Identification and Prevention of “Black Pad”

ABSTRACT

Purpose – The use of an electroless nickel, immersion gold (ENIG) surface finish comes with the inherent potential risk of Black Pad failures that can cause fracture embrittlement at the interface between the solder and the metal pad. As yet, there is no conclusive agreed solution to effectively eliminate Black Pad failures. The case studies presented are intended to add to the understanding of the Black Pad failure mechanism and to identify both the plating and the subsequent assembly processes and conditions that can help to prevent the likelihood of Black Pad occurring.

Design/methodology/approach – Scanning Electron Microscope (SEM) analysis of exposed pad surfaces on failed PCBs demonstrated a ‘mud-crack’ appearance, which is a characteristic of the Black Pad phenomenon. In addition, Energy Dispersive X-ray (EDX) analysis was used to identify the elemental composition of the fractured layer between the Ni_3P and Ni_3Sn_4 inter-metallic compound, confirming the presence of Black Pad.

Findings – Grain boundaries or ‘mud-cracks’ that can be clearly seen in a top view of the failed pad surface and corrosion spikes in the failed pad surface, as evident from the cross section sample, should be used as a guideline to confirm Black Pad failures. Maintaining an optimum and well-controlled electroless nickel (EN) and immersion gold bath, in addition to good process control prior to nickel-gold deposition is recommended as the best approach for minimizing the occurrence of Black Pad failures.

Research limitations/implications – Only Sn/Pb soldering processes using ENIG PCBs or package substrates were evaluated and discussed. Thus, the current case studies do not encompass Black Pad failures with lead-free soldering.

Practical implication – the work reported provides guidelines that can be used to identify Black Pad occurrence. It also proposes relevant approaches for minimizing the possible occurrence Black Pad.

Originality/value - The findings of these studies provide a basic understanding of the Black Pad failure mechanism. Subsequently, both the plating and the ensuing assembly processes and conditions that can help to prevent the likelihood of Black Pad occurrence were identified.

Keywords Black Pad, Nickel corrosion, ENIG, Electroless Nickel, Immersion Gold, SEM-EDX, tin-lead solder

Paper type Case study

INTRODUCTION

For many years, copper (Cu) has been the most common metal for interconnecting microelectronic devices due to its superior properties such as low reactivity with acids and good mechanical properties. However, copper is easily oxidized and does not form a self-passivating oxide layer under humid and oxygen-containing environments with gaseous acids, such as carbon dioxide (CO_2), sulfuric acid (H_2SO_4), and hydrochloric acid (HCl) (Bui *et al.*, 2010). With the advent of highly reliable printed circuit boards (PCBs), extensive research and development has been focused in an attempt to solve the copper oxidation problem prior to soldering. One solution, the Electroless Nickel/Immersion Gold (ENIG) surface modification technique has been popularly recommended as a preferred substrate surface finish in the electronics industry to protect copper from premature oxidation. Extensive use of the ENIG finish plating system to coat the solder pads of printed circuit boards, ball-grid arrays (BGA) and flip chip substrates in the electronics industry was undeniably attributed to its excellent properties in terms of wear, corrosion resistance, soldering/solderability, co-planarity, wire bondability and its ability to provide a flat and uniform surface for multiple reflow and wave soldering, allowing complex assembly operations to be undertaken (Zeng *et al.*, 2006; Yoon *et al.*, 2007; Kim *et al.*, 2010).

In spite of the fact that it is relatively expensive, ENIG surface finishes have been widely used to protect copper pads from premature oxidation and it was not until the late 1990s that a major original equipment manufacturer (OEM) brought an ENIG problem to the industry’s attention. The problem is associated with a corroded nickel surface which has resulted in the loss of solderability and that has caused poorly formed solder joints at the interface between the solder and the nickel interface (Milad, 2008a; Milad, 2010). When such weakened joints are subjected to mechanical or thermal-mechanical stress, they can be easily fractured. Under normal microscopic inspection (3 to 5X), a flat pad with a black spot at the failed location is observable. The exact composition of the black nickel corrosion material still remains elusive up to the present day, but the term Black Pad is now widely used to describe a specific type of nickel corrosion on an ENIG surface finish.

Due to the difficulty of reproducing Black Pad failures, the overall formation mechanism is not yet completely understood. Nonetheless, many research and literature reports over the past ten years have helped to provide a clearer picture of the Black Pad issue. Much of the literature reviewed has cited the presence of a phosphorus

(P) rich region at the surface of the electroless nickel (EN) coating surface, along with its growth during the ensuing soldering processes and other thermal excursions that have contributed to solder joint embrittlement. It is evident that Black Pad failure is likely to be initiated in the EN plating process and further developed during the gold (Au) plating process. Therefore, it is under the influence of the plating process parameters and controls therein (Bulwith *et al.*, 2002; Goosey, 2002; Zeng *et al.*, 2006; Milad, 2008a; Milad, 2010).

Data presented in this paper was compiled from a number of Black Pad incidents encountered over the past two years. 'Dye and Pry' analysis, optical inspection and SEM-EDX analysis were used to identify the Black Pad failures. Thereafter, the interpreted results were compared with those reported in the reviewed literature. Some of the basic chemical reactions of the plating process, factors contributing to Black Pad failures and recommendations for a possible approach to minimize Black Pad failures are presented and discussed, based on the correlation established with the cited literature. This paper is a continuation of the authors' preceding work that was presented at the SMTA South East Asia Technical Conference on Electronics Assembly Technologies held on the 19th to 20th May 2011 at Penang, Malaysia.

METHODOLOGY

Open solder joints, especially for area array packages were confirmed using the 'dye and pry' technique. The locations of the failed area array packages with suspected Black Pad failures were pre-determined and cut using a diamond saw. The sample specimens were later washed in running tap water and cleaned in an ultrasonic cleaner for approximately 15 minutes to remove debris from the earlier process. After which, the sample specimens were then dried using an air gun. Then, the entire samples were sufficiently soaked in Dykem Steel Red Layout Fluid from ITW. The samples were placed in a desiccator and vacuumed in a Sprayit Vacuum Impregnator for 15 to 30 minutes to ensure complete penetration of the red dye into the crack and fracture layer. After that, the samples were cured in an oven at 100°C for 3 to 4 hours and carefully pried apart using a flat head screwdriver for visual inspection.

An Hitachi S2150 SEM operating at 15 kV was used to investigate the top nickel layer morphology for excessive etching of grain boundaries and mud-cracking phenomena, while quantitative analysis of the elemental composition of different phases was performed using an EDAX XM2-60 EDX detector. For the surface morphology study, the top surface's gold coating was removed with a potassium iodide solution, (a specific chemical stripper that will only dissolve the gold coating). The samples were then sputtered with a layer of carbon (C) using a Palaron Range CC7650 sputter coater and analysed using the SEM. All cross section samples were prepared according to IPC-TM650 Test Method 2.1.1, “Microsectioning, Manual Method” and sputtered with a layer of carbon prior to SEM-EDX analysis (IPC-TM-650/2.1.1, 2005/04; Castello *et al.*, 2006).

RESULTS AND DISCUSSION

The Black Pad failure mode can occur on the BGA Nickel-Gold substrate or on the Nickel-Gold surface finish of a printed circuit board (PCB). Both case studies are presented and discussed in this paper. The ‘dye and pry’ analysis can provide easy to interpret results identifying which I/Os were broken completely and/or partially the specific location of the failure within the joint. It is popularly used for the testing of area array style package solder joints and, in most instances, the identified specific open I/Os have a good match with the diagnosed electrical ‘opens’ or ‘intermittents’ that accompanied the failures. However, process and failure analysis engineers should handle the investigation with careful and comprehensive inspection, identifying the Black Pad fracture mode at different layers which will eventually lead to two different directions of corrective action ie to work with the component OEM or the PCB plating company.

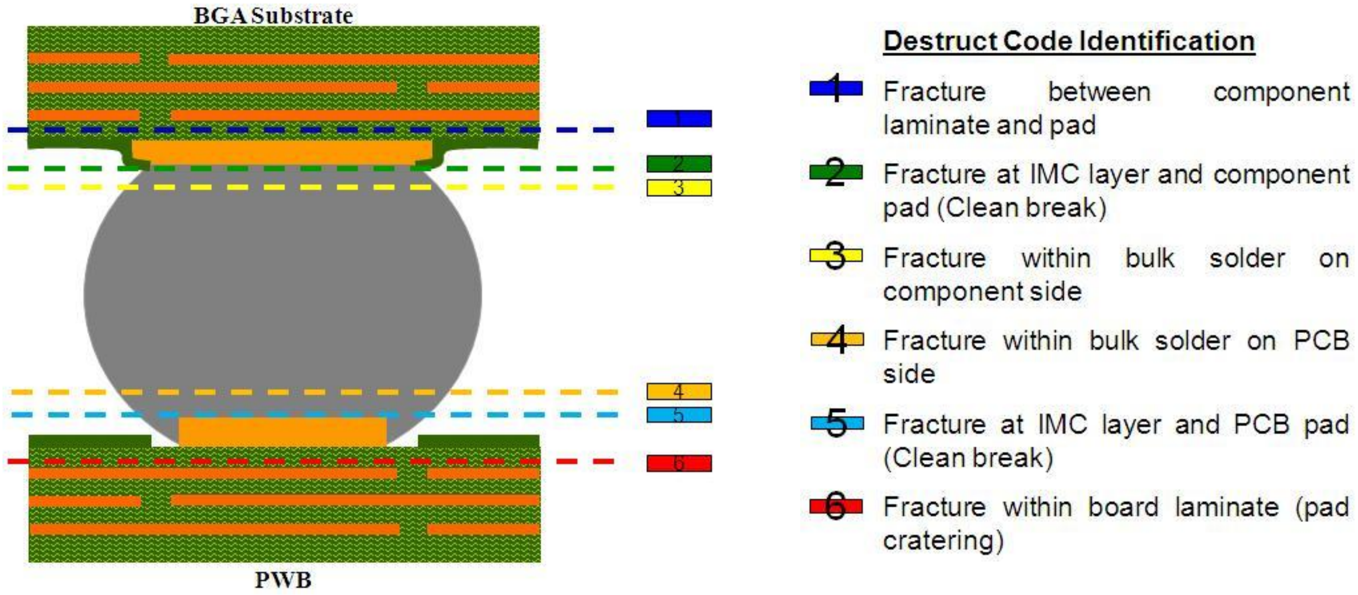

Figure 1: Destruct Code identification used as a guideline to check possible Black Pad failures side.

The authors had pre-identified six different destruct codes to be used as a guideline in our attempt to confirm the Black Pad failure mode. All of the six different destruct codes are shown in Figure 1. A direct fracture between the inter-metallic compound (IMC) layer and BGA substrate under Destruct Code 2 is an early sign of possible Black Pad failures on BGA substrate. However, Destruct Code 5 will be indicating a possible open joint at the PCB side due to Black Pad failure. (Destruct Codes 1, 3, 4, and 6 are not of interest for the present case studies.)

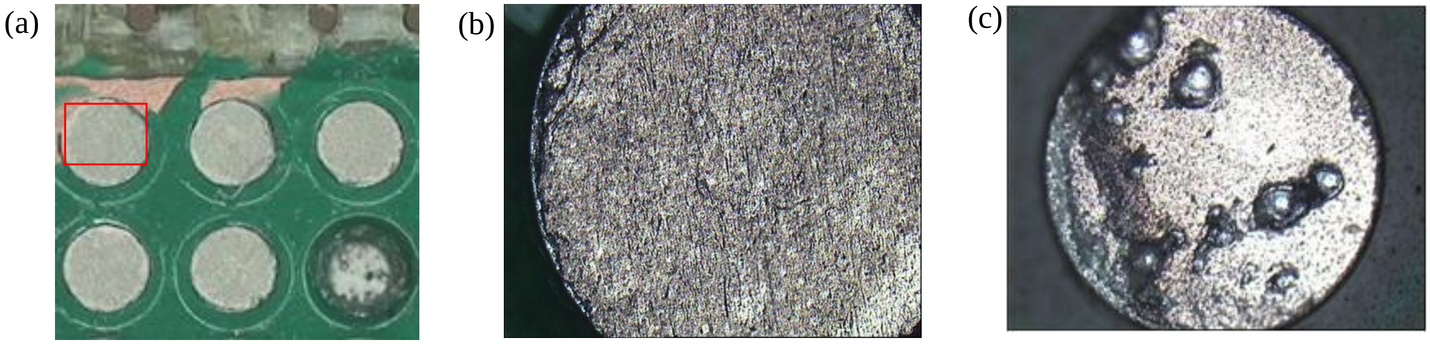

Figure 2: 'Dye and Pry' analysis on reflowed samples. (a) PCB BGA pad at 20X magnification. (b) PCB BGA pad at 200X magnification from (a). (c) PBGA package side substrate at 200X magnification.

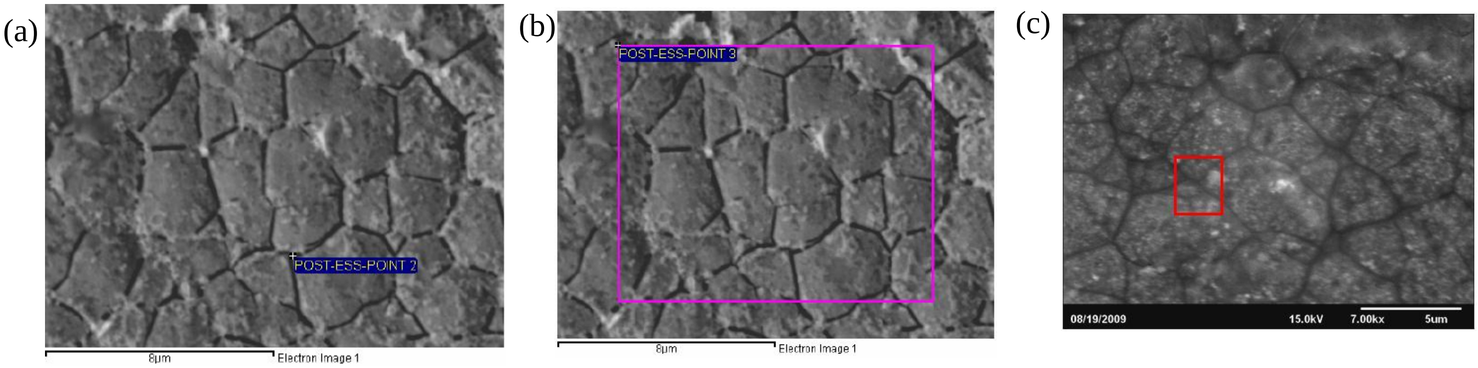

Figure 3: Top view inspection (a) Spot EDX analysis at PCB side, 8000X magnification. (b) Area EDX analysis at PCB side, 8000X magnification. (c) Area EDX analysis at PBGA package side substrate, 7000X magnification.

Figure 2(a) and 2(b) present an ENIG-plated pad on a PCB where a reflowed BGA package site was 'dyed and pried' prior to 20X and 200X magnification visual inspections. Visual inspection at 200X magnification had shown that there were some bright areas indicating a small amount of fine IMC particles (grey) at the centre and the perimeter of the BGA pads, but, it is at a much smaller ratio as compared to the area observed with pink or black spot. The smaller ratio of IMC particles was evidence of a weaker PCB-BGA package bond. Although some solder joint formation still existed and Black Pad failures are not apparent upon initial soldering, failures can be experienced at a later stage in the field when there are stresses induced by thermal and mechanical excursions. A similar phenomenon was observed on a PBGA package substrate as is evident from Figure 2(c). Visual inspection at 200X magnification had shown a pink or black coverage of more than half of the total substrate surface. Although partial IMC formation can be confirmed at random locations on the substrate surface, a much weaker BGA package-solder ball bond is expected.

In a top view, further magnification at 7000X and 8000X had shown a feature that looked like the boundaries of the ENIG plating nodules, as can be seen from Figure 3(a) to 3(c). The boundary-like feature was the commonly called 'mud-crack' phenomenon related to Black Pad failure in the electronics industry. Both the spot and area EDX analyses had shown a close match of elemental composition, as indicated in Table 1. With the assumption that all the tin signals were detected from the few Ni_3Sn_4 particles (for the SnPb joint chemistry) and they were negligible on the BGA pad surface (for PCB) and substrate (for PBGA), the calculated atomic ratio of Ni:P was approximately 48.0:16.0 for the former and 14.4:4.0 for the latter, which was very close to the stoichiometry value of Ni_3P. The obtained value is in good agreement with Zeng *et al.*, 2006. Hence, the presented result had confirmed that the joint was fractured between the Ni_3P and Ni_3Sn_4 layers, while the mud-crack phenomenon observed was in the Ni_3P layer.

Table 1: EDX analysis results for locations identified in Figure 3(a) to 3(c)

Elements	Figure 3 (a)		Figure 3 (b)		Figure 3 (c)	
	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %
C	14.55	42.18	13.97	41.87	26.87	61.61
O	3.09	6.74	2.65	5.96	1.99	3.43
Si	0.58	0.71	0.50	0.64	N/D	N/D
P	10.44	11.74	9.96	11.58	8.13	7.23
Ni	59.02	35.01	57.53	35.28	55.30	25.94
Sn	12.33	3.62	15.39	4.67	7.72	1.79

Note: N/D- Not detected

In fact, Black Pad failures are a nickel corrosion process which may occur as a result of hyper-corrosive attack of the nickel layer during the immersion gold plating process. The deposition mechanism of gold on electroless nickel (EN) is a displacement reaction. Due to the potential difference between the two metals, the less noble nickel will be oxidized to provide electrons that facilitate reduction of the more noble gold. Nickel will be dissolved into the immersion gold solution releasing two electrons to be reacted with gold atoms, while two gold atoms will be reduced to form a layer of metallic coating on nickel. This process will continue until all the nickel is covered by gold and no more nickel is available for further reaction. Thus, the immersion gold plating is a self-limiting process and ceases once the underlying nickel layer is effectively isolated from the immersion gold solution. The chemical reaction can be represented by Equations (1) and (2) below respectively.

Anodic reaction: $Ni^0 \rightarrow Ni^{2+} + 2e^-$ (1)

Cathodic reaction: $2\ Au^+ + 2e^- \rightarrow 2\ Au^0$ (2)

It had been reported that under such circumstances, compromised EN plating with fissures or grain boundaries is prone to gold attack in the immersion gold bath as the solution in the grain boundaries is not replenished at the same rate as the solution in contact with the surface of the nodules. The establishment of galvanic cells between the differing solution concentrations in the grain boundaries and on the surface has given rise to the possibility og hydrogen generation, which is an undesirable side reaction ie;

$$Ni + 2H^+ \rightarrow Ni^{2+} + H_2\uparrow (3)$$

Accelerated ion generation from galvanic interactions will release nickel cations without the deposition of gold. Thus, the gold bath dwell time is extended to complete the gold deposition to the point of compromising the nickel surface. In addition, the more nodular a deposit, the deeper will be the inter-granular grain boundaries. Hence, the greater tendency of excessive corrosion on the nickel surface to take place (Bulwith *et al.*, 2002; Goosey, 2002; Milad, 2008a; Milad, 2010).

To obtain more conclusive evidence for Black Pad failures, two cross-sections were performed on the as-received PCB sample at two different locations on the same board. One of the cross-section locations was on the suspected known-bad pad. Figure 4(a) shows that corrosion spikes were clearly seen on the suspected sample at 5000X magnification. It can be seen that a feather-like structure around the spike or crack had spread into the nickel-phosphorous (Ni-P) plating, while only a hair line spike defect was observable on the good sample, as had been shown in Figure 4(b). As EN and immersion gold process will submerge the whole PCB in the solution bath, it is expected that the metallic coating defects and thicknesses should be consistent across all areas of the surface. The only plausible explanation for the different observation may be due to the complexity of the PCB design at different locations or there may be an uneven movement in the solution bath, meaning that the solution bath was not maintained at a homogeneous concentration. On a separate note, cross sectional analysis was not performed on the failed PBGA when Black Pad failures happened, thus, comparative discussion of the two different destruct codes was not further explored in the present paper.

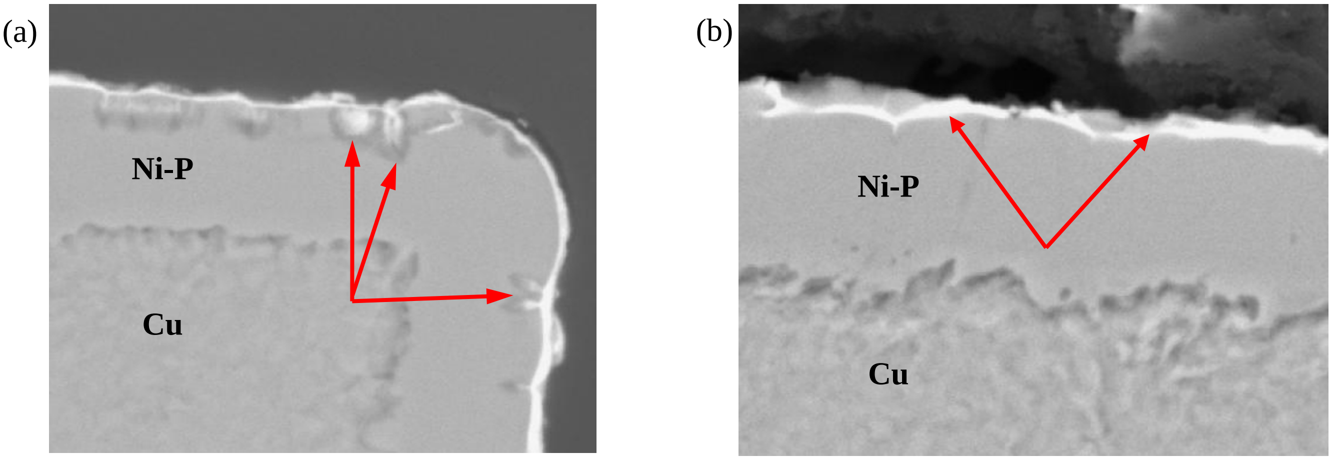

Figure 4: Cross section samples at PCB BGA pad. (a) Feather-like spikes or crack indicating the Black Pad phenomenon. (b) An acceptable good sample.

Normally, when a Black Pad related solder joint failure happens, a high phosphorus content is detected at the fractured pad surface. Due to this observation, there is common confusion in citing a high phosphorus content as evidence from failure analysis as the root cause of Black Pad failures. Thus, the ensuing suggestion of lowering the phosphorus content in the EN plating bath as a fix, which has rendered the issue, remains unsolved. In fact, in some of publications reviewed it has been reported that an increase in the phosphorus content to a range of 7-11% in the EN bath actually reduces the corrosion during immersion gold plating process. This is because above 7%, EN deposits have an amorphous structure and are corrosion resistant, whilst at levels below 7% EN the deposits exist with a microcrystalline structure. A phosphorus content of about 15 wt% (25 at.%), higher than the initial bulk EN plating is expected from EDX analysis after the soldering process irrespective of whether ENIG PCB exhibit a Black Pad defect or not (Johal *et al.*, 2004; Zeng *et al.*, 2006; Yoon *et al.*, 2007; Bath, 2007; Kang *et al.*, 2009).

It is necessary to understand the reaction mechanism when solder reacts with the ENIG plating in order to estimate the correct expected phosphorus content in an ‘after soldering’ sample and in the EN solution bath. During the soldering process, solder melts on the top most ENIG layer and the gold plating dissolves into the molten solder, leaving behind the Ni-P layer exposed to the molten solder. Tin (Sn) will be in direct contact with the Ni-P plating and an Ni_3Sn_4 IMC is formed at the interface upon cooling. Furthermore, the reaction of nickel atoms to form the Ni_3Sn_4 IMC enhances the crystallization of the amorphous Ni-P plating into the P-rich nickel layer such as Ni_3P. It is in this regard that after reflow, the crystallized composition of the Ni-P plating will become Ni_3P with an estimated ≥15wt% (25 at.%) phosphorus.

However, there is another very thin ternary layer between Ni_3Sn_4 and Ni_3P with an approximate thickness of 100 nm containing nickel, tin and phosphorus that has not been agreed upon yet in the literature due to the difficulty in confirming its composition. Nonetheless, two possible compositional formulae (Ni_3SnP or Ni_2SnP) have been reported and discussed by a few groups of researchers. One thing that has been agreed is that the ternary layer between Ni_3Sn_4 and Ni_3P will bring adverse effects causing the spalling of the IMC layer, which has significantly affected the reliability of the solder joint (Zeng *et al.*, 2006; Yoon *et al.*, 2007; Kang *et al.*, 2009).

Discussion of the presented results has supported that concept that the Black Pad failures are very likely attributable to a few possible factors, namely EN plating, immersion gold plating and the subsequent assembly process parameters working together to produce a perplexing failure mechanism that is yet to have a full comprehensive understanding of the subject matter. Based on a wealth of literature reviewed and the author's experience working with the plating manufacturers and the assembly engineers, there are basically three approaches that are applicable to prevent Black Pad failures.

Pre-nickel-gold Deposition Influences

A micro-etch treatment of the copper surface prior to the EN plating process is a fundamental pre-requisite to ensure a clean surface for the initial catalysation of the copper surface and the correct morphology for the subsequent EN and immersion gold plating process to take place. Micro-etching helps to provide a micro-roughened copper surface that will enable good adhesion of the EN plating to the copper layer. Thus, it is vital that the metallic etch resist used to protect the underlying copper is completely removed without leaving any copper-tin inter-metallic residues to prevent them from interfering with the subsequent plating processes. In addition, it is also critical to make sure that the acidic stripping chemistry is completely rinsed off and the PCBs (or package substrate) are properly dried to prevent localized corrosion of the copper stripper residues (Goosey, 2002; Milad, 2008a).

EN Deposition Control

Higher content of phosphorus in the EN plating bath can improve the corrosion resistance during the immersion gold plating process. Depending on the chemistry used, it had been commonly recommended to control the phosphorus content in the EN deposition bath within the range of 7 to 11%. This can be achieved by maintaining the EN plating rate at approximately 7 to 11 micro inches per minute. Phosphorus contents below 7% have been known to produce microcrystalline EN deposit structures, whilst above 7% the EN deposit exists as an amorphous structure and is corrosion resistant. In addition, slower EN deposition rates are preferred in order to minimize deep crevice formation at grain boundaries. This is in view of the fact that corrosion during immersion gold plating tends to happen more aggressively at grain boundaries with deeper crevices. Therefore, it is important to carefully control the reaction temperature and pH to achieve an optimum EN plating surface morphology with smaller nodule sizes and uniform, minimum crevice formation at grain boundaries. A few researchers had reported that the phosphorus content of the EN plating will decrease with increasing pH values and vice versa. The pH values between 4.0 and 4.5 have been found to be effective in achieving a phosphorus content of approximately 10.0% in the EN plating (Yoon *et al.*, 2007; Milad, 2008a; Saturn Electronics Corporation, 2009; Won *et al.*, 2010).

Immersion Gold Deposition Control

Although Black Pad defects may have been initiated since the micro-etch treatment of the underlying copper layer prior to EN plating process, Black Pad actually occurs during the immersion gold plating process. Immersion gold plating is a self-limiting process and ceases once the underlying nickel layer is effectively isolated from the immersion gold solution. The consistency of the oxidation-reduction reaction during immersion gold plating can influence the stoichiometry of the gold bath. The less noble nickel will be oxidized to provide electrons that facilitate reduction of the more noble gold. Nickel atoms released into the gold bath should follow the stoichiometry and remain consistent if the EN plating bath is properly maintained. Otherwise, if the stoichiometry reaction has shifted to the right producing more nickel atoms than it should, the immersion gold bath will become adversely more “aggressive” than is expected. Hence, chemical analysis of the gold bath is required so that corrective action can be taken to adjust the composition and to enable it to operate according to the recommended specification (chemical composition, concentration, pH, temperature, and run rate).

In addition, gold plating aggressiveness can also be prevented by controlling the appropriate gold plating thickness to within the range of 0.05 to 0.10 μm, in which the chemical supplier's recommended gold plating rate or dwell times in the gold plating bath should be followed and maintained accordingly. Prolonged dwell times or faster plating rates that are higher than the recommended specification to ensure complete EN plating should be avoided (IPC-4552, 2002; Milad, 2008b; Milad, 2010).

CONCLUSION

According to the author's experience and some of the published literature, the following criteria are proposed as a guideline to confirm Black Pad occurrence. Open solder joints and a flat pad should be detected at failure location and there is very little solder or no solder remaining on the pad. Under high magnification, SEM inspection, grain boundaries or 'mud-crack' phenomena are clearly seen in a top view of the failed pad surface. Cross sections of failed pads will reveal corrosion spikes in the failed pad surface and, if they can be correlated with the surface 'mud-crack' phenomenon, then the evidence is likely to be conclusive in confirming Black Pad failures.

Much work has been undertaken by various groups of researchers in an attempt to attain a comprehensive understanding of the Black Pad failure mechanism and to identify both the plating and the subsequent assembly processes and conditions that can prevent the likelihood of its occurrence. However, there is no conclusive solution that has been agreed upon to effectively eliminate Black Pad failures. Maintaining optimum and well-controlled EN and immersion gold baths, in addition to good process control prior to the nickel-gold deposition process are still considered to be the best approaches at the moment to minimize Black Pad failures.

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