AUGER ANALYSIS OF OXYGEN AND SULFUR INTERACTIONS WITH VARIOUS METALS AND THE EFFECT OF SLIDING ON THESE INTERACTIONS

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Various gases were adsorbed to copper, aluminum, and chromium surfaces. The gases included oxygen, hydrogen sulfide, methyl mercaptan, and sulfur dioxide. Chemisorption was conducted on static surfaces and during dynamic friction experiments. An Auger cylindrical-mirror analyzer was used to monitor surface films. The sulfur containing gases adsorbed readily to all surfaces. Exposures of as little as $10^{-6}$ (torr)(sec) (1 langmuir) were sufficient to reduce friction. Sliding contact did not affect chemisorption to copper or aluminum but did affect chemisorption to chromium surfaces. Oxygen removed sulfur films from all surfaces at room temperature ($23^\circ$ C). Gaseous exposures were from $10^{-6}$ to $10^{-2}$ (torr)(sec) (1 to $10^4$ langmuirs).
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SUMMARY

An investigation was conducted to determine the chemisorption of various gases to copper, aluminum, and chromium surfaces. The gases were oxygen, hydrogen sulfide, methyl mercaptan, and sulfur dioxide. They were chemisorbed at room temperature and exposures to $10^{-2}$ torr-second ($10^4$ langmuirs) both statically and during dynamic friction experiments. Once sulfide films were adsorbed, they were exposed to oxygen.

Results of these studies indicate that sulfur-containing gases will adsorb readily to all surfaces. Methyl mercaptan adsorbs dissociatively, and only sulfur remains on the surface. Oxygen removes sulfur from sulfide films at room temperature. Surface exposures to sulfur-containing gases of as little as $10^{-6}$ torr-second (1 langmuir) are sufficient to reduce the friction coefficient for sputter-cleaned materials. Sliding alters the chemisorption of all gases to chromium but not to copper or aluminum surfaces.

INTRODUCTION

Sulfur is one of the most frequently employed extreme-pressure additives in lubricants. The exact nature of the protective film formed on metal surfaces is not fully understood. On steel surfaces, iron sulfide has been observed by some investigators (refs. 1 to 4), while others find no evidence for sulfide films (refs. 5 and 6). A recent study indicates that oxygen can replace sulfur from an iron surface (ref. 7). Thus, for steels, the extreme pressure films may, in reality, consist of both sulfides and oxides.

In addition to steels, there are a host of nonferrous alloys that are employed in practical lubrication systems. These include copper- and aluminum-base alloys. Further, there are alloying elements which segregate at the surface and thereby control the chemistry of surface films formed (ref. 8). One such element is chromium. In austenitic stainless steels, this particular element is found on the surface of the alloys.
The objective of this investigation was to examine the formation of sulfide films on copper, aluminum, and chromium and the effect of oxygen on such films. Further, the influence of sliding during friction experiments on surface reactions was explored. Surface chemistry and changes therein were examined by Auger spectroscopy. Sliding friction experiments were conducted with a hemispherical rider specimen (metal-coated alumina) sliding on a flat disk surface. Disks of aluminum, copper, and a chromium film on iron were examined. Surface films were formed on these disks by using hydrogen sulfide, methyl mercaptan, sulfur dioxide, and oxygen.

MATERIALS

The copper and aluminum disks used in this investigation were fabricated from 99.999 percent pure zone-refined materials. The surfaces were polished with metallurgical papers down to 600 grit and then polished with 3-micrometer diamond paste to produce a mirrorlike surface.

All of the gases used were research grades. The oxygen contained 5 ppm nitrogen, 16 ppm argon, 8 ppm krypton, and 9.4 ppm methane. The hydrogen sulfide, methyl mercaptan, and sulfur dioxide were 99.6, 99.5, and 99.98 percent pure, respectively. The rider specimens were polycrystalline alumina with a sputter-deposited thin film of the disk material (10^{-7} m; 1000 Å).

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 by a magnetic drive assembly. The drive assembly provides various linear velocities (in this study, 30 cm/min). The rider (in this study, aluminum oxide) is mounted in a holder to one end of a stainless-steel shaft. Friction and wear experiments are conducted with the rider 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 load used in this investigation was 100 grams, and the temperature was 23° C.
Experimental Chamber

The experiments were 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 cold-cathode ionization gage. The vacuum system achieved a pressure of $10^{-10}$ torr after bakeout at 250° C. All friction experiments were conducted at this pressure.

Measurements

The friction force between the disk and rider specimen is continuously recorded during friction experiments. 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 strain-gage assembly. This assembly measures the frictional force between the disk and rider specimen. The friction force is then recorded on a strip chart.

Specimen Cleaning

The disk specimens are cleaned by ion bombardment in the experimental chamber. The disk specimen is insulated from ground. 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. This terminal establishes the positive potential in the glow discharge. The second rod has a beryllium-copper leaf attached to it which contacts the disk. The entire flat of the disk is cleaned by the sputtering as a result of being immersed in the glow discharge. The specimens 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 1000 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.

During sputter cleaning of the disk, the aluminum oxide rider remains exposed to the disk. Thus, sputtered metal from the disk deposits on the aluminum oxide rider, and the result is that during sliding a thin film of the metal is rubbing against itself.
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 contact of the rider on the disk passes under the Auger beam 20 seconds after the disk moves out of the contact zone. The disk was rotated to produce a sliding velocity of 30 centimeters per minute. 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.

A beam of electrons is directed at the disk surface by 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 150° away from the rider on the disk surface. The beam spot diameter is 0.2 millimeter. The gun contains deflection plates which allow positioning the beam on the disk surface. The beam current was 15 milliamperes, and the applied voltage was 2000 volts.

The secondary electrons come off the specimen surface and pass through the energy analyzer. They are collected by an electron multiplier. Elemental identification is accomplished by determination of the detected Auger-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 chemistry.

RESULTS AND DISCUSSION

Oxygen

In reference 7 the Auger peak intensities for oxygen are plotted as a function of oxygen exposure for iron. A linear increase of oxygen peak intensity was detected as a function of exposure. Similar results were obtained in this investigation with aluminum, and the results obtained are presented in figure 2 together with those for iron from reference 7.

An examination of figure 2 indicates that, while there is an increase in oxygen uptake with exposure to $10^{-2}$ torr-second ($10^4$ langmuirs), the total surface concentration is less than that observed for iron. As with iron, sliding does not alter adsorption characteristics. In this study by definition only chemisorption is involved.
The results of figure 2 indicate that a greater quantity of oxygen is present on an iron surface than on an aluminum surface for a given oxygen exposure. This, however, does not indicate anything about the stability or kinetics of formation of the respective metal oxides. It is well known that the oxides of aluminum are thermodynamically more stable than those of iron (ref. 9). The results of figure 2 do indicate that iron will adsorb a greater quantity for a given exposure.

Oxygen was also chemisorbed on a chromium film present on an iron surface. Data obtained for the adsorption characteristics were very similar to those for aluminum.

With copper, exposure of the surface to $10^{-2}$ torr-second ($10^4$ langmuirs) of oxygen resulted in no evidence for oxygen chemisorption. This was the case for both static and dynamic experiments.

Oxygen adsorption results indicate that iron, aluminum, and chromium all adsorb oxygen readily, while copper does not. Sliding has no effect on adsorption. In an earlier study, sliding was found to affect the adsorption of certain hydrocarbons (ref. 10). The effects accompanying sliding, that is, heating and strain, can affect adsorption.

Hydrogen Sulfide

An earlier study with iron (ref. 7) indicated that hydrogen sulfide chemisorbs very readily to iron. The adsorption process is dissociative, and only sulfur remains on the iron surface.

Similar chemisorption experiments were conducted in this investigation with copper, aluminum, and chromium surfaces. In figure 3(a) an Auger spectrum is presented for a clean copper surface after sputtering. In figure 3(b) the Auger spectrum for the same surface is shown after adsorption of $10^{-2}$ torr-second ($10^4$ langmuirs) of hydrogen sulfide. The copper Auger peaks appear in figure 3(a), but those same peaks are completely masked by the sulfur in figure 3(b) at these energies.

The chemisorption of hydrogen sulfide to copper and aluminum as a function of exposure, both static and during a dynamic friction experiment, was monitored with Auger spectrometry, and the results obtained are presented in figure 4. Reference data for iron are also presented in figure 4. Just as was observed with iron in reference 7, sliding did not affect adsorption.

In figure 4, iron exhibits the greatest amount of sulfur on its surface and aluminum the least. Aluminum forms a dense thin tenacious oxide when exposed to oxygen. A similar effect may exist with sulfur; that is, a film once formed may inhibit film growth. Both iron and copper form relatively porous oxides, and again by analogy, their sulfides may be porous and allow for easy film growth beyond a monolayer.

The friction coefficients for iron, aluminum, and copper at various hydrogen sulfide exposures are presented in figure 5. The friction coefficients do not appear to be related
to the sulfide coverage reflected by the data of figure 4. In figure 5, iron (the metal with the greatest amount of sulfur) exhibits the highest friction coefficient. Copper, with the intermediate film coverage in figure 4, has the lowest friction coefficient in figure 5. The curve shapes are, however, similar. Thus, even in very thin film form, the property of the particular metal sulfide plays an important role in friction behavior.

The iron and aluminum sulfide films are still increasing in thickness at a hydrogen sulfide exposure of $10^{-2}$ torr-second ($10^4$ langmuirs), and yet no changes in friction are detected beyond $10^{-5}$ torr-second (10 langmuirs). With iron, the significant reduction in friction occurs with the first exposure to hydrogen sulfide.

While sliding did not affect the chemisorption of hydrogen sulfide to iron, copper, and aluminum, it did affect chemisorption to chromium. This effect is shown in the data of figure 6. In figure 6, a greater quantity of sulfur is present on the surface during static chemisorption than during sliding. This could be due to frictional heating inhibiting adsorption, the enhanced tendency for desorption due to this same heating, or the rubbing away of the film formed during the sliding process.

In figure 6 at an exposure of $10^{-6}$ torr-second (1 langmuir), there was considerably more sulfur present on the chromium surface than was observed in figure 4 with copper and aluminum.

Copper in these studies exhibited interesting behavior in that, even at $10^{-2}$-torr-second ($10^4$-langmuir) exposure to oxygen, no evidence for oxygen chemisorption was observed. Yet, with hydrogen sulfide, chemisorption was detected with as little exposure as $10^{-6}$ torr-second (1 langmuir). These results would indicate that copper has a greater affinity for sulfur than it has for oxygen. While the thermodynamic stability of a metal oxide is generally greater than that for the sulfide of the same metal, with copper their stabilities are comparable.

Sliding under equivalent mechanical conditions resulted in an effect upon hydrogen sulfide chemisorption to chromium but no effect upon adsorption to iron, copper, or aluminum. Chromium is chemically very active. Despite this fact, less sulfur was observed on chromium during sliding for a given exposure to hydrogen sulfide than was observed without sliding.

**Methyl Mercaptan**

Sulfur additives present in lubricating oils are usually there as organic compounds. Chemisorption studies were therefore conducted with the simple sulfur-containing organic molecule methyl mercaptan. Methyl mercaptan ($\text{CH}_3\text{SH}$) was adsorbed to copper, aluminum, and chromium surfaces.

Figure 7 presents Auger sulfur peak intensities for adsorption of methyl mercaptan to copper. Three observations can be made. First, there was no difference at lower
exposures between dynamic and static experiments. Second, only sulfur was present on the surface; no carbon was detected. Just as in studies with iron, the methyl mercaptan adsorbed dissociatively, and the methyl group was not retained on the surface.

Third, the amount of sulfur present on the surface at any given exposure with methyl mercaptan was less than was present with hydrogen sulfide on the same surface. Thus, even though the methyl group was not retained in the film, its presence in the molecule inhibited sulfur adsorption.

Similar chemisorption experiments were conducted with methyl mercaptan on an aluminum surface, and results obtained are presented in figure 8. As with the iron and copper surfaces, the adsorption of the methyl mercaptan occurred dissociatively, and only the sulfur remained on the surface. There was no evidence of carbon.

Auger spectra for a clean aluminum surface and the same surface after exposure to methyl mercaptan are presented in figure 9. The clean surface (fig. 9(a)) exhibits a single peak, namely, that of aluminum. After exposure to methyl mercaptan, both sulfur and aluminum peaks are detected. The aluminum peak height has been reduced in size because of the presence of sulfur on the aluminum (fig. 9(b)). No carbon peak was detected.

Chemisorption results for methyl mercaptan on a chromium surface are presented in figure 10. Some differences between static adsorption and adsorption during sliding appear to exist. The data points during sliding fall consistently below those for static adsorption. The differences are, however, not as great as those observed for hydrogen sulfide on a chromium surface.

**Oxygen Displacement of Sulfide Films**

**Copper.** - After the surfaces of copper specimens were covered with sulfide films originating from methyl mercaptan and hydrogen sulfide, these same surfaces were exposed to various amounts of oxygen. Since oxygen is almost always present in practical lubrication systems, its effect on sulfide films was sought. The results obtained are presented in figure 11.

In figure 11 the Auger peak intensity for sulfur is plotted as a function of oxygen exposure. The first observation to be made is that, although a sulfide film is present on the surfaces, the source of the sulfur does not appear to affect its removal. The hydrogen sulfide generated film and the film originating from methyl mercaptan were both removed equally easily by oxygen. Second, there is a continuous decrease in the amount of sulfur with increasing quantities of oxygen.

It should be indicated that oxygen removed sulfur from the surface. Auger spectra indicated no evidence for the adsorption of oxygen to the surface. Earlier studies with
iron indicated, with mass spectrometry, that sulfur monoxide and sulfur dioxide were being formed and passing into the vacuum system. The same mechanism undoubtedly operates with copper.

It is of interest to examine the effect of sulfide film removal on friction. In figure 12 friction coefficient is plotted as a function of copper exposure to hydrogen sulfide and hydrogen sulfide film removal by oxygen. With increasing exposure to hydrogen sulfide, there is a continuous corresponding decrease in friction coefficient.

After the surface was exposed to $10^2$ torr-second ($10^4$ langmuirs) of hydrogen sulfide (fig. 12(a)), it was exposed to varying amounts of oxygen (fig. 12(b)). The effect of oxygen on friction is seen in the curve of figure 12(b). As sulfur is removed (see fig. 11), the friction coefficient increases.

**Aluminum.** - In figure 13 data are presented for the effect of oxygen on a sulfide film present on aluminum. Hydrogen sulfide was the source for the sulfide film. Oxygen removed the sulfide film just as it had done with copper. The amount of sulfur removed was enhanced by the sliding process.

When the source gas for the sulfide film on the aluminum surface was methyl mercaptan rather than hydrogen sulfide, the removal by oxygen was not the same. This is evidenced by the data of figure 14, which should be compared with the data of figure 13. Static exposure to oxygen did not displace the sulfide film in figure 14. Only with sliding (fig. 14) was a decrease in sulfur peak intensity observed.

**Chromium.** - Figure 15 presents data for the effect of oxygen on the removal of a sulfide film originally generated on a chromium surface from two sources, hydrogen sulfide and methyl mercaptan. With both film sources, oxygen removed sulfur. Just as was observed with copper, the sulfide film was more readily removed when the film was formed from methyl mercaptan.

In general, it has been observed in this investigation with copper, aluminum, and chromium and in reference 7 with iron that a sulfide film can be removed from these surfaces by oxygen. In the study of reference 7 after exposure to $10^{-2}$ torr-second ($10^4$ langmuirs) of oxygen, the sulfide film was completely removed from the iron surface. All of this removal occurred at 23° C. Thus, in practical lubrication systems where oxygen concentrations are sufficient, sulfide films can be removed or displaced by oxygen even at room temperature.

Another general observation to be made from the data with chromium (fig. 15) is that, although sulfide films resulted from exposure to two different source materials, namely, hydrogen sulfide and methyl mercaptan, susceptibility to static oxygen removal was different. Sulfur originating from hydrogen sulfide was easier to remove. In both instances, however, sulfur was still present on the surface after exposure to $10^{-2}$ torr-second ($10^4$ langmuirs) of oxygen.
In general, the process of sliding appears to promote oxygen removal of sulfide films. Friction coefficient decreases with increasing surface concentration of sulfur, while its removal by oxygen results in an increase in friction.

Sulfur Dioxide

Since oxygen displaces sulfur from metal surfaces, the question arises as to what type of adsorption would occur if both oxygen and sulfur were present in the same molecular structure. Is the adsorption dissociative, or does the entire molecule containing both sulfur and oxygen adsorb? Sulfur dioxide was adsorbed to copper, aluminum, and chromium surfaces.

With sulfur dioxide, adsorption was molecular in nature. Both oxygen and sulfur were detected on all metal surfaces. Figure 16 is an Auger spectrum for sulfur dioxide adsorbed to an aluminum surface during a sliding friction experiment. In addition to aluminum, both sulfur and oxygen peaks are detected. These results would indicate that adsorption of the sulfur dioxide is molecular in nature.

Figure 17 presents Auger peak intensities for sulfur as a function of exposure of chromium to sulfur dioxide. Just as with hydrogen sulfide and methyl mercaptan, sliding made a difference in adsorption behavior. With both hydrogen sulfide and methyl mercaptan, less sulfur was observed on the surface when adsorption was conducted during sliding than in the absence of sliding. With sulfur dioxide, the reverse was true. A greater amount of sulfur was detected on the surface during sliding than was observed in its absence.

Sliding results in heating and straining of metal surfaces. Both of these events tend to promote chemical reaction. Where only adsorption is involved, heating enhances desorption. The results in figure 17 therefore indicate that chemical reaction occurs at the metal surface with sulfur dioxide.

Adsorption to a chromium surface is influenced by the sliding process. The effect of sliding is dependent upon the adsorbing species. With some species, for example, hydrogen sulfide and methyl mercaptan, less sulfide film is observed than for static adsorption. In contrast with sulfur dioxide, sliding actually promoted sulfur adsorption. Thus, the effect of sliding is selective and determined by the adsorbing species.

SUMMARY OF RESULTS

From adsorption studies with oxygen, hydrogen sulfide, methyl mercaptan, and sulfur dioxide at exposures to $10^{-2}$ torr-second ($10^3$ langmuirs) of various metals (copper, aluminum, and chromium), the following results were obtained:
1. The sulfur-containing gases hydrogen sulfide and methyl mercaptan adsorbed very readily to all metal surfaces. Film coverage continued to increase with increasing exposure to $10^{-2}$ torr-second ($10^4$ langmuirs) of each of the gaseous species.

2. Sliding friction had no effect upon the adsorption of gases to copper, aluminum, or iron surfaces. With chromium, however, adsorption was affected by sliding. Film coverage at a particular exposure increased or decreased during sliding over that seen in static adsorption depending upon the particular adsorbing species. In some cases coverage was greater, while in others it was less.

3. Oxygen removed sulfur from all surfaces at room temperature ($23^\circ$ C) except for a sulfide film formed from methyl mercaptan on an aluminum surface. This occurred despite the source of the sulfur. The amount of sulfur removed was, however, dependent upon the source of the sulfur for one metal, chromium. The amount removed from chromium was less when the source was hydrogen sulfide than when it was methyl mercaptan.

4. Exposure to as little as $10^{-6}$ torr-second (1 langmuir) of a gas such as hydrogen sulfide was sufficient to reduce friction from that of the sputter-cleaned state of the metal surface for all metals examined except copper. With oxygen removal of a sulfide film from a surface, there was a continuous increase in friction with increasing exposure to oxygen.

5. Oxygen did not adsorb to a sputter-cleaned copper surface at exposures to $10^{-6}$ torr-second ($10^4$ langmuirs).

6. Methyl mercaptan adsorbed dissociatively to all the metals studied, and only sulfur remained on the surfaces.

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Figure 1. Friction apparatus with Auger spectrometer.

Figure 2. Auger spectroscopy detection of oxygen after exposure of two metals to various amounts of oxygen. Both stationary and sliding disks were exposed to oxygen; surfaces were sputter-cleaned prior to adsorption. Conditions for sliding friction experiments: sliding velocity, 30 centimeters per minute; load, 200 grams; ambient temperature, 23°C.
(a) Clean copper.

(b) Same surface after exposure to $10^{-2}$ torr-second ($10^4$ langmuirs) of hydrogen sulfide on copper.

Figure 3. - Auger spectra for sputter-cleaned copper surface and same surface after exposure to $10^{-2}$ torr-second ($10^4$ langmuirs) of hydrogen sulfide.
Figure 4. - Auger spectroscopy detection of sulfur after adsorption of hydrogen sulfide to metal surfaces. Both stationary and sliding disks were exposed to hydrogen sulfide; copper surface was sputter-cleaned prior to adsorption. Conditions for sliding friction experiments: sliding velocity, 30 centimeters per minute; load, 100 grams; ambient temperature, 23°C.

Figure 5. - Coefficient of friction for various sputter-cleaned metal surfaces exposed to hydrogen sulfide. Rider specimen was aluminum oxide. Sliding velocity, 30 centimeters per minute; load, 100 grams; ambient temperature, 23°C; ambient pressure, 10^-10 torr.
Figure 6. - Auger spectroscopy detection of sulfur after adsorption of hydrogen sulfide to chromium surface. Both stationary and sliding disks were exposed to hydrogen sulfide; chromium surface was sputter-cleaned prior to adsorption. Conditions for sliding friction experiments: sliding velocity, 30 centimeters per minute; load, 100 grams; ambient temperature, 23°C.

Figure 7. - Auger spectroscopy detection of sulfur after adsorption of methyl mercaptan to copper surface. Both stationary and sliding disks were exposed to methyl mercaptan; copper surface was sputter-cleaned prior to adsorption. Conditions for sliding friction experiments: sliding velocity, 30 centimeters per minute; load, 100 grams; ambient temperature, 23°C.
Figure 8. Auger spectroscopy detection of sulfur after adsorption of methyl mercaptan to aluminum surface. Both stationary and sliding disks were exposed to methyl mercaptan; aluminum surface was sputter-cleaned prior to adsorption. Conditions for sliding friction experiments: sliding velocity, 30 centimeters per minute; load, 100 grams; ambient temperature, 23°C.
Figure 9. - Auger spectra for sputter-cleaned aluminum surface and same surface after exposure to $10^{-2}$ torr-second ($10^4$ langmuirs) of methyl mercaptan.
Figure 10. - Auger spectroscopy detection of sulfur after adsorption of methyl mercaptan to chromium surface. Both stationary and sliding surfaces were exposed to methyl mercaptan; chromium surface was sputter-cleaned prior to adsorption. Conditions for sliding friction experiments: sliding velocity, 30 centimeters per minute; load, 100 grams; ambient temperature, 23°C.

Figure 11. - Auger spectroscopy detection of sulfur from hydrogen sulfide and methyl mercaptan and its displacement by exposure to oxygen. Disk surface was copper.
Figure 12. - Friction behavior of sulfide film on copper surface before and after exposure to oxygen. Sliding velocity, 30 centimeters per minute; load, 100 grams; ambient temperature, 23° C; ambient pressure, $10^{-10}$ torr.

Figure 13. - Auger spectroscopy detection of displacement of sulfide film from aluminum surface by oxygen. Original sulfide film was formed from methyl mercaptan with total exposure of $10^{-6}$ torr-second ($10^6$ langmuirs).
Figure 14. - Auger spectroscopy detection of sulfide film on aluminum surface after exposure to oxygen. Sulfide film preformed by exposing clean aluminum surface to $10^{-2}$ torr-second ($10^4$ langmuirs) of methyl mercaptan. Sliding velocity, 30 centimeters per minute; load, 100 grams; ambient temperature, 23° C; ambient pressure, $10^{-10}$ torr.

Figure 15. - Auger spectroscopy detection of preformed sulfide films on chromium surface with various exposures to oxygen. Two sulfide films were preformed, one from hydrogen sulfide and one from methyl mercaptan; total exposure to sulfur-containing gas, $10^{-2}$ torr-second ($10^4$ langmuirs).
Figure 16. - Auger spectrum for sulfur dioxide adsorbed on aluminum surface. Sputter-cleaned surface was exposed to $10^{-2}$ torr-second (10 langmuirs) of sulfur dioxide during sliding.

Figure 17. - Auger spectroscopy detection of sulfur after adsorption of sulfur dioxide to chromium surface. Both stationary and sliding surfaces were exposed to sulfur dioxide; chromium surface was sputter-cleaned prior to adsorption. Conditions for sliding friction experiments: sliding velocity, 30 centimeters per minute; load, 100 grams; ambient temperature, 23° C.
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—National Aeronautics and Space Act of 1958

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