NASA TECHNICAL MEMORANDUM

NASA TM X-52823

DEPOSITION OF ALLOY FILMS ON COMPLEX SURFACES BY ION PLATING WITH FLASH EVAPORATION

by Talivaldis Spalvins
Lewis Research Center
Cleveland, Ohio
June 1970

This information is being published in preliminary form in order to expedite its early release.

DEPOSITION OF ALLOY FILMS ON COMPLEX SURFACES BY ION PLATING WITH FLASH EVAPORATION

by Talivaldis Spalvins

Lewis Research Center
Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

DEPOSITION OF ALLOY FILMS ON COMPLEX SURFACES BY ION PLATING WITH FLASH EVAPORATION

by

Talivaldis Spalvins

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio

SUMMARY

A new method is described which basically incorporates a combination of two independent methods: (1) ion plating, and (2) flash evaporation. This ion plating with flash evaporation (IPFE) method can be used to deposit alloy films without a change in composition. Three alloys of the following weight percentages were deposited: Pb-Sn (50-50), Pb-Sn (34-65) and Cu-Au (85-15). These alloy films were analyzed quantitatively and the original composition was maintained. These films can be deposited on complex surfaces (into cavities and around corners) on a stationary specimen. Exceptionally strong adherence is obtained between the film and the surface. A graded interface is formed (diffusion type) due to the high penetrating effect of the plating material.

INTRODUCTION

This paper presents a new vacuum deposition method which extends the scope of the ion plating technique. In ion plating only elemental metals can be deposited, but this newly developed method can be utilized for depositing alloy films. This method basically incorporates a combination of two independent techniques: (1) ion plating and (2) flash evaporation; and will be referred as ion plating with flash evaporation (IPFE).

The objective of this method is to deposit an alloy film which retains the same composition as the original alloy. The method was tested on two different Pb-Sn alloys and one Cu-Au alloy. These three different alloys were deposited by IPFE and the alloy films formed were quantitatively analyzed. The composition of the alloy film was essentially the same as that of the original alloy.

The unique characteristics of the ion plating method are retained in terms of plating complex surfaces from all sides and obtaining the penetrating effect of the metal ions and forming a graded interface. The ionized metal ions accelerate towards the specimen (cathode) and arrive with an energy proportional to the potential difference between

the specimen (cathode) and boat (anode). Ion plating has been described previously (ref. 1 and 2) for depositing elemental metals. This method is based on thermal evaporation and condensation principles which create difficulties when alloys have to be evaporated. Normally, alloys when evaporated by conventional evaporation methods yield a vapor that is initially rich in the more volatile component. Because of the difference in vapor pressures, the composition of the vapor varies continuously during the evaporation process and the resultant film is chemically inhomogeneous (ref. 3). To prevent dissociation or decomposition of the alloy it is essential to reduce the time factor present during thermal evaporation. The solution here is to use flash evaporation. In flash evaporation the material is dropped continuously into a boat whose temperature is set as high as necessary to evaporate the least volatile component.

Flash Evaporation

The flash evaporation technique was first reported by Siegel and Harris (ref. 4). They evaporated α brass, β brass and Au-Cd alloys and obtained films which had the same chemical composition as the original alloy. More recently compounds such as $BaTiO_3$ (perovskites), Ni-Cr alloys of various compositions and the semiconductors III-V compounds like GaAs, InSb, GaP, GaSb, InAs, AlSb have been evaporated retaining their corresponding stoichiometries (refs. 3 and 5).

Flash evaporation differs from the conventional vapor deposition, in that the evaporant (powder) is fed continuously into a preheated boat. The temperature of the boat is set as high as necessary to evaporate the least volatile component. The constituents of the powder when coming in contact with the boat have to be vaporized instantaneously. This instantaneous evaporation prevents fractional decomposition or dissociation of the material. The feed of the powder into the boat should be continuous during the evaporation process. No material should accumulate in the boat and the vapor which is produced from the uniform powder feed into the boat forms a film with the same composition as the original one (ref. 6). The parameters which have to be controlled critically are the particle size of the powder, rate of powder delivery to the preheated boat and the temperature of the boat.

Ion Plating

Ion plating has been described previously in the literature (refs. 1 and2). Some of the basic characteristics will be reviewed here. Basically ion plating has a diode configuration. The specimen (cathode) to be plated is an integral part of the d-c high voltage circuit. The boat (anode) with the evaporant is heated by resistance heating. The anode to cathode distance is about 14 cm and the argon discharge pressure 20 μ, applied

voltage 5 kv. When a negative potential is applied to the specimen glow discharge is established and sputter etching of the specimen commences. Sputter etching is continued up to and during the evaporation. The plating material is evaporated by resistance heating and the evaporated metal atoms are injected into the flow discharge. A large percentage of the metal atoms are ionized and they follow the electric lines of force to all points on the specimen surface.

APPARATUS

The IPFE apparatus is shown schematically in figure 1(a) and photographically in figure 1(b). The apparatus is basically an ion plating unit with an added powder feed which is controlled by a vibrator. The powder has to be delivered at a constant rate to a preheated boat from which it must evaporate almost instantaneously.

The ion plating part has been described in detail in references 1 and 2.

The particle feed is a 304 stainless steel L-shaped trough about 1-inch wide and is water cooled. At the upper end of the trough is a cylindrical cup with a screen bottom. This cup is filled with a selected particle size powder, and the powder rests on a certain mesh screen. The particle size used in this study was 125 and 250μ and the screens were 120 mesh and 60 mesh, respectively. The particle feed is mounted outside the quartz cylinder on a bracket. A linkage arm connects the bracket to a connecting rod. This rod extends out of the vacuum system by means of a vacuum tight bellow type feedthru. A vibrator (rotating motor) is attached to the rod. By means of a power supply the rotating motor of the vibrator can be selectively rotated, at various speeds. By changing the speed of the rototing motor (vibrator) a wide range of frequencies can be transmitted to the feed by means of the rod and the linkage arm. A steady stream of powder at a selected rate can be delivered to the preheated boat. The temperature of the boat is controlled by a W-Re thermocouple which is directly welded to the boat.

PROCEDURE

The vacuum chamber is evacuated and purged several times with ultra pure argon. An argon pressure of 20μ is established by a variable leak valve. A potential of 3-5 kv is gradually applied across the specimen and boat (electrodes) and an argon discharge is established. The specimen is now sputter etched by the ionized argon. The surface of the specimen is thus cleaned from 20 minutes to several hours depending on the sputtering rate of the specimen. While a potential is across the electrodes and sputter etching is in progress, the boat is heated to a preselected temperature. For the Pb-Sn alloys the boat temperature was set

from 2,100 - 2,350° F depending on the particle size used and the delivery rate of the powder. For the Au-Cu alloy the temperature was set around 3,100° F. Once the desired temperature of the boat is reached the vibrator is initiated to transmit the preselected frequency to vibrate the powder feed. Uniform stream of powder drops down the feed at a constant rate. The temperature of the boat was adjusted so that the parricles upon falling into the boat could be seen to melt and then evaporate instantaneously. The temperature of the boat (in regard to a particular alloy used) and the evaporation rates should not be too high. The sudden evolution of the vapor blows the dropping powder away before reaching the boat when the temperature of the boat and the rate of powder delivery is too high.

After the alloy is evaporated the vapor is injected into the glow discharge. A high percentage of the evaporated atoms are ionized and accelerated to the specimen (cathode). These alloy ions plated by IPFE behave in the same manner as the elemental ions in ion plating. Plating is nondirectional, it plates into cavities and around corners. The adherence is exceptionally strong due to the high potential difference (high acceleration of the ions) between the specimen and the boat. Tensile tests and bend tests have shown that the coating flows with the bulk material without chipping or peeling.

RESULTS AND DISCUSSION

Three binary alloy systems with the following weight percentages were deposited by IPFE Pb-Sn (50-50), Pb-Sn (34-65), Cu-Au (85-15). The deposited alloy film was analyzed quantitatively by spectrographic methods, X-ray fluorescence and wet chemical analyses. The weight percentages of the constituent elements before and after plating are listed in Table 1. These analyses reveal that the close alloy composition can be maintained when deposited by IPFE. It should be realized that compositional changes may occur, since this strictly depends on the selected operational parameters. These parameters which have to be selected and closely controlled are particle size, rate and consistency of the powder delivery to boat and the temperature of the boat. When these deposition parameters are experimentally determined for a particular alloy, the composition of the plated alloy film will be very close to the original alloy. These IPFE plated alloy films have the same degree of adherence as elemental metal films plated by ion plating.

TABLE 1

QUANTITATIVE ANALYSES OF ORIGINAL ALLOY VERSUS PLATED FILM

Alloy	Original Composition Weight %	Composition of Film Weight %
Pb-Sn	48.24-48.10	49.61-50.39
Pb-Sn	33.87-65.42	36.78-64.20
Cu-Au	84.32-15.17	85.1 -14.8

SUMMARY AND RESULTS

A new vacuum deposition method ion plating with flash evaporation (IPFE) is introduced. By this method alloy films of the same composition as the original alloy can be deposited. Three binary alloys of the following weight percentages were successfully deposited: Pb-Sn (50-50), Pb-Sn (34-69), and Cu-Au (85-15). These films can be deposited on complex geometrical surfaces (into cavities and around corners) without rotating the specimen. Very strong adherence with a diffusion type interface between the film and surface is obtained, which is due to the highly penetrating effect of the plating material.

REFERENCES

1. Spalvins, Talivaldis; and Przybyszewski, John: Deposition of Thin Films by Ion Plating on Surfaces Having Various Configurations. NASA TN D-3707, 1966.

2. Mattox, D. M.: Design Considerations for Ion Plating. Rep. SC-R-65-997, Sandia Corp., Jan. 1966.

3. Richards, John L.; A Fundamental Study of Epitaxy by Flash Evaporation. Philco Corp. (AFCRL-63-524, DDC No. AD-426134), Sept. 30, 1963.

4. Harris, Louis; and Siegel, Benjamin M.: A Method for the Evaporation of Alloys. J. Appl. Phys., vol. 19, no. 8, Aug. 1948, pp. 739-741.

5. Richards, John: Flash Evaporation. The Use of Thin Films in Physical Investigations. J. C. Anderson, ed., Academic Press, 1966.

6. Campbell, D. S.; and Hendry, B.: The Effect of Composition on the Temperature Coefficient of Resistance of NiCr Films. Brit. J. Appl. Phys., vol. 16, no. 11, Nov. 1965, pp. 1719-1725.

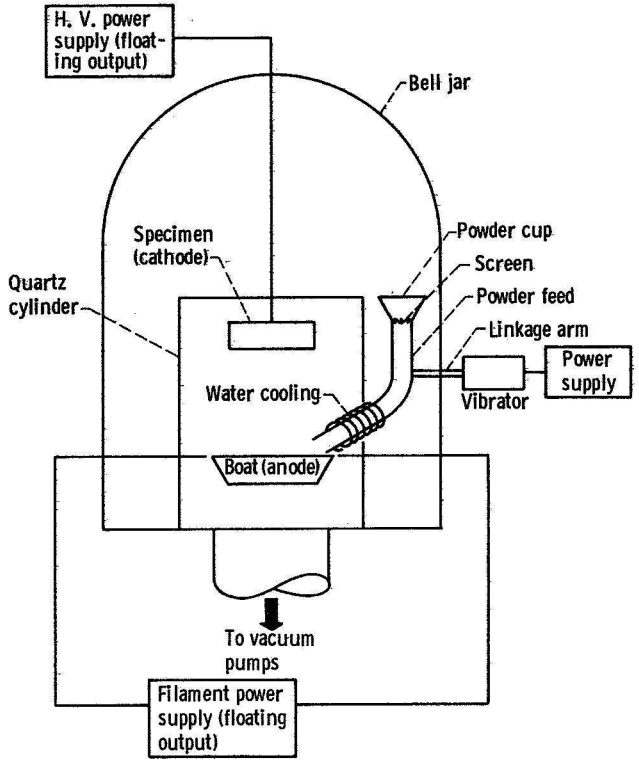

Figure 1(a). - Schematic of ion plating with flash evaporation (IPFE) apparatus.

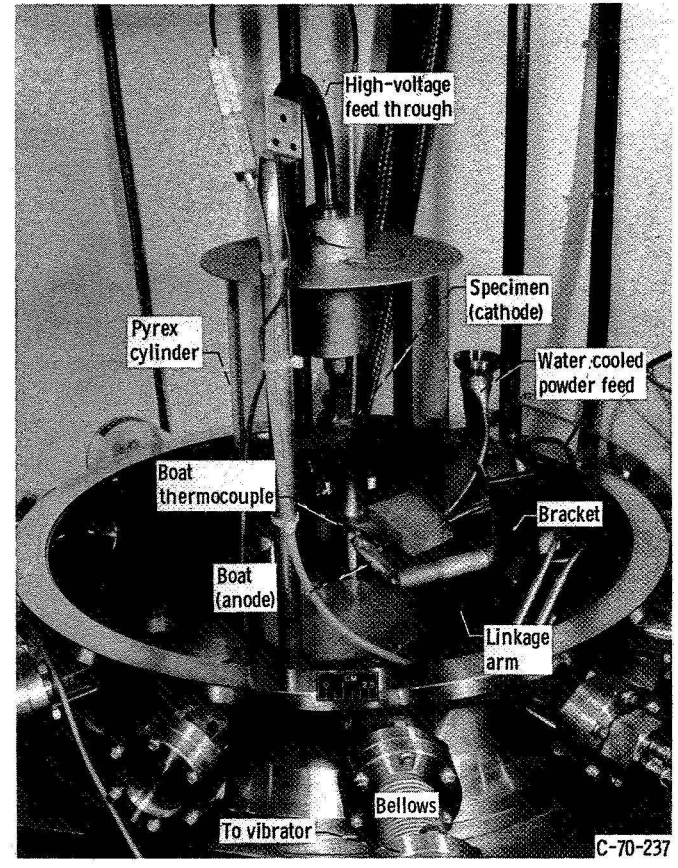

Fig. 1(b). - Ion plating with flash evaporation chamber.