The etching of *a*-plane GaN epilayers grown by metal–organic chemical vapour deposition*

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Morphology of nonpolar $(11\bar{2}0)$ *a*-plane GaN epilayers on *r*-plane $(1\bar{1}02)$ sapphire substrate grown by low-pressure metal–organic vapour deposition was investigated after KOH solution etching. Many micron- and nano-meter columns on the *a*-plane GaN surface were observed by scanning electron microscopy. An etching mechanism model is proposed to interpret the origin of the peculiar etching morphology. The basal stacking fault in the *a*-plane GaN plays a very important role in the etching process.

Keywords: crystal morphology, stacking fault, nonpolar GaN, chemical etching

PACC: 7280E, 7360L

1. Introduction

GaN-based semiconductors have recently attracted considerable attention due to their potential applications for visible or ultraviolet light-emitting diodes (LEDs), laser diodes and high-power transistors grown on either *c*-plane sapphire or SiC.[1,2] However, the wurtzite crystal structure group-III nitrides exhibit large spontaneous and piezoelectric polarization along the *c* direction. These fields are useful in the formation of two-dimensional electron and/or hole gases in order to achieve a higher carrier density. At the same time, with the spatial separation of electrons and holes in quantum well structures with *c*-axis orientation, the emission wavelength is redshifted due to the quantum confined Stark effect.[3,4] The polarization effects could be avoided by growing device structures along non-polar orientations. Therefore, the study and development of the nonpolar *a*-plane GaN is promising to improve the luminous efficiency of the LED.

The wet etching techniques are extensively used for defect study in the *c*-plane GaN due to its advantage of low cost and simple experimental process.[5−7] At the same time, the mechanism and morphology after chemical etching with regard to *a*-plane GaN epilayer are still lacking. In this paper, the etching behaviour in KOH solution for the GaN films prepared by metal–organic vapour deposition (MOCVD) has been investigated. The scanning electron microscopy (SEM) was used to study the morphology surfaces and to understand the dependence of etching property on the polarity. Based on the SEM analysis, an appropriate model is proposed to explain the mechanism of the selective etching on nonpolar *a*-plane GaN surface by KOH solution.

2. Experimental procedure

Growth of *a*-plane GaN films was achieved on *r*-plane $(1\bar{1}02)$ sapphire substrates using a cold-wall showerhead MOCVD system. After chemical cleaning, *r*-plane sapphire substrates were loaded into the chamber. Hydrogen was used as the carrier gas and triethylgallium, trimethylaluminium and ammo-

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nia (NH_3) were used as source compounds. A low temperature AlN nucleation layer of 25 nm, a high-temperature AlN layer of 100 nm and a 200-nm-thick high-temperature AlN/AlGaN SLs layer with five periods were grown at 620 °C, 1050 °C and 1020 °C, respectively. After buffer layer growth, subsequently a 1.5-μm thick a-plane GaN epitaxial layer was grown. For comparison, the Ga-face ($+c$) GaN and the N-face ($-c$) GaN were also grown on the c-plane sapphire, and then the substrate was cleaned under H_2 ambient at 980 °C. The Ga-face GaN and the N-face GaN are distinguished by nitridizing after the nuclear layer growth. Then, a 1.5-μm thick GaN epilayer was grown. Wet etching was performed in KOH solution for 30 min at 100 °C.

3. Results and discussion

The morphologies of as grown a-plane and c-plane GaN is shown in Fig. 1(a). The anisotropic surface texture is present in a-plane GaN, the stripe features along the c direction and surface undulation along the m direction [$1\bar{1}00$]. This morphology is the typical morphology of nonpolar materials grown on the nonpolar GaN/sapphire substrates. The anisotropic of our a-plane is not so pronounced which indicates the improved quality of our materials by the SLs layer. As shown in Fig. 1(b), the surface of the Ga-face GaN is very smooth. The morphology in Fig. 1(c) is the typical surface morphology of the N-face polar GaN, and the typical characteristics of the N-face GaN are the hexagonal features including hexagonal hillocks and platelets.[8,9]

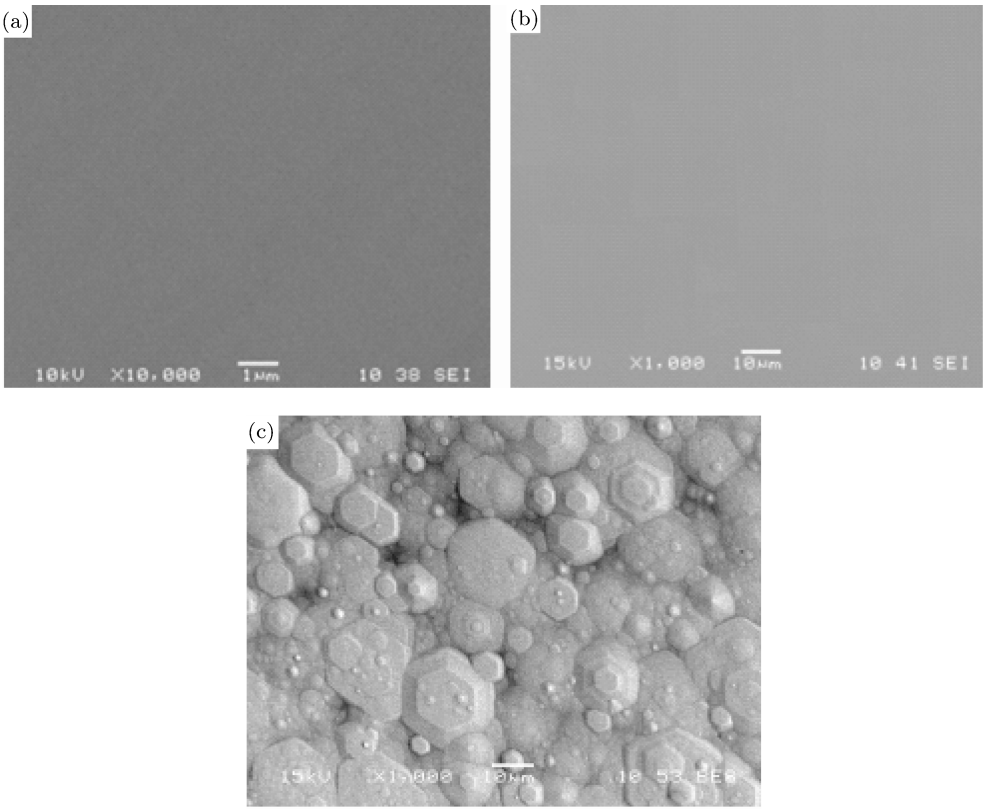


Fig. 1. The SEM images of as grown GaN (a) nonpolar a-plane GaN, (b) Ga-face GaN, and (c) N-face GaN.

All of the etching results are shown in Fig. 2. There are many micron- and nano-meter columns appearing on the a-plane GaN surface. The columns have different diameters, but all along the c-axis direction. The phenomenon is very interesting and shows a significant difference with that of chemical etching of the c-plane GaN. We have found that the TD terminates at the V-defects of the Ga-face GaN, and the α-type etching pit shows an inversed trapezoidal shape, the β one has a triangular shape, and the Υ-type one has a combination of triangular and trapezoidal shapes. The different types of etching pits originate from screw, edge, and mixed-

type TDs, respectively.[10] At the same time the N-face GaN has been etched into the substrate, leaving only a few grains. The different etching morphologies indicate the different etching models.

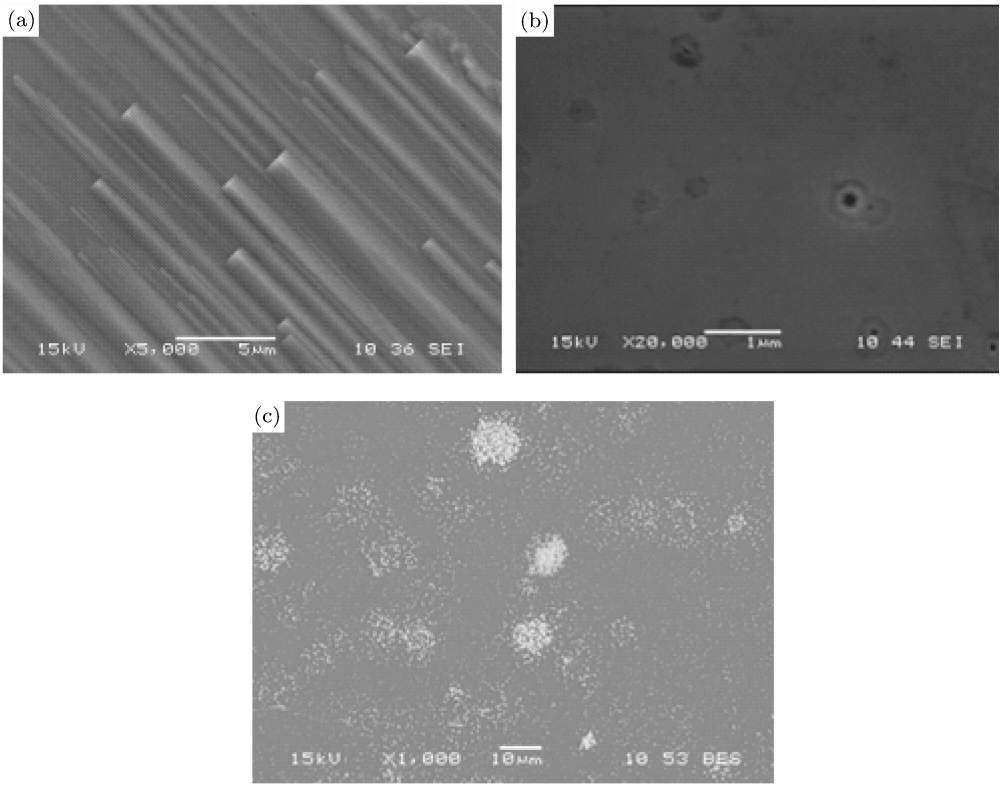


Fig. 2. The SEM images of GaN made by wet etching for 30 min: (a) nonpolar a-plane GaN, (b) Ga-face GaN, and (c) N-face GaN.

In order to interpret the origin of etching morphology, an etching mechanism model is proposed. For the a-plane GaN, there are both Ga and N atoms at the step edge in the [0001] direction, whereas there is either Ga or N atoms at the step edge in the $[1\bar{1}00]$ direction. The OH^- ions can attack the bonds of the Ga atoms and be adsorbed on the surface as shown in Fig. 3. The OH^- ion reacts with GaN, forming gallium oxide and NH_3, and the gallium oxide can be dissolved in alkali solution. Reaction equation is given as follows:[11]

$$2\mathrm{GaN} + 3\mathrm{H_2O} \xrightarrow{\mathrm{KOH}} \mathrm{Ga_2O_3} + 2\mathrm{NH_3}.$$

KOH is playing a role as a catalyst. The adsorbed and solved processes are shown in Figs. 3(b), 3(c) and 3(d). Furthermore, the OH^- ion attacks the Ga atoms of the next atom layer. The process is shown in Figs. 3(e) and 3(f), and then the surface structure of the a-plane GaN would be converted into Fig. 3(g). This surface structure is the same dangling bond configuration of nitrogen as that in Fig. 3(d). The etching of the a-plane GaN film is continued through repeating stages (d)–(g) in Fig. 3. However, the speed of the etching along the $[11\bar{2}0]$ direction is not the most dominant in the a-plane etching model. For MOCVD grown a-plane $(11\bar{2}0)$ GaN on r-plane $(1\bar{1}02)$ sapphire, at the beginning of growth, many large islands with discrete side facets are produced. As these islands continue to grow and coalesce, coalescence is incomplete and is a consequence of the $(000\bar{1})$ plane being of a different crystallographic family from the $(10\bar{1}1)$ and $(011\bar{1})$ planes.[12,13] The a-plane GaN typically has a threading dislocation density of 3×10^{10} cm^{-2} and a basal stacking fault (BSF) density of 3.5×10^5 cm^{-1}.[14–16] Johnston *et al.* proposed that the partial dislocations bound BSFs, which is necessarily confined to the N-face polar plane.[17] The N-face polar in a-plane GaN plays a very important role in the etching process. The chemical etching is performed not only along the a axis but also along the $-c$ axis. The chemical etching would be carrying out alone the N-face easily. There is a single dangling bond of nitrogen atom upward on the N-face surface. The chemical etching process of the N-face polar GaN

is shown in Fig. 4. The etching principle of the N-face is similar to the a-plane; the difference is that there

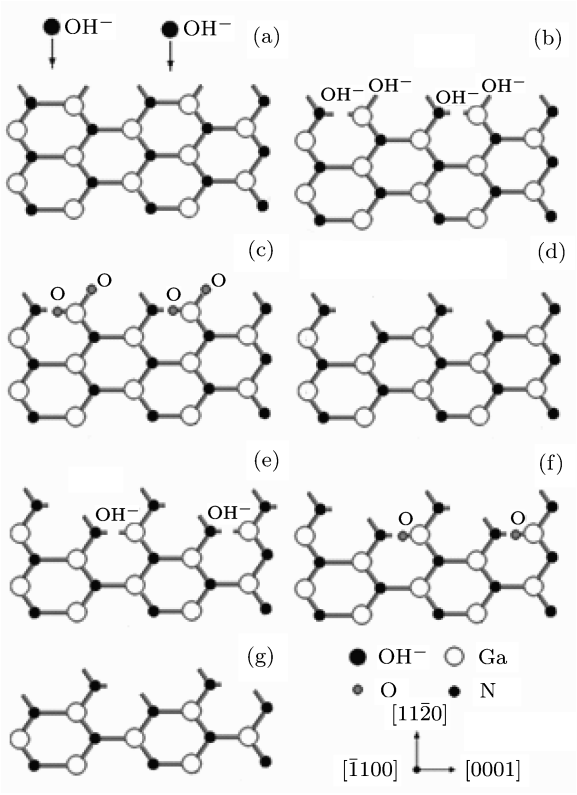


Fig. 3. Schematic diagrams of the a-plane GaN film viewed along the [1$\bar{1}$00] direction to explain the mechanism of the selective etching.

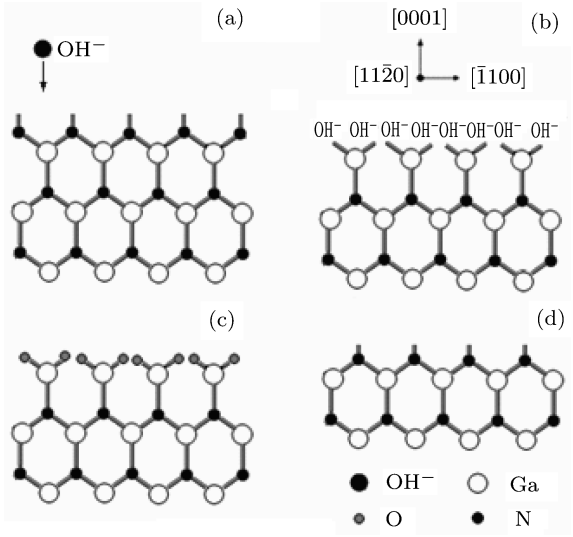


Fig. 4. Schematic diagrams of the cross sectional N-surface GaN film viewed along the [11$\bar{2}$0] direction to explain the mechanism of the selective etching.

are two dangling bonds of nitrogen atom upward on the nonpolar a plane surface. Therefore, the N-face etching rate will be certainly greater than the nonpolar a plane. So the N-face GaN has been etched into the substrate as shown in Fig. 2(c). The specific etching process of the N-face can be seen in Ref. [11].

Etching can be carried out along the dislocation in the Ga-face GaN, but the speed along the Ga-terminated is very slow. The different chemical etching results should result from the different arrangements of atoms. Since the hydroxide ions in KOH solution cannot attack the Ga-terminated surface because of large repulsion between OH^- and three occupied dangling bonds of nitrogen. The schematic diagram of the Ga-terminated GaN film viewed along the [0001] direction is shown in Fig. 5. Therefore, the speed of the a-plane GaN is between the speed of N-plane GaN and that of Ga-plane GaN.

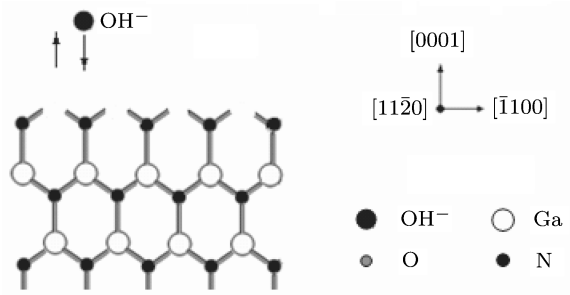


Fig. 5. Schematic diagrams of the Ga-face GaN film viewed along the [11$\bar{2}$0] direction to explain the mechanism of the selective etching.

4. Conclusions

In summary, morphology and microstructure of the a-plane GaN epilayers etched by KOH solution have been investigated by SEM. There are many micron- and nano-meter columns appearing on the a-plane GaN surface. An etching mechanism model associated with the BSF is proposed to interpret the origin of the distinctive etching morphology. The micron- and nano-meter columns are associated with the BSF of the a-plane GaN. Hydroxide ions in KOH solution is difficult to attack the Ga-face GaN because of large repulsion between OH^- and three occupied dangling bonds of nitrogen. The dangling bond configuration of nitrogen on the surface plays an important role in the selective etching. The etching speed of the a-plane GaN is greater than that of the Ga-face but less than the N-face GaN.

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