Fabrication of highly reliable joint based on Cu/Ni/Sn double-layer powder for high temperature application

Hongyan Xu[1, 4], Yaochun Shen[2], Yihua Hu[2], Jianqiang Li[3, 4]*, Ju Xu[1, 4]*

[1] Beijing Engineering Laboratory of Electrical Drive System & Power Electronic Device Packaging Technology, Micro-nano fabrication technology department, Institute of Electronic Engineering, Chinese Academy of Sciences Beijing, 100190, China, xuju@mail.iee.ac.cn

[2] School of Electrical Engineering, Electronics and Computer Science, University of Liverpool, Brownlow Hill Liverpool L69 3GJ, UK, ycshen@liverpool.ac.uk

[3] State Key Laboratory of Multiphase Complex System, Institute of Process Engineering, Chinese Academy of Sciences, Beijing 100190, China, jqli@ipe.ac.cn

[4] University of Chinese Academy of Sciences, Beijing, 100049, China

Abstract: A highly reliable three-dimensional network structure joint was fabricated based on Cu/Ni/Sn powder with double-layer coatings and transient liquid phase bonding (TLPB) technology for high temperature application. Cu/Ni/Sn joint is characterized by Cu metal particles embedded in the matrix of $(Cu,Ni)_6Sn_5/Ni_3Sn_4$ intermetallic compounds (IMCs), with a low void ratio and can be reflowed at low temperatures（<260℃）, but it can reliably work at a high temperature up to 415℃. Cu/Ni/Sn double-layer powders with different Sn layer and Ni layer thickness were fabricated and compressed as preform used for TLPB joint bonding. The microstructure and phase composition evolution for Cu/Sn and Cu/Ni/Sn system during reflow and aging were comparatively studied. Two kinds of interfacial structure designs were made, and corresponding interfacial microscopic morphology was analyzed and compared under once and twice reflow soldering process. The results indicated that Sn coating layer was completely consumed to form $(Cu,Ni)_6Sn_5/Ni_3Sn_4$ IMCs, and the Cu/Ni/Sn joint had a lower void ratio and a higher shear strength than that of Cu/Sn. The mechanism of Ni coating layer inhibiting phase transformation was studied. The high reliable three-dimensional network structure joint based on Cu/Ni/Sn double-layer powder was fabricated for high temperature application.

Key words: three-dimensional network, high reliability, interfacial structure design, transient liquid phase bonding, shear strength

1. Introduction

The third-generation wide-band gap (WBG) semiconductors such as SiC and GaN have emerged as potential substitutes for traditional Si semiconductors due to their superior properties, especially the stable electrical performance at high temperatures [1]. Particularly, SiC chip is able to work stably even at temperature up to 600 °C [2-3]. This calls for suitable bonding materials that are able to extract the best performance from SiC semiconductors and withstand high temperature in rigorous environments at the same time. Reliability is a critical consideration for power module designs [4]. Double-faced soldering structure design for two-sides cooling is one of the methods to solve the heat dissipation problem of high-power module and could effectively improve the reliability.

Multiple soldering materials with low processing temperature but high remelting temperature is required in the double-faced cooling structure. Traditional high temperature technologies are prone to damage the sensitive components owing to high processing temperature [5-7], for example

AuSn/AuSi/AuGe, ZnAl and BiAgX solders. Transient liquid phase soldering (TLPS) was characterized by low temperature soldering and high temperature service, which was considered to be potential candidates for the high temperature alloy soldering technology. However, since the bonding thickness is limited to no less than 20μm, the stress caused by thermal expansion coefficient mismatch between chip and substrate materials cannot be completely absorbed and conducted [8-12]. Composite powder was developed to fabricate complete or part of intermetallic joint, which could make thicker joint and withstand atmosphere temperature over 400 °C, but as known that the intermetallic compounds (IMCs) were hard and brittle, the complete IMCs layer with sufficient thickness is easily cracked [13-14], moreover, the joints made by paste with screen printing tended to produce large voids and contamination, which are caused by volatilization of the organic paste during reflow process [15-16].

The novel three-dimensional network structure joint made by two-time TLPS process based on Cu/Sn preform could be fabricated to a thickness of 100~400μm. The Cu particles in the joint were mounted in the matrix of IMCs, and the properties and reliability of as-reflowed samples have been proved [17-18] at room temperature. However, since the Cu_6Sn_5 IMC phase is easily transformed to Cu_3Sn during high temperature application, the porosity and crack will initiate and propagate along Cu particles, which seriously affects the high temperature reliability of the joint. This paper attempts to coat Ni on the surface of Cu spheres to make Cu/Ni/Sn double-layer micro particles, which will inhibit the reaction of Cu_6Sn_5 with Cu and reduce the formation of Cu_3Sn and void. The influence mechanism of Ni on interfacial morphology and the shear strength of the joint during aging test was investigated and analyzed, and the high-temperature reliability was proved by temperature cycling test.

2. Experimental

Cu lower plates with a dimension of 9 mm× 9 mm× 3 mm and upper plates with a dimension of 9 mm× 9 mm× 1 mm were polished to remove oxidation and then ultrasonically cleaned in acetone to remove oil contamination. Different Cu powders size of <15μm, 15~25μm and 30~50μm were chosen and mixed thoroughly in proportion of 3:2:1. Mixed sized Cu powders were electroplated firstly in $NiSO_4$ solution to form the Cu/Ni intermediate powder, and then it was electroplated in $SnSO_4$ solution to make the Cu/Ni/Sn double coating layer powder. The thickness of Ni coating layer was about ~1μm and Sn coating layer was 2~3μm and 3~5μm respectively. Electroplated Cu/Sn and Cu/Ni/Sn particles were then compressed to preforms with 7000 μm×7000μm× 350μm under 20MPa.

Pure Sn layer with about 1μm thick was sputtered on surface Cu substrates. The sample was placed on a heating plate and reflowed in vacuum atmosphere at 250 °C. In order to maintain reliable interconnect joint, different interfacial structural design was made, and Sn/Cu-Ni-Sn preform/Sn structure and Cu/Sn preform with gradient thickness Sn coating at bulk and interface were prepared. A pressure of 0.03MPa was applied to make the preform fully contacted with Cu substrate, and the bonding process lasted for 8 min. After bonding, the samples were divided into three groups for as-reflowed characterization, aging test and temperature cycling respectively.

The aging test for Cu/Sn and Cu/Ni/Sn samples were carried out in an air blowing thermostatic oven at the temperature of 250 °C for 24 h, 50h, 100h and 200h, respectively. The temperature cycling test were carried out in thermal shock oven from -70~200 °C for 50 cycles, 100 cycles and 150 cycles. The cross-sections of both as-reflowed and aged samples were ground and polished to

0.05μm with Al_2O_3 polishing suspension. The phase evolution for Cu/Sn and Cu/Ni/Sn samples, which were as-reflowed and aged for 200 hours respectively, were tested by X-ray diffraction (XRD) and differential scanning calorimetry (DSC). The microstructure and chemical composition of cross-sections were characterized by a scanning electron microscope (SEM, Hitachi-S4700) with an attached electron dispersive x-ray detector (EDX, EDAX XM4). The precision level of the EDX point is 1μm. In order to evaluate and compare the mechanical reliability of Cu/IMCs joint, two group of temperature cycling samples were prepared for shear tests, respectively.

3. Results and Discussion

3.1 Effect of the electroplate condition on the Cu/ Sn and Cu/Ni/Sn core-shell structure

Fig.1 a) shows the morphology and size distribution of spherical Cu powder. In order to improve the joint properties, different level size grains were distributed to obtain a uniform and compact bonding texture. The cross-section of Cu/Sn and Cu/Ni/Sn powder was shown in Fig.1 b) and c), and Sn coating was evenly covered on surface of Cu or Ni particle.

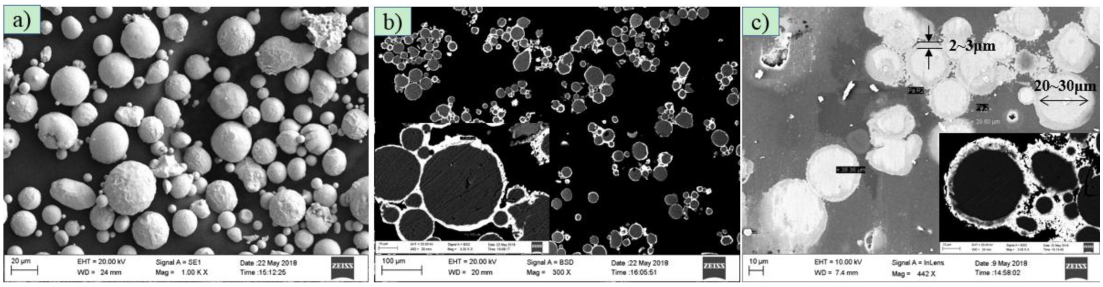


Fig.1 Surface and cross-section morphology of powders, a) was surface morphology of Cu particles, b) and c) was cross-section morphology of electroplated Cu@Sn and Cu@Ni@Sn particles, respectively

Morphology and element distribution of Cu/Sn core-shell structure was illustrated in Fig.2 a) and b). One transition area was found between Cu core and Sn shell, where Sn element increased and Cu element decreased gradually in Sn-Cu direction. The Cu/Ni/Sn double shell structure powder had two transition regions between Sn/Ni and Cu/Ni, as shown in Fig.2 c) and d), while the Cu/Ni/Sn tri-phase area was between Sn/Ni and Cu/Ni transition area. In the vicinity of the Sn/Ni transition area, the Cu concentration decreased, and the Sn content decreased significantly near the Ni/Cu transition area. According to the DSC curve of Cu/Sn and Cu/Ni/Sn powder, seen in Fig.3(a) and Fig.(b), an exothermic peak was found at about 522℃, so Cu-Sn interfacial metallic compounds were formed in the transition area during electroplated process. The Ni element played a little role in suppressing the inter-diffusion of Cu or Sn, which maybe because the Ni layer was too thin or insufficiently densified due to the short electroplating time.

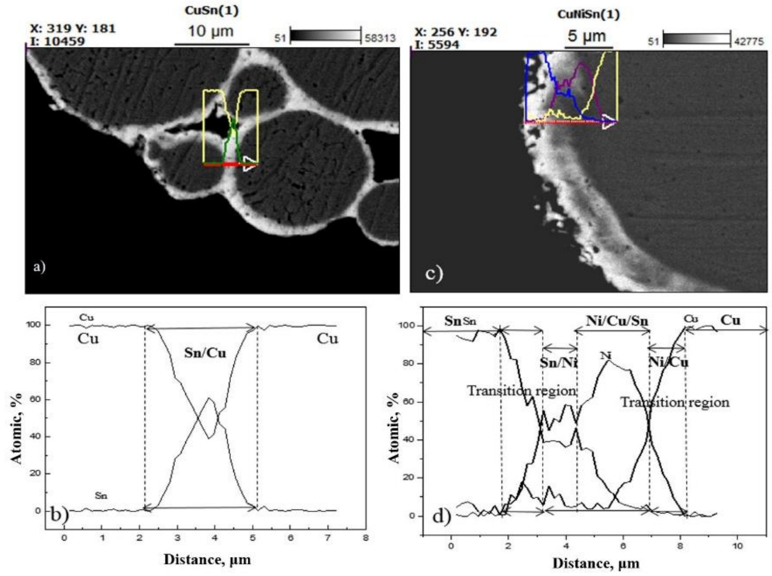


Fig. 2 Composition and morphology of electroplated Cu/Sn and Cu/Ni/Sn powders, a) and c) was morphology of Cu/Sn and Cu/Ni/Sn powders, respectively, b) and d) was the corresponding composition of a) and c)

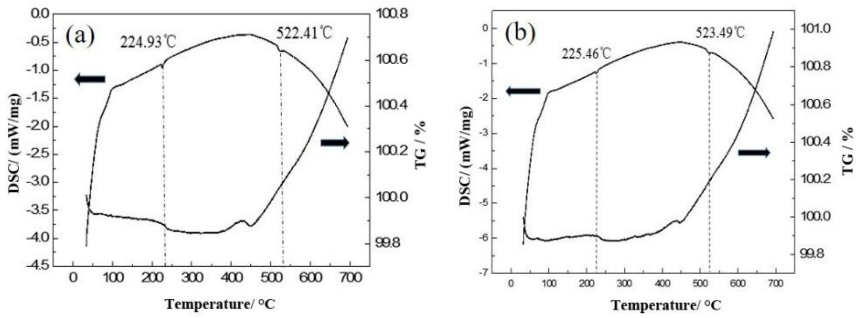


Fig.3 DSC and TG of (a) Cu/Sn and (b) Cu/Ni/Sn powder

3.2 Microstructure characteristic and evolution of Cu-Sn and Cu-Ni-Sn bonding bulk

Cu/Sn and Cu/Ni/Sn double coating layer powder were pressed under 10~30MPa to make the preforms, and then reflowed at 250 °C for different isothermal alloying times. Fig.4 is the joint morphology of Cu/Sn and Cu/Ni/Sn after reflowing at 250 °C for 8 minutes, three-dimensional network structure joint bulk was composed of different size Cu particle surrounded by interfacial metallic compounds (Cu/IMCs). As shown Fig.4 a) and b), Cu/IMCs joints have different microstructure, phase composition and porosity. For example, Cu/Sn joints were Cu/Cu_6Sn_5 or $Cu/Cu_3Sn/Cu_6Sn_5$ with different reflowing time. As the reflow beginning, the phase of the joint was $Cu/Cu_3Sn/Cu_6Sn_5$, and with the reflow time increased, Cu element diffused to Cu_6Sn_5 through Cu3Sn, so part of Cu_6Sn_5 reacted with Cu to form more Cu_3Sn. Since 1mol of Cu_6Sn_5 required 9mol of Cu to form 3mol of Cu_3Sn (see the equations in Fig.5), more voids formed with Cu element diffusing to Cu_6Sn_5 through Cu_3Sn, and voids on the Cu particles side became larger and larger with the increase of reacting time. The voids caused by diffusion was called the Kirkendall voids. Due to the formed Cu_3Sn had higher density than Cu_6Sn_5, so volume void was produced on the Cu_6Sn_5 side when Cu_6Sn_5 transforming to Cu_3Sn, which was called vacancy in the joint. The Kirkendall voids and vacancy was shown in the

Fig.6 a).

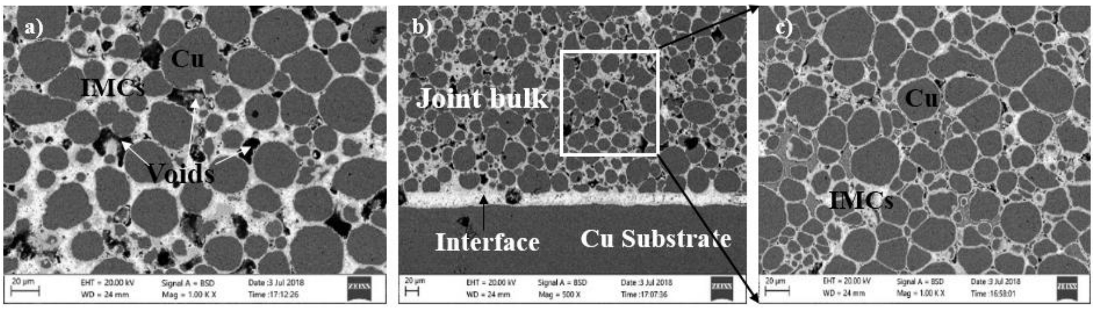


Fig. 4 Joint morphology of Cu/Sn and Cu/Ni/Sn preform reflowed at 250℃ for 8 minutes, a) was Cu/Sn joint, b) was cross-section of Cu/Ni/Sn joint, c) was the magnified image of b)

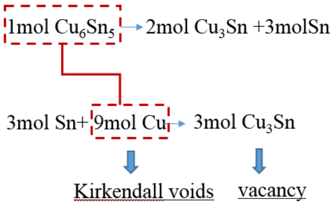


Fig. 5 Voids formation mechanism of Cu-Sn system joint

The morphology of the Cu/Ni/Sn joint was shown in Fig. 4 b). Fig.4 c) was a magnified image, which was more densified than the Cu/Sn joint, and the Ni barrier layer well inhibited the diffusion of Cu element and the forming of Cu_3Sn. Cu, Ni, Sn element area distribution was shown in Fig.6, Ni, Sn and Cu element was as shown in Fig.6 b), Fig.6 c) and Fig.6 d), respectively. Sn layer and Ni layer were coated outside the Cu particle, and Cu particles of different sizes were evenly distributed. There was distinct element transition district at IMCs and in the Cu particle, Ni and Cu inter-diffused to each other, as shown in Fig6. b) and d), and reacted with Sn to form the IMCs.

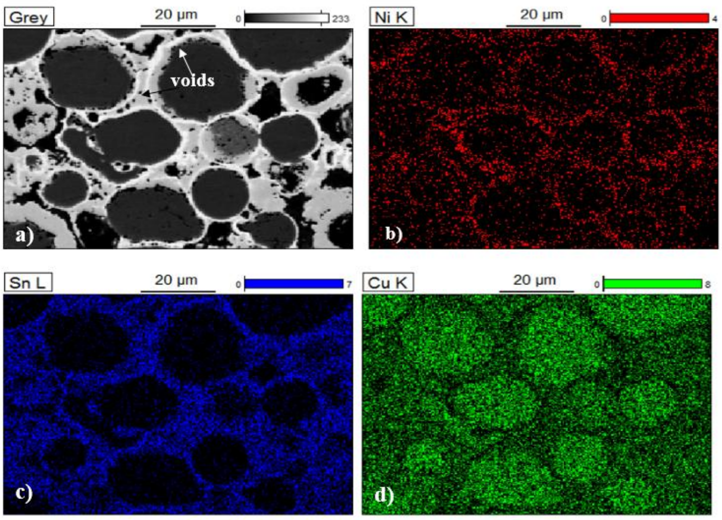


Fig.6 Area scanning results of Cu/Ni/Sn as-reflowed joint

According to the phase diagram of Cu-Ni-Sn, as shown in Fig. 7, there are Ni_3Sn_4, Ni_3Sn_2 and Ni_3Sn phases in Ni-Sn system at 240 °C, and Cu_6Sn_5 and Cu_3Sn phases in Cu-Sn system. The phase evolution of the CuNiSn system during reflow and aging process was shown in Fig.8. At the beginning of reflowing, Ni_3Sn_4 was first produced on the interface of Sn-Ni, and with the isothermal time increased, Cu diffused into Ni_3Sn_4 through Ni layer, and part of Ni_3Sn_4 reacted with Cu to form $(Ni,Cu)_3Sn_4$. With Cu and Ni diffusion concentration increased, part or all of $(Ni,Cu)_3Sn_4$ phase was transformed into $(Cu,Ni)_6Sn_5$. Finally, there are two potential results, one was that all $(Ni,Cu)_3Sn_4$ phases were completely translated to $(Cu,Ni)_6Sn_5$, and the interfacial compounds were $(Cu,Ni)_6Sn_5$ and Ni_3Sn_4. In another case, two layers of $(Cu,Ni)_6Sn_5$ was existed, while the inner layer came from Ni_3Sn_4 and the outer layer was from $(Ni,Cu)_3Sn_4$. The two layers $(Cu,Ni)_6Sn_5$ which have different densities are easier to initiate crack.

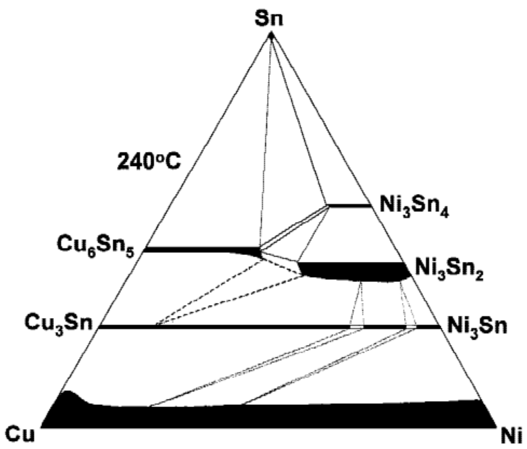


Fig. 7 Phase diagram of Cu-Ni-Sn system

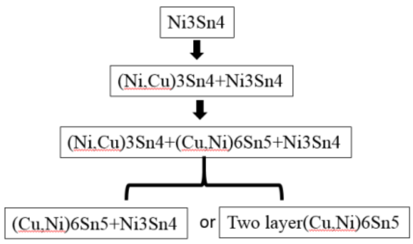


Fig. 8 IMCs phase evolution process of Cu-Ni-Sn system joint

For the time when the CuSn and CuNiSn systems to produce different phases, Fig.9 illustrates the results of DSC and TG of CuSn and CuNiSn system joint after reflow process. The CuSn and CuNiSn system only had one exothermal peak at about 522 °C, which meant only one phase was produced at reflowing process. According to IMCs phase evolution process of Cu-Sn and Cu-Ni-Sn system, $Cu_{11}Sn_5$ may be formed in Cu-Sn system and $(Cu,Ni)_6Sn_5$ in Cu-Ni-Sn system at 522 °C. The TG curve generated a weight loss at about 400 °C for both systems, which can be explained by the residual flux during soldering process. Fig.10 1) and Fig.10 4) showed the phase transformation of the Cu-Sn and Cu-Ni-Sn system for the as-reflowed samples, and Fig.10 2) and Fig. 10 3) showed the phase changing of Cu-Sn and Cu-Ni-Sn system for aging 200 hours samples, respectively. There are Cu, Cu_6Sn_5 and Cu_3Sn phases for Cu-Sn as-reflowed samples, and the Cu_6Sn_5 phase disappeared when aging for 200 hours. Compared to the Cu-Sn system, there are only Cu_6Sn_5 IMC in the Cu-Ni-Sn system. Ni element well inhibits the Cu diffusion to Cu_6Sn_5 to form Cu3Sn, and Cu or CuNi eutectic also existed. After aging for 200 hours, little Cu_3Sn phase existed in the CuNiSn system, indicating that Ni element had a certain inhibitory effect.

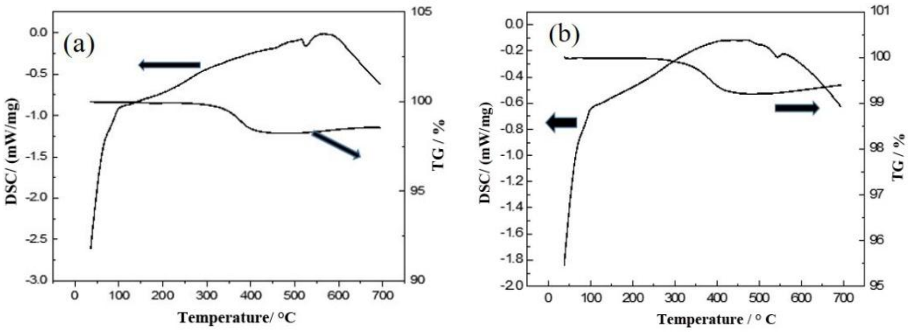
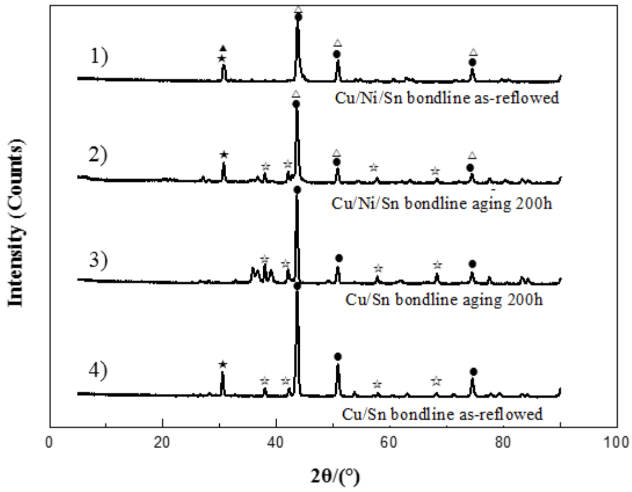


Fig.9 DSC and TG of Cu/Sn (a) and Cu/Ni/Sn (b) preform after reflowing process



●-Cu; ★-Cu6Sn5; ☆-Cu_3Sn; △-Cu_xNi_y; ▲-Ni_3Sn_4

Fig.10 Phases composition of Cu/Sn and Cu/Ni/Sn for as-reflowed and aging for 200 hours samples, respectively

3.3 Interfacial structure design and morphology of Cu-Ni-Sn preform/ Cu substrate

The joint of Cu-Ni-Sn preform/ Cu substrates consists of the three-dimensional network bulk and the interface of Cu-Ni-Sn preform/ Cu substrate, and the dense Cu/Ni/Sn three-dimensional network bulk have been prepared as shown in Fig.4. The different interfacial microscopic structure was dependent on different designs as shown in Fig.11, which presented the schematic diagram of two different interfacial structures of Cu-Ni-Sn preform/substrate, one of which was made by sputtering a thin Sn layer (about 1~2μm) on Cu substrates, and the bulk reacted with Sn coating on surface Cu substrate to form the connecting interface. The other one is made of Cu/Sn core-shell structure powders with gradient Sn coating layer. Sn coating thickness in solder bulk is about 2~3μm, while that in interfacial layer is 3~5μm. The bulk was compressed Cu/Sn powder, and the yellow sphere was Cu particles, white rectangle was gap between Cu/Sn powders, when bulk was soldered, the gap was filled by molten Sn. Different interfacial microstructure was obtained through different structural design and soldering processes, as shown in Fig.12. In the transient liquid phase soldering process, joint with sputtering Sn coating on surface of Cu substrates will produce a dense bulk but an uneven and discontinuous soldering interface, as shown in Fig.12 a), meanwhile high interfacial void ratio will result in big thermal resistant for module. This is attributed to the surface tension of molten Sn so that it did not spread on the surface of substrate/preform, so the interface was incomplete. While preform consists of Cu/Sn powders with gradient Sn coatings, a dense interfacial structure will be obtained, as shown in Fig.12 b), both the bulk and interface were uniform under two soldering processes, with the thickness of the interface being about 5~10μm and the thickness of the bulk being about 100~150μm.

Two soldering processing may solve the problem of non-wetting of the Sn coating layer on substrate. The solid phase sintering was adopted to make dense interface though solid Cu/Sn inter-diffusion, avoiding the flow of molten Sn, and then transient liquid phase soldering was chosen to melt Sn completely and fill the voids to obtained the dense and evenly interfacial microstructure. But for the gradient Sn coating thickness structure, they may have inconsistent microstructure and joint mechanical property. For the layer of Cu/Sn with 3~5μm Sn layer near preform will have a thicker IMCs layer than that of the bulk, which will be potential vulnerabilities of the joint. So, the two soldering processes has been chosen to make the interfacial structure.

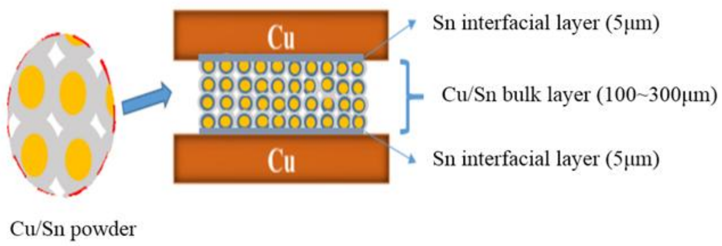


Fig. 11 Schematic diagram of three-dimensional network and interfacial structure of joint

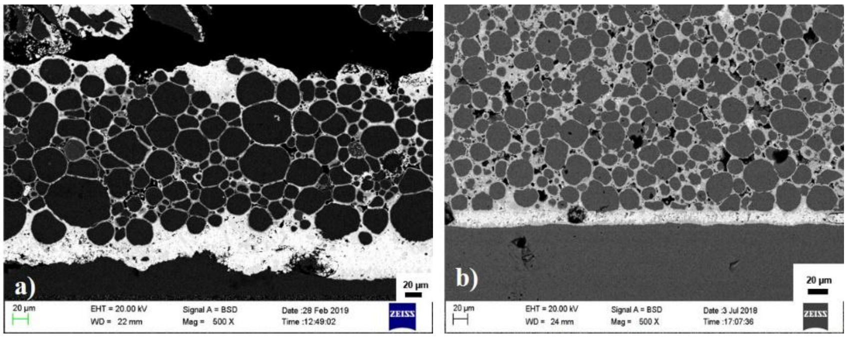


Fig. 12 Different interfacial morphology structure under different interfacial design, a) was for preform with sputtering Sn on substrate, b) was for preform with gradient Sn coating on Cu particles.

3.4 Shear strength of Cu-Sn and Cu-Ni-Sn system soldering joint

The Cu-Sn and Cu-Ni-Sn system joint were prepared by two soldering processing with preform and sputtering Sn on Cu substrate. The shear strength of joint of Cu-Sn and Cu-Ni-Sn system before and after temperature cycle (-70~200 °C) was tested, and the result was illustrated in Fig.13. It is about 200MPa for Cu/Ni/Sn as-reflowed samples, which decreased with increasing temperature cycling number and reached about 140MPa for 150 temperature cycles. However, for the Cu/Sn system, though the shear strength is about 220MPa before temperature cycling, and it decreased rapidly with the increasing temperature cycles, and reached about 80MPa for 150 cycles, which was smaller than that of Cu/Ni/Sn system. The void rate variation of Cu/Sn system was showed in the Fig.14, the void rate increased with aging time extension. Fig.14(a)~(d) was void evolving process of Cu/Sn system aging for 25h, 50h, 100h and 200h, respectively, with the aging time extended from 25h to 100h, void rate continuously increased, until aging time up to 200h, the void rate was comparably stable to about 12%~15%, as shown in Fig.14 (d), where the Cu6Sn5 phase has completely transformed to Cu3Sn. That is the reason why the shear strength decrease for Cu/Sn system with increasing temperature cycles.

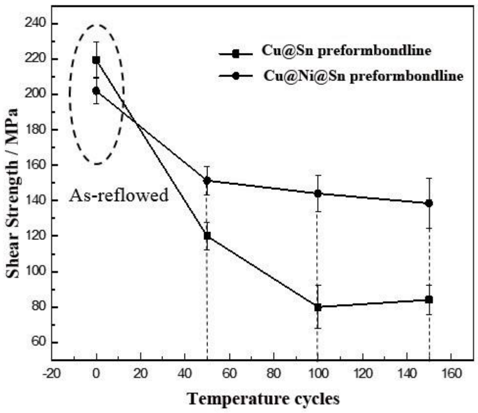


Fig. 13 Shear strength of Cu/Sn and Cu/Ni/Sn bondline at different temperature cycles (-70~200 °C)

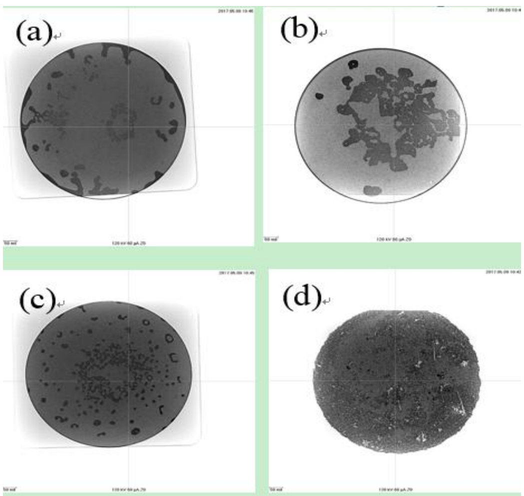


Fig.14 X-Ray results of Cu/Sn at different aging time, (a)~(d) was for Cu/Sn aging 20h, 50h, 100h, and 200h, respectively

The shear strength of Cu/Ni/Sn system decreased from 200MPa to 110MPa with aging time prolonging from 24h to 200h, but void rate was slight increased from 5% to 6.5%, as showed in Fig.14, the shear strength was still higher than that of Cu/Sn system, and void rate was lower than that of Cu/Sn system, It may be that the Ni element in the Cu/Ni/Sn system effectively inhibited the transformation of Cu_6Sn_5 phase into Cu_3Sn phase, voiding the kirkendall effect and vacancy formed and lower void rate, make the joint have long lifetime and high reliability for high temperature application. While the shear strength of high-lead alloy solder and sintered nano-silver was 28MPa and 20~80MPa respectively at room temperature, and deceased to 7.5 MPa and 20~40 MPa at 200 °C [19], which was lower than that of Cu/Sn or Cu/Ni/Sn system, and the Cu/Sn and Cu/Ni/Sn system have comparatively high joint reliability.

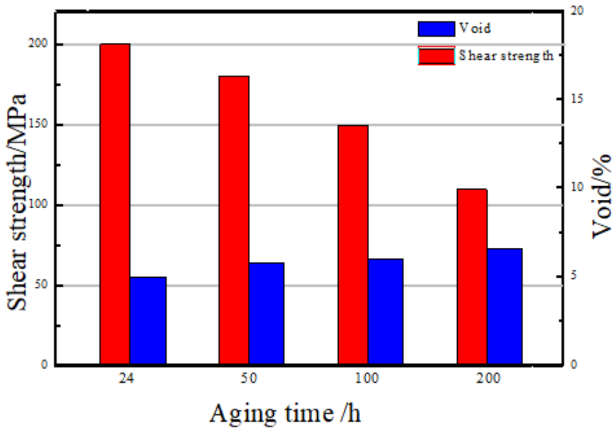


Fig.15 Shear strength and Void rate vs. aging time for Cu/Ni/Sn system

4. Conclusions

(1) Uniform and compact Cu/Sn and Cu/Ni/Sn coating layer structure powders were prepared by electroplating process. The thickness of the Ni layer was about ~1μm, and the thickness of the Sn layer was about 2~3μm and 3~5μm, respectively. The composition and element distribution were analyzed, and different transition districts were existed in Cu/Sn and Cu/Ni/Sn systems.

(2) The microstructure characteristics and phase evolution of Cu-Sn and Cu-Ni-Sn joints during reflow and aging processes was studied. Due to the Kirkendall voids and vacancies in the Cu-Sn system, relatively porous microstructure was obtained in the joint than Cu-Ni-Sn system, where Ni element effectively inhibited Cu_6Sn_5 transforming to Cu_3Sn. The interfacial compounds are $(Cu,Ni)_6Sn_5$ and Ni_3Sn_4, or two layers of $(Cu,Ni)_6Sn_5$ in Cu-Ni-Sn system.

(3) Two interfacial structure design of Sn/Cu-Ni-Sn preform/Sn and Cu/Sn preform with gradient Sn coating thickness were carried out. The corresponding interfacial micro-morphology in once or twice soldering processes was investigated. Dense and uniform interfacial connection was got under twice TLPS for Sn /Cu-Ni-Sn preform/Sn structure by sputtering Sn on surface of substrates

(4) The dense and uniform joint was fabricated under two soldering process, and the shear strength and void rate was compared for Cu/Sn and Cu/Ni/Sn systems. The shear strength was slightly higher for Cu/Sn system than Cu/Ni/Sn system as-reflowed samples. However, after 150 temperature cycles (-70~200 °C), it was reduced quickly to about 80MPa for Cu-Sn system while it was 140MPa for Cu/Ni/Sn system. The void rate of Cu/Ni/Sn system was smaller than that of Cu/Sn system. Therefore, Cu/Ni/Sn system has high reliability for high temperature applications.

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