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An inductively coupled plasma metal organic chemical vapor deposition based on showerhead structure for low temperature growth

Zixuan Zhang, Yi Luo, Jiadong Yu, Xiang Li, Jian Wang, Wangyang Yu, Lai Wang, Zhibiao Hao, Changzheng Sun, Yanjun Han, Bing Xiong and Hongtao Li

Beijing National Research Center for Information Science and Technology (BNRist), Department of Electronic Engineering, Tsinghua University, Beijing 100084, People’s Republic of China

* Authors to whom any correspondence should be addressed.

E-mail: luoy@tsinghua.edu.cn and yjd13@tsinghua.org.cn

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Abstract

An inductively coupled plasma metal organic chemical vapor deposition (ICP-MOCVD) based on showerhead structure is proposed for the low temperature growth of thin solid films including GaN. The flow field of precursors in the chamber of ICP-MOCVD was analyzed and the structure of showerhead was optimized by changing the showerhead diameter to obtain uniform velocity field above the substrate. The thickness non-uniformity of GaN films grown at 600 °C was improved from 5.14% to 1.86% after the optimization of showerhead. On that basis, the influence of triethylgallium (TEG) and trimethylgallium (TMG) on low-temperature GaN growth were investigated and TEG was proved to be the more appropriate Ga source in this case. Finally, GaN film with high c-axis and in-plane orientations was obtained on sputtered AlN/sapphire template and the full width half maximums of (002) and (102) x-ray rocking curves are 0.45° and 0.57° respectively. Our results provide a practicable method for the optimization of low-temperature MOCVD, which has potential to obtain large-scale crystalline films at low temperature.

1. Introduction

Large-scale semiconductor thin films have many applications in solar cells, backlight display, flexible devices and other thin film devices [1–4]. Generally, semiconductor thin films are grown on single-crystalline substrates and have relatively high costs and small sizes [5]. The growth temperature is relatively high to dissociate the precursors and promote the adatom surface migration [6], e.g., the GaN growth temperature in commercial metal organic chemical vapor deposition (MOCVD) is above 1000 °C [7]. However, although the large-scale amorphous substrates have low cost, the softening (or melting) points of which are relatively low. For example, the softening point of the float glass substrates is generally no more than 600 °C. Therefore, the low-temperature growth is a more promising way for fabricating large-scale, low-cost crystalline film compared with the traditional high-temperature growth, which has huge potential to expand the choice of substrates and promote the development of large-area electronics. In addition, high-temperature growth results in many other limitations, such as serious thermal mismatch between the epilayers and the substrates, and difficulty in integration with Si-based devices. Meanwhile, it is challenging to grow high-quality In-rich alloys at high temperature due to the low vapor pressure and decomposition temperature of InN. Obviously, low-temperature growth can circumvent such limitations [6].

In this work, an inductively coupled plasma metal organic chemical vapor deposition (ICP-MOCVD) based on showerhead structure is proposed for the low temperature growth of the large-scale semiconductor thin films. And the feasibility of the ICP-MOCVD is verified by growing crystalline GaN films at 600 °C, which is important for (opto)electronic devices [8, 9]. GaN low-temperature growth techniques usually require the
assistance of plasma for cracking the N source, in which N radical particles serve as the group V precursor in the low-temperature growth of GaN. Variety growth techniques have been employed for low temperature growth up to now, which can be classified into two categories. One is physical vapor deposition, such as plasma-assisted molecular beam epitaxy (PA-MBE), pulsed sputtering deposition (PSD), and pulsed laser deposition (PLD) [10–12]. However, physical vapor deposition generally need ultrahigh vacuum, which are not appropriate for the commercial fabrication. The other is chemical vapor deposition, such as low-temperature MOCVD, which is more suitable for industrialization. The key points of such low-temperature MOCVD are that uniform and high-density N plasma should be obtained and the high-energy ions which are harmful to growth should be filtered out [13]. In addition, a uniform distribution of both group III and V radicals in the growth chamber is also needed. Butcher et al designed a remote plasma chemical vapor deposition (RPCVD) to dissociate precursors from group V with electron cyclotron resonance (ECR) plasma in their MOCVD system, and the large gap (up to ~25 cm) between the plasma source and substrate surface was adopted to reduce the ions bombardments. The precursors of group III were injected into the growth chamber separately via the gas injection ring [14]. Nevertheless, the ECR plasma suffers from the mode hopping and has the limit in the application of large area epitaxy growth. Moreover, the gas injection ring has been gradually abandoned in commercial equipment due to the severe pre-reaction between the group III and V precursors. Yi Lu et al proposed a radical-enhanced metal organic chemical deposition (REMOCVD), in which a metal grid was adopted below the plasma to confine the ions [15]. However, the group III precursor was also uniformized by a gas injection ring, which limits the improvement of GaN crystallinity. Besides, there is no discussion about the compatibility between metal grid and gas fluid. K. Scott et al reported their migration enhanced afterglow chemical vapor deposition system (MEACVD). Although group V precursors could be dissociated by capacitively coupled plasma (CCP), the crystallinity of GaN was not mentioned in their report [16].

Several methods have been employed in various low-temperature MOCVD to release the ions bombardment [14–16]. However, there are few reports about the flow field in the CVD chamber which is a key point to obtain the semiconductor thin films with better crystallinity. As is known to all, the typical glowing discharge pressure of plasma technology is about 1 ~ 10^5 Pa [17], which is much different from the operating pressure (~10^3 Pa) of commercial MOCVD [18, 19]. Therefore, it is necessary to investigate the transport of precursors at low pressure in low-temperature MOCVD.

In our previous studies, it has been demonstrated that the showerhead structure can effectively filter out the ions in plasma [20]. Here, the effects of precursors transport in ICP-MOCVD have been investigated for the first time to optimize the showerhead structure and the growth process has been initially investigated. Firstly, the transport model was established based on the Navier–Stokes equations (N-S equations) and the laminar flow model. The difference of the flow field between low pressure (1 Pa) and conventional MOCVD growth (20000 Pa) was studied. The showerhead structure was then optimized for low pressure growth so as to ensure a uniform flow field, thus allowing GaN films with improved uniformity and crystalline quality. Moreover, preliminary experiments are carried out based on such ICP-MOCVD. The effects of triethylgallium (TEG) and trimethylgallium (TMG) on GaN low-temperature MOCVD growth were investigated and GaN film with high c-axis and in-plane orientations was obtained on sputtered AlN/sapphire template.

2. Transport model and experimental details

Although plasma consists of electrons, ions and neutral radical species, a large number of studies show that neutral radical species mainly dominate the growth of III-nitrides instead of ions [14, 20]. Therefore, dynamics of neutral species at low pressure need to be studied due to the few relevant research on the transport of precursors in the low-temperature MOCVD chamber. According to the mean free path (MFP) theory of the gas molecules, the Knudsen number (Kn) and the Reynolds number (Re) are the key factors for determining the motion characteristic of the gas [21, 22].

The Kn defined as the ratio of the MFP (l) to the diameter of chamber (D), which is a dimensionless number of the flow characteristic [21]:

$$Kn = \frac{l}{D}$$  \hspace{1cm} (1)

The MFP equations [23] can be expressed as:

$$\lambda = \frac{KT}{\sqrt{2} \pi d^2 P}$$  \hspace{1cm} (2)

Where d, P and T are the diameter of gas molecules, pressure, and temperature, respectively.

Nitrogen molecules (N_2) were adopted as the group V gas source and d = 0.304 nm. The range of the ICP glowing discharge pressure (P) is 1 ~ 40 Pa. The temperature of the chamber changes from 600 °C (growth
temperature) to 50 °C (cooling sidewall). D is set to 270 mm. Thus, the Kn is about 0.01 ∼ 0.09, which means the gas flow satisfies the N-S fluid continuity equations [21]. The Re is a measurement between the inertial force and the viscous force of the fluid:

\[
Re = \frac{\rho vD}{\mu}
\]

The gas density ρ and the gas velocity v could be estimated by the ideal gas state equation and the Bernoulli equation [24–26], μ is viscosity coefficient and the viscosity coefficient of N2 at different temperatures can be obtained from the reference [27]. Therefore, the estimated range of Re is about 0.5 ∼ 5, indicating the laminar flow state because Re is less than 2300 [22, 24–26]. In a word, the neutral species could be analyzed by computational fluid dynamics (CFD) in accordance with the N-S equations and laminar flow [28, 29].

Figure 1 shows the schematic diagram of the ICP-MOCVD. N source is injected into a ring-shaped nozzle and then diffuses uniformly, thus a relatively uniform spatial plasma in the discharge chamber can be obtained. The ionized N source plasma is then injected into the growth chamber through the group V gas holes in the showerhead. The MO source is injected into the showerhead and transported onto the graphite susceptor through the group III gas holes, which is thermally dissociated near the susceptor surface. The simulation was performed with the process parameters of the ICP-MOCVD, such as: chamber pressure of 1 Pa, susceptor rotation rate of 200 rpm and growth temperature of 600 °C. N2 was used as the N source because it could produce higher radical species density and TMG was used as the MO source. The flow rates of N2 and TMG were 320 sccm and 5 sccm, respectively.

All the GaN samples were grown in this ICP-MOCVD. Purified N2 was used as the N source. And MO sources were TMG and TEG. The substrates were firstly degassed in H2 atmosphere at 600 °C before GaN growth to remove the impurities adsorbed on the surface. The base pressure of growth chamber, the substrate temperature, the RF power and the N2 flow rate were set to 1 Pa, 600 °C, 950 W, and 320 sccm, respectively. The thicknesses of GaN films were about ~ 300 nm. The growth rate was measured by an in situ laser thickness monitor. The crystalline and the impurity properties of the as-grown GaN films were characterized by using x-ray diffraction (XRD), secondary ion mass spectroscopy (SIMS) and electron backscattered diffraction (EBSD) measurements.

3. Results and discussion

3.1. N2 flow field distribution

The distribution of flow field in the chamber was studied. The showerhead in the chamber has evenly distributed gas holes. Figure 2 shows the cross-sectional distribution of the flow field on account of the rotational symmetry of the cylindrical reactor chamber. The streamlines in the reactor chamber at 1 Pa and 20000 Pa are shown in figures 2(a) and 2(b), respectively, which indicate the distribution of the gas flow in the chamber. It is obviously
that there is a significant difference of the streamlines between the low pressure (1 Pa) and conventional MOCVD operation pressure (20000 Pa). From figure 2(a), the N₂ diffuses downward immediately after injecting into the discharge chamber, then flows through the gas holes of showerhead and finally flows out of the chamber into the exhaust pipeline. The streamlines are denser near the sidewall of chamber and become sparser toward the susceptor surface. There is almost no streamline through the inner gas holes of showerhead. Therefore, precursors may have a serious loss at low pressure. On the contrary, the eddy flow is generated in the discharge chamber under 20000 Pa, as shown in figure 2(b). Hence, the distribution of streamlines is more uniform in this case. Therefore, the streamlines extend toward inner gas holes of the showerhead, which increases the precursors arriving at the susceptor surface. Furthermore, the distribution of the velocity magnitude at 1 Pa and 20000 Pa are shown in figures 2(c) and 2(d), respectively. From figure 2(c), the distribution of velocity magnitude is rather non-uniform among the gas holes. The velocity increases gradually from the inner holes to the outer holes and the velocity magnitude at the edge of showerhead is about 11 times of that at the center. This characteristic makes the velocity of outer region is obviously greater than that of central region on the susceptor. However, the velocity distribution of gas holes under 20000 Pa shows a faster velocity in the inner region. As shown in figure 2(d), the velocity distribution on the susceptor is more uniform.

Figure 3 shows the distribution of the N₂ concentration fraction on the susceptor. It could be seen that the concentration of N₂ gradually increases from the center to the edge under 1 Pa, which is caused by the distribution of velocity magnitude on this condition. The inset is the typical cloud picture of N₂ distribution under 1 Pa and the difference of color on the surface of susceptor means that the N₂ distribution is uneven. However, the N₂ concentration is extremely uniform along the radial direction under 20000 Pa result from the uniform distribution of velocity. The different phenomenon of the N₂ concentration distribution between 1 Pa and 20000 Pa can be attributed to the flow velocity, which is rather faster under low pressure. The velocity under 1 Pa is about one order of magnitude higher than that under 20000 Pa. Therefore, the precursors diffuse immediately toward the direction of the exhaust pipeline at 1 Pa, leading to the serious loss of precursors and the non-uniformity concentration on the susceptor surface. Nevertheless, the diffusion velocity is relatively slow at 20000 Pa. So the showerhead has a blocking effect for the group V gas, resulting in a uniform velocity through the gas holes and a uniform concentration distribution on the susceptor surface.
3.2. The optimization of the showerhead

A majority of studies on MOCVD have revealed that the uniformity of precursors has a significant influence on the film uniformity and crystalline quality [24, 25]. It could be seen that the showerhead is a key issue to the distribution of precursors above the susceptor. Thus, the showerhead structure was optimized by reducing the showerhead diameter in order to improve the flow field. As shown in figure 4(a), the flow pattern of streamlines has a significant optimization. The precursors diffuse toward the inner of the discharge chamber, so the distribution region of the gas flow gathers toward the central region of the showerhead and the susceptor. In figure 4(b), the velocity through each gas hole is almost the same except for the central gas hole. According to the simulation results, the uniformity of velocity above the susceptor becomes extremely uniform from the center to the edge.

Figure 5 shows the N$_2$ concentration fraction distribution of the optimized showerhead structure. The concentration is much more uniform along the radial direction above the susceptor. What’s more, the numerical value of the N$_2$ concentration in the central region is also increased compared with the un-optimized results in figure 3. The inset shows the relatively uniform color, indicating a more uniform concentration above the susceptor.

3.3. The influence of the flow field on GaN ICP-MOCVD growth

GaN films were grown on 2-inch sapphire substrates using 5 sccm TMG to investigate the effects of the flow field on the crystalline quality and uniformity. The outermost gas holes were gradually removed to reduce the showerhead diameter and optimize the flow field. Figure 6 shows the full width half maximums (FWHMs) of...
(002) x-ray rocking curves (XRCs) and the growth rates of GaN films grown at different showerhead diameter. The un-optimized showerhead diameter is 0.2375 m. The FWHM of (002) XRC is reduced from 1.44° to 1.07° as the showerhead diameter reduced to 0.1325 m, illustrating the improved crystalline quality. In addition, the growth rate of GaN films is increased from 310 nm h$^{-1}$ to 475 nm h$^{-1}$ because more reaction sources reached onto the substrate surface. However, as the showerhead diameter is further reduced to 0.0975 m, the FWHM of (002) XRC increases and the growth rate decreases. The reason is that the surface recombination and collision loss of the active particles in the plasma will be greatly increased when the number of gas holes is insufficient, resulting in the decrease of the growth rate and the crystalline quality of GaN films. Thus, the optimized flow field is obtained when the showerhead diameter is 0.1325 m according to the experimental results.

Figure 7 shows the thickness profiles of GaN films along the radial direction on 2-inch sapphire substrates measured by an interferometric thickness gauge, the right side of the wafer in the inset is closer to the susceptor center during the growth. Here we define the film thickness non-uniformity as the ratio of the standard deviation of the film thickness at different position to the average film thickness. The thickness of GaN grown in the un-optimized flow field reduces gradually from the left region to the right region, corresponding to the changing of N$_2$ concentration in figure 3, and the thickness non-uniformity is about 5.14%. The thickness profile of GaN grown in the optimized flow field is more uniform and the thickness ranges from 290 ∼ 300 nm. The corresponding non-uniformity is improved to 1.86%, which is very close to the commercial MOCVD [30].
3.4. The influence of Ga sources and sputtered AlN buffer on GaN ICP-MOCVD growth

Based on the optimized showerhead structure, the influences of Ga sources on the low-temperature ICP-MOCVD growth of GaN films were studied. GaN samples were grown on 2-inch sapphire substrates using 62 sccm TEG and 5 sccm TMG, respectively. Both of the mole flow rates of TMG and TEG were 30 μmol min⁻¹.

Figure 8(a) shows the (002) XRCs of the two GaN samples. The (002) XRCs FWHMs of GaN samples grown using TMG and TEG are 1.07° and 0.81°, respectively. The GaN sample grown using TEG has lower (002) XRC FWHM and stronger intensity than that grown using TMG, indicating that TEG is more beneficial to obtain the GaN film with better crystalline quality at low growth temperature. The difference between different Ga sources in ICP-MOCVD may be ascribed to the carbon impurity concentration. TMG cannot be completely decomposed at 600°C due to the high thermal decomposition temperature. The decomposition products of TMG are alkyl radicals which are easy to incorporate into the crystal lattice. As alkyl radicals introduce a large amount of carbon impurity into the GaN films, it results in poor crystallinity [31]. Oppositely, TEG is easier to be decomposed at 600°C. The main crack way of TEG at low temperature is β-H elimination, and the main product is C₂H₄ which is stable and unlikely to be re-incorporated into the crystal lattice [32]. Figure 8(b) is the SIMS depth profiles of the carbon, hydrogen and oxygen normalized to the Ga signal in GaN samples grown using TMG and TEG.

Moreover, the sputtered AlN buffer layer was introduced in the GaN low-temperature MOCVD growth to further improve the crystalline quality, which is commonly used in GaN commercial production [33].
impact of the AlN buffer is shown in figure 9. As the large lattice mismatch between sapphire and GaN would result in great stress, the in-plane orientations of the GaN crystal nuclei are inconsistent when directly grown on the sapphire substrate at low temperature. As a result, the crystal islands would not completely merge during the subsequent growth and many amorphous regions with rough surfaces would form at the edges of the crystal islands. In contrast, the lattice mismatch between AlN and GaN is relatively small, so the GaN crystal nuclei have consistent in-plane orientations when grown on the AlN/sapphire template, and the crystal islands would merge during the subsequent growth.

10-nm-thick AlN buffer was sputtered on the sapphire substrate at 600 °C. GaN film was grown on such AlN/sapphire template using 62 sccm TEG. Figures 10(a) and 10(b) show the EBSD maps and $\{10\overline{2}\}$ pole figures of the GaN samples grown on the AlN/sapphire template and the sapphire substrate, respectively. The EBSD maps use different colors to show the c-orientations and red domains represent (002) c-oriented GaN. It can be seen that there are many black domains and some scattered green and blue dots in figure 10(a), which means there are many amorphous or rough GaN, (010) and (120) c-oriented GaN in the sample grown on sapphire substrate [34]. The corresponding $\{10\overline{2}\}$ pole figure has the pattern of ring, which indicates poor in-plane orientations. On the contrary, the EBSD map in figure 10(b) is all red color and the corresponding $\{10\overline{2}\}$ pole figure has a pattern of six discrete dots with rotational symmetry, indicating that the GaN film grown on AlN/sapphire template is hexagonal single-crystalline GaN with high c-axis and in-plane orientations. Those results are consistent with the results of (002) and (102) XRCs of the two samples (as shown in figure 10(c)). The (002) XRC FWHM of GaN film grown on sapphire substrate is 0.81° and there is almost no (102) XRC signals.
However, the (002) XRC FWHM of GaN film grown on AlN/sapphire template is 0.45° and there is an obvious (102) XRC with a FWHM of 0.57°. These results indicate that the sputtered AlN buffer can effectively improve the crystalline quality of GaN films grown at low temperature.

4. Conclusions

The transport of precursors was analyzed in our homemade ICP-MOCVD, and the CFD model was built based on the N-S equations and the hypothesis of laminar flow. Our simulation results reveal that a more uniform distribution of precursors can be obtained by optimizing the showerhead diameter. The enhancements of uniformity and crystalline quality were demonstrated by growing GaN films in the ICP-MOCVD chamber, which exhibited the significant improvement in the crystallinity compared with films grown at higher temperatures. The results indicate that the MOCVD technology can be optimized for the fabrication of GaN films, which can be applied in various fields, such as in-plane orientated GaN samples with good crystallinity were achieved on sputtered AlN/sapphire templates at 600 °C. Our results provide a practicable method for the optimization of low-temperature MOCVD. Such ICP-MOCVD technology has the potential to obtain large-scale crystalline film at low temperature, such as nitride, silicon, and oxide films, etc.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

ORCID iDs

Zixuan Zhang https://orcid.org/0000-0001-9023-3702

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