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Deposition of cubic boron nitride films by anode layer linear ion source assisted radio frequency magnetron sputtering

Shuai Tian[a], Feng Xu[a,*], Peng Ye[a], Jinxin Wu[a], Yousheng Zou[b], Dunwen Zuo[a]

[a] College of Mechanical and Electrical Engineering, Nanjing University of Aeronautics and Astronautics, Nanjing 210016, China
[b] School of Materials Science and Engineering, Nanjing University of Science and Technology, Nanjing 210094, China

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ABSTRACT

Anode Layer Linear Ion Source (ALLIS) can improve the ionization rate of working gases and enhance the activity of gas molecules. In the present work, the ALLIS assisted Radio Frequency Magnetron Sputtering (RFMS) method was proposed to deposit cubic boron nitride (cBN) films, and the effect of ALLIS on the microstructure and mechanical properties of the deposited cBN films was investigated. The phase component, chemical composition, bonding states, surface topography, deposition rate and nanohardness of the cBN films were characterized respectively by Fourier transformed infrared spectroscopy, X-ray photoelectron spectroscopy, scanning electron microscopy and nanoindentation. The results indicated that the sp^3 phase content in cBN films increased at first and then decreased with the increase of ALLIS power, and the maximum value was 83% when the ALLIS power was 200 W, the surface quality and growth rate of cBN films were enhanced markedly. The hardness and elastic modulus exhibited the same trend to variation of cubic phase content. Finally, the effect of ALLIS on the cBN synthesis was discussed systematically.

1. Introduction

Cubic Boron Nitride (cBN) attracts people's great research interest in recent years due to excellent physical and chemical properties, such as high hardness, which is second only to diamond, very high thermal stability and chemical stability. Besides, cBN has high thermal conductivity and low dielectric constant. So the cBN crystal is very suitable to be used as cutting tools and semiconductor materials [1,2]. The synthesis of cBN films has been widely studied in the past decades by physical vapor deposition (PVD) and chemical vapor deposition (CVD). W.J. Zhang et al. prepared cBN films with high-purity and high adhesive strength on diamond transition layer using fluorine-assisted CVD method. The selective etching of F element to sp^2 phase BN makes the synthesis of high-purity cBN a reality [3,4], H. Chen et al. found that cubic phase in the BN films increase with the increase of ion energy and ion current density by a 3 cm Kaufmann-type ion-assisted pulsed laser deposition method [5]. J. S. Ko and K. Nose also obtained high quality cBN films by radio frequency (RF) sputtering method [6,7]. In these methods, radio frequency magnetron sputtering (RFMS) is a frequently-used technology in PVD method for preparing cBN films at present, because of easy control of deposition conditions and the as-deposited films with compact surface and low friction coefficient [8]. But the low gas ionization rate restricts the quality and deposition rate of cBN films. For the past few years, many scholars have paid more attention to auxiliary ion sources, such as Kaufman ion source [9], end-Hall ion source [10] and anode layer linear ion source (ALLIS) [11,12], and aimed to improve the ion concentration during deposition. The ion sources utilized the orthogonal electromagnetic field to increase the probability of collision between electrons and gas molecules to increase the gas ionization rate. W.R. Kim et al. [11] found that the amount of sp^3 bonding in DLC films increased at first and then decreased by changing the anode voltages from 800 V to 1800 V using ALLIS assisted PVD. S. Paskvale et al. [12] observed an increase of deposition rate and adhesion for DLC coatings with increasing discharge voltage of anode layer source. P. Ye et al. [13] found that ALLIS effectively improved the sp^3 carbon bond content, deposition rate and hardness of a-CN_x film using ALLIS assisted RFMS technology.

Compared with other auxiliary ion sources, ALLIS has the advantage of simple internal structure and lower cost. It is the ideal auxiliary ion source for RFMS to deposit films. Up to now, there are no reports about the cBN films deposited by ALLIS assisted RFMS technology. In this work, cBN films were deposited by ALLIS assisted RFMS method. The influences of ALLIS on the phase component, bonding states, surface morphology, deposition rate and nanohardness of as-deposited cBN films were investigated systematically.

* Corresponding author.
E-mail address: xufeng@nuaa.edu.cn (F. Xu).

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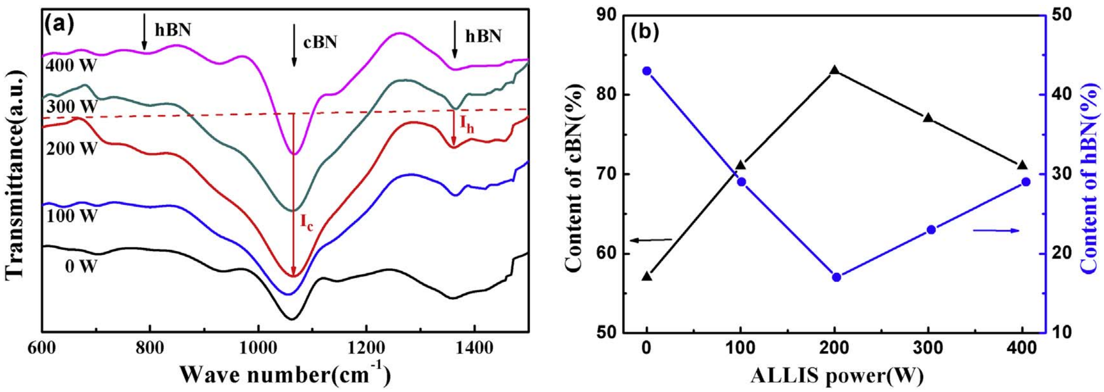

Fig. 1. (a) FTIR spectra of BN films deposited with various ALLIS power; (b) relative content of cBN and hBN with various ALLIS power.

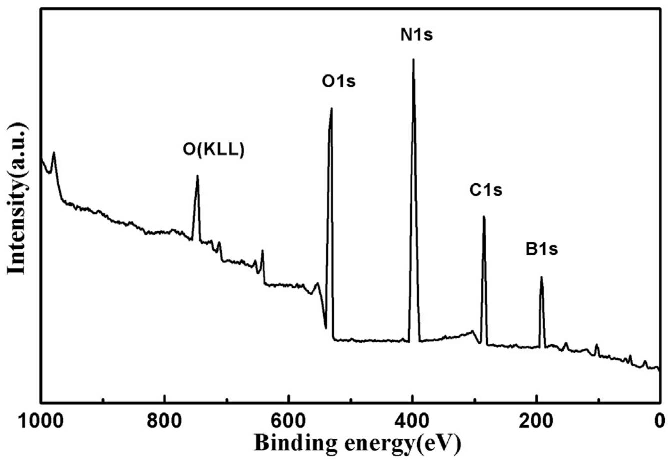

Fig. 2. The XPS spectra of the BN film.

2. Experimental details

The cBN films were deposited on (100) oriented Si wafers by ALLIS assisted RFMS (JSD450-III) method which was described in Ref. 13. The targets were sintered h-BN (with a purity of 99.99%), the working gases were Ar and N_2 (both with a purity of 99.99%), and the distance of target-to-substrate was 90 mm. The silicon wafer was cut to substrates with the shape of $1.0 \times 1.0\,cm^2$. First, the substrates were immersed into hydrofluoric acid solution (with a concentration of 5%) and ultrasonically cleaned for 15 min to wipe off the oxide on their surface. Then the substrates were cleaned ultrasonically in ethanol, acetone and deionized water for 10 min respectively to remove the surface contaminations. Finally the substrates were dried in dry nitrogen and then placed in the vacuum chamber for deposition. During deposition process, the background pressure was less than 5×10^{-4} Pa and deposition pressure maintained at 0.8 Pa, the flow rate of working gases was 35 sccm. The deposition process is divided into two steps, firstly, the substrates were preheated to the temperature of 900 °C and RF power was adjusted to 250 W. The substrates surface were bombarded with pure argon at ALLIS power 200 W and DC bias voltage −300 V to remove the contaminations and activate the substrate surface. After the pre-sputtering process, DC bias voltage was decreased to −220 V, the ratio of nitrogen to argon in gas mixture was 1:6 and deposition time was 2 h. The influences of ALLIS on cBN films were studied with ALLIS powers ranging from 0 to 400 W in deposition process.

The as-deposited cBN films were characterized by different detection techniques. Fourier transformed infrared spectroscopy (FTIR, Thermo Scientific Instrument Co, Nicolet 8700) measurements were used to check the formation of cubic boron nitride. The chemical composition and bonding states for the sample surfaces were characterized using a PERKIN-ELMER CHI 5300 X-ray photoelectron spectroscopy (XPS) with Al-Kα radiation line (1486.6 eV). The surface morphology and cross-sections images of the cBN films were obtained by scanning electron microscopy (SEM, Hitachi-S4800) to characterize the surface quality and deposition rate, the deposition rate was calculated by dividing the thickness by the deposition time. Finally, the hardness (H) and elastic modulus (E) were measured by nano-indentation (Agilent technologies, G-200) tests using continuous stiffness method (CSM).

3. Results and discussion

Fig. 1(a) shows the FTIR spectra of BN films prepared with different ALLIS power. It can be seen that hexagonal phase (hBN) infrared absorption peaks appear near 780 cm^{-1} and 1380 cm^{-1}, while the

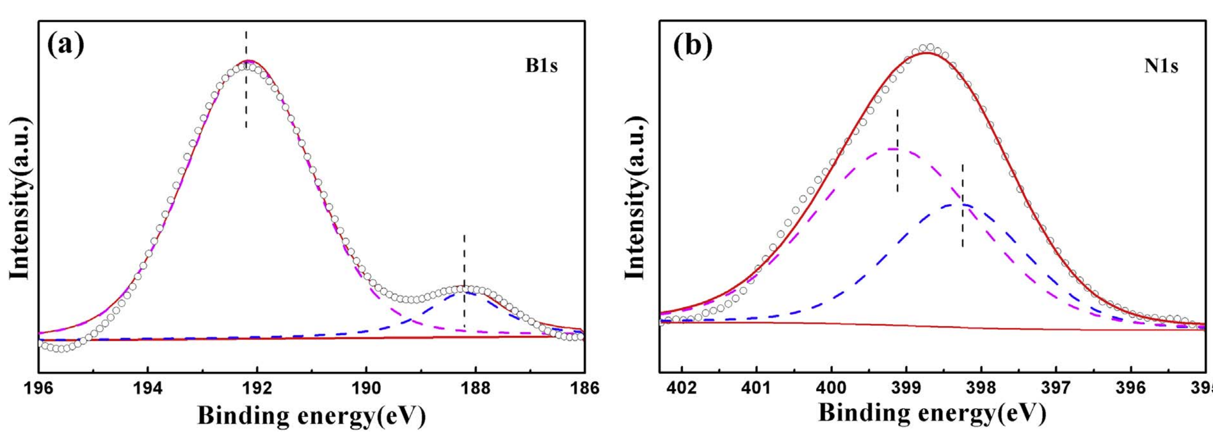

Fig. 3. XPS high resolution spectra of the BN film (a) B1s and (b) N1s.