IOP Conf. Series: Materials Science and Engineering **452** (2018) 022056 doi:10.1088/1757-899X/452/2/022056

Preparation of Porous Diamond Preforms for Infiltrating Aluminium Alloy

Caiyu Guo[1, *], Long You[2], Lin Chen[1], Shubin Ren[3] and Xinbo He[3]

[1]Beijing National Innovation Institute of lightweight Co. Ltd., Beijing, 100083, China
[2]Henan Engineering Research Center for Wear Of materials, Zhengzhou, 471000, China
[3]Institute for Advanced Materials and Technology, University of Science and Technology Beijing, 100083, China

*Corresponding author e-mail: 15101010588@163.com

Abstract. Diamond/Al composites with high volume fraction of diamond particles possess high hardness and so they are difficult to be machined subsequently. The near-net-shape preparation of diamond/Al composites can be successfully realized by combined process of diamond preforms by powder injection molding and then aluminium alloy infiltrating the preforms. The process of diamond preforms prepared by powder injection molding was studied, which involved the choice of the binder, the injection molding process and the sintering process. In the experiment, Cu was coating on diamond powder and then sintering diamond preforms with Cu-coated to obtain the strength for the handling operation. Research results showed that, the feedstocks had excellent rheological properties and higher solid volume fraction could be obtained, when the binder composed of 60% paraffin, 30% high density polyethylene and 10% stearic acid as the carrier was used in the powder injection molding. In powder injection molding, the best injection temperature was 160~170°C and the best injection pressure was 70~80MPa. When the diamond preforms were sintering at 950°**C** for 35min, its strength could reach 8MPa and open porosity was close to 100%.

1. Introduction

With the ceaselessly improvement of the current high-power microwave devices, the quantity of heat generated by the unit area of the chip rapidly increases. Package materials require a higher thermal conductivity [1-4]. Diamond-reinforced aluminium matrix composite is an ideal heat sink material, which possesses excellent properties and combines high thermal conductivity and low expansion coefficient of diamond and low weight and adjustable coefficient of thermal expansion of aluminium alloy [5-7]. The diamond/Al composites mainly depend on the high thermal conductivity of diamond, so the volume fraction of diamond particles in the diamond/Al composite is required to be as high as possible, usually more than 55vol%. Therefore it is difficult to be used in the future mechanical processing due to higher brittleness and hardness, which greatly limits its wide application in practical engineering [8, 9]. So aiming at the technical problems of composite materials, the authors put forward to adopt the powder injection molding process to prepare the diamond preforms, and then infiltrate diamond preforms with the aluminium alloy to realize the integrated shaping of diamond/Al composite devices, which can avoid or reduce the mechanical processing later. This paper focuses on the related process of diamond preforms with Cu-coated prepared by powder injection molding, including the determination of loading capacity, the rheological properties of feedstocks, the effect of injection process on the quality of injection diamond preforms and the effect of subsequent sintering process on the strength and pore structure of diamond preforms.

2. Experiment

The diamond powder used in the experiment is commercially available on MBD6 level and the content of N is 250ppm. Considering the effect of diamond particle size on the thermal conductivity of the composites, the diamond particle size was used to be about 80~120μm, which is widely reported in the literature [4, 6]. Because it was difficult to sinter to be formed by no layer on the diamond surface, which led to that the porous diamond preforms without strength could not be carried on the handling operation, we adopted the process of plating Cu on diamond powder and then sintering. The process could effectively improve the interface bonding between the diamond and diamond in diamond preforms, and greatly be convenient for infiltrating aluminium alloy. In this experiment, plating was carried out by using Cu layer with certain thick (about 0.5~2μm) on diamond prowers, which could be used as binder agent among the diamond particles. The Cu layer would be integrated with the matrix aluminium alloy, so it would not affect the thermal property of the composites. Fig. 1 (a) showed the optical microstructures of diamond particles without coating. Fig. 1 (b) showed the optical microstructures of diamond particles with Cu coating a. It could be seen that the coatings were both uniform and compact.

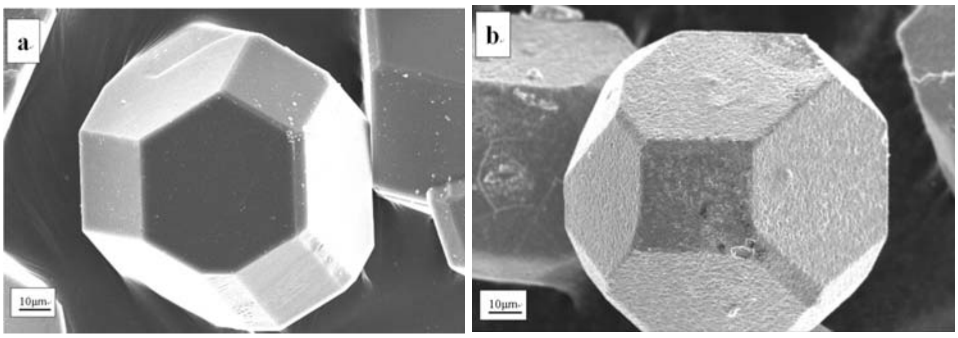

Figure 1. Optical microstructures of diamond powder
Without coating (a) and with Cu-coated (b) a

Table 1. Compositions of binder

Binder number (Feedstocks number)	Chemical composition (wt/%)			
	Paraffin (PW)	High-density polyethylene (HDPE)	Polypropylene (PP)	Stearic acid (SA)
F1	60	0	25	5
F2	60	30	0	10
F3	60	15	15	10

The diamond powdesr with Cu coating and injection molding binder were mixed as feedstocks. Three kinds of binder were used in this study as shown in Table 1. Through the investigation of the rheological behavior of feedstocks, the best composition of binder was confirmed. The diamond preforms were prepared with the best composition of binder by injection molding, and then the binder inside the diamond preforms was removed. Finally the diamond preforms get a certain strength by sintering at a certain temperature and the open porosity was closed to 100%.

The viscosity of feedstocks was measured by capillary rheometer. Scanning electron microscopy (SEM) was applied to examine the microstructures of the diamond particle and diamond preforms, which was carried out in a LEO1450 scanning electron microscopy. The porosity of diamond preforms was measured by drainage method.

3. Results and Discussions

3.1. Determination of diamond powder loading

In the process of preparating diamond performs by injection molding, the volume fraction of diamond powder in the feedstocks was the same as the volume fraction of diamond powder in the composite. As the volume fraction of diamond particles in the composites was required to be as high as possible, that is to say the volume fraction of diamond particles in the feedstocks (loading) was required to be as high as possible. But excessive loading (the volume fraction of diamond particles in the feedstocks) would reduce the viscosity of the feedstocks, which resulted in the separation between diamond powders and binder, and even was easy to generate many micro poroes and cracking. Through establishing the relationship between density and volume fraction for various feedstocks with different binders, the critical loading of fixed diamond particle size could be determined [10]. Fig.2 showed the relationship between density and volume fraction for various feedstocks with different binders in Table 1. It could be seen the effect of binder composition on the loading was significant. When F2# was used in the process, the loading of diamond performs could reach 66%; when F1# was used in the process, the loading of diamond performs could reach 64%. From Fig.2, we could also see the experimental density of feedstocks deviated from the theoretical density when the loading was excessive, which indicated there had been separation between diamond powders and binder and micro pores. The effect of binder composition on the loading is mainly related to the characteristics of the binder. High electrostatic polypropylene was easy to aging when it contacted with the Cu layer on diamond surface, which resulted in a change in its molecular structure and then made the contact force weak among the diamond powders and generated pores. But the high density polyethylene has good chemical stability, so it could increase the loading. We could see the feedstocks F2# was the most suitable as the organic carrier in diamond powder injection molding.

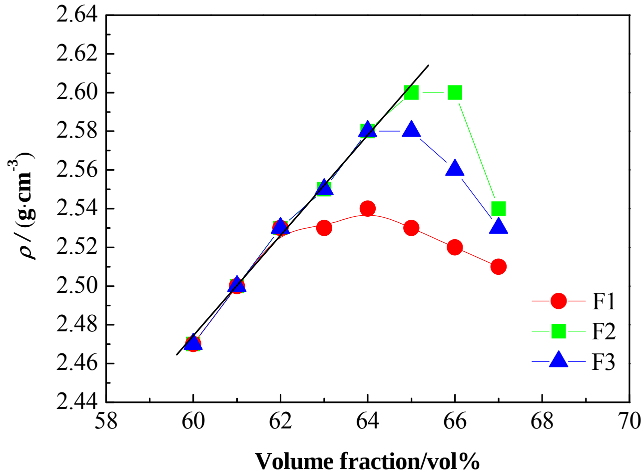

Figure 2. The relationship between density and volume fraction for various feedstocks with different binders

3.2. Effect of injection molding parameters on the quality of diamond perform

Injection molding process parameters directly related to the quality of diamond preforms. Table 2 showed the Effects of injection molding parameters on the quality of diamond preforms. It could be seen it was better complete the process of filling die and the surface of diamond preform was smooth.

However if the temperature and pressure were too low, it would increases the viscosity and be difficult to fill the cavity; inner pores and the flash could be caused by too high temperature and pressure

Table 2. Effects of injection molding parameters on the quality of diamond preform

Injection temperature	Injection pressure (MPa)			
	60	70	80	90
150	Not filled	some filled	some filled	some filled
160	Not filled	filled	filled	filled
170	Not filled	filled	filled	surface sink
180	some filled	flash	flash	inner pores

3.3. Effect of sintering process on the quality and porosity of diamond perform

After thermal debinding of the diamond preforms at 520°C, the binder had been completely cleared. But the strength was so low that it could not be carried for operation. In order to improve the strength of diamond preforms for operation, the diamond particles were coated Cu (0.5~2μm) by chemical plating. It should be noted that the sintering temperature should not be higher than 1083°C. So in this study, the sintering temperature was 950°C. In order to obtain the high strength and high precision of porous diamond preforms in a short sintering time, the effect of the thickness of Cu layer and the holding time on the strength and porosity of the diamond preform was studied in the experiment. Fig.3 showed the effect of the thickness of Cu layer on the strength and porosity of the diamond preform at 950°C for 35min. It could be seen the strength of the diamond preform increased with the increase of thickness of Cu layer. When the thickness was less than 1μm, the opening porosity was close to 100%. When the thickness was more than 1μm, the opening porosity sharply decrease and the precision of porous diamond preforms was low. When the thickness was 2μm, there was serious deformation of the diamond preforms.

Fig.4 showed the morphology of sintering diamond preforms with 0.5μm and 2μm Cu coating. We could see there was bonding among diamond particle with Cr coating due to Cu coating and open pore network was basically formed. When the thickness was thicker, the melting phenomenon of copper between diamonds particles was more obvious, which led to the formation of closed pores and the decrease of porosity.

Fig.5 showed the Correlation between strength and sintering time for porous diamond performs with 0.5μm Cu coating at 950°C. It could be seen the strength would gradually increase with the holding time extended. But the holding time was too long and it was easy to cause the decline of diamond performance and the deformation of diamond preforms. Fig.6 showed the microstructure of the preform for 45 minutes. It could be seen that the connection area of Cu among diamond particles increased. In addition, the deformation was also more serious.

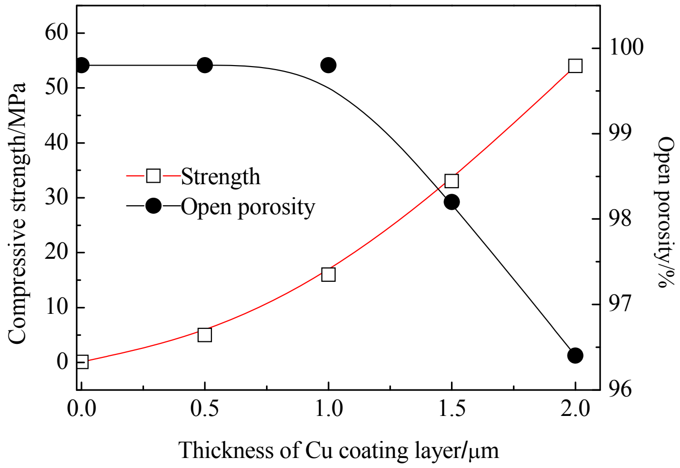

Figure 3. Density and porosity dependence of Cu layer thickness of Cr-coated and Cu-coated secondly diamond

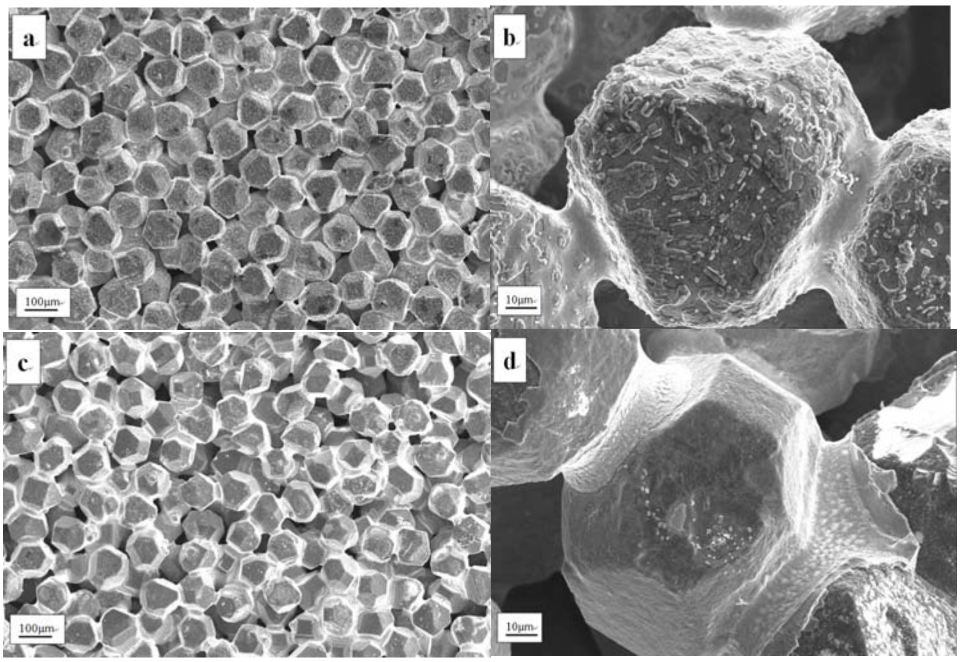

Figure 4. The morphology of sintering diamond preforms with 0.5μm and 2μm Cu coating a: Cu-0.5μm; b: magnified morphology in Figure (a); c: Cu-1μm; d: magnified morphology in Figure (c)

In consideration of open porosity, strength and size precision of the preform, the best sintering process could be determined. The best sintering temperature was 950°C and the best holding time was 35min. When the thickness of Cu plating was 0.5~1μm, the strength of the preform reached 8Mpa and open porosity was close to 100%.

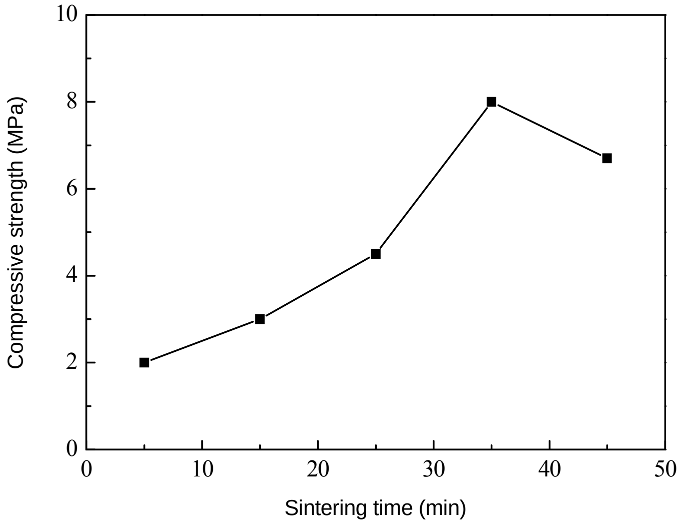

Figure 5. Correlation between strength and sintering time for porous diamond performs

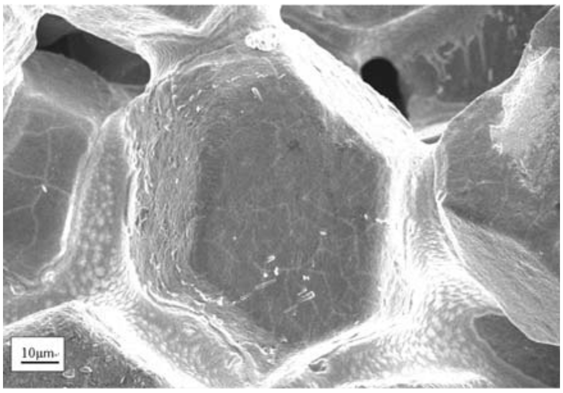

Figure 6. Microscopic morphology of diamond preforms by sintering for 45min

4. Conclusion

1) The effect of binder composition on the loading in the powder injection molding is mainly related to the characteristics of the binder. The loading of diamond performs could reach 65%, when the binder composed of 60% paraffin, 30% high density polyethylene and 10% stearic acid were used. The feedstocks composed this kind of binder and diamond powder were suitable as an organic carrier in injection molding.

2) Using the binder of 60% paraffin, 30% high density polyethylene and 10% stearic acid, it was better complete the process of filling die at 160 ~170°C and at 70~80Mpa and the surface of diamond preform was smooth when the loading was 66%. At too low temperature and pressure, it would be difficult to fill the cavity; inner pores and the flash could be caused by too high temperature and pressure.

3) In consideration of open porosity, strength and size precision of the preform, the best sintering process could be determined. The best sintering temperature was 950°C and the best holding time was 35min. When the thickness of Cu plating was 0.5~1μm, the strength of the preform reached 8Mpa and open porosity was close to 100%.

Acknowledgments
This work was supported by the Open Science and Technology Cooperation Project of Henan Province.

References

[1] Ruch PW, Beffort O, Kleiner S, Weber L and Uggowitzer PJ: Selective interfacial bonding in Al (Si)-diamond composites and its effect on thermal conductivity. Compos Sci Technol. 2006; 66 (15): 2677 - 2685.

[2] German RM, Hens KF and Johnson J L: Powder metallurgy processing of thermal management materials for microelectronic applications. Int J Powder Metall. 1994; 30 (2): 205 - 215.

[3] Beffort O, Khalid F A, Weber L, Ruch P, Klotz U E, Meier S and Kleiner S: Interface formation in infiltrated Al (Si)/diamond composites. Diam Relat Mater. 2006; 159 (9): 1250 - 1260.

[4] Weber L and Tavangar R: On the influence of active element content on the thermal conductivity and thermal expansion of Cu–X (X = Cr, B) diamond composites. Scripta Mater. 2007; 57 (11): 988 - 991.

[5] Yoshida K and Morigami H: Thermal properties of diamond/copper composite material. Microelectron reliab. 2004; 44 (2): 303 - 308.

[6] Yang ZL, Wang LG, Wang LM, He XB, Qu XH, Liu RJ, Hu HF: Microstructure and graphitization behavior of diamond/SiC composites fabricated by vacuum vapor reactive infiltration. Rare Metals. 2015; 34 (6): 400 - 406.

[7] Ekimov EA, Suetin NV, Popovich AF, Ralchenko VG: Thermal conductivity of diamond composites sintered under high pressures. Diam Relat Mater. 2008; 17 (4): 838 - 843.

[8] Schuberta T, Ciupińskib Ł, Zielińskib W, Michalskib A, Weißgärbera T, Kiebacka B: Interfacial characterization of Cu/diamond composites prepared by powder metallurgy for heat sink applications. Scripta Materi. 2007; 58 (4): 263 - 266.

[9] Mizuuchi K, Inoue K, Agari Y, Morisada Y, Sugioka M, Itami M, Kawahara M, Makino Y: Thermal conductivity of diamond particle dispersed aluminum matrix composites produced in solid-liquid co-existent state by SPS. Compos: Part B. 2011; 42 (5): 1029 - 1034.

[10] Abolhasani H, Muhamad N: A new starch-based binder for metal injection molding [J]. J Mater ProcessTech. 2010; 210 (6): 961 - 968.