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# Impact of Surface Treatments and Post-Deposition Annealing Upon Interfacial Property of ALD-Al<sub>2</sub>O<sub>3</sub> on a-Plane GaN

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**ABSTRACT** Optimization of interface characteristics between dielectric and non-polar GaN surface is very important and urgent for vertical GaN MOS device whose channel is perpendicular to the conventional c-plane. In this work, the effects of piranha cleaning and N<sub>2</sub> post deposition annealing (PDA) to the interface between atomic-layer-deposited (ALD)-Al<sub>2</sub>O<sub>3</sub> and a-plane GaN samples were comprehensively investigated by X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM) and photo-assisted capacitance-voltage (C-V) measurements. The piranha cleaning and N<sub>2</sub> annealing can improve interface characteristics through the reduction in surface roughness and Ga-O bonds, respectively. Therefore, the frequency dispersion and hysteresis are nearly suppressed with a low interface trap quantity (Q<sub>it</sub>) of  $4.1 \times 10^{11}$  cm<sup>-2</sup> and a low average interface state density (D<sub>it</sub>) of  $2.04 \times 10^{11}$  cm<sup>-2</sup> eV<sup>-1</sup> from photoassisted C-V measurements, showing the great promise of utilizing piranha pretreatment, buffered oxide etch (BOE) dip, and N<sub>2</sub> annealing as an effective route to improve the vertical GaN MOS interface properties.

**INDEX TERMS** GaN, a-plane, Interface trap density, ALD-Al<sub>2</sub>O<sub>3</sub>, photo-assisted C-V measurement.

### I. INTRODUCTION

Due to its wide bandgap, high electron mobility and saturation velocity [1], [2] gallium nitride (GaN) turns out to be a competitive candidate for both power switch and power amplifier applications [3]–[9]. In recent 20 years, both industry and academia have witnessed the significant progress of GaN high-electron-mobility transistors (HEMTs), mainly because of the polarization induced high density, high mobility and high velocity electrons at the c-plane [10]. On the other hand, vertical GaN power device is attracting more attention due to the maturity of the GaN native substrate and the demonstration of various state-of-art high performance power devices [11]–[13]. In particular, vertical metal-oxide-semiconductor field-effect transistors (MOSFETs) with the capability of delivering higher blocking voltage and on-current have shown their great promise to extend the application voltage beyond the general acquiesced 600 V from p-GaN gate-injection-transistors (GITs) [14], [15]. In general, this vertical non-polar a-plane or m-plane channel is perpendicular to the c-plane so that the interface between non-polar GaN and gate dielectric should be carefully investigated to gain a better understanding about the interfacial properties.

There are several studies about the band offset characterizations of atomic layer deposition (ALD)  $Al_2O_3$  on m-plane GaN by X-ray photoelectron spectroscopy (XPS) and the interface property investigations of  $Al_2O_3$ , SiO<sub>2</sub> and high-k dielectrics on non-polar GaN [16]–[18]. However, it seems there are still some frequency dispersion and hysteresis observed and there is no in-depth study on the effects



**FIGURE 1.** (a) Schematic cross-section of fabricated Al<sub>2</sub>O<sub>3</sub>/a-plane GaN MOSCAP. (b) C-V and 1/C<sup>2</sup>-V characteristics of the Al<sub>2</sub>O<sub>3</sub>/a-plane GaN MOSCAP with extract electron concentration of  $2.7 \times 10^{17}$  cm<sup>-3</sup>.

of surface treatment and post-deposition annealing process upon non-polar GaN and dielectrics interfaces, which is the key to reduce the interface state and obtain excellent vertical MOSFET characteristics.

In this work, we report on implementing various surface treatments and post-deposition annealing methodologies to improve the interface quality between the ALD-Al<sub>2</sub>O<sub>3</sub> and a-plane non-polar GaN. In addition, various characterization techniques by XPS, atomic force microscopy (AFM), conductance method and photo-assisted C-V measurements are incorporated to provide guidance for improving the performances of GaN MOS devices.

#### **II. EXPERIMENTAL PROCEDURE**

The four a-plane Si-doped GaN samples were sliced from c-plane GaN, which is grown by hybrid vapor phase epitaxy (HVPE). The samples preparation started with ultrasonic cleaning in acetone and ethanol for 10 minutes and DI water rinse. 15 nm Al<sub>2</sub>O<sub>3</sub> was deposited on all samples by ALD at 300°C with tri-methylaluminum (TMA) and ozone  $(O_3)$ as oxygen source. Five different treatments were adopted on those samples: (A) without treatment; (B) only 5 mins N<sub>2</sub> annealing at 500°C after Al<sub>2</sub>O<sub>3</sub> deposition; (C) only 1 min piranha ( $H_2O_2$ : $H_2SO_4 = 3:7$ ) cleaning before  $Al_2O_3$ deposition; (D) 1 min piranha cleaning and 30s BOE (49% HF:40% NH<sub>4</sub>F = 1:6) dipped before Al<sub>2</sub>O<sub>3</sub> deposition and followed the ALD Al<sub>2</sub>O<sub>3</sub> growth, 5 mins of N<sub>2</sub> annealing at 500°C was further used. Series MOS capacitors (MOSCAPs) were then defined by photo-lithography, depositing Ni/Au (60/120 nm) metal stacks and lift-off processes. The crosssection schematic view of MOSCAP for measurements is shown in Fig. 1(a) which is obtained by connecting one large and one small capacitor in series (the large capacitor can be ignored). The Keithley 4200A-SCS parameter analyzer with heated chuck was used for all the C-V measurements. A deep UV light with wavelength of 365 nm was used as the light source during the photo-assisted C-V measurements.

#### **III. RESULTS AND DISCUSSION**

Fig. 1(b) shows the doping concentration of the a-plane GaN, and it is determined to be  $2.7 \times 10^{17}$  cm<sup>-3</sup> from



FIGURE 2. (a) - (d) C-V (solid line) and G-V curves (dashed line) of  $Al_2O_3/a$ -plane GaN MOSCAPs with different treatments measured at 10 kHz, 100 kHz and 1 MHz. The sweep rate for the C-V hysteresis measurement is 0.1 second per 0.01 V step. (e) Current density-voltage (J-V) characteristics of a-plane GaN MOSCAPs.

the  $d(1/C^2)/dV$  [19] of the C-V measurements at 100 kHz. Fig. 2 shows the f-dependent C-V and G-V hysteresis loop of the Al<sub>2</sub>O<sub>3</sub>/a-plane GaN MOSCAPs, which were measured at room temperature (RT) and the hysteresis was calculated at 1 MHz. The f-dependent C-V measurements start from biasing the diode from depletion to accumulation with 0.01 V as a step and then sweeping back to depletion. Traps located at the interface of the oxide and bulk semiconductor may trap and de-trap electrons at this bi-directional sweeps. This feature leads to a shift of the flat-band voltage (V<sub>FB</sub>) during the C-V measurement. The hysteresis is determined by its maximum value at 1 MHz. Sample A without any treatment demonstrates large dispersion, hysteresis, and high interface trap quantity (Qit). The significant increase in capacitance at lower frequencies is caused by larger leakage as shown in Fig. 2(e). And samples without piranha clean show large conductance at forward bias which also indicates the leakage over the MOSCAPs. The hysteresis of sample B with N2 PDA is 0.23 V and small dispersion is also obtained in sample B. Sample C shows large hysteresis of 0.41 V but low leakage. The treatment combined N2 PDA and piranha cleaning in sample D obtains the smallest hysteresis of 0.13 V, low frequency dispersion and unobservable leakage.

The detectable  $Q_{it}$  can be roughly estimated by using the equation:  $Q_{it} = C_{ox} \times \Delta V/q$ , where  $C_{ox}$  is calculated oxide capacitance of 0.5  $\mu$ F/cm<sup>2</sup> and  $\Delta V$  is  $V_{FB}$  shift,



FIGURE 3. XPS spectra of (a) Ga 3d, (b) Al 2p, (c) O 1s, and (d) N 1s core-level of 2 nm ALD  $Al_2O_3$  on a-plane GaN with or without N<sub>2</sub> PDA, respectively. All binding energies have been charge referenced to the primary carbon C-C peak at 284.8 eV.

respectively [20]. The  $Q_{it}$  can be reduced from  $1.4 \times 10^{12}$  $\text{cm}^{-2}$  to 7.3 × 10<sup>11</sup> cm<sup>-2</sup> by N<sub>2</sub> PDA. As shown in Fig. 3, the bonding states in ALD Al<sub>2</sub>O<sub>3</sub> on a-plane GaN with or without N2 PDA were also studied with XPS. The ratio of Ga-N bond to Ga-O bond at interface increases from 8.13 to 9.77 by 5 mins of N<sub>2</sub> PDA at 500°C. The improvement in Qit may be due to the reduction of Ga-O bond after N2 PDA. The higher oxidation states of Ga reflected in Ga-O bond have been considered as one of the factors that introduce a high interfacial defect density and traps [21], [22]. The Al 2p peak position shifted from 75.5 eV to 74.7 eV after N2 PDA which suggests the increase of Al-N bonds during N<sub>2</sub> PDA [23]. The O 1s peak position shifted from 530.28 eV to 532.3 eV after N2 PDA also suggests the transition from Al-O (or Ga-O) bonds to Al-O-N (or Al-O-N) bonds [21], [24]. The XPS spectra of N 1s core levels is basically not affected by N2 PDA which may be due to the simultaneous increase of the bonds related to Ga-N and Al-N. The minimal  $Q_{it} = 4.1 \times 10^{11} \text{ cm}^{-2}$  of sample D may be attributed to 30 s BOE dipped after piranha cleaning which reduces residual oxides during the piranha treatment process. According to XPS data shown in Fig. 4(a), C-O (the peak around 286 eV may also be the C-N bonds) [25] and C = O [26] bonds are effectively reduced by piranha and BOE cleaning. The root mean square (RMS) surface roughness of a-plane GaN were measured by AFM and shown in Fig. 4(b). The results indicated that piranha cleaning can effectively improve surface RMS from 0.405 to 0.180 nm which contributes to the minimized leakage path [27].



FIGURE 4. (a) XPS C 1s core levels of a-plane GaN samples with piranha cleaning and with both piranha and BOE cleaning, respectively. All binding energies have been charge referenced to the primary carbon C-C peak at 284.8 eV. (b) Atomic force microscopy images of a-plane GaN pretreated with or without piranha cleaning, respectively.



**FIGURE 5.** F-dependent C-V measurements of different MOSCAPs at RT or 150°C: (a) and (e) without treatment, (b) and (f) N<sub>2</sub> PDA, (c) and (g) piranha cleaning, and (d) and (h) both piranha cleaning and N<sub>2</sub> PDA. Extracted G<sub>p</sub>/Aq $\omega$  of (i) sample A and (j) sample D at T = 150°C from AC conductance method. The step of the C-V-f measurement is 0.1 V.

Due to the wide bandgap nature of the GaN, the RT C-V characterization is not sufficient to detect the trap states [28], [29], which locates away from the conduction band ( $E_C$ ) minimal. In order to detect deeper trap states in the bandgap, a high-T C-V measurement is desirable. The C-V curves from 1 kHz to 1 MHz at RT and 150°C are presented in Fig. 5 (a) to (h). Sample A, sample B and sample C exhibit a large dispersion phenomenon,

and C-V characteristics are severely degraded at 150°C. Combining the N<sub>2</sub> PDA, piranha cleaning, and BOE dipped, the excellent interface characteristic has been obtained as shown in Fig. 5(d) and (h). The conventional conductance method was first thought to be able to accurately measure interface traps density (Dit). The parallel conductance  $(G_p/\omega)$  can be calculated from the measured capacitance  $(C_m)$  and conductance  $(G_m)$  by using the equation:  $G_p/\omega =$  $\omega C_{OX}^2 \text{Gm}/[G_m^2 + \omega^2(C_{OX} - C_m)]$  [19], where  $\omega$  is radial frequency. And the energy level of traps  $(E_T)$  with respect to GaN conduction band minimum  $(E_{C})$  of the interface states can be found by the SRH (Shockley-Read-Hall) model using the equation:  $(E_C - E_T) = k_B T \ln(\tau_{it} \sigma \nu_t N_C)$  [19], where  $k_B$  is the Boltzmann constant, T is the temperature,  $\tau_{it}$  is the lifetime of the interface states,  $\sigma$  is the capture cross section of the interface states,  $v_t$  is the thermal velocity, and N<sub>C</sub> is effective density of state in the conduction band. Although the conductance  $(G_p/Aq\omega)$  peaks of sample A can be obtained by the conventional conductance method at 150 °C as shown in Fig. 4(i), but no obvious normalized conductance  $(G_p/Aq\omega)$  peak is observable (shown in Fig. 5 (j)) even at T = 150 °C for sample D. It is a common phenomenon of wide bandgap materials with undetectable trap level due to the large bandgap in semiconductor and deep level of traps [30]-[32].

On the other hand, with the assistance of the ultraviolet (UV) illumination to generate hole and electron pairs, the photo-assisted C-V method can make sure that the traps at different energies in the bandgap can respond to the measurement signal during the C-V measurement, making it possible to evaluate the quality and calculate average Dit of our fabricated MOSCAPs. In order to avoid traps not being completely filled with electrons, the measurements started with biasing gate voltage at accumulation for 10 s. And then C-V measurements from deep depletion to accumulation at f = 1 MHz in dark to achieve dark C-V curve in Fig. 6. The automatic switching test program function of Keithley 4200A-SCS parameter analyzer is used to conduct continuous testing. Next, the gate bias is kept at -10 V, while holding UV light illumination for 10 minutes to force the generated holes toward the Al2O3/GaN interface. The large hole concentration at the interface under illumination enables the interface states to change from allowing electrons to occupy to recombine. After that, turning off the UV light and then the same C-V measurements were performed again to acquire a post-UV C-V curve. The holes generated by UV illumination recombined with those electrons captured in traps make Al2O3/GaN interface donor-like. Compared to acceptor-like interface before UV illumination, a shift of the C-V curves can be observed. This shift can be quantitatively described by  $\Delta V_F$ , while the averaged D<sub>it</sub> can be obtained by the following equation [33]:

$$D_{it} = \frac{C_{ox} \times \Delta V_F}{q \times E_g} \tag{1}$$



**FIGURE 6.** Photo-assisted C-V measurements of MOSCAPs with different treatments: (a) without treatment, (b) N<sub>2</sub> PDA, (c) piranha cleaning and (d) piranha cleaning and N<sub>2</sub> PDA. The insets illustrate the  $(C_{ox}/C)^2$ -1 as a function of voltage to extract the  $\Delta V$  of each sample. The sweep rate for the C-V measurement is 0.1 second per 0.01 V step.

The  $\Delta V_F$  is the shift of  $V_{FB}$ . And  $V_{FB}$  is determined through the extrapolated line of the  $(C_{ox}/C)^2 - 1 \sim V_G$  at  $(C_{ox}/C)^2 - 1 = 0$  [34]. The C-V curves both in dark and after UV irradiated for different treatments have been shown in Fig. 5, and the insets show the  $V_{FB}$  shift. The ideal  $V_{FB}$ of 15 nm Al<sub>2</sub>O<sub>3</sub> on a-plane GaN is 1.1 V simulated by TCAD. The negatively shifting in  $V_{FB}$  for the capacitors with N<sub>2</sub> PDA is due to the decreasing negative effective oxide charge at the Al<sub>2</sub>O<sub>3</sub>/GaN interface [35]. The piranha and BOE cleaning before Al<sub>2</sub>O<sub>3</sub> deposition and N<sub>2</sub> PDA can significantly reduce the  $\Delta V_F$  from 1.11 V in sample A to 0.21 V and achieve minimal averaged interface trap state density to  $2.04 \times 10^{11}$  cm<sup>-2</sup>·eV<sup>-1</sup>.

#### **IV. CONCLUSION**

The interfaces of ALD-Al<sub>2</sub>O<sub>3</sub> on a-plane GaN by various treatments were characterized by XPS, AFM and photo-assisted C-V measurements. The N<sub>2</sub> PDA reduces C-V hysteresis through the reduction of Ga-O bonds, and piranha cleaning suppressed leakage by improvement of surface RMS. Combining piranha and BOE cleaning before Al<sub>2</sub>O<sub>3</sub> deposition and N<sub>2</sub> PDA, a low average D<sub>it</sub> of  $2.04 \times 10^{11}$  cm<sup>-2</sup>·eV<sup>-1</sup> by photo-assisted C-V measurements were achieved, demonstrating its great potential as an effective approach to improve the vertical GaN MOS interface properties.

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