Jurnal Teknologi, 52 Mei 2010: 1–12

EFFECT OF SOLDER BUMP SIZE ON INTERFACIAL REACTIONS DURING SOLDERING BETWEEN PB-FREE SOLDER AND Cu AND Cu Ni/Pd/Au SURFACE FINISHES

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Abstract. *Flip chip* technology provides the ultimate in high I/O-density and count with superior electrical performance for interconnecting electronic components. Therefore, the study of the intermetallic compounds was conducted to investigate the effect of solder bumps sizes on several surface finishes which are copper and Electroless Nickel/Electroless Palladium/Immersion Gold (ENEPIG) which is widely used in electronics packaging as under-bump metallization (UBM) for flip-chip application nowadays. In this research, field emission scanning electron microscopy (FE-SEM) analyses were conducted to analyze the morphology and composition of intermetallic compounds (IMCs) formed at the interface between the solder and UBM. The IMCs between Sn–Pb and lead-free solder with Cu surface finish during reflowing were mainly $(Cu, Ni)_6Sn_5$ dan Cu_6Sn_5. While the main IMCs formed between Sn–Pb and lead-free solder on ENEPIG surface finish are $(Ni, Cu)_3Sn_4$ and Ni_3Sn_4. The results from FESEM with energy dispersive x-ray (EDX) have revealed that isothermal aging at 150°C has caused the thickening and coarsening of IMCs as well as changing them into more spherical shape. The thickness of the intermetallic compounds in both finishes investigated ware found to be higher in solders with smaller bump size. From the experimental results, it also appears that the growth rate of IMCs is higher when soldering on copper compared to ENEPIG finish. Besides that, the results also showed that the thickness of intermetallic compounds was found to be proportional to isothermal aging duration.

Keywords: Electroless nickel; electroless palladium; immersion gold (ENEPIG); flip chip; Ni/Pd/Au Under-bump metallization (UMB)

Abstrak. Teknologi *flip chip* memberikan ketumpatan I/O yang sangat tinggi dan mengambil kira prestasi elektrikal yang paling baik dalam penyambungan komponen elektronik. Oleh itu, kajian tentang sebatian antara logam dilaksanakan untuk mengkaji kesan saiz bebola pateri bagi beberapa penyudahan permukaan, iaitu Kuprum dan Nikel tanpa elektrod/Palladium tanpa elektrod/ Emas rendaman (ENEPIG). Pelogaman di bawah pateri (UBM) Ni/Pd/Au bagi aplikasi *flip chip* digunakan dengan sangat meluas dalam pembungkusan elektronik. Analisis FESEM dilakukan untuk menganalisis morfologi dan komposisi bagi sebatian antara logam (IMC). IMC yang terbentuk antara pateri Sn-Pb dan tanpa Pb dengan penyudahan permukaan kuprum semasa proses pematrian logam secara umumnya adalah $(Cu, Ni)_6Sn_5$ dan Cu_6Sn_5. Sementara IMC utama yang terbentuk antara pateri Sn-Pb dan tanpa Pb dengan penyudahan permukaan ENEPIG adalah $(Ni, Cu)_3Sn_4$ dan Ni_3Sn_4. Hasil daripada analisis morfologi menggunakan FESEM dengan EDX menyatakan penuaan sesuhu pada suhu 150°C menyebabkan penebalan dan pengasaran struktur IMC serta

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menjadikan bentuknya kepada lebih sfera. Tebal IMC bagi kedua-dua penyudahan yang dikaji adalah lebih tinggi bagi bebola patri yang lebih kecil. Daripada hasil kajian juga, didapati bahawa kadar pertumbuhan IMC adalah lebih tinggi apabila pematrian dilakukan atas penyudahan kuprum berbanding ENEPIG. Hasil kajian juga menunjukkan ketebalan IMC adalah berkadaran dengan masa penuaan sesuhu.

Kata kunci: *Flip chip*; Kumprum dan Nikel tanpa elektrod; Palladium tanpa elektrod; Emas rendaman (ENEPIG); Pelogaman di bawah pateri (UMB) Ni/Pd/Au

1.0 INTRODUCTION

The requirements of electronic packaging is toward higher I/O, greater performance, higher density, and lighter weight, the use of area array packaging technology is expected to increase. The type of packaging, flip chip, provides the ultimate in high I/O-density and count with superior electrical performance, and very small size [1]. It is well known that soldering involves a reaction between molten solder and substrate, which dissolves some of the substrate and which forms some sort of intermetallic layer [2]. The interfacial chemical reactions enhance the wettability between the solder and the substrate. The intermetallic compound (IMC), grows at the solder and at the under bump metallurgy (UBM) interface at the practical operating temperature during the reflow process and eventually forms the solder bump.

Due to its excellent conductivity and surface for soldering, copper has been widely used as the substrate materials. Though, surface finishes are still needed to be deposited onto the substrate surface as copper may oxidize easily. For electroless nickel/electroless palladium/immersion gold (ENEPIG) surface finish, nickel functions as the diffusion barrier with its low dissolution rate into tin. Meanwhile the palladium and gold layers can protect the underlying metals from oxidation.

ENEPIG is formed by the deposition of electroless nickel (305–610 μm), followed by 12.5–37.5 μm of electroless palladium with an immersion gold flash (2.5–5.0 μm). The electroless palladium layer prevents any probability of nickel corrosion that may caused by the immersion gold deposition reaction. This layer is much harder than gold, providing added strength to the surface finish for wire bonding and connector attachment, while protecting the underlying nickel from oxidation [3].

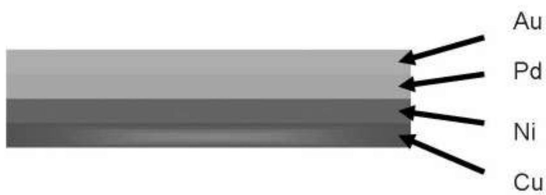


Figure 1 The deposition layers of ENEPIG surface finish

The nickel/palladium/gold plated boards have a shelf life of up to two years or more. The process is almost similar to the nickel/gold process, except that it uses palladium metal layer that is deposited after the nickel layer, but prior to the final gold layer. ENEPIG is the finish that has the widest latitude for a variety of applications. It provides a flat co-planar surface. Sometimes referred to as the universal finish, it is a good soldering surface, a gold wire bondable surface, aluminium wire bondable surface, as well as a contacting surface.

2.0 EXPERIMENTAL DETAILS

In order to investigate the effect of solder bump size on intermetallic compound (IMC) formation between solder bump and substrate, three different sizes of solder balls, namely 300, 500, and 700 μm in diameter were soldered on difference UBM; Bare Copper surface finish and Electroless Nickel/Electroless Palladium/Immersion Gold (ENEPIG) finishes. The Copper substrate is the copper-polymer sandwiched substrates (FR4). The experiments must start with depositing the under bump metallurgy first. The ENEPIG is an under bump metallurgy which consists of layers of Nickel, Palladium and Gold towards copper plate. The first step in the ENEPIG process is to catalyze the copper surface for Ni deposition. The copper substrates were first subjected to grind mechanically to remove the oxide layer and rust followed by rinse with running tap water. Then, the copper substrates were subjected to a pretreatment cleaning process to remove dust, grease and oxide layers as well as to activate the copper surface for ENEPIG plating preparation. Since Ni is less noble than Cu, activation of the copper surface for Electroless Ni plating can be achieved by seeding a noble metal such as palladium chloride (or palladium sulfate) [4]. The substrate was activated twice during the pretreatment before electroless nickel plating and before the electroless palladium plating was initiated with palladium solution. The immersion gold plating was performed right after electroless palladium without pretreatment except rinsing with water. The Nickel deposition is aided by a hyphophosphite (H_2PO_2) reducing agent that decomposes during this reaction and results in the decomposition of phosphorus in the electroless Ni layer [5]. In this research, medium phosphorus/midphosphorus (7–10%) was used. Several combinations of electroless Nickel plating solutions have been tried out before arriving at one most stable plating succession. The components of optimum nickel plating solution are one part of NIMUDEN 5X and four parts of distilled water. The plating process was conducted for 50 minutes until plating layer reached the thickness of 5–6 μm at temperature of 95°C and the range of pH is 4.3–4.5.

For palladium solution plating, it is used 2 g/L $PdCl_2$, 200 ml/L NH_4OH (28% NH_3), 3.5 g/L $Na_2EDTA.2H_2O$, 20 mg/L (8 drops) Thiodiglycollic acid, and 8.5 g/L $NaH_2PO_2H_2O$ where the operating temperature of the plating bath was set around 45°C and a pH within the range of 8.0–9.0. The parameters has been set up in order to obtain Pd thin film layer with the thickness < 0.1 μm. The final step for ENEPIG plating is immersion gold where the gold layer acts as an oxidation barrier. The immersion gold plating is conducted immediately after electroless palladium with no pretreatment, except rinsing in running tap water. The combination for the immersion gold solution are 2 g/L *Potassium cyanoaurate* (KAuCN2), 75 g/L *Ammonium chloride*, 50 g/L *dehydrate Sodium citrate*, and 10 g/L *dehydrate Sodium hyphophosphite*. The operating temperature of the plating bath was set around 93°C, and a pH within the range of 7.0–7.5 for 10 minutes immersion time in order to obtain a very thin layer of gold.

After preparing the substrate and under bump metallurgy, the differences sizes of solder balls were placed onto a solder mask-covered surface of the substrate followed by reflow soldering process at 250°C in a resistant furnace. The samples then subjected to isothermal solid state aging at a temperature of 150°C for different aging times. Analysis of the results obtained focused mainly on characterisation the intermetallic compounds formed between the Cu/Au under bump metallurgy and solders. This was made as function of process parameters such as thickness and morphology of IMC.

3.0 RESULTS AND DISCUSSIONS

3.1 Effect of Solder Bump Size on Intermetallic Compound Thickness

In order to study the effect to the intermetallics thickness, a measurement from the cross sections of different solder joints was conducted and shown in histograms in Figure 2. These histograms illustrate the effect of solder joint size on interfacial reactions occurring during soldering. It is clearly shown that the intermetallics relatively grow faster when soldering with a smaller solder volume (small solder bump) compared with those formed in larger solder volume. The results are similar with all solder joints which is a clear trend that can be observed here, where the IMC layer thickness decreases with increasing solder size from 300 μm to 700 μm for ENEPIG and copper surface finishes.

The dissolution phenomena of metals involved in the interfacial reactions are controlled by the ratio of solder volume to contact pad area [6]. With an increase in this V/A ratio the diffusion distance for Cu to saturate the liquid solder increases, thus resulting in slower interfacial reactions. This explanation could be applied in the present study. Even the metal pad used is different for all specimens and depends on the solder sizes, the ratio V/A still increases with increasing the solder ball diameter. This confirms the finding that with decreasing the solder volume i.e; increasing the V/A ratio will lead to high intermetallics growth [7].

The effect of isothermal aging shows a decrease in IMCs grow rate after 1000 hours for both surface finishes because the consumption of the copper substrate will decrease when there are other layers of IMCs existed, which prevent further diffusion between copper and solder material.

Between the Cu substrates and the solders, there were relatively thicker intermetallics formed as compared to ENEPIG substrates. Figure 2(b) shows that the intermetallics thickness has not shown significant increase after 1000hrs. This phenomenon occurred due to the fact that after soldering the IMC on copper finish has scallop-type morphology with a rough interface. However, after 1000 hours ageing process the IMC has somewhat leveled off and became more continuous and exhibit a smoother

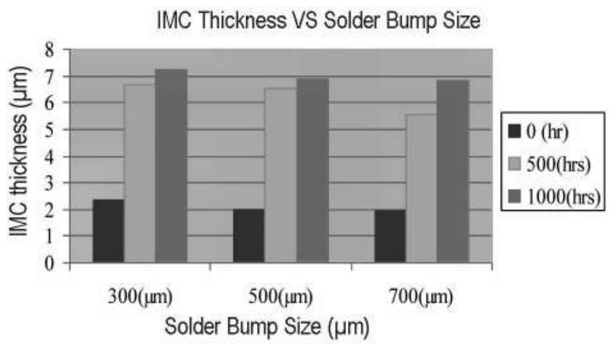


Figure 2(a) IMC thickness for Pb-free solder on Cu surface finish

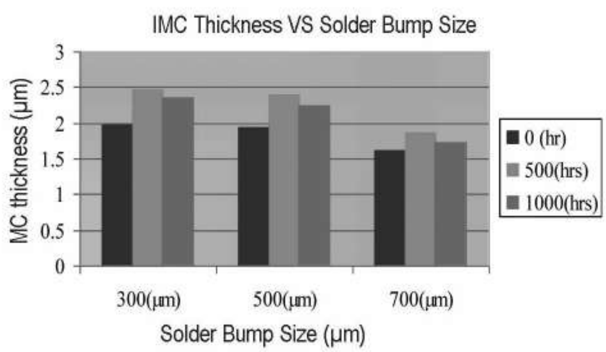


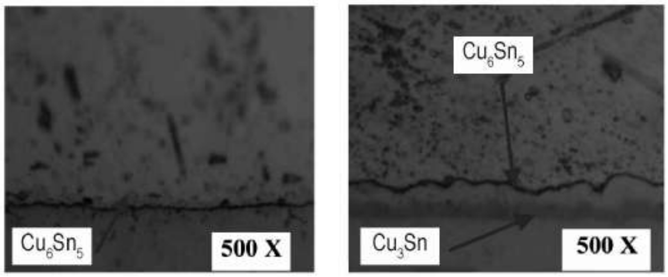
Figure 2(b) IMC thickness for Pb-free solder on ENEPIG surface finish interface [7]. IMCs produced in ENEPIG also decrease when the solder bump size increases. This is due to the existence of the Ni diffusion barrier layer between the solder and substrate. Since the reaction rate of Ni with liquid Sn solder is smaller than that of Cu, it acts to prevent the rapid interfacial reaction between solder and Cu conductor in electronic devices. This in turn, results in thinner intermetallic compounds. During reflow soldering, the molten lead-free Sn-4.0Ag-0.5Cu solder alloy dissolves the entire Au and Pd layer into the liquid solder, allowing Sn from the molten solder to react with the Ni layer to form Ni-Sn intermetallics.

3.2 Composition and Morphology of Intermetallic Compounds Analysis

During reflow soldering, the formation of the solder joint exists when there are interactions between liquid solder with the base metal, copper or nickel. This interfacial reaction results in the formation of intermetallic compounds. Besides, separate samples of all categories were aged at 150°C for 500 hours and 1000 hours. In all these samples several phases were identified by optical and electron microscopy together with EDX. The solid-state reactions and growth of intermetallics during long term exposure to high temperatures are also important because the morphology, distribution and thickness of these intermetallics will affect the solder joint reliability. The morphologies of the various intermetallics formed in the solder joint during soldering and after solid state ageing were examined on all specimens using the deep etching of the solder.

It is well known that soldering involves a reaction between molten solder and substrate, which dissolves some of the substrate and which forms some sort of intermetallic layer [2]. The interfacial chemical reactions enhance the wettability between the solder and the substrate. When tin-containing solder comes in contact with the copper pad surface, a layer of Cu-Sn IMC, consisting of the Cu_6Sn_5 phase adjacent to the solder and the Cu_3Sn phase next to the copper land pad surface is formed in between and serve as the bonding material for solder joint.

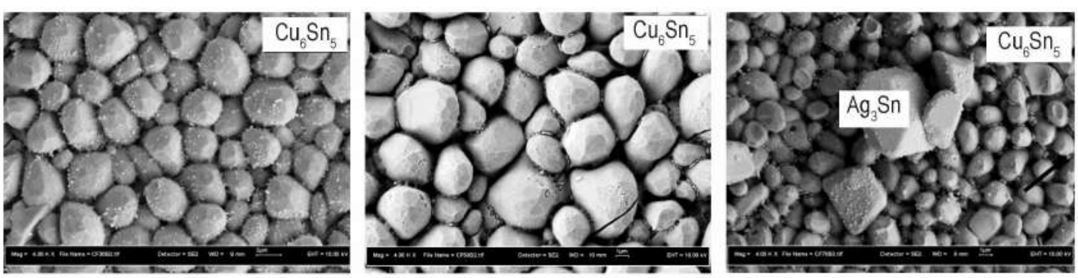
There is an extra IMC layer observed between the Cu_6Sn_5 and the Cu pad in all solder bump sizes studied after ageing for up to 500 hrs (Figure 3(b)) and 1000 hrs. This layer is confirmed by EDX analysis as Cu_3Sn. No such Cu_3Sn is observed at the interface after reflow in the cross sectional optical micrographs used in this study (Figure 3(a)). In the case of smaller solder, the bulk solder is saturated with Cu quickly, Sn supply also is limited than Cu from the substrate, the Cu_6Sn_5 layer that formed first will transform into Cu_3Sn. The same effect also happened for the other solder bump sizes which are the 500 μm and 700 μm.



(a) (b)

Figure 3 Cu_3Sn and Cu_6Sn_5 IMCs formed between lead-free solder and bare copper for 300 μm solder bump after (a) reflow soldering and (b) 500 hrs aging

For the solder joint at Cu suface, there are Cu-Sn intermetallics compound at the interface as result of the diffusion of copper into the solder after reflow soldering. However, the dissolution rates of Cu along the Cu surface are not uniform. There are areas where all the Cu atoms have not been used in the formation of Cu_6Sn_5 IMC. This Cu surplus may lead to the formation of long tubes or fibres of Cu_6Sn_5. Figure 4 shows the IMCs at the top surface on Cu surface finish after reflow with different solder bump sizes. It is clearly shown that the same IMC morphology is observed for all solder bump sizes investigated. Thus, it can be concluded that difference in solder bump size does not have an effect on the type of intermetallic compound formed between copper surface finish and lead free solder.

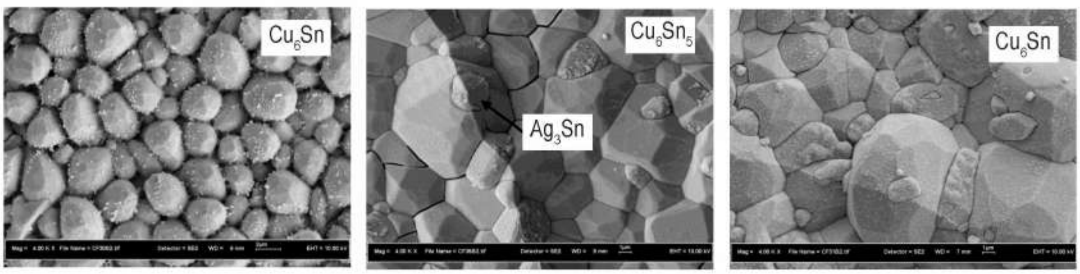


(a) (b) (c)

Figure 4 Morphology of Cu_6Sn_5 formed between Sn-Ag-Cu solder and bare copper after reflow for difference solder sizes; (a) 300 μm, (b) 500 μm, and (c) 700 μm

During isothermal aging, the IMCs continue to grow by inter-diffusion between the Sn in the solder and Cu in the substrate. This solid state aging resulted in a significant increase in the size of the Cu_6Sn_5 IMCs (Figure 5). Figure 5(b) shows that after 500 hours aging, the IMC has coarsened and became more uniform and denser as well as the IMC morphology after 1000 hours instead of the scallop-type observed after reflow soldering. Moreover, after heating the solder joint, a new IMC phase has formed between the Cu substrate and Cu_6Sn_5 IMC as shown by the thin grey layer in Figure 3(b) which is known as Cu_3Sn. In the temperature range of interest (e.g. below 350°C), the interfacial reaction with molten Sn-based solder results in the formation of $Cu_3Sn(\varepsilon)$ and $Cu_6Sn_5(\eta)$ layers [8] (Figure 6).

The same effect also happened with the solder bump sizes of 500 μm and 700 μm. In addition, there is the Ag_3Sn IMCs which was observed in solder-Cu joint on the Cu_6Sn_5. The shapes formed vary but generally they were plate-like. After aging, Ag_3Sn IMCs will become more spherical and smaller (Figure 5(b)).



(a) (b) (c)

Figure 5 Morphology on the top surface of IMCs formed between Sn-Ag-Cu solder and bare copper for 300 μm solder bump with difference aging durations; (a) as reflow, (b) 500 hrs, and (c) 1000 hrs

However, different phenomenon happened to ENEPIG surface finish. In general, at temperatures below 260°C (reflow temperature is 217°C) Ni_3Sn_4 is the first phase to form at the liquid Sn/Ni conductor interface. The first stage of the reaction is the dissolution of Ni in liquid solder, until solder is supersaturated with Ni. This is because the thin layer of gold and palladium dissolve into the bulk solder, exposing the nickel layer to react with the solder to form solder joint. In fact, there are more types of intermetallics were present at the interface of ENEPIG surface finish compared to the Bare Cu, making it much more complicated to identify the types of intermetallics that exist. This is because other than Sn, Ag and Cu from the solder also take part in the formation of intermetallic compounds after reflow.

Lead free Sn-Ag-Cu solders are capable of forming different types of intermetallics, which are present in circular boundary phases as shown in Figure 7. The contributing factor to the formation of these circular boundary regions is due to the different wetting

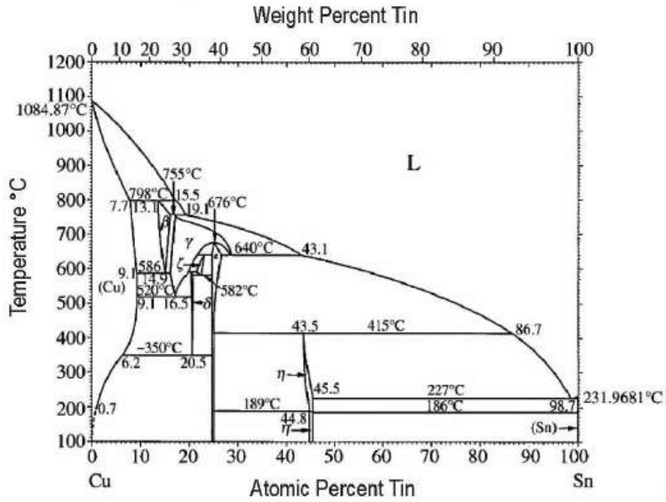


Figure 6 Cu-Sn binary phase diagram [9]

properties ranging from the edge to the centre of the solder bump. The different deep etching reaction at these regions also could be considered as one of the contribution factor.

Several intermetallics were found in the lead free Sn-Ag-Cu solder joint which are (Ni, Cu)$_3$Sn$_4$, Ni_3Sn_4, Cu_6Sn_5, and Ag_3Sn. Neither gold nor palladium-containing intermetallics were found in lead free solder joints. Although gold and palladium are also detected in EDX analysis, they are relatively very low and are usually ignored in the determination of the composition of intermetallic compounds.

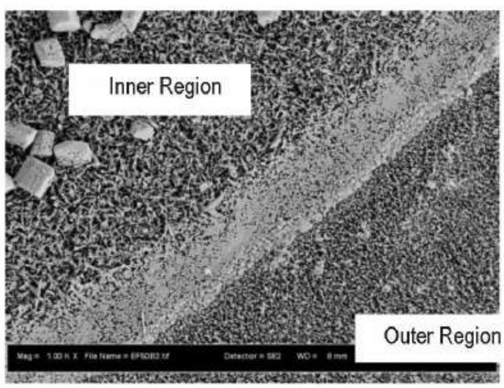
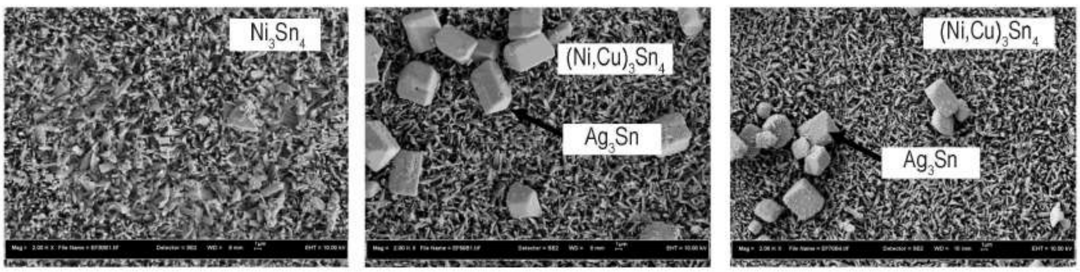


Figure 7 Different morphologies of intermetallics which form the circular boundary regions in lead free Sn-Ag-Cu solder joint

Generally, $(Ni, Cu)_3Sn_4$ and Ni_3Sn_4, take the forms of needle-like or rod shape after reflow. While blocky Ag_3Sn intermetallics were usually formed above them at the centre region. The morphologies of these intermetallics for the different solder sizes after reflow are shown in Figure 8. It was observed that Ag_3Sn were rarely found in the solder joints using 300 μm solder bumps. This is due to the fact that smaller solder volume has relatively low Ag content in the solder and thus forming less Ag_3Sn in the solder joint compared to that which uses bigger solder bump with higher solder volume.

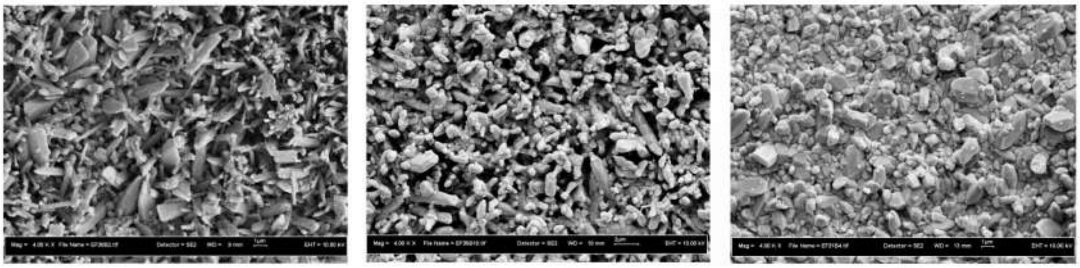


(a) (b) (c)

Figure 8 Top surface morphology of intermetallics formed in centre region after reflow using (a) 300 μm (b) 500 μm (c) 700 μm solder bumps

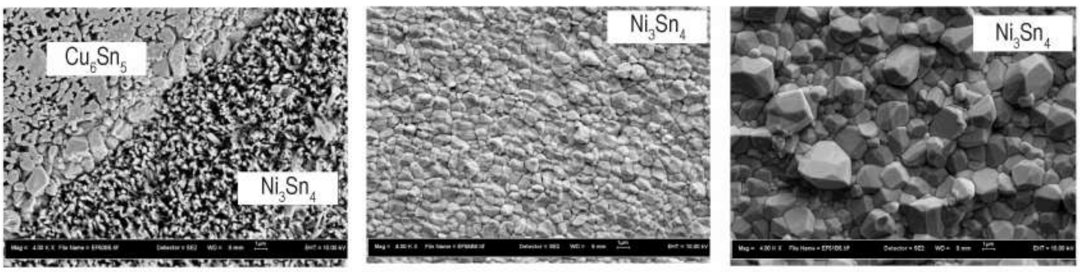
Moreover, isothermal aging will affect the morphologies of intermetallic compound in lead-free Sn-Ag-Cu solder joint. Figure 9(a) shows that the needle-like Ni_3Sn_4 were formed after reflow soldering for sample using 300 μm solder bumps. However, they changed to a more spherical and chunky shape after 500 hours aging treatment (Figure 9(b)). The intermetallics continued to grow and coarsen when subjected to further aging from 1000 hours aging treatment.

The same trend also applied to the solder joints using 500 μm and 700 μm solder bumps which experienced the transformation of intermetallics from a loose and needle-like shape to more compact, coarse and spherical shape with aging duration. Besides that, there is another intermetallic that has formed at the outer region which is known as Ni_3Sn. The same trend also applied at this region where from the needle shape of Ni_3Sn_4 after reflow to a more spherical and chunky shape after 500 hours aging treatment. The intermetallics continued to grow and coarsen when subjected to further aging from 1000 hours aging treatment (Figure 10). At circular boundary phases, it shows different intermetallic which is Cu_6Sn_5 (Figure 10(a)).



(a) (b) (c)

Figure 9 Top surface micrographs showing morphologies of in Sn-Ag-Cu solder joint using 300 μm solder bumps at inner region (a) after reflow (b) 500 hrs (c) 1000 hrs



(a) (b) (c)

Figure 10 Top surface micrographs showing morphologies of in Sn-Ag-Cu solder joint using 500 μm solder bumps at outer region (a) after reflow (b) 500 hrs (c) 1000 hrs

4.0 CONCLUSIONS

In this study, the mean thickness of the intermetallic compounds was found to be thicker in smaller solder volume compared to larger solder volume. On the other hand, ENEPIG surface finish produced thinner intermetallic compounds compared to the copper surface finish and the main type of intermetallic compounds formed are Ni_3Sn_4 and $(Ni,Cu)_3Sn_4$. The intermetallic compounds type and morphology are more or less the same for the different solder volumes investigated in this research. For both surface finishes, aging resulted in growth of the IMC in terms of overall thickness, coarsening of intermetallic compounds and also changes in morphology of intermetallic compounds to more spherical shape.

ACKNOWLEDGMENTS

The authors would like to thank Universiti Teknologi Malaysia for providing research facilities.

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