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Microstructure and mechanical properties of Sn–58Bi eutectic alloy with Cu/P addition

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Abstract

Sn–(58-x) Bi–x Cu/P ternary alloys were prepared by downward continuous casting, and the microstructure of the alloy was characterized using scanning electron microscopy (SEM), x-ray diffractometry (XRD) and differential scanning calorimetry (DSC). The results show that the addition of Cu and P can refine the eutectic structure and form rod-shaped Cu6Sn5 and P3Sn4 phases distributed in Sn matrix. The refined eutectic structure can be observed in Sn–(58–x) Bi–x Cu/P alloys, and this results in the elongation at break increases up. In addition, the wettability of Sn–58Bi alloy increases on Cu substrate with the addition of Cu and P elements. The improvement of the wettability of Sn–58Bi alloy by the addition of Cu element can be attributed to the increase of Cu–Sn IMC nucleation and growth rate. The addition of P element in Sn–58Bi alloy can improve its anti-oxidation performance, which is beneficial to the improvement of its wettability.

1. Introduction

Sn–Pb alloys have been widely used in electronic packaging for many years. Nevertheless, lead is harmful to human health and the environmental safety [1–3]. A suitable candidate for lead-free alloys have become a research hotspot to solve this problem in recent years, such as Sn–Ag–Cu [4], Sn–Cu [5], Sn–Zn [6], and Sn–Bi [7, 8] alloys. Among them, Sn–58Bi alloy has low melting point [9, 10], good fracture strength and creep resistance [11], which is considered as an ideal candidate for traditional Sn–Pb alloy [12, 13]. However, the extremely poor ductility renders Sn–58Bi alloy difficult to process into wires or welding sheets. The previous studies had shown that the wettability and elongation at break of Sn–Bi alloy can be effectively improved by micro-alloying, including Cu [14–16], Ag [17] Fe [18, 19] and Re [20], etc. In fact, several related studies have been conducted, focusing on various Sn–Bi hypoeutectic alloys. For example, Sn–40Bi hypoeutectic alloy was prepared as cast in iron mold by several researches. According to Wu et al [15] an optimized composition of Sn–40Bi–0.3Cu alloy has good elongation at room temperature and high temperature respectively. H Takao et al [21] reported that the elongation of Sn–40Bi–0.1Cu alloy reached 171% at room temperature after long-time heat treatment. Dong et al [22] reported that the anti-oxidation performance and wettability of Sn3.0Ag0.5Cu solder were improved with the addition of P. However, the research on the properties of Sn–(58-x) Bi–x Cu/P alloy needs to be further deepened. Many efforts are still in need to understand the comprehensive behavior related to Sn–(58-x) Bi–x Cu/P.

The improvement of preparation method can improve the processing behavior of Sn–Bi alloy. Research on Sn–58Bi eutectic alloy has focused mainly on traditional processes, such as iron mold casting and semi-solid squeeze casting. It is worth noting that there is little research exists on downward continuous casting. In previous studies, Chen et al [23] investigated the tensile properties of the eutectic Sn–58Bi alloy prepared by iron mold casting. Tensile tests were carried out at the drawing speed of 2 mm min−1. It was found that the ductility value was 20% for Sn–58Bi. S Sakuyama and co-authors [13] revealed ductility value of 10% for Sn–58Bi. Tensile tests were carried out at the drawing speed of 3 mm min−1. The downward continuous casting method permits the generation of continuous high-density wire rod with uniform composition, and reduces the defects,
e.g. shrinkage cavity or hole. In this paper, tensile tests were carried out at the drawing speed of 10 mm min$^{-1}$. It was found that the ductility value of Sn–58Bi alloy was prepared by downward continuous casting method can reach 30%. Moreover, the casting rod with a diameter of 6mm also has been prepared in self-developed downward continuous casting device. It can be inferred that Sn-58Bi casting rod with smaller diameter also can be prepared by adjusting the size of guide rod in a certain range, and that conducive to shorten the preparation process of wire.

In this paper, Sn–(58–x) Bi–x Cu and Sn–(58–x) Bi–x P alloys were prepared by self-developed downward continuous casting device. Furthermore, the effects of Cu/P on the microstructure and properties of Sn–58Bi alloy were studied with a further discussion on the influence mechanism.

2. Experimental procedure

Sn–(58–x) Bi–x Cu ($x = 0.1$–$1.0$) and Sn–(58–x) Bi–x P ($x = 0.01$–$0.1$) alloys were prepared with Sn (99%), Bi (99%), Sn–5P master-alloy and Sn–55Bi–3Cu master-alloy. According to ALAM et al [16], Cu has a very low solubility in both Sn and Bi. It is expected that addition of a higher amount of Cu to the Sn-Bi eutectic solder alloy will lead to the accumulation of Cu at the Sn-Bi grain boundary. Moreover, more Cu addition made the binary Sn-Bi eutectic alloy’s melting temperature increase. Therefore, the addition of Cu will not exceed 1 wt.%.

The addition of trace P can improve the anti-oxidation performance and wettability of the alloy [24]. However, P is easy to form brittle phase with Sn. It was found that when the content of P was 0.1 wt.%, the plasticity was poor. Therefore, in this paper three groups of alloy compositions were selected in the range of 0 $\sim$ 0.1 wt.%. The compositions of the alloys are listed in table 1. The casting rod with a diameter of 8mm was prepared in self-developed downward continuous casting device, as shown in figure 1. The pitch and casting speed of the traction unit are 0.3 mm and 50.4 mm min$^{-1}$ respectively with casting temperature of 190 $\pm$ 10 $^\circ$C.

The melting behavior of the alloys were determined by differential scanning calorimetry (DSC, HSC4, Beijing, HENVEN) under Ar atmosphere protection. The mass of the sample and heating rate were 30 mg and

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Table 1. Designated composition of the solder alloys.

| Number | Alloy                | Components (wt.%) |
|--------|----------------------|------------------|
|        |                      | Sn   | Cu | P  | Bi  |
| S1     | 42Sn58Bi             | 42   | 0  | 58 |
| S2     | 42Sn57.9Bi0.1Cu      | 42   | 0.1| 57.9|
| S3     | 42Sn57.5Bi0.5Cu      | 42   | 0.5| 57.5|
| S4     | 42Sn57.0Bi1.0Cu      | 42   | 1.0| 57.0|
| S5     | 42Sn57.99Bi0.01P     | 42   | 0.01| 57.99|
| S6     | 42Sn57.95Bi0.05P     | 42   | 0.05| 57.95|
| S7     | 42Sn57.9Bi0.1P       | 42   | 0.1| 57.9|
5 °C min \(^{-1}\) respectively. The alloy microstructure was observed using scanning electron microscopy (SEM, MLA650F, FEI Company, USA) equipped with energy dispersive spectrometer (EDS, QUANTAX400, Bruker, Germany). The alloy samples were polished with SiC abrasive paper from 600 mesh to 2000 mesh, and etched with a 3vol% \(\text{FeCl}_3\) + 6vol% \(\text{HCl}\) + 91vol% \(\text{H}_2\text{O}\) solution. In addition, energy-dispersive x-ray spectroscopy (EDX) and x-ray diffraction (XRD, Xpert powder, PANalytical B.V., The Netherlands) were performed to analyze the phase compositions of alloys.

Tensile tests were carried out on tensile testing machine (UTM5105) at room temperature with a strain rate of 10 mm min \(^{-1}\). Three samples were selected for each composition to measure the tensile strength and elongation at break. According to the Japanese Industrial Standard JIS Z 3198-3, the wettability of the alloy was measured on pure copper plate (C1220P) at 170 ± 2 °C for 30 s respectively. The flux is composed of rosin (25 ± 0.1 g) + acetone (75 ± 0.1 g) + diethylamine hydrochloride (0.39 ± 0.1 g). The spreading rate (\(S_R\)) of solder alloy can be defined by equation (1):

![Figure 2](image1.png)  
**Figure 2.** Tensile strength and elongation of: (a) Sn–(58-x)Bi–xCu alloys, (b) Sn–(58-x)Bi–xP alloys.

![Figure 3](image2.png)  
**Figure 3.** XRD patterns of: (a) Sn–58Bi, (b) Sn–57.5Bi–0.5Cu, (c) Sn–57Bi–1.0Cu, (d) Sn–57.99Bi–0.01P, (e) Sn–57.9Bi–0.1P.
where $S_R$ is spreading factor ($\%$), $D$ is the diameter (mm) of the same mass solder ball, and $H$ is the height (mm) of solder joint.

3. Results

3.1. Mechanical behavior

Figure 2 shows the curves of tensile strength and elongation at break of Sn–(58–$x$) Bi–$x$Cu and Sn–(58–$x$) Bi–$x$P alloys. According to figure 2(a), the tensile strength and elongation at break of Sn–58Bi alloy are 71.05 MPa and 30% respectively. The tensile strength increased with the increase of Cu content, while the elongation increased first and then decreased. The maximum elongation was 41.67% in Sn–57.5Bi–0.5Cu alloy, which was 38.9% higher than that of Sn–58Bi alloy. The addition of P element also affected the mechanical properties of Sn–58Bi alloy. In fact, the addition of P decreased the tensile strength slightly from 71.05 MPa to 67.57 MPa, whilst the elongation increased from 30% to the peak value of 38.33% increasing 27.8% compared with Sn–58Bi alloy, as shown in figure 2(b).

3.2. Microstructure characterization

The XRD patterns of Sn–(58–$x$) Bi–$x$Cu ($x = 0, 0.5, 1.0$) and Sn–(58–$x$) Bi–$x$P ($x = 0.01, 0.1$) alloys are shown in figure 3. It can be seen from figure 3(a) that Sn–58Bi alloy is primarily composed of $\beta$–Sn phase and pure Bi...
phase, while new diffraction peak can be observed in Sn–(58-x)Bi–xCu alloys. The diffraction peaks are Cu6Sn5 phase (figure 3(a)) and P3Sn4 phase (figure 3(b)) respectively after calibrating.

Figure 4 shows the SEM images of Sn–58Bi, Sn–(58-x)Bi–xCu (x = 0.5–1.0) and Sn–(58-x)Bi–0.1P alloys. EDS analysis were carried out to identify the phase compositions of the region marked in figure 4, and the corresponding results are listed in table 2. According to the Sn–Bi binary phase diagram, Sn–58Bi alloy is primarily composed of Bi-rich phase, \( \beta \)-Sn phase and a second precipitated Bi phase [24, 25]. The region 1 in Sn–58Bi alloy (figure 4(a)) was \( \beta \)-Sn phase dissolved with 2.61 wt.% Bi element from EDS analysis. The region 2 should be a Bi-rich phase with 99.88 wt.% Bi. The addition of Cu can refine the eutectic structure. The size of \( \beta \)-Sn phase in Sn–57.5Bi–0.5Cu alloy decreased with 0.5wt% Cu addition, as shown in figure 4(c). Moreover, the amount of \( \beta \)-Sn phase increased significantly, and the distribution was more uniform, which revealed that the microstructure was significantly improved. In addition, rod-shaped phases (region 3) were found in alloys with 0.5 wt.% and 1.0 wt.% Cu. EDS analysis showed that the rod-shaped phase was mainly composed of Cu (37.56 wt.%) and Sn (61.43 wt.%). Combined with the XRD analysis, the rod-shaped phase was Cu6Sn5 phase in region 3.

The size of Bi-rich phase exhibited a distinct refinement with the addition of P element, as shown in figures 4(d)–(f). In addition, rod–shaped phase was distributed in Sn–57.95Bi–0.05P alloy (figure 4(b)) and Sn–57.9Bi–0.1P alloy (figure 4(c)). Compared with Sn–57.95Bi–0.05P alloy, Bi–rich phase has spherical or irregular rod–shaped morphologies, and eutectic structure was significantly refined. The number of rod-shaped phases increased in Sn–57.9Bi–0.1P alloy (figure 4(f)). EDS analysis of Sn–57.9Bi–0.1P alloy showed that the

| Table 2. EDS analysis results. |
|-----------------------------|
| Point | Sn (%) | Bi (%) | Cu (%) | P (%) |
| 1     | 97.39  | 2.61   | 0      | 0     |
| 2     | 0.12   | 99.88  | 0      | 0     |
| 3     | 61.43  | 1.04   | 37.53  | 0     |
| 4     | 79.55  | 3.98   | 0      | 16.47 |

| Table 3. The DSC results of Sn–(58-x)Bi–xCu/P alloys. |
|-----------------------------|
| Alloy | Main peak/°C | Onset point/°C | Offset point/°C | Range/°C |
| S1   | 131.02        | 127.35          | 135.37          | 8.02     |
| S2   | 130.08        | 126.24          | 135.10          | 8.86     |
| S3   | 131.8         | 127.24          | 135.13          | 7.89     |
| S4   | 130.95        | 127.18          | 135.63          | 8.45     |
| S5   | 130.71        | 126.96          | 135.68          | 8.72     |
| S6   | 131.9         | 128.21          | 136.57          | 8.36     |
| S7   | 131.46        | 127.60          | 136.64          | 9.04     |
rod-shaped phase in Sn–57.9Bi–0.1P alloy was mainly composed of P (16.47 wt.%) and Sn (79.55 wt.%). Combined with the XRD analysis, the rod-shaped phase in Sn–57.9Bi–0.1P alloy was P3Sn4 phase.

3.3. Melting properties

Low-temperature solder forming a reliable solder joint should have low melting point and a narrow melting range, i.e., the difference between the onset point and the offset point. Figure 5 shows the DSC curves of Sn–58Bi, Sn–(58–x) Bi–x Cu (x = 0.1–1.0) and Sn–(58–x) Bi–x P (x = 0.01–0.1) alloys. The DSC results of Sn–(58–x) Bi–x Cu/P alloys was shown in table 3. Sn–58Bi binary eutectic peak decreased from 131.02 °C to 130.95 °C with the addition of Cu (figure 5(a)), whereas Cu addition extended melting range to 8.45 °C slightly. It can be seen from figure 5(b) that the addition of P lead to a 0.3°C decrease of the onset point and a 1.3°C increase of the offset point. The melting range reached 9.04 °C. It can be found that there is little difference in the

Figure 7. Typical SEM images of solder joints with: (a) interfacial morphology of Sn–58Bi/Cu, (b) EDS line scan of Sn–58Bi/Cu, (c) interfacial morphology of Sn–57Bi–1.0Cu/Cu, (d) EDS line scan of Sn–57Bi–1.0Cu/Cu, (e) interfacial morphology of Sn–57.9Bi–0.1P/Cu, (f) EDS line scan of Sn–57.9Bi–0.1P/Cu.
melting range and peak temperature of these alloys, indicating that the addition of trace Cu and P elements do not make an obvious change to the melting point and melting range.

3.4. Wettability
The wettability is an important characteristic for solder alloy. After preparing solder joint on Cu substrate, the height of Sn–58Bi, Sn–(58–x) Bi–x Cu (x = 0.1–1.0) and Sn–(58–x) Bi–x P (x = 0.01–0.1) solder joints were tested. The spreading rate (SR) of the alloy was calculated using formula (1) in wettability experiment, and the effect of the addition of Cu and P on wettability of the Sn–58Bi eutectic alloy was studied, as shown in figure 6. It can be found that Sn–58Bi alloy had good wettability, and the spreading rate reached about 70%. The spreading rate of the alloy increased slightly with the addition of Cu and P elements. When the Cu content was 1.0 wt.%, the spreading rate of Sn–57Bi–1.0Cu alloy was about 73% (figure 6(a)). The spreading rate of Sn–57.9Bi–0.1P alloy reached to 72.6% (figure 6(b)).

The solder melts into a liquid state and spreads on the surface of Cu substrate to form interfacial compounds (IMCs) during welding. To investigate the effect of Cu and P elements on IMCs, the interfacial compound layers (IMLs) of Sn–58Bi/Cu, Sn–57Bi–1.0Cu/Cu and Sn–57.9Bi–0.1P/Cu were examined using EDS line scan, as shown in figure 7. The microstructure of Sn–58Bi/Cu solder joint was primarily composed of eutectic structure and a small amount of β-Sn phase. A thin IMC layer was observed in the IML, as shown in figure 7(a). With the EDS line scan of IML of Sn–57Bi–1.0Cu/Cu, it confirmed that the IML was mainly composed of Cu and Sn elements, and Bi had not participated in the reactions of IML. In addition, a refined Bi-rich phase was observed in Sn–57Bi–1.0Cu alloy (figure 7(c)). The interface between Sn–57Bi–1.0Cu alloy and IML was mainly Bi-rich phase according to the EDS line scan of Sn–57Bi–1.0Cu/Cu solder joint (figure 7(d)). EDS analysis confirmed that the interface between Sn–57.9Bi–0.1P alloy and IML was mainly β-Sn phase.

4. Discussion

4.1. Improvement of elongation
The elongation of Sn–58Bi alloy was improved by adding appropriate amount of Cu and P elements according to mentioned results in figure 3. The fracture morphology of Sn–58Bi, Sn–57.5Bi–0.5Cu and Sn–57.99Bi–0.01P alloys at room temperature were analyzed in figure 8. Sn–58Bi alloy has large Bi-rich phase showing a mixed fracture mode of cleavage fracture and intergranular fracture with poor plasticity in figure 8(a). The fracture surfaces of Sn–57.5Bi–0.5Cu and Sn–57.99Bi–0.01P alloys were shown in figures 8(b) and (c). The result showed that Sn–57.5Bi–0.5Cu alloy exhibits a ductile fracture mode, and small dimples can be observed on the fracture surfaces. However, the fracture surface of Sn–57.99Bi–0.01P alloy was flat and performed some cleavage features. A better ductility compared with Sn–58Bi alloy, it is consistent with the research results of elongation. In addition, it is worth noting in figure 8(b), Cu₅Sn₃ IMCs seem to be inducement of the occurrence of premature cracks in Sn–57.5Bi–0.5Cu alloy.

The hard and brittle Bi-rich phase has not participated in deformation due to the quite different plasticity of Sn and Bi elements. The deformation of Sn–Bi based alloy was mainly contributed to Sn phase during processing. Therefore, the large Bi-rich phase will cause a sudden fracture resulting in poor plasticity with the cracks initiation and propagation. The reason for the better ductility of Sn–57.5Bi–0.5Cu and Sn–57.99Bi–0.01P alloys is the increased nucleation sites and refined eutectic structure with the addition of Cu and P elements. Moreover, Lai et al [26] investigated the fracture mechanism of Sn–25Bi and Sn–35Bi alloys. The results showed that the crack initiation and propagation of Sn–25Bi and Sn–35Bi alloys mainly occurred in brittle eutectic.
structure. Therefore, refining eutectic structure is considered to be an effective method to improve elongation in Sn–Bi alloy.

Chen et al [27] studied the deformation mechanism combined with in situ observation and nanoindentation. The result showed that the deformation might happen between Sn and Bi phases. The elongation at break of alloy might be affected by phase interface. Here the right amount of Cu6Sn5 phase provided more nucleation sites and introduced more interfaces. Therefore, it may be considered that better mechanical properties of Sn–57.5Bi–0.5Cu alloy was attributed to the refined grains. However, more Cu6Sn5 phases can promote the crack initiation, increase the brittleness and decrease the elongation of Sn–57.5Bi–1.0Cu alloy.

4.2. Improvement of wettability
Bi might exhibit significant improvement in the formation of IML in two aspects. One is that Bi might enrich on the surface of the solution in the molten state, reduce the surface tension and improve the wettability of the alloy [28]. The other is to hinder that Cu reacted with Sn to form Cu6Sn5 IMC to retain the IML with appropriate thickness [29]. EDS line scan confirmed that the interface between Sn–57.5Bi–1.0Cu alloy and IML was surrounded by Bi-rich phase, which can hinder the reaction between Sn element and Cu matrix. In addition, IML thickened in welding process. According to Shen et al [14], the wettability was improved with Cu addition. It might be due to the addition of Cu element promoted the local Cu concentration at the solder/Cu interface and accelerated the reaction of Cu–Sn IML. Nevertheless, the interface between Sn–57.9Bi–0.1P alloy and IML is mainly β–Sn phase, which has strong affinity with Cu element. Moreover, the addition of P element in Sn–58Bi alloy can improve its anti-oxidation performance [30]. Based on these characteristics, the wettability of Sn–57.9Bi–0.1P alloy was effectively improved.

5. Conclusion
The addition of Cu and P elements can improve the elongation at break and wettability of Sn–58Bi alloy. Among them, Sn–57.5Bi–0.5Cu and Sn–57.99Bi–0.01P alloys exhibit a better ductility. The reason for the better ductility of Sn–57.5Bi–0.5Cu and Sn–57.99Bi–0.01P alloys is the increased nucleation sites and refined eutectic structure with the addition of Cu and P elements. The fracture morphology showed that Sn–57.5Bi–0.5Cu alloy exhibited ductile fracture features, and small dimples can be observed on the fracture surfaces. Moreover, Sn–57.5Bi–0.5Cu alloy has better mechanical properties compared with Sn–57.99Bi–0.01P alloy. The wettability was improved slightly with Cu and P addition. In welding process, the solder melted into a liquid state and spreads on the surface of Cu substrate to form IML. The IML was mainly composed of Cu and Sn elements. Bi and P had not participated in the interfacial reactions. The interface between Sn–57.5Bi–1.0Cu alloy and IML was surrounded by Bi-rich phase. Nevertheless, the interface between Sn–57.9Bi–0.1P alloy and IML was mainly β–Sn phase.

The nucleation sites in Sn–58Bi alloy increase with Cu and P addition, and more Cu6Sn5 and P3Sn4 phases was detected in Sn–57.5Bi–1.0Cu and Sn–57.9Bi–0.1P alloys. It can hinder the grain boundary migration and inhibit the grain growth.

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