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LEED AND AUGER STUDIES OF EFFECT OF OXYGEN ON ADHESION OF CLEAN IRON (001) AND (011) SURFACES

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16. Abstract

Adhesion experiments were conducted with the (001) and (011) surfaces of iron. A 3.0-mm-diameter flat contacted a larger crystal surface at forces from negligible load to 350 dynes (0.0035 N). With the presence of even fractions of monolayer of oxygen, adhesion of iron to itself decreased appreciably. Adhesion decreased with increasing surface coverage. The minimum in adhesion was observed with a full FeO layer formed on the (001) surface of iron. A comparison of adhesion data (obtained in this study) with oxygen on the surface and that with hydrogen sulfide indicates that for, equivalent surface coverages, the relative effectiveness of the absorbed species in reducing adhesion may be related to the percent surface coverage.

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SUMMARY

Adhesion studies were conducted in a vacuum system incorporating LEED and Auger emission analysis of the surfaces. Two orientations of iron were examined in the presence of various amounts of oxygen on the surfaces. The orientations were the (001) and (011). Adhesion measurements were made with a 3.0-millimeter-diameter flat contacting a larger single-crystal flat of the same material and orientation. Applied load varied from negligible (<10 dynes or 0.0001 N) for clean iron to a maximum of 350 dynes (0.0035 N) where oxygen was present. All experiments were conducted in a vacuum of 10^{-11} to 10^{-10} torr.

The results of the study indicate that even fractions of a monolayer of oxygen are sufficient to reduce the adhesion of clean iron surfaces. Further, adhesion decreased with increases in the amount of oxygen surface coverage. The minimum in adhesion was observed with complete coverage of the iron (001) and (011) surfaces and the resulting formation of iron oxide (FeO). When adhesion data of this study for various oxygen surface coverages were compared with those obtained in an earlier study with hydrogen sulfide, the results indicate that the relative effect of the adsorbed species in reducing adhesion may be related to the percent surface coverage.

INTRODUCTION

As is well known, oxygen and oxides play a very important role in the adhesion, friction, and wear behavior of iron and iron-base alloys. Further, not only the presence of oxides but the type of oxide will exert an influence on these properties. In practical lubrication devices, nascent metal surfaces may be generated by wear, and chemisorption of oxygen may occur. The specific effect of chemisorbed oxygen on high purity iron has not been examined. Little is known about the influence of a monolayer or less of oxygen on the adhesion of iron. Although practical lubrication systems involve the use of complex alloys of iron, a better fundamental understanding of oxygen-iron interactions can be achieved if high purity iron with an atomically clean surface is used for the study of such interactions. Small concentrations of impurities or alloying agents in the bulk can markedly alter the adsorption process, particularly where "equilibrium segregation" occurs.

Since adhesion is fundamental to both friction and adhesive wear, adhesion experiments can be very effective in providing insight into the influence of adsorbed oxygen on the behavior of the friction and wear of iron and iron alloys. Even fractions of a monolayer of oxygen on an iron surface will reduce the valence electrons available for cohesive iron bonding across an interface. These surface changes should be reflected in reduction of measured adhesive forces.

The objective of this investigation was to determine the influence of both adsorbed oxygen and of iron oxide on the adhesion of iron single crystals. The iron surfaces were examined, before adsorption as well as before and after adhesion with LEED (low energy electron diffraction) and with AES (Auger emission spectrometry). Two orientations of iron were examined, a (001) and a (011) surface. Adhesion experiments were conducted with like atomic planes in contact. The applied load varied from negligible (<10 dynes or 0.0001 N) to 350 dynes (0.0035 N).

MATERIALS

The specimens were triple-zone refined iron and had 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 oxygen gas used was reagent grade containing a maximum impurity level of 60 ppm; of these, 15 were argon, 12 krypton, 15 nitrogen, 1 zeon, 2 water, 1 carbon monoxide or carbon dioxide, and total hydrocarbon of 12 ppm.

APPARATUS

The apparatus 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°. This rotatability allowed for the making of adhesion measurements on the crystal surface shown in figure 1, then rotating the crystal and obtaining both an Auger analysis and a LEED pattern from the crystal surface in the adhesion contact area. The crystal could also be moved in the lateral and vertical directions.

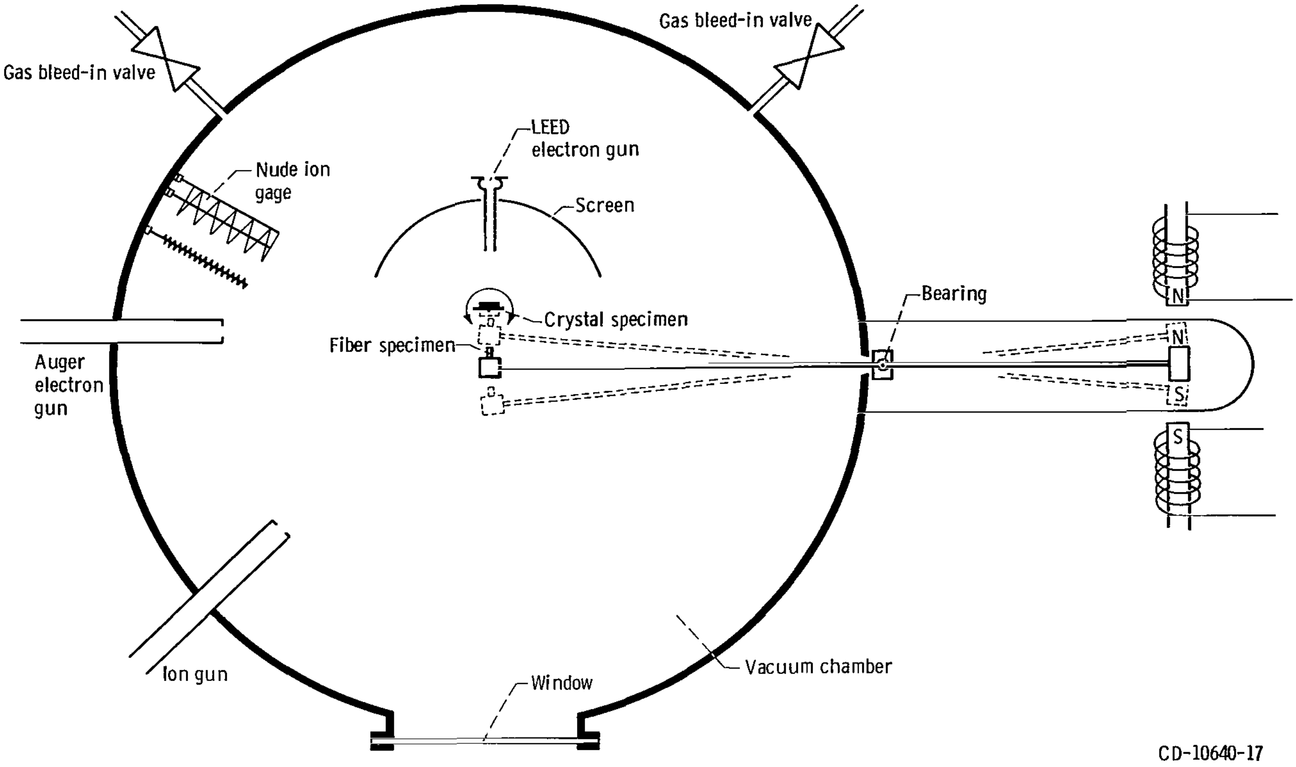


Figure 1. - Low-energy electron-diffraction (LEED) adhesion apparatus.

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 alternating current 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 wall were two electromagnets. The permanent magnet and the electromagnets were positioned so that like poles were 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 three-grid Varian with a fourth grid added for Auger analysis. The LEED beam diameter was 0.6 millimeter. The vacuum system consisted of vacuum adsorption 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 pressures 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 having a diameter of 8.0 millimeters and a thickness of 3.0 millimeters and the other specimen having 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 block diagram form in figure 2. 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

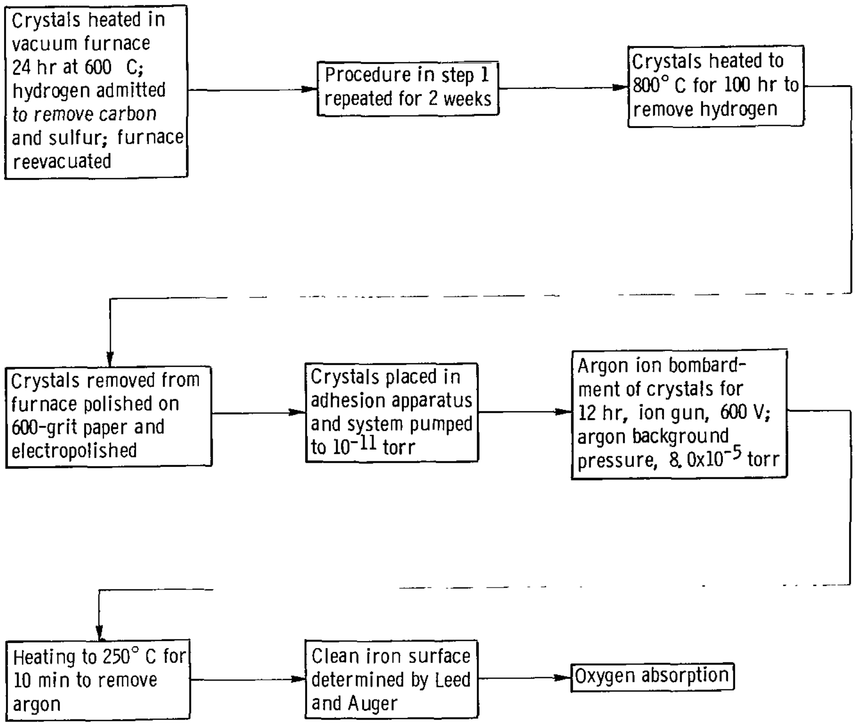


Figure 2. - Steps in iron surface preparation, cleaning, and adsorption.

then reevacuated. This procedure was repeated over 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 above process was completed, the crystals were removed from the vacuum tube furnace, repolished, and electropolished. They were then placed into 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 600 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.

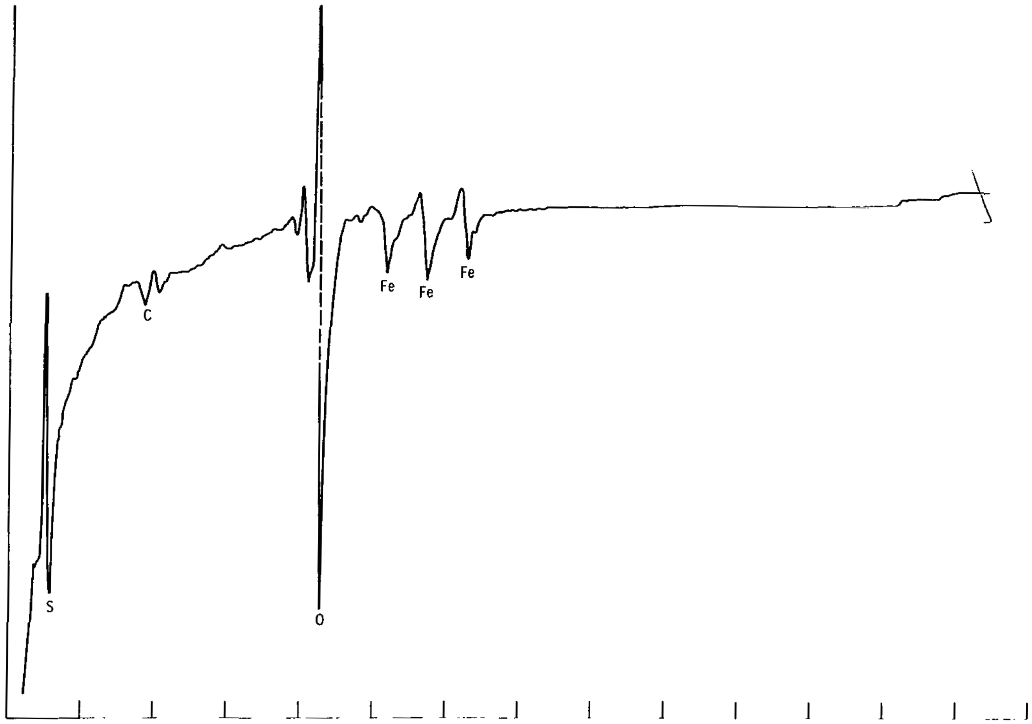
RESULTS AND DISCUSSION

Auger analysis of an iron (001) surface after heating for a prolonged period in vacuum revealed the presence of sulfur, carbon, and oxygen on the surface as indicated in the Auger trace of figure 3(a). Argon ion bombardment, at an argon pressure of 8.0×10^{-5} torr with an ionization voltage of 600 volts, resulted in the removal of these elements from the iron (001) surface. Figure 3(b) is the Auger trace of the surface after argon ion bombardment.

In figure 3 the derivative of the secondary electron energy distribution dN/dV is plotted as a function of electron energy (ev). The three characteristic peaks observed for iron are indicated in figure 3. In addition, after argon ion bombardment, small peaks (fig. 3(b)) for argon and nitrogen were observed. The argon peak is the result of embedded argon in the surface from ion bombardment. The nitrogen peak resulted in all probability from the vacuum chamber background gas. Each time the vacuum chamber is opened, it is bled to atmospheric pressure with dry nitrogen. Since the ionization potential of nitrogen is less than that for argon, some nitrogen is also bombarding the surface and being buried in it. Heating to 250° C for 10 minutes removed these gases from the surface. It is of interest to note that the iron peaks increase in intensity with the removal of sulfur, carbon, and oxygen.

The iron (001) surface is shown in the form of sphere model in figure 4. The lattice constant a_o is equal to 2.86 Å (ref. 1). The directions on the surface are also shown in figure 4.

Adhesion experiments were made with clean iron (001) surfaces in contact. The surfaces were simply brought into touch contact. Adhesive forces were sufficiently high



(a) After simple heating in vacuum.

(b) After argon ion bombardment.

Figure 3. - Auger emission spectrometric analyses of iron (001) surface after treatment.

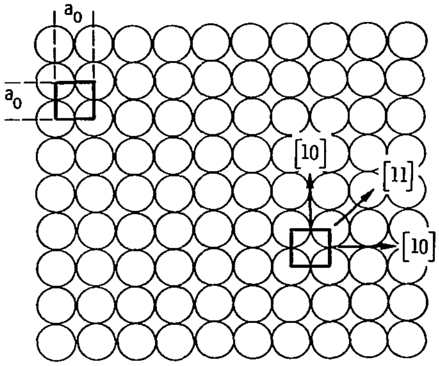
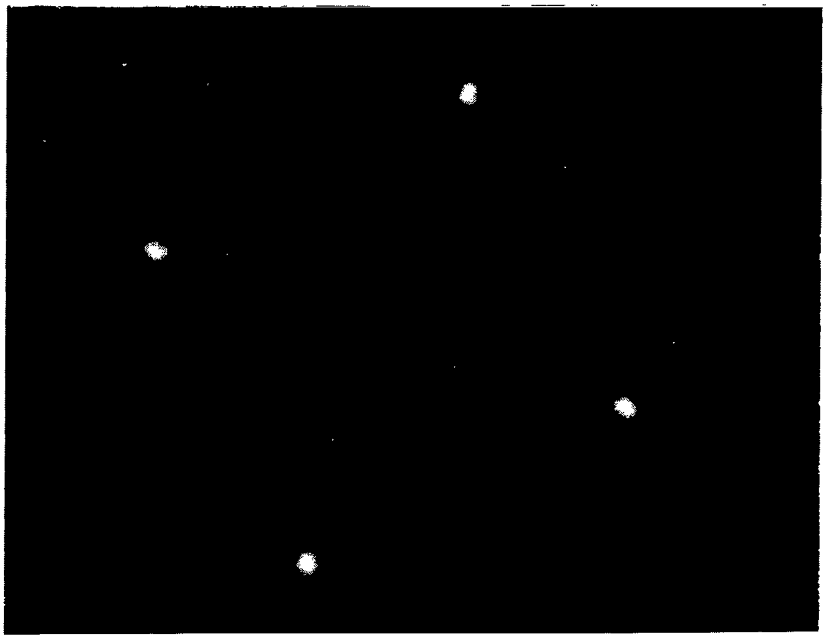


Figure 4. - Arrangement of atoms in iron (001) surface.

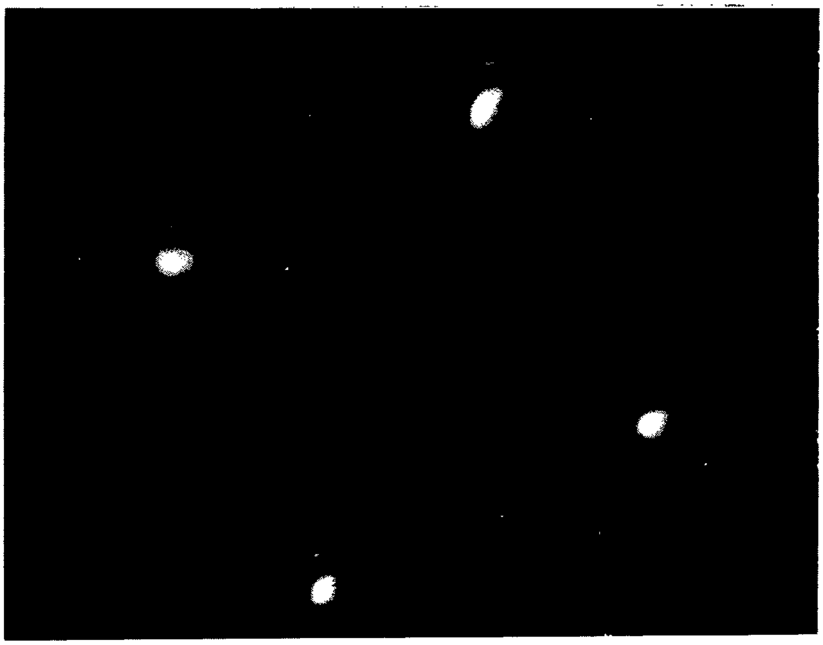
that they exceeded the measuring capability of the apparatus (400 dynes or 0.004 N). The strong forces holding the iron surfaces in contact are a function of the strength of the iron cohesive bond and the true area of interfacial contact when clean iron surfaces are brought together. The bonding force of iron to itself with the (001) surfaces and the (011) surfaces in contact in this study and with polycrystalline iron in another study (ref. 2) are stronger than has been observed in similar copper adhesion experiments on (100), (110), (111), and polycrystalline surfaces of copper.

Under negligible load conditions and with electropolished flats of crystal faces (representing a larger ratio of true to apparent contact area) the effect of differences in bulk elastic and plastic behavior is minimized. Calculations of contact area, assuming negligible loading, indicate that differences in contact area alone could not account for differences in observed adhesion forces. The differences in the adhesion of iron to iron and copper to copper will reflect differences in cohesive binding energies. The cohesive energies of the transition metals are usually larger than those of monovalent metals such as copper, silver, and gold. The cohesive energy for a metal is the difference between average energy of valence electrons and the energy of a completely free electron. The cohesive energy of iron is about 100 kilocalories per gram-atom (4.18×10^5 J/g-at.) and that of copper about 81 kilocalories per gram-atom (3.38×10^5 J/g-at.) (ref. 3).

The clean iron (001) surface prior to adhesive contact was examined with LEED. The diffraction pattern from the clean surface is presented in the photograph of figure 5(a). The origin of the pattern can be seen from an examination of the surface model with unit mesh shown in figure 4 by rotating the pattern 25° in the clockwise direction. The four diffraction spots are characteristic of the (001) surface. The absence of any background, the intensity of the diffraction spots, and the Auger analysis indicate the surface to be clean iron. The intensity of the iron (001) diffraction spots are greater than was observed with the clean iron (011) surface in reference 4. The intensity data of references 5 and 6 indicate a similiar effect.

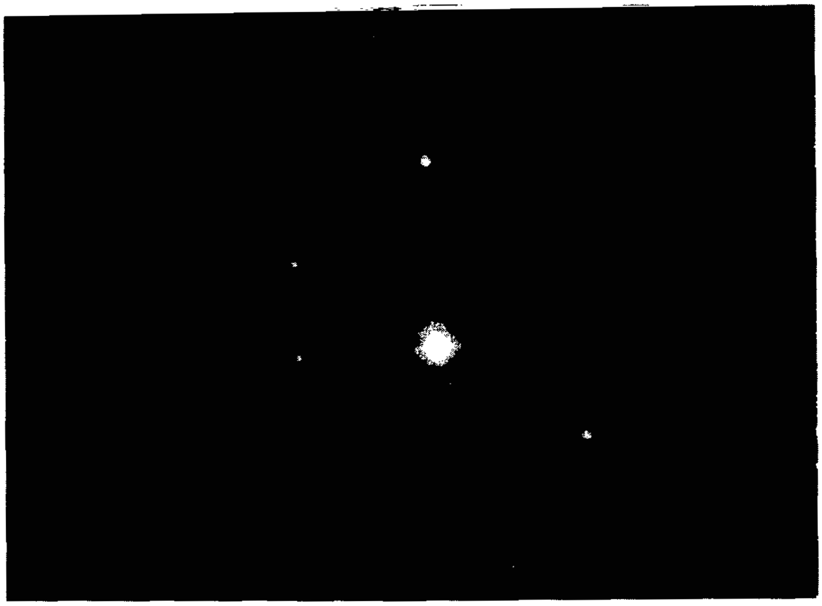


(a) Before adhesion contact.

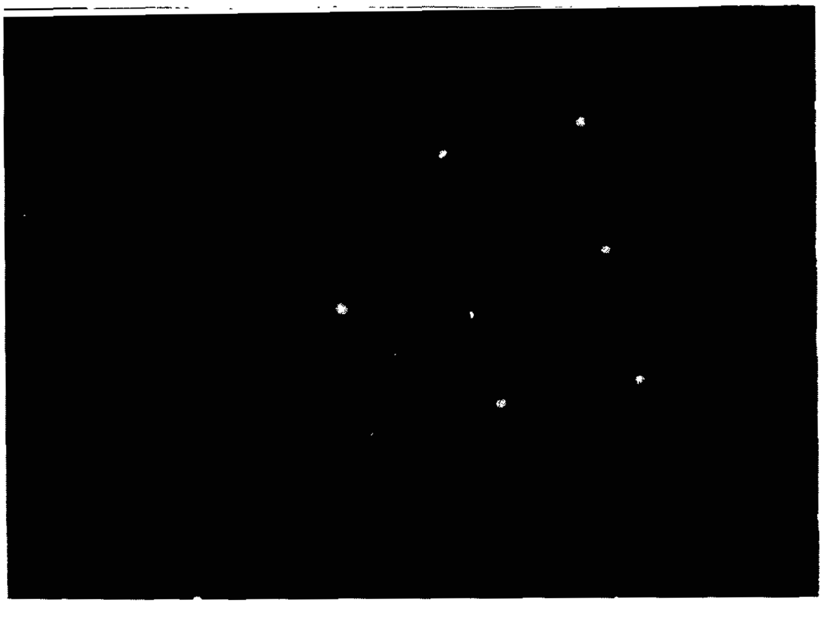


(b) After adhesive contact with itself.

Figure 5. - Photographs of LEED patterns obtained for clean iron (001) surface before and after adhesive contact. The contacting surface was 3.0 millimeter diameter iron flat with negligible contracting load.



(a) Fe(001)C(3x4)-O.



(b) Fe(001)C(2x2)-O.

Figure 6. - Photographs of LEED patterns for oxygen adsorbed to the iron (001) surface.

The (001) surface of iron after adhesive contact is shown in figure 5(b). The diffraction spots have become elliptical in shape. This elliptical shape is interpreted as resulting from strain in the surficial layer as a result of pulling to tensile fracture of the iron cohesive bonds at the interface. The spots have also increased in size, a further indication of electron scatter and strain. This same type of change in the character of the diffraction pattern has been observed with aluminum, nickel, copper, and silver in adhesion experiments (ref. 7).

Oxygen was adsorbed on the iron (001) surface to produce a Fe(001)C(3×4)-O surface structure. The LEED pattern obtained is shown in the photograph of figure 6(a). The suggested arrangement of the oxygen on the iron surface is as shown in the sphere model of figure 7. It indicates the sites occupied by the oxygen on the iron surface.

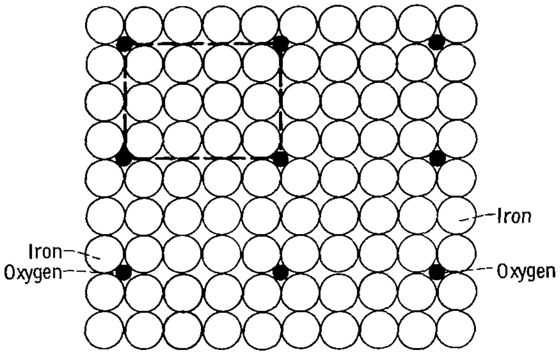


Figure 7. - Suggested arrangement of oxygen adsorbed on iron (001) surface producing Fe(001)C(3x4)-O structure.

Adhesion measurements were made to the surface structure shown in figure 6(a) at various applied loads and the results obtained are presented in figure 8. The first observation to be made is that the adhesive force of iron is appreciably reduced even with a fraction of a monolayer of oxygen present on the surface. The second observation is that the adhesive force appears to have some dependency on applied load at very light loads. At applied loads in excess of 50 dynes (0.05 mN), it was independent of applied force. With a fraction of a monolayer oxygen coverage, bonding at the interface is truly adhesive as well as cohesive. When the two surfaces are brought into contact, there will be sites where oxygen from one surface will bond to iron of the other surface; there will also be sites where oxygen-oxygen interactions can occur as well as iron-iron cohesive bonding. The iron-iron bonding will not be the same cohesive bonding observed for the clean surfaces because the presence of oxygen has reduced the surface energy and will affect interatomic separation.

The addition of more oxygen to the iron (001) surface produces a Fe(001)C(2×2)-O

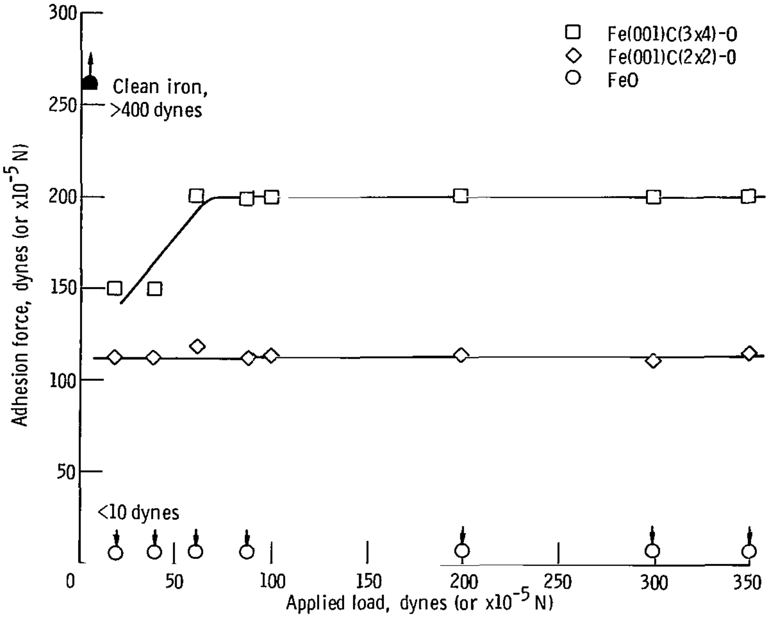


Figure 8. - Adhesion force of iron (001) plane to iron (001) plane at various applied loads with various amounts of oxygen present on iron surfaces. Ambient temperature, 20° C; contact time, 10 seconds.

structure as shown in the LEED pattern of figure 6(b). This particular structure was also observed by the authors of reference 6. In reference 6 the pattern was reported to reach maximum intensity at 140 electron volts and an exposure of 0.8 Langmuir (1×10^{-6} torr-sec), while, in this study, it occurred at 145 electron volts and exposure of 1.0 Langmuir.

The suggested sphere model interpreting figure 6(b) is presented in figure 9. The 2×2 structure represents a quarter monolayer of oxygen coverage on the iron surface.

The adhesion force was measured at various loads for the Fe(001)C(2×2)-O surfaces

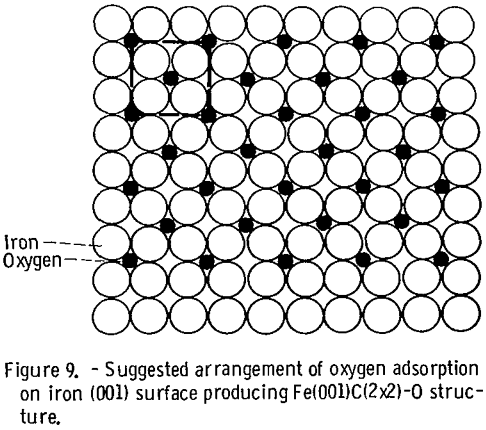


Figure 9. - Suggested arrangement of oxygen adsorption on iron (001) surface producing Fe(001)C(2x2)-O structure.

in contact, and the results obtained are presented in figure 8. The adhesive force was essentially independent of applied load over the range of loads investigated. It did not show, at light loads, the dependency of adhesion on applied load seen with the Fe(001)C(3×4)-O structure. A similar observation was made in reference 4 with hydrogen sulfide gas on the iron (011) surface. The adhesion force was a function of applied load until a full monolayer surface coverage of hydrogen sulfide was achieved. Figure 8 representing experiments conducted in the same load range indicates that the same effect is produced with a quarter of a monolayer of oxygen.

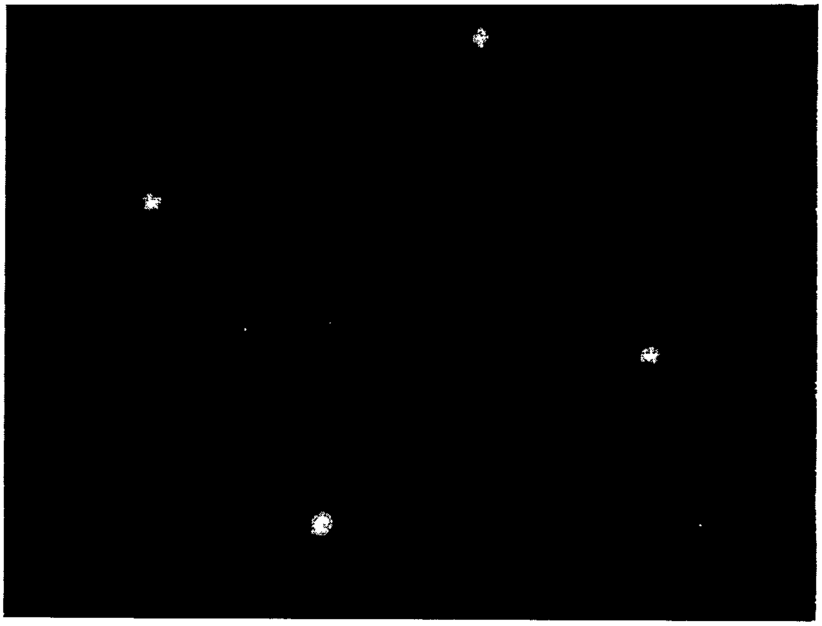
Sufficient oxygen was admitted to the system to produce a continuous weakening of the 2×2 structure. Eventually no pattern could be detected. The surface was heated to 500° C, and the iron oxide structure of figure 10(a) resulted. The intensity maxima were observed at 90 and 165 volts. FeO rather than Fe_3O_4 was also observed to form in the studies of reference 6. Bulk oxidation studies indicate that FeO would not be stable below 575° C in vacuum and that the oxide Fe_3O_4 would form (ref. 7). The formation of the oxide in figure 10(a) required an oxygen exposure of seven Langmuirs. These results coupled with those of reference 8 warn against the use of bulk behavior in attempts to explain surface phenomenon.

Figure 11 is an Auger analysis of the iron oxide (FeO) surface. The peaks detected were those of oxygen and iron. The pattern of figure 11 does not show the presence of sulfur and carbon observed in figure 3(a) with the iron surface simply heated in vacuum without any cleaning prior to oxidation. The results of figures 3(a) and 11 indicate that if oxide only is desired on an iron surface it may be necessary to clean the iron surface and then oxidize. The film formed on the iron surface in air at atmospheric pressure may contain other elements, as shown in figure 3(a).

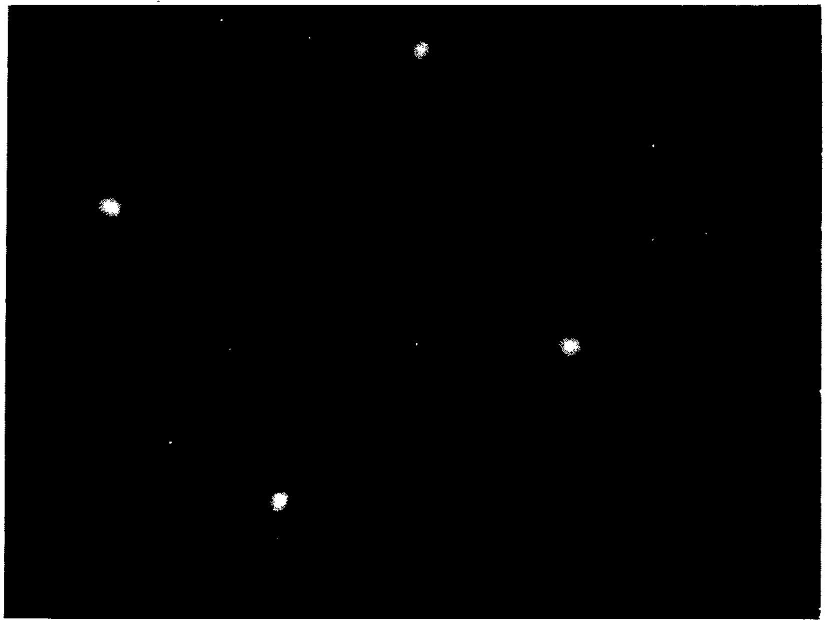
Adhesion experiments were conducted on the FeO surface. LEED patterns were obtained in the contact zone. The photograph of one such pattern is presented in figure 10(b). This pattern was obtained after adhesive loading to 350 dynes (3. 5 mN). While strain was evidenced in figure 5(b) after adhesive contact under negligible load, no such strain was observed in figure 10(b) with iron oxide (FeO).

The adhesive forces measured at various loads were less than 10 dynes (0. 1 mN) at loads to 350 dynes (3. 5 N) as shown in figure 7. Thus, as might be anticipated, as the oxygen concentration on the surface increases the adhesive force at any given load decreases (fig. 8).

Adhesion experiments were also conducted with the (011) surface of iron after various exposures to oxygen. The experimental results obtained are presented in figure 12. Three oxygen structures were observed, a Fe(011)C(2×4)-O, Fe(011)C(2×2)-O, and a Fe(011)C(1×2)-O structure. With all three structures, a dependency of adhesion on applied load was observed. This should be contrasted with the data of figure 8 where, with a (2×2) structure, adhesion was independent of applied load when examined in the same load range.



(a) Before adhesion contact.



(b) After adhesive contact with itself.

Figure 10. - Photographs of LEED patterns obtained for iron oxide (FeO) on an iron surface (001) before and after adhesive contact with itself.

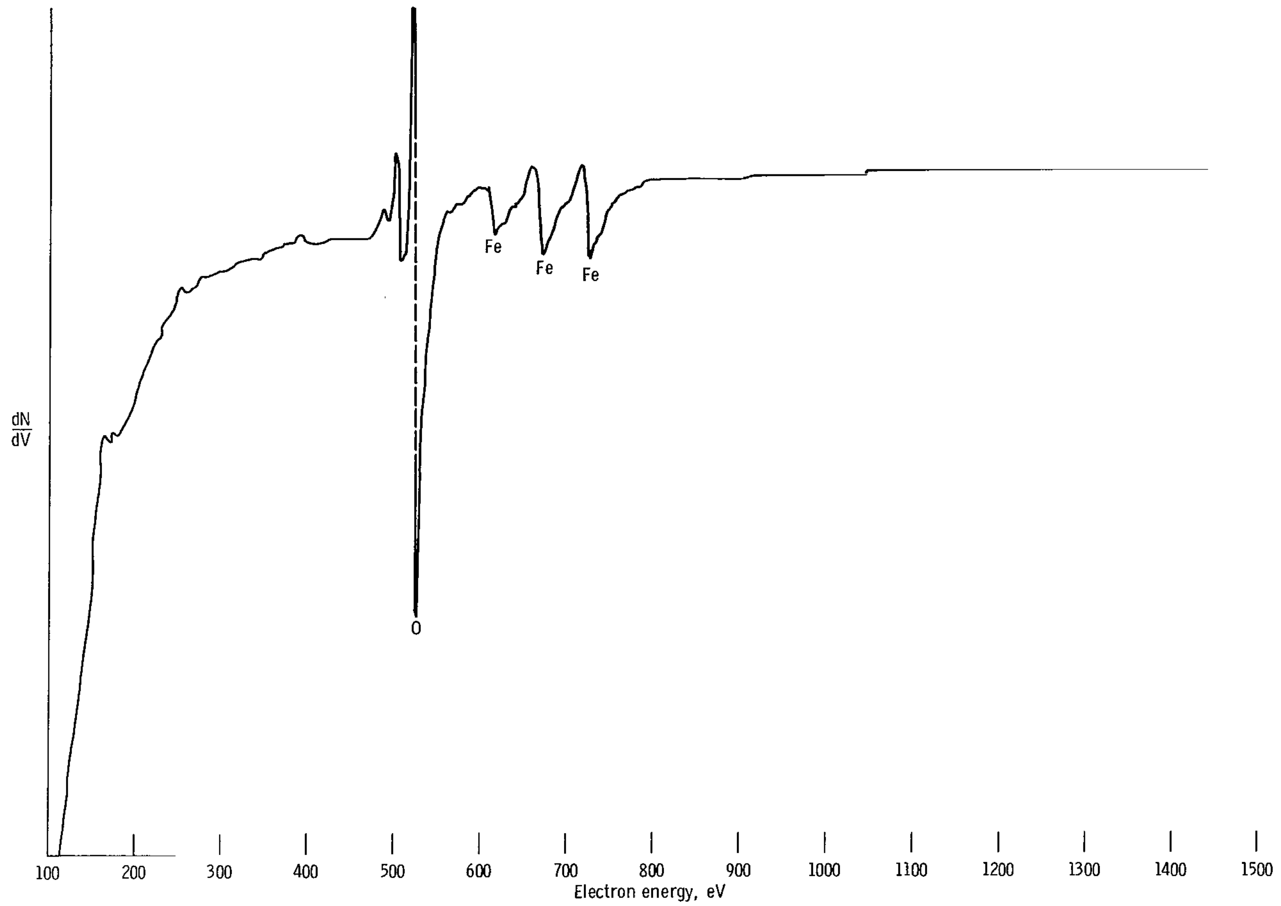


Figure 11. - Auger emission spectrometer analysis of iron oxide (FeO).

The difference in adhesion behavior on the two iron surfaces (001) and (011) with increasing load and a (2×2) oxygen structure cannot be related to differences in deformation behavior because the Young's modulus for the (001) surface is 13.2×10^{11} dynes/cm^2 (13.2×10^{6} N/cm^2) while that for the (011) surface is 22.2×10^{11} dynes/cm^2 (22.2×10^{6} N/cm^2) (ref. 9). While the adhesive force for the (011) surface is less at the lower loads (<200 dynes (2.0 mN)), at 200 dynes and higher the adhesive force is greater. The surface energy of the (001) surface is less than that for the (011) surface (ref. 9). The difference in adhesion behavior of the two surfaces may be related to the difference in surface energy at the higher loads. It would be of interest to know what effect a (2×2) oxygen structure has on the surface energy of the two iron surfaces. Unfortunately, such data are not available. The difference in adhesive behavior at the light loads may be due to greater resistance of the (011) surface to deformation thereby minimizing the amount of iron cohesive bonding through the quarter monolayer oxygen surface film. At higher loads, however, where metal cohesive bonding is more likely the effects of the surface

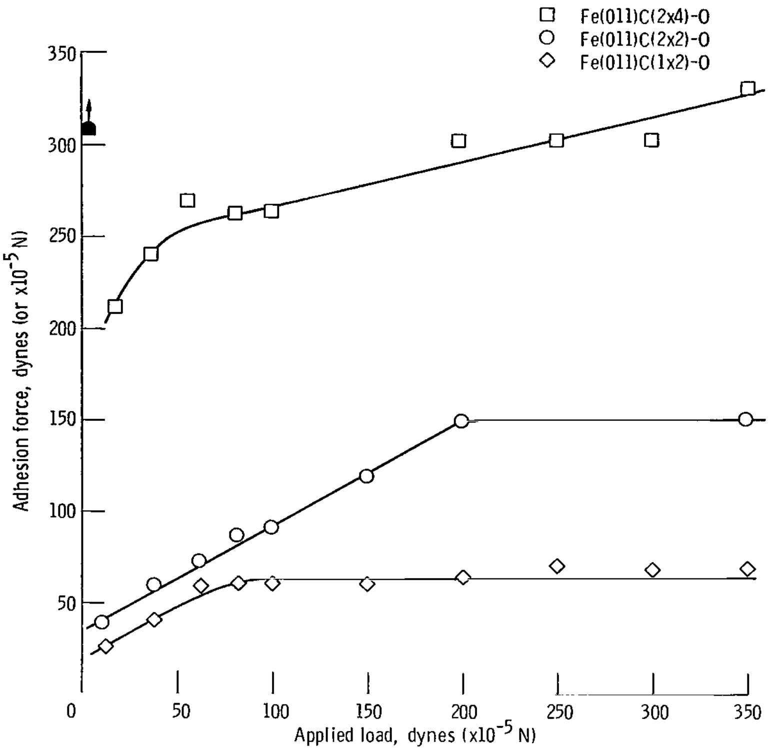


Figure 12. - Adhesion force of iron (011) to iron (011) in presence of various fractions of oxygen monolayer coverage. Contact time, 10 seconds; temperature, 20° C.

energy differences between the (001) and (011) surfaces will play a more important role in determining measured adhesion forces.

In reference 2 it is reported that (2×4) sulfur and hydrogen sulfide structures afforded better resistance to adhesion than did a (2×4) oxygen structure on an iron (011) surface. If, however, the (1×2) structure adhesive data of figure 11 are compared with adhesion data obtained in reference 3 for a (1×2) hydrogen sulfide structure, the adhesion forces measured to the two surfaces are nearly the same at loads from 100 to 300 dynes (1.0 to 3.0 mN). These results indicate that the relative effectiveness of equivalent surface coverages of two different gases on iron may be related to the percentage of surface coverage.

SUMMARY OF RESULTS

Based on the adhesion experiments conducted in this study with the (001) and (011) surfaces of iron in contact with themselves and the influence of oxygen on that adhesion, the following summary remarks are made:

1. The adsorption of oxygen on iron surfaces in coverages of less than a monolayer are sufficient to appreciably reduce the adhesion of iron to itself.

2. Adhesion decreased as the amount of oxygen surface coverage increased. Minimal adhesion was observed on an iron oxide (FeO) structure developed on the (001) surface of iron.

3. While lattice strain occurred to the clean iron surface (001) with the fracture of cohesive iron junctions equivalent experiments with iron oxide (FeO) revealed no evidence of surface strain.

4. A comparison of the effect of oxygen surface coverage in this study with equivalent surface coverages of hydrogen sulfide in an earlier investigation indicate that both the percentage of surface coverage and the composition of the chemisorbed species are important in reducing adhesion.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, January 30, 1970,
129-03.

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