Properties of High P ENIG process on Flexible boards

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Abstract

Because of their numerous functional design and application possibilities, the use of flex PCBs is increasing rapidly. However, this shift poses additional challenges within circuit board fabrication and assembly operations, particularly in terms of the surface finish on the PCB.
In response, to improve the reliability of flex circuit applications, alternative surface finish methods are increasingly being used, such as the electroless nickel/immersion gold (ENIG) process.

To avoid cracking of the nickel during bending, the use of ENIG for flex circuits has typically required a relatively thin electroless nickel deposit of medium phosphorus content. With regard to solderability thin medium P EN layers are potential candidates of causing ‘black pad’ and brittle fracture issues resulting in component fall-off.

In this technical paper the reliability of ENIG with a high-phosphorous electroless nickel layer is compared to a medium-phosphorous electroless nickel deposit. It is looked at the mechanical strength of the solder joints at one hand by means of ball shear test and at the other hand at bending capabilities by using results of bending tests, hardness measurements and SEMs.
In particular Young’s Modulus (stress-to-strain ratio) is evaluated to show a direct relationship between the phosphorus content of the electroless nickel deposit and the ductility of the overall ENIG finish. Electroless nickel deposits of varying phosphorous content and thickness were applied.

The ENIG process with high-phosphorus nickel is shown to offer a more reliable surface finish for flex applications.

Introduction

During the next several years, no other segment of the printed wiring board (PWB) industry is forecasted to grow as rapidly as the flexible circuit segment.

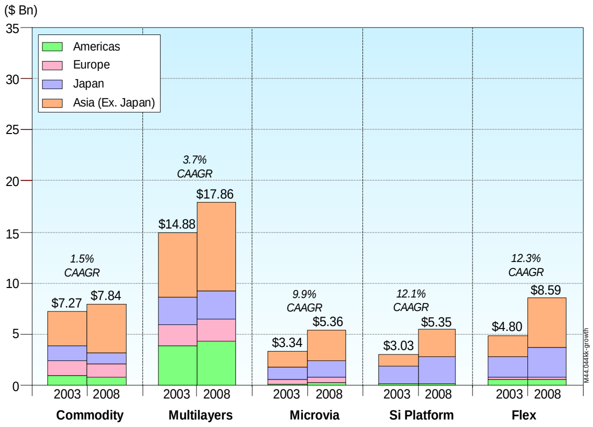


Fig 1 Projected PWB Market Profiles Growth 2003 vs. 2008 (Source: Prismark Partners LLC)

As shown in Figure 1, the flexible printed circuit market is forecasted to grow at an annualised rate of more than 12-percent, reaching a worldwide production value of $8.6 billion by 2008.

As indicated in Figure 2, this increase is concentrated in key areas, mainly attributed to the rapid growth in use of mobile phone, digital camera and LCD technologies.

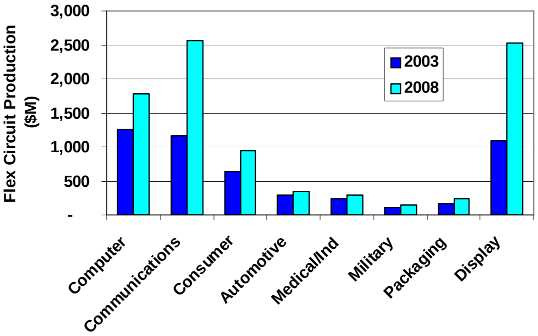


Fig 2 Flexible Circuit Market Growth by Application 2003 vs. 2008 (Source: Prismark Partners LLC)

Only a few years ago, Japan was recognized as the global center for fabrication of flexible circuits.

However, as expected, much of the growth in this market segment will be experienced most dramatically in other parts of Asia.

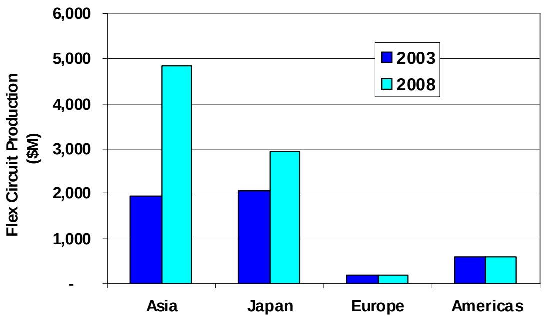


Fig 3 Flexible Circuit Market Growth by Global Area 2003 vs. 2008 (Source: Prismark Partners LLC)

As presented in Figure 3, the value of flexible circuits produced in Asia (countries other than Japan) is predicted to reach nearly five billion dollars by 2008.

Figure 4 presents a prediction of the expected flexible circuit market growth according to the type of circuit board.

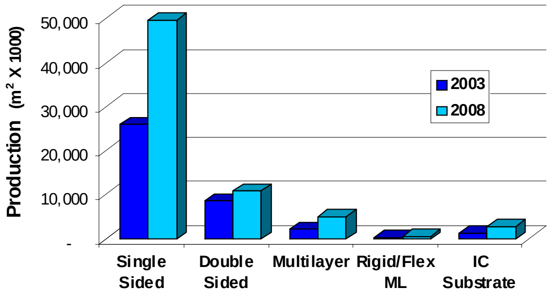


Fig 4 Flexible circuit market by type of material (Source: Prismark Partners LCC)

As shown, single-sided flexible material will continue to be the dominant format and will increase in terms of market share.

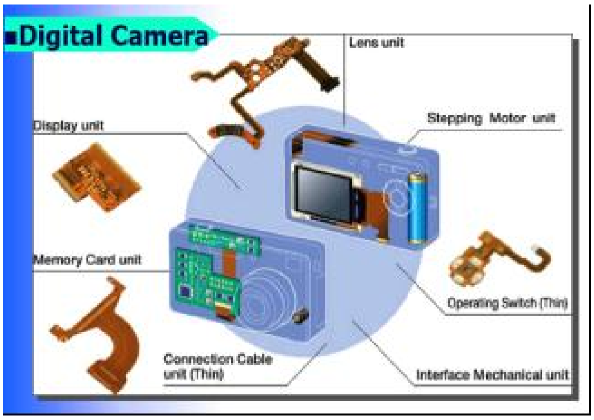


Fig 5 End-use applications for flexible and rigid-flex circuitries (Source: Mektec)

Surface Finish Alternatives for Flexible Circuit Applications

Currently, a variety of methods exist for surface finish of flexible circuits.

Among these are:

- Electrolytic Tin/Lead
- Electrolytic Tin
- Electrolytic Nickel/Gold
- Immersion Silver
- Immersion Tin
- Electroless Nickel/Immersion Gold
- Organic Solderability Preservative

There is no single deposit that provides the perfect surface finish, which explains the existence of these various alternatives.

For example, electrolytic nickel/gold is most predominant in flexible circuit applications where metallic surface finishes are used.

However, as with any electrodeposited metal, there are problems with surface distribution and plating in fine-line dimensions.

Likewise, OSPs are simple to use from a fabrication standpoint, although these coatings do not allow wire bonding and their ability to withstand multiple thermal excursions during component assembly is limited.

As a result, OEMs are frequently seeking ways to improve the reliability of the surface finish while reducing costs.

The electroless nickel / immersion gold (ENIG) process has been used for more than 20 years in the PWB industry.

As a finish, ENIG is now receiving increased attention because it meets requirements for Pb-free assembly while offering a coplanar surface that is both solderable and aluminum-wire bondable. ENIG is also well suited for hot bar soldering and anisotropic conductive film (ACF) bonding.

High-Phosphorous Electroless Nickel / Immersion Gold

In the application of electroless nickel, the nickel is commonly co-deposited with phosphorus.

Most ENIG processes currently used for circuit applications create a nickel deposit with medium-phosphorus content, in the range of 7-9 percent by weight.
In recent years there has been a gradual but consistent shift to the use of high-phosphorous electroless nickel/immersion gold (HP-ENIG) as a final finish. This acceptance is particularly evident in the telecommunications industry, which is experiencing a significant increase in the use of flexible circuits, as previously mentioned.

The immersion gold step of any ENIG process relies on the exchange of nickel ions for gold, which is essentially a corrosion action.
To compensate for the lower ductility of medium-P electroless nickel deposits, some fabricators of flexible PWBs finished with ENIG deposit at relatively low nickel thickness (2-3 microns) in comparison to deposits on standard rigid materials.

This reduced thickness has been necessary to avoid nickel cracking during normal bending of the flex circuit. However, because of the thin nickel layer, corrosion from the immersion gold step can frequently cause problems with solder joint integrity after assembly, a result commonly known as the “Black Pad” defect.

Although a thicker nickel deposit may eliminate the black pad issue, it compounds the problem of nickel cracking during bending of the flexible circuits.

This cracking is directly related to the nickel deposit properties, such as ductility and internal stress, which are primarily influenced by the composition of the nickel solution, the solution age as defined by number of metal turnovers (MTO) and the phosphorous content.

HP-ENIG involves the use of an electroless nickel deposit containing 10-13 percent phosphorus by weight. Because of the higher phosphorus content in the nickel deposit, it offers superior corrosion resistance compared to that of a low- or medium-phosphorous process.

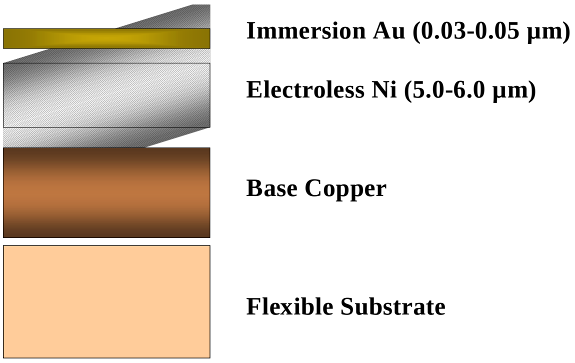


Fig 6 Layer build up for the high Phosphorous ENIG Process

Figure 6 illustrates the build-up of the electroless nickel and immersion gold layers on the base copper of the flexible circuit.

Table 1 presents information regarding the HP-ENIG process sequence and key operating parameters.

Table 1. HP-ENIG Process Sequence and Parameters

Process Step	**Treatment Time (min)**	**Treatment Temp (ºC)**
Clean	3-6	35-45
Micro etch	1 - 2	25-35
Acid Dip	>3	Ambient
Activate	1-3	20-25
Electroless Nickel	35-45	80-90
Immersion Gold	10-12	80-85

If it is accepted that a corrosion-resistant electroless nickel layer undergoes less attack by the immersion gold reaction, the resultant gold thickness will be lower in comparison to a nickel layer with less corrosion-resistance, providing the immersion gold solution parameters are equal.

This condition was previously observed[1] on a nickel deposit of 8.0-percent phosphorus that achieved a gold thickness of 0.08 µm, while a nickel layer of 11.2-percent phosphorus yielded a gold thickness of only 0.05 µm.
Although the tool used to measure the gold thickness is also limited in accuracy (typically +/- 0.01µm or greater) depending on pad size and collimator been used, it does suggest that thicker gold will be deposited on the nickel layer that is more readily attacked.

Ball Shear Tests for Solder Joint Integrity Evaluation

BGA substrates were prepared by systematically varying ENIG layer thickness and bulk phosphorus content.

The layers were characterized by X-ray fluorescence (XRF) and scanning electron microscope (SEM) in a cross-section.

Manufacturers and assemblers of the BGA-laminate typically utilize ball shear tests. Because the individual pads are solder mask-defined (SMD), shown in Figure 7, the chance of a pad pull-out is less in comparison to a non-solder mask-defined (NSMD) pad, as shown in Figure 8, since SMD pads are typically found on the board side.

Higher strength against pad pull-out will force the fracture to occur at the metallic layer, the IMC, the solder or any interface in between.

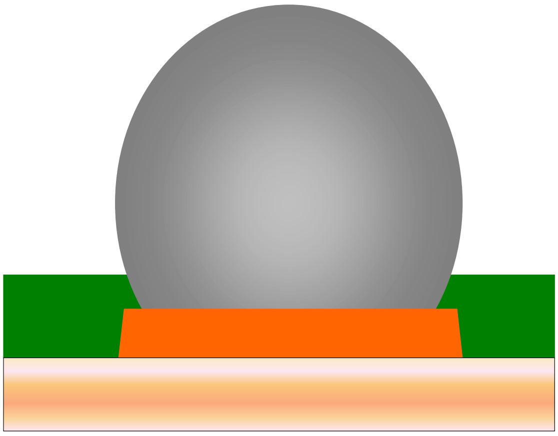


Fig. 7 Solder Mask Defined pad (SMD)

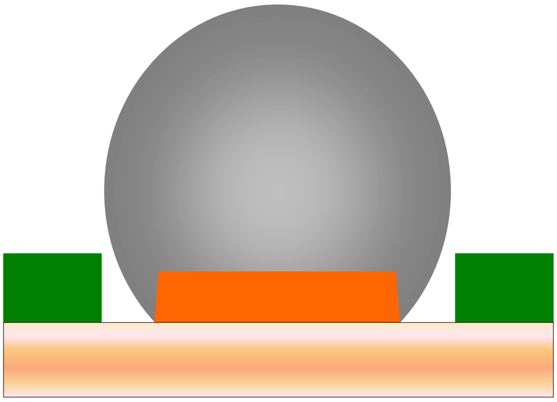


Fig. 8 Non Solder Mask Defined pad (NSMD)

A BGA solder ball (760 µm diameter) is soldered onto the SMD pad (600 µm diameter opening) or NSMD pad (600 µm diameter) and sheared off using a DAGE PC 2400 shear tester. Force/length diagrams are then plotted as presented in Figure 9. Diagrams with a steep descent after the maximum height represent “brittle” interfacial fracture, while a gradual descent represents “ductile” plastic deformation of the solder.

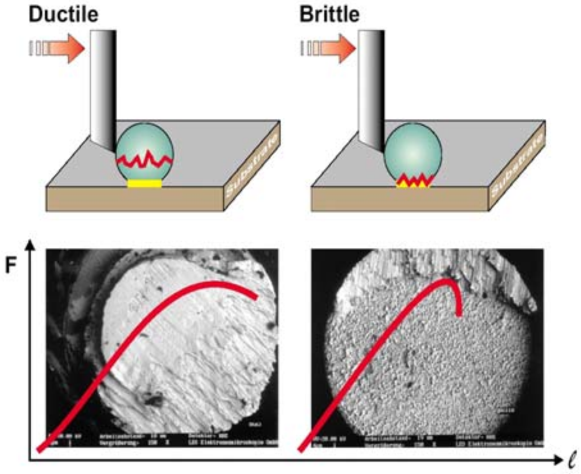
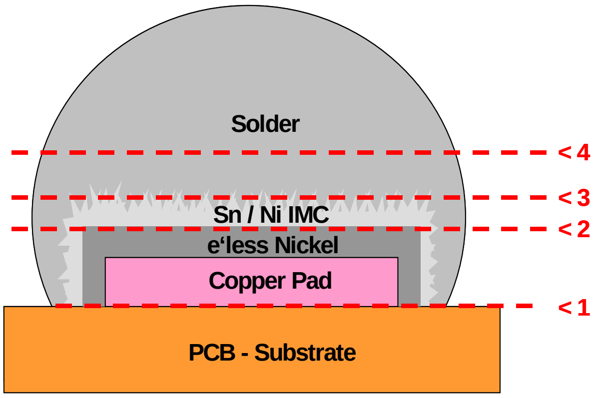


Fig. 9 Schematic diagrams of ball shear test and SEM micrographs of ductile and brittle fracture.

The surface of the remaining pad is analyzed (Figure 10) and the fracture is classified as “ductile” (fracture in the solder/pad pull-out) or “brittle” (fracture at the IMC).

Fracture modes 1 and 4 represent ductile fractures, where the fracture occurs by the shearing of the BGA pad from the PCB (Mode 1) or the fracture occurs within the solder (Mode 4). Brittle fractures occur between the electroless nickel layer and the Ni/Sn IMC (Mode 2) and at the interface between solder and Ni/Sn IMC (Mode 3).



Fracture mode:

1 pad pull out
2 between nickel and Sn / Ni IMC
3 between Sn / Ni IMC and solder
4 with in the solder

Fig. 10 Classification of fracture modes 1-4 after ball shear test.

In order to clarify the solder joint integrity for the tested ENIG layer systems, samples were prepared as follows:

Electroless nickel at 3 µm to 6 µm thickness
- Medium phosphorous content in the bulk nickel layer at 7-9 wt %
- High phosphorus content in the bulk nickel layer at 9.5 -13 wt %.

Immersion gold at 0.05µm to 0.15µm thickness

Samples were assembled with Sn/Pb solder balls (760-µm diameter) using Litton Kester 950 E3.5 (type F-SW33) as flux, in convection reflow oven.

As stated before, high gold thickness was not possible with high P content nickel without operating outside normal conditions.

Ball Shear / 600 µm SMD Pad

Figure 11 shows a series of ball shear results with 600-µm SMD pads. In this example, two ENIG systems are compared, one with a bulk phosphorus content of 7-9%, the other with 9.5-13% P.

The plated electroless nickel and immersion gold thicknesses were varied from 3.2 µm to 6.1 µm and from 0.06 µm to 0.15 µm, respectively. Solder balls were attached to samples directly after plating.

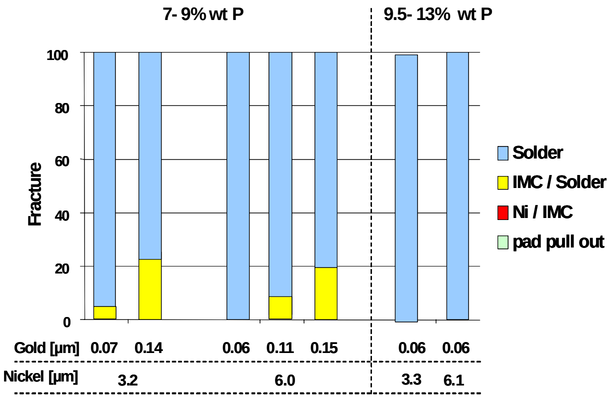


Fig. 11: Detected fracture modes of ENIG layers on 600-µm SMD pads.

Samples with a low gold thickness (0.06 µm and 0.07 µm) exhibit fracture primarily within the solder (Mode 4). However at low nickel thickness (3.2 µm and 3.3 µm) in 7-9 % wt P, a low number of Mode 3 fractures occurred, which was not the case for the high-P content nickel.

Samples with low nickel thickness (3.2 µm and 3.3 µm) and/or samples with high nickel thickness (6.0 µm) with high gold thickness (>0.14 µm) show the highest number of fractures at the interface between Ni/Sn IMC (Mode 3).

In the case of low nickel thickness (3.3 µm) with 9.5-13% P, samples exhibited a ductile fracture mode within the solder (Mode 4).

Ball Shear / 600 µm NSMD Pads

A series of ball shear with 600 µm NSMD pads is shown in Figure 7.As an example, two ENIG systems are compared: one with a bulk P-content of 7-9%, the other with 9.5-13% P.

The plated electroless nickel and immersion gold thicknesses varied from 3.2 µm to 6.1 µm and from 0.06 µm to 0.15 µm, respectively. Due to the higher corrosion resistance of the high-P, creating a thicker gold layer was not possible without operating the system outside its normal operating parameters.

Solder balls were attached to samples directly after plating.

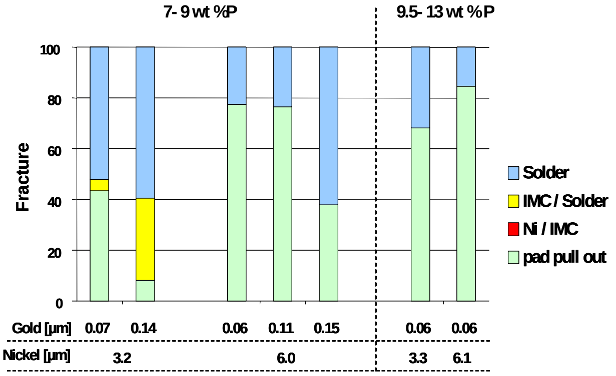


Fig. 7 Detected fracture modes of ENIG layers on 600-µm NSMD pads.

Only samples with P-content of 7-9% in the nickel layer and a nickel thickness of 3.2 µm showed brittle fracture (Mode 3) at the interface between the Ni/Sn IMC and the solder, regardless of the gold thickness.

Throughout the testing only 600-µm NSMD pads showed fractures of the Mode 1 type with pad pull-out. Nickel layers with 7-9% P, 6.0-µm thickness and 0.15-µm gold thickness exhibited the lowest frequency of pad pull-out, less than 40 percent.
By comparison, high-P nickel layers with 6.1-µm thickness and 0.06-µm gold thickness resulted in more than 80 percent pad pull-out.

All samples with an electroless nickel P-content of 9.5-13% (regardless of tested nickel thickness) and 0.06µm gold showed no brittle fractures regardless of the type pad formation (SMD or NSMD).

In the case of NSMD pads, even with the thicker immersion gold there were no brittle facture joints. This fact is attributed to the additional anchorage effect of the solder around the pad providing the joint greater bond strength.

The comparison showed that a low thickness medium-phosphorous electroless nickel layer as commonly plated on flex PCBs bears a higher potential risk for the occurrence of brittle fracture.

Flexible Circuit Bending Test Methods

Because flexible circuit designs are often unique for each application, the original equipment manufacturer (OEM) defines their specific performance criteria.

However, such specifications are typically based on accepted industry standards. The primary standard regarding performance testing for flexible circuits is IPC-6013A (Qualification and Performance Specification for Flexible Printed Boards).

Within this standard, Section 3.6 specifies “Physical Requirements” for such circuits, including “Bending Flexibility”.

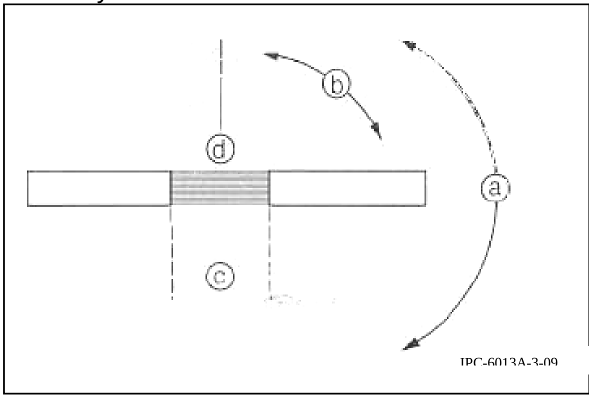


Fig 12 Bending test according to IPC-6013A

Figure 12 shows the basic premise for the 90º and 180º bend tests according to IPC-6013A, where direction of bend (a), degree of bend (b), number of bend cycles and the bend radius (d) are required.

Guidelines for determining the minimum bend radius (d) are set forth in IPC-2223-A (Sectional Design Standard for Flexible Printed Boards).

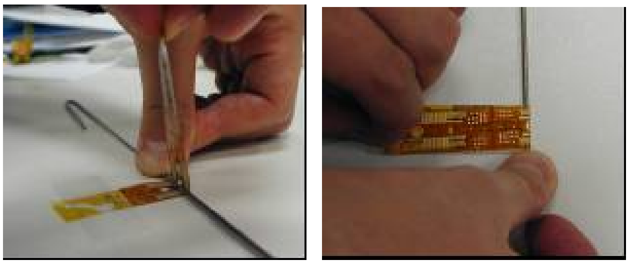


Fig 13 Performing the 90º bend test (left) and 180º bend test (right)

Figure 13 illustrates the performance of the 90º and 180º bend tests in practice for a selected bend radius.

Other dynamic testing is employed within the flex circuit industry and has been previously documented[3]. An illustration of each of these tests is shown in Figure 14.

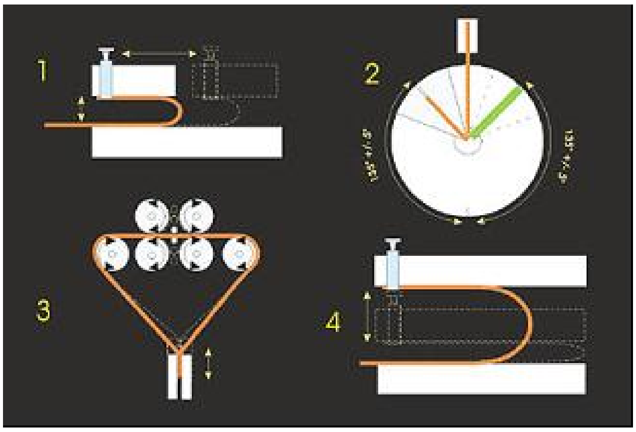


Fig 14 Illustration of various bending tests for flexible circuits: (1) Cyclical Rolling Flex Test, (2) Cyclical Bend Test, (3) Fatigue Ductility Flex Test and (4) Collapsing Radius Test

One flexible circuit fabricator has adopted a relatively simple method for testing the ENIG surface finish on flexible substrates.

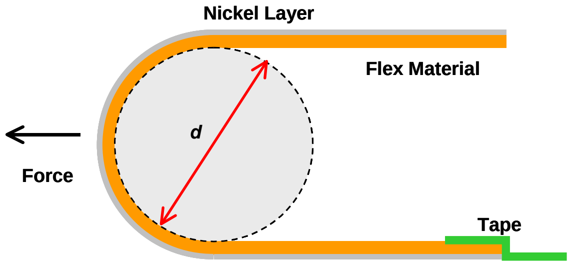


Fig 15 Wire pull/bend test for flexible circuits

The test involves fixing one end of the flexible circuit coupon and pulling a wire of known diameter (*d*) through the length of the sample as shown in Figure 15.

By simulating both a bending and "curling" effect, this test was considered to be a more demanding and accurate measure of the ductility required of the deposit.

Figure 16 shows the performance of this test in practice using a 2-mm wire to roll the bend through the flexible material.

Fig 16 Securing the flex circuit sample (left) and performing the 2-mm wire pull/bend test (right)

Bending Test Results

Wire pull/bend tests were performed on flexible circuits with surface finishes of medium-phosphorus and high-phosphorus ENIG.

Using a 2-mm wire, tests were conducted on both deposits with nickel deposit thicknesses of 1µm, 3µm and 5µm. In all cases, the gold thickness was held constant at 0.05 µm. All conductor widths were 0.5 mm.

Figure 17 shows the results of the 2-mm wire pull/bend test as performed on the ENIG deposit of medium phosphorus content.

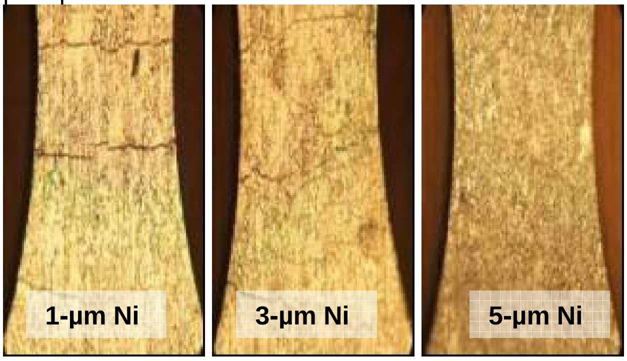


Fig 17 Medium-phosphorus ENIG deposit after 2-mm wire pull/bend test showing evidence of surface cracks

Cracks in the deposit are readily noticeable at a nickel thickness of 3 µm and the defect is exacerbated at the 5-µm nickel thickness, as seen in Figure 18, which presents a sample cross-section that clearly shows the magnitude of the defect.

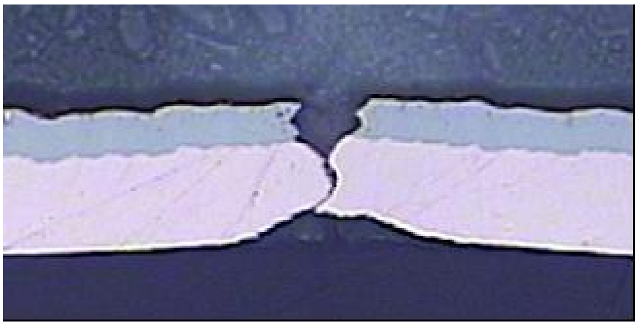


Fig 18 Cross section of 5-µm medium-phosphorus ENIG deposit on copper following 2-mm wire pull/bend test

As noted previously, it is for this reason that fabricators of flexible circuits will compensate for the lack of ductility in this type of deposit by reducing the electroless nickel thickness.

Unfortunately, such a reaction increases the probability of "black pad" effect as a result of corrosion by the immersion gold and the tendency to of brittle fracture as shown before.

In contrast, the wire pull/bend test results for the HP-ENIG deposit resulted in no discernible cracks at any thickness tested, as shown in Figures 19 and 20. The preferred minimum electroless nickel deposit of 5-µm can thus be applied without concern for surface cracking.

This is a significant advantage of the HP-ENIG deposit since it plays such a major role in the prevention of copper attack by the immersion gold step (and the resultant Black Pad effect).

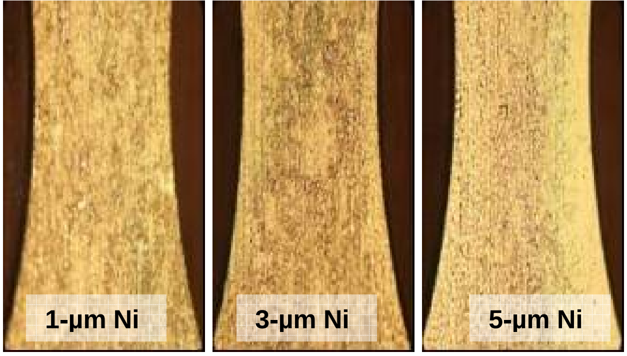


Fig 19 High-phosphorus ENIG deposit after 2-mm wire pull/bend test showing defect-free surface

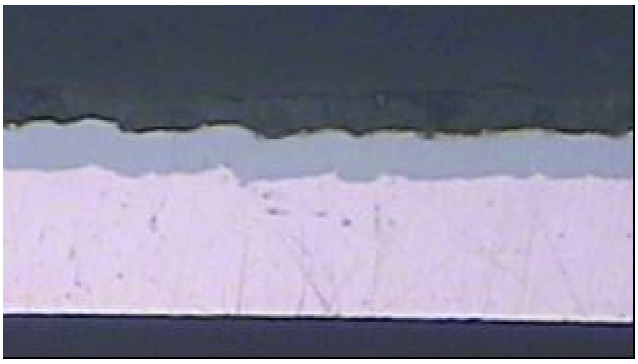


Fig 20 Cross section of 5-µm high-phosphorus ENIG deposit on copper following 2-mm wire pull/bend test

Ductility and Stress Analysis

Ductility is a measurement of the extent that a material can be “plastically” deformed before fracture occurs.

$$\%EL = \frac{l_f - l_0}{l_0} \times 100\% = \varepsilon_f \times 100\% \qquad \%RA = \frac{A_0 - A_f}{A_0} \times 100\%$$

It is commonly expressed as percent elongation (*%EL*) or percent reduction in area (*%RA*):

In flexible circuit applications, ductility is an important property of the ENIG deposit. The key to ductility is to maintain low internal stress to account for the required bending.

As shown in Figure 21, electroless nickel deposits with medium phosphorus content (7 - 9 percent) exhibited internal stress in the tensile range of approximately 60 N/mm².
Conversely, the high-phosphorus (10-12 percent) nickel deposit displayed internal stresses of a compressive nature and of lower values in comparison to the medium-phosphorus deposit.

In similar manner, the effect of nickel thickness was examined for medium- and high-phosphorus deposits.

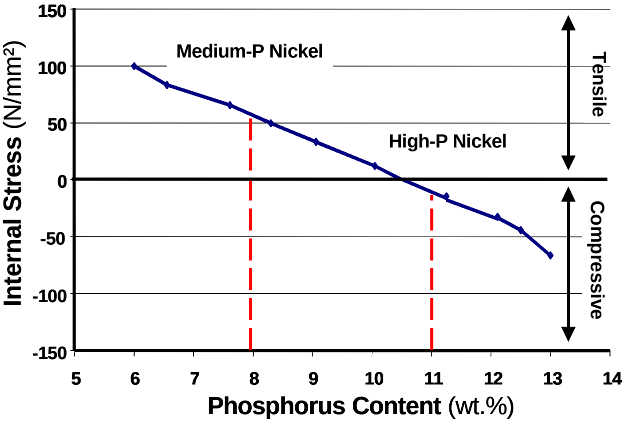


Fig 21 Internal stress of 5-µm electroless nickel deposits with varying degree of phosphorus content.

Figures 22 and 23 show the results of these tests for different nickel thicknesses and electroless nickel solution metal turnovers (MTO).

Comparing the two charts, it can be observed that the internal stress of the high-phosphorus deposit is lower and impacted to a lesser degree by both metal thickness and MTO.

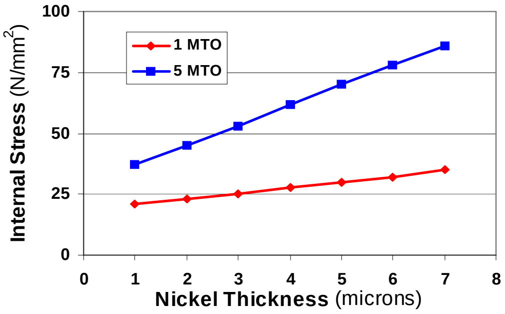


Fig 22 Relationship of internal stress and nickel thickness for medium-P nickel deposit

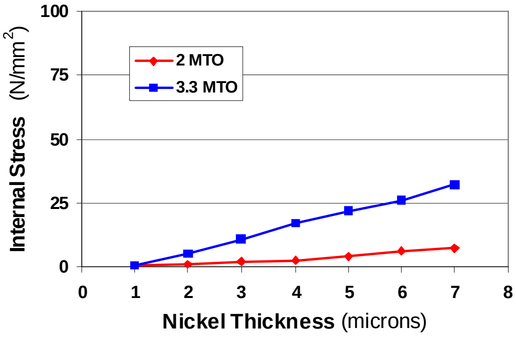


Fig 23 Relationship of internal stress and nickel thickness for high-P nickel deposit

Elasticity

For the description of the elastic properties of linear objects like wires, rods, columns that are either stretched or compressed, a convenient parameter of the material is the Young's Modulus.[4]

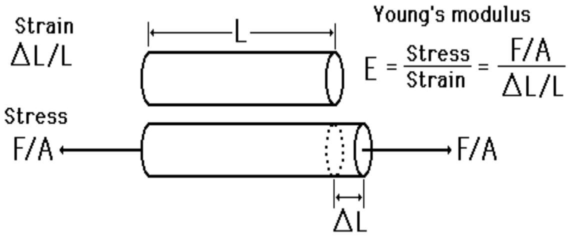
Young's modulus can be used to predict the elongation or compression of an object as long as the stress is less than the yield strength of the material.
Young's Modulus (*E*) is defined as the ratio of stress to strain:

$$E = \frac{[\text{stress}]}{[\text{strain}]},$$

This ratio can also be expressed as:

$$E = \frac{L_0}{\Delta L}\frac{F}{A},$$

where L_0 is the equilibrium length, ΔL is the length change under the applied stress, *F* is the force applied, and *A* is the area over which the force is applied.



Measured in Pascal or Newton per square meter (N/m²), Young's Modulus provides a relatively simple method for comparing the elastic properties of various materials.

As a means of comparing the elasticity of HP-ENIG deposit versus an ENIG deposit of medium phosphorus content, the Young's modulus for each case was measured.

A Fisherscope H100C was used to measure the resultant stress and strain.

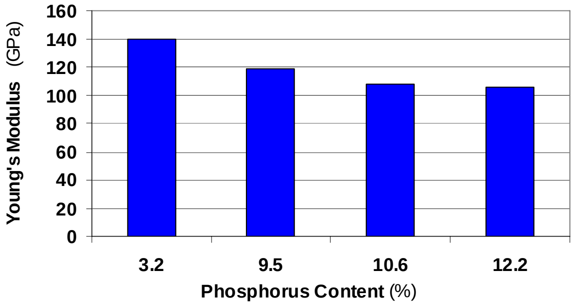


Fig 24 Comparison of Young's Modulus vs. phosphorus content of different electroless nickel deposits

Figure 24 shows a comparison of Young's Modulus values for electroless nickel deposits of varying phosphorus content.

As shown, the deposits of higher phosphorus content exhibit a lower Young's Modulus, indicating a higher degree of elasticity. The Young's Modulus of the HP-ENIG is within the range of that for electrolytically deposited copper, which is typically 70-110 Gpa.

Hardness

Hardness is defined as the measure of a material's ability to withstand indentation.

For measurements of microhardness, the Vickers unit of hardness is often used and results can be directly related to the strength of the material.
The following table compares the Vickers hardness values for electroless nickel deposits of varying phosphorus content.
As shown, no clear trend was observed regarding the relationship between phosphorus content of the nickel and the hardness of the deposit.

Comparison of Hardness for Electroless Nickel Deposits of Varying Phosphorus Content

Electroless Nickel Process Solution	**Phosphorus Content** (%)	**Vickers Hardness**
Low P	3.2	1026
Medium P	9.5	760
Medium-High P	10.6	1040
High P	12.2	802

Conclusion

Based on the investigations performed in this evaluation, the following conclusions are offered:

1. OEMs exert a major influence in determining the application requirements for flexible circuits. As such, design and functionality are often not assigned to a common industry standard.

2. From the standpoint of solder joint integrity the investigations have shown that the high-phosphorous deposit exhibits greater ductility and is less prone to brittle fracture than that obtained using a medium-P process.

3. Varying the content of co-deposited phosphorus directly influences the inherent stress in the electroless nickel deposit. The internal stress within the nickel deposit will shift from the tensile range at medium phosphorus content (7-9%) to the compressive range at 10-13% phosphorus. Furthermore, at the higher phosphorus content, the internal stress of the nickel is less affected by changes in deposit thickness and plating solution age.

4. Increasing the content of co-deposited phosphorus can improve the elasticity of the nickel deposit as measured by Young's Modulus. A more elastic (i.e. lower Young's Modulus) response can be achieved with the high-phosphorus electroless nickel deposit.

5. For all thicknesses examined, the HP-ENIG deposit showed superior performance in the 2-mm wire pull/bend test for flexible circuit applications in comparison to a medium-phosphorus deposit.

In summary, results of solder joint integrity investigation, internal stress analysis, measurement of elasticity and practical bending tests indicate that the HP-ENIG process is well suited for applications involving flexible circuitry.

Because of its (1) improved resistance to corrosion from the immersion gold step, (2) better solder joint integrity, (3) lower and compressive internal stress and (4) higher ductility, the HP-ENIG deposit was determined to be capable of withstanding more intensive flexural testing than conventional ENIG processes with nickel deposits of medium phosphorus content.

Further investigations are necessary and will be performed to fully assess the impact of different flexible circuit construction and design on testing results.

References

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