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Band alignment at AlN/Si (111) and (001) interfaces

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To advance the development of III-V nitride on silicon heterostructure semiconductor devices, we have utilized *in-situ* x-ray photoelectron spectroscopy (XPS) to investigate the chemistry and valence band offset (VBO) at interfaces formed by gas source molecular beam epitaxy of AlN on Si (001) and (111) substrates. For the range of growth temperatures (600–1050 °C) and Al pre-exposures (1–15 min) explored, XPS showed the formation of Si-N bonding at the AlN/Si interface in all cases. The AlN/Si VBO was determined to be -3.5 ± 0.3 eV and independent of the Si orientation and degree of interfacial Si-N bond formation. The corresponding AlN/Si conduction band offset (CBO) was calculated to be 1.6 ± 0.3 eV based on the measured VBO and band gap for wurtzite AlN. Utilizing these results, prior reports for the GaN/AlN band alignment, and transitive and commutative rules for VBOs, the VBO and CBO at the GaN/Si interface were determined to be -2.7 ± 0.3 and -0.4 ± 0.3 eV, respectively. © *2015 AIP Publishing LLC*.
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I. INTRODUCTION

Aluminum nitride (AlN) is a wide band gap III-V nitride (III-N) semiconductor[1] and insulating dielectric[2] that exhibits many outstanding properties including excellent chemical stability,[3] low coefficient of thermal expansion (CTE),[1] high thermal conductivity,[4] mechanical stiffness,[5] dielectric constant/refractive index,[6] and piezoelectric coefficient.[7,8] Accordingly, there is significant interest in AlN and AlN/III-N alloys for numerous electronic,[9,10] opto-electronic,[11,12] electro-mechanical,[13,14] electro-acoustic,[15,16] and energy harvesting[17,18] device applications. Due to inherent economic advantages, significant interest exists for fabricating these and related AlN based devices on large diameter silicon (Si) substrates.[19,20] Unfortunately, the relatively large mismatch between Si and AlN in lattice constant and CTE (19% and 60% for AlN (0001)/Si (111) interface, respectively[1,12]), and the high reactivity of Si with nitrogen[20,21] create significant interfacial engineering challenges.[22] Despite these challenges, AlN is also commonly utilized as a buffer or seed layer for the growth of GaN and III-N alloys on Si to prevent liquid Ga at the Si growth surface, facilitate elimination of threading dislocations, and enable growth of compressive GaN to counteract tensile stresses created by the large CTE mismatch between GaN and Si.[21,23]

In addition to the above challenges, charge transport and carrier recombination at the AlN/Si interface are a significant consideration for the performance and reliability of AlN/Si and other III-N/Si heterostructure devices.[24,25] However, there have been relatively few investigations of the valence and conduction band alignment at III-N/Si interfaces.[26] In this regard, we have utilized *in-situ* x-ray photoelectron spectroscopy (XPS) to investigate both the chemistry and band alignment at interfaces formed by ammonia (NH_3) gas-source molecular beam epitaxy (NH_3-GSMBE)[27] of AlN on Si (001) and (111) substrates.

II. EXPERIMENTAL

The *n*-type, phosphorous doped (8–1.2 Ω cm) Si (001) and (111) substrates utilized in this study were purchased from Virginia Semiconductor, Inc. Prior to loading into vacuum, the wafers were dipped in 10:1 buffered HF for 10 min to remove the native oxide. They were then subsequently degassed/annealed at 1050 °C in 10^{-9} Torr vacuum in the GSMBE system for 10–15 min to desorb any remaining surface oxide or other contaminants.[28,29] The oxygen, carbon, and nitrogen contamination of the thermally desorbed Si substrates were below the detection limits of XPS. *In-situ* low energy electron diffraction (LEED) of the same Si surfaces revealed (2×1) and (7×7) reconstructions, respectively, for the (001) and the (111) orientations.

The AlN NH_3-GSMBE was performed in a custom built system specifically designed for the heteroepitaxial growth of AlN, GaN, and ScN on Si and SiC substrates.[30,31] The details of this system and the AlN growth conditions have been previously described.[32,33] Briefly, source materials in the NH_3-GSMBE system relevant to this study consisted of NH_3 (99.9995%) and Al (99.9999%). The NH_3 was further purified via an inline metalorganic resin purifier connected directly to a leak valve mounted on the GSMBE chamber. Growth of AlN was performed in a back-pressure of 10^{-5}–10^{-4} Torr NH_3 at temperatures of 600–1100 °C. The Al Knudsen cell was operated at ~1050 °C. In an attempt to minimize/modulate possible interfacial Si-N bond formation, the Si substrate

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was first exposed to the Al flux for varying times (1–15 min) before introducing NH_3 into the system.[21,34,35] The background pressure during the Al pre-exposure was $<5\times10^{-8}$ Torr, and quadrapole mass spectrometer measurements showed Al ($m/e^- = 27$) and N_2 ($m/e^- = 28$) were the primary background species.

The XPS measurements have been described previously and were performed in a separate vacuum chamber attached to the main ultra high vacuum (UHV) transfer line.[33,36] Briefly, all XPS spectra were collected using Al Kα radiation ($h\nu = 1484.6$ eV) in a 2×10^{-10} Torr UHV system equipped with a 100 mm radius hemispherical electron energy analyzer (VG CLAMII). Conditions previously demonstrated to minimize charging and surface photovoltage effects for GaN/AlN and AlN/6H-SiC (0001) interfaces were utilized.[33,36] Calibration of the binding energy scale for all scans was achieved by periodically taking scans of the Au $4f_{7/2}$ and Cu $2p_{3/2}$ peaks from standards and correcting for any discrepancies with the known values (83.98 and 932.67 eV, respectively). A combined Gaussian-Lorentzian curve shape with a linear back-ground was found to best represent the XPS data. The thickness of the AlN films was estimated based on the attenuation of the substrate Si 2p core level (CL) and utilizing previously described relationships for determining the Si 2p attenuation length in AlN for layer by layer growth.[27]

The method of Kraut *et al.*,[37] previously described in detail,[33,36] was utilized to determine the VBO at the AlN/Si interface. The method relies on referencing distinct CLs in each material to their respective valence band maximum (VBM), and then measuring the relative position of these core levels with respect to one another at their interface. Specifically, the valence band offset (ΔE_v) for the AlN/Si interface was determined according to Eq. (1), as per

$$\Delta E_v(\mathrm{AlN/Si}) = (\mathrm{CL}-\mathrm{VBM})_{\mathrm{AlN}} - (\mathrm{CL}-\mathrm{VBM})_{\mathrm{Si}} + \Delta\mathrm{CL}_{\mathrm{int}}, \quad (1)$$

where (CL–VBM) is the relative position of the core level to the valence band maximum of the bulk material, and ΔCL_{int} is the relative position of the core levels in the two materials at the interface [i.e., $\Delta CL_{int} = (CL_{Si} - CL_{AlN})_{int}$]. To determine ΔCL_{int} for the AlN/Si interface, we deposited ~2 nm of AlN on the Si substrate and measured the relative position of the Al 2p, N 1s, and Si 2p core levels at the interface. For the Si (001) and (111) substrates, $(CL–VBM)_{bulk}$ for the Si 2p core level was determined from a high resolution scan of the Si valence band after the *ex-situ* HF clean. For AlN, we have previously determined $(CL–VBM)_{bulk}$ for the Al 2p core level to be 71.4 ± 0.2 eV for AlN grown at 1050 °C.[30]

III. RESULTS AND DISCUSSION

Figure 1 shows an XPS valence band spectrum acquired from a Si (001) substrate after the *ex-situ* HF clean and loading into the XPS system. A linear extrapolation from the inflection point of the valence band spectrum locates the valence band maximum at 1.1 ± 0.2 eV below the system Fermi level. As shown in Figure 2(a), the Si 2p core level

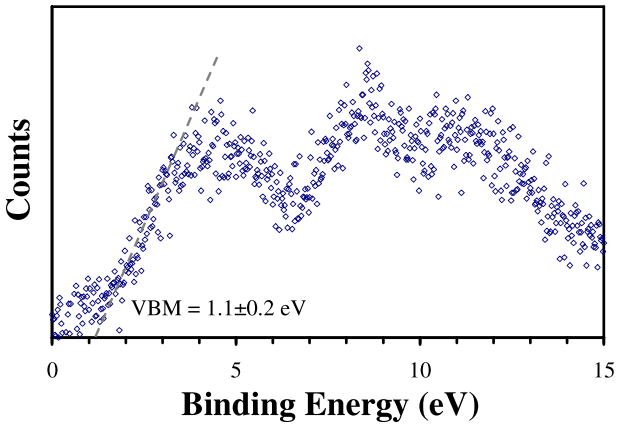

FIG. 1. XPS valence band spectrum for Si (001) surface after *ex-situ* HF clean.

from the Si (001) substrate was well fitted using a single Gaussian-Lorentzian line shape and located at 100.1 ± 0.03 eV. The corresponding Si 2p-VBM value was determined to be 99.0 ± 0.2 eV. This value is in excellent agreement with the values of 98.94 ± 0.04 eV reported by Bersch *et al.*[38] and 98.98 ± 0.05 eV reported by Chambers *et al.*[39] The same value of 99.0 ± 0.2 eV was determined for Si 2p-VBM of the Si (111) substrate surface after *ex-situ* HF cleaning.

Figure 2(b) shows the Si 2p core level after growth of AlN on the Si (001) surface at 1050 °C, with a long 15 min Al pre-exposure. The Si 2p core level in this case was fitted using two peaks at 99.9 and 102.6 eV. The former is attributed to Si-Si bonding in the Si (001) substrate and the latter to Si-N bonding formed at the interface between the Si (001) substrate and the AlN film. Based on the attenuation of the substrate Si 2p core level, the thickness of the AlN film was estimated to be ~2 nm.[27] Figure 2(c) shows the Si 2p core level after growth of ~1.5 nm of AlN on a Si (111) surface using the same growth conditions, but instead with a shorter 5 min Al pre-exposure. In this case, two Si 2p core levels attributable to substrate Si-Si and interfacial Si-N bonding were again observed.

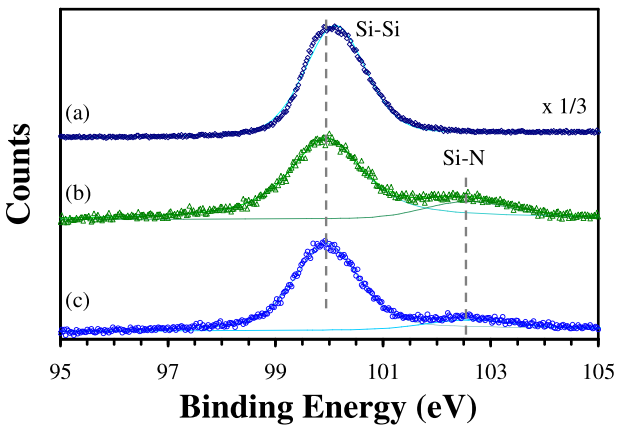

FIG. 2. XPS spectra of Si 2p core level after (a) *ex-situ* HF clean and after *in-situ* NH_3-GSMBE growth of AlN at 1050 °C on (b) Si (001) and (c) Si (111) substrates. Note: dashed lines are included to indicate portions of the Si 2p spectra attributed to Si-Si and Si-N bonding and not exact peak positions.

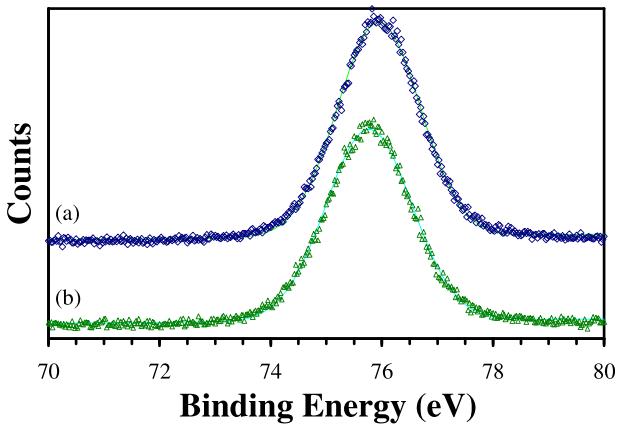

FIG. 3. XPS of Al 2p core level after NH_3-GSMBE growth of AlN at 1050 °C on (a) Si (001) and (b) Si (111) substrates.

The amount of interfacial Si-N bonding observed with Al pre-exposures as short as 1 min was similar to that for the longer exposures. This indicates that for the Al flux and growth temperature utilized, the amount of interfacial Si-N bond formation is relatively constant for Al pre-exposure times >1 min. Reducing the AlN growth temperature to as low as 600 °C was found to decrease but not eliminate the observation of some interfacial Si-N bonding in the Si 2p XPS spectra. This is consistent with other reports of AlN buffer layer growth on Si, where the growth temperature has been intentionally reduced to ~600 °C in order to minimize interfacial SiN_x formation.[40] We do note the possibility for some Si-N bond formation to occur via reaction with background NH_3 in the GSMBE during the Al pre-exposure. However, the amount of interfacial Si-N bonding formed in this manner was likely minimal as Si-N was not observed by XPS of the Si wafer after thermal desorption cleaning in the GSMBE with similar background NH_3 levels.

Figure 3(a) shows the Al 2p core level from the same 2 nm AlN film grown on the Si (001) substrate, as shown in Fig. 2(b). The Al 2p peak was well fitted using a single Gaussian-Lorentzian line shape centered at 75.9 ± 0.03 eV with a full width half maximum (FWHM) of 1.75 eV. Figure 3(b) shows a similar result for the Al 2p core level from the same 1.5 nm AlN film grown on the Si (111) substrate, as shown in Figure 2(c). The N 1s core level for both AlN films (not shown) was also well fitted using a single Gaussian-Lorentzian lineshape centered at 399.1 ± 0.1 eV with a FWHM of 1.8 eV. No LEED pattern was observed for AlN grown on the Si (001) substrate. However, a diffuse (1 × 1) LEED pattern was observed for AlN grown on the Si (111) substrate.

For the AlN/Si (001) and AlN/Si(111) interfaces, ΔCL_{int} was determined to be 24.0 ± 0.05 and 24.1 ± 0.05 eV, respectively, from the fitted position of the Al 2p and Si 2p core levels, as shown in Figures 2 and 3. Using Eq. (1) and the previously determined values for ΔCL_{int} and $(CL–VBM)_{bulk}$, the corresponding VBOs for the AlN/Si (001) and AlN/Si(111) interfaces were, respectively, determined to be −3.6 ± 0.2 and −3.5 ± 0.2 eV (see Table I). The negative sign for the VBO indicates that the AlN valence band resides below the Si valence band (see Figure 4). Identical AlN/Si VBO values were obtained using the N 1s and Si 2p core levels. This is consistent with the relative position of the N 1s and Al 2p core levels being constant at 323.2 ± 0.05 eV in this study. Factoring in additional repeat VBO measurements and taking the root mean square of the variances for all the measurements, we determined the VBO for both the AlN/Si(001) and AlN/Si(111) interfaces to be −3.5 ± 0.3 eV.

To determine the AlN/Si CBO, we utilize the literature values of 1.1 and 6.2 eV for the band gaps of Si and wurtzite AlN, respectively. The corresponding AlN/Si CBO was then directly calculated to be 1.6 ± 0.3 eV revealing a type I band alignment as shown in Fig. 4. Since AlN buffer layers for GaN heteroepitaxy on Si are sometimes grown at lower temperatures and then annealed at higher temperatures,[40] we performed an additional growth where the AlN was initially grown at 600 °C and then the growth temperature was ramped to 1000 °C. In this case, we determined a slightly lower VBO of −3.3 ± 0.2 eV with a correspondingly higher CBO of 1.8 ± 0.2 eV.

These values and trends are in reasonable agreement with prior internal photoemission (IPE) and photoconductivity (PC) measurements by Badylevich *et al.* for aluminum nitride grown by atomic layer deposition on a Si (001) substrate with a chemically grown 0.8 nm SiO_x surface layer.[26] For an amorphous AlN film grown at 325–400 °C, they determined a VBO of ~−2.6 eV and for the same film crystallized via annealing at 1100 °C, they determined a VBO of ~−3.3 eV. All of these similar values indicate that the AlN/Si VBO is relatively insensitive to both the substrate orientation ((100) vs. (111)) and the interfacial bonding (i.e., SiN_x vs. SiO_x). The strong agreement between XPS and IPE

TABLE I. Summary of CL-VBM, ΔCL_{int}, VBO, and CBO values determined for the Si and AlN surfaces and interfaces investigated in this study.

Surface/interface	Si2p-VBM (eV)	Al2p-VBM (eV)	Si2p-Al2p (eV)	VBO (eV)	CBO (eV)
Si (001)	99.0 ± 0.2	…	…	…	…
AlN	…	71.4 ± 0.2 (Ref. 30)	…	…	…
AlN/Si (001)	…	…	24 ± 0.05	−3.6 ± 0.2	…
AlN/Si (111)	…	…	24.1 ± 0.05	−3.5 ± 0.2	…
AlN/Si	…	…	…	−3.5 ± 0.3[a]	1.6 ± 0.3[b]
GaN/Si	…	…	…	−2.7 ± 0.3[c]	−0.4 ± 0.3[b]

[a]Average of additional repeat measurements for both AlN/Si (001) and AlN/Si (111) interfaces.
[b]Calculated using VBO and reported band gaps of AlN (6.2 eV) and GaN (3.4 eV).[1]
[c]Calculated using transitive and commutative rules and VBOs for AlN/Si and GaN/AlN interfaces determined in this and prior studies.[30]

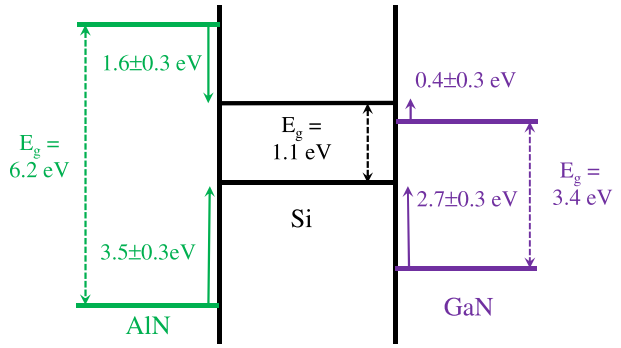

FIG. 4. Schematic flat band diagram showing the VBO and CBO for AlN/Si and GaN/Si interfaces.

measurements also suggests that the differential charging effects previously observed in XPS measurements of the band alignment of HfO_2 to *p*-type Si are likely negligible or within the error bar of these measurements.[38]

Using the above band alignment for the AlN/Si interface and prior measurements of the GaN/AlN band alignment,[30] the VBO at a GaN/Si interface can additionally be estimated using the rules of transitivity and commutativity.[41,42] The transitive and commutative rules for VBOs, respectively, state that

$$\Delta E_v(a/b) + \Delta E_v(b/c) + \Delta E_v(c/a) = 0, \quad (2)$$

$$\Delta E_v(b/c) = \Delta E_v(c/b), \quad (3)$$

where in this study, a/b signifies the GaN/Si interface in question, b/c signifies the Si/AlN interface, and c/a signifies the AlN/GaN interface. Using the above rules and the previously determined VBOs for AlN/Si and GaN/AlN ($\Delta E_v = 0.8 \pm 0.2$ eV),[30] we deduced a GaN/Si VBO of -2.7 ± 0.3 eV. Using the literature value of 3.4 eV for wurtzite GaN,[1] we determined a type II band alignment for the GaN/Si interface with a CBO of -0.4 ± 0.3 eV, where the negative sign in this case indicates the GaN conduction band resides below the Si conduction band (see Figure 4). This estimate for the GaN/Si band alignment can be further confirmed by again using the VBO transitive and commutative rules and prior reports for the Si_3N_4/GaN and Si_3N_4/Si VBOs of -0.5 ± 0.1 eV (Ref. 43) and -2.1 ± 0.1 eV,[44] respectively. Using these values, we calculated a GaN/Si VBO of -2.6 ± 0.1 eV, that is, within the error bar of the estimate based on our XPS measurements.

IV. CONCLUSIONS

In summary, we have utilized *in-situ* XPS to determine the VBO present at AlN/Si (001) and AlN/Si (111) interfaces. The VBO was determined to be -3.5 ± 0.3 eV and insensitive to Si orientation and interfacial bonding. Using the transitive and commutative rules for VBOs, the VBO and CBO at a GaN/Si interface were additionally deduced to be -2.7 ± 0.3 eV and -0.4 ± 0.3 eV, respectively.

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