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NASA TM X-71452

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(NASA-TM-X-71452) THE ATOMIC NATURE OF
POLYMER-METAL INTERACTIONS IN ADHESION,
FRICTION AND WEAR (NASA) 18 p HC \$3.00
CSSL 07D

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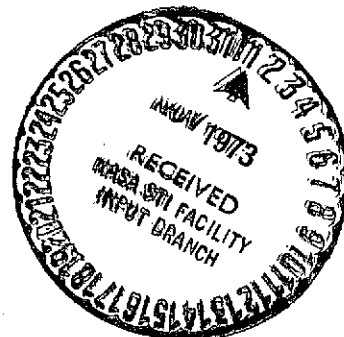
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**THE ATOMIC NATURE OF POLYMER-METAL INTERACTIONS
IN ADHESION, FRICTION AND WEAR**

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TECHNICAL PAPER proposed for presentation at
Symposium on Advances in Polymer Friction and Wear
sponsored by the American Chemical Society
Los Angeles, California, March 31-April 4, 1974



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ADHESION, FRICTION AND WEAR

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INTRODUCTION

In lubrication systems polymers are frequently used in sliding or rolling contact with metal surfaces. It is of interest therefore to study the fundamental surface interactions between these polymers and metals and the manner in which those interactions influence adhesion, friction and wear.

Of solids the polymers used, PTFE (polytetra-fluoroethylene) and polyimide are two structures with inherently good self-lubricating and transfer characteristics. Unlike polymers such as nylon they do not depend upon the presence of adsorbates such as water vapor or other environmental constituents for their low adhesion, friction and wear properties¹⁻³.

Another type of polymer-metal interaction that has been observed is associated with the formation of polymeric surface films from the interaction of monomers or (lower molecular weight species) with metal surfaces in sliding contact. Frictional energy provides the activation energy for polymerization. This phenomenon was first observed in electrical contacts⁴⁻⁵. And more recently it has been observed in friction and wear studies⁶⁻⁷.

The objectives of this paper are twofold, first, to examine the adhesion, friction and wear behavior of the solid polymers PTFE and polyimide with various metals and second, to gain insight into the role of the friction process in the generation of polymer surface films. Analytical surface tools including field ion microscopy, Auger emission spectroscopy and scanning electron microscopy were used to examine the atomic nature of polymer - metal surface interactions.

MATERIALS

The solid polymer bodies of PTFE and polyimide used in these studies were research grade materials. The polymer forming gaseous monomer vinyl chloride, was 99.95 percent in purity as was the ethyl chloride.

The metals used were iron, tungsten, gold, and aluminum. The tungsten, gold, and aluminum were all 99.999 percent and the iron was 99.99 + percent in purity.

EXPERIMENTAL RESULTS

Solid Polymers

Field ion microscopy. - The field ion microscope (FIM) is a powerful tool for studying the adhesion process. A combination of high magnification and resolution of 2-3 Å permits the adhesion process to be studied in atomic detail. The principals and operation of the FIM are described by Müller⁸, who also demonstrated that adhesion could be studied with the FIM⁹.

A schematic of the FIM used as an adhesion apparatus is shown in Fig. 1. A specially constructed contact device can be moved into place under the field ion tip by the compression of bellows. With suitable voltage applied to the

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electromagnets, the beam can be made to move upward and contact the field ion tip. The load applied during contact is determined by the change in output voltage from a solar cell. PTFE or polyimide is mounted on the surface of a thin (0.002 cm) gold foil. When contact is made, the foil deflects downward cutting off some of the light illuminating a solar cell surface thus changing the output voltage (see Ref. 10 for details). When the beam is moved downward away from the tip, the adhesion between the tip and the PTFE or polyimide pull the foil upward past the original zero load point until separation occurs. Calibration of the loads applied to the foil to cause given voltage changes was done with an electronic balance with a sensitivity of $<1 \mu\text{g}$.

A series of PTFE-tungsten contacts were made at loads between 20-30 μg and the force of adhesion measured. Figure 2 is a micrograph taken prior to contact of a tungsten tip. Figure 3 is a photomicrograph taken following contact with PTFE for a few seconds. Many extra image points are apparent on the post contact micrograph particularly on the (110) plane. Adsorbed or adhered atoms can be observed because the geometry of the extra atoms on the surface of the flat creates points of localized field enhancement resulting in increased probability of ionization. Thus, clusters visible on the (110) plane are fragments of PTFE which remain adhered to the tungsten surface after separation has occurred. The other bright image spots also represent PTFE on the metal surface but their cluster-like nature cannot be resolved. The fragments of PTFE have the appearance of the end of a PTFE chain which is normal to the (110) plane. The fact that the fragments are stable at the very high electric field required for helium ion imaging implies that the bonding between the PTFE and tungsten is very strong, otherwise field desorption of the adhered PTFE would occur.

To obtain a measure of the bonding between the PTFE and tungsten, the forces of adhesion were measured for a number of contacts over varying periods of contact time. The results are summarized in Fig. 4. For short contact times, the forces of adhesion were immeasurably small. After 2 minutes, however, the force of adhesion increased markedly. At contact times of 4-6 minutes, adhesion coefficients approaching those for clean metals in contact were obtained.

It was observed that negligible adhesion force was obtained when a second contact was made with a previously contacted tip indicating the adhesive polymer to metal bond is stronger than the cohesive polymer bond. Polymer radicals can be expected to occur as a result of the breaking of chains, by the chemical interaction between polymer and metal. Thus, for PTFE contacting a clean tungsten surface, the possibility of unsatisfied valence states of carbon atoms in PTFE bonding to tungsten exists.

A heavily loaded tungsten - PTFE (approximately three times more load $\sim 1 \text{ mg}$) contact gave the rather surprising result that extensive deformation of the tungsten occurred. The deformation extended far into the bulk of the material as shown in Fig. 5, which was taken after extensive field evaporation (an effective increase in tip radius of approximately 50 percent). Generally, during heavy loaded contacts the tip will bend so that the image is often displaced on the screen; however, there is no displacement of (110) in the micrograph of Fig. 5. The tip, when pressed into the PTFE, may be prevented from bending and thus absorbing the stress further up the shank by the very large area of contact (relative to metal-metal contacts).

Mechanical contacts with a polyimide polymer contacting tungsten tips were made in vacuum of 10^{-9} torr with both light and heavy loads. At light loads the results obtained were analogous to those obtained with PTFE. Random distribution of bright spots were visible indicative of polymer fragments adhering to the tungsten. Spots (polymer fragments) were particularly heavily clustered on the (110) surface as was observed with PTFE.

To determine if load would significantly influence the polyimide-tungsten contacts, an experiment was conducted with polyimide contacting tungsten using a load approximately three times greater than that used for light load contacts. Figure 6 is the image of the same tungsten tip after a heavily loaded polyimide contact. The results show a series of elongated spots all radially projected outward from a dark nonimaging area on the right side of the micrograph. As was also observed with PTFE, it appears that the polyimide polymer fragments have been oriented by the radial stresses projecting outward from the center of contact, where the center of contact is the dark region to the right in the micrograph.

Auger emission spectroscopy. - Auger emission spectroscopy (AES), provides a technique for determining the chemical composition of a surface for elements heavier than helium with a high degree of sensitivity (i.e., 1/100th of a monolayer). AES was used in conjunction with a vacuum friction apparatus to provide an instantaneous chemical analysis of a metal surface during both static and dynamic contact with PTFE. The experimental apparatus is shown in Fig. 7 and further details on the technique and equipment are available in the literature¹².

Static contact. - Transfer of PTFE to atomically clean metals by static contact was observed for all metals used. These included iron, tungsten, aluminum, and gold. Figure 8 shows three Auger spectrograms for a gold surface before and after adhesive contact. The presence of carbon and fluorine in Fig. 8(b) indicate the transfer of PTFE to the gold surface.

The possibility that the transfer of PTFE to metal might be adversely affected by the presence of an oxide film on the metal was investigated by two methods. In the first method, high-purity oxygen was admitted to the chamber after the disk surface had been sputter cleaned with the oxygen being chemisorbed on the surface to monolayer coverage. Static contact was then initiated, and again transfer of PTFE was observed. Thus, the presence of a monolayer of chemisorbed oxygen does not prevent the transfer observed.

The second method, involved the use of a pre-oxidized aluminum disk. It is known that the natural oxide layer on aluminum is many layers thick. Removing the adsorbed carbon dioxide and carbon monoxide by a short sputtering (20 min) exposed the "clean" aluminum oxide layer. Static contact was again initiated, and again PTFE was found on the surface. Thus, PTFE transfers to the oxide of aluminum as well as to the clean metal, which implies that the chemical activity of the substrate was not an important factor in the transfer observed in these static contact experiments.

Sliding contact. - Sliding contact was initiated on an atomically clean tungsten disk. The velocity was 1.0 centimeter per second, and the load was 100 grams. A transfer film of PTFE was generated on the disk during the first revolution. The Auger spectrogram is shown in Fig. 9(a). The carbon and fluorine peaks were much larger than those in the static contact experiments and indicated the presence of larger amounts of PTFE on the surface.

The film appeared uniform across the track, as indicated by the constance of the peaks when the deflection plates in the electron gun moved the beam across the track. The film also appeared uniform and continuous along the circumference of the track, as indicated by the constancy of the peaks throughout the first revolution of the disk.

Notice that the tungsten peak is still visible in the Auger spectrum. This indicates that the film was only a few monolayers thick, since AES is sensitive only to the first few layers on the surface.

Information on the structure of the film and its interaction with the substrate may be obtained from the time dependence of the Auger peaks when the

disk is held stationary and the electron beam impinges on one spot of the surface. In Fig. 9, the two spectra were taken 60 seconds apart. It can be seen that the fluorine peak has decreased while the carbon and tungsten peaks have grown. The incident 2000-eV electrons used to excite the Auger transitions have severed the carbon-fluorine bonds in the PTFE, and the fluorine has desorbed from the surface. With the departure of the fluorine, Auger electrons from the carbon and tungsten can leave the surface, enter the analyzer, and cause growth of these peaks. Exposure of the surface to the electron beam for about 1 minute resulted in complete disappearance of the fluorine peak (Fig. 2(b)).

Pepper¹³ calculated the cross section value for electron induced fluorine desorption from the time dependence of the fluorine Auger peak as 5×10^{-18} cm², thusly implying no interactions exist between the fluorine in the PTFE and the metal substrate. This indicates that bonding to the metal must be via the carbon atom.

Figure 10 shows the curve of friction coefficient versus number of passes of the disk for PTFE sliding on atomically clean tungsten and aluminum. The value of 0.08 obtained for PTFE on tungsten is consistent with values usually reported for PTFE sliding on metals in air. The friction for PTFE on aluminum, however, rose drastically from 0.08 at the start to over 0.5 in less than one complete revolution. Severe "machining" of the aluminum disk occurred and metal cut from the wear track could be seen at the rider-disk contact zone, as well as chips of aluminum covering the surface. The severe scoring of the aluminum occurred both in the presence or absence of a oxide film.

Scanning electron microscopy. - To further study the behavior of the PTFE-metal system, a series of experiments were conducted in air with face centered cubic aluminum single crystals. The crystals were of (111), (110), and (100) orientations. Friction tracks were generated by sliding PTFE over the electropolished single crystals and then examining the resulting wear tracks and rider wear scars in the scanning electron microscope (SEM).

Sliding wear tracks were generated on each of the three single crystal surfaces under loads of 100 and 200 grams. Single passes as well as multi-passes over the same track were made at a sliding velocity of 0.07 cm/sec. In all cases, large disordered areas of PTFE were observed adhering to the aluminum surfaces at the initial contact area. Extending out of these areas were linear fibers of PTFE parallel to the surface. These were oriented in the direction of sliding.

The friction coefficients for the three crystals were in the range of approximately 0.06 to 0.20 during sliding. The friction on the (111) surface was generally the lowest ($0.06 \leq f_k$).

On the (111) plane, no damage to the aluminum surface was observed with a single pass of the rider at either 100 or 200 grams loads. Further, repeated passes over the same track did not damage the metal surface. At 100 gram loads no scoring was observed on the (100) plane but scoring was observed on the (110) plane. On the (100) and (110) planes, fine score marks were observed on the aluminum surface after a single pass at 200 grams. Higher magnification micrographs taken for the 200 gram load showed areas where pieces of aluminum had been plucked out of the bulk.

For PTFE sliding on the (100) plane, although no scoring was observed for a single pass at 100 grams load, fine score marks were observed after three passes over the same wear track. On the (110) plane, repeated passes resulted in extensive scoring for a 200 gram load. In general, score marks on both planes were found to terminate with a fragment of PTFE which had been pulled out of the bulk.

It appears as if scoring of the aluminum is caused by hard particles lodged in the PTFE. Discounting Al_2O_3 as the source of the hard particles, since scoring was observed in vacuum when the oxide was removed by sputtering, fragments of work hardened aluminum are being pulled out of the bulk (by the strong adhesion between the PTFE and the metal) during sliding and these embed in the PTFE; since these particles are in rubbing contact with the crystal they become work hardened and thus cause scoring. The transfer of aluminum to PTFE is confirmed by SEM of the observation of aluminum fragments embedded in the PTFE rider after running (Fig. 11).

The differences observed in the scoring behavior of the three aluminum single crystals appears to be correlated with the different microhardness of the three single crystals. Microhardness measurements on the three surfaces showed the order of hardness to be: (111) > (100) > (110). The (111) plane which is the hardest (highest atomic density) showed no scoring. On a (110) surface where the hardness is the least, the scoring process is most severe. On the (100) plane which is intermediate, between the (111) and (110), the scoring is intermediate in degree. The protection afforded by the oxide breaks down at very light loads because of the hard brittle nature of the oxide. Thus, the PTFE penetrates to the nascent metal and as a consequence of the strong adhesion and the localized stress, plucks some of the metal off the surface and these metal fragments are pushed along with the rider. As sliding continues, these pieces of metal become strain hardened until they are harder than the softer metal surface from which they came. These hardened particles can then score the parent aluminum surface from which they came.

Friction Initiated Polymer Film Formation

A simple organic molecule containing chlorine is vinyl chloride. 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 was exposed to various amounts of vinyl chloride. Vinyl chloride adsorbed to the iron surface with an exposure of as little as 1 Langmuir. The adsorption is chemical in nature because the experiments were conducted at 23 C in a vacuum of 10^{-10} torr.

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 Fig. 12. The chlorine Auger peak intensity is plotted in Fig. 12 for various exposures of vinyl chloride. A marked difference in the quantity of vinyl chloride adsorbed is apparent for the two states of the metal surface. Surface cleaning of iron promotes greater adsorption.

An interesting aspect of vinyl chloride adsorption is that in addition to surface cleanliness, sliding exerted an influence on adsorption. Fig. 13 presents the Auger chlorine peak intensities with various exposure to vinyl chloride while the iron disk was stationary (static) and during sliding.

There is a marked difference (Fig. 13(a)) in the adsorption of vinyl chloride to 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 simple 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.

Vinyl chloride is a polymer forming material. Ethyl chloride, its saturated equivalent, is not. Both molecules contain two carbon atoms and one chlorine. An iron disk surface was exposed to ethyl chloride. The chlorine Auger peak intensity observed is plotted in Fig. 14.

An examination of Fig. 14 indicates that the iron surface becomes essentially saturated with ethyl chloride at an exposure of 1 Langmuir. No change in Auger chlorine peak intensity was observed beyond 1 Langmuir. Ethyl chloride is chemically more reactive than vinyl chloride. This may account for no further increase in Auger chlorine peak intensity with exposures to ethyl chloride greater than 1 Langmuir. This may also be a result of the limiting depth detectability of Auger analysis (four to five atomic layers).

The observation to be made from Fig. 14 is that even though vinyl chloride is less reactive than its saturated equivalent, ethyl chloride, a greater concentration of vinyl chloride develops on the surface of iron with sliding. This can be related to the ability of vinyl chloride to polymerize. 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 Fig. 14.

Static adsorption studies were conducted on clean iron surfaces with vinyl and with 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.

Hydrocarbons are generally in contact in practical lubrication systems 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 unsputtered 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 Figs. 15 and 16.

Figure 15 is a presentation of three Auger traces. In Fig. 15(a) the load employed was 100 grams. The chlorine Auger peak is present but small. When the load is increased to 500 grams (Fig. 15(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 Fig. 15(c).

The chlorine Auger peak intensities and friction coefficients for a number of loads are presented in the data of Fig. 16. In Fig. 16 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 Fig. 16 friction is at 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.

DISCUSSION

The results of the study with PTFE and polyimide polymers in contact with tungsten indicate that polymers will transfer to a clean metal surface on simple touch contact. The transferred polymers, at the high field for helium ion imaging imply that the bond of the polymer to the metal surface is chemical in nature. With PTFE it is hypothesized that the bonding is that of carbon to the metal surface because the carbon to carbon bond is the weakest bond in the PTFE structure and the one most frequently seen broken on polymer scission. Furthermore, the carbon could readily interact with the clean tungsten to form bonds stable at the imaging and field evaporation voltages applied in this study.

With the polyimide both oxygen and carbon bonding to the tungsten surface are possible. But again because of the imaging and evaporation voltages employed the bond must be chemical in nature.

The concept of chemical bonding of the polymers to the clean metal surface necessitates the breaking of bonds in the organic molecule and the subsequent formation of metal to carbon or oxygen bonds to form organometallics. The breaking of organic bonds by metal surfaces is observed with hydrocarbons contacting metals in the field of catalysis. The tendency for such reactions should be increased when the metal surface is atomically clean because of the enhanced surface activity of the metal.

The effect of loading in the transfer of polymers was examined. Larger amounts of polymers were observed with an increase in load. The polymer appears to remain on the tungsten surface in longer chain fragments indicating that fracture occurred deeper in the polymer body than was observed at light loads. When field evaporation was conducted the polymer chain length that adhered to the tungsten could be reduced to that observed with light loads.

It is of interest also to note that preferred orientation of the polymer chains toward the zone of contact has occurred. This resulted when the tungsten was pressed into the polymer under load. Adhesion of polymer occurred to the tungsten surface. Polymer bonds were broken in the bulk and the relative tangential motion of the polymer body along the radius of the tungsten tip resulted in texturing (preferred orientation of chain fragments).

The results obtained with vinyl chloride in sliding friction experiments indicate that the friction process itself can supply the necessary energy to initiate polymer formation with metal surfaces. Under certain conditions the formation of such films is highly desirable in that such films reduced adhesion, friction and wear. There formation has even been suggested as a form of extreme pressure lubrication. The formation of such polymer films in other areas such as sliding electric contacts is undesirable because the polymer films formed increase interfacial contact resistance.

CONCLUDING REMARKS

Based upon the results presented herein the following concluding remarks are made:

1. In adhesion experiments with PTFE and polyimide contacting tungsten results obtained in the field ion microscope indicate that the polymers bond chemically to the clean metal surface.
2. Polymer chain fragments which transfer to the surface of tungsten in field ion microscopy adhesion studies are highly oriented.
3. Auger emission spectroscopy analysis of PTFE transfer films to various metal surfaces indicate that the PTFE is bonded to the metal surface via the carbon atom.

4. With PTFE in sliding contact with different orientations of aluminum, metal orientation is found to influence surfaces in sliding. The lowest friction and least amount of surface damage is detected on the highest atomic density (111) plane.
5. The friction process itself can initiate polymer film formation from simple organic molecules. The tendency is more pronounced for polymer formation with clean metals than for oxide covered metal surfaces.

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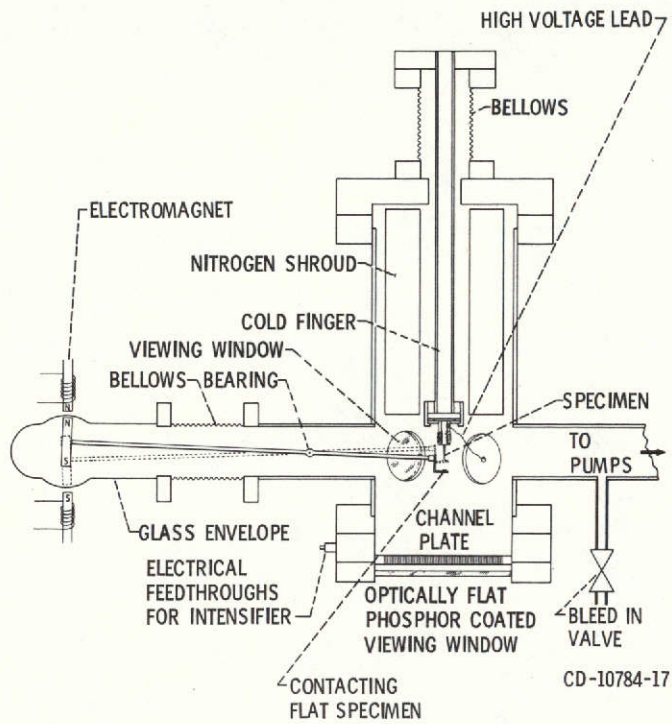


Figure 1. - Schematic of field ion microscope.

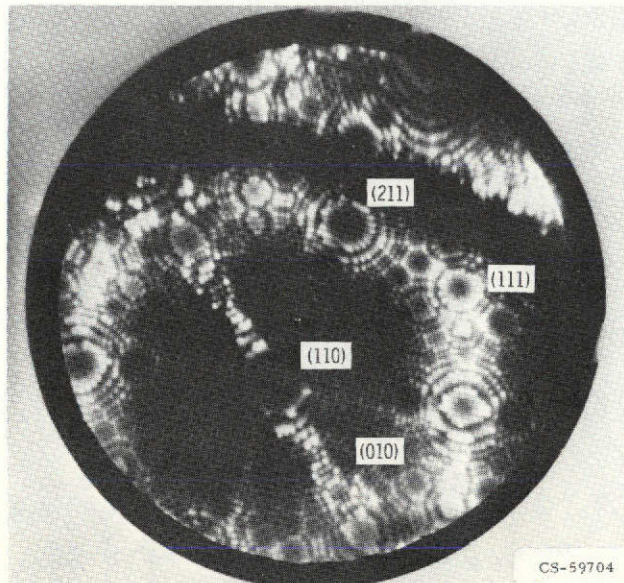


Figure 2. - FIM image of tungsten tip prior to contact (18.0 kV, helium image gas, liquid nitrogen cooling).

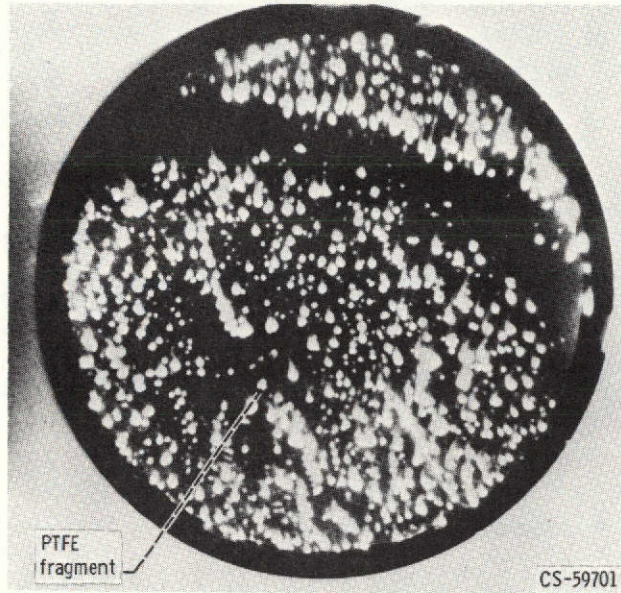


Figure 3. - FIM image of tungsten tip following contact with PTFE (16.5 kV, helium image gas, liquid helium cooling).

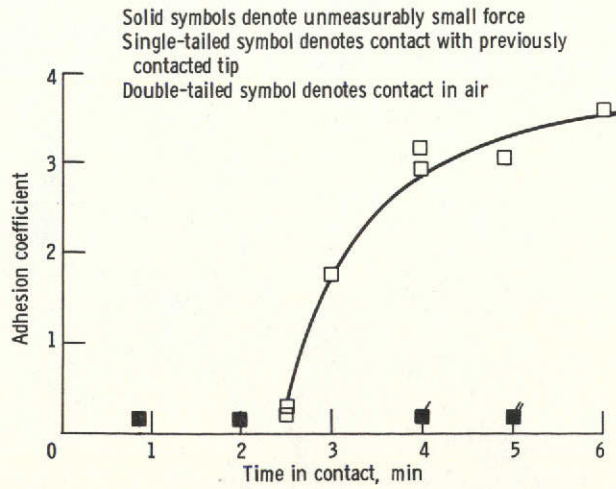


Figure 4. - Adhesion coefficients for PTFE-tungsten contacts.

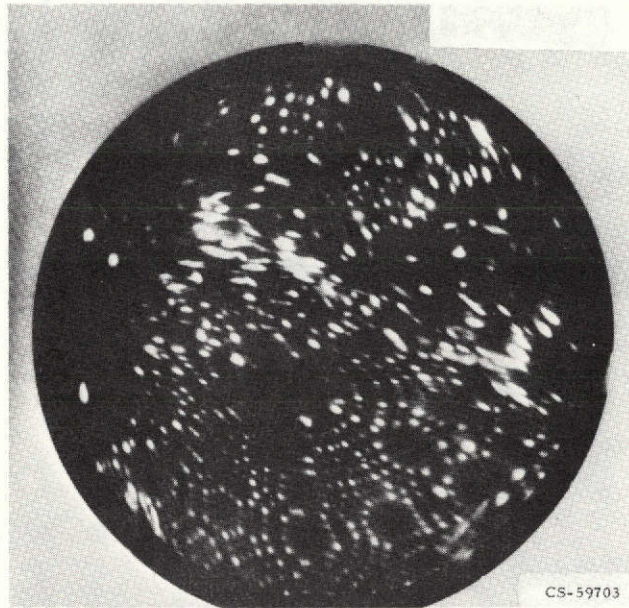


Figure 5. - Tungsten after heavy load PTFE contact.

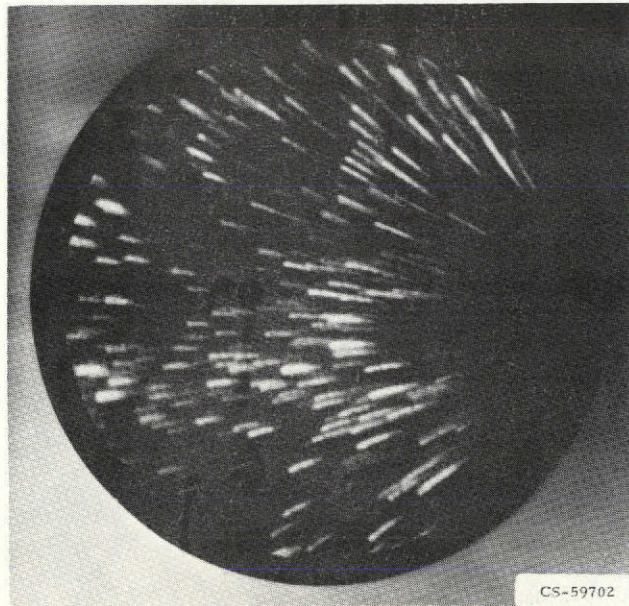


Figure 6. - Tungsten after polyimide contact (9.25 kV, helium image gas, liquid helium cooling).

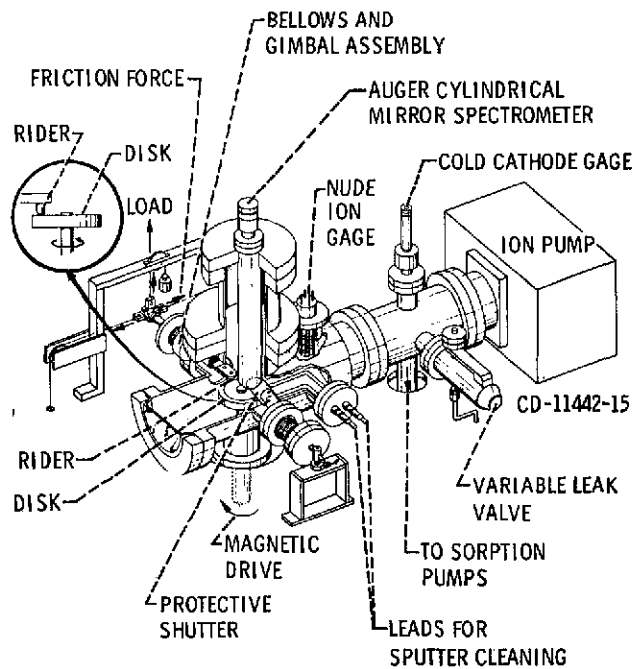
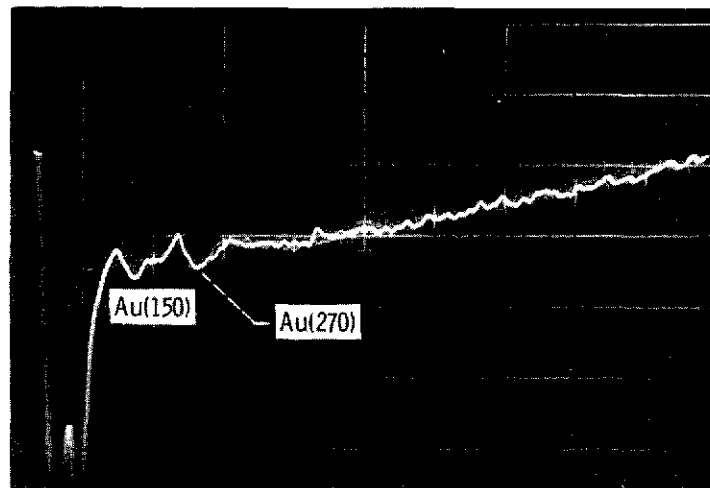
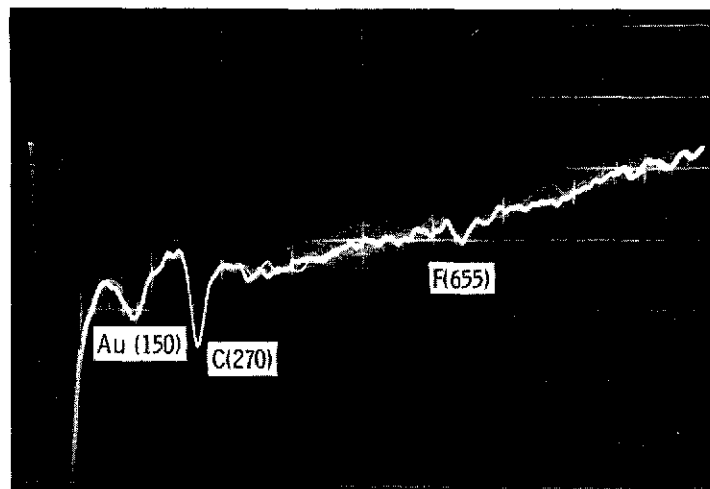


Figure 7. - Friction apparatus with Auger spectrometer.

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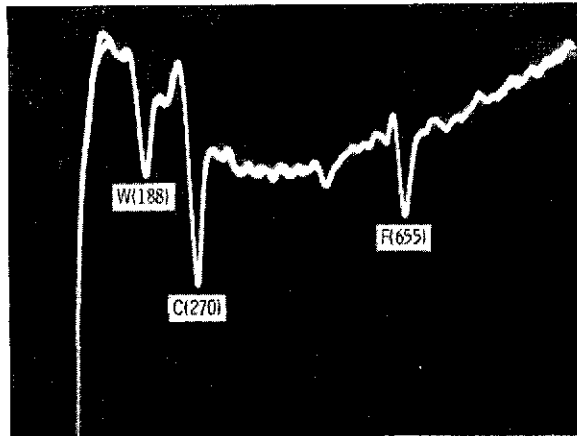
(a) CLEAN SURFACE.



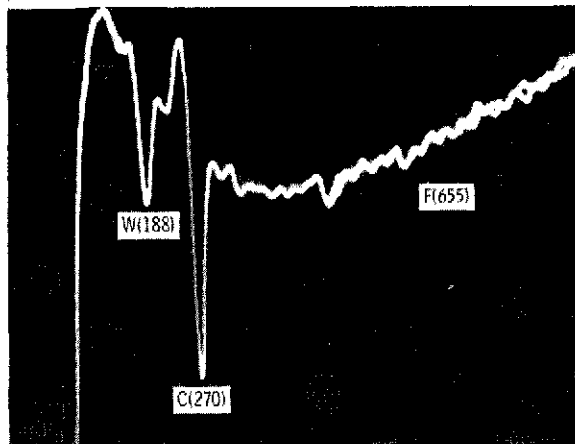
SECONDARY ELECTRON ENERGY, VOLTS →
 (b) AFTER 500-GRAM-LOAD STATIC CONTACT FOR 1 MINUTE WITH PTFE.

Figure 8. - Auger spectrogram of gold surface.

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DERIVATIVE OF SECONDARY ELECTRON ENERGY DISTRIBUTION, dN/dE 

(a) DISK WITH PTFE FILM GENERATED BY 100-GRAM-LOAD SLIDING AT VELOCITY OF 1 CENTIMETER PER SECOND AFTER 1 REVOLUTION OF DISK.



SECONDARY ELECTRON ENERGY, VOLTS →

(b) SAME SPOT DISK AS IN (A) AFTER 1-MINUTE TIME INTERVAL; 70-MICROAMPERE BEAM CURRENT.

Figure 9. - Auger spectrogram of tungsten disk.

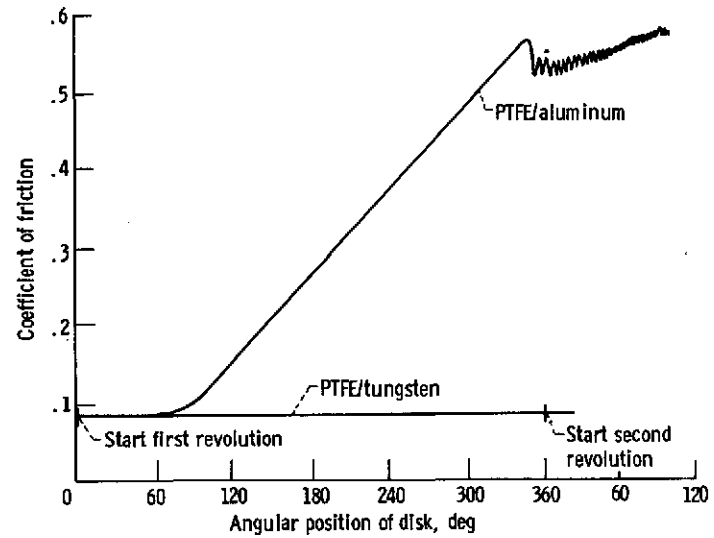


Figure 10. - Coefficient of friction of PTFE on aluminum disk and tungsten disk in vacuum. Sliding velocity, 0.07 centimeter per second; load, 250 grams.

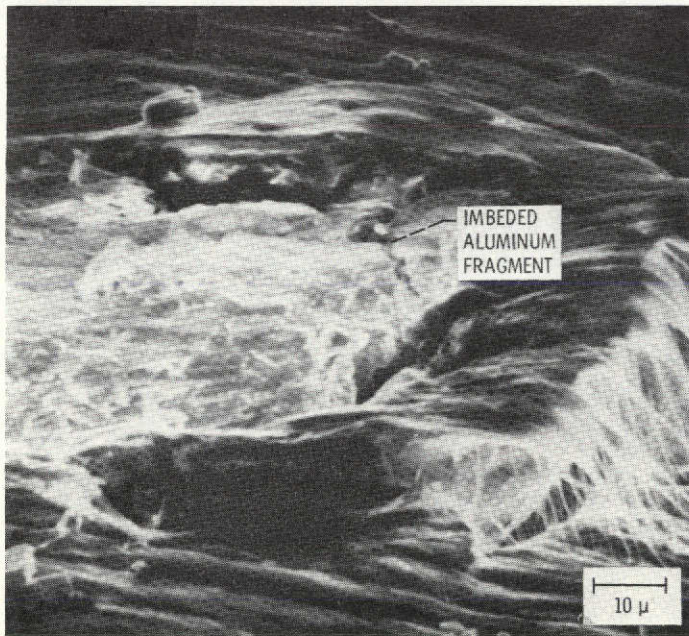
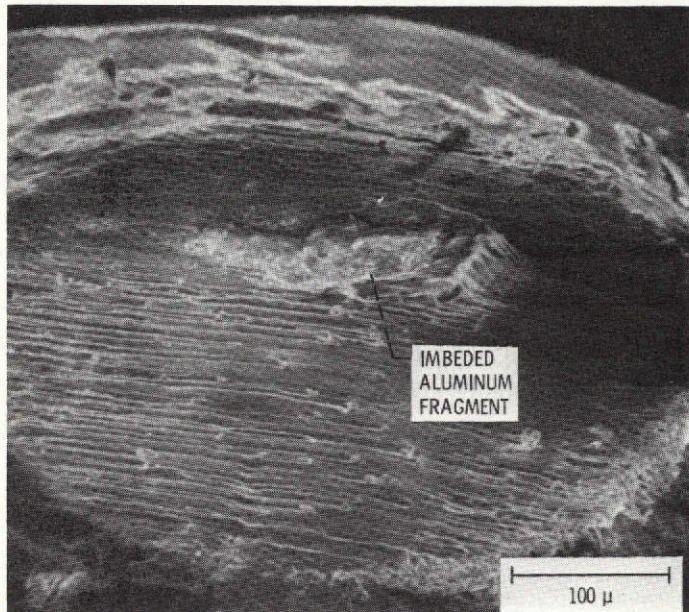


Figure 11. - PTFE - rider-wear scar showing lodged metal fragment run on (110) surface, single pass, 200 grams.

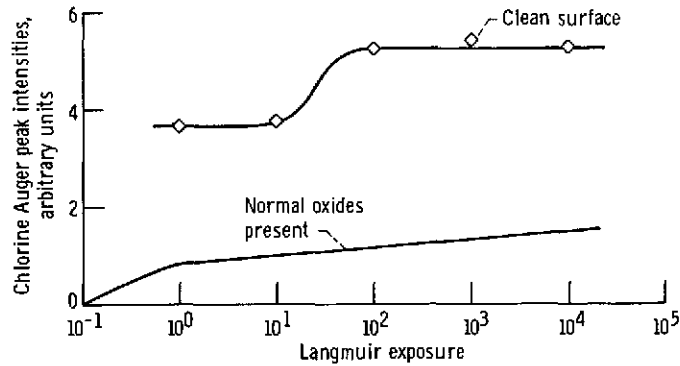


Figure 12. - Comparison of adsorption of vinyl chloride on iron surface in clean and oxidized states as evidenced by Auger analysis for chlorine. Adsorption conducted during sliding friction experiment.

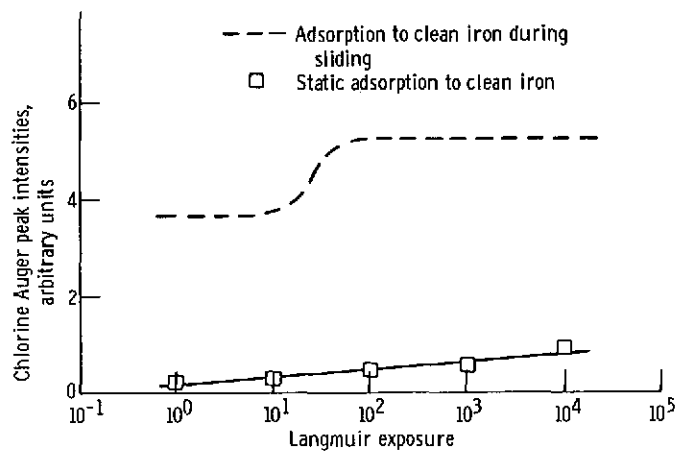


Figure 13. - Chlorine Auger peak intensity for vinyl chloride adsorption with and without sliding on iron surface. Rider specimen, aluminum oxide; sliding velocity, 30 centimeters per minute; load, 100 grams; temperature, 23° C.

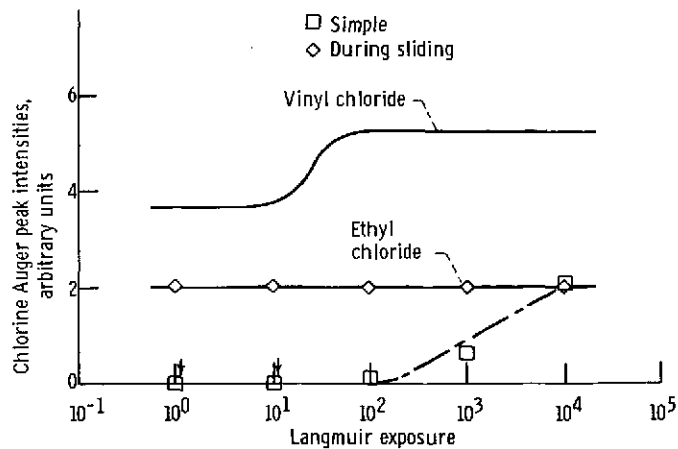
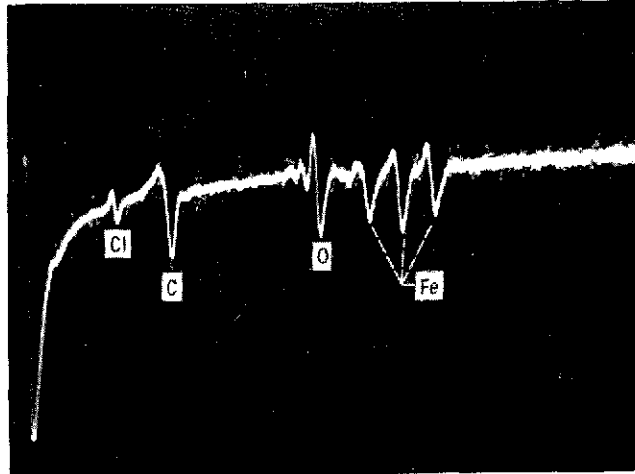
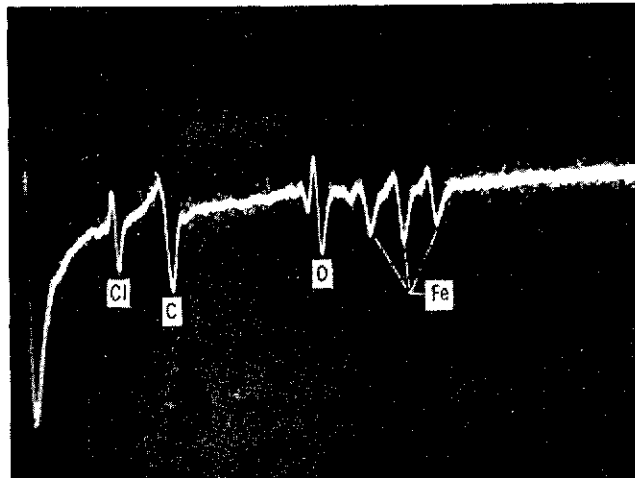


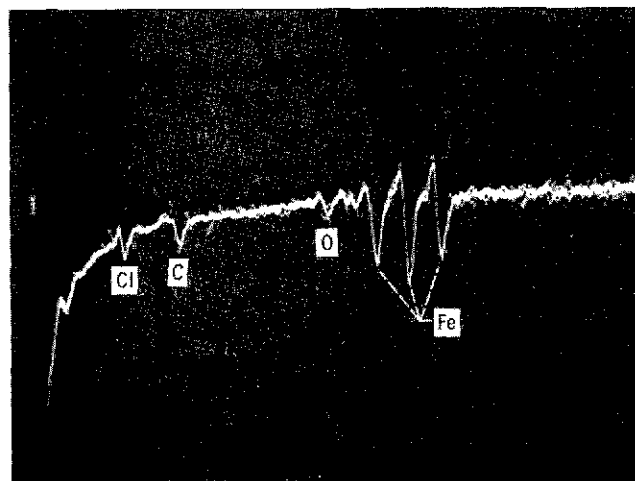
Figure 14. - Chlorine Auger peak intensities for vinyl chloride and ethyl chloride adsorbed on clean iron surface statically and during sliding. Rider specimen, aluminum oxide; sliding velocity, 30 centimeters per minute; load, 100 grams; temperature, 23° C.



(a) LOAD, 100 GRAMS.



(b) LOAD, 500 GRAMS.



(c) LOAD, 800 grams.

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Figure 15. - 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.

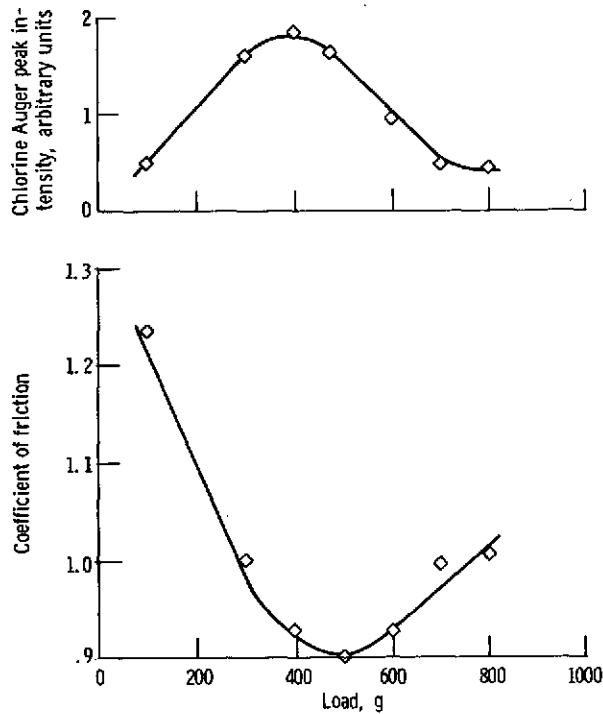


Figure 16. - Coefficient of friction and Auger chloride peak intensity as function of load for vinyl chloride on iron surface. Ambient pressure, 10^{-6} torr of vinyl chloride; rider specimen, aluminum oxide; sliding velocity, 30 centimeters per minute; temperature, 23° C; normal oxides present on iron surface.