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FRICTION AND WEAR LIFE PROPERTIES OF POLYIMIDE THIN FILMS

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16. Abstract

A transition in the friction coefficient and wear life properties of Pyralin polyimide (PI) thin films was found to exist at a temperature between 25° and 100° C. Above this transition, PI thin films gave long wear lives and low friction coefficients. The presence of H_2O in air improved the friction and wear life properties at 25° C; but at 100° C, H_2O had a detrimental effect. At 100° C and above, a dry argon atmosphere gave lower friction coefficients and longer wear lives than did a dry air atmosphere.

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FRICTION AND WEAR LIFE PROPERTIES OF POLYIMIDE THIN FILMS

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SUMMARY

A hemisphere-on-flat disk-type friction apparatus was used to evaluate the friction coefficient and the wear life of Pyralin polyimide (PI) thin films which were bonded to roughened 440C stainless steel disks. The films were evaluated in atmospheres of dry argon (10 ppm H_2O), dry air (20 ppm H_2O), or moist air (10 000 ppm H_2O).

It was found that a transition in the friction and wear life properties of PI thin films exists at a temperature between 25° and 100° C. Above this transition, the PI thin films performed very well as a solid lubricant. For example, at 25° C in a dry air atmosphere, the minimum value for the friction coefficient was 0.26, while at 100° C it was 0.04. At 100° C or above, even better results were achieved in a dry argon atmosphere. Both longer wear lives and lower friction coefficients were obtained.

It was found that moisture also had an effect on the lubricating properties of PI thin films. At 25° C, moisture in air had a beneficial effect, while at 100° C it had a detrimental effect. At 200° C, moist air and dry air gave equivalent results.

The effect of varying speed or load on the average friction coefficient at a temperature above the transition (100° C) was also studied. In dry air, increasing speed or load decreased the friction coefficient to a value approaching that obtained in dry argon. No effect of increasing speed or load was found in dry argon, the friction remained constant at the low value of 0.02.

The wear lives of PI thin films were compared to the results reported in reference 1, where PI was used as a binder for graphite fluoride $(CF_{1.1})_n$ or molybdenum disulfide (MoS_2). Above 100° C in dry air, better wear lives were obtained using PI alone rather than using it as a binder for MoS_2 (film composition: 75 wt. % MoS_2, 25 wt. % PI). The PI-bonded $(CF_{1.1})_n$ (film composition: 60 wt. % $(CF_{1.1})_n$, 40 wt. % PI) gave better wear lives at all temperatures than the PI thin films.

INTRODUCTION

Pyralin polyimide (PI-4701) has been shown to be a very good binder for the solid lubricant, graphite fluoride $(CF_{1.1})_n$ (ref. 1). Thin films of this lubricant on 440C stainless steel gave long wear lives and low friction coefficients in dry air (10 ppm H_2O) at temperatures up to 400° C (the decomposition temperature of PI in air). It has also been shown in several Lewis Research Center reports (refs. 2 to 5) that polyimide, in the form of a solid body, has good friction and wear properties.

There are many different kinds of polyimides (refs. 6 to 11). Basically they are cyclic chain polymers, which are formed by the chemical reaction of pyromellitic dianhydrides and aromatic diamines. The chains consist of aromatic rings alternated with heterocyclic groups. Their basic structure is shown in figure 1, the R in the figure represents a thermally stable group (refs. 6 and 9 to 12).

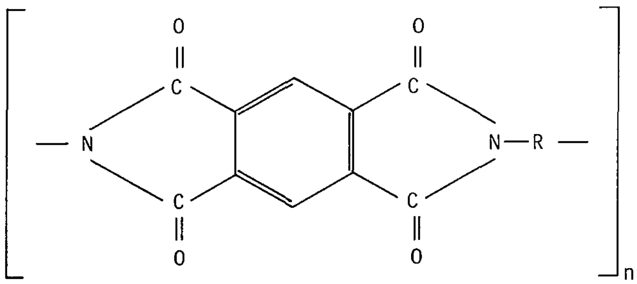


Figure 1. - Basic structure of aromatic polyimide.

Due to the multiple bonds between the aromatic and heterocyclic groups, the polyimides are characterized by a high thermal stability (400° C in air, 500° C in inert atmospheres) (refs. 1 and 13). They also have a high radiation stability (refs. 9, 10, and 13) and can withstand high exposure to neutrons, electrons, ultraviolet light, and gamma radiation. They are resistant to most common chemicals and solvents; but if attacked by alkalis (refs. 10 and 13) at the decomposition point, they crumble to a fine powder without melting. For a more detailed discussion of the physical properties, see references 9 to 14.

The object of this study was to determine the lubricating properties of thin Pyralin polyimide (PI) films which were bonded to roughened 440C stainless steel disks. The scope of the program included determining the effects of temperature, load, sliding speed, and type of atmosphere on the friction and wear life of the polyimide thin films.

FRICTION APPARATUS

A hemisphere-on-flat type of sliding friction apparatus was used to study the friction and wear life characteristics of the polyimide thin films. A diagram of the apparatus is shown in figure 2. The test specimens were made of 440C stainless steel.

The riders were hemispherically tipped pins with a radius of 0.475 centimeter. They were loaded with dead weights against a flat 6.3-centimeter-diameter disk, which could be rotated at various speeds. The rider slid on a 5-centimeter-diameter circular track on the disk.

The disk was heated by using a high frequency induction unit. The temperature was monitored by a thermocouple when the disk was not rotating and by an infrared pyrometer

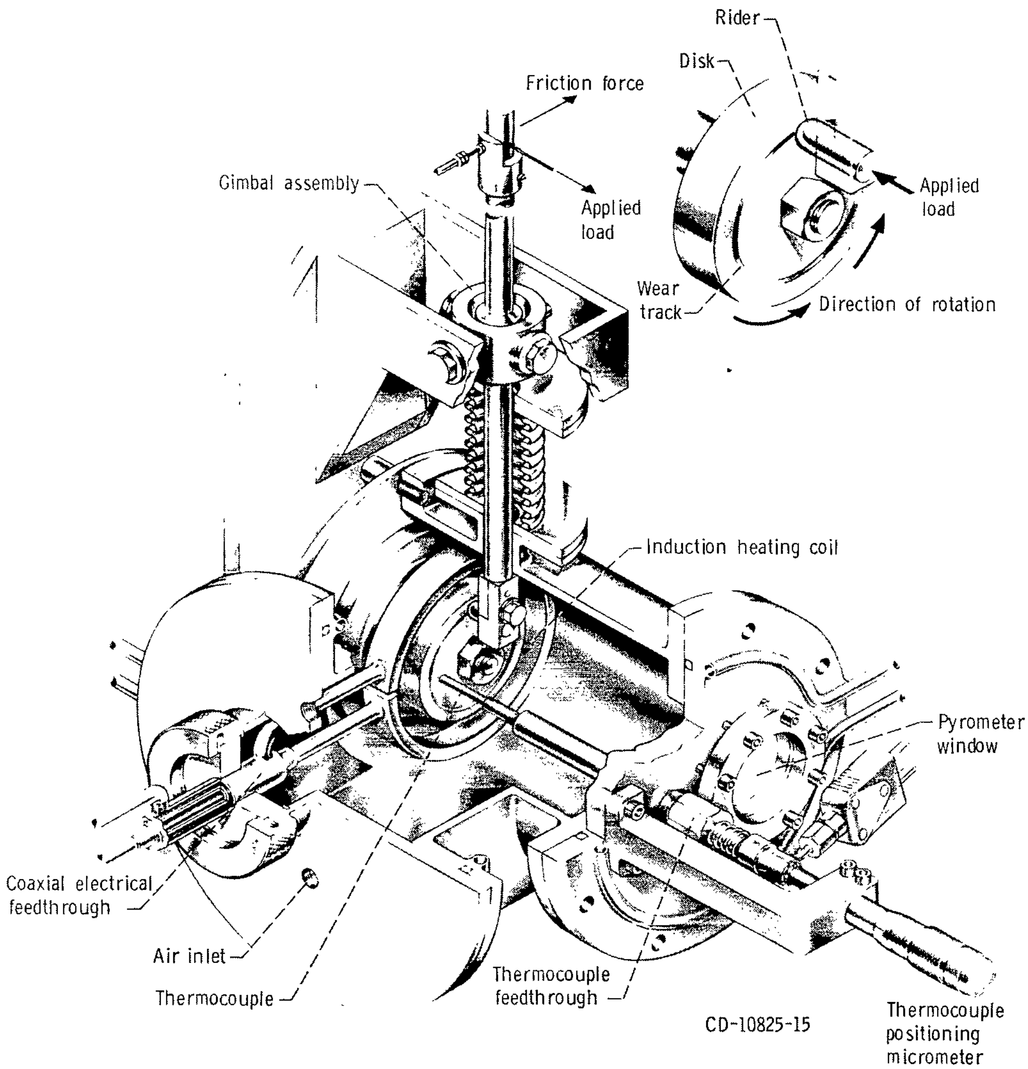


Figure 2. - Friction and wear testing device.

when in motion. A strain gage was used to measure frictional force, which was continuously recorded by a strip chart recorder.

PROCEDURE

Specimen Preparation

The 440C stainless steel specimens used in this investigation were hardened to Rockwell C-60. The surfaces of the disks were roughened to a rms of 0.90×10^{-6} to 1.25×10^{-6} meter (35×10^{-6} to 50×10^{-6} in.) by sandblasting.

After the surfaces were roughened, they were scrubbed with a brush under running water to remove any abrasive particles. A water paste of levigated alumina was next used to clean the surfaces. The disks were then rinsed in distilled water and stored in a desiccator until they were coated with the solid lubricant.

The riders were cleaned by washing them with ethyl alcohol and then scrubbing them with a water paste of levigated alumina. The riders were then rinsed in distilled water and also stored in a desiccator until used. Lubricant was not applied to the riders.

Film Application

An airbrush was used to apply the polyimide varnish to the disks. In order to obtain a sprayable solution, a thinner consisting of N-methypyrrolidone and xylene was added to the polyimide varnish.

The polyimide film did not dry rapidly. If too thick a coating were applied, the varnish "ran" and a nonuniform film resulted. To obtain uniform films and the desired coating thicknesses, it was necessary to apply the polyimide varnish in thin layers, bake the film at 100° C for 1 hour, and then to apply another thin layer, and repeat the procedure.

When the desired thickness of 10 to 20 micrometers was obtained, (usually three or four coats were applied) the remainder of the curing procedure was carried out. The procedure was to bake the coating at 100° C for 1 hour, and then to bake it for an additional hour at 300° C. The coated disks were stored in a desiccator until used.

The PI-bonded solid lubricant films (from ref. 1) were applied and cured in a similar manner. The PI-bonded MoS_2 films were prepared by mixing 75 weight percent MoS_2 powder with 25 weight percent PI solids. The PI-bonded $(CF_{1.1})_n$ films were prepared by mixing 60 weight percent $(CF_{1.1})_n$ powder with 40 weight percent PI solids. N-methypyrrolidone and xylene were added to each formulation to obtain a sprayable solution.

Experimental Method

The method used for conducting the friction and wear life tests was as follows: A rider and disk (with the applied PI film) were inserted into the friction apparatus (fig. 2). The test chamber was sealed and either dry argon (10 ppm H_2O), 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. 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 using induction heating. The temperature was held for 10 minutes to allow it to stabilize.

Some preliminary experiments were conducted to determine the friction characteristics of unlubricated 440C stainless steel disks. From these results, it was decided to make the criterion for failure 0.30. This value of the friction coefficient was less than the friction coefficient of unlubricated 440C stainless steel over the experimental temperature range of 25° to 500° C. An automatic cutoff system was used to shut down the apparatus when the friction coefficient reached 0.30.

A series of experiments were conducted at 100° C in a dry air atmosphere (20 ppm H_2O) and in a dry argon atmosphere (10 ppm H_2O) to determine the effect of load and speed on the coefficient of friction. When the effect of speed was studied, the load was held constant at 1 kilogram. When the effect of load was studied, the speed was held constant at 2.6 meters per second (1000 rpm).

The experimental method used for this series of experiments was to first "run-in" the PI films at a speed of 2.6 meters per second and a load of 1 kilogram for approximately 20 minutes. Then a series of 2-minute tests were performed at each of the following speeds: 1.6, 2.6, 3.9, 5.2, 6.5, 7.8, 9.1, and 10.4 meters per second. Two tests at each speed were made; once when increasing the speed, and once when decreasing the speed.

A similar series of tests were performed at each of the following loads: 0.5, 1, 1.5, 2.0, 2.5, and 3.0 kilograms.

RESULTS AND DISCUSSION

Effect of Temperature

A most interesting observation in this study was the existance of a transition in the friction and wear life properties of 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 was made in this study to exactly characterize the transition or to postulate the cause of its existance. However, friction and wear life tests conducted at 25° and 100° C in a dry air atmosphere do indicate that the transition occurs somewhere between these two temperatures. For example, at 25° C, the minimum friction coefficient of the PI thin films was 0.26 and the wear life was 8 kilocycles. At 100° C, the minimum friction coefficient dropped to a value of 0.04, and wear life increased to 480 kilocycles.

The wear life of PI thin films is shown as a function of temperature in figure 3. For comparison, the results of reference 1 are also given. In reference 1, PI was used as a binder for molybdenum disulfide (MoS_2) (75 wt. % MoS_2, 25 wt. % PI) and for graphite fluoride $(CF_{1.1})_n$ (60 wt. % $(CF_{1.1})_n$, 40 wt. % PI).

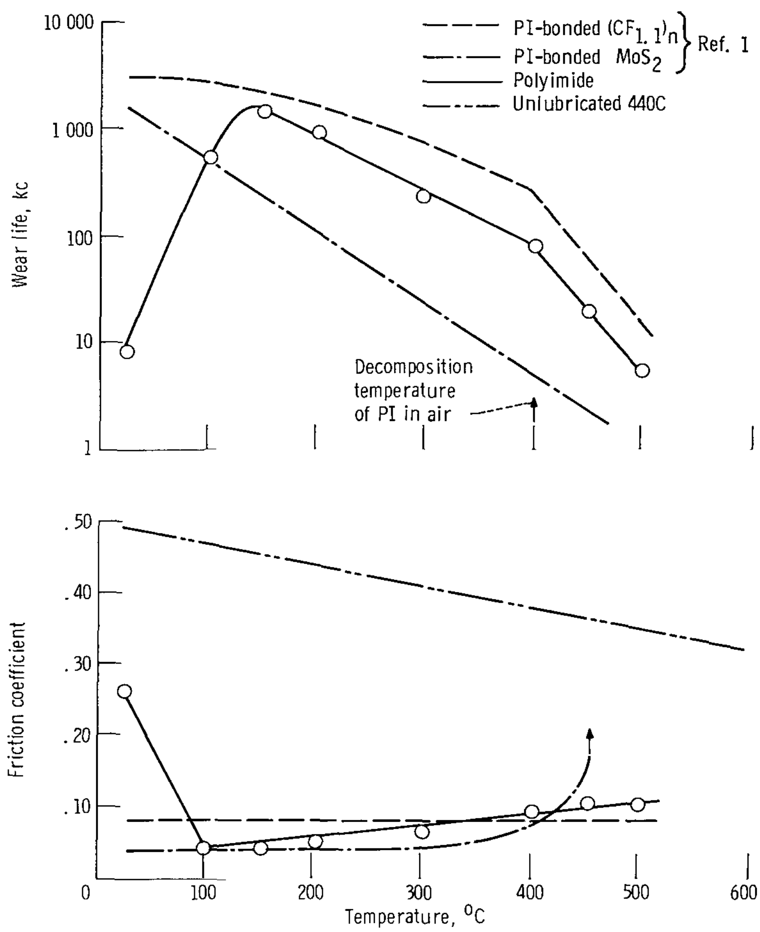


Figure 3. - Minimum friction coefficient and wear life as a function of temperature for three solid lubricant films run in dry air (20 ppm H_2O). Linear sliding speed, 2.6 meters per second (1000 rpm); load, 1 kilogram; 440C stainless steel riders and disks; failure criterion, friction coefficient of 0.30.

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 the 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, that is, 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 weight percent MoS_2 added to 25 weight percent PI gave decreased life above 100° C indicates that this might not be the correct lubricant-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 transition temperature.

TABLE I. - ENDURANCE PROPERTIES OF PYRALIN POLYIMIDE (PI) THIN FILMS, PI-BONDED MoS_2 THIN FILMS, OR PI-BONDED $(CF_{1.1})_n$ THIN FILMS

[Dry air atmosphere (20 ppm H_2O); linear sliding speed, 2.6 m/sec (1000 rpm); load, 1 kg; 440C stainless steel riders and disks.]

Friction coefficient	PI	PI-bonded MoS_2	PI-bonded $(CF_{1.1})_n$	Friction coefficient	PI	PI-bonded MoS_2	PI-bonded $(CF_{1.1})_n$
	Number of revolutions, kc				Number of revolutions, kc		
25° C				300° C			
0.10	<1	1000	1100	0.10	10	20	350
.20	<1	1250	1750	.20	210	25	400
.30	8	1400	2950	.30	250	30	450
100° C				400° C			
0.10	40	450	2300	0.10	9	<1	100
.20	350	500	2550	.20	60	2	150
.30	480	550	2750	.30	67	4	230
200° C				500° C			
0.10	10	60	1000	0.10	<1	<1	<1
.20	700	70	1350	.20	4	<1	10
.30	925	100	1800	.30	5	<1	20

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

The minimum friction coefficient as a function of temperature for the three polyimide films is also plotted in figure 3. 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 resulted from the oxidative degradation of the MoS_2.

Table I compares the wear lines of three lubricant films at 25°, 100°, 200°, 300°, 400°, and 500° C. Also given in the table are the number of kilocycles elapsed before the friction coefficient reached values of 0.10 and 0.20. The table thus gives an indication of how the friction coefficient varied during the life of the film.

Effect of Atmosphere

The effect of atmosphere on the minimum friction coefficient and the wear life of PI thin films is shown in figure 4. In this figure, the minimum friction coefficient and the wear life are plotted as a function of temperature for experiments conducted in dry argon (10 ppm H_2O), dry air (20 ppm H_2O), and moist air (10 000 ppm H_2O).

The results indicate, that in air at 25° C, moisture is beneficial in providing a longer wear life and a lower friction coefficient; however, at 100° C moisture seemed to have a detrimental effect. For example, at 25° C in moist air, the wear life was 300 kilocycles and the minimum friction coefficient was 0.12. Under the same conditions in dry air, the wear life was only 8 kilocycles and the minimum friction coefficient was 0.26. At 100° C in moist air, the wear life decreased to 210 kilocycles and the friction coefficient rose to 0.15. At the same temperature in dry air, wear life increased to 480 kilocycles and the friction coefficient dropped to 0.04. As the temperature was increased above 100° C, the moisture effect became less important, and had no significant effect at 200° C and above. Further work is needed to determine the mechanism by which moisture affects the lubricating properties of PI thin films; and what, if any, effect it has on the transition temperature.

The results obtained, at 25° C, in a dry argon atmosphere are nearly equivalent to those obtained in dry air at this temperature. At 100° C (a temperature above the transition) or at higher temperatures, the nonoxidizing atmosphere of dry argon gave much better results. The wear life in dry argon was up to ten times longer than in dry air. The

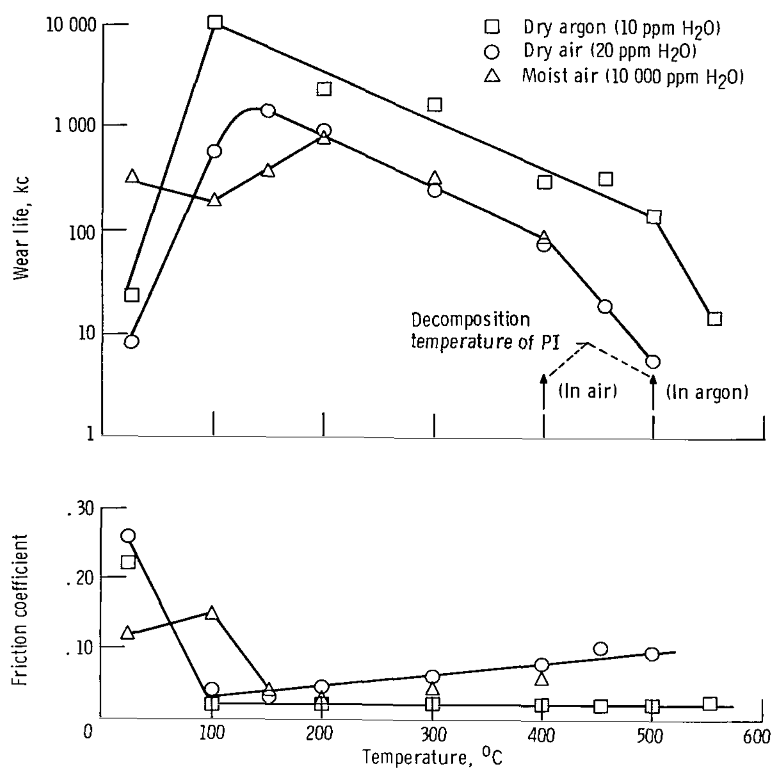


Figure 4. - Minimum friction coefficient and wear life as a function of temperature for thin films of polyimide (PI) run in atmospheres of dry argon, dry air, and moist air. Linear sliding speed, 2.6 meters per second (1000 rpm); load, 1 kilogram; 440C stainless steel riders and disks; failure criterion, friction coefficient of 0.30.

minimum friction coefficient in dry argon was constant at 0.02 from 100° to 550° C, by comparison in dry air the friction coefficient increased linearly from 0.04 to 0.09 as the temperature was raised from 100° to 500° C.

The upper temperature limit for PI thin films in argon was 500° C as compared to 400° C in air. The coatings became powdery at the upper temperature limits stated, but even in this powdery state, they did provide a certain degree of lubrication in both dry air and dry argon.

A comparison of the wear lives of PI thin films is given in table II. Results obtained from the experiments performed in the three different atmospheres and at temperatures of 25°, 100°, 200°, 300°, 400°, and 500° C are given. Also shown in the table are the number of kilocycles elapsed before the friction coefficient reached values of 0.10 and 0.20. As with table I, this table gives an indication of how the friction coefficient varied during the life of the films.

Representative plots of friction coefficients as a function of time at 25° and 100° C are given in figure 5. The traces shown are reproductions of actual friction traces, and are given for tests in dry argon, dry air, and moist air.

TABLE II. - EFFECT OF ATMOSPHERE ON THE ENDURANCE PROPERTIES OF PYRALIN POLYIMIDE (PI) THIN FILMS

[Linear sliding speed, 2.6 m/sec (1000 rpm); load, 1 kg; 440C stainless steel riders and disks.]

Friction coefficient	Dry argon (10 ppm H_2O)	Dry air (20 ppm H_2O)	Moist air (10 000 ppm H_2O)	Friction coefficient	Dry argon (10 ppm H_2O)	Dry air (20 ppm H_2O)	Moist air (10 000 ppm H_2O)
	Number of revolutions, kc				Number of revolutions, kc		
	25° C				300° C		
0.10	<1	<1	<1	0.10	190	10	30
.20	<1	<1	90	.20	950	210	230
.30	28	8	300	.30	1200	250	310
	100° C				400° C		
0.10	10 000	40	<1	0.10	261	9	2
.20	(a)	350	18	.20	261	60	31
.30	(a)	480	210	.30	261	67	67
	200° C				500° C		
0.10	650	10	250	0.10	148	<1	---
.20	1 670	700	700	.20	148	4	---
.30	1 820	925	820	.30	148	5	---

[a]Test terminated before friction coefficient reaches this value.

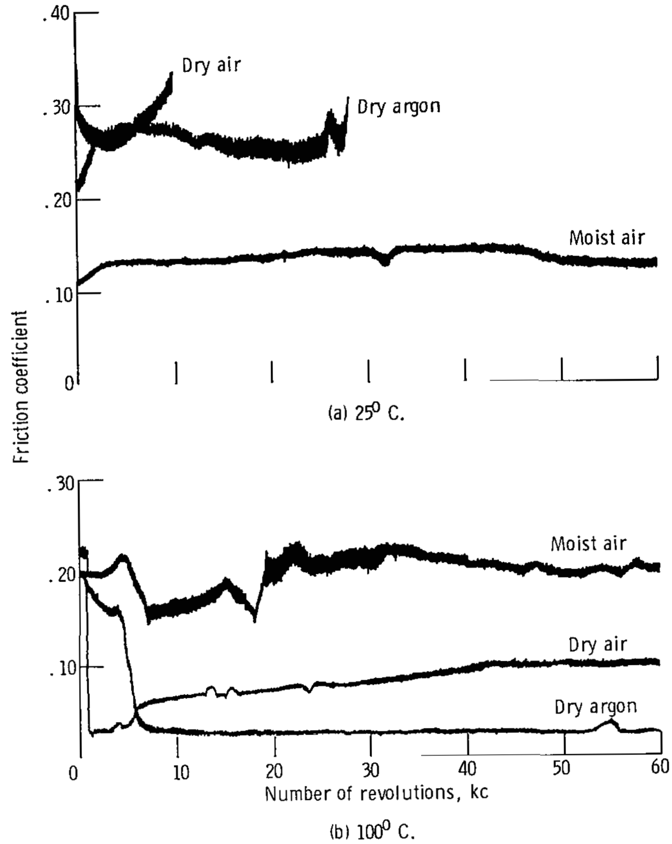


Figure 5. - Typical friction traces for polyimide thin films tested at 25° and 100° C in atmospheres of dry argon (10 ppm H_2O), dry air (20 ppm H_2O), and moist air (10 000 H_2O). Linear sliding speed, 2.6 meters per second (1000 rpm); load, 1 kilogram; 440C stainless steel riders and disks.

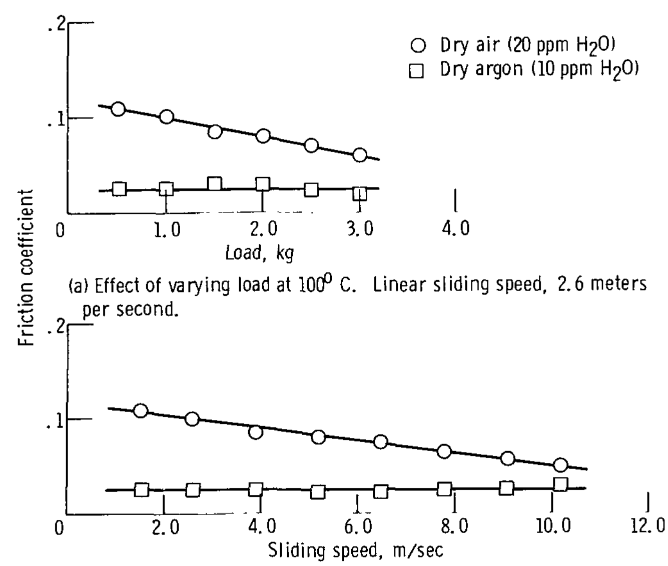
The traces illustrate that, at 25° C, the presence of moisture tends to make the friction trace somewhat "smoother" and also gives it a lower value. At 100° C, moisture is shown to be detrimental. The friction is higher and also "more erratic" when moisture is present.

A short "run-in" seems to be associated with tests conducted in the dry atmospheres at 100° C. Initially, the friction coefficient starts out high at 0.20 or above and then drops off to a value of 0.03 to 0.04 after 1 minute in dry air, and after 7 minutes in dry argon. There was no "run-in" for the tests conducted in moist air at 100° C or for any of the tests conducted at 25° C.

Effect of Load and Sliding Speed

A series of experiments were conducted in dry air and in dry argon at 100° C to determine the effect of load and sliding speed on the friction coefficient of PI thin films. In these experiments, when the load was varied the linear speed was kept constant at 2.6 meters per second; and when the linear speed was varied, the load was held constant at 1 kilogram. Figure 6 presents the results of these experiments. It should be noted that in this figure the values of the friction coefficient for tests run in dry air are the average values. As can be seen in figure 5, the friction coefficient in dry air drops to a minimum value after "running-in." It then rises gradually for a certain number of kilocycles and then fluctuates around an average position. This average value is the value presented in this figure.

In a dry argon atmosphere, no effect on the average friction coefficient was found when load or speed were varied. The value remained somewhere between 0.02 to 0.03 over the entire test range. In dry air, however, the effect of increasing load or linear sliding speed was to decrease the average friction coefficient. The friction coefficient at 1.5 meters per second was 0.11. As the speed was increased, the friction coefficient decreased in a linear manner until, at 10.2 meters per second, the friction coefficient became 0.05. (The 1.5- and 10.2-m/sec speeds were the lower and upper limits of the test apparatus.) Similarly, when the load was increased, the friction coefficient decreased in



(a) Effect of varying load at 100° C. Linear sliding speed, 2.6 meters per second.

(b) Effect of varying sliding speed at 100° C. Load, 1 kilogram.

Figure 6. - Average friction coefficient as a function of load and sliding speed for polyimide thin films run in dry air or dry argon atmospheres using 440C stainless steel test specimens. (Note that the values of the friction coefficient presented in this figure are average values, not minimum values as presented in the other figures.)

a linear manner. At a load of 0.5 kilogram the friction coefficient was 0.11; when the load was increased to 3.0 kilograms, the friction coefficient was 0.06.

SUMMARY OF RESULTS

Friction and wear life experiments conducted on thin films of Pyralin polyimide (PI-4701) indicate the following:

1. A transition exists (at a so far undetermined temperature between 25° and 100° C), above which polyimide (PI) thin films performed very well as a solid lubricant.
2. When moisture was present in air (10 000 ppm H_2O), improved friction and wear life results were obtained at 25° C; however, at 100° C moisture was detrimental. At 200° C and above no moisture effect was observed.
3. The nonoxidizing atmosphere of dry argon gave lower friction coefficients and longer wear lives (at temperatures above the transition) than did a dry air atmosphere.
4. PI-bonded $(CF_{1.1})_n$ in dry air gave better wear lives at all test temperatures than did PI thin films.
5. Above 100° C, better wear lives were achieved in dry air with PI thin films than with PI-bonded MoS_2 thin films.
6. At 100° C in dry air, the effect of increasing speed or load was to decrease the average friction coefficient to a value approaching that obtained in dry argon; in dry argon, no effect was found when load or speed were varied.

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National Aeronautics and Space Administration,
Cleveland, Ohio, June 8, 1972,
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