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Effect of Different Nickel Percentage in Solder Alloy towards **Intermetallic Compound Formation and Growth**

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Abstract: The growth of electronics product has increased rapidly and hence drive challenges among the designers as well as manufacturers to select the best electronic packaging materials for their products. Among those materials, lead free solders has become the best replacement for leadcontaining solders even though its performance still could not meet the original specification. Thus, this paper is aiming at studying the effect Nickel (Ni) additional with difference percentage into solder alloy toward intermetallic compound formation (IMC) and growth at the interface of the solder joint. The work was carried out by producing Sn-xNi (x = 0.0, 0.3, 0.5, 0.7, 1.0) through powder metallurgy method. The process started with powder milling at the speed of 1400 rpm for 3 hours. Then it was compacted into a thin pallet with size of 6mm diameter at the pressure of 5 ton. After that, the sample were subjected to reflow soldering for 250°C for 25min heating duration and 15min holding time. Solder joint strength was carried out using ASTM D1002. Finally, characterisation was made in terms of IMC morphology, thickness and solder joint strength using metallographic microscope as well as scanning electron microscopy-energy dispersive x-ray (SEM-EDX). Results showed that the addition of Ni with different percentage produced different morphology of IMC at the interface after reflow soldering which also affects its growth behaviour. The IMC's formed after reflow soldering produced closed and packed grain boundary when solder with higher Ni content was used. The type of IMC formed at the solder joint was Cu6Sn5 and its thickness increased with Ni addition. These results were the proof that Ni is a good candidate to improve solder joint performance by restricting the formation and growth of Cu3Sn IMC. In addition, the solder joint strength was found to be increasing upon Ni percentage increment. The highest strength value of 5.885MPa was produce when Ni was added up to 1.0%. This showed that Ni not only a good candidate to improve joint performance in IMC formation but also help in strengthening the solder joint.

Keywords

Solder Alloy, Soldering, and Intermetallic compound

1. Introduction

Leaded solder has been used may years ago in electronics manufacturing due to its performance in providing good joint reliability. However, there was an arise in environmental and human health concerns owing to



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the use of lead in solder allow since it known as heavy metal and its effect is very dangerous [2]. As a result, The European Union's Restriction of Hazardous Substances in Electrical and Electronic Equipment (RoHS) prohibited the further use of lead in electronics [3]. Since that, lead-free solder alloy has become the main stream of industries due to its environmentally friendly. Many changes in alloy composition are adopted in order to match the requirement of solder joint with Pb-free definition. Tin Copper (SnCu), Tin Silver (SnAg), Tin Nickel (SnNi) and Tin Silver Copper (SnAgCu or SAC) are the dominating Pb-free solder alloys adopted by the industries.

Among these solder alloy, SnAgCu solder alloy has become one of the best candidates since its composition close to the eutectic composition [4]. Nevertheless, this solder is quite expensive due to the high content of Cu and Ag. Therefore, this study chose solder with another element such as Ni. This is because, it is not only can reduce the cost of producing solder alloy but also eliminating the cost for substrate metallization. This solder poses a good mechanical properties and excellent strength as well as fatigue properties [5]. Other than that, the addition of Ni into the solder is known to be improved most of Sn-based solder alloy's wetting properties. It was mentioned by previous researchers that even a small amount of Ni in the solder managed to form fine Cu-Ni-Sn IMC which can retard aggressive growth of IMC during aging and at the same time improved its mechanical properties [6], [7]. In addition, according to research carried out by [8] mentioned that the small addition of Ni into SnCu solder alloy managed to prevent rapid diffusion between solder alloy and copper substrate and thus suppressed further growth of the IMC during aging. Besides, proper amount of Ni added into the solder may also reduce IMC thickness. These phenomena will eventually affect the solder joint strength since it is very much depending on IMC type and thickness.

Referring to the above information, the addition of Ni into the solder alloy has clearly affected the interfacial reaction which finally may affect the solder joint strength. However, the research on interfacial reaction between Sn-*x*Ni and copper substrate is still insufficient because Ni percentage need to as optimum as possible in order to control the IMC formation and growth without involving too many production costs. Hence, this study at focuses at determining the effect of different Ni addition into Sn-*x*Ni solder alloy towards IMC formation and growth. The IMC thickness, type and morphology were studied for different Ni percentage. Then, optimum Ni percentage will be recommended.

2. Methodology

Sample preparation methods used in this study were powder metallurgy and reflow soldering for solder joint formation. For the powder metallurgy process [9], mechanical mixing and compaction have been considered for this research. Second aim is to distinguish the effect of different solder alloy composition towards solder joint strength. Shear test was also conducted to determine the solder joint strength of each sample composition. The process starts with mechanical mixed the solder alloy until they become homogenous. The suitable blending duration for these studies was 6 hours, with the speed of 1400 RPM. The speed of rotational and mixing time for the jar is kept constant. The powder of Sn and Ni with difference percentage (Sn-xNi, with x = 0.0, 0.3, 0.5, 0.7, 1.0) weighted carefully and mixed and blended in a mechanical alloying machine. Upon completion of mixing process, the mixed powder was compacted 5 ton into a 6mm diameter thin pallet. Then, the green solder material was subjected to reflow soldering to perform solder joint. Then these specimens were mechanically tested using Instron Universal Tensile Machine in order to determine solder joint, following ASTM D1002 standard. After that, all specimens were characterized in terms of IMC thickness and IMC morphology using metallographic microscope, Scanning electron microscope-energy dispersive x-ray (SEM-EDX).

3. Results and Discussion

3.1 Effect of Ni Addition onto IMC Morphology

Tin-nickel (Sn-Ni) solders are tin concentrations between 0%, 0.3%, 0.5%, 0.7% and 1.0% by weight. The Sn-xNi (0%, 0.3%, 0.5%, 0.7% and 1.0%) solder alloy compositions will be annotated such as follows 100%Sn-0%Ni (SN00), 99.7%Sn-0.3%Ni (SN03), 99.5%Sn-0.5%Ni (SN05), 99.3%Sn-0.7%Ni (SN07), 99%Sn-1.0%Ni (SN10) for the purpose of discussion in this study.

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It is an established fact that the final structure of solder the joint depends on the material composition percentage [10]. In order to determine the influence of solder strength on the solder joint structure, five different material composition were used as shown in Figure 1. All samples undergone the same reflow process. After reflow soldering, the fastest IMC to form was Cu_6Sn_5 because it provides fast diffusion passageway for Cu atoms to diffuse towards solder alloy. This type of IMC was confirm using SEM-EDX as shown in Table 1. Meanwhile, according to figure 1, it was found that as the percentage of Ni increased, the morphology of IMC also changed. Furthermore, the existence of nickel did affect the IMC grain boundary as can be seen in Figure 1(a), whereby the grain size became finer as the percentage of Ni increasing.

However, the shape of IMC turnout to be needle-like shape instead of scallop shape. As the percentage of Ni increased, the numbers of needle-like shape IMC also increased. Nevertheless, the amount of Ni in Figure 1 was difficult to detect due to low Ni element content. At Ni percentage of 1.0%, Ni element started to react and thus producing $(Ni,Cu)_6Sn_5$ IMC. According to [8], the solubility of Ni in Cu₆Sn₅ is high and Cu can be easily replaced with Ni due to its atomic diameter of Ni is similar to Cu. That would the reason of $(Ni,Cu)_6Sn_5$ formation as well as needle-like shape IMC. The low Ni content in the solder might be the reason on difficulty of detecting Ni in Cu₆Sn₅ IMC.



Figure 1. Morhplogy of IMC Top View using SEM with Magnification of 4000X

Table 1. Atomic Percentage o	of EDX Top View
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 Atomic %		
Sn	Ni	Cu

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0% Ni - 100% Sn	38.256	0	53.774
0.3% Ni - 99.7% Sn	36.796	1.259	61.944
0.5% Ni - 99.5% Sn	36.055	6.134	57.809
0.7% Ni - 99.3% Sn	41.503	8.072	50.424
1.0% Ni - 99% Sn	47.087	11.271	41.64

According to Figure 2, it can be seen that the atomic percentage of copper at all composition was high, which means this element dominate the solder alloy during grain boundary formation. In fact, after reflow soldering, Sn element also become dominant after Cu element.

This explained how Cu element from board/substrate and Sn diffused and bonded together at the solder joint. Besides, Ni reading in solder alloy from the graph increased with the same amount specimen's Ni percentage. This is because the amount of nickel percentage increased proportionally to the atomic percentage during the solid-state diffusion.



Figure 2. Comparison of Average Atomic Percentage of EDX Top View

Furthermore, the size and shape of the IMC varies depending on material's composition [11]. Figure 3 shows the cross-sectional views of all specimens. It can be seen that, there was a difference in shape and size of the grain formed at the solder joint's interface. This is probably due to the effect of different Ni concentration in the solder alloy [5]. The differences were as expected in the terms of types of IMC formed, morphology, and their growth rate after reflow soldering. All of these were correlated to their mechanical and electrical integrity [12].

The results of the present work indicated that the cross section interfacial characteristics of composite solder joints are affected significantly by the difference composition of Nickel. This can be seen through Figure 3 that solder alloy with 0%, 0.3%, 0.5%, 0.7% and 1.0% Ni produced finer and denser grain size as Ni percentage increased. These fine grains structure may influence the diffusion of Ni by providing fast Ni diffusion channels in some particular locations.

The scallop-shape and flat-shape IMC's layers were generated on the all-sample's interfaces during initial reflow. The scallop-shape IMCs layer was transformed into the flat-shape one when Ni percentage increased on the solder alloy interface as shown in Figure 1 (d & e). The Ni in solder played a role of purification, and it could decrease the concentration gradient of Sn atom at the solder interface to inhibit the Sn-containing compounds growth.

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Figure 3. IMC Thickness Magnification of 500X

It was also found that the IMC thickness increased upon percentage increment in the solder alloy. As a result, IMC thickness grew thickener as Ni percentage increase after reflow soldering (Figure 4).



Figure 4. Comparison of IMC Thickness Cross Section View

3.2 Effect of Ni Additional into Solder Alloy towards Solder Joint Strength

In this study, lap shear test was carried out to determine the shear strength of adhesives for bonded materials when single-lap-joint specimen was used. It was also used to evaluate the material's strengths following ASTM D1002.

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From the results. It was found that the hhighest shear strength was dominated by 1.0% Ni sample followed by 0.3% Ni and 0.7% Ni as illustrated in Figure 5. Whereas, the lowest strength was produced by 0% Ni followed by 0.5% Ni. Knowing that shear strength from 1.0% Ni had the highest value of IMC thickness, this is somehow a fascinating situation to explore the reason behind it. The 1.0% Ni produced the highest value of shear strength which was 5.885MPa. It is understandable that large percentage Ni in the solder alloy could contributed to the higher shear strength due to its behaviour such as hard and ductile [13]. This could be the reason of higher shear strength value for 1.0% Ni sample.

Table 2: Average Result of Shear Stress		
Composition	Average Compressive stress at maximum force [MPa]	
0% Ni – 100% Sn	1.86	
0.3% Ni-99.7% Sn	5.21	
0.5% Ni-99.5% Sn	2.145	
0.7% Ni-99.3% Sn	3.155	
1.0% Ni – 99% Sn	5.885	

With 0% Ni, the shear strength recorded a value of 1.86MPa whereas 0% Ni depicted the lowest value of shear strength in this study. As shown in Figure 5, the overall solder strength was ascending curve as Ni percentage increase in solder alloy. Since more Ni percentage in solder alloy clearly appeared in the specimen SN10 were substantially improved, resulting in the joints with the greatest shear strength.



Figure 5. Strength Composition Solder Curve

4. Discussions

Result showed that different Ni composition gave different results in term of IMC thickness, morphology and type. This proved a proper mixing of solder alloy element is crucial in order to have a specific yet high reliable of solder alloy with good performance. All data obtained were compared and analyzed to obtain accurate and relevant findings. The three tests that will be conducted to strengthen the results of this study are shear test, IMC thickness and IMC morphology.

The mechanical properties of solder alloy produced through powder metallurgy is a successful method and proven from the results of this study. The SEM/EDX result depicted a crystalline structure of milled solder alloy before continue with pressing method. This has verified that the PM method successfully assemble each element to form homogeneous mixture of solder alloy.

The scallop-type of Sn-Ni still showed up at the solder joint's interface as the Ni increased, it changed into planar-type IMC due to Ni behavior where it is very strong, ductile, and malleable and it excellent for

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strengthening metal alloys. Furthermore, in the previous study, by employment of Ni-doped solder, the shear strength of SAC305-0.1Ni/Cu and SAC305-0.1Ni/Ni joints were future enhanced [14].

It is understandable that large percentage of Ni in solder alloy could contributed to a higher shear strength due to Ni behavior is hard and ductile. As the Ni increase, solder joint strength seemed to be higher [15]. This was due to the Ni manage to bond strongly with other element during reflow process.

5. Conclusion

A few conclusions can be produced from the study:

- i. Different Ni percentage produce different morphologies of IMC during reflow soldering which allowed influence the growth behavior of the IMC. The EDX result illustrate an interfacial characteristics of composite solder joints are affected significantly by the difference composition of Ni [16]. This has verified that the IMC type charged for each percentage on microstructure analysis. The scallop-type of Cu₆Sn₅ showed up at the interfacial solder joint for sample SN00 and SN03 composition but as the Ni percentage increased, it changed into needle-type (Ni,Cu)₆Sn₅.
- ii. The compressive stress reading trend showed that as the Ni percentage increased, the stress increased proportionally. This explained that large percentage of Ni in solder alloy produced higher shear strength. As the Ni percentage increased, solder strength seemed to be higher. In summary, the specimen SN07 with Ni percentage of 0.7% solder type Cu₆Sn₅ was recommended. This due to sufficient IMC thickness of 9.92µm and 3.155 MPa shear strength was good enough. This 0.7% Ni percentage demonstrated superior mechanical reliability, and good IMC needed which could benefit in electronic industry.

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