

A Novel Electroless Nickel Immersion Gold (ENIG) Surface Finish for Robust Solder Joints and Better Reliability of Electronic Assemblies

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Abstract

Conventional Electroless Nickel/Immersion Gold (ENIG) currently available in the market is prone to brittle solder joints failures. Due to these reasons, there are field failures and reliability concerns of electronic assemblies - component disconnection which lead to overall malfunction of electronic assemblies. The novel ENIG achieves robust solder joints and provides improved quality and reliability of electronic assembly. Also, it uses cyanide-free chemistry for the immersion gold process making it eco-friendly. This allows manufacturers to consume eco-friendly product while avoiding major field failures and resulting consequences.

With conventional ENIG, the immersion gold process operates on galvanic displacement process (displacing Ni atoms by Au atoms). If not controlled properly, the displacement can be aggressive leading to Ni corrosion, commonly known as “black pad” or hyper-corrosion of Ni. This leads to brittle solder joints failures. Also, during the reflow process the gold layer dissolves into solder and intermetallics form between tin and nickel. In conventional ENIG intermetallics, too much nickel diffuses into tin leaving behind soft Ni₃P layer at the interface. This soft layer is responsible for brittle solder joint failures. The novel ENIG employs an interfacial engineering approach which leads to 10X corrosion resistance of Ni surface helping to prevent black pad/hyper-corrosion. Also, it creates a barrier for Ni atoms to diffuse too much in tin forming distinct-thin intermetallics which are robust and eliminates brittle solder joints failures. Ball Shear and Ball Pull tests (Industry Standard Based testing: JESD22-B115 and JESD22-B117) have been conducted after (1, 3 and 6) reflow cycles during the soldering process. Also, as a part of simulating aging and evaluating long term reliability of solder joints/electronic assemblies, samples were subjected to 150°C for 500 hours and 1000 hours before conducting Ball shear test and Ball Pull test. The novel ENIG board surface finish achieves robust solder joints for better reliability of electronic assemblies.

Introduction

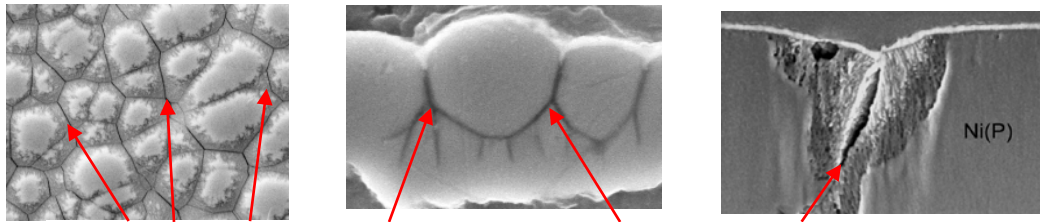
Electroless Nickel Immersion Gold (ENIG) is widely used throughout the electronic industry as a solderable surface finish. With continued circuit feature miniaturization, along with the need for maintaining signal attenuation, copper dissolution into solder joints is a critical issue. Barriers which prevent dissolution of copper into solder joints is very crucial. The principal benefit of ENIG is the role of its nickel layer, which acts as a barrier layer to the dissolution of the underlying copper into solder joints. The ENIG process includes an application of an electroless nickel-phosphorous layer followed by an immersion gold layer. The immersion gold process provides a protective barrier to passivation of the nickel surface. During subsequent reflow soldering processes, the gold layer is dissolved into molten solder and the intermetallic forms the bond between Ni-P atoms and tin atoms of the solder [1].

Currently available ENIG is prone to black-pad defects, a failure associated with a poorly formed joint at the solder/nickel-phosphorous interface, which lead to solder de-wetting and failures. Root causes of black-pad defects are identified as hyper corrosion activity of the immersion gold process on the nickel surface. Galvanic hyper-corrosion occurs between gold and nickel atoms resulting in nickel atom depletion and enrichment of phosphorous atoms in the localized area [2,3]. In the case of hyper corrosion, due to depletion of nickel atoms in the localized area, the intermetallic is not allowed to form and wetting of the surface (solder) does not occur. This leads to de-wetting, a solid-to-liquid bond failure, of the solder and interfacial fracture [2].

Moreover, the solder joints involving ENIG lead to brittle failures. Even without the hyper-corrosion between gold and nickel atoms, just the displacement of nickel atoms situated at the top layer(s) leads to enrichment of phosphorous atoms in localized regions. This phosphorous rich intermetallic (larger Ni₃P and Ni₃SnP phases) layer is responsible for brittle failures at the solder joints. Also, the presence of black pad or hyper-corrosion make the solder joints more prone to brittle failures [1].

The immersion gold process is a galvanic displacement in which gold atoms replace nickel atoms. This process is self-limiting in which once the surface is completely covered by gold atoms the displacement reaction stops. In the case of the Ni(P) layer of electroless nickel plating there are boundaries and crevices on the surface (as seen in the images below). If the boundary or crevice is deep, the supply of gold atoms is slowed down which creates a lack of concentration of gold atoms in crevices compared to the plating bath during the immersion gold process. Hence, the galvanic cell, 2 different metals

connected by a salt bridge, is set up between crevice and surface, leading to hyper active corrosion reaction (black-pad defect) at the crevice [3]. Also, sometimes crevices can be so deep that it can penetrate through the thickness of the electroless nickel coating. This results in coating porosity which degrades solderability and can allow copper migration (Figure 1).



Nickel Intergranular Boundaries Deep Crevices at Intergranular

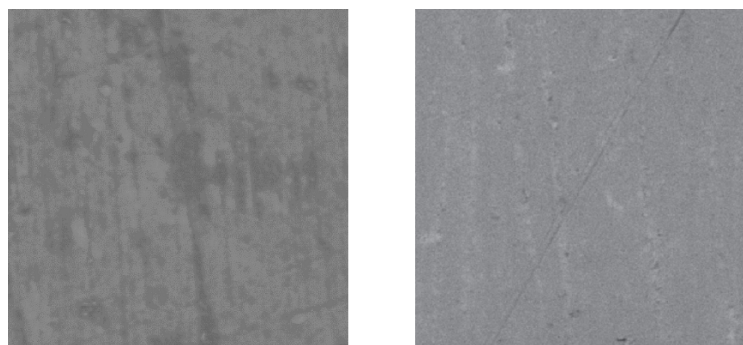
Figure 1: SEM micrograph showing corrosion assisted black pad (surface cracking)

It is essential to design a chemistry and process to curb the hyper-corrosion reaction at the Ni-P surface for a robust ENIG surface finish. Also, solder joints comprised of intermetallic compounds of nickel-tin can be regarded as prone to brittle solder joint failures depending on ENIG chemistry [1]. The newly developed novel ENIG includes a barrier layer at the Ni-P and gold interface that results in very high corrosion resistance (elimination of black-pad defects) and robust solder joints which show the desired ductile behavior and are not prone to brittle failures. An interfacial nano-engineering approach (inclusion of barrier layer) had been used to successfully eliminate black pad and achieve robust solder joints. The novel immersion gold chemistry is also cyanide-free (eco-friendly) unlike most ENIG finishes currently available in the market.

Analysis of nano-engineered sample (Novel ENIG) compared with conventional ENIG sample

SEM Imaging

The microstructure of Ni-P surface and nano-engineered Ni-P surfaces were evaluated by Scanning Electron Microscopy (SEM). The goal of the analysis was to evaluate the smoothening of the microstructure after nano-engineering the Ni-P surface and assessing each surface treatment. SEM imaging was conducted on the Ni-P plated sample and nano-engineered Ni-P sample (Novel ENIG). The SEM images (Figure 2) show Ni-P surface and nano-engineered Ni-P surface (Novel ENIG). As seen in the SEM images the bare Ni-P surface shows multiple pits and black regions which are very non-uniform. However, the nano-engineered (inclusion of barrier layer) Ni-P surface (Novel ENIG) looks very smooth and uniform without showing any black regions or pitting.



**SEM Image (5000X):
Ni-P surface (ENIG)**

**SEM Image (5000X):
Nano-engineered Ni-P**

Figure 2: SEM imaging of Ni-P surface (ENIG and Novel ENIG)

Profilometer analysis of electroless Ni-P surface

Surface roughness measurement was conducted to measure and evaluate surface evenness (unevenness) using the profilometer. Profilometer analysis provided a quantitative measure of surface roughness (Ra and RMS) and helped evaluate presence of deep crevices and intergranular boundaries. Both nano-engineered Ni-P surface (Novel ENIG) and bare Ni-P surface (conventional ENIG) were evaluated using the profilometer.

Observations: Profilometer analysis of conventional (ENIG) sample (bare): Electroless Ni-P layer on Cu surface - Surface roughness of conventional Ni-P sample (Figure 3) was Ra of 1000 Å and RMS of 1244.794.

Profilometer analysis of nano-engineered Ni-P sample (Novel ENIG) with inclusion of nano-engineering on electroless Ni-P layer of Cu surface led to the surface roughness of the nano-engineered sample (Figure 4) of Ra of 480 Å and RMS of 541.

As deduced from this analysis, the nano-engineered sample surface shows much smoother topography compared to the conventional sample (much lower surface roughness Ra and RMS for nano-engineered sample compared to conventional sample). Analysis of the conventional sample also shows the presence of intergranular deep crevices. This analysis suggests that nano-engineering (inclusion of barrier layer) on Ni-P layer helps smoothen the topography and shield deep crevices from potential attack of gold during the immersion gold process – a primary root cause of black-pad defects.

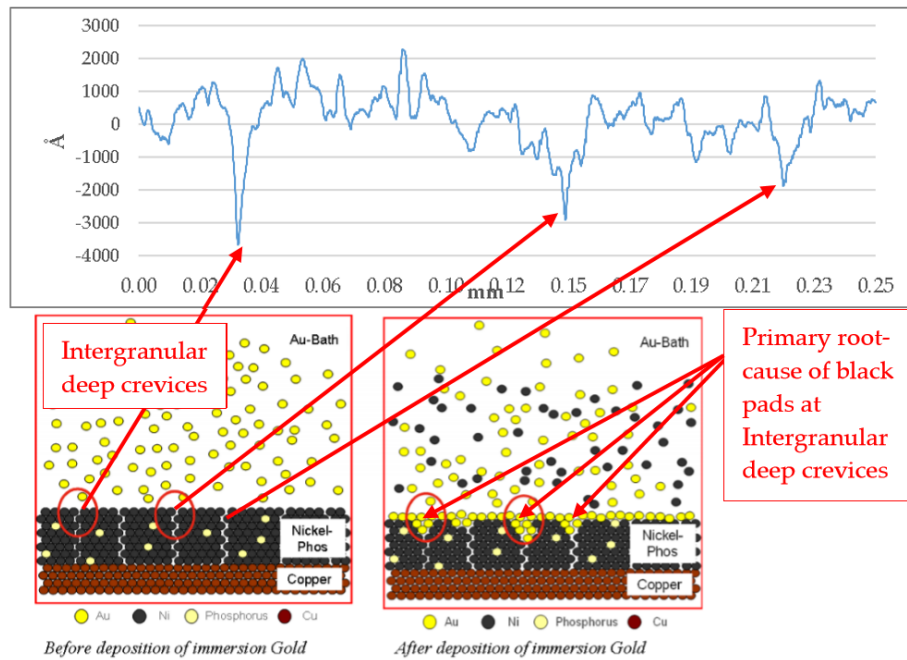


Figure 3: Profilometer analysis of conventional Ni-P surface (ENIG) and illustration of immersion gold reaction on electroless Ni-P layer [4]

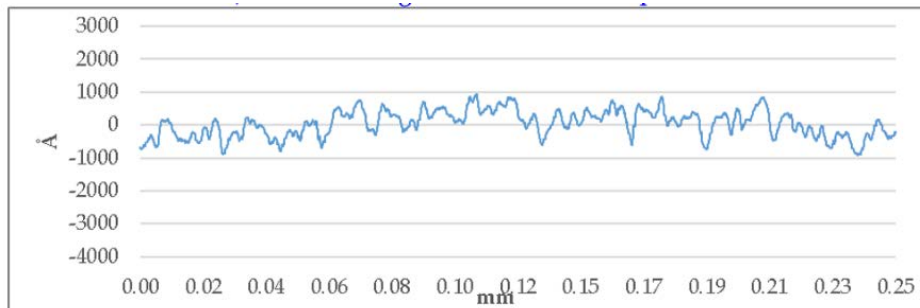


Figure 4: Profilometer analysis of nano-engineered Ni-P surface (Novel ENIG)

Electrochemical corrosion tests:

A potentiodynamic polarization technique using an electrochemical set-up was used to measure corrosion resistance quantitatively for the Ni-P surface. Electrochemical polarization testing was performed by recording anodic and cathodic potentiodynamic polarization curves. Investigations such as passivation tendencies of nano-engineering on Ni-P was assessed and their effectiveness compared to untreated conventional Ni-P samples. These measurements were used to determine corrosion characteristics of the nano-engineered (novel ENIG) and untreated Ni-P samples in aqueous environments such as E_{corr} and i_{corr} (corrosion potential and corrosion current). These characteristics were compared between nano-engineered (novel ENIG) samples and the untreated sample (conventional ENIG) and evaluated based on the corrosion inhibition efficiency.

Corrosion parameters of nano-engineered (novel ENIG) and untreated Ni-P samples were evaluated using the potentiodynamic polarization test in 7% NaCl solution. The test was conducted by sweeping the potential from -400 to +200 mV versus open circuit potential at a scan rate of about 1 mV/sec using the potentiostat. The reference electrode employed was Ag/AgCl electrode. Tafel plots (Figure 5) were obtained and E_{corr} & i_{corr} were deduced. The adjacent Table 1 lists the E_{corr} & i_{corr} values for treated and untreated samples.

Observations

E_{corr} for the treated sample shifts about 45 mV towards the positive direction compared to the as deposited Ni-P (untreated) sample. The i_{corr} of the treated Ni-P sample dropped by 8-10x which suggests that the nano-engineering (inclusion of barrier layer) dramatically lowered the corrosion current (i_{corr}) and improved the corrosion resistance. This data can be correlated with the improved black-pad resistance of the treated sample since the black-pad mechanism is hyper-active galvanic corrosion of Ni (Ni-P) by gold (immersion gold process).

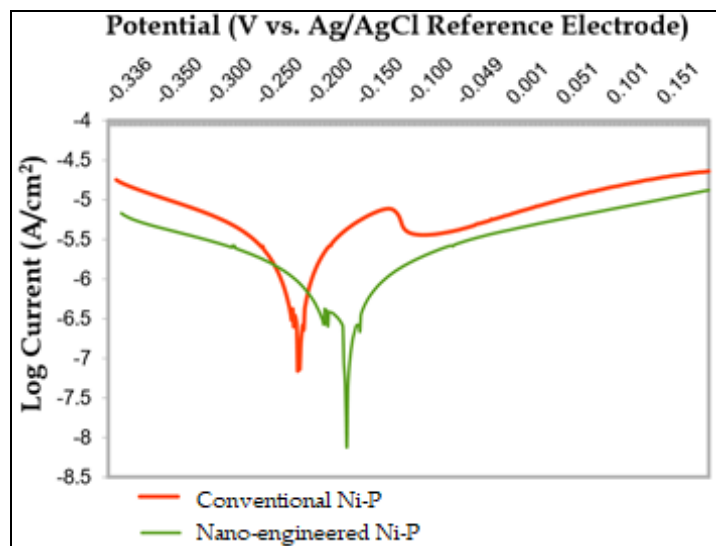


Figure 5: Tafel Plots of conventional Ni-P (ENIG) and nano-engineered Ni-P (novel ENIG) surfaces (Ag/AgCl reference electrode; sweeping potential from -400 to +200 mV versus open circuit potential at a scan rate of about 1 mV/sec)

Table 1: E_{corr} & i_{corr} values of conventional Ni-P (ENIG) and nano-engineered Ni-P (novel ENIG)

Sample Description	i_{corr} (A/cm ²)	E_{corr} (mV)
Ni-P (Untreated- Conventional ENIG)	2.138×10^{-6}	-207
Nano-engineered Ni-P (Novel ENIG)	2.75×10^{-7}	-162

Based on the SEM-EDX and surface roughness (Profilometer) analysis, the nano-engineered (novel ENIG) sample looks smoother and more uniform than the conventional Ni-P surface. The potentiodynamic polarization (electrochemical set-up) test concluded that the i_{corr} decreased by 10 times. This suggests the resistance to black-pad related failures have increased by about 10 fold. These analyses suggest that nano-engineered (novel ENIG) Ni-P surface has high corrosion resistance and will help to remove black-pad related failures in electronic assemblies.

Solder Joint Evaluation

Solder joint formation of the ENIG surface finish happens when the gold layer dissolves into the solder and intermetallic compounds are formed between nickel (nano-engineered (novel ENIG) Ni-P layer) and tin (solder). However, solder joints comprised of intermetallic compounds of tin-nickel-phosphorous can be prone to brittle solder joint failures due to poor ENIG chemistry.

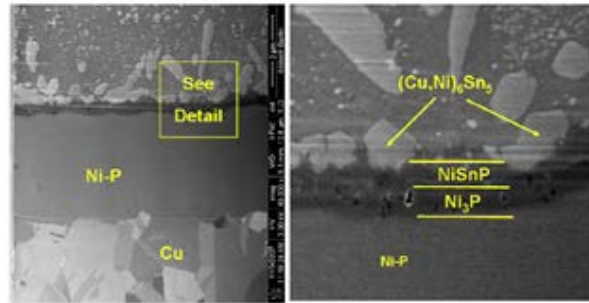


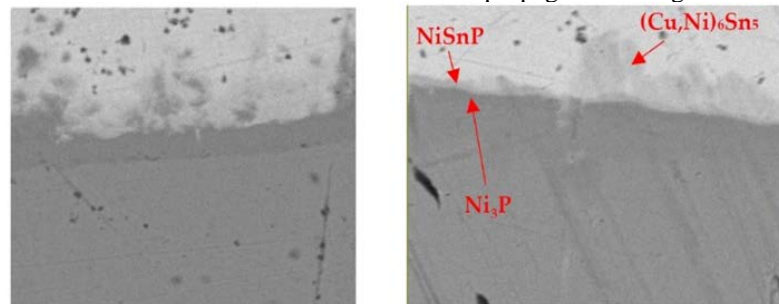
Figure 7: SEM cross-section image at 10,000x (left) of ENIG surface finish after solder ball attach and (right) detailed intermetallic formation [1]

It is essential to evaluate intermetallics in the case of the nano-engineered Ni-P layer (Novel ENIG) and reliability of solder joints to ensure the desired ductile solder joints. The solder ball composition used for this analysis was Sn96.5Ag3.0Cu0.5(SAC305) and the ball diameter was 0.6 mm. The solder balls were assembled on the novel ENIG/conventional ENIG plated copper pads of dummy PCBs using water soluble flux and were then subjected to reflow in an IR reflow oven (peak temperature around 255°C). Samples were subjected to thermal processes, including multiple reflows (up to 6 reflow cycles).

Cross-section and SEM-EDX analysis

Solder joint formation with the novel ENIG samples was done by cross-sectioning to evaluate intermetallic compound microstructures using SEM-EDX along with conventional ENIG (gold on bare Ni-P) samples. A detailed microstructure evaluation was performed to confirm the interfacial reaction between the solder and the nano-engineered (novel ENIG) Ni-P layer analyzing for formation of different intermetallic compound phases and nickel thickness degradation after multiple reflow cycles.

As seen from the SEM images below (Figure 8), the Ni₃P and NiSnP layers are more distinct and compact in a sample containing the barrier layer (Novel ENIG) due to presence of the barrier layer preventing Ni atom diffusion. However, in the case of conventional ENIG, both Ni₃P and NiSnP layers are scattered/diffused and of higher thickness (seen in Figure 2; image on the left). These thicker layers (Ni₃P and NiSnP) can have Kirkendall voids and when the stress is applied, it gets concentrated at the weakest link. The voids coalesce and the crack forms/propagates leading to brittle solder joint failures. [1]



Conventional ENIG

Novel ENIG

Figure 8: SEM Images (5000x) of cross-section solder joints for conventional ENIG and novel ENIG

Mechanical tests of solder joints (not aged)

Ball shear test and cold-ball pull tests were conducted to assess the solder joint reliability using the nano-engineered (novel ENIG) and conventional ENIG surface finish with SAC305 solder after multiple reflow cycles (6 reflow cycles). A failure mode evaluation was conducted to understand where the failures occur during these mechanical tests.

Ball Shear Test

Ball shear testing was performed as per JESD22-B117 by a third-party accredited testing laboratory using a ball shear testing speed of 0.5 mm/sec. The solder ball used in the test samples analyzed was lead-free solder (SAC305).

Observations

The force required to induce the ball failure was 1400 grams (avg.) for the novel ENIG compared to 944 grams (avg.) for the conventional ENIG. About 48% more force was required to generate the ball failure (shear) with the novel ENIG compared

to the conventional ENIG. 80% of the failures happened at the intermetallics for the conventional ENIG compared to no failures (0%) at the intermetallics for the Novel ENIG as shown in Table 2. No brittle solder joints failures were associated with the Novel ENIG finish.

Table 2: Failure locations during ball shear testing for conventional ENIG and novel ENIG surface finishes

Surface Finish	Failure Location		
	At Intermetallics (Solder Joints)	Within Solder	At Cu pad within PCB
Conventional ENIG	80%		20%
Novel ENIG		20%	80%

Ball Pull Test

Ball pull testing was performed as per JESD22-B115 by a third-party accredited testing lab. using a ball pull testing speed of 10 mm/sec. The solder ball used in the samples analyzed was lead-free solder (SAC305).

Observations

The force required to create the ball failure was 2717 grams (avg.) for the novel ENIG compared to 1698 grams (avg.) for the conventional ENIG finish. About 60% more force was required to generate the ball failure (pull) with Novel ENIG compared to the conventional ENIG. Eighty percent of the failures happened at the intermetallics for the conventional ENIG compared to no failures (0%) at the intermetallics for the novel ENIG as shown in Table 3. No brittle solder joints failures were associated with the novel ENIG finish.

Table 3: Failure locations during ball pull testing for conventional ENIG and novel ENIG surface finishes

Surface Finish	Failure Location		
	At Intermetallics (Solder Joints)	Within Solder	At Cu pad within PCB
Conventional ENIG	80%	20%	
Novel ENIG			100%

Long term reliability of Novel-ENIG (after aging):

Solder assemblies involving novel ENIG have been subjected to further reliability tests and analysis which included extended thermal exposure of solder joints (intermetallics) and conducting solder ball pull test and ball shear test to evaluate the aging and long terms reliability of solder joints. Half of the boards were subjected to 150°C for 500 hours and other half were subjected to 150°C for 1000 hours. Ball pull test and shear test were conducted after 500 hours and 1000 hours of exposure. Ball pull test was conducted in accordance to JESD22-B115 and ball shear test was conducted in accordance to JESD22-B117 by a third-party accredited lab.

Below is some few reference data from published scientific studies showing the brittle nature of solder joints. Figure 9 shows Ni-Sn (ENIG) intermetallics based solder joints as reflowed and after aging (1000 hours at 150°C). As seen in the figure (red oval), brittle failure dominates the failure mode after shear testing.

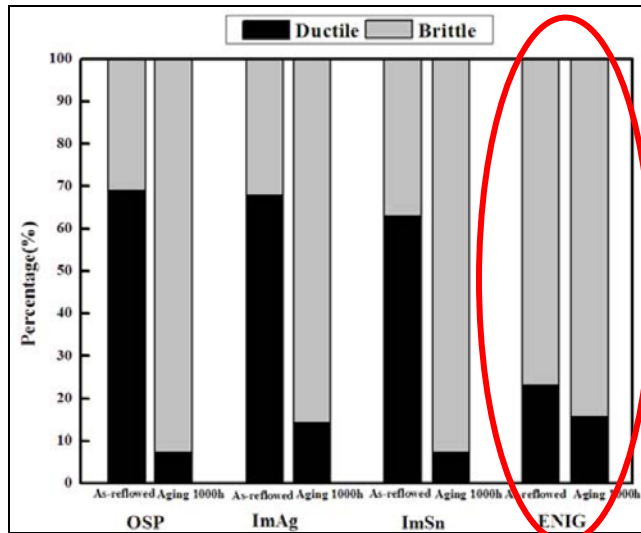


Figure 9: Solder-joints fracture modes with different PCB surface finishes after ball shear test [5]

Another published study (Table 4) showed two different varieties of Ni-Sn based intermetallics (ENIG) failing at the intermetallic/solder joints (brittle nature) after as reflowed (not aged) and after aging (1000 hours at 150°C) during shear and pull testing. Note: IMC: Intermetallics (solder joints).

	Fracture Location	
	Not aged	Aged
ENIG – SMD	IMC / Ni interface	IMC / Ni interface
ENIG – NSMD	IMC / Ni interface	IMC / Ni interface

Table 4: Fracture Locations for different sample types [6]

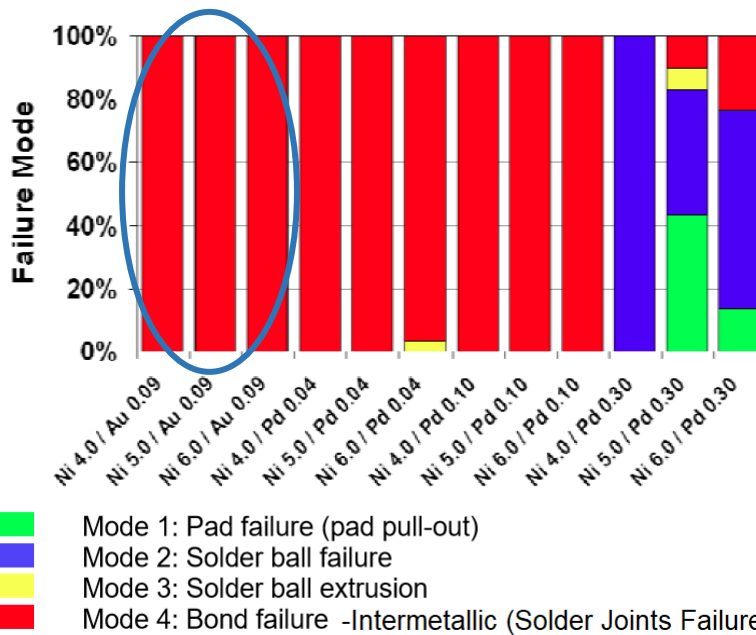


Figure 10: Summary of cold ball pull strength and fracture modes by surface finish for samples tested in "After 5x Reflow" condition [1]

Another study showed intermetallics failures during cold ball pull testing for Ni-Sn based solder joints after 5 reflow cycles (highlighted in the blue oval).

Below are the results of shear test and pull test results conducted by a third party accredited lab. on solder assemblies involving Ni-Sn (Novel ENIG) solder joints. For Figures 11-14, the failure modes are: Mode 1: Solder ball fracture, Mode 2: PCB pad lift/pad crater, Mode 3: Non-wetting failure, Mode 4: Brittle intermetallics failure.

As seen in the Figures 11-14, no Mode 4 failure (brittle solder joints failure) was observed after 1, 3 and 6 reflow cycles and after 500 hours and 1000 hours of 150°C exposure.

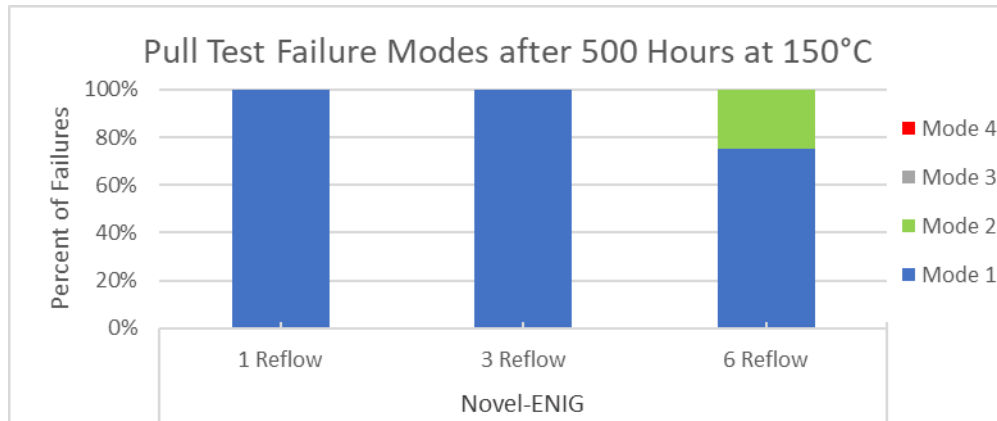


Figure 11: Pull Test Failure Modes after 500 Hours at 150°C

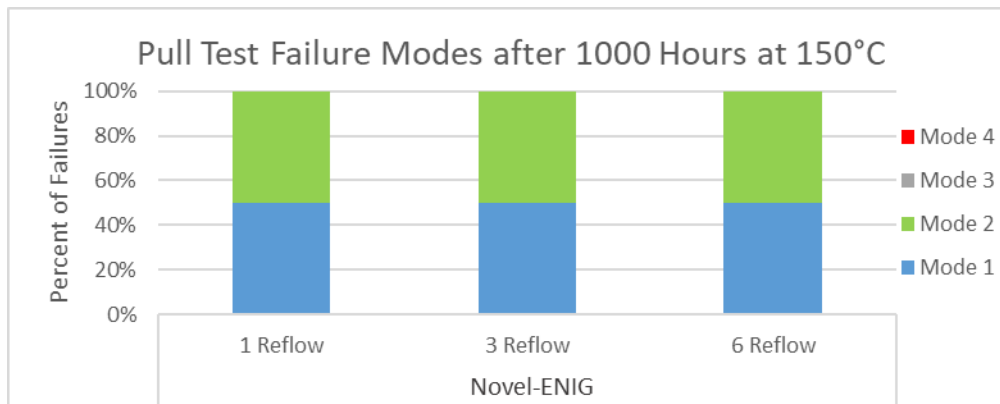


Figure 12: Pull Test Failure Modes after 1000 Hours at 150°C

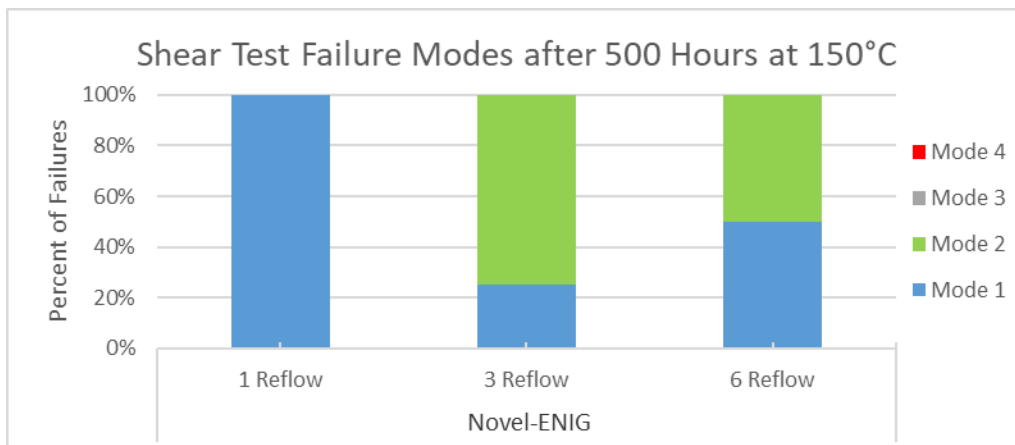


Figure 13: Shear Test Failure Modes after 500 hours at 150°C

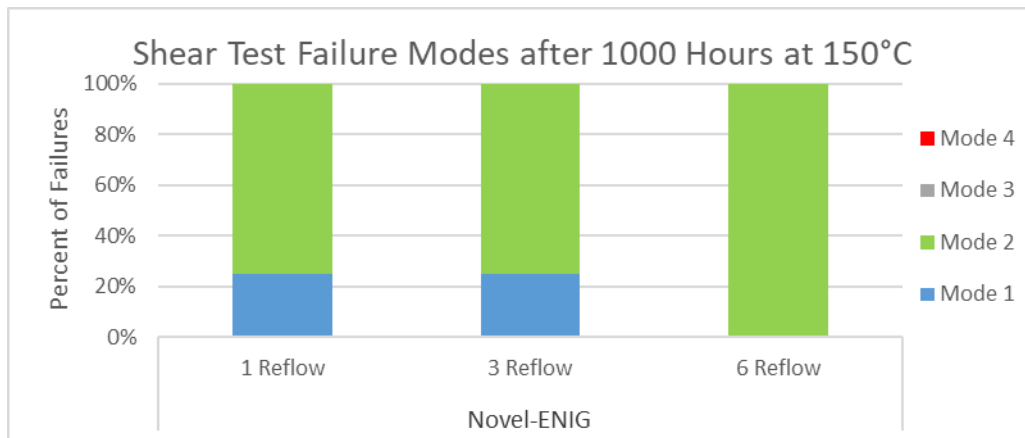


Figure 14: Shear Test Failure Modes after 1000 hours at 150°C

Long-term reliability testing on solder assemblies involving novel ENIG data suggests novel ENIG solder joints show robust behavior (no brittle failures) after 500 and 1000 hours of aging (150°C thermal exposure) which also ensures long term reliability (fatigue failure resistance) for electronic assemblies.

Conclusions

Solder joints have been evaluated using the cross-section/SEM analysis method in which the structure of physical intermetallics have been evaluated. Nano-engineered (inclusion of barrier layer) Ni-P led to compact and distinct layers Ni₃P-NiSnP phases which lead to robust solder joints. However, conventional ENIG had diffused intermetallics and thick layers of those phases.

Mechanical tests were conducted in terms of the solder ball shear test and ball pull test. About 48% more force was required to generate the ball failure (shear) with the novel ENIG compared to the conventional ENIG. Eighty percent of the failures happened at the intermetallics for the conventional ENIG compared to no failures at the intermetallics for the novel ENIG finish. About 60% more force was required to generate the ball failure (pull) with the novel ENIG compared to the conventional ENIG. Eighty percent of the failures happened at the intermetallics for the conventional ENIG compared to no failures (0%) at the intermetallics for the novel ENIG finish. Also, solder assemblies were subjected to extended thermal exposure to evaluate the aging and long-term reliability of solder joints. No brittle failures were observed after ball pull test and ball shear testing (according to JESD22-B115 and JESD22-B117) to the solder assemblies which had been subjected to 150 degrees Celsius temperature for up to 1000 hours and up to 6 reflow cycles during the soldering operation. Both physical evaluation of intermetallic using cross-section/SEM and mechanical tests (solder ball shear test and ball pull test) showed robust solder joints by compact-distinct intermetallic and improved mechanical performance (higher force to failure)-failure location (away from intermetallic).

Corrosion tests conducted in the form of potentiodynamic polarization test (electrochemical set-up) suggested that the corrosion current i_{corr} decreased by 10 times. This correlates to the resistance to black-pad related failures increasing by about 10 fold. These analyses show the nano-engineered (novel ENIG) Ni-P surface has high black-pad related failures resistance in electronic assemblies.

Elimination of black-pad related failures and ensuring robust solder joints with the novel ENIG can help to lead to improved reliability of electronic assemblies for various industry sectors. The novel ENIG involves inclusion of a barrier layer (nano-engineered) at the Ni-P and immersion gold interface which leads to smoothing of the Ni-P interface, passivation of the surface which increases corrosion resistance (helping to remove the black pad issue). Also, the barrier layer helps attain robust solder joints by minimizing the intermetallic thickness and preventing Ni atom diffusion even after long thermal exposure (aging). These benefits will help manufacturers to avoid major failures and the resulting consequences.

References:

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