Estimation of Thermal Fatigue Resistances of Sn–Ag and Sn–Ag–Cu Lead-Free Solders Using Strain Rate Sensitivity Index

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A tensile test was conducted to evaluate thermal fatigue resistances of Sn–Ag and several Sn–Ag–Cu lead-free solder alloys. The test is based on the strain rate change method to obtain a strain rate sensitivity index, m . The m values were investigated at various strains during the tensile test until fracture. The plots of m and strain where m is measured showed a linear relationship. Therefore, the m value at zero strain, m_0 , and the gradient of the fitting line, k , were obtained by extrapolation. Using m_0 and k values investigated, an estimation of the thermal fatigue resistance of the solder joint was attempted. The ranking of the reciprocals of m_0 and k in the solders is relatively in good agreement with that of the thermal fatigue resistance of the solder joints, and thus m_0 and k can be indexes to develop a new lead-free solder with the excellent thermal fatigue resistance. Moreover, the influences of the aging treatment on m_0 , k and the microstructure of the solder were also investigated.

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1. Introduction

Lead-free soldering has been widely spread in electronics assembly due to toxicity of the lead for human health. Sn–Ag–Cu and Sn–Ag alloys are used as a substitute of a conventional Sn–Pb eutectic solder, although the melting points of Sn–Ag system alloys are a little higher than that of the Sn–Pb eutectic solder. The Sn–Ag and Sn–Ag–Cu alloys have excellent pull strength,¹⁾ creep resistance²⁾ and fatigue resistance,³⁾ compared to the Sn–Pb eutectic solder.

For recent fine pitch solder joints, excellent mechanical and thermal fatigue properties are required, because the electronic devices are exposed under temperature cycle conditions. Especially, the thermal fatigue resistance is important to the reliability of the solder joint.^{4,5)} The thermal fatigue resistance is evaluated by a temperature cycle test and it takes much time. Thus, many studies have been conducted in order to predict the thermal fatigue life of the lead-free solder joint based on the Coffin–Manson equation.^{6,7)} Some studies conducted on the relationship between the thermal fatigue resistance and the mechanical properties of the solders^{8,9)} and it was clarified that the creep properties is very important factor in the thermal fatigue resistance of the solder. In particular, it was reported the ranking of the steady state creep rate at the same stress in several lead-free solders does not change in the creep rate ranging from 10^{-6} to 10^{-3} s^{-1} .^{10,11)} It means the possibility of the thermal fatigue life prediction of the solder joint using the creep properties of the solder.

A strain rate change test is simple mechanical test and can investigate the creep properties such as a strain rate sensitivity.¹²⁾ Takemoto *et al.*, who is one of the authors, have reported a good correlation between the strain rate sensitivities and the thermal fatigue resistances in several solder alloys.^{13–15)} Since the thermal cycle conditions used in the reliability test of electronics devices are various, it is often difficult to evaluate precisely the thermal fatigue resistances of the solder under various conditions even using

the Coffin–Manson equation.¹⁶⁾ However, the simple evaluation method of the thermal fatigue resistance of the solder is very effective for the quick development of new lead-free alloys. The strain rate change test can become the simple test to evaluate the thermal fatigue resistance of the solder, because the strain rate change test takes shorter time than the conventional creep test.

This study aimed to investigate the strain rate sensitivities of several Sn–Ag and Sn–Ag–Cu lead-free solders by the strain rate change test and also aimed to estimate their thermal fatigue resistances by the strain rate sensitivities. The effect of content of Ag and Cu, on the strain rate sensitivity and microstructure, and the effect of aging on them were also investigated. Moreover, similar investigation with the Sn–Pb eutectic solder was conducted to compare with the lead-free solders.

2. Experimental Procedures

The solders studied are several Sn–Ag–Cu alloys, Sn–3.5 mass%Ag and Sn–37 mass%Pb as listed in Table 1. The solidus and liquidus temperatures for the solders are also indicated in the table. The ingot of each alloy was melted at the temperature of $100 \pm 5^\circ\text{C}$ above the liquidus temperature of the alloy in air. The molten alloy was cast into a cylindrical

Table 1 Chemical compositions of solders studied.

Solder alloy	Amount of element (mass%)				Solidus Temp. (°C)	Liquidus Temp. (°C)
	Ag	Cu	Pb	Sn		
Sn–3.5Ag	3.5	—	—	Bal.	221	221
Sn–3Ag–0.5Cu	3.0	0.5	—	Bal.	217	219
Sn–2.8Ag–0.3Cu	2.8	0.3	—	Bal.	217	221
Sn–3.5Ag–0.7Cu	3.5	0.7	—	Bal.	217	217
Sn–3.8Ag–0.7Cu	3.8	0.7	—	Bal.	217	217
Sn–4Ag–0.9Cu	4.0	0.9	—	Bal.	217	217
Sn–37Pb	—	—	37	Bal.	183	183

stainless steel mold, and a cylindrical ingot was prepared. The diameter and length of the ingot were approximately 30 and 250 mm, respectively. A rod-shaped tensile test specimen, with 50 mm gauge length and 10 mm diameter, was machined from the cylindrical ingot. The tensile test specimen was aged at 120°C for one week and three weeks before the tensile test to investigate the influence of the aging treatment on the mechanical properties. Moreover, microstructural observations were performed for the center of the grip region of each specimen using electron probe X-ray microanalysis (EPMA).

The strain rate change test was performed at room temperature for specimens without and with aging treatment using an Instron-type tensile-testing machine. The test was started under a cross-head speed of 3 mm/min and a strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$. To obtain the strain rate sensitivity, m , the cross head speed was changed to 0.3 mm/min, with a strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$, repeatedly until fracture. Generally, the solder used in the fine pitch joint is deformed under the thermal cycle conditions at the strain rate ranging from approximately 10^{-6} to 10^{-5} s^{-1} .⁹⁾ Since, as described above, the ranking of the steady state creep rate at the same stress in several lead-free solders is relatively stable in the creep rate ranging from 10^{-6} to 10^{-3} s^{-1} ,^{10,11)} the strain rate range from 10^{-4} to 10^{-3} s^{-1} was selected in this study. Such a strain rate range is general one used in the conventional tensile test, and thus the test is relatively quick to perform.

Figure 1 shows an example of a load-strain curve of the Sn–3Ag–0.5Cu solder. At certain strain, the cross-head speed is lowered; then the speed is reversed to the former rate after the flow stress reached almost steady state. The flow stresses P_A and P'_A , at the same strain, are measured for the evaluation of m . The m was calculated from the following equation:¹³⁾

$$m = \{\log(P_1/P_2)\}/\{\log(v_1/v_2)\}$$

where P_1 is the load under a cross-head speed, $v_1 = 3 \text{ mm/min}$ (strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$) and P_2 is the load under a cross-head speed, $v_2 = 0.3 \text{ mm/min}$ (strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$).

When the obtained m is plotted against the strain where the m is measured, the plots usually shows a linear relation ship.^{13–15)} Thus, the m at strain zero, m_0 , is obtained by

Fig. 1 Example of load-strain curve (Sn–3Ag–0.5Cu).

extrapolating the relation between m and the strain where the m is measured.¹³⁾ Moreover, the gradient k is also investigated as the slope of the fitting line.¹³⁾

3. Results and Discussion

3.1 Microstructures

Figure 2 shows the back-scattered electron images of initial microstructures of the solders of as cast condition. The bright gray areas was identified as β -Sn phases by EPMA mapping analysis in all the solders. Those phases are primary Sn phases. In the Sn–3.5Ag eutectic alloy, Sn and Ag are detected in the dark gray area by EPMA mapping analysis. That area is inferred to be eutectic microstructure of Sn and Ag_3Sn by the EPMA analysis result, the Sn–Ag phase diagram¹⁷⁾ and the previous report by Suganuma *et al.*¹⁸⁾ Similar microstructures were observed in Sn–3Ag–0.5Cu, Sn–2.8Ag–0.3Cu and Sn–3.5Ag–0.7Cu. Cu is detected in eutectic microstructures in those solders by EPMA mapping analysis. The solid solubility of Cu in Sn is at most 0.01 at%,¹⁹⁾ and thus most of Cu are existed as Cu_6Sn_5 in the eutectic microstructure.²⁰⁾ For ternary Sn–Ag–Cu alloys, the ternary eutectic composition has been reported to be approximately Sn–3.5Ag–0.75Cu.^{21–23)} Therefore, the microstructures of the hypoeutectic and the eutectic Sn–Ag–Cu ternary alloys were similar to that of the binary Sn–Ag eutectic alloy in this study.

In the hypereutectic Sn–Ag–Cu alloys, Sn–3.8Ag–0.7Cu and Sn–4Ag–0.9Cu, the primary Sn phases are refined compared to those of the hypoeutectic ones. The refinement of the primary Sn phases develops with increasing the content of Ag and Cu. Moreover, those phases have dendritic structures.

Figure 3 shows back-scattered electron micrographs of the solders after aging at 120°C for 7 d. Coarsening of β -Sn phases and coarsening of Ag_3Sn phases in the eutectic microstructure are observed in all the solders. Ag_3Sn phases coarsen approximately to a several-micron meters. Coarsening of Cu–Sn compounds is also observed in Sn–3.5Ag–0.7Cu, Sn–3.8Ag–0.7Cu and Sn–4Ag–0.9Cu. In those solders, dark-gray and bright-gray phases in the eutectic microstructures correspond to Cu–Sn compounds and Ag_3Sn , respectively. Moreover, the growth of Ag_3Sn phases along to the β -Sn phases are observed in the Sn–3.8Ag–0.7Cu and the Sn–4Ag–0.9Cu solders.

Similar microstructures shown in Fig. 3 were also observed in the solders after aging at 120°C for 21 d. Therefore, the influence of aging time on the microstructures is negligible in the aging time ranging from 7 to 21 d investigated.

3.2 Strain rate sensitivity index of lead-free solders

Figure 4 shows the relationship between m and strain in specimens without aging, as cast condition. The m values of the Sn–Ag and the Sn–Ag–Cu lead-free solders are very lower than that of the Sn–37Pb solder. The gradients k of the lead-free solders are also lower than that of Sn–37Pb. This means the lead-free solders investigated, which have low m_0 and k , have better resistance to thermal fatigue than the Sn–37Pb solder.¹³⁾

Fig. 2 Microstructures of lead-free solders without aging.

Fig. 3 Microstructures of lead-free solders after aging at 120°C for 7 d.

In the lead-free solders, Sn–3.5Ag and Sn–3Ag–0.5Cu have the lowest m_0 . For m_0 , the value increases with deviating the composition of the solder from the ternary Sn–Ag–Cu eutectic composition. Higher m_0 means the microstructure is finer,¹³⁾ and this corresponds to the microstructural observation results shown in Fig. 2. For k , Sn–3Ag–0.5Cu has the highest value and it was found that k decreases with increasing the content of Ag and Cu in the

solder. Since low k means a constant low creep rate irrespective of strain,¹³⁾ the solder with a hypereutectic composition is expected to have the excellent thermal fatigue resistance.

3.3 Influence of aging on strain rate sensitivities

Figure 5 shows the influence of the aging treatment on m_0 . In the Sn–Pb eutectic solder, m_0 gradually decreases with

(a)

(b)

Fig. 4 Relationship between strain rate sensitivity and strain (without aging) (a) comparing lead-free solders with Sn–37Pb solder, (b) lead-free solders.

Fig. 5 Influence of aging treatment on strain rate sensitivity.

increasing aging time. For this tendency, it was already reported the decrease in m_0 with aging corresponds to the microstructure coarsening.¹³⁾ Although m_0 of the Sn–37Pb solder decreases with increasing aging time, it is much higher than those of the lead-free solders under aging conditions investigated.

Fig. 6 Relationship between m_0 and k values.

On the contrary, m_0 initially increases until aging time of 7 d and afterward, it saturates in the lead-free solders. Since the microstructures are relatively stable in aging time ranging from 7 to 21 d, the saturation of m_0 would be observed in the lead-free solders. The increase in m_0 usually means refining of the microstructure.¹³⁾ Coarsening of β -Sn, however, were observed in all the lead-free solders after aging treatment (refer to Fig. 3). In the case of the lead-free solders, the growth of Ag_3Sn and Cu–Sn compounds occurs under aging conditions. Such microstructures, in which the compounds grow up to several-micron meters, have lower creep resistance due to the degradation of dispersion hardening. Thus, the lead-free solders become to have a high creep rate by aging and the strain rate sensitivities are higher, resulting in the increases of m_0 .

Figure 6 indicates the relationship between m_0 and k . Each data shown in Fig. 6 is the average value of measurements which conducted with three specimens for each solder. In the lead-free solders, k decreases following the aging treatment. The influence of aging time on the decrease of k is negligible under aging conditions investigated. That is, as described above, because the microstructures are relatively stable in aging time ranging from 7 to 21 d. In the solders except the hypereutectic solders, Sn–3.8Ag–0.7Cu and Sn–4Ag–0.9Cu, the decreases of k are relatively large. This tendency means the creep rate becomes constant at lower level irrespective of strain, and consequently the solders with low k are expected to have better thermal fatigue resistance. However, m_0 values of those solders are higher than those without aging. Since the solder with high m_0 has a high creep rate, the excellent thermal fatigue resistance would not be expected for such a solder. Therefore, the aging treatment will degrade the thermal fatigue life for Sn–3.5Ag, Sn–3Ag–0.5Cu, Sn–2.8Ag–0.3Cu and Sn–3.5Ag–0.7Cu. On the contrary, the changes of m_0 and k in the hypereutectic solders are relatively smaller than those of other lead-free solders. Although the decrease of k and the increase of m_0 are also observed in the hypereutectic solders, those changes are a little. The influence of aging on the thermal fatigue resistance seems to be a little in Sn–3.8Ag–0.7Cu and Sn–4Ag–0.9Cu.

3.4 Estimation of thermal fatigue resistance of the solder with m_0 and k

Since m_0 and k correspond to the creep resistance of the solder, it is feasible to evaluate the thermal fatigue resistance of the solder using m_0 and k . It has been reported that Sn–36.7Pb–0.7Ag–0.6Sb with low m_0 and k has more excellent thermal fatigue life than Sn–37Pb with higher m_0 and k by Takemoto *et al.*²⁴⁾ Moreover, the study on ranking thermal fatigue lives of the several solders has been conducted with m_0 , k and the evaluation data of thermal fatigue lives.¹³⁾ Those previous studies have reported the estimation feasibility of the thermal fatigue resistance of the solder with m_0 and k . Analogous investigation was attempted in this study. Since microstructure coarsening has been observed in the solder joints under thermal cycle²⁵⁾ and heat exposure conditions,^{26,27)} m_0 and k values after aging are effective to investigate the thermal fatigue resistance of the solder. Thus, m_0 and k values after aging at 120°C for 21 d were used for the analysis in this study.

From the data after aging at 120°C for 21 d shown in Fig. 6, it is found that the lead-free solders have lower m_0 and k compared to the Sn–37Pb solder. It means that the lead-free solders have more excellent thermal fatigue resistances than the Sn–37Pb solder under thermal cycle conditions which maximum temperature is at most 120°C. For the lead-free solders, the solders with lower k have higher m_0 and the ones with lower m_0 have higher k . Therefore, it is not easy to rank in detail the lead-free solders in thermal fatigue resistance with m_0 and k , different from making a comparison the lead-free solders with the Sn–37Pb solder. Among the lead-free solders investigated, the difference of the thermal fatigue resistance seems to be a little compared to that of the lead-free solder and the Sn–37Pb solder.

Considering from the data of m_0 and k after aging at 120°C for 21 d shown in Fig. 6, Sn–3.5Ag and Sn–3Ag–0.5Cu would have the excellent thermal fatigue resistance at low strain because the solders have lower m_0 , which means higher creep resistance. On the contrary, Sn–2.8Ag–0.3Cu has the highest m_0 among the lead-free solders investigated. The Sn–2.8Ag–0.3Cu solder has the hypoeutectic composition and the eutectic microstructure is less than other lead-free solders. Thus, coarsening of Ag₃Sn by the aging treatment causes a decrease of a deformation resistance. At high temperatures, the deformation resistance will decrease in the solder. The Sn–2.8Ag–0.3Cu solder, however, would have the excellent thermal fatigue resistance at high strain due to low k . For Sn–3.5Ag–0.7Cu, Sn–3.8Ag–0.7Cu and Sn–4Ag–0.9Cu, they would have intermediate thermal fatigue properties between those of Sn–3.5Ag and Sn–3Ag–0.5Cu and that of Sn–2.8Ag–0.3Cu, compared to m_0 and k values.

Figure 7 shows the relationship of m_0 , k , which are investigated with specimens after aging at 120°C for 21 d, and the thermal fatigue life in Sn–37Pb, Sn–3.5Ag and Sn–3.5Ag–0.7Cu.⁶⁾ For m_0 and k , the reciprocals are shown in the figure because the solder with low m_0 and k have the excellent thermal fatigue life. Each value shown in Fig. 7 is normalized by the datum of the Sn–37Pb solder. For the thermal fatigue life, the data of CSP solder joints⁶⁾ were used. Since there were no data of the thermal fatigue lives

Fig. 7 Comparison reciprocals of m_0 and k investigated after aging at 120°C for 21 d with thermal fatigue lives (N_f) of solders.⁶⁾

evaluated by the same thermal cycle test using fine pitch solder joints with other lead-free solders, the comparison could not be conducted with other lead-free solders. Although the reciprocals of m_0 in Sn–3.5Ag and Sn–3.5Ag–0.7Cu are analogous values, the reciprocal of k in Sn–3.5Ag–0.7Cu is larger than that in Sn–3.5Ag. The change of the thermal fatigue life is good consistent with that of the reciprocal of k .

S. K. Kang *et al.*²⁸⁾ have reported that the hypereutectic Sn–Ag–Cu solder is superior to the hypoeutectic Sn–Ag–Cu solders in the thermal fatigue life as the result of thermal cycle test with BGA solder joints. Since the hypereutectic Sn–Ag–Cu solders have lower m_0 compared to the hypoeutectic ones (refer to Fig. 6), it was clarified that the solder with low m_0 after aging has the excellent thermal fatigue life in the Sn–Ag–Cu solders.

In this way, m_0 and k values seem to be valid to estimate the thermal fatigue resistance of the solder. Generally, the thermal fatigue resistance of the solder depends on solder composition,^{6,24,25)} thermal cycle conditions,^{6,16,29)} design of joints⁶⁾ and so on. Such specifications alter a plastic strain range applied in the solder joint, and consequently the thermal fatigue resistance of the solder changes according to modified Coffin–Manson equation.²⁹⁾ Therefore, it is not easy to estimate the thermal fatigue resistance of the solder in detail using only m_0 and k under various thermal cycle conditions. For the prediction method of the fatigue life of the solder joint, the method using rupture ductility has been already suggested,^{8,30)} and thus further investigation seems to be required including the comparison with such a method. However, m_0 and k are examined easily by a simple mechanical test and those correspond to the creep resistance of the solder. In particular, when the ranking of the steady state creep rate of the solder is relatively stable in the large creep rate range in the same alloy system, m_0 and k values are valid to estimate the thermal fatigue resistance of the solder. In such conditions, m_0 and k are feasible to become effective indexes developing a new lead-free solder with the excellent thermal fatigue resistance.

4. Conclusions

The strain rate sensitivities of several Sn–Ag and Sn–Ag–Cu lead-free solders were investigated by the strain rate change test. Moreover, the estimations of the thermal fatigue resistances of the solders were conducted using the strain rate sensitivities investigated. The results obtained are summarized as follows.

- (1) The m_0 and k values of the Sn–Ag and the Sn–Ag–Cu lead-free solders are lower than those of the Sn–Pb eutectic solder. It means the lead-free solders have the excellent creep resistances compared to the Sn–Pb eutectic solder.
- (2) The m_0 of the lead-free solder increases with deviating the composition of the solder from the ternary Sn–Ag–Cu eutectic composition. The k decreases when the contents of Ag and Cu increase and Ag_3Sn and Cu–Sn compounds grow in the eutectic microstructure.
- (3) The aging treatment at 120°C increases m_0 a little and decreases k in the lead-free solders. The influence of aging on the strain rate sensitivity and the microstructure of the lead-free solder are negligible in the aging time ranging from 7 to 21 d.
- (4) The ranking of the reciprocals of m_0 and k in the solders is relatively in good agreement with that of the thermal fatigue lives evaluated by the thermal cycle test with the solder ball joints.
- (5) The m_0 and k have the feasibility to become effective indexes developing a new lead-free solder with the excellent thermal fatigue resistance.

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