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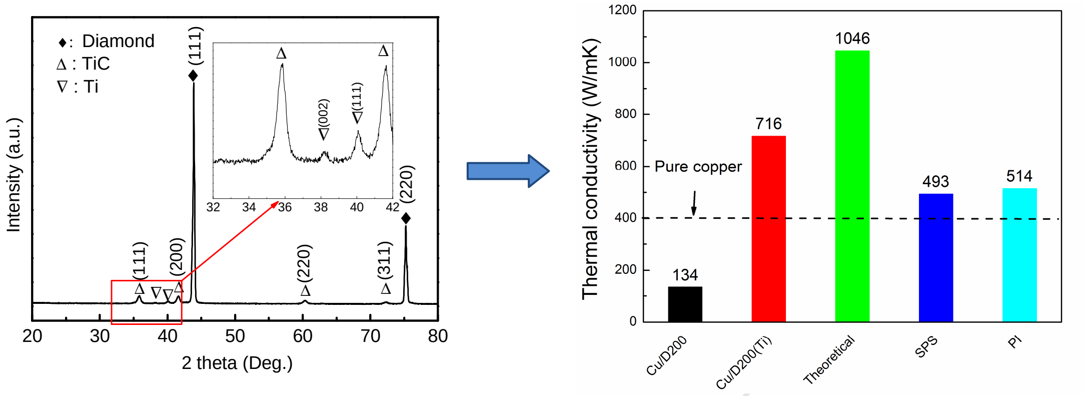
Graphical Abstract



Microstructure and thermal conductivity of Cu/diamond composites with Ti-coated diamond particles produced by gas pressure infiltration

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Abstract: As an attractive thermal management material, diamond particles reinforced Cu matrix (Cu/diamond) composites generally exhibit thermal conductivities lower than expected. To exploit the potential of heat conduction, a combination of Ti coating on diamond particles and gas pressure infiltration was used to prepare Cu/diamond(Ti) composites. A high thermal conductivity of 716 W/mK and a low coefficient of thermal expansion of 5.8 ppm/K at 323 K were obtained in the composites. Auger electron spectroscopy (AES) characterization shows that a TiC layer was formed between Cu matrix and diamond reinforcement, which is responsible for the enhancement of thermal conductivity. The results suggest that Ti coating can significantly promote interface bonding between Cu and diamond and gas pressure infiltration is an effective method to produce Cu/diamond composites.

Keywords: A. Metal matrix composites; B. Liquid-solid reactions; C. Heat conduction; Auger electron spectroscopy

1. Introduction

Microelectronic devices with high power density are demanding electronic packaging materials with high thermal conductivity (TC). Due to extraordinarily high TC of diamond (1200–2200 W/mK), diamond particles reinforced metal matrix composites (MMCs) become a popular candidate and are inspiring great interests. Compared with Al/diamond [1,2], Cu/diamond composites have higher TC and lower coefficient of thermal expansion (CTE) in theory and are especially suitable for applications at higher operating temperatures due to higher melting point of Cu [3]. Microelectronic devices can generate significant heat fluxes such as ~50 W/cm^2 in current electronic chips and up to 2000 W/cm^2 in semiconductor lasers [4]. Owing to high thermal conductivity, Cu/diamond composites as thermal conductive materials can dissipate heat from the microelectronic devices quickly and maintain reasonable temperature in the devices.

Despite the potential for excellent performance in Cu/diamond composites, poor wettability of copper with diamond acts as a barrier to processing the composites. Even at a high temperature of 1673 K, copper and diamond exhibits a contact angle as large as 128.7° [5]. In order to take full advantage of high thermal conductivity of diamond, surface metallization of diamond particles [6-11] and alloying of Cu matrix [12-15] are commonly used to improve interfacial bonding between Cu matrix and diamond reinforcements. Zhang et al. obtained an enhanced TC value of 493 W/mK by coating Ti on diamond particles [16]. He et al. achieved a high TC value of 677 W/mK in Cu/diamond composites by alloying Zr into Cu matrix [17].

In addition to interface modification, microstructure optimization of Cu/diamond composites is also crucial to the enhancement of thermal conductivity. So far several processing techniques like spark plasma sintering (SPS) [18-21], pulse plasma sintering [22], vacuum hot pressing [23], pressureless infiltration [9], high pressure high temperature (HPHT) infiltration [16] and gas pressure infiltration (GPI) [15] have been employed to optimize the microstructure of Cu/diamond composites. Among these processes, GPI is proved to be effective in achieving high TC in Cu/diamond composites [15]. Since Beffort et al. [24] firstly produced Al/diamond composites using GPI in 2006, this route has been followed by several groups [1,25]. However, because of high melting point of Cu and extreme chemical inertness between diamond and Cu, the production of Cu/diamond composites via GPI is rarely reported [15]. Besides, the effectiveness of Ti coating on diamond particles in enhancing thermal conductivity of GPI-produced Cu/diamond composites has not yet been proved. As an alternative route, the combination of surface metallization of diamond particles by Ti coating and promising technique of GPI could be helpful to modify Cu/diamond interface and to achieve high thermal conductivity in the composites.

In this article, we use gas pressure infiltration to produce Cu/diamond composites with Ti-coated diamond particles. The interface structure is elaborately investigated by using diamond particles dissolved from the produced Cu/diamond composites to determine the effect of Ti coating on thermal conductivity. As a result, a TC of 716 W/mK is achieved, which is 79% higher than 400 W/mK for Cu matrix.

2. Experimental

Commercially available bulk Cu (purity: 99.95 wt.%, Cuibolin Non-Ferrous Technology Co., China) was used as metal matrix. Synthetic single-crystalline diamond powders (particle size: 62-75 μm, mesh: 200-230, HWD40, Henan Huanghe Whirlwind International Co., China) were used as particulate reinforcements. The diamond has been characterized to contain 150-170 ppm N_2, giving a TC of 1700-1850 W/mK [26]. The diamond particles were coated with Ti by using vacuum vapor deposition. In doing so, diamond particles and Ti powders (particle size: ~5 μm, TaiYu Materials Science & Technology Co., China) were mixed and then heated at 1098 K for 15 min in a high vacuum induction- heating furnace.

The Cu/diamond(Ti) composites were produced by a home-made gas pressure infiltration equipment. The Ti-coated diamond particles were tap-packed in a graphite mold and the Cu bulks were placed on top of the diamond powder bed. The installed graphite mold was moved to a graphite heating muff for infiltration. A vacuum better than 0.1 Pa was evacuated before heating, and the graphite mold was then inductively heated to 1423 K and kept for 10 min. Afterwards, high-purity argon gas was pumped into the muff to maintain a pressure of 1.0 MPa. The pressure was kept for 15 min to enable sufficient contact of diamond crystals with copper melts, before switching off the heating source. The infiltrated Cu/diamond(Ti) sample was furnace cooled down to room temperature within 1-2 h.

The volume fraction of diamonds in the composite can be estimated as follows. Without consideration of pores in samples, the composite density is written as $\rho_c = \rho_d V_d$

+ $\rho_m(1-V_d)$, where ρ_c, ρ_d and ρ_m are densities of composite, diamond and copper, respectively, and V_d is the volume fraction of diamonds in the composite. For the estimation, ρ_c was examined by the Archimedes, and ρ_m = 8.90 g/cm^3 and ρ_d = 3.52 g/cm^3 were used. The phase structure of the coated diamond particles was detected by X-ray diffraction using Cu Kα radiation (XRD, Rigaku DMAX-RB, Japan). Auger electron spectroscopy (AES, PHI 700, Japan) was used to investigate interface diffusion and reaction between Ti coating layer and diamond. The produced Cu/diamond composite samples were electrochemically etched in a 10 vol.% HNO_3 and 90 vol.% H_2O solution for 15 min. The extracted diamond particles were then cleaned in acetone for 1 min and the cleaning was repeated for 3 times. The microstructure of the Cu/diamond composites was characterized by field emission scanning electron microscopy (FE-SEM, SUPRA 55, ZEISS, Germany). The samples as fabricated by GPI were grinded by using diamond wheels and then SiC papers, and finally were polished by using diamond pastes. The thermal conductivities of the composites were evaluated by using $K = \alpha\rho_c C_p$, where K is thermal conductivity, α is thermal diffusivity, ρ_c is bulk density of sample, and C_p is specific heat capacity. The thermal diffusivity was measured at room temperature by a laser flash apparatus (LFA427/3/G, Netzsch, Germany). For the measurement of thermal diffusivity, the as-cast composite samples were cut into disk-shaped specimens 10 mm in diameter and 3 mm in thickness. The specific heat capacity was measured by a differential thermal analysis (DSC 204, Netzsch, Germany). The CTE measurements were conducted on a NETZSCH DIL 402C thermo-mechanical analyzer at a heating rate of 5 K/min. The samples for CTE measurement were made with a cylinder shape of Φ3 mm×25 mm.

3. Results and discussion

The volume fraction of diamonds was estimated to be 65 vol.% by the density measurements. Fig. 1 shows the XRD patterns of the Ti-coated diamond particles. Besides diamond and TiC, a small amount of Ti phase was also detected with the diamond particles. This means that a layer configuration of diamond/TiC/Ti could be formed in the vacuum vapor deposition.

The polished surfaces of the Cu/diamond composites with uncoated and Ti-coated diamond particles are shown in Fig. 2. The diamond particles were homogeneously dispersed in the Cu matrix. Due to poor wettability between pure copper and diamond, some uncoated diamond particles were found to dropout from the composite during the polishing process (Fig. 2a). By contrast, the presence of TiC layer strengthens the Cu/diamond(Ti) interface, and no diamond particles were found to fall off, as seen in Fig. 2b. It is thus concluded that the formed TiC layer can improve adhesion between diamond particles and Cu matrix.

Fig. 3 shows the SEM observation of the fracture surfaces of the composites. The intact diamond particles shown in Fig. 3a indicate poor interfacial bonding in the unmodified Cu/diamond composite because the fracture often occurs at interfaces with weak bonding. For comparison, good interfacial bonding is revealed in the Cu/diamond composites with Ti-coated diamond particles, where plastic fractures occur within Cu matrix and Cu-adhered diamond particles are observed, as indicated by the circles in Fig.

3b. This indicates that the interfacial bonding strength of Cu/diamond(Ti) is even higher than the intrinsic fracture strength of Cu matrix.

The energy dispersive X-ray spectroscopy (EDS) scan across a Cu/diamond(Ti) interface is shown in Fig. 4. The EDS analysis was carried out on a grinded and polished surface. The result indicates that the intermediate zone, between Cu matrix and diamond reinforcements, was enriched in Ti. In addition, Ti signal detected in the matrix was higher than that on the diamond. This suggests that the coated Ti could diffuse to Cu matrix at the infiltration temperature of 1423 K. According to the Cu-Ti phase diagram, the maximum solubility of Ti in Cu is nearly 8 at.%. After cooling down, the dissolved Ti could stay in the crystals of Cu matrix.

Fig. 5 shows the AES results for the Ti-coated diamond particles before infiltration and dissolved from the produced Cu/diamond(Ti) composites. The formation of TiC can be confirmed by the Auger line shape analysis. As shown in Fig. 5c and 5d, C spectra consisted of three peaks at 256, 264 and 274 eV; as shown in Fig. 5e and 5f, Ti spectra exhibited one peak at 419 eV. The analysis suggests the existence of TiC [16]. The initial thickness of TiC layer is 532 nm (Fig. 5a), deduced from the sputtering time with a sputtering rate of 76 nm/min that is calibrated by SiO_2 film. For comparison, the thickness of TiC layer on the diamond particles dissolved from the Cu/diamond(Ti) composites increases to 1292 nm (Fig. 5b). The finding means that C and Ti inter-diffuse during infiltration to increase the thickness of TiC. Fig. 5b shows that oxygen was presented through the layer. This indicates that environmental O could be incorporated into the layer during infiltration owing to the limited vacuum used.

It is interesting to note that the chemical states of Ti and C on the surface of diamond particles have changed after infiltration. On the surface of the diamond particles before infiltration, the chemical states of Ti and C corresponds to TiC (Fig. 5c and 5e). In contrast, on the surface of the diamond particles dissolved from the composites, only one C peak was observed in the spectra (Fig. 5d) and the Ti peak almost disappeared (Fig. 5f). The difference could be explained as follows. In the electrochemical etching process to extract diamond particles, the Cu/diamond composites were designated as the anode. Besides the Cu matrix, the Ti in TiC is also electrochemically dissolved, leaving activated carbon on the surface of diamond particles [27]. As seen in Fig. 5b, the elemental profile of Ti exhibits an upward trend at the beginning of Ar^+ sputtering, which provides an evidence to support the explanation.

Fig. 6 shows a survey of thermal conductivity of Cu/diamond composites. The GPI-produced Cu/diamond composite reinforced with uncoated diamond particles showed a TC of only 134 W/mK, lower than half value of pure copper. Ti coating on diamond particles can greatly enhance the thermal conductivity of Cu/diamond composites. The TC of the Cu/diamond composites prepared by SPS and pressure infiltration (PI) are 493 W/mK [16] and 514 W/mK [28], respectively. For comparison, the TC of the Cu/diamond(Ti) composites prepared by GPI reached 716 W/mK. This suggests that TiC layer can improve the interface bonding effectively and then enhance the thermal conductivity.

We have calculated the theoretical value of thermal conductivity for Cu/diamond composites using the Maxwell-Eucken model [29] without considering interface thermal resistance:

$$K_c = K_m \left[\frac{2+K_d/K_m+2V_d(K_d/K_m-1)}{2+K_d/K_m+V_d(1-K_d/K_m)} \right] \quad (1)$$

where K_c, K_m and K_d are thermal conductivities of composite, matrix and particulate reinforcement, respectively, and V_d is volume fraction of reinforcements. The calculations take thermal conductivities of 1800 and 400 W/mK for diamond reinforcement and Cu matrix, respectively, and V_d of 65 vol.%. The calculated value is 1046 W/mK for the GPI-produced Cu/diamond composites, as shown in Fig. 6. It can be seen that the experimentally measured 716 W/mK is about 68% of the theoretical value. Since the thermal conductivity of TiC is 36.4 W/mK [30], much lower than those of both copper and diamond, the thermal conductivity of the composites is lower than the theoretical value.

While metal/diamond composites are highly densified, acoustic impedance between dissimilar materials will be the main source of interface thermal resistance. In this study, Ti was chosen as a coating element for the metallization of diamond particles because TiC has an acoustic impendance between diamond and Cu. In order to understand directly the effect of Ti coating, we calculate the interface thermal conductance of the Cu/diamond composites. Without considering the microstructure of interface, the interface thermal conductance can be deduced from the acoustic mismatch model (AMM) [31]. In a previous study [16], we obtain the interface thermal conductance of $h_{\text{copper/diamond}}=4.80\times10^{7}$ W/m^{2}K. Hasselman and Johnson [29] have proposed a theoretical model to predict the thermal conductivity of metal/diamond composites with consideration of interface thermal conductance:

$$K_c = K_m\left[\frac{2\left(\frac{K_d}{K_m}-\frac{K_d}{ah_c}-1\right)V_d+\frac{K_d}{K_m}+\frac{2K_d}{ah_c}+2}{\left(1-\frac{K_d}{K_m}+\frac{K_d}{ah_c}\right)V_d+\frac{K_d}{K_m}+\frac{2K_d}{ah_c}+2}\right] \tag{2}$$

where a is the radius of spherical dispersion and h_c is the interface thermal conductance. When the values of other parameters are known, h_c can be derived from Eq. (2). Then, the derived h_c value is regarded as a measure of interface thermal conductance. The values of K_c, K_m, K_d and V_d are previously given, and the diamond particles have an average size of a = 70 μm. The h_c values are thus calculated to be 3.36×10^5 W/m^{2}K and 2.99×10^7 W/m^{2}K for the Cu/diamond composites with uncoated and coated diamond particles, respectively. It can be seen that Ti coating has greatly increased the interface thermal conductance between Cu matrix and diamond reinforcements, and in turn enhanced the thermal conductivity of the composites. Even so, the 2.99×10^7 W/m^{2}K is still lower than the value of 4.80×10^7 W/m^{2}K for an ideal Cu/diamond interface.

Actually, the thickness of TiC layer should also be taken into account in the calculation of interface thermal conductance because TiC has a low thermal conductivity. To do so, a model of series electrical resistance analogy [32] is used to calculate the interface thermal conductance of Cu/diamond composites:

$$\frac{1}{h_{total}}=\frac{1}{h_{copper/carbide}}+\frac{1}{h_{caibide/diamond}}+\frac{l}{K_{carbide}} \tag{3}$$

where h is interface thermal conductance, K is thermal conductivity, and l is thickness of carbide layer. The thickness of TiC layer is characterized to be 1292 nm based on the AES analysis and the thermal conductivity of TiC is 36.4 W/mK [30]. The $h_{\text{copper/carbide}}$ and $h_{\text{carbide/diamond}}$ are deduced from the AMM model to be 2.1×10^8 W/m^{2}K and 5.9×10^8

W/m^{2}K, respectively. When the carbide layer is taken into consideration, the interface thermal conductance for the Cu/diamond composites is thus evaluated to be 2.38×10^7 W/m^{2}K, a bit lower than without consideration of TiC layer. This finding shows the negative impact of the coating. As such, the coating layer should be as thin as possible in order to improve the interface thermal conductance of Cu/diamond composites.

In a thermal management system, the packaging materials should have good thermal conductivities. The other concern is the match of CTE between microelectronic devices and electronic packaging materials. Fig. 7 shows the CTE values of the Cu/diamond(Ti) composites. The CTE values were obtained from the instantaneous derivative of the sample relative dilatation vs. temperature. The CTE of the composites increases gradually with temperature. When the temperature is lower than 600 K, the CTE is below 9 ppm/K. At 323 K, the CTE is as low as 5.8 ppm/K, meeting the requirements of electronic packaging materials. For the cooling of personal computer (PC), the temperature of the central processing unit (CPU) should be restricted to 353 K-363 K [33]. As such, we have also obtained the average CTE value in the temperature range from 323 K to 373 K. The CTE value was obtained to be 6.0 ppm/K, by averaging all data points measured in the temperature range. This CTE value matches that of semiconductors.

4. Conclusions

The Cu/diamond composites with Ti-coated diamond particles were fabricated by gas pressure infiltration. The results suggest that Ti coating on diamond particles enhances the interfacial bonding between diamond and copper in the produced Cu/diamond(Ti) composites. As a result, the thermal conductivity of the composites is dramatically increased from 134 to 716 W/mK. The average CTE values is 6.0 ppm/K in the temperature range from 323 K to 373 K. However, the measured thermal conductivity is still lower than the theoretical value. In future research, the thickness of TiC layer could be optimized by adjusting vacuum vapor deposition or gas pressure infiltration parameter to further enhance the thermal conductivity.

Acknowledgements

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Figure captions

Fig. 1. XRD patterns of the Ti-coated diamond particles.

Fig. 2. Microstructure of the Cu/diamond composites fabricated by gas pressure infiltration with (a) uncoated and (b) Ti-coated diamond particles.

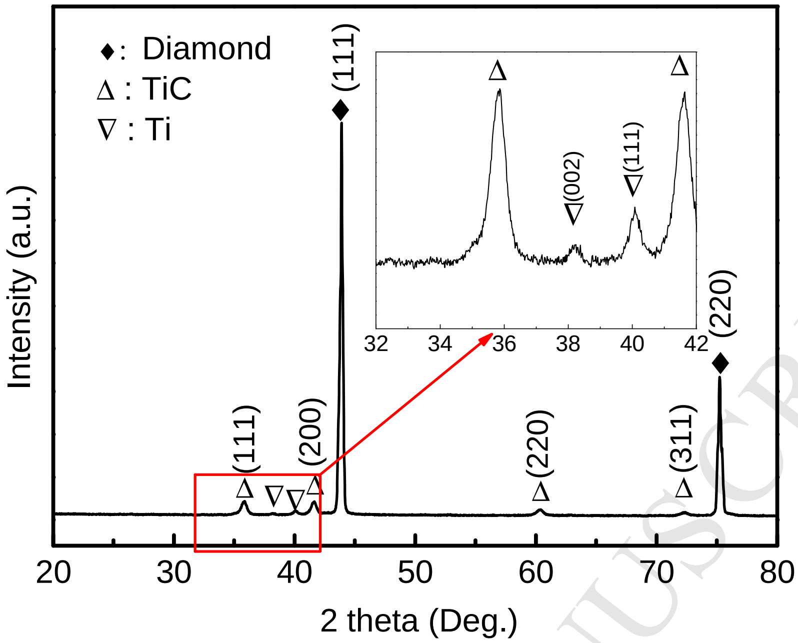
Fig. 3. Fracture surfaces of the Cu/diamond composites fabricated by gas pressure infiltration with (a) uncoated and (b) Ti-coated diamond particles.

Fig. 4. (a) Polished surface of the Cu/diamond(Ti) composites and elemental distribution of EDS scan across Cu/diamond interface for (b) Ti, (c) Cu, and (d) C.

Fig. 5. Auger electron spectroscopy detected elemental depth profile for Ti-coated diamond particles (a) before composite fabrication and (b) dissolved from the Cu/diamond(Ti) composites as well as the corresponding kinetic spectra of C and Ti at various depths: (c) C and (e) Ti for (a) and (d) C and (f) Ti for (b).

Fig. 6. Thermal conductivity of the Cu/diamond composites fabricated by gas pressure infiltration, compared with literature and theoretical value.

Fig. 7. Coefficient of thermal expansion of the Cu/diamond(Ti) composites fabricated by gas pressure infiltration.



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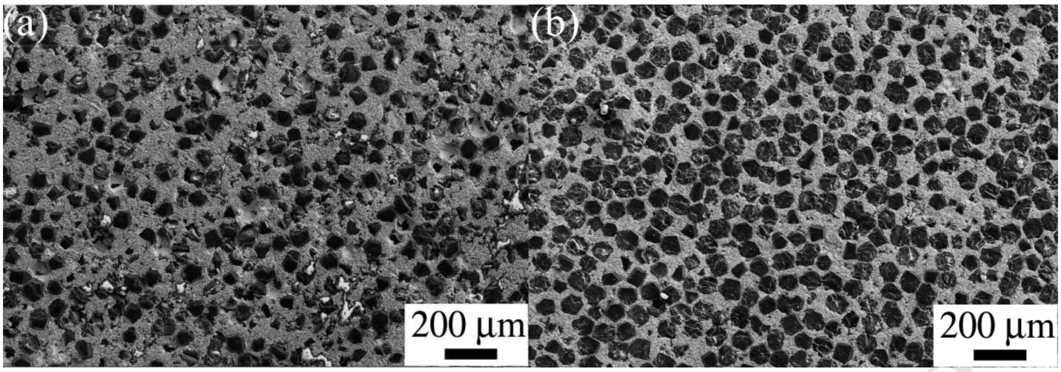

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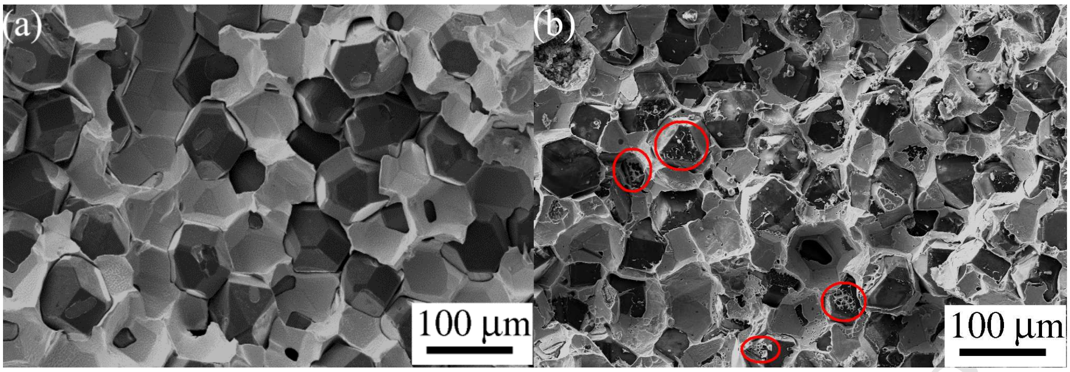

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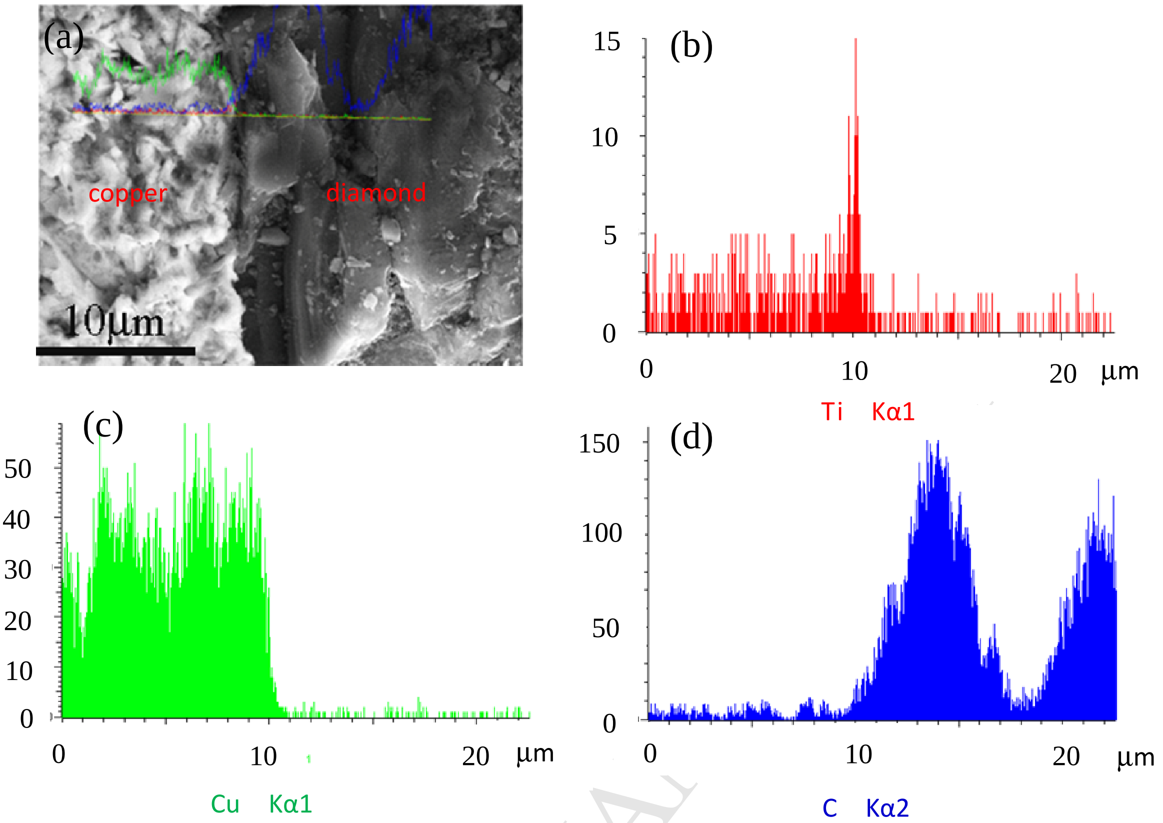

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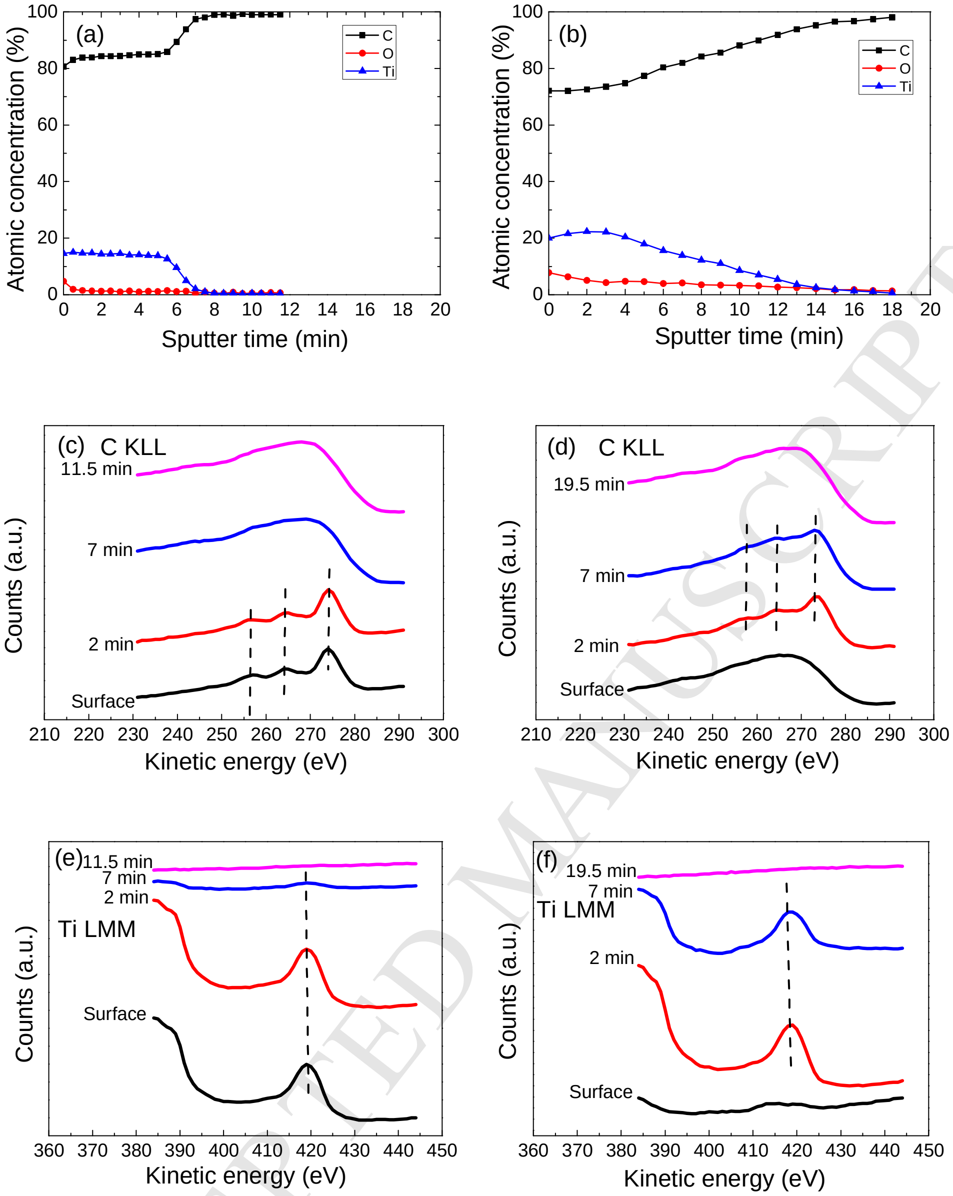

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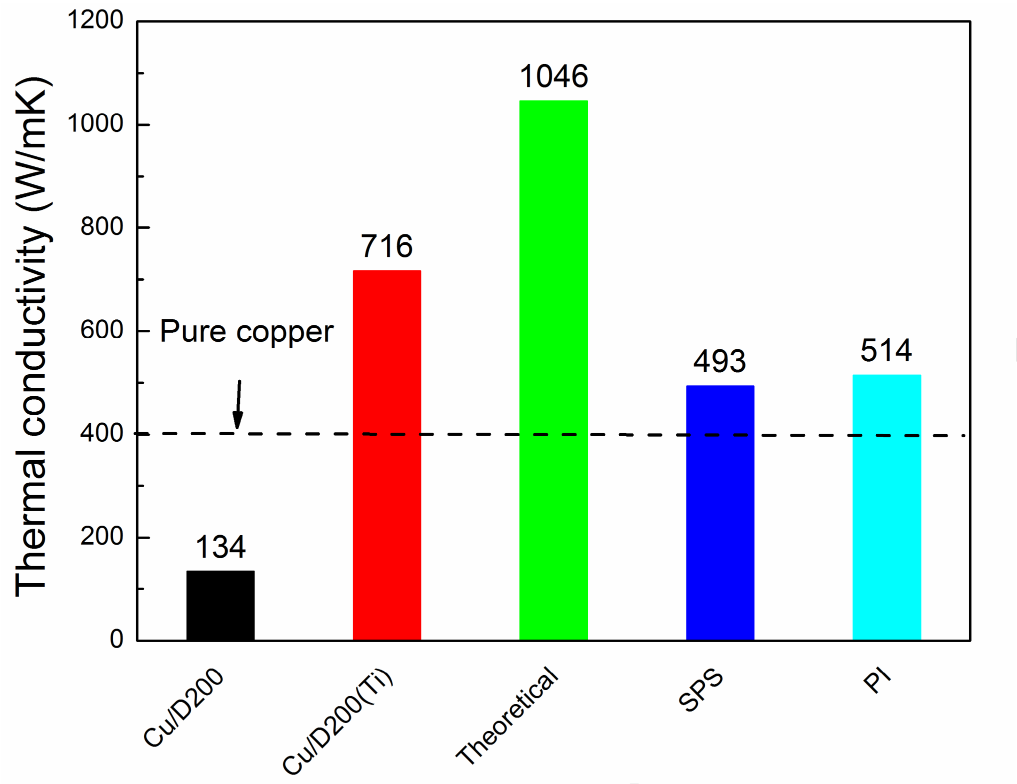

Fig. 6. Thermal conductivity of the Cu/diamond composites fabricated by gas pressure infiltration, compared with literature and theoretical value.

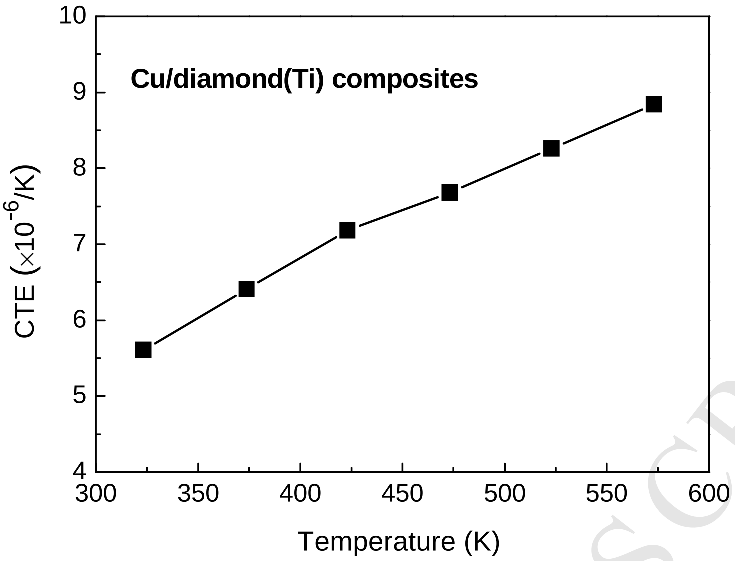

Fig. 7. Coefficient of thermal expansion of the Cu/diamond(Ti) composites fabricated by gas pressure infiltration.

Highlights

- The Cu/diamond(Ti) composites are produced by gas pressure infiltration.
- A TiC layer is formed between Cu matrix and diamond reinforcement.
- A thermal conductivity of 716 W/mK is obtained for the composites.
- A coefficient of thermal expansion of 5.8 ppm/K at 323 K was obtained.