Creep property of composite solders reinforced by nano-sized particles

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Abstract In the present work the creep properties of Sn37Pb and Sn0.7Cu based composite solders with nano-sized metallic Cu, Ag and nano-sized oxide Al_2O_3, TiO_2 reinforcement particles have been studied. First, a series of volume percentages of reinforcements were selected for optimizing the content of particles. Then, the composite solder with optimum volume fraction of the reinforcement particles, corresponding to maximum creep rupture life, is selected for investigating the effect of applied stress level and test temperature on creep rupture life of the composite solder joints. In the creep rupture life test, small single-lap tensile-shear joints were adopted. The results indicate that all the composite solders have improved creep resistance, comparing to the eutectic Sn37Pb solder and the Sn0.7Cu lead-free solder. The creep rupture life of the composite solder joints is first increased with the increase in the volume fraction of reinforcement in the composite solders. Then, the creep rupture life is decreased, as the reinforcement content exceeds a certain value. The creep rupture life of the solder joints is decreased with the increase of applied stress and testing temperature. Moreover, the reinforced efficiency of nano-sized Ag particles is the best in all the tested nano-sized reinforcements for the Sn37Pb based and Sn0.7Cu based composite solders, when the particles contents are in their own optimum content.

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1 Introduction

Solders under severe service environment are required to have excellent mechanical properties and high reliability. In automobile, solders used in the proximity of the engine experience a temperature excursion between –40 °C and 200 °C in winter. The solders are subjected to a thermal stress induced by the temperature cycling as well as a mechanical stress induced by the vibration from the engine and the occasional shocks or impact from the road condition during driving. In lightguide ocean cables, where the optical fiber is attached with solder, the precise alignment of each component in a plane perpendicular to the fiber axis requires that the solders have excellent creep resistance and dimensional stability. Moreover, in the fine pitch solder bump interconnect, the bump current density may approach 10^4 A/cm^2 level, where electro migration becomes a significant reliability issue. Thus, the solders with enhanced electrical and mechanical properties are required. These severe service environments make solders experience microstructure evolution, creep deformation, and thermo-mechanical fatigue damage. In order to improve the mechanical properties of solders, especially creep and thermo-mechanical fatigue resistance, composite solders have been developed recently.

Composite solders generally contain fine second-phase reinforcing particles dispersed uniformly in the solder matrix. In general there are two possible ways to manufacture the composite solders. They are the in-site reaction method and the mechanical mixing method. The particles obstruct movement of dislocation and pin grain boundaries so as to prevent the solder matrix from plastic deformation. Early efforts demonstrated that certain composite solders were with improved mechanical properties. Marshall et al. [1] found superior mechanical properties on conventional

Sn40Pb solders by introducing Cu_6Sn_5 intermetallic compounds (IMC). In addition, Ag, Ni, and Cu also have been used as reinforcing particles in the Sn40Pb based composite solder. In order to improve the service temperature capability of lead-free solders, Guo et al. [2, 3] studied the enhanced creep resistance of Sn3.5Ag solder by introducing micro-sized Ag, Ni, or Cu particles. It was found that Ni-particles reinforced composite solder joints were about five times more creep resistant than composite solder joints reinforced by Cu-particles, and about 30 times better than Sn3.5Ag solder and composite solder reinforced with Ag-particles. Recently, metallic Co particles were mechanically mixed with Sn3.5Ag solder to form composite solder [4].

With the development of nano technology, various nano-sized particles were selected as reinforcements in manufacturing composite solders. Mavoori and Jin [5] studied the Sn37Pb composite solders with the reinforcement of nano-sized oxide particles. The creep test was carried out in a constant compressive load and the composite solder was not subjected to reflow process. The result showed that the composite solders exhibited significantly enhanced creep resistance combined with increased strength. Moreover, the nano-sized metallic particles (Ni, Cu, and Mo) reinforced composite solders were developed, and the results showed that the mechanical properties, such as microhardness and creep resistance were increased [6–8]. Recently, multi-walled carbon nanotubes were chosen as the reinforced particles to synthesize composite solders [9]. The investigation showed that the mechanical properties of the composite solders showed a certain improvement in comparison to the un-reinforced SnAgCu solder. Moreover, a SnAg based composite solder with nano-structured polyhedral oligomeric silsesquioxanes (POSS) particles was investigated recently [10]. The POSS particles were surface active, and inert reinforcement. The initial results showed the improvement of shear strength for the POSS particles reinforced Sn3.5Ag solder joints [11].

As described above, many investigations have been carried out on the particles reinforced composite solders. Most of the published results are focused on effect of reinforcement particles on the conventional mechanical properties such as tensile strength, shear strength, or microhardness of composite solders. It is known that fine-grained microstructure may be beneficial for mechanical strength, but it may not be realistic for creep resistance, since creep deformation at the service temperature is by grain boundary sliding.

Although some results on effect of nano-sized reinforcements on mechanical strength and creep properties were given, there is lack of comparison of strength properties for composite solders between different reinforcements under the same test condition. Therefore, the present work is focused to investigate and compare the effect of different nano-sized reinforcements including metallic particles and oxide particles on the creep properties of Sn37Pb and Sn0.7Cu composite solders.

2 Experimental

2.1 Preparation of composite solders

The composite solders were prepared by blending pre-weighed matrix solder powders with different volume percentages of metallic reinforcement particles. The matrix materials used for this study were Sn37Pb and Sn0.7Cu powders, respectively with an average diameter of about 43 μm. Four kinds of nano-sized reinforced particles were used. Two kinds of metallic particles used as the reinforcements were pure Cu nanopowder having an average size of 50 nm, and pure Ag nanopowder having an average size of 60 nm. The two kinds of nanopowders were purchased from the Nano Zunye Ltd, Shenzhen. Moreover, two kinds of oxide particles used as the reinforcements were Al_2O_3 nanopowders having an average size of 70 nm and TiO_2 nanopowders having an average size of 80 nm. Both the oxide nanopowders were coated by a aluminium stearate film. The Al_2O_3 and TiO_2 nanopowders were purchased from the Beijing University of Chemical Engineering and the Panzhihua, Sichuan, respectively. A series of volume percentages were selected for optimizing the content of the reinforcement particles. The composite solders were blended very well prior to addition of a mildly activated RMA flux. The weight ratio of the composite solder powder and the flux was 7:1. The resultant composite solders were mechanically stirred for 30 min so as to ensure a homogeneous distribution of the reinforcement particles in the solder matrix. Finally, the pastes were immediately sealed and preserved in a refrigerator at –10 °C to delay degradation of the pastes.

2.2 Tensile shear test

The tensile shear test was conducted for investigating effect of reinforcement particles on mechanical strength of composite solder joints. The geometry of the specimens for tensile shear test is shown in Fig. 1. The composite solder paste was sandwiched between the two copper sheets. Two copper wires, which had diameter of 0.1 mm were used for fixing up the gap of the joint. The solder joints were heated to the peak temperature of 240 °C and 280 °C for 2 min for the Sn37Pb based and Sn0.7Cu based composite solders, respectively. All the test results were an average of three
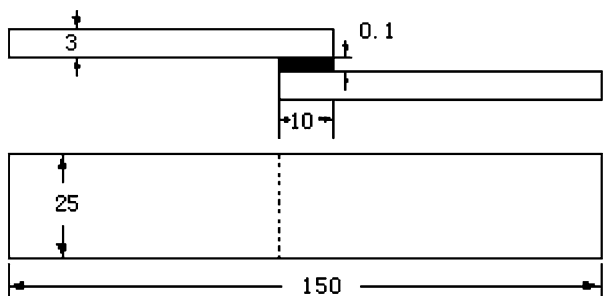
Fig. 1 Geometry of tensile shear test specimen (in mm)

specimens. The tensile test was conducted at ambient temperature with a strain rate of 5×10^{-4}.

2.3 Creep rupture life test

To simulate the size of solder joints in practical electronics assembly, a small single-lap shear joint was adopted in the creep rupture life test. The joint geometry for the test is shown in Fig. 2. Two pieces of ''dog-bone'' copper sheets were used as substrate. Thickness of the copper sheet was 0.1 mm. The copper substrates were polished using 400 grit sand paper, then the cleaning of the copper substrates with a solution of 50% HNO_3 and 50% H_2O, followed by rinsing with ethanol. A solder mask IF710 was spread on the surface of the narrow end of the copper substrate to achieve a cross-sectional area of 1 mm^2. Then the composite solder paste was sandwiched between the copper substrates. Thickness of solder layer or gap between the copper sheet was about 0.1 mm. Soldering was performed in an aluminum mold. For the Sn37Pb based composite solder, the solder joints were heated to peak temperature of 240 °C for a soak time of 2 min, then the aluminum mold was quickly removed from furnace and cooled at a rate of 50 °C/min. For the Sn0.7Cu based composite solder, the joints were heated to peak temperature of 280 °C for 2 min, then cooled quickly at a rate of 50 °C/min.

The creep rupture life test was performed at various temperatures with different dead loads. The fluctuation of temperature during the creep tests was not more than 2 °C. During testing the rupture life of specimens was recorded
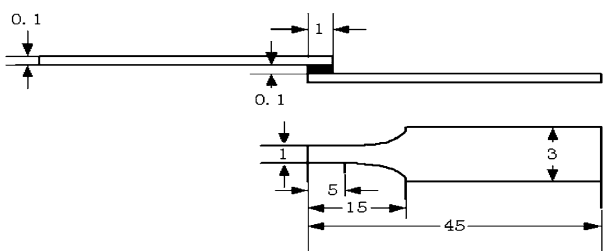

Fig. 2 Geometry of small single-lap joint (in mm)

automatically by a clock electrically connected in series with the solder joint. The test results were averaged from 12 specimens. Both Sn37Pb and Sn0.7Cu eutectic solders without reinforcement additions were also subjected to the same procedure as a comparison.

3 Results and discussion

3.1 Optimum of volume fraction of reinforcement particles

To archive an optimum creep strengthening, the composite solders with various volume fraction of reinforcement particles were examined. For the Sn37Pb based composite solders, the volume fraction of reinforcements were from 0 vol% to 4 vol% of nano-sized particles Cu, and from 0 vol% to 5 vol% of nano-sized Ag, Al_2O_3 or TiO_2. For the Sn0.7Cu based composite solders, the volume fraction of reinforcements were from 0 vol% to 3 vol% of nano-sized particles Ag, and from 0 vol% to 5 vol% of nano-sized Al_2O_3. For the Sn37Pb based composite solder joints, the creep rupture life test was carried out under a shear stress of 11.27 MPa at 25 °C. To shorten testing time, creep rupture test was carried out under the shear stress of 16.17 MPa at 75 °C for the Sn0.7Cu based composite solder joints. The test results of the creep rupture life of the composite solder joints are shown in Tables 1 and 2 respectively for the Sn37Pb based and Sn0.7Cu based composite solders.

The results indicate that the creep rupture life of the mini solder joints is first increased with the increase in the volume fraction of reinforcement in the composite solders. Then, the creep rupture life is decreased, as the reinforcement content exceeds a certain value. In other word, there exists a peak value of creep rupture life for a solder joint. For the Sn37Pb based composite solder, the optimum content of the nano-sized Cu reinforcement particles is 1 vol%, corresponding to the maximum creep rupture life. However, the optimum content of the nano-sized Ag, Al_2O_3 or TiO_2 reinforced particles is 3 vol%. For the Sn0.7Cu based composite solder, the optimum contents of the nano-sized Ag and Al_2O_3 reinforcement particles are 1 vol%, and 3 vol%, respectively, corresponding to their maximum creep rupture life. Thus, the composite solder with the optimized volume fraction of the reinforcement particles, corresponding to maximum creep rupture life, is selected for the further investigation on the effect of applied stress level and test temperature on creep rupture life of the mini solder joints.

The above results also reveal that the reinforced efficiency of nano-sized Ag particles is the best in all the four

Table 1 Effect of volume fraction of reinforcements on creep rupture life (min) for Sn37Pb based composite solder joints under shear stress of 11.27 MPa at 25° C

Nano-sized particles	Fraction of reinforcement, vol%						
	0	0.5	1	2	3	4	5
Ag	435 ± 42	–	1028 ± 140	3891 ± 357	6479 ± 495	2023 ± 211	998 ± 183
Al_2O_3	435 ± 42	–	968 ± 373	2287 ± 508	3125 ± 242	1938 ± 281	867 ± 128
TiO_2	435 ± 42	–	915 ± 135	1523 ± 504	2088 ± 211	1367 ± 387	812 ± 94
Cu	435 ± 42	1376 ± 260	2015 ± 338	1322 ± 159	1026 ± 137	410 ± 24	–

Table 2 Effect of volume fraction of reinforcements on creep rupture life (min) for Sn0.7Cu based composite solder joints under shear stress of 16.17 MPa at 75° C

Nano-sized particles	Fraction of reinforcement, vol%						
	0	0.5	1	2	3	4	5
Ag	294 ± 12	727 ± 24	1125 ± 123	845 ± 71	323 ± 22	–	–
Al_2O_3	294 ± 12	–	422 ± 20	654 ± 38	771 ± 30	675 ± 30	541 ± 14

kinds of nano-sized reinforcements for the Sn37Pb based composite solders, when the particles contents are in their own optimum content. Similarly, for the Sn0.7Cu based composite solders, the reinforced efficiency of nano-sized Ag particles is much better than that of nano-sized Al_2O_3 particles. Under the condition of testing stress and temperature, the creep rupture life of the nano-sized Ag reinforced Sn37Pb based composite solder joint is increased up to about 15 times more than that of Sn37Pb eutectic solder joint, when the nano-sized Ag particles account for 3 vol%. Moreover, the creep rupture life of the the nano-sized Ag reinforced Sn0.7Cu based composite solder joint is increased up to 4 times more than that of the Sn0.7Cu eutectic solder joint, when the nano-sized Ag particles account for 1 vol%.

Tables 3 and 4 show the tensile shear test results. For the Sn37Pb based composite solders in the test, the volume fraction of the nano-sized Ag, Cu, Al_2O_3 or TiO_2 reinforcements was from 0 vol% to 5 vol%. For the Sn0.7Cu based composite solders, the volume fractions of the reinforcements were nano-sized Ag from 0 vol% to 4 vol% and nano-sized Al_2O_3 from 0 vol% to 5 vol%, respectively.

The test results indicate that for the Sn37Pb based composite solder joints, the highest shear strength occurs in the content of 3 vol% of nano-sized Ag, Al_2O_3 or TiO_2 particles. The optimum content of reinforcements for the shear strength is the same as that for the creep strength. However, for the composite solder with nano-sized Cu reinforcement the optimum content of Cu is in 2 vol%, which is different from the creep test result. In the creep test the optimum content of Cu is in 1 vol%. For the Sn0.7Cu based composite solder joints, the highest shear strength occurs in the content of 1 vol% of nano-sized Ag, which is different from the creep test result. In the creep test the optimum content of Ag is 3 vol%.

3.2 Effect of test temperature and dead load stress on creep rupture life

To investigate the effect of applied stress and test temperature on creep rupture life of the composite solder joints, a series of tests with different dead load were carried out for the composite solder joints. Generally in the test, the applied stress was from 6.37 MPa to 18.13 MPa, and

Table 3 Effect of volume fraction of reinforcements on shear strength (MPa) for Sn37Pb based composite solder joints

Nano-sized particles	Fraction of reinforcement, vol%					
	0	1	2	3	4	5
Ag	23.33 ± 1.11	33.60 ± 0.61	35.77 ± 1.80	39.80 ± 0.61	35.27 ± 0.80	32.87 ± 0.67
Al_2O_3	23.33 ± 1.11	31.06 ± 1.91	32.13 ± 4.19	35.23 ± 1.78	31.00 ± 1.95	30.03 ± 1.83
TiO_2	23.33 ± 1.11	30.90 ± 1.28	32.07 ± 1.00	35.03 ± 1.55	30.83 ± 0.65	28.70 ± 1.30
Cu	23.33 ± 1.11	29.57 ± 0.93	32.57 ± 0.85	30.23 ± 0.87	27.37 ± 1.15	26.33 ± 2.84

Table 4 Effect of volume fraction of reinforcements on shear strength (MPa) for Sn0.7Cu based composite solder joints

Nano-sized particles	Fraction of reinforcement, vol%						
	0	0.5	1	2	3	4	5
Ag	17.07 ± 0.38	21.53 ± 0.40	25.77 ± 0.49	23.67 ± 1.40	20.33 ± 0.76	18.57 ± 1.14	–
Al_2O_3	17.06 ± 0.38	–	18.47 ± 0.55	20.77 ± 1.43	22.17 ± 0.76	18.07 ± 0.21	12.10 ± 0.95

the testing temperature was from 25 °C to 100 °C. For the Sn37Pb based composite solder, 1 vol% of nano-sized Cu and 3 vol% of nano-sized Ag, Al_2O_3 or TiO_2 were adopted as the reinforcement particles. For the Sn0.7Cu based composite solder, 1 vol% of nano-sized Ag and 3 vol% of nano-sized Al_2O_3 were used as the reinforcement particles, respectively. The test results for the Sn37Pb based and Sn0.7Cu based composite solders are shown in Figs. 3 and 4 respectively.

The results indicate that the creep rupture life is decreased with the increase of applied stress for all the solder joints tested. Moreover, the creep rupture life is decreased with the increase of testing temperature. The creep rupture life of all the composite solders is longer than that of the solder joint without reinforcement particles addition. That is, all the reinforced particles exhibit strengthening effect on the composite solders. Also, the results indicate that the strengthening action of reinforcement particles is more obvious under lower applied stress or at lower test temperature. It is found that different particles exhibit different strengthening effect. Among them, the strengthening effectiveness of nano-sized Ag particles is strongest for
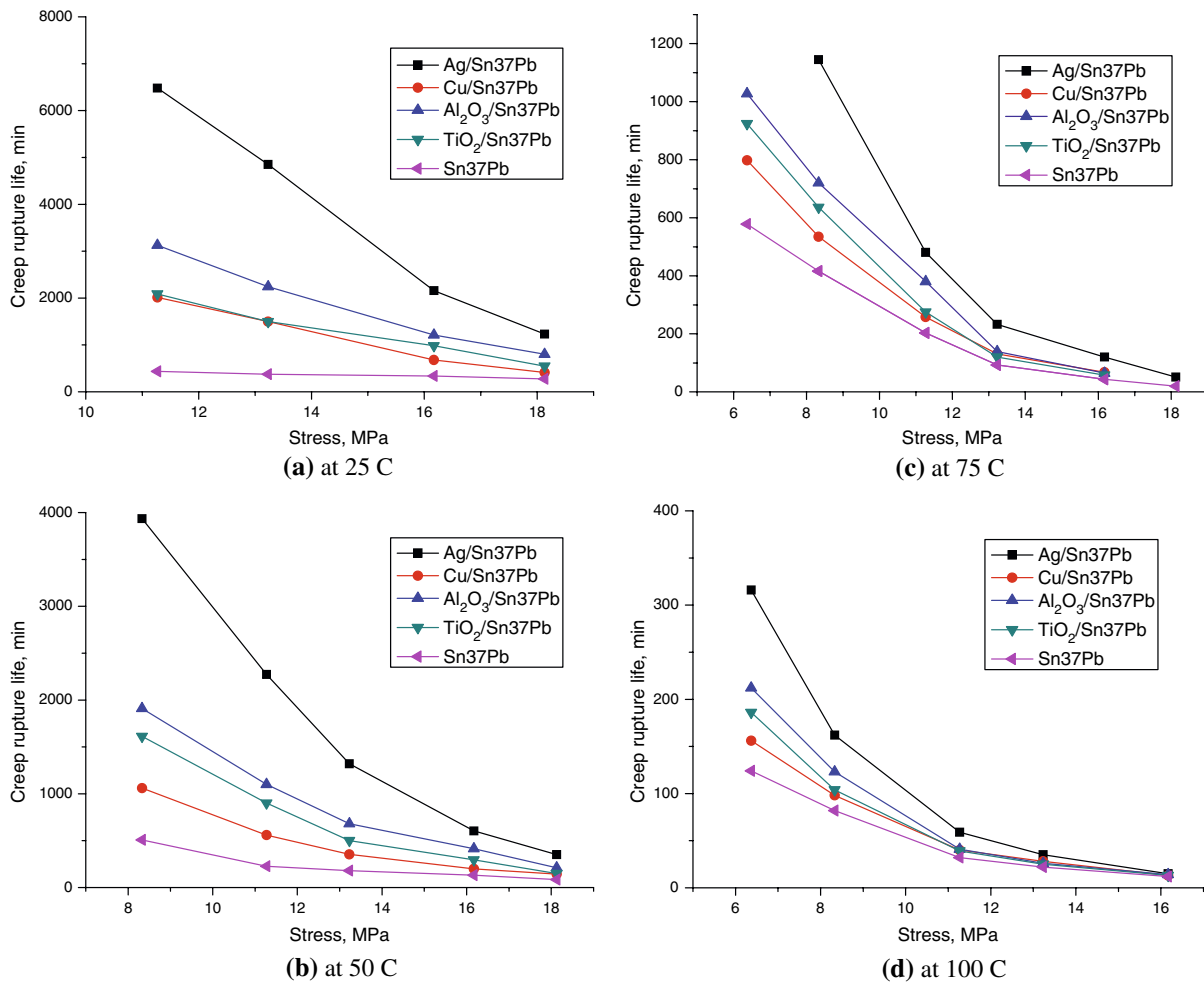

Fig. 3 Effect of applied stress on creep rupture life of Sn37Pb based composite solder joints at (**a**) 25 °C, (**b**) 50 °C, (**c**) 75 °C and (**d**) 100 °C
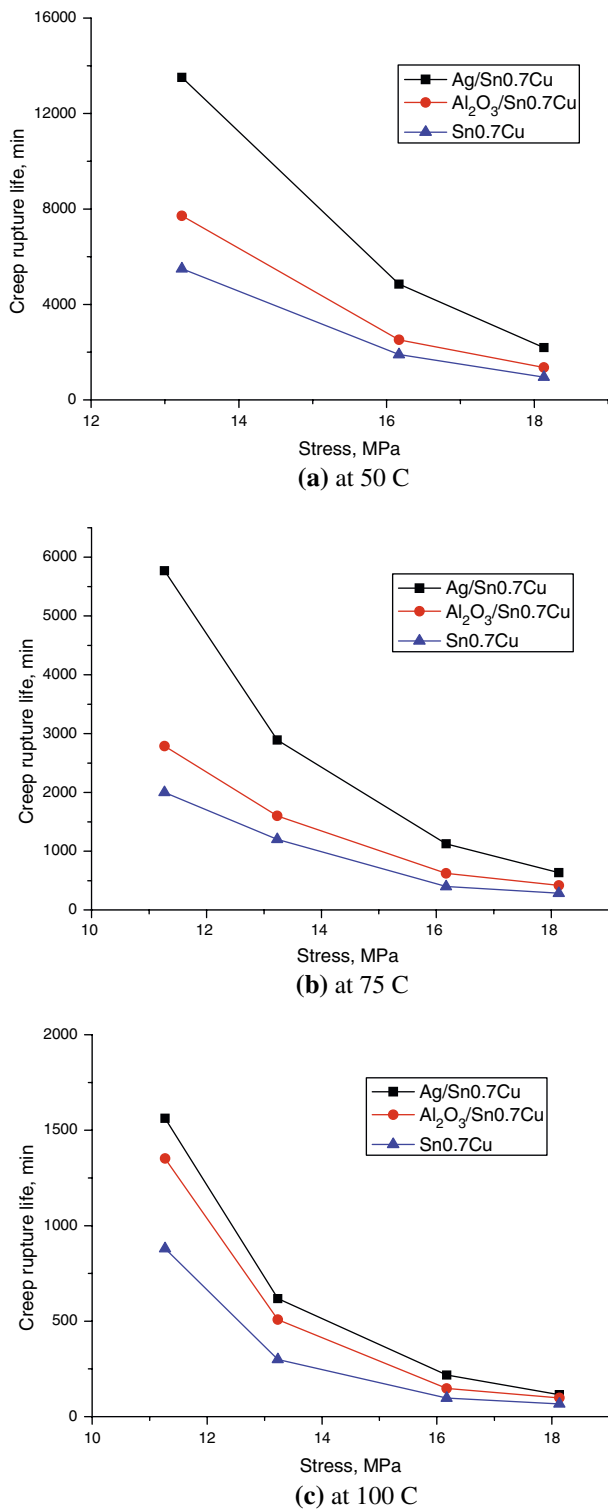
Fig. 4 Effect of applied stress on creep rupture life of Sn0.7Cu based composite solder joints at (**a**) 50 °C, (**b**) 75 °C and (**c**) 100 °C

both Sn37Pb based and Sn0.7Cu based composite. For the Sn37Pb based composite solder, the strengthening effectiveness of nano-sized Cu particles is relatively weaker.

The strengthening effectiveness of Al_2O_3 and TiO_2 particles is basically located between the nano-sized Ag and Cu particles.

Moreover, there exists different strengthening effectiveness for the Sn37Pb based and Sn0.7Cu based composite solder joints. For example, under the stress of 13.23 MPa and at the test temperature of 50 °C, the creep rupture life of the 3 vol% nano-sized Ag particles reinforced composite solder joint is increased up to 7.3 times more than that of Sn37Pb solder joint. At the same time the creep rupture life of the 3 vol% nano-sized Al_2O_3 particles reinforced composite solder joint is increased up to 3.8 times more than that of Sn37Pb solder joint. Under the same test condition, however, the creep rupture life of the 1 vol% nano-sized Ag particles reinforced composite solder joint is increased up to 2.5 times more than that of Sn0.7Cu solder joint. At the same time the creep rupture life of the 3 vol% nano-sized Al_2O_3 particles reinforced composite solder joint is increased up to 1.4 times more than that of Sn0.7Cu solder joint. It is clear that strengthening effectiveness of nano-sized Ag or Al_2O_3 particles in the Sn37Pb based composite solder is stronger than that of nano-sized Ag or Al_2O_3 particles in the Sn0.7Cu based composite solder, although the creep rupture life of Sn37Pb solder joint is shorter than that of Sn0.7Cu solder joint under the identical test condition.

Under a certain temperature, the relation between the applied stress, σ and creep rupture life, t_r is given by

$$t_r = A\sigma^{-\beta}$$

where A and β are constants related with materials and temperature, respectively. Table 5 gives the regressive results for the Sn0.7Cu based nano-sized particle reinforced solder joints based on the above creep rupture life tests. Thus, the creep rupture life of composite solder joints may be approximately evaluated.

3.3 Strengthening of nano-sized particles

There are two kinds of nano-sized reinforcements were used. One is the metallic particles such as Cu, and Ag. The other is the inert particles, such as TiO_2, Al_2O_3, POSS and multi-walled carbon nanotubes. Generally, pure metals particles have a common feature that they bond to Sn well. The above results show that the strengthening effectiveness of nano-sized Ag particles is better than that of nano-sized Cu particles. The reason may be related with the diffusion of metallic particles in Sn matrix. The diffusion of Cu in Sn is faster than that of Ag in Sn under the same condition. Thus, the nano-sized Cu will react with Sn, then the Cu/Sn IMCs formed may redistributed during creep test or aging process. However, the nano-sized Ag particles may be

Table 5 Parameters related with materials and temperature for the Sn0.7Cu based nano-sized particle reinforced solder joints based on regressive calculation

Reinforcement	Temperature° C	A	β
Ag	50	16102813.7	2.86359
	75	16895559.2	3.36728
	100	20612590.6	4.0068
Al$_2$O$_3$	50	9617799.5	2.8883
	75	3538723.4	3.00991
	100	36932415.7	4.30719
None	50	6174944.2	2.84365
	75	4069138.0	3.19359
	100	19856651.6	4.24267

relatively stable in distribution except the Ag/Sn IMCs formed at interface of particles. Figure 5 is the SEM fracture surface of creep rupture test specimen and EDAX
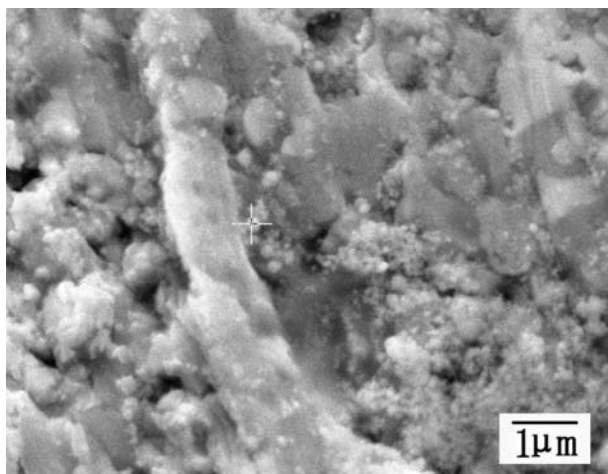
(a) SEM
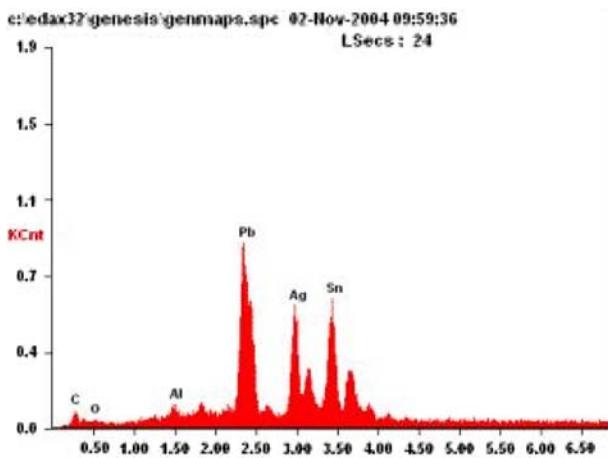
(b) EDAX

Fig. 5 Fracture surface of nano-sized Ag particles reinforced Sn37Pb based composite solder joint after creep rupture test (**a**) SEM and (**b**) EDAX

analysis of a particle in the nano-sized Ag particles reinforced Sn37Pb based composite solder. It is assured by the EDAX analysis that the particle is a nano-sized Ag.

Figure 6 shows the feature of fracture surface of the nano-sized Ag particles reinforced Sn37Pb based composite solder joint specimen after creep rupture life test under stress of 11.27 MPa at 25 °C. Part of the Sn phases is etched away to facilitate the SEM observation. It is found that nano-sized reinforcements mostly either in the matrix or at the grain boundaries in an agglomerated form. Each agglomeration of the Ag particles is distributed uniformly throughout the matrix, which hinders the dislocation motion as well as the grain boundary sliding. Thus, the composite solder joints are able to subject more strain before the onset of the tertiary creep that initiates the rupture of the solder joints.

For the inert particles reinforced composite solders, the bonding between particles and matrix is rather weak. Such inert reinforcements are lack of any metallurgical bond to the matrix. Sometimes, there may exist interfacial cracking between the reinforcement and matrix, as if nonmetallic inclusions are in steels. Decohesive may easily occur at the particles during plastic deformation [12, 13]. Thus, the nano-sized inert reinforcements may be not very effective to enhance the mechanical strength.

4 Conclusion

(1) Nano-sized metal Ag, Cu particles and nano-sized oxide Al$_2$O$_3$, TiO$_2$ particles can be used as the
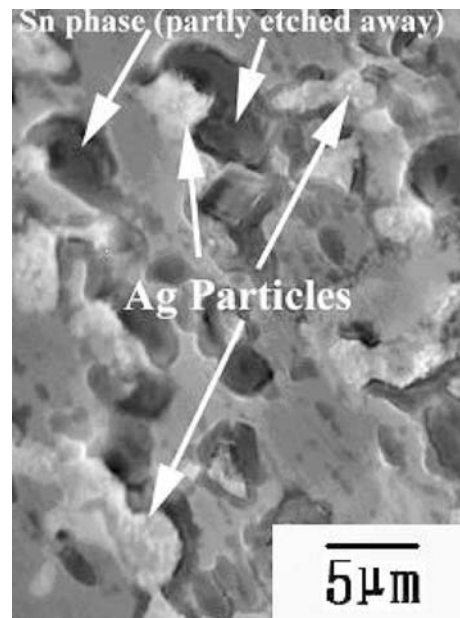

Fig. 6 Fracture surface of nano-sized Ag particles reinforced Sn37Pb based composite solder joint

reinforcements for the Sn37Pb or Sn0.7Cu based composite solders. The results indicate that all the composite solders have improved creep resistance, comparing to the eutectic Sn37Pb solder and the Sn0.7Cu lead-free solder.

(2) Creep rupture life is first increased with the increase in the volume fraction of reinforcement in the composite solders. Then, the creep rupture life is decreased, as the reinforcement content exceeds a certain value. Moreover, the creep rupture life of the composite solder joints is decreased with the increase of applied stress and testing temperature.

(3) Reinforced efficiency of nano-sized Ag particles is the best in all the tested nano-sized reinforcements for the Sn37Pb based and Sn0.7Cu composite solders, when the particles contents are in their own optimum content.

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