



Interfacial reactions of eutectic Sn3.5Ag and pure tin solders with Cu substrates during liquid-state soldering

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ARTICLE INFO

Article history:

Received 5 November 2011

Received in revised form

25 December 2011

Accepted 28 February 2012

Available online 22 March 2012

Keywords:

A. Intermetallics, miscellaneous

B. Texture

C. Crystal growth

D. Joining

D. Microstructure

ABSTRACT

The growth behaviors of the intermetallic compounds (IMCs) formed at the eutectic Sn3.5Ag/polycrystalline Cu and pure Sn/polycrystalline Cu interfaces are comparatively studied based on an experiment in which the liquid solder is removed before the end of soldering. This removal of the solder allows for the capture and visualization of the interfacial IMCs formed during liquid-state soldering and avoids the influence of Cu₆Sn₅ precipitated from the solder matrix during cooling. The results show that round, scallop-type Cu₆Sn₅ grains with a strong texture form at the molten solder/Cu interface and that their growth is controlled more by grain boundary (GB) diffusion at the beginning of the reaction followed by volume diffusion, whereas the growth of Cu₃Sn is only volume-diffusion-controlled. In addition, in contrast to the predictions of some studies, Ag does not inhibit interfacial IMC growth. Instead, by changing the interfacial energy between the molten solder and the interfacial IMC, the addition of Ag affects the growth orientation and coarsening behavior of interfacial Cu₆Sn₅ grains. These changes lead to more Cu₆Sn₅ GBs at the interface and therefore greater IMC formation and Cu consumption in the Sn3.5Ag/Cu reaction than in the Sn/Cu reaction under the same reflow conditions.

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1. Introduction

Soldering is a key process in the industrial manufacture of electronic devices [1]. Recently, because of environmental and health concerns, efforts to develop and promote lead-free soldering have significantly increased [1–12]. Eutectic Sn3.5Ag has been recommended as a replacement for SnPb solders in the chip-joint process for the manufacture of low-cost, large-volume consumer products [2,3]. To further improve the properties of solder joints, a wide range of quaternary additions, such as rare earths, Bi, In, Cu, Ni, and ZrO₂, are currently being alloyed with SnAg solders [4–12]. In these SnAg-based solders, Sn is the main element and usually reacts with Cu substrates to form two interposing layers of Cu–Sn intermetallic compounds (IMCs), e.g., Cu₃Sn and Cu₆Sn₅. The Cu₃Sn is formed next to the Cu, whereas Cu₆Sn₅ is formed immediately adjacent to the solder [3–11]. Understanding the growth behavior of IMCs is very important because IMCs are linked to several

reliability issues [4]. Today, because an increasing number of additives are being used, an empirical approach for finding a suitable lead-free solder for industrial needs is becoming infeasible. To better develop lead-free solders, investigations of the basic binary and ternary reaction systems, i.e., Sn/Cu and SnAg/Cu systems, are essential.

In the literature, many studies on the interfacial reactions of Sn3.5Ag and Sn solders with Cu interfaces during soldering have been published [13–19]. However, little information is available about the growth orientations of the interfacial Cu₆Sn₅ grains formed in the two reactions; moreover, the experimental methods that have been used in most studies are not particularly appropriate. Recently, some findings have indicated that the Cu₆Sn₅ layer formed at the interface shows strong texture, which is greatly affected by solder compositions [11,13], reaction temperatures [14], and substrate orientations [20]. Due to different electronic mobilities and atomic diffusivities along various crystal orientations, their growth orientations may have a significant influence on the electrical and mechanical characteristics of solder joints. In addition, because of the anisotropy of Cu₆Sn₅ crystals, the growth orientations of Cu₆Sn₅ grains at the interface may affect their morphology, coarsening behavior, and growth kinetics. Therefore, the growth orientations of the interfacial Cu₆Sn₅ grains formed in the two reactions are worthy of investigation.

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In addition, although the morphologies, coarsening behaviors, and growth kinetics of interfacial IMCs have been extensively studied [1–27], almost all of the studies published to date are based on samples cooled either in ambient air or in water. It should be pointed out that the formation of a soldered joint occurs in two stages: isothermal soldering and subsequent cooling. During cooling, a large amount of Cu_6Sn_5 will rapidly precipitate on the existing grains at the interface and significantly affect the morphology, size, and thickness of the Cu_6Sn_5 IMC formed during soldering [15]. Consequently, the studies based on such samples can hardly reveal the actual morphological evolution, coarsening behaviors, and growth kinetics of the interfacial IMCs formed during liquid-state soldering.

Thus, the aim of this study was to obtain information on interfacial reactants at an arbitrary moment during the liquid/solid reaction to monitor the evolution of interfacial IMCs formed at molten Sn3.5Ag solder/Cu (polycrystalline Cu unless otherwise specified) and molten Sn/Cu interfaces. Based on these results, the growth behaviors (e.g., morphological evolution, growth orientations, coarsening behaviors, and growth kinetics) of the IMCs during liquid-state soldering are discussed, and the effects of these behaviors on the extent of interfacial IMC growth and substrate consumption are evaluated.

2. Experimental procedures

2.1. Preparation of samples

For the sample preparation, 150 ± 5 mg of eutectic Sn3.5Ag solder beads or pure Sn alloy (99.95 wt.%) were placed on polycrystalline Cu foil ($5 \text{ mm} \times 5 \text{ mm} \times 0.3 \text{ mm}$) and then reflowed using hot air at peak temperatures of $240 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ or $280 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$, respectively, for durations of 2 min–600 min. The long durations of reflow were chosen to more clearly resolve the kinetic trends. To minimize the influence of the Cu_6Sn_5 precipitated from the solder matrix during cooling on the interfacial IMCs formed during liquid-state soldering, the molten solder was blown off the sample using a high-pressure air gun immediately before the end of soldering (Fig. 1). Two samples were prepared to study each parameter: one was mounted in epoxy and polished to reveal the cross-sectional microstructure; the other was immersed in 10 vol.% HNO_3 solution with ultrasonic waves to dissolve away the excess solder and expose the three-dimensional IMC morphology.

2.2. Identification and measurement of IMCs

Scanning electron microscopy (SEM), together with energy-dispersive X-ray spectroscopy (EDX), was employed to

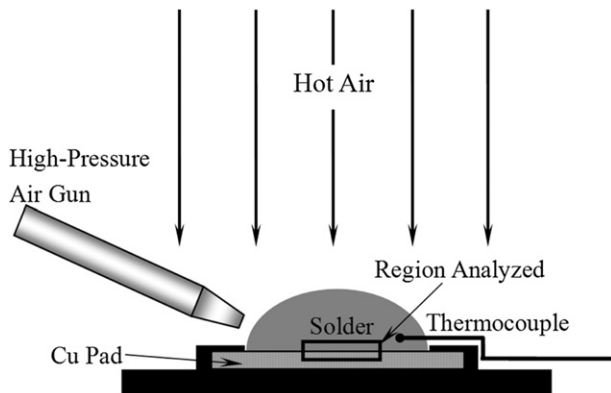


Fig. 1. Schematic illustration of reflow process.

characterize the IMC microstructure. For kinetic studies, the IMC thicknesses were measured from the SEM images of the metallographic cross-sections using the following image analysis procedure: (1) an SEM image of each sample was obtained at the appropriate magnification (Fig. 2a); (2) the grayscale SEM image was enhanced using Adobe Photoshop to clearly identify the interfaces between the different layers (Fig. 2b); (3) the mean thickness (H_{IMC}) of individual layers was calculated using the following equation:

$$H_{\text{IMC}} = H_{\text{SEM}} \times N_{\text{IMC}}/N_{\text{SEM}}, \quad (1)$$

where H_{SEM} is the actual height of the SEM image, and N_{IMC} and N_{SEM} are the number of pixels in the IMC layers and the entire image, respectively. To make the data of the IMC layer thickness more statistically reliable, dozens of SEM images that covered numerous grains in the middle of the interface (Fig. 1) were analyzed for each sample.

As presented below, the IMCs formed in the Sn3.5Ag/Cu and Sn/Cu systems include η -phase (Cu_6Sn_5) that grew at the solder side and ϵ -phase (Cu_3Sn) that grew at the Cu side. To study the coarsening behaviors of the interfacial Cu_6Sn_5 grains, the top view of the interfacial grains was first recorded by SEM as a function of the reaction time and the reaction temperature. The mean equivalent spherical radius and size distributions of the interfacial Cu_6Sn_5 grains were then determined using the program ImageJ; 500–800 grains were measured for each experimental condition.

2.3. Measurement of Cu_6Sn_5 growth orientation

Electron backscatter diffraction (EBSD) analyses were conducted to reveal the interfacial Cu_6Sn_5 orientations. To facilitate EBSD characterization, the samples with exposed interfacial Cu_6Sn_5 were mounted, and the tips of interfacial Cu_6Sn_5 grains were carefully ground and polished to faceted surfaces [14]. Because the Cu_6Sn_5 layer was quickly cooled after soldering, η - Cu_6Sn_5 with a hexagonal structure was used in our EBSD analyses [11,13,14,21]. Orientation data were collected from the selected area using an SEM equipped with a fully automatic EBSD analysis system; pole figures were then obtained using the program Orientation Imaging Microscopy (OIM). To obtain more statistically reliable orientation distributions, many grains that covered an area of at least $500 \mu\text{m} \times 500 \mu\text{m}$ were analyzed.

3. Results and discussion

3.1. Morphology formation of interfacial Cu_6Sn_5

Fig. 3 shows the Cu_6Sn_5 grains formed at the Sn3.5Ag/Cu and Sn/Cu interfaces during liquid-state soldering. Almost all of the interfacial Cu_6Sn_5 grains exhibited round surfaces for all of the experimental conditions used in this study. The shapes of these round grains formed at various reaction conditions were not identical, which can be observed more clearly from the cross-sectional view. The Cu_6Sn_5 grains formed at the Sn3.5Ag/Cu interface were higher but thinner than those formed at the Sn/Cu interface under the same reflow conditions. In addition, the shape of the Cu_6Sn_5 grains formed at the Sn3.5Ag/Cu interface at $240 \text{ }^\circ\text{C}$ was more uniform than that formed under the other three sets of conditions.

Because almost all of the solder was removed prior to cooling (Fig. 3), the interfacial Cu_6Sn_5 grains in this study should have been minimally affected by the Cu_6Sn_5 precipitated from the solder matrix during cooling. The Cu_6Sn_5 grains can therefore be observed as the interfacial grains formed during liquid-state soldering, which can be regarded as an equilibrium state. Therefore, the classical

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