



Optimized thermal properties in diamond particles reinforced copper-titanium matrix composites produced by gas pressure infiltration



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ABSTRACT

Interface modification is crucial to exploit high thermal conductive potential of diamond in the metal matrix composites reinforced with diamond particles (Cu/diamond composites). With an attempt to modify the Cu/diamond interface, we add a carbide-forming element of Ti to the Cu matrix and use a liquid-phase processing technique to attain sound interfacial bonding. The Cu-xTi/diamond composites were characterized by using scanning electron microscopy, transmission electron microscopy, and X-ray diffraction. The interface layer is confirmed as TiC, the amount of which increases with increasing Ti concentration in the Cu-xTi alloy matrix. As the Ti concentration increases, the thermal conductivity of the Cu-xTi/diamond composites first increases and then decreases, giving an optimized thermal conductivity of 752 W/m K and a coefficient of thermal expansion of $6.50 \times 10^{-6}/\text{K}$ at $x = 0.5$ wt.%. The results show that an appropriate amount of Ti addition in Cu matrix can enhance the thermal conductivity of Cu/diamond composites.

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1. Introduction

The miniaturization of electronics is urgently demanding packaging materials with superior thermal conductivity. Effective thermal management is fundamental to consistent long-term performance and reliability of electronic devices [1,2]. As a promising candidate, diamond particles dispersed copper matrix (Cu/diamond) composites are attracting more and more interests [3–17]. In preparing Cu/diamond composites, the major restriction originates from the mismatch in thermal expansion coefficient between diamond and copper, which easily induces large thermal stress. In addition, the non-carbide forming nature of copper and the large contact angle between diamond and Cu mean an absence of strong chemical bonds at the interface. Consequently, the diamond particles are inclined to delaminate from the pure copper matrix after cooling down. Poor interfacial bonding becomes a main obstacle to obtaining high thermal conductivity in the Cu/diamond composites. The thermal conductivity of the Cu/diamond composites is reduced even with increasing the amount of diamonds [4].

At present, it is popular to improve the interfacial bonding by introducing a third component between diamond and copper, usually through surface metallization of diamond particles [3,7,8,11,18] or through alloying of copper matrix [6,19,20]. Among broad researches, TiC is suggested to connect the diamond reinforcements with the metal matrix tightly. TiC has a Gibbs free energy of -170.5 kJ/mol [21], which is favorable to its formation at the interface. Through Ti coating on diamond particles, the derived Cu-Ti/diamond composites display a thermal conductivity as high as 630 W/m K, owing to TiC formation at the interface [22]. Nevertheless, the coating on diamond is expensive and demanding. Moreover, the coating is oxidized during high-temperature processing [7]. The presence of oxygen on TiC surface strongly inhibits the interaction between the carbide and the molten Cu matrix, causing non-wetting conditions [23]. By means of alloying Ti to Cu matrix, the diamond particles and the Cu-Ti alloys can react directly, and the formation of oxide layer on the diamond particles is thus avoided. From the above, the route of Cu matrix alloying is proved to be more feasible in the fabrication of Cu/diamond composites.

Here we add Ti to Cu matrix to demonstrate the enhancement of thermal conductivity in the Cu-Ti/diamond composites. Intuitively, the amount of Ti addition should be limited in order to maintain high thermal conductivity in the Cu matrix. Literature

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has not reported detailed information about the effect of alloying element content on the microstructure and thermal properties of the composites. Several groups [20,24] prepare the Cu-Ti/diamond composites using a solid-state sintering route, but obtain relatively low thermal conductivities. A liquid-state processing route of gas pressure infiltration has been successfully utilized to achieve high thermal conductivity in Cu/diamond composites [7,16]. The gas pressure infiltration is capable of realizing dense microstructure and promoting interfacial reaction in the diamond composites. So far the promising route has not been used to prepare the Cu-Ti/diamond composites.

Another difficulty in Cu/diamond composites community comes from delicate characterization of the Cu/diamond interface. Due to huge difference in hardness between diamond and metal, it is extremely tough to mill an eligible specimen for transmission electron microscopy (TEM) observations. For this reason, actual interface structure of the Cu/diamond composites is rarely reported [10,15], which is certainly critical to understand the thermal conducting mechanism involved. Taking advantage of focused ion beam (FIB) technique, we have developed thin foils containing the Cu/diamond interface and obtained the state-of-the-art images depicting carbides at the interface.

In this article, we produce the Cu-Ti/diamond composites by the gas pressure infiltration and investigate the effect of Ti addition on the microstructure and thermal properties with respect to diamond surface state and interface structure of the composites. The effect of interfacial layer thickness on thermal conductivity of the Cu/diamond composites is elucidated. The thermal conductivity and coefficient of thermal expansion (CTE) of the Cu-Ti/diamond composites are correlated with various modeling schemes.

2. Experimental

The Cu-*x*Ti/diamond composites were produced by a gas pressure infiltration method. Fig. 1 shows the schematic drawing of the gas pressure infiltration device. The starting materials were HHD90 synthetic single-crystalline diamond powders with an average particle size of 230 μm (Henan Huanghe Whirlwind Co., China) and Cu-*x*Ti alloys (*x* = 0.3, 0.5, 2.0 wt.%). The Cu-*x*Ti alloys were melted by a vacuum induction route using 99.999 wt.% Cu and 99.99 wt.% Ti bulks (TaiYu Materials Science & Technology Co., China). The infiltration was conducted at 1423 K for 30 min, under an Ar gas pressure of 1.0 MPa. The details of the infiltration are referred to elsewhere [7].

X-ray diffraction (XRD, Rigaku DMAX-RB, Japan) was used to characterize the phase structure of the composites. The polished surface and fracture surface were observed by field emission scanning electron microscope (SEM, ZEISS SUPRA 55, Germany). A dual beam FIB system (FEI Nova 200 FIB, USA) was used to mill the Cu-Ti/diamond samples to thin foils. The FIB-milled samples were characterized using a scanning transmission electron microscope (STEM, JEOL, ARM200, Japan). Besides, the metal matrix was electrochemically etched and the diamond particles were collected to directly characterize the interface structure [25]. The composites were electrochemically etched in a 10 vol.% HNO₃ and 90 vol.% H₂O solution for 5 min. The sample acts as the anode and a steel plate as the cathode. The current density was controlled by a direct current power supply and the current was fixed at 1 A. After electrochemical etching, the samples were cleaned ultrasonically in an acetone bath for 40 s.

The thermal diffusivity (α) was measured by a laser flash method (LFA427, NETZSCH, Germany) with an international standard using a disc 10 mm in diameter and 3 mm in thickness. Three times were repeated for one sample to get an average value of the thermal diffusivity. The thermal conductivity (K) can be calculated

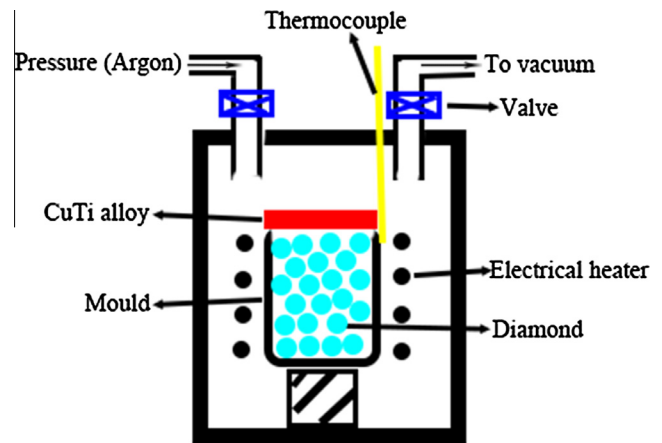


Fig. 1. Schematic drawing of the gas pressure infiltration device. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

according to the equation $K = \alpha \rho c$, where ρ is sample density and c is specific heat. The density was measured by the Archimedes method using alcohol as the immersion medium, and the measurement was repeated three times for each sample. The heat capacities were calculated from the rule of mixture based on mass fraction of each component. The CTEs of the composites were measured by a dilatometer (DIL 402C, NETZSCH, Germany) with a heating rate of 5 K/min in the temperature range between 323 K and 573 K, using a cylinder 5 mm in diameter and 25 mm in length.

3. Results and discussion

Fig. 2 shows the representative SEM images of the polished surface and fracture surface of the Cu-0.5 wt.%Ti/diamond composite. The diamond particles are found to be uniformly distributed in the Cu matrix (Fig. 2a) and no noticeable defects such as cracks or flaws are observed at the interface. The pull-out of diamond particles is rarely seen in the polished surface, which indicates strong interface bonding between the diamond particles and Cu matrix. The diamond particles maintain their original shape without any degradation. The observations demonstrate excellent quality of the composites produced by the gas pressure infiltration.

As seen from the fractured surface, the Cu matrix closely adheres to the surfaces of the diamond particles (red circles in Fig. 2b). Some diamond particles are found to fracture transgranularly. This fracture only occurs when the interfacial bonding strength is higher than the fracture strength of the diamond particles. The synthetic diamond has some flaws inside. It can be concluded that Ti addition has significantly improved the interfacial bonding between the Cu matrix and diamond particles.

In order to study the effect of Ti addition on the phase composition, the XRD patterns of the Cu-Ti/diamond composites were characterized, as shown in Fig. 3a. The results indicate the coexistence of Cu, diamond and TiC phases in the composites. Fig. 3b shows the XRD patterns of the diamond particles extracted from the Cu-Ti/diamond composites. Only diamond and TiC phases are detected. The morphology of the diamond particles is clearly seen in the inset Fig. 3b. The diamond particle surface is covered uniformly by the carbides. Owing to the interfacial reaction between the Cu-Ti matrix and the diamond, TiC is formed during the infiltrating process by the reaction between Ti and C atoms ($\text{Ti} + \text{C} \rightarrow \text{TiC}$). Thermodynamic calculation shows that titanium carbide will be produced by the reaction of carbon with copper-titanium alloys that have a titanium activity larger than 0.1 at 1400 K and close to 1.0 at 1423 K [26], which is the infiltration temperature

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