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Effect of Substrate Surface Finish on the Lubrication and Failure Mechanisms of Molybdenum Disulfide Films

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EFFECT OF SUBSTRATE SURFACE FINISH ON THE LUBRICATION AND FAILURE MECHANISMS OF MOLYBDENUM DISULFIDE FILMS

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ABSTRACT

An optical microscope was used to study the lubrication and failure mechanisms of rubbed (burnished) MoS_2 films applied to three substrate surface finishes - polished, sanded, and sandblasted - as a function of sliding distance. The lubrication mechanisms was the plastic flow of thin films of MoS_2 between flat plateaus on the rider and on the metallic substrate. If the substrate were rough, flat plateaus were created during "run-in" and the MoS_2 flowed across them. Wear life was extended by increasing surface roughness since valleys in the roughened substrate served as reservoirs for MoS_2 and a deposit site for wear debris. In moist air, the failure mechanism was the transformation of metallic-colored MoS_2 films to a black, powdery material that was found by X-ray diffraction to consist primarily of α-iron and MoO_3 powders. In dry argon, the failure mechanism was the gradual depletion of the MoS_2 film from the contact region by transverse flow. Analysis of the wear debris on the wear track at failure showed it consisted mainly of α-iron and some residual MoS_2. No molybdenum oxides were found.

INTRODUCTION

Molybdenum disulfide (MoS_2) is one of the most widely used solid lubricants. A considerable amount of research has been conducted on its friction properties and the reasons for its good lubricating behavior. Johnson (1), Winer (2), and Farr (3) have written excellent reviews on the history, the uses, and the fundamental knowledge of MoS_2 as a lubricant.

Many studies have been conducted on the lubrication mechanisms of MoS_2 films. Most of these studies have been on a molecular level; that is, they have related the good lubricating properties of MoS_2 to its hexagonal crystal structure and covalent nature (2 and 3) or to its highly polarized surface that adheres well to metals (4). Many studies have also been conducted on the failure mechanisms of MoS_2 films. Failure has been related to the chemical degradation of MoS_2, with the most predominant chemical reaction in air being oxidation (5 to 12). It has also been proposed that water vapor accelerates the oxidation process (10).

Very few studies have been conducted on how MoS_2 films lubricate and fail from a microscopic point of view. In one of the few, Salomon, De Gee, and Zaat (8 and 9) have observed and recorded morphological changes of MoS_2 films photographically by stroboscopic illumination. They have observed that MoS_2 compacts and sinters on the wear track to form a highly reflective film and that oxidation of the MoS_2 leads to embrittlement, blistering, and scaling of the film. This investigation was conducted to extend those results by determining the effect of substrate surface finish and by looking at greater detail into the lubrication and failure mechanisms of MoS_2 rubbed (burnished) films.

The uniqueness of the study involved stopping the tests at present sliding intervals and examining the surfaces at various stages of film wear life with an optical microscope. Using this technique it was possible to observe one particular area on the film (or rider) at high magnifications and to see how the film (or transfer) varied with sliding distance. Thus, even though the contact area could not be directly observed during sliding, it was possible to make deductions as to the lubricating mechanisms involved from these static observations.

This study was made possible, because in recent years the quality of optical microscopes has increased tremendously. With a good optical microscope, a surface can now be observed (without oil emersion) to 2000x with very good clarity. In fact, for the purposes of this study, it was found that an optical microscope gave much better visual results than did a scanning electron microscope (SEM). Because the wear surfaces were extremely smooth and featureless, the optical microscope was especially advantageous because any deviation from this smoothness showed up vividly. In addition, thin solid lubricant films are often transparent and with a microscope equipped with a vertical illuminator, interference color bands can be observed. These bands indicate that coalescing, plastic flow and sintering have taken place, since extremely smooth, continuous surfaces are needed for this to occur. Interference bands can also be used to measure the thickness of solid lubricant films and transfer films.

A previous investigation by this author, using this technique, was conducted to compare the lubricating mechanisms of MoS_2 to those of graphite fluoride (13). In general, it was found that the lubrication mechanism of both appeared to be the formation of thin, coalesced solid lubricant films on each sliding surface and the very dynamic plastic flow of these films between the sliding surfaces. In air, the failure mechanism of MoS_2 films was found to be caused by the transformation of metallic-colored, coalesced MoS_2 films to a black, powdery material. Water in the air atmosphere appeared to accelerate this transformation rate. In argon, no transformation of MoS_2

was observed (with the microscope), but cracking and spalling of the coalesced MoS_2 film resulted in its gradual depletion.

This study was conducted to obtain a better understanding of how substrate surface finish can effect the lubricating and failure mechanisms of a MoS_2 film and in turn how these mechanisms affect wear life, friction coefficient, and rider wear rate. A pin-on-disk apparatus and 400C HT stainless steel specimens were used. To separate any effects caused by oxidation of the MoS_2 film, experiments were conducted both in moist air (10 000 ppm H_2O) and in dry argon (<20 ppm H_2O). The experimental conditions were a temperature of 25° C, a sliding speed of 2.6 meters per second (1000 rpm) and a load of 9.8 newtons (1 kg).

MATERIALS

Technical-Grade MoS_2 powder with an average particle size of 10 micrometers was used in this study. The size ranged from less than 1 micrometer to 75 micrometers; however, the large particles appeared to be conglomerations of small particles.

The riders and disks were made of 440C HT stainless steel with a Rockwell hardness of C-58 to C-60. The disks were lapped and polished to a surface finish of 0.09±0.02 micrometer CLA (centerline average). Some disks were then roughened either by sanding them in random directions with number-150-grit wet sandpaper to 0.30±0.05 micrometer (CLA) or by sandblasting them to 1.2±0.2 micrometers (CLA).

The MoS_2 powder was applied to the polished or roughened disk surfaces by mechanically rubbing it over the surface at constant load (see section PROCEDURE). The thickness of the films obtained was estimated optically and by surface profilometry to be about 1 to 2 micrometers above the highest metallic asperty on the substrate surface.

APPARATUS

A pin-on-disk sliding friction apparatus was used in this study. This apparatus is described in reference 14. Basically the friction specimens (fig. 1) were a flat disk (6.3-cm diameter) in sliding contact with a stationary hemispherically tipped rider (0.476-cm radius). The rider slid on a 5-centimeter-diameter track on the disk for a linear sliding speed of 2.6 meters per second at a disk rotation of 1000 rpm.

The apparatus used to apply the MoS_2 powder to the disks is shown in figure 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 MoS_2 applicators. In these experiments, the backs of polishing cloths were used as applicators. The rubbing load was applied by placing two 1-kilogram weights on top of the metal plate.

The application apparatus was designed to fit under the bell jar of a vacuum system. The atmosphere in which the films were applied could thus be controlled by evacuating the bell jar and backfilling it with the desired atmosphere. Previous results (15) have shown that films applied in moist air are thicker and more dense than films applied under dryer conditions. Thus the films used in these experiments were applied in moist air (10 000-ppm H_2O).

PROCEDURE

Surface Cleaning

The cleaning procedure was as follows:

(1) Scrub surfaces under running tap water with a brush to remove abrasive particles. (2) Wash surfaces with pure ethyl alcohol. (3) Rub surface with a water paste of levigated alumina. Clean until water readily wets

surface. (4) Rinse under running tap water to remove levigated alumina. (Use brush to facilitate removal.) (5) Rinse in distilled water. (6) Dry surface with dry compressed air, since surfaces not dried quickly tend to oxidize.

Film Application

The procedure for applying the rubbed films was as follows:

(1) Apply a small amount of MoS_2 powder to the cleaned disk surface and spread it evenly over the surface with the back of a polishing cloth. (2) Apply approximately 1 gram of MoS_2 powder to the contact zone of the applicator (back of a polishing cloth attached to the floating metal plate) and distribute it evenly. (3) Assemble apparatus as shown in figure 2 and apply two 1-kilogram weights as the applied load. (4) Evacuate the bell jar to 1×10^3 N/m^2 and backfill it with moist air (10 000-ppm H_2O) to atmospheric pressure. Continue to purge the bell jar with moist air until application is complete. (5) Set disk into rotation, gradually increase the speed to 15 rpm, and rub for 1 hour. (6) Remove disk from apparatus and blow off loose MoS_2 debris from the surface with dry compressed air.

Friction and Wear Tests

A rider and disk (with applied MoS_2 film) were inserted into the friction and wear apparatus, and the test chamber was sealed. Moist air (10 000-ppm H_2O) or dry argon (<20-ppm H_2O) were purged through the chamber for 15 minutes before the tests; this purge was continued throughout the tests. Moist air was used as a control atmosphere since it is typical of average atmospheric conditions (approximately 50 percent relative humidity). Dry argon was used since it is inert and nonoxidizing.

The flow rate was 1500 cubic centimeters per minutes, and the volume of the chamber was 2000 cubic centimeters. After the 15-minute purge, the disk was set into rotation at 1000 rpm and a 1-kilogram load was gradually applied. The test temperature was 25° C.

Each test was stopped after 1 kilocycle (1 minute) of sliding. After the rider and disk were removed from the friction apparatus, the contact areas were examined by optical microscopy and photographed. Surface profiles of the film wear tracks were also taken. The rider and disk were then placed back into the apparatus, and the test procedure was repeated. The rider was not removed from the holder, and locating-pins in the apparatus insured that it was returned to its original position.

Each test was stopped and the test procedure repeated after sliding intervals of 1, 5, 15, 30, 60, 100, 200, 400, 700, 1500, 2700, and 3700 kilocycles or when failure occured. The failure criterion in this study was a friction coefficient of 0.30; thus wear life is defined as the number of kilocycles of sliding to reach a friction coefficient of 0.30. Rider wear was determined by measuring the diameter of the wear scar on the hemisperically tipped rider and then calculating the volume of material worn away.

Analysis of Sliding Surfaces

Optical microscopy techniques were used to study the lubricating films, the transfer films, and the wear particles. The surfaces were viewed at magnifications to 1100. At these high magnifications, the vertical resolution was low (~1 μm); this aspect was used to measure the heights of various features on the sliding surfaces, such as film thickness and wear track depth.

With vertical illumination of the surfaces, interference fringes could be seen in the films, both on the disk wear track and on the rider wear scar. Interference fringes indicated that the MoS_2 particles had flowed together to form a continuous film that was very smooth and that the film was being sheared thinner and thinner in some areas. For example, the gradual depletion of the fringes in the inlet area of the rider wear scar indicated that the film thickness was less than the wavelength of light (0.4 μm).

The powdery debris that remained on the disk wear tracks at failure was scraped off and mounted on a quartz fiber with apiezon. An X-ray diffraction pattern was then taken, by using the Debye-Scherrer method, to determine the composition.

RESULTS AND DISCUSSION

Moist-Air Results

The MoS_2 powder was rubbed onto 440C HT steel disks with three surface finishes: lapped and polished, sanded, and sandblasted. Figure 3 gives photomicrographs and surface profiles of the three substrate surfaces before the MoS_2 films were applied. Figure 4 shows photomicrographs and surface profiles after the MoS_2 films were applied to these surfaces.

The effect of substrate surface finish on friction coefficient and wear life in a moist-air test atmosphere (10 000-ppm H_2O) is shown in figure 5. The gaps in the traces represent the intervals when the tests were stopped so that wear measurements could be made and the sliding surfaces could be observed with an optical microscope. The sandblasted surface gave a lower friction coefficient for a longer time than did either the sanded surface or the polished surface.

The failure criterion for solid-lubricant films is usually arbitrary. Most often, failure is assumed as the point when the friction coefficient reaches some predetermined value or rapidly increases in value. In these experiments, failure was deemed to occur when the friction coefficient reached 0.30. This occurred for the polished surface after 3½ kilocycles of sliding, for the sanded surface after 5½ kilocycles, and for the sanblasted surface after 60 kilocycles. Sliding was continued past this point to determine how friction and rider wear were affected. The friction coefficient increased beyond 0.30 and then dropped below 0.30 (fig. 5).

At the preset sliding intervals, the tests were stopped, the rider and disk specimens were removed from the apparatus, the rubbing surfaces were examined, and the wear to the riders was calculated. Table 1 gives rider wear rates for each sliding interval. The rougher the substrate to which the MoS_2 was applied, the greater the initial wear to the rider. After 1 kilocycle of sliding on MoS_2 films applied to polished, sanded, and sandbalsted substrates the rider wear rates were 1.8×10^{-15}, 2.0×10^{-15}, and 8.8×10^{-15}, respectively.

Figure 6, which gives photomicrographs of rider wear scars and MoS_2 film wear tracks after 1 kilocycle of sliding, illustrates the difference in rider wear and transfer. Figure 7 gives high-magnification photomicrographs of the central area of the MoS_2 film wear tracks.

As shown in figures 6 and 7, MoS_2 did not produce a continuous film on the disk wear track, regardless of the substrate surface finish to which the MoS_2 was applied. On the polished substrate surface, individual platelets of MoS_2 formed and tended to coalesce, however a continuous film of MoS_2 was not produced on the wear track (fig. 7(a)). Excess MoS_2 was either displaced to the sides of the wear track or coalesced to form clumps (fig. 6(a)). The MoS_2 tended to fill up the scratches on the wear track of the sanded substrate surface (fig. 7(b)), and the area between the scratches was either void of MoS_2 or covered with a film too thin to observe. The amount of MoS_2 on the wear track of the sandblasted substrate surface appeared to be greater than for the other two surfaces; however, the film still did not cover the wear track completely. "Flats" on the metallic asperities can be seen in the wear track (fig. 7(c)). These flat plateaus were formed during the initial run-in phase of sliding by the interaction between the sharp sandblasted asperities on the substrate and the rider. This interaction was the most likely reason for the greater initial rider wear on the sandblasted substrate surface. Once flats had formed on the sandblasted asperities (sliding time greater than 1 kilocycle), rider wear was less for the sandblasted substrate surface than for the other two surfaces (table 1). Friction was lower because the valleys in the sandblasted substrate surface served as reservoirs for the MoS_2 and restricted its transverse flow from the contact zone.

With increasing sliding time, a feature common to all wear tracks was a distinct change in the appearance of the MoS_2 film. Initially, the MoS_2 film on the wear track was a bright, metallic-colored, coalesced film. As sliding progressed, the MoS_2 film on the wear tracks transformed to a blackish or darkish colored material (fig. 8(a)). The rate of this transformation appeared to be related to the roughness of the substrate surface. The rougher the surface, the longer it took for complete transformation to occur. The reason for this is that the valleys in the rough surfaces served as reservoirs for MoS_2 and deposit sites for wear particles or transformed material.

At failure, the film wear tracks on all three surface substrates looked much the same. There was a thick, powdery buildup on all three surfaces. Figure 8(b) shows the buildup on the sandblasted substrate, figure 9(a) shows the buildup on the polished substrate and figure 9(b) shows the buildup on the sanded substrate. Some of this powdery material from the sanded substrate was removed and analyzed by X-ray diffraction. No MoS_2 lines were found. The powdery material consisted mostly of α-iron and MoO_3. A few weak $FeMoO_3$ lines were found, but they were too weak for good analysis.

Heavy, coalesced transfer films of MoS_2 were found after 1 kilocycle on the riders that slid on the films applied to the polished and the sanded substrate surfaces (figs. 6(a), 6(b), and 10(a)); but the transfer was not nearly as thick on the riders that slid on films applied to the sandblasted substrate surface (fig. 6(c)). This was probably due to the scouring action of the sharp metallic asperities of the sandblasted surface. This thin transfer did not increase the friction coefficient; in fact a slightly lower friction coefficient was obtained (fig. 5).

High mangnification photomicrographs of the transfer in the entrance region of the rider which slid on the MoS_2 film applied to the sanded substrate is shown in figure 10 after (a) 1 kilocycle and (b) 10 kilocycles of sliding. After 1 kilocycle of sliding a large buildup of MoS_2 is seen in the entrance region of the scar. The MoS_2 is powdery at the leading edge, but it becomes highly compressed towards the contact region. In fact, in the contact region, the MoS_2 particles coalesce and it is impossible to distinguish individual particles. The coalesced MoS_2 film tends to plastically flow across the contact area of the rider and then break up into fine powdery material in the exit region. As was found on the substrate wear tracks, the MoS_2 had a bright, metallic looking appearance (fig. 10(a)).

As sliding time elapsed the metallic appearance of the MoS_2 transfer films gradually disappeared, and at failure the transfer appeared to be transformed into a more powdery, less coalesced, blackish colored film (fig. 10(b)). Thus at failure, the film wear tracks and the rider wear scars looked very similar. This transformed material did not completely cover the wear surfaces, however; it formed bands around the circumference of the disk wear track and a corresponding band on the rider.

Each test was continued past the arbitrarily chosen failure point - a friction coefficient of 0.30. In each case both the friction coefficient (fig. 5) and the rider wear rate (table I) decreased. Thus either the transformed MoS_2 was providing lubrication, or MoS_2 at the sides of the wear track or deeper in the valleys (of the rough surfaces) had found its way into the contact region. The small reductions in friction coefficients and rider wear rate for the MoS_2 film applied to the polished surface suggest that the main contribution probably came from exposure of new MoS_2 (from deeper in the valleys of the rough surfaces).

Dry-Argon Results

In a previous investigation (13), it was shown that the transformation of MoS_2 on the sliding surfaces did not occur in dry argon (<20-ppm H_2O). Thus, a series of experiments was also conducted in dry argon to determine the effect of substrate surface roughness on the lubricating properties of MoS_2 films when transformation of MoS_2 was not the principal mechanism of failure. Friction traces for these tests (fig. 11) illustrate that the friction coefficient was not affected by the roughness of the substrate - the value being slightly less than 0.02 for the first 700 cycles. Wear life, however, was affected, just as it was in moist air; that is, the rougher the surface, the longer the wear life. Even on the polished substrate, wear life was much greater in dry argon than in moist air. The wear lives obtained were 950 kilocycles for the polished substrate, 1860 kilocycles for the sanded substrate, and 4450 kilocycles for the sandblasted substrate.

Table II gives rider wear rates in dry argon for each sliding interval. As in moist air, the rougher the substrate, the greater the initial rider wear. However, the wear rate was lower in dry argon. The values obtained after 1 kilocycle of sliding were 0.10×10^{-15} m^3/m for the polished substrate,

0.30×10^{-15} m^3/m for the sanded substrate, and 4.5×10^{-15} m^3/m for the sandblasted substrate. After run-in, the rates dropped considerably, with the polished surface giving the lowest rate (0.002×10^{-15} m^3/m). The sandblasted substrate gave a low rate of 0.003×10^{-15} m^3/m, and the sanded substrate gave a low rate of 0.008×10^{-15} m^3/m.

The wear and transfer to the riders after 1 kilocycle of sliding in dry argon can be compared for the three surface finishes in figure 12. As in moist air, the smoother the substrate surface, the greater the transfer to the rider and the less the wear. Even though transfer and rider wear were different at this point, the friction coefficients were the same. After 15 kilocycles of sliding, the transfer films for riders on the polished and sanded substrates became like those for riders on the sandblasted substrate after 1 kilocycle of sliding (fig. 12(c)). That is, very thin films of MoS_2 were found on the rider scar.

Figure 13 gives high-magnification photomicrographs of the MoS_2 film wear tracks after 1 kilocycle of sliding in dry argon. On the polished and sandblasted substrates the MoS_2 film was not continuous; however, on the sanded substrate a more continuous film was produced. Because the longest lives were obtained on the sandblasted substrate, film continuity was not the most important factor in determining wear life.

In a previous investigation (13), it was found that for MoS_2 films applied to sanded substrates, the failure mechanism was the cracking and spalling of the continuous, metallic-colored MoS_2 films. This, coupled with the tendency of the MoS_2 to flow transversely outward on the film wear track, gradually depleted the films. For films applied to polished substrates, very little cracking or spalling was observed. The failure mechanism seemed to be solely the depletion of the coalesced MoS_2 films by transverse flow. Figure 14 gives high-magnification photomicrographs of the wear tracks on the MoS_2 films applied to a polished substrate after 60, 700, and 940 kilocycles of sliding. The thinning and gradual depletion of the films can be seen by comparing the surfaces at 60 and 700 kilocycles of sliding. When the films became too thin, metal-to-metal contact occurred and fine, powdery metallic debris was produced. If only a small amount of debris was produced, the films could reheal and only a spike in the friction coefficient trace resulted (fig. 11). However, at some point, the production of fine metallic debris became too great, causing the friction to increase and bands of powdery material to be created around the wear track. Figure 14(c) shows this powdery buildup on the wear track at failure.

On the sandblasted substrate, the MoS_2 film on the wear track tended to buildup around the highest metallic asperities. Figure 13(c) shows a typical region on the track. Smooth, compacted MoS_2 can be seen along with flat, metallic asperity plateaus. As in moist air, the interaction of the sharp, sandblasted asperities (during run-in) with the rider was the most likely reason for the greater initial rider wear when sliding on the sandblasted substrate. After run-in the lubrication mechanism was the plastic flow of the MoS_2 films between the flat asperity plateaus of the substrate and the flat rider scar. As sliding progressed, the MoS_2 in the contact region was gradually depleted, causing the metallic asperity plateaus to wear and thereby exposing new lubricant deeper in the valleys.

Figure 15 shows a high-magnification photomicrograph that illustrates the lubrication mechanism of MoS_2 films applied to sandblasted substrates. The photomicrograph, taken after 1700 kilocycles of sliding, shows a flat, metallic plateau with thin films of MoS_2 flowing over it. The MoS_2 is supplied from the valleys, becomes compressed in the entrance region of the plateau, and eventually coalesces into a very thin film as it flows across the plateau and then is deposited in a following valley. The same process occurs on the rider, which in effect is also a flat, metallic plateau - but much larger. The MoS_2 builds up in the entrance region of the rider scar, compresses, and eventually coalesces as it flows into and across the rider scar and is deposited in the exit region (fig. 12).

At failure, the film wear tracks on the sandblasted substrate and the rider wear scars looked much the same as they did for the other two substrates. That is, there was a heavy buildup of powdery material on both surfaces. The powdery material was scraped off the disk wear track and analyzed by X-ray diffraction. No molybdenum oxides were found for any test conducted in dry argon, and the powdery material was mostly α-iron. Also found were a few weak MoS_2 lines and a few FeS lines, although the FeS lines were too weak for good analysis. Thus, in dry argon, failure was deemed to be caused by the gradual depletion of MoS_2 and the consequent excessive production of very fine metallic wear debris.

CONCLUDING REMARKS

A theory for MoS_2 lubrication and failure mechanisms has been presented in this paper which is consistent with the observations of many other investigators. The theory, in essence is similar to that concluded by De Gee, Salomon and Zaat (9), except that the presence of oxygen was not found to promote sintering under the high stresses used in these experiments. However, it was found under the low stresses (as those used in applying the films), that oxygen and moisture in the atmosphere did promote sintering and the production of thicker, denser films. Johnston and Moore (15) also found this to be true.

The lubricating mechanism proposed in this paper and reference 13 also extends the results of De Gee et al. (9) by proposing that the lubricating mechanism is more than just a simple sintering together of the MoS_2 particles. The lubrication process was found to be very dynamic and appears to be due to the plastic flow of individual MoS_2 particles. The particles are capable of coalescing together to form continuous films on the contact areas (fig. 13(b)). However, the photomicrographs of figures 7, 13, 14, and 15 indicate that thick, continuous films are not a necessary criterion for good lubrication. What this study indicates is that a constant flow of MoS_2 is taking place between the sliding surfaces and that this flow is providing the lubrication. When observing the same spot on the substrate or on the rider contact area (during the intermittant testing), it was observed that the appearance of the film was constantly changing. On the polished substrate, the position and size of the MoS_2 platelets varied (fig. 14); and on the rough surfaces, the flow patterns on the asperity flat plateaus (fig. 15) were always different at the end of each sliding interval.

The lubricating mechanisms described in this paper are very similar to those observed for rubbed graphite fluoride films (16) and to those observed for polyimide-bonded graphite fluoride films under high stresses (17). Thus, in general, it appears that the the propensity of individual particles to coalesce together into a plastically flowing film is a prime prerequisite for a good solid lubricant material.

Roughness of the substrate is important in that it affords a reservoir for the solid lubricant; but it has a negative aspect in that during "run-in," rougher substrates tended to increase rider wear. After "run-in," rider wear and friction were minimally affected by substrate roughness (especially in an inert atmosphere), but wear life was increased considerably by the reservoir capability.

The flow properties of MoS_2 in moist air and dry argon did not appear to be significantly different after very short sliding distances (<1 kc). However, as others have also found, repeated passes over the film in air, caused the MoS_2 to chemically decompose (primarily to MoO_3), with the result that the color and flow properties also changed. The substrate surface became covered with a more powdery type of material that sintered together but did not possess the coalescing, flowing type of behavior that MoS_2 possessed in dry argon.

SUMMARY OF RESULTS

Friction, wear, and optical microscopy studies of molybdenum disulfide (MoS_2) rubbed films applied to 440C HT steel with different substrate surface finishes and then evaluated in moist air (10 000-ppm H_2O) or dry argon (<20-ppm H_2O) gave the following results:

1. The lubricating mechanism consisted of the coalescing and plastic flow of thin films of MoS_2 between flat plateaus on the rider and on the metallic substrate. If the substrate were rough, flat plateaus were created during run-in. The MoS_2 tended to flow across these flat plateaus.

2. Failure in moist air was due to the transformation of metallic-colored, coalesced films of MoS_2 to a black, powdery material that was found by X-ray diffraction to consist mainly of α-iron and MoO_3.

3. Failure in dry argon was caused by the gradual depletion of MoS_2 by lateral flow from the contact region and the consequent production of very fine powdery debris that was found by X-ray diffraction to be mostly α-iron. A very small amount of residual MoS_2 was also found in the powder, but no molybdenum oxides were found.

4. Wear life was about two orders of magnitude greater in dry argon than in moist air because of the difference in failure mechanisms. Increasing

the substrate surface roughness tended to extend wear life (both in moist air and in dry argon) by providing reservoirs for the MoS_2.

5. The friction coefficient was not affected by substrate surface roughness in dry argon; however, in moist air, rougher substrate surfaces gave lower friction coefficients than smoother substrate surfaces.

6. Rougher substrates caused higher wear rates during run-in in both moist air and dry argon. After run-in, rider wear rates in dry argon were not greatly affected by substrate roughness; however, in moist air, the rougher the substrate, the lower the rider wear rates.

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TABLE I. - RIDER WEAR RATES IN A MOIST-AIR ATMOSPHERE (10 000-ppm H_2O)

[Experimental conditions: rubbed MoS_2 films; 440C HT steel riders and disks; temperature, 25° C; load, 1 kg; speed, 2.6 m/sec (1000 rpm).]

Sliding interval, kilocycles	Sandblasted substrate	Sanded substrate	Polished substrate
	Surface finish, μm		
	1.2±0.2	0.30±0.15	0.09±0.02
	Rider wear rate, m^3/m		
0 - 1	8.80×10^{-15}	2.00×10^{-15}	1.8×10^{-15}
1 - 5	.64	.55	[a]3.6
5 - 10	----------	[a]2.40	2.8
5 - 15	.07	----------	
10 - 30		.49	---------
15 - 60	.11		
60 - 70	[a]1.60	----------	---------
70 - 100	.22	----------	---------

[a] Failure (friction coefficient of 0.30).

TABLE II. - RIDER WEAR RATES IN A DRY-ARGON ATMOSPHERE (<20-ppm H_2O)

[Experimental conditions: rubbed MoS_2 films; 440C HT steel riders and disks; temperature, 25° C; load, 1 kg; speed, 2.6 m/sec (1000 rpm).]

Sliding interval, kilocycles	Sandblasted substrate	Sanded substrate	Polished substrate
	Surface finish, μm		
	1.2±0.2	0.30±0.15	0.09±0.02
	Rider wear rate, m^3/m		
0 - 1	4.500×10^{-15}	0.490×10^{-15}	0.100×10^{-15}
1 - 15	.048	.062	.038
15 - 60	.013	.019	.018
60 - 200	.009	.012	.004
200 - 400	.003	.012	.002
400 - 700	.006	.008	.002
700 - 940	-----------	-----------	[a].010
700 - 1500	.010	.011	-----------
1500 - 1860	-----------	[a].018	-----------
1500 - 2700	.012	-----------	-----------
2700 - 3700	.009	-----------	-----------
3700 - 4450	[a].046	-----------	-----------

[a] Failure (friction coefficient of 0.30).

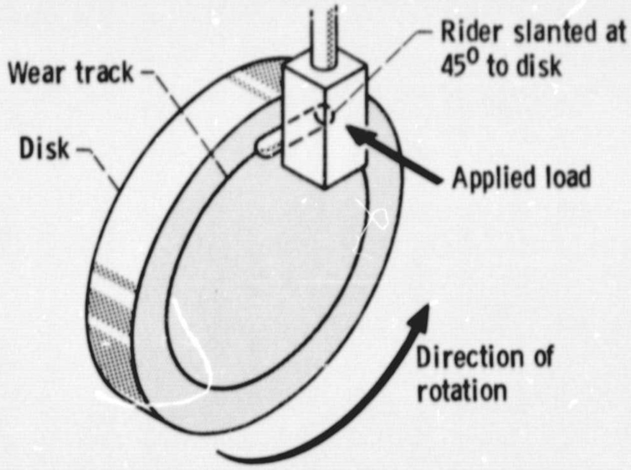


Figure 1. - Schematic diagram of friction specimens.

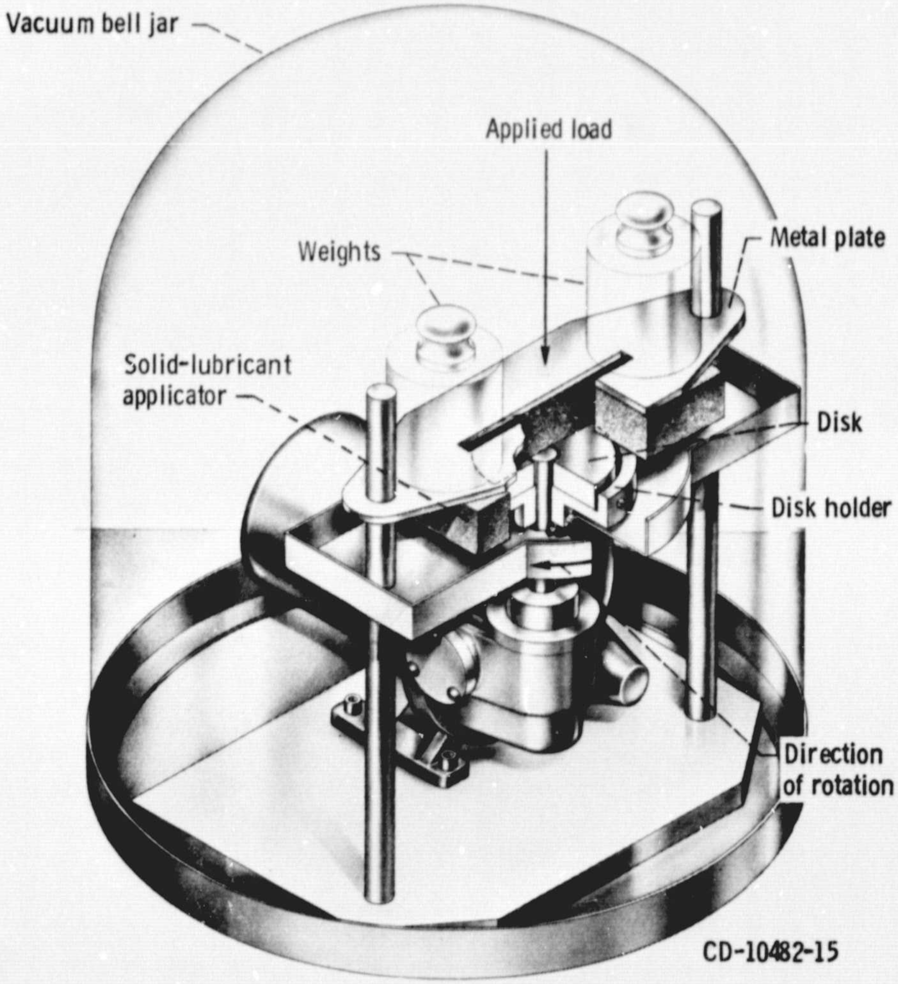
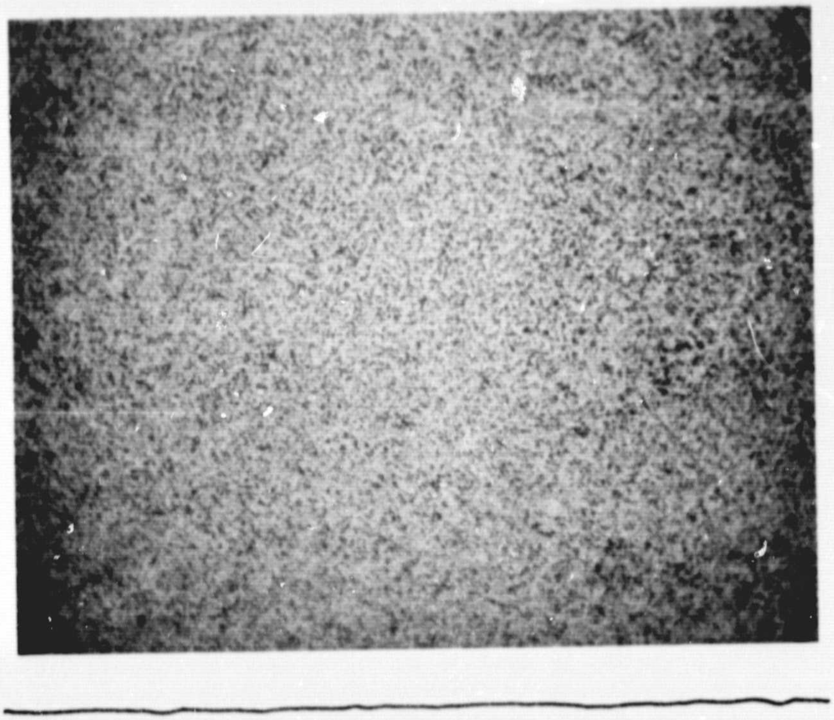
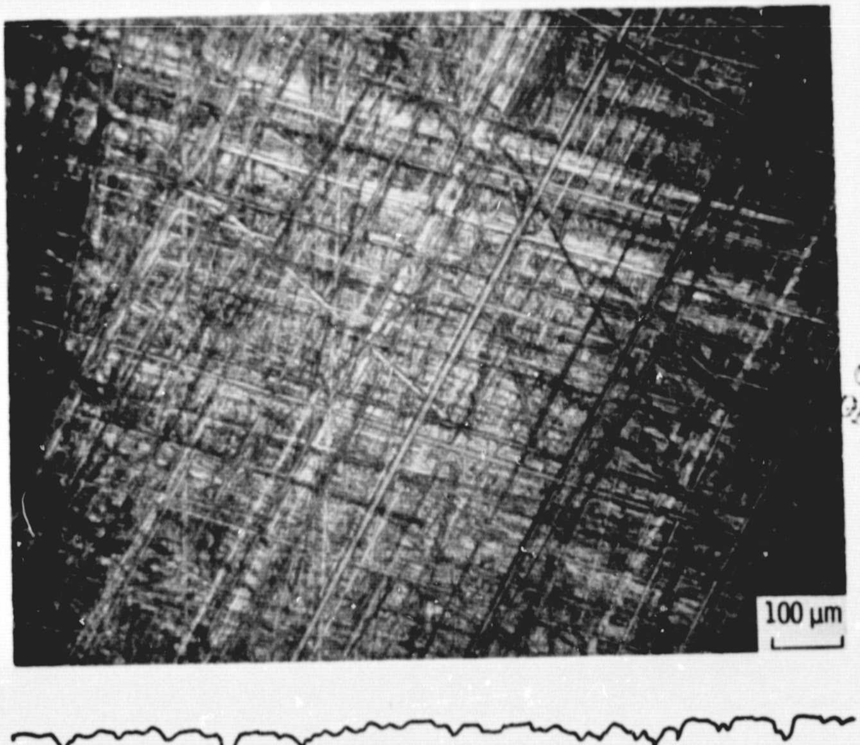


Figure 2. - Apparatus used to apply MoS_2 films.

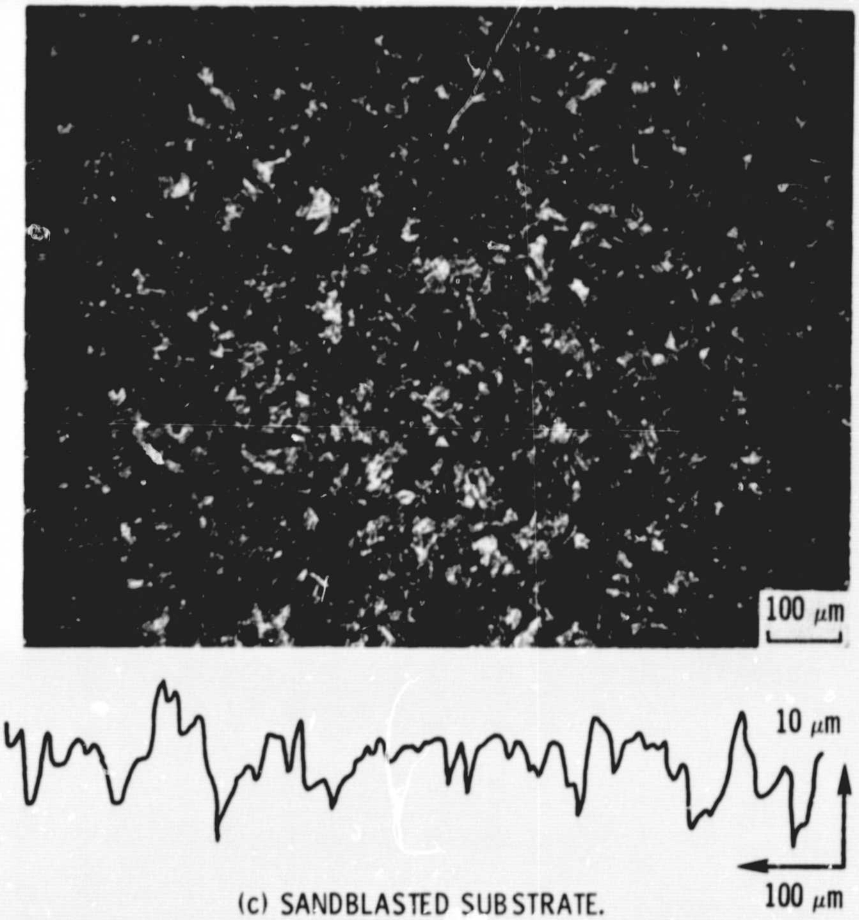


(a) POLISHED SUBSTRATE.



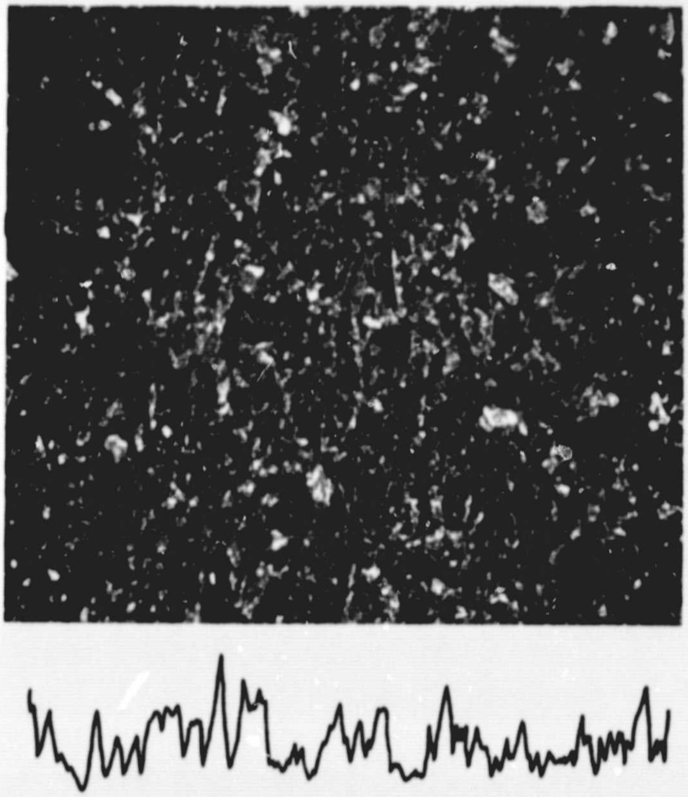
(b) SANDED SUBSTRATE.

Figure 3. - Photomicrographs and surface profiles of 440C HT stainless-steel disks with different surface finishes before application of rubbed MoS_2 films.

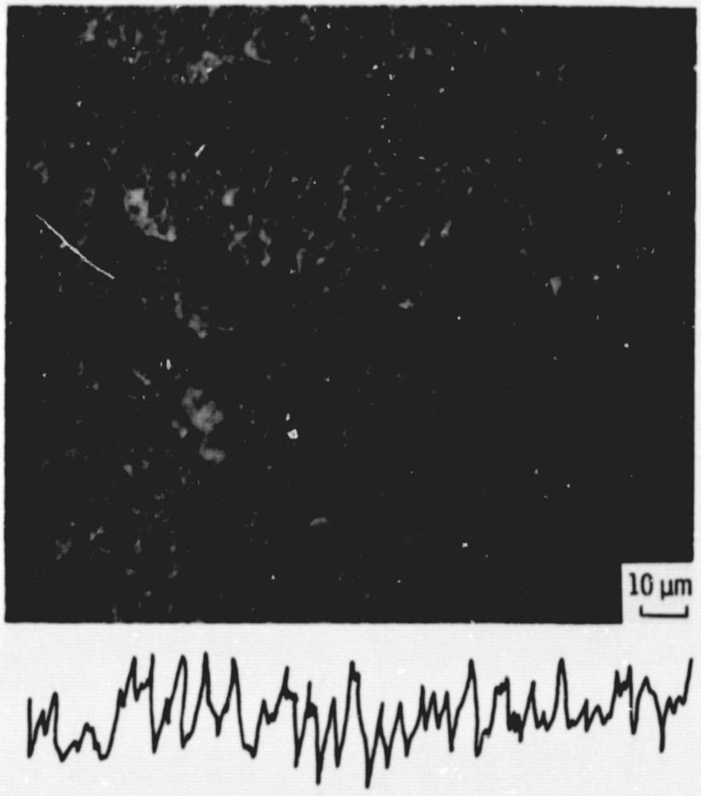


(c) SANDBLASTED SUBSTRATE.

Figure 3. - Concluded.

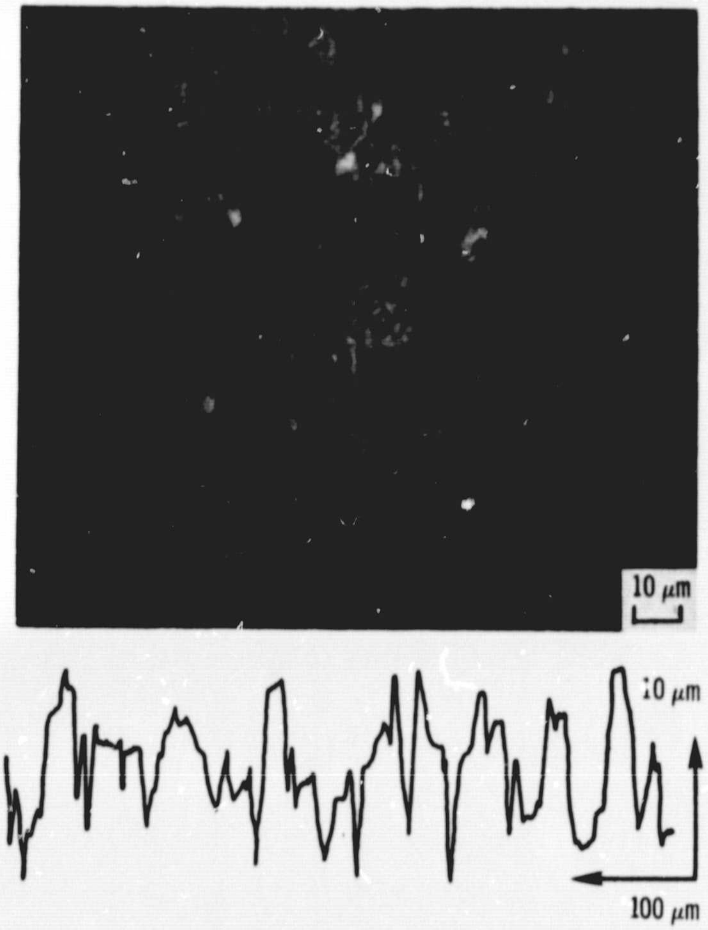


(a) POLISHED SUBSTRATE WITH MoS_2 FILM.



(b) SANDED SUBSTRATE WITH MoS_2 FILM.

Figure 4. - Photomicrographs and surface profiles of 440C HT stainless-steel disks with different surface finishes after application of rubbed MoS_2 films.



(c) SANDBLASTED SUBSTRATE WITH MoS_2 FILM.

Figure 4. - Concluded.

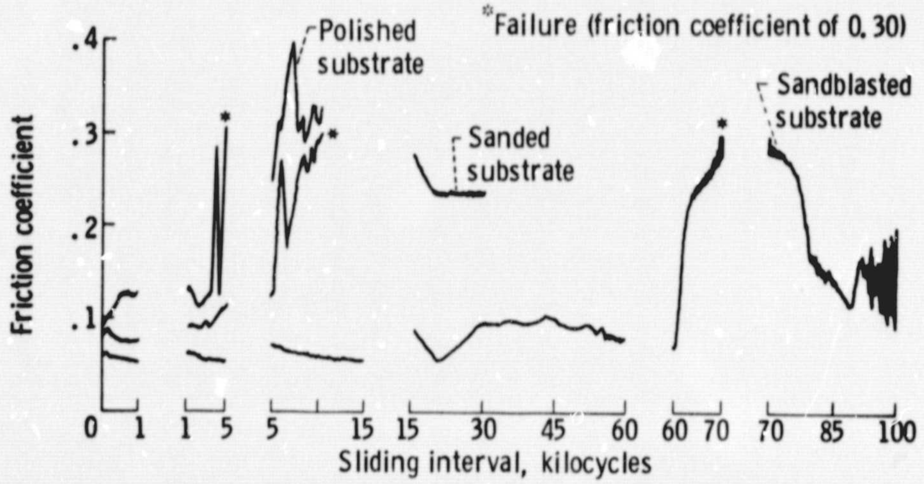
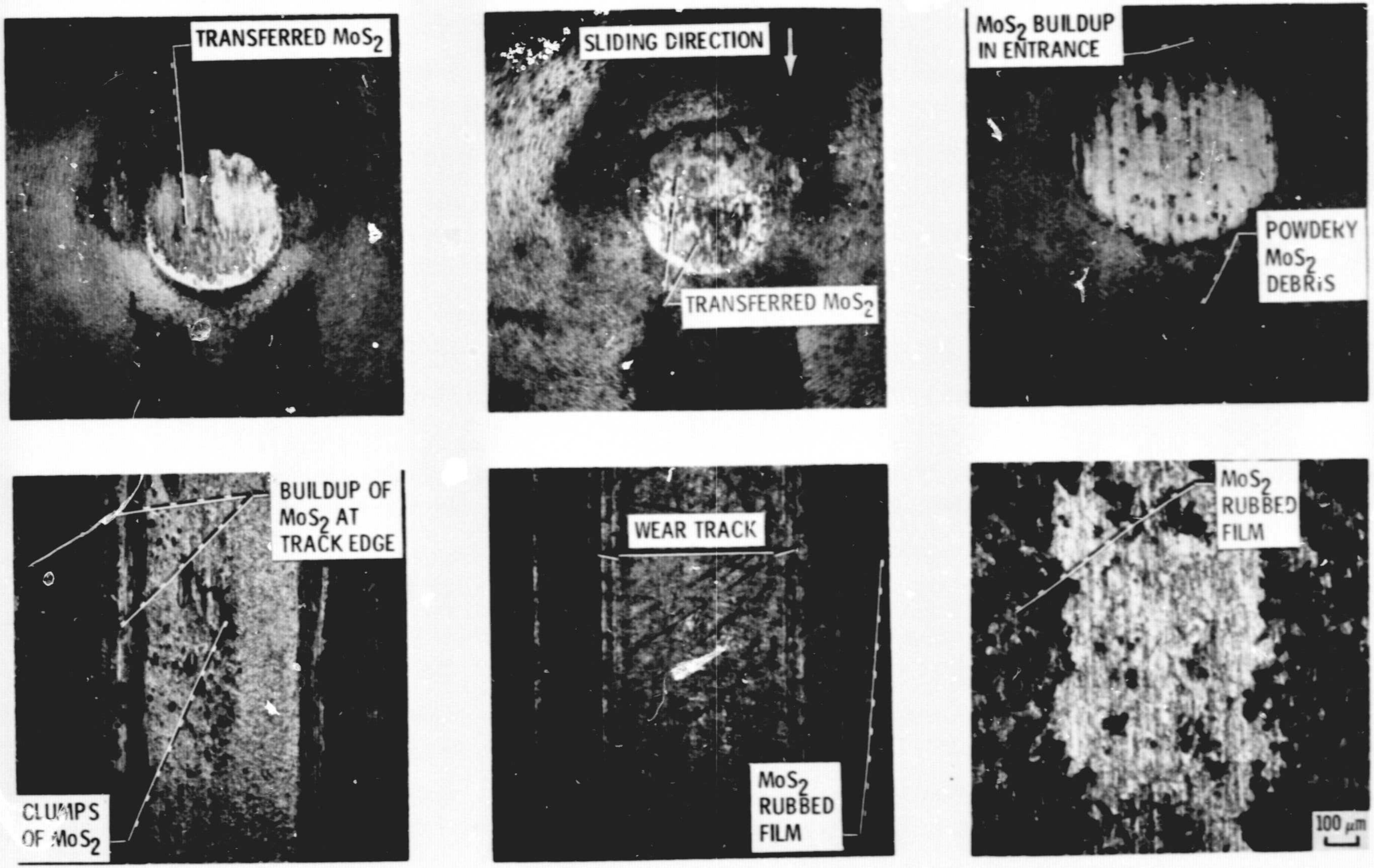


Figure 5. - Friction coefficient in moist air (10 000-ppm H_2O) as a function of kilocycles of sliding for rubbed MoS_2 films applied to 440C HT stainless-steel disks with different surface finishes.

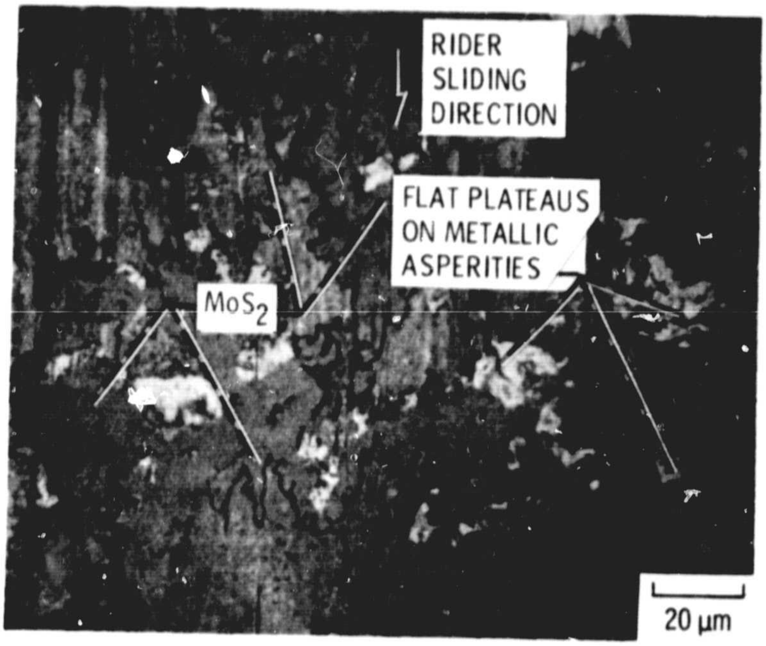


(a) POLISHED SUBSTRATE.

(b) SANDED SUBSTRATE.

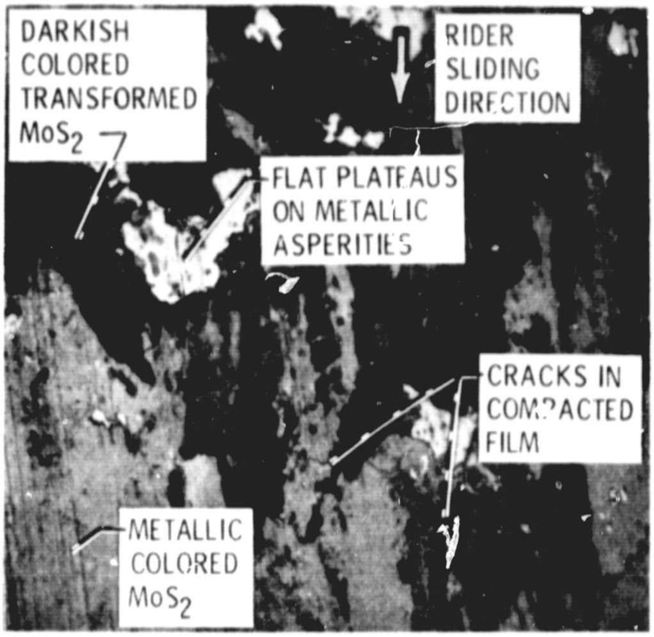
(c) SANDBLASTED SUBSTRATE.

Figure 6. - Photomicrographs (taken after 1 kilocycle of sliding in moist air (10 000-ppm H_2O)) of rider wear scars and wear tracks on rubbed MoS_2 films applied to 440C HT stainless-steel disks with different surface finishes.

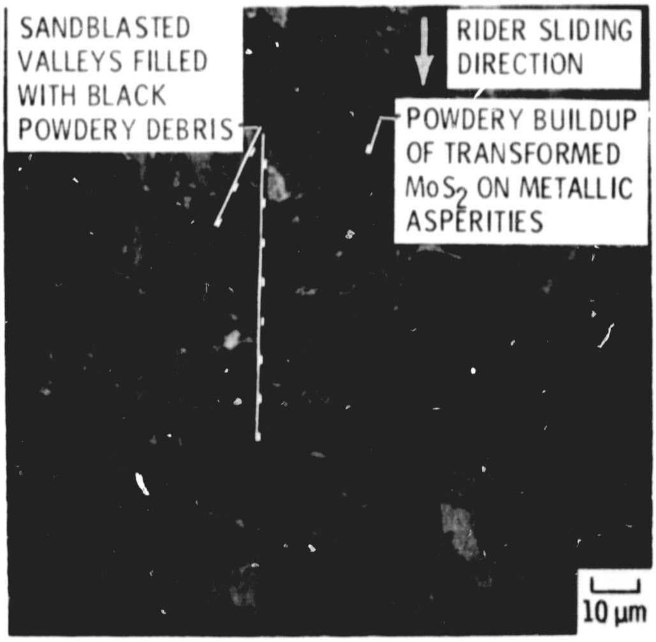


(c) SANDBLASTED SUBSTRATE

Figure 7. - Concluded.

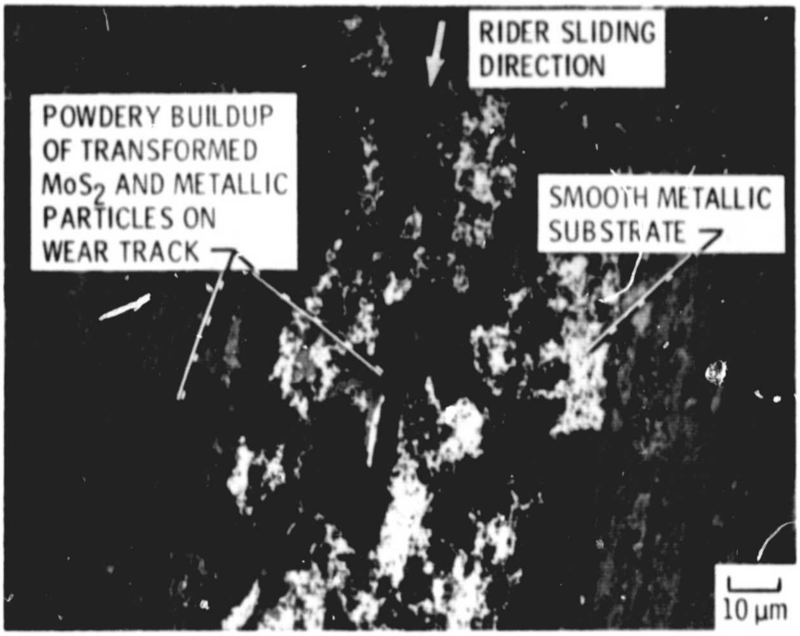


(a) AFTER 15 kilocycles OF SLIDING.

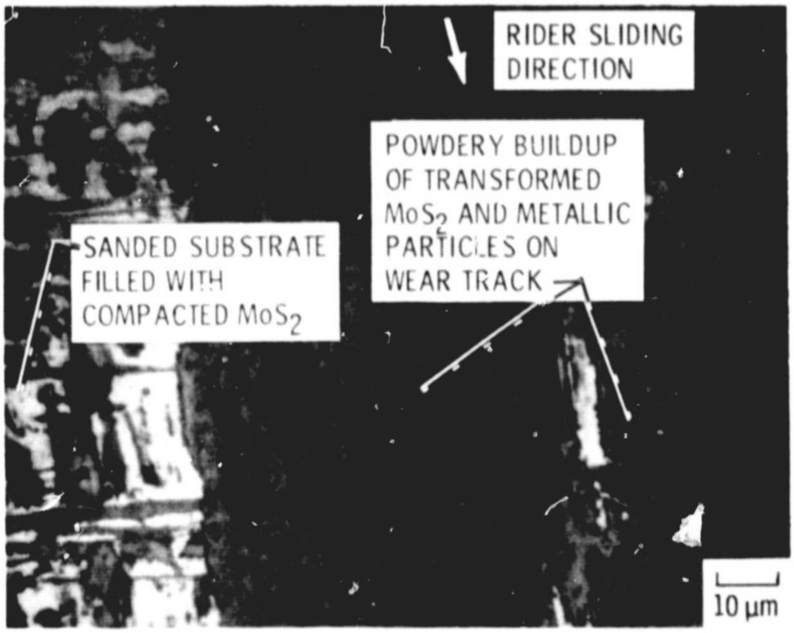


(b) AFTER 70 kilocycles OF SLIDING.

Figure 8. - High magnification photomicrographs of the wear tracks on rubbed MoS_2 films, applied to sandblasted 440C HT stainless-steel disks, after (a) 15 and (b) 70 kilocycles of sliding, indicating the gradual transformation of MoS_2 films in moist air.

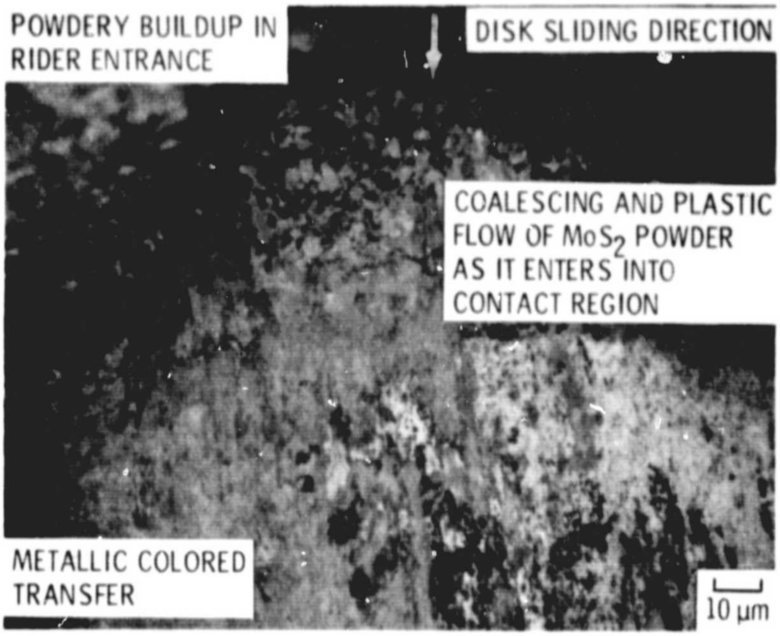


(a) SMOOTH SUBSTRATE-5 kilocycles OF SLIDING.

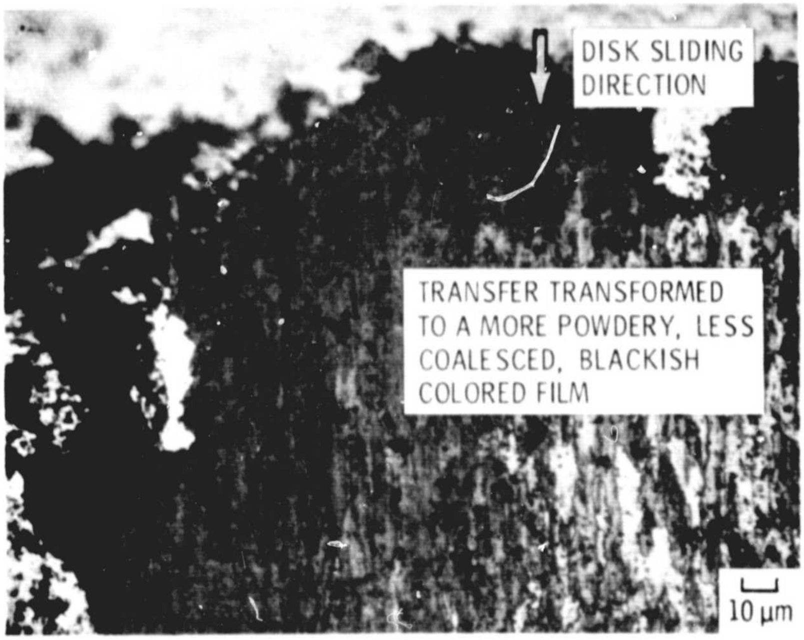


(b) SANDED SUBSTRATE-10 kilocycles OF SLIDING.

Figure 9. - High magnification photomicrographs of the wear tracks after failure occurred in moist air for MoS_2 rubbed films applied to (a) polished substrate and (b) sanded substrate.



(a) AFTER 1 kilocycle OF SLIDING.



(b) AFTER 10 kilocycles OF SLIDING (AFTER FAILURE).

Figure 10. - High magnification photomicrographs of the transfer in the entrance region to the 440C HT stainless steel rider which slid on the MoS_2 film applied to the sanded substrate showing the difference in transfer before and after failure in moist air.

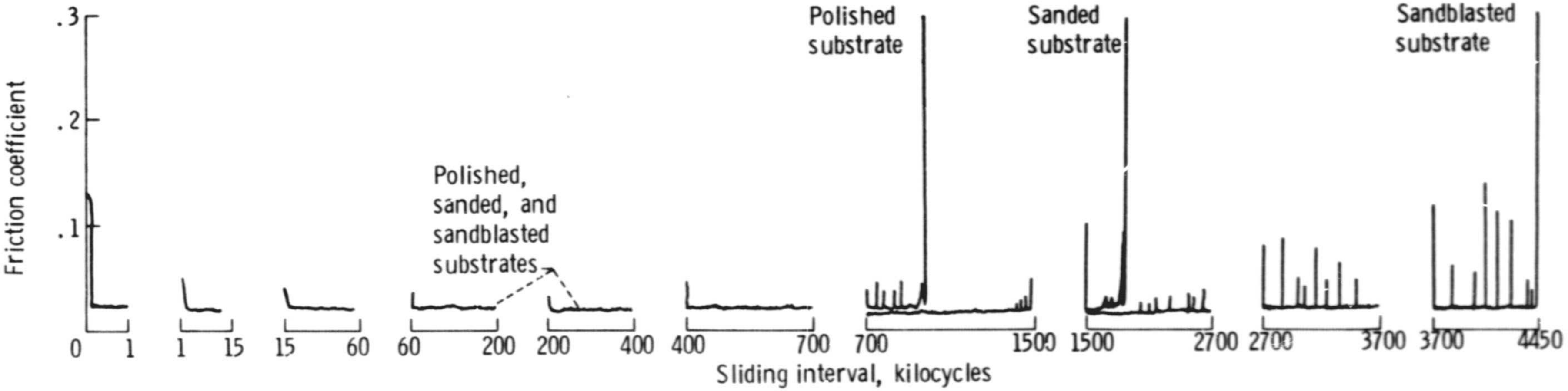
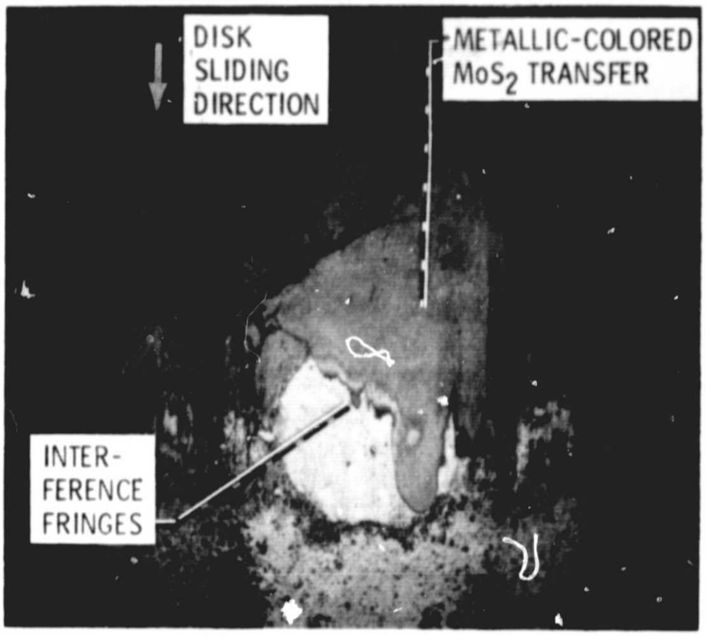
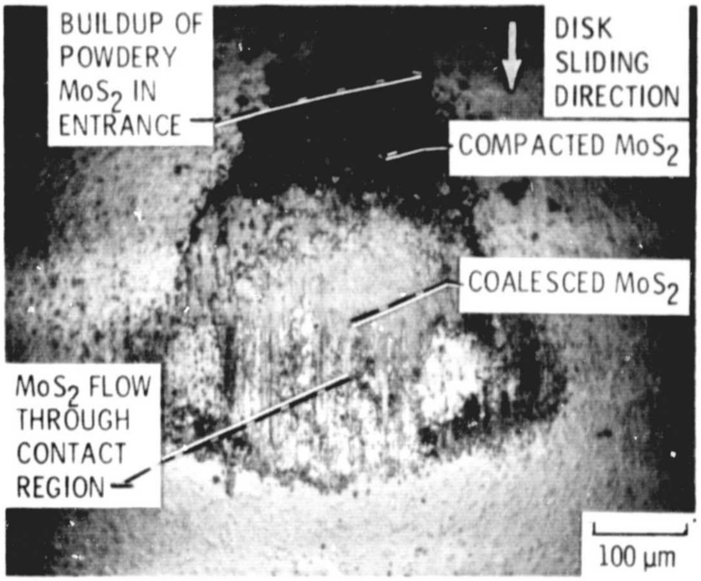


Figure 11. - Friction coefficient in dry argon (<20-ppm H_2O) as a function of kilocycles of sliding for rubbed MoS_2 films applied to 440C HT stainless-steel disks with different surface finishes.

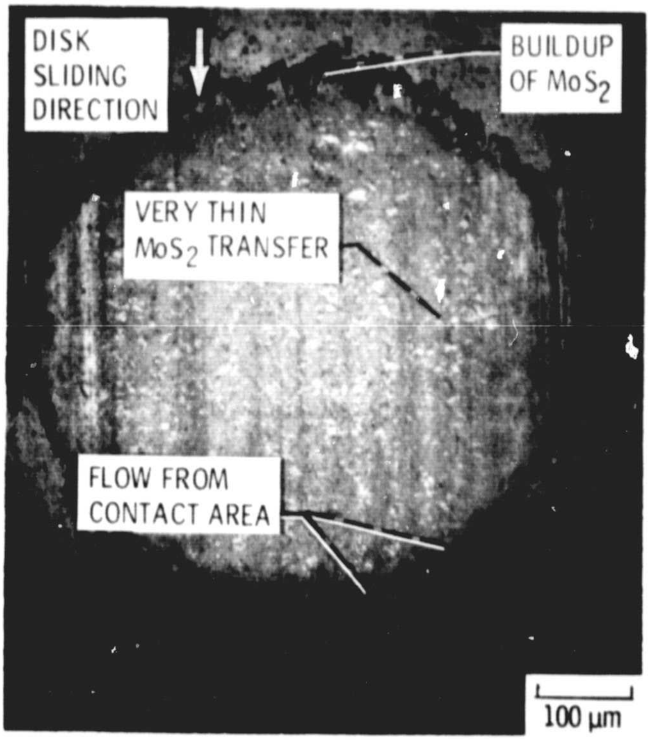


(a) POLISHED SUBSTRATE.



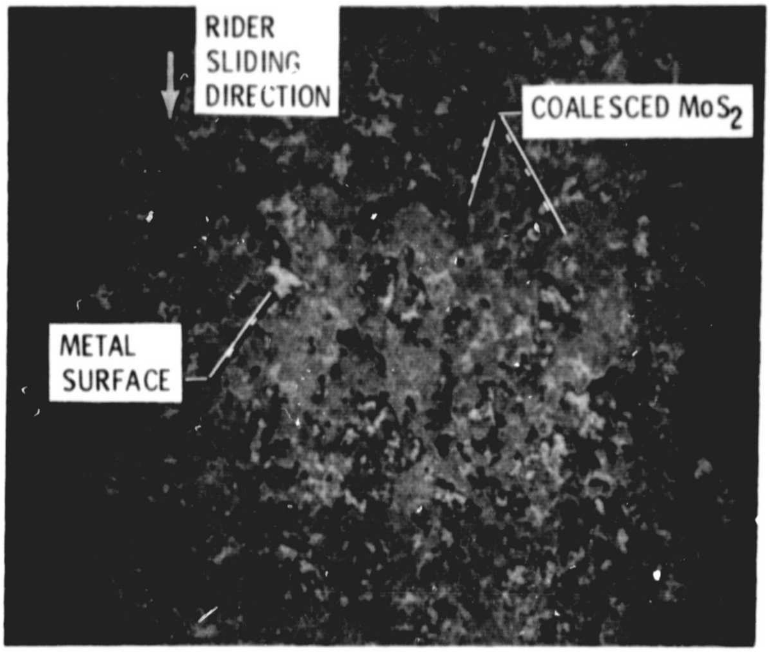
(b) SANDED SUBSTRATE.

Figure 12. - Photomicrographs (taken after 1 kilocycle of sliding in dry argon (<20-ppm H_2O)) of wear scars and transfer films on riders that slid on rubbed MoS_2 films applied to 440C HT stainless-steel disks with different surface finishes.

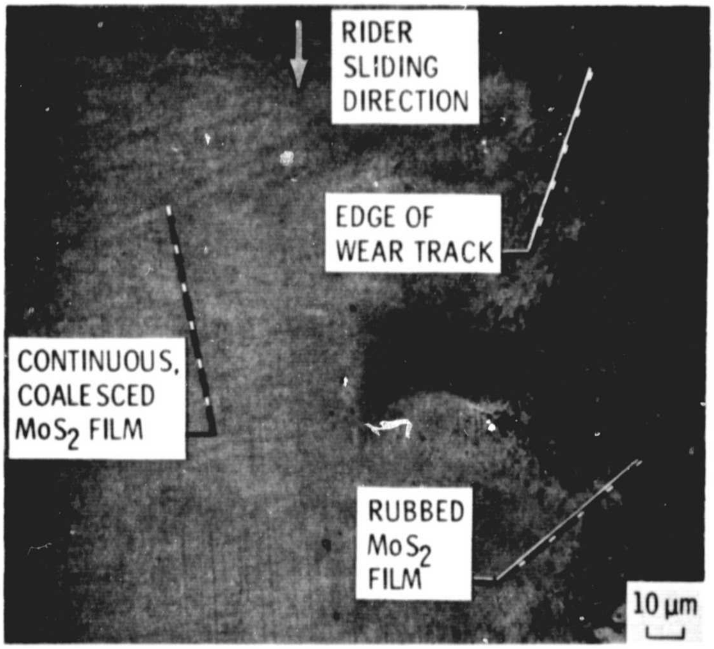


(c) SANDBLASTED SUBSTRATE.

Figure 12. - Concluded.



(a) POLISHED SUBSTRATE.



(b) SANDED SUBSTRATE.

Figure 13. - High-magnification photomicrographs (taken after 1 kilocycle of sliding in dry argon (<20-ppm H_2O)) of wear tracks on rubbed MoS_2 films applied to 440C HT stainless-steel disks with different surface finishes.

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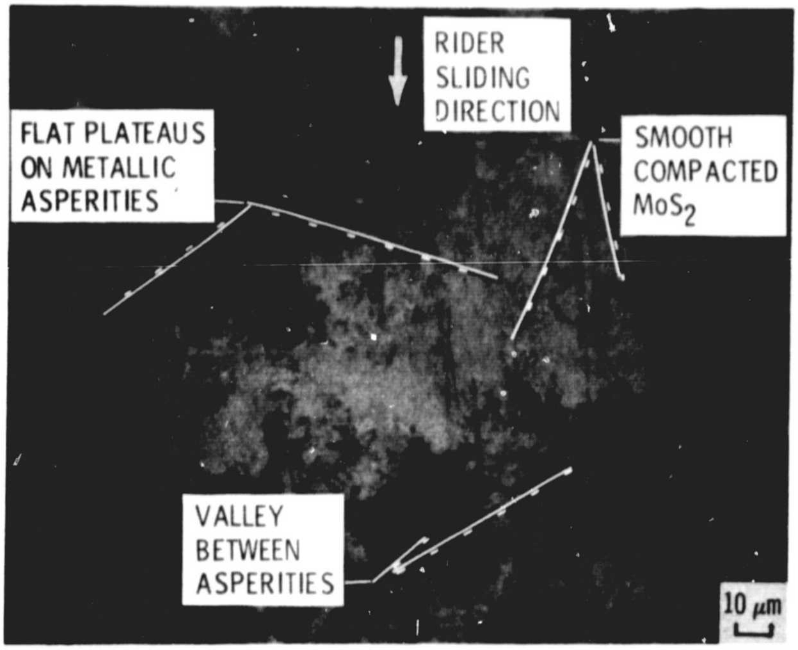
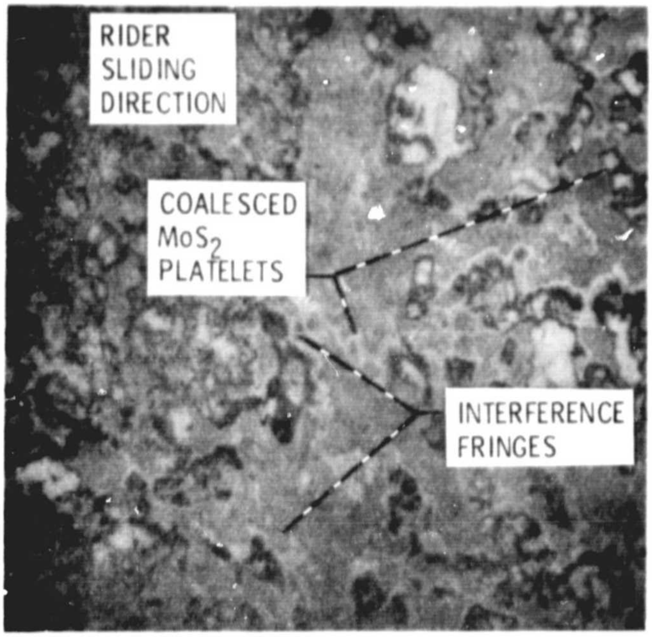
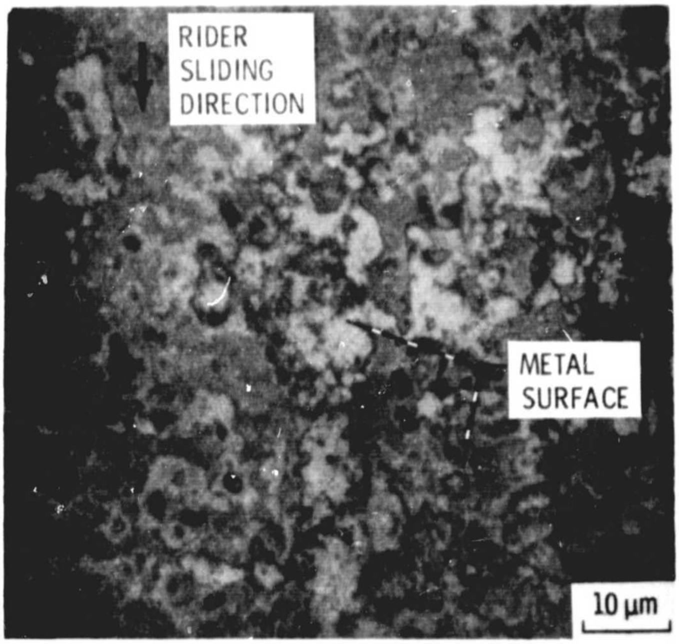


(c) SANDBLASTED SUBSTRATE.

Figure 13. - Concluded.

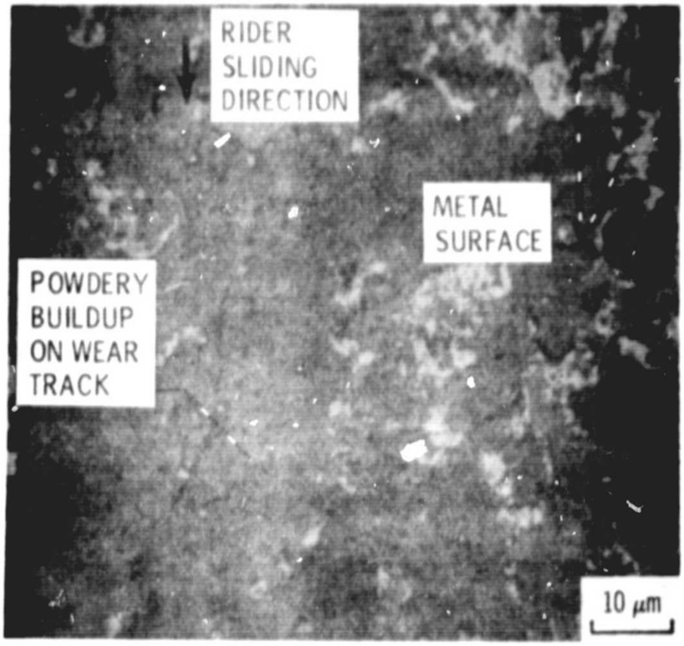


(a) AFTER 60 kilocycles OF SLIDING.



(b) AFTER 700 kilocycles OF SLIDING.

Figure 14. - High-magnification photomicrographs (taken in dry argon (<20-ppm H_2O) after various intervals of sliding) of wear tracks on rubbed MoS_2 films applied to polished 440C HT stainless-steel disk surfaces.



(c) AFTER 940 kilocycles OF SLIDING.

Figure 14. - Concluded.

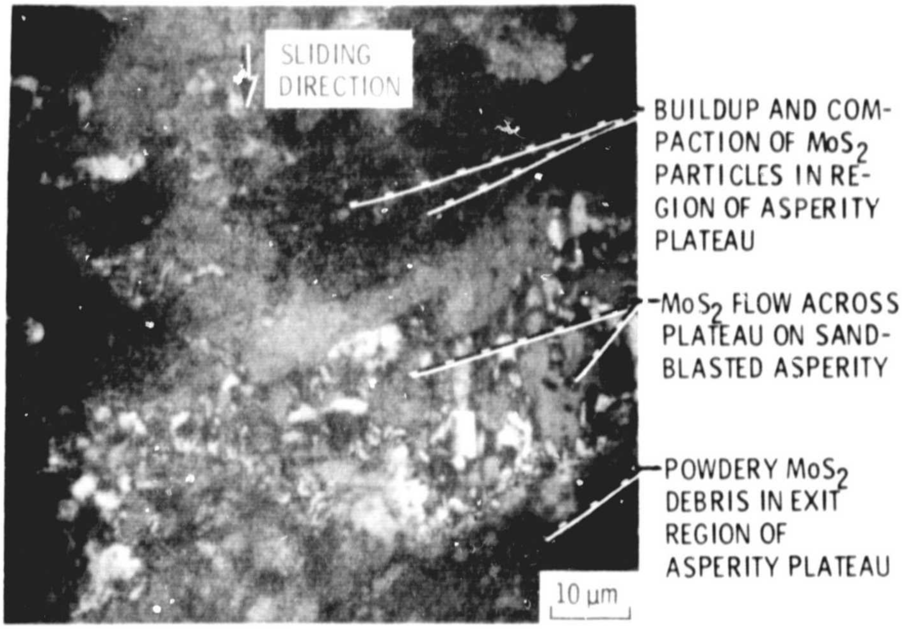


Figure 15. - High-magnification photomicrograph illustrating lubrication mechanism in dry argon (<20-ppm H_2O) of MoS_2 films applied to sandblasted 440C HT stainless-steel substrate surface.