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Effects of nitrogen deficiency on electronic properties of AlGaN surfaces subjected to thermal and plasma processes

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Abstract
Effects of device processing on chemical and electronic properties of AlGaN surfaces were investigated. The X-ray photoelectron spectroscopy analysis showed serious deterioration such as stoichiometry disorder and nitrogen deficiency (N deficiency) at the AlGaN surfaces processed by high-temperature annealing, H₂-plasma cleaning, dry etching in CH₄/H₂/Ar plasma and deposition of SiO₂. This resulted in high density of surface states at the processed AlGaN surface. Furthermore, the N deficiency introduced a localized deep donor level related to N vacancy (VN) at AlGaN surfaces. Such electronic states governed by a VN related deep donor and surface state continuum can cause strong Fermi level pinning at the AlGaN surface, reduction of the barrier height and excess leakage currents at the AlGaN Schottky interface and serious drain current collapse in AlGaN/GaN heterostructure field effect transistors. The SiNₓ or Al₂O₃-based passivation scheme with a combination of a remote N₂-plasma treatment was found to be effective in suppressing formation of VN-related surface defects at AlGaN surfaces.

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1. Introduction

The fabrication process of semiconductor devices involves various kinds of surface treatments and junction formation steps under high-temperature and/or high-processing energy conditions, which may disorder chemical composition and introduce defects on semiconductor surfaces. Although significant progress has been achieved in the GaN-based high-power/high-frequency electronic devices, additional works are required to solve surface-related problems. In particular, the so-called current collapse effects not only degrade microwave-output performance but also impede reliable operation of the GaN-based power devices [1-5]. To understand and control instability issues in AlGaN/GaN heterostructure field effect transistors (HFETs), it is important to clarify the properties of process-induced defects on AlGaN surfaces. However, experimental confirmation on the electronic states of such "surface defects" has largely been lacking.

The present paper reports effects of device processing on chemical and electronic properties of AlGaN surfaces, especially focusing on nitrogen deficiency caused by high-temperature annealing, plasma cleaning, plasma etching and plasma-assisted deposition processes. It is shown that a nitrogen-vacancy-related near-surface trap plays an important role in electronic states of AlGaN surfaces and operation stability of AlGaN/GaN HFETs.

2. Experiment

2.1 Samples

The Al_{0.25}Ga_{0.75}N/GaN heterostructure sample grown on sapphire substrate by metal organic vapor phase epitaxy (MOVPE) was used in this study. The AlGaN barrier consists of an undoped capping layer (5 nm), a Si-doped layer (15 nm, 4 \times 10^{18} \text{ cm}^{-3}) and an undoped spacer layer (5 nm). Typical values of electron mobility and sheet carrier concentration of the heterostructure samples at room temperature (RT) is 900 cm²/Vs and 1.1 \times 10^{13} \text{ cm}^{-2}, respectively. We also prepared Si-doped n-type Al_{0.20}Ga_{0.80}N layer (0.5 \text{ µm}) grown by MOVPE for metal-insulator-semiconductor (MIS) and Schottky-junction structures. The mobility and carrier concentration at RT were 100 cm²/Vs and 5 \times 10^{17} \text{ cm}^{-3}, respectively. The dislocation density of AlGaN layers ranged from 1 \times 10^{9} - 4 \times 10^{9} \text{ cm}^{-2}.

2.2 Surface processes

To reduce natural oxide, all the AlGaN surfaces were initially treated in NH₄OH solution at 50 °C for 10 min [6, 7]. We used this surface as a reference. Then the samples were processed by thermal annealing, plasma cleaning, dry etching and deposition of SiO₂ and SiNₓ films. The annealing was performed in N₂ at 900 °C for 1 min with and without a SiNₓ cap. The electron-cyclotron-resonance (ECR) excited H₂ plasma with a microwave power of 50 W was used as a dry plasma-cleaning process for AlGaN surfaces at 300 °C. The ECR-assisted reactive ion-beam etching (ECR-RIBE) of the AlGaN surfaces was carried out at RT using a CH₄/H₂/Ar gas system [8]. The deposition of SiO₂ and SiNₓ was performed at 280 °C by ECR-assisted chemical vapor deposition, using SiH₄ and N₂O as precursors for SiO₂, and SiH₄ and N₂ for SiNₓ, respectively. The thickness of the deposited films ranged from 2 to 70 nm.

3. Results and Discussion

3.1 XPS characterization of processed AlGaN surfaces

Figure 1 shows X-ray photoelectron spectroscopy (XPS) core-level spectra of the Al_{0.25}Ga_{0.75}N/GaN heterostructure surfaces after the annealing at 900 °C for 1min in N₂ flow. A monochromated Al Kα x-ray source (hv= 1486.6 eV) was used for the XPS measurements. The present annealing condition is typical for an alloying process for formation of ohmic contacts on AlGaN surfaces. As shown in Fig. 1 (a), the high-temperature anneal produced large amounts of oxide and metal elements in the Al₂p spectrum. A similar feature was observed in the Ga3d core level. This means that a residual O₂ or H₂O in N₂ atmosphere can cause oxidation reaction at the AlGaN surface under the high-temperature condition. From the N1s spectrum, a serious N deficiency was seen at the annealed AlGaN surface, as shown in Fig. 1 (c). In addition, the peak corresponding to the N-O bond [9] was detected at the annealed surface. Such degradation of chemical properties was also observed at
the AlGaN surfaces after dry etching in CH$_4$/H$_2$/Ar plasma, as shown in Fig.2. This process left large amounts of metal clusters on the AlGaN surfaces, resulting in pronounced disorder in the surface stoichiometry.

**Table I** summarizes the XPS analysis on the AlGaN/GaN heterostructure sample surfaces after various treatments. We firstly deconvoluted the measured XPS spectra into some chemical bonding components, as shown in Figs.1 and 2. The Al2p and Ga3d spectra were deconvoluted into the AlGaN bonds, the metallic bonds and oxide bonds, while the N1s peak was separated to the AlGaN bond, Ga AES peaks and additional NH or NO peaks. The composition and V/III ratio were then calculated from the integrated intensities of the deconvoluted XPS components taking account the sensitivity factors.

As mentioned above, the high-temperature anneal caused highly non-stoichiometric surface (V/III ratio of 0.87). The apparent Al composition became much higher than that before annealing. This seems to be due to formation of large amounts of metallic Al cluster and Al oxide, as shown in Fig. 1 (a). As seen in Table I, the degradation of chemical properties was also observed on the AlGaN surfaces after H$_2$-plasma, RIBE and SiO$_2$-deposition processes (samples C-E). A model for possible surface reactions during these processes is shown in Fig.3. During high-temperature annealing, residual impurities such as O$_2$ and/or H$_2$O in N$_2$ can cause oxidation reaction at the AlGaN surface. The N atoms dissociated from Al-N and Ga-N bonds due to the oxidation could react each other or with O atoms to form volatile molecules such as N$_2$ or NO$_x$. This led to the preferential loss of N atoms from the AlGaN surface, leaving a residue of oxides and metal clusters, as shown in Fig.3 (a). The increase of Al composition after the high-temperature annealing indicated that such surface processes also caused a Ga evaporation.

After the deposition of the SiO$_2$ film, we detected an enhancement of the oxide components on the AlGaN surface. In the initial stage of the deposition process, O-related radicals generated in the N$_2$O plasma may react with AlGaN surface, resulting in loss of N atoms and surface process partly similar to that in the high-temperature annealing.

During the H$_2$-plasma treatment, highly active H radicals can react with the AlGaN surface to form volatile NH$_x$ products, as schematically shown in Fig. 3 (b), even at low temperature around 300 °C. This process left the III-metal droplets or clusters and the N depletion at the topmost AlGaN surface [10, 11]. For the RIBE process using CH$_4$/H$_2$/Ar plasma, a similar surface reaction can occur at AlGaN surfaces, producing a heavily metal-rich AlGaN surface [8]. This also led to the pronounced deficiency of N atoms (V/III ratio of 0.56).

### 3.2 Electronic states of processed AlGaN surfaces

To investigate the effects of N deficiency on electronic properties of AlGaN surface, the C-V and I-V measurements were employed to the processed surfaces using metal-insulator-semiconductor (MIS) and Schottky-junction structures.

**Figure 4** shows the interface state density distributions (D$_s$) of the SiN$_x$/n-Al$_{0.2}$Ga$_{0.8}$N structures determined by applying the Terman method to the measured C-V data at room temperature. The SiN$_x$ film was deposited on the processed AlGaN. The measurement frequency was 100 kHz. The sample treated only in NH$_4$OH solution (reference sample) showed a normal continuous D$_s$ distribution [6, 10]. On the other hand, a localized surface level was found at around E$_C$-0.4 eV for the sample with the high-temperature annealed AlGaN surface, as shown in Fig.4. Such a discrete level was also detected in the H$_2$-plasma-processed and RIBE-processed samples where serious N deficiency was found. These results indicated that the N deficiency seems to introduce a nitrogen-vacancy (V$_N$) -related gap level at the processed AlGaN surfaces. Neugebauer and Van de Walle [12], and Boguslawski et al. [13] predicted that a simple V$_N$ defect creates donor levels in the conduction band, acting as a resonant or shallow donor levels in GaN. On the other hand, a possibility of an s-like discrete deep level within bandgap was proposed for a V$_N$ defect in GaN and AlGaN by Yamaguchi and Junnarkar [14].

Next, we have calculated the pinning positions of surface Fermi levels (E$_{FS}$) at the processed AlGaN surfaces based on the experimentally obtained surface state distributions shown in Fig.4. For simply formulating the distribution, we used a surface state model which consists of a U-shaped surface state continuum and a discrete donor level, as shown in **Fig.5** (a). For the U-shaped
distribution, the following formula was used in accordance with the disorder-induced gap state (DIGS) model [15].

\[
D_{SS}(E) = D_{SS \min} \exp\left(\frac{|E - E_{CNL}|}{E_0}\right)^n \quad (1)
\]

where \(D_{SS \min}\) is the minimum surface state density and \(E_{CNL}\) is the charge neutrality level. \(E_{CNL}\) acts as a branch point of surface or interface states since they can be related to the conduction band or the valence band. This represents that the state continuum distributed in energy above \(E_{CNL}\), which has conduction-band character, is acceptor-like charging state, while the one below \(E_{CNL}\), splitting off from the valence band has donor-like charging state [16]. \(E_0\) and \(n\) are the parameters related to distribution shape of a state continuum. The position of \(E_{CNL}\) was set at the midgap of AlGaN. For the discrete near-surface donor level, we employed a donor-type Gaussian peak [12-14] at \(E_c-0.4\) eV with a thermal distribution of 3kT (k: Boltzmann constant, T: absolute temperature). The Al composition was assumed to be 0.3 because it is often used for AlGaN/GaN HFET structures. The position of \(E_{FS} (= E_{CNL} - E_F^0\) at the surface) was calculated applying the charge neutrality condition to the AlGaN surface.

\[
E_{FS} = \left\{ D_{SS}(E)de = D_{SS}^{-} = D_{VN}^+ + D_{dep}^+ \right\}_{E_{CNL}} \quad (2)
\]

where \(D_{SS}^{-}\), \(D_{VN}^+\) and \(D_{dep}^+\) are the ionized acceptor-like surface states, ionized \(V_{N}\)-related level and ionized shallow donors in depletion region of the AlGaN surface. The calculated \(E_{FS}\) position is plotted as a function of the minimum value of surface state continuum, \(D_{SS \min}\), in Fig. 5 (b). The band bending at the \(\text{Al}_{x}\text{Ga}_{1-x}\text{N}\) surface becomes stronger with the density of state continuum. In general, semiconductor surfaces processed with high energy show non-stoichiometric and highly-disordered character, resulting in formation of high-density surface states. This can lead to the strong band bending at the surfaces. The \(E_{FS}\) values ranging from 1.3 eV to 1.7 eV have often been reported for \(\text{Al}_{x}\text{Ga}_{1-x}\text{N}\) surface (x: 0.24 - 0.41) [17-19]. From the calculated results shown in Fig. 5 (b), it is expected that surface states with densities higher than \(1 \times 10^{12} \text{ cm}^{-2} \text{ eV}^{-1}\) are formed on the processed AlGaN surfaces, thereby causing the firm Fermi level pinning.

When a \(V_{N}\)-related discrete donor level is introduced at the surface, the band bending is apparently reduced, as shown in Fig. 5 (b), assuming that the density of state continuum remains unchanged. An extremely high density of the \(V_{N}\)-related donor, in particular, can lower the \(E_{FS}\) position below 1.0 eV. This may be the case for molecular-beam-epitaxy (MBE) grown GaN surfaces [20] where the Ga-rich condition is likely due to the limitation of nitrogen supply via rf-plasma-excited source and highly non-equilibrium growth condition for MBE [21]. For practical processed surface, however, the \(N\) deficiency is accompanied by serious surface disorder described in the previous section. This may also cause an increase in density of the surface state continuum. In such a case, there is a possibility that the \(E_{FS}\) position almost remains unchanged even if the \(V_{N}\)-related defect was created at the processed surfaces.

The surface electronic states obtained here can cause various kinds of operation instabilities in AlGaN/GaN HFETs due to trapping and detrapping of electrons by surface states. In fact, Hasegawa et al. [22] have very recently reported that the electronic states consisting of a U-shaped state continuum and a \(V_{N}\)-related defect level were responsible for drain current collapse in AlGaN/GaN HFETs. According to this model, such surface states could be filled with high-energy electrons injected from the channel during on-state operation with a large drain voltage. This could form a "virtual gate" at the free AlGaN surface near drain edge and partially deplete the two-dimensional electron gas at the AlGaN/GaN interface. In the off-state operation, the strong gate stress may induce electron injection into surface states near gate edge between gate and drain, also causing similar virtual gating effects on the AlGaN surface. Thus, the drain current collapse was induced under both drain stress and gate stress [1-5, 22].

The effects of \(V_{N}\)-related defect level on Schottky properties of AlGaN were investigated. Figure 6 shows typical I-V characteristics of Ni/n-Al\(_{0.22}\)Ga\(_{0.78}\) N Schottky interfaces with and without the H\(_2\)-plasma treatment. The sample without the H\(_2\)-plasma treatment showed the I-V curve similar to
the published data [23-25]. On the other hand, the pronounced reduction of barrier height, $\phi_B$, was observed for the sample having the AlGaN surface exposed to the H$_2$ plasma. The RIBE process resulted in similar reduction of $\phi_B$. The high-density V$_{\text{N}}$-related donors induced during the plasma processes seem to be responsible for the serious thinning of potential barrier at the Schottky interface. In that case, electron transport across the interface is not governed by normal thermionic emission but by thermionic field emission [26], leading to significant reduction of the effective potential barrier against electrons. Such reduction of barrier heights were reported on the GaN and AlGaN surfaces processed by reactive ion etching (RIE) [23, 27] and inductively coupled plasma (ICP)-assisted etching [28, 29]. It is expected that high-density V$_{\text{N}}$-related donor levels are introduced at the surfaces during those etching processes. In addition, Hasegawa et al. [22] recently pointed out that introduction of high-density V$_{\text{N}}$-related donor level caused pronounced thinning of Schottky barrier, resulting in serious excess gate leakage currents in the AlGaN/GaN HFETs due to thermionic field emission transport [26].

3.3 Surface passivation processes for AlGaN surfaces

For reliability improvement of GaN-based electron devices such as AlGaN/GaN HFETs, it is inevitable to establish a suitable passivation structure for AlGaN surfaces especially for suppressing N deficiency.

Firstly, we employed a surface treatment process using N$_2$ plasma for AlGaN. The process utilized a remote ECR plasma where electrons and ions move with the same diffusion coefficient, i.e., the so-called ambipolar diffusion [30]. This means that a nearly neutrality condition can be maintained in the plasma stream. In addition, the ECR microwave discharges can be produced without large potentials. Thus, it is expected that a self-biasing effect is rather weak in the ECR plasma processes. In fact, Matsuoka and Ono [31] reported the self-biasing voltage less than -40 V in the gas pressure range from 1 x 10$^{-2}$ Pa to 1 x 10$^{-1}$ Pa. This fact as well as low ion energies of 10 to several tens of eV in the ECR plasma [31, 32] can suppress an ion-bombardment (sputtering) effect on the material surfaces.

**Figure 7** shows the XPS O1s and C1s spectra of Al$_{0.25}$Ga$_{0.75}$N surface before and after the ECR N$_2$-plasma treatment with a relatively low microwave power of 50 W. The process time and temperature was 1 min and 280 °C, respectively. Significant reduction of O1s and C1s peaks was observed at the AlGaN surface after the N$_2$-plasma treatment, indicating the process is effective in removing natural oxide from AlGaN surfaces. The Al composition and V/III ratio was maintained even after the N$_2$-plasma process. Furthermore, the band bending was reduced at the N$_2$-plasma-treated GaN surfaces [6]. These results indicate that the ECR N$_2$-plasma treatment seems to partially recover or terminate V$_{\text{N}}$-related defects, leading to the reduction of densities of surface states on GaN and AlGaN.

Next we investigated a surface passivation scheme utilizing the ECR N$_2$-plasma treatment and deposition of SiN$_x$ film. The capacitance-voltage analysis showed relatively low interface state density of 1 x 10$^{11}$ cm$^{-2}$eV$^{-1}$ or less at the SiN$_x$/GaN interface [6, 33]. This seems to be main reason that the SiN$_x$-based surface passivation effectively suppress current collapse in GaN MESFETs [34] and AlGaN/GaN HFETs [4, 5, 35-38]. From XPS analysis on the annealed AlGaN surface after removing a SiN$_x$ passivation layer, we found that the SiN$_x$ passivation was also effective in suppressing surface disorder of AlGaN during high-temperature annealing. In contrast to the result shown in Fig.1, no deterioration in chemical configuration was observed at the SiN$_x$-passivated surface even after the high-temperature annealing. In addition, the peak positions and line widths of the core-level spectra remained unchanged. Thus the SiN$_x$ film can act as a protective layer against chemical reaction at AlGaN surfaces during the annealing process.

The SiN$_x$ film has also been used as a gate insulator for AlGaN/GaN HFETs [5, 39]. However, it can be argued that its bandgap (E$_g$ ~ 5 eV) and dielectric constant ($\varepsilon$ ~ 7) are not suitable for utilization as a gate barrier to Al$_x$Ga$_{1-x}$N (e.g., E$_g$ ~ 4.1 eV and $\varepsilon$ ~ 9 for x = 0.3). In fact, we have very recently pointed out that there was a Fowler-Nordheim leakage problem at the SiN$_x$/Al$_{0.3}$Ga$_{0.7}$N interface due to a low value of conduction band offset [11]. As an alternative gate dielectric, we proposed the use of Al$_2$O$_3$ for AlGaN/GaN HFET since it has a wide bandgap (> 7 eV), a high
dielectric constant (~9) and a high breakdown field (>15 MV/cm). The fabricated Al₂O₃ insulated gate HFET showed an excellent gate control of currents with low gate leakage characteristics. In addition, the Al₂O₃-based surface passivation scheme including an N₂-plasma surface treatment achieved the drastic suppression of current collapse in AlGaN/GaN HFETs [11, 40].

4. Summary

The effects of device processing on chemical and electronic properties of AlGaN surfaces were investigated by XPS, C-V and I-V methods. Serious stoichiometry disorder and N deficiency were found at the AlGaN surfaces processed by high-temperature annealing, H₂-plasma cleaning, dry etching in CH₄/H₂/Ar plasma and deposition of SiO₂. The N deficiency could introduce a localized deep donor level related to N vacancy (V₅ₙ) at AlGaN surfaces. The electronic states governed by a V₅ₙ-related deep donor and surface state continuum can cause reduction of the barrier height and excess leakage currents at the AlGaN Schottky interface and serious drain current collapse in AlGaN/GaN heterostructure field effect transistors. The results obtained indicate that it becomes a key to control V₅ₙ-related defects for reliability improvement of GaN-based electronic devices. The SiNₓ- or Al₂O₃-based passivation scheme with a combination of a remote N₂-plasma treatment is promising for suppressing formation of V₅ₙ-related surface defects as well as reducing surface state continuum at AlGaN surfaces.

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References


**Table caption**

Table I  Summary of XPS analysis on the processed Al$_{0.25}$Ga$_{0.75}$N/GaN heterostructure surfaces.

**Figure captions**

Fig.1  XPS core-level spectra of Al$_{0.25}$Ga$_{0.75}$N surface before and after the annealing in N$_2$ at 900 °C for 1 min.

Fig.2  XPS Al2p and Ga3d spectra of Al$_{0.25}$Ga$_{0.75}$N surface after the RIBE process in CH$_4$/H$_2$/Ar plasma.

Fig.3  schematic illustration of possible surface reactions at the AlGaN surface for (a) the high-temperature annealing process and (b) the H$_2$-plasma process.

Fig.4  Interface state density distributions ($D_{it}$) of SiN$_x$/n-Al$_{0.20}$Ga$_{0.80}$N structures. For the annealed sample, the SiN$_x$ film was deposited on the AlGaN surface after the annealing.

Fig.5  (a) An electronic state model and (b) calculated surface Fermi level positions at the Al$_{0.3}$Ga$_{0.7}$N surface.

Fig.6  I-V characteristics of the Ni/n-Al$_{0.20}$Ga$_{0.80}$N junction with and without the H$_2$ plasma treatment at 300 °C for 1 min.

Fig.7  XPS O1s and C1s spectra of Al$_{0.25}$Ga$_{0.75}$N surface before and after the ECR-N$_2$ plasma treatment at 280 °C for 1 min.
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Fig. 2 XPS Al2p and Ga3d spectra of Al_{0.25}Ga_{0.75}N surface after the RIBE process in CH_{4}/H_{2}/Ar plasma.

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