Comparing Soldering Results of ENIG and EPIG Post Steam Exposure

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Abstract
ENIG, electroless nickel immersion gold is now a well-regarded finish used to enhance and preserve the solder-ability of copper circuits. EPIG, electroless palladium immersion gold, is a new surface finish also for enhancing and preserving solder-ability but with the advantage of eliminating Electroless Nickel from the deposit layer. This feature has become increasingly important with the increasing use of high frequency PWB designs whereby nickel’s magnetic properties are detrimental. We examine these two finishes and their respective soldering characteristics as plated and after steam aging and offer an explanation for the performance deviation.

Comparing the results of steam age test data shows a clear benefit of EPIG over ENIG. After even a short duration exposure to steam, ENIG finishes failed to solder. Much longer steam exposures produced little to no effect on EPIG plated samples. Rapid oxidation of the electroless nickel phosphorous layer when stressed with heat and moisture explains the superior EPIG result.

The ability to demonstrate excellent solder-ability following steam exposure represents increased fabrication reliability under non-ideal storage conditions.

Introduction
Assessing the solder-ability of a given final finish can range from a simple solder dip as coated or plated to quite complicated procedures involving the final finish type, solder alloy, flux characteristics, test preconditioning, soldering apparatus (wetting balance) and part geometries. Focusing on two types of solder-able finishes and one preconditioning procedure allows the clear reporting of results and expectations that those finishes afford.

Morris, Lukaszewski and Genthe1 describe a need for verification methods for accelerated testing of electronics. Specific to soldering operations, steam aging of PCBs prior to solder wick testing is seen as not only quick and inexpensive but also a reliable shelf life predictor. Eight hours steam aging is reported to be equivalent to twelve months “shelf life” exposure for tin lead (60/40) systems only2.

There are many solder-able surface finishes used on copper printed circuit boards (PCB). They range from thin organic coatings to heavy metal plates, 1-3 microns of gold plate as an example. Plated tin lead or molten tin lead coatings (HASL) are early predecessors of the diversified finishes that are available today. These leaded alloys consistently show high resistance to steam aging and solder quality retention. Most other PCB coatings do not fare well after steam exposure. Currently, all other final surface finishes for PCBs fail to maintain acceptable solder-ability performance after one hour steam exposure.2

Of the many solder-able finishes available two are tested here. One, ENIG has been available for over twenty years. It has a track record of robust soldering performance and is generally ascribed a twelve month shelf life before the degrading of its soldering performance. EPIG, on the other hand, is a new finish with EPIG test results showing very good solder wetting and force values. The overall shelf life has yet to be determined. However, test results in this paper comparing ENIG performance to EPIG performance under steam exposure stress will shed light on this question.
Steam exposure is not the only approach to determine how well a finish will hold up to environmental conditions. Other tests include: thermal cycling, which shows the effect of multiple solder applications; real time environmental exposures, which yields the most reliable data; and mixed flowing gas testing, a precise chamber test where gas type and concentration are stringently controlled. Steam exposure, on the other hand, provides a quick, reliable and inexpensive indicator of soldering shelf life.

**Methodology**

A steam aging preconditioning or stressing procedure was exacted upon both ENIG and EPIG coupons per IPC J-Std-002 and J-Std-003. Using the stress of steam exposure helps to determine the durability and robustness of each solder-able surface. Robustness is a difficult term to define and measure accurately. Here robustness is intended to mean that a PCBs shelf life vastly exceeds the typical 12 months, can withstand more solder refows and holds soldering properties regardless of the normal fabrication processes employed.

Soldering test materials used include the application of a mild flux just prior to solder testing and the use of lead free solder–Sn3Ag0.5Cu joining metal. These were limited to single non-variable options to simplify testing protocol. Both of these represent common commercial soldering practice.

This assessment observes the solder wicking onto a PCB test coupon plated with either ENIG or EPIG. The testing is conducted on an as plated condition and after exposure to steam. The duration of the steam exposure is in units of hours and continues until failure or a total of eight continuous hours of exposure. It is expected that failure to solder wet will coincide with the oxidation of plated films or the base copper to an extent whereby fluxing is no longer able to clean and remove those oxides.

The PCBs plated for testing consisted of standard solder wicking coupons as shown in - Figure 1.

![Figure 1: Solder test coupon used for solder wick testing](image)

The ENIG film thickness used equals 3.75 microns of nickel phosphorus with 0.075 to 0.1 microns immersion gold on top. Thickness levels were not changed so as not to be a variable – (Figure 2).

![Figure 2: Diagram of ENIG plating tested](image)
The EPIG film thickness used equals 0.375 microns palladium phosphorus with 0.025 microns immersion gold on top. Again, thickness was held to one level for testing duration – (Figure 3).

![Figure 3: Diagram of EPIG plating tested](image)

The solder flux used was composed of 25% +/- 0.5% colophony, 0.39% +/- 0.01% diethylammonium chloride and the balance isopropyl alcohol.

The solder alloy employed was a SAC 305 alloy consisting of 3% silver, 0.5% copper and the balance tin. The solder temperature was set at 255°C. The contact time between the solder and sample measures wetting time and soldering final force.

A production solder wetting balance was used to measure time to wet and solder force – Figure 4.

![Figure 4: Wetting balance used to test solderability](image)

A production XRF machine was used to measure plating thicknesses – Figure 5.

![Figure 5: XRF used to measure plating thicknesses of the ENIG and EPIG test samples](image)

The steam aging apparatus used was as outlined in IPC J-STD-002/003 as shown in – Figure 6.
The solder-ability evaluation procedure engaged was to first plate solder test coupons with ENIG and EPIG. Both of these plating processes utilized the exact same preparation cycle to clean and activate the base copper. Electroless palladium was simply substituted for electroless nickel to create an EPIG finish. The singular preparation and plating sequence utilized are outlined in Table 1.

### Table 1: Plating cycles used to prepare ENIG and EPIG test samples

<table>
<thead>
<tr>
<th>Plating Step</th>
<th>Temperature</th>
<th>Time</th>
<th>Agitation</th>
<th>Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clean</td>
<td>49°C</td>
<td>5 minutes</td>
<td>Stirring</td>
<td>Acidic</td>
</tr>
<tr>
<td>DI Water Rinse</td>
<td>21°C</td>
<td>1 minute</td>
<td>Stirring</td>
<td></td>
</tr>
<tr>
<td>Etch</td>
<td>27°C</td>
<td>1 minute</td>
<td>Stirring</td>
<td>Peroxide/Sulfuric</td>
</tr>
<tr>
<td>DI Water Rinse</td>
<td>21°C</td>
<td>1 minute</td>
<td>Stirring</td>
<td></td>
</tr>
<tr>
<td>Activate</td>
<td>27°C</td>
<td>2.5 minutes</td>
<td>Stirring</td>
<td>Acidic Dissolved Palladium</td>
</tr>
<tr>
<td>DI Water Rinse</td>
<td>21°C</td>
<td>1 minute</td>
<td>Stirring</td>
<td></td>
</tr>
<tr>
<td>Electroless nickel or Electroless palladium</td>
<td>82°C</td>
<td>20 minutes</td>
<td>Stirring</td>
<td>Nickel 7%Phosphorus Palladium 3%Phosphorus</td>
</tr>
<tr>
<td></td>
<td>49°C</td>
<td>15 minutes</td>
<td>Stirring</td>
<td></td>
</tr>
<tr>
<td>DI Water Rinse</td>
<td>21°C</td>
<td>1 minute</td>
<td>Stirring</td>
<td></td>
</tr>
<tr>
<td>Immersion gold</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Conditions used for EN (ENIG)</td>
<td>81°C</td>
<td>10 minutes</td>
<td>Stirring</td>
<td>Gold Potassium Cyanide 2g/l Gold Metal pH 5.2</td>
</tr>
<tr>
<td>Conditions used for EPd (EPIG)</td>
<td>85°C</td>
<td>20 minutes</td>
<td>Stirring</td>
<td></td>
</tr>
<tr>
<td>DI Water Rinse</td>
<td>21°C</td>
<td>2 minutes</td>
<td>Stirring</td>
<td></td>
</tr>
<tr>
<td>Dry</td>
<td></td>
<td></td>
<td></td>
<td>Forced Air</td>
</tr>
</tbody>
</table>

Plating times were adjusted in the immersion gold step as deposition is more vigorous on nickel versus palladium as predicted by the relative corrosion potentials of -0.250V for nickel and +1.498V for gold, a delta of -1.748V compared to +0.987V for palladium and +1.498V for gold a delta of -0.511V. The larger delta negative potential between nickel and gold shows that in the case of immersion plating gold deposits more rapidly on to nickel than on to palladium.

Post plating, solder coupons were dried and stored in a sealed and desiccated environment until steam stressing and solder wick testing was performed.

Coupons were steam aged, as outlined in J-STD-002/003, for one to eight continuous hours. This was conducted in one-hour increments until solder failure or until eight hours of total steam aging time had been achieved.

**Results and Discussion**

Test results show that ENIG is an outstanding final finish. ENIG, however, is susceptible to rapid solder non-wetting after steam exposure. On the contrary, EPIG shows little to no susceptibility in comparison. Speculation as to why such a difference between the two finishes suggests a strong interaction of steam on exposed nickel phosphorus. The nickel phosphorus is made available to steam penetration through the thin porous gold top coat present in the ENIG deposit.
Figures 7 through 12 show the results of solder wetting tests using the solder wetting balance. The X axis measures contact time between the plated copper pads and the liquid solder. The time units are in seconds. Total contact time for each measurement is ten or twenty seconds.

The Y axis of the wetting chart measures the wicking force of the solder as it wets the plated copper test pads. The force units are mN/mm for these tests. The force climbs as a greater area of the test pad is covered with solder. The initial dip in force represents the initial contact between the coupon and solder. As the test area wets with solder the force climbs to a maximum. That maximum is the wetting force for that particular test. The time needed for the force to cross or intersect the zero-force line is considered to be the total wetting time for that particular test.

Results are interpreted as positive when the wetting force rapidly ascends to a maximum and holds at that level. Should the wetting force not pass through zero for the duration of the test then a non-wetting solder failure is assigned. Likewise, solder failure is also observed when the wetting force reaches a maximum and then decreases. That type of chart represents solder dewetting for the test.

Comparing the solder-ability results of ENIG (Figure 7) and EPIG (Figure 8) show that both provide excellent wetting times and soldering forces in the as-plated condition.

![Figure 7: ENIG wetting time and force as plated](image)

![Figure 8: EPIG wetting time and force as plated](image)

After just one hour of steam aging the ENIG finished sample fails to wet solder (non-wet condition) – Figure 9. The EPIG sample continues to wet well to solder, unaffected by one-hour steam preconditioning – Figure 10.
Through eight hours steam aging the EPIG samples solder well with minor reduction in wetting force – Figure 11 (Note: this figure shows a twenty second contact duration).

These test results show that steam has a dramatic negative impact on the solder-ability of ENIG even after one-hour exposure. EPIG on the other hand remains quite solder-able with almost no change in wetting times and force after eight hours of continuous steam exposure. The reason for the performance difference is thought to be a rapid formation of a nickel oxide on the electroless nickel phosphorous surface during steam stressing inhibiting wetting and the formation of a nickel-tin intermetallic which reduced the ability to solder well. The formation of a tin nickel intermetallic is required for good solder wetting to occur.

K. Yokomine, et al⁴, describe results of testing the depth of oxygen found on the electroless nickel surface from an ENIG film as plated and after immersion in an aqueous cleaner at 80°C. Using ESCA (Electron Spectroscopy for Chemical Analysis) equipment, a three to four-fold increase in oxygen depth post dipping ENIG at 80°C versus room temperature measurements was recorded⁴.
Palladium’s inherent resistance to oxidation provides a robust oxidation free protective layer for solder applications. Also, palladium dissolves in the molten solder allowing the formation of a copper-tin intermetallic directly with the underlying copper surface.

Another contributing reason for the soldering performance difference between ENIG and EPIG is the tin intermetallic formed. ENIG forms a nickel-tin intermetallic whereas EPIG forms a copper-tin intermetallic. The gold and palladium layers readily dissolve into solder, so the bonded interface is directly between copper and tin. Hence, any thin oxidation or other environmental contamination of the palladium layer is essentially dissolved and thus inconsequential to the copper tin bonding interface.

A follow-up verification test was made by coating ENIG solder coupons with an organic anti-tarnish top coat. The top coat is a solderable organic anti-tarnish coating that is intended to be a barrier to oxygen penetration. The coating is applied on top of the immersion gold layer essentially sealing the thin porous gold.

Solder wetting tests using the organic top coat on ENIG substantially eliminates the adverse effects of steam conditioning. Starting at one-hour steam exposure and through to eight hours limited the degradation in the ENIG’s solderability which indicates that the nickel layer remained relatively oxide free with solder wetting occurring after eight hours steam stressing – Figure 12.

Figure 12: Organic anti-tarnish protected ENIG wetting balance test results after eight hours steam stressing

<table>
<thead>
<tr>
<th>Test Condition</th>
<th>Time to positive wetting (seconds)</th>
<th>Wetting Force (mN/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ENIG “as plated”</td>
<td>0.51</td>
<td>0.21</td>
</tr>
<tr>
<td>EPIG “as plated”</td>
<td>0.18</td>
<td>0.17</td>
</tr>
<tr>
<td>ENIG “after one-hour steam”</td>
<td>Null</td>
<td>-0.02</td>
</tr>
<tr>
<td>EPIG “after one-hour steam”</td>
<td>0.12</td>
<td>0.19</td>
</tr>
<tr>
<td>EPIG “after eight hours steam”</td>
<td>0.20</td>
<td>0.16</td>
</tr>
<tr>
<td>ENIG/Anti-tarnish “after eight hours steam”</td>
<td>2.0</td>
<td>0.25</td>
</tr>
</tbody>
</table>

Summary / Conclusions
The successful soldering to PCB’s is lessened with time and exposure to environmental conditions most notably heat and moisture. Excessive exposure renders boards unsuitable for final fabrication / assembly based on poor solder-ability resulting in disposal before or worse after expensive components have been mounted. Enhanced soldering reliability is achieved by rendering heat and humidity ineffectual in reducing solderability.
Steam aging per IPC J-STD-002 and J-STD-003 is a convenient and low cost and time method of evaluating the shelf-life of finishes used on PCBs. Two finishes, ENIG and EPIG were tested for solder wetting pre and post steam stressing exposure. Test data shows that the steam stressing had little influence on the EPIG board surface finish solderability while ENIG board surface finish rapidly becomes unsolder-able. The swift oxidation of the nickel layer by steam is thought to be the cause. Supporting evidence was provided by applying a secondary organic anti-tarnish coat to the ENIG samples prior to steam stressing. Oxidation of the nickel layer was then suppressed. The organic film reduced nickel oxidation after steam stressing with improved solder wetting.

References
2. O’Brian, G., Chairman of 5-23A, Responsible for IPC J-STD-003. Verbal communication

Click here for information on EPIG, ENIG, ENEPIG and other products from Uyemura, the global leader in PCB final finishes.