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The influence of the AlN film texture on the wet chemical etching

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ABSTRACT

The influence of the aluminum nitride (AlN) film texture on the chemical etching in KOH solution was invested. The AlN films with the different texture and crystal quality were prepared by sputtering. It is found that the chemical etching behaviors, including the etch rate, the activation energy, the surface morphology and the anisotropy, are strongly dependent on the film texture. There is a faster etching in the case of mixed (1 0 0) and (0 0 2) texture and a lower rate in the case of only (0 0 2) texture. The etch rate also decreases with the crystal quality. The sample with the only (0 0 2) texture forms discontinuous column structure after etching and exhibits lower porosity compared to that of the mixed (1 0 0) and (0 0 2) texture. Due to the strong anisotropy of the AlN wurtzite structure, the morphology of the film deposited at 700 °C shows the homogeneous pyramid shape after etching. The cross-section micrographs of etching patterns indicate that the anisotropy of the chemical etching is improved with the improving of the crystal quality.

1. Introduction

With the tremendous progress of III-nitrides research in terms of both fundamental understanding as well as devices applications, aluminum nitride (AlN) attracts increasing interest due to the band gap of approximately 6.2 eV and some excellent properties. The direct wide band gap is very attractive for the ultraviolet light-emitting diodes [1] and the photo detectors [2]. The high thermal conductivity (340 W m^{-1} K^{-1}) makes AlN suitable for the high power applications [3]. Furthermore, the polycrystalline AlN films have been utilized to fabricate the piezoelectric sensors/actuators [4] and the high-frequency electro-acoustic devices [5] due to the piezoelectric properties and high acoustic velocity. In the devices fabrication, the patterning of the materials is a key step because a number of device performances may be affected by the etching process. Generally, the reactive ion etching using chlorine can be highly anisotropic, an ideal characteristic for producing vertical profiles [6]. However, due to the strong physical component and the high chemical activity of the chlorine plasma, the dry etching has low etch-selectivity between materials and can cause subsurface damage by ion bombardment. In contrast, the wet etching, produces negligible damage, can be highly selective, is relatively inexpensive, and can be done with simple equipment. In addition, wet chemical etching is a reasonable, reliable, and simple method to analyze the defects and crystal polarity in III-nitrides [3]. Unfortunately, there was relatively little success in developing wet etching solutions for AlN because of their excellent chemical stability. The chemical etching was strongly dependent on the crystal quality and the etch temperature. Only KOH or NaOH containing solution can be etch epitaxial and single crystal AlN at the temperature below 80 °C [3,7]. The hot H_3PO_4 [8], and AZ400 K photoresist developer [9] were also reported can etch the polycrystalline AlN. However, more detail information about etching behaviors of polycrystalline AlN films is required for the micro-device fabrication.

In this paper, the influence of the film texture on the chemical etching in KOH solution was invested. The AlN films with the different texture and crystal quality were prepared by sputtering. It is found that the etch rate, the activation energy and the morphology of etching surface depend on the films texture and the crystal quality. The micrographs of the patterns were observed from the top view and the cross-section view to detect the etching evolution and anisotropy.

2. Experimental details

The AlN films were deposited using a RF sputtering system (ANEIVA SPF-210F). The target was a 4-in-diameter aluminum with 99.99% purity. Prior to the AlN deposition, the 300 nm W film was sputtering, on in. Si (1 1 1) wafers as the bottom electrodes. The chamber was evacuated until the base pressure decreased to

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Table 1
Sputtering parameters for AlN films deposition

Target-to-substrate spacing	6 cm
RF power	50–150 W
Pressure	0.55 Pa
Ar flow rate	5 sccm
N_2 flow rate	5 sccm
Substrate temperature	RT-600 °C

$<6\times10^{-5}$ Pa. High-purity argon was then introduced and pre-sputtered the target for 15 min before the film deposition. The sputtering parameters were summarized in Table 1. The thicknesses of all the films were controlled to about 1 μm. In order to investigate the effect of the crystal quality on the etching behaviors, the substrate temperature was turned from room temperature (RT) to 600 °C. The AlN etchant used in the experiment was 10 wt% KOH solution. In order to demonstrate the suitability of this wet etching for device applications, the test patterns were formed. The Ti layer with the thickness of 200 nm was deposited on the AlN surface and patterned by the conventional photolithography method as the mask for patterning AlN. The Ti layer is a better candidate mask for AlN-based devices because the high cohesion to AlN and the stability in AlN etchants.

The etch depths were obtained by Dektak stylus profilometer at different position after the removal of the mask with an approximate 5% error. The crystal structure and the texture of the films were identified by X-ray diffraction (XRD) and rocking curve (BRUKER-AXS) at wavelength of 0.15418 nm (Cu Kα). The morphological investigations were performed using field emission scanning electron microscopy (FE-SEM, FEI SIRION 200).

3. Results and discussion

3.1. The influence of the film texture

Fig. 1 displays the XRD patterns of the AlN films for three RF power levels. The substrate temperature was RT. In the case of 50 and 100 W, the diffraction peaks of AlN (1 0 0) and (0 0 2) were observed at 2θ around 33° and 36°, respectively. The peak at 40.3° corresponds to the W (1 1 0) orientation. With the increasing of RF power, the (0 0 2) peak becomes stronger and sharper, while the (1 0 0) peak becomes weaker. The sample deposited at 150 W exhibits the only (0 0 2) texture with the full-width at half-maximum (FWHM) of 0.31°, which indicates the preferred *c*-axis orientation. The result can be explained by that the AlN complex particles have sufficient kinetic energy to move to the lowest energy state and advantage the formation of the (0 0 2) texture crystalline structure at high power.

Fig. 2 shows the etch rates as a function of temperature in KOH solution for the films with the different texture. During etching, the bubbles were observed on the sample surface; meanwhile the white floccules were formed in the etchant. The etching occurred by the following reaction formula [10]:

$$AlN + 6KOH \rightarrow Al(OH)_3\downarrow + NH_3\uparrow + 3K_2O\downarrow$$

As expected, the etch rates of all the films increase sharply as the etch temperature increases from 30 to 80 °C. The etch rate of the film with the only (0 0 2) texture is around 55 nm/min at 30 °C and increase to 420 nm min^{-1} at 80 °C, which is faster than the reported value of the epitaxial films and single crystal [3]. The etch rate of the films with the mixed (1 0 0) and (0 0 2) texture is obviously higher than the case of (0 0 2) texture. Combining the etching rates and the XRD patterns, it can be concluded that the (1 0 0) plane are etched preferentially compared to the (0 0 2) plane. This selective etching behavior was also observed in AlN (1 0 1) and (0 0 2) planes by Ababneh et al. [11]. For the (0 0 2) texture, the *c*-axis is normal to the substrate and the plane parallel to the substrate is the close-packed basal plane, with either all aluminum or nitrogen atoms. Other planes should form the lesser-packed atomic array [12] and will suffer higher etch rate than the (0 0 2) plane in the etchant.

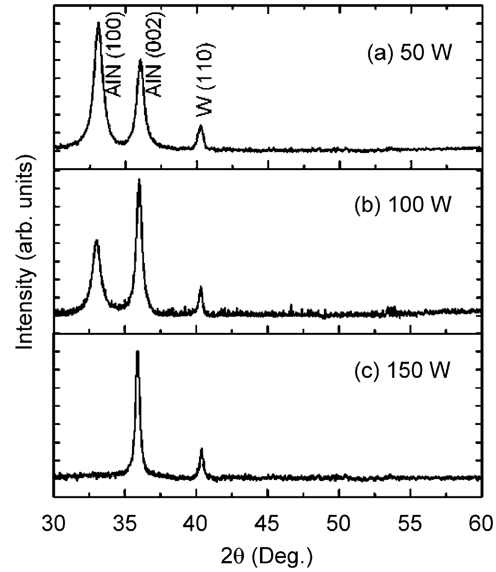

Fig. 1. The XRD patterns of AlN films prepared in three RF power levels. (a) 50 W; (b) 100 W and (c) 150 W. The substrate temperature was RT.

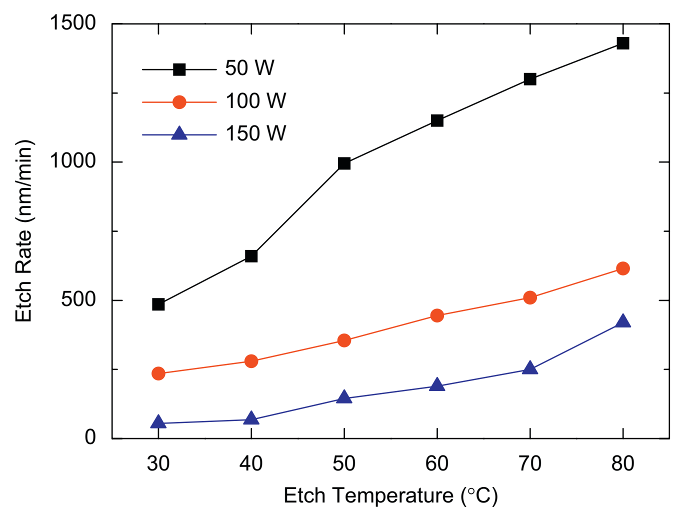

Fig. 2. The etch rate as a function of temperature in 10 wt% KOH solution for the films deposited at different RF power. (a) 50 W; (b) 100 W and (c) 150 W.

3.2. The effects of crystal quality

To get more detailed understanding for the correlation between the etching behaviors and the film texture, the substrate temperature was turned from 300 to 600 °C to get the higher (0 0 2) texture. The crystal quality was identified by X-ray rocking curve. As shown in Fig. 3, the rocking curves strengthen in intensity and become narrower with the increasing of substrate temperature, which means the reducing of defects and the improvement of crystal quality.

A parameter of interest for the etching experiments is the activation energy E_a. This is generally obtained by fitting an