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Phase transformation and morphology of the intermetallic compounds formed at the Sn–9Zn–3.5Ag/Cu interface in aging

Min-Hsiung Hon [a], Tao-Chih Chang [a,b], Moo-Chin Wang [c,*]

[a] *Department of Materials Science and Engineering, National Cheng Kung University, 1 Ta-Hsueh Road, Tainan 70101, Taiwan*
[b] *Electronic & Optoelectronics Research Laboratories, Industrial Technology Research Institute, Bldg. 11, 195, Sec. 4, Chung-Hsing Road, Chutung, Hsinchu, 310, Taiwan*
[c] *Faculty of Fragrance and Cosmetics, Kaohsiung Medical University,100 Shi-Chuan 1st Road, Kaohsiung 807, Taiwan*

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Abstract

The morphology and phase transformation of the intermetallic compounds (IMCs) formed at the Sn–9Zn–3.5Ag/Cu interface in a solid-state reaction have been investigated by X-ray diffraction (XRD), transmission electron microscopy (TEM), electron diffraction (ED), scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS). The monoclinic η'-Cu_6Sn_5 transforms to the hexagonal η-Cu_6Sn_5 and the orthorhombic Cu_5Zn_8 transforms to the body-centered cubic (bcc) γ-Cu_5Zn_8 as aged at 180 °C. The scallop-shaped Cu_6Sn_5 layer is retained after aging at 180 °C for 1000 h. In the solid-state reaction, Ag is repelled from η'-Cu_6Sn_5 and reacts with Sn to form Ag_3Sn, and the Cu_5Zn_8 layer decomposes. Kirkendall voids are not observed at the Sn–9Zn–3.5Ag/Cu interface even after aging at 180 °C for 1000 h.
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Keywords: Phase transformation; Intermetallic compound; Sn–9Zn–3.5Ag/Cu; Monoclinic; Hexagonal; Kirkendall voids

1. Introduction

To reduce the pollution caused by Pb, lead-free solders have been utilized to replace gradually the 63Sn–37Pb solder alloy in electronic industry. The morphology, distribution and thickness of the intermetallic compounds (IMCs) formed at the solder alloy/Cu interface affect the solder joint reliability seriously, as reported by Harris and Chagger [1]. Therefore, many researchers have engaged in investigating the interface reactions between solder alloys and Cu substrate or other under bump metallization (UBM) layers [2–5]. η-Cu_6Sn_5 is the most often observed IMC formed at the Sn-base solder/Cu interface and has a hexagonal structure [6–10].

η-Cu_6Sn_5 is an ordered IMC with the NiAs structure. Westgren and Phrahmen [11] have reported that Cu_6Sn_5 transforms from η to η' at a temperature around 170 °C. η'-Cu_6Sn_5 has a monoclinic structure and is a new superstructure belonging to the NiAs-Ni_2In structure, as reported by Larsson et al. [12] In the previous study [13], a bi-structural Cu_6Sn_5 is found at the Sn–9Zn–*x*Ag/Cu interface after soldering due to the dissolution of Ag and Zn in η-Cu_6Sn_5. Besides, orthorhombic Cu_5Zn_8 replaces bcc γ-Cu_5Zn_8 forming at the interface when Ag addition in solder alloy is 3.5 wt% [13].

However, the Cu–Sn compound formed at the Sn–9Zn–3.5Ag/Cu interface after aging at 180 °C for 1000 h is determined as η-Cu_6Sn_5 [14] and no η'-Cu_6Sn_5 is detected. Therefore, the phase transformation of bi-structural Cu_6Sn_5 is proposed to occur in the solid-state reaction. The scallop-shaped and planar IMC layers are the most stable morphology in the reflow and aging processes, respectively, as reported by Tu et al. [15]. The morphology and thickness of the IMCs affect the solder joint reliability [16]. Besides, Yu et al. [2] have shown that the formation of Kirkendall voids after aging at 150 °C for 600 h decreases the adhesion strength of the Sn–9Zn/Cu and Sn–Zn–Al/Cu interfaces. However, Kirkendall voids are not observed at the Sn–9Zn–*x*Ag/Cu interface even after aging at 150 °C for 750 h [17]. Hence, the addition of Ag in the Sn–9Zn solder alloy is beneficial to preventing the formation of Kirkendall voids.

The morphology and phase transformation of the IMCs formed at the Sn–9Zn–3.5Ag/Cu interface in the solid-state reaction have been investigated. The objectives of this study

* Corresponding author. Tel.: +886 6 2585663; fax: +886 6 2502734.
E-mail address: mcwang@kmu.edu.tw (M.-C. Wang).

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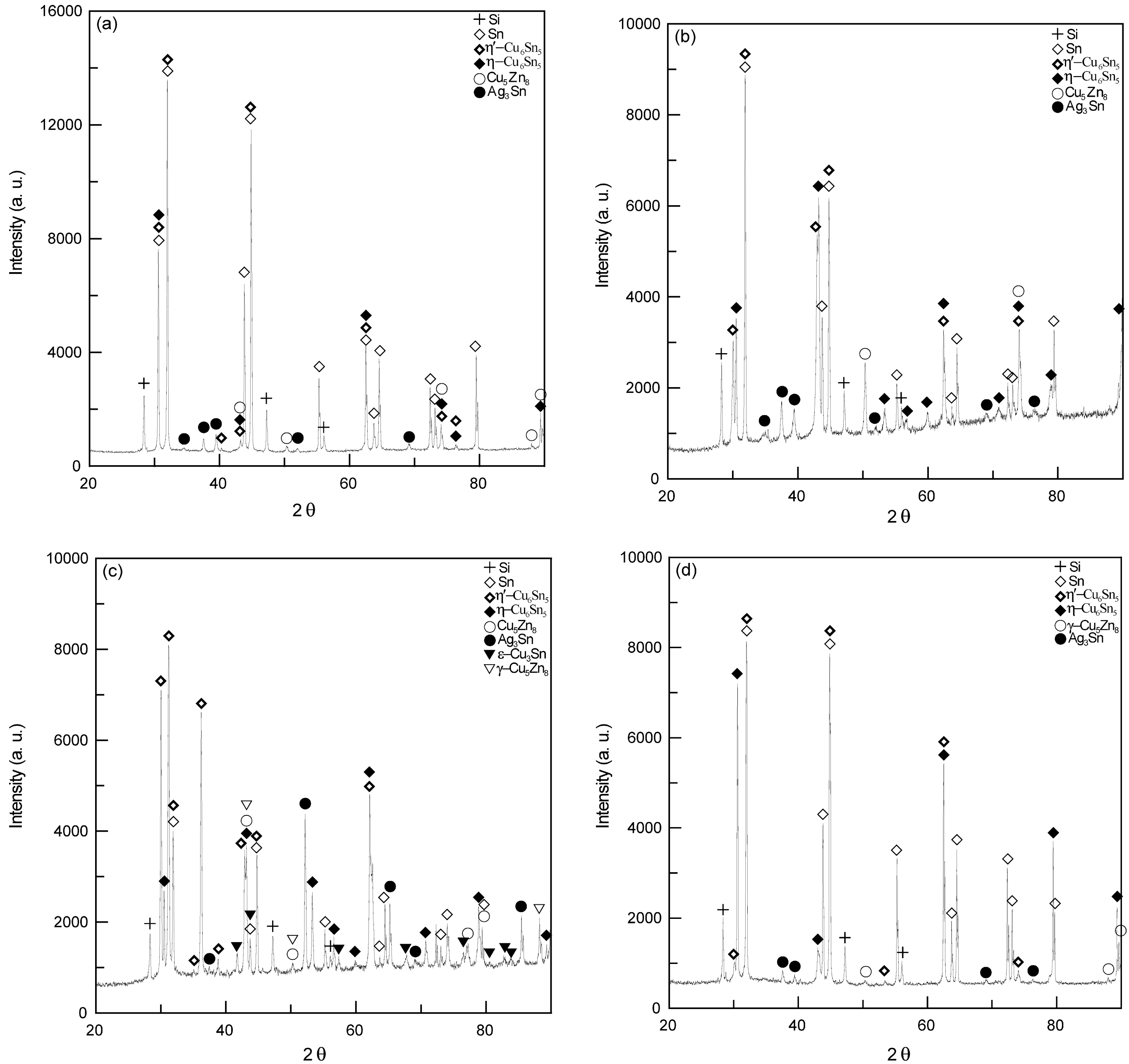

Fig. 1. XRD pattern of the Sn–9Zn–3.5Ag/Cu interface after soldering at 350 °C for 30 s and aged at 180 °C for (a) 0 h, (b) 100 h, (c) 250 h and (d) 400 h.

are: (1) to observe the phase transformation and morphology of the IMCs formed at the Sn–9Zn–3.5Ag/Cu interface in the solid-state reaction, (2) to prevent the formation of Kirkendall voids at the solder alloy/Cu interface as aged at 180 °C with Ag addition and (3) to estimate the adhesion strength of the Sn–9Zn–3.5Ag/Cu interface after aging at 180 °C for various times.

2. Experimental procedure

The Sn–9Zn–3.5Ag solder alloys used in this study were melted with pure metals of Sn, Zn and Ag (purity of 99.9%). They were degreased and deoxidized with 5 vol% HCl and 5 wt% NaOH solutions, respectively. After each step, the metals were rinsed with deionized water, then melted at 600 °C in a stainless steel crucible and stirred to homogenize.

The substrate was an oxygen-free, high conductivity (OFHC) Cu plate and cleaned by the same procedure as mentioned above for pure metals. After cleaning, the Cu substrate was fluxed in a 3.5 wt% DMAHCl solution (3.5 wt% dimethylammonium chloride and ethanol as a solvent) for 10 s and hot-dipped in the molten solder alloy at 350 °C for 10, 20 and 30 s, respectively, to obtain a flat surface, then cooled in air. Afterward, the samples hot-dipped for 30 s were aged at 180 °C for 100, 250, 400, 750 and 1000 h, respectively.

The unreacted solder alloy was removed with sandpapers and an etching solution (40 ml C_2H_5OH + 40 ml HCl + 10 ml CrO_3 + 10 ml $FeCl_3 \cdot 6H_2O$). XRD (D-MAXIIIB, Rigaku, Japan) was used to determine the structure of intermetallic compounds (IMCs) formed at the Sn–9Zn–3.5Ag/Cu interface at a scanning rate $1^\circ\ min^{-1}$ for 2θ from 20 to 90°.

The treated samples were cross-sectioned and mounted in an epoxy, then ground and polished with sandpapers and 0.3 μm Al_2O_3 powders, respectively. The polished samples were etched with an etchant (2 vol% HCl + 3 vol% HNO_3 + 95 vol% C_2H_5OH) for 10 s. SEM (JXA-840, JEOL, Japan) was utilized to observe the Sn–9Zn–3.5Ag/Cu interface and EDS (AN10000/85S,

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