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Electrochemical liftoff of freestanding GaN by a thick highly conductive sacrificial layer grown by HVPE*

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Separation technology is an indispensable step in the preparation of freestanding GaN substrate. In this paper, a large-area freestanding GaN layer was separated from the substrate by an electrochemical liftoff process on a sandwich structure composed of an Fe-doped GaN substrate, a highly conductive Si-doped sacrificial layer and a top Fe-doped layer grown by hydride vapor phase epitaxy (HVPE). The large difference between the resistivity in the Si-doped layer and Fe-doped layer resulted in a sharp interface between the etched and unetched layer. It was found that the etching rate increased linearly with the applied voltage, while it continuously decreased with the electrochemical etching process as a result of the mass transport limitation. Flaky GaN pieces and nitrogen gas generated from the sacrificial layer by electrochemical etching were recognized as the main factors responsible for the blocking of the etching channel. Hence, a thick Si-doped layer grown by HVPE was used as the sacrificial layer to alleviate this problem. Moreover, high temperature and ultrasonic oscillation were also found to increase the etching rate. Based on the results above, we succeeded in the liftoff of $\sim$ 1.5 inch GaN layer. This work could help reduce the cost of freestanding GaN substrate and identifies a new way for mass production.

Keywords: electrochemical etching, liftoff, hydride vapor phase epitaxy (HVPE), freestanding GaN

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1. Introduction

GaN has the advantages of high electron mobility, wide band gap, strong critical breakdown field and stable physical and chemical properties,[1] which are beneficial for light-emitting diode (LED), field-effect transistor (FET), Schottky barrier diode (SBD) and other power electronic devices.[2–7] However, due to the lack of native substrates, group III nitrides are generally grown on foreign substrates, such as sapphire, silicon and SiC, which results in the large lattice mismatch and thermal expansion mismatch between the substrate and epilayer. The dislocation density in a device on foreign substrate is much higher than that grown on native substrate.[8,9] Hence, freestanding GaN substrate is an effective way to improve the quality of the epitaxial layer and performance of the device.[10]

Hydride vapor phase epitaxy (HVPE) has been considered as one of the promising candidates for the commercial fabrication of freestanding GaN substrates, due to its high growth rate and large wafer size.[11] However, the cost of freestanding GaN substrate grown by this method is still high. Moreover, in the process of preparing freestanding GaN substrates, separation, rounding and polishing are indispensable steps, and the separation cost is particularly high. All of the above limit its large-scale commercial application.

To date, the laser lift-off (LLO) method has been utilized to prepare freestanding GaN substrates.[12,13] The LLO method uses a high-power UV laser to decompose the GaN at the interface between sapphire and GaN to realize the liftoff of the substrate and epilayer. However, the cost of this method is relatively high and the heat produced by the laser may cause the GaN to crack.[14] In addition, it is not compatible with epitaxial layers grown on SiC or bulk GaN crystals because of the similar band gap. Photoelectrochemical etching is also used as a stripping method, which requires illumination by intense UV light. There are several reports on the GaN etching without UV illumination, but they were performed on GaN with different polarities. However, these techniques are not appropriate for chemical liftoff because of their low selectivity, low etch speed and the necessity for light illumination. Moreover, such lateral etching usually exposes N-polar GaN, which is easily etched by many of the wet etching solutions.[15,16]

Electrochemical liftoff is a technique that utilizes lateral undercut etching and can be used to separate the GaN layers from the substrates regardless of the kind of substrate.[17–19]

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Many research groups have studied electrochemical techniques to prepare GaN substrates. Joonmo Park *et al.* prepared 20×20 μm freestanding GaN substrate by electrochemical etching sacrificial layer completely.[14] However the smallest size does not have much practical value. In order to obtain a large-size substrate, recently, Qin Hu *et al.* successfully separated 2 inch GaN substrate from sapphire substrate by introducing holes into 2 inch GaN sacrificial layer using electrochemical and chemical methods.[20] During cooling, the substrate is separated by mismatch stress. Jin-Ho Kang *et al.*[21] and Zeng Yin Dong *et al.*[22] used a similar method to get a large freestanding GaN substrate. However, by introducing large holes, the method of releasing stress is only suitable for heterogeneous substrates. Also, the release of stress easily leads to the cracking of freestanding GaN substrate.[23] In general, the size of the substrate prepared by complete etching is small, because the thin sacrificial layer hinders mass transportation. While the introduction of holes for large-size liftoff is only suitable for heterogeneous substrates and results in the cracking of GaN during cooling.

In this article, based on the rapid growth characteristics of HVPE, we propose to thicken the sacrificial layer to alleviate the mass transportation problem and combine temperature, oscillation and high voltage to promote electrochemical etching. Finally, we realized the electrochemical liftoff of ~ 1.5 inch freestanding GaN by etching a thick highly conductive sacrificial layer grown by HVPE and the surface roughness of the substrate is relatively low. So, we just need to grind and polish it a little, which will greatly reduce the grinding loss. This method provides a very promising technical route for the simultaneous etching of multi-layer Si-doped sacrificial layers to achieve multiple liftoff and reduce the production cost of freestanding GaN in the future.

2. Experiment

A sandwich structure composed of an Fe-doped freestanding GaN substrate (350 μm), a highly conductive Si-doped sacrificial layer (100 μm) and a top Fe-doped layer (320 μm) was grown by HVPE, as shown in Fig. 1(a). Metallic Ga and NH_3 were used as the Ga and N sources, respectively. $FeCl_2$ gas produced by a reaction between metallic Fe and gaseous HCl was used as the Fe source, while SiH_4 gas was used as the Si source and 2 inch Fe-doped freestanding GaN was used as the substrate. The temperature of the Ga and Fe sources was controlled at 850 °C, while the growth was carried out at 1040 °C.

After growth, the periphery of the sample was ground off by a spheronizator to expose the sacrificial layer for the convenience of the deposition of the electrode. The ohmic contact electrode was Ti/Al/Ni/Au (thickness: 20 nm/130 nm/50 nm/150 nm; annealed at 890 °C for 30 s) prepared by magnetron sputtering. The ohmic contact electrode was connected to the positive terminal of a voltage source, while a Pt electrode was connected to the negative terminal. Both the Pt electrode and GaN sample were immersed in the electrolyte (0.6 ml oxalic acid). The immersion depth of the GaN sample gradually increases with the etching process to realize complete liftoff. Good ohmic contact is crucial to this work, which can ensure that the voltage is applied at the interface between the sample and electrolyte. Electrochemical etching was performed using a potentiostat (K2400) at room temperature, as shown in Fig. 1(b).

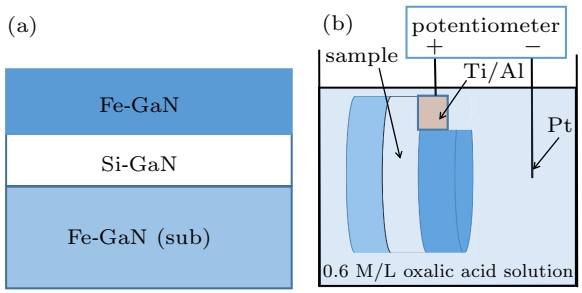


Fig. 1. Schematic of (a) the sample structure, and (b) the equipment used for the electrochemical etching.

Scanning electron microscopy (SEM) and atomic force microscopy (AFM) were used to characterize the surface morphology of GaN after electrochemical etching on both the Ga- and N-face. The etching current from anode to cathode is proportional to the etching rate. Hence, it could be utilized to reflect the fluctuation of the etching rate. The etching products from the sacrificial layer were measured by x-ray diffraction (XRD), energy dispersive x-ray spectroscopy (EDX) and Raman spectrometry.

3. Results and discussion

Figure 2(a) shows the cross-sectional SEM image of a sample etched by 100 V. It is found that the highly conductive sacrificial layer is completely etched off, while the highly resistive Fe-doped GaN remain unchanged, as shown in Fig. 2(b). The interface between the etched and unetched area is sharp. Figure 2(c) shows the Raman spectra of the substrate, sacrificial layer and top layer. For n-type GaN, the carrier concentration could be calculated from the peak position of the longitudinal optical phonon-plasmon coupled mode (LOPC) in the Raman spectrum.[24] The LOPC mode usually consists of upper- and lower-frequency branches (denoted by L+ and L−, respectively) and here the L-peak was used for calculation. The wavenumber of the L-peak is 424 cm^{-1} in the Si-doped GaN and the carrier concentration is calculated to be 6.2×10^{18} cm^{-3}. According to the relationship between resistivity and carrier concentration, the resistivity could be estimated to be in the order of 10^{-3} ohm·cm. However, the peak position of L− in the Fe-doped substrate and top layer could hardly be observed due to the lower carrier concentration. Moreover, the impurity concentration of Fe is measured by SIMS, which is 4×10^{18} cm^{-3} for Fe impurity, as shown in Fig. 2(d). Therefore, the carrier concentration (the resistivity)

in the Si-doped layer in much higher (much lower) than that in the Fe-doped layer, resulting in the big difference in etching rate.[25,26]

When the voltage is applied, it is equivalent to applying reverse voltage to the Schottky barrier at the interface between the GaN and electrolyte, leading to the generation of tunneling current in the space-charge region.[27] In order to realize large-scale liftoff, sufficient tunneling current is needed to form a lot of holes on the surface to oxidize the GaN.

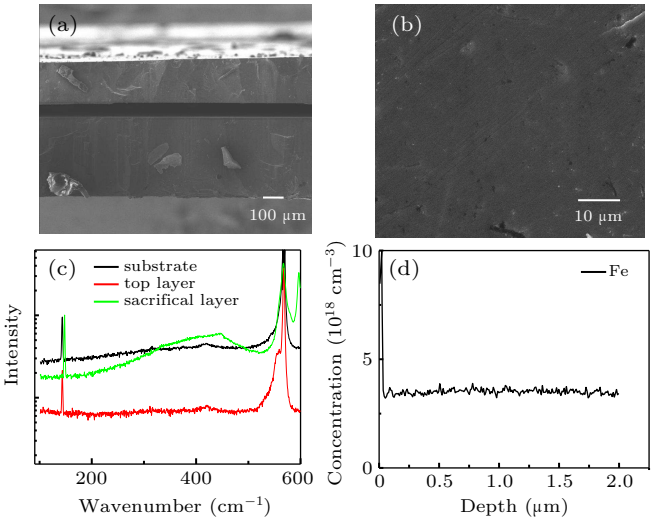


Fig. 2. (a) SEM image of the sample after etching by 100 V, (b) enlarged SEM image of Fe-GaN cross-section after being etched, (c) Raman spectrum of sacrificial layer, substrate and top layer, respectively, (d) SIMS shows the Fe impurity concentration of the substrate and top layer.

Figure 3 shows the influence of applied voltage on etching rate. All the samples are etched for 30 min at different voltage. The etching rate is calculated by dividing the etching depth by etching time, and the etching depth is measured by SEM. It is found that the etching rate increases nearly linearly with the applied voltage from 40 V to 110 V. The etching rate is as high as 30 μm/min at 110 V.

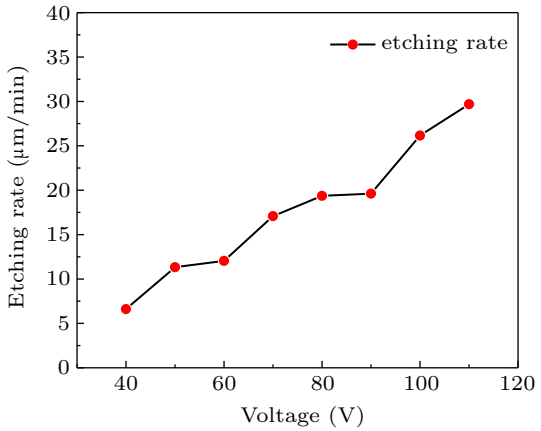


Fig. 3. Dependence of the etching rate on the applied voltage.

Figure 4(a) shows that the etching rate decreased with the increase of etching depth, especially when the depth is above 1 mm, which is a barrier for large-scale liftoff. It could also be found that the etching current decreased with time, as shown in Fig. 4(b). This problem may be concerned with mass transport limitation, because many flaky GaN pieces appear in the sacrificial layer after electrochemical etching, as shown in Figs. 4(c) and 4(d). Moreover, the flaky GaN pieces do not diminish or vanish with the increase in voltage. Figure 4(d) shows that the flaky GaN could be as high as 65 μm, about three-quarters of the thickness of the sacrificial layer, which is a very serious barrier to mass transport.

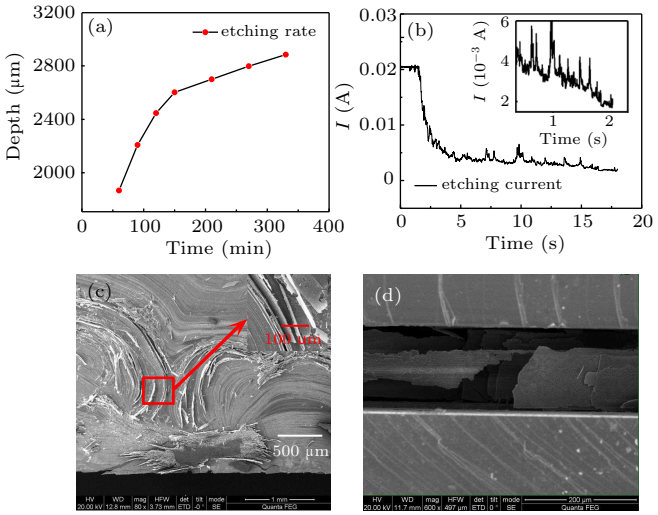


Fig. 4. (a) Dependence of etching rate on etching depth; (b) change of etching current with etching time; (c) plane-view SEM image of completely etched Si-doped GaN sacrificial layer at 100 V for 6 h. Inset shows a magnified image of the red region; (d) cross-sectional SEM image of the flaky GaN in the sacrificial layer.

It has been reported that the nanopore (NP) side wall formed by electrochemical etching GaN is oxide. Because no lattice fringes are presented in the bulk GaN/NP GaN interface through HRTEM, it was found that the sidewall of the NP GaN should be surrounded by an amorphous structure.[28] Figure 5(a) shows the cross-sectional SEM image of the sample after etching. Many flaky pieces appear in the sacrificial layer.

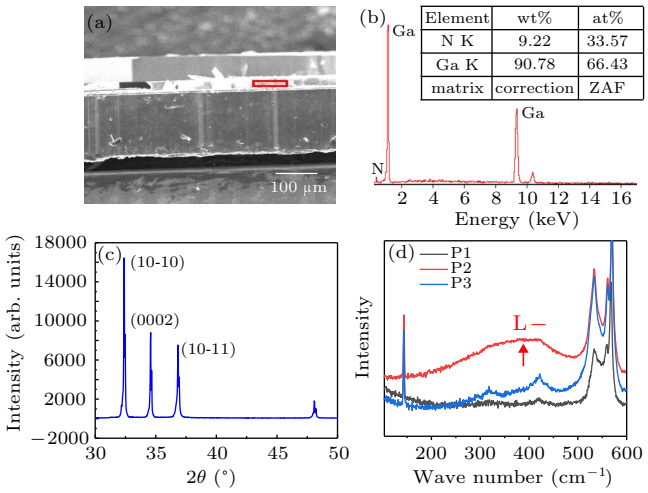


Fig. 5. (a) Cross-sectional SEM image of the sample after electrochemical etching. (b) EDX spectra from the area marked in the red box in (a). (c) Powder XRD data of the flaky products. (d) Raman spectrum of Fe-doped GaN (P1), Si-doped sacrificial layer (P2) and flaky GaN (P3), respectively.

The composition of the pieces is measured by EDX, which proves that they are GaN, but not oxide, as shown in Fig. 5(b). Powder XRD also verifies that the residual products in the sacrificial layer are GaN, as shown in Fig. 5(c). The flaky GaN was measured by Raman spectrum (P3), which indicates that the carrier concentration is extremely low, as shown in Fig. 5(d). Hence, the flaky GaN pieces were left behind because the etching rate is much slower than the highly conductive sacrificial GaN. The residual GaN could block the etching channel and reduce the etching rate.

Besides, the nitrogen gas generated from the electrochemical etching process is another reason for the decrease in etching rate. The nitrogen gas could attach to the interface between the etched and unetched area and isolate the unetched area from the electrolyte, resulting in the decrease in etching rate and the formation of flaky GaN. And, because the etching speed is different in each place, the etching front looks serrated at a certain moment, as shown in Fig. 6(b). There are three reasons for the formation of isolated flaky GaN. First, the uncertainty of the etching direction leads to the NP bending and connecting with adjacent NPs, resulting in the formation of isolated GaN, as shown at position 1 in Fig. 6(a). Second, the isolated GaN marked by position 2 in Fig. 6(a) is caused by etching along a direction that could also be found at position 1 and 2 in Figs. 6(c) and 6(d), respectively. Third, the isolated GaN marked by position 3 occurs as a result of the reason mentioned above.

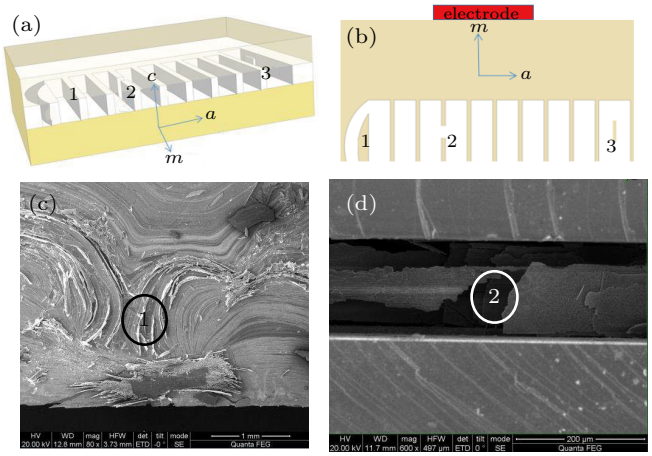


Fig. 6. (a) Schematic diagram of the topography of the etching front at a certain time. There are three isolated GaN marked by 1, 2, 3. Crystal orientation (*m*, *a*, *c*) of the sample is also indicated. (b) Top view of the etching front. (c) Plane-view SEM image of completely etched Si-doped GaN sacrificial layer at 100 V for 6 h. (d) Cross-sectional SEM image of the flaky GaN in the sacrificial layer.

In order to remove the flaky GaN to make the etching channel free enough, ultrasonic shaking is applied. Figure 7(a) shows the influence of ultrasonic shaking on the etching current. When the oscillation is turned on, the flaky GaN could be seen falling off from the sacrificial layer due to resonance and the etching current increases rapidly. When the oscillation is turned off, the etching current gradually decreases, indicating that the flaky GaN pieces begin accumulating and the etching channel becomes crowded.

Moreover, temperature could also affect the etching rate. Figure 7(b) shows that the etching current increases with temperature, because the ion transport in the electrolyte becomes faster with the increase in temperature. As the temperature increases from 20 °C to 60 °C, the etching current increases from 1.5 mA to 8 mA.

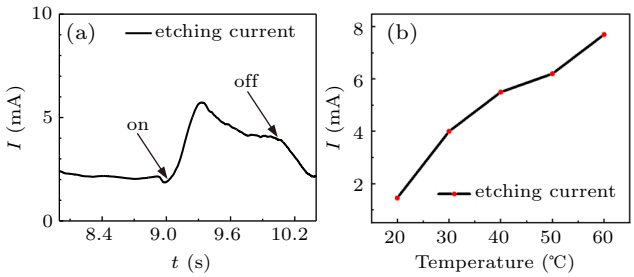


Fig. 7. (a), (b) Change of etching current with the oscillating switch and temperature.

Ultra-thick sacrificial layer grown by HVPE makes it possible to separate large-size GaN substrate, because it alleviates the mass transport limitation. So, under the condition of ultrasonic shaking and high temperature (60 °C), we succeed in the liftoff of nearly 1.5 inch freestanding GaN by electrochemical etching at 100 V. The separated substrate and top layer are shown in Fig. 8.

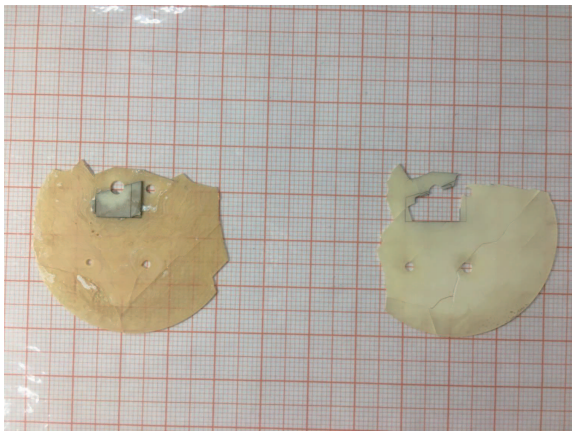


Fig. 8. Image of nearly 1.5 inch separated substrate and epitaxial layer. Inset is the liftoff of the 20 × 10 sample.

Figures 9(a)–9(c) show the SEM images of the Ga-face of the substrate, N-face of the top layer and Ga-face of the top layer, respectively. The SEM image of the Ga-face of the top layer before etching is also shown in Fig. 9(d) for comparison. The corresponding AFM images are shown in Figs. 9(e)–9(h). The roughness of the Ga-face of the substrate, N-face of the top layer and Ga-face of the top layer is 18.7 nm, 9.53 nm, and 2.04 nm, respectively, which is close to the previously reported results.[16] High roughness results in various micro defects in the epitaxial layer during high-temperature crystallization, which affects the quality of the epitaxial layer.[29] The roughness of the Ga-face of the top layer is 1.82 nm before etching, which is similar to the value after etching. This indicates that the Ga-face remains unchanged during the etching process.

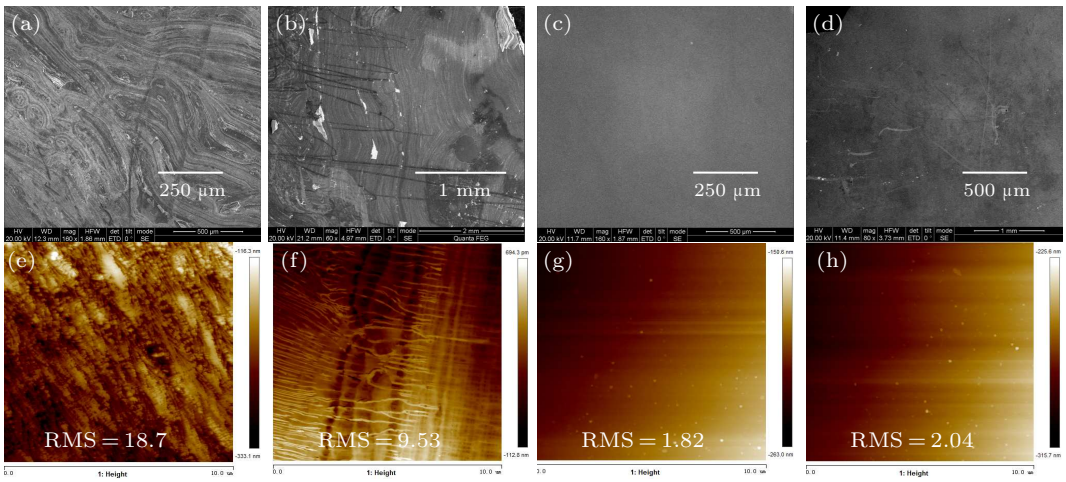


Fig. 9. (a)–(d) SEM images of the Ga surface of the substrate, N surface of the epitaxial layer and Ga surface before and after etching. AFM scans (10×10 μm) (e)–(h) correspond to SEM images (a)–(d), respectively.

In conclusion, we have demonstrated the feasibility of electrochemical liftoff of large-area freestanding GaN with a sandwiched structure, including an Fe-doped substrate, a highly Si-doped sacrificial layer and a top Fe-doped layer. The etching rate decreases with the etching process as a result of the mass transport limitation. A thick sacrificial layer, together with ultrasonic shaking, high voltage and high temperature, could help solve the problem of transportation jam, which is beneficial to lift off large-size GaN substrate. Finally, under the condition of ultrasonic shaking and 60 °C, we obtained more than 1.5 inch GaN substrate by electrochemical etching at 100 V. This work could help reduce the cost of GaN substrate and lays a foundation for engineering production of GaN substrate by electrochemical etching method.

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