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FERROGRAPHIC ANALYSIS OF WEAR DEBRIS
FROM BOUNDARY LUBRICATION EXPERIMENTS
WITH A FIVE-RING POLYPHENYL ETHER

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16. Abstract The types of wear particles generated by a five-ring polyphenyl ether in boundary lubrication experiments in various atmospheres were determined by Ferrographic analysis. The types of wear particles observed included cylindrical or rocklike organometallic debris, adhesive and cutting wear particles, and some spherical debris. Interpretations as to the mechanism of generation of the various types of particles are presented.			
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FERROGRAPHIC ANALYSIS OF WEAR DEBRIS FROM BOUNDARY LUBRICATION EXPERIMENTS WITH A FIVE-RING POLYPHENYL ETHER

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SUMMARY

The types of wear particles generated by a five-ring polyphenyl ether in boundary lubrication experiments were determined by Ferrographic analysis. Lubricant samples were obtained from tests conducted on a ball-on-disk sliding friction apparatus. Conditions included a 1-kilogram load, a 17-meter-per-minute sliding speed, 100^o to 250^o C disk temperatures, test atmospheres of air and nitrogen of varying moisture contents, and a test duration of 25 minutes. Test specimens were consumable electrode vacuum melted (CVM) M-50 steel.

In dry nitrogen (where high wear occurred) large quantities of an organometallic carbonaceous wear debris were observed; this debris consisted of either cylindrical or rocklike particles. The amount of the debris increased with increasing test temperature. In dry air (where lower wear occurred) smaller amounts of the carbonaceous debris were observed.

In wet atmospheres (air and nitrogen, where the lowest wear occurred) very little carbonaceous debris was generated. However, under these conditions, some spherical and a few severe (large) wear particles were observed.

Adhesive and cutting wear debris were observed under all conditions. However, the cutting debris appeared to be more prevalent in wet atmospheres (particularly wet air).

Therefore, it appears that a corrosive wear mechanism is operative with the polyphenyl ether under dry inert conditions. The presence of moisture (and to a lesser extent oxygen) inhibits this corrosive action. These observations are in accord with the radical anion wear model proposed by Goldblatt for polynuclear aromatics.

INTRODUCTION

The detection and analysis of wear debris generated in lubrication systems plays

an important role in elucidating what wear mechanisms are occurring. In addition, the sudden appearance of or an increase in the rate of production of a particular type of wear particle may be related to the incipient failure of a system component.

There are a number of methods available for detecting and analyzing wear debris in lubricants. The quantity and size distribution of particles may be measured by using ordinary particle counters which are based on a variety of different principles (refs. 1 and 2). Atomic absorption, emission spectroscopy, and X-ray fluorescence are also commonly used techniques for elemental analysis (refs. 3 to 6). Debris may be collected by filtration, examined microscopically, and subjected to other analytical procedures such as X-ray diffraction (ref. 7) and neutron activation analysis (ref. 8).

However, each of these methods has certain deficiencies. Particle counters are not able to differentiate between metallic and organic debris. Nor do they provide any information concerning particle morphology. Filtration often results in a massive pileup of all particulates (including those unrelated to the wear process) on the filter. Thus, it is very difficult to study individual particles. The spectroscopic and irradiation techniques are extremely sensitive but again do not differentiate between particles of different chemical nature.

Recently, a new instrument, the Ferrograph, has been developed which is capable of precipitating magnetic particles from a lubricant onto a transparent substrate to yield a Ferrogram (ref. 9). The precipitated particles range in size from approximately 0.02 to a few micrometers and are arranged according to size on the substrate. The density of the deposit may be measured optically, and individual particles may be observed with a unique bichromatic microscope, the Ferroscope. The Ferrogram may also be examined with an electron microscope.

Polyphenyl ethers are among the most thermally stable organic fluids known. Therefore, they have often been considered as possible high-temperature lubricants (refs. 10 and 11). However, these ethers have exhibited poor boundary lubricating characteristics in friction and wear tests (refs. 12 to 14), pump loops (ref. 15), and bearing tests (ref. 16). They are particularly poor lubricants under dry inert conditions (refs. 12, 15, and 17).

The objectives of this investigation were (1) to determine types of wear particles generated by a five-ring polyphenyl ether in boundary lubrication experiments by using Ferrographic analysis and (2) to relate the particle types to the wear mechanisms occurring with this fluid.

Lubricant samples were obtained from friction and wear tests conducted on a ball-on-disk sliding friction apparatus. Conditions included a 1-kilogram load (initial Hertz stress, 10^9 N/m^2), a 17-meter-per-minute (100-rpm) sliding speed, 100° to 250° C disk temperatures, test atmospheres of air and nitrogen of varying moisture contents, and a test duration of 25 minutes (in most cases). Test specimens were consumable

electrode vacuum melted (CVM) M-50 steel.

APPARATUS

The ball-on-disk sliding friction apparatus is shown in figure 1. The test specimens were placed in a stainless steel chamber. The atmosphere was controlled with respect to oxygen and moisture content. A stationary 0.476-centimeter-radius ball was placed in sliding contact with a rotating 6.3-centimeter-diameter disk. A sliding speed of 17 meters per minute (100 rpm) was maintained. A normal load of 1 kilogram (initial Hertz stress, 1×10^9 N/m²) was applied with a deadweight. Balls and disks were made of CVM M-50 tool steel. Disk and ball Rockwell hardness was C-62 to C-64.

The disk was partially submerged in a polyimide cup containing the test lubricant and was heated by induction. Bulk lubricant temperature was measured with a thermocouple. Disk temperature was monitored with an infrared pyrometer. Friction force was measured with a strain gage and was recorded on a strip-chart recorder.

PROCEDURE

Disks were ground and lapped to a root-mean-square surface finish of 10×10^{-8} to 20×10^{-8} meter (4 to 8 μ in.) and balls to 2.5×10^{-8} meter (1 μ in.). Specimens were scrubbed with a paste of levigated alumina and water, rinsed with tap water and distilled water, and then placed in a desiccator.

All lubricants tested under dry conditions were degassed at approximately 150^o C (302^o F) at 2 torr for 1 hour. The Karl Fischer method indicated that this degassing procedure reduces dissolved water content in the test fluids to less than 20 ppm.

The specimens were assembled, and 3×10^{-5} cubic meter (30 ml) of lubricant was placed in the lubricant cup. The test chamber (volume, 3.7×10^{-3} m³ (3.7 liters)) was purged with the test atmosphere for 10 minutes at a flow rate in excess of 5×10^{-2} cubic meter per hour (50 liters/hr). The disk was heated by induction to the test temperature while rotating. The ball was then loaded against the disk. The test atmosphere flow rate was reduced to 3.5×10^{-2} cubic meter per hour (35 liters/hr), and a 6.9×10^{-3} -newton-per-square-meter (1-psi) pressure was maintained in the chamber. The lubricant was heated only by heat transfer from the rotating disk. The bulk lubricant temperature was essentially the same as the disk temperature at disk temperatures to 100^o C (212^o F). At disk temperatures of 200^o and 300^o C (392^o and 572^o F), the bulk oil temperatures stabilized at approximately 150^o and 200^o C (302^o and 392^o F), respectively.

Friction force and bulk lubricant temperature were continuously recorded. Disk temperature was continuously monitored. Experiments were terminated after

25 minutes (in most cases) and rider wear scar diameter was recorded. All the used lubricant was retained for Ferrographic analysis.

Because of the extreme sensitivity of the Ferrographic technique, a rigorous cleaning procedure was adopted. All parts of the test apparatus that came into contact with the test lubricant were cleaned with a dilute acid solution. This acid solution was prepared by adding 10 drops of concentrated nitric acid and 10 drops of concentrated hydrochloric acid to 5×10^{-5} cubic meter (50 ml) of distilled water. This solution dissolved any metallic debris left from the previous test. In addition, the solvents and the unused oil were subjected to Ferrographic analysis to determine if they were free of metallic debris. Essentially no metallic contamination was found in these fluids.

TEST ATMOSPHERES

Four test atmospheres were used in this study. They were wet and dry air and wet and dry nitrogen. Dry air and dry nitrogen contained less than 50 and 20 ppm of moisture, respectively. Both wet gases had a relative humidity of greater than 95 percent at 25° C.

Relative humidity of the wet gases was monitored with an electronic hygrometer accurate to ± 4 percent. The moisture content of the dry gases was monitored by a moisture analyzer with an accuracy of ± 10 ppm. A wet gas was obtained by bubbling the dry gas through a water reservoir.

EXPERIMENTAL LUBRICANT

The polyphenyl ether used in these studies was an isomeric mixture of five-ring components. This fluid contained no additives. Typical properties of this fluid appear in table I. Previously reported wear results (ref. 17) for this fluid in dry air and dry nitrogen appear in figure 2(a). Figure 2(b) illustrates the effect of moisture on wear rate (ref. 12).

RESULTS

Several different types of wear debris were observed when the oil samples from the friction and wear tests were subjected to Ferrographic analysis. Table II summarizes the types of wear debris observed in this study. In addition, table II contains information regarding the morphology, size range, and appearance in the bichromatic microscope.

Wear Results

Figure 2(a) gives rider wear for the polyphenyl ether as a function of disk temperature for atmospheres of dry air and dry nitrogen. The effect of moisture on rider wear is shown in figure 2(b) at three disk temperatures: 150^o, 250^o, and 350^o C. It should be noted that the wear rates in the dry atmospheres of figure 2(b) do not exactly correspond to the rates shown in figure 2(a). The discrepancies arise because these results are from two separate studies and the wear rates were based on different test durations (25 min and 1 hr). In addition, the relative humidity of the wet gases in the study of figure 2(b) was 50 percent compared with greater than 95 percent in the present study. Nevertheless, the figures are qualitatively consistent and do illustrate the following points: High wear occurs under dry inert conditions. The presence of either oxygen or moisture in the test atmosphere reduces wear, with moisture having the greater effect. The abnormally low wear at 25^o C in figure 2(a) is due to the generation of an elastohydrodynamic film.

Ferrogram Results

Particles in the used oil are precipitated according to size on a transparent substrate, and the result is called a Ferrogram. The largest and the greatest density of wear particles occurs at the end of the Ferrogram where the used oil first contacts the substrate. For comparison purposes all photomicrographs were taken in this region.

The bichromatic (red-green) system of the Ferroscope facilitates differentiation among the types of wear debris. Free metal appears bright red, nonmetallic translucent particles appear yellow or green, and nonmetallic opaque debris appears black or dark red.

A typical photomicrograph from a Ferrogram appears in figure 3. At least three particle types are discernible. First, the horizontal red strings of material are adhesive wear particles. Second, the green material which appears in islands or randomly oriented strings is oxides. Third, the large black rocklike particles are carbonaceous debris. It should be noted that the term carbonaceous is used in this report in a descriptive rather than a chemical sense.

Effect of Temperature

Figure 4 illustrates the effect of disk temperature on the generation of wear debris in dry nitrogen. Some adhesive and cutting debris are evident. However, the most important observation is the large amount of carbonaceous debris present. Note the

large increase in this type of debris at 250° C. There also appears to be two distinct types. Some particles are irregularly shaped and rocklike. Others appear as small cylinders which are typically 1 to 3 micrometers in diameter and 10 to 15 micrometers in length. In general, the rocklike particles decrease and the cylinders increase with increasing temperature. The cylinders also decrease in size with increasing temperature.

Effect of Oxygen and Moisture

The effect of oxygen or moisture in the test atmosphere on wear debris generation at 100° C is illustrated in figure 5. It is immediately evident that the presence of moisture in the test atmosphere almost completely inhibits the formation of the carbonaceous debris. The presence of oxygen also decreases the production of carbonaceous debris, but to a much lesser extent than moisture. More metallic debris is noted in wet air than in wet nitrogen.

Cutting wear debris was observed in all Ferrograms but was more prevalent under moist conditions, particularly in wet air. Some examples of this type of debris appear in figure 6.

Another moisture effect manifests itself in the generation of spherical debris such as that illustrated in figure 7. Spheres were rarely observed in dry atmospheres but always under moist conditions.

Finally, a number of Ferrograms contained some severe (large) wear particles, such as the large wear fragment shown in figure 8. Again, these particles appeared more frequently in moist atmospheres.

DISCUSSION

The boundary lubrication behavior of polyphenyl ethers has been well documented. Polyphenyl ethers are poor boundary lubricants under dry inert conditions (refs. 12, 15, and 17). Under these conditions they also form large quantities of an insoluble carbonaceous debris (refs. 8, 12, 13, 15, and 18). The presence of either oxygen or moisture in the test atmosphere improves their boundary lubricating properties. Moisture, and to a lesser extent oxygen, reduces the production of the carbonaceous debris.

Polyphenyl ethers have also performed poorly in bearing tests (ref. 16). This is surprising. The aromatic structure of these fluids yields a high pressure-viscosity coefficient, which, combined with their high bulk viscosity, should make these fluids excellent elastohydrodynamic (EHD) film formers. Their poor bearing performance

may be related to their poor boundary characteristics. Polyphenyl ethers also exhibit poor wettability (ref. 12). Poor wetting can encourage lubricant starvation in the vicinity of the inlet to the Hertzian contact, which would lead to thinner than expected EHD films.

At any rate, the polyphenyl ethers appear to follow the general boundary lubricating behavior of polynuclear aromatics reported by Appeldoorn and Tao (ref. 19). The following sections discuss the various types of wear debris generated by the polyphenyl ether during the boundary lubrication experiments.

Carbonaceous Debris

Many investigators have observed the generation of carbonaceous debris in boundary lubrication experiments with polyphenyl ethers. Apparently, a wearing situation must exist for its production, since this type of debris is not observed in static thermal stability tests. Spar and Damasco (ref. 13) found large quantities of metallic debris in the carbonaceous product from their experiments. Analysis of the organic portion of their debris yielded a higher molecular weight structure similar to the base fluid. Analysis of the carbonaceous debris in the study of reference 18 yielded similar results. Therefore, this debris is not just a cokelike deposit caused by high-temperature degradation of the lubricant.

Goldblatt (ref. 20) proposed the following model to explain the lubricating behavior of aromatics: He theorized that aromatic radical anions are generated at the wearing surfaces. These highly reactive molecules can then react with the metal surfaces and produce a form of corrosive wear. The radical anions can also be "quenched" by reacting with either oxygen or water and are thus prevented from corrosively attacking the surface.

One may speculate that a similar phenomenon occurs with the polyphenyl ethers. In dry nitrogen, radical anions formed from the polyphenyl ether molecules attack the metal surface and produce an organometallic friction polymer (called surface resin by Goldblatt). This product is removed from the surface during the sliding process and accumulates around the wear scar and in the fluid. The fact that much of the observed carbonaceous debris is in the form of small cylinders tends to support this idea. Other investigators, such as Antler (ref. 21) and May, Morris, and Attack (ref. 22), have reported the production of roller-shaped debris in sliding experiments.

Another interesting observation is that, even though the wear (determined by the wear scar) is high in dry nitrogen, very little free metallic debris is evident in the Ferrograms. If a corrosive mechanism is operative, one would expect much of the worn metal to be contained in the carbonaceous debris in the form of an organometallic

reaction product. Of course, it would also be possible for free metallic particles to become embedded in the carbonaceous debris while it is being stripped off the surface. At any rate, when a Ferrogram containing carbonaceous debris is placed on a hotplate at 480° C, the debris becomes translucent and retains its original morphology. It also exhibits the orange-red color of iron oxide. The fact that large quantities of this carbonaceous debris are precipitated by the magnetic field of the Ferrograph further indicates the presence of iron. An electron micrograph of some of the carbonaceous particles appears in figure 9(a). An iron X-ray map of the same area appears in figure 9(b) and confirms the presence of iron in both types of particle.

The large decrease in wear rate at temperatures above 200° C (in dry nitrogen) may be a consequence of the increased reactivity of the radical anions at high temperature. If the rate of production of surface resin were greater than its effective removal rate during the sliding process, a reduction in wear could be effected. This would be a result of a quasi-hydrodynamic film formation or a microelastohydrodynamic effect such as proposed by Fein and Kreuz (refs. 7 and 23).

The decrease in carbonaceous debris in the presence of water or oxygen follows the Goldblatt model. Either water or oxygen "quenches" or ties up radical anions before they can react with the surface. The apparent greater effectiveness of water compared with oxygen in quenching the anions may be a concentration effect. The high solubility parameter of polyphenyl ethers indicates a high solubility for water but a low solubility for the fixed gases, such as oxygen (ref. 24).

Adhesive Wear Debris

The generation of wear particles by an adhesion mechanism has been proposed by many investigators (refs. 25 to 27). This mechanism is operative when the boundary film fails and metal-to-metal junctions are formed. Relative motion then causes the welded junction to shear. Material is transferred from one surface to another and is subsequently removed during the sliding process.

Suh (ref. 28) has proposed a new wear mechanism based on dislocation theory. It involves the formation of subsurface voids, crack propagation between the voids, and the subsequent shearing of this material to form a sheetlike wear particle. This sheetlike particle may then be broken into smaller particles or may be rolled into a larger particle during the sliding process. Many of the particles observed in this study which have been referred to as adhesive wear do appear as sheets.

Figure 10 is a photomicrograph of a portion of a wear scar from one of the experiments in this study. It was taken with the Ferroscope by utilizing an interference contrast technique. It appears that a thin layer of material was generated on the surface. This layer may have been related to the often mentioned but little understood

white or nonetching wear transformation layer (refs. 29 to 31) and may have been the source of so-called adhesive wear particles. In fact, there is an area in the center of the photograph where some of this layer was stripped off. The vertical resolution in this photograph is about 5×10^{-8} meter (500 Å).

Cutting Wear Debris

Another common wear mode is cutting (ref. 32). This type of wear results when a hard particle or surface plows out a softer surface. It is also often referred to as abrasive wear. Loose work-hardened wear particles are no doubt present in the fluid during the test. However, Scott, Seifert, and Westcott (ref. 33) have been unable to reproduce cutting wear particles by adding abrasive particles (sand) to lubricants. A possible explanation for this type of debris could be a micromachining process caused by work-hardened particles adhering to the rider prow. Another possibility may be hard inclusions in the metal itself, such as carbides. The increased generation of cutting wear particles in wet atmospheres may have been caused by the formation of abrasive oxides under these conditions.

Spherical Debris

Many investigators have noted the occurrence of spherical particles in bearing and friction and wear tests (refs. 34 to 40). Finken (ref. 34) reported the formation of large spheres (apparently iron oxide) in friction and wear tests with 1020 steel. Suh (ref. 28) observed spherical metallic debris and theorized that it was produced from metal particles which were trapped between the sliding surfaces and rolled into a ball. Spheres have also been observed in fretting studies (ref. 38). Broszeit and Hess (ref. 39) observed spheres in grinding debris and related their formation to a high-temperature melting process. Scott and Mills (refs. 36 and 37) observed metallic spheres during microscopic examination of bearing fatigue surfaces. They felt that the spheres are formed by a deformation process within propagating fatigue cracks. In fact, it has been suggested that their detection in a bearing lubricant may allow the prediction of incipient bearing fatigue failure (ref. 33).

The spheres observed in this study appeared as free metal in the bichromatic system (i.e., they appeared red). Energy-dispersion X-ray analysis confirmed their metallic content. These spheres may have been generated by a fatigue related process. Certainly, fatigue under sliding conditions has been demonstrated by a number of investigators (refs. 41 to 43). However, the number of stress cycles generated in the wear tests of this study was very low (generally less than 2500). And it is possible

that the disk surface was worn away before crack propagation could occur. Nevertheless, spheres were always observed under moist test conditions and rarely in dry atmospheres. The presence of moisture certainly accelerates the fatigue process (refs. 44 and 45). An electron micrograph of some typical spheres appears in figure 11(a).

Severe (Large) Wear Particles

Occasionally some severe (large) wear particles (diam, $>30 \mu\text{m}$) were observed in the Ferrograms. These particles may have been the result of processes such as surface fatigue or surface fracture. However, they were only rarely encountered and did not appear to be a major component in the wear processes of this study. An electron micrograph of one such particle appears in figure 11(b).

SUMMARY OF RESULTS

The major results of the Ferrographic analysis of the wear debris generated by a five-ring polyphenyl ether in boundary lubrication experiments may be summarized as follows:

1. In dry nitrogen (where high wear occurred) large quantities of carbonaceous organometallic wear debris were observed; this debris consisted of either cylindrical or rocklike particles. The amount of the debris increased with increasing test temperature.
2. In dry air (where lower wear occurred) smaller amounts of the carbonaceous debris were observed.
3. In wet atmospheres (air and nitrogen, where the lowest wear occurred) very little carbonaceous debris was generated. However, under these conditions, some spherical and a few severe (large) wear particles were observed.
4. Adhesive and cutting wear debris were observed under all conditions. However, the cutting debris appeared to be more prevalent in wet atmospheres (particularly wet air).
5. Therefore, it appears that a corrosive wear mechanism is operative with the polyphenyl ether under dry inert conditions. The presence of moisture (and to a lesser

extent oxygen) inhibits this corrosive action. These observations are in accord with the radical anion wear model proposed by Goldblatt for polynuclear aromatics.

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TABLE I. - SOME PROPERTIES OF A FIVE-RING
POLYPHENYL ETHER

Kinematic viscosity, m ² /sec (cS)	
At 38 ^o C (100 ^o F)	3.6×10 ⁻⁴ (360)
At 99 ^o C (210 ^o F)	1.3×10 ⁻⁵ (13)
At 350 ^o C (662 ^o F)	7.2×10 ⁻⁷ (0.72)
Pour point, ^o C (^o F)	5 (40)
Flash point, ^o C (^o F)	288 (550)
Fire point, ^o C (^o F)	350 (662)
Density at 38 ^o C (100 ^o F), kg/m ³ (g/ml)	1.19×10 ³ (1.19)
Thermal decomposition (isoteniscope), ^o C (^o F)	443 (830)
Vapor pressure at 343 ^o C (650 ^o F), torr	12
Surface tension at 25 ^o C (77 ^o F), N/cm (dynes/cm)	5×10 ⁻⁴ (50)

TABLE II. - DESCRIPTION OF DIFFERENT TYPES OF WEAR DEBRIS GENERATED BY
A FIVE-RING POLYPHENYL ETHER

Type of wear debris	Morphology (as debris appears in Ferrograms)	Size range, μm	Appearance in bichromatic microscope
Free metal			
Adhesive	Strings (actually made up of small asymmetrical flakes) which have agglomerated during precipitation	Length, <5	Bright red
Cutting	Individual particles (wire-like turnings and crescent-shaped chips)	Variable	Bright red
Spheres	Spherical particles (often found interspersed in adhesive strings)	Diameter, 1 to 10	Bright red
Severe wear	Large irregularly shaped fragments	Diameter, 30 to 50	Bright red
Inorganic (oxides)	Translucent islands or randomly oriented strings	Diameter, <1	Yellow-green
Organic	Carbonaceous particles (containing some metallic debris)		Dark red
	Rocklike	Diameter, 5 to 20	
	Cylindrical	Diameter, 1 to 3; length, 10 to 15	

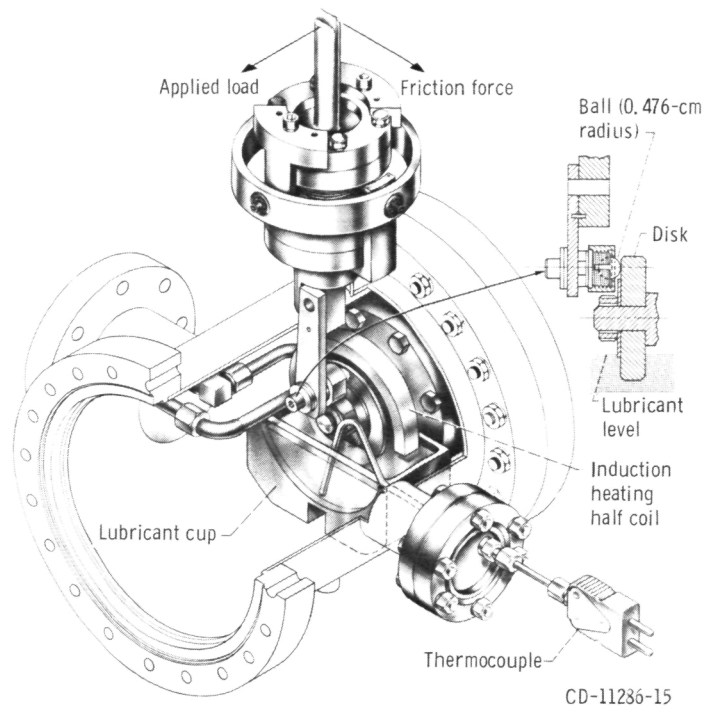
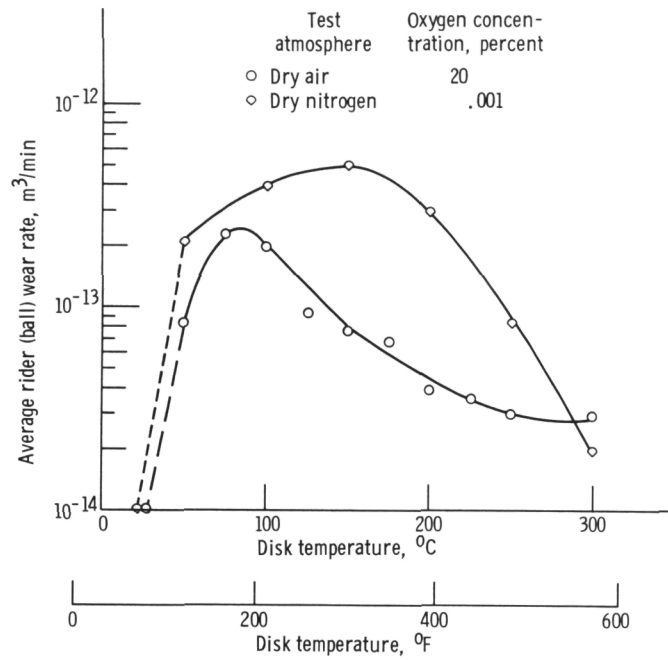
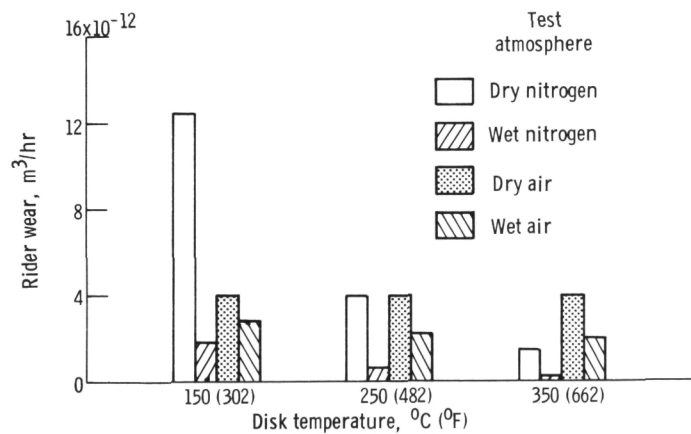


Figure 1. - Friction and wear apparatus.



(a) Test duration, 25 minutes (from ref. 17).



(b) Test duration, 1 hour (from ref. 12).

Figure 2. - Rider wear as function of disk temperature with five-ring polyphenyl ether as lubricant in various test atmospheres. Specimen material, CVM M-50 steel; load, 1 kilogram; sliding speed, 17 meters per minute (100 rpm).

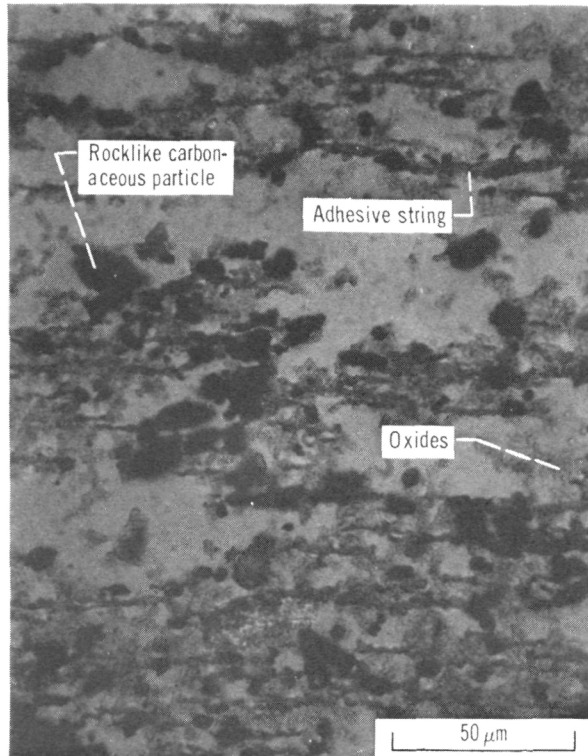
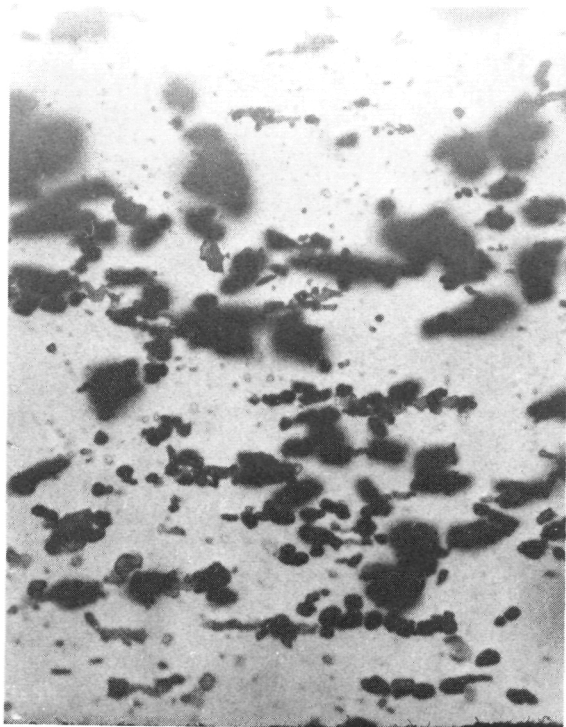
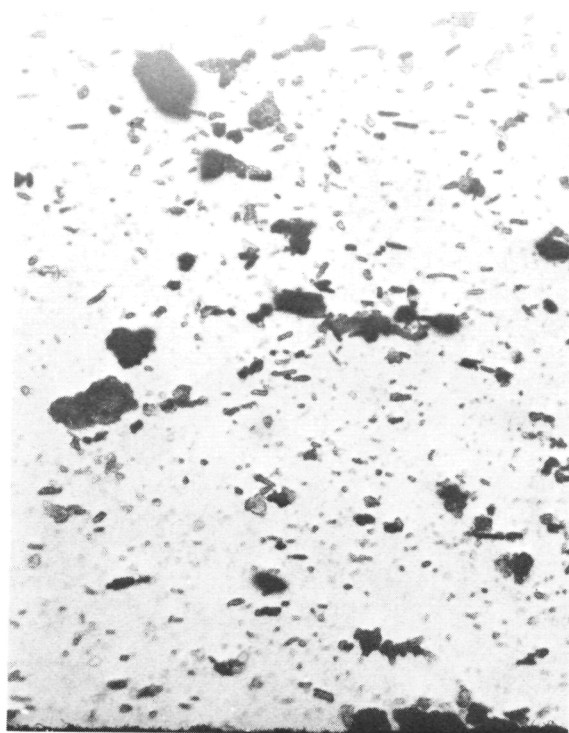


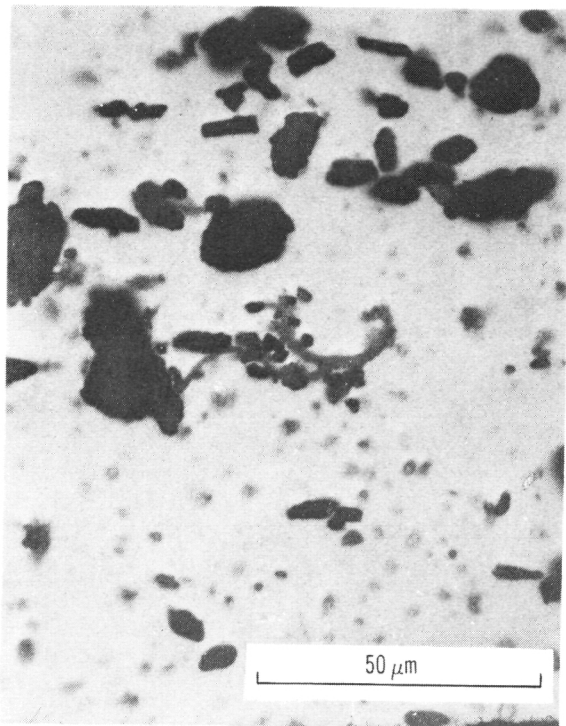
Figure 3. - Photomicrograph of wear debris generated by polyphenyl ether at 100° C in dry test atmosphere of 1 percent oxygen and 99 percent nitrogen (by volume).



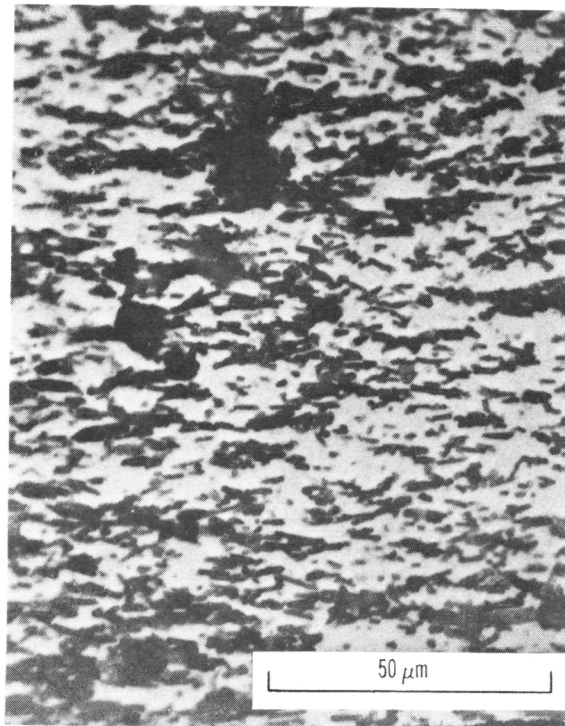
(a) 100° C.



(c) 200° C.



(b) 150° C.

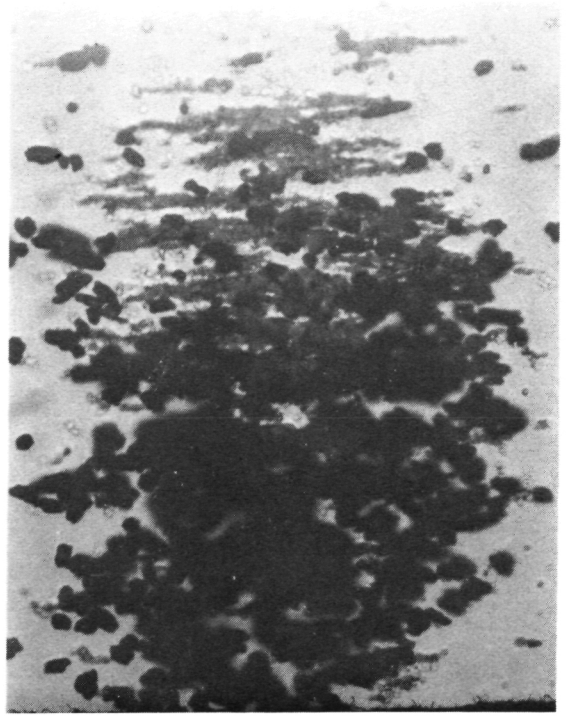


(d) 250° C.

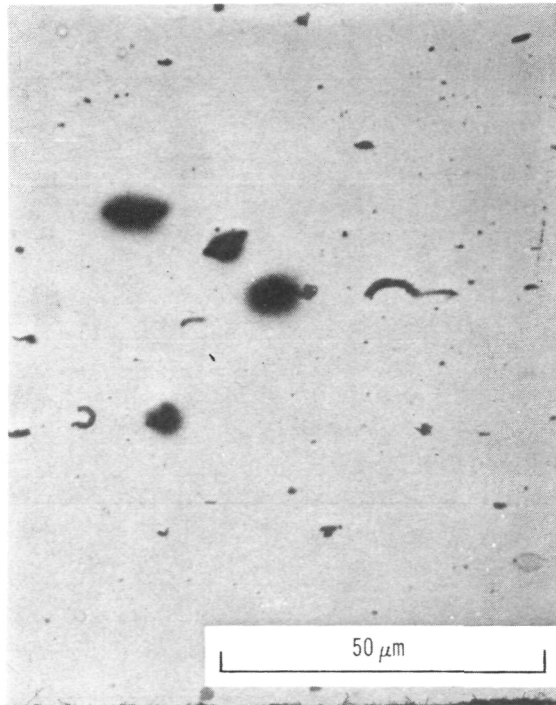
Figure 4. - Photomicrographs of wear debris generated by polyphenyl ether in dry nitrogen at four test temperatures.



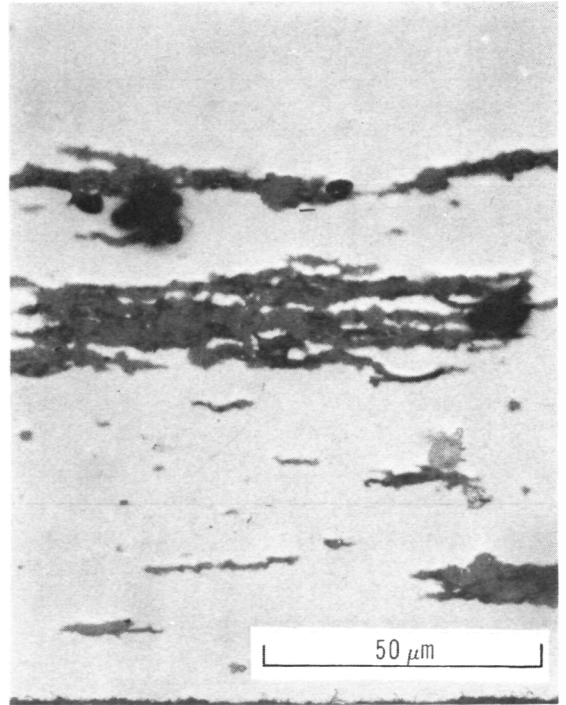
(a) Dry nitrogen.



(c) Dry air.

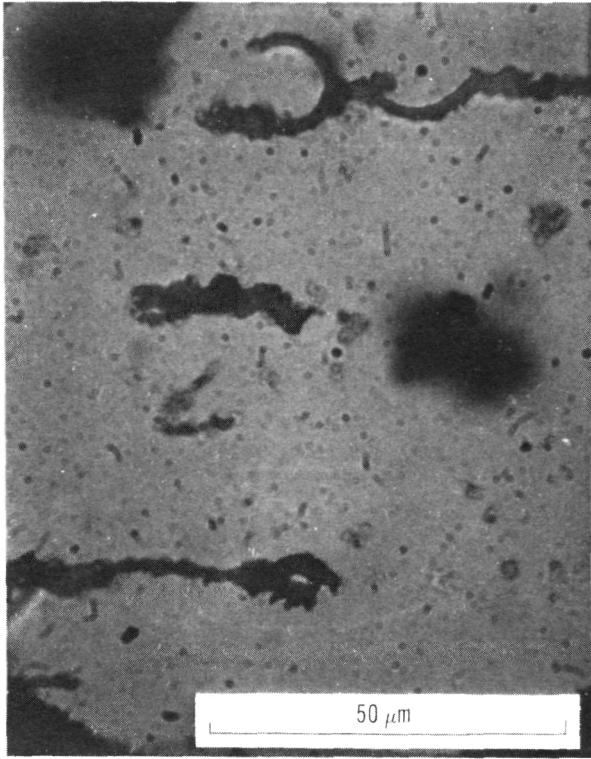


(b) Wet nitrogen (relative humidity, > 95 percent).

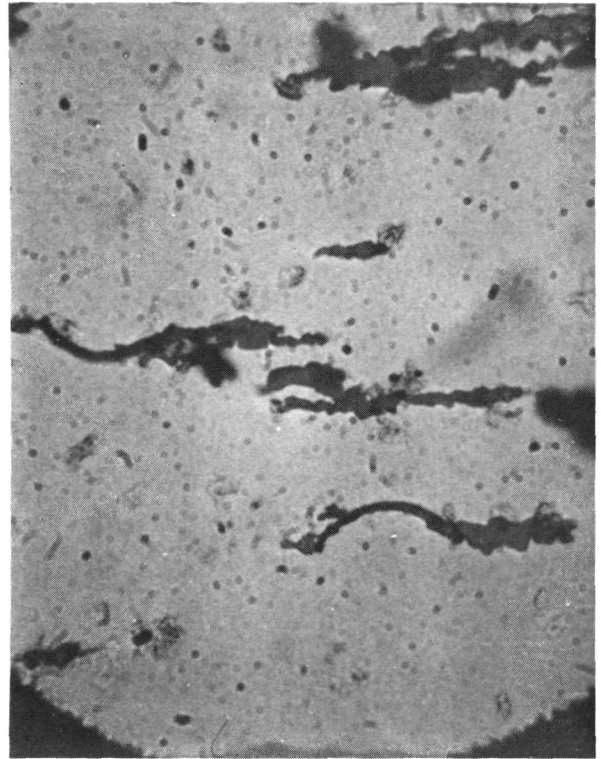


(d) Wet air (relative humidity, > 95 percent).

Figure 5. - Photomicrographs of wear debris generated by polyphenyl ether at 100°C in four test atmospheres.



(a) Nitrogen.



(b) Air.

Figure 6. - Photomicrographs of cutting wear debris generated by polyphenyl ether at 100°C in wet test atmospheres (relative humidity, > 95 percent).

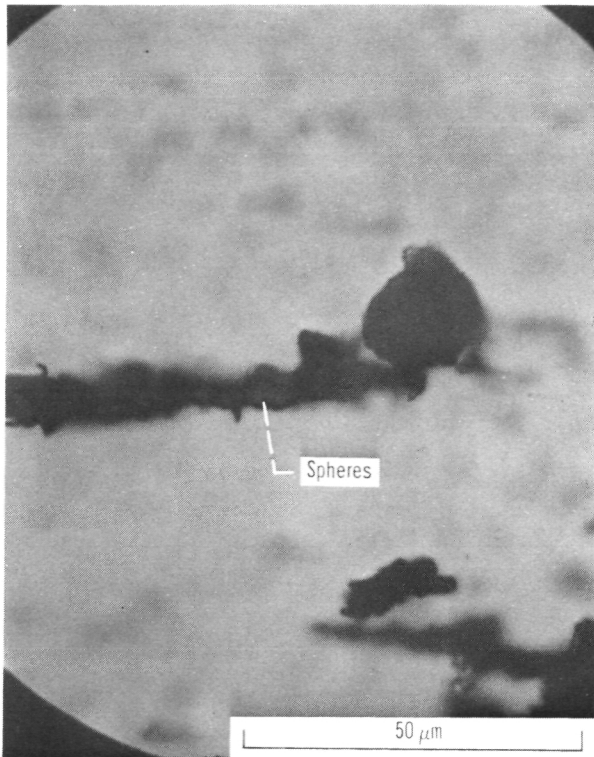


Figure 7. - Photomicrograph of wear debris containing metallic spheres generated by polyphenyl ether at 100° C in air (relative humidity, > 95 percent).

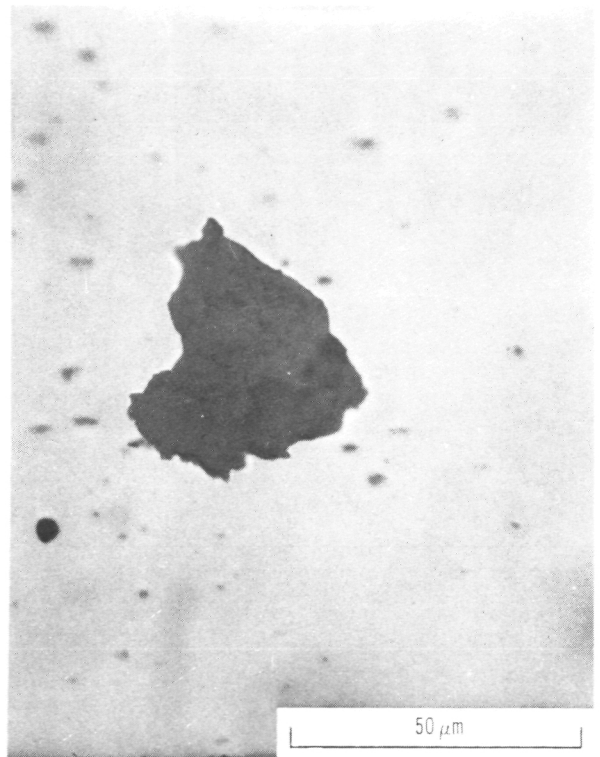
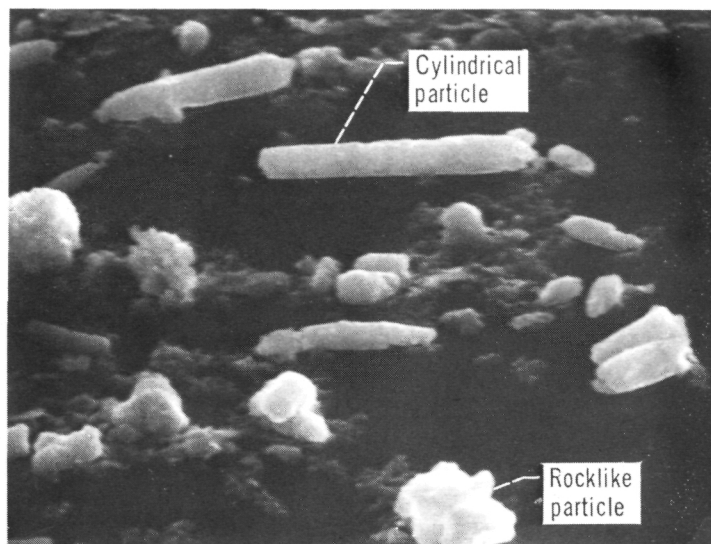
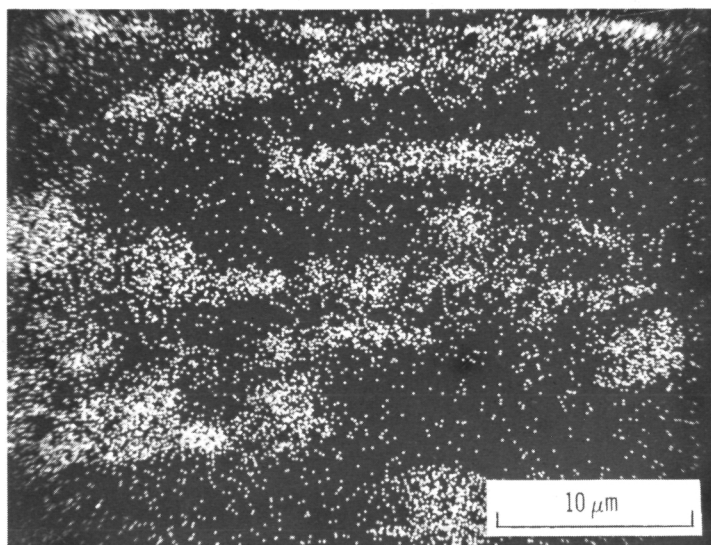


Figure 8. - Photomicrograph of large wear fragment generated by polyphenyl ether at 100° C in air (relative humidity, > 95 percent).



(a) Electron micrograph of carbonaceous wear debris.



(b) Iron X-ray map.

Figure 9. - Electron micrograph of carbonaceous wear debris and iron X-ray map of same area.

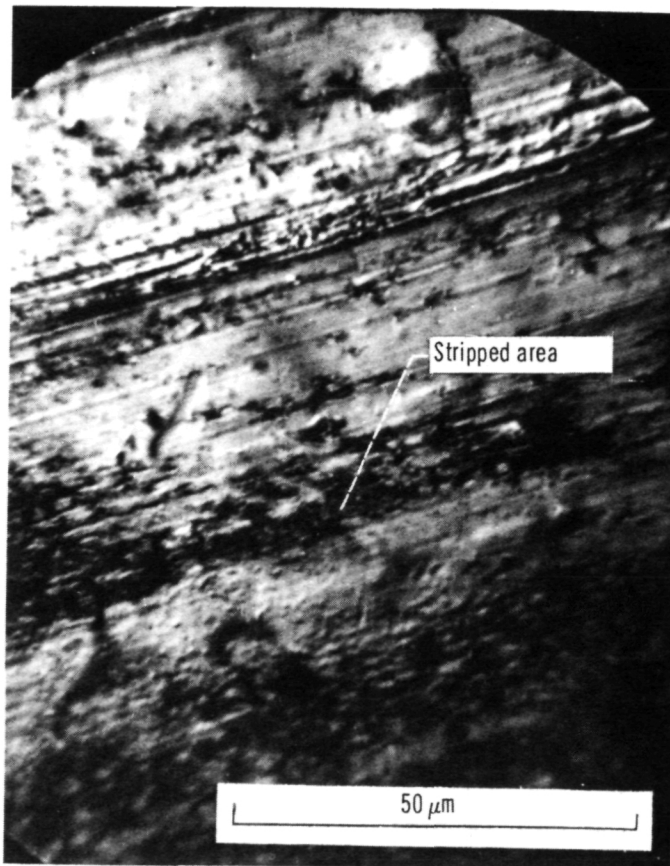
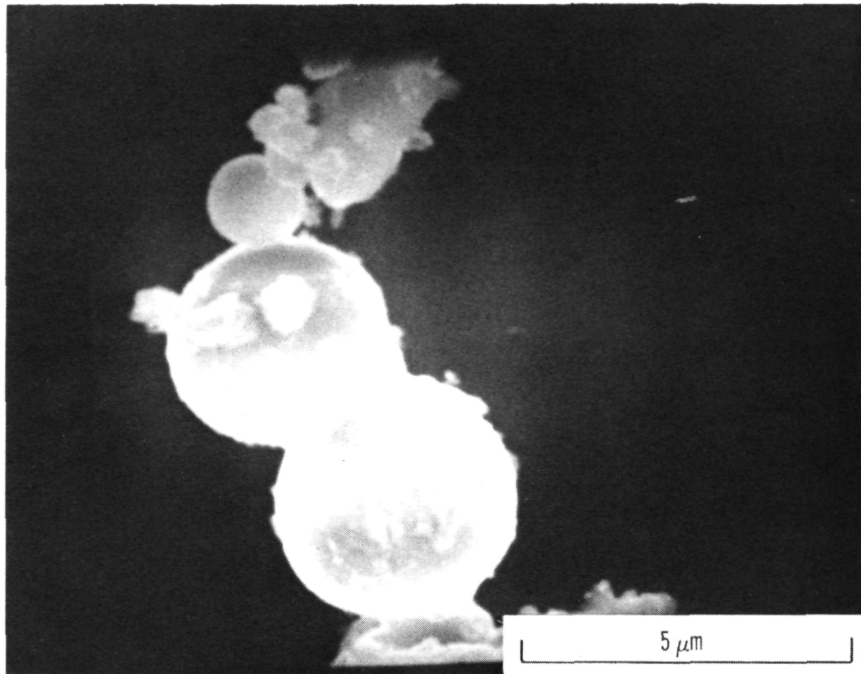
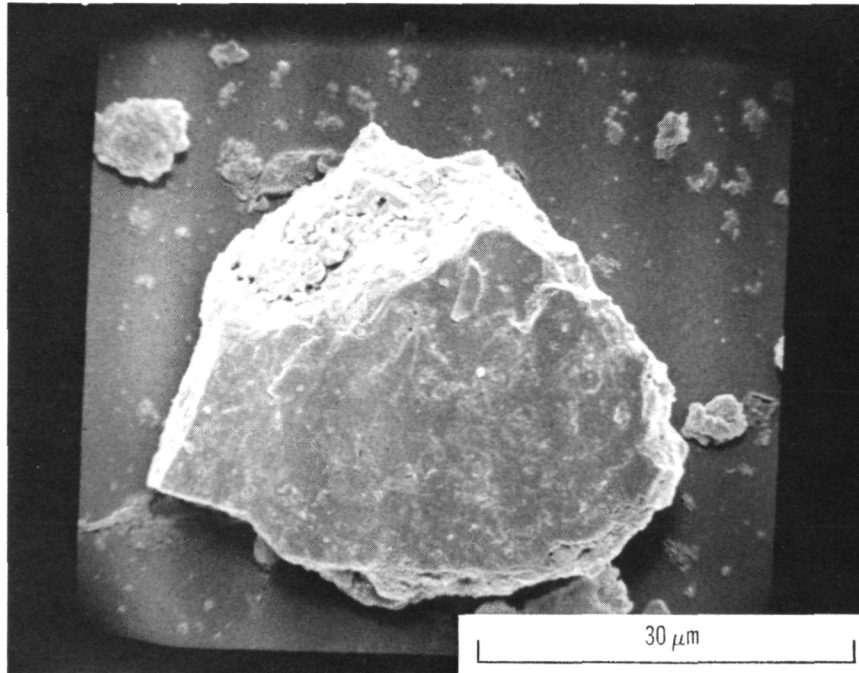


Figure 10. - Photomicrograph of wear scar obtained from interference contrast system. Lubricant, polyphenyl ether; specimen material, M-50 steel; temperature, 100° C; test atmosphere, nitrogen (relative humidity, 50 percent).



(a) Spherical particles.



(b) Large wear fragment.

Figure 11. - Electron micrographs of two types of metallic wear debris.



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