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Dependence of RF power on the phase transformation for boron nitride films deposited on graphite at room temperature

Xuxin Yang, Hongdong Li^{*}, Yingai Li, Xianyi Lü, Shiyong Gao, Pinwen Zhu, Qing Zhang, Tiechen Zhang, Guangtian Zou

State Key Laboratory of Superhard Materials, Jilin University, Changchun 130012, China

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1. Introduction

Boron nitride (BN) is one of the most important III-V binary compounds. Besides the well-known cubic (cubic boron nitride (cBN)) and hexagonal phase (hexagonal boron nitride (hBN)), other phases have also been reported, including wurtzitic (wBN), amorphous (α BN), turbostratic (tBN), rhombohedral (rBN), and explosion BN phase (eBN). It is known that hBN, tBN, and rBN are sp²-bonded, wBN and cBN are sp³-bonded, whereas eBN is the mixed sp³- and sp²-bonded structure [1–3]. Among them cBN has attracted most attention for wide technical applications due to its excellent physical and chemical properties such as wide band gap, extreme hardness, chemical inertness against ferrous-based metals, high thermal conductivity, high melting temperature, and negative electron affinity (NEA), etc. [1,2]. To realize cBN films, silicon was generally used as the substrate, and recently, polycrystalline [4] and single crystal diamond [5] were also adapted for heteroepitaxial cBN growth. Another important substrate candidate is graphite for obtaining cBN films performed

E-mail address: hdli@jlu.edu.cn (H. Li).

ABSTRACT

Cubic boron nitride (cBN) thick films deposited on mainly *c*-axis-oriented graphite substrate at room temperature and zero bias by radio frequency (RF) magnetron sputtering were studied. In the growth process, RF power plays a key role in determining the content of cubic phase in films, while the conventional substrate heating and biasing have been neglected. With increase in RF power, the dominated content of films converts from explosion boron nitride (eBN) to cBN. The transformation mechanism has been discussed. The unique structural properties of the "soft" graphite are favorable to propose simple conditions for growing "hard" cBN films. Furthermore, the optical band gap of BN films having ~90% cubic phase is of ~5.8 eV obtained from ultraviolet–visible optical transmission measurement. The electron field emission examination shows that cBN film on graphite has a high emission current density of 2.8×10^{-5} A/cm² at an applied field of ~30 V/µm.

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at high temperature (>500 °C), though the content of cubic phase was low in the samples [6]. Regardless of various substrates reported in the literature, the substrate subplantations (such as heating, biasing, and/or assisting high energetic ion bombardment to growing surface) were generally required to increase the content of cubic phase in BN products, either by physical vapor deposition (PVD) or by chemical vapor deposition (CVD) [1,2,7]. To a certain extent, these subplantations are disadvantageous to the fabrication and applications for cBN-based optoelectronic devices. For instance, the subplantations might damage the raw substrate (device wafer) structure and/or accumulate large stress, leading to the appearance of crack or delamination [1,2]. Since the recent findings of single-atomic graphite sheet [8], researches on nanographite-based devices have become one of the top scientific issues. In this case, "friendly" processes are thus particularly desirable to establish new combined structures of semiconductor and "soft" graphite with novel properties, and applied in optoelectronic fields.

In this article, by radio frequency (RF) magnetron sputtering (MS) technique, thick BN films (\sim 1.2 µm) with a high concentration of cubic phase (\sim 90%) have been deposited on graphite performed at room temperature (RT) in Ar/N₂ ambient atmosphere. It is demonstrated that the RF power determines the

^{*}Corresponding author. Fax: +86 431 85168095.

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content of cBN in the products. The growth mechanism of cBN related to the unique properties of graphite is discussed. The wide band gap and electron field emission properties examined for the BN films with a high cubic-phase concentration suggest that the cBN/graphite system is available for optoelectronic applications.

2. Experimental

The BN films were deposited by MS at a frequency of 13.56 MHz. The growth processes were carried out at RT, and the RF power varied from 100 to 180W. The sputter target disk was 99.9% pure hBN with a diameter of 50 mm. Mainly c-axisoriented graphite foil was used as the substrate, and its surface was mechanically polished before deposition. The distance between the target and the substrate was 50 mm. The chamber base pressure was below 3×10^{-3} Pa. During the deposition, Ar (86%) and N₂ (14\%) gas mixture was injected into the chamber and the working pressure was 2 Pa. The structure and the morphology of the products were tested by means of Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), scanning electron microscope (SEM), and atomic force microscope (AFM). The optical band gap was studied by UV-vis transmission spectrum using a UV-3150 spectrophotometer. The electron field emission measurements were carried out using a diode configuration, a cathode (sample), and a parallel anode plate at a distance of 100 µm, performed in a vacuum chamber maintained at a pressure of 2×10^{-7} Pa.

3. Results and discussion

The typical FTIR spectrum of the sample prepared for 120 min with an RF power of 150 W is shown in Fig. 1. A strong peak centered at 1075 cm^{-1} appears with a full-width at half-maximum (FWHM) of 170 cm^{-1} . This absorption band can be assigned to the characteristic transverse optical (TO) mode of sp³-bonded cBN [5]. The corresponding intensity of the band is significantly higher than that centered at 750 cm^{-1} related to the out-of-plane B–N–B bending in poorly crystalline sp²-bonded hBN (tBN) [9]. It suggests that the cubic phase dominates in the sample. This fact is further supported by the corresponding B 1s XPS data (Fig. 2). The component of the B 1s core level is presented at 191.2 eV, in good agreement with the value of cBN



Fig. 1. FTIR spectrum of cBN film prepared with RF power of 150W for 120 min.



Fig. 2. B 1s XPS spectrum of the cBN film. The inset is B 1s peak fitted with cubic (a) and turbostratic (b) phase components.

reported in the literature [10,11]. The fitting curves of cBN (curve a) and tBN (curve b) components are shown in the inset of Fig. 2. Estimated by deconvolution of the core-level peaks, \sim 90% cBN in volume is realized in the product. Furthermore, the ratio between the content of boron to nitrogen is about 1:1, which means that the composition of the BN product is approximately stoichiometric. Fig. 3(a) shows the cross-sectional SEM image of the cBN film. Clearly, a continuous cBN layer of 1.2 µm thickness was deposited on graphite substrate with a clean interface. The corresponding growth rate is estimated to be $\sim 10 \text{ nm/min}$. Observed by AFM (Fig. 3(b)), the cBN layer prepared consists of nanocrystalline grains (average size: $\sim 20 \text{ nm}$) with a root mean square (rms) surface roughness of 2 nm. Compared to the previous work of cBN growth on graphite by pulsed-laser deposition (substrate heating $> 500 \,^{\circ}$ C) [6], thick cBN films with fewer noncubic phases are reported in this article.

In order to study the growth evolution of cBN, the films deposited with different RF powers were characterized by FTIR (Fig. 4). It is found that for the samples fabricated with lower RF power (<150 W), the IR spectra are generally asymmetric in the scaled region of $800-1300 \text{ cm}^{-1}$ (Fig. 4(a) and (b)). As an example shown in the inset of Fig. 4, fitted by the Lorentzian profile for Fig. 4(a), two peaks at 1083 and 1186 cm⁻¹ are mainly presented assigning to the characteristic vibration of cBN [5] and eBN [12,13], respectively. Evidently, in this case, the eBN-related peak dominates the spectra and the peak for cBN is presented as a shoulder. Besides the evidence of IR spectroscopy, the appearance of eBN has also been demonstrated by the analysis of electron diffraction patterns (not shown) obtained from TEM. These results mean that the products consist of eBN in the majority as well as a small amount of cBN. With increase in RF power (150 and 180 W), the mode originated from cBN (at $\sim 1070 - 1075 \text{ cm}^{-1}$) dominates the spectra, while the mode from eBN is quenched, as shown in Fig. 4(c) and (d). It suggests that higher RF power is helpful to increase the content of cBN in the films. In our experiments, 150 W is the threshold for depositing highly purified cBN on graphite substrate. It is worth mentioning that except for the IR peaks originated from BN phases, no peaks related to carbon-based bonding vibrations (e.g., C-N and B-C) are found in the spectra of most products, meaning that there are little carbon or carbonaceous impurities appearing in the products.

The cBN films deposited at RT and assisted with a series of substrate negative bias voltages (0, 40, 80, and 120 V) were also

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