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Enhancing densification capacity and properties of Al/diamond composites by partial liquid hot pressing

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ABSTRACT

Al/50 vol% diamond composites are fabricated by hot pressing with the assistance of partial liquid phase for favoring densification through the addition of elemental copper. Copper is introduced by pure powder and secondary deposited coating (a tungsten based layer is coated directly on the surface of diamond particles), respectively. The results show that density, bending strength and thermal conductivity of Al/diamond composites are improved when the liquid phase forms homogeneously in the matrix. As copper powder is added, the liquid phase spreads through the particle boundaries under the applied press during sintering, finally fills the interfaces and voids. While as copper coating is conducted, a better densification capacity and more enhanced properties of the composites are received. Moreover, a theoretical model is proposed to estimate the effect of different copper addition routes on thermal conductivity of the composites, and the results show a good agreement with the experimental data.

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1. Introduction

In the past few years, aluminum based composites, particularly reinforced with high volume fraction of diamond particles (above 50 vol%), have attracted great attentions as thermal management material for electronic packaging applications [1–3], such as thermal dispersion for super luminescent diodes [4–6]. The Al/diamond composites show attractive performances, especially the combination of high thermal conductivity, tailorable coefficient of thermal expansion (CTE) and low density [2,7]. Nevertheless, the poor interfacial wettability between aluminum and diamond greatly decreases the mechanical and physical properties of Al/diamond composites. Previous studies indicated that surface modification of reinforcement with a metal coating can effectively improve the interfacial wettability between reinforcement and metal matrix, and then enhance the properties of consolidated composites [8–11]. Moreover, matrix alloying with carbide-forming elements, such as Ti [12], Cr [13], and Zr [14], is also introduced to improve the wettability between diamond and metal matrix.

Recently, various methods are applied for the preparation of Al/diamond composites, e.g., squeeze casting infiltration, pressure or pressureless infiltration and spark plasma sintering [3,7,15–17]. Although the infiltration process can obtain Al/diamond composites with high volume fraction, Al_4C_3 phase is unavoidably formed at the diamond/molten aluminum interfaces as a result of the high temperature (above the melting point of aluminum) [2]. The formed carbide has detrimental effects on properties of the composite, such as brittleness, unstableness and poor thermal conductivity. Spark plasma sintering (SPS) has become a prevalent method of fabricating Al/diamond composites to avoid the formation of Al_4C_3 [18,19]. However, the insufficient interface diffusion and poor interfacial bonding always existence due to the very short holding time, resulting in low thermal conductivity of the composites [20]. Therefore, further studies concerning the densification and thermal conductance should be carried out to improve the performance of Al/diamond composites.

To facilitate the application of Al/diamond composites, a fabrication method with reduced cycle time and cost is also demanded. Powder metallurgy (PM) technique can be a feasible route, especially via cold pressing followed by pressure or pressureless sintering. Additionally, composites reinforced with a wide range of volume fraction and uniform distribution of reinforcement can be achieved by PM method. In this respect, the hot pressing (HP) process, which involves relatively low sintering temperature, low cost and high productivity, provides some opportunities for economically fabrication of high performance Al/diamond composites [20,21].

During the progress of conventional hot pressing at solid state, the deleterious reaction between reinforcement and metal matrix can be well minimized. However, the composite powder with a high volume fraction of reinforcement have low densification capacity, thus the thermal properties of received composites are generally not satisfactory due to the residual porosity. It is expected that a eutectic liquid phase will form during sintering when elemental copper is added into aluminum

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matrix according to the Al-Cu binary phase diagram. This liquid phase improves the densification process as well as decreases the fabrication temperature [22]. In the present contribution, the Al-Cu/diamond composites reinforced with W-coated diamond particles are synthesized by partial liquid hot pressing (PLHP). Copper is added by two methods to achieve different microstructural features. On the one hand, elemental copper powder is mixed into the aluminum matrix mechanically. On the other hand, copper is added in the form of secondary coating on the surface of diamond particles with pre-existed tungsten coating. Density, microstructure, bending strength and thermal conductivity of the hot pressed Al/diamond composites are characterized.

2. Experimental procedure

Commercial gas-atomized aluminum powder (99.95 wt%) and electrolytic copper powder (99.995 wt%) with average size of 75 μm and 45 μm, respectively, were used as the matrix. MBD6-type synthetic diamond particles with an average size of 400 μm (Henan Huanghe Whirlwind Co., Ltd. of China) were utilized as the reinforcement. Two types of metal coating were deposited on the surface of diamond particles, one of that was a tungsten single layer, the other was a W-Cu dual-layers fabricated using a multi-step process. Diffusion method was applied to deposit the tungsten coating on diamond particles (diamond-W). After that, a secondary copper coating was deposited on the tungsten coated diamond to form a W-Cu dual-layers using electroless plating method (diamond-W/Cu). The coating method was described in detail in the previous works [23,24]. The thickness of tungsten coating is about 420 nm. The dual-layers coated diamond particles contain 3.6 wt% of copper, analyzed by inductively coupled plasma (ICP), and the coating thickness is about 0.9 μm.

Firstly, composite powder of PLHP-1#, PLHP-2# and PLHP-3# according to Table 1 was mixed mechanically, respectively. Secondary, the mixed powder was carefully transferred into a carbide mold (ø10 mm) to avoid separation of the reinforcement and matrix. A pressure of 320 MPa was applied on the powder and held for 20 s. Green compacts with a relative density of about 76% were obtained. Thereafter, the green compacts were consolidated in a graphite mold by hot pressing at 600 °C under a pressure of 30 MPa for 1 h in nitrogen atmosphere. The hot pressed samples were cooled in furnace with pressure until 200 °C.

The densities of as-received composites were measured by the Archimedes method. The microstructure and fracture surface of the composites were examined by scanning electron microscope (SEM, FEI SIRION 200) and energy dispersion spectrum (EDS). The elemental distribution in the composite samples was analyzed using an electron probe micro-analyzer (EPMA, JXA-8230). The three-point bending strength of composites was measured on the specimens with a dimension of 3 mm × 10 mm × 50 mm. The bending tests were carried out with an initial strain rate of 10^{-4} s^{-1} using Instron MTS 850 materials testing system. The thermal diffusivities and specific heat of the composites were measured by a heat flux technique using a JR-3 laser flash thermal analyzer. The thermal conductivity (λ) was calculated as the product of the density (ρ), thermal diffusivity (α) and specific heat (C_p), $\lambda = \alpha \cdot \rho \cdot C_p$.

Table 1
Composition of the Al/diamond composites in the present work.

Sample	Matrix	Reinforcement
PLHP-1#	Al	50 vol% diamond-W
PLHP-2#	Al-4wt%Cu	50 vol% diamond-W
PLHP-3#	Al	50 vol% diamond-W/Cu

3. Results and discussion

3.1. Microstructural characteristics

The morphologies of copper powder, original diamond particles, tungsten coated and W-Cu dual-layers coated diamond particles are shown in Fig. 1a to f, respectively. It can be observed that the diamond surface is evenly and entirely coated by single layer or dual-layers. The metal layers are formed by submicron particles, which lead to the smooth surface of diamond particles become somewhat rough. The outer layer is tightly absorbed on tungsten particles because the copper is deposited in the form of atoms. From the previous work [20,21], it is known that the tungsten carbide is directly formed during the deposited process of the layer. The tungsten layer structure coated on diamond consists of WC, W_2C, and W, from inside to outside respectively.

Fig. 2 shows the microstructures of the hot pressed Al/diamond composites with different surface modifications of diamond particles. It has been known that the surface modification of diamond with tungsten coating can greatly reduce the porosity in composite owing to the wetting ability between diamond and matrix is significantly increased [21]. However, as seen from Fig. 2a (PLHP-1#), poor junction presents between the diamond particles and the aluminum matrix, and small residual pores are also visible in the matrix. The high magnified image (Fig. 2b) shows that the interface gap is existence between the diamond and the matrix. It can be seen from Fig. 2c (PLHP-2#) that almost no porosity is presented in the matrix, but little gaps are formed when the two diamond particles contact with each other. The high magnified image of the interface between diamond and matrix (Fig. 2d) shows that a lighter contrasted phase is formed. This phase is identified as Al_2Cu precipitated at the interface according to the EDS results, which formed during cooling of the liquid Al-Cu eutectic phase. While, no pore and defect is observed in the composite and the interface between diamond and aluminum are well connected in the sample PLHP-3# (Fig. 2c and f). This result suggests that nearly full dense Al/diamond composite is obtained with dual-layers coating diamond particles using hot press sintering.

Ogel and Gurbuz [25] reported that the densification of pure aluminum powder by hot pressing was not successful, even increasing the sintering temperature up to 600 °C, due to the low diffusion rate at solid state. Moreover, the presence of surface oxide layer on aluminum powder also inhibits the inter-particle diffusion. It is suggested that the aluminum matrix composite reinforced with 50 vol% diamond is hard to be densified, hence, the residual pores and gaps are observed in the sample PLHP-1#. The studies of Tan et al. [17] concluded that the strong interfacial bonding was not formed in the Al/diamond composite until the sintering temperature increased to 650 °C. This is an indicative of pores and gaps form between the diamond and the aluminum matrix.

According to the Al-Cu binary phase diagram, the eutectic invariant lies at 548 °C. When the sintering temperature heats to 548 °C, partial melting occurs at the contacted areas between the aluminum powder and the copper powder due to mutual diffusion. With the applied pressure, the liquid phase spreads through the boundaries among the powder by a capillary force, and fills pores and voids during the sintering process. Additionally, with the presence of liquid phase in the composite powder, lower applied pressure is needed for the densification process. The results of Kim et al. [22] suggested that 4–10 wt% Cu addition into the aluminum matrix can form a persistent liquid phase during sintering. Accordingly, a persistent phase is more effective for improving the densification ability of the composite powder containing diamond particles. This phenomenon should ascribe to the liquid phase penetrate the boundaries and glue the particles before disappears into the aluminum matrix. Compares to Fig. 2b, a significantly better interface bonding is observed in Fig. 2d and f, indicating the copper addition can be employed to form a persistent liquid phase in the powder densification process, and then increase the sintering rate. Moreover, owing to the applied pressure, the pores formed by fast diffusion of copper

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