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Mechanism of Lubrication by Tricresylphosphate (TCP)

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National Aeronautics
and Space Administration
**Scientific and Technical
Information Branch**

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SUMMARY

A pin-on-disk tribometer equipped with an induction heater was used to study the coefficient of friction as a function of temperature for tricresylphosphate (TCP) on continuous-vacuum-melted (CVM) M-50 tool steel under the following conditions: TCP present in a liquid reservoir (bulk lubrication) and TCP applied as a liquid layer directly to the disk (limited lubrication). Under limited lubrication conditions, experiments were performed in dry air (<100 ppm H₂O), dry nitrogen (<20 ppm H₂O), dry nitrogen with the disks preheated to 700° C then cooled to room temperature before TCP application (preheated disks), and moist nitrogen using preheated disks. When the coefficient of friction was plotted as a function of the disk temperature, the friction decreased at a characteristic temperature, T_r , whose observed values were 265° C for bulk lubrication conditions in dry air, 225° C for limited lubrication conditions in dry air, and 215° C for limited lubrication conditions in dry nitrogen. No decrease in friction was observed with preheated disks; instead, a sharp failure temperature was observed at 218° C, which was taken as the temperature about which the behavior of TCP should be judged. X-ray photoelectron spectroscopy (XPS) confirmed the presence of phosphate on the surface of the iron pins used in the tribometer under TCP lubrication. Depth profile studies support the idea that a chemical reaction occurs between the TCP and the metal surface at T_r .

INTRODUCTION

The mechanism by which tricresylphosphate (TCP) acts as an antiwear additive in liquid lubrication conditions has been studied since 1940 when it was proposed that a eutetic phosphide was formed with the base metal (ref. 1). In 1965 Barcroft and Daniel suggested, as a result of phosphorus 32 studies, that a phosphate was formed on the surface rather than a phosphide (ref. 2). In the same year Godfrey used electron diffraction to study the wear particles from the surface of TCP-treated steel to demonstrate the presence of FePO₄ and FePO₄·2H₂O (ref. 3). Thin layer chromatography studies of TCP followed by neutron activation analysis for phosphorus 32 by Bieber, Klaus, and Tewksbury (ref. 4) led these authors to the conclusion that the acid phosphate impurities in TCP are the active lubrication ingredients. Subsequent work has resulted in a wide variety of detailed information (see ref. 5 for a recent literature survey), but a total understanding of the TCP-lubrication mechanism is not yet available.

One aspect of the TCP-lubrication mechanism that is not in the literature is the behavior of friction as a function of temperature over a wide enough range to show the chemical reaction between TCP and the lubricated surfaces. When such a chemical reaction occurs, the coefficient of friction should exhibit a sharp decrease at a characteristic temperature, T_r (ref. 6). The objective of the present investigation is to measure values of T_r for TCP under a variety of conditions and make a preliminary study of the reaction between the metal surface and TCP using X-ray photoelectron spectroscopy (XPS).

APPARATUS AND PROCEDURE

A pin-on-disk tribometer equipped with an induction heater was used for all friction measurements (fig. 1). The temperature was measured by an Ircon

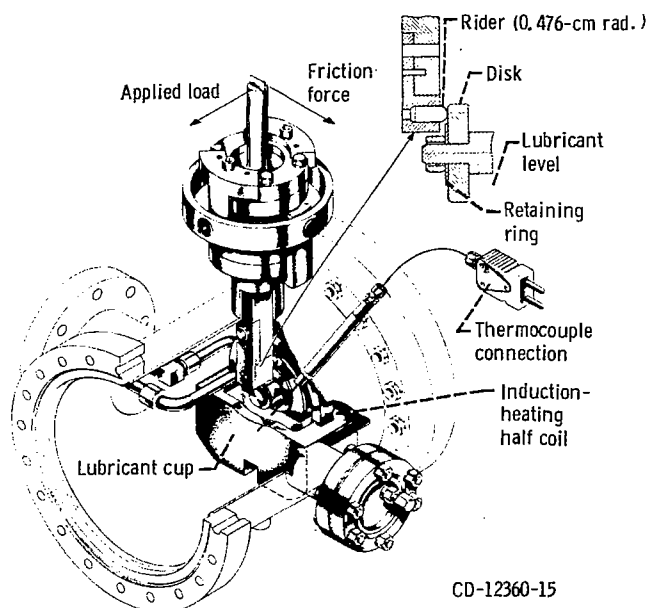


Figure 1. - The high temperature pin-on-disk tribometer.

model 300LC infrared pyrometer reading from the metal retaining ring on the disk. The TCP used in all experiments was Fisher Scientific Co. T-342.

X-ray photoelectron spectra were taken on an apparatus consisting of a commercial, cylindrical-mirror, electron-energy analyzer with a 25-eV pass energy and a magnesium target X-ray analyzer housed in a stainless-steel ultrahigh vacuum chamber. The X-ray source operated at 10 kV and 40 mA. An ion gun in the system could be used to ion etch the surface of samples with 5 keV argon ions. The pressure in the vacuum system was less than 1.33×10^{-6} Pa of argon. The analyzer was calibrated using the gold $4f_{7/2}$ binding energy of 84.0 eV. The binding energies and oxidation states of phosphorus were assigned according to the Handbook of X-Ray Photoelectron Spectroscopy (ref. 7).

Continuous-vacuum-melted, M-50 steel disks, 6.3 cm in diameter and 1.1 cm thick, were prepared by lapping (on a commercial lapping machine) followed by washing with Freon, scrubbing with levigated alumina paste and a clean felt cloth, and polishing with a polishing wheel charged with levigated alumina. The disks were then scrubbed with water and a clean, felt cloth, rinsed with distilled water, and dried in clean filter paper. The pins were washed with Freon, scrubbed with a paste of levigated alumina on a felt cloth, washed with warm water and a clean felt cloth, rinsed with distilled water, and dried in clean filter paper. The chamber of the tribometer was rinsed with 95 percent ethanol, followed by a liberal rinse with Freon to remove any trace of previous lubricants. Workers used polyethylene gloves to avoid contaminating the apparatus with skin oils during assembly.

The tribometer chamber was flushed with dry air or dry nitrogen for at least 2 hr before a run was begun. The dry air had less than 100 ppm H_2O , and the dry nitrogen less than 20 ppm H_2O . To remove adsorbed oxygen, disks were heated to $700 \pm 20^\circ C$ in dry nitrogen. After the disks had cooled to room temperature, the friction runs were made in either dry nitrogen or moist nitrogen.

The TCP was used as-received for dry air experiments, but, was deoxygenated and dehydrated by heating to approximately $150^\circ C$ under vacuum, then alternately flushing with dry nitrogen and evacuating at least three times for the nitrogen atmosphere experiments. For bulk lubrication measurements

25 ml of TCP was placed in a polyimide cup into which the disk rotated. Experiments were also conducted by placing less than 1 ml of TCP directly onto the rotating disk. These experiments are labeled "limited lubrication runs." For dry air measurements the TCP was placed on the disk using an ordinary pipette equipped with a rubber bulb. For nitrogen atmosphere measurements with limited lubrication, the degassed and dehydrated TCP was drawn into a clean, nitrogen-swept syringe. The TCP was placed on the disk by inserting the long needle of the syringe through a special tube placed into the thermocouple opening in the tribometer chamber. At least two runs were made under each set of conditions to check the reproducibility of the data.

All friction runs were made under a constant load of 9.8 N (1 kg) and a speed of 50 rpm (equivalent to a sliding speed of 13 cm/sec). All began with a run-in time of at least 25 min or until the friction trace showed no change in average value for the friction force or width of the friction trace. (A typical trace is shown in fig. 2.)

The infrared pyrometer was calibrated by placing a sample of mineral oil in the lubricant cup, inserting a Chromel-Alumel thermocouple into the oil, rotating the disk, and raising the temperature of the oil above 150° C. The heater was then turned off, and the emissivity of the pyrometer adjusted until the pyrometer matched the temperature of the oil, as measured by the thermocouple as the oil cooled. This procedure gave temperature readings that were reproducible to about 1°. Scale corrections were made for the pyrometer for each experiment by reading adjacent scales at the same temperature and noting any discrepancy between them. During an experiment, the temperature of the disk was increased by the induction heater at a rate of 3 to 8 C°/min. The temperatures were read at intervals of 1 min, while the temperature was increasing. Readings are valid to approximately 1°, and any uncertainty in the temperature is within the reproducibility of the data.

The friction force was detected by a strain gage and recorded on an XY recorder as a function of time. The friction force on the strain gage was calibrated using fixed weights, which can be attached directly to the pin mechanism of the tribometer. The coefficient of friction was determined for each temperature measurement and plotted as a function of temperature for each experiment.

The friction was measured until the temperature reached 400° C, until the friction increased to a point where metal-metal contact between the pin and the disk resulted in an audible screech, or until the ballast for the strain gage clattered against its support. One of these phenomena usually occurred when the coefficient of friction reached approximately 0.6 and showed a rapid rise in friction such that a temperature could be associated with the failure of the lubricant. These temperatures are referred to as failure temperatures.



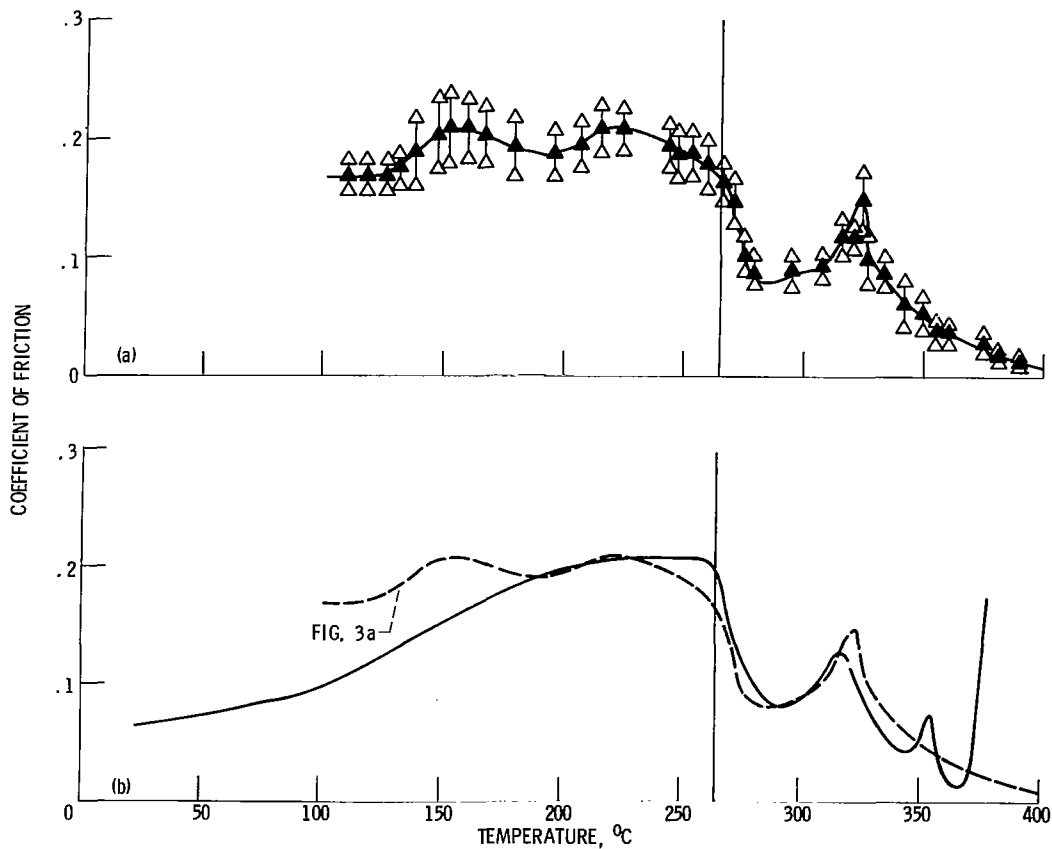
Figure 2. - Typical friction trace for CVM M-50 pin slid on CVM M-50 disk. Temperature range, 218° to 400° C. (See fig. 3(b) for friction-temperature curve.)

RESULTS AND DISCUSSION

Curves for the coefficient of friction as a function of temperature are shown in figures 3 to 7. All are for CVM M-50 pins sliding on CVM M-50 disks. The curves in figure 3 include the width of the friction trace to illustrate the variation in friction typically found in these experiments. For the sake of clarity other curves do not include the width of the friction trace.

Bulk Lubrication in Dry Air

Figure 3 shows the results of two experiments run in dry air under bulk lubrication conditions. The curves show a drop in friction that can be associated with chemical reactions at 265° and 320° C. One curve in figure 3(b) shows a third drop at 353° C. The characteristic temperature T_r was assigned the value 265° C because it most closely resembles curves for other materials (ref. 6). The succeeding peaks can be attributed to either other chemical reactions to which the friction is sensitive or to the disk picking up layers of lubricant from the bulk lubricant, wetting the surface, and failing at the temperatures observed. To minimize the latter possibility, subsequent runs were made under limited lubrication conditions as described in the Apparatus section.



(a) $T_r = 264^\circ \text{C}$. (The width of the friction trace is illustrated.)

(b) $T_r = 265^\circ \text{C}$. (Curves for two experiments are shown.)

Figure 3. - Coefficient of friction as function of temperature for CVM M-50 pin sliding against CVM M-50 disk in dry air under bulk lubrication with TCP.

Limited Lubrication in Dry Air

The coefficient of friction as a function of temperature for limited lubrication in dry air is shown in figure 4. Note that the first transition occurred at approximately the same temperature for both experiments, 225°C , but at a significantly lower temperature than in the bulk lubrication experiments, 265°C . Once again details are different in the higher temperature portions of the curves. It is possible that the lower values of T_r for limited lubrication conditions are more representative of the chemical reactions conditions because the bulk lubricant can dissipate the surface reaction energy in the greater bulk of the lubricant. The fact that subsequent limited lubrication runs show T_r values close to 225°C tends to substantiate this idea.

Limited Lubrication in Dry Nitrogen

Figure 5 presents data for limited lubrication of CVM M-50 pins on CVM M-50 disks in a dry (<20 ppm H_2O) nitrogen atmosphere. A decrease in friction was still observed, indicating a chemical reaction with the metal surface. Values of T_r are found at 215°C . There was some detail at temperatures above T_r but failure occurred at temperatures (about 250°C), well below failure temperatures in dry air (375°C). This behavior suggests a reaction for TCP that is essentially the same in both dry air and dry nitrogen. Sweeping out the tribometer chamber with nitrogen will not remove oxygen adsorbed on the metal surfaces. This oxygen could react with the TCP to produce a thin film of product and yield frictional behavior similar to that in air. The limited oxygen supply should result in significantly less reaction product and in a lower failure temperature as the product film is worn away by the pin sliding over it. This is precisely what was observed.

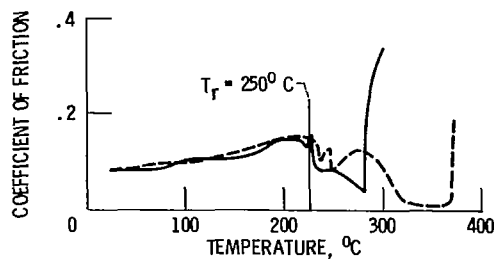


Figure 4. - Coefficient of friction as function of temperature for CVM M-50 pin sliding against CVM M-50 disk in dry air under limited lubrication with TCP. Curves for two experiments are shown.

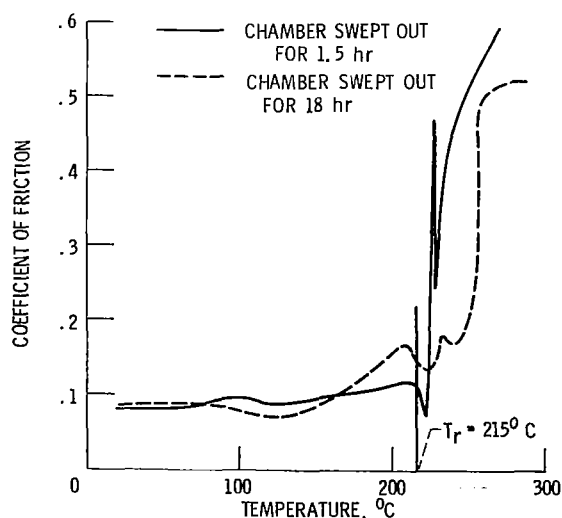


Figure 5. - Coefficient of friction as function of CVM M-50 pin sliding against CVM M-50 pin in dry nitrogen under limited lubrication with TCP. Curves for two experiments are shown.

Limited Lubrication in Dry Nitrogen with Preheated Disks

To verify the adsorbed oxygen idea, disks were heated to approximately 700° C in dry nitrogen before the friction run to remove the adsorbed oxygen. (This procedure is described in the Apparatus and Procedure section.)

Figure 6 presents the data for limited lubrication with degassed and dehydrated TCP for CVM M-50 pins on preheated CVM M-50 disks. There is no drop in friction in these curves. Instead, a sharp, reproducible failure temperature is observed at 218° C. The absence of a decrease in friction lends support to the idea that the chemical reaction of TCP at T_r is essentially a reaction with oxygen. The 218° C failure temperature was very close to the T_r values observed in both dry air and dry nitrogen under limited lubrication conditions (225° and 215° C, respectively). This suggests that 218° C may be a basis temperature around which the behavior of TCP revolves. Below 218° C TCP wets the surface and lubricates the metal with the adsorbed film. At 218° C the adsorbed film of TCP fails and chemical reaction must occur in order to promote further lubrication. The T_r values indicate that such a reaction does indeed occur when oxygen is present. Consequently, TCP (or its reaction products) continues to lubricate at temperatures greater than 218° C. If the reaction with oxygen were to occur at temperatures significantly above 218° C, the TCP would fail in its lubricating action before chemical reaction could occur to generate secondary lubricating species. Under these conditions, the friction would rise until the chemical reaction would occur, at which temperature the friction would decrease as the secondary lubricating species were generated. Of course, if reaction with oxygen were to occur below 218° C, no failure of the adsorbed layer would be observed, and the friction would decrease at T_r .

According to the model drawn from the frictional behavior of TCP in air and nitrogen with deoxygenated disks, the secondary lubrication species of TCP must be oxidation products of TCP. The phosphate moiety is not chemically reactive with oxygen. A likely candidate for reaction is the methyl group, which is readily oxidized into an aldehyde or carboxylic acid functional group. Either could easily react with the metal surface to continue

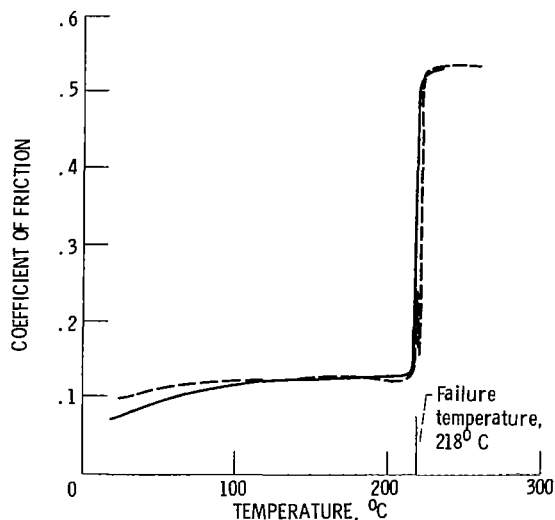


Figure 6. - Coefficient of friction as function of temperature for CVM M-50 pin sliding against preheated CVM M-50 disk on dry nitrogen under limited lubrication with TCP. Curves for two experiments are shown.

lubrication above T_r . Further reaction with oxygen may remove the organic section of TCP and leave the iron phosphate observed by Godfrey (ref. 3).

Limited Lubrication in Moist Nitrogen with Preheated Disks

The role of moisture in the action of TCP has been studied and is usually discussed relative to the hydrolysis of TCP to an acid phosphate (refs. 2, 3, and 8). To study the role of moisture under conditions where only hydrolysis can occur, experiments were conducted in moist nitrogen using preheated disks. The curves for these experiments are shown in figure 7. The experiments were performed in nitrogen with a relative humidity of 81 and 83 percent (at 21° C).

The curves in figure 7 exhibit no decrease in friction and therefore no chemical reaction at the surface. Each curve does show a sharp failure temperature that is lower than the 218° C T_r value observed in dry nitrogen and that seems to depend on the length of time the preheated disks are exposed to the moisture. In any event the results obtained here do not support the idea of a hydrolysis reaction between the water and the TCP. Such a reaction would generate an acid phosphate that would react with the metal and produce a decrease in friction. This was not observed.

The preheating of the disks produced on the surface a blue-black film which is most likely an oxide film since reaction between the metal and nitrogen is usually accomplished in the presence of hydrogen. If an acid phosphate were formed by the hydrolysis of the TCP, the reaction between the acid phosphate and the oxide would be at least as likely as the reaction between the acid phosphate and the metal. Since no such reaction was observed, we can conclude that TCP did not react with the water under the conditions of this experiment.

The failure temperatures of 202° and 190° C were lower than the 218° C found for dry nitrogen. This may be due to the TCP being displaced on the

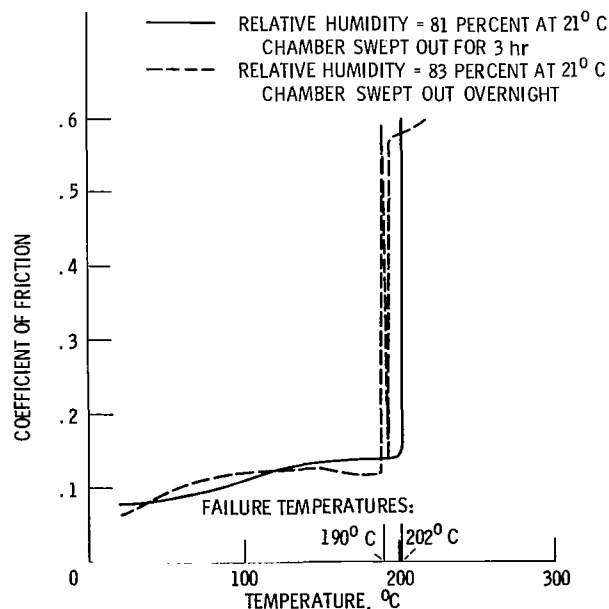


Figure 7. - Coefficient of friction as function of temperature for CVM M-50 pin sliding against preheated CVM M-50 disk in dry nitrogen under limited lubrication with TCP. Curves for two experiments are shown.

disk surface by the water. The longer the moisture is present, the greater the TCP displaced. Alternatively, the water may react with the metal oxide present and cause decreased adsorption of the TCP on the hydrous oxide.

X-Ray Photoelectron Spectroscopy Investigations

The presence of characteristic temperatures T_r for TCP on CVM M-50 steel suggests that a chemical reaction has occurred between the TCP and the metal surface. X-ray photoelectron spectroscopy (XPS) was used to investigate the product of this reaction. The wear scars formed by the M-50 pins sliding on M-50 disks were too small to be investigated adequately by XPS. Consequently, the XPS measurements were carried on the wear scars of iron pins that had been slid on M-50 disks under bulk lubrication conditions in dry air. The coefficient of friction as a function of temperature is shown in figure 8. The value of T_r is taken as 313°C .

The XPS study was made using three iron pins. The first was a pin taken from an experiment where the temperature was raised to 280°C , that is, lower than T_r and presumably before the chemical reaction occurs. The second pin was from an experiment where the temperature was raised to 345°C , that is, at the bottom of the valley in the coefficient of friction and presumably after the chemical reaction had occurred. The third pin was from an experiment which was run to the highest temperature, that is, above 390°C and to failure.

An XPS study was made on each pin to identify the material on the surface of the pin and to establish the relative amounts of chemical product formed. The XPS spectrum and the depth profile are shown in figure 9. Note that the binding energy observed in the XPS spectrum for the phosphorus was 134 eV, the energy expected for phosphate, but that no peaks were observed

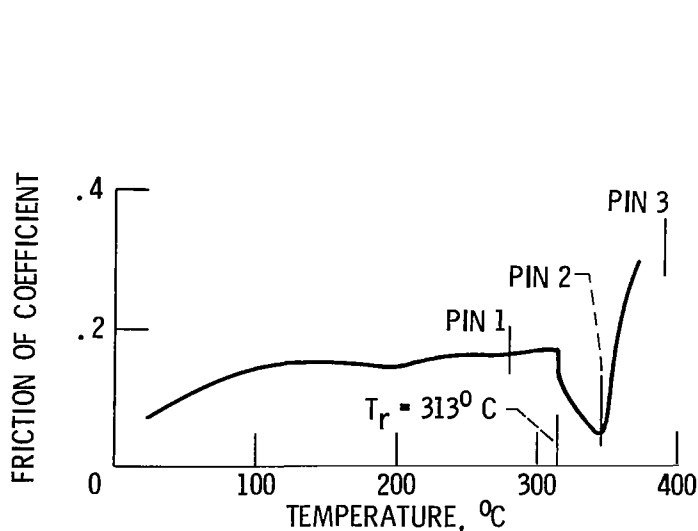
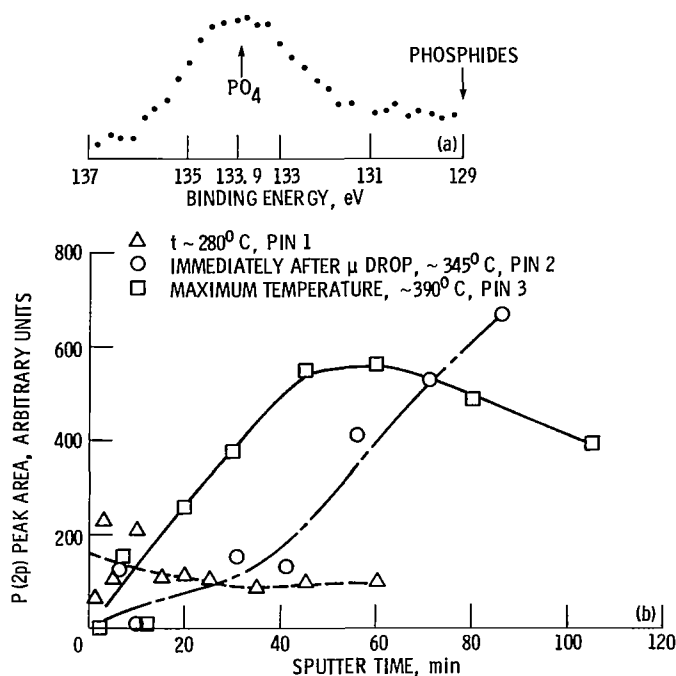


Figure 8. - Coefficient of friction as function of temperature for an iron pin sliding against CVM M-50 disk in dry air under bulk lubrication with TCP.



(a) X-ray photoelectron spectrum.
(b) X-ray photoelectron depth profile.

Figure 9. - X-ray photoelectron spectrum and depth profile for P(2p) phosphate peak on iron pins.

at the position expected for phosphide (ref. 7). This is in agreement with the conclusions of previous workers on the identity of the TCP-steel reaction product (refs. 2 and 3). The depth profiles for the phosphorus peak of each pin are presented in figure 9(b).

The profile for pin 1 (i.e., the pin heated to only 280° C) showed surface phosphate, which decreased as the surface was sputtered away. Compared with the second and third pins, the first pin showed only a small amount of phosphorus on the surface. The presence of any phosphorus before T_r was reached is in agreement with the observations of other workers who detected phosphate on metal surfaces under quite mild temperature conditions (refs. 3, 5, 9, and 10) and suggests that TCP is capable of reacting with metal surfaces under asperity conditions to form a very thin surface film. The second pin revealed a surface layer which grew richer in phosphate as the surface was sputtered away. The increasing concentration of phosphate is revealing of a chemical reaction occurring between the TCP and the metal surface at a T_r of 313° C.

The third pin exhibits a maximum in the phosphate concentration in the depth profile. The sputtering rate of gold was measured at approximately 1 nm/min. Sputtering rates for various materials are relatively constant. Using the sputtering rate for gold as the rate for the iron pins, the maximum phosphate concentration occurs approximately 50 nm into the surface. The presence of this maximum is consistent with the reaction between the TCP and the metal surface at T_r . The film formed on the surface by the reaction does not grow after the temperature is raised well above T_r and is worn away as the pin continues to slide over the disk. Not all the phosphate is worn away, and the sputtering manages to penetrate the phosphate film, thereby producing the maximum in the curve. Continued sliding would wear away all the phosphate.

CONCLUSIONS

The results of investigations carried out with TCP lubricating CVM M-50 steel in various environments lead to the following conclusions.

1. The characteristic temperature T_r , where friction is reduced due to the chemical reaction between the lubricant and the metal surface, are 265° C in dry (<100 ppm H₂O) air under bulk lubrication conditions, 225° C in dry air under limited lubrication conditions, and 215° C in dry (<20 ppm H₂O) nitrogen under limited lubrication conditions.
2. Bulk lubrication conditions tend to raise the values of T_r for TCP on CVM M-50 steel in dry air.
3. Oxygen is required for TCP to react with the metal surfaces.
4. Sweeping out the tribometer chamber did not remove the oxygen adsorbed on the metal surfaces. Heating to 700° C in dry nitrogen did effectively eliminate the adsorbed oxygen.
5. The coefficient of friction for preheated disks in dry nitrogen exhibited a sharp, reproducible failure temperature at 218° C. This failure temperature is suggested as a basis temperature around which the behavior of TCP can be judged.
6. The presence of moisture in dry nitrogen with preheated disks caused sharp failure temperatures that were lower than 218° C and that seemed to depend on the length of time the metal surfaces were exposed to the moisture.
7. The presence of moisture does not result in a decrease in friction in nitrogen on preheated disks and does not provide evidence to support the hydrolysis of TCP by moisture.

8. X-ray photoelectron spectra (XPS) of iron pins which had been slid against CVM M-50 disks lubricated with TCP in dry air exhibited a strong phosphate peak, but no phosphide peak.

9. A depth profile study of the phosphate layers on the iron pins by XPS supported the view that TCP reacted chemically with the metal surfaces at the temperature T_r .

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, October 28, 1982

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