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A LEED STUDY OF THE ADHESION OF GOLD TO COPPER AND COPPER-ALUMINUM ALLOYS

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ABSTRACT

The adhesion of the (111) surface of gold to clean (111) surfaces of copper, aluminum, and copper-aluminum alloys was studied with the aid of LEED. Concentrations in copper as low as 1.0 at. % are sufficient to increase the adhesive bonding of gold to copper fivefold. This adhesion is characteristic of what is observed for gold contacting aluminum. The gold also transferred epitaxially to the copper-aluminum alloys, taking on the lattice characteristics of the alloy. Increases in contact load resulted in increases in the amount of gold adhering to the alloy surface.

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SUMMARY

An investigation was conducted to determine the influence of an alloying element on the adhesion of copper to gold. Aluminum was added to copper in concentrations to 10 atomic percent. Experiments were conducted with (111) crystal surfaces of the alloys contacted by the (111) surface of gold. The gold was a 1-millimeter-diameter crystal that was brought into contact with copper, aluminum, and copper alloys under loads of 20 to 300 milligrams in a vacuum of 1.0×10^{-11} torr after the surfaces had been cleaned. The surfaces were examined with LEED (low-energy electron diffraction) before and after adhesive contact.

The results of the investigation indicated that the force of adhesion of gold to copper is markedly increased with aluminum present in the copper.

The addition of 1.0 atomic percent aluminum was sufficient to increase the force of adhesion fivefold. The gold adhered to the alloy surface epitaxially with the gold taking on the alloy lattice structure. The amount of gold transferred to the alloy surface was a function of the load. The greater the applied load, the greater the amount of gold that remained adhered to the alloy surface. The distribution of gold on the alloy surface was fairly uniform in the contact area, as determined by electron microprobe analysis.

INTRODUCTION

It has been demonstrated with the adhesion of metals to themselves that the crystallographic orientations influence the cohesive forces measured (ref. 1). Thus, the surface atomic arrangement exerts an influence on the degree of solid surface interaction, probably by determining the amount of electron interaction that will form across the interface. With metals contacting themselves across an interface, the maximum cohesive forces might be anticipated where the orientations are the same; with mismatching, the cohesive forces are less. This mismatching has been substantiated as being the case with copper (ref. 1).

When dissimilar metals contact across an interface, the tendency is to think of the adhesive forces as being less than cohesive forces because of differences in atomic size and lattice spacings in addition to the normal orientation mismatch encountered in cohesion. This is, however, not the case, as the data of reference 2 demonstrate. With dissimilar metals in contact, the interfacial layers may consist of alloys or intermetallic compounds that form in the adhesion process and whose bonding is stronger than the cohesive bonding in the weaker of the two host metals. Thus, when the surfaces are separated, it is cohesive bond forces that are measured.

The presence of small amounts of alloying elements in metals can markedly alter their surface activity. The addition of aluminum to copper in the bulk, for example, results in an expansion of the copper lattice (ref. 3) and a decrease in the number of surface free electrons with an accompanying decrease in surface energy (ref. 4). Aluminum also forms some intermetallic compounds with copper (ref. 5). It increases the resistance to deformation of copper even with relatively small additions of aluminum (ref. 6). All these factors may be significant in the adhesion of copper to other surfaces.

The objective of this investigation was to extend the study of the copper-gold contacting couple in reference 2 to determine the influence of the alloying element aluminum in copper on the adhesion of copper to gold. Aluminum was alloyed with copper from 0.1 to 10.0 atomic percent. The single-crystal orientation examined in these studies was the (111) surface of the copper-aluminum alloys, and this surface was contacted by a (111) orientation of gold. Experiments were conducted in a vacuum of 10^{-11} torr with clean surfaces. A 1-millimeter-diameter gold flat contacted the copper-aluminum alloys under loads of 20 to 300 milligrams for various contact times.

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 adhesion measurements to be made on the crystal surface shown in figure 1. Then the crystal could be rotated 180° to obtain a LEED pattern 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 two insulated metal rods, and a thin tantalum holder was used to resistance heat the crystal. A 100-ampere ac power supply was used for resistance heating.

The fiber, which contacted the single crystal metal surface, was mounted in a

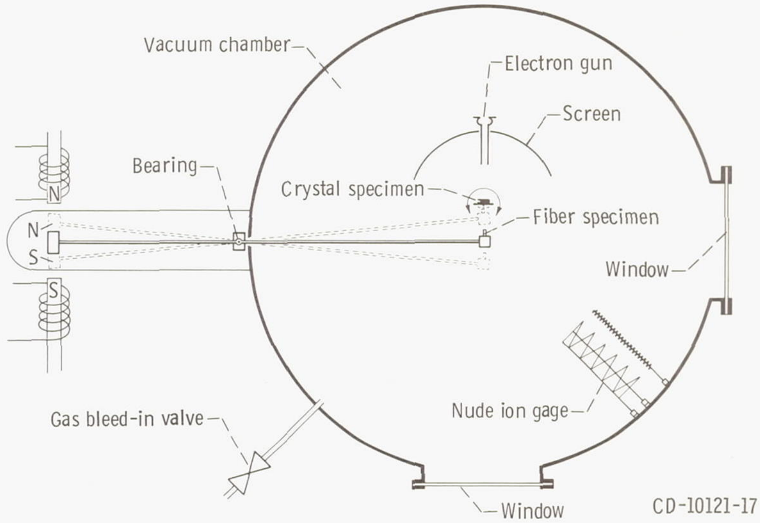


Figure 1. - Leed adhesion apparatus.

stainless-steel holder that 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 fiber specimen was a small permanent magnet. Outside the chamber wall were two electromagnets. The permanent and electromagnets were positioned in such a manner as to have like poles facing each other. A simple 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 LEED electron optics and the vacuum system were the standard type used by those engaged in LEED studies and are adequately described in the literature. The basic LEED system was obtained commercially. The electron optics system was the Varian three-grid type. The diameter of the beam 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 pressures from 1.0×10^{-11} to 8.0×10^{-1} torr. No cryopumping was used.

MATERIALS

The copper, gold, and copper-aluminum alloys used in this investigation were all of triple zone refined material. The single-crystal alloys were cut from rods whose axes were normal to the (111) surface. Cutting slices normal to the rod axis gave specimens

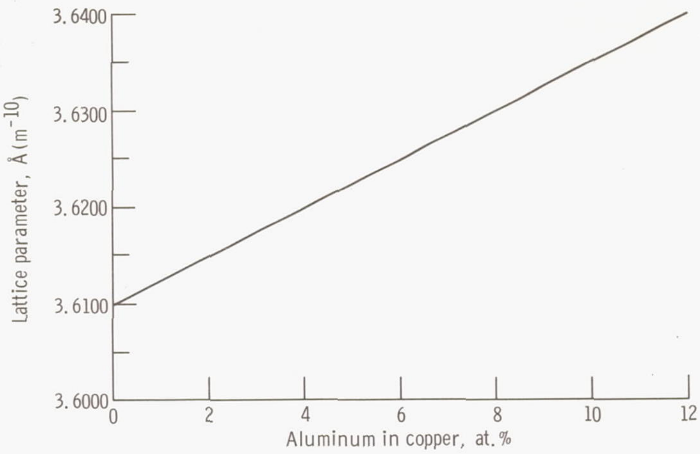


Figure 2. - Influence of aluminum additions on lattice parameter of copper (ref. 3).

5 millimeters thick with diameters varying from 5 to 7 millimeters, depending on the particular alloy rod. The crystal flats were polished to 600 grit on metallurgical papers and then electropolished in orthophosphoric acid. Orientations were checked with the Laue X-ray technique before and after finishing the specimens. The gold was treated in essentially the same manner except that the electropolishing solution was an aqueous solution of hydrofluoric and nitric acids.

The expansion of the copper lattice with the addition of aluminum is shown in figure 2 (data from ref. 3). In the solid solution, the addition of aluminum indicates a continuous expansion of the copper lattice.

EXPERIMENTAL PROCEDURE

The crystal surfaces were cleaned in the LEED adhesion apparatus after bakeout of the system, and the pressure in the chamber reached 10^{-11} torr. The crystal surfaces were heated initially for 3 hours at 500° C to outgas the crystal thoroughly. The pressure rose in the chamber and with prolonged crystal heating would subsequently decrease. The temperature was decreased to 450° C, and hydrogen gas was admitted to the chamber to reduce surface copper oxides. The pressure in the chamber was raised to 10^{-6} torr with hydrogen, and after 15 minutes of hydrogen exposure, the pressure was reduced and the crystal heated to 550° C to remove the surface hydrogen.

The copper and copper-aluminum alloys were mounted in tantalum holders. The gold specimen was cleaned by contacting the cylindrical side of the gold crystal near the contacting flat with the tantalum specimen holder containing the copper alloy specimen. The crystals were heated by resistance heating the tantalum. When the specimens were cooled to room temperature, the pressure in the system was 1.0×10^{-11} torr.

The aluminum crystal surfaces were cleaned by heating the crystal to 600° C and holding it at this temperature for 72 hours to allow the oxygen to diffuse into the metal. Cleaning by this technique was found to be equally effective to ion bombardment (ref. 7), and it was used herein in preference to ion bombardment because it does not disturb the surface topography as ion bombardment is known to do.

EXPERIMENTAL RESULTS

The data of figure 3 indicate the force required to separate the gold specimen from copper, aluminum, and various copper-aluminum alloys. At 0, 0.1, and 0.5 atomic percent aluminum, the force to fracture the contact was 100 milligrams or less. When, however, the aluminum content in the copper was increased to 1.0 atomic percent, a marked increase in adhesive force occurred.

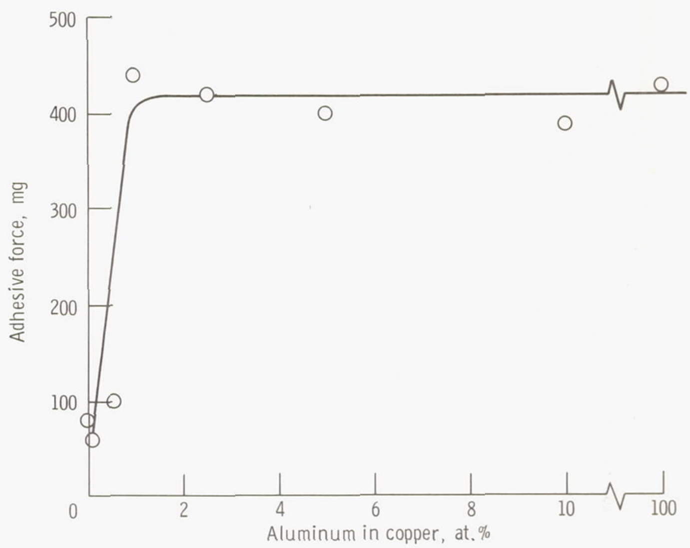


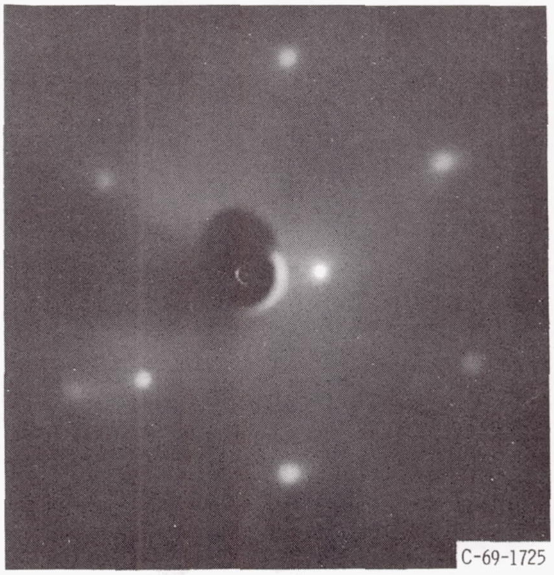
Figure 3. - Adhesive force of (111) gold to (111) surface of copper, aluminum, and various aluminum-copper alloys. Initial applied load, 20 milligrams; contact time, 10 seconds.

Examination of the contact surface with LEED after the adhesive junction had been broken revealed the structure shown in the photographs of figure 4 for the 0.5 and 1.0 atomic percent aluminum in copper alloys. With the 0.5 percent aluminum (fig. 4(a)), a double set of diffraction spots indicative of epitaxial adherence of gold to the copper-aluminum alloy was observed. This same type of diffraction pattern was observed with both 0.1 atomic percent aluminum in copper and pure copper. The pattern for the adherence of gold epitaxially to pure copper is shown in reference 2.

The LEED pattern photograph of figure 4(b) indicates a single set of diffraction spots that characterized the pattern for the 0.5 atomic percent aluminum in copper before adhesive contact. Gold is present on the surface of the copper alloy as shown in the section EXPERIMENTAL RESULTS.

The presence of a double set of diffraction spots in figure (4) is indicative of the mismatch in the lattice parameters of copper and gold, as was observed in reference 2. The

(a) Clean (111).



(b) After 20-milligram contact with gold.

Figure 4. - LEED patterns of aluminum (111) surface before and after adhesive contact with gold.

gold initially adheres epitaxially but with its own lattice parameter, which deviates from copper and accounts for the double diffraction spots. This same effect has been observed by others with vapor deposition of one metal on another metal surface (refs. 8 and 9). When one material deposits or adheres to another surface and sufficient material is present but no change in the substrate pattern occurs as in figure 4(b), this is indicative of the adhered material taking on the lattice of the substrate material.

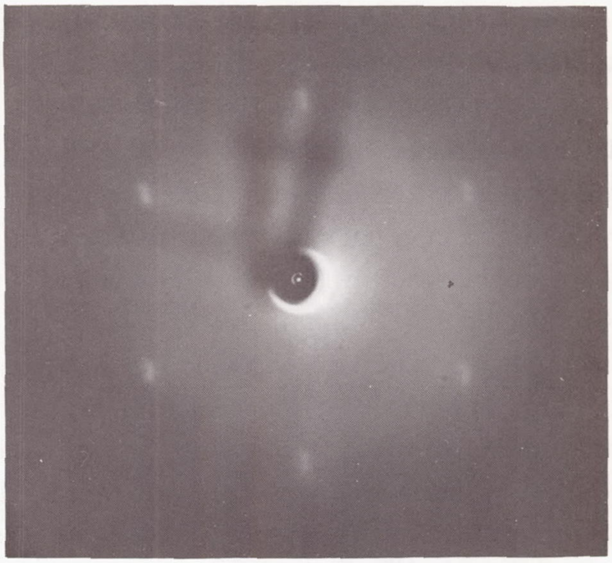
Further increase in the aluminum content in the copper from 1.0 to 2.5, 5.0, and 10.0 atomic percent aluminum resulted in larger adhesive forces than that obtained for the 1.0 atomic percent aluminum in copper alloy, as shown in figure 3. The forces to separate the surfaces were approximately 20 times the applied load.

The force of adhesion of gold to the aluminum (111) surface is presented in figure 3. The force measured is comparable with that obtained for gold contacting the copper-aluminum alloys with 1.0 atomic percent or more aluminum. It may well be that the copper alloy surfaces are aluminum rich. Work with iron has shown that the addition of as little as 1.0 atomic percent aluminum to the bulk can result, due to diffusion, in a surface concentration of 35 atomic percent aluminum. A similar effect may account for the results of figure 3.

Photographs of the aluminum (111) surface before and after adhesive contact with gold are presented in figure 5. The clean aluminum surface, the pattern of figure 5(a), and the pattern for that same surface after adhesive contact with gold is presented in figure 5(b). Although gold adhered epitaxially to copper and copper-aluminum alloys, no adherence was observed to occur with aluminum. The aluminum, less resistant to strain than the gold, underwent strain, as indicated by the streaking of some of the diffraction spots in figure 5(b).

The LEED photographs of figure 6 indicate the effect of adhesion on the surface structure of the 2.5 atomic percent aluminum in copper alloy. Figure 6(a) shows the pattern for that surface after adhesive contact with gold under a load of 20 milligrams. The diffraction spots are slightly elongated. After the surface of figure 6(a) was heated to 400° C for 5 minutes, the pattern of figure 6(b) resulted. Examination of figure 6(b) revealed the presence of a double set of diffraction spots. Figures 6(a) and (b) indicate that, although gold initially adheres epitaxially, with the gold taking on the copper-aluminum alloy lattice, the addition of sufficient energy by heating will bring about rearrangement of the gold so that it will develop its own lattice characteristics.

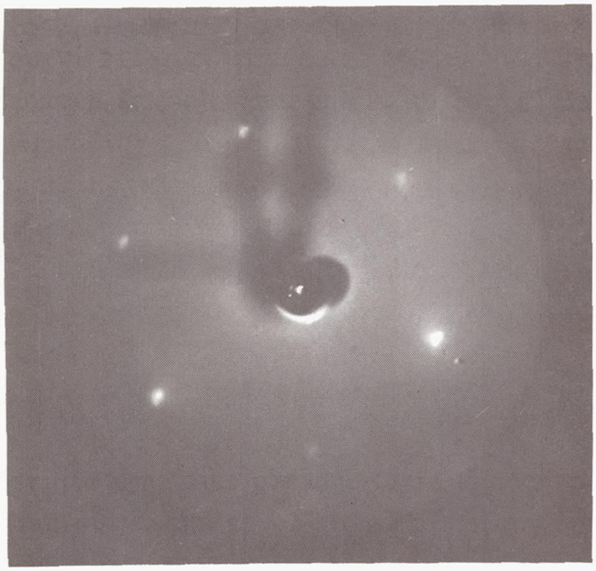
The difference in lattice spacing shown in figure 6(b) could be achieved without heating by simply increasing the contact load of gold to copper. Figure 6(c) presents the 2.5 atomic percent aluminum in copper alloy surface after gold had contacted it under a load of 200 milligrams. Two interesting observations can be made from figure 6(c): the first is that a difference in the gold and copper alloy lattice is observed. This difference was essentially achieved under a 20-milligram load (fig. 6(a)) only after heating (fig. 6(b)).



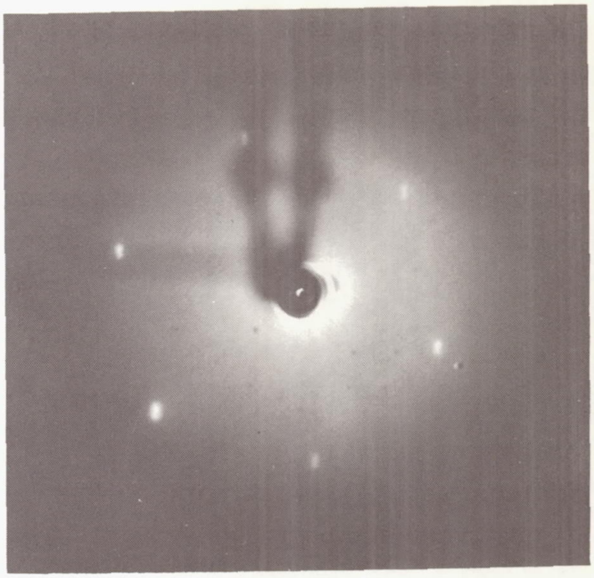
(a) 0.5 Atomic percent aluminum in copper.

(b) 1.0 Atomic percent aluminum in copper.

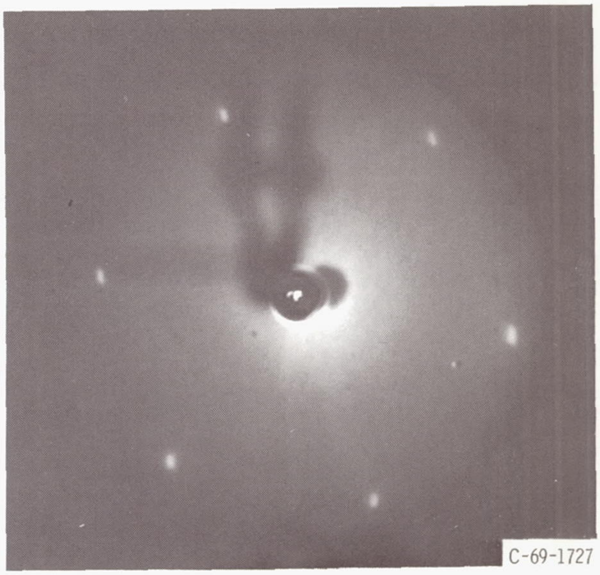
Figure 5. - LEED patterns of two aluminum-copper alloy (111) surfaces after adhesive contact with gold. Load, 20 milligrams; contact time, 10 seconds.



(a) Load, 20 milligrams.



(b) Load, 20 milligrams with subsequent heating to 400° C.

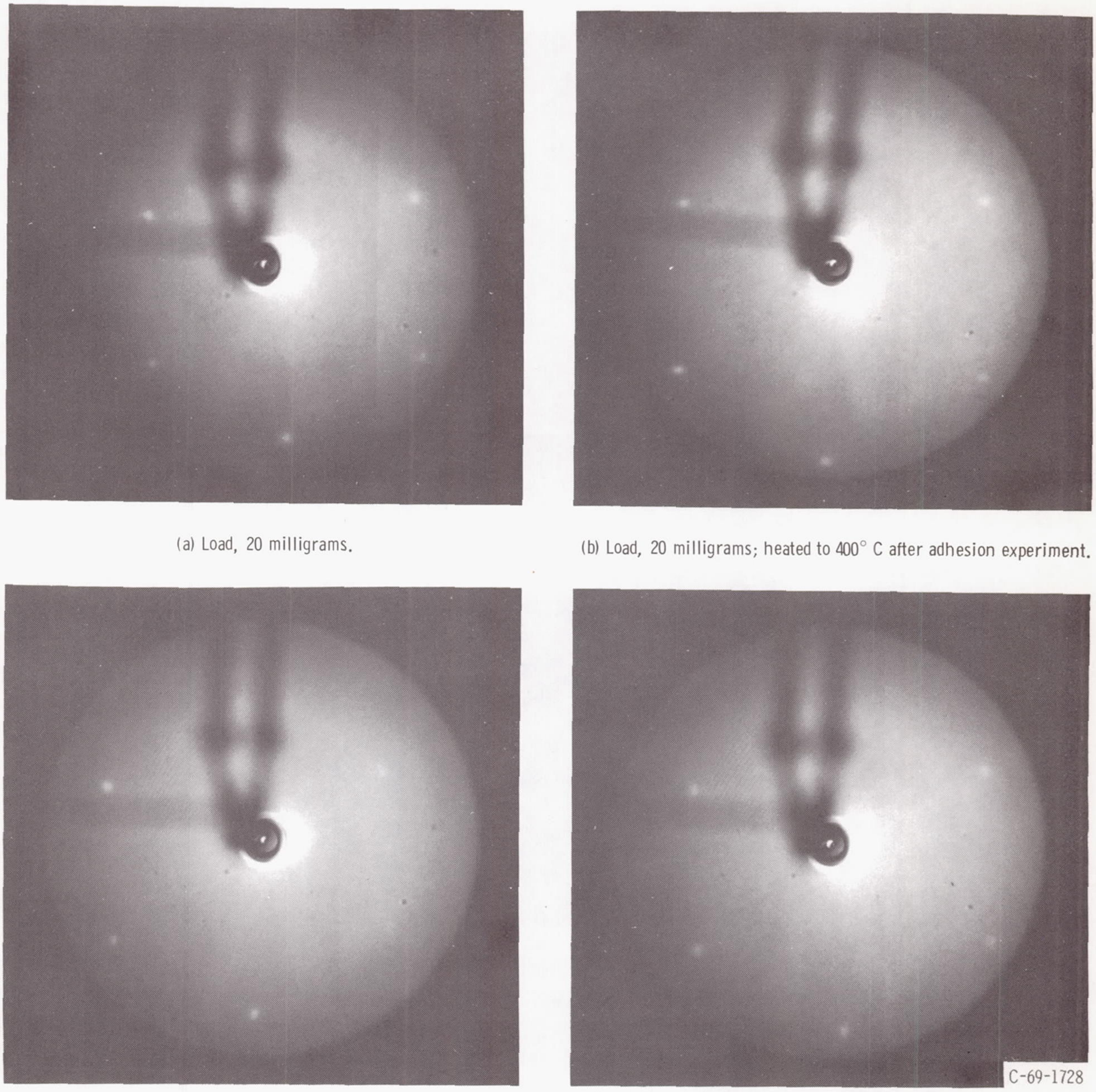


(c) Load, 200 milligrams.

Figure 6. - LEED patterns of surface of 2.5 atomic percent aluminum in copper alloy after contact with gold at two loads.

Thus, apparently the energy associated with the higher load appears to bring about the lattice change without the necessity of heating the surface. The second observation is that, even at loads as high as 200 milligrams, no noticeable strain appeared to take place in the 2.5 atomic percent copper-aluminum alloy. This observation is in keeping with those made earlier, in reference 2, that the cohesively weaker metal (in this study, the gold) is straining under load.

The alloy of 5.0 atomic percent aluminum in copper exhibited a very high adhesive



(a) Load, 20 milligrams.

(b) Load, 20 milligrams; heated to 400° C after adhesion experiment.

(c) Load, 100 milligrams.

(d) Load, 300 milligrams.

Figure 7. - LEED patterns of surface of 5.0 atomic percent aluminum in copper alloy after adhesive contact with (111) gold surface at various loads.

bonding force to gold, as shown by the data of figure 3. Photographs of LEED patterns obtained before and after adhesive contact for the 5.0 atomic percent alloy are presented in figure 7. The pattern obtained after contact under a 20-milligram load is shown in figure 7(a). The pattern after the surface was heated for 5 minutes at 400° C is shown in figure 7(b). Figures 7(c) and (d) present the patterns obtained from the 5.0 atomic percent alloy surface after contact with gold under loads of 100 and 300 milligrams, respectively. Although difficult to discern from the photographs, very close double diffraction spots exist in the patterns of figures 7(c) and (d).

Although the LEED patterns of figure 7 indicate the presence of gold on the alloy surface, they give no indication of the quantity being transferred and its distribution. Figure 8 is a plot of the current collected on the LEED screen by the electrons that have been

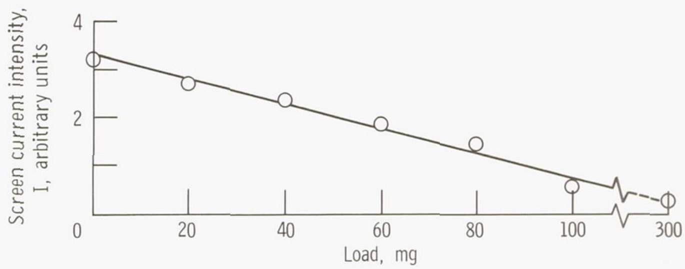
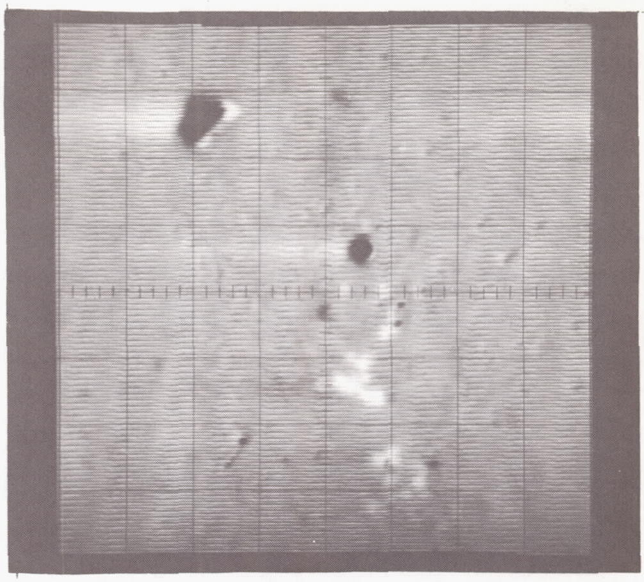


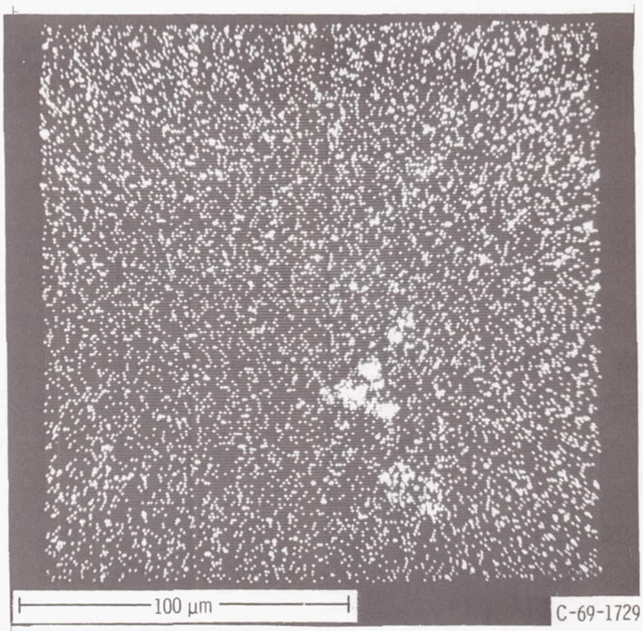
Figure 8. - LEED screen current intensity as function of contact load for (111) surface of gold contacting (111) surface of 5.0 atomic percent aluminum in copper alloy. Beam voltage, 23 volts.

elastically diffracted from the specimen surface. With an increase in load, a direct relation to screen current intensity is seen. This relation, in essence, indicates that, as the load is increased, the amount of gold transferred to the alloy surface continues to increase. With increasing quantities of gold on the surface, the intensity of the copper patterns continues to decrease. The amount of transferred gold appears to be a linear function of applied load.

An electron microprobe analysis of the 5.0 atomic percent aluminum-copper alloy surface was performed to determine the distribution of the gold on the alloy surface. The results are presented in figure 9. The photograph of figure 9(a) is a pattern of specimen surface current, and figure 9(b) is a ratemeter surface scan showing gold distribution (note white spots). The most interesting result of the microprobe analysis is the relatively uniform distribution of the gold on the copper surface shown in figure 9(b). The only area where any nonuniform transfer seems to have occurred is that near the center of the photographs in figures 9(a) and (b), where some clumping of the gold appears. Even here, however, the thickness of the transferred gold was less than 1000 Å (10^{-7} m) as determined by microprobe peak intensities.



(a) Specimen current.



(b) Ratemeter scan.

Figure 9. - Electron microprobe analysis of 5.0 atomic percent aluminum in copper alloy surface revealing presence and distribution of gold on surface after adhesive contact. Load on gold specimen, 20 milligrams; contact time, 10 seconds.

The adhesion force measured for metals in contact is frequently a function of contact time; the longer the time in contact, the greater the force required to separate the surfaces. This principle was demonstrated in reference 1 for copper crystals in contact. The adhesion force as a function of contact time was measured for gold in contact with the 5.0 atomic percent aluminum in copper alloy, and the results obtained are presented in figure 10. These results indicate that, even for relatively short contact times, marked differences in adhesion forces can be measured with variations in contact time.

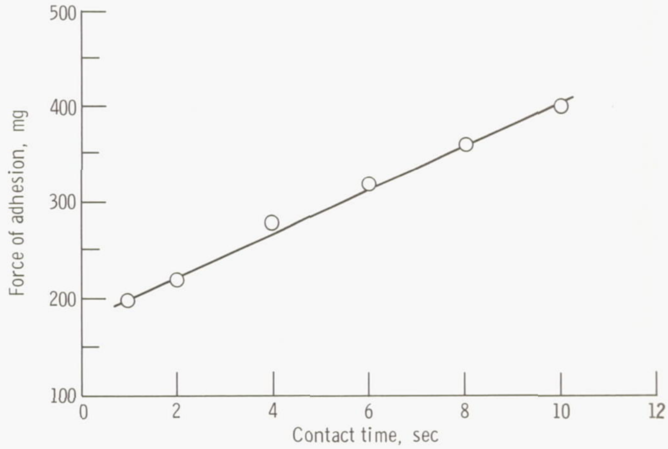
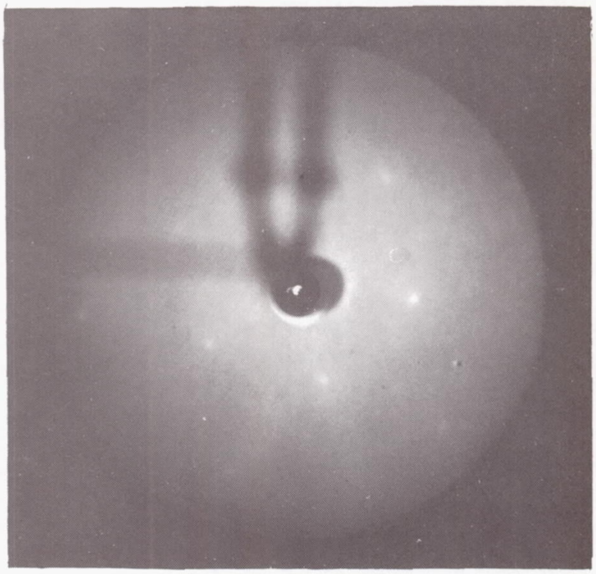


Figure 10. - Effect of contact time on adhesion of gold (111) surface to 5.0 percent aluminum in copper alloy (111) surface. Load applied, 20 milligrams.

With the adhesion of gold to the 10 atomic percent aluminum in copper alloy, an adhesive force was measured similar to that measured with the 5.0 atomic percent alloy, as shown by the data of figure 3. Analysis revealed that less gold had transferred to the 10.0 atomic percent alloy than to the 5.0 atomic percent alloy under equivalent loads and contact times. Figures 11(a) and (b) present photographs of LEED patterns after contact at two loads of 20 and 200 milligrams, respectively. The results indicate that gold is still transferring epitaxially at 10 atomic percent aluminum. The pattern of figure 11 is contracted because the beam voltage was increased from the 63 volts used in some of the prior figures to 100 volts.

Although the amount of gold transferred to the 10 atomic percent alloy was less than that observed with adhesion to the 5.0 atomic percent alloy, it was greater than that observed with the 0.5 atomic percent alloy. This is demonstrated in terms of mechanical strength properties by the data of figure 12, where the force required to fracture adhesive junctions is shown as a function of time required to fracture the junctions in tension. This figure basically presents the results of a tensile test on the junction. Since fracture is occurring in the cohesive gold bonds, the values indicated for 300 seconds and beyond



(a) Load, 20 milligrams.

(b) Load, 200 milligrams.

Figure 11. - LEED patterns of 10 atomic percent aluminum in copper alloy after adhesive contact with gold at two loads.

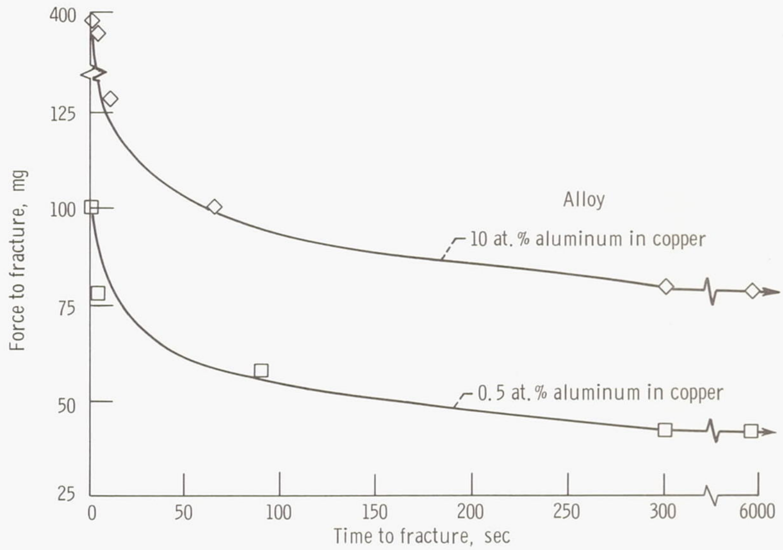


Figure 12. - Force to fracture contact of gold to copper alloy as function of time under tensile load. Initial applied force for adhesion, 20 milligrams; both gold and copper alloy of (111) orientation.

represent the force below which fracture of the junctions will not occur because the junction is elastic. The elastic limit is a known value for gold, and therefore the difference in limiting values presented in figure 12 for the two alloy compositions represents the difference in area under stress (see ref. 2). With the addition of aluminum to the copper in excess of 1.0 atomic percent, the extent of bonding across the interface appears to increase with an increase in interaction of the Fermi surfaces.

DISCUSSION

The results of this investigation indicate that gold adheres to copper-aluminum alloys in much the same way as it does to pure copper, namely, in an epitaxial manner. The data of reference 2 indicated that the orientation of the copper did not alter epitaxial adhesion. With copper, gold did take on the copper lattice but retained its own characteristic lattice parameters. In this study, however, gold was observed in the adhesion process to take on the lattice parameters of the copper-aluminum alloy. With the addition of energy by heating, surface rearrangement of the gold could be brought about to develop a lattice differing from that of the copper alloy. The tendency toward such rearrangement is not unusual since this may be the more stable state, and the work of others seems to indicate that epitaxial films, even monolayers, are observed to deposit with a retention of their own lattice parameters (refs. 8 and 9).

With the expansion of the copper lattice by the addition of aluminum to the copper toward that of the gold lattice, it was anticipated that a continued increase in surface adhesion would occur. The results of figure 3, however, seem to indicate that this is not the case.

It is of interest to note that, with all the copper alloys studied, except for the 0.1 atomic percent aluminum, the forces required to separate the surfaces were higher than that observed in reference 2 for the copper (111) surface. Thus, the presence of aluminum as an alloying element exerts an influence on the adhesion process; however, it does not appear to be related simply to alterations in lattice parameter. Rather, with the addition of 1.0 atomic percent or more aluminum, the adhesive forces appear to be those measured for aluminum. Again, these forces may be due to a preferential segregation of aluminum at the surface.

The adhesive force of one surface to another should be a function of the Fermi surfaces that interact across an interface. This will be determined in part by a number of the closest interatomic approaches necessary for bonding. With a metal like copper, as shown in reference 2, simply changing the orientation will result in a change in the number of interatomic approaches necessary to achieve bonding across the interface. With copper, surface arrangement will principally be responsible for the forces of surface adherence, since it will alter the nature of bonding occurring.

The situation with respect to alloy surfaces is different. If, for example, the (111) surface of the 10 atomic percent aluminum in copper alloy is considered, a minimum of 1 out of 10 atoms on the surface should be aluminum. As just discussed, the adhesion results indicate that the number of aluminum atoms is much higher. The nature of the Fermi surface has been altered and the interaction of aluminum with gold must be considered. In the noble metals, copper, silver, and gold, the cohesive energy is derived mainly from the electrons in the d-bands, and hence these metals are tightly bound. In aluminum, on the other hand, the d-band is unoccupied, and the electrons that contribute to the cohesive energy come from the conduction band alone, causing this metal to be more compressible than either copper or gold (ref. 10).

If the interaction of aluminum is considered with respect to its affinity for gold, some insight into bonding forces may be obtained from a consideration of the distance of the closest approach and alterations of lattice spacing. When aluminum alloys with copper, a contraction occurs because of Brillouin zone overlaps. A 20-percent greater contraction, however, occurs when aluminum is alloyed with gold (ref. 10). Further, as shown in figure 2, a lattice expansion occurs on the addition of aluminum to copper. When aluminum alloys with gold, a contraction in lattice is observed to occur.

Thus, from the foregoing, it may be concluded that, in copper-gold contact, the gold undergoes compression at the interface. With the contact of the gold to the copper-aluminum alloy, the compressibility of the aluminum is greater than that of gold. This will affect the band at the interface across which interaction will occur. The stronger affinity of aluminum for gold than of gold for copper or copper for gold will result in stronger interfacial bonding. In simple terms, the aluminum adheres better to gold than does copper; thus the increase in adhesive force to fracture.

It must be pointed out that the fracture in all of these experiments is occurring in the cohesive bond of gold; that is, gold subsurface bonds are being broken. The number of subsurface bonds broken is related to the amount of bonding that occurs at the interface (ref. 2), which is related to the atomic surface arrangement and to the affinity of the surfaces for each other. The presence of aluminum in copper enhances surface affinity because of the strong bonding of aluminum to gold. If an alloying element were added to copper, an element that has a weaker affinity than copper for gold, a reduction in adhesive forces with alloying might be anticipated. The dichotomy here is that, although this reduction is the case for clean surfaces in contact, just the opposite may be true in an active gas environment such as air. The aluminum in the copper enhances the formation of surface passive oxides that would tend to reduce adhesion.

The data of figure 10 are interesting in that they show a marked increase in the adhesive bonding with changes in contact time. The changes in time are only a matter of seconds, and yet drastic changes in adhesion forces occur. Such changes might be throught of as being associated with any one or all three of the following mechanisms: (1) diffusion, (2) reconstruction, and (3) creep of the gold. All these mechanisms are time-dependent phenomena. In light of the fact that here only a few atomic layers are involved the times involved in figure 10 may be meaningful for all three of these processes.

SUMMARY OF RESULTS

LEED was used in an investigation of gold contacting clean aluminum in copper alloys, and the following results were obtained:

1. The addition of small concentrations of aluminum to copper markedly increased the adherence of the copper to gold. The addition of 1.0 atomic percent of aluminum to copper resulted in a greater than fivefold increase in the force of adhesion. With the 1.0 atomic percent aluminum, the adhesive force was comparable with that obtained for aluminum. This increase may be related to the strong affinity of aluminum for gold.

2. The amount of gold transferred to the copper-aluminum was directly related to the load applied to the surfaces in contact. The higher the load, the greater the amount of gold transferred. An electron microprobe analysis revealed that the distribution of the gold on the surface was fairly uniform.

3. The adhesion forces measured depended very strongly on the time the surfaces were in contact. The adhesion force increased with increasing contact time.

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