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Special Issue Paper

Interfacial Reactions Between Pb-Free Solders and In/Ni/Cu Multilayer Substrates

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This study investigates the interfacial reactions between Sn-3.0wt.% Ag-0.5wt.%Cu (SAC) and Sn-0.7wt.%Cu (SC) on In/Ni/Cu multilayer substrates using the solid–liquid interdiffusion bonding technique. Samples were reflowed first at 160°C, 180°C, and 200°C for various periods, and then aged at 100°C for 100 h to 500 h. The scalloped Cu_6Sn_5 phase was formed at the SAC/In/Ni/Cu and SC/In/Ni/Cu interfaces. When the reflowing temperatures were 160°C and 180°C, a ternary Ni-In-Sn intermetallic compound (IMC) was formed when the samples were further aged at 100°C. This ternary Ni-In-Sn IMC could be the binary Ni_3Sn_4 phase with extensive Cu and In solubilities, or the ternary Sn-In-Ni compound with Cu solubility, or even a quaternary compound. As the reflow temperature was increased to 200°C, only one Cu_6Sn_5 phase was formed at the solder/substrate interface with the heat treatment at 100°C for 500 h. Mechanical test results indicated that the formation of the Ni-In-Sn ternary IMC weakened the mechanical strength of the solder joints. Furthermore, the solid–liquid interdiffusion (SLID) technique in this work effectively reduced the reflow temperature.

Key words: Sn-3.0wt.%Ag-0.5wt.%Cu and Sn-0.7wt.%Cu, lead-free solder, In/Ni/Cu multilayer substrates, solid–liquid interdiffusion bonding technique

INTRODUCTION

Sn-Pb alloys are the most commonly adopted solders in the electronics industry. However, Pb harms the environment and human health.[1,2] Hence, the European Union (EU) has passed regulations, such as the Waste Electrical and Electronic Equipment (WEEE) directive and the Restriction of Hazardous Substances (RoHS) directive to ban the sale of Pb-containing electronic products in the EU after July 1st, 2006.[3,4] Sn-3.0wt.%Ag-0.5wt.%Cu (SAC) and Sn-0.7wt.%Cu (SC) alloys are popular lead-free solders used in electronic packaging to join electronic devices to metallic substrates. Both ball grid array (BGA) and flip-chip (FC) techniques are advanced packaging methods and can provide larger input/output (I/O) numbers and good reliability. BGA and FC are frequently used in high-power electronic devices or portable products.[5] In BGA and FC techniques, underbump metallization (UBM) layer structures are very important. The UBM layer enhances the wettability and solderability between the solder and substrate, and prevents substrate oxidation and interdiffusion between the solder and the substrate. An electroplated Ni layer is widely adopted as a diffusion barrier layer material in UBM layer structures.

Numerous efforts have been made to study the interfacial reactions between SAC or SC with Cu or Ni substrates.[6–13] However, the liquidus temperatures of SAC or SC solders are about 30°C higher

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than that of conventional Sn-Pb solder (eutectic temperature of 183°C). Thus, reducing the liquidus temperature of Pb-free solders is a very popular research subject.[6,8,9,11,12] Recently, one common approach for lowering the liquidus temperature of the Pb-free solder has been to add In, Bi or other metals with lower melting points. Huang et al.[8] and Koo et al.[11] studied interfacial reactions of the Sn-In/Ni systems, and both observed the formation of the Ni_3Sn_4 phase at the interface. However, Wang et al.[12] found a planar intermetallic compound (IMC) layer at the Sn-In/Ni interface and asserted that it was the Sn-In-Ni ternary IMC. These Sn-In/Ni interfacial reaction results were inconsistent.

Although adding In to Pb-free solders can effectively reduce the solder liquidus temperature, it raises another problem: because In is an expensive metal, adding In to solders markedly increases the manufacturing cost. A new bonding method has been proposed to increase the range of In applications in the electronics industry. Chen et al.[14] adopted Pb-free composite solders with an In layer and found that this bonding technique reduces not only the cost but also the reflow temperature.

The solid–liquid interdiffusion (SLID) technique is adopted to bond the In/Ni/Cu multilayer structure substrate and two Pb-free solders, Sn-3.0wt.%Ag-0.5 wt.%Cu and Sn-0.7wt.%Cu, in this study. Specimens are reflowed first, then undergo a long period of heat treatment. Furthermore, this study investigates mechanical properties of solder joints in the Pb-free solder/In/Ni/Cu systems.

EXPERIMENTAL PROCEDURES

Preparation of Solder Balls and In/Ni/Cu Multilayer Substrate

The Sn-3.0wt.%Ag-0.5wt.%Cu (SAC) solder ball with a diameter of 760 μm was purchased commercially from Yeh-Chiang Technology Corp. in Taiwan. However, the Sn-0.7wt.%Cu solder ball was prepared by the authors. Sn and Cu metals with high, 99.9 wt.% purity, used in the Sn-0.7wt.%Cu (SC) alloy, were weighed. SC alloys were encapsulated in a quartz tube in a 1.0×10^{-5} Pa vacuum. Then, the samples were placed in a tube furnace at 400°C for 2 h. After the heat treatment, the sample tubes were quenched in ice water and cut down to a mass of about 1.69 mg. The SC alloy was dipped first in rosin mildly activated (RAM) flux (commercial model: Deltalux 523) and then placed in silicon oil at 250°C. Thereby, the SC solder ball with a diameter of 760.0 μm was obtained.

A Cu rod with a diameter of 6.35 mm was cut into a disk with a thickness of 1.00 mm. Then, the Ni layer with a thickness of 5.0 μm and In layer with a thickness of 14.0 μm were deposited on the Cu substrate by an electroplating method to complete the In/Ni/Cu multilayer structure.

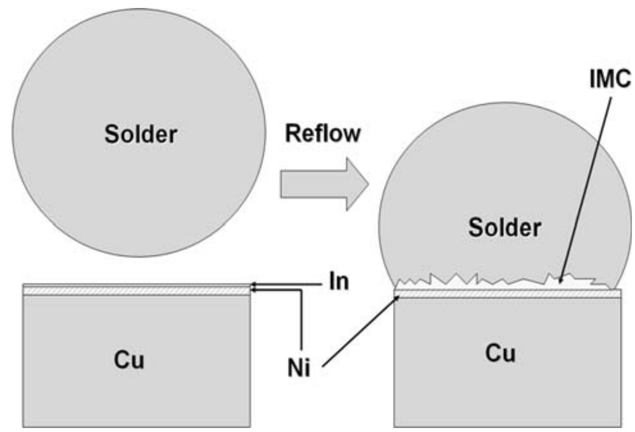

Fig. 1. Schematic diagram of solid–liquid interdiffusion bonding in the solder/In/Ni/Cu system.

Preparation of Interfacial Reactions and Analysis

The SAC and SC solder balls were immersed in RAM flux and then placed on the In/Ni/Cu surface, as presented in Fig. 1. The samples were initially reflowed at 160°C, 180°C, and 200°C for 3 min, 4 min, and 5 min and then aged at 100°C for 100 h to 500 h. Following heat treatment, each sample was metallographically examined. Therefore, the cross-section of the solder/substrate interface was clearly observed. The morphology at the solder/ substrate interface was examined by optical microscopy (OM) and field-emission scanning electron microscope (FE-SEM). The intermetallic compounds (IMCs) formed at the interface were identified by an energy-dispersive spectrometer (SEM/EDS) and an electron probe x-ray microanalyzer (EPMA), which provided their composition. The IMC was identified based on the phase diagram and experimental data.

Testing Mechanical Properties

SAC solder specimens were reflowed at 160°C for 3 min, 4 min, and 5 min and aged at 100°C for 100 h to 500 h, before undergoing mechanical testing using a universal testing machine (Shimadzu AI-GS). The testing parameters were set according to the industry standard-JESD22-B117.[15] The shear rate of the mechanical properties testing was 300.0 μm/s.

RESULTS AND DISCUSSION

Interfacial Reactions in Sn-3.0wt.% Ag-0.5wt.%Cu Solders with In/Ni/Cu Multilayer

Figure 2 shows a backscatter electron image (BEI) of the Sn-3.0wt.%Ag-0.5wt.%Cu solder (SAC) reacting with the In/Ni/Cu substrate at 160°C for 4 min. As shown in Fig. 2, a dark phase spread over the substrate. The EPMA analysis revealed that its composition was Sn-35.4at.%Cu-16.9at.% Ni-2.5at.%In (the compositions in this work are all

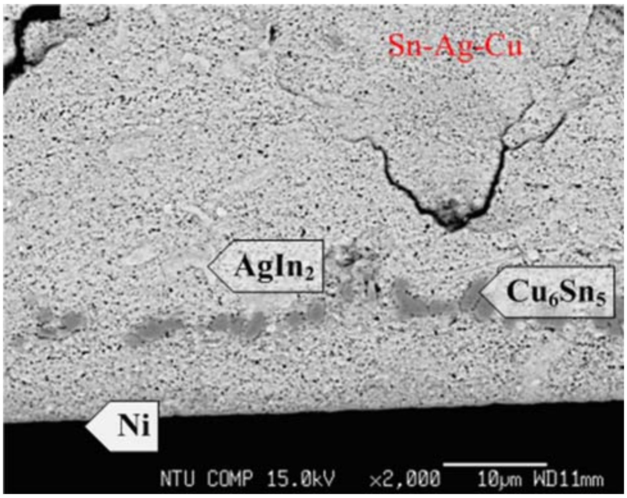

Fig. 2. BEI micrograph of the SAC/In/Ni/Cu system reflowed at 160°C for 4 min.

in at.% hereafter), which corresponds to the Cu_6Sn_5 phase.[16] Bright, irregular, and micro-island-shaped particles of Sn-31.1Ag-2.1Cu-59.5In were found in the solder, corresponding to the $AgIn_2$ phase.[16] The region between the Cu_6Sn_5 phase and the substrate is the $InSn_4$ phase, as determined by EPMA.[16] Meanwhile, the In layer was completely consumed during the reflowing process. When the reflowing time was 3 min at 160°C, or samples were reflowed at 180°C for 3 min, 4 min or 5 min, the results were similar to those for other SAC/In/Ni/Cu specimens.

Following reflowing, all the specimens were heat-treated for a long period. Figure 3 shows a BEI micrograph of the SAC/In/Ni/Cu specimen reflowed at 160°C for 4 min and then aged at 100°C for 300 h. A brighter and planar layer was formed at the Ni side. It is a homogeneous phase and its composition is Sn-9.4Cu-22.3Ni-24.1In. Chen et al.[8,9] and Koo and Jung[11] reported the formation of only the Ni_3Sn_4 phase in the Sn-In/Ni couples. However, Wang et al.[12] studied the interfacial reactions in the Sn-51.0 at.%In/Ni couples at 150°C to 240°C for 15 min to 240 min, and they found the ternary phase, $Sn_{47}In_{20}Ni_{47}$, was formed at the interface.

Since Cu and Ni have the same face-centered cubic structure and form a continuous solid solution, it is likely that a large amount of Cu can be incorporated into the Ni sublattice in the Sn-In-Ni ternary intermetallic compound. Therefore, the (Cu + Ni) concentration in this phase could be considered as the Ni concentration in the Sn-In-Ni ternary system. The composition of the compound found in this study is thus Sn-31.7Ni-24.1In. It falls in the single Ni_3Sn_4 phase region in the In-Sn-Ni isothermal section at 160°C.[8] However, its composition is also similar to that found in Wang's study.[12] This brighter phase formed in the SAC/In/Ni/Cu couple could be the binary Ni_3Sn_4 phase with extensive Cu and In solubilities, or the ternary Sn-In-Ni compound with Cu solubility, or even a quaternary compound. Further phase characterizations, such as x-ray diffraction (XRD) or transmission electron microscope (TEM) analyses, need to be performed for better understanding. This brighter phase could be a metastable phase as well. The formation of metastable phases has been observed in various similar interfacial reaction studies.[17] A gray scallop-shaped phase region is observed adjacent to the homogeneous brighter phase layer in Fig. 3. Compositional analysis indicated that this is the Cu_6Sn_5 phase with Ni solubility. In addition to these two phases, both $AgIn_2$ and Ag_2In phases were formed and mixed in the solder phase. It has been observed that the size of the Ag_2In phase became larger with aging.

Figure 4 shows a BEI micrograph of the SAC solder reacting with the In/Ni/Cu multilayer at 200°C for 3 min. Only a dark and scallop-shaped region was found at the interface. The EPMA analysis and the Sn-Cu phase diagram[16] indicate that this region is the Cu_6Sn_5 phase of Sn-36.2 Cu-17.9Ni-1.8In. Comparing the surface morphology of the SAC/In/Ni/Cu system reflowed at 200°C with that at 160°C or 180°C, we found that the Cu_6Sn_5 phase did not spread over the interface at 200°C but that the Ni layer remained residually on the Cu surface. Additionally, the Ag_3Sn phase was found in the solder and its composition was Sn-74.0Ag based on EPMA analysis. Figure 5 shows the microstructure of the Ag_3Sn phase following etching with a solution (93.0 CH_3OH + 2.0 HCl + 5.0 HNO_3, in vol.%) for a few minutes to remove the solder. The dendrite-shaped microstructure in Fig. 5 is the typical solidification structure.[18] A similar result was found in the Sn-Ag/Ni system.[19]

A different surface morphology was observed between the SAC solder and the In/Ni/Cu multilayer when the SAC/In/Ni/Cu systems were reflowed at

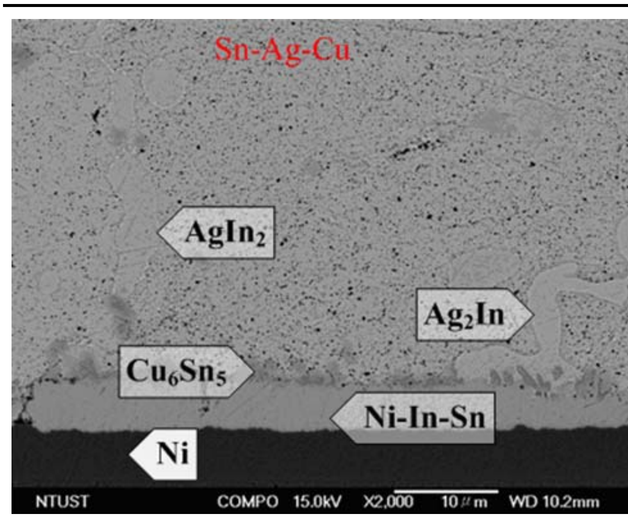

Fig. 3. BEI micrograph of the SAC/In/Ni/Cu system reflowed at 160°C for 4 min and then aged at 100°C for 300 h.

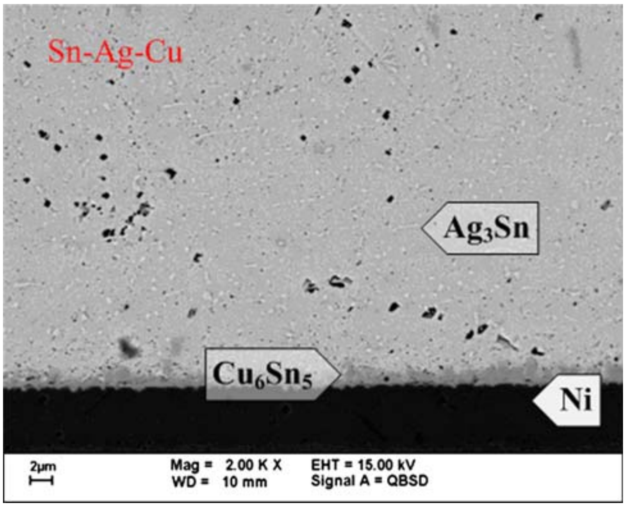

Fig. 4. BEI micrograph of the SAC/In/Ni/Cu system reflowed at 200°C for 4 min.

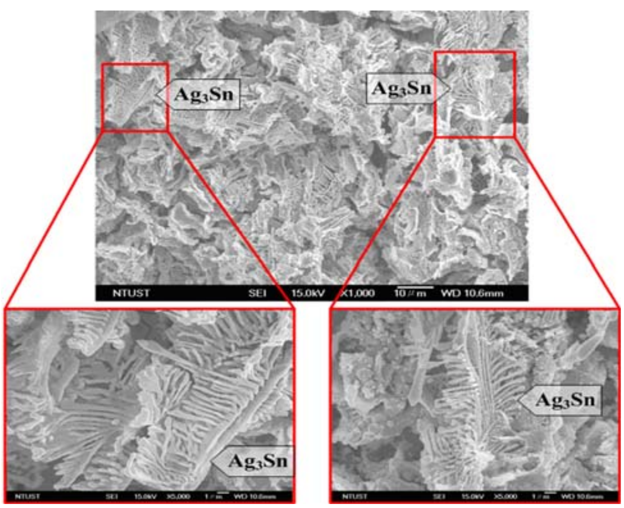

Fig. 5. Microstructure of the Ag_3Sn phase in the SAC/In/Ni/Cu system that was reflowed at 200°C for 3 min following use of an etching solution (93.0 CH_3OH + 2.0 HCl + 5.0 HNO_3, in vol.%) to remove the solder.

200°C and then aged at 100°C for a long period. As described above, the Ni-In-Sn ternary IMC was formed when the SAC/In/Ni/Cu system was reflowed at 160°C or 180°C and then aged at 100°C for a long period of heat treatment; the Cu_6Sn_5 phase still adhered to the Ni-In-Sn ternary IMC. However, only the Cu_6Sn_5 phase was formed at the interface and adhered to the Ni side, as shown in Fig. 6, which presents a BEI micrograph of the SAC/In/Ni/Cu system reflowed at 200°C for 4 min and then aged at 100°C for 400 h. The Ag_2In phase still can be observed in Fig. 6. Similar results were obtained when the SAC/In/Ni/Cu specimens were reflowed at 200°C for 3 min or 5 min and then heat-treated at 100°C.

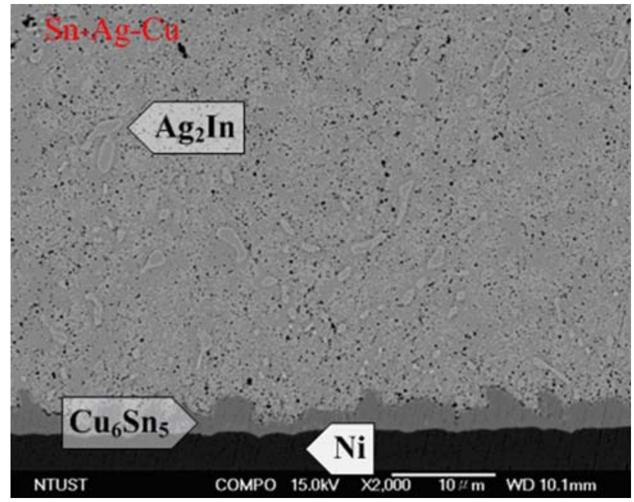

Fig. 6. BEI micrograph of the SAC/In/Ni/Cu system reflowed at 200°C for 4 min and then aged at 100°C for 400 h.

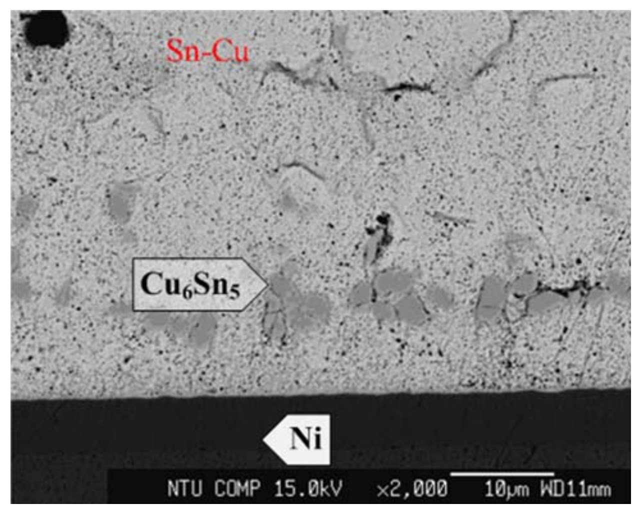

Fig. 7. BEI micrograph of the SC/In/Ni/Cu system reflowed at 160°C for 3 min.

Interfacial Reactions in Sn-0.7wt.%Cu Solders with In/Ni/Cu Multilayer

Figure 7 shows a BEI micrograph of the SC/In/Ni/Cu specimen that was reflowed at 160°C for 3 min. The spalling and dark color region of Sn-41.1Cu-11.9Ni-5.8In was formed at the interface. It is the Cu_6Sn_5 phase.[16] This result is very similar to that obtained in the SAC/In/Ni/Cu systems which were reflowed at 160°C for 3 min to 5 min.

Figure 8 shows a BEI micrograph of the SC/In/Ni/Cu specimen that was reflowed at 160°C for 3 min and then aged at 100°C for 300 h. Similar to the SAC/In/Ni/Cu specimen, the dark region spreading over the solder and the bright plane layer close to the Ni side were observed. The composition of the dark region is Sn-40.1Cu-12.7Ni-3.7In and it is the Cu_6Sn_5 phase.[16] The composition of the bright layer is Sn-14.0Cu-14.0Ni-26.0In, as revealed by EPMA

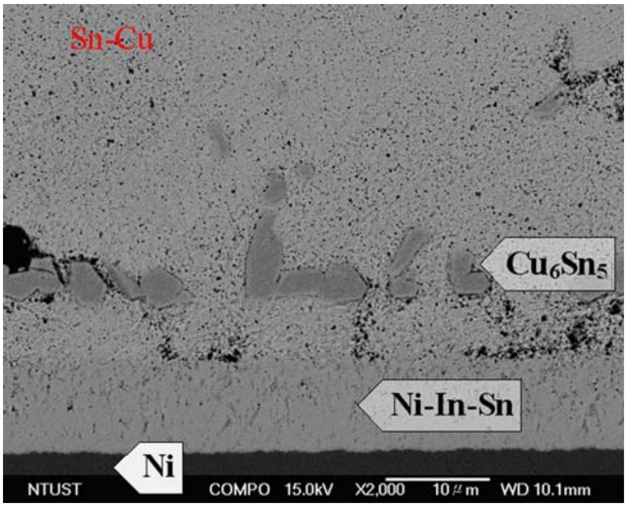

Fig. 8. BEI micrograph of the SC/In/Ni/Cu system reflowed at 160°C for 3 min and then aged at 100°C for 300 h.

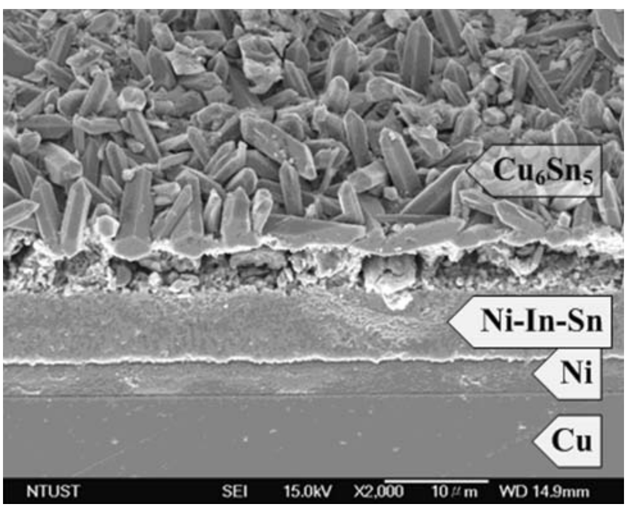

Fig. 9. Deep-etched SEI of the microstructure of the SC/In/Ni/Cu system that was reflowed at 160°C for 3 min and then aged at 100°C for 100 h.

analysis. Because the Cu can dissolve into the Ni lattice, the ratio of (Ni + Cu) to In to Sn is 28:26:46. This ratio is similar to that of the bright-layer region in the SAC/In/Ni/Cu specimen that was reflowed at 160°C and then aged at 100°C for a long period. The two compounds in the SC/In/Ni/Cu and SAC/In/Ni/Cu couples are likely the same. Figure 9 presents a deep-etched secondary electron image (SEI) of the microstructure of the interface between the SC solder and In/Ni/Cu multilayer that was reflowed at 160°C for 3 min and then aged at 100°C for 100 h. As presented in Fig. 9, the hexagonal prism is the Cu_6Sn_5 phase and is peeled off the interface, as well. Due to ripening of the Cu_6Sn_5 phases, the peeling-off phenomenon of the Cu_6Sn_5 phase from the substrate surface was commonly found in the SAC/Au/Ni/Cu and SC/Au/Ni/Cu systems.[10,13] The Ni-In-Sn ternary IMC was still planar.

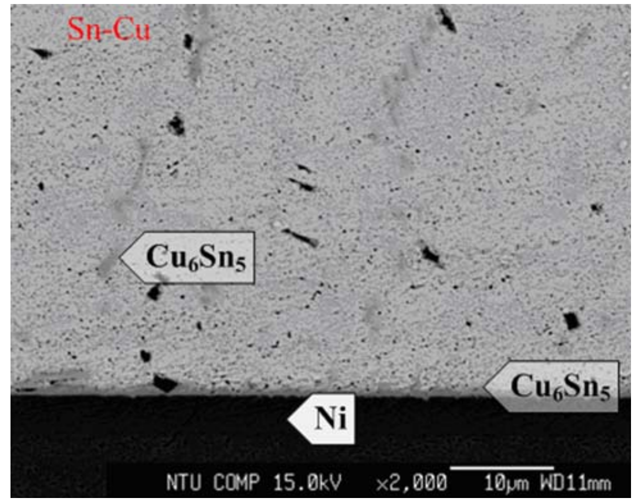

Fig. 10. BEI micrograph of the SC/In/Ni/Cu reflowed at 200°C for 3 min.

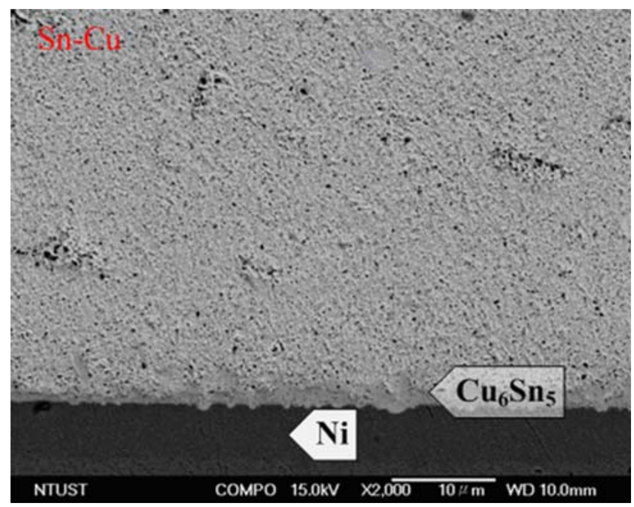

Fig. 11. BEI micrograph of the SC/In/Ni/Cu reflowed at 200°C for 3 min and then aged at 100°C for 300 h.

Figure 10 shows the surface morphology of the SC solder upon reaction with the In/Ni/Cu multilayer at 200°C for 3 min. Only the dark continuous layer was found; its composition was Sn-36.2Cu-17.9Ni-1.8In. As for the SAC/In/Ni/Cu systems, it is the Cu_6Sn_5 phase.[16] Figure 11 shows a BEI micrograph of the SC/In/Ni/Cu system that was reflowed at 200°C for 3 min and then aged at 100°C for 300 h. Similar to the SAC/In/Ni/Cu systems, only the Cu_6Sn_5 phase was formed and no Ni-In-Sn ternary IMC can be found at the solder/substrate interface. Similar results were obtained in the SC/In/Ni/Cu specimens that were reflowed at 200°C for 4 min or 5 min and then aged at 100°C for a long period.

Mechanism of IMC Growth in Solder/In/Ni/Cu Systems

The observation of different surface morphologies in both the SAC/In/Ni/Cu and SC/In/Ni/Cu systems

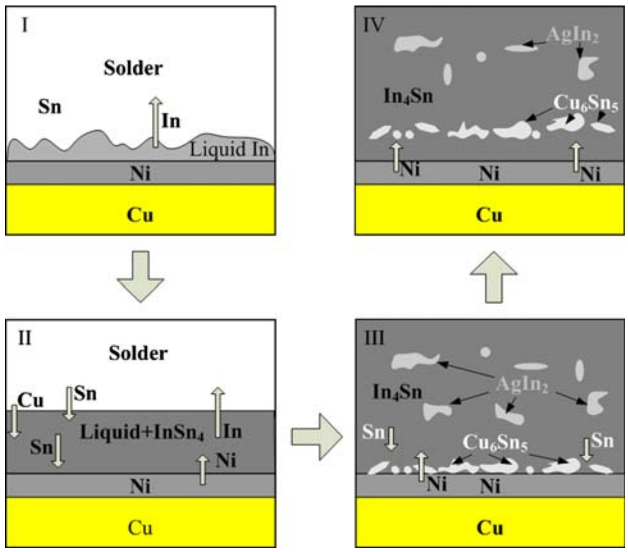

Fig. 12. Schematic diagrams of IMC formation of solder/In/Ni/Cu system.

reflowed at 160°C, 180°C, and 200°C and then heat-treated for a long period is interesting. When the reflowing temperature was below 180°C but above the melting point of In, the solid–liquid reaction occurred at the interface. In this stage, the Cu content in the solder was about 0.5 wt.% and the solder/substrate system was the SAC/Ni/Cu system. The interdiffusion of Cu and Sn atoms occurs, forming the Cu_6Sn_5 phase, as shown in Fig. 12I and II. According to the interfacial reaction results in the SAC/Ni couple by Ho et al., only the Cu_6Sn_5 phase is formed at the interface.[10] The results of our work are consistent with the literature. In atoms diffused to the SAC solder and reacted with Ag. The $AgIn_2$ phase formed in the SAC solder. Therefore, the In content in the solder decreased, and the Sn-In composition shifted toward that of the Sn-rich region in the Sn-In phase diagram. The solder/substrate interface remained in the liquid state; the Cu_6Sn_5 phase peeled off the substrate. More and more Sn atoms diffused toward the Cu side, diluting the concentration of In. The Sn-In composition reached the $InSn_4$ phase region. Finally, the $InSn_4$ phase was formed between the Cu_6Sn_5 phase and the Ni side, as shown in Fig. 12III and IV.

When the reflowed sample underwent heat treatment, Ni atoms diffused to the solder through the $InSn_4$ phase. However, the Cu_6Sn_5 phase acted as a diffusion barrier to prevent the diffusion of Ni atoms toward the solder. The Ni-In-Sn ternary IMC was formed between the Cu_6Sn_5 phase and the Ni side. However, the question of why the Ni-In-Sn ternary IMC is not formed when the reflowing temperature is 200°C remains. As in the solder/In/Ni/Cu sample that was reflowed at 160°C or 180°C, the In layer became liquid and the Cu_6Sn_5 phase formed at the interface. The In content decreased and shifted toward the Sn-rich region. At 200°C, compared with 160°C or 180°C, the stable phase more easily reaches the Sn-rich region, i.e., the β-Sn phase. Thus, only the Cu_6Sn_5 phase can be found at the solder/Ni interface.

Mechanical Properties Testing and SLID Technique

Figure 13 plots the tensile strength of the SAC/In/Ni/Cu system first reflowed at 160°C for 3 min to 5 min and then aged at 100°C for 100 h to 500 h. As presented in Fig. 13, the tensile strength clearly decreased as the heat treatment continued for over 100 h. The drop in the strength may have been caused by the formation of IMCs at the interface, as IMCs are hard and brittle. Thus, the formation of IMCs with excessive thickness reduces the solder-joint strength.[13] In this study, the Ni-In-Sn ternary IMC is observed at the interface after 100 h of heat treatment. Therefore, the formation of the Ni-In-Sn ternary IMC weakened the mechanical strength of the solder joints.

Another important aim of this study is to reduce the reflowing temperature applied to connect the SCA or SC solder with the Cu pad in the BGA or FC process. The In and Ni layers were deposited on the Cu surface by the electroplating method. Then, SAC or SC solders were reflowed below 200°C to connect the In/Ni/Cu multilayer substrate. This reflowing temperature was 70°C lower than the typical reflowing temperature of about 270°C. According to these results, the surface forms an effective connection between the solder and substrate, and the solder-joint strength is also good enough after a long period of heat treatment. The connection method used in this work is similar to the solid–liquid interdiffusion (SLID) technique. In the reflowing process, the In layer becomes liquid and Ni, Cu or solder remain in the solid state below 200°C.

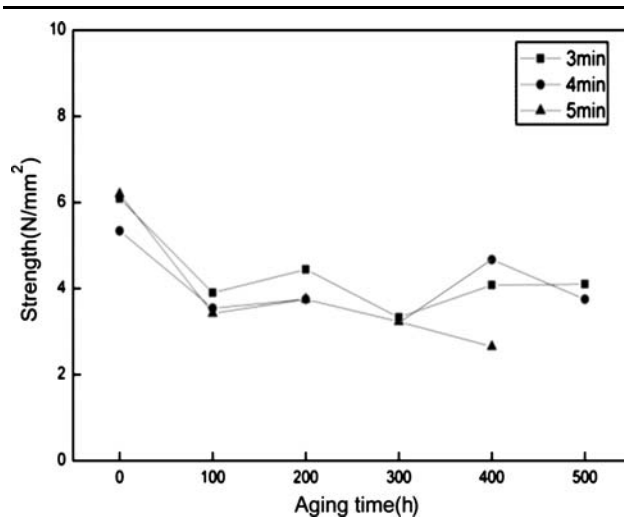

Fig. 13. Relationship between the tensile strength and the aging time in the SAC/In/Ni/Cu system that was reflowed at 160°C for 3 min to 5 min and then heat-treated for a long period.

Therefore, solid–liquid interdiffusion occurs between the solder-In and In-Ni/Cu interface. The local equilibrium condition allows the IMCs to form at the solder/substrate interface. The Cu_6Sn_5 phase is formed in both SAC/In/Ni/Cu and SC/In/Ni/Cu systems in the reflowing process in this work. Then the Cu_6Sn_5 phase or the Ni-In-Sn ternary IMC is formed after a long period of heat treatment. The IMCs enhance the bonding strength between the solder and substrate. The connecting solder/substrate method herein is similar to that adopted by Chen,[14] who used composite solders with an In layer to connect the solder and substrate. The SLID technique in this work effectively reduced the reflowing temperature and the cost of soldering. It can be used in BGA and FC electronic packaging.

CONCLUSIONS

Two kinds of lead-free solders, Sn-3.0Ag-0.5Cu and Sn-0.7Cu, were reacted with the In/Ni/Cu layer at 160°C, 180°C, and 200°C and then aged at 100°C for 100 h to 500 h. A scallop-shaped Cu_6Sn_5 phase was formed in all specimens and this phase spread over the substrate. Following a long heat treatment, planar Ni-In-Sn ternary intermetallic compounds (IMC) and a spalling Cu_6Sn_5 phase were found at the interface because the reflowing temperature was less than 200°C. This ternary Ni-In-Sn IMC could be the binary Ni_3Sn_4 phase with extensive Cu and In solubilities, the ternary Sn-In-Ni compound with Cu solubility, or even a quaternary compound. Further phase characterizations, such as XRD or TEM analyses, need to be performed for better understanding. Only the scallop-shaped Cu_6Sn_5 phase close to the Ni side was formed when the reflow temperature was 200°C. The formation of the Ni-In-Sn ternary IMC weakened the mechanical strength of the solder joints. Furthermore, the SLID technique in this work effectively reduced the reflow temperature.

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