Inhomogeneous deformation and microstructure evolution of Sn–Ag-based solder interconnects during thermal cycling and shear testing

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ABSTRACT

Orientation imaging microscopy was adopted to characterize the microstructural changes in Sn–Ag-based solder interconnects during thermal cycling and shear testing. The deformation and microstructure evolution of Sn–Ag-based solder interconnects are inhomogeneous, depending on the orientations of β-Sn grains in the as-solidified microstructure. Recovery or recrystallization can take place even under pure shear stress at room temperature, and it tends to occur at high-angle grain boundaries in multi-grained solder interconnects, while it localizes in near-interface region in solder interconnects with only one grain inside. During thermal cycling, the hardness of recrystallized microstructure decreased significantly due to the segregation of Ag3Sn IMC particles towards the newly-formed recrystallized boundaries, increasing the ease of localized deformation in this weakened microstructure. As a consequence, cracks were propagated intergranularly in the recrystallized microstructure.

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1. Introduction

As electronic devices evolve continuously towards miniaturization, high-density, and multi-function, solder interconnects are subjected to more severe thermo-mechanical and electrical loading conditions [1]. One of the most important reasons for failures of solder interconnects is CTE (coefficient of thermal expansion) mismatch between different packaging materials, such as chip and PCB (printed circuit board) [2]. An electronic device may contain hundreds of solder interconnects, and if any one of them fails, it will lead to the malfunction of the whole device. Therefore, the performance of solder interconnects is of great importance for the reliability and service life of electronic products.

Nowadays, SnPb interconnect materials have been replaced by lead-free solder alloys with Sn concentration generally over 90 wt.%. The performance of Sn-rich solder interconnects is determined jointly by the interfacial IMC (intermetallic compound) and the microstructures of the β-Sn solder bulk embedded with IMC particles, such as Ag3Sn or Cu6Sn5 [2–4]. Extensive research has been done to study the interfacial IMCs because they can induce brittle failure when accidental mechanical shock takes place, especially for those portable electronic devices [3,5]. However, cracks are frequently observed in the solder bulk during thermal cycling, which is a typical service environment for most electronic devices due to power switches and temperature change of external environment [6–10]. Moreover, the initiation and propagation of cracks are frequently accompanied with recrystallization until the final failure of lead-free solder interconnects, therefore, it has attracted considerable interest in recent years [6–16]. Localized recrystallization could also be observed under different mechanical stresses, such as four-point bending, tensile and shear tests [17]. However, recrystallization does not occur in SnAgCu solder slabs when compressed up to 50% of the original height followed by annealing immediately after the deformation [18]. The findings and observations are inconsistent regarding recrystallization in lead-free solder interconnects, and it may be related to the differences of interconnects in size, metallization, geometry, solidification condition, etc.

Recrystallization significantly changes the shape, size, and orientation of β-Sn grains, which play a significant role in dictating the thermomechanical responses of Sn-rich solder interconnects because the body-centered tetragonal β-Sn shows strong anisotropic thermal and mechanical properties in different crystallographic directions [19,20]. Furthermore, microstructure and mechanical properties of lead-free solder interconnects are not constant and they can evolve dramatically along with recrystallization [11,12]. Therefore, a lead-free solder interconnect cannot be viewed as a homogeneous material as usually done in FEM (finite element modeling). Therefore, more information, such as grain orientation and microstructural changes, needs to be added to the model to accurately describe the deformation behavior of lead-free solder interconnects.
Although much important work has been carried out on recrystallization behavior of lead-free solder interconnects, the details of underlying mechanism of recrystallization are still not fully understood. Recrystallization behavior in practical lead-free solder interconnects requires further study in terms of microstructure and mechanical properties to obtain a better understanding of the failure mode and reliability of lead-free solder interconnects. In this study, we examined the evolution of microstructures and the accompanying change of mechanical properties in Sn–Ag-based solder interconnects during thermal cycling and shear testing.

2. Experimental procedures

As shown in Fig. 1, the components in this study were chip scale packaged (CSP) ball grid array (BGA) packages with 228 peripheral array Sn–3.0Ag–0.5Cu solder bumps with pitch of 0.5 mm. The dimensions of the package and chip were 12 mm × 12 mm and 10 mm × 10 mm, respectively. The solder bump size was 0.3 mm in diameter. Surface finish of the package side substrate was electrolytic NiAu. The BGA packages were soldered onto the non-solder in diameter. Surface finish of the package side substrate was electrolytic NiAu. The BGA packages were soldered onto the non-solder

1. Orientation evolution of Sn–3.0Ag–0.5Cu solder interconnects during thermal cycling

As shown in Fig. 2, an as-solidified microstructure of Sn–3.0Ag–0.5Cu solder interconnect is typically composed of a few grain or only one grain though the orientations after solidification varied from interconnect to interconnect due to the complicated solidification conditions. The small number of grains in lead-free solder interconnects is caused by the difficulty in β-Sn nucleation and the associated large undercooling during solidification [21,22]. Several low- or high-angle grain boundaries were observed in the grain boundary map.

The definition of recovery and recrystallization is provided by a review [23], i.e. recovery includes all the processes of releasing stored energy without the movement of a high-angle grain boundary, while recrystallization involves the formation and migration of high-angle grain boundaries driven by the stored energy of deformation. Based on this definition of recovery and recrystallization, as shown in Fig. 3a, we define three different microstructures in the thermally cycled solder interconnect, i.e. recrystallized microstructure separated with high-angle boundaries, recovered microstructure with many small subgrains inside (characterized by a large density of low-angle boundaries), and nonrecrystallized microstructure without significant change after solidification (with few or no low-angle boundaries inside).

After 2815 thermal cycles, it can be seen from the EBSD orientation map, misorientation angle distribution, and grain boundary map in Fig. 3a, c, and d that the orientations of the recrystallized grains appear to be randomly distributed. Many small subgrains, which are typical products of recovery, were formed around the recrystallized grains, as shown in Fig. 3a. These subgrains show similar orientations with slight misorientations with each other, and can not be clearly distinguished in EBSD orientation map. They are not the same with the cell wall structures, which should be much smaller and can not be clearly characterized under SEM. However, the small subgrains (with misorientations larger than ~0.5° from the parent grains or neighboring subgrains) can be revealed by the grain boundary map of OIM. Moreover, the stored energy distribution can also be roughly estimated with this EBSD data [24], however, this is beyond the scope of this study.

Fig. 3b shows the misorientation along the path from the non-recrystallized microstructure to the recrystallized microstructure (indicated by the black arrow in Fig. 3a) the misorientation in the nonrecrystallized region is quite low (generally below 10°); however, it increased dramatically after reaching the recrystallized region. The cracks mainly follow the newly formed grain boundaries produced by recrystallization, and sometimes, cracks were
also observed at the boundaries of the recrystallized and non-recrystallized regions, as shown at the upper right corner of the solder interconnect (indicated by the white arrow in Fig. 3a).

3.2. Microstructural evolution during thermal cycling

The microstructure of an as-reflowed Sn–3.0Ag–0.5Cu solder interconnect is composed of Sn-rich phase ($\beta$-Sn), Cu$_6$Sn$_5$, and Ag$_3$Sn IMCs. The $\beta$-Sn cells are surrounded mainly by Cu$_6$Sn$_5$ and Ag$_3$Sn IMCs, which are the strengthening phases of SnAgCu solder alloy. After thermal cycling, as shown in Fig. 4a, grain boundaries in localized recrystallized regions were revealed clearly by the etchant. The interiors of the recrystallized grains appear to be smooth with only a few Ag$_3$Sn IMC particles inside, while no significant change was observed in the non-recrystallized region. The element mapping result of the solder interconnect is shown in Fig. 4b. It is
interesting to note that Ag element segregated in the localized recrystallized region, i.e. the finely dispersed Ag₃Sn IMC particles coalesce into larger ones with decreasing number of particles (also can be clearly seen in Fig. 4a). However, no obvious segregation of Ag element is identified in the nonrecrystallized microstructure. As shown in Fig. 4a, the coarsening extent of Ag₃Sn particles in the recovered region (with many small subgrains inside) falls between that in the recrystallized and nonrecrystallized regions.

A closer examination of the microstructure shows that most of the Ag₃Sn IMC particles have a tendency to segregate along the grain boundaries of the localized recrystallized microstructure. The grain boundaries of the recrystallized microstructure offered defects, such as vacancies and dislocations, to accommodate the Ag atoms to decrease the Gibbs free energy. It looks like the original finely dispersed Ag₃Sn IMC particles were “pushed” to the grain boundaries to agglomerate into larger ones. However, as shown in Fig. 4b, no Cu element segregation was found at the grain boundaries in both the recrystallized and nonrecrystallized microstructures. However, it has been reported that Cu diffusion rate in Sn is 15 times faster than that of Ag in Sn at 180 °C [25]. The likely reason is that the concentration of Cu in SnAgCu solder alloy is relatively low (0.5 wt.%) compared with that of Ag (3.0 wt.%), and thus the segregation of Cu is negligible [26].

3.3. Hardness test

We have tried nanoindentation method previously to characterize the mechanical properties of the recrystallized and nonrecrystallized microstructures. However, the data show a relatively large discrepancy due to the limitation of the indentation area, i.e. a lower value was obtained when the indentation was performed on the soft β-Sn matrix, whereas a higher value was achieved when the
indenter contacted the hard IMCs. Therefore, to get the overall performance of the recrystallized and nonrecrystallized microstructures as a whole, we adopted the Vickers microhardness test to characterize the mechanical properties of these microstructures and corresponding degradation during thermal cycling. The microstructure after indentation testing is shown in Fig. 5. The area of indentation in the recrystallized microstructure was larger than that of the nonrecrystallized counterpart, indicating microstructural degradation takes place in the recrystallized microstructure due to the weakened dispersion strengthening effect of the coarsened Ag3Sn IMC particles. As shown in Fig. 6, the hardness values of the microstructures in the solder interconnects dropped down significantly after 2815 thermal cycles; compared with the microstructure of as-reflowed solder interconnects, the microhardness of the recrystallized microstructure was decreased by about 39%, and that of the nonrecrystallized microstructure was decreased by about 20%.

3.4. Orientation evolution of Sn–3.0Ag solder interconnects during shear testing

Many small subgrains characterized by a high density of low-angle boundaries (Fig. 7a and b) or recrystallized grains separated by high-angle boundaries (Fig. 7c) were formed at the near-interface region in the Sn–3.0Ag solder interconnects after shear testing. For the 100 solder interconnects tested in this experiment, nineteen of them show recrystallization, while the rest show recovery with subgrains near the interface. That is to say, localized recrystallization can take place in solder interconnects even under pure shear stress. This can be attributed to the high homologous temperature of Sn–3.0Ag solder at room temperature ($T_{\text{room}}/T_{\text{m}} > 0.6$, in K) because recrystallization is a thermally activated process.

It should be noted that the two solder interconnects in Fig. 7a and b show different mechanical responses; many low-angle boundaries were formed near the original grain boundary in the solder interconnect in Fig. 7b, indicating deformation behavior has a close relationship with the orientations of the as-solidified solder interconnects. Recrystallization tends to occur in β-Sn phases, as shown in Fig. 8, while no obvious recrystallization was observed in the wall structures surrounding the β-Sn cells, which are composed of fine Ag3Sn and Cu6Sn5 IMC particles embedded in the β-Sn matrix. This indicates that the fine closely-spaced particles can inhibit the initiation of recrystallization by limiting the movement of dislocations.

4. Discussion and analysis

4.1. Recrystallization facilitated by subgrain formation, rotation and growth

The subgrains (or cell structures), which are typical products of recovery, may act as the nucleation sites for recrystallization. The subgrains are relatively small compared with the size of the recrystallized grains in thermal cycling and shear testing, as shown in Fig. 3 and Fig. 7. It is reasonably to infer that (sub)grain growth is accompanied with the rotation from subgrains to recrystallized grains. After subgrain formation, subgrains tend to grow up to reduce the area of low-angle boundaries to lower the stored energy, making subgrain coalesce with their neighbors. The possibility of the rotation of a subgrain with respect to its neighbors during recrystallization has been confirmed thermodynamically and kinetically, respectively [27]. As a consequence, the boundaries originally present between the neighboring subgrains disappear, and the misorientation of the rotated (sub)grains with their neighbors is enlarged after (sub)grain rotation and coalescence. In this way, small subgrains with low-angle boundaries can have an energy advantage (usually a large size) and high local misorientations with the neighboring (sub)grains, which are necessary conditions for them to have large mobility to finally evolve into the large recrystallized grains [23]. The new recrystallized grains grow from the recovered subgrains or cell structures rather than nucleate by the atom by atom construction. That is to say, the subgrains and recrystallized grains are the consequence of rotation from the parent grain by different degrees along different axes. The final recrystallized grains are randomly distributed, and no exact crystallographic relationship between the recrystallized and nonrecrystallized grains was found.

Grain boundary sliding can be seen between the rotated grains under the cyclic thermomechanical stress (Fig. 9a). The observation in this study is different with the incremental recrystallization reported by Telang et al. [15]. This may relate to the geometry of the lap joints, which are different from the practical solder interconnects from the aspects of interaction between the complicated geometry and corresponding complex loading conditions, such as, cyclic shear, torsion and bending. Moreover, the size, metallization, and solidification condition of solder interconnects also play
an important role in determining the thermomechanical responses during thermal cycling [28]. It should be noted that the subgrain boundaries produced after recovery are different from the low-angle boundaries in the as-solidified microstructure in that these subgrain boundaries have a much larger mobility to coalesce with the neighboring grains.

Fig. 7. Subgrain formation at (a) near-interface region; (b) original high-angle grain boundary, and (c) recrystallized grain formation at near-interface region in Sn–3.0Ag solder interconnects after shear testing.

Fig. 8. Near-interface microstructure of Sn–3.0Ag solder interconnect after shear testing.
From the results discussed above, it can be seen that the subgrains produced by recovery facilitates the formation of recrystallized grains. However, recovery and recrystallization are generally assumed to be two competitive processes; recovery is easy to take place in high stacking fault energy metals, such as tin, by the cross-slip and climb of dislocations, especially at high temperature [29]. Therefore, recovery process releases some stored energy preceding recrystallization. However, in this study, the residual stored energy remains high enough to induce following recrystallization in the deformed solder alloy after subgrain formation. The transition process from subgrains to recrystallized grains is continuous, and recrystallization cannot be initiated without subgrain formation in the preceding recovery.

4.2. Degradation and failure modes of Sn–Ag-based solder interconnects during thermal cycling

The segregation process of Ag element is accelerated by plastic deformation. It has been reported that the segregation can be enhanced by plastic deformation by two possible means, i.e. carrying of the solute atoms to the grain boundaries by dislocation movement, and strain enhanced diffusion of solute atoms along the dislocation pipes [30–32]. For the mechanism of dislocation carrying solute atoms, solute atmospheres around mobile dislocations form by the elastic interaction of the strain fields between the dislocations and the solute atoms [33]. When deformation occurs, the dislocation movements are blocked by the grain boundaries, and then the solute atoms dragged by these dislocations are also deposited and accumulated at the boundaries [31]. In addition, the segregation of the solute atoms could also be accelerated by the increased diffusion short circuits, such as [sub]grain boundaries [32]. In deformed area, the diffusion short circuits increased dramatically with dislocation density, leading to the accelerated diffusion of the solute compared to an undeformed or less deformed counterpart. Combined with the previous discussion about the formation mechanism of localized recrystallization, it is most likely that both mechanisms are involved in the segregation process of Ag element. Sometimes, the trace of the coalescence of Ag₃Sn IMC particles can be identified, as shown in Fig. 4a. As a consequence of Ag element segregation, the dispersion strengthening effect of Ag₃Sn IMC particles is significantly weakened, leading to the decreased hardness in the recrystallized microstructure. In fact, hardness is an approximate measure of plastic deformation resistance, and it can be used to give a rough estimation of lead-free solder interconnect reliability because the final failure of solder interconnects is mainly brought by the cyclic plastic deformation under thermomechanical fatigue loading.

It has been reported that random boundaries could be considered as potential cracks due to the high energy, and grain boundary sliding is more likely to occur at these boundaries [34]. The differences in crystallographic orientation and mechanical properties between the recrystallized grains may induce stress peaks at the grain boundaries that may exceed the yield strength. Once the cracks are initiated in the recrystallized region, they can rarely be blocked by the sparsely distributed coarsened Ag₃Sn IMC particles during the propagation process, leading to the eventual intergranular fracture. Furthermore, the degraded properties make the deformation localize in the recrystallized region, and the deformation behavior will change fundamentally once recrystallization occurs in solder interconnects. As shown in Fig. 9, wedge cracks are frequently observed at the fracture surfaces of solder interconnects, indicating grain boundary sliding becomes the most important deformation behavior at high homologous temperature [6,12]. At the same time, diffusion transport is also associated with the grain boundaries, i.e. diffusion creep, leading to the reduced cohesion of grain boundaries [35]. The coarsened Ag₃Sn IMC particles were revealed at the cracked grain boundaries on the fractography, which correlates well with the segregation behavior observed in the cross-sections of the specimens. The cracks mainly initiate and propagate intergranularly in the recrystallized microstructure due to the more boundaries and microstructural degradation.
4.3. Inhomogeneous deformation of lead-free solder interconnects

The deformation behavior of solder interconnects in thermal cycling and shear testing is not homogeneous depending on the shape, size, and crystallographic orientations of $\beta$-Sn grains. Solder interconnects show different thermomechanical or mechanical responses due to the varied orientations in the as-solidified microstructure even they are subjected to the exact same loading. It is interesting to note that only several solder interconnects survive 10,000 thermal cycles without obvious cracking in the cross-sections, and they all have multi-grained structure with grain boundaries almost perpendicular to the interfaces. No significant recrystallization was observed in these interconnects, and they seems to be more resistant to the cyclic deformation due to the geometrical relationship between the grain boundaries and shear stress, however, more research needs to be done to study the effect of grain orientation on the solder interconnect reliability. Similar to what has been found in shear testing (Fig. 7), during thermal cycling, recrystallization tends to take place at the high-angle grain boundaries in the solder bulk of multi-grained solder interconnects, while it was localized at the near-interface region in single-grained solder interconnects (Fig. 10). Subsequently, the degradation of recrystallized microstructure makes further deformation easier to take place in this local area with the rest part of the solder interconnect showing little influence. The inhomogeneous responses of solder interconnects with varied orientations under corresponding complicated superimposed stress could be one of the reasons for the large differences in service life of the same bulk electronic devices.

5. Conclusions

The results show that the recrystallized grains evolve from subgrains by rotation and coalescence, and subgrains formation seems to be a necessary step before recrystallized grains can take into shape. The subgrains and recrystallized grains are formed by rotation from the parent grain by different degrees along different axes. Recrystallization can take place in Sn–Ag-based solder interconnects at a very quick speed under pure mechanical stress even at room temperature. The grain boundaries in multi-grained solder interconnects are preferable sites for recrystallization, while recrystallization took place at the near-package-interface region in solder interconnects with only one grain inside. The inhomogeneous deformation of Sn–Ag-based solder interconnects strongly depends on the size, shape and crystallographic orientation of the $\beta$-Sn grains. The microhardness of recrystallized microstructure is significantly reduced with the segregation of Ag$_3$Sn IMC particles towards the newly-formed boundaries. Compared with the microstructure of as-reflowed solder interconnects, the microhardness values of the recrystallized and nonrecrystallized microstructures were decreased by about 39% and 20%, respectively. The reduced microhardness makes the deformation and subsequent intergranular cracking concentrate in the recrystallized
microstructure, facilitating the inhomogeneous deformation of Sn-Ag-based solder interconnects.

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