18 to 20). But before polyimides can be synthesized with improved lubrication properties, a 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 atmospheric dependent (ref. 21). It was shown in reference 21 that a transition from low friction to high friction and from long wear lives to short wear lives occurred in dry argon or in dry air (<20 ppm H₂O) somewhere between 25° and 100° C. At temperatures above this transition, the films exhibited much longer wear lives and considerably lower friction coefficients than at temperatures below the transition.

A more detailed study of the friction transition was reported in reference 22, and the transition was found to occur in dry argon (<20 ppm H₂O) at 40°±10° C. It was postulated in reference 22 that the transition was due to a reordering of the surface molecules into a structure conducive to easy shear, such as an extended chain molecular structure with the chains parallel to the sliding direction. For orientation 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 H₂O), dry air (<20 ppm H₂O), and moist air (10 000 ppm H₂O). To accomplish this, a photographic 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

The apparatus used to measure the friction coefficient and to evaluate the wear lives of the solid lubricant films is illustrated in figure 1. Basically, the device

consists of a flat (6.3-cm-diam) disk in sliding contact with a stationary (0.476-cm-rad) hemispherically tipped rider. A 1-kilogram load is applied to the rider as the disk rotates at 1000 rpm. The rider slides on a 5-centimeter-diameter track on the disk and obtains a linear sliding speed of 2.6 meters per second.

Induction heating is used to heat the disk. This is accomplished by placing an induction coil around the circumferential surface of the disk. The temperature is monitored by a thermocouple in contact with the disk surface. A micrometer is used to move the thermocouple away from the disk before it is set in motion. The temperature is then monitored by an infrared pyrometer focused on the wear track of the disk.

A strain gage senses the frictional force, which is 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} to 1.25×10^{-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 then 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 the desiccator. No polyimide films were 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 to 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 H₂O), dry air (<20 ppm H₂O), or moist air (10 000 ppm H₂O) 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 (ref. 21), a transition from high friction to low friction and from long endurance lives to short endurance lives was found to occur in either dry argon (<20 ppm H₂O) or dry air (<20 ppm H₂O) somewhere between 25^o and 100^o C. The same study revealed that when water vapor was present in the air (10 000 ppm H₂O) the transition was shifted upward to somewhere between 100^o and 200^o C. An objective of this study was to attempt to correlate friction properties to wear properties of polyimide films to ascertain if a wear transition accompanied the friction transition.

To accomplish this, an experimental program was conducted in each of the previously mentioned atmospheres at test temperatures of 25^o, 100^o, and 200^o 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 tem-

perature 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^o 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 this reduction appears to be that absorbed H₂O reduced the adhesion occurring between the sliding surfaces. Further evidence of this is seen in dry air and dry argon tests. On startup, the friction coefficient always started out at a lower value than its resultant average value. Most likely this reduction was due to H₂O 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^o C. Comparing these results to those at 25^o 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^o C the films seemed to exhibit some sort of "run-in" phenomena, whereas at 25^o 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 reference 22 to be due to orientation taking place at the sliding interface. The exception to this was in moist air; on subsequent startups after stopping the tests to measure wear, no "run-in" was observed. Apparently, moisture in this case is detrimental to the orientation phenomena.

At 200^o 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^o C. In moist air, however, the average value obtained at 200^o C was much lower than that obtained at 100^o C; this indicates that between 100^o and 200^o C a 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^o C than at 100^o 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 what happened was that 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 reorientation took place.

Figure 5 gives the results of the effects of temperature and atmosphere on the friction coefficient. This figure shows the transition regions and the large variation in the friction coefficient that can occur just by varying the test atmosphere.



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 6 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 consisted of a gradual process of wearing the film away until metallic contact occurred.

Similar wear tests were conducted at 100° and 200° C. Figure 7 compares the wear at each test temperature and in each test atmosphere after 60 kilocycles of sliding. At 100° 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° and 200° C. The surface profiles of figure 7 indicate that a similar effect has occurred to the wear transition in moist air.

Photomicrographs of the polyimide film wear tracks, which were taken after 60 kilocycles of sliding, are shown in figure 8. The photomicrographs compliment what was previously discussed concerning the surface profiles. The white looking material on the film surfaces is polyimide wear debris.

Photomicrographs of rider transfer films were also taken after the same interval of sliding. Figure 9 compares those photomicrographs. Transfer films were found to be present both above and below the transition temperature. In general, transfer films seemed to form very rapidly, and regardless of when the tests were stopped (as long as the polyimide film on the metallic disk was not worn through), a transfer film of some sort was always present. They were tenaciously bound and very hard to remove; however, when they were removed, the rider surface was still spherical underneath, indicating macroscopic wear was only occurring on the polyimide film.

One variation in transfer was found in moist air at 25° C. Transfer to the rider was minimal; the polyimide wear debris seemed to have flowed through the contact zone and was deposited in the exit area. This difference in transfer could possibly be the reason for the lower friction coefficient which was obtained in moist air as compared to dry argon or dry air at 25° C.

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. This can be seen in the surface profiles of figure 8 and the photomicrographs of figure 9. Most likely this film formed from polyimide wear debris. When this occurred, a rider transfer film formed on the metallic rider which corresponded to the newly formed film on the metallic disk surface (fig. 9). It was found that lubrication by this mechanism could continue for an extended period of time (up to 400 kilocycles); however, the friction coefficient also rose gradually with time.

Wear Rate

In order to quantify the wear process, film wear was calculated by measuring the cross-sectional area of the polyimide film wear tracks (from the surface profiles) after each sliding interval. These values for the 25^o C tests are plotted in figure 10 as a function of the number of sliding revolutions (expressed in kilocycles). The general trend of the polyimide film wear 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 the slope, the wear rate was found to be 4×10^{-6} square centimeter per kilocycle. The 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 was felt that these parameters were more representative of the wear process involved in this test configuration.

In order to quantify the wear at 100^o and 200^o C, sliding was continued until the films were worn away. At various intervals the tests were stopped and surface profiles of the wear tracks were taken. Figure 11 presents the results of those tests. The figure indicates that, in general, for any particular combination of atmosphere and temperature the wear rate was constant. The one exception existed at 200^o 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} square centimeter per kilocycle. Since this change was found to be reproducible, the wear process must have changed.

As mentioned previously, the wear rate at 25^o C did not depend on the type of atmosphere in which the experiments were conducted; however, at 100^o and 200^o C the atmosphere did have a very pronounced effect on the wear rate. Figure 12 presents the polyimide film wear rate as a function of temperature for each test atmosphere. The figure illustrates the wide variations in wear rates that were obtained.

If figure 12 is compared to figure 5, the friction properties of the films and the wear properties of the films are shown to correlate very well. The main difference is at 25^o C where the wear rate is relatively insensitive to atmosphere but the friction

coefficient is quite dependent on atmosphere. The figures illustrate 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° and 100° C. In moist air this transition has been translated to between 100° and 200° C.

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 H₂O) 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 H₂O) at the same temperature. The results of reference 21 and this study indicate that a friction transition also takes place in moist air (10 000 ppm H₂O); however, for this to occur, a higher temperature must be obtained (somewhere between 100° 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 dependent on 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. Since the wear rates in dry argon and dry air were equivalent at 25° C, it implies that oxidation may play a role in the wear process at elevated temperatures.

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 tend to absorb H₂O, and polyimide is of this type. It was found by torsional braid analysis (ref. 22) that when even a very small quantity of H₂O was present in the testing atmosphere a new mechanical relaxation peak occurred. Bernier and Kline (ref. 23) believe that this peak is caused by water molecules which become hydrogen bonded to the carbonyl oxygen present in the polyimide structure. These extra molecules, hydrogen bonded in the structure, most likely are the cause of the transition being shifted to higher temperatures.

In reference 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. The presence of H₂O molecules, which are hydrogen bonded to the polyimide chains, puts a further constraint on the molecules ability to reorder. Thus, only when a sufficiently high temperature is reached, so as to remove the H₂O molecules (or at least make their presence negligible), can the friction and also the wear be reduced.

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, H₂O 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 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, however, it was found that when H₂O was present in air (10 000 ppm H₂O), the transition was shifted to a temperature somewhere between 100° and 200° C.

3. It was postulated that in moist air the H₂O molecule, hydrogen bonded to the polyimide chains, constrained the molecular mobility so that orientation did not take place until a sufficiently high temperature was obtained.

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

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

6. Essentially no wear occurred to the riders until the films were worn away and the metal substrate exposed. Flat areas on the riders consisted of transferred polyimide material.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, March 8, 1976,
505-04.

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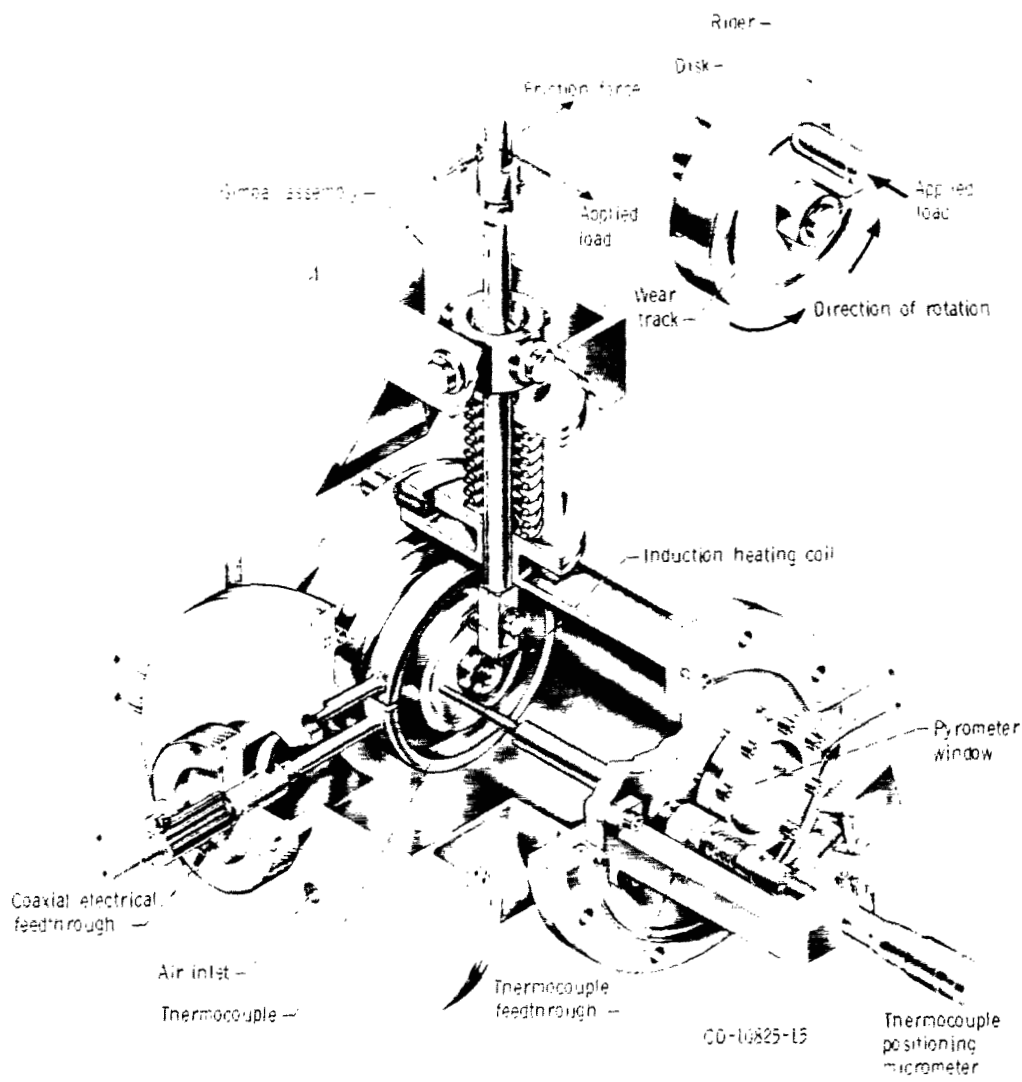


Figure 1. - Friction and wear apparatus.

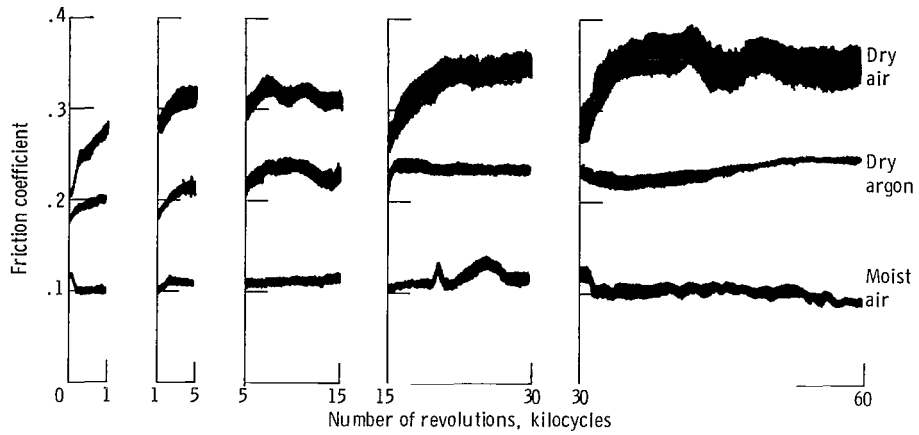


Figure 2. - Effect of atmosphere on friction coefficient of polyimide films at 25⁰ C as function of repeated passes (number of revolutions). Rider and disk material, 440C stainless steel; sliding velocity, 2.6 meters per second; load, 1 kilogram; dry argon (<20 ppm H₂O), dry air (<20 ppm H₂O), moist air (10 000 ppm H₂O).

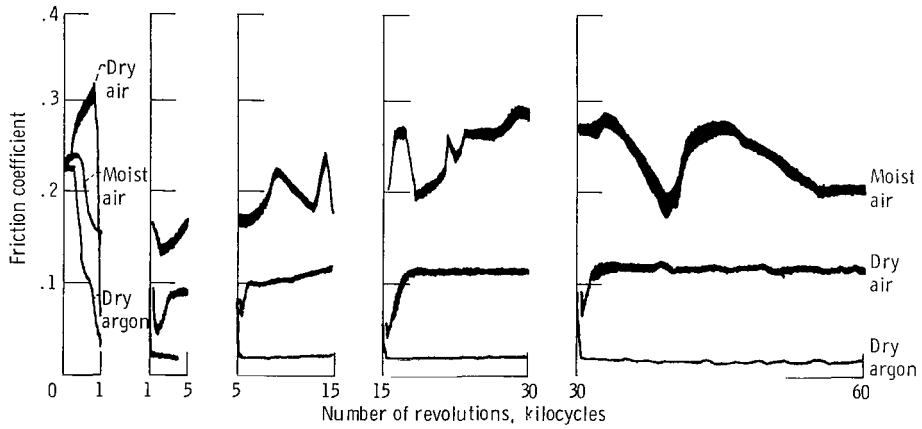


Figure 3. - Effect of atmosphere on friction coefficient of polyimide films at 100⁰ C as function of repeated passes (number of revolutions). Rider and disk material, 440C stainless steel; sliding velocity, 2.6 meters per second; load, 1 kilogram; dry argon (<20 ppm H₂O), dry air (<20 ppm H₂O), moist air (10 000 ppm H₂O).

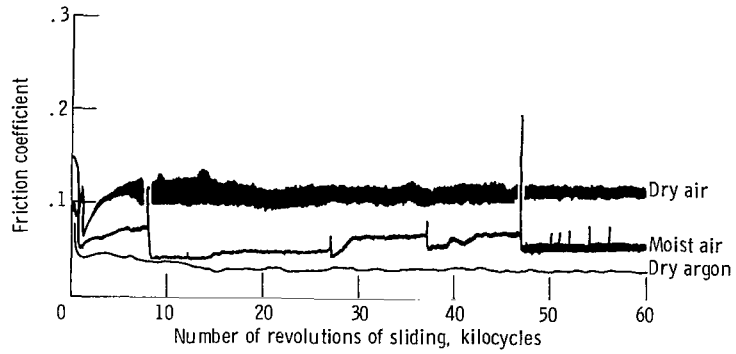


Figure 4. - Effect of atmosphere on friction coefficient of polyimide films at 200° C as function of repeated passes (number of revolutions). Rider and disk material, 440C stainless steel; sliding velocity, 2.6 meters per second; load, 1 kilogram; dry argon (< 20 ppm H₂O), dry air (< 20 ppm H₂O), and moist air (10 000 ppm H₂O).

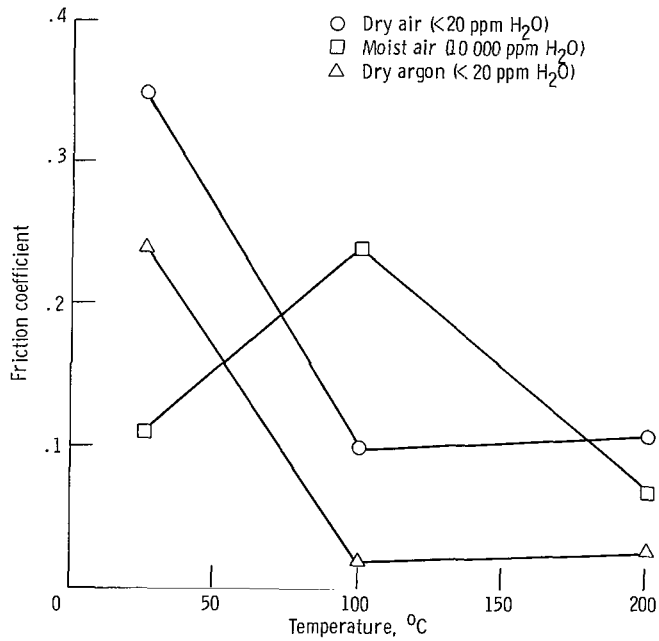


Figure 5. - Effect of temperature and atmosphere on friction coefficient of polyimide films.

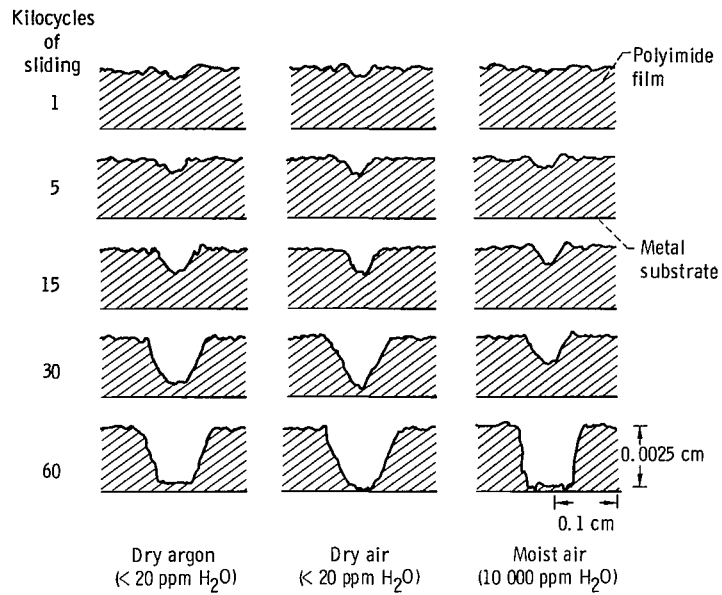


Figure 6. - Effect of atmosphere on wear of polyimide films at 25^o C. Rider and disk material, 440C stainless steel; sliding speed, 2.6 meters per second; load, 1 kilogram.

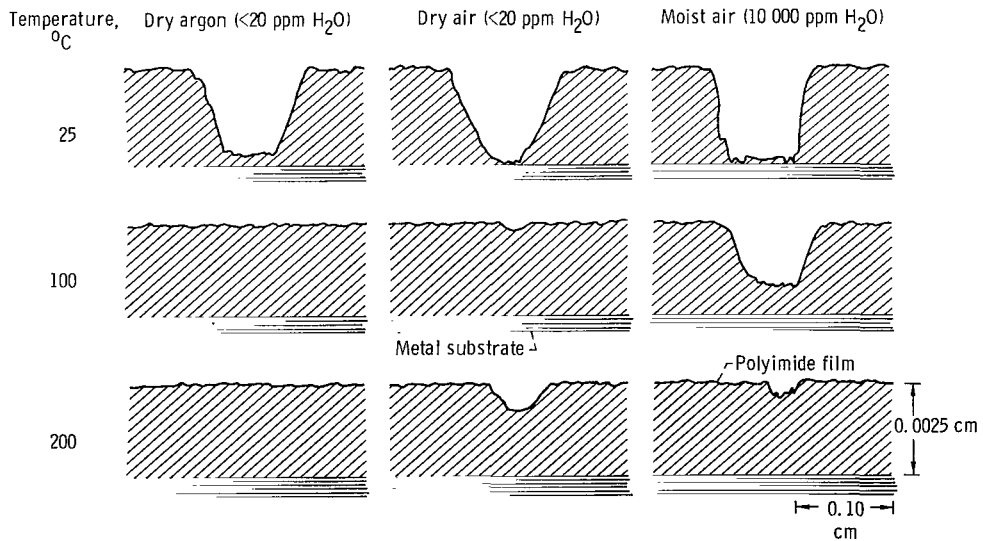


Figure 7. Surface profiles of polyimide film wear tracks after 60 kilocycles of sliding.

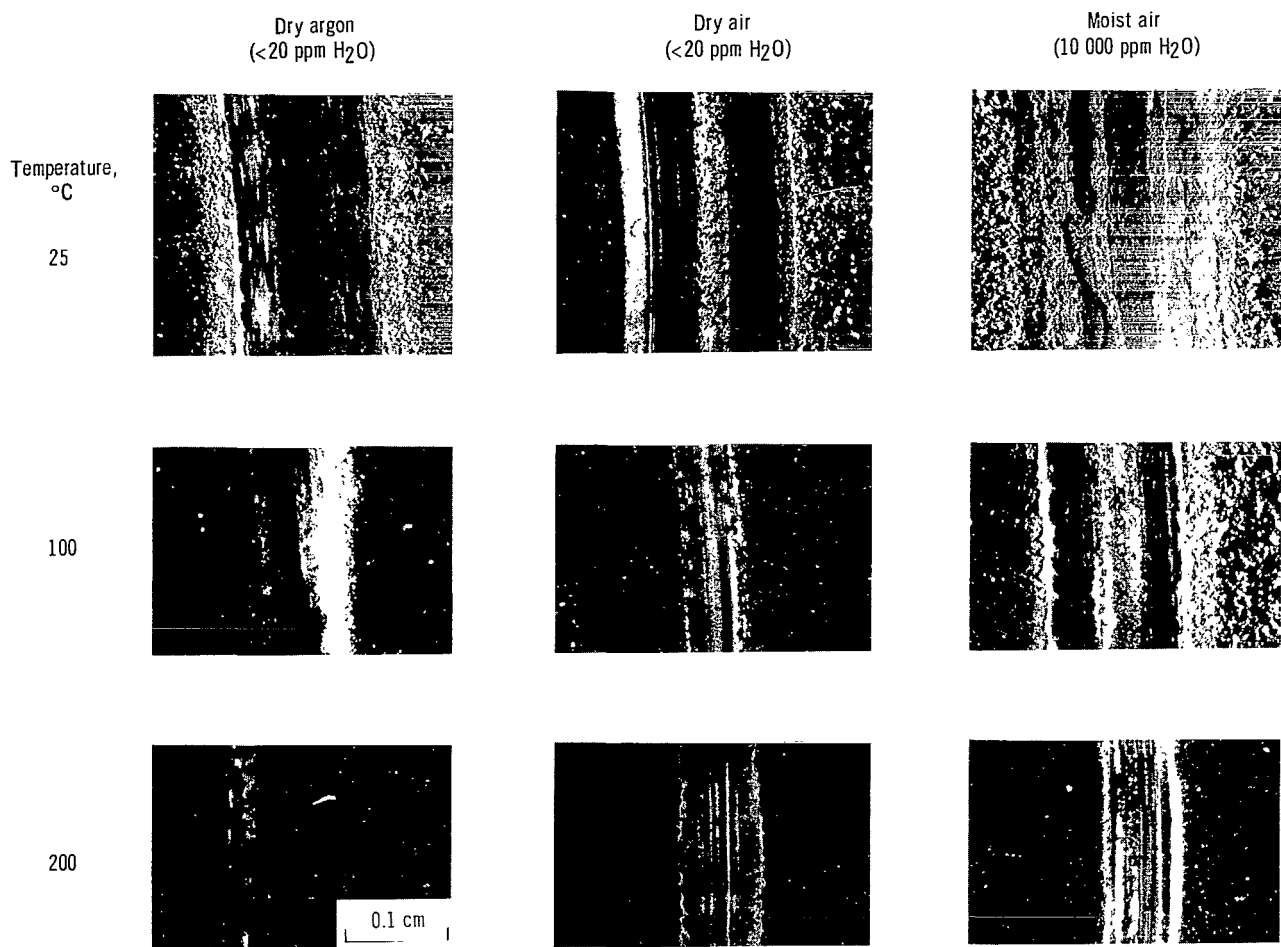


Figure 8. - Photomicrographs of polyimide film wear tracks after 60 kilocycles of sliding.



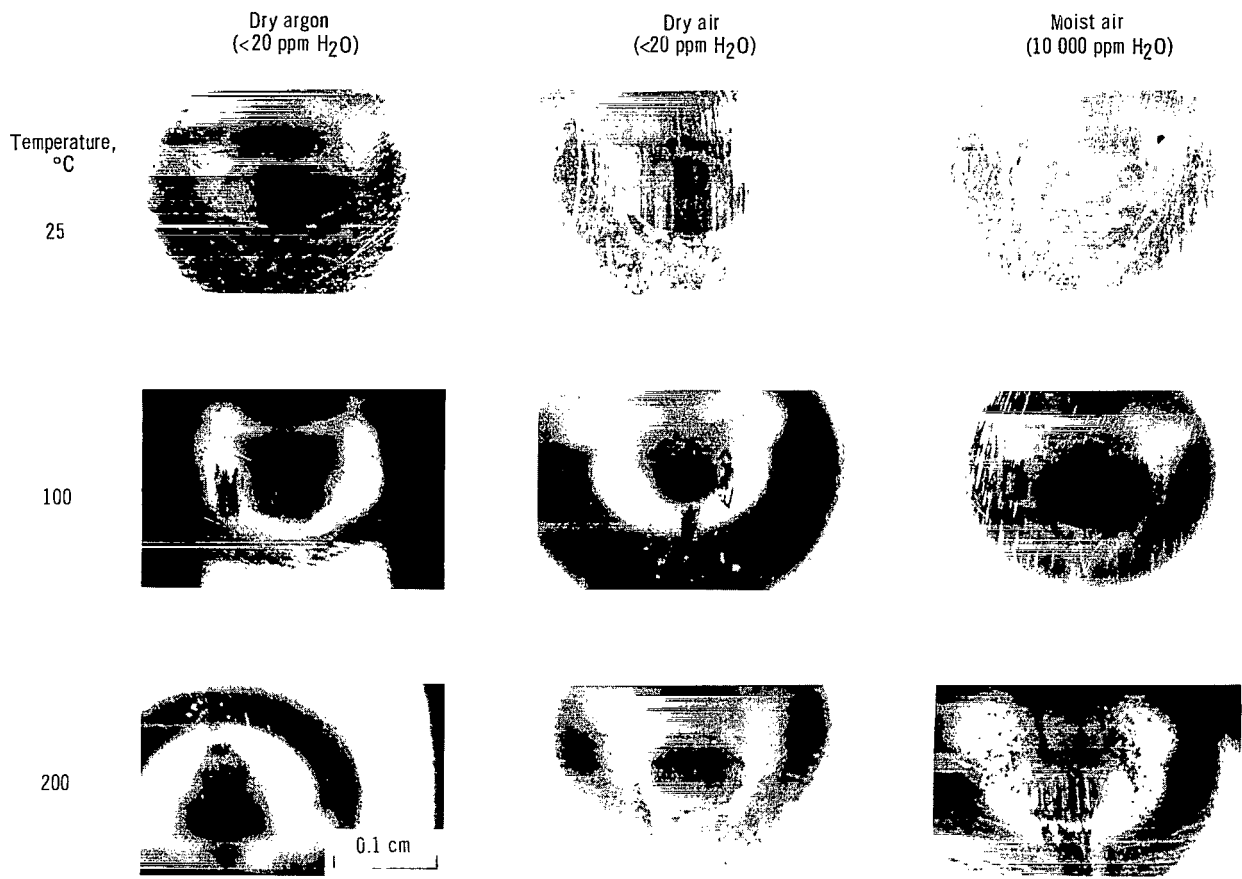


Figure 9. - Photomicrographs of rider transfer films after 60 kilocycles of sliding.

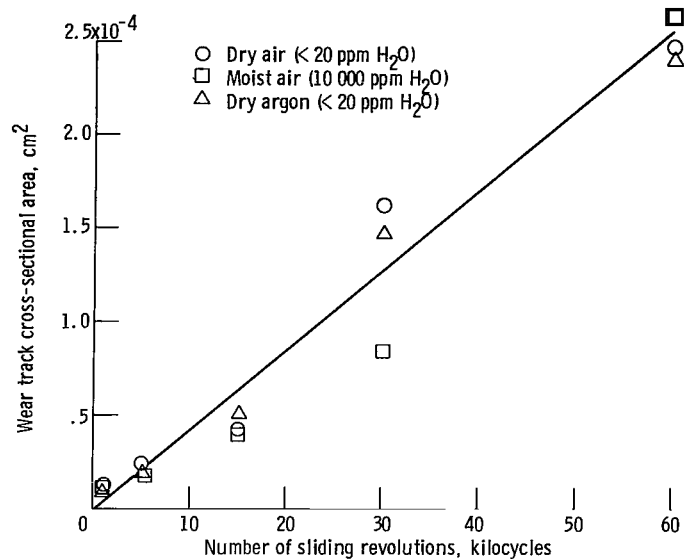


Figure 10. - Polyimide film wear at 25°C as function of kilocycles of sliding. Rider and disk material, 440C stainless steel; sliding velocity, 2.6 meters per second; load, 1 kilogram.

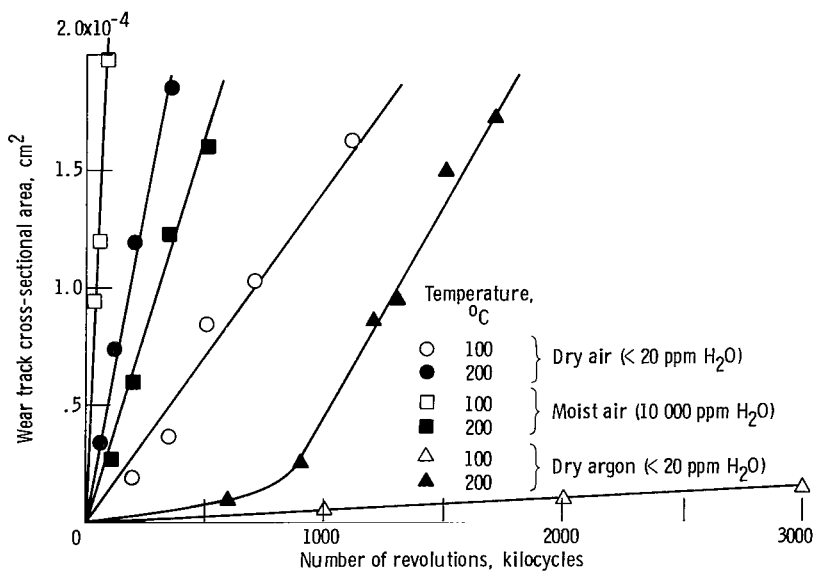


Figure 11. - Polyimide film wear of 100°C and 200°C as function of kilocycles of sliding. Rider and disk material, 440C stainless steel; sliding velocity, 2.6 meters per second; load, 1 kilogram.

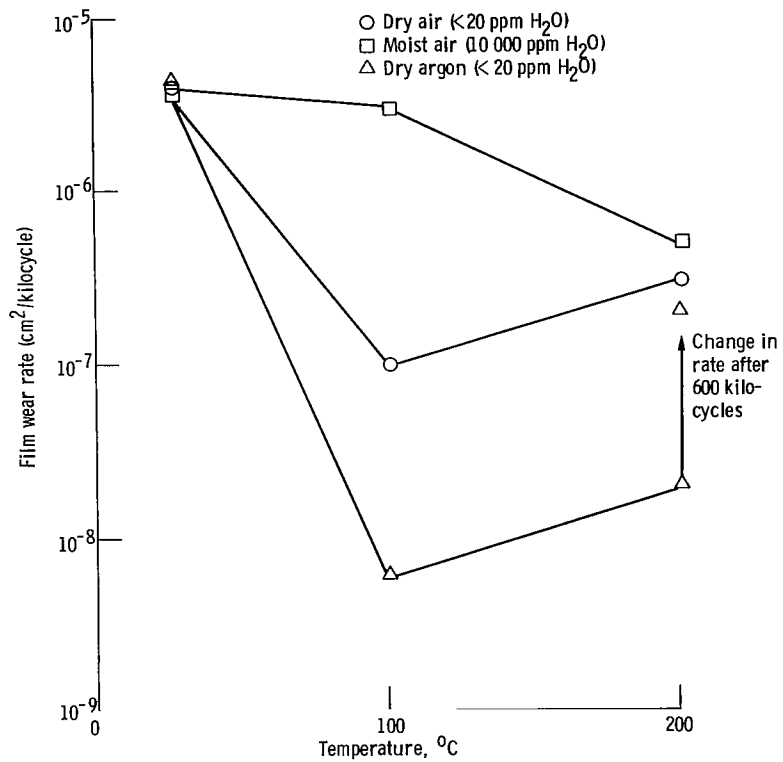


Figure 12. - Effect of temperature and atmosphere on wear rate of polyimide films.



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