Metal-Organic Chemical Vapor Deposition of Aluminum Scandium Nitride

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Dedicated to the memory of D. Koleske, who authored a detailed and very useful report on his attempts to grow GaScN

Alloys of scandium with AlN exhibit an enhanced piezoelectric coefficient that can boost the performance of nitride-based electronic and optoelectronic devices such as high electron mobility transistors (HEMTs). Consequently, there is increasing interest in the epitaxial growth of high-quality AlScN/GaN heterostructures. So far, only very recent reports on AlScN HEMT structures grown by molecular beam epitaxy (MBE) have been published. Herein, the motivation for depositing AlScN epitaxial layers by metal-organic chemical vapor deposition (MOCVD) as well as the challenges associated with this approach are explained. For the first time, the successful deposition of epitaxial layers with a Sc content up to 30% [Al<sub>x</sub>Sc<sub>1-x</sub>N] is reported. It is shown that the deposited films consist of wurtzite-type AlScN with high crystalline quality, demonstrating that MOCVD is suitable for the growth of HEMT structures with Sc-based ternary nitrides.

Recently, ternary metastable nitrides, such as AlScN and GaScN, have started to gain attention due to enhanced properties attractive for electroacoustic (AlScN)<sup>1</sup> as well as optoelectronic (GaScN) applications.<sup>2–4</sup> Such Sc-based nitrides could boost the performance of conventional group III-nitrides (AlN, GaN, and InN) in optoelectronic applications, power electronics, and high-frequency electronics.<sup>5</sup> Mixing enthalpy calculations<sup>6,9</sup> have shown that the wurtzite-type structure of GaN or AlN is preserved in an alloy with ScN for a certain composition range. One of the most prominent effects of incorporating ScN into group III-nitrides is the strong increase in the piezoelectric coefficient<sup>1,7,8</sup> AlScN has a three times higher spontaneous polarization<sup>9</sup> and a four to five times higher piezoelectric coefficient<sup>10</sup> compared with AlN. Nitride-based high electron mobility transistors (HEMTs) would strongly benefit from the increased piezoelectric coefficient, which would generate a much higher density of carriers in the 2D electron gas (2DEG) formed at the interface between a barrier layer (e.g., AlScN) and a channel layer (e.g., GaN).<sup>11</sup> AlGaN- or AlN-based HEMTs have already been adopted at an industrial level, thanks to their high power density and output power at high frequencies.<sup>12</sup> Even so, further improvements can be expected from AlScN-based HEMTs. Furthermore, such heterostructures are typically deposited on top of a GaN buffer layer, yet when the barrier layer (e.g., AlN) has a thickness above its critical value, excessive stress is generated and the layer cracks. Al<sub>0.82</sub>Sc<sub>0.18</sub>N has the same a-lattice parameter as GaN,<sup>2</sup> so that no constraints exist for the adoption of barrier layers thicker than 5 nm, which is the limit in the case of AlN-based HEMTs. Recently, the first proof-of-concept AlScN-based HEMTs were demonstrated using molecular beam epitaxy (MBE). These HEMT structures have much higher sheet carrier densities (above 3 × 10<sup>13</sup> cm<sup>−2</sup>) compared with standard AlGaN/GaN HEMTs while maintaining high electron mobilities (close to 1000 cm<sup>2</sup> V<sup>−1</sup> s<sup>−1</sup>).<sup>13–15</sup> To the best of our knowledge, electrical results for devices manufactured from these structures have been published for the first time this year.<sup>16,17</sup> AlScN/GaN HEMT devices with a current density of 2.4 A mm<sup>−1</sup> and a transconductance of 0.67 S mm<sup>−1</sup> have been demonstrated with cutoff frequencies up to 88 GHz<sup>17</sup> comparable with the values reported for state-of-the-art AlGaN/GaN HEMTs.<sup>12</sup>

The growth of crystalline Sc-based nitrides’ epitaxial layers may be accomplished through different methods: magnetron sputtering, hydride vapor phase epitaxy (HVPE), MBE, and metal-organic chemical vapor deposition (MOCVD). Which

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technique is most suited for manufacturing these epitaxial heterostructures in an industrial setting is open to question.

Even though high-quality AlScN thin films can be deposited by magnetron sputtering, as demonstrated by various researchers during the past few years,\cite{18-21} this approach may not work for the fabrication of light-emitting diode (LED) and HEMT structures, which depend on the sharpness of the interfaces between the epitaxial layers, a low defect density, and high material purity. AlScN nanowires\cite{22} and ScN layers\cite{23,24} have been grown by HVPE using metallic Sc as source material reacting with an HCl/H₂ mixture. However, ScN layers deposited using this method suffer from a high incorporation of impurities, which is detrimental for the realization of HEMT structures. In addition, HVPE facilitates very high growth rates (i.e., \( \geq 10 \mu m h^{-1} \)), which are unnecessary or even disadvantageous for this applications, because for HEMT and LED structures, very thin layers (i.e., below 10 nm) must be grown with accurate control of the composition.

So far, MBE was the only technique that was successfully used to fabricate AlScN-based HEMT structures because it can grow epitaxial layers of high crystal quality as well as abrupt and smooth interfaces.\cite{13-15} However, the typically very slow growth rates (\( \leq 1 \mu m h^{-1} \) for GaN layers) and, to some extent, the limitation on the number of wafers that can be processed simultaneously result in a low throughput. Together with the large delays associated with any planned or unplanned maintenance, MBE may result in an unattractive growth technique for large-scale industrial production.

To bring AlScN semiconductor materials from fundamental research to industrial production, the technique used to grow these layers must provide high quality, uniform heterostructures on multiple large-diameter wafers per growth run, and reasonably short process times. MOCVD reactors typically fulfill these requirements. MOCVD can achieve relatively high growth rates (very controllable in a range from 0.01 to 5 \( \mu m h^{-1} \)) and high productivity, is considered as a well-established method for large-scale production, and is therefore the technique of choice for manufacturing AlN- and GaN-based nitride semiconductor structures. However, the very few reported attempts to grow group-III-nitride layers alloyed with ScN by MOCVD have been unsuccessful. Saidi et al.\cite{3} as well as Koleske et al.\cite{4} attempted to grow GaScN by MOCVD, but achieved only doping levels (Sc concentrations below 1 \( \times 10^{19} \) cm\(^{-3} \)), which are not at all close to the requirements of HEMT or LED structures (Sc concentration above 10%).

The difficulty of producing AlScN or GaScN by MOCVD observed in the previously published works indicates a fundamental challenge that has to be overcome before the industrialization and the widespread use of such novel ternary nitrides becomes a reality. Therefore, the approach of our work was to first understand what steps have to be taken prior to the growth to achieve high crystalline quality wurtzite-type AlScN epitaxial layers by MOCVD and only then to proceed with the growth experiments. Thereafter, we demonstrate successful incorporation of Sc up to 30% in AlScN layers, the highest incorporated concentration of Sc into AlN reported so far. We explain that these results could be achieved on account of these three factors: 1) the choice of a proper precursor of Sc; 2) the rebuilding of the MOCVD reactor to have a sufficient amount of Sc in the gas phase to achieve such high concentrations; and 3) the understanding of the crystal structure, quality, and purity of MOCVD grown epitaxial layers through structural and compositional analysis.

**Choice of a precursor for Sc:** High vapor pressure is needed to let a relevant amount of Sc-molecules enter the MOCVD reaction chamber and be incorporated in the epitaxial layer at an alloy level. Sc compounds using arene ligands may lead to high vapor pressure, but their synthetic route is highly complicated and demands a very specific setup,\cite{25,26} making the synthesis impractical from an industrial production point of view. Other potential Sc precursors exist, but to be suitable for the MOCVD of AlScN layers for HEMT structures, they must be free of oxygen to prevent its incorporation into the epitaxial layers. This restricts the choice of precursors considerably. For example, the molecule tris(N,N-di-prop-2'-yl-formamidinato)scandium (C\(_{21}\)H\(_{48}\)ScN\(_{3}\)) is oxygen-free but has a vapor pressure even lower than typical metalloocene complexes.

The synthesis and characteristics of scandiocene (tris(methylcyclopentadienyl)Sc or (Cp)\(_{3}\)Sc) and its analogue with MeCp instead of Cp as the ligand have been studied several years ago,\cite{27} and the precursor synthesis is nowadays established. Blom et al.\cite{27} did not report any vapor pressure for Cp\(_{3}\)Sc or MeCp\(_{3}\)Sc, but rather only for Cp\(_{3}\)Yb and MeCp\(_{3}\)Yb, which are quoted as \( \approx 10^{-7} \) mm Hg at room temperature. This value is very low, much lower than that of Cp\(_{3}\)Mg and Cp\(_{2}\)Fe, typically used for doping GaN with Mg and Fe. Nevertheless, based on these findings and although no data were available for their vapor pressure, we concluded that Cp\(_{3}\)Sc and or (MeCp)\(_{3}\)Sc should be tested at first as the most promising potential precursors for the MOCVD of AlScN epitaxial layers. Cp\(_{3}\)Sc is a commercially available precursor, whereas its analogue (MeCp)\(_{3}\)Sc requires a more difficult synthetic route. Therefore, it is not used in this work.

**Rebuilding of the MOCVD reactor:** As mentioned earlier, both Saidi et al. and Koleske et al. tried the epitaxial growth of GaScN by MOCVD with Cp\(_{3}\)Sc and (MeCp)\(_{3}\)Sc\cite{3,4} but they achieved only doping levels of Sc in GaN. Assuming that Cp\(_{3}\)Sc has a similar vapor pressure as Cp\(_{3}\)Yb,\cite{27} we calculated that the molar flow of Cp\(_{3}\)Sc in the gas phase in the setup used by Saidi et al. (Cp\(_{3}\)Sc bubbler heated to 90°C and held at atmospheric pressure) would be in the range only from 5 \( \times 10^{13} \) moles min\(^{-1} \) to 10 \( \times 10^{8} \) mol min\(^{-1} \). This should typically result in Sc concentrations <10\(^{19}\) cm\(^{-3} \), validating the results reported by Saidi et al.

The molar flow can be calculated using the general gas equation:

\[
P V = nRT, \quad \text{where} \quad P \text{ is the pressure (in pascals),} \quad V \text{ is the volume (in cubic meters),} \quad n \text{ is the amount of moles,} \quad R \text{ is the gas constant equal to 8.314462 J K}^{-1} \text{mol}^{-1}, \quad \text{and} \quad T \text{ is the temperature (in Kelvin).}
\]

In our specific case, we define the molar flow rate of Sc (\( n_{sc} \)) as the amount of moles per minute

\[
n_{sc} = \frac{P_{sc} \times V_{tot}}{R \times T} \quad \text{(1)}
\]

where \( P_{sc} \) is the pressure of the Sc precursor, which depends on the vapor pressure of the precursor at the temperature \( T \) at which the bubbler is kept. \( V_{tot} \) is the volume flow rate of the gas mixture leaving the bubbler (carrier gas and Sc precursor). A more precise calculation also considers the increased molar flow from the bubbler, which operates at a lower pressure than atmospheric. Accordingly, the molar flow rate should be multiplied by the ratio between atmospheric pressure and the bubbler pressure.
We then calculated that the $\text{C}_p\text{Sc}$ bubbler should be heated to a temperature exceeding 120 °C to reach a molar flow close to $10^{-4}$ mol min$^{-1}$. At 150 °C, the molar flow would become comparable with that of trimethylaluminum (TMAI), which is used for the deposition of the AlN barrier layer of a HEMT structure grown in an MOCVD reactor (typically $10^{-6}$ to $10^{-5}$ mol min$^{-1}$). This would make a large difference in the Sc amount that can be incorporated into AlN during growth, in contrast to the very low levels of incorporation reported by Saidi et al. and Koleske et al., in whose works the precursors were heated only to temperatures <90 °C.

Controlling a bubbler and its gas flow at these elevated temperatures is, however, an additional technological challenge. MOCVD reactors are usually equipped with mass flow controllers (MFCs), pressure controllers, valves, gas injection systems, and other components that are necessary for the precise control of the gas quantities of the precursors and to ensure a high control and reproducibility of the epitaxial growth process (thickness and composition of the epitaxial layers). Most of these components contain either sealing materials or electronic parts that cannot be heated to temperatures higher than 70 °C or possibly 90 °C. As explained earlier, one of the biggest challenges in the growth of Sc-based ternary nitrides is to have a high enough molar flow, which is possible only by maintaining both the Sc precursor and the gas lines extending to the reaction chamber at a suitable elevated temperature. In addition, careful control of the pressure and amount of gas which flows inward and outward from the Sc-precursor bubbler has to be ensured.

In this study, with $\text{C}_p\text{Sc}$ as the precursor, we wanted the bubbler and the gas lines to be heated up to 150 °C. Consequently, MFCs and pressure controller either had to be installed in a way that they were not affected by the high temperature or they had to be compatible with such conditions. An MFC to regulate the amount of hydrogen flowing into the Sc bubbler was relocated upstream of the heated line and the bubbler. A heated pressure gauge was installed downstream of the bubbler to measure the pressure in the bubbler and estimate the molar flow of $\text{C}_p\text{Sc}$ transported in the gas phase. All the components were checked to be compatible with the temperature of 150 °C used for the bubbler and the gas lines to the reaction chamber. The injection system of the reaction chamber was also heated to prevent any condensation of the $\text{C}_p\text{Sc}$ vapors.

**Growth and characterization of AlScN epitaxial layers:** Epitaxial growth experiments to deposit AlScN (0001) layers in a multiwafer MOCVD reactor with a showerhead injection system were performed using sapphire $\text{Al}_2\text{O}_3$ (0001) 4” substrates. A typical structure consisted of a 1.5 μm GaN epitaxial layer, followed by a 5–10 nm thin AlN spacer, a 10–100 nm-thick AlScN layer and capped with a few nanometers of AlN or GaN. The whole epitaxial structure was grown in the same MOCVD reactor unless otherwise specified. The entire growth process took 3–5 h depending on the thickness of the layers. The typical growth rate of GaN layers was 2 μm h$^{-1}$, whereas that of AlScN layers was ≈0.07 μm h$^{-1}$. Although the growth rate of the AlScN layers was compatible with that achieved with MBE,[21] the rate of GaN growth by MOCVD is much higher. While with our MOCVD setup, the deposition of a complete HEMT structure requires ≈3 h the same structure could be grown by MBE in probably no less than 6 or 7 h.

Different values of epitaxial growth parameters, such as temperature, pressure, V/III ratio (ratio of the molar flow of the group V precursor nitrogen vs the molar flow of the group III precursors Al and Sc), carrier gas flow, molar flow of the Al precursor, and the amount of $\text{H}_2$ flowing in the $\text{C}_p\text{Sc}$ bubbler, were tested. Their full effects are still under investigation and an in-depth study will be published in future. The structures investigated and discussed in this work have been deposited using a growth temperature of 1000 °C, a pressure of 40 mbar, a V/III ratio of 3000, and a standard carrier gas flow of 20 slm.

The composition of the AlScN layers was analyzed by secondary-ion mass spectrometry (SIMS), where Ca$^+$ ions with 5 keV energy were used as the primary ion beam to identify secondary ions or molecules containing Sc, Al, Ga, N, C, and O in the epitaxial layers. Magnetron-sputtered AlScN thin films[21] were used as calibration samples for the SIMS setup to ensure the correct determination of the Al/Sc ratio. The structural properties of the layers were analyzed by high-resolution X-ray diffraction (HRXRD). The HRXRD measurements were carried out with a Cu radiation source providing Cu Kα radiation and a two-bounce Ge 220 monochromator. An optical microscope equipped with Nomarski differential interference contrast (NDIC) and an atomic force microscope (AFM) in tapping mode were used to evaluate the surface morphology of these layers and to determine the surface roughness in form of the root mean square (RMS) average.

In Figure 1, we show the SIMS profile of a 70 nm-thick $\text{Al}_0.3\text{Sc}_{0.7}\text{N}$ layer with a 10 nm-thick AlN spacer and a 5 nm AlN cap deposited on a 4” $\text{GaN/Al}_2\text{O}_3$ sample. Such a high Sc concentration has never been demonstrated in material grown by MOCVD before. It clearly shows that in this study, we have succeeded in introducing a high molar flow of $\text{C}_p\text{Sc}$ into the MOCVD reactor. The flow resulted in a growth rate of up to 0.07 μm h$^{-1}$, which would be sufficient to deposit a 10 nm barrier layer of a HEMT structure in less than 10 min. It is shown that the amount of Sc incorporated in the 70 nm AlScN layer is constant, which indicates that the Sc supply system was stable during the deposition process. A diffusion of Sc and Al into the GaN layer and vice versa is visible. This is quite common
in MOCVD growth and is largely due to the high growth temperatures. For example, such diffusion was also previously observed in high-quality AlN/GaN heterointerfaces without impeding the functionality of HEMTs[28]; therefore, it was not caused specifically by Sc incorporation. SIMS analysis has also shown that a significant amount of carbon and oxygen is incorporated in the epitaxial layers (in the order of $10^{19}$–$10^{20}$ atoms cm$^{-3}$), calculated using pure AlN standard (Figure S1, Supporting Information). We could not quantify the exact concentration of C and O due to the current unavailability of SIMS calibration standards for AlScN. A systematic SIMS investigation of C and O in AlScN layers is planned in the near future, using C- and O-implanted AlScN layers. Carbon incorporation is most likely due to the Cp$_3$Sc molecule, which is used as the precursor for Sc. This molecule contains 15 atoms of C and only one atom of Sc. Consequently, a large amount of C is present in the gas phase in the MOCVD reaction chamber and may be incorporated in the epitaxial layer, too. In the case of GaN and AlN epitaxial growth, C incorporation can also happen due to the use of typical precursors ((CH$_3$)$_3$Ga and (CH$_3$)$_3$Al) that contain C atoms, although at a lower ratio (3:1) of carbon atoms to metal atoms. This incorporation phenomenon is called autodoping[29,30] and can be controlled by the growth parameters, either to intentionally deposit C-doped GaN layers with a concentration up to $10^{19}$ cm$^{-3}$ or to reduce it to less than $10^{15}$ cm$^{-3}$[31]. We expect that by further optimizing the epitaxial growth conditions of AlScN, it should be possible to reduce the amount of unintentionally incorporated C. Regarding the incorporation of oxygen, we have observed that AlN layers, grown under the same conditions except for the absence of a Sc supply, have a comparable concentration of O. Therefore, we conclude that the main source of O is not the Sc source. Similar to the case of C, the incorporation of O could be controlled by the proper choice of growth parameters,[32] e.g., higher substrate temperatures and higher V/III ratios, as well as using electronic-grade sources with oxygen concentrations <1 ppm. Currently, the Cp$_3$Sc source material used in this study was classified as “research grade” by the manufacturer and the exact quantity of O was not measured. However, an electronic-grade Cp$_3$Sc bubbler can be obtained, and its use and effects on the amount of O incorporated in the AlScN epitaxial layers will be presented in future work. Further actions, such as using an optoelectronic grade TMAl source and installing a finer purifier for NH$_3$, will be taken and tested with the electronic-grade Cp$_3$Sc bubbler, to reduce the oxygen concentration in the AlScN layers.

HRXRD measurements were performed on a 12 nm-thick Al$_{0.8}$Sc$_{0.2}$N layer having a 3 nm AlN spacer and a 2 nm GaN cap layer (Figure 2). The HRXRD phase analysis shows that pure wurtzite-type phase AlScN with high crystalline quality has been deposited, as indicated by the presence of thickness fringes for the 0002, 0004, and 0006 reflections. The full width at half maximum (FWHM) of omega-scans of AlScN peak is largely determined from the FWHM of the GaN epitaxial layer underneath, as already described for AlInN and AlInGaN on GaN heterostructures.[33] In the aforementioned sample, the FWHM of GaN 00.2 $\omega$-scan is 251 arcsec, whereas that of AlScN 00.2 $\omega$-scan is 257 arcsec, which is indicative of very high crystal quality.

For analysis of the lattice parameter of the AlScN layer, HRXRD reciprocal space maps (RSMs) of the 0002 and $\bar{1}$ $\bar{1}$ 24 reflection range were recorded for the same sample (Figure 3). The lattice parameters for the Al$_{0.8}$Sc$_{0.2}$N were estimated to be $a = 318.21$ pm and $c = 498.80$ pm, whereas for the GaN buffer layer, $a = 318.21$ pm and $c = 519.14$ pm were determined, respectively. The identical in-plane lattice parameter $a$ of the AlScN layer and the GaN buffer layer proves that pseudomorphic growth was achieved for the AlScN layer by MOCVD. In addition, both the RSM of the 0002 and $\bar{1}$ $\bar{1}$ 24 reflections show well-resolved thickness fringes confirming the high structural quality of the AlScN layer.

The optical microscopy images (Figure S2, Supporting Information) indicate that depending on layer composition,
different behavior is observed: 1) for epitaxial layers having a Sc content of 10–15%, multiple cracks appear at thicknesses above \( \gtrsim 15 \text{ nm} \) (Figure S2a, Supporting Information) because the thickness of the layers exceeded its critical thickness at this point (matching the theoretical calculations reported by Moram and Zhang[5]). 2) When the Sc content is 15–30% (similar to the sample used for the HRXRD analysis in Figure 2), crack-free layers with a thickness of 20nm or higher could be deposited (Figure S2b, Supporting Information).

AFM analysis of a 12 nm \( \text{Al}_{0.8}\text{Sc}_{0.2}\text{N} \) epitaxial layer having a 3 nm AlN spacer and a 2 nm GaN cap layer (Figure S3, Supporting Information) indicates a relatively rough surface, with an RMS roughness of 2.9 nm measured for a \( 10 \times 10 \text{ \mu m}^2 \) area and an RMS roughness of 2.8 nm for a \( 2 \times 2 \text{ \mu m}^2 \) area. This surface roughness suggests that the AlScN layers tend to grow in a 3D mode, which is probably not due to optimized growth conditions used in this study. Nevertheless, the structure is very uniform and no other defect features can be observed. Furthermore, a similar sample morphology was also reported by Hardy et al. for the growth of AlScN layers by MBE with a high V/III ratio.[14] A different V/III ratio, a higher supersaturation, and investigation of the growth temperature effect should allow addressing this issue and further experiments will be conducted to promote the lateral growth mode. The 3D structure of the AlScN barrier layer is expected to affect the electrical characteristics of the 2DEG, and the process should be further optimized before high-performance HEMT structures can be achieved. Even so, a HEMT test structure with a 12 nm \( \text{Al}_{0.8}\text{Sc}_{0.2}\text{N} \) barrier layer and a 3 nm GaN cap layer was grown. Capacitance–voltage measurements done with a mercury-probe revealed the presence of a 2DEG, whereas sheet resistance measurements conducted using a Leighton eddy-current system showed a very low sheet resistance of 230 \( \Omega \text{ sq}^{-1} \). A detailed analysis of the growth parameters for the deposition of high-quality HEMT structures will be presented in future work.

In conclusion, in this work, we have identified cyclopentadienyl-coordinated Sc molecules as the most suitable and available precursor for MOCVD of Sc-containing nitrides and calculated that a set-up temperature of 150 \( ^\circ \text{C} \) is required to have a relevant amount of the Sc precursor in the gas phase. We have assembled the gas supply system of our MOCVD reactor in such a way that the Sc source and its gas lines could be heated to that temperature and the flow of the Sc-precursor vapor could be controlled reproducibly.

In this way, we have demonstrated for the first time—to the best of our knowledge—that it is possible to deposit epitaxial layers of wurtzite-type AlScN layers by MOCVD with a Sc content of up to 30% and high crystalline quality. Further optimization of the growth process will allow making HEMT structures or optoelectronic devices with increased performance for the future of energy-efficient nitride-based electronics.

**Supporting Information**

Supporting Information is available from the Wiley Online Library or from the author.

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**Conflict of Interest**

The authors declare no conflict of interest.

**Keywords**

AlScN, aluminum scandium nitride, high electron mobility transistors, metal-organic chemical vapor deposition, ScAIN, vapor pressures

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