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2015

Duan, T. L., Pan, J. S., & Ang, D. S. (2015). Effect of post-deposition annealing on the interface electronic structures of Al₂O₃-Capped GaN and GaN/AlGa_N/GaN heterostructure. ECS Journal of Solid State Science and Technology, 4(9), P364-P368.
doi:10.1149/2.0081509jss

<https://hdl.handle.net/10356/85429>

<https://doi.org/10.1149/2.0081509jss>

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Effect of Post-Deposition Annealing on the Interface Electronic Structures of Al₂O₃-Capped GaN and GaN/AlGaIn/GaN Heterostructure

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The interface electronic structures of GaN and GaN/AlGaIn/GaN samples, capped with atomic-layer-deposited Al₂O₃ and subjected to varying post-deposition annealing (PDA) temperatures, are investigated by X-ray photoelectron spectroscopy (XPS) and Hall-effect measurement. XPS study shows a decrease in the Ga 3d and N 1s core level binding energy after a high-temperature PDA, implying a decrease of the interface defect density and the associated positive trapped-charge. From Hall-effect measurement, the density and mobility of the two-dimensional electron gas (2DEG) are found to decrease and increase, respectively, after PDA, supporting the XPS results. The XPS data, however, reveal a clear Ga-O signal for all samples and no apparent change in the Ga-O to Ga-N bond ratio for the range of annealing temperatures studied. These findings imply that the observed improvement in 2DEG mobility (and the associated decrease of interface defect density) should be ascribed to the structural change of the disordered GaO_x interfacial layer formed during the atomic-layer-deposition of the Al₂O₃.

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Manuscript submitted June 8, 2015; revised manuscript received July 16, 2015. Published July 29, 2015.

The last decade has seen a growing interest in the gallium nitride (GaN) high-electron-mobility transistor (HEMT). An important advantage of GaN lies in its higher breakdown field, ~5 times more than that of Si and GaAs. As a consequence, larger drain voltages can be applied to the GaN HEMT for improving the output impedance per watt of radio-frequency power, resulting in easier matching and lower loss matching circuits.^{1,2} The larger bandgap of GaN also enables the GaN HEMT to be operated at much higher temperatures as compared to the Si and GaAs counterparts. Further enhancement in the GaN HEMT performance has been achieved by the insertion of a dielectric material between the metal gate and the underlying semiconductor,^{1,3-10} leading to what is commonly known as a metal-insulator-semiconductor or MIS-HEMT. Among the various dielectric materials investigated (e.g. SiO₂,³ Si₃N₄,^{4,5} Al₂O₃,^{1,7,10} HfO₂,⁹ etc.), Al₂O₃ has emerged as one of the most favorable choices, by virtue of its high breakdown field (~10 MV/cm) and bandgap energy (~7–9 eV).^{7,11} The former enables a higher operation voltage while the latter gives rise to larger conduction and valence band offsets that further help suppress the gate leakage current.

Apart from functioning as an insulator that lowers the gate leakage current, the Al₂O₃ is also believed to have another benefit of reducing the native gallium oxide and arsenic oxide (in the case of GaAs), which are the main sources of surface defects.¹³⁻¹⁵ As shown via X-ray photoelectron spectroscopy (XPS), GaAs sample exposed to trimethylaluminum (TMA), the Al precursor used in the atomic-layer deposition (ALD) of Al₂O₃, exhibited a reduction in the As-O and Ga-O spectra, indicating removal of the arsenic and gallium oxides. A similar “TMA-cleaning effect” is believed to also apply to the GaN sample, by virtue of the more negative Gibb’s free energy of Al₂O₃ (−1582.3 kJ/mol) as compared to that for Ga₂O₃ (−998.3 kJ/mol).^{16,17}

On the other hand, there are studies which did not observe the TMA-cleaning effect¹⁸ and the discrepancy has been ascribed to the regrowth of an interfacial oxide upon exposure to atmosphere during ex situ characterization.¹⁹⁻²¹ The regrowth may be particularly pronounced for samples with thin insulator (<1.5 nm) layers. As the formation of the Al₂O₃ insulator in the MIS-HEMT typically involves the use of an oxidizing species (e.g. ozone or H₂O vapor in the case of ALD), there would be inevitable exposure of the GaN surface to the oxidizing species during subsequent Al₂O₃ deposition after a TMA-cleaning step to remove the native Ga oxide was performed. It is then essential to clarify (1) the interface reactions that take place, especially during the initial few cycles where only a thin Al₂O₃ layer is present; (2) the origin of the improved electrical characteristics of MIS-HEMTs employing Al₂O₃ as the gate dielectric.^{10,12}

In this study, we examine the interface electronic structure of Al₂O₃-capped GaN and GaN/AlGaIn/GaN samples subjected to varying post-deposition annealing (PDA) temperatures, using XPS and Hall-effect measurement. The growth of an interfacial GaO_x layer was observed during Al₂O₃ deposition, in accordance to a recent finding.¹¹ More importantly, the GaO_x interfacial layer (IL) has a structural change caused by the annealing at 800°C while the XPS intensity of the Ga-O bonds remains almost unchanged. Hall-effect measurement clearly reveals a lower density and higher mobility of the 2DEG after PDA, indicating that reduction in the interface defect density has occurred. These findings imply that the higher 2DEG mobility observed in Al₂O₃-capped GaN devices is not a direct consequence of the deposited Al₂O₃, but may be the outcome of a thermal induced change of the interfacial GaO_x (e.g. conversion to Ga₂O₃), which in turn lowers the interface defect density. In addition, the binding energy (BE) of the Ga 3d core level XPS spectra corresponding to the Ga-O and Ga-N bonds are observed to decrease with increase in the PDA temperature. This observation could be attributed to the associated reduction in the positive interface trapped charge, which modulates the energy band bending in the space-charge region at the GaN surface^{22,27} and in turn the BE measured with respect to the 0 eV Fermi reference level.

The test samples were fabricated on two different Ga-face GaN-on-Si(111) wafers (A and B) purchased from NTT Advanced Technology Corporation. The Ga-face wafer was chosen in our study because of its smooth surface morphology and high material quality,²⁸ making it a preferred choice for device applications. Wafer A (lot number: N2551-1) consists of a 1-μm Si-doped GaN layer sitting on top of a 0.2-μm intrinsic GaN layer. Wafer B (lot number: N3301-2) has an 18-nm Al_{0.26}Ga_{0.74}N layer sandwiched between a 2-nm top GaN layer and a 1-μm bottom GaN layer. All layers were unintentionally doped n-type. In both wafers, a transition layer separates the top layers from the underlying Si substrate. All wafers were prepared at a temperature over 1000°C and thus the epilayers were expected to be stable under the range of annealing temperatures used in this work. The wafers were initially degreased for 5 minutes in acetone and subsequently in isopropyl alcohol. They were then dipped in dilute hydrofluoric acid (HF:H₂O = 1:20) for ~3 minutes to etch away the pre-existing native oxide, followed by a rinse in flowing de-ionized water. The Al₂O₃ was grown by atomic layer deposition (ALD), with trimethylaluminum (TMA) and H₂O as the metal precursor and oxidant, respectively. The temperature was 250°C. After ALD, the samples were subjected to PDA in N₂ for 30 s at varying temperatures. The Al₂O₃ thickness is ~3 nm for all samples, as measured by the Spectroscopic Ellipsometer VB-250. For wafer A, the sample with as-deposited Al₂O₃ (i.e. without PDA) is denoted as S1, while samples subjected to PDA at 400, 500,

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Figure 1. High-resolution cross-sectional transmission electron micrographs of the $\text{Al}_2\text{O}_3/\text{GaN}$ interface region, and the associated Al and Ga profiles (measured by energy-dispersive X-ray spectroscopy) in the (a) as-deposited (S1); (b) 500°C-annealed (S3) and (c) 800°C-annealed samples. The shaded regions denote the “as-measured” spread of the Ga profile. Samples were protected from the focused-ion-beam cutting by a top Pt layer.

600, and 800°C are denoted as S2, S3, S4 and S5, respectively. For wafer B, the bare sample (i.e. without Al_2O_3 deposition) is denoted as H1 and the as-deposited sample is denoted as H2. Samples subjected to PDA at 500 and 800°C are denoted as H3 and H4, respectively. XPS measurements were carried out using a monochromatic Al $K\alpha$ X-ray source of energy 1486.7 eV. The XPS spectra reported here are referenced to C 1s at 285.0 eV and the spectra are curve-fitted with a combination of Gaussian and Lorentzian line shapes, using a Shirley-type background subtraction.

The physical structure of the as-deposited Al_2O_3 -capped GaN sample (S1) and those annealed at 500°C (S3) and 800°C (S5) were examined by high-resolution transmission electron microscopy (Fig. 1, left panels). The chemical composition of the $\text{Al}_2\text{O}_3/\text{GaN}$ interface structure was examined by XPS measurements. The Ga 3d, N 1s and Al 2p core-level XPS spectra for S1-S5 are depicted in Figs. 2a, 2b and 2c, respectively. The corresponding results for H1-H4 are shown in Figs. 3a–3c. Tables I and II summarize the key parameters (e.g. BE of the spectral peaks). The presence of a Ga-O signal in the Ga 3d spectra of Figs. 2a and 3a should be noted. Since the Ga-O/Ga-N bond ratio is relatively constant over the range of PDA temperatures examined, one may rule out the possibility of Ga out-diffusion into

Figure 2. (a) Ga 3d, (b) N 1s, and (c) Al 2p core-level XPS spectra of samples S1-S5. Gaussian-Lorentz curve fitting was used. Except for the Al 2p peak which is not affected by PDA, the Ga 3d and N 1s peaks can be seen to shift toward lower binding energy with increase in the PDA temperature.

the Al_2O_3 layer (during ALD) and to the surface of the Al_2O_3 (during subsequent PDA) as the origin of the Ga-O signal. The presence of a Ga-O signal therefore implies the existence of a GaO_x layer at the $\text{Al}_2\text{O}_3/\text{GaN}$ interface. To check this inference, energy-dispersive

Table I. Binding energy of XPS spectra peaks, Ga-O/Ga-N bond density ratio and the interface barrier height Φ_b for samples S1-S5. Φ_b is defined as the energy difference between the Fermi level and the conduction band minimum at the surface or interface (cf. Fig. 5b). It may be expressed as $E_g + E_{\text{Ga } 3d}^{\text{diff}} - \text{BE}_{\text{Ga } 3d}$, where E_g is GaN bandgap (3.4 eV⁴³), $\text{BE}_{\text{Ga } 3d}$ is binding energy of the Ga 3d peak, $E_{\text{Ga } 3d}^{\text{diff}}$ is difference between the valence band maximum and $\text{BE}_{\text{Ga } 3d}$ (independent of Al_2O_3 passivation⁴⁴).

Sample no.	PDA temperature (°C)	Binding energy (eV)					
		Ga 3d		N 1s	Al 2p	Bond ratio	Φ_b (eV)
		Ga-N	Ga-O	Ga-N	Al-O		
S1	as-dep	19.5	20.2	397.6	74.3	2.8	2.8
S2	400	19.6	20.2	397.3	74.3	2.7	2.7
S3	500	19.4	19.9	397.1	74.4	2.9	2.9
S4	600	19.4	20.1	397.1	74.3	2.9	2.9
S5	800	19.1	19.8	396.8	74.3	3.2	3.2

Table II. Binding energy of XPS spectra peaks, Ga-O/Ga-N bond density ratio and the interface barrier height Φ_b for samples H1-H4.

Sample no.	PDA temperature (°C)	Binding energy (eV)					Bond ratio	Φ _b (eV)
		Ga 3 <i>d</i>		N 1 <i>s</i>	Al 2 <i>p</i>			
		Ga-N	Ga-O		Al-O	Al-N		
H1	bare	19.7	20.6	397.2		73.5	0.12	2.6
H2	as-dep	19.4	20.0	396.9	74.4	73.5	0.24	2.9
H3	500	19.4	20.1	397.0	74.4	73.6	0.21	2.9
H4	800	19.3	19.9	396.8	74.4	73.5	0.22	3.0

X-ray spectroscopy (EDX) was carried out to map the distribution of Ga and Al across the two materials (Fig. 1, right panels). As expected, intermixing of Al and Ga within the transition region can be observed. However, it should be mentioned that the transition region is artificially broadened to ~ 3 nm, due to the finite electron-beam size of ~ 2 nm. Inside the Al_2O_3 , it is clear that the Ga concentration is below the background detection level. More importantly, the Ga concentration in Al_2O_3 remains below the detection level and the width of the transition region is relatively unchanged in S5 after 800°C PDA, showing no apparent diffusion of Ga during PDA. These observations

support our inference that the Ga-O signal in the Ga 3d spectra is a result of a GaO_x interfacial layer.

Since the formation of GaO_x was inevitable during pre-ALD processing, the XPS measurement results of the as-dep sample (H2) are compared to those for a bare sample (H1) which underwent the same pre-ALD processing steps but without Al_2O_3 deposition. Relative to H1, the higher Ga-O to Ga-N bond density ratio in H2 (Table II) implies that additional GaO_x was formed at the $\text{Al}_2\text{O}_3/\text{GaN}$ interface during ALD.¹¹ In control experiments where TMA-only cycles were applied at the beginning of the ALD process, a similar increase of the Ga-O/Ga-N ratio was observed.¹¹ This implies that the finite delay between the introduction of the sample into the ALD chamber and the start of the ALD process is not the main reason for the GaO_x formation, since the TMA-only cycles would have reduced the GaO_x formed at this stage.^{16,17} The results therefore show that GaO_x growth also happened during ALD as the oxidizing species may penetrate the initially thin Al_2O_3 layer. The tendency for the increase of the Ga-O/Ga-N bond density ratio to slow down, indicating that the GaO_x growth tends toward saturation beyond a certain Al_2O_3 thickness,¹¹ supports this inference. While GaO_x formation may happen during the ALD of Al_2O_3 , it is interesting to point out that the Ga-O to Ga-N bond density ratio remains almost unchanged after PDA (Tables I and II). This shows that there is no apparent reduction of GaO_x by the deposited Al_2O_3 after PDA.

To examine the impact of PDA on the electrical properties of the Al_2O_3 -capped GaN/AlGaIn/GaN heterostructure, Hall-effect measurements were performed on H1-H4 (based on the van der Pauw method²⁵) to extract the density and mobility of the 2DEG. Results are shown in Fig. 4. For the as-deposited sample (H2), the density and mobility of the 2DEG are comparable to those of the bare sample (H1). The observation suggests that the properties of the GaN surface are not significantly altered by the deposited Al_2O_3 but are determined mainly by the first few monolayers of GaO_x formed. Moreover, there seems to be no major differences between the GaO_x/GaN interface formed during the ALD and that on the bare sample, due possibly to the relatively low ALD temperature (250°C). The lack of any substantial impact of the 500°C PDA on the 2DEG density and mobility corroborates this inference. The increase in the 2DEG mobility after the 800°C PDA implies that the surface scattering effect and thus the interface defect density are reduced.^{10,26} The corresponding decrease in the 2DEG density supports this conclusion. It has been widely accepted that the surface defects are a main source for the 2DEG and the density of 2DEG is related to the potential barrier Φ_b .²⁷⁻²⁹ Therefore, the increase in Φ_b (in Tables I and II) and thus the decrease in 2DEG density indicate that the surface defect density is being reduced after PDA.²⁹

Studies on the Al_2O_3 -capped AlGaIn/GaN heterostructure have broadly attributed the improved 2DEG mobility to the surface passivation effect of the deposited Al_2O_3 layer.^{7,10} The detailed mechanisms, however, are not yet clarified. One explanation, based on Gibb's free energy calculation, attributes the passivation effect to the removal of GaO_x by the Al_2O_3 , thus resulting in a lower interface defect density.^{17,30,31} On the other hand, our results show that an interfacial GaO_x layer is formed at the same time as the Al_2O_3 and that the GaO_x layer remains after PDA (for the range of temperature studied). These results imply that it is not Al_2O_3 deposition but the

Figure 3. (a) Ga 3d, (b) N 1s, and (c) Al 2p core-level XPS spectra of samples H1-H4 showing similar behavior as those for samples S1-S5 in Fig. 2.

Figure 4. (a) Concentration and (b) mobility of the two-dimensional electron gas in samples H1-H4, as determined from Hall-effect measurement (300 K).

subsequent thermal treatment that leads to the improved 2DEG mobility. It is reported that a GaO_x layer is formed through a reaction between GaN and oxygen species by oxygen absorption or oxygen penetration through the thin Al_2O_3 layer formed during the initial stage of the ALD process.^{11,32} At the relatively low ALD temperature, a disordered GaO_x layer intermixed with Al_2O_3 is formed.³²⁻³⁴ This disordered GaO_x layer is believed to be the source of interface states.^{32,35,36} A high-temperature annealing process provides the necessary thermal activation that enables the conversion of the disordered GaO_x layer into a more stable form (e.g. Ga_2O_3).^{27,32,36} In conjunction with the charge redistribution in disordered GaO_x that occur during PDA, an improved match between the Ga_2O_3 and the GaN surface may then be achieved.³⁷ It has been shown that the defect density at

the SiO_2/GaN interface could be substantially reduced by the growth of an ultrathin Ga_2O_3 layer prior to the SiO_2 deposition.³⁸ Thus, it is believed that the commonly reported mobility enhancement of the $\text{Al}_2\text{O}_3/\text{GaN}$ HEMT stems mainly from the improved $\text{Ga}_2\text{O}_3/\text{GaN}$ interface after subsequent thermal treatment. Nonetheless, the extent by which structural transformation of the GaO_x layer could happen under a given PDA condition is expected to depend on the type of dielectric used and the associated interface strain. Further study is in progress to understand this.

The impact of PDA on the BE of the core-level spectra peaks is also examined (Tables I and II). No visible impact on the BE of the Ga 3d peak, relative to the as-deposited sample, can be seen for PDA temperature up to $\sim 400^\circ\text{C}$. A slight reduction of the BE is observed at the 500°C PDA and the decrease becomes evidently larger at the 800°C PDA. A similar observation applies to the N 1s peak. Given that PDA has no apparent impact on the BE of the Al 2p peak, the observed BE decrease is mainly due to changes at the $\text{Al}_2\text{O}_3/\text{GaN}$ interface. Similar results are obtained on the GaN/AlGaIn/GaN samples (H3 and H4) shown in Fig. 3. For these samples, the Al 2p spectra can be deconvoluted into two components – other than the usual Al-O component, another component due to the Al-N bonds can be observed (Fig. 3c), since the AlGaIn layer was also probed. The substantially lower Al-N peak of H2 as compared to that of H1 indicates that with the deposition of the 3-nm Al_2O_3 , the XPS probe depth is now confined to the interface region between the 2-nm top GaN layer and the Al_2O_3 . Since PDA also has no apparent effect on the BE of the Al 2p peak, the BE decrease for the Ga 3d and N 1s peaks may thus be ascribed to changes at the GaN/ Al_2O_3 interface.

The change in the BE of the XPS spectra may be explained in terms of the change in the GaN surface band bending, as a result of the decrease in the interface defect density after PDA, as illustrated in Fig. 5. Spontaneous polarization of the Ga-face GaN results in a negative surface bound charge (of density $\sim 10^{13} \text{ cm}^{-2}$)^{22,29,39} which depletes the n-type GaN (or AlGaIn), causing the energy band at the surface to bend upwards. Fig. 5a illustrates how this upward surface band bending, in conjunction with a varying XPS probe depth in GaN, influence the BE obtained from XPS measurement. For a shallower probe depth (long-dash), surface photoelectrons having smaller BEs (measured with respect to the 0 eV Fermi level) made up a greater proportion of the photoelectrons detected during XPS measurement, as compared to the case of a deeper probe depth (dotted). Thus, the measured BE would be smaller when the XPS probe depth is shallower. This explains the

Figure 5. (a) Schematic diagram showing upward bending of the energy band near the surface (line AA') of the Ga-face GaN. The effect of a shallow (long-dash) and deep (dotted) XPS probe depths on the measured XPS BE is illustrated. (b) The positive trapped charge at the $\text{Al}_2\text{O}_3/\text{GaN}$ interface reduces the upward surface band bending in the as-deposited sample (solid) and hence increases the measured XPS BE relative to that of the annealed sample (short-dash) in which the density of the positive interface trapped charge is decreased.

BE decrease of the Ga 3d and N 1s peaks of H2, relative to those of H1,¹¹ since the XPS probe depth in GaN is reduced after the deposition of the 3-nm Al₂O₃. It is essential to note that the upward band bending is lowered by the surface states which charge up positively^{22,27} and thus partially screen the negative surface bound charge (Fig. 5b). The presence of positively charged surface states is commonly revealed through the negative-voltage shifted capacitance-voltage (C-V) curve of the metal/Al₂O₃/GaN structure.^{39,40} After PDA, the density of the interface states is reduced, as can be deduced from the changes in the density and mobility of the 2DEG, as well as the positive-voltage shift of the C-V curve reported by other studies.^{38,41,42} As a consequence, the surface band bending is increased and the effect of the reduced XPS probe depth (after Al₂O₃ deposition) on the XPS BE decrease becomes more prominent, as depicted in Fig. 5a. A higher PDA temperature results in a greater reduction of the interface defect density^{26,38,41} and thus a more apparent decrease of the XPS BE. Changes in the surface band bending after PDA may be manifested through the corresponding changes in the interface barrier height Φ_b (Fig. 5b), as summarized in Tables I and II.

In summary, the interface electronic properties of Al₂O₃-capped GaN and GaN/AlGaN/GaN heterostructure after PDA at varying temperatures are investigated by XPS and Hall-effect measurements. The similar 2DEG density and mobility in the bare and as-deposited samples imply that the deposited Al₂O₃ has no direct impact on the interface electronic properties, which are mainly controlled by the interfacial GaO_x. More importantly, the lack of any significant reduction of the GaO_x after a relatively high annealing temperature (800°C) further implies that the decrease in the interface defect density has mostly resulted from structural changes that happen to this GaO_x layer during thermal treatment, as opposed to its elimination by the deposited Al₂O₃ as commonly perceived. The decrease of the XPS BEs for the Ga 3d and N 1s core levels after PDA may be explained by the increase of surface energy band bending arising from the reduction in the interface positive trapped charge.

Acknowledgments

The authors are grateful to Z. Zhang for his assistance with XPS measurements and M. Lin for his assistance with TEM measurements. This work is partially supported by a Singapore Ministry of Education research grant RG 78/12.

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