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# The nucleation-controlled intermetallic grain refinement of Cu–Sn solid-liquid interdiffusion wafer bonding joints induced by addition of Ni particles

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## ABSTRACT

In this study, Ni particle was validated as an effective grain refiner for the Cu–Sn solid-liquid interdiffusion (SLID) wafer bonding joints, which induced a dispersive non-interfacial nucleation of intermetallic grains, effectively preventing the grain mergence phenomenon. With increasing the Ni particle addition, there were significant reductions in both the  $(\text{Cu}, \text{Ni})_6\text{Sn}_5$  grain size and the  $\text{Cu}_3\text{Sn}$  layer thickness, 6 wt% addition of Ni particles even led a formation of the intermetallic joints only consisted of  $(\text{Cu}, \text{Ni})_6\text{Sn}_5$  grains with an average grain size of 1.3  $\mu\text{m}$ .  $(\text{Cu}, \text{Ni})_6\text{Sn}_5$  grain refinement could notably enhanced the shear strength of the intermetallic joints.

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In recent years, wafer bonding has been seemed as an important bonding method in the manufacture of micro-electro-mechanical systems (MEMS) because it enables the fabrication of silicon-on-insulator (SOI) substrates and allows the three-dimensional (3D) packaging of micro-components [1]. In general, the wide variety of wafer bonding techniques includes three most commonly used bonding methods: direct bonding, anodic bonding and intermediate-layer bonding [1–3]. Among the intermediate-layer bonding methods, solid-liquid interdiffusion (SLID) bonding, also known as transient-liquid-phase (TLP) bonding, is an especially attractive bonding method for the packaging of the MEMS devices which operate at high temperatures [4,5]. SLID bonding is performed in a structure of sandwiching a low melting-temperature solder interlayer between two base metals or metallizations having high melting temperature, and its bonding principle is based on the continuously formation of intermetallic compounds (IMCs) on the cost of the solder consumption, which eventually results in the intermetallic joints entirely consisted of high melting-temperature IMCs and hence offers better thermal stability than other traditional solder bonding methods. Although Au–Sn and Cu–Sn material systems both have been proven to be the promising candidates for SLID wafer bonding, Cu–Sn material system was the more popular choice not only due to its attractive bonding conditions (low bonding

temperature and high performance results) but also owe to its cost and availability [2,6].

For further integration of the high-density packaging micro-components, the vertical thickness of Cu–Sn SLID wafer bonding joints has been reduced to 10  $\mu\text{m}$  or even less, which is one tenth or less compared with the flip-chip bonding joints having a vertical thickness commonly more than 100  $\mu\text{m}$  [7]. The reduced vertical thickness of Cu–Sn SLID wafer bonding joints has introduced many new challenges, a critical concern is the significant influence of the anisotropy of  $\text{Cu}_6\text{Sn}_5$  grains on the mechanical properties of the entire joint [8–10]. The studies of Liu [11] and Zhang [12] have revealed that, in the Cu–Sn SLID wafer bonding joints with a vertical thickness of 12  $\mu\text{m}$ , the average diameter of the contained  $\text{Cu}_6\text{Sn}_5$  grains could be as large as 10  $\mu\text{m}$ , which means that the entire joint was only consisted of a very limited number of  $\text{Cu}_6\text{Sn}_5$  grains, and the studies of Mu [9,10] demonstrated that the mechanical properties of hexagonal  $\text{Cu}_6\text{Sn}_5$  grains were strongly related to the crystal orientation, as a result, the anisotropy of  $\text{Cu}_6\text{Sn}_5$  grains inevitably led a nonunity of mechanical properties in the formed intermetallic joints and induced unforeseen fluctuations on the reliability of bonded devices. Consequently, there is a necessity to develop an effective method to achieve the intermetallic grain refinement of Cu–Sn SLID wafer bonding joints.

In this study, Ni particle was utilized as an effective grain refiner for the Cu–Sn SLID wafer bonding joints, the influence of Ni particle addition on the nucleating pattern of intermetallic grains during the bonding process was investigated, and then the correlations between

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the amount of Ni particle addition and the magnitude of intermetallic grain refinement as well as the shear strength of formed intermetallic joints were also studied.

A sandwiching structure of the Si/Sn-Ni particles/Si bonding system was utilized in this study. The Si bond components were metallized with 50 nm (Ti)/50 nm (Ni)/8  $\mu$ m (Cu) by evaporation and cut into  $5 \times 5$  mm<sup>2</sup> in area. The commercial pure Ni powder having an average particle size of 2  $\mu$ m was mixed with the pure Sn powder having an average particle size of 10  $\mu$ m at the ratios of 0.5 wt%, 2 wt%, 4 wt% and 6 wt% by the dry milling in a N<sub>2</sub> atmosphere for 30 min. The resulted powder mixture was compacted and uniaxially pressed into the pallet with a thickness of 20  $\mu$ m, which was then cut into  $5 \times 5$  mm<sup>2</sup> in area and utilized as the solder interlayer for SLID wafer bonding process. The mounted samples were then soldered in a vacuum reflow oven at 260 °C with a variety of bonding times from 15 min to 45 min. The formed joint samples were subsequently mounted, ground and polished, the cross-sectional microstructures of the joints were characterized by scanning electron microscope (SEM) and the distribution of Ni, Sn and Cu atoms in the intermetallic joints were detected by electron probe micro-analyzer (EPMA). In order to identify the intermetallic grains which formed at the Ni/Sn and Cu/Sn interfaces, focused ion beam (FIB) milling system was utilized to prepare the foil samples for the characterization of transmission electron microscope (TEM). Electron backscatter diffraction (EBSD) detector was used to examine the grain structure of the formed intermetallic joints, and the lineal intercept model gain sizes were calculated. Micro-shear tester was utilized to measure the average shear strengths of the formed intermetallic joints at room temperature.

Fig. 1(a)–(c) presents the backscattered electron (BSE) images of the cross-sectional microstructures of the joints bonded using the Sn-4 wt% Ni particles solder interlayer at 260 °C with a variety of bonding times from 15 min to 45 min, which clearly indicates that, during the bonding process, the nucleation and growth of the intermetallic grains simultaneously occurred both inside the Sn-Ni particles interlayer and at its interfaces with the Cu metallizations. When the bonding time was 15 min, as shown in Fig. 1(a), the IMCs nucleated around the Ni particles and exhibited an incompact core-halo structure. The phase of the intermetallic grains presented in the bright-field TEM (BF-TEM) image of Fig. 2(a), was identified by selected-area diffraction pattern (SADP) and energy dispersive X-ray spectroscopy (EDS) as shown in Fig. 2(b)–(f). The results of SADP indicate that the lattice structures of the two selected

intermetallic grains A and B both matched well with the hexagonal close-packed (HCP)  $\eta$ -Cu<sub>6</sub>Sn<sub>5</sub> structure (space group:  $P6_3/mmc$ ;  $a = 0.4192$  nm;  $c = 0.5037$  nm), the TEM-EDS shows that the two selected IMC grains, which were adjacent to and distant from the Ni particle, had the compositions of 44.82 at% Cu–10.63 at% Ni–44.55 at% Sn and 52.37 at% Cu–3.15 at% Ni–44.48 at% Sn, respectively. Therefore, the phase of the intermetallic grains nucleated around the Ni particle could be identified to be (Cu, Ni)<sub>6</sub>Sn<sub>5</sub>. This result is consistent with the findings of Nogita et al. [13], demonstrating that the incorporation of Ni into Cu<sub>6</sub>Sn<sub>5</sub> stabilized the  $\eta$ -Cu<sub>6</sub>Sn<sub>5</sub> and prevented its transformation from  $\eta$ - to  $\eta'$ -Cu<sub>6</sub>Sn<sub>5</sub> (monoclinic), even at temperatures below 186 °C. At the interfaces between the Sn solder and Cu metallizations, there exhibited a bilayer structure consisting of two different kinds of IMCs as shown in Figs. 1(a) and 3(a). The phases of the intermetallic grains C and D were identified by SADP and TEM-EDS as shown in Fig. 3(b)–(f). The selected bottom-layer intermetallic grain C had a lattice structure matched well with the orthorhombic  $\varepsilon$ -Cu<sub>3</sub>Sn structure (space group:  $C6_3/mmc$ ;  $a = 0.55298$  nm;  $b = 4.7756$  nm;  $c = 0.4323$  nm) and a composition of 77.28 at% Cu–22.72 at% Sn, which could be identified to be Cu<sub>3</sub>Sn. Whereas, the selected top-layer intermetallic grain D had a lattice structure matched well with the hexagonal close-packed (HCP)  $\eta$ -Cu<sub>6</sub>Sn<sub>5</sub> structure and a composition of 53.45 at% Cu–1.88 at% Ni–44.67 at% Sn, which could be identified to be (Cu, Ni)<sub>6</sub>Sn<sub>5</sub>. After the bonding process of 30 min, as shown in Fig. 1(b), the halos of (Cu, Ni)<sub>6</sub>Sn<sub>5</sub> dispersively expanded and the number of the primary grains dramatically redoubled, leading the (Cu, Ni)<sub>6</sub>Sn<sub>5</sub> grains uniformly dispersed inside the entire solder interlayer, whereas the interfacial layers of (Cu, Ni)<sub>6</sub>Sn<sub>5</sub> and Cu<sub>3</sub>Sn had not underwent an apparent growth. When the bonding time exceeded 45 min, the SEM image and EPMA analysis presented in Fig. 1(c)–(f) show that the Sn solder had been completely consumed out and an intermetallic joint consisted of a majority of (Cu, Ni)<sub>6</sub>Sn<sub>5</sub> and a minority of Cu<sub>3</sub>Sn was formed.

During the bonding process, the reactions at the Sn/Ni and Sn/Cu interfaces would occur simultaneously and interact mutually [14,15]. The dissolved Ni and Cu atoms could quickly diffuse across the molten Sn interlayer and establish solubility gradient between the Ni particles and the Cu metallizations, which would strongly affect the nucleation and growth of intermetallic grains at those interfaces [7,14–18]. In particular, at the Sn/Ni interfaces, the arrived Cu atoms continuously dissolved into the initial Ni<sub>3</sub>Sn<sub>4</sub> grains under the driving force of the entropy increase caused by the mixture of Cu and Ni atoms in the ternary

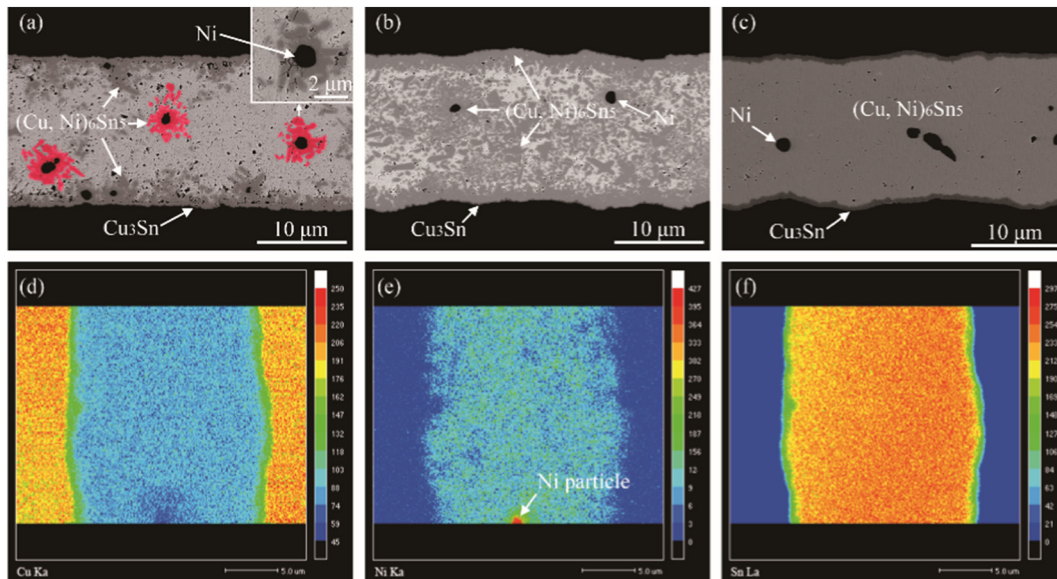


Fig. 1. SEM images of the cross-sectional microstructures of the joints bonded using the Sn-4 wt% Ni particles solder interlayer at 260 °C with a variety of bonding times, (a) 15 min, (b) 30 min and (c) 45 min, and EPMA analysis of element distribution, (d) Cu, (e) Ni and (f) Sn.

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