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SURVEY OF ION PLATING SOURCES

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ABSTRACT

Ion plating is a plasma deposition technique where ions of the gas and the evaporant have a decisive role in the formation of a coating in terms of adherence, coherence, and morphological growth. The range of materials that can be ion plated is predominantly determined by the selection of the evaporation source. Based on the type of evaporation source, gaseous media and mode of transport, the following will be discussed: resistance, electron beam, sputtering, reactive and ion beam evaporation. Ionization efficiencies and ion energies in the glow discharge determine the percentage of atoms which are ionized under typical ion plating conditions. The plating flux consists of a small number of energetic ions and a large number of energetic neutrals. The energy distribution ranges from thermal energies up to a maximum energy of the discharge. The various reaction mechanisms which contribute to the exceptionally strong adherence - formation of a graded substrate/coating interface are not fully understood, however the controlling factors are evaluated. The influence of process variables on the nucleation and growth characteristics are illustrated in terms of morphological changes which affect the mechanical and tribological properties of the coating.

INTRODUCTION

When ion plating was first reported in the literature by Mattox in 1963[1] it immediately captured interest and technological experimentation on a laboratory scale. During the last 16 years ion plating has gradually left the laboratory confines and is finding an increasing number of specialized industrial applications where other deposition techniques have not been successful. For instance, ion plating is used as a production technique in the aircraft industry to aluminize various steel fasteners.[2] The path, however, from the research laboratory to practical industrial use has progressed in a cyclic sequence.

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The extensive variation in instrumental requirements has precluded the manufacture of a standard ion plating unit in volume production. One of the basic uncertainties may be the selection of the most appropriate evaporation source. Proper selection offers the same degree of versatility in materials deposition as does the dc or rf sputtering techniques. As a result, each laboratory or industrial operation uses their own unit construction with their own specialized instrumental features to best meet their requirements.

The intense interest in the ion plating process stems from the unique features which it offers: (1) the high energy flux of ions and neutrals, and (2) the high throwing power which is responsible for the excellent coating adherence and the three-dimensional coverage. The exceptionally strong adherence and the ability to coat complex three-dimensional surfaces without rotation are difficult to match with any other available coating technique. Ion plating is an energetic activation process where the energetic ions and energetic neutrals of the gas and the evaporant have a decisive role in the nucleation and growth of the coating in terms of adherence, coherence, and morphological growth. The range of materials that can be ion plated is predominantly determined by the selection of an appropriate evaporation source and a gaseous media, to best match the end results desired.

The activity in ion plating has primarily developed at two levels, the technological and the fundamental. It is the objective of this survey to evaluate the various evaporation sources and describe the characteristics of ion plated films and their performance for use at both levels.

TECHNIQUE

Ion plating is generally defined as a plasma or ion activated deposition technique where the substrate is the cathode immersed in a glow discharge which is subjected to a flux of energetic ions and energetic neutrals to cause appreciable sputtering before and during the film formation. A basic ion plating configuration is shown schematically in Fig. 1(a) and photographically in Fig. 1(b). It consists of a dc diode configuration with a resistively heated vaporization source (boat or filament). Both electrodes (substrate and evaporation source) are isolated from the ground by utilizing a

filament and high voltage power supply which are at floating output. The details of the apparatus construction have been previously described.[3,4] The dc gas discharge is established by applying a negative potential of 2 to 5 kV to the substrate. The surface is then sputter cleaned for a selected time period depending on the substrate material and surface condition.

Sputter cleaning is the most useful technique for obtaining an atomically clean metal surface. The cleaning effectiveness can be generally monitored during the sputtering process. When sputtering begins, the initial discharge current is high and some arcing may take place. The initial high value of the current is partially dependent on the presence of surface oxides and generally these oxides have a higher secondary electron emission values than pure metals. A gradual decrease in current will occur as the oxides are sputtered from the surface until a constant minimum value is reached; at this sputter cleaning stage the substrate can be regarded as being free of oxides and the film deposition sequence is commenced.

Without interrupting the sputtering process which is an integral part of the plating process, the coating material is evaporated into the glow discharge. Essentially, ion plating consists of two processes: sputtering and ion implantation as shown in Figs. 2(a) and (b). While sputter cleaning of the specimen continues, the film deposition by implantation is begun without interrupting the sputter cleaning process. Obviously, for the film to form, the deposition rate must exceed the sputtering rate. At the beginning of film evaporation a high potential (3 to 5 kV) is set to the substrate to obtain a deeper ion implantation and this is subsequently reduced for effective plating. The plating conditions which are most commonly used in industrial ion plating are: a negative potential of 2 to 5 kV dc; an argon pressure 2 to 2.5 Pa; and a current density 0.3 to 0.6 mA/cm^2.

The above description pertains to coating substrates which are conductors. To coat insulators (ceramics, plastics, etc.) and retain a dc bias a simple modification is necessary. The insulating substrate is surrounded by a metallic screen, which is attached to the high voltage electrode.[5] To avoid contamination of the insulator surface from the sputtered screen material, it is advisable to construct the screen cage

ion current, but is a combination of ion current and secondary electron current.[8] The width of the dark space can be expanded or contracted either by decreasing or increasing the pressure.

The most effective condition for ion plating is obtained when the edge of the dark space is close to the boat and as wide as possible. Since the mean free path for the ejected material is short because of the collision processes in the dark space field, the evaporant ions will lose energy and arrive at the substrate with a distribution of energies considerably less than the applied potential.[8] The distribution will depend to a large extent on the ratio d/L where d is the dark space width and L the collisional mean free path.[7] A cathode to anode spacing of 15 cm is adequate to establish a glow discharge.

Recent studies by Teer[9] which have been based on several assumptions (e.g., all ions originate in the negative glow and that the electric field varies linearly from the cathode to the edge of the negative glow region, that symmetrical charge transfer collisions occur only, are responsible for the energy spectrum, and that the collision cross section does not change with energy) estimate that under typical ion plating conditions the ions carry only 10 percent of the energy dissipated while the neutrals carry 90 percent. Thus the plating flux consists of a small number of energetic ions and a large number of energetic neutrals. The ions and neutrals may have a distribution of energies from thermal up to the maximum energy of the discharge. The average energies of ions and neutrals were estimated to be of the order of 100 eV.

TYPES OF EVAPORATION SOURCES

The developments in ion plating instrumentation have not only been aimed at incorporating the various available evaporation sources, but also designing the vapor sources so as to increase the electron-atom collisions for enhanced ionization. The object is to expand the range of materials which can be deposited either directly or synthesized in the process and then deposited, and optimize the process parameters in terms of deposition rates and ionization efficiencies. Each evaporation source has its advantages and disadvantages and the trend has been to select a specific evaporation source to meet the requirements for the end product in terms of coating performance and economic considerations.

Based on the type of evaporation source, gaseous media and mode of transport, one may classify ion plating as shown in Fig. 4.

Resistance Heating Evaporation

Resistance heating sources, using refractory metal boats or filaments (usually tungsten or molybdenum) are preferred, because they are comparatively sturdy, inexpensive, reliable and simple to operate. However, the range of materials which have been ion plated by resistance heating evaporation is rather limited. It has been confined to the elemental metals with melting temperatures of less than 1300° C. Of course, resistance heating has obvious limitations due to fractionization of evaporating alloys or compounds, alloying or reacting of some evaporating materials with the boat and incapability to evaporate high temperature metals such as the refractories. To overcome some of the above difficulties and expand the range of materials which can be evaporated by resistance heating several modifications have been used such as flash evaporation[10] and utilization of multiple evaporation sources.

In flash evaporation powder from a reservoir is slowly and continually fed onto a preheated boat. Binary alloys of (Pb-Sn) and (Cu-Au) have been successfully ion plated retaining the original stoichiometry.[10] The use of multiple source evaporation where the relative evaporation rates are controlled by temperature adjustments at each source has not gained much interest in ion plating.

Electron Beam Evaporation

Of the various electron beam techniques the hot filament type schematically shown in Fig. 5 has very significantly increased the potentials of ion plating and is one of the principal evaporation sources used in commercial ion plating. By using the conventional electron beam gun high melting point materials exceeding melting temperatures of 3000° C have been deposited at high evaporation rates. Evaporation is generally confined to water cooled copper crucibles which have to be used for the evaporation of reactive or refractory metals.

An expanded range of usable materials mainly stems from such process extensions as evaporation with a continuous feed of alloys or compounds, coevaporation and reactive evaporation. When a rod-fed type of mechanism is used to feed the

plating material for electron beam evaporation, it is possible to produce alloy and compound coatings of closely controlled compositions.[11,12] The only complication that arises is that the electron emitter cannot be operated at the pressure where the glow discharge exists, due to extensive sputtering and possible arcing. To circumvent this difficulty the electron beam gun is isolated from the plasma region and is differentially pumped. In spite of a more sophisticated pumping system which is required, the technique offers the greatest flexibility in materials evaporation when compared to the other available sources.

The hollow cathode electron beam source schematically shown in Fig. 6 has been also used but more selectively.[13] The gun consists of a hollow cylinder or sphere that has an aperture. The gun is operated in an argon atmosphere by applying a negative potential. As soon as the glow discharge is generated, an electron beam is emitted from the aperture. The main advantages of the hollow cathode guns are that they can operate at the glow-discharge pressures, do not use a hot filament and are easy to design and construct. However, the primary disadvantage is that the gun is also sputtered in the process which contaminates the coatings. Shielding has to be used in most instances.

Radiofrequency Induction Evaporation

The induction heated evaporation source consists essentially of a ceramic crucible with the plating metal or alloy which is inductively evaporated as shown in Fig. 7. A bare induction coil without shielding can be used directly in the glow discharge region without producing any arcs. However, the conventional rf induction generator with a frequency of 450 kHz cannot be used during ion plating, because severe arcing of the coil occurs. To overcome this difficulty, the operating frequency has to be reduced to about 75 kHz which is accomplished by utilizing an appropriate induction generator. The instrumental setup is relatively simple and many metals have been successfully evaporated.[14] The induction heating source can also increase the ionization efficiency of the evaporant flux.

Impact Evaporation-Use of Sputtering Targets

The use of sputtering targets as an evaporation source in ion plating has not found any pronounced acceptance. Ion plating with a sputtering target as a vapor source is very similar to bias sputtering, with the exception that higher bias voltages are used. The limiting aspect of this process is that high substrate potentials (2000 to 5000 Vdc) that are commonly used in ion plating cannot be applied. The substrate potential has to be considerably decreased so that at some point the deposition rate becomes greater than the resputtered rate. An alternative would be to use a planar magnetron sputtering source which offers high sputtering rates.

Reactive Ion Plating

Reactive ion plating as shown schematically in Fig. 8 is ion plating with a reactant gas. The evaporant atoms can be injected in the glow discharge by resistance heating, electron-beam gun, or any of the other evaporation sources. The reactant gas is introduced in the vacuum chamber during deposition in controlled amounts. The vaporized atoms and the reactant gas are activated by the plasma and the reaction can occur in the gas phase before the material is deposited, or it can occur on the substrate forming a stoichiometric coating. This technique is most commonly used for the formation and deposition of carbides, nitrides, silicides, and oxides.[15-19] By varying the reactant gas concentration a graded composition region between the substrate and the coating can be formed to eliminate sharp interfaces between the two different materials.

Ion-Beam Evaporation

When an ion beam source is used a large portion of the evaporated atoms are ionized in the ion source in a high vacuum of 10^{-4} to 10^{-5} Pa and deposited on a negatively charged substrate. The ionization occurs as the evaporated atoms pass near the filament and collide with the emitted electrons. Ionization rates of 10 to 20 percent have been achieved. Various design configurations of the ion sources have been reported.[20]

Similar ion gun source which uses rf power has been reported.[21] The evaporated material is ionized as it evaporates, and the evaporant creates its own plasma which

permeates the deposition chamber. Deposition system pressure is 10^{-4} Pa. Evaporation by ion beam guns in high vacuum are not commonly used in ion plating.

Additional Designs

In addition to the various evaporation sources, a number of additional instrumental modifications have been designed to increase the ionizing rate. For instance, single or multi auxiliary cathodes with hot filaments are placed near the substrate to increase the ionization by electrons emitted from the cathode.[22] Inserting an rf coil between the evaporation source and substrate to promote ionization by the high frequency magnetic field[18] or inserting auxiliary positive electrodes to form a triode-assisted ion plating system.[23]

FILM CHARACTERISTICS

Ion plated films can be characterized as highly adherent, coherent, and with a high packing density. The exceptionally strong film adherence, regardless of film/ substrate combination, is attributed to the formation of a graded-fused interface and this is well documented in the literature. All interfaces represent a break in the normally uniform crystallinity and/or composition. A graded interface as compared to an abrupt one has the tendency to reduce the dispersing stresses which are created due to the differences in chemical, mechanical, and thermal properties such as crystal structure, hardness, coefficient of thermal expansion and thermal conductivity. It is also important to recognize that the adhesive strength of the film/substrate combination depends not only on the bonding across the interface, but also on the morphological growth of the film.

The graded interface and consequently the strong adherence is generally attributed to the sputter etched surface and the high energy flux of the energetic evaporant ions and neutrals striking the substrate. The exact reaction mechanism for the formation of such an interface is not fully understood but the controlling factors are, sputter etched surface, diffusion, implantation, atomic mixing and nucleation and growth characteristics as shown in Fig. 9. These factors can act separately or in various combinations, depending on the film/substrate compatibility. A number of various film/substrate combinations have been reported where the depth of the interfacial

region has been estimated. In all the following combinations Au/Al,[24] Al/Ti,[25] In/Cu,[26] Au/Cr,[27] Cr/Ni,[28] Cu/steel,[29] the depth of the graded interface ranged from a few micrometers to about 15 μm.

It should be noted that a penetration depth in the micrometer range cannot be achieved simply by implantation. Even if the impinging evaporant ions had the maximum energy of 5000 eV at an applied potential of 5000 V, the maximum depth of implantation would be 50 to 100 Å.[30] This indicates that other factors are responsible for the deeper penetration effects. Recently it has been reported[31] that the depth of the interface is affected by the degree of solid solubility which the film/substrate combination possesses. In the combination where the film/substrate has a complete solid solubility such as Cu/Ni combination the surficial heating effects and the high defect concentration introduced by the high energy flux contribute to the enhanced diffusion forming an interface in the micrometer range. On the other hand, incompatible film/substrate combinations such as Ag/Ni[31] had a narrow interfacial region in the order of 500 Å. The interface formation for this combination can be possibly explained in terms of implantation and atomic mixing of the depositing and resputtered substrate atoms.

The morphological growth of the film is influenced by the impinging ions since they transfer energy, momentum and charge to the substrate and the forming film. This affects the nucleation and growth characteristics of the film, by increasing the nucleation density. As a result the film becomes continuous at lower thickness, has a smaller crystallite size and has a higher degree in crystallite uniformity. These effects contribute to a film of higher density, reduced porosity, and increased cohesive strength.

In practical ion plating one difficulty normally arises, the inability to produce defect-free or pore-free coatings over large surface areas. This is primarily due to the difficulty in preparing surfaces which are atomically smooth over an appreciable area. The coating defect growth is basically dominated by the substrate finish and topography[32] whereas the coating-matrix morphology is controlled by the process parameters primarily the substrate temperature. To improve the homogeneous growth

morphology by reducing the film defect concentration extra effort should be placed in preparing a highly polished and smooth surface.

APPLICATIONS AND PERFORMANCE

The applications of ion plated films are basically derived from the two unique features which the technique offers. The range of materials which can be deposited directly or synthesized, has considerably widened due to the selection of a proper evaporation source and gaseous media. The areas where ion plating is most widely used are in the mechanical and metallurgical fields. For instance, application of tribological coatings (Au, Ag, Pb, TiN, TiC, etc.) to prevent mechanical failures of sliding and rotating components, increased corrosion resistance (Al, Cr, Ti, Co-Cr-Al-Y alloy, etc.), for electronic components (SiO_2, Al_2O_3, etc.), decorative purposes and in many other areas for film deposition on intricate components which otherwise would be difficult or impossible to coat by the other techniques.

The ion plated films have a great impact on the tribological and mechanical properties. The characteristics of these films which improve performance warrants a brief discussion.

Tribological Properties

Ion plated soft, metallic lubricating films such as Au, Ag, Pb, and Sn offer great improvements when applied to sliding or rotating mechanical components. When thin gold films (2000 Å) are ion plated on tool steel, Ni-Cr alloy surfaces and friction tested (pin and disk) under vacuum conditions the following improvements are observed: (a) increased endurance life, (b) lower coefficient of friction, and (c) avoidance of catastrophic failure. A typical friction curve is shown in Fig. 10 for a niobium rider sliding on a (Ni-Cr) alloy surface ion plated with a 2000 Å thick film. The increased endurance life is attributed to the improved adherence, the lower coefficient of friction (0.18) is attributed to surface hardening primarily due to a decrease in the crystalline size, shape, and an increase in cohesive strength, and the gradual increase of the coefficient of friction rather than an abrupt one due to the formation of an interfacial region, not observed with a gold film deposited by vapor deposition.

Mechanical Properties

The impact which the graded interface exerts on the mechanical properties has been shown by tensile,[33] fatigue,[34] and creep[35] tests. Typical load elongation curves for nickel and Inconel tensile specimens with an ion plated copper film 1500 Å thick and without such a film are shown in Fig. 11. The ion plated specimens show an increase in yield and tensile strength from 5 to 8 percent. Similar results have been reported[34] where 316 stainless steel and tungsten wires were ion plated with Cu and Ti. The higher strength properties are explained in terms of the interfacial and surface effects which inhibit the motion of the slip planes during sliding. Fatigue life can also be extended by ion plating as shown in Fig. 12. Nickel and Inconel fatigue specimens plated with a 1500 Å thick gold and copper film had 23 to 27 percent increase in fatigue life, respectively. Practically all fatigue failures start at the surface, therefore the increase in the fatigue life is attributed to solid solution strengthening and surface residual stresses induced during ion plating. Similar results have been reported wherein steel fatigue specimens were ion plated with gold where the fatigue limit is considerably increased. The creep resistance has been also improved when a Ti-6242 alloy was ion plated with platinum and tested at room and elevated temperatures up to 455° C.[35]

CONCLUDING REMARKS

The ion plating process offers the highest ionization efficiencies and ion energies of the plating material when compared to all other deposition techniques. As a result three-dimensional coverage with excellent adherence is obtained regardless of coating/substrate combination. The future extension of industrial applications will depend on careful evaluation of the highly diversified equipment configurations available with the intent to establish a standard ion plating unit with a reasonably fixed set of operational parameters.

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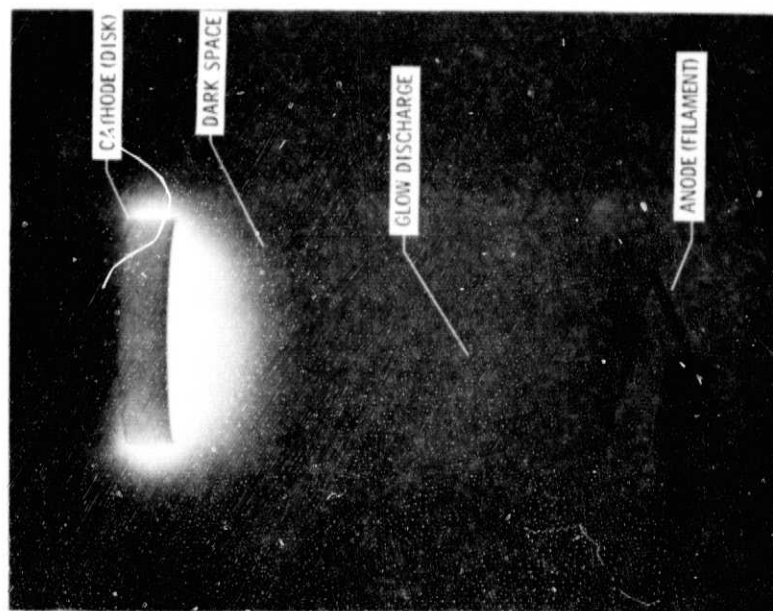35. S. Fujishiro and D. Eylon, Thin Solid Films, $\underline{54}$, 309–315 (1978).

FIGURE LEGENDS

(a) Schematic.
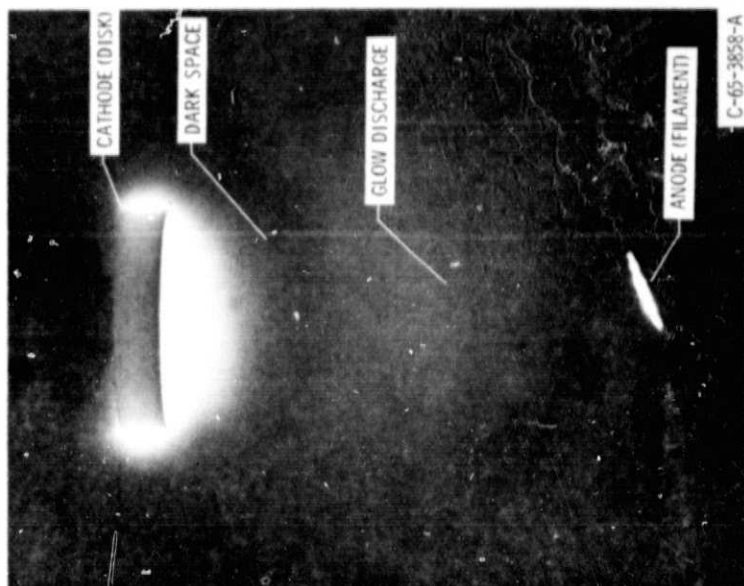
(b) Ion plating chamber.

Figure 1. - Ion plating system.

(a) Glow discharge sputter etching.

(b) Glow discharge during ion plating process.

Figure 2. - Sequence of ion plating.

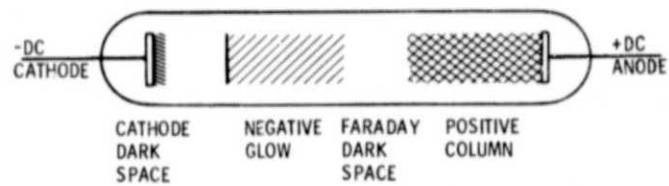
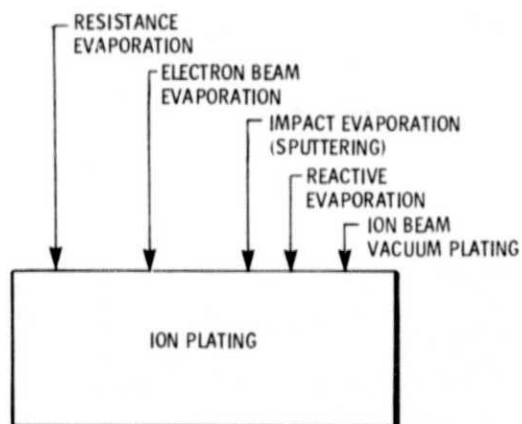Figure 3. - Most prominent light and dark zones of an abnormal glow discharge.

Figure 4. - Classification of ion plating as to the source and media of evaporation.

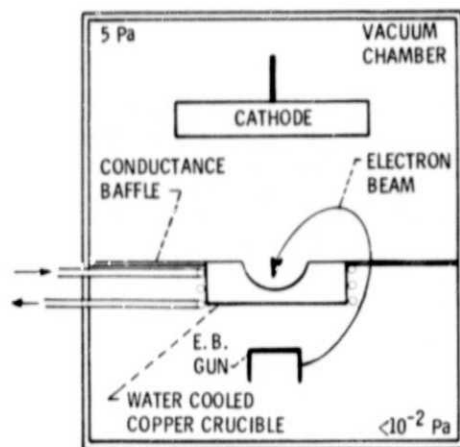Figure 5. - Schematic layout of ion plating by electron beam gun.

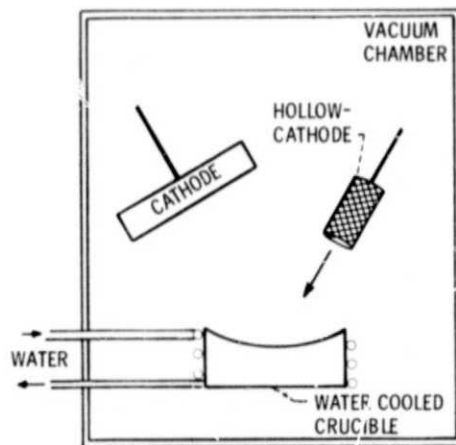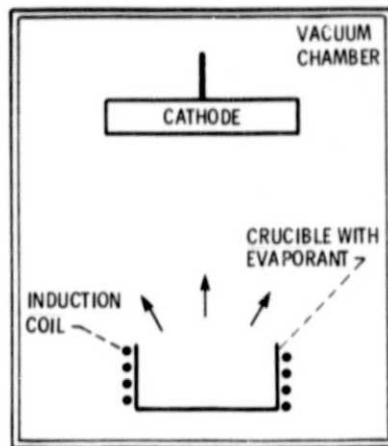Figure 6. - Schematic of ion plating by hollow-cathode.

Figure 7. - Schematic of layout of ion plating by induction heating.

Figure 8. - Schematic layout of reactive ion plating.

Figure 9. - Illustration of the factors which influence adherence and interface formation during ion plating.

Figure 10. - Coefficient of friction of niobium sliding on (Ni-Cr) alloy with gold deposited by vapor deposition, and ion plating about 2000 Å thick (load, 250 g; speed, 1.52 m/min; ambient temp. 10^{-11} torr).

Figure 11. - Load elongation curves during tensile tests.

Figure 12. - Effect of copper and gold ion plated films (1500 Å) on the fatigue life of nickel.

(a) SCHEMATIC.

(b) ION PLATING CHAMBER.

Figure 1. - Ion plating system.

(b) GLOW DISCHARGE DURING ION PLATING PROCESS.

C-65-3858-A

Figure 2. – Sequence of ion plating.

(a) GLOW DISCHARGE SPUTTER ETCHING.

CATHODE   NEGATIVE FARADAY POSITIVE
DARK   GLOW   DARK   COLUMN
SPACE   SPACE

Figure 3. - Most prominent light and dark zones of an abnormal glow discharge.

CS-79-3332

Figure 4. - Classification of ion plating as to the source and
media of evaporation.

CS-79-3335

Figure 5. - Schematic layout of ion plating by electron beam gun.

CS-79-3341

Figure 6. - Schematic of ion plating by hollow-cathode.

CS-79-3340

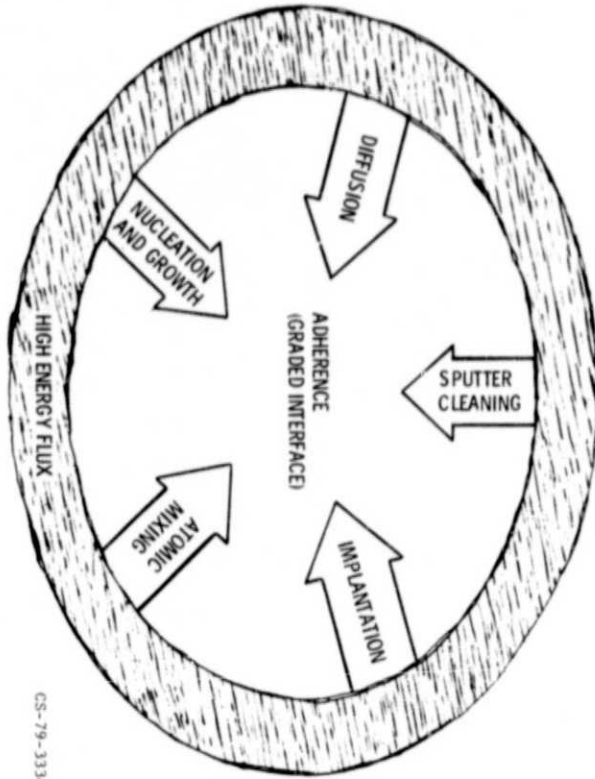Figure 7. - Schematic of layout of ion plating by
induction heating.

CS-79-3336
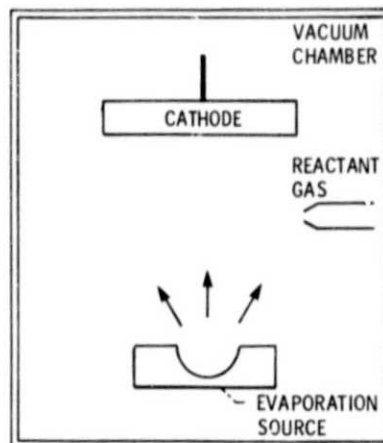
Figure 8. - Schematic layout of reactive ion
plating.

CS-79-3342

Figure 9. - Illustration of the factors which influence adherence and interface formation during ion plating.
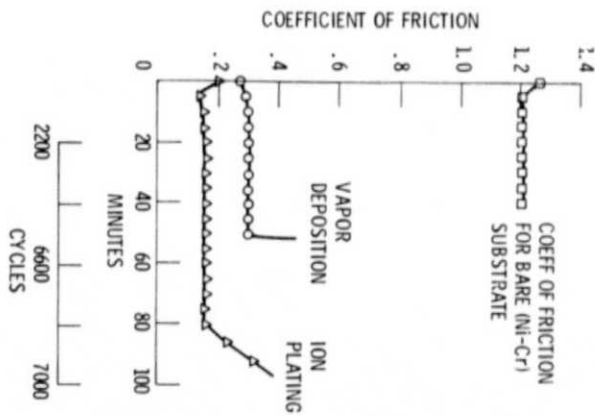
CS-79-3334

Figure 10. - Coefficient of friction of niobium sliding on (Ni-Cr) alloy with gold deposited by vapor deposition, and ion plating about 2000 Å thick (load, 250 g; speed, 1.52 m/min; ambient temp. 10^{-11} torr).
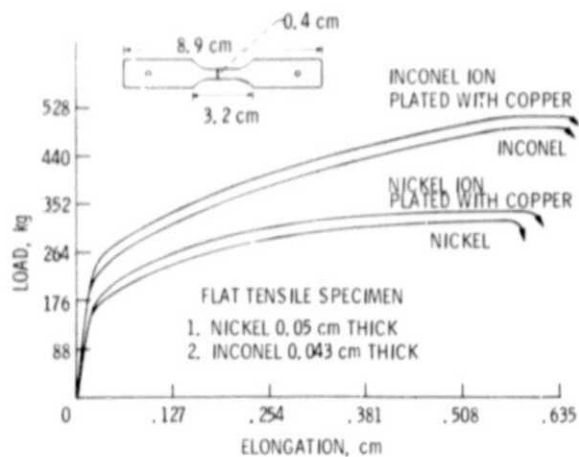
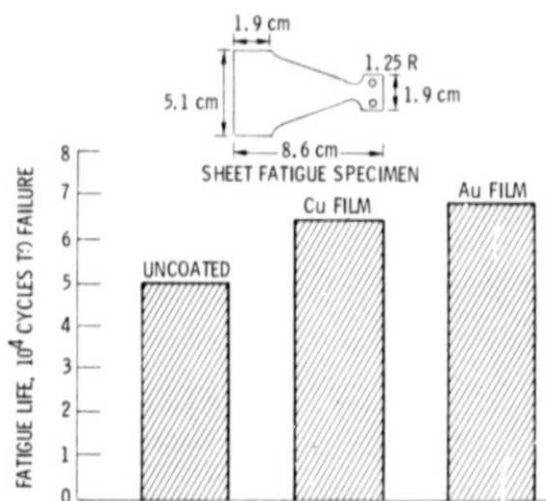Figure 11. - Load elongation curves during tensile tests.

Figure 12. - Effect of copper and gold ion plated films (1500 Å) on the fatigue life of nickel.