Reliability Study of Lead Free Sn-3.8Ag-0.7Cu and Copper (Cu) Substrate based on the Microstructure, Physical and Mechanical Properties.

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ABSTRACT

Solder alloys are vital in any electronic packaging industry especially in terms of its connectivity with the substrate. Mechanical properties are usually associated to prove and test the reliability of the solder and substrate. Ultimately, the customary Sn-Pb solders are known to be the ideal solders which solves the issue as mentioned. Sadly, this plumbum (Pb) contained solder alloy is harmful to the environment and human, leading to application of lead free solder alloys. Keeping that in perspective, this research investigates the mechanical properties of the lead free Sn-3.8Ag-0.7Cu (SAC) lead free solder alloy comprising the study in melting temperature, microstructure, wettability, shear strength and hardness. Melting temperature

ISSN 1823- 5514, eISSN 2550-164X

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of the SAC solder alloy falls below the required soldering temperature of 250°C. Investigation shows that this solder alloy joint with the copper (Cu) substrate provides a low contact angle of 24.8°. Microstructure study of SAC meanwhile shows a well-defined structure with dendrite shaped Sn-matrix with wider eutectic range, which contributes to the mechanical strengthening effect. The shear strength of this solder alloy was also noted to be as high as 44.84MPa together with high hardness value of 14.4Hv. All these results clearly satisfies the environment concern and importantly confirming the reliability of the solder and substrate.

Keywords: Sn-Ag-Cu Lead Free Solder Alloy, Wettability, Shear Strength, Hardness, Intermetallic Compound (IMC)

Introduction

Good mechanical properties together with good bonding between the SnPb solder alloy and substrates categorises this solder alloy system as the reference solder for other solders [1,2]. Phase diagram of the SnPb is shown in Figure. 1, which points the melting temperature together with the phases and compounds presence. Yet, its harmful effect has led to the banning of this solder alloy [3]. On that concern, many lead free solder alloys are being researched to comply as replacement for the lead solder alloy [4,5]. Nevertheless, the next problem statement arises when these lead free solder alloys needs to be having a good contact with the substrate to provide long lasting reliability. Lead free solders such as Sn-Zn, Sn-Cu, Sn-Zn-Bi and more are being studied and tends to be the alternative to say [6]. The Sn-Zn and Sn-Zn-Bi solder alloys prevails high tensile strength but these solder alloys are prone to oxidation causing corrosion and lack in the bonding between substrate [7, 8]. Therefore, the Sn-Ag-Cu solder system claimed by Chuang et al. produces better bonding between substrate and is commonly used as the alternative solder. Yet, research and evidence of the reason behind its profitability are less discussed. Such claims should be studied and versed for better evidence and understanding. In this research, the Sn-3.8Ag-0.7Cu (SAC) solder was investigated through its wetting, shear strength and hardness.



Figure 1: Phase Diagram of the Sn-Pb solder alloy.

Research Procedure

The preparation of SAC solder alloys were based on the weight percentage of 95.5% Tin (Sn), 3.8% Argentum (Ag) and 0.7% Copper (Cu), taken for a 20g overall composition. A small amount of composition was used to imitate the usage of small amount of solder pastes in the microelectronic industries. These elements were then melted in an induction furnace for 90 minutes at a constant temperature of 1100°C to ensure a homogeneous mixture of all elements. The SAC solder alloys were then compacted into billets forms for tests purposes. The SAC solder alloy billets later were polished and etched in Nital for 60 seconds to avoid debris before the tests and produce clean surface finish. The optical microscope (Meiji Techno) with the magnification of $10 \times$ and $20 \times$ was used for the microstructural analysis. The Differential Scanning Calorimetry (DSC) tests were conducted using the DSC 4000 (Perkin Elemer) to study the melting temperatures of the SAC solder alloy, which is important in determining the melting temperature. This experiment was done with a heat flow of 20.00°C/min with temperature from 100°C to 300°C for the SAC solder alloy. The graphs of the DSC curves are presented as the heat flow against the temperature. From the DSC test, the melting temperature was analyzed based on the onset, peak and end temperature. The onset temperature is the temperature where the solder starts to melt and is termed solidus temperature, T_S , while the peak temperature is the melting temperature, T_M and the end temperature is when the solder fully solidifies and termed as liquidus temperature, T_L. Wettability test was done to study the contact angle, Figure 2. (b) between the solder alloy and substrate, upon soldering onto the copper (Cu) substrate with dimensions of 10×10mm at 250°C, the suggested soldering temperature in the electronic packaging industry complying JIS H 3260 standard. The hardness test was conducted based on Vickers hardness (Wilson Wolpert) with 1kgf load applied to the SAC solder billet. Shear strength test meanwhile was done using the single shear lap test based on the ASTM D1002 specification for mechanical testing, Figure.2 (a) with the SAC solder billets soldered on the Cu substrate at the same temperature of 250°C using a hotplate. Crosshead speed of 0.01mm/s was applied to the SAC solder and Cu substrate to duplicate the slow fatigue usually faced by the solders in the electronic industries. The maximum shear strength was calculated by the aid of the Universal Testing Machine (Instron). Additionally, the X-ray diffraction (XRD) using the (ARL EQUINOX 3000 X-ray Diffractometer) analysis was done to confirm the presence of phases and compound for the SAC solder joint. All these result were analysed and discussed further in later in this paper.

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Figure 2. (a) Shear Test Specimen and Method and (b) Contact angle

Result and Analysis

The DSC in Figure. 3 indicates a single sharp endothermic peak which is the melting temperature of 218.98°C, 0.9% higher than the eutectic temperature in the SAC phase diagram. This result is due to the different atmosphere medium which is the nitrogen gas used in this DSC test. However, compared to other researchers such as Chuang et al., Chang et al. and Tsao & Chang, the melting temperature in this research was lower. The onset temperature of $T_s = 218.2^{\circ}C$ and an end temperature of $T_L = 221.9^{\circ}C$ was also plotted on the DSC curve. The pasty range is the difference between the melting temperature and solidus temperature and is one of the important factors that influences in providing a reliable joint [14]. The pasty range was noted to be 3.7°C for this SAC solder alloy. A short pasty range provides a faster solidification for the solder and it is important in providing better grain refinement and was observed for the SAC solder alloy [28, 29]. This result was supported by the research of Fouzder et al. for the Sn-3.0Ag-0.5Cu solder alloy. Phase diagram of Sn-Ag-Cu solder alloys confirms the melting temperature obtained in this research. Comparing to the Sn-Pb solder alloy ($T_M = 183^{\circ}C$), this solder produced higher temperature due to ternary element application, yet the desired soldering temperature still falls under desired soldering temperature (< 250°C) [11].



Figure 3. DSC result of SAC solder alloy, (a) Phase diagram of Sn-Ag-Cu.

Wettability

Wettability is the ability of a solder alloy to spread on the substrate which forms metallurgical bond influenced by surface tension, wetting time, contact angle and spreading area of a solder joint [10,11,12,13]. In this research the contact angle used as the parameter to study the wettability. The SAC solder alloy produces contact angle values below ($<30^\circ$) with an average of 24.81°, as shown in Table 1. Similarly, this result was supported by the research of Kotadia et al. and Fallahi et al. by producing contact angles of 28.38 and 29.6° for the Sn-3.8Ag-0.7Cu and Sn-3.6Ag-0.9Cu solder alloys respectively. In another research by Shen and Chan, the contact angle of the Sn-3.5Ag-0.5Cu was 38°, contradicts with the result in this study. The reason is that the temperature used in that research was 230°C, less 20°C than the applied temperature in this study and therefore at low temperature soldering, it produces high surface tension which denies the solder to spread easily and increase the contact angle [17]. Thus, the SAC for this research clearly being compatible with the soldering temperature and at the same time producing low contact angle which produces thinner intermetallic compound (IMC) layer and improves the mechanical property. This statement was likewise supported by [18].

Trials	Contact Angle (°)	
1	24.98 ± 1	
2	26.50 ± 1	
3	26.00 ± 1	
4	24.15 ± 1	
5	22.42 ± 1	
Average	24.81 ± 1	

Table 1. Contact angle of SAC solder alloy.

Microstructure

The microstructure analysis of the SAC solder alloy, Figure. 4 shows dendrite like light phases and dark phases. The microstructure of this SAC alloy consist of coarse Sn-grains, which is identified to be the light phase, while the dark phase is the eutectic regions which consists of two phases mainly in the form of intermetallic compounds. This observation correlates with the study of Yu et al. and Che et al. as in Figure. 4(a) and (b) in this study. The reason for this different phase and microstructure formation is due to the solidification process [33]. During solidification, a sufficient driving force will be built up and then, Sn nucleation will start to occur [34]. Later, the nucleation will be

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followed by the growth of the dendrite Sn in the molten solder where by the dendritic structure is appeared to be dependent on the content of the Ag and Cu [28]. The Sn dendrites will form earlier than the Sn matrix in eutectic phase, while at the Sn matrix; the dendrites grow quickly and form large dendrite structure [35]. The uniform microstructure of solder alloy increases the hardness by resisting any deformation and dislocation from taking place easily in the solder [26]. Also noted in this study is the uniform microstructures as portrayed in Figure. 4. This particular characteristic is important in giving a good strengthening effect to a solder especially in terms of hardness. The result was in agreement with the study of Roubaud et al. and Laurila et al. Apparently, these findings are supported and noted in this research and explained in later part of this paper. The microstructures of the SAC solder alloy gives an initial reason for the high hardness that was produced during the hardness test. Once more, it is due to the uniform distribution of the microstructure with Sn-matrix and the eutectic phase finely dispersed which gives the strengthening effect of the solder alloy.



Figure 4: Microstructures of SAC solder alloy, (a) $10 \times$ and (b) $20 \times$, magnification.

Shear Strength

Shear strength of a solder alloy primarily depends on the microstructure of the solder alloy itself and also the IMC layer formation between the solder alloy and substrate [19], Figure 5. The average maximum shear strength for the SAC solder alloy joint was 46.68MPa (Table 2) in this research. One of the factor is because of the eutectic composition of SAC used in research. Typical characteristics of a eutectic compositions are known to have a higher surface area per unit volume and this directly contributes in providing higher strength [20]. Adding to that, the low contact angle of the SAC solder alloy enables the joint to be more reliable by producing thin IMC layers containing the Cu₆Sn₅ intermetallic layer as confirmed by the XRD result in Figure 6. Contrasting to a thick IMC layer which have more exposed surface which acts as stress concentrator that will cause the crack to initiate and propagate. It is understood

that a thick IMC layer for the Sn-Ag-Cu solder system usually occurs when the Cu₃Sn is be formed due to the solid-state reaction, where a new layer of stacks will be formed between the solder and substrate, in between the solder/Cu₆Sn₅ and Cu₆Sn₅/Cu. This process happens when Cu atoms from the substrate arrive at the interface of Cu₆Sn₅ by diffusion through grain boundaries and then a part of Cu₆Sn₅ will be converted to a Cu₃Sn IMC layer. Opposing to that, the Cu₃Sn from the XRD analysis in this study denotes less peak confirming that this layer is not having a profound effect. This is because the Cu₃Sn layer forms in a minor scale and its presence are hardly noticed, contributing to thin IMC layer. The production of the Cu₆Sn₅ layer occurs as a part of diffusion of the Sn element in the solder towards the Cu element in the substrate during soldering [21]. A thin IMC layer consists of these Cu₆Sn₅ IMC layers providing barrier for sliding to occur easily and such action increases the strength of the solder joint. This explanation was agreed by Yang et al. and in the same research mentioned that a thin continuous and uniform IMC layer is an essential requirement for a good bonding while a thick IMC layer is prone to cracking and endures failure to the solder joint. Adding to that, the SAC solder alloy joint produces better strength which can be witnessed as some of the sample for the shear test had ruptures/break on the Cu substrate rather than at the joint. This physical observation as in Figure. 7 verifies a good bonding between the SAC solder and substrate. In a normal shear test, as the force is concentrated to the solder alloy joint area, the ruptures take place at that point. Vice versa, a strong joint will withstand the stress at the joint making the rupture to occur elsewhere, at the Cu substrate in this study. This observation was noted in the research of Ervina and Singh.



Figure 5. Solder/Substrate joint compounds representation.

Table 2. Shear Strength of the SAC solder alloy joint.

Trials	Shear Strength (MPa)
1	44.90
2	42.99
3	46.68
Average	44.86



Figure 6. XRD of SAC solder alloy joint.



Figure 7. Crack at the Copper (Cu) substrate.

Hardness

The ability of a material to resist impact or fracture during service time can be termed as a material's hardness [23]. The Vickers hardness test was used in this research to portray the load resistance of the SAC solder alloy. The results of the SAC hardness are shown in Table 3 with an average value of 14.4Hv attained. This result was supported by the research of Chang et al. suggesting that the SAC solders system are highly resistance to plastic deformation due to its ternary composition [25]. Moreover, the SAC solder alloy contains the IMC of Cu_6Sn_5 and Ag_3Sn after diffusion had taken place between the elements during meting and soldering. Thus, the pivotal reason for this high hardness is

the presence of these IMC that takes the role as obstacles and hinders the motion of dislocation of an applied impact or load. Existence of the IMC are noted in the XRD result in Figure. 2. These characteristics was also alike to the explanation made the study of Lin et al..

Table 3. Vickers hardness of SAC solder alloy.

Conclusion

Trials	Vickers Hardness (Hv)
1	14.6
2	14.4
3	14.3
4	14.8
5	14.1
Average Vickers Hardness (Hv)	14.4

Electronic packaging industries are facing major problem of getting the ideal lead free solder alloy to replace the lead solder because of some mechanical reliability issue with the alternative lead free solder alloys. On that, this research produces the solution with the aim to solve the problem by introducing the SAC lead free solder alloy. The low melting temperature produced by the SAC ($T_M = 218.98^{\circ}C$) denotes the ability of this solder alloy to be used in the electronic packaging industry without damaging other component due to high temperature and at the same time sustains thermal conductivity. Essential properties such as the wettability produces a low contact angle of 24.8° assuring a better bonding of this SAC solder alloy. Microstructure of the SAC produced dendrite like shape Sn matrix combined with eutectic area that assists in better mechanical properties. The XRD test confirm the presence of the IMC's that also contributes to a better mechanical properties. Mechanical tests on shear strength and hardness were continued in this research to prove the SAC solder alloys reliability where most research's lack to convey. High shear strength of 44.84MPa with some physical evidence of rupture on the substrate ultimately proves the SAC solder alloy is well joined with the substrate. These attributes are correlated to the low contact angle produced, assuring a thin IMC layer that avoids crack propagation. In addition, the resistance of the SAC solder alloy to produce high Vickers hardness value of 14.4Hv at the same time provides the confirmation of the mechanical beneficiary of this solder alloy. In a general conclusion and with the results as affirmed above, this SAC solder alloy can be the solution implemented to the problem faced in the electronic packaging industry.

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