

MICROSTRUCTURE AND METALLURGICAL PROPERTIES OF LOW TEMPERATURE SNBI SOLDER JOINT AT DIFFERENT REFLOW SOLDERING TEMPERATURES

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Abstract

With the rise in trend of miniaturisation of electrical components and the ban on lead-based solders, low melting temperature solders are in demand to minimize damage to electrical components. SnBi solder has gained attention for its excellent mechanical and electrical properties as well as its low melting point. There is minimal research done on the effect of reflow temperature on the microstructural and metallurgical properties of SnBi/Cu solder joint. Existing conflict on the understanding of the behaviour of bismuth during the reflow soldering process motivates the initiation of this research. This paper aims to close the gap in research regarding the SnBi/Cu solder joint by analysing the microstructural and metallurgical properties of SnBi/Cu solder joint subjected to three different reflow temperatures. It was discovered that at 160 °C, there was no IMC present due to insufficient temperature. IMCs were present at 170 °C and 180 °C, with Cu₆Sn₅ being present at both reflow temperatures and Cu₃Sn being present only at 180 °C as revealed by the EDS. The average thickness of Cu₆Sn₅ was 0.909 and 1.404 µm at 170 °C and 180 °C respectively, where it was found to have nearly a 54% increase in thickness. The findings show the possibility of adapting the SnBi/Cu for a low temperature solder alternative.

Keywords: Cu₆Sn₅ IMC, Low temperature soldering, Metallurgical, Microstructure, SnBi solder joint.

1. Introduction

Soldering is defined as the process where metal parts are joined together using a filler metal, also known as a solder, at temperatures below 450 °C [1]. Intermetallic compounds (IMC) will form at the contact surface between the solder and metal part, and solder connection will allow for facilitation of electrical connections [1, 2]. Reflow soldering is a type of soldering process which involves the melting of solder paste to form joints between components and the printed circuit board (PCB) without modification to the original attributes. There is a growing importance for reflow soldering, which is steadily replacing traditional manual soldering process in production as the result of miniaturisation of electronics product.

The reflow soldering profile is often generated to configure the heating parameters and control the effect of heating on the PCB boards. Generally, there are four independent controlled heating zones: preheating, soaking, reflow and cooling zones. Significant errors and defects are concentrated at the reflow soldering process, where defects such as warped areas, tin (Sn) particles, bridges, PCB delamination and blistering occur [3]. Therefore, optimization of the temperature profile has been a major area of investigation for the improvement of solder joint quality [3].

A reliable solder should consist of low melting temperature, good wettability and a good hardness value [4]. Traditionally, eutectic tin-lead (SnPb) alloy was an ideal choice because of its favourable properties. However, the rising concern of environmental impact and human health issues caused by the toxicity of lead solders have led to regulatory actions being implemented. As a result, there has been substantial effort on the research of lead-free solders as an alternative for the traditional lead solders [5]. The aim of lead-free solder material is to possess properties that either suppress or have equal capabilities of lead-based alloys and fulfil the demands for the electronics industry [6].

Solders possessing low melting points effectively addresses this issue by allowing lower reflow temperature, providing greater technological flexibility for upcoming packaging structure. Recent research has identified tin-bismuth (SnBi) as a potential environmentally friendly alternative solder material due to its low melting point (138 °C) and favourable properties, including high yield strength (25–35 MPa), cost-effectiveness and strong reliability [7]. Experimental investigations on reflow process on SnBi can offer additional insight on the microstructural and metallurgical properties of the solder joint formed. Jaynesh and Elias [8] concluded at higher temperatures at 185 °C, porous Cu₃Sn will form, while it will not form at 170 °C.

Therefore, the reflow temperature selected will influence the type of IMC formed. Zou et al. [9] performed an investigation on six reflow temperatures ranging from 180 to 300 °C and its effect on SnBi/Cu solder joint properties, where the results suggest that an increase in reflow temperature result in significant transition of Bi segregation. The findings from this study can serve as valuable contribution towards the reduction in defection occurring during the reflow process, enhancing soldering quality and increasing manufacturing efficient in the microelectronics industry while fulfilling the criteria of environmental sustainability [5, 10].

Research on the properties of SnBi/Cu solder joint remain limited despite its critical role in determining the reliability of the electronic product. This research revolves in investigating the SnBi/Cu solder joint's microstructural and metallurgical subjected to three different reflow temperatures.

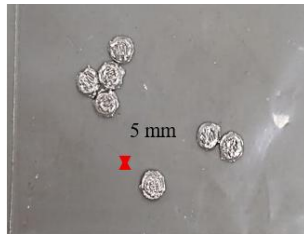
2. Materials and Method

A 10.5 cm by 10.5 cm mold, which consisted of sixteen circle protrusion with a diameter of 0.5 cm with a thickness of 0.1 mm, was designed in SOLIDWORKS and 3D printed. The mold is designed to ensure consistency of size and volume of the solder billets for testing purpose. 50 grams of eutectic SnBi was placed into the ceramic crucible and heated to 300 °C using a hotplate for five minutes. The melted solder was then poured into the mould and left to solidify at room temperature for one hour. The billets were then removed from the mould and sandpapered to remove the excess material as shown in Fig. 1(a).

Five milligrams of SnBi were prepared for thermogravimetric (TGA) analysis. The TGA analysis was performed from 100 °C until 300 °C, with a heating rate of 10 °C/min under purified oxygen for 20 minutes. The TGA analysis has concluded several aspects of thermal properties. Samples 1, 2 and 3 were reflowed at 160 °C. Samples 4, 5 and 6 were reflowed at 170 °C, while samples 7, 8 and 9 were reflowed at 180 °C. The reflow process is completed with three reflow temperatures: 160 °C, 170 °C and 180 °C. Soldering was performed three times at each reflow temperature. A thermologger attached to a thermocouple is placed onto the copper (Cu) substrate.

Hoboware software was used to collect the temperature of the Cu substrate with a one second interval as in the setup shown in Fig. 1(b). The soldering process was done using the hotplate. The hotplate is heated to 100 °C and sustained for 120 second. After 120 seconds, the temperature is raised to the reflow temperature and is sustained for 45 seconds. After the limit, the hotplate is then switched off to allow the SnBi/Cu piece to cool down. Figure 1(c) shows the soldered sample of SnBi and Cu.

The solder joints were cold mounted to accommodate the metallurgical analyses. The sectioned samples of cold mount shown in Fig. 1(d) are finely grinded and polished for 5 minutes at 150 rpm using a grinding and polishing machine. The scanning electron microscopy (SEM) to capture microstructural image of the SnBi/Cu solder joints and the image's elemental composition was analysed using the elemental dispersive spectrum (EDS).



(a) SnBi billet.



(b) Soldering setup.



(c) SnBi/Cu soldered piece.



(d) Sectioned sample.

Fig. 1. SnBi sample preparation and setups.

3. Results and Discussion

3.1. Thermogravimetric analysis (TGA)

TGA results are as shown in Fig. 2. Upon heating and exposure to oxygen gas, the sample experiences weight loss from the initial weight of 2.825194 mg to 2.683968 mg over a period of 20 minutes at a heating rate of 10 °C/min. The overall percentage loss in mass is calculated as shown in Eq. (1).

$$\frac{2.683968 - 2.825194}{2.825194} \times 100 = -4.99881\% \quad (1)$$

A 5% decrease in mass is seen over a period of 20 minutes. The resulting mass could be because of the evaporation of surface water adsorbed and from the decomposition of impurities in the sample. The sample is said to be 99.95% in purity and along with impurities obtained from handling and during the creation of the solder, which may have accounted for. This agrees with investigations carried out by Rami et al. [11], Bouzerafa et al. [12], and Fruth et al. [13], where the investigation shows that at similar temperatures, mass loss was the result in the evaporation of water and decomposition of impurities.

The decrease in mass is not steady though, instead, a sigmoidal weight loss is observed, as seen with the S-shaped curve, illustrated in Fig. 3. The maximum rate of mass loss occurs at the steepest part of the curve and is found through the analysis of the first derivative. 3. For analysis, mass changes with temperature are studied. The rate of change is using Eq. (2).

$$\text{mass change} = \frac{\text{change in mass (mg)}}{\text{change in temperature (°C)}} \quad (2)$$

The tabulated data shows that mass loss occurs at 130 to 140 °C when a consistent negative trend starts to form. The area of maximum mass loss occurs between 170 to 230 °C. After 230 °C, the rate of mass loss begins to decrease. Eutectic SnBi has a melting point of 138 °C, which provides an explanation for the onset of decline of sample mass. This can be seen in the onset temperature graph, where 138 °C is determined to be the beginning of mass loss. Common impurities in metal include sulphur, phosphorus, oxygen, and nitrogen. When SnBi begins to melt, the volatile impurities may start to evaporate, resulting in mass loss. Rapid mass loss occurs during 170 to 230 °C, with the peak at 210 °C, when the sample has fully melted and liquefied. After 200 °C, rate of mass loss reduces, indicating that majority of the volatile impurities with low melting points have been evaporated or decomposed.

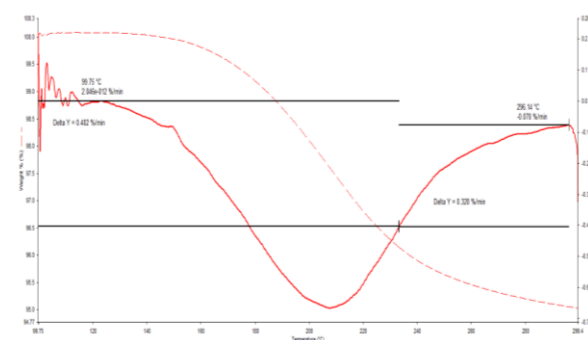


Fig. 2. Rate of change of mass.

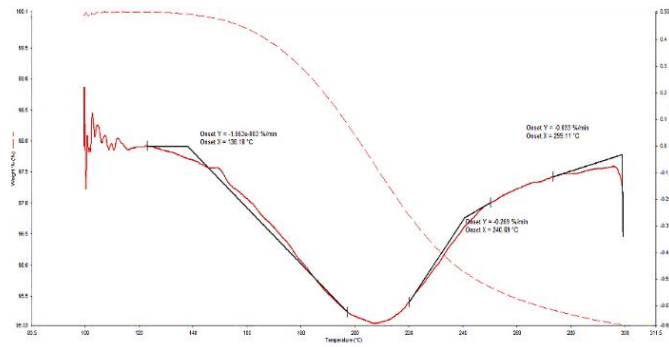
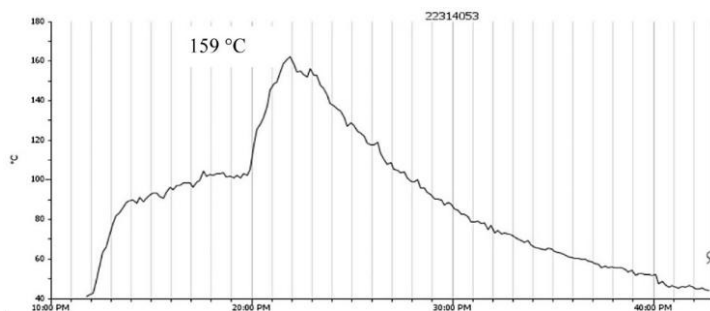


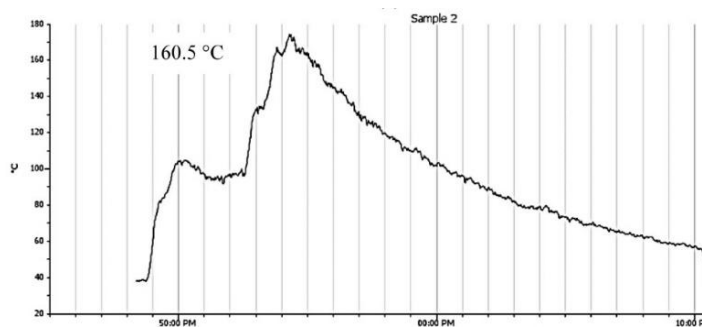
Fig. 3. Thermal stability.

3.2. Soldering temperature profile

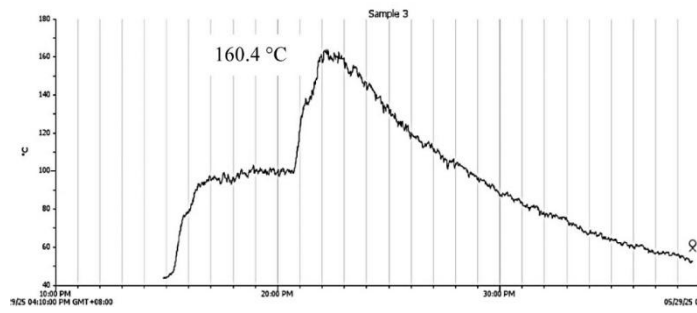
The results from the soldering process are shown in Figs. 4. The experiment conducted is to simulate the conditions to generate results like the actual reflow temperature profile. The average temperature of the reflow process was calculated for each sample and tabulated in Table 1. Cooling rate and reflow temperature have significant effect on the microstructural and metallurgical properties of the solder joint. The cooling rate will impact the growth rate of β -Sn and the grain features [14-16]. To ensure precision of experiment, the cooling rate for all samples are at approximately 7.5 °C/min. Results show a cooling rate between 5 °C/min to 10 °C/min, which is an acceptable difference to maintain accuracy for this investigation.



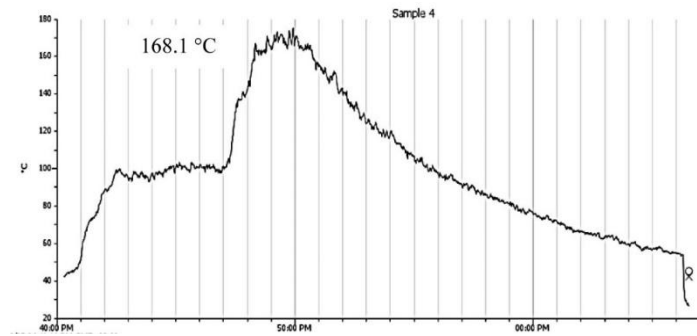
(a)



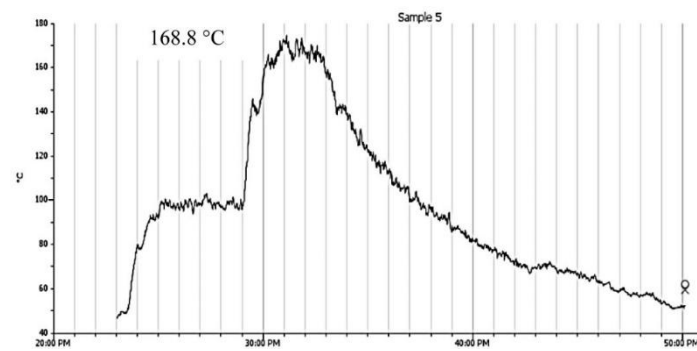
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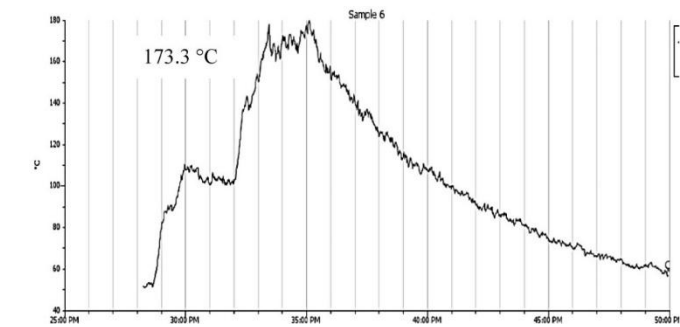
(c)



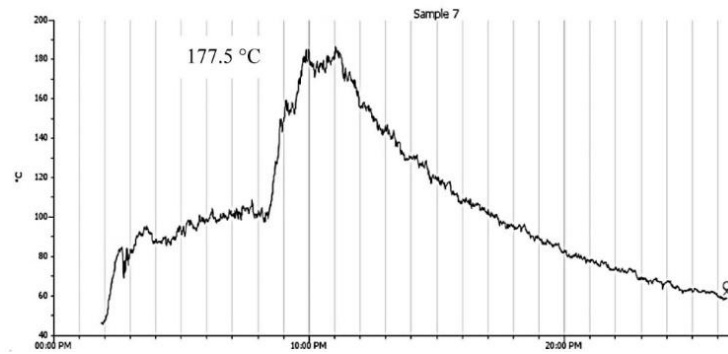
(d)



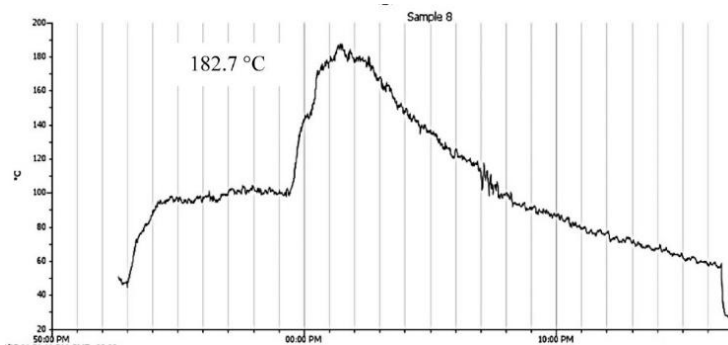
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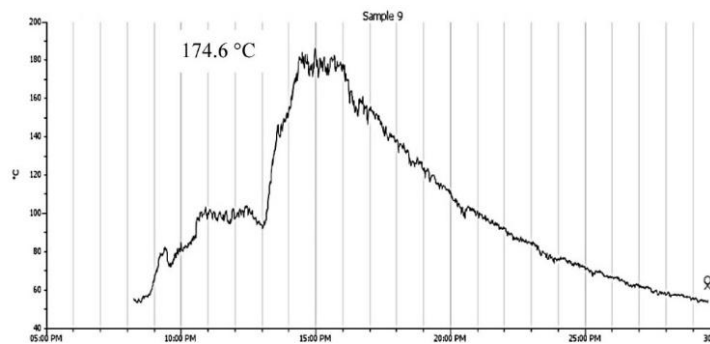
(f)



(g)



(h)



(i)

Fig. 4. Reflow profile of samples (a-c) at 160 °C, (d-f) at 170 °C and (g-i) at 170 °C.

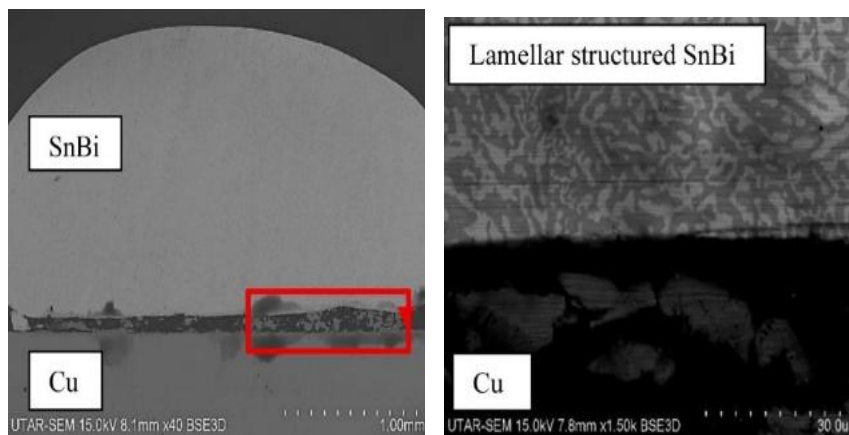
The average temperature of the reflow process was calculated for each sample and tabulated in Table 1. Cooling rate and reflow temperature showed significant effect on the microstructural and metallurgical properties of the solder joint. The cooling rate will impact the growth rate of β -Sn and the grain features [14-16]. To ensure precision of experiment, the cooling rate for all samples is at approximately 7.5 °C/min. Results show a cooling rate between 5 °C/min to 10 °C/min, which is an acceptable difference to maintain accuracy for this investigation.

Table 1. Average soldering temperature and cooling rate.

Sample	Average Soldering Temperature, (°C)	Average Cooling Rate (°C/min)
1	159.00250	5.42005
2	160.52000	7.56360
3	160.36543	6.70647
4	168.10931	8.60502
5	168.83891	6.30652
6	173.34435	7.38111
7	177.47630	7.49489
8	182.65717	10.19669
9	174.64326	8.34332

3.3. Microstructural and metallurgical properties

The IMC formation and thickness are a crucial aspect in the reliability of a solder joint. When observed using the SEM graphics, it was found that there was no IMCs form between the solder and substrate. Figure 5 which is also the SEM image of the solder joint at 160 °C depicts the overall image of SnBi/Cu substrate, with the bright white part of the structure consisting of Bi elements and the grey part is the Sn elements.

**Fig. 5. SnBi/Cu solder joint at 160 °C.**

The region between the SnBi solder and substrate at the 160 °C show complete contrast, suggesting that no or negligible IMCs are formed. The point EDS analysis at the point in Fig. 6 reveal that the contrasted area consisted mostly of carbon particles with no minimal Sn, Bi or Cu elements, indicating that the completely contrasted areas consist of voids. There are several reasons for this possibility. Firstly, the formation of voids is the result of the trapped gas in the molten solder. During the soldering process, the flux is heated. Decomposition of flux will occur, and the gas is released. However, at 160 °C, incomplete outgassing may occur as the temperature is too low to fully activate the flux, causing the gas to be trapped when the solder solidifies. Furthermore, the presence of impurities can further exacerbate the formation of voids by preventing good solder wetting, leading to gas entrapment.

Growth kinetic theory had originally suggested that SnBi follows a parabolic principle, whereby the growth rate of IMC will initially accelerate rapidly before slowing down [17]. Therefore, at 138 °C and above, rapid formation of IMC begins. However, the theory may not hold universally as various factors can influence the diffusion process, leading to different behaviours than previous theorised. Yu et al. [18] and Zeng et al. [19] noted that formation of voids reduce the parabolic growth rate of the at the void effected interfaces.

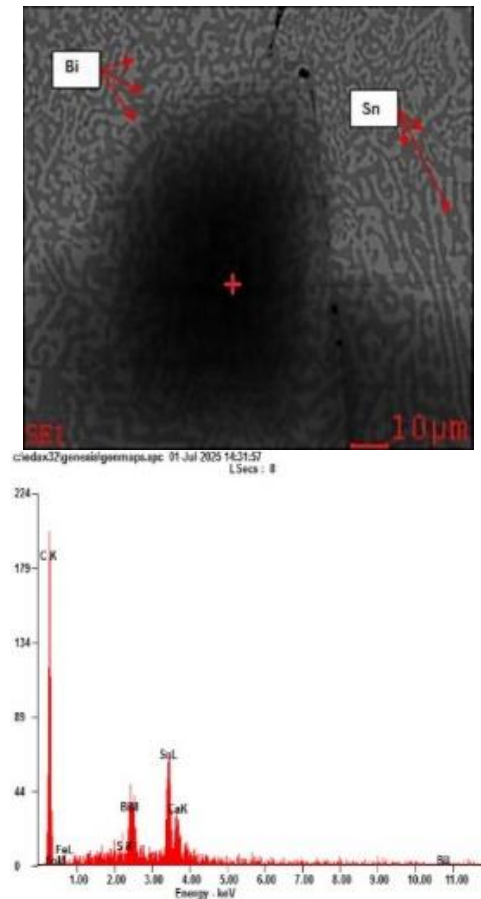


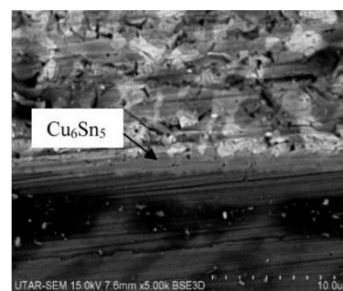
Fig. 6. SEM image and EDS SnBi/Cu solder joint at 160 °C.

The results obtained suggests that the formation of voids due to the insufficient activation of flux have prevented proper formation of IMCs. Furthermore, 160 °C is above the melting point of the solder, allowing eutectic SnBi to melt, however, it may be suboptimal for the solder to react with the Cu substrate to form complete visible IMCs. IMCs are supposedly formed at the interface, where the Sn atoms and the Cu atoms start to diffuse into each other. Theoretically, diffusion is a thermally activated process therefore higher temperature will result in a higher diffusion rate. In this investigation, the SnBi may have melted, but it had insufficient energy for diffusion to form the IMC layers. Along with the formation of voids, the conditions for IMC formation were suboptimal.

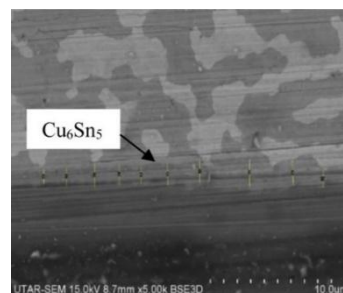
Figure 7 shows the cross-section of SnBi/Cu solder joints heated at 170 and 180 °C respectively. The EDS analyses at the point in Fig. 8(a) and Fig. 8(b) reveals the presence of Cu_6Sn_5 at the interfaces. During the reaction of the between the Cu substrate and SnBi solder, Cu_6Sn_5 is produced as per Eq. (3). As soldering time or temperature increase, the Cu atoms are consumed for the growth of Cu_3Sn IMC as seen in Eq. (4) and Cu_6Sn_5 will begin to transform into Cu_3Sn as in Eq. (5). This was observed at the SnBi/Cu solder joint at 180 °C.



The formation of IMC signals good bonding between the substrate and solder, but excessive bonding is detrimental to the reliability of the solder. Cu_3Sn IMC layer was not found in the morphology of 170 °C due to the low temperature and short reflow soldering time during the reflow soldering process. This is aligned with the research conducted by Kang et al. [20] where only Cu_6Sn_5 was found after reflow soldering at 170 °C for 5 minutes. This indicates good reflow soldering as the appearance of Cu_3Sn will decrease the structural integrity of the solder joint because of its weak mechanical properties. However, Cu_3Sn was found to appear at 180 °C as seen in the EDS. Shang et al. [21] similarly noted such IMC formation. However, the Cu_3Sn layer formed during the reflow soldering process was very thin and was unable to be captured in the SEM imaging but could only be identified through the EDS as seen in Fig. 8(c). The IMCs at both 170 and 180 °C had relatively smooth morphology, with rounded, lumpy scallop-like curve shape.

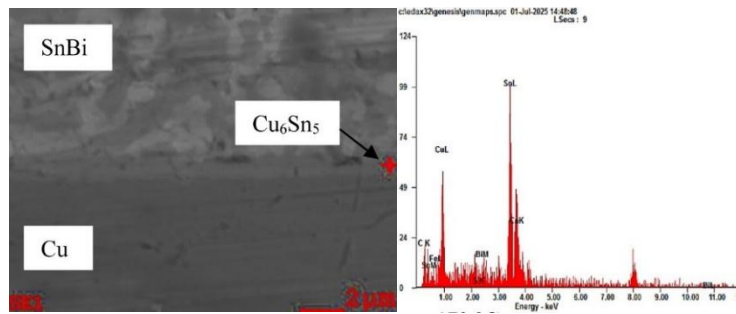
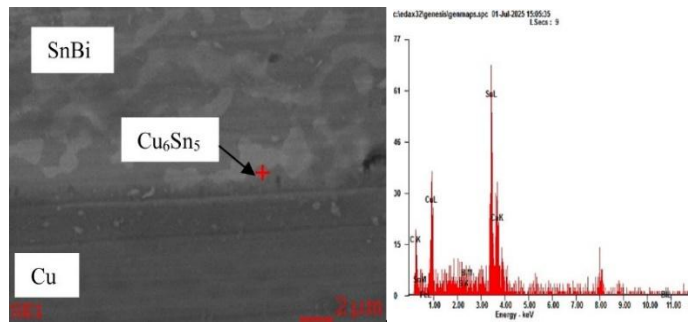
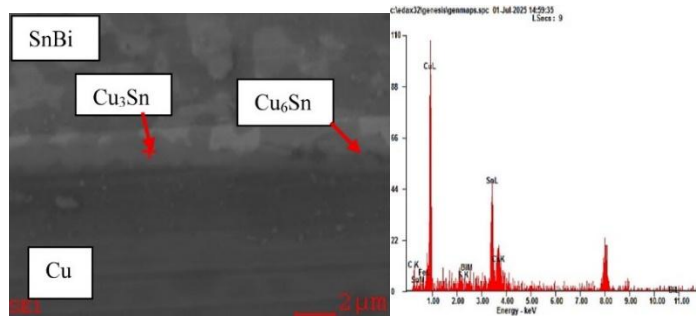


(a) 170 °C



(b) 180 °C

Fig. 7. Interfacial microstructure of SnBi/Cu.

(a) Cu₆Sn₅ at 170 °C.(b) Cu₆Sn₅ 180 °C.(c) Cu₃Sn and Cu₆Sn₅ at 180 °C.**Fig. 8. IMC layer and EDS result of SnBi/Cu solder joint.**

The IMC layer thickness increases with the increase in reflow temperature, as shown in Fig. 9. At 170 °C, the average thickness of the Cu₆Sn₅ layer was 0.909 μm and at 180 °C, the average thickness was 1.404 μm, showing an increase of 54.45%. This aligns with the theory whereby the IMC thickness increases with the increase in temperature [22]. The study conducted by Kang et al. [23] shows the average thickness of the Cu₆Sn₅ layer was 2 μm after reflowing for 5 minutes, which had better control compared to 180 °C which rapidly grows during short reflow times. Furthermore, a parabolic growth rate can also be seen, where rapid growth is seen during the initial phases of IMC formation. A 54.45% increase in thickness is excessive, as solder joints in the microelectronics industry are often subjected to thermal aging, which further facilitates the formation of IMCs.

From the EDS analysis of 180 °C, some Cu₃Sn IMCs were found to exist more on the SnBi solder side rather than the Cu substrate side. This phenomenon can be attributed to the diffusion theory, where Cu will typically have a higher diffusion rate and will diffuse to the solder alloy matrix [21]. Excess Cu may have diffused to the solder side during the reflow soldering process, where it is consumed by the Sn atoms to form Cu₃Sn IMCs. The SEM images show a distinctive visualization of IMC changes with the change in temperature. The growth kinetics of the Cu-Sn IMCs can be calculated from the IMC thickness, shown in Eq. (6).

$$X_t = \sqrt{Dt} \quad (6)$$

where X_t is the IMC thickness at time t , and D is the diffusion coefficient.

At 170 and 180 °C, the diffusion coefficient of SnBi/Cu is 0.0184 $\mu\text{m}^2/\text{s}$ and 0.0438 $\mu\text{m}^2/\text{s}$ respectively. This aligns with the theory that the increase in temperature will result in faster diffusion rates. Observation of the SEM images show that the effect of diffusion coefficient is apparent in the thickness of the IMC layer and the area of Cu₃Sn formation but has minimal effect on the morphology of the IMCs as at both reflow temperatures, as both images depict the layers with similar morphologies of scallop-like structure.

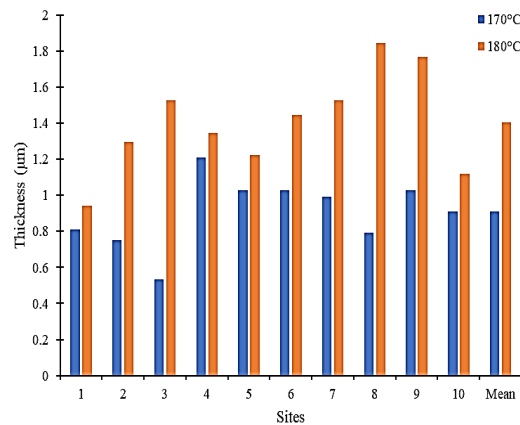


Fig. 9. Cu₆Sn₅ IMC layer thickness at 170 and 180 °C.

4. Conclusion

The TGA concluded that the melting temperature is between 130 and 140 °C and highlighted that impurities are present in the alloy which will appear during the SEM imaging and EDS analysis. It was discovered that at 160 °C, the temperature was insufficient to properly activate the flux and form IMCs, resulting in a layer of voids between the solder and the substrate instead of IMC layers. At 170 °C, Cu₆Sn₅ IMC were found, with an average thickness of 0.909 μm . The diffusion coefficient for 170 °C was found to be 0.0184 $\mu\text{m}^2/\text{s}$. At 180 °C, both Cu₆Sn₅ and Cu₃Sn were found between the Cu substrate and the SnBi solder. The average thickness of Cu₆Sn₅ was found to be 1.404 μm , which was 54% more than the thickness of IMC at 170 °C. The average diffusion coefficient found was 0.0438 $\mu\text{m}^2/\text{s}$. Cu₃Sn IMCs were not visible in the SEM indicating the less presence of these IMCs. From the in-situ results of this research, it suggests that there is a potential for the SnBi as a low temperature solder alternative.

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