Influence of indium and copper in Sn3.5Ag0.4CuIn solder on its interaction with copper

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Abstract

The influence of indium (0–29.5 wt.%) and copper (0; 0.4 wt.%) in the lead-free Sn3.5Ag solder was studied on both the temperature of transition from the solid to liquid state and vice-versa and the wetting of copper substrate. The influence of indium and copper on the strength of Cu-solder-Cu joints and on the microstructure of the Cu substrate/solder interface is also presented. The results are compared with those for the copper-free Sn3.5Ag solder. Indium and copper were found to decrease the transition temperature from solid to liquid state and vice-versa, to decrease the wetting angle of copper substrates and mildly to decrease the strength of copper-solder-copper joints. Influence of copper is stronger compared with indium.

Key words: lead-free solder, wetting, indium, Cu-solder joint, interface

1. Introduction

An increasing demand for Pb-free solder for electronic industry is due to environmental problems caused by lead. Many of Sn based Pb-free solders were studied to replace Sn-Pb eutectic solder.

There is a strong effort to find a solder with the closest properties to the ones of Sn-Pb solder. Several alloys are considered as promising candidates for replacing this system.

Two eutectic solder systems Sn-Ag [1] and Sn-Zn [2] are providing a melting temperature range similar to the Sn-Pb eutectic (456 K). Melting temperature of Sn-3.5Ag (494 K) is higher than that of eutectic Sn-37Pb and the wetting property is poorer. To improve properties (e.g. wetting, strength of the joints) additional elements as Bi, Cu, In, and Zn [3] are added in the Sn-3.5Ag solder.

Choi et al. [4] studied effect of indium in Sn3.5Ag solder and found out that adding 9 wt.% of In lowers melting temperature of the solder to 483 K. The influence of indium (6.5 and 9 wt.%) on the wetting of Cu substrate by Sn3.5Ag-base solder in the temperature interval 523–593 K and on the solder shear strength of Cu-solder-Cu joints was studied in [5]. The increase of both wetting temperature and amount of indium in the solder was found to decrease the wetting angle of copper. Joint strength moderately decreases with increasing joining temperature and the amount of indium.

Šebo et al. [6] studied the influence of thermal cycling on shear strength of Cu-Sn3.5AgIn-Cu joints for 0; 6.5 and 9 wt.% of indium in the temperature range from room temperature (RT) to 423 K for up to 1000 cycles. Shear strength of Cu-Sn3.5Ag-Cu joints decreases with increasing number of cycles. Addition of indium to the Sn3.5Ag solder gives rise to the increase of shear strength of the joints with increasing number of cycles. The decrease (increase) of the shear strength with increasing number of cycles reflects the increase (decrease) of the Cu_6Sn_5 phase thickness at the boundary between copper substrate and solder.

Another important factor increasing the solder reliability from the electronic properties point of view is the product of interfacial reactions between the substrate and the solder. Wojewoda et al. [7] studied the formation of intermetallic phases in joining process Cu-Cu with the eutectic alloy In-48 at.% Sn. It was

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revealed that η [$Cu_6(Sn,In)_5$] phase appeared as the first one in the solid-liquid reaction between Cu and In-Sn liquid and possessed dual morphology of fine and coarse grains. Chuang et al. [8] studied the Sn-51wt.%In/Cu couple at 423–673 K, and they found ε-$Cu_3(In,Sn)$ and η-$Cu_6(In,Sn)_5$. Šebo et al. [9] studied influence of indium in amounts 4–75 at.% In in Sn3.13Ag0.74CuIn solder on the microstructure of the interface between solder and copper substrate. The phase at the interface is changing with increasing the amount of indium. For In concentration up to 30 at.% In interface is formed by Cu_6Sn_5 phase, although for 30 at.% In there was found a copper rich layer which was adjacent to copper substrate. No X-ray diffraction, however, from this layer was obtained. For higher In concentration (50 and 75 at.% In) an interface is formed by copper rich phase $Cu_{41}Sn_{11}$.

The aim of this contribution is to present the results concerning the influence of indium (0–29.5 wt.%) and copper (0.4 wt.%) on some properties of Sn3.5Ag-base solders.

2. Experimental

Lead-free solders based on Sn-3.5Ag-0.4Cu eutectic alloys were prepared by melting appropriate amount of relevant metals of the purity 99.99 and better. Melting was done in induction furnace under argon atmosphere. Part of each solder was produced in the form of ribbon 5 mm wide and ~ 0.05 mm thick by rapid quenching, the rest was in bulk form. Five different solders were prepared containing constant amounts of silver and copper and 0; 6.5; 9; 14.7 and 29.5 wt.% of indium. The differential scanning calorimetry (DSC) was used to study the melting and solidification phenomena, especially to determine the melting and solidification temperatures of all solders. Continuous heating regimes with the heating and cooling rates ± 10 ℃ min^{-1} were used. The calibration for the heating regime was applied also during the subsequent cooling and each transformation temperature was recalculated.

The bulk form of solders was used for wetting experiments (cube of the solder with the edge length ~ 4 mm); the solder in ribbon form was used for joint preparation. The wetting of copper substrate was studied by sessile drop method in air atmosphere. Copper substrate was mechanically polished and cleaned in alcohol followed by etching in 10 % sulphuric acid in methanol. Substrate as well as the cube of solder prior to insert them into the furnace were daubed by rosin moderately activated flux. The drop of solder was photographed by digital camera at the temperatures 523, 553 and 593 K up to 1800 s and wetting angle was measured by personal computer. Copper substrates for making joint were treated by the same way as for wetting.

Prior to placing with solder in ribbon form into metallic holder they were daubed by rosin moderately activated flux. Holder was placed into the furnace and the joints were prepared at the temperature 553 K and time 1800 s in the air atmosphere. For each set of joints four specimens were prepared. Three of them were used for shear strength measurement by Zwick testing machine using the push-off method, one was used for the microstructure study. Microstructure of the original solders, interface between the substrate and solder after wetting at 553 K for 1800 s and interface between the copper and solder of the joints prepared at the temperature 553 K and 1800 s were studied by light microscopy, scanning electron microscopy (SEM) equipped with energy dispersive X-ray analyser (EDX) and X-ray diffraction. The samples from wetting experiments were cut perpendicularly to reveal the cross section which was further reduced in thickness by removing most of Cu and solder in order to maximize the interface area for X-ray diffraction analysis.

3. Results and discussion

3.1. Differential scanning calorimetry

Results obtained by differential scanning calorimetry are summarized in Table 1. Quantities T_{xm} and T_{xf} are onset temperatures of main melting and freezing effects, respectively.

The melting of the Sn3.5Ag alloy free of copper and indium is characterized by the large endothermal peak starting at $T_x = 499.8$ K (226.8 ℃) having the enthalpy $\Delta H_m = 64.3\ J\ g^{-1}$, however, it is not a single peak. After extensive undercooling of $T_{xm} - T_{xf} = 29.8$ K, the corresponding exothermal effect with the same enthalpy ($\Delta H_m = -63.2\ J\ g^{-1}$) appeared. The significantly sharper peak reflects the typical solidification of the alloy at cooling. The added 0.4 vol.% of Cu decreases the melting temperature 8 K. The temperature of indium added originates in several additive effects accompanying both melting and solidification of the concerned solder. Thus, in the case of 6.5 or 9 vol.% of In minor endothermal effect precedes the main melting peak, which, however, vanishes after one melting-and-solidification cycle. On the other hand, in the case of 14.7 vol.% of In a small peak appears before the main melting effect in each measurement following the first melting-and-solidification cycle. The melting of the solder Sn3.5Ag0.4Cu29.5In is characterized by a complicated complex phenomenon containing minimally four elementary effects, changing intensity proportions by the repeated cycling. Besides, a widespread tail follows the melting for all samples.

Table 1. Onset temperatures and transformation enthalpies of main melting and freezing effects of relevant solders Sn3.5Ag0.4Cu and Sn3.5Ag (in brackets) with various amounts of indium in the as-prepared state

In (wt.%)	0	6.5	9	14.7	29.5
T_{xm} (℃)	218.3 ± 0.2 (226.8 ± 1.4)	202.8 ± 0.1 (211.4 ± 0.9)	200.5 ± 1.0 (208.2 ± 0.6)	184.1 ± 0.3	146.7 ± 0.2
T_{xf} (℃)	197.5 ± 7.7 (197.0 ± 0.6)	184.1 ± 1.2 (185.4 ± 1.7)	183.6 ± 2.2 (177.8 ± 0.5)	178.6 ± 5.0	143.1 ± 4.2
$T_{\mathrm{xm}} - T_{\mathrm{xf}}$ (℃)	21.3 ± 7.9 (29.8 ± 2.0)	18.7 ± 1.3 (26.0 ± 2.6)	16.9 ± 3.2 (30.4 ± 1.1)	5.5 ± 5.3	3.6 ± 4.4
ΔH_{m} (J g^{-1})	65.3 ± 0.4 (64.3 ± 0.7)	59.9 ± 0.1 (58.9 ± 0.3)	60.3 ± 0.5 (58.6 ± 0.1)	57.9 ± 0.3	55.7 ± 0.5
ΔH_{f} (J g^{-1})	–64.9 ± 0.3 (–63.2 ± 0.5)	–58.8 ± 0.1 (–57.8 ± 0.4)	–56.7 ± 0.9 (–55.5 ± 0.5)	–47.9 ± 0.5	–36.5 ± 0.3

Table 2. Influence of copper in Sn3.5Ag solder type in the amount of 0 and 0.4 wt.% Cu containing 0; 6.5 and 9 wt.% of indium on the wetting angle (deg) of copper substrate after 1800 seconds of wetting

In (wt.%)		0			6.5			9	
Temperature (K)	523	553	593	523	553	593	523	553	593
Sn3.5Ag	62	55	49	45	45	43	42	38	36
Sn3.5Ag0.4Cu	28	27	27	26	26	26	26	24	26

The exothermal solidification effects of the SnAgCuIn solders reflect their complex melting; however, the number of the peaks does not always correspond to the number of the endotherms obtained by the preceding heating. This phenomenon might indicate the eventual modification of the phase composition of the samples during the first melting and solidification cycles. Moreover the temperature of several of these exothermal peaks has a very pure reproducibility which in fact might correspond to the variable nucleation effects on the surface of the viscous melt forming several smaller beads during its crystallization.

Both Cu and In were found to decrease the temperatures of the main melting and solidification effects, to minimize dramatically the undercooling by freezing and to decrease slightly the transformation heat. Comparing with the indium and copper free Sn3.5Ag ribbon, the decrease makes 80.1 K (melting) and 53.9 (freezing), resulting in 3.6 K undercooling for Sn3.5Ag0.4Cu29.5In solder only. In this case, the heat of fusion is 55.7 J g^{-1}, while the heat of solidification is only –36.5 J g^{-1}, which difference might signalize that some of the thermal effects accompanying the complex endothermal and/or reversal exothermal transformation either were not accounted or they do not represent the reciprocal melting and solidification.

Comparing with the master alloys, i.e. the bulk form of each sample, the procedure of rapid quenching resulting in the ribbon form did not modify the number and the position of the melting peaks of the solder; it slightly changed the shape of the main melting endotherm, i.e. the proportionality or the quantity of the small peaks. However, after the first melting and solidification cycle of the initially ribbon form solder, its melting thermogram matches that of the bulk sample.

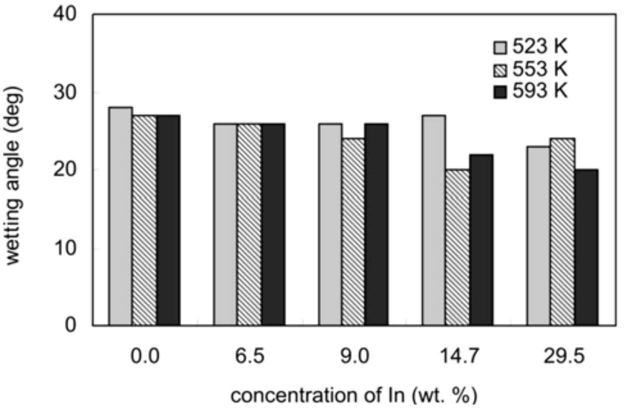
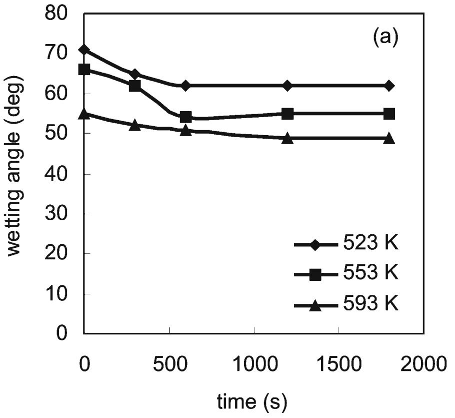


Fig. 1. Wetting angle of copper substrate by Sn3.5Ag0.4CuIn solder in dependence on indium concentration for various temperatures after 1800 s wetting.

3.2. Wetting

Wetting angles of copper by Sn3.5Ag0.4Cu solder with various amounts of indium for temperatures 523, 553 and 593 K after 1800 s wetting are shown in Fig. 1. For comparison, the wetting angles of copper by



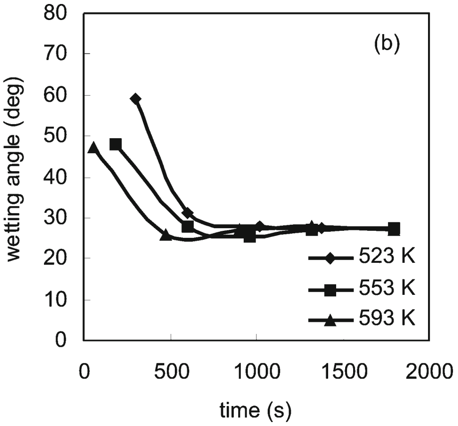


Fig. 2. Time dependence of wetting angle of copper substrate by Sn3.5Ag (a) and Sn3.5Ag0.4Cu (b) solders for various temperatures.

copper-free (Sn3.5Ag) solder [5] with various amount of indium for the same temperatures and time of wetting are given in Table 2. The data given in Fig. 1 and Table 2 show the decrease of wetting angle with the increase of wetting temperature and with the increase of indium content. The influence of indium in copper-free solder is stronger compared with the one in copper contained solder (the decrease of wetting angle is for copper-free solder deeper). An increase of indium from 0 to 9 at.% in copper free solder results in the decrease of wetting angle of 20 degrees at 523 K whereas in the copper containing solder this decrease is 2 degrees only. Figure 2a,b shows the time dependence of wetting angle of copper by copper-free solder (a) and solder containing copper (b). Copper present in solder markedly enhances the decrease of the wetting angle even in indium-free solder (Fig. 2b). Wetting temperature affects the wetting angle much more strongly in the case of copper-free solder. In Sn3.5Ag solder the increase of temperature of 70 K decreases the wetting angle by 13 degrees. For Sn3.5Ag0.4Cu solder practically there is no effect of temperature on the wetting angle. Indium exhibits similar effect on the wetting angle.

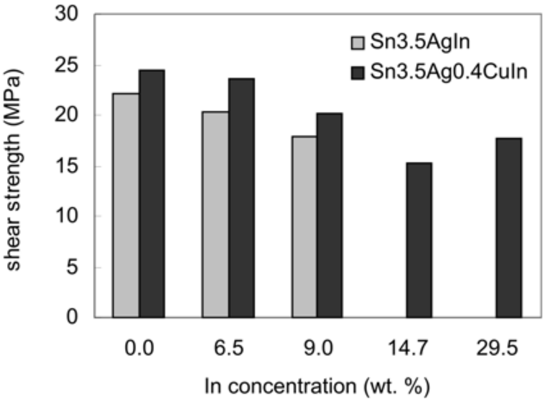


Fig. 3. Shear strength of the joints Cu-Sn3.5AgIn-Cu and Cu-Sn3.5Ag0.4CuIn-Cu for various concentrations of indium in solders made at 553 K and 1800 s.

3.3. Joints strength

Influence of indium in the solders Sn3.5Ag [5] and Sn3.5Ag0.4Cu on the shear strength of the Cu-solder-Cu joint is shown in Fig. 3. Joints were prepared from various solders in between Cu substrates at the temperature 553 K and 1800 s in air atmosphere. Shear strength of the joints decreases with increasing the indium content in the solder. This is valid for both solders investigated. Indium supports the diffusion of copper into the solder; as a result, the amount of Cu_6Sn_5 phase increases. As can be seen from Fig. 3, the presence of copper in the solder increases the shear strength comparing with the solder free of copper. For the highest indium contents the joint strength starts to increase. As it was shown in [6] the decrease or the increase of the joint strength is controlled by the increase or the decrease of the amount of Cu_6Sn_5 phase at the boundary between the Cu substrates and the solder, respectively. One can suppose that the addition of copper into the solder hinders the creation of Cu_6Sn_5. So, the higher bulk copper content in the solder, the higher strength of the joint. For the highest indium concentration (29.5 wt.%) a new copper rich phase forms, partially at the expense of Cu_6Sn_5 phase, and the strength of the joint increases (Fig. 3).

3.4. Microstructure of the solders, interfaces and soldered joints

3.4.1. *Microstructure of the as-prepared solders*

The microstructure of as-prepared Sn3.5Ag0.4Cu solders without indium and with 29.5 wt.% indium is shown in Fig. 4a,b. X-ray diffraction profiles for both solders are presented in Fig. 4c. Profile No. 1 represents Sn3.5Ag0.4Cu solder free of indium. Profile No. 2 characterizes the same solder containing 29.5 wt.%

Table 3. Chemical composition of phases in as-prepared Sn3.5Ag0.4CuIn solders

In (wt.%)	0	6.5	9.0	14.7	29.5
Phases	Sn, Ag_3Sn	Sn, Ag_3Sn	Sn, In_4Ag_9	Sn, In_4Ag_9, $InSn_4$	Sn, $AgIn_2$, $InSn_4$, In_3Sn

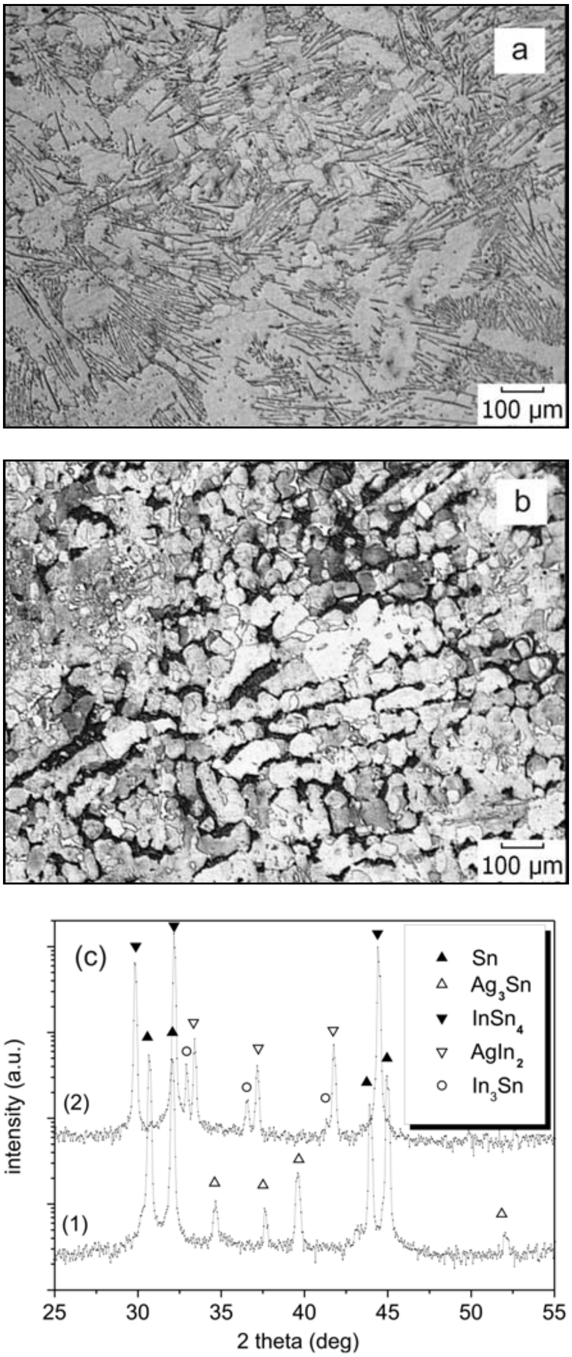


Fig. 4. Microstructures of as-prepared Sn3.5Ag0.4Cu (a) and Sn3.5Ag0.4Cu29.5In (b) solders and the corresponding X-ray diffraction profiles (1 – Sn3.5Ag0.4Cu, 2 – Sn3.5Ag0.4Cu29.5In) (c).

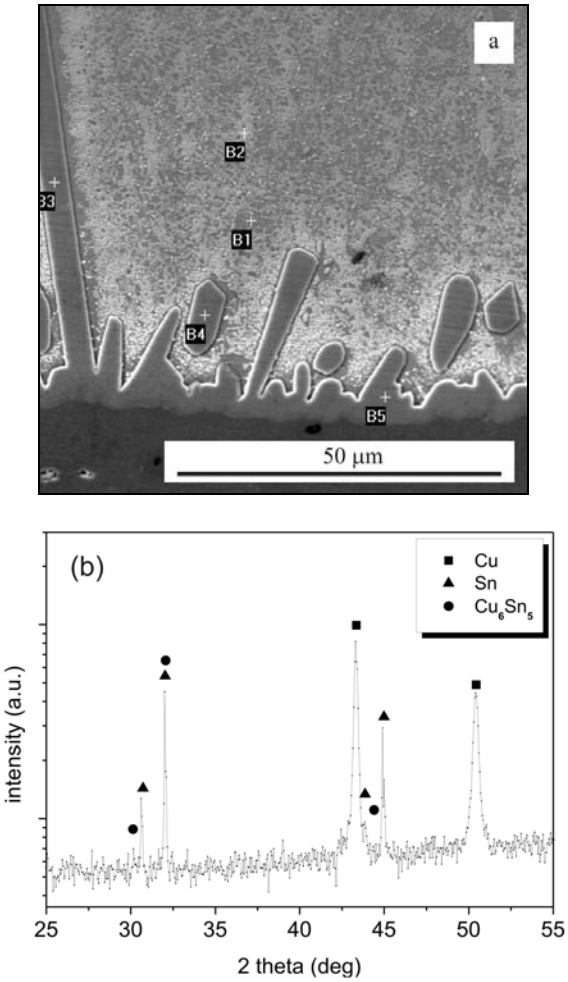


Fig. 5. Microstructure of interface between Cu substrate and Sn3.5Ag0.4Cu (a) and the corresponding X-ray diffraction profile (b).

indium. Phases identified in these as well as in other as-prepared solders are summarized in Table 3.

3.4.2. Microstructure of the interface

Figure 5a shows SEM image of interface between the copper substrate and solder Sn3.5Ag0.4Cu after wetting the copper substrate at 553 K for 1800 s. The composition of several particles measured by energy dispersive X-ray analyser is given in Table 4. Possible phases occurring in the microstructure can be Sn (B1 and B2 points in Fig. 5a) and Cu_6Sn_5 (points B3–B5 in Fig. 5a).

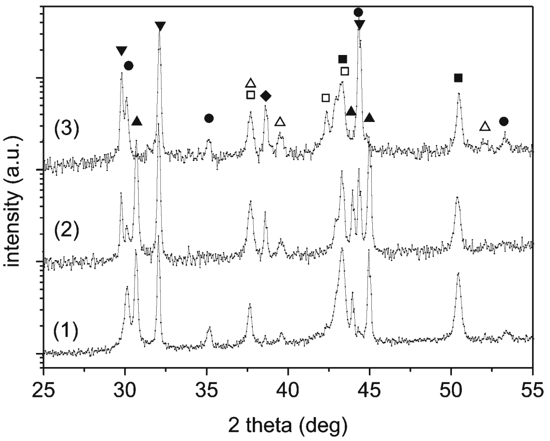


Fig. 6. X-ray diffraction profiles from the interfaces between Cu substrate and Sn3.5Ag0.4Cu solder containing 6.5 (1), 9.0 (2) and 14.7 (3) wt.% indium after wetting at 553 K for 1800 s. The meaning of the symbols used: ▼ – $InSn_4$, ■ – Cu, ▲ – Sn, ● – Cu_6Sn_5, △ – $Ag_3(Sn,In)$, □ – Cu_3Sn, ◆ – In_4Ag_9.

Table 4. Chemical composition of several particles at the interface Cu substrate-Sn3.5Ag-0.4Cu solder. See Fig. 5a

At.%	Cu	Ag	Sn
B1	0.2	1.0	98.8
B2	1.5	0.9	97.6
B3	54.7	1.2	44.1
B4	55.6	0.5	43.9
B5	55.1	0.4	44.4

Table 5. Chemical composition of selected points in the interface Cu substrate-Sn3.5Ag0.4Cu29.5In solder. See Fig. 7a

At.%	Cu	Ag	In	Sn
A1	55.2	0	0.8	44.0
A2	63.2	0	8.7	28.1
A3	0	1.9	1.3	96.8
A4	57.3	0	2.3	40.3
A5	50.9	8.0	7.6	33.5
A6	85.8	0	1.3	12.9
A7	85.1	0	0.8	14.1
A8	83.3	0	3.9	12.8

Figure 5b gives the X-ray diffraction profile taken from the interface Cu-Sn3.5Ag0.4Cu solder after wetting at 553 K for 1800 s. Diffraction confirmed the presence of Sn and Cu_6Sn_5 phases at the interface. Copper lines are from the substrate. Figure 5a shows that the phase at the interface adjacent to the copper substrate is Cu_6Sn_5.

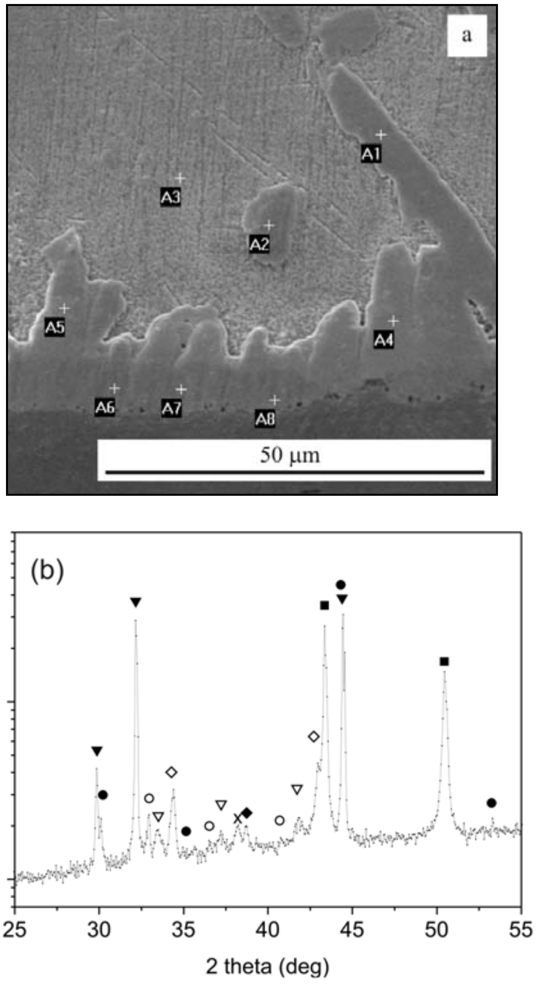


Fig. 7. Microstructure of the interface between Cu substrate and Sn3.5Ag0.4Cu29.5In (a) and the corresponding X-ray diffraction profile (b). The meaning of the symbols used: ▼ – $InSn_4$, ■ – Cu, ◇ – $Cu_{41}Sn_{11}$, ● – Cu_6Sn_5, ○ – In_3Sn, ▽ – $AgIn_2$, ◆ – In_4Ag_9. The mark (X) indicates an unidentified diffraction maximum.

X-ray diffraction profiles from the interfaces between Cu substrate and solders Sn3.5Ag0.4Cu containing indium 6.5 (1), 9.0 (2) and 14.7 (3) wt.% are illustrated in Fig. 6.

The interface between the Cu substrate and the drop made of Sn3.5Ag0.4Cu29.5In solder is documented in Fig. 7a. The composition of several particles measured by EDX analyser is given in Table 5. Possible phases occurring in the microstructure can be Sn (point A3) and Cu_6Sn_5 (points A1, A2, A4, and A5). EDX measurement shows the existence of another layer with higher amount of copper $Cu_{41}Sn_{11}$ adjacent to the copper substrate (points A6–A8). X-ray diffraction from the solder on copper substrate after wetting at 553 K for 1800 s gives these and also other phases: $AgIn_2$, In_4Ag_9, $InSn_4$, In_3Sn (Fig. 7b).

Phases identified in individual In containing solders are given in Table 6. In all these solders (for indium

Table 6. Phases identified in Sn3.5Ag0.4CuIn solders with various amounts of indium as-prepared solders and in interface between the substrate and the drop

In (wt.% in solder)	As-prepared solder	After wetting at 553 K for 1800 s
0	Sn, Ag_3Sn	Sn, Cu_6Sn_5
6.5	Sn, Ag_3Sn	Sn, Cu_6Sn_5, Ag_3Sn
9	Sn, In_4Ag_9	Sn, Cu_6Sn_5, $Ag_3(In,Sn)$, In_4Ag_9, $InSn_4$
14.7	Sn, In_4Ag_9, $InSn_4$	Sn, Cu_6Sn_5, Ag_3Sn, In_4Ag_9, $InSn_4$, Cu_3Sn
29.5	Sn, $AgIn_2$, $InSn_4$, In_3Sn	Sn, Cu_6Sn_5, AgIn2, In_4Ag_9, $InSn_4$, In_3Sn, $Cu_{41}Sn_{11}$

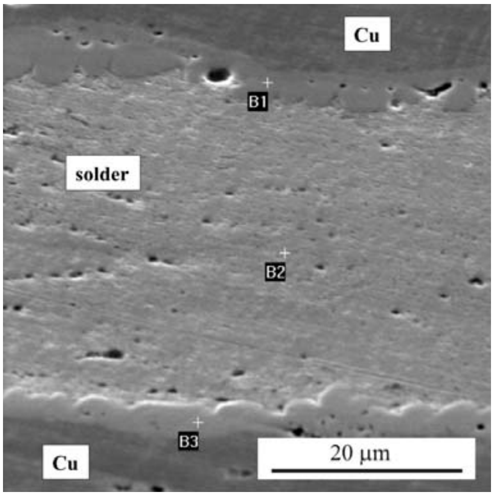


Fig. 8. Microstructure of the joint Cu-Sn3.5Ag0.4Cu-Cu made at 553 K and 1800 s.

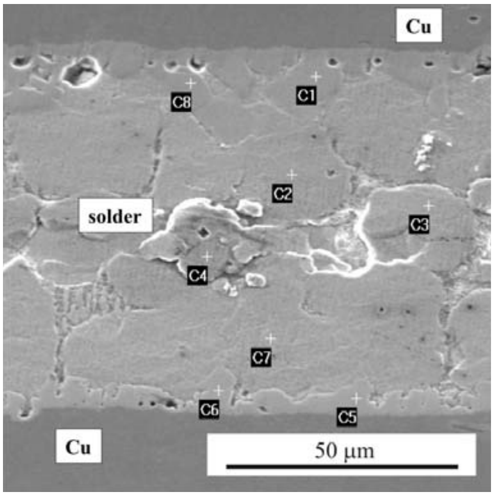


Fig. 9. Microstructure of the joint Cu-Sn3.5Ag0.4Cu-29.5In-Cu made at 553 K and 1800 s.

concentration 0, 6.5, 9.0 and 14.7) phase adjacent to the copper substrate is Cu_6Sn_5. For the highest indium concentration the phase adjacent to the copper substrate is $Cu_{41}Sn_{11}$.

Table 7. Chemical composition of selected points in the joint Cu-Sn3.5Ag0.4Cu-Cu. See Fig. 8

At.%	Cu	Ag	Sn
B1	54.6	0.7	44.7
B2	2.2	8.6	89.3
B3	49.6	1.1	49.4

Table 8. Chemical composition of selected points in the joint Cu-Sn3.5Ag0.4Cu29.5In-Cu. See Fig. 9

At.%	Cu	Ag	In	Sn
C1	61.4	0.3	6.2	32.1
C2	0.9	0.7	5.7	92.7
C3	0	0.2	5.7	94.1
C4	0	1.1	3.5	95.3
C5	68.5	0.2	8.5	22.8
C6	60.7	0.5	4.1	34.6
C7	2.7	0.3	11.2	85.9
C8	60.9	0	10.2	28.9

3.4.3. Microstructures of the joints

Figures 8 and 9 show microstructures of the joints Cu-Sn3.5Ag0.4Cu-Cu and Cu-Sn3.5Ag0.4Cu29.5In-Cu, respectively, both prepared at 553 K and 1800 s. The microstructure of the joint is composed of the interface between Cu substrate and solder on both sides of the joint as well as of the solder alone. Tables 7 and 8 give the chemical composition in given points of the interface and the solder for both joints, respectively.

Possible phases suggested in these points can be the Sn + Ag_3Sn eutectic (points B1 and B3) and Cu_6Sn_5 (point B2) in Fig. 8 and Sn (points C2, C3 and C4), $Cu_6(Sn,In)_5$ (points C1, C5, C6, and C8) and $InSn_4$ (points C7 and C8) in Fig. 9. Phases present in joints for given indium concentrations have practically the same composition as those observed after wetting experiment. Both kinds of the specimens were prepared at the same conditions.

4. Conclusions

Influence of indium and copper on the transition from solid to liquid state, on wetting of copper substrate by solder as well as on the shear strength on Cu-solder-Cu joints was studied for the lead-free Sn3.5Ag-type solder. The microstructure of both as-prepared solders as well as interfaces between solder and copper substrate after wetting at 553 K for 1800 s was also studied.

The obtained results can be summarized as follows:

1. Indium decreases the transition temperatures from the solid to liquid state and vice-versa.

2. Copper in the Sn3.5Ag0.4Cu type solders decreases the transition temperatures from solid to liquid state and vice-versa more expressively compared with indium.

3. Indium and copper in the Sn3.5Ag0.4CuIn solders decrease the wetting angle of copper substrate. Copper possesses more expressive response. The wetting temperature increase lowers the wetting angle.

4. Shear strength of the Cu-solder-Cu joints mildly decreases with increasing indium content in the Sn3.5Ag0.4CuIn solders except for the solder with the highest amount of indium (29.5 wt.% In). The shear strength of the solder with highest In content is slightly higher comparing with the solder containing 14.7 wt.% In.

5. Addition of copper into the solder in the amount 0.4 % moderately increases the Cu-solder-Cu joint shear strength.

6. Cu_6Sn_5 phase arises at the interface between the copper substrate and solder for all indium concentrations. This phase is adjacent to the copper substrate except for the highest indium concentration where the adjacent phase is $Cu_{41}Sn_{11}$.

7. The shear strength of the joints increases or decreases with the decrease or the increase of the Cu_6Sn_5 phase occurrence at the boundary between copper substrate and solder, respectively.

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