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Study on the preparation of boron-rich film by magnetron sputtering in oxygen atmosphere

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ABSTRACT

In this paper, the growth of boron (^{10}B) oxide films on (1 0 0) silicon substrate were achieved by radio frequency (r.f.) magnetron sputtering under the different oxygen partial pressure with a target of boron and boron oxide. The structure and properties of deposited films were characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy spectrometer (FTIR), X-ray photoelectron spectroscopy (XPS), respectively. The results showed that the substrate was covered with boron-rich films tightly and the surface of films was covered with B_2O_3. And the growth mechanism of boron-rich film in oxygen atmosphere was also analyzed.

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1. Introduction

Boron, as an oxyphilic element, exists only in the forms of oxide (B_2O_3) and fluoride (BF_3) under natural conditions. Boron and boron-rich boride films have attracted considerable attention in the past few years for their unique properties arising from their unusual three-center electron-deficient bonds [1–3]. These fascinating properties include high melting point (~2500 K) with low density, high hardness close to diamond, and excellent thermoelectric property. In addition, the previous study also shows that boron or boride containing a certain amount of ^{10}B is also an ideal conversion layer material for neutron radiation detectors [4–6]. Since ^{10}B (n,α) ^{7}Li nuclear reaction is considered to be a most widely used neutron conversion method, uncharged neutron can be changed into charged α particles by the nuclear reactions mentioned above, then α detector detects neutrons indirectly.

Boron and boron-rich boride films have been successfully prepared by several methods. For instance, pure boron coating films were prepared successfully by chemical vapor deposition (CVD) technique [7,8]. McGregor et al. utilized evaporation method to product B films and the thickness of the films ranged between 1000 and 18400 Å [9]. Wang et al. employed laser ablation method to fabricate B films [10]. As compared to other methods, sputtering, especially direct current (DC) magnetron sputtering, with advantages of controllable preparation processes, low cost and high stability, is more suitable for industrial process.

In this work, boron oxide films have successfully been prepared on (1 0 0) Si substrate by reactive magnetron sputtering methods. The effect of oxygen partial pressure on properties of boron oxide films is also discussed.

2. Experimental

Boron-rich films were deposited on Si (1 0 0) substrates by radio-frequency (r. f.) magnetron sputtering. An 76-mm-diameter target with the mixture of boron and B_2O_3 ($B{:}B_2O_3 = 2{:}3$ w.t.) was employed. Prior to deposition, the base pressure of the deposition chamber was kept below than 1×10^{-3} Pa. During deposition, the Ar pressure, sputtering power and the distance between substrate and target were kept at 0.5 Pa, 200 W and 50 mm, respectively. All the samples were prepared at room temperature for 2 h. The effect of oxygen partial pressure in the boron-rich film preparation was studied by different flow ratio of oxygen to argon (O_2/Ar), such as 0, 10:90, 20:80.

The structure, chemical composition and relative content of boron-rich film were measured by X-ray diffraction (18KW-D/MAX2500V+/PC), FTIR spectrometer (AVATAR-380 FTIR) and X-ray photo-electron spectrometer (ESCALAB 250Xi).

3. Results and discussion

Fig. 1 shows the growth rate of boron-rich films under different oxygen concentrations of $O_2/(O_2 + Ar)$. The decrease of growth rate

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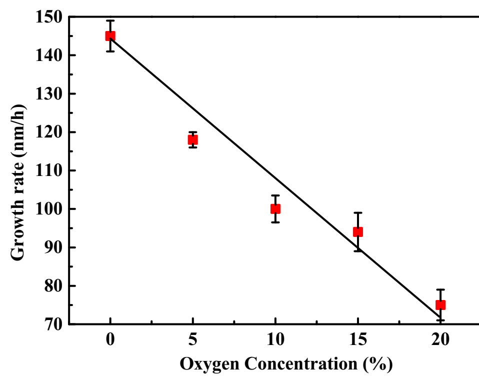

Fig. 1. The growth rate of boron-rich films under different oxygen concentration of $O_2/(O_2 + Ar)$.

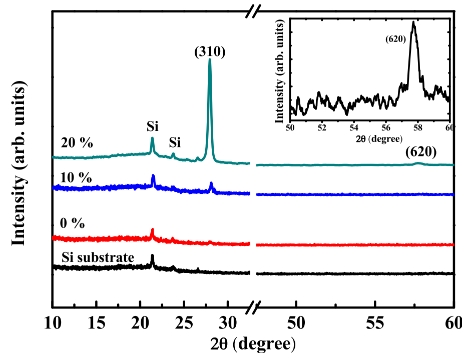

Fig. 2. XRD spectra of boron-rich films under different oxygen concentration of $O_2/(O_2 + Ar)$.

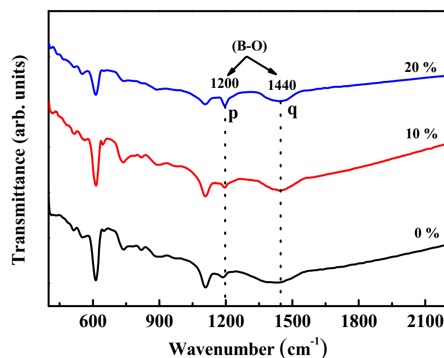

Fig. 3. IR spectra of boron-rich films under different oxygen concentration of $O_2/(O_2 + Ar)$.

linearly with oxygen concentration is caused by the decrease of ionization rate of argon ion and then the lowering of sputtering yield.

Fig. 2 shows the XRD spectra of boron-rich films deposited under different oxygen concentrations of $O_2/(O_2 + Ar)$. Only the diffraction peak at 28.0°, corresponding to the (3 1 0) plane of B_2O_3 crystal, indicates well (3 1 0) preferential orientation of films. Moreover, the (3 1 0) peaks increases with the increasing of oxygen concentration obviously, and the peak at 2θ = 57.64°, corresponding to (6 2 0) orientation, appears under the oxygen concentration of 20%. The grain size of samples, calculated using Scherrer formula, is 19.02 nm, 31.04 nm and 24.77 nm, respectively.

In order to study the changes of chemical bonds in the boron-rich film, three samples are characterized by AVATAR-380 FTIR spectrometer (Fig. 3). The main absorption bands of boron-rich films in the range of 400–2200 cm^{-1} locate at 1200 cm^{-1} (p peak) and 1440 cm^{-1} (q peak). This two absorption peaks correspond to the B—O group and a chain of B—O—B group [11]. The absorption peak frequencies of two peaks are listed in Table 1.

Table 1
IR data of boron-rich films under different oxygen concentration.

Oxygen concentration	0%	10%	20%
Wavenumber (cm^{-1})	1186.07	1193.78	1195.71
Intensity of p peak/I	0.26	0.34	1.23
Intensity of q peak/I	0.65	0.57	0.54

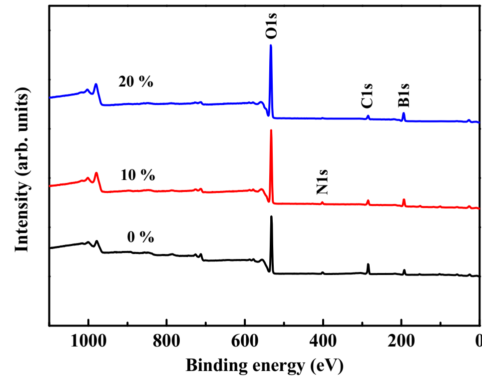

Fig. 4. XPS survey spectra of boron-rich films under different oxygen concentration.

A slight blue-shift of the absorption peak can be seen with increasing the oxygen concentration, as show in Table 1. The reason of this phenomenon can be explained by the increasing of B—O bonding strength with the oxygen concentration [12]. The absorption band at 1107 cm^{-1} corresponds to the absorption peak of Si substrate [13]. The relative intensity of p and q peaks is normalized to this Si peak. It is also worthwhile noting that the intensity of p peak increases with oxygen concentration while the intensity of q peak decreases.

Fig. 4 shows XPS survey spectra of boron-rich films. For all XPS analysis, the C1s (284.6 eV) from surface contamination is chosen to be a reference energy [14–17]. Only O, C, B and a small amount of Si and N elements on the surface of the films can be found. The C, O and N come from the contamination under air environment. The appearance of Si signal from the substrate likely indicates that the surface coverage for samples with the oxygen concentration of 0% and 10% is not complete.

Fig. 5 shows the B1s and O1s XPS spectra of B_2O_3 of boron-rich films, from which it can be seen that with the increase of oxygen concentration the B1s peak moves toward the direction of high energy. The reason for this is that the boron is more oxidized with the increase of oxygen concentration, well consistent with the FTIR absorption spectra. It makes boron atoms lost more electron and leads to the peak moves toward high energy [18].

The relative content of elements in Table 2 is calculated from Fig. 5 according to elemental sensitivity factors. It can be seen that the B atomic percent of sample obtained without oxygen is much closer to the natural B_2O_3 (40%). With the increase of oxygen concentration, the content of B on surface further increases.

In order to further study the film's composition in depth profile, the etching process was used. The process was divided into two steps: the first step, the etching energy is kept at 2000 eV, and continuous etching is carried out for 10 times with each etching time is 30 s; the second step, etching energy is 2000 eV, and continuous etching is carried out for 5 times with each etching time is 60 s. The etching rate of standard sample is 0.29 nm/s [19]. All layers from the surface layer to the last layer are, respectively, denoted as L0-L15. L1, L6, L9, L15 are selected to list in Fig. 6.

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