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# Study of dry etching for GaN and InGaN-based laser structure using inductively coupled plasma reactive ion etching

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## Abstract

Dry etching of undoped, n-GaN, p-GaN and InGaN laser structure was investigated by inductively coupled plasmas reactive ion etching (ICP-RIE) using Ni mask. As  $Cl_2/Ar$  gas flow rates were fixed at 10/25 sccm, the etched surface roughness has the lowest value of 0.2 nm at constant ICP/bias power = 300/100 W and 5 mTorr chamber pressure for undoped GaN. The highest etching rate of 12,000 Å/min for n-GaN was achieved at 30 mTorr, 300 W ICP, 100 W bias power using low  $Cl_2$  flow rate ( $Cl_2/Ar = 10/25$  sccm) gas mixtures. The surface roughness was dependent of bias power and chamber pressure, and shows a low root mean square (rms) roughness value of about 1 nm at 50 W of bias power for n-GaN and p-GaN. For etching of InGaN laser structure using high  $Cl_2$  flow rate ( $Cl_2/Ar = 50/20$  sccm) and low chamber pressure 5 mTorr, a smooth mirror-like facet of InGaN laser diode structure was obtained. Using these etching parameters, mirror-like facets can be obtained which can be used for the fabrication of nitride-based laser diodes. Moreover, at the fixed  $Cl_2/Ar$  flow rate of 10/25 sccm, ICP/bias power of 200/100 W and chamber pressure of 30 mTorr, the InGaN-based materials nanorods were fabricated with a density of about  $10^8$  cm<sup>-2</sup> and dimension of 50-100 nm.

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Keywords: GaN; Inductively coupled plasma (ICP); Mirror-like facet; Laser diode; Nanorods

## 1. Introduction

GaN and InGaN-based compound semiconductors and other group III-nitrides have been successfully employed to fulfill blue-green light-emitting diodes and blue laser diodes [1–4]. Due to their excellent chemical and thermal stabilities, the wet etching on GaN and InGaN-based material are difficult with no other assistance, and results in low etching rate and isotropic etching profile. The dry etching technique for GaN-based material shows anisotropic etching profile and fast etching rates. A number of studies on the dry etching of GaN and related compounds have been reported earlier [5–8] using different dry etching technology including reactive ion etching (RIE), electron cyclotron resonance (ECR) etching, chemically assisted ion beam etching, and inductively coupled plasma (ICP). It is known that using ECR plasma etching could obtain higher degree of anisotropy profile and faster etching rate than RIE etching because of the higher plasma density of ECR.

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But, compared to ICP system, ICP have several advantages than ECR including improved plasma uniformity, lower cost and easier scale-up production [9]. Previous dry etching investigations on GaN using the ICP system have been obtained the fastest etching rate and high quality mirror-like facet [10-12]. Improved etch results for GaN in the Ni mask using ICP system have reported [13] and show a high selectivity and a good surface morphology. Additionally, for GaN-based materials, the dry etching technology also could fabricate nanostructure [14,15] and become an alternate new method to form nanorods or nanowires. The fabrication and synthesis of GaN nanowires and nanorods using various methods have been reported, for example, carbon nanotube-confined reaction, metal-catalyzed growth assisted by laser ablation, the high-temperature pyrolysis approach and so on [16-21]. The photoenhanced wet etching technique to produce GaN whiskers [22-24] was also reported recently. However, all these reported methods are relatively complicated and mostly use the synthesis approach with the aid of catalysts. In this paper, a parametric study etching results including etching rate, etched surface morphology and sidewall were investigated as functions of Cl<sub>2</sub>/Ar gases flow rate for undoped-GaN films, ICP power, bias power and

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chamber pressure for n-GaN and p-GaN films. Moreover, we also apply the etching result to fabricate very smooth and highly anisotropic facets on InGaN laser structure, and create the InGaN-based material nanostructure as earlier reported [15].

# 2. Experiment

The undoped-GaN and n-GaN samples with a thickness of 2-3 µm, p-GaN samples with 1 µm thickness and InGaN laser structure were all grown by metalorganic chemical vapor deposition (MOCVD) on c-face sapphire substrates. Fig. 1 depicts schematic a diagram of the InGaN laser structure, including sapphire substrate, a 30 nm-thick GaN buffer layer, 2 µm-thick n-GaN:Si, 0.4 µm-thick n-Al<sub>0.1</sub>GaN:Si, MQW structure with 2.5 nm-thick InGaN wells sandwiched between 7.5 nm-thick GaN barrier, 0.4 µm-thick p-Al<sub>0.1</sub>GaN:Mg and 0.3  $\mu$ m-thick p-GaN:Mg. The 300  $\mu$ m  $\times$  150  $\mu$ m Ni mask pattern was formed by means of photoresist lithography, and then the Ni layer with a thickness of about 1000 Å was deposited by E-gun. Finally a lift-off technique was used to form the Ni mask in acetone. After the fabrication of Ni mask, the GaN samples were placed inside a load-lock chamber, which is connected to the ICP reactor chamber for etching. The ICP etching equipment was a planar ICP-RIE system (SAMCO ICP-RIE 101iPH). The ICP power and bias power source with rf frequency were set at 13.56 MHz. The etchant gases were Cl<sub>2</sub> and Ar. Both Cl<sub>2</sub> and Ar gases were introduced into the reactor chamber through independent electronic mass flow controllers (MFCs) that can control the flow rate of each gas with an accuracy of about 1 sccm. An automatic pressure controller (APC) was placed near the exhaust end of the chamber to control the chamber pressure. In this paper, the surface morphology was measured by atomic force microscopy (AFM), scanning electron microscopy (SEM), and the etching rates were calculated from the etched depth of samples from the Dektek II stylus profilometry measurements after the removal of the Ni mask.

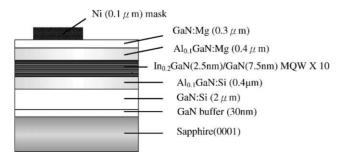


Fig. 1. Schematic diagram of the InGaN/GaN MQW laser structure.

# 3. Results and discussions

## 3.1. Functions of Cl<sub>2</sub>/Ar gases flow rate

First, we investigate the effects of  $\text{Cl}_2$  and Ar gases flow rate on the etching rate and surface morphology for undoped-GaN. Fig. 2(a) and (b) showed the etching rates and etched surface root mean square roughness for undoped-GaN film as a function of  $\text{Cl}_2$  and Ar gases flow rate respectively at constant ICP/bias power =  $300/100\,\text{W}$  and 5 mTorr chamber pressure. In Fig. 2(a), the Ar flow rate was maintained at 25 sccm during the etching process, and it can be observed that the surface roughness has the lowest value of 0.2 nm at 10 sccm of  $\text{Cl}_2$  and has no major change with increasing the  $\text{Cl}_2$  flow rate. It seems that the etching rate increased with increasing the  $\text{Cl}_2$  flow rate because of higher concentration of  $\text{Cl}_1$  radicals in the plasma.

In Fig. 2(b), the  $\text{Cl}_2$  flow rate was fixed at 10 sccm during the etching process. The surface roughness remained relatively constant as the Ar flow rate increased up to 25 sccm. As shown in Fig. 2(b), the addition of Ar below 25 sccm

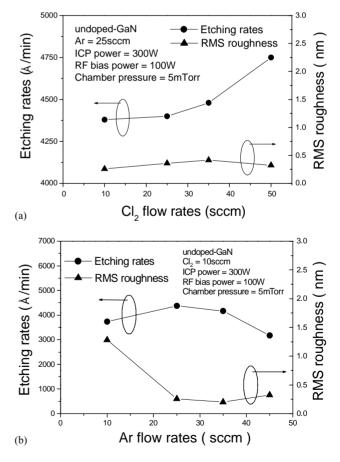


Fig. 2. (a) Effect of  $\text{Cl}_2$  flow rate on etching rates and rms surface roughness for undoped-GaN with constant ICP/bias power =  $300/100\,\text{W}$ , 25 sccm of Ar and 5 mTorr chamber pressure, and (b) effect of Ar flow rate on etching rates and rms surface roughness for undoped-GaN with constant ICP/bias power =  $300/100\,\text{W}$ ,  $10\,\text{sccm}$  of  $\text{Cl}_2$  and 5 mTorr chamber pressure.

increased the undoped-GaN etching rate and obtained the highest etching rate of about 4300 Å/min at 25 sccm of Ar. However, the addition of Ar more than 25 sccm decreased the etching rate, and it could be explained that the addition of a small amount of Ar can enhance the removal of etch products such as  $GaCl_x$  by physical ion bombardment. As further increased the Ar flow rate, the reduction of etching rate was due to less available Cl radicals. Therefore, the  $Cl_2/Ar$  gas flow rates were fixed at 10/25 sccm in the following experiments.

# 3.2. Functions of ICP/bias/chamber pressure

Fig. 3 showed the etching rates and etched surface rms roughness for n- and p-type GaN film as a function of the ICP source power. During the etching process, the chamber pressure, bias power, and the gas mixture were held constant at 5 mTorr, 100 W, and 10 sccm  $\text{Cl}_2/25$  sccm Ar, respectively. The etching rates of n-GaN and p-GaN increased rapidly with the ICP source power increased from 100 to 300 W. It is primarily due to higher concentrations of reactive chlorine

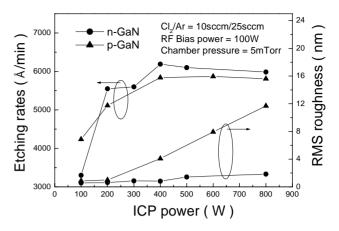


Fig. 3. Effect of ICP source power on etching rates and rms surface roughness for n-GaN and p-GaN with Cl<sub>2</sub>/Ar plasma chemistries.

radicals, which increased the chemical component of the etching mechanism, and higher ion flux, which increases the bond breaking and sputter desorption efficiency of the process. However, the etching rates have also been observed

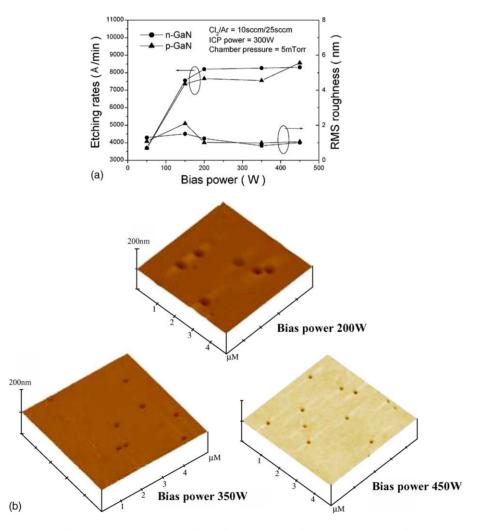


Fig. 4. (a) Effect of bias power on etching rates and rms roughness for n-GaN and p-GaN with  $Cl_2/Ar$  plasma chemistries, and (b) the AFM measurement result of n-GaN for various bias powers of 200, 350, 450 W.

to stabilize and gradually decrease over 400 W of ICP power, it result from the saturation of reactive radicals at the surface or sputter desorption of reactive species from the surface before the process occurring. The n-GaN and p-GaN rms roughness was also smoother about 1–2 nm from 100 to 300 W.

The n-GaN and p-GaN etching rates and rms roughness as a function of bias power at the chamber pressure of 5 mTorr, ICP source power of 300 W, and the same gas mixture of 10 sccm Cl<sub>2</sub>/25 sccm Ar were shown in Fig. 4(a). The n-GaN and p-GaN etching rates increased monotonically as the bias power and became stabilization over 200 W. The ion bombardment energy was mainly depended on the bias power. With increasing the bias power, ion bombardment energy was increased and resulted in an increase in etching rate. However, the rms roughness was equal smooth of about 1 nm and is independent of the bias power up to 200 W.

Furthermore, as shown in Fig. 4(b), it can be observed that the increase of ion bombardment over the saturated bias power of 200 W would induce more pits or dislocations density on the surface of samples. The variation in the density of the pits seems to be related to the bias power. In our experiment, the pits density is about  $2.4 \times 10^9 \, \mathrm{cm}^{-2}$  at 200 W of bias power, increases to about  $3.2 \times 10^9 \, \mathrm{cm}^{-2}$  at 350 W and  $4 \times 10^{10} \, \mathrm{cm}^{-2}$  at 450 W. It could be explained that the addition of ion bombardment energy did not affect the surface roughness, but cause more pits or dislocations.

The effect of chamber pressure on the etching rates and surface roughness was shown in Fig. 5. While the ICP power and Bias power were fixed at 300, 100 W, respectively, the etching rates increased as the chamber pressure increased from 2.5 to 40 mTorr and reach to the maximum etching rate of about 12000 Å/min. The increase in etching rate suggested a reactant limited regime at the chamber pressure of below 40 mTorr. However at higher chamber pressures of over 40 mTorr, the etching rates gradually decreased due to lower plasma densities resulting from recombination. The SEM data shows the etching surface of n-GaN and p-GaN

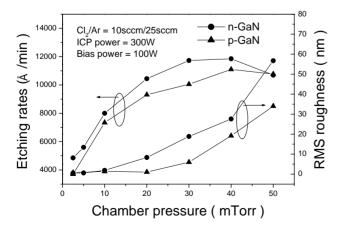


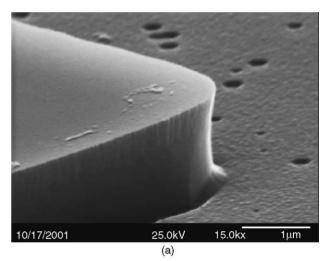
Fig. 5. Effect of chamber pressure on etching rates and rms surface roughness for n-GaN and p-GaN with  $\rm Cl_2/Ar$  plasma chemistries.

has a very smooth profile with sharp vertical sidewall for all etching conditions. However, the chamber pressure was suggested to be below 10 mTorr for etched surface with low surface roughness of ranging from 1 to 3 nm.

## 3.3. InGaN laser structure facet and nanostructure

For the etching of the InGaN laser structure, we chose high  $\text{Cl}_2$  gas flow rate to enhance the chemical etching because of In element in MQW layer. The etching conditions were  $\text{Cl}_2/\text{Ar} = 50/20\,\text{scm}$ , and bias power was 30 W, ICP source power was 30 W, and the chamber pressure was 5 mTorr for 4 min etching time. The etching depth was 11000 Å and the calculated etching rate was about 2750 Å/min, and etched surface roughness was about 1 nm. The SEM micrograph of such an etched mirror is shown in Fig. 6(a) and the mirror-like facet of GaN can be obtained using this etching parameter.

The formation of InGaN-based materials nanostructure was shown in Fig. 6(b). The etching was conducted under



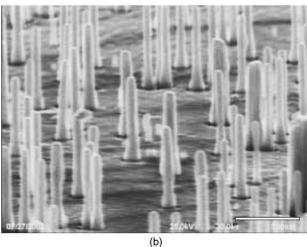


Fig. 6. (a) The SEM micrograph of the mirror-like facet of etched InGaN laser structure, and (b) the SEM micrograph of nanorods of InGaN-based materials

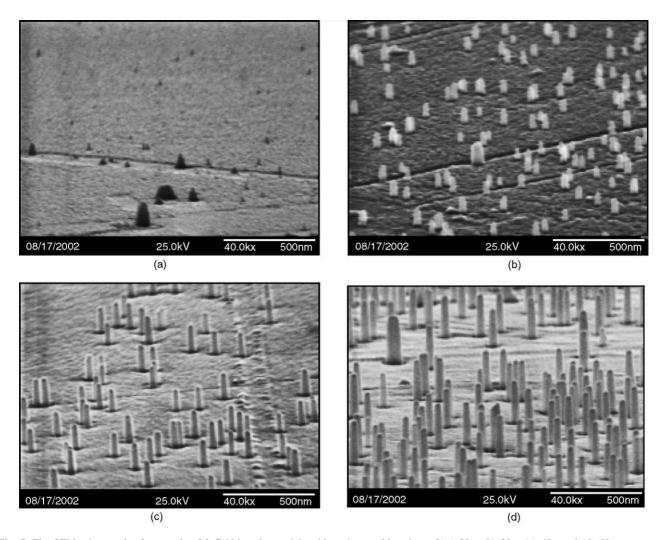


Fig. 7. The SEM micrograph of nanorods of InGaN-based materials with various etching time of (a)  $20 \, \text{s}$ , (b)  $30 \, \text{s}$ , (c)  $40 \, \text{s}$  and (d)  $50 \, \text{s}$  at constant etching condition of  $\text{Cl}_2/\text{Ar} = 10/25 \, \text{sccm}$ , the ICP source power set at  $200 \, \text{W}$ , bias power set at  $100 \, \text{W}$  and chamber pressure set at  $30 \, \text{mTorr}$ .

a gas mixture condition of  $Cl_2/Ar = 10/25$  sccm with the ICP source power set at 200 W, bias power set at 100 W and chamber pressure set at 30 mTorr for a 2 min etching time. The InGaN-based materials nanorods have a near hexagonal structure with a height of about 0.6 µm and the dimension and density of about 50–100 nm and 10<sup>8</sup> cm<sup>-2</sup>, respectively, as estimated from the SEM image of Fig. 6(b), and obtain calculated etching rate of about 3000 Å/min. The exact mechanism of the formation of nanostructure is not fully understood yet. However, the creations of nanorods seem to be related to the crystalline quality of epitaxially grown GaN sample material and the ability of the ICP process to dissociate GaN bonding. Generally, MOCVD-grown GaN on a sapphire substrate is known to have high dislocations and defect density on the order of  $10^8-10^9$  cm<sup>-2</sup> due to a lattice mismatch between the GaN and substrate. These dislocations and defects tend to have weaker binding energy and could be easily dissociated by the ICP etching process leaving behind the rigid crystalline GaN region [15]. To further investigate the creation of the InGaN nanorods, we vary different etching time of 20, 30, 40, 50 s to be tend to interpret the formation mechanism of nanorods. As shown in Fig. 7, the variation in the height of the nanorods seems to be related to the etching time under the same etching condition, and the dimension and density of the GaN nanorods have no major change. The result suggests that the rigid crystalline GaN region is persisted under this etching condition and is consist with the mention before.

## 4. Conclusion

Dry etching of undoped, n-GaN, p-GaN and InGaN laser structure has been carried out in  $\text{Cl}_2/\text{Ar}$  inductively coupled plasmas using Ni mask. As  $\text{Cl}_2/\text{Ar}$  gas flow rates were fixed at 10/25 sccm, the etched surface roughness has the lowest value of  $0.2 \, \text{nm}$  at constant ICP/bias power =  $300/100 \, \text{W}$  and 5 mTorr chamber pressure for undoped GaN. The highest etching rate of  $12,000 \, \text{Å/min}$  for n-GaN was achieved at  $30 \, \text{mTorr}$ ,  $300 \, \text{W}$  ICP,  $100 \, \text{W}$  bias power using low  $\text{Cl}_2$ 

flow rate ( $\text{Cl}_2/\text{Ar} = 10/25\,\text{sccm}$ ) gas mixtures. The surface roughness was dependent of bias power and chamber pressure, and shows a low rms roughness value of about 1 nm at 50 W of bias power for n-GaN and p-GaN. For etching of InGaN laser structure using high  $\text{Cl}_2$  flow rate ( $\text{Cl}_2/\text{Ar} = 50/20\,\text{sccm}$ ) and low chamber pressure 5 mTorr, a smooth mirror-like facet of InGaN laser diode structure was obtained by ICP system. Using these etching parameters, mirror-like facets can be obtained which can be used for the fabrication of nitride-based laser diodes. Moreover, at the fixed  $\text{Cl}_2/\text{Ar}$  flow rate of  $10/25\,\text{sccm}$ , ICP/bias power of  $200/100\,\text{W}$  and chamber pressure of 30 mTorr, the InGaN-based materials nanorods were fabricated with a density of about  $10^8\,\text{cm}^{-2}$  and dimension of  $50-100\,\text{nm}$ .

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