

# Fabrication of GaN high-aspect fine nano-hole array structures by hydrogen atmosphere anisotropic thermal etching with ammonia gas

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## Abstract

We developed a high-aspect GaN nano-hole fabrication technique based on low-pressure hydrogen assisted thermal decomposition of GaN with SiO<sub>2</sub> selective etching mask. It was confirmed that cross-sectional etching profile was strongly dependent on the NH<sub>3</sub> concentration in H<sub>2</sub> and the etching temperature. As the NH<sub>3</sub> was increased from 5% to 15%, the incline angle of the etched side facets approached 90°. In the case of 10% NH<sub>3</sub> addition, as the temperature increased from 875 to 975 °C, the etch rate increased from 5 to 60 nm/min and the side facets becoming more vertical. The optimized NH<sub>3</sub> addition HEATE conditions were used to demonstrate the fabrication of 100 nm pitch triangular lattice high-aspect fine GaN nano-hole array with a diameter of 55 nm and a depth of 900 nm. This etching technique is expected to be applicable to GaN fine nanostructures such as GaN-based photonic crystal in visible wavelength region.

## 1. Introduction

GaN based III-nitride materials has excellent light emission characteristics in the green to near-UV region, and their high-brightness LEDs and laser diodes has been widely commercialized. The introduction of photonic crystal (PhC) structure is attractive for further improvement and functionalization of these optical devices. Many researches regarding to GaN PhC, e.g., emission direction control, vertical cavity laser [1], emission rate control by high Q cavity [2] has been reported. On the other hand, the PhC structure in the visible region requires ultra-fine fabrication technology of 100 nm or less, and although dry etching is mainly used, etching damage and shape controllability are strongly concerned.

Recently, we have proposed a low-damage dry etching technique of hydrogen environment anisotropic thermal etching (HEATE) which based on hydrogen assisted thermal decomposition of GaN using thin SiO<sub>2</sub> for selective etching mask [3, 4]. This HEATE technique could fabricate ultrafine GaN/InGaN/GaN nanopillars and nanowalls with almost without etching damage. However, the stable etched facets of HEATE tend to be {20 21} facets which are 15° tilted plane from m-plane. Therefore, it was difficult to form high-aspect nano-hole structure commonly used for PhCs.

In this study, we investigated the etching conditions of GaN by HEATE with changing NH<sub>3</sub> addition concentration and temperature, consequently succeeded a fabrication of fine high-aspect GaN nano-hole array suitable for PhC application.

## 2. Experiments

A Si-doped (0001) GaN epitaxial wafer grown on (0001) sapphire substrate by metal-organic chemical deposition was used as a starting material. After atomic deposition of a 15-nm-thick SiO<sub>2</sub> layer on the wafer, Cr nano-patterns were formed by a lift-off process using electron-beam lithography and ion-beam sputtering. The Cr nano-patterns were transferred to SiO<sub>2</sub> via ion-beam etching using CF<sub>4</sub> and O<sub>2</sub> gases and then Cr mask was removed by wet etching. Two types of SiO<sub>2</sub> nano-masks, one is stripe windows with various width and another is triangular nano-holes with various diameter and periods were prepared. The samples were loaded in a tubular furnace with a quartz reactor, and then heated at the temperature (T) of 875 or 975 with duration (t) of 30 or 100 min. The total pressure of H<sub>2</sub> and NH<sub>3</sub> was maintained at 100 Pa and NH<sub>3</sub> concentration (N) was varied to be 5, 10, or 15%. After the HEATE process, the cross sectional shape of nano-trenches and nano-holes were observed by scanning electron microscope (SEM).

## 3. Results and Discussions

Fig.1(a)-(c) show typical cross sectional SEM images of nano-trenches fabricated by HEATE using stripe masks. The HEATE conditions were T=875 °C, N=15 %, t=100 min for (a), T=975 °C, N=15 %, t=30 min for (b), and T=975 °C, N=15 %, t=30 min for (c). It is worth noting that cross sectional shape of nano-trenches very different. For the case of (a), about 45° slanted stable facets were formed and deep undercut were observed under the SiO<sub>2</sub> masks. It can be considered that etch rate of depth direction was relatively slower

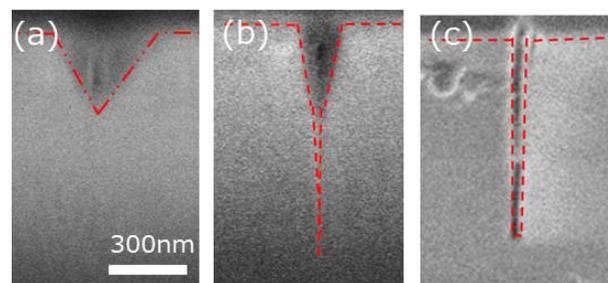


Fig. 1. Cross sectional SEM images of nano-trenches fabricated by HEATE with conditions of (a): T=875 °C, N=15 %, t=100 min, (b): T=975 °C, N=15 %, t=30 min, and (c): T=975 °C, N=15 %, t=30 min.

than that of normal direction toward side facets. For the case of (b), which etched at higher temperature and shorter duration than (a), two side facets with different incline angles were formed. Both side incline angles were steeper than that of (a), suggesting that higher the temperature higher the etching rate toward depth direction. For the case of (c), which is higher  $\text{NH}_3$  concentration than (b). The side facets were almost normal to the  $c$ -axis and indicating that  $\text{NH}_3$  tend to prevents side etching. Fig. 2 shows etching rate of  $c$ -axes direction of nano-trenched fabricated by stripe masks with different opening width of 140 and 80 nm at the  $\text{NH}_3$  concentration of 5%. The etching rates at 875°C were 2-7nm/min and increased about 10 times to 52-65 nm/min at 975 °C. It was also note that etching rate tend to higher for wider trenches. Fig.3 shows etching rate of  $c$ -axes as a function of  $\text{NH}_3$  concentration. The etching rate was decreased with increasing  $\text{NH}_3$  concentration from 5 to 10 %, but almost saturated at 15 %. For the case of  $N=15\%$ , high density of residual fine nanopillar structures with 20-30 nm diameters were formed in exposed area.

Fig. 4 shows top- and bird's eye-view SEM images of GaN nano-hole arrays fabricated at a HEATE conditions of  $T=975$  °C,  $N=10$  %,  $t=30$  min. Uniform and high-aspect ver-

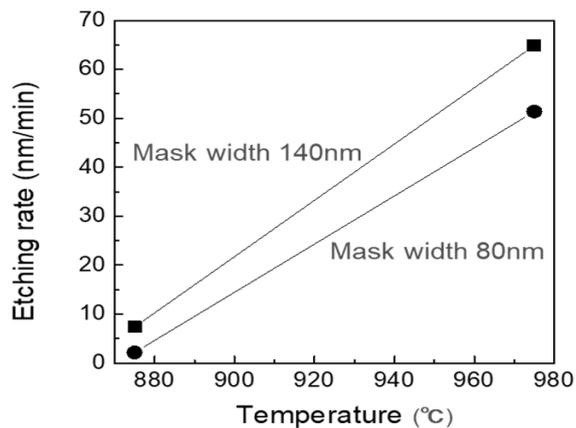


Fig. 2. Depth etching rate of GaN nano-trenches for different temperatures of 875 and 980 °C. The  $\text{NH}_3$  concentration was 5%.

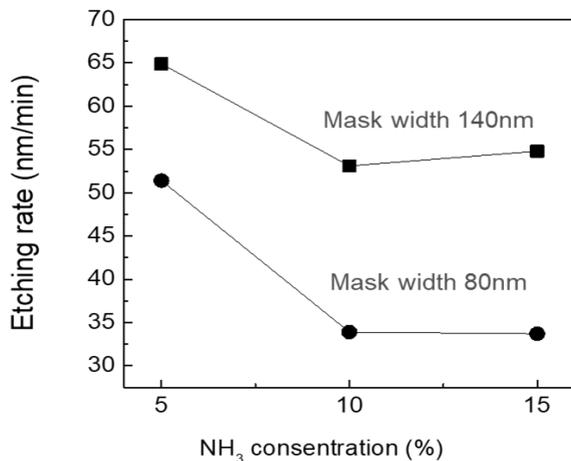


Fig. 3. Depth etching rate of GaN nano-trenches as a function of  $\text{NH}_3$  concentration. The etching temperature was 975 °C.

tical holes with diameters of 55, 72, and 91 nm were successfully fabricated. Fig.5 shows nano-hole depth dependency on the hole diameter. The depth tends to reduce with reducing the hole diameter, but the fluctuation was as small as 5%.

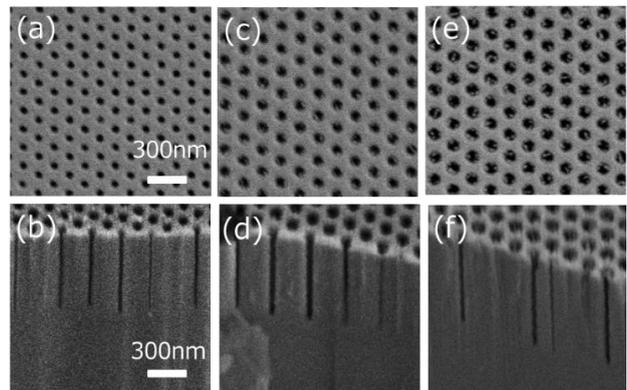


Fig. 4. Top- and bird's eye view SEM images of GaN nano-hole array fabricated by HEATE with  $\text{NH}_3$  addition. The hole diameters of (a, b), (c, d), (e, f) are 55, 72, and 91 nm.

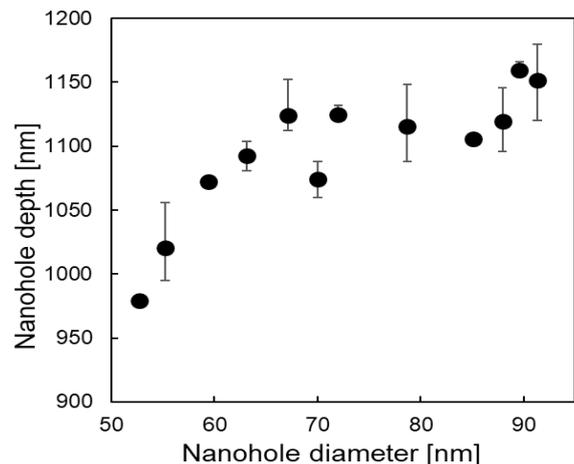


Fig. 4. Nano-hole depth as a function of hole diameter.

#### 4. Conclusions

Etching characteristics of GaN by HEATE was investigated with changing the  $\text{NH}_3$  addition concentration and temperature. It was confirmed that the cross sectional shape of nano-hole strongly dependent on the etching condition. A high-aspect small nano-hole array with hole diameter of 55 nm and depth of 900 nm could be fabricated at a HEATE condition of 100 Pa in total pressure at 975 °C with 10%  $\text{NH}_3$  addition.

#### Acknowledgements

This work was partially supported by JSPS KAKENHI Grant Numbers 16K14260, 17L02747, and JST CREST Grant Number JPMJCR18T4, Japan.

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