Incorporation Behaviors of In and Ga in the Two-Heater MOVPE Growth of InGaN Films

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The promising electronic and optical properties of InGaN alloy, such as high two-dimensional electron gas (2DEG) electron mobility, wide tunability of photon emission energy from 0.7 to 3.4 eV, have given rise to numerous applications in the past decade. These include high power and high electron mobility transistor (HEMT), visible light emitting devices, tandem solar cell, and solid state lighting.1–7 To realize a method that capable of growing device-quality III-V nitrides is essential. Among all growth methods, metal-organic vapor-phase epitaxy (MOVPE) has proved itself to be the most reliable and mature technique.1–20,21 Metal-organic vapor-phase epitaxy (MOVPE) has proved itself to be the most reliable and mature technique. In our previous work, we have employed a so-called two-heater MOVPE horizontal reactor, consisting of a pair of paralleled ceiling and substrate heaters,16 to grow InGaN films. We demonstrated its ability in preparing high In-content thick InGaN films with emission wavelength extended into infrared color region, and more importantly exhibiting good optical-quality and high spectral uniformity. For instance, a sample with x = 0.40 In,Ga1−xN film has a mean emission wavelength of 808 ± 6 nm and FWHM of 229 ± 4 nm at 18 K over the entire 2-inch wafer. Such a deviation of peak wavelength is relatively small, which can compete to that of the conventional MOVPE-grown nitride film emitted at 425 nm.19 Despite of the accomplishment, it has been reported that gas-phase parasitic reaction is one of the major side effects needed to be concerned in growing nitride, in particular InN and AlN, which could cause unequal depletion of group III species and result in composition fluctuation in the deposited films. This situation is considered to be severer in our two-heater MOVPE reactor due to the use of high ceiling temperature over the substrate. In this work, we therefore continue our study on InGaN growth with the aim of understanding of the individual incorporation behaviors of In and Ga in this type of reactor since it is fundamentally important and essential for CVD process.

Experimental

The InGaN samples studied here were grown on GaN/(0001) c-plane sapphire templates by using the aforementioned two-heater MOVPE horizontal reactor, where the ceiling and substrate temperatures are thermostated independently and well regulated by temperature controllers. High purity trimethylgallium (TMGa), trimethylindium (TMIn), and purified ammonia (NH3) were used as source precursors for Ga, In, and N, respectively, and nitrogen gas was used as the carrier gas. During the deposition, the disk that held the substrate was installed directly on the SiC-coated susceptor to ensure good thermal contact so that the substrate temperature is nearly the same as the susceptor temperature. After the growth, pieces of ~1 cm² samples cut from the center of 2-inch wafers were examined by scanning electron microscopy (SEM) to obtain the film thicknesses and by X-ray diffraction (XRD) in 0–20 scan mode of (002) reflection peak to estimate the In contents in In,Ga1−xN films, assuming the Vegard’s law is valid.

Results and Discussion

Since the temperature is known to be one of the key parameters for film growth, which affects the reaction routes and chemical species involved in the growth and parasitic reactions both homogeneously and heterogeneously, the first series in this study is the substrate-temperature series. In this series, the InGaN films were prepared by both the two-heater and the conventional growth modes. For the former, the substrate temperature Tsub was varied from 550 to 700 °C under a fixed ceiling temperature Tceil of 800 °C, and a reactor pressure of 200 mbar. The maintained molar flow rate for the TMGa, TMIn and NH3 are at 5.9, 8.8 μmol/min and 4.8 slm, respectively. For the latter, where the heater of the ceiling temperature is simply turned off, the growths were carried out at substrate temperatures between 575 and 700 °C with all other growth parameters kept unchanged. It is worth mentioning that inaccuracy arises in the substrate temperature, which is primarily due to the bowing effect of sapphire substrate during the deposition. The estimated inaccuracy is about ±2 °C in our interested temperature range, based on the results of Brunner et al.22

Figure 1 depicts the XRD 0–20 scan curves of the (002) reflection peaks for the above samples. The measured X-ray patterns are scanned from 31° to 35°, covering the InN and GaN diffraction peaks (31.3° and 34.5°). No In droplets were observed for above samples. As expected, when substrate temperature is increased, the In content is observed to
InGaN films on substrate temperature, grown by (a) conventional and (b) two-heater MOVPE growth modes. For the latter, the ceiling temperature was set at 800 °C.

descend accordingly from 0.36 to 0.17 and 0.37 to 0.07, respectively, for films prepared by the conventional and the two-heater MOVPE growth modes.

From the above X-ray data, we may at the first glance deem the incorporation behaviors of Ga and In in both growth modes differing insignificantly against substrate temperature. After introducing a parameter, a so-called incorporation efficiency, to elucidate the experimental outcomes, more clear pictures are revealed. The incorporation efficiency for each group III element here is defined as,

\[ \gamma_i = \frac{\gamma_{in}}{f_i}, \]

where \( \gamma_{in} \) and \( f_i \) are growth rate (\( \mu \text{m/min} \)) and molar flow rate (mol/min) of a given group III element \( i \). The individual growth rate \( \gamma_{in} \) is calculated from the film thickness and solid composition determined by SEM and XRD measurements. The derived results are drawn logarithmically against reciprocal substrate temperature \( T_{sub} \) and are shown in Fig. 2.

As can be seen in the Figure 2, the GaN incorporation efficiency \( \gamma_{GaN} \) displays a typical mass-transport limited growth behavior in the substrate temperature range of interest, regardless of the growth modes being used. These \( \gamma_{GaN} \) bear almost the same values at ~225 ± 14 \( \mu \text{m/mol} \). This implies that the use of ceiling heater at \( T_{cel} = 800^\circ \text{C} \) does not bring Ga loss during the InGaN growth. Thus, no gas-phase parasitic reactions can therefore be inferred between TMGa and NH3 when the two-heater MOCVD growth is employed to grow InGaN films under this growth environment.

More interesting results are observed for InN incorporation efficiency \( \gamma_{InN} \) in InGaN. As shown in Fig. 2, two growth regimes are observed, divided at a substrate temperature \( T_{sub} \approx 625^\circ \text{C} \). For substrate temperatures lower than 625 °C, the \( \gamma_{InN} \) appears to be insensitive to temperature, suggestive of the growth lying in the mass-transport growth regime, where nearly the same values are observed for both growth modes, similar to the case of the \( \gamma_{GaN} \). For temperatures above 625 °C, the corresponding \( \gamma_{InN} \) in InGaN readily enters into the high temperature growth regime, where the efficiency begins to drop considerably with increasing substrate temperature. One should notice that the \( \gamma_{InN} \) by the two-heater holds a steeper fall-off curve than the conventional one. This discloses a fact that the introduction of ceiling temperature brings an additional In loss for InGaN films when grown at high substrate temperatures.

For the two-heater growth mode, ceiling temperature \( T_{cel} \) perhaps is one of most attentive growth parameters for InGaN growth. To verify its effects, we thus conducted a second series of experiments at \( T_{sub} = 600^\circ \text{C} \), termed ceiling-temperature series, by ranging the ceiling temperature from 600 to 950 °C. In these runs, substrate temperature was cautiously chosen within the mass-transport limited regime with the purpose of avoiding any high substrate temperature effects to intervene the consequences.

Figure 3 shows the variations of incorporation efficiencies of GaN and InN on ceiling temperature. One can note that the incorporation of Ga in InGaN is again not affected considerably by the use of ceiling temperature. The \( \gamma_{GaN} \) remains nearly constant throughout the ceiling temperature range employed here, reassuring minimal or no parasitic reactions presented for TMGa at this growth ambient. In contrast, a distinct change is observed for InN. As illustrated in the figure, the InN incorporation efficiency \( \gamma_{InN} \) keeps nearly unchanged at ~96 \( \mu \text{m/mol} \) until \( T_{cel} \) reaches ~800 °C, where a visible drop occurs, signifying the existence of gas-phase parasitic reactions between TMIn and NH3 in this growth regime.

On the basis of above findings, we thus define the constant InN incorporation efficiency for \( T_{cel} \leq 700^\circ \text{C} \) as \( \gamma_{InN}^0 \) (~96 \( \mu \text{m/mol} \)) where complete absence of parasitic reactions is presumed. Then, the amount of In loss with respect to ceiling temperature due to the gas-phase parasitic reactions can be written as

\[ \gamma_{InN} = \gamma_{InN}^0 - \gamma_{InN}. \]

The result is shown in Fig. 4, where an increase of the In loss with increasing ceiling temperature is clearly observed.

Naturally, chemical kinetic reactions that govern the transitions from initial input gases to final product involve sequential steps of various processes in pursuit of the lowest energy state. Whether the
processes proceed via unimolecular or bimolecular, it is required that the participating active species have sufficient energy to surmount the energy barrier during the transition. To get more insights into the parasitic reactions associated with the In loss here, we further assume that there exhibits a rate limiting step in these chain reactions, which follows the first-order kinetics. Remind that all of the data used in this study are taken in the vicinities of centers of the substrates, thus the In parasitic loss \( \gamma_{\text{In}} \) for a given run in the deposition zone above the center of substrate can be expressed by integrating the reaction rate over the height \( h \) of the reactor

\[
\gamma_{\text{In}} = A \int_{0}^{h} \gamma_c \left( y \right) dy,
\]

in which \( \gamma_c \left( y \right) \) stands for the normalized profile of the concentration of active specie in this region, and \( k \) is the corresponding rate constant, depending exponentially on the temperature, which varies with the position along the central line perpendicular to the substrate due to the graded temperature distribution imposed by the use of ceiling temperature. The equation of \( k \) is given as

\[
k = A \exp \left( -\frac{E_a}{RT} \right),
\]

here \( A \) is the prefactor, which is related with the reactor geometry as well as the growth conditions in this study, \( E_a \) is its activation energy in unit of kcal/mol and \( R \) is the general gas constant.

For film deposition using a horizontal rectangular-shape reactor by the two-heater growth mode, the active species will deposit on both ceiling and substrate surfaces due to the existence of high operating temperatures, which causes the depletion of the reactants and hence zero concentrations of active species at both growing surfaces. Consequently, the normalized concentration profile of reactant and temperature distribution profile over the substrate under a fully developed flow condition can be approximated by the following equations

\[
c \left( y \right) = \sin \left( \frac{\pi y}{h} \right),
\]

and

\[
T \left( y \right) = \left[ T_{\text{sub}}^{\text{ref}} - \left( T_{\text{sub}}^{\text{ref}} - T_{\text{cel}}^{\text{ref}} \right) \frac{y}{h} \right] \frac{\pi}{h},
\]

respectively, based on the chemical deposition models developed by Mohammadi et al.\(^{24}\) and van de Ven et al.\(^{25}\) Here, \( y \) is vertical position perpendicular to gas flow direction and \( \beta \) is a constant, equals approximately to 0.7.\(^{26,27}\)

By substituting the temperature profile Eq. 5 into Arrhenius-type form of rate constant \( k \), and taking integration with sinusoidal concentration profile over the \( y \)-direction, we now have

\[
\gamma_{\text{In}}^P = A \int_{0}^{h} \exp \left( -\frac{E_a}{RT \left( y \right)} \right) \sin \left( \frac{\pi y}{h} \right) dy.
\]

By inserting sets of the experimental \( \gamma_{\text{In}}^P \) and corresponding \( T_{\text{sub}} \) and \( T_{\text{ceil}} \) in Fig. 4 into above equation, the prefactor \( A \) and activation energy \( E_a \) for In parasitic reactions can then be determined, which are of \( \sim 4.8 \times 10^8 \mu \text{m}^{-2} \cdot \text{mol}^{-1} \) and 34.2 \pm 0.1 kcal/mol, respectively.

Before further analysis of the experimental data, it is worthwhile to pause at this point and have a brief review of parasitic reactions involved in nitride epitaxy. When M(CH\(_3\))\(_3\) and NH\(_3\) are used as source precursors, where M = Al, Ga, or In, two types of parasitic reactions were proposed.\(^{28}\) No matter which one is dominant, the Lewis acid-base adducts (CH\(_3\))\(_3\)M:NH\(_3\) are agreed to be the first product formed upon the precursor mixing at the entrance inlet.\(^{29}\) When these adducts undergo irreversible decomposition into amide (CH\(_3\))\(_2\)MN followed by imine (CH\(_3\))MNH intermediates, further association (oligomerization) could bring them to initiate nanoparticle formations. Such a reaction channel is referred to as “adduct parasitic pathway”. As a sub-structure, if the Lewis acid-base adducts proceed reversible dissociation, they detach back into M(CH\(_3\))\(_3\) and NH\(_3\) completely at temperatures \( \geq 300 \)°C.\(^{30}\) The M(CH\(_3\))\(_3\) then proceed chains of homogenous homolysis fissions to form radical fragments as temperature is elevated.\(^{3,12}\) Further increasing the temperature may cause parts of them to recombine together, which act as nucleation, and provoke agglomeration or coalescence processes for particle growth.\(^{30}\) This parasitic reaction route is termed as “radical parasitic pathway”.

We consider the gas-phase In loss in our InGaN study is by means of “radical parasitic pathway”. This is rational and the explanation is given as follows. Because of weak dative bonding energy, the TMIn:NH\(_3\) adducts formed at the inlet detach back into TMIn and NH\(_3\) completely at \( \sim 269 \)°C.\(^{31}\) When TMIn arrive deposition zone over the substrate, they subsequently release two CH\(_3\) radicals and transform into MMIn molecules (In(CH\(_3\))\(_3\)). At temperatures higher than 480°C, gas-phase MMIn can either associate into (CH\(_3\))\(_3\)In or into M(CH\(_3\))\(_3\) radicals and form into IMIn (In(CH\(_3\))\(_3\)) at temperatures \( \geq 365 \)°C.\(^{32}\) Because of the unlikeliness of formation of (In(CH\(_3\))\(_3\)), any MMIn molecule that is formed would finally decompose into In atom at sufficient high temperatures.\(^{33}\) During the deposition, increasing the temperature certainly would add more In atoms into gas phase; once the In partial pressure reaches super-saturated state, nucleation initiates, followed by an almost immediate vapor condensation on nuclei surface to form In clusters,\(^{34}\) which is what the gas-phase In loss comes from. On the basis of the nature of radical pyrolysis reactions of TMIn, in conjunction with the fact of close agreement in between the activation energy of cleavage of last In-CH\(_3\) bond and the energy of the In parasitic loss derived from Fig. 4, it could be implied plainly that atomic In play an integral role in participating the particulate formation and the decomposition of MMIn is most likely the rate-limiting step of the consecutive parasitic reactions for In during InGaN MOVPE growth.

Beside the gas-phase In parasitic loss, it is generally accepted that the high-temperature loss occurred on the growing interface is another major factor responsible for the In loss when InGaN growth is performed at high substrate temperatures (\( T_{\text{sub}} \geq 625 \)°C). To examine the effects of such an In loss on InGaN growth in the substrate-temperature series, we thus include the parasitic loss into the equation of the InN incorporation efficiency in Fig. 2, which relates to the high temperature In loss by

\[
\gamma_{\text{In}} = \gamma_{\text{In}}^{0} - \gamma_{\text{In}}^{P} - \gamma_{\text{In}}^{HT}.
\]

Similar to the gas-phase parasitic reactions, the high temperature loss is assumed to be thermally activated process and follow the first-order kinetics

\[
\gamma_{\text{In}}^{HT} = B \exp \left( -\frac{E_{\text{HT}}}{RT_{\text{sub}}} \right),
\]
where $B$ is a constant, $E_{\text{HT}}$ is the corresponding thermal activation energy. After substituting sets of the measured $\gamma_{\text{AlN}}$ and calculated parasitic In loss $\gamma_{\text{InN}}$ got from the predetermined $E_{\text{HT}}$ and $A$, the activation energies $E_{\text{HT}}$ of high temperature loss at the growing interface are found to be 28.0 ± 0.1 and 21.7 ± 0.1 kcal/mol for the conventional and the two-heater growth modes, as shown in Fig. 5, respectively. The similar activation energy seems to imply identical mechanism ruling the high-temperature In loss here, no matter the conventional or the two-heater growth method is employed. Moreover, these values are comparable to the reported activation energies of InN decomposition, 25–26 kcal/mol,36–38 suggesting that the steps involved in the cleavage of In-N bonds in InN follow almost the same as that in InN, does not influence imposingly by the existence of adjacent Ga-N bonds. The calculation also indicates that at a reactor pressure of 200 mbar the gas-phase parasitic reaction can be ignored for the conventional InGaN MOVPE growth, since it contributes to < 3% In loss in reference to $\gamma_{\text{AlN}}$ when $T_{\text{sub}}$ is less than 700°C.

Note that the derived activation energy for high-substrate temperature In loss is substantially lower than the binding energy of In-N bond (44.4 kcal/mol).39 This points to a truth that the outermost surface In atom on the growing interface is not cleaved directly from In-N bond itself, it should react with other species before leaving the surface, which are capable of lowering the activation energy for bond breaking. It has been stated that the GaN growing surface under NH$_3$ ambient is either hydrogenated or nitridated by adsorbed H or NH$_3$ generated from the heterogenous decomposition of NH$_3$,40,41 by which could bring marked influences on surface growth chemistry. Because of the similarity in chemical properties of GaN and InN, same situation is believed to be also present in InGaN growth.

Using the first-principle calculations based on the density function theory, Togashi et al. reported that the desorption energies of hydrogenated and nitridated surfaces for InN are 23.0 and 66.7 kcal/mol for InH and NH$_3$, respectively.42 The measured activation energies here (21.7 ± 0.1 and 28.0 ± 0.1 kcal/mol) seems to indicate that the high-temperature In loss is connected to the desorption of InH, not the desorption of NH$_3$ from the growing surface. The existence of terminated-InH can be attributable to the presence of large concentration of active surface H atoms produced from the decomposition of adsorbed NH$_3$.

Finally, the dependences of incorporation efficiencies for both InN and GaN in InGaN on reactor pressure were also investigated at a substrate temperature of 625°C, since if gas-phase parasitic reactions actually occur, normally a notable drop in incorporation efficiency with increasing pressure should be observed. One representative example is MOVPE AlN growth using TMAl and NH$_3$ sources, where the AlN incorporation efficiency falls drastically to almost zero as pressure is increased to ~400 mbar.33,44 Thus, in this series two sets of experiments were performed; one at ceiling temperature of 600°C used as a reference and the other at 950 °C on purpose to observe the characteristics of parasitic reactions on pressure linked to In and Ga.

As depicted in Fig. 6, when the growths were conducted at $T_{\text{sub}} = 625\text{°C}$ almost no variation in InN incorporation efficiency is yielded at $T_{\text{cel}} = 600\text{°C}$. The experimental evidence indicates the parasitic reaction is insignificant under this circumstance, as can be foreseen by above discussion. Instead, the $\gamma_{\text{AlN}}$ at $T_{\text{cel}} = 950\text{°C}$ show a slow declining characteristic with increasing reactor pressure and tend to saturate at high operating pressures. Obviously, this slow declining feature in InN departs considerably from the case in AlN, where a sharp fall-off is reported.43,44 We attribute such a discrepancy to the different mechanisms behind their parasitic reactions. As proposed by Coltrin et al.,45 the gas-phase particle growth for TMAl in AlN growth favors “adduct parasitic pathway”, where the formation of particle nucleus requires the collision of two Al(CH$_3$)$_2$NH$_2$ molecules, which is categorized as a bi-molecular process. That means the parasitic reaction rate depends on the result of square of Al(CH$_3$)$_2$NH$_2$ partial pressure, and hence the square of reactor pressure under given gaseous molar flow rates. Consequently, this yields a pronounce reduction in AlN incorporation efficiency with increasing reactor pressure. On the contrary, the parasitic chemical reactions for TMIn undergoes “radical parasitic pathway”, which forms In clusters to deplete the In source in the gas phase at high temperatures. The relevant rate limiting step, as mentioned earlier, is the removing of the last CH$_3$ from energetic MMIn (In(CH$_3$)$_3$) itself. Such a type of reaction is a unimolecular process, of which the reaction rate is proportional to the first power of the partial pressure of reacting species. This explains for the less dependency of InN incorporation on reactor pressure in InGaN growth in comparison with AlN.

Compared to InN, more interesting and unusual results were obtained for GaN incorporation efficiency. For the case of ceiling temperature 600°C, the $\gamma_{\text{GaN}}$ remains unchanged throughout the entire reactor pressure range, agreed well with the prediction based on the simple diffusion growth model,34 reconfirming the absence or unimportance of gas-phase parasitic reactions for TMGa reactant under this growth ambient. Nonetheless, at $T_{\text{cel}} = 950\text{°C}$ the $\gamma_{\text{GaN}}$ is no longer a constant. The value of $\gamma_{\text{GaN}}$ jumps firstly at 50 mbar, when compared to that at 600°C, and then descends gradually with reactor pressure with a tendency analogous to that in $\gamma_{\text{AlN}}$ at 950°C. The likeness in declining curve not only signifies the presence of premature

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**Figure 5.** Arrhenius plot of the high-temperature InN loss of InGaN films grown by the two-heater ($T_{\text{cel}} = 800\text{°C}$) and the conventional MOVPE. Standard deviation of activation energies and the error bars are due to the inaccuracy of the substrate temperature.

**Figure 6.** (a) GaN and (b) InN incorporation efficiencies of InGaN films as a function of reactor pressure prepared at $T_{\text{sub}} = 625\text{°C}$ with $T_{\text{cel}} = 600$ and 950°C.
reactions for TMGa at $T_{\text{gas}} = 950$ °C, but also is suggestive of a similar parasitic chemistry behind Ga and In. As a matter of fact, the pyrolysis of TMGa, resembling to TMIn in many aspects, proceeds also by radical homolysis. The last methyl of TMGa is not cleaved in the gas phase until the temperature reaches $\sim 800$ °C, which suggests that the clustering of Ga should take places at still higher temperatures. This statement is consistent with the observation by Creighton and co-workers, who using in-situ laser light scattering unveiled the formation of Ga-bearing nanoparticles during GaN growth functioning in the temperatures of 900--1000 °C.

The jump of $\gamma_{\text{GaN}}$ at 50 mbar mentioned above certainly shows the presence of the driving forces advantageous to Ga solid incorporation at $T_{\text{gas}} = 950$ °C. Such an improvement in $\gamma_{\text{GaN}}$ definitely is connected to the employment of high ceiling temperature as well as low reactor pressure imposed during the deposition. The origins accounted for it are not clear at present yet. One likely cause is thought to be the pressure imposed during the deposition. The origins accounted for portions of gaseous Ga(CH$_3$)$_x$ molecules are converted into atomic Ga, at 50 mbar where parasitic reactions are greatly suppressed, large portions into atomic Ga. The lighter molecular weight of Ga, together with its better diffusion capability, and almost zero surface kinetic barrier when react with adsorbed NH, or NH$_2$. Consequently, an improved Ga incorporation efficiency is resulted.

The improvement in $\gamma_{\text{GaN}}$ at 50 mbar may have a direct impact on the efficiency of parasitic chemistries of the Ga and In in our MOVPE InGaN growth. That both an enhanced and a declined growth conducts with reactor pressure are veiled the formation of Ga-bearing nanoparticles during GaN growth with its better diffusion capability, and almost zero surface kinetic barrier when react with adsorbed NH, or NH$_2$. Consequently, an improved Ga incorporation efficiency is resulted.

Summary

In summary, we have conducted sets of InGaN growths in our two-heater MOVPE reactor, using TMGa, TMIn, and NH$_3$ as source precursors and N$_2$ for growth ambient. From considering both the concentration and temperature profiles above the substrate, the rate of the In parasitic-loss mechanisms is found to exhibit an activation energy $\sim 34.2 \pm 0.1$ kcal/mol. Becoming significant for temperature $\geq 950$ °C, the rate-limiting step of parasitic processes is plausibly related to the ceiling temperature and low reactor pressure during the deposition. The last methyl of TMGa is not cleaved at $950$ °C, in particular at 50 mbar where parasitic reactions are greatly suppressed, large portions of gaseous Ga(CH$_3$)$_x$ molecules are converted into atomic Ga, which have a lighter molecular weight, better diffusion capability, and almost zero surface kinetic barrier when react with adsorbed NH, or NH$_2$. Consequently, an improved Ga incorporation efficiency is resulted.

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