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INTERCONNECT TECHNOLOGIES

Development of Novel Immersion Gold for Electroless Nickel Immersion Gold Process (ENIG) in PCB Applications

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ABSTRACT

Electroless nickel and immersion gold (ENIG) is one of the most common final finishes used in PCBs. ENIG surfaces exhibit excellent planarity, solderability and wire bondability and tolerate multiple lead free solder reflows during assembly processing. However, the price of gold has increased rapidly from around \$400 US per troy ounce in 2004 to over \$1200 US in 2010. This change has increased ENIG operating costs for PCB manufacturers. While many customers have considered operating their immersion gold baths at levels well below the normal product operating ranges, making this change without proper qualification may have a negative impact on ENIG coating performance. In order to avoid such risks, we have developed a new immersion gold product, designed to operate at lower gold concentration, while ensuring that performance characteristics, including deposition rate, the impact on the electroless nickel layer during the displacement reaction and the coverage of the final deposit, are maintained at the required levels.

INTRODUCTION

In ENIG processes, the strong influence of both electroless nickel bath formulation and process control on the properties of the final deposit have been addressed by many researchers [1, 2]. The immersion gold step also has a strong influence on performance. The bath formulation controls the gold deposit thickness and also affects the underlying electroless nickel interfacial layer, due to the immersion reaction. Both of these factors can influence the strength of solder joints formed on ENIG finishes. Past studies of immersion gold have included the effect of reducing agent [3, 4], bath pH and temperature on the deposition process [5]. While non-cyanide electroless gold processes have also been examined [6-8], cyanide-based systems remain the most widely used products, due to their stability, long bath life and excellent deposit quality.

In this paper, the performance of this novel immersion gold bath will be described. Even though the required gold content is much reduced, plating rate is not reduced. In addition, the new formulation has been found to substantially reduce the occurrence of hyperactive corrosion at the electroless nickel immersion gold interface, which minimizes the risk of solder joint failure during subsequent assembly processes. The bath is also very well suited to use in selective ENIG processes, due to the excellent gold coverage and low porosity of the immersion gold film. These attributes allow the deposit to withstand corrosive attack during the Organic Solderability Preservative (OSP) process.

The characteristics of ENIG deposits will be presented, together with test results for solderability, solder joint ball shear and immersion gold porosity.

The performance of the deposit after multiple OSP process exposures was also examined, to demonstrate process capability for Selective ENIG applications.

IMMERSION GOLD COST ANALYSIS

The majority of commercially available immersion gold products operate at gold metal concentrations between 1.5 – 2.0 g/l. In contrast, the new immersion gold bath operates at much lower gold concentrations, in the range between 0.5 – 0.7 g/L. The gold salt used is gold potassium cyanide.

Table 1 shows a comparison of the total gold metal running cost for immersion gold baths operated at 2 g/L, 1.2 g/L and 0.7 g/L of gold metal. The analysis is based on an assumption of 5 ml per feet square of drag out volume and 0.05 micron gold deposit thickness. Since the amount of plated gold will vary greatly, depending on whether all surface features are plated (Normal ENIG) or only those features intended for use as keypad contacts (Selective ENIG), this factor must also be included in the analysis.

Figure 1 shows a graph of total gold cost against different concentrations of gold. When gold operating concentration is reduced from 2 g/L to 0.7 g/l, the total gold metal costs drop by 28% and 45% for normal and selective ENIG respectively. The cost savings are larger for selective ENIG process as the drag out loss of gold metal is relatively more significant compared the amount of metal deposited on the board.

(a)	Normal ENIG (15% Copper Exposed Area not covered by soldermask)		
	2 g/l	1.2 g/l	0.7 g/l
Gold Price (USD/troy Oz)	1150	1150	1150
Gold Price(USD/g)	40.3	40.3	40.3
Drag out (ml/ft ²)	5	5	5
Au thickness (micron)	0.05	0.05	0.05
Effective Plating Area (%)	15	15	15
Au density (g/cm ³)	19.32	19.32	19.32
Au Metal Drag out (g/ft ²)	0.01	0.006	0.0035
Au Metal Deposited on Board (g/ft ²)	0.01346	0.01346	0.01346
Spent bath of Au used per square feet of board processed (g/ft ²)	0.00235	0.00195	0.00170
Total Au consumed (g/ft ²)	0.02581	0.02141	0.01866
Au metal running cost (USD/ft ²)	1.04	0.86	0.75

(b)	Selective ENIG (5% Copper Exposed Area not covered by soldermask and dry film)		
	2 g/l	1.2 g/l	0.7 g/l
Gold Price (USD/troy Oz)	1150	1150	1150
Gold Price(USD/g)	40.3	40.3	40.3
Drag out (ml/ft ²)	5	5	5
Au thickness (micron)	0.05	0.05	0.05
Effective Plating Area (%)	5	5	5
Au density (g/cm ³)	19.32	19.32	19.32
Au Metal Drag out (g/ft ²)	0.01	0.006	0.0035
Au Metal Deposited on Board (g/ft ²)	0.00449	0.00449	0.00449
Spent bath of Au used per square feet of board processed (g/ft ²)	0.00145	0.00105	0.00080
Total Au consumed (g/ft ²)	0.01594	0.01154	0.00879
Au metal running cost (USD/ft ²)	0.64	0.46	0.35

Table 1: Gold Metal Running Cost for (a) Normal ENIG (b) Selective ENIG.

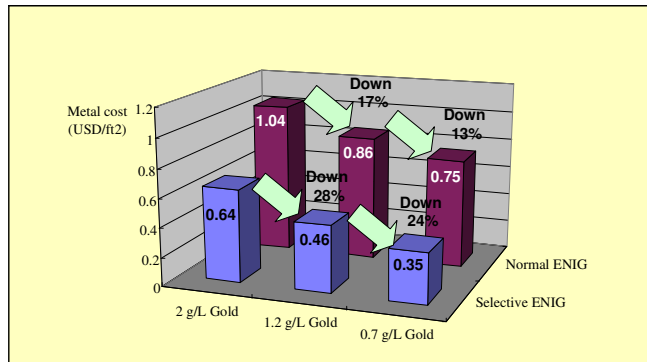


Figure 1: Operational Cost Comparison for Different Gold Concentrations.

EXPERIMENTAL

Sample Preparation

Two standard test panel designs were used for the immersion gold process characterizations reported in this paper. One substrate has 0.47 mm diameter BGA pad features suitable for ball shear test and the other has 4 mm pads designed for globule wetting balance test.

All the chemicals used for preparing the ENIG deposits were Dow Electronic Materials products. Prior to EN plating, standard copper surface cleaning and surface activation steps appropriate for ENIG were carried out.

The thicknesses of the electroless nickel and immersion gold layers were 4-5 μm and 0.05-0.07 μm respectively. Gold and nickel thickness were measured using X-ray fluorescence (XRF). To examine the structure of the EN interfacial layer, the IG layer was first stripped off from the ENIG deposits, using a proprietary cyanide based room temperature stripper.

Ball Shear Test

The strength of solder joints formed on the ENIG deposits was evaluated using Ball Shear testing. Prior to attachment of 0.63 mm diameter SAC305 (tin / silver 3% / copper 0.5%)¹ lead-free solder balls, a soluble non-activated flux was applied to the pad surfaces. To form the bond between the ENIG surface and solder, the panel was processed through a reflow oven with a profile peak temperature of 260°C. In addition to an as-plated sample, other parts were exposed to either three cycles of lead-free reflow or a 4 hour bake at 150°C (an accelerated shelf life aging condition).

A Dage 4000 Bond tester was used to evaluate solder ball shear force. The test speed and shear height were set at 200 μm/s and 100 μm respectively. The shear force and the fracture interface location were recorded for each location tested.

Wetting Balance Test

The solderability performance of the deposits was evaluated using a globule wetting balance test. The sample was first cleaned with analytical grade isopropanol to remove surface contaminants. A Must III Wetting Balance unit was used and the test parameters were as shown in Table 2.

Test Time (s)	10	Temperature (°C)	260
Immersion Depth (mm)	0.5	Solder Alloy	96.5Sn/3Ag/0.5Cu ²
Immersion Speed (mm/s)	1.0	Size of Pellet	4 mm (200mg)
Removal Speed (mm/s)	10.0	Flux	Alpha 100 ³

Table 2: Test Parameters for Wetting Balance Test.

The solderability was assessed by recording values for T_b (Time to Buoyancy corrected to zero) and Time to 2/3 F_{max} (Time for the sample to reach 2/3 maximum force F_{max}).

Porosity Test

A nitric acid porosity test was used to evaluate immersion gold deposit coverage. A modification of an IPC test method (IPC-TM-650 : 2.3.24.2, Method 3), using a 30 minute immersion in 15% nitric acid solution, instead of sample exposure to nitric acid vapor for 1 hour, was used. After that the nitric acid immersion, samples were dipped into a polysulphide solution and then rinsed with DI water. Optical microscope images were used to record whether black or red corrosion products were present on the surface

RESULTS AND DISCUSSION

The low gold immersion gold bath is capable of depositing about 0.05 – 0.06 μm in 5 – 6 min at 83°C. Gold deposition rate fell less than 10% when the dissolved nickel ion concentration in the bath was raised to 600 ppm. With the new formulation, the variation in gold thickness between different pad sizes (2mm x 2mm to 8mm x 8mm) was found to be very small throughout the bath life. Results are shown in Figure 2.

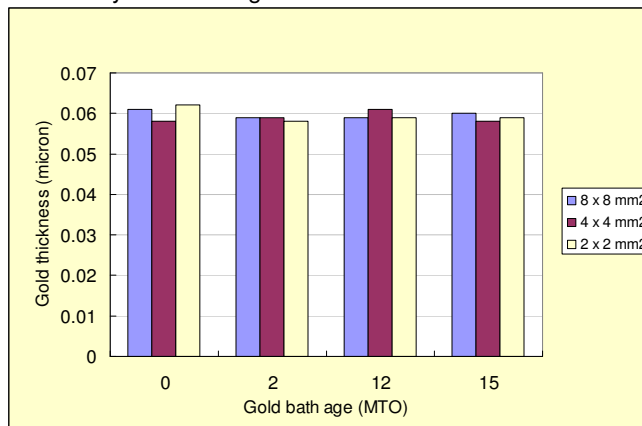


Figure 2: Impact of Pad Size on Immersion Gold Thickness.

To assess the condition of the electroless nickel layer, the immersion gold was stripped and the top surface was examined by scanning electron microscope (SEM) as shown in Figure 3. The new low gold chemistry induced no observable nickel corrosion.

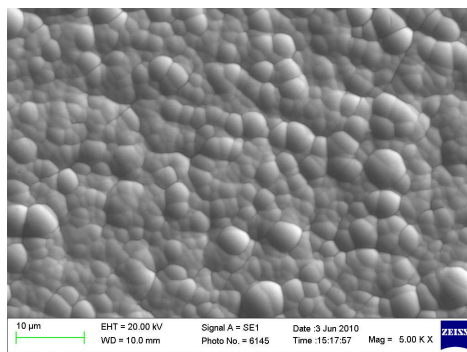


Figure 3: Typical SEM Image of EN Surface after Gold Stripping.

Ball shear test and wetting balance test were carried to measure the solder joint performance of the ENIG deposits. Thirty measurements were made for each ball shear test condition. Figure 4 shows the average shear forces obtained at different gold bath ages for as-plated samples and samples exposed to two different pre-conditionings. The values of force were high and no significant change in shear force was observed for either of the preconditioned samples, implying that the quality of the deposits was good.

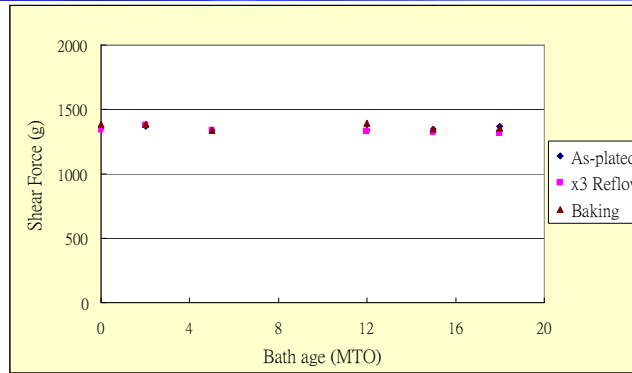


Figure 4: Shear Force Result for Ball Shear Test.

The fracture mode was also recorded during the ball shear tests. In all cases, the failures occurred within the solder, rather than in the IMC layer at the solder joint interface, showing that all the samples had good solder joint formation with SAC305 alloy. The results of wetting balance tests (average of four samples) for as-plated and preconditioned samples (150°C for 4 hours) are shown in Table 3.

Au Bath Age (MTO)	Tb (sec)		Time to 2/3 Fmax (sec)		Fmax (mN)	
	As plated	Baked	As plated	Baked	As plated	Baked
0	0.04	0.16	0.48	0.88	2.00	1.43
2	0.04	0.31	0.53	0.72	1.85	1.43
5	0.05	0.36	0.50	0.83	1.98	1.50
12	0.05	0.44	0.49	0.86	2.02	1.54
15	0.05	0.45	0.49	0.83	2.93	1.60
18	0.09	0.32	0.60	0.68	2.11	1.75

Table 3: Wetting Balance Results.

While the baked samples showed slightly poorer wetting performance than the as-plated samples, the difference was relatively small. Time to 2/3 Fmax was less than 1 seconds for all samples tested. These results indicate that the solder wetting ability of the ENIG deposits can be maintained throughout the bath life.

Another important property of ENIG deposit is the coverage of the electroless nickel by the gold deposit. Good gold coverage will minimize the possibility for nickel oxidation during long time storage and block attack on the electroless nickel layer by the OSP process during selective ENIG application. Oxidation of the EN surface can reduce the solder wetting performance and reduce solder joint reliability.

Figure 5 shows the results of porosity tests comparing ENIG deposits produced using the new immersion gold process with those from a previous generation process. A smaller number of locations showing brown corrosion products were observed for the new process, indicating that the deposit has improved gold coverage.

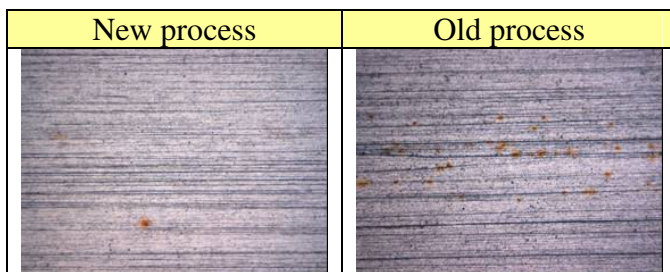


Figure 5: Results of Porosity Test on New and Old Processes.

INTERCONNECT TECHNOLOGIES TECHNICAL COMMUNICATIONS

The performance of deposit after exposure to OSP processing was also investigated. ENIG samples prepared using the new and old process and then passed three times through an OSP production line. The microetch chemical used in the OSP process was sodium persulfate, with a measured etch rate on copper of 18 microinches per cycle. The samples were then cross-sectioned and examined under SEM to determine the condition of the ENIG surface after OSP exposure. Figure 6 shows the results of the comparison.

It was found that the sample prepared using the new chemistry was undamaged after OSP process exposure, while the older process showed evidence of localized "spike" corrosion. This difference in performance can be attributed to the reduced porosity of the immersion gold deposit from the new process.

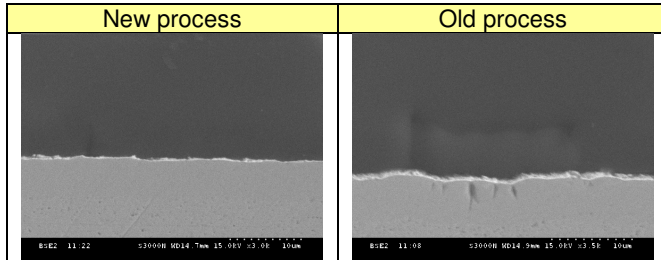


Figure 6: Sample Cross-sections after 3 x OSP Process Exposure.

During the development, it was observed that use of suitable combinations of additives in the bath can provide a comparative faster initial gold deposition rate, but still allow complete coverage to be reached after only ten minute deposition. Deposition times from 10 minutes to 15 minutes only lead to a small increase in gold thickness as the majority of the electroless nickel surface has already been covered with gold. This kind of gold bath formulation can minimize the risk of hyperactive attack on electroless nickel, while providing good deposit coverage. Figure 7 showed the effect of bath formulation on deposition behavior.

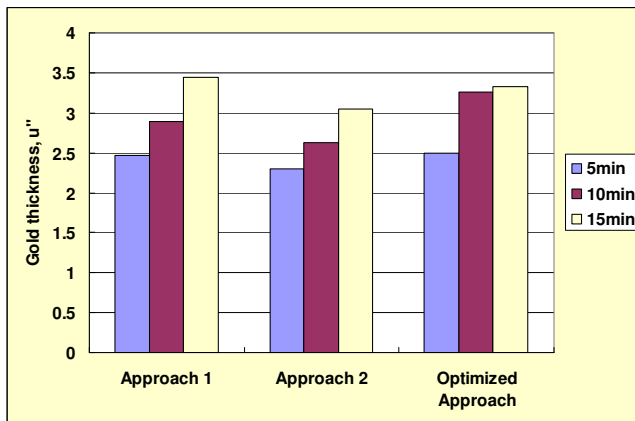


Figure 7: Effect of Bath Formulation on Gold Deposition Behavior.

CONCLUSION

A novel immersion gold process has been developed for ENIG process. The low gold concentration used in the formulation substantially reduces the process cost for PCB manufacture. The process has been optimized to provide stable plating rate and a deposit with excellent uniformity, low porosity and stable solderability performance. It has been proven to have excellent and stable performance both in normal and selective ENIG applications in high volume ENIG production installations.

REFERENCES

1. R.W M. Kwok, K.C.M. Chan and M.W. Bayes (2004), "Development of an electroless nickel immersion gold for PCB final finishes", *Circuit World*, Vol. 30, pp 37 – 42.
2. G. Milad and J. Martin (2000), "Electroless nickel/immersion gold, solderability and solder joint reliability as functions of process control", *Circuitree*, pp 56 – 62.
3. T. N. Vorobyova, S.K. Poznyak, A.A. Rimskaya, O.N. Vrublevskaya (2004), "Electroless gold plating from a hypophosphite-dicyanoaurate bath", *Surface and Coatings Technology*, Vol. 176, pp 327 – 336.
4. N. Shaigan, S.N. Ashrafizadeh, M.S.H. Bafghi and S. Rastegari (2005), "Elimination of the Corrosion of Ni-P Substrates during Electroless Gold Plating", *Journal of The Electrochemical Society*, Vol. 152, pp C173 – C178.
5. H. Liu, N. Li, S. Bi and D. Li (2007), "Gold Immersion Deposition on Electroless Nickel Substrates – Deposition Process and Influence Factor Analysis", *Journal of The Electrochemical Society*, Vol. 154, pp D662 – D668.
6. J. Sato, M. Kato, H. Otani, T. Homma, Y. Okinaka, T. Osaka and O. Yoshioka (2002), "Substrate (Ni)-Catalyzed Electroless Gold Deposition from a Noncyanide Bath Containing Thiosulfate and Sulfite (II) Deposit Characteristic and Substrate Effects", *Journal of The Electrochemical Society*, Vol. 149, pp C168 – C172.
7. A.M. Sullivan and P. A. Kohl (1995), "The Autocatalytic Deposition of Gold in Nonalkaline, Gold Thiosulfate Electroless Bath", *Journal of The Electrochemical Society*, Vol. 142, pp 2250 – 2255.
8. H.P. Liu, N. Li, S.F. Bi, L.T. Kong and Q. Tan (2007), "Study of Non-Cyanide Immersion Gold Plating". *Diandu Yu Huanbao*, Vol. 27, pp 26 – 28.