Isothermal Fatigue Properties of Sn–Ag–Cu Alloy Evaluated by Micro Size Specimen

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Micro-bulk fatigue testing developed to investigate the fatigue lives and damage mechanisms of Sn–3.0Ag–0.5Cu and Sn–37Pb solder alloys. The fatigue life of micro-bulk solder obeyed Manson–Coffin’s empirical law, and the fatigue ductility exponents were about 0.5 for both Sn–Ag–Cu and Sn–Pb alloys. The fatigue life of Sn–3.0Ag–0.5Cu alloy was 10 times longer than that of Sn–37Pb alloy under symmetrical cycling at 298 K, although fatigue resistance of Sn–3.0Ag–0.5Cu alloy was not very superior under asymmetrical wave and elevated temperature condition. The fatigue crack was developed from extrusion and intrusion of slip band in Sn–3.0Ag–0.5Cu alloy, while the crack was observed at colony boundary and grain boundary in Sn–37Pb alloy. The difference in damage mechanism may affect the susceptibility to fatigue life test condition of reversibility.

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1. Introduction

Understanding fatigue properties of solder alloys is important to evaluate reliability of solder joints, since cyclic mechanical deformation can occur at solder joints due to thermomechanical fatigue (e.g., environmental-temperature cycles on joined materials with different coefficients of thermal expansion). In addition to thermomechanical fatigue, the solder joint can also be subjected to mechanical fatigue, typically when the board or substrate is bent during mechanical handling or from vibration forces. Therefore, a number of studies on mechanical fatigue of solder alloys to understand fatigue failure of solder joints have been performed to date. In general, mechanical properties of solder alloys such as creep and fatigue are measured using larger volume bulk specimens than real solder joints as there are many difficulties in sample preparation and mechanical testing. However, the microstructure of the real joint is totally different to that of the large size mechanical testing specimen, since solidification conditions of real joints cannot be duplicated in large size. We proposed a tensile testing using micro size specimen to obtain the mechanical behavior of Sn–Ag–Cu lead free solder alloy, and the testing resulted in that the mechanical behaviors of the micro size and the large specimens are not similar. In addition to such differences in the mechanical properties, specimen size also affects crack initiation and propagation behaviors in low cycle fatigue. Therefore, the low cycle fatigue test should also be performed using micro size specimen. A number of studies on low cycle fatigue of solder alloys were reported to date, whereas there are no studies on that using small volume specimen.

In this study, we developed micro-bulk fatigue testing method for solder alloys, and the isothermal fatigue properties of lead-free solder alloy were tested to understand fatigue damage of lead-free solder alloy in small volume.
cally in Fig. 2. The specimens were heat-treated at 423 K for 2 h to stabilize the microstructure at high temperature.

2.2 Isothermal fatigue tests

Total strain controlled isothermal fatigue tests were carried out in air at 298 and 398 K with the application of a precise displacement controlled mechanical testing machine (Saginomiya MFT-01). Figure 3 shows schematic illustration of the fatigue testing machine. A linear DC motor was employed for an actuator of the machine, and the displacement resolution of the actuator and maximum load capacity are 20 nm and 200 N, respectively. The total strain range chosen was between 0.3 and 1.3 percent. Fatigue life was defined as the number of cycles at which the load decreases to 20 percent from initial value. The total strain was obtained by measuring a displacement between specimen fixing jigs with a capacitance displacement sensor that has a resolution of 0.04 mm. Two types of symmetrical (Fast–Fast) and asymmetrical (Slow–Fast) continuous strain cycling as shown in Fig. 4 were employed in this study to investigate an effect of irreversibility of loading profile on the fatigue life. A fast ramp rate of $\frac{10}{s}$ and a slow ramp rate of $\frac{3}{10}s$ were used in asymmetrical cycling. Test temperature was controlled by an infrared image furnace, and was maintained within 0.2 K throughout the test.

2.3 Microstructural observation

The specimens were ground with two grades of SiC papers (#500, and 1200) and then mechanically polished with diamond paste (15 μm). Finally, the specimens were polished with colloidal silica suspension. After polishing, they were observed using an optical microscope without any chemical etching.

3. Result and Discussion

3.1 Microstructure

Figure 5 shows optical micrographs for the Sn–3.0Ag–0.5Cu (polarized image) and Sn–37Pb. The microstructure of Sn–3.0Ag–0.5Cu alloy consists of Sn dendrites surrounded by a eutectic of Sn, Ag$_3$Sn and Cu$_6$Sn$_5$. Very fine spherical intermetallic compounds were formed in the eutectic region. Morphology of the Sn dendrite is columnar and equiaxial dendrites were not observed in the specimen as can be seen in Figs. 5(a) and (b). The Sn dendrites with similar crystal orientation are clustered (same contrast region), making large grains in the miniature specimen. Only a few grains were observed in the gauge section in the micro bulk specimen, whereas a large volume cast specimen seems to be in polycrystalline state with equiaxial Sn dendrites.6)

Figures 5(c) and (d) show optical micrograph of as solidified Sn–37Pb alloy. The microstructure consists of fine lead and tin solid solutions. The classic eutectic structure (two paralleled lamellar structure) was suppressed by substantial undercooling, and fine two phase mixed structure and relatively small size colonies were observed in the micro bulk specimen. The size of Pb phases was about 1–5 μm in the microstructure.

3.2 Fatigue life of micro bulk specimen

Figure 6 shows hysteresis loop observed at total strain range of 1.2% at three cycles for the micro bulk specimen. Typical hysteresis loop of low cycle fatigue was observed as shown in Fig. 6. Young’s modulus obtained from the hysteresis loop were about 42 GPa that well agrees with a typical value measured in a bulk specimen.8,9) The modulus means that strain measurement method adopted here is appropriate, and that the testing method enables to precisely
measure mechanical response of micro bulk solder specimen.

Figure 7 shows relationship between inelastic strain range and fatigue life of Sn–3.0Ag–0.5Cu alloy at 289 and 398 K. The data on strain range versus fatigue life are plotted on log–log scales against the inelastic strain range. All data obeyed Manson–Coffin’s law as follows.

$$\Delta \varepsilon_{\text{in}} \cdot N_f^\alpha = C$$  \hspace{1cm} (1)

where $\Delta \varepsilon_{\text{in}}$ is the inelastic strain range, $N_f$ is the fatigue life, $\alpha$ is the exponent and $C$ is the fatigue ductility. The exponent for all condition is about 0.5, which is similar to that measured in large bulk specimen of Sn–Ag solders.\(^6\)–\(^12\) As can be seen in Fig. 7, the fatigue life of the alloy is sensitive

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**Fig. 5** Optical micrographs for the micro bulk specimens of Sn–3.0Ag–0.5Cu and Sn–37Pb.

**Fig. 6** Hysteresis loop for the miniature specimen at strain range of 1.2% at 298 K for Sn–3.0Ag–0.5Cu.

**Fig. 7** Manson–Coffin plots for Sn–3.0Ag–0.5Cu alloy.
to symmetry of strain cycling and test temperature. The fatigue life in asymmetrical cycling is approximately one-tenth of that in symmetrical strain cycling, and increase in test temperature reduces the life to one-third of that at lower temperature. Thus, asymmetrical cycling dramatically reduces the life of Sn–3.0Ag–0.5Cu alloy, whereas a deleterious effect of temperature upon fatigue life is less than that of reversibility in strain profile. Figure 8 shows relationship between inelastic strain range and fatigue life of Sn–37Pb alloy at 289 and 398 K. All data for Sn–37Pb also obeyed Manson–Coffin’s law, and the exponent is about 0.5 for each condition similar to Sn–3.0Ag–0.5Cu alloy. The life of Sn–37Pb is also very sensitive to symmetry of strain cycling as shown in Fig. 8, while the deleterious effect of asymmetrical cycling upon fatigue damage is more moderate than that in Sn–3.0Ag–0.5Cu alloy. On the other hand, the test temperature does not have deleterious effect on the fatigue life of the alloy. The fatigue life at 398 K in symmetrical cycling is obviously longer than that at 298 K. The result shows that test temperature enhances the life in symmetrical mode, which does not have deleterious effect on fatigue life in asymmetrical mode for Sn–37Pb alloy, whereas an increase in test temperature has deleterious one for Sn–3.0Ag–0.5Cu. The Sn–37Pb is widely known as one of the superplasticity alloys. The alloy exhibits exceptional ductility (over a thousand percent of the failure elongation) when pulled under tension at high temperature. The exceptional ductility is induced by the grain boundary sliding with fine microstructure. The low cycle fatigue life strongly depends on the fracture ductility, and high ductility leads superior low cycle fatigue endurance. In this study, the grain boundary sliding may govern deformation of the alloy at 398 K, since very fine microstructure was obtained as shown in Fig. 5. The mechanism leads high ductility which enhances the fatigue life of Sn–37Pb at 398 K.

Figure 9 shows comparison between Sn–3.0Ag–0.5Cu and Sn–37Pb for low cycle fatigue endurance. The fatigue life of Sn–3.0Ag–0.5Cu is much longer than that of Pb–Sn eutectic in symmetrical cycling at 298 K and the life of Sn–3.0Ag–0.5Cu is about seven times of that of Pb–Sn eutectic. However, the life of Sn–3.0Ag–0.5Cu is inferior to the life of Sn–37Pb at 398 K in symmetrical cycling. In addition, the fatigue life of Sn–3.0Ag–0.5Cu in slow–fast cycling is very similar to that of Pb–Sn eutectic as is evident in Fig. 9. Thus, superior fatigue resistance of Sn–3.0Ag–0.5Cu alloy was obtained only in symmetrical cycling at 298 K.

3.3 Fatigue damage formation mechanism

3.3.1 Damage mechanism of Sn–3.0Ag–0.5Cu alloy

Figure 10 shows surface morphology and cross-sectional optical micrograph near crack initiation site for Sn–3.0Ag–0.5Cu alloy subjected to the total strain range of 1.0% at 298 K in fast–fast cycling. The formation of a pair of intrusion and extrusion was observed on fatigued surface of Sn–3.0Ag–0.5Cu alloy. The displacements within the slip band were not fully reversed, which leads to the fatigue crack nucleation. The fatigue crack initiation along the slip band was revealed by the cross-sectional observation as shown in Fig. 10(b). Thus, the fatigue crack started from the slip bands and propagated within grain interior in the symmetrical cycling at 298 K. However, the crack propagated along a high angle grain boundary, when the crack arrives at the boundary as shown in Fig. 11. Thus, the mixed mode of transgranular and intergranular failure is predominant for Sn–3.0Ag–0.5Cu alloy at 298 K in the symmetrical cycling.

Figure 12 shows fatigue crack initiation and propagation in Sn–3.0Ag–0.5Cu alloy at 398 K. The formation of an intrusion and extrusion pair was also observed, and the fatigue crack initiation along the slip band was visible at 398 K in fast–fast cycling. However, the fatigue crack prefers to propagate along a high angle grain boundary rather than to propagate within grain interior as can be seen in Fig. 12(a). Especially, the crack nucleation prefers the high angle grain boundary, which makes a large angle with the loading axis. Any significant recrystallization was not observed in the symmetrical cycling at 298 K, even though the test temper-
ature corresponds to $0.8T_m$ of the alloy. Thus, the intergranular failure is predominant at 398 K in the symmetrical cycling. An increase in the test temperature may promote the crack propagation due to intergranular failure, and contributing to the reduction in fatigue life as shown in Fig. 7.

On the other hand, the recrystallization of tin was observed around the fatigue crack in the asymmetrical cycling at 398 K as shown in Fig. 12(b), even though that was not observed in the symmetrical cycling. As the recrystallization requires a high strain energy accumulation, the result suggests that the strain energy due to cyclic deformation in the asymmetrical cycling is much higher than that in the symmetrical cycling. The slip deformation in the asymmetrical cycling may be irreversible, so that plastic strain is highly accumulated around crack nucleation site by irreversible slip deformation, which leads to the recrystallization of tin phase. The crack propagation rate at the grain boundary is faster than that in grain interior at elevated temperature condition, which contributes to the reduction in fatigue life as shown in Fig. 7. Thus, the asymmetrical cycling induces the recrystallization of tin phase, and the recrystallization promotes the crack propagation due to intergranular failure for Sn–3.0Ag–0.5Cu alloy. However, the deleterious effect of asymmetrical cycling upon fatigue life of Sn–Ag–Cu alloy is yet to be elucidated.

### 3.3.2 Damage mechanism of Sn–37Pb alloy

Figure 13 shows surface morphology and cross-sectional optical micrograph of Sn–37Pb alloy subjected to fatigue at total strain range of 1.0% at 298 K as shown in Fig. 12(b), even though that was not observed in the symmetrical cycling. As the recrystallization requires a high strain energy accumulation, the result suggests that the strain energy due to cyclic deformation in the asymmetrical cycling is much higher than that in the symmetrical cycling. The slip deformation in the asymmetrical cycling may be irreversible, so that plastic strain is highly accumulated around crack nucleation site by irreversible slip deformation, which leads to the recrystallization of tin phase. The crack propagation rate at the grain boundary is faster than that in grain interior at elevated temperature condition, which contributes to the reduction in fatigue life as shown in Fig. 7. Thus, the asymmetrical cycling induces the recrystallization of tin phase, and the recrystallization promotes the crack propagation due to intergranular failure for Sn–3.0Ag–0.5Cu alloy. However, the deleterious effect of asymmetrical cycling upon fatigue life of Sn–Ag–Cu alloy is yet to be elucidated.
cycling. As can be seen in the figure, the fatigue crack was initiated and propagated along colony boundary, which is totally different to the failure mechanism in Sn–3.0Ag–0.5Cu alloy. Similar failure mode is often observed in actual solder joints made using Sn–37Pb alloy.16)

Figure 14 shows cross-sectional optical micrograph of Sn–37Pb alloy subjected to fatigue at total strain range of 0.5% at 398 K.

4. Summary

Micro-bulk fatigue testing was developed to investigate the fatigue lives and damage mechanisms of Sn–3.0Ag–0.5Cu and Sn–37Pb solder alloys. The fatigue life of Sn–3.0Ag–0.5Cu was much longer than that of Pb–Sn eutectic in symmetrical cycling at 298 K, and the life of Sn–3.0Ag–0.5Cu was about seven times of that of Pb–Sn eutectic. However, the fatigue life of Sn–3.0Ag–0.5Cu at 398 K was somewhat shorter than that of Pb–Sn eutectic, since the life of Sn–37Pb was increased with increasing temperature. In addition, the fatigue life of Sn–3.0Ag–0.5Cu in asymmetrical cycling was very similar to that of Pb–Sn eutectic. Thus, Sn–3.0Ag–0.5Cu alloy exhibits superior fatigue resistance compared with Pb–Sn eutectic in symmetrical cycling at 298 K, whereas the lives were almost equivalent in asymmetrical cycling or at the elevated temperature.

The fatigue crack started from the slip bands and propagated within grain interior in the symmetrical cycling at 298 K. However, an increase in the test temperature promoted the crack propagation due to intergranular failure, and contributed to the reduction in fatigue life. In addition, the asymmetrical cycling induced the recrystallization of tin phase, and the recrystallization promoted the crack propagation due to intergranular failure for Sn–3.0Ag–0.5Cu alloy. On the other hand, the crack was observed at colony boundary in Sn–37Pb alloy at 298 K. The fatigue crack propagated at the boundary between Pb and Sn phases, which is induced by the grain boundary sliding. The grain boundary sliding enhanced fatigue resistance of Sn–37Pb, since the grain boundary sliding induces exceptional ductility. The difference in damage mechanism between Sn–Ag–Cu and Pb–Sn may affect the susceptibility of test condition to fatigue endurance.

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