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FRICTION-INDUCED SURFACE ACTIVITY OF SOME HYDROCARBONS WITH CLEAN AND OXIDE-COVERED IRON

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16. Abstract

Sliding friction studies were conducted on a clean and oxide-covered iron surface with exposure of that surface to various hydrocarbons. The hydrocarbons included ethane, ethylene, ethyl chloride, methyl chloride, and vinyl chloride. Auger cylindrical mirror analysis was used to follow interactions of the hydrocarbon with the iron surface. Results with vinyl chloride indicate friction induced surface reactivity, adsorption to surface oxides, friction sensitivity to concentration and polymerization. Variation in the loads employed influence adsorption and accordingly friction. In contrast with ethyl and vinyl chloride, friction induced surface reactivity was not observed with ethane and ethylene.

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FRICTION-INDUCED SURFACE ACTIVITY OF SOME HYDROCARBONS WITH CLEAN AND OXIDE-COVERED IRON

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SUMMARY

An investigation was conducted with the adsorption of some simple and substituted hydrocarbons to a clean and oxide-covered iron surface. Adsorption was studied with and without sliding. Auger analysis was used to follow surface interactions. The hydrocarbons examined included ethane, ethylene, methyl chloride, ethyl chloride, and vinyl chloride. Sliding friction experiments were made with an aluminum oxide rider sliding on an iron disk surface both in the clean state and with normal oxides present.

Results of the study indicate that friction initiates surface reactivity of both ethyl and vinyl chloride with iron. Vinyl chloride will adsorb to both clean and oxide-covered surfaces. Polymerization of vinyl chloride appears to occur on an iron surface with sliding. Adsorption and friction with vinyl chloride are load sensitive. Friction induced surface reactivity is not observed with ethane and ethylene.

INTRODUCTION

The interaction of hydrocarbons with iron and iron base alloys is of fundamental interest because the vast majority of practical lubrication systems involve such interactions. Adsorption and chemical interaction of hydrocarbons with iron have been examined by various investigators (refs. 1 to 4). These investigations have not, however, employed surface tools (e.g., Auger) to define the nature of the iron surface prior to adsorption or reaction with the hydrocarbons. Further, these experiments have been of adsorption and reaction with "static" iron surfaces.

In practical lubrication systems the adsorption and reactions of hydrocarbons with iron surfaces occur with those surfaces in sliding, rolling, or rubbing contact. Mechanical and metallurgical changes that take place during such motion can alter adsorption and reactions.

The objectives of this investigation were to study the adsorption and reaction of some simple and substituted hydrocarbons to and with an iron surface during dynamic friction experiments using cylindrical mirror Auger analysis to define the surface. Further, the role of friction in the initiation of polymerization of hydrocarbons was studied by the same technique. Surface chemistry and changes therein were continuously monitored during sliding.

Sliding friction experiments were conducted with a hemispherical rider of aluminum oxide sliding on an iron disk. Aluminum oxide was selected as a rider to minimize destruction of the surficial layers (surface layers) of the iron disk. The amount of surface disruption is much less than it would be with an iron rider. A cylindrical mirror Auger spectrometer was focused into the wear track for elemental analysis. The hydrocarbons examined included ethane, ethylene, methyl chloride, ethyl chloride, and vinyl chloride. The disk specimen was rotated at a sliding speed of 30 centimeters per minute and loads from 100 to 800 grams were used.

EXPERIMENTAL APPARATUS AND PROCEDURE

Experimental Chamber

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

Specimens

The friction and wear specimens consisted of a disk specimen 6.5 centimeters in diameter and 1.2 centimeters in thickness and a hemispherical rider with a 0.5-centimeter radius. The specimens are shown in the apparatus schematic in figure 1(a). 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, 30 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 were 100 to 800 grams, and the temperature was 23° C.

Measurements

The friction force between the disk and rider specimen is continuously recorded during the experiment. The beam which contains the rider specimen is welded in 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(a)). 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 temperature compensated strain gage. These gages measure the frictional force between the disk and rider specimen. 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 (micron) and finally 3-micrometer (micron) diamond paste. The disks are rinsed with acetone and then with absolute ethyl alcohol.

The rider specimens were acid cleaned prior to use with aqua regia to remove metal from finishing. They were then scrubbed with levigated alumina, rinsed in water and finally in ethyl alcohol.

The specimens are cleaned by argon 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 specimens are ion bombarded by bleeding research-grade argon gas into the system until a pressure of about 0.02 torr is achieved. A dc 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. Sputtering was continued until only iron peaks were detected by Auger spectroscopy.

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 12 seconds after that point moves out of the contact zone. The Auger analyzer is a commercial unit, the essential elements of which are described in the literature.

The primary beam of electrons is directed at the disk surface by a beam from the electron gun in the Auger cylindrical mirror analyzer (fig. 1(b)). 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 the outer cylindrical can opening (see fig. 1(b)), and then pass through slits in an inner cylinder which serves as an energy analyzer. They are collected by the electron multiplier. Elemental identification is accomplished by analysis of the detected secondary-electron energies. The Auger electrons that appear in the secondary-electron distribution chemically identify 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 0.10 second. Thus, there exists the capability of monitoring a moving or rotating surface and detecting dynamic variations in surface chemistry. In this investigation, surfaces were examined before, during, and after sliding.

RESULTS AND DISCUSSION

An iron disk surface was examined with the Auger spectrometer with the normal oxides and adsorbates present on its surface. A typical Auger spectrum is presented in figure 2(a). The elements sulfur, carbon, oxygen, and iron were detected. The carbon can originate from two sources, (1) adsorbed carbon monoxide and/or carbon dioxide and (2) as an iron impurity. The oxygen can result from adsorbates and iron oxide.

Argon ion sputter cleaning of the iron disk surface resulted in the Auger spectrum of figure 2(b). Examination of figure 2(b) reveals only peaks for iron. The surface is free of oxygen, carbon, and sulfur.

Auger peak heights were measured from the oscilloscope photographs directly. In all experiments, Auger primary beam voltage and current were held constant so as to allow for peak height comparison. Thus, based upon the pattern of figure 2(b) the iron disk surface is atomically clean.

Methyl Chloride

The substituted hydrocarbon methyl chloride, CH_3Cl, was selected for adsorption to a clean iron surface. It has only a single carbon atom and contains chlorine, a well known constituent of extreme pressure lubricant additives (ref. 5). The iron surface was exposed to various concentrations of methyl chloride. Exposure was in Langmuirs. One Langmuir is an exposure at a pressure of 10^{-6} torr for 1 second.

In figure 3 an Auger spectrum is presented for a clean iron surface after being exposed to 10^4 Langmuirs of methyl chloride. There are, in addition to the iron peaks, Auger peaks for chlorine and carbon. Methyl chloride adsorbs very readily to a clean iron surface. Even with exposure to only 1 Langmuir of methyl chloride, chlorine appeared in the Auger surface spectrum.

Of interest in lubrication is the tenacity of the adsorbed film on the surface. A sliding friction experiment was therefore conducted on the film indicated by the chlorine and carbon peaks of figure 3. Sliding over the same surface was continued for a total of one hundred passes without any additional methyl chloride exposure. The friction and Auger analytical results are presented in figure 4.

Examination of figure 4 indicates essentially no change in friction coefficient or chlorine Auger peak intensity during one hundred passes over the same surface. The sliding friction experiment of figure 4 was conducted at a vacuum of 10^{-10} torr. The residual methyl chloride film did not desorb, indicating the tenacity of the film to the surface. The friction coefficient for the clean iron surface was 2.0 when in contact with the aluminum oxide rider. Thus, the adsorbed film accomplished a marked reduction in friction coefficient.

After completion of one hundred passes over the surface, the iron disk was heated during bakeout in vacuum to 250° C for 12 hours. There was no evidence of a decrease in either chlorine or carbon Auger peak intensity.

In table I the chlorine and carbon Auger peak intensities are presented after 10 000 Langmuirs exposure to methyl chloride for the static case and with the surface under sliding contact. Sliding contact does not appear to alter the adsorptive behavior of methyl chloride to iron. It appears to promote neither adsorption nor desorption.

Vinyl Chloride

Another simple organic molecule containing chlorine is vinyl chloride ($CH_2 = CHCl$).

In addition to containing chlorine, vinyl chloride contains two carbon atoms and is unsaturated. Its unsaturated nature permits it to polymerize very readily.

An iron surface containing the normal surface films shown in figure 2(a) was exposed to varied amounts of vinyl chloride. Vinyl chloride adsorbed to the iron surface with an exposure of as little as 1 Langmuir. The adsorption must be chemical in nature because the experiments were conducted at 23° C in a vacuum of 10^{-10} torr. By definition, physical adsorption cannot exist at room temperature and 10^{-10} torr.

The uptake of vinyl chloride by the contaminated iron surface and its influence on friction coefficient is indicated by the data of figure 5. With increasing exposure to vinyl chloride, there is an increase in chlorine Auger peak intensity and a corresponding decrease in friction coefficient. Thus, strong bonds of chemisorption form between vinyl chloride and oxide-covered iron surfaces.

If the residual surface oxides are removed from the iron surface, larger quantities of vinyl chloride are taken up by the surface. This is demonstrated by the data of figure 6. The chlorine Auger peak intensity is plotted in figure 6 for various exposures to vinyl chloride. A marked difference in the quantity of vinyl chloride adsorbed exists for the two states of the metal surface. Surface cleaning of iron promotes adsorption.

An interesting aspect of vinyl chloride adsorption is that, in addition to surface cleanliness, sliding exerted an influence on adsorption. Figure 7 presents the Auger chlorine peak intensities with various exposures to vinyl chloride while the iron disk was stationary (static) and during sliding.

There is in figure 7 a marked difference in adsorption of vinyl chloride to the iron surface. Sliding and the associated strain of the iron results in an increased quantity of vinyl chloride on the iron surface. In both instances, adsorption is to a clean iron surface.

With static adsorption there is a linear increase in the vinyl chloride on the surface with increasing exposure times. In sliding, however, this behavior is not observed. There is with sliding a notable increase in the chlorine peak intensity when the surface exposure was increased from 10 to 100 Langmuirs. Beyond 100 Langmuirs no further increase in the chlorine Auger peak intensity was observed.

Accompanying the increase in uptake of vinyl chlorine with sliding at 100 Langmuirs was a corresponding decrease in friction coefficient. This decrease is indicated in the data of figure 8.

A very marked decrease in friction coefficient occurs in figure 8 in the surface exposure range of 10 to 100 Langmuirs. Up to 10 Langmuirs there appeared to be essentially no change in friction. After the sharp decrease in friction from 10 to 100 Langmuirs, no further reduction in friction was observed.

Ethyl Chloride

Vinyl chloride is a polymer forming material. Ethyl chloride, CH_3-CH_2Cl, its saturated equivalent is not. Both molecules contain two carbon atoms and one chlorine. An iron disk surface was exposed to ethyl chloride and the chlorine Auger peak intensity is plotted in figure 9.

An examination of figure 9 indicates that the iron surface becomes essentially saturated with ethyl chloride at an exposure of 1 Langmuir during sliding. No change in Auger chlorine peak intensity was observed beyond 1 Langmuir. Ethyl chloride is chemically more reactive with iron than vinyl chloride (ref. 6). This may account for no further increase in Auger chlorine peak intensity with exposures beyond 1 Langmuir of ethyl chloride.

The observation to be made from figure 9 is that even though vinyl chloride may be expected to be less reactive with a metal surface than its saturated equivalent, ethyl chloride, a greater concentration of vinyl chloride develops on the surface of iron with sliding. This may be the result of the ability of vinyl chloride to polymerize. The absence of a marked increase in carbon peak intensity in table I with the increase in chlorine for vinyl chloride may be associated with the molecular arrangement of the vinyl chloride on the surface. The carbon may be shielded by the chlorine.

Chemisorption is a monolayer process. That is, once a monolayer has formed, chemisorption to the iron surface is complete. Subsequent increases in hydrocarbon surface concentration must result from chemical reaction or polymerization in these experiments. The lesser chemical reactive nature of vinyl chloride would seem to indicate that polymerization is accounting for the differences in surface concentration for vinyl and ethyl chlorides seen in figure 9.

Static adsorption studies were conducted on a clean iron surface with both vinyl and ethyl chlorides. After 10 000 Langmuirs exposure, there was a greater concentration of ethyl than vinyl chloride on the iron surface. Thus, the normally greater reactivity of ethyl chloride was confirmed. This is also additional evidence for the polymerization of vinyl chloride. The sliding friction process appears to be initiating this polymerization.

Vinyl Chloride Adsorption and Mechanical Effects

In practical lubrication systems, hydrocarbons are generally in contact with oxide-covered surfaces. Some sliding friction experiments were conducted with iron containing its naturally occurring oxides in an environment of vinyl chloride at various loads. The objective of the experiments was to determine the influence of a mechanical parameter such as loading on surface reactions. The aluminum oxide rider was loaded against the iron disk at loads from 100 to 800 grams. Sliding was done in vinyl chloride at a pressure of 1×10^{-6} torr. Results obtained in these experiments are presented in figures 10 and 11.

Figure 10 is a presentation of three Auger traces. In figure 10(a) the load employed was 100 grams. The chlorine Auger peak is present but small. When the load is increased to 500 grams (fig. 10(b)), the chlorine Auger peak increases indicating a higher concentration of vinyl chloride on the surface. A further increase in load results in a reduction in the chlorine Auger peak intensity as seen in figure 10(c).

The chlorine Auger peak intensities and friction coefficients for a number of loads are presented in the data of figure 11. In figure 11, an increase in load results in an increase in chlorine Auger peak intensity to 500 grams. Beyond 500 grams a decrease in chlorine peak intensity occurs. Friction coefficient is strongly dependent upon the concentration of vinyl chloride present on the surface. Where the chlorine peak intensity is maximum in figure 11, friction is nearing a minimum. To a point, increasing the load has a beneficial effect in the adsorption of vinyl chloride and in the decrease of friction. There is, however, an optimum beyond which load is detrimental to both. At higher loads the frictional interfacial energy may be sufficient to bring about desorption of the adsorbed species. Such desorption would result in less film coverage of the iron surface, greater adhesions, and consequently higher friction.

Ethane and Ethylene

In order to gain some insight into the influence of the chlorine in the vinyl and ethyl chloride structures on interaction with the iron surface, experiments were conducted with ethane, $CH_3\text{-}CH_7$, and ethylene, $CH_2{=}CH_2$. These two hydrocarbons contain two carbon atoms and ethylene is unsaturated but neither has chlorine in its molecular structure.

In figure 12 the carbon Auger peak intensity is plotted as a function of exposure to ethane for both the static and sliding cases. An examination of figure 12 indicates, first, that there is essentially no friction activated adsorption of ethane to the iron surface. With ethyl chloride, there was friction activated adsorption. This would tend to confirm that the bonding of the molecule to the iron surface may be via the chlorine.

The second observation to be made from figure 12 is that the amount of material (ethane) adsorbed does not increase rapidly with exposure. Ethane uptake by the surface for both the static and sliding case is slow. Friction coefficient is also higher than when chlorine is present as indicated by the data of table I.

The coefficient of friction is presented in figure 13 for the iron with ethane and ethylene adsorbed to the surface. Very little, if any, difference in friction exists for these two gases.

Similar experiments were conducted with ethylene and some of the results obtained are presented in table I. Again as with ethane, there appeared to be no friction activated adsorption. Further, the friction coefficient is comparable to that obtained with ethane.

The results obtained with ethane and ethylene indicate that, first, chlorine in the molecule affects friction initiated surface reactions. Neither ethane nor ethylene increased in surface concentration with sliding while both ethyl and vinyl chloride did. Secondly, ethylene, which is unsaturated as is vinyl chloride, did not increase in surface concentration despite its polymer forming ability. Thus, polymerization of molecules on an iron surface may, with sliding, depend upon molecular structure as well as the simple ability of the molecule to polymerize.

CONCLUSIONS

Based upon the results obtained in this investigation with various hydrocarbons exposed to an iron surface both statically and during a sliding friction experiment, the following conclusions are drawn:

1. With respect to the hydrocarbon vinyl chloride:

   a. The sliding friction process increases the hydrocarbon concentration observed on the surface.

   b. Friction coefficient is sensitive to surface concentration.

   c. Chemisorption of the molecular species occurs to both clean and oxide-covered surfaces. The surface concentration is, however, greater for the clean surface.

   d. Polymerization of the structure appears to occur on the surface.

2. While ethyl chloride is chemically more reactive with a metal surface than vinyl chloride, a greater concentration of vinyl chloride is seen on the surface with sliding. This may be due to the polymer forming tendencies of vinyl chloride.

3. Mechanical parameters such as load affect the surface concentration and accordingly the friction of vinyl chloride on an iron surface. There is an optimum load for maximum surface coverage and minimum friction.

4. While friction initiated adsorption of ethyl and vinyl chlorides to an iron surface was observed, such activation was not seen with ethane and ethylene. Thus, chlorine in the molecular structure of the hydrocarbon promotes friction initiated surface adsorption.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, January 15, 1973,
502-01.

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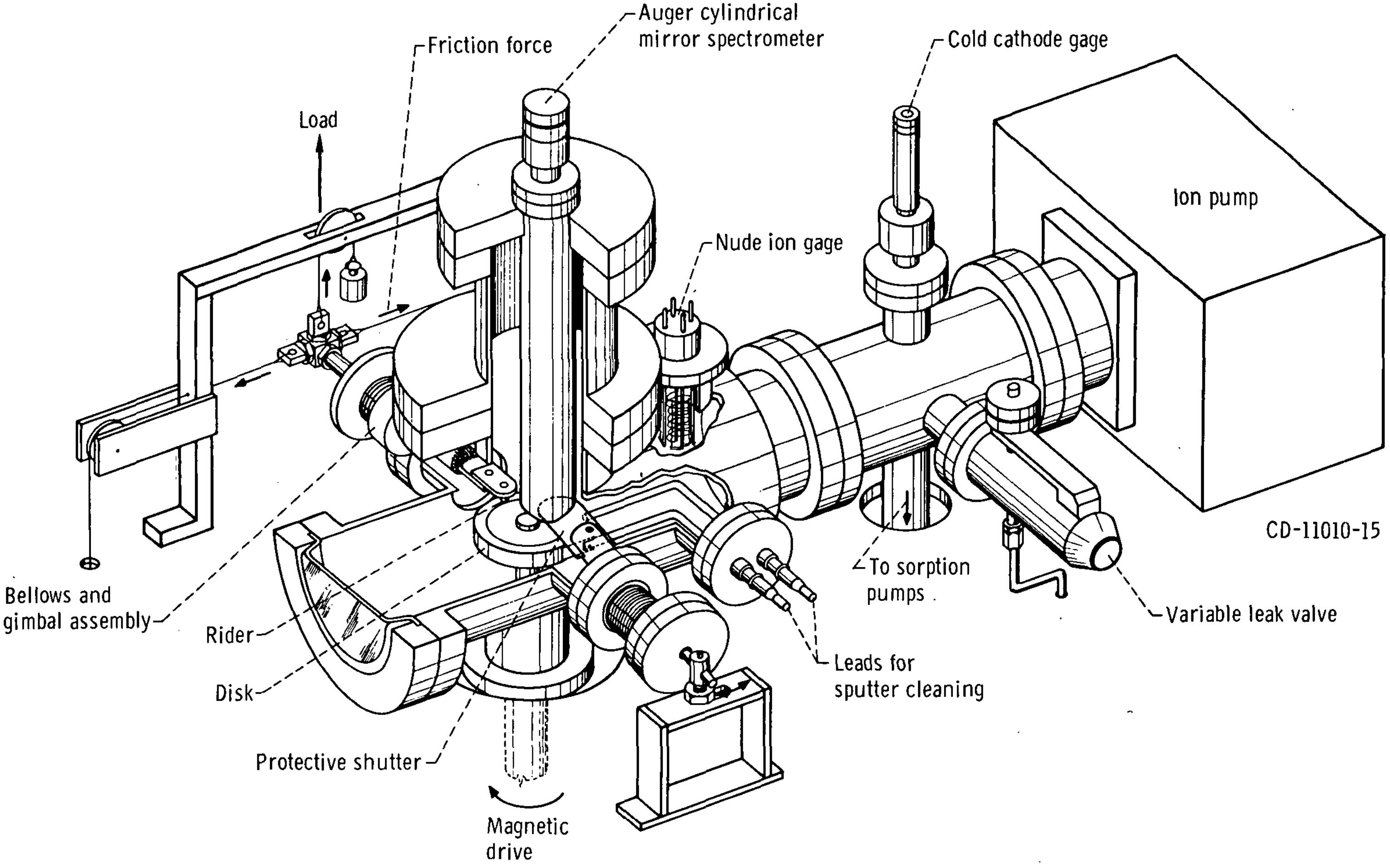
TABLE I. - COEFFICIENT OF FRICTION AND AUGER PEAK INTENSITIES FOR VARIOUS HYDROCARBONS ADSORBED TO A CLEAN IRON SURFACE

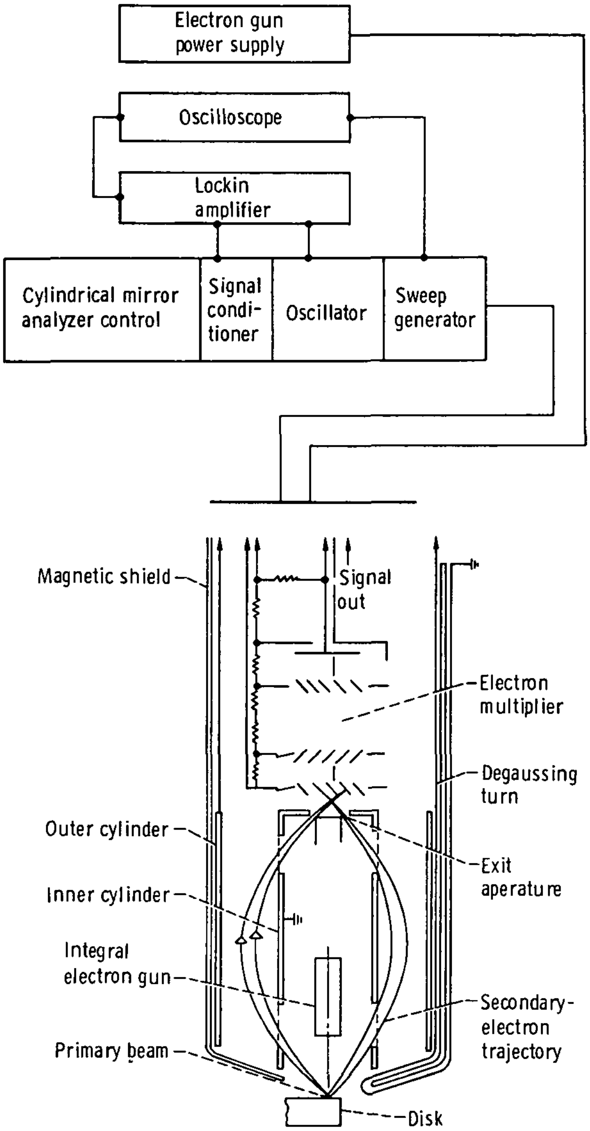
Hydrocarbon adsorbed	Auger peak intensity for 10 000-Langmuir exposure, arbitrary units				Coefficient of friction[a]
	Static		During sliding		
	Carbon	Chlorine	Carbon	Chlorine	
Methyl chloride CH_3Cl	0.4	2.4	0.3	2.3	1.2
Ethane CH_3-CH_3	.9	---	.4	---	1.4
Ethylene $CH_2=CH_2$	.8	---	.9	---	1.4
Ethyl chloride CH_3-CH_2Cl	.8	2.2	.7	2.0	1.1
Vinyl chloride $CH_2=CHCl$	.9	1.0	1.2	5.3	1.2
None					2.0

[a] Sliding velocity, 30 cm/min; load, 100 g.



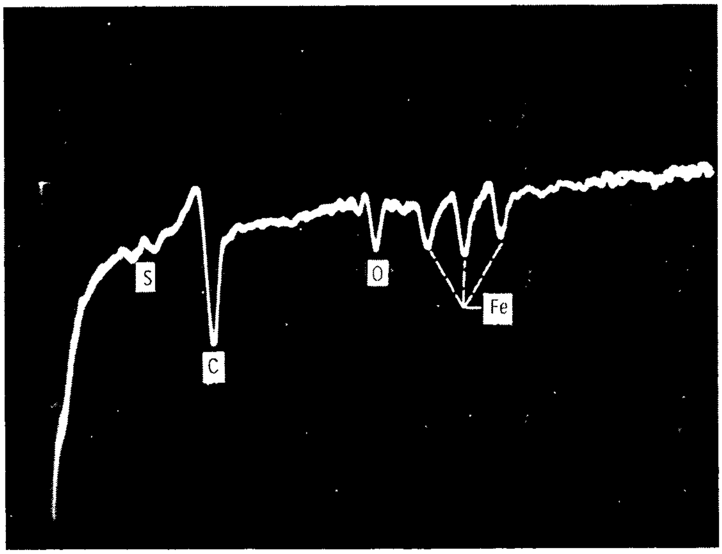
(a) Friction apparatus.

Figure 1. - Friction apparatus with Auger spectrometer.

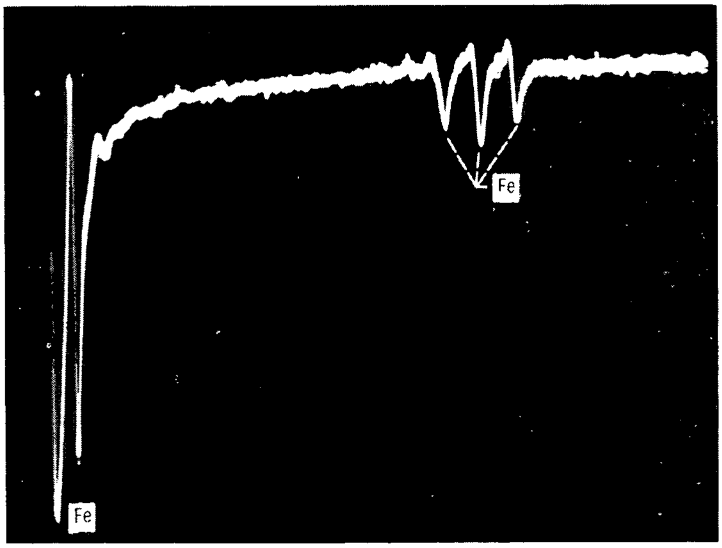


(b) Schematic of Auger cylindrical mirror analyzer.

Figure 1. - Concluded.



(a) Before sputter cleaning.



(b) After sputter cleaning.

Figure 2. - Auger spectra for an iron surface before and after sputter cleaning.

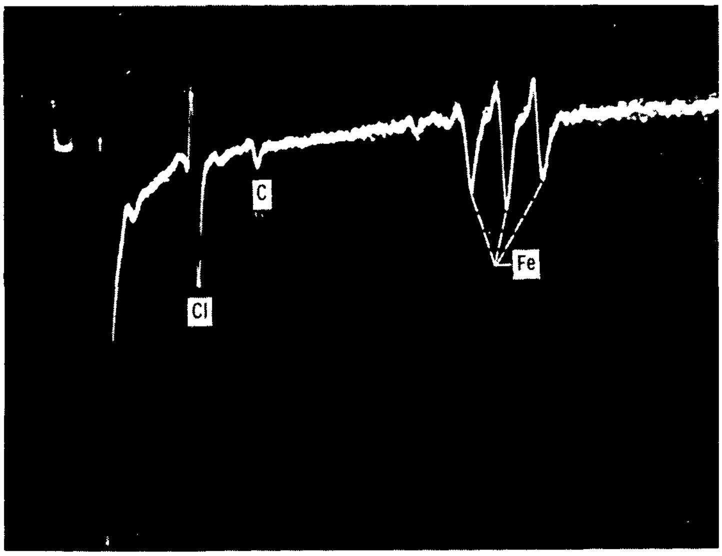


Figure 3. - Auger spectrum for iron surface with methyl chloride adsorbed. Iron surface sputter cleaned prior to exposure of 10 000 Langmuirs of methyl chloride.

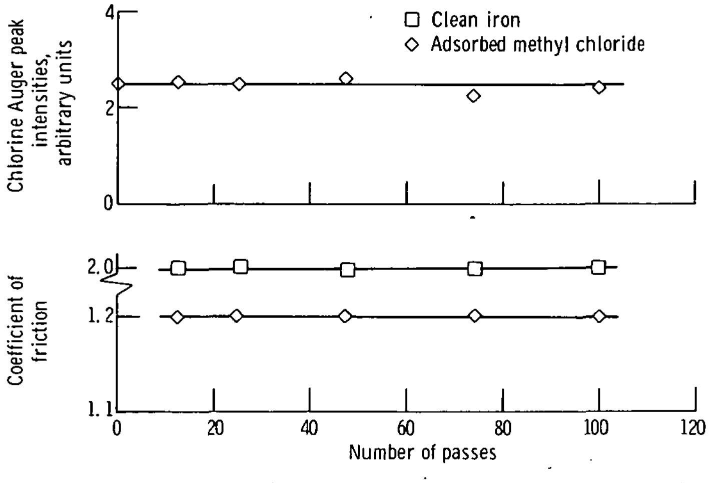


Figure 4. - Coefficient of friction and Auger peak intensity of chlorine for methyl chloride adsorbed on clean iron. Initial exposure prior to start of sliding, 10 000 Langmuirs of methyl chloride; sliding velocity, 30 centimeters per minute; load, 100 grams; temperature, 23^0 C.

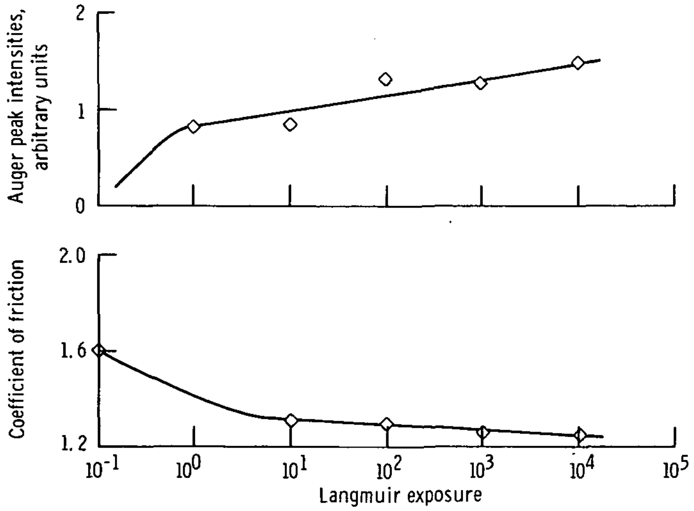


Figure 5. - Coefficient of friction and chlorine Auger peak intensities for various exposures of iron to vinyl chloride. Normal oxide present on iron, rider aluminum oxide; load, 100 grams; sliding velocity, 30 centimeters per minute; temperature, 23° C.

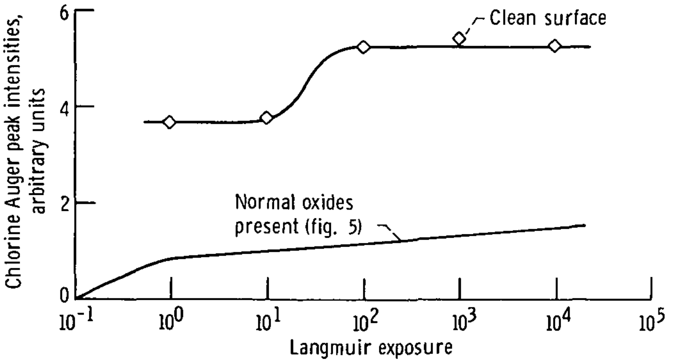


Figure 6. - Comparison of adsorption of vinyl chloride on iron surface in clean and oxidized states as evidenced by Auger analysis for chlorine. Adsorption conducted during sliding friction experiment.

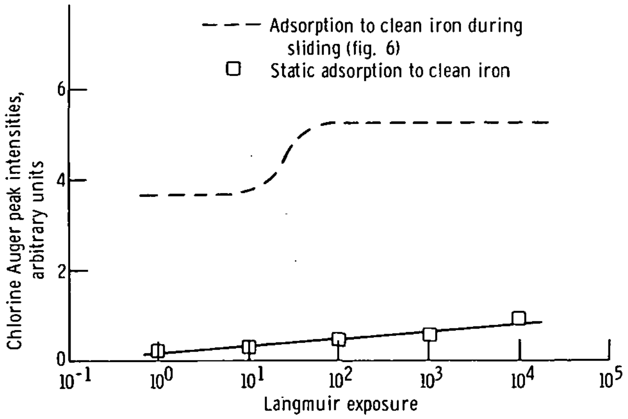


Figure 7. - Chlorine Auger peak intensity for vinyl chloride adsorption with and without sliding on iron surface. Rider specimen, aluminum oxide; sliding velocity, 30 centimeters per minute; load, 100 grams; temperature, 23° C.

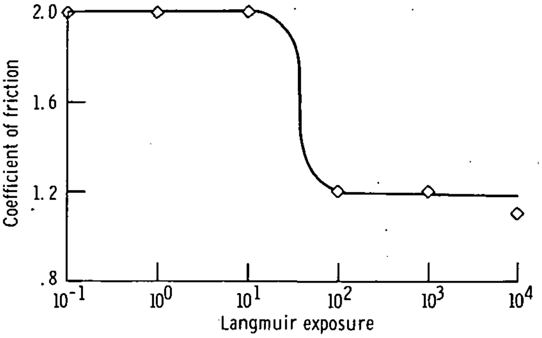


Figure 8. - Coefficient of friction for aluminum oxide rider sliding on clean iron in presence of vinyl chloride. Sliding velocity, 30 centimeters per minute; load, 100 grams; temperature, 23° C.

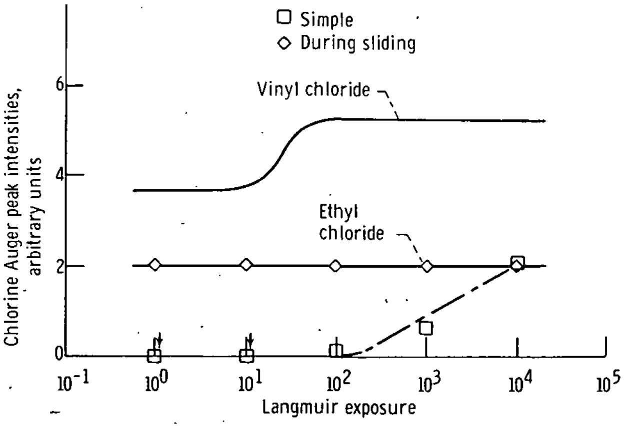
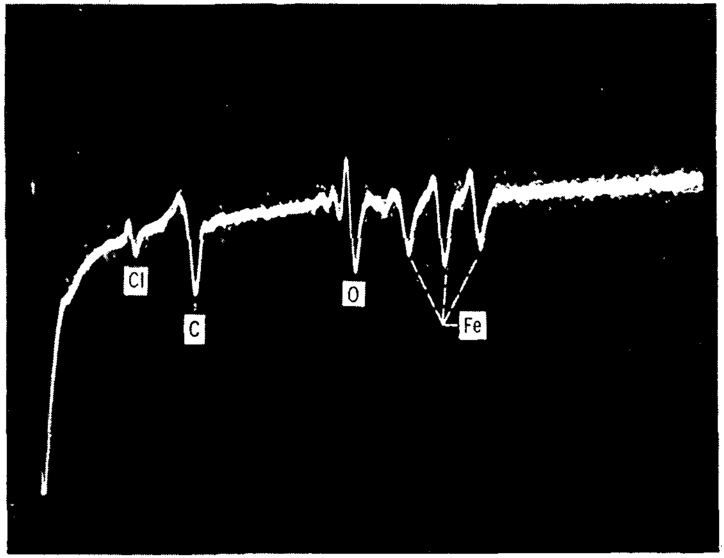
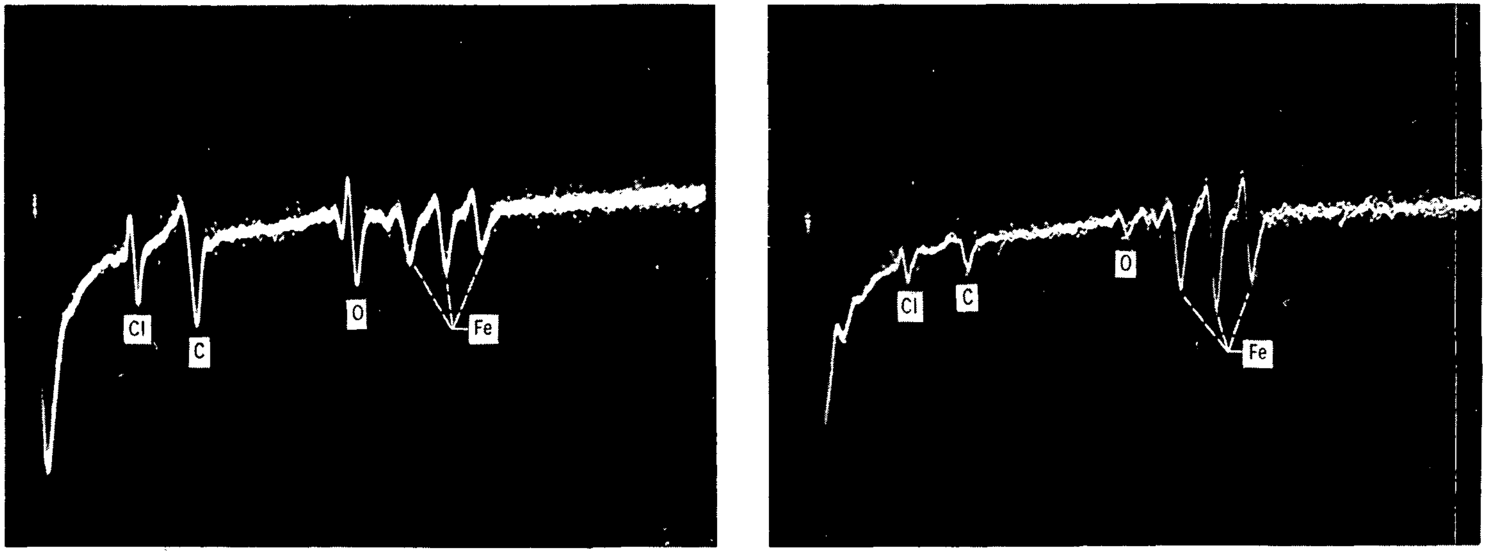


Figure 9. - Chlorine Auger peak intensities for vinyl chloride and ethyl chloride adsorbed on clean iron surface statically and during sliding. Rider specimen, aluminum oxide; sliding velocity, 30 centimeters per minute; load, 100 grams; temperature, 23° C.



(a) Load, 100 grams.



(b) Load, 500 grams.

(c) Load, 800 grams.

Figure 10. - Auger spectra for oxide covered iron surface with vinyl chloride adsorbed during sliding at various loads. Ambient pressure, 10^{-6} torr of vinyl chloride; rider specimen, aluminum oxide.

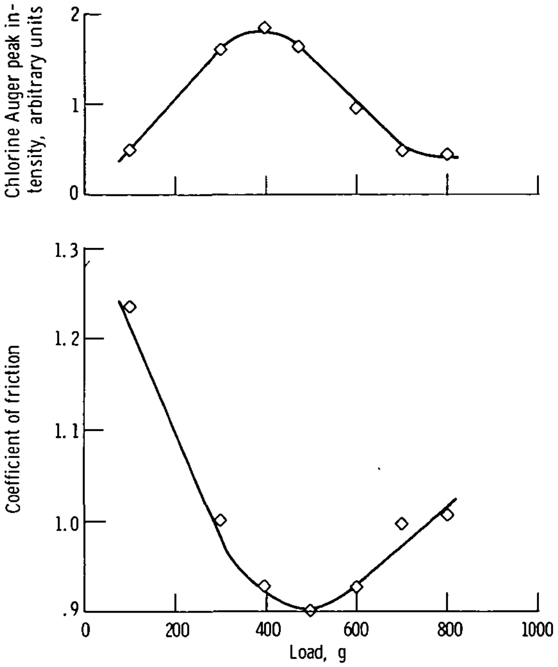


Figure 11. - Coefficient of friction and Auger chloride peak intensity as function of load for vinyl chloride on iron surface. Ambient pressure, 10^{-6} torr of vinyl chloride; rider specimen, aluminum oxide; sliding velocity, 30 centimeters per minute; temperature, 23° C; normal oxides present on iron surface.

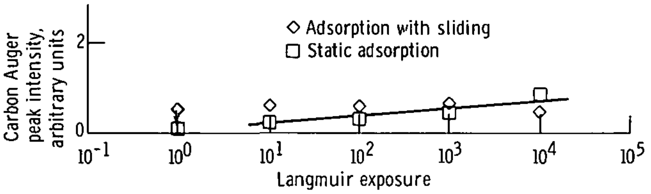


Figure 12. - Carbon Auger peak intensities for ethane adsorbed to clean iron surface statically and with sliding. Rider specimen, aluminum oxide; sliding velocity, 30 centimeters per minute; load, 100 grams; temperature, 23° C.

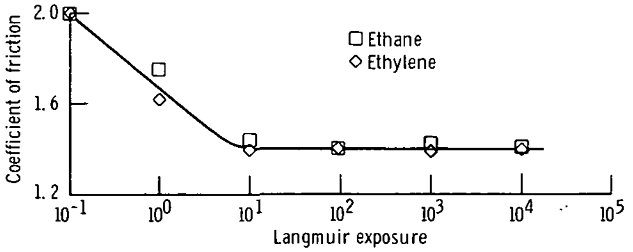


Figure 13. - Coefficient of friction as function of exposure for iron with ethane and ethylene adsorbed on surface. Ambient pressure, 10^{-10} torr; sliding velocity, 30 centimeters per minute; load, 100 grams; temperature, 23° C.

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