ACHIEVING A SUCCESSFUL ENIG FINISHED PCB UNDER REVISION A OF IPC 4552 MACDERMID ENTHONE

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ABSTRACT
The 4-14 IPC Standards Committee recently created a revision to the IPC4552 specification for Electroless Nickel/Immersion Gold (ENIG) finished Printed Circuit Boards (PCB). Revision A brings a more comprehensive evaluation of metal layer thicknesses measurement, composition and introduces, for the first time, a quality aspect for nickel corrosion which has been historically connected to a defect called black line nickel or black pad.

The introduction of these revisions will ensure a higher level of quality for conforming ENIG deposits but it will also present some challenges in achieving and consistently delivering the required level of quality from PCB fabrication.

IPC4552 Revision A requires that the PCB fabricator demonstrate capability in measuring and maintaining electroless nickel thickness and composition. The reduced minimum gold thickness and newly introduced maximum gold thickness specifications challenge not only measurement systems but also process control to comply with statistical restrictions on conforming ENIG product.

The PCB Fabricator will also have new responsibility to establish control of the electroless nickel corrosion levels within given acceptability guidelines.

Key words: IPC4552, ENIG, deposit thickness, electroless nickel composition, electroless nickel corrosion.

INTRODUCTION

Electroless Nickel/Immersion Gold

In the mid 1990's, the IPC started a project to evaluate alternative surface finishes to Hot Air Solder Level (HASL). The project was designed as a response to the looming European ban of lead for the electronics industry. The ban would eliminate lead in components, solder materials and the HASL surface finish. The impending doom of a transition from lead hung over the industry for almost 10 years, it still plagues some market segments today. The release of the 1996 ITRI/October project which tested the reliability of alternative surface finishes, enabled a growth of those finishes [1]. Companies were more comfortable with the short and long term reliability of "alternative" finishes. Today at least five are used regularly with new ones coming to the market sporadically. The main finishes include organic solderability preservative (OSP), immersion tin, immersion silver, electroless nickel/immersion gold, and electroless nickel/electroless palladium /immersion gold (ENEPIG). From the perspective of volume, OSP is the most heavily used, from a monetary perspective, even excluding the cost of gold metal, ENIG is the leading surface finish [2].

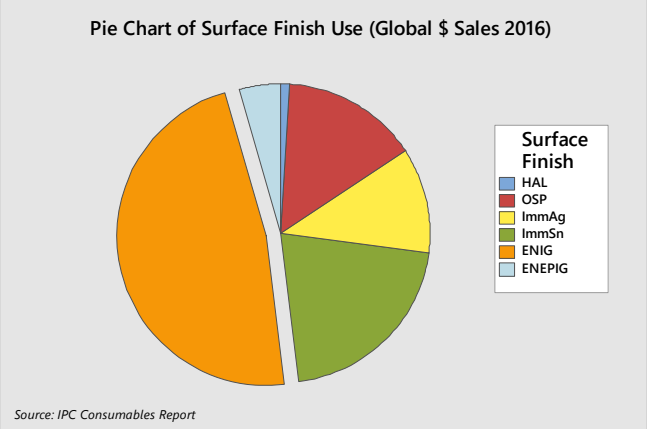


Figure 1: Surface Finish Process Chemistry by $.

All surface finishes have their strengths and weaknesses. There is no one finish that suits all needs for designers, PCB fabricators, assemblers and end users. With that said, this paper focuses on ENIG and how to achieve the best process control and product performance from the finish and compliance to IPC4552 Revision A.

Historically, Hot Air Solder Level (HASL) was the surface finish of choice to protect copper from oxidation prior to soldering components to the surface. As circuits become more complex and smaller features were introduced, the industry required alternatives to that finish that were flatter, less aggressive on the substrate material and more environmentally friendly. The industry began using electroless nickel/immersion gold (ENIG) in greater volumes. The chemical mechanism of plating a two layer deposit and chemical interactions with other materials of the

PCB build made this a much more challenging finish than HASL. Even with the challenges, the cost and the time associated to finishing boards with ENIG, the finish is still in high demand today.

In the 1990's with the growth in new designs, the launch of BGA components for the telecommunications industry [3] and the overall increase in ENIG usage, the industry learned a great deal about the chemistry, variables in fabrication that affected the resultant product and the reliability of this surface finish. Yet, there still seems to be a level of uncertainty or unpredictability that the industry deals with and accepts. The IPC 4552A document creates a guideline to decrease any uncertainty and create a more predictably performing ENIG finish. This paper is designed to better explain the need for the revisions, methods for achieving a successful deposit and a new process with much better control and consistency to bring more ease to the PCB fabrication process increasing conformance to the new IPC 4552 specification.

ENIG maintains its popularity and heavy usage due to many advantages including long shelf life, resistance to tarnish and whisker growth, consistent solderability especially after multiple lead free reflow excursions, it is aluminum wire bondable and its suitability for contacts /key pads. Another important advantage as explained by end users is that many areas on a finished printed circuit board assembly (PCBA) are left unsoldered. ENIG provides strong resistance to environmental conditions and leaves an exposed metal with a reliable contact surface that does not discolor.

A closer look at the disadvantages of the conventional process will create an outline for the main discussion points of this paper. For an end user, it is an expensive step in the PCB manufacture process despite being limited to 15% or less of the outer board area. For the PCB fabricator, the challenges are greater; high temperature chemistry, a complex process with multiple steps and a final finish that is not reworkable make ENIG one of the more challenging production finishes. Finally, and potentially the biggest concern for the PCB fabricator, assembler and end user alike is the potential for Black Line Nickel and resultant reliability issues not detected until after the board has been populated and is at its most expensive state. It was a painful and costly lesson in the 90's which a hangover for most involved at the time still remains.

	HASL	OSP	Imm Silver	Imm Tin	ENIG	ENEPIG
Planarity	No	Yes	Yes	Yes	Yes	Yes
Solderjoint	Cu-Sn	Cu-Sn	Cu-Sn	Cu-Sn	Ni-Sn	Ni-Sn
Relative Cost	$	$	$$	$$	$$$	$$$
IPC Shelf Life	No Specification	No Specification	12 months	12 months	12 months	12 months
Reflows	6	4	6	2	6	6
Contact	E-Test, ICT	Difficult	E-Test, ICT, Keypad	E-Test	E-Test, ICT, Keypad	E-Test, ICT, Keypad
Press Fit	Good	Good	Good	Good	Good	Good
Au Wirebond	No	No	No	No	No	Yes
Al Wirebond	No	No	Yes	No	Yes	Yes

Figure 2: Surface Finish Attribute Comparison Chart

IPC 4552 Revision A

Overall, the IPC4552 Revision A [3] increases the level of scrutiny for the ENIG process and final deposit. The first order of change is the gold deposit thickness control and measurement. An introduction to measurement system error and provisions for non-conforming tools has been described. The specification draws attention to the accuracy of the equipment currently in the field. Significant advancements have been made in X-ray fluorescence measurement equipment but that technology costs on the order of $100k which not all PCB fabricators can justify owning. Older equipment may not bring the same level of accuracy.

For the gold layer, there has been a reduction of the minimum allowable thickness and a new maximum thickness set. The range will put barriers on some who drive gold deposits down to save money. Extremely low gold thickness contributes to performance failures as a thin deposit may not last through thermal conditioning and environmental exposure, remembering this was two of the desired advantages offered by the ENIG finish. The upper limits help to combat the potential nickel corrosion caused with excessive galvanic displacement required to drive the deposition mechanism, which will be discussed further in this paper.

Today's specification contains a section dedicated to measuring the phosphorous content of the electroless nickel deposit. Historically, people referenced ranges of nickel phosphorous (%P) in terms of low, medium and high but there was no industry standard to test for this level or control it through bath life. The 4552 revision introduces a new measurement system for phosphorous content and process control requirements that the PCB fabricator is required to demonstrate.

Lastly, there is a clear explanation for evaluating an ENIG deposit for corrosion. It contains a suggested evaluation method and levels of acceptability. The IPC committee has executed detailed studies to create this new document which add clarity to a grey area that was absent in the previous 4552 specification. What has not been done is an industry analysis to determine how challenging this may be to maintain in production environments globally with existing equipment and chemical processes.

Understanding nickel corrosion

There are two types of nickel corrosion to consider when discussing an ENIG finish. With aggressive environments and mobility of electronics today, most think first about the finishes resistance to the environment. Due to its nobility, gold is not a metal that tarnishes. Yet, there is a possibility that with a thin gold deposit or poor coverage of the underlying copper, ENIG can suffer corrosion. Again, this can occur either through pores in the thin gold deposit resulting in nickel oxidation/passivation or corrosion of underlying copper which was not properly covered in the electroless nickel plating process. The focus of the 4552 revision with respect to corrosion and the focus of this paper is the electroless nickel layer's resistance to corrosion

caused by a poorly controlled electroless nickel plating process or excessive galvanic attack of the electroless nickel deposit during the immersion gold stage. To better understand nickel corrosion as a result of plating immersion gold, one must understand the chemical mechanism of how the gold is deposited.

Anodic Reaction

$Ni \rightarrow Ni^{2+} + 2e^-$

Cathodic Reaction

$2[Au(CN)_2]^- \rightarrow 2Au^+ + 4CN^-$

$2Au^+ + 2\,e^- \rightarrow 2\,Au$

Overall Reaction

$Ni + 2[Au(CN)_2]^- \rightarrow 2Au + [Ni(CN)_4]^{2}$

Gold is deposited onto a nickel surface through an exchange reaction also called an immersion or galvanic displacement reaction [5]. Galvanic displacement reactions are driven by electron transfer and governed by the electromotive series, which dictates that more noble metals will naturally plate on those that are less noble without the use of chemical reducing agents to facilitate the reaction.

To deposit gold onto the electroless nickel surfaces, electrons must be transferred from the metallic electroless nickel deposit to the gold ions in solution producing a metallic gold deposit. This electron transfer results in removal of metallic nickel (corrosion) from the surface of the electroless nickel deposit and builds up nickel ions in the immersion gold solution as the reaction proceeds. Traditional immersion gold systems can allow uncontrolled dissolution of nickel metal and result in localized undesired spikes of corrosion which can be viewed in top down microscopy or through cross section.

Due to the nature of the gold plating mechanism, there will always be some level of corrosion as nickel must be removed from the electroless nickel deposit surface to facilitate the gold metal deposition. It is the level as seen both in amount over the surface and depth of that corrosion that is the concern.

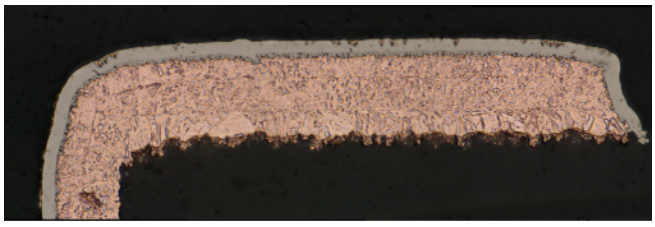
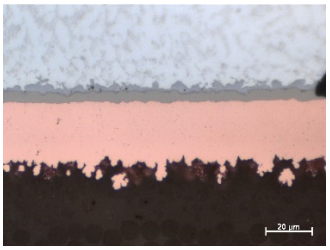


Figure 3: Example of ENIG Corrosion by X-Section.

The amount of the phosphorous in the electroless nickel layer will dictate the speed of gold deposition. Interaction between electroless nickel phosphorous content and absorbed organic stabilizer will determine the degree of corrosion that the gold imparts on the electroless nickel deposit surface. Generally speaking higher phosphorous contents and lower absorbed organics produce a more corrosion resistant electroless nickel deposit.

Excessive corrosion can result in poor IMC formation and weaker solder joint formation which in turn can cause components to fall from the Printed Circuit Board Assembly (PCBA) surface [6].



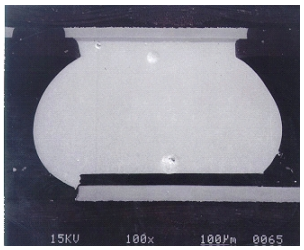


Figure 4a and 4b: Examples of poor IMC Formations and Solderjoint Failure

Deposit Thickness Measurement Capability

IPC4552 introduces XRF measurement capability, in the form of a type 1 gage study, to ensure PCB fabricators are using a reliable tool to control ENIG deposit thickness. It is recommended that conforming XRF equipment shall display gage capability (Cg) ≥ 1.33. There is no mention of Cgk in the document, which takes into account any measurement bias, therefore demonstrating measurement repeatability is the only requirement for XRF measurement. Repeatability is the ability to consistently make the same measurement on the same part, using the same gage, under the same conditions. Some measurement system variation will always be present even in a capable gage, but if the variation from measurement is too large in relation to the specification tolerance, the space available for process variation (between the upper and lower specification limits) will be reduced and create more strain maintaining a capable process [7].

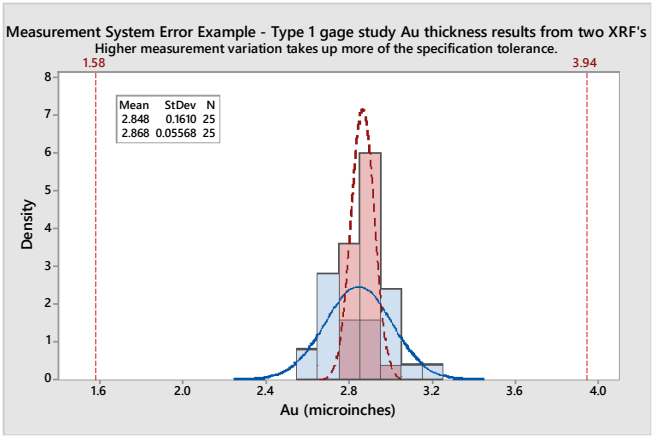


Figure 5: Histogram analysis comparing measurement system error from two different XRF's.

The Cg/Cgk metrics can be calculated by executing a type 1 gage study of the XRF tool to be used for ENIG thickness measurements as described in Section 3.1.1.2 of the new revision [4].

An experiment was executed to quantify the effect of XRF count time on gage capability. It was found that reducing the count times reduced repeatability and the bias. Extending

the count time from 30 to 60 seconds resulted in an increased Cg from 1.12 to 1.92 and Cgk 0.89 to 1.90 respectively.

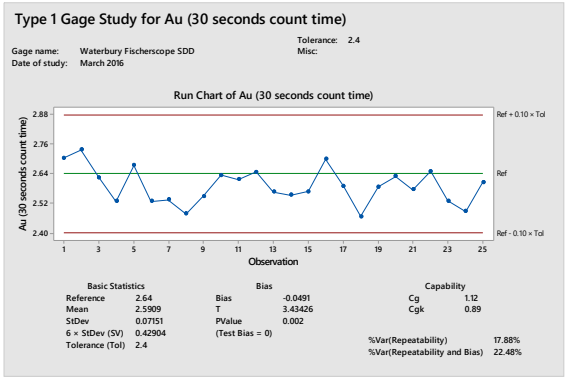


Figure 6: Type 1 XRF gage study for gold thickness (30 secs count time).

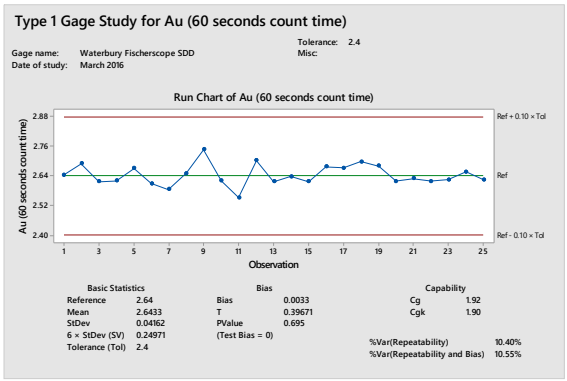


Figure 7: Type 1 XRF gage study for gold thickness (60 secs count time).

For non-conforming XRF equipment IPC4552A will allow two options….

1. Increase number of measurements by $[2 / Cg]^2$.

The Cg achieved from the XRF gage study can be used to calculate the number of measurements required as follows.

- 30 seconds count time - $[2 / 1.12]^2 = 4$ measurements
- 60 seconds count time - $[2 / 1.92]^2 = 2$ measurements

As gage capability reduces the amount of measurements required will increase as more measurements are needed to ensure that the average thickness calculated is close to the true value. Having operators taking many measurements may not be workable in a production environment, Therefore IPC4552A provides a second option.

2. The use of 3 sigma guard bands.

Using the standard deviation from the gold thickness measurements obtained from the XRF gage study, IPC4552A prescribes that three sigma guard bands should be introduced to create a new tighter working specification to reduce the effect of measurements system error creating out of specification thickness occurrence.

Thus the use of 3-sigma guard bands will reduce the specification tolerance and reduce the "room" for process variation in metal thickness observed from a production ENIG process.

Gold thickness data was evaluated from a production ENIG process according to IPC4552A and found to show 0.0126% of expected measurements to be outside of the new thickness specification. However, applying the 3-sigma guard band rule (Cg = 1.12, Std Dev = 0.07151) it was determined that the expected out of specification measurements rose to 0.31 %. This experiment does not take into account that there will be a widening of the gold thickness distribution using an XRF with Cg – 1.12, and in reality it can be expected that more measurements will fall outside of the gold thickness specification.

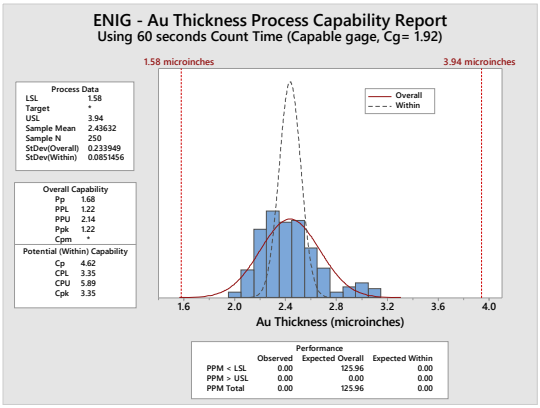


Figure 8: Gold Thickness data from production process measured using capable XRF equipment (Cg = 1.92).

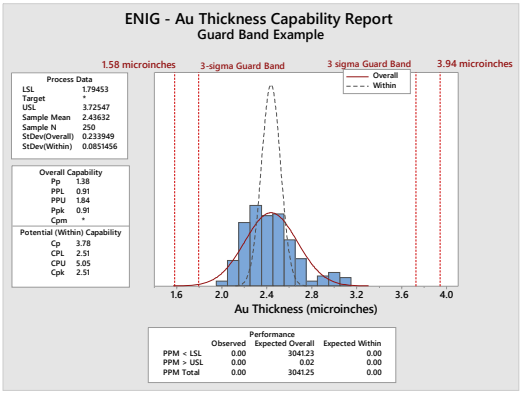


Figure 9: Gold Thickness data from production process measured using incapable XRF equipment (Cg = 1.12).

Having capable XRF equipment is considered key to minimizing the amount of measurements and maximizing the working specification for gold thickness. Without adequate deposit thickness capability there is increased pressure on the ENIG production process.

To prove instrument capability, a type 1 gage study was undertaken using a Fischerscope SDD XRF over the IPC

4552A gold thickness range. The minimum Cg calculated for this study was 1.50 (Cgk = 1.47) which meets the IPC4552A requirements. This enables accurate testing and execution of various PCB designs and changes to process formulations and conditions in respect to gold thickness.

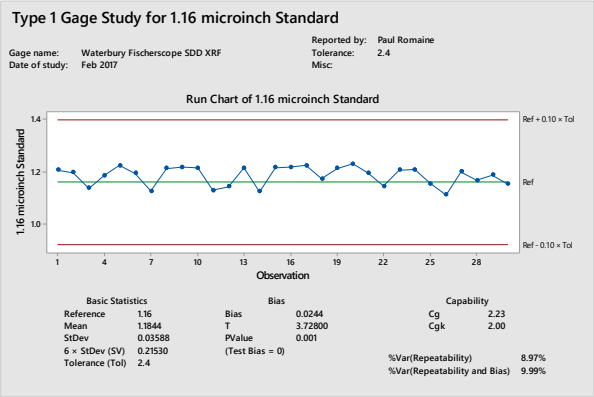


Figure 10: Type 1 gage study for 1.16 microinch gold standard**.**

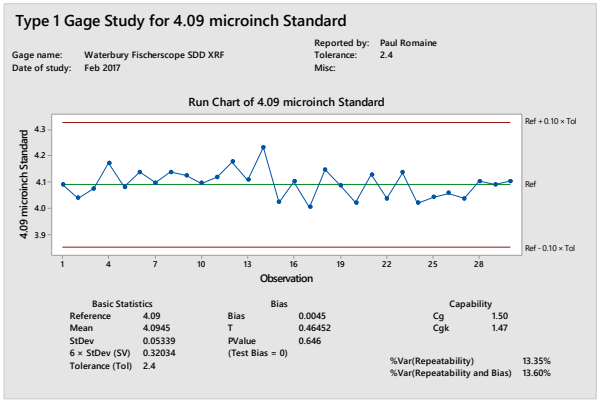


Figure 11: Type 1 gage study for 4.09 microinch gold standard.

New Gold Thickness Specification

IPC4552A includes a new minimum and introduces a maximum gold thickness for the first time. The average gold thickness measured should be three standard deviations above 1.58 microinches and three standard deviations below 3.94 microinhes [4].

Gold thickness should have average of $\tilde{\boldsymbol{x}}$ …

$\tilde{\boldsymbol{x}} - 3\ s \geq 1.58$ µinches,

and $\tilde{\boldsymbol{x}} + 3\ s \leq 3.94$ µinches

With the addition of a maximum specification limit and the statistical restraints needed based on thickness standard deviation, controlling gold thickness produces a larger challenge for PCB fabrication. This means measurement system variation and process variation must be low. As the ENIG process delivers lower standard deviation gold distribution, the average gold thickness can be reduced closer to the lower specification limit. This offers potential savings in gold consumption.

Variation from measurement systems, ENIG process chemistry selection and process control of the chosen chemistry will all contribute to the variation observed in gold thickness and the ability to meet the IPC 4552A.

Corrosion Evaluation

IPC 4552A section 3.6 discusses and tries to add some uniformity to the ongoing ENIG corrosion acceptability and reliability argument. The key points are as follows:

- Some occurrences of hyper-corrosion will always be found if enough samples are taken from a printed circuit board or if excessive magnification is used for evaluation. A single occurrence of hyper-corrosion is NOT rejectable.
- The defect associated with hyper-corrosion is non-wetting (although the gold is consumed) as defined by a lack of intermetallic compound (IMC) formation.
- Hyper-corrosion may be evident but if it does not interfere with the formation of a reliable solder joint as defined by continuous IMC formation, it is NOT considered to be rejectable.
- Severe hyper-corrosion whereby the soldering is impacted negatively is a rejectable condition. Inspection of hyper-corrosion for acceptance criteria shall use optical microscopy at a maximum of 1000x magnification.

Non-wetting soldering defects can occur for many reasons independent of the PCB final finish. Thus a consistent and low level of EN corrosion is advisable to avoid any solderability failure being diagnosed as “hyper-corrosion” and thus rejected.

The IPC document includes levels of ENIG corrosion rating and an acceptability assessment flow chart for a more defined observation and identification of corrosion. The chart gives a direct stepwise determination of corrosion and acceptability for release or submission for failure analysis.

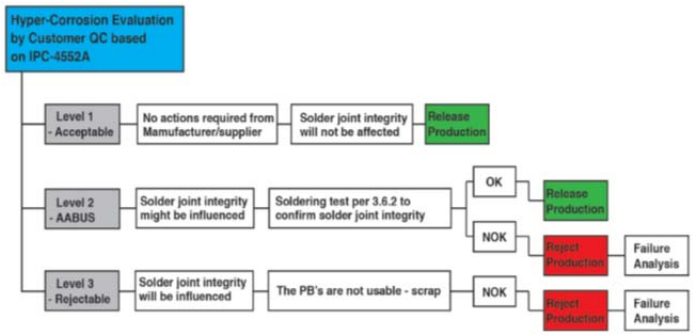


Figure 12: IPC Corrosion Acceptability Evaluation Decision Tree**.**

Table 3-4 in the IPC 4552 A diagnoses various degrees of hyper-corrosion based on frequency and depth of the corrosion spikes within a cross sectional area, and the document also contains visual guidelines of the three levels defined.

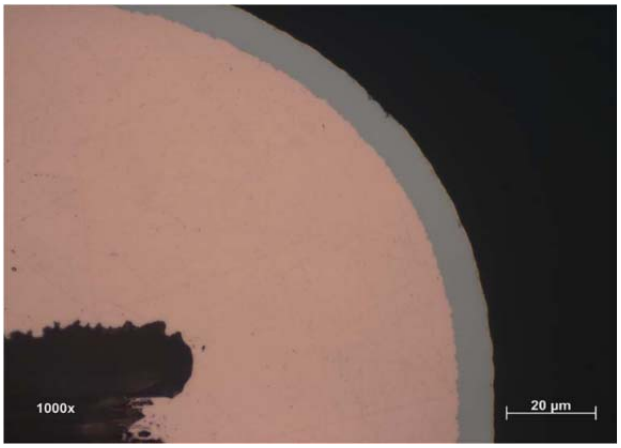


Figure 13: Example of Level 1 Hyper Corrosion (Preferred).

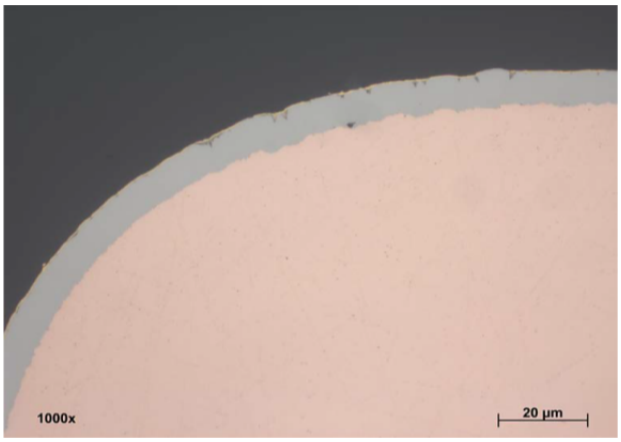


Figure 14: Example of Level 2 Hype Corrosion (AABUS).

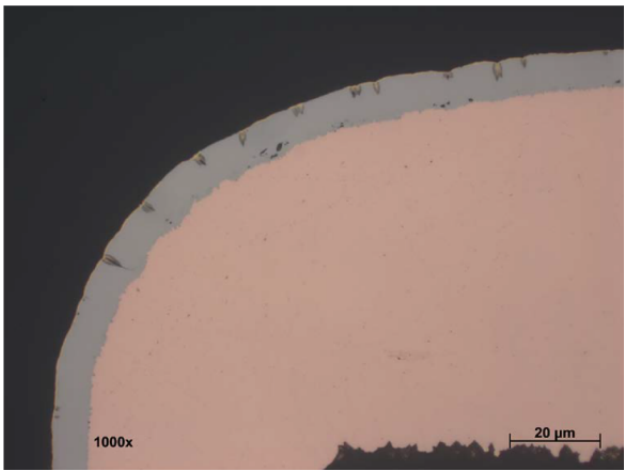


Figure 15: Example of Level 3 Hype Corrosion (Rejectable).

Electroless Nickel Phosphorous Content

The final change to the IPC 4552 document which is an addition over the previous version is Section 3.1.6 Measuring Phosphorous content in an ENIG deposit. Revision A casts doubt over the traditional use of Energy-dispersive x-ray spectroscopy (EDS) as a suitable method of measuring phosphorous but suggests chemical stripping of the EN deposit and analysis by Inductively Coupled Plasma (ICP) or Atomic Absorption Spectroscopy (AAS) of the resultant stripping solution. In this section, an XRF method is also introduced for analysis for the EN's phosphorous deposit as plated but the equipment must utilize a Silicon Drift Detector to be capable of this analysis. It is stated that the phosphorous content of a plated sample shall be checked quarterly at a minimum and should cover the working metal turnover operating range for the electroless nickel chemistry. It should be noted that for many electroless nickel formulations, the co-deposited phosphorous content can change a few percent within a full bath life.

CASE STUDY

Electroless Nickel/Immersion Gold

To better understand the capabilities and quality of present day equipment and processes, a case study was run with a PCB manufacturer in North America. The first step was to assess their thickness measurement capability for gold thickness. A Type 1 gage study was undertaken using a 5 second reading time which was the standard procedure for this facility. A 60 second count time was also used as the recommended time for comparison. Increasing the count time from 5 to 60 seconds significantly improved the repeatability of the readings from a Cg of 0.5 to 1.44. By removing some of the measurement system error, the variation in gold deposit thickness is reduced. Despite demonstrating improved gold thickness measurement repeatability, there was still considerable bias observed from the XRF unit.

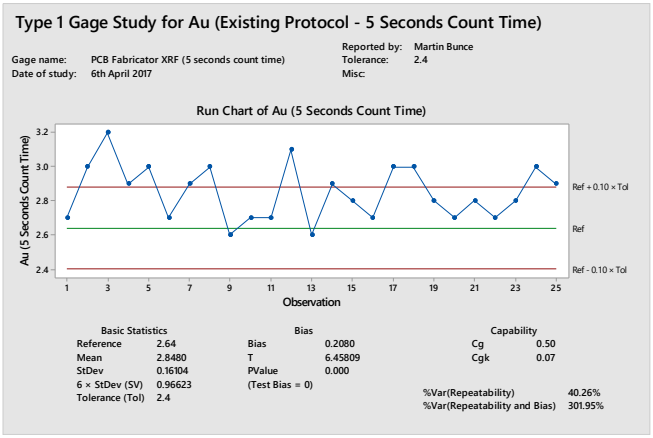


Figure 16: Type 1 gage study with 5 seconds XRF count time.

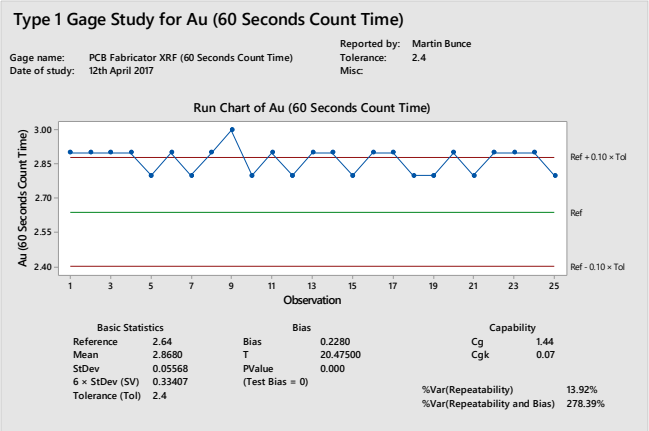


Figure 17: Type 1 gage study with 60 seconds XRF count time.

The next step was a full ENIG deposit performance evaluation including gold thickness distribution, electroless nickel phosphorous content and solderability performance with the existing production process.

Gold thickness distribution

- XRF readings on each test coupon feature size at various panel locations
- Total of 100 XRF readings per test panel
- Thickness analyzed as per the IPC 4552 requirements (consistent pad size)
- Pad-Pad thickness analysis (within panel distribution)

Corrosion Analysis

- Cross section of 10 metal defined BGA pads
- Cross section through a row of 10 PTHs
- Each BGA and PTH rated as per the IPC 4552A
- The % corrosion observations recorded for each analysis.
- Cyanide stripping of gold from 10 metal defined BGA pads
- Cyanide stripping of gold from 10 PTHs
- Each BGA and PTH evaluated with SEM at 300x magnification

For this case study, two test panels were processed through the customer's existing ENIG process one at 0.72 electroless nickel MTOs and 2.2 MTOs. Based on this "snapshot" of the process, it is expected that 6.97% of the gold thickness measurements from this process would be outside of the IPC4552A specification (consistent pad size for measurement). When looking at the process from the thickness capability based on varying pad sizes on a particular test coupon it was determined that 12.37% of the gold thickness measurements are expected to be outside of the IPC 4552A specification.

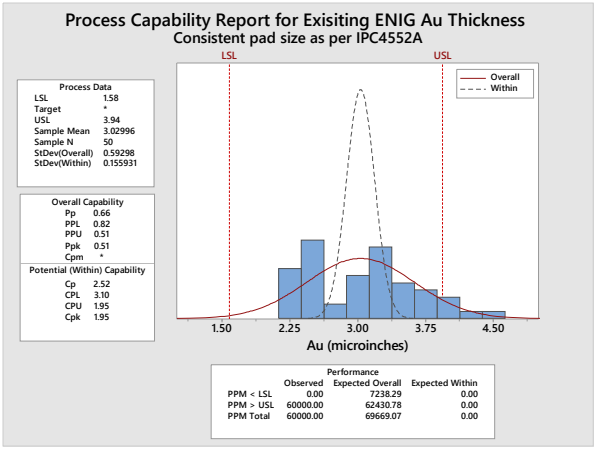


Figure 18: Existing ENIG Gold Thickness Capability as per IPC4552A.

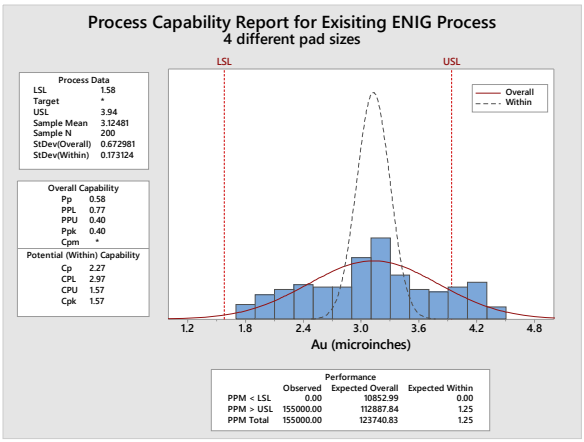


Figure 19: Existing ENIG Gold Thickness Capability on various pad sizes.

The majority of the cross section corrosion evaluations from SMT pads was spilt approximately 50/50 between acceptable and ABBUS. However, there was an increased in the level 2 type corrosion observed when PTH areas were evaluated.

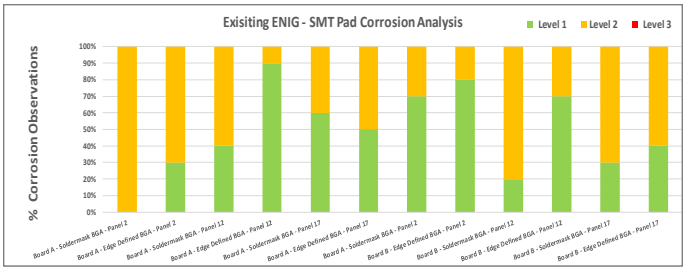


Figure 20: Existing ENIG Corrosion Evaluation on SMT features.

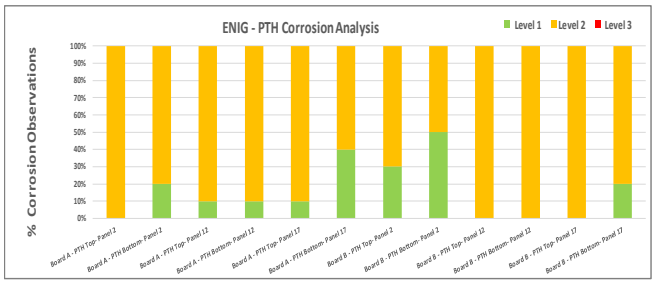


Figure 21: Existing ENIG Corrosion Evaluation on PTH features.

Figure 22: Typical ENIG Corrosion Evaluation on PTH feature.

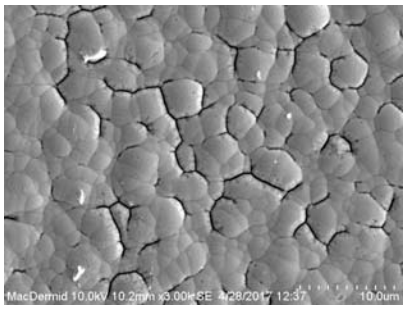


Figure 23: Typical ENIG Corrosion Evaluation on PTH feature.

INTRODUCING A NEW CHEMICAL SET

Reduced Variation

In parallel to the creation of IPC 4552 Revision A, a new ENIG chemical process was being developed as a result of a larger pool of technical knowledge within the MacDermid Enthone organization. Combining this expertise with a six sigma approach to development, an ENIG technology with significantly improved conformance to IPC4552A has been delivered.

Many traditional electroless nickel systems allow phosphorus content to drift over the life of the chemical plating bath. As byproducts from the electroless nickel deposition reaction build up in the process chemistry a reduction in plating speed is observed. It is not the desire of the PCB fabricator to have a changing plating rate as this can effect throughput and often plating times in each bath are dictated by a fixed time way. Common methods used to maintain productivity and compensate for reduced plating speed are to increase temperature or make chemical pH adjustment of the electroless nickel chemistry. The disadvantage of manipulating plating rate is its effect on resultant phosphorus content. Some methods deliver less of a change to the phosphorous than others (figure 24), your chemical supplier should be consulted. As plating rate changes, the deposited phosphorous content shifts.

As mentioned in the Introduction of this paper, the level of phosphorous in the deposit dictates the corrosion resistance level of the deposited EN. This is true for environmental corrosion resistance as well as corrosion resistance to the gold plating bath. The higher the phosphorous content of the electroless nickel deposit, the greater the resistance. Care needs to be taken when approaching levels above 12 percent as the ability to plate an adherent gold and maintain solderability becomes more challenging, not impossible but more challenging.

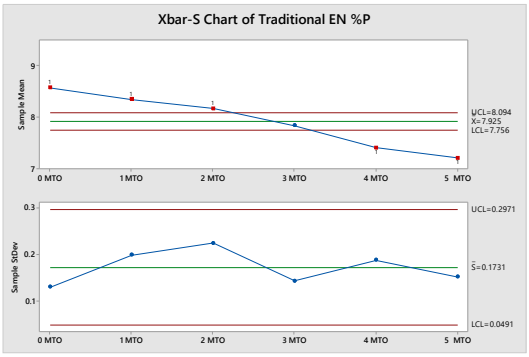


Figure 24: Variable EN %P with chemistry age due to pH manipulation.

Resultantly, when using a traditional electroless nickel technology, the Immersion Gold galvanic displacement reaction needs to manage variability in the electroless nickel corrosion resistance as it is changing over time. It many cases, this can per a few percentages. This can lead to difficultly in achieving good control of gold deposit thickness and potentially limit the PCB manufactures ability to consistently meet IPC4552 Revision A.

A new approach to electroless nickel formulation and process control provides consistent deposited phosphorous content over the life of the chemistry with a narrow standard deviation. By delivering a consistent phosphorous level over a board, board to board, and through a nickel bath life, the gold deposit is inherently more uniform. The Immersion Gold galvanic displacement mechanism plates on a surface with consistent corrosion resistance. Figure 25 shows the very narrow range of deposited phosphorous content over the new electroless nickel bath life for five metal turn overs. This data holds true for a board with various pads sizes and board to board. In turn, this yields a significant increase in ability to maintain gold deposit thickness over the life of the electrodes nickel chemical life. As with any chemical processes, attention should be paid to process control parameters to deliver the greatest performance and resultant uniformity of the system for each chemical bath.

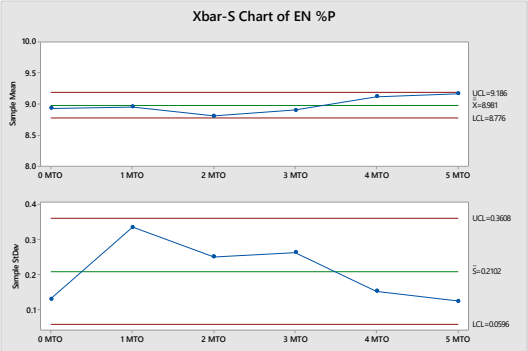


Figure 25: X-Bar S chart showing stable EN %P with chemistry age.

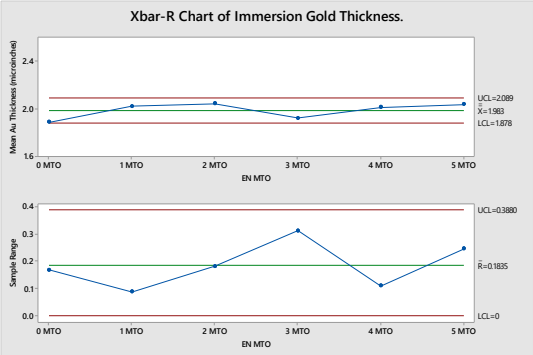


Figure 26: X-Bar S chart showing stable Au thickness with Electroless Nickel chemistry age.

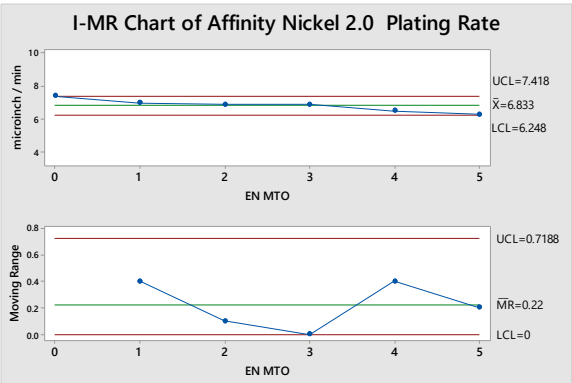


Figure 27: Stable EN plating rate with chemistry age

The ability to create a more corrosion resistant electroless nickel and a process controlled gold plating can be observed by the improved gold thickness distribution (figure 30) on varying pad sizes as well as a reduction in observable corrosion by SEM and cross section analysis (figures 28 and 29).

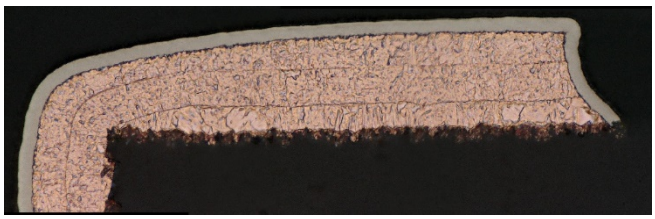


Figure 28: Typical ENIG Corrosion Evaluation on PTH feature from newly developed ENIG system.

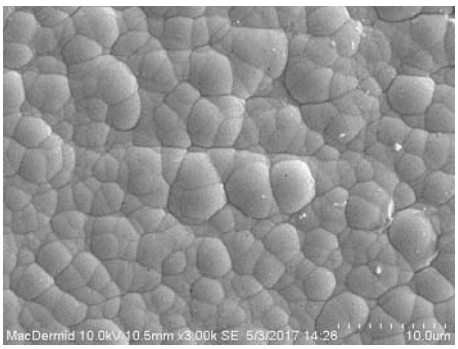


Figure 29: Typical ENIG Corrosion Evaluation on PTH feature from newly developed ENIG system.

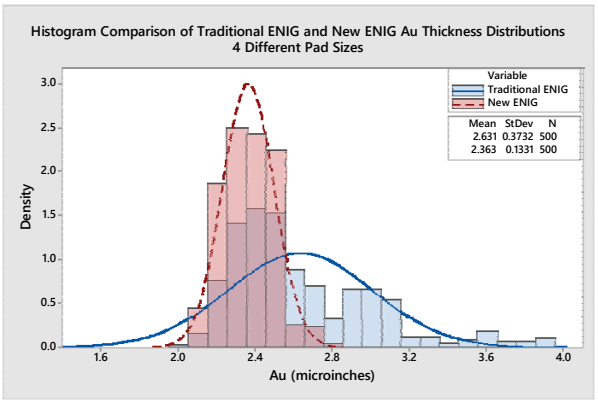


Figure 30: Gold thickness distribution of traditional ENIG and New ENIG using 4 different pad sizes.

CONCLUSIONS

IPC4552 Revision A will raise the bar for ENIG quality in terms of corrosion, metal deposit composition and thickness control. Some traditional ENIG systems may be challenged to consistently produce the required quality at the PCB fabrication level. Close attention should be paid to the chemical process conditions and the equipment being used to for analysis.

To bring more ease and consistency to the ENIG process within PCB fabrication and ensure compliance to the IPC 4552 Revision A, a new chemical ENIG process has been developed. It brings significant improvements in deposit composition uniformity and ability to control critical to quality characteristics required of conforming ENIG product.

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