

Effect of Flux on the Wetting Characteristics of SnAg, SnCu, SnAgBi, and SnAgCu Lead-Free Solders on Copper Substrates

MARIO F. ARENAS,¹ MIN HE,¹ and VIOLA L. ACOFF^{1,2}

1.—Department of Metallurgical and Materials Engineering, The University of Alabama, P.O. Box 870202, Tuscaloosa, AL 35487-0202. 2.—E-mail: vacoff@eng.ua.edu

The effect of flux on the wetting characteristics of four lead-free solders, Sn-3.5Ag, Sn-0.7Cu, Sn-3.5Ag-4.8Bi, and Sn-3.8Ag-0.7Cu (wt.%), on copper substrates have been studied at 240, 260, and 280°C. The fluxes investigated were rosin (R), mildly activated rosin (RMA), and activated rosin (RA). The wetting tests were conducted using the sessile-drop method. Results showed that fluxes significantly affect the wetting properties of the solders. Contact angles ranging from 10° to 30° for RMA, 20° to 30° for RA, and 35° to 60° for R were obtained. The effect of temperature on contact angle depended on the type of flux used. The contact angle decreased with increasing temperature; however, in some cases the contact angle was independent of temperature. The Sn-3.5Ag-4.8Bi exhibited the lowest contact angles indicating improved wettability with addition of bismuth. The microstructure of the solder/copper interface was analyzed by scanning electron microscopy.

Key words: Pb-free solder, contact angle, flux, copper substrate, Cu-Sn intermetallics

INTRODUCTION

Sn-Pb solders have been extensively used in the assembly of modern electronic circuits. However, because lead and Pb-containing compounds have been regarded as chemicals posing great threats to human life and the environment, research activity for development of substitute alloys has been intense. At present, Sn-rich alloys with such alloying elements as Ag, Cu, and Bi are the most promising candidates for replacing Sn-Pb alloy in electronic assemblies.^{1,2}

For most soldering processes, the use of fluxes is needed to clean and prepare the surface of the substrate so that it can be wetted by molten solder. Fluxes react with any oxide or tarnish present and form a continuous film over the surface that inhibits access of oxygen to the substrate. Fluxes also affect the different interfacial tension forces present during wetting because of the change of the environment at the solder/substrate interface. In the presence of a flux, the classical Young-Dupré equation for the balance of surface tensions at the interface takes the following form

$$\gamma_{fs} = \gamma_{ls} + \gamma_{fl} \cos \theta \quad (1)$$

where γ_{fs} is the surface tension of the flux/substrate interface, γ_{ls} is the interfacial energy between the liquid solder and the substrate, γ_{fl} is the surface tension of the flux/liquid interface, and θ is the equilibrium contact angle formed at the solder/substrate/flux triple point. The flux/substrate surface tension (γ_{fs}) depends upon the cleanliness of the substrate surface, γ_{ls} is intrinsically related to the metallurgical reaction at the solder/substrate interface, and γ_{fl} is a function of the chemistries of the flux and solder.³ It is known that fluxes can reduce the surface tension of the solder/substrate/flux triple point,⁴ thus reducing the contact angle. However, the extent of this reduction depends on the compositions of both the solder and the flux used.

Fluxes for the soldering of electronic components with Sn-Pb alloys have been primarily based on rosin-based mixtures. Rosin is a natural product derived from pines trees consisting of roughly 80% of resin acids having a tricyclic diterpene structure with formula $C_{19}H_{29}COOH$. The effect of rosin fluxes on the wettability of Sn-Pb alloy over typical substrates, such as copper, has been extensively studied.⁴ However, information on the relationship between wetting performance of lead-free solders and flux used is scarce. Lead-free soldering

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processes face additional challenges due to the higher temperature of operation and reduced wettability compared to conventional Sn-Pb solder alloys.⁵ The flux formulation will require higher flux capacity, higher oxygen barrier capability, and higher thermal stability. Therefore, development of suitable fluxes is important for implementation of lead-free soldering alloys in electronic applications.

Solder wettability can be experimentally assessed by measuring the contact angle of wetting. In addition to the type of flux used, the measurement of the contact angle values involves control of a number of parameters that can affect the results, namely, surface roughness, time, and temperature of measurements. A review of the literature on data of contact angles for Pb-free solder alloys shows that the reported values are quite disparate and hard to compare to each other, due to the different conditions used during testing. Furthermore, there is a need to obtain data for the alloys that are leading candidates to replace lead-containing solders in microelectronic applications. These alloys are based on the binary eutectic systems Sn-3.5Ag and Sn-0.7Cu (wt.%) and the ternary systems Sn-3.5Ag-0.7Cu and Sn-3.5Ag-4.8Bi (wt.%). The objectives of this study are (1) to determine the wetting behavior of selected lead-free soldering alloys as a function of flux and process temperature by measuring the contact angle on copper substrates and (2) to characterize the structure of intermetallic compounds formed at the solder/substrate interface.

EXPERIMENTAL PROCEDURES

Four different lead-free alloys were used for this investigation: Sn-3.5Ag, Sn-0.7Cu, Sn-3.8Ag-0.7Cu, and Sn-3.5Ag-4.8Bi (all in wt.%). These alloys, except Sn-3.5Ag-4.8Bi, were obtained from Johnson Manufacturing Company (Princeton, IA) in the form of solid wires. The Sn-3.5Ag-4.8Bi alloy was prepared from pure metals (purity > 99.9%) using arc melting under an argon atmosphere. The alloys were remelted three times in order to obtain homogeneous structure and composition. The conventional solder employed in this study was commercial Sn-37Pb wire. The solder alloys were remelted in an argon atmosphere and cast into cylindrical bars 0.3 cm in diameter. Small solder beads (0.3 cm height × 0.3 cm diameter) were cut from the cylindrical bars for reflowing into molten drops. The beads were cleaned with acetone in an ultrasonic bath. The substrate consisted of Cu plates of 99.9% purity and area 2.5 cm × 2.5 cm. These small plates were polished sequentially with SiC paper up to 1200 abrasive number, which corresponds to a particle size of 5 μm, and then cleaned ultrasonically in acetone and alcohol for 10 min.

The measurement of the contact angle was performed using the sessile drop method in two different environments. One set of experiments was performed under vacuum (10^{-3} Pa) with no fluxes in a specialized furnace manufactured by Centorr

Associates, Inc. Heating was achieved by increasing the current passing through the heater until the desired temperature was reached. The wetting tests were performed at a temperature 10°C above the nominal melting point of the solder tested. This temperature was held for 2 min., allowing spreading of the solder onto the substrate, and then the sample was furnace-cooled to room temperature. In another series of experiments, wetting was carried out on a hot plate in air with the use of fluxes. Three different commercial liquid fluxes were used: rosin (R), mildly activated rosin (RMA), and activated rosin (RA). Several drops of flux from a pipette were placed on each copper plate to form a thin layer of flux on the copper surface. Two drops of flux were also placed on each solder alloy. In preparation for reflow, the substrate containing the solder and the flux was first preheated to 100°C on a hot plate to evaporate the volatile constituents of the flux. It was then moved to a second hot plate set at the desired reflow temperature. Three different temperatures were used for a heating time of 2 min.: 240°C, 260°C, and 280°C. Following the time of reflow, the specimen was quickly removed, allowed to solidify, and later quenched to room temperature.

The morphology and contact angle of the solder bead on Cu were studied using a video camera that filmed the spreading process. The camera was connected to a computer and a video recorder, enabling time and temperature stamps on each image. A frame grabber system was used to capture individual video frames as TIFF files for further processing. Contact angles were measured from the shape of the drop in the pictures. For each set of experimental conditions, the contact angle measurements were repeated three times and the mean value is reported. Experimental error was determined using statistical analysis. A schematic of the apparatus is shown in Fig. 1.

After each test, the solder drop was cut perpendicular to the interface, mounted in conductive resin, and polished to examine the possible interfacial reactions. Microstructural analysis was performed using scanning electron microscopy (SEM) equipped with energy-dispersive spectroscopy

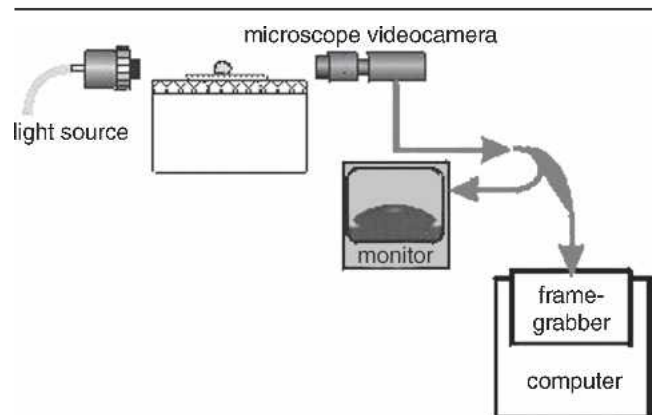


Fig. 1. Schematic of the sessile drop method used in this study for measuring contact angles.

(EDS). All SEM images were obtained using the backscattered electron mode. SEM/EDS analysis was utilized to investigate the spatial distribution of chemical species for identification of the intermetallics at the interface.

RESULTS

Contact Angle Measurements

Figure 2 shows the results of contact-angle tests using vacuum atmosphere for the four types of lead-free solders and Sn-37Pb solder. The contact angles for the lead-free solders investigated range from 33° to 42°, which are significantly higher than the value of 13° obtained for the conventional Pb-Sn alloy. The contact angle value measured for the Sn-37Pb alloy is in close agreement with previous results published in the literature.⁴ The higher contact angles observed for lead-free solders might be explained by the increase of the solder-vapor interfacial tension caused by the absence of lead, as concluded by Vianco and Frear.³ These results also indicate the need to use suitable fluxes to improve the wetting behavior of lead-free soldering alloys.

Figure 3 shows the results of contact angle measurements for Sn-3.5Ag solder as a function of flux. In this figure, the magnitudes of the contact angles ranged from 17° to 63°. It was evident that the contact angles for flux R were considerable higher than

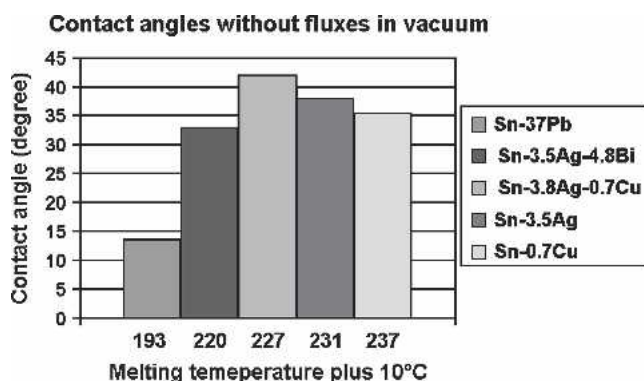


Fig. 2. Contact angles of solders on copper substrates measured in vacuum atmosphere. Temperatures were 10°C higher than the nominal melting point of the solders.

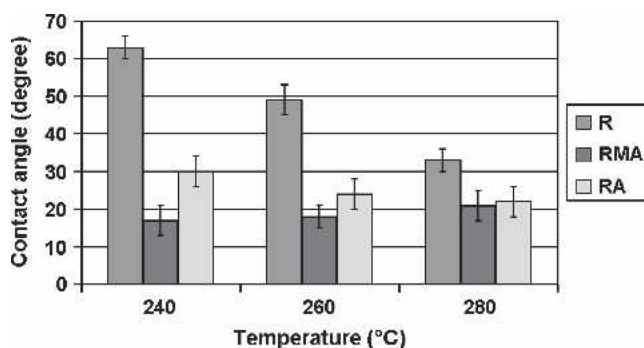


Fig. 3. Contact angles as a function of flux for Sn-3.5Ag solder on Cu substrate.

those observed for fluxes RMA and RA, in the range of temperatures investigated (240–280°C). This indicates that fluxes containing activated elements in their structures are necessary to improve the wettability of lead-free soldering alloys. RMA and RA fluxes contain ionic additives that clean the surface of the substrate better and faster. Interestingly, the RMA flux yielded slightly lower contact angles compared to the RA flux at 240°C and 260°C. However, the difference between RMA and RA was within the range of experimental error at 280°C. Figure 3 also shows that the increase in temperature results in lower contact angles. This seems to be in agreement with the observed reduction in surface tension of solders, and consequently the solder-substrate surface tension (γ_{ls}), when the temperature is increased.⁶ A smaller γ_{ls} results in an increase in the $(\gamma_{fs} - \gamma_{ls})$ term in Eq. 1, and accordingly the contact angle θ is reduced.

Figure 4 illustrates the contact angles obtained after wetting of Sn-3.5Ag-4.8Bi alloy as a function of flux used. In this figure, contact angles ranging from 15° to 20° are observed for RMA and RA fluxes. These values approach the typical contact angles reported for Sn-Pb alloy,⁷ thus indicating good wettability. Moreover, R flux yielded larger contact angles varying from 38° to 45°. The improved wettability of this alloy can be attributed to the addition of bismuth in the solder composition as a previous report has shown.³ RMA and RA fluxes gave similar contact angles except at the temperature of 240°C, which resulted in a slightly lower contact angle for RMA flux. A close examination of the data as a function of temperature show that, for R and RMA fluxes, the contact angle is relatively independent of temperature, because differences appeared to be within the experimental scattering. However, when RA flux was used, the contact angle decreased as temperature increased from 240°C to 260°C. Further increase to 280°C did not produce a significant change in contact angle.

Figure 5 shows the contact angle measurements for the ternary Sn-3.8Ag-0.7Cu alloy. In this case and similarly to other alloys studied, the use of R flux resulted in higher contact angles, indicating poor wetting. However, a significant improvement in

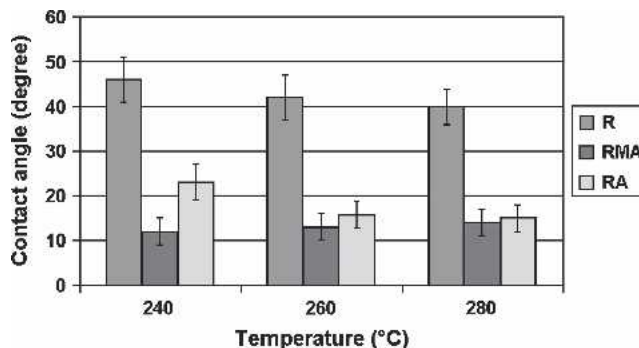


Fig. 4. Contact angles as a function of flux for Sn-3.5Ag-4.8Bi solder on Cu substrate.

wetting was achieved using RMA and RA fluxes. RMA yielded contact angles in the range of 14° to 20°, whereas the values for RA ranged from 21° to 31°. At a temperature of 280°C, both fluxes yielded the same contact angle. The increase in temperature resulted in a small decrease of the contact angle for fluxes R and RA. However, in the case of flux RMA, the contact angle measured was independent of temperature. Because these contact angles were similar to those obtained for Sn-3.5Ag, it can be concluded that copper additions of 0.7% have no effect on wettability. Lower contact angles were obtained using RMA flux at the temperatures of 240°C and 260°C; however, at 280°C, both fluxes yielded the same contact angle.

Figure 6 shows the contact angles measured for Sn-0.7Cu solder. This figure reveals a similar trend in the results compared to the previous alloy: high contact angles for R flux (53–62°) and lower values for RMA and RA fluxes (17–30°). The lowest contact angles were obtained when RMA flux was used at 240°C and 260°C. The variation in contact angle was almost independent of temperature for the three fluxes analyzed. It was expected that in all cases the increase in temperature would result in greater spreading; however, it is possible that for some fluxes, higher temperatures will increase the flux–solder surface tension due to chemical reaction. Similar findings have been reported by Loomans et al. for some solder systems.⁸

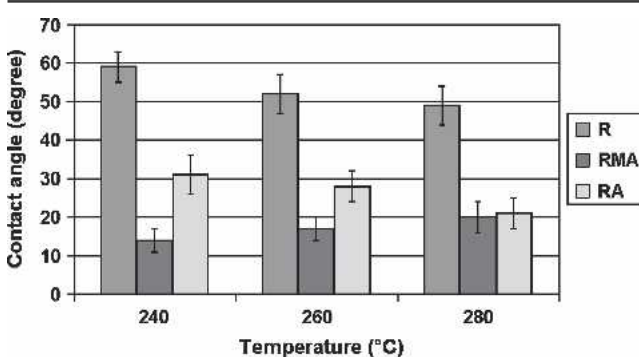


Fig. 5. Contact angles as a function of flux for Sn-3.8Ag-0.7Cu solder on Cu substrate.

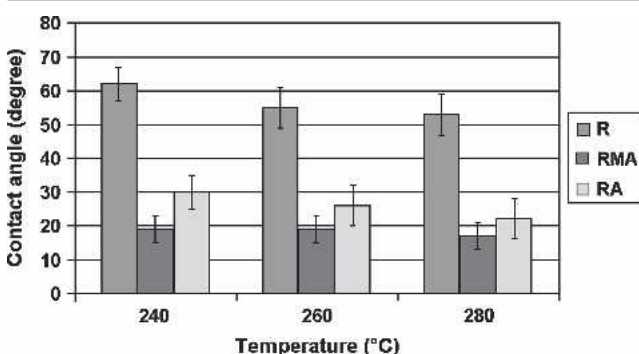


Fig. 6. Contact angles as a function of flux for Sn-0.7Cu solder on Cu substrate.

Microstructural Analysis of the Interface

Figure 7 shows the backscattered electron images of the cross section of the Sn-3.5Ag lead-free solder/copper interface obtained after reflow at 240°C. These pictures show the presence of intermetallic compounds at the interface. Two types of intermetallics can be observed: grains with a scallop-type morphology and a smooth, darker phase adjacent to the copper substrate (Fig. 7c). SEM/EDS analysis revealed that the composition of the scallop-type microstructure corresponds to Cu_6Sn_5 , while the layer adjacent to the Cu substrate is Cu_3Sn . This phase was only observed when RA flux was used. The Cu_6Sn_5 grains ranged from 2 μm to 5 μm in size, and the total thickness of the Cu_3Sn was less than a micron. The formation of intermetallics is attributed to diffusion of copper into the liquid solder. When the boundary layer of the molten solder, adjacent to the copper metal, becomes saturated with Cu, intermetallic compounds start to form at the copper substrate.

Figure 8 shows micrographs of the solder/copper interface at 260°C. It can be observed that the predominant intermetallic phase is Cu_6Sn_5 . The other compound, Cu_3Sn , is only shown in Fig. 8c, which illustrates the microstructure after reflow with RA flux. This might suggest that the RA flux promotes the formation of the Cu_3Sn . The grain size of Cu_6Sn_5 ranges from 1 to 5 μm , and the larger intermetallic grains are observed for the RMA flux. Microstructures of the interface obtained at 280°C are presented in Fig. 9. At this temperature, Cu_6Sn_5 is the main intermetallic phase for all fluxes, and Cu_3Sn is observed only for RMA and RA fluxes. The grain size of the Cu_6Sn_5 phase in decreasing order is $\text{RMA} > \text{RA} > \text{R}$.

Figure 10 shows micrographs of the wetting interface for the Sn-3.5Ag-4.8Bi alloy at 240°C. The micrographs for 260°C and 280°C are similar to those shown for 240°C. Despite the addition of a third element to the composition of the solder, only the scallop-type Cu_6Sn_5 intermetallic was identified at the detection limits of the SEM. This suggests that Cu reacts selectively with Sn and does not react with Bi. However, EDS analysis showed ~1–2 at.% of Bi in the intermetallic phase. No precipitation of Bi-rich particles was observed, thus it could be assumed that the Bi present was simply adsorbed onto the surface of the intermetallic phase. Micrographs for RMA flux showed an irregular contact line of the interface with large Cu_6Sn_5 grains (Fig. 10b).

The microstructures of samples corresponding to the Sn-3.8Ag-0.7Cu system at 240°C are presented in Fig. 11. The results for 260°C and 280°C are similar to the results for 240°C. This figure shows the Cu_6Sn_5 phase with grain sizes varying from 1 μm to 5 μm . The same micrographs reveal that the Cu_3Sn phase is detected only after reflow with RA flux, similar to the observation made for Sn-3.5Ag solder. Furthermore, the largest intermetallic grains were observed for RMA flux. The increase in temperature

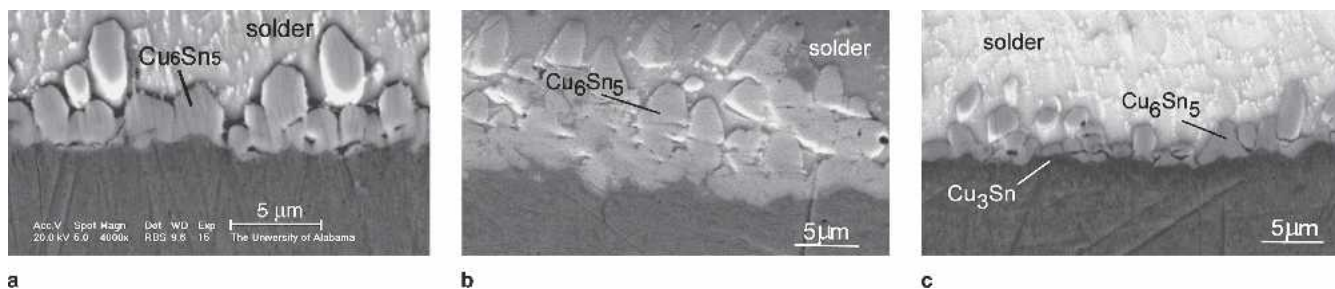


Fig. 7. Cross-sectional SEM images of the Sn-3.5Ag/Cu interface after reflow for 2 min. at 240°C for fluxes (a) R, (b) RMA, and (c) RA.

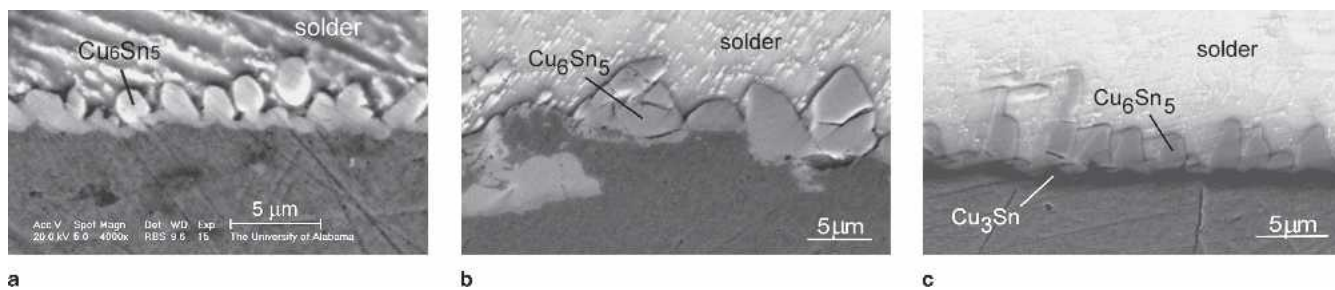


Fig. 8. Cross-sectional SEM images of the Sn-3.5Ag/Cu interface after reflow for 2 min. at 260°C for fluxes (a) R, (b) RMA, and (c) RA.

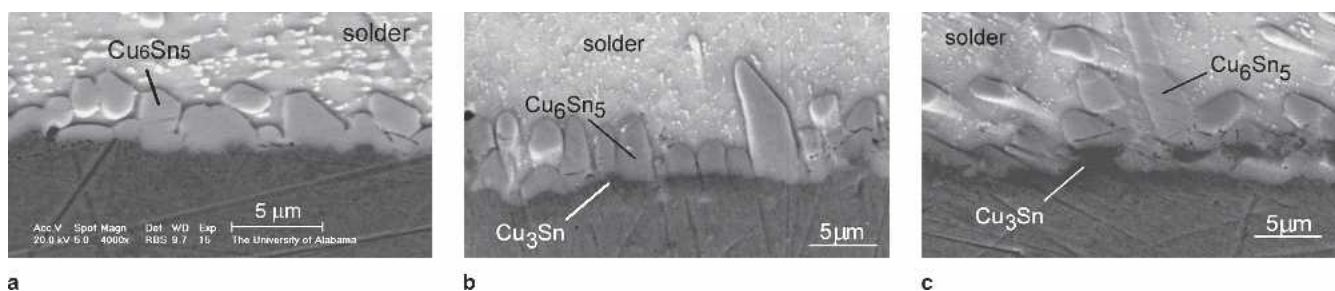


Fig. 9. Cross-sectional SEM images of the Sn-3.5Ag/Cu interface after reflow for 2 min. at 280°C for fluxes (a) R, (b) RMA, and (c) RA.

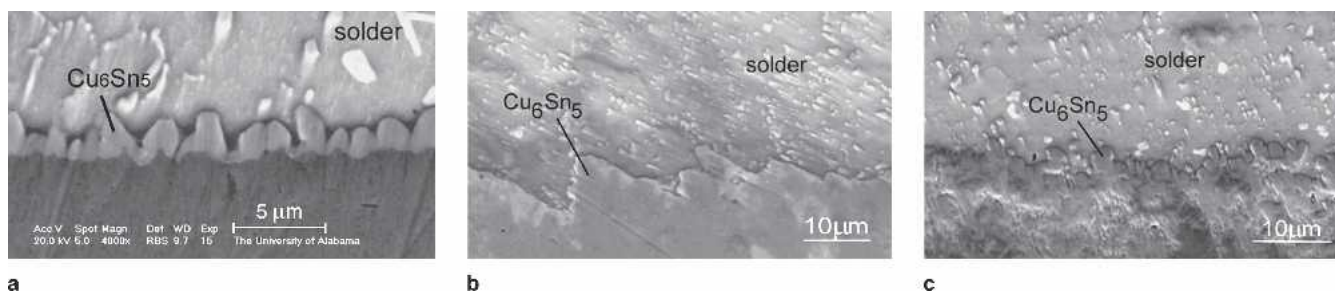


Fig. 10. Cross-sectional SEM images of the Sn-3.5Ag-4.8Bi/Cu interface after reflow for 2 min. at 240°C for fluxes (a) R, (b) RMA, and (c) RA.

promoted the growth of the scallop-like grains. They grow bigger but fewer with time, thus suggesting a ripening reaction among the scallop-type grains. During the wetting reaction, the neighboring scallops will not join together to form grain boundaries. Instead, the larger grains will grow at the expense of the smaller ones. While the formation of many small crystals is kinetically favored, large crystals are thermodynamically favored. However, growth is limited by the reduction in the number of channels between the scallops which provide the supply of copper atoms.⁹ Tu et al. have reported that the growth kinetics of Cu_6Sn_5 has a $t^{1/3}$ dependence on time.¹⁰

Figure 12 shows the microstructures obtained at the Sn-0.7Cu solder/substrate interface after reflow of 2 min. for a temperature of 240°C. This figure indicates that the main intermetallic phase present is Cu_6Sn_5 . However, a very thin layer of Cu_3Sn was observed for RA flux at temperatures of 240°C and 260°C. Although data of the physical properties of the fluxes was not available, it might be possible that the RA flux promotes the formation of the Cu_3Sn due to the higher heat-transfer coefficient. This could allow more heat for the formation of Cu_3Sn , which is known to require higher temperatures to be formed.

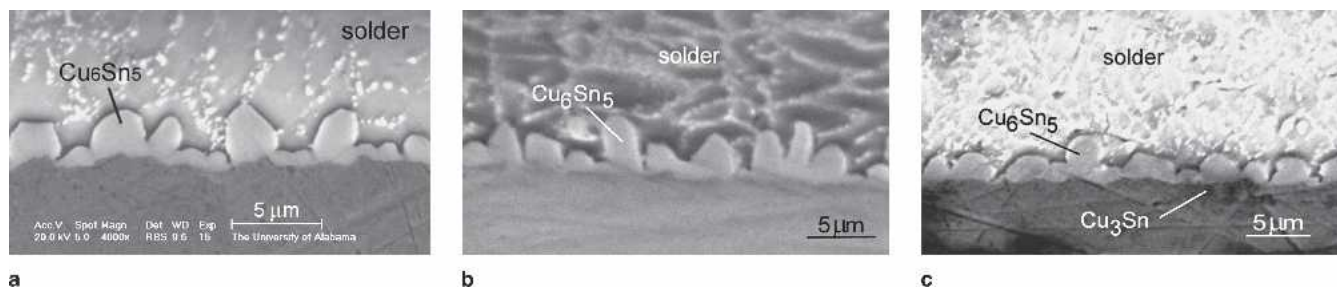


Fig. 11. Cross-sectional SEM images of the Sn-3.8Ag-0.7Cu/Cu interface after reflow for 2 min. at 240°C for fluxes (a) R, (b) RMA, and (c) RA.

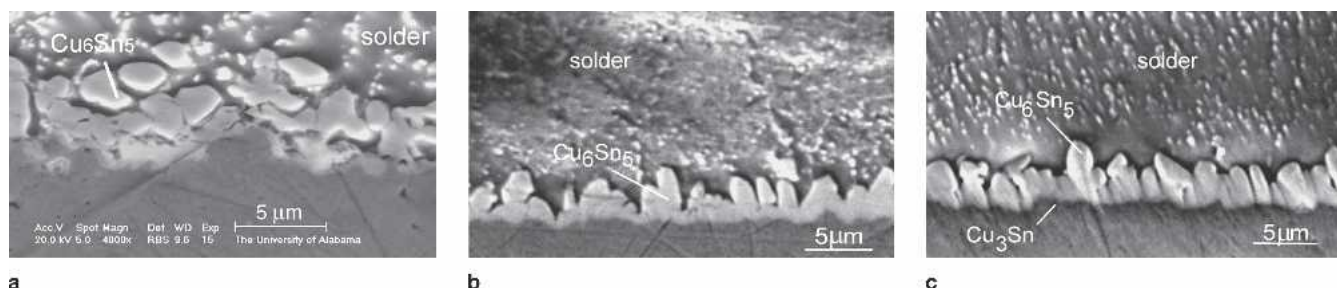


Fig. 12. Cross-sectional SEM images of the Sn-0.7Cu/Cu interface after reflow for 2 min. at 240°C using fluxes (a) R, (b) RMA, and (c) RA.

DISCUSSION

Usage of different fluxes, different solder compositions, or different reflow parameters affects the wettability of a solder alloy by changing the contact angle between the solder and its substrate, as shown in Figs. 3–6. Variation of the contact angle can be understood by the changes in surface tension originated by the fluxes. It can be explained by the Young-Dupré equation (Eq. 1) for the solder-flux-copper system. As in Eq. 1, imbalance of the surface tension at the solder/flux/substrate triple joint is the driving force for the spreading of the solder. An increase in γ_{fs} or decrease in γ_{fl} will decrease the contact angle, thus increasing wettability.

A flux addition that promotes those changes will result in improved wettability. The first role of the flux is to remove the nascent oxide layer from the substrate surface, so that the solder will wet and spread. The parameter γ_{fs} would be affected by using different fluxes because it represents the condition of the substrate surface. Strong aggressiveness of a particular flux would remove the oxide layer more efficiently and increase the value of γ_{fs} . According to Lau, R flux needs more time to be fully activated than do RMA and RA fluxes,¹¹ because the RMA and RA fluxes have ionic additives formulated to improve the flux action. Therefore, in the same solder/Cu system, the contact angle with R flux is significantly larger than that with RMA and RA fluxes under the same reflow temperature (Figs. 3–6). The second role of the flux is to remove the oxide skin on the molten solder and modify the surface chemistry of the molten solder. The parameter γ_{fl} would be reduced with this property of the flux. It can be speculated that RA and RMA fluxes promote reduction of γ_{fl} due to an increased fluxing activity as a result of their more powerful cleaning action.

However, the contact angles obtained for RMA were slightly lower than those for flux RA. There is no explanation at the moment for this behavior, although it might be related to the intrinsic properties of the fluxes, such as amount of halogen in the chemical composition and level of viscosity. In a previous study, it was suggested that the viscosity of the flux influences the wetting characteristics of a solder on a copper substrate.¹² The use of RMA flux instead of RA flux could be beneficial because the latter, due to its active ingredients, produces residues that are generally corrosive and potentially deleterious for the performance of the electronic assembly.¹³

The experimental results in Figs. 3–6 show that not only the variation in flux type but also the change of reflow temperature influences the contact angle between a solder alloy and Cu substrate since γ_{fs} has been changed. After removal of the oxide layer from the Cu substrate, the molten solder can make direct contact with the substrate and permit a metallurgical reaction between them. The production of this reaction is the formation of a layer of Cu-Sn IMC at the interface between the solder and Cu. This IMC layer also contributes to the driving force for the wetting and spreading of the solder by increasing the γ_{fs} .¹⁴ When reflow temperature increases, formation of the IMC layer is earlier and faster than the low reflow temperature process. Moreover, the effectiveness of flux increases with increasing temperature.¹⁵ Therefore, the contact angle decreases with increase of reflow temperature for the R and RA fluxes. However, for the RMA flux with Sn-Ag, Sn-Ag-Bi, and Sn-Ag-Cu solder systems, the contact angle increases slightly with increasing the reflow temperature (Figs. 3–5). It may be because the RMA flux begins to thermally decompose at the reflow temperature. The first consequence of the decomposition is the loss of oxide

removal and interfacial tension modification functions of the flux. The second consequence is that the decomposition products deposit on the substrate and inhibit further wetting and spreading of molten solders.¹⁴ For the case of Sn-Cu solder and Cu substrate, as shown in Fig. 6, the fast reaction between Sn-Cu and Cu may compensate the consequences of the decomposition of RMA flux. Therefore, the contact angle between Sn-Cu solder and Cu decreases with increasing reflow temperature.

The effect of Cu-Sn IMC formation on the contact angle and wettability of solder has been discussed above. In Figs. 3, 5, and 6, the contact angles for Sn-Ag, Sn-Ag-Cu, and Sn-Cu solder/Cu systems are quite close. However, the contact angle between Sn-Ag-Bi solder and Cu substrate is lower than the other three systems with the same flux and reflow temperature. According to Mackay, Bi addition in Sn-40Pb solder decreased wetting time and increased wetting force.¹⁶ Therefore, Sn-Ag-Bi solder has a much smaller contact angle than the other systems.

The results in Figs. 3–6 are important for the usage of a particular type of flux in a lead-free solder/Cu system. Because the optimum temperature condition, at which the specific flux/solder alloy couple with Cu substrate could be used to achieve acceptable wetting performance of the system, can be determined by these results. For example, Sn-Ag solder with RMA flux reflow at 240°C is the best working condition for the Sn-Ag/Cu system because the lowest contact angle can be achieved under this condition. However, it should be emphasized again that for the short, nonequibrated soldering process, not only the balance of interfacial tension and reaction between solder and its substrate but also the fluid viscosity and dissolutions of substrate can affect the wettability of a solder alloy.

CONCLUSIONS

The contact angles of four lead-free alloys, Sn-3.5Ag, Sn-3.5Ag-4.8Bi, Sn-3.8Ag-0.7Cu, and Sn-0.7Cu (all in wt.%), were measured on copper substrates. Three different rosin fluxes, namely R, RMA, and RA, were tested. Contact angles between 10° and 35° were obtained using RMA and RA fluxes whereas higher values, 30–65°, were obtained for R flux. The stronger cleaning action of RMA and RA fluxes is the main reason for obtaining a better wettability. Accordingly, R flux is not suitable for lead-free solders on Cu substrate. Among the sol-

ders tested, Sn-3.5Ag-4.8Bi exhibited the lowest contact angles, indicating excellent wettability. This improvement in wettability was attributed to addition of Bi, which led to the decrease of the surface tension of the system. For all soldering alloys studied, lower contact angles were obtained using RMA flux. The effect of temperature on contact angle in the range of 240–280°C depended on the type of flux used. In many cases, especially for R and RA fluxes, when the temperature was increased the contact angle decreased. However, in other results for RMA flux, the contact angle was independent of temperature. Intermetallics formed at the solder/Cu interface were identified as Cu₆Sn₅ and Cu₃Sn, the former in the form of scallop grains and the latter in layer-type structure. The use of RA flux promoted the formation of Cu₃Sn at the solder/copper interface.

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