GRAPHITE FLUORIDE
AS A SOLID LUBRICANT
IN A POLYIMIDE BINDER

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Polyimide resin (PI) was shown to be a suitable binder material for the solid lubricant graphite fluoride \((\text{CF}_1.1)_n\). Comparisons were made to similar tests using PI-bonded MoS\(_2\) films, \((\text{CF}_1.1)_n\) rubbed films, and MoS\(_2\) rubbed films. The results showed that, at any one specific temperature between 25\(^\circ\) and 400\(^\circ\) C, the wear life of PI-bonded \((\text{CF}_1.1)_n\) films exceeded those of the other three films by at least a factor of 2 and by as much as a factor of 60. Minimum friction coefficients for the PI-bonded films were 0.08 for \((\text{CF}_1.1)_n\) and 0.04 for MoS\(_2\). The rider wear rates for the two PI-bonded films at 25\(^\circ\) C were nearly equal.
A solid lubricant film, consisting of a polyimide resin (PI) as the binder and graphite fluoride (CF$_{1.1}$)$_n$ as the lubricant, was formulated. The lubricant was applied to roughened 440C stainless-steel disks (0.90x10$^{-6}$ to 1.25x10$^{-6}$ m, rms) using an artist’s airbrush. The thicknesses of the films were in the range of 10 to 20 micrometers. A pin-on-disk friction apparatus was used to evaluate the friction coefficient, the wear life, and the rider wear rate. The test conditions were a dry-air atmosphere (moisture content, 20 ppm), 440C stainless-steel riders, a 1-kilogram load, and a 2.6-meter-per-second (1000-rpm) sliding speed.

For comparison, similar experiments were conducted using PI-bonded molybdenum disulfide (MoS$_2$) films, (CF$_{1.1}$)$_n$ rubbed films, and MoS$_2$ rubbed films. The results indicated that PI is a suitable binder material for the solid lubricant (CF$_{1.1}$)$_n$. Comparison of the wear lives at any one specific temperature between 25$^\circ$C and 400$^\circ$C showed that the PI-bonded (CF$_{1.1}$)$_n$ films were at least two times better than the other three films at 25$^\circ$C and as much as 60 times better than either MoS$_2$ film at 400$^\circ$C.

The minimum friction coefficient for the PI-bonded (CF$_{1.1}$)$_n$ film was 0.08 over the entire test temperature range of 25$^\circ$ to 500$^\circ$C. The minimum friction coefficient for the PI-bonded MoS$_2$ film was 0.04 over the range of 25$^\circ$ to 300$^\circ$C. Above this temperature, the friction coefficient increased gradually until, at 450$^\circ$C, no minimum value was recorded (i.e., the film failed immediately).

The rider wear rates of the two PI-bonded films at 25$^\circ$C were nearly equivalent. These wear rates were, however, only one-tenth the wear rate of the rubbed-on films. The upper temperature limit, in air, for the PI-bonded films was deemed to be 400$^\circ$C, since at this temperature the coatings became powdery.

INTRODUCTION

In a previous report (ref. 1), it has been shown that burnished (rubbed-on) films of graphite fluoride (CF$_{1.1}$)$_n$ have excellent lubricating properties. The experiments
described in reference 1 consisted of rubbing \((\text{CF}_{1.1})_n\) powders onto roughened 440C stainless-steel disks and then evaluating the resulting films with a pin-on-disk friction apparatus (fig. 1). The results indicated that the minimum value of friction coefficient for these \((\text{CF}_{1.1})_n\) lubricated disks was comparable to, or better than, the friction coefficients for disks similarly lubricated with molybdenum disulfide (MoS\(_2\)) or graphite. The wear life of the rubbed \((\text{CF}_{1.1})_n\) films was as much as six times better than that of either of the two other lubricant films.

Solid lubricants, such as MoS\(_2\) or graphite, usually show increased wear life when they are incorporated into a binder system. The object of this investigation was, therefore, to determine whether \((\text{CF}_{1.1})_n\) would also have improved wear life when used with

![Figure 1. - Friction and wear testing device.](image-url)
a binder material. A secondary objective was to compare the results to those obtained when MoS$_2$ was used with the same binder material.

The binder material used in these experiments was a polyimide resin (manufacturer's designation, PI-4701) (ref. 2). Polyimide has been shown in previous Lewis Research Center reports (refs. 3 to 6) to be a good friction and wear material. Also reference 7 has reported good results using it as a binder material for MoS$_2$. 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.

The experimental conditions used in these tests were a 2.6-meter-per-second (1000-rpm) sliding speed, a 1-kilogram load, a dry-air atmosphere (moisture content, 20 ppm), and 440°C stainless-steel riders and disks.

**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, the existence of a patent entitled "Dry Lubrication" (ref. 8) was discovered. The patent was granted on July 25, 1961; it describes a product with a carbon-to-fluorine ratio of 1:1. The patent states, "Fluorinated graphite has outstanding dry lubricating properties." However, no data supporting this statement were included with the patent and quantitative information was not available prior to reference 1.

More recently, Ishikawa and Shimada (ref. 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 (ref. 10) have also conducted some exploratory tests 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**

Most polymers lose their rigidity and strength at elevated temperatures (refs. 11
and 12). One of the reasons for this is that their chain-like structure is weakened by the increased molecular vibrations associated with increased temperatures. There are two possible consequences of these vibrations: Either the bonds between the monomer units break and the polymer disintegrates, or the polymer melts.

To obtain polymers with improved thermal stability, it is necessary to devise a means of stiffening the molecules so that heat and its resulting vibrations will have less effect (ref. 12). A method of accomplishing this is by the insertion of aromatic rings (rings of carbon atoms) into the polymer chains (refs. 13 to 15). These resulting polymers are designated aromatic polymers. Polymers of this type have an upper temperature limit of about 300°C (refs. 12 and 15).

To make polymers which will withstand temperatures above 300°C, the parent molecule itself is made into a ring. These rings (called heterocyclic molecules) are then alternated with the aromatic rings to form cyclic-chain polymers. One class of these polymers has been designated aromatic polyimides (refs. 14 to 20). Their basic structure is shown in figure 2, where R represents a thermally stable group (refs. 14, and 18 to 21). These polymers can be formed chemically by the reaction of pyromellitic dianhydrides and aromatic diamines.

Due to the multiple bonds between the aromatic and heterocyclic rings, the polyimides are characterized by a high thermal stability (400°C in air; 500°C in inert atmospheres) (refs. 19, and 22 to 24). They also have a high radiation stability (refs. 18, 19, and 22) 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 (refs. 19 and 22). At the decomposition point, they crumble to a fine powder without melting. For a more detailed discussion of the physical properties, see references 18 to 24.

Figure 2. - Basic structure of aromatic polyimide. The structure consists of aromatic rings and heterocyclic molecules.
APPARATUS DESCRIPTION

The apparatus used to measure the friction coefficient and to evaluate 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, 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 MoS₂ films were prepared by mixing three parts (by weight) of MoS₂ powder with one part (by weight) of polyimide solids. The PI-bonded (CF₁.₁)ₙ films were made by mixing three parts (by weight) of (CF₁.₁)ₙ powder with two parts (by weight) of polyimide solids. The density of (CF₁.₁)ₙ was only about one-half that of MoS₂; 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 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 clean 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. Cleaning continued until no trace of sediment from the rider appeared on the cloth. The riders were then rinsed in distilled water and stored in the desiccator until used.

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 and solid lubricant was sprayed onto each disk using an artist's airbrush. The coating did not dry rapidly. Thus, if more than a thin coat was applied, the liquid "ran" and a nonuniform coating resulted. To eliminate this "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 in an oven for 1 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 1 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. This procedure was the same as that used for the burnished 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. 1). The test chamber was sealed, and dry air (moisture content, 20 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 criterion for failure in these tests was a friction coefficient of 0.30. An auto-
matic cutoff system shut down the apparatus when the friction coefficient reached this value.

In order to calculate the rider wear rate at 25°C, the tests were stopped after 1 hour of sliding (60 kilocycles). 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

Friction Coefficient

The variation of the friction coefficient with time, at 25°C, in a dry-air atmosphere is shown in figure 3. The figure depicts actual friction traces for both the PI-bonded (CF$_{1.1}$)$_n$ film and the PI-bonded MoS$_2$ film. The test conditions were a load of 1 kilogram, a sliding speed of 2.6 meters per second (1000 rpm), a dry-air atmosphere (moisture content, 20 ppm), and 440C stainless-steel riders and disks.

These friction traces are typical of all the tests conducted. Usually, at the start of each test, there was a run-in period (not shown in the figure) where the friction coefficient could attain a value as high as 0.20. This run-in lasted a few minutes, after which the friction coefficient fell to some minimum value. It then rose slightly and fluctuated around an equilibrium position. The remainder of the test consisted of a gradual in-
crease in the value of the friction coefficient until the cutoff friction coefficient of 0.30 was reached.

The friction coefficient of the PI-bonded (CF$_{1.1}$)$_n$ film seemed to be characterized by a more gradual increase with time than did the PI-bonded MoS$_2$ film. The friction coefficient of the PI-bonded MoS$_2$ film at some point during the test suddenly became erratic (e.g., at 600 min in fig. 3). This erratic friction is probably due to metallic contact through the lubricant film, and the subsequent rehealing of the film due to additional lubricant being brought into the contact region from the sides of the disk wear track.

The friction coefficients of PI-bonded (CF$_{1.1}$)$_n$ and PI-bonded MoS$_2$ as a function of temperature are presented in figure 4. The values of the friction coefficient in this figure and all following figures are the minimum values obtained for each test. Also shown in figure 4 are friction coefficient values for unlubricated 440C stainless steel sliding against itself. This figure illustrates that the friction coefficient of PI-bonded (CF$_{1.1}$)$_n$ remained constant at 0.08 over the entire test temperature range of 25°C to 500°C. The friction coefficient of PI-bonded MoS$_2$ remained constant at 0.04 over the range of 25°C to 300°C. The friction coefficient increased to 0.05 at 350°C and to 0.08 at 400°C. At 450°C, the friction coefficient rose steadily, and no minimum value was recorded.

Coating Wear Life

One of the most important properties that a good solid lubricant must possess is
the ability to remain intact on the surface to be lubricated for as long as possible. This quality, the wear life of the lubricant film, can be designated either as the time or the number of cycles until failure occurs.

Failure is an arbitrary term. It is usually the discretion of the experimentor which determines the onset of failure. 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 wear lives of four solid lubricant films were evaluated in this investigation. The films included rubbed-on (burnished) films of MoS$_2$ or (CF$_{1.1}$)$_n$ and PI-bonded films of (CF$_{1.1}$)$_n$ and MoS$_2$. Figure 5 shows the wear life as a function of temperature for each of these four films. At each test temperature, the PI-bonded (CF$_{1.1}$)$_n$ films proved to be superior to the other three films.

The wear life of rubbed-on (CF$_{1.1}$)$_n$ was found to be at least four times the wear life of rubbed-on MoS$_2$ over the entire test temperature range. PI-bonded (CF$_{1.1}$)$_n$ was found to be up to 10 times better than rubbed-on films of (CF$_{1.1}$)$_n$ over the same test temperature range. When the wear lives of the PI-bonded films were compared, it was found that, at 25°C, the wear life of the PI-bonded (CF$_{1.1}$)$_n$ was about twice that of the PI-bonded MoS$_2$ film. As the temperature was increased, the relative difference
between the wear lives became greater; and at 400 °C, the difference was about a factor of 60.

At 450 °C, the wear life of both PI-bonded films dropped off. Upon examination of the disks after test completion, it was found that the coatings had become powdery. Thus, the upper temperature limit for these PI-bonded films, in a dry-air atmosphere, is about 400 °C. It is 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 rubbed-on solid lubricant films.

A comparison of the wear lives, at 25 °C, for the four solid lubricant films is given in table I. The wear life of a commercially available sodium silicate bonded MoS₂ lubricant, which was tested under the same conditions, is also presented.

The rubbed-on films gave the shortest wear lives at 25 °C. The wear life of rubbed MoS₂ was 60 kilocycles; rubbed-on (CF₁.₁)ₙ films gave much better results, with a wear life of 230 kilocycles. The commercially available sodium silicate bonded MoS₂ film proved to be better than either of the rubbed-on films; its wear life was 500 kilocycles.

By using polyimide as the binder material for MoS₂, further improvement in the wear life was achieved. The wear life for this film was 1400 kilocycles. When (CF₁.₁)ₙ was substituted for MoS₂ in the polyimide film formulation, the results were even better. The wear life for these PI-bonded (CF₁.₁)ₙ films was 2950 kilocycles.
It is important to note that no attempt was made to optimize the weight ratio of $(\text{CF}_1)_{1n}$ to polyimide in formulating the film. An optimization study could result in an even better formulation.

The number of kilocycles at which the friction coefficient reached values of 0.10, 0.20, and 0.30 are given in table II. This information is given for each of the four lubricant films and for each of the test temperatures of 25°C, 100°C, 200°C, 300°C, 400°C, and 500°C. The wear-life ranking of the four films is the same at all three values of friction coefficient. Therefore, the choice of a friction coefficient of 0.10 or 0.20 instead of 0.30 as the failure criterion would not alter the conclusions concerning the relative durability of the coatings.

### Wear

In addition to providing a low friction coefficient and a long wear life, a good solid lubricant should also minimize wear. Thus, a series of experiments were performed to compare the wear occurring on the sliding riders.

#### TABLE II. NUMBER OF REVOLUTIONS ELAPSED BEFORE FRICTION COEFFICIENT

OF FOUR DIFFERENT FILMS REACHED VALUES OF 0.10, 0.20, AND 0.30

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<th>Friction coefficient</th>
<th>PI-bonded $(\text{CF}<em>1)</em>{1n}$</th>
<th>PI-bonded MoS$_2$</th>
<th>Rubbed $(\text{CF}<em>1)</em>{1n}$</th>
<th>Rubbed MoS$_2$</th>
<th>Number of revolutions, kilocycles</th>
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*Failed immediately.*

[Dry-air atmosphere (moisture content, 20 ppm); 440°C stainless-steel disks and riders; 1-kg load; 2.6-m/sec sliding speed.]
Once a solid lubricant film fails, gross damage to the metal surface can take place. This damage is, of course, not characteristic of the wear prior to failure of the solid lubricant. The wear tests were thus stopped after 1 hour of sliding (60 kilocycles). The wear was determined by measuring the circular scar diameter on the hemispherically tipped rider and then calculating the wear volume of the material removed from the rider.

Figure 6 gives the results of tests which were run at 25°C with a 1-kilogram load and in a dry-air atmosphere (moisture content, 20 ppm). Four solid lubricant films were tested: rubbed-on (CF$_{1.1}$)$_n$, rubbed-on MoS$_2$, PI-bonded (CF$_{1.1}$)$_n$, or PI-bonded MoS$_2$. The base metal, 440C stainless steel, was also run in the unlubricated condition.

The best results were obtained with the PI-bonded films. The wear occurring to the riders which slid on PI-bonded (CF$_{1.1}$)$_n$ films was $1 \times 10^{-13}$ cubic meters, while the wear to the riders which slid on PI-bonded MoS$_2$ films was $2 \times 10^{-13}$ cubic meters. The wear occurring to the riders which slid on rubbed-on films was about 10 times greater than for riders which slid on PI-bonded films. The values were $1.5 \times 10^{-12}$ cubic meters for riders which slid on (CF$_{1.1}$)$_n$ rubbed-on films and $4.0 \times 10^{-12}$ cubic meters for riders which slid on rubbed-on films of MoS$_2$. 
The wear of riders sliding on the unlubricated 440C stainless steel was $1 \times 10^{-9}$ cubic meters per hour of sliding time (60 kilocycles); the PI-bonded films thus reduced the wear to $1/10,000$th that of the unlubricated surface.

A photomicrographic comparison, at $25^\circ$ C, of the wear occurring to the riders and disks is shown in figure 7. The disks were lubricated with PI-bonded MoS$_2$, or PI-bonded $(\text{CF}_1)_n$. They were run in a dry-air atmosphere (moisture content, 20 ppm) until a friction coefficient of 0.30 was reached. This figure illustrates the fact that, even though the PI-bonded $(\text{CF}_1)_n$ film lasted over two times longer, the wear occurring to the riders and disks during their effective lives was nearly equivalent. The surface damage to the specimens was not particularly severe (fig. 7). Some metal-to-metal contact has probably taken place, but enough solid lubricant has found its way into the interface to provide protection.
SUMMARY OF RESULTS

A study was made of graphite fluoride as a solid lubricant in a polyimide binder. The results were as follows:

1. Polyimide (PI) is a suitable binder material for the solid lubricant graphite fluoride \((\text{CF}_{1.1})_n\).
2. The wear life of PI-bonded \((\text{CF}_{1.1})_n\) is superior to the wear life of similar films of PI-bonded molybdenum disulfide \((\text{MoS}_2)\).
3. The wear life of PI-bonded \((\text{CF}_{1.1})_n\) is 10 times greater than the wear life of rubbed-on films of either \((\text{CF}_{1.1})_n\) or \(\text{MoS}_2\).
4. The PI-bonded \((\text{CF}_{1.1})_n\) films performed exceptionally well at high temperatures; at 400 °C for example, they lasted about 60 times longer than PI-bonded \(\text{MoS}_2\) films.
5. The rider wear rates of the PI-bonded \((\text{CF}_{1.1})_n\) and PI-bonded \(\text{MoS}_2\) films were nearly equivalent at 250 °C.
6. The reduction in the wear life of PI-bonded \((\text{CF}_{1.1})_n\) above 400 °C is due to the degradation of the polyimide.

Lewis Research Center,
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