Characterization of Electrical Current Stress and Indentation Creep of Carbon Nanotubes-reinforced Low Melting Temperature Sn-58Bi Composites

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Abstract. Sn-58Bi eutectic alloy emerges as one of the potential lead-free solder material candidates due to its low melting temperature that enables low temperature soldering reflow process during the manufacturing of fine-pitch and low z-height advanced microelectronic devices. Its low melting temperature provides feasible solution for a two-step reflow process in the assembly of 3D-stacked advanced packaging technology, and at the same time reduces dynamic warpage-induced quality and reliability issues. However, miniaturization of these microelectronic devices has triggered significant reliability risk to its solder interconnects due to the requirement of high current density that resulted in severe physical failures, such as electromigration (EM), and thermomigration (TM). To mitigate the risk, carbon nanotubes (CNT) are incorporated into elemental Sn and Bi powders using planetary ball milling technique, and subject to liquid phase melting to form low melting temperature CNT-reinforced eutectic Sn-58Bi composites. Electrical resistance and current density of the eutectic alloy and its composites were investigated, and results show that Sn-58Bi-0.03CNT composite has the lowest electrical resistance and was able to survive 26 times longer duration of current stressing, even with the supply of higher stressed current. Creep performance of eutectic Sn-58Bi system was also examined in this study due to its high homologous temperature of 0.72 at room temperature. Marginal improvement in indentation creep performance was seen in the composites.

1 Introduction

Under the Restriction of Hazardous Substrates (RoHS), the use of lead (Pb) has been prohibited in the electronic packaging since July 2006 [1]. Alternative solders based on Sn-Ag-Cu lead-free materials were an immediate replacement of conventional Sn-Pb solders since then. While adverse reliability impacts were reported in these lead-free solder joints [2-3], its higher melting temperature (T_m~217°C) requires higher soldering reflow temperature, as compared with that of eutectic Pb-Sn solder (T_m~183°C). This translates to a higher risk of thermally induced damages in the microelectronic devices, such as moisture...
escaped related “pop-corning” phenomenon, as well as dynamic warpage of the polymer substrate [4].

In fact, recent development of coreless technology in polymer package substrate for meeting the performance demand in advanced microelectronic device has further increased dynamic warpage risk [5]. Kim et. al. [5] has demonstrated the correlation between dynamic warpage behaviour and the soldering reflow temperature of a typical flip-chip packaging technology. In this case, the warpage of the component was the highest at the temperature range of around 240°C that used for conventional lead-free solders metallurgy. Based on this learning, it highlights the opportunity to innovatively reduce the dynamic warpage of the electronic device through an introduction of low melting temperature solder metallurgy that requires a lower soldering reflow temperature. While Indium is extremely expensive, the use of eutectic Sn-58Bi alloy appears to be a promising candidate based on its low eutectic temperature of 139°C. However, it has been reported that the addition of Bi in Sn-3.7Ag solder resulted in reduction of its elongation and creep properties [6-7]. This signifies the concern of using eutectic Sn-58Bi alloy in high performance microelectronic devices, as high homologous temperature (T/Tm > 0.5) at service environment may cause significant degradation to its creep performance.

Furthermore, the trend with continued pursuit of miniaturization and improved performance of microelectronic devices also triggered the requirement of higher input/output (I/O) density, thus smaller solder interconnects with increased reliability risk is anticipated. The increment of current density through solder interconnections could easily result in EM [8,9]. This could lead to the bottleneck in carrying high current density due to several physical failures including EM and TM. According to International Technology Roadmap for Semiconductors 2007 (ITRS 2007) [10], EM is considered an upper constrain of high current density packages in wafer-level packaging (WLP) for Microelectromechanical systems (MEMS). In eutectic Sn-58Bi solder system, mass accumulation of Bi in eutectic Sn-58Bi was observed in an accelerated test of EM when it is under current density of 5×10^3 A/cm^2 at 75°C [8]. This results in Bi segregation-induced brittle behaviour at intermetallic compound of solder interconnections [11].

To address the above concerns, multi-walled CNT are incorporated into Sn and Bi elemental powders using planetary ball milling technique, and subject to liquid phase melting to form low melting temperature CNT-reinforced eutectic Sn-58Bi composites. While CNT are known to possess superior Young’s modulus and electrical conductivity [12], it is the objective of the present study to characterize the effect of CNT reinforcement on electrical current density and indentation creep performance of low melting temperature CNT-reinforced Sn-58Bi composites.

2 Experimental details

2.1 Sample preparation

Based on the established method that used by Dele-Afolabi et al. for the mixing of CNT-reinforced Sn-5Sb solder composites [13], planetary ball milling technique was carried out at 400rpm with ball-to-powder ratio of 20:1 for up to 2 hours to prepare the mixture of Sn-58Bi, Sn-58Bi-0.01CNT, and Sn-58Bi-0.03CNT from high purity (99.9% purity) Sn and Bi elemental powders, and multi-walled CNT with an average diameter of ~26nm. Figure 1 shows the micrographs and energy dispersive x-ray spectrum of as-received Sn, Bi and multi-walled CNT. The mixtures were then melted in a crucible at 300°C with the addition of soldering flux to form eutectic Sn-58Bi monolithic solder alloy, 0.01wt.%CNT-reinforced Sn-58Bi and 0.03wt.%CNT-reinforced Sn-58Bi composites, Figure 2.
2.2 Electrical current stressing

The V-shape solder joint technique that developed by Yue et al. [14] for detecting electromigration under direct current stressing of up to $1.5 \times 10^4\ A/cm^2$ was referred, but the fabrication technique and the area determination for the definition of current density were
not clearly defined. Hence, an improved alternative experimental setup, together with appropriate design of test coupon was carried out in this investigation. Figure 3 shows the design of the test coupon, whereby a copper trace of 0.03mm thick x 0.3mm width x 20mm length was printed on a single layer of flame retardant graded 4 (FR4) printed circuit board (PCB) with two copper pads at both ends. A hole of 0.4mm diameter was drilled through the center of the trace to electrically isolate the trace so that solder sample could be melted to form a conductive solder bridge between the traces.

![Diagram of test coupon](image1)

**Fig. 3.** Test coupon for electrical current stressing experiment; (a) the design of copper trace with two pads at both ends and a hole of 0.4mm diameter at the center of the trace; (b) section A-A of the copper trace.

Figure 4(a) shows the actual experimental setup of the test coupon where the pads of the copper trace were soldered with American Wire Gauge (AWG) 26 copper wire using Sn-0.7Cu solder to connect to the DC power supply (GW INSTEK GPS-3303) via crocodile clips. Sn-0.7Cu solder of higher melting temperature ($T_m=227^\circ C$) was selected in this case to ensure the failure location for the test sample would occur at low melting temperature eutectic Sn-58Bi solder bridge, instead of at solder pad connection. 1mg of solder sample was then transferred to the hole and reflowed under a soldering rework station with the presence of soldering flux to form the solder bridge in order to complete a close-loop circuit. The nature of surface tension of molten solder bridge went through self-alignment to form a controlled shape, Figure 4(b).

![Actual experimental setup and SEM micrograph](image2)

**Fig. 4.** (a) Actual experimental setup of the test coupon where the pads were soldered with copper wire and connected to a DC power supply via crocodile clips; (b) SEM micrograph of the solder bridge (i.e. solder sample) of self-aligned shape due to its surface tension at molten stage.

### 2.3 Indentation creep test

Using a Vickers hardness tester, the hardness indentation was made on the polished samples at room temperature. The indentation was made with a pyramid indenter under a
constant load of 10N. Both the hardness value and the diagonal length of the impression were recorded for each dwell time. In this study, the dwell time of the indentation was taken as a parameter, and its variation was made within a range of 10s to 60s. For each sample, a minimum of four indentations were taken, and the distance between adjacent indentations were at least 2.5 times the diagonal length of the impression, in accordance to BS 6286: 1982 [15].

3 Results and discussion

The electrical current response for test coupons of various solder samples were plotted against voltages, as demonstrated in Figure 5(a). Based on the gradient, electrical resistance of 0.138Ω, 0.135Ω, and 0.132Ω were found on eutectic Sn-58Bi monolithic alloy, Sn-58Bi-0.01CNT, and Sn-58Bi-0.03CNT composites, respectively. From the trend, the incorporation of CNT into Sn-58Bi solder alloy resulted in lower electrical resistance, hence improving the conductivity of composites solder bridge by allowing more current to pass through it at constant voltage. This result matches the finding that obtained in other study [8].

Fig. 5. (a) Voltage against current of eutectic Sn-58Bi monolithic alloy, Sn-58Bi-0.01CNT composite, and Sn-58Bi-0.03CNT composite; (b) Time taken for samples to fail under constant electrical current supply.

Based on the reference data that obtained from Ye et al. [16] and Yue et al. [14] on different lead-free solder system, the test coupon of eutectic Sn-58Bi solder bridge was initially stressed with a current of 1.5A for up to 30 minutes, but the solder remained intact. To accelerate the failure, 2.5A of current was supplied (i.e. equivalent to current density of 2.78×10⁴ A/cm²) and failure was observed on eutectic Sn-58Bi solder bridge shortly after 55s, but not for the composites. To further accelerate the failure of the composites, 3.24A of current, which equivalent to current density of 3.6×10⁴ A/cm², was supplied to Sn-58Bi-0.01CNT and Sn-58Bi-0.03CNT composites solder bridges. Results show that Sn-58Bi-0.01CNT composite fails after 330s of stressed duration, whereas Sn-58Bi-0.03CNT fails after 1440s, as illustrated in Figure 5(b). Even with higher magnitude of the supplied current, Sn-58Bi-0.03CNT composite clearly showed significant improvement in its capacity in carrying high current density of 26 times longer duration than its monolithic alloy.

As for the indentation creep performance investigation, indentation length was found to increase with dwell time for all solder materials tested, Figure 6(a). By comparing with the monolithic alloy, the composites exhibit lower indentation length. Based on power law
relationship that applied for an indentation creep of a solid [17], for each dwell time, the same set of data in Figure 6(a) was plotted into indentation creep rate, as illustrated in Figure 6(b). In this figure, it is noted that for all the materials, the indentation creep rate decreases rapidly initially, and the rate of reduction trending down substantially with further increased in dwell time. This indicates the occurrence of primary creep in the initial stage where higher indentation creep rate is expected, and the indentation creep rate reduces subsequently to suggest a steady-state creep. This observation is in good agreement with that observed by Zaki et.al. [18]. By focusing on steady-state creep, marginal improvement in indentation creep rate was observed on the composites with 0.01wt% CNT and 0.03wt% CNT reinforcement, as compared with its eutectic Sn-58Bi monolithic alloy.

![Fig. 6](image)

**Fig. 6.** (a) Indentation length increases with dwell time; (b) Indentation creep rate was higher in the monolithic alloy than that of the composites.

### 4 Conclusions

Planetary ball milling and liquid phase melting techniques were used to synthesize low melting temperature eutectic Sn-58Bi solder alloy and its CNT-reinforced composites. Using in-house developed test coupon and experimental setup, the presence of CNT in eutectic Sn-58Bi solder alloy was found to improve its electrical conductivity, and its capacity to withstand higher electrical current density. In addition, marginal improvement in indentation creep rate was also seen in CNT-reinforced eutectic Sn-58Bi composites.

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