Estimation of the Thermal Fatigue Resistance and Creep Properties of the Co/Ni-Bearing SAC305 Lead-Free Solders by the Strain Rate Change Tensile Test

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In the present paper, in order to evaluate the creep properties of Co/Ni-bearing SAC305 lead-free alloys, the strain rate change tensile (SRCT) test was conducted. Two parameters, m_0 and k , obtained from the SRCT test could be applied to estimate the thermal fatigue resistance and the creep properties for lead-free solder alloys. The m_0 value implied the material's creep resistance, and the k value revealed the stability of the creep resistance during the whole tensile test process. The Co/Ni-bearing SAC305 solder alloys showed much lower m_0 and k values than the conventional Sn37Pb solders which indicated that the Co/Ni-bearing SAC305 solder alloys possessed of excellent thermal fatigue resistance and higher creep resistance. For the lead free solder alloys, high temperature aging led to microstructural coarsening. After aging, the m_0 value increased meanwhile the k value decreased for the Co/Ni-bearing SAC305 solders. Furthermore, the two parameters showed a converse change tendency between the conventional Sn37Pb and Co/Ni-bearing SAC305 solders after high temperature aging, which implied that they have different creep mechanism. It can be deduced that, for the conventional Sn37Pb solder, the grain boundary slide is the dominant creep mechanism, whereas the Co/Ni-bearing SAC305 solders showed a dislocation glide/climb creep mode. [doi:10.2320/matertrans.MF200803]

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1. Introduction

With the miniaturization of the electronic products, better mechanical performance and higher reliability are required for the solder materials. Even during service conditions, the solder alloys are subjected to creep and fatigue deformation duo to their low melting point. Accordingly, the anti-creep ability and the thermal fatigue resistance of the solder alloys are much concerned.¹⁻³⁾ Since the test parameters used in the traditional thermal fatigue properties test are various, the results are not universal, in addition, the test cost is high and the test period is long.⁴⁾ The SRCT test is a simple mechanical test method developed by professor T. Takemoto *et al.*⁵⁾ In this test method, the strain rate is alternated between two given values at given time intervals. The strain rate sensitivities index (SRSI), *i.e.* the m value, and its gradient k can be conveniently calculated using the load-strain curve obtained from the SRCT test. The m_0 value was accordant with the creep resistance while the k value implied the stability of the creep resistance during the deformation process. Because the relative low melting temperature, even in the room temperature the homologous temperature of Sn-based solders is over 0.6, which indicates that the time-dependent creep deformation is dominant during the thermal fatigue damage for the Sn-based lead free solder alloys. So, the two parameters obtained from the SRCT test, m_0 and k could be applied to evaluate the resistance of thermal fatigue and the creep properties of lead free solder alloys. T. Takemoto and I. Shoji *et al.* used the two parameters to evaluate the thermal fatigue resistance for several lead-free solder alloys and obtained satisfactory results.⁶⁻¹⁰⁾

It has been proved that the minor element addition is a simple and effective way of modifying the microstructural and mechanical properties of lead free solder alloys. Owing to the relatively low cost and toxicity, the Co and Ni have

driven more concerns in the recent years.^{11,12)} In the present work, it was noticed that the m_0 and k values showed different change tendency for the SAC305 solder alloys and the traditional SnPb solders before and after high temperature aging. The two parameters, m_0 and k and their change tendency were used to analyze the creep properties, as well as the estimation of the thermal fatigue resistance for the Co/Ni-bearing SAC305 solders.

2. Experimental Procedures

Four sorts of lead-free solders, Sn-3.0 mass%Ag-0.5 mass%Cu, Sn-3.0 mass%Ag-0.5 mass%Cu-0.2 mass%Co, Sn-3.0 mass%Ag-0.5 mass%Cu-0.1 mass%Ni, and Sn-3.0 mass%Ag-0.5 mass%Cu-0.2 mass%Co-0.1 mass%Ni (Hereafter, these alloys were designated as SAC305, SAC305-0.2Co, SAC305-0.1Ni, and SAC305-0.2Co.1Ni, respectively.) were prepared for the experiments. The solders were remelted at about 317°C in a graphite crucible and then cast into a dumbbell-shaped steel moulds. The specification of the tensile test specimens is according to JIS Z 6198-2.

The SRCT test was conducted at room temperature using the Instron tensile test machine. Figure 1(a) depicted a typical test load-strain profile obtained from SRCT test. There are two strain rates applied at intervals in the tensile procedure. The high strain rate is $\dot{\epsilon}_1 = 1 \times 10^{-3} \text{ s}^{-1}$ (the corresponding cross-head speed is 3 mm/min.) and the low strain rate is $\dot{\epsilon}_2 = 1 \times 10^{-4} \text{ s}^{-1}$ (the corresponding cross-head speed is 0.3 mm/min.). The standing intervals are 50 s for $\dot{\epsilon}_1$ and 30 s for $\dot{\epsilon}_2$, respectively. 30 s is enough to achieve quasic-steady state for $\dot{\epsilon}_2$.

Figure 1(b) illustrated the procedure how to calculate the SRSI, *i.e.* the m value, using a typical load-strain curve obtained from the SRCT test. The m was defined by the following power-law equation:

Fig. 1 Schematic diagram of the strain rate change tensile test method (a) the typical stress-strain test plot, and (b) the obtained m - ε plot.

$$\sigma = A\dot{\varepsilon}^m \quad (1)$$

Here, σ denotes the tensile stress, $\dot{\varepsilon}$ the strain rate, A material constant, and m is the SRSI.

So, the m value at certain strain value could be calculated by the following equation:

$$m = \frac{\log(\sigma_1/\sigma_2)}{\log(\dot{\varepsilon}_1/\dot{\varepsilon}_2)} = \frac{\log(P_1/P_2)}{\log(\dot{\varepsilon}_1/\dot{\varepsilon}_2)} \quad (2)$$

Where P_1 is the load force under the high strain rate $\dot{\varepsilon}_1 = 1 \times 10^{-3} \text{ s}^{-1}$, and P_2 is the load force under the low strain rate $\dot{\varepsilon}_2 = 1 \times 10^{-4} \text{ s}^{-1}$. The P_2 could be directly obtained from the stress-strain profile in Fig. 1(a). Meanwhile, the P_1 could be calculated by extrapolating the two contextual profile segments under the high strain rate $\dot{\varepsilon}_1$ at the same strain point, ε_A . The plot between m and strain ε showed an almost linear relationship within the range of present experiment as shown in Fig. 1(b). The m at strain rate zero, *i.e.* m_0 , can be obtained by extrapolating the m - ε plot to the vertical axial. Moreover, the gradient k of this fitting line is also obtained.

In order to investigate the effect of high temperature storage on the thermal fatigue resistance and the creep properties, the solders were aged in an oil bath at 150°C for 168 h. The composition of Co-Sn intermetallics was identified by electron probe micro-analysis (EPMA).

3. Results and Discussion

3.1 Strain rate sensitivity index m and its gradient k

As depicted in the experiment procedures, the strain rate sensitivity index, m , can be calculated from the designed SRCT test. According to the test results, the m value is not a constant during the whole tensile process. Generally, with the increase of the elongation deformation, the m value becomes larger, that means the m is strain-dependent. Obviously, the m_0 obtained from the SRCT test eliminated the effect of strain, *i.e.*, strain-independent. The m value is an effective

Table 1 The quantitative analysis results of Co/Ni-Sn intermetallics in Co/Ni-bearing SAC305 solders. (unit: at%).

	Sn	Cu	Ag	Co	Ni	Phase
SAC305-0.2Co	66.4	1.2	0	32.6	—	CoSn ₂
SAC305-0.1Ni	47.1	39.8	0.3	—	12.8	(Cu,Ni) ₆ Sn ₅
SAC305-0.2Co0.1Ni	65.7	0.9	0	23.2	10.2	(Co,Ni)Sn ₂

parameter to analyze the materials creep properties. Generally, the lower the m value is, the higher the creep resistance is. The dislocation climb (DC) creep showed a much higher m value than that of the grain boundary slide (GBS) creep pattern.^{13,14}

Furthermore, the gradient k reveals the stability of the m value when the deformation increasing continuously during the tensile test process. In theory, zero k means the m is a constant during the whole tensile process before fracture, which implied the creep resistance is not deteriorated during the whole tensile test process. On the contrary, a plus k value indicated the decrease of the creep resistance with the increase of the deformation. So, the gradient k is a good index to evaluate the stability of the creep process.

3.2 The microstructural characterization of Co/Ni-bearing SAC305 solder alloys

It has been widely known that several phases co-exist in the SAC305 solder alloy, namely, the β -Sn, Ag₃Sn and Cu₆Sn₅. The Co/Ni additives induced the formation of some new compound phases and microstructural modification for the SAC305 solder alloy. Figure 2 illustrated that some new intermetallics were generated after the addition of minor Co/Ni. The quantitative analysis results by EPMA indicated that the new intermetallics were CoSn₂ and (Co,Ni)Sn₂ for the SAC305-0.2Co and SAC305-0.2Co0.1Ni alloys, respectively. For the SAC305-0.1Ni alloy, the new intermetallics was (Cu,Ni)₆Sn₅, as shown in Table 1.

Figure 3 shows the microstructure after high temperature aging at 150°C for 168 h. It is clear that substantial microstructural coarsening had occurred. For the rapid diffusion of Ag and Cu in the Sn matrix under high temperature, the superfine Ag₃Sn and Cu₆Sn₅ particles inside the eutectic network grew and coalesced rapidly.

Generally, the mechanical properties are correlated with the microstructures, the formation of the new intermetallics after the addition of Co/Ni and the microstructural coarsening after high temperature storage will change the thermal fatigue resistance, as well as the creep properties.

3.3 The effects of Co and Ni additives on the m_0 and k

The relationship between the m value and the strain ε was illustrated in Fig. 4. The discrete m value data are fitted by least-squares method and the intercept of the fitting lines on the vertical axial is the corresponding m_0 value. It is clear that the SAC305 solder has the lowest m_0 value, as well as the lowest k value. The sole addition of Co or Ni showed similar effect on the m_0 and k . Both of them increased the m_0 slightly, but scarcely affected the k value. However, the dual addition of Co-Ni raised the k value significantly.

Figure 5 depicted the location of the four tested lead-free solders and the conventional Sn37Pb solder in the m_0 - k

Fig. 2 The microstructure in the as-cast condition of (a) SAC305, (b) SAC305-0.2Co, (c) SAC305-0.1Ni, and (d) SAC305-0.2Co0.1Ni.

Fig. 3 The microstructure after 168 h aging at 150°C of (a) SAC305, (b) SAC305-0.2Co, (c) SAC305-0.1Ni, and (d) SAC305-0.2Co0.1Ni.

coordinate plane. The open symbols and the correlated solid symbols denoted the as-cast condition and after aging condition for the same alloy, respectively. Compared with the conventional Sn37Pb eutectic solder, the four sorts of lead-free solders showed much lower m_0 and k values. In addition, it is noticed that the SAC305-0.2Co0.1Ni solder showed a larger k value than the other lead-free solder alloys.

High temperature aging treatment has notably effect on the m_0 and k value for the solder alloys. For the lead-free solders, the m_0 value increased while the k value decreased. For the Sn37Pb solder, the effects were just contrary, the m_0 decreased meanwhile the k increased after high temperature aging treatment.

3.4 Estimation of the thermal fatigue resistance

Based on the earlier experiment results conducted by T. Takemoto *et al.*, the m_0 and k are the effective indices for the thermal fatigue resistance rank of several solder alloys, that is, the lower the m_0 and k values are, the higher the thermal fatigue resistance rank is. Compared to the SAC305 baseline, the Co/Ni additives just changed the m_0 and k values slightly. Accordingly, it can be deduced that the effect of Co/Ni additives on the thermal resistance is very slight. Compared to the Sn37Pb solder, all the four sorts of lead-free solder alloys should possess of excellent thermal fatigue resistance due to their relatively lower m_0 and k value. After aging at 150°C for 168 h, the parameters were still much lower than

Fig. 4 The relationship between the m and the strain ε .

Fig. 5 The location of several solders in the m_0 - k coordinate plane.

that of the Sn37Pb, which indicated that the SAC305-based lead-free solder alloys possessed excellent thermal fatigue resistance even after high temperature storage.

3.5 Evaluation of the creep properties

As shown in Fig. 5, the Co/Ni-bearing SAC305 solder alloys showed much higher m_0 value than the conventional Sn37Pb solders both in the as-cast condition and after high temperature aging. Such a result indicated that the Co/Ni-bearing SAC305 solder alloys possessed of better creep resistance than the Sn37Pb solder.

After aging, the m_0 and k value of lead-free solders and the conventional Sn37Pb solders showed different change tendency, which implied different creep mechanism for them. The conventional Sn37Pb solder consists of two constituent phase, *i.e.*, the alternated tin-rich phase and lead-rich phase. During high temperature aging, the average grain size both of the tin-rich and the lead-rich phases increased. Meanwhile, the m_0 also decreased, as shown in Fig. 5. In other words, the creep resistance increased after aging for the conventional Sn37Pb solder. It is accordant with the grain boundary slide (GBS) type creep mechanism, that is, the larger the average grain size, the higher the creep resistance is. Accordingly, it

can be conclude that GBS is the dominant creep mechanism for the Sn37Pb solders.

The SAC-based lead-free solders consist typically of β -Sn and Ag_3Sn - Cu_6Sn_5 - β -Sn ternary eutectic network. As-cast condition, the superfine Ag_3Sn and Cu_6Sn_5 intermetallics dispersed in the eutectic network uniformly, as shown in Fig. 2. These hard and well-dispersed intermetallics acted as the discrete obstacles to block the movement of the dislocation, which is the main reason that the SAC-based lead-free solders presented better creep resistance than the Sn37Pb solders under the room temperature. During high temperature aging, the superfine Ag_3Sn and Cu_6Sn_5 particles in the eutectic network grew and coalesced into together significantly. So, the microstructural coarsening of the lead-free solder alloys after aging attributed to the coalescence and the amount decrease of these intermetallics instead of the growing of the β -Sn dendrites. On the other hand, the increase of m_0 after aging implied that the anti-creep ability decreased, which is very different to that of the Sn37Pb solder. A reasonable explanation for such a result is the growing and coalescence of the Ag_3Sn and Cu_6Sn_5 intermetallics reduced the obstacles for the dislocation movement after aging treatment. In addition, the m_0 value of the lead-free solder alloys is much lower than that of the conventional Sn37Pb solder alloy. It is accordant with the dislocation glide/climb (DC) type creep, that is, the lower the amount of the obstacle particles, the lower the creep resistance is. Accordingly, it can be concluded that the dislocation glide/climb is the dominant creep mechanism for the SAC-based lead-free solders.

Compared with the SAC305 baseline, the SAC305-0.2Co0.1Ni alloy showed a higher k value. A higher k value indicated a lower stability of the creep resistance during deformation. It could be explained by the different of the microstructure. As shown in Fig. 2(d), the CoSn_2 intermetallics in SAC305-0.2Co0.1Ni solder are very thin and sharp, in addition, these intermetallics are harder and more brittle than the Sn matrix. Along with the increase of the deformation during tensile test, the micro-cracks are apt to initiate along these CoSn_2 intermetallics, as shown in Fig. 6. The easily initiating of the micro-cracks along the sharp and brittle CoSn_2 intermetallics is the main reason for the degradation of the creep resistance.

Fig. 6 The cross-sectional micrographs near the fracture surface after tensile test of the SAC305-0.2Co0.1Ni.

4. Conclusions

To evaluate the effect of the Co/Ni additives on the thermal fatigue resistance and the creep properties of the SAC305 lead-free solder alloys, a strain rate change tensile test method was conducted. The results are summarized as follows.

(1) The m_0 and k values of the Co/Ni-bearing SAC305 lead-free solders are lower than that of the Sn37Pb solder. It indicates that the Co/Ni-bearing SAC305 solders showed a better thermal fatigue resistance than the conventional Sn37Pb solder.

(2) The Co/Ni-bearing SAC305 solder alloys showed much lower m_0 value than the conventional Sn37Pb solders both in the as-cast condition and after high temperature aging. Such a result indicated that the Co/Ni-bearing SAC305 solder alloys possessed of better creep resistance than the Sn37Pb solder.

(3) After high temperature aging, the contrary change tendency of the m_0 and k values for the SAC305 lead-free solders and the conventional Sn37Pb solders revealed that they have different creep mechanism. It can be deduced that the GBS is the dominant creep mechanism for the Sn37Pb eutectic solder, meanwhile, the SAC305 lead-free solders showed DC type creep mode.

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