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The influence of boron implantation into silicon substrate on the internal stress and adhesion strength of c-BN films

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Abstract

Cubic boron nitride(c-BN) films were deposited on silicon substrate with boron implanted buffer layer by RF-magnetron sputtering. The most serious problems for c-BN film is high residual stress and low adhesion strength to a substrate. In order to improve the adhesion of the c-BN film, the boron implanted buffer layer was introduced to improve the interface state between the film and substrate. The experiment results showed that the boron implanted buffer layer can reduce the internal stress and improve the adhesion strength of the films obviously. The critical load of scratch test rises to 44.5 N, compared to 7.5 N of c-BN film on the unimplanted silicon. Then the composition and organization of the boron implanted layer was analyzed by XPS. And the influence of boron implanted layer on the internal stress and adhesion strength of c-BN films were investigated.

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Keywords: Cubic boron nitride; Silicon substrate; Ion implantation; Internal stress

1. Introduction

Thin c-BN films are highly attractive, because their characteristics, such as hardness, wear-resistance and oxidation resistance, are outstanding. To date, a number of deposition techniques have been used to synthesize c-BN films, so c-BN thin films have been obtained successfully under optimal deposition conditions [1,2].

But thin film application of c-BN are mainly limited by its poor adhesion. One of the main problems is high residual compressive stress existing in c-BN film [3]. Irrespective of the deposition technique used, c-BN is only formed under a low energy ion irradiation. This ion bombardment leads to significant compressive stress in the layers [4]. To relax the stress to improving the adhesion to the substrate, Various types of interlayers have been used for adhesion improvement. These include conventional layers such as Ti, TiN, B/N or B/C/N used as buffer interlayer [5–7]. But all these interlayers will bring one weak interface between the film and substrate, which will make the delamination occurring between the interlayer and the substrate.

In this paper, boron implanted buffer layer was introduced to relax the compressive stress and improve adhesion of the c-BN films, which was prepared by recoil implantation techniques. The boron implanted buffer layer mixed with the silicon substrate very well, which avoids one additional weak interface. Then the influence of boron implanted buffer layer on the residual stress and adhesion strength of c-BN films was investigated.

2. Experimental details

c-BN films were prepared on silicon substrates by RF-magnetron sputtering. The schematic diagram of the apparatus used is shown in Fig. 1.

A target of hot pressed h-BN was adhered to the cathode. Radio-frequency power was applied to the silicon substrate to generate a negative self-bias voltage. The magnetic coil was used to produce magnetic field that could confine the movement of electric particles in the plasma so as to increase the density of plasma near target and substrate. Finally the c-BN films were deposited under the conditions given in Table 1.

The implanted buffer layer was formed by first depositing a pure boron layer about 100 nm on silicon substrate by one Kaufman ion sources. Then the argon ion implantation into

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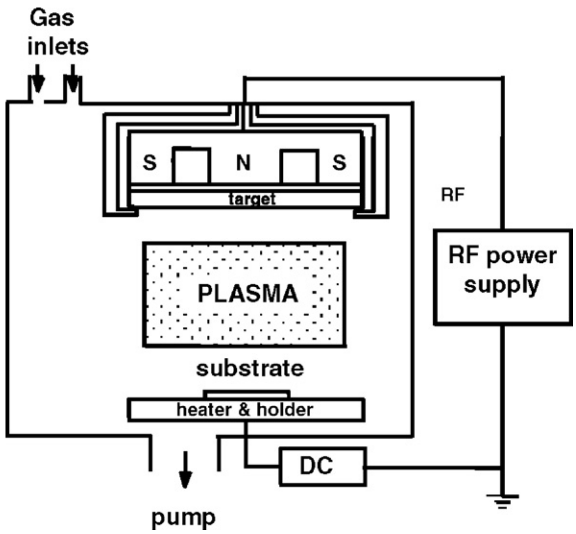

Fig. 1. Schematic illustration of the RF sputtering system for the deposition of BN films.

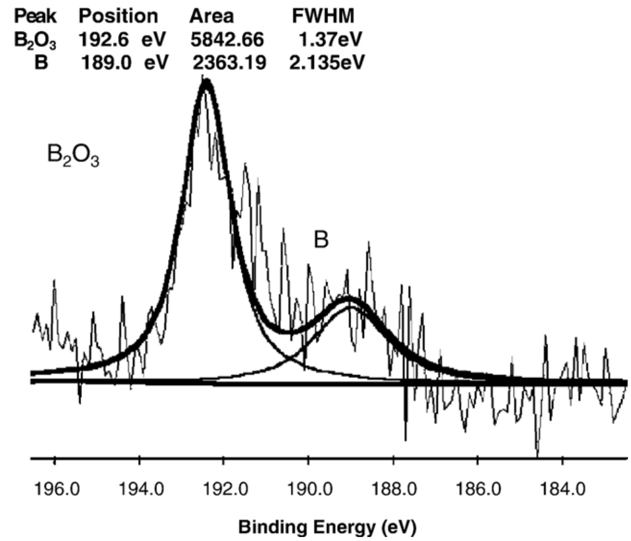

Fig. 2. XPS analysis of boron implanted buffer layer.

silicon substrate were carried out at the energy of 50 keV. And the argon ion dose were varied from 1.6×10^{17} to 12.8×10^{17} ions/cm^2 corresponding to implantation time from 0.5 h to 4 h. Finally the c-BN films were deposited under the conditions given in Table 1.

The composition and valence state of the boron implanted layer were analyzed using X-ray photo-electron spectroscopy (XPS). Phase identification of the BN films was characterized by Infrared Transform Absorption spectra (FTIR). And compressive stress in the films was evaluated by measuring the shift of the c-BN absorption peak The adhesive strength of c-BN films was determined with a scratch test apparatus and a Rockwell diamond stylus with a tip radius of 0.1 mm. Testing was conducted at a loading rate of 100 N/min with a scratch rate of 4 mm/min.

3. Results and discussion

3.1. XPS analysis of ion implanted layer

The composition and valence state of the boron implanted layer in silicon substrate was analyzed by XPS. According to XPS results, the variation of boron valence in implantation layer changed greatly with different argon dose. And when the argon dose equals to 9.6×10^{17} ions/cm^2, the boron valence in implantation layer showed in Fig. 2.

From Fig. 2, the XPS spectrum can be divided into two peaks by Gauss simulation: One peak is in 188.6–189.4 eV, which corresponds to pure boron element; another peak is in 192.5–193.4 eV, which corresponds to B_2O_3 oxide.

And the content of borides in the implanted layer increased with the increase of the argon dose. It can be seen from Table 2 that When it is 1.6×10^{17} ions/cm^2, the relative content of the single boron element is 86% and the relative content of borides is 14%. However, when the dose equals to 9.6×10^{17} ions/cm^2, the content of borides comes to 82% and the content of single boron element drops to 18%. And the existence of single boron element in the implanted layer may bring about favorable lubrication effects [8]. It will reduce the internal stress of the c-BN films greatly.

3.2. IR analysis of c-BN films

As a non-destructive method, FTIR can provide information on the phase composition of the films since cubic BN is characterized by a strong absorption peak about 1080 cm^{-1}, whereas h-BN exhibits absorption around 780 and 1380 cm^{-1}. And it is generally accepted that the intensity ratio $I_{1080}/(I_{1080}+I_{1380})$ provides a relative measure for the percentage of c-BN in the films [9]. And based on the strain-induced shifts of the TO-phonon frequency calculated by S. Fahy [10], the intrinsic strain in the c-BN layer due to compressive stress accumulated during film growth was evaluated by measuring the shift of the c-BN absorption peak as a function of film thickness. And the compressive stress will increase about 1 GPa when the wavenumber will increase at 4.5 cm^{-1}.

Fig. 3 shows infrared absorption spectra of the c-BN film on the boron implanted buffer layer with different argon implantation dose. The absorption bands appeared at approximately

Table 1
Experimental conditions for the deposition of c-BN films

Parameter	Pre-sputtering	Deposition
RF power (W)	350	300
Substrate bias voltage (V)	−300	−200
Substrate temperature (°C)	25	400
Ar/N_2 gas flow ratio	Pure Ar	5:2
Deposition time (min)	10 min	120

Table 2
The B1s analysis of implanted layer under different dose

Partition	1.6×10^{17} ions/cm^2	6.4×10^{17} ions/cm^2	9.6×10^{17} ions/cm^2
B (%)	86	53	18
B_2O_3 (%)	14	47	82