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Wettability and strength of In–Bi–Sn lead-free solder alloy on copper substrate

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ABSTRACT

This work studied the surface and interfacial properties of a proposed lead-free solder material, the In–31.6Bi–19.6Sn system. Surface tension and contact angle of In–31.6Bi–19.6Sn lead-free solder with melting temperature of 61.33 °C was measured on copper substrate at different reflow temperatures. Sessile drop measurements showed that the contact angle depended on reflow temperature. The contact angle gradually decreased from 38.34° to 17.25° as reflow temperature increased from 80 to 140 °C. Energy-dispersive X-ray analysis indicated two layers of intermetallic compound between the solder and the Cu substrate: one of Cu_6Sn_5 and $Cu_{11}In_9$ (scallop shaped) and the other of $Cu_{11}In_9$ (brightly coloured). As the reflow temperature increased, the shear strength of the In–31.6Bi–19.6Sn/Cu solder joint improved due to reduced contact angle and larger spreading area.

1. Introduction

In modern microelectronic technology, soldering continues to play an important role. Manko [1] defines soldering as a metallurgical joining method using solder with a melting point of below 315 °C as a filler. To achieve this, wetting of the solder on a base metal (or substrate) is essential to ensure that metallurgical bonding is formed, ideally, without formation of intermetallic compound (IMC). Wetting refers to the capacity of molten solder to react with a substrate, at the interface of solder and substrate, to form a certain amount of intermetallic compound that acts as an adhesion layer to join the solder and the substrate [2]. The reaction between the solder and substrate is important as it may affect the microstructure and eventually the mechanical strength of the solder joint [3]. The extent of wetting is measured by the spreading area and the contact angle that is formed at the juncture of a solid and a liquid in a particular environment [4]. The Young equation has been used to determine contact angles and the balance of surface tension at the juncture [4].

Tin-lead (Sn–Pb) solders are widely employed in microelectronic industries due to their superior wetting properties, moderate melting temperature and low cost. However, it is widely known that solder based on Pb is toxic to health and the environment. Therefore, an alternative solder free from Pb needs to be developed [5–9]. Sn–Ag solder alloy (221 °C) and Sn–Ag–Cu solder alloy (217 °C) have been investigated as replacements [10–15]. However, despite the general acceptance of these solders as the leading Pb-free solders, they do have a number of disadvantages, including a relatively high melting temperature. A higher melting point leads to a 20–30 °C increase in the peak reflow temperature required for assembly compared to that required for Pb-Sn eutectic solder. Such an increased temperature is detrimental to many microelectronic components, making assembly very difficult.

Another alternative is Sn–Zn solder alloy (Zn about 9 wt%) with a melting point of about 198 °C [16–19]. This alloy offers significant benefits in terms of cost and the good mechanical properties on Sn–Zn solder alloy [16,17]. However, Sn–Zn alloys suffer from low oxidation resistance and poor wettability [16].

Despite development of Sn–Ag, Sn–Ag–Cu and Sn–Zn solders, their properties still are not good enough for these alloys to be employed in temperature-sensitive components, optoelectronics modules, step soldering processes and thin printed wiring boards (PWBs) [20], which require a relatively low melting temperature. Low temperature soldering is necessary when electronic devices to be soldered are prone to thermal damage. Low temperature soldering can also reduce the risk of thermal shock induced by the thermal expansion mismatch among different materials in an electronic package.

Step soldering, another application for low temperature solders, is commonly used when soldering a device requires more than one step. In this application the solder used for subsequent step(s) should have a lower melting point than that used for the preceding step [21]. The alloying element employed to lower the melting temperature of any solder alloy must conform to the Restriction

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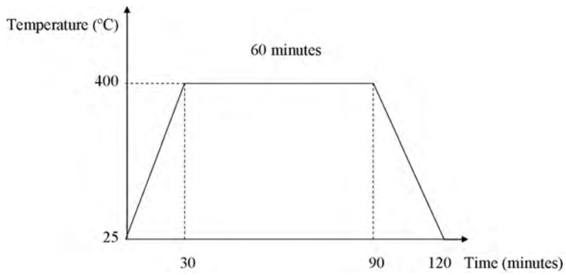

Fig. 1. Typical temperature profile for preparing the ingot.

of Hazardous Substances (RoHS) directive of the Europe Union and have a low melting point, as do bismuth (Bi), gallium (Ga), and indium (In). According to Kanlayasiri and Ariga [22], Bi and In can be employed to lower solidus and liquidus temperatures of solder alloys. Therefore in the past several years a number of studies have been carried out on the binary, ternary and quaternary systems of low temperature soldering such as Sn3.13Ag0.74CuIn [23], Sn–Zn–Bi [24,25], Bi.22In [26], Sn–Zn [17–19,27,28], Sn–Zn–*x*Bi [29], and Bi–In–Zn [30]. Although lead-free solder has been extensively studied, information about the In–31.6Bi–19.6Sn solder alloy is not yet available in the literature.

Therefore, a new solder with relatively low melting temperature, good wettability and good shear strength needs to be developed. This work proposes In–31.6Bi–19.6Sn as a candidate for a good low temperature lead-free solder alloy. Since this solder alloy has low liquidus temperature, it may be used for low temperature soldering. Furthermore, its low liquidus temperature suits it for use as an organic-based transistor such as liquid crystal display (145–178 °C), in the new generation of nano silicon chips (100–300 °C), and in polymeric conductive boards (140–400 °C). Using this alloy, outer space nano satellites to be used under cryogenic conditions (−147 to 447 °C) can be manufactured at low temperature. This paper reports the results of an investigation of the microstructural features and wettability performance of In–31.6Bi–19.6Sn solder on Cu substrate for different reflow temperatures. Results show that this ternary solder fulfills two of the requirements; microstructural and wettability results on Cu substrate for different reflow temperature of the ideal solder. Since the technology relies on the formation of intermetallic compounds, it is important to identify microstructural characteristics during joining and their influence on the mechanical properties of the micro-joints.

2. Experimental procedures

In–31.6Bi–19.6Sn solder was prepared from indium (99.9% pure, Alfa Aesar), bismuth (99.9% pure, Strem Chemicals), and tin (99.9% pure, Malaysia Smelting Corporation). These elements were melted in a furnace at 400 °C before being cast in a

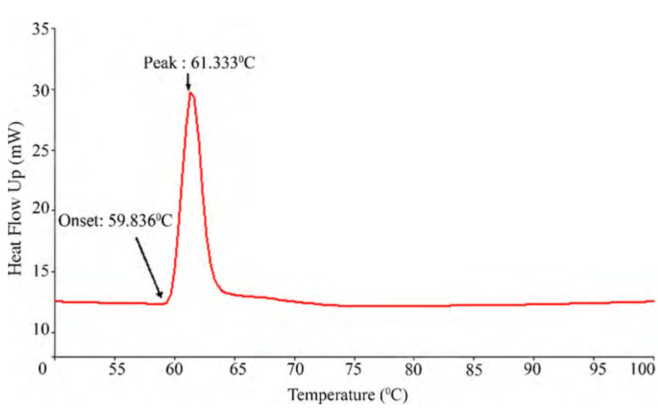

Fig. 2. Differential scanning calorimetry analysis (DSC) profile of In–30Bi–20Sn lead-free solder system.

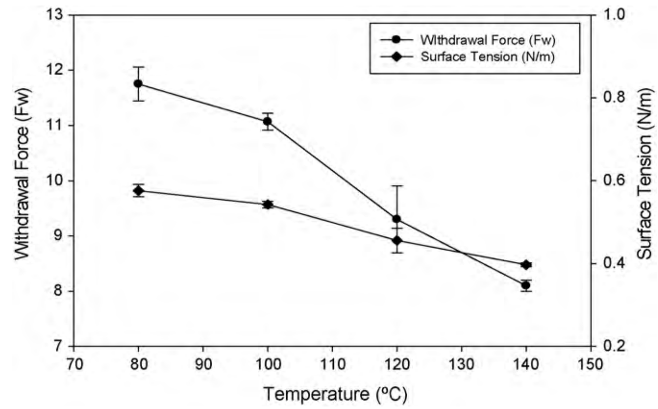

Fig. 3. Surface tension and withdrawal force (F_W) for In–30Bi–20Sn lead-free solder on Cu substrate at different reflow temperatures.

cylindrical steel mould to form the solder. The size of the ingot was 59.0 mm^3; the temperature profile for preparing the ingot is shown in Fig. 1. To obtain a homogeneous composition, the ingot was re-melted three times. Cooling was done in a similar environment for all samples. The cooling rate was controlled by two thermocouples, the first at the furnace to control furnace temperature and the second inside the solder to control its internal temperature. Both thermocouples were used to measure and control the temperature; temperature readings were recorded. In this way, the solder was controlled at a constant rate without environmental effects, and the solder samples could be prepared from the same ingot. As the cooling rate was held constant, it would not affect the microstructure and properties of the solder alloys. The ingot was then mechanically cleaned by scratching. The ingot was then rinsed with pure ethanol in an ultrasonic bath for 5 min.

A sample was taken from the ingot of the solder alloy for DSC (differential scanning calorimetry) measurement. DSC measurement with a heating rate of 10 °C/min under a nitrogen atmosphere was used to determine the liquidus temperature of the sample.

0.01-mm thick copper plates (Mitsui Mining and Smelting Co., Ltd, Japan) were used as substrates for the wetting balance test. Prior to the tests, the substrates were polished using 1 μm fine alumina powder, degreased in water for 1 min, ultrasonically cleaned in ethanol and then dried.

The surface tension of the copper substrate was measured using a wetting balance (solder checker; SAT-5100 Rhesca) in accordance with EIA-ET-740 standards. Temperatures of the solder checker were set at 80, 100, 120 and 140 °C. For each temperature, the In–31.6Bi–19.6Sn ingot was heated in a crucible until it melted and achieved setting temperature. During the test, the ingot was placed in a crucible to maintain the axial symmetry of the liquid metal drop. Prior to the test, a fixed amount of flux was applied onto the copper substrate. Then the substrate was dipped in liquid solder for 30 s.

Three of the most commonly used wetting indicators are wetting time (T_0), maximum wetting force (F_{max}) and withdrawal force (F_W) [20]. In this study, wetting force was measured as a function of time. The data acquisition was performed by a SAT/WET Data Analysis System program.

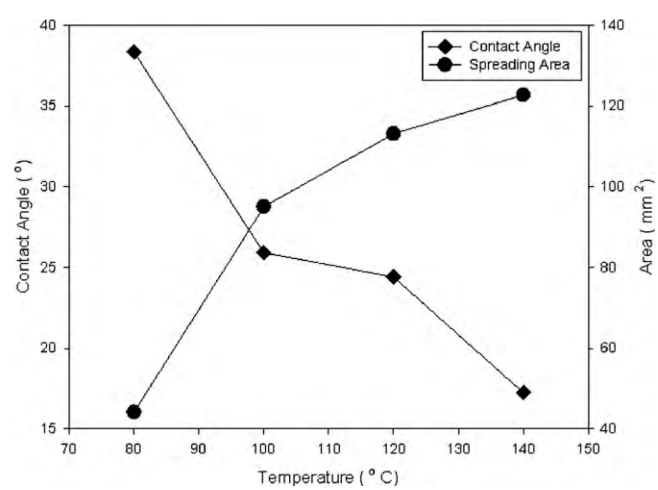

Fig. 4. Contact angle and spreading area for In–30Bi–20Sn lead-free solder on Cu substrate at different reflow temperatures.

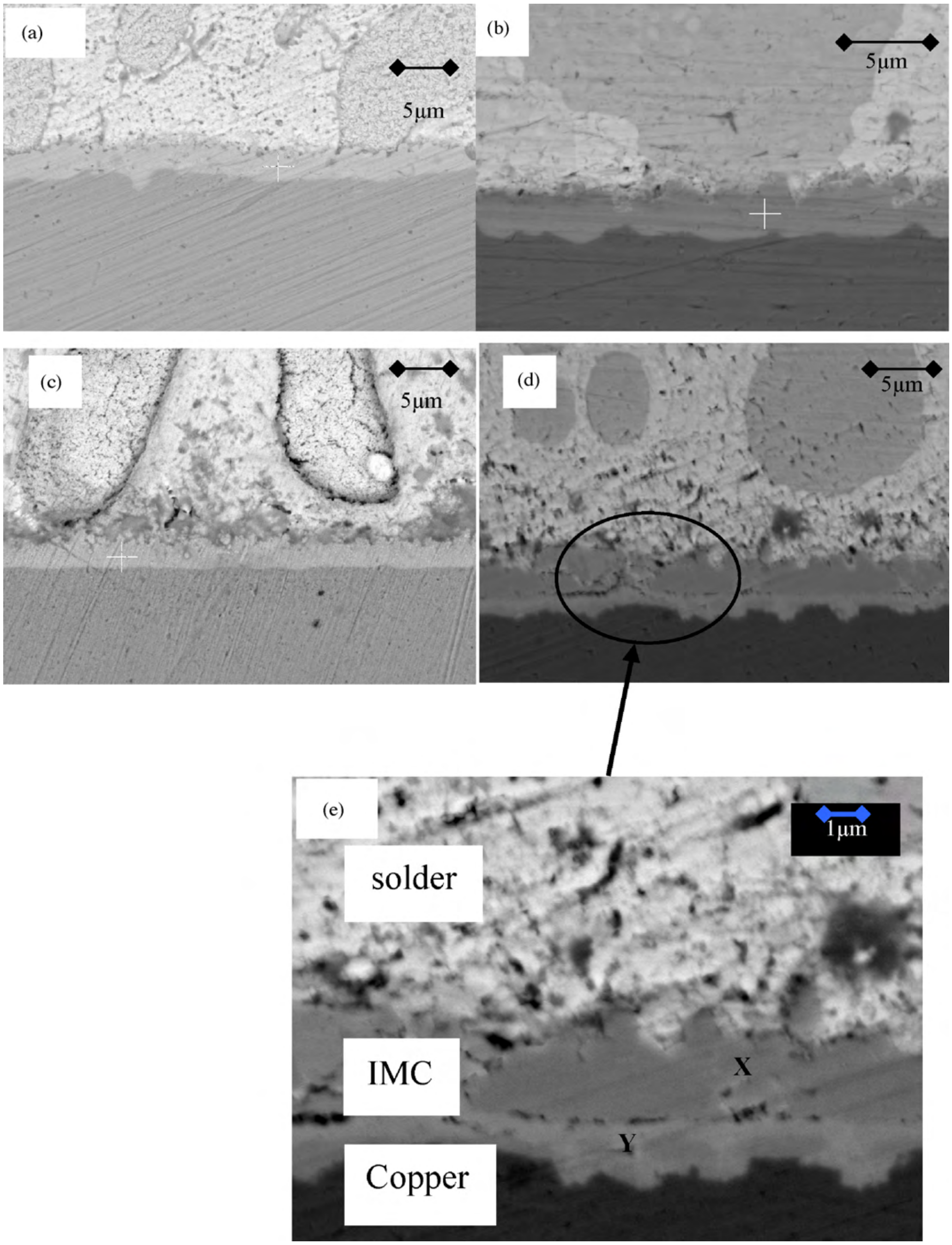

Fig. 5. SEM backscattered images at different reflow temperatures: (a) 80 °C, (b) 100 °C, (c) 120 °C, (d) 140 °C at 3000× magnification and (e) 140 °C reflow temperature at 5000× magnification.

Wetting behavior of In–31.6Bi–19.6Sn alloy was investigated using pure copper sheet (99.9%) as the substrate. The solder volume was fixed as 59.0 mm^3 and a drop of $ZnCl_2$ flux was used in this research before testing by the sessile drop method. The wetting test was conducted at 80, 100, 120 and 140 °C reflow temperature. Reflow time for the wetting test was fixed at 30 s. Specimens were mounted in epoxy resin, cross-sectioned, mechanically ground and polished using 1.0 μm Al_2O_3 to observe the interface between the solder and substrate. The wetting angle of the solder on substrate was determined by measuring the contact angle. Areas of spreading and contact angle were measured by an optical microscope with the aid of analytical software (Visver 2.9 (Plus) at 0.6× magnification).

To measure the spreading area, the diameter of the solder on the copper plate after the spreading test was measured. In order to use the analytical software, a scale needs to be defined so that measurement results can be presented in calibrated units, such as millimetres. In order to set the scale, the straight line selection tool was used to make a line selection that corresponds to a known distance. The spreading area was calculated using the software-measured diameter.

The morphology of the interface between solder and substrate were examined using a scanning electron microscope (SEM) equipped with an energy-dispersive X-ray (EDX). IMC thickness at the interface between solders and Cu substrate was measured and analyzed to identify the phases in a cross-section of the specimen. The average thickness was calculated by measuring the area of the compound and dividing it by the total length of the image. In this research, the thickness of the IMC layers was taken as an average over three locations. Elemental composition of the

Table 1
EDX analysis for points X and Y at 140 °C reflow temperature.

Point	At%		
	Cu	Sn	In
X	58.87	21.23	19.89
Y	44.85	–	55.15

IMC was investigated by EDX, and the IMC layers were proved by X-ray diffraction (XRD) analysis.

The shear strength of the In–31.6Bi–19.6Sn/Cu solder joint was evaluated by a single lap shear test using monotonic software. Solder joints were made according to the conventional method for making shear test specimens, and the lap shear test was carried out to estimate bonding strength. To perform the test, solder was applied between two Cu pieces with a length of 40, a width of 10 and a thickness of 0.5 mm. The specimen was pulled in shear mode until fracture. The method of shear testing followed the ASTM E8/8M standard (physical and mechanical testing standard). Prior to testing, the samples were ground using 1200 grit SiC paper and polished to remove any excess solder at the edges. The crosshead speed used was 1 mm/s. The fracture surface morphology of the specimens was examined under optical microscope and SEM operated at 20 keV.

3. Results and discussion

Fig. 2 presents the results of differential heat flow as a function of temperature for the In–31.6Bi–19.6Sn solder. A single sharp endothermic peak was recorded within the studied temperature range. The peak and onset temperatures are 61.333 and 59.836 °C, respectively. The former temperature is associated with liquidus temperature of the solder while the latter is related to its solidus temperature.

The measured withdrawal force (F_W) of the solder on Cu substrate at different reflow temperatures is shown in Fig. 3. The force was then converted into surface tension (γ) (Fig. 3), using the following equation [31]:

$$\Delta F_W = \gamma P \quad (1)$$

where P is the perimeter of the area of the sample covered by molten solder, calculated from the width (10 mm) and the thickness (4.0 mm) of the Cu substrates. The withdrawal force was used to calculate the surface tension of the solder using Eq. (1). The result showed that surface tension γ decreased as the reflow temperature increased. A similar observation has been reported for 60Bi–24Cu–16Sn solder [32], where surface tension γ decreased as the temperature increased from 747 to 777 °C. However, the percentage of reduction (3.9%) is much lower if compared with that of this solder (29.92%). This finding may be attributed to the increment of fluidity and higher reaction rate of the flux as the reflow temperature increases.

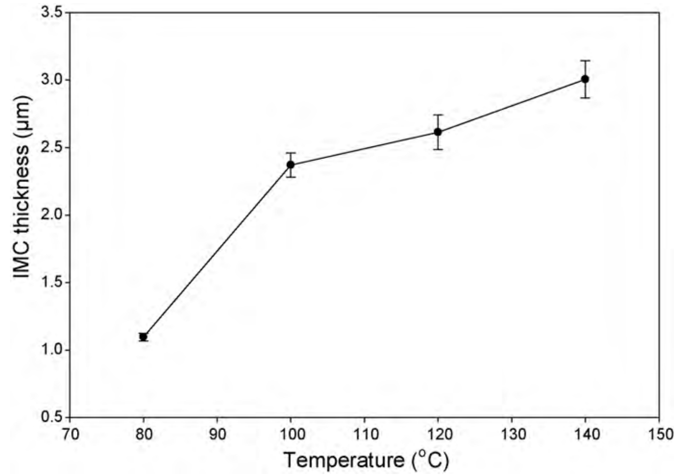

Fig. 6. Intermetallic compound layers thickness of Bi–In–Sn lead-free solder on Cu substrate at different reflow temperatures.

Wettability of the solder on Cu substrate, which can be determined by the spreading area and the contact angle of the solder on the substrate, was evaluated as a function of reflow temperature (Fig. 4). As the temperature increased from 80 to 100 °C, a significant increment was seen in the spreading area of the solder on the substrate. The increment of the spreading area became smaller as the reflow temperature increased. An inverse trend was observed in the contact angle between the solder and the substrate. The reduction of contact angle and the increase in the spreading area with increasing reflow temperature may both be attributed to the increase in the reaction rate of the flux. In addition, elements of In and Bi in the solder probably act as agents to reduce surface tension of the molten solder (γ_{SL}) at higher temperatures. As γ_{SL} is reduced, a smaller contact angle and better wetting behavior can be obtained, as expressed by Eq. (1).

Since the contact angle between solder and substrate in the investigated reflow temperature range is much lower than 90° (between 19° and 35° for reflow temperatures between 80 and

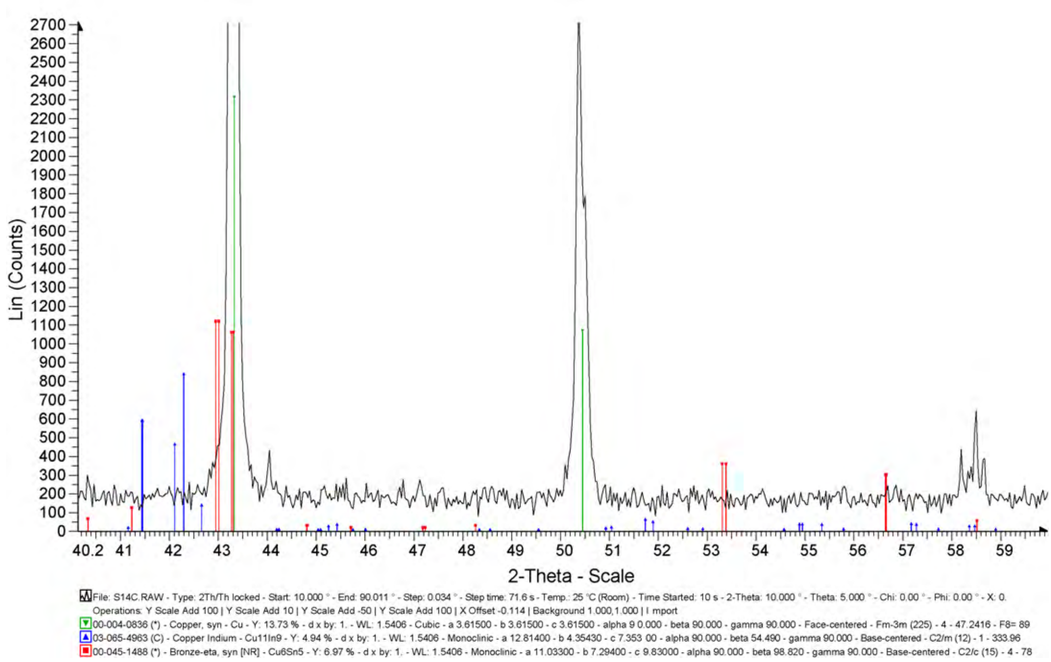

Fig. 7. XRD image of In–30Bi–20Sn/Cu joint with 140 °C reflow temperature.

140 °C), it can be concluded that the solderability of this novel solder is good. These results are comparable with those obtained by other researchers for contact angle for Sn–3.5Ag–4.8Bi (12–23° for reflow temperature between 240 and 260 °C) [8] but slightly better than the results for 95.5Sn–3.9Ag–0.6Cu solders (42° and 39° for reflow temperature between 230 and 260 °C) [33].

Fig. 5(a)–(e) illustrates a backscattered electron micrograph of the cross-section of the In–Bi–Sn/Cu interface obtained after spreading at different reflow temperatures. IMC thickness was found to increase with increasing reflow temperature. The IMC thickness in the In–Bi–Sn solder system is prominent, especially at high temperature (140 °C). This observation shows that as temperature increases, Sn reacts with Cu at a faster rate.

The results of EDX analysis at points X and Y are summarized in Table 1. Two layers of IMC were found to form between the solder and Cu substrate. Two types of IMCs could be observed: a scallop-type morphology and a brighter continuous layer formed underneath. Based on SEM and EDX analysis at point X of this solder, it is suggested that the scallop-type microstructure IMC is Cu_6Sn_5 mixed with $Cu_{11}In_9$ while the IMC layer underneath is $Cu_{11}In_9$ in bright colour (point Y). The element of composition at point X, chosen from the upper IMC layer, is 19.89 at%In–21.23 at%Sn–58.87 at% while at point Y, the IMC layer is 55.15 at%In–44.55 at%Cu.

The concentration of Sn was measured by EDX. At point Y, Sn was not detected. These results are in agreement with the binary phase diagrams for Cu–In [34] and Cu–Sn [35]. According to Frear et al. [36], Cu_6Sn_5 is observed in the case of soldering of Sn–Ag–Cu on Cu substrate, forming a rough layer, then extending into the solder. No Bi concentration was detected in the IMC region at either point X or point Y, suggesting that Bi was not present in the IMC. Therefore, no data on Bi concentration is included in Table 1. These results agree well with the findings of Islam et al. [37], who explained that Bi does not take part in the interface reactions and therefore does not form any IMC layer with Cu.

According to Suganuma [20], it is possible for two types of IMC (Cu_6Sn_5 and Cu_3Sn) to be formed between Sn alloy and Cu substrate at temperatures lower than 350 °C. In this work, a Cu_6Sn_5 layer was observed instead of Cu_3Sn because Cu_6Sn_5 grows much faster than Cu_3Sn in the molten state and at room temperature while a Cu_3Sn layer is usually formed at high temperature [36]. The formation of these IMCs may be attributed to the saturation of molten solder in contact with Cu [8]. Based on Fig. 6, it was found that the average thickness of the total IMC layer increased as the reflow temperature increased. However, the variation in thickness is not significant. In addition, the increment in reflow temperature promotes the growth of scallop-like grains. This may be due to the result of a ripening reaction between the scallops.

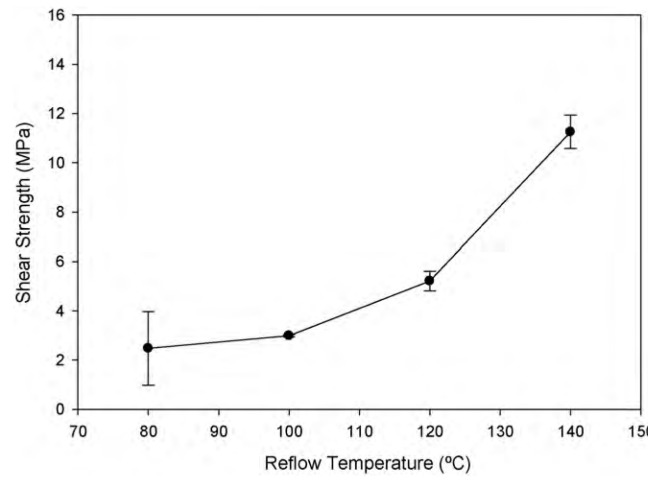

Fig. 8. Shear strength of In–30Bi–20Sn/Cu joint.

The presence of $Cu_{11}In_9$ (ICDD 03-065-4963) was confirmed by XRD analysis (Fig. 7). Currently, no research has been reported on the formation of $Cu_{11}In_9$ at low temperature. However, Chen [38] has explained that Bi addition decreases the melting temperature of Sn–Zn alloy and with this lower melting temperature the alloy experiences a longer molten period during reflow. Both this statement and XRD analysis suggest that the Bi addition has reduced the temperature for $Cu_{11}In_9$ formation.

A shear strength test was conducted to evaluate the effect of different reflow temperatures on the reliability of the solder joint. Fig. 8 shows the variation of shear strength with reflow temperature. The strength of the In–31.6Bi–19.6Sn/Cu solder joint increased with increasing reflow temperature. A significant increase in shear strength from 2.4 to 11.3 MPa was seen as the temperature rose from 80 to 140 °C. Previous studies [39,40] have indicated that the interfacial reaction, producing intermetallic compounds between solders and substrates, has a significant effect on the mechanical properties and reliability of the solder joints. Results of the present work show that the IMC thickness did not influence the shear strength of the joint as the reflow temperature increased from 80 to 140 °C.

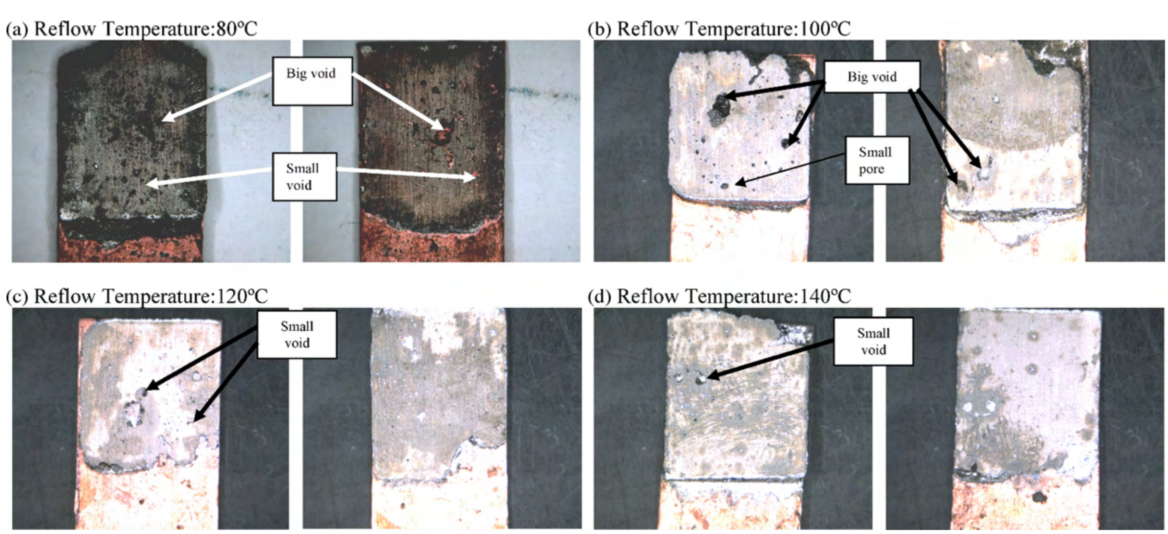

Fig. 9. Fracture surface of In–30Bi–20Sn/Cu joint with different reflow temperatures as observed under optical microscope.

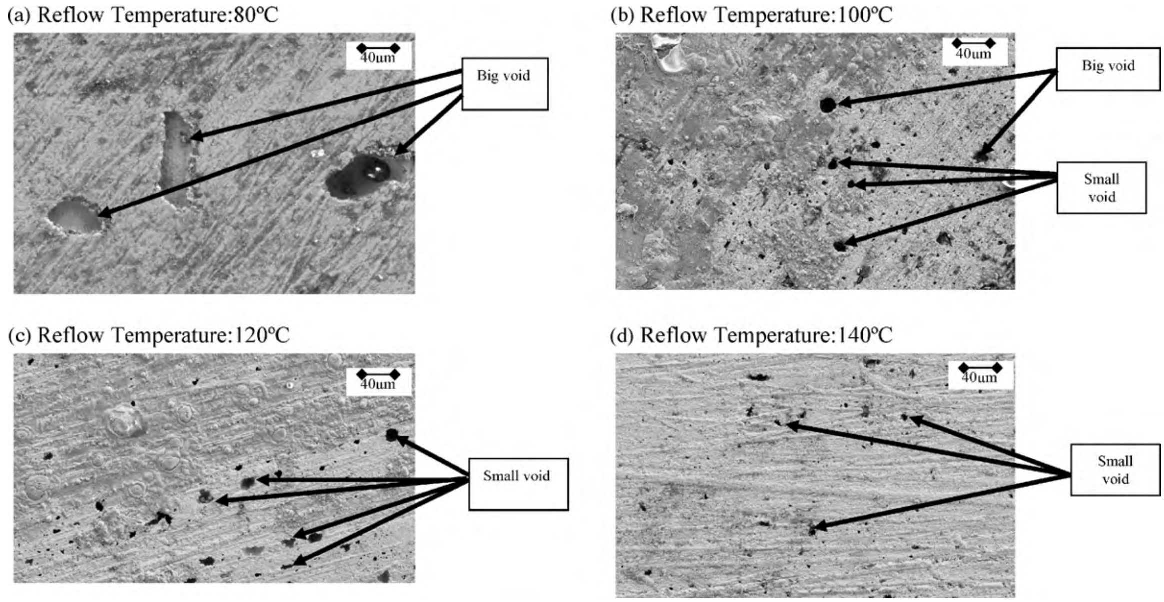

Fig. 10. Fracture surface on In–30Bi–20Sn/Cu joints with different reflow temperatures as observed under SEM.

To investigate the dependence of shear strength on reflow temperature, fracture surfaces were examined under an optical microscope and SEM. Fig. 9 and Fig. 10 show fracture surfaces of In–31.6Bi–19.6Sn solder joints with different reflow temperatures as observed under optical microscope and SEM, respectively. The fracture surfaces were pock-marked with voids, with more numerous and smaller voids for 80 and 100 °C reflow temperatures than those for the 120 and 140 °C reflow temperatures. Lin et al. [41] have indicated that the presence of larger and continuous voids at the interface between the solder alloy and IMC are correlated with lower solder joint strength.

The micrograph shows that as the reflow temperature increased, the number of voids in the fracture surface decreased, possibly because of the wettability between solder with Cu substrate; if a solder joint is improperly soldered due to poor wetting, then failure can occur directly at the interface [38]. As observed in all samples at all reflow temperatures, the fracture typically occurred at the bulk solder. This argument is in agreement with the result of contact angle and spreading area of the In–31.6Bi–19.6Sn solder on Cu substrate. In both cases, when the spreading area became smaller with a larger contact angle, wettability reduced, which also resulted in more voids. However, when the spreading area was larger with a lower contact angle, wettability became excellent (i.e. with fewer voids). Consequently, the shear strength of this solder on Cu substrate increased as reflow temperature increased.

4. Conclusions

The wettability and microstructural characteristics of In–31.6Bi–19.6Sn (low melting temperature ∼60 °C), a novel lead-free solder, were investigated. Surface tension and contact angle of the solder on Cu were evaluated in a reflow temperature range of 80–140 °C. As the temperature increased, the surface tension was found to decrease. A reduction in contact angle and an increment in spreading area in this solder improved wettability were observed as the temperature increased. Two types of IMC were detected between the solder reflow and Cu substrate: Cu_6Sn_5 and $Cu_{11}In_9$ (scallop shaped) and $Cu_{11}In_9$ (brighter thin flat layer). The shear strength of the In–31.6Bi–19.6Sn/Cu solder joint strengthened by voids and wettability. Higher wettability, with lower contact angle and larger spreading area, gave higher shear strength as the reflow temperature increased.

Acknowledgements

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