Specification for
Electroless Nickel / Electroless Palladium / Immersion Gold
(ENEPIG) Plating for Printed Circuit Boards

Plating Subcommittee (4-14)
1 SCOPE

1.1 Statement of Scope
This specification sets the requirements for the use of Electroless Nickel/Electroless Palladium/Immersion Gold (ENEPIG) as a surface finish for printed boards. This specification sets requirements for ENEPIG deposit thicknesses for applications including solderability, wire bonding and as a contact finish. It is intended for use by suppliers, printed board manufacturers, electronics manufacturing services (EMS) and original equipment manufacturers (OEM).

1.2 Description
ENEPIG is a surface finish consisting of an electroless nickel layer plated with an electroless palladium layer which is then capped with a thin layer of immersion gold (for deposition process details, see APPENDIX 1). It is a multifunctional surface finish, applicable to soldering and to gold, aluminum and copper wire bonding. In addition, it is also suitable as the mating surface for soft membrane and steel dome contacts, Low Insertion Force (LIF) and Zero Insertion Force (ZIF) edge connectors and for press-fit applications. The electroless palladium layer forms a diffusion barrier that impedes nickel diffusion to the gold surface. The immersion gold prevents the palladium layer from reacting with contaminants that might otherwise affect joining processes, such as wire bonding and soldering.

1.2.1 Electroless Nickel Reducing Agents - Phosphorus Content
Phosphorus containing reducing agents are typically used for the reduction of the electroless nickel during the deposition process and phosphorus is incorporated in the nickel deposit. The level of this co-deposited element should be controlled within the suppliers specified process limits. Variation of phosphorus levels, outside the specified process limits, may have adverse effects on the performance of the finish.

1.2.2 Electroless Palladium Reducing Agents
There are two distinct classes of reducing agents used in electroless palladium baths currently available for use in the ENEPIG process, those that produce deposits that contain a co-deposited element such as phosphorus, and those that produce an essentially pure palladium deposit. The level of the co-deposited elements should be controlled within the specified process limits. Examples of electroless palladium deposit from both these classes have been evaluated during the development of this specification and no perceivable differences in performance were observed in the tests of solderability or wire bondability.

1.3 Objective
This specification sets the requirements for ENEPIG as a surface finish (see Table 3-1 for a summary of these requirements). As additional surface finishes require specifications, they will be addressed by the IPC Plating Processes Subcommittee as part of the IPC-455X specification family. This and other surface finish specifications are under continuous review. The 4-14 subcommittee will make appropriate amendments or revisions to these documents as required. The 4-14 Plating Processes Subcommittee undertook a “Round Robin” study to generate data to support the recommendations sited for the various aspects of this specification. For an outline of the study, refer to APPENDIX 2.
1.3.1 Order of Precedence
In the event of conflict, the following order of precedence shall apply:
1. The purchase order. This includes exceptions to this specification as agreed to by both user and supplier.
2. The master drawing. This includes exceptions to this specification as agreed to by both user and supplier.
3. This specification.
4. Applicable documents as detailed in section 2 of this document.

1.3.1.1 Appendices
This specification contains ten Appendices that are provided for use and are shown after the main body of this document is fully shown. Be aware that all appendices to this specification are not binding requirements unless separately and specifically required by the purchase order, the master drawing or other applicable documents, as agreed to by both the user and supplier.

1.4 Performance Functions

1.4.1 Solderability
One of the two primary functions of ENEPIG is to provide a solderable surface finish capable of providing IPC Category 3 shelf life (minimum 12 months). This shelf life is suitable for all surface mount, hybrid and through-hole assembly applications. The use of electroless palladium as a diffusion barrier between the nickel and the gold, mitigates the potential for hyper-corrosion of the electroless nickel deposit, thus enhancing its solderability and solder joint reliability. The minimal thicknesses of the electroless palladium and of the immersion gold deposit make the possibility of embrittlement of the solder joint by palladium or gold negligible.

1.4.2 Wire Bonding
The second primary function of ENEPIG is to provide a wire bondable surface finish, suitable for Gold, Aluminum and Copper wire applications. ENEPIG has been found to meet the requirements of MIL-STD-883, Method 2011.7. The committee has performed extensive testing with gold wire (see APPENDIX 8) and has received additional anecdotal data from members confirming the suitability of this finish for aluminum and copper wire bonding. Variables that are likely to affect performance include cleanliness, substrate materials, wire thickness and substrate copper surface morphology, as ENEPIG deposits are not capable of completely levelling the substrate surface. For this specification, surfaces with Ra values up to 389 nm were tested with no negative impact on gold wire bond strength.

1.4.3 Contact Surface
There is a substantial amount of experience using ENEPIG for the applications listed below in 1.4.3.1 and 1.4.3.2.

1.4.3.1 Membrane Switches
ENEPIG is a de facto standard for soft touch membrane switches.
ENEPIG plated samples tested for 2 million contact actuations showed negligible change in contact resistance. For these actuations, the deposit thicknesses were nominally: 4.65 µm [183 µin] of electroless nickel (EN), 0.13 µm [5.2 µin] of electroless palladium (EP) and 0.053 µm [2.1 µin] of immersion gold (IG).

Note: This test was run to the full number of cycles with no visible impact on the ENEPIG deposit.
1.4.3.2 Metallic Dome Contacts
ENEPIG has been demonstrated to be suitable for use with metallic (stainless steel), circular dome contacts, based on testing to 1 million cycles. For this testing, the ENEPIG was deposited at nominal thicknesses of 5.38 µm (212 µin) of electroless nickel (EN), 0.081 µm (3.17 µin) of electroless palladium (EP) and 0.015 µm (0.58 µin) of immersion gold.

The committee continues to be interested in any additional test data using other metals and contact shapes for this particular application.

1.4.4 EMI Shielding
ENEPIG can be used as a surface finish at the interface between electromagnetic interference (EMI) shielding materials and printed boards (PBs).

1.4.5 Interface for Conductive and/or Anisotropic Adhesives
ENEPIG is suitable for use as an interface for conductive adhesives used as an alternative to solder. It is also suitable for anisotropic adhesive applications.

1.4.6 Connectors
1.4.6.1 Press-Fit Applications
Press-fit performance should meet Telcordia GR-1217-CORE. Excessive electroless nickel thicknesses can lead to concerns with excessive press-fit insertion forces. The additional thickness of the palladium specified either by this specification and/or a customer-specific requirement should be added to the electroless nickel thickness when considering the type of application.

1.4.6.2 Edge Tab Contact Applications
The ENEPIG surface finish is suitable for plug-to-install applications (twenty insertions/withdrawals or less) using low insertion force (LIF) or zero insertion force (ZIF) connectors. ENEPIG is not believed to be suitable as a surface finish for edge connectors that require multiple insertion/withdrawals (more than twenty) or high insertion force applications. This application generally requires an alternate metallic finish, such as electrolytic hard gold.

1.4.7 Limitations of ENEPIG
ENEPIG, similar to the other alternate surface finishes addressed by the IPC-455X series specifications remain susceptible to creep corrosion in high sulfur, high temperature and humidity atmospheres. It is recommended to test specific design/applications if the end use environment is considered harsh.
2 APPLICABLE DOCUMENTS

2.1 IPC
J-STD-003 Solderability Tests for Printed Boards
IPC-2221 Generic Standard on Printed Board Design
IPC-6011 Generic Performance Specification for Printed Boards
IPC-6012 Qualification and Performance Specification for Rigid Printed Boards
IPC-6013 Qualification and Performance Specification for Flexible Printed Boards
IPC 5704 Cleanliness Requirements for Unpopulated Printed Boards
IPC-TM-650 Test Methods
  2.3.25 Detection and Measurement of Ionizable Surface Contaminants by Resistivity of Solvent Extract (ROSE)
  2.4.1 Adhesion, Tape Testing
  2.6.3.5 Bare Board Cleanliness by Surface Insulation Resistance
  2.6.14.1 Electrochemical Migration Resistance Test

2.2 American Society for Testing and Materials (ASTM International)
ASTM B733, Standard Specification for Autocatalytic (Electroless) Nickel-Phosphorus Coatings on Metal
ASTM B 568, Standard Test Method for Measurement of Coating Thickness by X-Ray Spectrometry

2.3 JEDEC
JESD 213, Common Test Method for Detecting Component Surface Finish Materials (Made for what you are doing in lab for pure tin and referenced in GEIA)

2.4 Defense Standardization Program
MIL-STD-883, Method 2011.7 Test Method Standard, Microcircuits – Bond Strength (Destructive Bond Pull Test)
MIL-STD-1580, Department of Defense Test Method Standard (Destructive Physical Analysis for Electronic, Electromagnetic, and Electromechanical Parts)

2.5 Telcordia Technologies, Inc.
Telcordia GR-1217-CORE Generic Requirements For Separable Electrical Connectors Used In Telecommunications Hardware

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1. www.ipc.org
2. Current and revised IPC Test Methods are available on the IPC Web site (www.ipc.org/html/testmethods.htm)
3. www.astm.org
4. www.jedec.org
5. www.dsp.dla.mil
6. www.telcordia.com
2.6 International Organization for Standardization (ISO)\textsuperscript{7}


\textsuperscript{7} www.iso.org
3 REQUIREMENTS

A summary of requirements are provided in Table 3-1 Requirements for Electroless Nickel Electroless Palladium Immersion Gold Plating at the end of section 3.

3.1 Visual
ENEPIG surfaces shall be inspected in accordance with the visual examination sections of the IPC-6011 series specifications, specifically IPC-6012 which specifies the use of a nominal magnification of 1.75X (approx. 3 diopters). The coverage shall be complete and the finish shall be uniform on the surfaces to be plated (see Figure 3-1). For higher magnification analysis, Figures 3-2 to 3-4 are offered for reference purposes. There shall be no extraneous plating or nickel foot (see Figure 3-2), edge pull back (see Figure 3-3) or skip plating (see Figures 3-4, 3-5 and 3-6) on the surfaces of all classes of product.

![Figure 3-1 Uniform Plating](image)

Figure 3-1 Uniform Plating
Figure 3-2  Extraneous Plating or Nickel Foot

Figure 3-3  Edge Pull Back
Figure 3-4  Skip Plating

Figure 3-5  Skip Plating of Gold Over Palladium
3.1.1 High Magnification Reference Images
It may be necessary to evaluate the ENEPIG deposit using scanning electron microscopy (SEM), transmission electron microscopy (TEM) or focused ion beam (FIB) as part of a qualification plan and or for failure analysis. The images in the following section are for reference purposes and were supplied by several different suppliers of ENEPIG.
Figure 3-7  1000X SEM Image of a Normal ENEPIG Surface

Figure 3-8  4000X SEM Image of a Normal ENEPIG Surface
Figure 3-9  2500X FIB Image of a Normal ENEPIG Deposit

Figure 3-10  100,000X FIB Image of a Normal ENEPIG Deposit
The Phos-Palladium film is amorphous and very uniform whereas the gold (Au) deposit is crystalline in nature.

This is due to excessive dwell time in the gold bath.
3.2 Finish Thickness
Thickenesses of the electroless nickel, electroless palladium and immersion gold layers **shall** be measured and verified following the completion of the ENEPIG plating operation in the printed board fabrication process. The use of X-ray fluorescence (XRF) methodology for thickness determination **shall** be in accordance with APPENDIX 4 of this document and shall employ XRF instrumentation equipped with software and hardware appropriate for ENEPIG measurement. The committee has performed extensive round robin testing of equipment capability to accurately measure this tertiary deposit (see APPENDIX 3). The results showed a number of critical issues with equipment, set-up, measuring protocols and reference standards. It is imperative to demonstrate measurement capability in order to meet the following thickness specification.

3.2.1 Electroless Nickel Thickness
The electroless nickel thickness **shall** be 3 to 6 µm [118.1 to 236.2 µin] at ± 4 sigma (standard deviations) from the mean as measured on a nominal pad size of 1.5 mm x 1.5 mm [0.060 in x 0.060 in] or equivalent area, where standard feature size tolerances, as expressed in the IPC-6010 standard series, apply. This upper end of this thickness specification has been chosen based on concerns with insertion force issues for compliant pin applications ONLY. Use of thicknesses outside of this range **shall** be AABUS.

3.2.2 Electroless Palladium Thickness
The electroless palladium thickness **shall** be 0.05 to 0.15 µm [2 to 6 µin] at ± 4 sigma (standard deviations) from the mean as measured on a nominal pad size of 1.5 mm x 1.5 mm [0.060 in x 0.060 in] or equivalent area, where standard feature size tolerances as expressed in the IPC-6010 standard series, apply. The upper thickness limit may be exceeded to meet special design criteria (for example, wire bonding and no soldering). However, the committee has data showing that excessive electroless palladium thicknesses may impair solder joint reliability.

3.2.3 Immersion Gold Thickness
The minimum immersion gold thickness **shall** be 0.025µm [1.2 µin] at - 4 sigma (standard deviations) below the mean as measured on a nominal pad size of 1.5 mm x 1.5 mm [0.060 in x 0.060 in] or equivalent area, where standard feature size tolerances as expressed in the IPC-6010 standard series, apply. The specification is one sided, based on performance test results showing impaired solderability performance after accelerated aging at lower gold thicknesses. The nature of the reaction of immersion gold with electroless palladium is such that substantially greater thicknesses may not be achievable.

NOTE: Some designs may not have feature sizes of this specified pad area and will require the use of alternate pad sizes. The XRF collimator used **shall** always be smaller than the pad used for measurement; and specifically, the collimator **shall not** exceed 30% of the feature pad size being measured. For smaller sized pads, the measurement time will need to increase proportionately as a function of collimator area reduction. Consistency of the pad size used for measurement is of primary concern in order to obtain a statistically valid result, due to the typical plating thickness variation seen as a function of pad size. In general, smaller features will plate thicker than larger features. The use of fiducial pads for this measurement is suggested for those designs not having the specified pad feature areas, described above.
3.2.4 XRF Calibration Standards
The use of National Standards traceable calibration standards with similar thicknesses to the specified ENEPIG thicknesses which are to be measured, is recommended. Tri-layer standards where Au and Pd are plated directly on Ni/Cu/PCB should be used for calibration for Cu thicknesses > 30µm. Tri-layer foil standards where Au and Pd are plated on a Ni-foil should be used if boards with varying Cu-thicknesses are to be measured. Additional information can be found in Appendix 10.

For Cu thickness > 30 µm, a combination of a minimum of two calibration standards with approximate thicknesses, as below, should be used.

- Au/Pd/Ni/Base 50nm/20nm/3µm
- Au/Pd/Ni/Base 50nm/90nm/3µm
- Au/Pd/Ni/Base 50nm/300nm/3µm
- Au/Pd/Ni/Base 10nm/20nm/3µm

For Cu thicknesses < 30 um a combination of a minimum of two calibration foil standards with approximate thicknesses as below, should be used

- Au/Pd/Ni/Base 60nm/20nm/4µm
- Au/Pd/Ni/Base 60nm/60nm/4µm
- Au/Pd/Ni/Base 50nm/100nm/4µm
- Au/Pd/Ni/Base 50nm/250nm/4µm

For additional details, see APPENDIX 9.

3.2.5 Use of Non Statistical Thickness Specification
For production lot sizes of ten printed boards or less, i.e. prototype builds, the need to demonstrate compliance to the statistical requirements above are negated, provided that measurements are taken on all printed boards/panel coupons, thus providing population parameters rather than sample statistics. All such readings shall fall within the thickness ranges specified above for all three deposits. Any readings below or above the specified thickness range shall be cause for rejection of that board.

3.2.6 Production Overruns and/or Inventory Items
When compliance testing of individual printed board(s) remaining in inventory is required, use the protocol of 3.2.4. For production overruns larger than ten, the sample statistic constraints shall apply.

3.3 Porosity
An ENEPIG immersion gold layer is not totally impervious and is not able to pass the requirements of a ‘classic’ porosity test (for example, Nitric Acid per ASTM B 735).

3.4 Adhesion
The purpose of adhesion testing for ENEPIG finished boards is two-fold.

1) The primary purpose of the testing is to verify that there is no adhesion failure between the metallic layers of the finish, or the finish and the base metal. The tape testing shall be in carried out in accordance with IPC-TM-650, Test Method 2.4.1, using a strip of pressure sensitive tape. There shall be no evidence of any portion of the surface finish being removed, as shown by particles of plated deposit/pattern adhering to the tape. If overhanging metal (slivers) or soldermask undercut breaks off and adheres to the tape, it is evidence of overhang or slivers, but not of adhesion failures.
2) The secondary purpose is for testing the adhesion of the solder mask to both traces and laminate. Both the chemistry used in the ENEPIG process and the required dwell times and temperatures can have a negative impact on solder mask adhesion – Note: Not all solder masks are compatible with an ENEPIG process. In both cases, testing should be conducted on regions of high feature density on the board, such as BGA sites, dams between fine pitch leaded devices or over areas of high trace density. Using the same approach as for the test for metal adhesion, check for evidence of the soldermask being removed, as shown by particles of soldermask adhering to the tape.

Because of the requirement to test in high density areas, the potential to leave tape residue that could interfere with soldering exists, especially if an incorrect type of tape is used. Verification of zero impact to solderability must be demonstrated for the areas tested, or the area tested shall not be used for final product.

3.5 Solderability

The thickness specification described herein shall meet the Category 3 coating durability requirements of J-STD-003, i.e. greater than 12 months shelf life.

NOTE 1: The use of steam exposure (conditioning) is not an applicable accelerated stress test method for ENEPIG.

NOTE 2: While adherence to the IPC 1601 recommendations for baking prior to assembly may have a negative impact on solderability performance, the requirements for solderability testing in this specification are for samples tested PRE-BAKE. Any deviation from this shall be AABUS.

For details of the solder spread testing data from the round robin, see APPENDIX 6. Ball shear data from the same study is documented in APPENDIX 7.

3.5.1 Force Measurement Testing (Wetting Balance Testing)

The following requirements are based on the use of the Standard IPC coupon, Figure 3-13 below, immersed to a depth of 0.6mm at 90 degrees incidence to the solder surface. The dwell time shall be ten seconds. For details of the wetting balance data from the round robin study, see APPENDIX 5.

- Force measurement testing of an “as received” sample with Eutectic SnPb solder, using the prescribed Test Flux # 1 at 235°C, shall produce a positive wetting force within 2 seconds and a minimum wetting force of 0.14 mN/mm.
- For force measurement testing of samples stressed for 8 hours @ 72°C [162°F] and 85% R.H, the increase in wetting time shall not exceed 1 second. The wetting force shall be a minimum of 0.12 mN/mm.
- Force measurement testing of “as received” samples with SAC305 solder, using the prescribed Test Flux # 2 at 255°C, shall produce a positive wetting force within 1.5 seconds and a minimum wetting force of 0.19 mN/mm.
- For force measurement testing of samples stressed for 8 hours @ 72°C [162°F] and 85% R.H., the increase in wetting time shall not exceed 1 second. The wetting force shall be a minimum of 0.17mN/mm.
3.6 Cleanliness
The surface of an ENEPIG finish is typically cleaner than most other surface finishes. Ionic cleanliness **shall** be established per the requirements of IPC-5704, which specifies a sample size of 11. It is recommended that SEC testing per IPC-TM-650 method 2.3.25 also be performed at the same time as the IPC-5704 qualification, to establish process control parameters. SIR testing **shall** be per IPC-TM-650 Method 2.6.3.5. The requirements for SIR test performance **shall** be as specified in Table 3-1. Failure to meet this cleanliness specification should trigger immediate process control corrective action.

3.7 Electrolytic Corrosion
The ENEPIG process contains chemistries that are ionic in nature, but which are readily rinseable. Both the quality and volume of water used for the rinsing steps between process baths, and for the final rinsing of the product are critical if the potential for electrolytic corrosion is to be avoided. The use of IPC-TM-650, TM 2.6.14.1 “Electrochemical Migration Resistance Test” is recommended for high reliability applications, or for designs with high feature density. The resistance values **shall not** drop more than 1 decade after the test has been completed.

3.8 Chemical Resistance – Creep Corrosion Resistance
Extensive testing of ENEPIG for creep corrosion resistance has been reported. This surface finish is not suitable for applications in corrosive atmospheres with high contents (ppm levels) of sulfur compounds or chlorine.

3.9 High Frequency Signal Loss
Higher frequency (>3 GHz) applications may experience signal loss on full build (i.e. where ENEPIG is applied over both traces and pads) designs. Signal loss is minimal when only the pads (but not the traces) are plated with the finish.
<table>
<thead>
<tr>
<th>Inspection</th>
<th>Class/Test Frequency (A.Q.L.)</th>
<th>Test Method</th>
<th>1</th>
<th>2</th>
<th>3/3A</th>
<th>Paragraph</th>
<th>Requirements</th>
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<tbody>
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<td>General:</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Visual</td>
<td></td>
<td>Visual</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Uniform color and complete coverage of surface to be coated</td>
</tr>
<tr>
<td>Electroless Nickel Thickness</td>
<td>Appendix 4</td>
<td>6.5</td>
<td>4.0</td>
<td>2.5</td>
<td>3.2.1</td>
<td></td>
<td>be 3 to 6 µm [118.1 to 236.2 µin] at ± 4 sigma (standard deviations) from the mean as measured on a nominal pad size of 1.5 mm x 1.5 mm [0.060 in x 0.060 in] or equivalent area,</td>
</tr>
<tr>
<td>Electroless Palladium Thickness</td>
<td>Appendix 4</td>
<td>6.5</td>
<td>4.0</td>
<td>2.5</td>
<td>3.2.2</td>
<td></td>
<td>0.05 to 0.15 µm [2 to 12 µin] at ± 4 sigma (standard deviations) from the mean as measured on a nominal pad size of 1.5 mm x 1.5 mm [0.060 in x 0.060 in] or equivalent area,</td>
</tr>
<tr>
<td>Immersion Gold Thickness</td>
<td>Appendix 4</td>
<td>6.5</td>
<td>4.0</td>
<td>2.5</td>
<td>3.2.3</td>
<td></td>
<td>0.025µm [1.2 µin] at - 4 sigma (standard deviations) below the mean as measured on a nominal pad size of 1.5 mm x 1.5 mm [0.060 in x 0.060 in] or equivalent area,</td>
</tr>
<tr>
<td>Physical:</td>
<td></td>
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</tr>
<tr>
<td>Adhesion/Tape Test</td>
<td>IPC-TM-650, TM 2.4.1</td>
<td>3.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>No evidence of plating and/or solder mask removed.</td>
</tr>
<tr>
<td>Solderability</td>
<td>J-STD-003</td>
<td>3.5</td>
<td></td>
<td></td>
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<td></td>
<td>Refer to the applicable performance specification.</td>
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<tr>
<td>Force Measurement Test</td>
<td></td>
<td>3.5.1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.14mN/mm minimum for Eutectic SnPb testing. 0.19 mN/mm minimum for SAC 305 testing.</td>
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<tr>
<td>Environmental</td>
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<tr>
<td>Cleanliness</td>
<td>IPC- 5704</td>
<td>3.6</td>
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<td></td>
<td></td>
<td></td>
<td>Refer to applicable performance specification.</td>
</tr>
<tr>
<td>SIR</td>
<td>IPC-TM-650, TM 2.6.3.5 GR78-Core</td>
<td>3.6</td>
<td>3.6</td>
<td>1.0E+08 ohms 1.0E+10 ohms</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
4 QUALITY ASSURANCE PROVISIONS

4.1 General Quality Assurance Provisions
These are specified in IPC-6011 and each IPC-60XX sectional specification. Additional requirements for printed boards with electroless nickel/electroless palladium/immersion gold (ENEPIG) plating are specified herein for qualification, acceptance, and quality conformance.

4.1.1 Qualification Recommendations
Qualification of a printed board supplier, supplying ENEPIG to this specification shall be AABUS (see IPC-6011). The process capability of a supplier of PWB with ENEPIG finish shall be established by demonstrating process control over time. APPENDIX 3 provides a detailed discussion on qualification aspects of the ENEPIG process by a printed board supplier. Table 4-1 provides a listing of suggested qualification elements that should be exercised by the supplier. It includes the associated requirements and/or test methods when applicable.

4.1.2 Sample Test Coupons
Test specimens used for qualification of printed boards with ENEPIG finish may be found in IPC-2221 and/or noted in IPC-6010 sectional specifications. Alternatively, the supplier may create test patterns or process control coupons, to accommodate qualification testing and/or lot conformance deposit thickness measurements. Specimen(s) should be obtained or developed for qualification testing to the requirements listed in Table 4-1.

Table 4-1 Suggested Fabricator Qualification Plan

<table>
<thead>
<tr>
<th>Requirement</th>
<th>Test Reference</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>XRF Certification</td>
<td>Suitably calibrated XRF instrument with detector capable of measuring tri-level thin coatings. Use of NIST certified ENEPIG standards as referenced in Appendix 3. Note: Do not use electrodeposited standards or ones with thicknesses outside the target thickness range under evaluation.</td>
<td>Perform XRF certification by performing a gauge R&amp;R or equivalent statistical approach. Use of the correct NIST standards is critical. This data may be used as to begin building a suitable deposit thickness distribution database.</td>
</tr>
<tr>
<td>Deposit Thickness Distribution</td>
<td>Suitable test pattern or production board/panel having the correct surface area pads as described in Appendix 3.</td>
<td>The deposit thickness database to demonstrate 4 sigma distribution should include sample readings from product processed through the production baths. Include readings from both front and back of the panels. Also map thickness as a function of panel position within a basket. It is suggested that thickness measurements be made at panel locations that provide as complete panel coverage as possible, e.g., diagonal, four corners and center, each PWB within a panel.</td>
</tr>
<tr>
<td>Visual</td>
<td>Ref. paragraph 3.1</td>
<td>Perform visual inspection per applicable requirements at a production final inspection workstation and document the appearance of the finish. It is recommended to create an inspection checklist.</td>
</tr>
<tr>
<td>Topic</td>
<td>Description</td>
<td>Notes</td>
</tr>
<tr>
<td>------------------------------------------------</td>
<td>-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------</td>
<td>-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>Solderability (Wetting Balance)</td>
<td>Use suitable coupon(s) to perform J-STD-003 Category 3 testing. See paragraph 3.5 and 3.5.1. Note: IPC-1601 provides guidance on packaging and handling of coupons.</td>
<td>It is recommended to qualify to Category 3, the highest level of storage coating durability in J-STD-003.</td>
</tr>
<tr>
<td>Wire Bondability</td>
<td>Test specimen suitable for use with MIL-STD 883, METHOD 2011.8 or User defined test pattern/specification Method 2011.8 of MIL-STD-883 is the most commonly used test method requirement for determining wire bond strength. Other user-defined AABUS measurements may be required. Document a sufficient quantity of bond pulls to establish capability. Assistance by the user may be required to support this method.</td>
<td></td>
</tr>
<tr>
<td>Ionic Cleanliness</td>
<td>Suitable specimen for IPC-5704 Ion Chromatography (IC) Testing IC testing per IPC-5704 is recommended during qualification to confirm that the process and associated rinses are adequate to ensure that no contaminates remain that might not meet next level assembly requirements. Periodic re-qualification may be necessary to account for changes in materials and processes.</td>
<td></td>
</tr>
<tr>
<td>PTH Coverage</td>
<td>Suitable Through Hole specimens for microsectioning Microsection a number of “as received” specimen(s) using darkfield illumination (before solder float). Utilize coupon specimens from a variety of locations, for example, from the outside and center panels of a basket load.</td>
<td></td>
</tr>
<tr>
<td>Effect on Soldermask Adhesion</td>
<td>Para 3.4 - Suitable coupon of IPC-2221 or Production Part containing high density features. Utilizing an appropriate specimen, benchmark adhesion of solder mask. Inspect to Quality conformance requirements, or as specified by User. Note whether the solder mask was applied before or after ENEPIG processing.</td>
<td></td>
</tr>
<tr>
<td>Repeatability at Various Stages of Loading and Bath Life</td>
<td>Applicable specimens of the Qual Plan Repeat aspects of the Qualification Testing at various bath life (MTO) stages, as applicable, looking for any impacts on deposition thickness or distribution, (for example, for new baths and late MTO).</td>
<td></td>
</tr>
</tbody>
</table>
4.1 Quality Conformance Testing

Quality conformance testing shall consist of inspections as specified in Table 3-1 and the IPC-6010 sectional specification.

4.1.1 Thickness measurement frequency shall be in accordance with Table 3-1. The number of readings per panel are to be enough to calculate a significant standard deviation. A minimum of 20 readings per lot is suggested.

4.1.2 If a qualified process has been established, the requirements of 4.1.1 for a minimum number of readings and calculation of an associated standard deviation can be waived. For a qualified process, demonstrating a process capability (+/- 4 sigma) within the appropriate specification, a sample measurement consistent with Table 3-1 may be used for lot conformance, provided all readings fall within the Qualified Process population (+/- 4 sigma). Alternative thickness inspection methodologies shall be AABUS.

For gauge capability and Gauge R & R, see APPENDIX 10.
Chemical Definitions

Electroless Process – This chemical process promotes sustained deposition of a metal or metal alloy onto the PWB surface through an oxidation-reduction chemical reaction, without the application of an external electrical potential. Reducing agents, such as sodium hypophosphite or sodium formate, react at catalytic surfaces to release electrons, which immediately reduce the positively charged metal ions (e.g. nickel ions in ENIG and ENEPIG and palladium ions in ENEPIG), promoting their deposition onto the PWB.

This type of reaction is described as “autocatalytic”, as the deposition process will continue even after the substrate is completely covered by a continuous layer of the plated deposit. The deposit thickness will therefore continue to rise in the presence of source metal ions and a reducing agent, until the board is removed from the plating bath. The thickness of plated deposits will vary depending on the bath temperature, chemical parameters (such as solution pH) and the amount of time spent in the plating bath.

Immersion Process – This chemical process uses a chemical displacement reaction to deposit a layer of a second metal onto a base metal surface. In this reaction, the base metal dissolves, releasing the electrons that reduce the positively charged ions of the second metal present in solution. Driven by the electrochemical potential difference, the metal ions in solution (e.g. gold ions in ENIG or ENEPIG process) are deposited onto the surface of the board, simultaneously displacing ions of the surface metal into solution.

This type of reaction is described as “self-limiting” because, once the base metal is covered with a continuous layer of the deposited metal, there is no longer a source of electrons and the reaction ceases.

Process Sequence

1. **Cleaner** – The purpose of this step is to clean the copper surface in preparation for processing. The cleaner removes oxides and light surface contaminants, and ensures that the surface will be in a condition allowing it to be uniformly micro-etched. Vendor specifications for temperature, dwell time, agitation and bath chemical control should be followed.

2. **Microetch** – The purpose of this step is to produce a surface that may be uniformly catalyzed and plated with good deposit adhesion by removing some copper from the surface. A variety of different etchant types may be used (e.g. sodium persulfate, peroxide/sulfuric). Vendor specifications for temperature, dwell time, agitation and bath chemical control should be followed.

3. **Catalyst** – The purpose of this step is to deposit a material that is catalytic to electroless nickel plating on the copper surface. The catalyst lowers the activation energy for nickel deposition and allows plating to initiate on the copper surface. Examples of metal catalysts include palladium and ruthenium (typically deposited by an immersion reaction with the copper surface). Vendor specifications for temperature, dwell time, agitation and bath chemical control should be followed.
4. **Electroless Nickel** – The purpose of this bath is to deposit the required thickness of electroless nickel on the catalyzed copper surface. The nickel thickness should be adequate to cover the copper with a substantially pore-free coating, to create a diffusion barrier to copper migration, and also serve as a solderable surface, depending on the intended application.

The nickel bath has a relatively high deposition rate and its active chemical components must be maintained in balance on a continuous basis, by addition of appropriate replenishment components. Electroless nickel baths typically run at high temperatures and extended dwell times to achieve the required deposit thickness. It is therefore important to ensure that compatible PWB substrate and solder mask materials are used. Vendor specifications for temperature, dwell time, agitation and bath chemical control should be followed.

5. **Electroless Palladium** - The purpose of this bath is to deposit the required thickness of electroless palladium onto the initial electroless nickel deposit. The palladium thickness should be adequate to provide a surface with the desired solderability and/or wire bonding characteristics, depending on the intended application. This bath runs at moderately high temperatures. Dwell times will vary, depending on the required deposit thickness. Vendor specifications for temperature, dwell time, agitation and bath chemical control should be followed.

6. **Immersion Gold** – The purpose of this step is to deposit a thin, continuous layer of immersion gold. The gold protects the underlying electroless nickel / electroless palladium layers from oxidation or passivation, and also serves as a contact surface, depending on the intended application. This bath runs at relatively high temperatures and dwell times. Ensure compatibility of substrate, and solder mask if used. Vendor specifications for temperature, dwell time, agitation and bath chemical control should be followed.

7. **Rinsing** – The purpose of these steps is to remove residual process chemicals from the PWB surface after each chemical processing step. This may be achieved in either a single step or with multiple rinses. In some instances, pre-dip and/or post-dip process steps may also be required for optimum process performance. Vendor specifications for temperature, dwell time, agitation and turn-over rate should be followed.

8. **Drying** – The purpose of this step is to ensure the boards are completely dry. This may be achieved by use of either in-line vertical, or off-line horizontal drying. Off-line horizontal drying should be preceded by a horizontal rinsing step and should be dedicated to the boards from the ENIG / ENEPIG processes. The time and temperature should be optimized to suit the type of product.

9. **ENIG / ENEPIG Process Combination** – Due to the very substantial overlap between the process flows for production of ENEPIG and ENIG, single process lines can be designed to be capable of producing either product type, by use of different line programs.
Objective: To determine the appropriate specification limits for electroless palladium in ENEPIG for soldering and wire bonding applications.

Supplier Participation:

Six suppliers namely: Atotech, Cookson Electronics, Dow Electronics Material, MacDermid, OMG and UIC Uyemura, committed to providing samples of the required finish for the Round Robin testing:

Thickness Measurement:

Industry capability to accurately measure the thickness of palladium in thickness ranges desired to be applied to the test vehicle (TV) values was questioned by members of the sub-committee. Prior to the preparation of the samples for the round robin testing of solderability and wire bonding performance, it was decided to obtain an ENEPIG test sample to be sent to interested participants with measuring equipment to determine industry capabilities to reproducibly measure thickness values. The test sample had 16 pads designated for measurement and recorded values were sent to Gerard O’Brien for compilation. The results are provided in the specific Appendix 3, authored by Gerard O’Brien. Participants in this study were able to use this data to confirm their own measurement capability and to identify any need for changes in their measurement protocols.

Solderability and Solder Joint Reliability Testing

The Test Coupon

The “W Coupon” described in the IPC-6012 document used for this phase of the Round Robin. The coupon is 6.5 X5.5”, it has 18 sub-units as shown above, as well as 2 detachable BGA coupons. All submitted samples will be subjected to the stress or conditioning for 8 hours at 72°C [162°F] and 85%RH.

Thickness of the ENEPIG to be tested:

After additional discussions, the thicknesses of the multiple layers to be tested were agreed upon and all suppliers targeted the desired thicknesses seen next.

- Electroless Nickel (EN): 5 µm +/- 1 µm
- Electroless Palladium (EP): 0.1 µm, 0.2 µm, 0.3 µm and 0.5 µm*
  *(from only one supplier).
- Immersion Gold (IG): Supplier preferred immersion gold process (one supplier chose to supply a second ENEPIG sample set with a very thin immersion gold layer).
All combinations will be tested as follows:

- Soldering with eutectic solder
- Soldering with Lead Free SAC305 alloy

**Sample Size**

Each supplier agreed to provide 6 “W Coupons” per thickness iteration. All samples were shipped to Gerard O’Brien for coding, prior to being sent to the test locations, to ensure anonymity of the supplier. Gerard will also maintain a record of the actual thickness plated on each set of coupons, based on measurements made at a single (referee) test location.

**Solderability/Wettability**

This was evaluated using “Wetting Balance” as well as Spread test coupons. Wetting Balance was carried out by G. O’Brien of ST and S Group (see APPENDIX 5). Solder spread testing was conducted by Brian Madsen of Continental Automotive Systems (see APPENDIX 6).

**Solder Joint Reliability**

Ball Shear testing on the BGA portion of the coupon was used to make this assessment. The testing was carried out at Rockwell Collins by David Hillman. Both force and fracture mode were recorded. Cross sections of representative samples were prepared to examine the composition of the intermetallic compound (IMC) and its configuration. The data is reported by David Hillman in APPENDIX 7.

**Wire Bonding Evaluation**

**Wire Bonding Coupon**

The test coupon design was provided by Horst Clauberg of Kulick & Sofa. James Monarchio at TTM manufactured the test coupons. Jim supplied sets of test coupons at 2 different levels of surface roughness. One to be particularly coarse (RA ~ 380), utilizing mechanical scrubbing. The surface roughness was measured by profilometry at Enthone by Karl Wengenroth.

**The Thickness of the ENEPIG to be tested:**

The thickness is the same as the solderability test [see (1) above]. All combinations were tested as follows:

- Gold wire bonding utilizing ball, wedge or crescent.
- All samples are pre-conditioned for 4 hours at 150°C [302°F], before bonding.

The wire bonding testing was done by Stephen Meeks of St. Jude Medical. For the protocol and results of the wire bonding evaluation, please refer to the APPENDIX 8 by Stephen Meeks.
APPENDIX 3

ENEPIG PWB Surface Finish XRF Round Robin Testing

Gerard O’Brien – President S T and S Group

Introduction:

Measurement capability for thin immersion coatings has long been a concern for the 4-14 Subcommittee and with the introduction of Electroless Nickel/Electroless Palladium/Immersion Gold (ENEPIG), this has become a bigger issue with the need to measure three plating deposits. Additionally there is the impact of the phosphorus content in the Electroless Palladium that was in the majority of samples submitted for the generation of this specification. For previous 455X family specifications, the committee has typically verified the deposits submitted by the chemical suppliers using a reference XRF (X-ray Fluorescence) and the delta between the readings shared with the suppliers and a general comparison chart generated. There were a significant number of ENEPIG users and printed board (PB) suppliers as well chemical suppliers involved with the generation of this specification who expressed an interest in understanding the variability and accuracy issues with measuring using XRF and so it was decided to perform a round robin test with all interested parties measuring a previously “certified” coupon.

A previously plated test coupon used for switch pad testing, but never tested, was chosen for this purpose at random and sixteen locations on the coupon were identified for measuring. A letter detailing the protocol was supplied with the sample and sent initially to the six chemical suppliers prior to plating their test submissions. The test vehicle was then forwarded around the world to OEMs, CEMs and PB suppliers. The data was returned to S T and S and coded to protect the source of the data. Feedback was provided to each test site as to where there readings were relative to the actual values for Ni, Pd and Au. Based on some of the data generated, the test location asked for another chance to read the coupon after their XRF had been adjusted or repaired.

The XRF capability used for this round robin testing ranged from simple proportional counter systems all the way through vacuum assisted focused capillary systems costing $200K+. It should be noted that the higher cost systems did not always produce the correct answers and that a correctly set up XRF system of any type with sufficient measurement time can be capable of correctly measuring the ENEPIG deposit.

It was discovered during this testing that the use of incorrect calibration standards is still a reality. Non phosphorus containing nickel standards were a common occurrence as well as gold foils outside the measurement range. It cannot be emphasized enough that correct measurements
can only be obtained when using ENEPIG standards with the phosphorus content in the nickel and palladium matching that in the plated deposit.

Figure 1 shows the test coupon and pertinent information from the protocol sent to the test locations. Figures 3, 4, and 5 show the recorded test data for the Au, Pd, and Ni measurements, respectively. Level C shows the reference data.

![Figure 1. Test Coupon Used for XRF Round Robin Measurements](image)

**Pertinent Information from the Protocol Sent to All Round Robin Test Locations:**

1) Record the collimator used for the measurements.
2) Record the measurement time.
3) Take only one reading per pad.
4) If multiple readings are taken to examine variability of the readings, record them on a separate sheet.
5) Record what material the coupon sat on when measured.
6) Include the calibration file parameters used to create the file, whether standards were used or just fundamental parameters.

Note: There were no measurements taken in the pads to the immediate left of pad #s 13 & 15, as they have a centered hole in them. For pads #s 14 and 16, there are also holes present but they were offset enough so as not to interfere with the XRF readings.
### Means and Standard Deviations

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<th>Std Dev</th>
<th>Std Err Mean</th>
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Figure 2. Au Thickness Readings in Microinches for the Sixteen Test Pads Where C Is the Reference XRF
Figure 3. Pd Thickness Readings in Microinches for the Sixteen Test Pads Where C Is the Reference XRF
<table>
<thead>
<tr>
<th>Level</th>
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<th>Mean</th>
<th>Std Dev</th>
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<td>6.01565</td>
<td>1.5039</td>
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</table>

Figure 4. Ni Thickness Readings in Microinches for the Sixteen Test Pads Where C Is the Reference XRF
Results:

Table 1 - Comparison of the Accuracy of Measurements Relative to the C Readings for All Three Deposits

<table>
<thead>
<tr>
<th>XRF machine</th>
<th>Nickel Thickness</th>
<th>Palladium Thickness</th>
<th>Gold Thickness</th>
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<tbody>
<tr>
<td>A</td>
<td>Low</td>
<td>Low</td>
<td>High</td>
</tr>
<tr>
<td>B</td>
<td>Low</td>
<td>High</td>
<td>High</td>
</tr>
<tr>
<td>C</td>
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<td>Low</td>
<td>Correct</td>
<td>High</td>
</tr>
<tr>
<td>E</td>
<td>Correct</td>
<td>Low</td>
<td>High*</td>
</tr>
<tr>
<td>F</td>
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<td>Low</td>
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</tr>
<tr>
<td>G</td>
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</table>

*Note – Readings were so far out as to be meaningless

From the round robin testing, the following can be stated:

1) For Immersion Gold readings, the majority of the readings were on the high side with only one location reading the thickness correctly. Two locations produced readings; one high and one low; that were so far out of the range as to be meaningless.

2) For Electroless Palladium, four of the machines recorded readings that were correct with the majority of the other readings being low. Again, one of the machines that produced meaningless gold readings also produced similar readings for Palladium and maintained consistency regarding the error reading being low.

3) For the Electroless Nickel, there were eight participants that were within an acceptable range of the actual readings. The remainder of the locations read the sample LOW.

In addition to this testing, another series of XRF measurements were taken to evaluate the impact of feature size on the deposit thickness plated at the same time on the same board. The board was again measured on the same reference XRF unit. A summary of the results is shown in Figure 5.
Figure 5. The Impact of Feature Size on Pd and Au Deposition Thickness Plated on the Same PB

Summary:

- Based on the round robin testing, there should be concern regarding accuracy of ENEPIG measurements. If the protocols outlined in the XRF APPENDIX 4 are not followed, then some equipment will at best be a random number generator.
- It is imperative that Gauge Repeatability & Reproducibility protocols be run on all XRF equipment used in the supply chain and comparison testing using known NIST traceable standards to ensure accuracy of measurement and hence performance of the ENEPIG deposit.
- Some equipment currently in use for other surface finishes will not meet the requirements for accuracy and repeatability and will need to be replaced – this is the cost of supplying and using ENEPIG as a multifunctional surface finish.
- Feature size affects the deposit thickness for ENEPIG similar to other finishes. – The use of the specified 60 mil X 60 mil pads cannot be emphasized enough to minimize variation between measurements that will make hitting the proposed specification limits nearly impossible.
- The XRF supplier base was absent from the study and their lack of participation in correctly setting up, evaluating equipment and suggesting alternatives for older equipment is troubling, to say the least. Without their support, ENEPIG performance will be open to criticism that is not warranted, due to incorrect thicknesses being supplied.
APPENDIX 4

Factors Affecting Measurement Accuracy of ENEPIG Coatings by XRF

Frank Ferrandino, Technical Mgr.
Calmetrics Inc.

XRF has become ubiquitous in the plating industry for measuring plated layer thicknesses. Among its many virtues, the XRF method tends to typically be robust for a wide range of plating applications. However, the plating industry in response to increasing demand for reliability, continues to develop new coating combinations that can make the analysis of thickness by XRF more complicated and therefore increasingly subject to error if not calibrated and used properly. In particular, the use of ENIPIG coatings in the PCB industry, to improve shelf life and solderability, present some challenges to the typical XRF instrument used to measure it.

Typical XRF Instruments Used for Plating Thickness Measurements

A sizable majority of the XRF instruments used by PCB manufacturers are good general purpose plating thickness testers equipped with either a tungsten (W) target or molybdenum (Mo) target x-ray source and gas filled proportional detectors. Such products are capable of achieving reasonably good accuracy when measuring ENIPIG plating thicknesses. On the other hand, it is also easy for measurement accuracy to be compromised when such XRF instruments do not have certain software functions or are not properly calibrated.

Below is a list of potential sources of errors when measuring ENIPIG coatings with proportional counter XRF systems using W or Mo x-ray sources:

(1) The PCB epoxy laminate often contains fire retardant compounds of bromine. The default measurement condition for these types of XRF instruments is to count the number of Au x-rays detected for the Au L-β line (energy ~ 11.4 keV). This is because the Au L-α peak overlaps the Cu x-ray peaks which originate from the Cu layer. In XRF analysis, we try to avoid interferences caused by such overlaps when possible, hence the usual choice of analyzing the Au L-β line.

Normally when plating on Cu or Cu alloy substrates there is no interference with the Au L-β peak. However, in the case of PCB samples, there is a reasonably good chance that some Br x-ray emission will be detected from the epoxy substrate. Given the relatively poor energy resolution of proportional counter x-ray detectors, Br, with a K-α energy of ~ 11.9 will produce a spectral peak that overlaps or interferes with the usually reliable Au L-β peak. Normally, Br x-ray intensity will be low since the Br x-rays must pass through the Cu, Ni, Pd, and Au layers to reach the detector and be counted. These layers provide significant reduction in Br intensity due to shielding, resulting in only a small, minor Br peak intensity.

In the case of immersion Au layers, which only produce a low intensity Au L-β peak, this small Br contribution to the peak intensity of Au, if not corrected, can be significant since
the Br contribution to the Au peak intensity is roughly the same level of magnitude or even greater compared to the Au peak intensity itself.

When measuring immersion Au layers in the 0.05 µm – 0.13 µm [2 – 5 microinch] range uncorrected Br interference can add anywhere from a few sub-microns or microinches for the gold measurements to as much as tenths of microns or tens of microinches. Therefore, a Au layer which is truly 0.1 micron [4 microinches] for example, may measure anywhere from 0.15 µin to 0.25 µm [6 µin to 10 µin ] or more, if the Br interference is not accounted for and corrected. The amount of error will depend primarily on the thickness of the Cu layer (thinner copper results in larger errors in an Au measurement), the amount of Br compound in the epoxy and the spatial resolution of the x-ray beam and its position relative to the plated areas being measured.

Fortunately, most if not all the XRF instruments used for plating thickness measurements offer peak deconvolution software. This software will allow the instrument to breakdown the composite Au + Br peak into its component parts. Therefore, it is possible to extract Au L-β peak intensity information independent of the contributing interference from Br. For accurate measurement of immersion Au on PCB’s, use of a peak deconvolution routine is prudent and in many cases absolutely vital to maximizing the accuracy of the Au thickness measurement. It should be noted that significant errors in Au thickness measurement due to uncorrected Br interference will contribute to errors in the calculation of underlying electroless Ni and Pd thicknesses, as well.

(2) A second source of potential error lies with the electroless nickel layer. Proportional counter based XRF instruments cannot measure phosphorous content in the Ni layer directly. Therefore it is generally assumed in the calibration of the instrument that the Ni layer contains some constant amount of phosphorous composition, typically about 8%. However, if the sample does not contain 8% phosphorous in the Ni layer, errors in electroless Ni thickness will occur. As a very rough rule of thumb, one can expect about a 4% thickness error for each 1% difference in phosphorous content between the sample under test and the standards used for calibration.

The electroless nickel thickness measurement will be erroneously high if the sample phosphorous content is less than the calibration standard. The measurement of thickness will be erroneously low if the sample contains more phosphorous than the standards used for calibration. Since most platers maintain the phosphorous content at approximately 8%, the electroless nickel thickness measurement error is normally minor.

Although electroless Ni thickness errors due to phosphorous content variations are normally less than 10%, it is possible to correct for changes in sample phosphorous content if they are known. Some XRF software does offer such corrections which allow the user to enter the known % P content in the nickel layer and the thickness measurement will automatically be corrected. Alternatively, suppliers of such XRF instrumentation should be able to provide correction factors for changes in sample % P which can be entered as “density” factors that can correctly compensate for changes in sample % P.
Measurement of the immersion Pd layer is subject to two possible errors. The more significant potential error is caused by variations in the x-ray background level in the spectral region where the Pd K-\(\alpha\) peak is detected. The Pd K-\(\alpha\) peak is added to this background level. Typically, XRF plating thickness measuring instruments integrate this area to obtain Pd intensity and relate that intensity to Pd thickness. In the case of immersion Pd where thicknesses are in the order of 0.05 \(\mu\)m – 0.15 \(\mu\)m [2 \(\mu\)in – 6 \(\mu\)in], the spectrum background level is often as intense or more intense than the Pd peak itself. If the background level is constant, this influence may be included in the calibration. However, the background level may, in fact, vary. This potential background level variation is a function mainly of the Cu layer’s thickness below the Pd.

The background scatter originates mainly from the epoxy substrate. The W or Mo target x-ray sources provide a broad band of x-ray energies to the sample during the measurement. It is mainly the higher energy x-rays that will penetrate the thick Cu-clad laminate, scatter back from the epoxy through the Cu layer to be detected and seen in the measured spectrum. Lower energy x-rays originating from the source, do not have enough energy to penetrate the Cu layer twice and be detected. Therefore background “noise” is only an issue in the higher end of the x-ray energy spectrum.

It is in this high end of the spectrum that the Pd K-\(\alpha\) appears (~ 21.1 keV). The amount of background scatter that reaches the detector is again a function of the Cu thickness since the scattered x-rays are shielded primarily by this relatively thick layer. As with the Br interference with Au, the background scatter will be a function of the underlying Cu thickness as well as the spatial resolution of the x-ray beam and the position of the beam relative to the edges of the sample plated area.

Again, to be prudent and to optimize accuracy of Pd thickness measurements, the potential varying background level should be compensated for to obtain net Pd intensity information. Most XRF instruments are equipped with background correction software to deal with this issue and obtain reasonably accurate net Pd intensities. The user should be familiar with how to access this feature if its use is optional, or know if automatic background correction is always used by their XRF instrument to measure plating thickness. There are a wide variety of background correction schemes that are available, some of which are more effective in this case than others.

Other approaches to mitigate Pd measurement errors due to varying background levels include the use of primary beam filters to attempt to suppress the background level to a magnitude far below that of the Pd K-\(\alpha\) peak intensity. This method can work well when using for example a Mo filter with a larger collimator. Since primary filters reduce overall signal intensity the filter material and thickness must be selected with an attempt to balance the gains from reducing the background and the loss due to reduced Pd signal intensity. Typically, primary filters are effective only when using larger x-ray beam collimators. Users should consult with the manufacturer of the instrument before attempting to use a primary filter.
Less significantly for the commonly used XRF instrument is the error bias that may be introduced by the fact that immersion Pd layers are not pure Pd. Instead, it is common that such layers contain about 2% phosphorous. Like the electroless nickel measurement, the level of phosphorous in the plating has some effect on the thickness measurement.

In the case of immersion Pd however, we can normally assume this effect is insignificant for two reasons:

a) The Pd thickness is very low where the thickness measurement is not very dependent on the layer purity

b) The Pd x-ray energy is almost 3X higher than Ni so that the absorption of Pd x-rays within the layer by phosphorous is much smaller than it is for nickel. This combined with (a) above will result in errors of about 5% assuming the calibration standards are pure Pd (which is typically true).

Much of this error originates with the density difference between immersion Pd and pure Pd used for calibration standards. By requesting standards with the Pd certified to a density of 11.6 g/cc instead of the conventional 12.0 g/cc, the bias error is reduced to less than 2%.

Long term stability of XRF calibrations using proportional counter detectors for ENIPIG applications is often compromised primarily by the use of the peak deconvolution method for the Au. This method places higher demands on the detector electronics stability, making the measurement more sensitive to instrument drift. To avoid additional error due to this effect, one should check calibration standards frequently, at least twice a day, to assure that the instrument is still within calibration tolerances. Use supplier provided drift correction features to correct for drift if standard measurements are outside their tolerances. If drift correction fails to restore the calibration to within tolerance, the instrument should be recalibrated.

**Summary for Common Plating Thickness Measuring XRF Instruments**

The predominantly used XRF type in the PCB industry to measure ENIPIG coatings should be calibrated with the following considerations in mind:

1. Use of peak deconvolution for the Au layer measurement.
2. Use of an appropriate background subtraction model for the Pd layer.
3. Use of appropriate corrections for differences in phosphorous content between calibration standards and test samples for the nickel layer and, less importantly, the Pd layer.
4. Use of calibration standards with similar thicknesses to the specified ENIPIG thicknesses which are to be measured. If possible, request Pd thickness certification to the level of % P expected in the Pd layer and request electroless nickel layer(s) with % P content within 1% of the expected % P content for the samples which are to be measured.
5. It is also helpful if the standards are plated over a Cu laminated epoxy substrate containing Br if the samples to be tested will also contain Br.

By requesting standards with these parameters, one can most closely approximate the inter-element matrix effects and spectral effects discussed above, resulting in an optimized calibration which minimizes thickness measurement error. Use calibrations standards to check and if necessary correct for long term stability problems. Calibrations standards are
used to check that the calibration is still within tolerance from day-to-day, to assure that long
term stability limitations of the instrument do not compromise results.

**Alternative XRF Instruments & Configurations: Advantages and Disadvantages**

In recent years, more capable XRF instruments have been introduced for plating thickness
measurement. The most significant advance involves the use of solid state detectors, typically
silicon PIN diodes and most recently silicon drift detectors (SDD). These detectors offer a major
advantage over the common proportional counter used in XRF’s employed in the plating
industry in terms of energy resolution. It is possible to achieve energy resolutions of about 5X
better than the proportional counter with these detectors. The advantage instruments with these
detectors offer is primarily in the Au layer measurement. In the case of Au, the Au L-β peak and
the Br K-α peaks can be better resolved, reducing overlap interference.

Furthermore, the Au L-α peak is fully resolved from any overlap with the Cu K peaks.
As a result, the Au L-α peak may be used without resorting to deconvolution techniques. While
peak deconvolution routines needed for proportional counters minimize error due to peak
interference from other elements like Br, they include their own inherent potential errors, smaller
but nevertheless present. Peak deconvolution is especially difficult to employ well when plating
thickness becomes very thin. For Au layers less than 3 microinches, peak deconvolution
methods can struggle to achieve good accuracy when Br is present.

In addition, the nature of the peak deconvolution method requires excellent electronics stability
to maintain measurement accuracy without frequent recalibration. In some cases, instrument
design does not offer the needed stability to avoid frequent drift correction and recalibration.
Use of solid state detector based XRF systems eliminates the need for peak deconvolution
methods with respect to Au thickness measurements and permits Au measurements of layers less
than 1 microinch. Therefore, solid state detector XRF systems tend to offer much better long
term measurement stability and are much less reliant on operator know-how and vigilance to
achieve accurate Au results.

Solid state based XRF instruments offer some advantage for Pd measurement as well.
Although the varying background scatter issue is still present, the overall background level tends
to be lower compared with proportional counter systems, again allowing a clearer view of the Pd
peak above the background. This translates into the ability to measure lower thicknesses of Pd
using solid state detectors relative to proportional counters. The disadvantage in terms of Pd
measurement is that solid state detectors are not as efficient as proportional counters in detecting
the high energy Pd K-line x-rays. As such, overall x-ray intensity for the same Pd thickness will
be less when using a solid state detector than a proportional counter, given all other factors being
equal. This disadvantage is partially negated by the overall lower background levels observed
with solid state detectors.

Other considerations noted for the proportional counter based XRF systems must be applied as
well with solid state detector based systems. These include corrections for phosphorous content
in both the nickel and again, to a lesser extent, the Pd layer. The advantages offered by the solid
state detector when measuring ENEPIG come with some disadvantages as well. First, the cost
for solid state detector based systems is higher than proportional counter based systems. Second,
the size of the detection area is smaller than proportional counters. As a result, less x-rays are
detected per unit time under the same excitation conditions. Generally, larger x-ray collimators must be used to compensate for the smaller detection area. This means that measurement of sample areas less than 12 mils wide or in diameter can be problematic with solid state detector based XRFs. Typically longer measurement times must be used to achieve the same level of measurement repeatability as a proportional counter based XRF. However, if collimators that are 20 mils or larger can be used, then measurement time for both types of XRF’s can be comparable to achieve similar repeatability.

**XRF Instruments Using Cr Target X-ray Source**

Some proportional counter based XRF systems have been provided by at least one manufacturer using a Cr target x-ray source. The principle advantage of the Cr target source is its ability to excite the Pd L series peaks (~ 3 keV). Once these peaks are sufficiently excited, measurement precision is significantly improved for the Pd layer since the sensitivity of Pd L line x-ray intensity, in terms of unit thickness change, is excellent. This high sensitivity to Pd thickness change is especially advantageous in the working range for immersion Pd thicknesses used in typical ENEPIG applications. The improved measurement repeatability and shorter measurement times that may be used, allow for better process control. Furthermore, by shifting the Pd analysis from the high energy K line typically used to the low energy L line, the issue of background scatter variations from the substrate is eliminated. This is because low energy scatter will not vary in this case.

Thus the measurement of Pd is less dependent again on operator know-how and vigilance. By using the Cr target source with a solid state detector, one may have the same advantages the Cr target offers for the Pd layer and achieve the advantages for the Au layer offered by the solid state detector. Again there is a down side to this strategy as well. Very small areas (< 12 mils) are difficult, if not, impractical to measure (at least long measuring times are required). Secondly, Cr target sources typically do not have as long an operating life as the common W or Mo target sources. Therefore, cost of ownership is higher.

**Vacuum Based XRF Systems**

Even higher cost XRF systems are available that use only solid state detectors and offer the ability to evacuate the X-ray chamber of air. By evacuating the X-ray chamber, one gains the ability to directly detect phosphorous x-rays. Detection and counting phosphorous x-rays allows for the direct measurement of phosphorous content in electroless nickel. This provides the user with direct, accurate composition measurement and allows the XRF to simultaneously calculate electroless nickel thickness based on the actual phosphorous content. This represents the most accurate way to measure electroless nickel layers.

The same advantages and disadvantages described above for measurement of Au and Pd layers using solid state detectors applies with one exception. If one chooses to configure the vacuum XRF with a Cr target source to analyze the Pd L peaks intensity or alternatively uses x-ray optics to produce microbeams to measure very small sample areas, the use of an evacuated environment reduces background substantially for the Pd L peak. When measuring Pd L intensity in atmosphere, argon fluorescence interferes with Pd L peaks (Ar is 1% of the atmosphere). By evacuating the sample chamber, this effect is eliminated, making Pd L peak intensity determination more precise.
To summarize the various effects one must address when calibrating and measuring ENIPIG coatings by XRF, and the various solutions offered by different XRF configurations, the following table is offered. In addition, the supporting section to this article includes some example spectra that illustrate some of the effects discussed.

**Table 1 – Summary of XRF Configuration Solutions Offered for Measurement of ENIPIG Plating on PCB's with Advantages and Disadvantages**

<table>
<thead>
<tr>
<th>ENIPIG Layer</th>
<th>Proportional Counter XRF</th>
<th>Solid State Detector XRF</th>
<th>Vacuum &amp; Solid State Detector XRF</th>
</tr>
</thead>
</table>
| Au           | 1- Use peak deconvolution to correct for Br interference  
2- Check calibration at least twice a day – drift correct or recalibrate as needed  
3- Difficult to measure < 2 mi  
4- Measure time 60 – 90 sec depending on collimator size  
5- Can measure areas as narrow as 3 mils wide or 4 mils diameters with longer measurement times (120 sec) | 1- No deconvolution needed – can measure < 1 mi – better overall accuracy  
2- Good long term stability  
3- Measure areas < 12 mils wide impractical unless instrument configured with x-ray optic  
4- Measure time 60 – 90 sec; longer for layers < 1 mi. Less time if collimator is 20 mils or larger or if x-ray optic used. | 1- No deconvolution needed – can measure < 1 mi – better overall accuracy  
2- Good long term stability  
3- Measure areas < 12 mils wide impractical unless instrument configured with x-ray optic  
4- Measure time 60 – 90 sec; longer for layers < 1 mi. Less time if collimator is 20 mils or larger or if x-ray optic used |
|              | 1- Must use background correction methods unless Cr target source used  
2- Measurement of Pd < 2 mi is problematic unless Cr target source used | 1- Must use background correction methods unless Cr source or x-ray optics used  
2- Measurement of Pd < 2 mi is possible with longer measurement times  
3- Measure areas < 12 mils wide impractical unless instrument configured with x-ray optic  
4- Use of Cr target source or x-ray optics improves precision, reduces time | 1- Must use background correction methods unless Cr source or x-ray optics used  
2- Measurement of Pd < 2 mi is possible with longer measurement times  
3- Measure areas < 12 mils wide impractical unless instrument configured with x-ray optic  
4- Use of Cr target source or x-ray optics improves precision, reduces time and allows for measurement of minimum 0.3 mi |
| Ni-P         | Correct thickness measurement for difference in % P between calibration standard and sample | Correct thickness measurement for difference in % P between calibration standard and sample | Most accurate – measures % P and corrects thickness of Ni layer automatically |
Above Figure SS-1 displays overlay of one spectrum collected on half ounce Cu/epoxy and spectrum of one ounce Cu/epoxy. Blue trace is from half ounce Cu/epoxy, red trace is from one ounce Cu/epoxy. Note the significant reduction in Br peak when Cu thickness increases from half ounce to one ounce. Also note the difference in background levels in the indicated region of the spectrum where Pd intensity will be counted. Again, background “noise” decreases as Cu thickness increases.
Above Figure SS-2 displays spectrum from typical ENIPIG plated Cu sample (no epoxy) and spectrum from one ounce Cu/epoxy (blue trace). Note the Red peak near Br is the Au L-β peak without any Br interference since the substrate for this sample had no epoxy. The figure illustrates how close the energies are between the Au L-β peak and the Br K-α (blue trace, marked as Br). Without peak deconvolution these peaks would be summed as one peak and erroneously interpreted as immersion Au thickness.
Above Figure SS-3 displays spectrum from typical ENIPIG plated on pure Cu (no epoxy) and typical ENEPIG plated on one ounce Cu/epoxy (red trace). In the case of ENEPIG on one ounce Cu, the Br peak is added to the Au peak (seen in blue with no Br interference). The red peak (Br + Au) is higher which results in a higher thickness measurement if peak deconvolution is not used. Also note the Pd peak. The red Pd peak from the one ounce Cu sample is slightly higher than the blue Pd peak because the background level scattered from the epoxy base raises the Pd peak a bit higher. Again without correction or proper calibration, this would be interpreted as slightly higher Pd thickness. Both effects will vary as the Cu thickness varies. Also x-ray beam size and position on the sample relative to its edges can affect the level of these interferences.
Above Figure SS-4 shows typical ENIPIG plated on one ounce Cu/epoxy sample when measured with a pin diode detector. Note how Au L-β and Br K-α peaks are better resolved and the Au L- α peak is visible and distinct from Cu K- β, allowing measurement of Au intensity without need for peak overlap corrections like peak deconvolution. Also note clear Pd peak with low background noise.
APPENDIX 5

ENEPIG PWB Surface Finish Wetting Balance Testing


Introduction:

Consistent with all previous specification generations by the 4-14 Subcommittee for PWB surface finishes, solderability testing using a wetting balance was performed to evaluate ENEPIG both in the “as received” condition and post temperature / humidity stressing. There were six chemical suppliers who submitted samples for testing and because of their geographical locations and the time needed to complete the plated test vehicles, some sample groups had significant “shelf life” prior to testing of up to eight months. During this time, the samples remained in their original packaging.

Test Vehicle:

The 4-14 Subcommittee which works closely with the 5-23A Task Group has settled on a wetting balance coupon that has changed little over the last 5 years or so. The latest iteration of the test coupon was built with 18 wetting balance coupons and four ball shear coupons per array (see Figure 1). The wetting balance coupon also contains the NPL spread test vehicle, which was also used during the generation of this specification and retains through holes at the opposite side (which were not used in these tests). The acid copper plating and soldermask operations required to fabricate these coupons were generously provided to the group by Mr. Luc Beauvillier, who was then with Via Systems - Oregon.

Figure 1. Example of the Wetting Balance Coupon Used for the Testing of ENEPIG
Following extensive intra-committee discussions on the target thicknesses for the specification, it was proposed that initial testing would be on samples plated to the following thickness requirements:

1) Electroless Nickel – 6 microns ±1 micron
2) Electroless Palladium – 0.1, 0.2 and 0.3 microns nominally – no tolerance was set
3) Immersion Gold – the thickness would be “as supplied”
4) Electroless Palladium at 0.5 microns nominal from two of the six suppliers, in order to evaluate a “heavy” palladium deposit

Six chemical suppliers took part by providing plated test vehicles:

1) Atotech
2) Dow
3) Enthone
4) MacDermid
5) OMG
6) Uyemura (both domestic and Japanese plated)

Some of these suppliers submitted ENEPIG samples that used a phosphorus-containing reducing agent in the electroless palladium bath, whose deposits contain approx. 2 to 4 wt% phosphorus, while others submitted samples using other non-phosphorus containing reducing agents, whose deposits are sometimes classified as “pure palladium”.

The samples remained in their original packaging until the final ones had been received for testing. This took some time in the case of some suppliers and, when ready to commence the test, some ENEPIG samples had 8 months of “natural” shelf life. Once all samples were received, the packages were opened and each group assigned a Roman numeral corresponding to a specific nominal electroless palladium thickness and supplier – this was marked on the edge rail of the array with an electric pencil to avoid surface contamination of the plating. XRF readings, using a PIN diode system with a measurement time of sixty seconds, were generated for all sample groups. As can be seen from the graphs (Figures 2 through 4) below, some of the suppliers did not correctly measure the deposit at their location, something that has been a major issue with the introduction of ENEPIG. Additionally, one supplier chose to submit samples with two different immersion gold thicknesses. The thinner of these two samples turned out to be of great value to the committee, as it produced solderability failures when stressed.
Figure 2. XRF Measurements of Gold and Palladium Thicknesses Supplied as Nominal 0.1 micron Electroless Palladium

Figure 3. XRF Measurements of Gold and Palladium Thicknesses Supplied as Nominal 0.2 micron Electroless Palladium
Figure 4. XRF Measurements of Gold and Palladium Thicknesses Supplied as Nominal 0.2 micron Electroless Palladium

Table 1 - XRF Measurements of the Nominal 0.5 micron Electroless Palladium Samples

<table>
<thead>
<tr>
<th>Supplier Identifier</th>
<th>Immersion Gold Thickness (microns)</th>
<th>Electroless Palladium Thickness (microns)</th>
<th>Electroless Nickel Thickness (microns)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Target Thickness Per individual supplier</td>
<td>0.5</td>
<td>5 - 6</td>
</tr>
<tr>
<td>F</td>
<td>0.038</td>
<td>0.51</td>
<td>4.98</td>
</tr>
<tr>
<td>G</td>
<td>0.016</td>
<td>0.45</td>
<td>6.67</td>
</tr>
</tbody>
</table>

Solderability testing was performed on a Metronelec ST88 wetting balance (see Figure 5), using a solder bath for coupon testing. Testing followed the requirements of the IPC J-STD-003 B Solderability Standard and samples were tested with both SAC305 and Eutectic SnPb solders. As per the standard, test temperatures of 255°C and 235°C were used for the SAC305 alloy and the Eutectic SnPb alloy, respectively. For the testing, a new charge of solder was used for each alloy. Similarly per the Standard, Test Fluxes # 1 and # 2 were used for Eutectic SnPb and SAC305 alloys, respectively.
Figure 5. Metronelec ST88 Wetting Balance Used for the Testing

The test coupons were removed from the array, the edges carefully “wiped” over 1200 grit silicon carbide paper and then wiped with a paper towel to remove any dust, to ensure that no epoxy smear was present on the edge of the SMT pads on the coupon that might prevent contact with the solder and produce a false result.

Following coupon preparation, each coupon was immersed into the appropriate flux for the alloy being used for five seconds, excess flux being allowed to drain. The coupon was then placed into a suitable tool holder and immersed to a depth of 0.5 mm at 90 degrees incident to the solder surface. A dwell time of ten seconds was used for each coupon. Following the test, the sample was inspected to confirm the wetting balance results and the next coupon from that group run. Ten samples per group were run for the “as-received” condition. Results are shown in Figures 6 - 15).
Figure 6. Example of the Wetting Performance for a Nominal 4 µin Electroless Palladium Tested with SnPb Solder

Figure 7. Example of the Wetting Performance for a Nominal 8 µin Electroless Palladium Tested with SnPb Solder
Figure 8. Example of the Wetting Performance for a Nominal 20 µin Electroless Palladium Tested with SnPb Solder

Figure 9. Example of the Wetting Performance for a Nominal 4 µin Electroless Palladium Tested with SAC305 Solder
Figure 10. Example of the Wetting Performance for a Nominal 8 µin Electroless Palladium Tested with SAC305 Solder

Figure 11. Example of the Wetting Performance for a Nominal 12 µin Palladium Tested with SAC305 Solder
Summary of solderability testing of ENEPIG samples in as-received condition:

1) The wetting times for SnPb solder ranged between 1 and 2 seconds, while those for SAC305 were typically below 1 second – this reduction in wetting times is attributed to the increased solder pot temperature used for SAC testing.

2) The majority of SnPb tested samples showed a continual rise to the final wetting force, some sample groups reached their maximum force after five seconds and then leveled out.

3) For the SAC test groups, the time to reach the maximum force was reduced by 1 to 2 seconds and the shape of the curve was flatter overall.

4) There were no failures in any of the test groups and most samples showed excellent intra-group consistency.

5) Samples tested with 8+ months of “natural” shelf life performed better than some newer samples with similar gold and palladium thicknesses.

To evaluate the robustness of the ENEPIG deposits, wetting balance coupons were stressed using a protocol of 8 hours of 72°C [162°F]/85% R.H exposure. The wetting balance coupons were sent to Continental’s Seguin Texas location where the wetting balance coupons and the solder spread coupons were stressed together in a single chamber in one stressing cycle, in order to reduce variability. Following stressing, the wetting balance samples were returned to S T and S for completion of testing. No additional baking was done on the stressed coupons prior to testing. The previously used protocol was used for testing with SAC305 and Eutectic SnPb.

![Figure 12. Example of a Nominal 4 µin Electroless Palladium ENEPIG Post Temperature and Humidity Stressing, Tested with SnPb – Showing Excellent Robustness](image-url)
Figure 13. Example of a Nominal 4 µin Electroless Palladium ENEPIG, with Very Thin Immersion Gold after Temperature and Humidity Stressing & Tested with SnPb.
(Note: This sample did not tolerate stressing, most likely due to the very thin gold deposit. The performance of this group was used to define the lower immersion gold limit for the specification.)

Figure 14. Example of a Nominal 6 µin ENEPIG Deposit Tested with SAC305 Post Stressing – Showing Excellent Robustness of Deposit
Summary of Soldering ENEPIG Post 8 Hours of 72°C/85% R.H Exposure:

1) The stressing protocol was able to differentiate “good” from “bad” ENEPIG deposits
2) The test results were capable of defining a minimum gold thickness for the specification
3) For the more robust ENEPIG deposits, the exposure to the stressing protocol had little impact on wetting times and final wetting forces, however the time to reach the maximum force increased.
4) Stressing deposits that were already 8 months old had little impact on the “good” samples, whereas the “bad” samples were significantly affected.

Summary:
The continuing use of the wetting balance by the 4-14 Committee to define acceptable deposit thickness and performance characteristics was once again confirmed with this round of extensive testing. From this testing, a pass-fail criterion has finally been established for the wetting balance testing of ENEPIG, which will also be included in the upcoming revision of IPC J-STD-003C.

Six sample groups of ENEPIG were evaluated and were found, in the majority of cases, to exhibit very little difference in performance as a function of electroless palladium deposit thickness. However immersion gold thickness was found to have a major impact on robustness and potentially shelf life. Differences were evident between the suppliers, especially post stressing of the deposit. Each supplier was provided only with the identifier for their own ENEPIG sample(s), allowing them to know the performance of their own process, but not of any specific competitor. It is hoped that the lower performing groups use this information to improve their process performance, especially post stressing.
APPENDIX 6
Solder Spread Testing

Brian Madsen, Continental Automotive Systems

Solder spread testing was performed on printed boards with seven different ENEPIG chemistries. Samples with a range of electroless palladium thickness (targeted thicknesses from 0.1 to 0.5 μm) were provided for each of the seven chemistries. While the intention had been to evaluate 3 to 4 electroless palladium thicknesses per chemistry through four test conditions, unfortunately a shortage of samples for some chemistries resulted in incomplete data sets (see Table S1). Two solder pastes were used to evaluate the solder spread on the ENEPIG deposits - a Sn/Pb/Ag eutectic alloy solder paste with flux activity level ROL1 and a SAC300 family Pb-free paste with flux activity level ROL0. For each solder paste, one test board was subjected to 8 hours of damp heat conditioning at 72°C [162°F] and 85% relative humidity prior to printing the solder paste, while a second board was printed with no pre-conditioning. Each individual test board contained 16 solder spread coupons (layout shown in Figure 1).

Table S1 - Preconditioning and Solder Paste Matrix Used for Solder Spread Testing for a Given ENEPIG Chemistry and Electroless Palladium Thickness
(Note: When sufficient samples available)
Solder Spread Test Method:

The solder spread test pattern consists of 6 parallel traces, each 0.5 mm thick and 25 mm long. The key aspect of the spread test is the solder paste stencil design shown in Figure 2, which consists of 18 solder paste deposits (0.5 mm x 0.5 mm) on each trace (108 total). The distance between the 18 paste deposits increases linearly across the pattern, from a minimum of ~0.15 mm [6 mils] to a maximum of nearly 1 mm [38 mils].

During reflow, solder deposits that contact each other tend to coalesce. Across the test pattern, the variation in the gaps between paste deposits requires the solder to spread more at one end of the pattern for coalescence to occur. The overall solder spread is calculated by first counting the number of distinct solder deposits remaining in each row of the pattern after reflow. Then, the counts for the rows (“x”) are entered into the following equation:
The result is a solder spread value expressed as a percent. A pattern exhibiting complete coalescence of the 18 deposits on each row would achieve a sum of 6 (a count of 1 solder deposit per row x 6 rows), yielding a solder spread of 100%. If none of the deposits coalesce, the sum is 108 and the solder spread is 0%, as expected. Figure 3 shows a solder spread result of 14%.

\[
\phi = \frac{108 - \sum_{i=1}^{6} x_i}{102} \cdot 100\%
\]

Figure 3. An Actual Solder Spread Result Showing the Deposits Counted for Each Line of the Pattern

**ENEPiG Solder Spread Results:**

**SnPb Solder**

The ROL1 Sn/Pb/Ag solder paste (using a SnPb solder profile) achieved very high solder spread percentages (> 90%) for all samples, regardless of ENEPIG chemistry, electroless palladium thickness, or pre-conditioning. There was no meaningful differentiation between any of the samples (see Figures 4 and 5).
Figure 4. Solder Spread Results Using an ROL1 Sn/Pb/Ag Solder Paste on ENEPIG Deposits

(Note: The ENEPIG deposits are from 7 different chemistry suppliers (with different electroless palladium thicknesses) using the solder spread pattern. The Figure 4 - Top is for boards as-received. The Figure 4 – Bottom is for soldering after pre-conditioning of the boards for 8 hours at 72°C [162°F] and 85% RH).
**Pb-free Soldering**

Figure 5. Solder Spread Results Using an ROL0 Pb-free Solder Paste of the SAC 300 Family on ENEPIG Deposits

(Note: The ENEPIG deposits are from 7 different chemistry suppliers (with different electroless palladium thicknesses) using the solder spread pattern. The Figure 5 – Left is for boards as-received. The Figure 5 - Right is for soldering after pre-conditioning the boards for 8 hours at 72°C [162°F] and 85% RH).

The solder spread results using a Pb-free solder paste (ROL0) showed more variation between the different ENEPIG suppliers. For boards just out of the package (no pre-conditioning), solder spread values ranged from 60-100%. The differentiation between ENEPIG deposits is interesting, and may indicate some differences between the process chemistries, but 60% would still be considered ‘good’ solder spread. For comparison, past measurements of OSP materials with the same solder paste achieved ~20% spread with no pre-conditioning. Note: A high spread value may not be necessary to achieve an acceptable solder joint, depending on the situation (pad, stencil aperture, component, etc.).

There was a negative impact to the solder spread from the damp heat pre-conditioning (8 hours, 72°C [162°F]/85% RH) on the ENEPIG deposits for the Pb-free solder paste and reflow. For samples with the same ENEPIG chemistry and target electroless palladium thickness, the solder spread for the damp heat conditioned sample was lower every time. As seen in Figure 5 above, solder spread percentages on some ENEPIG samples after damp heat dropped to values as low as 24%, while others achieved spreads near 90%.

Industry pass/fail criteria are not yet established for the solder spread test. The 24% spread value represents an average spread over 16 spread coupons (96 lines); within this board, some individual lines had very low spread, which is indicative of high wetting angles and possibly localized dewetting. Figure 6 confirms the localized dewetting, as some solder deposits after coalescing, can be seen to have pulled back from their original deposit positions along the trace.
Using XRF data collected for samples from the same batches (not the exact same boards used for spread testing), the solder spread results were plotted against measured immersion gold (IG), electroless palladium (EP) and electroless nickel (EN) layer thicknesses. No correlation was found between the achieved solder spread and the IG or EP thicknesses (all $R^2$ values were less than 0.1). The EN layer thicknesses, however, did show a relationship to the solder spread results (see Figure 7). Thicker layers of EN resulted in the highest solder spread results with the Pb-free solder paste. The lowest Pb-free spread results were observed for the thinnest EN layers – this effect was most apparent for the damp heat conditioned samples.
Figure 7. Solder Spread Results for Pb-free Solder Paste as a Function of the EN Layer Thickness in the ENEPIG Deposit
Conclusions

- The solder spread on ENEPIG with the Sn/Pb/Ag solder paste (ROL1) was very high (good), regardless of ENEPIG chemistry, deposit layer thickness, or damp heat conditioning.

- The solder spread on ENEPIG with the Pb-free solder paste (ROL0) was lower than the Sn-Pb results.

- Damp heat conditioning prior to solder paste printing negatively impacted the spread for the Pb-free solder paste. The worst samples exhibited localized areas of dewetting.

- No correlation (linear $R^2 < 0.1$) was found between the solder spread and the electroless palladium layer thickness (range tested: 0.05-0.45 μm) or IG layer thickness (range tested: 0.015-0.085 μm).

- Some correlation (linear $R^2 \sim 0.45$) was observed between the Pb-free solder spread and the EN layer thickness (range tested: 4.5-9.5 μm), with the thickest EN deposits having the highest spread and the lowest spread observed for low EN thicknesses.
APPENDIX 7

ENEPIG PWB Surface Finish Shear Test Project

Dave Hillman, Brad Williams, Tim Pearson, Ross Wilcoxon and Jennet Volden

Rockwell Collins Inc.

Introduction
Rockwell Collins conducted solderball shear testing for the IPC 4-14 Plating Processes Subcommittee as part of their efforts to create a specification for electroless nickel/electroless palladium/immersion gold (ENEPIG) circuit card finishes. A test program was conducted to assess the solderball shear strength of a series of test coupons with different electroless palladium plating thicknesses using both tin/lead and lead-free soldering processes.

Test Vehicle
The IPC 4-14 subcommittee supplied solderball shear test coupons that were 2.4 inches long x 0.93 inches wide x 0.063 inches thick. The solderball pad was 0.025 inches in diameter. The palladium thickness varied for each coupon. Table 1 lists the measured palladium thickness/coupon combinations and Figure 1 illustrates the shear test coupon.

Table 1: Test Coupon Serial Number and Palladium Thickness

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Figure 1: Left - Shear Test Coupon – Note Right Portion of the Test Coupon is Missing Due Removal for Cross-sectional Analysis; Right – Close Up View of Solder Paste Deposit Reflowed on Coupon Pads (smaller solder joints) and a Pad with a Solderball Reflowed on Pad (large solder joint)
**Solder Processes**

The shear test coupons were manually stenciled using low residue tin/lead (Sn63Pb37) and lead-free (SAC305) solder pastes per IPC-JSTD-004 and a 0.005 inch thick stainless steel stencil. A 0.025 inch diameter Sn63Pb37 alloy solder sphere and a 0.035 inch diameter SAC305 alloy solder sphere were manually placed on a random selection of stenciled deposits. The test coupons were then processed in a five zone Electrovert Omniflow reflow oven using a nitrogen reflow atmosphere. The peak reflow temperature for the Sn63Pb37 alloy was 220°C and for the SAC305 alloy was 245°C. The Time Above Liquidus for the Sn63Pb37 alloy was 30-60 seconds and for the SAC305 alloy was 45-90 seconds. The test coupons were cleaned using an in-line cleaner that used Kyzen Aquanox A4625 saponifier/deionized water solution.

**Shear Test Parameters**

The solderball shear testing was conducted using a DAGE 5000 shear test system. A BS5KG cartridge was used with a 50 gram ball shear load. A shear speed of 500 um/second, a shear height of 50 um and an over travel distance of 100 um were used in the test. Figure 2 illustrates the DAGE 5000 shear system used in the test.

![Figure 2: DAGE 5000 Shear Test System: Left – Macro View, Right – Close Up of Shear Head](image)

![Figure 3: Solderball Shear Results: Left – Coupon Pad Ripped Out of Board During Shear Test, Right – Solderball Sheared Through Solder Joint During Shear Test](image)
Test Results

Table 2 through Table 5 list the solderball shear test results. All of the shear test failures were a cohesive mode with no failure at the solder joint/coupon pad interface. The test coupon shear test results populations were consistent with the Sn63Pb37 alloy results having standard deviations that were 1-5% of the average while the SAC305 alloy results had standard deviations that were 10-30% of the average. There are no industry specification minimum values available for results comparison use.

Table 2: Sn63Pb37 Solderball Shear Test Results Coupon Set 1 (grams)

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Average 1354.29 1335.48 1337.81 1245.54 1344.19
Std Dev 29.98223 42.61057 49.26176 81.98731 50.50496

Table 3: Sn63Pb37 Solderball Shear Test Results Coupon Set 2 (grams)

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Average 1320.765 1370.1 1350.65 1323 1338.61
Std Dev 49.71872 16.777697 29.891889 37.450679 40.204324
Table 4: SAC305 Solderball Shear Test Results Coupon Set 1 (grams)

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Average 1973.06 1534.69 2129.83 2269.385 2041.14
Std Dev   557.7766 299.5225 436.0666 460.6777 213.892

Table 5: SAC305 Solderball Shear Test Results Coupon Set 2 (grams)

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Average 2520.14 2198.3333 2081.525 2252.2456 2171.64
Std Dev 331.9375 367.41668 529.90524 412.21435 546.8967
Metallographic Cross-sectional Analysis
A number of the non-tested solderball solder joints for each coupon were randomly selected for metallographic cross-sectional analysis. The purpose of the analysis was to determine if any of the solder joints contained the palladium/tin intermetallic (IMC) phase PdSn₄, which has been shown to cause embrittlement and degradation of solder joint integrity [1]. The PdSn₄ IMC structure is a blocky, angular phase in the scanning electron microscopy (SEM) image of a Sample 19 Sn63Pb37 solder joint with arrows indicating the PdSn₄ IMC phase illustrated in Figure 4. The Sn63Pb37 solder joints contained more of the PdSn₄ IMC structure than the SAC305 solder joints. The difference in the resulting microstructure between the solder processes is due to the fact that the higher SAC305 reflow temperature and greater tin composition percentage of the SAC305 alloy allows for better distribution of the palladium within the solder microstructure rather than forming the PdSn₄ IMC phase. The thickest palladium plating (Sample 19) also contained the most PdSn₄ IMC phase. Figure 5 through Figure 16 illustrate typical solder joint microstructures observed for each of the test coupons for both the Sn63Pb37 and SAC305 solder joints. The arrows shown in the Figures point to some of the observed PdSn₄ IMC phase contained in the solder joints.

Figure 4: Sn63Pb37 Test Coupon 19, 1690X Magnification SEM View

Figure 5: Sn63Pb37: Left - Test Coupon 1, Right – Test Coupon 2, 500X Magnification Cross-section Views
Figure 6: Sn63Pb37: Left - Test Coupon 3, Right – Test Coupon 4, 500X Magnification Cross-section Views

Figure 7: Sn63Pb37: Left - Test Coupon 5, Right – Test Coupon 6, 500X Magnification Cross-section Views

Figure 8: Sn63Pb37: Left - Test Coupon 7, Right – Test Coupon 8, 500X Magnification Cross-section Views
Figure 9: Sn63Pb37 Test Coupon 18, 500X Magnification Cross-section View

Figure 10: Sn63Pb37 Test Coupon 19, Left – 200X Magnification, Right - 500X Magnification Cross-section View

Figure 11: SAC305 Left -Test Coupon 1, Right – Test Coupon 2, 500X Magnification Cross-section Views
Figure 15: SAC305 Test Coupon 18, 500X Magnification Cross-section View

Figure 16: SAC305 Test Coupon 19, Left – 200X Magnification, Right - 500X Magnification Cross-section View
Discussion

Figure 17 shows the solderball shear data as a function of palladium plating thickness for both the Sn63Pb37 and SAC305 soldering processes. The error bars in the plot represent one standard deviation. The Sn63Pb37 soldering process shear force values are very consistent with little variation over the range of palladium plating thickness. The SAC305 soldering process shear force values are more variable but greater than the Sn63Pb37 values. A solder joint integrity issue due to PdSn$_4$ IMC phase embrittlement would have revealed itself as a loss of shear force with increasing palladium plating thickness. The metallographic cross-sectional analysis confirms the Figure 17 data as the presence of the PdSn$_4$ IMC phase was not segregated at the solder joint/test coupon pad and was only found in minor concentration levels. The test results recorded in this investigation are in agreement with recently published testing palladium plating reliability results by M. Wolverton [2] and previous Rockwell Collins investigations [3].

![Solderball Shear on ENEPIG Surface Finish](image)

Figure 17: Solderball Shear Force as a Function of Palladium Plating Thickness

Conclusion

The palladium plating thickness range on the test coupons submitted by the IPC 4-14 Plating Processes Subcommittee did not introduce any loss of solder joint integrity for Sn63Pb37 or SAC305 soldering processes as measured by solderball shear testing and metallographic cross-sectional analysis.

References

The tasks identified by a consensus of the IPC 4-14 Plating Processes Subcommittee members to evaluate the suitability of the Electroless Nickel Electroless Palladium Immersion Gold (ENEPIG) plating finish for wire bondability were as follows:

- Round Robin Task Group Members Roles and Responsibilities
- Development of a Wire Bond Test Vehicle
- Commercial ENEPIG Plating Finish Supplier Contributions
- ENEPIG Plating Finish Attributes Selected to Evaluate Wire Bonding
- 1 mil Gold Ball Wire Bonding and Destructive Pull Testing Evaluations per MIL-STD-883

**Round Robin Task Group Member Roles and Responsibilities**
The wire bond test vehicle artwork was supplied by Kulicke and Soffa (K&S). Fabrication of the test vehicles was performed by TTM Technologies, Inc.. Commercial suppliers provided the ENEPIG plating finish test vehicles. St. Jude Medical conducted the auto wire bonding and destructive pull testing evaluations.

**Development of the Wire Bond Test Vehicle**
The artwork for the round robin wire bond test vehicle was provided by K&S. The test vehicle/coupon was a double sided Printed Circuit Board (PCB) with no inner planes. The dimensions of an individual test coupon were 1 inch by 1 inch square and 0.034 inches thick. The coupon was designed for wire bonding to be conducted only on one side of the part. Two levels of wire bond pad surface roughness were selected for the test matrix --- high and low.

FR-4 was the laminate material used for the test vehicle. No solder mask was incorporated. All coupons were fabricated by TTM. A tooling hole was added to each coupon within the panel to provide for a pin #1 location. The wire bonding areas of each coupon were separated into 4 quadrants for wire bonding and accurate destructive pull data tracking (as shown in Figure 1).
Note: The red lines indicate the general wire bond locations and sites used for Destructive Pull Testing (DPT).

**Figure 1. Wire Bond Test Vehicle Showing pin #1, quadrants 1, 2, 3 and 4.**

**Commercial ENEPIG Plating Finish Suppliers**
Following the fabrication of the ENEPIG panels by TTM, the panels containing the wire bond test coupons were shipped to the following commercial ENEPIG finish suppliers who had agreed to participate in the Round Robin testing:

1. Uyemura
2. DOW
3. MacDermid
4. Enthone-Cookson
5. OMG
6. Atotech

The electroless nickel and immersion-gold surface finish thickness metal layers were selected as a control; the electroless palladium thickness and wire bond pad surface roughness were the variables chosen by consensus of the subcommittee for evaluation of the suitability of ENEPIG as a plating finish for gold wire bonding.

The electroless palladium thickness layer varied based upon the following target thicknesses:
   a. 4 micro inches
   b. 8 micro inches
   c. 12 micro inches
   d. 20 micro inches
ENEPIG Plating Finish Attributes Used to Evaluate 1-Mil Gold Wire Bonding

1 mil gold ball auto wire bonding for evaluation of the ENEPIG finish was based upon using the established volume manufacturing wire bond processes used at St. Jude Medical (Scottsdale, AZ). Experienced and certified wire bond associates and technicians performed all wire bond setups, calibrations and conducted the actual wire bonding of the ENEPIG test vehicles.

The wire bond testing was configured as a blind test. The suppliers, plating thicknesses and other properties were unknown before and after actual processing. Twenty-one (21) groups of panels were received with special labeling used for designation of the coupons and wire bonding data collection (Table I).

Development of 1 mil gold wire bond parameters and attributes used for wire bonding onto the coupons with the ENEPIG plating finish was consistent with the same approaches used to establish auto wire bonding parameters for standard electrolytic gold plating finish substrates.

Auto Wire Bond Process Design Point Areas of Focus Included:

- ✔ Preheat Stage Temperature
- ✔ Power /Force /Time
- ✔ Visual Inspection Before and After Wire Bonding
- ✔ Selection of Coupons within the Panels
- ✔ One Set of Parameters to be used for ALL Wire Bond Samples (Standardization Target)
- ✔ No Effort would be Placed into “Making Wires Stick”
- ✔ Wire Bond Equipment was Calibrated Before Processing
- ✔ Certified Wire Bond Operators Used for Wire Bonding
- ✔ 1 mil Diameter Gold Wire Used for Bonding
- ✔ Destructive Pull Testing per MIL-STD-883 Would be Incorporated
- ✔ Pre-conditioning of Test Vehicle Before Wire Bonding
The samples in the package marked “Hand Cut” were not evaluated.

The individual wire bond coupons were immediately labeled with the same designation used for the panel prior to removal of the coupon from the panel and packaging (Table I and Figure 2). To ensure maximum traceability from coupon to coupon and supplier to supplier, a standard label approach was adopted for wire bonding to each test vehicle.

The identical wire bond locations on each coupon were used for wire bonding for each of the 6 different ENEPIG suppliers followed by destructive pull testing. For example, wire bond pad location #1 in the X-direction per Figure 1 was the same location used for all 21 panel groups. Wire bond location #2 corresponded to location number 2 for all 21 panel groups. Panel groups were composed of six (6) Suppliers, four (4) different palladium thicknesses and two (2) levels of surface roughness (Table II).

1 mil Gold Wire Bonding and Destructive Pull Testing Evaluation per MIL-STD-883

The 1 mil gold wire bond parameters developed for the ENEPIG plating finish test vehicles were based upon the same 1 mil gold wire bond approach used in the development of the wire bond parameters designed for production level substrates with gold plating finish.

Manufacturing processes and operations are inherently specific. While automatic wire bonders are similar, they may be different in specific aspects. Wire bond fixtures and capillaries are critical and play a significant role in a successful wire bonding operation. The experience levels of the wire bond operator are also important.
Following removal of the coupons from the panel and marking of the coupons, the parts were baked in a nitrogen oven at 150 ºC [302 ºF] for 4 hours. The parts were then plasma cleaned and wire bonded. 120 wires were placed onto each conditioned coupon. A minimum of 10 each 1 mil gold wires per coupon in the X-direction and 10 wires in the Y-direction were bonded, followed by DPT (see Figure 1).

The 1 mil gold wire bonding parameters selected for bonding on substrates with an electrolytic gold plating finish were modified for the bonding of ENEPIG coupons with the immersion gold finish, as follows:

- Reduction in Power by 7%
- Force was increased by 10%
- Time was increased by 100%
- Stage temperature at the substrate surface was 150 °C ± 5 °C [302 °F ± 9 °F].

MIL-STD 883 destructive pull test requirements for all ENEPIG test vehicles were met (see Figures 3 and 4).

Figure 2. Example of 6 in x 8 in Panel Containing 1 in x 1in ENEPIG Wire Bond Coupons
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Figure 3. Visual Evaluation of Wire Bonding Showing Classical Crescents Resulting in Neck Breaks

(Top) Destructive Pull Test value of 12.5 grams / (Lower) DPT is 5.5 grams

Upon completion of the final wire bonding and submission of data, the plating finish sample thicknesses were obtained and summarized. The results are shown in Table III.
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Figure 4. (Top) Summary of 1 mil Gold Wire ENEPIG Destructive Pull Test (DPT) Results and (Lower) Comparison of X-Y Directional DPT Values
Summary

The tasks identified through consensus of the IPC 4-14 Plating Processes Subcommittee to evaluate the suitability of Electroless Nickel Electroless Palladium Immersion Gold (ENEPIG) as a finish for gold ball wire bond bondability included development of an industry Round Robin with roles and responsibilities, development of a wire bond test vehicle, participation of commercial ENEPIG plating finish suppliers, establishment of ENEPIG plating finish attributes to evaluate the wire bonding and 1 mil gold ball wire bonding destructive pull testing evaluation per MIL-STD883.

The wire bond test vehicle artwork was supplied by Kulicke and Soffa, fabrication of the test vehicle was conducted by TTM Technologies, Inc. and the ENEPIG plating finish was supplied by commercial suppliers, Uyemura, DOW, MacDermid, Enthone-Cookson, OMG and Atotech.

The electroless palladium target thicknesses were 4, 8, 12 and 20 microinches. The nickel and immersion gold thicknesses were not considered variables.

One set of 1 mil gold ball wire bond parameters were selected for testing all six (6) ENEPIG supplier finishes evaluated in this study.

All destructive pull testing results for all 6 commercial ENEPIG plating finish suppliers were in compliance with MIL-STD-883 requirements.
APPENDIX 9

XRF Thickness Measurements of thin Au and Pd (ENEPIG):
Recommendations for Instrumentation (Detectors) and their Limitations

Michael Haller
Chief Operating Officer
Fischer Technology

Table 1 - XRF Detectors and Their Limitations at Typical Count Rates

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<tr>
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<td>&lt; 200 eV</td>
<td>&lt; 150 eV</td>
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- The given limitations in Table 1 for the minimal measurable thickness is based on measurements of a typical sample with 50 nm Au and 100 nm Pd plated on 4 µm Ni, over 30 µm Cu on a substrate of fiberglass-reinforced epoxy resin with Br for the given total intensities. Measuring time 120 s.

- Intensities are given for reference. If lower intensities are realized for a certain XRF setup appropriate adjustments should be made, by either increasing collimator size (while still meeting spot size specifications) or by increasing the measurement time.

- Detector limits are derived from repeatability precision (standard deviation) of the XRF instruments and based on counting statistics which directly relate to total intensity and measurement time. As a general rule, in order to improve the repeatability precision by a factor of 2 an increase in intensity or measurement time of a factor of 4 is required.

- The minimum measurable thickness for proportional counter detectors is limited due to the strong influence of the PCB base material (Br) and Cu thicknesses (background signal and peak overlap).
The use of calibration standards with similar thicknesses to the specified ENEPIG thicknesses which are to be measured is recommended. Tri-layer standards where Au and Pd are plated directly on Ni/Cu/PCB should be used for calibration for Cu thicknesses > 30µm. Tri-layer foil standards where Au and Pd are plated on a Ni-foil should be used if boards with varying Cu-thickness are to be measured. The foils allow for great flexibility since they can be placed on various base materials, therefore achieving optimal accuracy. With decreasing Cu thickness the influence of the PCB material becomes more and more significant and requires careful calibration.

For Cu thickness > 30 µm a combination of a minimum of two calibration standards with approximate thicknesses as below should be used.

- Au/Pd/Ni/Base 50nm/20nm/3µ
- Au/Pd/Ni/Base 50nm/90nm/3µ
- Au/Pd/Ni/Base 50nm/300nm/3µ
- Au/Pd/Ni/Base 10nm/20nm/3µ

For Cu thicknesses < 30 µm a combination of a minimum of two calibration foil standards with approximate thicknesses as below should be used.

- Au/Pd/Ni 60nm/20nm/4µ
- Au/Pd/Ni 60nm/60nm/4µ
- Au/Pd/Ni 50nm/100nm/4µ
- Au/Pd/Ni 50nm/250nm/4µ

To verify accuracy, it is most important after the XRF has been calibrated for the appropriate measurement range, that the instrument will read Au and Pd values of an uncoated board of the measurement sample as statistically zero. Only in this way can one ensure that no systematic offset in the calibration and setup exists. (i.e. measure on uncoated Ni/Cu/PCB or Cu/PCB and record Au and Pd values obtained)

For samples with thin coatings (< 100 nm) and small structures (< 0.2 - 0.3 mm) an XRF instrument with semiconductor detector and special X Ray optics (poly-capillary) is required to achieve sufficient high intensities.

Due to the influence of the base material on the measurements (Br in PCB materials, different composition of the PCB base materials, varying Cu thicknesses) an evaluation software with a peak deconvolution, flexible background correction and the ability to take the P in the Ni, and Pd layer into account, as well utilizing Pd L and Au M emissions is absolutely necessary for the accuracy of the measurements. The influence is especially large for proportional counter instruments, therefore, proportional counter XRF systems are not recommended for Cu thicknesses < 30 µm (~ 1 oz).
Goal: Test of gage capability with respect to repeatability and mean of measurement values for a
given tolerance. Preferably the gage capability is conducted with a calibrated reference standard,
with its reference value approximately in the middle of the tolerance field. At defined
measurement points, the reference standard is to be measured with \( n \geq 25 \) times under repeatable
conditions.

For measurement criteria with Upper and Lower Specification Limits (USL and LSL): \( T = \text{USL-}
\text{LSL} \). For measurement criteria with only a one sided specification limit (USL or LSL): \( T \) does
not exist. In this case the allowable measurement value lies below \( \text{USL} - 4 \cdot s \) or above \( \text{LSL} + 4 \cdot s \).
The value of the reference standard should be within +/- 10% of the USL or LSL.

If Gage capability Indexes are to be calculated, use the following formulas:
The instrument capability is checked thru the \( C_g \) and \( C_{gk} \) values which are defined as:

\[
C_g = \frac{0.2 \cdot T}{6 \cdot s}
\]

\[
C_{gk} = \frac{0.1 \cdot T - \left| \bar{x} - x_m \right|}{3 \cdot s}
\]

where: \( T = \) tolerance, \( s = \) standard deviation, \( x_m = \) mean of standard, \( \bar{x} = \) mean value
measurement
A gage is considered capable if \( C_g \geq 1.33 \) and \( C_{gk} \geq 1.33 \)

Typical measurement data achievable with properly calibrated SDD-Detector XRF

Nominal values of standard measured, Au49nm/Pd100nm/Ni4387nm

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<td>Observation</td>
<td>Au  [nm]</td>
<td>C.O.V. (%)</td>
<td>Measuring time</td>
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**Basic Statistics**

- **Mean:** 48.92 nm  
- **Standard deviation:** 0.286 nm  
- **C.O.V. (%)**: 0.59  
- **Range:** 1.07 nm  
- **Number of readings:** 25  
- **Min. reading:** 48.4 nm  
- **Max. reading:** 49.4 nm  
- **Measuring time:** 120 sec
Type 1 Gage Study for Pd

Gage name: XRF
Date of study:

Reported by:
Tolerance: 100
Misc: ENEPIG-Pd Measurements

Run Chart of Pd

Basic Statistics
Reference 100
Mean 99.68
StdDev 0.837
6 * StdDev (SV) 5.021
Tolerance (Tol) 100

Capability
Cg 3.98
Cgk 3.86

% Var(Repeatability) 5.02%
% Var(Repeatability and Bias) 5.19%

Type 1 Gage Study for Ni

Gage name: XRF
Date of study:

Reported by:
Tolerance: 3000
Misc: ENEPIG-Ni Measurements

Run Chart of Ni

Basic Statistics
Reference 4387
Mean 4388.02
StdDev 5.112
6 * StdDev (SV) 30.670
Tolerance (Tol) 3000

Capability
Cg 19.56
Cgk 19.50

% Var(Repeatability) 1.02%
% Var(Repeatability and Bias) 1.03%