Effect of Addition of Small Amount of Zinc on Microstructural Evolution and Thermal Shock Behavior in Low-Ag Sn–Ag–Cu Solder Joints during Thermal Cycling

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The effects of adding small amounts of Zn on the microstructural evolution of low-Ag Sn–Ag–Cu solders and thermal shock behavior during thermal cycling between −40 and 125°C were investigated. Observation of the visual appearance of the solder joints revealed that the probability of solder deformation and crack initiation in the solder fillets after thermal cycling was lower in Sn–1Ag–0.3Cu–1Zn than in Sn–3Ag–0.5Cu. Microstructural observation of the interface of the solder joints revealed that the addition of Zn to a low-Ag Sn–Ag–Cu solder inhibited the growth of the intermetallic compound layer between the solder and the Cu pad in printed circuit boards. The addition of small amounts of Zn improved the thermal shock behavior of the low-Ag Sn–Ag–Cu solder.

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1. Introduction

Eutectic Sn–Pb solder has been the most widely used material for interconnecting and packaging electronic components. However, Pb is considered toxic. Owing to concerns about the toxic effects of Pb on the natural environment and human body, several studies have focused on Pb-free solders and soldering techniques aiming at replacing Sn–Pb eutectic solders with Pb-free alternatives.1

Several compositions including Sn–Ag, Sn–Ag–Cu, Sn–Zn and Sn–Bi have emerged as candidates for Pb-free solders, and various properties such as wettability, interfacial reactions, tensile properties, and thermal fatigue have been researched.2–11 Consequently, near-ternary eutectic Sn–Ag–Cu solders such as Sn–3Ag–0.5Cu are regarded some of the most attractive candidates.12,13 Sn–3Ag–0.5Cu is the most well-known Pb-free solder worldwide, which is used in numerous electronic devices. However, the solder contains 3 mass% Ag (a precious metal), and hence, its market price is much higher than that of other lead-free solders. Moreover, fluctuations in the market price of Ag have a significant impact on the price of Sn–3Ag–0.5Cu. Sn–Ag–Cu solders with lower Ag content, referred to as low-Ag Sn–Ag–Cu solders (such as Sn–1Ag–0.7Cu), have been developed recently in order to reduce the material cost.14,15 Manufacturers are attempting to use low-Ag Sn–Ag–Cu solders in their products that are subjected to severe price competition. Since the decrease in the Ag content reduces the mechanical strength (e.g., tensile strength, creep resistance, flow stress),16–18 there are concerns regarding deterioration of joining reliability. Consequently, recent research has focused on finding a fourth element to boost the mechanical strength of low-Ag Sn–Ag–Cu.19–23

When changing the solder composition for electronic devices (e.g., replacing Sn–3Ag–0.5Cu with Sn–1Ag–0.7Cu), a thermal cycling is performed in order to evaluate the joining reliability of the printed circuit boards (PCBs) that are soldered with various electronic components. A thermal cycling involves the repeated loading of a soldered PCB at high and low temperatures to evaluate the thermal fatigue property. During thermal cycling, solder joints are subjected to the thermal stress arising from the difference in the coefficients of thermal expansion between the electronic components and the substrate. A solder alloy in solder joints is required to suppress plastic deformation caused by thermal stress, as well as to sustain the electrical continuity; however, the solder alloy starts to deform with increasing numbers of thermal cycles, resulting in the crack initiation. Cracks propagate with increasing numbers of thermal cycles, and finally, electrical disconnecting occurs in the solder joints.

When a crack starts to propagate in a solder joint, the electrical resistance of the solder joint area gradually increases. Although there is also a method for evaluating thermal fatigue properties based on changes in electrical resistance values during a thermal cycling,24,25 this method is not often adopted because of the long testing time required to change the electrical resistance values; also, there are numerous parts on PCBs; therefore, it is not possible to perform a complete evaluation. There is an alternative method widely accepted by many electronic assembly companies. First, the temperature range and number of cycles for thermal shock are determined based on the environment in which the products are used and the quality assurance term and/or the supposed life of the products, respectively, and then, thermal cycling is applied. After thermal cycling, only for the principal electronic components, visual appearance of solder joints is observed in order to verify changes of the surfaces of the solder fillets, and then, the thermal fatigue properties are evaluated from the extent of appearance change. Subsequently, microstructural observation is often carried out to confirm the growth of the intermetallic compound layers (IMC layers) of joint interface. Intense growth of the IMC layer sometimes involves the occurrence of Kirkendall voids, which results in deterioration of the joint strength; hence, it is also important to confirm the behavior of the solder joints during thermal cycling.

For many solder joints that were created using a solder with poor thermal shock behavior, such as Sn–1Ag–0.7Cu,
the original smooth, bright surface changed to one with a dull orange-peel-like appearance arising from plastic deformation in solder fillets.\(^{29}\) Cracks are often observed in solder fillets after long thermal cycling. It has been reported that plastic deformations of the solder that occur in the solder joint during thermal cycling are mainly caused by creep deformation.\(^{25}\) Since the decrease in the Ag content of Sn–Ag–Cu solders results in deterioration of the creep resistance,\(^{15}\) after thermal cycling, obvious deformation in the solder fillets is observed for Sn–1Ag–0.7Cu solder joints, and therefore improvements are urgently required.

In order to improve the thermal shock behavior of lower-Ag Sn–Ag–Cu solders, it would be effective to develop a low-Ag composition that has the same level of creep resistance as Sn–3Ag–0.5Cu. We have already developed low-Ag alloys that exhibit the same level of creep resistance as Sn–3Ag–0.5Cu,\(^{28}\) by adding a small amount of Zn to the low-Ag Sn–Ag–Cu solder. The Sn–1Ag–0.3Cu–1Zn exhibited higher flow stress than the Sn–3Ag–0.5Cu, and the flow stresses of Sn–1Ag–0.1Cu–0.4Zn and Sn–1Ag–0.3Cu–1Zn were higher than that of Sn–3Ag–0.5Cu after aging for 500 h at 125°C.\(^{29}\) Although the developed solder has a low-Ag composition, its thermal shock behavior could be considered to be equivalent to that of Sn–3Ag–0.5Cu. However, no studies have yet reported on that aspect. This research, therefore, aims to investigate the behavior of Sn–Ag–Cu–Zn solders subjected to thermal cycling and compare it with that of the Sn–3Ag–0.5Cu solder. Moreover, in order to clarify the effects of Zn addition, the microstructural evolution of the solder alloy and the joint interfaces were investigated before and after thermal cycling.

2. **Experimental Procedure**

2.1 **Preparation of specimens**

Sn–3 mass% Ag–0.5 mass% Cu, Sn–1 mass% Ag–0.7 mass% Cu, Sn–1 mass% Ag–0.1 mass% Cu–0.4 mass% Zn and Sn–1 mass% Ag–0.3 mass% Cu–1 mass% Zn solders were used in this study (hereafter, these solders are designated as SAC305, SAC107, SAC101-0.4Zn and SAC103-1Zn, respectively). The SAC305 and the SAC107 solders are recommended as Pb-free solders by the Japan Electronics and Information Technology Industries Association (JEITA). For the preparation of these solders, high-purity Sn (99.98 mass%), Ag (99.99 mass%), Cu (99.99 mass%) and Zn (99.9 mass%) were used as the starting materials. The solder billets were initially produced by the ingot casting technique. The molten solders were cast into an iron mold having a diameter 72 mm and length 200 mm. The billets were extruded into cylindrical bars of 12 mm diameter, filling an RMA (rosin mildly activated type) class\(^30\) flux, and then drawn into wire of 0.8 mm diameter. The flux-cored solder wire was cut into pieces of length 7 mm and processed into ring shape. The chemical compositions of the prepared solders are listed in Table 1.

Specimens for thermal cycling were prepared using PCBs, connectors, and ring solders. They are shown in Fig. 1. The connector leads were made from brass with electrolytic Ni plating on the brass and Sn plating on the Ni plating. The chemical composition of the brass was examined using X-ray fluorescence analysis (XRF; SEA1000A, SII). The brass consisted of 75.4 mass% Cu and 24.6 mass% Zn. The thicknesses of the Ni plating and the Sn plating were about 1 and 2 µm, respectively. Single-sided PCBs were used to reduce the amount of solder supplied to the solder joints and to increase the load to the solder joints during thermal cycling. Connector leads were inserted into the PCBs, and the ring solder was inserted into the connector leads. A light beam from a xenon lamp was used as the heating source. The light from the xenon lamp converged with a reflection mirror and the converged light was passed through an optical fiber connected to a converging lens. The spot diameter of the converged light was approximately 2 mm. The output power and irradiation time were 40 W and 2 s, respectively. After heating, solder fillets, which had smooth surfaces, were confirmed at each soldering point, as shown in Fig. 2. Connector leads were distinguished by labeling them, from left to right, as the first lead (L1), second lead (L2), and so on, to the tenth lead (L10). The flux residue was removed by ultrasonic cleaning after soldering. The labelings of the solder joints are shown in Fig. 2.

2.2 **Thermal cycling**

Thermal cycling was applied to the specimens up to 1000 cycles such that the high and low temperatures were 125 and −40°C, respectively. Each specimen was held at the
high and low temperatures for about 30 min for each, and therefore, one cycle lasted for about 60 min. In order to confirm the temperature profile of thermal cycling, a thermocouple was installed at the edge of connector lead. A typical temperature profile is shown in Fig. 3. When a change occurred from a high temperature to a low temperature, temporarily dropping to near $-45^\circ$C, the temperature then converged with the set temperature of $-40^\circ$C. When a change occurred from a low temperature to a high temperature, the profile curve was gradually converged with the set temperature of 125°C. The temperature profile indicated that specimens were subjected to an intended thermal loading.

2.3 Measurement of pull strength

When the unexpected intensive growth of IMC layers or the formation of the IMC layers with a low melting point at the interface of the solder joints occurred during thermal cycling or isothermal aging, the joint strength suddenly deteriorated. Pull strengths were therefore measured after 0, 500 and 1000 thermal cycles at room temperature as shown in Fig. 4. The pull tests were conducted for 10 leads under a pull rate of 10 mm/min using a pull testing machine (BT-085, YASUI KIKAI).

2.4 Observation of visible appearance and microstructure

All the connector leads, from the first to the tenth, were observed using an optical microscope (KH-1300, Hirox) in order to verify changes in the visible appearance that occur on the surface of the solder fillet as a result of thermal cycling. Specimens for microstructural observation were prepared by sectioning PCBs perpendicularly and then mechanically polished and finished using ion milling (E-3200, Hitachi). The microstructures of the interface of the solder joint (solder/Cu interface and solder/lead interface) and the microstructures of the solder alloys were observed using a scanning electron microscope (SEM; SSX-550, Shimadzu). A field-emission electron-probe microanalyzer (FE-EPMA; JXA-8530F, JEOL) was used to perform elementary mapping for the interface of the solder joint.

3. Results

3.1 Appearance of solder fillets

After thermal cycling, many solder fillets, which originally have smooth, bright surfaces, have a dull orange-peel-like appearance as a result of plastic deformation of the solder. Cracks are often observed on deformed solder fillets after long thermal cycling. The visual appearances of the SAC305 and SAC103-1Zn, before and after the thermal cycling, are shown in Figs. 5 and 6, respectively. Solder fillets that did not exhibit any solder deformations or cracks on the surface following the thermal shock test are classified as "A", the fillets in which solder deformations were observed only on the surface are classified as "B", and the fillets in which both solder deformations and cracks were observed on the surface are classified as "C", as shown in the Figures. The difference between "B" and "C" is whether or not there is obvious crack opening. For SAC305, solder deformations on the surfaces of the solder fillets were confirmed for the first through to the sixth and the tenth leads. Cracks, which are pointed with arrow signs, were also observed for the leads that had solder deformations. For SAC103-1Zn, deformations on the surfaces of the solder were confirmed for the first, second and
eight through to the tenth leads. Cracks were observed on the first, second and eighth, and, tenth leads. Cracks occurred in connection with solder deformations for both SAC305 and SAC103-1Zn. The solder deformation and crack initiation were mostly observed at the outer side of the connector leads for all examined solders. This is because the PCB and the connector expand and contract from their center during thermal cycling and the thermal strain as a result of the differences between the coefficients of thermal expansion is larger than that in the joints of the outer leads. The probability of solder deformation and/or crack initiation on the solder fillets for all ten leads is summarized in Table 2. Solder deformations and cracks on the solder fillets were most suppressed for SAC103-1Zn, whereas the probabilities of both solder deformation and crack initiation were 70 to 80% for SAC305, SAC107 and SAC101-0.4Zn.

### 3.2 Microstructural observation

#### 3.2.1 Microstructural evolution of solders

The microstructures of the SAC305, before and after the thermal cycling, are shown in Fig. 7. The eutectic structure of \( \beta\text{-Sn, Ag}_3\text{Sn and Cu}_6\text{Sn}_5 \) exists surrounding the \( \beta\text{-Sn phase before thermal cycling}.^{34} \) Significant coarsening occurred with the second-phase particles of \( \text{Ag}_3\text{Sn and Cu}_6\text{Sn}_5 \) in the eutectic structure after 1000 thermal cycles. Figure 8 shows the microstructures of SAC107 before and after thermal cycling. After thermal cycling, obvious coarsening of the second-phase particles is confirmed, just as in the case of SAC305. The microstructures of SAC101-0.4Zn and SAC103-1Zn are shown in Figs. 9 and 10, respectively. The \( \beta\text{-Sn phase existed more widely than SAC305 and finely dispersed second-phase particles were confirmed before thermal cycling. The second-phase particles were coarsened after 1000 thermal cycles; however, this coarsening was not as extensive as for SAC305.} \n
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<th>Probability (%)</th>
<th>Deformation</th>
<th>Crack</th>
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<td>SAC305</td>
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<td>SAC107</td>
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<td>SAC101-0.4Zn</td>
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<td>80</td>
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<td>SAC103-1Zn</td>
<td>50</td>
<td>40</td>
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<td>Before thermal cycling</td>
<td>After 1000 thermal cycles</td>
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Fig. 6 Visual appearance before and after thermal cycling for SAC103-1Zn.

Fig. 7 Microstructural evolution for SAC305: (a) shows the microstructures before the thermal cycling and (b) shows the microstructures after 1000 thermal cycles.

Fig. 8 Microstructural evolution for SAC107: (a) shows the microstructures before the thermal cycling and (b) shows the microstructures after 1000 thermal cycles.

Fig. 9 Microstructural evolution for SAC101-0.4Zn: (a) shows the microstructures before the thermal cycling and (b) shows the microstructures after 1000 thermal cycles.

Fig. 10 Microstructural evolution for SAC103-1Zn: (a) shows the microstructures before the thermal cycling and (b) shows the microstructures after 1000 thermal cycles.
3.2.2 Microstructural evolution of the interface

The microstructures of the solder/Cu interface for SAC305, SAC107, SAC101-0.4Zn and SAC103-1Zn, before and after thermal cycling, are shown in Fig. 11. The IMC layer of the solder/Cu interface for SAC305 and SAC107 had obviously thickened after 1000 thermal cycles. For SAC101-0.4Zn and SAC103-1Zn, on the other hand, growth of the IMC layer of the solder/Cu interface was suppressed in comparison with the case of SAC305 and SAC107. The microstructures of the solder/lead interface for SAC305, SAC107, SAC101-0.4Zn and SAC103-1Zn before and after the thermal cycling are shown in Fig. 12. No significant growth in the IMC layer was observed following the thermal cycling for all examined solders. The changes in thickness of the IMC layer during the thermal cycling at the interfaces of all solders are shown in Fig. 13. Obvious growth of the IMC layer was observed at the interfaces of all solders in Fig. 11. The IMC layer was observed regardless of the composition of the solder alloy. The results of an FE-EPMA analysis of the solder/Cu interface for SAC103-1Zn are shown in Fig. 14. IMC layer was composed of two regions. One layer in which Zn and Cu reacted existed on the solder side of the IMC layers, and the other layer in which Cu and Sn reacted existed on the side of the Cu on PCB. The results of an FE-EPMA analysis of the solder/lead interface for SAC103-1Zn are shown in Fig. 15. A Ni-plated layer was clearly verified. A layer in which Cu, Ni and Sn reacted was observed on the solder side of the Ni-plated layer.

3.3 Pull strength

The ranges of pull strength of each solder are shown in Table 3. There was a tendency for the pull strength to decline gradually in all the examined solders with increasing number of thermal cycles. The ranges of pull strength for SAC305 were higher than those for the other examined solders before and after thermal cycling. The appearances of the fractured solder joints for SAC305 and SAC103-1Zn, which subjected to 1000 thermal cycles, are shown in Fig. 16. For all examined solders, each connector lead was drawn out after the pull test.

4. Discussion

4.1 Relationship between probability of solder deformation and crack initiation and creep resistance

The results of this study show that solder deformations and cracks on the solder fillets were suppressed for SAC103-1Zn following 1000 thermal cycles. It has been reported that creep strain dominates the total strain of plastic deformation during
Fig. 13 Variation in thickness of intermetallic compound layer as a function of thermal cycles: solder/Cu interface (a) and solder/lead interface (b).

Fig. 14 Field-emission electron-probe analysis of solder/Cu interface for SAC103-1Zn after 1000 thermal cycles.

Fig. 15 Field-emission microprobe analysis of solder/lead interface for SAC103-1Zn after 1000 thermal cycles.

Table 3 Variation in pull strength as a function of thermal cycles for Sn–Ag–Cu and Sn–Ag–Cu–Zn solders.

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<th>Pull strength, F/N</th>
<th>SAC05</th>
<th>SAC107</th>
<th>SAC101-0.4Zn</th>
<th>SAC103-1Zn</th>
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Fig. 16 Appearances after pull test: SAC05 (a) and SAC103-1Zn (b).
It is well known that in Sn–Ag–Cu solders, the interfacial structure is a solder/Cu$_2$Sn$_5$/Cu$_6$Sn$_5$/Cu under soldering conditions and that Cu$_6$Sn$_5$ and Cu$_2$Sn layers grow during isothermal aging or thermal cycling. This is because Cu on substrate diffuses sufficiently toward the solder phase, resulting in the continuous formation of the Cu–Sn IMC layer.

Zn-containing solders such as Sn–8Zn–3Bi and Sn–9Zn have a peculiar interfacial structure unlike Sn–Ag–Cu solders. Isothermal aging has been reported to result in the formation of a layered solder/Cu$_2$Zn$_8$/Cu$_6$Sn$_5$/Cu interface structure in Sn–8Zn–3Bi and Sn–9Zn. Since these solders contained 8–9 mass% of Zn, Zn atoms diffused to the joint interfaces sufficiently during isothermal aging; consequently, the Cu$_2$Zn$_8$ IMC layer grew and the total IMC layer became progressively thicker. The interfacial structures of Sn–3 mass%Ag–0.5 mass%Cu–1 mass%Zn after isothermal aging also were reported and they are identical to those of Sn–8Zn–3Bi and Sn–9Zn; however, the growth of the IMC layers was suppressed. The suppression mechanism could be considered to be as follows: small Zn content of the solder alloy leads to insufficient diffusion of Zn to the joint interface, resulting in suppression of the growth of Cu$_2$Zn$_8$ layer during isothermal aging. Similarly, in Sn–3.8 mass%Ag–0.7 mass%Cu–0.4 mass%Zn and Sn–0.7 mass%Cu–0.4 mass%Zn, the growth of the IMC layer during isothermal aging was suppressed.

In the case of SAC103-1Zn, interfacial structure consists of the SAC103-1Zn solder/Cu–Zn layer/Cu–Sn layer/Cu after thermal cycling, as shown in Fig. 14. These results suggest that the interfacial structure of SAC103-1Zn also consisted of solder/Cu$_2$Zn$_8$/Cu$_6$Sn$_5$/Cu. The reason for the suppression of the growth of the total IMC layer for SAC103-1Zn is considered that the Zn content was low and the insufficient supply of Zn atoms to the interface.

### 4.2.2 Solder/leads interface

The thicknesses of the IMC layers between the solder/lead and the interface were less than 1 µm in all the examined solders after 1000 thermal cycles. The interfacial structure between Ni plated on a Cu pad and Sn–Ag–Cu solders has been researched; the interfacial structure consist of solder/(Cu,Ni)$_3$Sn$_5$/Ni-plating/Cu. It has also been reported that the (Cu,Ni)$_3$Sn$_5$ IMC layer suppress the diffusion of Ni toward the solder and obviously suppresses the growth of the IMC layer during isothermal aging. In Fig. 15, an IMC layer, in which Cu, Ni and Sn exist, is confirmed on the solder side of the Ni-plating layer after thermal cycling. This IMC layer is considered to be (Cu,Ni)$_3$Sn$_5$, and the layer could suppress the growth of IMC layer during thermal cycling.

### 4.3 Relationship between pull strength and thermal cycling

After the tensile tests, all the connector leads were drawn out after the pull tests for all examined solders. These results indicated that fractures occurred in the solder/lead interface and the pull strength is considered to be influenced by the microstructures of the solder/lead interface.

The interfacial structure between Ni plating on a Cu pad and Sn–Ag–Cu solders has been studied and reported that the
microstructure consists of solder/(Cu,Ni)Sn/ Ni-plating/Cu pad.46,47) The FE-EPMA analysis for SAC103-1Zn, shown in Fig. 15, indicated that an IMC layer, in which Cu, Ni and Sn exist, is confirmed on the solder side of the Ni-plating layer after 1000 thermal cycles. This result indicated that the microstructure of the solder/lead interface for SAC103-1Zn is the same as that of Sn–Ag–Cu solders. Figure 12 shows the microstructures of the solder/lead interface before and after thermal cycling. The microstructural evolution of IMC layer was not observed for all examined solders. The variation in the thickness of the IMC layer in the solder/lead interface during thermal cycling is shown in Fig. 13(b). For all examined solders, the growth of the IMC layer was suppressed during thermal cycling. The above-mentioned results indicate that the microstructures of the solder/lead interface of all the examined solders are the same before and after thermal cycling. Therefore, the pull strength is not influenced by the microstructures of the solder/lead interface.

The ranges of pull strengths of SAC305, which are listed in Table 3, were higher than those of the other solders in each thermal cycle. The factor that influences pull strength is considered to be the size of the contact area, i.e., the wetting area, between the solder and the lead. The pull strength decreases with decreasing size of the wetting area. The size of the wetting area decreases according to the deterioration of the wettability of solder alloys. It is well known that a Zn-rich solder, such as Sn–9Zn, exhibits poor wettability, arising from Zn oxidation sensitivity.48,49) Because SAC101-0.4Zn and SAC103-1Zn contain small amounts of Zn, the wettability deteriorated slightly, so the pull strength of SAC101-0.4Zn and SAC103-1Zn were lower than that of SAC305. The wettability of SAC101-0.4Zn and SAC103-1Zn can be easily improved by a flux technique because these solders contain a small amount of Zn. This issue will be discussed in future papers.

5. Conclusion

The thermal shock behavior and microstructural evolution of SAC305, SAC107, SAC101-0.4Zn and SAC103-1Zn during thermal cycling between −40 and 125°C were investigated. The results of this study are summarized as follows.

(1) SAC103-1Zn had the lowest probability of solder deformation and crack initiation after thermal cycling and the best thermal shock behavior among all the examined solders.

(2) The probability of solder deformation and crack initiation has a correlation with the flow stress of the solder alloys.

(3) The addition of a small amount of Zn suppressed the growth of IMC layers at the solder/Cu interface during thermal cycling.

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REFERENCES

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