


Effects of nano-SiO₂ particles addition on the microstructure, wettability, joint shear force and the interfacial IMC growth of Sn3.0Ag0.5Cu solder

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Abstract In the present study, the nano-composite solder with SiO₂ nanoparticles into eutectic Sn3.0Ag0.5Cu solder were prepared. The addition concentration of SiO₂ nanoparticles ranges from 0 to 1 wt%. The effects of SiO₂ on the microstructure, the wettability, joint shear force of the composite solder under different working temperature and the growth of interfacial intermetallics compounds (IMCs) at solder/Cu substrate during thermal cycling were investigated respectively. The results show that appropriate addition of nano-SiO₂ particles can refine the β -Sn matrixes and enhance the wettability of nano-SiO₂ composite solder. Additionally, slight addition of nano-SiO₂ particles can inhibit the formation and the growth of the interfacial IMCs layer between the solder and Cu substrate during reflow and thermal cycling. Moreover, nano-SiO₂ can also increase the shear force of composite solder joint. However, excessive addition of SiO₂ nanoparticles in the Sn3.0Ag0.5Cu solders degrade the wettability, joint shear force and the inhibition effect on the interfacial IMCs. There is an optimum addition concentration of SiO₂ nanoparticles in Sn3.0Ag0.5Cu solder alloys, which is 0.05 wt%, and Sn3.0Ag0.5Cu–0.05 wt% SiO₂ solders possess the biggest wetting force, highest shear force and the best inhibition effect on the interfacial IMCs formation and growth.

1 Introduction

Lead-free solder alloys have been widely used to replace toxic Sn–Pb solder in electronic industry. Sn–Ag–Cu (SAC) solder alloy system has been regarded as the most promising replacement due to its environmental friendly property and relatively good soldering performance as well as their compatibility with the current microelectronics components [1]. Among all SAC solders, Sn3.0Ag0.5Cu solder alloy has a low melting temperature, near-eutectic composition and good wettability, thus is widely applied in the novel electronic products and systems [2, 3]. However, with the trend toward high integration, multifunction and further miniaturization in microelectronic products, solder joints will be applied under harsh and fierce environments [4], such as high environment temperature and rapid change of temperature. Thus, the application reliability of SAC solder becomes a crucial issue for its further application. Under high temperature, the growth rate of the brittle intermetallic compounds (IMCs) in the solder or at the solder/substrate interface will accelerate and weaken the mechanical properties of solder joints [5]. Besides, current or voltage fluctuation through solder joints change the temperature surrounding an electronic product inducing a thermal stress because of the mismatch of the thermal expansion coefficient between solders, substrates and other components, which can lead to a low cycle thermo-mechanical fatigue (TMF) [6]. During thermal cycles, the solder joints undergo continuous deformation and crack or plastic deformation will appear resulting in the failure of the joints [7].

A method of improving the comprehensive performance of SAC lead-free solder alloys by adding nonmetal nanoparticles into the solder alloys is widely used. In addition, the microstructure, interfacial IMC formation and

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growth and mechanical properties of the nano-composite solder, which is prepared by adding inert nanoparticles into the SAC solder, were studied by lots of researchers. Chang et al. [8] and Chuang et al. [9] studied the properties of SAC solder alloy reinforced by Al_2O_3 nanoparticles and found that adding Al_2O_3 nanoparticles can suppress the growth rate of the IMCs and decreased grain sizes. Liu et al. [10] found that the SiC particles can absorb on the grain of primary β -Sn phase decreasing the sizes of the β -Sn dendrites and can play the role of a secondary reinforcing phase to enhance the microhardness of the solders. However, there are few researches focusing on the properties of nano-composite solders under the action of harsh environment. Hence, it is meaningful to investigate the interfacial IMC growth of nano-composite solder/Cu during thermal cycling and the mechanical properties of nano-composite solder joint under higher temperature environment.

SiO_2 nanoparticles are potential reinforcement particles, and they are helpful in reducing the effect of the CTE mismatch between the solder, substrate and components, which makes a great contribution to significantly enhancing the creep resistance and mechanical properties of SAC solders. However, studies on the effect of nano- SiO_2 addition on the wettability, the growth of interfacial IMCs during thermal cycling and the joint shear force of lead-free composite solder joint are scarce. In this paper, the nano-composite solders were prepared by mechanically incorporating 0.05, 0.1, 0.5, and 1 (wt%) nano- SiO_2 particles into the Sn3.0Ag0.5Cu solder paste. The effect of SiO_2 concentration on microstructure, wettability, joint shear force of nano-composite solder and the IMC growth at composite solder/Cu interface were systematically investigated.

2 Experiments procedure

2.1 The preparation of nano-composite solders

The nano-composite solders were prepared by mechanically mixing nano- SiO_2 particles with Sn3.0Ag0.5Cu solder paste (fabricated by Senju Metal Industry Co., Ltd.). The addition concentrations of nanoparticles are 0.05, 0.1, 0.5, and 1 (wt%). Figure 1 presents the scanning electron microscope (SEM) image of SiO_2 nanoparticles, which can investigate that the SiO_2 nanoparticles were spherical shape and about 50 nm in diameter.

2.2 Microstructure investigate

The Sn3.0Ag0.5Cu- $x\text{SiO}_2$ ($x = 0, 0.05, 0.1, 0.5, 1$ wt%) solder were heating from room temperature to 300 °C at a heating rate of 1 °C/s in a vacuum induction furnace with mechanical stirring, holding the maximum temperature for

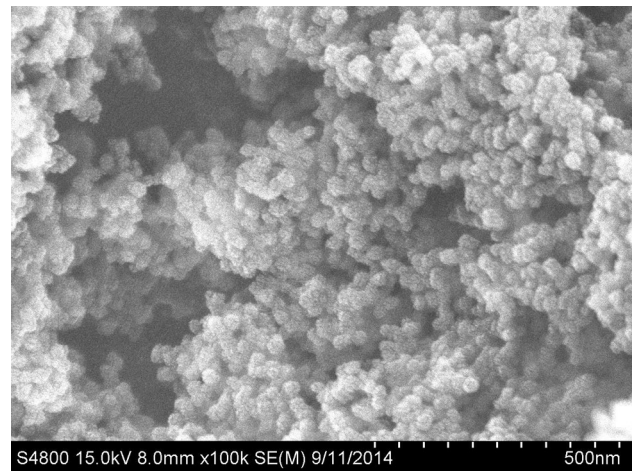


Fig. 1 SEM of the SiO_2 nanoparticles

5 min, and then cooled down to room temperature. A common metallographic practice of grinding and polishing was used to prepare the specimens of Sn3.0Ag0.5Cu- $x\text{SiO}_2$ nano-composite solders [11]. Then the specimens were etched by a mixed solution of 5 ml HCl, 30 ml H_2O , 60 ml $\text{C}_2\text{H}_5\text{OH}$ and 2 g FeCl_3 for approximately 30 s. The microstructure investigations were performed by optical microscopy (OM). X-ray diffraction (XRD) was adopted for the determination of phase composition of the specimens.

2.3 Wetting test

The wettability of the nano-composite solders on Cu substrate was measured by a wetting balance tester (SAT-5200T, Rhesca Co. Ltd., Japan). In this paper, the wettability test was conducted in accordance with Japan industry standard C0099 by Profile Temperature Rising Method. In a wettability test, a Cu coupon with a size of 30 mm \times 5 mm \times 0.3 mm was immersed in the solder paste added nano SiO_2 particles, and then the molten solder climbed up the coupon. At the same time, wetting force was measured and recorded. In this study, wetting temperature was set as 270 °C, the immersion depth was 0.5 mm, the immersion time was 5 s, the immersion speed was 1 mm/s, and the lifting speed was 5 mm/s. The measurement of the maximum wetting force (F_{max}) was used to assess the wetting behavior of the solder alloys. Generally, a large value of the wetting force means a good wettability.

2.4 Interfacial IMC layers growth under thermal cycling

In order to explore the influence of the nano- SiO_2 addition concentration on the interfacial IMC growth, solder/Cu joints were prepared by a traditional reflow process. Pure

Cu plates were used as substrate in this study. Prior to reflow, Cu substrates were immersed into 15 % HCl (by volume) solution in order to remove the oxide layer which exists on Cu substrate surface. Sn3.0Ag0.5Cu–xSiO₂ (x = 0, 0.05, 0.1, 0.5 and 1 wt%) nano-composite solders were placed on Cu substrate, undergoing reflow process with the peak temperature of 250 °C in condition of free spreading, which was shown in Fig. 2. The prepared solder joints were subjected to a thermal cycling test in the Programmable Constant Temperature and Humidity Tester (GDJS-100) for 20, 40 and 60 thermal cycles at the temperature range from –55 to 125 °C, and maintaining for 10 min at each extreme temperatures. During the thermal cycling tests, the heating rate and cooling rate are 1 and 2 °C/min respectively. Scanning electron microscopy (SEM) was used to investigate IMCs formation at the interface between the solder and Cu substrates for both samples before and after thermal cycling, and the elemental compositions of microstructure of nano-composite solder alloy and interfacial IMC layer were determined by energy dispersive X-ray spectroscopy (EDX, Oxford) analysis. The thickness of IMC layers is determined by the area of the IMC layers dividing it’s length after measuring the area and length of interfacial IMC layers by Auto CAD technology software.

2.5 Shear force test of nano-composite solder joint

In order to test the shear force of the SAC–xSiO₂ nano-composite solder, the 0603 resistant components were soldered using SAC–xSiO₂ nano-composite solder pastes on the Cu substrate (the reflow temperature profile showed in Fig. 2) and tested by the Bonding Tester (PTR-1100, Rhesca Co. Ltd., Japan). Figure 3 shows the schematic diagrams of shear force test. To investigate the joint shear

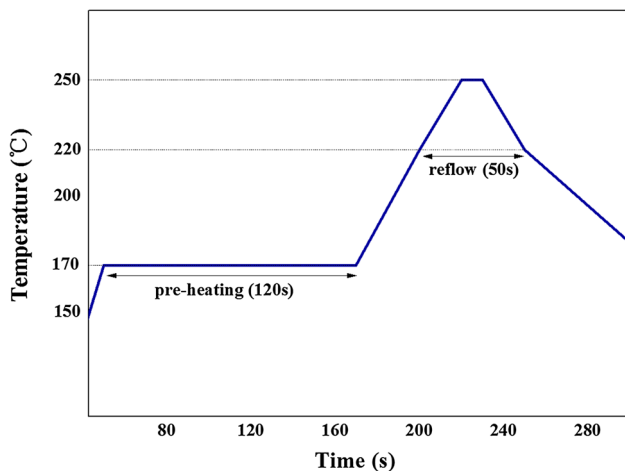


Fig. 2 Typical reflow temperature profile of composite solder/Cu joint

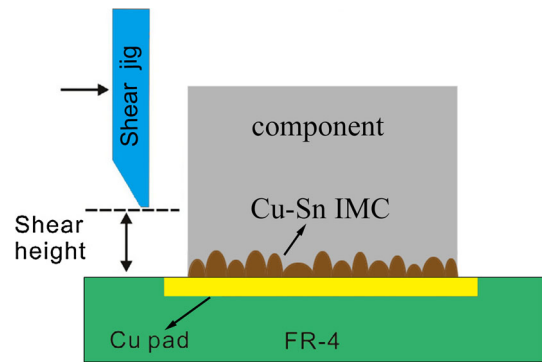


Fig. 3 The schematic diagram of shear force test

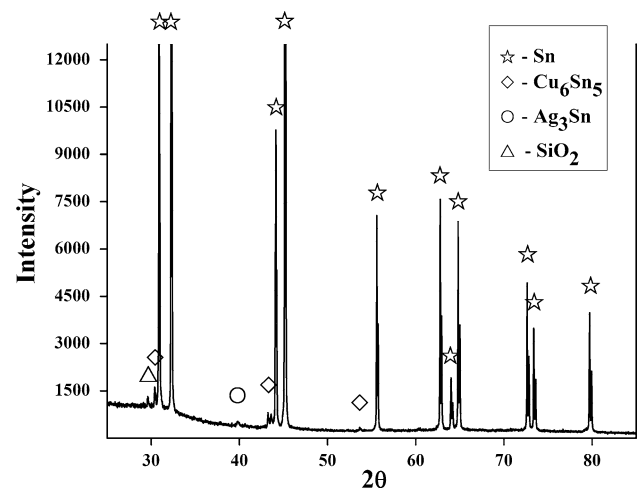


Fig. 4 XRD spectra of SAC305–0.5SiO₂ solder

force of nano-composite solder under different temperature, we conduct the test at the working temperature of room temperature, 75, 100 and 125 °C respectively.

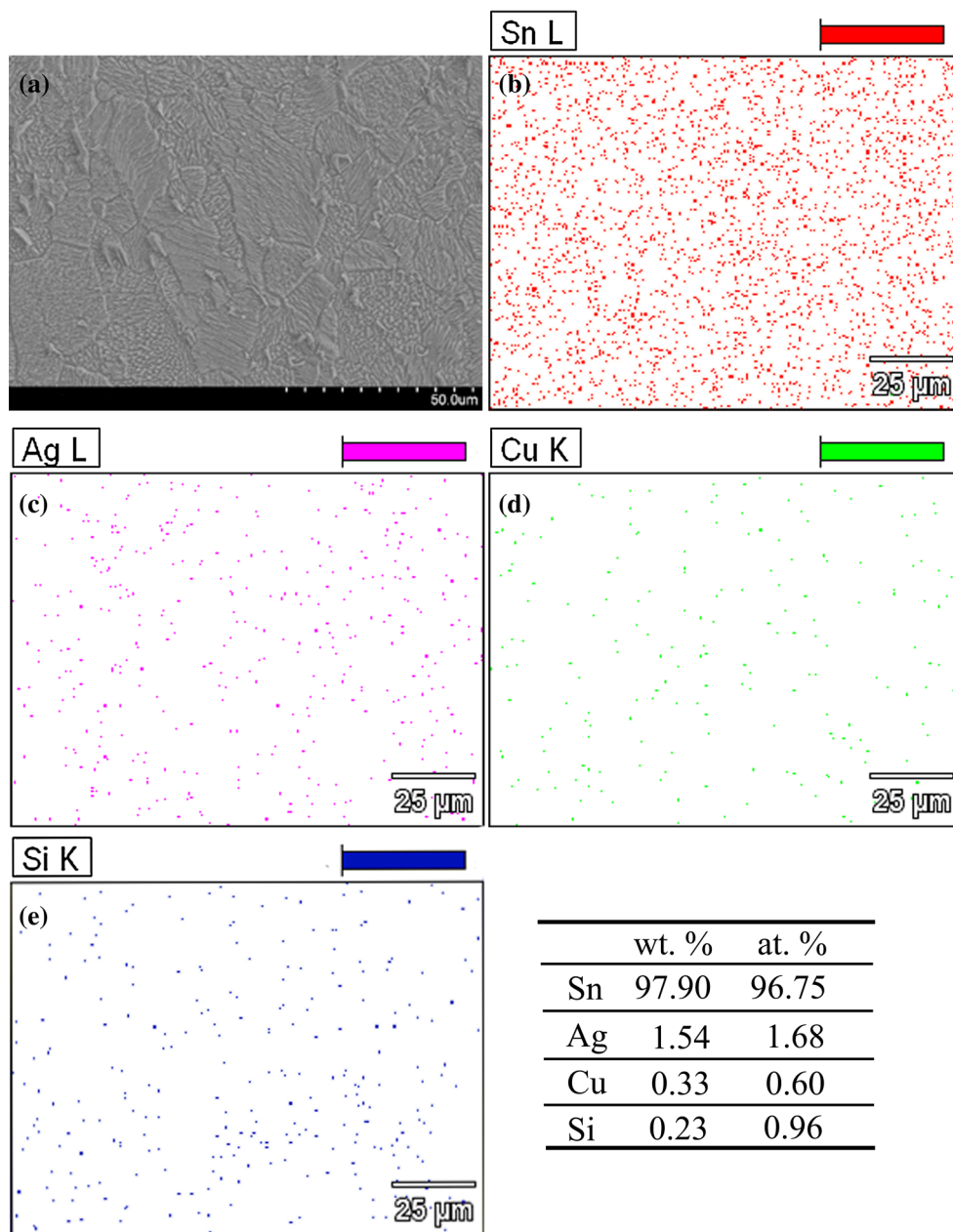
3 Results and discussions

The XRD pattern of the SAC305–0.5 wt% SiO₂ solders are shown in Fig. 4. It can be obviously seen that the nano-SiO₂ particles were successfully blended with the SAC solder.

From the EDX elemental mapping of the Sn3.0Ag0.5Cu–1 wt% SiO₂ after reflow, shown in Fig. 5, it was found that Si can be found in the solder alloy, which indicates that SiO₂ nanoparticles were successfully added into the solder alloy. Additionally, the mapping for Si is approximately the same as the mappings for Cu and Sn, and mainly appear in the boundaries of β-Sn grains.

Figure 6 presents the microstructures of Sn3.0Ag0.5Cu–xSiO₂ nano-composite solders. The island-type structure is

Fig. 5 EDX elemental mapping analysis of the Sn3.0Ag0.5Cu–1 wt% SiO₂: **a** SEM image, **b** mapping for Sn, **c** mapping for Ag, **d** mapping for Cu, **e** mapping for Si. Elemental concentration decreases with decreasing color intensity



made of β -Sn matrix [12, 13]. In general, with the addition of nano-SiO₂, the size of the island-type β -Sn gets small which means the nano SiO₂ particles can refine the microstructure of the composite solders. It is noteworthy that the addition concentration of nano-particles into the solder has relationships with the refinement effect of the microstructure. When the addition concentration is 0.05 wt%, the best grain refinement effect is achieved, as shown in Fig. 6b. According to the theory of heterogeneous nucleation, nano SiO₂ particles can serve as heterogeneous nucleation substrate for the nucleation of primary β -Sn phases, which explains the refinement effect of the solder microstructure after slight addition of SiO₂ nano-particles.

However, when the addition concentration of SiO₂ nanoparticles increases, the agglomeration possibility of nano-SiO₂ particles increases [14, 15]. Agglomerated nano-particles decrease the surface energy of the particles reducing the amount of surface-active nano-particles, which can explain the increased size of β -Sn matrix.

The variation curves of the wetting force of the nano-composite solders with the increase of SiO₂ addition concentration are shown in Fig. 7. Compared to the original SAC solder, the wetting force of the nano-composite solders increased significantly when only adding small amount of SiO₂ (0.05 and 0.1 wt%) nanoparticles. It means that adding slight amount of the nano SiO₂ particles into

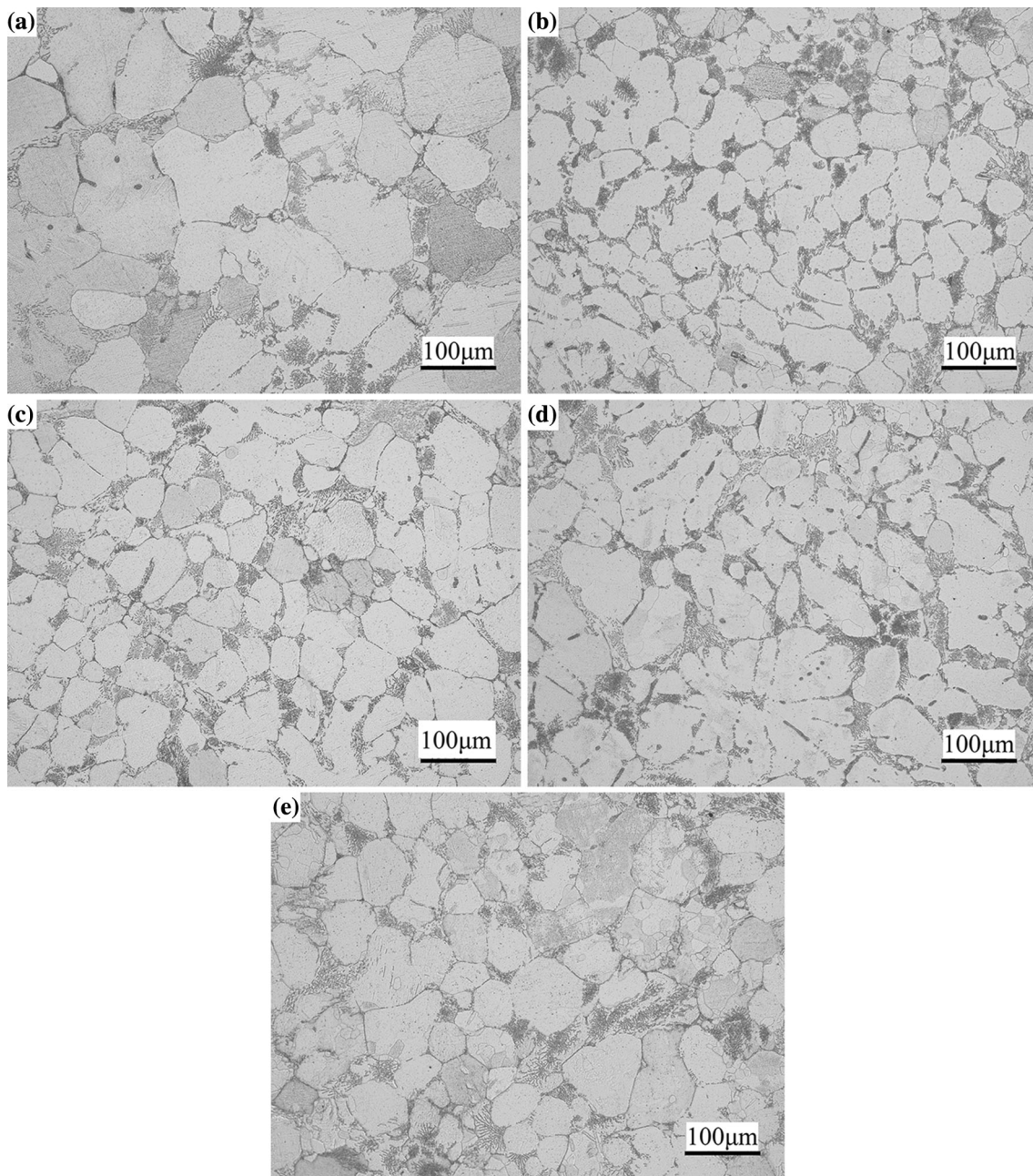


Fig. 6 Micrographs showing the microstructures of nano-composite solders. **a** SAC, **b** SAC–0.05 wt% SiO₂, **c** SAC–0.1 wt% SiO₂, **d** SAC–0.5 wt% SiO₂, **e** SAC–1 wt% SiO₂

SAC solder could improve the wettability of the nano-composite solder. However, with excessive addition (0.5 and 1 wt%) of nano SiO₂ particles, wetting force decreases and become even smaller than the original solder. An explanation is that excessive addition of nanoparticles in the solders increase the viscosity of the molten nano-composite solders, which degrades the wettability. Additionally, the adsorption of SiO₂ on Cu substrates will increase the wetting force of the solder alloy. With excessive addition of nanoparticle in the solder, the nanoparticles will be more

easily to agglomerate and transform into large particles, which will not trend to adsorb on the surface of Cu substrates due to their lower surface energy. Therefore, the real adsorption amount of SiO₂ nanoparticles declines, which decreases the improvement extent of the wettability of nano-composite solder. The wetting force of nano-composite solders first increased significantly and then decreased with the increasing addition concentration of the nano-SiO₂. In other words, there is an appropriate addition concentration of nano-SiO₂ in order to enhance the

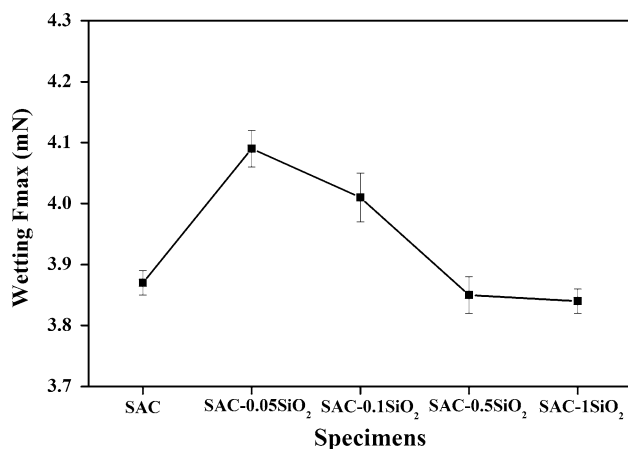


Fig. 7 The wetting force of the SAC- x SiO₂ nano-composite solders

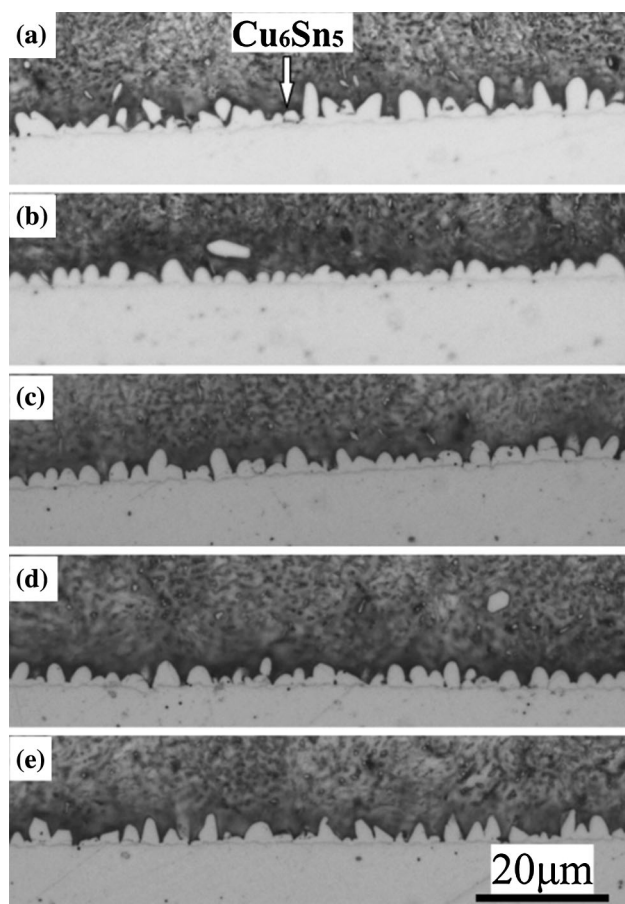


Fig. 8 Cross-section images of the SAC- x SiO₂ nano-composite solder/Cu interfaces. **a** SAC, **b** SAC-0.05 wt% SiO₂, **c** SAC-0.1 wt% SiO₂, **d** SAC-0.5 wt% SiO₂, **e** SAC-1 wt% SiO₂

wettability of SAC composite solders most effectively. The optimal SiO₂ addition concentration is 0.05 wt%. The wetting force is 4.09 mN by adding 0.05 wt% nano-SiO₂ particles, compared with that of the original SAC solder which is only 3.87 mN.

In order to investigate the effect of SiO₂ addition concentration on the interfacial IMC growth between SAC- x SiO₂ nano-composite solders and Cu substrate under condition of reflow soldering and thermal cycling respectively, cross-section images of composite solder/Cu joint were observed and measurements of the interfacial IMC thicknesses were carried out. Figure 8 shows microstructure of the SAC- x SiO₂/Cu interface after reflow soldering. It can be seen that the scallop-shaped Cu₆Sn₅ IMC layer is formed at the SAC/Cu interface after soldering reaction. The interfacial Cu₆Sn₅ layer is not uniformly distributed. Some scallop Cu₆Sn₅ grains are obviously bigger than the others. Figure 8b shows that with slight nano SiO₂ addition (0.05 wt%), the interfacial IMC layers form evenly thin scallop type Cu₆Sn₅. The morphology transformation is related to the reinforcement effect of the nano SiO₂ particle addition. Comparing the IMC thickness shown in Fig. 8a, b, it is noted that the interfacial IMC thickness between SAC nano-composite solder and Cu substrate reduce dramatically, from 3.4 μm of original SAC solder (Fig. 8a) to 2.3 μm of SAC-0.05 wt% SiO₂ solder (Fig. 8b).

The EDX result of IMC layer analyzed using line scanning mode is given in Fig. 9b. The elemental analysis of the interfacial IMC at point A and B in Fig. 9a are shown in Fig. 9b. The positions of point A and B present β-Sn and interfacial Cu₆Sn₅ respectively, which can be proved by the elemental analysis results shown in Fig. 9b. It can be found that both inside β-Sn and on the surface of Cu₆Sn₅, Si can be detected, which indicates that SiO₂ nanoparticles can exist in these places.

After thermal cycle test, the interfacial IMC layer increased and the Cu₆Sn₅ grains were coarsen obviously with uniformly distribution in scallop shape. Figure 10 shows the microstructure of the SAC- x SiO₂/Cu interface after thermal cycle tests. After 80 times cycle, the interfacial IMC of SAC-0.05 wt% SiO₂ (Fig. 10b) grows thicker slightly. The thickness of the nano-composite solders/Cu substrate interfacial IMCs after thermal cycle test also decreases with increasing addition concentration of nano-SiO₂, which has a consistent law with the variation law of that of the IMC thickness at solder/Cu substrate after soldering. The interfacial IMC growth amount at SAC-0.05 wt% SiO₂ solder/Cu interface after 80 times cycle is 1.5 μm, which is smaller than that of the original SAC solder which is 2.5 μm.

The aforementioned phenomenon indicates that SiO₂ addition can suppress interfacial IMC growth significantly during both solder reaction and thermal cycling. However, when the SiO₂ addition concentration continues to increase over 0.05 wt%, the interfacial IMC thickness increases slightly with the addition concentration of nano SiO₂ particles going up. The interfacial IMC thicknesses of each nano-composite solders after reflow and different

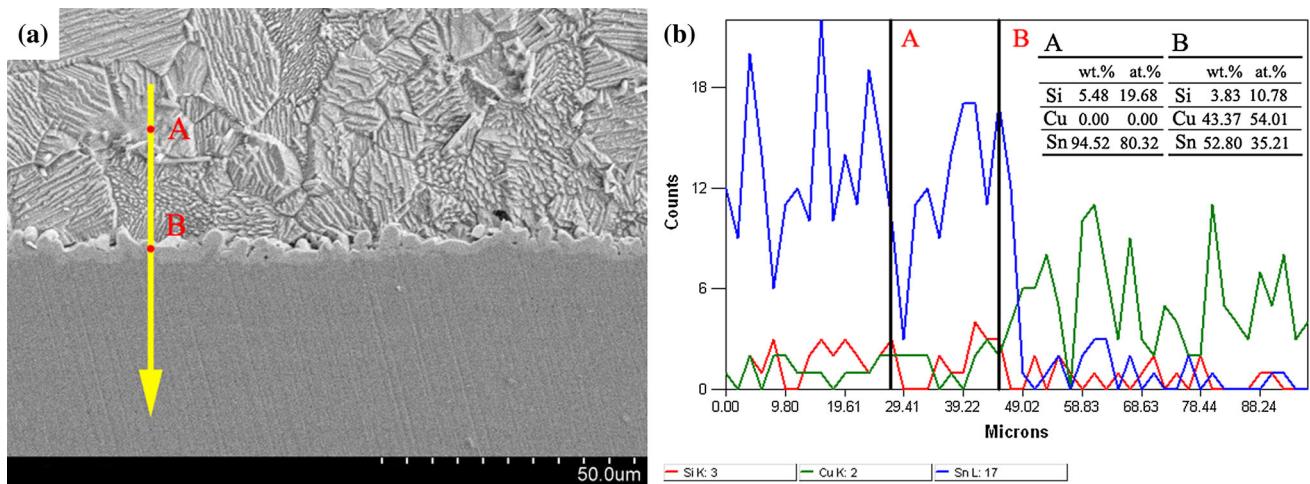


Fig. 9 **a** SEM line scanning image of the micrograph of the IMC layer at SAC305–1 wt% SiO₂ solder/Cu interface; **b** EDX analysis of the line scanning area of the IMC layer at SAC305–1 wt% SiO₂ solder/Cu interface

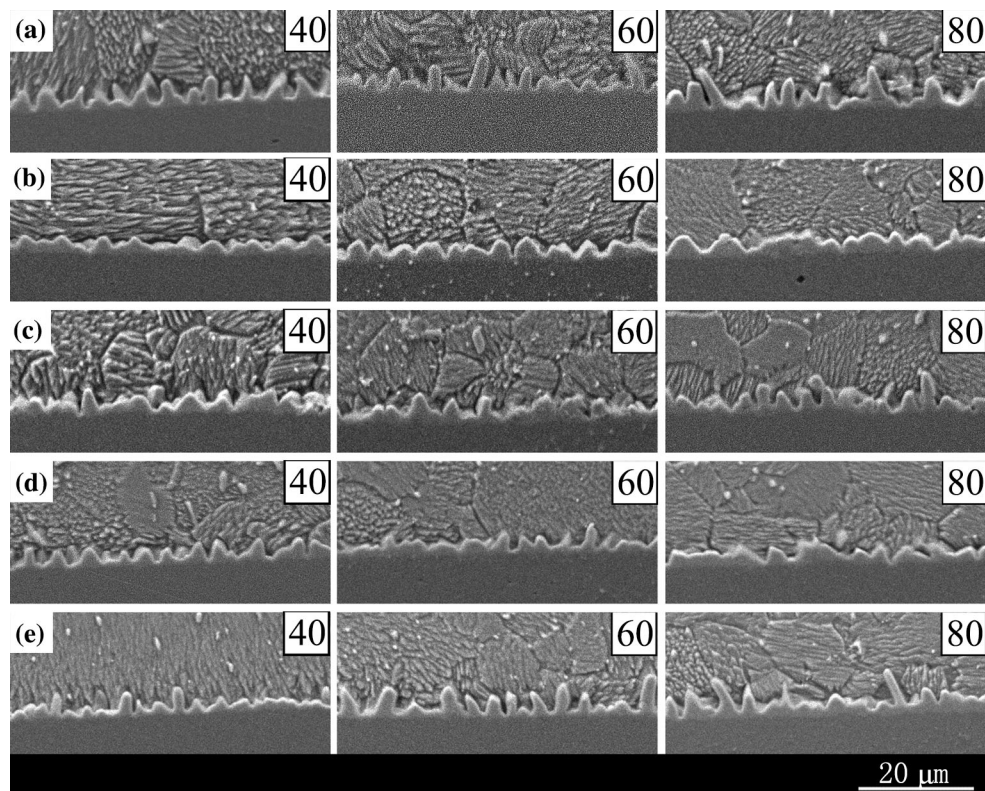


Fig. 10 Cross-section images of the SAC–xSiO₂ solder/Cu interfaces after 40, 60 and 80 thermal cycles. **a** SAC, **b** SAC–0.05 % SiO₂, **c** SAC–0.1 % SiO₂, **d** SAC–0.5 % SiO₂, **e** SAC–1 % SiO₂

thermal cycles are given in Fig. 11. The interfacial IMC layer thickness of SAC305–xSiO₂ nano-composite solders after thermal cycling firstly decreases and then increases with the increasing of the SiO₂ addition concentration. The interfacial IMC layer thickness reaches the smallest value when the addition concentration of nano-SiO₂ in

nano-composite solders reaches 0.05 wt% for all test conditions. The results indicate that SiO₂ can suppress interfacial IMC formation during reflow solder reaction and growth during thermal cycling, and the suppression extent is greatly dependent on nano-SiO₂ addition concentration.

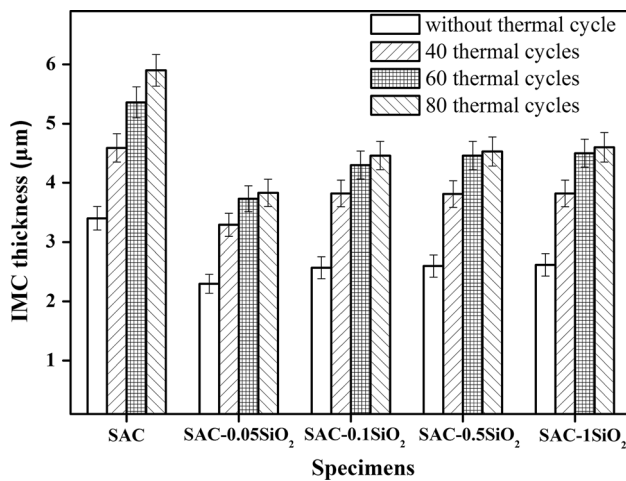


Fig. 11 The IMC thickness of Sn_{3.0}Ag_{0.5}Cu-*x*SiO₂ solders/Cu substrate

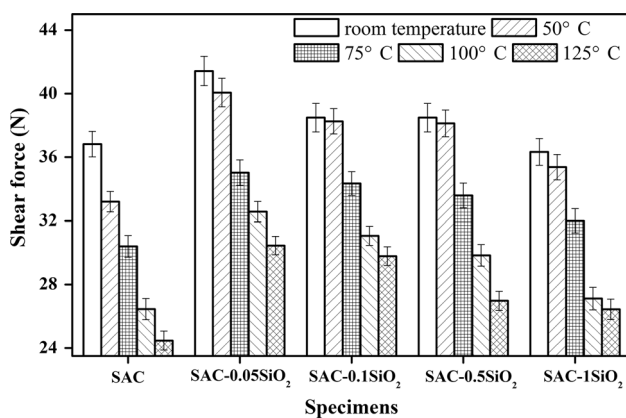


Fig. 12 The shear force of SAC-*x*SiO₂ with Cu substrate at different temperature

Based on adsorption theory, a lot of researchers investigated the adsorption phenomenon of nanoparticles in the solder alloys, which manifests that nanoparticles, such as TiO₂ [4], Fe₂O₃ [11] and SiC [16], can adsorb on the solid surfaces including the surfaces of IMCs and Cu substrate, thus decrease the growth of the IMCs between Cu/solder interface. At the same time, excessive nanoparticles added in the nano-composite solder can agglomerate to transform into large size particles, and as a result influence the adsorption effect of nanoparticles [5]. In this study, the addition of SiO₂ nanoparticles make effects on the interfacial IMC growth. The SiO₂ nanoparticles adsorbed on the interface between the solder and Cu substrate, and became an obstacle for inter-diffusion of Sn and Cu atoms through the interfacial IMC layer during thermal cycling, which slowed down the interfacial IMCs growth. However, with excessive addition of SiO₂ nanoparticle in the nano-composite solder, the nanoparticles tended to agglomerate into larger particles. The larger particles reduced the adsorption of

nano-SiO₂ on the surfaces of Cu substrates because of their relatively low surface energy, which will decrease the obstruction effect of the reaction and diffusion of Sn and Cu atoms.

Figure 12 shows the joint shear force of SAC305-*x*SiO₂ nano-composite solder with Cu substrate at different temperatures. Compared to the original SAC305 solder, the joint shear force of the SAC305 nano-composite solder at all working temperature is enhanced due to nano SiO₂ particles addition and the SiO₂ concentration play critical role on the increase of the shear force of nano-composite SAC solder. A slight addition of SiO₂ nanoparticles in SAC305 solder can obviously enhance the joint shear force, while excessive addition of SiO₂ nanoparticles decreases the degree of enhancement of the joint shear force. When the addition concentration of the nano-SiO₂ in the nano-composite solder is 0.05 wt%, the shear force of joint is improved from 36.82 N to 41.43 N at room temperature and from 24.47 N to 30.45 N at 125 °C working temperature respectively. The improvement of the shear force could be attributed to: (1) SiO₂ nanoparticles pinning in grain boundaries and thus impeding the sliding of the grain boundaries, (2) the increase of dislocation densities and obstacles to restrict the motion of dislocation and (3) the hardening mechanism of SiO₂ nanoparticles [17, 18]. In nano-composite solders, because of the existence of dispersing nanoparticles, the dislocations are transformed from line shape into dislocation loops, around these particles. The interaction between dislocations and enhanced particles improve the shear level of composite solder [19].

4 Conclusions

The effect of nano-SiO₂ particles addition concentration on Sn_{3.0}Ag_{0.5}Cu-*x*SiO₂ nano-composite solder has been studied in this work. The main conclusions obtained are as follows:

1. The appropriate addition concentration of SiO₂ nanoparticles can effectively refine the microstructure and enhance the wettability of the SAC305 solder as well as inhibit the overgrowth of the interfacial IMCs and increase the shear force of the SAC305 solder joints.
2. The addition of SiO₂ nanoparticles can effectively refine the microstructure of SAC305 solder. The grain refinement extent improved first and deteriorated with the increase of SiO₂ nanoparticles in composite solders. When the addition is 0.05 wt%, the best grain refinement effect is achieved.
3. Appropriate addition concentration of SiO₂ nanoparticles can enhance the wettability and joint shear force,

and suppress interfacial IMC growth dramatically. The effect of nanoparticles on wettability, joint shear force and interfacial IMC growth is dependent on nano-SiO₂ addition concentration. A slight addition concentration of SiO₂ nanoparticles in SAC305 solder can obviously increase the wetting force of composite solder, the shear force of composite solder joints under different working temperature and effectively inhibit the interfacial IMC formation and growth of composite solder/Cu substrate. However, excessive addition of SiO₂ nanoparticles in SAC305 solder will degrade the wettability, joint shear force and the inhibition effect on the interfacial IMCs. The optimal addition concentration of SiO₂ nanoparticles in SAC305 solder is about 0.05 wt%.

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Conflict of interest The authors declare that there is no conflict of interests.

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