Physical properties of Sn-3.0Ag-0.5Cu lead-free solder with the additional of SiC particles

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Abstract. Composite solder has gained researcher’s attention due to its promising improvement in physical and mechanical properties for lead-free solder. To improve the properties of Sn-3.0Ag-0.5Cu (SAC) with the promising lead-free candidate, addition of silicon carbide (SiC) as a reinforcement was used in this study. This study was carried out to identify the effect of SiC particle on microstructure evolution and physical properties of SAC based solder alloys. SAC-SiC composite solder was synthesized by powder metallurgy method (PM), which consists of several processes such as mechanical blending, compaction and sintering. Three different weight percentages of SiC particles; 0.00, 0.50, and 1.00 were mechanically blended with SAC lead-free solder. The results show that the additional of particle SiC was able to refine the microstructure and reduced the size of β-Sn.

1 Introduction

It has long been well-known that tin-based solders, including SnAg, SnAgCu, and Sn-0.07Cu, were the most significant solders for electronic applications in industries [1, 2]. Many researchers still study the selection for Pb-free alternatives. SAC have attracted considerable attention as leading alloy Pb-free solder because of its greater mechanical properties [3]. Besides, SAC solder alloy was chosen due to their melting point which is relatively low and closer to Sn-Pb. However, formation of large Ag3Sn IMC within the bulk solder matrix due to the high amount of Ag is known to easily initiate cracks. Ag3Sn IMC which is brittle in nature can cause degradation in term of hardness performance and commonly will causes failure in portable electronic devices that used the solder [4]. In this research, SiC particles were chosen as reinforcement for SAC solders due to its light weight and ease of handling. The additional of SiC particles is believed be able to improve the microstructural, physical, electrical, mechanical properties, solder joint and IMC formation.

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issues. There is still insufficient data on the effect of the nano-size or micron-size SiC particle as reinforcement for SAC in the microstructure, thermal behaviour and mechanical properties when applied in soldering application [5]. Due to SiC have no solubility in β-Sn matrix, it is predicted that SiC will have significant effect on the SAC alloys. Thus, the aim of this study is to investigate the influence of micron-size SiC particles addition on microstructure, and microhardness properties of SAC[6].

2 Materials and Processing

SAC and SiC powder with particle size of < 45 µm were used in this research for the base matrix material and reinforcement respectively. The various composition of SiC reinforcement, 0.00 wt% as reference, 0.50 wt% and 1.00 wt% were used to fabricate new SAC-SiC composite solder via powder metallurgy technique (PM) [7,8]. The matrix of SAC solder was mixed with different composition of SiC in an airtight container using a planetary mill rotated at a speed 200 of RPM for an hour. After that, 12 mm diameter mould was used for compaction at a load of 4.5 tons (44.21 × 106 MPa) by using manual hydraulic press [9]. Next, the samples were sintered using a microwave at 185 °C for 2 minutes [10]. The sample of pure SAC which is without SiC also was prepared as reference sample [11].

The comparison between both pure and composite lead-free solders was studied on Cu-substrate (PCB FR-4 type). All the samples were cut into smaller pieces approximately 1 gram. Next, the pieces of bulk samples were placed on Cu-substrate with a no-clean (NC) paste flux type AFM037A that was provided by ASAHI Flux Medium. Finally, the samples were heated to a maximum peak temperature 250 °C in desk lead-free reflow oven [12].

3 Microstructure Analysis and Hardness Test

All samples were mounted for microstructure observation under the optical microscope. To observe the microstructure, mounted samples were ground by using sand paper from grit 600 to 2000. The samples that already ground need were soaked in ultrasonic cleaning to remove the impurities that remains on the surface of the samples. To reveal the microstructure, all samples were etched. The solution needs to be prepared for etching is the mixture of 2% of HCl, 3% of HNO₃ and 95% of methanol. Each of the samples were etched for 5 second to reveal the microstructure. This testing was refer based on ASTM B933-09 standard [13]. The compacted 12-mm diameter samples were tested using the Vickers Microhardness Test Model FV-700e. The testing was measured on the flat surface sample with 1 Kgf indenting load and 10 second dwell time [14].

4 Results and Discussion

4.1 Microstructure

Fig. 1 shows the optical images of pure SAC and SAC-\(x\)SiC (\(x = 0.5\) and 1.0 wt. %) composite solder. Based on result, β-Sn region for composite solder was decreased by increasing the amount of SiC particles. These findings proved that the addition of SiC particles able to retard the grain growth and gave rise to a finer β-Sn phase. Reinforcement concentration between grains will tend to prevent and limit grain boundary sliding and retard the grain growth[15]. Gain et al., [16] studied the addition of ZrO₂ nano-particles into
SAC found that the grain size was decreased due to precipitation of ZrO$_2$ at the grain boundary and change relative relationship of the growth velocity between the crystalline direction of the IMC particles, which reduces the IMC particles size [17]. Moreover, the SiC particles also functioned as the diffusive barrier to suppress metal atom diffusion on the surface of the grains. This process also contributed to delaying the growth of the grains, leading to finer microstructures [18]. The refined microstructure also contributed to the enhancement of microhardness value in the composite solder. Besides, Chen et al., [19] also found the addition of fullerene nanoparticles (FNSs) into the SAC were able to refine microstructure of the composite solder. The microstructure of SAC composite lead-free solders was observed by using SEM backscattered image.

![Fig. 1](image_url)

**Fig. 1.** Optical images of SiC as a reinforcement in; (a) pure SAC, (b) SAC-0.5SiC, and (c) SAC-1.0SiC

Fig. 1 (a), (b) and (c) shows the grain boundary and β-Sn matrix on the surface of pure SAC, SAC-0.5SiC and SAC-1.0. Fig. 2 (b) shows the observation area of SiC particle that were located between grain boundaries for SAC-SiC composite solders. Fig. 3 illustrates the location of SiC particles in SAC-SiC composite solder. The dark element is SiC particles. The SEM-EDX analysis result also support the existence of SiC particles within solidified SAC solder. The existence of Cu$_6$Sn$_5$ and β-Sn were also detected in SAC-SiC composite lead-free solder.
Fig. 2. SEM microstructures of: (a) pure SAC solder, and (b) SAC-1.0SiC composite solder.

Fig. 3. Point analysis of SAC-SiC.

4.2 Microhardness Test

Fig. 4 showed the comparison of microhardness’s data between the various compositions of SiC as a reinforcement for SAC lead-free solder. According to the Fig. 4, the microhardness of the composite solder rises with the increased of SiC particles compare to the pure SAC. The properties of SiC, a ceramic type material with high strength and high hardness, is the cause for enhancement of the properties of the solder. It believed the decreasing size of $\beta$-Sn region which has increased the microhardness of composite solder due to the classical dispersion strengthening theory[20]. From that theory, the foreign particles that located in the solder alloy matrix and grain boundaries are likely to change the solder alloy’s deformation characteristics by retarding dislocation movement as well as impeding grain-boundary sliding in solder matrix, resulting in enhancement in microhardness [19].
The additional of SiC particles influence the growth and size of the $\beta$-Sn region in SAC-SiC composite lead-free solder. Apart from that, the presence of reinforcement contributes to decreasing of solder matrix grain size. The microhardness of composite lead-free solder was increased because of dislocation path are disturb by reinforcing particle.

![Fig. 4. Microhardness value comparison](image)

The enhancement of microhardness value was attributed by the role of SiC particles as the pinning grain boundaries. The reinforcement that located in the solder matrix and grain boundaries were able to change the solder alloy’s deformation characteristics by retarding dislocation movement, as well as impeding grain boundary sliding in solder matrix, resulting improved in the microhardness [21]. That mechanism become an obstacle to the movement of dislocations and increased dislocation densities. The microhardness value of composite lead-free solders displayed consistently higher value than both pure SAC lead-free solders due to homogeneous distribution of SiC particles and refinement of the IMC particles[22]. In conjunction with the dispersion strengthening mechanism, the fine IMC particles and ceramic reinforcement can improve the microstructure and mechanical properties of a lead-free solder [16].

5 Conclusion

This study has proven with the addition of SiC particle in SAC lead-free solder are able to retard the grain growth and gave rise to the number of finer $\beta$-Sn phase. With that, the soft area of $\beta$-Sn matrix was decreased that able to enhance the microhardness properties of composite lead-free solder.

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