# Intermetallic formation in solder joints

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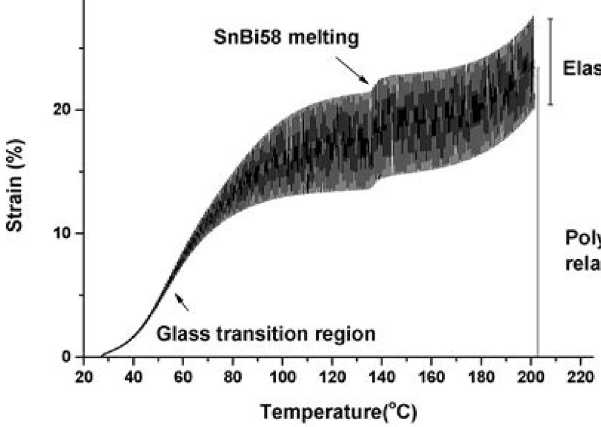


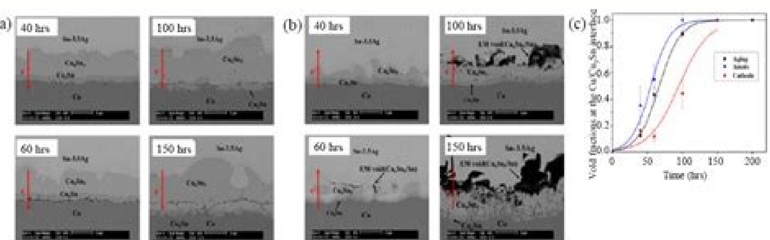

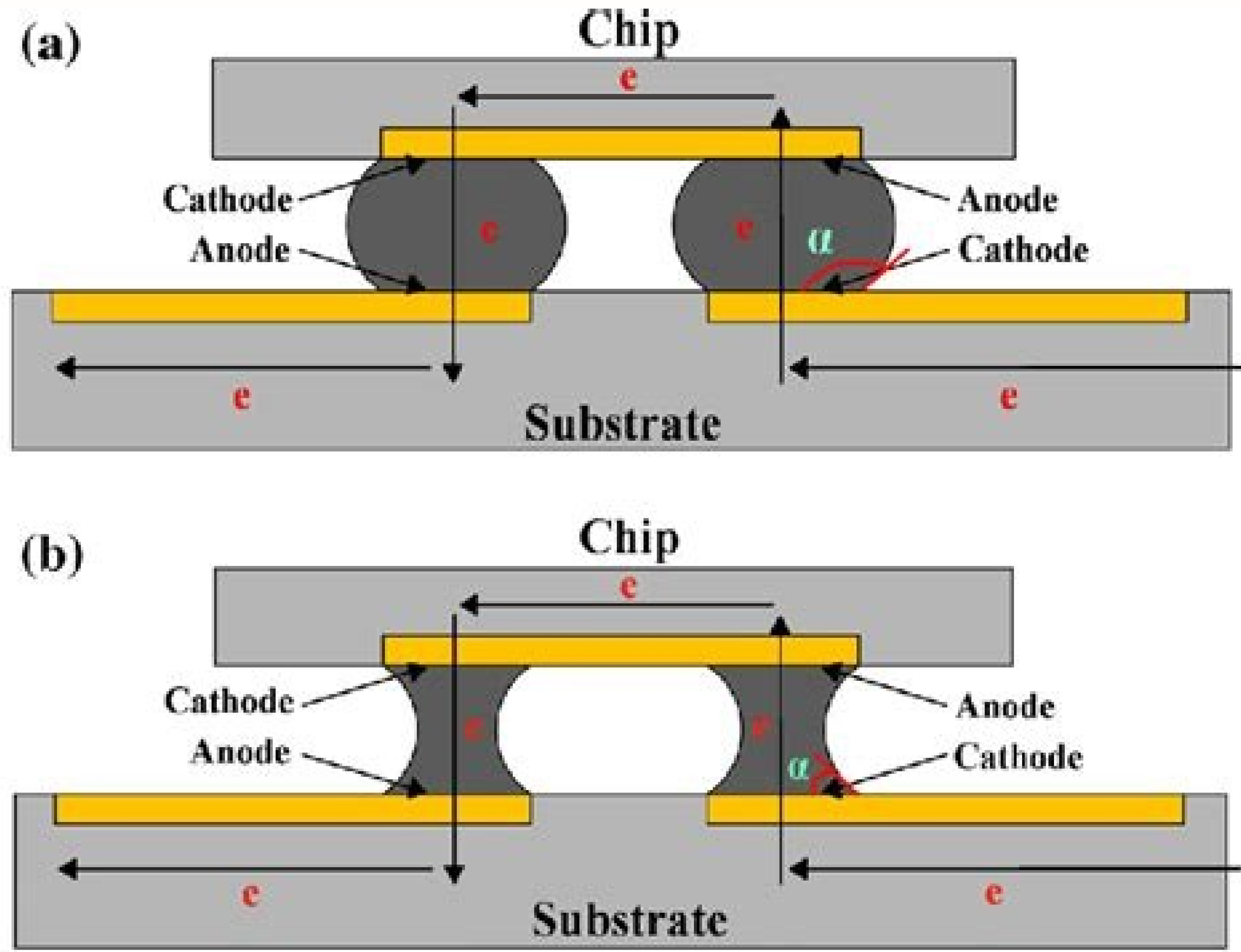


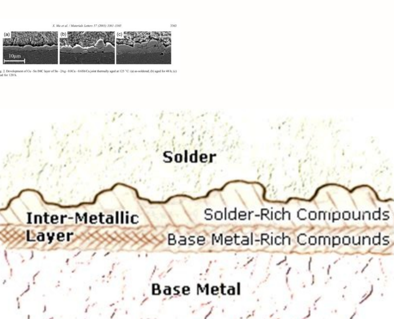


How to solder joints. Can you solder a copper joint. Can solder joints touch. Are solder joints strong.

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By the addition of bismuth to SAC257 solder, the spreading ratio increased from 80.46% to 85.97, indicating an improvement in wettability. In order to investigate the joint properties, alloy solders were bonded to copper substrate, and the structure of the interface, tensile-shear strength and the fractured surfaces were studied. It was observed that the thickness of the intermetallic compounds of Cu6Sn5 at the interface decreased with the addition of bismuth, and the lowest thickness of the interfacial IMCs was found in the SAC257-1Bi solder joint, which decreased about 14% compared to the base solder. Also, the Cu/SAC257-1Bi/Cu bond had the highest tensile-shear strength and elongation percentage among the alloy solders, which has a tensile-shear strength of about 30% and an elongation percentage of about 38% higher than the base solder joint. In recent years, the use of lead-free solders has increased to replace with the common Sn-Pb solder1,2,3. Among the various groups of lead-free solders, the Sn-Ag-Cu (SAC) triple alloy has the highest acceptability4. Due to this fact that these solders are easy to use and have relatively suitable mechanical and physical properties, they are the most widely used alloy solders5,6,7,8; However, there are still many unresolved issues with these solders9,10. SAC solders with high Ag content (>3 wt.%) have good cyclic thermal properties. However, the formation of a large amount of Ag3Sn intermetallic compound (IMC) results in non-homogeneous stress distribution and reduction of reliability in these solders11,12. Also, the high silver content has decreased the wettability and has increased the price of these solders. Recently, the SAC solder problems with high percentage of silver have been solved by SAC solders containing low silver content (1–3 wt.%). By reducing the percentage of Ag, despite an increase in elastic compliance and the plastic energy dissipation ability, thermal properties such as thermal fatigue and creep life of SAC solders have been reduced11,13. Therefore, to improve the properties of these solders, series of investigations have been carried out which show that the properties of SAC solders can be improved by adding a small amount of alloying elements such as Ni, Ti, Sb, Bi, Zn, Ni, Ga. Adding micro-alloying elements can modify the microstructure of SAC solders and it can limit the formation of Ag3Sn intermetallic by affecting the solidification rate11,14,15.Zhang et al.16 studied the effect of Zn on the wettability of Sn-3.8Ag-0.7Cu solder. They have shown an increase in the wettability of the mentioned solder by adding up to 0.8 wt.% Zn, but decreasing at higher percentages. This is due to the oxidation of Zn during soldering that reduces wettability16. Sungkhaphaitoon and Plookphol17 studied the effect of Sb addition on microstructure, mechanical and thermal properties of Sn-3Ag-0.5Cu solder. They reported that with the addition of Sb to the mentioned solder, more homogeneous distribution of IMCs was obtained, and the nature of IMCs changed to Ag3(Sn,Sb) and Cu6(Sn,Sb)5 compounds. Moreover, tensile strength increased and the ductility of the solder was reduced. It is worth to mention that by increasing the concentration of Sb from 0 to 3 wt.%, the melting temperature slightly increased17. El-Daly et al.9,10 showed that with the addition of bismuth to the SAC157 solder, the eutectic temperature decreases and the microstructure improves, which is a cause of decrease in the degree of undercooling. In addition, the tensile and creep properties of bismuth-containing solders improve as well9,10.Adding alloying elements to the SAC solders have three major effects on the interface reactions and the formation of IMCs18: It can increase or decrease the rate of reaction/growth. It can change the physical properties of the formed phases. It can create additional reactive layers at the interface or even replace reactive products with commonly formed IMCs. These elements generally fall into two general categories: (a) Elements that only affect the activity of the elements participating in the interactions of the interface, and these elements do not affect the formation of these phases. These elements generally have no significant solubility in the IMC layer, such as iron and silver. (B) Elements that participate in the interface interactions and have broad solubility in IMCs, such as gold and nickel. T. Laurila et al.18 showed that the elements of group (B) have a small effect on the growth of IMCs, while the elements of group (b) have a significant effect. The results showed that the addition of silver and iron, only reduced the diffusion coefficient, while in the case of gold and nickel, the reaction layer changed to (Cu, Au)6Sn5 and (Cu, Ni)6Sn5, respectively, and even in the case of nickel, the curvature of IMC growth did not show any other parabolic behavior with time, indicating a change in the growth mechanism18. Tao et al.19 added Ni and Sb to the Sn-3.8Ag-0.7Cu-3Bi alloy solder. They showed that shear strength increases with the solid solution mechanism of Sb in Sn matrix, and reinforcement of new intermetallic compounds (Cu, Ni) 6Sn5 and Ag3(Sn, Sb)19. A. Kantarcıoglu and Y.E. Kalay20 have shown that by adding micro alloying elements, Fe (0.01–0.1 wt%) and Al (0.05 wt%) to the eutectic SAC solder, the microstructure of the solder improved, the formation of Ag3Sn compounds was limited, and the new FeSn2 and Al-Sn-Cu intermetallic compounds were formed in the microstructure. Also, the thickness of the IMCs at the interface was reduced and the shear properties of soldered copper improved20. Developments of adding Bi and other common elements in Sn-Ag-Cu alloy are summarized in Table 1.Table 1 Common elements/additives in SAC lead-free solder and their effects5,9,10,15,16,19,32,49,50,51,52.In this study, bismuth element was used to improve the properties of SAC solder alloy. Previous studies suggest that a significant reduction in the weight percent of Ag in SAC solders causes problems like an increase in the solidification range. For this reason, Sn-2.5Ag-0.7Cu solder alloy with moderate percentage of Ag was chosen as the base alloy. Therefore, in this study, the physical and mechanical properties of SAC257-xBi solder and their joints were investigated.In this study, the pure elements of tin, silver, copper and bismuth (purity 99.99 wt.%) were used for the synthesis of Sn-2.5Ag-0.7Cu-xBi solder alloys (SAC257-xBi, x = 0, 1, 2.5, 5). For this purpose, the pure elements of Sn, Ag, Cu and various Bi percentages were placed in the alumina crucible in an atmospheric furnace at 650 °C. KCl-LiCl (1:1) was used as a flux to prevent molten oxidation and melt vaporization. For homogenization, the melt was kept at this temperature for 1 hour and experienced remelting for three times after the solidification of the alloyed solder. Casting the final melt was carried out in a cylindrical steel mold with a diameter of 4 cm and a height of 1 cm with a cooling rate of 6–8 °C/s. The chemical composition of the alloy solders after solidification is given in Table 2. The as cast samples were rolled and turned into sheets of 1 mm and 300 microns for use at the following stage (bonding). The rolled sheets were annealed in oven at 100 °C for 1 hour.Table 2 Chemical composition of synthesized solder alloys (wt.%).Characterization of the solder alloysIn order to investigate the microstructure of alloyed solders, the distribution of grains and IMCs before and after alloying, the samples were sectioned in 5 mm * 5 mm dimensions and then cold mounted. Then, the specimens were prepared according to the standard methods of metallography. For etching, a solution of ethanol [96Vol%], hydrochloric acid [2Vol%] and nitric acid [2Vol%] was used and the microstructure of the solder alloys was investigated by a scanning electron microscopy (Philips-Xl30- & FEI-Quanta -EDS Element silicon drift) and optical microscopy (Olympus-BX51M). MIP software was used for analyzing the microstructure images.The DSC test was used to measure the thermal properties of SAC257-xBi solder alloys (x = 0, 1, 2.5, 5) in accordance with ASTM D3418/E1356 standard by Pishtaz Engineering Co. model TA-1A instrument. Samples with equivalent weight between 15 to 25 mg were used. The test was carried out in the argon atmosphere in the form of sweep (heating-cooling). The heating rate was 10 °C/min and the maximum temperature reached 300 °C during the DSC test. To determine the wettability of the solder alloys according to Japanese Industrial Standard, (JIS 23198-3,2003), the spreading ratio was calculated. For this purpose, the alloyed solder disks of the same weight (200mgr) containing flux were placed inside a ceramic crucible and melted in the furnace to obtain a spherical state after solidification. After the production of spherical shaped solders, they were placed on the copper substrates with flux (RMA) and according to the temperature program represented in Fig. 1; the process was carried out in the furnace. After cooling the specimens, the diameter (D) and height (H) of the solidified droplets were measured and the spreading percentage of the solder alloys was calculated using equation (1). Figure 2a shows how to measure the spreading ratio.$${\rm{Spreading}}\,{\rm{ratio}}=({\rm{D}}-{\rm{H}}/{\rm{D}})\ast 100$$Where D is the diameter of spread solder (mm), and H is the height of spread (mm).Figure 1The temperature program to measure the spreading ratio.Figure 2(a) The procedure for measuring the spreading ratio, (b) the geometry of sub-size samples for tensile test.Tensile test was used to examine the mechanical strength of SAC257-xBi (x = 0, 1, 2.5, 5) solder alloys. For this purpose, the sub-size samples were prepared in accordance with Fig. 2b. The Instron 5500 R test device was used for this test. The test was performed at ambient temperature with a 1 mm/min jaw speed. Three samples were tested for each case and finally the average stress-strain obtained was reported.Characterization of the Cu/SAC-xBi/Cu jointsMaterialsIn order to investigate the properties of the solder alloy joints, 2 mm copper sheets with a purity of 99.999 wt.% were used as substrate. For deoxidation and reduction of the surface roughness of copper sheets, the specimens were grinded and washed in sulfuric acid solution for 30 minutes. Finally, in order to ensure the absence of fats and other surface contaminants, the samples were washed with alcohol and kept in the neutral atmosphere of argon until they were bonded to maintain surface conditions. The synthesized solder alloy interlayers were cut in 12* 12 mm2 and 300 µm in thickness. In order to remove surface contaminations, the interlayers were washed with alcohol and were stored in the argon atmosphere until bonding process. The RMA flux was used to prevent the base metal and solder alloys against the oxidation during bonding process.Resistant furnace was used to provide the required temperature for bonding, and bonding temperature program is in accordance with the IEC 60068-2-58 2015 standard, as shown in Fig. 1.Microstructure of the interfaceIn order to investigate the microstructure of the base solder/ base metal interface, a 300 µm solder foil and flux was placed between two copper samples with dimensions of 10 * 10 * 2 mm, and the system experienced temperature cycle in the furnace in accordance with the temperature program given in Fig. 1. According to the melting temperature of SAC solders, the maximum soldering temperature was chosen at 250 °C. In order to investigate the morphology of the intermetallic compounds formed at the interface after the bonding process was completed, the samples were sectioned perpendicular to the thickness by a cutting machine. The standard metallographic operations were performed on them using a mixed acidic etching solution containing ethanol [96Vol%], hydrochloric acid [2Vol%] and nitric acid [2Vol%]. SEM and FE-SEM were used for microstructural studies (see Fig. 3a). SEM equipped with an EDS system with a silicon drift sensor was used for semi-quantitative analysis of phases. In order to investigate the microstructure, MIP software was used for quantitative calculations of microscopic images such as the size of the interfacial zone and the volume fraction of IMCs and precipitates.Figure 3(a) The procedure for microstructural characterization of solder joint, (b) The details of solder joint set up.Calculation of the IMC thicknessIn order to determine the average thickness of the IMCs, the MIP image analyzer software was used. Because the thickness of the IMC layer was not uniform throughout the interface, the mean value of the thickness (Y) of IMC was obtained from the Equation (2), where A is the total area of the IMC layer in the image, L is the length of the IMC in the interface direction. For each of the thicknesses reported in the solder alloys, the length of 400 µm (6 photos) was taken from different parts of the interface region and then calculated and reported. Figure 4 shows the overall trend of the IMCs thickness calculation.$${\rm{Y}}={\rm{A}}/{\rm{L}}$$Figure 4The overall trend of the IMCs thickness calculation by software.Tensile-Shear testThe tensile-shear specimens were prepared in accordance with the ASTM D1002 standard which is provided in Fig. 3b. In this test, 300 µm solder alloy foils were used as interlayer, copper samples were used with dimensions of 35 mm * 10 mm * 2 mm, and the soldered area was 10 cm * 10 cm. The temperature program represented in Fig. 1 was used for bonding process. Two pieces of copper with 2 mm thickness were placed on both sides of the sample in order to align the sample in the jaws of the tensile machine. Instron tensile machine was used for tensile-shear test. Test was performed at ambient temperature with 1 mm/min jaw speed. The application of the tensile force with the tensile machine causes a shear force at the interface. Thus, by means of load-displacement diagram, the tensile strength of the joint was measured.FractographyIn order to investigate the failure mechanism of the joint, the tensile-shear fractured samples were sectioned, cleaned in ultrasonic bath for 15 minutes to eliminate the surface contaminations and were investigated by FE-SEM.To study the thermal behavior of solder alloys during cooling and heating, the DSC test was performed. Figures 5, 6 and 7 show the results of this test. The melting temperature is one of the critical factors for solder selection in microelectronic industries, for this reason, this should be taken into consideration to improve the base solder properties21,22.Figure 5Cooling and heating curves of solder alloys during the DSC test.Figure 6The melting temperature and pasty range of solder alloys obtained from DSC test.Figure 7Onset cooling and heating temperatures and undercooling for solder alloys obtained from DSC test.In this study, with the addition of bismuth to the SAC257 base solder, despite the fact that the change in pasty range is small, the melting point decreases from about 219 °C in the SAC257 base solder to 207 °C in the alloy SAC257-5Bi, which indicates the positive effect of bismuth on the melting point. According to Lindemann's criterion, melting occurs when the second root of the atom displacement in the network exceeds a certain atomic distance. Therefore, it is possible that with the addition of bismuth to the SAC257 solder, and the formation of new Sn-Bi bonds resulting from the reaction between tin and bismuth, the atomic displacement in crystalline network occurs easier and the overall melting temperature of the system decreases23,24,25. Another parameter affecting the solidification behavior of the solder and its microstructure is undercooling, which is defined as ΔT = TL − TS, where TL is the temperature at which the melting starts during heating stage, and TS is the solidification temperature during cooling stage. The lower the ΔT, the better the nucleation of solidified phases, which increases the tendency for heterogeneous nucleation and makes the microstructure finer9,10,25,26. According to Fig. 7, the addition of bismuth to the base solder SAC257 has reduced the degree of undercooling, which indicates that the presence of bismuth is very effective in improving the microstructure. As shown in the optical microscopy images in Fig. 8, by adding bismuth to the SAC257 base solder, the eutectic region expands and the β-Sn region is reduced. In SAC257-xBi solder alloys, the decrease in β-Sn size due to the reduction of ΔT can be attributed to the presence of bismuth. Increasing the heterogeneous nucleation sites by primary IMCs, causes faster solidification rate of Ag3Sn and β-Sn phases and results in the finer microstructure9,10,25.Figure 8The optical microstructure of solder alloys: (a) SAC257, (b) SAC257-1Bi, (c) SAC257-2.5Bi, and (d) SAC257-5Bi.Figure 9 shows the back-scatter electron SEM images to characterize the microstructure of the base solder and SAC257-xBi solder alloys. According to this fact that the SAC257 base solder chemical composition is close to the eutectic composition, the Ag3Sn and Cu6Sn5 phases are formed in the β-Sn matrix, which are shown in Fig. 9a. By adding bismuth to the SAC257, the microstructure changes so that, according to Fig. 9b–d, the morphology of the Ag3Sn and Cu6Sn5 intermetallic compounds tends to change from needle to equiaxial shape. Also, due to the decrease in the degree of undercooling, the size of the intermetallic compounds decreases9,10,25. On the other hand, the Bi solubility is also limited in Sn, and in the case of more than 1 wt.% Bi, bismuth is deposited separately27,28, which is visible as white phase in Fig. 9c,d. The semi-quantitative EDS analysis of the phases in the base solder and SAC257-5Bi solder alloys is shown in Fig. 10, which also confirms the presence of white bismuth phase.Figure 9The back-scatter electron SEM images of microstructure for the base solder and SAC257-xBi solder alloys: (a) SAC, (b) SAC1Bi, (c) SAC2.5Bi, and (d) SAC5Bi.Figure 10The semi-quantitative EDS analysis of the phases in the base solder and SAC257-5Bi solder alloys.Figure 11a–d, represents the results of the solidification sequence through JMATPRO software. As it is evident in SAC257, the intermetallic compounds of Ag3Sn and Cu6Sn5 are formed in the final stages of solidification. But, by adding Bi in the case of SAC257-1Bi, SAC257-2.5Bi, and SAC257-5Bi, the Ag3Sn and Cu6Sn5 intermetallic compounds are formed from the beginning of the solidification process25. Studies have shown that the formation of the β-Sn matrix phase in Sn-based alloys requires high degrees of undercooling11; therefore, in SAC257 solder with a relatively high degree of undercooling, first, the β-Sn phase is formed as the primary solidification product, and then the intermetallic compounds as the secondary phases. Talking about SAC-Bi solder alloys, Bi affects the solidification behavior of solder and reduces the degree of undercooling, which results in the formation of a portion of the intermetallic compounds as the primary solidification products, which act as the heterogeneous nucleation sites. On the other hand, reduction of undercooling limits the growth of β-Sn, and leads to the finer grain structure. Another part of the intermetallic compounds are formed simultaneously during a Sn-Ag-Cu ternary eutectic reaction. Therefore, two types of morphologies of the intermetallic compounds are expected to be observed in the microstructure of the SAC257-Bi alloys25. According to Fig. 12a–d, it can be seen that in the microstructure of Bi-containing solder alloys, in addition to the large IMCs and Bi precipitates, series of fine precipitates are observed in the matrix. For further examination of these fine particles, FE-SEM images were taken at high magnification from these fine precipitates, as shown in Fig. 12a,b. In the microstructure of the SAC257-1Bi solder, according to the Bi solubility limit in Sn and the contrast of the atomic numbers in BSE mode, it is likely that these nanosize particles are the same IMCs. As mentioned before, the reason for the formation of these nano-precipitates is the decrease in the degrees of undercooling, the increase in heterogeneous nucleation and the formation of intermetallic compounds during solidification. In the SAC257-2.5Bi and SAC257-5Bi solders (Fig. 12c,d), the Bi white nanosize precipitates were also formed in the matrix during a solid state process due to the reduced solubility of Bi in Sn by reducing the temperature. Therefore, Bi, due to the change in the solidification rate, the growth rate of eutectic phases and the reduction of the eutectic point, makes the microstructure finer.Figure 11Analysis of the Solidification behavior of solder Alloys: (a) SAC 257, (b) SAC 257-1 wt.%Bi, (c) SAC 257-2.5 wt.%Bi, and (d) SAC257-5 wt.%Bi solder alloys by using JMATPRO® software.Figure 12High magnification FE-SEM image from fine IMC and Bi precipitate distribution in SAC 257-Bi Solder alloy: (a,b) SAC257-1Bi (c) SAC257-2.5Bi (d) SAC257-5Bi.By calculating the phase fraction in the solder alloys, which is provided in Fig. 13, it can be seen that by increasing the bismuth from 2.5 wt.% to 5 wt.%, the area fraction of bismuth precipitates increases from 4.46% to 7.54%, respectively. Bismuth, on the other hand, has inherent brittleness, and by addition of high Bi percentage to the base solder, coarse bismuth deposits are created in the microstructure, which can reduce the mechanical properties of the solder alloys. These precipitates are well visible in the microstructure of SAC257-5Bi solder alloy.Figure 13Calculating phase fraction in the solder alloys: (a) SAC257-2.5Bi (b) SAC257-5Bi.WettabilityTo achieve a sound solder joint, liquid phase should properly wet the surface of the solid base materials. This means that there should be a special reaction between the liquid solder and the solid surfaces that are supposed to be bonded. The ability of liquid solder for spreading and flow during the soldering process is one of the most important factors for the formation of a suitable bond between solder and the base material29,30. Therefore, for improving the properties of SAC solders with alloying elements, the alloying elements should be selected in such a way that the wettability of the solder does not decrease and even improves. The spreading ratio is one of the important factors for investigation of the wettability of alloy solders. The results of spreading ratio in different cases are shown in Fig. 14. It is observed that with the addition of bismuth to the base solder, the percentage of spreading ratio increases and ranges from 80.46% in SAC257 to 85.97 in the SAC257-5Bi solder.Figure 14The results of spreading ratio in different solder alloys.The variation of the spreading ratio from 2.5 wt.% Bi to 5 wt.% Bi alloy solders is eligible, indicating that more than 2.5 wt.% Bi does not change the wettability. Generally, proper wettability occurs when there is the lowest amount of surface free energy. This means that the surface energy of the solder will be reduced by forming an interface with lower free energy. On the other hand, in order to liquid solder wets and bonds with the base material surface, the external forces (between liquid and the substrate) must overcome the internal forces (liquid interatomic forces). As the molten inner bonds become weaker, wettability can improve. In alloy solders, with the addition of bismuth and the formation of a weaker Sn-Bi bond, instead of Sn-Sn, the wettability can be improved. For this reason, the addition of bismuth to the base solder will increase the wettability25,31.In terms of classical wettability model, for determining the wettability of molten solder on a solid surface, the competition between the cohesive forces (molten internal forces) and adhesive forces (the force between the atoms of the molten solder and the substrate), as well as the molten surface tension, is decisive. Therefore, in order to molten solder wets the substrate, the adhesive forces must overcome the cohesive forces of the molten solder. From the classical wetting point of view, the weaker the cohesive forces of the molten solder, the higher the wettability of the liquid phase on the solid substrate and the lower the wetting angle. As mentioned before, the melting temperature decreases in the solder alloy by adding Bi. Given this, addition of Bi weakens the internal forces (likely to form a weaker Sn-Bi bond instead of Sn-Sn) and therefore, external forces can overcome more than before. In other words, from the classical wetting perspective, by increasing the weight percentage of Bi as the melting point depressant, the spreadability of the liquid phase increases. Bi also has a lower surface tension and, by adding it to the base solder, the overall surface tension of the molten solder is reduced31,32,33.But, in addition to the classical theory of wettability, reactive wettability should also be taken into account, because the molten solder reacts with the substrate. Thus, the reaction of the molten solder with the copper substrate also affects the wettability and spreading ratio. Studies have shown that Bi and Cu have a very poor reactivity, but Sn and Cu have a strong interaction. In general, the presence of bismuth in small concentrations, increases the reactivity between Sn and Cu, and the Sn-base solder alloys containing Bi exhibit better wettability comparing to the base solder (So not only from the classical wettability, but also from the reactive wettability point of view, the wetting should increase). But by increasing the amount of Bi in the solder alloy, the rate of increase in the wettability decreases and high concentrations of Bi, negatively affects the wettability. The reason for such a behavior can be explained based on the following schematic. Regarding Fig. 15a–d, for the reaction and the formation of equilibrium intermetallic compounds at the interface, because of the weak reactivity of Bi with Cu, Bi atoms dissolved in β-Sn will be removed from this structure due to the reduction of the local solubility. The greater the amount of Bi in the solder, the more Bi atoms accumulate at the interfacial molten phase reducing the effective reactivity of Sn and Cu34. As shown in the results of the spreading coefficient, the rate of increase in wettability decreases with increasing Bi weight percent (see Fig. 14).Figure 15Schematic of the bismuth effect on reactivity and spreading in the interface of molten solder/Cu substrate: (a) SAC257/Cu (b) SAC257-1Bi/Cu (c) SAC257-2.5Bi/Cu (d) SAC257-5Bi-Cu.Tensile properties of the soldersFigure 16 shows the tensile test results of the SAC257 base solder and SAC257-xBi alloy solders. According to the results, for the alloy solders with bismuth up to 2.5 wt.%, the yield stress and the tensile strength increase, and the percentage of elongation shows a gradual decreasing trend. However, in the SAC257-5Bi alloy solder, the yield and tensile strengths are reduced and the percentage of elongation is also significantly reduced. According to the SEM microscopic images (Fig. 9d), the large bismuth precipitates are visible in the microstructure of SAC257-5Bi alloy solder. Bismuth is inherently brittle, dissolves in small amounts in the Sn matrix (about 1 wt.%) and the high percentage of bismuth in the Sn alloy forms large precipitates in the matrix by solid state precipitation of Bi. Therefore, the high amount of Bi is detrimental for the mechanical properties. In low percentages of bismuth (

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