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Preparation of extended microtunnels in GaN by wet chemical etching

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Extended microtunnels with triangular cross sections are demonstrated in GaN layers on sapphire substrates. The depths of the tunnels can easily reach several hundred micrometers by using wet chemical etching. To obtain this result, patterned growth of specially designed GaN layers is carried out on sapphire substrates with metalorganic chemical vapor deposition and subsequently hydride vapor-phase epitaxy techniques. The prepared samples are then chemically etched in molten potassium hydroxide, and microtunnels with triangularly etched cross sections are formed. The planes of the triangular bevels belong to the $\{11\bar{2}2\}$ family. The etch rate of the tunnel can be as high as 10 μm/min under proper etching conditions.

1 Introduction

Group III nitride semiconductors have stimulated great research interest in the last decade due to their unique properties and potential applications in short-wavelength optoelectronic devices as well as in high-temperature and high-frequency devices. However, the unusual chemical stability of GaN has also made it difficult for wet chemical etching that is often necessary for device fabrication [1]. Several dry etching methods, such as: inductively coupled plasma (ICP) etch, reactive ion etch (RIE), and chemically assisted ion beam etch (CAIBE), have been developed for effective etching of GaN [2, 3]. But dry etching techniques often cause ion-bombardment damage and usually have difficulties in obtaining smoothly etched side walls. Wet chemical etching can provide the advantages of low damage, low cost, and anisotropic etch rates of different crystallographic planes [4]. It is therefore an important complement to dry etching techniques. However, to date, feasible wet chemical etch rates of GaN are still slow. Even with the assistance of ultraviolet light, as those applied in the photoelectrochemical (PEC) etching techniques demonstrated by many research groups, the fastest achievable etch rate is still limited to several tens of nanometers per minute [5]. In the present study, we have develop a new wet etching technique to obtain extended microtunnels (EMTs) in GaN with high etch rates in molten KOH.

2 Experimental

All the samples used in this study consist of a roughly 90 μm-thick GaN layer grown on sapphire substrates. Conventional metal-organic chemical vapor deposition (MOCVD) was first used to grow a 4 μm-thick GaN template on a (0001) *c*-plane sapphire substrate, followed by the deposition of a 300 nm SiO_2 layer prepared by plasma-enhanced chemical vapor deposition (PECVD). The SiO_2 layer is then patterned,

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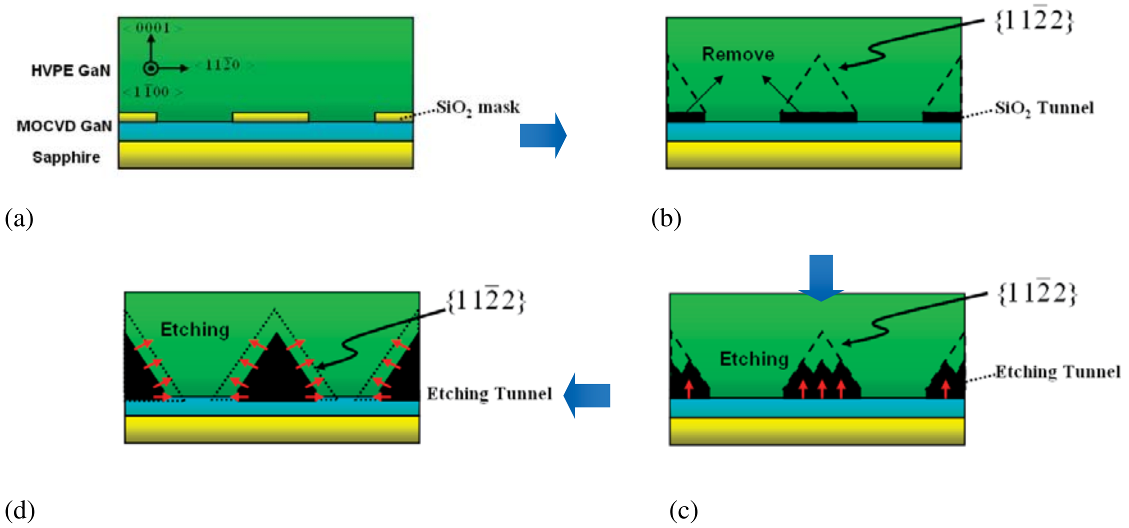

Fig. 1 (online colour at: www.pss-b.com) Schematic of GaN EMT formation process: (a) ELOG structure of HVPE GaN, (b) etching of SiO$_2$ mask, (c) formation of GaN EMTs, (d) lateral etching of GaN along the $\langle 11\bar{2}2\rangle$ direction.

using a standard photolithographic technique and BOE etch, into 5 μm wide strips along the $\langle 1\bar{1}00\rangle$ direction of GaN with 5 μm windows, which expose the underlying MOCVD GaN layers, in between the strips. On top of this patterned MOCVD GaN template, hydride vapor-phase epitaxy (HVPE) is adopted to perform an epitaxial lateral overgrowth (ELOG) of a 85 μm-thick GaN film. Figure 1a illustrates the cross section of the completed structure. The sample is then sliced into several smaller pieces for wet chemical etching. While most of the samples are etched in molten potassium hydroxide (KOH) at temperatures from 170 to 250 °C, some samples are etched in BOE for comparison.

3 Results and discussion

3.1 Formation of extended microtunnels

Extended microtunnels (EMTs) with triangular cross sections are observed when samples are etched in molten KOH at high temperature. The formation process of EMTs is illustrated in Fig. 1. The process consists of several steps. First, the SiO$_2$ mask is removed by molten KOH, as shown in Fig. 1b. Secondly, GaN right above the SiO$_2$ mask starts to be etched away, as in Fig. 1c. Then, tunnels with well-shaped triangular cross sections were formed with the $\{11\bar{2}2\}$ family of planes as the etch-stop planes, as illustrated in Fig. 1d. In the axial direction of EMTs, it is believed that high etch rates are obtained mainly with the assistance of the fast etch rates of SiO$_2$ and the GaN on its top. Figure 2 is an optical microscopic (OM) image of an etched sample from its top. The depths of the tunnels have extended beyond 300 μm, which corresponds to an average tunnel etch rate of more than 10 μm/min. Even with such extended depths of EMTs, adjacent tunnels remain separated. This indicates that while the etch rates along the tunnel's axial direction are exceedingly fast, the etch rates along the tunnel's sidewalls are very slow.

3.2 Reaction mechanism of GaN EMTs

Figure 3a and b are cross-sectional SEM images of GaN EMTs after being etched in molten KOH at 230 °C for 30 min. Both images show very uniformly repeated EMTs. Such repeated EMTs are observed in different positions of the sample, which indicates that the etching reactions are very uniform across the whole sample. For comparison, Fig. 3c shows the cross-sectional SEM image of a sample after being

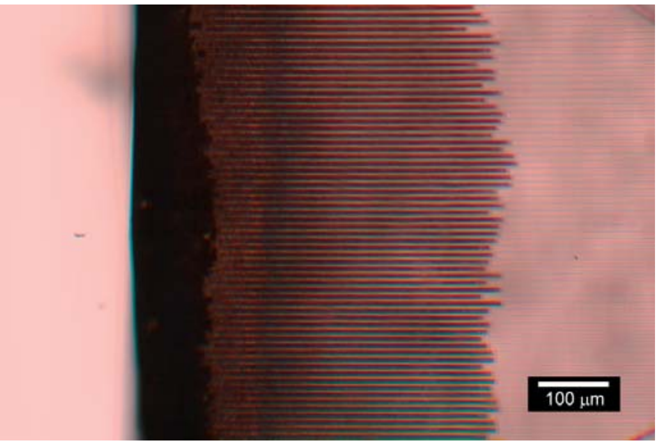

Fig. 2 (online colour at: www.pss-b.com) Optical microscopic image of GaN EMTs after 30 min etch by KOH at 250 °C.

etched in BOE for 18 h at room temperature. No well-shaped triangular tunnels are observed in the samples etched by BOE. For BOE etched samples, the average etch rate of the tunnel along the axial direction is found to be about 0.25 µm/min. It is very slow compared to the etch rate of the tunnels in molten KOH. Energy dispersive X-ray (EDX) measurements have been applied to the tunnels' cross-sectional regions of these samples and no traces of Si or oxygen were found, which implies that the SiO_2 mask has been removed by both molten KOH and BOE. Blohowiak et al. [6] have proposed a possible reaction mechanism between SiO_2 and KOH. After SiO_2 is removed, molten KOH starts to etch the N-polar GaN right above the SiO_2 mask region. A number of studies have shown that the N-polar GaN can be etched

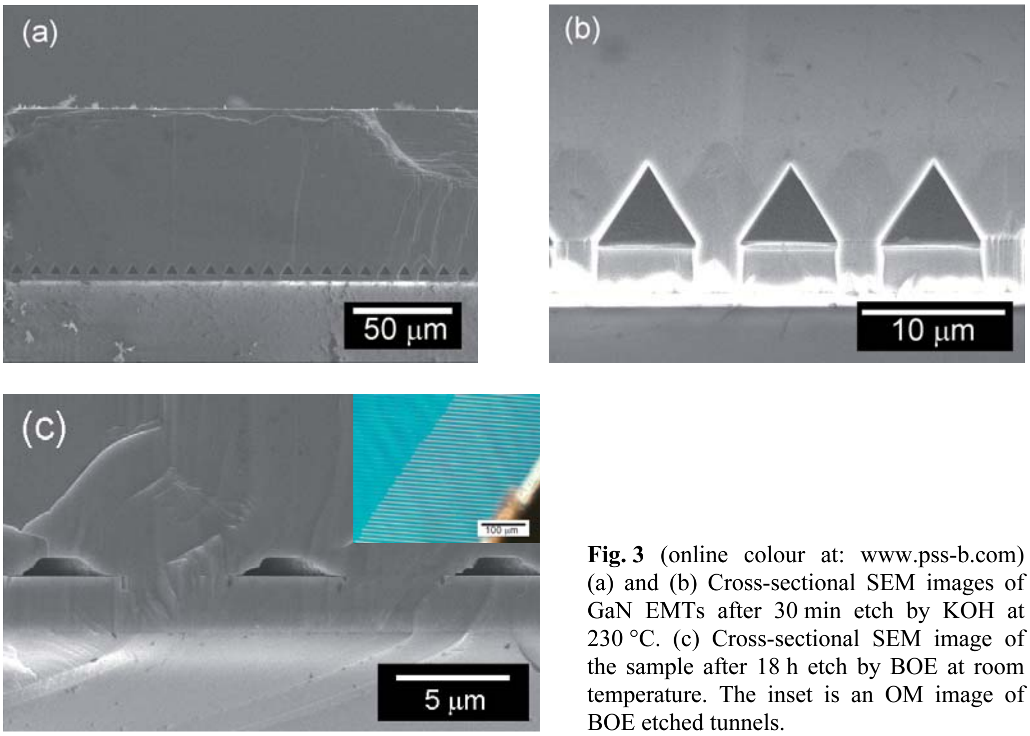

Fig. 3 (online colour at: www.pss-b.com) (a) and (b) Cross-sectional SEM images of GaN EMTs after 30 min etch by KOH at 230 °C. (c) Cross-sectional SEM image of the sample after 18 h etch by BOE at room temperature. The inset is an OM image of BOE etched tunnels.

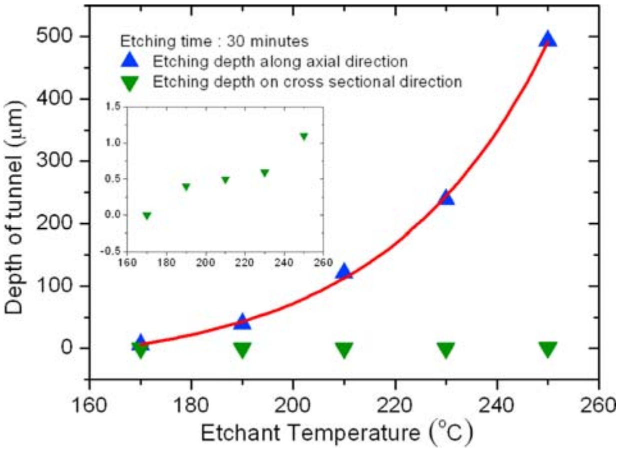

Fig. 4 (online colour at: www.pss-b.com) Etch depth of GaN EMTs along the axial direction and etching distance of the bevel planes in the cross-sectional direction at different etchant temperatures. The inset magnifies the etching distance in the cross-sectional direction.

more readily than the Ga-polar GaN [7–9]. Li et al. have interpreted the mechanism of such polarity-selective etching [10, 11]. It is suggested that hydroxide ions (OH^-) are first adsorbed onto the N-polar GaN surface to form Ga_2O_3, which can then dissolve in KOH solvent. In the formation process of GaN EMTs in the present study, the etch rate becomes very slow when the etchant reaches the $\{11\bar{2}2\}$ family planes. Figure 4 compares the etch depths of the tunnels along the axial direction and the extent of the etching distance of the $\{11\bar{2}2\}$ planes in the cross-sectional direction in a series of isochronal etching experiments. The average etch rate of the tunnel in the axial direction determined from this figure is larger than 10 μm/min, while the etch rate of the planes is only 0.5 μm/min.

3.3 Separation of GaN from sapphire

Figure 5 shows the cross-sectional SEM image of GaN EMTs after a 30 min etch in molten KOH at 250 °C. The GaN EMTs have coalesced and hence caused separation of the GaN layer from the sapphire substrate. The bottom of the N-polar GaN forms hexagonal pyramids that have been observed by other researchers in wet chemical etching of N-polar GaN [8].

4 Conclusions

A novel GaN wet chemical etching technique is developed by using molten KOH and specially designed structures of GaN layers. It is shown that well-shaped triangular microtunnels extended hundreds of micrometers can be easily obtained. The bevels of the tunnels' triangular cross section belong to the

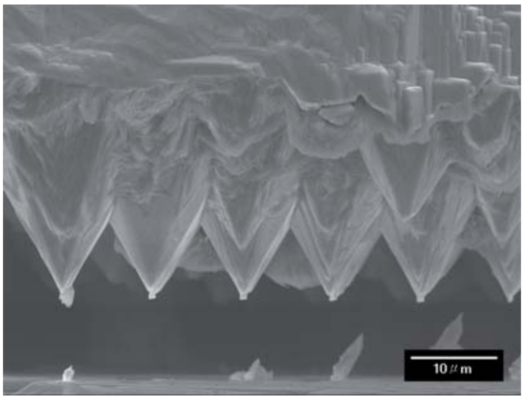

Fig. 5 Separation of GaN from sapphire after 30 min etch by molten KOH at 250 °C.

$\{11\bar{2}2\}$ family of planes of GaN. The etch rate of the tunnels in the axial direction can be more than 10 μm/min.

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References

[1] D. Zhuang and J. H. Edgar, Mater. Sci. Eng. R **48**, 1 (2005).
[2] C. Y. Fang, W. J. Huang, and E. Y. Chang, Jpn. J. Appl. Phys. **42**, 4207 (2003).
[3] S. Tripathy and S. J. Chua, J. Appl. Phys. **91**, 3398 (2001).
[4] D. A. Stocker and E. F. Schubert, Appl. Phys. Lett. **73**, 2654 (1998).
[5] L. H. Peng and C. W. Chuang, Appl. Phys. Lett. **72**, 939 (1997).
[6] K. Y. Blohowiak, D. R. Treadwell, and B. L. Muller, Chem. Mater. **6**, 2177 (1994).
[7] T. Palacios, F. Calle, and E. Munoz, Semicond. Sci. Technol. **15**, 996 (2000).
[8] H. M. Ng, N. G. Weimann, and A. Chowdhury, J. Appl. Phys. **94**, 650 (2003).
[9] M. Shimizu, A. Suzuki, and K. Onabe, in: International Symposium on Blue Laser and Light Emitting Diodes II, Chiba, Japan, September 29–October 2, 1998, p. 723.
[10] D. Li, M. Sumiya, and S. Fuke, phys. stat. sol. (a) **180**, 357 (2000).
[11] D. Li, M. Sumiya, and Y. Fukuda, J. Appl. Phys. **90**, 4219 (2001).