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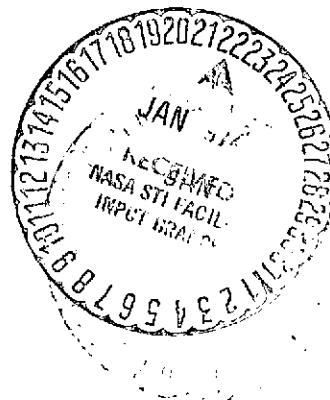


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16. Abstract An iron surface was exposed to the extreme-pressure type lubricant additives benzyl chloride, dichlorophenyl phosphine, dichlorophenyl phosphine sulfide, and dichlorophenyl phosphine oxide. Iron, in the sputter-cleaned state, was exposed to these materials statically and during dynamic friction experiments. With benzyl chloride only chlorine adsorbed to the surface, and with dichlorophenyl phosphine no adsorption occurred, while the addition of sulfur to that same molecular structure resulted in the promotion of carbon and chlorine adsorption. Substitution of oxygen for sulfur in the dichlorobenzyl phosphine molecule resulted in carbon, chlorine, and oxygen adsorption. With none of the phosphorus containing molecules was phosphorus detected on the surface. Sliding in an atmosphere of benzyl chloride promoted adsorption of chlorine to the iron surface. Increases in load resulted in a decrease in the surface concentration of iron chloride.			
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INTERACTION OF SOME EXTREME-PRESSURE TYPE LUBRICATING COMPOUNDS WITH AN IRON SURFACE

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SUMMARY

An investigation was conducted to determine the interaction characteristics of four extreme-pressure type organic molecules with an iron surface. The molecular species were benzyl chloride, dichlorophenyl phosphine, dichlorophenylphosphine sulfide, and dichlorophenyl phosphine oxide. A sputter-cleaned iron surface was exposed to these materials statically and during dynamic friction experiments. Auger electron spectroscopy was used to monitor the surface chemistry.

Results of surface analysis indicate that benzyl chloride dissociates with only chlorine adsorbing to the iron surface. Dichlorophenyl phosphine does not adsorb, but the addition of sulfur to the molecule promotes the adsorption of both carbon and chlorine. Substitution of oxygen for sulfur in the molecule results in the adsorption of oxygen as well as carbon and chlorine. The labile nature of the chlorine in the molecule determines whether simply chlorine or an organic radical will adsorb. Sliding in an atmosphere of benzyl chloride promotes adsorption of chlorine to the iron surface from benzyl chloride. Increased loading after exposure to a fixed quantity of benzyl chloride causes a decrease in the amount of chloride film on the iron surface.

INTRODUCTION

Extreme-pressure lubricant additives are contained in a wide variety of oils. The number of compounds used as additives is even greater. Despite the extensive use of extreme-pressure additives to extend the use of oils, very little is really known about the fundamental chemical interactions of these materials with metal and alloy surfaces. Most of the studies on additive behavior that have been done are empirical in nature (ref. 1). The procedure appears to be to select a particular compound, examine its performance in some type of test machine, and then put it into use in an actual application. A better fundamental understanding of the mechanism of interaction of these materials with metal

surfaces may lessen the necessity for extensive experimental testing.

Some additives contain a single elemental species of the component that interacts with the metal surface to form the antiseizure film (refs. 2 to 4). Others contain two or more of the active elements sulfur, phosphorus, and chlorine (refs. 5 and 6). Even in those cases where only a single active element is involved oxygen from the environment is dissolved in the oil. In a strict sense oxygen is an extreme-pressure agent. The question which arises and requires resolution is what kinds of metal films form? Are they single compounds or do they comprise two or more compounds? Further, what is the mechanism by which these films form?

The objective of this investigation was to examine the nature of the initial interaction of some extreme-pressure type compounds with an iron surface. Auger spectroscopy was used to follow the interactions, and surfaces were examined for those interactions that occur with static as well as dynamic exposure (shearing or sliding friction). The effect of the films formed on friction coefficient was also measured.

MATERIALS

The disk specimens used in this study were prepared by vacuum casting 99.99-percent-pure iron into slabs slightly over disk size. The disks were then machined from these slabs, polished down to 600 grit on metallurgical papers, and finally diamond polished.

The rider specimens were high-purity aluminum oxide. During sputter-cleaning of the disk specimen, sputtering was continued until a clean layer of iron deposited on the aluminum oxide surface. Thus, an iron covered aluminum oxide rider was in sliding contact with an iron disk.

EXPERIMENTAL APPARATUS AND PROCEDURE

Specimens

The friction and wear specimens consisted of a disk specimen 6.5 centimeters in diameter and 1.2 centimeters thick and a hemispherical rider with a 0.5-centimeter radius. The specimens are shown in the apparatus schematic in figure 1. The disk specimen is mounted on a drive shaft which is rotated with a magnetic drive assembly. The drive assembly provides for rotation at various speeds (in this study, 20 cm/min). The rider specimen is mounted in an insulated holder to one end of a stainless-steel shaft. Friction and wear experiments are conducted with the rider specimen loaded against the

disk surface. As the disk is rotated, the rider scribes a circular wear track on the flat surface of the disk. The loads used in this investigation varied from 100 to 1000 grams, and the temperature was 23° C.

Experimental Chamber

The experiments are conducted in a vacuum chamber (see fig. 1). The vacuum system is pumped by sorption pumps and an ion pump. Pressure in the vacuum system is read with a nude ionization gage. The vacuum system achieved a pressure of 1×10^{-10} torr after bakeout at 250° C.

Measurements

The friction force between the disk and rider specimens is continuously recorded during the experiment. The beam which contains the rider specimen is welded into a bellows assembly which is gimbal mounted to the vacuum system. The gimbal mounting permits deadweight loading of the rider against the disk surface (fig. 1). At right angles to the deadweight loading, the beam containing the rider can move in two directions in the horizontal plane. Movement of the rider (with the disk as it rotates) is restrained by a cable which is attached to a beryllium-copper ring. The ring contains four sets of strain gages. These gages measure the frictional force between the disk and rider specimens. The friction force is recorded on a strip chart.

Specimen Preparation and Cleaning

The disk specimens are finish ground on metallurgical papers to a grit of 600. They are then diamond polished with 6-micrometer and finally 3-micrometer diamond paste. The disks are rinsed with acetone and then with absolute ethyl alcohol.

The specimens are cleaned by ion bombardment in the experimental chamber. The disk specimen is insulated from ground on the drive shaft. Two copper rods are brought to the disk from feed-throughs insulated from ground. The end of one rod terminates 0.5 centimeter from the circumferential edge of the disk specimen. This terminal establishes the positive potential in the glow discharge. The second rod has a beryllium-copper leaf attached to it. The end of the leaf opposite its attachment to the rod makes a wiping type of contact with the circumferential edge of the disk. The entire flat of the disk is cleaned by the sputtering as a result of being immersed in the glow discharge. The spec-

imens are ion-bombarded by bleeding research-grade argon gas into the system until a pressure of about 0.02 torr is achieved. A direct-current power supply is used to supply 400 to 500 volts between the disk and the floating electrode. With the negative potential on the disk positively charged, argon ions bombard and sputter clean the specimen surfaces. Variations in the argon pressure can alter the voltage necessary for efficient sputtering. The higher the pressure, the lower the voltage needed.

Auger Analysis

Elemental analysis of the disk specimen surface can be made before, during, and after the friction and wear experiment by using an Auger cylindrical mirror analyzer with an integral electron gun. The point of rider to disk contact passes under the Auger beam 20 seconds after the disk moves out of the contact zone. This time period can be reduced by increasing the speed at which the disk rotates. The disk could rotate over a broad range of speed, and Auger analysis could still be performed. The Auger analyzer is a commercial unit, the essential elements of which are described in the literature (refs. 7 and 8).

The primary beam of electrons is directed at the disk surface by a beam from the electron gun in the Auger cylindrical mirror analyzer. The beam is focused on the wear track scribed by the rider in sliding contact with the disk. The beam contact is 180° away from the rider on the disk surface. The beam spot diameter is 0.2 millimeter. The gun contains deflection plates which allow positioning of the beam on the disk surface.

The secondary electrons come off the specimen surface, pass through a cylindrical can opening, and then pass through slits in an inner cylinder which serves as an energy analyzer. They are collected by an electron multiplier. Elemental identification is accomplished by analysis of the detected secondary-electron energies. The Auger electrons that appear in the secondary-electron distribution "fingerprint" the surface elements to a depth of approximately four atomic layers.

Auger traces are displayed on an oscilloscope. The sweep control module permits a full-spectrum scan for those elements of interest in friction, wear, and lubrication in 0.10 second. Thus, there exists the capability of monitoring a moving or rotating surface and detecting dynamic variations in surface and detecting dynamic variations in surface chemistry.

RESULTS AND DISCUSSION

Benzyl Chloride

It has been established that the antiwear properties of chlorine-containing additives depend very strongly on the labile nature of the carbon-to-chlorine bond. The antiwear properties improve with decreasing strength of the carbon-to-chlorine bond (ref. 1). The chlorine atom in benzyl chloride is highly reactive since it is joined to the alkyl group rather than to the benzene ring. Though an alkyl-aryl compound, it is more reactive than the alkyl halide ethyl chloride (ref. 9). Thus, it is a good candidate for examination.

Since during the sliding or rubbing process associated with two metals in contact nascent metals are exposed, experiments were conducted with clean iron surfaces. The iron disk specimen was sputter-cleaned with argon ion bombardment. A typical Auger spectrum for a clean iron surface is presented in figure 2(a). There are four Auger energy peaks in the spectrum associated with iron, one low-energy and three high-energy peaks. There is a complete absence of any other elements on the iron surface.

The surface of figure 2(a) was exposed to 10^{-2} torr-second (10^4 langmuirs) of benzyl chloride. The resulting Auger spectrum is presented in figure 2(b). The same four iron peaks are present as in figure 2(a), and in addition there is a peak associated with the presence of chlorine on the surface. The chlorine comes from the benzyl chloride. Note in figure 2(b) that there is no indication of the presence of carbon on the surface. If carbon were present, an Auger peak would appear in the spectrum between the chlorine and the first of the three high-energy iron peaks.

Auger emission spectroscopy is particularly sensitive to the presence of carbon on surfaces. Very small amounts of carbon on an iron surface can be readily detected by Auger analysis (ref. 10). Thus, if carbon were present on the iron, it should have appeared in the spectrum of figure 2(b).

The absence of carbon on the iron surface indicates that benzyl chloride adsorbs dissociatively. The molecule dissociates with the chlorine remaining chemisorbed to the iron surface and the hydrocarbon portion of the molecule going into the vacuum system. Benzyl chloride needs only to contact a clean iron surface for iron chloride to form.

The amount of chlorine on the iron surface is a direct function of the exposure or concentration of benzyl chloride. This is evidenced by the data of figure 3. Results are presented in figure 3 for exposure to benzyl chloride, both static and during sliding. At each exposure a greater concentration of chlorine adsorbs on the surface during sliding. Sliding contact thus promotes dissociative adsorption.

The coefficient of friction continuously decreases with increasing exposure to benzyl chloride. This is seen in the friction data of figure 4. Thus, the more chlorine present on the iron surface, the lower the friction.

The data of figure 4 were obtained at a load of 100 grams. Chlorine when reacted with iron is purportedly a good extreme-pressure lubricant; that is, it is an effective boundary lubricant at heavy loads. In order to determine the tenacity of chloride films formed from benzyl chloride, experiments were conducted at various loads with a chloride film formed by exposure of iron to 10^{-2} torr-second (10^4 langmuirs) of benzyl chloride. Sliding was conducted repeatedly over the same track. Changes in Auger chlorine peak intensity with repeated passes over the same surface at three loads are presented in figure 5.

An examination of figure 5 indicates that a 100-gram load sliding over the same surface to 100 repeated passes does not change very notably the chlorine surface coverage. With a load of 300 grams, however, the Auger chlorine peak intensity continuously decreases with successive passes over the surface. After 100 passes the chlorine peak intensity is approximately one-half the intensity prior to the commencement of sliding.

If the load was increased from 300 to 1000 grams in the sliding friction experiments, even further reductions in chlorine peak intensity were observed, as indicated in the data of figure 5. At only 27 passes the Auger chlorine peak intensity is less than one-third the value obtained before the start of sliding. After 100 passes, while chlorine is still present on the surface, a considerable amount has been removed during sliding.

It should be indicated that the Auger beam spot diameter is 0.6 millimeter, and at the light load of 100 grams the beam is sampling not only the wear track but also a region to either side of it. Notwithstanding this fact, chlorine is still being removed from the surface, as evidenced by the Auger data of figure 5. At the heavier loads of 300 and 1000 grams a wear track will develop more rapidly.

There are three possible explanations for the reductions in chlorine peak intensity with sliding shown in figure 5. Any one or all three may be contributing to the reduction observed in chlorine peak intensity. First, sufficient interfacial energy may develop at the interface to cause dissociation of the iron chloride into iron and chlorine. This mode of chlorine removal appears unlikely because of the stability of iron chloride (ref. 11).

Second, chlorine removal may involve the burial of the chloride film by metallic iron. As load is increased and with repeated passes, an increasing amount of iron-to-iron contact occurs through the film. Elemental iron being relatively soft, it flows plastically very readily. With an increasing exposure of the area scanned by the primary electron beam consisting of iron there is an accompanying decrease in the amount of chloride being sampled.

Third, the film may simply be reduced in thickness with repeated passes. The sliding process is simply wiping chloride out of the contact zone. This latter mechanism appears to be the most plausible. Evidence for its being the operable mechanism responsible for the results shown in figure 5 is presented in figure 6.

In figure 6 the friction coefficient is presented for various loads. These data were

obtained during the first 100 passes over the surface. Despite the reduction in chlorine peak intensity shown in figure 5 with sliding at various loads, no change in friction coefficient was observed, as indicated in the data of figure 6. If the second proposed mechanism were responsible for the reduction in chlorine peak intensity, then some change (increase) in friction could be expected. None was observed, however.

Structures of Dichlorophenyl Phosphine, Dichlorophenyl Phosphine

Sulfide, and Dichlorophenyl Phosphine Oxide

Most commercial extreme-pressure additives employed industrially contain more than one active element in the molecular structure (refs. 5 and 6). A question which arises in the use of such additives is do all the active elements form films on the surface? In order to attempt to answer this question experiments were conducted with three organic structures, dichlorophenyl phosphine, dichlorophenyl phosphine sulfide, and dichlorophenyl phosphine oxide. The first molecule contains the active elements chlorine and phosphorus; the second chlorine, phosphorus, and sulfur; and the third, chlorine, phosphorus, and oxygen. The basic structure of these molecules and that of benzyl chloride are presented in figure 7.

An iron surface was exposed statically and during dynamic friction experiments to a total of 2×10^{-2} torr-second (2×10^4 langmuirs) of dichlorophenyl phosphine. No evidence of chlorine or phosphorus was detected on the surface.

Exposure of iron to dichlorophenyl phosphine sulfide resulted in adsorption with as little as 10^{-5} torr-second (10^1 langmuirs). Auger peak intensities are presented in figure 8 for those elements detected on the iron surface. It is of interest to note that only carbon and chlorine were detected on the surface. There was no evidence of the presence of phosphorus or sulfur with the least effective of the molecules examined. Again dissociative adsorption appears to have occurred, as was observed with benzyl chloride. Unlike benzyl chloride, however, sliding did not appear to influence adsorption of the chlorinated phenyl radical to the iron surface.

The data of figure 8 are particularly interesting in that they show that only one of the three active elements in the parent molecule is actually present on the surface. Further, it is present with the carbon of the phenyl group. With benzyl chloride only the chlorine remained on the surface.

The iron surface was next exposed to dichlorophenyl phosphine oxide. The Auger peaks detected are presented in figure 9. Three elements were detected on the iron surface: carbon, chlorine, and oxygen. Again, as with dichlorophenyl phosphine sulfide no sulfur or phosphorus was detected. At 10^{-6} -torr-second (10^0 -langmuir) exposure both carbon and chlorine were detected. Oxygen did not appear in the Auger spectrum until

the surface had been exposed to 10^{-3} torr-second (10^3 langmuirs) of dichlorophenyl phosphine oxide.

Iron oxides are thermodynamically much more stable than iron sulfide. This may account for the presence of oxygen on the surface in figure 9 and the absence of sulfur in figure 8.

In figure 9 the carbon Auger peak intensity increases linearly with exposure. No increase in the chlorine peak occurs beyond 10^{-5} torr-second (10^1 langmuirs). The chlorine is undergoing electron-induced adsorption. It must therefore be concluded that it is the carbon and not the chlorine that is bonding to the iron surface since in figure 3 with benzyl chloride there was no evidence for electron-induced desorption of the chlorine where chlorine adsorption was directly to iron.

The experimental results obtained in this investigation with benzyl chloride, dichlorophenyl phosphine sulfide, and dichlorophenyl phosphine oxide indicate that dissociative adsorption to an iron surface can occur with extreme-pressure organic molecules. The results also indicate that not all of the extreme-pressure agents in the molecule (e.g., sulfur in the dichlorophenyl phosphine sulfide) are present in the surface film. Further, with benzyl chloride the chlorine is bonded directly to the iron to form an iron chloride film. With the dichlorobenzyl phosphine sulfide it appears that bonding is by the phenyl group. Thus, molecular structure of the hydrocarbon portion of the organic molecule may play an important role in forming extreme-pressure lubricating films.

As mentioned in the section Benzyl Chloride, with benzyl chloride the chlorine is extremely reactive and therefore can be readily removed from the benzyl group by interaction with the iron. In contrast, the same chlorine attached directly to the benzene ring as it is in dichlorophenyl phosphine sulfide is relatively inactive and difficult to remove from the benzene ring.

Chlorine is a good extreme-pressure lubricant which will reduce both friction and wear (ref. 11). It is more desirable to have it bond to the iron surface than the carbon of the hydrocarbon portion of the molecule.

In three of the molecular structures examined in this study phosphorus was present. However, no phosphorus was ever detected on the iron surface.

SUMMARY OF RESULTS

The following results were obtained in an investigation in which iron surfaces were exposed to benzyl chloride, dichlorophenyl phosphine sulfide, and dichlorophenyl phosphine oxide statically and during sliding:

1. Benzyl chloride and dichlorophenyl phosphine sulfide adsorbed to a clear iron surface dissociatively. With benzyl chloride only chlorine was detected on the surface.

There was no evidence of the hydrocarbon portion of the molecule on the surface. With dichlorophenyl phosphine sulfide only chlorine and carbon were detected by Auger spectrometry. There was a complete absence of phosphorus and sulfur.

2. The position of the chlorine in the organic molecule determined whether a metal chloride film formed or another portion of the molecule bonded to the surface.

3. Removal of an unreplenished iron chloride film from a surface was load sensitive. An increase in load resulted in a decrease in chlorine concentration on the surface.

4. Sliding in an atmosphere of benzyl chloride promoted the dissociation of benzyl chloride and the subsequent adsorption of chlorine to an iron surface.

5. The presence of sulfur in the dichlorophenyl phosphine sulfide molecule promoted the adsorption of the chlorinated phenyl group to the iron surface since with dichlorophenyl phosphine no adsorption to iron occurred.

6. While sulfur did not bond to the iron surface with dichlorophenyl phosphine sulfide, oxygen from dichlorophenyl phosphine oxide did bond because of its strong affinity for iron.

7. Three of the molecules examined in this study contained phosphorus, yet phosphorus was never detected on the iron surface.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 17, 1973,
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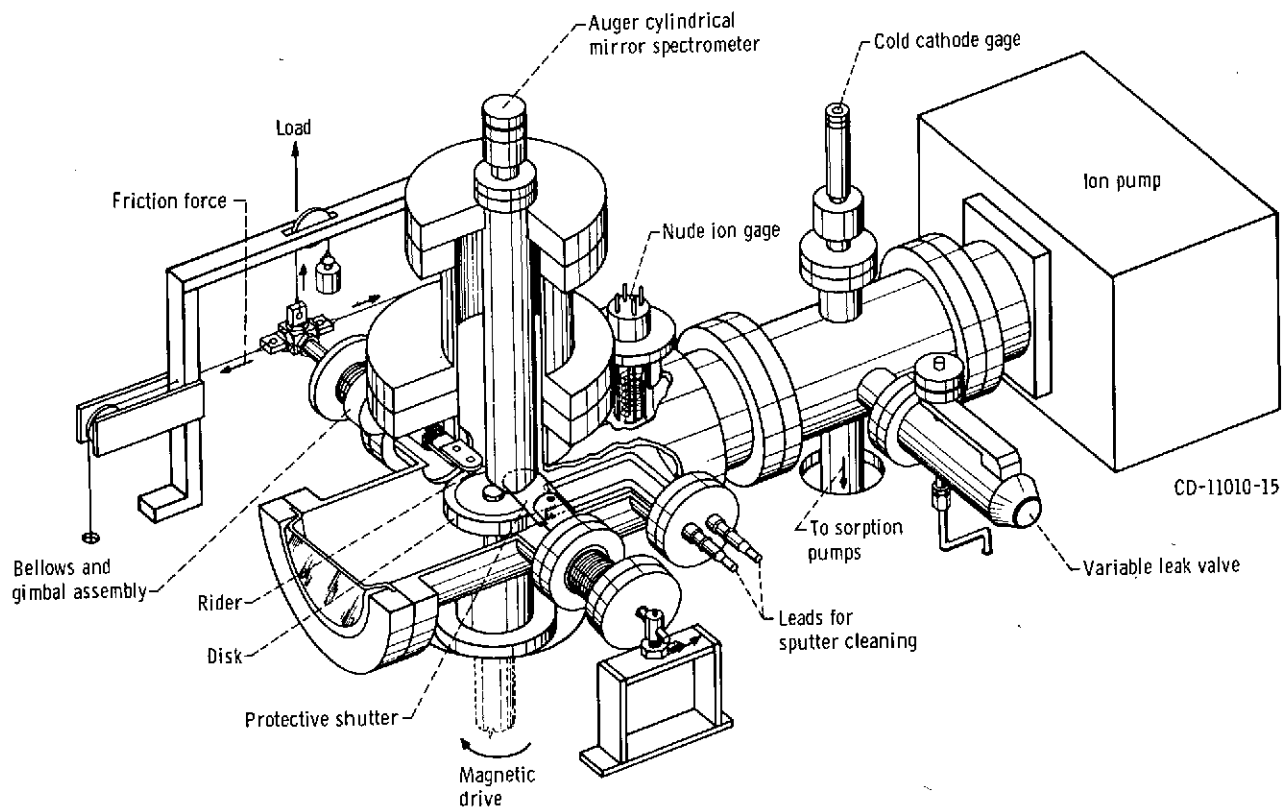
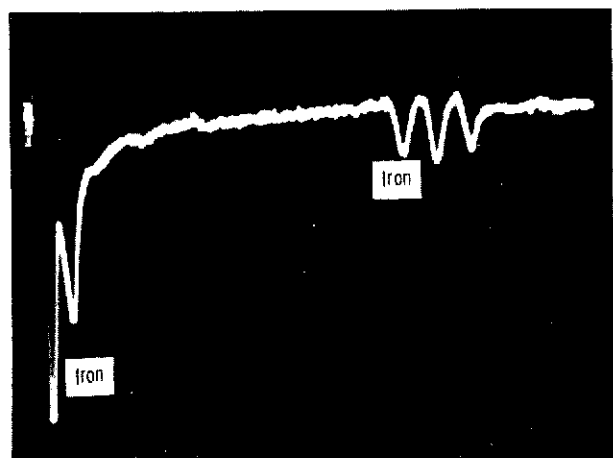
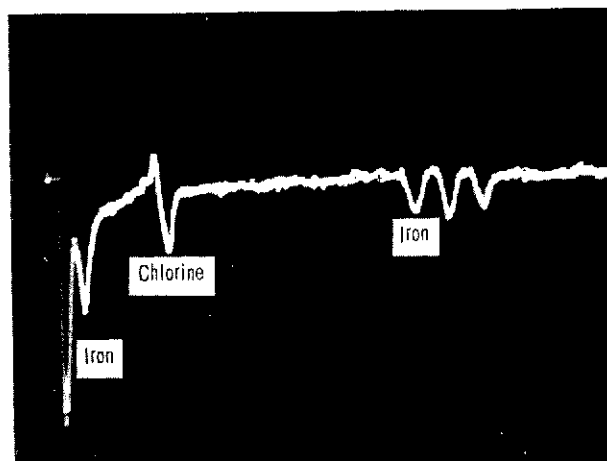


Figure 1. - Friction apparatus with Auger spectrometer.



(a) Sputter-cleaned iron.



(b) Iron exposed to 10^{-2} torr-second (10^4 langmuirs) of benzyl chloride.

Figure 2. - Auger spectra of clean and chloride-covered iron.

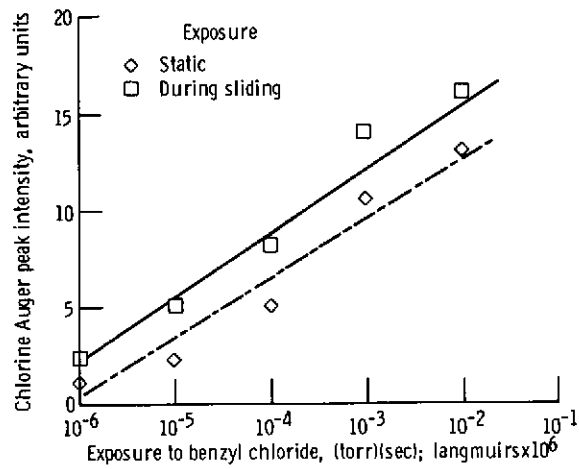


Figure 3. - Chlorine Auger peak intensity as function of iron exposure to benzyl chloride, both static and during sliding. Sliding velocity, 30 centimeters per minute; load, 100 grams; temperature, 23^o C.

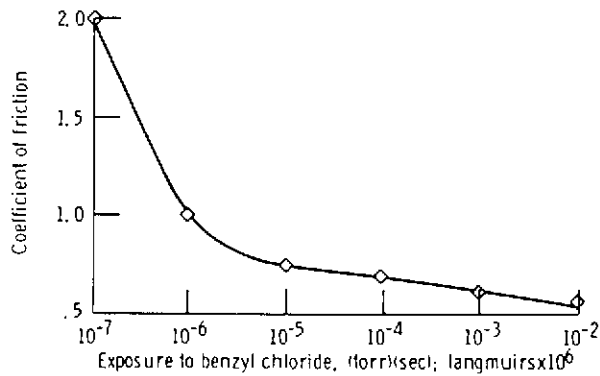


Figure 4. - Coefficient of friction as function of exposure to benzyl chloride for iron-coated aluminum oxide sliding on iron. Sliding velocity, 30 centimeters per minute; load, 100 grams; temperature, 23^o C.

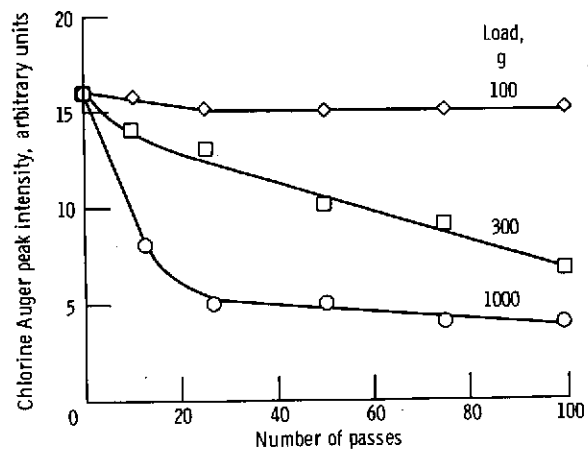


Figure 5. - Change in Auger chlorine peak intensity with number of repeated passes over same iron surface at various loads. Surface exposed to 10^{-2} torr-second (10^4 langmuirs) of benzyl chloride prior to sliding.

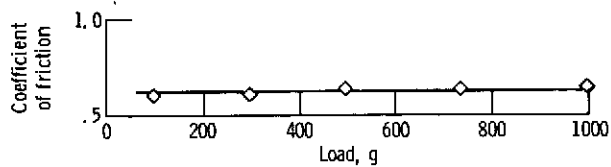


Figure 6. - Coefficient of friction as function of load for iron surface exposed to 10^{-2} torr-second (10^4 langmuirs) of benzyl chloride. Sliding velocity, 30 centimeters per minute; total of 100 passes; temperature, 23° C.

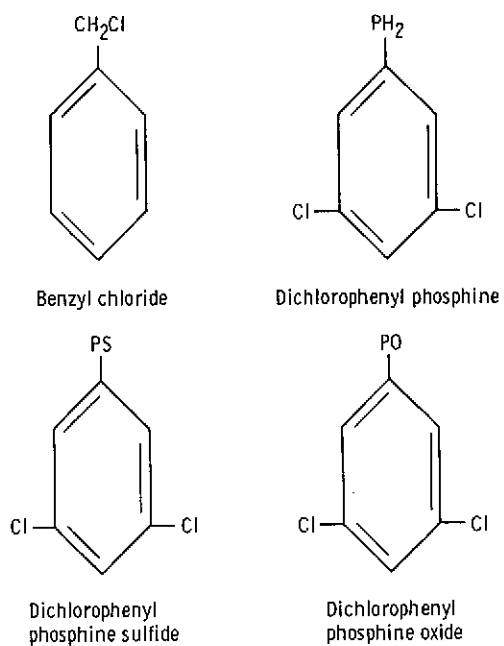


Figure 7. - Structure of additive compounds used in this study.

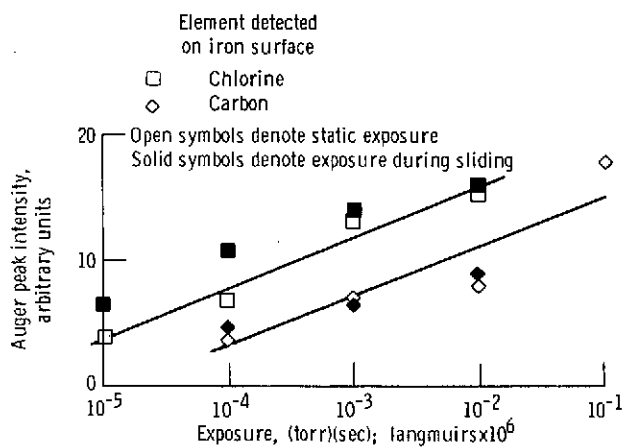


Figure 8. - Auger spectroscopy analysis of iron surface exposed to various amounts of dichlorophenyl phosphine sulfide.

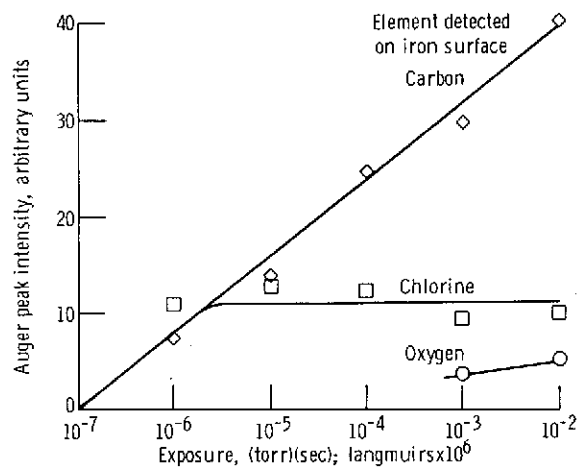


Figure 9. - Auger spectroscopy analysis of iron surface exposed to various amounts of dichlorophenyl phosphine oxide.