Effect of Process Variations on Solder Joint Reliability for Nickel-based Surface Finishes

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ABSTRACT

Following the adoption of recent Pb-free requirements, PWB and IC substrate fabricators, assemblers and OEMs continue to search for a surface finish that will minimize solder joint reliability concerns. Historically, electroless nickel / immersion gold (ENIG) has provided a reliable finish for soldering applications. The nickel layer provides the added benefit of serving as a barrier to minimize copper dissolution during the soldering operation. Preservation of the underlying copper will become increasingly important as circuit features continue to shrink and signal integrity becomes a more critical issue. However, ENIG is not trouble-free; as the immersion gold reaction can often excessively corrode the nickel layer, leading to the occurrence of “black pad” and tendencies toward brittle failure in solder joints. As an alternative to ENIG, an electroless palladium layer over the nickel (NiPd) should provide a solderable surface while minimizing impact to the nickel deposit. By eliminating the corrosive immersion gold reaction, the resultant intermetallics should contribute to improved solder joint integrity, even after multiple Pb-free reflow operations.

This paper summarizes the results of recent investigations to examine the effect of electroless nickel process variations with respect to Pb-free (Sn-3.0Ag-0.5Cu) solder connections. These investigations included both ENIG and NiPd as surface finishes intended for second level interconnects in BGA applications. Process variations that are suspected to weaken solder joint reliability, including treatment time and pH, were used to achieve differences in nickel layer composition. Immersion gold deposits were also varied, but were directly dependent upon the plated nickel characteristics. In contrast to gold, different electroless palladium thicknesses were independently achieved by treatment time adjustments.

Results of investigations include: (1) Cold ball pull testing to evaluate solder joint integrity, (2) SEM examinations of the underlying nickel surface and (3) IMC examinations to quantify nickel thickness degradation after multiple solder reflow cycles. The paper discusses the relatively simple approach that, if proven effective in large-scale fabrication, may significantly reduce or eliminate many of the well-known reliability concerns associated with nickel-based intermetallics in a Pb-free assembly environment.

BACKGROUND

As a surface finish for BGA applications, electroless nickel / immersion gold (ENIG) has been traditionally accepted for use on both IC substrates and PWBs. The principal benefit of ENIG as a surface finish relates to the role of the nickel layer, which functions as a barrier to limit the dissolution of underlying copper into the solder joint. With continued miniaturization of circuit features, coupled with the need for maintaining signal attenuation, copper dissolution into the solder joint is a critical issue and a barrier to such dissolution is a key objective. Solder joint formation of ENIG surface finishes occurs by dissolution of gold into the solder, forming an IMC between the nickel and tin. However, solder joints comprised of nickel-tin intermetallic compounds (IMC) are generally regarded as more prone to brittle fracture in comparison to copper-tin IMCs. Therefore, it is imperative to control formation of the nickel/tin IMC to produce the desired ductile solder joint.

With ENIG, if not properly controlled, the immersion gold step can significantly corrode the nickel layer. In the worst cases, such corrosion can result in the so-called “black pad” effect. Such attack of the nickel layer can lead to increased occurrence of brittle fracture and serious solder joint reliability concerns as a result. Considerable effort has been devoted to limiting or preventing the occurrence of such nickel attack. One such technique promotes the use of higher phosphorous concentrations in the nickel deposit, making it more resistant to the corrosive nature of the immersion gold solution and resulting in a denser and more evenly formed nickel-tin IMC [1]

Another method for controlling nickel corrosion involves the use of a thin layer of palladium, deposited autocatalytically on the nickel surface, thus protecting it during the immersion gold reaction. This electroless nickel / electroless palladium / immersion gold (Ni/Pd/Au) surface finish was actually introduced in the mid-1990s. However, with the widespread adoption of

Pb-free soldering, this finish has again demonstrated improved performance in comparison to ENIG and has gained considerable attention.

The suitability of the Ni/Pd/Au finish (employing a pure palladium deposit) for both wire bonding and soldering has been previously reported 2,3]. More recently, the findings of a major design of experiment (DoE) that compared the performance of several ENIG and Ni/Pd/Au finishes using different solder ball pull and shear investigations were reported [4]. The results of those investigations indicated that the Ni/Pd/Au process provides improved bonding results for meeting the more demanding BGA soldering requirements for the Sn-Ag-Cu alloy compositions.

In light of the renewed interest in nickel-based surface finishes, investigations were initiated to determine if a more simplified process comprised of electroless nickel / electroless palladium (Ni/Pd) could provide equivalent benefits at a lower cost.

DESCRIPTION OF INVESTIGATIONS

As part of a more comprehensive DoE, investigations were conducted to compare the performance of two different surface finishes: ENIG and Ni/Pd. The cold ball pull test was used as the primary method to evaluate the solder joint reliability of these two nickel-based surface finishes.

Test Vehicle

Figure 1 shows an example of the BGA test vehicle used for these investigations. Also shown are the locations of the 30 solder balls tested. As shown, four balls are located at each of the remote corners of the grid array and the remaining 14 balls are located diagonally across the center of the array.

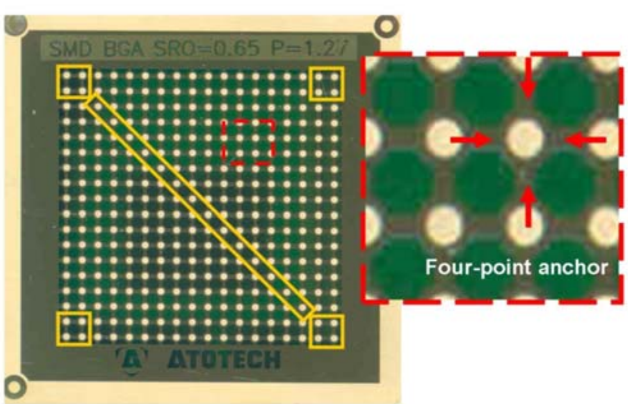

Fig. 1 BGA test vehicle for cold ball pull testing showing location of solder balls tested and detail of four-point anchor design (under solder mask).

The objective of the DoE was to specifically evaluate deposit characteristics and solder joint reliability in relation to variations in surface finish. Build-up material used for IC substrate fabrication cannot typically provide sufficient adhesive strength to withstand the forces of the cold ball pull testing. Most failures occur as a result of the loss of adhesion between the copper pad and the underlying dielectric and the bond at the surface finish is not adequately tested. Therefore, it is important to note that the test vehicle used for these investigations, including both the base material and pad design, was intentionally developed to be conducive to interfacial fractures between solder and pad finish (or failure within the bulk solder). FR4 was used as the test vehicle dielectric because of its inherently higher adhesive strength (50-70% greater than build-up materials). Furthermore, all pads were designed to include a "four-point anchoring" feature. This construction method has been shown to consistently promote the occurrence of interfacial failures by firmly securing the test pads to the underlying dielectric material.

Surface Finish

The test vehicles were fabricated with two different surface finishes, ENIG and Ni/Pd. In the case of ENIG, three different surface thickness variations of electroless nickel were used; depositing 4.0 µm, 5.0 µm and 6.0 µm of nickel, while maintaining a relatively constant 0.09-µm immersion gold thickness. For the Ni/Pd finish, nine different deposit variations were used, with nickel thicknesses of 4.0 µm, 5.0 µm and 6.0 µm in combination with palladium thicknesses of 0.04 µm, 0.10 µm and 0.30 µm. In all cases, the nickel and palladium were both co-deposited with phosphorus; 8-10 percent phosphorus by weight in the nickel deposit and 4-6 percent by weight in the palladium deposit. The nickel deposit thickness was controlled by adjusting the plating rate of the electroless nickel solution. The thickness of the palladium deposit was adjusted according to treatment time. Table 1 presents a summary of the surface finishes included in these investigations.

Table 1 – Summary of Surface Finish Specifications

Surface Finish	Ni	Pd	Au
Ni 4.0 / Au 0.09	4.0 µm	-	0.09 µm
Ni 5.0 / Au 0.09	5.0 µm	-	0.09 µm
Ni 6.0 / Au 0.09	6.0 µm	-	0.09 µm
Ni 4.0 / Pd 0.04	4.0 µm	0.04 µm	-
Ni 5.0 / Pd 0.04	5.0 µm	0.04 µm	-
Ni 6.0 / Pd 0.04	6.0 µm	0.04 µm	-
Ni 4.0 / Pd 0.10	4.0µm	0.10µm	-
Ni 5.0 / Pd 0.10	5.0µm	0.10µm	-
Ni 6.0 / Pd 0.10	6.0µm	0.10µm	-
Ni 4.0 / Pd 0.30	4.0µm	0.30µm	-
Ni 5.0 / Pd 0.30	5.0µm	0.30µm	-
Ni 6.0 / Pd 0.30	6.0µm	0.30µm	-
Deposit thickness variations: Ni: ± 5%, Pd: ±10% Au: ±10%			

Figure 2 shows a comparison of the ENIG and NiPd finish topographies. In the figure, the ENIG surface is shown after the immersion gold deposit has been stripped. As seen, the NiPd surface is very similar in topography to the underlying nickel of the ENIG.

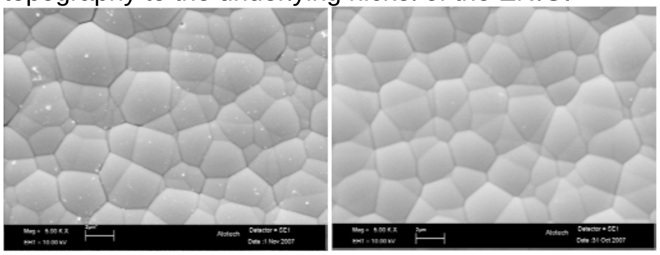

Fig. 2 Comparison of ENIG after gold stripping (left) and NiPd (right) at 5000x

Assembly Parameters

The key information regarding assembly of the solder balls to the BGA test vehicle is summarized in Table 2. As noted, a Pb-free Sn-4.0Ag-0.5Cu (SAC405) alloy was used for all investigations. All bond testing was performed within four hours of reflow. Figure 3 shows the solder reflow profile that was used for all investigations.

Table 2 – Solder Ball Reflow Specifications	
Solder balls	Senju Sn4.0Ag0.5Cu
Solder Flux	Kester TSF6502
Reflow profile	TSF6502 Pb-free linear profile
Reflow Atmosphere	Nitrogen (< 100 ppm O_2)
Peak temperature	258°C
Time to peak	241sec
Time above liquidus	57sec

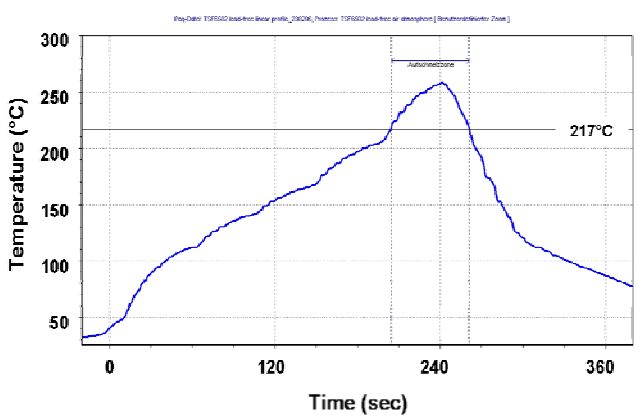

Fig. 3 Pb-free solder reflow profile

Test Procedures - Cold Ball Pull

As previously noted, cold ball pull investigations included the testing of 30 Pb-free solder balls from each test sample. The test samples differed only according to their respective surface finish. Solder balls of 760-µm diameter were mounted on solder resist-defined pads with solder resist openings (SRO) of 650µm.

Cold ball pull testing was performed on the Dage 4000 Multi-Function Bond Tester. The ball pull test mechanism and a detailed view of the ball pull device are shown in Figures 4 and 5, respectively.

Table 3 presents a summary of the important parameters related to the cold ball pull investigations.

Table 3 – Cold Ball Pull Test Parameters	
Equipment	Dage Series 4000
Ball pull speed	5 mm/sec
Solder Ball Diameter	760 µm
Solder Resist Opening	650 µm
Test Conditions	After Pb-free ball attach After 5 additional Pb-free reflow cycles

Fig. 4 Dage Series 4000 Multi-Purpose Bond Tester

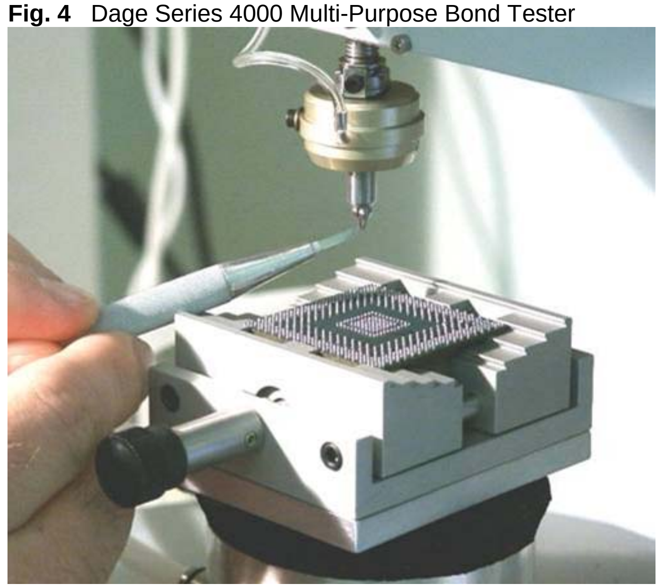

Fig. 5 Detailed view of the solder ball pull device on the Dage Series 4000 Multi-Purpose Bond Tester

Cold ball pull failures were categorized according to the type and location of failure. The preferred type of failure occurs entirely within the solder ball (Mode 2). A bond failure (Mode 4) is indicative of brittle fracture; although unlike ball shear interfacial fractures, in the case of cold ball pull testing the complete IMC is typically exposed. Pad failure or pad "pull-out" (Mode 1) only indicates that the bond of the solder ball to the pad was stronger than the pad-to-substrate adhesive strength. Ball extrusion (Mode 3) may indicate a problem with the clamping force of the device or that the solder composition is too soft.

Figure 6 illustrates the location of the four different fracture modes within the solder ball joint.

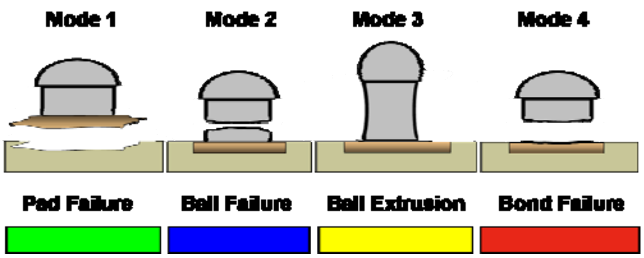

Fig. 6 Illustration of cold ball pull failure modes and their location within the solder ball joint

TESTING RESULTS

Cold ball pull test results are reported and evaluated in terms of both pull strength and failure mode. The graphics legend for qualifying the four types of cold ball pull failures is as follows:

- Mode 1: Pad failure (pad pull-out)
- Mode 2: Solder ball failure
- Mode 3: Solder ball extrusion
- Mode 4: Bond failure

Testing Results

The results of cold ball pull testing performed on samples following "ball attach" (i.e. prior to any additional reflow cycles) are presented in Table 4. These same pull strength data and results of the failure mode analysis are graphically examined in Figure 7.

As shown in the upper part of the figure, there is not a significant difference in the pull strength for the sample group. The ENIG samples did achieve slightly lower average pull strengths and those results also showed a wider variation in comparison to the NiPd results. Generally, the pull strength results for the NiPd samples were very consistent. However, a significant difference exists in terms of fracture mode. Nearly all the ENIG samples exhibited interfacial (brittle) fracture, while the majority of the NiPd samples displayed ductile fracture or pad failures. In particular, there is a noticeable trend of increased ductile fractures as the palladium deposit thickness increases.

Table 4 – Cold Ball Pull Strength Test Results - After Ball Attach

Surface Finish	Mean	Std Dev	Min	Max
Ni 4.0 / Au 0.09	3383.9	216.4	2639.3	3851.8
Ni 5.0 / Au 0.09	3239.3	332.0	2553.3	3734.8
Ni 6.0 / Au 0.09	3420.7	256.2	2777.8	3887.3
Ni 4.0 / Pd 0.04	3708.5	108.1	3571.5	3931.3
Ni 5.0 / Pd 0.04	3644.1	93.3	3492.1	3823.7
Ni 6.0 / Pd 0.04	3663.1	62.3	3510.6	3780.1
Ni 4.0 / Pd 0.10	3647.4	76.1	3475.5	3766.6
Ni 5.0 / Pd 0.10	3638.2	80.7	3496.9	3809.1
Ni 6.0 / Pd 0.10	3608.5	70.9	3446.1	3736.2
Ni 4.0 / Pd 0.30	3557.6	138.8	3070.1	3777.8
Ni 5.0 / Pd 0.30	3584.7	79.6	3397.9	3700.2
Ni 6.0 / Pd 0.30	3600.6	106.3	3359.8	3809.8

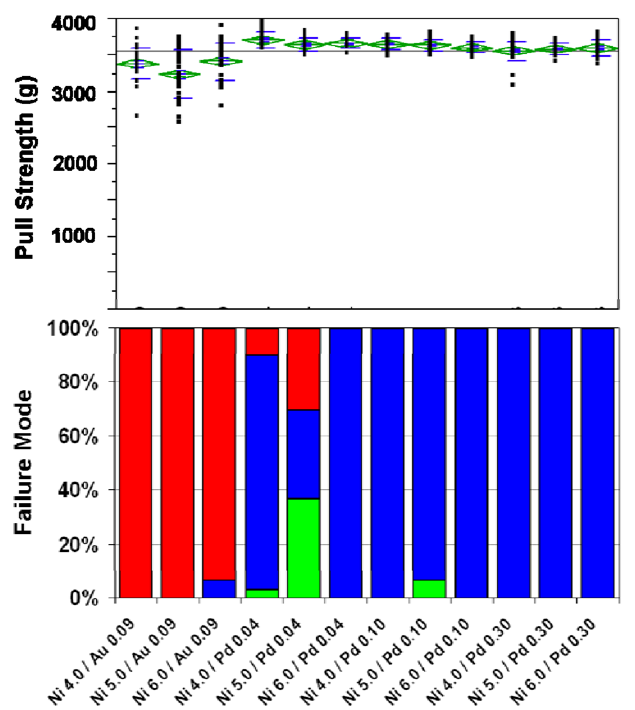

Fig. 7 Summary of cold ball pull strength and fracture modes by surface finish for samples tested in "After Ball Attach" condition

Similarly, Table 5 and Figure 8 present the results of ball pull testing performed on samples following ball attach plus five additional reflow cycles. In this case, comparing the results for the NiPd and ENIG surface finishes, a noticeable trend can be seen in terms of increasing pull strength. An increase in palladium thickness appears to correspond to measurable increases in pull strength. In particular, the NiPd finishes with 0.3 µm palladium exhibited higher and more consistent pull strengths. The failure mode analysis indicated that interfacial fractures occurred for all samples except the NiPd finishes with 0.3 µm palladium. It is significant that the pull strength results, alone, would not seem to indicate such a significant difference in performance as compared to the failure mode analysis.

Table 5 – Cold Ball Pull Strength Test Results - After Ball Attach + Five Reflows

Surface Finish	Mean	Std Dev	Min	Max
Ni 4.0 / Au 0.09	1884.7	259.9	1260.3	2331.5
Ni 5.0 / Au 0.09	2036.7	257.7	1519.2	2603.7
Ni 6.0 / Au 0.09	2301.4	358.4	1792.0	3311.3
Ni 4.0 / Pd 0.04	2686.4	315.7	2236.3	3423.1
Ni 5.0 / Pd 0.04	2495.3	220.6	2094.9	2952.5
Ni 6.0 / Pd 0.04	2927.2	281.4	2331.4	3484.4
Ni 4.0 / Pd 0.10	3160.3	301.2	2393.3	3701.7
Ni 5.0 / Pd 0.10	3085.3	256.2	2578.7	3525.3
Ni 6.0 / Pd 0.10	3076.5	314.0	2266.4	3601.6
Ni 4.0 / Pd 0.30	3595.5	108.1	3405.2	3784.2
Ni 5.0 / Pd 0.30	3516.3	141.9	3024.4	3679.2
Ni 6.0 / Pd 0.30	3623.7	91.4	3459.1	3842.5

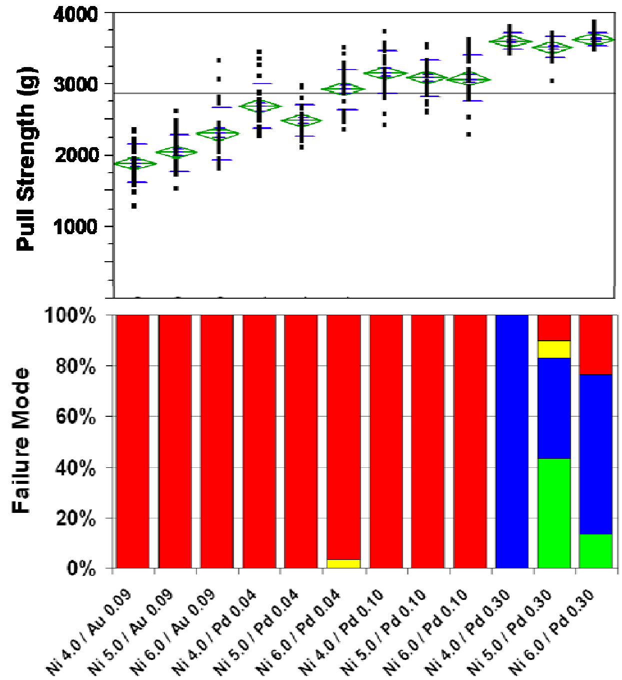

Fig. 8 Summary of cold ball pull strength and fracture modes by surface finish for samples tested in “After 5x Reflow” condition

It is interesting to note in the failure mode chart in Figure 8 that the incidence of brittle fracture appears to increase with increasing nickel thickness for the NiPd finishes with 0.3 µm palladium. It is theorized that the slower plating rate (used for the thinner nickel deposit) results in a higher phosphorus content in the nickel layer, improving the formation of the IMCs.

Testing Results – Intermetallic Examination

The results presented to this point indicate that the Ni/Pd finishes with thicker palladium deposits achieved superior cold ball pull results in comparison to a conventional ENIG finish. To investigate the reason for this apparent performance improvement, the intermetallic compounds (IMC) of the finishes were examined. All samples were prepared by focused ion beam (FIB) technique prior to examination by SEM.

Figure 9 shows one SEM cross-section image of an ENIG surface finish sample following Pb-free solder ball attachment, according to the previously described reflow profile. As shown, both the phosphorus-rich Ni_3P and NiSnP IMCs have been formed. The $(Cu, Ni)_6Sn_5$ IMC measures several microns in thickness.

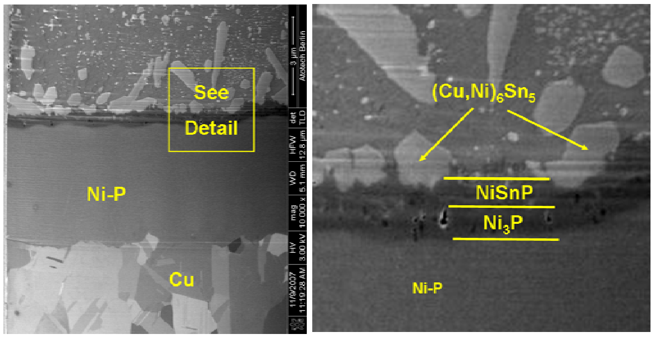

Fig. 9 SEM cross-section image at 10,000x (left) of ENIG surface finish after ball attach and detail (right) showing formation of Ni_3P, NiSnP and (Cu, $Ni)_6Sn_5$ IMCs

Similarly, Figure 10 shows an SEM cross-section image of the ENIG surface finish sample after five additional Pb-free solder reflow cycles. As shown, the Ni_3P and NiSnP IMCs have grown as a result of the thermal excursions. Also, the (Cu, $Ni)_6Sn_5$ IMC has become significantly larger and more dense.

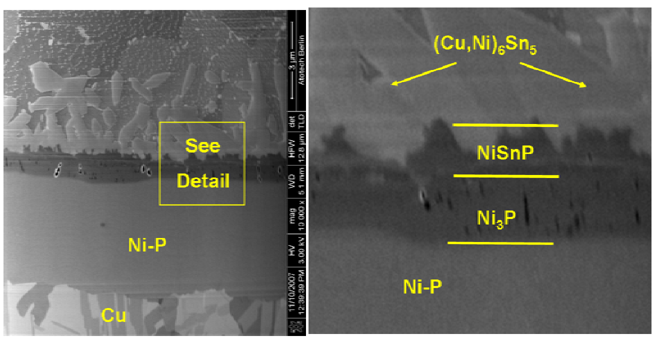

Fig. 10 SEM cross-section image at 10,000x (left) of ENIG surface finish after 5x reflow cycles and detail (right) showing formation of Ni_3P, NiSnP and (Cu, $Ni)_6Sn_5$ IMCs

By comparison, Figure 11 shows a similar SEM cross-section image of a NiPd surface finish sample following Pb-free solder ball attachment. Again, the Ni_3P and NiSnP IMCs have been formed and are comparable to those in the ENIG sample. In the case of the NiPd sample, there appears to be a more distinct boundary between these two IMCs. More significant, however, is the fact that the (Cu, $Ni)_6Sn_5$ IMC is almost not detectable, which is quite different in comparison to the similarly treated ENIG sample.

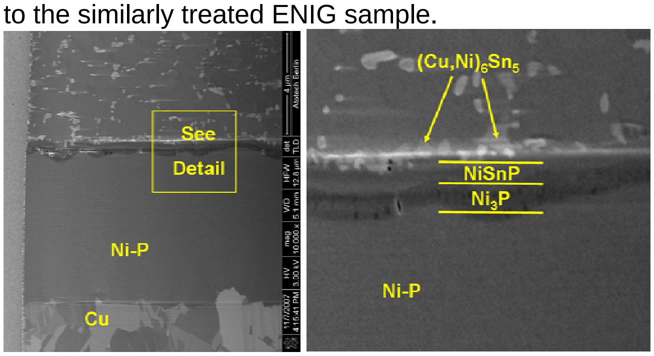

Fig. 11 SEM cross-section image at 10,000x (left) of NiPd surface finish after ball attach and detail (right) showing formation of Ni_3P, NiSnP and (Cu, $Ni)_6Sn_5$ IMCs

In similar fashion, Figure 12 shows an SEM cross-section image of the NiPd surface finish sample after the five additional Pb-free solder reflow cycles. As shown, the Ni_3P and NiSnP IMCs have grown as a result of the thermal excursions. Again, there appears to be a more distinct boundary and composition between these two IMCs in comparison to the similarly treated ENIG sample. More significantly, the (Cu, $Ni)_6Sn_5$ IMC remains extremely thin and does not appear to have grown in comparison to the NiPd sample examined after ball attachment. The palladium layer and/or its contribution to the formed IMCs appear to have limited and nearly prevented any additional growth of the (Cu, $Ni)_6Sn_5$ IMC.

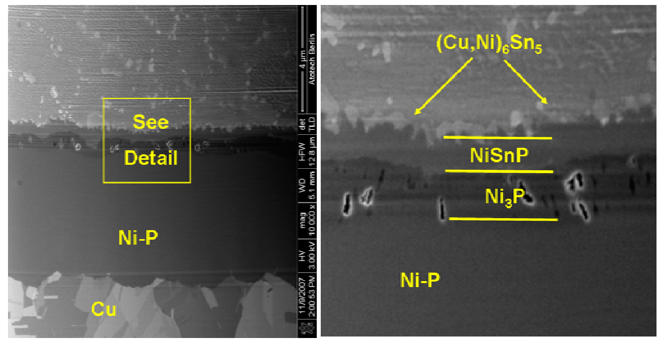

Fig. 12 SEM cross-section image at 10,000x (left) of NiPd surface finish after 5x reflow cycles and detail (right) showing formation of Ni_3P, NiSnP and (Cu, $Ni)_6Sn_5$ IMCs

DISCUSSION

The investigations presented in this paper are part of a continuing comprehensive DOE focused on enhancement of surface finishes for high-density IC substrates (ball-side and C4-side) used in flip chip BGAs applications.

In these investigations, one common denominator for all samples (ENIG and NiPd) was the electroless nickel plating solution. As such, the layer composition was identical for similar nickel deposit thicknesses, including variations of the phosphorus content due to the plating rate used to achieve 4.0 µm, 5.0 µm or 6.0 µm of nickel. The only difference in the surface finishes was the protection layer (gold or palladium) deposited on the nickel layer.

Palladium enrichment in the bulk solder or at the interface is not recognized as the root cause for the improved mechanical performance of the solder joints. This statement is supported by the fact that studies of NiPdAu finishes incorporating a “pure” palladium deposit do not show results that are similar to those demonstrated with the palladium co-deposited with phosphorus.

Through dissolution into the liquid solder alloy, each layer will typically change the local equilibrium composition at the liquid/solid interface or in the bulk solder itself. Therefore, each reaction between solder and substrate metals will alter the chemical composition and mechanical properties of the solder-to-nickel interface. These differences in composition and properties play an important role from a solder joint reliability standpoint.

In comparison to ENIG, the improved performance of the NiPd surface finish in this examination can be more readily understood by reviewing the reactions that occur during soldering and subsequent thermal excursions. After dissolution of the protection layer (i.e. gold or palladium) the nickel diffuses directly into the solder. At the same time, phosphorous from the nickel deposit becomes enriched in the effective joint region. In the case of NiPd, because the palladium layer is co-deposited with phosphorus, the amount of phosphorous required to form the stable Ni_3P IMC is obtained without excessive dissolution of the Ni-P layer. It is theorized that by offering additional phosphorous at the interface, the nickel atoms diffusing into the solder react with the phosphorous from the palladium, forming the Ni_3P IMC. In this manner the formed IMCs are dense and unlike ENIG, the required amount of phosphorus is not dependent on excessive dissolution of the Ni-P layer. Consequently, in the case of NiPd a relatively small amount of nickel dissolves into the solder to form the Ni_3P IMC. Nickel that did not react to form the Ni_3P IMC now forms a NiSn IMC. Unlike ENIG, excessive nickel at the solder interface is not present and, therefore, explains the absence of any significant amount of the $(Cu, Ni)_6Sn_5$ IMC. These IMCs are seen as effective diffusion barriers, which appear to inhibit or slow the dissolution of nickel into solder.

CONCLUSIONS

Based on the investigations performed in this evaluation, the following conclusions are offered:

1. Each of the three NiPd surface finishes exhibited improved solder joint reliability in terms of cold ball pull test performance following Pb-free solder ball attachment. This improved performance was primarily reported in terms of the achieved fracture mode.

2. Following five additional Pb-free reflow cycles, the three NiPd surface finishes were found to withstand greater pull forces in comparison to the ENIG finish. However, only tests of the NiPd finish with the thicker palladium deposit (0.3 µm) were observed to achieve ductile failure modes.

3. The thickness of the electroless nickel deposit was not found to be directly related to cold ball pull performance. However, the rate of electroless nickel plating may have a direct impact on the overall ductility of the Pb-free solder joint. This condition, which is likely related to varying phosphorus content at different plating rates, will be evaluated further.

4. The palladium deposit thickness of the NiPd surface finish has a direct and measureable impact on the cold ball pull test performance. Increasing the palladium thickness from 0.04 µm to 0.3 µm yielded increasingly greater pull strengths and a significantly lower incidence of interfacial (brittle) fracture resulting from this test.

5. Based on the IMC examination of NiPd samples (after ball attachment and after five additional reflow cycles), the relative absence of the $(Cu, Ni)_6Sn_5$ IMC appears to be directly related to its superior performance in cold ball pull testing in terms of both pull strength and failure mode.

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REFERENCES

1. K. Johal, S. Lamprecht, H.J. Schreier and H. Roberts; "Impacts of Bulk Phosphorous Content of Electroless Nickel Layers to Solder Joint Integrity and their Use as Gold- and Aluminum-Wire Bond Surfaces", SMTA 9th Annual Pan Pacific Microelectronics Symposium; January 2004.
2. K. Johal, S. Lamprecht and H. Roberts; "Electroless Nickel / Electroless Palladium / Immersion Gold Plating Process for Gold- and Aluminum-Wire Bonding Designed for High-Temperature Applications"; SMTA 9th Annual Pan Pacific Microelectronics Symposium; January 2004.
3. K. Johal, H. Roberts and S. Lamprecht; "Electroless Nickel / Electroless Palladium / Immersion Gold Process for Multi-Purpose Assembly Technology"; SMTA International Conference; September 2004.
4. K. Johal, H. Roberts, T.Beck, S. Lamprecht, H.J. Schreier, and C. Sebald; "Evaluation of Pb-free BGA Solder Joint Reliability on Ni-based Surface Finishes Using Alternative Shear and Pull Metrologies", SMTA 12th Annual Pan Pacific Microelectronics Symposium; January 2007.