Band alignment of sputtered and atomic layer deposited SiO₂ and Al₂O₃ on ScAIN

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២ Xinyi Xia, ២ Jian-Sian Li, ២ Md Irfan Khan, et al.

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Band alignment of sputtered and atomic layer deposited SiO_2 and Al_2O_3 on ScAlN

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Xinyi Xia,¹ D Jian-Sian Li,¹ Md Irfan Khan,² Kamruzzaman Khan,² Elaheh Ahmadi,² D David C. Hays,³ Fan Ren,¹ and S. J. Pearton^{4,a)}

AFFILIATIONS

¹Department of Chemical Engineering, University of Florida, Gainesville, Florida 32611, USA

²Department of Electrical Engineering and Computer Science, University of Michigan, Ann Arbor, Michigan 48109, USA

³Nanoscale Research Facility, University of Florida, Gainesville, Florida 32611, USA

⁴Department of Materials Science and Engineering, University of Florida, Gainesville, Florida 32611, USA

^{a)}Author to whom correspondence should be addressed: spear@mse.ufl.edu

ABSTRACT

The band alignments of two candidate dielectrics for ScAlN, namely, SiO₂ and Al₂O₂, were obtained by x-ray photoelectron spectroscopy. We compared the effect of deposition method on the valence band offsets of both sputtered and atomic layer deposition films of SiO₂ and Al₂O₃ on Sc_{0.27}Al_{0.73} N (bandgap 5.1 eV) films. The band alignments are type I (straddled gap) for SiO₂ and type II (staggered gap) for Al₂O₃. The deposition methods make a large difference in relative valence band offsets, in the range 0.4–0.5 eV for both SiO₂ and Al₂O₃. The absolute valence band offsets were 2.1 or 2.6 eV for SiO₂ and 1.5 or 1.9 eV for Al₂O₃ on ScAlN. Conduction band offsets derived from these valence band offsets, and the measured bandgaps were then in the range 1.0–1.1 eV for SiO₂ and 0.30–0.70 eV for Al₂O₃. These latter differences can be partially ascribed to changes in bandgap for the case of SiO₂ deposited by the two different methods, but not for Al₂O₃, where the bandgap as independent of deposition method. Since both dielectrics can be selectively removed from ScAlN, they are promising as gate dielectrics for transistor structures.

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I. INTRODUCTION

Recent advances in the growth of ScAlN by molecular beam epitaxy (MBE)¹⁻³ and metal organic chemical vapor deposition (MOCVD)^{4,5} have led to significant renewed interest for its use in high frequency, thin film, surface acoustic wave (SAW) resonators^{6–15} and ferroelectric memories.¹⁶ The improved purity and crystalline quality of these films lead to improved ferroelectric switching characteristics compared to previous sputter-deposited films.⁶ AlScN alloys with a high concentration of scandium can significantly improve the piezoelectric properties relative to the more common AlN piezoelectric films used in micro-electromechanical systems (MEMS), such as acoustic resonators for sensor and actuator applications.¹⁻⁷ Sc enhances the spontaneous polarization in hexagonal AlN and increases the piezoelectric and pyroelectric coefficients.¹⁵⁻²⁰ The AlScN/GaN heterostructure has also been used for high electron mobility transistors (HEMTs) with excellent dc and RF characteristics.^{21–23} In that case, the typical Sc mole fraction is typically in the range 17%–25%.²

A key requirement for any advanced device application is to have dielectric films for ScAlN that provide carrier confinement in heterojunctions, surface passivation and can be selectively patterned or removed without damaging the ScAlN. Two of the most promising dielectrics for ScAlN are SiO₂ and Al₂O₃, due to their well-developed deposition processes and their large bandgaps. SiO₂ has a bandgap of ~8.7 eV, which is a prerequisite for achieving large valence and conduction band offsets, blocking hole, and electron transfer. Although Al₂O₃ (~6.3 eV) has a smaller bandgap than SiO₂, its dielectric constant is higher, making it advantageous in various applications. For devices such as ScAlN metal–oxide– semiconductor field effect transistors (MOSFETs), the integration of higher dielectric constant (κ) materials in the gate structure can lower the effect of interface defects, lower the device's power consumption, and increase the capacitance density of the gate oxide.

In this paper, we report the band alignment on ScAlN of these two dielectrics, namely, SiO_2 and Al_2O_3 , which were each deposited by two different methods—sputtering or atomic layer deposition





(ALD). The band alignments are type I (straddled gap) for SiO_2 and type II (staggered gap) for Al_2O_3 . The magnitude of the valence and conduction band offsets depends on the dielectric deposition method. This shows that careful attention must be paid to the process integration of these dielectrics with ScAlN to optimize the quality of the heterointerfaces between this material and the dielectrics.

II. EXPERIMENTAL

0.739 nm

The ScAlN sample was grown on a Veeco GENXplore MBE system with conventional Al, Ga, and Sc effusion cells and a radio-frequency (RF) plasma source to supply active nitrogen. The N source consisted of ultrahigh-purity (99.9995%) N₂ gas flowing at 1 SCCM through the RF-plasma source with 350 W RF power, which corresponded to a growth rate of ~6 nm/min for metal rich GaN layers. Commercially available on axis semi-insulating Ga polar GaN on sapphire substrate was used for the growth of Sc_{0.27}Al_{0.73}N. Before the growth, the substrate was coated with 500 nm of e-beam evaporated Ti on the backside to ensure uniform heating by the substrate heater. Then, the substrate was exposed to the ultraviolet (UV) ozone and diluted buffered hydrofluoric acid (BHF) etching to remove possible polishing damages and



FIG. 3. High-resolution XRD plot of Sc_{0.27}Al_{0.73}N.

impurities from the substrate surface. After that, the substrate was solvent cleaned with 4 min soak of acetone, methanol, and isopropanol to remove all the organic residues from the substrate surface. After cleaning, the substrate piece was bonded to a Si wafer with molten In. The prepared substrate was then loaded into the MBE load lock chamber. An hour of baking was performed at 400 °C in the buffer chamber to remove any water prior to transferring the substrate to the growth chamber. During the growth, the substrate



FIG. 4. EDS spectra of ScAIN.

6.0 nm 6.0 nm

FIG. 2. AFM image $(2 \times 2 \mu m^2)$ of Sc_{0.27}Al_{0.73}N films grown at 750 °C.



FIG. 5. High-resolution XPS spectra for the vacuum-core delta regions of $Sc_{0.27}AI_{0.73}N$.

temperature was measured and monitored using the thermocouple. The growth was monitored *in situ* via reflection high-energy electron diffraction (RHEED).

The growth was initiated by five cycles of Ga deposition and desorption to improve the surface quality and uniformity by removing residues from the substrate surfaces. After that 200 nm of n+ metal rich GaN was grown at 740 °C using a Ga beam equivalent pressure of 6.4×10^{-7} Torr to ensure a smooth and clean surface. The excess Ga was desorbed every 17 min by closing the Ga shutter while keeping the nitrogen shutter open until a bright and streaky RHEED pattern was observed.²⁴

After the n⁺ GaN growth, 200 nm of $Sc_{0.27}Al_{0.73}N$ was grown under N-rich conditions, which are beneficial to improve phase purity and surface roughness. The Sc composition is accurate to ±1%. The growth temperature of ScAlN was 750 °C. Figure 1 shows a schematic of the structure. The bandgap of ScAlN at this composition is 5.1 eV.^{25,26} We did not employ x-ray photoelectron spectroscopy (XPS) to determine the bandgap of ScAlN by looking at the energy loss region of the lattice elements because both Sc and Al have spin–orbit splitting, which makes it difficult to determine the "zero" point. Also, the N 1s peak was too close to the Sc peak, overlapping the onset of inelastic losses.

A Veeco Dimension ICON atomic force microscopy (AFM) was employed to characterize the surface morphology of the



FIG. 6. High-resolution XPS spectra for the vacuum-core delta regions of (top) sputtered SiO_2 compared to (bottom) ALD SiO_2 . The intensity is in arbitrary units (a.u.).



FIG. 7. REELS spectra for sputtered and ALD SiO_2 (top) and sputtered and ALD Al_2O_3 (bottom).

samples. A smooth surface morphology with an rms roughness of ~0.74 nm has been obtained for the ScAlN film as shown in the $2 \times 2 \mu m^2$ AFM image in Fig. 2. High-resolution x-ray diffraction (HRXRD) ω -2 θ scan of the sample was recorded on Rigaku Smartlab XRD. The XRD plot presented in Fig. 3 clearly shows the GaN and the ScAlN peaks. This also confirms the pure wurtzite phase of ScAlN present in the samples. The second peak close to ScAlN peak can be attributed to AlN buffer layer present in the substrate. Figure 4 shows the energy dispersive spectroscopy (EDS) spectra of ScAlN film measured in a Hitachi SU8000 scanning electron microscope (SEM). The EDS shows 26% Sc in the ScAlN sample. However, 27% Sc has been obtained from secondary ion mass spectrometry (SIMS) of a ScAlN thin film grown in similar growth condition.

To measure band alignments of SiO₂ or Al₂O₃ on ScAlN, these dielectrics were deposited by two different methods, RF magnetron sputtering or ALD. The deposition conditions have been described previously.²⁷ In brief, the sputtering was performed near room temperature with pure Si or Al targets in a $3\%O_2/Ar$ ambient. The ALD layers for both dielectrics were deposited at 200 °C using trimethylaluminum or tris (diethylamino) silane as precursors, respectively, for SiO₂ or Al₂O₃. The bandgaps of the dielectrics were 8.7 eV for ALD SiO₂ and 8.3 eV for sputtered SiO₂ and 6.3 eV for Al₂O₃ deposited by either method. These were determined by reflection electron energy loss spectroscopy (REELS) and by the O1s peak from the reference dielectric data and were consistent with those reported previously.²⁷ Thus, subsequent differences in conduction band offsets were not due to differences in the Al₂O₃ bandgaps deposited by the two methods but were affected in the case of SiO₂. Both thick (200 nm) and thin (1.5 nm) layers of the SiO₃ or Al₂O₃ were deposited for measuring their bandgaps and core levels for these layers on ScAlN. REELS has an advantage over UV/Vis since it only requires the film to be at least as thick as the sampling depth, which is typically a few nanometers. In addition, most UV/Vis systems have a cutoff at ~6 eV, but REELS does not have such an energy restriction.

XPS performed with a Physical Instruments ULVAC PHI system was used to obtain valence band offsets with the standard method of Kraut,²⁸ employing an Al x-ray source (energy 1486.6 eV) with a source power 300 W, an analysis size of $20\,\mu$ m diameter, a takeoff angle of 50°, and an acceptance angle of \pm 7°. The electron pass energy was 23.5 eV for high-resolution scans and 93.5 eV for survey scans. The total energy resolution of this XPS system is about 0.5 eV, and the accuracy of the observed binding energy is within 0.03 eV.

III. RESULTS AND DISCUSSION

To measure the band alignment using the Kraut method, three samples are needed. First, precise core level and valence band edge data must be measured from thick samples of ScAlN and also both dielectrics under investigation.^{28–30} Then, the same core level locations measured in these bulk samples are re-measured within a heterostructure of SiO₂/ScAlN and Al₂O₃/ScAlN. The shift of the core level binding energy locations within the heterostructures as compared to the initial bulk binding energies can be used to determine the respective valence band offsets.^{28–30} Figure 5 shows the high-resolution valence band maximum (VBM)-core delta region in ScAlN. We used the Sc peak for both SiO₂ and Al₂O₃ band alignment. Similarly, high-resolution XPS spectra of the valence band maximum (VBM)-core delta region are shown in Fig. 6 for the sputtered and ALD SiO₂ (top) and for the corresponding Al₂O₃ cases (bottom).

The REELS spectra for all the thick dielectrics are shown in Fig. 7. The dielectric bandgap is obtained from the onset of the electron energy loss spectra. The energy where the onset of inelastic losses occurs is obtained by extrapolating the linear-fit line and calculating its intersection with the "zero" level. The bandgap is the difference between the centroid of elastic scattering and the calculated intersection. As shown in Fig. 7, while the bandgaps of the Al_2O_3 films are independent of the deposition method, there is a difference of 0.3 eV for SiO₂.

Figure 8 shows the XPS spectra for the ScAlN to (top) sputtered SiO_2 and ALD SiO_2 and to (bottom) sputtered and ALD Al_2O_3 core delta regions in the heterostructure samples. These values are summarized in Table I and were used to calculate the valence band offsets for the different structures used in this study.³¹ The separation between the reference core levels can be translated





directly into a value for the valence band offsets (VBOs) using the previously measured single layer sample core-level to valence band maximum (VBM) energies. There are shifts of 0.4-0.5 eV in the VBO from sputtered to ALD dielectrics in each case. This is commonly observed in these dielectrics on other materials, showing the effect of the deposition method on the VBO, which is directly measured and not derived from other measurements. As suggested previously,²⁹ the valence band offset can be changed by modification of the interface between the sputtered dielectrics and ScAlN. In the case of sputter deposition, it is highly likely that the impingement of energetic ions during the process can cause changes at this interface, compared to the more benign environment of ALD. The ion energies during sputtering are up to ~500 eV, well above the threshold for atomic displacements to occur in ScAlN. Another possible source of interfacial changes would be metallic contamination during the sputtering process, originating from the electrodes in the chamber. However, the XPS data did not show any presence of metallic contamination in the dielectric films deposited by either sputtering or ALD. The VBO at the interfaces

of the latter with ScAlN is probably the normal values. It will be interesting to measure interlace state densities in capacitor structures to quantitatively establish the difference between the two types of deposition on ScAlN. Clearly, however, ALD appears to be a better choice for dielectric deposition on this material relative to sputtering.

Having measured the VBOs and bandgaps, we then derived the conduction band offsets from these values. Figures 9 and 10 show the band alignment for the SiO₂/Sc_{0.27}Al_{0.73}N heterostructure in which SiO₂ was deposited by sputtering (Fig. 9) or ALD (Fig. 10). Both are nested type I alignments. There is a difference of 0.5 eV in the VBOs and 0.1 eV in the conduction band offsets (CBOs) between the two deposition methods. Both the VBO and CBO are >1 eV, which is a rule-of-thumb for having effective carrier confinement of both electrons and holes in electronic devices. In our experience, SiO₂ can be selectively removed from ScAlN with standard buffered oxide etches, and F₂-based plasma etching, so these are advantages from a practical device processing view.

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	ScAIN (27%So	(2			Reference SiO	2		Thin SiO_2	2 on ScAlN	
VBM	Core level peak (Sc 2p)	Core-VBM	Film	VBM	Core level peak (Si 2p)	Core-VBM	Core level peak (Sc 2p)	Core level peak (Si 2p)	\triangle Core level	Valence band offset
-0.6	397.8	398.4	Sputt. SiO ₂ ALD SiO ₂	4.8 5.7	103.5 104.3	98.7 98.6	398.7 398.8	101.1 101.6	297.6 297.2	2.1 2.6
	ScAlN (27%So	(2			Reference Al ₂ C)3		Thin Al ₂ O	3 on ScAlN	
VBM	Core level peak (Sc 2p)	Core-VBM	Film	VBM	Core level peak (Al 2p)	Core-VBM	Core level peak (Sc 2p)	Core level peak (Al 2p)	\triangle Core level	Valence band offset
-0.6	397.8	398.4	Sputt. Al ₂ O ₃ ALD Al ₂ O ₃	$1.6 \\ 1.1$	72.8 72.3	71.2 71.2	398.2 398.2	72.5 72.9	325.7 325.3	1.5 1.9











FIG. 11. Band diagram for sputtered Al₂O₃ on Sc_{0.27}Al_{0.73}N.



FIG. 12. Band diagram for ALD Al₂O₃ on Sc_{0.27}Al_{0.73}N.

Similar results for the Al₂O₃/ScAlN heterostructures are shown in Fig. 11 for the sputtered dielectric and Fig. 12 for the ALD dielectric. These are type II, staggered alignments. However, while the VBOs are 1.5 eV, the CBOs are small, between 0.3 and 0.7 eV, which means electron confinement would not be as effective as hole confinement. Al₂O₃ can also be selectively removed from ScAlN by the same wet and dry processes for SiO₂. Given the larger CBOs for SiO₂, it appears this is a superior choice as a dielectric on ScAlN for electronic device applications.

IV. SUMMARY AND CONCLUSIONS

In device designs that utilize a gate dielectric, one of the most crucial parameters for the dielectric/semiconductor system of interest is that the dielectric acts as a barrier to both holes and electrons to prevent leakage current. SiO_2 has type I band alignments while Al_2O_3 has type II band alignments on a composition of ScAlN (27 at. % Sc) typical of actuator and RF transistor applications and both would provide good carrier confinement. We find that there is a significant difference in valence band offsets between dielectrics deposited by sputtering compared to ALD. This has been observed in other ultra-wide bandgap materials and indicates that these semiconductors are susceptible to surface disorder during sputtering.^{29,30}

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Xinyi Xia: Conceptualization (equal); Data curation (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Writing – original draft (equal). Jian-Sian Li: Conceptualization (equal); Data curation (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Writing – original draft (equal). Md Irfan Khan: Conceptualization (equal); Data curation (equal); Formal analysis (equal); Funding acquisition (equal); Investigation (equal); Writing – original draft (equal). Kamruzzaman Khan: Conceptualization (equal); Data curation (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Writing – original draft (equal). **Elaheh Ahmadi:** Conceptualization (equal); Data curation (equal); Formal analysis (equal); Funding acquisition (equal); Investigation (equal); Methodology (equal); Writing – original draft (equal). **David C. Hays:** Conceptualization (equal); Data curation (equal); Formal analysis (equal); Investigation (equal); Writing – original draft (equal). **Fan Ren:** Conceptualization (equal); Data curation (equal); Formal analysis (equal); Funding acquisition (equal); Writing – original draft (equal). **S. J. Pearton:** Conceptualization (equal); Data curation (equal); Formal analysis (equal); Funding acquisition (equal); Methodology (equal); Writing – original draft (equal).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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