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Comparison of Deposition Behavior and Properties of Cyanide-free Electroless Au Plating on Various Underlayer Electroless Ni-P films

Dong-Huyn Kim*

MSC Co., Ltd.

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Abstract

Internal connections between device, package and external terminals for connecting packaging and printed circuit board are normally manufactured by electroless Ni-P plating followed by immersion Au plating (ENIG process) to ensure the connection reliability. In this study, a new non-cyanide-based immersion and electroless Au plating solutions using thiomalic acid as a complexing agent and aminoethanethiol as a reducing agent was investigated on different underlayer electroless Ni-P plating layers.

As a result, it was confirmed that the deposition behavior and film properties of electroless Au plating are affected by grain size and impurity of the electroless Ni-P film, which is used as the plating underlayer. Au plating on the electroless Ni-P plating film with a dense surface structure showed the highest bonding strength. In addition, the electroless Au plating film on the Ni-P plating film has a smaller particle size exhibited higher bonding strength than that on the large particle size.

Keywords : Cyanide-free plating; Electroless Au plating; Immersion plating; Autocatalytic plating, Deposition rate; Solderability; Particle size.

1. Introduction

The recent development of the electronics industry requires miniaturization of the high functionality of electronic devices. To respond, refinement of components, miniaturization of substrates, isolation of circuits, narrower pitch, and various methods of bonding for semiconductor components are being made. In addition, there is an increasing demand for environmental friendliness and low carbon in the manufacture of electronic components. In particular, in semiconductor packaging technology, bonding technology, and mobile communication, high-integration and miniaturized components are required according to the trend of miniaturization, multi-band, and high frequency of mobile communication devices [1] ~ [6].

In the field of surface-mounting technology, surface treatment of a printed circuit board (PCB) has become very important to ensure connection reliability in internal connection of devices and packages, external terminals for connection between packaging and printed substrates, and directly mounting the bare chip on substrates without packaging [7].

Although various methods such as wire bonding, flip-chip, and bump are used for the connection method, when viewed from the terminal side, the main

surface treatment used two-layer of Ni/ Au plating through the electroless nickel and immersion gold process (ENIG). While circuits and connection terminals, including semiconductor devices, are being refined and narrow pitched, lead wires for electroplating are essential conventional electrolytic Au plating, and thus, the inductance of this part is hindered in high-speed signals. For this reason, the necessary electroless Ni/Au plating technology of lead wires is attracting attention as an important technology. Accordingly, the fields requiring electroless Au plating are diversified, and various types of plating solutions suitable for each property and application have been researched and developed to become practical [8], [9]. In addition, many studies have reported the effect of underlying layer on solder bonding in electroless Ni/Au plating as the P content, vacancy position in the Ni-P plating, and the deterioration of the surface of the underlying layer by immersion Au plating treatment on solder bonding [10], [11].

The reasons for the weakening strength of the solder joint include (1) weakening of the P-rich layer between the electroless Ni-P layer and the solder layer [8] ~ [12], (2) generating (Cu, Ni)₆Sn₅ intermetallic compounds (IMC) [10], [12], [13] that are very fragile between the Ni-P layer and the solder layer during solder bonding.

Conventional electroless Au plating baths are operated at a high pH bath, using gold potassium cyanide (KAu(CN)₂) as a metallic salt, potassium cyanide (KCN) as a complexing agent, sodium hypophosphate, dimethylamine borane (DMAB), or others as reducing agent. KCN and Au(I) cyanide complex, KAu(CN)₂, which are used as a complexing agent, play a very important role in ensuring the stability of the plating solution in electro- and electroless gold plating. However, cyanide is a highly toxic chemical, and cyanine-containing plating solutions are generally operated in the alkaline range, which can erode the photoresists and other materials used in the packaging of highdensity circuits. In addition, there is a problem in that bath stability is remarkably reduced due to the incorporation of a small amount of Ni_{2+} ions. Due to this difficulty, it is required to develop a new electroless Au plating bath that can operate at a neutral pH, it does not contain a cyanide compound but still ensures good stability [14] ~ [21].

It should be noted that two critical components of an electroless gold plating bath are a complex ligand to bond with gold ions and the reducing agent. The most stable complex with Aun+ ions is a CN with the standard reduction potential $E^0 = -0.65$ V (vs. SHE). Some other gold complexing agents such as AuCl²⁻, AuCl⁴⁻, Au(SCN)²⁻, Au(thiourea)²⁺, Au(S_2O_3)₂³⁻, Au(SO_3)₂³⁻ have the standard reduction potential of 1.15, 0.92, 0.67, 0.64, 0.38, 0.15 and 0.06 V (vs. SHE), respectively. The thio compounds (R-SH) exhibit the lower reduction potentials of a complex with gold among non-cyanide compounds, of about -0.5 to -0.6 V (vs. SHE), and are expected to replace the cyanide compound for complexing with gold ions in electro- and electroless gold plating bath [21] ~ [23].

A new type of non-cyanide electroless Au plating using thiomalic acid as a complexing agent and aminoethanethiol as a reducing agent has been reported together with its electrochemical properties and complex binding mechanisms [24] ~ [26]. However, in the ENIG process, the effect of the substrate materials, particularly the electroless Ni-P layer, on the properties of immersion and electroless gold complexed with thiomalic acid has not been reported. In this study, the properties of cyanide-free electroless gold on the four underlayer electroless nickel films were systematically investigated in deposition rate, surface properties, defects, adhesion, and solderability.

2. Experimental

The process of electroless gold on the four different underlayer electroless nickel films is presented in Fig. 1. First, a copper specimen with a size of 2.5×2.5 cm was degreased in an alkaline cleaning solution at 60 °C for 5 min, followed by acid cleaning in 100 g/L H_2SO_4 solution at R.T for 5 min. Next, the Cu sample was etched by 100 g/ $L Na_2SO_5 + 50 g/L H_2SO_4$ solution at R.T for 30 s, followed by pre-dipping treatment in 20 g/L H_2SO_4 solution at R.T for 30 s. The copper surface was then activated by Pd²⁺ ion in solution containing 20 ppm PdCl2 at R.T for 120 s. Next, electroless nickel film was formed on activated Cu sample from four different plating baths, named A, B, C, and D. The electroless nickel was conducted at 80 °C for 30 min to obtain a Ni-P film with a thickness of 5.0 μ m. The electroless Ni-P plating solutions contained 22.5 g Ni_2SO_4 ·6H₂O as a nickel ion source, 25 g/L NaH₂PO₂·H₂O as reducing agent, 18 g/L lactic acid as a complexing agent, 10 g/L CH₃COONa as a buffer, and some additives. Gold was then deposited on the electroless Ni coated samples through two coating processes: immersion Au and autocatalytic electroless Au plating. The immersion gold process to obtain gold film thickness of 0.05 μ m was conducted in a solution containing 0.01 M Au⁺, 0.27 M thiomalic acid, 0.15 M potassium hydrogen phosphate, and additives, at 80 °C for 20 min. The autocatalytic electroless Au plating solution contained 0.01 M Au⁺, 0.27 M thiomalic acid, 0.4 M 2-aminoethanol, 0.15 M hydrogen phosphate, and additives, temperature of 80 °C for 60 min, to achieve the Au film thickness of 0.6 μ m. The compositions of immersion Au and electroless Au were also listed in Tables 1 and 2.

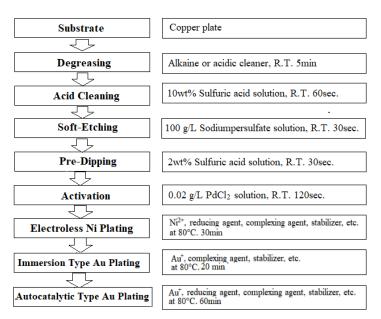


Fig. 1. Process of Electroless Ni/Au Plating

Table 1. Composition of immersion type Au plating solution and plating conditions.

Component	Concentration	
Au(I)Thiomalate	0.01 mol/L	
Thiomalic Acid	0.27 mol/L	
Potassium Dihydrogen Phosphate	0.15 mol/L	
pH at 25 °C	7.0	
Bath temperature	80 °C	
pH adjustment	КОН	

Table 2. Composition of autocatalytic Au plating	solution
and plating conditions.	

Concentration
0.01 mol/L
0.27 mol/L
0.40 mol/L
0.15 mol/L
7.0
80 °C
КОН

Tester	Dage Series 4000			
Method	Heated bump pull test			
Heating temperature	170 °C			
Heating time	10 s			
Cooling off	50 °C			
Full range	5000 gf			
Pull speed	300 µm/s			

Table 3. Conditions for testing solder ball bonding strength of solder ball.

The change in the concentrations of Au and Ni ions in the immersion gold plating solution was analyzed using inductively coupled plasma (ICP). The thickness of the deposited Au film was measured by X-ray fluorescence, the surface morphology and film composition were observed by scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDAX) analysis.

To confirm the soldering characteristics, the zero-crossing time and solder ball bonding strength were measured. As for the solder ball bonding strength, the high-temperature bonding strength between the solder ball and the Au film was measured by mounting a 0.75 mm Sn_{3.5}Ag_{0.7}Cu solder ball in a 0.40 mm pad-sized BGA package. Table 3 shows the conditions for testing the bonding strength of solder balls.

3. Result and discussion

3.1. Deposition behavior of immersion Au layer

The process of gold coating is followed: electroless nickel plating on copper substrate \rightarrow Immersion gold plating \rightarrow Electroless gold plating. In this case, immersion gold plating and electroless gold plating are affected by the deposition behavior or soldering properties of the electroless nickel plating bath, such as the type of nickel plating bath, the pretreatment of nickel plating, and under improper pretreatment conditions, defects such as discoloration and pinholes [24], [25]. Fig. 2 illustrates the correlation of various factors affecting the electroless Au plating film characteristics.

Fig. 3 shows the deposition rate and P content of electroless Ni-P film obtained from four different electroless nickel plating baths. The deposition rate was about 10 to 12 μ m/h at a plating temperature of 80 °C. The P content in the Ni-P alloy film obtained from the four kinds of electroless Ni-P plating baths was in the range of 6 to 8 wt.%. Fig. 4 presents the deposition rates of immersion Au plating and electroless Au plating on the four Ni-P coated Cu substrates. The thickness of Ni-P film was about 5.0 μ m. The immersion Au plating was conducted at 80 °C for 20 min, and then electroless Au plating was performed at 80 °C for 60 min.

When immersion Au plating is performed on the Ni-P alloy plating films obtained from different electroless Ni-P plating baths, the deposition rate of Au film proportional increases to increase the deposition rate of electroless nickel. This is believed to be because the higher the deposition rate of the electroless Ni-P plating provided, the larger the particle size on the surface, and in the case of the underlayer Ni-P film with large particle size, the grain boundary dissolution of the Ni-P film is faster during the immersion Au plating reaction.

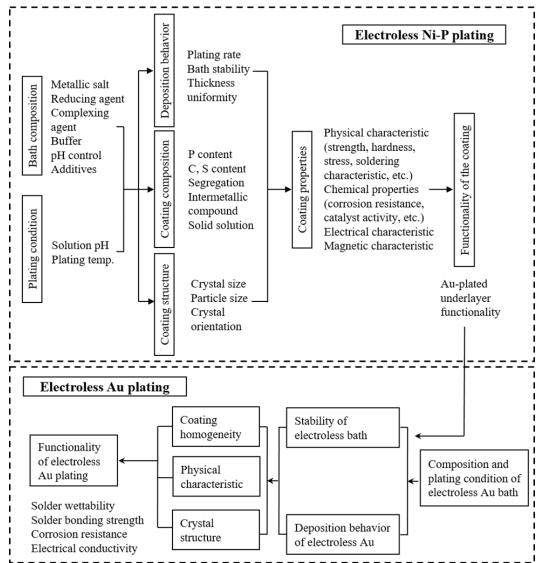


Fig. 2 Correlation of various factors of electroless Ni-P plating on the function of electroless Au plating film

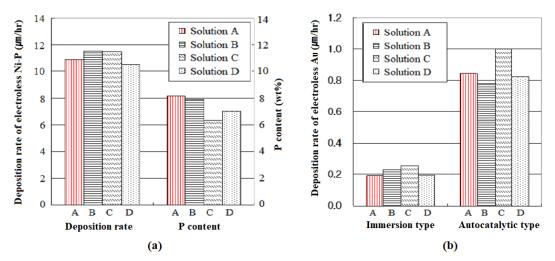


Fig. 3. Plating behavior of electroless Ni-P plating and electroless Au Plating.
(a) Deposition rate and P content of electroless Ni-P plating obtained from various different bath.
(b) Effects of under layer electroless Ni-P plating films on deposition rate of electroless Au plating.

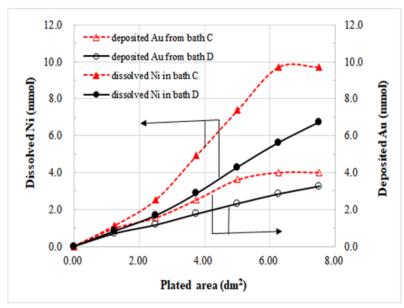


Fig. 4. Changes of consumed Au concentration and dissolved Ni concentration in immersion Au plating solution.

Electroless nickel plating bath D has a plating rate of 10.8 μ m/h and P content of 7 % exhibited the best performance for the immersion gold reaction compared to the other electroless Ni-P plating baths (A, B, and C).

Fig. 5 shows the results of analyzing the concentrations of Au consumed and Ni dissolved during immersion Au plating process on the Ni-P film obtained using the electroless nickel plating baths C and D. The Ni-P film thickness of about 5 μ m on both sides of the Cu specimen size of 2.5×2.5 cm², immersion in immersion Au

plating solution, and performing up to 60 sheets at 80 \degree for 20 min.

For immersion Au process with Ni-P film obtained from electroless plating bath C, the amount of Au consumption increased with increasing plated area up to 47.5 dm²; after that amount of Au consumption is constant, it means that the deposition of Au is stopped. However, Ni²⁺ concentration increases with increasing plated area up to 60 dm². For bath D, both the consumed Au and Ni dissolved continue to increase with increase plated area up to 80 dm². On the other hand, comparing the concentration

Ni soln.	А	В	С	D
SEM image	A16 - 100 - 104			
Deposition rate	10.89 µm/hr	11.52 µm/hr	11.46 µm/hr	10.52 µm/hr
P content	8.15 wt%	7.93 wt%	6.33 wt%	6.99 wt%
Mor- phology	Small particle size below 1.0 µm	Particle size from 2.0 µm to 5.0 µm	Smooth surface, black point	Smooth sur- face,very fine particle

Fig. 5. SEM images and film properties of electroless Ni-P plating films obtained from various different bath.

of Ni dissolved and the amount of Au deposited of immersion Au from bath C and D, it can be seen that the Ni-P deposited from bath C has a larger amount dissolved than that of bath D, even in the same amount of Au deposited.

Considering the exchange plating reaction in which dissolved metal ions in the plating solution are deposited by receiving electrons, the oxidation reaction of Ni \rightarrow $Ni^{2+} + 2e^{-}$ generated two-electron, and the reduction reaction of $Au^+ + e^- \rightarrow Au$ consumed one-electron. Thus, theoretically, to reduce 2 moles of Au+ needs 1 mole of Ni; however, as seen in Fig. 5, it needs 1.2 to 2.2 moles of Ni oxidized to deposit 1 mole of Au⁺. This suggests that electrons generated by Ni oxidation are not only used for reducing Au⁺ ions but also for oxidation of other components such as a complexing agent, an additive, or others during the immersion gold plating process.

This is because the Ni-P alloy film having a larger particle size on the surface lowers the oxidation potential of the Ni-P film in the exchange Au plating bath, tends to lower corrosion resistance, and increases the oxidation reaction (dissolution) rate of nickel. This particle size also has a relationship with the content of P in the Ni-P alloy film, and as the content of P increases, the grain size of the film tends to decrease. Although this correlation can indeed be obtained from many plating baths other than exchanged Au plating, the addition of additives in the electroless Ni-P plating bath developed for use as a base film for electroless Au plating, etc. In some cases, the physical properties of the Ni-P alloy film may be controlled, resulting in different results from this tendency. This is because the electroless Ni-P plating film for the base of the electroless Au plating is a medium P-type (P content of about 6 to 10 wt.%) in Ni-P alloy plating film and has a microcrystalline film structure. Alternatively, the dissolution rate in the immersion Au plating bath must be suppressed, and an appropriate bath composition and additives must be selected to control the crystal structure or dissolution potential of Ni-P film.

As for the film in which the grain size is controlled, the difference can be seen in Fig. 6. In electroless nickel baths A and B, the P content is almost the same as about 8.0 wt.%, but the grain size of the film obtained from bath A (1.0 μ m or lower) is smaller than the grain size of the film obtained from bath B (about 2.0 μ m to 5.0

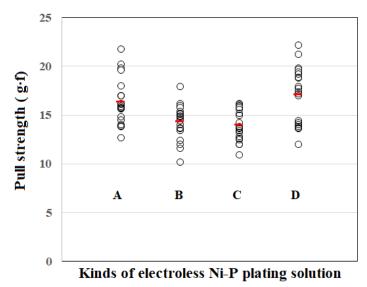


Fig. 6. Comparison of solder ball bonding strength of immersion type Au plating film on various electroless Ni-P plating films.

µm). In addition, the content of P in the film obtained from the bath D is about 6.99 wt.% but exhibited a smooth surface structure and fine particles. Although it is an alloy composition in which a film with a large particle size is generally easy to obtain, it shows the fine particle and smooth surface structure. On the other hand, in the film obtained from bath C, the addition of additives is believed to appear as black spots.

Fig. 7 compared bonding strength after electroless Au plating on different electroless Ni-P films, where electroless Au was conducted on 0.05 μ m immersion Au layer. From this result, the bonding strength is high in the order of bath D \rangle bath A \rangle bath B \rangle bath C, and the case where the electroless Ni-P film obtained from bath D showing a dense surface structure is the highest bonding strength. In association with the result of the P content of the electroless Ni-P film, the bonding strength of the Au plating on the nickel film from bath C with the lowest P content is lower than that of bath A or B, which have higher P content.

In general, when Au plating is performed on a Ni-P film having a high content of P, the P-rich layer is easily formed by diffusion of Ni into the solder layer or formation of an intermetallic compound with Sn, that is, the bonding strength of the solder is reduced [10], [11]. On the other hand, considering the result of the bonding strength in relation to the surface state of the electroless Ni-P film, the Au plating on the Ni-P film obtained from the bath D showed a dense surface structure has the highest bonding strength. The Au plating film on bath A with a small particle size showed higher bonding strength than the film from bath B with relatively larger particle size.

The bond strength of the electroless Au plating film plated on the Ni-P film from bath C, where impurities are visible on the surface, is the lowest value. Thus, it would be a problem to evaluate the ENIG process only by the P content of the electroless nickel layer due to the significant impact of the impurities on the bonding strength.

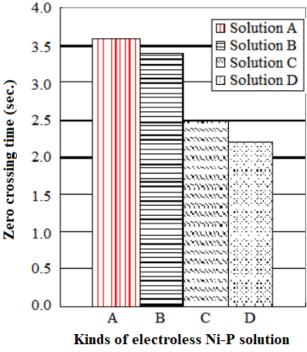


Fig. 7. Comparison of zero crossing time of immersion type Au plating film on various electroless Ni-P plating films.

To select the best electroless Ni-P plating film, it was confirmed that a film must have dense crystal grains, exhibiting some high solubility resistance to exchange with gold metal plating bath, and homogeneous dissolution of Ni was preferred.

In addition, the solder wettability of the immersion-type Au plating film was measured with zero-crossing time, and the results are shown in Fig. 8. From the result of the zero-crossing time of the Au plating film, it can be seen that the Au plating on the Ni-P film obtained from bath D, which has a dense surface structure, showed the best solder wettability. However, in the case of thick Au plating of the coating on Ni-P film from bath C, where impurities are visible on the surface, the solder wettability is decreased. It can be seen that the value of the zero-crossing time was lower than that of the Au plating film on the obtained Ni-P plating film, indicating that the solder wettability did not deteriorate. Comparing the underlying layer from the results of Figs. 7 and 8, the solder ball bonding strength of the immersion-type Au plating film in the order of bath D \rangle bath A \rangle bath B \rangle bath C. The zero-crossing time of the immersiontype Au plating film is good in the order of bath D \rangle bath C \rangle bath A \rangle bath B. These

results suggest that the excellent wettability of the solder does not necessarily mean that the solder joint strength is also strong. In recent electronic component mounting technology, bonding by solder of a limited diameter on a limited pad area, such as ball grid allay (BGA) or bump of flipchip, the bonding strength is evaluated as more important in terms of reliability than wettability.

Fig. 9 presents EDAX analysis of bonding interface after mounting and reflow of solder ball on immersion type Au plating films after using a solder ball with a composition of SnAg3.5Cu0.7 mounted on the electroless Au plating on the different electroless Ni-P baths. According to this result, almost all Au plating films are diffused into the Sn-containing layer of solder, and are not observed on the surface of the electroless Ni-P plating layer. When using electroless nickel bath C, the P-rich layer was observed mainly at the interface between the Ni-P alloy and the solder layers. Even when using electroless nickel bath A, the P-rich layer still can be seen. However, the P-rich layer was not observed when used the electroless Ni-P plating bath B or D.

When this result is considered about

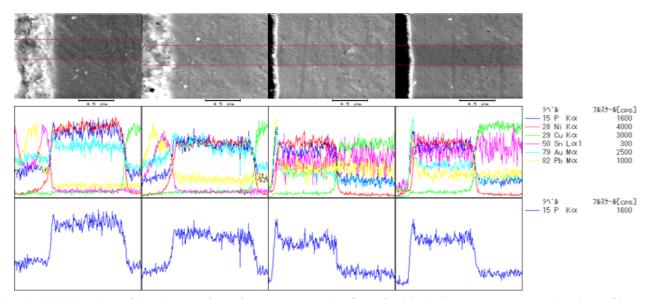


Fig. 8. EDAX analysis of bonding interface after mounting and reflow of solder ball on immersion type Au plating films.

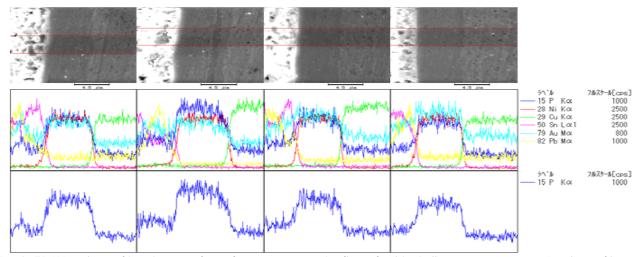


Fig. 9. EDAX analysis of bonding interface after mounting and reflow of solder ball on immersion type Au plating films.

the soldering as mentioned above, the soldering characteristics are not weakened in baths A and C, where the formation of the P-rich layer is observed. The particle size and grain boundary corrosion of the underlayer Ni-P plating film greatly influence the soldering characteristics. It can be considered that the particle size and intergranular corrosion of the film have a more significant influence on the soldering properties. Particularly, when electroless Ni-P from bath C was used, the results of the zero-crossing time show good characteristics, but the bonding strength shows the weakest results. From the SEM image of Fig. 6, the Ni-P plating film from bath C has a small particle size so that the grain boundary is not reliably distinguished, and black spots are observed on the surface. In this way, the zero-crossing time is good when the particle size is small. However, still, it is known that the organic component vacated in the film, particularly the S-containing organic material, increases the dissolution of the Ni-P film in the immersion Au plating and forms a black pad after Au plating [1],[2],[11],[24].

Fig. 10 displays EDAX analysis of bonding interface after mounting and reflow of solder ball on electroless-type Au plating films after using a solder ball with a composition of SnAg_{3.5}Cu_{0.7} mounted on

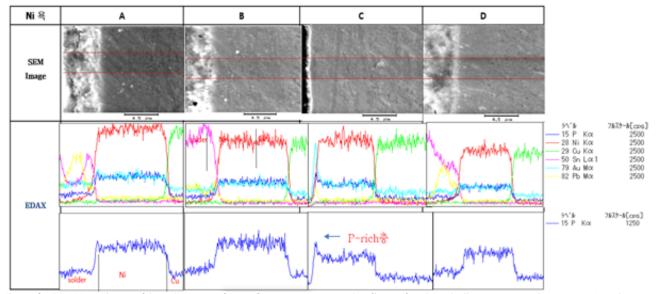


Fig. 10. EDAX analysis of bonding interface after mounting and reflow of solder ball on autocatalytic type Au plating films.

the electroless Au plating on the different electroless Ni-P baths. Although the electroless type Au plating film thickness the soldering as mentioned above, the soldering characteristics are not weakened in baths A and C, where the formation of the P-rich layer is observed. The particle size and grain boundary corrosion of the underlayer Ni-P plating film greatly influence the soldering characteristics. It can be considered that the particle size and intergranular corrosion of the film have a more significant influence on the soldering properties. Particularly, when electroless Ni-P from bath C was used, the results of the zero-crossing time show good characteristics, but the bonding strength shows the weakest results. From the SEM image of Fig. 6, the Ni-P plating film from bath C has a small particle size so that the grain boundary is not reliably distinguished, and black spots are observed on the surface. In this way, the zero-crossing time is good when the particle size is small. However, still, it is known that the organic component vacated in the film, particularly the S-containing organic material, increases the dissolution of the Ni-P film in the immersion Au plating and forms a black pad after Au plating [1],[2],[11],[24].is about 0.6 μ m, compared to the 0.05 μ m of immersion type Au film, almost all Au is diffused into the solder layer, so that the electroless Ni-P plating high solder layer is not observed at the interface. In addition, when soldering is performed after electroless Au plating, the P-rich layer is not observed in any plating bath regardless of the type of the electroless Ni-P plating solution used in the Ni-P plating process of the underlayer. As described above, when the thickness of the Au plating film is sufficiently thick, the P-rich layer is not observed at the bonding interface with the solder layer. This result shows that as the Au plating thickness increases, a petal-shaped aggregate is increased in the solder layer. The formation of Au6Sn5 intermetallic compounds forms the petalshaped aggregate, and the increase of the intermetallic compounds is known to cause a decrease in solder bonding strength [27] ~ [30]. Therefore, since it is industrially bonded to solder in ENIG processes such as printed circuit boards, the Au plating thickness is set to 0.05 μ m, and in wire bonding to which Au wire is bonded, the Au plating thickness is increased to about 0.3 μ m.

4. Conclusion

A new non-cyanide-based electroless Au plating solution using thiomalic acid as a complexing agent and aminoethanethiol as a reducing agent was deposited on four electroless Ni-P plating layers having different crystal structures. The effects of the electroless Ni-P plating film used as the base layer on the deposition behavior and film properties of immersion type and electroless type Au plating were investigated. The following conclusions were reached.

- The crystal structure of the electroless Ni-P plating film used as the underlying layer greatly affected on the deposition rate and soldering characteristics, particularly the deposition behavior of the immersion Au plating.
- 2) When a film obtained in e an electroless nickel bath has a higher deposition rate, the immersion Au plating deposition rate tends to increase. Also, the Ni-P alloy film having a larger particle size tends to have the lower corrosion resistance of the Ni-P film in the immersion-type Au plating bath. Since the oxidation reaction (dissolution) rate of nickel increases, the immersion-type Au plating deposition rate becomes higher.
- 3) Au plating on an electroless Ni-P plating film with a dense surface structure and

a smaller grain size showed the highest bonding strength. On the other hand, even in a Ni-P plating film having a small grain size, the bonding strength of the electroless Au plating film plated on the Ni-P film having impurities on the surface shows the lower value.

There is a problem in evaluating electroless Ni-P plating used as the underlying layer simply with the content of P. To select the best underlying electroless Ni-P plating film, the crystal grains are dense, it has a high degree of dissolution resistance to the immersion precious metal plating bath, and homogeneous dissolution over the entire surface of the Ni-P film in the substituted Au plating solution is possible. It was confirmed that the film formed was good.

4) The soldering properties of the Au film on the electroless Ni-P plating film, where the P-rich layer is observed, are not necessarily weakened, and the particle size and grain boundary corrosion of the underlayer Ni-P plating film has a more significant influence on the soldering characteristics.

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