The effect of nickel doping on SAC305 Lead-free Solders and EN(B)EPIG Surface Finish

O. Saliza Azlina[1,a], A. Ourdjini[2,b] and M.A Azmah Hanim[3,c]

[1]Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia, Johor, Malaysia

[2]Faculty of Mechanical Engineering, Universiti Teknologi Malaysia, Johor, Malaysia

[3]Faculty of Engineering, Universiti Putra Malaysia, Serdang, Malaysia

[a]salizaz@uthm.edu.my, [b]ourdjini@fkm.utm.my, [c]azmah@eng.upm.edu.my

Abstract

Recently, there are many portable electronics product such as i-pad, smart phone and tablet were widely used due to growing needs of busy lives and demanding, more functionalities and compatibility. The growing of these smart technologies made electronic packaging are moving parallel with current technology and expanding rapidly because of global competitive pressure. Thus, flip chip technology is one of the important element need to be considered in order to produce high performance and quality of electronic devices due to shorter electrical connections between the chip and substrate and very high input/output capacity and the smallest possible package size. In flip chip applications, solder bump was used to connect the die to the substrate or circuit board. The addition of doping element in lead-free solder has been discussed have a big influence on the solder joint quality including solder microstructure. It also can enhanced the properties and improve the performance of interfacial reaction at interface and made a reliability of lead-free solder especially on Sn-3.0Ag-0.5Cu was increased. Therefore, this study investigates the effect of nickel doping on Sn-3.0Ag-0.5Cu (SAC305) lead-free solders and electroless nickel (boron)/electroless palladium/immersion gold (EN(B)EPIG) surface finish. In this study, two types of lead-free solders was used which are Sn-3.0Ag-0.5Cu (SAC305) and Sn-3.0Ag-0.5Cu-0.05Ni (SACN30505) with solder size diameter of 500 µm in order to examine the effect of nickel on interfacial reaction during soldering process. Reliability of solder joint has been assessed by performing solid state isothermal aging at 125°C for 250, 500, 1000 and 2000 hours. Several characterization techniques will be conducted including image analyzer, optical microscope, field emission scanning electron microscopy and energy dispersive x-ray analysis to characterize the intermetallic formed. After reflow soldering process, it was found that the $(Cu,Ni)_6Sn_5$ and $(Ni,Cu)_3Sn_4$ intermetallic compound (IMC) is formed at interface. $(Ni,Cu)_3Sn_4$ dominates in the outside of solder joint while $(Cu,Ni)_6Sn_5$ dominates in the centre of joints. Besides that, after soldering and isothermal aging process, SAC305 solder with additions of 0.05% of Ni (SACN30505) made the intermetallics grew slightly faster than in the solder without Ni additions. Hence, analysis by optical microscope revealed that the IMC thickness of the SACN30505 solder produced thicker IMC compared to SAC305 for both situations. This observation is confirmed by the increase in grain size of intermetallics with Ni additions. Moreover, aging time resulted in an increase in thickness and changed the morphology into more spherical, dense and large grain size. In addition, the results also revealed that the thickness of intermetallics formed is proportional to the aging duration.

1. Introduction

As electronic packaging technology evolves, flip-chip interconnect becomes popular in dealing with requirements of high input/output (I/O) connect density and high operating frequency. Recently, nearly all large scale integrated circuit (IC) chips are packaged with flip-chip interconnect joints. The chip is flipped over and the active (front) side is connected to the package substrate using a large number of tiny solder joints [1].

The Sn-Ag-Cu (SAC) family have risen through the ranks and popular as the most promising candidates for miroelectronic application because of its excellent properties. However, trace rare earth (RE) elements were also added into lead-free solder alloys to form better solder alloys. With the addition of RE, the properties such as microstructure, mechanical and wetting behavior will improved [2,3]. Nickel (Ni) is one of the RE element which can show marked solubility in the intermetallic compound layer [4,5]. Indeed, Ni has attracted a great deal of interest as an alloying addition to lead-free alloys due to its excellent performance in improving solder microstructure, increasing the drop lifetime of electronic assembly, as well as their comparatively low cost [6].

The selection of surface finish on copper is very important in soldering process. Electroless nickel such as nickel phosphorous (Ni-P) has been widely used in ENIG and ENEPIG as surface finish on printed circuit board. However, nickel boron (Ni-B) also has been received considerable attention in interconnection because it reacts in the same manner with Ni-P [7,8].

Therefore, this study is performed to examine the effect of doping element (0.05wt% Ni) in the Sn-3.0Ag-0.5Cu (SAC305) lead-free solder on intermetallic growth and thickness, where EN(B)EPIG (electroless nickel-boron/ electroless palladium/immersion gold) as a surface finish. Besides that, the effect of different aging duration on interfacial reaction during reflow soldering and isothermal aging also has been investigated.

2. Experimental Procedure

The soldering reaction between Sn-3.0Ag-0.5Cu (SAC305) and Sn-3.0Ag-0.5Cu-0.05Ni (SACN30505) solders ball and Ni-B (EN(B)EPIG) surface finish were examined in this study. The copper polymer sandwich substrate (FR-4) with dimensions 45 x 50 x 1 mm was prepared and then was subjected to a pretreatment process in order to remove oxides and activate the copper substrate surface before the electroless Ni-B/electroless Pd/immersion Au (EN(B)EPIG) plating process is started. The Ni-B solution was conducted at 85°C and after that, electroless palladium were applied on Ni-B layer and followed by deposited with gold layer through immersion plating without any pretreatment except rinsing in running tap water with temperature was set up at 45°C and 93°C respectively. Then, all samples were laminated with a layer of solder mask to restrict the molten solder from flat spreading during reflow. Next step is the solder mask together with the patterned film was cured by ultraviolet (UV) light in order to produce small openings. After curing samples, a thin layer of no-clean flux is applied onto the substrate to remove the oxide layer and also to improve the wetting of molten solder during reflow. Then, the substrates were manually populated with solder balls with a diameter of 500µm arranged in several rows. Bonding to form the solder joints was made by reflow soldering in a furnace at temperature ~230°C. Then, each sample was subjected to aging treatment at 125°C for 250 hours, 1000 hours and 2000 hours. Characterisation of samples involved both at top surface and cross section of solder joints. Several techniques including optical microscopy, NIKON optical microscope, scanning electron microscopy (SEM) and energy dispersive x-ray analysis (EDX), image analyzer and field emission scanning electron microscope (FESEM) were used for the intermetallics characterization.

3. Results and discussion

In soldering process, the interfacial reaction will be occurred between solders and substrate where the solder joint known as intermetallic compound (IMC) will be formed at the interface. Fig. 1 shows top surface views of intermetallic compound (IMC) on EN(B)EPIG after reflow for SAC305 and SACN30505. From EDX spectrum analysis represents that the IMC formed at interface were $(Ni,Cu)_3Sn_4$ and $(Cu,Ni)_6Sn_5$. $(Cu,Ni)_6Sn_5$ dominates in the centre of joints in fig 1(a) and fig 1(c) while $(Ni,Cu)_3Sn_4$ dominates in the outside of solder joint as can be seen in fig 1(b) and fig 1(d). Besides that, needle-like $(Ni,Cu)_3Sn_4$ was also observed underneath the $(Cu,Ni)_6Sn_5$ IMC shows in Fig 1(a). The formation of $(Cu,Ni)_6Sn_5$ at the interface is consistent with the results of previous studies (Laurila *et al.*, [9], Ho *et al.*, [10] and Ho *et al.* [11]) where the concentration of Cu in the solders is greater than or equal to 0.5wt.%.

Fig. 2 shows the cross sections view of IMC on EN(B)EPIG after isothermal aging at 125°C for 1000 hours. The effect of aging treatment on intermetallics at the interface can be cleared examined by cross-sectioning of solder joints, selectively etching the solder away so that IMC morphology can be revealed. As is clearly shown in fig 2 (a) and fig 2(b) for SAC305 and SACN30505 on EN(B)EPIG substrates respectively, aging process increases the intermetallics is more leveled. This is because of the formation of palladium tin intermetallics at the interface. For the formation of intermetallics, the copper and tin elements must diffuse through the palladium tin intermetallics to form Cu_6Sn_5 based intermetallic at the interface even after aging for 2000 hours. This will slow down the diffusion process, and automatically turns the palladium tin intermetallics layer into a diffusion barrier layer [15].

Besides that, SACN30505 shows thicker intermetallics layer than SAC305. The present results reconcile well with the findings of Yao *et al.* [14] who found that the interfacial layer $(Ni,Cu)_3Sn_4$ grew more thicker with the presence of Ni when exposed to isothermal aging, and that the interfacial $(Cu\ Ni)_6Sn_5$ layer decreased correspondingly.

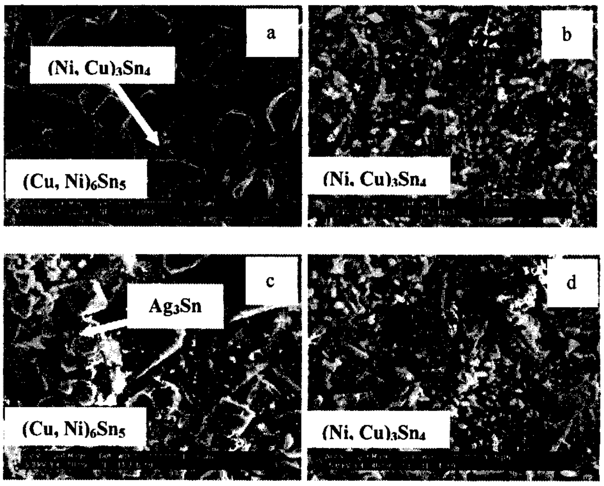

Fig.1 SEM top surfaces of IMC on EN(B)EPIG after reflow with solder size of Ø500 (a) SAC305 in center, (b) SAC305 near outside, (c) SACN30505 in near center,(d) SACN30505 in near outside.

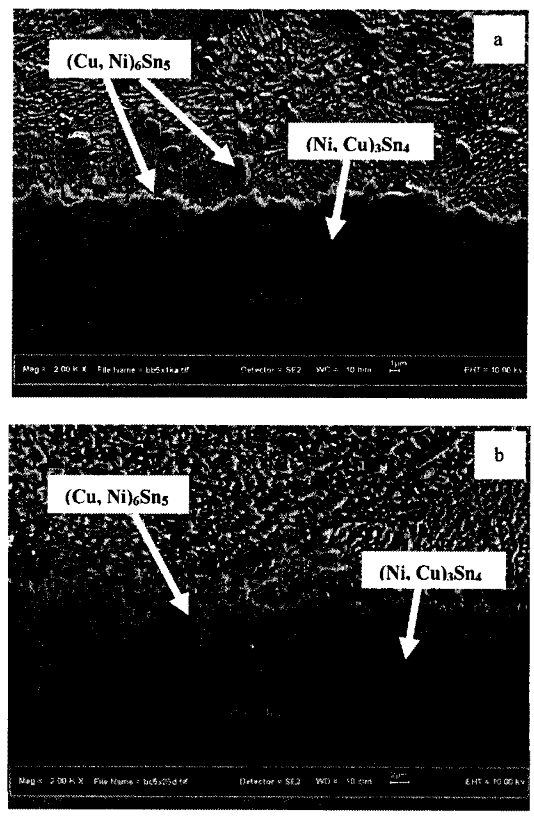

Fig. 2 FESEM Cross sections of IMC on EN(B)EPIG after aging at 125°C for 1000 hours: (a) SAC305 (b) SACN30505

As mentioned before, when the substrate is exposed to isothermal aging, the intermetallic formation (IMC) formed during reflow soldering will grow continually but at a much slower rate. According to ternary diagram, both IMCs, $(Cu,Ni)_6Sn_5$ and $(Ni,Cu)_3Sn_4$ are based on the Cu_6Sn_5 and Ni_3Sn_4 crystal structures, respectively where $(Cu, Ni)_6Sn_5$ and $(Ni, Cu)_3Sn_4$ are stable ternary formed at the interface. Fig 3 represents SEM top surface views of EN(B)EPIG aged at 125°C with solder size Ø500 at near center of solder. It is clear from these micrographs that the effect of aging did not induce changes in the type of IMC formed but the morphology has become more spherical, compact, dense and large grain size.

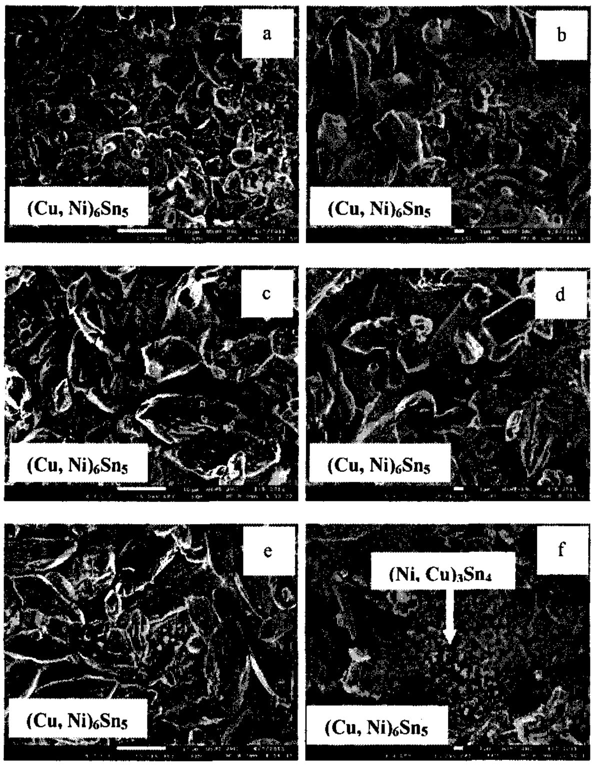

Fig 3 SEM top surface of EN(B)EPIG aged at 125°C with solder size Ø500 at near center of solder (a) SAC305 aged at 250 hours, (b) SACN30505 aged at 250 hours (c) SAC305 aged at 1000 hours (d) SACN30505 aged at 1000 hours (e) SAC305 aged at 2000 hours (f) SACN30505 aged at 2000 hours

Moreover, there is also evidence that the formation of duplex IMC layers, $(Ni,Cu)_3Sn_4$ and $(Cu,Ni)_6Sn_5$ is maintained as shown in Fig 3(f). These morphological evolutions of IMC's are similar to the findings of Yoon *et al.* [12] and Wang et *al.* [13]. These authors reported that with longer reflowing time (up to 60min), $(Ni,Cu)_3Sn_4$ IM3C showed extensive growth by reaction of the molten solder and Ni substrate layer. In addition, the $(Cu,Ni)_6Sn_5$ IMCs were sequentially transformed into $(Ni,Cu)_3Sn_4$ IMCs during prolonged reflow times, due to the limited Cu supply from the solder and continuous Ni diffusion from the Ni layer. They concluded that growth of the $(Ni,Cu)_3Sn_4$ IMC consumed the $(Cu,Ni)_6Sn_5$ IMC during prolonged reflow reactions and that the remaining $(Cu,Ni)_6Sn_5$ IMCs were embedded with $(Ni, Cu)_3Sn_4$ IMCs.

By observing the qualitative data, the interfacial IMC layer formed when soldering SAC305 on EN(B)EPIG finished substrate is thinner than SACN30505 solders as illustrated in Fig 4. From the graph, it can be seen that with increased addition of Ni percentage, the IMC thickness also increased significantly during reflow soldering as well as isothermal aging. The results in the present study are in agreement with those reported by Yao *et al.* [14].

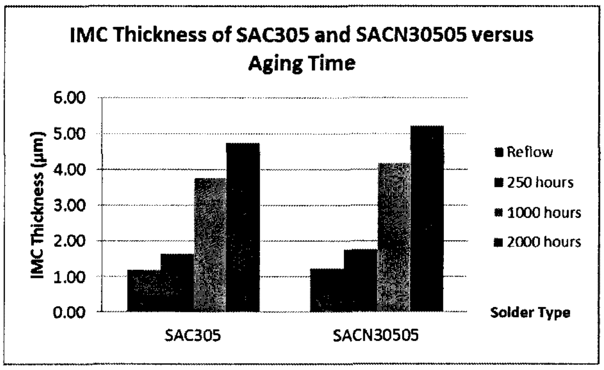

Fig 4 IMC Thickness of SAC305 and SAC3050.05Ni on EN(B)EPIG at the aging temperatures 125°C

4. Conclusions

In the as-reflow condition the major intermetallics formed between SAC305 and EN(B)EPIG were $(Ni,Cu)_3Sn_4$ and $(Cu,Ni)_6Sn_5$. $(Ni,Cu)_3Sn_4$ dominates in the outside of solder joint while $(Cu,Ni)_6Sn_5$ dominates in the centre of joints. After soldering and isothermal aging process, SAC305 solder with additions of 0.05% of Ni (SACN30505) made the intermetallics grew slightly faster than in the solder without Ni additions. Hence, IMC thickness of the SACN30505 solder produced thicker IMC compared to SAC305 for both situations. This observation is confirmed by the increase in grain size of intermetallics with Ni additions. Besides, aging time resulted in an increase in thickness and changed the morphology into more spherical, dense and large grain size and made intermetallics formed is proportional to the aging duration

Acknowledgments

This work was financially supported by Universiti Tun Hussein Onn Malaysia (UTHM), Universiti Teknologi Malaysia, Microelectronics and Nanotechnology-Shamsuddin Research Centre (Mint-SRC, UTHM), Faculty of Mechanical and Manufacturing Engineering (FKMP, UTHM) and Advanced Manufacturing and Materials Centre (AMMC, UTHM) for providing the research facilities.

References

1. Chu-Hsuan Sha & Chin Lee, C. Demonstration of silver micro-joints for flip-chip interconnects. Microelectronic Engineering. Volume 99, November 2012, pp. 11–17

2. Wu C.M.L et al. Improvements of microstructure, wettability, tensile and creep strength of eutectic Sn–Ag alloy by doping with rare-earth elements. Journal of Materials Research. Vol 17. 1292002, pp. 3146-3154
3. Yu, D.Q et al. Improvement on the microstructure stability, mechanical and wetting properties of Sn–Ag–Cu lead-free solder with the addition of rare earth elements. Journal of Alloys and Compounds. Volume 376, 1-2 (2004), pp. 170-175
4. Wang, L, et al. Morphology and Growth Mechanisms of SAC305-xNi/pad joints Intermetallic Compounds. 2010 11th International Conference on Electronic Packaging Technology & High Density Packaging (2010)
5. Laurila, T., et al.. Impurity and alloying effects on interfacial reaction layers in Pb-free soldering. Materials Science and Engineering. Vol. 68 (2010), pp.1–38.
6. Che F.X. The study of mechanical properties of Sn–Ag–Cu lead-free solders with different Ag contents and Ni doping under different strain rates and temperatures. Journal of Alloy Compoundd. Vol 507 (2010), pp. 215–224
7. Saliza Azlina, O. et al., Effect of Solder Volume On Interfacial Reaction Between SAC405 Solders and EN(B)EPIG Surface Finish, Advanced Material Research Vol. 845, (2014), pp. 76-80.
8. Tsukimura, M. et al, (2002) Materials Transactions. Vol. 43, pp 1615-1620.
9. Laurila, T., et al,. Interfacial Reactions between Lead-Free Solders and Common Base Materials. Materials Science and Engineering. Vol 49 (2005), pp 1-60
10. Ho, C.E. et al,. J. Electron. Mater. Vol. 31, (2002) pp 584
11. Ho, C.E., et al J. Mater. Sci.: Mater. Electron. Vol 18, (2007), pp. 155
12. Yoon, J.W et al. Sequential interfacial intermetallic compound formation of Cu_6Sn_5 and Ni_3Sn_4 between Sn–Ag–Cu solder and ENEPIG substrate during a reflow process. Journal of Alloys and Compounds. Vol 509. (2011): pp. L153-L156
13. Wang, L, et al. Morphology and Growth Mechanisms of SAC305-xNi/pad joints Intermetallic Compounds. 2010 11th International Conference on Electronic Packaging Technology & High Density Packaging (2010)
14. Yao, P. et al.. Interfacial reaction and shear strength of SnAgCu–xNi/Ni solder joints during aging at 150C. Microelectronic Engineering. Vol 86. (2009), pp. 1969–1974.
15. Gaurav S.& K. Vaidyanathan, Kinetics of Intermetallics Growth between Lead Free Solders and Silver-Palladium Metallizations, Proceedings of Electronics Packaging Technology Conference. (2006). pp. 190-195