Intermetallic Compounds Formed during the Reflow of In-49Sn Solder Ball-Grid Array Packages

T.H. CHUANG,[1,2] S.Y. CHANG,[1] L.C. TSAO,[1] W.P. WENG,[1] and H.M. WU[1]

1.—Institute of Materials Science and Engineering, National Taiwan University, Taipei 106, Taiwan.
2.—E-mail: tunghan@ccms.ntu.edu.tw

The intermetallic compounds formed at the interfaces between In-49Sn solder balls and Au/Ni/Cu pads during the reflow of In-49Sn solder, ball-grid array (BGA) packages are investigated. Various temperature profiles with peak temperatures ranging from 140°C to 220°C and melting times ranging from 45 sec to 170 sec are plotted for the reflow processes. At peak temperatures below 170°C, a continuous double layer of intermetallics can be observed, showing a composition of $Au(In,Ni)_2$/Au(In,Ni). Through selective etching of the In-49Sn solders, the intermetallic layer is made up of irregular coarse grains. In contrast, a number of cubic-shaped $AuIn_2$ intermetallic compounds appear at the interfaces and migrate toward the upper domes of In-49Sn solder balls after reflow at peak temperatures above 200°C for longer melting times. The upward floating of the $AuIn_2$ cubes can be explained by a thermomigration effect caused by the temperature gradient present in the liquid solder ball. The intermetallic compounds formed under various reflow conditions in this study exhibit different types of morphology, yet the ball shear strengths of the solder joints in the In-49Sn BGA packages remain unaffected.

Key words: In-49Sn solder balls, BGA, Au/Ni/Cu pads, intermetallic

INTRODUCTION

Gold thin films are widely used in electronic packaging to prevent oxidation and promote the wettability of Cu pads or Ni reaction layers. However, Au can be easily dissolved in Sn-based solders to form $AuSn_4$ precipitates, which can lead to the embrittlement of solder joints.[1] It has been reported that such a gold embrittlement failure in advanced packaging can be inhibited in In-Sn solders because of the lower solubility of Au in In-Sn.[2] Shohji et al. studied the solid/solid reaction between Au and In-49Sn solder and showed that an $AuIn_2$ layer and a γ-phase layer were formed at the Au/In-49Sn interface.[3] The activation energy for the formation of $AuIn_2$ was 42.8 kJ/mol, equivalent to the activation energy for the diffusion of Au in $AuIn_2$. Such a continuous interfacial $AuIn_2$ layer was believed to act as a barrier to the dissolution of Au in In-based solders.[4]

The Au/Ni/Cu is a popular pad structure of ball-grid array (BGA) packages. Owing to the merits of greater ductility, longer fatigue life, and lower melting point than the more traditional Pb-Sn solders,[5–9] the eutectic In-49Sn alloy is considered a Pb-free solder-ball material for BGA packages. This study is concerned with the characterization of intermetallic compounds formed at the interfaces between liquid In-49Sn solders and Au/Ni/Cu pads during the reflow of BGA packages under various conditions.

(Received May 30, 2002; accepted July 27, 2002)

EXPERIMENTAL

The geometry of BGA specimens used in this study is shown in Fig. 1. A silicon die of size of 4.5 mm × 4.5 mm × 0.25 mm was attached to a bismaleimide triazine (BT) resin substrate and encapsulated in a molding compound. Each package was fitted with 49 Cu pads electroplated with 5-μm-thick Ni and 0.5-μm-thick Au. The In-49 (wt.%) Sn solder balls of 0.4 mm in diameter were dipped in rosin, mildly activated flux, placed on the Au/Ni/Cu pads, and then reflowed in an infrared furnace. The reflow furnace had five heating zones with a typical temperature profile, as shown in Fig. 2. The reflow process was performed at various peak temperatures ($T_{max.}$) ranging from 140°C to 220°C for various melting time periods (Δt). As the temperature rose above the eutectic point of the In-49Sn solder (Te = 120°C), liquid/solid reactions took place at the interfaces between In-49Sn solder balls and Au/Ni/Cu pads.

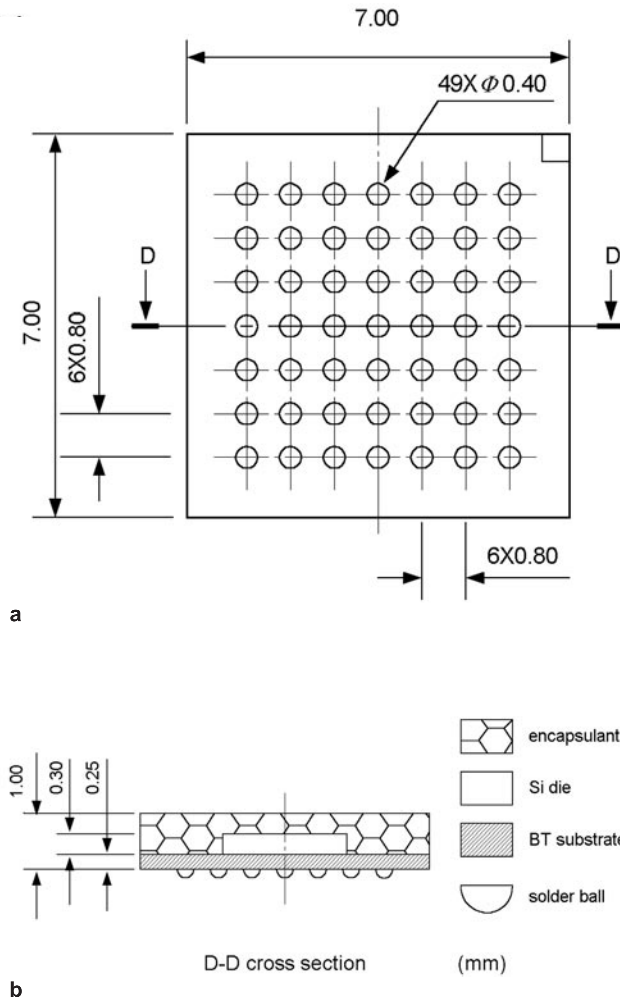

Fig. 1. Geometry of BGA specimens used in this study: (a) solder ball distribution and (b) package cross section.

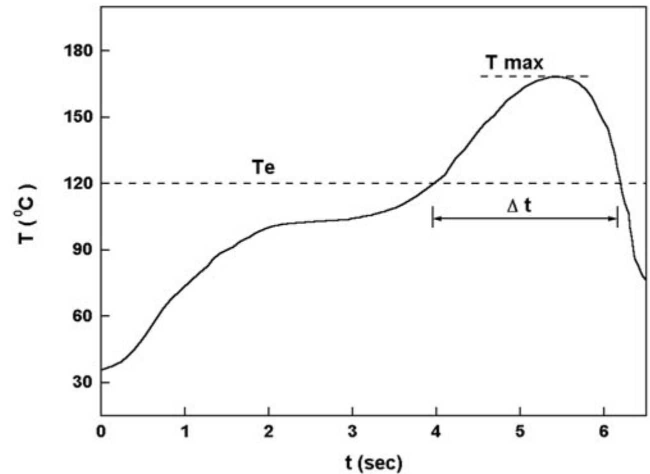

Fig. 2. Typical temperature profile for the reflow of In-49Sn solder BGA packages in this study ($T_{max.}$: peak temperature, Δt: melting time period, and Te: eutectic point of In-49Sn).

After reflow, the specimens were cross sectioned through a row of solder balls, ground with 1,500-grit SiC paper, and polished with 0.3-μm Al_2O_3 powder. The intermetallic compounds formed during reflow were observed with a scanning electron microscope (SEM) and analyzed using an electron probe microanalyzer (EPMA). The three-dimensional morphology of the intermetallic compounds was also probed through the selectively etched specimens. For this purpose, an etching solution of 10-mL HF, 10-mL H_2O_2, and 40-mL H_2O was employed to dissolve the In-49Sn solder and retain the intermetallic compounds at the solder/pad interfaces.

The bonding strengths of In-49Sn solder balls on Au/Ni/Cu pads under various reflow conditions were measured via ball shear tests. The measurements were taken at a shear rate of 0.1 mm/sec and a shear height of 80 μm (about 1/4 the reflowed ball height). The fractography of the solder joints after ball shear tests was examined in the SEM.

RESULTS AND DISCUSSION

Figure 3 shows the typical morphology of intermetallic compounds formed at the interfaces between In-49Sn solder balls and Au/Ni/Cu pads after reflow at the peak temperature of 170°C for various melting time periods. The intermetallic compounds appear in the form of a continuous double layer at this peak temperature even after a long melting time. The EPMA analyses show that the composition (at.%) of the outer layer is Au:In:Ni = 34.3:60.6:5.1, corresponding to a $Au(In,Ni)_2$ phase. However, the inner layer of the intermetallic compounds achieves a composition of Au:In:Ni = 51.2:41.5:7.3, which corresponds to an Au(In,Ni) phase.

As the peak temperature is increased to 220°C, the intermetallic compounds are found to change from a continuous layer into a nearly square shape, as shown in Fig. 4. Figure 5 shows the morphological variation of the intermetallic compounds formed during reflow at different peak temperatures for the BGA In-49Sn solder. The continuous intermetallic layers remain intact below 170°C, but at temperatures above 200°C, the intermetallic compounds begin to transform into a cubic shape. The cubic-shaped intermetallics can keep growing with the increase of peak temperature and melting time to a size larger than 10 μm, as shown in Figs. 4 and 5. To highlight such an interesting observation of intermetallic changes in morphology for the BGA In-49Sn solder reflowed under various conditions, Fig. 6 offers a clearer picture of the continuous $Au(In,Ni)_2$/Au(In,Ni) intermetallic double layer and the cubic-shaped $AuIn_2$ intermetallics formed, respectively, at the peak temperatures of 170°C and 200°C. The EPMA analyses indicate that the composition (at.%) of these cubic-shaped intermetallics is Au:In = 33.5:66.5, which matches the $AuIn_2$ phase. After the formation of the cubic $AuIn_2$ intermetallics, the Au film disappears, while the liquid In-49Sn solder continues to react with the Ni layer.

With the In-49Sn solder selectively etched away from the BGA specimen reflowed at the peak temperature 170°C for a melting time of 70 sec, Fig. 7a demonstrates that the continuous interfacial inter-

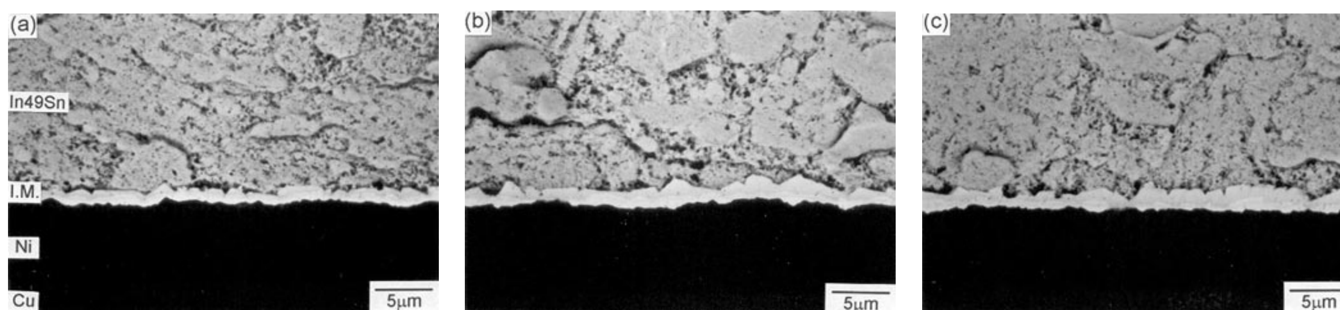

Fig. 3. Morphology of intermetallic compounds formed at the interfaces between In-49Sn solders and Au/Ni/u pads after reflow at 170°C for various melting time periods: (a) 60 sec, (b) 100 sec, (c) 120 sec.

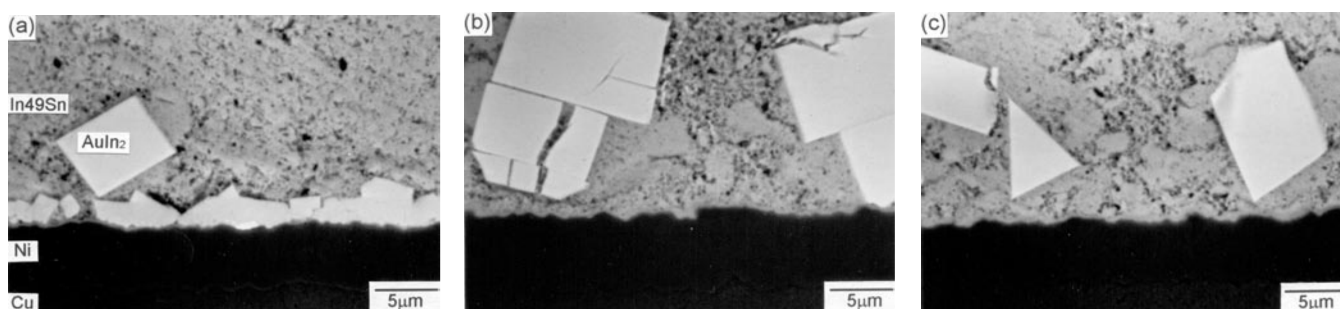

Fig. 4. Morphology of intermetallic compounds formed at the interfaces between In-49Sn solders and Au/Ni/Cu pads after reflow at 220°C for various melting time periods: (a) 105 sec, (b) 140 sec, and (c) 170 sec.

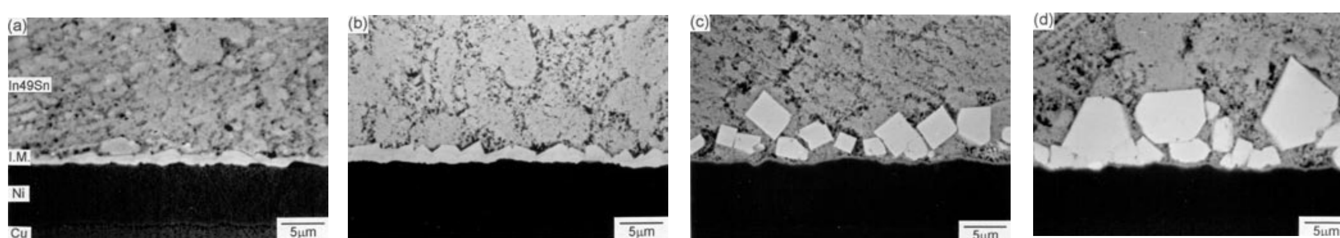

Fig. 5. Morphology of intermetallic compounds formed at the interfaces between In-49Sn solders and Au/Ni/Cu pads after reflow at various peak temperatures for various melting time periods: (a) 150°C, 125 sec; (b) 170°C, 140 sec; (c) 200°C, 160 sec; and (d) 220°C, 120 sec.

metallic layer from Fig. 3 is composed of irregular coarse grains with an average size of about 3 μm. Their typical chemical composition (at.%) as analyzed at Location A is Au:In:Ni = 75.4:8.3:16.3 , which corresponds to an ordered ε′ phase. In Fig. 7a, a few regions in the coarse-grained matrix are covered with fine-grained clusters, which have a composition of Au:In:Ni = 87.6:10.4:2.0, corresponding to the α_1 phase. With the melting time increased to 140 sec at the peak temperature of 170°C, it is found in the selectively etched specimen that those irregular coarse grains have grown further to an average size of about 6 μm. The composition as analyzed from Location C is Au:In:Ni = 31.7:60.1:8.2, corresponding to the $Au(In,Ni)_2$ phase similar to that of the outer intermetallic layer in Fig. 3. Accompanying the grain growth in the matrix, the fine-grained clusters in the α_1 phase disappear, resulting in the exposure of a number of small areas beneath the $Au(In,Ni)_2$ intermetallic coarse grains, as shown in Location D. The composition of these areas is Ni:In:Sn = 75.1:17.1:7.8, corresponding to the $Ni_3(In,Sn)$ phase. Although Ni_3In and Ni_3Sn are listed in the Ni-In and Ni-Sn phase diagrams, they have never been reported as the products of soldering reactions between In (or Sn, InSn) and Ni substrates.[11–13] During the interfacial reactions between liquid In-49Sn solders and Ni substrates at temperatures ranging from 150°C to 300°C, a $Ni_{33}In_{18.5}Sn_{48.5}$ intermetallic compound was observed, rather than the $Ni_3(In,Sn)$ phase.[13] The Ni_3Sn intermetallic compound, was reported by Bader et al. in their study on the intermetallic formation during the interdiffusion between Ni and Sn thin films, respectively, of 4.1 μm and 2.6 μm in thickness.[14] After heating at 400°C for 90 h, the Sn film was exhausted, which spurred the reaction of the formerly created Ni_3Sn_2 phase with the remainder of the Ni film to form a columnar-grained Ni_3Sn intermetallic. In the present study, the appearance of the $Ni_3(In,Sn)$ phase is believed to also result from insufficient In and Sn supply caused by the barrier effect of the $Au(In,Sn)_2$ layer wrought on the In-49Sn solder. The limited amount of In and Sn from the $Au(In,Sn)_2$ layer reacts with the thicker Ni

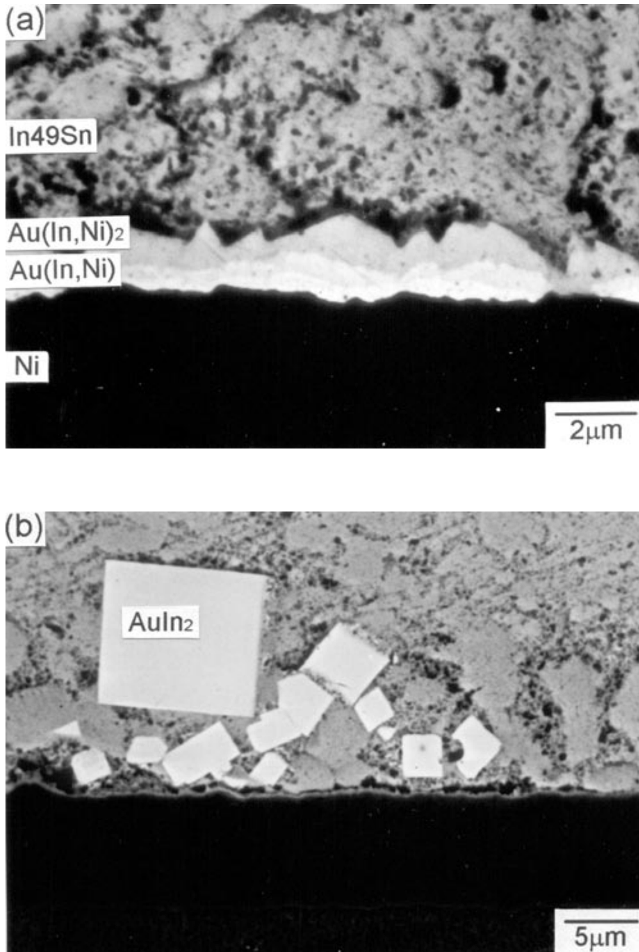

Fig. 6. Continuous double layer $Au(In,Ni)_2$/Au(In,Sn) and cubic shape $AuIn_2$ intermetallic compounds formed during the reflow of BGA in-49Sn solder at (a) 170°C for 140 sec and (b) 200°C for 160 sec.

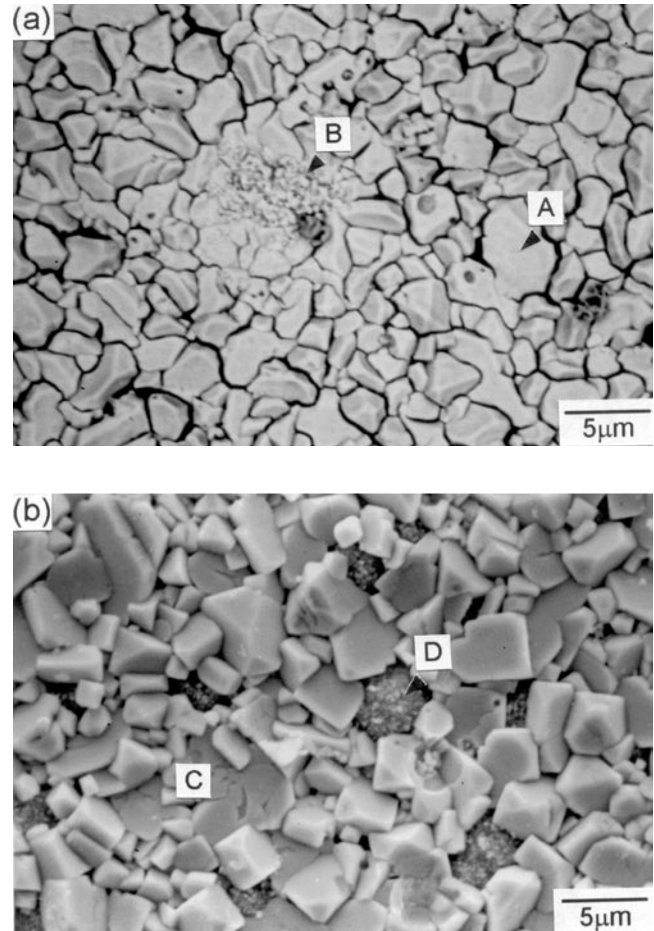

Fig. 7. Selective etching of the In-49Sn solders showing that irregular granular interfacial intermetallic compounds are formed in BGA specimens after reflow at 170°C for various melting time periods: (a) 70 sec and (b) 140 sec.

layer to form the Ni-rich Ni_3(In,Sn) intermetallics, conducive also to the transformation of particle $Au(In,Sn)_2$ into Au(In,Sn) adjacent to the Ni_3(In,Sn) phase.

Figure 8a shows a selectively etched specimen whose three-dimensional morphology is given as evidence of the $AuIn_2$ intermetallic compounds of cubic shape formed at peak temperatures above 200°C. It has been reported that $AuIn_2$ has a CaF_2 (C1)-type structure.[10] The appearance of cubic-shaped $AuIn_2$ intermetallic compounds (Fig. 8a) implies that the surface energy of their (100) planes is quite low. Figure 8b is a magnified view of the matrix from Fig. 8a, which reveals a morphology of very fine grains. The EPMA analysis identifies the composition of the fine-grained matrix as Ni:Sn:In = 43.4:39.2:17.4, which corresponds to the $Ni_3(In,Sn)_4$ phase. The $Ni_3(In,Sn)_4$ intermetallic compound at the interface of the In-49Sn solder BGA package is consistent with the reaction product Ni_3Sn_4, which appeared during the soldering reactions between liquid Sn and Ni substrates, contrasting that in the literature to the formation of $Ni_{33}In_{18.5}Sn_{48.5}$ intermetallic at the In-49$Sn_{(L)}$/$Ni_{(S)}$ interface.[13] The discrepancy can be attributed to the excessive consumption of In from the In-49Sn during its reaction with Au film, which results in an In-Sn alloy with high Sn content adjacent to the Au/Ni/Cu pads to react with the Ni layer.

Another interesting phenomenon can be observed from Fig. 9, i.e., the $AuIn_2$ intermetallic cubes float toward the top of the In49-Sn solder ball. As the peak reflow temperature increases, more $AuIn_2$ cubes migrate through the In-49Sn solder ball and settle on its upper dome. Because $AuIn_2$ is specifically much denser than In-49Sn solder, it seems unreasonable that the heavier $AuIn_2$ is able to float upward in liquid In-49Sn solder. An explanation is proposed based on the thermomigration theory:[15,16] the upper dome of the solder ball is irradiated with infrared heat during reflow, which gives rise to a temperature gradient between the top and bottom of the solder ball. Because of this temperature gradient, the liquid In-49Sn solder migrates downward, and the $AuIn_2$ cubes are squeezed upward instead to settle on the upper side of the solder ball.

Fractography on all BGA specimens reflowed under various conditions shows ductile failure char-

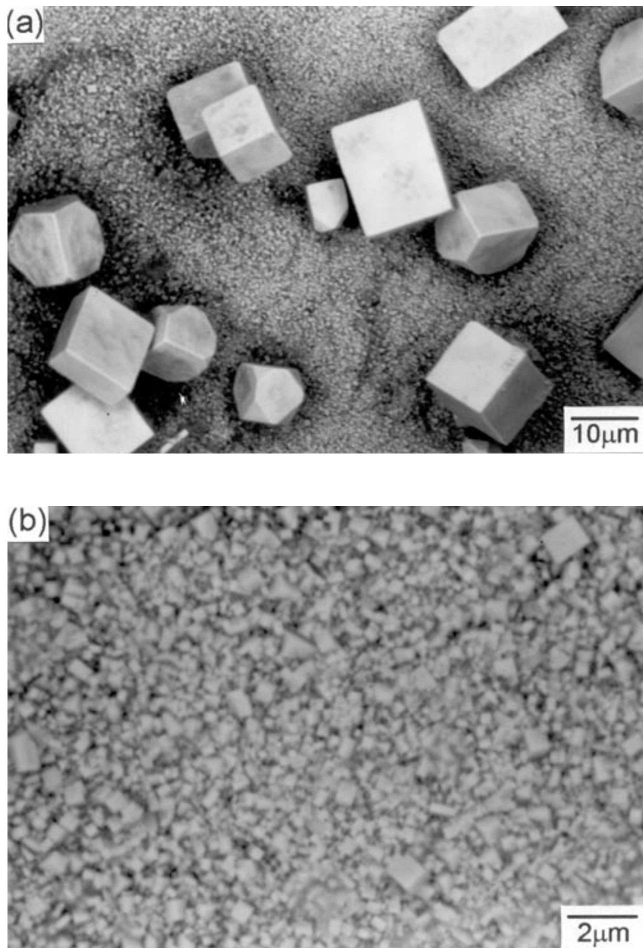

Fig. 8. (a) Selective etching of the In-49Sn solder showing that the cubic $AuIn_2$ interfacial intermetallic compounds are formed in BGA specimens after reflow at 220°C for 140 sec. (b) Magnified morpohology of the matrix in Fig. 8a.

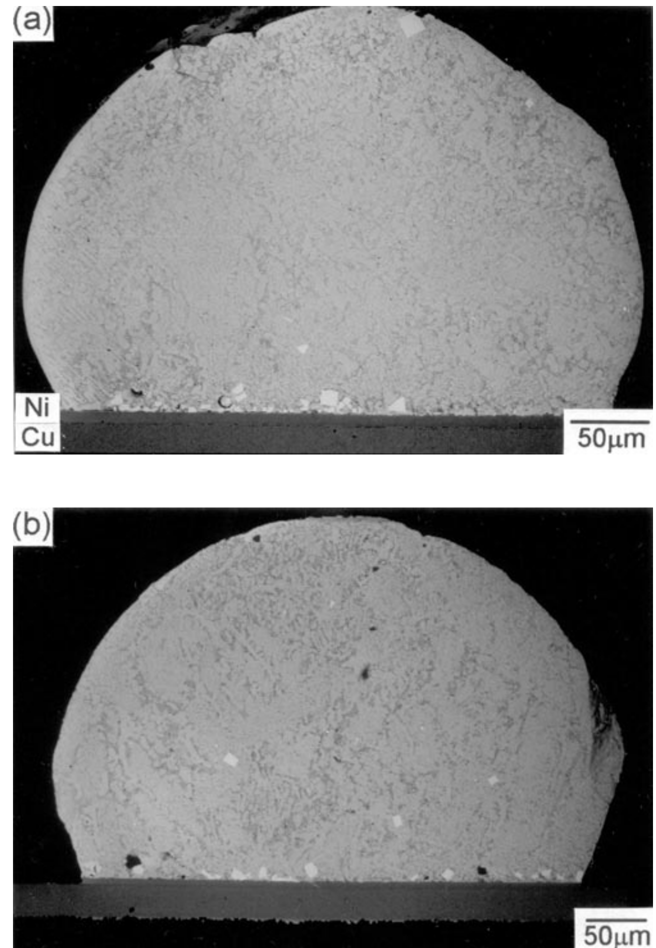

Fig. 9. Migration of $AuIn_2$ cubes toward the upper domes of In-49Sn solder balls after reflow at high peak temperatures: (a) 200°C, 160sec, (b) 220°C, 140sec.

acteristics after ball shear testing (Fig. 10). Fracture occurs to all the solder joints as cracks propagate through the In-49Sn solder balls. The fracture behavior of such BGA In-49Sn is affected by neither the continuous intermetallic layer formed below 170°C nor the cubic-shaped $AuIn_2$ formed above 200°C. Independent of various reflow conditions, the ball shear strengths of all In-49Sn BGA packages used in this study are in the vicinity of 3 N, as listed in Table I.

CONCLUSIONS

During the reflow of In-49Sn BGA packages, interfacial reactions occur between the liquid In-49Sn solder balls and Au/Ni/Cu pads. As the experimental results indicate, there are two types of intermetallic compounds formed after reflow under various reflow conditions. At peak temperatures below 170°C, a continuous bilayered $Au(In,Ni)_2/Au(In,Ni)$ intermetallic appears at the interface. However, a cubic-shaped $AuIn_2$ intermetallic can be observed at peak temperatures above 200°C. By selectively etching the In-49Sn solder balls, the remaining intermetallic compounds formed at the early stage at 170°C show a composition of $Au_{87.6}In_{10.4}Sn_{2.0}$, which are later transformed into the double layer $Au(In,Ni)_2/Au(In,Ni)$ phases. Underneath the granular $Au(In,Ni)_2/Au(In,Ni)$ intermetallics, there also exists an $Ni_3(In,Sn)$ phase. Through selective etching of the specimens, the cubic-shaped $AuIn_2$ intermetallics formed at 200°C are shown to grow in a fine-grained $Ni_3(In,Sn)_4$ matrix. The $AuIn_2$ cubes are observed to float toward the top of the In-49Sn solder ball, which might be attributed to the thermomigration phenomenon resulting from the temperature gradient between the top and bottom of the solder ball. Ball shear tests on these BGA specimens indicate that the strengths of In-49Sn solder joint are unaffected by morphological differences in the intermetallic compounds formed under various reflow conditions.

ACKNOWLEDGEMENTS

We thank the National Science Council (NSC) in Taiwan for sponsoring this research project under Grant No. 90-2216-E002-032.

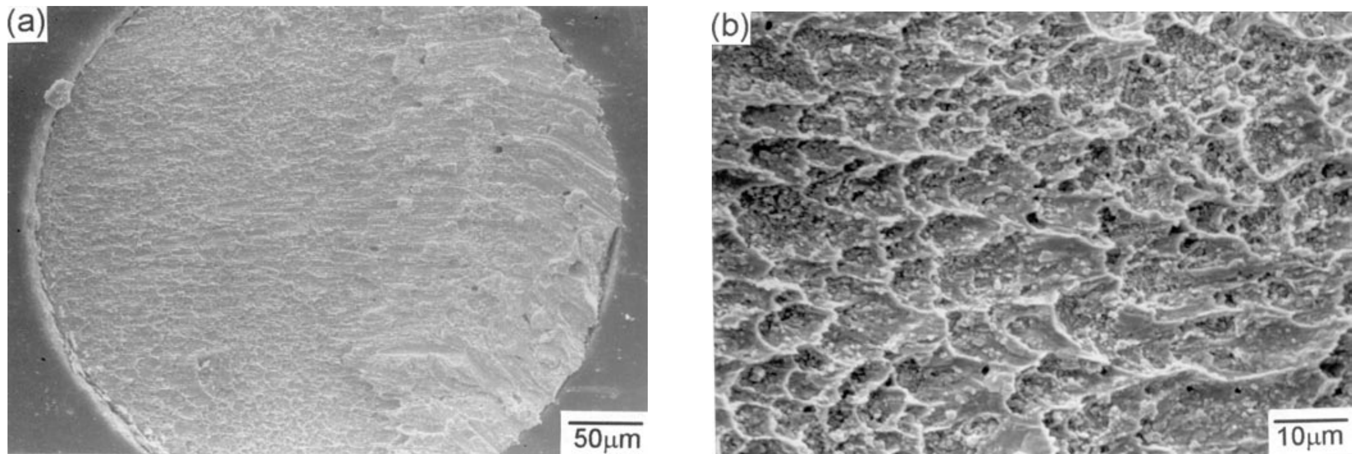

Fig. 10. Typical fractography of In-49Sn solder joints of the BGA packages used in this study after ball shear tests.

Table I. Ball Shear Strengths (Newtons) of the In-49Sn Solder Ball-Grid Array Packages in This Study after Reflow at Various Peak Temperatures for Various Melting Time Periods

Peak Temperature* (°C)	Melting Time Periods** (sec)						
	45	60	70	100	120	140	170
140	2.55	2.86	2.97	2.85	3.12	—	—
150	—	2.73	2.84	2.94	2.96	3.20	—
160	—	2.53	2.97	3.00	2.94	2.98	—
170	—	2.74	3.30	3.22	3.10	3.19	—
200	—	—	—	—	—	3.01	3.02
220	—	—	—	2.79	2.79	2.66	2.89

* Peak temperature: The maximum temperatures on the reflow temperature profiles.
** Melting time periods: The time periods in which the reflow temperatures are above the melting point of In-49Sn solder (120°C).

REFERENCES

1. D.M. Jacobson and G. Humpston, *Gold Bull*. 22, 9 (1989).
2. N.C. Lee, *Solid Surf. Mount Technol.* 26, 65 (1997).
3. I. Shohji, S. Fujiwara, S. Kiyono, and K.F. Kobayashi, *Scripta Mater.* 40, 815 (1999).
4. F.G. Yost, F.P.G. Anyard, and M.M. Karnowsky, *Metall. Trans. A* 7A, 1141 (1976).
5. Z. Mei and J.W. Morris, *J. Electron. Mater.* 21, 599 (1992).
6. S.K. Kang and A.K. Saarkhel, *J. Electron. Mater.* 23, 701 (1994).
7. J. Glazer, *Int. Mater. Rev.* 40, 65 (1995).
8. K. Shimizu, T. Nakauishi, K. Karasawa, K. Hashimoto, and K. NiWa, *J. Electron. Mater.* 25, 39 (1996).
9. Z. Mei and J. Morris, *J. Electron. Mater.* 21, 401 (1992).
10. E. Zintl, A. Harder, and W. Haucke, *Z. Phys. Chem. B* 35, 354 (1937).
11. S.K. Kang and V. Ramachandran, *Scripta Metall.* 14, 421 (1980).
12. Y.H. Lin, Y.L. Chen, and T.H. Chuang, *J. Electron. Mater.* 29, 1047 (2000).
13. Y.H. Tseng (Ph.D. thesis, National Taiwan University, 2000).
14. S. Bader, W. Gust, and H. Hieber, *Acta Metall. Mater.* 43, 329 (1995).
15. G.J. Van Gurp, P.J. de Waard, and F.J. du Chatenier, *J. Appl. Phys.* 58, 728 (1985).
16. T.Y. Tan, *Appl. Phys. Lett* . 73, 2678 (1998).