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Micro-raman spectroscopy and atomic force microscopy characterization of gallium nitride damaged by accelerated gallium ions

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Gallium nitride on sapphire was characterized using AFM, SEM and micro-Raman spectroscopy after etching by a NOVA 200 FEI Focused Ion Beam (FIB). Various probe beam currents were used at a 30 kV acceleration voltage. The sidewall of the etched area was rougher and the roughness on the surface of the etched area increased when the probe beam current was increased. The intensity of the E_2^2 phonon of micro-Raman spectroscopy decreased when the probe beam current was increased from 10pA, 100 pA, 1 nA to 20 nA. Therefore, it is very important to control the FIB probe current to maximize the etch rate and minimize the damage induced by accelerated Gallium ions.

Key words: Focused Ion Beam, Gallium Nitride, Damage.

Introduction

Gallium nitride (GaN) has been widely researched for high frequency, high power and high breakdown voltage applications [1-5]. GaN-based electronic devices in hostile environments, such as high temperature, proton-irradiation and gamma-irradiation, outperform conventional Silicon- and GaAs-based electronic devices [6-8]. These good electrical and mechanical properties of GaN make it an ideal candidate for advanced sensor systems [9, 10]. However, one of the drawbacks in the GaN material system is the difficulty in both wet-etching and dry-etching. GaN is chemically very stable, mainly due to the strong Ga-N bond strength (8.92 eV/atom) [11]. For dry etching, plasma-based ICP (Inductively Coupled Plasma), MRIE (Magnetron Reactive Ion Etching), ECR (Electron Cyclotron Resonance) and RIE (Reactive ion Etching) techniques have been employed to etch strong GaN materials [12].

Focused-Ion-Beam has been used to prepare TEM (Transmission electron microscopy) samples [13]. The FIB system employs a Ga-ion beam to mill samples. One of the merits of FIB is that it enables direct etching of the sample without photomasks. Also, Pt can be directly deposited on the sample without the photolithography process. Recent FIB systems employ dual beams, which can cut and image the species without rotating the sample back to the original position inside the chamber. The etch rate can be controlled by adjusting the probe current. Since a sample can be damaged by exposure to accelerated Ga-ions even for a short time, it is very important to characterize the damage induced on GaN at various probe currents. AFM (Atomic Force Microscopy) is very helpful in characterizing the roughness of a surface that was damaged by FIB at various probe currents. Also, micro-Raman spectroscopy is useful in characterizing the quality of semiconductor materials. This technique is extremely sensitive to the crystal quality, residual strain, doping concentrations and temperature of semiconductors [14, 15]. The resolution of the micro-Raman scattering technique is typically less than 2 μm. We combined SEM (scanning electron microscope), AFM and micro-Raman Spectroscopy for detailed characterization of GaN damaged by FIB.

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Experimental

GaN (2 μm thick, n ~ 3×10^{17}) was grown by a Metal-Organic Chemical Vapor Deposition technique on an Al_2O_3 substrate. For FIB etching, a NOVA 200 (FEI Company) was used to etch GaN samples. The probe current was increased from 10 pA, 100 pA, 1 nA to 20 nA. The dwell time was 1 μs. The Volume/Dose was 0.15 μm^3/nC and the overlap of X- and Y-position was 50%. The stage was tilted at 52°. Each position was etched for 10 minutes to compare the etch rates at 10 pA, 100 pA, 1 nA and 20 nA. The acceleration voltage was 30 kV. Etch rate and roughness were measured by AFM after each FIB process. The micro-Raman spectroscopy system with a He-Ne laser (632.8 nm wavelength) by Jobin Yvon (LabRam High Resolution) was used to characterize the damage after each FIB etching inside the ring and outside the ring. The E_2^2 high phonon was monitored because it is the most intensive in this back-scattering geometry. The laser power applied to the sample was about 0.64 mW. GaN sample was positioned using a computer-controlled motorized stage. The CCD of the micro-Raman spectroscopy system was cooled with liquid nitrogen.

Results and Discussions

Figure 1 shows SEM pictures of GaN damaged by gallium-ions at various probe currents. Figure 1(A) was taken after FIB etching at a probe current of 10 pA for 10 minutes. Figure 1(D) was taken after FIB etching at a 20 nA probe current for 10 minutes. In Fig. 1(B) and Fig. 1(C), the probe current was 100 pA, and 1 nA, respectively. The morphology of the sidewalls became rougher as the probe current was increased as seen in Fig. 1(A~D). Therefore, the probe current must be controlled to generate the smooth sidewall needed for applications in waveguides, laser diodes and Vertical-Cavity Surface-Emitting Lasers (VCSEL) to minimize the signal loss at the surface. The etch rate also increased when the probe current was increased. There was a strong relationship between the etch rate of GaN and the FIB probe current. The islands seen in Fig. 1(C) and 1(D) were created due to re-deposition of etched materials because the residue could not be removed efficiently at high probe currents.

Figure 2(A) shows an AFM image of a ring-type etched area. Figure 2(B) shows the image at the bottom of the ring. The RMS (Root-Mean-Square) roughness of each ring is given in Table 1. As expected from the SEM images, the surface morphology of the bottom of the etched area was rougher at the higher probe current. Therefore, it is important to optimize the etch rate and

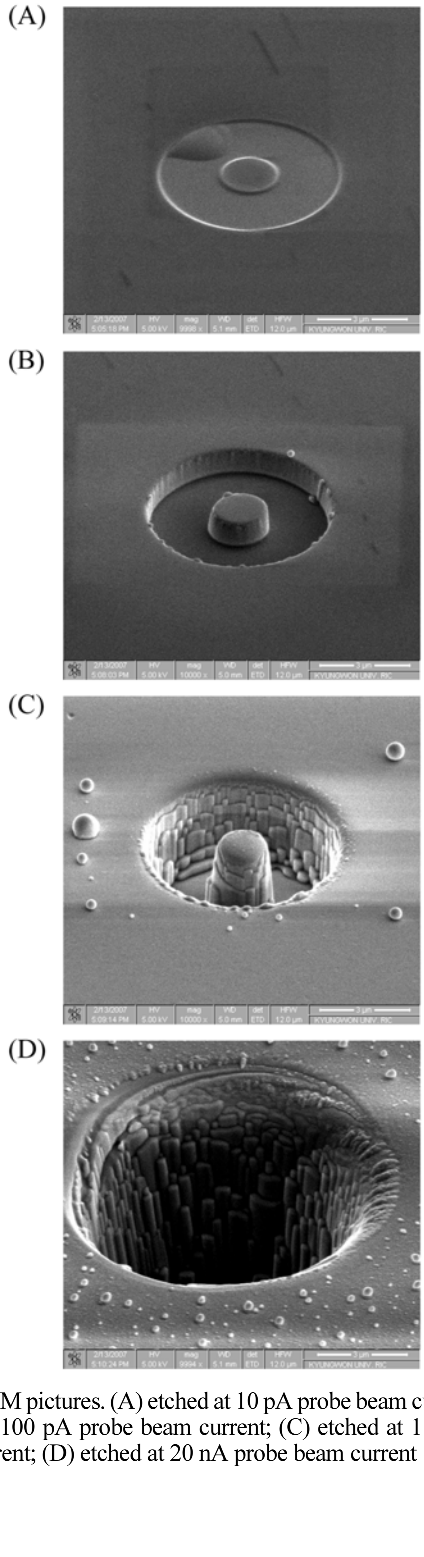

Fig. 1. SEM pictures. (A) etched at 10 pA probe beam current; (B) etched at 100 pA probe beam current; (C) etched at 1 nA probe beam current; (D) etched at 20 nA probe beam current

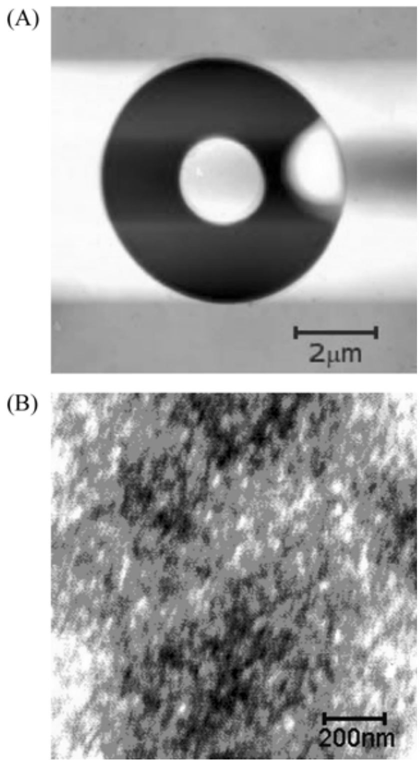

Fig. 2. AFM pictures after etching at 10 pA probe beam current entire area etched area (bottom of the ring).

Table 1.

FIB Probe Currents	10 pA	100 pA	1 nA	20 nA
RMS Roughness	194.6 pm	1.375 nm	11.62 nm	20.31 nm

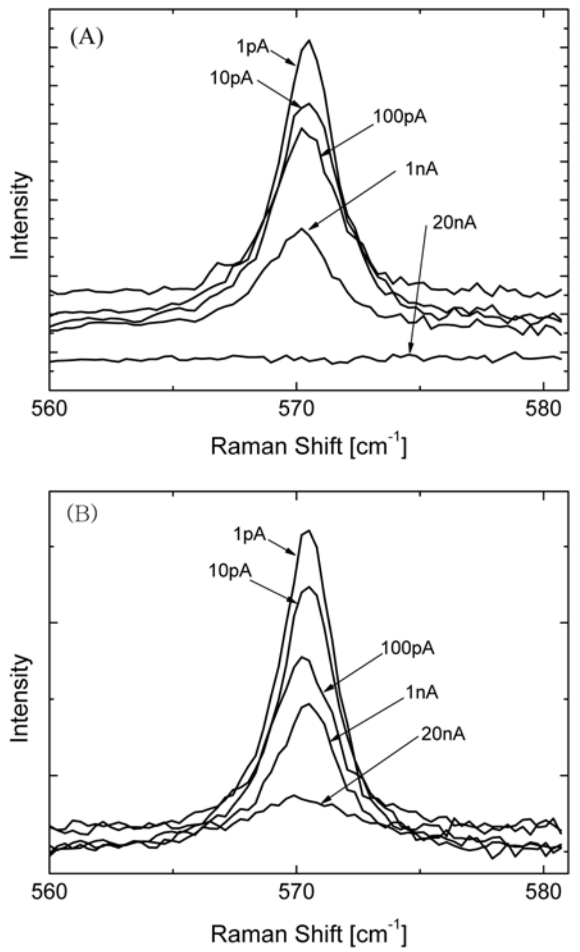

Fig. 3. Micro-Raman spectroscopy data in the etched area (Ring) close to the etched area (Close to ring).

the surface roughness in FIB etching of GaN, as these can result in signal loss in optical devices.

Micro-Raman spectroscopy was employed to characterize the damage to the crystal after each FIB etching. Figure 3(A) shows the intensity of micro-Raman spectroscopy after each FIB etching at the ring. The intensity of micro-Raman Spectroscopy decreased when the FIB probe current was increased. Figure 3(B) shows micro-Raman Spectroscopy data measured close to the ring (close to the FIB-etched area). We found that the signal intensity of micro-Raman spectroscopy also decreased in the vicinity of the FIB-etched area. Future studies will include characterizations of the performance of GaN-based devices when these devices are damaged by FIB etching under various conditions.

Conclusions

MOCVD-grown GaN/sapphire was etched by NOVA 200 Focused-Ion-Beam equipment. The etch rate was larger at higher FIB probe currents. The sidewall was also rougher at higher FIB probe currents. The RMS roughness was monitored using AFM after each FIB etching. The RMS roughness increased when the FIB probe current was increased. The micro-Raman spectroscopy signal intensity decreased in the FIB-etched area and in the vicinity of the FIB-etched area at higher FIB probe currents.

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