Optimization of Ag Composition in Cu Pillar Bumps with Sn-xAg Solders

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Abstract

In order to minimize the large plate growth of Ag_3Sn intermetallic compounds (IMCs) adversely affecting the mechanical behavior and reducing the reliability of solder joints, the Ag content of less than 3wt% has been suggested in ball grid array (BGA) Sn-Ag solders. The suggestion (< 3wt% Ag in the solders) is invalid in small solder bumps, and moreover there is no available guideline of the Ag content in the small Sn-Ag solders. In this study, the optimum level of Ag content in small Sn-Ag Cu pillar bumps (CPBs) of less than 50um in diameter was investigated to suppress the large plate growth of Ag_3Sn. Since the undercooling of Sn-Ag solders increases with a decrease of solder ball size, the Ag_3Sn IMCs are more prone to become largely plate-like in small solders. Actually, in small Sn-Ag CPBs, the large plate growth of Ag_3Sn was observed on the top surface of solder bumps even though the Ag content was less than 3.0wt%. For suppression of Ag_3Sn plates in Sn-Ag CPBs, two ways were suggested; increasing the cooling rate of the reflow profile and reducing the Ag content in the solders. Reducing the Ag content was effective on reducing the large plate growth of Ag_3Sn, while a high cooling rate was not effective. Through the thermal analysis and thermodynamic calculations, the optimized Ag content in Sn-Ag CPBs of less than 50um was suggested, and the large plate growth of Ag_3Sn was effectively reduced within the suggested Ag content.

***Keywords*:** Cu Pillar Bump, Sn-Ag Pb-free Solders, Intermetallic Compound, Undercooling, Thermodynamics

1. Introduction

Many studies for Pb-free solder alloys have been conducted in the last several years [1-5]. The Pb-free solders are now common subject in electronic packaging, and various Pb-free solders have been used from surface-mounted technology (SMT) to chip-level interconnections. In addition, Sn-rich Pb-free solders with various alloying elements, such as Ag, Cu, Zn, Ni, Co, and Bi, are continually evaluated to produce reliable Pb-free solder joints [3-5]. A silver (Ag) alloying element among the various elements has been broadly used in Pb-free solders because it is beneficially effective on decreasing the melting point [6] and improving wettability [6], and mechanical properties [7]. However, the formation of pro-eutectic Ag_3Sn intermetallic compounds (IMCs) is a big problem in using Ag-containing Sn-rich Pb-free solders, because Ag3Sn IMCs form a large plate in the matrix of solders during reflow. The large Ag_3Sn plates can adversely affect the plastic deformation properties of the solder [9] and cause plastic-strain localization at the boundary between the Ag_3Sn plates and the surrounding β-Sn phase [9]. In a previous report, the crack is actually initiated at the interface of a large Ag_3Sn plate and the β-Sn phase, and then propagated along the interface [10]. Therefore, lots of studies have been conducted to suppress the large plate growth of Ag_3Sn IMCs in the past few years, and a proper guideline for suppressing the large plate growth of Ag_3Sn IMCs has been established in surface-mounted card assembly such as BGA solder joints: a higher cooling rate (> 1.5°C/sec), a lower Ag content (< 3wt%), and a small amount of undercooling (< 5°C) [10-12].

The Ag alloying element is important in a chip-level interconnection as well. The Sn-based Pb-free solders used in the chip-level interconnection usually have a higher melting temperature than Sn-Ag-Cu solders (215°C) used in SMT, and then a Sn-Ag or Sn-Cu binary solder is proposed as the chip-level Pb-free solders. In recent studies, the Sn-Ag binary solders are more reliable than the Sn-Cu solders in terms of electromigration [13], microstructure, and mechanical proprieties [14]. Nevertheless, many industries experience the large plate growth of Ag_3Sn IMCs in using Sn-Ag binary solders, and spend lots of time and efforts to suppress Ag_3Sn plates. Moreover, although Sn-Ag binary solders with low Ag content are suggested in a chip-level interconnection, there is no available guideline on the optimum level of Ag content for suppressing the large plate growth of Ag_3Sn IMCs in C4 solder bumps and Cu pillar bumps (CPBs) with the diameter of less than 50um.

In this present work, the formation of large Ag_3Sn plates was investigated with small Sn-Ag CPBs (< 50um). To understand why the large plate growth of Ag_3Sn IMCs formed in Sn-Ag solder with 2.3-2.5wt% Ag, the thermal behaviors, such as heating peak, cooling peak, and undercooling, were measured experimentally with thermal analysis. For suppression of Ag_3Sn plates in Sn-Ag CPBs, two ways were suggested; increasing the cooling rate of the reflow profile and reducing the Ag content in the solders. In addition, the thermodynamic calculations were conducted to suggest the optimum level of Ag content, and the suggested Ag content was experimentally verified.

2. Experimental Procedures

The height and diameter of Sn-Ag solders on CPBs were less than 50um. The Sn-Ag CPBs were fabricated by a typical wafer-level bump process: sputtering for the formation of under bump metallurgy (UBM), photo lithography (photo resist coating, exposure, develop), electroplating for Cu pillars and Sn-Ag solders, etch, and reflow. After reflow, the bumped wafer was inspected by optical inspection systems to measure the 3D height of CPBs. The Ag content in Sn-Ag CPBs was 2.3-2.5wt%, which was confirmed by X-ray fluorescence (XRF) after reflow.

The secondary electron mode of scanning electron microscopy (SEM) was used to observe Ag_3Sn IMCs formed on the surface of Sn-Ag CPBs, and energy-dispersive X-ray spectroscopy (EDS) was used for their identification. To measure the undercooling of Sn-Ag CPBs, differential scanning calorimetry (DSC) experiments were performed in a diamond DSC calorimeter (Perkin-Elmer, Inc.), which was heated and cooled at the rate of 6°C/min under a nitrogen atmosphere with hundreds of CPBs.

In addition, thermodynamic calculations were performed to understand the experimental results and suggest the optimum level of Ag content by using the Thermo-Calc thermodynamic software developed at the Royal Institute of Technology, Stockholm, Sweden [15].

3. Results and Discussion

Large Plate Growth of Ag_3Sn Intermetallic Compounds in Cu Pillar Bumps

Figure 1 is the yield results for 3-D height of Sn-Ag CPBs to confirm whether all bumps formed on a 12” wafer meet a specification. In Fig 1, a few chips were out of the specification owing to low height of some CPBs. However, through an optical microscope, the shape and height of CPBs were definitely normal (as shown in Fig 1), but the surface brightness of solder bumps was abnormal and a little dark, compared to nearby shiny solder bumps. Since the optical inspection system for measuring the 3-D height of CPBs basically sense the surface brightness of CPBs, the decrease of the surface brightness leaded to errors measuring the height of CPBs

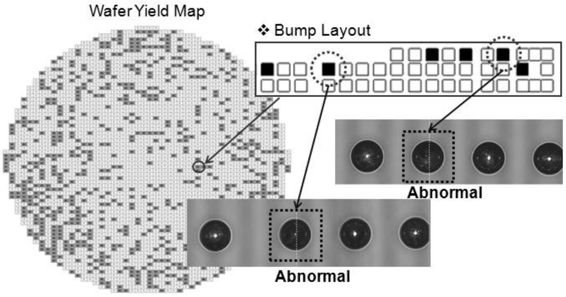


Figure 1. The yield results for 3-D height of Sn-Ag CPBs on a 12” wafer and the optical images of defective bumps.

To find out the reason for the decrease of the surface brightness in the CPBs, the surface of solder bumps were observed by SEM. The formation of one IMC phase was found on the top surface of solder bumps (as shown in Fig 2), and the morphology appeared large plate-like. The IMC phase consisted Sn and Ag atoms through the EDS analysis, and the compositional ratio of Sn and Ag matched to Ag_3Sn IMCs. Consequently, the decrease of the surface brightness was resulted from the large plate growth of Ag_3Sn IMCs.

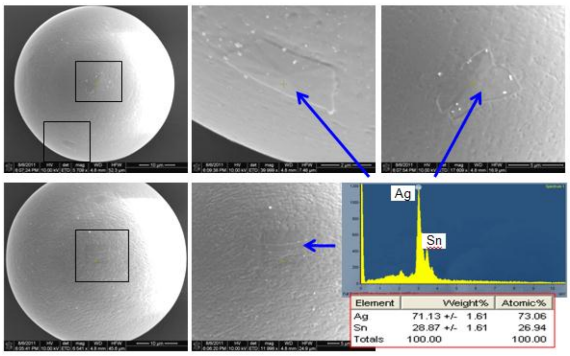


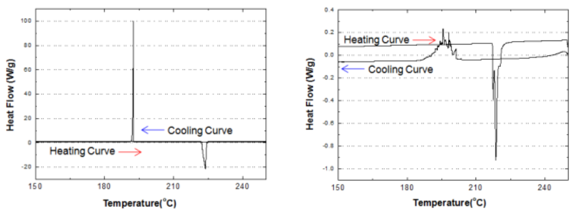
Figure 2. SEM images for the surface on defective bumps and phase identification by EDS.

In general, the large plate growth of Ag_3Sn IMCs is a natural phenomenon in Sn-Ag solders over 3.5wt% Ag (eutectic content), because the Ag_3Sn IMC is a first-forming solid phase as a pro-eutectic phase during solidification. However, it is reported that the large plate-like Ag_3Sn IMCs can be formed under 3.5wt% Ag, which is caused by slow cooling rate and a large amount of undercooling in Sn-Ag binary solders [10-11]. Since the nucleation of β-Sn phases is more difficult than that of Ag_3Sn IMCs during cooling of Sn-based molten solders, the large amount of undercooling can cause the large plate growth of Ag_3Sn IMCs in a supersaturated liquid during cooling [10,16].

Thermal Behaviors of CPBs with Sn-xAg Solders through DSC Measurements

To understand why the large plate growth of Ag_3Sn IMCs forms in Sn-Ag solders with 2.3-2.5wt% Ag content (under the eutectic composition of 3.5wt% Ag), the undercooling of Sn-Ag CPBs (< 50um) was measured by DSC. Figure 3(a) is the typical thermal profile recorded during heating and cooling of Sn-rich solders. In general, the amount of undercooling is referenced to the equilibrium melting point: The amount of undercooling means the difference between the equilibrium melting point and the real solidification temperature. In previous literatures, the amount of undercooling can be determined by comparing the onset temperature of the cooling curve to the peak temperature of the heating curve [20].

In case of small Sn-Ag CPBs, they are too small to detect their thermal changes, and therefore hundreds of CPBs should be used for DSC measurements. As a result, the cooling curve of the small Sn-Ag CPBs displays small multiple peaks while their heating curve does one or two large peaks, as shown in Fig 3(b). This DSC curve for the hundreds of solder bumps is quite consistent with the previous report [18-19]. The small multiple cooling peaks are attributed to several or individually solidifying solder bumps. To determine the undercooling of small CPBs, we measured the difference between onset temperature of heating curve and peak temperature of cooling curve, and considered both of the first cooling peak and the last cooling peak.



(a) (b)

Figure 3. (a) A typical DSC curve of Sn-Ag bulk solders and (b) A DSC curve of small Sn-Ag CPBs (< 50um).

Table 1. The DSC results of Sn-Ag CPBs as a function of Ag content (from 1.5wt% to 2.3wt%).

Ag Content	Heating Curve			Cooling Curve			Undercooling	
	Onset	Peak	Endset	First	Median	Last	Onset-to-First	Onset-to-Last
Sn-2.3wt% Ag	217.1	218.5	221.1	203.7	195.6	183.3	13.4	33.8
Sn-1.8wt% Ag	217.1	218.7	223.7	205.7	199.9	182.3	11.4	34.8
Sn-1.5wt% Ag	217.1	218.5	223.0	204.3	199.5	184.1	12.8	33

Table 1 summarizes the DSC results of Sn-Ag CPBs as a function of Ag content (from 1.5wt% to 2.3wt%). In case of Sn-Ag CPBs with 2.3wt% Ag, the first and last cooling peak were 200°C and 180°C respectively, yielding the undercooling of 15-35°C. It is reported that the undercooling of bulk Sn-rich solders is around 30°C and that of Sn-rich solders reacted to under bump metallurgy (UBM) is around 15-20°C [21-22]. Therefore, for Sn-Ag CPBs of less than 50um, the undercooling of some Sn-Ag CPBs (< 50um) displays the double undercooling of Sn-rich solders with UBMs. This is a very good agreement with the previous report: the amount of β–Sn undercooling in Sn-rich solders is inversely proportional to sample size, suggesting a larger undercooling in a smaller solder joint (such as flip chip versus BGA solder joints) [17-19]. It is explained that the probability that several clusters grow to nuclei would be reduced as the volume of molten solders decreases, and as a result the solidification of molten solders with small volume occurs at a lower temperature. Through the DSC results, large Ag_3Sn plates in Sn-Ag CPBs with 2.3-2.5wt% Ag are attributed to the large undercooling accompanied by small volume of Sn-Ag solders (< 50um).

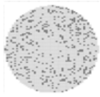
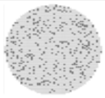
In addition, the onset temperature of heating curve in Table 1 was similar regardless of Ag content, while the peak temperature and the endset temperature were slightly changed by the Ag content. Moreover, the first peak temperature during cooling was observed at around 200°C, and the last peak temperature was around 180°C. Both of first and last temperature were similar regardless the Ag content, yielding the undercooling of 15-35°C by the onset-to-peak measurement. It is found that the undercooling of Sn-Ag CPBs is independent on the Ag content in the CPBs.

Suppression of Ag_3Sn IMCs by Controlling the Cooling Rate during Reflow

To suppress the large plate growth of Ag_3Sn IMCs, controlling cooling rate during reflow could be suggested because the fast cooling restricts the growth time of the Ag_3Sn plate even though small Sn-Ag CPBs exhibit the undercooling of about 15-35°C. In the previous report, the fast cooling of more than 1.5°C/sec is recommended in the Sn-Ag solder balls (> 300um in diameter) with the Ag content of 3.0wt% [10-11]. The cooling rate of a reflow profile used in this study was higher than the suggested cooling rate. However, the cooling rate used in this study was not effective on reducing the Ag_3Sn plates, and then we increased the cooling rate by 1.66 times.

Table 2 summarizes the 3-D height yield for Sn-Ag CPBs changed by the cooling rate. Although the cooling rate increased 1.6 times higher, the 3-D yield was scarcely improved. The ineffectiveness of controlling the cooling rate might be due to the thermal mass of a 12” wafer.

Table 2. The 3-D height yield in Sn-Ag CPBs changed by the cooling rate.

Wafer Slot	Cooling Conditions	Change of 3-D Height Yield	Wafer Map
#1	Ref.	-	
#2	46% Higher	1.8% Down	
#3		5.3% Down	
#4	66% Higher	1.8% Down	
#5		0.8% Up	

Thermodynamic Calculations for Optimization of Ag Content

To fundamentally retard the plate growth of Ag_3Sn IMCs, the Ag content of Sn-Ag CPBs should be reduced. Actually, the yield of 3-D height declined by the formation of Ag_3Sn IMCs increased as the Ag content in the Sn-Ag CPBs decreased, as shown in Fig. 5. Then, how much do we need to reduce the Ag content in the Sn-Ag CPBs? We thermodynamically estimated the proper level of Ag composition at which the large plate of Ag_3Sn IMCs does not grow up in Sn-Ag CPBs of less than 50um. The thermodynamic calculation was conducted with the ThermoCalc software and the data base released by Scientific Group Thermodata Europe (SGTE, revised 2004) was used.

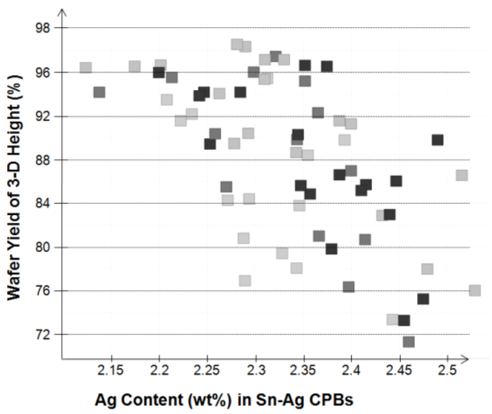


Figure 5. The relation between the yield of 3-D height and the Ag content in Sn-Ag CPBs.

Based on the classical of nucleation [23], total free energy change during solidification is written as

$$\Delta G = -\frac{4}{3}\pi r^3 \cdot \Delta G_B + 4\pi r^2 \gamma \qquad (1)$$

where ΔG_B is the free-energy change upon solidification, r is radius of nuclei in liquid phases and γ is the surface tension between solid and liquid. If the liquid is cooled below the melting point, the free energy of the solid (G_s) become smaller, and as a result ΔG_B becomes a positive number below the melting temperature. However, a surface free energy induced by nuclei in liquid phases during solidification naturally increases the total free energy change (ΔG), acting as a barrier to the formation of small nuclei. Therefore, more positive ΔG_B is needed for the phase transformation (from liquid to solid), and naturally the liquid phases are undercooled below melting point.

Ag_3Sn IMCs formed in the undercooled molten solders also follows the general nucleation theory of the equation (1). In the equation (1), the ΔG_B is the difference between liquid and Ag_3Sn IMCs, and the $4\pi r^2\gamma$ is the surface energy of Ag_3Sn nuclei. To find out the critical composition of Ag at which large Ag_3Sn plates do not form in the Sn-Ag CPBs, thermodynamic calculations were performed with two assumptions below;

1. The minimum of the freezing temperature in the Sn-Ag CPBs with less 50um is around 180°C, and it is similar values regardless the Ag content. Then, the values of ΔG_B at 180°C are calculated and compared as a function of Ag content.

2. In the equation (1), the surface free energy would be similar regardless the Ag content, and consequently ΔG is only dependent on ΔG_B.

Figure 6 is the calculated driving force of Ag_3Sn IMCs in the supersaturated liquid at 180°C as the Ag content in molten solders decreases. As expected, the driving force of Ag_3Sn nucleation decreases with decrease of the Ag content in the solders. It is interesting that as the Ag content in the Sn-Ag solders is below 2.1wt%, the driving force of Ag_3Sn IMCs changes from a positive value to negative. It means that if the Ag content of Sn-Ag solders is less than 2.1wt%, Ag_3Sn IMCs is not formed thermodynamically in the undercooled liquid at 180°C.

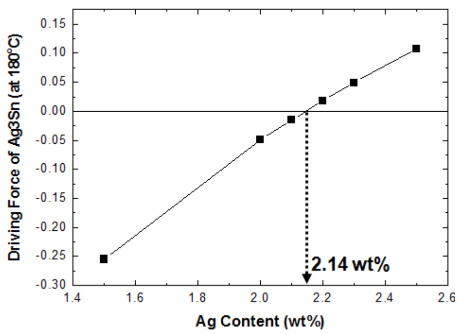


Figure 6. The calculated driving force of Ag_3Sn IMCs in the supersaturated liquid at 180°C as a function of the Ag content in the liquid.

Optimization of Ag Content in Sn-Ag Solder Bumps

Through the previous DSC results and thermodynamic calculations, the Ag content of less than 2.1wt% is suggested to retard the large plates of Ag_3Sn IMCs in Sn-Ag CPBs with less than 50um. To prove this suggestion, we controlled the content of Ag in the electroplated Sn-Ag solder at below 2.1wt%, and then verified whether the formation of Ag_3Sn plates could be actually suppressed or not.

Figure 7 is the drop of 3-D height yield for Sn-Ag CPBs in which the Ag content was controlled at 2.1wt% or lower. Actually, the yield drop was hardly observed within the maximum Ag content of 2.1wt%. Therefore, it is proved that large plate growth of Ag_3Sn IMCs is substantially reduced by controlling the Ag content of below 2.1wt% in small Sn-Ag CPBs (< 50um).

	Slot #1	Slot #2	Slot #3	Slot #4	Slot #5
Wafer Map					
Maximum of Ag Composition by XRF	1.4 wt%	1.6 wt%	1.8 wt%	1.9 wt%	2.0 wt%
Yield drop of 3-D Height	0%	0%	0%	0%	0%

Figure 7. The drop of 3-D height yield for Sn-Ag CPBs in which the Ag content was controlled within about 2.1wt% or lower.

4. Summary

The formation of large Ag_3Sn plates was investigated with small Sn-Ag CPBs (< 50um). In the small Sn-Ag CPBs, the large plate growth of Ag_3Sn was observed on the top surface of solder bumps even though the Ag content was less than 3.0wt%.

Through the DSC results, large Ag_3Sn plates in Sn-Ag CPBs with 2.3-2.5wt% Ag are attributed to the large undercooling accompanied by small volume of Sn-Ag solders. For suppression of Ag_3Sn plates in Sn-Ag CPBs, two ways were suggested; increasing the cooling rate of the reflow profile and reducing the Ag content in the solders. Reducing the Ag content was very effective on reducing the large plate growth of Ag_3Sn, while a high cooling rate was not effective.

Through the thermodynamic calculations, the Ag content of less than 2.1wt% is suggested as the optimized Ag content in small Sn-Ag CPBs, and the large plate growth of Ag_3Sn IMCs was substantially reduced within 2.1wt% Ag or lower.

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