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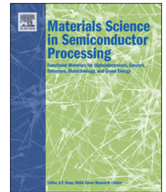

Role of pre-annealing treatment in improving the porosity of gallium nitride on cubic silicon (100) substrate

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A B S T R A C T

This report describes the improved properties of porous gallium nitride (GaN) via pre-annealing treatment using a conventional furnace system. Prior to this work, non-porous GaN samples were annealed at the temperature of 600–1000 °C in order to rise the quality of the samples. From the microscopic, structural and optical measurements, the optimum annealing temperature was found to be 800 °C. Next, the sample that was annealed at the optimum temperature was fabricated into a porous structure by using an electrochemical etching technique. The characteristic of the porous GaN was then investigated by observing its morphology and crystallography properties. For a comparative analysis, a porous GaN sample without the annealing treatment and a porous GaN sample that was then annealed at 800 °C (post-annealing treatment) were also prepared. It was found that the pre-annealing treatment promotes a better quality in porosity of the GaN than other counterparts.

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1. Introduction

A lot of information of gallium nitride (GaN) has been available in the literature since few decades ago and this is due to its fascinating prospectives that allow devices based on it to operate at high temperature, power, and frequency [1]. In general, sapphire (Al_2O_3) is a common substrate for GaN. However, this substrate is quite costly and nitrides based devices grown on it need to undergo complicated fabrication processes. Alternatively, silicon (Si) has been identified as a suitable substrate for GaN [2] and its devices since it is cheap, eases the device fabrication processes and provides a high current injection to the devices [2,3]. Despite these, the GaN layer grown on Si substrate commonly suffers from high defects density and cracks due to large difference in lattice constant and thermal expansion coefficient between both materials [3]. This limits the efforts of using GaN for advanced processes and applications. On the other hand, porous GaN would promote strain-relaxed overgrown layer [4] while ‘drain-out’ the threading dislocations from propagating into the next layer. Therefore, porous GaN has a potential to be used as a template for subsequent nitrides growth. Moreover, porous GaN has a high surface to volume ratio and shows improvement in optical properties [5], which make it a suitable structure for advanced sensor and optical devices.

It is worth noting that one of the critical issues to produce porous GaN nowadays is the surface of the porous structure which tends to damage during the etching process. As a consequence, this leads to unwanted non-stoichiometric conditions; either Ga- or N-rich on the surface. Few research groups such as Vajpeyi et al. [6] and Hartono et al. [4] have demonstrated that thermal annealing can restore the quality of the porous GaN after being damaged by the etching process (so-called post-annealing treatment). However, this kind of treatment may reduce the pore density and distort the pore shape, especially when the sample is annealed at higher temperature [6].

In relation to that, we propose a new technique to improve the quality of porous GaN by annealing the GaN sample prior

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to the etching (so-called pre-annealing treatment). To determine the optimum annealing temperature, the non-porous GaN sample will be annealed at the temperature in the range of 600–1000 °C using a conventional furnace system. Unlike rapid thermal annealing (RTA), a common annealing technique for nitrides materials, the conventional furnace provides a more uniform heating distribution [7]. The sample annealed at the optimum temperature is then etched by electrochemical etching to form porous GaN on the surface. For comparison to pre-annealed GaN, two porous GaN samples are also prepared: 1) without any annealing treatment and 2) with the post-annealing treatment. In the end of this work, the approach that gives a better porosity of the GaN will be proposed.

2. Experimental procedures

An undoped GaN film was grown on Si by RF-plasma assisted molecular beam epitaxy (MBE) using nitrogen plasma operated at 300 W. In this work, we used an undoped Si (100) substrate with the thickness and resistivity ranging from 250 to 300 μm and 1 to 10 Ω cm, respectively. Initially, the surface of the substrate was exposed to Ga flux at a temperature of 850 °C. This was purposely done to remove SiO_2 by forming Ga_2O_3, which was then de-absorbed from the surface [8]. To promote better GaN epitaxial layer, AlN buffer layer was grown at a temperature of 840 °C for 15 min. Subsequently, the GaN growth took place at the same temperature with a thickness of 0.2 μm. After the growth process, the sample was cut into several pieces and annealed at different temperatures of 600 °C, 800 °C and 1000 °C within 5 min under nitrogen ambient.

Next, the annealed GaN samples were characterized in order to investigate the effect of temperature on the crystalline properties of the samples. This subsequently allows the optimum annealing temperature to be determined. The surface morphology of the samples was observed via FE-SEM with accelerating voltage varying from 0.05 kV to 30 kV. The images were magnified in the range of 5000 × to 50,000 ×. Then, the surface roughness of the samples was measured by atomic force microscopy (AFM). This was followed by x-ray diffraction (XRD) measurement to study the crystalline properties of the samples. Besides, photoluminescence (PL) measurement at room temperature allowed us to study the optical behavior of the samples. A HeCd laser source with an emission wavelength λ of 325 nm was used to excite the samples.

The GaN sample annealed at the optimum temperature was fabricated into a porous structure by electrochemical etching. The electrochemical etching setup is illustrated in Fig. 1. In this experiment, a home-made Teflon cell was used to fix the sample as the anode while Pt wire as the cathode. The etching was carried out for 15 min using NaOH electrolyte of 5%. The etching process was conducted at 5 mA with assistance from a 500 W ultra-violet (UV) light for promoting holes at the surface. Note that the distance between the UV light and the electrolyte was kept constant for all porous GaN. For comparison, a porous GaN sample without annealing treatment and a porous GaN with post-annealing treatment were also demonstrated. This is carried out to observe the role of using the pre-annealing treatment to improve the porosity of the GaN, as compared to the other two techniques.

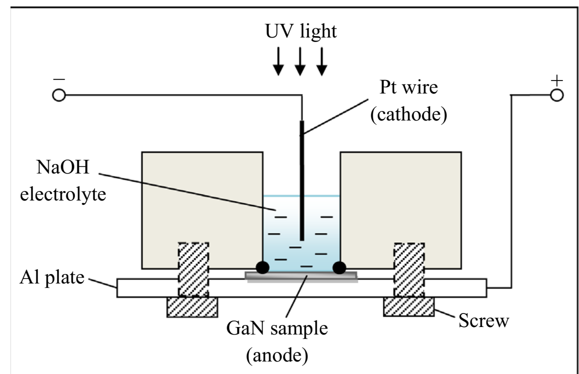

Fig. 1. Schematic diagram of the electrochemical etching experiment to produce porous GaN.

3. Results and discussions

Fig. 2 shows the dependence of the surface roughness (in RMS value) of the samples on the annealing temperature. Overall, the surface roughness decreases with temperature. Further investigation was carried out by FE-SEM measurement. The measurement reveals that the grains size increases by increasing the temperature, which results in smoother surface, similar to what have been observed in AFM measurement. This clearly indicates that the surface morphology has been improved by annealing the sample at higher temperature. When the temperature is increased, the disordered atoms in the GaN layer will re-arrange to form a more well-orderly crystal structure while the threading dislocations were partially removed. The image of the non-annealed sample is also included for comparison.

Fig. 3 shows the XRD data of the non-porous GaN sample. Clearly, the sample was preferably grown in [002] direction. All annealed samples also show the same characteristic (data not shown here). The crystalline quality of all samples was determined by measuring the full width half-maximum (FWHM) from the scans of (002) and (102) XRD rocking curve. The results are shown in Fig. 3(b). It can be seen that increase in the annealing temperature helps to reduce the amount of the threading dislocations and hence raises the crystalline quality to the samples. This is in a good agreement with the results from AFM and FE-SEM measurements. However, it should be noted that the FWHM from the (002) scan slightly increases when the sample was annealed at 1000 °C. This may indicate the initial loss of the Ga atoms from the surface at elevated temperature [9]. As a result, non-stoichiometric III/V ratio has been created on the surface and thus contributes to the broadening of the XRD peak.

PL spectra of all samples are presented in Fig. 4(a). Apparently, a sharp and strong peak at 364 nm (3.40 eV) is observable in all samples. This peak corresponds to the near band-edge related emission in GaN. As shown in Fig. 4(b), the PL peak increases in intensity with temperature but then decreases at 1000 °C. Furthermore, by referring to Fig. 4(a), there is a broad and weak peak at a longer wavelength (~427 nm)

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