

Influence of AlN buffer layer on molecular beam epitaxy growth of wurtzite $Al_{1-x}Sc_xN$ thin films

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Abstract. Wurtzite– $Al_{1-x}Sc_xN$ thin films deposited by solid-state alloying of AlN with ScN exhibit high piezoelectric coefficient and large band gap that makes it a promising material for a variety of applications in piezo-electronics, electronic, acoustoelectric devices, etc. Research on epitaxial $Al_{1-x}Sc_xN$ growth in wurtzite crystal structure is still at an early stage and achieving high scandium (Sc) concentrations in epitaxial films without any phase separation or secondary phase formation is still a critical challenge. Moreover, as most of the reports of wurtzite– $Al_{1-x}Sc_xN$ growth thus far relies on low-vacuum growth techniques, such as magnetron sputtering that are prone to large impurities and contaminants detrimental for device applications, high-vacuum deposition techniques, such as molecular beam epitaxy method needs to be developed. In this paper, we report the epitaxial growth of wurtzite– $Al_{1-x}Sc_xN$ on sapphire (Al_2O_3) substrates under different Sc fluxes using ultra-high vacuum plasma-assisted molecular beam epitaxy. To prevent ScN phase separation, a 30 nm AlN buffer layer is deposited *in situ* on GaN epilayers as well as Al_2O_3 substrates that result in phase-pure wurtzite– $Al_{1-x}Sc_xN$ thin films without any phase separation or secondary phase formation. The structural and compositional analyses performed with high-resolution X-ray diffraction (HRXRD) and secondary ion mass spectroscopy (SIMS), reveal epitaxial wurtzite– $Al_{1-x}Sc_xN$ on AlN buffer layers will enable the development of devices with improved efficiencies.

Keywords. Wurtzite– $Al_{1-x}Sc_xN$; molecular beam epitaxy; phase separation.

1. Introduction

Solid-state alloys of compound semiconducting materials have been an important research topic for band gap engineering, lattice-matching, dielectric permittivity engineering in the heterostructure materials for a variety of electronic, optoelectronic and energy conversion applications [1]. Such semiconductor alloys have led to a wide variety of novel devices, such as diode lasers, light emitting diodes (LED), high electron mobility transistors (HEMT), heterojunction bipolar transistor, photo detectors, etc. [2,3], and are increasingly studied for the next generations of industrial applications, such as quantum computing, quantum information processing, optical communications, sensing, etc. [4]. Though the initial efforts to deposit semiconductor alloys were focussed on the technologically important III–V compounds, such as arsenides, antimonides and phosphides for optoelectronic applications in 1950s–1980s [5–8], over the past couple of decades, the attention has shifted to III-nitrides (III-N) due to their attractive properties, such as wide band gap, high breakdown voltage, remarkable chemical and thermal stabilities, etc. [9]. LEDs and power electronic devices based on GaN have been commercialized, and several new III-N alloys and their devices, such as InGaN-based blue-green LEDs for efficient solid-state lighting [10], AlGaN-based UV LEDs for water purification [11] and InAlN-based radio-frequency and power electronic devices [12,13] were also currently developed.

On the other hand, extensive research directed towards finding new materials with improved properties and capabilities to expand the device application space have led to a new class of nitride semiconductors based on

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group III-(B) transition metals [14,15]. Among this class of materials, ScN and its alloys with GaN and AlN are reported to show fascinating properties and have potential device applications [16,17]. Since ScN and traditional III-Ns such as AlN, GaN and InN are isovalent, but nonisostructural (in stable phase) compounds, their alloys have rocksalt (Sc-rich phase) or wurtzite (Al- or Ga-rich phase) crystal structure [18]. The rocksalt phase ScN and Al_{1-x} Sc_xN are actively being researched for thermoelectric applications [19–21], whereas the wurtzite phase of Al_{1-x} Sc_rN with Sc <0.5 mole fraction exhibits a larger piezoelectric coefficient [22,23] i.e., higher than that of AlN. According to modelling predictions and limited experimental results, $Al_{1-x}Sc_xN$ has three times higher spontaneous polarization and a four to five times higher piezoelectric coefficient in comparison to AlN [23,24]. Such high piezoelectric response makes wurtzite $-Al_{1-x}$ Sc_xN attractive for use in resonators for wide-band width, high-frequency RF filters, energy harvesting, lead-free hightemperature piezoelectric applications, etc.

In $Al_{1-x}Sc_xN$, however, since the binary compounds (AlN and ScN) exhibit different crystal structures, they do not readily dissolve in one another, and hence, the majority of efforts to deposit epitaxial $Al_{1-x}Sc_xN$ alloys were based on non-equilibrium process, such as magnetron sputter deposition [25–27], hybrid vapour phase epitaxy (HVPE) [28], metal-organic chemical vapour deposition (MOCVD) [29], etc. Structural characterizations have showed that the Al-rich $Al_{1-x}Sc_xN$ films exhibit wurtzite structures and grow with *c*-axis orientations with very good crystalline quality on Al_2O_3 substrates [30]. However, high growth rate and low-vacuum growth process in such techniques resulted in impurities and defects that limits their suitability for device applications. Molecular beam epitaxy (MBE) technique has several advantages in terms of control over the thickness, reduced unintentional impurity incorporation, high crystalline quality with smooth and sharp interfaces, etc. However, so far only a couple of research reports exist on $Al_{1-x}Sc_xN$ growth using MBE [31,32]. Hardy *et al* [31] relied on the metal-rich conditions, while varying the substrate temperature to optimize the growth parameters to obtain phase-pure $Al_{1-x}Sc_xN$. They concluded that lowering the Sc flux suppresses the cubic phase inclusion in the films, and a low growth temperature window from 520 to 730°C improves the crystal quality and morphology. However, the metal-rich growth condition is found to degrade the phase purity due to the formation of Al/Sc intermetallic compounds and also incorporation of metallic Al. To avoid such metallic inclusions, Frei et al [32] used pulsed metal supply and studied the effect of metal-tonitrogen ratio on the properties of $Al_{1-r}Sc_rN$. Their results showed that the pulsed supply did not entirely prevent the formation of metallic inclusions in the films. Therefore, even though high-crystalline quality of $Al_{1-x}Sc_xN$ has been obtained, phase purity is still a critical issue in $Al_{1-x}Sc_xN$ growth. In this paper, $Al_{1-x}Sc_xN$ film growth is demonstrated on Al₂O₃ substrates in nitrogen-rich growth conditions. Further, the effect of Sc flux on the structure and morphology of the $Al_{1-x}Sc_xN$ thin films is investigated. Phase-pure wurtzite $Al_{1-x}Sc_xN$ is obtained, further, by depositing a thin (30 nm) AlN buffer layer on Al₂O₃ and GaN epilayer substrates that prevents any ScN phase separation. Nitrogen-rich growth conditions are particularly important to suppress the nitrogen vacancies in the films that otherwise dope the III-nitrides with high electron concentrations and limits their electronic device efficiencies.

2. Experimental

Epitaxial $Al_{1-x}Sc_xN$ thin films were deposited with a IIInitride plasma-assisted MBE (SVTA, USA) system with high temperature Al and Sc K-cells, RF plasma source for nitrogen and with a base pressure of 3×10^{-11} Torr. Highpurity Sc (purity 99.999%) and Al (purity 99.99999%) flakes were loaded into alumina and PBN crucibles, respectively, inside the Sc and Al K-cells, respectively. Since the objective of the work was to deposit $Al_{1-r}Sc_rN$ thin films in wurtzite crystal structure (w-Al_{1-x}Sc_xN), c-plane Al₂O₃ was chosen as the substrate. The substrates were thoroughly cleaned prior to deposition by degreasing with organic solvents-acetone and propanol, rinsed with de-ionized water, and blow dried. The substrates were transferred to the preparation chamber, where it was degassed at 600°C for an hour before transferring them into the growth chamber. Before commencing the growth, the substrate was degassed again at 800°C for 30 min and subsequently, the substrate temperature was brought down to 200°C and the substrate was exposed to nitrogen plasma for a duration of 2 h with 375 W RF power and 3 sccm N₂ flow. Such nitridation leads to the formation of a few monolayers of AlN on Al₂O₃ substrates, as confirmed by the streaky patterns of in situ reflection high energy electron diffraction (RHEED) used to monitor the substrate and epilayer surface during the growth. Following the nitridation step, the substrate temperature was ramped up to the growth temperature of 660°C. For the main growth, the N₂ flow was reduced to 1 sccm and the RF power to 300 W. The total growth duration was limited to 60 min with an aim of a layer thickness of ~ 300 nm. A pristine AlN sample and a set of three $Al_{1-x}Sc_xN$ samples were grown by varying the Sc K-cell temperature and keeping the rest of the parameters as constants. The Al K-cell temperature was kept at 1250°C which corresponds to a beam equivalent pressure (BEP) of 1×10^{-7} Torr. Details of the samples with different Sc K-cell temperatures and corresponding flux are presented in table 1. Furthermore, a separate set of $Al_{1-x}Sc_xN$ samples were prepared with a thin AlN buffer layer on Al₂O₃ and GaN epilayer substrates. The growth parameter used for the buffer layer was same as that of pristine AlN thin film described earlier. Post the deposition

Sample	Sc K-cell, T (°C)	Sc flux (Torr)	Composition (based on flux)
A	_	_	AlN
В	1225	8.5×10^{-8}	$Al_{0.54}Sc_{0.46}N$
С	1250	9.5×10^{-8}	Al _{0.51} Sc _{0.49} N
D	1295	1.0×10^{-7}	Al _{0.50} Sc _{0.50} N

Table 1. Sc K-cell temperature, corresponding beam equivalent pressure (BEP) and composition based on the flux.

process, several *ex situ* characterization techniques were employed to study the samples as follows. Field emission scanning electron microscope (FESEM, FEI Quanta 3D, The Netherlands) was used to study the morphology and to measure the film thickness. High resolution X-ray diffraction (HRXRD, Bruker D8 Discover, USA) was employed to determine the structural properties and confirm the epitaxial nature of growth. Secondary ion mass spectrometer (SIMS) depth profiling was used to determine the $Al_{1-x}Sc_xN$ composition.

3. Results and discussion

3.1 Growth of $Al_{1-x}Sc_xN$ on Al_2O_3 substrates

Figure 1 shows FESEM image of the samples' morphology and final RHEED pattern of the films. The RHEED patterns are composed of streaks and spots indicating 3D structures with flat tops. The spotty nature of the pattern increases from sample A to D. The sample D shows additional spots that are slightly misaligned in the vertical axis, indicating the presence of additional phases. The FESEM images show



Figure 1. Surface morphology and corresponding RHEED pattern of (a) pristine AlN, and $Al_{1-x}Sc_xN$ films with Sc K-cell temperatures of (b) 1225, (c) 1250 and (d) 1295°C.

that the AlN film has a columnar morphology, whereas the typical morphology of the $Al_{1-x}Sc_xN$ samples as coalesced flat-topped islands. With the increase in Sc flux, the thin films were found to become flatter and more compact. A difference in contrast was seen in some portions of the samples that could arise from a difference in the level of charging, possibly due to the presence of separate phases. The density of such dark contrast regions increases with the applied Sc flux. The film thicknesses have been determined from cross-sectional FESEM images (not shown) and are tabulated in table 2.

The composition of $Al_{1-x}Sc_xN$ films were analysed with SIMS, where oxygen ions with 5 keV energy were used as primary beam to eject and identify secondary ions or molecules containing Sc, Al, and N in the epitaxial layer. A magnetron sputter-deposited Al_{0.62}Sc_{0.38}N thin film (with composition characterized by Rutherford backscattering spectroscopy (RBS) previously) was used as reference sample for the determination of Sc composition in the MBE grown AlScN layers. Figure 2 shows the SIMS depth profiles of samples B and C grown with different Sc flux. The dotted line in figure 2 indicates the interface between thin film and the substrate. The atomic percentage of the Sc in $Al_{1-x}Sc_xN$ determined by the SIMS is presented in table 2. As can be noted from the SIMS profiles, the distribution of constituent elements is nominally uniform throughout the thickness of the layers except for the sample D (not shown here) indicating that film with high Sc flux had highly nonuniform composition through the depth, possibly due to phase separation or secondary phase formation. Also, Sc profile in all the samples have shown slight bump at the middle and near the substrate surface indicating that there is small variation in Sc composition in the samples grown with lower Sc flux.

To determine the epitaxial relationship and lattice parameters of the films, high resolution $2\theta - \omega$, ω and ϕ XRD scans of each of the films were performed. Figure 3 shows $2\theta - \omega$ scans of samples A to D in symmetric geometry. The XRD patterns show the sharp 006 peak of the Al₂O₃ substrate and several sample-related peaks. The AlN sample shows only an AlN 002 reflection apart from the substrate peak. The AlN 002 peak has a high intensity comparable to the substrate peak and is sharp. For the Al_{1-x}Sc_xN samples, the dominant sample peak is identified as Al_{1-x}Sc_xN 002 reflection occurring in the range of

Table 2. Summary of results obtained from cross-section FESEM image, SIMS and HRXRD analysis of AlN and $Al_{1-x}Sc_xN$ layers with different Sc compositions.

Sample	Thickness (nm)	Sc at%	<i>c</i> (Å)	a (Å)	FWHM (deg.) ω-scan
A	349		4.990	3.106	0.03
В	332	0.40	4.981	3.094	0.46
С	292	0.47	4.985	3.110	0.13
D	303	—	4.988	3.105	0.23



Figure 2. SIMS depth profiles of (a) pristine AlN and $Al_{1-x}Sc_xN$ films grown on Al_2O_3 substrates with Sc K-cell temperatures of (b) 1225 and (c) 1250°C.



Figure 3. (a) $2\theta - \omega$ scans of AlN and $Al_{1-x}Sc_xN$ samples with different Sc flux, (b) ω -scan (rocking curve) of the same samples and (c) representative ϕ -scan of $Al_{1-x}Sc_xN$ (i.e., sample C) showing six-fold symmetry.



Figure 4. XRD patterns of (a) $Al_2O_3/AlN/Al_{0.6}Sc_{0.4}N$ and (b) $Al_2O_3/GaN/AlN/Al_{0.6}Sc_{0.4}N$. Surface morphology of (c) $Al_2O_3/AlN/Al_{0.6}Sc_{0.4}N$ and (d) $Al_2O_3/GaN/AlN/Al_{0.6}Sc_{0.4}N$.

35.98–36.04° and is accompanied by smaller peaks around 34.5 and 37.8°. The former is identified as 111 reflection of ScN and the latter is believed to be originated from the substrate, which will be evidenced in the following section. The $2\theta-\omega$ scan confirmed that 002 reflection of the Al_{1-x}Sc_xN layers is aligned to the 006 reflection of the Al₂O₃, i.e., the out-of-plane epitaxial relationship is $[0001]_{Al_{1-x}SC_xN} \parallel [0001]_{Al_2O_3}$ between the film and substrate. To probe the in-plane epitaxial relationship, ϕ scan of 101 reflection of Al_{1-x}Sc_xN was performed. Figure 3c shows the ϕ scan for sample C and is representative of all the samples. The six peaks in the ϕ scan reflect the six-fold symmetry of the parent AlN lattice.

The ScN 111 peak is at least an order of magnitude smaller in intensity compared to the primary 002 $Al_{1-x}Sc_xN$ peak. As the Sc flux is increased from sample A to D, the intensity of these peaks increases monotonically, while the $Al_{1-x}Sc_xN$ 002 peak intensity reduces. The in-plane (*a*) and out-of-plane (*c*) lattice parameters of the prevalent $Al_{1-x}Sc_xN$ phase were calculated from the symmetric 002 and asymmetric 101 $2\theta-\omega$ XRD spectra, and the values have been tabulated in table 2. With increase in Sc flux, the out-of-plane (*c*) lattice parameter of the samples is found to increase, whereas no proper trend is seen for the in-plane (*a*) lattice constant. Figure 3 also shows the rocking curves of the 002 reflection of AlN and $Al_{1-x}Sc_xN$ films. The rocking curve width for the AlN 002 reflection was measured as 1.6 arc min (0.027°). As the Sc flux is increased, the corresponding width of the rocking curve also increases in general, although sample B shows an anomalously large FWHM. Since the width of the rocking curve is an indicator of crystalline quality and especially micro-strain, we can conclude that the increase in Sc flux leads to deterioration in thin film quality. Geometric mis-orientation among the coalesced islands which make up the film also influences the rocking curve width. From table 2, it can be seen that the lattice parameters are close to the relaxed lattice parameters of AlN which means that in this growth regime, by employing high Sc flux, there is a complete segregation of the material between an AlScN phase with scant Sc, and nearly pure ScN phase. The clear observation of phase separation in XRD data are also in agreement with RHEED pattern for the $Al_{1-x}Sc_xN$ with high Sc flux. In addition, SIMS analysis indicated the presence of inhomogeneity in the Sc composition in the thin films. This may be due to the use of comparatively high Sc flux during the growth. Strain arising due to the large lattice mismatch between the Al₂O₃ substrate and $Al_{1-x}Sc_xN$ layer is believed to have played a role in the observed phase separation.

3.2 Growth of $Al_{1-x}Sc_xN$ on AlN buffer layers on GaN and Al_2O_3 substrates

To reduce lattice mismatch between $Al_{1-x}Sc_xN$ and the substrate and to achieve phase-pure $Al_{1-x}Sc_xN$ thin films,

a thin (30 nm) AlN buffer layer was deposited prior to $Al_{1-x}Sc_xN$ growth on Al_2O_3 substrate as well as on commercial 3-µm thick GaN epilayers on Al₂O₃ substrates. The growth condition for $Al_{1-x}Sc_xN$ was same as the sample B deposited previously that resulted at 60% AlN mole-fraction. Comparison of the XRD patterns of Al₂O₃/AlN/Al_{0.6}Sc_{0.4}N and Al₂O₃/GaN/AlN/Al_{0.6}Sc_{0.4}N with their respective substrates (see figure 4a and b) reveal only one film peak at 36.1° corresponding to the (0002) oriented growth of $Al_{1-r}Sc_rN$ and a *c*-axis latticeconstant of 4.98 Å. Interestingly, there is no ScN 111 peaks that were otherwise present on films deposited directly on Al₂O₃ substrates confirming that realization of phase-pure wurtzite-Al_{0.6}Sc_{0.4}N with AlN buffer layers. The surface morphology of Al₂O₃/AlN/Al_{0.6}Sc_{0.4}N film appear similar to the $Al_2O_3/Al_{1-x}Sc_xN$ i.e., the one deposited without the buffer layers. However, Al₂O₃/ GaN/AlN/Al_{0.6}Sc_{0.4}N film exhibit columnar morphology similar to sputter-deposited wurtzite $-Al_{1-x}Sc_xN$ [27]. Thus, it is clear that with the introduction of AlN buffer layer, high quality phase-pure wurtzite-Al_{0.6}Sc_{0.4}N layer was achieved. Importantly, the growth of wurtzite- $Al_{1-x}Sc_xN$ on GaN/AlN buffer layer will help to develop its integration for device applications.

4. Conclusion

 $Al_{1-x}Sc_xN$ thin films with varying Sc compositions have been deposited in wurtzite crystal phase and their crystal qualities are compared with that of pristine AlN. HRXRD and SIMS analysis revealed high degree of ScN phase separation when $Al_{1-x}Sc_xN$ is deposited directly on Al_2O_3 substrates. However, with the introduction of a thin 30 nm AlN buffer layer on Al_2O_3 and GaN epilayers deposited on Al_2O_3 substrates, phase-pure $Al_{1-x}Sc_xN$ thin films were achieved. The development of phase-pure wurtzite $Al_{1-x}Sc_xN$ thin films will help to understand different growth modes of $Al_{1-x}Sc_xN$ thin films and help to design devices for industrial applications.

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