

The microstructure and mechanical properties of Zn-25Sn-XAl (X=0–0.09 wt%) high temperature lead free solder

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ARTICLE INFO

Article history:

Received 27 July 2016

Received in revised form

13 September 2016

Accepted 15 September 2016

Available online 16 September 2016

Keywords:

High temperature lead free solder

Mechanical properties

Microstructure

ABSTRACT

The microstructure and mechanical properties of Zn-25Sn-XAl (X=0, 0.01, 0.03, 0.05, 0.09 wt%) high temperature Pb-free solders were investigated. The addition of Al tends to refine the grain size and depress the undercooling behavior of the solder. The needle-like zinc-rich phase of the eutectic region was coarsened upon Al addition. The increasing addition of Al up to 0.09 wt% enhanced the ultimate tensile strength (UTS) of the alloy from 67.28 to 78.61 MPa (16.84% improved), and the yield strength from 42.52 to 52.81 MPa (24.2% improved). The strain of the solder degraded from 39.02–32.83% when the addition of Al ranged from 0 to 0.09 wt%.

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1. Introduction

While Pb-Sn solders are widely used in the micro-electronics industry, lead is hazardous and harmful to both the environment and human health [1], and laws have thus been passed in many countries to restrict its use [2,3]. As such, there is a clear need for a high temperature lead-free solder [4–6]. Several potential candidates for high temperature applications have been proposed, such as Bi-Ag, Au-Sn and Zn-based alloys [7]. Zn has an appropriate melting range, good thermal and electrical conductivity, and low cost, and alloying with Sn can improve the ductility and wettability of Zn-containing solders [8,9], and thus Zn-Sn system is considered as a potential substitute for solders containing Pb [10–18]. Zn-25Sn alloy has also been investigated for the feasibility of its use [6,19,20]. The main drawback of Zn-containing solder is its easy oxidation, but the minor addition of Al and Pr can improve the oxidation resistance of such materials, with over 0.05 wt% Al addition impedes the oxidation dewetting of Zn-25Sn alloy. The excellent oxidation resistance contributed by the addition of Al can be ascribed to the forming of a compact Al_2O_3 film [6,21].

The addition of Al will also modify the microstructures and mechanical properties of the solder alloys through phase transformation. The addition of a small amount of Nano-Al particles could refine the microstructure and improve the tensile/shear strength and creep property of Sn-Ag-Cu solders [22,23]. The addition of 0.01–0.25 wt% Al coarsens the needle-like Zn-rich phase of the Sn-8.55Zn-1Ag solder alloys [24]. With 0.45 wt% Al addition, the Zn-rich phase becomes rod-like and the diamond-shaped Al-

rich phase appears. The ultimate tensile strength (UTS) increases from 45 to 60 MPa when the Al content is increased up to 0.45 wt%. Due to the dissolution of Al in Sn, the maximum strain of 58% is achieved with 0.25 wt% Al.

The addition of 0.5 wt% Al has been shown to refine the microstructure of Sn-9Zn [25]. The $\text{Al}_6\text{Zn}_3\text{Sn}$ phase distributes uniformly in β -Sn, which increases the tensile strength, while the elongation of this alloy slightly drops. The addition of Al refines the microstructure of Sn-0.7Cu alloy, and also improves the wettability, although the mechanical properties degrade slightly. Besides, the tensile strength and the creep resistance of Sn-0.7Cu-(0.05–0.075)Al are better than that of Sn-0.7Cu-(0.01–0.025)Al due to the formation of the strengthening phase Al_2Cu [26].

The Zn-Al-Sn ternary system has also been investigated [27,28], with the results showing that the Sn that is added does not dissolve in Zn or Al [29]. The α Al phase, Zn-Al structure and Zn-Al-Sn peritectic structure were observed in Zn-6Al-XSn solders [8]. In addition to $\text{Al}_6\text{Zn}_3\text{Sn}$, Al also forms various phases with Zn and Sn in YSn-(Zn-5Al) (Y=10–91 wt%) alloys [30].

Mechanical properties are essential for solder performance. However, the mechanical properties of Zn-25Sn high temperature lead-free solder have rarely been reported in the literature. Based on the effects of Al addition in other alloys, as reviewed above, in this study Al was selected as the alloying element to improve the oxidation resistance and modify the microstructure and mechanical properties of Zn-25Sn alloys. The as-cast microstructures and the fractographies of these were investigated to assess the tensile behaviors of the Zn-25Sn-XAl (X=0–0.09 wt%) solders.

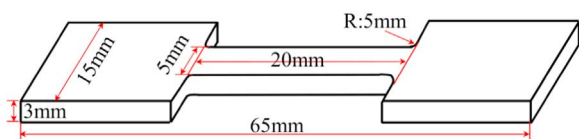
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Table 1

The chemical composition analyzed by ICP-OES (wt%).

Alloys	Zn	Sn	Al
ZS	76.768	23.226	ND
ZS01A	77.103	22.881	0.015
ZS03A	74.741	25.221	0.037
ZS05A	77.938	22.008	0.046
ZS09A	77.041	22.877	0.076

**Fig. 1.** The schematic of sample for tensile test.

2. Materials and methods

The Zn-25Sn-XAl (ZSXA, X=0, 0.01, 0.03, 0.05, 0.09 wt%, the 'wt.' will be omitted for weight percentage in the following) alloys were prepared from Zn (99.99%), Sn (99.99%) and Al (99.999%). The compositions of the alloys prepared were analyzed by ICP-OES, as shown in Table 1.

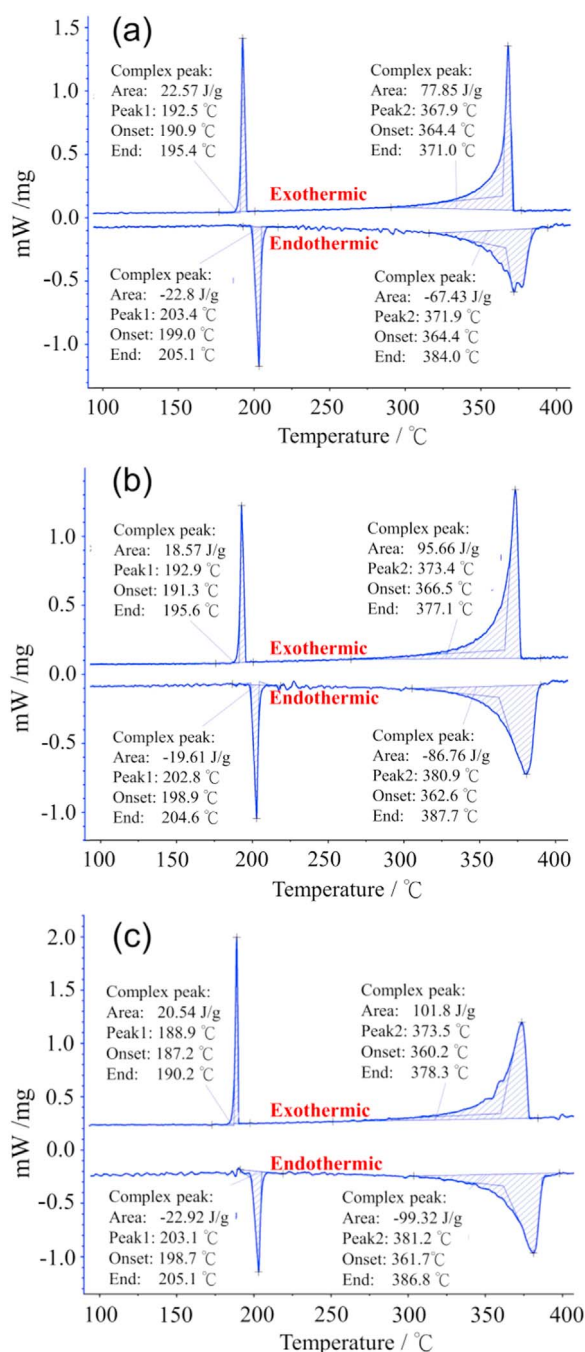
Differential scanning calorimetry (DSC) was conducted to acquire the thermal properties of the alloys from 30 °C to 435 °C. The heating and cooling rates were fixed at 10 K/min under N₂ atmosphere with the flow rate of 60 ml/min.

The samples used for electron backscatter diffraction (EBSD) observation were prepared as follows. The weighed Zn, Sn and Al were sealed in a quartz tube in a high purity Ar atmosphere, heated to 500 °C and held at this temperature for 5 h, being shaken at the second and third hours for homogenization. The alloys were quenched in liquid nitrogen after homogenization. The solidified alloys were machined by electro spark wire-electrode cutting and carefully ground with sandpaper up to 4000 grits and then polished by 0.05 μm Al₂O₃. After that, the specimens for EBSD investigation were polished by colloidal silica suspension. The dimensions of the grains of the Zn-rich phase were estimated by image analysis software. The samples used for SEM microstructure observation were prepared following the same procedure as mentioned above, but cooled in a furnace. For the tensile test, 2 kg of the metals were heated in a crucible and homogenized at 500 °C for 5 h. The specimens were cast as cuboids with the dimensions of 65 × 15 × 100 mm in a metal mold, and then machined by electro spark wire-electrode cutting to the dimensions shown in Fig. 1 for the tensile tests. These tests were conducted at a cross-head speed of 1 mm/min according to the ASTM-A370 standard. The initial strain rate was about 8.33×10^{-4} (s⁻¹). The fracture morphology was observed by SEM.

3. Results

The compositions of the prepared solders were analyzed by ICP-OES, with the results shown in Table 1. The Zn contents of the prepared solders ranged from 74–78%, while the Sn contents were between 22% and 26%, and the Al contents were between 0.015% and 0.076%.

Thermal analysis was performed for the solders ZS, ZS01A and ZS09A. Fig. 2 shows the DSC curves of the various alloys. The onset, end and peak temperatures during cooling and heating cycles are tabulated in Table 2. It can be seen that the peak 1 temperature of the three alloys fluctuates slightly for both heating and cooling processes. This fluctuation may be ascribed to the experimental

**Fig. 2.** The differential scanning calorimetry (DSC) of: a) ZS; b) ZS01A; c) ZS09A.

deviations among tests. For example, the temperatures of ZS, ZS01A and ZS09A on the onset point of peak 1 in heating are 199, 198.9 and 198.7 °C, respectively. The results deviate slightly from, but are close to, the reported eutectic temperature of 198 °C. The values in the literature also show slight deviations from each other [28,31].

The endothermic areas of peak 2 of ZS, ZS01A and ZS09A during heating are 67.43, 86.76 and 99.32 J/g, respectively. The increase in the endothermic area is attributed to the addition of Al, which has a melting temperature of 660.4 °C. This increasing trend also occurs to the areas of exothermic peak 2. The eutectic reaction at about 278 °C reported for the Sn-Zn-Al system [8,28] was not observed in this study. The absence of this eutectic reaction is attributed to the minor addition of Al. However, the eutectic reaction of the Sn-Zn-Al ternary system is reflected by peak 1.

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