

Research Article

Effect of Indium Additions on the Formation of Interfacial Intermetallic Phases and the Wettability at Sn-Zn-In/Cu Interfaces

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The wettability of copper substrates by Sn-Zn eutectic solder alloy doped with 0, 0.5, 1, and 1.5 at.% of indium was studied using the sessile drop method, with flux, in air, at 250°C and reflow time of 3, 8, 15, 30, and 60 min. Wetting tests were performed at 230, 250, 280, 320, and 370°C for an alloy containing 1.5 at.% of indium, in order to determine activation energy of diffusion. Solidified solder/substrate couples were studied using scanning electron microscopy (SEM), the intermetallic phases from Cu-Zn system which formed at the solder/substrate interface were identified, and their growth kinetics was investigated. The ϵ -CuZn₄ was formed first, as a product of the reaction between liquid solder and the Cu substrate, whereas γ -Cu₅Zn₈ was formed as a product of the reaction between ϵ -CuZn₄ and the Cu substrate. With increasing wetting time, the thickness of ϵ -CuZn₄ increases, while the thickness of ϵ -CuZn₄ does not change over time for indium-doped solders and gradually disappears over time for Sn-Zn eutectic solder.

1. Introduction

The continuous development of microelectronics in respect to miniaturisation and improved efficiency motivates the search for new materials and technologies capable of allowing both cost reductions and increased density of connections and efficiency, along with improved reliability. The European Parliament and the Council of Europe directives issued in [1–3] 2003 and in 2008 prohibited the use of solders containing metals such as lead and cadmium, which are detrimental to human health. Therefore, in recent years intense research has been conducted worldwide, the aim of which is to devise new alternatives to hazardous solders such as those containing lead.

Interest in alloys based on Sn-Zn eutectic is the result of the search for the optimum replacement of Pb-Sn solders. Sn-Zn eutectic's greatest advantages are its low melting temperature (198°C), which is close to the melting temperature of Pb-Sn eutectic, and the low cost per mass unit. In addition, Sn-Zn alloys exhibit relatively high mechanical strength, corrosion

resistance, and resistance to thermal fatigue [4–8]. On the other hand, these alloys wet soldered surfaces poorly and are prone to oxidation unless soldering is carried out under a protective atmosphere or with a sufficiently aggressive flux [9–11]. Well-chosen alloying additions may improve joint properties, so in designing joints it is essential to understand the effect of alloying additions on processes occurring at the interface of solder and substrate. The addition of indium may lower the melting temperature of solders but also improve the wettability and durability of solder joints [12]. We earlier investigated the effect of indium addition on the properties of soldered joints: Cu/SnAgCu + In [13], Ni/Sn-Zn + In [14], and to some extent Cu/Sn-Zn + In [15, 16]. In the case of Cu/SnAgCu + In [13], it was found that the Cu₆Sn₅ was formed at the solder/Cu interface for solders with low In concentration, whereas for high In concentrations it was the Cu₄₁In₁₁ phase that developed. Also, higher In content leads to the formation of In-based phases (InSn₄), which in turn hinder the diffusion of Cu and resulted in the decreased thickness of the interfacial Cu-rich layer [13]. The addition of

In to Sn-Zn eutectic leads to a lower wetting angle on Ni and Cu substrates [14]. Earlier [15], we found that the spreading area increases with In content over a relatively short wetting time, but for longer wetting times this effect was absent.

The authors of [16] studied the effect of indium on the surface tension, electrical conductivity, and viscosity of liquid In-Sn, and the correlation between these properties and the structure of the liquid. They found anomalies in concentration dependence of viscosity near indium concentrations corresponding to intermetallic phases existing in the In-Sn system.

Taking the above into account, the aim of this work is to investigate the effect of indium addition to Sn-Zn eutectic on spreading on copper substrates, as well as on the phenomena occurring at solder/substrate interface during soldering.

2. Experimental

For wetting tests, copper substrates (99.9 wt.%, Alfa-Aesar) were used and their surfaces prepared in accordance with EN ISO 9455-10:2000. Copper plates (40 × 40 × 0.25 mm) were etched in 99.5% copper sulphide solution for 8 min, subsequently ground with sand paper (1,000), and degreased with methyl alcohol and acetone directly before wetting tests.

Alloys were prepared by melting carefully weighed amounts of pure (99.999 wt.%) Sn, Zn, and In in alumina crucibles under high purity (99.9999 vol.%) Ar atmosphere. The wetting tests were carried out with the use of the apparatus earlier described in [17], at 250°C, for different annealing times (3, 8, 15, 30, and 60 minutes), without a protective atmosphere and with the use of the Alu-33® flux produced by Amasan. According to ISO 9454-1, this is a 2.1.2-type flux, that is, organic, water-soluble, and activated with halides. In order to determine the activation energy of the growth of the Cu₅Zn₈ phase for an alloy containing 1.5 at.% of indium, wetting test were performed at 230, 250, 280, 320, and 370°C. For each set of the experimental conditions (sample composition, temperature, and time) 5–7 independent wetting tests were performed. Solidified samples were washed with tap water to remove flux residue and photographed from two perpendicular directions. Wetting angle data are the average of individual measurements for all samples. The wetting angles were determined from photographs using the application ImageJ. The spreading areas of solders were determined from photographs of the top view of drops. Selected solidified solder/substrate couples were cut perpendicular to the plane of the interface, mounted in conductive resin, polished for microstructural characterisation, and sputtered with a thin carbon layer. Microstructural and energy-dispersive spectroscopy (EDS) analysis was performed with the FEI E-SEM XL30 system, at 20 kV, with the use of the standardless Analysis EDAX System based on Genesis 2000 software. The intermetallic phases of CuZn₄ and Cu₅Zn₈ were identified based on analysis of results EDS and knowledge of the binary Cu-Zn phase diagram. The thickness of intermetallic (IMC) layers was determined from micrographs with the use of ImageJ software.

3. Results and Discussion

Figure 1(b) illustrates the wetting angle of Sn-Zn-In alloys on copper substrate at 250°C, for varying indium content and time. It is clear that the wetting angles of In-doped alloys are lower than those of the alloy without In addition. This difference is more pronounced than in our earlier work [14], where we also found that the wetting angle decreased with increasing temperature. However, this earlier investigation was carried out with different flux. For the alloys in the present study, the wetting angle seems to be practically independent of wetting time. Similarly, we earlier [15] observed, with the use of the same flux as in this study, that the spreading area does not increase over time. Figure 1(a), based on the example of an alloy containing 1.5% of In, illustrates that the spreading area increases with increasing temperature

$$\sigma_{SV} = \sigma_{SL} + \sigma_{LV} \cos \phi. \quad (1)$$

According to the Young-Dupre equation (1), as illustrated in Figure 2, wettability can be improved (wetting angle ϕ lowered) by increasing the surface energy of the solid substrate σ_{SV} , by lowering the surface tension of the liquid alloy σ_{LV} , or by lowering solid-liquid interfacial tension σ_{SL} . The increase of surface energy of solid σ_{SV} occurs as the oxide layer is removed by flux action. The surface tension of the alloy, on the other hand, can be lowered by doping with surface active components or metals with substantially lower surface tension. From Figure 1 we can conclude that, with increasing In content, the wettability improves, despite the fact that authors of [16, 18] did not observe surface tension decreasing with indium concentration. The authors of [13] claim that increasing concentration of In in liquid solder (Sn-Ag-Cu alloy in their case) improves wettability on copper. This is because In lowers solid-liquid interfacial tension, even if it does not lower the surface tension of the solder.

Figure 1(b) presents the spreading area of (SnZn)_{eut}+1.5% at. In solder on Cu after 8 min of wetting at 230, 250, 280, 320, and 370°C. It is clear that the spreading area increases with temperature, which could be a result of the surface tension of Sn-Zn-In alloy decreasing with temperature [18] and may also be due to changes in interfacial tension.

After wetting tests, the microstructure of the solder/Cu interface of selected samples was investigated. According to Lee et al. [19], Gibbs free energy of the formation of the γ -Cu₅Zn₈ phase is -212.10 kJ/mol (at 150°C), while Gibbs free energy of the formation of Cu₆Sn₅ is -26.26 kJ/mol, which explains why the intermetallic phase from the Cu-Zn system is formed at the liquid Sn-Zn/solid Cu interface. At 250°C, in the Sn-Zn-Cu system the liquid phase (composition close to eutectic Sn-Zn) coexists in equilibrium with two intermetallic phases: γ -Cu₅Zn₈ and ϵ -CuZn₄ [20, 21]. In a study of Sn-Zn-Cu/Cu interfaces over short wetting time [22], we found that, at the earliest stage of the soldering process, liquid solder reacts with the Cu substrate and ϵ -CuZn₄ is formed. After 60 s, as a result of the diffusion of elements, γ -Cu₅Zn₈ starts to appear. In the present study, both ϵ -CuZn₄ and γ -Cu₅Zn₈ were found at the solder/substrate interface.

Figures 3 and 4, respectively, illustrate the interfacial microstructure of Sn-Zn/Cu and Sn-Zn-1.5% In/Cu couples

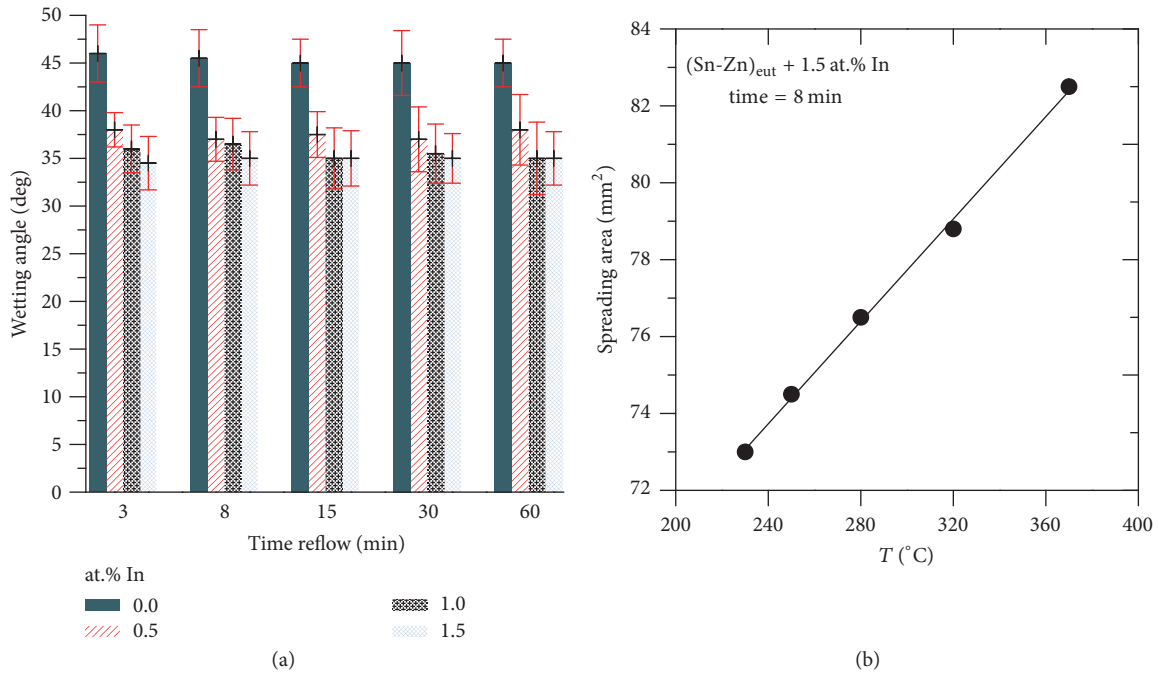


FIGURE 1: Wetting angle (a) and spreading area (b) of 0.5 g liquid Sn-Zn-xIn ($x = 0, 0.5, 1, 1.5$) on copper substrate: (a) 250°C, 3–60 min, (b) 230–370°C, 8 min.

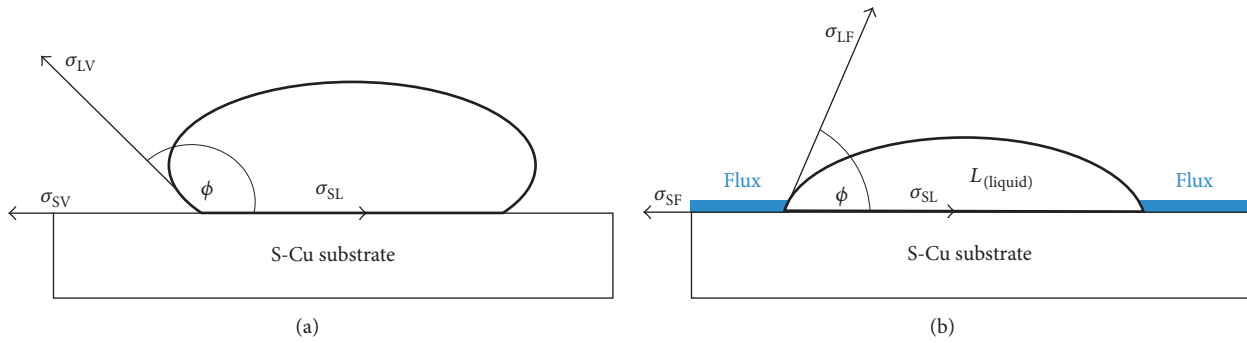


FIGURE 2: Sketch of Young-Dupre equation.

after (a) 3, (b) 15, and (c) 30 min at 250°C. The growth of intermetallic layers over time at the solder/substrate interface of the abovementioned couples is illustrated in Figure 5. For both examples, there are two intermetallic phases at the interface in the early stage (up to 15 min). These are ϵ -CuZn₄ on the side of the solder and γ -Cu₅Zn₈ adjacent to the Cu substrate. At this stage, the thickness of the γ -Cu₅Zn₈ phase increases over time, while the thickness of ϵ -CuZn₄ remains practically the same. For longer wetting times, the thickness of the γ -Cu₅Zn₈ phase continues to increase, while ϵ -CuZn₄ in the case of Sn-Zn/Cu couples begins to disappear and grows very little in the case of Sn-Zn-1.5% In/Cu couples. Moreover, just 0.5% of In is sufficient to keep the ϵ -CuZn₄ stable even after 60 min of wetting (Figures 6 and 7), as we observed earlier [15]. The bright spots between the ϵ -CuZn₄ and γ -Cu₅Zn₈ in Figure 6 are, according to EDS analysis,

composed mostly of Sn with In content somewhat higher than the In content in the Sn-Zn-In solder.

According to the Gibbs phase rule, a maximum of three phases can coexist in equilibrium under the conditions of constant pressure and temperature ($T, p = \text{const}$) in a three-component system. Although the solder/substrate interfaces are not under equilibrium conditions, it can be assumed that, as wetting time increases, Sn-Zn/Cu couples are getting closer to equilibrium. This might explain why, after 60 min, three phases coexist in the Sn-Zn/Cu couples. These phases are liquid (solder), Cu₅Zn₈, and Cu (substrate). The addition of the fourth component, In in the present case, allows the fourth phase to coexist (CuZn₄ in the present case).

In the course of the experiment, Sn, Zn, and In diffuse from the liquid solder towards the solid Cu, while Cu diffuses in the opposite direction. As a result of diffusion and

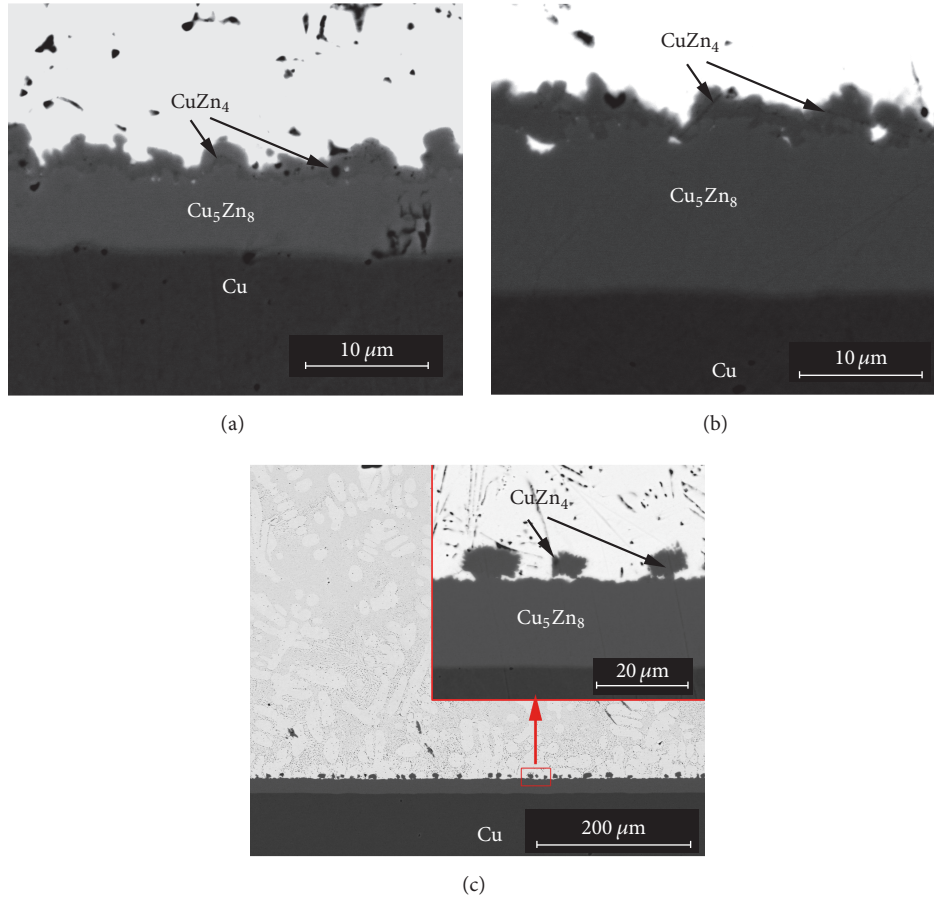


FIGURE 3: Interfacial microstructure of Sn-Zn/Cu couples, $T = 250^{\circ}\text{C}$, reflow time: (a) 3 min, (b) 15 min, and (c) 30 min.

reactions between the liquid solder and solid substrate, one or more intermetallic layers are formed at the solder/substrate interface. As explained earlier [22], at the earliest stage of wetting $\epsilon\text{-CuZn}_4$ appears first, and only after 30 s of wetting can the $\gamma\text{-Cu}_5\text{Zn}_8$ be distinguished. Diffusion processes continue and lead to the increased thickness of intermetallic layer(s). Moreover, the $\gamma\text{-Cu}_5\text{Zn}_8$ is thermodynamically more stable than $\epsilon\text{-CuZn}_4$, so it can be speculated that, for Sn-Zn/Cu couples, after 15 min the $\epsilon\text{-CuZn}_4$ transforms to $\gamma\text{-Cu}_5\text{Zn}_8$. This can be expressed as the reaction $5\text{CuZn}_4 \rightarrow \text{Cu}_5\text{Zn}_8 + 12\text{Zn}$. The freed Zn atoms go to the liquid solder, locally increasing Zn concentration near the interface. In the case of $(\text{Sn-Zn})_{\text{eut}} + 1.5\% \text{In}$ /Cu couples, In atoms diffusing through the $\epsilon\text{-CuZn}_4$ layer towards substrate may stop at the $\epsilon\text{-CuZn}_4/\gamma\text{-Cu}_5\text{Zn}_8$ interface. Earlier, we discussed the growth mechanism of intermetallic layers at the Sn-Zn-Cu/Cu interface [22] as a result of Cu and Zn diffusion in the opposite direction. It is possible that the indium atoms form a kind of diffusion barrier for Cu diffusing towards the solder, but not a barrier for Zn diffusing towards the substrate. As a result, the $\epsilon\text{-CuZn}_4$ does not disappear after a prolonged time for Sn-Zn-In/Cu couples, but neither does its thickness increase substantially.

Evolution of intermetallic layer thickness with time is described with [23–25]

$$\delta = ke^{-Q/RT}t^n, \quad (2)$$

where δ is the thickness of the intermetallic layer [μm], k is the growth constant, Q is the activation energy of growth [kJ/mol], R is the universal gas constant [8.314 J/mol], T is soldering temperature [K], t is soldering time [min.], and n is the time exponent.

Figure 8 illustrates the growth kinetics of $\epsilon\text{-CuZn}_4$ and $\gamma\text{-Cu}_5\text{Zn}_8$ for $(\text{Sn-Zn})_{\text{eut}}$ and $(\text{Sn-Zn})_{\text{eut}} + 1.5\% \text{In}$ in comparison to literature data [26, 27]. It is clear that the growth constant of the $\gamma\text{-Cu}_5\text{Zn}_8$ phase ($k = 3.18$) in the case of Sn-Zn/Cu is the same as in the case of Sn-Zn-1.5% In/Cu and is more than ten times higher than the growth constant of the $\epsilon\text{-CuZn}_4$ phase. The present k of the $\gamma\text{-Cu}_5\text{Zn}_8$ phase is slightly higher than that reported in [26]. On the other hand, it can be assumed that growth constant k increases with temperature, since for Sn-Zn/Cu at 230°C the reported k is 1.72.

In order to determine the activation energy of the growth of the $\gamma\text{-Cu}_5\text{Zn}_8$ phase, samples were held at 230, 250, 280, and 320°C for 8 min. Figure 9 illustrates the relation between

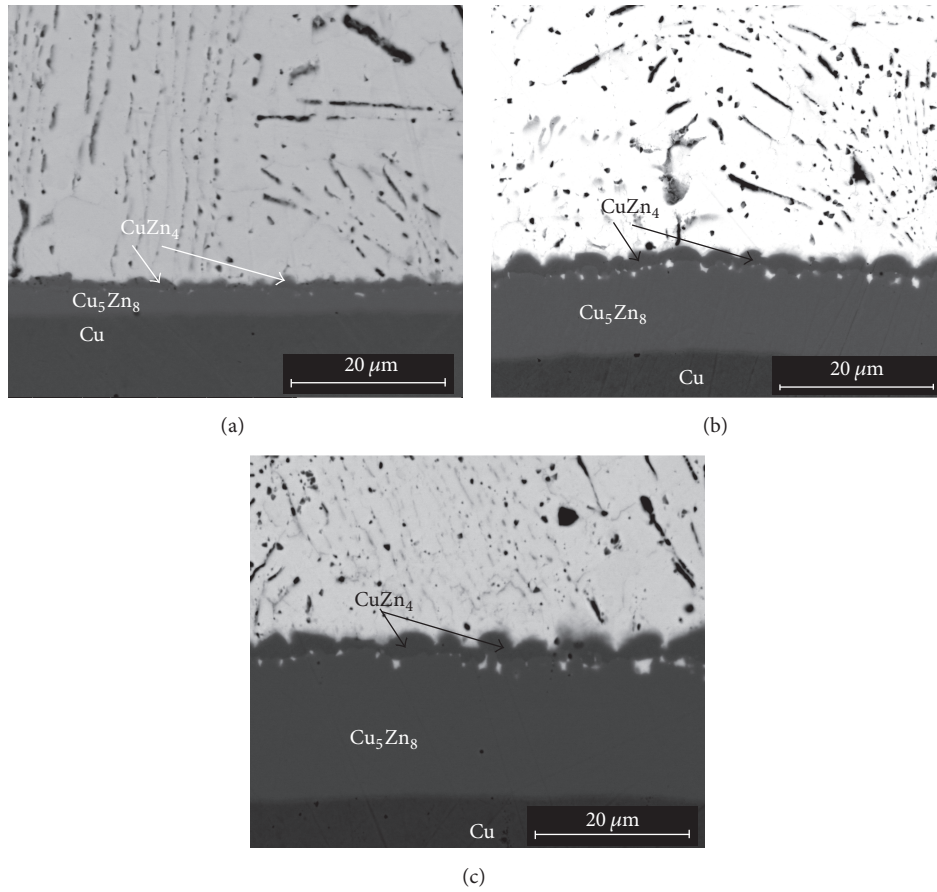


FIGURE 4: Interfacial microstructure of Sn-Zn-1.5% In/Cu couples, $T = 250^{\circ}\text{C}$, reflow time: (a) 3 min, (b) 15 min, and (c) 30 min.

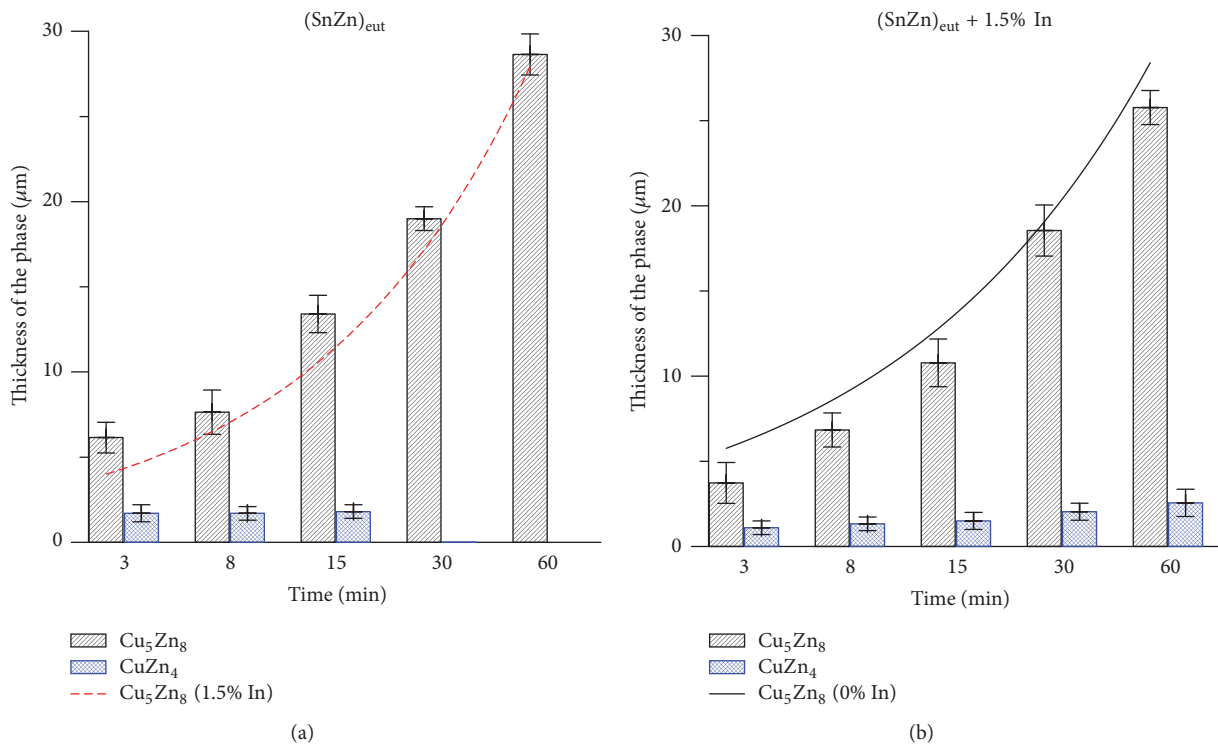


FIGURE 5: Thickness of intermetallic layers (Cu_5Zn_8 , CuZn_4) at $(\text{Sn-Zn})_{\text{eut}}/\text{Cu}$ and $(\text{Sn-Zn})_{\text{eut}} + 1.5\% \text{In}/\text{Cu}$ interface at 250°C .

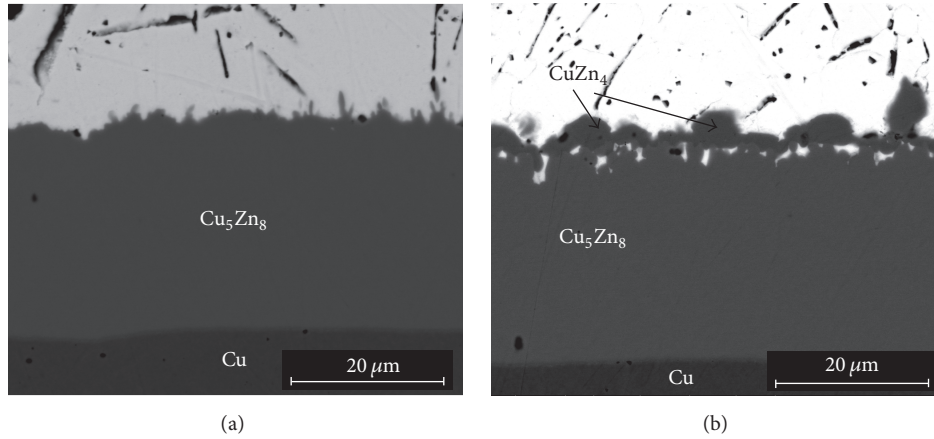


FIGURE 6: Interfacial microstructures of (a) Sn-Zn/Cu couples and (b) Sn-Zn-0.5% In/Cu couples, $T = 250^\circ\text{C}$, reflow time 60 min.

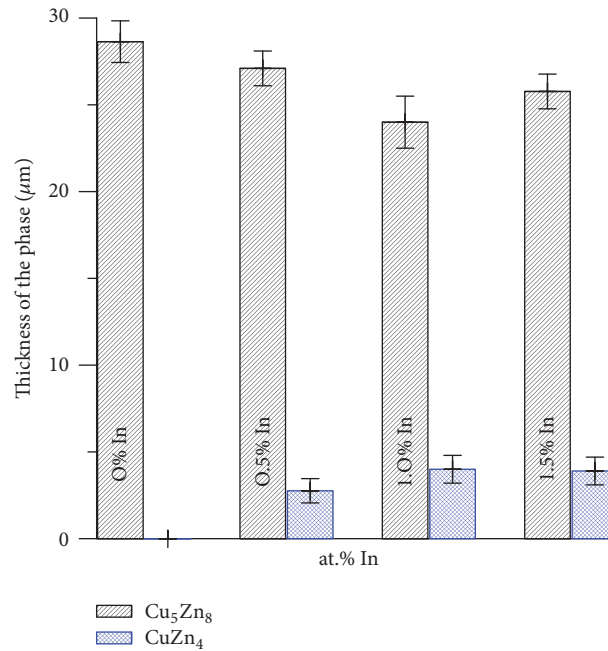


FIGURE 7: Thickness of the CuZn_4 and Cu_5Zn_8 intermetallic layers at the $(\text{Sn-Zn})_{\text{eut}} + x\text{In}/\text{Cu}$ ($x = 0, 0.5, 1, 1.5\%$) interfaces after 60 min at 250°C .

the growth rate and reciprocal soldering temperature, according to (3). The activation energy of growth of the phase is, according to our results, equal to 29.5 kJ/mol , which is close to 26 kJ/mol reported in [26].

$$\ln \delta = \ln k + n \ln t - \frac{Q}{RT}. \quad (3)$$

4. Conclusion

Wettability studies of Sn-Zn eutectic doped with In on Cu substrate were performed in the presence of flux over a temperature range of $230\text{--}370^\circ\text{C}$ for wetting times in the range 3–60 min. It was found that additions of indium lower

the wetting angle, but the wetting time does not affect the wetting angle.

The microstructure of solidified solder/Cu interfaces was investigated with scanning electron microscopy. Two intermetallic phases are observed at the interface: $\epsilon\text{-CuZn}_4$ on the side of the solder and $\gamma\text{-Cu}_5\text{Zn}_8$ adjacent to the Cu substrate. At the early stage (up to 15 min), the thickness of the $\gamma\text{-Cu}_5\text{Zn}_8$ phase increases much faster. For longer wetting times, greater than 15 min, the thickness of the $\gamma\text{-Cu}_5\text{Zn}_8$ phase continues to increase, while $\epsilon\text{-CuZn}_4$ in the case of Sn-Zn/Cu couples begins to disappear and grows very little in the case of Sn-Zn-1.5% In/Cu couples. Therefore, the addition of indium to Sn-Zn solder stabilises $\epsilon\text{-CuZn}_4$ at the interface of solder and substrate, even after 60 min of wetting.

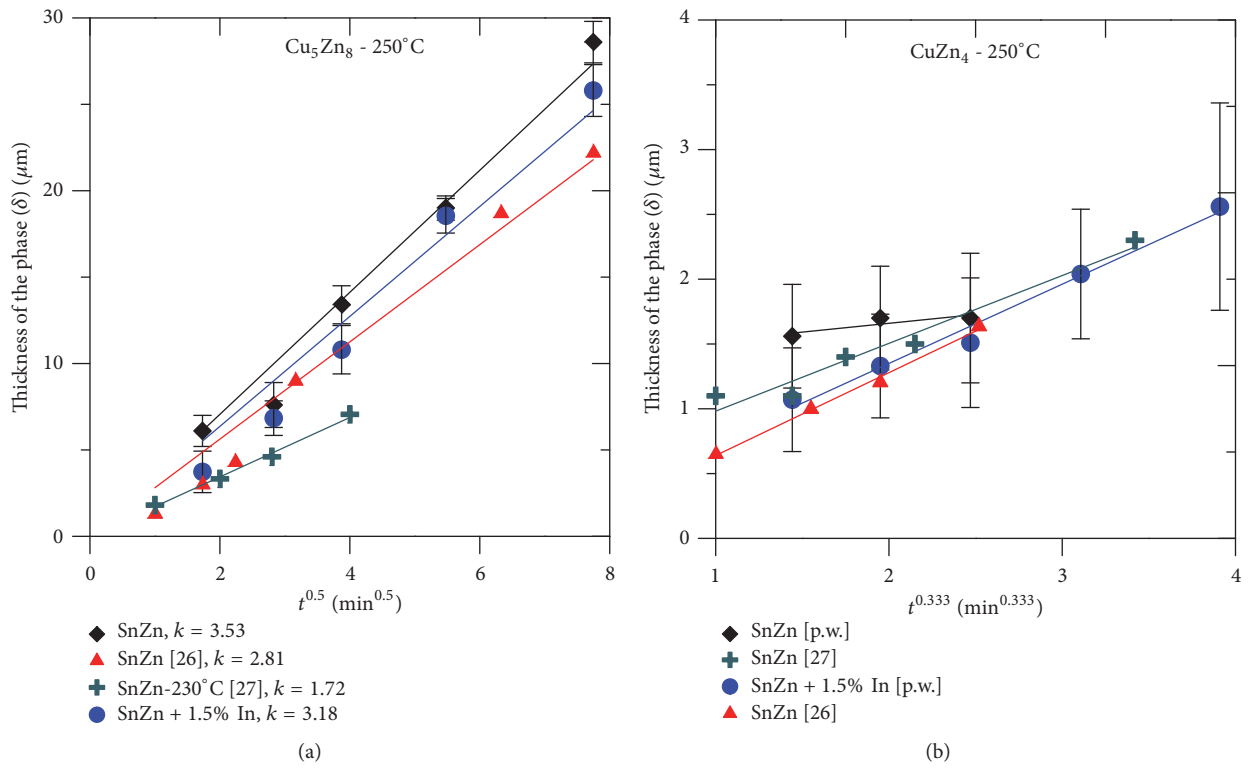


FIGURE 8: Growth kinetics of $\epsilon\text{-Cu}_5\text{Zn}_8$ (a) and $\gamma\text{-CuZn}_4$ (b) at 250°C , for $(\text{Sn-Zn})_{\text{eut}}/\text{Cu}$ and $(\text{Sn-Zn})_{\text{eut}} + 1.5\% \text{In}/\text{Cu}$ interfaces. Present work (p.w.) and [26] at 250°C , [27] at 230°C .

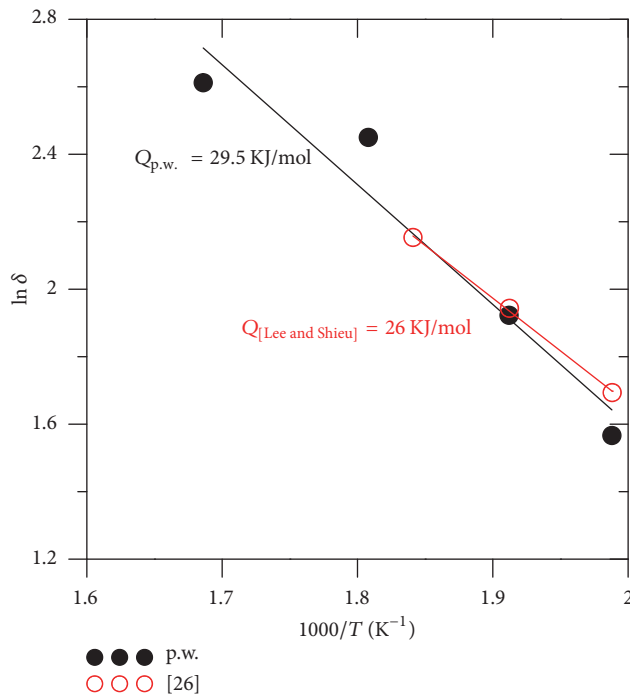


FIGURE 9: Arrhenius plot for the activation energy of Cu_5Zn_8 phase layer at the interface between molten $(\text{Sn-Zn})_{\text{eut}}$ [26] or molten $(\text{Sn-Zn})_{\text{eut}} + \text{In}$ [present work -p.w.] and Cu.

Conflicts of Interest

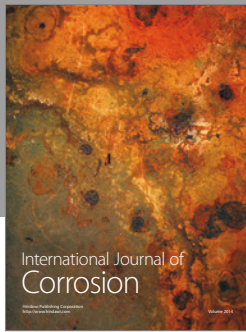
The authors declare that there are no conflicts of interest regarding the publication of this article.

Acknowledgments

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