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Wetting and Interfacial Chemistry of SnZnCu Alloys with Cu and Al Substrates

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Wetting of Cu and Al pads by Sn-Zn eutectic-based alloys with 0.5, 1, and 1.5 wt.% of Cu was studied at 250 °C, in the presence of ALU33® flux, with wetting times of 15, 30, 60, and 180 s, respectively. With increasing wetting time the wetting angle decreases only slightly and the angles on Cu pads are higher than those on Al pads. Selected, solidified solder-pad couples were cross-sectioned and subjected to SEM-EDS study of the interfacial microstructure. The results revealed that the microstructure of the SnZnCu/Cu interface is much different from SnZnCu/Al interface. In the first case continuous interlayers are observed while in the latter case there is no interlayer but the alloy dissolves the substrate along grain boundaries.

Keywords microstructure, Sn-Zn-Cu, wetting

1. Introduction

Among Pb-free solders Sn-9Zn (wt.%) solder alloy is considered the best with respect to cost per mass unit. Also, its melting temperature (199 °C) makes it a close match to conventional Sn-37Pb solder. Mechanical and thermal properties of Sn-Zn solder alloys and wetting behavior on Cu were thoroughly studied by numerous authors, in particular good mechanical properties were reported but the wettability was inferior to Sn-37Pb (Ref 1, 2). Some researchers studied corrosion behavior of Sn-Zn alloys in gaseous mixtures (Ref 3) and high-humidity conditions (Ref 2) and found Sn-Zn alloys prone to corrosion. In order to improve corrosion resistance and wetting properties alloying additions were introduced including for example: Ag (Ref 4, 5), Bi (Ref 2, 6), Cu (Ref 7), and In (Ref 8). The results of some of these efforts were reviewed by Zhang and co-workers (Ref 9). Despite intensive development of new joining techniques such as ultrasonic-assisted joining (Ref 10), Sn-Zn-based solders were found to be useful in joining of aluminum with dissimilar materials, in particular copper (Ref 11).

Yu et al. (Ref 12) were among the first to study wetting properties and interfacial microstructure of Sn-9Zn-*x*Cu solders (*x* = 0, 2, 4, 6, and 10 wt.%) and Cu substrate. Based on reflow test with peak temperature 260 °C, they found that increased copper content (2 wt.% and more) improves wetting and increases Cu_6Sn_5 IMC in the interlayer on expense of the Cu_5Zn_8. The γ-Cu_5Zn_8 phase is the only one that they found at the eutectic Sn-Zn/Cu interface. Chou et al. (Ref 7) studied interaction between Sn-9Zn-*x*Cu alloys (*x* = 0, 1, and 10 wt.%) and Cu substrate at 250 °C with reaction time of several hours, and they found up to three intermetallic (IMC) layers at the interface: ε-$CuZn_5$, γ-Cu_5Zn_8, and β-CuZn. IMCs layer growth is observed in the case of all Sn-Zn-*x*Cu solders on Cu with γ-Cu_5Zn_8 being the dominant one, yet as pointed in (Ref 7) the literature data on remaining IMCs are inconclusive. What is particularly interesting is the early stage of IMC layer growth i.e., occurring in the first seconds of wetting, as this should help explain the order in which IMCs are grown. To study this is not easy, as it requires to hold a sample at high temperature for specified time and then to cool it rapidly below solidus temperature of solder to preserve the interfacial microstructure. In this work we attempt to study evolution of the interface between liquid Sn-Zn-*x*Cu (*x* = 0.5, 1.0, and 1.5 wt.%) solders and Cu substrate held at 250 °C for a short time of 15, 30, 60, and 180 s. Also, to the best of the present authors knowledge there are no reports in the available literature on the wetting and interfacial microstructure of Sn-Zn-Cu/Al couples. Therefore, the aim of this work is to study the effect of time on microstructure evolution of the interfaces Sn-Zn-Cu/Cu and Sn-Zn-Cu/Al.

2. Materials and Method

Three alloys based on Sn-8.8Zn (eutectic) containing small addition of copper: 0.5, 1.0, and 1.5 wt.%, respectively, were used for a study of wetting on Cu (99.9%) and Al (99.5%) substrates with the sessile drop method (SD). Solders were prepared by melting pure metals (99.999%) in graphite crucibles, under Ar (99.9992%) protective atmosphere to avoid oxidation. The as-cast alloys were cut into suitable pieces of approximately 0.2 g and degreased with acetone prior the tests. Except for degreasing, there was no special treatment of

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substrates (25 × 20 × 0.2 mm). The wetting tests were carried out at 250 °C on aluminum and copper substrates, for 15, 30, 60, and 180 s, respectively, with the setup earlier used in studies of Sn-Zn-X alloys (Ref 13). The advantage of the setup is that it enables quick transfer of the sample to the already heated furnace, so the heating rate is very fast, as well as the sample is quickly transferred out after the designated time of the test.

Wetting tests were performed with ALU33® flux (~0.05 cm^3) applied to the solder sample and the surrounding part of the substrate. According to ISO 9454-1 this is the 2.1.2-type flux i.e., organic, water-soluble, and activated with halides. Its components are aminoethylethanolamine ($C_4H_{12}N_2O$) and ammonium fluoroborate (NH_4BF_4). The reported wetting angles are the average of three independent measurements on solidified samples after washing flux residue with tap water. After wetting tests, the selected solidified solder/substrate couples were cut perpendicular to the plane of the interface, mounted in conductive resin, and polished for microstructural characterization. The microstructural and energy-dispersive spectroscopy (EDS) analysis was performed with Quanta 3D FEG system, at 20 kV, with the use of the standardless Analysis EDAX System based on Genesis 4000 software.

3. Results and Discussion

3.1 Cu Substrate

The results of wetting angle measurements on Cu substrates are shown in Fig. 1; one could see that for all the investigated solder/substrate pairs the effect of time on wetting angle is negligible. Wetting angles of Sn-Zn-Cu alloys on Cu are generally lower than the wetting angles of Sn-Zn on Cu. On the other hand, the results of different Sn-Zn-Cu alloys on Cu are within the experimental error, which is particularly well seen after 180 s of wetting. The present wetting angle data for Sn-Zn-*x*Cu alloys are significantly lower than the data of (Ref 12) for alloys of similar composition. Similarly, different fluxes used in the present study are most likely the reason why wetting angles of Sn-8.8Zn alloy are lower than the angle of the same alloy on the same type of substrate reported earlier (Ref 13). Such a difference is an indication that deoxidation of metallic surfaces by the flux used earlier (Ref 13) is not sufficient.

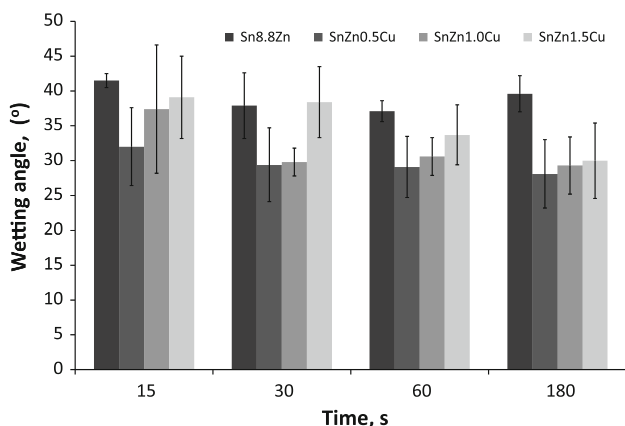

Fig. 1 Wetting angle of Sn-Zn-*x*Cu alloys on Cu pads, *x* = 0.5, 1.0, and 1.5 wt.%

Figure 2 illustrates the interfacial microstructure of Sn-Zn-1.0Cu/Cu couples after 15, 30, 60, and 180 s of wetting, respectively. Similar results were obtained for *x* = 0.5 wt.% Cu and *x* = 1.5 wt.% Cu. One could see that with increasing time of wetting the observed number and thickness of interlayers (formed of reaction products between liquid alloy and solid substrate) varies. More on reactive wetting can be found in review (Ref 14). Although there are a few intermetallic phases in the sub-binaries of Sn-Zn-Cu (namely: $CuZn_4$, CuZn, Cu_5Zn_8, $CuSn_3$, and Cu_6Sn_5), regarding the Sn-Zn/Cu interface Zn has stronger than Sn affinity for Cu, the Cu_5Zn_8 has the lowest Gibbs free energy of formation (Ref 15) and also low activation energy of growth (Ref 16). As shown in Fig. 2, there is a certain difference between the interfaces after 15, 30 s and the interfaces after 60, 180 s of wetting. In the first case (wetting time up to 30 s) a single interlayer can be observed. According to EDS analysis results collected in Table 1 its composition is close to ε-$CuZn_4$ phase assuming that Sn substitutes portion of Zn. In the second case (wetting time 60 s and more) there are two distinct interlayers. The upper layer is clearly scalloped from the side of the solder and according to EDS (Table 1) its composition is close to $CuZn_4$ phase. The thickness of the layer adjacent to the substrate is greater and increases with time of wetting. Its composition determined with EDS matches Cu_5Zn_8, although some content of Sn is observed. This phase is flat on both sides and is known to be a barrier for diffusion of Sn toward substrate thus preventing formation of Cu_6Sn_5. Since it is prone to fractures which enable diffusion of Sn, its growth should be controlled. According to thermodynamic calculations (Ref 17) Cu_5Zn_8 is the phase that should form first, i.e., before $CuZn_4$, at the Sn8.8Zn/Cu interface, however present results after 15 and 30 s of wetting do not confirm this. According to the present results, the ε-$CuZn_4$ phase is the first that is formed at the interface and the γ-Cu_5Zn_8 appears after 60 s of wetting and grows faster than the ε-$CuZn_4$. It can be speculated that γ-Cu_5Zn_8 is formed as a result of copper diffusion from solid substrate to ε-$CuZn_4$ and subsequent nucleation and crystallization within the ε-$CuZn_4$, as in the micrograph (Fig. 3) showing particles of ε-$CuZn_4$ in the matrix of Sn-Zn-1.5Cu solder after 180 s at 250 °C with grains of γ-Cu_5Zn_8 inside. Figure 4 shows the Sn-Zn-1.0Cu/Cu couple after 180 s at 250 °C with the line indicating the position of the original interface at the beginning of wetting. The fact that the thickness of intermetallic layer from the side of solder is approximately the same as thickness from the side of substrate suggests that diffusion of Cu from substrate into γ-Cu_5Zn_8 is similar to that of Zn from the solder.

Generally, if there is more than one intermetallic formed at the interface, the intermetallic in direct contact with liquid at the triple line determines wetting (Ref 18). The fact that wetting angles (Fig. 1) after 15 and 180 s are close means that intermetallic in contact with liquid is the same after 15 and 180 s, which is in agreement with EDS results (Table 1). Thickness of the intermetallic layers in Fig. 2 was measured at 10 spots at least at two separate micrographs per each solder/substrate couple with Axio Vision and later averaged. It has to be noted that for wetting times 15 and 30 s the intermediate layer is thin, as can be seen in Fig. 2(a), (b), with EDS analysis we found its composition to be close to $CuZn_4$. For this reason in Fig. 5 there is only one data bar at 15 and 30 s, while for 60 and 180 s the two data bars represent thickness of $CuZn_4$ and Cu_5Zn_8 layers. After 15, 30 s the thickness of intermetallic layer is similar. Starting from 60 s, with increasing time of

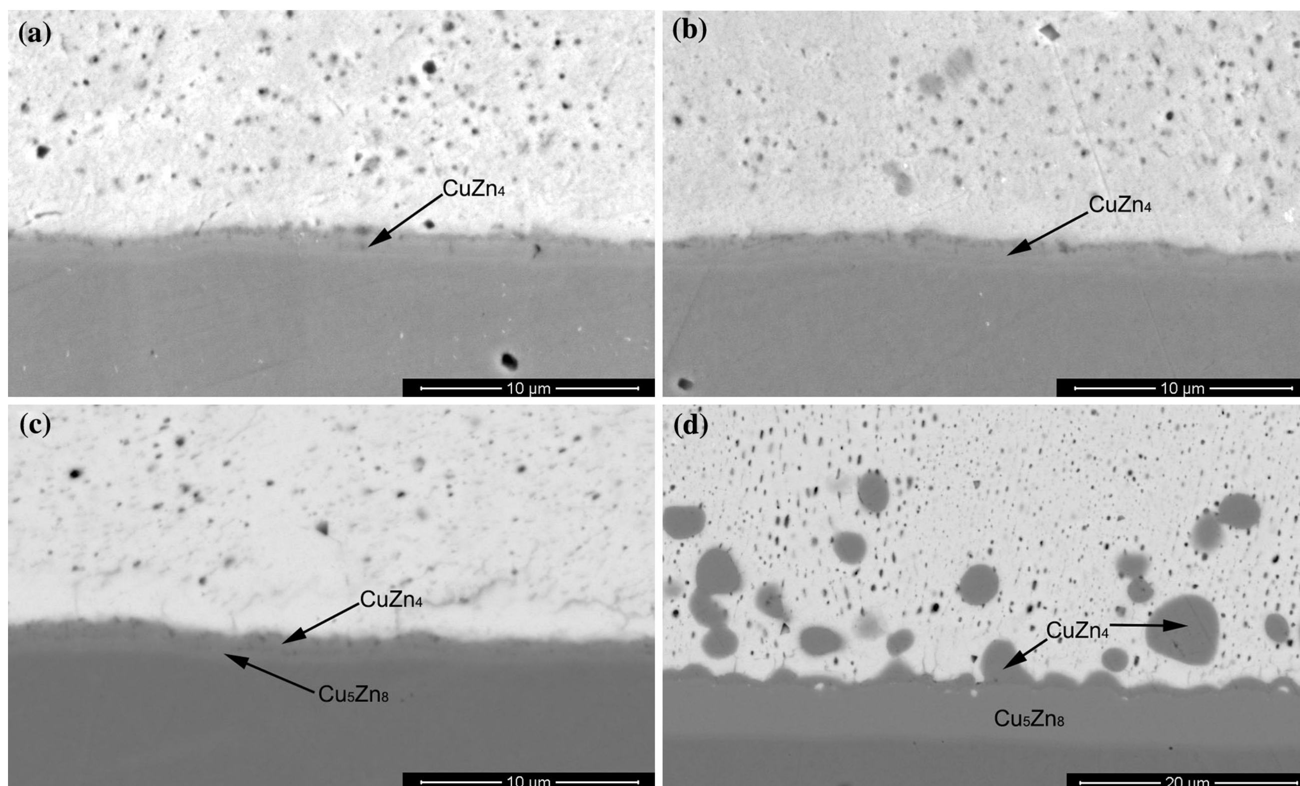

Fig. 2 Interfacial microstructure of Sn-Zn-1.0Cu/Cu couples, (a) 15 s, (b) 30 s, (c) 60 s, and (d) 180 s

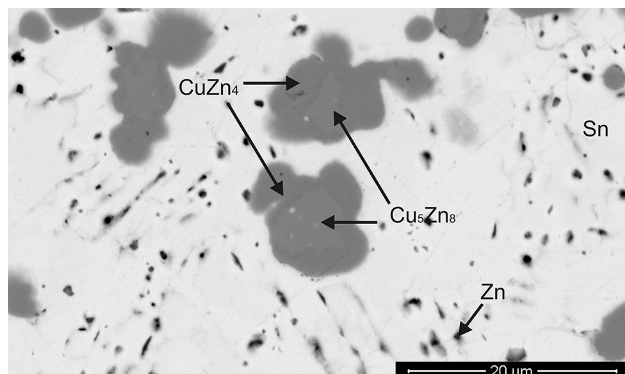

Fig. 3 ε-$CuZn_4$ precipitate in Sn-Zn-1.5Cu solder matrix with γ-Cu_5Zn_8 nucleated inside

wetting thickness of Cu_5Zn_8 layers significantly increases as shown in Fig. 5, whereas thickness of $CuZn_4$ remains small. Similar results were obtained for x = 0.5 wt.% Cu and x = 1.5 wt.% Cu.

Based on the present experimental results the following order of events at the interface of liquid Sn-Zn-xCu (x up to 1.5 wt.%) with Cu substrate is proposed. Initially, liquid Zn from the solder reacts with Cu dissolved from solid substrate and thin $CuZn_4$ layer is formed (Fig. 6a). The most likely reason that it is $CuZn_4$ rather than Cu_5Zn_8 is that the influx of Zn toward substrate exceeds the dissolution rate of Cu. Therefore, each Cu atom is instantly consumed as the fresh Zn atoms are coming from the bulk of the solder. Next (Fig. 6b), Zn diffuses through the $CuZn_4$ toward substrate (at

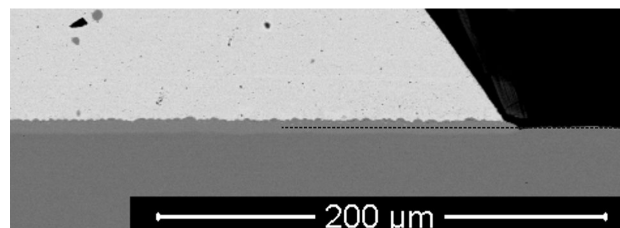

Fig. 4 Sn-Zn-1.0Cu/Cu interface after 180 s of wetting at 250 °C. Dashed line indicates the position of the original interface

the same time Cu diffuses the other way) and the Cu_5Zn_8 is formed adjacent to the substrate. Because of diffusion of Zn from the solder the Cu_5Zn_8 keeps growing and its growth is faster than the $CuZn_4$ as the system tends to reach the minimum energy (Fig. 6c).

In the case of alloys containing 1.0 and 1.5 wt.% Cu after 180 s of wetting, isolated precipitates of $CuZn_4$ phase are present in the solder particularly numerous in the vicinity of the interface. According to Huang et al. (Ref 19) for Sn-9Zn-xCu alloys (x = 0.5-3.0 Cu) Cu_5Zn_8 is the primary crystallizing phase. Although the $CuZn_4$ precipitates seem to be concentrated in the vicinity of interface they are randomly distributed in the solder. If the samples were kept in liquid for a long time sedimentation would be observed due to a density difference between precipitates (for instance, Cu_5Zn_8 is ~8.0 g/cm^3) and the solder matrix (~7.0 g/cm^3) as explained by Song (Ref 15). Song (Ref 15) studied sedimentation behavior of Cu-Zn precipitates in the above-mentioned solders held at 250 °C for 20 min. Since the temperatures at which the intermetallics from

Table 1 **EDS analysis results of the interlayer at the Sn-Zn-*x*Cu/Cu interface (*x* = 0.5, 1.0, and 1.5 wt.%)**

t (s)	Composition (at%) Sn	Cu	Zn	Phase	Figure no.
Sn-Zn-0.5Cu					
15	10.0	22.1	67.8	ε-$CuZn_4$	
30	5.6	22.0	72.4	ε-$CuZn_4$	
60 (#)	4.6	22.2	73.2	ε-$CuZn_4$	
60		35.4	64.6	γ-Cu_5Zn_8	
180 (#)	3.3	21.3	75.4	ε-$CuZn_4$	
180	0.2	35.1	64.7	γ-Cu_5Zn_8	
Sn-Zn-1.0Cu					
15	9.5	27.0	63.5	ε-$CuZn_4$	2(a)
30	9.7	21.7	68.5	ε-$CuZn_4$	2(b)
60 (#)	10.5	24.1	65.4	ε-$CuZn_4$	2(c)
60	2.5	42.8	54.7	γ-Cu_5Zn_8	2(c)
180 (#)	2.6	24.5	72.9	ε-$CuZn_4$	2(d)
180	0.9	34.2	64.9	γ-Cu_5Zn_8	2(d)
Sn-Zn-1.5Cu					
15	8.3	20.6	71.0	ε-$CuZn_4$	
30	5.3	19.5	75.2	ε-$CuZn_4$	
60 (#)	4.2	23.0	72.8	ε-$CuZn_4$	
60	0.5	36.1	63.3	γ-Cu_5Zn_8	
180 (#)	3.2	23.7	73.1	ε-$CuZn_4$	
180		35.1	64.9	γ-Cu_5Zn_8	

(#) Upper layer

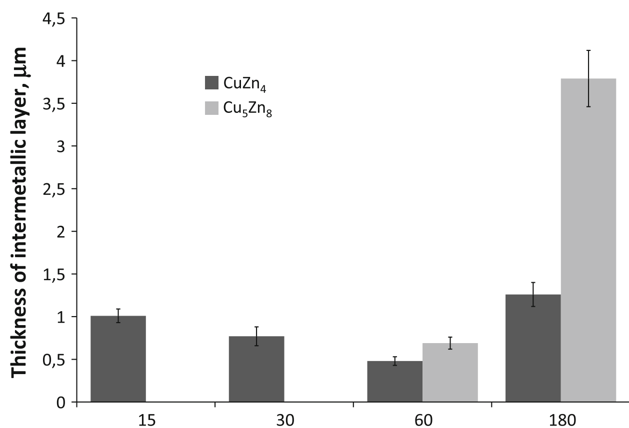

Fig. 5 Thickness of the interlayer at 250 °C at the Sn-Zn-1.0Cu/Cu interface

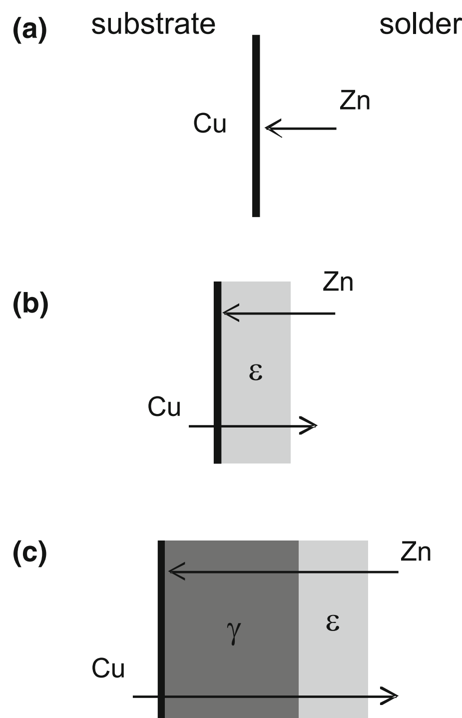

Fig. 6 Sequence of growth of IMC at the Sn-Zn-*x*Cu/Cu interface (*x* up to 1.5 wt.%) at the early stage of liquid-solid interaction. Sketch is not in scale

Cu-Zn crystallize are much higher than 300 °C, it was assumed (Ref 15) that at 250 °C the originally precipitated "as-cast" IMPs would sediment. They observed both $CuZn_4$ and Cu_5Zn_8 in the SnZn-0.5Cu solder but only Cu_5Zn_8 in solders containing ≥ 1 wt.% Cu. In their experiment they found that the "as-cast" IMPs dissolve into Sn-Zn matrix at 250 °C and precipitate again. After 20 min of holding at 250 °C they found rounded precipitates of ε-$CuZn_4$ which sediment in 0.5 and 1.0 wt.% Cu samples, and dendritic precipitates of Cu_5Zn_8 randomly distributed in Sn-9Zn matrix. They explained this dissolution-precipitation behavior by high solubility of Zn in liquid Sn.

3.2 Al Substrate

The wetting angles of Sn-Zn-1.0Cu on Al, shown in Fig. 7, are much lower than the wetting angle of the respective alloy on Cu. Considering wetting of Sn-Zn-1.0Cu on Al substrate, the present data are close to those of Sn-8.8Zn and Sn-Zn-In reported earlier on the same type of substrate and in the

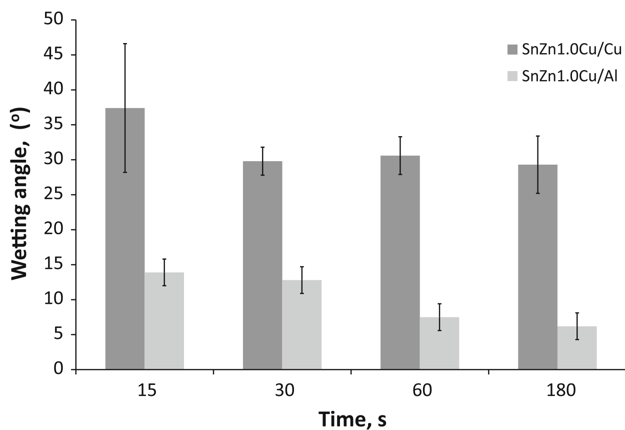

Fig. 7 Wetting angles of Sn-Zn-1.0Cu alloy on Cu and Al pads

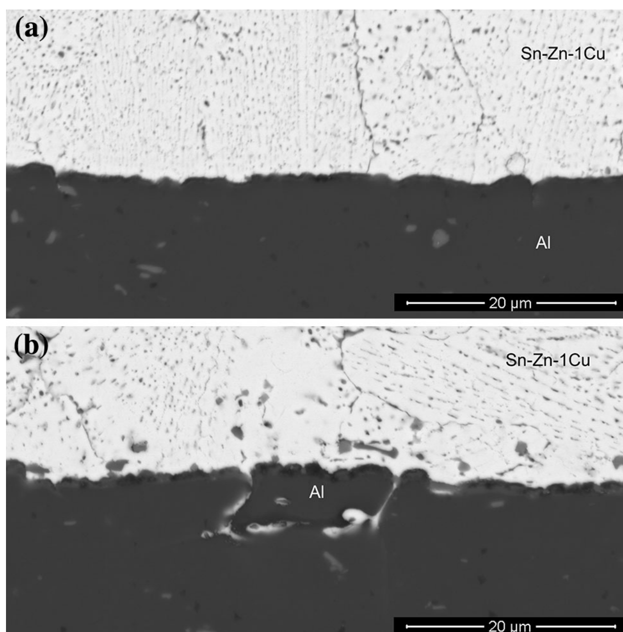

Fig. 8 Interfacial microstructure of Sn-Zn-1.0Cu/Al couples after wetting time of (a) 15 and (b) 180 s

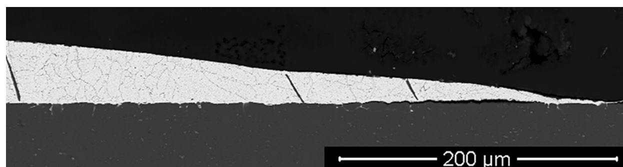

Fig. 9 Sn-Zn-1.0Cu/Al interface after 180 s of wetting at 250 °C

presence of the same flux, but longer time of wetting (Ref 20). Figure 8(a) and (b) illustrates interfacial microstructure of Sn-Zn-1.0Cu/Al couples after 15 and 180 s of wetting, respectively. One could see that the interface is much different than in the case of Sn-Zn-Cu/Cu couples, i.e., no interlayer is observed at solder/substrate interface in agreement with Al-Sn and Al-Zn phase diagrams.

The interface between Sn-Zn-1.0Cu solder alloy and Al substrate is rather rough and the substrate roughness increases with increasing time of wetting. This system is an example of dissolutive wetting, theory and examples of which can be found in review (Ref 21). It is observed that at 180 s, solder grooves and penetrates the substrate along grain boundaries leading to some grains being separated, at least partially, from the surrounding substrate. This kind of dissolution of substrate may lead to Al grains detached and floating near the substrate as shown in Ref 11, 22. The Sn-Zn-1.0Cu/Al interface after 180 s of wetting resemble the Sn-Zn/Al and Sn-Zn-In/Al interfaces after 300 s of wetting and more, discussed in our earlier work (Ref 20, 22). The EDS analysis performed near the interface confirmed that Al dissolved in Sn-Zn-1.0Cu alloy. Nevertheless, as pointed before, one has to be cautious regarding the EDS results because of small size of microstructure features and the associated errors of measurement. Figure 9 shows the Sn-Zn-1.0Cu/Al couple after 180 s at 250 °C. No significant dissolution of the substrate, except for grooves, can be observed. This and the fact that the wetting angle after 180 s is close to that after 15 s is an indication that dissolution kinetics is much slower than the kinetics of wetting.

4. Conclusion

The study of microstructure evolution of the interface between Sn-Zn-*x*Cu alloys on Cu and Al substrates was performed with the sessile drop method. It was found that Sn-Zn-*x*Cu/Al interface is different from Sn-Zn-*x*Cu/Cu. In the first case there are no reaction products at the interface but the alloy dissolves the substrate along grain boundaries similar to Sn-Zn alloys reported earlier. In the case of copper, substrate formation of intermetallic interlayers is observed. Initially (15 and 30 s) only single layer is present with the composition close to $CuZn_4$. After a long time (60 and 180 s) two interlayers can be distinguished at the interface one Cu_5Zn_8 adjacent to the substrate, and the other $CuZn_4$ adjacent to SnZnCu alloy. The thickness of the $CuZn_4$ remains small during the experiment while the thickness of Cu_5Zn_8 increases as observed in the literature.

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