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LUBRICATING CHARACTERISTICS OF POLYIMIDE BONDED GRAPHITE FLUORIDE AND POLYIMIDE THIN FILMS

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ABSTRACT

A friction and wear life study was conducted on the use of a polyimide (PI) varnish as a binder for the solid lubricant, graphite fluoride $(CF_{1.1})_n$. Also studied were the lubricating properties of PI thin films with no solid lubricant additives. For comparison, similar experiments on PI-bonded MoS_2 and burnished films of MoS_2 or $(CF_{1.1})_n$ were conducted. The best results were obtained with the PI-bonded $(CF_{1.1})_n$ films. The polyimide thin films (with no solid lubricant additives) did not lubricate well at 25 C; however, when heated to 100 C, low friction and long wear lives were obtained.

INTRODUCTION

A previous investigation (1) has shown that burnished (rubbed-on) films of graphite fluoride $[(CF_{1.1})_n]$ powders have excellent lubricating properties. The results of these experiments indicated that the minimum value of the friction coefficient for $(CF_{1.1})_n$ lubricated 440C stainless steel specimens was comparable or better than specimens similarly lubricated with molybdenum disulfide or graphite. The wear lives of the $(CF_{1.1})_n$ films were found to be up to six times better than either of the other two films.

Solid lubricants such as MoS_2 or graphite usually show improved wear life when they are incorporated into a binder system. The object of this investigation was, therefore, to determine whether $(CF_{1.1})_n$ would also have improved wear life when used with a binder material.

The binder material used in these experiments was a polyimide varnish (manufacturer's designation, PI-4701)(2). Polyimide has been shown in References 3 to 6 to be a good friction and wear material. Also, good results have been obtained when using it as a binder material for MoS_2 (7). In addition, $(CF_{1.1})_n$ was found to mix readily with the polyimide varnish. For these reasons and because it was stable in air to over 400 C, it was chosen as the binder material.

Since polyimide has been shown to have good friction and wear properties when used as a solid body (3 to 6), a secondary objective of this study was to determine how polyimide would function as a thin film lubricant without any solid lubricant additives.

MATERIALS

Graphite Fluoride

A brief review of the history and physical properties of graphite fluoride $(CF_{1.1})_n$ is given in Reference 1. After the publication of that work, a U.S. patent "Dry Lubrication", (8) (July 25, 1961), was discovered. It describes a product with a carbon-to-fluorine ratio of 1:1 but gives almost no quantitative data on lubricating characteristics.

More recently, Ishikawa and Shimada (9) have used graphite fluoride as an additive in grease, mechanical carbons, and polytetrafluoroethylene (PTFE) - fibrous carbon composites. In each instance, beneficial results were reported. They found increased load-carrying capacity, reduced surface temperatures, or seizure prevention.

Gisser, Petronio, and Shapiro (10) have also conducted experiments on the friction and wear properties of graphite fluoride. They found that, by adding 2-percent graphite fluoride to a lithium soap - diester grease, low friction could be attained at temperatures up to 344 C. The grease alone or with graphite added failed at 215 C. They also tested graphite fluoride in a silicate binder and in an epoxy-phenolic binder. In general, they found that films formulated with graphite fluoride gave better friction and wear results than similar films formulated with graphite.

Polyimides

There are many varities of polyimides (12 to 17). Basically, they are cyclic chain polymers, which are formed chemically by the reaction of pyromellitic dianhydrides and aromatic diamines. The chains consist of aromatic rings alternated with heterocyclic groups. Their basic structure is shown in Fig. 1, where R represents a thermally stable group (12, 15 to 18).

Due to the multiple bonds between the aromatic and heterocyclic molecules, the polyimides are characterized by a high thermal stability [400 C in air, 500 C in inert atmospheres (18)]. They also have a high radiation stability (15, 16, and 19); and can withstand high exposure to neutrons, electrons, ultraviolet light, and gamma radiation. They are resistant to most common chemicals and solvents, but are attacked by alkalis (16 and 19). At the decomposition point, they crumble to a fine powder without melting. For a more detailed discussion of the physical properties, see References 15 to 21.

APPARATUS DESCRIPTION

The apparatus used to measure the friction coefficient and to evaluate wear lives of the solid lubricant films is illustrated in Fig. 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, which gives it 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 which is focused on the wear track of the disk.

A strain gage senses the frictional force, which is continuously recorded on a strip-chart recorder.

PROCEDURE

Coating Formulation

The PI-bonded MoS_2 films were prepared by mising three parts (by weight) of MoS_2 powder with one part (by weight) of polyimide solids which were dissolved in a varnish-like concentrate. The PI-bonded $(CF_{1.1})_n$ films were made by mixing three parts (by weight) of $(CF_{1.1})_n$ powder with two parts (by weight) of polyimide solids. The density of $(CF_{1.1})_n$ was only about one-half that of MoS_2; therefore, the two film formulations contain equal volume percents of solid lubricant. A thinner consisting of N-methylpyrrolidone and xylene was added to each formulation to obtain a sprayable solution.

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 solid film lubricants to the disks, they were roughened by sandblasting to an rms of 0.90×10^{-6} to 1.25×10^{-6} meter.

After the disk surfaces were roughened, they were scrubbed with a brush under running water to ensure that no abrasive particles remained. A water paste of levigated alumina was next rubbed gently 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 solid lubricant.

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 until used. No solid lubricant coating was applied to the riders.

Application of Lubricant Films

Some tests were conducted using disks burnished with MoS_2 or $(CF_{1.1})_n$ powders. These coatings were applied by rubbing the powder onto the disk surface with the back of a napped polishing cloth. The polishing cloth was made of open-weave fabric (twilled) and thus served as a good applicator.

The liquid mixture of polyimide or PI-bonded solid lubricant was sprayed onto each disk using an airbrush. The coating did not dry rapidly. Thus, if more than a thin coat were applied, the liquid "ran" and a nonuniform coating resulted. To eliminate "running" and to obtain the desired coating thickness, it was necessary to spray a thin coating of the formulation onto the disk, bake it at 100 C for one hour, and then spray another thin coating on the disk and repeat the procedure.

When the desired thickness of 10 to 20 micrometers was obtained, the remainder of the curing procedure was carried out. This procedure was to bake the coating at 100 C for one hour and then to bake it for an additional hour at 300 C. After the disks had cooled, some additional solid lubricant powder was rubbed onto the polyimide - solid lubricant film by the same procedure as that used for burnishing directly on the metal surfaces. This practice was intended to increase the concentration of lubricant at the surface and thereby to improve the friction and wear life characteristics of the films.

Test Procedure

The procedure for conducting the wear life tests was as follows: a rider and a disk (with the applied solid lubricant film) were inserted into the friction apparatus (Fig. 2). The test chamber was sealed, and dry air (moisture content, 20 ppm), moist air (moisture content, 10 000 ppm), or dry argon (moisture content, 10 ppm) was purged through the chamber for 15 minutes. The flow rate was 1500 cubic centimeters per minute. This flow rate maintained a slight positive pressure in the chamber, which had a volume of 2000 cubic centimeters.

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

The arbitrary criterion for failure in these wear life tests was a friction coefficient of 0.30. An automatic cutoff system shut down the apparatus when the friction coefficient reached this value.

In order to calculate the wear rate at 25 C, the tests were stopped after one hour of sliding (60 000 cycles). The wear scar diameter on the hemispherically tipped rider was measured and wear volume per hour was calculated.

For comparison, the wear rate and friction coefficient for unlubricated 440C stainless steel were also similarly determined. The same procedure was followed; but no cutoff friction coefficient was used.

RESULTS AND DISCUSSION

Polyimide-Bonded Graphite Fluoride

Figure 3 shows representative friction traces from experiments conducted on PI-bonded graphite fluoride and PI-bonded molybdenum disulfide thin films. The experiments were conducted at 25 C in a dry air atmosphere (moisture content, 20 ppm). The speed was 2.6 m/sec (1000 rpm) and the load, 1 kilogram.

Usually at the beginning of each test, there was a "run-in" period (not shown in this figure) where the friction coefficient could attain a value as high as 0.20. This "run-in" lasted only a few minutes, then the friction coefficient decreased to some minimum value. This was followed by a slight increase in friction, and then a period where the friction fluctuated around an equilibrium position. During the remainder of the test there was a gradual increase in the friction coefficient until the arbitrary cutoff value of 0.30 was reached.

In this study, the criterion for failure was a friction coefficient of 0.30. This value was less than the friction coefficient of unlubricated 440C stainless steel over the entire test temperature range.

The friction coefficient of the PI-bonded $(CF_{1.1})_n$ film was characterized by a more gradual increase with time than was the PI-bonded MoS_2 film. The friction coefficient of the PI-bonded MoS_2 film at some point during the experiment suddenly became erratic (e.g., at 600 minutes in Fig. 5). This erratic friction is probably due to metallic contact through the lubricant film.

The wear lives of the PI-bonded films were also compared to burnished (rubbed-on) films of MoS_2 or $(CF_{1.1})_n$. Figure 4 gives the wear life as a function of temperature for each of these four films. At each test temperature, the wear life of the PI-bonded $(CF_{1.1})_n$ film was superior to any of the other three films. The wear life of burnished $(CF_{1.1})_n$ was at least four times the wear life of burnished MoS_2 over the test temperature range of 25 to 400 C; and the wear life of PI-bonded $(CF_{1.1})_n$ was up to ten times longer than for burnished films of $(CF_{1.1})_n$ over the same temperature range. The wear life of PI-bonded MoS_2 was longer than that of burnished $(CF_{1.1})_n$ at the lower temperatures; but above 200 C, the burnished $(CF_{1.1})_n$ films were better.

The decomposition temperature of polyimide in dry air is 400 C. When experiments were conducted above this temperature, it was found that the coatings became powdery. It is also interesting to note, that above 400 C, the wear lives of the

PI-bonded films approached the values of the wear lives of their respective burnished solid lubricant films.

Polyimide Thin Films

A most interesting observation in this study was the existence of a transition in the friction and wear life properties of polyimide (PI) thin films. Above some minimum temperature, a PI thin film performed extremely well as a solid lubricant. That is, no solid lubricant additive was needed to obtain low friction and long wear life. Below this temperature, friction was high and wear life short.

No attempt has been made in this study to characterize the transition or to postulate the cause of its existance. Friction and wear life tests conducted at 25 and at 100 C, do indicate that it occurs somewhere between these two temperatures.

Representative friction traces from experiments which were conducted at 25 and at 100 C in a dry air atmosphere are shown in Fig. 5. The traces illustrate the difference in friction properties of the film above and below the transition temperature.

A short "run-in" seems to be associated with the tests conducted at 100 C. The friction coefficient is initially 0.22, then drops to a minimum value of 0.03 after about one minute of sliding. At 25 C, no "run-in" was observed. The friction coefficient started at a high value (0.29) and never decreased significantly for the duration of the experiment.

The effect of atmosphere on the wear life and friction coefficient of PI thin films is shown in Fig. 6. The data are from experiments conducted in atmospheres of dry argon (moisture content, 10 ppm), dry air (moisture content, 20 ppm), and moist air (moisture content, 10 000 ppm).

The results indicate that at 25 C, moisture is beneficial in providing a longer wear life and a lower friction coefficient. However, at 100 C and at 150 C longer wear lives were obtained in a dry air atmosphere. At 200 C and above, essentially no difference was found in wear life or friction coefficient in the two atmospheres.

The results at 25 C in dry argon are nearly equivalent to those in dry air. Above the transition temperature, however, the nonoxidizing atmosphere of dry argon gave better results. The wear life was from two to ten times longer and the friction coefficient was lower. Also, the friction coefficient remained constant at 0.02 as the temperature was increased. In dry air, the friction coefficient tended to increase with increasing temperature.

Another observed effect of the nonoxidizing atmosphere of argon was the extension of the upper temperature limit of the film to 500 C, compared to 400 C in dry air. Above 500 C in dry argon, the coating tended to flake off the metal substrate.

Effect of Solid Lubricant Additives

A comparison of the results obtained when using PI alone to those obtained from equivalent experiments with PI-bonded MoS_2 or PI-bonded $(CF_{1.1})_n$ is given in Fig. 7. Adding either MoS_2 or $(CF_{1.1})_n$ greatly increased wear life at 25 C. The wear life of PI-bonded MoS_2 was 1400 kilocycles while that of PI-bonded $(CF_{1.1})_n$ was 2950 kilocycles. This compared to an 8-kilocycle life for the PI film with no solid lubricant additives.

Above the transition temperature, however, the addition of a solid lubricant was not as beneficial. The wear life of the PI thin film at 100 C was nearly equivalent to that of the PI-bonded MoS_2 film, i.e., 480 kilocycles compared to 550 kilocycles. At 150 C, the effect of adding MoS_2 to the polyimide became detrimental. The wear life of PI alone was 1400 kilocycles, while that of the PI-bonded MoS_2 was about 300 kilocycles. This detrimental effect prevailed at all higher temperatures.

The fact that 75 wt percent MoS_2 added to 25 wt percent PI gave decreased life above 100 C indicates that this might not be the correct lubricant to binder ratio for these two materials above the transition temperature of the polyimide. An optimization study should be made to determine the most beneficial lubricant-binder ratio at temperatures above the friction transition temperature.

At each test temperature, the wear life of PI-bonded $(CF_{1.1})_n$ exceeded that of the PI used alone. An optimization study could conceivably result in an even better formulation for this lubricant-binder system.

The friction coefficient as a function of temperature for the three polyimide films is also plotted in Fig. 7. In addition, the friction coefficients for unlubricated 440C stainless steel sliding against itself are given.

The figure shows that at 25 C (a temperature below the transition), the addition of a solid lubricant to the PI film formulation greatly reduced the friction coefficient. However, at 100 C (a temperature above the transition), no solid lubricant was required to give low friction. The PI-bonded MoS_2 film at 450 C failed immediately, this was due to the degradation of the MoS_2.

Comparison of Wear Lives

A comparison of the wear lives obtained from experiments performed in a dry air atmosphere at temperatures of 25, 100, 200, 300, 400, and 500 C is given in Table I. Results are given for each of the three PI films and for each of the two burnished films. Also shown is the wear life (at 25 C only) for a commercially available sodium silicate-bonded MoS_2 lubricant, which was tested under equivalent conditions.

Both PI-bonded films gave longer wear lives at 25 C than did the sodium silicate-bonded MoS_2 film. The wear life of PI-bonded $(CF_{1.1})_n$ was about six times that of the sodium silicate-bonded MoS_2 film.

At elevated temperatures the two films containing $(CF_{1.1})_n$ gave superior wear lives. This is probably due to the good stability of $(CF_{1.1})_n$ at these higher temperatures.

Rider Wear

In addition to providing a low friction coefficient and a long wear life, a good solid lubricant film should also minimize wear. To compare the wear of the sliding riders, a series of one hour tests (60 kilocycles of sliding) were conducted at 25 C in a dry air atmosphere.

Figure 8 presents the results of these experiments. Five solid lubricant films were evaluated: burnished $(CF_{1.1})_n$, burnished MoS_2, PI-bonded $(CF_{1.1})_n$, PI-bonded MoS_2, or PI films with no solid lubricant additive. The base metal 440C stainless steel, was also tested in the unlubricated condition.

The best results were obtained with the PI-bonded solid lubricant films. The rider wear on PI-bonded $(CF_{1.1})_n$ and PI-bonded MoS_2 were nearly equivalent. The rider wear on the burnished films or on the PI film without solid lubricant additives was about ten times greater than rider wear on the PI-bonded solid lubricant films. Rider wear on unlubricated 440C stainless steel was 10 000 times greater than rider wear on PI-bonded solid lubricant films.

SUMMARY OF RESULTS

Friction and wear life experiments conducted on thin films of polyimide (PI) resin (with no solid lubricant additives) and thin films of PI-bonded graphite fluoride $(CF_{1.1})_n$ indicate that:

1. Polyimide is an excellent binder material for the solid lubricant $(CF_{1.1})_n$.

2. A transition in the friction and wear life characteristics of PI thin films exists (at a so far undetermined temperature between 25 and 100 C). Above this transition, polyimide thin films performed very well as a solid lubricant.

3. The wear life of PI-bonded $(CF_{1.1})_n$ is superior to the wear life of similar films of PI-bonded MoS_2 or to PI films containing no solid lubricant additives.

4. Above 100 C in a dry air atmosphere, longer wear lives were obtained with polyimide thin films than with a film containing 75 wt percent MoS_2 and 25 wt percent PI.

5. Rider wear at 25 C is nearly equivalent for riders sliding on PI-bonded $(CF_{1.1})_n$ and PI-bonded MoS_2 films.

6. The maximum useful temperature for polyimide in sliding studies in dry air is 400 C. At higher temperatures degradation occurs.

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TABLE I. - COMPARISON OF WEAR LIVES AT 25°, 100°, 200°, 300°, 400°, and 500° C FOR SIX SOLID LUBRICANT FILMS

Failure criterion, friction coefficient of 0.30; 440C stainless steel riders and disks; dry air atm (moisture content, 20 ppm); speed, 2.6 m/sec; load, 1 kilogram; rotational speed, 1000 rpm.

Lubricant film	Wear life (kilocycles)					
	Temperature, °C					
	25	100	200	300	400	500
PI-bonded $(CF_{1.1})_n$	2950	2750	1800	450	320	20
PI-bonded MoS_2	1400	550	100	30	4	(a)
PI film	8	480	925	250	67	5
Sodium silicate-bonded MoS_2[b]	500	---	---	---	---	---
Burnished $(CF_{1.1})_n$	230	180	100	80	40	16
Burnished MoS_2	60	31	16	8	5	(a)

[a]Failed immediately.

[b]Commercially available lubricant (contents 71% MoS_2, 7% graphite, 22% sodium silicate).

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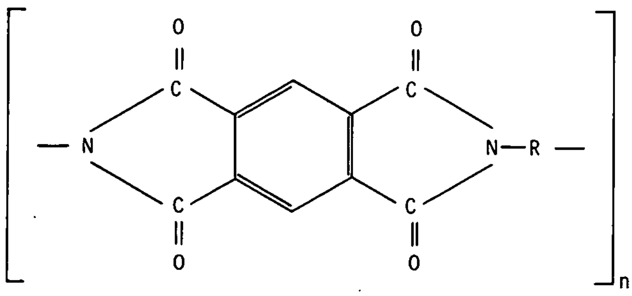

Figure 1. - Basic structure of aromatic polyimide.

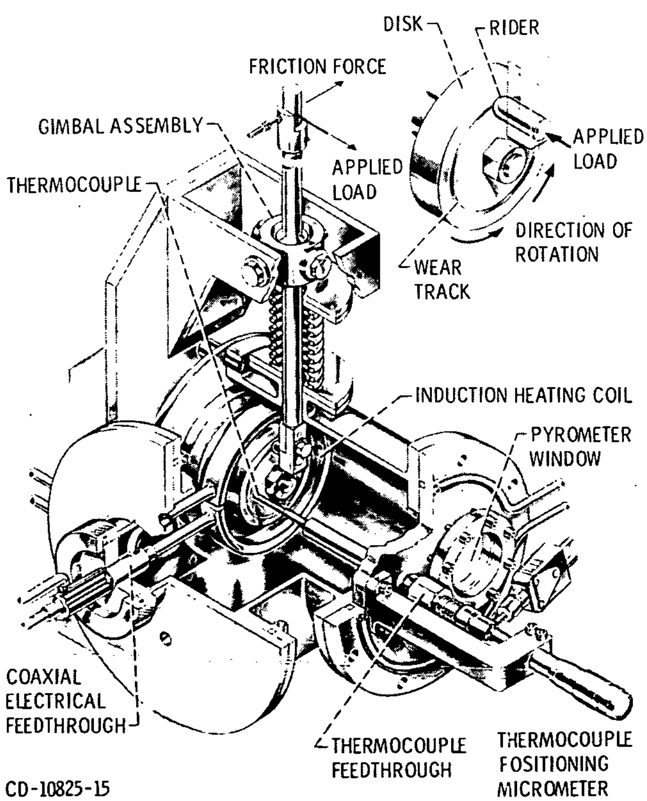

Figure 2. - Friction and wear testing device.

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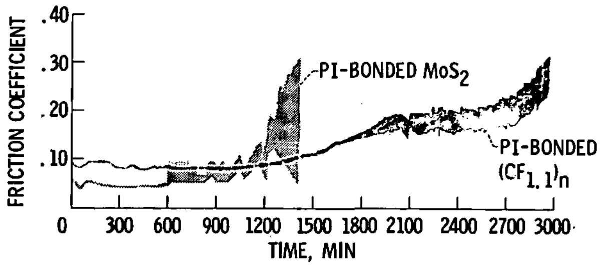

Figure 3. - Variation of friction coefficient with time for polyimide (PI)-bonded films of graphite fluoride ($(CF_{1.1})_n$) and molybdenum disulfide (MoS_2). Test temperature, 25° C; rider and disk material, 440C stainless steel; load, 1 kilogram; linear sliding speed, 2.6 meters per second; rotational speed, 1000 rpm; dry-air atmosphere (moisture content, 20 ppm).

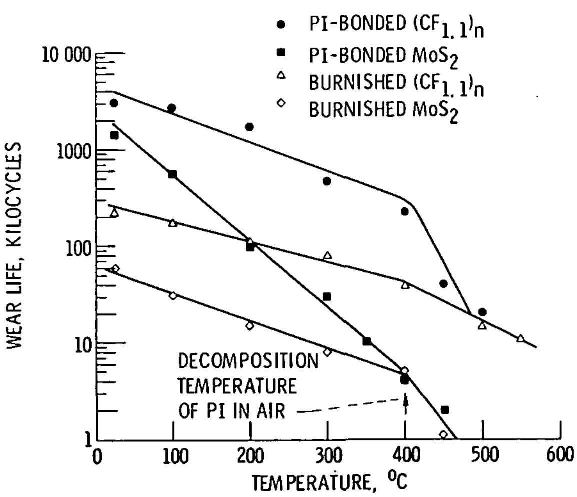

Figure 4. - Wear life as a function of temperature for 440C stainless steel disks lubricated with burnished films or polyimide-bonded films of graphite fluoride $(CF_{1.1})_n$ or molybdenum disulfide (MoS_2). Riders, 440C stainless steel; linear sliding speed, 2.6 meters per second; load, 1 kilogram; atmosphere, dry air (moisture content, 20 ppm); failure criterion, friction coefficient of 0.30.

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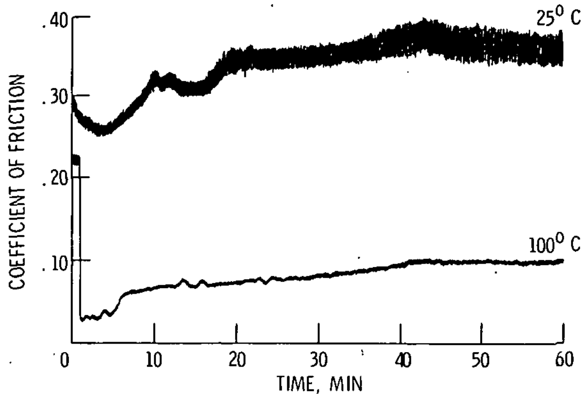

Figure 5. - Effect of temperature on the friction coefficient of thin films of polyimide. Dry air atmosphere (20 ppm, moisture content); linear sliding speed, 2.6 meters per second (1000 rpm); load, 1 kilogram; 440C stainless steel riders and disks.

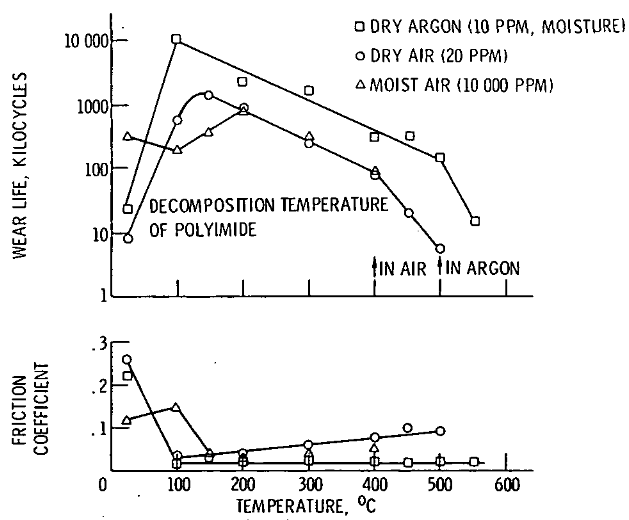

Figure 6. - Friction coefficient and wear life as a function of temperature for thin films of polyimide (PI) run in atmospheres of dry argon, dry air, and moist air. Load, 1 kilogram; linear sliding speed, 2.6 meters per second (1000 rpm); 440C stainless steel riders and disks; failure criterion, friction coefficient of 0.30.

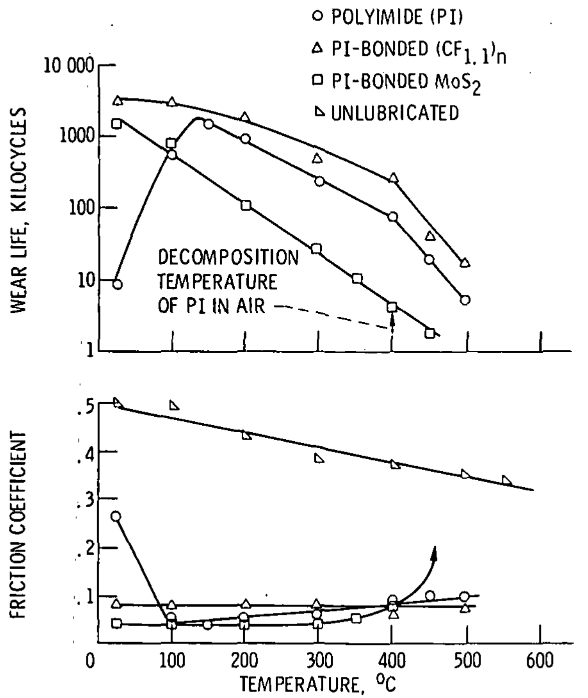

Figure 7. - Friction coefficient and wear life as a function of temperature for three solid lubricant films run in dry air (moisture content, 20 ppm). Linear sliding speed, 2.6 meters per second (1000 rpm); load, 1 kilogram; 440C stainless steel riders and disks; failure criterion, friction coefficient of 0.30.

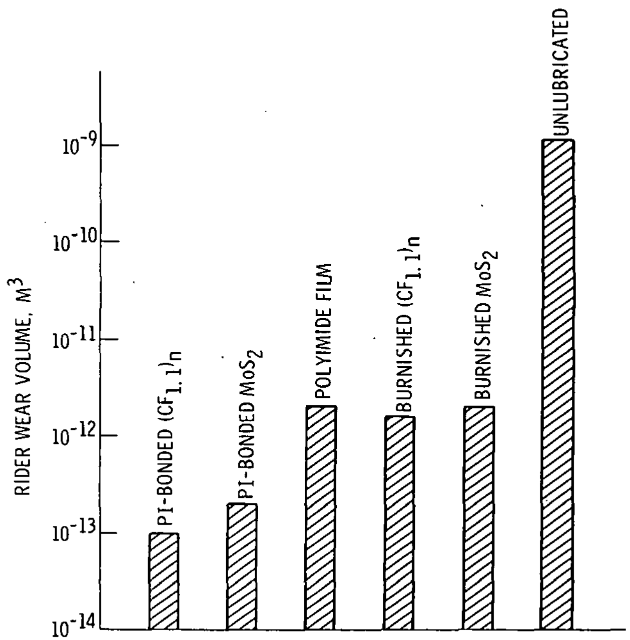

Figure 8. - Comparison of wear to 440C stainless steel riders which slid for one hour against 440C stainless steel disks coated with the above solid lubricant films. Temperature, 25° C; load, 1 kilogram; sliding speed, 2.6 meters per second (1000 rpm); atmosphere, dry air (moisture content, 20 ppm).

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