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The Variation of Ohmic Contacts and Surface Characteristics on p-GaN Induced by Reactive Ion Etching

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Electron cyclotron resonance (ECR) reactive ion etch (RIE) was used to etch p-type GaN under a chlorine-based plasma. Rapid thermal annealing (RTA) and nitrogen plasma were used as post-RIE treatments to investigate the variation of Ni/Au contacts on p-GaN. RIE deteriorated the contact very much due to the induced nitrogen vacancies and damage on the p-GaN surface. The poor contact was improved by RTA treatment at 700°C for 3 min although the current-voltage curve was still nonlinear. The results from X-ray photoelectron spectroscopy (XPS), atomic force microscopy, and grazing incidence X-ray diffraction measurements indicated that RTA at 700°C could reconstruct the ordered structure from the damaged p-GaN surface. Annealing at 500 or 900°C did not improve the contact due to the high oxygen content of the surface. ECR-N_2 plasma treatment could scarcely improve the contact. XPS and photoluminescence analyses revealed that the nitrogen plasma treatment increased the number of nitrogen vacancies as well as the nitrogen content of a p-GaN surface. These nitrogen atoms did not form tight bonds with GaN, and easily escaped from the surface by annealing.
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GaN-based semiconductors have received much attention because of their superior properties such as wide bandgap, high breakdown field, high electron mobility,[1] and good thermal stability. These properties make GaN a promising material not only for optical devices in the short wavelength range but also for electronic power devices.[2] High-quality ohmic contacts are required in these devices. For n-type GaN, Ti/Al based metallization schemes[3] are widely used with a contact resistance as low as $10^{-6}\ \Omega\ \text{cm}^2$. For p-type GaN, the typical ohmic contact is Ni/Au, which is normally annealed at 500-700°C with a specific contact resistance of 10^{-3} to $10^{-2}\ \Omega\ \text{cm}^2$. Recently, Ho *et al.*[4] reported that the specific contact resistance of Ni/Au could be reduced to $4 \times 10^{-6}\ \Omega\ \text{cm}^2$ after annealing in an oxygen ambient. The reliability[5] and reproducibility of their results must be further verified.

Most studies of metal contacts have focused on the treatment of an as-grown GaN surface. For practical application in heterojunction bipolar transistors, high electron mobility transistors, and other optoelectronic devices, a GaN wafer must be etched to a particular epitaxy layer for metallization. The etching process is normally undertaken in a reactive ion etch (RIE) system with a high density plasma, due to the chemical inertness of GaN. This process would change the structure and stoichiometry of the GaN surface and affect the performance of the following metal contacts. In n-type GaN, the ohmic contact could be improved after an RIE process because the preferential loss of nitrogen renders the surface highly n-type.[6,7] However, Chen *et al.*[8] demonstrated a contrary result that was probably due to the disorder and damaged surface caused by RIE exposure. Some of the RIE-induced damage could be recovered by rapid thermal annealing (RTA).

In this work, we studied the effects of RIE on Ni/Au ohmic contacts of p-type GaN. Electron cyclotron resonance (ECR) was used as the plasma source to reduce the surface damage caused by ion bombardment and to maintain a high etching rate.[9] RTA and N_2 plasma were used as post-RIE treatments to recover the surface disorder and supplement the lost nitrogen, respectively. X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), photoluminescence (PL), and grazing incident X-ray diffraction (GIXD) were utilized to analyze the variation of the surface state.

Experimental

The samples used for this study were p-type GaN grown on sapphire substrates by metallorganic chemical vapor deposition (MOCVD). The grown epitaxial layers consisted of (0001)-oriented unintentionally doped GaN, Si-doped n-GaN, and Mg-doped p-GaN. The thicknesses of these layers were 2, 2, and 0.2 μm, respectively. The hole concentration of the p-type layer was $1 \times 10^{17}\ \text{cm}^{-3}$ with the mobility of $10\ \text{cm}^2\ \text{V}^{-1}\ \text{s}^{-1}$. As shown in Table I, all of the samples were initially cleaned by acetone, HCl (17 vol %), and NH_4OH (5 vol %) in turn. The etched samples were loaded into the ECR-RIE chamber, and processed under a chlorine-based plasma. The etching depth was 40 nm at 1.33×10^{-3} mbar working pressure, 250 W ECR power, 6 standard cubic centimeters per minute (sccm) CH_4, 30 sccm Cl_2, 30 sccm Ar, 140 W cathode radio frequency power, and 120 V direct current bias. Following the RIE process, two different techniques were adopted to improve the final ohmic contact. One involved RTA treatment at temperatures of 500, 700, and 900°C in a nitrogen ambient to recover the damaged surface. The other involved putting the sample under ECR-N_2 plasma for 5 min to supplement the nitrogen atoms preferentially lost from the p-GaN surface due to ion bombardment. The samples were patterned with a design based on the circular transmission line model (CTLM[10]) to measure the electrical characteristics. This pattern was finally transferred to the metal contacts by lift-off technique. Ni/Au (5/5 nm) contacts were deposited by electron beam evaporation and then alloyed at 550°C for 10 min. The current-voltage (I-V) measurement was performed using an HP-4156 analyzer. AFM was employed to measure the roughness of the native and processed surfaces. The variation of the surface composition was observed by XPS. In the PL measurements, the focused beam of a He-Cd 325 nm laser excited the GaN films, which were cooled at 10 K. GIXD were conducted to analyze the near surface structure with the incident angle of 0.5°.

Results and Discussion

Figure 1 shows the I-V characteristics of the Ni/Au contacts on different p-GaN samples. The thickness of p-GaN layers should be adjusted to the same level to compare the samples. Namely, the data points of the native sample were modified to a new curve based on CTLM by considering a p-GaN thickness of 0.16 um rather than 0.2 um. The curve of the native sample demonstrates ohmic behavior, while the other curves of RIE samples display nonlinear and poor contact characteristics. In addition, ECR-N_2 plasma treatments deteriorate the contacts. Only by RTA treatment at 700°C, can the poor property be improved much although it does not become ohmic yet.

The XPS survey spectra show that the p-GaN surface mainly consisted of gallium, nitrogen, oxygen, and carbon impurities. Figure 2 displays the XPS spectra of Ga $2p_{3/2}$, O 1s, and N 1s photoelectrons from the samples. The absolute surface contents were not

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Table I. Samples processed by different treatments, and the results from XPS and AFM measurements. The relative variations of surface nitrogen and oxygen contents can be obtained by taking the native sample as the basis.

Sample no.	Post-RIE treatments	Normalized N/Ga	Normalized O/Ga	AFM roughness (nm)
1	None (native)	1	1	1.1
2	None (RIE)	0.92	3.19	10.5
3	RTA 500°C	0.53	3.49	16.8
4	RTA 700°C	0.70	1.15	81.4
5	RTA 900°C	0.66	4.23	16.2
6	N_2 plasma	1.61	4.06	33.3
7	N_2 plasma + RTA 700°C	0.64	3.00	51.6

acquired from the calculation of peak areas, due to the lack of precise atomic sensitivity data. Accordingly, only the relative contents of nitrogen and oxygen were obtained by taking the native sample for the basis (Table I). The nitrogen content declined slightly after RIE treatment. However, the oxygen content tripled probably because of the RIE-induced Ga dangling bond, which would adsorb oxygen from the ambient even though the sample was carefully stored in the nitrogen cabinet before XPS measurements were made. When the etched samples were RTA-treated at 500°C, the nitrogen content was further reduced because a few of the nitrogen bonds had been destroyed and the surface structure became very loose under the earlier RIE treatment. By annealing at 700°C, the nitrogen and oxygen contents were recovered nearly to the native amounts. This finding implied that the disordered p-GaN surface underwent reconstruction[11] such that improved I-V characteristics could be observed. When the sample was annealed at 900°C, the GaN surface started to decompose and gallium oxide tended to form by the residual oxygen.[12] ECR-N_2 treatment increased the nitrogen content, but these nitrogen atoms did not bond tightly to the GaN surface. As long as the sample was annealed, the nitrogen atoms would escape from the surface. Consequently, ECR-N_2 treatment did not improve the contact.

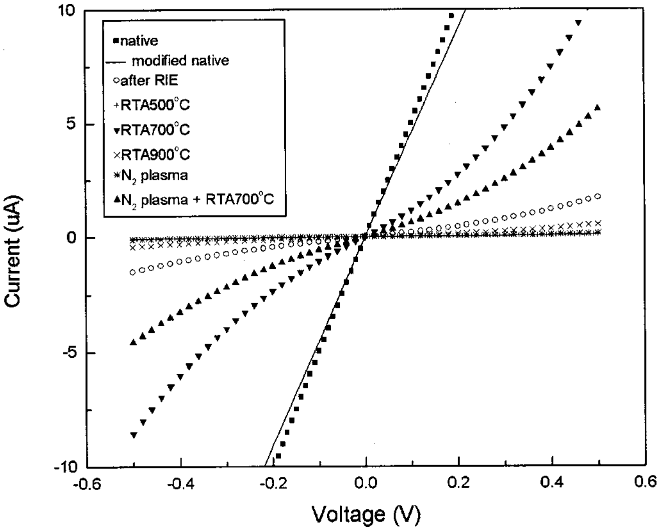

Figure 1. Current-voltage (I-V) characteristics of Ni/Au (5/5 nm) contacts to p-type GaN after alloying in N_2 ambient at 550°C for 10 min. The inner radius of the measured pattern is 200 μm with the spacing of 20 μm to the outer ring. The inset at upper left indicates the treatment conditions.

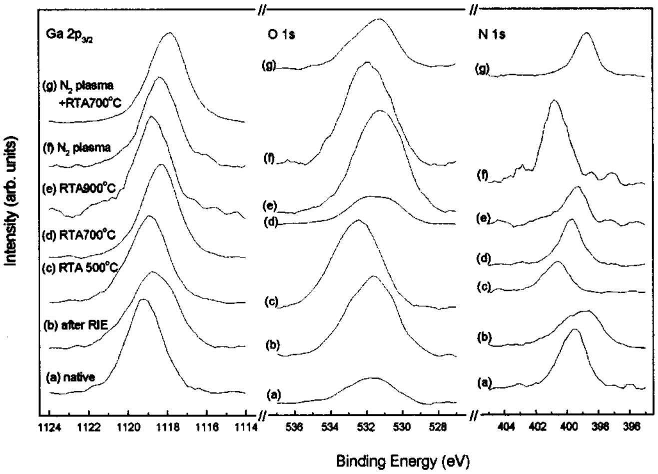

Figure 2. XPS spectra of Ga $2p_{3/2}$, O 1s, and N 1s.

Table I also displays the roughness data from AFM surface scans. The native sample had a very smooth surface with a roughness of 1 nm, while this value became 10 nm after RIE treatment. After RTA treatment at 700°C, the p-GaN surface turned very rough with a value of 81 nm. This roughness variation should relate to the formation of the new surface structure. GIXD measurements were taken on the 700°C-annealed sample and the ones with and without N_2 plasma treatments. The diffraction pattern of Fig. 3 shows that the surface corresponding to RIE and N_2 plasma treatments is disordered. This result is consistent with the one obtained by Chen *et al.*,[8] who utilized the rocking curve method. After annealing at 700°C, the surface might undergo reconstruction and turn to the ordered structure. These peaks located at 35.8, 50.1, and 64.2° represent (002), (102), and (103) faces of the GaN phase, respectively. Trivial geometric calculation shows that the orientation of the surface lattice is very close to the orientation of the underlying epilayer lattice after the RTA treatment at 700°C. For the sample ever treated by N_2 plasma, the surface orientation is different from the epilayer orientation and the diffraction noise is obvious. Namely, the RTA treatment could not recover the surface structure efficiently once the surface had been bombarded by N_2 plasma. This phenomenon should be the main reason why the RTA-treated sample, which had been processed by N_2 plasma, had a poorer I-V characteristic than the sample without N_2 plasma treatment.

As shown in Fig. 4, PL analysis was made on the native, as-etched, and RTA-treated samples. The peak located at 3.479 eV represents the free exciton in GaN. The broad peak around 3 eV corresponds to the Mg-related donor-acceptor pair transition (DAP). The DAP peaks of RIE and N_2 plasma-treated samples are located at 3.0529 and 3.0950 eV which are higher than 2.9983 eV of the native sample. Many studies[13-15] have claimed that the activation energy of Mg ranges from 120 to 510 meV, and the oxygen impurity and

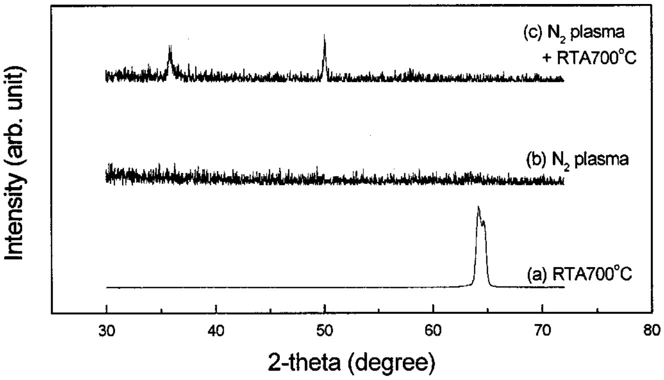

Figure 3. GIXD analysis of the samples treated by (a) RTA 700°C, (b) N_2 plasma, and (c) N_2 plasma + RTA 700°C.

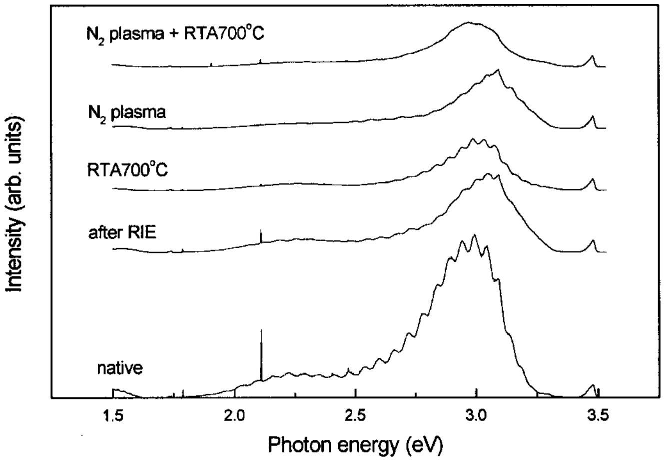

Figure 4. PL spectra of the treated p-type GaN samples at 10 K.

the nitrogen vacancy act as donors with activation energies of 78 and 30 meV, respectively. Therefore, the fact that the peak shifts toward higher energy can be attributed to the higher concentrations of nitrogen vacancies and oxygen impurities on the p-GaN surface after RIE or N_2 plasma treatments. Notably, the higher nitrogen content does not mean fewer nitrogen vacancies certainly. In the case of N_2 plasma treatment, the instilled nitrogen atoms did not form tight bonds with GaN, so the number of nitrogen vacancies still increased. After RTA treatments, the DAP peaks return to the positions of 2.9880 and 2.9710 eV, which were slightly lower than those of the native sample. The damaged surface was reconstructed, but its oxygen content was moderately higher than the one of the native surface.

Conclusion

The RIE process significantly degraded the Ni/Au contact with p-GaN by inducing nitrogen vacancies and damage on the p-GaN surface. The poor contact was improved much by RTA treatment at 700°C for 3 min, although the I-V curve remained nonlinear. The results from XPS, AFM, and GIXD measurements indicated that RTA at 700°C reconstructed the ordered structure from the damaged p-GaN surface. However, ECR-N_2 plasma treatment could scarcely improve the ohmic characteristics. XPS and PL analyses revealed that the N_2 plasma treatment increased the number of nitrogen vacancies and the nitrogen content of the p-GaN surface at the same time. These nitrogen atoms did not form tight bonds with GaN, and easily escaped from the surface by annealing.

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