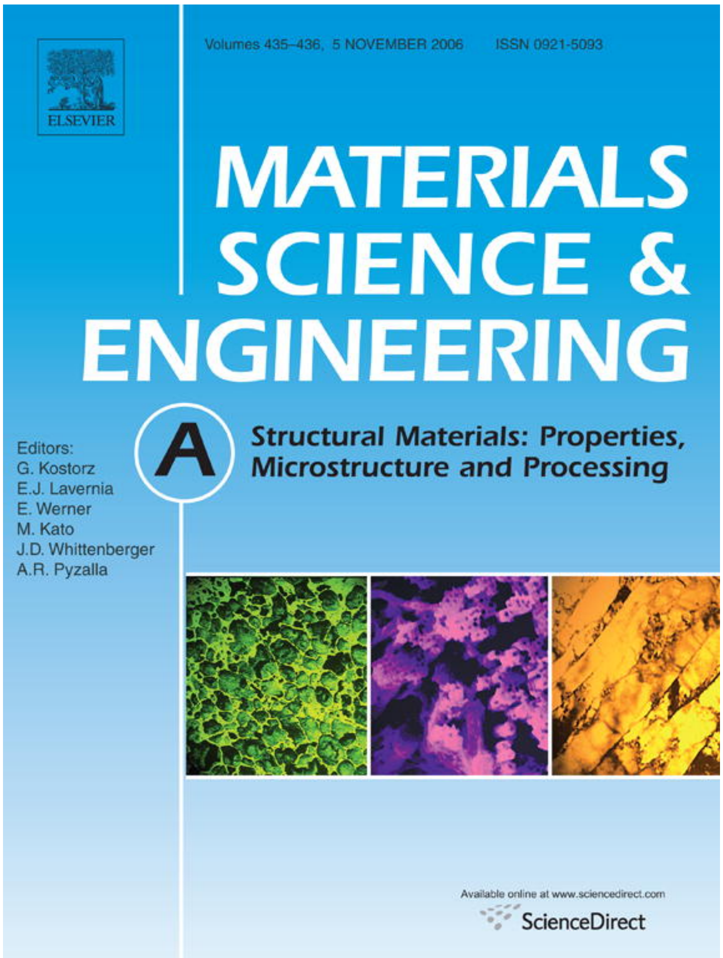
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Fatigue damage mechanisms of copper single crystal/Sn–Ag–Cu interfaces

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Abstract

Sn–Ag–Cu solder/copper single crystal joints were prepared to reveal the interfacial fatigue damage mechanisms. Scallop-type Cu_6Sn_5 and planar-type Cu_6Sn_5/Cu_3Sn intermetallic compounds (IMCs) interfaces were formed between SnAgCu solder and copper single crystal after reflowing at 240 °C and subsequent aging at 170 °C, respectively. Under cyclic loading, persistent slip bands (PSBs) were activated in copper single crystal and continuously impinged the interfaces of the SnAgCu solder/copper single crystal joint. Two types of fatigue cracking modes, i.e. interfacial cracking between solder and IMC and fracture within IMC, were observed. Based on the experimental observations above, the corresponding interfacial fatigue cracking mechanisms were discussed.

Keywords: Sn–Ag–Cu solder; Copper single crystal; Intermetallic compounds (IMCs); Interface; Cyclic deformation; Persistent slip bands (PSBs); Fatigue cracking

1. Introduction

The primary function of solder interconnection used in electronic assembles is not only to provide the electrical connection, but also to ensure the mechanical bonding [1–4]. In recent years, the advance in high density electronic packaging technology has increased the demand on mechanical reliability of this interconnection [5]. Usually, this interconnection is made in a metallurgical way by the formation and growth of intermetallic compounds (IMCs) between the copper pad and solder, and its reliability depends strongly on the morphology and kinetics of IMCs formation [6–17]. Therefore, one of the major concerns to the integrity of solder interconnection is the damage mechanism of IMCs interfaces in practical service.

Sn–Ag–Cu solder alloy is one of the most promising lead-free solder candidates because of its relatively low melting temperature, good solderability and excellent mechanical properties [1–3,18,19]. Therefore, the reliability of its interconnection is an important issue for its wide use in electronic industry. In recent years, the reliability evaluation of soldering interconnection has attracted great interest and been extensively studied by means of tension [9–11], shear [12–14], isothermal or thermo-mechanical fatigue tests [15–17]. However, most of these related studies are only based on the experiments performed to evaluate the reliability of the solder joints in macro-scale. Although the IMC interface is crucial for the mechanical integrity of the joints, very few studies so far have focused on the interfacial damage behavior in micro-scale. Thus, there is a need to better understand the interfacial damage mechanisms in micro-scale for the interconnection reliability.

It is well known that persistent slip bands (PSBs) is a typical feature of plastic strain localization in copper single crystals subjected to cyclic deformation [20–22]. PSB can be regarded as carrier and channel transporting the residual dislocations and can reach the free surface during cyclic deformation [21–24]. In the present study, we designed the IMC interface between the Sn–Ag–Cu solder and copper single crystal to investigate the interfacial damage mechanisms under the interaction of PSB with the interface. During cyclic deformation, PSBs will continuously impinge the interfaces, leading to a high stress concentration nearby. The locally high stress concentration will induce the different interfacial fatigue damage behavior on a micro-scale level. Therefore, the intrinsic fatigue cracking mechanisms along the brittle IMC and interfaces among copper, IMCs and Sn–Ag–Cu solder can be well understood. As far as we know, such an evaluation method about the interfacial fatigue damage mechanisms in micro-scale has never been reported previously. It is expected that the current

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research on the fatigue damage behavior of the interface between the Sn–Ag–Cu and copper single crystal will provide a new approach for the evaluation of interfacial reliability in microscale.

2. Experimental procedure

The copper single crystal plate with a dimension size of 150 mm × 50 mm × 10 mm was grown from oxygen-free-high-conductivity (OFHC) copper of 99.999% purity by the Bridgman method in a horizontal furnace. Some rectangular specimens with a size of 50 mm × 6 mm × 5 mm were spark-cut from the copper single crystal plate. By the X-ray Laue back-reflection method, the orientation of the specimen along the loading direction in the present study was determined as $[\bar{1}\,6\,8]$. A lead-free solder alloy with the composition of Sn3.8Ag0.7Cu was prepared by melting high purity (>99.99%) tin, silver, and copper in vacuum at 800 °C for 30 min. Before the reaction of molten solder with copper single crystal, the copper surface was electropolished carefully and then rinsed in water and ethanol. Upon air drying, a commercial eutectic Sn–Ag–Cu paste was dispersed on the selected area of polished copper surface, and a SnAgCu alloy sheet was placed on the paste to ensure sufficient wetting reaction. The graphite plates were clamped on the sides of copper specimens with the solder paste to avoid the outflow of molten solder. Then the specimens were heated in an oven, where the reflow temperature was controlled at 240 °C for 15 min to evaporate the rosin flux before the specimens were cooled down in air. The isothermal aging of the as-reflowed specimens was conducted at 170 °C for 4, 7, and 16 days, respectively. Before fatigue tests, the specimens were carefully polished only in a mechanical way for the microstructure observations of solder, solder/copper interface and copper single crystal. All the specimens were cyclically deformed in push–pull on a Shimadzu servo-hydraulic fatigue testing machine under a constant axial plastic strain amplitude of 10^{-3} at room temperature in air. The axial cyclic loading of the prepared sample can be illustrated in Fig. 1. Cyclic deformation was interrupted at different cycles, then the surfaces of those specimens were observed with a LEO Super35 scanning electron microscopy (SEM) to examine the slip morphology of copper crystal and fatigue crack initiation at the interfaces. Finally, the bonding solder on the copper crystal was removed by etching deeply with a 10% nitric acid and 90% methanol solution, so as to observe the morphology of the IMCs and the cracking propagation path in the IMCs at the interface between the solder and copper single crystal substrate.

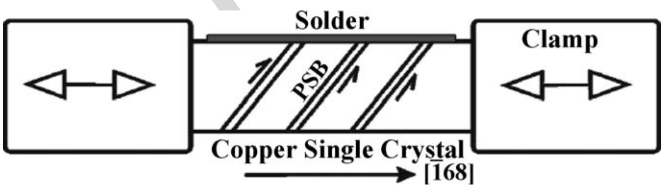

Fig. 1. Illustration of the fatigue specimen with an IMC interface between the solder and copper single crystal.

3. Results and discussion

3.1. Morphology and growth of interfacial IMCs

After reflow, a scallop morphology IMC layer was formed along the interface of SnAgCu/Cu single crystal joint, as shown in Fig. 2(a). EDX analysis indicates that the IMC layer is mainly Cu_6Sn_5 phase, but Cu_3Sn phase was not observed. The average thickness of the scallop-type Cu_6Sn_5 layer is about 4 μm. It reveals that the IMC Cu_6Sn_5 layer at the interface between SnAgCu solder and copper single crystal grew relatively rapidly during initial soldering. After isothermal solid-state aging at 170 °C for different times, the solder/copper single crystal interface layer exhibits a duplex layers of Cu_6Sn_5 and Cu_3Sn IMCs, as shown in Fig. 2(b)–(d). Obviously, the scallop-type Cu_6Sn_5 phase was changed to planar-type Cu_6Sn_5/Cu_3Sn during solid-state aging, which requires the growth of IMC in the valleys of the scallop. In other words, the IMC growth should be faster in the valley than that at the peak of the scallop in the initial stage of aging [25]. As a result, the top surface of the Cu_6Sn_5 layer remains somewhat wavy. Besides, some Cu_6Sn_5 phase was transformed into Cu_3Sn phase near the interface of IMC/copper, where the diffusion of Cu or Sn atoms through bulky layers of IMC would become difficult. The interface between Cu_6Sn_5 and Cu_3Sn phases is also quite wavy, but the interface between Cu_3Sn phase and copper single crystal becomes rather flat. With further thermal aging, the total IMCs layer continued to grow, albeit much more slowly. While a number of empirical relations have been proposed to predict the thickness of IMCs layer as a function of aging time and temperature for solder/polycrystalline copper interfaces [26,27], there is no related study on solder/copper single crystal interface. Fig. 3 shows the dependence of the thickness of interfacial IMCs on the aging time for the joint between SnAgCu solder and copper single crystal. It can be seen that the thickness of the IMCs increases linearly with the square root of the aging time. In addition, the layer of Cu_3Sn phase grew slightly more quickly than that of Cu_6Sn_5 phase. In general, it is empirically thought that the growth process of the IMCs layer was mainly controlled by the diffusion mechanism. This diffusion-controlled solid-state Cu–Sn IMC growth can be expressed by the following one-dimensional empirical equation [28,29]; i.e. $d = \sqrt{Dt}$, where d is the IMC thickness, D the diffusion coefficient, t is aging time. Based on the results in Fig. 3, the values D of diffusion coefficient were calculated as 7.24×10^{-17}, 1.68×10^{-17} and $1.94 \times 10^{-17}\,m^2\,s^{-1}$ for the whole Cu_6Sn_5/Cu_3Sn layer, Cu_6Sn_5 layer and Cu_3Sn layer respectively. The current diffusion coefficient D for the whole IMCs layer is approximately equal to the previous results of SnAgCu/polycrystalline copper interfaces, i.e. $8.1 \times 10^{-17}\,m^2\,s^{-1}$ at 190 °C [28] and $4.7 \times 10^{-17} m^2\,s^{-1}$ at 125 °C [29]. From the above results, it can be recognized that the morphology and growth of IMCs at solder/copper single crystal interface are similar to those at solder/polycrystalline copper, indicating that the microstructure of the different copper crystals did not play a significant role in the IMC growth during thermal aging.

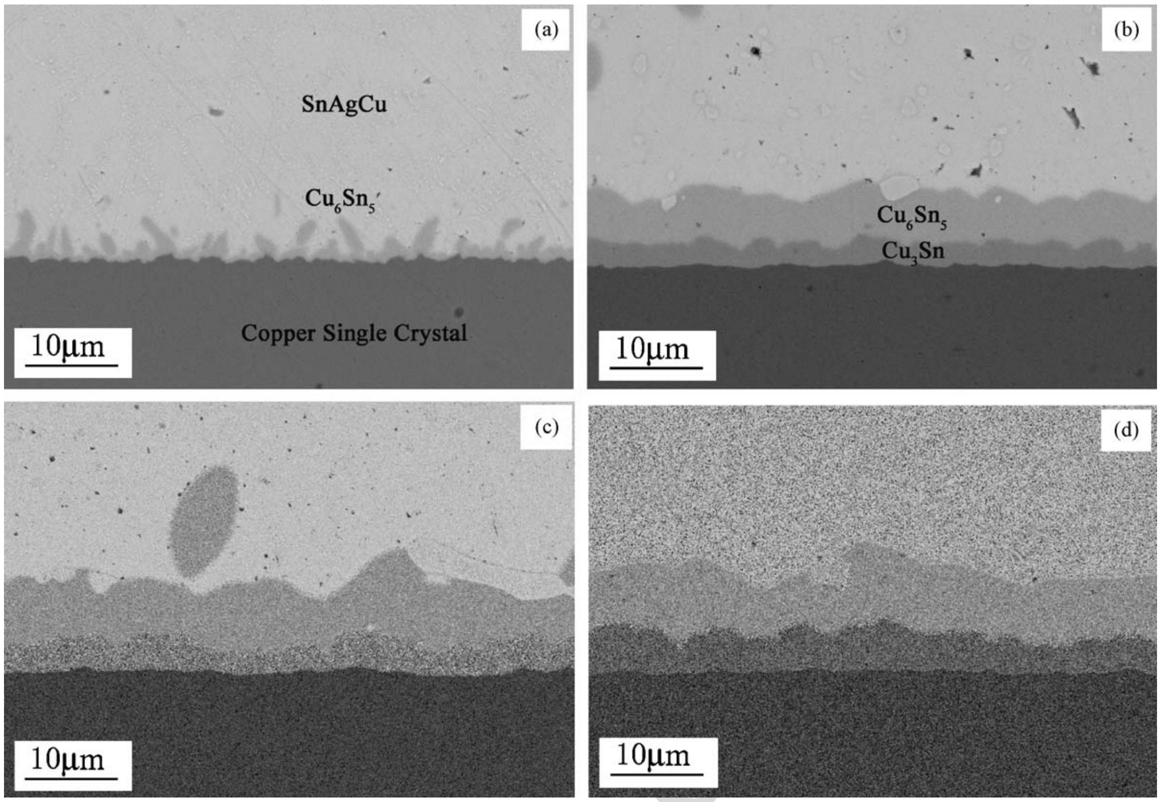

Fig. 2. SEM-backscattered electron micrographs of the morphology of IMCs at the SnAgCu/copper single crystal interface: (a) as-reflowed, (b) aged at 170 °C for 4 days, (c) 7 days, and (d) 16 days.

3.2. Interfacial fatigue damage behavior after reflow

During cyclic deformation of the reflowed specimen, some PSBs were activated on the copper single crystal surface. With increasing cyclic number, the density of PSBs increases. After only 650 cycles, the PSBs had accumulated to a high density, as shown in Fig. 4(a). Normally, PSBs can lead to the surface roughness by distinct extrusions and intrusions [30]. However, all the PSBs had not yet arrived at the interface of SnAgCu solder/copper single crystal during initial cyclic deformation. Even now, it is noted that all the cracks had first nucleated in the waist of the protruding region of the scallop-like Cu_6Sn_5 layer. The cracking orientation usually depends strongly on the morphology of the IMC layer itself, but is not associated with the impingement of PSBs to the interface. Such a cracking behavior is similar to that under tensile loading reported by Lee et al. [10,31]. With further cyclic deformation, the density of PSBs became higher and higher. Some PSBs could grow through the whole crystal and eventually terminated at the interface of IMC/copper single copper, as shown in Fig. 4(b). Surface observation showed that the cracking did not occur at the intersection sites of PSBs with interface, even though the interface was impinged continually by PSBs. On the contrary, the fracture would continue to develop within the scallop-like IMC layer. Some fragments of the Cu_6Sn_5 scallops were embedded into the adjacent solder matrix, and then the interface seems to be more planar than the initial state. In the study of Prakash et al. [32], these fragments of IMC phase were identified at the base of the microvoids in tensile fracture surface. After cycling for about 4000 cycles, it can be seen from Fig. 4(c) that there is a serious plastic deformation within both SnAgCu solder and copper single crystal. The transverse cracks in individual scallops of IMC started to extend into the adjacent solder. Meanwhile, it is found that some cracks began to initiate at the intersection sites of PSBs with the interface of IMC/copper single crystal. Under cyclic loading, these cracks propagated into residual thin

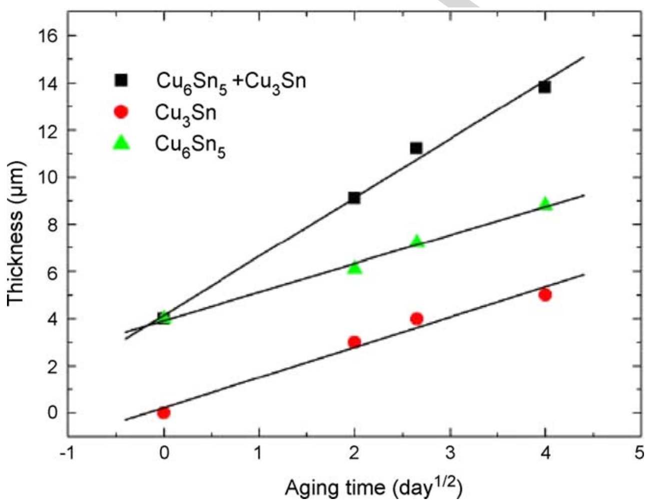

Fig. 3. Dependence of the thicknesses for the IMC layers at the SnAgCu/copper single crystal joint on the aging time at 170 °C.

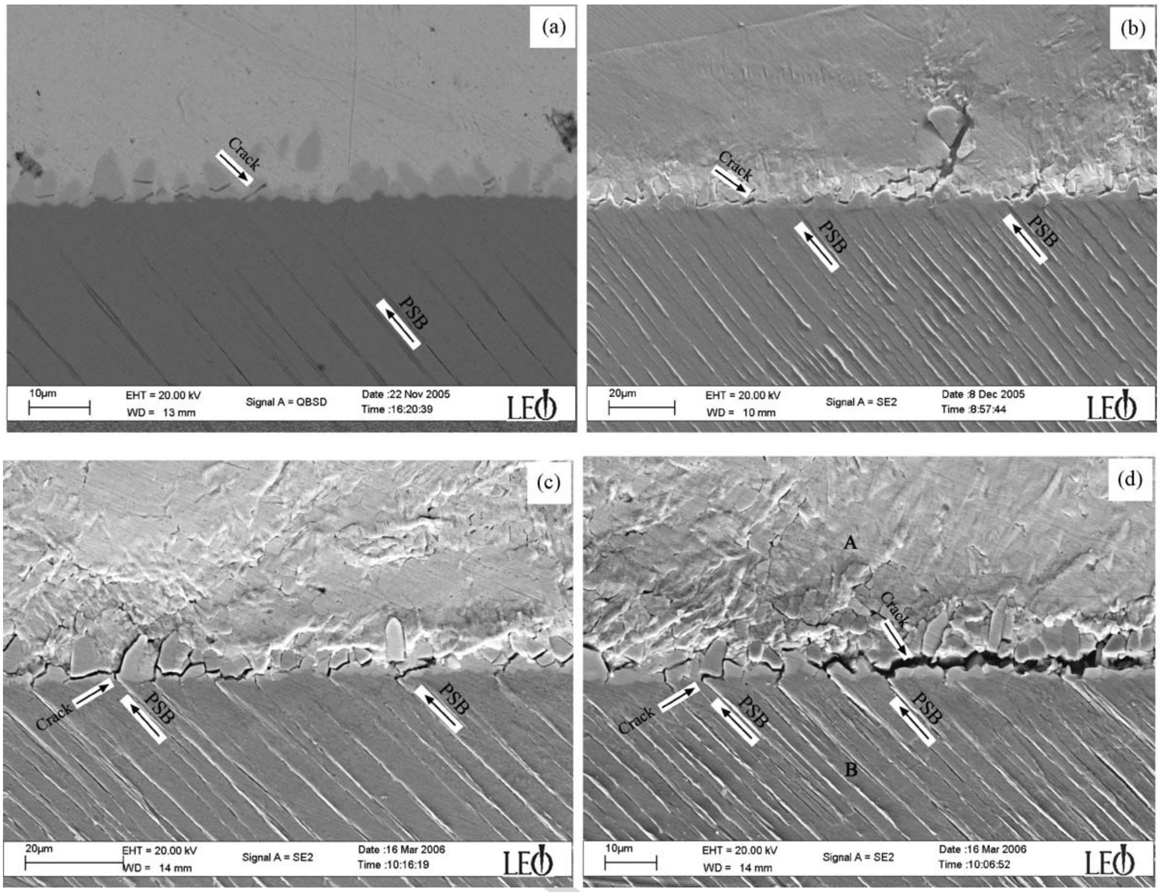

Fig. 4. SEM micrographs of the damage processes at the as-reflowed SnAgCu/copper single crystal interface during cyclic deformation: (a) 650 cycles, (b) 3000 cycles, (c) and (d) 4000 cycles.

IMC layer along direction approximately vertical to the interface. On the other hand, when the PSBs were blocked by the interface, it is noted that the PSBs in the vicinity of interface would become quite rough. In local regions, when a number of transverse cracks propagated into the solder and linked each other, the interfacial fracture between A and B would occur easily, as shown in Fig. 4(d). Similarly, Lee et al. [31] also pointed out that the fracture occurred first at the IMC bulge and then the crack propagated into solder, forming a dimple-like fractured morphology for the scallop-type IMC interface under tensile loading. From the results above, it can be concluded that the cracking in the extrusion of IMC layer is the preferential damage mode for the scallop-type IMC interface. Though the cracks occurred at the interface of IMC/copper single crystal after a continuous impingement of PSBs to the interface, these cracks had never propagated along the interface, indicating a strong bonding property between copper single crystal and the IMC.

3.3. Interfacial fatigue damage behavior after aging

The interaction of PSBs with the planar-type IMCs interface after aging is presented in Fig. 5. There are only a few PSBs appearing irregularly on copper single crystal surface after about 150 cycles, as shown in Fig. 5(a). The fatigue crack began to initiate within the thick IMCs layer perpendicular to the interface obviously by the impingement of PSBs. Normally, the fatigue cracks often originated initially from the intersection sites of PSBs with the interface and have a good one-to-one relationship with PSBs. From that, it seems that these cracks should be attributed primarily to the interacting of PSBs with the thick IMCs layer. Zhang et al. [33,34] investigated the fatigue cracking behavior of copper bicrystals, and found that the fatigue cracks first nucleated at the intersection sites of PSBs with grain boundary (GB) and then propagated along the GB plane. However, in the present case, since the fracture toughness (K_{IC}) of the IMCs is very low, if a crack or a groove was formed at an intersection site of a PSB with IMC layer, the crack or the groove would be split easily across the IMCs layer, instead of propagating along the interface of IMC/copper single crystal joint. The path of crack propagation in the interior of IMC is quite straight and approximately vertical to the interface, i.e. in a mode I fracture, as shown in Fig. 5(b) under a higher magnification. From that, it is also noted that there is a serious strain incompatibility near the interface of solder/IMC, where the interfacial cracks initiated when the propagation of crack within the IMC terminated at the interface. With further cyclic deformation, the strain incompatibility between the solder and IMC became more serious, which often leads to a severe stress concentration in the vicinity of solder/IMC interface [31]. The cracks propagated rapidly along the interface of solder/IMC, as shown in Fig. 5(c), and the interface was broken eventually after a high plastic strain accumulation,

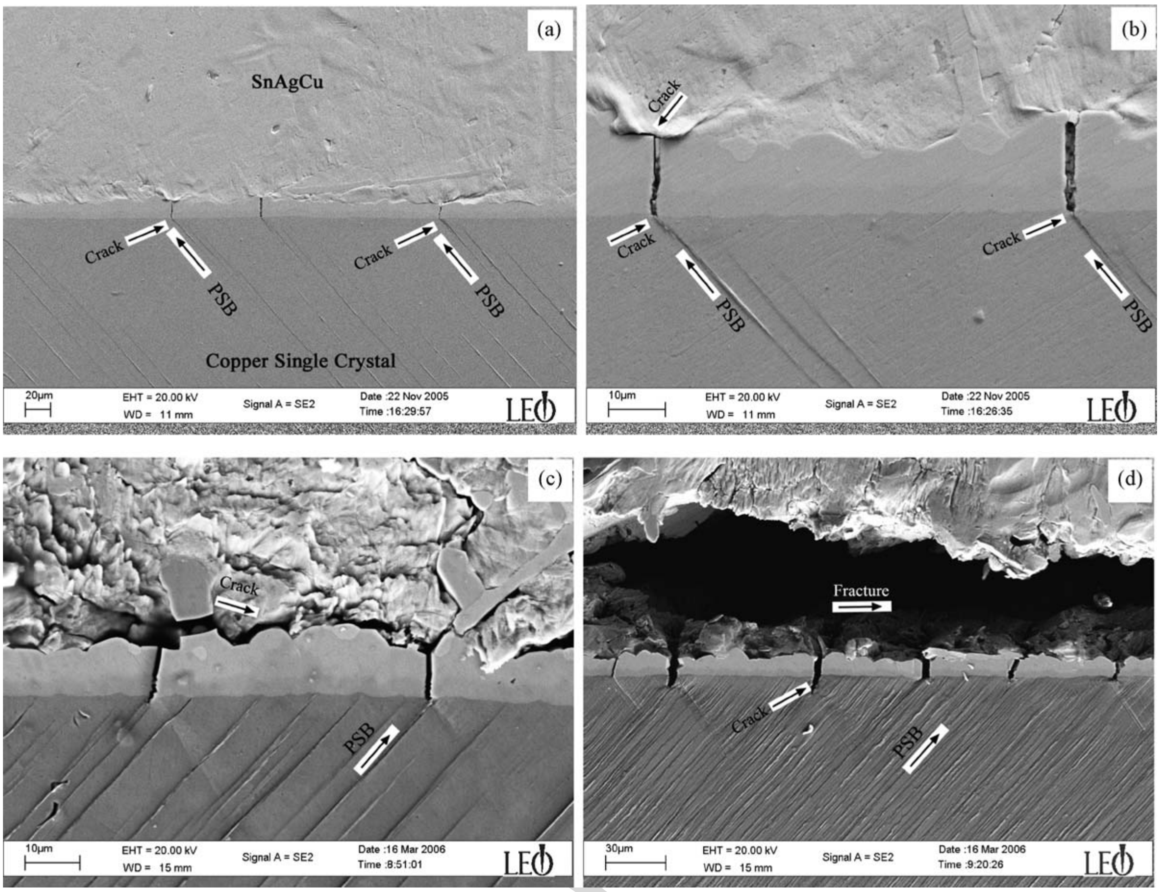

Fig. 5. SEM micrographs of the damage processes at the aged SnAgCu/copper single crystal interface during cyclic deformation: (a) 200 cycles, (b) 200 cycles, the magnified image of (a), (c) 2000 cycles, and (d) 3000 cycles.

as shown in Fig. 5(d). On the other hand, it is observed that the number of cracks in IMCs did not increase remarkably even though the PSBs had accumulated to a high density. Under cyclic loading, the gaps of these cracks can be obviously widened, and even some of them began to propagate into the copper substrate, as indicated by the arrow in Fig. 5(d). From the present results, it can be concluded that under cyclic loading the thick planar-type IMC interface produced by aging is first damaged by the interacting PSBs, indicating a typical brittle-fracture feature. It was reported [10,31,32] that when joints were subjected to tensile or shear loading, the cracks usually initiated and propagated laterally through the thick planar Cu_6Sn_5 layer, leaving a cleavage fracture morphology. It seems that this thick planar IMC layer is easier to be damaged than the thin scallop-type layer, which is supposed to be associated with the detrimental effect on the cohesive strength for the aged joint [10,14,32]. Therefore, it can be concluded that quite different damage mechanisms operate at the scallop and planar-type interfaces under cyclic deformation, which is an interesting phenomenon and will be further discussed in Section 3.5.

3.4. Fatigue cracking path within IMC

When the top solder was etched away, the microstructure of Cu_6Sn_5 IMC and the propagation of fatigue cracks within IMC layer can be well shown in Fig. 6. It is seen that the microstructure of Cu_6Sn_5 IMC layer is composed of equiaxed grains with an average grain size of about 8 μm by image analysis. Besides that, some residual β-Sn grains with bright contrast could also be obviously identified from the gray Cu_6Sn_5 grains. These fatigue cracks propagated through the thick IMCs layer with a quite straight path, as shown in Fig. 6(a), exhibiting a typical feature of brittle fracture. Although it was previously shown that the grains in IMC layer have a preferred crystallographic orientation [35], it seems that the growth of fatigue cracks is independent of the crystallographic orientation. In addition, from Fig. 6(b), it is clearly observed that the fatigue cracking path within Cu_6Sn_5 IMC layer consists of both intergranular and transgranular manners, indicating that the fatigue crack propagation within Cu_6Sn_5 layer should be a random or abrupt process. The observations above further confirm that the Cu_6Sn_5 IMC is a very brittle phase, and is harmful to the interfacial strength of interconnection. On the other hand, it is also noted that the fatigue crack is difficult to pass through the soft Sn grains, and had to occasionally change the propagation path, indicating that the Sn solder has a better fracture toughness than the IMC phase. This result agrees well with the observation in Fig. 5 that the propagation of cracks within the IMCs terminated at the solder/IMC interface and then turned to propagate along the interface.

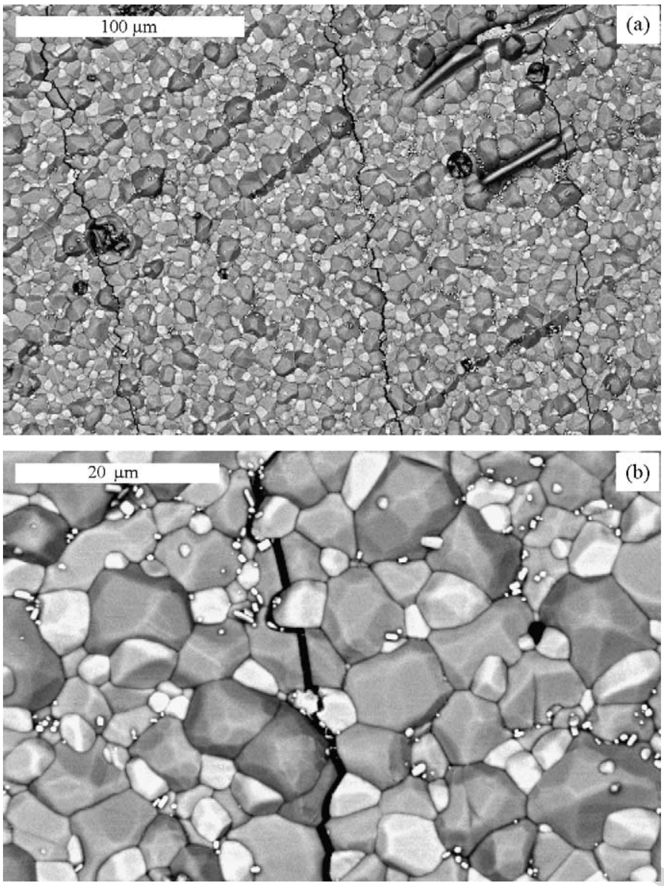

Fig. 6. SEM-second electron micrographs of the crack propagation within the Cu_6Sn_5 IMC layer: (a) straight propagation path and (b) intergranular and transgranular manners.

3.5. Fatigue cracking mechanisms of scallop and planar-type IMCs interfaces

During cyclic deformation, the extrusions and intrusions are often the preferential sites for the nucleation of fatigue cracks in copper single crystal [30,36]. But in most cases, the dislocations carried by PSBs are often blocked by the interfaces, such as grain boundaries [33]. In the present case, when PSBs met with the IMC layer, it is natural that PSBs could not pass through the interface. As a result, with continuous cyclic deformation, the residual dislocations within the PSBs would pile-up at the interface, forming a high stress concentration zone near the intersection site of PSBs with the interface. From that, it can be known that the essence of the interactions of PSBs with interface in the present study should be the process of accumulating dislocations at the interfaces. Accordingly, the IMC layer had to bear an accumulating elastic strain under a continuous impinging of PSBs. On the other hand, since there is a serious strain incompatibility between the brittle protrudes of IMC and the adjacent soft solder, the cracking of protrudes should be the preferential damage mode for the scallop-type IMC layer, as schematically illustrated in Fig. 7(a), so as to release the elastic energy in good time. Thus, the cracking at the intersection of PSBs with IMC interface can be effectively delayed. Unfortunately, unlike the scallop-type IMC layer, the accumulating elastic deformation energy within the thick planar IMC layer could not be released in time through the local cracking of protrudes. Consequently, the thick IMC layer can be easily broken by the impingement of PSBs at the interface, as illustrated in Fig. 8(a).

Generally speaking, the fatigue failure always occurred in the vicinity of the interface of IMC/solder, as illustrated in Fig. 7(b) and Fig. 8(b), respectively, which is basically attributed to the serious strain incompatibility between the

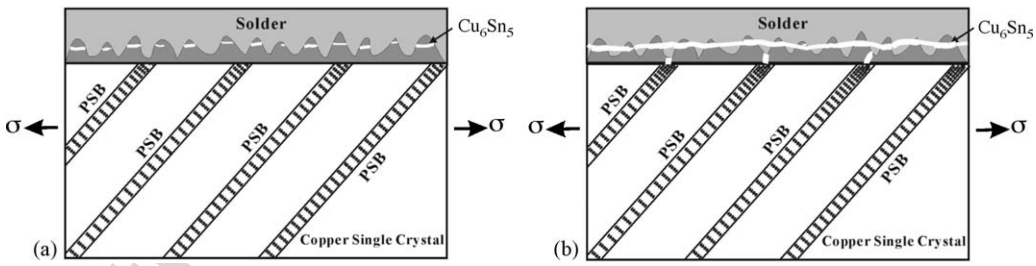

Fig. 7. Illustration of the damage process at the as-reflowed interface under the interaction of PSBs with the interface: (a) fracture of the protrudes of IMC and (b) cracking at the intersection site of PSBs with the interface and failure along the interface of solder/IMC layer.

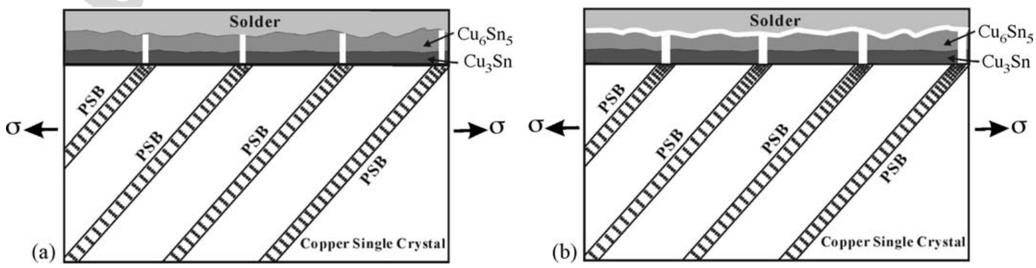

Fig. 8. Illustration of the damage process at the aged interface under the interaction of PSBs with the interface: (a) cracking at the intersection site of PSBs with the interface and (b) failure along the interface of solder/IMC layer.

solder and IMC during cyclic deformation. However, it can be obviously noted that the fatigue crack growth along the interface of solder/scallop-type IMC is much more difficult than that along the planar interface because of its rougher IMC/solder interface. As is also reported in Yao and Shang's work [37], the fatigue crack growth resistance increases with increasing the roughness of the IMC/solder interface at the low strain energy release rate. In other words, if the fracture occurs exactly on the irregular interface, it will have to change direction abruptly and frequently to propagate, which would be energetically unfavourable [32]. It appears clearly that, when the scallop-like morphology of IMC grows into planar-like, the fatigue crack growth resistance decreases, resulting in the decrease in the fatigue life of the solder joints. From the comparison above, it can be concluded that the growth of IMC layer can significantly deteriorate the mechanical integrity of the joints because of the excessive thickness as well as the morphology of the IMC layer.

4. Conclusions

Based on the experimental results and analysis above, the following conclusions can be drawn.

1. The IMC interface between the SnAgCu solder and copper single crystal was successfully designed. During solid-state aging, the thin scallop-type Cu_6Sn_5 IMC layer produced in re-flowing was transformed into thick planar-type layer with a duplex structure of Cu_6Sn_5 and Cu_3Sn IMCs. The growth of thickness of IMC had a good linear relationship with square root of aging time with diffusion coefficients D of 7.24×10^{-17}, 1.68×10^{-17} and 1.94×10^{-17} $m^2 s^{-1}$ respectively for the whole Cu_6Sn_5/Cu_3Sn layer, Cu_6Sn_5 layer and Cu_3Sn layer.
2. During cyclic deformation, the fatigue cracking first occurred at the protuberance of IMC layer for the scallop-type IMC interface after reflow, and subsequently nucleated at the intersection sites of PSBs with the interface after high cycles, which can be attributed to the piling-up mechanism of dislocations carried by PSBs.
3. The thick planar IMC layer after aging was easier to fracture approximately perpendicular to the interface due to the impingement of PSBs to the interface. However, fatigue cracks were not observed to propagate along the interface of IMC/copper single crystal. The fatigue failure always occurred along the interface of solder/IMC for both types of IMC interfaces. However, the fatigue crack propagation resistance along the interface of solder/scallop-type IMC is better than that along the planar-type interface. These results obtained in a micro-scale damage condition could have important implications in interconnection engineering, particularly for controlling the interfacial IMC layer in manufacturing process.

Acknowledgements

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