New Immersion Gold Technology for Uniform Au Thickness Distribution

Jian Zhang*, Ivan Hsu^ and Crystal Li*

Rohm and Haas Electronic Materials Asia Ltd (a Member of The Dow Chemical Company)

15 On Lok Mun Street, On Lok Tsuen, Fanling, N.T. Hong Kong*

Rohm and Haas Electronic Materials Taiwan Ltd., The Dow Chemical Company

No.6, Lane 280, Chung Shan North Road, Ta Yuan Hsiang, Taoyuan Hsien, Taiwan (R.O.C)^

jzhang23@dow.com

IMPACT Conference Proceedings, October 2016.

ABSTRACT

Electroless Nickel Immersion Gold (ENIG) is one of the important final finish techniques that is used in the printed circuit board industry. Despite its relatively expensive cost compared to other final finishing processes such as OSP and immersion Tin etc., ENIG has many advantages. The low reactivity of Au can protect the underlying Ni and Cu surfaces from oxidation and keeps the board suitable for long storage time. It provides excellent surface planarity suitable for soldering, especially for tiny parts such as BGA and Flip-Chip, and the surface remains solderable even after multiple reflow cycles. It is useful for contact surfaces such as membrane switches and contact points and may also be used for wire bonding.

The Immersion Gold (IG) bath provides the protective Au layer after the Electroless Ni (EN) process following the below equation: $2Au^{+} + Ni \rightarrow 2Au + Ni^{2+}$. As an electrochemical process, the deposition rate (or Au thickness per unit of time) is highly dependent on the redox potential of Au^{+} and electropotential of the Ni surface. The former can be considered as a fixed value in a Au plating solution while the latter will vary depending on the design of boards (pad size, connection of pad to the inner-layer Cu, etc.). The differences of surface potential, or so-called galvanic effect, makes the deposition of Au non-uniform, which may influence the properties of the board in many aspects. For example, chromatic aberration of the Au surface appears and some of the extremely thick Au pads may appear as red color; solderability may become uneven across all pads; and Au may be wasted due to the poor gold thickness distribution. More seriously, the galvanic effect may cause Au skip-plating, or black Ni because of Ni over-dissolution on specific pads, resulting in the solderability problem.

Our new immersion gold technology can effectively reduce the galvanic effect by introducing a new chemical system. The electropotential differences were minimized among pads with various surface areas or connected to different inner-layer copper areas thus providing a uniform gold deposition. Meanwhile, the plated Au thickness can be steadily increased from less than 1 microinch to 3 microinches and metallic lustre of the Au surface remained normal, from the pale yellow of thinner Au to the lemon yellow of thicker Au.

Ni corrosion resistance remained good for a range of Au thickness from 1 microinch to 3 microinches. Modified HNO_3 porosity test yielded good results. Solderability test using SAC305 solder showed good reliability after 0x, 3x reflow and baking (150 ºC, 4 hours) conditions in ball shear and ball pull test.

INTRODUCTION

Final finishing, as the name implies, is the last plating step in the process of printed circuit boards (PCB) production. Its main function is to provide a protective surface that prevents the copper outer-layers from oxidation, meanwhile, this layer of protection must be solderable when assembling the components on the PCB. As one of the important final finish techniques, Electroless Nickel Immersion Gold (ENIG) can afford very good protection as a result of low reactivity of noble metal Au toward corrosives, thus providing much longer storage life for a PCB. The excellent surface planarity is suitable for soldering of very tiny parts such as Flip-Chip BGA and the good thermal resistance make it tolerate multiple reflow cycles. Besides, ENIG can also be used for aluminum wire bonding or serve as contact surfaces such as "gold fingers".

On the other hand, compared with other final finish techniques, ENIG is much more expensive because of Au consumption. In fact, the price of gold has increased by ~400% from the lowest point during the last 15 years and this value reached its historical high point of more than USD 1800/oz in the year of 2011 (Figure 1).[1] In this way, controlling the Au usage is a key point for cost reduction.

FIGURE 1. AU PRICE TREND IN THE LAST 15 YEARS

There are generally two approaches to reduce the cost on Au consumption. One is to operate the Au bath under low Au content. In reality, many PCB manufactures have lowered down the Au concentration from 1.5-2.0 g/l to 0.5-1.0 g/l. If Au content is decreased from 1.5 to 0.5 g/l, 1 g/l Au would be saved per bath life. The other approach is to deposit a uniform Au layer on the Ni/P surface. According to the IPC standard, the more uniform of a gold layer, the less Au would be used to fulfill the minimum Au thickness requirement. [2]

Both approaches requires minimizing the influence of galvanic effect. Due to the board design, the electropotential of Cu pads are not evenly distributed depending on the pad sizes and connections to inner-layer copper etc. Some are more cathodic and Au^+ ion tends to be more easily reduced and have a higher deposition rate, leading to a much thicker Au layer. On the other hand, if the pad is less cathodic, Au layer would be much thinner (Figure 2). Moreover, it was observed that the galvanic effect would be more serious at low gold content, although the mechanism was not clear yet.

FIGURE 2. INFLUENCE OF GALVANIC EFFECT ON AU THICKNESS

The influence of galvanic effect is not only reflected on the Au thickness uniformity, but also on the metallic lustre of Au. Some pads with extremely quick Au deposition rate would be at a high risk for reddish pads while chromatic aberration may be observed for pads with much thinner Au coverage. Functional issues would also be encountered. For example, poor coverage of Au would result in oxidation of Ni underlayer. In an extreme situation, galvanic effect may lead to over-dissolution of Ni (Figure 3). In both cases, solderability issue may appear.

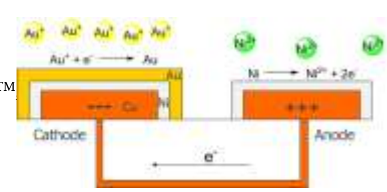


FIGURE 3. NI CORROSION IN A GALVANIC CELL

In this regard, we delivered a new immersion type gold bath (New Bath) that can reduce the influence of galvanic effect. It can minimize the electropotential differences on the Ni/P surface among several of pads on the same boards or different boards, providing uniform Au depositions among pads with different sizes and connections to inner-layer copper areas. The Au color is normal without chromatic aberration. The Ni corrosion resistance is also good and passed ball shear and ball pull solderability test.

EXPERIMENTAL & RESULTS

ENIG Plating Test Condition

All the internal test panels and customer test panels were plated on a 280-L pilot line according to the standard process (Figure 4). Au content was set at 0.5 g/l. The test conditions were based on different Ni bath aging conditions, and the Au bath aging test was simulated by contamination with Ni^{2+} (using $NiSO_4 \cdot (H_2O)_6$) and Cu^{2+} (using $CuSO_4 \cdot (H_2O)_5$) and the six test conditions were listed in Figure 5. For each condition, we also plated test panels with three different Au thickness requirements (1 µ'', 2 µ'' & 3 µ'') by controlling the immersion time. All panels obtained after plating test were in normal golden color. These panels were used for Au thickness measurement (plating rate, uniformity), Ni corrosion resistance and other functional tests.

Process Flow	Tank size	Temp.	Time
Cleaner: **RONACLEAN™ HCP-208 Cleaner**	280 L	40 ºC	3 min
Water Rinse	250 L	rt	1 min
Water Rinse	250 L	rt	2 min
Microetch: **NaPS/H_2SO_4**	280 L	25 ºC	1.5 min
Water Rinse	250 L	rt	2 min
Acid Rinse: **H_2SO_4**	280 L	rt	1 min
Water Rinse	250 L	rt	1 min
Predip: **H_2SO_4**	280 L	rt	1 min
Catalyst: **RONAMERSE™ SMT Catalyst CF**	280 L	25 ºC	3 min
Water Rinse	250 L	rt	1 min
Postdip: **H_2SO_4**	320 L	25 ºC	1 min
Water Rinse	250 L	rt	1 min
Electroless Ni: **DURAPOSIT™ SMT 88 ELECTROLESS NICKEL**	280 L	82~84 ºC	~20 min
Water Rinse	250 L	rt	1 min
Water Rinse	250 L	rt	1 min
Immersion Gold: **New Bath**	280 L	84 ºC	varied

Water Rinse	250 L	rt	1 min
Water Rinse	250 L	rt	1 min
Hot Water Rinse	250 L	60 ºC	1 min
Dry		85 ºC	

FIGURE 4. ENIG PROCESS FLOW CHART

Test Condition		1	2	3	4	5	6
Electroless Ni: **DURAPOSIT™ SMT 88 ELECTROLESS NICKEL**	MTO	0.3	1.1	2.3	2.6	2.8	2.9
Immersion Gold: **New Bath**	Ni^{2+}	NA	NA	NA	200 ppm	700 ppm	700 ppm
	Cu^{2+}	NA	NA	NA	NA	1 ppm	5 ppm

FIGURE 5. ENIG TEST CONDITIONS

		< 2x2 mm	2x2 mm	> 2x2 mm	overall
Customer panel A 3 µ"	Average, u"	3.41	3.47	3.60	3.50
	Max, µ"	3.49	3.57	3.69	3.69
	Min, µ"	3.33	3.37	3.51	3.33
	R, µ"	0.16	0.20	0.18	0.36
	STD, µ"	0.06	0.06	0.06	0.10
	STD%	1.6%	1.6%	1.8%	2.9%
Customer panel B 2 µ"	Average, u"	2.40	2.58	2.40	2.46
	Max, µ"	2.42	2.62	2.43	2.62
	Min, µ"	2.36	2.53	2.36	2.36
	R, µ"	0.06	0.09	0.07	0.26
	STD, µ"	0.02	0.03	0.02	0.09
	STD%	0.9%	1.1%	1.0%	3.6%
Customer panel C 1 µ"	Average, u"	1.20	1.18	1.16	1.18
	Max, µ"	1.25	1.19	1.17	1.25
	Min, µ"	1.15	1.16	1.13	1.13
	R, µ"	0.10	0.03	0.04	0.12
	STD, µ"	0.04	0.01	0.02	0.03
	STD%	3.1%	1.0%	1.4%	2.4%

FIGURE 7. AU THICKNESS VARIATION FOR DIFFERENT THICKNESS REQUIREMENTS

Gold Deposition Thickness

Au thickness data for plotting its relationship versus time were collected with a Hitachi FT9550X XRF Coating Thickness Gauge on a 4x4 mm pad of internal panel B.

Generally speaking, a thin gold of 1-microinch Au thickness was achieved in 2 min for the New Bath, and 8 min was required for a 2-microinch panel. The plated Au thickness could be steadily increased as gold plating went on, and 3-microinch thick gold would be achieved in 15 min even for an aged Ni bath and an aged Au bath as in Condition 6 (Figure 6).

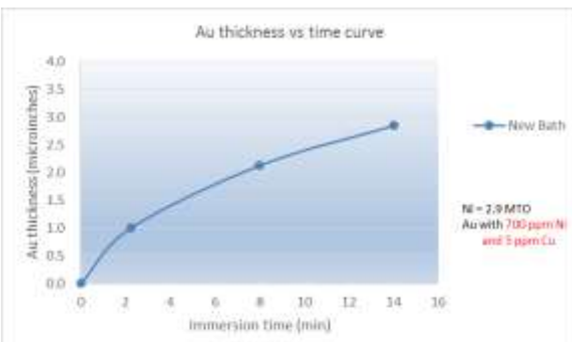
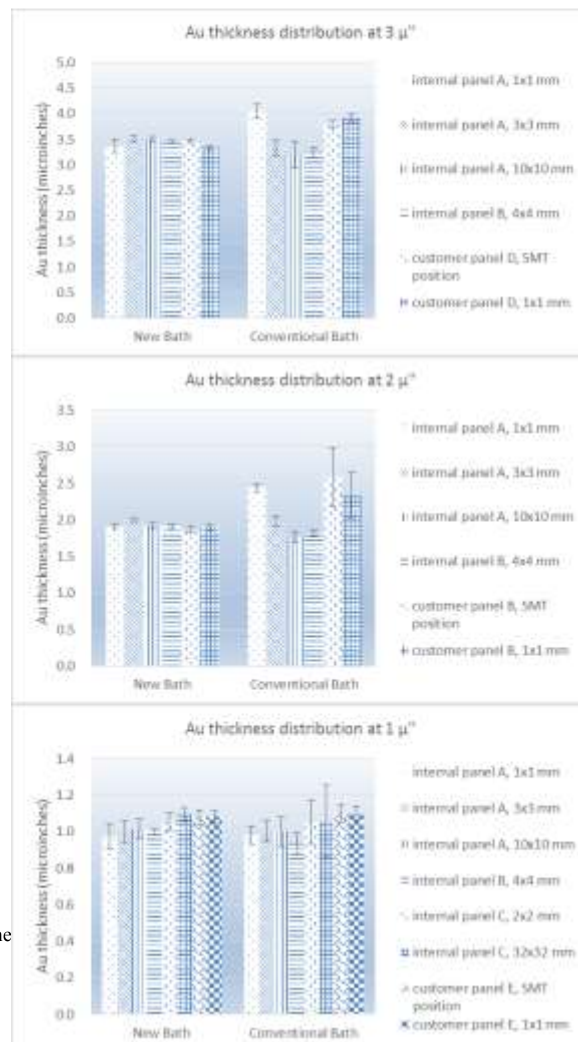


FIGURE 6. PLATING RATE FOR NEW BATH

Uniform Gold Thickness Distribution

Three types of customer test panels (multi-layer board) were used to evaluate Au thickness uniformity for different Au thickness requirements, respectively. We measured Au thicknesses on small pads (<2x2 mm), 2x2 mm pads, and large pads (>2x2 mm) for each test panels. These pads were connected to different inner-layers via through-holes, and there were 10 samples for each group of pad size. The results were listed in Figure 7. No matter within the same size or with different size among the test board, the standard deviation of Au thickness keeps very small (< 4%) as Au deposition grows from 1-microinch thin gold to 3-microinch thick gold.

In addition, we also made a comparison of Au thickness uniformity between New Bath and a conventional IG bath (Conventional Bath) for the three Au thickness levels (Figure 8). The most remarkable advantage of the New Bath compared to the Conventional Bath is that the Au thickness distribution is much more uniform not only within the pads (with different sizes) in the same test board (small standard deviation), but also for pads among different test boards (small difference of Au thickness).



RONACLEAN™, RONAMERSE™, DURAPOSIT™, SUPERSTRIP™ are Trademark of the

FIGURE 8. COMPARISON OF AU THICKNESS UNIFORMITY BETWEEN NEW BATH & CONVENTIONAL BATH

Good Ni Corrosion Resistance

SEM inspection of Ni/P surface was recorded at 1500x magnification after removal of IG layers using a SUPERSTRIP™ 100 gold stripping bath. There was no significant corrosion on the grain boundary nor pitting nor pin-hole type corrosion detected on pad as-plated (Figure 9). We also examined its performance after 1xOSP simulation based on 30 µ'' micro-etch by NaPS. SEM inspection still afforded good results indicative of the low porosity of IG layer and New Bath can be used in selective-ENIG.

Porosity test was also performed using a modified HNO_3 test method according to ASTM B 735 standard. [3] The ENIG samples was carefully prepared from a test panel with 2-microinch Au thickness without any damage on the gold surfaces. It was cleaned by DI water then dipped in 15% HNO_3 solution for 30 min. Then it was immersed in 10% NaOH solution for 1min and rinsed with DI water. After dipping it in Na_2S_x solution for 1 min and cleaned with DI water, the sample was dried and examined at 100x magnification. The results showed that no obvious black or red pore was detected on the lemon yellow Au surface (Figure 10), which showed low porosity of the IG layer and it was in accordance with the SEM results after 1xOSP simulation test.

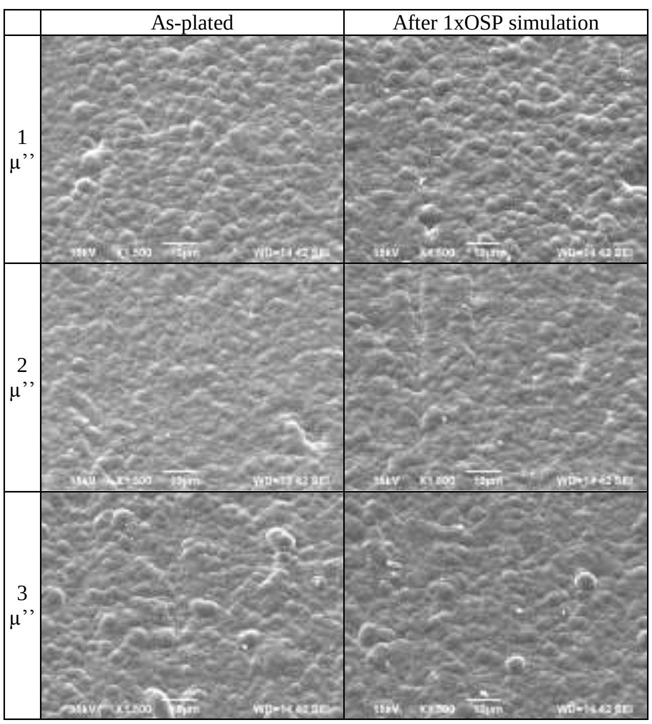


FIGURE 9. SEM INSPECTION OF THE NI/P SURFACE OF PAD OBTAINED FROM NEW BATH

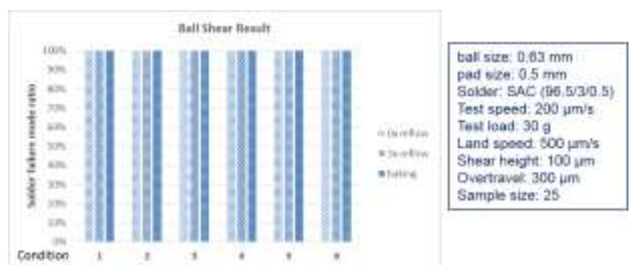
RONACLEAN™, RONAMERSE™, DURAPOSIT™, SUPERSTRIP™ are Trademark of t

FIGURE 10. POROSITY TEST RESULT FOR NEW BATH

Good Solderability Test Result

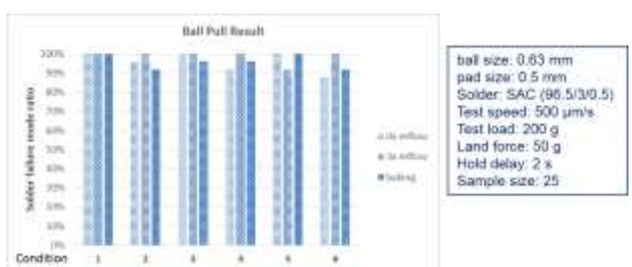
Solderability test was performed using Pb free solder SAC305 on internal test board B with Au thickness of 2 microinches. For the test boards obtained under each test condition, we did the test for 0x reflow, 3x reflow and baking under 150 °C for 4 h.

Both solder ball shear and ball pull test conditions and results were shown in Figure 11 and Figure 12, respectively. For ball shear test, 100% solder failure mode was observed for panels obtained from different Ni and Au aging conditions. Ball pull test also afforded good results with an average of >90% solder failure mode.



Average force (g) (passing rate)	Condition					
	1	2	3	4	5	6
0x reflow	1159 (24/24)	1202 (25/25)	1265 (24/24)	1197 (25/25)	1224 (25/25)	1248 (25/25)
3x reflow	1237 (24/24)	1231 (25/25)	1219 (25/25)	1256 (25/25)	1186 (25/25)	1206 (25/25)
Baking	1299 (24/24)	1331 (25/25)	1354 (25/25)	1355 (25/25)	1346 (25/25)	1334 (25/25)

FIGURE 11. BALL SHEAR TEST RESULT



Average force (g) (passing rate)	Condition					
	1	2	3	4	5	6
0x reflow	1668 (22/22)	1710 (24/25)	1817 (24/24)	1754 (23/25)	1801 (25/25)	1817 (22/25)
3x reflow	1761 (24/24)	1827 (25/25)	1810 (25/25)	1891 (25/25)	1816 (22/24)	1813 (25/25)
Baking*	1973 (25/25)	1960 (23/24)	2047 (24/25)	2055 (24/25)	1972 (25/25)	1997 (23/25)

FIGURE 12. BALL PULL TEST RESULT

CONCLUSION

In summary, the new immersion type gold plating technology can minimize the influences of galvanic effect. This technology can provide uniform Au thickness distribution among several of pads within or among different test boards. It not only make cost saving for Au consumption but also reduce possible negative influences caused by galvanic effect, such as chromatic aberration and other functional issues. Meanwhile, the Ni corrosion resistance was good and can pass 1xOSP simulation based on 30 u'' SPS micro etch. Ball shear and ball pull solderability test also showed good results.

REFERENCES

[1] Data was obtained from http://goldprice.org/gold-price-history.html

[2] *Specification for Electroless Nickel/Immersion Gold (ENIG) Plating for Printed Circuit Boards*. IPC-4552 Amendment 1.

[3] *Standard Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor*. ASTM B735-06(2011).