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Tribological Characteristics of Gold Films Deposited on Metals by Ion Plating and Vapor Deposition

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Abstract

Recent work on the graded interface between an ion-plated film and a substrate is discussed as well as the friction and wear properties of ion-plated gold. X-ray photoelectron spectroscopy (XPS) depth profiling and microhardness depth profiling were used to investigate the interface. The friction and wear properties of ion-plated and vapor-deposited gold films were studied both in an ultra-high-vacuum system to maximize adhesion and in oil to minimize adhesion. The results of the investigation indicate that the solubility of gold on the substrate material controls the depth of the graded interface. Thermal diffusion and chemical diffusion mechanisms are thought to be involved in the formation of the gold-nickel interface. In iron-gold graded interfaces the gold was primarily dispersed in the iron and thus formed a physically bonded interface. The hardness of the gold film was influenced by its depth and was also related to the composition gradient between the gold and the substrate. The graded nickel-gold interface exhibited the highest hardness because of an alloy hardening effect. The effects of film thickness on adhesion and friction were established. A minimum coefficient of friction was found in the thin-film region. No graded interfaces were detected in this investigation between vapor-deposited gold films and substrates.

INTRODUCTION

This paper is concerned with a fundamental understanding of the interface between an ion-plated gold film and a metal substrate as well as the friction and wear properties of ion-plated gold.

To understand the mechanisms of adhesion and interface formation and to improve the tribological properties of thin-film coatings, it is important to know the compositional variation in the film as well as in the interface between the film and the substrate. Various techniques and surface analysis tools for studying the atomic nature of a graded interface have been reported in the literature (1-9). Such techniques include optical microscopy, Auger electron spectroscopy (AES), secondary ion mass spectrometry (SIMS), ion scattering spectroscopy (ISS), and electron microscopy. A number of graded interfaces, namely gold on aluminum, aluminum on titanium, indium on copper, gold on copper, gold on chromium, chromium on nickel and aluminum, and copper on nickel have been investigated (1-9). Because these material pairs have solid solubility, thermal diffusion is possible. A few attempts have been made to detect and analyze the interface for materials where diffusion is negligible (9). The surface analysis techniques used in the present study were X-ray photoelectron spectroscopy (XPS), depth profiling, and microhardness depth profiling (10, 11).

The friction and wear properties of ion-plated gold were determined with flat specimens (composed of the film, the graded interface, and the substrate) sliding against silicon carbide pins in two processes. The first was an adhesive process, in which friction arose primarily from adhesion between sliding interfaces. This was done in an ultra-high-vacuum system to maximize adhesion. The second was a nonadhesive process (i.e., abrasion), in which friction occurred as a result of the silicon carbide sliding against the film, indenting it, and plowing a series of grooves. This was done in mineral oil to minimize adhesion.

DESCRIPTION OF EXPERIMENT

Materials

The plating material was 99.99-percent-pure gold. The substrates were 99.999-percent-pure copper and 99.99-percent-pure nickel and iron. The rider was 99.9-percent-pure single-crystal silicon carbide. The mineral oil used for lubrication was a pharmaceutical grade that had been degassed. Typical properties of the mineral oil are given in Table 1.

Apparatus

Coating. - The ion-plating chamber used in this study is described in (11). The chamber is evacuated by a mechanical-oil and diffusion pumping system and a liquid-nitrogen trap. An alternative pumping system was also used to eliminate any possible external contamination, such as oil backstreaming, during the pumping cycle. The mechanical and diffusion pumps were isolated from the chamber, and the pumping was performed directly by two vacuum sorption pumps. The chamber was also used to vapor deposit gold.

Friction. - The two apparatus used in this investigation are described in (11). Each was basically a pin (rider) sliding on a flat. One was capable of measuring adhesion, load, and friction in vacuum; it also contained X-ray photoelectron spectroscopy for surface analysis and an ion gun for ion sputter etching (depth profiling). The second apparatus was capable of measuring friction in oil; load was applied by placing deadweights on a pan that rested on top of the rod containing the pin specimen.

Procedure

Coating and XPS depth profiling. - The ion-plating and XPS depth profiling procedures are described in (10). The ion-plating experimental configuration was also used to vapor deposit the gold films. Before vapor deposition the substrates were dc sputter cleaned for about 10 min, and then the chamber was pumped to a pressure of 67 mPa (5 μtorr), at which pressure the gold vapor deposition was performed.

Friction experiments. - In situ friction experiments were conducted in a vacuum of 30 nPa with loads to 0.2 N. The radius of the silicon carbide pin specimen was 0.79 mm. The time in contact before sliding was 30 s. The frictional force was continuously monitored. The sliding velocity was 5×10^{-2} mm/s with a total sliding distance of about 3 mm. The pin specimens were also sputter cleaned in vacuum.

In the oil experiments a 0.025-mm-radius silicon carbide pin was used. Both the silicon carbide surfaces and the ion-plated gold surfaces were rinsed with absolute ethyl alcohol before use. The time in contact before sliding was 30 s. Each friction experiment was a single pass over a total sliding distance of 3 to 10 mm at a sliding velocity of 0.7 mm/s. The experiments were conducted in mineral oil at 25° C.

The vacuum environment was used to maximize the adhesion effect; the oil environment was used to minimize the adhesion effect. The 0.79-mm-radius silicon carbide pin was used to lessen the plowing effect on friction; the 0.025-mm-radius silicon carbide pin was used to heighten the plowing effect. The surfaces of the pin specimens were polished with 3-μm-diameter diamond powder and 1-μm-diameter aluminum oxide (Al_2O_3) powder.

Hardness. - The conventional microhardness (Vickers) tester used was mounted on a vibration-free table. The indentation experiments were conducted at loads to 3 N at 25° C in air with a relative humidity of 40 percent. The hardness depth profiling was conducted at a load of 0.1 N. The ion-plated gold specimen was etched by argon ion sputtering. The hardness was obtained by averaging three to five indentation measurements.

RESULTS AND DISCUSSION

X-Ray Photoelectron Spectroscopy Depth Profiling

XPS survey spectra of gold films ion plated on nickel and iron substrates, obtained before sputter cleaning, revealed a carbon peak caused by atmospheric contamination as well as an oxygen peak (11, 12). A layer of adsorbate present on the outermost surface consisted of water vapor and carbons from the atmospheric environment. The XPS spectra of ion-plated gold surfaces that had been sputter cleaned for 5 min showed no contamination peaks. Typical XPS spectra of the Au 4f, Ni 2p, and Fe 2p peaks obtained from narrow scans for gold ion-plated on nickel and iron are presented in Figs. 1 and 2. The XPS spectra taken 5 min after the gold surfaces had been sputter cleaned clearly reveal the gold but no nickel or iron peaks. After subsequent sputter etching the height of the nickel and iron peaks increased with longer sputtering times, but the gold peaks decreased and finally disappeared from the spectra. For the nickel substrate the gold peak had disappeared from the spectrum after a sputtering time of 3.25 hr; and for the iron substrate, after 72 min. The average depths of the areas analyzed by XPS were approximately 6 μm for 3.25 hr of sputtering and 1.5 μm for 72 min.

Composition of surface (at. %) for gold films ion-plated on nickel and iron were obtained as functions of the sputtering time from XPS analyses. Typical exampl[illegible] presented in Fig. 3. The compositi[illegible] gold did not change in the first 20 min of sputtering and thereafter gradually decreased with increasing sputtering time. However, the nickel and iron increased with increasing sputtering times.

The graded interface for gold on nickel was approximately 6 μm thick; that for gold on iron was approximately 1.5 μm thick. The deeper graded interface between a gold coating and a nickel substrate is due to the solid solubility of the material pairs. Gold has a continuous solid solubility in nickel but has limited solubility in iron (the solubility of gold in α-Fe at 850° C being 1.5 at. % (13)). Gold and nickel in a graded interface can form an alloy.

Thermal and chemical diffusion are thought to be involved in the formation of the gold-nickel interface. At moderate gold deposition rates (several hundred angstrom per minute) the nickel substrate temperature can reach 300° to 500° C (14). On the other hand, the gold in the graded gold-iron interface is atomically dispersed in the iron and limited thermal diffusion results.

Compositions of the surface (at. %) for gold films vapor deposited on copper, nickel, and 440C stainless steel were obtained as functions of sputtering time. Typical examples are presented in Fig. 4. The composition of gold rapidly decreased with increasing sputtering time; the composition of copper, nickel, and 440C stainless steel rapidly increased with increasing sputtering time to 0.2, 0.4, and 0.5 hr, respectively. No graded interface between the vapor-deposited film and substrate is evident in Fig. 4. However, the figure does show a longer sputtering time to remove gold from copper and nickel than from 440C stainless steel. This may be due to the high chemical affinity of gold for copper and nickel.

Indentation Hardness Depth Profiling

Micro-Vickers hardness measurements were conducted in air at atmospheric pressure on gold films both ion-plated and vapor deposited on nickel substrates.

Fig. 5(a) presents the microhardness of ion-plated gold films, graded nickel-gold interfaces, and nickel substrates as a function of depth from the surface of the film. The gold film was gradually removed by argon ion sputtering. The hardness is influenced by the depth from the film surface and is also related to the composition gradient between the ion-plated gold and the substrate. As one might expect, the microhardness of the film increased with increasing depth from the surface because the load borne by the hard graded interface and the substrate increases as the film becomes thinner, and deformation accordingly decreases during indentation (15). Furthermore, in the graded interface, the hardness at first increased slightly with increasing depth. The graded interface exhibited the highest hardness. Below a certain depth (about 3 μm in Fig. 5(a)) in the graded interface, the hardness decreased with increasing depth and gradually approached the hardness of the nickel substrate. The behavior of hardness in the graded interface is due to an alloy hardening effect (16, 17).

Fig. 5(b) presents the microhardness of vapor-deposited gold films and nickel substrates as a function of depth from the surface of the film. The gold film surface had the lowest hardness value. The hardness of the gold film increased with increasing depth from the surface. The hardness of the substrate, after the gold film had been removed by argon ion sputtering, was almost the same as that of the uncoated bulk nickel substrate. No graded interface between the vapor-deposited film and substrate is shown in Fig. 5(b).

Friction and Wear Under Adhesive Conditions

Friction. - Single-pass sliding friction experiments were conducted with gold ion-plated on nickel and with bulk gold (solid) flats in sliding contact with metal and nonmetal pin specimens in vacuum at a pressure of 30 nPa. Friction force traces resulting from sliding in vacuum were generally characterized by randomly fluctuating behavior.

The coefficients of friction measured in vacuum are presented in Fig. 6. They are strongly influenced by material combinations as well as film thickness. Fig. 6(a) shows that gold exhibits the highest adhesion and friction in sliding contact with itself. The adhesion and friction for nickel-gold contacts are higher than those for iron-gold contacts. This difference in friction may be due to the compatibility or affinity of the material pairs (18-21). The nonmetallic materials, aluminum oxide and silicon carbide, in contact with gold had relatively lower adhesion and friction than did metal-to-gold contacts.

The adhesion and friction strongly depend on the thickness of the gold film, as indicated in Fig. 6. The coefficient of friction for a film 0.6 μm thick is generally lower than that for a film 0.1 μm thick. On the basis of coefficient of friction for a gold pin on an ion-plated gold flat and a bulk gold flat (Fig. 6(a)) and a silicon carbide pin on an ion-plated gold flat and a bulk gold flat (Figs. 6(a) and (b)), the variation of film thickness can be divided into two regions. These are an ultra-thin-film region and a thin-film region with an effective or critical film thickness at which the coefficient of friction reaches a minimum value.

In the ultra-thin-film region (up to 0.6 μm in Fig. 6(b)) the coefficient of friction increases for thicker films because the larger apparent contact area decreases the load-carrying capacity of the substrate surface.

Effect of substrate on friction. - Single-pass sliding friction experiments were conducted with the surfaces of clean ion-plated gold films, graded nickel-gold interfaces, and nickel substrates sliding against a clean 0.79-mm-radius silicon carbide spherical pin in vacuum at a pressure of 30 nPa. Friction force traces resulting from sliding in vacuum were generally characterized by stick-slip with fluctuating behavior. Fig. 7(a) presents the coefficient of friction as a function of the depth from the film surface for ion-plated gold films, graded nickel-gold interfaces, and nickel substrates. The results of XPS elemental analysis and depth profiling are also schematically summarized in Fig. 7 with a structural model. The coefficient of friction

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for a film 0.6 μm thick was lower than that for a surface that was ion etched about 0.5 μm from the ion-plated surface.

At the graded interface the coefficient of friction generally increased because of the presence of the substrate element relative to that obtained for the ion-plated gold film. The high friction is associated with high adhesion at the graded interface caused by the raising of the Peierls stress and an increase in lattice friction stress, which result in resistance to shear fracture of the cohesive bonding in the alloy of the graded interface by nickel atoms of the substrate. The present observation is consistent with the authors' earlier work (22). In the nickel substrate region the coefficient of friction was considerably lower than that for the graded interface. The coefficient of friction generally decreases markedly with the absence of the ion-plated gold.

Fig. 7(b) presents the coefficients of friction for the surfaces of clean vapor-deposited gold films and nickel substrates sliding against a clean 0.79-mm-radius silicon carbide hemispherical pin in vacuum at a pressure of 30 nPa. Inspection of the wear track on the vapor-deposited gold film clearly revealed breakthrough of the film in the contact area caused by the sliding action. The breakthrough of the film induced direct contact of the silicon carbide pin with the nickel substrate.

Fig. 8 shows the influence of the substrate on friction for gold films ion-plated and vapor deposited on copper, nickel, and 440C stainless steel substrates in contact with a 0.79-mm-radius hemispherical silicon carbide pin in vacuum. With ion-plated gold films (Fig. 8(a)) the coefficient of friction was lower with 440C stainless steel than with copper or nickel because gold films on 440C stainless steel have higher load-carrying capacity (namely, greater hardness).

With vapor-deposited gold films (Fig. 8(b)) the breakthrough of the gold film on 440C stainless steel induced direct contact of the silicon carbide pin with the substrate and resulted in a higher coefficient of friction. With the metal substrates (Fig. 8(c)) the coefficient of friction was generally higher with 440C stainless steel than with the pure metals copper and nickel. The coefficients of friction for vapor-deposited gold films correlated strongly with those for the substrates themselves (Figs. 8(b) and (c)).

Wear and transfer. - Inspection of the ion-plated gold films after single-pass sliding contact with metals and nonmetals in vacuum revealed wear and transfer of gold to the mating surfaces. All the metal and nonmetal pin specimens that had contacted the ion-plated gold in this investigation contained the element gold in their surfaces. This occurred with a single pass of the pin specimen.

Fig. 9 presents scanning electron micrographs of the wear track on an ion-plated gold film and the wear scar on a nickel pin specimen generated by a single pass of sliding. It is obvious that copious amounts of gold transferred to the nickel and that backtransfer of gold to gold occurred even with single-pass sliding. The wear of gold and its transfer to nickel were however, much less than for gold-to-gold contacts. Gold exhibited high adhesion and friction when it was brought into contact with itself. This resulted in a large amount of wear and subsequently a considerable amount of transfer and backtransfer (10). With the iron-to-gold contact, the wear of gold and its transfer to iron were slightly lower than for nickel-to-gold contacts. Silicon carbide and aluminum oxide pins caused the least amount of wear and transfer of the gold film. The wear tracks on the gold surfaces sliding against the nonmetals were smooth and exhibited considerably lower friction than was observed with the metals (10).

Wear tracks on the gold surface in sliding contact with metals and nonmetals in argon atmosphere were very smooth, and much less gold transferred to the mating surfaces in vacuum (10).

Friction and Wear Under Abrasive Conditions

Friction and wear. - Sliding friction experiments were conducted with gold both ion plated and vapor deposited on nickel substrates in contact with a 0.025-mm-radius, spherical silicon carbide pin with a mineral oil lubricant at loads of 0.05 and 0.1 N. The sliding action resulted in permanent grooves in the gold films and in the metal substrates with deformed metal piled up along the sides of the grooves, as typically shown in Fig. 10. Under these conditions the friction is due primarily to plowing of the gold film and the substrate. The friction-force traces obtained in this investigation were characterized by randomly fluctuating behavior with no evidence of stick-slip.

Fig. 11(a) presents the coefficients of friction for an ion-plated gold film, a graded interface, and a nickel substrate in contact with a silicon carbide pin lubricated with mineral oil. The coefficient of friction initially decreased as the depth from the film surface increased. The gold film had the highest coefficient of friction; and the graded interface, the lowest. However, in the substrate region the coefficient of friction increased slightly. The trend of these data indicates that the coefficient of friction is inversely related to hardness and directly related to the apparent contact area during sliding. The greater the hardness of the metal surface, the lower was the coefficient of friction. This is consistent with the authors' earlier work (16).

Fig. 11(b) presents the coefficients of friction for gold vapor deposited on nickel and for the nickel substrate as a function of the depth from the film surface at a load of 0.1 N. Again, the coefficient of friction is inversely related to the hardness, or the load-carrying capacity, of the vapor-deposited gold surface and the substrate.

Effect of substrate on friction. - To examine the influence of hardness on friction with various substrates, additional sliding friction experiments were conducted with gold films ion plated and vapor deposited on copper, nickel, and 440C stainless steel substrates in contact with a 0.025-mm-radius, spherical silicon carbide pin lubricated with mineral oil. The coefficients of friction are presented as a function of hardness measured at normal loads of 0.1 and 3 N in Fig. 12. The coefficients of friction correlate with the hardness of the gold film on the substrate, and the relationship is a linearly decreasing one.

CONCLUSIONS

The following conclusions were drawn from the data presented herein on the tribological characteristics of gold films deposited on metals by ion plating and vapor deposition:

1. The solubility of material pairs controls the depth of the graded interface between an ion-plated gold film and a metal substrate. With gold and nickel the graded interface can be an alloy of the metals. Thermal diffusion and chemical diffusion mechanisms are thought to be involved in the formation of the gold-nickel interface. In gold-iron graded interfaces the gold is primarily dispersed in the iron and thus forms a physically bonded interface.

2. The hardness depth profile for gold ion-plated on a metal substrate indicated that the hardness is influenced by the depth from the surface of the gold film. The graded interface exhibited the highest hardness because of an alloy hardening effect. No graded interface was detected between the vapor-deposited gold film and the substrate.

3. The effects of film thickness on adhesion and friction were established. A minimum in the coefficient of friction was found in the thin-film region.

4. The friction related to adhesion and that related to abrasion are influenced by the depths of the graded interface and the film. The surface of a gold film exhibited the lowest coefficient of friction in the adhesive process. The highest coefficient of friction due to abrasion was inversely related to hardness. The harder the surface, the lower was the abrasion and friction.

5. No graded interface was detected between the vapor-deposited gold film and the substrate.

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TABLE 1. - MINERAL OIL PROPERTIES[a]

Chemical type	Naphthenic mineral oil
Viscosity, m^2/s(cS):	
At 38° C	73.4×10^{-6} (73.4)
At 99° C	8.35×10^{-6} (8.35)
Specific gravity:	
At 16° C	0.880
At 25° C	0.875
Pour point, °C	-18
Flashpoint, °C	224

[a]Manufacturer's analysis.

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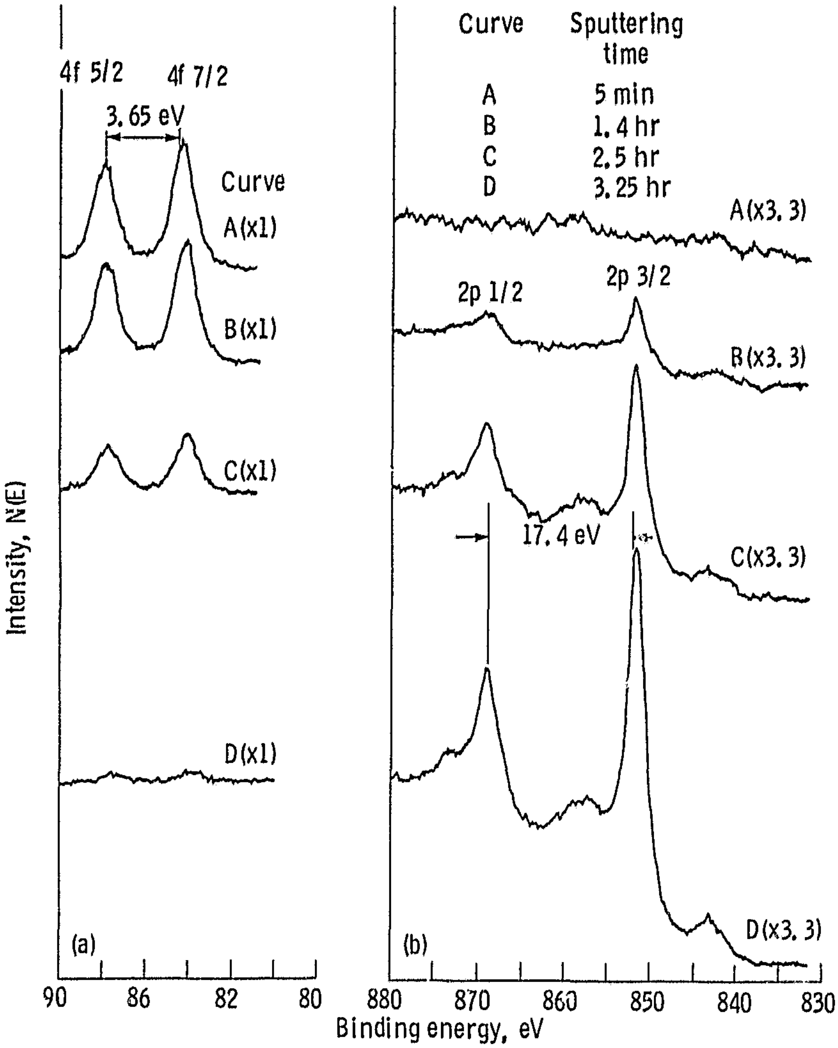

(a) Au 4f.
(b) Ni 2p.

Figure 1. - Representative Au 4f and Ni 2p SPS peaks for gold ion plated on nickel after various sputtering times. Film thickness, 0.6 μm.

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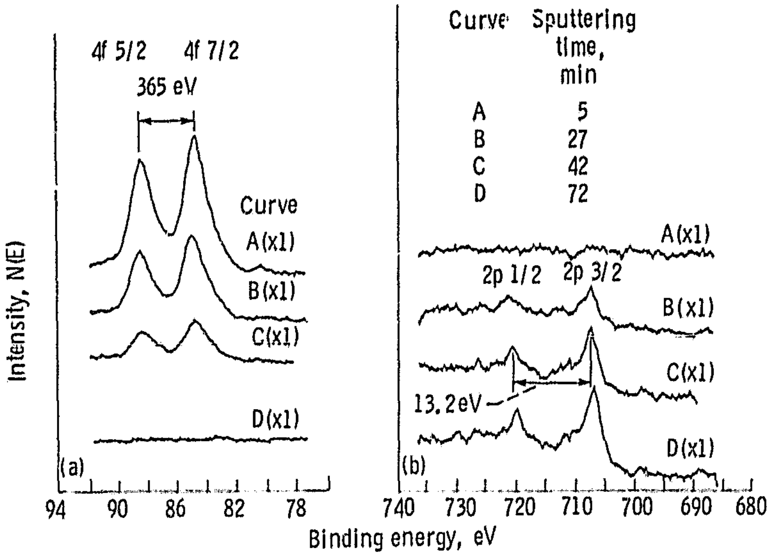
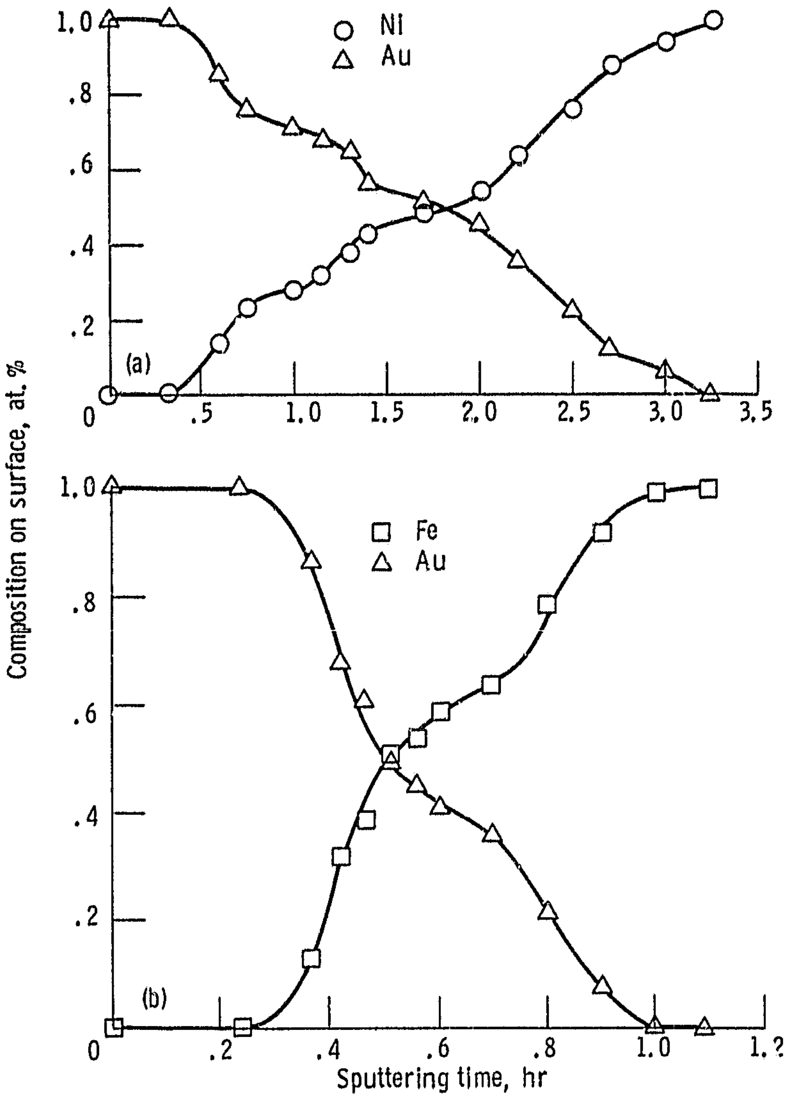


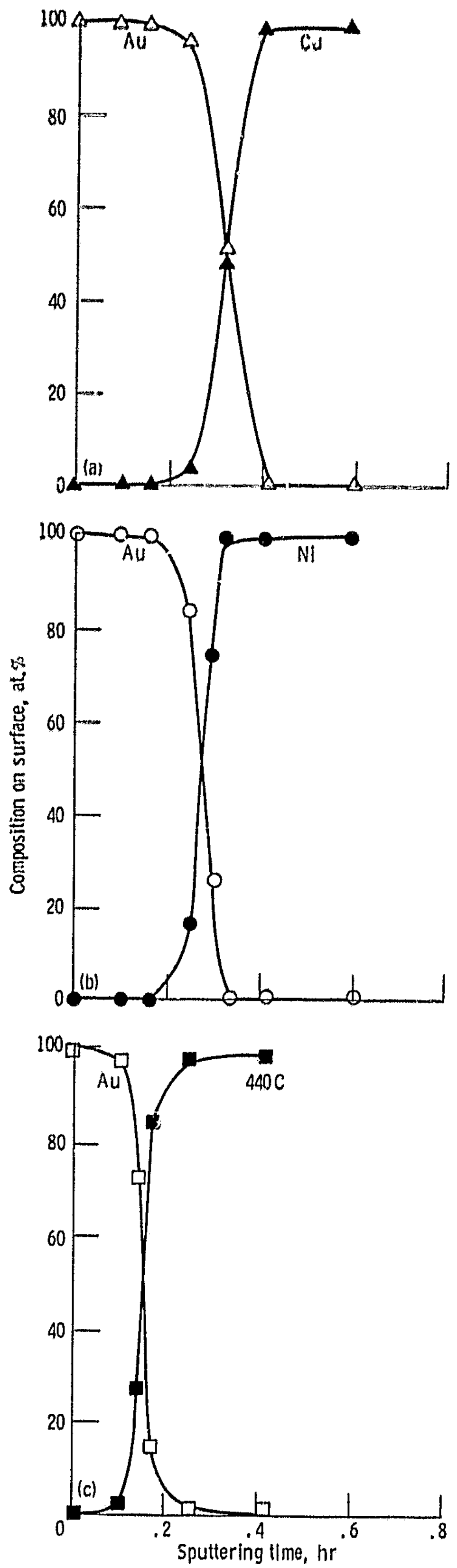
(a) Au 4f.
(b) Fe 2p.

Figure 2. - Representative Au 4f and Fe 2p XPS peaks for gold ion plated on iron after various sputtering times. Film thickness, 0.6μm.



(a) Nickel substrate.
(b) Iron substrate.

Figure 3. - Elemental depth profiles for gold ion plated on nickel and iron. Film thickness, 0.6μm.



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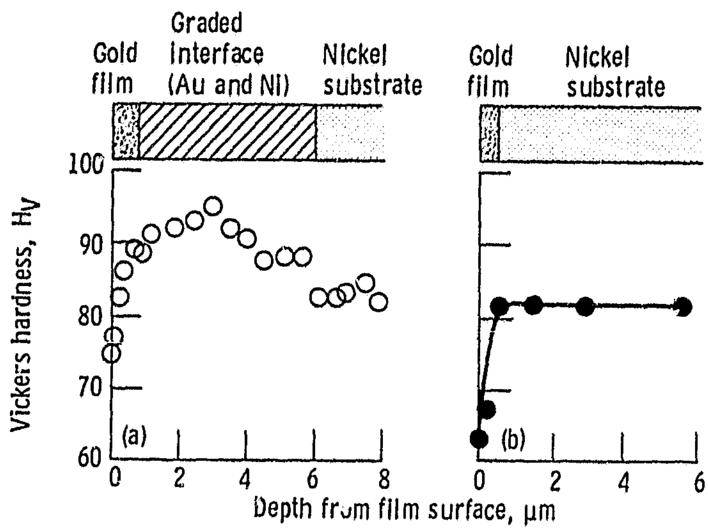
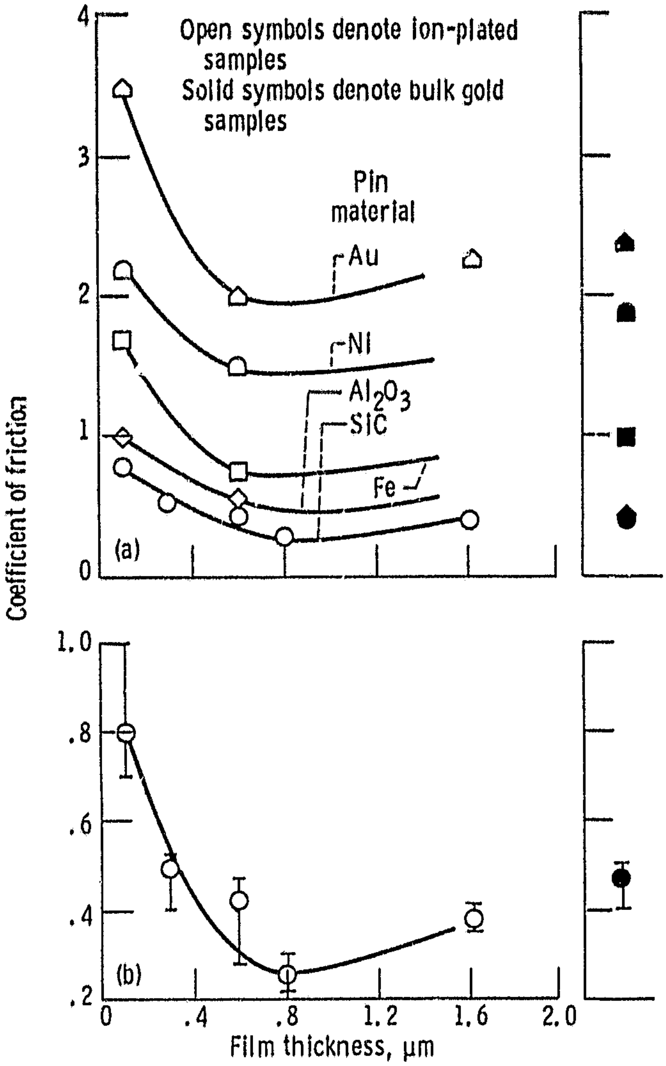
(a) Copper substrate.
(b) Nickel substrate.
(c) 440C substrate.

Figure 4. - Elemental depth profiles for gold vapor deposited on copper, nickel, and 440C stainless steel. Film thickness, 0.3 μm.



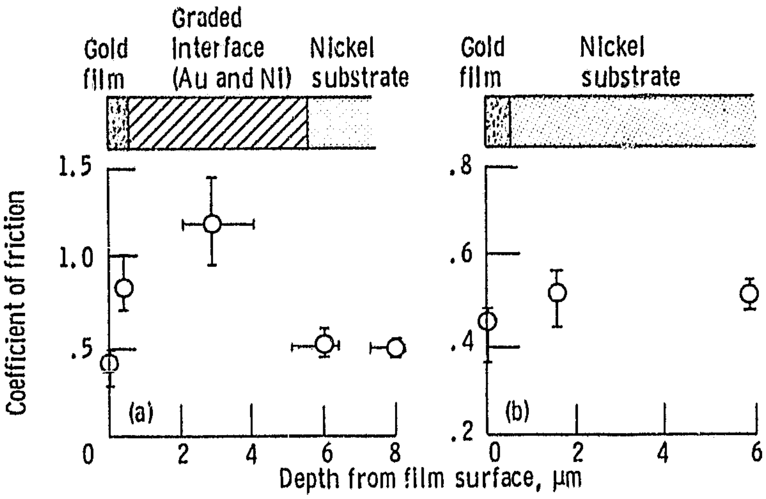
(a) Ion-plated gold film.
(b) Vapor-deposited gold film.

Figure 5. - Hardness depth profiles for gold ion plated and vapor deposited on nickel substrate. Hardness measuring load, 0.1 N.



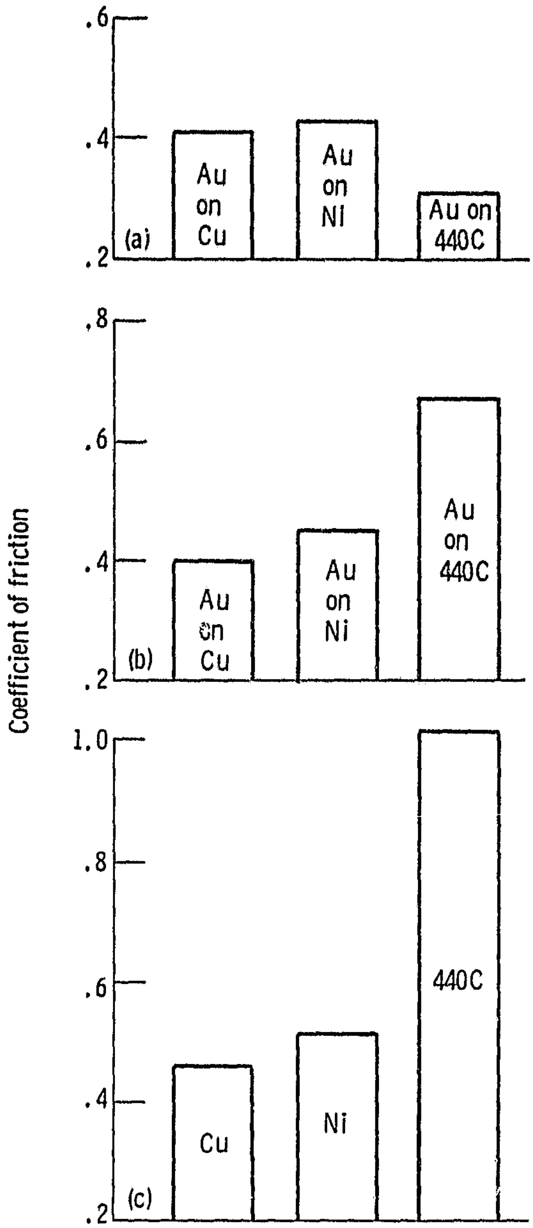
(a) Metal and nonmetal pins.
(b) Silicon carbide pin.

Figure 6. - Coefficient of friction as function of film thickness for single-pass sliding of gold, nickel, iron, aluminum oxide and silicon carbide pin specimens on gold surfaces ion plated on nickel and on bulk gold surfaces in ultrahigh vacuum. Pin radius, 0.79 mm; Sliding velocity, 5×10^{-5} ms^{-1}; load, 0.1 to 0.2 N; vacuum pressure, 30 nPa; temperature, 23° C.



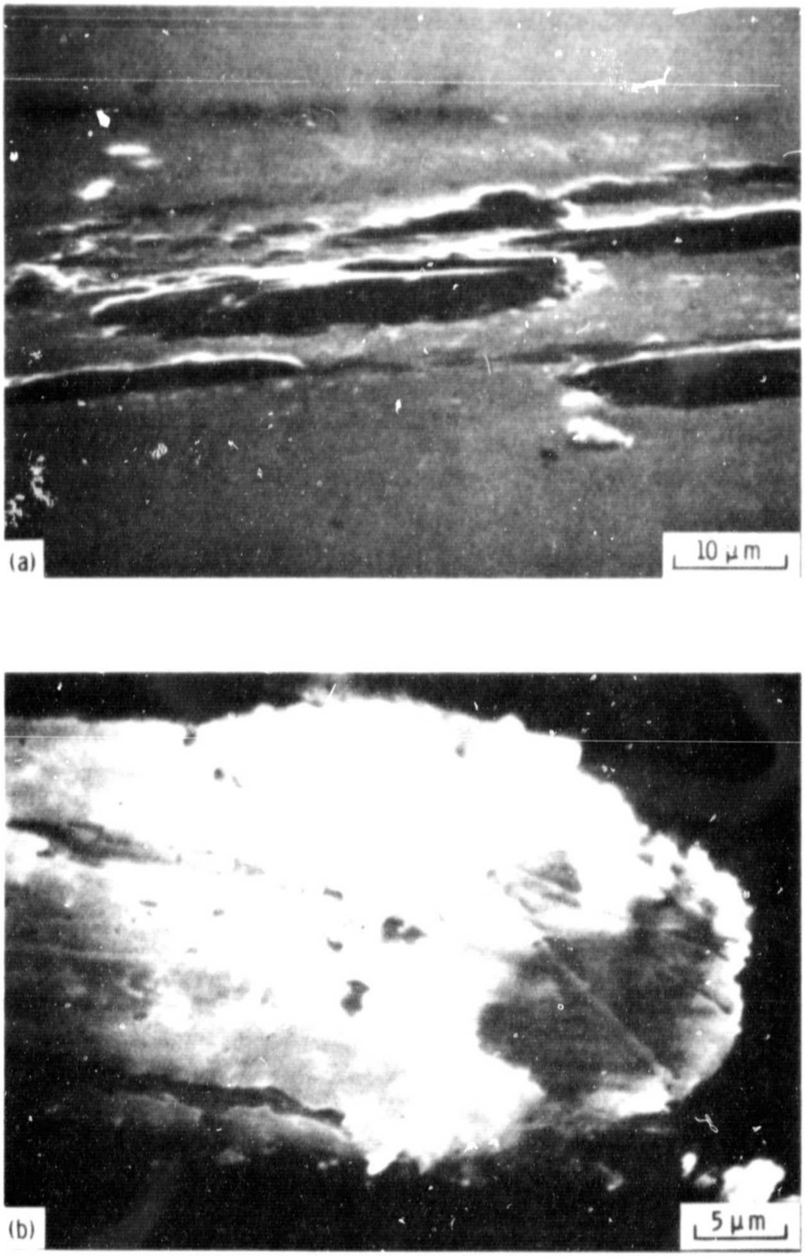
(a) Ion-plated gold film.
(b) Vapor-deposited gold film.

Figure 7. - Coefficient of friction as function of depth from film surface for single-pass sliding of silicon carbide pin specimen on gold surfaces ion plated and vapor deposited on nickel in ultrahigh vacuum. Pin radius, 0.79 mm; sliding velocity, $5x10^{-5}$ ms^{-1}; load, 0.2 N; vacuum pressure, 30 nPa; temperature, 23^{0} C.



(a) Ion-plated gold film.
(b) Vapor-deposited gold film.
(c) Uncoated metal substrates.

Figure 8. - Coefficient of friction for gold ion plated and vapor deposited on copper, nickel, and 440C stainless steel and for uncoated copper, nickel, and 440C stainless steel in contact with SiC hemispherical pins in ultrahigh vacuum. Pin radius, 0.79 mm; sliding velocity, 5×10^{-5} ms^{-1}; load, 0.1 to 0.2 N; vacuum pressure, 30 nPa; temperature, 23^{0} C.



(a) Wear track on ion-plated gold surface on nickel.

(b) Wear scar on nickel pin specimen.

Figure 9. - Scanning electron micrographs of wear tracks produced during single-pass sliding of nickel pin specimen in ultrahigh vacuum. Sliding velocity, $5x10^{-5}$ ms^{-1} ; load, 0.2 N ; sliding distance, 3 mm ; vacuum pressure, 30 nPa ; temperature, 23^{0} C ; film thickness, 0.6 μm.

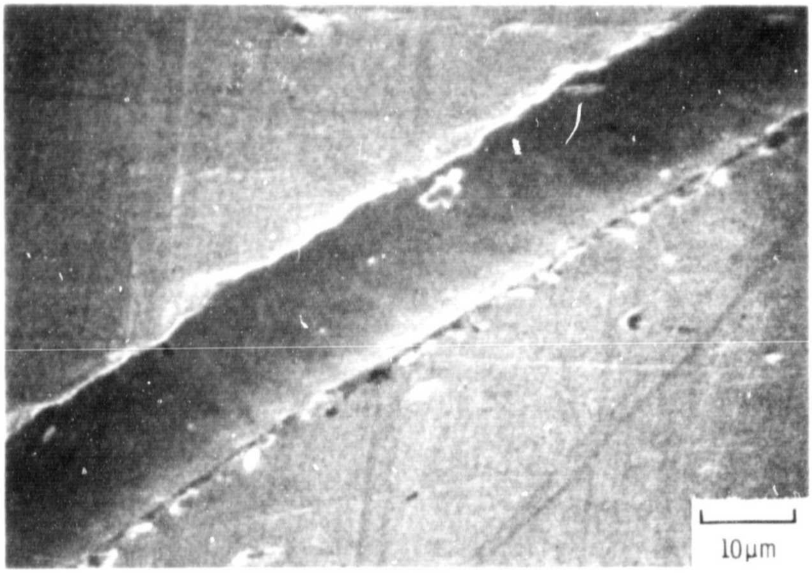
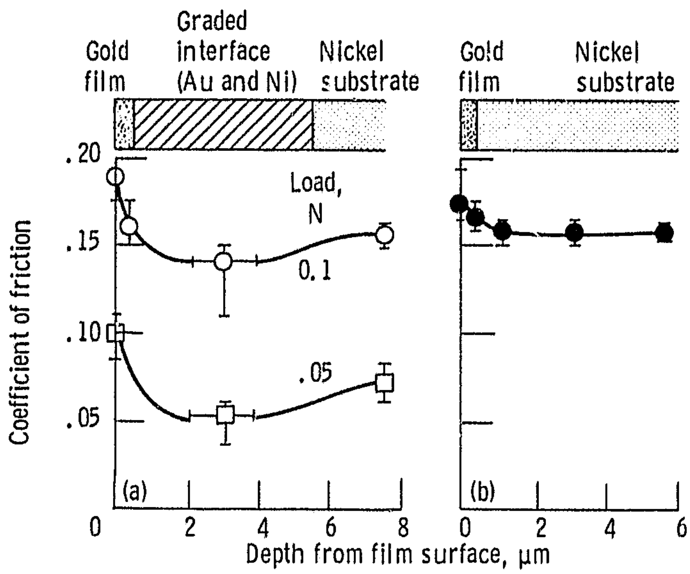


Figure 10. - Scanning electron micrograph of wear track on gold surface ion plated on nickel. Single pass of 0.25-mm-radius hemispherical pin; lubricant, mineral oil; sliding velocity, 7×10^{-4} ms^{-1}; load, 0.1 N; film thickness, 0.6 μm.



(a) Ion-plated gold film (0.6 μm thick) on nickel substrate. Load, 0.1 and 0.05 N.
(b) Vapor-deposited gold film (0.3 μm thick) on nickel substrate. Load, 0.1 N.

Figure 11. - Coefficient of friction as function of depth from film surface for single-pass sliding of silicon carbide pin specimen on gold surfaces ion plated and vapor deposited on nickel. Pin radius, 0.025 mm; lubricant, mineral oil; sliding velocity, $7x10^{-4}$ ms^{-1}.

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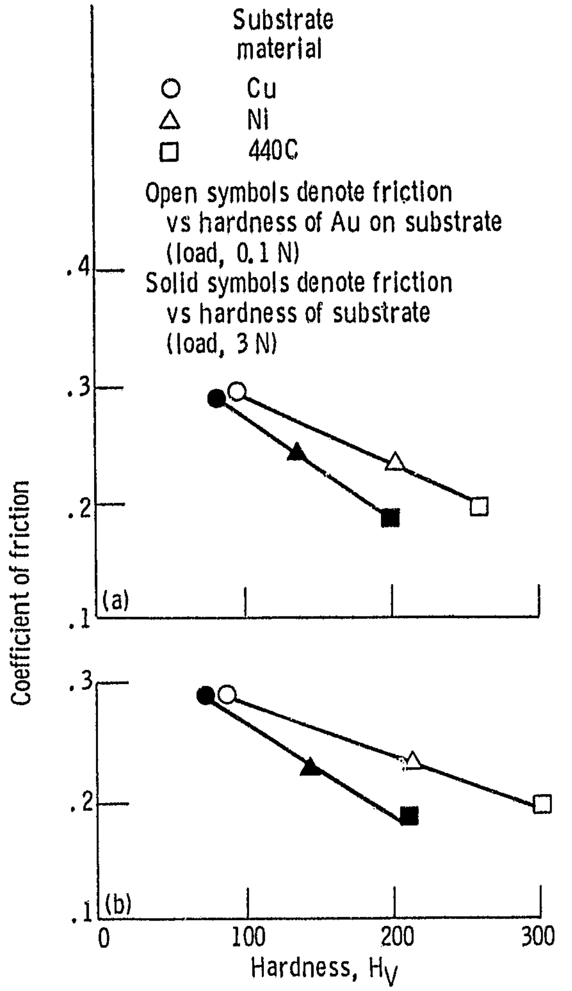

(a) Ion-plated gold. Film thickness, 0.6 μm.
(b) Vapor-deposited gold. Film thickness, 0.3 μm.

Figure 12. - Coefficient of friction for gold ion plated and vapor deposited on nickel, copper, and 440C stainless steel as function of hardness. Single-pass sliding of 0.025-mm-radius hemispherical pin; lubricant, mineral oil; sliding velocity, $7x10^{-4}$ ms^{-1}; load, 0.2 N.