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.

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.

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 test results after 2x simulated solder reflow conditions were used.
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](image1)

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 Al2O3 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 ft²/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**

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

**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

<table>
<thead>
<tr>
<th>Ni plating mins</th>
<th>Nickel stir bar</th>
<th>Gold bath used</th>
<th>Gold plating mins</th>
<th>Gold stir bar</th>
<th>Avg. Ni µin</th>
<th>Avg. Au µin</th>
<th>Wet time as rec'd</th>
<th>Wet force as rec'd</th>
<th>Wet time 2x reflow</th>
<th>Wet force 2x reflow</th>
</tr>
</thead>
<tbody>
<tr>
<td>22 fast</td>
<td>Gold A</td>
<td>8</td>
<td>Fast</td>
<td>150</td>
<td>2.0</td>
<td>2.0</td>
<td>0.20</td>
<td>2.6</td>
<td>0.18</td>
<td></td>
</tr>
<tr>
<td>24 fast</td>
<td>Gold A</td>
<td>18</td>
<td>Fast</td>
<td>160</td>
<td>3.0</td>
<td>1.8</td>
<td>0.23</td>
<td>2.7</td>
<td>0.21</td>
<td></td>
</tr>
<tr>
<td>24 fast</td>
<td>Gold A</td>
<td>25</td>
<td>Fast</td>
<td>180</td>
<td>4.0</td>
<td>1.9</td>
<td>0.12</td>
<td>2.7</td>
<td>0.19</td>
<td></td>
</tr>
<tr>
<td>24 fast</td>
<td>Gold C</td>
<td>6</td>
<td>Fast</td>
<td>150</td>
<td>2.4</td>
<td>1.3</td>
<td>0.22</td>
<td>2</td>
<td>0.22</td>
<td></td>
</tr>
<tr>
<td>24 fast</td>
<td>Gold C</td>
<td>12</td>
<td>fast</td>
<td>160</td>
<td>3.7</td>
<td>1.2</td>
<td>0.23</td>
<td>1.9</td>
<td>0.22</td>
<td></td>
</tr>
<tr>
<td>22 slow</td>
<td>Gold B</td>
<td>30</td>
<td>Slow</td>
<td>170</td>
<td>3.4</td>
<td>1.6</td>
<td>0.23</td>
<td>1.9</td>
<td>0.23</td>
<td></td>
</tr>
<tr>
<td>24 slow</td>
<td>Gold C</td>
<td>24</td>
<td>Slow</td>
<td>190</td>
<td>5.6</td>
<td>1.1</td>
<td>0.25</td>
<td>1.2</td>
<td>0.25</td>
<td></td>
</tr>
<tr>
<td>24 slow</td>
<td>Gold A</td>
<td>25</td>
<td>Slow</td>
<td>160</td>
<td>3.9</td>
<td>1.6</td>
<td>0.22</td>
<td>1.9</td>
<td>0.22</td>
<td></td>
</tr>
<tr>
<td>24 slow</td>
<td>Gold A</td>
<td>40</td>
<td>Slow</td>
<td>190</td>
<td>6.3</td>
<td>1.4</td>
<td>0.21</td>
<td>1.6</td>
<td>0.23</td>
<td></td>
</tr>
</tbody>
</table>

**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.

**Corrosion Level 2**: All other observations.

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

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

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.

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**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.
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.

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).

**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.