Effects of Sb Addition on Phase Transformation and Thermal Fatigue in Sn-Ag-Bi-In Solder Joints

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Abstract
We studied the effects of adding various amounts of Sb to Sn-3.5Ag-0.5Bi-6.0In-0.8Cu (SABIC) solder in terms of the phase transformation temperature for the $\beta$-Sn and $\gamma$(InSn$_4$) transformation, mechanical properties such as strength and ductility, and thermal fatigue properties under thermal cycling ($-40^\circ$C/$175^\circ$C). With increasing amounts of added Sb, the temperature at which the phase transformation occurred increased. At 150 and 175°C, breaking elongation values of solders as a measure of ductility became the highest for an Sb content of 0.5 wt.%. As the phase transformation temperature increased, deformations of the solder joints after the thermal fatigue test were suppressed. The thermal fatigue properties were best at an Sb content of 1.0 wt.%, at which the phase transformation temperature was greater than 175°C. It is considered that raising the phase transformation temperature to a temperature higher than the maximum operating temperature of electronic devices should be the topmost priority to improve crack extension behavior.

Keywords: Thermal Fatigue Property, Sb, Sn-In Solder, Phase Transformation Temperature, Ductility

1. Introduction
The number of electronic devices used in automobiles is increasing year by year,[1] resulting in them being miniaturized so that they can be mounted in limited spaces, such as an engine room, which is a high temperature environment. Because of both the high heat density of miniaturized electronic devices and the high ambient temperature, thermal fatigue properties that enable the devices to operate at temperatures greater than 150°C are required in the future.[2] For automotive electronic devices to resist thermal fatigue during operation, it is important to ensure thermal fatigue when being packaged on circuit boards. In order to achieve this, studies are being carried out to improve heat resistance, an important property for high-temperature use of electronic components, boards/substrates, and solders used to join components. However, it is difficult to use automotive electronic devices at temperatures greater than 150°C, because the solder joints on circuit boards are not able to withstand thermal fatigue caused by cyclic temperature changes. Therefore, it is necessary to improve the thermal fatigue properties of the solder joints.

We previously studied the effects of adding Bi and In to Sn-Ag solders and reported that the Sn-3.5Ag-0.5Bi-6.0In-0.8Cu (SABIC) solder has good thermal fatigue properties and can be used in a high-temperature environment up to 150°C.[3–5] The phase transformation from $\beta$-Sn to $\gamma$ (InSn$_4$) and the reverse phase transformation, which are intrinsic properties of the Sn-In solder, affected its thermal fatigue properties. The SABIC solder had better ductility at high temperatures than other Sn-based solders, including those containing solid solution elements such as Bi or Sb. The reason for this might be that the phase of SABIC varies from $\beta$-Sn single phase to $\beta$-Sn and $\gamma$ binary phases via phase transformation with an increase in temperature.[5] It is considered that this high-temperature ductility had a positive influence on the thermal fatigue properties of solder joints.

On the other hand, excess repetition of phase and reverse phase transformation causes self-collapse of the solder due to the repeated large-volume change between the $\beta$-Sn and $\gamma$ phases during repeated temperature
changes.[6, 7] This self-collapse may have a negative influence on the thermal fatigue properties of solder joints and lead to short circuits by deformation. In addition, the phase transformation behavior influences the mechanical properties of solders. Past research has suggested that the collapse of the SABIC solder occurs as a result of repeated exposure to temperatures greater than 150°C.[3] However, few studies have focused on the mechanical properties and phase transformation behavior of the SABIC solder at temperatures greater than 150°C.[6, 7]

Therefore, the phase transformation behavior of SABIC and an increase in the phase transformation temperature should be studied to ensure good thermal fatigue properties of SABIC above 150°C. Moreover, it is important to clarify the mechanical properties of the solder.

For the above-mentioned purpose, it is necessary to find an additive element that can raise the phase transformation temperature without decreasing the In content, while also maintaining or improving mechanical properties. A calculated phase diagram indicated that the phase transformation temperature of the Sn-3.5Ag-0.5Bi-6.0In-0.8Cu solder seems likely to rise by the addition of Sb.

Therefore, we studied the effects of Sb addition on the phase transformation behavior of the SABIC solder by analyzing its phase transformation temperature and mechanical properties such as strength and ductility. We also investigated the thermal fatigue properties of the solder joints using that solder. In this paper, we report detailed results of thermal analyses, tensile tests, and thermal fatigue tests, and discuss the mechanism behind the observed thermal fatigue properties.

2. Experimental Methods

2.1 Materials

The following two solders have been used in this study: (i) SABIC and (ii) SABIC with different amounts of added Sb (0.5, 1.0, and 5.0 wt.%). These specimens have been abbreviated as SABIC and SABICXSb, respectively, where X denotes the wt.% of added Sb.

Solders were produced as follows: about 1 mm diameter metal blanks of each element were prepared and weighed as each composition dictates. Firstly, Sn metal blanks were melted at 600°C in an N₂ atmosphere in a ceramic pot. Then, the other elements were added to the melted Sn. After keeping the mixture at this temperature for 5 min, the solders were cooled to room temperature. The cooling rate was about 10°C/s.

2.2 Methods

2.2.1 Differential scanning calorimetry

The phase transformation temperatures of the β-Sn and γ phases were measured by differential scanning calorimetry (DSC, Seiko Instruments DSC6200). About 20 mg of each solder was cut and put into an aluminum pan. For the DSC measurements, the temperature was varied between 150 and 200°C at a heating rate of 10°C/min. From the obtained DSC curves, endothermic peaks at temperatures below the solidus temperature of each solder were extracted, and the phase transformation temperatures were determined by calculating the inflection points of the peaks.

2.2.2 Tensile tests

Tensile tests were carried out in order to determine the mechanical properties of each solder. The specimen for these tests was made by heating each solder at 300°C, followed by casting in a graphite template (Fig. 1). The cooling rate was about 5°C/s. After casting, the specimens were annealed at 150°C for 1 day, and the tensile tests were performed within 3 days. The testing conditions were as follows: temperatures were 25, 150, and 175°C, the strain rate was 2 × 10⁻⁴ s⁻¹, and the number of replicates was 3 for each sample type. The strain was measured using a strain gage (Kyowa Dengyo, KFR-1-120-C1-23R3M2) to 0.5% and by stroke strain up to 0.5%. After their fracture, the lengths of specimens were measured and their breaking elongations were calculated.

2.2.3 Metallographic observation

The structure of each solder was observed using a scanning electron microscope (SEM, JEOL JXA-8700) or a metallograph. The distribution of each element in the solders was observed using an electron probe X-ray microanalyzer (EPMA, JEOL JXA-8700). For SEM and EPMA observations, the SABIC and SABICXSb solders were surface polished using polishing papers from #80 to #2000, a 3.0 μm diamond polishing suspension, and a 0.05 μm alumina polishing suspension.

2.2.4 Thermal fatigue tests

Next, we studied the effect of adding Sb to the SABIC solder on its thermal fatigue properties. Figure 2 is a sch-
matic representation of a test element group (TEG) board for the thermal fatigue tests. The TEG boards were 190 mm × 125 mm × 1.2 mm in size and made of FR-5 type glass-epoxy (Panasonic, R-1755D), whose glass transition temperature (Tg) was 163°C, and their electrodes were Cu treated with an organic solderability preservative. Each test specimen was composed of a TEG board and 1005- and 3216-size chip resistors joined by means of each solder type. Table 1 shows the reflow profile of each solder. The testing cycle used was a 60-min cycle (−40°C for 30 min and 175°C for 30 min, heating and cooling time at the change of temperature was about 10 min) and the total number of cycles was 3,000.

The solders tested in these thermal fatigue experiments were SABIC, SABIC0.5Sb, SABIC1.0Sb, and Sn-3.0Ag-0.5Cu (SAC).

3. Results
3.1 Phase transformation behavior
Figure 3 shows the DSC curves of SABIC and SABICXSb from 150 to 200°C. In Fig. 3 (a), which shows the heating curve, each solder shows endothermic peaks attributed to their phase transformation and melting behavior. A slight endothermic peak, attributed to the phase transformation, can be seen at 162°C for SABIC, at 170°C for SABIC0.5Sb, and at 178°C for SABIC1.0Sb. Thus, the temperature at which the endothermic reaction occurs increased with an increase in Sb content. SABIC5.0Sb, however, does not have a clear endothermic peak. SABIC and SABIC0.5Sb each also have a large endothermic peak at about 200°C, derived from their melting behavior.

In Fig. 3 (b), which shows the cooling curve, each solder has exothermic peaks attributed to their phase transformation and solidification behavior. Slight exothermic peaks, attributed to the phase transformation, can be seen at 151°C for SABIC, at 131°C for SABIC0.5Sb, at 136°C for SABIC1.0Sb, and at 131°C for SABIC5.0Sb.

3.2 Mechanical properties
Figure 4 shows the stress-strain curves of SABIC at 25°C and 150°C. At 25°C, the 0.2% proof stress and breaking elongation of SABIC are 55.0 MPa and 31%, respectively. At 150°C, the 0.2% proof stress is 12.7 MPa, which is smaller than that at 25°C, while the breaking elongation is...
43%, which is greater than that at 25°C. Figures 5 and 6 show the 0.2% proof stress and breaking elongations, respectively, of SABIC and SABICXSb at 25°C, 150°C, and 175°C.

The 0.2% proof stress increases slightly with an increase in Sb content at 25°C, but does not change significantly with an increase in Sb content at 150 and 175°C. The largest breaking elongation, on the other hand, is observed with 0.5 wt.% added Sb, especially at 150 and 175°C. However, this value decreases when the solders contain more than 0.5 wt.% Sb.

### 3.3 Microstructure

Figure 7 shows the results of EPMA analysis. From the In images, it can be seen that In is present at the β-Sn phase, and intermetallic compounds (IMCs) that contain more In are present in all the solders. From the Sb images, it can be seen that Sb is also present, except in the IMCs (light gray in the backscattered electron (BSE) images), with the amount of distributed Sb increasing with the Sb content. In SABIC1.0Sb and SABIC5.0Sb, other IMCs (dark gray in BSE images) that contain more Sb could be observed. The dark gray IMCs contain Sb and In at a ratio of around 1, found by quantitative analysis. Therefore, it is inferred that these dark gray IMCs are composed of InSb.

From these results, it can be concluded that Sb dissolved in the β-Sn phase first; then, with an increase in Sb content, Sb also reacted with In, and InSb IMCs formed simultaneously with the dissolution of Sb in the β-Sn phase. The reason for the preferential generation of InSb may be the lower mixing enthalpy of In-Sb compared to that of Sn-Sb and Sn-In.[8]

### 3.4 Thermal fatigue properties

Figure 8 shows the optical cross-sectional images of the R3216, R1005 solder joints, as well as the surface images of the 2 mm × 2 mm electrode of SABIC, SABIC0.5Sb and SABIC1.0Sb after 3,000 cycles of the thermal fatigue test. In the solder surface views, the SABIC surface appears bumpy. In contrast, the SABIC0.5Sb and SABIC1.0Sb surfaces retain a metallic luster, which indicates the suppression of surface deformation by the phase transformation. On the cross-sectional views, it can be seen that none of the SABICXSb solder joints fractured in R3216 and R1005 resistors. In the case of SABIC1.0Sb, the crack lengths at both sizes are shorter than for SABIC. Furthermore, although the views for SAC are omitted, the SAC solder joints fractured for both sizes.

The deformation of the SABIC1.0Sb joint fillet is less than that of SABIC and SABIC0.5Sb joint fillets. Especially for R1005 joints, deformation of the SABIC1.0Sb joint fillets is almost never observed.

From these results, it is found that an increase in Sb content prevented the deformation of solder surfaces as well as the disappearance of metallic luster. Moreover, crack growth was prevented, especially for SABIC1.0Sb compared with SABIC0.5Sb.
4. Discussion

4.1 Changes in the phase transformation behavior

As shown in Fig. 3, the phase transformation temperature of SABICXSb increased with an increase in Sb content. This result can be explained as discussed below.

Figure 9 shows the SABIC-Sb phase diagram calculated with the CALPHAD method[9, 10] using Pandat5.0 software. In Fig. 9, the border temperature of the mixing area of $\beta$-Sn and $\beta$-Sn/$\gamma$ phases, shown by a thick red dotted line, increased with an increase in Sb content. The phase
transformation temperatures obtained from the DSC curves in Fig. 3 are indicated by the large red dots and red dashed lines. It can be seen that the gradients of the line connecting the dots and the border line are similar. Therefore, it appears that the free energy of SABICXSb at higher temperatures is more stable in the β-Sn phase with an increase in Sb content, and the phase transformation temperature of SABICXSb increased because of the lower mixing enthalpy of In-Sb.

On the other hand, the temperatures obtained from the DSC curves are different from the border temperatures. The temperatures obtained from the DSC curves might not represent the starting temperatures of the phase transformation, but instead the temperatures at which the phase transformation had already progressed to a significant degree. In addition, the temperatures obtained from the DSC curves may have been affected by the heating rate, and the phase diagram, as opposed to the DSC data, represents equilibrium-state phases.

4.2 Changes in mechanical properties with Sb addition

We hypothesized that the solid solution and generation of InSb IMCs affected the 0.2% proof stress of the solders. Figure 7 shows that the amount of solid-dissolved Sb increased with an increase in Sb content, and that the amount of InSb IMCs also increased at ≥ 1.0 wt.% Sb. Therefore, the 0.2% proof stress of SABIC and SABICXSb increased as a result of the effects of solid solution formation and precipitation.

The reason why the 0.2% proof stress did not increase drastically upon Sb addition may be as follows: the amount of solid solute Sb was lower than that of In, and thus its effect was smaller than that of In. Moreover, IMCs composed of Ag, In, and Sn, as well as of Cu6Sn5, existed in the SABIC solder, and thus the effect of InSb precipitation, which occurred upon Sb addition, was not noticeable.

In a previous study,[4] we reported an increase in breaking elongation of the Sn-3.5Ag-0.5Bi-6.0In (SABI) solder, which was similar to SABIC, at high temperatures below 150°C. SABI exhibited an increase in breaking elongation with an increase in temperature, similar to that observed with SABICXSb. In that report, we also showed that the microstructure of SABI after tensile tests at 150°C, in spite of high breaking elongation, exhibited slight grain deformation, indicating the effect of grain boundary sliding on the elongation mechanism.

Figure 10 shows the SABIC0.5Sb microstructures before and after tensile testing at 150°C. In the microstructure after tensile testing, significant grain deformation can not be seen, despite the large breaking elongation. Therefore, grain boundary sliding may also have affected the elongation of SABICXSb. We therefore considered the mechanism of grain boundary sliding in terms of the metallographic structure and analyzed the crystallite orientation of SABIC, SABIC0.5Sb, and SABIC1.0Sb samples by measuring their electron backscatter diffraction (EBSD) patterns. Figure 11 shows the inverse pole figure (IPF) maps (obtained using EBSD patterns) of the solders prepared by the procedure described in section 2.2.2. From Fig. 11 (a), it can be seen that the crystal size of SABIC was relatively large. On the other hand, SABIC0.5Sb and SABIC1.0Sb crystal orientations, shown in Fig. 11 (b) and (c), were more inhomogeneous, and the grain sizes of SABIC0.5Sb and SABIC1.0Sb were smaller than that of SABIC.

We therefore consider the following hypothesis for the
Deformation by grain boundary sliding tends to occur for small grain sizes\cite{11} and more inhomogeneous crystallite orientations.\cite{12} It is said that the grain boundary diffusion is the dominant factor affecting grain boundary sliding in the case of high temperatures and low-stress environments.\cite{13} Grain boundary sliding may have been facilitated by the effects of grain boundary diffusion with the miniaturization of grains. Furthermore, it has been reported that grain boundary diffusion is facilitated by greater grain boundary energy if the crystallite orientation is more inhomogeneous.\cite{14, 15} Therefore, the grain boundary sliding might be more significant in SABIC\textsubscript{X}Sb, which has a smaller grain size and a more inhomogeneous crystallite orientation than SABIC.

The breaking elongation of SABIC\textsubscript{1.0}Sb, on the other hand, is smaller than that of SABIC\textsubscript{0.5}Sb. Figure 12 shows the BSE image of the SABIC\textsubscript{1.0}Sb cross-sectional microstructure. InSb IMCs, which might prevent grain boundary sliding, were generated at the grain boundary.

The effect of grain boundary sliding was evaluated with the strain rate sensitivity exponent $m$ as shown in equation (1):\cite{16–18}

$$m = \frac{\partial \ln \sigma}{\partial \ln \dot{e}}$$
where $\sigma$ is the steady stress during strain deformation and $\dot{\varepsilon}$ is the strain rate. In order to get a rough estimate for the $m$ value of SABIC and SABIC0.5Sb, we carried out tensile tests at 150°C, after which we calculated $m$ values of $2 \times 10^{-3}$ s$^{-1}$ and $2 \times 10^{-4}$ s$^{-1}$, respectively. The $m$ values of SABIC and SABIC0.5Sb calculated from the above tensile tests were 0.11 and 0.18, respectively, indicating that the effect of grain boundary sliding is more significant for SABIC0.5Sb than for SABIC. However, for a more in-depth discussion on the effects of $m$ on grain boundary sliding, it is necessary to evaluate its values more precisely. This remains to be solved.

4.3 Changes in microstructures with Sb addition

In this section, we discuss the mechanism behind grain miniaturization of SABICXSb upon Sb addition. Figure 13 shows a schematic of the solidification process of SABIC and SABIC0.5Sb, based on Fig. 9. Focusing on the $\beta$-Sn and $\gamma$ phases, in the case of SABIC, the $\beta$-Sn phase is generated first, and only the $\gamma$ phase is present at the solidus temperature after the phase transformation from the $\beta$-Sn to $\gamma$ phase. After solidification, the $\gamma$ phase undergoes phase transformation to the $\beta$-Sn phase upon cooling, and the $\gamma$ phase disappears, leaving behind only the $\beta$-Sn phase at room temperature.

In the case of SABIC0.5Sb, a mixture of the $\beta$-Sn and $\gamma$ phases exists at the solidus temperature. At room temperature, however, SABIC0.5Sb consists of only the $\beta$-Sn phase, as observed with SABIC.

Grain miniaturization upon phase transformation has been reported previously,[19] and we consider that the same mechanism existed in the case of SABICXSb. For SABICXSb, the $\gamma$ phase was generated in addition to the $\beta$-Sn phase until solidification, thus preventing the $\beta$-Sn phase from coarsening. In addition, precipitation nuclei that become the origin of solidification may have been generated.

The exothermic peak of SABIC in Fig. 3 (b) may represent the phase transformation temperature from the $\gamma$ phase to the $\gamma$ and $\beta$-Sn phases, while the exothermic peaks of SABIC0.5Sb and SABIC1.0Sb may represent the phase transformation temperature from the $\gamma$ and $\beta$-Sn phases to the $\beta$-Sn phase. Therefore, the exothermic energy of SABIC is greater than that of SABIC0.5Sb and SABIC1.0Sb.

4.4 Influence of added Sb on thermal fatigue property

As shown in Fig. 8, an increase in the amount of Sb prevented the solder surface and joint fillets of R1005 from deformation, while preserving the metallic luster. This result can be explained by the increase in phase transformation temperature. Since the onset temperature of the phase transformation increased with an increase in Sb content, the degree of repeated volume changes for the duration of thermal cycling decreased. Therefore, the deformation of the surface and joint fillets was suppressed, preventing short-circuiting of the printed circuits. Nonetheless, a slight deformation was observed for R3216 due to increased thermal strain as a result of the larger size of R3216 compared to R1005. However, additional discussion about the underlying mechanisms is needed.

The crack growth of the SABIC1.0Sb solder joints was suppressed compared with SABIC. However, the ductility and strength of SABIC1.0Sb and SABIC, which influence crack extension behavior, were not very different. Therefore, we considered that the phase transformation temperature may have influenced the crack extension behavior. In the high-temperature region of the thermal fatigue test, the phase transformation causes an improvement in ductility. If excessive phase transformation occurs due to a low phase transformation temperature, extra deformation by large volume changes may cause crack extension, which could decrease thermal fatigue life.

Nonetheless, the crack extension behavior of SABIC0.5Sb was not much suppressed, despite SABIC0.5Sb exhibiting good ductility at high temperatures. Therefore, increasing the phase transformation temp...
perature of SABICXSb to temperatures higher than the maximum operating temperature of electronic devices should be the topmost priority for improving its crack extension behavior. We also believe that improvements in mechanical properties such as ductility and strength are needed to achieve even better thermal fatigue properties. Finally, the $T_g$ of the TEG board may have affected thermal fatigue behavior, because the maximum temperature of the thermal test exceeded this $T_g$. However, the influence of $T_g$ needs to be investigated further, since there currently is insufficient evidence to discuss its effects.

5. Conclusions

We here studied the phase transformation mechanism and mechanical properties of Sn-3.5Ag-0.5Bi-6.0In-0.8Cu (SABIC) and SABIC with different amounts of added Sb (SABICXSb, with X indicating the wt.% of added Sb) in order to create a new solder that can be adapted for use in high-temperature environments (> 150°C). The results were as follows:

(1) The addition of Sb to the SABIC solder increased the phase transformation temperature, with that of SABIC1.0Sb being greater than 175°C.

(2) The breaking elongation of SABICXSb at temperatures above 150°C changed with the Sb content, and SABIC0.5Sb had the greatest ductility. In contrast, the 0.2% proof stress of SABICXSb was not sensitive to Sb content.

(3) The miniaturization of SABICXSb grains upon Sb addition was observed with electron backscatter diffraction (EBSD) measurements. This miniaturization may have promoted an increase in grain boundary sliding, thereby possibly increasing the breaking elongation of SABIC0.5Sb.

(4) The thermal fatigue of SABIC1.0Sb, which had a phase transformation temperature above 175°C, was superior to that of SABIC0.5Sb, which had a phase transformation temperature below 175°C and greater ductility than SABIC1.0Sb. Therefore, increasing the phase transformation temperature of SABICXSb to a temperature higher than the maximum operating temperature of electronic devices should be the topmost priority to improve crack extension behavior.

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