Hydride Vapor-Phase Epitaxy Reactor for Bulk GaN Growth

Vladislav Voronenkov,* Natalia Bochkareva, Andrey Zubrilov, Yuri Lelikov, Ruslan Gorbunov, Philipp Latyshev, and Yuri Shreter

An hydride vapor-phase epitaxy reactor for the growth of bulk GaN crystals with a diameter of 50 mm is developed. Growth rate nonuniformity of 1% is achieved using an axisymmetric vertical gas injector with stagnation point flow. Chemically resistant refractory materials are used instead of quartz in the reactor hot zone. High-capacity external gallium precursor sources are developed for the nonstop growth of the bulk GaN layers. A load-lock vacuum chamber and a dry in situ growth chamber cleaning are implemented to improve the growth process reproducibility. Freestanding GaN crystals with a diameter of 50 mm are grown with the reactor.

1. Introduction

Chloride–hydride epitaxy is the primary method of bulk GaN substrate production. The hydride vapor-phase epitaxy (HVPE) method allows us to grow epitaxial layers of high purity and to produce uncompensated semi-insulating material.[11] The structural perfection of HVPE-grown layers is determined by the seed substrate quality: the dislocation density in the epitaxial layers grown on the bulk GaN substrate does not exceed the dislocation density in the substrate.[2–4] The HVPE method is also promising for the epitaxy of high-purity device structures.[1]

The high process temperature together with the chemically aggressive substances involved in the HVPE process makes the design of the reactor a challenging task. Gallium, ammonia, and hydrogen chloride react to some extent with most heat-resistant materials, including quartz, often used in HVPE reactor design,[5–15] which leads to the degradation of reactor parts and unintended doping of the grown crystal. The exclusion of quartz from the hot zone of the reactor is known to reduce the level of unintentional doping significantly.[1,16] The typical HVPE reactor layout with a gallium source located inside the hot zone of the reactor, near the growth chamber, imposes considerable restrictions on the growth chamber design, limits the source capacity, and complicates the reactor maintenance.

The process is usually performed at near-atmospheric pressures, which makes the gas flow prone to free convection and recirculation[10,12,17] and makes it difficult to obtain a uniform thickness distribution and suppress the parasitic growth on the injector nozzle at the same time.

In this article, a reactor designed for growing bulk GaN layers with a diameter of 2 in. is described. To ensure long-term nonstop growth processes, a high-capacity external precursor source was developed, and the gas flow pattern in the growth chamber was optimized to suppress the parasitic growth at the injector nozzle completely. Heated exhaust lines and a large-capacity powder trap were used to prevent clogging. To reduce the time between growth processes, a vacuum load-lock chamber was used. Chemically resistant materials were used in the hot zone of the reactor to reduce possible crystal contamination and to increase the lifetime of the reactor parts. Uniformity of the deposition rate and a V/III ratio of less than 1% were obtained.

2. Growth System Design

2.1. Modeling Procedure Details

2.1.1. Chemical Equilibrium Analysis

To calculate the chemical equilibrium, a hierarchical method was used.[18–31] Thermochemical properties of substances were sourced from previous studies.[22–28] No preliminary assumptions were made about the composition of the system; all species for which thermochemical data were available in the sources[22–28] were accounted for in the calculation. Ideal gas behavior was assumed.

2.1.2. Computational Fluid Dynamics Calculation

The self-consistent calculation of gas flow, heat transfer, particle diffusion, and surface reactions was performed in a 2D radially symmetric approximation by the finite volume method.[29] Surface reactions were taken into account under the assumption of a kinetically limited thermodynamic equilibrium: surface reactions were assumed to be infinitely fast except for the decomposition of ammonia into hydrogen and nitrogen, which was assumed to be kinetically inhibited. This approximation is applicable at typical GaN growth temperatures, which are in the range of 900–1100 °C. The GaN deposition process at these temperatures is limited by diffusion through the boundary layer or thermodynamically limited,[30–34] and the process of ammonia decomposition on the surfaces is kinetically limited.[35,36]

Dr. V. Voronenkov, Dr. N. Bochkareva, Dr. A. Zubrilov, Y. Lelikov, Dr. R. Gorbunov, Dr. P. Latyshev, Prof. Y. Shreter
Division of Solid State Electronics
Ioffe Institute
Politehnicheskaya 26, St. Petersburg 194021, Russia
E-mail: voronenkov@mail.ioffe.ru

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2.2. External Boat

The aim of implementing the external boat was to simplify the reactor design and service. The external boat volume is not constrained by the dimensions of the reactor hot zone, and precursor refilling can be conducted without disassembling the reactor. The main technical limitation when designing an external source is the need to transport gallium precursors at temperatures compatible with metal pipelines and conventional vacuum elastomeric or metal seals, i.e., at $T < 200\, ^\circ C$. This requirement cannot be fulfilled using a conventional chlorination process performed at temperatures above 600 °C, where the main product of the chlorination reaction (Figure 1a) is gallium monochloride, formed by the reaction

$$\text{Ga}_{\text{liquid}} + \text{HCl} \rightarrow \text{GaCl} + \frac{1}{2}\text{H}_2 \quad (1)$$

that decomposes with the condensation of metallic gallium, at temperatures below the chlorination temperature (Figure 1b).

One known way to overcome this problem is to use an organometallic[37–39] or organometallic chloride[40–42] precursor that is converted to gallium monochloride in the hot zone of the reactor by reaction with hydrogen chloride or by decomposition, respectively. The drawback of this approach is the possible carbon contamination and the formation of condensed carbon coproducts.[43]

Another possible way is to use gallium trichloride,[41,44–46] which can be evaporated from a bubbler[41,44,45] or produced in a high-temperature boat by chlorination in excess of chlorine[46] or by chlorination at temperatures below 300 °C, where the main products of the chlorination reaction are GaCl$_3$ and its dimer Ga$_2$Cl$_6$ (Figure 1a) formed in the reaction

$$\text{Ga}_{\text{liquid}} + 3\text{HCl} \rightarrow \text{GaCl}_3 + \frac{3}{2}\text{H}_2 \quad (2)$$

Gallium trichloride condenses only at temperatures below 200 °C (Figure 1c), which makes it possible to transport it through pipes heated to moderate temperatures in the range of 100–200 °C, depending on the degree of dilution by the carrier gas and the process pressure.

Gallium trichloride can be used directly for the synthesis of GaN by the reaction

$$\text{GaCl}_3 + \text{NH}_3 = \text{GaN} + 3\text{HCl} \quad (3)$$

However, high supersaturation of this reaction can lead to a parasitic reaction in the gas phase;[47] also, the growth of the Ga-polar GaN surface is unstable due to the steric effect.[48] To avoid this, GaCl$_3$ can be reduced to GaCl by reaction with hydrogen in the hot zone[49]

$$\text{GaCl}_3 + \text{H}_2 \rightarrow \text{GaCl} + 2\text{HCl} \quad (4)$$

In this case, GaN deposition proceeds by the same reaction as in the reactor in the internal boat

$$\text{GaCl} + \text{NH}_3 \rightarrow \text{GaN} + \text{HCl} + \text{H}_2 \quad (5)$$

Two types of external sources of gallium trichloride were successfully tested: an external boat with gallium chlorination at a temperature of about 200 °C and a bubbler evaporator filled with dry GaCl$_3$. In both cases, GaCl$_3$ was delivered to the growth chamber through pipes heated to a temperature below 200 °C.

The external boat was a vacuum-tight ceramic vessel with metal flanges with an elastomeric seal. Inside was a container filled with gallium; the surface area of the liquid gallium was about 100 cm$^2$. The gallium container was heated to a temperature of about 200 °C by an external resistive heater. During growth, 100–300 sccm of HCl diluted in nitrogen was fed into the boat, which corresponded to a 33–100 sccm GaCl$_3$ flow at the outlet of the external boat. The chlorination efficiency at a given temperature and flow was close to 100%. The total flow at the exit of the boat was about 500 sccm. The entire system was thermally insulated to maintain the temperature of the walls and piping above the condensation point.

An alternative GaCl$_3$ delivery method using a bubbler evaporator was also tested. A bubbler with a capacity of 1 L was filled with gallium trichloride with a purity of 99.9999%. Before the growth process, the bubbler was heated to a temperature above the melting point of GaCl$_3$, usually 90–110 °C, which was maintained by a thermostat. The carrier gas was hydrogen or a mixture of nitrogen and hydrogen. The total gas flow at the bubbler outlet was also maintained at around 500 sccm. Like the external boat,
the bubbler-based system was placed in a heated enclosure to prevent condensation. Although the bubbler system allowed us to achieve results similar to that obtained using the external boat, it had much more stringent requirements for temperature stabilization accuracy and was more difficult to service.

2.3. Growth Chamber Design

The reaction chamber (Figure 2a) is vertical with hot walls and external resistive heating that provides a growth temperature of up to 1500 °C. All components of the growth chamber are made of nonoxide ceramics and refractory metals. The substrate with a diameter of 50 mm is placed horizontally, growth surface up. The gas injector consists of a set of coaxial tubes, implementing the stagnation point flow pattern (Figure 2b). Nitrogen is used as a carrier gas. Gallium chloride is fed through the inner tube.

To completely prevent parasitic growth on the nozzle of the injector, a relative saturation \( S \) of less than unity is maintained on the surface

\[
S = \frac{P_{\text{GaCl}}}{P_{\text{NH}_3}} \frac{P_{\text{HCl}}}{P_{\text{H}_2}} K(T) < 1
\]

where \( P_x \) are the partial pressures of the corresponding substances and \( K(T) \) is the equilibrium constant of the GaN deposition reaction (5). The inert gas shielding between the ammonia supply line and the gallium supply separates GaCl₃ and NH₃ and reduces \( P_{\text{GaCl}} P_{\text{NH}_3} \) in the vicinity of the injector. To fulfill condition (6), the presence of hydrogen chloride and hydrogen in the injected gases is also necessary. Since excess hydrogen is added to the carrier gas transporting gallium trichloride from the external boat, hydrogen chloride formed in reaction (4) and the remainder of the excess hydrogen are always present in the internal tube of the injector during growth. The calculation of the relative saturation field near the injector nozzle is shown in Figure 2e. The relative saturation on the surface of the injector \( S < 1 \), which corresponds to the observed suppressed parasitic growth. Growth campaigns with total deposited GaN layer

Figure 2. a) Schematic drawing of the reactor growth chamber. b) Calculated velocity field. The color represents the magnitude of the absolute gas velocity; streamlines show the laminar flow pattern without recirculation. c) Concentration field of GaCl. Lines of equal GaCl concentration near the substrate are near parallel to the surface, demonstrating a uniform boundary layer. d) Concentration field of NH₃. e) Relative saturation field near the injector nozzle. The black contour line represents the level of relative saturation \( S = 1 \). On the surface of the injector nozzle, \( S < 1 \), and parasitic GaN growth is thermodynamically unfavorable. f) Distribution of the growth rate and ammonia concentration across the wafer surface, and a cross-section thickness profile of a 41 µm thick GaN film, measured by an optical microscope.
thickness of 5.8 mm were conducted, showing no signs of parasitic deposition on the injector.

During the growth process, the substrate can be rotated at a rate of 10–100 rpm; the typical rotation rate used during the bulk crystal growth process was 20 rpm. The purpose of the substrate rotation is to average minor inhomogeneities caused by a possible slight displacement of the substrate or substrate holder relative to the axis of symmetry of the reactor or uneven wear of the heating elements. The gas flow pattern remains unaffected by the substrate rotation since, at a rotation rate of 20 rpm, the azimuthal velocity at the edge of the substrate holder is approximately 7 cm s$^{-1}$, which is an order of magnitude lower than the forced gas flow velocity for the typical growth parameters used (Figure 3b).

2.4. Growth Rate and Ammonia Partial Pressure Uniformity

The advantage of the used injector design is a high achievable homogeneity of the deposition rate and the V/III ratio. The critical factor in achieving high deposition homogeneity in this geometry is the suppression of free convection. The driving force of free convection is the difference in gas density associated with the temperature gradient and the significant difference in the molecular weight of the carrier gas and the precursors.

The predominance of free convection over forced convection is expected at high values of the Rayleigh number$^{[50]}$

$$Ra = \frac{gL^3 \delta \rho}{\nu D \rho} > 1708$$

and with a high ratio of the Grashof number to the Reynolds number$^{[50]}$

$$\frac{Gr}{Re^2} = \left(\frac{gL^3 \delta \rho}{\nu^2 \rho}\right) \left(\frac{\mu L}{\nu}\right)^2 \gg 1$$

where $L$ is the reactor height, $u$ is the gas flow velocity, $p$ is the pressure, $\nu$ is the kinematic viscosity, $g$ is the gravitational acceleration, and $\delta \rho / \rho$ is the relative inhomogeneity of the gas density associated with inhomogeneity of the temperature $\delta T / T$ and gas mixture composition. The nature of the diffusion coefficient $D$ depends on the origin of the density inhomogeneity. If the density inhomogeneity is determined mainly by the temperature inhomogeneity, then $D$ is the thermal diffusivity, which for nitrogen gas at a temperature of 1000 °C is $\approx 2.5$ cm$^2$ s$^{-1}$. If the density inhomogeneity is determined mainly by the concentration of a nonuniformly distributed substance with a molecular mass substantially different from the mass of the carrier gas, then $D$ is the diffusion coefficient of this substance. In the described reactor, the temperature gradients in the growth chamber are relatively small and the density inhomogeneity is determined by the concentration of GaCl; the diffusion coefficient of GaCl in nitrogen at $T = 1000$ °C is $\approx 1.3$ cm$^2$ s$^{-1}$.

The Rayleigh number depends quadratically on pressure

$$Ra \sim p^2$$

(9)

and $Gr / Re^2$ scales quadratically with pressure and the mass flow rate $Q$

$$\frac{Gr}{Re^2} \sim \frac{p^2}{Q^2}$$

(10)

Therefore, reducing the process pressure and increasing the total gas flow rate are an effective method of suppressing free convection. At the given growth chamber dimensions and the gas flow rate, no free convection is observed at pressures below 200 mbar.

The gas flow in the growth chamber of the reactor was calculated, taking into account heat transfer, chemical species diffusion, and surface reactions. The total process pressure was 150 mbar; the substrate temperature was 1000 °C. The

Figure 3. Equilibrium partial pressure of gaseous decomposition products of various materials in a mixture of GaCl:H$_2$HCl in a ratio of 1:1:1 at atmospheric pressure: a) quartz, b) alumina, c) zirconia, d) boron nitride, e) molybdenum, f) tungsten, and g) platinum; h) summary diagram: total partial pressure of gaseous decomposition products over each of the materials.
Reynolds number was $\text{Re} \approx 30$, the Rayleigh number was $\text{Ra} \approx 500$, and $\text{Gr}/\text{Re}^2 \approx 0.5$, which corresponds to laminar flow without recirculation and any evidence of free convection (Figure 2b). The calculated concentration fields of GaCl and ammonia are shown in Figure 2c,d. Ammonia is distributed uniformly over the surface of the substrate, where the lines of equal concentration of GaCl are almost parallel to the surface of the substrate. The estimated inhomogeneity of the deposition rate and ammonia concentration along the substrate diameter does not exceed 1%, as shown in Figure 2f.

2.5. Materials

Special attention was paid to the choice of reactor materials. Quartz, in addition to being a potential source of contamination, is fragile and limits the ability to produce parts of complex shape and high accuracy. The chemical resistance of commonly used refractory materials was studied using chemical equilibrium analysis. For example, the chemical resistance of quartz, aluminum oxide, zirconium oxide, boron nitride, molybdenum, tungsten, and platinum in the gallium chloride injector nozzle was estimated by calculating the equilibrium reaction products in a model mixture containing gallium monochloride, hydrogen, and hydrogen chloride in a ratio of 1:1:1 (Figure 3).

As can be seen, quartz and other oxide materials have equilibrium pressures of decomposition products of more than $10^{-4}$ atm at a temperature of 1000 °C (Figure 3a–c). The main decomposition products are water vapor and metal chloride. Replacing quartz with boron nitride can significantly reduce possible oxygen contamination; however, the equilibrium pressure of BCl$_3$ at 1000 °C is still higher than $10^{-4}$ atm (Figure 3d).

Molybdenum, tungsten, and platinum exhibit a significantly lower pressure of the decomposition products in the presence of hydrogen (Figure 3e–g). It should be noted that in the absence of hydrogen, molybdenum and tungsten are not resistant to HCl.$^{[29]}$

Replacing quartz with refractory metals and BN allows excluding one of the potential sources of silicon and oxygen contamination, extends the service life of the injector nozzle, and adds flexibility in the design of the growth chamber.

A detailed description of the thermodynamic study of the chemical resistance of materials, as well as a secondary ion mass spectrometry study of the concentration of impurities in the metals and ceramics used, precursors, and the resulting GaN layers, will be published separately.

2.6. Exhaust Line Heating to Prevent Clogging with Ammonium Chloride

The gases at the exit of the growth chamber contain hydrogen chloride and ammonia, which react to form solid ammonium chloride with decreasing temperature

$$\text{HCl} + \text{NH}_3 = \text{NH}_4\text{Cl} \quad (11)$$

The condensation of ammonium chloride in the exhaust leads to reactor clogging and must be prevented to ensure long, continuous growth processes. Heating the exhaust lines down to the powder trap prevents premature condensation.

The condensation temperature can be defined as

$$T_{\text{cond}} = \frac{-R \ln \left( \frac{P_{\text{NH}_3}}{P_{\text{NH}_4}} \right)}{\left( \mu_{\text{NH}_3} - \mu_{\text{NH}_4} - \mu_{\text{HCl}} \right)} \quad (12)$$

where $P_{\text{NH}_3}$ and $P_{\text{NH}_4}$ are the partial pressure of GaCl and ammonia, respectively, and $\mu_{\text{NH}_3}$, $\mu_{\text{HCl}}$, and $\mu_{\text{NH}_4}$ are the standard chemical potentials of the species. The dependence of the temperature required to prevent NH$_4$Cl condensation on the partial pressures of hydrogen chloride and ammonia is shown in Figure 4.

If gallium chloride does not completely react in the growth chamber, the formation of the GaCl$_3$:NH$_3$ complex is possible in the exhaust. This substance has a boiling point of 438 °C$^{[31]}$ and requires significantly higher heating of the exhaust compared to ammonium chloride to prevent condensation.

3. Load-Lock and Growth Chamber In Situ Cleaning

The vacuum load-lock chamber and the procedure of the growth chamber dry etching do not directly affect the crystal growth process, but improve the reproducibility of the process and prolong the service life of the growth chamber and the heater, and significantly accelerate and facilitate the workflow. The load-lock chamber is located in the lower part of the reactor, where the substrate holder is moved before and after the growth process for loading and unloading. Load-lock allows the reactor to be reloaded without exposing the growth chamber to the atmosphere and without thermal cycling. The use of the load-lock also allowed us to implement in situ dry etching of the growth chamber before the growth campaign and between the growth processes. The etching is performed at a temperature of 1000–1100 °C in a mixture of hydrogen chloride with hydrogen.
and allows us to remove the GaN deposits and the contaminants that have volatile chlorides.

4. Bulk GaN Layers

A bulk GaN crystal with a thickness of 2.8 mm, grown on a GaN-on-sapphire template using the two-stage growth process\[52\] is shown in Figure 5a. The growth rate was 200 μm h\(^{-1}\); the GaCl:HCl:H\(_2\) ratio was 1:2:2. The crystal separated from the substrate during cooling after growth by spontaneous stress-induced self-separation.\[13,53\] The as-grown surface was smooth, and the density of V-shaped pits was less than 3 cm\(^{-2}\). Microscopic examination showed that the V pits were associated with macrodefects of the seed template and that no new pits were formed during the bulk layer growth. The (0002) X-ray rocking curve full width at half maximum (FWHM) was 51 arcsec. The threading dislocation density estimated using cathodoluminescent microscopy by measuring dark spot density was \(2 \times 10^6\) cm\(^{-2}\) (Figure 5b). The optical quality of the crystal was estimated by photoluminescence (PL). The low-temperature (10 K) PL spectrum of the Ga face of the crystal is shown in Figure 5c. The dominant emission line at 3.473 eV that is usually attributed to the exciton bound to a neutral donor\[54\] has an FWHM of 2.5 meV.

5. Conclusions

The developed reactor has proven itself in long-term operation, with more than a thousand growth processes conducted. Vertical geometry with stagnation point flow provided high uniformity for both the growth rate and the III/V ratio. Vacuum load-lock and in situ growth chamber etching proved to be useful in improving process repeatability. High-temperature chemical-resistant materials of the growth chamber used instead of quartz made it possible to increase the service life and provided an opportunity to reduce the unintentional doping level. High-capacity external precursor sources, a gas injector with suppressed parasitic growth, and the heated exhaust line allowed us to grow bulk layers with thickness up to 3 mm and higher in a single growth process.

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Figure 5. a) Photograph of a freestanding GaN crystal with a thickness of 2800 μm and a diameter of 50 mm. b) Cathodoluminescence micrograph of the Ga-face surface. The dark spot density is below \(2 \times 10^6\) cm\(^{-2}\). c) Low-temperature (10 K) PL spectrum of the Ga face of the crystal.
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Conflict of Interest
The authors declare no conflict of interest.

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