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Effect of Substrate Chemical Pretreatment on the Tribological Properties of Graphite Films

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EFFECT OF SUBSTRATE CHEMICAL PRETREATMENT ON THE TRIBOLOGICAL PROPERTIES OF GRAPHITE FILMS

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Abstract

Rubbed films of natural flake Madagascar graphite were applied to ASTM A-355(D) steel with chemical surface pretreatments of zinc phosphate, gas nitride, salt nitride, sulfo-nitride, and with mechanical pretreatment (sandblasting). SAE 1045 steel pins were slid against these films using a pin-on-disk tribometer. The results indicate that two different lubricating mechanisms can occur. In one the chemical surface pretreatment, the graphite can mix together to form a surface layer of the two constituents; this plastically flowing layer provides the lubrication. The longest endurance lives and the lowest pin wear rates were obtained with this mechanism. In the other, surface topography appeared to control the mechanism: A rough surface was necessary to serve as a reservoir to supply the graphite to the flat metallic plateaus where it was sheared in very thin films between the plateaus and the sliding pin surface. For this mechanism, chemical pretreatment seemed to do little more than serve as a means for roughening the surface. Mean friction was not significantly influenced by chemical pretreatment, but surface roughness effects were observed.

INTRODUCTION

The object of solid film lubrication is to insert between two relatively moving surfaces a solid or solids which have low shear strength. The most widely used solid lubricants with low shear strengths are the layer-lattice or laminar solids, such as graphite and molybdenum disulfide (1-3). Both have hexagonal crystal structures, and, when rubbed under pressure, the basal planes orient on the surface so that they become parallel to the sliding interface (4-7). This facilitates shear and the crystals' ability to flow into themselves.

The fact that a solid has a layer-lattice crystal structure does not ensure good lubricating properties. Both boron nitride and mica have layer-lattice structures, yet neither is considered a good solid lubricant (1,3,4,8,9). Some evidence suggests they are not good lubricants because they do not adequately adhere to the surfaces to be lubricated (8). Thus, besides having low shear strength, the ability of the solid lubricant to adhere to the surfaces is very important.

Adherence to the surfaces may be chemical and/or physical in nature. Even such low energy surfaces as polytetrafluoroethylene (PTFE) can chemically bond to a metal surface if the metal is sufficiently clean (10). Physical bonding occurs by the solid lubricant simply filling up the valleys between the metallic asperities on the surface (2,11,12). The amount of physically bonded solid lubricant can often be increased by a controlled increase in the roughness of the surface. Physical bonding has also been suggested to occur for layer-lattice compounds such as graphite or MoS_2 by the sharp edges of the crystallites embedding into the metallic surfaces (4,13).

Chemical pretreating of metallic substrates has been shown to increase the endurance life of solid lubricants (2,14,15). The mechanisms by which this occurs are not well understood. Is it because chemical pretreating of the surfaces increases the adherence of the solid lubricant, or is it because the chemical pretreatment roughens the surface and provides microreservoirs for the solid lubricant?

The purpose of this investigation was to obtain a better understanding of the mechanisms by which graphite provides lubrication by studying the effect of rubbed (burnished) films of graphite on various chemically pretreated surfaces. Friction coefficients, endurance lives, pin wear rates, film wear surface morphology, and energy dispersive x-ray (EDS) analysis of the film wear tracks were determined for graphite films applied to sandblasted, zinc phosphated, gas nitrided, salt nitrided (rough and smooth surfaces), and sulfo-nitrided surfaces.

MATERIALS

Natural flake Madagascar graphite with a mean particle size of 15 μm (size distribution, 4 to 40 μm) and a specific gravity of 2.25 was used in this study. The pin specimens were made from SAE 1045 steel with a Rockwell hardness of B-90. The disk specimens were made from ASTM A-355(D) steel with a Rockwell hardness of C-28. The surface treatments to the steel, sandblast, zinc phosphate, gas nitride, salt nitride, and sulfo-nitride, were applied before coating with graphite. The surface roughnesses of the disks are given in Table 1 before and after coating with graphite. Graphite was applied to salt nitrided disks with two surface roughnesses. The surface pretreatments were applied by commercial manufacturers who specialized in providing these pretreatments.

The moist air used in these experiments was of 50-percent relative humidity.

APPARATUS

A pin-on-disk sliding friction apparatus was used in this study. The apparatus has been described in previous reports (11,12). Basically the friction specimens (Fig. 1) consisted of a flat disk (6.3 cm diam) and a stationary, hemispherically tipped pin (0.476-cm rad.). The pin slid on a 5-cm-diam track on the disk at a linear sliding speed of 2.6 m/sec (for a disk rotation of 1000 rpm).

The apparatus used to apply the solid lubricant powder to the disks is shown in Fig. 2. The disk was attached to the vertical shaft of a small electric motor by means of a cup-shaped holder. Two vertical rods were used to restrain a floating metal plate to which were attached the solid lubricant applicators. The backs of polishing cloths were used as applicators. The rubbing load was applied by placing two 1-kg weights on top of the metal plate.

The rubbing apparatus was designed to fit within the bell jar of a vacuum system. The bell jar was evacuated to 1000 Pa and then backfilled to atmospheric pressure with 50-percent relative humidity air.

PROCEDURE

Specimen Cleaning

The pretreated steel disk surfaces were cleaned by washing with ethyl alcohol and then by brushing under running distilled water to remove dust particles. Clean, dry air was used to quickly dry the surfaces. The disks were stored in a desicator until they were ready for coating with graphite. The pins were washed with ethyl alcohol and then scrubbed with a water paste of levigated alumina. The pins were then rinsed in distilled water and dried with clean dry compressed air. Graphite was not applied to the pins.

Film Application

The procedure for applying the rubbed films was as follows:

(1) Apply a small amount of graphite powder to the cleaned disk surface and spread it evenly over the surface with the back of a polishing cloth.

(2) Apply about 0.5 g of graphite to the contact zone of the applicator and distribute it evenly.

(3) Assemble the apparatus as shown in Fig. 2 and apply two 1-kg weights for the load.

(4) Evacuate the bell jar to 1000 Pa and then backfill it to atmospheric pressure with an atmosphere of moist air (50 percent relative humidity). Continue to purge the bell jar with moist air until the disk is removed from the apparatus.

(5) Set the disk into rotation by gradually increasing the speed to 15 rpm and rub for 1 hr.

(6) Remove the disk from the apparatus and blow the loose graphite debris from the surface with dry compressed air.

Test Conditions

The specimens were inserted into the apparatus, and the chamber was sealed. Moist air was purged through the chamber of 2000 cm^3 at the rate of 1500 cm^3/min for 15 min before commencing the test. The disk was set into rotation at 1000 rpm (2.6 m/s), and a 9.8-N load was gradually applied. The test temperature was 24°±3° C (ambient).

Each test was stopped after 1 km of sliding and at failure (when the friction coefficient reached a value of 0.25). One test for each surface pretreatment was also stopped after 3 km of sliding. The pin and disk were removed from the apparatus (after stopping the tests), and the contact areas were examined by optical microscopy and photographed. Surface profiles of the disk wear track were also taken. The specimens were then remounted in the apparatus, and the test procedure was repeated. The pin was not removed from its holder, and locating pins ensured that it was returned to its original position.

The specimens were also observed in a scanning electron microscope (SEM), and EDS spectra of undisturbed films and wear tracks were taken.

RESULTS

Endurance Life

Endurance life was arbitrarily defined as the number of sliding revolutions (in kilocycles) to reach a friction coefficient of 0.25. The endurance lives obtained with the various pretreatments were compared (Fig. 3 and Table 2). Graphite films applied to the zinc phosphated surfaces provided much longer endurance lives than the other pretreatments. The sulfo-nitrided and the rough, salt-nitrided surface gave the shortest endurance lives. The sandblasted, gas nitrided, and the smooth, salt nitrided surfaces gave intermediate results. Possible reasons for these results will be discussed in a later section.

Friction Coefficient

For all of the experiments the friction coefficient started out at some low value and then varied about this value for an extended period. At some point the friction would then rise gradually with increasing sliding time until the failure point (0.25 friction coefficient) was reached. Table 2 gives mean values for the low, constant friction coefficients obtained in each test. Figure 4 gives the variation in friction coefficient obtained from test to test. Except for the smooth, salt-nitrided surface (which is slightly higher), the friction coefficient variation for any one particular surface generally overlaps the friction coefficients obtained for the other surfaces (Fig. 4). The sandblasted, gas-nitrided, and sulfo-nitrided surfaces produced greater variations in friction coefficient than the other surfaces.

Wear Rates

Pin wear volume was calculated after 1 and 3 km of sliding and after failure. Table 2 gives calculated pin wear rate values in terms of wear volume per unit distance of sliding for sliding intervals of 0 to 1 km, 1 to 3 km, and 1 km to the failure point. A bar graph comparison of these pin wear rate values shows that during the first kilometer of sliding, the pin wear rate is considerably higher than it is for the remainder of any individual test (Fig. 5). Obviously, this is due to run-in effects. The sliding interval from 1 to 3 km represents the steady-state period of wear and a low value for any particular test. The sliding interval from 1 km to the failure point represents the wear obtained during the steady-state period and the increased wear that occurs near the end of the test when the lubricant is gradually depleted. This interval is given, rather than 3 km to the failure point, because some tests were not stopped after 3 km of sliding and it was desirable to compare all tests for the same sliding intervals.

The zinc-phosphated surface pretreatment gave by far the lowest pin wear rates, after run-in (Table 2 and Fig. 5). The sandblasted and the smooth, salt-nitrided surface pretreatments produced the next best results, although the sandblasted surface appeared to give lower wear during the later sliding intervals, as indicated by the pin wear rates obtained during the 1-km-to-failure sliding interval. The gas nitrided surface gave the next best pin wear rates, followed by the rough, salt-nitrided surface and the sulfo-nitrided surface. Possible reasons for these differences in wear rates will be discussed in a later section.

The wear of the pretreated disk surfaces was studied by taking surface profiles of the wear tracks at the end of each test. Figure 6 gives representative profiles for each surface pretreatment. Considerable wear of the zinc phosphated surface occurred, but the rest of the surfaces tended to only have the high spots worn off. Wear morphology of the graphite-rubbed, pretreated surfaces will be discussed in the next section.

DISCUSSION

Film Wear Surface Morphology

Photomicrographs of the rubbed graphite films applied to the various pretreated ASTM A-355(D) steel disk surfaces are shown in Fig. 7. Coating tended to smooth the surfaces, but the relative order of roughness remained the same before and after coating (Table 1). Both mechanical and chemical pretreatment influence the surface morphology of the rubbed graphite films (Fig. 7).

Photomicrographs of small surface areas of the wear tracks of the rubbed graphite films applied to the various pretreated steel disks are shown in Figs. 8 to 12. The gas-nitrided, salt-nitrided (both rough and smooth), and the sulfo-nitrided wear track surfaces were not covered by a continuous film of graphite after 6 kcycles of sliding (Figs. 8 to 10). Small metallic plateaus can be seen in the wear track for the gas-nitrided surface (Fig. 8(a)) and the smooth, salt-nitrided surface (Fig. 9(a)). Larger, ridgelike plateaus can be seen for the rough, salt-nitrided disk (Fig. 9(b)), and the sulfo-nitrided surface is covered with large metallic plateaus (Fig. 10).

The sandblasted and the zinc phosphated surfaces were covered with a much more continuous film of graphite than the others after 6 kcycles of sliding (Figs. 11(a) and 12(a)). In fact, the zinc phosphated surface wear track gives the appearance that the graphite and the zinc phosphate have mixed to form a continuous layer on the surface.

Failure of the films appeared to be caused by the depletion of the graphite and the exposure of larger metallic regions. At failure, all the surfaces were covered by a black, powdery debris (Fig. 8(a)) which EDS analysis showed to be composed of iron or iron compounds.

The endurance lives of the films appear to be related to the film forming ability of the graphite on the pretreated surface and to the fact that the rough surface acts as a reservoir for the graphite. Defining an optimum surface roughness value is difficult, however, since radically different surface structures can produce the same numerical surface roughness value (such as R_a). For example, the sandblasted and the rough, salt-nitrided surface have similar surface roughness values (Table 1), but the distribution of the metal on the surface is entirely different (Figs. 9(b) and 11(b)). The rough, salt-nitrided surface tends to have long ridges running parallel to the pin sliding direction; while the sandblasted surface tends to have flat plateaus randomly scattered throughout the wear track region. Since the pin is sliding parallel to the metal ridges of the rough, salt-nitrided surface, it is difficult (if not impossible) for the graphite to get into the contact area once the original film has worn away. But on the sandblasted surface, since graphite surrounds the metallic plateaus, it is easy for the graphite to flow into the contact. In Fig. 11(b) thin layers of graphite can be seen on the metallic plateaus.

The zinc phosphate surface pretreatment tended to function differently from the others. The fact that it was thicker (about 15 μm) may have helped the graphite mix with the zinc phosphate to form a continuous film. The wear process tended to be gradual through the zinc phosphate layer, during which crumbling of the layer was found to occur (Fig. 12(b)). The EDS spectra of the wear track area on the graphite-film - zinc-phosphated - steel surface (Fig. 13) show that the phosphorus and zinc were gradually depleted and the amount of iron increased with sliding duration. At failure there was essentially no phosphorus or zinc on the wear track.

Transfer Film Morphology

Photomicrographs of the transfer films after 1 km of sliding are shown in Fig. 14. All pins showed a buildup of graphite in the contact inlet region and very thin plastically flowing films across the contact (Fig. 14). The zinc phosphated surface produced graphite transfer films that were thicker and more continuous than the others. The gas-nitrided and sulfo-nitrided produced transfer films were more discontinuous than the others. And the rough, circumferentially grooved, salt-nitrided surface produced stress risers (grooves) in the pin.

Failure was characterized by the buildup of powdery wear debris on the pin. This debris did not coalesce to produce plastically flowing films. Again, EDS analysis showed the debris to be composed of iron or iron compounds.

CONCLUSIONS

Friction, wear, and wear surface morphology studies of rubbed graphite films applied to pretreated ASTM A-355(D) steel disk surfaces indicate that

1. The zinc phosphate chemical pretreatment provided the longest endurance lives and the lowest pin wear rates.

2. The zinc phosphate (approx. 15 μm thick) and the graphite mixed together to formed a thin, plastically flowing surface layer (approx. 3 μm thick). Some crumbling of the zinc phosphated-graphite surface occurred with sliding, but residual graphite from the wear debris and from that distributed deep into the very rough pretreated surface enabled a new, thin, plastically flowing surface layer to form. Failure occurred once the zinc phosphated layer had been depleted.

3. The surface layers of the other pretreated specimens showed minimal mixing with the graphite. The pin wear and the endurance life seemed to be controlled by the pretreated surface topography. The results indicate that surface topography should be optimized so that the valleys in the surface serve as reservoirs to supply graphite to small, metallic plateaus.

4. Sandblasted surfaces provided longer endurance lives and lower pin wear rates than the chemical pretreated surfaces (except for the zinc phosphated surfaces), indicating that the chemical treatment did not increase the adherence of the graphite to the disk; and, even if it did, the surface topography factor was of more importance.

5. Chemical pretreatment of the surfaces did not markedly affect the mean value of friction coefficient obtained. Surface roughness seemed to have a slight effect, as evidenced by the higher friction obtained for the smooth, salt-nitrided disk (0.18 versus 0.14 for the rough disk).

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Table I

Summary of Materials Properties

Disk surface treatment	Rockwell hardness		Surface roughness of disk, CLA, μm	
	Pin	Disk[a]	Before coating	After coating
Sandblasted	B 90	C 28	1.1 - 1.2	0.85 - 1.0
Gas-nitrided	B 90	C 57	0.45 - 0.70	0.45 - 0.60
Zinc-phosphated	B 90	C 26	3.2 - 3.8	1.5 - 1.8
Salt-nitrided, smooth surface	B 90	C 29	0.32 - 0.39	0.27 - 0.34
Salt-nitrided, rough surface	B 90	C 29	1.2 - 1.4	1.1 - 1.2
Sulfo-nitrided	B 90	C 27	1.3 - 2.5	1.0 - 1.5

[a]After treatment.

Table II

Summary of Experimental Results

Disk surface pretreatment	Test	Mean friction coefficient	Endurance,[a] kilocycles	Pin wear rate, m^3/m		
				0 to 1 km	1 to 3 km	1 km to failure point
Sandblasted	1	0.11	167	530×10^{-18}	----------	80×10^{-18}
	2	.15	176	320×10^{-18}	----------	44×10^{-18}
	3	.13	179	630×10^{-18}	20×10^{-18}	70×10^{-18}
Gas-nitrided	1	0.12	99	$1\,200 \times 10^{-18}$	----------	570×10^{-18}
	2	.14	112	$1\,700 \times 10^{-18}$	----------	530×10^{-18}
	3	.16	78	$1\,300 \times 10^{-18}$	120×10^{-18}	910×10^{-18}
Zinc-phosphated	1	0.12	412	270×10^{-18}	----------	6.8×10^{-18}
	2	.13	507	210×10^{-18}	----------	7.7×10^{-18}
	3	.13	470	280×10^{-18}	5×10^{-18}	10×10^{-18}
Salt-nitrided, smooth surface	1	0.19	150	540×10^{-18}	----------	280×10^{-18}
	2	.18	155	490×10^{-18}	20×10^{-18}	148×10^{-18}
Salt-nitrided, rough surface	1	0.15	30	$7\,200 \times 10^{-18}$	----------	1100×10^{-18}
	2	.14	31	$6\,300 \times 10^{-18}$	3900×10^{-18}	3900×10^{-18}
Sulfo-nitrided	1	0.11	46	$9\,900 \times 10^{-18}$	----------	1640×10^{-18}
	2	.12	33	$11\,000 \times 10^{-18}$	----------	4700×10^{-18}
	3	.14	20	$6\,800 \times 10^{-18}$	----------	5200×10^{-18}

[a]Kilocycles of sliding to reach a friction coefficient of 0.25.

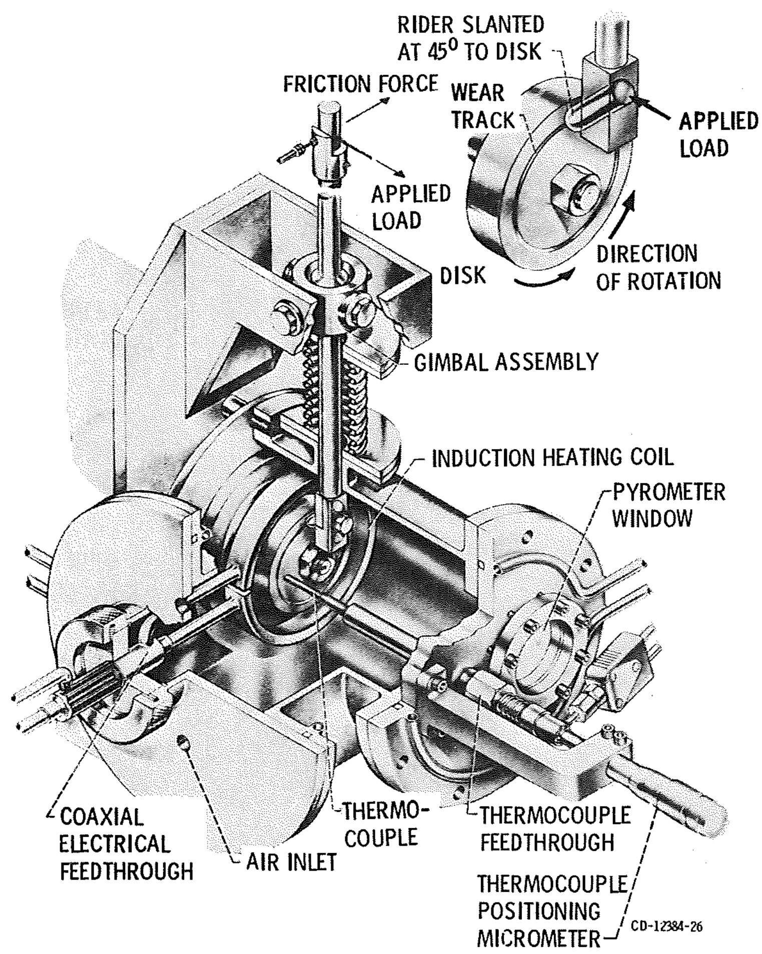

Fig. 1. - High speed friction and wear rig.

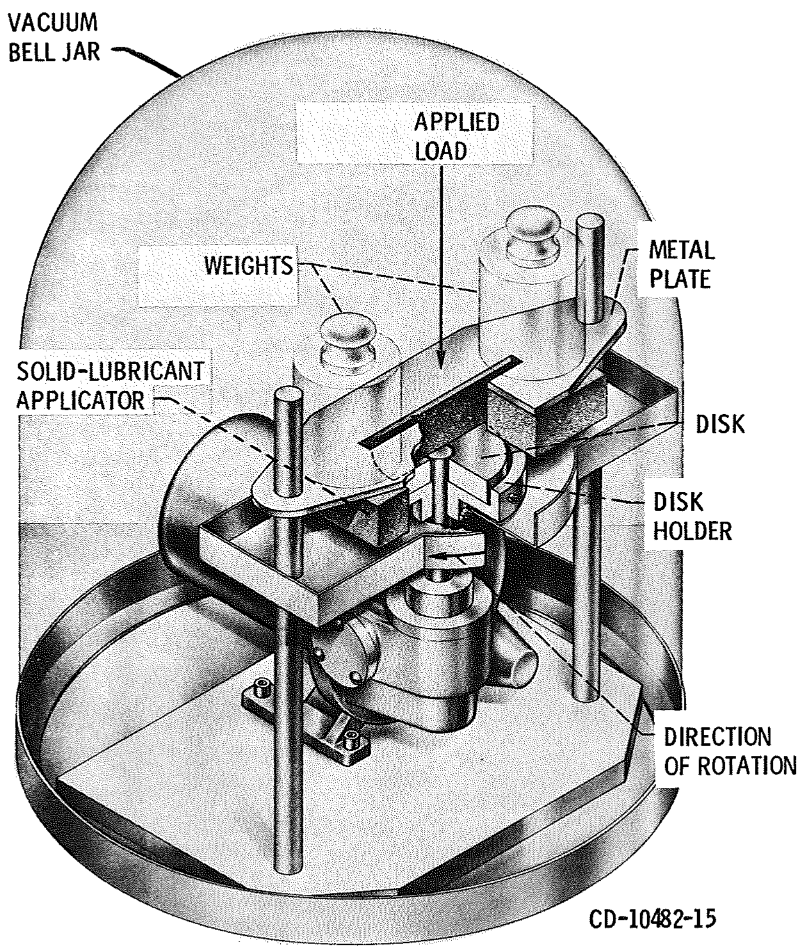

Fig. 2. - Burnishing aparatus.

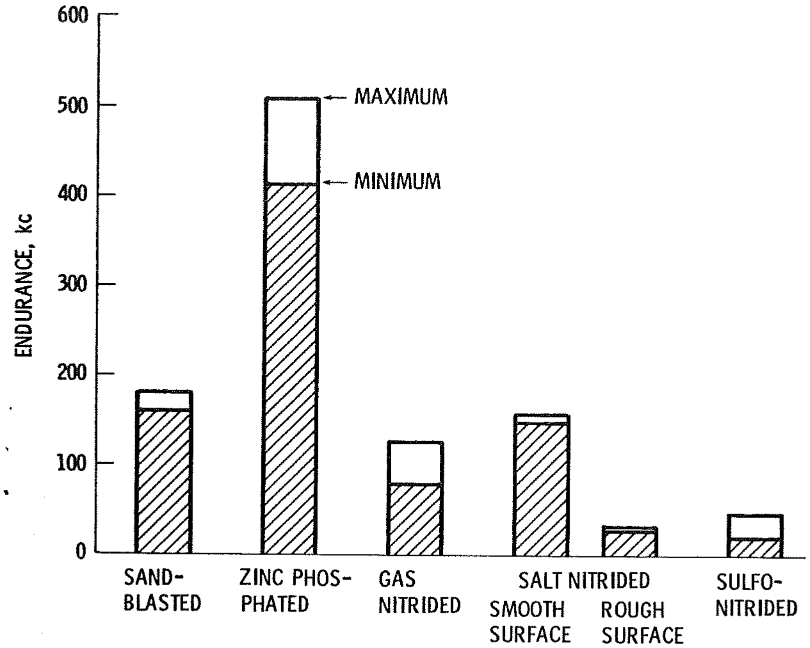

Fig. 3. - Endurance of rubbed graphite films on pretreated steel disks.

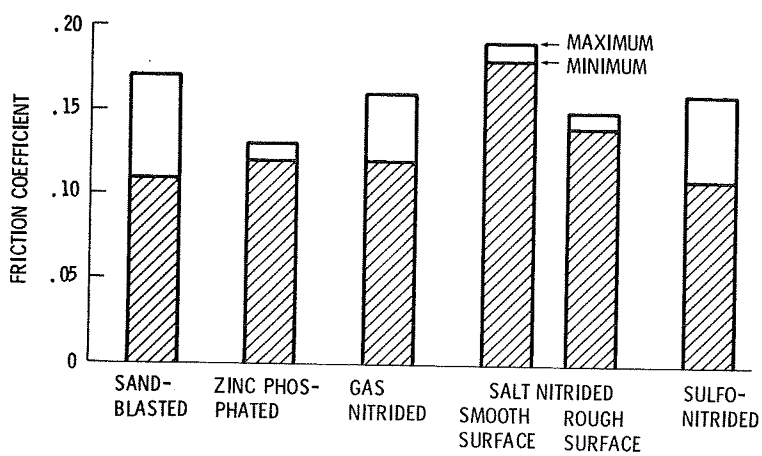

Fig. 4. - Friction coefficient for SAE 1045 pins sliding against graphite films rubbed onto pretreated steel disks.

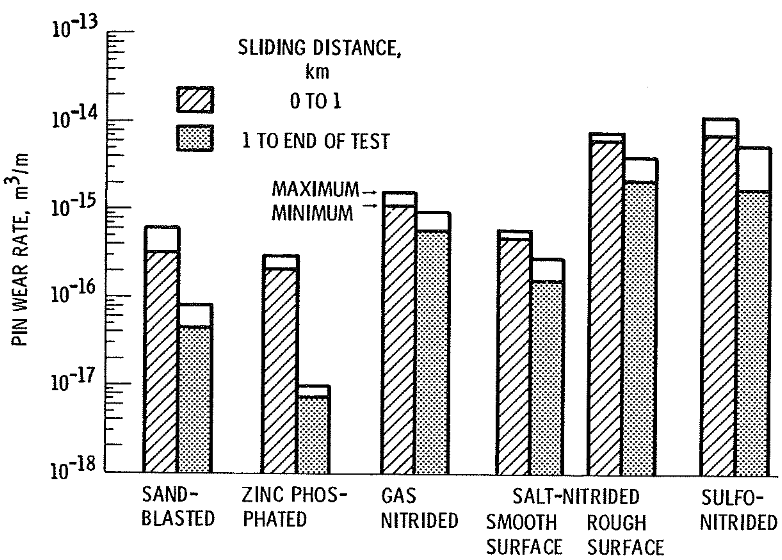

Fig. 5. - Pin wear rates for hemispherically tipped pins sliding against rubbed graphite films on pretreated steel disks.

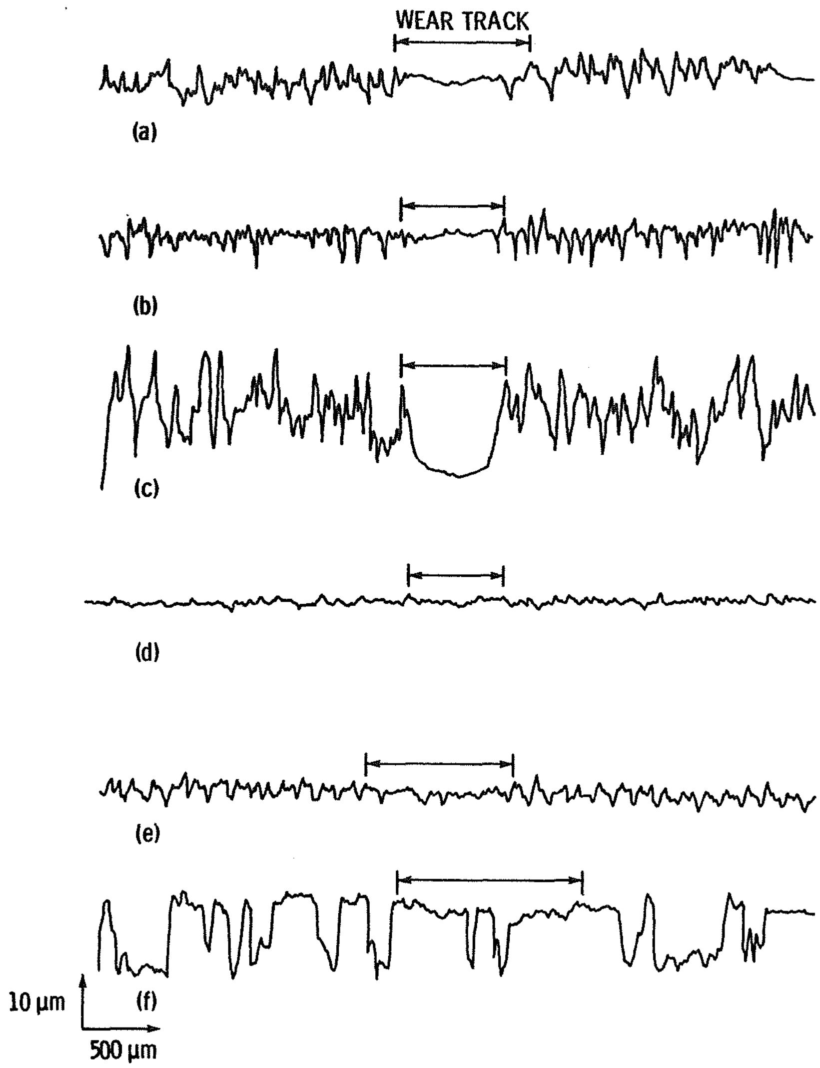

Fig. 6. - Surface profiles of wear tracks on graphite films rubbed onto pretreated steel disks. Pretreatment: (a) Sandblasted; (b) gas nitrided; (c) zinc phosphated; (d) salt nitrided, smooth surface; (e) salt nitrided, rough surface; (f) sulfo-nitrided.

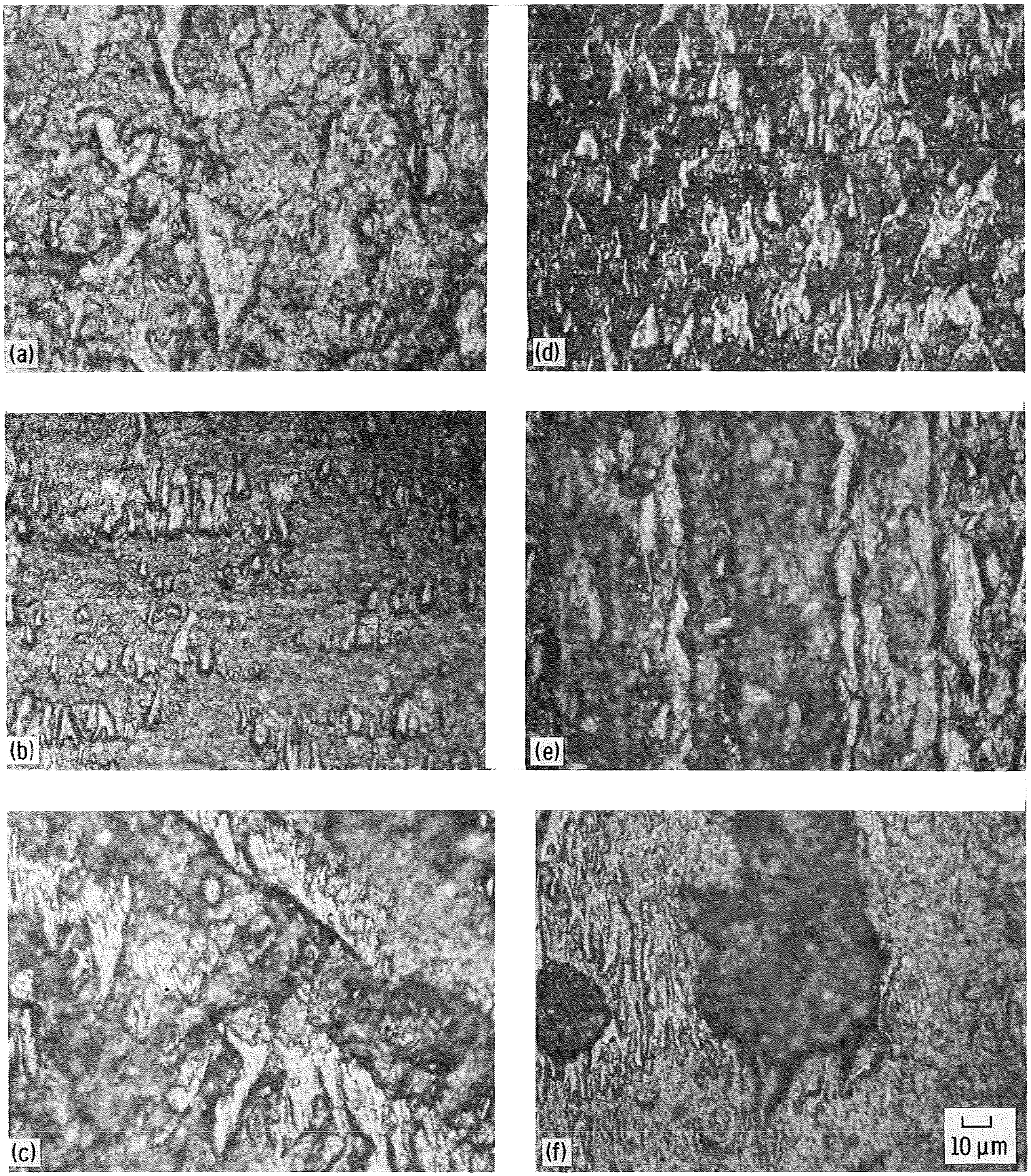

Figure 7. - Photomicrographs of graphite films rubbed onto pretreated steel disks. Pretreatment: (a) Sandblasted; (b) gas nitrided; (c) zinc phosphated; (d) salt nitrided, smooth surface; (e) salt nitrided, rough surface; (f) sulfo-nitrided.

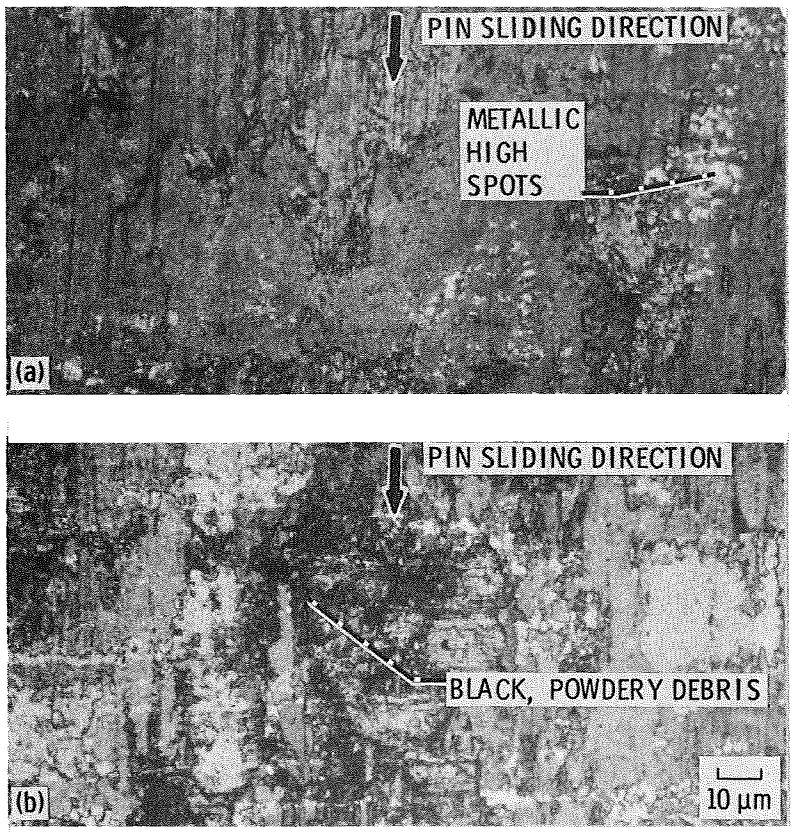

Figure 8. - Wear tracks of graphite films rubbed onto gas nitrided steel disks (a) after 6 kcycles of sliding and (b) after 78 kcycles of sliding (failure coefficient of friction, 0.25).

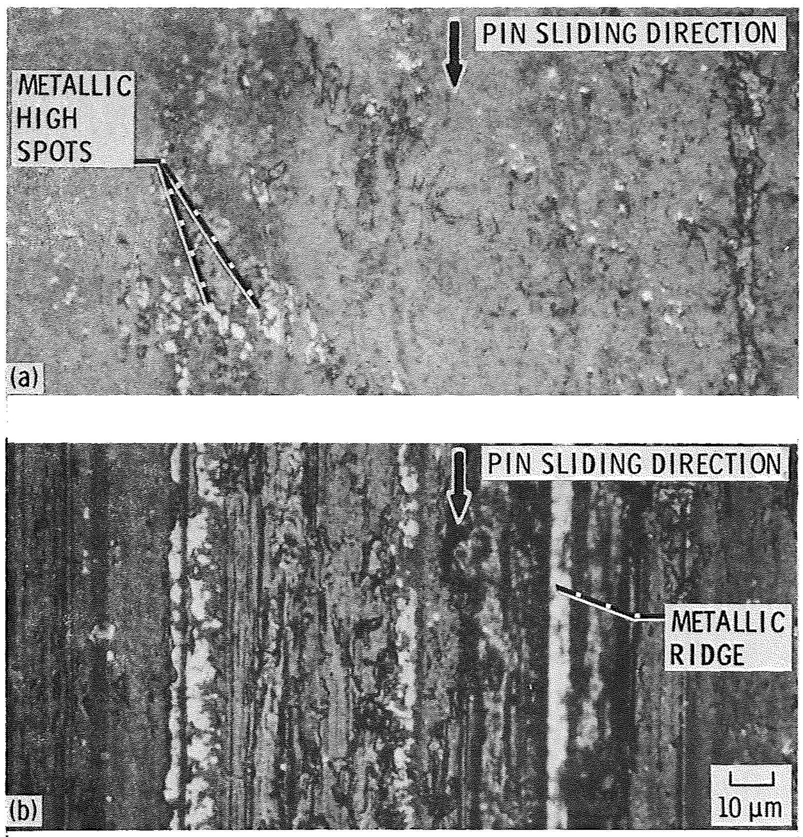

Figure 9. - Wear tracks of graphite films rubbed onto (a) smooth-surface salt nitrided and (b) rough-surface salt nitrided steel disks; after 6 kcycles of sliding.

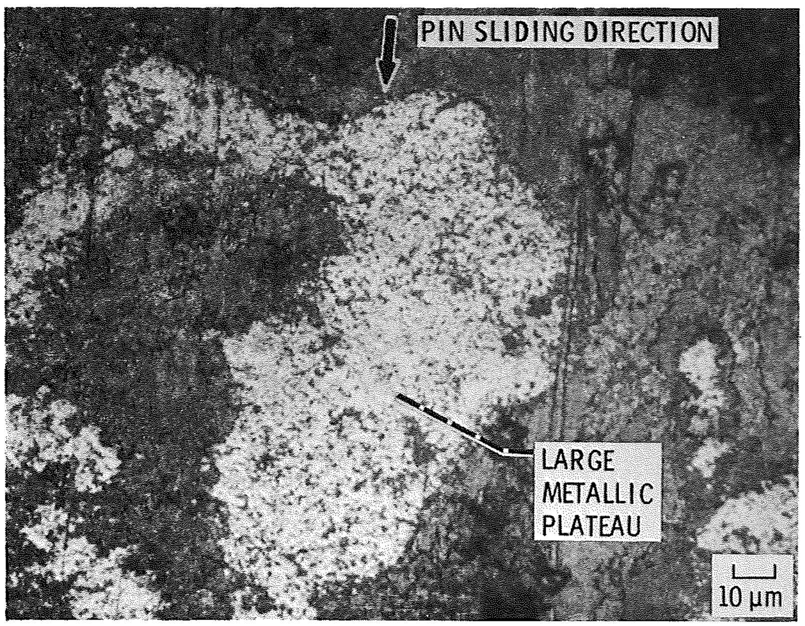

Figure 10. - Wear tracks of graphite films rubbed onto sulfo-nitrided steel disks after 6 kcycles of sliding.

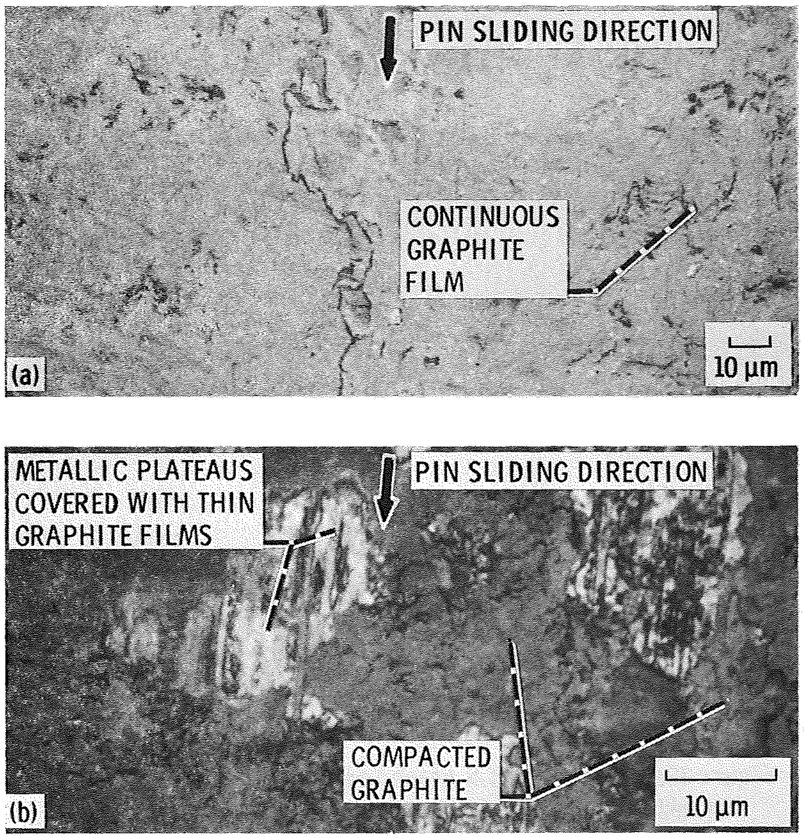

Figure 11. - Wear tracks of graphite films rubbed onto sandblasted steel disks after (a) 6 kcycles of sliding and (b) 90 kcycles of sliding.

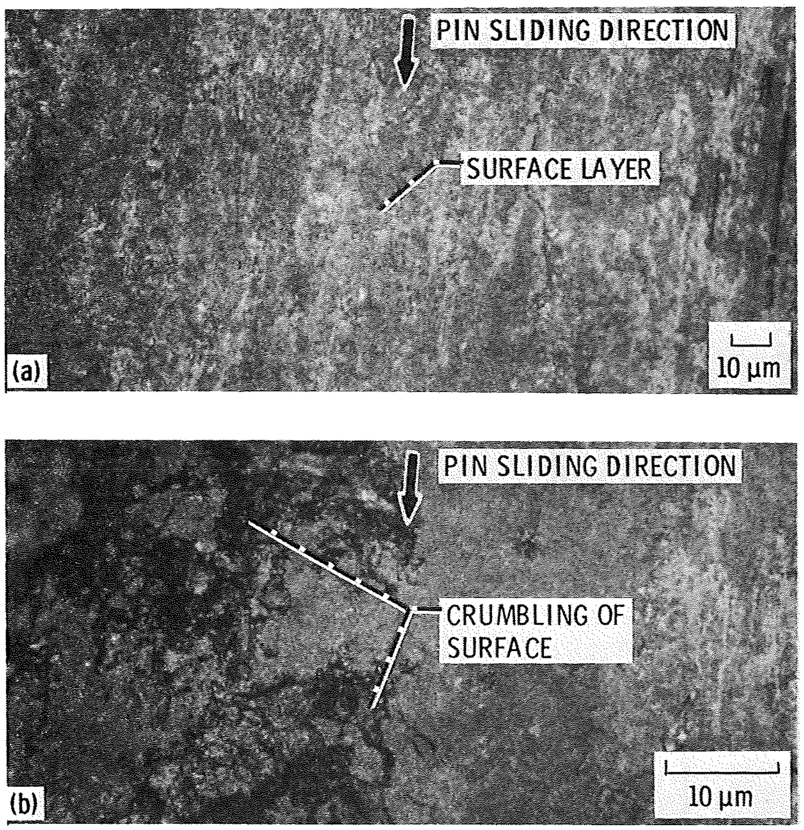

Figure 12. - Wear tracks of graphite films rubbed onto zinc phosphated steel disks after (a) 6 kcycles of sliding and (b) after 250 kcycles of sliding. The surface layer in part (a) appears to be a mixture of graphite and the zinc phosphated surface.

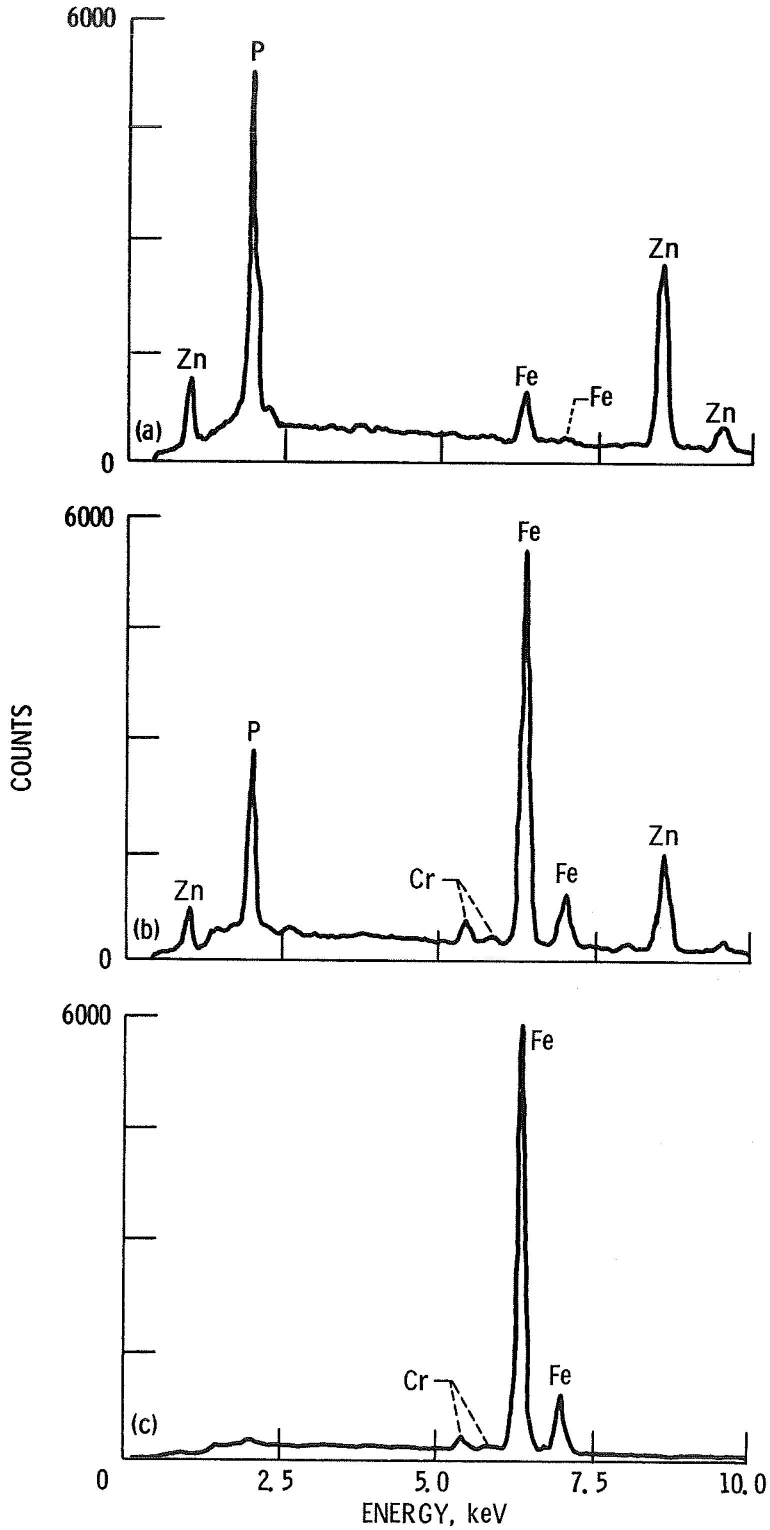

Fig. 13 - Energy dispersive x-ray spectra of wear tracks of graphite films rubbed onto zinc phosphated steel disks (a) before testing, (b) after 250 kcycles of sliding, and (c) after failure.

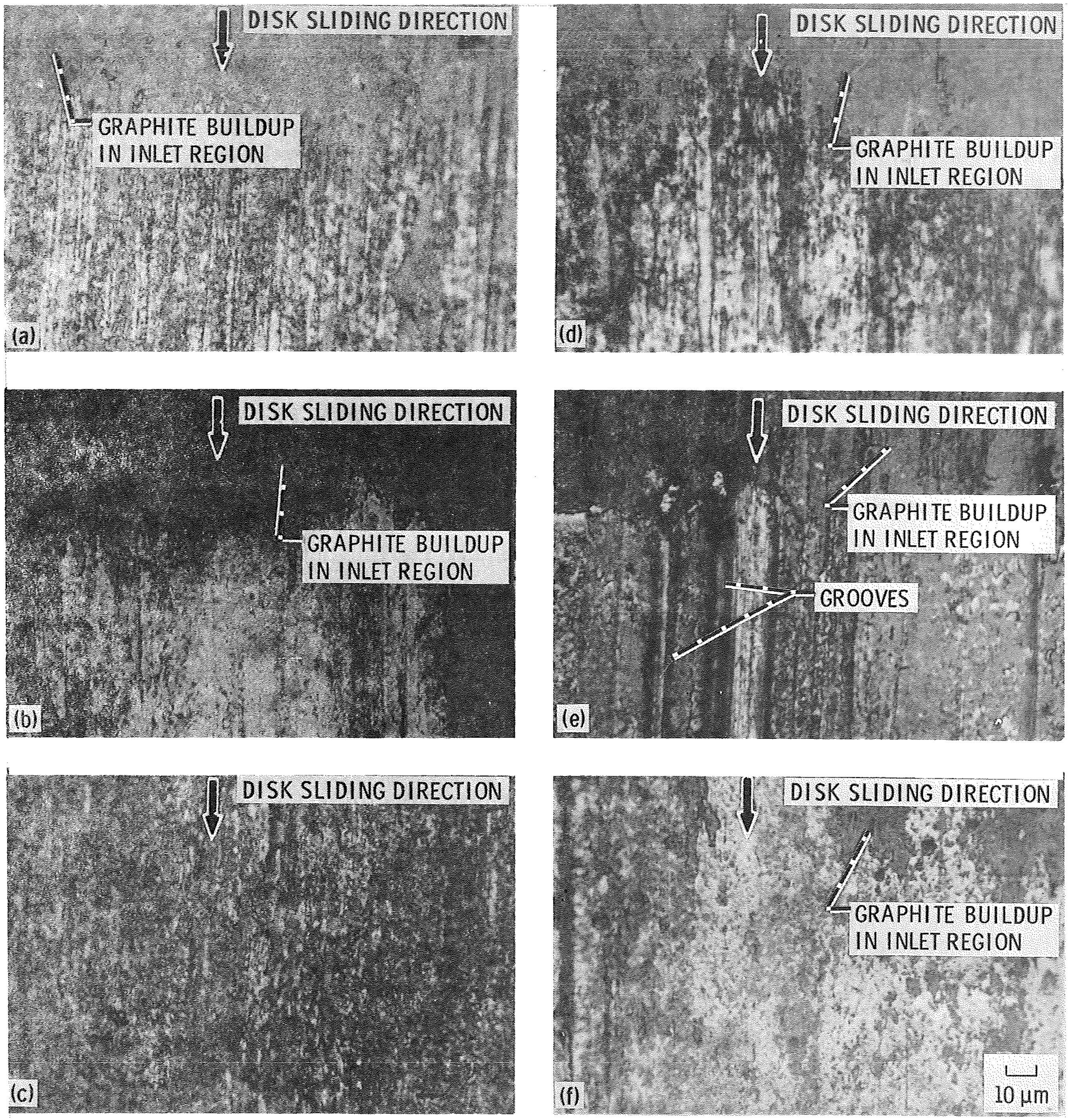

Figure 14. - Transfer films on pins after 1 km of sliding on graphite films rubbed onto pretreated steel disks. Pretreatments: (a) sandblasted; (b) gas nitrided; (c) zinc phosphated (Note thin transfer in middle of wear scar.); (d) salt nitrided, smooth surface; (e) salt nitrided, rough surface; (f) sulfo-nitrided (Note less continuous transfer in middle of wear scar.). Dark areas in photographs are graphite films, bright areas are metallic protrusions.

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16. Abstract

Rubbed films of natural flake Madagascar graphite were applied to ASTM A-355(D) steel with chemical surface pretreatments of zinc phosphate, gas nitride, salt nitride, sulfo-nitride, and with mechanical pretreatment (sandblasting). SAE 1045 steel pins were slid against these films using a pin-on-disk tribometer. The results indicate that two different lubricating mechanisms can occur. In the chemical surface pretreatment, the graphite can mix together to form a surface layer of the two constituents and this plastically flowing layer provides the lubrication The longest endurance lives and the lowest pin wear rates were obtained with this mechanism. In the other, surface topography appeared to control the mechanism. A rough surface was necessary to serve as a reservoir to supply the graphite to the flat metallic plateaus where it was sheared in very thin films between the plateaus and the sliding pin surface. For this mechanism, chemical pretreatment seemed to do little more than serve as a means for roughening the surface. Mean friction was not significantly influenced by chemical pretreatment, but surface roughness effects were observed.

17. Key Words (Suggested by Author(s))
Graphite; Solid lubricant; Surface pretreatment; Friction; Wear; Tribological properties; Lubricating mechanisms; Transfer films

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