Effects of post-deposition annealing ambient on band alignment of RF magnetron-sputtered Y$_2$O$_3$ film on gallium nitride

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Abstract

The effects of different post-deposition annealing ambients (oxygen, argon, forming gas (95% N$_2$ + 5% H$_2$), and nitrogen) on radio frequency magnetron-sputtered yttrium oxide (Y$_2$O$_3$) films on n-type gallium nitride (GaN) substrate were studied in this work. X-ray photoelectron spectroscopy was utilized to extract the bandgap of Y$_2$O$_3$ and interfacial layer as well as establishing the energy band alignment of Y$_2$O$_3$/interfacial layer/GaN structure. Three different structures of energy band alignment were obtained, and the change of band alignment influenced leakage current density-electrical breakdown field characteristics of the samples subjected to different post-deposition annealing ambients. Of these investigated samples, ability of the sample annealed in O$_2$ ambient to withstand the highest electric breakdown field (approximately 6.6 MV/cm) at $10^{-6}$ A/cm$^2$ was related to the largest conduction band offset of interfacial layer/GaN (3.77 eV) and barrier height (3.72 eV).

Keywords: Yttrium oxide, Gallium nitride, Post-deposition annealing, Band alignment, Conduction band offset

Background

Increasing concerns regarding the escalating demand of energy consumption throughout the world has triggered the needs of developing energy-efficient high-power and high-temperature metal-oxide-semiconductor (MOS)-based devices. It has been projected that gallium nitride (GaN) has the potential of conforming to the needs of these MOS-based devices due to its promising properties, which include wide bandgap (3.4 eV), large critical electric field (3 MV/cm), high electron mobility, as well as good thermal conductivity and stability [1-6]. The fabrication of a functional GaN-based MOS device requires a high-quality gate oxide that is capable of resisting a high transverse electric field [7,8]. Native oxide (GaN$_2$O$_3$) of GaN [9-13] and a relatively low-dielectric-constant ($k$) SiN$_2$O$_y$ [2] or SiO$_2$ [14-19] have been successfully grown and deposited, respectively, as gate oxides in GaN-based MOS devices. However, these gate oxides are not the preferred choices. The shortcoming encountered by the former gate is the slow growth gate, high oxidation temperature (>700°C), and high leakage current [12,13] while the latter gate with a relatively low $k$ is unable to withstand the high electric field imposed on GaN [7,20,21]. Thereafter, numerous high-$k$ gate oxides [3,20-28] have been selected for investigation on GaN-based MOS devices. Recent exploration on the employment of radio frequency (RF) magnetron-sputtered Y$_2$O$_3$ gate subjected to post-deposition annealing (PDA) from 200°C to 1,000°C for 30 min in argon ambient has revealed that the Y$_2$O$_3$ gate annealed at 400°C has yielded the best current density-breakdown field ($J-E$) characteristic as well as the lowest effective oxide charge, interface trap density, and total interface trap density [25]. It is noticed that the acquired $J-E$ characteristic for this sample is better than majority of the investigated gate oxide materials [25]. The ability of Y$_2$O$_3$/GaN MOS structure to be driven at a high $E$ and low $J$ is attributed to the fascinating properties possessed by Y$_2$O$_3$, such as high $k$ value ($k = 12$ to 18), large bandgap (approximately 5.5 eV), and large conduction band offset (approximately 1.97 eV) [25,29-31]. Despite that, the presence of oxygen-related defects, changes in compositional homogeneity of Y$_2$O$_3$, and formation of interfacial layer (IL) are of particular concern as...
either of these factors might alter the bandgap of Y₂O₃ and band alignment of Y₂O₃ with respect to the GaN, which would influence the J-E characteristic of the MOS structure. Li et al. has reported previously that J-E characteristic of the MOS structure is dependent on the thickness of IL, wherein interface quality of the atomic layer deposited HfO₂ on Si can be altered via the IL thickness [32]. In order to reduce oxygen-related defects and restore compositional homogeneity of Y₂O₃, it is essential to perform post-deposition annealing on the oxide [33]. Besides, the oxygen content near the Y₂O₃/GaN interface can be regulated by varying the post-deposition annealing ambient and eventually controlling the formation of IL. Therefore, engineering of the bandgap of Y₂O₃ gate and band alignment of Y₂O₃ with GaN through different PDA ambients is of technological importance. In this work, effects of different PDA ambients (oxygen (O₂), argon (Ar) [25], nitrogen (N₂), and forming gas (FG; 95% N₂ + 5% H₂)) at 400°C for 30 min on the Y₂O₃/GaN structure in modifying the bandgap of Y₂O₃ gate and band alignment of Y₂O₃/GaN are presented. A correlation on the bandgap of Y₂O₃ gate and band alignment of Y₂O₃/GaN with regard to the J-E characteristics is also discussed in this paper.

Methods

Prior to the deposition of 60-nm thick Y₂O₃ films on the commercially purchased Si-doped (n-type) GaN epitaxial layers with thickness of 7 μm and doping concentration of 1 to 9 × 10¹⁹ cm⁻³ grown on sapphire substrates, the wafer, which was dicing into smaller pieces, were subjected to RCA cleaning. Subsequently, these samples were loaded into a vacuum chamber of RF magnetron sputtering system (Edwards A500, Edwards, Sanborn, NY, USA). A comprehensive description on the deposition process of Y₂O₃ films has been reported elsewhere [29,30]. Then, PDA was performed in a horizontal tube furnace at 400°C in different ambients (O₂, Ar, N₂, and FG (95%N₂ + 5% H₂)) for 30 min. The heating and cooling rate of approximately 10°C/min was used for the PDA process. After the PDA process, X-ray photoelectron spectroscopy (XPS) measurements were conducted on the samples at the Research Center for Surface and Materials Science, Auckland University, New Zealand, using Kratos Axis Ultra DLD (Shimadzu, Kyoto, Japan) equipped with a monochromatic Al-Kα X-ray source (hv = 1486.69 eV). The spectra of the survey scan were obtained at a low pass energy of 160 eV with an energy resolution of 0.1 eV, and the photoelectron take-off angle was fixed at 0° with respect to the surface normal. Chemical depth profiling was performed by etching the samples using an Ar ion gun operated at 5 keV in order to identify the boundary of Y₂O₃ and interfacial layer between the oxide and GaN. To further determine the bandgap of Y₂O₃ and IL, a detailed scan of O 1s was first performed at the same pass energy of 20 eV with an energy resolution of 1.0 eV. The energy loss spectrum of O 1s would provide the bandgap of Y₂O₃ and IL by taking into consideration the onset of a single particle excitation and band-to-band transition. Kraut’s method was utilized in the extraction of the valence band offset of Y₂O₃ and IL [34,35]. In order to fabricate MOS test structure, the Y₂O₃ film was selectively etched using HF/H₂O (1:1) solution. Next, a blanket of aluminum was evaporated on the Y₂O₃ film using a thermal evaporator (AUTO 306, Edwards). Lastly, an array of Al gate electrode (area = 2.5 × 10⁻³ cm²) was defined using photolithography process. Figure 1 shows the fabricated Al/Y₂O₃/GaN-based MOS test structure. The current–voltage characteristics of the samples were measured using a computer-controlled semiconductor parameter analyzer (Agilent 4156C, Agilent Technologies, Santa Clara, CA, USA).

Results and discussion

Bandgap (E₉) values for Y₂O₃ and IL are extracted from the onset of the respective energy loss spectrum of O 1s core level peaks. The determination of E₉ values for Y₂O₃ and IL is done using a linear extrapolation method, wherein the segment of maximum negative slope is extrapolated to the background level [36]. Figure 2a shows typical O 1s energy loss spectra of Y₂O₃ and IL for the sample annealed in O₂ ambient. The extracted E₉ values are in the range of 4.07 to 4.97 eV and 1.17 to 3.93 eV with a tolerance of 0.05 eV for Y₂O₃ and IL, respectively, for samples annealed in different post-deposition annealing ambients (Figure 3a).

Typical valence band photoelectron spectra of Y₂O₃ and IL for the sample annealed in O₂ ambient are presented in Figure 2b. By means of linear extrapolation method, the valence band edges (Eᵥ) of Y₂O₃ and IL could be determined by extrapolating the maximum negative slope to the minimum horizontal baseline [36]. The acquired valence band offset (ΔEᵥ) values of

![Figure 1 Al/Y₂O₃/GaN MOS test structure.](http://www.nanoscalereslett.com/content/8/1/53)
Y$_2$O$_3$ and IL with respect to GaN substrate are in the range of $-0.04$ to $-1.43$ eV and $-0.21$ to $-3.23$ eV with a tolerance of 0.05 eV, respectively, for all of the investigated samples. The $\Delta E_v$ values of Y$_2$O$_3$/GaN and IL/GaN are shown in Figure 3b as a function of PDA ambient.

The determination of both $E_g$ of Y$_2$O$_3$ and IL as well as $\Delta E_v$ of Y$_2$O$_3$/GaN and IL/GaN enables the calculation of the conduction band offset ($\Delta E_c$) of Y$_2$O$_3$/GaN, IL/GaN, and Y$_2$O$_3$/IL using the following equation: $\Delta E_c$(oxide or IL) = $E_g$(oxide or IL) - $\Delta E_v$(oxide/GaN or IL/GaN) - $E_g$(GaN), where $E_g$(GaN) is 3.40 eV for GaN [37]. The obtained values of $\Delta E_c$(Y$_2$O$_3$/GaN), $\Delta E_c$(IL/GaN), and $\Delta E_c$(Y$_2$O$_3$/IL) for all of the investigated samples are presented in Figure 4. In general, a reduction in $E_g$(Y$_2$O$_3$), $E_g$(IL), $\Delta E_v$(Y$_2$O$_3$/GaN), and $\Delta E_v$(IL/GaN) is observed when different PDA ambients are performed, as indicated by O$_2$ > Ar > FG > N$_2$. The IL has been proven using XPS to be comprised of a mixture of Ga-O, Ga-O-N, Y-O, and Y-N bonding (HJQ and KYC, unpublished work).
and Ga-O-N bonding in the region of IL. Sample subjected to PDA in O₂ ambient attains the largest $E_g(Y_2O_3)$ and $E_g(\text{IL})$ as well as the highest values of $\Delta E_c(Y_2O_3/GaN)$ and $\Delta E_c(\text{IL}/GaN)$. This is related to the supply of O₂ from the gas ambient during PDA, which has contributed to the reduction of oxygen-related defects in the $Y_2O_3$ film and the improvement in the compositional homogeneity of the oxide film. The absence of O₂ supply during PDA in Ar (inert) and reducing ambient, such as FG and N₂, may be the reason contributing to the attainment of lower $E_g(Y_2O_3)$, $E_g(\text{IL})$, $\Delta E_c(Y_2O_3/GaN)$, and $\Delta E_c(\text{IL}/GaN)$ values than the sample annealed in O₂. The presence of N₂ in both FG and N₂ ambient has caused the formation of O₂-deficient $Y_2O_3$ film, wherein N atoms dissociated from N₂ gas may couple with the oxygen-related defects in the $Y_2O_3$ film [30,38]. In addition, the presence of N₂ in both FG and N₂ ambient is also capable of performing nitridation process to diminish the tendency of O₂ dissociated from the $Y_2O_3$ film during PDA to diffuse inward and react with the GaN substrate [30]. Thus, the interfacial layer formed in between the $Y_2O_3$/GaN structure for these samples could be O₂ deficient. Despite the fact that FG and N₂ ambient are capable of providing nitridation and coupling process, the percentage of N₂ in FG ambient (95% N₂) is lower than that in pure N₂. Hence, PDA in N₂ ambient will enhance the nitridation process and coupling of N atoms with the oxygen-related defects in $Y_2O_3$, which leads to the formation of more O₂-deficient $Y_2O_3$ film and IL when compared with the sample annealed in FG ambient. This could be the reason leading to the attainment of the lowest $E_g(Y_2O_3)$, $E_g(\text{IL})$, $\Delta E_c(Y_2O_3/GaN)$, and $\Delta E_c(\text{IL}/GaN)$ values for the sample annealed in N₂ ambient.

A schematic illustration of the energy band alignment of the $Y_2O_3$/IL/GaN structure that had been subjected to different PDA ambients is presented in Figure 5. Three different energy band alignment structures were obtained due to the effect of PDA ambient. It is noticed that the conduction band edge of IL is higher than that of $Y_2O_3$ for the sample annealed in O₂ ambient, but it is lower in samples annealed in Ar, FG, and N₂ ambient. This band alignment shift would influence the leakage current density-electrical field ($J$-$E$) characteristics of the samples (Figure 6). The dielectric breakdown field ($E_B$) is defined as the electric field that causes a leakage current density of $10^{-6}$ A/cm², which is not related to a permanent oxide breakdown but representing a safe value for device operation [39]. Of all the investigated samples, the sample annealed in O₂ ambient demonstrates the lowest $J$ and the highest $E_B$ (approximately 6.6 MV/cm) at $J$ of $10^{-6}$ A/cm². This might be attributed to the attainment of the largest $E_g(Y_2O_3)$ and $E_g(\text{IL})$ as well as

![Figure 5 Schematic diagram showing the energy band alignment of the $Y_2O_3$/IL/GaN system. Energy band alignment of the $Y_2O_3$/IL/GaN system for the sample annealed in (a) oxygen, (b) argon and forming gas, and (c) nitrogen ambient.](http://www.nanoscalereslett.com/content/8/1/53)
the highest values of $\Delta E_c(Y_2O_3/GaN)$ and $\Delta E_c(IL/GaN)$, while for other samples, a deterioration in $J$ and $E_B$ is perceived. The reduction is ranked as $Ar > FG > N_2$.

In order to determine whether the $E_B$ of the investigated samples is either dominated by the breakdown of IL, $Y_2O_3$, or a combination of both $Y_2O_3$ and IL, Fowler-Nordheim (FN) tunneling model is employed to extract the barrier height ($\Phi_B$) of $Y_2O_3$ on GaN. FN tunneling mechanism is defined as tunneling of the injected charged carrier into the conduction band of the $Y_2O_3$ gate oxide via passing through a triangular energy barrier [7,8,30]. This mechanism can be expressed as

$$J_{FN} = A e^B \exp(-B/E),$$

where $A = q^3m_o/8(h\Phi_B)$, $B = 4(2 m^{1/2} \Phi_B^3/(3qh^2))$, $q$ is the electronic charge, $m$ is the effective electron mass in the $Y_2O_3$ ($m = 0.1m_o$, where $m_o$ is the free electron mass), and $h$ is Planck’s constant [8,40]. In order to fit the obtained experimental data with the FN tunneling model, linear curve fitting method has been normally utilized [8,20,41]. Nevertheless, data transformation is needed in this method owing to the limited models that can be presented in linear forms. Hence, nonlinear curve fitting method is employed using Datafit version 9.0.59 to fit the acquired $J$-$E$ results in this work with the FN tunneling model. It is believed that the extracted results using nonlinear curve fitting method is more accurate due to the utilization of actual data and the minimization of data transformation steps required in the linear curve fitting [42,43]. Figure 7 shows the $J$-$E$ results for the samples annealed in $O_2$, Ar, and FG ambient, which fitted well with FN tunneling model. The extracted $\Phi_B$ values of these samples are presented in the Figure 4. The highest $\Phi_B$ value attained by the sample annealed in $O_2$ ambient (3.72 eV) was higher than that of metal-organic decomposed $CeO_2$ (1.13 eV) spin-coated on n-type GaN substrate [20]. No $\Phi_B$ value has been extracted for the sample annealed in $N_2$ ambient due to the low $E_B$ and high $J$ of this sample, wherein the gate oxide breaks down prior to the FN tunneling mechanism.

Table 1 compares the computed $\Delta E_c$ values from the XPS characterization with the $\Phi_B$ value extracted from the FN tunneling model. From this table, it is distinguished that the $E_B$ of the sample annealed in $O_2$ ambient is dominated by the breakdown of IL as the obtained value of $\Phi_B$ from the FN tunneling model is comparable with the value of $\Delta E_c(IL/GaN)$ computed from the XPS measurement. For samples annealed in Ar and FG ambient, the acquisition of $\Phi_B$ value that is comparable to the $\Delta E_c(Y_2O_3/GaN)$ indicates that the $E_B$ of these samples is actually dominated by the breakdown of bulk $Y_2O_3$. Since the leakage current of the sample annealed in $N_2$ ambient is not governed by FN tunneling mechanism, a conclusion in determining whether the $E_B$ of this sample is dominated by the breakdown of IL, $Y_2O_3$, or a combination of both cannot be deduced. Based on the obtained values of $\Delta E_c(Y_2O_3/GaN)$, $\Delta E_c(IL/GaN)$, and $\Delta E_c(Y_2O_3/IL)$, the $E_B$ of this sample is unlikely to be dominated by IL due to the acquisition of a negative $\Delta E_c$ (IL/GaN) value for this sample. Thus, the $E_B$ of this sample is most plausible to be dominated by either $Y_2O_3$ or

| Table 1 Comparison of the obtained $\Delta E_c$ and $\Phi_B$ values |
|------------------|------------------|------------------|------------------|
| XPS: conduction band offset | $J$-$E$ result |
| $Y_2O_3/GaN$ | $IL/GaN$ | $Y_2O_3/IL$ | $\Phi_B$ (eV) |
| $O_2$ | 3.00 | 3.77 | 0.77 | 3.72 |
| $Ar$ | 1.55 | 1.40 | 0.15 | 1.58 |
| $FG$ | 0.99 | 0.68 | 0.31 | 0.92 |
| $N_2$ | 0.70 | $-2.03$ | 2.73 | * |

*Not influenced by FN tunneling. Therefore, barrier height is not extracted from the FN tunneling model.
a combination of Y2O3 and IL. However, the attainment of $\Delta E_f(Y_2O_3/IL)$ value which is larger than that of $\Delta E_f(Y_2O_3/GaN)$ value obtained for the samples annealed in Ar and FG ambient eliminates the latter possibility. The reason behind it is if the $E_B$ of the sample annealed in N2 ambient is dominated by the combination of Y2O3 and IL, this sample should be able to sustain a higher $E_B$ and a lower $J$ than the samples annealed in Ar and FG ambient. Therefore, the $E_B$ of the sample annealed in N2 ambient is most likely dominated by the breakdown of bulk Y2O3.

Conclusions
In conclusion, three different energy band alignment models of Y2O3/interfacial layer/GaN structure subjected to post-deposition annealing at 400°C in different ambient conditions have been established using X-ray photoelectron spectroscopy. It was proven that the dielectric breakdown field ($E_B$) of the sample annealed in O2 ambient was dominated by the breakdown of IL, while the $E_B$ of the samples annealed in Ar, FG, and N2 ambient was dominated by the breakdown of bulk Y2O3. The sample annealed in O2 ambient demonstrated the best leakage current density-breakdown field due to the attainment of the largest bandgap, the largest conduction band offset, and the highest barrier height value.

Competing interests
The authors declare that they have no competing interests.

Authors’ contributions
HJQ carried out all of the experimental work, data analysis of the obtained experimental results, and drafting of the manuscript. KYC had played a vital role in assisting HJQ in the experimental work and data analysis as well as in revising and approving the submission of the final manuscript for publication. Both authors read and approved the final manuscript.

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