[Technical Paper]

Influences of Electroless Nickel Film Conditions on Electroless Au/Pd/Ni Wire Bondability

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Abstract

Electroless Au/Ni plating is intensively applied to high-density printed boards. In this process, local corrosion often occurs between the deposited nickel and the deposited gold. Generally, nickel tends to diffuse from the local corroded areas to the deposited gold surface after thermal treatment due to its strong affinity for oxygen. These areas cause surface mounting failures. Recently, electroless Au/Pd/Ni plating has been actively studied as a substitute for electroless Au/Ni plating because it suppresses the nickel corrosion reaction. In this study, we investigate the influence of the nickel microstructure and the thickness of the palladium and gold on wire bondability. The wire bonding strength is increased with increased palladium and gold film thickness. The deposited nickel microstructures also influence the wire bonding properties after thermal treatment. It was confirmed that good wire bonding properties can be achieved using a nickel film with a layered microstructure rather than a columnar microstructure. From the AES analysis, we confirm that preparation of a uniform layered microstructure of the nickel film is a key factor to keep the gold concentration on the gold film surface after thermal treatment.

Keywords: Electroless Au/Pd/Ni plating, Diffusion, Nickel Film Condition, Wire Bondability

1. Introduction

Gold has excellent electrical properties and chemical stability. Therefore, gold plating is used for the final surface finishing of various electronic parts. Recently, electroless Au/Ni plating process has been applied to the copper patterns on high-density printed boards. In this plating process, local nickel corrosion occurs between the deposited nickel and gold films. Nickel tends to diffuse into the deposited gold surface at the locally corroded area after thermal loading such as occurs during soldering.[1–5] These diffused areas often cause surface mounting failures. Therefore, the gold film thickness has to be increased since the diffusion of nickel occurs more easily with decreasing gold film thickness. However, for the purpose of reducing costs, gold films in electronics devices should be thin. To overcome these issues, we investigated electroless Au/Pd/Ni plating as a substitute for electroless Au/Ni plating.[6–9]

We focused on the relationship between wire bondability and the Pd/Au film thickness, and on the influence of

the deposited nickel film conditions (ex. Nickel deposition structure, phosphorus content) on wire bondability. Immersion potential was measured to study the depositing electroless palladium and immersion gold depositing behaviour.

2. Experimental Details

2.1 Preparation of substrate for wire bonding

In this study, PCB bonding pads and solder ball pads are used to evaluate the wire bonding properties. The electroless Au/Pd/Ni plating process is shown in Table 1. After preparation of the substrate using a conventional pre-treatment, a 5 μm -thick electroless nickel film was deposited on the Cu, followed by the deposition of electroless palladium and gold films, each 0.05 to 0.2 μm thick. The basic compositions and operating conditions of the electroless nickel, electroless palladium, and electroless gold plating baths are shown in Table 2. In this study, immersion-type and auto-catalytic-type baths are used for the electroless gold plating. The rinsing treatment

was performed by using ion-exchanged water between each process.

Table 1 Electroless Au/Pd/Ni plating process.

Degreasing treatment (CL-120 (Sanyo Chemical ind.) 2 g/dm ³ , NaH ₂ PO ₄ •2H ₂ O 10 g/dm ³ , 40°C, 4 min.)
↓
Soft etching treatment (Na ₂ SO ₃ 125 g/dm ³ , H ₂ SO ₄ 10 mL/dm ³ , 30°C, 1 min.)
↓
Acid rinsing treatment (10 vol.-%H ₂ SO ₄ , 30°C, 1 min.)
↓
Pd catalyzing treatment (PdCl ₂ 0.1 g/dm ³ , 30°C, 1 min.)
↓
Electroless Ni Plating (Table. 2-1 to 4, Ni : 5 μm)
↓
Electroless Pd Plating (Table. 2-5, Pd : 0.05 to 0.2 μm)
↓
Immersion Au Plating (Table. 2-6, Au : 0.05 to 0.1 μm)
↓
Auto catalytic Au Plating (Table. 2-7, Au : 0.1 μm over)

2.2 Characteristics of deposited nickel film

The electroless nickel film microstructure affects the subsequent deposition film morphologies and wire bondability. Therefore, electroless nickel with various microstructures was plated on the copper patterns in this study. We selected the complexing agents to control the nickel film microstructure. Nickel films with various phosphorus contents were also prepared by changing the concentration of sodium hypophosphite. As shown in Fig. 1, the deposited nickel films display a columnar structure

Fig. 1 SEM images of nickel microstructure on various electroless Ni plating solutions.

Table 2 Basic plating bath compositions and operating conditions.

Electroless nickel plating				
Ni plating bath	(1)Bath A	(2)Bath B	(3)Bath C	(4)Bath D
NiSO ₄ •6H ₂ O	1.0 mol/dm ³			
Complexing agents	Lactic acid and Succinic acid 0.2 mol/dm ³ and 0.1 mol/dm ³		Lactic acid and Malic acid 0.2 mol/dm ³ and 0.1 mol/dm ³	
Pb(NO ₃) ₂	0.2 ppm (as Pb concentration)			
NaH ₂ PO ₂ •H ₂ O	0.25 mol/dm ³	0.28 mol/dm ³	0.25 mol/dm ³	0.28 mol/dm ³
Bath pH and Temperature	4.5 and 85°C			

(5)Electroless Pd Plating		(6)Immersion type Au Plating		(7)Auto catalytic type Au Plating	
PdCl ₂	0.01 mol/dm ³	KAu(CN) ₂	0.01 mol/dm ³	Na ₂ [Au(SO ₃) ₂]	0.01 mol/dm ³
NH ₂ CH ₂ CH ₂ NH ₂	0.08 mol/dm ³	K ₃ (C ₆ H ₅ O ₇)•H ₂ O	0.08 mol/dm ³	Na ₂ SO ₃	0.1 mol/dm ³
HCOOH	0.3 mol/dm ³	[CH ₂ N(CH ₂ COOH) ₂] ₂	0.3 mol/dm ³	Na ₂ SO ₃	0.1 mol/dm ³
pH	7.0	TISO ₄	1ppm (as TI)	[CH ₂ N(CH ₂ COOH) ₂] ₂	0.1 mol/dm ³
Temperature	60°C	pH	4.5	L-Ascorbate	0.25 mol/dm ³
		Temperature	90°C	TISO ₄	3 ppm (as TI)
				pH	7.0
				Temperature	60°C

when using lactic acid and succinic acid mixed bath as the complexing agents (Baths A and B). On the other hand, the deposited nickel films have a layered microstructure when prepared using lactic acid and malic acid mixed bath (Baths C and D). Electroless nickel films containing 6 wt% or 8 wt% of phosphorus were deposited by changing the concentration of sodium hypophosphite.

2.3 Evaluation of wire-bonding properties

The wire-bonding strength and wire failure mode were evaluated before and after thermal treatment (200°C, 1 hour). Gold wire 28 μm in diameter was used for the evaluation of wire-bonding strength. The bonding loads were 50 gf for 50 msec at the ball-bonding side and 85 gf for 60 msec at the wedge side. The bonding stage temperature was set at 125°C. The wire-bonding strength was evaluated using a wire pull tester at a pull speed of 12.5 mm/min. Schematic diagrams of the gold wire failure mode are shown in Fig. 2. The fracture mode of the gold wire was observed using a stereomicroscope after the bonding strength measurement.

2.4 Analysis of electroless plated Au/Pd/Ni films

The surface morphology and surface roughness of electroless Au/Pd/Ni plated films were observed with atomic force microscopy (AFM, SPI4000, SII). The behaviour of local nickel corrosion was observed by two methods. The nickel film surface was observed using field emission scanning electron microscopy (FE-SEM, JSM-7000F, JEOL) after removing the deposited gold and palladium films with a cyanide-type remover (GSS-7P, Kojima Chemical Co., Ltd.). The cross-sectional views were observed by FE-SEM after preparation with a cross-section polisher (CP, SM-09010, JEOL). The thermal diffusion conditions of the electroless Au/Pd/Ni films were observed with an auger electron spectroscopy analyzer (AES, JAMP-7810, JEOL). The surface hardness of the electroless Au/Pd/Ni film was measured using the Martens hardness test (Nano Indentation Tester, ENT-1100a, ELIONIX Inc.). In this study, the measurement load was set at 0.1gf to examine the surface hardness to a depth of about 70 nm.

Fig. 2 Schematic diagram of gold wire failure mode.

2.5 Observation of palladium and gold plating behaviour

Immersion potential was measured to study the depositing electroless palladium and immersion gold depositing behaviour. For the measurement of the initial electroless palladium plating behaviour, each nickel deposited copper electrode was dipped in the electroless palladium plating solution, and the initial electroless palladium plating reaction was observed. To observe the immersion gold plating reaction behaviour, the electroless Pd/Ni plated copper electrode was immersed in the immersion gold plating solution and the initial electroless gold plating reaction behaviour was recorded.

3. Results and Discussion

3.1 Relationship between wire bondability and Au/Pd films thickness

We investigated the wire bonding strength by changing the thickness of the electroless Au and Pd plating films in order to understand the relationship between wire bondability and Au/Pd film thickness. In this study, we used Bath A as the electroless nickel plating bath with 6 wt% phosphorus content. The deposited nickel showed a columnar structure. Palladium and gold were deposited on the nickel plated copper patterns using auto-catalytic type electroless palladium plating, immersion type electroless gold plating, and auto-catalytic type electroless gold plating. Firstly, we investigated the relationship between wire bondability and the electroless gold film thickness. A 5 μm -thick electroless nickel film and a 0.05 μm -thick electroless palladium film were plated, and subsequently, electroless gold films of 0.05 to 0.2 μm were plated. After thermal treatment (200°C, 1 hour, in air), wire bondability was evaluated. As shown in Fig. 3, the wire bonding strength increases with increasing thickness of the deposited gold film, and the dispersion of wire bonding

Fig. 3 Influence of gold film thickness for wire bonding properties after thermal treatment.

strength values becomes small. When the gold film thickness exceeded $0.1\ \mu\text{m}$, a gold wire test indicated that 80% of failure modes were type C. From these results, it is confirmed that the thickness of the deposited gold film affects the wire bonding strength. Secondly, we examined the relationship between the wire bonding properties and the palladium film thickness. As evaluation samples, the thickness of the palladium was varied from 0.05 to $0.2\ \mu\text{m}$ on the $5\ \mu\text{m}$ -thick nickel plated copper patterns, and followed by the $0.05\ \mu\text{m}$ gold immersion plating. For a palladium film thickness of $0.05\ \mu\text{m}$, wire bonding strength values showed a large dispersion as shown in Fig. 4. On the other hand, with palladium film thicknesses over $0.1\ \mu\text{m}$, dispersion became small and the wire bonding strength showed consistent results. Also, the type C gold wire failure mode increased with increasing palladium film thickness. From these results, it is confirmed that electroless palladium is a key factor to improve the wire bonding strength.

3.2 Influence of electroless nickel film microstructure on wire bondability

From the above results, wire bondability was degraded with thinner gold and palladium plating ($0.05\ \mu\text{m}$). However, thinner gold and palladium plating is required for cost reduction of the electroless Au/Pd/Ni plating process. We have reported that the wire bondability is also affected by the nickel microstructure of the electroless Au/Ni plating.[10] Accordingly, we investigated the influences of the electroless nickel microstructure on the wire bondability with electroless Au/Pd/Ni plating. The wire bondability values using various electroless nickel-plating baths are shown in Fig. 5. All samples showed excellent wire bonding strength before thermal treatment. On the other hand, both the wire bonding strength and the range of wire bonding strengths were decreased after thermal treatment. After thermal treatment, the electroless nickel

microstructure influenced the wire bondability. Good wire bonding strength was obtained with the layered nickel microstructure but not the columnar nickel microstructure. From these results, we obtained excellent wire bondability by selecting a suitable electroless nickel plating solution, even with thinner gold and palladium plating ($0.05\ \mu\text{m}$). It is suggested that wire bondability is influenced by the nickel microstructure.

3.3 Observation of electroless Au/Pd/Ni plating film after thermal treatment

Generally, local nickel corrosion, the gold concentration on the deposited gold surface, the deposited film morphology, and the surface hardness are reasons for differences of wire bondability. Accordingly, we analyzed the electroless Au/Pd/Ni film before and after thermal treatment to examine the correlation of the electroless nickel film microstructure and wire bondability. Firstly, we observed the local nickel corrosion condition after immersion gold plating. Figs. 6-I and II show the nickel surface morphology after removing the gold and palladium deposited films, and a cross section of the electroless Au/Pd/Ni film before thermal treatment with the columnar nickel microstructure. The local nickel corrosion area was not detected with the thinner gold and palladium plating (each thickness was $0.05\ \mu\text{m}$). As shown in Fig. 6-III, nickel was not detected on the gold film surface after thermal treatment. However, the palladium peak was detected at the gold film surface. Secondly, we analyzed the thermal diffusion conditions between gold, palladium, and nickel before and after thermal treatment. These results are shown in Fig. 7. Changes in the thermal diffusion conditions were not observed on all samples. Therefore, Bath A was chosen as a reference for measurement of the AES depth profile before thermal treatment. After thermal treatment, with the electroless Au/Pd/Ni film, the diffusion reaction between gold and palladium tends to develop more with the electroless nickel film having a columnar microstructure than with

Fig. 4 Influence of palladium film thickness for wire bonding properties after thermal treatment.

Fig. 5 Influence of nickel film microstructure for wire bonding properties after thermal treatment.

Fig. 6 Observation of nickel local corrosion after electroless Au/Pd/Ni plating (Au/Pd are 0.05 μm, respectively). I: SEM image after peeled off Au and Pd film. II: Cross sectional image of electroless Au/Pd/Ni film. III: AES analysis of electroless Au/Pd/Ni film surface after thermal treatment.

Fig. 7 Thermal diffusion conditions between Au and Pd after heat treatment.

that having a layered microstructure. Especially when using Bath A or B as an electroless nickel-plating solution, thermal diffusion occurred between the gold and palladium. Therefore, we examined the ratio of gold and palladium on the plated gold surface after thermal treatment. As shown in Fig. 8, for the columnar nickel microstructure, the ratio of gold and palladium on gold film surface was about 45% to 55%. On the other hand, for the layered nickel microstructure, the gold and palladium ratio was about 50% to 50%. The ratio of gold was increased by 5% for the electroless nickel film with a layered microstructure. From these results, we speculated that the thermal diffusion is influenced by the morphology, the roughness, and the surface hardness of the electroless Au/Pd/Ni plating film. Therefore, we observed the surface morphology of the electroless Au/Pd/Ni film after thermal treatment. AFM

Fig. 8 Existence ratio of gold and palladium on gold film surface after thermal treatment.

images are shown in Fig. 9. Surface morphologies differed on various samples because these morphologies originated from the deposited electroless nickel film surface. However, significant differences in roughness were not seen on all samples. Subsequently, we investigated the sur-

Fig. 9 Surface morphologies and surface roughness of electroless Au/Pd/Ni film after thermal treatment.

Fig. 10 Surface hardness of electroless Au/Pd/Ni film after thermal treatment.

face hardness of the electroless Au/Pd/Ni film before and after thermal treatment using the Martens hardness test. As shown in Fig. 10, Martens hardness values increased after thermal treatment on all samples. The surface hardness of the deposited Au/Pd/Ni films increased with increasing phosphorus content of the nickel film. However we could not find significant differences in the surface morphology and surface hardness of the electroless Au/Pd/Ni films.

From these results, it can be seen that wire-bonding strength values decrease with decreasing thickness of gold and palladium because the palladium concentration on the gold film surface is increased by thermal diffusion after heat treatment.

3.4 Observation of palladium and gold plating behaviour

As the above results show, the properties of electroless Au/Pd/Ni plated film after thermal treatment changed depending on various nickel film microstructures. Therefore, we investigated the initial plating reaction behavior

Fig. 11 Immersion potential behavior of electroless Pd plating on various deposited Ni films.

on electroless palladium plating and immersion gold plating to understanding the mechanism of thermal diffusion between gold and palladium. Firstly, to observe the influence of palladium, the initial deposition behavior of palladium was measured using nickel films with a columnar microstructure and with a layered microstructure. The electrode potential was measured using plated nickel on a copper electrode. The working electrode is immersed in the electroless palladium plating solution. Fig. 11 shows the potential shift on the plated nickel surface. The immersion potential of the nickel film with the layered microstructure changed more rapidly than that of the nickel film with the columnar microstructure. It is anticipated that the deposited thin film of palladium has many pinholes due to the columnar microstructure of the nickel. Therefore, the surface elements of the plated palladium film that is deposited on each type of nickel film were analysed by AES. As shown in Fig. 12, nickel peaks were not detected on the deposited palladium film surface. From these results, it appears that the potential of the palladium plated onto the layered nickel reached the immersion potential of palladium because the deposited palladium film was uniformly distributed. On the other hand, for the columnar nickel, reduction of the palladium ion and oxidation of the dissolved nickel film occurred on the working electrode surface because of local displacement reactions. Therefore, for thin layers of palladium ($0.05\text{ }\mu\text{m}$), the electrode potential showed more negative due to the exposure of nickel at the local corrosion area. Accordingly, we examined the electrode potential using the deposited palladium film on each type of nickel microstructure to observe the influence of the initial gold deposition behaviour on the palladium. As shown in Fig. 13, the immersion potential gradually decreased and stabilized at -210 mV (gold plating time of 90 sec.) for the layered microstructure nickel. On the other hand, with the columnar structure, the electrode

Fig. 12 AES analysis of Pd film surface after thermal treatment.

Fig. 13 Immersion potential behavior of immersion Au plating on each electrodes Pd/Ni films.

potential was shifted in the negative direction and reached -350 mV at 30 sec of gold plating time. The immersion potential decreased and then gradually increased with gold plating time. Finally, the immersion potential reached about the same working electrode potential with the layered microstructure nickel film. With the columnar microstructure nickel, gold is rapidly deposited around the local corroded area. Therefore, it causes non-uniform deposition of gold. On the other hand, with the layered microstructure nickel, immersion gold plating progresses gradually. Therefore, uniform gold deposits can be obtained.

From these results, as shown in Fig. 14, the electroless palladium deposition reaction occurs uniformly on the nickel film with a layered microstructure. On the other hand, for the nickel film with a columnar microstructure,

Fig. 14 Schematic illustration of the mechanism of Pd diffusion for Au film surface at electroless Au/Pd/Ni film.

palladium displacement deposition progresses more aggressively on the nickel films compared to that with the layered microstructure due to the porous deposited surface. Furthermore, whereas the immersion gold deposited grains were distributed uniformly on the palladium surface after deposition of the layered microstructure nickel, for the nickel with a columnar structure, the grain distribution of deposited gold becomes disordered due to the unevenness of the gold grain size. For the above-mentioned reason, we conclude that diffusion between the gold and palladium on the columnar nickel microstructure proceeded more aggressively after thermal treatment than that on the layered nickel microstructure.

4. Conclusion

The following findings resulted from our investigation of electroless Au/Pd/Ni plating process for wire bondability.

- (1) Excellent wire bondability was obtained when the thicknesses of the palladium and gold films are both over $0.1\ \mu\text{m}$.
- (2) Wire bondability was degraded with decreasing thickness of the gold and palladium films due to the thermal diffusion between gold and palladium.
- (3) Good wire bondability was obtained when an electroless nickel film with layered microstructure was applied. Excellent wire bondability was obtained with the electroless nickel plating solution even with thin layers of gold and palladium ($0.05\ \mu\text{m}$).
- (4) We confirmed that the preparation of a nickel film with a uniform layered microstructure is a key factor in maintaining the gold concentration on the gold film surface after thermal treatment.

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