The 56th International October Conference on Mining and Metallurgy
22-25 October 2025, Bor Lake, Bor, Serbia
www.ioc.tfbor.bg.ac.rs

DOI: 10.5937/IOC25430N
Original scientific paper

MICROSTRUCTURES AND WETTABILITY BEHAVIOR OF Sn-0.7Cu SOLDER DOPED WITH GRAPHENE NANOSHEETS

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Abstract

The effect of graphene nanosheets (GNS) on the wettability and microstructure of Sn-0.7Cu-xGNS (x = 0, 0.02, 0.04, 0.06, 0.08, and 0.1 wt.%) nanocomposite solders was investigated. The composites were produced using the powder metallurgy (PM) technique. The microstructures of the solder joints between the solders and copper substrates were examined using a scanning electron microscope (SEM), and the presence of intermetallic compounds (IMCs) was confirmed by energy-dispersive spectroscopy (EDS). The thickness of the intermetallic layer formed at the solder/Cu interface was reduced by 64.6% with the addition of up to 0.1 wt.% GNS, compared to the Sn-0.7Cu base solder. The wettability of the samples was evaluated by measuring the spreading area of the solders on the copper substrates. The results showed that the addition of up to 0.1 wt.% GNS significantly improved wettability, as evidenced by a 95.2% increase in spreading area compared to the unreinforced solder.

***Keywords**: (powder metallurgy, nanocomposite, graphene nanosheets, intermetallic compounds)*

1. INTRODUCTION

Tin–lead (Sn–Pb) solders have been widely used in the electronics industry due to several advantages, including a low melting point, good wettability, and cost-effectiveness [1]. However, growing environmental and health concerns related to lead toxicity, along with international regulations, have led to a global shift toward the development of lead-free solder materials. As a result, significant research efforts have been focused on identifying suitable alternatives that can match or exceed the performance of traditional Sn–Pb solders [2, 3].

Among lead-free solder systems, Sn–Cu, Sn–Bi, Sn–In, Sn–Sb and Sn–Ag–Cu alloys, particularly Sn-0.7Cu, are considered promising due to their low cost, environmental compatibility, and acceptable mechanical properties [4]. However, they often exhibit poor wettability and the formation of coarse IMCs, which can reduce joint reliability. To improve these properties, recent studies have explored the addition of various types of carbon nanoparticles, such as fullerenes (FNS), graphene nanosheets (GNS), carbon nanotubes (CNT), single-walled carbon nanotubes (SWCNT) and multi-walled carbon nanotubes (MWCNT). Among these, GNS has garnered significant attention due to its exceptional properties, including high thermal and electrical conductivity, mechanical strength and large surface area [5, 6]. GNS exhibits poor dispersibility in metal matrices due to strong Van der Waals forces, which lead to low wettability and particle agglomeration [7]. To overcome these limitations, the mechanical alloying (MA) process is commonly employed to enhance the interaction between graphene and the metal matrix. This method involves cold welding, fracturing, and rewelding of powder particles in a high-energy ball mill. However, the effect of GNS addition on the Sn-0.7Cu solder system has not been thoroughly investigated. According to the available literature, only Yang and his associates have studied the effect of varying GNS contents (0.02, 0.05, 0.075, and 0.10 wt.%) on the properties of the Sn–0.7Cu base alloy. Their findings indicated improvements in wettability, microhardness and microstructural refinement of the solders [8, 9].

In this study, the wettability and microstructure of the solder joints formed between Sn-0.7Cu-xGNS solders and the copper substrates were investigated. The graphene-reinforced composite solders were synthesized using the PM technique, which included MA, cold pressing and sintering.

2. EXPERIMENTAL

Materials used in this work were powders of tin (Sn), copper (Cu) and GNS. First, Sn and Cu powders (particle size up to 75 µm) were mixed in a specific ratio using a "Turbula T2F" three-axis mixer at 50 rpm for 30 hours to form the Sn-0.7Cu base alloy. The mixture was then MA with GNS (with an average diameter of 5-10 µm and a thickness of 3-5 nm) in a high-energy planetary ball mill "FRITSCH Planetary Ball Mill P-7" at 100 rpm for 3 hours. After MA, the obtained powders were compacted under 300 MPa using a hydraulic press to form green compacts with a diameter of 12.7 mm. Finally, the green compacts were sintered in an argon atmosphere at 185 °C for 3 hours. As a result, the Sn-0.7Cu-xGNS nanocomposite materials (x = 0, 0.02, 0.04, 0.06, 0.08, and 0.1 wt.% GNS) were obtained.

The wettability of the composite solders was determined by measuring the spreading area of the solders on copper substrates. Cylindrical sintered samples (3 mm in diameter and height) were placed at the center of the copper substrates (20×20×1 mm). To prevent oxidation during soldering, RMA223 flux was applied. The soldering process was performed on a hot plate at 250 °C for 1 minute in air. After soldering, the spreading areas were measured using ImageJ software. Subsequently, the microstructures of the solder joints at the solder/Cu interface were examined using a scanning electron microscope (SEM), and the presence of IMC was identified through energy-dispersive spectroscopy (EDS). The microstructural analysis was performed on standard metallographically prepared samples using a "Tescan Vega 3 LMU" SEM equipped with an "Oxford Instruments X-act" EDS detector.

3. RESULTS AND DISCUSSION

Figure 1 shows the spreading behavior of Sn-0.7Cu-xGNS solders on Cu substrates after soldering, while Figure 2 presents the changes in the spreading area as a function of GNS content in the composite solders.

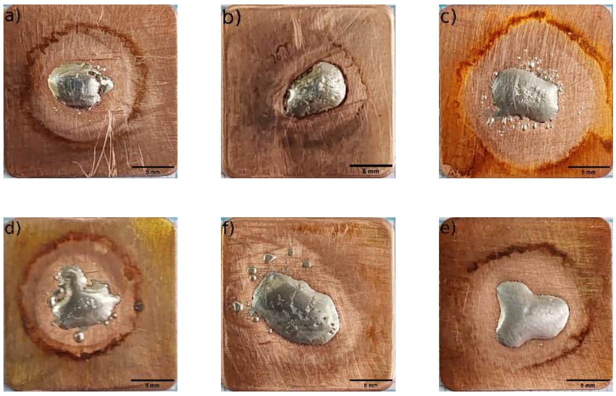


Figure 1. Spreading of solders on the Cu substrates: (a) Sn-0.7Cu; (b) Sn-0.7Cu-0.02GNS; (c) Sn-0.7Cu-0.04GNS; (d) Sn-0.7Cu-0.06GNS; (e) Sn-0.7Cu-0.08GNS; (f) Sn-0.7Cu-0.10GNS

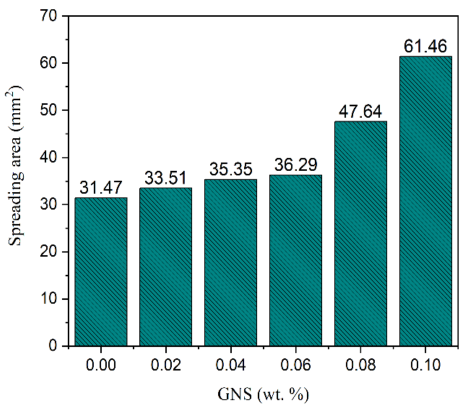


Figure 2. Changes in the spreading area as a function of GNS content in Sn-0.7Cu-xGNS solders

The results presented in Figures 1 and 2 demonstrate that the addition of GNS increases the spreading area of the composite solders, indicating improved wettability. The base Sn-0.7Cu solder exhibited a spreading area of 31.47 mm^2, while the composite solder with a GNS content of 0.10 wt.% has a spreading area of 61.46 mm^2, which is approximately 95.3% higher than the base solder. This improvement can be attributed to the reduction in surface tension between the solder and Cu substrate due to the presence of GNS, as well as enhanced oxide layer removal due to the action of the flux. These results suggest that the addition of GNS leads to the formation of a wider and more stable bond between the solder and the substrate, enhancing the mechanical reliability of the solder joint.

Following the wettability test, the microstructure of the intermetallic layer at the solder/Cu interface was examined. Figure 3 presents the corresponding SEM images obtained after the soldering process.

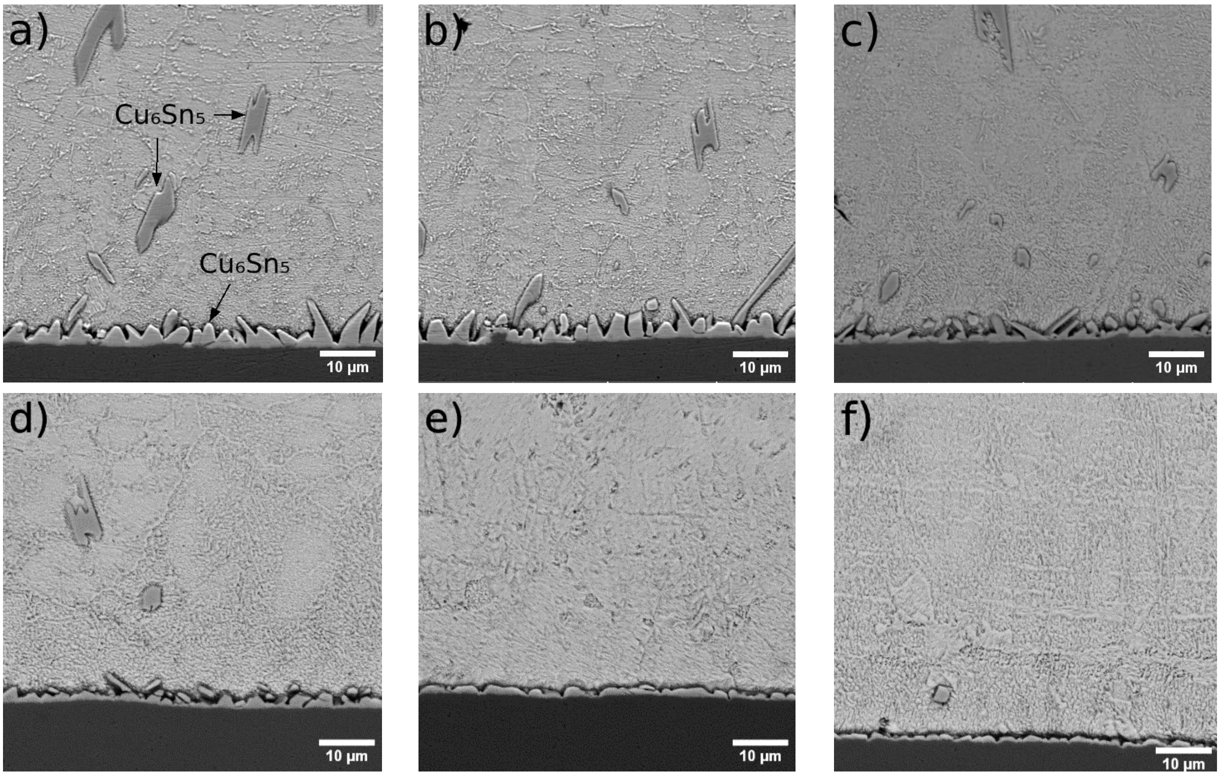
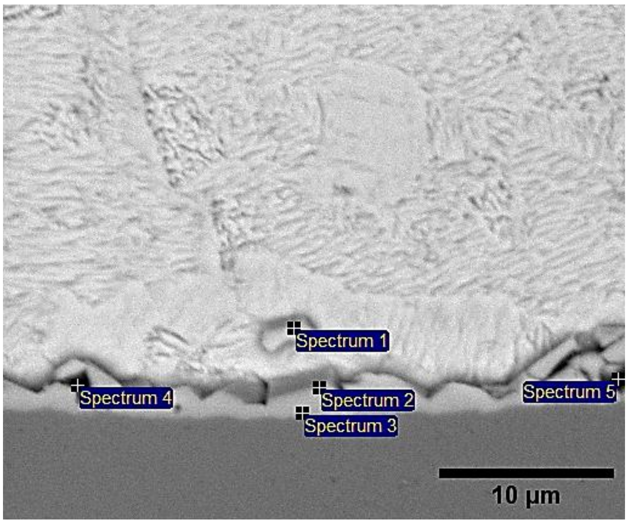


Figure 3. SEM micrographs of the intermetallic layer between Sn-0.7Cu-xGNS solders and Cu substrates (a) Sn-0.7Cu; (b) Sn-0.7Cu-0.02GNS; (c) Sn-0.7Cu-0.04GNS; (d) Sn-0.7Cu-0.06GNS; (e) Sn-0.7Cu-0.08GNS; (f) Sn-0.7Cu-0.10GNS

As observed, a typical wave - serrated Cu_6Sn_5 intermetallic layer was formed at the solder/Cu interface. ImageJ software was used to measure the thickness of the intermetallic layer. The analysis showed that the thickness decreased by 64.62% with an increase in GNS content up to 0.1 wt.%. The presence of GNS in the solder leads to a reduction in the thickness of the IMC layer due to several factors. The high specific surface area of GNS inhibits the diffusion of metal atoms by acting as a barrier to the diffusion of Sn and Cu atoms. Additionally, due to their low density and tendency to agglomerate, GNS accumulate at the solder/substrate interface, further limiting the diffusion of Cu atoms from the substrate. As a result, the reduced diffusion of Cu slows down the formation and growth of the IMC layer at the solder/substrate interface. This is consistent with the results that have been reported in the literature [10, 11]. The presence of the Cu_6Sn_5 intermetallic phase (spectrum 1-3) was confirmed by EDS analysis, as shown in Figure 4. Furthermore, the detection of carbon at the solder/substrate interface (spectrum 4 and 5) suggests that GNS serves as a diffusion barrier for Cu and Sn atoms, leading to the formation of a thinner intermetallic layer.



Spectrum	C (wt.%)	Cu (wt.%)	Sn (wt.%)
Spectrum 1	/	35.28	64.72
Spectrum 2	/	40.57	59.43
Spectrum 3	/	43.48	56.52
Spectrum 4	82.32	12.17	5.51
Spectrum 5	87.15	7.14	5.71

Figure 4. EDS analysis of the intermetallic layer between Sn-0.7Cu-0.1GNS solder and Cu substrate

4. CONCLUSION

In this study, Sn-0.7Cu-xGNS (x = 0, 0.02, 0.04, 0.06, 0.08, and 0.1 wt.%) nanocomposite solders were successfully synthesized using the powder metallurgy technique. The experimental results show that increasing the GNS content up to 0.1 wt.% improves wettability and significantly reduces the thickness of the Cu_6Sn_5 intermetallic layer formed at the solder/substrate interface. These improvements promote the formation of a wider and more stable solder joint, leading to enhanced mechanical reliability of the connection.

ACKNOWLEDGEMENTS

The research presented in this paper was done with the financial support of the Ministry of Science, Technological Development and Innovation of the Republic of Serbia, with the funding of the scientific research work at the University of Belgrade, Technical Faculty in Bor, according to the contract with registration number 451-03-137/2025-03/200131.

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