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Interfacial Reaction and Intermetallic Compound Growth Behaviour of CNT-Filled Composite Solder on the Oxidizing Substrate

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ABSTRACT

The poor performance of traditional solder joints, which can be attributed to the formation of oxide layers on the substrate surface during the die attach process, has driven research into alternative materials like CNT-composite solder, which offers improved wetting and mechanical properties without compromising electrical performance. This study investigates the performance of CNT-composite solder (CCS) after the reflow process on the oxidizing substrate. The performance of the CCS was evaluated by comparing the morphology of the IMC layer of CCS with the IMC layer of SAC305. The morphology of the IMC layer was observed using Scanning Electron Microscopy (SEM) equipped with Energy Dispersive X-ray (EDX). The thickness and the roughness of the IMC layer for both the SAC305 solder and the CCS solder also were measured. The results reveal that the CCS solder has produced a thin IMC layer but rougher than the IMC layer from SAC305 solder. It concluded that the presence of CNT within the solder matrix plays a major role in the thin and rougher IMC layer. The thin layer of the IMC layer at an initial stage will benefit by increasing the solder joint's lifespan.

1. Introduction

Nowadays, the demands for smaller electronic packaging have escalated, pushing manufacturers to evolve the integrated circuit (IC) from the flip-chip package to the 3D-stacking and wafer-level [1]. The solder joint is important and plays a part in the interconnect in the IC [2]. It gives mechanical support and allows the movement of electrical from the chip to the copper lead frame.

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However, the chip was easily detached from the substrate due to the poor solder joint. The poor solder joint was related to the occurrence of an oxide layer on the substrate's surface [3]. This circumstance commonly happens during the die attach process. Lately, the attraction towards CNT-composite solder has increased due to its good wetting and mechanical properties [4,5]. The CNT nanoparticles were studied capable of improving the properties of the solder without affecting the electrical properties [6].

Despite the potential advantages, the research findings regarding the effectiveness of CNT-composite solder in mitigating the oxide layer problem remain inconsistent. Some studies report significant improvements, while others show minimal or no improvement in joint reliability [7,8]. This inconsistency indicates a gap in understanding the interfacial reactions and intermetallic compound (IMC) growth behaviour when using CNT-filled composite solder on oxidizing substrates.

Understanding this interaction is crucial as it directly impacts the reliability and lifespan of solder joints in electronic packaging. The significance of this study lies in addressing these inconsistencies by providing a comprehensive analysis of the CNT-composite solder's performance on the oxidized copper lead frame. This will be achieved by observing the morphology of the IMC layer formation and comparing it with the formation of the IMC layer from SAC305 solder.

For this purpose, the performance of the CNT-composite solder to encounter the oxide layer on the substrate's surface was studied. The CNT-composite solder's performance will be evaluated by observing the morphology of the IMC layer formation and compared with the formation of the IMC layer from the SAC305. Additionally, a solid understanding of the formation of the oxide layer and implementation of the current solder alloy is essential to be comprehended as it is the key to solve the lifespan of the solder joint.

2. Methodology

2.1 Preparation of the Sample

Composite solder was prepared by homogeneously mixing 0.04 wt.% of CNT nano-powder into commercializing 96.5 (wt%) of Sn, 3.0 (wt%) of Ag and 0.5 (wt%) of Cu (SAC305) using a paste mixing machine (Thinky mixer, ARE-310). The mixture was agitated for 400s at 1000 rpm to produce the CNT-composite solder (CCS). Simultaneously, the heat-treatment process has carried towards the copper lead-frame to promote the oxide layer on the copper lead frame's surface. The copper lead frame used in this study consisted of 97.05% Cu, 2.6% Fe, 0.15% P and 0.2% Zn, also known as C194. The C194 was exposed to three different temperatures, which are 120 ℃, 180 ℃ and 240 ℃ for 3 hours. Table 1 tabulated the detail of the temperature used to promote the oxide layer. The exposed copper lead frame was then characterized using Atomic Force Microscopy (AFM) to ensure the oxide layer's presence.

Table 1
The detail on exposure temperature and duration of the treatment

Condition	Temperature (℃)	Duration of the treatment
1	120	3 h
2	180	3 h
3	240	3 h

The CCS was manually soldered onto the oxidizing substrate using the dispensing approach. The deposited CCS paste will then undergo the reflow process to solidify the CCS solder. Figure 1 depicts the process preparation of the sample.

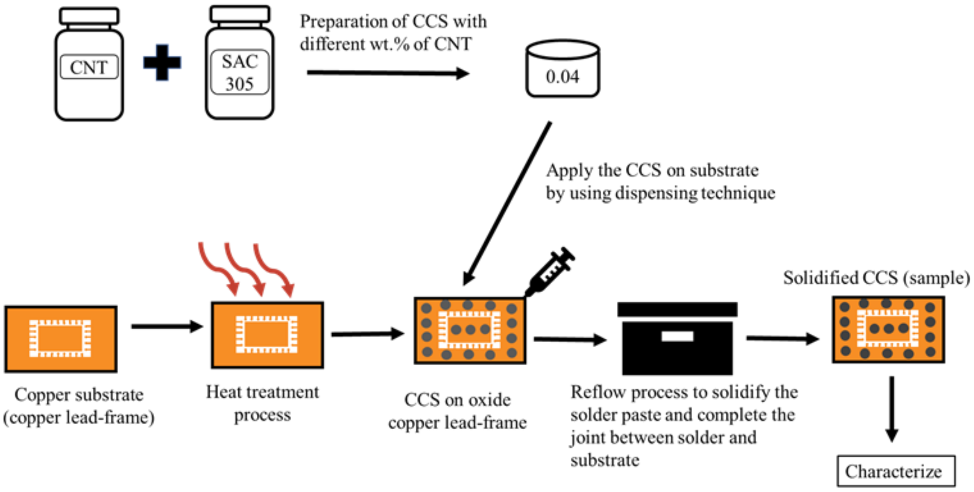


Fig. 1. Process of sample preparation

2.2 Characterization of the Composite Solder

All of the samples will be cross-section using a cross-section polisher (JEOL IB-19510CP). The accelerating voltage has been set to 5kv, with the ion beam set to 4.0. The cross-section polisher was performed for 6 hours per sample. Afterwards, the microstructure of all samples was observed via a scanning electron microscope (SEM, JEOL) with a back-scattered electron (BSE) imaging mode. Additionally, the energy-dispersive X-ray spectrometer (EDX) was used to detect the presence of the oxide layer on the substrate surface.

3. Results and Discussions

3.1 Formation of Oxide Layer on the Substrate Surface

The substrate that underwent the heat treatment under the temperatures of 120 °C, 180 °C and 240 °C were characterised with EDX analysis. The objective of the analysis was to ensure the presence of the oxide layer on the substrate surface. The substrate that is exposed under 120 °C will be referred to as Substrate 1. Meanwhile, the substrate exposed under 180 °C and 240 °C will be referred to as Substrate 2 and Substrate 3, respectively. Table 2 shows the details of weight % and atomic % for Substrate A, Substrate B and Substrate C. It was found that the element O was detected on all sample surfaces.

Interestingly, the atomic percentage of the O element increases with the increase of the temperature. The atomic percentage on Substrate 1 was 8.01 % and the value was increased to 37.71 % and 42.66 % for Substrate 2 and Substrate 3. The presence of the O element resulted from the inward diffusion of oxygen gaseous (O_2) into the substrate surface to form Cu_2O [9]. The fact of the O element on all substrate surfaces proves the oxide layer has formed on the substrate surface. A previous study used the heat-treatment process to promote the oxide layer and found a similar finding [10].

Table 2
Details of weight % and atomic % for substrate 1, substrate 2 and substrate 3

Element	Substrate 1		Substrate 2		Substrate 3	
	Wt.%	At.%	Wt.%	At.%	Wt.%	At.%
Cu	97.57	89.18	83.53	55.08	82.84	54.47
O	1.66	8.01	14.4	37.71	16.33	42.66
C	0.77	2.81	2.07	7.21	0.83	2.87

3.2 Surface Roughness and Thickness of the Oxide Layer

For a better insight into the matter, the surface roughness and thickness of the oxide layer for all three substrates were analysed through AFM and AES analysis. The graph for both surface roughness and thickness of the oxide layer is shown in Figure 2. Interestingly, the thickness of the copper oxide for substrate 2 was thicker than substrate 1 with 110 nm and the thickness gap between substrate 1 and substrate 2 was massive. Substrate 3 also has a thick copper oxide layer with 350 nm depth, as illustrated in Figure 2. During the heat-treatment for substrate 1, the oxide layer was slowly grown but stopped in an additional few nanometres. The temperature only provided little energy for oxygen atoms to diffuse through the native oxide layer [10]. However, the formation of the copper oxide was quick on substrate 2's surface and substrate 3's surface. Zheng and Wang [11] has stated that the oxygen atoms were active at high temperatures and tend to diffuse with a low energy barrier. This explained why the formation of the copper oxide layer on substrate 2 and substrate 3 was thicker compared to substrate 1.

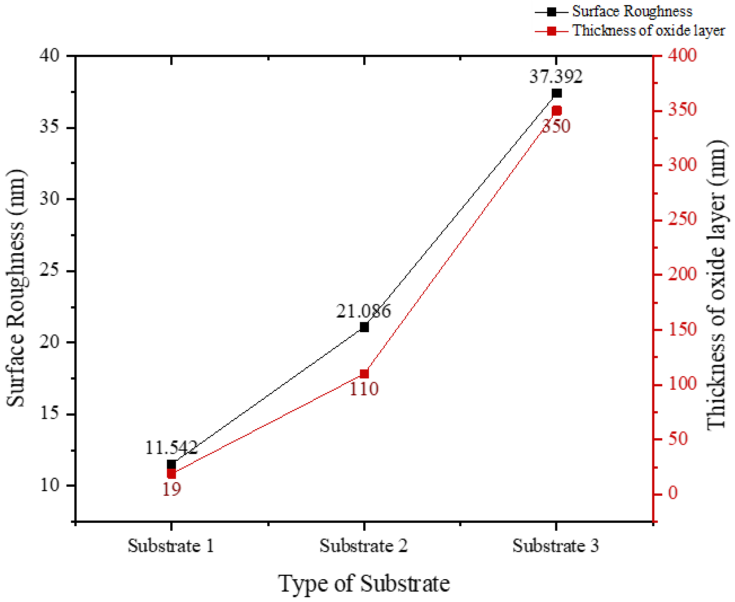
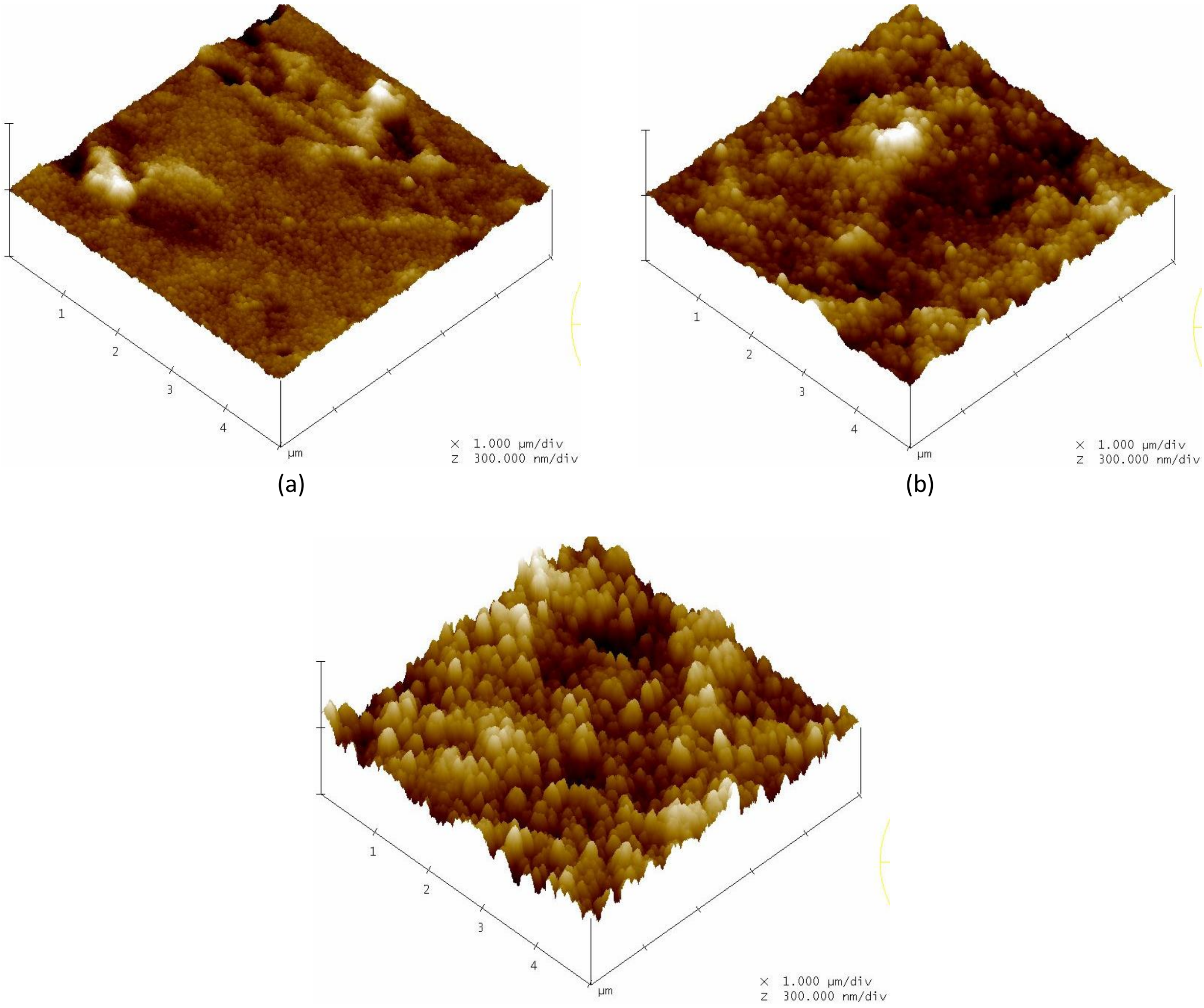


Fig. 2. The graph of surface roughness and thickness of oxide layer on all substrate's surface

The surface roughness of all samples was measured using Atomic Force Microscopy (AFM) for further details. Figure 2 shows a plotted graph of surface roughness for all samples subjected to the type of substrates. The surface roughness of substrate 1 was the smoothest compared to all samples with 11.542 nm. However, substrate 2 and substrate 3 depicted a rougher surface with the value of surface roughness was 21.086 nm and 37.392 nm. Figure 3 represents the 3-D AFM image for all samples with a scanning area of 5 µm x 5 µm.

Figure 3 shows the surface topology image of Substrate 1, Substrate 2 and Substrate 3. Figure 3(a) depicts a topology image for Substrate 1, which has a flat surface but is filled with dense particles on top of its surface. Different from Substrate 1, the surface of Substrate 2 is seen to be filled with bigger particles and a hilly surface. The surface on Substrate 3 was found to have a high hill-valley structure compared to other samples. Zheng and Wang [11] have found a similar finding where they found a hill-valley structure on the surface of their sample during the oxidation process. They believed the formation of the hill-valley structure resulted from the Cu-Cu_2O interfacial strain energy [12]. This statement was agreed upon by Daria *et al.,* [13].



(a) (b)

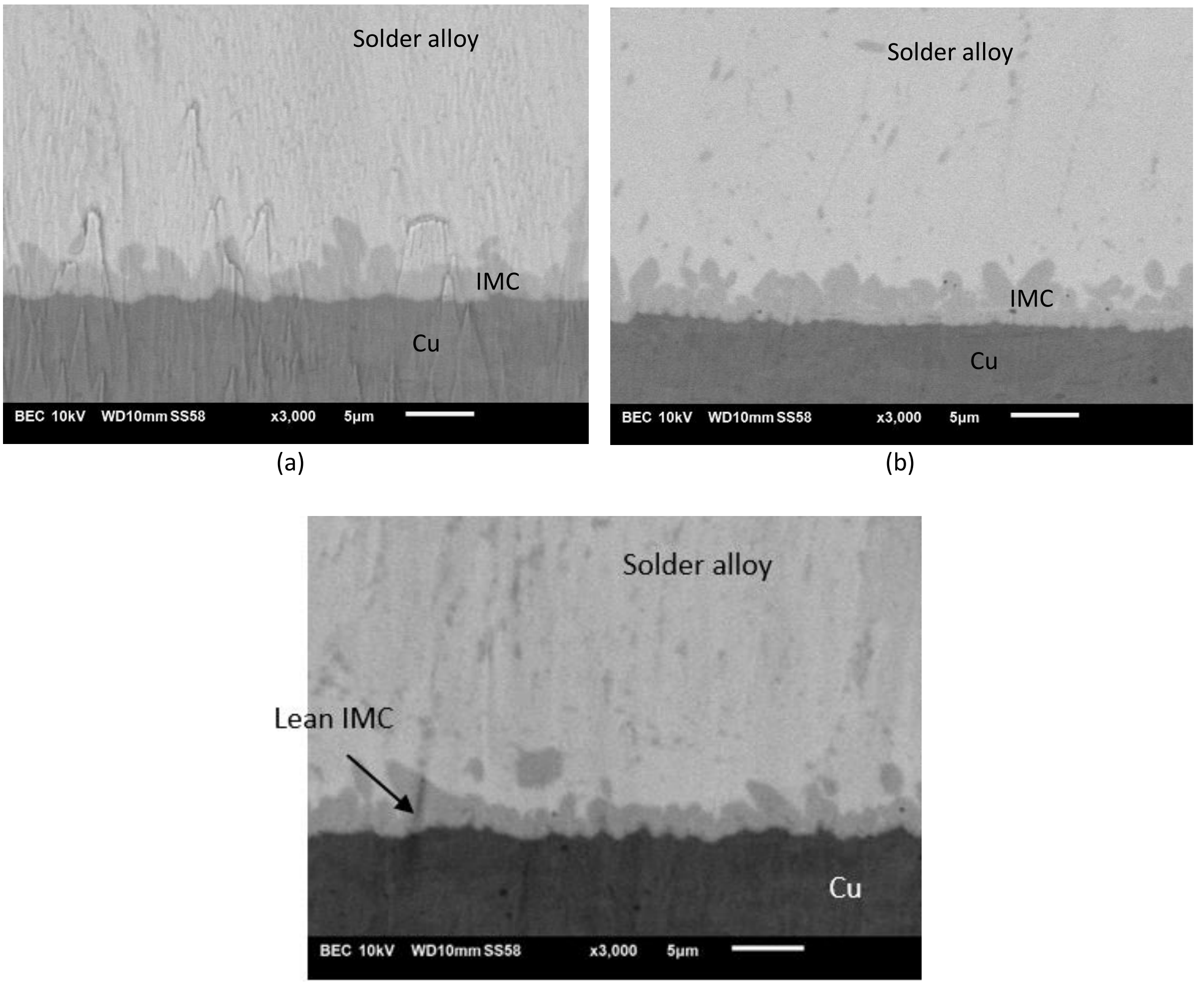
(c)

Fig. 3. Micrograph of AFM for substrate 1, substrate 2 and substrate 3

3.3 Microstructure Evolution of IMC Layers

There are three colonies within the reflowed SAC305 and CCS solder: the solder matrix, IMC layer and the Cu substrate. The IMC layer usually forms at the interface of the solder matrix and substrate [14]. This layer will represent whether the joint has been successfully made or not [15]. It was observed that the IMC layer formed for both SAC305 solder alloy and CCS solder regardless of the initial condition of Cu substrates, as depicted in Figure 4 and Figure 5.

Figure 4 shows an SEM image of SAC305 solder after reflowed on Substrate 1, Substrate 2 and Substrate 3. It was interesting to observe that the IMC layer's formation shape for reflowed SAC305 solder alloy was different for each substrate. The IMC layer of the SAC305 solder alloy comprises the Cu_6Sn_5 compounds [16]. Figure 4(a) is a figure of SAC305 solder alloy reflowed on Substrate 1 and the formation shape of the Cu_6Sn_5 was highly irregular with an average thickness of 3.9 µm. The irregular shape of Cu_6Sn_5 becomes even with an average thickness reduced to 3.6 µm after SAC305 is reflowed on Substrate 2. The thickness of the Cu_6Sn_5 decreased again to 2.73 µm when the SAC305 solder alloy reflowed on Substrate 3, containing a thicker oxide layer. At this time, the Cu_6Sn_5 was in a uniform shape and continuous.



(a) (b)

(c)

Fig. 4. SEM image of IMC for SAC305 solder alloy reflowed on (a) substrate 1 (b) substrate 2 (c) substrate 3

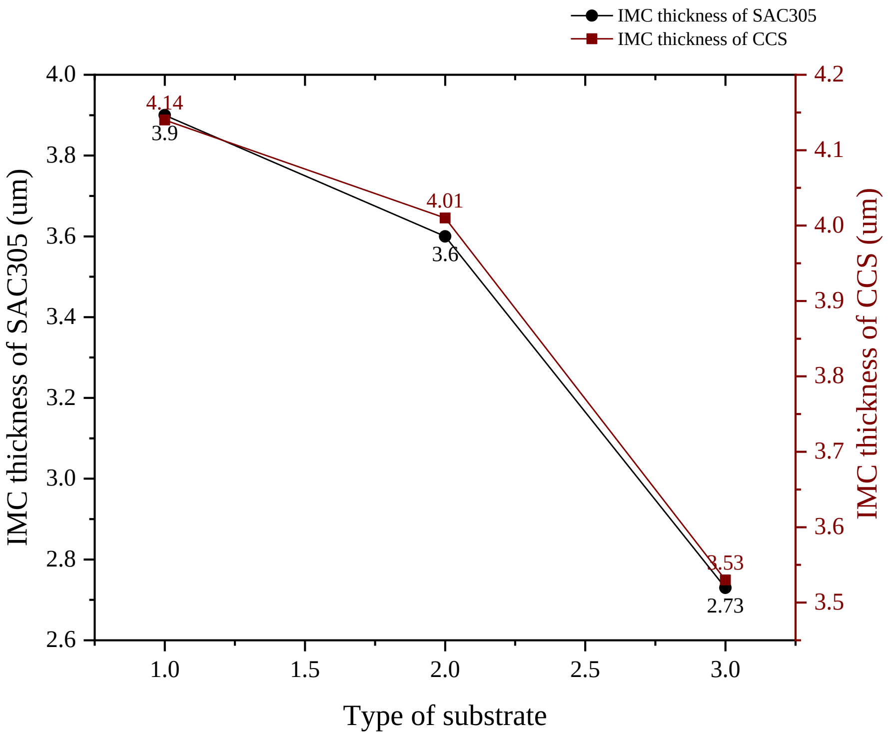


Fig. 5. The graph of the type of substrate against the IMC thickness of SAC305 and IMC thickness of CCS

The decreasing thickness of the Cu_6Sn_5 with increasing the oxide layer on the Cu substrate might depend on the growth rate of Cu_6Sn_5. Tu and Zeng [17] proposed the microstructure and morphology of Cu_6Sn_5 depending on the energy change rate (ΔG). They also suggested the Cu_6Sn_5 with scallop shape was formed due to the high rate of growth at the interfacial of Cu substrate. The ΔG is defined as

$$\Delta G = \int_0^{\tau} \frac{dG}{dt} dt \tag{1}$$

Where dG/dt was the rate of free energy reaction and τ was the reaction period. The mechanism and factors affecting growth rate were hard to explain and were still debated among the researchers [18,19]. A few researchers believe that IMC formation happens through the grain boundary diffusion process and volume diffusion process [20]. The grain boundary diffusion occurs during the melting temperature of the solid-liquid interface separated by a planar boundary. In this study, the solid was referred to as a Cu substrate and the liquid was the solder matrix. During T_m, the Cu atom from the Cu substrate will move forward to the solder matrix and the Sn atom from the solder matrix will move towards the Cu substrate. At the beginning of the reaction, the IMC η-phase will be forming near the Cu substrate's interface. The η-phase of IMC consisted of the Cu_6Sn_5 compound and was the first stage of IMC's growth [21]. Most of the Cu atoms were likely to react with Sn atoms because the Sn atom was the most rapidly diffusing species in the soldering joint [22]. Then, the reaction will continue and the ε-phase of the IMC will start to grow. The ε-phase of the IMC consisted of Cu_3Sn proliferates [23]. Due to the Gibbs-Thomson effect, the IMC grains will continue to grow and dissolve into the molten solder to form IMC's scallop shape [24]. This effect explained the scallop shape formation of IMC for both SAC305 solder alloy and CCS solder.

Both the IMC thickness for SAC305 solder alloy and CCS solder decreased when reflowed on Substrate 1, Substrate 2 and Substrate 3. This case could be related to the substrate's surface roughness before both of the solders were reflowed on it. The surface roughness of Substrate 1, Substrate 2 and Substrate 3 was 11.542 nm, 21.086 nm and 37.392 nm. The measurement of the substrate's surface roughness can be referred to in sub-topic 3.2. It is shown that the surface roughness of Substrate 3 was rougher compared to Substrate 2 and Substrate 1. The coarser substrate's surface tends to allow the molten solder (either SAC305 or CCS) to penetrate with a higher degree than the smoother surface [25]. The reason was coarser surface has a high asperity level which increases the capillary action during solder solidification. This mechanism concluded that a rougher substrate surface would produce uniform and thin IMC than the smoother substrate surface. Bhat and Satyanarayan [26] agree that surface roughness plays an important factor in forming IMC's shape and IMC thickness. They state that the solder's IMC formation would change from a long needle to a uniform scallop when reflowed the solder from a smooth surface to a rougher surface.

It was noticed that the IMC thickness of the CCS solder was higher than the IMC thickness of the SAC305 solder alloy. To explain the finding, the rate of diffusion coefficient for both SAC305 solder alloy and CCS solder when reflowed on the three different substrates was measured through Eq. (2). Figure 6 shows the diffusion coefficient (D) graph for both SAC305 solder alloy and CCS solder when reflowed on Substrate 1, Substrate 2 and Substrate 3. It can be seen that the D value of the reflowed SAC305 on Substrate 1 was slightly higher than the reflowed CCS on Substrate 1. However, the D value of reflowed SAC305 on Substrate 2 and Substrate 3 was lower than the D value of reflowed CCS on Substrate 2 and Substrate 3. It states that the IMC layer's growth rate for CCS solder when reflowed on Substrate 1 was slower compared to the growth rate of the SAC305 solder when reflowed on Substrate 1. But, the difference in the growth rate between SAC305 and CCS when reflowed on Substrate 1 was extremely small, with differences was only 3.5%. Meanwhile, IMC's growth rate for CCS when reflowed on Substrate 2 and Substrate 3 was higher than the IMC growth rate for SAC305 when reflowed on Substrate 2 and Substrate 3. It explained the IMC thickness of the CCS was higher than the IMC thickness of the SAC 305.

$$h = \frac{A}{L} \tag{2}$$

It is well understood that the growth of copper oxide mainly caused the different roughness of the substrate's surface [27]. This copper oxide layer will act as the barrier that slows down the solder (both SAC305 and CCS) to interact with the substrate's Cu atom. During the reflow process, the Cu atom diffuses into the oxide layer and breaks the copper oxide layer to interact with the molten solder and form the IMC layer [28]. Therefore, the D value for substrate with a thicker copper oxide layer tends to be lower than the thin copper oxide layer. Figure 7 shows the mechanism of the process.

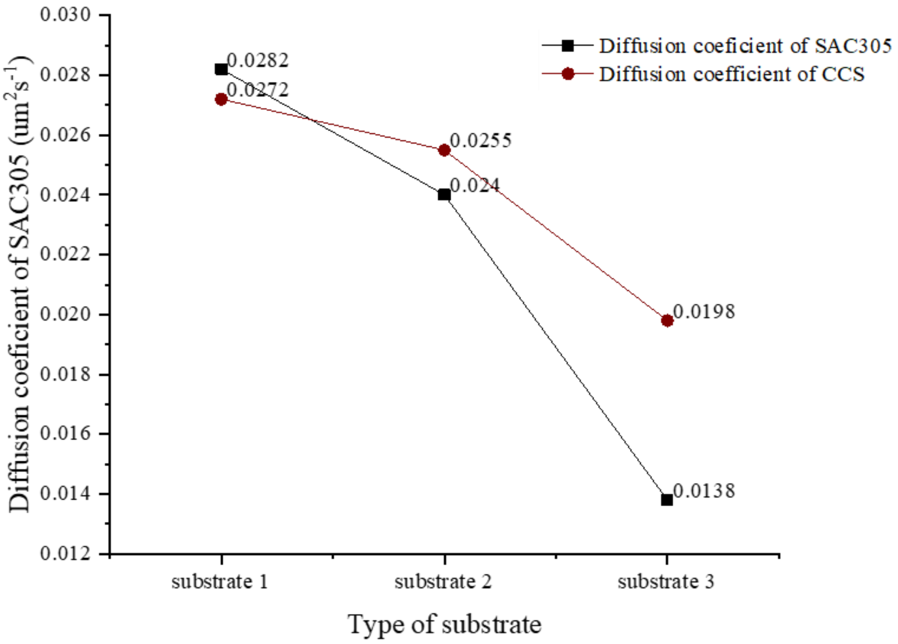


Fig. 6. The graph of diffusion coefficient for both SAC305 and CCS on three different substrates

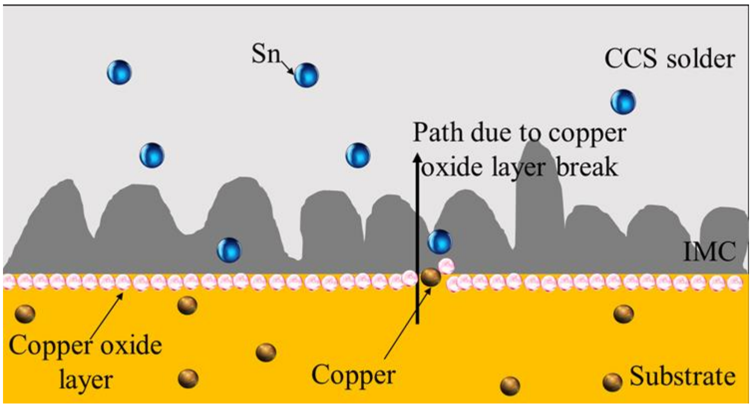


Fig. 7. Illustration of the copper atom breaking the oxide layer to interact with Sn atom

Figure 8 shows that the CCS solder has a higher growth rate than the SAC305 and the different growth rates are suspected related to the presence of the CNT within the solder matrix. When the CCS solder underwent the reflow process, the CNT will be pushed toward the grain boundary of the solder matrix [29]. The presence of the CNT within the grain boundary will weaken the interaction between the grains, allowing the Cu atom from the substrate to diffuse through the grain boundary towards CCS easily. This mechanism explained the high-rate diffusion growth of CCS compared to SAC305 solder alloy. However, at the same time, the presence of CNT at the CCS solder/substrate interface had given a random path for the diffusion, resulting in the rough surface of the IMC layer, as shown in Figure 8.

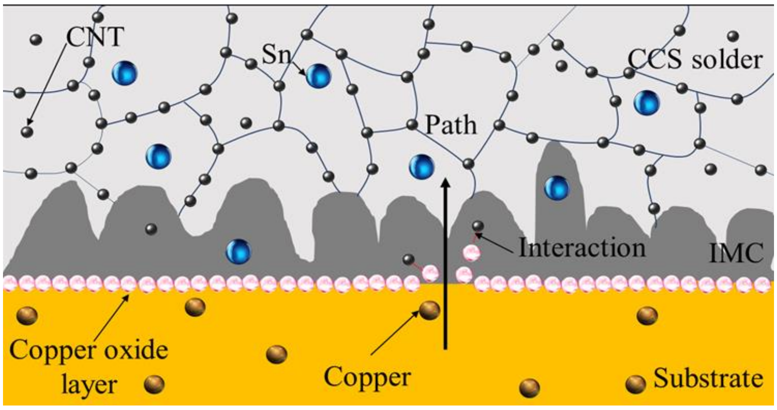


Fig. 8. Illustration of interaction between the CNT and CuO has opened the path of the copper and Sn

4. Conclusions

Two major findings can be concluded from the study:

i. In this study, the oxide layer was successfully deposited on the copper lead frame's surface by the heat-treatment method. Different temperature exposures will alter the substrate's surface. This study reveals that the surface roughness of the substrate was different with different temperatures during the heat-treatment process. High temperatures lead to a rougher surface because of the formation of Cu_2O. Other than that, the thickness of the Cu_2O also increased with the increase in temperature. It was believed that high temperatures would function as a catalyst to fasten the growth of Cu_2O.
ii. The formation of the IMC layer after CCS was reflowed on the oxidizing substrate was thinner compared to the IMC layer thickness of SAC 305. However, the IMC layer of the CCS was rougher compared to the IMC layer of SAC305. It was found that the CNT within the CCS solder matrix has lost the interaction within the solder matrix and gives many direct paths for the Cu substrate to diffuse into the solder through grain boundary diffusion. But the CNT also will be a barrier for Cu to diffuse with the Sn within the solder matrix, which causes the formation of the IMC for CCS to become rougher.

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