

Effect of film thickness on the temperature dependence of thermal conductivity for diamond/BeO composites

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Abstract

Polycrystalline diamond films of different thickness have been deposited on the surface of beryllium oxide (BeO) substrates by hot filament chemical vapor deposition (CVD). Thermal conductivity (κ value) of various samples has been measured. The biggest increase of 17.5% in thermal conductivity relative to the substrate is obtained in the composite with 5.0 μm film at room temperature. The temperature dependence of the thermal conductivity for the composites with films of different thickness has been investigated. Comprehensive effect of film purity and residual stress is found to be responsible for the variation of temperature dependence for the composites. Stress-induced defects play an important role in the thermal conductivity at high temperature from our analysis. This work is of great significance on the preparation and thermal application of composites with CVD diamond in such a film/substrate structure.

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1. Introduction

The increasing integration and power density of electronic devices make the heat dissipation a challenging problem, especially in those applications where materials with both high electrical resistivity and high thermal conductivity are demanded. Take traveling-wave tube (TWT) as an example, both the above properties are required for its supporting rods. Moreover, since the local operating temperature of TWT could reach over 600 K, materials with high conductivity in a broad temperature range are favored for the performance and longevity of the tube. This applies in many aerospace materials in demanding environment as well.

Much effort has been paid for the pursuit of high thermal conductivity materials, among which several structures

have been developed. Diamond/copper composites fabricated by mixing diamond/copper powders and subsequent sintering method demonstrate an isotropic conductivity of 742 W/(m K) [1]. Abyzov et.al. [2] developed a filler/coating/matrix structure for various composites and a thermal conductivity of 900 W/(m K) was obtained. However, due to the metal components, none of the above materials could be adopted in the applications where high electrical resistivity is demanded.

The pioneer work of combining BeO with diamond films can be traced back to 1993 by Alberto [3]. Beryllium oxide (BeO) possesses a thermal conductivity of about 370 W/(m K) at room temperature (RT) [4], higher than any other known practical ceramics and even most metals. It has already been broadly applied in high-power and high-density electronics in the last decades. Another material that shows both high thermal conductivity (~ 2000 W/(m K), RT) and electrical resistivity is diamond. Owing to the scarcity of natural diamond, polycrystalline diamond films fabricated by CVD methods are preferred for their low cost and outstanding conductivity.

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In this paper, polycrystalline diamond films of different thickness are fabricated by hot filament CVD on BeO plate substrates, forming a series (film/substrate) structure. We mainly investigate the effect of temperature and film thickness on the thermal conductivity of diamond/BeO composites. The relationship between diamond film structure and thermal conductivity of the composites are revealed.

2. Material and methods

The polycrystalline beryllium oxide substrates, of which the density was 2.89 g/cm^3 and the size was $\phi 12.5 \text{ mm} \times 2 \text{ mm}$, were manufactured by a hot pressing technique. Identical pretreatment were performed before deposition of all the samples. The substrates were first etched by hydrofluoric acid (analytical pure) for 10 min to produce a certain amount of micro-cavities on the surface, making the diamond films better bonded to the substrates by mechanical interlocking. Then the substrates were ultrasonically agitated in micro-diamond colloid for another 10 min. Immediately afterwards, the samples were ultrasonically rinsed for 2 min in deionized water and absolute ethanol respectively and blown dry before subsequent diamond deposition.

Diamond films of different thickness were deposited on only one side of the beryllium oxide substrates in a hot filament chemical vapor deposition system, using a precarburized W filament coil. To obtain diamond films with higher thermal conductivity (higher purity), the growth rates need to be controlled on a relatively slow level [5]. A gas mixture with a total flow of 50 sccm consisting of methane and hydrogen (2:98 vol%) was introduced with a total pressure of 2 kPa. The filament-to-substrate distance was 7–8 mm and the substrate temperature was maintained at $750 \pm 10^\circ \text{C}$. All the above conditions were controlled as identical as possible during every deposition. And the deposition times of 2, 4 and 8 h were employed to obtain composites with diamond films of different thickness. For convenience, the samples were denoted as S1, S2 and S3, respectively.

Scanning electron microscopy (SEM; FEI Sirion200) was employed to characterize the surface and cross-section morphology of the diamond films. The micro-Raman analysis (LabRAM HR800) was performed at room temperature. The 10 mW, 488 nm line of argon ion continuous laser was utilized and the spot size was $\sim 1 \mu\text{m}$ in diameter.

The perpendicular thermal conductivity (in the axis direction) of various composites (substrate included) in the temperature range of 298.15–653.15 K were measured by laser flash technique utilizing Netzsch LFA 457 [6], which have been broadly employed to characterize high conductivity materials with either simple or complex structures [7,8], especially in a wide temperature range. The principle of this measurement is utilizing a pulsed laser to heat the front side of plate-shape material and recording the temperature rise versus time on its rear surface, thus we can directly obtain thermal diffusivity D by using equation given in reference [8], and specific heat C by using a comparative method [9]. By multiplying D , C and the composite density ρ , we got the

thermal conductivity value ($\kappa = \rho \cdot D \cdot C$). Here we simply took the composite density as 2.89 g/cm^3 (equal to that of bare substrate) since both components had similar density and the diamond content was relatively low. The applied laser size was 12.7 mm in diameter, same as the substrate dimension. Measurement results were calibrated by the Cowan modal [10].

3. Results

The surface and cross-section morphologies (the upper right insets) of diamond films deposited for 2, 4 and 8 h are shown in Fig. 1. It is obvious that both the crystal size and film continuity increase along with the deposition time. Apparent discontinuity between the well faceted diamonds can be observed in S1, but the surface coverage of 100% have been reached for the films deposited for 4 h and 8 h. The average film thickness determined by cross-section images of the above 3 samples are about 0.9, 1.4 and $5.0 \mu\text{m}$.

Despite the efficiency of micro-Raman tests in detecting structural defects and stress distribution [11,12], care must be taken to obtain the Raman spectra. A large number of spectra are recorded [13] over the surface of diamond films to get more reliable information. The Raman spectra of all the diamond films are given in Fig. 1(d). Both upward shifting and splitting of normal diamond peak (1332 cm^{-1}) into singlet and doublet peaks are observed for all films. The details of shifting and splitting are better shown in the insets. The broad bands at 1350 cm^{-1} , 1480 cm^{-1} and 1580 cm^{-1} are also present, the band of 1480 cm^{-1} is assigned to transpolyacetylene at grain boundaries and the rest two bands are referred to sp^2 -bonded amorphous carbon (D and G bands) [12].

The thermal conductivity of various composites as a function of absolute temperature is shown in Fig. 2, and the results from Ref. 15 are also included for comparison. Error bars are not shown here because the standard deviations are extremely low ($\sim 0.2 \text{ W/(m K)}$) and do not influence the results. For every material illustrated, the thermal conductivity exhibits obvious decrease when the temperature increases. By comparing the composites with diamond films of different thickness to substrates, it is plain that although S2 and S3 show apparent increase in thermal conductivity, S1 shows obvious reduction at every temperature. By calculating the rates of increase in thermal conductivity of composites relative to the BeO substrates at every temperature, very interesting phenomenon can be discovered. The results are given in Fig. 3. The maximal increase is obtained at 293.15 K in S3, reaching approximately 17.5%. At 293.15 K and 333.15 K, the rates of increase for S3 are higher than S2. From 398.15 K to 653.15 K, the rates of increase for S3 become slightly lower than S2, both around 7%, while the reduction of S1 is between 6% and 8%.

4. Discussion

Thermal conductivity in the device operating temperature range is the key index for further thermal management

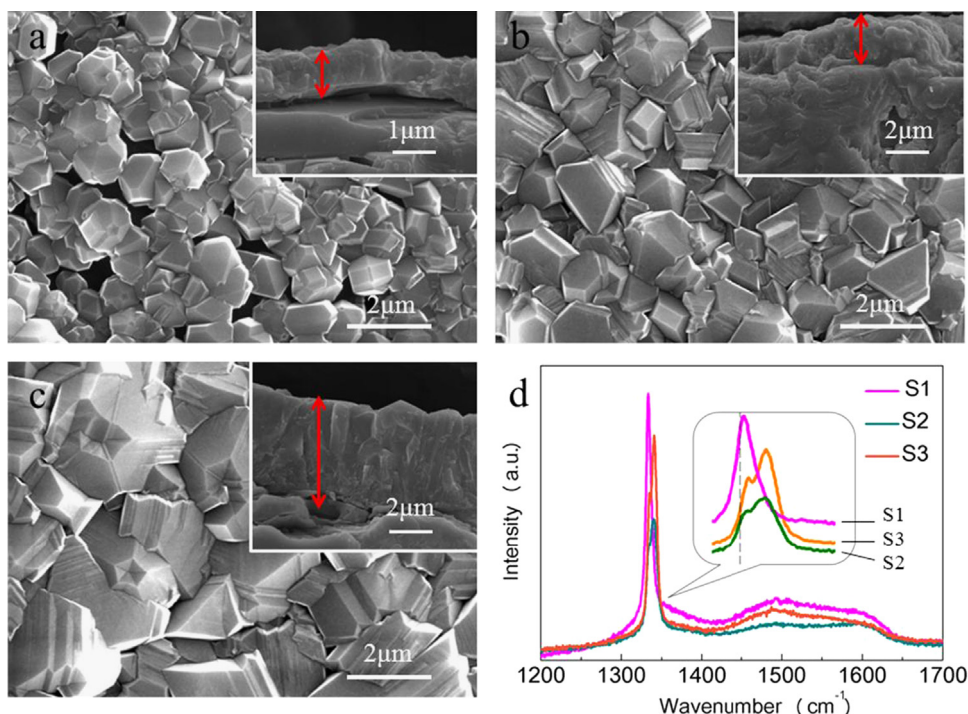


Fig. 1. The surface and cross-section (upper right insets) morphology of diamond films deposited for (a) 2 h, (b) 4 h and (c) 8 h. (d) The corresponding Raman spectra of as-deposited films. The inset shows the details of shifting and splitting of diamond peak.

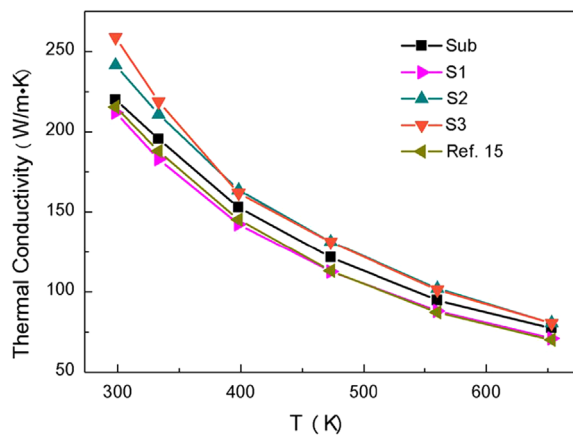


Fig. 2. Thermal conductivity of various samples in the temperature range from 298.15 K to 653.15 K. The dark yellow triangles represent the results in Ref. [15]. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

applications. For deeper investigation on the temperature dependence of the conductivity for the composites, further data processing is needed on the basis of above results. From the published papers on the temperature dependence of dielectric materials, especially for ceramics [14,15] and natural diamond [16], we know that the relationship between thermal conductivity and absolute temperature could be approximated by the following equation:

$$\kappa = a/T^b \quad (1)$$

where κ is the thermal conductivity in W/(m K) and T is in kelvins, a and b are both constants for given materials. It

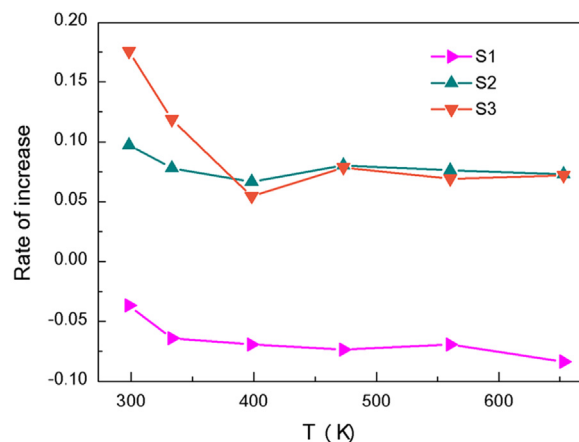


Fig. 3. The rates of increase in thermal conductivity of diamond/BeO composites relative to BeO substrates in the measured temperature range.

should be specifically pointed out that Eq. (1) applies well in the temperature range of 298.15–653.15 K and the b values are ~ 1 , for both diamond film [16] and beryllium oxide [15].

The fitting processes using Eq. (1) have been performed for BeO in Ref. [15], our substrates and as-prepared composites. The fitting results are listed in Table 1. The curves of our composites fit extremely well with the measured results. It indicates that the two-phase composites in such a series structure also demonstrate the same characteristics as single-phase ceramics. The temperature dependence of materials can be well reflected by the b value, i.e., the larger b value is, the stronger the dependence would be. It is quite clear that the BeO we employed as substrates possess weaker temperature

Table 1

The a and b values for the BeO in Ref [15], natural diamond, our substrates and as-prepared composites.

Sample	BeO (Ref. [15])	Diamond (Ref. [16])	BeO (substrates)	S1	S2	S3
a ($\times 10^5$)	7.392	28.33	5.820	6.533	6.995	13.52
b	1.427	1.245	1.377	1.408	1.396	1.503

dependence than those measured by Ditmars et.al. [17], this is because the density of our substrates (2.89 g/cm^3) is larger than theirs (2.62 g/cm^3). Another conclusion is that S3 has stronger temperature dependence than S2 and S1.

In fact, the equation recommended by Harris [16] applies better to natural diamond than CVD diamond. Massive work [18–20] has been done on the temperature dependence of CVD diamond films of various grades. But most of them did not give the exact relationship and only focused on the temperature range from several kelvins to $\sim 400 \text{ K}$. For hot-filament CVD diamond, the effect of impurity and defects on the temperature dependence becomes particularly noticeable [21]. Therefore, it is quite possible that the b value for CVD diamond film would be larger than 1.245. According to results listed in Table 1, the diamond/BeO composites all have greater b value than BeO substrate, indicating stronger dependence in the measured temperature range. This can be attributed to the thermal boundary conductivity [22] of the diamond–BeO interface or diamond films with various defects, or the summation of both. The influence of BeO is excluded for its stability in the experimental temperature range; so as for the interfacial thermal resistance, because the interfaces between substrates and films are expected to be the same under identical deposition condition. In this paper we try to explain the results of thermal conductivity mainly by the following investigation on the diamond film quality and thickness.

Typically, thicker diamond films demonstrate higher thermal conductivity than thin films of the same grade [23], thus it is reasonable for us to expect higher conductivity in the composites with thicker films, which is exactly the situation observed at 295.15 K and 333.15 K. However, the inverse situation appears in the higher temperature range. In view of the virtually identical deposition parameters in our experiments, we think this is closely related with the structure evolution of diamond films in the growth direction.

The columnar structure [24] of CVD diamond films and cross section morphology of S3 are given in Fig. 4. With growth time increasing, the unfilled spaces between crystals in the early stages remain at the interface. Increasing crystal sizes and diminishing of grain boundaries are observed, also depicted in the picture for better view. It has been proven that impurities like amorphous and graphitic phases mainly accumulate at grain boundaries [25–27]. Also, the regular defects including dislocations and stacking faults had been proved to be located preferentially near grain boundaries [23,28,29]. Thereby, concentrations of all the above defects would decrease with increasing thickness in the growth direction.

This trend would be further justified in the following analysis on Raman data. It implies that none of the above defects should be accounted for the inverse situation mentioned above, since the scattering of phonons from them would also decrease in the upper part of the thick film. Moreover, the thermal resistance resulted by the phonon scattering from point defects, grain boundaries and the above defects had been calculated and analyzed in a broad temperature range [21], the conclusion is that scattering from grain boundaries dominates at low temperature while point defects become the main factor at high temperature. The above analysis invokes us to focus on the influence of point defects in various films on thermal conductivity.

As mentioned above, Raman test is an effective way in the analysis of diamond film. The splitting observed is usually attributed to the existence of anisotropic stress throughout the films [30], which are probably generated by the inhomogeneous distribution of defects like stacking faults and dislocations [31]. This is consistent with above description that both of these defects aggregate preferentially at or near boundaries. By fitting the Raman spectra with Lorentzian line shapes [32–34], we can calculate the relative amount of diamond content (sp^3) and residual stress in the diamond films by using following equations [13,33]:

$$C_d = 100A_d / \left(A_d + \sum A_i / 50 \right) \quad (2)$$

$$\sigma = -0.567(\omega_m - \omega_0) \text{ GPa} \quad (3)$$

where A_d and A_i are the integrated area of the fitted curves corresponding to diamond peak and non-diamond peaks, ω_m is the average of singlet and doublet peaks and ω_0 is the unperturbed position of diamond peak.

The diamond content and residual stress in the diamond films of various composites are displayed in Fig. 5. The purity of diamond films increases from 86% to 91.5%, consistent with the columnar structure [24] revealed by the morphology characterization, i.e., with increasing crystal sizes and diminishing of crystal boundaries in the growth direction, the impurities that reside in the boundaries [27] show a decrease [35]. The decreasing of thermal conductivity in S1 (compared to substrate) could now be well explained by its low-purity and discontinuous film. While for S2 and S3, the improving thermal conductivity is the result of purer and continuous thick film. Moreover, due to the increasing surface continuity and film thickness [36], the residual compressive stress increase from 1.8 GPa to 3.3 GPa. The stress is probably resulted from the differences between the thermal expansion coefficients of diamond film ($\sim 1 \times 10^{-6}/\text{K}$) and BeO ($6.8 \times 10^{-6}/\text{K}$). It has been proposed in Adler's [37] simulation that the thermal conductivity of diamond films with residual stress are lower than those without. Since the buildup of stress would result in the increasing of defects concentration and change of point defects like intermediate carbons (isolated three-fold carbon atoms) [38] and split interstitials [39,40], leading to significant scattering of phonons and the comprehensive effect is the reduction of thermal conductivity. So that

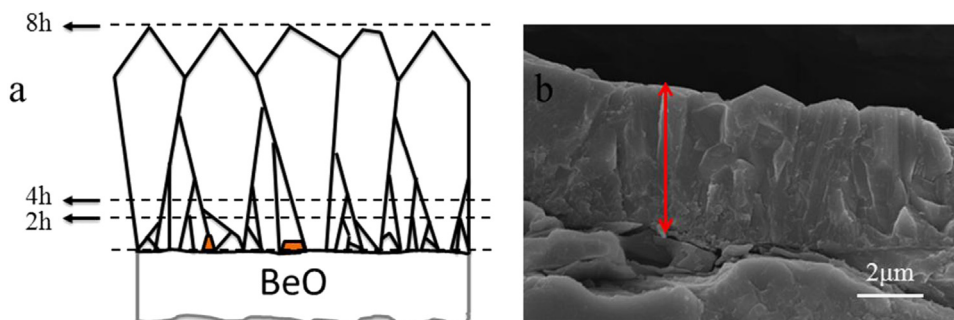


Fig. 4. (a) Schematic view of cross section morphology of CVD polycrystalline diamond film deposited on BeO. The orange parts represent the unfilled space (cavities) between diamond crystals. The lowest dashed line is the interface between film and substrate. The rest three lines give the thickness of films deposited for 2 h, 4 h and 8 h respectively. (b) Cross section image of the 5.0 μm film in our experiments. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

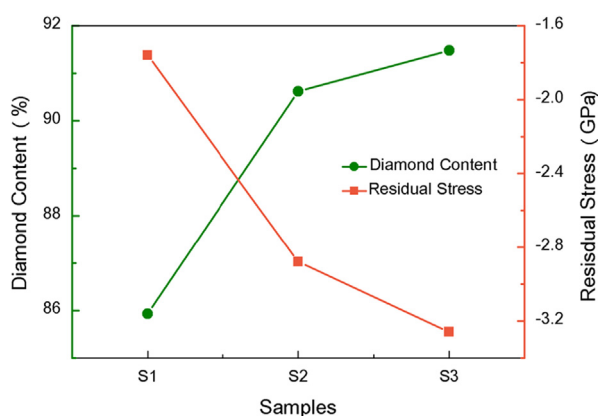


Fig. 5. Diamond content and residual stress in the films of various composites. The results are calculated by using Eqs. (2) and (3), based on the fitting results of Raman spectra with Lorentzian line shapes.

for films with larger stress, the concentration of these point defects would increase, resulting in greater reduction in thermal conductivity.

Considering the above analysis, we would like to give the following explanation. In the lower temperature range, the dominating factor that determines the thermal conductivity is the purity and continuity of diamond films, so that highest conductivity is obtained in S3. While in the temperature range of 398.15–653.15 K, the fact that the conductivity of S3 becomes slightly lower than S2 is ascribed to the stress-induced defects. Since phonon density increases with temperature, it is possible that the phonon density reaches a critical value at or near 398.15 K, concerning that the concentration of stress-induced defects in S3 is significantly higher than that in S2 due to the larger stress, the scattering of phonons from these defects becomes prominent enough to counteract (or even slightly overpassing) the improving effect of increasing purity and thickness. While for S2, this scattering is far less intense. In other words, due to the increasing of phonon density with temperature, the stress-induced defects become a pronounced factor for S3 in the higher temperature range. It also accounts for the stronger temperature dependence within S3 for the same reason.

So, the above discussion gives a guidance (or prediction) for the preparation and application of diamond films in the as-designed structure of the composites, i.e., if the composites (with continuous films) are to be applied at high temperature (> 400 K) and residual stress of diamond film unable to be eliminated or reduced, composites with thinner films will outweigh thick ones since they are equally efficient while much more time-saving and cheaper. Put another way, on the basis of minimizing residual stress, improving the film thickness would result in better thermal performance, especially in the high temperature range.

5. Conclusions

In summary, the thermal conductivity of diamond/BeO composites with different film thickness are investigated in the temperature range of 298.15–653.15 K. A maximum increase of 17.5% is obtained. The temperature dependence of the composite is explained by the comprehensive effects of films purity and residual stress, both of which vary with the change of thickness. At 296.15 K and 333.15 K, the fact that composite with the thickest diamond film exhibits the highest thermal conductivity is attributed to its high-purity thick film. While in the higher temperature range, the scattering of phonons by stress-induced defects in S3 (like intermediate carbons and split interstitials) surpasses the improving effect of its high purity and thickness, resulting in lower conductivity than S2. As a consequence, to achieve prominent performance in the high-temperature thermal management application, diamond films with high purity and low stress are preferred in the diamond/BeO composites.

Acknowledgments

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