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ADSORPTION OF ETHYLENE OXIDE AND VINYL CHLORIDE ON AN IRON (011) SURFACE AND EFFECT OF THESE FILMS ON ADHESION

by Donald H. Buckley

Lewis Research Center
Cleveland, Ohio 44135

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16. Abstract

The influence of chlorine and oxygen in the ethylene molecule on adsorption to iron and the effect of these films on adhesion was studied with LEED and Auger analysis. The displacement of these films from an iron surface by oxygen was also examined. Results indicate that the presence of chlorine or oxygen in the ethylene molecule reduces the adhesion of iron to itself. Greater amounts of oxygen were necessary to displace vinyl chloride and ethylene oxide from an iron surface than was needed to displace ethylene. Ethylene oxide formed a close packed structure whereas a (9 × 2) structure on the (011) surface of iron was observed with vinyl chloride.

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SUMMARY

An investigation was conducted to determine if the polymer forming gases, vinyl chloride and ethylene oxide, would chemisorb to a clean iron (011) surface and if they did, their effect on the adhesion of these iron surfaces. A further objective was to establish the stability of these films in the presence of oxygen. Both LEED and Auger spectrometer analysis were incorporated in the adhesion vacuum apparatus to study surface changes.

The results of the study indicate that both vinyl chloride and ethylene oxide will chemisorb to a clean iron (011) surface at 20^{o} C. Ethylene oxide formed a close packed and vinyl chloride a (9×2) structure. Adhesion forces between iron surfaces were lower in the presence of chemisorbed vinyl chloride and ethylene oxide than with chemisorbed ethylene. Both vinyl chloride and ethylene oxide were less readily displaced from an iron surface by oxygen then was ethylene. Greater amounts of oxygen were needed to displace ethylene oxide than were needed to displace vinyl chloride.

INTRODUCTION

The hydrocarbons methane, ethane, ethylene, and acetylene will chemisorb to a clean iron surface. The effectiveness of these hydrocarbons in reducing adhesion varies. Increasing the number of carbon atoms from one in methane to two in ethane results in a decrease in adhesive forces of iron to itself for the same number of hydrocarbon molecules on the surface. The degree of bond saturation also influences adhesive forces of iron to itself. With the hydrocarbons ethane, ethylene and acetylene, the adhesive forces were greatest with ethane and least with acetylene for equivalent surface coverage (ref. 1).

The stability of hydrocarbon films on a clean iron surface in the presence of oxygen also varies. All the previously mentioned hydrocarbons can be displaced from an iron surface by oxygen. The amount of oxygen necessary to achieve displacement of the hydrocarbon varies in the same manner as did resistance to adhesion. Methane was easiest to remove with oxygen and acetylene required the greatest amount of oxygen for removal. These results seem to indicate that there may be a relationship between the ability of hydrocarbons to reduce the adhesion of iron and the chemical bond strengths of the hydrocarbons with iron (ref. 1).

A logical extension of the work of reference 1 would be an examination of the effect of adding an active atom to the two carbon molecule and determine its influence on adhesion and resistance to displacement by oxygen. A surface active element widely used in lubricants is chlorine (ref. 2). Vinyl chloride ($CH_2 = CHCl$) is basically ethylene examined in reference 1, containing chlorine in place of one hydrogen. It is therefore an ideal structure to examine in studies of adhesion and oxidation stability.

Many practical hydrocarbon lubricants contain oxygen in their molecular structure (e.g., ethers). It would therefore be of interest to know what effect oxygen in a hydrocarbon molecule has on the interactions of the molecule with a clean iron surface. Ethylene oxide (CH_2OCH_2) is basically the ethylene structure in which the two carbon atoms are also bonded to a single oxygen. It is likely that the presence of oxygen would increase the binding energy of the moleucle to the iron surface.

The objectives of this investigation were to determine (1) if ethylene oxide and vinyl chloride will chemisorb to clean iron, (2) the influence of these hydrocarbons on adhesion of iron, and (3) should the films chemisorb, their stability in the presence of oxygen.

LEED (low energy electron diffraction) and Auger emission spectrometer analysis were used to determine the cleanliness of iron and to follow, structurally and chemically, the nature of surface changes that took place with chemisorption. Adhesion experiments were conducted with a 3.0-millimeter-diameter iron flat contacting a larger 8.0-millimeter-diameter flat surface. Both iron surfaces were single crystals having a (011) orientation. All experimentation was conducted at 20° C.

MATERIALS

The specimens were of triple zone refined iron with the following impurities in ppm: carbon, 8; oxygen, 7; nitrogen, 7; calcium, 2; nickel, 1; sulfur, 10; and all others in concentrations of less than 1 ppm. The ethylene oxide and vinyl chloride gases were reagent grade having a minimum purity of 99.9 percent. The oxygen gas used was reagent grade 99.995 percent minimum purity.

APPARATUS

The apparatus was used in these studies is shown schematically in figure 1. The single crystal surface mounted in the center of the chamber could be rotated 360°. Adhesion measurements were made on the crystal surface as shown in figure 1. The crystal was then rotated and both an Auger analysis and a LEED pattern were obtained from the crystal surface in the adhesion contact area. The crystal could also be moved in the lateral and vertical directions.

The crystal specimen was supported in the chamber by means of two metal rods (insulated) which were used to resistance heat the crystal. A 100-ampere ac power supply was used for resistance heating.

The 3.0-millimeter-diameter flat-ended iron single crystal, which contacted the single crystal metal surface, was mounted in a stainless steel holder which was in turn mounted to a 1.5-millimeter-diameter stainless steel beam. The beam was mounted in a bearing containing yoke. At the end of the beam beyond the pivot point, and opposite the smaller cylindrical specimen, was a small permanent magnet. Outside the chamber

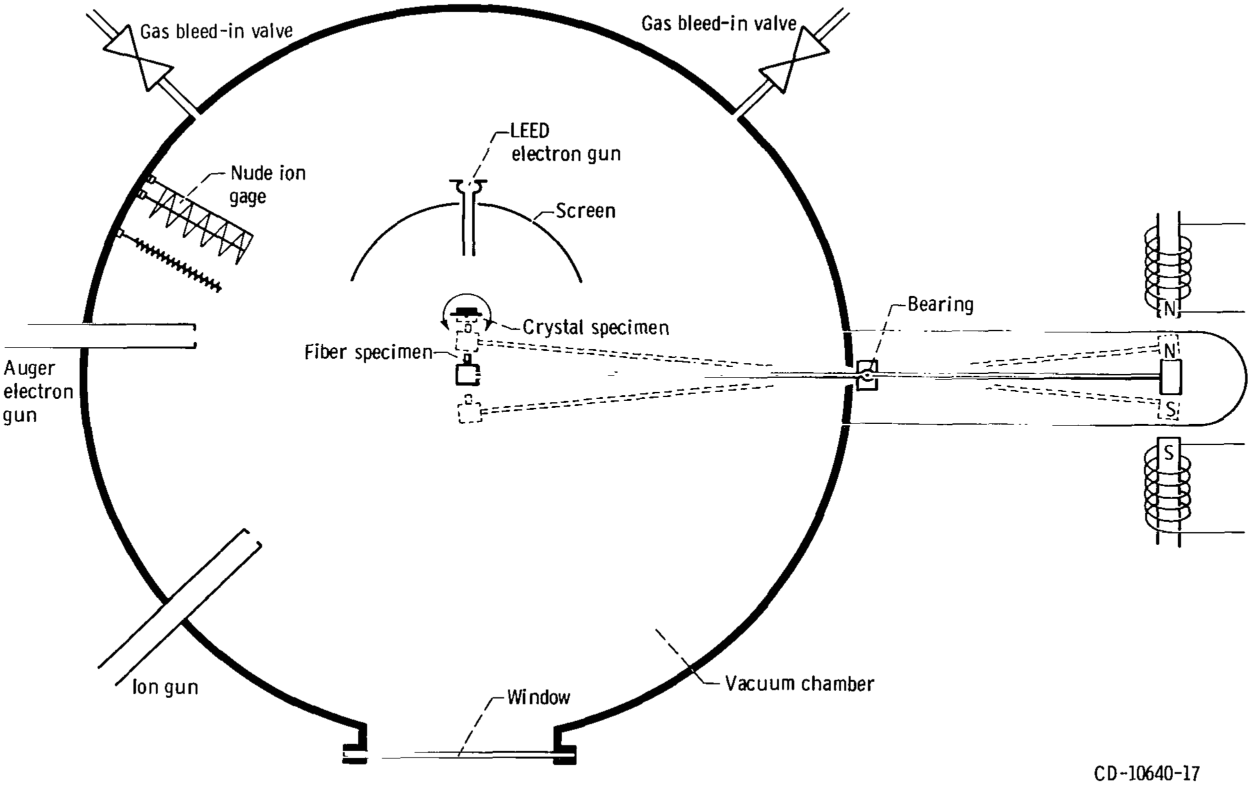


Figure 1. - Low-energy electron-diffraction (LEED) adhesion apparatus.

wall were two electromagnets. The permanent magnet and electromagnets were positioned in such a manner as to have like poles facing each other. A variation in the current applied to the magnets could be used to move the beam.

The current applied to the electromagnets was calibrated in terms of the force applied in the adhesion experiments. Load applied to the surfaces in contact was measured by current, as was the force required to separate the crystal surfaces.

The basic LEED and Auger systems were obtained commercially. The LEED electron optics and the vacuum system were of the standard type used by those engaged in LEED studies. The Auger spectrometer gun was located at a position 90° from the LEED gun (see fig. 1). The electron optics of the LEED was of the Varian three grid type with a fourth grid added for Auger analysis. The LEED beam diameter was 0.6 millimeter. The vacuum system consisted of vacsorb pumps, an ion pump, and a sublimation pump. The system pressure was measured with a nude ion gage and all experiments were conducted with the vacuum system in the range of pressure from 10^{-11} to 10^{-10} torr. No cryopumping was used.

EXPERIMENTAL PROCEDURE

The iron crystals used in this study were cut from a rod into specimens: one had a diameter of 8.0 millimeters and a thickness of 3.0 millimeters, and the second had a diameter of 3.0 millimeters and a length of 6.0 millimeters. The steps used to prepare the iron surfaces for adsorption studies are outlined in reference 1. The crystal orientations were checked after electropolishing and the specimens were placed in a vacuum tube furnace. They were heated to 600° C and held under vacuum for 24 hours, at which time hydrogen gas was admitted. The system was then reevacuated. This procedure was repeated for a two week period. The purpose of heating was to attempt to drive carbon and sulfur from the bulk to the surface. The admitted hydrogen was to remove the carbon and sulfur from the surface by reaction. After this treatment, the crystals were held in vacuum at 800° C for a prolonged period to remove hydrogen from the iron.

When the previous process was complete, the crystals were removed from the vacuum tube furnace, repolished, and electropolished. They were then placed in the apparatus for adhesion, LEED and Auger studies.

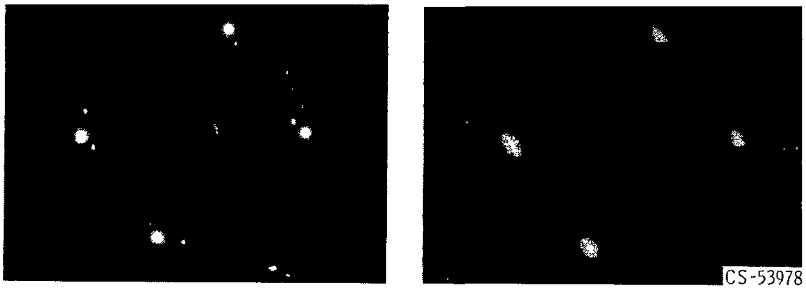
The vacuum system was evacuated to 10^{-11} torr and the crystals were ion bombarded for 12 hours after the system pressure was raised to 8.0×10^{-5} torr with argon gas. The ion gun voltage was 250 volts. The crystals were then heated to 250° C for 10 minutes to remove trapped argon and a clean iron surface for adsorption studies was obtained as indicated by LEED and Auger analysis.

The gases vinyl chloride, ethylene oxide, and oxygen used in this investigation were bled into the vacuum chamber through a variable leak valve. The pressure in the system was raised from 1×10^{-10} to 1×10^{-6} torr and maintained at that pressure for a sufficient period of time to achieve the desired Langmuir exposure.

RESULTS AND DISCUSSION

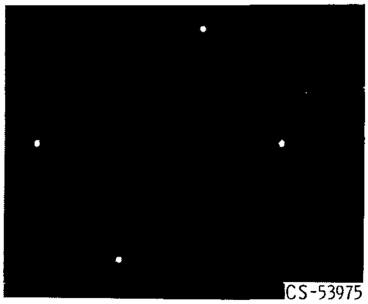
Clean Iron

Iron is an extremely difficult material to obtain in the clean state. Extremely small amounts of bulk impurity, such as sulfur, carbon, and nitrogen, will diffuse to the surface from the bulk on heating. An earlier study has shown that sulfur present in concentrations as low as 10 parts per million in the bulk will diffuse to an iron surface producing ordered structures readily detectable by LEED (ref. 3).



(a) Carbon containments.

(b) Argon bombarded.



(c) Clean surface (110 V).

Figure 2. - LEED patterns of iron (011) surface with carbon present and after argon ion bombardment.

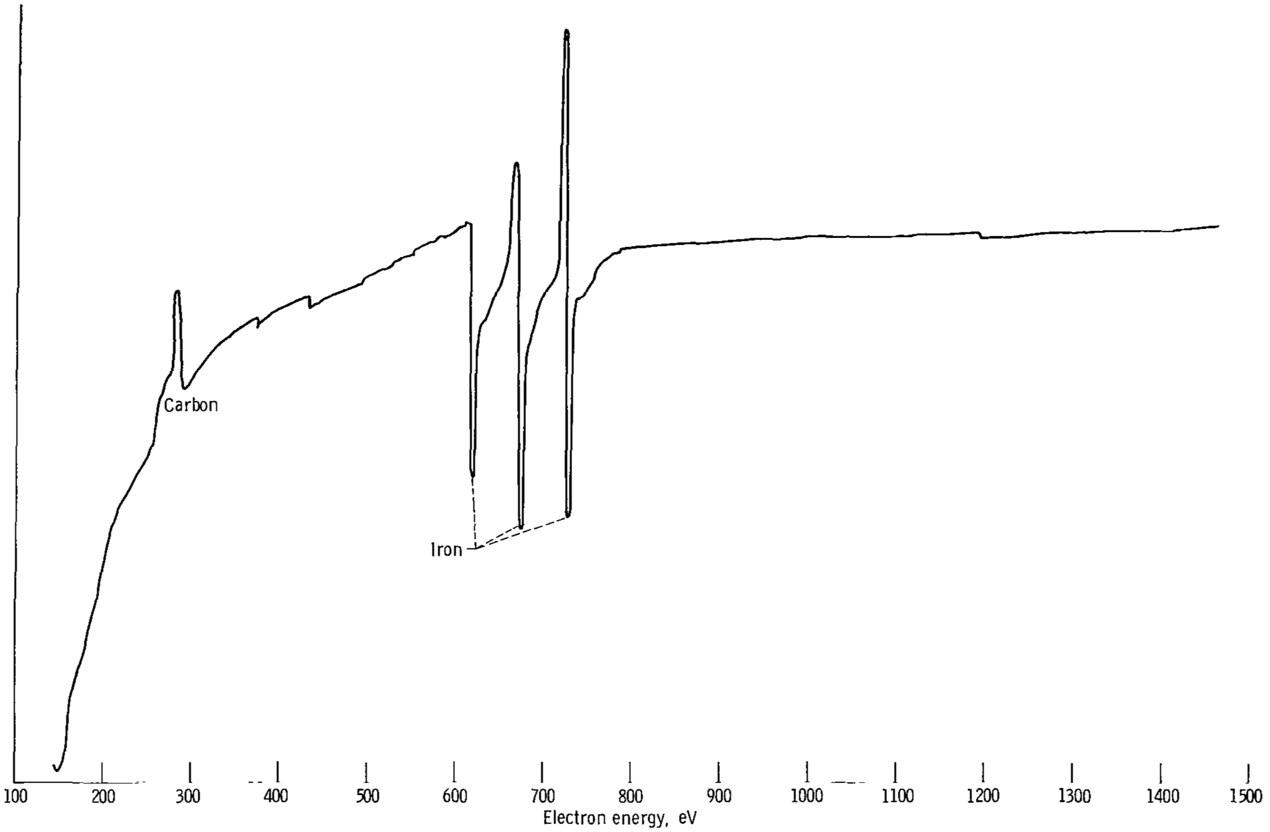


Figure 3. - Auger spectrometer trace of iron (011) surface with carbon present on surface.

When the sulfur concentration in iron has been depleted sufficiently, heating of the material will result in segregation of carbon on the iron surface. The iron used in this study contained initially only 8 parts per million of carbon in the bulk. After obtaining a clean surface and then heating the clean iron, carbon diffused to the surface producing the ringed LEED pattern of figure 2(a). Carbon was also observed to produce a ringed structure on the face centered cubic metal platinum in reference 4. Figure 3 is an Auger spectrometer trace of the iron surface with the ringed structure of figure 2(a) present. The Auger peaks detected are for iron and carbon.

Argon ion bombardment of the surface shown in figure 2(a) resulted in removal of the carbon as indicated by the LEED pattern of figure 2(b). The diffuse nature of the four iron diffraction spots in figure 2(b) is due to argon which has embedded in the iron. Heating of the iron surface in figure 2(b) for 10 minutes at 250° C removed the argon as shown by the sharp bright iron diffraction spots of figure 2(c). Auger spectrometer analysis of the iron surface revealed the absence of all peaks other than those attributable to iron in the range of 0 to 1500 electron volts.

Vinyl Chloride Adsorption

Earlier reported studies on the adsorption of hydrocarbons to iron indicate that the adsorption required an activation energy and that chemisorption did not occur at 20° C (ref. 5). In reference 1, however, where iron surfaces were clean, the hydrocarbons methane, ethane, ethylene, and acetylene were all found to chemisorb to iron at 20° C. Pure hydrocarbons are generally not good lubricants and surface active atoms are frequently added to the molecular structure of hydrocarbons to improve their lubricating characteristics (ref. 6).

Vinyl chloride and ethylene were adsorbed to the clean iron surface of figure 2(c) to determine the effect of the chlorine in the ethylene molecule on adsorption. The iron was exposed to from 1 to 1000 Langmuirs ($1L = 1\times10^{-6}$ torr-sec) of vinyl chloride. With as little as 10 Langmuirs exposure, carbon and chlorine were detected by a Auger analysis of the surface. Monitoring the Auger chlorine and carbon to iron peak intensities indicated that adsorption was nearly complete after an exposure of 100 Langmuirs of vinyl chloride. The specimen was exposed to a total of 1000 Langmuirs of vinyl chloride. Figure 4 is an Auger trace of the iron surface with the vinyl chloride present on the surface. Large chlorine and carbon peaks appear in the trace in addition to the iron. Figure 4 shows that both chlorine and carbon are present on the surface. This indicates that dissociation probably did not occur but, if it did, the fragments remained on the surface.

With the adsorption of vinyl chloride on the iron surface at 20° C, the background showed evidence of random adsorption. The black background between iron diffraction spots in figure 2(c) became white with increased exposure to vinyl chloride. Heating for 10 minutes at 200° C brought about surface rearrangement to develop the (9×2) structure in the LEED pattern of figure 5(a). The LEED pattern of figure 5(a) is interpreted in figure 6.

In figure 6 the arrangement of iron atoms in the (011) plane is indicated. The unit mesh or basic structure which completely defines the surface with respect to LEED is indicated. The unit mesh represents the LEED pattern seen in figure 2(c) for clean iron. The unit mesh of iron has a dimension of a_0 in the [20] direction (see fig. 6 insert) and $\sqrt{2}\, a_0$ in the [02] direction.

With vinyl chloride on the iron (011) surface producing the LEED pattern of figure 5(a), the suggested arrangement of the hydrocarbon is as indicated in figure 6 (left side). The vinyl chloride takes on the same lattice spacing as the iron in the [02] direction. The lattice spacing in the [20] direction is $9a_0$ or nine times the spacing of the iron. Using the terminology of surface crystallography, this would be referred to as a $Fe(011)C(9 \times 2)\text{-}CH_2CHCl$ structure (ref. 7).

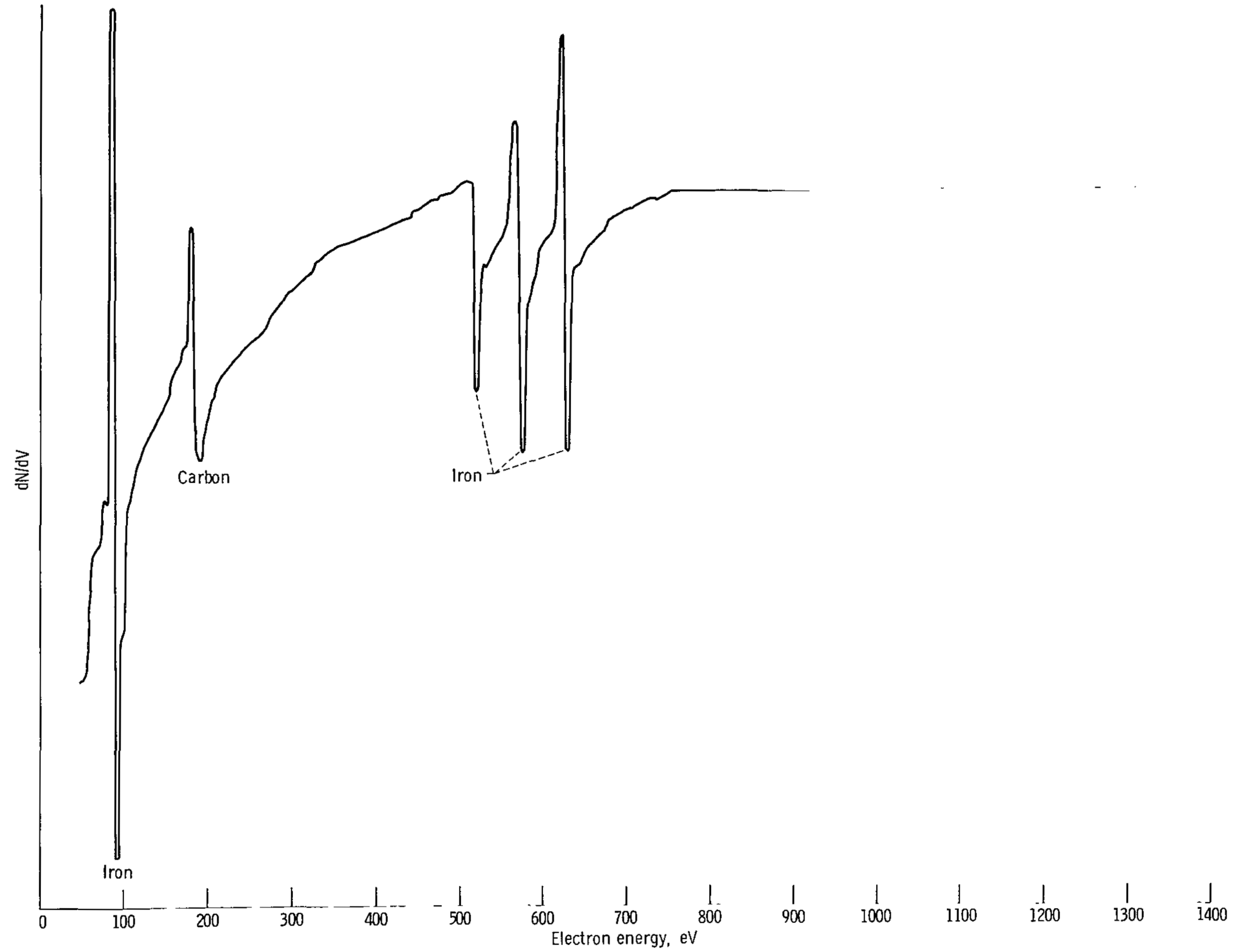
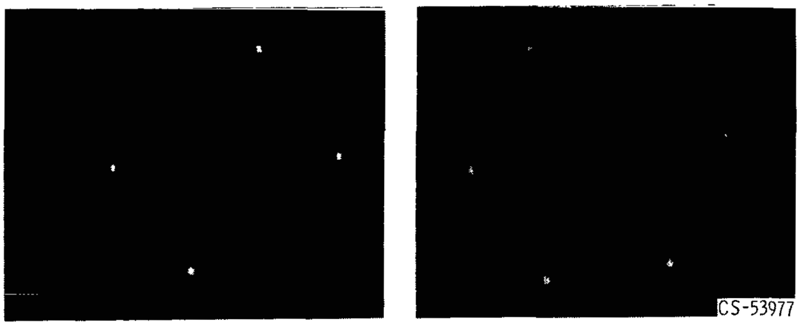


Figure 4. - Auger emission spectrometer trace in iron (001) surface with vinyl chloride present.



(a) $CH_2 = CHCl$.

(b) $\overset{O}{CH_2 - CH_2}$.

Figure 5. - LEED patterns obtained with two hydrocarbons on iron (011) surface (exposure, 1000 Langmuirs).

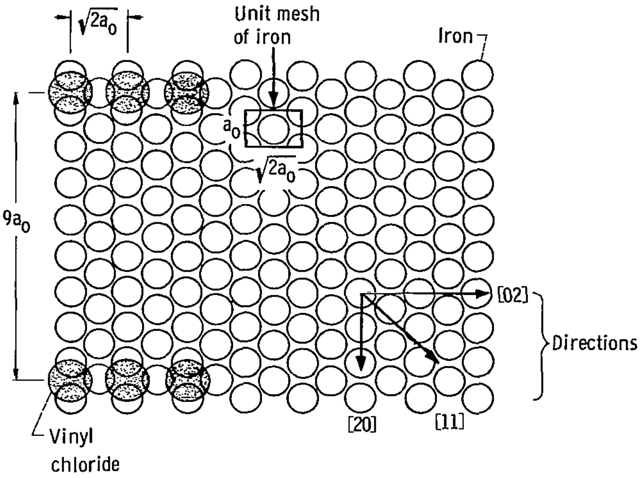


Figure 6. - Atomic model of iron (011) surface with suggested locations of adsorbed vinyl chloride molecules.

TABLE I. - EFFECT OF VARIOUS HYDROCARBONS ON ADHESION OF CLEAN IRON

Hydrocarbon chemisorbed to iron	Adhesive force,[a] dynes
Methane CH_4	<400
Ethane C_2H_6	280
Ethylene $H_2C=CH_2$	170
Acetylene $HC\equiv CH$	80
Vinyl chloride $H_2C=CHCl$	30
Ethylene oxide $\overset{O}{H_2C-CH_2}$	<10

[a] Contact load, 20 dynes; ambient pressure 10^{-10} torr; temperature, 20° C; both surfaces (011) planes.

Two sources of energy were involved in arriving at the structure of figure 5(a) and these could have produced a loss of some of the adsorbed hydrocarbon by desorption. The first is that the surface was heated to 200° C for 10 minutes to produce the ordered structure of figure 5(a). The second is the primary beam of the Auger electron gun (2000 V) which could also contribute to desorption.

Adhesion measurements were made with an ordered film of vinyl chloride present on the iron (011) surface and these results are presented in table I together with adhesion results obtained with methane, ethane, ethylene, and acetylene. It can be seen from table I that the number of carbon atoms and the degree of bond saturation influence adhesion. Comparing the adhesion results for ethylene with that of vinyl chloride, the addition of chlorine to the molecule has a marked influence on adhesion. It might be anticipated that the addition of more chlorine to the molecule would reduce the adhesion of iron to itself further. The more chlorine present in the ethylene molecule the more reactive it becomes (ref. 8).

Effect of Oxygen on Adsorbed Vinyl Chloride Film

In order to determine the stability of the vinyl chloride film in the presence of oxygen the surface containing the ordered adsorbed vinyl chloride film was exposed to various amounts of oxygen. Auger spectrometer traces were taken with each exposure and the relative intensity of the chlorine and carbon to oxygen with oxygen exposure is presented in figure 7. Complete removal of the carbon occurred at 1000 Langmuirs

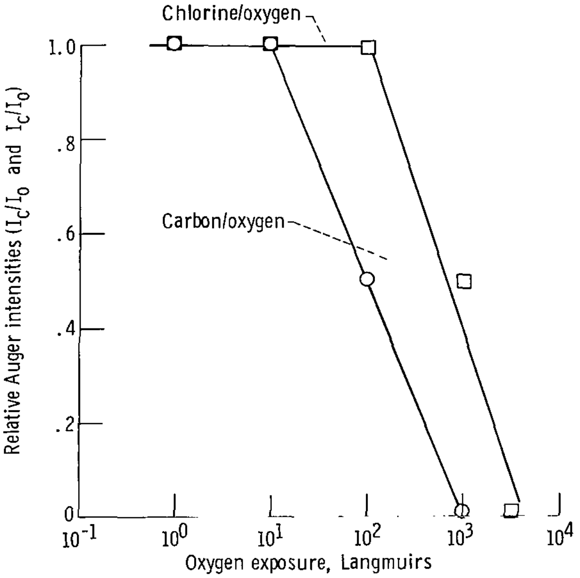


Figure 7. - Auger emission spectrometer analysis of oxygen interaction with layer of vinyl chloride present on an iron (011) surface.

oxygen exposure while 5000 Langmuirs exposure was necessary for complete chlorine removal. The data of figure 7 indicate two effects. The first is that oxygen will completely displace the vinyl chloride from the iron surface. The second observation to be made from figure 7 is that the hydrocarbon portion of the molecule is removed more readily than the chlorine.

The relative resistance of vinyl chloride to displacement by oxygen is compared with the resistance of ethylene to displacement by oxygen in figure 8. In figure 8 the carbon peak intensity to the oxygen peak intensity is compared for the two hydrocarbons. Figure 8 shows that ethylene containing chlorine in its molecular structure is more resisttant to displacement from the iron surface than is ethylene. The results of figures 7 and 8 indicate that chlorine must bond to the iron surface. If this were not the case it would be difficult to explain the presence of chlorine on the iron surface after carbon was completely removed in figure 7. Further, in figure 8, if only carbon were bonding to the iron surface, marked differences in the displacement of ethylene and vinyl chloride would not be expected.

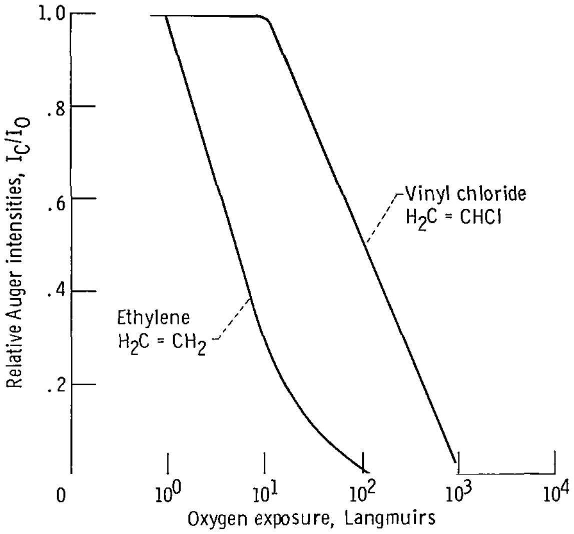


Figure 8. - Auger emission spectrometer analysis of interaction of oxygen with chemisorbed layer ethylene and vinyl chloride.

Adsorption of Ethylene Oxide on Iron

The iron (011) surface was exposed to various amounts of ethylene oxide just as it was to vinyl chloride. The ethylene oxide chemisorbed very readily to the iron surface at 20° C. At an ethylene oxide exposure of 10 Langmuirs, ethylene oxide could be readily detected on the iron surface with Auger analysis. Figure 9 is an Auger spectrometer trace of the iron surface after exposure to 100 Langmuirs of ethylene oxide. Both the carbon and the oxygen in the ethylene oxide structure were detected (see fig. 9).

The basic iron LEED pattern of figure 2(c) showed evidence of random adsorption of the ethylene oxide on the iron surface. The background between iron diffraction spots became brighter with each additional ethylene oxide exposure. After 1000 Langmuirs exposure, the surface was heated for 10 minutes at 200° C to determine if the ethylene oxide would order on the surface. The LEED structure of figure 5(b) resulted. The ethylene oxide underwent rearrangement to produce an ordered close packed surface structure. Thus, the ethylene oxide provides a full film of maximum density upon heating. This type of packing was not observed with ethylene. The presence of the oxygen in the molecule promotes fuller coverage.

It might be anticipated that a structure such as that of figure 5(b) would provide iron with good resistance to adhesion since the iron is completely covered by a close packed

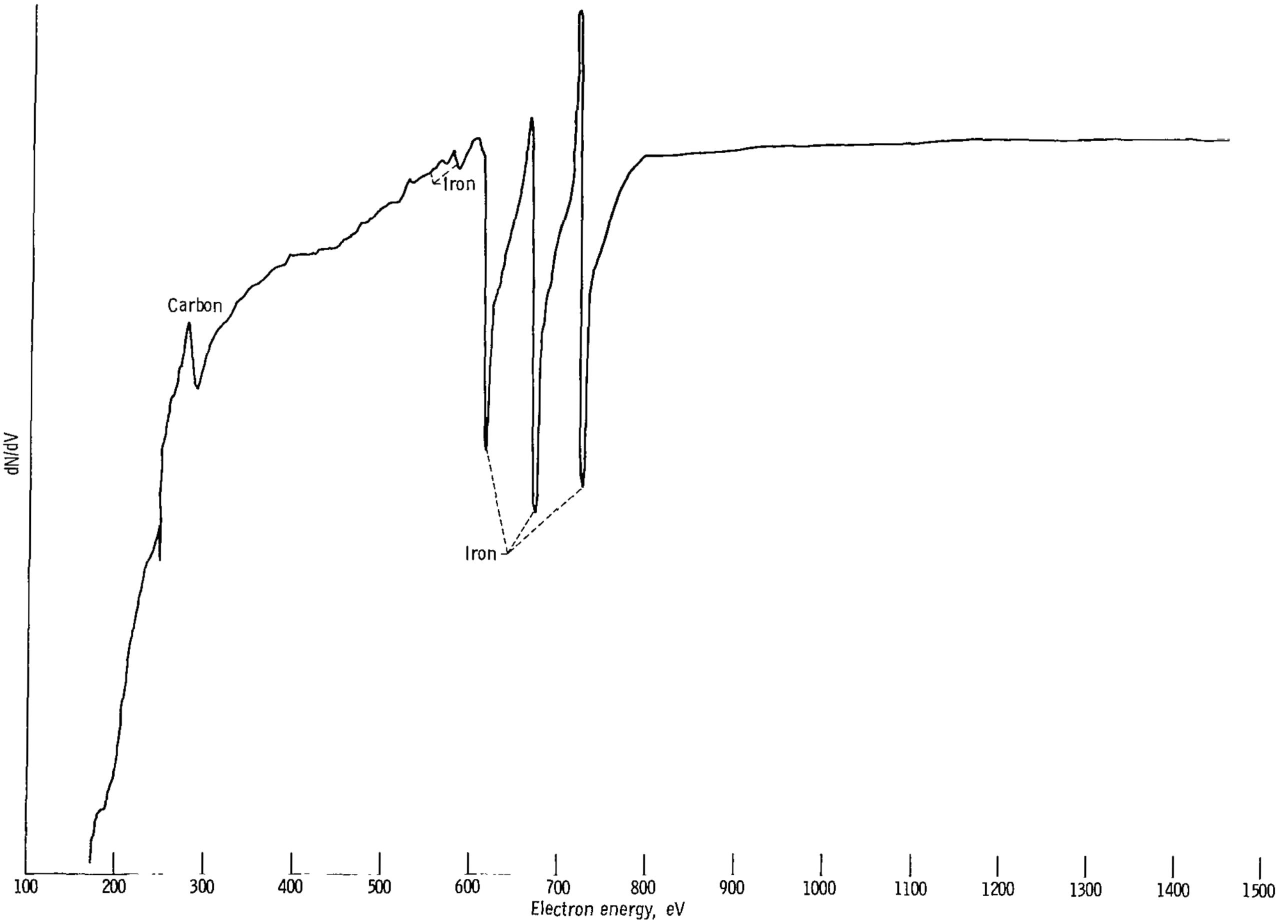


Figure 9. - Auger emission spectrometer trace of iron (001) surface with ethylene oxide present.

structure. The adhesion data of table I for iron to itself with ethylene oxide present indicates that it does. The lowest adhesive forces for iron in the presence of a hydrocarbon were achieved with an ethylene oxide surface film. These data, together with those obtained in reference 3 with sulfur adsorbed to the (011) surface of iron, indicate that a close packed structure is ideal for the achievement of a minimum in the adhesion of iron surfaces. It would be of interest to know what characteristics of a molecular structure leads to close packed formation and further if close packing of an adsorbing molecule will occur on all orientations.

Oxygen Displacement of Ethylene Oxide

In order to determine the stability of the ethylene oxide film of figure 5(b) in the presence of oxygen, the film was exposed to various amounts of oxygen. After varying oxygen exposures, the Auger carbon and oxygen peaks were monitored and the results of relative Auger intensities are presented in figure 10. The results of figure 10 indicate that, with a 5000 Langmuir oxygen exposure, the ethylene oxide film was completely displaced. Figure 11 shows the growth of the oxygen peak at 100 Langmuirs oxygen exposure compared to that observed with only ethylene oxide present on the surface, figure 9.

The presence of oxygen in the basic ethylene structure increases the hydrocarbon's resistance to displacement from the iron surface by oxygen. The relative ability of the

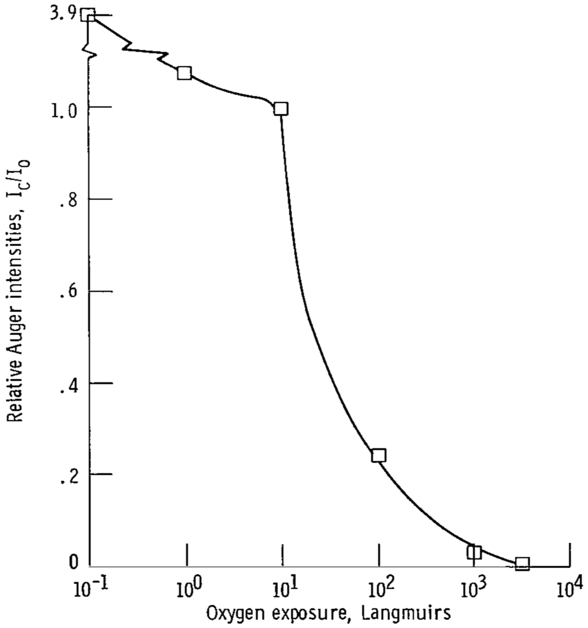


Figure 10. - Auger emission spectrometer analysis of interaction of oxygen with chemisorbed layer of ethylene oxide on an iron (011) surface.

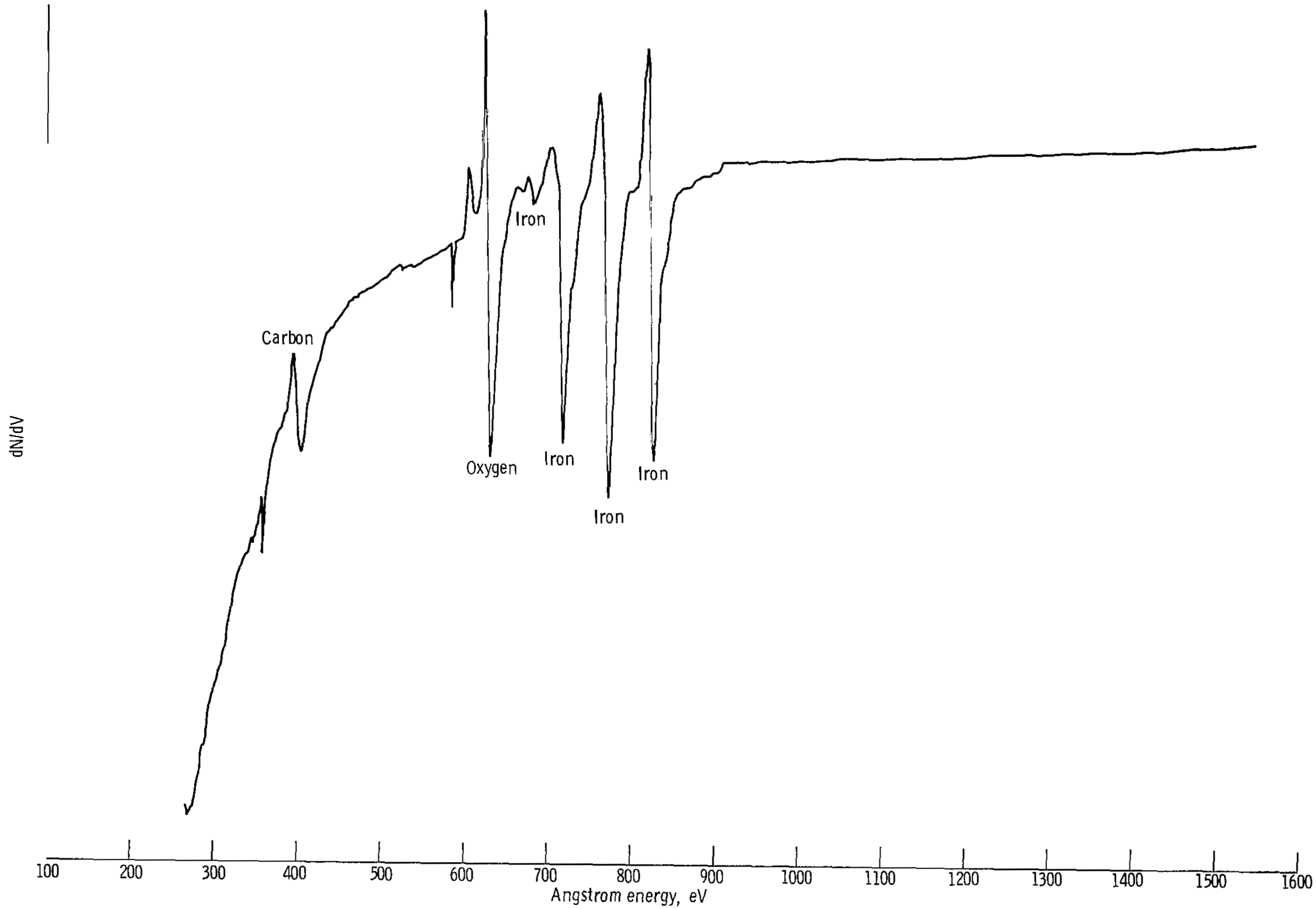


Figure 11. - Auger emission spectrometer trace of iron (011) surface with ethylene oxide and exposed to oxygen (100 Langmuir).

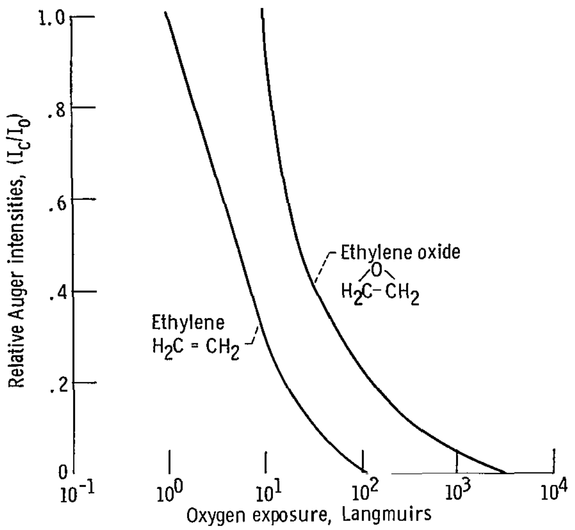


Figure 12. - Auger emission spectrometer analysis of interaction of oxygen with chemisorbed layers of ethylene and ethylene oxide with an iron (011) surface.

two hydrocarbons to withstand displacement by oxygen is shown in figure 12, where the carbon to oxygen Auger relative peak intensities are compared. The presence of the oxygen in the ethylene molecule increases the exposure necessary to achieve displacement by oxygen. While 100 Langmuirs of oxygen was adequate for the complete displacement of ethylene, 5000 Langmuirs were necessary to accomplish the same effect with ethylene oxide. These data indicate that oxygen present in the hydrocarbon molecule is desirable where stronger bonding of a hydrocarbon to an iron surface is desired.

CONCLUSIONS

Based on the adsorption and adhesion measurements made in this investigation with iron (011) surfaces, the following conclusions are drawn:

1. The presence of chlorine or oxygen in the basic ethylene hydrocarbon structure reduces the adhesion of iron to itself. The adhesive forces with ethylene was more than five times that obtained with vinyl chloride and ethylene oxide.

2. The addition of chlorine or oxygen to the ethylene structure increases the resistance of the hydrocarbon to displacement from the iron surface by oxygen. With both vinyl chloride and ethylene oxide, a greater amount of oxygen was necessary to displace the film than was necessary for ethylene.

3. With ethylene oxide full monolayer surface coverage, heating of the iron resulted in the formation of a close packed layer. Vinyl chloride after heating formed a Fe(011)C(9 × 2)-$H_2C = CHCl$ structure on the surface of iron.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 24, 1970,
129-03.

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