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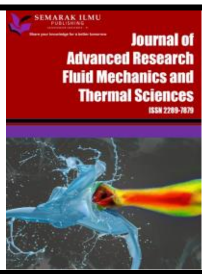

Influence of TiO_2 and Al_2O_3 Nanoparticles Addition on Thermal, Wettability and IMC Growth of Sn-3.0Ag-0.5Cu Lead-Free Solder at Different Reflow Temperature

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ABSTRACT

This study investigated the influence of adding 0.50 wt% and 1.0 wt% titanium dioxide (TiO_2) and aluminium oxide (Al_2O_3) nanoparticles on the properties of Sn3.0Ag0.5Cu (SAC305) lead-free solder alloy. In terms of thermal properties, SAC305 lead-free solder with or without addition of TiO_2 and Al_2O_3 nanoparticles, the melting temperature is very similar and comparable. The wetting performance of the SAC305 nanocomposite solder was determined by the contact angle. The best contact angle recorded is 31.65° at reflow temperature of 250°C for 0.50 wt% addition of TiO_2 and Al_2O_3 nanoparticles. The thickness of the IMC layer was observed using an Optical Microscope and measured. Results show that the thickness of the IMC layer decreased when TiO_2 and Al_2O_3 nanoparticles were reinforced into the SAC305 solder system, which shows significant decrease when 0.50wt% of TiO_2 and Al_2O_3 nanoparticles were added. However, beyond 0.50 wt% the thickness of IMC layer increases along with increasing reflow temperature. The results reveal that the adsorption effect of TiO_2 and Al_2O_3 nanoparticles will restrict the growth of the IMC layer under controlled concentration of the nanoparticles and reflow temperature. The optimal parameters to enhance the performance of SAC305 solder by reinforcing TiO_2 and Al_2O_3 nanoparticles are reflowed at 250°C at 0.50 wt% concentration of TiO_2 and Al_2O_3 nanoparticles.

1. Introduction

Tin-lead (Sn-Pb) solders are being widely used due to they are cost-effective, possess low melting point, good wettability and have distinctive mechanical properties in most of all in the electronic packaging industries [1-6]. Owing to environmental consequences (toxicity of Pb), thus, it contributed to a ban on electronics products in majority of countries. The use of harmful substances in electronic products have been prohibited by the European Union's Restriction of Hazardous Substances (RoHS) in 2006 [1,7]. As a result of the lead restriction in the electronic industry, there has been an increasing number of lead-free solders been investigated and developed to replace them [8,9]. Sn-Ag-Cu lead-

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free solder is one of the most promising lead-free solders due to its superior reliability, creep resistance and thermal fatigue properties [10].

Lead-free candidate solders based on Sn-Ag-Cu alloys are promising for variety of applications [11,12]. The properties of interfacial IMC layers are constantly being explored and improved in order to improve the reliability of Sn-Ag-Cu solder joints. Various IMCs are formed from all common substrate materials in electronic products during soldering [11]. Furthermore, understanding on the solder/substrate interface evolution process and mechanism in the solder joints is also critical for understanding reliability issues from the metallurgical standpoint and optimizing the soldering process. Besides, an appropriate thickness of IMC layer is needed for a good solder joint bonding between the solder and substrate. Reliability of a solder could significantly be degraded attributed to the excessive or thicker IMC layer. Thus, it is important to control the growth of the excessive IMC layer at the solder joints. Solders with minor alloying is one other possible method for improving the performance of the interfacial IMC layers in solder system.

Multiple research efforts have revealed that one of the potential solutions is producing nanocomposite solders by incorporating a relatively small number of nanoparticles into SAC solder [12-14]. The use of ceramics as reinforcement in the solder joints have been a novel trend in the development of solder alloys. The use of ceramic reinforcement in nanoparticle form usually below 100nm can promote heterogeneous nucleation. A wide selection of ceramics nanoparticles such as TiO_2, ZnO, ZrO_2, Al_2O_3, La_2O_3, SiC and Fe_2O_3 can be used as reinforcement to create lead-free nanocomposite solder alloy [13,14]. As a result, there has been an increased demand for the development of high-performance lead-free solder that can strengthen the characteristics they possess.

Al_2O_3 nanoparticles was found can restrict the growth of IMC layer, reduce the grain size and improve the reliability of solder joints [5,14,15]. Besides, TiO_2 nanoparticles could also inhibit the growth of IMC layer, have better wettability, strengthening the solder joints and increase the hardness of the solder [16-18]. Chuang *et al.,* [19] found that additions of Al_2O_3 nanoparticles decreases the surface energy of Ag_3Sn IMC grains and restricts the growth of IMCs. Chang *et al.,* [16] discover that growth of IMC layer was suppressed effectively by altering the morphology of Cu_6Sn_5 IMC layer from continuous scallop-shaped to a thinner discontinuous scallop-shape with additions of Al_2O_3 nanoparticles. Chang *et al.,* [16] and Tsao [20] studied the effects of TiO_2 nanoparticles on the IMC growth, thermal analysis, microstructure and tensile properties, and reported a reduction in grain size of β-Sn and Ag_3Sn, increase in liquidus temperature, and significant improvement in microhardness. Tang *et al.,* [18] investigate the influence of TiO_2 nanoparticles on IMC growth of Sn-3.0Ag-0.5Cu-xTiO_2 and observe significant decrease in both the thickness and grain size of IMC by means of adsorption, heterogeneous nucleation, and Ostwald ripening due to additions of TiO_2 nanoparticles. These ceramic nanoparticle materials that act as reinforcing agents were found to have good characteristics and enhanced the performance of solder alloy.

This work aims to investigate the influence of both TiO_2 and Al_2O_3 nanoparticles addition into Sn-3.0Ag-0.5 Cu solder on the thermal, wettability, and IMC growth of SAC305 lead-free nanocomposite solder.

2. Methodology

SAC305 solder was produced with 96.5% Tin (Sn), 3.0% Argentum (Ag), and 0.5% Copper (Cu) in weight percentages (wt%). The SAC305 lead-free nanocomposite solder was prepared by adding 0.50 wt% and 1.0 wt% of TiO_2 and Al_2O_3 nanoparticles respectively into the lead-free SAC305 nanocomposite solder at 250°C in a graphite crucible for 1 hour, and these nanocomposite solders were designated as Sn-3.0Ag-0.5Cu-0.50TiO_2-0.50Al_2O_3 and Sn-3.0Ag-0.5Cu-1.0TiO_2-1.0Al_2O_3. Then, the prepared solder alloy was cut into 5mm diameter billet with 1mm thickness. The wettability of the nanocomposite solders was evaluated by measuring their contact angles. The nanocomposite solders were melted on copper (Cu) substrate at size of 10x10mm at different reflow temperatures of 230, 250, 270 and 290°C on a laboratory hotplate for 60s. The contact angle was examined by using optical microscope and measured using VIS Pro software. The thickness of the intermetallic compound layer was obtained using an optical microscope. The melting details of the lead-free SAC305 nanocomposite solders were investigated using Differential Scanning Calorimetry (DSC, Perkin Elmer). In an Argon atmosphere, each solder alloy was heated at a rate of 10°C/min to temperatures ranging from 30-300 °C.

3. Results

3.1 Thermal Properties

Figure 1 shows the differential scanning calorimetry (DSC) curves for specimens reinforced with 0.50 and 1.0 wt% composition of TiO_2 and Al_2O_3 nanoparticles are shown in Figure 1. Figure 1(a) shows the DSC curve of SAC305 lead-free solder without any addition of TiO_2 and Al_2O_3 nanoparticles. Figure 1(b) and (c) shows the DSC curves for SAC305 lead-free solder reinforced with 0.5 and 1.0 wt% of TiO_2 and Al_2O_3 nanoparticles respectively. The melting temperature of SAC305 lead-free solder was measured to be 222.5 °C during the heating process of DSC analysis. The melting temperature was slightly changed 222.6 to 231.2 °C for SAC305 nanocomposite solder as shown in Figure 1(b) and (c). The slight increase in melting temperature was due to high melting point of TiO_2 (TM=1843°C) and Al_2O_3 (TM=2072°C) nanoparticles. Since both TiO_2 and Al_2O_3 nanoparticles has high melting point, it suggests that this is due to the local dissolution of TiO_2 and Al_2O_3 nanoparticles [16]. SAC305 solder and SAC305 nanocomposite solders begin to melt at 217°C, reaching its fully liquid state in range between 228.5 to 233.5°C. It can be observed that the solidus (TS) for all SAC nanocomposite solders, the solidus temperature was similar to the lead-free SAC305 solder at 217 °C. Meanwhile, the liquidus (TL) temperature of SAC305 nanocomposite solder are increasing slightly from 231 to 233.5 °C as shown in Figure 1(b) and (c). Similar findings were also found that the melting point of SAC lead-free solder increase slightly when reinforced with TiO_2 and Al_2O_3 nanoparticles [15,17]. Yet, the slight increment in the melting temperature is not a major concern as it still falls under the typical reflow temperature range for Pb-free solder which is 240-250°C.

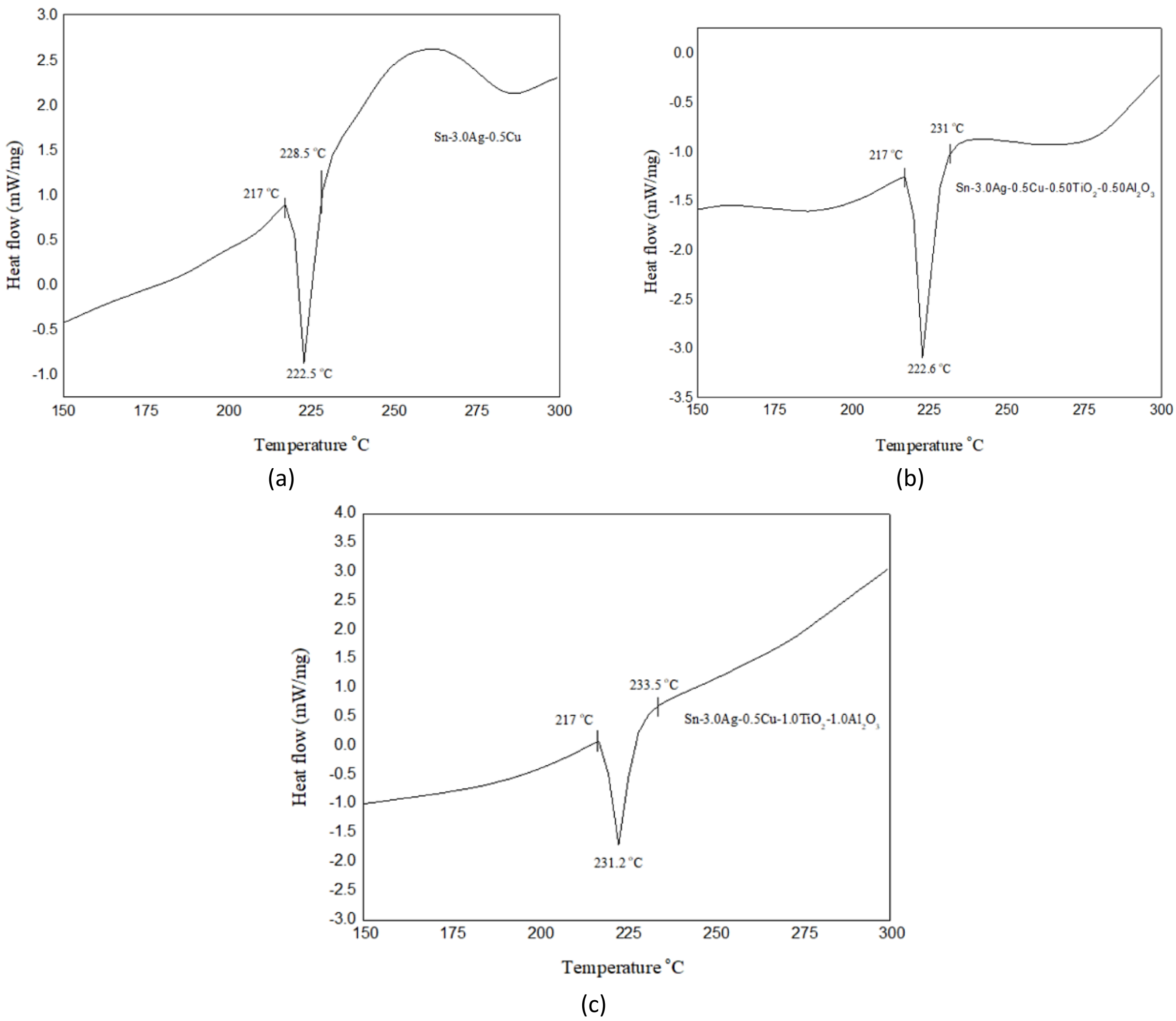

Fig. 1. DSC Curves of SAC305 solder with different wt% of TiO_2 and Al_2O_3 nanoparticles concentrations, (A) SAC305, (B) Sn-3.0Ag-0.5Cu-0.5TiO_2-0.5Al_2O_3 and (C) Sn-3.0Ag-0.5Cu-1.0TiO_2-1.0Al_2O_3

3.2 Wettability

The wettability of the solder, or its capacity to spread molten solder onto a substrate and maintain contact with it, can have an impact on the solder joint's reliability. The contact angle between the molten solder and the Cu substrate can be used to determine the wettability of the solder. Solder has very good wettability when the contact angle is between 0° and 20°. When the value of the wetting angle is between 20° and 40°, it is deemed acceptable and good [10]. Temperature plays a crucial role in determining wettability, spreading characteristics, and interfacial morphology. A good wetting behaviour of the solder by means the contact angle measurement can be achieved by controlling the temperature. Table 1 and Figure 2 present the SAC305 nanocomposite solder's wettability behaviour under a variety of contact angles (ϴ) and reflow temperatures, respectively. As shown in Figure 2, the best contact angle recorded is 31.65° at reflow temperature of 250°C for 0.50 wt% addition of TiO_2 and Al_2O_3 nanoparticles. The largest contact angle 46.09° was observed at 290°C for SAC305 solder. Decrease in contact angle from 230°C to 250°C was initially observed, yet an increasing trend was noticed afterward when the reflow temperature increase from 250°C to 290°C. According to these findings, the nanocomposite solder's wetting performance was enhanced by adding nanoparticles of TiO_2 and Al_2O_3 at concentrations of up to 0.50 weight percent at 250 °C.

In other words, the wetting performance can be increased by a small amount at a limited temperature by adding more nanoparticles. In the presence of the TiO_2 and Al_2O_3 nanoparticles, the decreased surface tension between the molten solder and the Cu substrate may be the cause of this occurrence [21,22]. However, upsurge in the contact angle happen as a result due to higher concentrations of TiO_2 and Al_2O_3 nanoparticles that raised the viscosity of the molten solder. Thus, this finding suggested that 0.50 wt% was the optimal TiO_2 and Al_2O_3 nanoparticles concentration at 250°C for the best result. The relationship between concentration of TiO_2 and Al_2O_3 nanoparticles and reflow temperature on the contact angle was excessive concentration of TiO_2 and Al_2O_3 nanoparticles and higher reflow temperature will increase the surface tension and also the viscosity of the molten solder and thus affecting the wetting performance of the nanocomposite solder.

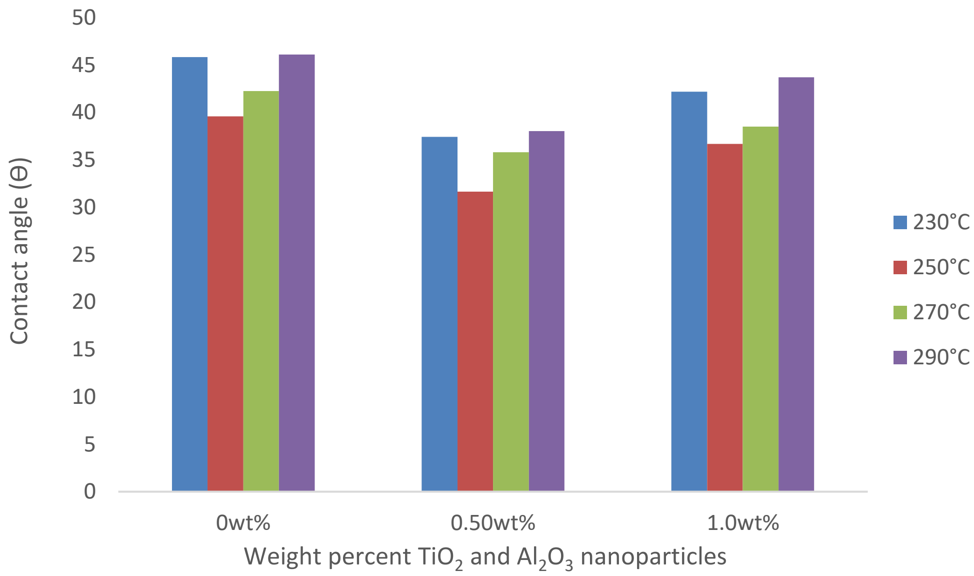

Fig. 2. Contact angle of SAC305 nanocomposite solder at different reflow temperature

Table 1
Contact angle of SAC305 nanocomposite solders at different reflow temperature

	Contact angle (Θ)			
Temperature (°C) / Sample	230	250	270	290
SAC305	45.82	45.45	45.97	46.09
Sn-3.0Ag-0.5Cu-0.5TiO_2-0.5Al_2O_3	37.41	31.65	35.81	38.02
Sn-3.0Ag-0.5Cu-1.0TiO_2-1.0Al_2O_3	42.17	36.69	38.49	43.72

3.3 IMC Growth Formation

Figure 3 shows the graph of IMC thickness under different reflow temperature at different concentration of TiO_2 and Al_2O_3 nanoparticles. IMC thickness of the solder was found to decrease at 0.50 wt% under 250°C reflow temperature then increase with increasing reflow temperature up to 290°C. For all SAC305 nanocomposite solder, it was observed that the IMC thickness in the solder is thickest at the highest temperature (290°C) as listed in Table 2. This observation shows that the TiO_2

and Al_2O_3 nanoparticles reacts with Cu substrate as the reflow temperature increases. It can be seen that thinnest IMC layer formed in SAC305-0.50TiO_2-0.50Al_2O_3 solder, a decrease from 1.44 to 1.40 µm at 250°C. When the weight percent of TiO_2 and Al_2O_3 increases, the IMC thickness increase from 1.80 to 1.95 µm for SAC305 solder, 1.44 to 1.55 µm for 0.50 wt% and 3.24 to 3.42 µm for 1.0 wt%. The findings show that the growth of IMC in liquid-state reactions can be inhibited by the addition of TiO_2 and Al_2O_3 nanoparticles, and that this inhibition is most effective when the concentration of TiO_2 and Al_2O_3 nanoparticles is increased to about 0.50 wt% at 250°C. Ceramic nanoparticles give significant enhancements on the properties of lead-free solder alloy include microstructures, thermal behaviours, mechanical properties, and growth of IMCs [10,13,23]. The refined Ag_3Sn IMCs phases proves TiO_2 and Al_2O_3 nanoparticles significantly restrict the growth of IMCs by acting as barrier between solder and substrate [13,14]. Thickness of the IMC layer decreases significantly when reinforced with 0.50 wt% of TiO_2 and Al_2O_3 nanoparticles but further addition of TiO_2 and Al_2O_3 nanoparticles could promotes growth of the IMC [13,14,18]. The thickness of the overall IMC layer decreases with an increase in TiO_2 and Al_2O_3 concentration along with the rise in reflow temperature. The decline trend starts when the weight percentage of TiO_2 and Al_2O_3 is at 0.50 wt%. IMC layer growth is elevated by an incremental increase in TiO_2 and Al_2O_3 concentration up to 1.0 wt%, though it remains thinner than that of the SAC305 solder joints.

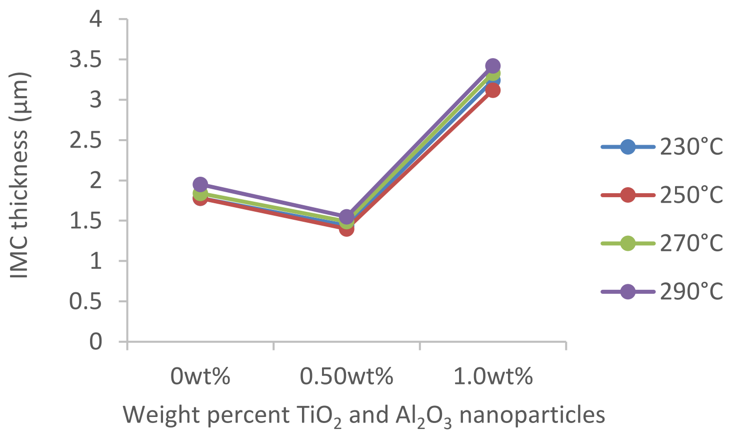

Fig. 3. IMC thickness of SAC305 nanocomposite solder at different reflow temperature

Table 2
IMC thickness of SAC305 nanocomposite solder at different reflow temperature

	IMCs thickness (µm)			
Temperature (°C) / Sample	230	250	270	290
SAC305	1.80	1.78	1.84	1.95
Sn-3.0Ag-0.5Cu-0.5TiO_2-0.5Al_2O_3	1.44	1.40	1.49	1.55
Sn-3.0Ag-0.5Cu-1.0TiO_2-1.0Al_2O_3	3.24	3.12	3.33	3.42

Similar findings by Tsao *et al.,* [14] and Chuang *et al.,* [19] imply that the reinforcement effect of Al_2O_3 nanoparticles is reduced once the amount reaches 1.0 wt%. The results discover that reinforcing TiO_2 and Al_2O_3 nanoparticles will restrict the growth of IMC layer under controlled concentration of TiO_2 and Al_2O_3 nanoparticles and reflow temperature. The growth of the IMC layer is restricted by the increased adsorption of Al_2O_3 nanoparticles, which decreases the surface free energy of the Ag_3Sn IMC grains, in accordance with the theory of the adsorption of surface-active materials [13]. Same goes for TiO_2 nanoparticles where it acted as reinforcements and barrier for solder dislocation [13]. TiO_2 nanoparticles adsorption effect inhibits the growth of the IMC layer [8,13,24]. This result was in accordance with other findings where the IMC layer thickness decreased by approximately by 50% when reinforcing 0.50 wt% of TiO_2 nanoparticles to SAC305 lead-free solder [13,15]. The adsorption effect of TiO_2 and Al_2O_3 nanoparticles not only restrict the growth of the IMC layer but also serves as a diffusion barrier at the interface between molten solder and Cu substrate.

4. Conclusions

SAC305-0.50TiO_2-0.50Al_2O_3 and SAC305-1.0TiO_2-1.0Al_2O_3 lead-free nanocomposite solders were successfully produced by reinforcing 0.50 wt% and 1.0 wt% of TiO_2 and Al_2O_3 nanoparticles respectively. At 217°C, SAC305 nanocomposite solders had a solidus temperature that was nearly identical to lead-free SAC solders. The SAC305 nanocomposite solders solidus and liquidus temperatures rise as the percentage of TiO_2 and Al_2O_3 nanoparticles increases, but still falls under the typical reflow temperature range for Pb-free solder, which is 240–250°C. The wetting performance of the solder system were improved under 250°C at 0.50 wt% concentration of TiO_2 and Al_2O_3 nanoparticles and falls under good standard of wetting angle between $20° < \theta < 40°$. This occurrence could be because of the reduced surface tension between the molten solder and Cu substrate in the presence of the TiO_2 and Al_2O_3 nanoparticles. On top of that, the IMC layer thickness was also improved under 250°C at 0.50 wt% concentration of TiO_2 and Al_2O_3 nanoparticles. The growth of the IMC layer thickness was restricted and controlled due to the adsorption effects of TiO_2 and Al_2O_3 nanoparticles and their roles as diffusion barrier and reduce the diffusion rate of Cu atoms to dissipate to the molten solder and react with Sn resulting in formation of Cu_6Sn_5 and Cu_3Sn IMC layer. Thus, this finding proposed that the addition of TiO_2 and Al_2O_3 nanoparticles improves the performance of SAC305 solder alloy under controlled concentrations of TiO_2 and Al_2O_3 nanoparticles and reflow temperature.

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