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Effect of Indium on Wettability of Sn-2Ag-0,5Cu-1In Quaternary Pb-Free Solder Alloy

Ahmet Mustafa ERER[a,*], Okan UYANIK[a], Yunus TÜREN[b]

[a]Karabuk University, Science Faculty of Physics, 78100 Karabuk, Turkey
[b]Karabuk University, Faculty of Engineering, Metallurgy and Materials Engineering, 78100 Karabuk, Turkey
uyanik_okan@hotmail.com

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Abstract: This study reports the investigation on indium addition into Sn–3Ag-0.5Cu (SAC305) ternary Pb-free solder alloy to evole its varied performances. The effects of indium addition on wettability of the solder alloy was studied. The experimental results exhibited that when the addition of indium was 1.0 wt.%, the change in melting temperature of Sn–2Ag-0.5Cu–1In (SAC-1In) solder was negligible, but the contact angles (θ) of the solder alloy decreased which were measured by using of the sessile drop method at various temperatures (250, 280 and 310 °C) on Cu substrate in Ar atmosphere. Inter-metallic phases, microstructures, and melting temperatures of alloy was characterized by X-ray diffraction (XRD), scanning electron microscope and energy dispersive X-ray spectroscopy (SEM + EDX), and differential scanning calorimeter (DSC), and effects of the amount of In on microstructure were investigated. The lowest θ was obtained as 35,55° at 310 °C. The formation of intermetallic compounds (IMC's) between the Pb-free solder alloy and the Cu substrate was observed. As a result, the studies show that the wetting capability of Sn–2Ag-0.5Cu–1In quaternary solder alloy is better than Sn–3Ag-0.5Cu ternary alloy.

Key words : Wettability, Pb-free Solder Alloys, Contact Angle.

Sn-2Ag-0,5Cu-1In Dörtlü Kurşunsuz Lehim Alaşımının Islatabilirliğine İndiyumun Etkisi

Öz: Bu çalışma, Sn – 3Ag-0.5Cu (SAC305) üçlü kurşunsuz lehim alaşımına indiyum ilavesinin, onun bazı özelliklerine etkisinin araştırıldığını bildirmektedir. İndiyum ilavesinin lehim alaşımının ıslatabilirliği üzerindeki etkileri çalışılmıştır. Deneysel sonuçlar, indiyum ilavesi ağırlıkça %1,0 olduğunda, Sn – 2Ag-0.5Cu-1In (SAC-1In) lehim alaşımının ergime sıcaklığındaki değişimin ihmal edilebilir olduğunu göstermiştir ancak; Ar atmosferinde Cu altlık üzerinde önceden belirlenen sıcaklıklarda (250, 280 ve 310 ° C) sesille damla yöntemi kullanılarak elde edilen lehim alaşımının temas açı değerleri(θ) azalmıştır. Alaşımın metalik fazları, mikroyapıları ve ergime sıcaklıkları X-ışını difraksiyonu (XRD), taramalı elektron mikroskobu ve enerji dağılımlı X-ışını spektroskopisi (SEM + EDX) ve diferansiyel taramalı kalorimetre (DSC) ile karakterize edilmiştir ve indiyum miktarına bağlı mikroyapı üzerindeki etkileri araştırılmıştır. En düşük θ açı değeri 310 ° C'de 35,55 ° olarak elde edilmiştir. Kurşunsuz lehim alaşımı ve Cu altlık arasında intermetalik bileşiklerin (IMC's) oluşumu gözlenmiştir. Sonuç olarak, çalışmalar SAC-1In dörtlü lehim alaşımının ıslatma kabiliyetinin SAC-305 üçlü alaşımından daha iyi olduğunu göstermektedir.

Anahtar kelimeler: Islatabilirlik, Kurşunsuz Lehim Alaşımları, Temas Açısı.

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1. Introduction

The lead element, the toxic substance in the conventional Sn-Pb solder alloy used in the electronics industry has been proved to be harmful to the environment and human health and is restricted for use under the RoHS directives [1]. During the past decade, the Sn-3.0(wt.%) Ag-0.5Cu composition has been a appropriate material to the electronic industry as a substitute of Pb-free solder alloy. Sn-Ag-Cu (SAC) Pb-free solder alloys are extensive utilized in the electrical and electronics industries due to their solderable and mechanical properties. Therefore, the use of SAC solder alloys in place of traditional Sn - Pb eutectic solder alloys have been supposed appropriate by many surroundings. On the other hand, due to the high cost of silver and the melting of lead-free solder alloys at elevated temperatures, it is also necessary to work on the SAC305 solder alloy [2]. Low Ag concentration lead-free solder alloys have been reported to have improved durability reliability. The use of the Ag element in the SAC triple eutectic lead-free solder alloy causes the melting point of the alloy to decrease and the mechanical properties to increase. As the work done, it can be said that indium add it onto solder alloys decreases the melting temperature and increase the wettability [3]. On the other hand, the alloy element may have other impressions on the features of the alloys, like mechanical and microstructural features, when forming solder alloys, the RoHS directive must be followed and solder alloys with a low melting point must be formed [4-7]. Because of the low Ag content, SAC solder alloys have a superior property in forming a more brittle Ag_3Sn intermetallic phase during soldering. This study ensures developed wettability results obtained in low Ag and In-containing Sn-2 (wt%) Ag-0.5 (wt%) Cu-1 (wt%) In (SAC-1In). [8-10]. Microstructural properties and interfacial reactions are also investigated. The optimal value of In (1.0wt.%) supplemented relating to the wettability is so recommended.

2. Experimental Methods

In this project, while amount (1wt.%) of indium into the near-eutectic SAC305 alloy were added, new quaternary lead-free solder alloy was produced. The chemical compositions obtained from the XRF analyzes of the studied alloy is listed in Table 1. The wettability tests were realized for the SAC-1In Pb-free solder alloy by using the sessile drop technique [11]. Through the medium of this technique, the SAC-1In solder alloy were dropped on copper substrate at various temperatures of 250^{o}C, 280 ^{o}C, and 310 ^{o}C. Casio Made Pro EX-F1, 300 FPS Model camera utilized to capture views of drops at the 0th, 5th, 10th, 15th, 30th, 60th, 90th, 120th, 150th, 300th seconds and these images were transferred into AutoCAD-2017 Software to measure contact angles of each drop from the right and left profiles.

Table 1. The chemical compositions of alloy (wt.%).

Alloys	Al	Cu	Ag	In	Pb	Cd	Sn
Sn-2Ag-0,5Cu-1In	0.0302	0.4363	2.0822	0.9940	0.0217	0.0839	96.3202

As a result of these processes repeated at least three times foreach temperature, mean angle values were calculated and new diagrams were drawn through the Sigma Plot 12.0 Software. In order to determine melting temperatures of alloy, the Differential Scanning Calorimetry (DSC) analysis were carried out on specimens in maximum 45 mg weight and at 50 ^oC – 400 ^oC temperature range (6min). Standard metallographic processes were carried out for microstructure examinations. Prepared specimens were etched with 100 ml H_2O, 2 ml HCl, 10 g $FeCl_3$ solution for 45 s. The etched specimens were characterized by the scanning electron microscope with energy dispersive X-ray spectroscopy (SEM+EDS) and X-ray diffraction (XRD).

3. Results and Discussion

In different experiments, wetting angles and relaxation curves are shown in Fig. 2 at varied temperatures. As the temperatures are 250 ^oC, 280 ^oC and 310 ^oC, it can be said that for SAC-1In the balance contact angles at temperatures of 44.85°, 43.06° and 35.55° decrease, respectively. The rates of the contact angle represent the degree of wettability [12]. Moser et al. [13] committed that when the wetting angle of a solder is between 0° and 30°, it can be said that the wetting quality is very good. It is good and receivable when the wetting angle is between 30° and 40°. However, when the wetting angle passes over 70°, the solder is unacceptable. Furthermore, the wettability grade for the SAC-1In / Cu system in the Ar atmosphere is quite good.

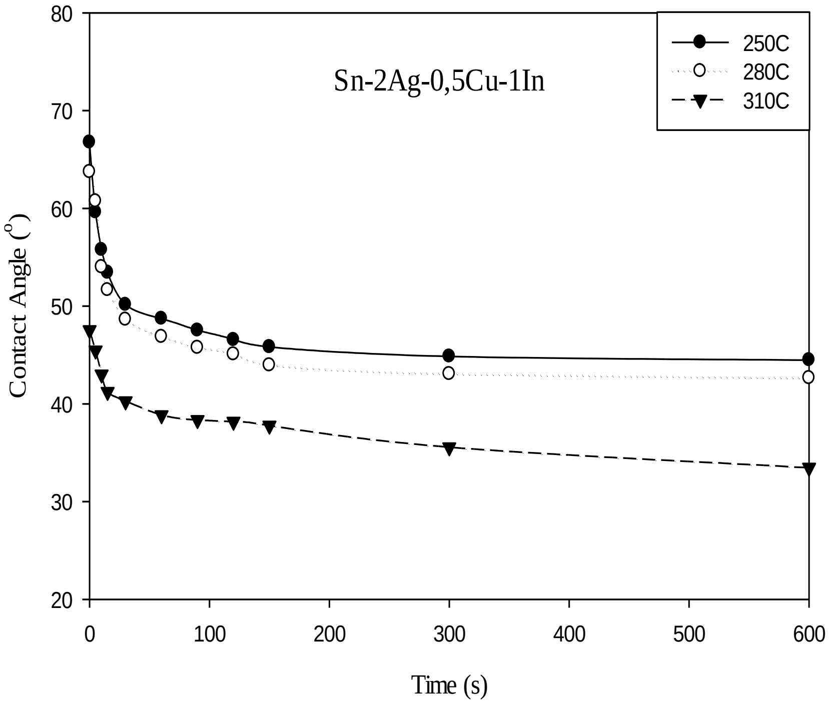

Figure 1. Time dependence of the contact angle of SAC-1In and alloy on Cu substrate at tests temperatures, 250 ^oC, 280 ^oC and 310 ^oC.

In Figure 2, DSC analysis show that the melting temperature of SAC-1In alloy. Thus, the wettability of SAC-1In alloy is good to replace SAC305 solder alloys.

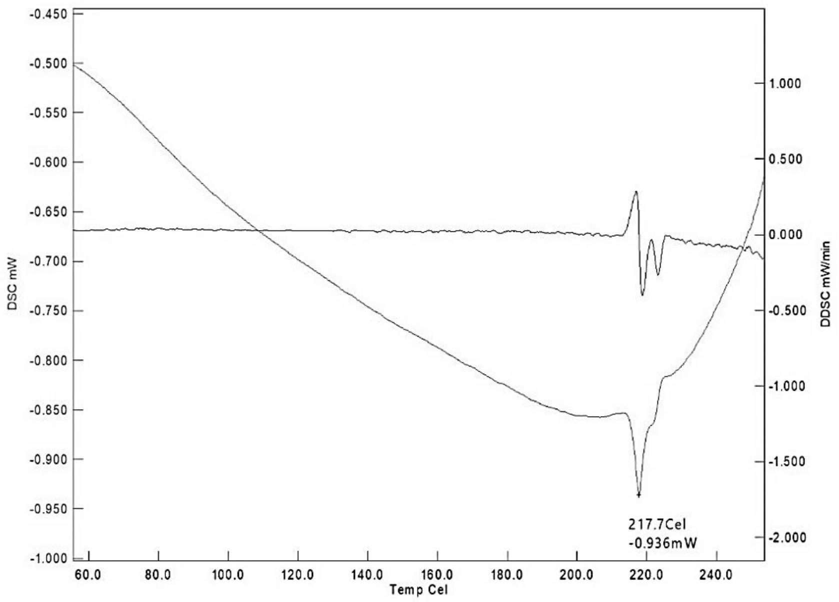

Figure 2. DSC analysis results of SAC-1In alloy.

Figure 3 shows that there are given XRD resultof the near-eutectic SAC305 alloy with added one amount of In (1 wt.%). It was determined that Cu_3Sn, Cu_6Sn_5, and $Ag_3(Sn,In)$ phases were formed in the alloy structure in accordance with the XRD results.

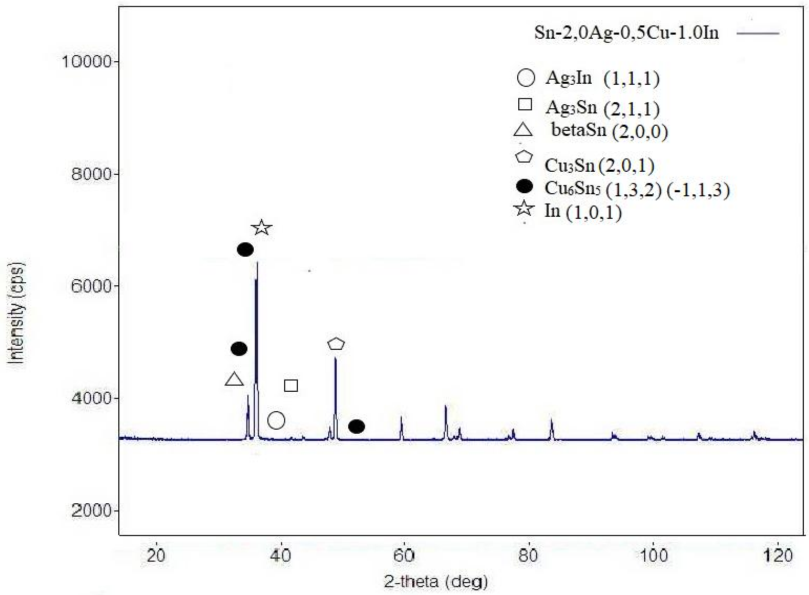

Figure 3. XRD result of the SAC-1In alloy.

Figure 4 shows the addition of indium (1% wt.) and SEM interface changes at three different temperatures. Intermetallic layers are found in the interface, which is a combination of SAC-1In / Cu matrix. These intermetallic layers formed are intermetallic components at the interface (Cu_6Sn_5, Cu_3Sn and Ag_3(Sn, In)) during the soldering process [14].

As can be seen from the SEM images, the interface details are more clearly observed as the growth rate increases. The wetting angle increases with the diameter of the intermetallic coats composed at the interface as the measurement temperatures (250, 280 and 310 °C) increase. In addition, at 310 °C, where the measurement temperature is the highest, it is seen that the interfacial intermetallic layer and substrate interface roughness become more uniform by decreasing the roughness.

On the other hand, it was also determined that the intermetallic layers were separated from each other at a temperature of 310 °C and moved within the solder alloy. This means that the diffusion rate increases at the interface with the increase of the process temperature Interfacial growth during soldering, thermal morphology and interrelationships of intermetallics are very important.

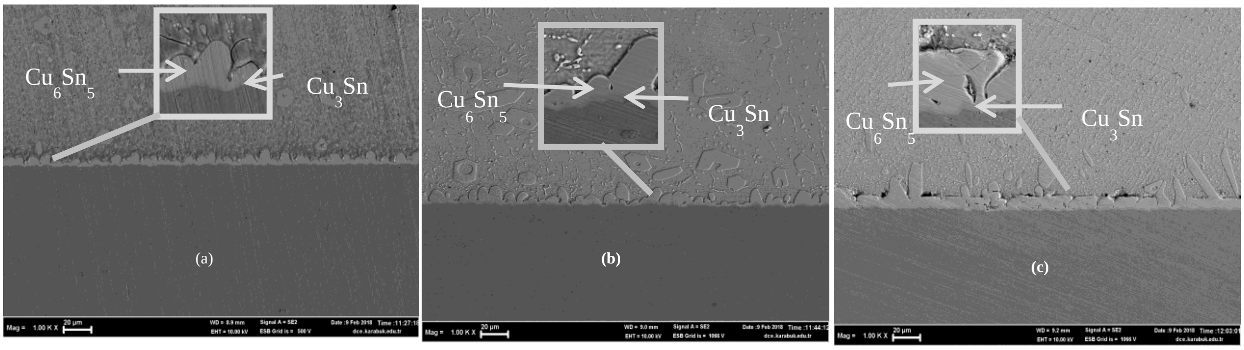

Figure 4. SEM images of the Sn-2.0Ag-Cu-1In/Cu interfaces subsequently of the drop process SAC-1In alloy at a)250°C, b)280°C, c)310°C.

The formation of intermetallics in Sn-Ag-Cu-In / Cu soldering systems occurs in two stages; Firstly, Cu_6Sn_5 occurs at the interface during soldering, while in the other step Cu_3Sn is formed between Cu_6Sn_5 and Cu substrate [15]. Consequently, of the more rapid diffusion of Cu atoms relative to the Sn atoms, the interface causes the formation of a rich intermetallic layer by the element Cu.

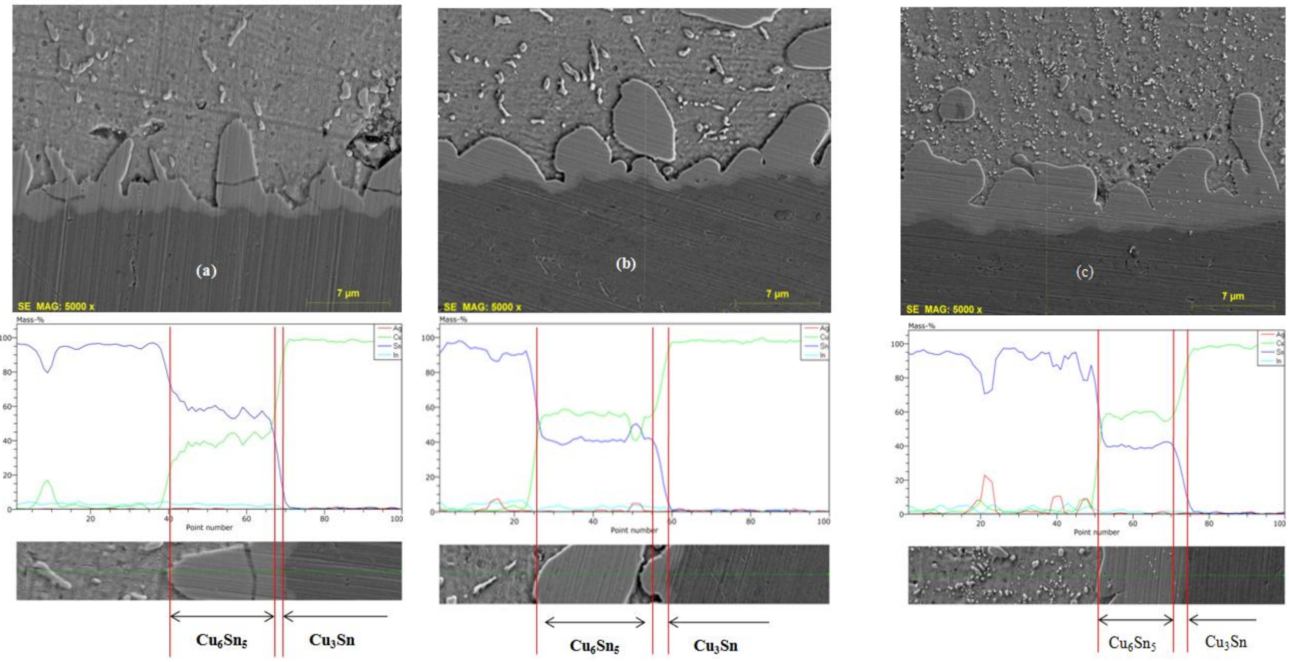

Figure 5. Element distributions across the SAC-1In/Cu interface, a)250°C, b)280°C, c)310°C measured by EDX.

The cross-sectional chemical composition is presented in Fig. 4 by EDX analysis. Indium has negligible effect on chemical composition of intermetallic phases. This condition is most likely to be caused by low concentration. The item is mostly found in the solder bulk. The Cu_3Sn layer thickness is greater than the Cu_6Sn_5 layer composed at the SAC-In / Cu interface.

4. Conclusions

The chemical compositions of the SAC-1In quaternary solder alloy which have produced for this project are given in Table 1. The wettability of SAC305 near-eutectic alloys with the supplement of In was enquired with the use of sessile drop technique. In all of the repeated experiments, it was ensured that all test conditions were at the optimum level and the same, ie. the combination of the alloys, the naivety and preparation of the substrate, the temperature of the wetting test and the amount of Argon gas applied were kept at the same values for each experiment. It was found that SAC-1In alloy shows good wetting of the copper substrate in Ar atmosphere. The contact angles for the alloys obtained from the sessile drop technique. Growing the supplement of In (up to %1) resulted in the lowering of the contact angles. The lowest θ was obtained as 33.47°for SAC-1In alloy at 310°C. Also, Micro-structures, intermetallic phases taking place in the structure (IMC) and melting temperatures of alloys were defined, and the results obtained have been correlated with recent studies of other research groups and general references to the characteristics of Pb-free solder alloys. Interfacial reactions between the working alloys and Cu substrate lead to IMC's which are Cu_6Sn_5, Cu_3Sn and Ag_3(Sn, In). The interface layer taken place of double parallel layers – Cu_3Sn and Cu_6Sn_5. The Cu_3Sn layer is composed between $Cu_6Sn_5$and the Cu substrate and it's diameter is thinner comparing to Cu_6Sn_5 layer composed at the SAC-In/Cu interface. If the Ag and In amount are kept at optimum values, the SAC-1In solder alloy can be a firm applicant to relay the conventional Sn-3.0Ag-0.5Cu solder.

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