



# Enhanced thermal conductivity and stability of diamond/aluminum composite by introduction of carbide interface layer



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## ABSTRACT

W-coated diamond/aluminum composites were manufactured by a pressure infiltration method. A continuous and homogeneous carbide coating was formed on the surface of diamond particles, and the selective bonding between the aluminum matrix and different diamond faces was no longer observed. The obtained composites exhibited thermal conductivity as high as  $476 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ . It was attributed to the carbide layer which increased the amount of reactive interfacial bonding and improved the mean interfacial thermal conductance. In addition, compared with the W coated diamond/aluminum composites, the thermal conductivities of uncoated ones were seriously declined by immersing the composites in moisture circumstances. SEM and XRD results indicated that the stabilities of composites thermal behaviors were closely related to the interface of composites. The W carbide layer in the interface region played a critical role in improving the stability of the composite exposure to moist environments.

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## 1. Introduction

The diamond/metal composites have drawn much interest in multi-functional electronic packaging due to their excellent thermal properties, such as low coefficient of thermal expansion and high thermal conductivity [1–5]. Different processes, such as vacuum hot press, spark plasma sintering and pressure infiltration, are introduced to fabricate the composites, and liquid infiltration is considered to be the most efficient process for fabricating the high volume fraction diamond reinforced aluminum matrix composites among all available techniques [6–8].

Unfortunately, the diamond is difficult to be wetted by molten pure aluminum at low temperature, therefore, a minimum external pressure should be applied to complete the infiltration [9]. Although the wettability can be improved at elevated temperature, thermodynamically unstable phase  $\text{Al}_4\text{C}_3$  will be formed on the interface [10]. It tends to react with the atmosphere moisture or water, which results in degradation of the performance of the composites. Therefore, some methods have to be employed not only to improve the wettability between aluminum and diamond but also to suppress the formation of  $\text{Al}_4\text{C}_3$ . The addition of an alloy element is a widely used method to enhance the wettability between preforms and the matrix [11–14]. But the addition of alloying element significantly decreases the thermal conductivity of the matrix. Recent researches indicate that surface modification of particles by a

coating transition element is a direct and more effective method to improve the wettability and thermal conductivity of carbon/metal composites [1,6,15–18]. The diamond/aluminum composites with TiC coating on diamond particles were fabricated by gas pressure infiltration. The TiC layer improved the interface adhesion between the diamond particles and the matrix, and enhanced the performance of the composites [1]. However, these results were obtained in lower volume fraction (30–55 vol.%) diamond composites. The higher volume fraction (>55 vol.%) diamond/aluminum composites fabricated by a liquid infiltration process and the long-term stability of the interface in a moist environment have not been investigated. W and its corresponding carbides have higher thermal conductivity than other commonly used carbide forming elements, and it is well wetted by the molten aluminum. Therefore, tungsten is selected as a carbide forming element in this paper. The purpose of our work is to check experimentally the possibility of obtaining composites with high thermal conductivity and good stability exploiting the liquid infiltration by means of the W coated diamond particles.

## 2. Experimental

### 2.1. Materials

Commercial purity aluminum (99.99% in purity) was used as a matrix material. Cubo-octahedral synthetic diamond crystal particle of the MBD-4 grade was purchased from Zhong Nan Diamond Industries, with a designated average particle diameter about 100  $\mu\text{m}$ . The diamond volume fraction amounted to approximately 62% of all samples,

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corresponding to the tap packed diamond powder density. The W coating on diamond particles was obtained by magnetron sputtering. Sputtering from a tungsten target was performed 120 W in Ar working gas. The sputtering pressure was 1.0 Pa and the sputtering time was 1 h. The particles were continually moved by vibration and translation motions during the deposition process to obtain a homogeneous coating. The thickness of the coating layer was deduced from the mass increase of the coated diamond particles, which is estimated to 200 nm. After the coating process, the diamond particles were annealed in vacuum (better than 1 Pa) at 1273 K for 2 h, both heating and cooling rate were 5 K/min.

## 2.2. Composites preparation

Diamond/aluminum composites were fabricated by a mechanical pressure assisted liquid metal infiltration method. For this approach, a special mold was designed for infiltration and schematically shown in Fig. 1. During the infiltration process, the conical shape of the mold and the fine graphite powder could prevent the molten metal from spilling along the inwall of the die. The composites were fabricated at three different infiltration temperatures, 973 K, 1023 K, and 1073 K respectively, and the heating rates were all 10 K/min. The piston moved downwards to infiltrate the molten aluminum into the diamond powder bed when the infiltration temperature had been reached. The temperature maintained 10 min until the infiltration process was completed. After that, the specimen was furnace-cooled to room temperature.

## 2.3. Characterization and test

X-ray diffraction (XRD) patterns, recorded by a D5000 Siemens diffractometer, were used to investigate the phase composition of the W coated diamond particles before and after heat treatment. FEI QUANTA 200 scanning electron microscopy and energy dispersive spectrometer (EDS) were employed to characterize the morphology of the coating layer and the distribution of the diamond particles as well as the interface area of the composites. The samples for investigating the interface area were prepared by the electrochemical etching method [19]. Absolute ethyl alcohol solution of perchloric acid at concentration of 10 vol.% was used as an electrolyte, while the voltage was 26 V DC and the current density was 1 A/cm<sup>2</sup>. After electrochemical etching, the samples were cleaned ultrasonically in an acetone bath for 10 s immediately, and quickly dried in an infrared furnace at 120 °C for 20 min.

In order to study the influence of a moist environment on the long-term stability of the composites, the composites were immersed in 100 ml deionized water at room temperature for 48 h. The thermal conductivity of the composites was calculated from composite density ( $\rho$ ),

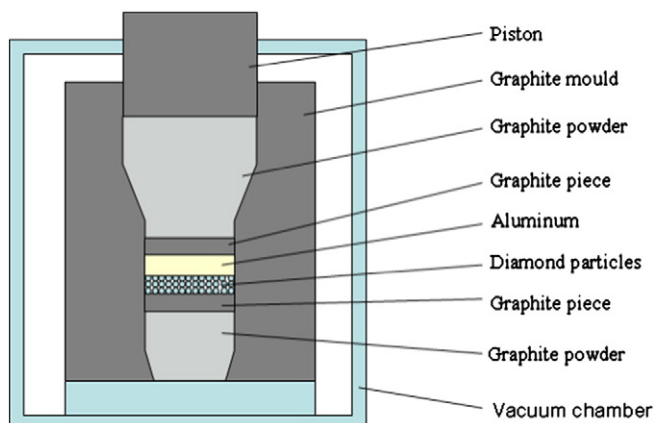


Fig. 1. The set-up for the infiltration experiments.

thermal diffusivity ( $\alpha$ ), and specific heat capacity ( $C$ ), using the equation  $\lambda = \alpha\rho C$ . The room temperature thermal diffusivity ( $\alpha$ ) of the composites before and after immersing were measured by a Flashline™ 3000 thermophysical testing machine. The density was determined by the Archimedes technique. Specific heat capacity was calculated by means of the linear rule of mixture using the values of the different components. The coefficient of thermal expansion was measured on a DIL 402C thermal dilatometer (NETZSCH Co.) from room temperature to 573 K with a heating rate of 5 K/min.

## 3. Results

### 3.1. Microscopy and phase composition of W coated diamond particles

To improve the wettability and inhibit the formation of Al<sub>4</sub>C<sub>3</sub> which results from direct contact with the molten aluminum on the surface of the diamond particles, a method of the carbide forming element tungsten deposited on diamond was adopted. Fig. 2(a), (c) and (e) illustrates the morphologies of the diamond as received, the W coated diamond before and after heat treatment, respectively. It can be seen a continuous and homogeneous coverage layer is deposited on the surface of the particle in Fig. 2(c) and (d). XRD analyses, as shown in Fig. 3, indicates that a carbide layer has not been formed after magnetron sputtering at room temperature, and subsequent heat treatment in vacuum at 1273 K for 2 h causes intensive coating carbidization. Since the carbon of the diamond diffuses into the tungsten coating unidirectionally [20], the coated diamond particle with a diamond–WC–W<sub>2</sub>C core–shell is formatted during the heat treatment process. As seen in Fig. 2(e) and (f), the carbides bond to both the {100} and the {111} surfaces of a diamond. The homogeneous carbide layer is expected to prevent the diamonds from direct contact with molten aluminum, and protect the diamond particles from possible graphitization and formation of undesirable Al<sub>4</sub>C<sub>3</sub>.

### 3.2. Microstructure and interface of composites

The diamond/aluminum composites and the composites reinforced by W coated diamond particles were fabricated under different infiltration temperatures. An electrochemical etching method was used to prepare the samples for microstructure and interface investigation, selective removal of aluminum from the composites. As seen in Fig. 4(g), the distribution of the diamond particles was almost uniform. A near-net-shaped matrix was formed, and no clear pores and cracks were found in the composites. The relative density of all the samples was greater than 99.3%. Generally speaking, the infiltration quality of the composites was fine and the molten matrix metal was able to penetrate the pore of the particles fully under the designed pressure assisted system.

More interfacial information of pure diamond/aluminum composites, fabricated at 1023 K, is presented in Fig. 4(a) and (b). The {100} surfaces show the rough surface topography relative to the {111} surfaces on the uncoated diamond particles due to selective interfacial reaction. The formation of the interfacial phase Al<sub>4</sub>C<sub>3</sub> on {100} surfaces had been identified by P.W. Ruch. A possible explanation for the selective interfacial reaction is that carbon atoms are bonded to the bulk by two carbon–carbon bonds at the {100} surfaces, while the carbon atoms are bonded by three carbon–carbon bonds at the {111} surfaces [21]. Thus, the reactivity of the {100} surfaces is higher, and the reaction rate of diamond {100} surfaces is expected to be greater than that of the {111} surfaces.

In contrast, the tungsten coated one which was also fabricated at 1023 K, as shown in Fig. 4(c) and (d), the difference in morphology on the {111} and {100} surfaces could be ignored. Note that no plate-like interfacial phase was formed on the diamond surface. It might be attributed to homogeneous tungsten carbides, which were formed during the heat treatment. Fig. 5 is a backscatter micrograph of fracture surfaces of

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