EFFECTS OF SINTERING TEMPERATURE ON THE PHYSICAL AND MECHANICAL PROPERTIES OF INJECTION-MOLDED COPPER/GRAPHENE COMPOSITE

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Article History: Received 26 October 2017; Revised 5 January 2018; Accepted 15 May 2018

ABSTRACT: The low thermal resistance of graphene nanoplatelets (GNps), which results in high thermal conductivity, is a promising behavior for thermal management applications, such as heat sinks, in electronic devices. In this study, a Cu/GNps composite was produced by powder injection molding (PIM). A suitable sintering temperature in PIM process is essential to be obtained with the intention of achieving the optimum physical and mechanical properties of the Cu/GNps composite. Therefore, the effects of the sintering temperature on the mechanical and physical properties of Cu/GNps were investigated. The Cu/GNps composite was prepared with a binder system, and later, such a system was removed during the debinding prior to sintering. The effects of sintering temperatures of 950°C, 1000°C and 1050°C on the physical and mechanical properties of Cu/GNps were examined. It was found that the highest density and tensile strength were obtained when the Cu/GNPs was sintered at 950°C.

KEYWORDS: Copper; Graphene; GNps; Sinterin; Powder Injection Moulding

1.0 INTRODUCTION

GNps are carbon nanoparticles with platelet-shaped stacks of graphene sheets. Such platelets provide a low thermal resistance, thereby resulting in high thermal conductivity which is desirable for thermal management applications, such as heat sinks, in electronic devices. GNps have the capacity to be used in the production of electronics and composites [1-2]. GNps are also suitable for use as fillers in composites because they need to be well-dispersed because large agglomerations are difficult to control. The density and strength of such composites need to be optimized in order to obtain a higher thermal conductivity. The common materials used for heat sinks are aluminium and copper [3]. These days, copper is gaining much attention as it provides a higher thermal conductivity compared to aluminium. Copper has also been widely used in many studies and applications because of its ease of fabrication [4]. Therefore, studies on the fabrication of a graphene filler into the Cu matrix have been reported in recent years where such combination seems promising and has a high potential to produce a composite with multiple properties [4–13].

Due to advanced technology, the demand for complex and small parts of components is increasing rapidly [14-15]. The reliability of an electronic component is closely related to the temperature, which is an important factor in thermal management where one of the common components in thermal management is heat sinks [16-17]. In order to meet the current demand, the mass production of heat sinks is highly preferred. Therefore, powder injection molding (PIM) is a suitable candidate due to its capability to produce parts with complex shapes at a high volume and level of precision [18-19]. PIM is also a powder metallurgy process that enables the final components to have the desired high density at the end of the process [20]. The main processes in PIM are mixing, injection, debinding and sintering [21-24]. Based on these processes, sintering plays a major character in achieving the anticipated mechanical and physical properties of the sintered part. In this work, powder injection molding (PIM) was used to fabricate the Cu/GNps composite due to its capability at economic mass production.

Sintering was the main focus in this study as the sintering phase is the final bonding phase of the particles. The main parameters of sintering are the heating temperature, heating rates, holding time and cooling rates. During sintering, the powder particles are bonded together by diffusion, where the diffusion itself is reliant on the sintering temperature and also the time or duration of the sintering process [25]. Solid state diffusion is important to the growth of the inter-particle bonding. The gas environment during sintering is also important to ensure the samples are sintered in a safe and suitable condition [26]. Argon gas is used due to its inert properties and low carbon content. Therefore, it was necessary to determine the suitable sintering temperature in order to achieve the optimum physical and mechanical properties of the Cu/GNps composite. The goal of this study was to obtain a composite with good grain growth, less porosity, high density and high tensile strength from the choice of the optimum results for sintering temperatures of 950°C to 1050°C.

2.0 METHODOLOGY

2.1 Materials

Cu powder, with an average particle size of 22 μ m, was supplied by Sandvik Materials Technology. Meanwhile, GNps (XGnP-M grade), with an average particle size of 15 μ m, was supplied by XG Sciences, Inc., and was used as the filler. The feedstock composition was 57.5 vol.% Cu, 0.5 vol.% GNps and 42 vol.% binders. The binder system consisted of 73 vol.% of polyethylene glycol (PEG) with 1.21 g/cm³ density, 25 vol.% of polymethyl methacrylate (PMMA), with 1.16 g/ cm³ density, and 2 vol.% of stearic acid (SA) with 0.96 g/cm³ density. The field emission scanning electron microscope (FESEM) image of the Cu powder is shown in Figure 1. It showed that the particles in the Cu powder had a spherical shape, while the FESEM image of the GNps powder is shown in Figure 2, where the thickness of the GNps sheets was 6~8 nm.



Figure 1: FESEM of as-received Cu powder



Figure 2: FESEM of as-received GNps

2.2 Powder injection Molding Process

A Cu/GNps composite was prepared accordingly by mixing, injection, debinding, and sintering. Density and tensile tests were conducted to determine the physical and mechanical properties of the sintered Cu/GNps parts. It was reported that GNps strengthened the composite and demonstrated outstanding tribological properties [27]. Sonication and stirring methods were used to disperse the GNps, where the GNps were sonicated at 55°C for 1 hour in distilled water, followed by stirring together with PEG at the same temperature for 21 hours using a magnetic stirrer. The GNps/PEG mixture was then mixed with Cu powder and a binder at 150°C for 1 hour using a Brabender mixer. Figure 3 shows the FESEM image of the Cu/GNps feedstock that was homogeneously mixed.



Figure 3: FESEM of the feedstock

The feedstock was then injected into a tensile bar using Xplore injection molding. The mold temperature, injection temperature, injection pressure and holding time were 40°C, 150°C, 9 bars, and 7s, respectively. The injected part was known as the green part. The green part was then subjected to solvent and thermal debinding to remove the binder prior to sintering. Distilled water was used to submerge the green part, and the solvent was debound for 7 hours in an oven. For the thermal debinding, the same green part was placed in a Quartz furnace at 450°C for 2 hours and 45 minutes. Figure 4 shows the double-phased graph of the thermal debinding and sintering phases. The heating rate and holding time for the thermal debinding were 3.5°C/min and 45 minutes, respectively. Once the binder was removed from the green part, it was known as the brown part. Finally, the brown part was sintered in an argon environment at different temperatures, such as

950°C, 1000°C and 1050°C, using a Quartz furnace. The heating rate and holding time were 1°C/min and 2 hours, respectively. The sintered part was known as the final part.



Figure 4: Double-phased graph of thermal debinding and sintering

3.0 RESULTS AND DISCUSSION

Different sintering temperatures were used to determine the effects of sintering at such temperatures in enhancing the mechanical properties and morphology of the sintered Cu/GNps parts. Figure 5 shows the brown part which was the final part before the sintering of the Cu/GNps, where the particles were not bonded together and had a high porosity which resulted in low tensile strength. The density and tensile strength for such part were 5.630 g/cm³ and 23.651 MPa, respectively.



Figure 5: FESEM of the brown part

Figure 6 illustrates the sintered surface of the Cu/GNps parts at various sintering temperatures such as (a) 950°C, (b) 1000°C and (c) 1050°C, respectively. From Figure 6(a), it can be seen that the Cu/GNps was well-sintered where the grain growth was uniform, and the grains were well-bonded with each other. When the sintering temperature was increased to 1000°C, some pores were observed, as shown in Figure 6(b). Such pores resulted in an uneven grain growth. Finally, as shown in Figure 6 (c), defects were formed on the surface of the Cu/GNps sintered at 1050°C, although the grains were well-bonded.



Figure 6: FESEM of sintered Cu/GNps parts at several sintering temperatures: (a) 950°C, (b)1000°C and (c) 1050°C

The graph of the density at different sintering temperatures is shown in Figure 7. It shows that as the sintering temperature increased, the density of the sintered Cu/GNps decreased. The density is defined by how tightly the particles are bonded to each other [28]. In addition, due to the formation of pores, as observed in Figure 6(b), it was proven that the sintering temperature of 950°C resulted in the highest density as no obvious pores were observed. Therefore, diffusion occurred successfully during this sintering temperature where the particles grew closer to each other and reduced or prevented the occurrence of porosity between the particles. As the temperature increased, the density decreased because the particle size had already accomplished the maximum sintered density before 1000°C as small particle bulks had an outsized mass carriage that reduced porosity [29]. Effects of Sintering Temperature on The Physical and Mechanical Properties of Injection-Molded Copper/Graphene Composite



Figure 7: Density of sintered part at different sintering temperatures

In addition, Figure 8 shows the results of different sintering temperatures on the tensile strength of Cu/GNps, where a similar trend as shown during the density test, was observed. The presence of porosity can be determined by the fracture surface obtained from the tensile test. Figure 9 shows the fracture surface of sintered Cu/GNps at various sintering temperatures, such as (a) 950°C, (b) 1000°C and (c) 1050°C. Porosity occurred in all the images. Although no porosity was observed on the surface of the sintered Cu/GNps at 950°C, it could be concluded that porosity still occurred and it was difficult to control during the sintering process [28]. Too much porosity is undesirable because it will lead to poor physical and mechanical properties [29]. In addition, it could be seen that the degree of severity of the porosity was enlarged as the temperature increased from 950°C to 1050°C. Therefore, the sintering temperature of 950°C appeared to be the most suitable temperature to obtain a Cu/GNps composite with a high density and tensile strength. In addition, the value of the tensile strength measured at a sintering temperature of 950°C was near to the theoretical value. The sintering temperature of 1050°C might have been too high for Cu because the melting temperature of Cu was 1086°C. A sintering temperature that is too high might lead to the formation of bloating and cracks on the final part.

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Figure 9: Fracture surface of the final sintered parts at different sintering parameters: (a)950°C, (b)1000°C and (c) 1050°C

4.0 CONCLUSION

In conclusion, Cu/GNps composite has been successfully fabricated by the PIM method. The influence of different sintering temperatures on the density and tensile strength is also investigated. Cu/GNps composite has the highest density and tensile strength when it is sintered at 950°C. Such sintering temperature also leads to better grain growth during the sintering of the Cu/GNps composite where less porosity was observed.

ACKNOWLEDGMENTS

The authors would like to thank the Universiti Kebangsaan Malaysia for the financial support (TRGS/2/2014/UKM/02/4/1) and (FRGS/1/2013/TK04/UKM/01/2).

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