# Impact of selective thermal etching in mixed H<sub>2</sub>/NH<sub>3</sub> atmosphere on crystal quality of N-polar AlGaN/GaN heterostructures

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# Abstract

We investigated thermal selective etching of N-polar GaN/Al<sub>0.27</sub>Ga<sub>0.73</sub>N/GaN double heterostructures in mixed  $H_2/NH_3$  atmosphere. We monitored etching behaviors using an in-situ surface reflectance measurement system, which enabled us to estimate etching rates and detect etching endpoints. The etching rate of N-polar GaN was lower than that of Ga-polar GaN, while the activation energy for etching was almost the same for both. The AlGaN layer inserted between the GaN layers acted as a selective etching stopper and exhibited smooth etched surface similar to that of an as-grown surface.

## 1. Introduction

High-power and high-frequency GaN HEMTs are promising devices for amplifiers for the next generation of millimeter-wave wireless communications systems. In particular, N-polar GaN HEMTs have attracted a great deal of attention for applications at higher frequencies because of their good tolerance against short channel effects [1]. In the several reports on the epitaxial growth of N-polar GaN HEMTs [2], high-quality material growth was limited to specific substrates with large off-cut angles. In addition, N-polar nitrides are known for their high incorporation of residual impurities.

To avoid these growth issues, we previously proposed a fabrication method for N-polar GaN that uses an epitaxiallayer transfer technique [3]. N-polar layers are formed by transferring Ga-polar epitaxial layers upside down to a host substrate and subsequently removing the growth substrate [4]. To use this method for actual device fabrications, N-polar GaN buffer layers with a thickness of several micrometers must be etched to expose the buried device layer, whose thickness is on the order of tens of nanometers. For this precise etching depth control, a highly selective etching technique is required. One possible approach is to use the selective thermal etching method reported by Arita et al [5], which provides high selectivity of around 10<sup>3</sup> and has achieved selective etching of Ga-polar GaN and successful exposure of nm-order ultra-fine structures. However, as far as we know, N-polar GaN etching by this method has not been reported yet.

In this paper, we report selective thermal etching of N-polar GaN/AlGaN/GaN double heterostructures in a mixed  $H_2/NH_3$  atmosphere and its impact on material qualities.

#### 2. Experiments

All the N-polar samples were grown by metal-organic

chemical vapor deposition (MOCVD) on 3-inch sapphire substrates with the miscut of 4° towards the a-plane. At the initial stage of growth, the substrate surface was nitrided for polarity control. The use of substrates with a large miscut enabled us to obtain smooth surfaces without hexagonal hillocks. We used N-polar GaN single-layer samples with the thickness of around 1  $\mu$ m to investigate etching characteristics. We grew N-polar double heterostructures consisting of 78-nm GaN/9-nm Al<sub>0.27</sub>Ga<sub>0.73</sub>N/1- $\mu$ m GaN to examine the etch-stopper behavior of Al<sub>0.27</sub>Ga<sub>0.73</sub>N. An N-polar 9-nm Al<sub>0.27</sub>Ga<sub>0.73</sub>N/1- $\mu$ m GaN heterostructure was also grown as a reference.

MOCVD growth and etching experiments were performed in the same reactor. After epitaxial growth, sample wafers were once removed from the reactor and exposed to the atmosphere. After wet chemical surface treatments and reloading of samples into the reactor, thermal etching was performed in a mixed  $H_2/NH_3$  atmosphere at 100 Torr. The etching temperatures were between 1080 and 1210°C. The etching was monitored in real time with an in-situ optical growth monitoring system. Sample qualities were examined by X-ray diffraction (XRD) and atomic force microscopy (AFM).

#### 3. Results and Discussion

Figure 1 shows the temperature dependence of the N-polar and Ga-polar [6] GaN etching rate. The H<sub>2</sub>/NH<sub>3</sub> supply



Fig. 1. Temperature dependence of etching rate of Gaand N-polar GaN. The inset shows typical time dependence of the signal of the in-situ monitoring system.



Fig. 2. Time dependence of reflected light intensity monitored by in-situ monitoring system. The etching was performed at 1160°C.

ratio was 74. The etching rate was estimated from the oscillation periods of light intensity reflected from samples, such as shown in the inset of Fig. 1. The activation energy estimated from the data in Fig. 1 was 234 kJ/mol for N-polar GaN etching, which was similar to that of Ga-polar GaN of 251 kJ/mol. These values are close to the calculated activation energies reported in the literature [7]. These results indicate that the etching process for N-polar GaN is governed by a reaction energy barrier similar to that in Ga-polar GaN. The difference in etching rates is probably due to the bonding arrangements on the GaN surface. In our experiments, the etching rate was limited by nitrogen atom desorption as NH<sub>3</sub>. Owing to the three back bonds of the topmost N atoms, nitrogen desorption is more suppressed on the N-polar surface than on the Gapolar surface, where N atoms have one back bond. Considering the results shown in Fig. 1, we choose the etching temperature at 1160°C for further experiments because the etching rate of around 5 nm/min is good enough to monitor the etching process.

Figure 2 shows an example of the time-dependence of reflectance intensity from an N-polar GaN/AlGaN/GaN double heterostructure during the etching process. The signal oscillation from 0 to around 650 s indicates the etching of the top GaN layer. From 650 s, the signal intensity becomes almost constant. This means that the etching stopped at the top of the AlGaN layer. The duration of constant intensity was defined as the AlGaN over-etching time ( $t_{OE}$ ). We examined the properties of samples treated with the  $t_{OE}$  of 500 s.

Figure 3 shows omega-2-theta XRD profiles of the GaN/AlGaN/GaN double heterostructures before and after etching and that of the as-grown AlGaN/GaN reference sample. In the higher angle region, the fringes due to the top GaN layer appeared before etching. They disappeared after the  $t_{OE}$  of 500 s. The profile of the etched sample is almost the same as that of the as-grown reference. This result indicates that the AlGaN layer acts as an etching stopper and remains after the  $t_{OE}$  of 500 s. The results are consistent with the reflectance monitoring results in Fig. 2.

Figure 4 shows an AFM image of the AlGaN surface after the over-etching and an image of the reference. On both samples, there are small surface pits originating from the N-polar



Fig. 3. XRD profiles of samples before and after etching and reference.



Fig. 4. AFM images of the AlGaN layer surface (a) after etch-stop ( $t_{OE} = 500$  s) and (b) reference.

growth process. The average surface roughnesses are 0.22 and 0.21 nm, respectively. The sample surfaces are atomically flat on average. This result suggests that etching depth is uniformly controlled by the thermal selective etching. In Fig. 4(a), the etched surface exhibits small bumps with a height of several nanometers. Further optimization is required to reduce these defects.

## 4. Conclusions

We examined the feasibility of thermal selective etching of N-polar GaN/Al<sub>0.27</sub>Ga<sub>0.73</sub>N heterostructure in a mixed  $H_2/NH_3$  atmosphere. The temperature dependence of the etching rate of GaN was investigated. The etching rate of N-polar GaN was lower than that of Ga-polar GaN, while the activation energy was almost the same. It was found that the AlGaN layer effectively acted as selective etching stopper. The Al-GaN etched surface was as smooth as that of the as-grown AlGaN surface. These results suggest the feasibility of using the presented method for practical microdevice fabrication.

## References

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