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Influence of small addition of antimony (Sb) on thermal behavior, microstructural and tensile properties of Sn-9.0Zn-0.5Al Pb-free solder alloy



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ABSTRACT

Eutectic Sn-Zn alloy is considered as one of the best lead free solder alloys in microelectronic industry. That motivates our group to select different weight percentage of Antimony (Sb) (0.5, 1.0, and 1.5 wt%) as an alloying to Sn-9.0Zn-0.5 Al solder alloy. The thermal behavior, microstructure modification as well as tensile properties of the new developed solder alloys were investigated. A slight increment of the melting temperature (~ 1 °C) was recorded using differential scanning calorimetry (DSC) after additions of Sb. For 1.5 wt% of Sb, two endothermic peaks at 200.8 °C and 201.5 °C were observed, which are assigned as hypoeutectic Sn-Zn composition. X-ray diffraction (XRD) measurements confirm the existence of β-Sn phase, α-Zn phase, and Sb-Sn intermetallic compounds (IMCs). Scanning electron microscope (SEM) images indicate that the Sb additives refine the microstructure and form a uniform distribution of IMCs in the matrix of solder. The road-like α -Zn phase, Al₆Zn₃Sn and SbSn IMCs were clearly appeared in β-Sn matrix, which are responsible of the enhancement in tensile strength. Moreover, α -Zn phases in the Sn-9Zn-0.5Al-1.5Sb alloy were modified as needle-like, broken enormously, depleted, and circle shapes. Generally, The Sb-containing alloys have higher ultimate tensile strength (UTS) and lower elongation than Sb-free solder alloy due to the solid solution and second phase dispersion strengthening effect. The relationship between UTS and temperature follow the Arrhenius law. The average activation energies (Q) were found to be $44.4 \pm 1.0 \, \text{kJ/mol}$, and the average stress exponents (n) were usually around 5.3 \pm 0.45, which are close to pipe diffusion controlled creep in β -Sn matrix.

1. Introduction

For the sake of the environmental protection and human health concerns, the international legislations prevent using of lead (Pb) at any products due to its high toxicity [1]. So, eco-friendly microelectronics industry encourages the lead-free solder(LFS) alloys products [1,2]. Despite the great efforts of many research groups to find a suitable alternative to Sn-Pb alloys, the investigation of LFS alloys are still competed and need more efforts. Over the past years, most researchers have been focused on Sn-Ag, Sn-Cu, Sn-Sb and Sn-Zn alloys as possible alternatives [1–4]. Notably, the eutectic Sn-Zn solder alloy was introduced as special interest because of its good mechanical properties, creep resistance, reliable soldering performance and low melting point (198 °C) which close to the Sn-37Pb solder alloy (183 °C) [5–8]. Even though, the employment of Sn-Zn solder alloys in microelectronic applications have remained restricted due to their poor wettability, low oxidation resistance and high corrosion rates due to high activity of Zn atoms [9]

In recent years, to overcome the shortfalls of Sn-9Zn alloy, some

authors have tried to add a third element, such as Bi [6–10], Ag [11–17], Cu [18,19], Ni [20], Ga [21–27], Ce [28] and rare earth elements [29–31], to improve their properties. Lin and Liu [32,33] added the Al element into the Sn-Zn alloy to improve the wettability property and oxidation resistance. Chen et al. [22] observed that, the ultimate tensile strength (UTS) and ductility of Sn-9.0Zn solder alloy were lowered after adding 0.45 wt% Al element. Meanwhile, McCormack and Jin [34] reported that alloying addition of 5 wt% In to Sn–Zn system reduced the melting temperature to 188 °C as well as enhanced their ductility and wettability. Recently, Antimony has been identified as one of the effective alloying element because of its considerable melting temperature, higher boiling point and excellent wettability [2].

Chen and Li. [35] reported that minor additions of Sb into Sn-Ag-Cu solder alloy depressed the activity of Sn by forming Sb-Sn IMC which dispersed in β -Sn matrix. Moreover, the presence of Sb-Sn IMC increased the rate of nucleation and refined the grain size [35–37]. Furthermore, Shifiq et al. revealed that adding small amounts of Sb to the Sn-Zn system refined the bulky needles of Zn as well as the β -Sn matrix and improved their strengthening by solid solution hardening [19].

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Table 1
Compositions of the solder alloy (wt%).

Alloy	Sn	Zn	Al	Sb
Sn - 9Zn - 0.5Al	Bal.	9.02	0.51	0.00
Sn - 9Zn - 0.5Al - 0.5Sb	Bal.	9.01	0.50	0.51
Sn - 9Zn - 0.5Al - 1.0Sb	Bal.	9.02	0.52	1.03
Sn - 9Zn - 0.5Al - 1.5Sb	Bal.	9.02	0.52	1.52

So far, the literature survey exhibited rare studies have been performed to reveal the effect of Sb alloying element on the physical properties of Sn-Zn-Al solder alloys. Therefore, the present study aimed to investigate the effect of Sb alloying element on the physical characteristics of Sn-9.0Zn-0.5Al solder alloy based on thermal analysis, microstructural, and tensile properties. The tensile deformation behavior carried out in terms of various testing temperature and different strain rates $(\dot{\epsilon})$ for all alloys. In this study, we have chosen small percentages of the Sb added to Sn-Zn-Al solder alloy for several considerations. First, small percentage make a solid solution of Sb with Sn which is a major constituent of the solder alloy. If the percentage of Sb increased to 10%, an extensive formation of the intermetallic phase will appear, which effects on desired mechanical properties of our solder alloy. Furthermore, The National Center for Manufacturing Sciences (NCMS) reported that "Sb is highly toxic when inhaled or ingested and has the same Lethal Dose, Lower Limit (LDLo) as Cd." [1]. So, we must use a small amount of Sb.

2. Experimental procedures

The Sn-9.0Zn-0.5Al-x Sb (x = 0.0, 0.5, 1.0, and 1.5 wt%) solder

Temperature °C

(b) Sn-9Zn-0.5Al-0.5Sb (a) Sn-9Zn-0.5Al T_c=198.7 °C T = 205.2 °C 204.7°C 198°C 0 0 -5 Heat Flow (mw) -5 mw) A/m = 76.2 J/g-10 A/m = 66.2 J/g-10 Heat 15 -15 T_m= 201.1 °C -20 -20 T = 200.2 °C -25₁₉₀ 206 208 -25₁₉₀ 192 194 196 198 200 202 204 200 202 204 206 208 Temperature °C Temperature °C (c) Sn-9Zn-0.5Al-1.0Sb (d) Sn-9Zn-0.5Al-1.5Sb T₁ = 204.3 °C T₂= 198.3 °C T_i = 206.1 °C T₀ =198.1 °C 0 Flow (mw) Heat Flow (mw) A/m = 62.7 J/gA/m = 74.1 J/gT_m= 201 °C -15 -20 **-**20 T_{m1}= 200.8 °C = 201.5 °C 192 194 198 200 202 204 206 208 196 200 202 204 206 208

Fig. 1. DSC curves for the various solder alloys with the following composition (a) Sn-9Zn-0.5Al, (b) Sn-9Zn-0.5Al-0.5%Sb, (c) Sn-9Zn-0.5Al-1.0%Sb and (d) Sn-9Zn-0.5Al-1.5%Sb.

Table 2 Comparison of solidus temperature (T_{onset}), liquidus temperature (T_{end}), the melting range, fusion heat Δ H for various solder alloys.

Solder alloy	T _{onset} °C	T_{end} $^{\circ}C$	Melting range (°C)	∆ H (J/g)	References
Sn – 37Pb Sn – 3Ag – 0.5Cu Sn – 9Zn – 0.5Al Sn – 9Zn – 0.5Al – 0.5Sb Sn – 9Zn – 0.5Al – 1.0Sb Sn – 9Zn – 0.5Al – 1.5Sb	179.5 217.0 198.0 198.7 198.1 198.3	191.0 221.0 204.7 205.2 204.3 206.1	11.5 4.0 6.7 6.5 6.2 7.8	104.2 184.8 163.9 142.3 134.8	[38] [38] This study This study This study

alloys were prepared from bulk Sn, Zn, Al, and Sb rods with high purity (99.99%). The process of melting was performed in electric furnace at 650 °C for 1 h. Then, each alloy was remelted three times to obtained more homogeneity and casting in steel mold. The chemical compositions of the investigated solder alloys are listed in Table 1. The cooling rate (~ 5 °C/s) is estimated by using a thermocouple placed in the solder which is equivalent to practical reflow process in industry. For metallographic observations, as-solidified specimens were neatly polished using 3 μ m and 1 μ m alumina powder which suspended in distilled water as a lubricant. Nearly mirror-like surface was obtained by using 0.3 μ m diamond paste. The polished samples were chemically etched in a solution of 80% glycerin, 10% nitric acid and 10% acetic acid for 10 s. A scanning electronic microscope (SEM) [JEOL model JSM-5410, Japan] was employed to examine the microstructure of the prepared alloys. An energy dispersive X-ray spectroscopy (EDX) was

Temperature °C

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