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Preparation of highly *c*-axis oriented AlN films on Si substrate with ZnO buffer layer by the DC magnetron sputtering

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ABSTRACT

Highly *c*-axis oriented AlN thin film was manufactured on Si (111) substrate using ZnO buffer layer by the direct current (DC) magnetron sputtering method. The X-ray diffraction results showed that the AlN/ZnO thin films had a perfect *c*-axis preferred orientation, and the full width at half maximum (FWHM) of the rocking curves of the (002) AlN peak decreased remarkably. Atomic force microscopy displayed that the AlN films with ZnO buffer layer had a dense, uniform and crack-free uniform microstructure compared to the microstructure of the AlN films grown on Si substrate. The average grain size and RMS surface roughness of the ZnO layers were, respectively, 90 nm and 3.4 nm. The scanning electron microscopy images showed that the AlN/ZnO thin films presented an obvious and quite uniform columnar structure, which are well aligned to the surface normal direction. The current–voltage curves results indicated that the ZnO buffer layer highly improved the insulating properties of the AlN films.

1. Introduction

Recently, surface acoustic wave (SAW) devices have been widely employed in a variety of communication systems due to their importance for telecommunication and sensor applications. However, SAW devices with high frequency ranges, low insertion loss, large electromechanical coupling factor (k_s^2), and stable follow temperature are necessary for the above mentioned applications [1]. Many researchers have been endeavored to propose or implement different kinds of layered SAW devices [2,3]. Among the piezoelectric materials developed for SAW devices, AlN films have been shown to be satisfactory for SAW applications because of their high acoustic velocity, high electrical resistivity, high thermal conductivity, and good piezoelectric characteristics [4]. In view of its different applications, AlN films have been deposited on various substrates such as tungsten [5], sapphire [6] and diamond [7] substrates. In the meantime, AlN and ZnO are also promising material for spintronics since it can possess the ferromagnetic properties. Boris et al. [8,9] has done a great deal of works about the ferromagnetic properties of nano-grained pure and Mn-doped ZnO films. Their results implied that oriented growth and crystal quality were needed for thin film device in order to take advantage of the best properties for a given application. Now, several researches on the influence of buffer layers on the structure and SAW properties of the AlN films have been reported [10–12]. However, less work about the preparation of AlN films on Si substrate with ZnO buffer layer were reported so far. It is possible that AlN and ZnO films are continuously deposited since both of them can be prepared by sputtering method. They are not only having the same crystal structure, but also with small lattice mismatch.

In this work, highly *c*-axis oriented AlN films were successfully deposited on Si substrate with ZnO buffer layer by the DC magnetron sputtering method. X-ray diffraction (XRD), atomic force microscopy (AFM), and scanning electron microscopy (SEM) measurements were performed to investigate the structure, composition and surface morphology of the AlN/ZnO films.

2. Experimental details

The AlN/ZnO films were fabricated by the magnetron sputtering method on Si substrates. Firstly, the ZnO thin films were deposited on Si substrates with the RF magnetron sputtering in gas mixture of Ar and O_2. The deposition conditions of the ZnO buffer layer were listed in Table 1. Then the AlN thin films were deposited on ZnO layers with the DC magnetron sputtering in gas mixture of Ar and N_2. The detailed parameters were also shown in Table 1. The AlN/ZnO bilayered films were continuously deposited in same apparatus. The purity of all gases was 99.999%. Both aluminum and zinc targets were 4 cm in diameter and their purity was 99.99%. The substrates used in this experiment were Si (111) wafers ($1.0 \times 2.0\ \text{cm}^2$). Degreasing of substrates was carried out in the ultrasonic baths of acetone, ethanol, and

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de-ionized water, successively. The substrates were blown out in dry N$_2$ gas and immediately inserted into the vacuum chamber.

X-ray diffraction (XRD, BEDE D1) analysis was performed to reveal the crystal structure of the films and their preferred orientation by using Cu Kα radiation in the mode of θ–2θ scan. The surface morphology was imaged by atomic force microscopy (AFM, Seiko SPA-300HV) and the scanning electron microscope (SEM, HITACHI S3400). The current–voltage (I–V) characteristics were measured using a standard ferroelectric test system (Radiant Precision LC2000).

3. Results and discussion

The structures of AlN/Si and AlN/ZnO bilayered films were characterized by XRD analysis. Fig. 1(a) showed the XRD profiles of $\theta-2\theta$ scans. Only the peak revealing (002) plane was observed, which suggested that the two samples had *c*-axis preferred orientation normal to the substrates. However, the AlN films deposited on the Si substrate showed a weak and broad (002) AlN peak at $2\theta=36.19^\circ$. The AlN films grown on Si substrate using a ZnO buffer layer had a stronger and sharper (002) AlN peak, which indicated that the ZnO buffer layer was conductive to obtaining highly *c*-axis-oriented structure for the AlN films. The results also could be confirmed from the Fig. 1(b). Fig. 1(b) showed the full width at half maximum (FWHM) of the rocking curves of the (002) AlN peak. The value of FWHM of AlN/Si (002) rocking curve measured by X-ray ω scan was 5.2°. This meant that the *c*-axis orientation of AlN deviates from normal to the substrate. Nevertheless, the value greatly decreased to 3.8° when the AlN films grown on ZnO buffer layer, which indicated that the crystal quality of AlN thin film was remarkably improved as a result of the ZnO buffer layer. The average grain size of the ZnO films was estimated from the full width at half maximum (FWHM) of the (002) XRD peak by using the well-known Scherrer's formula [13]. The calculated grain size of the AlN films grown on Si substrate was 39 nm, while for the AlN films grown on Si substrate using a ZnO buffer layer, the size was 79 nm.

Table 1
Processing parameters for AlN/ZnO thin films employed during DC sputtering.

Target used	Zn	Al
Target size	110 mm in diameter, 4 mm thick	110 mm in diameter, 4 mm thick
Base pressure	4×10^{-4}Pa	4×10^{-4} Pa
Plasma pressure	0.5 Pa	0.4 Pa
Sputtering power	RF:110 W	DC:110 W
Sputtering gas (sccm)	Ar:O$_2$=10:4	Ar:N$_2$=7:3
Substrate temperature	350 °C	650 °C
Target-substrate distance	100 mm	50 mm

Atomic force microscopy in tapping mode was employed to characterize the surface morphology of all layers. Fig. 2(a) and (b) shows the plan view of the AlN/Si films and AlN/ZnO films, respectively, and Fig. 2(c) and (d) shows the two-dimensional AFM images for them. It was noted that the grain size of ZnO films analyzed from AFM was larger than that from XRD. However, it can be seen from Fig. 2(a) that the microstructure of the AlN films without ZnO buffer layer was composed of small and nonuniform crystalline grains; Besides, there was a small quantity of grain clumps as shown in Fig. 2(a). The average grain size was 50 nm. Fig. 2(c) indicated that the root-mean square (RMS) surface roughness was 7.6 nm. Fig. 2(b) and (d) showed the AlN films with ZnO buffer layer had a dense, uniform and crack-free uniform microstructure. Furthermore, the crystal face of the AlN films could be observed obviously in the Fig. 2(b), which suggested that the ZnO buffer layer could help grow AlN films along (002) crystal orientation. The average grain size and RMS surface roughness of the AlN layers were respectively, 90 nm and 3.4 nm.

The SEM photographs of the cross-section (a) and the surface (c) for the AlN films without ZnO buffer layer were displayed in Fig. 3. Fig. 3(b, d) shows SEM cross-sectional views of the microstructure for the AlN films with ZnO buffer layer and the corresponding surface morphology, respectively. It can be seen from the Fig. 3(a) that the thickness of the AlN films was about 1.5 μm and there was no clear columnar structure in the films. From the surface, as shown in Fig. 3(c), it was visibly observed that the films were composed of nonuniform AlN grains, and a few excessively grown-up grains appeared in the surface. The result may be caused by the grain clumps, as the above analysis of AFM results. However, the AlN films grown on Si substrate with ZnO buffer layer showed an obvious and quite uniform columnar structure, which are well aligned to the surface normal direction as shown in Fig. 3(b). The grain boundary intensity of the sample was higher than the AlN films without ZnO buffer layer. Some researchers have demonstrated recently that the adsorption capacity of the grain boundaries was rather high and thus the total Mn solubility increases as compared to that in the single crystal [14,15]. The ferromagnetic properties of the films will be improved effectively. In spite of this, it also can be found that the thickness of the ZnO buffer and the AlN films was respectively about 150 nm and 1.5 μm, and there was a clear interface between ZnO and AlN films. The surface was covered with fine and uniform AlN grains, and a continuous AlN film was obtained as shown in Fig. 3(d).

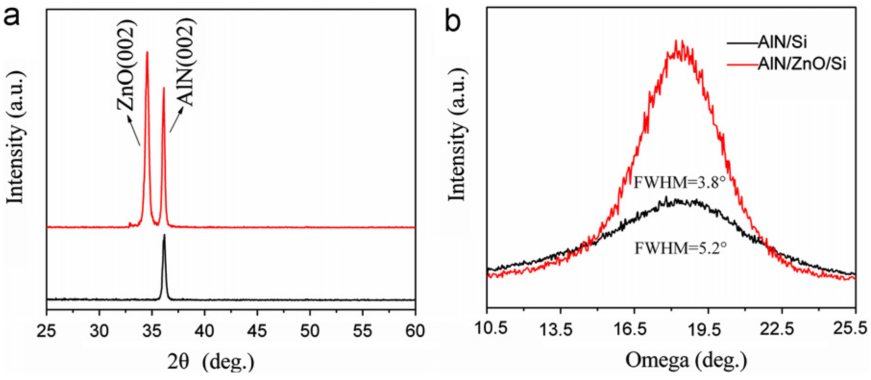

Fig. 1. (a) X-ray diffraction θ–2θ pattern and (b) rocking curve of the AlN films.