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Gallium Nitride Nanorods Fabricated by Inductively Coupled Plasma Reactive Ion Etching

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We report a novel method of fabricating gallium nitride (GaN) nanorods of controllable dimension and density from GaN epitaxial film using inductively coupled plasma reactive ion etching (ICP-RIE). The GaN epitaxial film was grown on a sapphire substrate by metal-organic chemical vapor deposition. Under the fixed Cl_2/Ar flow rate of 10/25 sccm and ICP/bias power of 200/200 W, the GaN nanorods and array were fabricated with a density of 10^8–10^{10} cm^{-2} and dimension of 20–100 nm by varying the chamber pressure from 10 to 30 mTorr. The technique offers one-step, controllable method for the fabrication of GaN nanostructures and should be applicable for the fabrication of GaN-based nano-optoelectronic devices.
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KEYWORDS: gallium nitride (GaN), nanorod, inductively coupled plasma (ICP)

Gallium nitride (GaN) and other group III-nitride-based semiconductors have been successfully employed to realize blue light-emitting diodes and blue laser diodes.[1–3] Additionally, due to quantum confinement effects, fabrication and studies of nanostructures have attracted considerable interest for potential application to electronic and optoelectronic devices. With the recent progress in semiconductor process technology, various nanostructure fabrication methods have been investigated such as optical lithography, atomic force microscopy (AFM) machining tools, and metal-catalyzed nanostructure synthesis by a vapour-liquid-solid growth process on different materials.[4,5] For GaN-based materials, 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.[6–11] Furthermore, the use of the postgrowth thermal annealing process to induce quantum dots in multiple quantum well layers[12–14] and the photoenhanced wet etching technique to produce GaN whiskers[15–17] was also reported recently. However, all these reported methods are relatively complicated and mostly use the synthesis approach with the aid of catalysts, and there is no mention of the control of the dimension and density of these fabricated GaN nanorods or nanowires. In this paper, we report a novel and straightforward technique of fabricating GaN nanorods with controllable dimension and density of and obtain bundles of vertically aligned nanorods from GaN epitaxial film using inductively coupled plasma reactive ion etching (ICP-RIE).

The GaN epitaxial film was grown by metal-organic chemical vapor deposition (MOCVD) on a *c*-axis sapphire substrate. The grown GaN sample has a 1-μm-thick p-type GaN layer on top of a 2.0–3.0-μm-thick undoped GaN layer and a 30-nm-thick GaN buffer layer. The experimental procedure for the fabrication of GaN nanorods is described in the following. First, for measurement of the etching depth, a Ni mask was fabricated on top of the GaN samples. The Ni mask was used because of its high etching selectivity.[18] The 300 μm × 150 μm 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_2 and Ar. Both Cl_2 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.

The etching was conducted under a gas mixture condition of $Cl_2/\mathrm{Ar} = 10/25$ standard cubic centimeter min (sccm) with the ICP source power and bias power set at 200 W for a 2 min etching time. GaN nanorods of various dimensions and densities of were obtained by controlling the chamber pressure from 2.5 to 30 mTorr. After the etching process, the etching depth was measured by stylus profilometry. Atomic force microscopy (AFM) was used to investigate the surface morphology and to estimate the nanorod density. The dimensions of the nanorods were estimated by a scanning electron microscope (SEM).

As shown in the SEM image of Fig. 1(a), at and below the chamber pressure of 2.5 mTorr, a uniform etched surface with no nanorod formation was observed and the surface roughness was about 1.5 nm. As the chamber pressure was increased to 10 mTorr, the GaN nanorods began to form and the density of nanorods increased as the chamber pressure was further increased to 20 mTorr and 30 mTorr. The nanorods have a near hexagonal structure with a height of about 1 μm as estimated from the SEM image of Figs. 1(b) and 1(c), and about 0.4 μm at 30 mTorr from Fig. 1(d). The variation in the height of the nanorods seems to be related to the etching rate. In our experiment, the etching rate is about 3000 Å/min at 2.5 mTorr, increases to about 5000 Å/min at 10 mTorr and 20 mTorr, and then decreases to 1450 Å/min at 30 mTorr. This seems to correspond to the variation in height of the nanorods. At 30 mTorr as shown in Fig. 1(d), the nanorods form a two-dimensional array of uniform density. To further illustrate the nanorod array formation and density, a magnified SEM image of Fig. 1(d) and its corresponding AFM image are shown in

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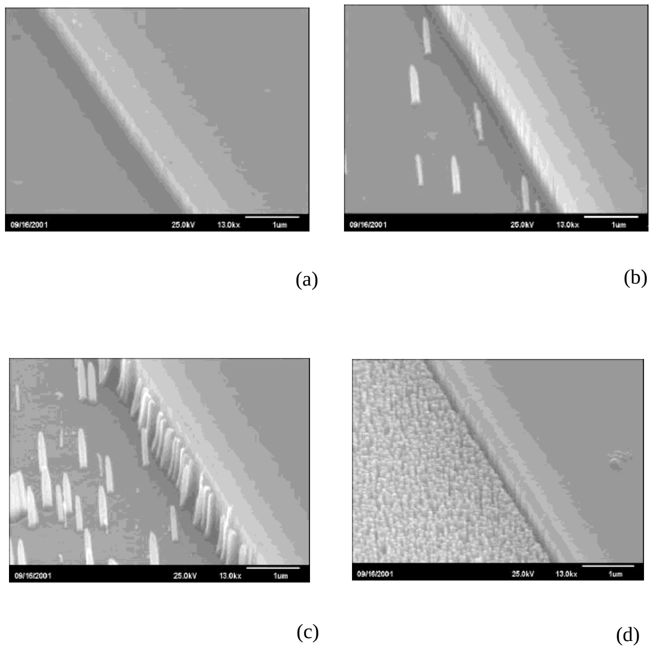

Fig. 1. SEM image of the etched GaN sample surface morphology with different chamber pressures: (a) 2.5 mTorr, (b) 10 mTorr, (c) 20 mTorr, and (d) 30 mTorr at the same Cl_2/Ar flow rate of 10/25 sccm, ICP/bias power of 200/200 W for a 2 min etching time.

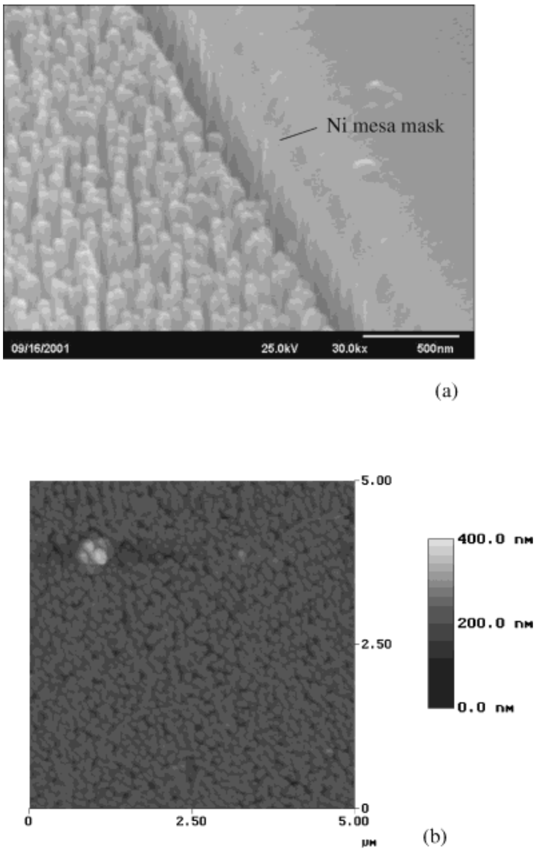

Fig. 2. (a) SEM and (b) AFM images of the etched GaN sample surface morphology with the chamber pressure of 30 mTorr, mixed gas Cl_2/Ar flow rate of 10/25 sccm, and ICP/bias power of 200/200 W for a 2 min etching time.

Figs. 2(a) and 2(b) respectively. The bright spot in the left-upper corner of Fig. 2(b) was believed to be caused by the micromask effect during the etching process.[19]

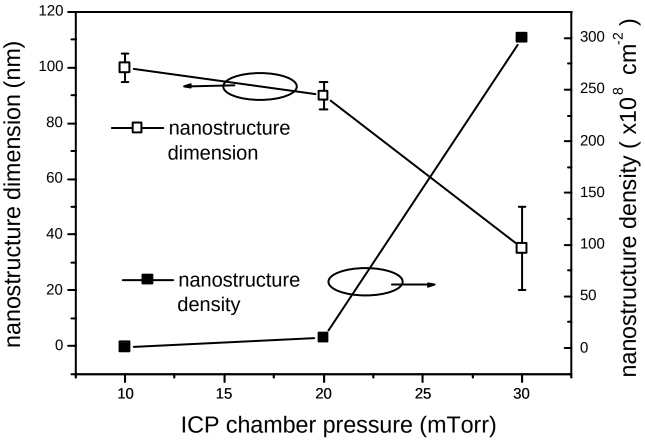

Fig. 3. Mean dimension and density of GaN nanostructure as a function of the chamber pressure which is varied from 10 to 30 mTorr.

From the SEM and AFM data, we estimate the mean dimension and density of the GaN nanorods as a function of the chamber pressure as shown in Fig. 3. The nanorod density increases from 1×10^8 cm^{-2}, 1×10^9 cm^{-2}, to 3×10^{10} cm^{-2} as the pressure increases from 10 mTorr, 20 mTorr, to 30 mTorr, respectively. The dimension of the GaN nanorods decreases from about 100 nm to the 20–50 nm range as the chamber pressure increases from 10 to 30 mTorr. The result suggests that the chamber pressure seems to play a major role in the formation and controlling of the dimension and density of GaN nanorods. The exact mechanism of the formation of nanostructures is not fully understood yet. However, the creation of nanorods and the variations in the dimension and density 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^{-2} 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.[20] As for the increase in the nanorod density on raising chamber pressure, it could be attributed to the degradation of the ICP dissociation ability at a higher chamber pressure.[21,22] According to refs. 21 and 22, the plasma density is a strong function of the chamber pressure. As the chamber pressure is increased, the mean free path decreases and the collisional frequency increases. This tends to result in changes in both ion energy and plasma density which strongly influence the dissociation of GaN bonding during the ICP etching process.

In summary, we report a novel method of fabricating GaN nanorods of controllable dimension and density from GaN epitaxial film using ICP-RIE. The GaN epitaxial film was grown by MOCVD on a sapphire substrate. Under the fixed Cl_2/Ar flow rate of 10/25 sccm and ICP/bias power of 200/200 W, the GaN nanorods and array were fabricated with a density of 10^8–10^{10} cm^{-2} and dimension of 20–100 nm by varying the chamber pressure from 10 to 30 mTorr. The technique offers one-step, relatively straightforward method for the fabrication of GaN nanostructures of controllable dimension and density. Although the current GaN sample is a p-type GaN on undoped GaN materials, it should be applicable for the fabrication of other types of GaN-based materials and structures including p-n junctions, heterojunctions and laser devices.

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