EFFECTS OF ENIG NICKEL CORROSION ON WETTING BALANCE TEST RESULTS AND INTERMETALLIC FORMATION

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ABSTRACT
Since the IPC-4552 rev A for ENIG was introduced there have been many requests for clarification of acceptable and unacceptable levels of nickel corrosion. This paper attempts to further clarify the effects of nickel corrosion on solder wetting balance test results and the resultant intermetallic formed. The study will attempt to produce level 1, level 2, and level 3 corrosion as denoted by IPC-4552 rev A and tabulate wetting balance results and congruity of intermetallic formed.

Key words: ENIG, corrosion, intermetallic formation, solder wetting balance

INTRODUCTION
ENIG was introduced to the industry in the mid-1990s whereby an electroless means of nickel and gold deposition was desired as a replacement for electrolytic nickel and gold for final finish of high density circuits. The advantages of ENIG are well known. In the late 1990s, the issue of "black pad" was discovered and steps were taken by chemical suppliers and board manufacturers to reduce or possibly severely limit the occurrence of this phenomenon. The establishment of the IPC-4552 ENIG specification in 2002 helped steer the industry to the establishment of a more robust ENIG product. Unfortunately, a low level of black pad defects still existed within the industry. Determining root causes of field failures was laborious and many times involved heated discussions between board manufacturers, assembly houses, consultants, and OEMs regarding root cause of solderability failures from boards with an ENIG final finish.

More recently, the IPC 4-14 committee has worked on establishing guidelines for determining acceptable or unacceptable levels of nickel corrosion, which might be an indicator for a poor soldering nickel surface, from cross section analysis of bare boards and the IPC-4552 rev.A was completed in 2017. Unfortunately, some confusion remained regarding determination of Level 1, Level 2, or Level 3 nickel corrosion and more effort was required by the 4-14 committee for clarification.

The latest ENIG specification revision states that Level 2 corrosion must be examined for establishment of a continuous intermetallic formed between the nickel layer and bulk solder by dipping the suspect coupon in molten solder or solder floated for 30 seconds. This paper discusses the attempt to determine if the use of solder wetting balance testing might be able to predict the acceptability of ENIG deposits with Level 2 corrosion or Level 3 corrosion. Solder wetting balance test results after 2x simulated solder reflow conditions were used.

EXPERIMENTAL AND RESULTS
The objective was to create level 2 and level 3 product. The latest version of the amendment of IPC 4552 A (in progress) describes how to assess the level of corrosion (level 0, 1, 2 and 3). After the assessment is made, 7 locations in the worst through hole or 5 locations on the worst SMT feature are classified based on corrosion levels. Based on this classification a "product rating" is extrapolated into: Acceptable or Rejectable, with a category in between that may need further evaluation. The product rating is based on statistically coming up with a single value for the product from the 7 or 5 evaluations of the corrosion levels found.

To produce a Rejectable "Product Rating" a highly corroded ENIG sample was needed. In the effort to produce a rejectable product rating; 3 main variables were explored:

- The first variable examined was type of immersion gold. Three (3) different types of immersion golds were used; the first "A" was considered a more aggressive type with a higher deposition rate, the second "B" was milder, high efficiency product with a more controlled immersion reaction and the third "C" was a "Reduction Assisted" immersion gold that exhibited minimum corrosion.
- The second variable was the dwell time in the gold bath (increasing the dwell time beyond vendor specifications). This was based on the concept that increased dwell time in the gold bath is the primary contributor to nickel corrosion.
- The third variable was solution agitation in both the electroless nickel and the immersion gold baths; this was achieved by using a fast and a slow stirring bar in the beakers at time of plating.

The test vehicle of choice was the solder wetting balance coupons shown in Figures 1 and 2. The coupons are double sided boards plated with 25 microns of copper.

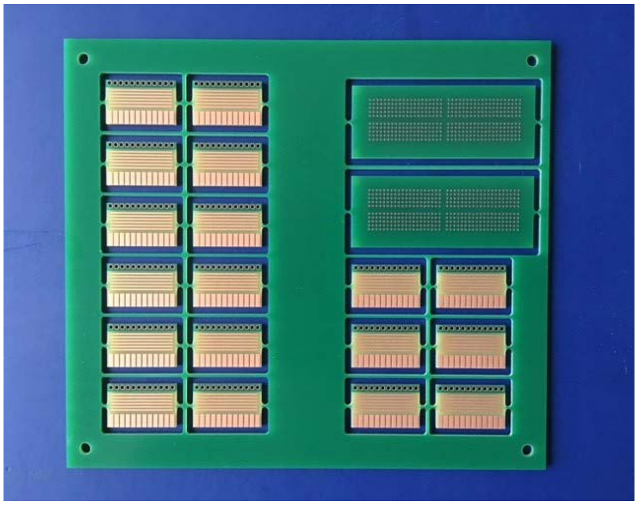


Figure 1. Wetting Balance coupon array

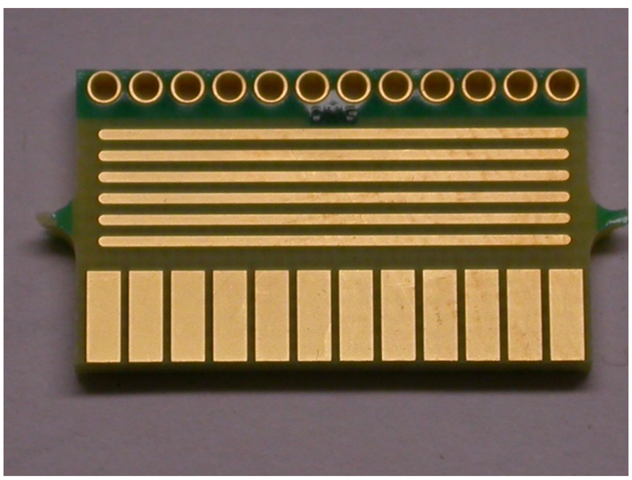


Figure 2. Individual wetting balance coupon from the array

Thickness measurements were taken using a Fischer XDV-u XRF utilizing a 20 micron spot size with capillary optics. Wetting balance testing was performed according to J-STD-003 and IPC-4552 rev A specifications using a Metronelec ST-88 Wetting Balance tester. Cross section analysis was performed with epoxy cured mounts using a standard sequence of grinding and polishing media: 120, 240, 320, 600, 1000, and 2000 grit SiC and 0.3micron and 0.05 micron Al_2O_3 media. Cross section images were taken using a Nikon Epiphot 200 metallograph and JEOL JSM-6010LA Scanning Electron Microscope.

ENIG Plating

ENIG plating was performed at 0.2 ft^2/gallon bath loading for the electroless nickel solution. The coupons were plated according to the standard ENIG plating sequence shown in Table 1.

Table 1. Plating Process Sequence

Process Step	**Time, min.**	**Temp deg. F**
Mild Acid Clean	5	120
DI Rinse	2	RT
Persulfate Etch	2	RT
DI Rinse	2	RT
2% Sulfuric Acid	1	RT
Pd Catalyst	1.25	RT
DI Rinse	2	RT
Mid-P E'less Nickel	22, 24	176
DI Rinse	1	RT
Immersion Gold A	8, 18, 25, 40	185
Immersion Gold B	30	185
Immersion Gold C	6, 12, 24	176
DI Rinse	4	RT
Dry		

Results

Table 2 depicts the wetting balance results for the variable conditions used in this study. Slight trends were seen for a decrease in wetting time versus solution speed while plating, however no real differences were seen with final wetting forces. All coupons appeared to be wetted properly, even the sample with extremely thick gold from immersion gold Solution A. Figures 3, 4, and 5 show the wetting balance curves after 2x reflows as noted. These are typical of the results seen throughout the testing.

Table 2. Wetting test results as plated and after 2x reflow simulation

EXPERIMENTAL DESIGN					THICKNESSS		WETTING BALANCE RESULTS			
Ni plating mins	**Nickel stir bar**	**Gold Bath used**	**Gold plating mins**	**Gold stir bar**	**Avg. Ni µin**	**Avg. Au µin**	**Wet time as rec'd**	**Wet force as rec'd**	**Wet time 2x reflow**	**Wet force 2x reflow**
22	fast	Gold A	8	Fast	150	2.0	2.0	0.20	2.6	0.18
24	fast	Gold A	18	Fast	160	3.0	1.8	0.23	2.7	0.21
24	fast	Gold A	25	Fast	180	4.0	1.9	0.12	2.7	0.19
24	fast	Gold C	6	Fast	150	2.4	1.3	0.22	2	0.22
24	fast	Gold C	12	fast	160	3.7	1.2	0.23	1.9	0.22
22	slow	Gold B	30	Slow	170	3.4	1.6	0.23	1.9	0.23
24	slow	Gold C	24	Slow	190	5.6	1.1	0.25	1.2	0.25
24	slow	Gold A	25	Slow	160	3.9	1.6	0.22	1.9	0.22
24	slow	Gold A	40	Slow	190	6.3	1.4	0.21	1.6	0.23

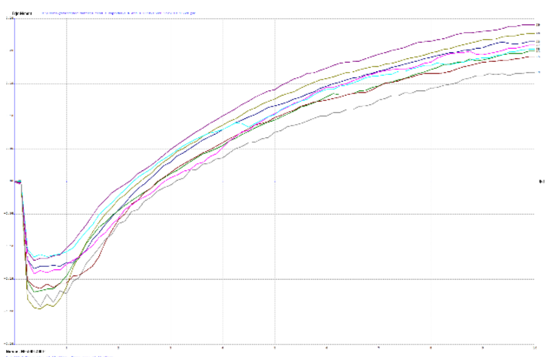


Figure 3. WB curve after 2x reflow for gold bath A at 4 microinches with high solution agitation

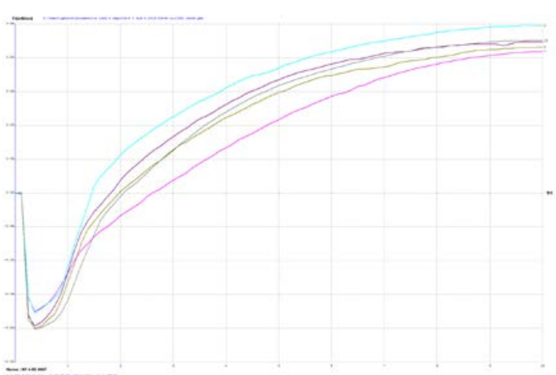


Figure 4. WB curve after 2x reflow for gold bath B at 3.4 microinches with low solution agitation

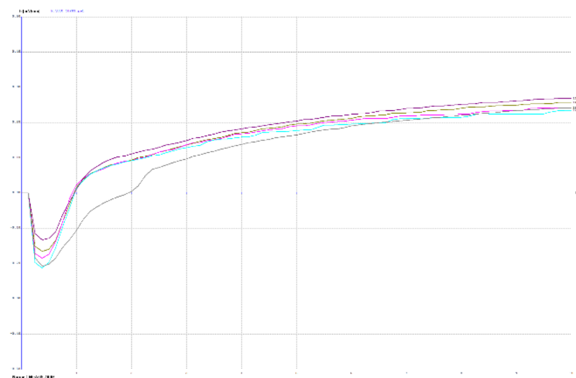


Figure 5. WB curves after 2x reflow for gold bath C at 5.6 microinches with low solution agitation

Coupons were examined at 200 X for corrosion. The worst sample (maximum dwell time in gold bath "A") was examined in great detail at 1000 X in 7 different locations refer to Figure 2. Each location was assigned a Corrosion Level. Corrosion evaluation into 3 Levels was accomplished as follows according to a pre-view of the Amended IPC specification 4552 Rev A Figure 6 shows the areas of investigation, seven locations per hole.

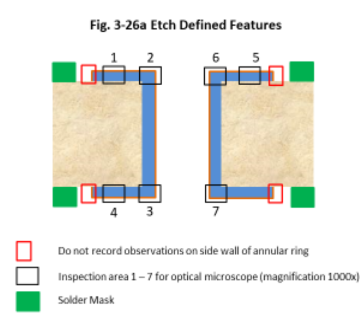
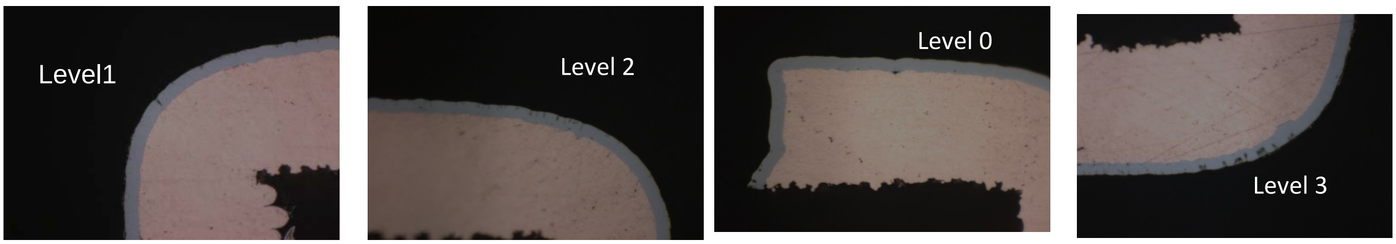


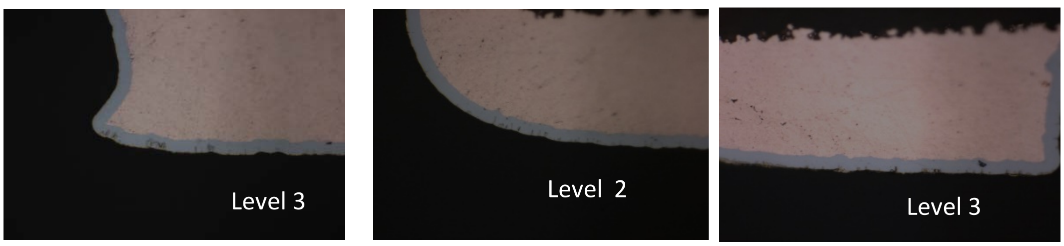
Figure 6. Locations of cross section checking at 1000x

Corrosion Level 1: Number of spikes defects <10. With all spike depth <20% of the nickel thickness Figure 6 shows an example of a corrosion spike

Figure 7 shows the results for the corrosion checking for immersion gold bath A with maximum immersion gold dwell time.

Corrosion Level 3: Number of spikes >10. And >5 defects penetrate more than 40% of the nickel deposit
Corrosion Level 2 : All other observations





Level 0 1
Level 1 1
Level 2 3
Level 3 2

Product Rating = 2

Figure 7. Examination of the seven locations from the longest dwell time in immersion gold bath A

The 7 corrosion ratings were tallied to give a "Product Rating" according to the following:

Level 0: One location noted
Level 1: One location noted
Level 2: Three locations noted
Level 3: Two locations noted

Based on the results, less than 40% of the locations saw level 3 corrosion and less than 60% of the locations saw level 1 corrosion.

Figure 8 shows a common set of small corrosion spikes.

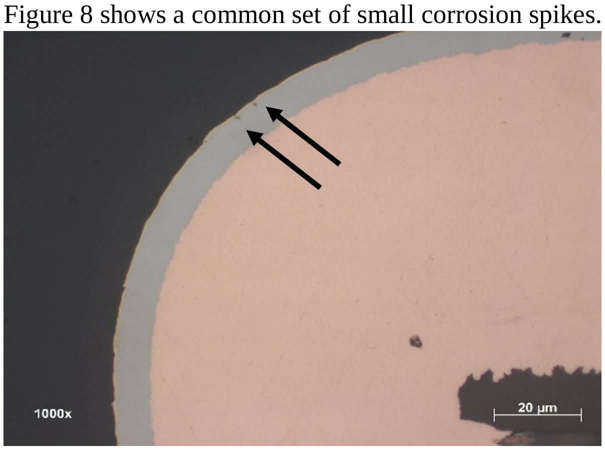


Figure 8. Two corrosion spikes noted, for reference

It has been known in the industry that the holes generally show more nickel corrosion than SMT pads, thus the requirement to examine holes rather than SMT pads if holes are available for analysis. For the case of examining wetting balance coupons, additional cross sectional analysis was undertaken for the samples with the longest dwell time in gold solution A. Figures 9, 10, and 11 show cross section results using backscatter SEM analysis at 1000x. One side of the wetting balance pad shows preferential nickel damage while the rest of the pad appears normal. This may have been due to the high solution flow rate making the nickel susceptible to corrosion in the immersion gold.

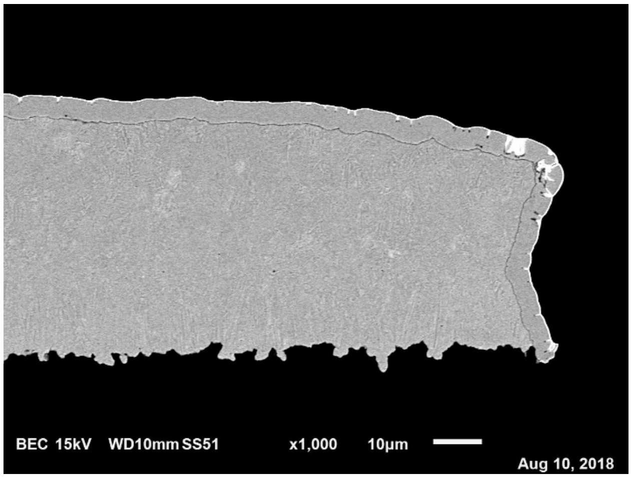


Figure 9. SEM backscatter image at 1000x for immersion gold A plated at 6.3 microinches of gold, right side of pad level 3 corrosion

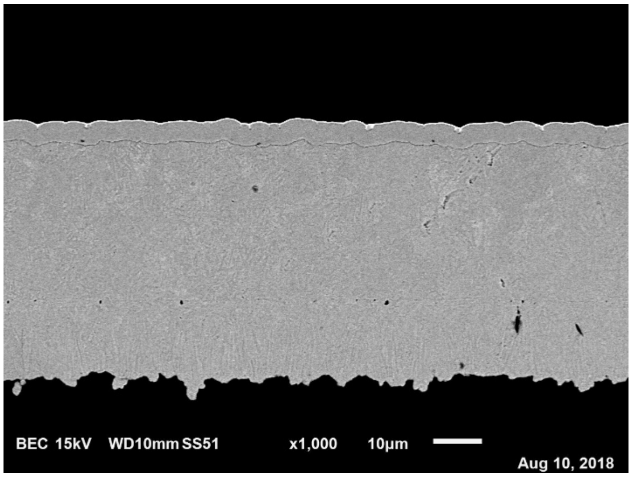


Figure 10. SEM backscatter image for immersion gold A plated at 6.3 microinches, middle of pad, level 1 corrosion

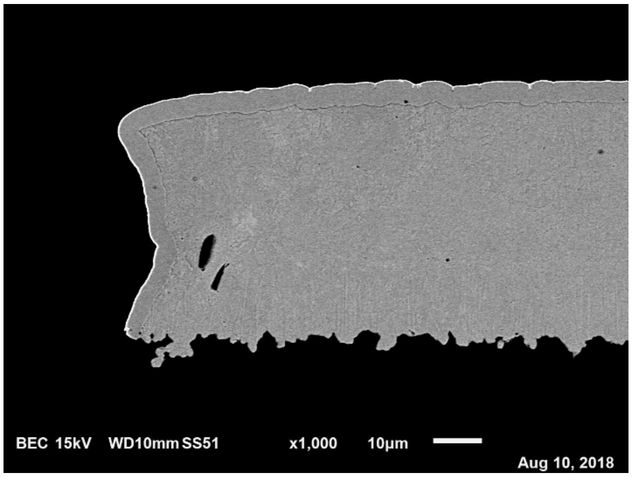


Figure 11. SEM backscatter image for immersion gold A plated at 6.3 microinches, left of pad, level 1 corrosion

An additional level of examination required is solder dipping and evaluation of contiguous IMC (intermetallic compound formed between nickel and tin). In all the tests that were run, no "Rejectable" product rating was produced. All samples examined were deemed acceptable. The coupon in figure 7 (level 2 corrosion) was subjected to a solder dip and the IMC was examined. The IMC formed was contiguous indicating an acceptable level 2 corrosion as shown in Figure 12.

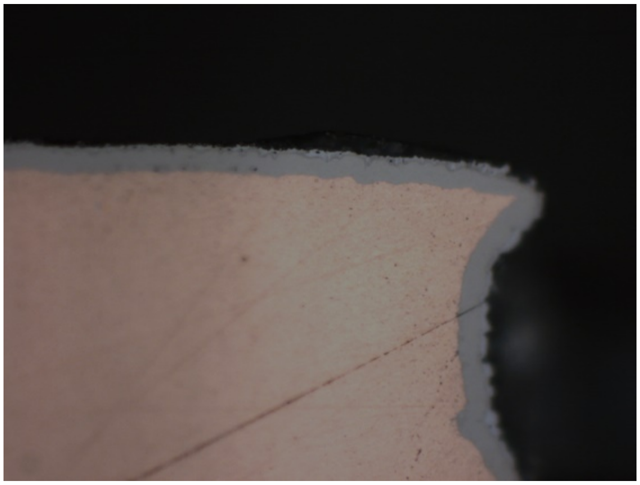


Figure 12. Contiguous IMC for sample with level 2 corrosion (sample etched to remove bulk solder)

EDS mapping was also used to check for continuity of intermetallic formation, in this case for bath A with 3.9 microinches of gold. Results shown in Figure 13. A continuous intermetallic was seen (sample etched to remove bulk solder).

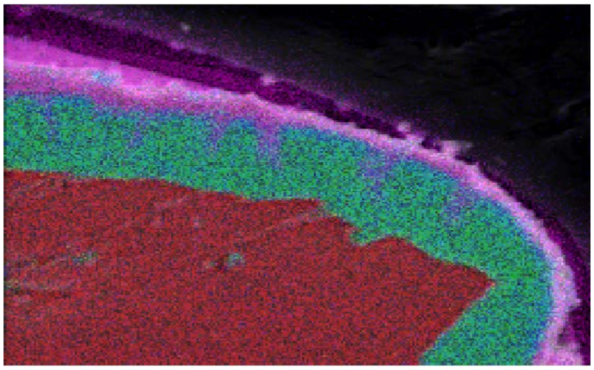


Figure 13. EDS mapping cross section image after 2x reflow and 30 second solder dip on level 2 corrosion deposit (immersion gold A, 3.9 microinches). Red = copper, green = nickel, pink = IMC

Conclusions

Increased dwell time in the immersion gold alone did not produce rejectable product. Solution agitation did not influence the degree of corrosion overall although on a micro scale it may have given localized corrosion on the leading edge of solution flow. The type of gold showed differences in nickel corrosion as evidenced by the cross-section analysis. Under the most aggressive dwell time with the more aggressive gold bath "A" occasional Level 2 and level 3 corrosion was found. The occurrences and frequency of these levels of corrosion were not enough to classify the product as "Rejectable". It is worth noting that the "Reduction Assisted" immersion gold "C" showed no level 2 or level 3 corrosion on any of the cross-sections that were evaluated, regardless of dwell time and final gold thickness.

Further Work

Due to time constraints, the authors decided to publish their findings without achieving the objective of determining if Wetting Balance testing could be a referee for ENIG corrosion acceptability.

Special acknowledgement and thanks given to Gerard O'Brien of ST&S Group for performing the wetting balance testing and cross sectional analysis.