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On the dynamics in chemical vapor deposition of InN

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ABSTRACT
Epitaxial nanometer-thin indium nitride (InN) films are considered promising active layers in various device applications but remain challenging to deposit. We compare the morphological evolution and characterizations of InN films with various growth conditions in chemical vapor deposition (CVD) by both a plasma atomic layer deposition (ALD) approach and a conventional metalorganic CVD approach. Our results show that a time-resolved precursor supply is highly beneficial for deposition of smooth and continuous InN nanometer-thin films. The time for purging the reactor between the precursor pulses and low deposition temperature are key factors to achieve homogeneous InN. The gas exchange dynamics of the reactor is further studied using computational fluid dynamics. According to our study, 320 °C is found to be the upper temperature where the dynamics of the deposition chemistry can be controlled to involve only surface reactions with surface species. The results highlight the promising role of the ALD technique in realizing electronic devices based on nanometer-thin InN layers.

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I. INTRODUCTION

III-nitrides (AlN, GaN, and InN) and their alloys are prominent semiconductor materials. AlN and GaN are highly important for optoelectronic applications and form the backbone in modern light-emitting diode technology. The revised bandgap of InN to 0.7 eV1 and its high electron mobility2,3 open up new opportunities, such as IR emitters, sensors,4 solar cells,5 thin-film transistors,6 and high-electron mobility transistors.7 As all such applications are based on heterostructures with homogeneous coverage of nanoscale active layers, highly controlled InN epitaxy is paramount to realize any application.

The growth of InN is problematic due to its low thermal stability; InN decomposes to In metal and nitrogen gas at about 550 °C.8 This has also been manifested by its high equilibrium pressure with nitrogen which has led to attempts at depositing InN at very high nitrogen pressures.9–11 Due to slow decomposition kinetics of ammonia (NH3),12 the most common nitrogen precursor in chemical vapor deposition (CVD), N/In ratios in the order of >104 have often been used in CVD of InN.13 Alternative N-precursors, such as dimethylhydrazine and tertiarybutylhydrazine, have been shown ineffective for the growth of InN.14 To allow more reactive N species, plasma-assisted approaches have been adapted in various growth techniques, such as molecule beam epitaxy,15–17 sputtering,18 and metalorganic CVD19 for the growth of InN.

We have previously hypothesized that steering the deposition chemistry fully to the surface could be an alternative route for low temperature CVD of InN as it could circumvent the problem of the slow kinetics in the gas phase decomposition of NH3. A CVD approach fully relying on surface chemistry is atomic layer deposition (ALD), which uses a sequential supply of the precursor gases to make the deposition chemistry surface controlled.20 ALD of crystalline InN films has been demonstrated using trimethylindium (TMI) and either nitrogen plasma21,22 or NH3 plasma.23 In addition, complex precursors with chelating ligands with In–N bonds have also been employed to prepare polycrystalline24 or epitaxial InN25 with NH3 plasma. Recently, we demonstrated that epitaxial and homogeneous wurtzite InN films on 4H-SiC substrate with a thickness down to 5 nm can be grown by using plasma ALD at 320 °C,26 which has never been reported by other growth techniques. ALD thus seems to be more promising than other techniques
to achieve homogeneous, epitaxial, nanometer-thin InN films. However, the understanding of why ALD holds such advantage over conventional thermal CVD is not properly addressed.

Here, we present an experimental study in which we compare the outcomes from ALD and conventional CVD process using TMI and NH₃ for both approaches. Our results demonstrate that nanometer-thin homogenous InN can only be deposited by a time-resolved CVD approach. The morphology of InN is heavily controlled by the purge time between the precursor pulses and the surface temperature. The transient gas exchange of the reactor is further studied using computational fluid dynamics (CFD). The deposition chemistry dynamics is suggested as a critical factor for the deposition of nanometer-thin InN layer which has the potential for device applications.

II. METHODS

A. InN deposition by ALD

A Picosun R-200 ALD system was employed to grow InN films using trimethyl indium, In(CH₃)₃ (TMI), and plasma discharged NH₃/Ar gas mixture. A detailed process development can be found in our earlier work. In this work, 4H-SiC (0001) was used as the substrate, which allows epitaxial growth of InN. The samples are loaded in the system without a load lock chamber. The plasma is generated within a quartz tube surrounded by a cylindrical rf coil. The heated substrate holder is located approximately 70 cm below the plasma source. N₂ (99.999%) and further purified by a getter filter) was used as the purge gas for the TMI (Pegasus Chemicals, Alpha grade). As previously described, a fill-empty pulsing scheme was used to get a sufficient flow of TMI in to the deposition chamber. The NH₃ plasma was ignited using a mixture of 50 SCCM NH₃ (99.9999%) and further dried with a getter filter) and 100 SCCM Ar (99.9997%) and further purified with a getter filter). The applied rf power for the NH₃ plasma is 2800 W. The rf power used in our work is much higher than the typical values (few hundred Watts) found in papers by other research groups. The power used stems from the design of our Picosun plasma ALD reactor where the ICP tube is located approximately 70 cm above the substrate holder in a relatively large volume reactor. Prior to the deposition of InN, the substrates were subjected to a 2-min plasma exposure (2800 W) with a 100 SCCM Ar + 50 SCCM N₂ plasma gas mixture, followed by 550 s, 1000 s interaction time. The InN ALD cycle comprises 4 s TMI pulse, 10 s N₂ purge, 12 s plasma exposure using 50 SCCM NH₃ + 100 SCCM Ar and 2800 W, and 6 s N₂ purge. The deposition temperatures were between 250 and 450 °C while the pressure was kept at 6 mbar for all growth runs. A small pressure fluctuation jumping from 6 to 10 mbar was observed while the TMI pulse is being introduced into the chamber. The pressure fluctuation was removed automatically within 2 s.

In some experiments which were done to demonstrate the importance of controlled surface reaction, the purge time between TMI and NH₃ plasma pulses was varied between 0.1 and 8 s with growth temperature of 320 °C. To facilitate the comparison on the surface morphology of initial deposition stage, 150 ALD cycles (with a nominal thickness of 6 nm at 320 °C and 10 s purge time) were performed for each condition.

For the sake of simplicity, we will refer to all samples deposited in this manner as samples deposited by ALD, even if the process conditions are such that a self-limiting behavior is not observed.

B. InN deposition by continuous CVD

An Aixtron/Epigress horizontal hot-wall MOCVD reactor was used to grow InN for comparison. TMI (SAFC Hitech, Epi-pure grade) and NH₃ (99.9990% and further dried with a getter filter) were used as the precursors, and 4H-SiC (0001) was used as the substrate. The deposition was performed with N₂ (99.999% and further purified by a getter filter) as process gas between 320 and 450 °C with continuous supply of both precursors for 30 min. The process pressure was kept at 50 mbar. The gas flows are 15 slm N₂, 2 slm NH₃, and 0.2 SCCM In(CH₃)₃, giving rise to a N/In ratio of 10 000. The growth time of 30 min combined with the total flow of TMI was chosen so that the same amount of TMI molecules was introduced into both the ALD and CVD experiments.

C. Film characterization

The surface morphologies of the InN samples were studied by high-resolution LEO 1530 Gemini field emission scanning electron microscope (SEM). The crystalline quality, thickness, and the macro-scale roughness were characterized by using x-ray diffraction (XRD) (PANalytical X’Pert Pro with a Cu-anode x-ray tube). Grazing-incidence XRD (GIXRD) and symmetric Bragg−θ scans were used to study the crystalline quality. The film thickness was determined by analyzing the x-ray reflectivity (XRR) results. The XRR data were fitted by software PANalytical X’Pert reflectivity using a two-layer model of the InN film and the substrate (InN/substrate).

D. Gas transport modeling of ALD reactor

The gas exchange dynamics of the TMI pulse-purge ALD-half cycle at 320 and 450 °C was modeled using computational fluid dynamics (CFD). The time-dependent concentration of the gaseous species in the TMI precursor pulse in the close vicinity of the substrate was investigated. The reactor geometry was discretized to a grid of 7 × 10⁸ elements. The governing equations of mass, momentum, and energy conservation were numerically solved together with the species transport equation using the finite volume solver Ansys Fluent 2020R2 (ANSYS Inc., Canonsburg, PA, USA). Due to the low vapor pressure of TMI in combination with the vacuum draw bubbler used for precursor supply, TMI was modeled as a passive concentration diluted in the carrier gas with an ideal, square-wave pulse. No chemical reactions were considered in the residence time model since the chamber volume is large (in the order of 10⁵ cm³ including the plasma cone) and the disk-shaped substrate holder where the flow impinges have a large gas volume directly on top due to the plasma cone [see Fig. 6(b)]. Hence, the bulk of the precursor is expected to pass over the substrate and through the chamber without any reactions and give negligible impact on the flow pattern. The main objective of this modeling is to study the time needed to purge the deposition chamber from TMI before the start of the NH₃ pulse to avoid overlap of
precursors; and due to the idealizations, the model cannot provide quantitative results such as growth rate. Additional computational details are given in the supplementary material.

III. RESULTS AND DISCUSSION

Figures 1(a)–1(c) show the surface morphologies of the resulting materials on SiC grown by both ALD (upper right parts) and CVD (lower left parts) approaches between 320 and 450 °C. For the cases of ALD, weak but homogeneous particle-like features can be seen on the substrate surface at 320 °C [Fig. 1(a)] and below (not shown). With increasing temperature, the grain size increases, and the surface coverage decreases, leading to a non-continuous film as illustrated in Fig. 1(b). At 450 °C and above (not shown), micro-size droplets/particles are found dominating the surface [Fig. 1(c)]. For the films from continuous CVD, barely any surface features are seen on the sample deposited at 320 °C [Fig. 1(a)]. At 400 °C, very low density of nanoparticles can be found on the substrate [Fig. 1(b)]. At 450 °C, separated, hexagonal grains with diameter ranging from a few nm to 50 nm can be observed [Fig. 1(c)]. Note that the dimension of the islands (few tens nm) deposited by CVD at 450 °C is much smaller than the droplets (1–2 μm) found on the sample deposited by ALD at 450 °C.

XRR has been performed to study the growth rate of all InN films. However, only samples grown by ALD at 320 °C revealed clear x-ray intensity fringes induced by the smooth and continuous film. The thickness fringes are barely seen for samples by ALD at 400 °C and thereafter disappear completely for all other samples (Fig. S1 in the supplementary material). We believe this is a consequence of high surface roughness observed in most of the studied samples as shown in Fig. 1. Meanwhile, the absence of noticeable thickness fringes also indicates that the existence of the buffer layer between InN and the substrate is negligible.

Crystalline, hexagonal InN can be found by XRD in the samples deposited by ALD as seen from the InN (0002) diffraction peak at 31.3° in Fig. 2(a). The satellite peaks in the vicinity of InN (0002), observed in the samples deposited at 320 °C, are thickness-correlated fringes. The presence of thickness fringe can be ascribed to heterojunctions with smooth surface and sharp interface. Such thickness fringes disappear in InN grown at higher temperatures due to higher surface roughness. Despite that the roughness increases with increasing temperature, the stacking coherence of the InN crystal was not affected as shown by the ω-scan against the InN(0002) between 320 and 400 °C. The full-width-half-maximum of the InN (0002) peak in ALD deposited films being nearly identical to the SiC substrate in the range of 20–50 arcsec [Fig. 2(b)]. For samples grown by ALD at 450 °C, the crystalline quality of InN is notably degraded by showing a broad FWHM of 600 arcsec. The additional peaks at 30.58° and 32.93° in the 2θ-ω scan can be indexed to In₂O₃ and metallic In, respectively. In droplets, as observed in Fig. 1(c), have been previously reported during growth of InN under elevated temperatures and In-rich conditions. Among the samples deposited by CVD, only the sample deposited at 450 °C shows XRD peaks that can be indexed to InN [Fig. 2(a)]. The FWHM of the InN (0002) in the ω-scan for the sample deposited by CVD is roughly 350 arcsec [Fig. 2(b)], which is significantly wider than for films prepared by ALD at lower temperatures in this work but is comparable to previously reported CVD films.

In Fig. 2(a), a down shift of the InN (0002) peak for approximately 0.15° with respected to the strain-free InN (0002) at 31.3°
is observed for all samples except the one by ALD at 320 °C [Fig. 2(a)]. Such a XRD peak shift is most likely due to strain caused by the lattice mismatch to the substrate and/or defects within the films. According to our earlier study, the InN layer becomes relaxed efficiently via the formation of misfit dislocations. A strain-free InN can be indexed for samples thicker than 5 nm by XRD. Instead of strain from the substrate, we suggest that oxygen could play a significant role for this. Owing to the very oxyphilic nature of In, seen from the standard Gibbs free energy of formation of $\text{In}_2\text{O}_3$ and $\text{InN}$, any residual O in the reactor and post-oxidation from air exposure can result in In$_x$O$_y$N$_z$ or even In$_2$O$_3$. Peak down shift with respect to InN (0002) in the XRD symmetric scan has been observed for InO$_x$N$_y$ before it turns completely into In$_2$O$_3$, which typically forms the cubic structure that is very different from the wurtzite structure of InN. According to EDX element mapping (Fig. 3), In, N, O, and Si are uniformly distributed for samples deposited at 320 °C by ALD [Fig. 3(a)] while an aggregation of oxygen together with In and N can be observed for samples deposited at 400 °C by ALD [Fig. 3(b)]. It has been found that the non-c plane surfaces are more favorable for oxygen incorporation in GaN. Assuming InN behaves similarly as GaN, the scattered small crystal grains seen in the sample deposited at 400 °C afford a higher surface area leading to enhanced oxygen incorporation and oxidation. We, therefore, suggest that the shift of the XRD peak in Fig. 2(a) is due to the formation of InO$_x$N$_y$ caused by the residual oxygen.
presented in the reactor during the growth and possibly also post-deposition air exposure.

The results in Figs. 1 and 2 show that the ALD process holds advantages over the conventional continuous CVD for deposition of InN. There are two major differences between the ALD and CVD approaches: the ALD approach uses plasma activation for the ammonia and the time-resolved supply of the precursors. The difference in the film deposition seen in Fig. 1 is likely explained by one or both differences. When a time-resolved precursor supply was used in the CVD reactor, no film was obtained at any temperature. When the plasma was disabled in our ALD reactor and a thermal ALD process for InN was attempted, no sign of InN deposition could be seen even at 450 °C. On the other hand, with plasma activation of the NH₃ pulse, InN can be deposited even at 180 °C in the ALD reactor, albeit with a reduced deposition rate and crystalline quality. This demonstrates the importance of the plasma activation of the NH₃ pulse. The utilization of plasma, affording more reactive N species, has been shown to increase the growth rate as well the crystalline quality of InN in both ALD and CVD systems. The plasma activation, therefore, seems paramount for the InN deposition at low temperature.

In continuous CVD processes, deposition of InN requires higher temperatures than in ALD (Figs. 1 and 2). The growth of InN by continuous CVD is suggested to depend on gas-phase reactions initiated from the adducts formed by mixing TMI and NH₃. Interestingly, pulsed flow of TMI into a continuous flow of NH₃ and a pulses of TMI and NH₃ with slight overlap have been reported to deposit good quality continuous InN films when the thickness exceeded 75 nm. Separating TMI and NH₃ was concluded advantageous for InN deposition in these studies. The separation of TMI and NH₃ should reduce the formation of adducts in the gas-phase and change the deposition chemistry. Consequently, we suggest that purge time is an important parameter which modulates reaction mechanism and its dynamics and strongly influences the deposited InN quality.

An ALD process needs to be done at relative low temperature to ensure the stability of the chemisorbed monolayer. Meanwhile, a sufficiently long purge time is also required to eliminate unwanted
chemical reactions of physiosorbed molecules. In fact, our ALD work was done in a temperature range higher than the typical InN ALD window of 150–300 °C.21,22,38–44 Considering the difference in film deposition observed in Figs. 1 and 2 at different temperatures and the thermal stability of TMI, our ALD of InN is likely to involve several In-species. TMI is considered to be stable up to 300 °C. Between 300 and 325 °C, TMI is reported to decompose fractionally to dimethylindium (DMI) and methyl radicals by homolytic cleavage of one In–C bond.39 DMI has also been suggested as a stable surface specie in ALD of InN from TMI.40 We, therefore, consider DMI as the main chemisorbed In-species on the surface below 325 °C and the overall surface reaction remains controllable. The reaction will eventually become uncontrollable by further increasing the temperature.

Excess precursors, reaction by-products, and physiosorbed surface species should be removed and evacuated by purging the ALD system with inert gas, N₂, in our experiments. To further study the influence of purge time and in the best possible attempt to mimic continuous CVD in our ALD reactor, we did InN ALD experiments with varied purge time. After the TMI pulse and the subsequent purge, the surface was exposed to NH₃ plasma, which nitridized the surface and immobilized all In species. The surface morphologies of InN ALD grown with different purge times are then a reflection of the behavior of the adsorbed In-species on the surface.

When 0.1 s purge time is used after the TMI pulse in a 320 °C deposition, a very high density (1.1 × 10¹¹ cm⁻²) of scattered particles is seen by top-view SEM [Fig. 4(a)]. A longer purge of 2 s results in a reduced density (1.6 × 10⁹ cm⁻²) of averagely larger particles [Fig. 4(b)]. With a long purge time of 8 s, a high-density particle-like feature (7.5 × 10¹³ cm⁻²) can be seen uniformly on the surface [Fig. 4(c)]. The particle-like surface feature is likely due to a Stránský–Krastanov growth mode where 3D growth takes over after a critical thickness of approximately 1 nm.41 The particle density, particle size, and inter-particle distance can be used to depict the distribution of adsorbed In-species on the surface, which changes with time, reflecting their mobility. According to our analysis, the average particle size increases from 5 to 16 nm [Fig. 4(d)] and the inter-particle distance also increases significantly [Fig. 4(e)] by increasing the purge time from 0.1 to 2 s. The increase in diameter of particles and inter-particle distance with longer purge time is an indication that the physiosorbed In-species on the surface are mobile until they are immobilized by the NH₃ plasma. According to the work by Muneshwar and Cadien, an ALD precursor pulse results in physiosorbed species and able to diffuse on the surface before being chemisorbed.42 With a long purge, all physiosorbed surface species should either chemisorb or desorb from the surface, leaving only chemisorbed In-species for nitridation. The growth per ALD cycle increases with increasing purge time: 0.1 s purge renders 0.22 Å/cycle, 2 s purge renders 0.31 Å/cycle, and 8 s purge gives 0.41 Å/cycle as shown in Fig. 5. Such a trend has been previously seen by Muneshwar and Cadien42 and suggests that the surface dynamics, producing chemisorbed species, is of very high importance for the film deposition. In addition, 8 s is adequate for the purge time in our case as the growth rate becomes saturated (Fig. 5). The SEM image shown in Fig. 4(c) reflects the outcome of InN deposition with an adequate purge time to have, presumably, no physiosorbed In-species. According to our analysis [Figs. 4(d) and 4(e)], the averaged diameter of InN crystal grains is 9 nm. This result is comparable to the value of InN grown by ALD using N₂ plasma on the GaN surface as reported by Woodward et al.43 A finite inter distance (smaller than 7 nm) between particles together with high particle density of 7.5 × 10¹⁴ cm⁻² indicates that the InN crystal grains are homogeneously distributed on the surface [Fig. 4(c)].

To better illustrate this coupled gas phase-surface dynamics in our ALD process, we utilized CFD modeling of the gas exchange in the reactor chamber. Figure 6 shows how the concentration of In species in the gas phase changes near the surface during the TMI pulse and the following N₂ purge. With a purge time of 0.1 s, the concentration of In species near the surface is only reduced slightly, while a 2 s purge time afford a significant but not complete removal of In species on the surface and in the chamber. A 2 s purge reduces the concentration to approximately one-eighth of the concentration just after the TMI pulse. This result coincides with the reduction in particle density observed between 0.1 and 2 s purge [Figs. 4(b) and 4(c)]. A purge time of 8 s and longer afforded a surface free from In species except those chemisorbed on the surface. It should be noted that the estimated necessary purge time is heavily dependent on the specific reactor design and carrier gas mass flow rate.

With increasing temperature, the degree of TMI decomposition increases and monomethyl indium (MMI) can form.45 MMI can undergo polymerization, forming poly-MMi, which has very low volatility.46 The formation of poly-MMi on the surface is not a self-limiting reaction and, thus, can continuously absorb In-species on to the surface as long as their concentration permits. Any formation of poly-MMi on the surface in an ALD process will, therefore, lead to formation of large crystal grains or droplets. At 450 °C, the degree of TMI pyrolysis is even higher and the last In–CH₃
bond in MMI can break. This results in elemental In as reported by Jacko and Price and evidenced in our XRD of films deposited at 450 °C by ALD [Fig. 2(a)]. The reductive environment of the NH3 plasma makes the formation of InN not favored for elemental, zero valent, In as an oxidation to In(III) is needed for formation of InN. Elemental In, on the other hand, can be easily oxidized upon exposure to both residual oxygen in the chamber and ambient after deposition, resulting in the observation of In2O3 in our XRD [Fig. 2(a)].

IV. CONCLUSION

We show that a time-resolved CVD approach by plasma ALD affords InN films with superior morphology and crystalline quality compared to a conventional, continuous CVD approach by standard thermal metalorganic CVD. We show that the key difference between the processes is not only the plasma activation of the ammonia but also the time-resolved precursor supply and how this is used to control the dynamics of the deposition chemistry. InN with smooth morphology can be obtained by the plasma ALD approach at 320 °C, provided that adequate time is given between the In- and N-precursors, presumably to allow physisorbed surface species to either chemisorb or desorb. We suggest that the detailed surface dynamics of In species is studied in detail by modeling to provide more insights. This study and previous findings in the literature demonstrate the importance of using a time-resolved precursor supply in CVD of InN in which the purge time plays an important role. It should also be noted that the best results in this study are outside the window where the deposition per ALD cycle is constant in temperature. Such observation suggests that a true, self-limiting ALD behavior is not the most critical factor but rather the dynamic precursor supply. Meanwhile, the experimentally optimal purge time and precursor pulse time should vary from reactor to reactor based on their geometry and volume.

SUPPLEMENTARY MATERIAL

See the supplementary material for full details on the XRR measurements and the CFD modeling.

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AUTHOR DECLARATIONS

Conflicts of Interest

The authors have no conflicts to disclose.

Author Contributions

C.-W.H and P.D. contributed equally to this work.
DATA AVAILABILITY

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

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