Article

Creep Properties Assessment by Shear Punch Creep Test and IMC Morphology of Aged Pb-Free Solder Joint/UBM

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Abstract: In this paper, we investigated the intermetallic compound (IMC) morphology of aged (0, 200, 600 h) conventional Sn-37Pb and Pb-free Sn-4Ag-0.5Cu solder joints/(Ni-P/Au) UBM; the creep properties of the solder joints were evaluated using a shear punch creep test (SPCT) method. The creep displacement-time curves of the solder joints exhibited different behaviors depending on stress application and aging treatment conditions. Empirical formulas such as the Power-law and Monkman-Grant relationship have been used to analyze the SPCT data. Furthermore, the IMC behavior of solder joints was investigated using energy-dispersive X-ray spectroscopy (EDS) and a scanning electron microscope (SEM). The result showed that with an increase of the aging time, the stress exponents (n) of solder joints were decreased, but the IMC thickness and size were increased. In most of the experimental conditions, the creep properties of Pb-free solder joints were superior to the conventional Sn-37Pb solder joints.

Keywords: Pb-free solder joint; aging treatment; IMC; UBM; shear punch creep test (SPCT); creep

1. Introduction

Conventional Sn-Pb solder joints are commonly used as the most outstanding solder joint for the electronic packaging industry. Over the past decades, they have been used as the most effective bonding material of electronic devices due to their excellent mechanical properties such as a low melting point, wettability and fatigue resistance, and low cost [1,2]. However, conventional Sn-Pb solder alloys are treated as general waste and therefore have bad effects on the environment, such as air and water pollution; they especially have a bad effect on human health [3].

As a result, the European Union (EU) has enacted environmental regulations, which include the restriction of hazardous substances (RoHS), as well as the waste electrical and electronic equipment (WEEE) legislation, to forbid the use of lead (Pb) in electrical and electronic products. For that reason, research on Pb-free solder has received much widespread attention [4,5]; in particular, Sn-Ag-Cu solder alloys are currently known as being the most promising Pb-free solder joints [6]. Sun et al. [7] suggested that in the reflow processing, the eutectic Sn-Pb solder joints can be replaced with a eutectic Sn-Ag-Cu solder joint. In addition, for the wave soldering process, the eutectic Sn-Pb solder joint can possibly be replaced with the eutectic Sn-Cu alloy as well. In the electronic packaging, Ni-P/Au multilayers have been considered as a promising UBM with a micro-solder bump for flip chip technology due to their functions. This is because they have provided a good solderable surface and also have a function for protecting the underlying copper (Cu) from reacting with the solder, at a low-cost [8]. The IMC behavior is considered a crucial factor for predicting the mechanical properties of solder joints. After 200 °C HTS aging, the phase microstructure, thickness, and fracture morphology of IMC on the

interface of SAC solders/UBM have an influence on the pull strength of the joint [9]. On the other hand, the shear punch creep test method has been investigated by many researchers for over twenty years, and it has been applied to evaluate the ductile and brittle materials. As a result, this method is considered as an acceptable tool for determining material creep properties [10]. However, there have been few reports about applying this creep test method using the SPCT for solder joints.

The main purpose of this study is to assess and investigate the creep mechanical properties of aged Pb-free Sn-4Ag-0.5Cu solder joints/(Ni-P/Au) UBM using the SPCT method at 30 °C. The isothermal aging was conducted at 150 °C during 0, 200, 600 h. Furthermore, the IMC behavior of solder joints was observed using SEM and EDS. The mechanical properties of Pb-free Sn-4Ag-0.5Cu solder joints were compared with the conventional Sn-37Pb solder.

2. Experimental Section and Preparation of Test Specimens

2.1. Materials and Preparation of Test Specimens

In this research, the conventional Sn-37Pb solder joints and Pb-free Sn-4Ag-0.5Cu solder joints were used to evaluate the creep properties of the solder joint. Figure 1 shows the fabrication process of the specimen for the SPCT method. (Step. 1) The Cu substrates were machined and polished by sandpaper from # 800 to # 2000 grit, to obtain a $10 \times 10 \times 0.8$ mm size and a center hole of $\varnothing 3.2$ mm. (Step. 2) Multi-layers of Ni-P/Au UBM, about 5 and 0.1 μm , respectively, were deposited on the Cu substrates. (Step. 3) The solder balls were reflowed into the Cu substrate at reflow temperature (Sn-37Pb: 200 °C, Sn-4Ag-0.5Cu: 240 °C) for 30 s and were cooled at room temperature for 10 min. (Step. 4) The surfaces of specimens were polished to obtain a final size ($10 \times 10 \times 0.5$ mm). (Step. 5) Finally, SP specimens were treated to isothermal aging (AJ-SB4/9908) at 150 °C for 0, 200, and 600 h. The chemical composition of the solder joints is shown in Table 1. The experiment conditions and melting point temperature of the solder joints is summarized in Table 2.

Figure 1. Schematic view for SPCT specimen machining.

Table 1. The chemical composition of solder joints.

Solder Joint	Chemical Composition (wt. %)											
	Sn	Pb	Sb	Cu	Bi	Zn	Fe	Al	As	Cd	Ag	Ni
Sn-37Pb	62–64	Bal-	0.10	0.03	0.03	0.002	0.02	0.002	0.03	0.002	0.002	0.01
Sn-4Ag-0.5Cu	Bal-	0.05	0.10	0.50	0.03	0.002	0.02	0.002	0.03	0.002	4.00	0.01

Table 2. The experimental condition and material melting point temperature.

Material		SPCT Method			
Solder Joint	Melting Point Temp (°C)	Reflow Condition		SPCT Temp (°C)	Aging Time (h) and Temp (°C)
		Reflow Temp (°C)	Reflow Time (s)		
Sn-37Pb	183	200 (± 5)	30	30	0, 200, 600 (At 150 °C)
Sn-4Ag-0.5Cu	223	240 (± 5)			

2.2. Experimental Method and Conditions

Figure 2 shows the schematic diagram of the experimental setup for the SPCT method and the deformation of the solder joint during applied stress. Figure 3a shows the photo of the experimental setup for the SPCT equipment, and Figure 3b shows the enlarged view of the photo of the specimen setup device. Figure 2a shows a schematic diagram of the whole SPCT composed system. The crucial devices of SPCT are composed of the lower and upper die, furnace, control panel, linear variable differential transformer (LVDT), loading system and computer. Figure 2b shows the lower and upper die used to clamp the specimen and to apply a load on the punch. The creep displacement-time curve behavior of both solder joints exhibits the same typical stages as those found in previous research on conventional creep tests, such as primary, secondary and tertiary stages [10]. According to Figure 2b, many stresses (i.e., compressive, tensile, and shear stress) were found in Region A. However, the fractures were mostly generated by shear stress followed by both compressive and tensile stresses on the solder joints.

Figure 2. Schematic diagram of the experimental setup for the shear punch creep test and the deformation of the solder joint during applied stress.

Figure 3. Photo of the experimental setup for the shear punch creep test.

Hence, the main stress applied to the specimens is the average shear stress (τ), as shown in the following equation [11]:

$$\tau = \frac{P}{\pi dt} \quad (1)$$

where P = punch load, t = thickness of the specimen, d = average of d_1 and d_2 (d_2 is the diameter of the punch ($\varnothing 2.8$ mm), and d_1 is the diameter of the Cu substrate hold ($\varnothing 3.2$ mm)). The shear strain (ϵ) shown in Figure 2a (Region A) was obtained using the following equation [12]. Here, δ is the creep displacement that occurs during creep deformation.

$$\epsilon = \frac{\delta}{(d_1 - d_2)/2} \quad (2)$$

Figure 4 shows the preparation of a schematic view of the cross-section specimen on Region B. Meanwhile, to analyze the IMC behavior, the cross-section of the specimen was mounted to a thermosetting resin and polished with sandpaper and 0.05 micro aluminum powders. Finally, the interface of the mounted solder joint specimens was etched for 15 s using a solution (93 mL of methanol, 2 mL of HCl and 5 mL of NH_4OH). After that, The IMC behavior at the interface of the cross-section specimens (Region B) was investigated using SEM and EDS.

Figure 4. Schematic view of the cross-section specimen on region B.

3. Result and Discussion

3.1. SPCT Displacement-Time Curve

The creep data obtained from the SPCT method for different aging times is shown in Figures 5 and 6. The creep displacement-time curves of the Sn-37Pb and Sn-4Ag-0.5Cu solder joints are shown in Figures 5 and 6, respectively. The creep curve behavior exhibits the primary, secondary and tertiary stages. It can be seen that the overall creep curves are significantly different from each other according to the aging time and applied load. One can also see that the rupture time significantly decreases with the increase of aging time and applied load. This can be explained due to the brittleness of solder joints and the matrix grain coarsening of IMC that occurred on the interface between the solder joint and Cu substrates after isothermal aging, thus decreasing the bonding strength between the solder joint and Cu substrates. In the case of applied loads 49 N and 53.9 N, the displacements (δ) of the curves are higher than for other conditions (see Figure 5b,c). Because the solder joints have a higher elongation property than for other aging time conditions (200 h and 600 h), the ductile fractures were predicted to occur in this case.

Figure 5. Displacement-time curves for Sn-37Pb for different aging times (a) 0 h, (b) 200 h, and (c) 600 h.

Figure 6. Displacement-time curves for Sn-4Ag-0.5Cu for different aging time (a) 0 h, (b) 200 h, and (c) 600 h.

3.2. Power-Law Relationship

Figure 7 shows the power-law relationship between the rupture time of the solder joint (t_r) and the average shear stress (τ) for the SPCT method. Figure 6 shows that the creep rupture time of the

solder joint decreased with an increase of the punch pressure. Equation (3) is a general power law relationship [13]:

$$t_r = A\tau^{-n} \quad (3)$$

where, A = material constant, τ = average shear stress, n = stress exponent. Generally, the stress exponent (n) decreased with an increase of the aging time (see Figure 7). This can be explained by the behavior of the IMC, which changed with an increase in the aging time. This causes the strength of the solder joint to get weaker. The mean thickness of the interfacial Cu_5Zn_8 IMC layers in the specimen increased with an increase in the thermal aging time, following a parabolic law [14]. The stress exponent (n) of the Pb-free solder joints is higher than Sn-37Pb at the same temperature. Commonly, the creep life of Pb-free Sn-4Ag-0.5Cu solder joints are longer than that of Sn-37Pb solder joints.

Figure 7. Power law relationships for both aged solders joints.

The creep rupture time (t_r) of the solder joints with the aging time was calculated using the following power law relationship equation:

The power law relationship equation of Sn-37Pb and Sn-4Ag-0.5Cu solder joints:

	Sn-37Pb solder joint	Sn-4Ag-0.5Cu solder joint	
-Aging 0 h	$t_r = 4.48 \times 10^{14} \cdot \tau^{-12.75}$	$t_r = 2.75 \times 10^{24} \cdot \tau^{-18.49}$	(4)
-Aging 200 h	$t_r = 7.4 \times 10^9 \cdot \tau^{-9.2}$	$t_r = 1.78 \times 10^{20} \cdot \tau^{-17.5}$	
-Aging 600 h	$t_r = 9.94 \times 10^5 \cdot \tau^{-6.1}$	$t_r = 7.49 \times 10^{12} \cdot \tau^{-11.6}$	

3.3. Monkman-Grant Relationship

The Monkman-Grant relational equation is widely used in the creep failure modeling. It has also been used to evaluate this experiment to predict the creep rupture time [15]. This relationship is obtained from the relationship between the creep strain rate ($\dot{\epsilon}$) and the creep rupture time (t_r), as follows.

$$t_r \dot{\epsilon}^m = b \quad (5)$$

where, t_r = fracture time, m = Monkman-Grant exponent, b = material constant. The slope of the relationship plot is approximately 1 and indicates that it is valid for the SPCT method [16]. Figure 8 shows that the Monkman-Grant relationship of both solder joints in relation to the creep strain rate ($\dot{\epsilon}$) and the creep rupture time (t_r) for different aging times. The Monkman-Grant exponents (m) of the solder joints are 0.71 and 1.1, which is close to 1. This means that the SPCT method applies in the

assessment of the creep mechanical properties of both the conventional and the Pb-free solder joints. The Monkman-Grant relationship of each solder joint is as follows:

$$\begin{aligned} \text{Sn-37Pb: } t_r \dot{\epsilon}^{0.71} &= 0.18 \\ \text{Sn-4Ag-0.5Cu: } t_r \dot{\epsilon}^{1.1} &= 0.03 \end{aligned} \quad (6)$$

Figure 8. Monkman-Grant relationship of (a) Sn-37Pb and (b) Sn-4Ag-0.5Cu.

3.4. Observation of IMC Behavior

We also investigated the IMC formation during the high-temperature reflow process in order to assess and evaluate the properties of the solder joints. However, the overgrowth of IMC leads to brittleness, which causes brittle fracture and the deterioration of the bonding strength between the solder joint and substrate. As a result, the observation of the IMC formation is an essential factor for solder joints [9,17]. Figures 9 and 10 show the phase and morphology of IMC of each solder joint using SEM and EDS after aging at 150 °C for 0, 200, and 600 h respectively. The results obtained from Figures 9 and 10 show that for both solders the thickness of the IMC layer at the Cu interface gradually increases with the aging time. The IMC morphology changed significantly from a stable to irregular state and from a small round to long needle-like shape when the aging time increased. Figure 9 shows the IMC Ni_3Sn_4 , Sn-rich, and Pb-rich phases for 0 and 200 h aging. In addition, voids and other IMC phases $(\text{Cu}, \text{Ni})_6\text{Sn}_5$ were observed at 600 h. This changed behavior of the IMC at the Cu interface shows that the initial ductility is gradually evolving to brittleness with increasing aging time. Figure 10 shows that the IMC phase $(\text{Cu}, \text{Ni})_6\text{Sn}_5$, Ni_3Sn_4 was observed from an aging time of 200 h, and that the voids appeared for an aging time of 600 h. Overall, the IMC interface of the Sn-4Ag-0.5Cu solder joints was found to be more stable than that of the Sn-37Pb solder.

Figure 9. SEM images of Sn-37Pb after aging time (a) 0 h, (b) 200 h, and (c) 600 h.

Figure 10. SEM images of Sn-4Ag-0.5Cu after aging time (a) 0 h, (b) 200 h, (c) and 600 h.

4. Conclusions

In this research, we evaluated the creep mechanical properties of Sn-37Pb and Sn-4Ag-0.5Cu solder joints for different aging times (0, 200, 600 h) by investigating IMC behavior and using the SPCT method. The conclusions of this study are summarized as follows.

1. Based on the results of the SEM micro images, the thickness of the IMC increased, and the shape of the IMC morphologies changed from round to needle-like with an increase of the aging time. After isothermal aging (200 and 600 h), the IMC $(\text{Cu, Ni})_6\text{Sn}_5$ phases and the micro voids were

found on the solder joint/Cu substrate interface. This means that the brittleness of the solder increased, thus causing the deterioration of mechanical properties.

2. The power-law relationship indicated that the stress exponent (n) and creep properties of solder joints decreased with the increase of aging time, and that the values (n) of Sn-4Ag-0.5Cu solder joints are higher than those of Sn-37Pb solder joints. It can thus be confirmed that the creep properties of Sn-4Ag-0.5Cu solder joints are better than those of conventional Sn-37Pb solder joints.
3. The Monkman-Grant relationship shows that the exponent value (m) solder joints are close to 1. It can thus be confirmed that the SPCT method is a reliable method, which can be used to evaluate the creep properties of solder joints.
4. Based on our results, the obtained mechanical properties of Pb-free Sn-4Ag-0.5Cu solder joints are greater than those of Sn-37Pb solder joints for all isothermal aging time conditions (0, 200, 600 h). In addition, the melting point temperature of Sn-4Ag-0.5Cu solder joints is similar to that of conventional Sn-37Pb solder joints. Therefore, it is possible to use Pb-free Sn-4Ag-0.5Cu solder joints in electrical applications, rather than Sn-37Pb solder joints.

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