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VAPOR DEPOSITED GOLD THIN FILMS AS LUBRICANTS IN VACUUM (10^{-11} mm Hg)

by T. Spalvins and D.H. Buckley
Lewis Research Center
Cleveland, Ohio

TECHNICAL PREPRINT prepared for Twelfth Annual Vacuum Symposium sponsored by the American Vacuum Society
New York City, September 29 - October 1, 1965

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23

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ABSTRACT

Gold thin films of 1800 A° to be used as lubricants were vapor deposited on Ni, Ni-Cr and Ni-Re substrates. Strong bonding (adhesion) and durability between the film and substrate were found to be essential when thin films are used as a lubricant. Factors which were investigated included the selection of the film and substrate material. Strong durablilty of the thin film is directly related to the type and structure of the interfacial region. Two methods of substrate preparation prior to vapor deposition were investigated: (1) mechanically polished surface and (2) electron bombarded surface. Gold was vapor deposited on the mechanically polished surface at room temperature and on the thermally etched surface at an elevated temperature approximatly (800°F). Strength and durability of the films were investigated in sliding friction experiments with a hemispherical niobium rider sliding on the films at a velocity of 5 feet per minute. Results obtained in these friction experiments indicated that the film endurance life was considerably better on the thermally etched surface. This increased film durability with the thermally etched surface is believed to be due to the formation of a diffusion type interface between the film and the substrate. Due to the disregistry at grain boundaries, a higher rate of diffusion and preferential trapping in and around the grain boundaries occurs, with these regions acting as "lubricant reservoirs" during the friction experiments.

INTRODUCTION

Thin, soft metal films may be used as lubricants in outer space for rotating or sliding components to reduce the coefficient of friction and to eliminate complete seizure of the moving parts. Previous experiments have shown that thin metal films deposited conventionally have very limited operational lives because of the weak adhesion of film to substrate (ref. 1). This weakly bonded film, when brought into sliding contact with another surface, is subject to easy rupturing, and thus its use as a lubricant is limited.

References 2 to 4 describe in great detail the deposition parameters for thin-film preparation, mostly on glass substrates, with special emphasis on the structural, mechanical, optical, and magnetic properties of the thin film. Less consideration has been given to vapor thin films vapor-deposited on metal substrates. The objective of this investigation was to study these vapor-deposited thin films and to obtain strong bonding (adhesion) between film and substrate. Strong bonding is essential for film durability in lubrication. Two factors were considered: (1) material (both film and substrate) selection and (2) preparation of the substrate material.

The substrate and the film materials were selected on the basis of solid-solubility principles (ref. 5) and thermal-expansion coefficients. If two materials are mutually soluble, diffusion and alloying take place at elevated temperatures, and, if the surface is atomically clean, vacuum deposition can then result in adherent films (ref. 6).

Adhesion of a vapor-deposited metal film to a substrate depends on the type and structure of the interfacial region and the nature of the bonding forces across this region (ref. 6). Hence, surface cleaning was investigated in order to achieve strong bonding (adhesion) between the substrate and the film.

The friction characteristics of vapor-deposited 1800-A^{o} gold films on nickel, nickel - 10-percent-chromium, and nickel - 5-percent-rhenium substrates were determined under ultra-high-vacuum conditions (10^{-11} mm Hg). The experiments were conducted with a hemispherical rider of niobium (which is slightly miscible with gold or nickel) sliding on the thin film. The rider was run at a load of 250 grams, a speed of 5 feet per minute, and ambient temperature.

APPARATUS

Vacuum Vapor-Deposition Apparatus

The vacuum vapor-deposition apparatus in figure 1 basically consists of a commercial evaporator unit with an 18-inch-diameter by 30-inch-high bell jar. A number of modifications were incorporated inside the bell jar for proper specimen mounting, inversion, electron bombardment, vapor deposition, and pumpdown to lower pressures.

The $2\frac{1}{2}$ inch diameter disk specimen is mounted on a stainless-steel specimen holder through which a circular rod is inserted for support and inversion. This inversion gives a proper positioning of the disk for electron bombardment and vapor deposition.

The specimen-mounting assembly has a fixed adjustment; that is, when the disk is positioned for electron bombardment, it is always 3 inches from the electron gun, and when the disk is inverted for vapor

deposition, it is of 3.5 inches from the filament. The water-cooled electron gun is located directly above the specimen disk and is controlled by a power supply. The filament is constructed from two 0.023-inch molybdenum wires, 2 inches in length and interwound. The evaporating material used is a 3-inch-long, 0.020-inch-diameter gold wire of 99.999-percent purity. This gold wire is wound around the molybdenum filament, which is inserted in the filament holders. Disk specimen temperature is measured by two Chromel-Alumel thermocouples placed inside the specimen holder and is then recorded by a temperature recorder.

Before vapor deposition, a movable shield is placed in front of the substrate so that both filament and gold can be well degassed before any material is deposited on the substrate.

In addition to the mechanical pump, the three-stage oil diffusion pump and the liquid-nitrogen baffle that are part of the usual vacuum system, two additional pumping systems were introduced: (1) a stainless steel dome that was placed just below the top of the bell jar and functioned as a cryopump while being cooled by liquid nitrogen and liquid helium, and (2) a liquid-nitrogen-cooled titanium sublimation pump. The pressure was measured with a nude, hot-cathode ionization gage, and the vacuum was approximately 10^{-8} millimeter of mercury.

Ultra-High-Vacuum Friction Apparatus

The vacuum-friction apparatus (fig. 2) was used for determining the coefficient of friction for the coated and uncoated surfaces. The apparatus has two distinct chambers, the specimen chamber and the bearing chamber, both of which are connected to the forepumping system.

The forepumping system of the apparatus consists of a cold trap which is made up of molecular sieves backed by liquid-nitrogen-cooled containers. This system is connected to two mechanical pumps through a 2-inch stainless-steel vacuum valve.

The specimen chamber, which is connected to the mechanical pumping system by a bakable high-vacuum valve, is provided with an ion pump (400-liter-per-second), as well as cryopumping surfaces (liquid nitrogen and liquid helium). The specimen chamber contains a cold-cathode (Kreisman) vacuum gage for measuring pressures. The pressure in the specimen chamber is approximately 10^{-11} millimeter of mercury. The specimen and bearing chambers are bakable at 700° and 400° F, respectively.

The bearing chamber, which is connected to the forepumping system by a 2-inch valve, is equipped with an ion pump (125-liter-per-second), which is placed in operation only after the mechanical pumping system has reduced the chamber pressure to about 10^{-4} millimeter of mercury. The bearing chamber also contains a liquid-nitrogen-cooled titanium sublimation pump. The combination of pumps is used to pump the bearing chamber to about 10^{-10} millimeter of mercury in preparation for an experiment.

The rotating shaft, upon which the $2\frac{1}{2}$-inch disk specimen is mounted, is supported on bearings in the bearing chamber. The shaft support bearings have a large clearance and are mounted in a cartridge so that the shaft expansion takes place into the test chamber and the possibility of the magnet striking the diaphragm is eliminated. Since loading was applied by deadweights, the expansion did not change the load on the specimens. The bearing cartridges are water cooled to prevent any

damage to the bearings during the bakeout cycle.

The rotating shaft projects through the rear wall of the test chamber first and then through a molecular flow seal (fig. 2). On the bearing-chamber end of the shaft is mounted a 20-pole magnet, which is separated from a similar magnet, outside the vacuum chamber, by a 0.030-inch diaphragm (0.160-in. air gap). The drive magnet, outside the chamber, is powered by a hydraulic motor with a variable speed capability of 4000 rpm (sliding velocity at the friction specimens, 2000 ft/min). Because of instabilities in the drive motor at low speeds (10, 20, and 100 rpm), these speeds were obtained by utilizing a speed reducer with a ratio of 10.

The disk specimen is mounted on the end of the horizontal shaft in the test chamber. Against the disk, a 3/16-inch hemispherical rider specimen is loaded. The rider is held in place by a rigid arm, which projects through a port in the side of the vacuum chamber. The seal is made at the wall by utilizing a bellows connection between the chamber wall and the rigid arm. A removable gimbal assembly, which is used to load the rider against the disk surface and to monitor the frictional force through a strain-gage assembly, is fastened to the rigid arm outside the vacuum chamber.

PROCEDURE

Specimen Preparation and Mechanical Polishing of Surfaces

The nickel disks used in this investigation were prepared from electrolytic nickel. Small nickel slugs were placed into a zirconium oxide crucible and the crucible was placed in an induction vacuum furnace which was then evacuated. After evacuation, the furnace was filled

with argon and the metal was melted. Once molten, the liquid nickel was poured into a copper mold and cooled to room temperature. The nickel-alloy disks used in this investigation were prepared in a similar manner. Afterward, the castings were machined to the required dimensions. Before the disk specimen was mounted the high-vacuum evaporation apparatus for electron-bombardment cleaning and vapor deposition, the disk was mechanically ground with 400- and 600-grade emery papers. It was then finished with levigated alumina on a lapping wheel.

Once the surface was micropolished, vacuum deposition was preformed at two temperatures on two surfaces: (1) at room temperature, on a micropolished surface which had not been cleaned by electron bombardment, and (2) at 800° F, on a micropolished surface which had been cleaned by electron bombardment.

Thermal Etching

The substrate surface was cleaned under high-vacuum conditions (10^{-6} to 10^{-7} mm Hg) by electron bombardment. During bombardment, the disk was kept at ground potential and served as the anode; the electrons were supplied by an annular tungsten cathode, which was kept at applied voltages of 3.5 to 4.5 kilovolts and a beam current of 70 to 80 milliamperes. The electron-bombardment cleaning was performed for approximately 30 to 45 minutes, and the disk temperature was in the range 1100° to 1200° F. Electron bombardment can be used for both cleaning the surface and heating the substrate material, if the electron beam is kept on continuously.

During electron bombardment the surface is cleaned by dissociation of the contaminants (i.e., adsorbed gases or oxides)(ref. 4). Considerable

energy is then liberated, the substrate temperature is increased and surface topography is changed. This thermal process, known as thermal etching (refs. 7 and 8), develops grooves where the grain boundaries intersect the surface and produces selective etching on the faces of individual grains. This selective-etching effect occurs because different planes and grain boundaries have different surface energies (ref. 9). Grain boundaries have a relatively high surface energy with respect to the crystals and they do not have the normal coordination number. Therefore, grain boundaries are the preferential sites for thermal etching and solid-state reactions (ref. 7). It is believed (ref. 9) that evaporation (either of metal or of oxide) appears to be the dominant mechanism in the formation of the etched structure.

Vapor Deposition

After thermal etching, the electron-beam intensity was gradually reduced, and thus the temperature of the disk was lowered. Once a temperature of approximately 800° F was reached, the electron beam was cut off, and the disk was inverted for vapor deposition of gold. The rate of evaporation depended on the filament current. A current of about 35 to 40 amperes was passed through the filament until the temperature was high enough to melt the gold wire and thus wet the molybdenum filament. Once this was achieved, evaporation was allowed for 2 minutes. The thickness of the films deposited was kept at approximately 1800 Å. After deposition, film thickness was measured with an interference microscope.

RESULTS AND DISCUSSION

The strength and the durability of thin films are determined largely be the degree of adhesion. Adhesion of a vapor deposited metal film to a metal substrate depends on the formation of an alloyed surface. Very weak adhesion between the film and the substrate occurs when the substrate surfaces are not properly cleaned. The gold films deposited on the "uncleaned" surfaces of nickel, nickel-chromium, and nickel-rhenium disks were poorly adherent, as was shown by the scotch tape test and by the friction test. In the first test, if adhesion was poor, the film adhered to the tape rather than to the substrate. This test, although qualitative, gave a good comparison between films with good and poor adhesion.

Figure 3 shows typical thermally etched copper surfaces. Copper is a particulatly suitable metal with which to illustrate the general phenomenon of thermal etching since it etches readily in vacuum during electron bombardment. If figures 3(b) and (c) are compared, it can be seen that, when the intensity and the duration of the electron beam bombardment are increased, thermal etching will occur, particularly at the grain boundaries. The micrograph of figure 3(a) was taken at a higher magnification to illustrate the thermal faceting on the various grains.

The groove formation at the grain boundaries is illustrated in figure 4. The groove formed can be measured interferometrically (refs. 10 and 11) by the boundary groove angle θ. The angle θ depends not only on the temperature and time of thermal etching, but also on the orientation

of the planes at the particular grain boundary. Since it is believed that evaporation (either of metal or of oxide) is the dominant mechanism for the formation of the etched surface in vacuum (ref. 9), surface cleaning must take place at the same time. Figures 5(a) and (b) show etched surfaces of a nickel - 10-percent-chromium alloy disk which was etched for 5 and 10 minutes, respectively. As the time was increased, the etching became more selective at the grain boundaries.

The micrographs were taken after vapor deposition of gold on the thermally etched nickel - 10-percent-chromium alloy surface (fig. 5(c)). The preferential accumulation of gold in and around the grain boundaries which is indicated can be attributed to the fact that, during vapor deposition, the impinging gold atoms migrate on the surface because they have kinetic energy and accumulate in the grooves of the grain boundaries (ref. 12). Another factor is the elevated temperature of the substrate (approximately 800° F), which enhances the surface migration of the impinging atoms. This phenomenon indicates that grain boundaries may act as trapping sites and become saturated with deposited atoms.

Since nickel and gold are mutually soluble (ref. 13) and deposition is performed at elevated temperatures, diffusion takes place. It is believed that the strong bonding found between the substrate and the film on the electron-bombarded surfaces is due to the cleanliness of the surface and to the formation of a diffusion type of interface. The diffusion-type interface is characterized by a gradual change in composition across the interfacial region (ref. 6) rather than by a sharp boundary between the film and the substrate. This was also confirmed by an

electron-beam microprobe on the gold-deposited nickel alloy disks used in this investiation. The gold diffusion was detectable in the bulk to a depth of about 2700 Å. As is well known, the rate and depth of diffusion are dependent on both temperature and time. As already described, the time and the temperature for diffusion are relatively short.

Intimate contact between the film and the substrate is essential for this diffusion bonding. A vacuum deposited film, however, is in good contact with the substrate. The diffusion rate of gold is not uniform over the entire etched surface. At the grain boundary, the average distance between the atoms is somewhat larger than in the perfect crystal, and therefore a disregistry is formed at the grain boundaries. Because of the presumably "open structure" of the grain boundaries, the rate of diffusion is higher there than it is through crystals (ref. 14). In addition, it has been found that grain-boundary diffusion depends on orientation difference across the boundary, diffusion being least for small orientation differences (ref. 15). Considering the preferential etching of grain boundaries, the preferential saturation around the grain boundaries of the impinging atoms, and the higher rate of diffusion through the grain boundaries, it can be said that grain boundaries act as "reservoirs" for the deposited material.

Very few quantitative studies of the effects of solute on grain boundary diffusion have been done. Some qualitative results have been published in the literature (ref. 16 and 17). An investigation had been conducted with silver diffusing into polycrystalline copper alloys. It

was observed that the alloying elements in copper had decisive effects on the rate of silver diffusion along the grain boundaries. According to the above investigators, a faster rate of silver diffusion was observed along the copper-alloy grain boundaries. From the solid solubility principles the gold-nickel combination used in this investigation would be similar to the copper-silver combination and, should, therefore, reveal similar effects. An electron-beam microprobe study was performed on the gold-nickel specimens of this investigation to differentiate between diffusion in the grain boundaries and diffusion in the grains. By the use of the absorbed electron method, one grain boundary, in one instance, appeared to have a higher gold concentration than the adjacent matrix; further attempts on other grain boundaries failed to show such an affect, however. The difficulties are probably the same as those that beset other methods: the large beam width, which activates a relatively large area and gives inadequate resulution; and interference from other elements in the specimen, which presents standardization problems.

The friction curves for the gold film on pure nickel (fig. 6(a)) indicate that the films had relatively short durability. The coefficient of friction did not remain at a constant low value but steadily rose to higher values. This continuous rise of the coefficient of friction revealed that pure nickel was a poor friction material and that not even the gold films on a clean surface could afford protection. This might be attributed to the fact that pure nickel is relatively soft (Brinnel 75) when compared with nickel alloys which have a higher hardness (Brinell 98-103).

A comparison of gold-film strength and durability on thermally etched (cleaned) and unetched (uncleaned) surfaces for nickel alloys (figs. 6(b) and (c)) indicated that the gold film was broken immediately on contact of the niobium rider on the rotating disk. The coefficient of friction at the beginning of the experiment for the gold films on uncleaned nickel alloy substrates was about 1.2. This value was indicative of the unlubricated sliding (1.2 and 1.25 for Ni-Cr and Ni-Re respectively). This indicated that the gold film was broken and metal-to-metal contact through the film had occurred. Based on the above evidence it was obvious that very weak adhesion existed between the gold film and the uncleaned surface.

The thermally etched nickel alloy substrates of nickel-chromium and nickel-rhemium with gold films maintained a coefficient of friction between 0.3 and 0.4 for a relatively long time (45 min.). This indicated that there was strong bonding (adhesion) due to the formation of a diffusion-type interface, as discussed previously.

As the film was broken, the coefficient of friction did not abruptly rise to the value for the bare metal but increased gradually and became erratic. The absence of abrupt change may be attributed to the depth of diffusion and the grain boundary reservoirs, which can supply additional lubricant during the friction test.

SUMMARY OF RESULTS

The following results were obtained from an investigation of vapor-deposited thin gold films as lubricants on nickel and nickel-alloy substrates in vacuum:

1. Marked improvement in film endurance life during friction experiments was obtained with 1800-Å gold films when substrates were cleaned by electron bombardment and thermally etched prior to deposition of the gold.

2. Vapor deposition of gold on nickel and nickel alloys at approximately 800° F formed a diffusion type interface in which there was a mutual solubility of the substrate and the film. Because of the disregistry at grain boundaries, a higher rate of diffusion and preferential trapping in and around the grain boundaries occurred with these regions acting as lubricant reservoirs during the friction experiments.

3. Nickel-chromium and nickel-rhenium alloy substrates produced longer film durability in friction studies than did pure nickel substrates, presumably because of increased substrate hardness.

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VACUUM VAPOR DEPOSITION APPARATUS

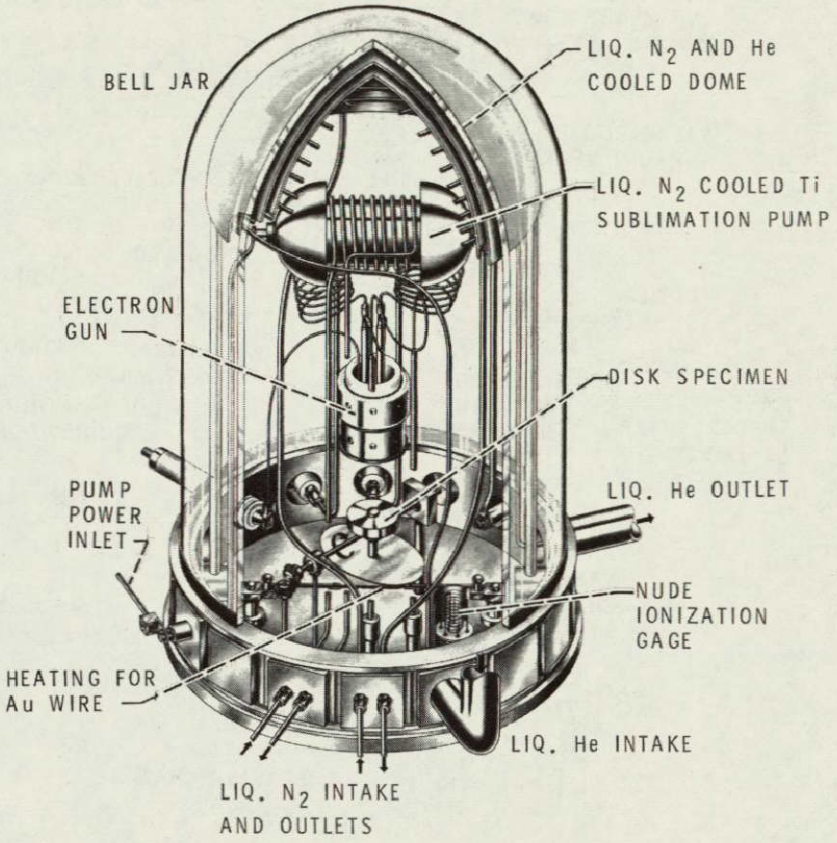
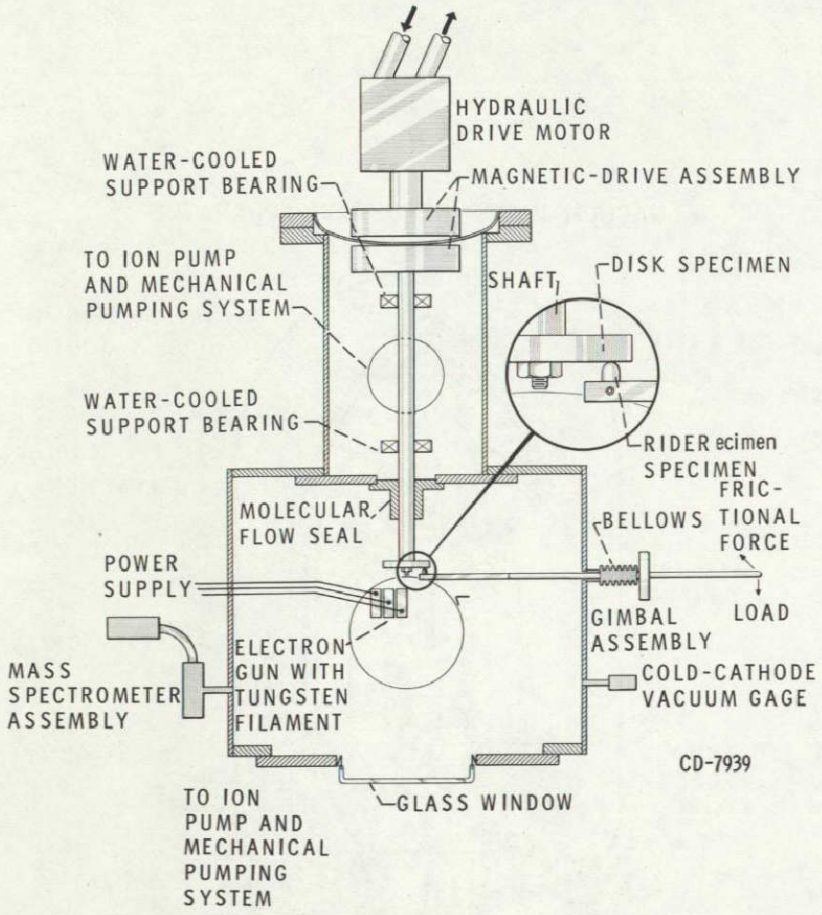


Figure 1.

ULTRA-HIGH-VACUUM FRICTION APPARATUS.



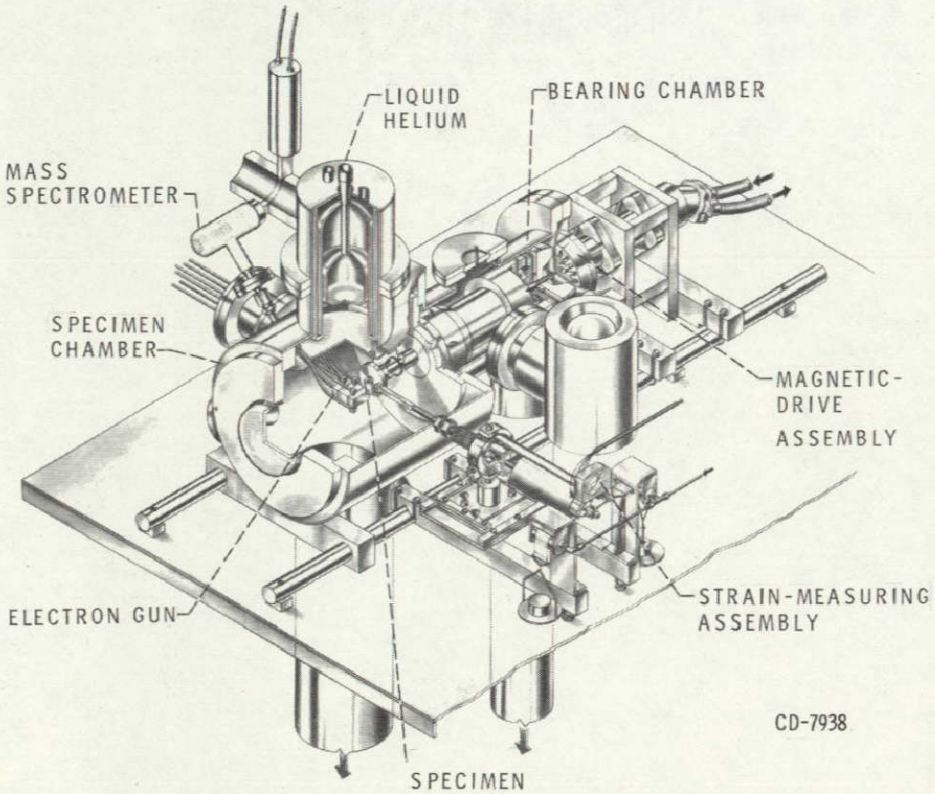
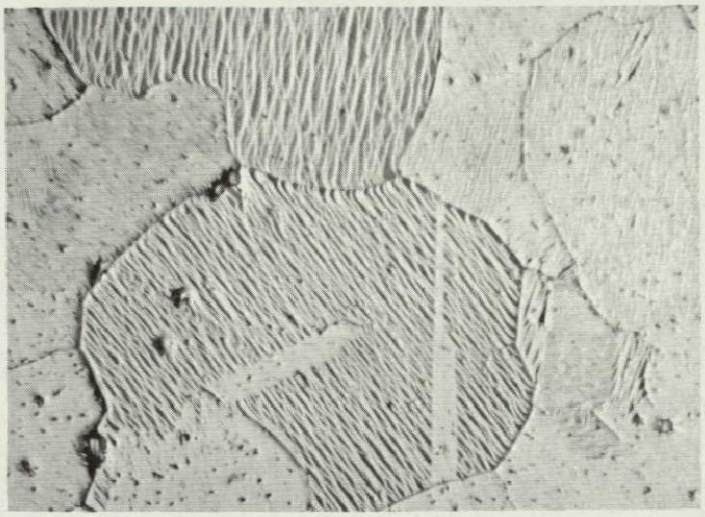


Figure 2.

ELECTRON BOMBARDED SURFACES



Cu DISK ELECTRON BOMBARDED IN HIGH VACUUM AT 1200° F FOR 3 MIN (x500)

Figure 3(a).

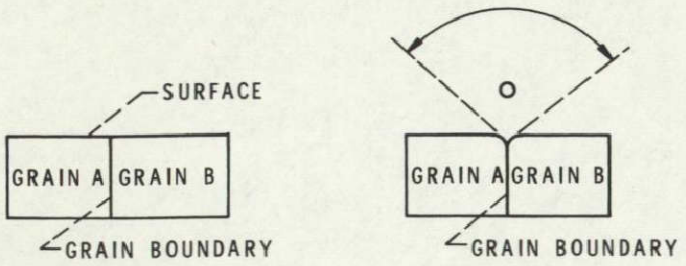
ELECTRON BOMBARDED SURFACES

CU DISK ELECTRON BOMBARDED IN HIGH VACUUM AT 1200° F FOR 3 MIN (x250)

CU DISK ELECTRON BOMBARDED IN HIGH VACUUM AT 1450° F FOR 20 MIN (x250)

Figure 3(b)(c).

GRAIN BOUNDARY REGION INTERSECTING SURFACE

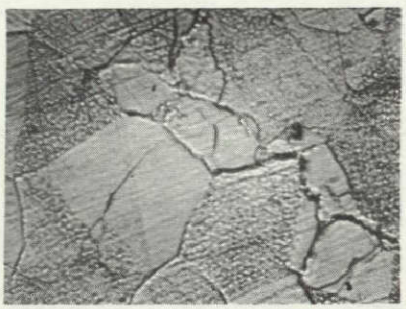


BEFORE ELECTRON BOMBARDMENT

AFTER ELECTRON BOMBARDMENT

Figure 4.

90% Ni-10% Cr ALLOY, ELECTRON BOMBARDED AT 1150° F FOR 5 MIN (x625)



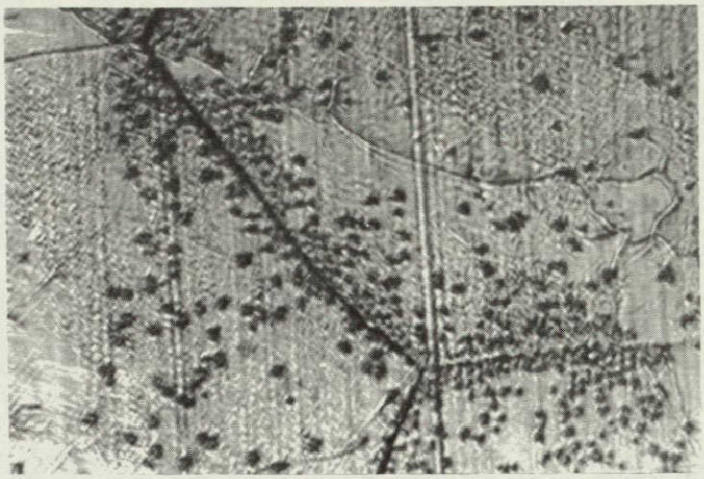
90% Ni-10% Cr ALLOY, ELECTRON BOMBARDED AT 1100° F FOR 10 MIN (x625)

ELECTRON BOMBARDED SURFACES

Figure 5(a)(b).

90% Ni–10% Cr ALLOY, WITH Au DEPOSIT

SUBSTRATE ELECTRON BOMBARDMENT



(x625)

Figure 5(c).

COEFFICIENT OF FRICTION FOR Nb SLIDING ON Ni
WITH AND WITHOUT Au FILMS

(10^{-11} MM Hg), 250 GMS, 5 FT/MIN, 75° F

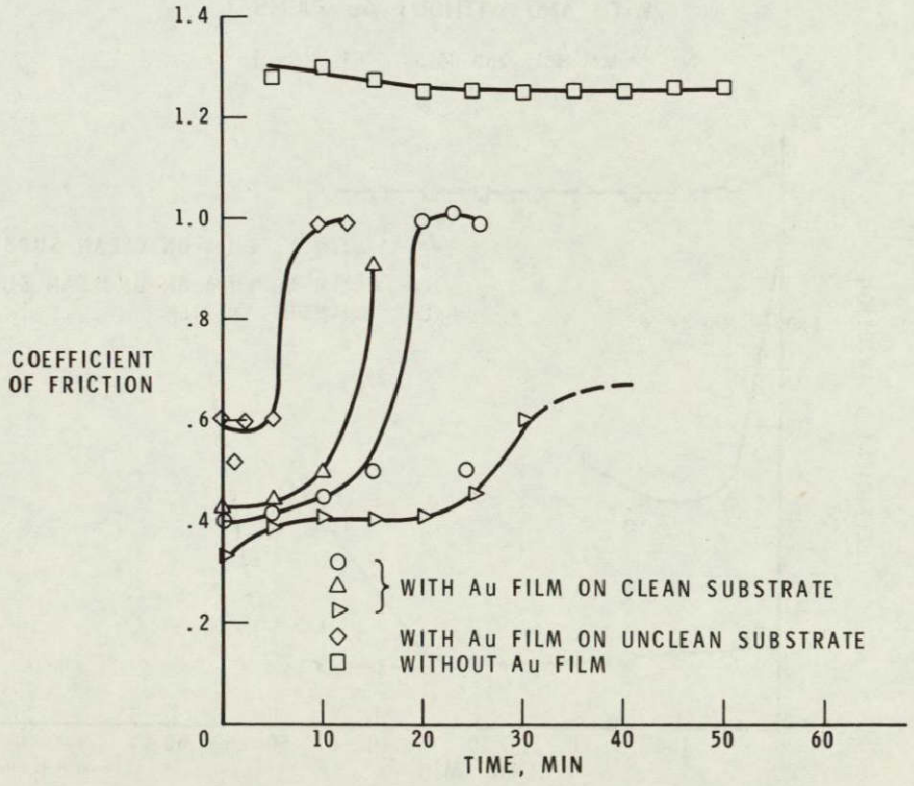


Figure 6(a)

COEFFICIENT OF FRICTION FOR Nb SLIDING ON 90% Ni – 10% Cr ALLOY
WITH AND WITHOUT Au FILMS

(10^{-11} MM Hg), 250 GMS, 5 FT/MIN, 75° F

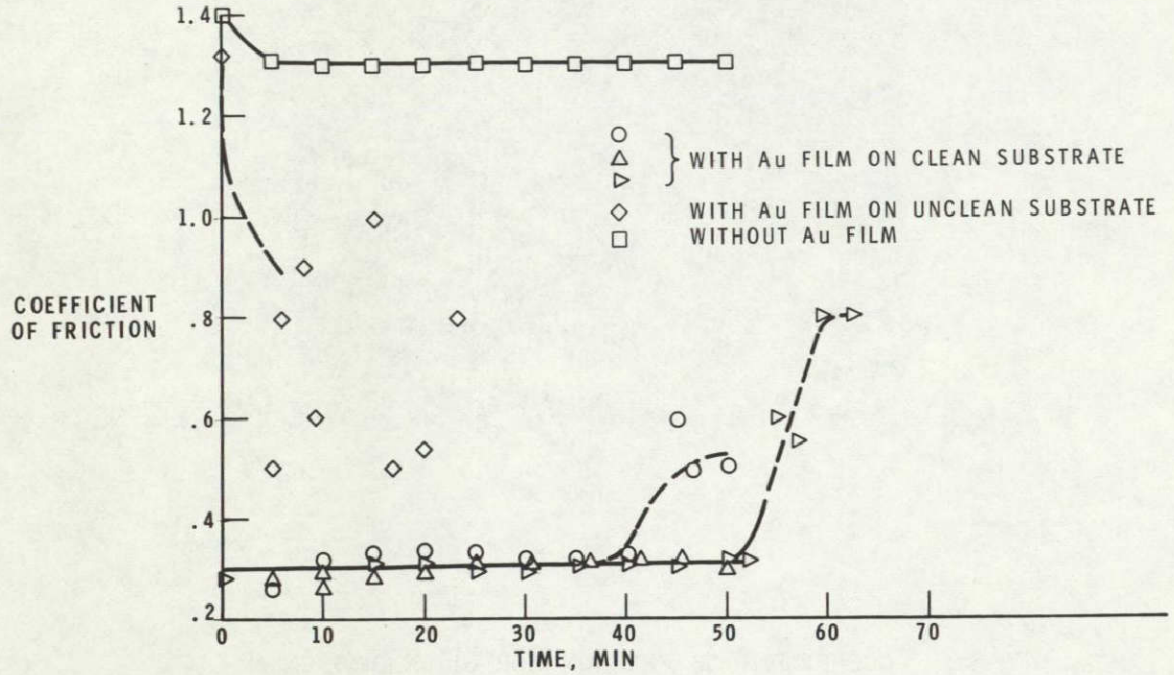


Figure 6(b).

COEFFICIENT OF FRICTION FOR Nb SLIDING ON 95% Ni – 5% Re ALLOY
WITH AND WITHOUT Au FILMS

(10^{-11} MM Hg), 250 GMS, 5 FT/MIN, 75° F

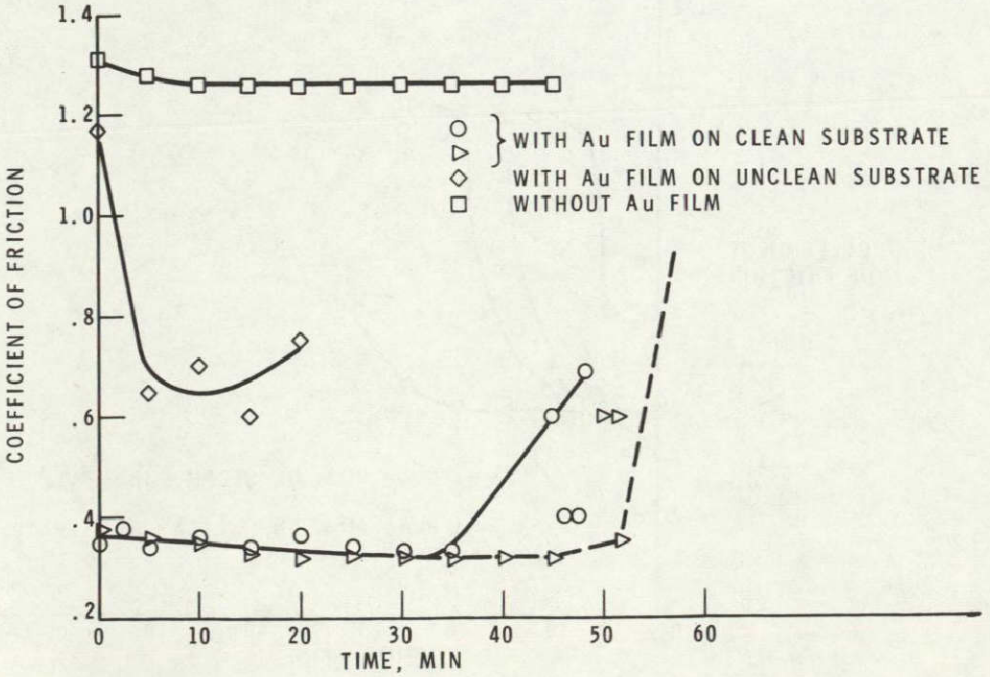


Figure 6(c).

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