Effect of Deposition Method on Valence Band Offsets of SiO₂ and Al₂O₃ on (Al₀.14Ga₀.86)₂O₃

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There are often variations in reported valence band offsets for dielectrics on semiconductors and some of the reasons documented include metal cation contamination, interfacial disorder, variations in dielectric composition, thermal conditions, strain, and surface termination effects. In this paper we show that there are differences of up to 1 eV in band alignments for the common gate dielectrics SiO₂ and Al₂O₃ on single crystal (Al₀.14Ga₀.86)₂O₃, depending on whether they are deposited by sputtering or Atomic Layer Deposition. In the case of Al₂O₃, this changed the band alignment from nested (type I) to staggered gap (type II). The valence band offset at each heterointerface was measured using X-Ray Photoelectron Spectroscopy and was determined to be −0.85 ± 0.15 eV for sputtered Al₂O₃ and 0.23 ± 0.04 eV for ALD Al₂O₃ on β(Al₀.14Ga₀.86)₂O₃, while for SiO₂ it was 0.6 ± 0.10 eV for sputtered and 1.6 ± 0.25 eV for ELD. These results are consistent with recent results showing that the surface of Ga₂O₃ and related alloys are susceptible to severe changes during exposure to energetic ion environments.

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The β-(Al₁Ga₁₋x)₂O₃/Ga₂O₃ heterostructure is attracting attention for field-effect transistors (FETs) that have shown impressive and improving performance. Due to the wide range of options for Ga₂O₃ based electronics, which is a welcome addition to potential ultra-wide bandgap semiconductors with power figure-of-merits well beyond SiC and GaN, the absence of p-type doping for Ga₂O₃ and its alloys means only majority carrier devices are possible and the availability of (Al₁Ga₁₋x)₂O₃/Ga₂O₃ heterostructures for scaled field effect transistors is important if radiofrequency applications are to be addressed by Ga₂O₃. The options for gate dielectrics are limited in the case of (Al₁Ga₁₋x)₂O₃ (referred to here in short as AGO), which exhibits a bandgap of around 5 eV and must utilize dielectric materials with a bandgap of 7 eV or higher to have adequate offsets in both the conduction and valence bands. Additionally, the gate dielectric must also be able to be deposited without disrupting the oxide surface. The two most common gate dielectrics in general for Ga₂O₃ and specifically for β-(Al₁Ga₁₋x)₂O₃/Ga₂O₃ have been Al₂O₃ and SiO₂. Both of these are thermodynamically stable on the AGO. We have previously noted on Ga₂O₃ that the band alignment for Al₂O₃ was different for deposition by Atomic Layer Deposition (ALD) relative to sputtering. The valence band offsets on Ga₂O₃ were 0.07 eV ± 0.04 eV for ALD relative to sputtering. The valence band offsets for ALD Al₂O₃ on β-(Al₀.14Ga₀.86)₂O₃, while for SiO₂ it was 0.6 ± 0.10 eV for sputtered and 1.6 ± 0.25 eV for ELD. These results are consistent with recent results showing that the surface of Ga₂O₃ and related alloys are susceptible to severe changes during exposure to energetic ion environments.

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The β-(Al₁Ga₁₋x)₂O₃/Ga₂O₃ heterostructure is attracting attention for field-effect transistors (FETs) that have shown impressive and improving performance. The formation of a two-dimensional electron gas enhances the transport properties, with advantages in device performance. Due to miscibility issues, the value of x is typically limited to the range 0.12-0.17. These characteristics, together with the necessity of having a wide bandgap semiconductor, have led to the development of potential high performance FETs. The options for Ga₂O₃ based electronics, which is a welcome addition to potential ultra-wide bandgap semiconductors with power figure-of-merits well beyond SiC and GaN, the absence of p-type doping for Ga₂O₃ and its alloys means only majority carrier devices are possible and the availability of (Al₁Ga₁₋x)₂O₃/Ga₂O₃ heterostructures for scaled field effect transistors is important if radiofrequency applications are to be addressed by Ga₂O₃. The options for gate dielectrics are limited in the case of (Al₁Ga₁₋x)₂O₃ (referred to here in short as AGO), which exhibits a bandgap of around 5 eV and must utilize dielectric materials with a bandgap of 7 eV or higher to have adequate offsets in both the conduction and valence bands. Additionally, the gate dielectric must also be able to be deposited without disrupting the oxide surface. The two most common gate dielectrics in general for Ga₂O₃ and specifically for β-(Al₁Ga₁₋x)₂O₃/Ga₂O₃ have been Al₂O₃ and SiO₂. Both of these are thermodynamically stable on the AGO. We have previously noted on Ga₂O₃ that the band alignment for Al₂O₃ was different for deposition by Atomic Layer Deposition (ALD) relative to sputtering. The valence band offsets on Ga₂O₃ were 0.07 eV ± 0.04 eV for ALD relative to sputtering. The valence band offsets for ALD Al₂O₃ on β-(Al₀.14Ga₀.86)₂O₃, while for SiO₂ it was 0.6 ± 0.10 eV for sputtered and 1.6 ± 0.25 eV for ELD. These results are consistent with recent results showing that the surface of Ga₂O₃ and related alloys are susceptible to severe changes during exposure to energetic ion environments.

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Figure 1. XPS survey scans of thick sputtered SiO$_2$ and Al$_2$O$_3$, 1.5 nm sputtered SiO$_2$ and Al$_2$O$_3$ on (Al$_{0.14}$Ga$_{0.86}$)$_2$O$_3$, and a (Al$_{0.14}$Ga$_{0.86}$)$_2$O$_3$ reference sample. The intensity is in arbitrary units (a.u.).

Charge compensation was performed using an electron flood gun and ion beam simultaneously. The adventitious carbon (C-C) line in the C 1s spectra at 284.8 eV was used for charge correction. Since only the relative energy position in each sample is needed to determine the valence band offsets, the absolute energy calibration for a sample has no effect on the ultimate result. The samples and electron analyzers were electrically grounded to provide a common reference Fermi level. Differential charging was not observed with this use of an electron gun. Reflection electron energy loss spectroscopy (REELS) was used to obtain the SiO$_2$ and Al$_2$O$_3$ bandgaps, using a 1 kV electron beam and hemispherical electron analyzer. The bandgap of the AGO was obtained from XPS energy loss measurements of the O1S peak. REELS could have been used for the AGO bandgap determination, but we have found that it is more accurate for the wider bandgap layers, like the dielectrics used here.

Results and Discussion

All of the XPS survey scans from the different structures and materials are shown in Figure 1, including the thick dielectrics of sputtered (200 nm) SiO$_2$ and Al$_2$O$_3$, 1.5 nm sputtered SiO$_2$ and Al$_2$O$_3$ on β-AGO, and an AGO reference sample. To the detection limit of XPS, there were no metallic contaminants in the films whose oxides might lower the overall bandgap of the dielectrics and affect the band alignment. There are numerous studies that the presence of surface defects can strongly influence band alignment.

The valence band maximum (VBM) for the dielectrics and the AGO were obtained in standard fashion using linear fitting of the leading edge of the valence band. These XPS spectra of core levels to valence band maximum (VBM) are shown in Figure 2 for (a) reference (Al$_{0.14}$Ga$_{0.86}$)$_2$O$_3$, (b) thick film sputtered Al$_2$O$_3$ compared to thick film ALD Al$_2$O$_3$, and (c) thick film sputtered SiO$_2$ compared to thick film ALD SiO$_2$. The VBM was 3.0 ± 0.2 eV for β-(Al$_{0.14}$Ga$_{0.86}$)$_2$O$_3$, 2.35 ± 0.3 eV and 3.25 ± 0.4 eV, respectively, for the sputtered and ALD Al$_2$O$_3$, and 4.80 ± 0.5 eV for both sputtered and ALD SiO$_2$. Thus, there is substantial difference between Al$_2$p-VBM for sputter vs PEALD Al$_2$O$_3$. However, there is no difference between Si$_2$p-VBM for sputter vs PEALD SiO$_2$. The former could easily account for the different band alignments we find below, but not for the SiO$_2$/AGO interface. The REELS data in Figure 3 showed no difference in bandgap for both types of deposition method for both Al$_2$O$_3$ and SiO$_2$ with

### Table 1. Summary of measured core levels in these experiments (eV). Error ranges are ~15% in each entry. The referenced core levels are Si$_2$p and Al$_2$p for the SiO$_2$ and Al$_2$O$_3$, respectively.

| Core Level | Reference (Al$_{0.14}$Ga$_{0.86}$)$_2$O$_3$ | Reference Dielectric | Thin Dielectric on (AlGa)$_2$O$_3$ |
|------------|----------------------------------|---------------------|----------------------------------|
| Core Level | VBM | Core Level Peak | Core - VBM | Film (Core) | VBM | Core Level Peak | Core - VBM | Δ Core Level | Valence Band Offset |
| Ga$_2$p/2  | 3.00 | 1117.60 | 1114.60 | Sputt. Al$_2$O$_3$ | 2.58 | 74.33 | 74.32 | 1043.7 | −0.85 |
| ALD Al$_2$O$_3$ | 3.25 | 103.5 | 103.4 | Sputt. SiO$_2$ | 4.80 | 103.5 | 98.70 | 1015.3 | 0.6 |
| ALD SiO$_2$ | 4.80 | 103.4 | 98.60 | ALD Al$_2$O$_3$ | 1014.4 | 1.6 |
values of 6.9 ± 0.4 eV for the former and 8.7 ± 0.5 eV for the latter, respectively. The bandgap of the AGO was found to be 5.0 ± 0.3 eV from the onset of the plasmon loss feature in the O 1s photoemission spectrum. The difference in bandgaps between SiO2 (Al2O3) and AGO is therefore 3.7 (1.9) eV.

We used the standard method due to Kraut to obtain the valence band offsets by XPS measurement of the shift of the core levels from the individual materials when these dielectrics and AGO form the respective heterojunctions. This method measures the energy difference between a core level and the VBM for both the single layer dielectric and semiconductor (AGO in this case). The separation between the reference core levels can be translated directly into a value for the valence band offset (VBO) using the previously measured single layer sample core-level to valence band maximum (VBM) energies:

\[ \Delta E_V = (E_{core}^1 - E_{VBM}^1) - (E_{core}^2 - E_{VBM}^2) = (E_{core}^1 - E_{core}^2) \]

The primary source of uncertainty in XPS measurements of band offsets arises from determining the position of the valence band maximum. The binding energy for the core level peaks were taken as the energy corresponding to their maximum intensity. The VBM values were determined by linear extrapolation of the leading edge to the baseline of the valence band spectra. A root sum square relationship was used to combine the uncertainties in the different binding energies to determine the uncertainty of calculated results.35 High resolution XPS spectra of the VBM-core delta region are shown in Figure 4 (a) for the β-(Al0.14Ga0.86)2O3, sputtered and ALD Al2O3 (b) and sputtered and ALD SiO2 samples (c). Figure 5 shows the XPS spectra for the β-(Al0.14Ga0.86)2O3 to (a) SiO2 and (b) Al2O3 core delta regions of the heterostructure samples for both types of deposition technique. These values are summarized in Table 1 and were used to calculate ΔEV for the different structures used in this study. What is immediately noticeable is the shift of ∼1 eV in the valence band offsets between sputtered and ALD films for both dielectrics. Previous reports have shown the presence of interface trap states or Fermi level pinning in ALD SiO2 and Al2O3/Ga2O3 structures,35–40 but there have not been comparable measurements on capacitors with sputtered dielectrics, so there is no available data on differences in the two types of surfaces. As we discussed earlier, sputtering is more prone to creating interfacial disorder and also have metallic contamination that affects the bandgap of the dielectric, although the latter effect is not present in our samples. As pointed out earlier, difference between AL2P-VBM for sputter vs PEALD Al2O3 accounts for the band alignment difference in this system, but not for the SiO2/AGO interface. One difference in the sputter deposition of the two types of films are the lower deposition rates for Al2O3 (by about a factor of two in our system) and thus the AGO surface is likely subject to more ion bombardment during the initial stages of dielectric deposition with a higher density of surface states created. We have tried to vary the deposition rate in the case of Al2O3, but the threshold power for obtaining any deposition is high enough that we cannot get an adequate range to explore this in detail.

The top of Figure 6 shows the band alignment of the Al2O3/β-(Al0.14Ga0.86)2O3 heterostructures. For the sputtered films, the alignment is type II, staggered gap with a valence band offset of −0.85 eV and conduction band offset of 2.75 eV. For comparison, the ALD deposited Al2O3 has type I alignment with valence band offset of 0.23 ± 0.04 eV and conduction band offset of 1.67 ± 0.30 eV. The SiO2/β-(Al0.14Ga0.86)2O3 remained type I for both types of dielectric deposition, with valence band offsets of 0.6 ± 0.10 eV (1.6 ± 0.30 eV) for sputtered (ALD) and corresponding conduction band offsets of 3.1 ± 0.40 eV for sputtered and 2.1 ± 0.40 eV for ALD films on the AGO. Since we kept the sample preparation and measurement conditions the same in all cases, these differences arise only from the deposition method for the dielectric.

Conclusions

In summary, XPS was used to measure the valence band offsets of SiO2/AGO and Al2O3/AGO heterojunctions in which the dielectrics were deposited by either sputtering or ALD. There were differences of −1 eV in valence band offsets measured directly by XPS between the sputtered and ALD films on AGO. These differences represent a major consideration when designing device fabrication sequences. While all the conduction band offsets obtained would provide excellent electron confinement, the hole confinement would be non-existent in the case of sputtered Al2O3 and ineffective for ALD films of either SiO2 or Al2O3 on this composition of AGO. It has been previously
Figure 4. High resolution XPS spectra for the vacuum-core delta regions of (a) \((\text{Al}_{0.14}\text{Ga}_{0.86})_2\text{O}_3\), (b) sputtered \(\text{Al}_2\text{O}_3\) compared to ALD \(\text{Al}_2\text{O}_3\), and (c) sputtered \(\text{SiO}_2\) compared to ALD \(\text{SiO}_2\). The intensity is in arbitrary units (a.u.).

Figure 5. High resolution XPS spectra for the \((\text{Al}_{0.14}\text{Ga}_{0.86})_2\text{O}_3\) to (a) \(\text{Al}_2\text{O}_3\) and (b) \(\text{SiO}_2\) core delta regions. The intensity is in arbitrary units (a.u.).

reported that sputtered films may contain metallic contaminants and interfacial disorder due to sputter-induced damage, and may have Fermi level pinning.\(^{34-36}\) Contamination is not an issue in our case, which suggests the main factor influencing the variation in band offset are interfacial defects such as metal or oxygen vacancies. Previous literature also demonstrated that variations of more than 1 eV difference in band alignment depending on interface preparation due to high defect concentration in the material along with the cation effect.\(^{24,34-36}\) There are few studies of band alignment and interfaces of AGO with gate dielectrics, so this speculation for the differences is not definitive. We do plan to try annealing experiments to see if the defects can be annealed in the sputtered \(\text{Al}_2\text{O}_3/\text{AGO}\) and remove the difference in band alignments. These results are an example of how ion-induced changes in the surface of \(\text{Ga}_2\text{O}_3\) are important.

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Figure 6. Band diagrams for (a) sputtered Al$_2$O$_3$ compared to ALD Al$_2$O$_3$ and (b) sputtered SiO$_2$ compared to ALD SiO$_2$ on (Al$_{0.14}$Ga$_{0.86}$)$_2$O$_3$. 
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