

# Effects of Zn addition on the Intermetallic Formation and Joint Strength of Sn-3.5Ag-1.0Cu Solders

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## ABSTRACT

*The need to replace lead-based solders has received considerable attention among researchers because of their toxicity. The Sn-Ag-Cu family is the most promising candidate. However, these solder systems require some improvement in terms of their performances in terms of mechanical properties. In this study, Sn-3.5Ag-1.0Cu solder was investigated with the addition of 0.1, 0.4 and 0.7 wt.% Zn. The solder was prepared using a powder metallurgy method to form the discs. An intermetallic compound study was conducted by melting a solder disc on a Cu substrate at 250 °C for 1 min. For the shear strength study, the solder was melted at 250 °C on two pieces of the Cu substrate. Aging process was conducted in an oven at 150 °C for 1000 h under an air atmosphere. For the intermetallic study, the aged solder joint was cross-sectioned, mounted in epoxy resin, and observed under a Scanning Electron Microscope. The solder joint specimens were tested using an Instron machine and the fracture surfaces were examined under a Scanning Electron Microscope. The  $Cu_6Sn_5$  intermetallic compound was initially formed with scalloped morphology. This intermetallic structure transformed into a flat structure at higher aging temperatures and durations. As aging progressed, another thin and flat intermetallic layer was formed near the Cu interface. The intermetallic compound was identified to be  $Cu_3Sn$ . The solder joint strength degraded at a later stage of aging owing to the excessive growth of intermetallics with the 0.7 wt.% exhibit higher values. The fracture mode displays ductile failure at lower aging temperatures and times, but shows brittle failure at higher aging temperatures and longer times.*



*Keywords: Shear Strength; Intermetallic; SnAgCuZn Solder; Cu-Sn; Fracture Surface*

## INTRODUCTION

Sn–Pb is the solder alloy system most widely used as an interconnection material in the electronic packaging industry. This is because Pb-containing alloys have an attractive combination of reliability, are well tested, and are cheap. However, Pb-based solders also have certain disadvantages. Apart from the undeniable toxicity of Pb to the human nervous system, it is also damaging to the environment owing to its potential for groundwater contamination [1].

With rapid economic growth, the rapid upgrading of electrical and electronic equipment has resulted in an increase in the quantity of waste electrical and electronic equipment (WEEE). This poisonous “heavy metal” from lead-based solder leaches out to solid-waste landfills and pollutes the underground water supply. Legislation such as restricting the use of Hazardous Substances (ROHS) has been implemented in the European Union. The ROHS Directive limits Pb content to less than 0.1 wt.% (1000 ppm) in the solder [2]. Consequently, Pb-containing solder alloys are either banned or phased out from the electronic industry.

Furthermore, solder joints must withstand high-stress conditions and severe thermal expansion mismatches. These challenging environments impose higher demands on the mechanical strength of solder joints and the solderability of the solder material [3-5].

Various lead-free solders have been proposed as a replacement for Sn-Pb solders, such as Sn-Ag, Sn-Cu, Sn-Zn-Bi, and Sn-Ag-Cu solders. Sn-Ag-Cu lead-free solders with various compositions have received considerable attention as substitutes for Sn-Pb solders. This solder has excellent properties compared with other lead-free solder candidates. These solder compositions have received considerable attention from researchers owing to their low cost, superior properties, and low melting temperature, which is close to that of Sn-Pb solder. However, some improvements, either in terms of the material choice or process operation, are required to

improve their properties. Sn-Ag-Cu lead free solders have certain drawbacks compared to Sn-Pb solders. These drawbacks include the rapid growth of brittle intermetallic compounds both in the bulk and at the solder/substrate interface during processing and use.

During the aging process, several reactions such as coarsening, growth of intermetallic compounds, and formation and growth of pores and cracks significantly affect the life expectancy of the joint and considerably influence the properties of the joints. It has been demonstrated that cracks usually appear near the intermetallic layer and propagate along its interface of the intermetallic layer [6]. The formation of an intermetallic compounds (IMC) layer is desirable to achieve good bonding between the solder and substrate. However, owing to the high brittleness of IMCs, an excessively thick layer of intermetallics may deteriorate the mechanical properties of the joint. Efforts have been made to improve solder properties by adding minor alloying elements. These minor alloying elements usually have three major effects on the reaction between the solder and the substrate. First, they can increase or decrease the intermetallic growth rate; second, they can change the physical properties of the formed phase; and third, they can form additional reaction layers at the interface or they can displace the binary phases that would normally appear and form other reaction products instead. However, research reports related to these additions of minor alloying elements are still limited, and further explanation is required.

In this study, three different percentages of Zn were added in Sn-3.5Ag-1.0Cu and the composite solder was soldered onto a Cu substrate. The solder joints were aged for 1000 h at 150 °C. Intermetallic interfaces were observed and the joint strength and fracture surface were investigated.

## METHODS

Sn-3.5Ag-1.0Cu-xZn solders were prepared from micron-sized ( $< 45 \mu\text{m}$ ) powder samples supplied by Sigma Aldrich. Three weight percentages of Zn were used; 0.1, 0.4 and 0.7 wt.%. A total of 5 g of powder samples with proportion weight were and placed in a small container. The solders were prepared using a powder metallurgy route that included mixing, compression, and sintering. The mixer was set up at a speed of 200 rpm

and mixed for 2 h using Panasonic DVUX 960 W roll mill. Then, 1 g of the powder was compressed at 140 bar using hydraulic press to produce a disc with a diameter of 12 mm and thickness of 1 mm and then sintered in a furnace for 2 h at 150 °C in a hydrogen gas environment to avoid oxidation. This sintering process hardens solder discs.

For the intermetallic study, the solder disc was placed on a copper substrate and melted at 250 °C using ZnCl<sub>2</sub> flux. The solder/copper substrate interaction was performed on a hot plate. The solder and copper substrate were allowed to react for 1 min and then cooled on a hot plate. The samples were then cleaned and placed in an oven for ageing. Aging was performed at 150 °C for 1000 h and the solder joint was mounted and polished to smooth the surface and analyzed under a Scanning Electron Microscope model Zeiss-Supra 35VP-24-58 operating in back scatter mode. Energy dispersive X-ray (EDX) was used to determine the elemental compositions.

For the tensile test study, two copper strips were cut and soldered together using 0.2 g solder. The joints were prepared by melting the solder at 250 °C for 1 min on a hot plate using a zinc chloride flux, as shown in Figure 1. The aging process was repeated as previously described. Six samples were tested under each aging condition. A lap area (8 x 4) mm<sup>2</sup> and initial length (l<sub>0</sub>) of 4 mm were used as input data for the stress/strain calculations. After 100 h, the samples were removed.

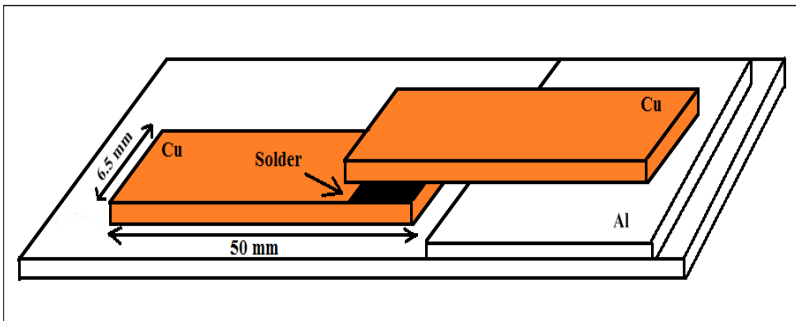


Figure 1: Solder joint for tensile test

For the test, an Instron machine (Instron 5582) was used to pull the ends of the Cu joints at a rate of 0.2 mm/min. The initial gauge length was set to 40 mm, and Cu joints were placed in the middle of the gauge. The thickness and width of the reacted solder on the Cu plate were measured and filled in a dialog box during set up. The values of maximum stress at fracture (failure) and strain were recorded. After the test, the fracture surface was observed using Scanning Electron Microscope to determine the fracture mode.

## RESULTS AND DISCUSSION

The reliability of electronic devices in relation to the mechanical strength of joints is vital. This is especially true for portable products, such as hand phones, laptops, mp3 players and others. These devices frequently experience mechanical shock loadings caused by dropping during operation. This section explains the formation of interface intermetallic and shear tests of solder joints in terms of the stress and strain values between the two copper substrates after the aging process. A detailed explanation of intermetallic formation between Sn-3.5Ag-1.0Cu-xZn and Cu substrate is presented elsewhere [7]. Shear testing was performed to determine the force at which a joint component can be “sheared,”- thus indicating the joint strength. The greater the force required to shear the component, the better and stronger the solder joint. However, excessive intermetallic growth may have a negative effect on the joint strength owing to its brittle nature. Therefore, aging degradation of solder joints in electronic packages is a critical concern in the microelectronics industry.

### Intermetallic Formation

Figure 2 to 5 show the intermetallic formation evolution of solder materials with different Zn contents on the Cu substrate. When the Sn-based solder reacted with the Cu substrate, the initial intermetallic compound formed at the interface was  $\text{Cu}_6\text{Sn}_5$ . This intermetallic has been reported by several researcher [8-11]. Figure 2(a) shows SEM images of the Sn-3.5Ag-1.0Cu solder that reacted with the Cu substrate after aging at 150 °C 100 h. EDX analysis and the Cu-Sn phase diagram confirmed the formation of  $\text{Cu}_6\text{Sn}_5$  intermetallic. With the addition of Zn (Figure 2b-d), the formation

of the  $\text{Cu}_6\text{Sn}_5$  intermetallic was slightly larger with the scallop structure. A second layer below  $\text{Cu}_6\text{Sn}_5$  was observed. This layer was identified as  $\text{Cu}_3\text{Sn}$ . However, the  $\text{Cu}_3\text{Sn}$  layer was much thinner than the  $\text{Cu}_6\text{Sn}_5$  layer. Furthermore, the formation of  $\text{Cu}_3\text{Sn}$  can be easily distinguished, as it has a darker contrast than the  $\text{Cu}_6\text{Sn}_5$  intermetallic. According to Kotadia *et al.* [12], the  $\text{Cu}_6\text{Sn}_5$  intermetallic is thermodynamically unstable; therefore,  $\text{Cu}_3\text{Sn}$  intermetallic forms overtime in the presence of heat by consuming the  $\text{Cu}_6\text{Sn}_5$  compound and Cu from the substrate, as shown in Equation (1). From the equation, it can be seen that the over-saturation of Cu atoms leads to the formation of  $\text{Cu}_3\text{Sn}$  intermetallic [13].

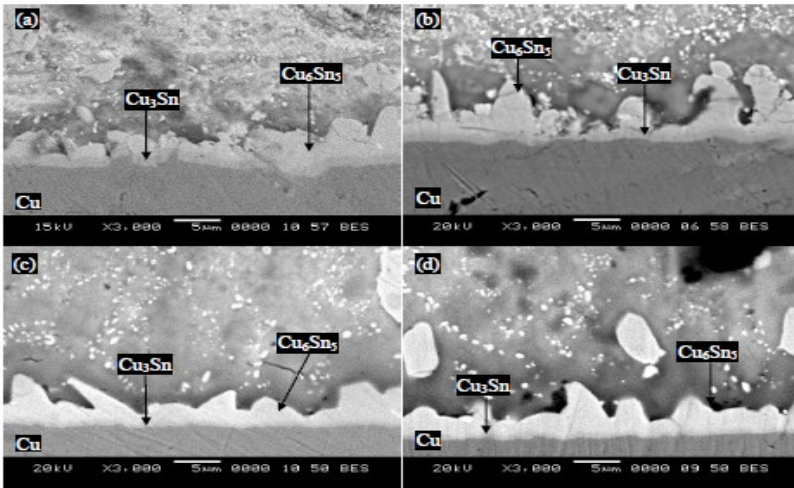
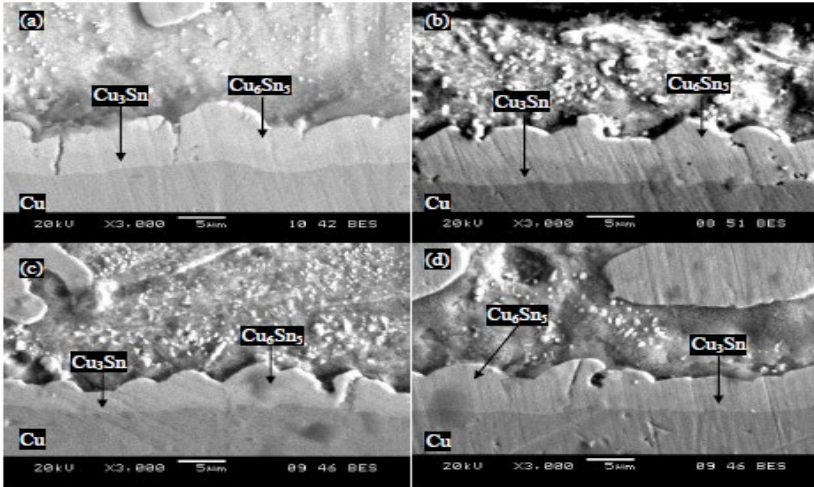


Figure 2: SEM images after aging for 100 h at 150 °C: (a) SAC (b) SAC-0.1Zn (c) SAC-0.4Zn and (d) SAC-0.7Zn.

When the aging time was increased to 500 h (Figure 3), two intermetallic layers were clearly observed. However, the  $\text{Cu}_6\text{Sn}_5$  layer transformed into a more planar morphology. Both layers have a unique contrast, which can be easily distinguished.



**Figure 3: SEM images after aging for 500 h at 150 °C: (a) SAC (b) SAC-0.1Zn (c) SAC-0.4Zn and (d) SAC-0.7Zn.**

Figures 4 and 5 clearly show the transformation from scallop-to-planar morphology for the  $\text{Cu}_6\text{Sn}_5$  intermetallic. As the aging progresses, the scallop morphology becomes unfavorable because of the high interfacial energy between the bulk solder and the  $\text{Cu}_6\text{Sn}_5$  intermetallic. Therefore, during solid-state aging, the grain disappears and the scallop changes to a layer type with a flat surface.

The presence of Zn in the solder is expected to form a  $\text{Cu}_5\text{Zn}_8$  intermetallic compound as a result of the reaction between the solder and the substrate. However, in this study, this intermetallic compound was not observed. This was because the maximum amount of Zn added was only 0.7 wt. %.

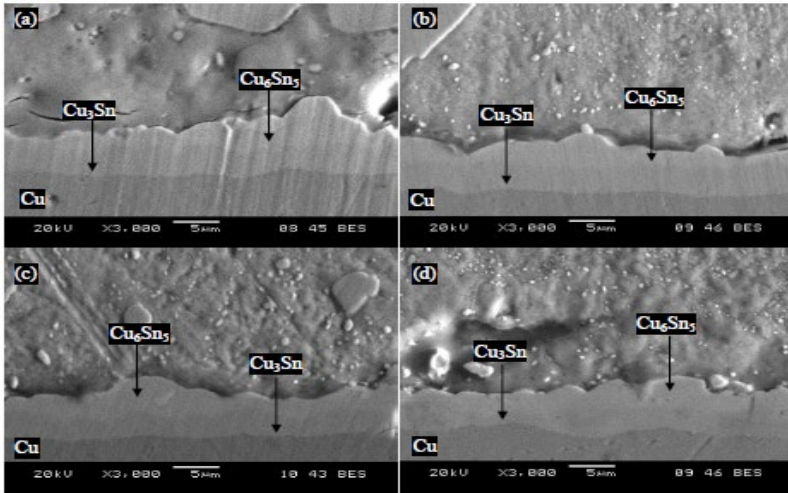


Figure 4: SEM images after aging for 700 h at 150 °C: (a) SAC (b) SAC-0.1Zn (c) SAC-0.4Zn and (d) SAC-0.7Zn.

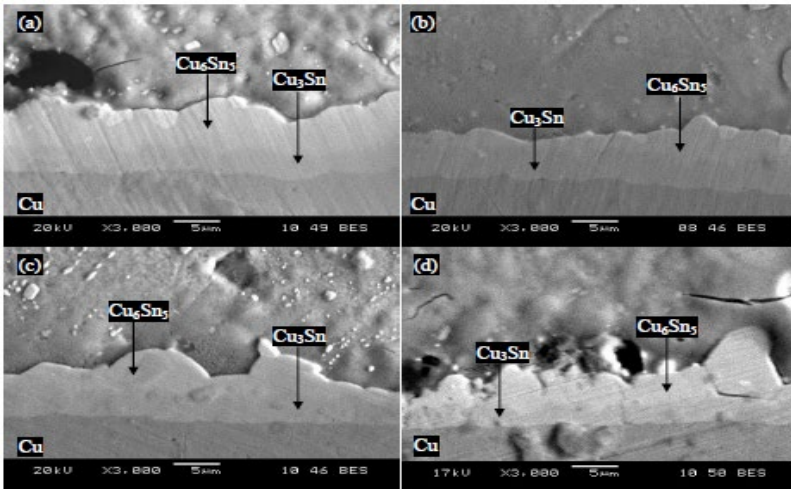


Figure 5: SEM images after aging for 900 h at 150 °C: (a) SAC (b) SAC-0.1Zn (c) SAC-0.4Zn and (d) SAC-0.7Zn.



The thickness of the  $\text{Cu}_6\text{Sn}_5$  intermetallic generally decreased with the addition of Zn [7]. The mechanism of Zn retardation remains unclear. Zn might have formed a Cu-Zn intermetallic, which reduces the ability of Cu to form Cu-Sn intermetallics. Some studies have shown that with the addition of minor amounts of Zn in Sn-based solder, the formation of the  $\text{Cu}_6(\text{Sn},\text{Zn})_5$  phase is possible. This intermetallic phase is formed during thermal aging, whereby Zn atoms diffuse into the Cu-Sn intermetallic [14].

Some studies have shown that the addition of Zn to Sn-Ag-Cu solder retards the growth of  $\text{Cu}_3\text{Sn}$  intermetallic. According to Kang *et al.* [15], Zn atoms accumulate at the interface, forming a thin layer and acting as a diffusion barrier for Cu atoms that contributes to the retardation of the  $\text{Cu}_3\text{Sn}$  intermetallic. Cho *et al.* [16] concluded that the Zn accumulated layer might be a Cu-Zn intermetallic layer. This thin layer was not detectable under a Scanning Electron Microscope.

## Stress-strain Results

Figure 6 shows a graph of the stress at max for the Sn-3.5Ag-1.0Cu-xZn solders. The stress at max is the maximum load per unit area required to break the solder joint. The graph shows a decreasing trend as aging time increases. This trend is expected because aging of the solder joint will decrease the joint strength. For the as-soldered solders, the stress values for all the compositions were above 20 MPa. This decreasing trend continued until the aging process ended at 1000 h. This final stress values were in the range of 14 - 16 MPa. Decreasing stress values indicated that the joint was brittle, and only a small load was required to break the joint.

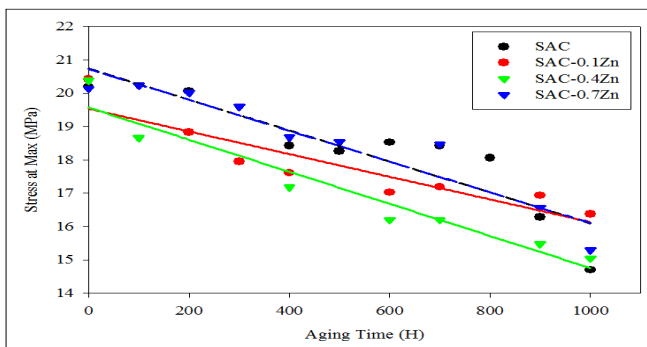


Figure 6: Stress at Max values for lead free Sn-3.5Ag-1.0Cu-xZn.

The stress at max values for the Sn-3.5Ag-1.0Cu solder varies from 14.70 to 20.19 MPa. With the addition of 0.1 and 0.4 wt.% Zn, the values vary between 16.38 to 20.42 MPa and 15.05 to 20.39 MPa, respectively. When there is 0.7 wt.% addition of Zn, the stress values at max are between 15.30 to 20.14 MPa.

The addition of Zn to the Sn-3.5Ag-1.0Cu solder was expected to improve the solder joint because the intermetallic layer thickness was thinner than that of Sn-3.5Ag-1.0Cu. However, no significant changes were observed as predicted. From the graph, only the Sn-3.5Ag-1.0Cu-0.7Zn solders exhibited higher values than Sn-3.5Ag-1.0Cu. The stress at max for the Sn-3.5Ag-1.0Cu-0.1Zn and Sn-3.5Ag-1.0Cu-0.4Zn solders was lower than that for Sn-3.5Ag-1.0Cu, indicating that they are brittle. It is believed that the nature of Zn affect solder joints. Morgan stated that the resistance of Zn to prolonged stress is not high, and that the metal is prone to creep [17].

Liu *et al.* suggested that the brittle fracture that occurred in the intermetallic layer resulted from the combined effect of the fragile intermetallic layer and stress concentration at the interface [18]. In general, intermetallics tend to be hard and brittle materials;- these properties are related to their ionic or covalent bonding. If they are present in large quantities or lie along the grain boundaries, the overall alloy can be extremely brittle. If the same intermetallic can be uniformly distributed throughout the structure in small particles, the alloy can be considerably strengthened [19]. This explains that even 0.4 wt.% Zn addition has the thinnest intermetallic, but it joints tend to be brittle.

Figure 7 shows the graph of strain at max for the Sn-3.5Ag-1.0Cu-xZn solders after the aging process. The strain at max indicates the maximum change in length before the solder joint breaks. The graph shows a decreasing trend of the strain at max for all solder alloy systems during the aging process. The solder system without the addition of Zn appeared to have the highest strain rate compared to the solder system with the addition of Zn.

The maximum strain for Sn-3.5Ag-1.0Cu solders decreased from 9.09 % to 3.25 % during the aging process. With 0.1 wt.% addition of Zn, the strain at max is in the range of 8.85 to 3.84 %. Among the three Sn-3.5Ag-1.0Cu-xZn solders, Sn-3.5Ag-1.0Cu-0.4Zn had the lowest strain

values, ranging from 5.31 to 3.20 %. For Sn-3.5Ag-1.0Cu-0.7Zn, the as-soldered solder showed a strain value of 6.06 %, which decreased to 3.84 % when aged for 1000 h. The degradation of solder joint is related to the intermetallic thickness and kinetics of intermetallic growth during aging. The addition of Zn significantly retarded the growth of  $\text{Cu}_6\text{Sn}_5$ . This shows that the zinc was effective to suppress the IMCs in solder sample. The mechanism of the retardation by Zn is still very unclear. Zn might have formed Cu–Zn intermetallic which reduces the ability of Cu to form Cu–Sn intermetallic. But the Cu–Zn intermetallic was not detected in the EDX analysis. Furthermore, researchers [20-22] found that, after solid state aging, severe voiding, known as the Kirkendall void, was found at the interfacial  $\text{Cu}_3\text{Sn}$  intermetallic layer, which greatly degraded the solder joint. A voided  $\text{Cu}_3\text{Sn}$  layer cannot provide the mechanical support needed to maintain the integrity of solder joints [23].

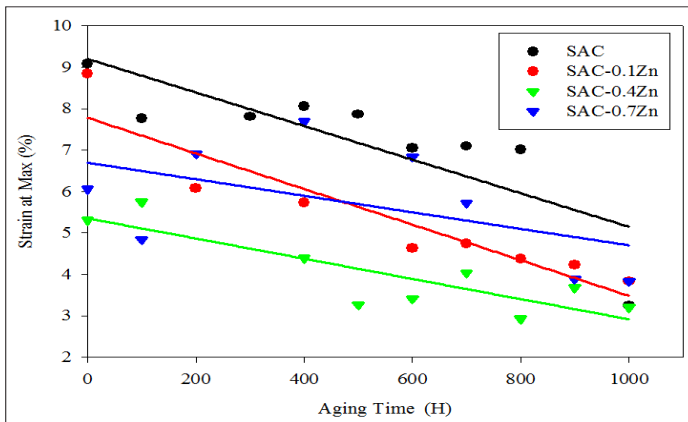


Figure 7: Strain at Max values for lead free Sn-3.5Ag-1.0Cu-xZn.

The mechanical behavior of a solder joint depends on its layered structure. The solder in the middle of the sandwich was yielding and ductile. When the stress and strain are high, the solder is ductile, which means that it can absorb stresses that may occur. However, when the stress and strain are low, the solder consists of crystals that are strong, hard, or brittle. The solder joints in this state are strong in shear and tension, but because of their brittle nature, they are relatively easy to peel apart [24]. Therefore, the solder joint will shift from ductile at lower aging times and become brittle at higher aging times.

## Fracture Surface of Solder Joint

Figure 8 and 9 show the difference in the fracture surface of the joint strength for the as-soldered and aged for 1000 h, respectively. Different structures are shown in both figures.

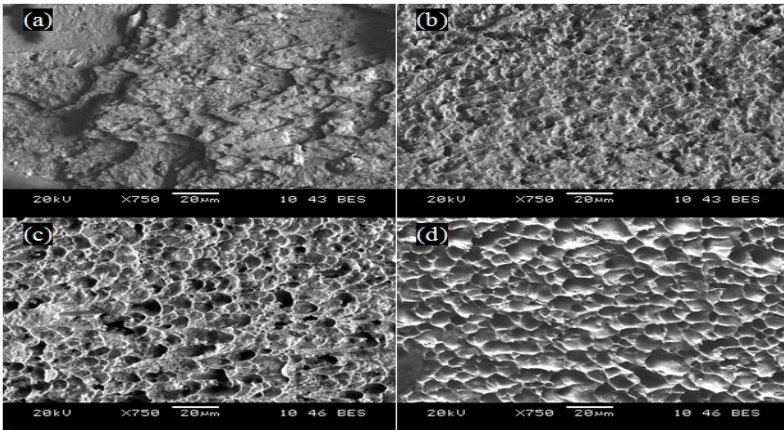


Figure 8: Fracture surface of solder joint by SEM for as-soldered: (a) SAC, (b) SAC-0.1Zn, (c) SAC-0.4Zn, (d) SAC-0.7Zn.

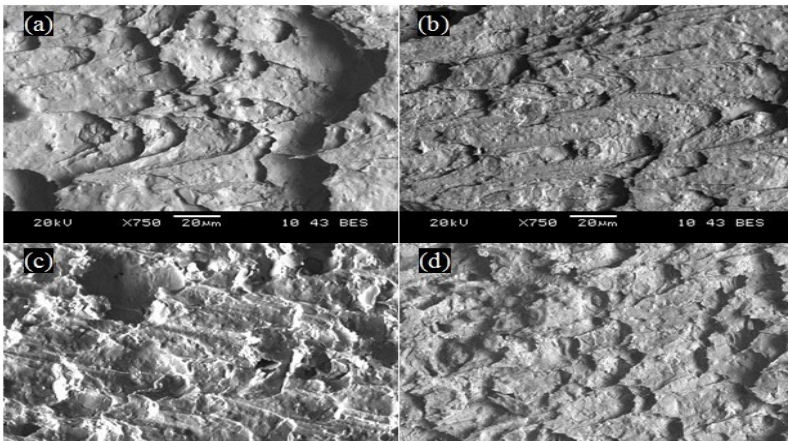


Figure 9: Fracture surface of solder joint by SEM for 1000 h aging time: (a) SAC, (b) SAC-0.1Zn, (c) SAC-0.4Zn, (d) SAC-0.7Zn.

For the as-soldered solder, the surface morphology is dimpled, which indicates that the solder is ductile. The ductile mode was displayed for all as-soldered solders as shown in Figure 8. According to DeGarmo *et al.* [19], the extent to which a material exhibits plasticity is significant for evaluating its suitability for certain manufacturing processes. For example, metal deformation processes require plasticity; the more plastic a material is, the more it can be deformed without rupture. The ability of a material to deform plastically without fracture is known as ductility [19].

Meanwhile, after 1000 h of aging, the structure became coarser, indicating that the all solders were brittle. This appears to agree with the research conducted by Takemoto *et al.* [25], who claimed that aging at elevated temperatures coarsened the microstructure. Coarsening of the microstructure is generally expected to decrease the tensile strength. DeGarmo *et al.* [19] claimed that brittleness should not be considered a lack of strength, but simply a lack of significant plasticity. In many cases, brittle fractures occur because of defects in the metal. These defects are either formed during the manufacturing stage or developed during service [26]. The transition from ductile to brittle behavior is called ductile-to-brittle transition (DBT).

During the soldering and aging processes, the solder constituents diffused towards the Cu substrate. The thickness of the diffusion zone depended on several factors. The first factor is the maximum temperature reached during soldering and secondly is the length of time during which the substrate was exposed to the molten solder which was termed as “aging time”. The higher the temperature reached during soldering, the longer the aging time, and the thicker the intermetallic layer [24]. According to Strauss [24], aging and the consequent grain coarsening lower the mechanical strength and ductility of solder joints. The thickening of the brittle intermetallic layer reduces its ability to absorb repeated deformation without cracking [24]. This can be explained by the addition of 0.4 wt. % of Zn, although this composition successfully suppressed the intermetallic growth [7], its joint strength is low compared to other composition.

## CONCLUSIONS

In this study, the effects of minor Zn (0, 0.1, 0.4, 0.7 wt.%) addition into Sn-3.5Ag-1.0Cu lead free solder in terms of intermetallic formation and joint strength. Minor Zn addition has a significant impact on intermetallic formation. The first intermetallic layer formed on the copper substrate was  $\text{Cu}_6\text{Sn}_5$ . After the aging process, there was formation of  $\text{Cu}_3\text{Sn}$  in between the copper substrate and the  $\text{Cu}_6\text{Sn}_5$  intermetallic compound. Initially,  $\text{Cu}_6\text{Sn}_5$  appeared as scallop before changing to a planar morphology.  $\text{Cu}_3\text{Sn}$  has a planar morphology and remains throughout the aging process. The stress and strain showed a decreasing trend as the aging time increased. The solder joint was ductile at low aging times and its properties became brittle as the aging time increased. Although 0.4 wt% Zn beneficial in suppress the intermetallic thickness, but its tensile strength is lacking compared with Sn-3.5Ag-1.0Cu, Sn-3.5Ag-1.0Cu-0.1Zn and Sn-3.5Ag-1.0Cu-0.7Cu. The solder with 0.7 wt% of Zn gives highest stress at max while for strain at max, Sn-3.5Ag-1.0Cu solder give the highest value. The fracture mode displays ductile failure at lower aging temperatures and times, but shows brittle failure at higher aging temperatures and longer times.

## ACKNOWLEDGMENT

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