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ADHESION AND FRICTION OF IRON AND GOLD IN CONTACT WITH ELEMENTAL SEMICONDUCTORS

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16. Abstract

Adhesion and friction experiments were conducted with single crystals of iron and gold in contact with single crystals of germanium and silicon. Surfaces were examined in the sputter-cleaned state and in the presence of oxygen and a lubricant. All experiments were conducted at room temperature with loads of 1 to 50 grams, and sliding friction was at a sliding velocity of 0.7 mm/min. Results indicate that the friction nature of metals in contact with semiconductors is sensitive to orientation, that strong adhesion of metals to both germanium and silicon occurs, and that friction is lower with silicon than with germanium for the same orientation. Surface effects are highly sensitive to environment. Silicon, for example, behaves in an entirely brittle manner in the clean state, but in the presence of a lubricant the surface deforms plastically.

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SUMMARY

An experimental program was conducted to determine the adhesion and friction behavior of the metals iron and gold in contact with the semiconductors germanium and silicon. Both metals and semiconductors were in single-crystal form. Experiments were conducted with the surfaces sputter cleaned in a vacuum of 10^{-8} N/m^2 (10^{-10} torr), in oxygen at atmospheric pressure, and in the presence of a lubricant (0.2-percent oleic acid in mineral oil). The sliding velocity in friction experiments was 0.7 mm/min, the loading on the specimens was from 1 to 50 grams, and the temperature in all experiments was 23° C.

The results of this investigation indicate that the friction of metals in contact with semiconductors is sensitive to orientation. Friction was lower on the (111) than on the (100) plane of the silicon semiconductor. Strong adhesion was observed for both iron and gold in contact with germanium and silicon. Lower friction was measured on the (111) surface of silicon than on the (111) surface of germanium. The effect of metal-semiconductor contact on the semiconductor surface is highly sensitive to environment. Brittle fracture of the silicon surface was observed with dry sliding on a clean surface; in the presence of the lubricant the surface of the silicon deformed in an entirely plastic manner.

INTRODUCTION

The semiconductors silicon and germanium are widely used in the electronics industry and yet very little is known about the adhesion and friction of these materials in contact with metals. Strong adhesion of noble metal wires to silicon and germanium has been observed in connecting electrical leads to these semiconductors (ref. 1). Adhesion has also been observed in the vapor deposition of metallic films of gold, aluminum, and nickel on silicon surfaces (ref. 2). More information is needed on the adhesion and more particularly the friction of metals in contact with silicon and germanium as they are affected by such things as orientation, doping, and surface films.

This investigation was conducted to examine the adhesion and friction behavior of two metals, gold and iron, in contact with silicon and germanium. All materials were in single-crystal form. The friction behavior of silicon was compared with that of germanium. Orientation and doping of silicon were examined relative to their effect on metal-semiconductor friction. All experiments were conducted with light loads of 1 to 50 grams, and the sliding velocity for friction measurements was 0.7 mm/min. The surfaces were examined in the sputter-clean state and with contaminant films present. Studies were all at room temperature.

MATERIALS

The gold single-crystal pin specimen used in this investigation was 99.999-percent gold, and the iron was 99.99-percent iron. The gold pin was oriented with the (111) plane parallel to the contacting interface and the iron with the (110) plane parallel to the interface.

Undoped germanium crystals of the (111) orientation were examined, as well as undoped silicon crystals of both the (111) and (100) orientations. Doped single crystals of silicon were also examined. These were of both the P and N types. The P type were doped with boron and the N type with phosphorous. The resistivity of the P type was from 1 to 10 ohm-cm, and that of the N type was from 3 to 15 ohm-cm.

APPARATUS

The apparatus used in this investigation was a vacuum system capable of measuring adhesion, load, and friction and capable of Auger and low-energy electron diffraction (LEED) surface analyses. The mechanism for measuring adhesion, loading, and friction is shown schematically in figure 1.

A gimbal-mounted beam projected into the vacuum system. The beam contained two flats machined normal to each other with strain gages mounted thereon. The end of the rod contained the iron or gold metal single-crystal pin specimen. The load applied by moving the beam toward the disk was measured by the strain gage. The adhesion force, that is, the force necessary to separate the pin and the disk after they are loaded together, was measured in the direction opposite to that of load application by the same strain gages.

Tangential motion of the pin along the disk surface was accomplished through the gimbal assembly. Under an applied load the friction force was sensed by the strain gage normal to that used to measure load. Multiple wear tracks could be generated on the disk specimen surface by the translational motion of the disk or pin. Pin sliding was in the vertical direction of figure 1.

The vacuum apparatus in which the components of figure 1 were contained also had a LEED diffraction system and an Auger spectrometer. The electron beam of both could be focused on any disk site by manipulating the disk.

The vacuum system was a conventional vacsorb and ion-pumped system capable of readily achieving pressures of 1×10^{-8} N/m^2 (10^{-10} torr) as measured by a nude ionization gage within the specimen chamber. Sublimation pumping was also used to more rapidly achieve the desired pressure.

EXPERIMENTAL PROCEDURE

The metal and semiconductor single-crystal surfaces were mechanically polished on metallurgical papers down to 600 grit. The crystals were then electropolished. The surfaces were rinsed with water and 200-proof ethyl alcohol. The specimens were placed in the vacuum chamber and the system was evacuated. It was baked out overnight at 250° C, after which the pressure was in the 10^{-8} N/m^2 (10^{-10} torr) range.

Argon gas was bled into the vacuum system to a pressure of 10^{-1} N/m^2 (10^{-3} torr). A 1000-volt direct-current potential was applied to the specimen and it was sputter bombarded for a period of 30 minutes. After sputtering the specimen surface was examined with Auger emission spectroscopy to establish its cleanliness. Where the surface was not clean, the sputtering process was repeated. Since the silicon and germanium were semiconductors, they were placed in a tantalum harness to achieve surface sputter cleaning. Both the metals (iron and gold) and the semiconductors (silicon and germanium) were sputter cleaned. All friction experiments were conducted with the system reevacuated to a pressure of 10^{-8} N/m^2 (10^{-10} torr).

The metal pin was loaded against the semiconductor flat by mechanically deflecting the beam through a gimbal assembly with the pin in contact with the flat. When the proper load, as indicated on a strip-chart recorder, had been applied, sliding was begun by starting a drive motor that moved the beam in a vertical direction parallel to the disk surface. The friction force was continuously recorded during sliding.

Auger emission spectroscopy analysis of the germanium (111) surface before sputtering revealed the presence of carbon and oxygen surface contaminants. The contaminating layer is thick enough (4 to 5 layers) that germanium peaks are not detected (fig. 2(a)). After this same germanium surface was sputter cleaned by argon bombardment for 30 minutes, the Auger spectrum of figure 2(b) was obtained. In this spectrum, germanium peaks are seen and carbon and oxygen surface contaminants are absent. Gold single-crystal pins were also sputter cleaned and subjected to adhesion experiments.

RESULTS AND DISCUSSION

Adhesion and Friction with Germanium

Photomicrographs of the germanium surface after adhesion contact with gold under a load of 30 grams are shown in figure 3. Adhesion of gold to the germanium was very strong and, when the two surfaces were separated, fracture had occurred in the germanium. In the top photomicrograph of figure 3 the area on the germanium that was contacted by the gold can be seen. To the right of the contact, slip lines appear, indicating plastic deformation in the germanium. Increasing the magnification (lower photomicrograph of fig. 3) clarifies changes in surface features in the contact zone. An X-ray energy-dispersed analysis of this region showed an absence of gold and the presence of only the element germanium. Thus, with adhesive contact the interfacial bonds formed between gold and germanium were stronger than the cohesive bonds in the germanium and fracture occurred in the germanium when the materials were pulled in tension.

The literature on the cohesive binding energies of gold and germanium states that these two materials have binding energies that are very close to one another (ref. 3). It might, therefore, be anticipated that fracture could occur in either material where the interfacial adhesion is stronger than the cohesive binding in the elements.

Adhesion experiments were also conducted with iron. Again, fracture was observed in the germanium.

Sliding friction experiments were conducted with both iron and gold crystals in contact with the germanium (111) surface. The friction traces were characterized by marked stick-slip behavior, as shown in figure 4. This type of friction is anticipated where strong adhesion occurs at the interface. The increase in stick and friction in the trace represents the force required to tangentially overcome the interfacial adhesive bond. The slip reflects fracture of the adhesive bond.

Friction experiments were conducted at various loads for iron in contact with the (111) surface of germanium both sputter cleaned in vacuum (10^{-8} N/m^2 (10^{-10} torr)) and in oxygen at atmospheric pressure. The results obtained are presented in figure 5. At a 1-gram load on clean surfaces a friction coefficient of 5 was obtained. With increases in the applied load the friction coefficient decreased. This behavior has been observed elsewhere (ref. 4). With loads in excess of 10 grams the friction did not change markedly.

In the presence of oxygen the friction coefficient in figure 5 was less at all loads than was obtained with the clean surfaces. Above 10 grams there was not a marked difference in the friction coefficient for the iron-germanium couple either clean or in oxygen. This is not too surprising since strong adhesion has been observed for metals in contact with germanium even in the presence of surface contaminants (ref. 1).

Detailed examination of the wear track on the germanium surface after sliding with iron revealed evidence of both plastic deformation and fracture in the germanium. Etch pitting was used as a method to detect plastic deformation. Figure 6 is a photomicrograph of a germanium wear track that was etch pitted to show dislocations. The dislocations have been generated with sliding. Surface cracks appear to emerge from the bands of dislocations. The fracture cracks originate at the dislocations, as shown by the arrows. A cluster of dislocation etch pits appears on the right of the photomicrograph around a particle of debris that apparently had been trapped at the interface during sliding.

Figure 6 suggests that, with sliding, frictional energy is initially dissipated by the plastic deformation of the germanium. At some point, however, the force to fracture must be less than that for plastic strain, and fracture cracks begin to appear. This is not uncommon. In fact, with iron and steel, there are three prerequisites for the formation of cleavage microcracks: (1) plastic deformation, (2) crack initiation, and (3) crack propagation (ref. 5). All three are evident on the germanium surface in figure 6.

Adhesion and Friction with Silicon

Silicon has many properties that are very similar to germanium properties. It does, however, differ from germanium in certain properties important in adhesion and friction. For example, it has a higher modulus of elasticity (ref. 3), higher cohesive binding energy (ref. 2), and higher surface energy (ref. 6). Adhesion and friction experiments were, therefore, also conducted with iron and gold contacting silicon.

Auger emission spectroscopy analysis of the silicon surface reveals that it contains the same surface contaminants as germanium, namely, carbon and oxygen (fig. 7(a)). When the surface is argon sputter cleaned, the silicon Auger peak appears, the oxygen disappears, and the carbon all but disappears (fig. 7(b)).

Adhesive contacts were made by bringing clean gold and iron in contact with a clean silicon (111) surface and measuring adhesive bonding. Both gold and iron were found to adhere to clean silicon. Photomicrographs (e.g., fig. 8) revealed that gold adhered to silicon, and an X-ray energy dispersive analysis for gold on the silicon surface indicated its presence. In contrast with germanium, copious amounts of gold remained on the silicon surface after the adhered junction was fractured in tension. Silicon has a considerably higher cohesive binding energy than does germanium, and this may account for the transfer of gold to silicon.

Comparison of Results with Both Semiconductors

Friction experiments were conducted with gold sliding on the (111) surfaces of both germanium and silicon to determine if friction behavior differed with the two semiconductors. Friction coefficients measured at various loads are presented in figure 9.

The friction coefficient for gold sliding on germanium was higher at all loads than for gold sliding on silicon (fig. 9). The friction differences are significant. Further, the friction coefficient for gold in sliding contact with germanium was considerably higher than for iron in sliding contact with germanium (fig. 5).

With iron sliding on silicon, fracture cracks formed in the silicon surface just as with iron sliding on germanium. Unlike the germanium results, very little evidence for plastic deformation was observed as indicated by the top photomicrograph in figure 10. Close examination of the cracks (bottom photomicrograph in fig. 10) shows no plastic behavior of the silicon with sliding. The friction coefficient for iron in sliding contact with silicon was measured in vacuum, in oxygen, and with an oil containing oleic acid. Results are presented in figure 11. The vacuum results are higher than for iron sliding on germanium (fig. 5). Comparing the vacuum results for clean surfaces in figure 5 and figure 11 suggests that cohesive fracture occurs more readily in silicon than in germanium, which is consistent with their basic properties. Even in the presence of oxygen, the friction coefficient for iron sliding on silicon was twice that for iron sliding on germanium in figure 5. With oxygen present, fracture cracks were observed in the silicon surface just as for the clean surface in figure 10.

Effect of Lubrication

Adhesion plays a role in silicon undergoing brittle fracture, so lubricating the surface to reduce adhesion should reduce crack formation in the silicon. Friction experiments were, therefore, conducted with the silicon surface lubricated with 0.2-percent oleic acid in mineral oil. The friction coefficients measured for the lubricated iron-silicon contacts at various loads are presented in figure 11.

The friction coefficient for the lubricated surface was relatively unaffected by load. Sliding was extremely smooth, with no evidence of stick-slip behavior. Further, examination of the silicon surface revealed a complete absence of fracture cracks. Etch pitting of the surface disclosed that a band of dislocations was generated in the sliding contact region. These are shown in the photomicrograph of figure 12 by a series of delta-shaped etch pits. Slip bands also appear to the right of the etch pits.

The plastic deformation of silicon (fig. 12) and germanium (fig. 6) occurred in experiments conducted at room temperature. Both silicon and germanium are brittle at room temperature, and deformation experiments on these materials are usually conducted at elevated temperatures (refs. 7 to 9). This is true even in the easy-slip stage (ref. 10). Further, with silicon, bond rupture occurs more easily than bond shear at room temperature (ref. 11). The dislocations generated herein with sliding at room temperature are, therefore, unusual.

Examination of the entire specimen surface revealed a complete absence of fracture cracks, indicating entirely plastic behavior of the silicon. The only other occurrence we could find of plastic deformation behavior of these materials at room temperature was in abrasion studies of silicon (ref. 12). In the abrasion studies, damage varied with orientation. In some instances, only dislocations were generated; in other instances, chips and cracks formed in addition to dislocations.

Effect of Orientation

Since semiconductors exhibit orientation-sensitive behavior in abrasion, it is reasonable to anticipate that similar behavior would be exhibited by these materials in sliding contact with metals. Friction experiments were thus conducted with gold sliding on the silicon (100) surface. The results were compared with those for gold in contact with the silicon (111) surface. The friction behavior of silicon is sensitive to orientation as indicated by the data of figure 13. A marked difference in friction coefficients exists for the two orientations. Thus, the semiconductor silicon exhibits orientation-sensitive friction behavior like other crystalline solids (ref. 13).

In addition to differences in friction behavior for the two orientations of silicon, differences also existed in the nature of the wear surfaces, as shown in figure 14. The debris present in the wear track region in the top photomicrograph and about the surface cracks in the bottom photomicrograph were not iron particles. Neither Auger spectroscopy analysis nor X-ray energy dispersion analysis indicated the presence of iron on the surface.

Effect of Doping

Doping of semiconductors with other elements such as boron and phosphorous can produce either N- or P-type conducting materials. To determine if doping affects friction behavior, sliding friction experiments were conducted with both N- and P-type silicon (111) surfaces in sliding contact with iron. The results are presented in figure 15. The friction coefficient was higher with N-type than with P-type silicon.

The doping itself places two different contaminants in the silicon, boron in the P type and phosphorus in the N type. These elements could account for friction differences. Another possible explanation for the differences in the surface changes produced on contact with metals is a negative surface charge on the N-type semiconductor and a positive surface charge on the P-type semiconductor. More definitive experiments are needed to identify the source of these differences.

CONCLUSIONS

Based on the adhesion and friction experiments conducted in this investigation with single crystals of iron and gold in sliding contact with single crystals of silicon and germanium, the following conclusions were made:

1. The friction characteristics of metals in contact with semiconductors are sensitive to orientation. With gold sliding on silicon (111) and (100) surfaces, friction as well as surface damage was markedly different with the two orientations.

2. The friction coefficients for gold-silicon contacts are lower than for gold-germanium contacts. The friction coefficient was three times greater for gold sliding on the germanium (111) surface than for gold sliding on the silicon (111) surface.

3. With silicon, friction was higher with the N- than with the P-type crystal.

4. Both silicon and germanium deformed plastically upon sliding contact with metals. With an oil lubricant, the silicon surface behaved in an entirely plastic manner. In the absence of lubricating surface films, brittle fracture alone was observed on the silicon surface as a result of sliding.

5. The metals became strongly bonded to the semiconductor surfaces. Fracture of the adhesive junction removed material from the germanium surface. However, gold adhered to the silicon surface.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 27, 1976,
506-16.

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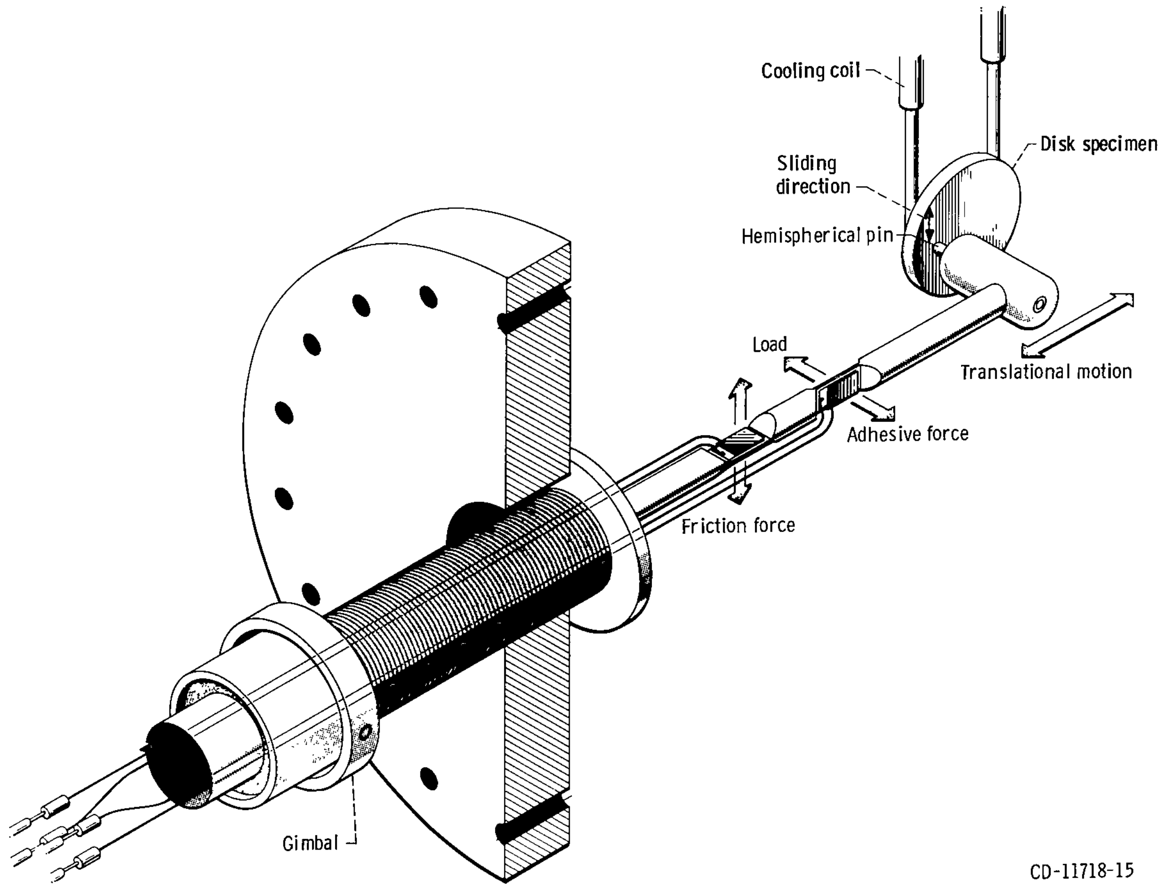
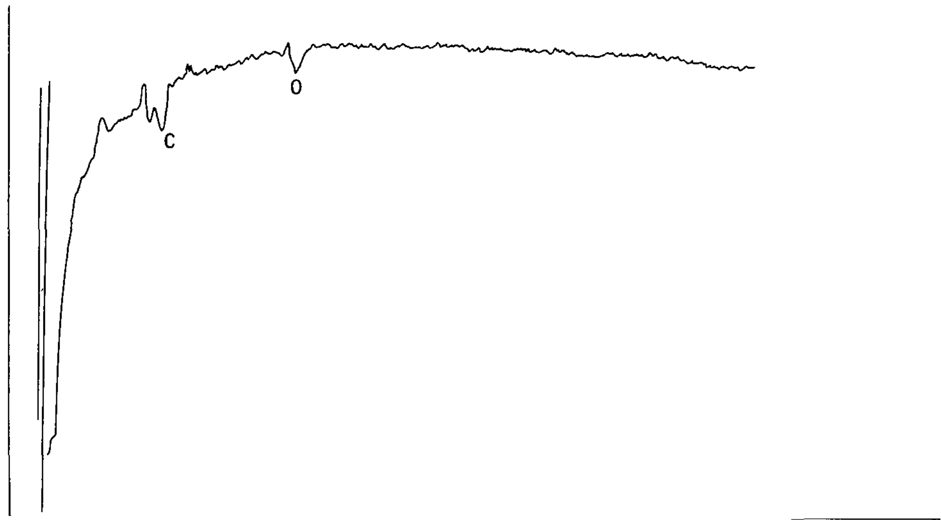
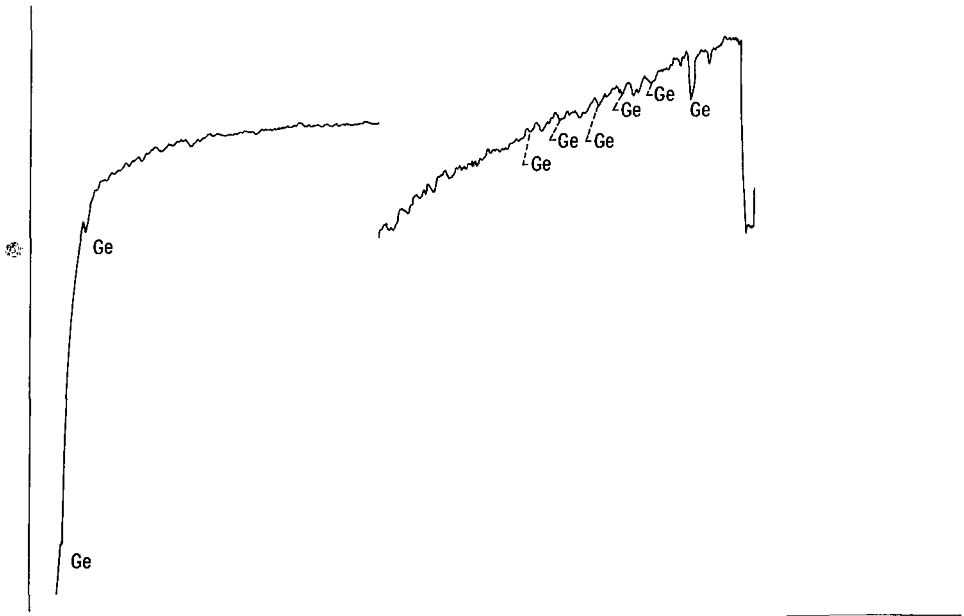


Figure 1. - High-vacuum friction and wear apparatus.



(a) Before sputter cleaning.



(b) After sputter cleaning.

Figure 2. - Auger emission spectroscopy spectra for a germanium single-crystal surface.

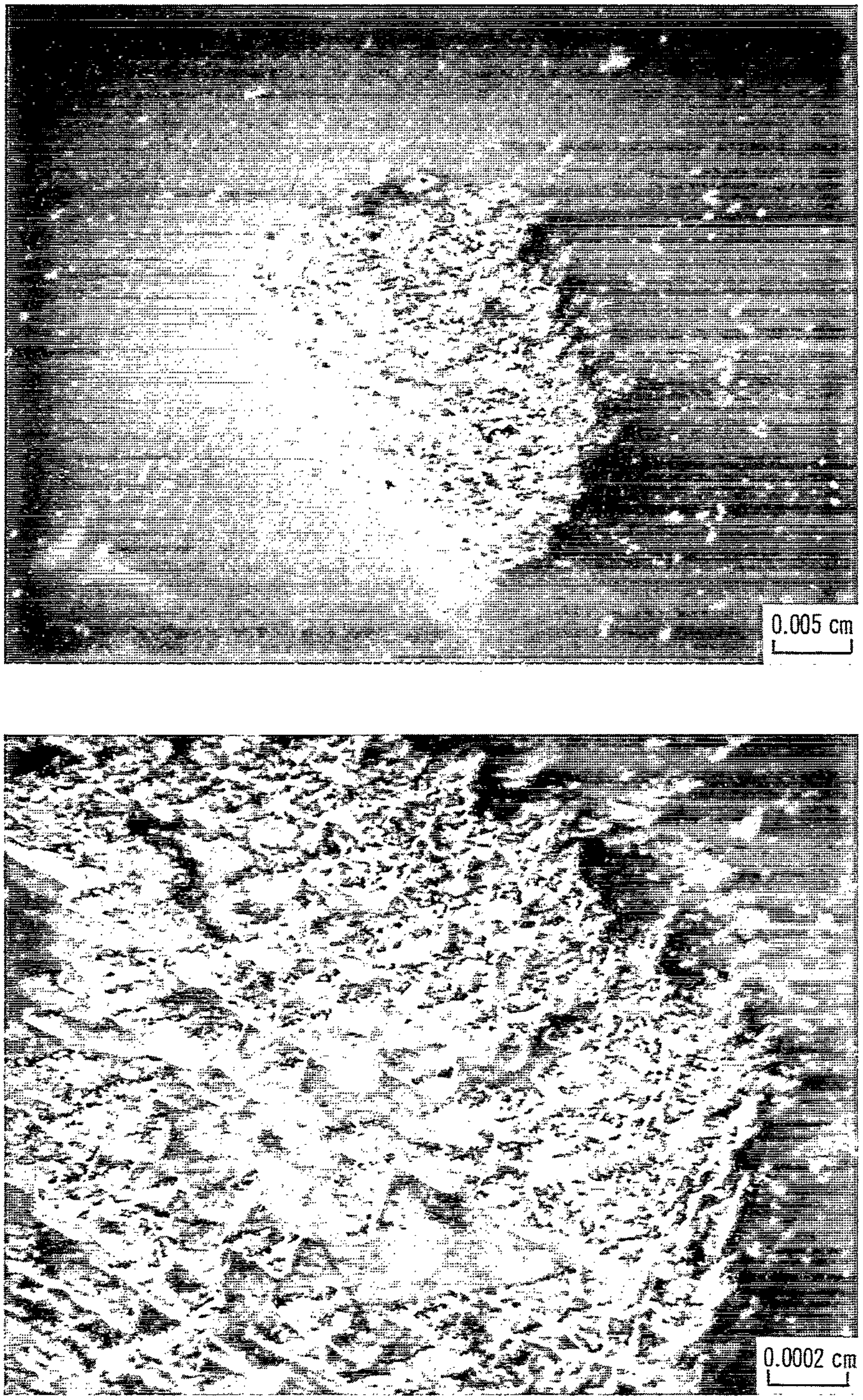


Figure 3. - Adhesion of gold (111) surface to germanium (111) surface. Load, 30 grams; temperature, 23° C; pressure, 10^{-8} N/m^2. (Fracture occurs in germanium.)

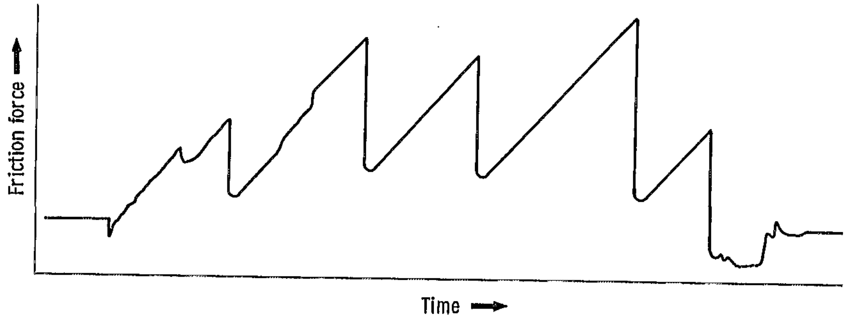


Figure 4. - Friction trace for single-crystal iron (110) sliding on a germanium (111) single-crystal surface. Sliding velocity, 0.7 mm/min; load, 30 grams; temperature, 23° C; pressure, 10^{-8} N/m^2.

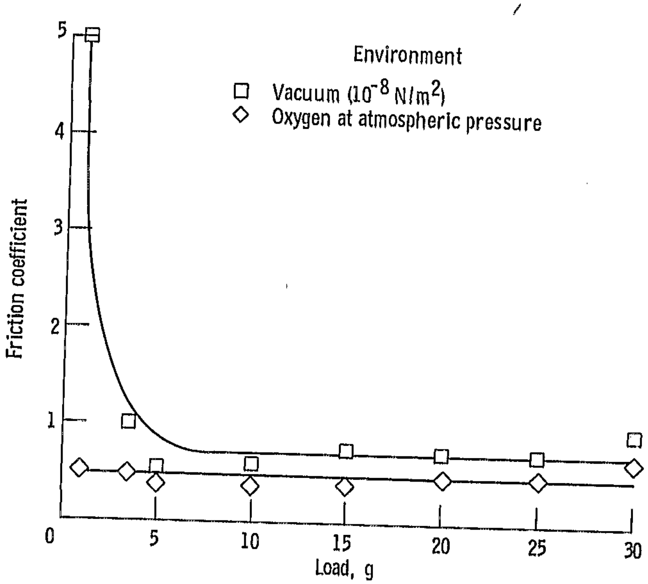


Figure 5. - Friction coefficient as function of load for single-crystal iron (110) sliding on single-crystal germanium (111) surface in vacuum and in oxygen. Sliding velocity, 0.7 mm/min; temperature, 23° C.

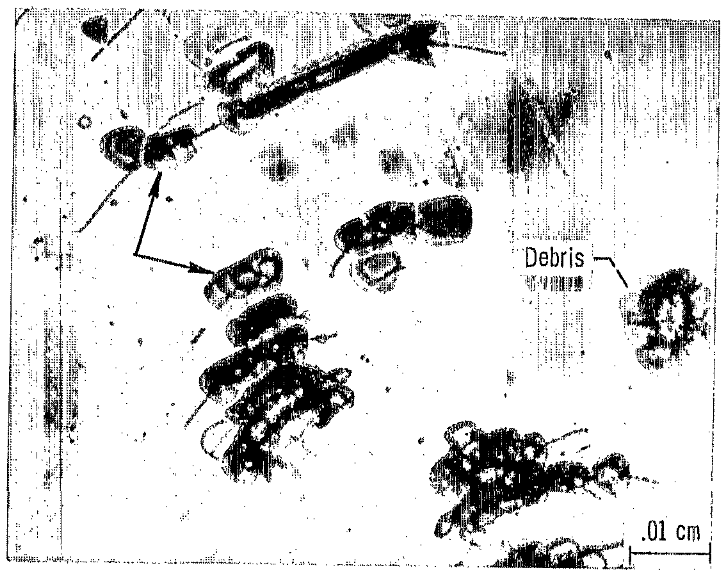
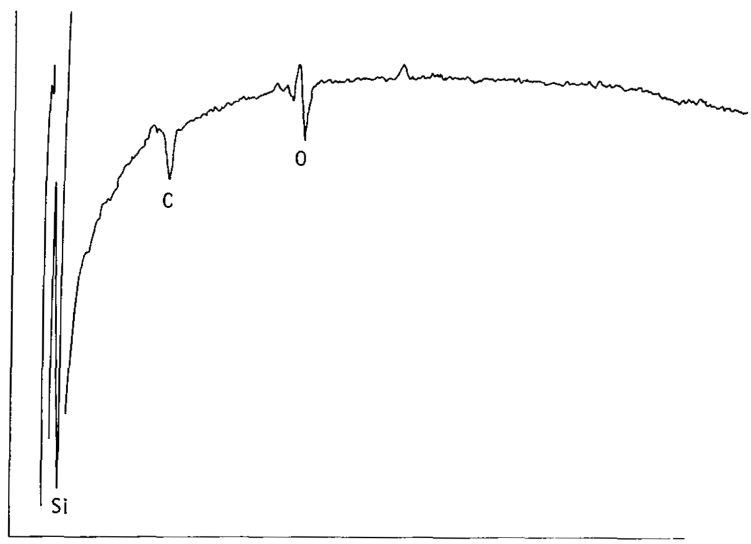
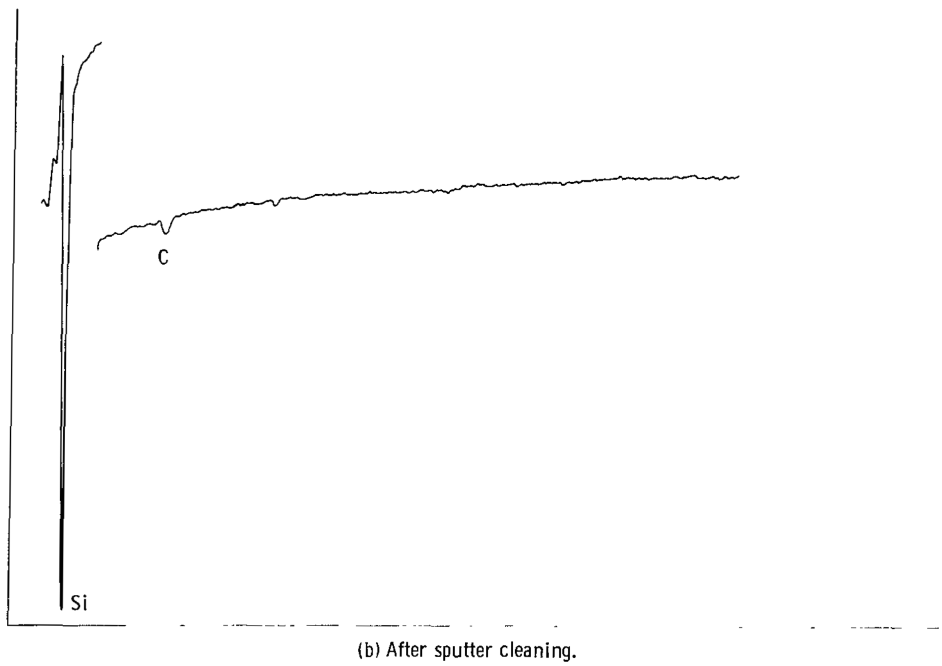


Figure 6. - Etch-pitted wear track made by single-crystal iron (110) sliding across germanium (111) surface. Sliding velocity, 0.7 mm/min.; load, 30 grams; temperature, 23° C; pressure, 10^{-8} N/m^2.



(a) Before sputter cleaning.



(b) After sputter cleaning.

Figure 7. - Auger emission spectroscopy spectra for a silicon single-crystal surface.

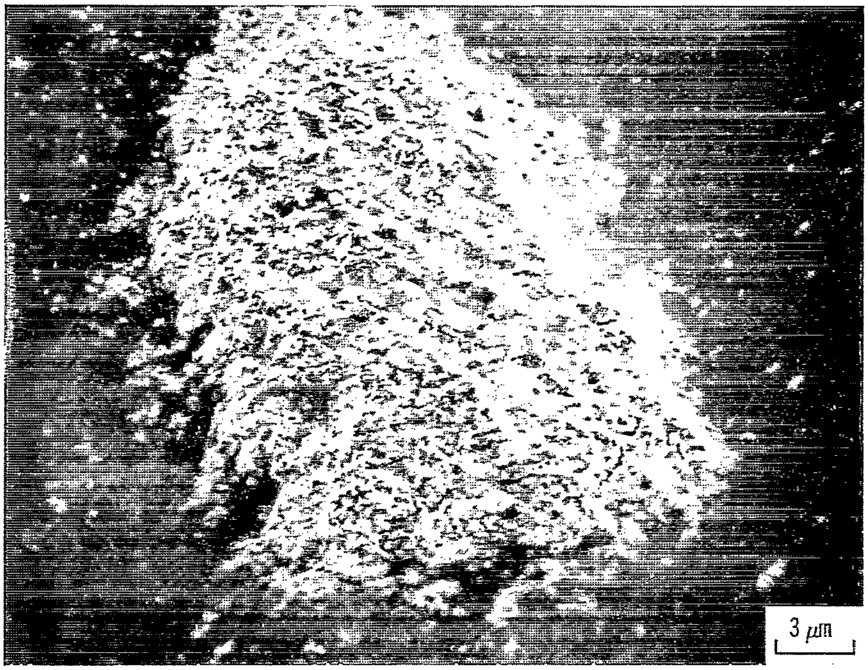


Figure 8. - Gold transferred to a clean silicon (111) surface after adhesive contact. Load, 30 grams; temperature, 23° C; pressure, 10^{-8} N/m^2.

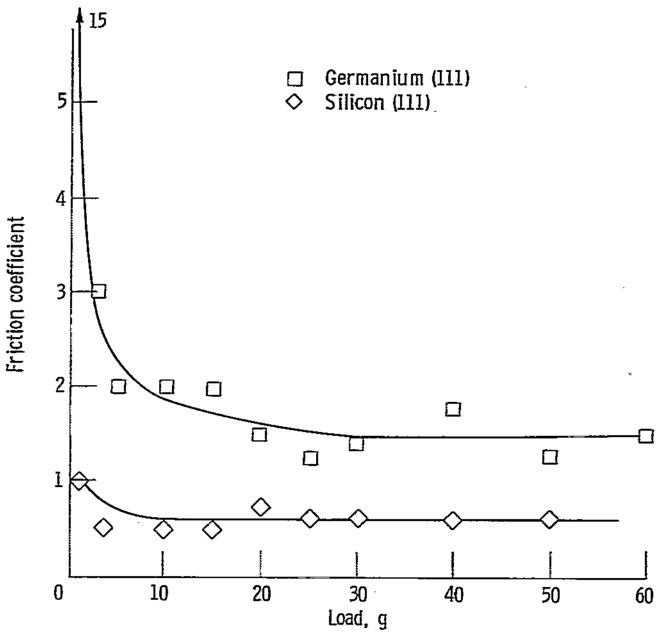


Figure 9. - Friction coefficient as function of load for single-crystal gold (111) sliding on single-crystal germanium and silicon (111) surfaces in vacuum (10^{-8} N/m^2). Sliding velocity, 0.7 mm/min; temperature, 23° C.

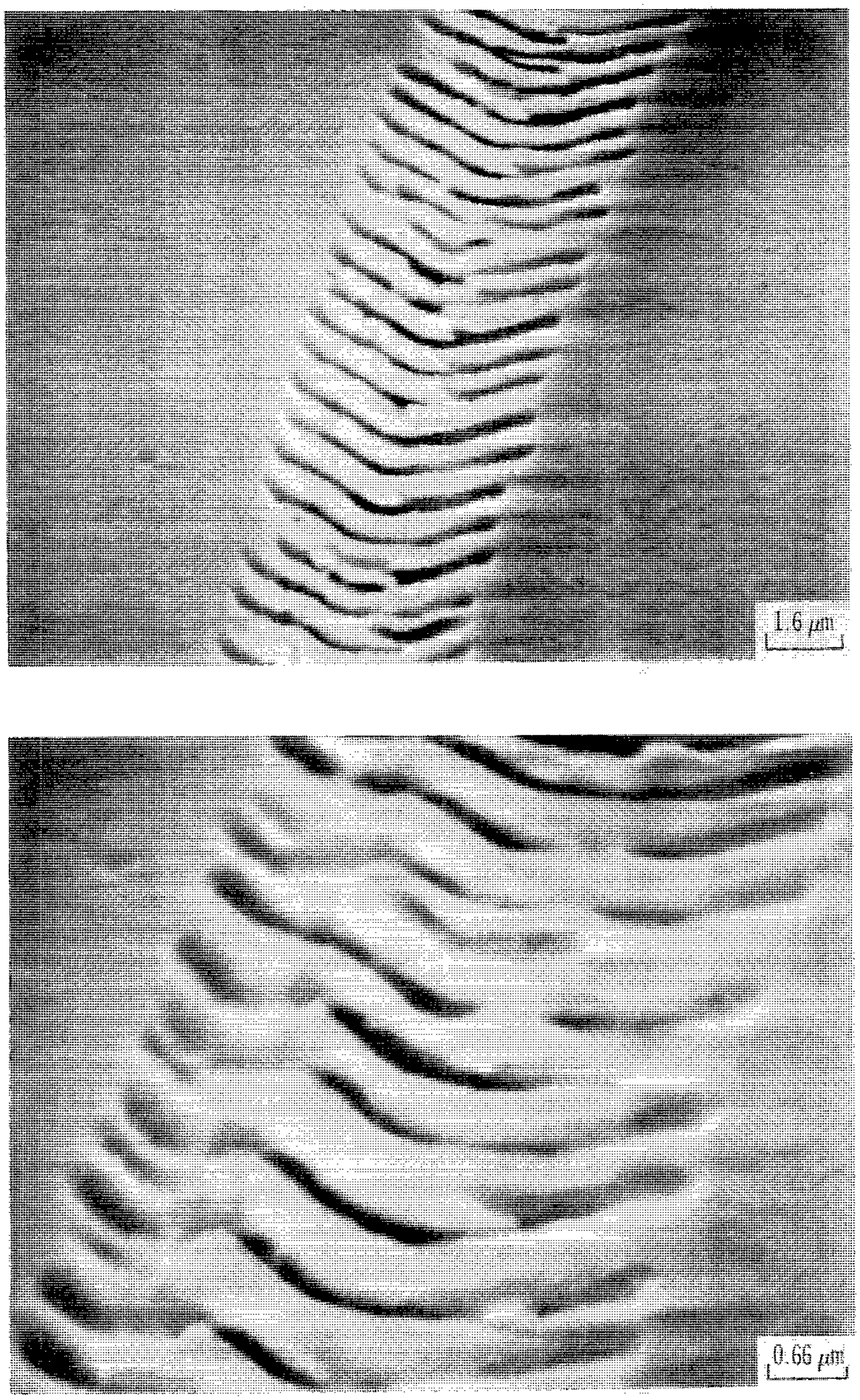


Figure 10. - Wear tracks made by single-crystal iron (110) sliding across silicon (111) surface. Sliding velocity, 0.7 mm/min.; temperature, 23° C; pressure, 10^{-8} N/m^2.

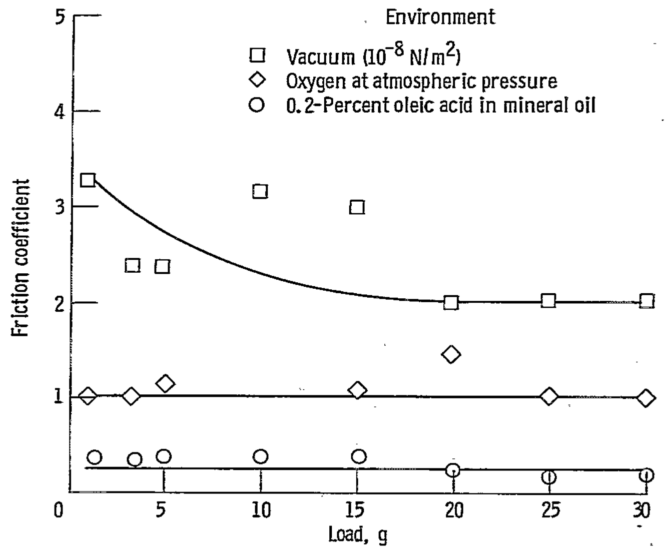


Figure 11. - Friction coefficient as function of load for single-crystal iron (110) sliding on a single-crystal silicon (111) surface in vacuum, in oxygen, and lubricated with 0.2-percent oleic acid in mineral oil. Sliding velocity, 0.7 mm/min; temperature, 23° C.

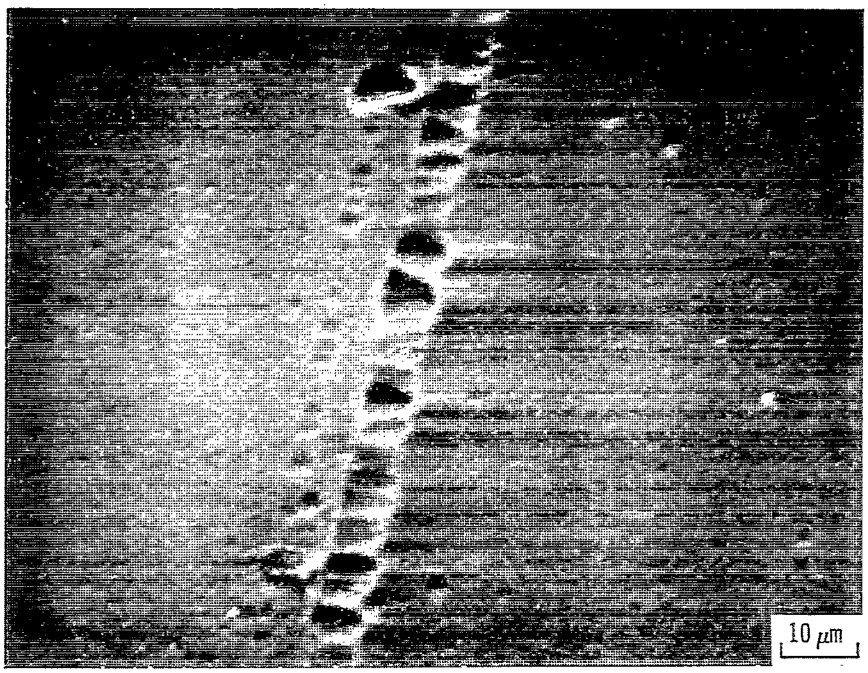


Figure 12. - Etch-pitted wear track made by single-crystal iron (110) sliding across silicon (111) surface, which was lubricated by mineral oil containing 0.2-percent oleic acid. Sliding velocity, 0.7 mm/min.; load, 30 grams; temperature, 23° C; environment, argon at atmospheric pressure.

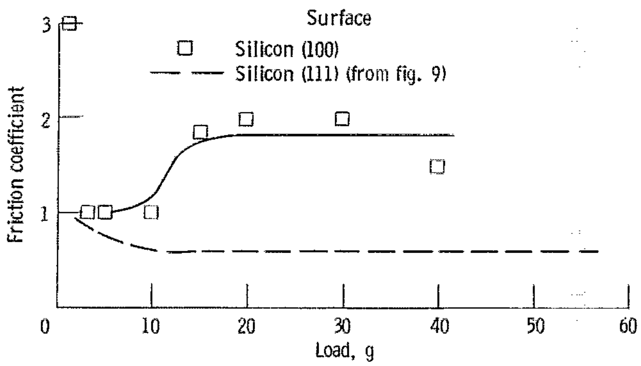


Figure 13. - Friction coefficient as function of load for single-crystal gold (111) sliding on single-crystal silicon (100) and (111) surfaces. Sliding velocity, 0.7 mm/min; temperature, 23° C; pressure, 10^{-8} N/m^2.

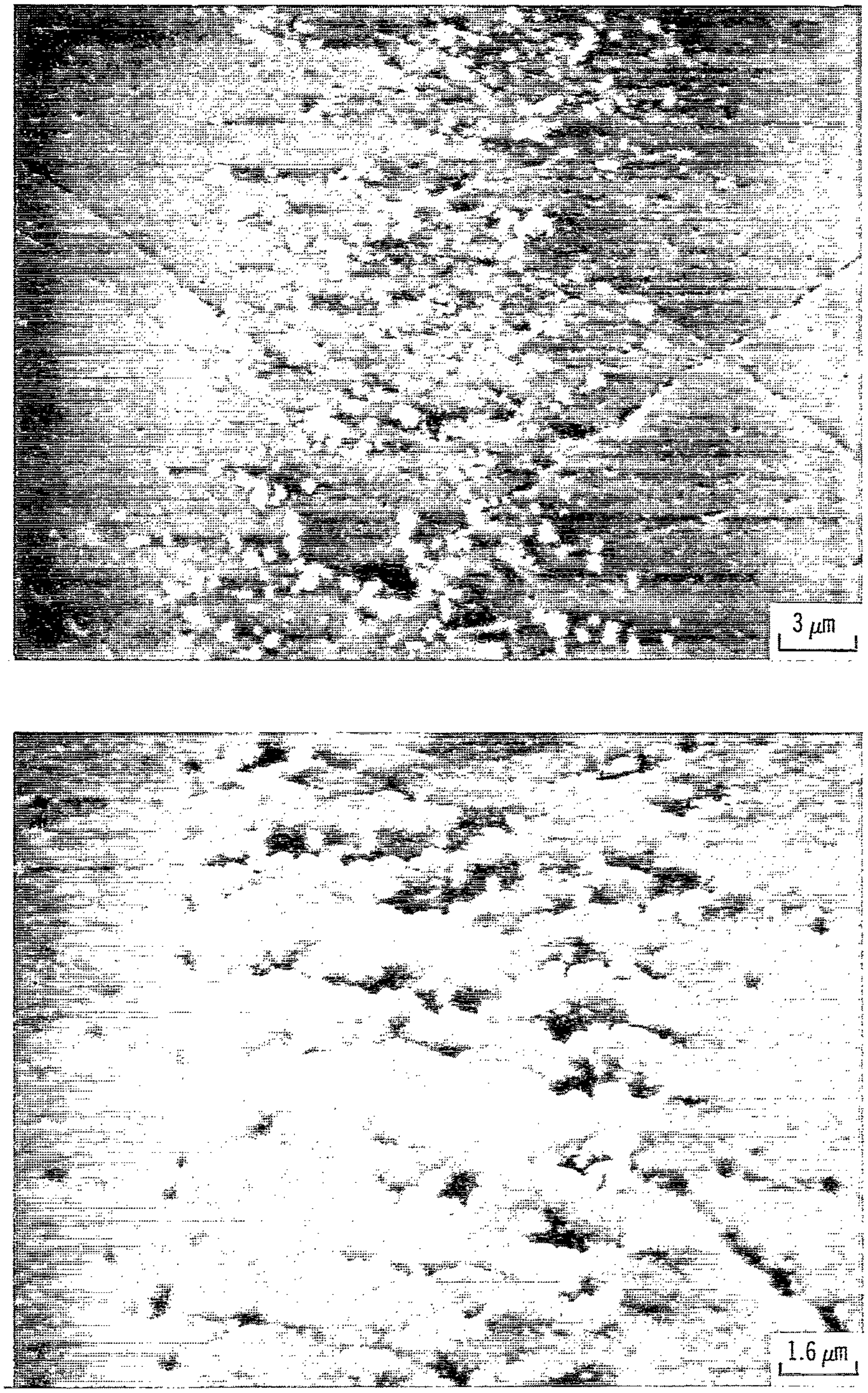


Figure 14. - Wear track made by single-crystal iron (110) sliding across silicon (100) surface. Sliding velocity, 0.7 mm/min.; load, 30 grams; temperature, 23° C; pressure, 10^{-8} N/m^2.

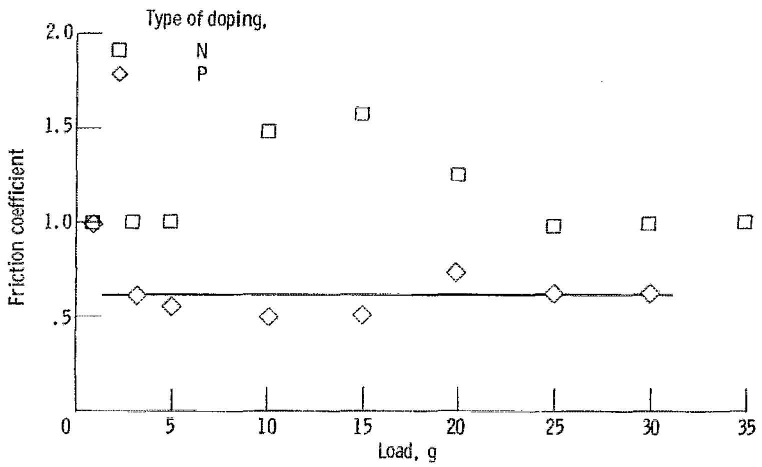


Figure 15. - Friction coefficient as function of load for single-crystal gold (111) sliding on doped single-crystal silicon (111) surfaces. Sliding velocity, 0.7 mm/min; temperature, 23° C; pressure, 10^{-8} N/m^2.

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