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MASTER

FUNDAMENTAL PROCESSES IN ION PLATING*

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ABSTRACT

Ion plating is a generic term applied to film deposition processes in which the substrate surface and/or the depositing film is subjected to a flux of high energy particles sufficient to cause changes in the interfacial region or film properties compared to a nonbombarded deposition. Ion plating is being accepted as an alternative coating technique to sputter deposition, vacuum evaporation and electroplating. In order to intelligently choose between the various deposition techniques, the fundamental mechanisms, relating to ion plating, must be understood. This paper reviews the effects of low energy ion bombardment on surfaces, interface formation and film development as they apply to ion plating and the implementation and applications of the ion plating process.

DISCLAIMER

[illegible]

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INTRODUCTION

Ion plating is a generi term applied to film dêposition processes in which the substrate surface and/or the depositing film is subjected to a flux of high energy particles sufficient to cause changes in the interfacial region or film properties compared to a nonbombarded deposition. Such changes may be reflected in the adhesion, film morphology, film stress, film density or the coverage of the surface by the depositing film material. The ion plating technique was first reported in the technical literature in 1964.[1]

This definition of ion plating refers only to processes and effects which occur on the substrate or film and does not specify the source of the depositing material nor the origin of the bombarding species. Variations in the ion plating technique have given rise to terms such as Vacuum Ion Plating, Reactive Ion Plating, Chemical Ion Plating, Bias Sputtering and others which refer to specific environment, sources or techniques. In addition, other terms are used for the ion plating process such as Ion Vapor Deposition, Ion Evaporation, Accelerated Ion Deposition and others. The fundamentals of the application of the ion plating process have been discussed elsewhere[2,3,4,5] and a rather extensive, though not complete, bibliography on ion plating and related subjects has been prepared.[6]

The concepts upon which the development of the ion plating process were based were extensions of studies of the sputtering

process where momentum transfer process are used to remove surface material by ion bombardment.[7] Recently, the ion implantation scientists have used high fluence beams to create the field of "Ion Implantation Metallurgy,"[8] which has many of the same considerations as ion plating.

Figure 1 depicts the most simple ion plating equipment. The conducting surface to be coated is biased at a negative potential with respect to the DC abnormal glow discharge plasma of argon gas such as is used with sputter deposition. Ions and high energy neutrals created by charge exchange processes bombard the surface prior to and during the deposition of film particles which are obtained from a resistively heated evaporator filament. Some of the film particles are ions which are formed while traversing the plasma but in most cases, the film particles are primarily neutral atoms. For a coating to be formed, it is necessary that the flux of depositing particles be greater than the flux of sputtered particles. If not, only those depositing particles which penetrate the eroding surface will remain.

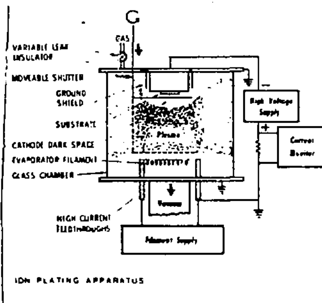

Fig. 1. Simple Ion Plating Apparatus

When the ion plating process was first introduced (1964), the origin of the benefits derived from ion bombardment, prior to and during deposition, were the subject of speculation since the effects of low energy ion bombardment on surfaces were poorly understood. Since that time, a great deal of information and some understanding of ion bombardment effects have developed and allows more informed speculations to be made.

ION BOMBARDMENT EFFECTS: PRIOR TO DEPOSITION

Ion bombardment of a surface results in a number of effects including: 1) sputtering, 2) defect production, 3) crystallographic disruption, 4) surface morphology changes, 5) gas incorporation, 6) temperature rise, 7) changes in surface composition and 8) physical mixing of near-surface materials.

SPUTTERING:

Physical sputtering, which is usually referred to as sputtering,[7] is a momentum transfer process in which an incoming particle creates a collision cascade which intersects the surface causing the ejection of a surface atom. The sputtering yield is defined as the number of atoms ejected per incident particle and is a function of the mass of the impinging particles, particle energy, angle of incidence surface morphology and the bombardment history of the surface.

Increasing mass and energy of the impinging particles will give increasing sputtering yields up to the point where

most of the energy is dissipated so deeply in the surface region that the major portion of the collision cascade produced does not intersect the surface. The sputtering yield will increase with off-normal angles of incidence up to 60-70° and then decrease as the incident ions are reflected from the surface. Typically, angular dependence can account for a factor of two variations in the sputtering yield.

Sputtering yield may also be bombardment dose dependent, increasing with bombardment dose to some equilibrium value.[9] The increased sputtering yield with dose is attributed to the introduction of defects and foreign atoms into the near-surface region which reduces the local atom bonding. The variation of sputtering yield is also a function of the bombarding environment. For instance, if there is a high residual gas pressure, a portion of the sputtered atoms will be backscattered to the surface.

If materials having a low sputtering yield are motile on the target surface, then the low yield material may agglomerate into an island structure giving cone formation on the surface.[10] The surface mobility may allow appreciable amounts of bulk material to be removed without removing the initial surface material. This has led to problems with composition profile measurements by sputter-Auger and secondary ion mass spectrometry in some systems.

Chemical sputtering of a surface results when the energetic and activated bombarding species form a chemical bond with the

surface species giving a volatile or more easily sputtered material. This effect enhances the apparent sputtering yield. It is difficult to separate the physical and chemical sputtering components of the sputtering of materials by reactive gases.

Sputtering of a surface results in the removal of surface material, hence may be called sputter cleaning: The use of a reactive gas gives reactive plasma etching[11] or reactive plasma cleaning[12] of the surface.

DEFECT PRODUCTION:

The amount of energy transferred to the lattice atom (E_t) by the bombarding particle depends on the relative masses of the particle and is given by:

$$E_t = \frac{4M_i \; M_t}{(M_1 + M_t)^2} E ,$$

Where E is the energy of the incident particle of mass, M_1 and M_t is the mass of the target atom. If the transferred energy exceeds about 25 eV, the lattice atom may be displaced to an interstitial site and point defects are formed (damage threshold). If less than 25 eV, the energy will all appear as heat.

The combination of thermal agitation and displacement will create migrating interstitials and vacancies which agglomerate and result in the formation of dislocation networks.[13] Even with agglomeration of defects, there remains an extremely high

residual concentration of point defects in the surface region of an ion bombarded surface. These defects have been studied by the trapping and desorption of low energy helium ions and show that the trapping sites are vacancies with varying binding energies for the trapped particles.[14]

CRYSTALLOGRAPHIC DISRUPTION:

If the defects produced by ion bombardment are sufficiently immobile, the surface crystallography will be disrupted into an amorphous structure.[15,16] Also, gas incorporation will tend to disrupt the surface crystallography. These effects are well known to investigators concerned with Low Energy Electron Diffraction (LEED) studies of surface crystallography. These studies normally require that ion bombarded surfaces be extensively annealed in order to restore the surface crystallography.[17]

SURFACE MORPHOLOGY CHANGES:

Ion bombardment of both crystalline and amorphous surfaces results in a wide variety of topographical changes[10] giving increased surface roughness and changes in sputtering yield. Generally, there is a strong dependence of surface morphology on ion bombardment dose, particle energy, species and angle of incidence, as well as surface condition such as crystallography, microstructure and impurity concentration.

GAS INCORPORATION:

Incorporation of gases into surfaces and growing films by low energy ion bombardment has long been recognized and

studied and is the basis for inert gas pumping by "sputter ion" type vacuum pumps. Inert gases have been deliberately incorporated into surfaces and films by low energy ion bombardment for a variety of reasons. The ability to incorporate the insoluble gases into the lattice of the surface or depositing film depends on the mobility, trapping sites and temperature, as well as the energy of the depositing species. Generally, amorphous materials trap more gas than do crystalline materials.[18] In bias sputter deposition, done with argon, the gas concentration may be as high as several percent and may cause blistering on subsequent heating. Gas incorporation increases with particle energy until heating by bombardment causes the release of the entrapped gas.

TEMPERATURE RISE:

Most of the energy of bombarding particles appear as surface heating. The surface to mass ratio, thermal properties of the system and the energy input to the system determines the resulting bulk temperature of the bombarded material. The atoms in an ion bombarded surface are being displaced from their equilibrium positions with energies up to or greater than 25 eV (damage threshold) and the meaning of a surface temperature with such great agitation is uncertain.

SURFACE COMPOSITION:

Without diffusion, atoms will be removed from a surface by sputtering in a stoichiometric ratio after an initial conditioning period. Because of differential sputtering yields

of the components of the system, the surface composition may differ from that of the bulk.[19] Ion bombardment itself may result in surface composition changes due to preferential "knock-on" or recoil effects, sometimes called "recoil implantation."[20]

Diffusion in the surface region can have a pronounced effect on composition. The presence of a high defect concentration and a high temperature will enhance diffusion such as is found with radiation enhanced diffusion.[21] Since the surface acts as a sink for point defects, the defect flow may allow solute segregation with small ions segregating to the surface.[22]

PHYSICAL MIXING:

Physical mixing of the near-surface material results in what may be termed a "Pseudodiffusion"[23] layer where the mixing occurs without the need for solubility or diffusion. Recoil implantation results in surface materials such as oxygen or carbon being embedded into the surface region.[20] If the sputtered atoms are returned to the surface by backscattering,[24] there will be an intermixing. If the sputtered atoms are ionized and accelerated back to the surface,[5] they will become embedded in the surface region.

ION BOMBARDMENT EFFECTS: INTERFACE FORMATION

Ion bombardment of a surface, while adding coating atoms, results in: 1) physical mixing, 2) enhanced diffusion, 3) modified nucleation modes, 4) preferential removal of loosely bound atoms and 5) improved surface coverage.

PHYSICAL MIXING:

Mixing of the near-surface region by nondiffusion type mechanisms will occur due to implantation of high energy particles,[8] backscattering of sputtered atoms[24] and recoil implantation of surface atoms.[25] The high energy particle may arise from a variety of sources. The coating atoms may be ionized and accelerated while traversing the plasma in a glow discharge[26] or may be ionized in an ion source then extracted and accelerated through a vacuum (ion beam plating). High energy particles may also be formed by being sputtered from the surface, ionized in the plasma, then accelerated back to the surface. The net effect of this intermixing will be to form the aforementioned "Pseudodiffusion" type interface.

ENHANCED DIFFUSION:

The high defect concentration and temperature of the near-surface region will enhance the diffusion rates.[27] Diffusion into the bulk or surface segregation may be enhanced by solute segregation effects where small ions will tend to segregate to the surface which acts as a sink for point defects.[22]

NUCLEATION MODES:

The behavior of an atom condensing on the substrate surface is determined by its interaction with the surface. If there is no strong bonding between the condensing atom and the surface, the atom will diffuse over the surface until it nucleates at a high energy site or by collision with other

diffusing atoms. This may be termed a nonreaction nucleation mode and will lead to widely spaced nuclei. The growth of the nuclei will possibly lead to a "dewetting" type growth[28] with interfacial porosity[29] as the nuclei grow together. The nuclei will not grow together until they have reached an appreciable thickness. This type of nucleation and growth leads to the island-channel-continuous growth pattern often studied with electron microscopy.[30].

If the condensing atom reacts strongly with the surface, the surface mobility will be limited and the nucleation density will increase and, in the limit, the depositing atoms will form a continuous monolayer on the substrate surface.[31] If there is chemical reaction or diffusion, the condensing atoms will react with the surface to form compound or alloy layers which extend both normally and laterally to the surface. This mode of growth will tend to decrease interfacial porosity and "grade" the properties of the interfacial region.[23] Extensive interfacial reaction may degrade the interface by the formation of porosity due to differential diffusion rates or by high intrinsic stresses generated by thick interfacial compound layers.[32] Very thin contamination layers on the surface can convert the diffusion/reaction nucleation mode to the nonreaction mode with an attendant undesirable interfacial region.[28]

An ion bombarded surface, with its surface morphology and disruption probably provides more nucleation sites than an undisturbed surface giving a higher nucleation density even

for nonreactive systems. Ion bombardment of the surface will tend to remove and disrupt contamination and barrier layers which prevent the diffusion/reaction nucleation mode.[33] Implanted atoms may also act as nucleation sites.

PREFERENTIAL REMOVAL:

Since sputtering of surface atoms is dependent on the local bonding, it is not unexpected that ion bombardment of a surface will tend to sputter the more loosely bound atoms. This is particularly evident when the reaction/diffusion type of interface is being formed and has been used to deposit pure Pt-Si layers by sputter depositing platinum on silicon and backsputtering the excess platinum.[34] It has also been noted that "high quality" SiO_2 films can be formed by bias sputtering and this is attributed to backsputtering of loosely bonded atoms,[35] though other effects may be important.

SURFACE COVERAGE:

The increased "throwing power" of the ion plating process over large areas and out of line-of-sight of the vapor source, when used in a gas discharge, is most probably due to gas scattering. The effect has been noted at high gas pressures without the discharge present[36] but ion bombardment is probably important in obtaining an adherent coating. Evaporation into a high residual gas environment results in deposition of a fine powder and is called "gas evaporation."[37] Vapor phase nucleated particles in a gas discharge will assume a negative

potential and be repulsed by the cathodically charged ion plating substrate[5] or may be used for "Ionized Cluster Beam" deposition processes.[38]

On a microscopic scale, the enhanced surface coverage is probably due to sputtering of the deposited atoms into regions out of line-of-sight of the source.[39] Backsputtering has been used to preferentially deposit materials on vertical sidewalls even to the extent that all deposited material on the surfaces normal to the impinging ion beam is completely removed.[40]

ION BOMBARDMENT EFFECTS: FILM GROWTH

Deposited materials often have properties and behaviors which are appreciably different than those found in bulk material. These differing properties and behaviors may include: 1) small grain size, 2) high defect composition, 3) low recrystallization temperatures (metals), 4) low strain point (glasses), 5) high intrinsic stresses, 6) metastable crystallography and phase composition and many others. Ion bombardment of a film during deposition may affect film morphology, crystallographic composition, physical properties and many other properties.

MORPHOLOGY:

The morphology of a depositing film depends on how the depositing adatoms are incorporated into the existing structure. On a smooth surface, preferential growth of one area over another may result from varying surface mobilities of the depositing atoms usually as a result of grain orientations.

Preferential growth leads to a dominant grain orientation and a surface roughening with thickness. As the surface becomes rough, geometrical shadowing will lead to preferential growth of the elevated regions giving a columnar morphology to the deposit.[39] A pre-existing surface roughness or preferential nucleation will also lead to shadowing and a columnar morphology. An elevated substrate temperature will affect the morphology by increasing surface mobility, enhancing bulk diffusion and allowing recrystallization to occur. These effects have led to the structure zone model of deposited materials of Movchan and Demchiskan.[41] The model consists of the formation of three zones which depend on the ratio of the surface temperature (T) to the melting point of the deposited material (T_m).

Zone 1 results when adatom diffusion is insufficient to overcome the effects of shadowing, giving a columnar structure with low density boundaries between the columns.[42] The individual columns are polycrystalline and are usually highly defected with small grain size. The surface morphology is generally rounded.

Zone 2 is defined as the range of T/T_m where adatom diffusion dominates and the column structure consists of less defected and larger grains with higher density boundaries between the columns. The surface morphology of the Zone 2 material is generally more angular than that of the Zone 1 material.

Zone 3 is the T/T_m region where bulk diffusion and recrystallization dominate.[42] The material is more equiaxed with high density grain boundaries and large grains.

Rough surfaces,[39] high gas pressures, oblique adatom flux[43] and reactive contaminant gases will tend to lower the T/T_m values for the zone boundaries. Ion bombardment, during deposition, may tend to raise the T/T_m values for the zone boundaries[44] though this effect may be offset by gas incorporation and smaller initial grain size.

CRYSTALLOGRAPHY:

Structural order is generally controlled by the surface mobility of the adatoms. Low mobility will lead to amorphous or fined grained material. High mobility will lead to large grained material and more structural perfection. Bulk diffusion will determine the crystallography of materials deposited at elevated temperatures (Zone 3). Preferential crystallographic orientation will result from the differential surface mobilities at low temperatures or the recrystallization kinetics at high temperatures.

Ion bombardment of the depositing material may influence crystallography by enhancing diffusion and recrystallization or by altering the nucleation stage of the film growth.

COMPOSITION:

As previously discussed, ion bombardment can alter the composition of the depositing material by preferentially sputtering loosely bound atoms or implanting atoms into the

growing surface region to form metastable phases. In the extreme case, many atomic percent of normally insolüble gases can be incorporated into a depositing film by ion bombardment.

PHYSICAL PROPERTIES:

Intrinsic stress is probably one of the most obvious properties which is affected by ion bombardment. Intrinsic stress is generated by atoms which are not in their lowest energy configurations. Generally, evaporative deposited films have a tensile stress while sputter deposited films have a compressive stress. The magnitude of the stresses often approaches the yield point of the bulk materials and, in some cases, may exceed the yield point of pure bulk materials because of incorporated impurity atoms. Compressive stresses occur when the atoms are in closer proximity to one another than they would be under more equilibrium circumstances. Tensile stresses develop when atoms are further apart than they would be under equilibrium circumstances. Some care must be taken with these interpretations since stress is often measured by x-ray analysis of lattice parameters which tell nothing about intergranular forces. Beam deformation measurements give an integrated stress measurement which may be more meaningful.[45]

For sputter deposited films, an elevated gas pressure, an oblique adatom flex or an elevated temperature will reduce the compressive film stress.[46] In some cases, stress may be reduced by a relief mechanism such as diffusion or recrystallization. Recrystallization temperature and rate are dependent on the intrinsic strain energy and defect concentration. In other cases, the structure may be unable to support the stress such as the low density structure of Zone 1 material where the weak columnar boundaries will not be able to transmit a stress. In such a case, the x-ray analysis may show high stress but a bending beam test will show little integrated stress.

Ion bombardment, during deposition, may increase the stress by forcing atoms into nonequilibrium positions and suppressing Zone 1 formation, or may decrease stress by enhancing stress relief mechanisms such as diffusion or recrystallization. Generally, the more refractory the material, the more likely ion bombardment is to increase stress. A low intrinsic stress is usually desirable for good adhesion[23] though, in some cases, a compressive stress may be beneficial since brittle materials under compression are generally stronger than unstressed materials, since fractures will not tend to propagate.

Other physical and mechanical properties such as hardness, yield strength, etc. are probably affected by ion bombardment during deposition, but there have been few studies in this area.

IMPLEMENTATION

The equipment needed for ion plating is essentially the same as that needed for sputtering or sputter deposition. The

principal concerns are in obtaining uniform high energy particle bombardment and deposition over the surface to be coated. Where a plasma is used to provide particle bombardment, this means obtaining a uniform plasma density over the surface. The presence of contaminants in the plasma may be highly detrimental to "cleaning" the surface.[33]

Figure 1 depicts an ion plating system using a resistively heated thermal evaporation source. Other sources which may be used in a plasma environment are shown in Figure 2. When a sputtering cathode is used, the process is often called bias sputtering. An electron beam evaporation source is popular particularly when using reactive gases to deposit compounds, carbides and nitrides (reactive ion plating). The use of a compound gas which decomposes in the plasma is called chemical ion plating and is analogous to plasma-enhanced chemical vapor deposition.[47]

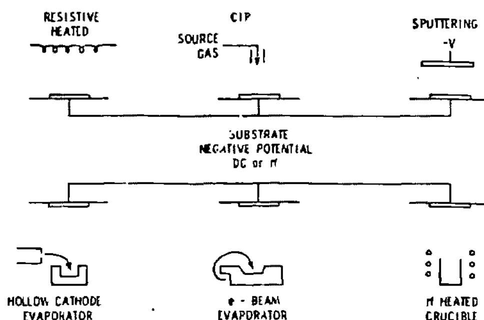

Fig. 2. Sources for Ion Plating[6]

One of the problems with ion plating, using a plasma environment, is separating the deposition parameters from the particle bombardment parameters. Figure 3 shows how ion plating may be done in a vacuum environment (vacuum ion plating) using an ion gun for the bombarding source. This allows the bombardment parameters and the deposition parameters to be independently varied.[48] A pure ion beam of the film material may be used for ion plating. At higher energies the impinging ions clear the surface of contaminants and depositing atoms. By lowering the energy of the bombarding ions a resulting film may be formed.

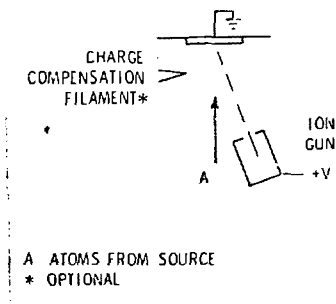

Fig. 3. Vacuum Ion Plating

Fig. 4. Enhanced Ionization Sources

Figure 4 shows a variety of techniques that have been used to enhance ionization for higher plasma densities and increased ionization of the depositing particles. Most of these techniques use electron emitters to enhance ionization. An exception to this is the positively biased electrode used to attract secondary electrons emitted from the e-beam evaporation and thus expand the plasma region. Most of these techniques are more fully described in Reference 6.

APPLICATIONS

Many of the applications of ion plating are covered in Reference 6. One of the greatest attributes of the ion plating process is the good adhesion which can be obtained with a wide variety of coating-substrate combinations. Metallurgical applications of ion plated coatings include: corrosion resistant coatings, wear and erosion resistant coatings, lubrication coatings, coatings for bonding and joining, and coatings to

improve creep and fracture properties of materials. Table 1 summarizes some of the film-substrate combinations which have been used for these purposes.

Bombardment during deposition can influence many of the metallurgical properties of the deposited materials including: microstructure, grain size and orientation, intrinsic stress, elastic modulus, hardness, and fracture properties. Control of these properties requires careful study of the bombardment and deposition parameters.

SUMMARY

From the standpoint of obtaining good adhesion, ion plating may be divided into surface preparation and interface formation. It seems that the most common applications of ion plating use DC glow discharges to provide the ion bombardment flux. It has been demonstrated that it is almost impossible to obtain an atomically clean surface using a DC discharge.[50] Apparently, this is due to backscattering and the ionization and return of the contaminant atoms in the discharge. Contributing factors are the low gas throughput normally used in DC discharge systems, such that contaminants are not flushed from the system.[33] Clean surfaces can be prepared by RF sputtering[50] and the use of reactive plasma cleaning, but good adhesion results are often obtained by using the DC discharge technique. This would indicate that cleaning of the surface is not the most important effect for obtaining good adhesion but rather the intermixing (pseudo-diffusion) may be the important factor.

As to the effects of ion bombardment on interface formation, little is really known. Analytical techniques which are capable of resolving atomic composition on a monolayer scale have only recently been developed and have not been applied to the problems of ion bombardment during deposition. It has been established that ion bombardment during sputter profiling can result in intermixing and surface effects which can give erroneous results to sputter profiling types of analysis.[27] To date, the studies which have been made on ion plating effects have been generally unsatisfactory. Invariably, the deposition environment is poorly defined with the energy distribution of the bombarding ions being unknown as is the ratio of the depositing atoms to bombarding particles. In some studies, the ratio is low even to the point that all of the deposited material is sputtered off. In other studies, the ratios are extremely high and the ion bombardment effects are relatively inconsequential.

Ion plating should be treated as just another deposition technique with advantages and disadvantages. The only way to intelligently utilize ion plating is to understand the fundamental considerations involved and how they could apply to a specific problem. Unfortunately, there is little quantitative information on many aspects of the mechanisms of ion plating. Defining the deposition environment is extremely complicated and the relative importance of the factors involved in ion plating are not defined since the effects of low energy ion bombardment are usually confined to surface and nearsurface

regions. Analytical techniques, applicable to studying ion plating effects, are rather limited. Another problem is that the ion bombardment parameters and the deposition parameters are usually not independent for most applications of ion plating. This makes process control rather difficult.

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TABLE I

APPLICATIONS OF ION PLATING

Optical	Electrical	Mechanical	Corrosion
Cr-Plastics	Contact	Lubrication	Gas
In(Sn)O-Glass	Pt,Al,Au,Ag-Si	Au,Ag,MoS_2 - Metals	Al-U,Ti,Steel
-Plastics	Al - GaAs		Cr-Steel
TiN - Metals	In(Ga) - CdS	Wear/Erosion	Al_2O_3-Steel
ZrC-Metals	Cu - Al_2O_3 (VIAS)	Ti(C,N), Zr(C,N), Cr(C,N)	Si_3N_4-Mo
		-Metals, Carbides	Cd-Steel
	Passivation		Ti-Steel
	Si_3N_4 - Si	Bonding	
		Ag-Be	Biological
		Cu-Ta,W,Nb,Oxides	C-Metals
		Au,Cr-Mo	Ti-Metals
		Creep	Chemical
		Pt-Ti	Ta-Steel

FIGURE CAPTIONS

Figure 1: Simple Ion Plating Apparatus

Figure 2: Sources for Ion Plating (6)

Figure 3: Vacuum Ion Plating

Figure 4: Enhanced Ionization Sources (6)

TABLES

Table I: Applications of Ion Plating