Enhanced Wet Etching of Patterned GaN with Ion Implantation

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We present the enhanced wet etching of GaN epilayer implanted with Au+ ion. Patterned GaN with 2 μm-wide sink-like strips was achieved by using 500 keV Au+ ion implantation and KOH etching. The Dependence of etching depth on etching time for the implantation at different ion fluences was investigated. The experiment showed that the damaged GaN area could be almost etched out at high ion fluence, and the etching depth could exceed the project range of incident 500 keV Au+ ion. The etch pits could be observed at the bottom of the etched area. The ∼400 nm depth etching could be achieved with high fluence implantation after a long etching time, and the edge of etched area could remain clear until the etching process had passed 40 min. As-deposited SiO2 spheres were used to mask the GaN sample in implantation process to investigate the etching effect. ∼70 nm wave of the GaN surface was observed. The results of our experiments may suggest an approach to the fabricating of GaN devices.

Keywords: GaN, Implantation, Wet Etching, KOH.

1. INTRODUCTION

Gallium nitride (GaN) is a wide-band gap semiconductor material with high thermal conductivity, high melting point, and a high degree of hardness. The outstanding physical and chemical properties of GaN make it a suitable material in advanced optoelectronic and microelectronic device applications, e.g., light-emitting devices, laser diodes, and ultraviolet detectors.1 Various dry etchants have been investigated on GaN for its excellent chemical stability as characterized by its invulnerabilities, such as electron cyclotron resonance (ECR), inductively coupled plasmas (ICP), reactive ion etching (RIE) and photoassisted dry etching.2–4 Nevertheless, there are several disadvantages to dry etching including ion-induced damage and difficulty in obtaining smooth sidewalls.5,6 Wet etching has the advantage for providing low equipment cost and complexity, low surface damage and selectiveness of different materials. As an important complement to dry etching, wet etching has a variety of applications to wide band gap semiconductor technology, including defect decoration, polarity and polytypic identification by producing characteristic pits or hillocks, and device fabrication on smooth surfaces.7 However, only very low etch rates have been reported for GaN films using wet chemical etchants.8,9 In this paper, we have developed the enhanced wet etching with ion implantation on the GaN surface with photoresist or SiO2 spheres patterned. The Dependence of etching depth on etching time at different ion fluences was investigated. The experiment showed that the damaged GaN area induced by ion implantation could be almost etched out by KOH solution, and the etching depth could exceed the project range of incident 500 keV Au+ ion. Moreover, As-deposited SiO2 spheres were used to mask the GaN sample in implantation process to investigate the etching effect. ∼70 nm wave of the GaN surface was observed. The results of our experiments may suggest an approach to the fabricating of GaN micro and nano devices.
2. EXPERIMENTAL DETAILS

A ∼2 μm-thick wurtzite undoped GaN (0001) epilayer used in this research program were epitaxially grown on a c-plane sapphire substrate by metal-organic chemical vapor deposition (MOCVD). The mass density of the GaN layer is about 6.1 g/cm³. Implantation of 500 keV Au⁺ ions was performed by using a 2 × 1.7 MV tandem accelerator (NEC, 5SDH-2) at room temperature (RT) with ion fluence from 5 × 10¹⁴ to 3 × 10¹⁶ cm⁻². The implantations were carried out at a constant beam flux of 2.5 × 10¹⁴ cm⁻²·s⁻¹. During implantation, to avoid channeling effect, the sample crystallographic axes (0001) were titled by ∼7° relative to the incident ion beam. As shown in Figure 1, before implantation, some GaN samples were masked by a 2 μm-thick photore sist (AZ 6130) patterned with 2 μm-wide strips and 5 μm-wide spacing, and were removed after implantation by organic solvent (Acetone). The other virgin GaN samples were masked with as-deposited SiO₂ spheres (d = 340 nm) in implantation. The implanted GaN samples were wet etched for 20–120 min in 2 mol·L⁻¹ KOH at 80 °C. The surface morphology of the implanted and etched GaN was studied with contact-mode atomic force microscopy (AFM, SPA400 SPI3800N) and scanning electron microscopy (SEM, STARTA DB235). Because the unimplanted area of GaN samples is hardly etched by KOH solution, the etch depth could be considered to be equal to the step height between implanted and unimplanted area measured with AFM.

3. RESULTS AND DISCUSSION

Figure 2 shows the dependence of etching depth on etching time for the implantation at three different ion fluences 5 × 10¹⁴, 5 × 10¹⁵ and 3 × 10¹⁶ cm⁻². Before etching, the ∼40 nm swelling and ∼200 nm depth erosion on patterned GaN surface after being implanted at the fluence of 5 × 10¹⁵ cm⁻² and 3 × 10¹⁶ cm⁻² have been investigated respectively (The step height has been marked at axes 0 minute as shown in Fig. 2). The details about swelling or erosion will be shown in another article.¹⁰ The evolution of etch rate could be divided into three stages. At the first 20 min etching, there was a negligibly small etch rate of GaN implanted at the fluence of 5 × 10¹⁴ cm⁻². The average etch rate of GaN implanted at the fluence of 5 × 10¹⁵ and 3 × 10¹⁶ cm⁻² was ∼8 nm/min, which was higher than the rate (<1 nm/min in 50% KOH solution at 83 °C) mentioned in Ref. [11]. After etched for 20 min, there was an increase of etch rate of GaN implanted at the fluence of 5 × 10¹⁴ cm⁻², but still kept less than 2 nm/min. There was only 20 nm increase of etching depth in GaN implanted at the fluence of 5 × 10¹⁵ and 3 × 10¹⁶ cm⁻². After etched for 80 min, the roughness of etched GaN sidewalls increased to a high point, and the
etching pattern was distorted, which made the increase of etching depth no sense.

Figure 3 shows the surface morphology and section of the GaN wet etched for 20 min in KOH at 80 °C after being implanted with 500 keV Au⁺ at fluence of 5 × 10¹⁵ cm⁻² The smooth, unimplanted surface and the etched implanted area could be observed in the AFM and SEM micrograph of Figure 3. Microscopy images show that the damaged area where the anomalous swelling mentioned above was observed had been taken off by applying wet etching approach. The etching depth is ~130 nm, and the 2 μm-wide strip of the implanted area, comparatively, maintained its original width The strip sidewall was vertical and smooth, indicating the usefulness of this etching approach in device fabrication. It is interesting to note that the etching depth has exceeded the peak projected range of incident 500 keV Au⁺ ion (~70 nm, stimulated based on SRIM2006¹). Considered with the threshold displacement energy for both Ga and N is 25 eV, at the etched bottom corresponding with the projected range 130 nm, the average number of Ga and

![Fig. 3. AFM image with cross sectional profile (a) and SEM image (b) of the GaN sample surface implanted through the mask with 500 keV Au⁺ at RT to the ion fluence of 5 × 10¹⁵ cm⁻² and etched for 20 min in 2 mol·L⁻¹ KOH at 80 °C.](image)

![Fig. 4. SEM image of the patterned GaN sample implanted through the mask with 500 keV Au⁺ at RT to the ion fluence of 5 × 10¹⁵ cm⁻² and etched for 40 min in 2 mol·L⁻¹ KOH at 80 °C.](image)

![Fig. 5. SEM image of the patterned GaN sample implanted through the mask with 500 keV Au⁺ at RT to the ion fluence of 3 × 10¹⁶ cm⁻² and etched for 40 min in 2 mol·L⁻¹ KOH at 80 °C.](image)
N vacancies induced by one Au\(^+\) ion is 0.25, and the lattice damage induced by ion implantation at the fluence of 5 \(\times\) 10\(^{15}\) cm\(^{-2}\) is \(~3\) dpa (3 displacement per atom, simulated based on SRIM2006). The result maybe suggested the threshold of lattice damage at which level the enhanced etching by ion implantation could be achieved.

The etch pits were obvious at the bottom of the sink-like strip with a very low density (about 10\(^7\) cm\(^{-2}\)) as shown in Figure 3(b), and the detail of pit was observed in the GaN sample etched for 40 min as shown in Figure 4. The etch pits exhibited hexagon composed by 6 competing etch planes with intersecting the c plane, suggesting the sites having original defects which etched by KOH solution associated with the ion implantation damage as mentioned in Ref. [13]. Figure 5 shows the surface morphology of GaN wet etched for 40 min in KOH at 80 °C after being implanted with 500 keV Au\(^+\) at the fluence of 3 \(\times\) 10\(^{16}\) cm\(^{-2}\). The etching depth is \(~400\) nm including the ion erosion depth. It is obvious that the width of the implanted area increased after etching, and the sidewall was eroded heavily by KOH solution. The density of etch pits at strip bottom increased to 10\(^8\) cm\(^{-2}\), which may be attributed to the damage induced by higher ion implantation fluence.

As-deposited SiO\(_2\) spheres (\(d = 340\) nm) were also used to mask the GaN sample in implantation process to investigate the etching effect. The SiO\(_2\) spheres had been removed before GaN was etched. The \(~70\) nm wave of the GaN surface was observed after being implanted with 500 keV Au\(^+\) at RT to the fluence of 5 \(\times\) 10\(^{15}\) cm\(^{-2}\) and wet etched for 20 min in 2 mol \(\cdot\) L\(^{-1}\) KOH at 80 °C as shown in Figure 6. The results suggest an approach to making micro or even nano-level structures, and the patterned local doping or electrical isolation may also be tried, which is under investigation in our project.

4. CONCLUSIONS

An enhanced wet etching process of GaN epilayer has been developed by ion implantation approach. The dependence of etching depth on etching time for the implantation at different ion fluences was investigated. The average etch rate of GaN implanted at the fluence of 5 \(\times\) 10\(^{15}\) and 3 \(\times\) 10\(^{16}\) cm\(^{-2}\) was \(~8\) nm/min in 2 mol \(\cdot\) L\(^{-1}\) KOH at 80 °C at the first 20 min. AFM and SEM analysis of the etched area indicated the usefulness of this etching approach in device fabrication when the ion influence was 5 \(\times\) 10\(^{15}\) cm\(^{-2}\). The etch pits were observed at the bottom of the sink-like strip, and the threshold of lattice damage at which level the enhanced etching by ion implantation could be achieved was suggested to be 3 dpa. The SiO\(_2\) spheres mask was also used to investigate an approach to produce nano-level structures.

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References and Notes


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