Deformation Fracture Characteristics of Microelectronic Sn-3.0Ag-0.5Cu-xNi Solders

Fei-Yi Hung1,*1, Truan-Sheng Lui2,*2, Li-Hui Chen2 and Cheng-Wei Chan2

1 Institute of Nanotechnology and Microsystems Engineering, Center for Micro/Nano Science and Technology, National Cheng Kung University, Tainan, Taiwan 701, R. O. China
2 Department of Materials Science and Engineering, National Cheng Kung University, Tainan, Taiwan 701, R. O. China

The effect of Ni content on the microstructure, as well as the tensile and vibration fracture mechanisms of a potential lead-free solder, Sn-3.0Ag-0.5Cu-xNi (0.02, 0.07, 0.1, 0.2 and 0.3 mass%), are examined in this study. The results show that both Sn-Ni-Cu and Sn-Cu-Ni-Ag intermetallic compounds (IMC) increased with increasing the Ni content. The IMCs mostly formed in the eutectic zones and a few in proeutectic Sn-rich phases. Notably, the Ni content of the bar-like Sn-Ni-Cu compounds was higher than that of the particle-like Sn-Cu-Ni-Ag compounds. In addition, the tensile deformed resistance of Sn-3.0Ag-0.5Cu-xNi solders decreased when the Ni content was increased. Adding Ni obtained finer structures, however the hard massive Sn-Ni-Cu in the eutectic zone deteriorated the tensile deformation resistance. For the lower Ni content specimens, the 0.07Ni specimen not only possessed finer structures but a large number of compounds which congregated were able to increase the crack tortuosity, which in turn increased the crack propagation resistance and the vibration life.

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1. Introduction

The Sn-Ag-Cu solder alloy has recently been considered as a candidate for a lead-free solder material because of ease of manufacture, mechanical properties and reliability.1–3) Under drop tests and pull tests, many reports have shown that the fracture resistance of Sn based solders can be enhanced by a very small addition of Ni, RE etc.4–8) Also, related studies4,5,9–13) have revealed that Ni addition (>3000 ppm) has a significant influence on the microstructure, creep, shear strength, fatigue properties and the characteristics of intermetallic compounds (IMC) in the interface. However, the studies emphasized the interfacial effect between solders and substrate; the relationship between the bulk solder and microsegregation has not been examined. Our previous studies14–17) have found the vibration fracture crack propagation of several Sn-based solders (Sn-Ag, Sn-Zn systems) was closely related to the second phase of the matrix. This is why fracture cracks were found either in the IMC layer or in the bulk solder after reliability tests. According to the above, when the IMC layer is undamaged, the crack will propagate into the bulk solders during drop tests and pull tests. So, understanding the deformation resistance of the bulk solders is an important subject. Many studies have discussed the characteristics of the (CuNi)xSn1 IMCs layer in Ni/Au pad and OSP pad,4,5,9–13) however, the addition effect of Ni (≤ 3000 ppm) in the bulk microstructure and the relevant fracture properties have still not been examined. Owing to the effect of Ni addition being closely related to the solder microstructural characteristics and the solder joint reliability, this study uses Sn-3.0Ag-0.5Cu-xNi solders not only to analyze the characteristics of (CuNi)xSn1 compounds, but also investigate the deformation fracture mechanism under tensile testing and vibration testing.

2. Experimental Procedure

Master alloys of Sn-3.0Ag-0.5Cu-xNi solders (x = 0.02, 0.07, 0.1, 0.2 and 0.3 mass%) were prepared by melting pure tin, pure silver, pure copper and pure nickel in a high-frequency induction furnace. The alloy ingots were then remelted and cast into a Y-shaped graphite mold. Hereafter, the specimens will be designated according to Ni content as 0.02Ni, 0.07Ni, 0.1Ni, 0.2Ni and 0.3Ni. In order to collect tensile test data, rectangular specimens (gauge length section: 20 mm × 4.3 mm × 2.1 mm) were also prepared, and the tensile test results of each sample had a constant strain rate of 7.5 × 10−4 s−1. For vibration testing, a simple cantilever beam vibration system was used for the vibration experiments. The test specimens, (Fig. 1), which were rectangular with dimensions 100 mm × 10 mm × 4 mm, were mounted and fixed on end to the vibration shaker. Two circular-
notches near the clamp were made for observing microstructural evolution in the vicinity of the notch front. For vibration testing, a diagram of observation direction is shown in Fig. 2. On the vibration frequency vs. the deflection amplitude curve, the maximum deflection amplitude always occurs at resonant frequency. The resonant frequency is taken as the frequency leading to the largest deflection and is determined by varying the vibration frequency continuously.\textsuperscript{18,19) With an aim to understanding the difference in chemical composition of the matrices, the structures of the affected regions were examined using EPMA/EDS/WDS. Each analysis datum was the average of at least 3~7 test results.

3. Results and Discussion

3.1 Microstructural characteristics

The microstructures of the solders are shown in Fig. 3. The average grain size of the $\beta$-Sn had an obvious tendency to decrease as the Ni content was increased. Except in the proeutectic Sn-rich phases, bar-like intermetallic compounds (IMC) and particle-like IMCs are observed in the Sn-Ag eutectic zones. In order to understand the feature and the composition of the IMCs, the BEI and EPMA of the Sn-3.0Ag-0.5Cu-0.3Ni specimen were observed. Figure 4 shows the microstructures of the particle-like IMCs (A) and the bar-like IMCs (B). Table 1 shows the EDS of the IMCs and the matrix. The results indicate that the particle-like IMC (A) was a compound of Sn-Cu-Ni-Ag and the bar-like IMC (B) was a compound of Sn-Ni-Cu. According to relevant reports,\textsuperscript{3–5) it can be inferred that the intermetallic compounds are the (CuNi)$_x$Sn$_y$ IMCs (Table 1). To obtain the distribution of elements, EPMA analysis of the structure was performed (Fig. 5). The results reveal that the Ni content in the bar-like Sn-Ni-Cu phase was higher than that in the particle-like Sn-Cu-Ni-Ag phases. The (CuNi)$_x$Sn$_y$ IMCs existed mostly in the eutectic zones with a few in the proeutectic Sn-rich phases.

![Fig. 2 Observation direction of the vibration fracture.](image)

![Fig. 3 Microstructures of Sn-3.0Ag-0.5Cu-xNi solders: (a) 0 N, (b) 0.02 N, (c) 0.2 N and (d) 0.3 N.](image)
Regardless of the Ni content, a very small amount of Ni was solid solution in the β-Sn phases. Notably, a large amount of Cu existed in the particle-like IMCs (A) and a little Cu in the bar-like IMCs (B). According to the above findings, it can be confirmed that the Ni attracted Cu to form IMCs easily. If the Ni microsegregation raised the concentration of Ni in the interface of the pad during reflow, it resulted in hard massive Sn-Ni-Cu intermetallic compounds forming and caused fracturing in the IMC layer or pad peeling to occur easily. In order to understand the effect of Ni microsegre-
gation on the deformation resistance, this study controlled the Ni content from 0.02 mass% to 0.3 mass%.

3.2 Tensile properties

Figure 6 shows a comparison of the tensile results of the specimens with various Ni content at room temperature (Ultimate Tensile Strength (UTS), Yield Strength (YS), Uniform Elongation (UE) and (d) Total Elongation (TE)). The figures reveal that the tensile strength of the Sn-3.0Ag-0.5Cu-xNi specimen decreased as the Ni content was increased (Fig. 6(a) and Fig. 6(b)), while some specimens had an inverse tendency towards elongation (Fig. 6(c) and Fig. 6(d)). For both the 0.02N specimen and the 0.07N specimen, the addition of Ni had a contribution to ductility. The tensile fracturing of some specimens was observed to understand the deformation behavior. Figure 7 shows a comparison of the tensile fracture subsurface of specimens with varying Ni content, and the fracture characteristics show that the β-Sn phase possessed a tensile plastic deformation flow after tensile testing, thus the β-Sn phase was the main deformation mechanism. According to relevant reports\(^{8-16}\) and comparing Fig. 3 with Fig. 6, the deformation resistance of the present solders did not show any improvement and the \((\text{CuNi})_x\text{Sn}_y\) IMCs had no contribution to deformation resistance. In Fig. 7, the precipitated IMCs acted as a magnet for fracture crack propagation and the cracks propagated mostly in the eutectic zones. The precipitated phase near cracks was examined using line scanning-WDS, shown in Fig. 8. The results show that the precipitated phase was the Sn-Ni-Cu IMC.

Increasing the content of Ni to more than 0.07 mass% made the structures finer, however, the hard massive Sn-Ni-Cu deteriorated the tensile deformation resistance. In addition, the Ni attracted Cu to form IMCs, resulting in the distinctive strength of \((\text{CuNi})_x\text{Sn}_y\) phase (non-Ni specimen) decreasing,\(^{5,17}\) thus there was no obvious improvement in the deformation resistance. The ductility data of the low Ni specimens (0.02Ni and 0.07Ni) show an obvious improvement (Fig. 6). According to observations, the size of dimples on the tensile fracture surface had an obvious tendency to increase as the Ni content was increased (Fig. 9). The most likely explanation for this is a combination of the refinement of β-Sn phase and no hard massive \((\text{CuNi})_x\text{Sn}_y\) IMCs forming.

3.3 Vibration behaviors

The low Ni specimens (0.02Ni and 0.07Ni) possessing better ductility were underwent vibration fracture testing under a fixed vibration force of 1.8 G (the resonant frequencies of the specimens have no change with variation of the composition, and the values are 49 ± 1 Hz). Figure 10 shows the D-N curves of the specimens and the initial deflection amplitude of the 0.07Ni specimen was lowest (damping capacity was highest), followed by 0.02Ni and SAC305. In addition, the vibration number of cycles (vibration life) increased with increasing the Ni content. Notably, this study avoided the effect of damping capacity by controlling the vibration force and measuring the D-N curves of the specimens under constant initial deflection conditions. Under both constant force and initial-deflection
conditions, the 0.07Ni specimen showed the greatest vibration life. After vibration tests, the surface crack propagations and the fracture characteristics were observed as illustrated in Fig. 2.

Figure 11 and Figure 12 showed the vibration fractures of the specimens under a fixed vibration force of 1.8 G. The results reveal that the vibration crack propagation went through the eutectic phases and β-Sn phases. The gathered IMCs were able to increase the crack tortuosity (Fig. 11), which in turn increased the crack propagation resistance. In addition, a large number of splinterly characteristics were observed on the vibration fractures of the SAC305 (0Ni) specimen (see the marks, Fig. 12(a)). This shows that the brittleness effect was obviously active in the 0Ni specimen and the splinterly characteristics had an obvious tendency to decrease as the Ni content was increased.

3.4 Vibration characteristics vs. tensile deformation resistance

From Fig. 6 and Fig. 10, we see that the vibration life of the specimens increased with increasing the Ni content (< 700 ppm). But the tensile strength of the specimens had an inverse relationship with the vibration life. In another words, the vibration life of the Sn-3.0Ag-0.5Cu-xNi solders cannot
be assessed from the tensile mechanical properties. For many engineering materials, the vibration fracture resistance increases with increasing the tensile deformation resistance and Young’s modulus of the materials.\textsuperscript{20,21} However, this is not true for all materials.\textsuperscript{22–24} The most likely explanation for this is that vibration testing not only involves fatigue, but also involves a vibration force with frequency of $\frac{1}{C_2}$ Hz, unlike tensile testing which has a strain rate of $\frac{1}{C_0}$. Although the present solders had different structures, damping capacity and mechanical properties, the 0.07Ni specimen not only possessed finer structures but also the congregated compounds (no massive (CuNi)$_y$Sn$_x$ IMCs form) were able to increase the crack tortuosity, which in turn increased the crack propagation resistance and lengthened the vibration life. Due to some of the (CuNi)$_y$Sn$_x$ IMCs growing unusually in the higher Ni specimens, the hard massive IMC phase was not advantageous to tensile deformation resistance. The lower Ni content specimens had a higher ductility, and the gathered IMCs were able to increase the crack propagation resistance and lengthened the vibration life. Under both constant force and initial-deflection conditions, the 0.07Ni specimen showed the longest vibration life.

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