Microelectronics Reliability 84 (2018) 230–237

Contents lists available at ScienceDirect

Microelectronics Reliability

journal homepage: www.elsevier.com/locate/microrel

Investigation on nano-reinforced solder paste after reflow soldering part 1: Effects of nano-reinforced solder paste on melting, hardness, spreading rate, and wetting quality

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ARTICLE INFO

Keywords:
Nanocomposite solders paste
Spreading rate
Wetting angle
Reflow soldering
Nanoindentation
Solder reliability

ABSTRACT

In this study, the effects of three distinctive contents of metal oxide [Iron Nickel Oxide (Fe_2NiO_4), Iron Oxide (Fe_2O_3), Nickel Oxide (NiO), and Indium Tin Oxide (ITO)] and carbon nanostructure [diamond (C)]-reinforced solder paste on melting behavior, hardness, spreading rate, and wetting angle after reflow soldering were differentiated. Each nanoparticle was mechanically mixed with SAC 305 solder paste to produce homogeneous nanocomposite solder alloys. Based on the results, the addition of nanoparticles slightly increased the melting point; except for the SAC 305-ITO and SAC 305-Diamond composite solder pastes. The SAC 305-ITO composite solder showed negligible effects, but the SAC 305-Diamond composite solder reduced the melting point. The SAC 305-Diamond composite solder exhibited a very high increment in solder hardness compared with other nanocomposite solder pastes. Reinforcement of 0.5 wt% diamond nanoparticles increased solder viscosity higher than other metal oxide nanoparticles. The flow rate of SAC 305-Diamond molten solder on Cu substrate decreased upon the nanoparticle addition during reflow soldering. The reduction in the spreading caused the enlargement in the wetting angle of solder paste. However, very low percentage of carbon nanostructure addition into molten solder alloys reduced the melting point and increased solder hardness with tolerable spreading rate and wetting quality, which enhanced solder reliability after reflow.

1. Introduction

The world is embarking on a fast-track in technology transformation. One of the fundamental factors for this was the ever developing microelectronic fields. The evolution of advanced electronic packages well demonstrated via promising reduction in pitch sizes between components and substrates, among components leads-frames, and solder balls arrangements. Hence, the huge jump from wide to narrow space in electronic assemblies greatly influenced the quality of the solder paste.

Pb-free solder paste was well established in the global market because of health and environmental concerns [1]. Among Pb-free solder pastes, Sn-3.0Ag-0.5Cu (SAC 305) solder alloys rank high in the field of surface mount technology because of their cost and efficiency. However, the applications of SAC 305 alloys were also limited in advanced assembly processes, specifically during reflow soldering. The dominant challenging scenarios of advanced assembly materials were high melting temperature of solder alloys, de-wetting, coefficient of thermal efficiency mismatch, head-in-pillow defects, and poor joining strength. The highly reliable components inside each high-technology product were basic customer requirements toward microelectronic industries. Weak solder joints directly influence the reliability of products after an efficient production process. Recently, researchers homogeneously reinforced different types of particles into solder alloys to upgrade solder joint strength. Previous studies showed that the optimal size range of reinforced particles to distribute into molten solder is 1–5 μm. Submicron particles have a higher possibility to expel with flux toward the surface [2]. Nowadays, the implementation of micro-sized solder alloys has slowly diminished, and such alloys were substituted by nano-sized solder alloys for highly reliable joining performance in research and development.

Currently, researchers were also studying the efficiency of nanocomposite solder paste after reflow. They had attempt to mix various types and amounts of nanoparticles into Pb-free solder alloy (SAC 305) to analyze the effects on joining strength. Priority was given to ceramic or metal oxide-type nanoparticles (that is, TiO_2, ZrO_2, $SrTiO_3$, and Fe_2NiO_4 etc.), which were mechanically mixed with SAC 305 to enhance solder strength [3–11]. Tang et al. [3,4] investigated the effects of TiO_2-reinforced SAC 305 solder paste on intermetallic (IMC) growth rate, microstructure evaluation, micro hardness, and tensile behavior.

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https://doi.org/10.1016/j.microrel.2018.03.027
Received 19 August 2015; Received in revised form 16 March 2018; Accepted 19 March 2018

Table 1
Average particle size distribution of nanoparticles via Nanophox with photon cross-correlation spectroscopy.

Type of nanoparticles	x_{10} (nm)	x_{16} (nm)	x_{50} (nm)	x_{84} (nm)	x_{90} (nm)	x_{99} (nm)	SMD (nm)	VMD (nm)
Fe_2NiO_4	8.77	9.17	10.54	12.13	12.69	14.67	10.45	10.65
Diamond	2.74	2.84	3.20	3.59	3.73	4.16	3.18	3.22
NiO	34.00	34.19	35.24	36.53	37.44	38.80	35.41	35.43
Fe_2O_3	3.04	3.22	3.94	4.81	5.09	6.28	3.86	4.02
ITO	1.09	1.15	1.41	1.76	1.88	2.39	1.40	1.46

The addition of 0.1 wt% TiO_2 composite solder alloys can increase the hardness of plain solder by 37%. However, the reinforcement of 1.0 wt % of TiO_2 into SAC 305 increases the melting temperature only by 0.3% [9]. Furthermore, Fouzder et al. [10] also studied the IMC, microstructure, and shear strength of SAC 305 solder paste with the addition of $SrTiO_3$ nanoparticles. The shear strength of plain solder increases by 3.0% when added with 0.5 wt% of $SrTiO_3$ after one reflow cycle. Gain et al. [7,8] also reported that a SAC 305 composite solder paste added with ZrO_2 nanoparticles does not significantly affect the thermal properties, but both the melting and peak temperatures slowly increase. ZrO_2 nanoparticles in SAC 305 nanocomposite solder paste increase shear strength and reduce grain sizes. However, the nonreactive carbon nanostructure (e.g., graphite, diamond, and carbon nanotubes) particles significantly influence the reliability of solder joint. Bukat et al. [12] focused on the effect of self-prepared nanocomposite SAC 305 solder paste after dispersion with multiwall carbon nanotubes (MWCNTs). As the amount of modified MWCNT increases in SAC 305 solder paste, the wettability of solder joint is reduced. However, the solder joint becomes stronger and reliable if the addition of MWCNT in SAC 305 solder paste was < 0.1 wt%. The spreading area of MWCNT-dispersed SAC 305 solder paste was reduced compared with that of plain solder. The wetting properties oppose the spreading behavior as the amount of nanoparticles in solder alloys increases. Shafiq et al. [13] delineated the addition of a small amount of diamond nanoparticles (0.5 and 1.0 wt %), which considerably increases the mechanical properties of plain solder (i.e., SAC 305 solder paste). However, there were few limitations in the implementation of nanoparticles as reinforcement alloys. The Zener pinning effect of nanoparticles into solder alloys was influenced by surface condition of the nanoparticles and size variations of nanoparticles [14]. Therefore, Mokhtari et al. [15] was used gold coating on silica nanoparticles to increase pinning effect with plain solder. Overall, previous studies outlined the positive and negative effects of reinforcing different types of nanoparticles into SAC 305 solder. Results on the correlation between different types of nanoparticles added with SAC 305 solder paste toward melting behavior, spreading rate, wettability, and mechanical properties to enhance the reliability of solder joint have not yet been reported in the previous work until this report. Thus, further studies were needed to fulfill the research gap, thereby escalating the reliability issues in microelectronics.

It is known that lead-free solder composites are becoming interesting. It is remarkable that is little the known on the effects of the cooling rate during the solidification stage of the soldering on the resulting microstructural array and their interaction with nanoparticles in order to constitute composites of these alloys, as well as on the microstructure-properties relationships. It is well-known that there is a great challenge when developing alternative alloys/composites focusing on the improvement of a specific property. This generally induces to a deleterious effect in another one. The effect of microstructural array on metallic alloys properties has previously been reported, particularly considering the grain size and dendritic array on both the mechanical and corrosion response for a number of alloys [16,17].

In the present investigation, multi-element metal oxide (Fe_2NiO_4, ITO), single-element metal oxide (NiO, Fe_2O_3), and carbon nanostructure (diamond) nanoparticles were used as reinforcement in SAC 305 solder paste. The effective pinning stress of nanoparticles in solder after mechanical mixing was assumed negligible. The reliabilities of these newly produced nanocomposite solder pastes at different aspects (spreading rate, wettability, melting behavior, and increment in hardness values) were analyzed.

2. Experimental procedure

To prepare the different types of nanocomposite solder paste, Sn–3.0Ag–0.5Cu (SAC 305) solder pastes that had been held for 1 h in the refrigerator were brought out to room temperature at 25 ± (2)°C. Subsequently, each type Fe_2NiO_4, C (Aldrich, ≥98% trace metals basis), Fe_2O_3, NiO, and ITO nanoparticles (Zhongke Leiming (Beijing) Science and Technology Co., Ltd.) were mixed with SAC 305 solder paste at a nominal percentage of 0.5, 1.5, and 2.5 wt%, respectively. The nano-reinforced solder was mixed using a mechanical stirrer for approximately 20 min to achieve homogeneity. The average particle sizes of the as-received SAC 305 were within the range of 20–38 μm (400–625 mesh). Meanwhile, the average particle sizes of Fe_2NiO_4, C, NiO, Fe_2O_3, and ITO nanoparticles were simultaneously measured by Nanophox analyzer prior to mixing, as reported in Table 1. The range of nanoparticle sizes was < 50 nm. The diameters of Fe_2NiO_4 nanoparticles were also measured via high-resolution transmission electron microscope (200 kV with field emission, TECNAI G2 20 S-TWIN, FEI) to validate the Nanophox results. The results deduced from both methods almost matched each other, which showed that the average particle size of Fe_2NiO_4 nanoparticles was nearly 10.54 nm (Fig. 1). Thus, the subsequent sizes of the remaining types of nanoparticles were measured by Nanophox analyzer. Furthermore, the homogeneity of reinforced nanoparticles in SAC 305 solder paste after mechanical mixing was confirmed by high-resolution scanning transmission electron microscope equipped with energy-dispersive X-ray mapping on the solder sample. Elemental mapping of SAC 305-Fe_2NiO_4 composite solder paste expressed that Fe_2NiO_4 nanoparticles were homogeneously spread via mechanical mixing (Fig. 2). Therefore, the four other types of nanoparticles were also mechanically mixed with SAC 305 for further investigations.

The melting behavior of each SAC 305 composite solder reinforced with SAC 305-Fe_2NiO_4, SAC 305-Diamond, SAC 305-NiO, SAC 305-Fe_2O_3, and SAC 305-ITO nanoparticles were investigated using a differential scanning calorimeter (DSC) [TA Instrument Q Series, US]. For DSC analysis, < 5 mg of each paste was placed in a sealed hermetic aluminum pan and scanned from room temperature to 260 °C at a rate of 10 °C/min under an argon and helium atmosphere.

The nanocomposite solder mixtures were transferred to a 30 mm × 30 mm × 0.3 mm Cu substrate using a disc plate. The disc plate was machined with a diameter and height of 6.5 and 1.24 mm (0.04 cm^3 in volume), respectively (Fig. 3a and b). Prior to the solder printing process, the Cu substrates were ground with silicon carbide paper and polished until a shining surface was obtained. The polished Cu substrates were then placed in an ultrasonic cleaner with acetone to remove the remaining polishing agents, washed with deionized water, and dried at room temperature.

The printed nanocomposite solder shapes on Cu substrates (Fig.3c) were placed in a laboratory-scale desktop Pb-free reflow oven, which was programmed according to the valid temperature zones. The SAC 305 solder pastes reinforced with different types of nanoparticles with