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# Texture evolution and its effects on growth of intermetallic compounds formed at eutectic Sn37Pb/Cu interface during solid-state aging

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#### ABSTRACT

The growth orientation of  $Cu_6Sn_5$  intermetallic compounds (IMCs) formed at the eutectic Sn37Pb/ polycrystalline Cu interface during solid-state aging was investigated. The results indicate that the interfacial  $Cu_6Sn_5$  grains exhibit textured growth under solid-state conditions, and their preferred orientations are affected by the initial joint preparation conditions.  $Cu_6Sn_5$  grains in the [0001] direction normal to the interface are stable in solid and molten Sn37Pb solder at 200 °C, but are rapidly consumed at 280 °C. This effect leads to the formation of different textures in the  $Cu_6Sn_5$  layer during the solid-state aging treatment of joints formed at 200 and 280 °C. In addition, the influence of texture evolution on the growth of interfacial IMCs was evaluated. The results indicate that Sn diffusion is faster along the [0001] direction of the  $Cu_6Sn_5$  crystal than along an angle of 25–45° to the [0001] direction; therefore, more IMCs are generated at the interface of the joints formed at 200 °C than at those formed at 280 °C under the same solid-state reaction conditions.

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#### 1. Introduction

Due to environmental and health concerns, the European Union enacted legislation on July 1, 2006 banning the use of lead (Pb) and several other substances in electronic products [1,2]. However, certain electronic products - including servers, storage and storage array systems, network infrastructure equipment and telecommunication equipment for network management - that are referred to as high-performance electronic products are so essential to modern society that their operational integrity must be maintained. For these products, the introduction of new and unproven materials could pose a significant reliability risk [1–4]. Accordingly, the European Commission (EC) has granted an exemption permitting the continued use of Pb in solders for highperformance equipment [1,2]. In such applications, the SnPb solder usually reacts with Cu to form two interposing layers of Cu-Sn intermetallic compounds (IMCs), e.g., Cu<sub>3</sub>Sn and Cu<sub>6</sub>Sn<sub>5</sub>, at the interface [2-9]. These layers tend to grow via solid-state diffusion even at ambient temperatures. Due to excessive growth during storage and service, these IMC layers may adversely affect the reliability of the solder joints. Specifically, the brittle nature of the IMCs can mechanically weaken the solder joints. Therefore, an understanding of the growth behavior of the IMCs formed at the SnPb/Cu interface during solid-state aging is necessary. In addition, because SnPb solders have been used on Cu for several decades and many studies have been performed on the ternary system of SnPb—Cu, an understanding of the solder reactions of SnPb on Cu can serve as a reference for the development of Pb-free solders [2—4].

To date, numerous studies have been reported on solid-state IMC growth [10–21]. However, nearly all studies regarding interfacial IMCs focus on the crystal structure [10–14], growth kinetics [15–22], and their effects on mechanical properties [20–22]. Little information is available regarding the growth orientations. Due to differences in electronic mobility and atomic diffusivity along the various crystal orientations, the growth orientations may have a significant influence on the electrical and mechanical properties of solder joints. In addition, because of the anisotropy of  $\text{Cu}_6\text{Sn}_5$  crystals, the growth orientations of the  $\text{Cu}_6\text{Sn}_5$  grains at the interface may affect their coarsening behavior and growth kinetics.

Therefore, this work attempts to understand the growth orientations of the  $\text{Cu}_6\text{Sn}_5$  grains formed at the eutectic  $\text{Sn}_5$ 7Pb/Cu (polycrystalline Cu, unless otherwise specified) interface during solid-state aging. In addition, the effects of the growth orientation of the interfacial  $\text{Cu}_6\text{Sn}_5$  grains on the coarsening behavior and growth kinetics of the interfacial IMCs are evaluated.

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#### 2. Experimental

#### 2.1. Preparation of samples

Square  $5 \times 5 \times 0.3$ -mm<sup>3</sup> specimens were cut from a commercial-grade polycrystalline, oxygen-free Cu plate (C1020P) with an average grain size of approximately 16 um. The specimens were then ground with SiC paper, polished with 1-um Al<sub>2</sub>O<sub>3</sub> powders. and etched with a 10 vol% HCl/alcohol solution in an ultrasonic bath. To study the interfacial reactions,  $150 \pm 5$  mg of eutectic Sn37Pb beads were placed on the polished surface of a Cu substrate in the presence of commercial rosin mildly activated (RMA) flux, then reflowed using hot air station (Pace, ST325) at the peak temperatures of 200 or 280 °C for 2 min. The temperature was measured using a thermocouple to the accuracy of  $\pm 2$  °C. The samples were cooled in air after soldering, placed in an oven (Binder, FD23), maintained at a constant temperature of 150  $\pm$  1  $^{\circ}$ C and aged for 1 day to 81 days. Two samples were prepared to study each parameter: One was mounted in epoxy, ground with SiC paper, and polished with 1-µm and 0.05-µm Al<sub>2</sub>O<sub>3</sub> powders to reveal the cross-sectional microstructure. The other was immersed in 10 vol% aqueous HNO3 and placed in an ultrasonic bath to dissolve the excess solder and expose the IMC film. The IMC grain morphology was examined in top view and then the same sample was analyzed using X-ray diffraction (XRD, Rigaku D/max-2500PC with Cu  $K\alpha$  radiation) and electron backscattering diffraction (EBSD, EDAX-TSL Hikari).

#### 2.2. Identification and measurement of IMCs

Scanning electron microscopy (SEM, Hitachi S-4700) and energy-dispersive X-ray spectroscopy (EDX, EDAX Genesis XM2 60) were employed to characterize the IMC microstructure. As presented below, the IMCs formed in the Sn37Pb/Cu system include  $\eta$ -phase (Cu<sub>6</sub>Sn<sub>5</sub>) that grew at the solder side and  $\varepsilon$ -phase (Cu<sub>3</sub>Sn) that grew at the Cu side.

For kinetic studies, the IMC thickness was measured using the SEM images of the metallographic cross-sections and the following image analysis procedure. (1) An SEM image of each sample was obtained at the appropriate magnification. (2) The grayscale SEM image was enhanced using Adobe Photoshop to clearly identify the interfaces between the different layers. (3) The mean thickness  $(H_{\rm IMC})$  of the individual layers was calculated using the following equation:

$$H_{\rm IMC} = H_{\rm SEM} \times N_{\rm IMC}/N_{\rm SEM} \tag{1}$$

in which  $H_{\rm SEM}$  is the actual height of the SEM image, and  $N_{\rm IMC}$  and  $N_{\rm SEM}$  are the number of pixels in the IMC layers and the entire image, respectively. To improve the statistical reliability of the IMC layer-thickness data, dozens of SEM images covering numerous grains in the middle of the interface were analyzed for each sample.

To study the coarsening behavior of the interfacial  $\text{Cu}_6\text{Sn}_5$  grains, their top view was monitored via SEM as a function of reaction time. The mean equivalent spherical radius and size distributions of the interfacial  $\text{Cu}_6\text{Sn}_5$  grains were then determined using the ImageJ program; 500-800 grains were measured for each experimental condition.

#### 2.3. Measurement of the Cu<sub>6</sub>Sn<sub>5</sub> growth orientation

XRD and EBSD analyses were conducted to determine the interfacial Cu<sub>6</sub>Sn<sub>5</sub> orientations. According to the Cu-Sn phase diagram, the  $\eta$ -Cu<sub>6</sub>Sn<sub>5</sub> phase is stable above 186 °C and primarily exhibits a B8-type structure (P63/mmc with the prototype NiAs-Ni<sub>2</sub>In) [10–12], then rearranges to monoclinic structures (C2 and  $P2_1/c$ ) [13]. The  $\eta'$ -Cu<sub>6</sub>Sn<sub>5</sub> phase is stable below 186 °C and exhibits a monoclinic structure (C2/c). Tu et al. [2] and Laurila et al. [4] reported that the  $\eta$ -Cu<sub>6</sub>Sn<sub>5</sub> phase could be maintained at room temperature in the quenched specimens due to insufficient time for the solid-state transformation of the  $\eta$  to  $\eta'$  phase. Recently, Li et al. [14] found that this transformation is affected by the release of volumetric strain energy. In the actual solid-state aging process of solder joints, these four structures co-exist for a long time because the release of the volumetric strain energy, which allows for the transformation from  $\eta$  to  $\eta'$ , is difficult. In addition, the four Cu<sub>6</sub>Sn<sub>5</sub> structures are similar and are all based on the hexagonal NiAs structure; thus, they exhibit similar XRD and Kikuchi patterns. Therefore, it is difficult to determine how much of the  $\eta$  phase has transformed into the  $\eta'$  phase. Because this study focuses on the orientational evolution of the interfacial Cu<sub>6</sub>Sn<sub>5</sub> grains during solid-state aging, only the n-Cu<sub>6</sub>Sn<sub>5</sub> with a hexagonal structure was analyzed via XRD and EBSD.

To facilitate the EBSD characterization, the samples with exposed interfacial  $Cu_6Sn_5$  were mounted, and the tips of the interfacial  $Cu_6Sn_5$  grains were carefully ground and polished to obtain faceted surfaces [23,24]. Orientation imaging microscopy (OIM) data were collected using TSL OIM Data Collection 5.2

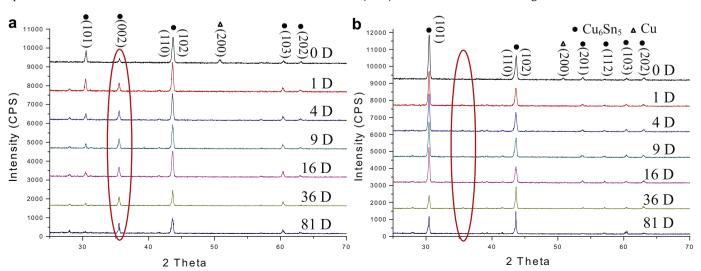


Fig. 1. XRD patterns of Cu<sub>6</sub>Sn<sub>5</sub> grains formed at the Sn37Pb/Cu interface at 150 °C for different aging times. The initial joints formed at (a) 200 °C and (b) 280 °C for 2 min.

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