The Evolution of Microstructures and Mechanical Properties of SnAgCu/Cu Weld Interface during Isothermal Aging

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Abstract. Based on the model of the diffusion flux ratio of Cu and Sn at the Cu/Cu₃Sn/Cu₆Sn₅/Sn interface, the evolution of microstructures, the behavior of formation and growth of intermetallic compound (IMC) for Sn95.5Ag3.8Cu0.7 solder aged at 150 °C were studied. The nano indentation was applied to measure the hardness and elastic modulus of the IMC. The tensile strength and the low cyclic fatigue properties of the weld joint were also measured. The results showed that the thickness increments of Cu₃Sn and Cu₆Sn₅ phases of IMC are mainly controlled by diffusion mechanism. The growth rates of Cu₃Sn and Cu₆Sn₅ are 7.86×10⁻¹⁹ m²/s and 7.80×10⁻¹⁷ m²/s, respectively. The hardness and elastic modulus of IMC enhance with increasing aging time. For 24h aging time, the microstructure of the Cu₆Sn₅ keeps scalloped shape and the elastic modulus of IMC layer are similar to the copper substrate, which result in high fatigue resistance of the welded joint and its tensile strength of 69.50 MPa. With the mechanical hardening cumulative effects resulting from aging and cyclic strain, the fatigue performance and tensile strength of the welded joint gradually worse with the further increase of the aging time.

1. Introduction
The Sn-Ag-Cu alloys have been found wide application in electronic industry as mainstream standard lead-free solders replacing the conventional Sn-Pb solders [1-3]. The working temperature of solder joints will reach 90-150 °C as a result of thermal cycle and mechanical vibration impact [4-5]. As the service time increases, the microstructure, deformation, and fracture mechanism of solder joints will change. The interaction of cyclic strain and high temperature leads to the reduction of mechanical properties of solder joints and the disequilibrium of physical and electrical properties of solder joints [6-10]. In this paper, the isothermal aging treatments were performed at 150°C for the Sn95.5Ag3.8Cu0.7/Cu solder joints. The microstructures, compositions, and growth rates of the IMC (intermetallic compound) of solder joints were studied. The evolutions of the mechanical properties and fatigue performances of IMC were also analyzed. It can attribute to the evaluation of the lifetime and reliability of micro-joints in advanced packaging technology.

2. Experimental Method
The brazing substrates used in this experiment were copper sheets with size of 25 × 25 × 0.2 mm³. The TU1 boards were cleaned ultrasonically in acetone for 15 min. After water washing, the boards were soaked in alcohol for 30 min and then dried in the drying oven. The solders with a mass of 0.2 g were covered completely by scaling powders. The mass ratio of solders and scaling powders was 9:1. The samples were transferred slowly into a furnace with temperature of 350 °C for fusion. Then, the brazing state samples were putted into a vacuum oven for aging treatment. The temperature of aging...
treatment was 150 °C and the time varied from 1 d to 5 d. After the aging treatment, the cross sections of solder joints were grinded, polished, and etched. The corrosive liquid was composed of C₂H₅OH (100 mL), HCl (25 mL), and FeCl₃ (10 g). The compositions of samples were measured by energy dispersive spectrometer (EDS). The microstructures of prepared samples were analyzed by electron microscope (AMRAY-100B). The average thickness of IMC in sample interface was measured by element line scan analysis method of the EDS.

According to the stretching and shear experiment method of lead-free solder joint, the tensile strength of welding joints was measured by microcomputer-controlled electronic universal testing machine (Shenli WDW-100). During the stretching experiment, the force control of 0.5 kN/sec was performed in the first stage while the second stage was displacement control (1.5 mm/sec). The hardness and elastic modulus of IMC layers in the solder/Cu interface were measured by a Nano indentation experiment system (Nano Indenter G200) with the pressure head of Berkovich shape, the loading rate of 10 nm/s, and the poisson ratio of 0.35. Based on the mechanical displacement method, the low cycle fatigue tests were performed for the welded joints by an electro-hydraulic servo fatigue machine (Shimadzu, EHF-EM100K-020-1A). The temperature and relative humidity of fatigue tests were 293K and 80%, respectively. A triangular waveform with the frequency of 1 Hz and the displacement of 0.03 mm was used for the fatigue tests.

3. Results and Discussion

3.1. The Morphology Changes of IMC

Figure 1 shows the SEM images of microstructures of solder/Cu interfaces after aging for different time. The EDS measurements were carried out for the scalloped compounds A and columnar compounds B in figure 1(a). The results show that in both scalloped and columnar compounds, the atomic ratio of copper and tin is about 6:5. It can conclude that the IMC of point A and point B is Cu₆Sn₅ phase. Moreover, the atomic mass percentage of silver at point A and point B are 0.87% and 0.34%, respectively, indicating that the silver atoms participate in the interface reaction of IMC. Figure 1(b) reveals the disappearance of long cylindrical IMC after aging for 24 h. The IMC at the solder side presents semicircular scallop shape. As shown in figure 1(c), after 48 h of aging treatment, the large diffusion migration of Cu and Sn atoms produces compressive stress. It results in the accumulation of Cu atoms as well as high nucleation rate of Cu₆Sn₅ in the high Cu ratio region. Therefore, the scalloped compounds grow into bamboo-like Cu₆Sn₅. Some IMCs fall into the solders due to the present cracks at the bottom of IMC. For the samples after 72 h of aging treatment, there are Kirkendall voids at the bottom of the Cu₆Sn₅ columnar crystal and grain shape Ag₃Sn phase segregation along grain boundaries of the solder (figure 1(d)). The welding interface of IMC after for 96 h can be seen in figure 1(e), due to the lower diffusion of tin atoms from solders than that of copper atoms, it needs a great deal of tin to grow Cu₃Sn IMC. Meanwhile, some long columnar or bamboo-like Cu₆Sn₅ fractures into the solders in the form of debris owing to the concentrated compressive stress of Cu₆Sn₅. Hence, the thickness of Cu₆Sn₅ phase reduces. The prolonged high-temperature aging increases the imbalanced interdiffusion between copper and tin atoms of Cu₃Sn and Cu₆Sn₅. It results in the formation of Kirkendall voids in IMC layer. The stress concentrates on the Kirkendall voids. The voids lead to the formation of cracks which are perpendicular to the growth direction of the IMC layer. When the aging time increases to 120 h, as shown in figure 1(f), there are many Kirkendall voids in Cu₃Sn and Cu₆Sn₅. Serious segregation happens for the granular Ag₃Sn. The Kirkendall voids [11] and segregation will significantly affect the mechanical property of the welding.
Figure 1. SEM images of the microstructures of solder/Cu interfaces after aging for different time

3.2. Dynamics Model of IMC

During the aging process, if the growth of IMC layer was controlled by atomic diffusion, the growth will follow the formula (1) [12-14]

\[ Y = Y_0 + kt^{1/2} \]  

where \( Y \), \( Y_0 \), \( k \), and \( t \) are the thickness of IMC, initial thickness, growth rate constant, and reaction time, respectively.

Figure 2 shows the relation between the thickness increment of Cu₆Sn₅ and Cu₃Sn in the IMC layers of Sn₉₅.₅Ag₃.₈Cu₀.₇ solder / Cu substrate and the aging time \( t^{1/2} \). For the aging time between 0-72 h, the thickness of Cu₆Sn₅ phase increases with the aging time, but when the aging time larger than 96 h, the thickness of Cu₆Sn₅ phase reduces owing to the partial exfoliation and reaction consumption. Therefore, the thickness increments of Cu₆Sn₅ are fitted limitedly in the aging time between 0-72 h. By multiple linear regression analysis methods, the relationship between thickness increment and square root of aging time is approximate linear and the slope \( k^2 \) is \( 7.80 \times 10^{-17} \text{ m}^2/\text{s} \). It indicates that the growth mechanism of Cu₆Sn₅ is parabola thickening, that is, the growth of Cu₆Sn₅ approximately follows the atomic diffusion law. In addition, the thickness of Cu₃Sn increases continuously throughout the aging process. As seen in figure 1, there are a small quantity of Kirkendall voids and cracks in Cu₃Sn only after 96 h of aging treatment. But the Cu₃Sn do not peel off after 96 h. By regression analysis, the thickness increment of Cu₃Sn phase and the aging time are approximately linear relationship with a slope \( k^2 \) of \( 7.86 \times 10^{-19} \text{ m}^2/\text{s} \), which is 2 orders of magnitude lower than that of the Cu₆Sn₅ phase.
According to the above results, the aging process of Cu$_3$Sn and Cu$_6$Sn$_5$ IMC of Sn$_{95.5}$Ag$_{3.8}$Cu$_{0.7}$ / Cu solder follows the atomic diffusion theory. In order to simplify the calculation model, the interfaces of Cu/Cu$_3$Sn ($\varepsilon$), Cu$_3$Sn ($\varepsilon$)/Cu$_6$Sn$_5$($\eta$), and Sn/Cu$_6$Sn$_5$($\eta$) are assumed to be flat. Figure 3 shows the model of diffusion of Cu and Sn atoms. If the thicknesses of Cu$_6$Sn$_5$($\eta$) phase and Cu$_3$Sn($\varepsilon$) phase are $m$ and $n$, respectively, according to the Fick’s first law:

$$J = D \frac{dC}{dx}$$ (2)

In the steady-state diffusion condition, we assume that at the two phase interface, the atomic diffusion fluxes of Sn in $\eta$ phase and Cu in $\varepsilon$ phase are $J_{Sn}^\eta$ and $J_{Cu}^\varepsilon$, respectively. By the integration of Eqn. (2), the diffusion fluxes at the $\varepsilon$ / $\eta$ interface are given by

$$J_{Cu}^\varepsilon = \frac{D_{Cu}^\varepsilon (C_{Cu}^{\varepsilon/\eta} - C_{Cu}^{\varepsilon/\eta})}{n}$$ (3)

$$J_{Sn}^\eta = \frac{D_{Sn}^\eta (C_{Sn}^{\eta/\varepsilon} - C_{Sn}^{\eta/\varepsilon})}{m}$$ (4)

where $D$ is the diffusion coefficient, $C$ is the molar concentration of Cu or Sn at the interface. The diffusion flux ratio ($r$) of Cu and Sn at the interface is given by

$$r = \frac{J_{Cu}^\varepsilon}{J_{Sn}^\eta} = \frac{mD_{Cu}^\varepsilon (C_{Cu}^{\varepsilon/\eta} - C_{Cu}^{\varepsilon/\eta})}{nD_{Sn}^\eta (C_{Sn}^{\eta/\varepsilon} - C_{Sn}^{\eta/\varepsilon})}$$ (5)

The atomic ratio is transformed into molar ratio as shown in Table 1. The molar quantities of Sn atoms and the molar volume ($V_n^\varepsilon$) of Cu$_3$Sn ($\varepsilon$) phase are assumed to be invariant after the Cu atoms.
dissolved into Cu$_3$Sn (ε) phase. The molar ratios of Cu atoms at the two interfaces of Cu$_3$Sn layer can be obtained as $C_{\text{Cu}}^{\epsilon} = \frac{n_{21}^{\text{Cu}}}{n_{21}^{\text{Sn}}} \frac{V_{\epsilon}^m}{V_{m}^{\epsilon}}$ and $C_{\text{Cu}}^{\eta} = \frac{n_{23}^{\text{Cu}}}{n_{23}^{\text{Sn}}} \frac{V_{\eta}^m}{V_{m}^{\eta}}$. The molar ratios of Sn atoms at the interface are $C_{\text{Sn}}^{\epsilon} = \frac{n_{34}^{\text{Sn}}}{n_{34}^{\text{Cu}}} \frac{V_{\epsilon}^m}{V_{m}^{\epsilon}}$ and $C_{\text{Sn}}^{\eta} = \frac{n_{32}^{\text{Sn}}}{n_{32}^{\text{Cu}}} \frac{V_{\eta}^m}{V_{m}^{\eta}}$.

The relevant molar volumes of the Cu$_3$Sn(ε) and Cu$_6$Sn$_5$ (η) phases are calculated by their density (8.90 g/cm$^3$ and 8.28 g/cm$^3$ for Cu$_3$Sn and Cu$_6$Sn$_5$ respectively) and molar mass. The obtained molar volumes of Cu$_3$Sn(ε) and Cu$_6$Sn$_5$ (η) phases are $V_{m}^{\epsilon} = 34.94$ cm$^3$/mol and $V_{m}^{\eta} = 118.24$ cm$^3$/mol, respectively[15]. Following formula is then available:

$$r = \frac{\frac{n_{21}^{\text{Cu}}}{n_{21}^{\text{Sn}}} \frac{V_{\epsilon}^m}{V_{m}^{\epsilon}} \times \frac{m}{n}}{\frac{n_{34}^{\text{Sn}}}{n_{34}^{\text{Cu}}} \frac{V_{\epsilon}^m}{V_{m}^{\epsilon}}}$$

The value of $\frac{D_{\text{Cu}}^{\epsilon}}{D_{\text{Sn}}^{\eta}}$ can be considered as a constant during the isothermal aging process. The value of $k = \frac{V_{m}^{\eta} D_{\text{Cu}}^{\eta}}{V_{m}^{\epsilon} D_{\text{Sn}}^{\eta}}$ is between 6.77 and 10.14 (taking $k=8$ for the calculation). By measuring the thicknesses of Cu$_3$Sn and Cu$_6$Sn$_5$ phases at different aging time, the diffusion fluxes ratio (r) of Cu and Sn at the interface are obtained and the results are listed in Table 2. The diffusion flux of Sn in the Cu$_6$Sn$_5$ phase is far lower than that of Cu in Cu$_3$Sn phase. After the welding of Sn$_{95.5}$Ag$_{3.8}$Cu$_{0.7}$, the value of $m/n$ is the maximum and the diffusion fluxes ratio (r) of Cu and Sn is large because of the less thickness ($0.08$ μm) of Cu$_3$Sn phase. The thicknesses of Cu$_3$Sn and Cu$_6$Sn$_5$ phases increase when the aging time increases from 24 to 72 h. But the value of $r$ reduces with increasing aging time, indicating that the diffusion flux of Sn atoms enhances at the Cu$_3$Sn/Cu$_6$Sn$_5$ interface. The reaction is Sn + 3Cu $\leftrightarrow$ Cu$_3$Sn. The diffusion flux of Sn atoms can consume 3 times the quantity of Cu at the interface. Furthermore, the Cu$_6$Sn$_5$ phase is reduced and its growth rate is inhibited. Furthermore, combination with the microstructures in Fig. 1, after 48 h of aging treatment, the Cu$_6$Sn$_5$ phase in some Cu-rich regions rapidly grow as bamboo accompanied by fracturing into the solder. The increment rate of the thickness of Cu$_6$Sn$_5$ phase reduces, leading to the decreased diffusion fluxes of Cu and Sn.

### Table 1. EDS analysis results of the solder/Cu interface after soldering

| element | $n_{21}$ (at%) | $n_{23}$ (at%) | $n_{32}$ (at%) | $n_{34}$ (at%) |
|---------|----------------|----------------|----------------|----------------|
| Cu      | 76.62          | 75.05          | 52.33          | 50.26          |
| Sn      | 23.38          | 23.85          | 47.65          | 48.87          |

### Table 2. The ratio of Cu to Sn diffusion fluxes at the SnAgCu/Cu interface

| Aging time/h | 0 | 24 | 48 | 72 |
|--------------|---|----|----|----|
| m/μm         | 2.28 | 5.11 | 5.78 | 6.88 |
| n/μm         | 0.08 | 0.20 | 0.28 | 0.35 |
| r            | 231.19 | 207.26 | 167.45 | 159.46 |

### 3.3. The Mechanical Properties of Welded Joint

#### 3.3.1. The Nano Indentation Hardness and Elastic Modulus

Due to the micron sized weld microstructure, the hardness-displacement curves are by Nano indentation continuous measurement and shown in figure 4. In figure 4, the material hardness is high when the depth of Berkovich
indentation is shallow, and the hardness tended to stabilize with increasing indentation depth. In the initial pressing stage, the material surface appears work hardening characteristics due to a series of grinding and polishing process. Without aging treatment, the hardness of Cu$_6$Sn$_5$ phase in IMC layer is 0.98 GPa, which is between the hardness of pure copper and solder. After aging for 24 h, the hardness of IMC is 1.34 GPa, which is very close to the hardness of pure copper substrate. When the aging time is 48 h, the hardness of IMC (1.40 GPa) is slightly higher than that of the copper. The hardness enhances continuously when the aging time increases to 72, 96, and 120 h.

Table 3 lists the elastic modulus of each soldered joint. The elastic modulus of Cu$_6$Sn$_5$ in IMC layer enhances gradually with increasing aging time. It reveals that the increasing aging time results in reducing elastic deformation of IMC layer in solder joint interface. The Cu$_6$Sn$_5$ IMC, which is dominating in the solder joint interface, has hexagonal prism crystal structure [16-17]. The hexagonal prism structure has less dislocation width and less and sparse atoms in the interface. After aging treatment, some impurity elements, such as oxygen, carbon, and hydrogen, are easily aggregated on the dislocation area. It hinders the dislocation action and increases the slip resistance. The difficult deformation of IMC leads to the reduced plasticity. The twin and cleavage fracture occur easily by external force. Hence, the IMC is hardening brittleness. The elastic modulus of pure copper substrate declines slowly during aging. The atoms arrange densely in the lattice plane of pure copper due to the face-centered cubic crystal structure. The dislocation width is large and the dislocation pileup is uneasy to happen. The vacancy mobility increases during the heating process. Therefore, more dislocations jump out of the pileup, resulting in the pure copper softening. Compared with the pure copper and IMC, the elastic modulus of Sn$_95.5$Ag$_{3.8}$Cu$_{0.7}$ solder is the minimum. The elastic moduli of IMC and pure copper substrate are closest for aging time of 24 and 48 h.

![Figure 4](image-url)

**Figure 4.** The hardness-displacement indentation curves for materials in solder/Cu interfaces

| Table 3. Elastic modulus E (GPa) of the different material in the welded joint |
|---------------------------------|------------------|------------------|------------------|------------------|------------------|------------------|
| Materials                      | Aging time/h     |
|                                | 0    | 24   | 48   | 72   | 96   | 120  |
| IMC                            | 70.01| 104.12| 118.34| 128.51| 130.06| 134.78|
| Cu                             | 124.25| 110.03| 108.00| 107.32| 107.00| 102.76|
| Sn$_{95.5}$Ag$_{3.8}$Cu$_{0.7}$| 48.25| 47.75| 42.88| 45.00| 46.05| 43.98|
3.3.2 The Tensile Strength. During the soldering process, The IMCs are formed from the reaction between the solder and the copper substrate. The difference in thermal expansion coefficient among IMC, Cu substrate, and solder affects the deformation resistance (tensile strength) of solder joints.

Figure 5 shows the change of tensile strength of Sn95.5 Ag3.8 Cu0.7 / Cu solder joints with aging time. The tensile strength of welded joint is the largest (69.50 MPa) after 24 h of aging. For aging time of 48 h, the tensile strength is 68.29 MPa, which is the secondly largest. For the aging time increases from 0 to 48 h, the Cu5Sn and Cu6Sn5 in the IMC layer of solder joint interface grow continually and the thickness of IMC increases. As can be seen from the SEM images in Fig. 1, the IMC at the solder side presents semicircular scallop shape for aging time of 24 h. When the aging time increases to 48 h, a small amount of bamboo-like Cu6Sn5 phases appear in the interface. But the IMC still maintains complete and smooth scallop shape. There is a "bite" effect between the IMC and solder. It indicates that the microstructures of Cu6Sn5 IMC with scallop characteristics are essential condition to ensure metallurgical bonding force of weld joint and interface strength of welding. Table 3 shows that the elastic moduli of IMC and copper substrate are the closest for aging time of 24 h. The elastic modulus is external reflection of the interatomic bonding force. The elastic modulus and thermal expansion coefficient closely relate to the crystal structure and inter-atomic forces. Their relation is determined by

$$E = E_0 e^{m \int \alpha(t)dt}$$  

Where $m$ is a constant, $t$ is the temperature, $\alpha$ is the thermal expansion coefficient, and $E_0$ is the elastic modulus of material at Debye temperature [18]. For aging time of 24 h and 48 h, the difference in thermal expansion coefficient between IMC layer and pure copper is small, which result in the tensile strength of welding spot interconnected with different materials is high. A large number of copper atoms have cluster to produce Cu6Sn5 compound in a long columnar way toward the internal solder, which makes the IMC brittle and its tensile strength begins to reduce after 72 h aging. For the aging time larger than 96 h, the elastic modulus of IMC is far higher than the elastic modulus of pure copper substrate which causes their mismatch of expansion coefficients between IMC and pure copper substrate. With increasing aging time, the Cu6Sn5 phase becomes harder, more Ag3Sn cluster in the solder, and more new Kirkendall voids form or the primary voids enlarger in the IMC, which give rise to the welded joint fracture easier, consequently the tensile strength of welding joint decreases to 35.02 MPa when the aging time is 120h.

Figure 6 shows that the maximum tensile stress and compressive stress of the hysteresis loop at

3.4. The Low Cycle Fatigue Properties of Welded Joints

In the symmetric cyclic strain test with constant amplitude (0.03mm), a series of stress and strain were obtained through monitoring the response of stress and strain and the results are shown in figure 6. Due to the clamping state of fixture and other components of the device could be achieved after the absorption of their own space, the hysteresis loop curves in this experiment begin from the 10th cycle. Figure 6 shows that the maximum tensile stress and compressive stress of the hysteresis loop at
different aging time of solder joints are different. The Bauschinger's effect [19] appears due to the residual stress and dislocation pileup formed by the repeated plastic deformations. As seen from the tensile cyclic stress-strain curve of solder tensile samples without ageing treatment in figure 6(a), the difference is very small in the 200th cycle and fifth cycle. When the number of cycles reaches 3500, the plastic strain amplitude is 0.008 mm and the elastic strain amplitude is 0.022 mm. It indicates that the Sn95.5Ag3.8Cu0.7/Cu samples have a wide range of elastic deformation by the displacement of ±0.030mm. Compared with welded joint without aging, the areas of the 5th cycle and 200th cycle of stress-strain curves of welded sample after 24 h aging treatment are smaller. It reveals that in the early stages of the tensile fatigue test, the strain energy density that the plastic deformation requires is smaller than that of welded joint without aging. For the cycle increases to 1200-3500, the fatigue properties are similar with figure 6(a). Figure 6(c-f) show the cyclic stress-strain curves of solder joint for aging time varies from 48 to 120 h. With the increase of aging time, the maximum tensile stress of hysteresis loop reduces and the plastic strain amplitude enhances. The plastic deformation occurs easily because the hardness of Cu6Sn5 in IMC layer enhances with increasing aging time. The initial hardness of IMC is superimposed with the cyclic strain hardening in the fatigue tests, leading to the IMC layer becomes harder and more brittle with increasing number of deformation. As a result, the samples are broken in the 2000th cycle for the aging time of 96 h. In the same condition, the fatigue life is only 1300 cycles when the aging time is 120 h.

Figure 6. SnAg3.0 Cu0.5 / Cu welding tensile samples in each cycle hysteresis loop

4. Conclusion
(1)During the aging time of Sn95.5Ag3.8Cu0.7/Cu increases from 0 to 120 h, the diffusion flux ratio of Cu and Sn atoms \( r \) is far larger than 1. But the value of \( r \) reduces with increasing aging time. The growth of Cu3Sn and Cu6Sn5 phases approximately follows the diffusion law. The growth rates of Cu3Sn and Cu6Sn5 are \( 7.86 \times 10^{-19} \) m\(^2\)/s and \( 7.80 \times 10^{-17} \) m\(^2\)/s, respectively.

(2)With the increase of aging time, the hardness and elastic modulus of Cu6Sn5 phase in IMC increase while the hardness and elastic modulus of pure copper substrate reduce slightly. For aging time of 24 h, the IMC layer still maintains the Cu6Sn5 phases with scallop. The hardness and elastic modulus of IMC are 1.34 and 104.12 GPa, respectively. The hardness and elastic moduli of IMC layer and that of the pure copper substrate are close. The low cycle fatigue resistance of solder joint is the best and its tensile strength reach the maximum (69.50 MPa). When the aging time is larger than 24 h, the elasticity modulus and hardness of IMC enhance with increasing aging time. The Cu6Sn5 phases
with column and bamboo shape and Kirkendall voids increase, leading to the reduced tensile strength and low cycle fatigue resistance.

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6. References
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