

Study of growth conditions effect on GaN doping with carbon from propane and methane

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A comprehensive study of intentional GaN carbon doping from propane and methane during MOVPE was performed in a wide range of growth conditions using both hydrogen and nitrogen carrier gas with growth rate varies from 0.8 to 62 $\mu\text{m/h}$. Carbon concentration raise with growth rate was revealed both precursors. For the same conditions carbon incorporation from methane is about one order lower than from propane. However, methane produced by trimethylgallium pyrolysis was revealed to be an important source for background carbon incorporation, especially at high growth rate. Character of the dependencies of carbon incorporation on concentration of carbon precursor and ammonia is significantly different for nitrogen and hydrogen carrier gases. Temperature dependencies of carbon incorporation from methane and background incorporation are similar while propane is more effective precursor at high temperature.

Keywords: Doping, Metalorganic vapor phase epitaxy, Nitrides.

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1. Introduction

Doping of GaN epilayers with carbon is widely used for growth of semi-insulating buffers for electronic applications [1–4], significantly influencing properties of High Electron Mobility Transistors (HEMT) [2,5–7]. Carbon in GaN is an amphoteric impurity and may substitute gallium (C_{Ga}) or nitrogen (C_{N}) in the lattice. It looks like semi-insulating properties of GaN:C originate from autocompensation through interplay of $C_{\text{N}}-C_{\text{Ga}}$ states incorporated during growth with comparable concentrations [7]. However, the details of this process are unclear yet.

Commonly used MOVPE process utilizing trimethylgallium (TMGa) as a precursor for both gallium and carbon to grow carbon-doped GaN (background doping) suffers from principal disadvantage: growth conditions necessary for high carbon concentration are far away from promoting high material quality. Potentially this problem diminishes if doping is performed using a separate carbon precursor.

It was published that high-quality GaN:C epilayers and HEMTs with intentionally doped GaN:C buffer may be grown using propane [8–11] or other hydrocarbons [8,12–15] as carbon precursors in MOVPE. Using of methane [16,17], ethylene [18], propane [19], and pentane [20] for carbon doping of GaN during HVPE was also reported. However, most of publications discuss material properties and/or device applications. Still little is known about peculiarities of carbon incorporation in GaN from hydrocarbons. At the same time, detailed experimental study of this process may be very useful in the development

of CFD models, optimization of epitaxial condition and reactors design.

In [21] we have reported a study of GaN doping using propane as precursor in very wide range of reactor conditions. It was revealed that carbon incorporation depends on process parameters in a manner, strongly differs from doping with common impurities substituting gallium atoms like silicon, magnesium, iron etc., which is not so strange for amphoteric impurity.

First of all, carbon incorporation is strongly increased with the growth rate. It should be stressed, that increase of TMGa flow results in strong increase of carbon incorporation from propane in spite of reduction of propane to TMGa ratio. It means that this ratio widely used as a process parameter is totally senseless.

Another unusual behavior is a dependence of carbon incorporation on propane concentration. At low and moderate growth rates carbon concentration in GaN is roughly proportional to propane concentration to the power 3/2. With increase of ammonia concentration and, especially, of growth rate, a linear component of the dependence becomes prominent. It was revealed, also, that carbon incorporation efficiency is inversely proportional to the square of ammonia concentration and weakly dependent on reactor pressure.

In [21] we have proposed that the observed effects may results from additional incorporation of carbon from methane generated by propane pyrolysis [22], and from the interaction of hydrogen with GaN resulting in formation of gallium coverage on the surface and quazi-equilibrium nature of the epitaxial process [23].

This study was performed to get more experimental data and clarify assumptions mentioned above. GaN doping from methane was studied in much wider range of reactor conditions than in [8]. Carbon incorporation from methane and propane using hydrogen and nitrogen carrier gas was compared. It should be stressed that we study the overall incorporation of carbon without separation of incorporation in various sites.

2. Experimental Details

GaN:C epitaxial layers were grown on (0001) sapphire substrates in a Dragon-125 MOVPE system with inductively-heated horizontal-flow reactor with 3×2 -inch or 1×100 mm substrates capacity. The growth temperature was measured by two pyrometers focused on the backside of the thin (11 mm) graphite susceptor. Growth rate was measured by multi-laser-beam *in-situ* optical reflectance monitoring (ORM). Trimethylgallium (TMGa), ammonia, propane, methane, hydrogen and nitrogen were used as precursors and carrier gases. Growth was initiated using a standard low-temperature GaN nucleation layer followed with $1 \mu\text{m}$ -thick GaN undoped epilayer grown at temperature of 1100°C and pressure of 800 mbar. These stages were identical for all structures studied in this work. Then carbon-doped GaN layers were grown. Reactor conditions for GaN:C growth were varied around our standard ones for undoped or silicon-doped GaN layers ($T = 1100^\circ\text{C}$, NH_3 concentration in the gas phase 0.35–0.37, total gas flow ~ 13 slm, growth rate $\sim 6 \mu\text{m}/\text{h}$) as described below. Most of GaN:C epilayers were grown with 100 mbar reactor pressure to allow reaching of high GaN growth rate (up to $62 \mu\text{m}/\text{h}$) by simple rise of TMGa flow. When doping efficiency with pressure was studied, all gas flows remains unchanged. When ammonia concentration was varied, total gas flow through the reactor was kept constant by substitution of ammonia by carrier gas (and vice versa) to keep other precursors concentration unchanged.

SIMS study was performed using a CAMECA IMS-7F instrument. Cs^+ ions were used as primaries while secondary negative carbon ions were used as analytical ones. Quantitative analysis was done using calibration to a GaN:C implanted standard [24]. Calibration of sputtering rates was done by measuring the depth of the sputtering craters by a mechanical stylus profilometer AMBIOS XP-1.

Samples for SIMS measurements were composed of a number of GaN:C layers grown under different conditions separated with undoped layers serving as markers for the measurements. Every dependence described below was revealed from data obtained from one multi-layer epitaxial structure to exclude influence of measurements precision on the character of the dependence.

Methane, propane, TMGa, ammonia concentrations in the gas phase and carbon atomic concentration in GaN are denoted later in text, figures, and equations as X_{CH_4} , $X_{\text{C}_3\text{H}_8}$, X_{TMGa} , X_{NH_3} , and C_{GaN} , respectively.

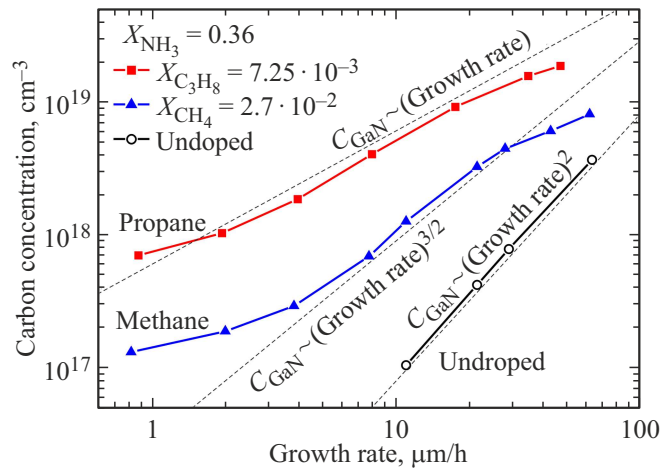


Figure 1. Dependence of carbon concentration on GaN growth rate for methane and propane doped GaN and undoped layers.

3. Results

In [8] it was reported that efficiency of carbon incorporation from methane is very low. However, as we have observed a 40 times rise of propane doping efficiency with increase of GaN growth rate [21], similar effect for methane was expected. The dependence of carbon concentration on growth rate for GaN doped from methane is presented in the Figure 1 together with the data for propane-doped and undoped epilayers. Note, that increase of growth rate was performed by rise of TMGa flow only, and all other gas flows including dopants were unchanged. Thus, for faster grown layers ratio of doping gas flow to TMGa flow was reduced. Note also, that for the data presented in the Figure 1, methane concentration in the gas phase was 3.7 times higher than propane concentration. As it can be seen carbon concentration in methane-doped GaN rises with growth rate even faster than in propane-doped ones. However, even for very fast growth ($40\text{--}60 \mu\text{m}/\text{h}$) doping from methane is approximately 10 times less effective than from propane.

Our analysis of data reported in [8] allows concluding that carbon incorporation from CH_4 is proportional to X_{CH_4} in $3/4$ power. However, our experiments have shown that carbon concentration in GaN is linear with methane concentration in the gas phase for high and low growth rate, for both H_2 and N_2 carrier gases (see Figure 2). The only difference between these two carrier gases in terms of carbon incorporation from methane is an increase of a background level in the case of nitrogen.

All this means that methane produced by propane pyrolysis cannot significantly influence doping process and should be accounted as small correction factor only. However as it will be shown below, data presented in the Figures 1 and 2 proves that doping from methane produced by TMGa pyrolysis may play a very important role in GaN background doping with carbon.

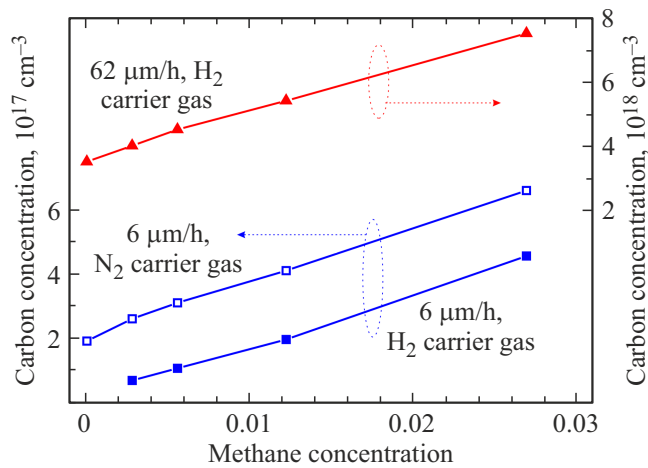


Figure 2. Dependence of carbon concentration on methane concentration. Note the difference in left and right axis scales.

In contrast to the case of doping from methane, change of the carrier gas type principally changes the character of GaN doping from propane. In the Figures 3, *a* and 3, *b* one can compare the $C_{\text{GaN}}(X_{\text{C}_3\text{H}_8})$ dependencies for GaN:C epilayers grown with moderate growth rate under low NH_3 concentration using two different carrier gases. Following $3/2$ power law in the case of hydrogen carrier gas is changed to strict linear dependence in the case of nitrogen.

It was revealed that the character of the dependence of carbon concentration in GaN on ammonia concentration in the reactor also strongly depends on the type of carrier gas. In [21] we have reported that if hydrogen is used as a carrier gas and propane is used for doping, $C_{\text{GaN}} \sim [X_{\text{NH}_3}]^{-2}$ for any GaN growth rate studied (data points are shown in gray in Figure 4). In contrast, if nitrogen is used as a carrier gas, carbon concentration is inversely proportional to ammonia

concentration. The same character of the dependence is observed for doping from methane for both types of carrier gas and for background carbon incorporation (only hydrogen case was studied) [21,25]. So, we can conclude that generally $C_{\text{GaN}} \sim [X_{\text{NH}_3}]^{-1}$, and $C_{\text{GaN}} \sim [X_{\text{NH}_3}]^{-2}$ law is specifically attributed to the very practically important case of propane as a precursor and hydrogen as a carrier gas.

One more difference between propane and methane as carbon precursors is a dependence of carbon incorporation on reactor pressure. In [21] we have reported that if doping is performed from propane, carbon concentration is independent of reactor pressure. In the case of doping from methane carbon concentration increases with pressure (Figure 5). The effect is not very strong (2 times for pressure rise from 100 to 400 mbar) but noticeable.

We have also studied an effect of reactor temperature on carbon incorporation in wider range that presented in [8]. To increase background carbon concentration ammonia concentration was reduced to 0.175 from our typical value of 0.35, and GaN growth rate was reduced to $3.5 \mu\text{m/h}$ to improve GaN planarity at reduced temperatures. In this set of experiments TMGa flow was adjusted ($\pm 15\%$ in the whole temperature range) to keep growth rate constant with temperature variation. Carbon concentration dependence on temperature for undoped, propane, and methane doped GaN are presented in the Figure 6, *a*. It is clearly seen that temperature dependencies for methane and background doping are very similar and carbon concentration rapidly drops with temperature. In contrast, the reduction of doping efficiency with temperature rise for propane is much less pronounced in agreement with short-range results of [8]. A variation of doping efficiency with temperature is shown in the Figure 6, *b* for both propane and methane. Added carbon concentration there means the difference between measured C_{GaN} and background concentration for the same temperature. The data for 900°C are very rough due to

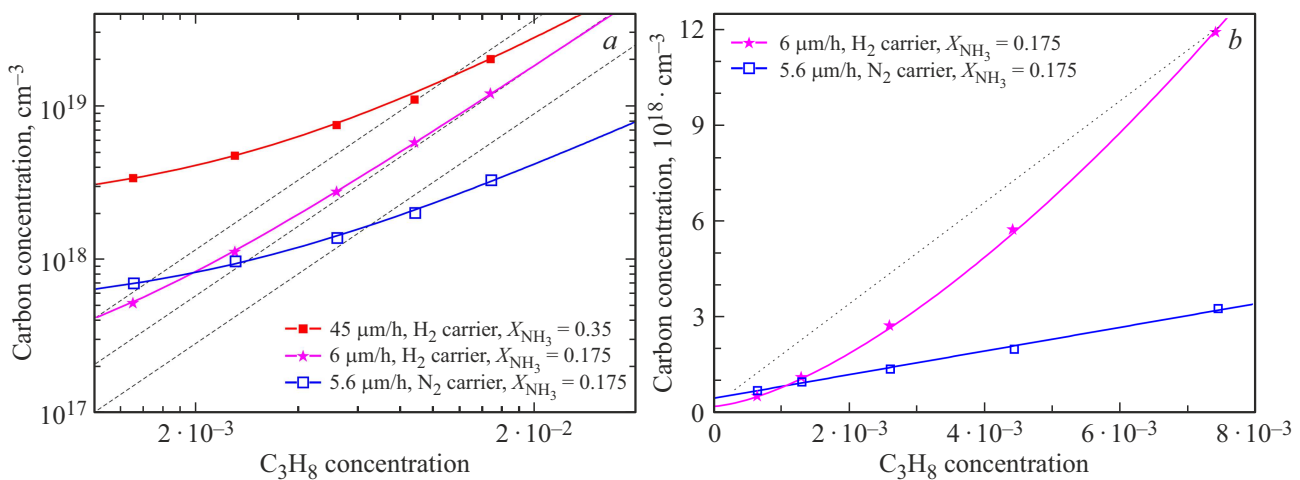


Figure 3. Dependence of carbon concentration on propane concentration for hydrogen and nitrogen carrier gases in logarithmic (*a*) and linear (*b*) scales. In (*a*) dashed lines denote dependences $C_{\text{GaN}} \sim [X_{\text{C}_3\text{H}_8}]^{3/2}$, and solid lines are given as eye guide. In (*b*) solid lines denote linear dependence for nitrogen carrier gas and $C_{\text{GaN}} \sim [X_{\text{C}_3\text{H}_8}]^{3/2} + C_{\text{background}}$ for hydrogen carrier gas; dashed line represents a linear dependence.

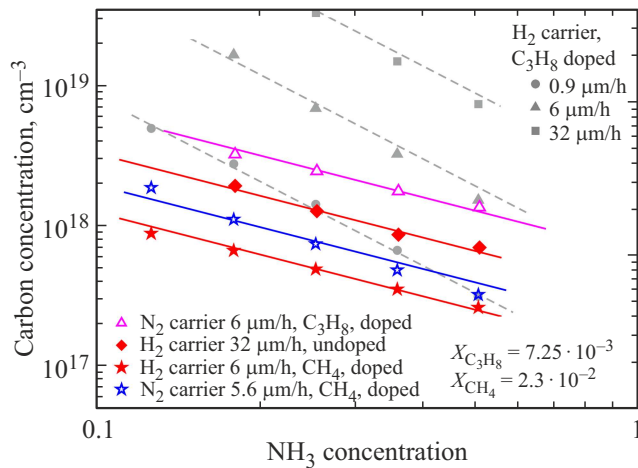


Figure 4. Dependence of carbon concentration on NH_3 concentration. Dashed and solid lines denotes the dependences $C_{\text{GaN}} \sim [X_{\text{NH}_3}]^{-2}$ and $C_{\text{GaN}} \sim [X_{\text{NH}_3}]^{-1}$ with various proportional coefficients, respectively.

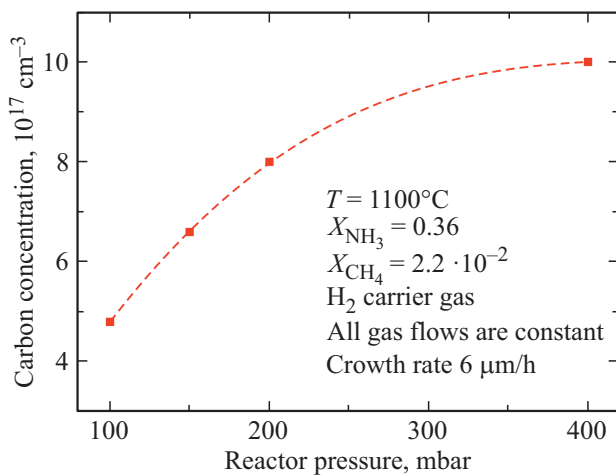


Figure 5. Dependence of carbon concentration in methane-doped GaN layers on reactor pressure.

low value of added carbon concentration comparing to the background level. However, even with this accuracy it is clear that propane is most effective at the temperature about 1000°C , and with temperature reduction its efficiency is reduced. For methane this maximum is not clear, but if exists it corresponds to lower temperatures. It was also revealed that for reduced temperatures (1000°C and below) $C_{\text{GaN}} \sim X_{\text{C}_3\text{H}_8}$, superlinearity develops only at higher temperatures.

4. Discussion

Presented data allows some speculations on the process of GaN background doping with carbon.

It is widely recognized that during MOVPE in the presence of hydrogen prior to reaching the wafer TMGa de-

composes to MMGa by methane elimination and thus every molecule of TMGa injected into the reactor produces two molecules of methane. However, it is commonly believed for GaN and proved for GaAs [26] that carbon background incorporation occurs only from monomethylgallium and methane is not involved in the background doping. We can doubt against this statement.

Data presented in the Figures 6, a and 6, b corresponds to relatively low GaN growth rate. TMGa concentration was $2.4 \cdot 10^{-4}$ (TMGa flow $140 \mu\text{mol}/\text{min}$, total flow of 13 slm). Thus, concentration of methane generated from TMGa is $4.8 \cdot 10^{-4}$. In these experiments at $X_{\text{CH}_4} = 6.6 \cdot 10^{-3}$ and 1050°C additional carbon concentration is $3.1 \cdot 10^{17} \text{cm}^{-3}$. Carbon incorporation from methane is linear with methane concentration, so we can calculate the concentration of carbon incorporated into GaN from methane generated by TMGa pyrolysis to be $2.3 \cdot 10^{16} \text{cm}^{-3}$. This value is about 10% of the background concentration ($2 \cdot 10^{17} \text{cm}^{-3}$) at this conditions, typical for GaN growth. So, methane is not a primary source of carbon, but should be accounted e.g. in detailed analysis of the growth process. We can assume that this proportion will be independent on X_{NH_3} because doping from methane and background doping demonstrate the same dependence on ammonia concentration.

Moreover, at fast growth the role of this „background“ methane becomes really significant. The same calculations for data corresponds to fast growth in the Figures 1 and 2 demonstrate, that in the growth rate range of $10\text{--}60 \mu\text{m}/\text{h}$ methane, generated from TMGa, is responsible for 40–60% of total background carbon concentration.

So, carbon background incorporation occurs not only from monomethylgallium. At elevated growth rate when doping from methane is relatively efficient, there is no big difference between incorporation rate of carbon atoms coming to the surface as metallorganic molecules or as methane already generated from TMGa.

The next question is why carbon incorporation from propane so strongly depends on the type of carrier gas. Generally, it may results from change in gas the phase chemical reactions in the reactor volume or in the surface chemical reactions. Some speculations on this issue are possible by combining our experimental data with a few publications of other groups.

Pyrolysis of propane (pure, diluted with argon and hydrogen) in the temperature range of $656\text{--}746^\circ\text{C}$ was studied in [27]. The temperature of gas in the hot zone of horizontal MOVPE reactor is more or less in this range. The reaction time in these experiments is much higher than in our MOVPE reactor but we do not have other data for comparing. It was shown [27] that propane pyrolysis products are ethylene, propylene, ethane and methane. Doping from methane is ineffective so it will not be discussed below. At 656°C the main product of propane pyrolysis are ethylene and propylene, produced in nearly equal quantities. Production of ethane is only a few percent of ethylene. With temperature raise the

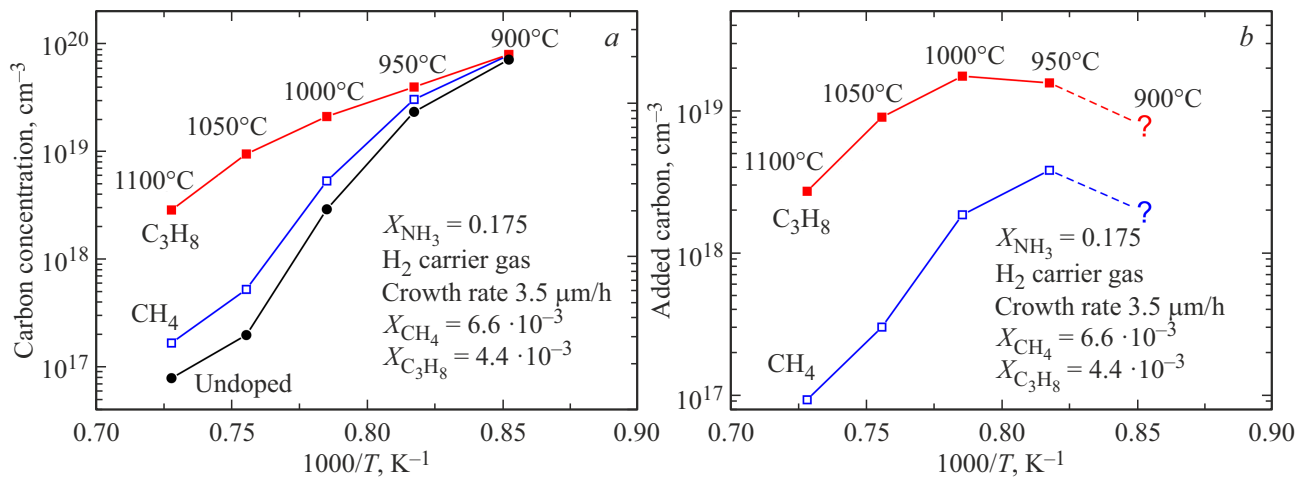
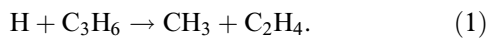


Figure 6. Dependences of carbon concentration (a) and differences of carbon concentration in doped and undoped layers (b) on temperature for doping from propane and methane.

pyrolysis rate is increased, and ethylene becomes the main product of the process. At 746°C production of ethylene is 2.7 times higher than those of propylene, production of ethane remains low. Dilution of propane with argon does not noticeably influence the process at any temperature in the range. At low temperature hydrogen does not affect the process also. In contrast, at 746°C the presence of hydrogen results in increase of ethylene to propylene ratio above 5. Production of ethane is increased a little bit but remains very low ($\sim 5\%$ of ethylene). It is proposed that increase of ethylene/propylene ratio in the case of hydrogen dilution is due to the reaction



It looks natural to assume that there is no big difference between dilution by argon and nitrogen. Also we can assume that in our horizontal-flow reactor using of nitrogen as a carrier gas results in some reduction of gas temperature in the hot zone comparing to the case of hydrogen. So, we can assume that in our epitaxial process when nitrogen is used as a carrier gas, propane is decomposed to ethylene and propylene in similar quantities, while in the case of hydrogen the main product is ethylene.

Efficiency of GaN doping with carbon was studied in [8] for a number of hydrocarbons. Our quantitative analysis of the experimental data presented there shows that for acetylene, ethylene, propane, and *i*-butane incorporated carbon concentration is proportional to the precursor flow in 3/2 power. This law is executed even stricter than in our experiments. Then, doping efficiency of acetylene, ethylene, and propane is absolutely the same if plotted against precursor mole flow, not the amount of added carbon atoms as in [8]. For *i*-butane it is also so if its mole flow is multiplying by approximately 1.3.

These speculations well match experimental results described in [8], where hydrogen was used as a carrier gas. If in this case propane is decomposed mostly to ethylene

and concentration of produced ethylene is close to the concentration of propane fed into the reactor, efficiencies of doping from these two precursors should be equal. Exactly the same is reported in [8]. We do not have any data on doping from ethane — a third product of propane decomposition — but it cannot be responsible for any of the observed effects because ethylene is produced in much higher quantities and is proved to have the same doping efficiency and character as propane [8]. Under nitrogen carrier gas, ethylene and propylene should be produced from propane in similar quantities. A possible assumption that propylene is ineffective as a precursor can only explain the observed reduction of carbon concentration in the epilayer, not the change from 3/2 power to linear law.

Moreover, it looks like propylene is as efficient precursor as ethylene.

First of all, described above transformation of propylene to ethylene (1) is a secondary process and may not occur in a MOVPE reactor with short residence time. Then, this assumption is proved by data on GaN doping with carbon from *i*-C₄H₁₀ [8,14]. Pyrolysis of *i*-butane (pure, diluted in argon and hydrogen) was studied in [28,29]. It was shown that the main products of this reaction are methane and propylene, and the products composition does not significantly changed in the presence of hydrogen. Ethylene is produced in much less quantities. Moreover, for propane pyrolysis it is generally accepted that both ethylene and propylene are produced simultaneously being two products of the same chain of reactions [see e.g. 30]. Under significant hydrogen concentration reaction (1) leads only to increase of ethylene and decrease of propylene concentrations. In contrast, in isobutane pyrolysis ethylene is a product of secondary transformations only [28,29]. It is formed from propylene and observed only after significant degree of *i*-C₄H₁₀ pyrolysis. In MOVPE reactor this process does not have enough time for development. So, from one side we can conclude that in the case of

doping from *i*-butane the dominating source of carbon is propylene. On the other side, in accordance to [8,14] doping from *i*-butane is very similar to doping from propane and ethylene.

So, returning to doping from propane, we can conclude that switching of the carrier gas type from hydrogen to nitrogen should result in change of gas-phase reactions of propane pyrolysis but it cannot result in the transition from 3/2 power to linear law.

Speculating about the influence of the carrier gas on the surface reactions resulting in change of the process of GaN doping with carbon from propane it is very attractive to suppose that hydrogen (and also concentrations of ammonia and TMGa) affect nitrogen vacancies formation on GaN surface and thus carbon incorporation into nitrogen sites (C_N). However, in accordance to the compensation model proposed in [7] and some other publications, formation of semiinsulating GaN:C requires comparable concentrations of C_N and C_{Ga} states. During our work we have grown GaN:C epilayers under various conditions resulting in very strong difference in carbon incorporation efficiency, and all of them were semi-insulating. Thus there should be either mechanism of increasing of C_{Ga} incorporation rate increase simultaneously with C_N , or the model described in [7] requires revision (like in [31]), or other explanations of the observed phenomena should be proposed.

Alternative speculations may be performed on the basis of our study of graphene deposition from propane [32]. There we have reported that under the reactor conditions very similar to used in the present work but without ammonia and TMGa supply, graphene is deposited on sapphire substrate as a result of propane pyrolysis. However, it occurs only if hydrogen was used as a carrier gas. Using of nitrogen results in amorphous carbon layers at any temperatures in the investigated range. It was observed also, that even as small ammonia concentration in hydrogen as 0.2% inhibits graphene deposition on sapphire, but on preliminary deposited graphene deposition continues at least up to 2% of ammonia content. Already deposited graphene layers are stable under $NH_3:H_2$ mixtures with up to 10% NH_3 concentration, at 20% very slow etching starts. Surprisingly, under $NH_3:N_2$ mixtures graphene is much less stable, only 0.2% of ammonia is enough to start etching, which rapidly accelerates with ammonia concentration rise. Thus, hydrogen and ammonia influence the surface processes of carbon incorporation in graphene layer. We cannot exclude that something similar happens during doping of GaN with carbon. One more effect was observed during our study of graphene deposition. Presence of gallium either in a form of GaN deposits in the reactor decomposing to gallium by hydrogen or as very low TMGa supply prior to graphene deposition accelerate graphene deposition process. (It was difficult to use this effect in the reproducible manner, it results in some deterioration of graphene properties, the process occur without any traces of gallium also, so the effect remains unpublished.)

In accordance to [21], rise of H_2 and reduction of NH_3 concentration leads to increase of GaN surface coverage with gallium adatoms. This effect also may be responsible for the observed dependencies of carbon incorporation on the type of carrier gas and ammonia concentration.

Possible mechanisms of the influence of carrier gas composition on the surface processes of carbon incorporation are not mutually exclusive.

Concluding this section we understand that strict explanation of our experimental results is impossible without application of complex methods of experimental study of chemical components distribution in the reactor volume and/or detailed numerical simulations of physical and chemical processes in the reactor volume and on the surfaces. Unfortunately, we do not have neither resources nor expertise to follow any of these ways. However, we hope that the above speculations as well as our experimental results may help other groups in development of advanced model of GaN carbon doping from hydrocarbons.

5. Conclusions

A doping of GaN with carbon using methane and propane as carbon precursors was studied in a wide range of reactor conditions using hydrogen and nitrogen as carrier gas. It was revealed that carbon concentration is raised with growth rate both for doping from propane and methane. It was demonstrated that the combination of propane as a precursor and hydrogen as a carrier gas is very specific. In this case carbon concentration in GaN epilayers is roughly proportional to propane concentration to the power 3/2 for low and moderate growth rates, and inversely proportional to the square of ammonia concentration at any growth rate in the studied range. In contrast, for the rest three cases (propane and nitrogen, methane and both types of carrier gas) carbon concentration linearly depends on the precursor concentration and inversely proportional to the ammonia concentration. If methane is used as a precursor, carbon concentration in GaN layers rise with pressure while for propane it is independent of pressure. Finally, it was revealed that temperature dependencies of carbon incorporation from methane and background incorporation are similar while propane is more effective precursor at high temperature. Analysis of the experimental data shows that methane produced by trimethylgallium pyrolysis is an important source for carbon incorporation in GaN (especially at high growth rates) and that the surface chemical processes are responsible for the dependence of doping efficiency from propane on the type of carrier gas.

Conflict of interest

The authors declare that they have no conflict of interest.

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