

# **New Process Approaches to Metalorganic Vapor Phase Epitaxy of III-nitrides for High Power HEMTs**

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# Chapter 1

## Introduction

### 1.1 Motivation

In the last 60 years semiconductor technology developed from *one germanium based, matchbox sized single transistor* at Bell Labs to *millions of silicon based transistors on an area the size of a thumbnail* in every day use. Thanks to Moore's Law the cycle of increasing performance and shrinking size keeps going. The leading role in the semiconductor business is still played by silicon, because it has many beneficial properties for device applications. It is abundant in nature, non toxic, easy to process and is one of the best characterized material systems. However, silicon also has significant weaknesses when it comes to optoelectronics or high frequency applications. Silicon is an indirect semiconductor, which greatly reduces its efficiency in light generation like in light emitting diodes (LEDs). Also, many III-V compounds have far smaller effective electron masses than the group IV materials like Si and Ge. Simplified, electronic transport depends on the maximum obtainable charge carrier velocity, which is inversely proportional to the effective mass. Thus, the ultimate speed of integrated circuits depends on the carrier velocity, so the low effective mass is one of the fundamental reasons that GaAs and other III-V compounds are used instead of Si in high-bandwidth and low noise applications like satellite communication and radar. If additionally high power applications are envisaged, *wide bandgap semiconductors* are employed such as the III-nitrides.

Recent progress in epitaxial and bulk growth of GaN and related compounds make these materials viable contenders for a variety of different applications from light emitters in the visible range (where III-nitride based technology is already commercialized) to high frequency and high power devices. The properties of the group III nitrides have often been cited: They cover a wide bandgap range of 0.7eV (InN) to 6.2 eV (AlN). In comparison to other materials they have higher binding energies, making them mechanically very stable. The 3.4 eV bandgap of GaN predicts that it withstands 7.5 times the electric field that

GaAs can before avalanche breakdown occurs. The good electron transport properties of GaN, with an electron mobility value *possibly* in excess of  $2000\text{cm}^2/\text{Vs}$  and an average electron saturation velocity above  $2 \times 10^7\text{cm/s}$  make the material attractive for high frequency application. All in all the nitrides are favorable when it comes to applications in hazardous environments, in high-temperature -frequency and -power regimes. Yet, GaN based RF components are not commercialized and will have to compete with the prevailing silicon based LDMOS devices or even SiGe, with less favorable properties in this application field but low production costs.

Despite these prospects, single crystalline GaN films still need to be grown heteroepitaxially on substrates like sapphire or SiC, which match more or less closely to its lattice constants and thermal expansion coefficient, since -up to now- there is no commercially available substrate for homoepitaxy. In the beginning of GaN epitaxy the heteroepitaxy significantly affected the quality of the material. High defect and dislocation densities ( $\sim 10^{10}\text{cm}^{-2}$ ) resulted from the lattice mismatch. High impurity levels had the effect of a very high n-type background carrier concentration ( $\sim 10^{19} - 10^{20}\text{cm}^{-3}$ ), which made the conductivity control, necessary for device applications, impossible. The problems related to the heteroepitaxial growth of GaN were first overcome by Amano and Akasaki in 1986 [Ama86]. They introduced a thin AlN layer, grown on sapphire at lower temperature (LT) than the subsequent GaN layer, in a so-called two-step growth technique. Later, Nakamura used GaN as low temperature layer [Nak91]. In fact, this LT-layer, the so called nucleation layer, acts like a template between the substrate and the epitaxial layer, resulting in a significant improvement of the material quality of the subsequent GaN layers with dislocation densities  $\sim 10^8\text{cm}^{-2}$  and a dramatic reduction of the background doping concentration ( $\leq 10^{17}\text{cm}^{-3}$ ). These developments also set the stage for the p-type GaN doped with Mg in 1989, which was not possible until then. With these findings the feasibility of the nitrides for device application came into focus and gave a great boost to nitride research.

Nowadays, the quality improvement of GaN-based materials is at a point where high performance electronic devices can be produced. Of great technological interest for applications such as sensors, radar, satellite, wireless base stations and power applications are high electron mobility transistors (HEMTs), which are by far the best investigated group-III nitrides electronic devices. The electron mobility is quite high in  $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$  heterostructures where two-dimensional electron gases (2DEG) form. The sheet conductivity in GaN based HEMTs, as the product of carrier concentration and mobility is further enhanced by the strong piezoelectric and polarization effects present, which account for the high carrier concentration in the 2DEG without doping. The nitrides therefore intrinsically fulfill another demand on HEMT structures: For high power and high speed performance it is of primal importance to maximize the channel sheet carrier concentration without reducing electron mobility and saturation velocity. These facts in combination with the aforementioned material properties of the wide band gap nitrides allow a proportionately higher operating voltage and current for the same load impedance

in comparison to GaAs based heterostructures. When voltage and current are taken together, a power delivery greater than 50 times that of GaAs devices is possible using GaN-based HEMTs. It is this potential increase in power performance that acts as a great motivator for pursuing GaN-based devices and circuits.

In order to achieve this goal, special care must be taken in the choice and preparation of a template. An ideal template for  $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$  heteroepitaxy with respect to the HEMT application must possess:

1. good structural quality with low dislocation densities for high amplification by high channel conductivity at high frequencies;
2. a smooth surface with a low defect density, important for the growth of sharp interfaces and flat surfaces as well as for device processing;
3. semi-insulating properties to avoid any parallel conductivity to the channel layer and to ensure high HEMT performance at high frequency;
4. high thermal conductivity to improve the dissipation of heat produced in the device in high power applications;
5. "manufacturing uniformity" in terms of homogenous layer characteristics and reproducible layer growth with a high yield, because high cost represent a significant road block in the development of RF devices.

Moreover, the thickness and the composition of the subsequent  $\text{Al}_x\text{Ga}_{1-x}\text{N}$  layer must be finely tuned in order to obtain a structural perfection at the  $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$  interface. The material grower must understand and solve all these growth issues related to group-III nitrides in order to create a suitable nitride heterostructure for HEMT application.

Metal organic chemical vapor deposition (MOVPE) is the most successful and suitable growth method for obtaining high quality group III nitride based materials. Nevertheless, the number of process parameters to tune the growth is very large (e.g. temperatures, thicknesses, molar flow rates and ratios...) and due to the coupling between them, the role of each is not clearly understood. Although a tremendous amount of work has been done these last years on optimizing the process parameters, a lot is still to be understood regarding the nitride growth mechanisms, particularly with regard to satisfying the aforementioned demands on HEMT structures.

Many failure mechanisms, originating from growth, limit the properties and lifetimes of HEMT structures due to several reasons:

- Dislocation density: Dislocations are indeed electrically active acting as acceptor like states and additionally as scattering centers for limiting transverse mobility. Control of dislocation types and densities is a prerequisite.

- Interface effects: the close proximity of the channel to the gate, essential for a high transconductance, makes the device susceptible to surface effects, e.g. trapping of mobile electrons. Passivation layers can help control these effects.
- DC-RF dispersion: Despite excellent DC and small signal characteristics of HEMTs, the measured large-signal power is much lower than expected. Power degradation is most likely caused by a loss of mobile electrons due to defect formation or impurities, thus some kind of trap in the material. For example a sufficiently high but comparably lower resistivity, i.e. lesser deep level states in the buffer, lowers this effect [Fan03].
- Layer characteristics uniformity: In large scale epitaxial equipment this factor is now widely controlled, however in single wafer reactors this is still an issue, not only affecting the yield but the distribution of layer characteristics. New process approaches become necessary.

## 1.2 Scope of this Thesis

Research aim of this work is to develop a MOVPE based growth process that allows the reliable and efficient deposition of high quality material for HEMT devices based on AlGaIn/GaN heterojunctions on sapphire substrates. The main task is to establish a broad comprehension of its epitaxial process based on numerical and experimental data and eventually to prove its functionality in terms of HEMT characteristics.

Epitaxy takes place in an Aixtron 200/4 RF-S horizontal reactor system. Beginning in chapter 2 the main structural and electrical properties of GaN are presented to establish the basis for device applications. The MOVPE process is discussed in chapter 3. Particularly this chapter deals with the growth limiting factors such as the hydrodynamics and the mass transport. Chapter 4 presents the applied characterization methods, since they provide the main tools to investigate the GaN properties and introduces the experimental setup used in this work. The following chapters deal with the growth optimization of HEMT device relevant thin films on sapphire substrates. In this context a lot of work has already been done in our labs [Ste03, Kal03] thus this thesis ties in with earlier results.

Since power HEMT devices consume large areas of typically several square-millimeter reproducibility and uniformity in terms of thickness and epilayer characteristics are paramount. However these are still open issues. Usage and development of adequate growth control and monitoring tools are essential to meet these premises especially with respect to III-nitride growth.

To this end a new approach for temperature determination will be investigated in chapter 5, that makes growth conditions on transparent substrates comparable, independent of the reactor type used. Moreover and more importantly for the following discussion, a reliable

basis for temperature determination is established, that facilitates in-situ measurement and control of the substrate temperature which is crucial for reproducible layer quality.

A new deposition approach is introduced in chapter 6 that takes care to avoid parasitic depositions on the reactor walls as these have been found to be the origin of irreproducibility [Har04]. Numerical simulations and growth modelling will be presented and the commonly applied growth process is compared to the new one.

Homogeneity in terms of thickness and epilayer characteristics is addressed next in chapter 7. In order to achieve a homogeneous distribution of growth species over the substrate, flow mechanical aspects in our reactor system are investigated. The carrier gas nature is therefore an important optimization parameter. For that purpose the role of different carrier gases - nitrogen and hydrogen - on III-nitride growth is examined. A necessity for the application in HEMT structures is a highly conductive GaN channel. Thus -in addition to a highly uniform GaN template- the optimization of morphology, structural and electrical quality is required and addressed as last subject in chapter 7.

In chapter 8 AlGaIn/GaN heterojunctions are grown with the main focus on controlled Aluminium content and AlGaIn thickness. The abrupt- and smoothness of the heterostructure interface will be studied. However, HEMT structures have additional needs in the form of highly resistive buffers to avoid parasitic parallel conduction in the device structure. Therefore the influence of the morphological development on the resistivity is studied in chapter 8, concurrently not losing sight of the above mentioned layer qualities. Finally HEMT device results will demonstrate the suitability of the growth approaches used.

At last all results will be summarized and an outlook on future research will be provided.



## Chapter 2

# Properties of III-Nitrides and their Application in HEMTs

Wide bandgap semiconductors afforded by group III-nitrides pave the way for high temperature and high power devices, because of their chemical and thermal durability. In this chapter the main structural and electrical properties will be devised. Furthermore a brief introduction to high electron mobility transistors (HEMT) based on GaN/AlGaN heterojunctions is given.

### 2.1 Physical Properties

In contrast to cubic III-V semiconductors, such as GaAs and InP, which crystalize in the cubic *zincblende* structure, the thermodynamically stable structure at ambient conditions for the three main compositions AlN, GaN and InN is the hexagonal *wurtzite* (so called  $\alpha$ -phase) [Amb98] [Mor98].

The wurtzite structure has a hexagonal unit cell and thus two lattice constants, the height of the hexagonal prism  $c$  and the edge length  $a$ . An internal parameter  $u$  is defined as the anion - cation bond length along the (0001) axis in units of  $c$ . It contains 6 atoms of each type. The space group for the wurtzite structure is  $P6_3mc$  ( $C_{6v}^4$ ). The wurtzite structure consists of two interpenetrating hexagonal close packed (HCP) sublattices, each with one type of atom, offset along the  $c$ -axis by  $5/8$  of the cell height (Figure 2.1).

Due to the lack of inversion symmetry, the wurtzite structures has a polar axis on [0001] direction. This results in planes terminating with either all group III atoms or all nitrogen atoms as seen in 2.2. The two different polarities may be defined: Ga-polarity (designated [0001], pointing from the cation to the anion along the long bond ( $c$ -axis) and corresponding to a gallium layer on the top position of the (0001) plane and N-polarity

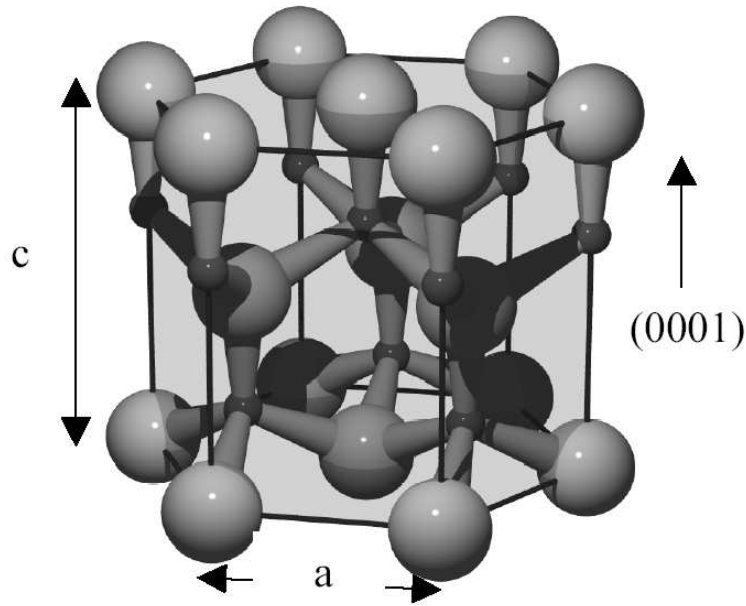


Figure 2.1: Stick and ball diagram of wurtzitic GaN. According to the ionic radii the small spheres represent nitrogen while the big ones represent Gallium.

(designated  $[000\bar{1}]$ ) for which nitrogen atoms are on top (Figure 2.2).

It is worth noticing that Ga-faced does not mean Ga-terminated, termination being a surface property connected to the possible reconstructions. The discussed polarities might be obtained by using different growth techniques and substrates. It has been reported that high quality epitaxial GaN layers deposited by MOVPE on *c*-plane sapphire substrates grow in the (0001) direction with Ga-faced surfaces, while MBE growth commonly occurs in the  $(000\bar{1})$  direction, yielding an N-faced GaN layer [Amb98].

The polarity, the non-centrosymmetric structure and the partial ionicity of the chemical bonding in the group III nitrides are responsible for the polarization effects which appear in the nitride layers. The physical properties of bulk and surface, as there are piezoelectric effects (described in subsection 2.2.1) depend significantly on the surface polarity [Amb98].

An overview of important values for crystal properties, mechanical and thermal constants and coefficients is given in table 2.1.

## 2.2 Electrical Properties of Nitrides

AlN, GaN and InN have different lattice constants, bandgaps and binding energies, because of the different ionic radii. The wurtzitic polytype of GaN, AlN and InN forms a

|                                                       | AlN               | GaN                     |
|-------------------------------------------------------|-------------------|-------------------------|
| Crystal Structure                                     | Wurtzite          | Wurtzite                |
| Lattice Constant $a_0$ [Å]                            | 3.112             | 3.189                   |
| Lattice Constant $c_0$ [Å]                            | 4.982             | 5.185                   |
| $a_0/c_0$                                             | 1.6010            | 1.6259                  |
| $u_0$                                                 | 0.380             | 0.376                   |
| $a_{Bohr}$ [Å]                                        | 5.814             | 6.04                    |
| Static Dielectric Constant $\epsilon_r$               | 8.5               | 8.9                     |
| High Frequency DC $\epsilon_{r,HF}$                   | 4.77              | 5.35                    |
| Energy Gap [eV]( $\Gamma$ Valley)                     | 6.2               | 3.39                    |
| Effective Mass $m_e$ ( $\Gamma$ Valley)               | 0.48              | 0.2                     |
| Electron mobility [cm <sup>2</sup> /Vs] (300K)        | 135               | 1000                    |
| Hole mobility [cm <sup>2</sup> /Vs] (300K)            | 14                | 30                      |
| Saturation velocity [cm/s]                            | $1.4 \times 10^7$ | $2.5 \times 10^7$       |
| Peak velocity [cm/s]                                  | $1.7 \times 10^7$ | $3.1 \times 10^7$       |
| Breakdown field [V/cm]                                |                   | $>5 \times 10^6$        |
| Thermal Conductivity [W/cmK]                          | 2                 | 1.5                     |
| $\rho$ [g/cm <sup>3</sup> ]                           | 3.29              | 6.07                    |
| Melting Point $T^M$ [K]                               | 3487              | 2791                    |
| Decomposition Point $T_E$ [K]                         | 1313              | 1123                    |
| Activation Energy $E_{MN}$ [kJ/mol]                   | 414               | 379                     |
| Spontaneous Polarization $P_{SP}$ [C/m <sup>2</sup> ] | -0.081            | -0.029                  |
| Piezoelectric Constants:                              |                   |                         |
| $e_{14}$ [C/m <sup>2</sup> ]                          | 0.92              | 0.375                   |
| $e_{15}$ [C/m <sup>2</sup> ]                          | -0.58/-0.48       | -0.3/-0.33/-0.22        |
| $e_{31}$ [C/m <sup>2</sup> ]                          | -0.48/-0.60/-0.58 | -0.49/-0.36/-0.33/-0.22 |
| $e_{33}$ [C/m <sup>2</sup> ]                          | 1.46/1.55         | 0.73/1/0.65/0.44        |
| Piezoelectric Coefficients:                           |                   |                         |
| $\epsilon_{11}$                                       | 9.0               | 9.5                     |
| $\epsilon_{33}$                                       | 10.7              | 10.4                    |
| Elastic constants:                                    | exp. cal.         | exp. cal.               |
| $C_{11}$                                              | 345 396           | 374 367                 |
| $C_{12}$                                              | 125 137           | 106 135                 |
| $C_{13}$                                              | 120 108           | 70 103                  |
| $C_{33}$                                              | 395 373           | 379 405                 |
| $C_{44}$                                              | 118 116           | 101 95                  |

Table 2.1: Crystal properties of GaN and AlN [Edg94] [Fou03].

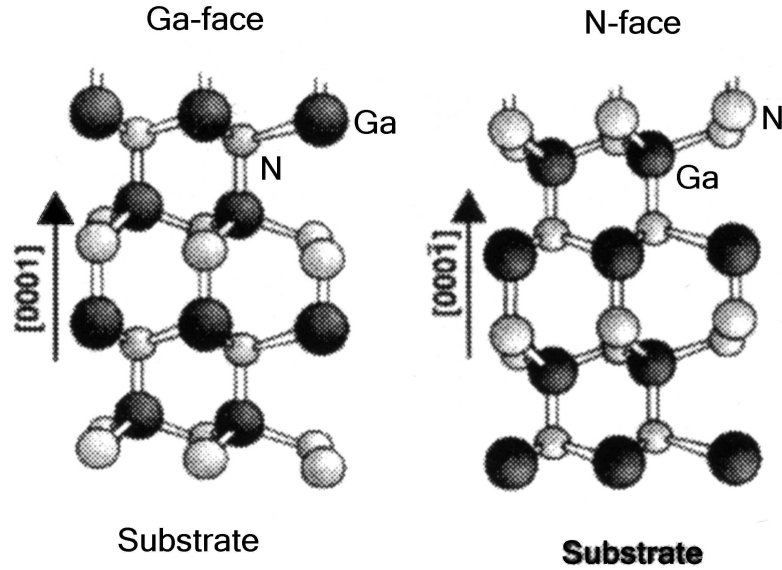


Figure 2.2: Different polarities (Ga- and N faced) of wurtzite GaN [Amb98].

continuous alloy system (AlGa<sub>x</sub>N<sub>1-x</sub>, InGa<sub>x</sub>N<sub>1-x</sub> and InAlN) whose direct bandgaps ranges from 0.7 eV for InN and 3.4 eV for GaN to 6.2 eV for AlN. Figure 2.3 displays the bandgaps versus the lattice constant.

Control of the electrical properties of group III nitrides remains the foremost obstacle hindering device efforts.

### 2.2.1 Spontaneous and Piezoelectric Polarization

Polarization related properties, such as spontaneous and piezoelectric polarization, are important for device applications. The electric field caused by polarization influences the shape of the band edges and the carrier distribution of heterostructure interfaces.

Pyroelectric materials exhibit polarization without an external electric field such as the *spontaneous polarization* in wurtzitic group III nitrides. For a crystal to exhibit spontaneous polarization it has to have a certain symmetry. Of the 32 point groups, which describe all crystalline systems, 11 are centrosymmetric, i.e. they contain an inversion center. In those crystals polar properties are not present, because there is no preferred direction. Ten of the initial 32 groups have a polar axis, which means they can display spontaneous polarization.

The wurtzite structure of the group III-nitrides is one of them. The origin of spontaneous

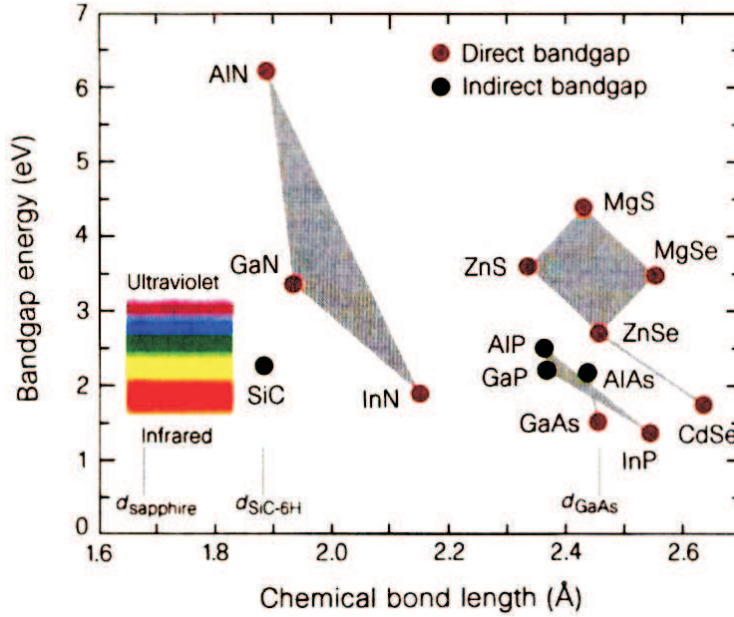


Figure 2.3: Bandgaps of important binary semiconductors.

polarization lies in the ionic or dipole character of the metal nitrogen bond and is always oriented along the crystal  $c$ -axis. The direction of the polarization (up or down the  $c$ -axis) can be controlled by controlling the polarity of the crystal. Spontaneous polarization can have significant influence on carrier concentration as well as band edge profiles, since a spontaneous polarization is connected with a surface charge. The polarization strength is dependent on the alloy composition and in general increases from GaN over InN to AlN and depends on the ionic character of the metal nitrogen bond (dipole character). Moreover every pyroelectric material is piezoelectric. In theory the strain-induced piezoelectric polarization in III-nitrides can equal the value of the spontaneous polarization. Spontaneous  $P_{Sp}$  and piezoelectric polarization  $P_{Pe}$  along the  $c$ -axis add up to the macroscopic polarization  $P$ :

$$P = P_{Sp} + P_{Pe} \quad (2.1)$$

The sign of the spontaneous polarization is determined by the polarity and is opposite to the  $[0001]$  for metal-face layers. The piezoelectric polarization along the  $c$ -axis can be calculated by using the piezoelectric coefficients<sup>1</sup>  $e_{33}$  and  $e_{13}$  as

$$P_{Pe3} = e_{33}\varepsilon_3 + e_{31}(\varepsilon_1 + \varepsilon_2) \quad (2.2)$$

<sup>1</sup>The calculated values of the piezoelectric constants in GaN, InN, and AlN are up to ten times larger than in GaAs based crystals and the sign is opposite to other III-V compounds.

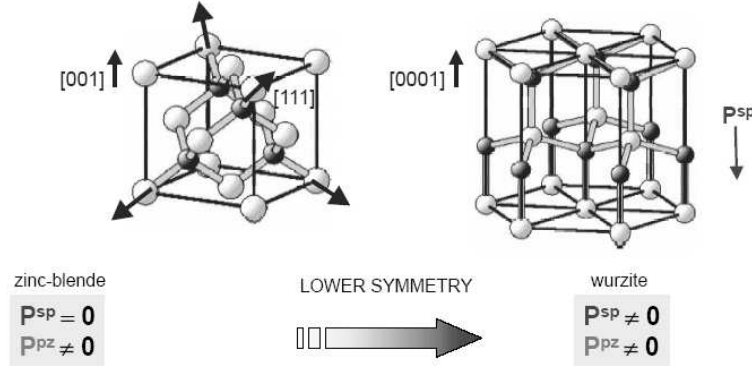


Figure 2.4: Sum of the microscopic dipoles, resulting in a spontaneous polarization  $\neq 0$  in the wurtzite structure (right) and  $= 0$  in the zincblende (left).

where  $\varepsilon_3 = (c - c_0)/c_0$  is the strain along the  $c$ -axis,  $\varepsilon_1 = \varepsilon_2 = (a - a_0)/a_0$  is the isotropic in-plane strain,  $a_0$  and  $c_0$  are the equilibrium values of the lattice parameters.

For a hexagonal structure, the relation between the lattice constants is given by

$$\frac{c - c_0}{c_0} = -2 \frac{C_{13}}{C_{33}} \frac{a - a_0}{a_0} \quad (2.3)$$

where  $C_{13}$  and  $C_{33}$  are the elastic constants (table 2.1 ).

Using equations 2.2 and 2.3 the amount of piezoelectric polarization in the direction of the  $c$ -axis can be determined by

$$P_{Pe_3} = 2 \frac{a - a_0}{a_0} \left( e_{31} - e_{33} \frac{C_{13}}{C_{33}} \right) \quad (2.4)$$

The macroscopic polarization results in an electric field:

$$E = - \frac{P}{\varepsilon_{Material} \varepsilon_0} \quad (2.5)$$

where  $\varepsilon_0$  is the dielectric constant of vacuum and  $\varepsilon_{material}$  is the relative dielectric constant of the employed material.

For  $Al_xGa_{1-x}N$  alloys the piezo polarization equals

$$\left( e_{31} - e_{33} \frac{C_{13}}{C_{33}} \right) < 0 \quad (2.6)$$

and is valid over the whole range ( $x$ ) of compositions. Therefore from Eq. (2.4) the piezoelectric polarization is negative for tensile and positive for compressive strained AlGa<sub>N</sub> films respectively. As a consequence, the orientation of the piezoelectric polarization is parallel to the spontaneous polarization for tensile strain and antiparallel in case of compressively strained AlGa<sub>N</sub> layers.

Summarizing, polarization itself and gradients in polarization at interfaces and surfaces of AlGa<sub>N</sub>/Ga<sub>N</sub> heterostructures induce fixed sheet charges, which in turn cause strong electric fields inside every heterostructure. In the nitrides the electric field can reach a strength of  $3 \cdot 10^6 \text{ V/cm}$  and therefore enhance electron or hole accumulation (depending on the polarity of the material) at AlGa<sub>N</sub>/Ga<sub>N</sub> interfaces.

### 2.2.2 HEMT-Structures Based on AlGa<sub>N</sub>/Ga<sub>N</sub> Heterojunctions

The High Electron Mobility Transistor (HEMT) has emerged as a promising candidate for microwave power amplification. HEMTs are an integral part of a new generation of group III nitride transistors formed between semiconductors of different compositions and band gaps. These devices offer advantages for instance in microwave and high speed digital integrated circuit (IC) applications but most of all as high power devices.

#### HEMT Heterostructures

The unique feature of the HEMT is channel formation from carriers accumulated along a heterojunction [Sze81, Hei01]. Whatever the physical origin of the carrier accumulation, the carriers have high mobility due to their separation from their source region and their location in a region absent of impurity scattering. Where carriers accumulate a so called two-dimensional electron gas (2DEG) is formed. Usually the origin of the carrier is a junction between a heavily doped high bandgap and a lightly doped low bandgap region. In HEMTs based on AlGa<sub>N</sub>/Ga<sub>N</sub> structures, this carrier accumulation is mainly due to polarization charges developed along the heterojunction in the high bandgap AlGa<sub>N</sub> side. A heterostructure as described above is shown in figure 2.5.

In inset (a) the Fermi levels  $E_F$  of the two semiconductors do not coincide. At a real heterostructure in thermodynamic equilibrium, this will lead to a bending of the bands, until the structure will have just one common Fermi level (inset (b)). The heterostructure results in the formation of a discontinuity in the conductance ( $E_C$ ) and valence ( $E_V$ ) band at the heterojunction. A triangular quantum well emerges, filled with electrons diffusing from semiconductor II and thus the strongly localized 2DEG develops. The term 2DEG refers to the condition in which electrons have quantized energy levels in one spatial direction but are free to move in the other two directions, parallel to the interface. While figure 2.5 is valid for all compound semiconductors for group III nitrides also polarization

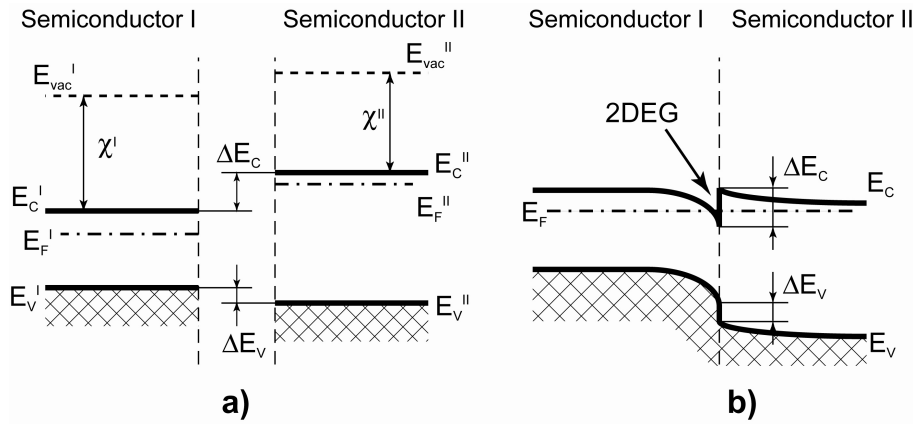


Figure 2.5: Band diagram of the heterostructure formed by lightly n-doped narrow gap semiconductor I and heavily n-doped wide gap semiconductor II divided one from another (a) and together in thermodynamical equilibrium [Iba99].

effects have to be taken into account.

### AlGaIn/GaN HEMTs

The carrier accumulation in the quantum well in the AlGaIn/GaN heterostructure is caused by the sum of spontaneous and piezoelectric polarization in the strained<sup>2</sup> AlGaIn layer and the spontaneous polarization in the GaN layer. At the heterojunction the polarization induced sheet charge is positive, thus free electrons will compensate this charge resulting in the formation of a 2DEG. Since polarization effects depend on the Al-content, higher carrier concentrations can be induced with higher Al amounts in the solid. For the Ga-face polarity the induced sheet charge is found to be in the AlGaIn layer near the interface (see figure 2.6 a) and therefore the 2DEG is formed in the GaN layer. For the N-face polarity the polarization has the opposite direction what results in the formation of the 2DEG in the GaN close to the top interface (figure 2.6 b) [Amb00].

The reason why the AlGaIn/GaN heterostructure has been so intensively studied for the last years is the fact, that the polarization in the layer system is large enough to form a 2DEG with high sheet carrier concentrations *without* the necessity of a doped layer in the structure.

Due to these material properties applications at elevated temperature (HEMT operation at 750°C have been shown [Dau98]) are possible and in comparison with GaAs etc., higher breakdown voltages, high saturated electron drift velocities and much higher thermal and

<sup>2</sup>The piezoelectric polarization, present solely in the thin AlGaIn layer, originates from the tensile strain imposed on the AlGaIn layer due to the GaN layer underneath.

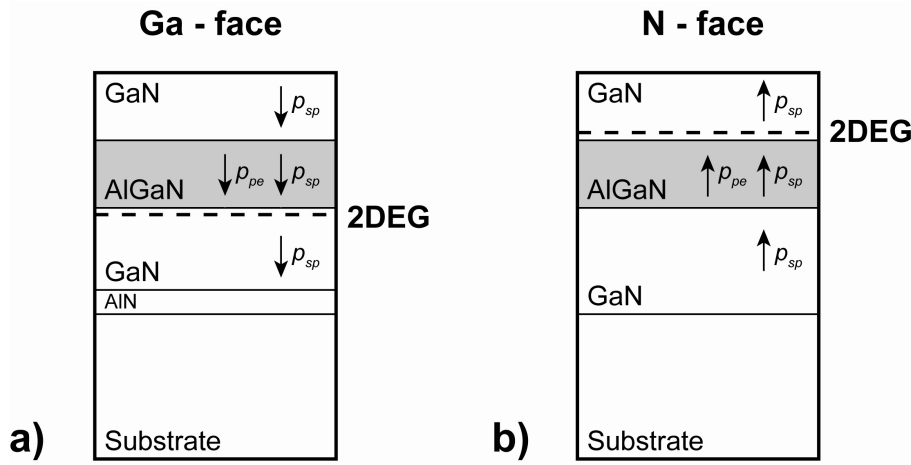


Figure 2.6: Polarisation induced charge in a Ga-faced (a) and N-faced (b) AlGaIn/GaN heterostructure

chemical stability are achieved. For the AlGaIn/GaN system simulations have predicted a high peak electron velocity of  $2.7 \cdot 10^5 m/s$  at electric fields of  $10^5 V/cm$ . A mobility of  $\mu_e \approx 2000 cm^2/Vs$  has been calculated for the 2DEG in the GaN channel at room temperature and a sheet carrier concentration of  $n \approx 10^{13} cm^{-2}$ . Together, these material properties yield the capability of about ten times as much microwave power density in  $W/mm$  channel periphery [Eas02].

In order to grow a HEMT with such sophisticated features several requirements on growth have to be fulfilled:

1. homogenous layer characteristics
2. Semi-insulating GaN layers to avoid any parallel conductivity to the channel layer and to ensure high HEMT performance at high frequency;
3. good structural quality with low dislocation densities for high amplification by high channel conductivity at high frequencies.
4. A smooth, defect free surface, important for the growth of sharp interfaces and flat surfaces as well as for device processing;
5. Tight alloy composition and thickness control in the heterostructure
6. Maximization of the sheet conductivity  $\mu \times n_s$ , thus maximizing both the sheet charge density  $n_s$ , necessitating the maximization of the spontaneous and piezoelectric polarizations with the Al mole fraction and the electron mobility  $\mu$  requiring the minimization of scattering centers due to dislocations, interface roughness and alloy disordering at the heterojunction.

For device applications an intermediate Al content of about 30 % seems most promising, typically resulting in a sheet carrier density of  $1 \times 10^{13} \text{cm}^{-2}$  and a 2DEG mobility of 1200 to 1500  $\text{cm}^2/\text{Vs}$  at 300K for an AlGa<sub>N</sub> layer thickness of 30 nm grown on 2  $\mu\text{m}$  semi-insulating Ga<sub>N</sub>. Usually the sheet carrier density increases with Al content in the AlGa<sub>N</sub> layer. The mobility of the 2DEG though, decreases with increasing Al-content. Following the model of Zhang and Singh [Zha99], the higher the sheet carrier density and the sheet charge, the closer the centroid of the 2DEG density is to the interface, and the more the electron transport is affected by the surface roughness. Hence, 30 % content is assumed to represent an optimum composition. Heterostructure growth is completed with the deposition of a Ga<sub>N</sub> cap layer.

### Electron Transport in AlGa<sub>N</sub>/Ga<sub>N</sub>

The formation of a 2DEG without intentional doping in AlGa<sub>N</sub>/Ga<sub>N</sub> heterostructures offers a novel phenomena that must be taken into account when determining the transport properties in the channel. However, in comparison to AlGaAs/GaAs 2DEGs, where the mobility is limited by Coulomb scattering induced by remote ionized donors [Smo99, Hir86], the electron mobilities in AlGa<sub>N</sub>/Ga<sub>N</sub> heterostructures are still lower. A short review of scattering events will be given:

**Ionized Impurities** The separation of electrons from their donors due to the polarization charges reduces the scattering and improves the electron mobility. Therefore scattering due to ionized impurities is solely caused by the background doping concentration that is usually in the order of  $10^{16} \text{cm}^{-3}$ .

**Alloy Scattering** Alloy disorder scattering originates from the randomly varying alloy potential in the barrier. In AlGaAs/GaAs heterostructures this scattering effect is negligible but in AlGa<sub>N</sub>/Ga<sub>N</sub> heterostructures, the combination of the large electron mass, the high sheet carrier concentration, and the large alloy potential make this scattering strong.

**Phonons** The phonon scattering limits the mobility in the 2DEG in the range of temperatures above 80K. The transport is affected by potential acoustic phonons, piezoelectric acoustic phonons, and polar optical phonons [Nag80].

**Dislocations** Due to the lattice mismatch between substrate and film, Ga<sub>N</sub> layers contain typically  $10^8$  to  $10^{10} \text{cm}^{-2}$  of dislocations. Thus changes in the periodicity of the lattice lead to local potential changes and so to scattering events. Some threading dislocations form acceptor like states and act as coulomb scattering centers.

**Charged Surface Donors** In an undoped heterostructure such as AlGa<sub>N</sub>/Ga<sub>N</sub> the question arises where free electrons originate from. A possible source of electrons seems to be surface donor states[Amb99], since the mentioned background carrier concentration is not high enough to produce such a sheet carrier density in the 2DEG. The surface donor states will form a source of scattering [Riz00] since they diminish the polarization induced sheet charge and transport scattering occurs similar to a delta-doped remote donor layer.

**Interface Roughness** Interface roughness scattering limits electronic transport of high density 2DEG's ( $10^{13} \text{cm}^{-3}$ ), because the 2DEG shifts closer to the interface as the sheet carrier concentration increases.

Thus, the electron transport properties of the 2DEG are limited by small angle scattering for low sheet carrier concentrations and by large angle scattering for high sheet carrier concentrations. Dependence of the 2DEG mobility on the temperature and scattering mechanisms is shown in figure 2.7.

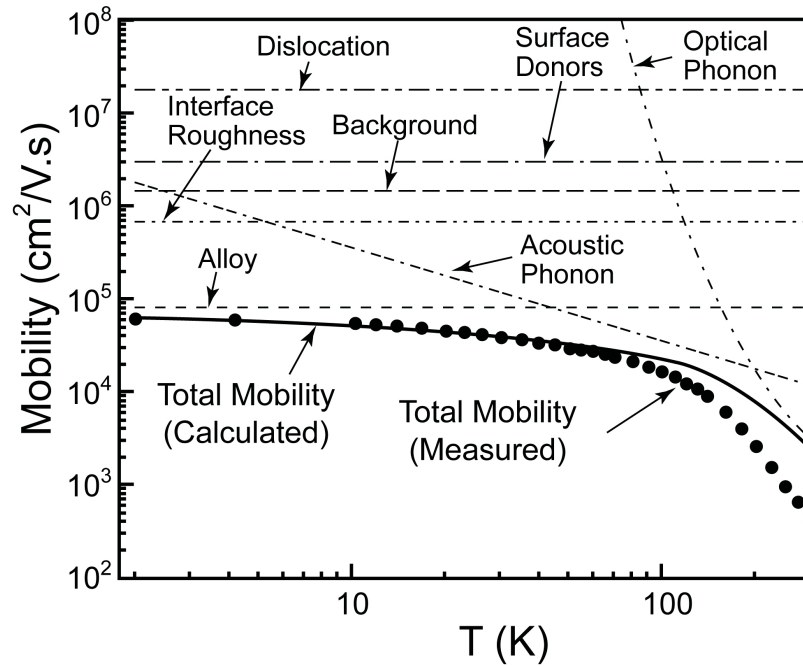


Figure 2.7: Dependence of the 2DEG mobility on the temperature and scattering mechanisms for AlGa<sub>N</sub>/Ga<sub>N</sub> heterostructures [Jen01].



# Chapter 3

## Principles of MOVPE GaN Growth

Usually, the production of compound semiconductor based devices requires the growth of high quality epitaxial layers (low defect and impurity levels, precise thickness of the layers close to the monolayer scale, abrupt transitions in composition and sharp interfaces). Metal-organic vapor phase epitaxy<sup>1</sup>(MOVPE) has been developed in the 1960's by Manasevit et al. [Man69], MOVPE, also known as metal-organic chemical vapor deposition (MOCVD), is a deposition method in which a thin solid film is synthesized by the chemical reaction of molecules from the gaseous phase on a heated surface. CVD processes are very versatile and suitable for the deposition of both semiconductor materials and coatings. In this work MOVPE has been used to grow (Al)GaN films on sapphire substrates. These films can be produced from several microns down to a thickness on the atomic layer scale, which is important in order to produce atomically sharp heterointerfaces e.g. for HEMT applications.

In the following the MOVPE process for III-V semiconductors is briefly introduced and the governing fundamentals are emphasized. Subsequently MOVPE will be discussed with particular interest in GaN growth.

### 3.1 MOVPE Process

Among different deposition methods employed for obtaining epitaxial layers such as vapor phase epitaxy (VPE), liquid phase epitaxy (LPE), molecular beam epitaxy (MBE), hydride vapor phase epitaxy (HVPE), MOVPE is the most versatile and suitable method. The MOVPE has evolved into a leading growth technique for III-V compound semicon-

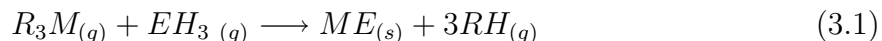
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<sup>1</sup>The term epitaxy derives from the Greek. *Epi*, meaning *on top* or *onto* and *taxis* which translates to *ordering*. Therefore epitaxy denotes the controlled growth of a substance on a single crystalline base, the substrate.

ductor materials, such as GaAs, AlAs, InP and their alloys. Beside the fact that MOVPE meets all the quality criteria for the epitaxial layers required for device applications, it also possesses the advantage to grow on large area substrates. Due to the developments in the area of optoelectronic applications, the MOVPE became the most successful and mature growth technique for group III nitrides based materials. All the breakthroughs in the MOVPE growth of group III nitrides such as heteroepitaxial growth on mismatched substrates and introduction of a low temperature nucleation layer, achievement of p-type doping, control of doping level in the nitride layers and the possibility to grow high quality (defined from structural, optical and electrical point of views) GaN, AlGaN and InGaN layers qualified the method for the growth of GaN based HEMT structures. It is the growth method used in this thesis and its basic principles will be discussed in the next sections.

### 3.1.1 General Considerations

MOVPE is a non-equilibrium growth technique which is based on the vapor transport of the precursor molecules, i.e. for group III-V based compounds, group III alkyls (denoted  $R_3M$  where  $R$  is an organic radical, typically  $CH_3$  or  $C_2H_5$  and  $M$  is a metal from group III, Al, Ga or In) and binary hydrogen compounds of group V (denoted  $EH_3$  where  $E$  is a nonmetal from Group V, N, P or As) and their subsequent reactions on a heated substrate resulting in epitaxial growth of a thin solid film. In a simplistic manner, which neglects the complexity of the MOVPE process, a general reaction can be written as follows:



The precursors are transported by an accurately metered flow of carrier gas (usually highly pure  $H_2$  and/or  $N_2$ ) into a reaction chamber. The carrier gas passes through the group III sources<sup>2</sup> to saturate with vapor from the source and carry a certain amount of material into the reactor. Because of the high vapor pressure the group V precursor solely flows by pressure difference.

The MOVPE process is very complex and consists of a number of reactions involving both homogeneous and heterogeneous reactions of the group III and group V source molecules as well as growth related surface processes such as adsorption/desorption of the chemical species and surface migration. The carrier gas will transport the source molecules from the reactor inlet to the heated zone of the reactor where they will undergo gas phase reactions, typically decomposition reactions (in the nitride growth case even adduct formation reactions). The resulting species diffuse due to concentration gradients

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<sup>2</sup>Alkyls of group III elements are usually in liquid form contained in so called "bubblers", which are held in thermostats with a controlled temperature and thus possess a well defined vapor pressure.

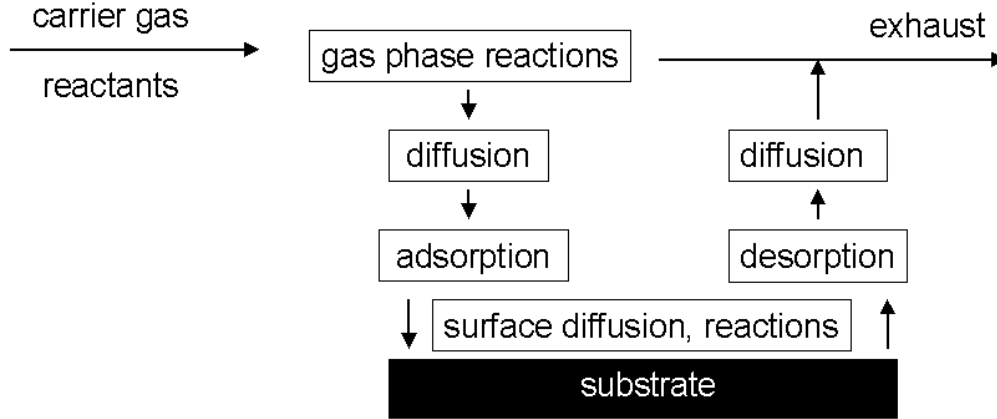


Figure 3.1: Process steps in CVD reactions

in the gas phase to the substrate surface where they are adsorbed at a vacant site. At this point, the species can either desorb or react with other surface species (or simply decompose), then diffuse along the surface before the final incorporation reactions take place and the resulting product is incorporated into the film forming a new bond. The gaseous byproducts that form desorb from the sample surface and diffuse in the carrier gas away from the deposition zone towards the reactor exhaust. The basic processes in MOVPE are displayed in figure 3.1.

The MOVPE growth process consists of many parallel and series reaction steps occurring simultaneously. For series reactions, the slowest step controls the overall reaction rate. For parallel reactions, the overall reaction rate is the sum of the individual reaction rates, hence the fastest reaction controls the overall reaction rate. For such a complex system, the most important elementary steps determining the overall reaction rate is controlled by an interplay between the three components: thermodynamics, kinetics and hydrodynamics (also including mass transport)[Hol96, Har03, Str99].

- The *thermodynamics* of growth, which describe the driving force for the overall growth process by indicating in which direction the reactions will proceed and predicting the solid stoichiometry, as depicted in section 3.1.2.
- The *kinetics* of growth, shown in section 3.1.3, define the rates at which the possible gas phase and surface reactions occur.
- The *hydrodynamics* (section 3.1.4) of the growth process controls not only the mass transport of the molecules in the gas phase, but also the temperature distribution, the velocity profile and total pressure in the reactor.

Examining the crystal growth mechanisms it is apparent that a major parameter in CVD growth is the deposition temperature. As MOVPE is an exothermic process, the growth rate can be expected to be a complicated function of the temperature according to the Arrhenius equation:

$$k = A \exp(-E_a/RT) \quad (3.2)$$

where  $k$  is the rate constant,  $A$  is the pre-exponential factor,  $E_a$  is the activation energy,  $R$  is the gas constant and  $T$  is the absolute temperature. Qualitatively, three growth temperature regimes [Sha75] can be established from a plot imaging the growth rate versus reciprocal temperature (Fig. 3.2):

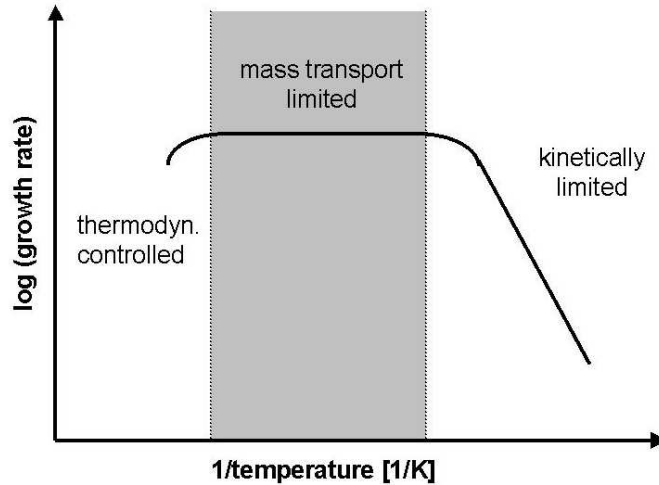


Figure 3.2: Typical Arrhenius plot of a CVD deposition process

- *kinetically limited* regime: At low temperatures the growth is determined (limited) by the reaction rates at the substrate surface. The growth rate increases exponentially with the reciprocal temperature.
- *mass transport limited* regime: at intermediate temperatures the chemical kinetics becomes faster, and the mass transport (gas diffusion to and away from the surface) is the rate limiting step. The growth rate becomes independent of the substrate temperature. Hydrodynamics influence the layer growth.
- *thermodynamically limited regime*: at high temperatures the thermodynamic equilibrium shifts to educt formation and hence the growth rate decreases. Also alternative processes such as depletion of reactants must be considered.

For each growth regime there are also other factors which may influence the overall growth rate besides temperature. In the kinetically limited case the surface plays an important role. When the mass transport limits the overall growth rate, parameters such as total flow, flow velocity and reactor pressure must be taken into account. For the thermodynamically limited regime where growth rates decrease due to possible depletion routes (adducts formation, wall deposits), parameters such as reactor geometry, including gas inlet design and time residence may influence considerably the growth.

All these parameters define together the optimal parameter window for a growth process. Usually the mass transport limited growth regime is favored, since variations in temperature will not affect the epilayer thickness. However, an analysis of the three components which govern the MOVPE process can provide a better understanding. Hence, in the next subsections few generalities about thermodynamics, kinetics and hydrodynamics for an MOVPE growth process will be discussed.

### 3.1.2 Thermodynamics

In CVD, growth is simply a phase transition from the gaseous to the solid phase. The basic goal of thermodynamics as applied to epitaxy, is to define the relationship between the compositions of the phases in an equilibrium system at constant temperature and pressure and hence thermodynamics determine the maximum growth rate for all growth processes. The driving force for deposition is the striving of the whole system towards equilibrium [Pie92]. It is therefore useful to know the equilibrium conditions. The Gibbs free energy function  $G$  per mole - also called free enthalpy - provides the true measure of chemical affinity under conditions of constant temperature and pressure. A system will be in equilibrium when  $G$  is at a minimum [Car94]. The free energy change of a chemical reaction can be described as

$$\Delta G_r = G_{products} - G_{reactants} \quad (3.3)$$

When the free enthalpy change is zero, there is no net work obtainable. The system is in a state of equilibrium. If the free energy change is positive for a reaction, net work must be put into the system to effect the reaction, otherwise the reaction cannot take place. If the free energy change is negative, the reaction can proceed spontaneously with accomplishment of net work. Therefore  $\Delta G_r$  is called the driving force of the reaction<sup>3</sup>.

For the calculation of the free energy of the reaction mixture,  $\Delta G_r$ , first the standard free energies of formation for the pure reactants and the pure products are employed, giving the standard reaction free energy,  $\Delta G_r^0$  :

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<sup>3</sup>The partial derivative of the free energy  $\frac{\partial G}{\partial n_i}$  with respect to the number of moles  $n$  per component  $i$ ,  $n_i$ , is known as the *chemical potential*  $\mu_i$ . The chemical potential denotes the required amount of energy for the phase transition.

$$\Delta G_r^0 = \sum n \Delta G_f^0(\text{products}) - \sum n \Delta G_f^0(\text{reactants}) \quad (3.4)$$

where  $n$  denotes the stoichiometric coefficients in the chemical equation. While  $\Delta G_r^0$  is the difference in molar free energy between the pure products and the pure reactants in their standard states (for a temperature  $T$  and a pressure of 1 *atm*),  $\Delta G_r$  refers to any chosen, fixed composition of the reaction mixture. Thus, the value of  $\Delta G_r$  varies with the composition of the reaction mixture. If the reaction mixture contains relatively more reactants than it would have at equilibrium, then  $\Delta G_r$  at that composition will be negative, and the system will have the tendency to form more products. For a higher proportion of products than at equilibrium, then  $\Delta G_r$  will be positive and the reverse reaction, the formation of the reactants, will be spontaneous. At an equilibrium composition of the reaction mixture,  $\Delta G_r = 0$  and there is no tendency for the reaction to occur in either direction.

The dependence of  $\Delta G_r$  on the composition of the reaction mixture can be written as

$$\Delta G_r = \Delta G_r^0 + RT \ln Q \quad (3.5)$$

where  $Q$  is the reaction quotient and  $R$  is the gas constant (8.31451 kJ/K·mol). The reaction quotient is defined in the same way as the equilibrium constant,  $K$ . If  $K$  is the ratio of the concentrations (or partial pressures) of the products to those for the reactants, each raised to a power equal to its stoichiometric coefficient in the balanced chemical equation, the reaction quotient defines the same ratio but at any stage of the reaction.  $Q$  is expressed in terms of partial pressures,  $p_i$  (where  $p_i = x_i P_T$ ,  $x_i$  being the molar fraction of species  $i$  and  $P_T$  the total pressure) for any gases or  $a_i$ , activity of species  $i$  ( $a_i = x_i \gamma_i$ , where  $\gamma_i$  is a non-ideality factor called also activity coefficient).

At equilibrium,  $\Delta G_r = 0$ , therefore the left-hand term of Eq. 3.5 is equal to 0 and  $Q = K$ , the equilibrium constant for the reaction and the equation becomes

$$\Delta G_r^0 = -RT \ln K \quad (3.6)$$

This is the link between the equilibrium constant and the standard reaction free energy for any change, physical or chemical. The value of  $\Delta G_r^0$  gives us information about the yield of the desired reaction at the equilibrium conditions for the reaction mixture. Moreover, from all the tabulated thermodynamic data, phase diagrams of the system in equilibrium state can be constructed, helping to determine the energetically favorable phases as a function of experimental variables (temperature, composition).

A disequilibrium situation is intentionally created by supersaturation of the reactants that drives the system to produce the solid desired. This is the driving force for epitaxy.

The maximum amount that can be produced is simply the amount that would establish equilibrium and is, thus, fundamentally limited by thermodynamics.

### 3.1.3 Kinetics

Thermodynamics define the equilibrium conditions but cannot give information on the course of a reaction. The kinetics of growth gives more insight on the reaction paths. In MOVPE homogeneous (gas phase) and well as heterogenous reactions (reactions between gas and sample surface) take place. Where homogeneous reactions have been extensively studied by mass spectroscopic techniques, the investigation of the heterogeneous reactions is still deficient because of lack of experimental tools needed for the measurement of the processes at the growing surface. Stringfellow [Str01] notes that "*the least understood aspect of the entire MOVPE growth process is probably the array of surface reactions and physical phenomena occurring at the solid/vapor interface during growth*". The difficulties arise from the fact that both homogeneous and heterogeneous reactions occur simultaneously, involving several precursors and their intermediates, apart from inhomogeneities in temperature and so forth. Also, reaction mechanism will be affected by so-called "system effects" such as system pressure, reactor geometry and flow conditions. In this context an universally valid reaction mechanism for the same growth process in different reactors is hardly conceivable. To this end numerical simulations and process modelling are useful to understand possible growth pathways together with the transport phenomena in the reactor. As a result, kinetics will here be described with a rather simplistic approach.

For a simple bimolecular reaction with reactants  $A$  and  $B$  which form the product  $P$ , the equilibrium constant  $K$  can be written as [Moo78]:

$$K = \frac{k_1}{k_{-1}} = \frac{[P]}{[A][B]} \quad (3.7)$$

here  $k_1$  and  $k_{-1}$  are the reaction rate constants of the reaction in forward (towards the product) and reverse direction. Each reaction rate constant is not dependent on the concentration of the participating species but is dependent on the temperature according to the equation of Arrhenius (3.2)[Ben68, Ben82]:

The difference in activation energy for both reactions  $E_{a,1}$  and  $E_{a,-1}$  is equal to:

$$\Delta H_r = E_{a,1} - E_{a,-1} \quad (3.8)$$

with the reaction enthalpy  $\Delta H_r$ . The schematics of the formation heat as a function of the distance along the reaction path for a simple bimolecular reaction is shown in figure 3.3.

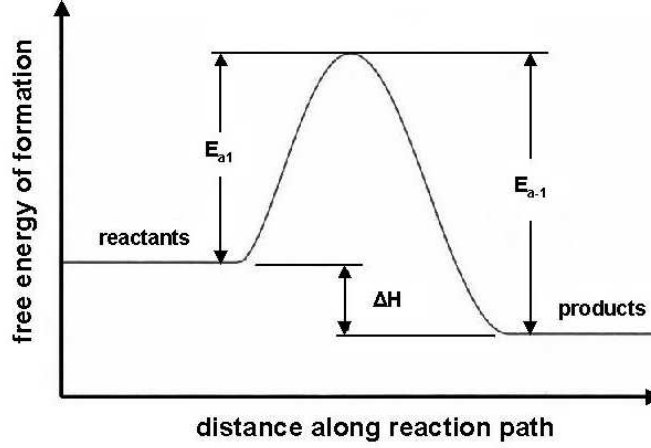


Figure 3.3: Schematic of the formation heat as a function of the distance along the reaction path.

For a **homogeneous reaction** (gas phase reaction) the rate constant  $k_1$  can be resolved as [Str99]:

$$k_1 = P_A P_B \frac{\pi d_{AB}^2}{\sigma_{AB}} \sqrt{\frac{8RT}{\pi \mu_{AB}}} \cdot N_A \cdot e^{-E_a/RT}, \quad (3.9)$$

where  $P_A$  and  $P_B$  are the probabilities that a collision between molecules  $A$  and  $B$  will proceed to reactants (basically depending on orientation factors),  $d_{AB}$  is the distance between the centers of mass of  $A$  and  $B$  in the collision complex,  $\sigma_{AB}$  is the symmetry number for the pair (1 for  $A \neq B$ , and 2 for  $A = B$ ), and  $\mu_{AB}$  is the reduced molecular weight ( $M_A M_B / (M_A + M_B)$ ) and  $N_A$  is AVOGADRO's number.

The **heterogeneous reactions** at the sample surface dominate the overall process. The surface has been thought to consist of atomically flat regions separated by steps, which can be visualized by atomic force microscopy. In this mode growth occurs by propagation of steps as well as by the formation and propagation of two- and three-dimensional islands. The physical procedure occurring on the surface during growth is depicted in figure 3.4. A simple model for the interaction at the surface is presented by the LANGMUIR adsorption. Providing that adsorption and desorption in a stationary state are equal, the surface coverage  $\Theta$  can be expressed by the LANGMUIR isotherm:

$$\Theta_i = \frac{K p_i}{1 + K p_i} \quad (3.10)$$

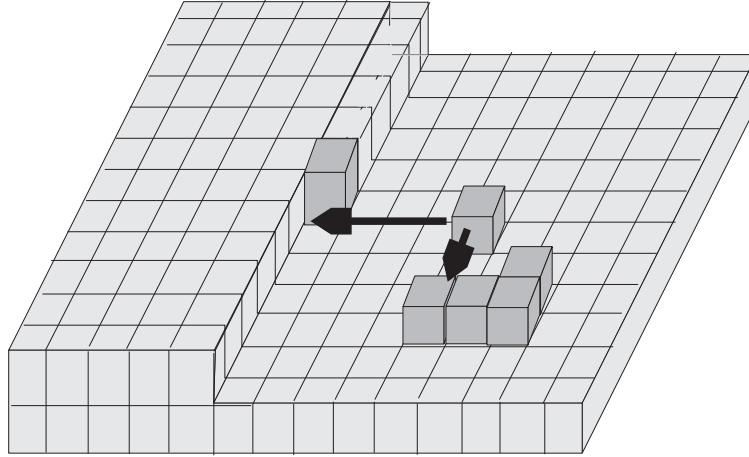


Figure 3.4: Adatom motion and preferred adsorption at kinks and islands.

$K$  is the equilibrium constant at the surface, here also the adsorption constant and  $p_i$  the partial pressure in the vapor of species  $i$ . Since this chemisorption process is exothermic the value of  $K$  and hence the surface coverage decreases with increasing temperature. Thus, the reaction rate is proportional to  $\Theta$ . Assuming that the reaction would be bimolecular for the III-V semiconductor growth process, the reaction rate  $r_g$  is proportional to the coverage of anion and cation species

$$r_g = k_1 \Theta_A \Theta_B \quad (3.11)$$

It should be mentioned that the LANGMUIR model is too simplistic to describe the actual adsorption of gas molecules on the surface. It has to be taken into account that the adsorbed atoms are known to diffuse to the lowest energy position of the surface before being incorporated into the growing film. Thus, imperfections such as screw dislocations, stacking faults and so forth, may be preferred sites. Other effects due to impurities collected on the surface (surfactant effects) also disturb ideal growth.

It was shown that the chemistry of the CVD process is quite complicated since a number of homogeneous and heterogeneous chemical reactions take place. However the slowest reaction is most important since it will determine the growth rate of the film and is therefore often mainly accounted for in the modelling of the growth process.

### 3.1.4 Hydrodynamics and Mass Transport

The hydrodynamics are the last fundamental component which describe the overall MOVPE process. Transport phenomena (fluid flow, heat transfer and mass transport) finally gov-

ern the access of the reactive species to the sample surface and influence the degree of gas phase reactions taking place before deposition. The knowledge of the phenomena can be obtained by modelling the growth process [Kle95]. To this end conditions are put into calculation which are defined by the CVD experiment. The constraints are the process properties such as reactor geometry, total flow, reactor pressure and substrate temperature and thermodynamic properties belonging to the reactants and products (this presupposes that the chemical model of deposition is known) and their partial pressures. In this way it becomes possible to determine the temperature distribution in the gas phase, the flow profiles and the distribution of the important chemical species for growth.

### CVD Model Equations

Modelling can be used to find a set of start parameters in the case of a growth process developed for a new material system or a new reactor type. In case of a well known material system, modelling can help find the optimal operating conditions in order to achieve significant growth rates of high quality layers and spatial uniformity of the layer thickness over large-area substrates. However, modelling cannot totally replace the experiments. Information from both experiment and modelling are needed to develop a better understanding of the growth process.

The physical treatment of the hydrodynamic aspects in the growth of III-V semiconductors is governed by the simultaneous solution of non-linear, coupled, partial differential equations for the mass transport, the homogenous and heterogenous kinetics and heat transfer. The transport phenomena are described through *mass, momentum and energy conservation* [Bir60].

Conservation of mass:

$$\frac{\partial}{\partial t}\rho_i = -\nabla \cdot (\rho_i \vec{v} + \vec{j}_i) = -\nabla \cdot \vec{n}_i \quad (3.12)$$

The total mass flux  $\vec{n}$  from equation (3.12) is the sum of convective and diffusive fluxes. Where  $\rho$  is the mass density,  $i$  denotes the species,  $\vec{v}$  the average mass velocity and  $\vec{j}$  the diffusive flux .

Navier-Stokes equation (conservation of momentum):

$$\frac{\partial}{\partial t}\rho \vec{v} = -[\nabla \cdot \{\rho \vec{v} \vec{v} + \mathbf{\Pi}\}] + \sum_i \rho_i \vec{g}_i \quad (3.13)$$

With  $\mathbf{\Pi}$  as the pressure tensor and  $\vec{g}_i$  as sum of all external forces, equation (3.13) states that any momentum change results from convective flow, internal pressure, viscous forces and external forces such as gravity.

Conservation of energy:

$$\frac{\partial}{\partial t} \rho \left( E + \frac{1}{2} v^2 \right) = -\nabla \cdot \left\{ \rho \left( E + \frac{1}{2} v^2 \right) \vec{v} + \vec{q} + [\Pi \cdot \vec{v}] \right\} + \sum_i (\vec{n}_i \cdot \vec{g}_i) \quad (3.14)$$

From the first law of thermodynamics the rate of gain in energy is principally formed from convection, conduction ( $\vec{q}$  is the conductive heat flux), the energy dissipation due to compression and friction, and external forces.

To specify the system it is assumed that the ideal gas law  $pV = nRT$  is applicable since precursor and product partial pressures are low. The diffusive flux  $\vec{j}_i$  to the substrate surface in equation (3.12) combines ordinary FICKIAN diffusion (driven by concentration gradients) and thermodiffusion, or SORET diffusion (driven by thermal gradients).

$$\vec{j}_i = \vec{j}_i^c + \vec{j}_i^T \quad (3.15)$$

Simplified the flux of the growth limiting species  $i$  can be described as [Ric86]

$$\vec{j}_i = \frac{D_i}{kT} \nabla p_i + \frac{\alpha_i D_i}{kT} p_i \nabla T, \quad (3.16)$$

where  $D_i$  is the diffusivity,  $\alpha_i$  is the thermodiffusion factor and  $k$  Boltzmann's constant.

Thermodiffusion usually has a weak effect on the overall diffusive flux, however, it plays a role in MOVPE processes, since the steep temperature gradient in a reactor is in a several hundred degrees Celsius range. In a nonisothermal reactor the gas is suddenly heated by the susceptor. The resulting change in density by virtue of gas expansion, gives rise to *buoyancy* driven convection (*natural* convection). The thermodiffusion results in the transport of heavy molecules (in respect to the gas mixture, eg. heavy metalorganics) into cooler regimes opposed to the concentration driven flux. This depletion of the group III compounds in the region close to the substrate results in a reduction of the growth rates by 10 - 20% relative to the growth rate achievable in the absence of thermal diffusion [Jen94].

### Characteristic Parameters

The basic transport processes are characterized by dimensionless parameter combinations that arise from the scaling of the governing transport equations (3.12), (3.13) and (3.14):

The *Knudsen number*  $Kn$  denotes the mean free path  $\lambda$  between collisions for a gas molecule relative to the characteristic length  $L$ . In a horizontal reactor with a rectangular profile (height  $h$ , width  $w$ )  $L$  can be calculated by use of the hydraulic diameter. Thus,  $Kn$  is small for MOVPE reactors.

$$Kn = \frac{\lambda}{L} < 0.01 \quad \text{with} \quad L = 4 \frac{hw}{2(w+h)} \quad (3.17)$$

The continuum description in equation (3.12),(3.13) and (3.14) is then appropriate, otherwise molecules would not undergo gas phase reactions, since they are rarely subject to gas phase collisions.

The dynamical behaviour of the reactant gas can be characterized by the *Reynolds number*. The Reynolds number  $Re$  is the relation between kinetic energy and frictional loss

$$Re = \frac{vL}{\varrho}, \quad (3.18)$$

where  $\varrho = \frac{\eta}{\rho}$  stands for the kinematic viscosity, defined as ratio of viscosity  $\eta$  and density  $\rho$ .  $Re$  gives information whether a flow is laminar or turbulent. In case of an isothermal liner an empirical value of  $Re > 2300$  has been determined above which the flow becomes chaotic. The transport regime will be dependent on the nature of the carrier gas employed ( $\varrho$ ) in the MOVPE growth process. Giling calculated the Reynolds number for 4 different carrier gases ( $H_2$ , He,  $N_2$ , Ar). He found that the Reynolds number depends on temperature, as a consequence of the variation of the density and viscosity of each gas with temperature and that for  $N_2$  and Ar, at high flow rates, a turbulent regime can be obtained. Also, for He or  $H_2$ , at high flow rates, a so called "entrance effect" can be observed, which means that the gas flow and temperature profiles are not fully developed and turbulent zones can appear inside of the reactor. However,  $Re$  is typically low for conventional MOVPE reactors. In the setup used in this work the Reynolds number is at least one order of magnitude lower than necessary for the tip over to turbulent flow.

The *Rayleigh number*  $Ra$  serves as a measure of the strength of natural convection in the system, therefore characterizing the thermal behaviour of the gas flow.  $Ra$  gives the relation between buoyancy force and viscous force

$$Ra = \frac{\alpha g c_p h^3 \rho^2 \Delta T}{\eta \kappa}, \quad (3.19)$$

where  $\alpha$  is the thermal expansion coefficient,  $c_p$  is the specific heat of gas,  $h$  is the free height of top wall above substrate,  $\Delta T = T_{hot} - T_{cold}$ ,  $\kappa$  is the thermal conductivity of gas, and  $g$  is the gravitational constant. When heated gas is beneath cold gas, for example in a MOVPE reactor due to the heated susceptor and cold walls, a driving force for natural convection exists. With no forced convection, the transition from stagnation to convection occurs for values  $Ra > 1707$ . The  $Ra$  number for several temperatures for  $H_2$  and  $N_2$  were calculated by [Gil86a]. For normal geometries and temperatures no convection is predicted for  $H_2$ , but  $N_2$  is expected to be highly convective. Of course if the gas encounters an obstacle such as a tilted heated susceptor or if the reactor height is too large, natural convection can result even in  $H_2$ . Temperature gradients are the primary cause of density variation in CVD systems, since the reactants are often used in such low concentrations that the depletion of reactants by film growth is incapable of creating significant density variations in the fluid.

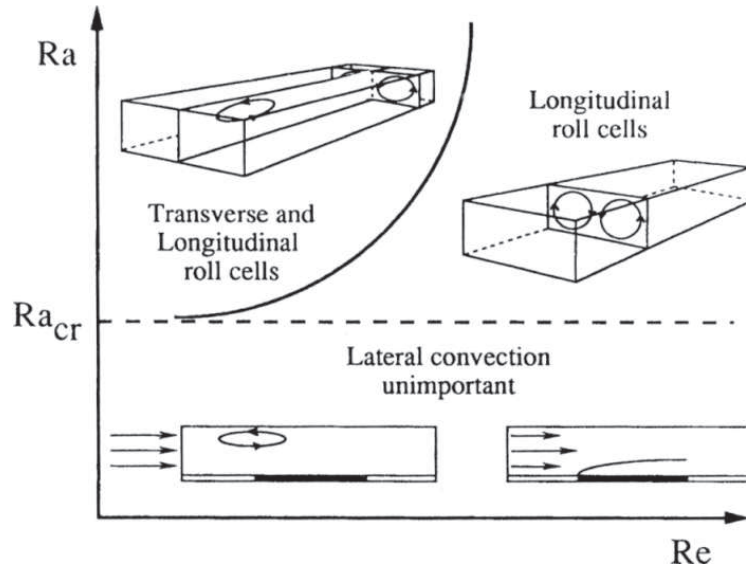


Figure 3.5: Schematic diagram of the Rayleigh number versus the Reynolds number, classifying the different flow behavior expected in horizontal CVD reactors [Jen93].

### Flow Phenomena in Horizontal Reactor Systems

The aforementioned critical values give a first impression of transport phenomena, but for real MOVPE reactors (in our case horizontal), a mixed convection mechanism is responsible for the transport. Density gradients in reduced and atmospheric pressure reactors are caused primarily by temperature gradients and thus may produce buoyancy driven flows that superimpose on the forced flow entering the reactor. This results in mixed convection transport and hence has significant effects on film thickness and uniformity. In this situation, the flow dynamics will be affected by each detail of the reactor geometry (dead spaces, obstacles or angles between the internal surfaces larger than  $10^\circ$ ) and flow recirculation cells, roll cells or turbulent(vortex) motions can be observed. Figure 3.5 shows the flow regions in a horizontal geometry based on the Rayleigh-Bénard analysis.

Above a critical value  $Ra_{cr}$  two dimensional convection cells, so called longitudinal rolls, develop downstream of the entrance region. The direction of rotation depends on the wall thermal boundary condition, as well as the inlet conditions. Jensen [Jen93] found out that for an aspect ratio of 2 for the reactor cross-section and *insulated side walls* the rolls rotate inwardly, thus increasing deposition in a midplane. For *cold side walls*, where flow rotates outwardly deposition increases near the walls and thus decreasing in a midplane. As the roll pattern develops, the downward flow in the reactor center causes lateral nonuniformity in growth rate.

The hydrodynamics approach of the MOVPE growth process demonstrates the complexity

of the flow phenomena in an MOVPE reactor. Consequently, most deposition characteristics are reactor dependent. The "manufacturing uniformity" of semiconductor layers is regulated by the reactor geometry coupled with the fluid dynamics and heat and mass transfer. This makes the translation of experimental data for one reactor to another reactor type and also the scale-up of reactors very uncertain. As a result of this complexity and despite their disadvantages (time consuming, high costs), trial-and-error methods are still important in developing new MOVPE growth processes in these circumstances.

### 3.1.5 Influence of Carrier Gases

The carrier gas plays a decisive role in MOVPE. It transports the group III metalorganic and the group V source compounds into the heated zone of the reactor where decomposition processes take place to form the desired highly pure materials. The gas phase (i.e. the reaction medium) in which the chemicals partly decompose mostly consists of carrier gas (around 70 – 95%)<sup>4</sup>. This has several consequences for growth:

- The hydrodynamical properties of the carrier gas such as density, viscosity, heat conductance and specific heat capacity determine the flow velocity- and temperature distribution profiles in the reactor chamber. Therefore the transport and the residence time of the growth species in the gasphase is significantly influenced.
- Through their respective masses carrier gas molecules initiate reactions of the growth species in the gasphase when acting as collision partners. Besides the number of collisions the impulse transmission is of importance for activation.
- The carrier gas itself might be product of one of the many reaction paths, thus reaction rates are determined by the carrier gas partial pressure.
- An influence of the carrier gas on surface reaction, absorbtion and desorption is also conceivable

Since we employed hydrogen and nitrogen (as well as arbitrary mixtures of both gases) as the carrier gas in our reactor system, some fundamental properties of these are reviewed here. These characteristics have a significant influence not only on hydrodynamics but on the gasphase reactions taking place in the reactor. The ratio for several properties between the two applied carrier gases hydrogen and nitrogen is given as

$$\frac{\rho_{N_2}}{\rho_{H_2}} = 14 \quad \text{Density} \quad (3.20)$$

---

<sup>4</sup>lesser amounts of carrier gas in nitride growth than for classical III-V's, see section 3.3)

$$\frac{\eta N_2}{\eta H_2} = 2.5 \quad \text{Viscosity} \quad (3.21)$$

$$\frac{\lambda N_2}{\lambda H_2} = 0.15 \quad \text{thermal conductivity} \quad (3.22)$$

$$\frac{c_p N_2}{c_p H_2} = 0.08 \quad \text{specific heat capacity} \quad (3.23)$$

These differences have several consequences in growth:

For instance, the greater mass of  $N_2$  relative to  $H_2$  transmits higher kinetic energies to its collision partner, which in turn leads to a higher decomposition of the precursors.

Lower thermal conductivity of  $N_2$  in comparison to  $H_2$  helps to achieve a uniform temperature distribution on the substrate surface.

The diffusion constant of reactive species in nitrogen is lower than that for hydrogen which can account for lower growth rates.

Especially for nitrogen flows in a high temperature ambient (i.e high Rayleigh numbers as seen in equation 3.19) the existence of complex flow structures were observed by Giling [Gil86b]. Namely the presence of a region of colder gas (so called *cold finger*) penetrating from the inlet into the deposition zone. This cold finger arises from radiative heating of the top wall and the subsequent heat transfer to the incoming gas. Hence, if the flow rate is low and the thermal conductivity is high, heat is conducted sufficiently fast relative to convection (3.19), so that there are no large temperature gradients from susceptor to the reactor top wall. However, if the gas velocity is fast and/or a gas with lower thermal conductivity (e.g.  $N_2$ ) is used a thermal boundary layer develops along the top wall.

Furthermore  $N_2$  is a nonexplosive gas, leading to a safer overall MOVPE process.

## 3.2 Sapphire as Substrate for (Al)GaN growth

While for GaAs and InP the existence of large area substrates makes homoepitaxial growth possible, for GaN this option is not available. The high dissociation pressures and melting temperatures are the main obstacles in obtaining large group III nitride single crystals, which can serve as substrates for homoepitaxial growth. Therefore single crystalline GaN films have been grown heteroepitaxially on several substrates, which match more or less closely to its lattice constants. Also different coefficients of thermal expansion between substrate and nitrides cause residual stress upon cooling, which leads to additional structural defects. There is *no ideal or particularly suitable* substrate available in that matter.

However, providing the thickness  $h$  of the epitaxially grown film is sufficiently small a pseudomorphic layer can be grown. The structure of a mismatched layer is shown in figure

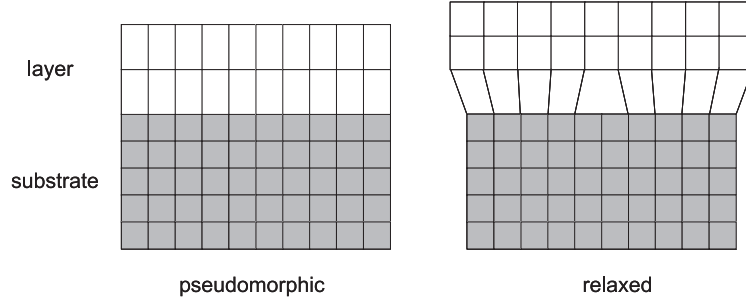


Figure 3.6: Structure of an epilayer under biaxial compression. Pseudomorphic (left) and relaxed (right) with misfit dislocations.

3.6. For such a pseudomorphic layer the misfit is accommodated by either *compressive* or *tensile* stress. The strain is tensile if the lattice constant of the epilayer is smaller than that of the substrate and compressive vice versa. For pseudomorphic layers the in-plane lattice constant adjusts to that of the substrate. Nevertheless distortion energy is caused by the stress in the grown layer. Increasing the layer thickness, therefore generating even more strain, results in more distortion energy. When the thickness exceeds a certain value, known as critical thickness  $h_c$ , plastic deformation is energetically favorable. Accordingly, the misfit strain begins to relax by the introduction of misfit dislocations [Mor98, Lant00].

Table 3.1 reviews the prospective substrate properties in comparison to GaN. However, sapphire is the only employed substrate in this work, therefore only its properties are discussed in more detail.

| Material                | Lattice constant<br>a ( $\text{\AA}$ ) | Lattice constant<br>c ( $\text{\AA}$ ) | thermal expansion<br>coefficient ( $\text{K}^{-1}$ )               |
|-------------------------|----------------------------------------|----------------------------------------|--------------------------------------------------------------------|
| GaN                     | 3.189                                  | 5.185                                  | $\alpha_a = 5.59 \cdot 10^{-6}$<br>$\alpha_c = 3.17 \cdot 10^{-6}$ |
| $\text{Al}_2\text{O}_3$ | 4.758                                  | 12.991                                 | $\alpha_a = 7.5 \cdot 10^{-6}$<br>$\alpha_c = 8.5 \cdot 10^{-6}$   |
| 6H-SiC                  | 3.08                                   | 15.12                                  | $\alpha_a = 4.2 \cdot 10^{-6}$<br>$\alpha_c = 4.7 \cdot 10^{-6}$   |
| 3C-SiC                  | 4.36                                   | -                                      | $\alpha = 2.7 \cdot 10^{-6}$                                       |
| GaAs                    | 5.653                                  | -                                      | $\alpha = 6.0 \cdot 10^{-6}$                                       |
| Si                      | 5.42                                   | -                                      | $\alpha = 2.56 \cdot 10^{-6}$                                      |

Table 3.1: Lattice constant and thermal expansion coefficient for heteroepitaxy of GaN on different substrates [Gil98].

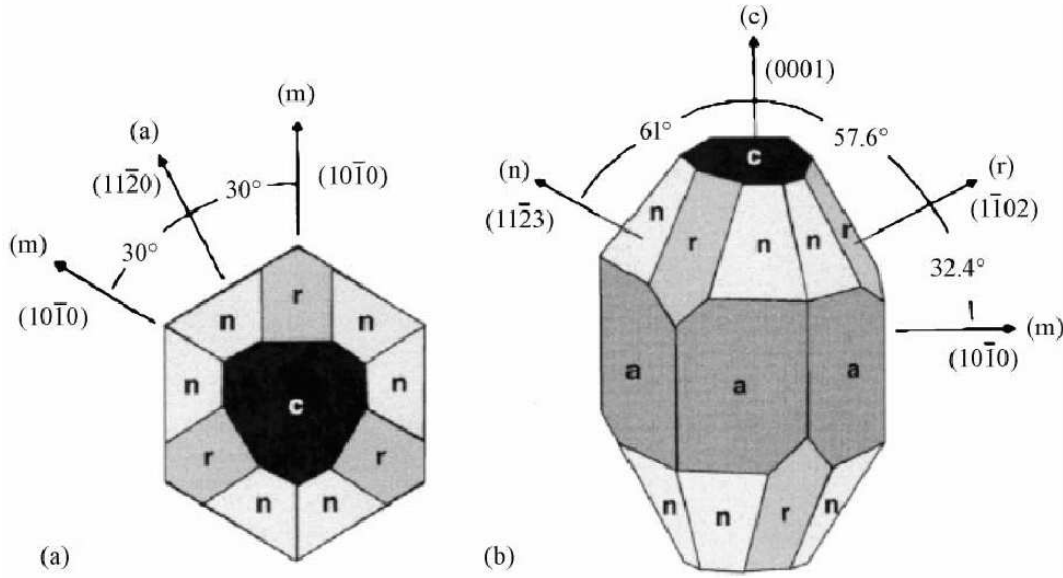


Figure 3.7: Rhombohedral structure and surface planes of the sapphire substrate.

### Sapphire $Al_2O_3$

Sapphire is the most extensively used substrate for growth of the group III nitrides and GaN in particular. Large area good quality crystals of sapphire are available at low cost. They are transparent, stable at high temperatures, and the growth technology of GaN on sapphire has reached a quite sophisticated level.

GaN can be deposited on one of the four planes of the corundum structure of sapphire, as there is the basal,  $c$ -plane (0001),  $a$ -plane ( $11\bar{2}0$ ),  $r$ -plane ( $1\bar{1}02$ ) and  $m$ -plane ( $10\bar{1}0$ ). The latter two are technologically not important due to the obtained low film quality. Figure 3.7 demonstrates the planes and their respective  $hkl$ -values.

The lattice mismatch between basal GaN and sapphire plane (both  $c$ -plane) can be calculated using  $\Delta a = (a_{\text{layer}} - a_{\text{substrate}})/a_{\text{substrate}}$ , where  $a_{\text{substrate}}$  is the effective lattice parameter of the substrate in the epitaxial plane. This mismatch leads to high dislocation densities ( $10^{10} \text{ cm}^{-2}$ ). Through a variety of techniques, such as low-temperature (LT) AlN or GaN nucleation layers [Ama86, Nak91], multiple intervening LT layers [Ama98], epitaxial lateral overgrowth [Bea01] and pendeoepitaxy [Zhe99] GaN layers with dislocation densities as low as  $10^7 \text{ cm}^{-2}$  have been produced. However, these defects have a number of effects on the layer characteristics. The electrical properties of the material and devices made from the material are altered, since defects lower sheet charge carriers concentrations, reduce the charge carrier mobility and thermal conductivity in heterostructure field effect transistors [Liu02]. Yet, defects are unavoidable and thus precise control of defect types and densities is a prerequisite for high quality GaN growth with respect to HEMT

application.

In this work all GaN films were grown on the *c*-plane, as it is the most common deposition direction.

### 3.3 MOVPE III-nitride Growth

The growth of group III nitrides is carried out by introducing a laminar flow of alkyls of the group III elements, such as trimethyl-gallium TMGa), and hydrides or hydrogen compounds of the group V elements, e.g. ammonia ( $NH_3$ ) into the reactor. The reactor itself consists of a quartz tube that contains a substrate. The substrate is situated on a silicon-carbide coated graphite susceptor heated by a radio frequency coil.

Neglecting the reactions of the precursors with the carrier gas, the net reaction between TMGa and  $NH_3$  taking place during growth of GaN can be indicated as:



In general, the supply of group III sources is lower than that of group V sources. For one thing, this is because of inferior decomposition of the group V molecules in comparison to the group III, since  $NH_3$  is thermally very stable. Even at the growth temperature used for HT growth only a few percent of ammonia is decomposed. On the other hand, more group V elements desorb from the substrate surface due to their volatility. Thus, high ammonia flows stabilize the stoichiometry of the growing layer.

However, decomposition and adduct formation of the source materials occurs in the gas phase even before film growth on the susceptor proceeds. In order to suppress these reactions caused by gas phase mixing a separation plate (see chapter 4) is introduced into the reactor, right in between the inlets of group III and V sources. Formation of adducts and so forth would lead to untimely depletion of source materials in the gas phase and hence to reduced growth rates. Using the separation plate, mixing takes place right before the heated susceptor.

Besides the aforementioned growth affecting parameters even the carrier gas has a distinct effect on growth. Obviously hydrodynamical properties of the carrier gas change the transport but at the elevated temperatures, where GaN growth takes place, hydrogen decomposes and influences the growth chemistry in the gasphase and at the surface level, for example by etching the layer. This is a particularly useful effect in the early stages of heteroepitaxial growth, e.g. during the so-called annealing step, where single islands form that coalesce later on. In contrast, when growing InGaN/GaN Multi-Quantum-Wells nitrogen is used as the carrier gas in order to increase the indium incorporation rate, when growing the active regions. Thus, in literature there have been many reports how  $H_2$  or

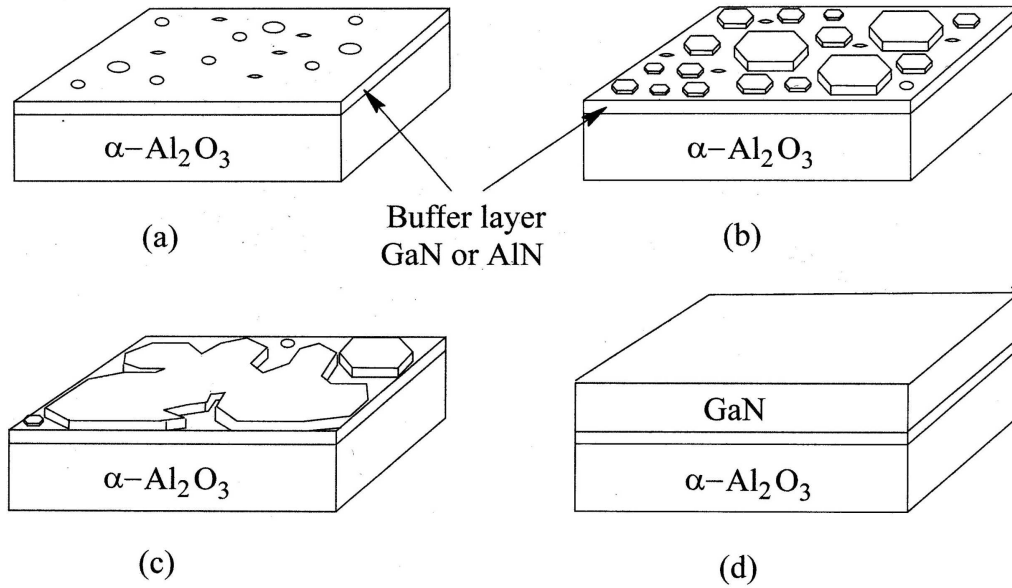


Figure 3.8: Growth model for GaN on sapphire substrates.

$N_2$  carrier gas changes the growth and properties of GaN epilayers. Some of them are quite controversial. In chapter 7 we will have a closer look at these issues.

Surface reactions however dominate GaN growth. GaN surfaces have a catalytic effect on the decomposition of ammonia adsorbed on the surface. The reaction products  $NH_x$  produce the active nitrogen necessary for GaN growth [Liu78]. At low temperatures 400–600°C used for nucleation layer deposition (see below), growth is kinetically controlled by the catalytic decomposition of the absorbed  $NH_3$  on the sample surface and by methyl radicals blocking Ga sites. At intermediate temperatures 600–1050°C growth is limited by the diffusive transport of the group III species to the surface. At higher temperatures growth becomes thermodynamically controlled and is limited by Ga desorption from the sample surface. Since both the LT and the HT GaN growth are carried out in regimes where surface kinetics are important and where growth is extremely temperature dependent, substrate surface temperature is the most important growth parameter which needs to be controlled. Additionally since GaN deposits have a catalytic effect on especially  $NH_3$  decomposition, the absence and the presence of deposits in the reactor chamber as well as on the substrate also will strongly influence growth.

In contrast to the typical III-V compound semiconductors (phosphides, arsenides and antimonides), for which the epitaxial growth process is well understood, the MOVPE growth of group III nitrides is challenging. This fact is due the constraint of heteroepitaxy imposed by the lack of suitable lattice matched substrates. In order to achieve good nitride epilayers, a two steps growth procedure was proposed by Amano and Akasaki

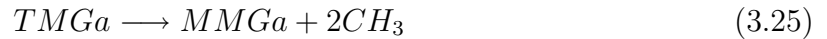
[Ama86, Aka89] (Figure 3.8).

In the first step, a GaN nucleation layer is deposited on the substrate at low temperatures (400-600°C for sapphire), followed by annealing. Then the main epilayer is grown in the second step at a higher temperature (usually  $> 1000^\circ\text{C}$ ). The nucleation layer must act as a template between the substrate and the main epilayer. First, the sapphire is covered with GaN nuclei, randomly distributed which furthermore will develop during the annealing step and finally will coalesce in the first part of the high temperature step, allowing the growth of a smooth nitride epilayer with improved structural and electrical properties. This development of morphology can conveniently be monitored by means of in-situ reflectometry as introduced in chapter 4.

### 3.3.1 Modelling of GaN Growth

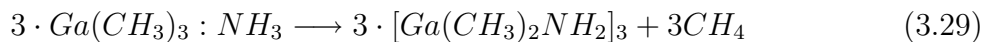
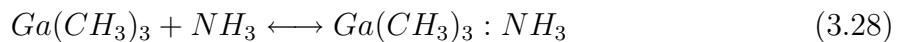
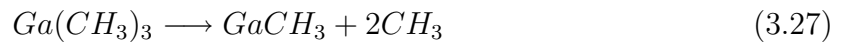
The principal characteristics of the inverted and conventional inlet growth processes were studied and compared using modelling. The modelling was performed by the Semiconductor Technology Research GmbH in Erlangen. The mathematical model is based on the solution of the 2D/3D Navier-Stokes conservation equations describing flow, heat transfer, and multi-component diffusion of the gaseous mixture inside the reactor [Bir60], taking into account the specific reactor geometry, the inlet design and the presence of wall deposits and particles. However, numerical simulation can only be performed for the high temperature deposition regime.

The chemical growth process is modelled starting from the following reaction equations. The products of TMGa decomposition are MMGa and atomic gallium. Gas-phase chemical reactions include two-step pyrolysis of the initial metal-organic precursor TMGa as presented in (3.25) and (3.26).



Fairly high temperatures in the reactor result in intensive TMGa pyrolysis producing MMGa, which, in turn, also decomposes to form atomic gallium which is the species providing the greatest contribution to growth.

Additionally, the set of Gas-phase reactions for GaN growth from TMGa and  $NH_3$  used in the computations is [Mih99]:



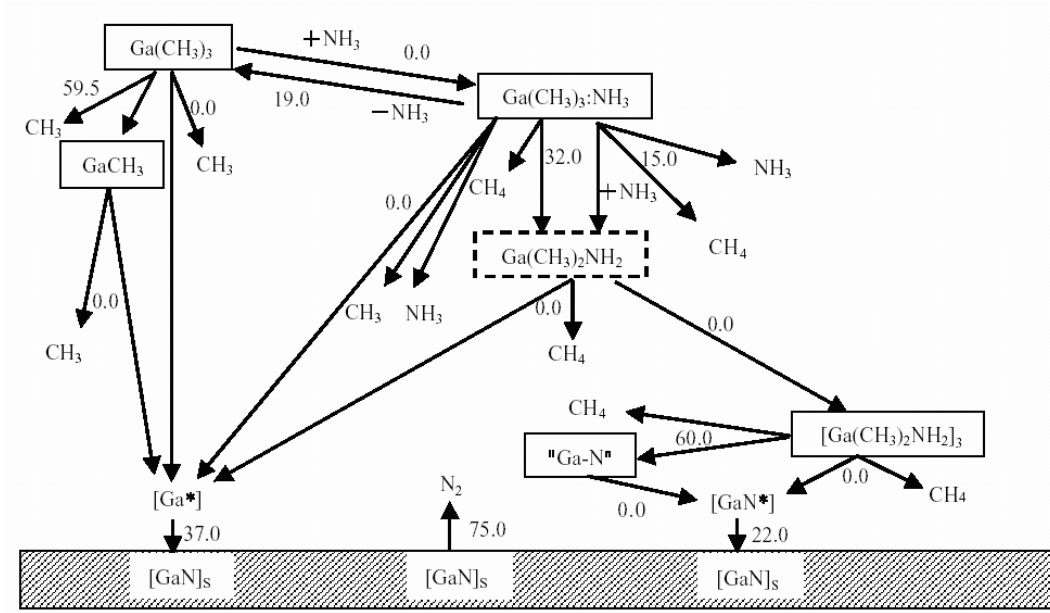
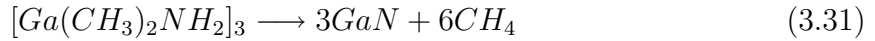
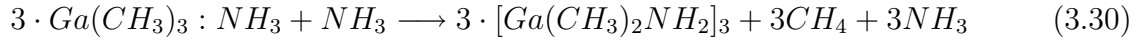


Figure 3.9: Schematic of the GaN decomposition and reaction pathways. Activation energy ( $kcal/mol$ ) is shown for each reaction pathway. Here,  $GaN^*$  and  $Ga^*$  are adsorption species on the surface. Based on the work of Mihopoulos [Mih99]



The kinetic mechanism of MOVPE gas-phase reactions is based on the work of Mihopoulos [Mih99]. The mechanism consists of six reactions and eight species which are shown in the above equations. The schematic of the GaN decomposition and reaction pathways is shown in figure 3.9.

To simulate the surface chemistry, a quasi-thermodynamic approach is used. This approach is based on the following assumptions:

- The atoms in the adsorption layer on the surface are nearly in equilibrium with the crystal bulk. This means that the rates of atomic incorporation into the crystal and of the crystal decomposition (occurring at the step edges) are much higher than their net difference (growth rate).
- Kinetic effects occurring at the stage of adsorption/desorption can be accounted for by the sticking/evaporation coefficients of the individual species. Generally, these coefficients are dependent on temperature and the partial pressures of the species.

A further discussion of hydrodynamic effects is found in section 3.1.4.

# Chapter 4

## Experimental Setup and Characterization Methods

In this chapter the setup of the employed MOVPE system as well as the applied methods for characterization of the deposited layers are discussed. In particular the reactor itself is described as well as the in-situ tools (reflectometry and pyrometry) emphasized since they represent the main tool to control, investigate and interpret the GaN growth in this work.

### 4.1 The Horizontal MOVPE Reactor (Aix 200 RF-S)

The MOVPE system used for the epitaxial growth of thin GaN films in this work was designed for the deposition at reduced pressures. The main constituents of the equipment are

- reactants and gas handling system,
- the reactor,
- the vacuum and exhaust system,
- the control unit

A more detailed description of these compounds and their application can be found in several publications [Jen94] [Sch03a]. In this work, all GaN growth runs were carried out in an AIX 200/4 RF-S horizontal reactor built by Aixtron.

## Gas Handling System and Sources

The reactant handling system mixes and meters the gases to be used in the reactor unit. According to the requirements the carrier gas for the gaseous sources can be switched between nitrogen ( $N_2$ ) and hydrogen ( $H_2$ ). For the growth of GaN the precursors trimethylgallium (TMGa), as gallium source, and ammonia ( $NH_3$ ), as nitrogen source, were used. Furthermore trimethylaluminum (TMAI) as aluminium source as well as  $n$ -type ( $SiH_4$ ) dopants are available. By use of mass flow and pressure controllers the total gas flow is accurately metered and controlled. The metal-organic group III compound and the group V nitrogen source are fed to the gas inlet in the reactor head via two separate lines (*Run MO* and *Run Hydrides*), in order to avoid any reactions between the educts. The gas inlet of the reactor was additionally equipped with valves in order to be able to choose between the conventional <sup>1</sup> and the inverted inlet configuration <sup>2</sup>(see below). Parallel to the two main lines that lead into the reactor a third is available that directs the gas flow alongside the reactor, the *Vent line*. As a result the flows can be stabilized before they are introduced into the reactor. Both Run and Vent line are pressure-wise controlled in order to afford equal pressure conditions and hence preventing pressure surge in the event of switching operation.

## The Reactor

The core of the MOVPE system consists of a cylindrical steel mantled, horizontal cold wall reactor. This steel setup was chosen in order to provide us with three optical windows. The whole construction is required to tolerate typical growth conditions for the deposition of high quality GaN with temperatures of 1100 °C and above, and at low pressures of 200 mbar and below.

The stainless steel reactor has three optical ports (as seen in figure 4.1), through which in-situ reflectometry measurements are accomplished as well as ellipsometry could be carried out. These ports are constantly rinsed with the carrier gas in order to avoid deposition with particles from inside the reactor which in turn would hinder in-situ measurement efforts. The reactor contains a quartz liner (which is the *actual* reaction chamber) with a rectangular cross section (13.1 cm × 4.5 cm). The void between liner and steel mantle is constantly flushed. Thus, the process gas cannot leave the liner.

Within the liner a so called *separation plate* is installed so as to reduce adduct formation between the group III and group V source, as shown in figure 4.2. The carrier gas, loaded with TMGa and  $NH_3$ , is transported onto the heated susceptor, where growth takes place. Usually, the  $NH_3$  enriched flow enters the reactor beneath the separation plate, while the MO flow enters from above the separation plate. The inlet configuration may as

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<sup>1</sup>Metalorganic sources through the upper channel, ammonia through the lower channel.

<sup>2</sup>Vice versa to the conventional inlet.



Figure 4.1: The AIX 200/4 horizontal reactor equipped with in-situ reflectometry and pyrometry (EpiR-TT)

well be permutated. Using a radio frequency coil, which is situated underneath the liner, the graphite (or molybdenum) susceptor is heated by means of electromagnetic induction of eddy currents. The maximum power of the RF-generator is  $30\text{ kW}$  at a frequency of  $55\text{ kHz}$ . Gas foil rotation enables the susceptor to rotate at values around  $60\text{ rpm}$ , hence resulting in homogeneous growth on the substrate. During growth, temperature is indirectly monitored and controlled underneath the susceptor using a Luxtron lightpipe temperature sensor. Through the upper optical port a vertical access to the substrate is given through a small hole in the liner ceiling and thus direct determination of the sample surface temperature is possible in connection with an emissivity corrected pyrometer and reflectometer (Laytec's EpiR-TT).

### Exhaust of Byproducts

The gaseous byproducts that form desorb from the sample surface and diffuse in the carrier gas away from the deposition zone towards the reactor exhaust. Very often CVD chemicals and the process byproducts require special precautions since they are toxic and/or environmentally hazardous. To this end toxic gas monitors and exhaust treating

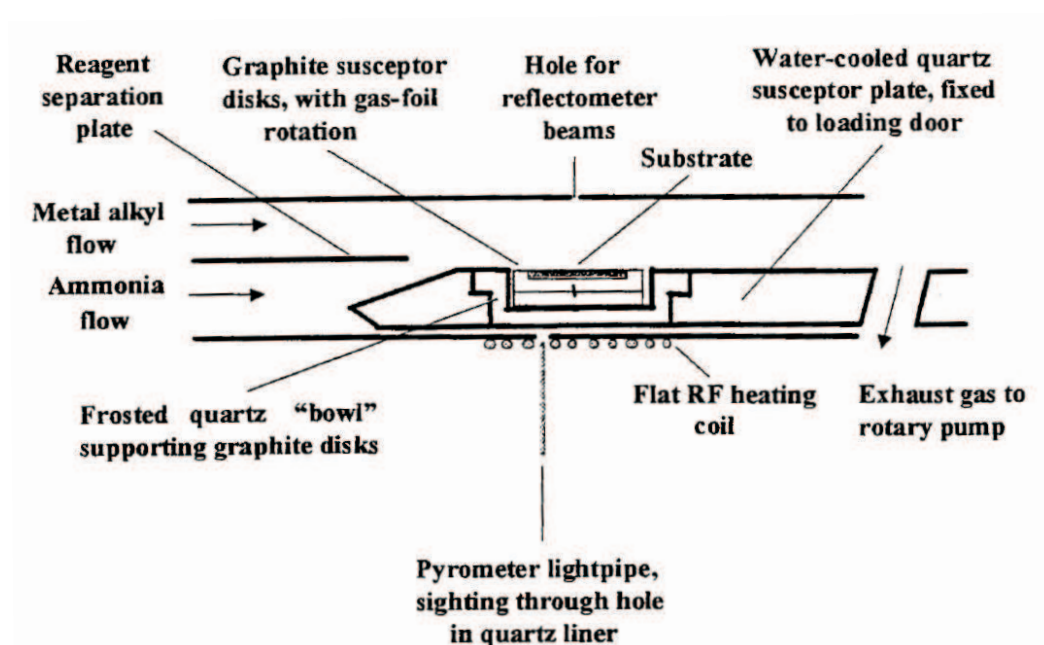


Figure 4.2: Schematic representation of the AIX 200/4 RF-S horizontal reactor employed for the MOVPE growth of group III nitrides. Note, that the inlet of group V and III is the conventional state on this sketch.

systems are used. The exhaust treating system will either deal with the gases by wet or dry chemical reaction. In the exhaust system (so called *scrubber*) used in this work, the neutralization of  $NH_3$  and the hydrolysis of the metal-organic sources is achieved with 30%  $H_2SO_4$  in water.

## 4.2 In-situ Monitoring

Unlike Molecular Beam Epitaxy (MBE) no electron-based techniques can be utilized for in-situ monitoring and growth control in gas phase environments like MOVPE. Due to the gas present, the free mean path of the electrons is reduced strongly. Therefore optical techniques are the methods of choice [Hab02a, Zet98, Zor95]. These methods include, among others, *spectroscopic ellipsometry* and *normalized reflectometry*. In this work normalized reflectometry has been used for in-situ monitoring. In general these optical measurement techniques are not surface sensitive probes, since the photons irrupt considerably into the layers beneath the surface (the penetration depth of visible light is in the micron range). In connection with a pyrometer, reflectometry allows the calculation of the emissivity corrected temperature of the sample surface, which is of particular interest, since it offers the possibility to accurately *determine and control* the temperature during all growth steps.

In the following section the measurement principle and the setup used for *in-situ normalized reflectometry* and *emissivity corrected pyrometry* will be described.

### 4.2.1 In-situ Normalized Reflectometry

The plain reflectance signal offers a convenient method of determining growth rates and ternary compositions as well an easy tool to monitor growth steps [Hab02b].

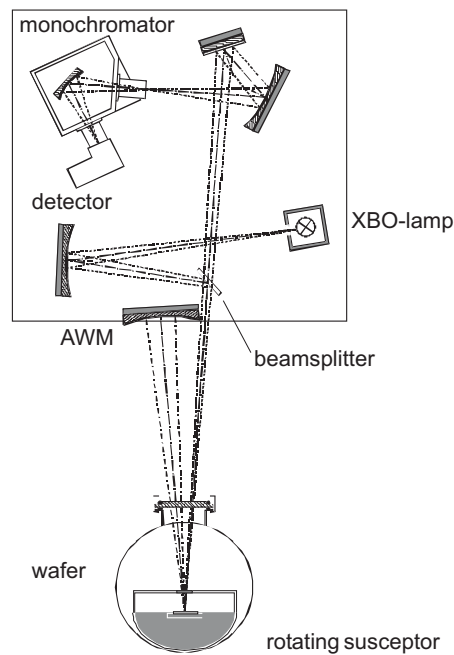


Figure 4.3: Schematic of the in-situ reflectometer as used for measurements on rotating samples to detect the reflection [Hab02].

Figure 4.3 is a schematical description of the arrangement. The reflectometer consists of a xenon lamp which is used as light source with a spectral range of 1.5 eV to 6.0 eV. Next to the source several filters are installed. Every couple of seconds the source is shaded and a dark signal is measured to determine the background signal. Additionally a band edge filter can be introduced in front of the xenon lamp in order to suppress light above 2 eV. Hence, diffraction maxima of higher order caused by low wavelengths are disabled. Then the light travels via a spherical mirror to a beamsplitter, where it is focused almost perpendicular to the sample surface onto the susceptor. From here it is reflected onto a toggle mirror that compensates the tipping of the susceptor. Finally the reflected intensity is detected. A more accurate description of this arrangement can be found in [Thi02].

Due to the lack of GaN substrates the growth of GaN can only take place on substrates of

a different material such as sapphire. Thus it is assumed that a three-phase-model (air/ layer/ substrate), describing the optical properties of the layer substrate configuration, can be employed to depict the reflections occurring in the layers during growth. As long as the penetration depth of the light is larger than the thickness of the layer and the layer can be treated as a homogeneous film, the optical properties of all three phases contribute to the reflectance.

The three-phase model can be reduced to a two-phase model<sup>3</sup> (only substrate and ambient) by defining an *effective dielectric function* describing the integral effect of the layer and the substrate therefore simplifying the reflection occurring during the growth process on multi layer structures. Using the FRESNEL equations the reflectance can be calculated [Bor80]. In the case of perpendicular incidence the reflectivity  $R$  equals to

$$R = |r|^2 = \frac{I_r}{I_i} = \frac{E_r^2}{E_i^2} = \left( \frac{n_1 - n_0}{n_1 + n_0} \right)^2, \quad (4.1)$$

where  $I_r$  and  $E_r$  is the reflected light intensity and reflected field component, respectively.  $I_i$  and  $E_i$  is the incident intensity and the incident field, respectively. Here  $n$  is the refractive index of the ambient medium.

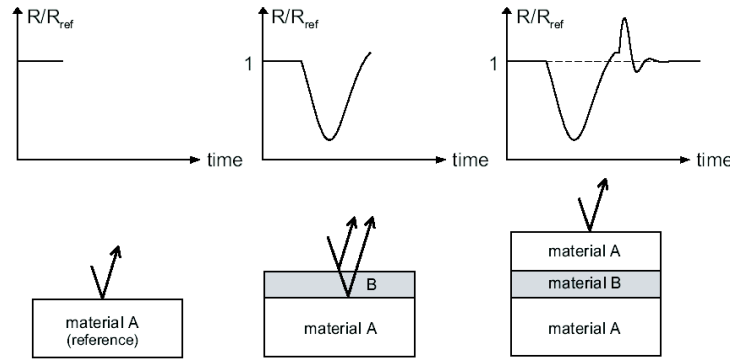


Figure 4.4: Schematic diagram illustrating the normalized reflectance. Layer B is normalized to the reflectance of layer A [Hab02].

Due to the fact that the actually measured signal is the detector voltage  $U$ , some way of calibrating this voltage to the sample's reflectivity  $R$  is needed. A *normalized reflectance*  $R/R_{ref}$  is used here for in-situ measurement, which is the reflectivity of the growing layer divided by the reflectivity of the substrate. This is illustrated in figure 4.4

If the reflectivity is continuously measured during growth a transient graph, as exhibited in figure 4.5, is acquired.

<sup>3</sup>This is also known as the effective media model [Bor80]

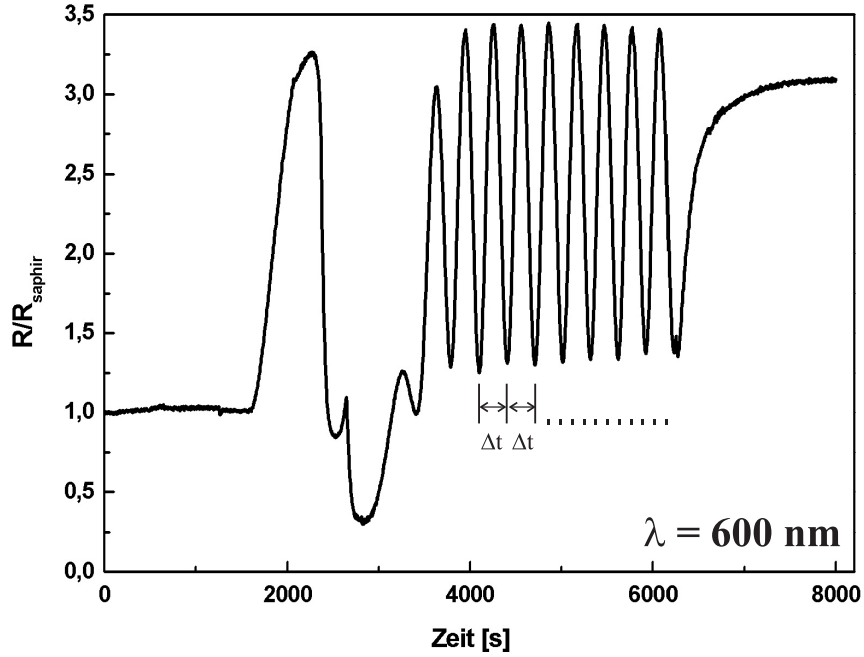


Figure 4.5: Typical reflectance transient measured at a wavelength of 600 nm.

By means of the transient growth rates and, where applicable, surface roughness can be determined. If  $\lambda$  is the wavelength of the used light and  $n$  the refractive index of the layer, then the maximum of the FABRY-PEROT oscillations due to interference, (following BRAGG's relation) of the reflected light can be found at thicknesses  $d$  such as

$$n \cdot d = (m) \cdot \frac{\lambda}{2} \quad (4.2)$$

with  $m > 1$  as integer. During growth layer thickness increases constantly so that a maximum of reflection is reached every time (4.2) is fulfilled. Thus the growth rate  $\mu_{gr}$  can be calculated by using the interval between two of these maxima

$$\mu_{gr} = \frac{\lambda}{2n\Delta t} \quad (4.3)$$

The reflectance signal offers not only the opportunity to measure such a crucial parameter as the growth rate, in addition information about the growth process itself can be obtained as will be discussed in chapter 6

### 4.2.2 Pyrometry

All substances at temperatures above absolute zero emit radiant energy as the result of the atomic and molecular agitation that is associated with the temperature of the substances. The rate of emission per unit area increases with increase in temperature. Accordingly, the temperature of a substance can be measured by means of detecting such radiant energy. This thermal radiation is characterized by a spectrum that depends on the temperature  $T$ . Pyrometry or radiation thermometry is the determination of temperature by analyzing this emission spectrum. Depending on the wavelength range studied, this procedure is called either optical or infrared pyrometry and is performed with an instrument called pyrometer. Because of its fast response this method is often used in thin film processing. In our experimental setup there are two pyrometers. One is integral part of the reflectometer setup and the other measures underneath the susceptor via a lightpipe sensor (compare section 4.1).

#### Principles of Pyrometry

The pyrometry measurement is based on detection of the incandescence from a surface according to the Planck equation and Kirchhoff's law [Pla00, Kir59]. The Planck spectral radiance ( $L$ ) emitted from a surface of a blackbody in thermal equilibrium at temperature  $T$  is

$$L_{blackbody}(\lambda, T) = \frac{2hc_0^2}{\lambda^5 \exp(hc_0/\lambda kT) - 1} \quad (4.4)$$

where  $\lambda$  is the emitted wavelength,  $h$  is Planck's constant,  $c_0$  is the speed of light in vacuum and  $k$  is the Boltzmann's constant. Figure 4.6 shows the spectral radiance of blackbody emission for several wavelengths. Hence, determining the surface temperature from a blackbody is a straightforward task.

However, real materials are seldom ideal blackbodies, i.e. bodies that absorb all incident radiation and at the same time are isotropically diffusive emitters. The optical pyrometer measures the spectral radiance of a real body  $L_\lambda$ , according to

$$L_\lambda(\lambda, T) = \varepsilon L_{bb} \quad (4.5)$$

where  $\varepsilon$  is called the spectral directional emissivity. It represents the ratio of the thermal radiation emitted from a real object to that from a blackbody. According to equation 4.5 the thermal emission is corrected by the emissivity factor.

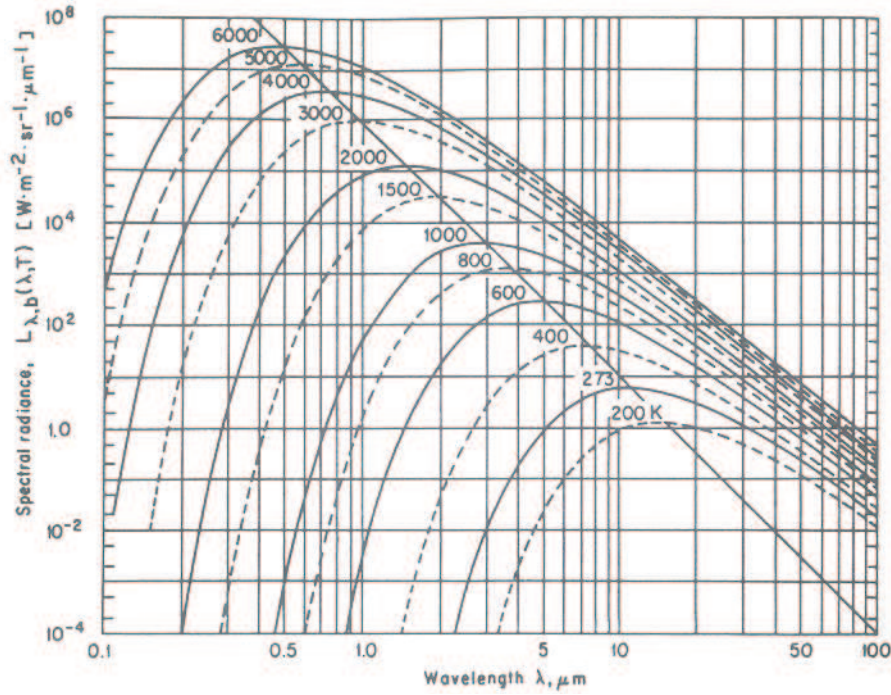


Figure 4.6: Spectral radiance of a blackbody from Planck's radiation law for several temperatures.

The emissivity  $\varepsilon$  depends on the reflectivity ( $\rho$ ) and transmittance ( $\tau$ ) of the material. Following the radiation balance

$$\rho + \alpha + \tau = 1 \quad (4.6)$$

and Kirchhoff's law stating that at thermal equilibrium, the emissivity of a body (or surface) equals its absorptivity  $\varepsilon = \alpha$ , we find

$$\varepsilon = 1 - \rho - \tau \quad (4.7)$$

For opaque media the transmittance becomes zero, therefore epsilon strongly depends on the reflectivity.

When pyrometric measurements are made at photon energies higher than the peak radiance the high-energy approximation of Planck's radiation law can be used. Using the Wien approximation [for  $\lambda T < \lambda_{max} T$ ] the inferred temperature from the surface of the object with the emissivity  $\varepsilon$  can be determined as

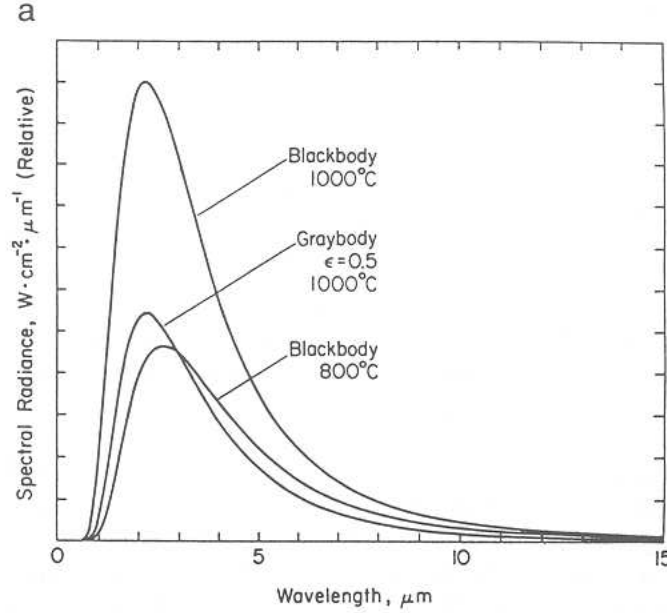


Figure 4.7: Spectral radiances for a blackbody and a graybody with emissivity of 0.5 at several temperatures, illustrating why accurate knowledge of the emissivity is essential.

$$T^{-1} = \frac{1}{T_\lambda} + \frac{\lambda}{c_2} \ln \varepsilon(\lambda, T) \quad (4.8)$$

From equation 4.8 it is obvious that successful pyrometry requires accurate knowledge of the spectral directional emissivity of the target. Errors of hundreds of degrees are possible in pyrometry if the emissivity is not accurately known, as seen in Figure 4.7. It shows that the flux near  $3\mu m$  is the same for a graybody<sup>4</sup> at  $1000^\circ C$  with  $\varepsilon = 0.5$  and a blackbody at  $800^\circ C$ .

Since the emissivity of the target depends upon such factors such as the incident light angle, material, temperature, surface roughness, doping level, dopant used, etc., it is not always accurate to assume that the emissivity is a known quantity. Even the roughness of the unpolished side of a wafer can alter the emissivity. Direct pyrometry is also complicated due to the fact that optical interference occurs since the index of refraction of the substrate differs from the deposited film. Thus, the emissivity *changes*.

<sup>4</sup>Graybodies are objects whose emissivity is equal at all wavelength

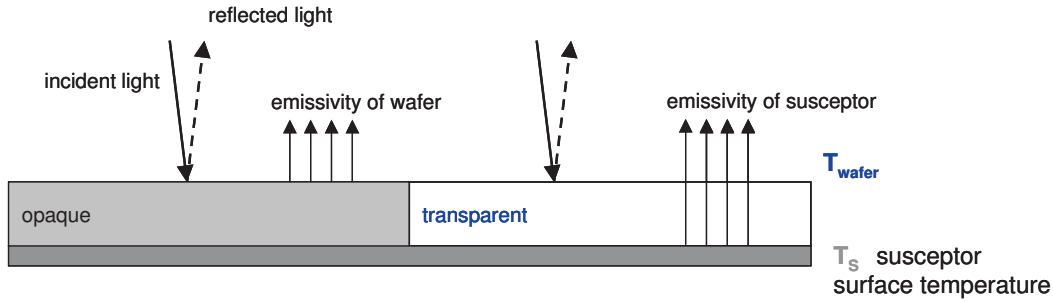


Figure 4.8: Pyrometry from opaque wafers such as Si or GaAs is a straightforward task as seen on the left half. The emissivity ( $\varepsilon$ ) of the substrate surface can be calculated from the measured reflectivity. On transparent substrates the pyrometer detects the emissivity of the underlying holder rather than that of the transparent substrate.

### Emissivity Corrected Pyrometry

These issues are overcome with *emissivity corrected pyrometry* (ECP). Simply stated, this in-situ measurement technique requires the combined functionality of a conventional pyrometer and a reflectometer. The reflectance of the substrate is measured at the same wavelength at which the pyrometer measures the thermal radiance. By using the equation (4.7)<sup>5</sup> the spectral directional emissivity ( $\varepsilon$ ) of the substrate surface can be calculated from the measured spectral directional reflectivity. Now, it is apparent why transparent substrates cannot be measured pyrometrically. At the employed detection wavelength (here: 950nm) the optical radiation from the susceptor passes through the transparent substrate and the pyrometer cannot distinguish between substrate radiation and susceptor radiation. The second problem is that the substrate emissivity at our detection wavelength is negligible yielding no usable blackbody radiance. Thus a calibration of the ECP is needed.

### Calibration Methods

Presently, the most effective methods to calibrate real-time emissivity corrected pyrometers are procedures based on melting points or phase transitions. To the latter one belongs the very convenient method based on the Si-Al-eutectic formation at  $T = 577^\circ\text{C}$ . This phase transition of a thin Al-layer on Si is accompanied by a sudden roughening of the surface, which leads to a dramatic loss in reflectance that can be seen with the naked eye, or more conveniently, can be detected by a reflectance sensor. Figure 4.9 shows the reflectance drop measured at the Ferdinand Braun Institute Berlin (FBH) performed with a LayTec EpiRAS-200 spectrometer that allows combined reflectance anisotropy spec-

<sup>5</sup>with negligible transmittance (with an opaque susceptor)

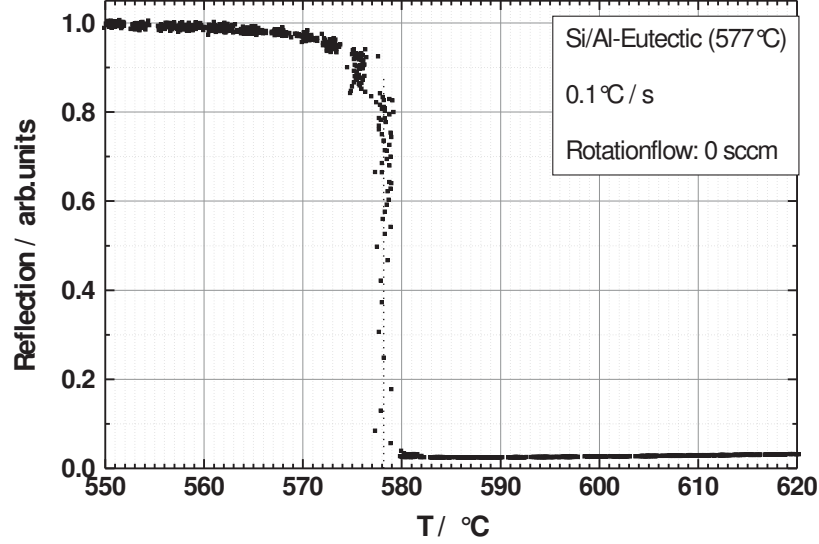


Figure 4.9: Calibration run based on the Si-Al-eutectic formation at  $T = 577^\circ\text{C}$  where a sudden roughening of the surface leads to a dramatic loss in reflectance. Experiment conducted at the Ferdinand Braun Institute Berlin in an AIX 200/4

troscopy (RAS) [Asp95] and reflectance (R) measurements between 826nm (1.5 eV) and 248nm (5.0 eV). However this method is rather time consuming and yields only one data point at  $577^\circ\text{C}$ . Also, the calibration accuracy could be influenced by Al-layer thickness.

## 4.3 Ex-situ Characterization Methods

### 4.3.1 Film Thickness and Homogeneity

As mentioned earlier optical methods such as reflectometry facilitate the actual layer thickness measurement. Using a Bio-Rad RPM2000 system, thickness mappings were carried out to obtain the thickness distribution of GaN samples. The system can scan wafers up to 1 mm thickness at a resolution of up to  $0.1\ \mu\text{m}$ .

Light from the sample is collected by a lens and fed to a spectrometer. The latter measures a definable wavelength range. These spectra exhibit typical interference effects because of multiple beam interference, where the spectral intensity is a function of the film thickness. With respect to the wavelength extrema are found. In principle the thickness  $d$  can now be calculated by use of equation (4.2). Therefore, if the film's optical constants are

known the thickness can be determined from the measured reflectivity. The basis of the measurement method is similar to the already discussed reflectometry.

Furthermore weakly absorbing films and multilayer films can also be evaluated by use of the two phase model as mentioned in subsection 4.2.1, therefore defining an effective dielectric function which describes the lower layers as an effective substrate.

### 4.3.2 Differential Interference Contrast Microscopy

Optical microscopy with all its derived types such as phase-contrast microscopy, differential interference-contrast microscopy or fluorescence microscopy is widely applied in physics, chemistry and biology. For our purpose, i.e. the layer morphology inspection after growth, the differential interference-contrast (DIC) microscopy<sup>6</sup> was employed. This technique is well suited topography evaluation of semiconductor layers and devices, as well as for locating defects on the surfaces.

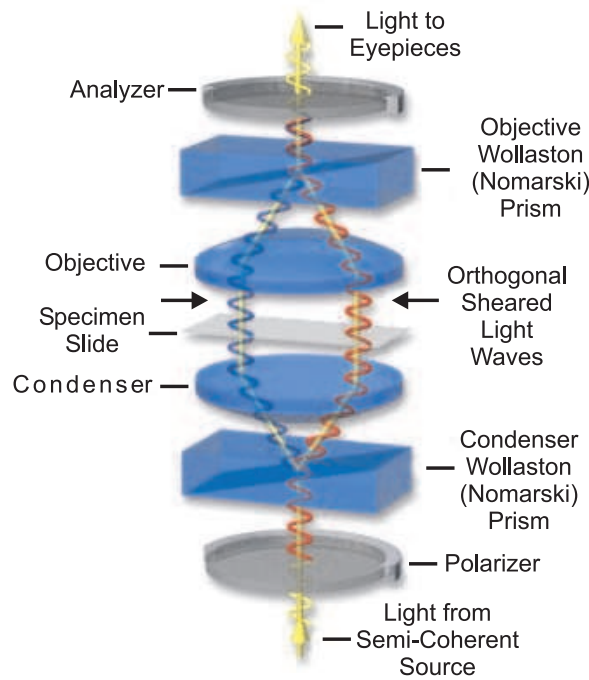


Figure 4.10: Schematic of Differential Interference Contrast.

**Brightfield illumination** is the normal, most even illumination mode for microscopy. A full cone of light is focused by an objective on the sample. The sample is uniformly

<sup>6</sup>sometime referred to as *Nomarski microscopy* after the French optics theoretician named Georges Nomarski.

illuminated. The picture observed is the result of differences in reflectivity created by material properties of the sample, e.g. transmission and reflection through surface films, and by the surface contour of the sample.

**Interference contrast** is a special type of brightfield illumination. Figure 4.10 shows the apparatus. Differences in height result in laterally different intensities and therefore in image contrast. Etch pits and cracks which are not detectable in brightfield will stand out clearly enough to be photographed in interference contrast. The Nomarski technique is especially good for observing the topography on semiconductors since it is capable of locating defects, such as pits, hillocks, cracks and other defects. This is very beneficial for group III nitride layers grown on sapphire substrates which are almost transparent.

### 4.3.3 Atomic Force Microscopy

Atomic force microscopy (AFM) provides three-dimensional surface topography at nanometer lateral and sub-angstrom vertical resolution on semiconductors and other materials. A sharp tip<sup>7</sup> on the end of a flexible cantilever scans a sample surface, while maintaining a constant force. The surface is scanned by a piezoelectric tube that moves the tip in a raster pattern with respect to the sample. The tip-sample interaction is monitored by reflecting a laser off the back of the cantilever into a split photodiode detector. By detecting the difference in the photodetector output voltages, changes in the cantilever deflection or oscillation amplitude are determined. A more detailed discussion is found in [Bin86, Ste03]

The two most commonly used modes of operation are *contact mode* and *tapping mode*. In this work the samples were investigated using the tapping mode. Here the cantilever is oscillating close to its resonance frequency (typically  $\sim 300$  kHz) and lightly tapping on the surface during the scan. The laser deflection method is used to detect the *root-mean-square* (rms) amplitude of the cantilever oscillation. A feedback loop maintains a constant oscillation amplitude by moving the scanner vertically at every  $x, y$  data point. Recording this movement forms the topographical image.

For investigation of atomically smooth surfaces, the AFM is very suitable, since the AFM possesses a very good vertical resolution. It can resolve monoatomic steps on the sample surface as well as calculate an rms roughness of the surface. Figure 4.11 displays parallel terrace like steps of epitaxially grown GaN.

Surface morphology investigations for the grown GaN and  $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$  layers were performed by atomic force microscopy (AFM) with a DI Nanoscope IIIa microscope operating in tapping mode. The rms roughness of the surface was calculated using the AFM software.

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<sup>7</sup>The tips typically have an end radius of 2 nm to 20 nm, depending on the tip type.

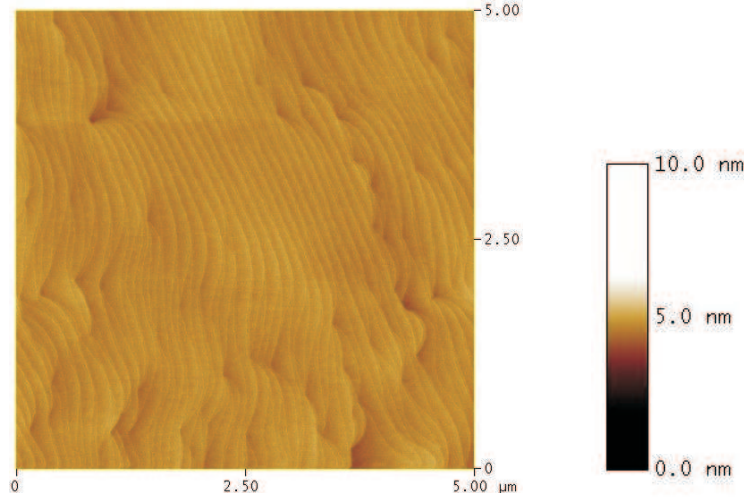


Figure 4.11: A smooth surface revealing terrace like steps of a GaN layer, as seen by AFM.

#### 4.3.4 Photoluminescence Spectroscopy

Photoluminescence Spectroscopy (PL) is a contactless, nondestructive method of probing the optical properties of materials. Photoluminescence is the emitted radiation from a sample as a consequence of light absorption<sup>8</sup>. The spectral analysis of this light allows statements on (in case of direct semiconductors) the band gap, crystal-quality, strain, impurities and defects. For more detailed information on PL on GaN refer to [Thi02].

It has to be kept in mind that due to the limited penetration depth of light only a certain depth of the specimen may be analyzed. The main recombination processes (as shown in figure 4.12) for a direct band gap semiconductor are:

- Band Band Transition
- Excitons
- Band Acceptor Transition
- Donor Acceptor Pair Transition

Strain measurements by PL are particularly easy for GaN. GaN layers grown on sapphire suffer from compressive strain. The band gap increases in case of biaxial in-plane compression compared to fully relaxed GaN. The donor bound exciton (DBX) energy therefore increases (from  $\sim 3.466$  eV) with increasing compressive strain in the layers.

<sup>8</sup>During a PL experiment usually a direct band gap semiconductor is excited with light of a higher energy than the bandgap,  $\hbar\omega_{Laser} > E_{Gap}$ , usually laser light.

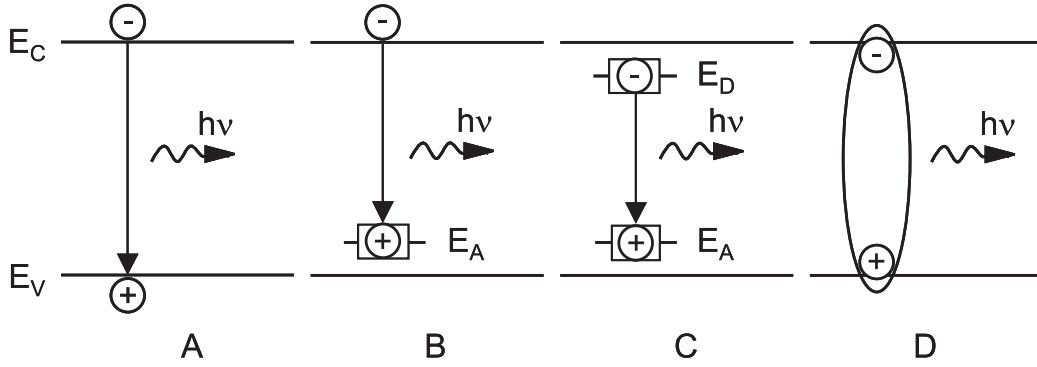


Figure 4.12: Recombination processes for a direct band gap semiconductor. (A) Band Band Transition ( $e, h$ ), (B) Band Acceptor Transition ( $e, A^0$ ), (C) Donor Acceptor Pair Transition ( $D^0, A^0$ ), (D) Excitons.

The spectra collected in the near band edge range (3.42 to 3.52 eV) were obtained using an excitation energy of 50  $\mu\text{W}$ , with a 1200 *lines/mm* grid.

### 4.3.5 X-ray diffraction

X-ray diffraction (XRD) is a widely-used technique in materials science, as a highly sensitive tool for detailed structural studies.

X-ray diffraction can be explained in simple terms by the "reflection" of an incident X-ray beam from a stack of parallel equidistant atomic planes. The electrons that surround the atoms, rather than the atomic nuclei themselves, are the entities which physically interact with the incoming X-ray photons. At each atomic plane a small portion of the beam is reflected. If those reflected beams do not cancel each other by interference, a diffracted beam can be observed. The condition where the reflected beams interfere positively (add up in phase) to give a strong diffracted beam is given by Bragg's law (compare equation (4.2)).

The Bragg law may be written as :

$$n\lambda = 2d_{hkl}\sin\theta_B \quad (4.9)$$

where  $n$  is an integral number describing the order of reflection,  $\lambda$  is the wavelength of the X-rays,  $d_{hkl}$  is the spacing between the lattice planes and  $\theta_B$  is the Bragg angle where a maximum in the diffracted intensity occurs. At other angles there is little or no diffracted intensity, because of negative interference. Since we know  $\lambda$  and we can measure  $\theta_B$ , we

can calculate the interplanar spacings. The characteristic set of  $d_{hkl}$  spacings generated in a typical X-ray scan provides a unique "fingerprint" of the material.

Using adequate incidence angles, various information about the crystal structure, composition or imperfections can be obtained. In this work X-ray diffraction has been used to determine the crystal quality of GaN layers and qualitatively compare dislocation densities (see Chapter 7). The structural characteristics of the GaN epilayers were evaluated by X-ray diffraction (XRD), using the symmetric (002) and asymmetric (102) reflection. The full width at half maximum (FWHM) of which are sensitive to all types of threading dislocations (TDs), such as screw, edge type and mixed TDs, and to pure edge and mixed types of TDs, respectively [Hey96].

#### 4.3.6 Rutherford Backscattering Spectroscopy

Rutherford Backscattering Spectroscopy (RBS) analysis is performed by bombarding a sample target with a mono-energetic beam of high energy particles, typically  $\text{He}^+$  ions, with an energy of a few  $\text{MeV}$ . A fraction of the incident atoms scatters backwards from heavier atoms in the near surface region of the target material, and is usually detected with a solid state detector that measures their energy. The energy of a backscattered particle is related to the depth and mass of the target atom, while the number of backscattered particles detected from any given element is proportional to its concentration. This relation is used to generate a quantitative depth profile of the upper 1 to 2  $\mu\text{m}$  of the sample. The primary applications of RBS are the quantitative depth profiling of thin film structures and their crystallinity

RBS was employed to analyze the obtained  $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$  layers. The spectra were acquired under random incidence (i.e. the angle of incidence does not coincide with a low index direction and channelling does not occur). In these conditions, many sub-surface atomic layers are hit and backscattering occurs from many deeper atoms. The backscattering spectrum exhibits a high intensity continuum of scattered energies up to a maximum defined by the surface peak due to scattering on the topmost layer [Iba99]. Figure 4.13 shows an RBS spectrum acquired for an  $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$  layer along a random direction in order to determine the layer thickness and composition.

The acquired spectrum can be simulated under the assumption of a stoichiometric ratio of group V (N) and group III (Al,Ga) elements, by inserting different relative concentrations of Ga and Al until the best fit is obtained (see Fig. 4.13). The  $\text{Al}_x\text{Ga}_{1-x}\text{N}$  layer composition can be determined with an accuracy of  $\pm 2$  atomic percent for Al. The relative precision of the Al level from sample to sample measured in the same RBS analysis is approximately  $\pm 1$  atomic percent.

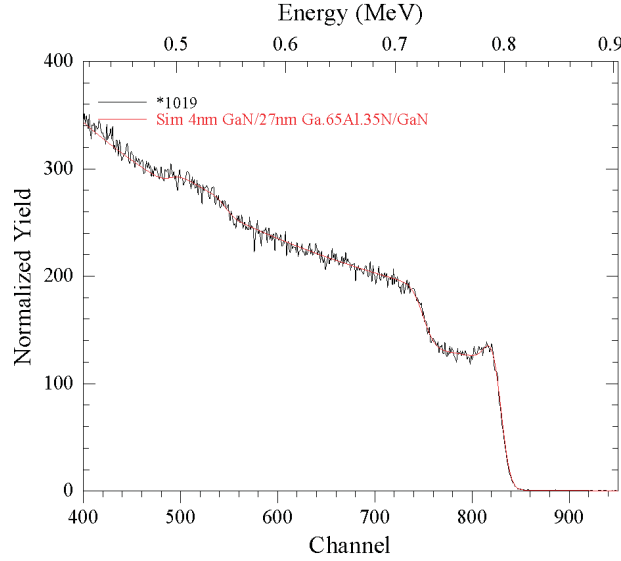


Figure 4.13: RBS spectrum of an  $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$  layer. The zigzag line is the RBS spectrum acquired in random geometry and the continuous line represents the best fit from which the  $\text{Al}_x\text{Ga}_{1-x}\text{N}$  layer composition (35 at% Al, 65 at% Ga) and the thickness (27 nm) were determined.

#### 4.3.7 Contactless Sheet Resistance Measurement

Resistivity is a primary quantity for characterization of semiconductor material. An eddy-current instrument directly measures nondestructively the conductance of a specimen. Values of sheet resistance and resistivity are calculated from the measured conductance, with the resistivity values also requiring a measurement of specimen thickness. The effective sheet resistance of a bulk substrate is its bulk resistivity  $\rho$  (in  $\Omega\text{cm}$ ) divided by its thickness  $d$  in  $\text{cm}$ .

$$R_s = \frac{\rho}{d} = \frac{RA}{ld} \quad (4.10)$$

$R$  is the electrical resistance of a uniform specimen of the material (measured in ohms),  $l$  is the length of the specimen (measured in meters) and  $A$  is the cross-sectional area of the specimen (measured in square meters). LEI Eddy Current systems measure the conductivity of a sample by producing an RF magnetic field perpendicular to and through the sample. The measurement principle is based on the absorption and dissipation of the magnetic field in the sample. The change in energy is measured and thus conductivity calculated. Since the magnetic energy passes through the sample, bulk wafers and multi-layered wafers can be measured. The bulk specimens may be single crystal or polycrystalline and of either conductivity type ( $p$  or  $n$ ) in the form of slice wafers (round

or other shape) that are free of diffusions or other conducting layers that are fabricated thereon, that are free of cracks, voids or other structural discontinuities. The substrate on which the thin film is fabricated should have an effective sheet resistance at least 1000 times that of the thin film. LEI systems can measure sheet resistance from below  $0.1 \Omega/\square$  to above  $3000 \Omega/\square$ . When measured within the proper resistance ranges LEI systems measure these sheet resistances to within  $\pm 3\%$ .

### 4.3.8 Hall Effect Measurement

Hall effect measurements are commonly used to determine electrical properties of solids such as concentration and mobility of charge carriers [Hal79]. The Hall effect is caused by the deflection of moving charged particles (i.e. electrons) by a magnetic field (Lorentz force). The carriers are accumulated at the edges of the samples resulting in an electric field which compensates the Lorentz force. By measuring the voltage associated with this the carrier concentration can be obtained. For a bar shaped sample with the thickness  $d$  the carrier concentration is given by the following expression,

$$n = -\frac{BI}{edV_H} \quad (4.11)$$

where  $I$  is the current through the sample,  $B$  is the magnetic field,  $e$  is the electronic charge and  $V_H$  is the measured Hall voltage. When measuring GaN samples, it is worth mentioning that the measured carrier concentration is an effective one (donors minus acceptors), which does not take compensation effects (traps and such) into account. The sign of the Hall voltage, positive or negative, gives the p- or n-type semiconductor.

The Hall mobility (depending on electron transport as discussed in chapter 2) is obtained, when additionally the resistance  $R$  of the sample is measured. Then  $\mu$  is given by

$$\mu = \frac{1}{en} \cdot \frac{l}{wd} \cdot \frac{1}{R}, \quad (4.12)$$

where  $e$  is the electronic charge,  $n$  is the charge density,  $l$  the length,  $w$  the width and  $d$  the thickness of the sample.

According to (4.12) for a bar shaped sample the actual dimensions ( $d$ ,  $w$  and length  $l$ ) of the sample are necessary to determine all parameters. For everyday characterization purposes usually arbitrary shaped samples are used. There the so called van der Pauw method is applied for the Hall measurements [Pau58]. With this method the mobility and carrier concentration of arbitrary shaped samples can be determined as long as there are no holes, nonconducting islands or inclusions (i.e. the sample is simply connected), the ohmic contacts are placed on the periphery (preferably in the corners of the plate)

and the contacts are sufficiently small. For detailed discussions of this method please see [Lant00, Ste03].

Hall effect measurements were done at 300 K and 77 K using the van der Pauw configuration, to determine the resistivity of the layers as well as the electron mobility and the carrier concentration, using In balls for ohmic contacts. The In balls were annealed at 400°C for 3 - 4 minutes in order to achieve ohmic contacts. AlGa<sub>N</sub>/Ga<sub>N</sub> heterojunctions were contacted using a procedure in an RTP oven [Ber05].

## Chapter 5

# Determination of Surface Temperature on Transparent Substrates

Accurate monitoring of growth temperatures is essential during GaN MOVPE growth. The layer qualities, growth rates, abruptness of heterointerfaces and ternary alloy compositions are all dependent on growth temperature. Hence, in-situ measurement and control of the substrate temperature in nitride MOVPE is crucial for obtaining reproducible layer quality. This chapter deals with temperature measurement on transparent substrates and the difficulties associated with commonly applied calibration methods. A new method for temperature calibration by using the in-situ measured band-gap shift of SiC in conjunction with real-time emissivity corrected pyrometry for transparent substrates is demonstrated. Eventually limitations of this new method are addressed.

### 5.1 Standard Measurement Techniques vs. Emissivity Corrected Pyrometry

For temperature determination thermocouples and pyrometers are the most widely used tools for measuring and controlling the process temperature, likewise in our system, which is equipped with a Luxtron light pipe sensor (compare chapter 4). Thermocouples are often inaccurate because they make no thermal contact with the sample. Due to susceptor and/or wafer rotation the thermocouple is far away from the growing layer. Therefore, the true wafer temperature has an offset to the process control temperature. This offset is not constant since changes in the susceptor setup or increasing reactor wall coatings and even different rotation gas flows alter the actual wafer surface temperature [Har04a] and hence, this offset *cannot* be compensated by calibration. Fig 5.1 demonstrates this effect.

As the process temperature is constant the real surface temperature changes drastically as a function of the rotation gas flow. All these indicated issues also hold for light-pipe sensors, which pyrometrically sense the temperature indirectly from underneath the susceptor setup.

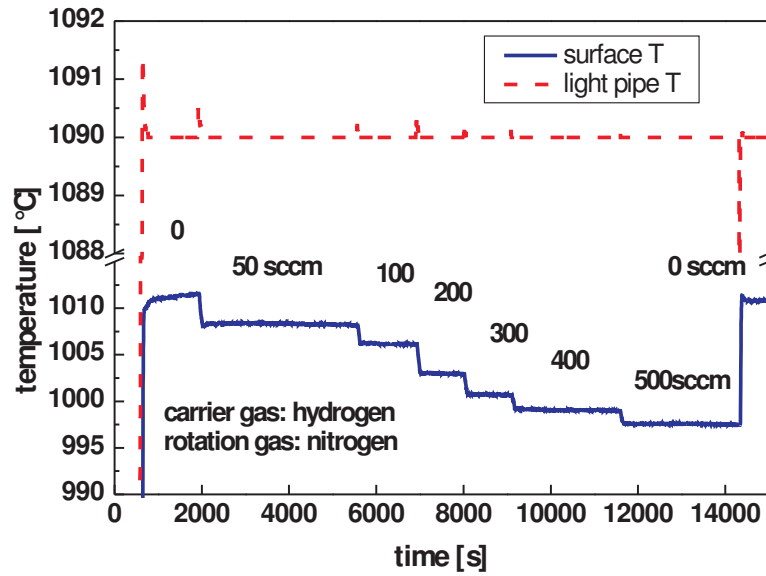


Figure 5.1: Temperature offset between wafer surface and process control temperature as determined by ECP and a light pipe pyrometer respectively for increasing rotation flows.

Real-time emissivity corrected direct pyrometry (ECP) [Bob94] of the susceptor and the wafer surface overcomes these problems. However, since optically transparent substrates such as sapphire or SiC are often employed for GaN MOVPE growth, the measured surface temperature represents the susceptor surface temperature and has to be corrected for the temperature difference between wafer and susceptor. Presently, calibration of real-time emissivity corrected pyrometers is achieved by procedures based on melting points or phase transitions [Hab02b] as demonstrated in chapter 4. It will be shown that this approach is not always applicable.

## 5.2 Conventional Calibration Methods

Presently, the most effective methods to calibrate real-time emissivity corrected pyrometers are procedures based on melting points or phase transitions as presented in chapter 4.

However this method is rather time consuming and yields only one data point at the respective melting point or phase transition of the employed material. Also, the calibration accuracy could be influenced by the layer thickness of the employed material.

In order to calibrate the pyrometer for the temperature difference between wafer and susceptor,  $\Delta T_{wafer,susceptor}$ , experiments with different phase transitions and melting points, as mentioned above, were used. For the calibration based on the eutectic- or melting point in our RF heated system, 150nm thin layers of Ag and Au on both sapphire and Si substrates were used in order to achieve data points in different temperature regimes close to GaN epitaxy conditions. The melting point for Ag and Au on sapphire wafers is 962°C and 1063°C, respectively. The phase transition of Ag and Au on Si occurs at 830°C and 370°C, respectively [Had90]. These samples have been carefully and stepwise heated up to their respective melting or phase transition points.

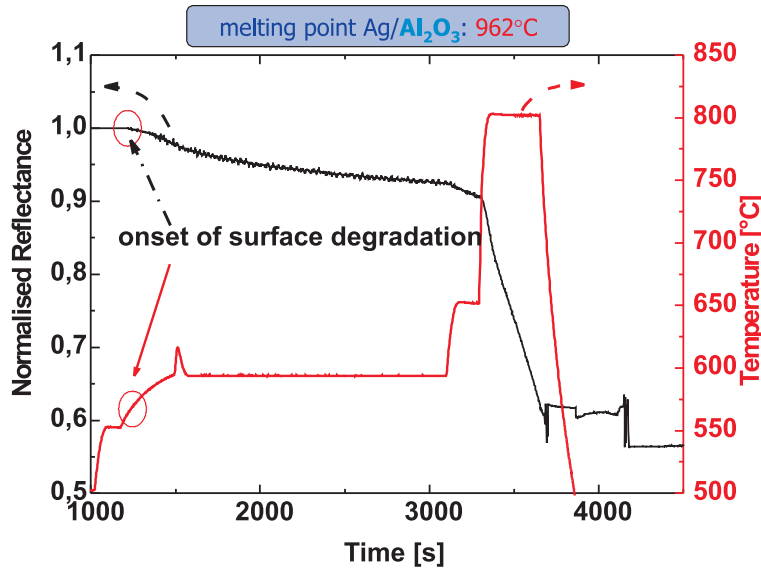


Figure 5.2: Reflectance and temperature transient of calibration run based on melting point determination of a thin Ag layer on sapphire. The unexpected and untimely slow degradation of the Ag-layer at a temperature of 570°C is highlighted.

Fig. 5.2 displays the reflectance transient for the melting-point experiment of Ag on sapphire as well as the respective *lightpipe* (or process control temperature), which yields a higher temperature than the actual surface bears. The sudden degradation of the Ag-layer surface is expected at 962°C. However, surface roughening occurs already at lower lightpipe temperatures of about 570°C. Keeping temperature constant at 590°C the reflectance slowly drops. With increasing temperature this effect becomes more evident and at around 730°C an abrupt change in the reflectance is observed. Apparently there is a huge difference between the nominal melting point and the measured actual value.

While many reactors are heated via irradiation of the susceptor with IR lamps our horizontal reactor system is inductively heated via a RF-coil situated underneath the susceptor setup. Thus, the untimely slow degradation of the surface is attributed to inductive heating of the metallic film due to the RF-heating of the susceptor material. Hence, methods based on melting points and phase transitions for absolute temperature calibration are not applicable for our RF-heated system. Another way for absolute temperature calibration has to be found.

### 5.3 New Calibration Approach with SiC Band Temperature Dependence

The band gap of many IV and III–V materials varies strongly with temperature. Therefore, spectral reflectometry holds possibilities for utilizing this shift to reference the substrate temperature during MOVPE growth [Tim92, Bal03, Joh93]. To be more precise the temperature dependent absorption edge is measured which is directly related to the bandgap energy.

The energy bandgap of semiconductors tends to decrease as the temperature is increased. This behavior can be better understood if one considers that the interatomic spacing increases with the thermal vibrations in the lattice. An increased interatomic spacing decreases the average potential seen by the electrons in the material, which in turn reduces the size of the energy bandgap. The temperature dependence of the energy bandgap has been experimentally determined [Var67] yielding the following empirical expression for the bandgap  $E_g$  as a function of the temperature  $T$

$$E_g(T) = E_g(0) - \frac{\alpha T^2}{T + \beta} \quad (5.1)$$

where  $E_g(0)$ ,  $\alpha$  and  $\beta$  are material dependent fitting parameters. While both empirical and theoretical equations exist for band gap dependence on temperature for most materials of interest, in practise these equations do not consider doping effects, scattering or more simply the instrument response. As a result it is much more accurate to generate experimental calibration curves.

In this thesis a new method for temperature calibration is demonstrated by using the in-situ measured temperature-dependent band-gap shift of SiC in conjunction with real-time emissivity corrected pyrometry. SiC was chosen since its a semiconductor and its bandgap is in the detection range of our reflectometer (unlike sapphire). It is transparent for visible light, just as sapphire wafers used throughout this work. Moreover SiC is the substrate of choice for many applications in opto- and power electronics for III-nitrides.

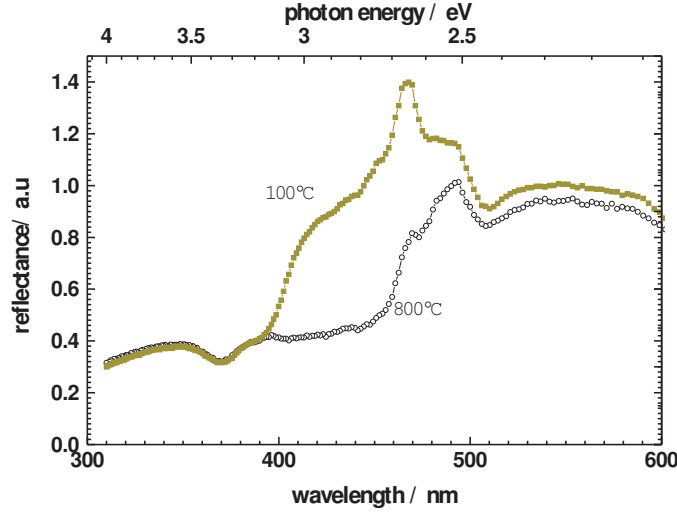


Figure 5.3: Reflection raw signal from SiC (double side polished, undoped) for two temperatures.

The temperature-dependent SiC band gap shift measurement were carried out using two inch, double side polished, 450  $\mu\text{m}$  thick, 6H-SiC substrate, supplied by NovaSiC. Double side polished SiC substrates were used to enhance the detected reflectance signal level, since less light is scattered by the rough wafer backside.

The band-gap<sup>1</sup> can be determined from the radiation reflected from the susceptor and transmitted through the substrate. The optical measurements at Forschungszentrum Juelich (FZJ) were performed at 950nm with an EpiR-M-TT optical in-situ sensor having two separate measurement channels for emissivity corrected pyrometry and high accuracy reflectance measurements at a wavelength range from 826 (1.5 eV) to 248 nm (5.0 eV). These were compared to measurements performed at the Ferdinand Braun Institute Berlin (FBH) with a LayTec EpiRAS-200 spectrometer that allows combined reflectance anisotropy spectroscopy (RAS) and reflectance (R) measurements in the same wavelength range from 1.5 to 5 eV.

In this set of experiments reflectance spectra with a resolution of 0.01eV were recorded for a range of true temperatures at FBH and nominal light pipe temperatures at FZJ. The first set of experiments is conducted at FBH. The IR heating system was calibrated by means of phase transition measurements with a non-rotating susceptor in order to exclude cooling effects due to rotation gas flow. The measurement error due to the phase transition calibration is assumed to be less than  $\pm 2K$ . Thus, the detected temperature

<sup>1</sup>To be more precise the temperature dependent absorption edge is measured which is directly related to the bandgap energy.

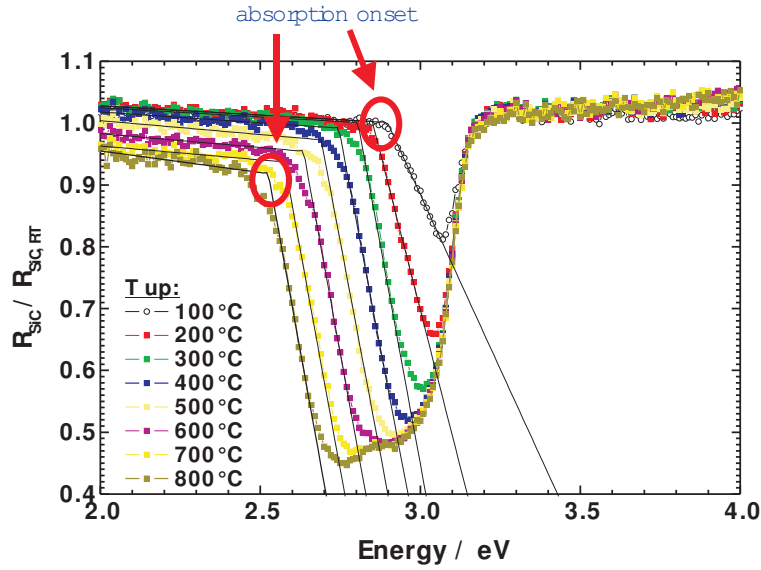


Figure 5.4: Normalized reflectance spectra referenced to the room temperature spectrum of an undoped double side polished SiC wafer for a range of absolute temperatures. The straight lines represent the fit function used to determine the onset of the absorption.

is very close to the *absolute* temperature.

Looking at the reflectance spectra, the "band gap" becomes clearly visible as a step in the reflection signal. Figure 5.3 displays the reflection raw signal from SiC (double side polished, undoped) for two temperatures. Superposed is the spectrum of the white light source yielding the maximum intensity around 2.65 eV. However, the onset of absorption ("band gap") is found at around 400 nm ( $\sim 3.2$  eV) and 470 nm ( $\sim 2.7$  eV) for wafer temperatures of 100°C and 800°C respectively.

Fig. 5.4 displays the inferred reflectance spectra from 2.0 to 4.0 eV for an unintentionally doped SiC wafer for a range of temperatures. The spectra are normalized and have been referenced to the room temperature spectrum. Thus, the difference in reflectance due to the different transmission/ absorption is emphasized and the superposed white-light source spectrum filtered. These spectra display a sharp falling edge which corresponds to the onset of absorption in the layer. This onset is correlated to the band gap. With increasing substrate temperature the position of the falling edge moves to lower energies as the band gap decreases. It should be noted that even at high temperatures the absorption edge is still in the visible spectrum range. Using a suitable fit function (straight lines in Fig.5.4) the position of the absorption onset versus the absolute temperature was determined as seen in Fig.5.5. This fit is basically a *fermi function* with material dependent characteristic reflection values (instrument response) that gives the "bandgap" energy.

Now, a gauging normal is established since the correlation of the band gap shift (or more

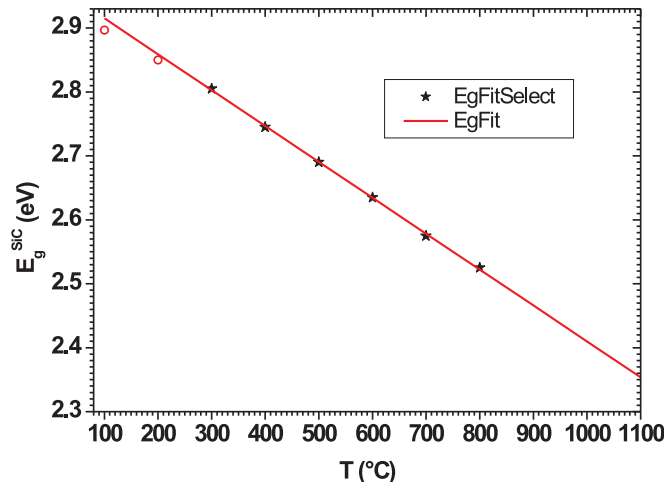


Figure 5.5: The resulting correlation between absorption onset and absolute temperature as found at the reference reactor (Ferdinand-Braun-Institut).

correctly “absorption onset”) with the absolute temperature is determined. Using this reference the reactor at FZJ can be calibrated. For that purpose the SiC measurements were repeated in the FZJ reactor in a similar way and the absorption onset was determined. Fig. 5.6 shows the comparison of the two resulting band gap plots versus the temperature. The resulting process temperature correction for the reactor at FZJ is displayed. The true surface temperature of the SiC wafer at a process control temperature  $T_1 = 620^\circ\text{C}$  is at  $550^\circ\text{C}$  and for  $T_1 = 1050^\circ\text{C}$  it is  $950^\circ\text{C}$ . Assuming that sapphire wafers of equal thickness qualitatively show the same thermal behavior our calibration method can also be employed to determine the surface temperature of sapphire wafers during growth. Once calibrated in this manner our reactor measures the correct wafer temperature for all growth runs on SiC and sapphire, provided that the wafer has full contact to the susceptor. Now this calibration method can be applied each time the reactor geometry or sensor position is changed.

## 5.4 Limitations

It must be pointed out that great care must be taken when doped substrates are used for calibration. High doping densities cause the bandgap to shrink. This effect is explained by the fact that the wavefunctions of the electrons bound to the impurity atoms start to overlap as the density of the impurities increases. For instance at a doping density of

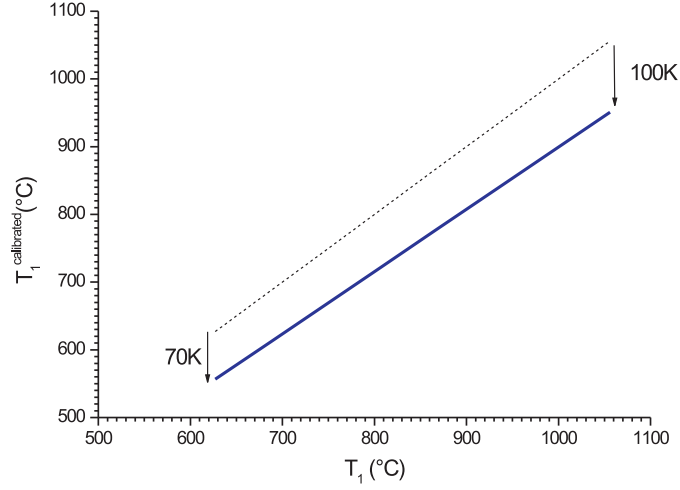


Figure 5.6: Referencing our temperature, empty circles, to the absolute temperature, full circles. The true surface temperature of the SiC wafer at a process control temperature  $T_1=620$  °C is at 550 °C and for  $T_1=1050$  °C it is 950 °C.

$10^{18} \text{cm}^{-3}$ , the average distance between two impurities is only  $10 \text{nm}$ . This overlap forces the energies to form an energy band rather than a discrete level. This fact is easily proven. Transmission measurements of non intentionally ( $< 5 \times 10^{17}$ ) and doped SiC ( $> 1 \times 10^{18}$ ) show significant changes of the absorption behavior of the substrate. Fig. 5.7 demonstrates the normalized reflectance spectra of an undoped SiC wafer versus two other substrates of higher doping concentration. The band edge is strongly broadened by the dopants and/or the free carriers make the material opaque.

Moreover the ECP system as it is described here cannot account for substrate temperature differences due to wafer bowing effects, that might occur especially for thin substrates. In our case, where non lattice matched materials (as GaN on sapphire) are grown, a strong concave wafer bowing might occur. The inhomogeneous contact or distance between wafer and susceptor leads to considerable differences in the wafer surface temperature. Combined curvature and ECP measurements give access to this effect [Kro05].

In addition, in this setup pyrometry depends on the reliable measurement of the wafer reflection and therefore effects such as wafer wobbling or rough surfaces have to be eliminated, since they reduce the accuracy of the measurement.

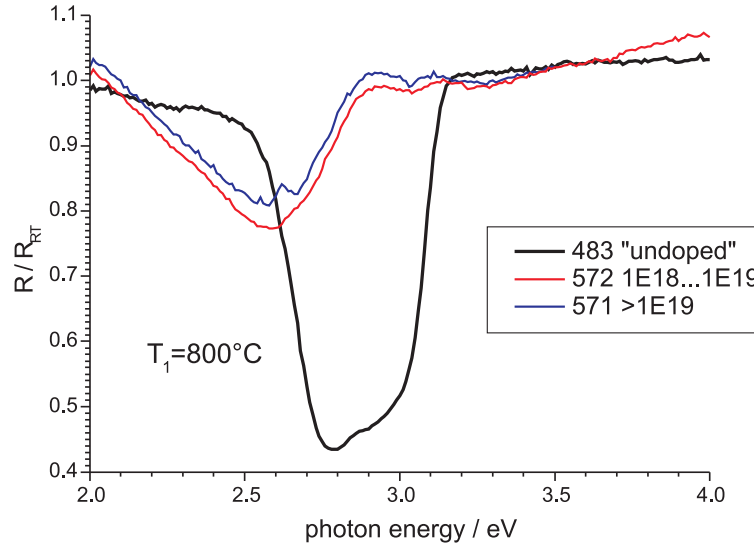


Figure 5.7: Doping dependence of the energy bandgap of SiC

## 5.5 Conclusion

It is shown that direct temperature measurement is a must though it is challenging for transparent substrates. Commonly applied calibration methods for absolute temperature determination of the wafer surface *can* be erroneous when highly conductive metallic layers are heated by induction as in our RF-heated MOVPE systems. Therefore, a new method for temperature calibration for transparent substrates has been demonstrated by using the in-situ measured, temperature dependent and band-gap related reflectance shift of SiC, in conjunction with real-time emissivity corrected pyrometry. The direct transfer of process temperature to any reactor becomes possible.

Moreover and more importantly for the following discussion, this technique allows to adjust the MOVPE system to identical run-by-run wafer temperatures. A reliable basis for absolute temperature determination is established. In-situ measurement and control of the substrate temperature is facilitated which is crucial for reproducible layer quality.



# Chapter 6

## New Growth Approach for GaN

This chapter deals with the development and optimization of a new MOVPE process approach on sapphire substrates with respect to AlGaN/GaN HEMT application.

Following this goal, the chapter is organized as follows: Beginning with a presentation of the applied MOVPE growth process on sapphire substrates the morphological development of GaN growth is discussed. Moreover, the reproducibility of the AIX 200/4 RF-S system is addressed. The source of irreproducibility will be investigated using the new approach for temperature determination (chapter 5). Counteractive measures are examined and implemented: A new deposition approach is introduced that takes care to avoid parasitic depositions on the reactor walls. Numerical simulations and growth modelling will be presented to prove the principal feasibility of the counteractive measures and to establish a broad comprehension of the epitaxial process. At last the commonly applied growth process is compared to the new one.

### 6.1 Morphological Development of MOVPE GaN

In the following section the standard MOVPE GaN growth process and the morphological development is discussed. The morphological development during GaN deposition will be monitored by in situ normalized reflectometry. This in situ method will be applied to optimize MOVPE of GaN on sapphire.

The high dissociation pressures and melting temperatures are the main obstacles in obtaining large group III nitride single crystals, which can serve as substrates for homoepitaxial growth. Therefore they are not yet commercially available and other substrates have been employed with considerable mismatch to GaN regarding lattice constants, thermal expansion and so forth. However, sapphire is the most extensively used. In general the quality of films grown *directly* (i.e. without any transition layer) on any plane of sapphire is poor.

To name just a few effects the degradation can be evidenced by XRD measurements (high FWHM values for symmetric and asymmetric rocking curves), morphology investigation (surface contains hillocks), and photoluminescence spectra (considerable yellow emission). In 1983 Yoshida et al. [Yos83] started the deposition of AlN buffer layers prior to the growth of GaN. Amano et al. [Ama86] adopted this process to MOVPE growth. They introduced a thin AlN layer, grown on sapphire at lower temperature than the subsequent GaN layer, in a so-called two-step growth technique. Nakamura [Nak91] suggested 1991 that the quality of GaN layers improves if low temperature GaN nucleation layers are used. Hence, with a proper surface preparation and an optimized accommodation between substrate and main layer, good quality GaN films can be obtained.

The complexity of the GaN growth process requires a precise and fast control. The in-situ reflectometry method employed during growth is capable of providing information for real time identification of the growth stages which can be helpful to optimize the growth process. The evaluation of the information extracted from the reflectometry transients allows a faster optimization of the growth parameters which influence the properties of the layer. Furthermore, by combining the in-situ with ex-situ characterization methods (such as AFM, HALL or XRD), a better understanding of the GaN growth mechanism on sapphire can be obtained. In order to identify and analyze the GaN growth stages for our process, the in-situ reflectometry method was employed during a standard GaN growth run. A complete reflectance transient for GaN growth in Fig. 6.1 (black line) is presented together with the AFM pictures which show the morphology at different growth stages. Also, the temperature profile recorded during the entire growth process is presented (grey line).

In our case, GaN growth was performed in an AIXTRON 200/4 RF-S single wafer reactor using TMGa and  $\text{NH}_3$  as precursors and  $\text{H}_2$  or  $\text{N}_2$  as carrier gas. Although the GaN growth MOVPE process is originally called a two-step process, the entire growth process in fact is a succession of more than the two mentioned steps:

### **Substrate pretreatment:**

The sapphire substrate is heated up to above  $1000^\circ\text{C}$  where *desorption* takes place. This is performed under hydrogen ambient in order to free the substrate of absorbed gaseous molecules and moreover to chemically clean the substrates by the reaction of carbon to alkanes and oxygen to water. Desorption, as the most common preparation procedure prior to MOVPE growth is reported to anneal the wafer surface and thus to reduce the roughness of the substrate which can present damages from the applied mechanical polishing procedure [Cui00].

The exemplary growth transient in figure 6.1 reveals no change in reflection due to the substrate pretreatment.

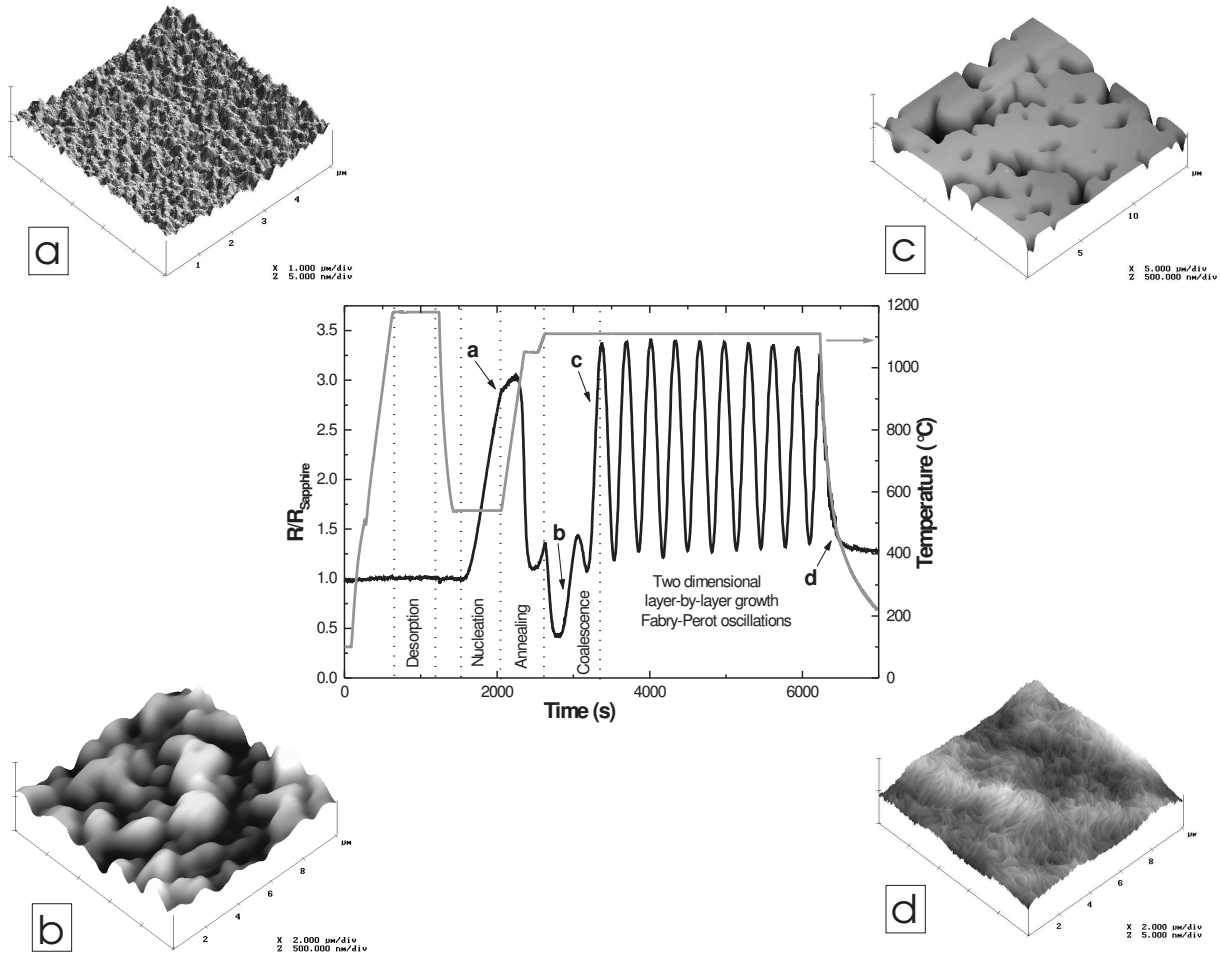


Figure 6.1: Typical reflectance transient (black line) and temperature (grey line) recorded during a standard growth run of GaN on sapphire. AFM insets show the morphology at different stages of growth: Nucleation layer before (a) and after recrystallization (b), during the evolution of island coalescence (c) and the final surface with atomic steps after coalescence (d).

### Nucleation layer growth:

Following the desorption step the reactor is ramped to a pressure of typically 200 *mbar* at a temperature of around 500°C. At this temperature a thin nucleation layer of about 20 to 30 *nm* is grown. The nucleation layer (which acts as buffer or accommodation layer between the main GaN film and the substrate) provides the high density of nucleation centers and, in growth steps following the nucleation, it promotes the *lateral* growth of the main layer. As a result of the low growth temperature the crystal quality is very poor. The nucleation layer consists of both small crystallites embedded in amorphous material. The

thickness of the nucleation represents the most important parameter for the subsequently grown main epilayer, in terms of surface morphology, electrical and structural properties [Hir97]. The layer thickness determines the crystallite size after annealing.

Figure 6.1 displays the detected in-situ reflectometry data during growth. Apparently the reflectance increases, due to the higher refractive index of GaN ( $n_{\text{GaN}} \approx 2.45$ ) in comparison to sapphire ( $n_{\text{saph}} \approx 1.75$ , compare equation (4.1)). A film covering the substrate is formed and therefore a first FABRY-PEROT oscillation is recorded. AFM studies show (Inset (a) in figure 6.1) a very smooth surface, containing very small grains.

### Annealing:

Right after the nucleation layer growth the temperature is intentionally slowly ramped up to growth conditions. As the temperature increases the surface is stabilized with  $NH_3$ . This annealing or recrystallization process turns the amorphous layer into a wurtzitic film with preferential orientation (i.e [0001] for c-sapphire) having an improved crystallinity. Also some of the material decomposes and evaporates at high temperatures, thus etching the material. During this step the GaN nuclei further develop to islands. The first islands form from the desorbed (gas-phase) Ga atoms generated during the NL decomposition (evidence for this mechanism is presented in [Kol04]). The most important parameter here is the annealing time. An extended annealing time will promote the reevaporation of GaN and etch the layer, decreasing the density of islands until only the bare sapphire is left.

In figure 6.1 the decrease in reflectivity is caused both by film etching (reversed growth) and scattering effects which are due to film roughening as islands appear.

The coalescence of the annealed layer is completed in the subsequent layer growth.

### GaN growth, Part I: 3D growth, "Coalescence"

The final step, the GaN epilayer growth, also sometimes referred to as the high temperature (HT) growth, takes place at elevated temperatures of usually above  $1000^\circ C$ . This step begins with the coalescence of the annealed layer's islands. Because of the large difference in the lattice constants of the grown layer and the substrate, the free energy of the system is lowered by three dimensional growth. The GaN layer starts to grow simultaneously both laterally and vertically in a three-dimensional growth mode, adjacent islands flow together to form larger complexes. Where islands merge dislocations form. Therefore the fewer the boundaries, the lower the dislocation density in the bulk GaN layer. However, due to the lattice mismatch a high density of threading dislocations ( $\sim 10^{10} \text{ cm}^{-2}$ ) propagates directly through the as-grown layer. At the end of coalescence, the growth mode is rapidly changed to a two-dimensional growth mode which allows a

smooth GaN epilayer. In this transition step the most important parameters are the growth temperature, growth rate, reactor pressure and the V/III ratio since they strongly influence the speed of coalescence which in turn determines the structural characteristics and therefore the electrical and optical properties of the film.

The transition to a 2D growth mode is illustrated by the AFM inset (c) in figure 6.1. In the beginning a three-dimensional (3D) growth mode is favored, hence the average roughness of the epilayer increases as long as the grain size stays below the nucleation site spacing, which becomes apparent in a further decrease of the reflectance signal. At this point, the surface appears dim and shows no specular reflectivity.

### GaN growth, Part II: 2D growth

This step begins with the completion of the previously started coalescence. Now that the transition from 3D to 2D growth occurred the surface of the layer is optically smooth. The two dimensional growth mode is rather insensitive to most of the aforementioned parameters of coalescence as it will be shown later on, however the temperature of course strongly affects the deposition since growth is carried out in the thermodynamically limited growth regime.

As the layer thickness increases, lateral growth is promoted, the reflectivity rises again and begins to oscillate (FABRY-PEROT oscillations) due to interference in the GaN/sapphire layer. The amplitude of this oscillation is at its highest when a compact layer with a smooth surface has been established (AFM picture (d)). Eventually, when the deposition step has ended and the reactor cools down, the surface is stabilized with  $NH_3$  in  $N_2$  ambient. The transient displays this cooling-down as a "tail".

## 6.2 Growth Reproducibility Issues

The foremost parameter for the deposition of GaN buffer layers for the use in HEMT structures is the run to run reproducibility. Reproducibility comprises that equal results are achieved under identical growth conditions (growth temperature, precursor flow, V/III ratio etc.). Furthermore reproducibility allows to pinpoint that changes observed in the growth are directly related to changes of the respective growth parameter. Solely with reproducible results the actual growth process can be optimized by comparing data collected during (in-situ) and after (ex-situ) the deposition of the GaN film. Hence, the investigation of system response to intentional variation of growth parameters is therefore a key issue in growth of high quality GaN layers. Thus, once a set of parameters is established, the reproducibility of which is paramount, resulting in reproducible layer characteristics. This is of particularly interesting for HEMT device growth with its high demands on material quality. In addition, a reproducibly high yield of adequate material is required,

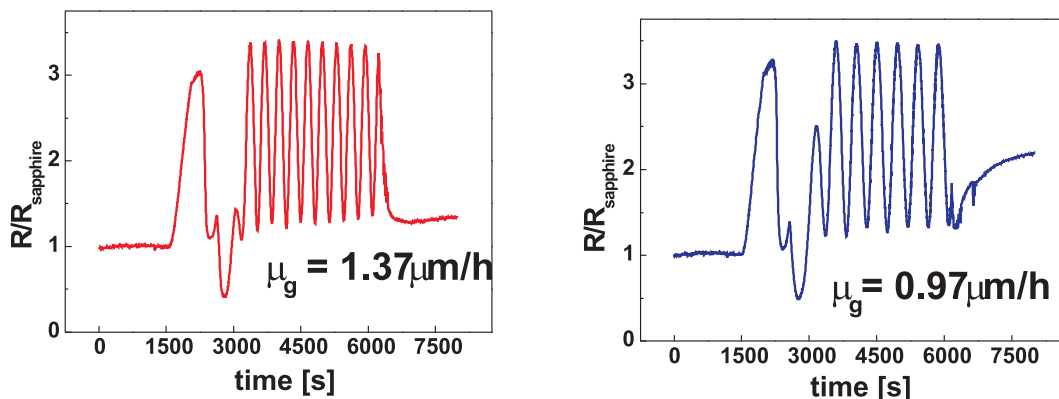


Figure 6.2: Comparison of two nominally identical growth runs: The right transient demonstrates a higher growth rate. The coalescence mode is also distinctly changed.

since high production costs represent a significant road block in the development of RF HEMT devices for high power applications.

While the material quality may be improved by optimization of various growth parameters as has been described in this chapter, the stability of the growth process is mainly related to the ability of the equipment to allow a successful and reproducible control of the parameters crucial for growth. However, the aforementioned aspects are still open issues. The reproducibility needs to be tested either by performing consecutive GaN growth runs or by repeating a chosen test-run in the same liner after a certain number of experiments. Only runs performed under identical experimental conditions (growth temperature, reactor pressure, precursor flows, V/III ratio are kept constant) are considered for the reproducibility analysis. Earlier investigations revealed a drastic shift of growth parameters [Har04, Kal03, Ste03]:

A gradual systematic change of the transients from run to run was observed. This is best resolved for two identical runs. Figure 6.2 presents the transients of the 3rd and the 12th run in the reactor. According to a higher growth rate the spacing of the Fabry-Perot oscillations in the HT growth regime is narrower for the 3rd run than for the 12th. The coalescence is also distinctly changed as can be evidenced by the morphology development in this stage. In the LT growth regime the ascent to the first oscillation is higher for the 12th run than for the 3rd run according to a higher growth rate. These observations can be explained by an increase in temperature in the LT and HT growth steps. The effect however is different. In the LT step, growth is kinetically controlled and the increase in temperature leads to a higher growth rate. In the HT step, growth is thermodynamically controlled so that an increase in temperature leads to a decrease in growth rate and faster coalescence. These results indicate that the temperature controlled by the MOVPE equipment is not identical with the substrate temperature. The reproducibility of sub-

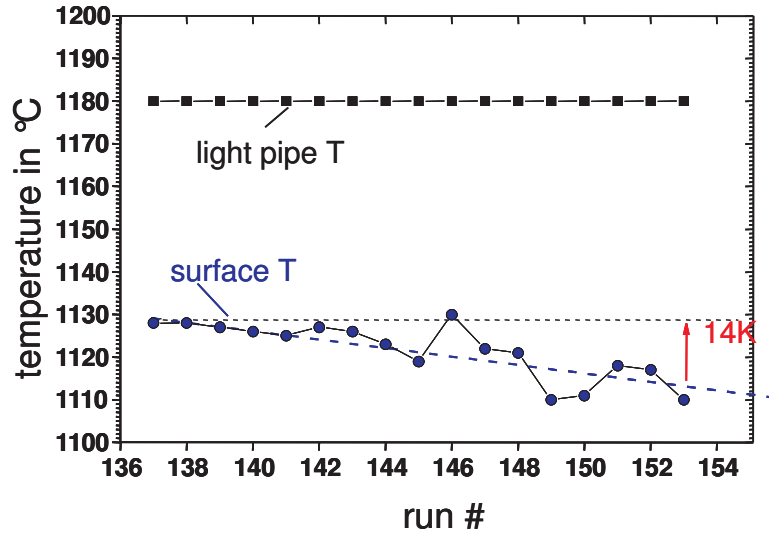


Figure 6.3: Comparison of control temperature (light pipe) at 1180 °C with surface temperature for a series of successive growth runs. The surface temperature changes here in the range of 14 K and the absolute temperature difference is more than 50 K.

strate surface temperature versus light pipe temperature (1180°C) is presented in Fig. 6.3. The surface temperature varied in the high temperature regime by up to 14K and an absolute temperature difference of more than 50K was observed. As a result the strongly temperature dependent growth needs to be controlled by means of in situ temperature monitoring of the substrate as discussed in chapter 5.

The only systematic change from run to run in the reactor is the increasing amount of parasitic deposits which form on the quartz parts of the chamber. The deposits will change the sample temperature [Hab02b, Har04] due to the change of heat radiation away from the sample as mentioned afore. Therefore the deposits are the real origin of the observed temperature effects. However that's not their only influence on growth. The role of parasitic deposits in MOVPE reactors has been a matter of great discussion in the past [Har04]. These deposits act as a catalytic surface, depleting the gasphase from growth species. Furthermore parasitic deposits impede the continuous usage of the employed reactor parts since the deposits tend to cause particles and/ or lead to a flaking which in turn ruins the surface morphology of the growing layer. The uptime and therefore the efficiency of the growth process is decreased. The accumulation of these films therefore needs to be prevented.

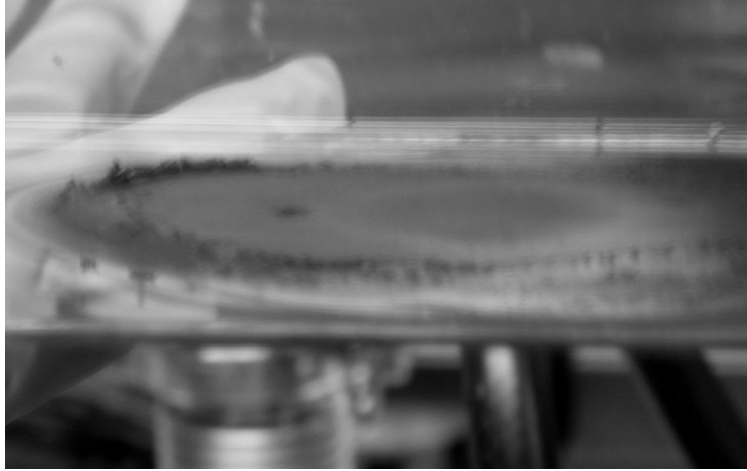


Figure 6.4: Parasitic liner deposits at the liner ceiling

### 6.3 New Process Approach: The Inverted Inlet Configuration

Usually, in horizontal MOVPE reactors, the group III and group V sources are injected through two separate channels. The upper of which contains the group III source materials and the lower of which contains the group V sources. This is done in order to prevent pre-reactions and to increase the group V source gas concentration near the substrate surface. We, however, propose to invert the source gas supply (Fig. 6.5) with the intention of reducing parasitic deposition and increasing reactor uptime and process efficiency [Har04].

Of all the possible chemicals in an MOVPE reactor the metalorganic source is mainly responsible for parasitic deposition. To get an idea of the constituents of the deposits a quantitative analysis of several samples from the liner ceiling and the quartzbowl was assessed. As a result, all samples were Ga-rich "GaN layers" with traces of oxygen and carbon coming from the MO sources.

Thus, the aim of this study is to avoid the contact of the growth limiting species with the hot reactor walls. Here the deposition process is kinetically limited due the relatively low temperatures on the ceiling. If reactor wall temperatures rise higher than  $400^{\circ}\text{C}$ , metalorganics and group V hydrides deposit by a mechanism similar to the growth of the nucleation layer on the substrate. In order to avoid parasitic deposition we therefore recommend to inject the metalorganic sources close to the heated substrate and far away from the unintentionally heated reactor parts. To establish a broad comprehension of the new epitaxial process approach, numerical simulations and growth modelling were employed. The commonly applied growth process is compared to the new one by comparison of experimental data. The main task of this section is to prove the functionality

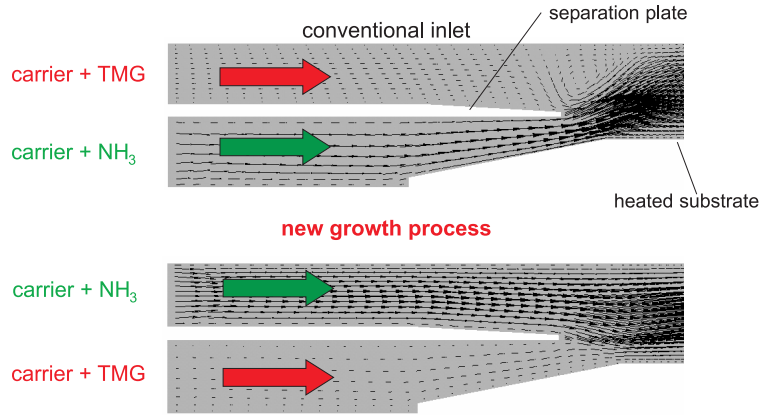


Figure 6.5: Sketch of inlet configuration and modelling of flow patterns: comparison for the conventional (a) and the inverted inlet (b) geometry. The flow velocity is high on the ammonia channel side because of a high mass flow and a comparably small cross sectional area where the mass flow passes through. Thus, for the conventional inlet the flow is directed towards the ceiling by a vortex. The inverted case demonstrates no such effects.

of the growth approach.

### 6.3.1 Growth Modelling

The principal characteristics of the inverted and conventional inlet growth processes were studied and compared using modelling. The modelling was performed by the Semiconductor Technology Research GmbH in Erlangen. The mathematical model is based on the solution of the 2D/3D Navier-Stokes conservation equations describing flow, heat transfer, and multi-component diffusion of the gaseous mixture inside the reactor [Bir60], taking into account the specific reactor geometry, the inlet design and the presence of wall deposits and particles. However, numerical simulation can only be performed for the high temperature deposition regime. A brief introduction is found in chapter 3. The kinetic mechanism of MOVPE gas-phase reactions is based on the work of Mihopoulos [Mih99]. To simulate the surface chemistry, a quasi-thermodynamic approach was used as described in chapter 3. A further discussion of hydrodynamic effects is also found in chapter 3.

For high temperature GaN growth, the boundary conditions were defined by the experiments and they are summarized below (Table 6.1). For the established set of parameters in table 6.1 solely the inlet was switched from the conventional setting to the inverted one, thus facilitating the investigation of principal transport characteristics for the inlet geometries and their respective impacts on growth. In figure 6.6 the temperature distribution in the reactor and on the ceiling is presented. It is not influenced by the inlet

|                       |                                                                                                         |
|-----------------------|---------------------------------------------------------------------------------------------------------|
| Precursors flow rates | TMGa = $6.63 \times 10^{-5} \text{ mol/min}$<br>NH <sub>3</sub> = $5.80 \times 10^{-2} \text{ mol/min}$ |
| Carrier gas           | H <sub>2</sub>                                                                                          |
| Total flow            | 7.8 <i>slm</i>                                                                                          |
| MO/ Hydride flow      | 6.8/ 1 <i>slm</i>                                                                                       |
| Growth temperature    | 1100°C                                                                                                  |
| Reactor pressure      | 200 mbar                                                                                                |
| Substrate:            | $\alpha$ -sapphire                                                                                      |

Table 6.1: Boundary conditions for a standard GaN growth process

configuration.

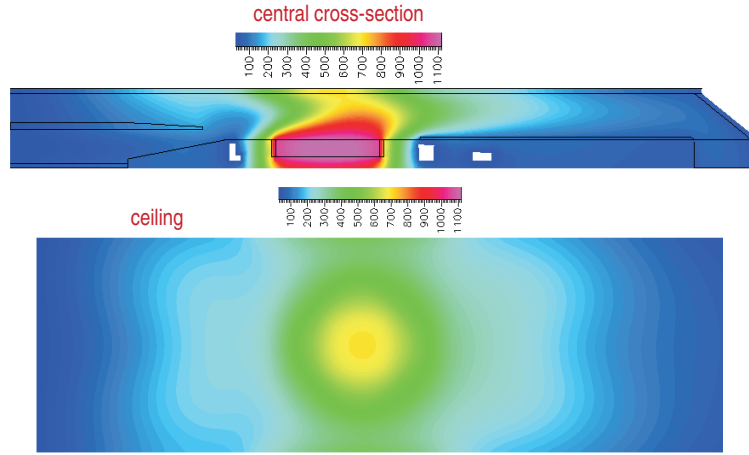


Figure 6.6: Temperature distribution in a cross sectional view and on the liner ceiling.

The inlet variation changes the transport, which governs the access of the reactive species to the sample surface. Thus, the flow patterns, as presented in Fig. 6.5, are compared for both processes. In the ammonia channel the total flow is higher than on the MO channel. The upper channel has a larger cross-section than the lower channel in the vicinity of the separation plate edge, where the mixing of the two flows takes place. This leads to a much higher gas velocity at the separation plate end in the ammonia channel for the conventional inlet than for the inverted one. A turbulence at the tip of the separation plate results from this gas distribution. The total flow distribution <sup>1</sup> together with the density of the gas and the temperature gradient in the reactor leads to buoyancy driven convection (or natural convection) as is clearly seen by the vortex. As for the inverted inlet, the flow seems to distribute evenly into the heated susceptor area, forming a laminar flow. These flow phenomena, of course, have an immediate impact on the distribution of

<sup>1</sup>governed by forced convection and diffusion, as discussed in chapter 3.

the species in the gasphase as we will discuss in the following.

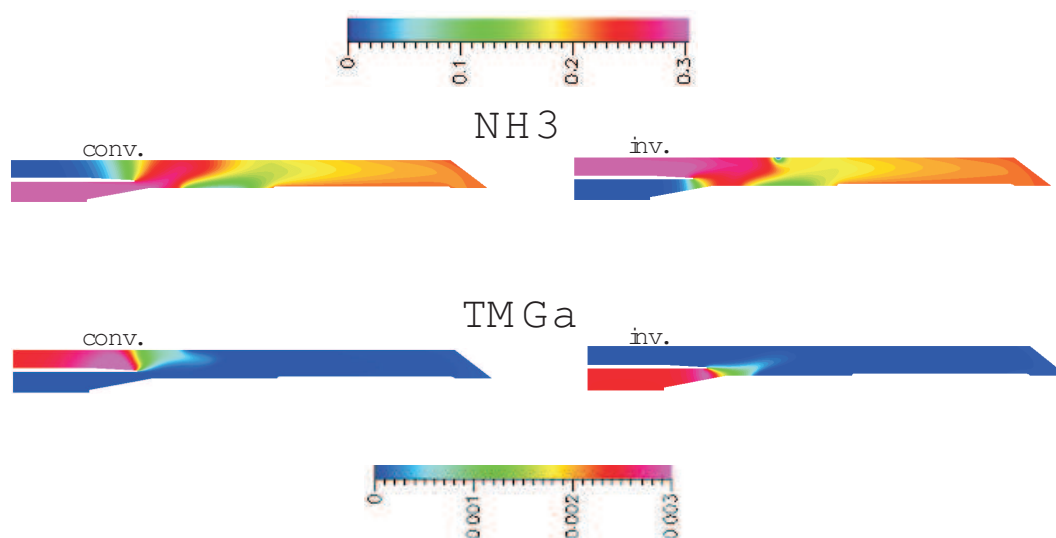


Figure 6.7: Comparison of  $\text{NH}_3$  and  $\text{TMGa}$  distribution for the conventional (a) and the inverted inlet (b) geometry. The concentrations are translated into colors: blue for low and red for high concentrations. The ammonia and  $\text{TMGa}$  concentration close to the ceiling is higher for the conventional inlet configuration.

Figure 6.7 serves as an illustration of growth species distribution in the central cross section of the reactor for the conventional and inverted inlet case. The molar concentrations are translated into colors: red for high and blue for low concentrations. Ammonia spreads towards the liner ceiling for the conventional case, whereas it is more homogeneously distributed throughout the growth area for the inverted inlet. Qualitatively the same behavior is predicted for the distribution of  $\text{TMGa}$ . Since it is injected close to the liner top it stays there. Its respective concentration is higher at the ceiling than for the inverted inlet, where the color scheme shows the highest concentration is to be found near the substrate.

Fig. 6.8 presents the transversal cross-section of the reactor at the location of the middle of the wafer and the distribution of monomethylgallium ( $\text{MMGa}$ ), the main gallium reagent for growth. For the conventional inlet growth process  $\text{TMGa}$  readily reaches the heated regions of the reactor ceiling above the substrate providing favorable conditions for the formation of parasitic deposits. Therefore the concentration of  $\text{MMGa}$  is higher near the ceiling of the reactor. In the inverted case the concentration of  $\text{TMGa}$  molecules is higher closer to the susceptor and react there to form the growth species  $\text{MMGa}$ .

Fig. 6.9 predicts the growth rate distribution in flow direction over the middle of the wafer according to modelling of non-rotated samples. The main reason for the change of profiles is found in the distribution of the growth determining species near the heated

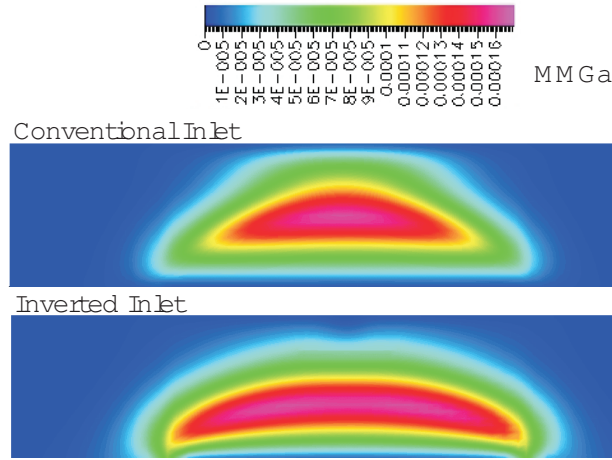


Figure 6.8: Modeling: Comparison of MMGa distribution for the conventional (a) and the inverted inlet (b) geometry. A transversal cross-section of the reactor is presented. The concentrations are translated into colors: blue for low and red for high concentrations. The MMGa concentration is lower towards the ceiling and higher towards the substrate in the inverted inlet configuration

substrate. The susceptor region creates a strongly heated area on the reactor bottom. The growth pattern is determined by the transport of growth limiting species to the susceptor surface. The close injection of TMGa to the substrate for the inverted inlet leads to a shorter diffusion time for MMGa to reach the substrate. Therefore deposition starts directly at the rim of the heated susceptor, a deposition maximum is found. Afterward the gas phase is gradually depleted of the source chemicals in flow direction. The gas phase is less strongly depleted of the nutrients toward the side walls of the reactor, because the gas phase at the sidewalls experiences the heated zone later, due to the disc shape of the susceptor. Therefore the layers are thicker towards the edge of the wafer. The growth maximum for the *conventional inlet* is positioned on the middle of the substrate. The MMGa has a longer diffusion length since it is delayed through a denser ammonia containing gas phase and forced convection carries the growth species further into the reactor.

All in all the effect of the conventional inlet on growth is an enhanced parasitic deposition by a factor of more than 3 on the quartz ceiling of the reactor as can be seen in Fig. 6.10. The maximum of this deposition is unfortunately upstream of the wafer for the conventional process so that it can react catalytically with the process gases before they reach the substrate whereas it is displaced downstream for the other geometry. Therefore it is predicted that the inverted inlet process should be advantageous for high reactor uptimes and good morphology with respect to flaking and particle formation and a higher run to run reproducibility.

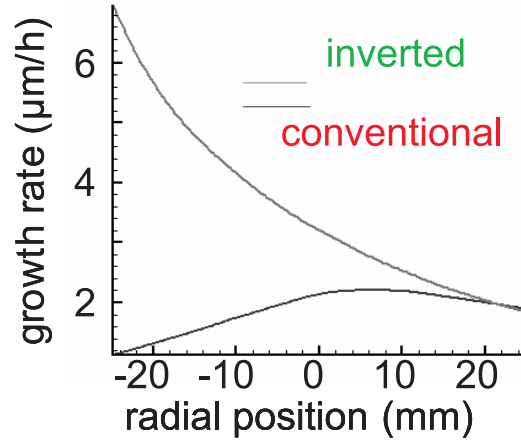


Figure 6.9: Expected growth profiles according to modelling in flow direction over the middle of the wafer. For the conventional inlet (lower line) a growth maximum is expected in the middle of the wafer. The inverted inlet (upper line) geometry exhibits a maximum at the front of the wafer.

### 6.3.2 Experimental Verification: Inverted to Conventional Inlet

Our experiments verify the predicted greatly reduced parasitic deposition in the inverted inlet geometry. Typically, at least 40 runs of  $2\mu\text{m}$  thick GaN can be carried out without removing parasitic deposits from the reactor ceiling instead of less than 10 growth runs for the conventional growth process. Also the surface temperatures and the growth rates remained constant from run to run with the new process. There are further characteristics which differ when comparing the transients (figure 6.11) for both growth processes:

- The growth rate of the nucleation layer is about one third higher for the inverted inlet geometry. This can be explained taking into account the different MMGa concentrations near the substrate surface. In the conventional inlet, the MMGa concentration is higher near the ceiling of the reactor and just a limited amount will diffuse through the boundary layer contributing to the GaN on the substrate. In contrast, in the inverted inlet, where the TMGa is supplied through the lower channel, the MMGa concentration in the vicinity of the substrate is higher, it will react more efficiently with the N-containing species, resulting in an increase of the nucleation growth rate [Kal04].
- The coalescence time is significantly shorter for the inverted inlet geometry. A reason for the shorter coalescence might be the higher availability of MMGa near the substrate therefore enhancing lateral growth, in turn leading to faster coalescence (compare chapter 7).
- The growth rate in the HT regime is lower for the inverted inlet geometry. As for the

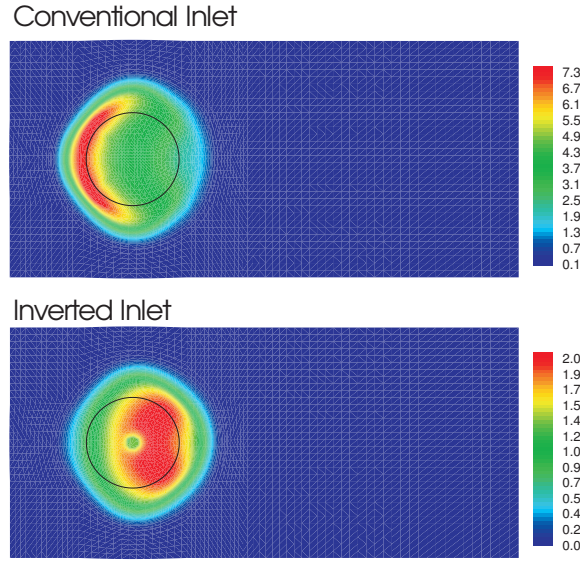


Figure 6.10: Comparison of growth rate distribution over the ceiling of the horizontal reactor above the substrate (marked with a circle) for the conventional (upper image) and the inverted inlet (lower image) geometry. The growth rates are translated into colors: blue for low and red for high rates. The growth rate is more than three times as high for the conventional inlet compared to the inverted inlet. The deposition maximum is positioned upstream of the substrate for the conventional inlet.

lower growth rate in the HT regime some mechanism of material losses seems to be activated at the reversed precursor supply associated with the total species flow and gas residence times. It might as well be due to the fact that the measuring point for growth rate determination is located in the middle of the wafer, whereas the maximum deposition rate is at the wafer rim. Therefore the growth rate depends, in short, on the configured growth parameters, which need to be optimized.

Fig. 6.12 presents thickness mappings of both runs. Considering the thickness distribution it becomes apparent that the growth profiles differ considerably. For the conventional growth process the growth profile is convex whereas it is concave for the inverted inlet geometry.

Starting from figure 6.9 and using wafer rotation, concave growth profiles are produced in the inverted inlet. The standard deviation in thickness with  $2mm$  edge exclusion is 6.5% and 10.4% for the conventional and the inverted inlet geometry, respectively. Due to the different convective to diffusive reactive species' flow ratios, each inlet geometry will need its own strategy to obtain optimum uniformity (compare chapter 7).

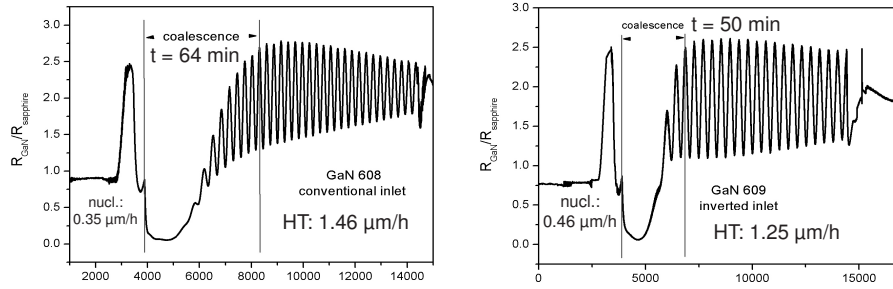


Figure 6.11: Comparison of transients (reflection measurement taken from the wafer middle) for both growth processes. The coalescence time is significantly shorter and the growth rate in the HT regime lower for the inverted inlet geometry.

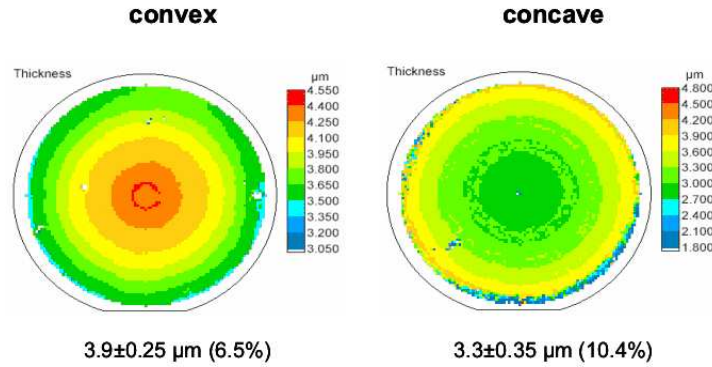


Figure 6.12: GaN growth profiles obtained using the conventional inlet (a) and the inverted inlet (b) geometry. For the conventional inlet a convex growth profile is observed, for the inverted inlet a concave profile is found.

The surface roughness is comparable at the microscopic and nanometer scale for both processes as figure 6.13 demonstrates. The AFM inset shows a slightly different surface morphology for the inverted inlet. Considering that the coalescence phase, which can contribute to the surface morphology, is somewhat shorter and the overall thickness is thinner for the "inverted" growth run, the change in morphology may be explainable.

The structural quality of the material is state of the art and very similar for both samples taking the different total layer thicknesses into account. The GaN layer grown by the conventional process the full-width at half maximum (FWHM) of the 002 rocking curve is 233 for the inverted inlet GaN film the FWHM is 244 *arcsec*.

The electrical properties of the grown material were investigated by room temperature Hall measurements and Leighton contactless sheet resistance mappings. It was found that the sheet resistance is lower by 10% for the inverted inlet at an absolute value of around  $360 \frac{\Omega}{\square}$  and its uniformity over the wafer is quite comparable for both process

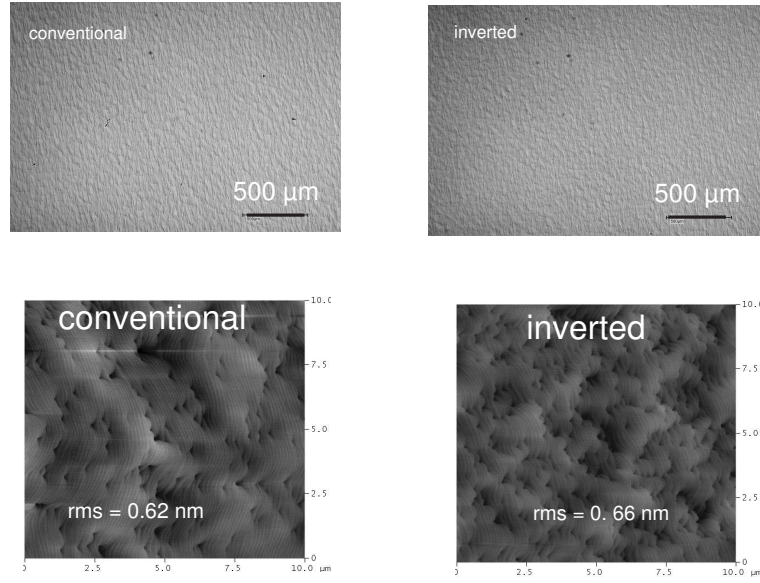


Figure 6.13: Comparison of surface morphology at microscopic (upper image) and nanometer scale (lower image) for both processes. The AFM inset shows a slightly rougher surface for the inverted inlet.

approaches. Intentionally Si doped samples exhibited a carrier concentration of  $1.0$  and  $1.2 \times 10^{17} \text{ cm}^{-3}$  and an electron mobility of  $605$  and  $544 \text{ cm}^2/\text{Vs}$  for the conventional and the inverted geometry, respectively. Here again the samples are state of the art and comparable for both growth processes. Table 6.2 summarizes the characteristics of both processes. However, it is important to further characterize the layer properties, so far these results prove the functionality of the new process.

| Parameter                                                      | Conventional inlet                 | Inverted inlet                      |
|----------------------------------------------------------------|------------------------------------|-------------------------------------|
| Growth rate at $550^\circ\text{C}$ ( $\mu\text{m}/\text{h}$ )  | 0.35                               | 0.46                                |
| Growth rate at $1100^\circ\text{C}$ ( $\mu\text{m}/\text{h}$ ) | 1.46                               | 1.25                                |
| Coalescence time (min)                                         | 64                                 | 50                                  |
| Growth profile                                                 | Convex                             | Concave                             |
| Thickness ( $\mu\text{m}$ )                                    | $3.90 \pm 0.25 \mu\text{m}$ (6.5%) | $3.32 \pm 0.35 \mu\text{m}$ (10.4%) |
| Roughness, rms (nm)                                            | 0.622                              | 0.655                               |
| XRD: FWHM (0002) (arcsec)                                      | 233                                | 244                                 |
| Sheet resistance ( $\frac{\Omega}{\square}$ )                  | $402 \pm 129$ (32%)                | $362 \pm 101$ (28%)                 |
| n300 ( $\times 10^{17} \text{ cm}^{-3}$ )                      | 1.0                                | 1.2                                 |
| $\mu_{\text{Hall}} 300\text{K}$ ( $\text{cm}^2/\text{V s}$ )   | 605                                | 544                                 |

Table 6.2: Comparison of morphological, structural, electrical and uniformity data for the conventional and inverted inlet geometries

### 6.3.3 Conclusion

Improving reproducibility was the aim of this chapter. A new growth approach for horizontal reactors is therefore introduced, in which the metalorganic source is injected near the substrate unlike the conventional process, resulting in reduced parasitic deposition. Experiment and modelling verify the suitability of the new growth approach for enhanced reproducibility. Moreover the new approach leads to a fourfold reactor uptime without changing reactor quartz parts. The structural, morphological and electrical characteristics of the grown layers are comparable for both processes. The main difference in the growth processes are the growth profiles which indicate that growth optimization must be carried out differently for both processes.



# Chapter 7

## Optimizing GaN Growth

Now that the functionality of the new GaN growth approach is proven the optimization of layers with respect to HEMT application can be tackled. In this context the major ingredients for an ideal GaN template should be reviewed:

1. "Manufacturing uniformity" in terms of homogeneous layer characteristics and reproducible layer growth with a high yield.
2. Good structural quality with low dislocation densities for high amplification by high channel conductivity at high frequencies;
3. A smooth surface with a low defect density, important for the growth of sharp interfaces as well as for device processing;

This chapter deals with the fulfillment of these criteria. First of all the homogeneity in terms of thickness distribution is addressed and then the improvement of electrical, structural and morphological properties is faced.

### 7.1 Homogeneity Optimization

The new process prevents parasitic GaN growth on the hot reactor walls by inverting the source gas supply and therefore leads to enhanced reproducibility and higher reactor uptimes. As reported in chapter 6 electrical and structural layer characteristics are quite comparable for both inlet geometries. However, the inversion of the precursor supply has a drawback consisting in a fairly high growth rate non-uniformity, with a typical standard deviation in thickness of around 10%. However, HEMT devices for power applications consume large areas of typically several  $mm^2$ . Thickness uniformity is therefore

paramount. Due to different flow dynamics and species distribution in comparison to the conventional process, the inverted growth process also needs different strategies to obtain uniform layers.

Flow-mechanical aspects in our reactor system are investigated in order to achieve a homogeneous thickness distribution. Due to the strong temperature dependence of the nucleation layer as well as the HT-GaN, temperature uniformity over the wafer is a prerequisite [Shi04]. Besides this, the most important factor is the distribution of the growth rate determining species in the gas phase close to the substrate. This distribution is also temperature dependent ("cold finger" effect) and moreover relies on parameters such as the carrier gas nature and flow velocities [Dau01, Har01]. For homogeneity optimization we particularly focus on the role of different carrier gases - nitrogen and hydrogen - during III-nitride growth.

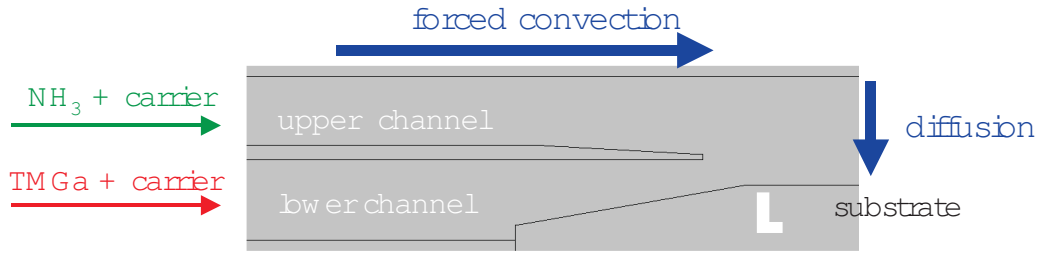


Figure 7.1: Forced convection and Fickian diffusion dominate the distribution of growth limiting species in the reactor and thus the homogeneity over the wafer. Forced convection moves the species in flow direction and parallel to the substrate surface, while diffusion moves the species perpendicular to the flow direction and to the substrate surface.

### 7.1.1 Optimization Strategy

Apparently, a flow parameter optimization becomes necessary to enhance the uniformity. Starting point for our considerations is the expected growth profiles according to modelling in flow direction over the middle of the wafer. The inverted inlet geometry exhibits a maximum at the front of the wafer with a nonlinear decrease over the wafer as seen in figure 6.9 in chapter 6. In order to linearize the growth rate and thus enhance the thickness uniformity the deposition maximum needs to be shifted closer to the substrate. A beneficial side effect is the enhanced growth efficiency.

According to chapter 3 the distribution of chemical species in the mass transport limited growth regime is determined by the hydrodynamics of the given flow. As depicted in figure 7.1 the mass transport is governed by forced convective (imposed pressure gradient)

and diffusive (Fickian diffusion) flux<sup>1</sup> of the growth limiting chemical species within the reactor, thus, determining the thickness homogeneity over the wafer [Str99]. A peculiarity arises when nitrogen is used as the carrier gas: In a high temperature process, as it is the case for GaN deposition, the presence of a region of colder gas (so called *cold finger*) is observed, penetrating from the inlet into the deposition zone (compare chapter 3). If the flow rate is low and the thermal conductivity is high, heat is conducted sufficiently fast relative to convection, so that there are no large temperature gradients around the susceptor. However, if the gas velocity is fast and/or a gas with lower thermal conductivity (e.g.  $N_2$ ) is used a thermal boundary layer develops. This can influence the decomposition of growth species and therefore change the supply of species to the surface.

In our reactor setup forced convection is driven by the pressure gradient between gas in- and outlet and the gas flow in the liner, thus dominating the flow velocity  $v_g$ , which is defined as

$$v_g = \frac{Q_{tot} \cdot p_{atm}}{A \cdot p_{tot}} \quad (7.1)$$

The flow velocity is controlled by the total flow rate  $Q_{tot}$  and the reactor pressure  $p_{tot}$ . Here,  $p_{atm}$  is the atmospheric ambient pressure ( $= 1bar$ ) and  $A$  stands for the cross-sectional area of either the inlet channel or the whole liner, depending on the gas velocity which is to be determined. Increasing the total flow leads to a different flow profile and hence to a different growth species distribution in the reactor.

Mass transport to the substrate itself is basically driven by Fickian diffusion and proportional to the mass flow  $m$ , which is defined as

$$m = \sum \frac{Q_i \rho_i}{A_i} \quad (7.2)$$

where  $Q_i$  is the total flow in channel  $i$  (either group III or V inlet) and  $\rho_i$  its respective density in the cross section  $A_i$ . Thus changing the density of the gas in a channel will yield a change of diffusion of the species towards the substrate.

Following the aforementioned dependencies two approaches are chosen.

1. The flow velocity is enhanced by increasing the total flow of  $H_2$  carrier gas in the reactor. Thus the gas phase depletion of growth species is reduced, due to shorter residence times.
2. Enhancing flow velocity and changing the diffusion rate by increasing the total flow in  $N_2$  ambient, thus increasing both viscosity and density of the carrier gas.

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<sup>1</sup>and natural convective flux (due to thermal gradients) which is, however, rather negligible.

Concurrently, the thermal conductivity within the gasphase is reduced, thus  $\text{MMGa}^2$  decomposition delayed.

Process modelling is employed to clarify the flow dynamics in this inlet configuration.

### 7.1.2 Experimental Results

In order to exclude effects due to different nucleation parameters, nucleation was always carried out with  $\text{H}_2$  carrier gas at  $550^\circ\text{C}$  until a layer thickness of about  $25\text{nm}$  was reached. After layer annealing HT growth was performed at constant conditions of  $200\text{mbar}$  reactor pressure and  $1100^\circ\text{C}$ . For the uniformity investigation the total amount of hydrogen or nitrogen carrier gas on the MO channel was stepwise increased right before HT growth, according to figure 7.2. The alkyl inlet was chosen since the growth limiting species is the MO precursor. The main growth parameters for our GaN growth process are summarized in table 7.1

|                  | GaN nucleation layer growth | Annealing            | GaN epilayer growth (HT growth) |
|------------------|-----------------------------|----------------------|---------------------------------|
| Temperature      | $550^\circ\text{C}$         | $1000^\circ\text{C}$ | $1100^\circ\text{C}$            |
| TMGa             | $15\text{ sccm}$            | -                    | $25\text{ sccm}$                |
| $\text{NH}_3$    | $2.2\text{ slm}$            | $2\text{ slm}$       | $3.3\text{ slm}$                |
| V/III ratio      | 1482                        | -                    | 1334                            |
| Total flow       | $8\text{ slm}$              | $8\text{ slm}$       | $9\ldots 15\text{ slm}$         |
| Flow MO side     | $1\text{ slm}$              | $1\text{ slm}$       | $1\ldots 7\text{ slm}$          |
| Flow Hyd side    | $7\text{ slm}$              | $7\text{ slm}$       | $8\text{ slm}$                  |
| Reactor pressure | $200\text{ mbar}$           | $200\text{ mbar}$    | $200\text{ mbar}$               |
| Time             | $1.5\text{ min}$            | $3\text{ min}$       | -                               |
| Thickness        | $25\text{ nm}$              | -                    | $\sim 2\text{ }\mu\text{m}$     |

Table 7.1: Growth parameters for a standard GaN growth process in an AIX 200/4 RF-S reactor (TMGa bubbler was used at a pressure of  $1000\text{ mbar}$  and a temperature of  $0^\circ\text{C}$ )

The wafers are characterized ex situ using the film thickness measurement method introduced in chapter 4. The standard deviation of the thickness distribution, as determined by film thickness measurements, is a measure of the achieved thickness uniformity. Growth rates are calculated with the in-situ recorded reflectance transient at  $600\text{ nm}$ .

Figure 7.3 presents growth rate (right) and thickness uniformity (left) versus the amount of additional carrier gas introduced through the lower inlet. Adding hydrogen, the growth

<sup>2</sup>MMGa is considered as the growth limiting species.

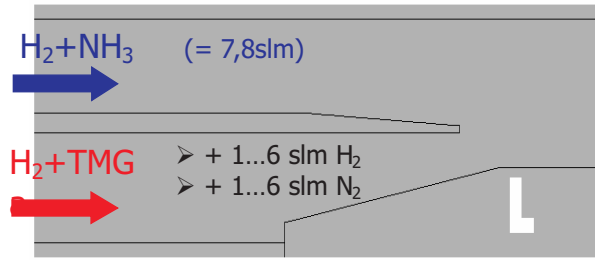


Figure 7.2: Inverted inlet geometry. The group III and group V sources are injected through two separate channels. The upper channel contains the group V source ( $NH_3$ ) and the lower one the group III (TMGa) sources. For the uniformity investigation the total amount of hydrogen or nitrogen carrier gas on the MO channel was increased stepwise

rate steadily increases in comparison to the starting point. In total the growth rate is enhanced by 20% at a total flow of 15 *slm*. For additional nitrogen in the carrier gas, the growth rate initially increases significantly by 35 % at an additional flow of 2 *slm*. After this peak it drops again with the heightened nitrogen flow, ending at similar growth rates as for the starting point. In pure hydrogen ambient, independent of the additional  $H_2$  flow, the standard deviation in thickness slightly increases (up to  $\sim 12$  %) and then is barely altered with a value around 11%. As nitrogen is injected in the MO channel the thickness deviation distinctly improves from 11% to  $\sim 3,5$  %. It is worth mentioning, that for the first added liter of nitrogen a similar behavior with the hydrogen series is found.

Thus, adding hydrogen shifts the maximum deposition rate towards the wafer center, since forced convection carries the growth species further into the reactor. Apparently this enhanced growth efficiency does not touch the homogeneity values. In contrast, adding nitrogen gas on the MO channel causes a simultaneous increase in forced convection, attenuated diffusion of the growth limiting species towards the heated substrate surface and probably a temperature change in the gas phase due to the cold finger effect. Basically the growth front is first pushed towards the wafer center (increasing growth rates) before diffusion limitation and delayed MMGa decomposition rivals the higher mass transport.

Summarizing, the enhanced uniformity is achieved due to the carrier gas nature, i.e. the carrier gas properties such as density, viscosity and thermal conductivity, and not due to the increase in forced convection with the higher total flow. Otherwise also the increased hydrogen flow would have led to improved uniformity. Process modelling is employed to verify the experimental findings and to elucidate the influence of nitrogen on mass transport in this inlet configuration.

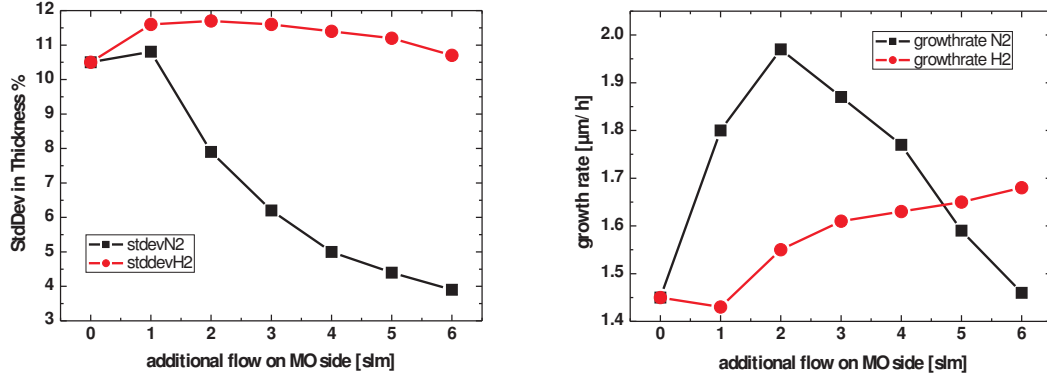


Figure 7.3: Standard deviation in thickness (left) and growth rate (right) in the inverted inlet as a function of different amounts of hydrogen (boxes) and nitrogen (circles) in the carrier gas.

### 7.1.3 Modelling Verification

Two runs are considered with  $1slm$  of  $H_2$  in the alkyl flow for the first case and an additional flow of nitrogen ( $1slm H_2 + 3slm N_2$ ) in the other case. These conditions are investigated, since convective effects can already be excluded as the origin of uniformity enhancement. Just the influence of nitrogen on species transport needs to be explained. Therefore, these two runs compare the starting condition to the enhanced growth species distribution.

Numerical simulations of the temperature distributions for both cases in the central cross-section of the reactor show a colder region ("cold finger") reaching from the separation plate tip to the susceptor when nitrogen is employed in comparison to the hydrogen case. This changes the growth species decomposition in the reactor. The main Ga-containing species contributing to the growth are the products of TMGa pyrolysis, within our considerations this species is MMGa. This distribution is analyzed next.

A horizontal cross-section located  $2mm$  above the growth surface is shown in figure 7.4. Here, the MMGa distribution is presented in a color code. A red shift means a higher MMGa molar fraction. Without nitrogen a concentration peak of MMGa is found at the wafer edge. The gradient of MMGa over the wafer is comparatively high. With nitrogen the concentration at the wafer edge is not so pronounced and thus the MMGa fraction gradient in flow direction is reduced. One can also see less curved isolines of the MMGa concentration in transversal direction. This leads to a more evenly distributed concentration of growth species over the wafer, with the concentration maximum being shifted downstream. These findings are responsible for the improved uniformity.

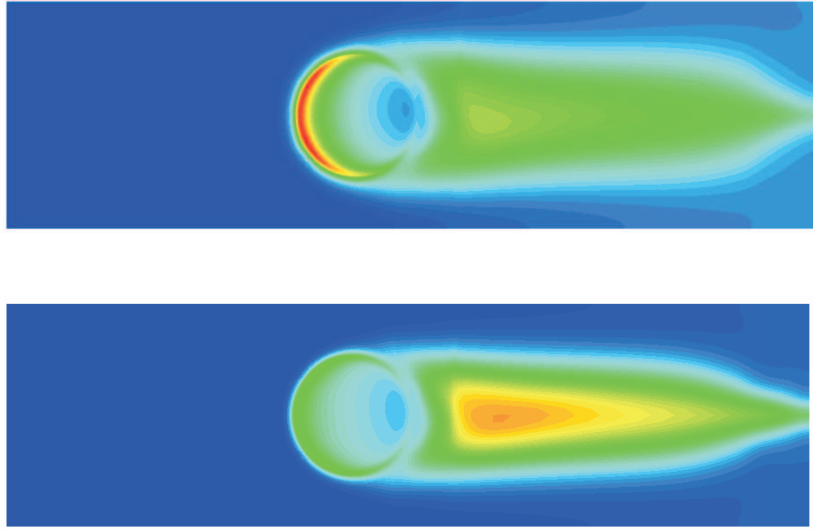


Figure 7.4: MMGa molar fraction distribution in a horizontal cross-section of the reactor. Without nitrogen (upper image) and with nitrogen (lower image) supply through the lower inlet. Red shift means higher MMGa molar fraction.

Figure 7.5 shows the predicted instant growth rate distributions over the wafer. The gradient of the instant deposition rate appears to be noticeably lower when nitrogen is employed. Moreover, less curved isolines [Ste03] are achieved that allows for improved uniformity. Ideally the profile should be block like [Dau01], since non uniformities perpendicular to the flow direction cannot be compensated by rotation.

The averaged distributions of the growth rate along the wafer centerline in figure 7.6 for the regimes without nitrogen (upper graph) and with nitrogen (lower graph) supply demonstrates that adding nitrogen into the alkyl mixture may improve considerably the layer thickness uniformity. Note, that absolute growth rate values are over estimated by modelling.

So far the importance of nitrogen for homogeneous growth in the inverted inlet configuration is experimentally and numerically proven, however, the growth conditions as depicted here with a maximum total flow of about  $15 \text{ slm}$  are above an often applied standard total flow for this type of horizontal reactor of around  $6\text{--}9 \text{ slm}$ . Furthermore the overall MOVPE process is more efficient at lower total flows. As stated earlier, the nature of the carrier gas, nitrogen which attenuates diffusion and delays decomposition, causes the improvement in uniformity. It is not necessary to employ these high flow rates in the reactor. Consequently growth was performed in pure nitrogen carrier gas at a reduced total flow of about  $9 \text{ slm}$  with growth parameters as summarized in table 7.2. Doing so the standard deviation in thickness reaches the minimum value of  $\sim 2.7 \%$ .

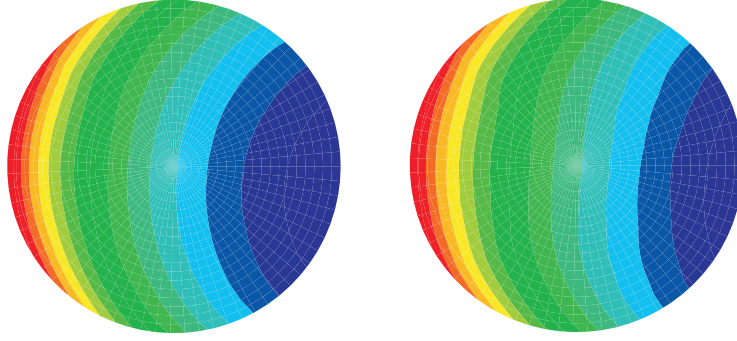


Figure 7.5: Instant growth rate mappings for the regimes without nitrogen (left) and with nitrogen (right) supply through the lower inlet. Less curved isolines are achieved with nitrogen.

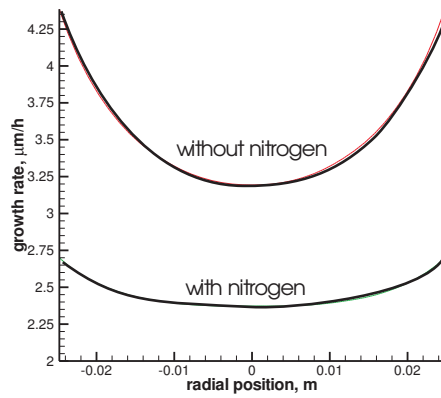


Figure 7.6: Growth rate distribution for rotated wafers along the wafer centerline for the regimes without nitrogen (upper line) and with nitrogen (lower line) supply through the lower inlet. Absolute growth rate values are over estimated by modelling.

#### 7.1.4 Conclusion

It has been found that inversion of the precursor supply has a drawback when considering the thickness uniformity. Two approaches were looked at to change the growth species distribution: An increase in forced convection and/or change of diffusive transport and delayed growth species decomposition. Uniformity can only be improved by changing the nature of the carrier gas, thus, modifying the diffusive transport and decomposition of growth species by adding nitrogen into the alkyl mixture. Thus, the growth species are distributed more uniformly above the susceptor. This way, in  $N_2$  atmosphere a standard deviation in thickness of less than 3% is achieved in comparison to 10% for  $H_2$  ambient. The investigation of layer properties is necessary to understand the influence of the nitrogen carrier gas on GaN epilayers characteristics.

## 7.2 On the Effect of $N_2$ on GaN Growth

For the MOVPE of GaN the majority of epitaxy groups report the usage of hydrogen as the preferred carrier gas. Successful growth processes in hydrogen have long been established. Nevertheless, there are others as well. Amano et al. used nitrogen as the ambient gas [Ama84]. Nakamura et al. reported the use of a gas mixture of  $H_2$  and  $N_2$  as the sub-flow gas [Nak91a]. These GaN epilayers, grown in the presence of  $N_2$ , show good uniformity and high quality. However, the majority of growth experiments in nitrogen led to the opinion that pure  $N_2$  is not only detrimental to the morphology and the structural quality of GaN but also to the electrical properties [Wan01]. The reason for these controversial findings [Muk98, Nak99, Jav02] may be due to different MOVPE reactor setups and non-adapted processes to the nitrogen ambient (one-to-one copy of hydrogen process). There is no detailed study of the influence of nitrogen on GaN growth. Thus, in this section GaN growth in different atmospheres, from pure hydrogen to pure nitrogen, will be systematically investigated.

### 7.2.1 Experimental Results

|                  | GaN epilayer<br>growth (HT growth) |
|------------------|------------------------------------|
| Temperature      | 1100°C                             |
| TMGa             | 25 <i>sccm</i>                     |
| NH <sub>3</sub>  | 2.2 <i>slm</i>                     |
| V/III ratio      | 877                                |
| Carrier gas      | 0...100% $N_2$                     |
| Total flow       | 9 <i>slm</i>                       |
| Flow MO side     | 6.5 <i>slm</i>                     |
| Flow Hyd side    | 2.5 <i>slm</i>                     |
| Reactor pressure | 200 <i>mbar</i>                    |
| Thickness        | > 2 $\mu\text{m}$                  |

Table 7.2: Growth parameters for the GaN growth process with increasing nitrogen amount in the gas phase

For all samples nucleation layer growth and annealing were carried out using  $H_2$  carrier gas under identical conditions as stated in table 7.1 in section 7.1. After the annealing process, growth was resumed and the successive high temperature growth step (HT-growth) was performed with different ratios of  $H_2/N_2$  carrier gas mixtures, ranging from 0% (pure hydrogen atmosphere) to 100% nitrogen at 1100°C while the total flow was kept constant at 9*slm*. In this way, the carrier gas composition was changed right after the annealing

step. The main growth parameters for the HT growth process are summarized in table 7.2

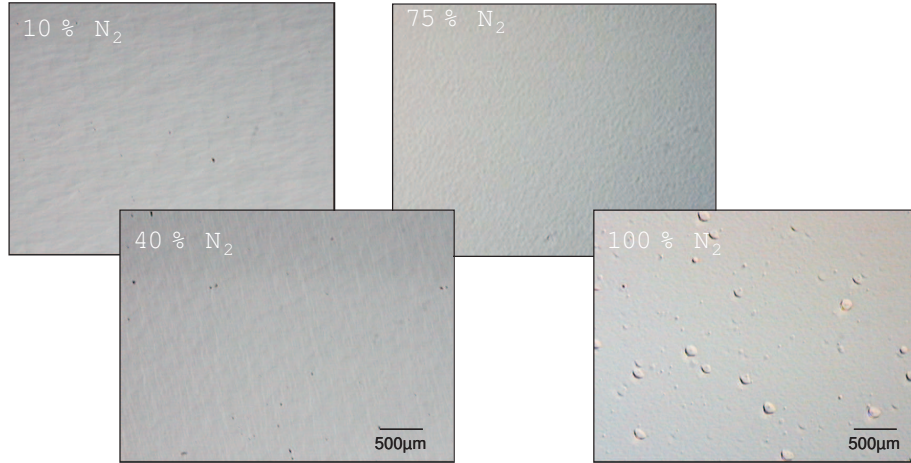


Figure 7.7: Nomarski-microscopy of samples grown with increasing amount of nitrogen in the ambient. In case of pure nitrogen the surface drastically worsens. Hexagonal structures and even small "turrets" can be observed.

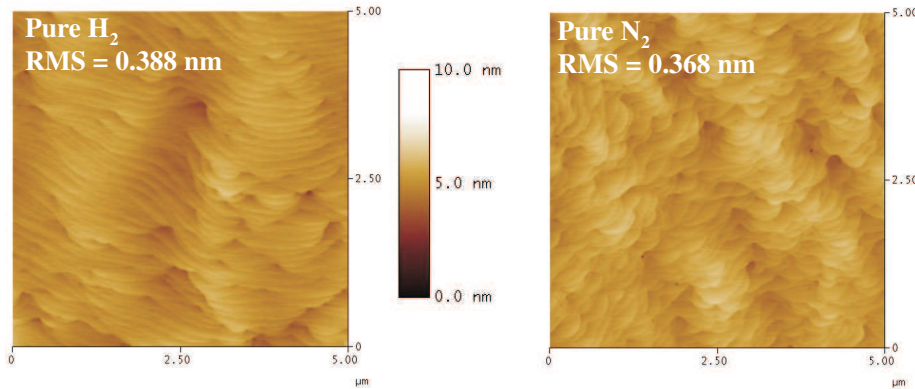


Figure 7.8: AFM micrographs of GaN layers grown under pure  $H_2$  (left) or  $N_2$  (right).

Figure 7.7 reveals the surface morphology as seen via Nomarski-microscopy of samples grown with increasing amount of nitrogen in the ambient. The morphology on the micrometer scale is rather smooth for any sample grown under hydrogen or mixtures of hydrogen and nitrogen. In case of pure nitrogen the surface drastically worsens. Hexagonal structures and even small "turrets" can be observed. Any further processing steps towards HEMT devices, such as metallization steps etc., are out of question for such a sample. Morphology optimization becomes necessary. However, *all* samples are specular when examined by the eye.

On the nanometer scale, the surface morphology also changes from  $H_2$  to  $N_2$  (Figure 7.8). For pure  $H_2$ , the surface exhibits smooth and parallel atomic steps. The higher the  $N_2$  content the more the steps become round and pits are observed. This is a first indication of an increased dislocation density. It is emphasized, however, that the roughness differs slightly but is comparably low (RMS:  $\sim 0.3nm$ ) for both samples.

The often reported surface degradation develops probably due to the different diffusion length of the adatoms on the surface. Due to the increased gas density in nitrogen, the adatoms have smaller mean free paths and cannot migrate over long distances to find favorable adsorption sites [Hal01, Wan01]. The structural properties may also be affected:

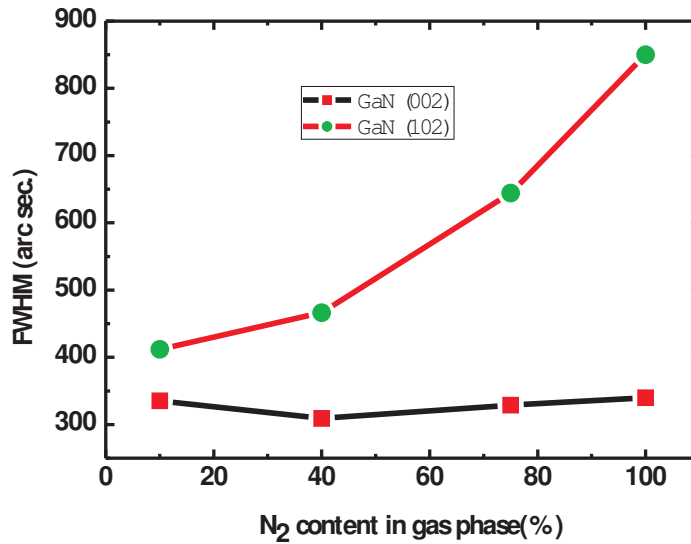


Figure 7.9: The rocking curves of the (002) demonstrate similar FWHM values of about  $330arcsec$ , while the (102) peaks increases significantly.

The crystal properties of the GaN epilayers were probed by X-ray diffraction using the symmetric (002) and asymmetric (102) reflection: The rocking curves of the (002) demonstrate similar FWHM values of about  $330arcsec$ , with only slight variations as the nitrogen content in the gas phase is changed, as seen in figure 7.9. In contrast, the FWHM of the (102) peaks increases significantly from 400 to  $900arcsec$ . The (102) rocking curve is broadened by all types of threading dislocations (TDs) such as the edge, screw and mixed types of TDs, while the (002) rocking curve is only broadened by pure screw and mixed types of TDs [Wan01, Bot01]. Obviously, mainly the edge dislocation density increases with increasing  $N_2$  content.

Grain boundaries are causal for dislocation formation. Apparently, in comparison to a

pure hydrogen atmosphere, smaller grains coalesce and dislocations arise at the edges of the islands. Higher dislocation densities, in turn, might be associated with strain relaxation, since dislocations accommodate the grown film to the substrate. Strain relaxation can be proven by, for example, PL measurements.

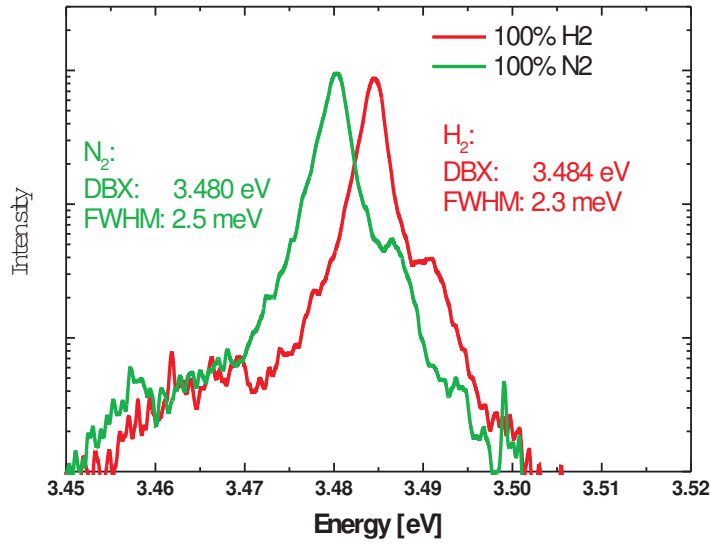


Figure 7.10: The donor bound exciton (DBX) peak is shifted towards lesser energies for an epitaxial growth run in pure nitrogen ambient in comparison to a growth run in pure  $H_2$ .

Photoluminescence measurements (figure 7.10) reveal a donor bound exciton (DBX) peak shifted towards lesser energies for an epitaxial growth run in pure nitrogen ambient in comparison to a pure  $H_2$  ambient. GaN layers grown on sapphire suffer from compressive strain. The GaN band gap increases in case of biaxial in-plane compression compared to fully relaxed GaN. The DBX energy therefore increases with increasing compressive strain in the layers. A lesser compressive strain is thus found in the layer [Cho06]. Similar results were obtained by Raman spectroscopy. The amount of strain decreases with increasing  $N_2$  content. These results indicate that internal compressive strain is released possibly by dislocation formation. Other groups also reported [Li04] different stress states when using nitrogen as the carrier gas, however did not mention the potential cause.

The degradation of the structural quality should also lead to a measurable impact on the electrical data, which is investigated next.

The electrical properties of the grown material were investigated by room temperature Hall measurements. Intentionally Si-doped samples exhibited a distinct impact of the

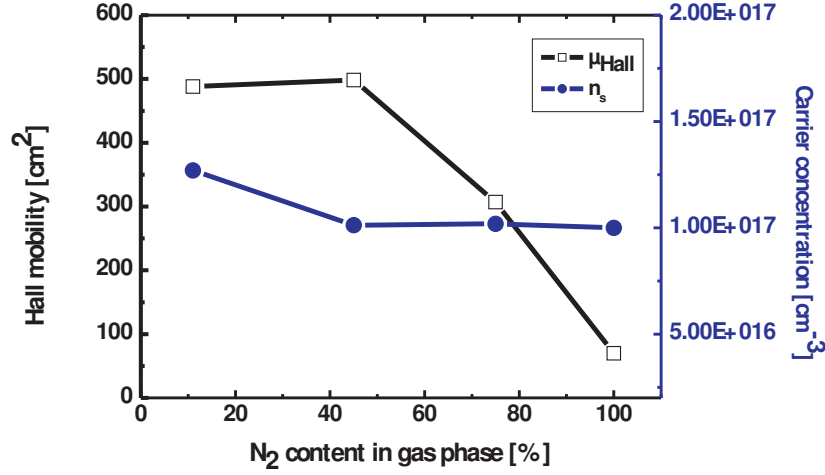


Figure 7.11: Hall measurements at room temperature.

carrier gas on the mobility of the GaN film. As the nitrogen content increases the mobility drops strongly from  $488 \text{ cm}^2/\text{Vs}$  at constant carrier concentration around  $1 \times 10^{17} \text{ cm}^{-3}$ . Obviously the deterioration of the electrical characteristics is related to a reduction of crystal structure quality with increasing nitrogen content. In particular the generated edge dislocations are electrically active, acting as acceptor like traps (compare chapter 8), and as scatter centers and thus carrier mobility is reduced (Eastman et al [Ng98]).

A lot of evidence has already been presented that the layer morphology, the structural quality and the electrical properties degrade and more dislocations are induced with increasing nitrogen content. The origin of these effects must be correlated with growth and thus with the morphology development. Hence, the growth transients will clarify the underlying growth mechanism.

The in situ measured reflectance transients at a wavelength of  $600 \text{ nm}$  for several growth runs with different carrier gas mixtures are presented in figure 7.12. The low temperature growth step, as well as the annealing step, use hydrogen as carrier gas. After these growth steps the coalescence phase starts in which the nuclei grow together. The beginning of this phase is denoted by the first dotted line in the transients. Gradually oscillations form which reach a constant intensity after complete two dimensional growth is observed. In each of the transients the second dotted line denotes the onset of complete 2D growth. With increasing nitrogen content in the gas phase (from bottom to top) the coalescence time (and therefore the “coalescence thickness”) decreases drastically from 2200 to 100 seconds. The coalescence time and thickness correlates with the size of the nuclei before

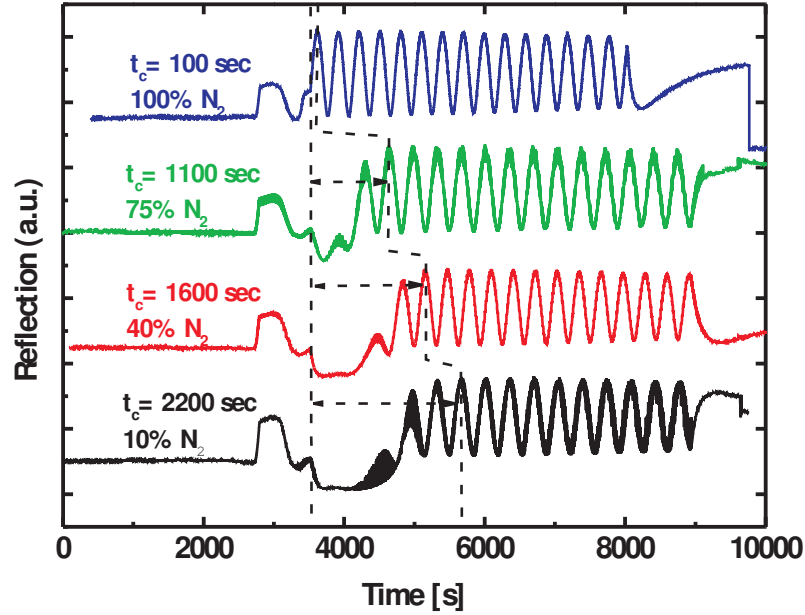


Figure 7.12: Effect of nitrogen on the coalescence time and thickness. With increasing nitrogen content in the gas phase the coalescence time (and therefore the “coalescence thickness”) decreases drastically.

neighboring grains merge: the smaller the thickness the smaller the grain size and the more dislocations can be found. Hence, the decrease in coalescence thickness with increasing nitrogen content leads to early strain relaxation.

These findings may be verified by results of other groups: Schenk et al. [Sch03] concluded that rapid coalescence leads to an increase of dislocations as determined by plan-view TEM. Böttcher et al. [Bot01] found that the density of edge threading dislocations decreases along with increasing “coalescence thickness” while the compressive stress increases. And finally Grzegorzczuk et al. [Grz05] correlate electrical properties to the influence of the carrier gas on the ratio of edge to mixed and screw type threading dislocations. However, each of these groups focused on single aspects of growth or epilayer properties, such as the coalescence phase or the resistivity. This study therefore clarifies the details of the influence of nitrogen on electrical, structural, morphological and optical GaN epilayer characteristics and correlates this with the overall morphology development in the deposition process.

### 7.2.2 Conclusion

Nitrogen is crucial for improved thickness homogeneity, but apparently nitrogen not only changes the surface morphology but it affects the growth mode in the early stages of deposition. From the presented data it is found that untimely recovery of the reflectance signal, i.e. start of 2D growth, leads to GaN layers which contain a larger number of edge type dislocations. Dislocations, in turn, influence the strain in the layer, leading to more relaxed GaN layers when grown in nitrogen ambient. Dislocation scattering is the very probable source of the decline in electrical data. As a result a larger "coalescence thickness" is preferred, since it reduces the density of dislocations. Therefore a strategy needs to be developed with which coalescence can be controlled despite the nitrogen ambient needed for uniform layers.

## 7.3 Electrical and Structural Optimization with Nitrogen

For high power and high speed performance of HEMTs it is of primal importance to maximize the channel sheet carrier concentration without reducing electron mobility and saturation velocity, thus, to form a highly conductive GaN channel. Therefore -in addition to a highly uniform GaN template- the optimization of morphology, structural and electrical quality is required, allowing for control of dislocation types and densities.

In this section the strategy is developed with which highly uniform and state of the art material quality GaN films are grown, despite the effects of nitrogen on morphology development.

### 7.3.1 Aim to Prolong Coalescence in $N_2$

An often reported approach towards prolonged coalescence is either lowering the deposition temperature and/or the V/III ratio [Fig00, Yan00, Kal03] during the coalescence phase. Originating from growth in pure  $N_2$  ambient, as shown in the foregoing discussion, we applied both methods as summarized in table 7.3. Nucleation layer growth and annealing conditions take place in hydrogen ambient and are the same as stated in table 7.2. During the coalescence phase the temperature was reduced to 1000 °C and afterwards increased to epilayer growth temperature of 1100 °C. In the next growth run the V/III ratio was distinctly reduced to 485 in comparison to 877 during the HT-GaN epilayer growth.

The resulting reflectance transients are presented in figure 7.13. In either case an immediate onset of small Fabry-Perot oscillations is observed. This suggests that, contrary

|                 | Coalescence    |                | HT-GaN         |
|-----------------|----------------|----------------|----------------|
|                 | lower T        | lower V/III    |                |
| Temperature     | 1000 °C        | 1000°C         | 1100 °C        |
| TMGa            | 25 sccm        | 25 sccm        | 25 sccm        |
| NH <sub>3</sub> | 2.2 slm        | 1.2 slm        | 2.2 slm        |
| V/III ratio     | 877            | 485            | 877            |
| Carrier gas     | N <sub>2</sub> | N <sub>2</sub> | N <sub>2</sub> |
| Total flow      | 9 slm          | 9 slm          | 9 slm          |
| Thickness       |                |                | > 2 μm         |

Table 7.3: Growth parameters for the prolonged coalescence run in nitrogen ambient.

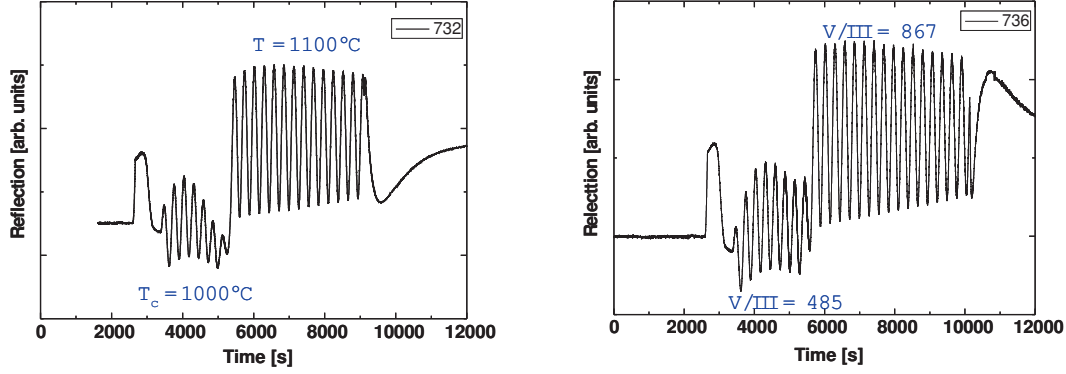


Figure 7.13: Reflectance transients of two growth runs in pure nitrogen ambient. Prolonged coalescence in neither achieved by applying a lower coalescence temperature  $T_c$  nor lower V/III ratio.

to our intention, 2D growth begins. On a morphological scale the coalescence mode is not controllable in nitrogen carrier gas. The electrical characterization by Hall measurements at room temperature of the intentionally Si-doped GaN yields very low electron mobilities. Applying a lower coalescence temperature results in an electron mobility of  $69 \text{ cm}^2/\text{Vs}$  at carrier concentrations of  $1 \times 10^{17} \text{ cm}^{-3}$ . Using a lower V/III ratio during coalescence results in a mobility of  $110 \text{ cm}^2/\text{Vs}$  at a carrier concentration of  $5.5 \times 10^{17} \text{ cm}^{-3}$ . In either growth run the surface of the samples reveal similar features to the pure nitrogen sample from figure 7.7: Hexagonal forms and turrets are observed.

A lengthened coalescence under nitrogen influence becomes impossible in our inlet configuration. Lacking control of coalescence results in poor electrical data. So the question arises whether it is an inherent property of nitrogen (thus a matter of growth chemistry) or due to accelerated coalescence that the crystalline quality of GaN layers degrades.

### 7.3.2 Role of Nitrogen in 2D Growth

To clarify this matter GaN was grown on a "hydrogen template", meaning the carrier gas composition was stepwise changed towards pure nitrogen, after the GaN film fully coalesced in  $H_2$  ambient. Consequently the coalescence time and "coalescence thickness" is the same for all growth runs. Figure 7.14 illustrates this. The line marks the transition (if applicable) between the carrier gases.

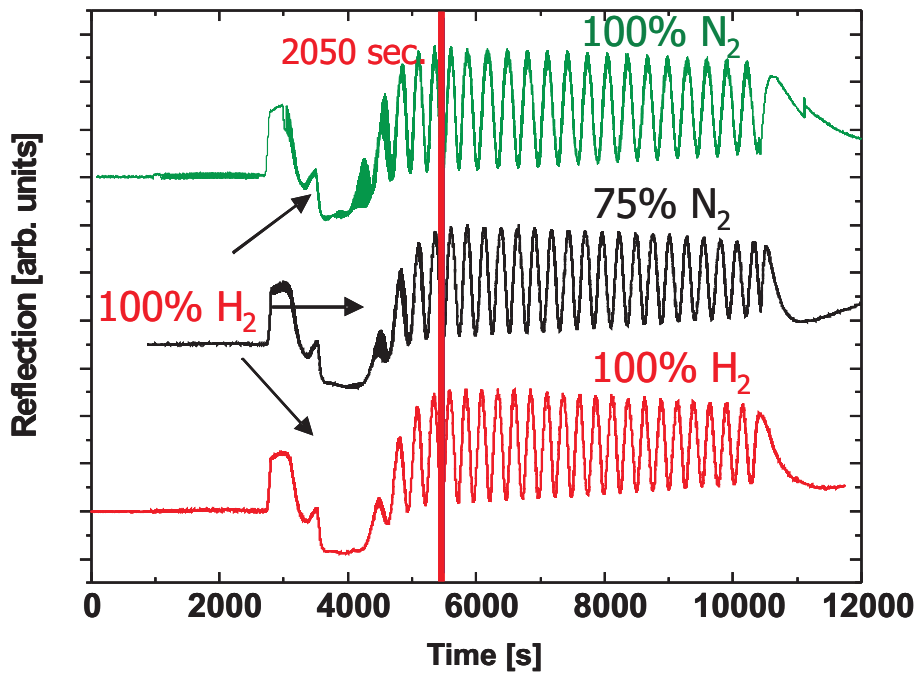


Figure 7.14: Growth transients: GaN is grown in different carrier gas mixtures on a "hydrogen template". The carrier gas composition was changed stepwise towards pure nitrogen, after the GaN film fully coalesced in  $H_2$  ambient

Table 7.4 summarizes several properties of the resulting layers. Considering the error of experiment, the electrical and structural data exhibit almost no disadvantageous influence of nitrogen on the 2D growth mode. The electrical data are comparable and state of the art at mobilities of around  $600 \text{ cm}^2/Vs$  for a carrier concentration around  $7 \times 10^{16} \text{ cm}^{-3}$ . The FWHM of the (002) reflections are also state of the art and decrease slightly from 262 down to 237 arcsec whereas the FWHM of the (102) reflections increase slightly from 388 to 415 arcsec. There are only minor changes of the surface morphology on the nanometer scale, which also holds for the RMS value. As expected, the thickness uniformity improves with increasing nitrogen content in the gas phase during 2D growth. However, since the GaN layers grow on top of a rather inhomogeneous "hydrogen template" even the high nitrogen amount cannot fully compensate that. Therefore uniformity is still improvable.

|                                                | <b>100 %H<sub>2</sub></b> | <b>75 %N<sub>2</sub></b> | <b>100 %N<sub>2</sub></b> |
|------------------------------------------------|---------------------------|--------------------------|---------------------------|
| <b>Electron mobility [cm<sup>2</sup>/Vs]</b>   | 609                       | 587                      | 598                       |
| <b>Carrier concentration [cm<sup>-3</sup>]</b> | $9 \times 10^{16}$        | $7 \times 10^{16}$       | $6 \times 10^{16}$        |
| <b>XRD: FWHM (002) [arcsec]</b>                | 262                       | 264                      | 237                       |
| <b>FWHM (102) [arcsec]</b>                     | 388                       | 387                      | 415                       |
| <b>AFM: RMS [nm]</b>                           | 0.26                      | 0.28                     | 0.4                       |
| <b>Std. Dev. [%]</b>                           | 9                         | 5.2                      | 4.3                       |
| <b>Thickness [<math>\mu m</math>]</b>          | 3.3                       | 3.2                      | 3                         |

Table 7.4: Properties of GaN layers with hydrogen coalescence and different amount of nitrogen during 2D growth.

It is concluded that the first growth steps (from nucleation to island coalescence) are more sensitive to growth parameter variation than the two dimensional layer by layer growth mode. Thus, there is no significant influence of the carrier gas composition on layer characteristics after full film coalescence is achieved. Its influence in the coalescence phase however is strong, here the layer characteristics are prefixed and determined. How nitrogen affects the coalescence mode in the early growth stages has to be subject to further investigations. We find hydrogen is crucial for the control of coalescence thickness. The general importance of hydrogen for GaN growth has been elaborated by works of Han et al. and Figge et al. [Han97, Fig00]. Hydrogen usually leads to larger crystallites with an overall lower spatial distribution and density on the surface. The presence of hydrogen in the gas phase is a necessity for controlled 3D nucleation. However, hydrogen is essential *solely* during the coalescence.

The task now is to combine the advantage (uniformity) and to avoid the disadvantage (fast coalescence) of N<sub>2</sub> as carrier gas in one process.

### 7.3.3 Optimizing GaN

Starting point for our considerations in this section is a "pure nitrogen process", as depicted in table 7.2. Here also nucleation layer growth and annealing take place in hydrogen, and gas switching takes place right after the annealing step has ended. Adding an intentionally low value of 15% H<sub>2</sub> to an otherwise nitrogen ambient increases the coalescence time to over 26 minutes. The Hall mobility improves significantly in comparison to growth in pure nitrogen from  $\mu = 69$  to  $401 \text{ cm}^2/\text{Vs}$  with  $n_s = 1 \times 10^{17} \text{ cm}^{-3}$ . Also the morphology becomes defect and "turret" free again and the surface is specular as probed by Nomarski microscopy. This result is the basis now for further optimization.

A further increase of the coalescence thickness becomes necessary to obtain good structural quality with low dislocation densities as postulated for the primary target of a high quality GaN template. Therefore, the influence of the growth rate at a constant V/III ratio on

| GaN epilayer<br>growth (HT growth) |                                         |
|------------------------------------|-----------------------------------------|
| Temperature                        | 1100°C                                  |
| TMGa                               | 15 ... 20 ... 25 <i>sccm</i>            |
| NH <sub>3</sub>                    | 1.3 ... 1.7 ... 2.2 <i>slm</i>          |
| V/III ratio                        | 877                                     |
| Carrier gas                        | 85% N <sub>2</sub> / 15% H <sub>2</sub> |
| Total flow                         | 9 <i>slm</i>                            |
| Thickness                          | > 2 $\mu\text{m}$                       |

Table 7.5: Growth parameters for prolonged coalescence in a 85% N<sub>2</sub>/ 15 % H<sub>2</sub> carrier gas mixture.

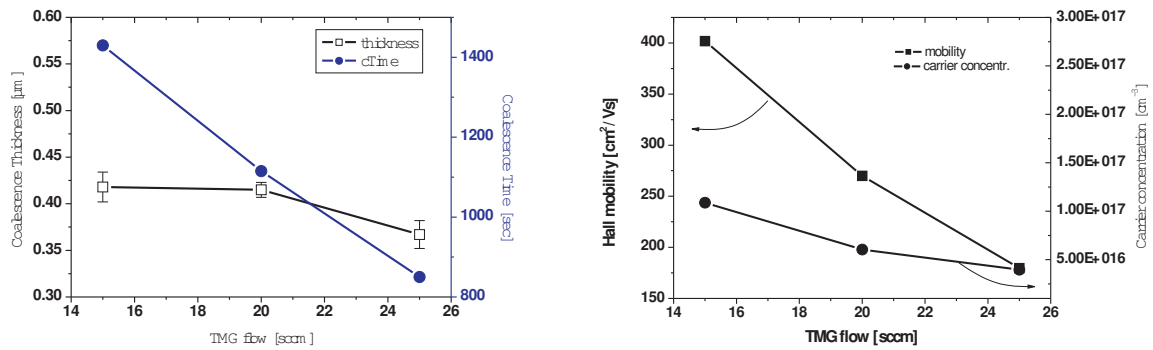


Figure 7.15: With increasing TMGa flow in the gasphase the coalescence time is almost halved, while the coalescence thickness is constant (left). This affects also the electrical data (right): The shorter the coalescence time (with higher TMGa flow) the lower the mobility.

coalescence thickness and time as well as on material properties were studied. Table 7.5 summarizes the growth conditions during the HT-GaN deposition.

Figure 7.15 (left) shows the effect of growth rates on the coalescence thickness and time. The thickness was calculated from the coalescence growth rate determined by the in-situ reflectometer at 600 nm.

The coalescence time decreases as a function of the TMGa amount (thus the growth rate) while the coalescence thickness is constant. That means the islands coalesce more rapidly at a high growth rate, due to a lowered aspect ratio (ratio of vertical to lateral growth rates). Thus, lateral growth of the islands is enhanced [Yan00] or vertical inhibited. A fast transition towards 2D growth is promoted. A lower growth rate increases the

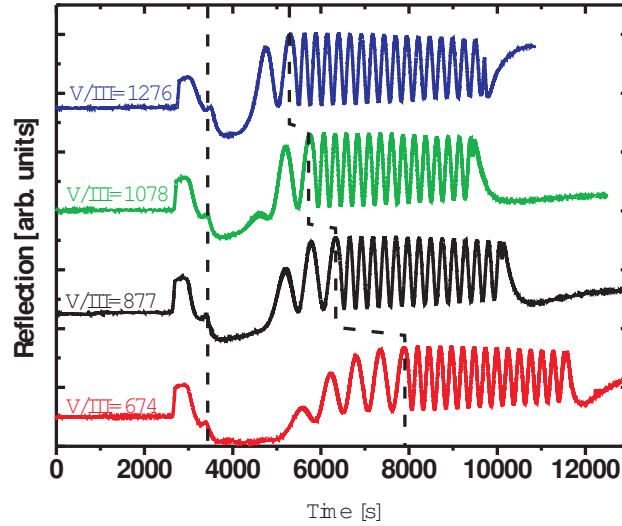


Figure 7.16: Applying a lower growth rate in combination with a reduced V/III ratio during the initial stages of growth results in an optimized coalescence growth rate and a thicker “coalescence layer”

aspect ratio which leads to larger islands in the early stages of HT-growth. Datta et al. [Dat04] propose that larger islands mean grains with inclined side facets in comparison to those with higher V/III ratio or growth rate. These inclined facets cause dislocations to bend  $90^\circ$ , therefore the probability for mutual annihilation of TDs is raised and therefore structural quality enhanced. If this assumption is true also the electrical data should improve. Indeed, Hall measurements at room temperature (figure 7.15 (right)) show that with lower growth rates, (thus increasing coalescence thickness) the hall mobility increases from  $\sim 200$  to  $\sim 400 \text{ cm}^2/\text{Vs}$  at carrier concentrations  $\sim 5$  to  $8 \text{ cm}^{-3}$ , respectively. Thus, low growth rates during coalescence are beneficial for layer properties.

Next, the combined effect of lower V/III ratio and lower growth rate during the initial stages of growth is investigated. During coalescence the ammonia amount is varied from 1.5 down to 0.8 *slm* at a fixed TMGa flow as summarized in table 7.6. After the film has fully coalesced the growth parameters of HT-GaN are used.

The in situ measured reflectance transients at a wavelength of  $600 \text{ nm}$  for several growth runs with decreasing V/III ratio are presented in figure 7.16. After identical nucleation layer growth and annealing (first dotted line) gradually oscillations form which reach a constant intensity after complete two dimensional growth is observed (second dotted line). The coalescence is prolonged with decreasing V/III ratio. With reducing the V/III ratio

|                 | Coalescence<br>lower V/III          | HT-GaN                                  |
|-----------------|-------------------------------------|-----------------------------------------|
| Temperature     | 1080 °C                             | 1100 °C                                 |
| TMGa            | 12 <i>sccm</i>                      | 25 <i>sccm</i>                          |
| NH <sub>3</sub> | 1.5 ... 1.3... 1 ... 0.8 <i>slm</i> | 2.2 <i>slm</i>                          |
| V/III ratio     | 1276...1078...877...674             | 877                                     |
| Carrier gas     | N <sub>2</sub> /H <sub>2</sub>      | 85% N <sub>2</sub> / 15% H <sub>2</sub> |
| Total flow      | 9 <i>slm</i>                        | 9 <i>slm</i>                            |
| Thickness       |                                     | ~ 2.5 μm                                |

Table 7.6: Growth parameters for prolonged coalescence run in nitrogen ambient.

(from bottom to top) the coalescence time (and therefore the “coalescence thickness”) increases. Thus, applying a lower growth rate in combination with a reduced V/III ratio during the initial stages of growth results in an optimized coalescence growth rate, hence a thicker “coalescence layer”. This should lead to improved structural quality.

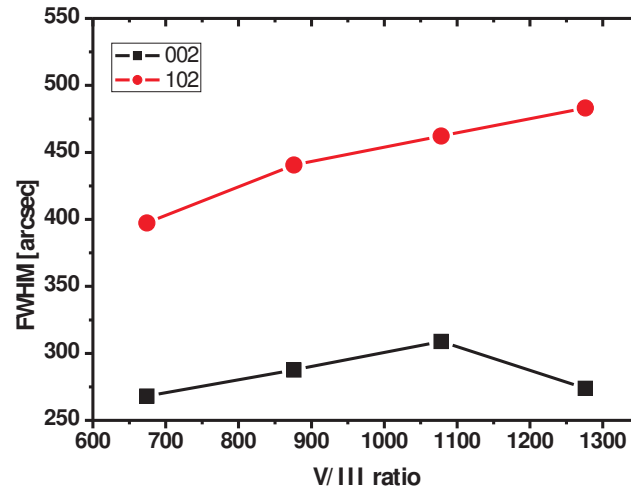


Figure 7.17: Symmetric (002) and asymmetric (102) reflections of GaN grown with decreasing V/III ratio during coalescence reveal a significant structural improvement.

XRD measurements of both symmetric (002) and asymmetric (102) reflections reveal the expected tendency (figure 7.17). While the (002) is rather insensitive to changes, the FWHM of the asymmetric peak decreases. Hence, both the electrical and structural data are significantly improved as summarized in table 7.7.

The last requirement as stated in the beginning of this chapter for further processing towards a HEMT structure is a smooth surface with low defect densities. Figure 7.18 displays the Nomarski images of the wafer surfaces. All wafers are specular and smooth. However, the longer the coalescence, with decreasing V/III ratio, the more wavy the surface gets on a micrometer scale. Thus there is a "natural end" to optimization. For one thing increased coalescence means a longer growth run period and for the other a too long coalescence can worsen the surface due to too large islands that will dominate the surface features. However, in our case we still find smooth and state of the art surfaces at acceptable growth run duration times. On a nanometer scale the wafers look quite comparable with parallel steps and so do the RMS values. Finally, we compare the "optimized GaN" surface to growth in hydrogen: Figure 7.19 demonstrates comparable step width in either case, however the "optimized GaN" surface tends to the well known round "cauliflower"-like structures. Also the RMS values for both surfaces are alike (0.26 and 0.35 nm).

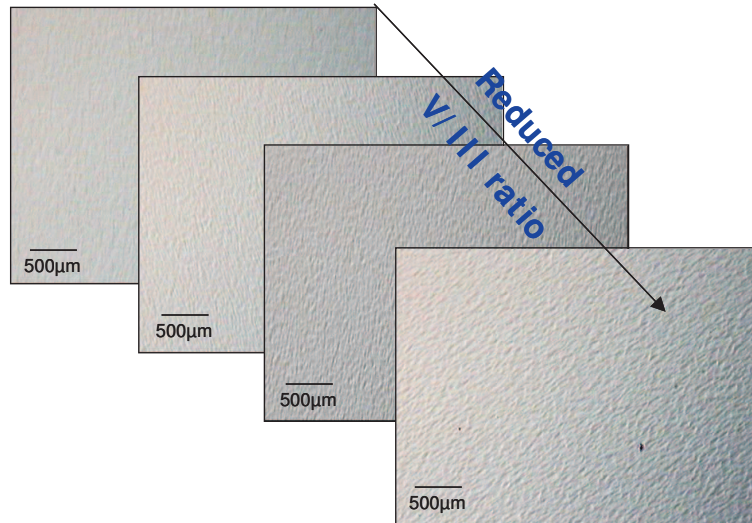


Figure 7.18: Nomarski images: The longer the coalescence the more structured the surface gets.

| V/III ratio                        | 1276               | 1078               | 877                | 674                |
|------------------------------------|--------------------|--------------------|--------------------|--------------------|
| Electr. Mob. [cm <sup>2</sup> /Vs] | 600                | 572                | 655                | 671                |
| Carrier Conc. [cm <sup>-3</sup> ]  | $6 \times 10^{16}$ | $6 \times 10^{16}$ | $7 \times 10^{16}$ | $9 \times 10^{16}$ |
| RMS [nm]                           | 0.23               | 0.34               | 0.27               | 0.35               |

Table 7.7: Improved electrical and structural data of the GaN film with lowering the V/III ratio, thus prolonging coalescence.

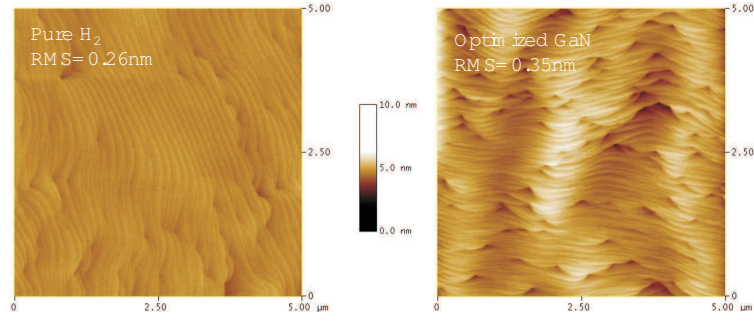


Figure 7.19: Comparison between the GaN grown in pure hydrogen to the finally fully "optimized GaN".

### 7.3.4 Conclusion

For the "inverted inlet geometry" a high nitrogen content in the gas phase is essential for improved thickness uniformity. A pure nitrogen ambient decreases the coalescence thickness and degrades crystal quality. However, it is not detrimental to the 2D growth phase. State of the art electrical and structural quality material as well as uniform layers with smooth surfaces are achieved by using only small amounts of hydrogen ( $\sim 15\%$ ) during the coalescence phase in otherwise nitrogen ambient. We managed to even increase the Hall mobility at room temperature up to  $671 \text{ cm}^2/\text{Vs}$  with a carrier concentration of  $9 \times 10^{16} \text{ cm}^{-3}$  and a FWHM for the (002) and (102) peaks of 268 and 397 *arcsec* respectively at a standard deviation in thickness of 3%. In this way, a high quality GaN template is produced. Further investigations need to be carried out to check the suitability as a buffer for HEMTs.



# Chapter 8

## HEMT growth

In chapter 7 an "optimized GaN" template was introduced with state of the art structural and electrical properties. In order to obtain devices of high power, the following parameters must be maximized:

1. Semi-insulating properties of the GaN to avoid any parallel conductivity to the channel layer and to ensure high HEMT performance at high frequency;
2. Tight alloy composition and thickness control in the heterostructure, yielding optimal AlGaIn/GaN heterojunctions;
3. Sheet conductivity  $\mu \times n_s$ , thus maximizing both the sheet charge density  $n_s$ , necessitating the maximization of the spontaneous and piezoelectric polarizations (PSE and PPE) with the Al mole fraction and the electron mobility  $\mu$ , requiring the minimization of scattering centers due to dislocations, interface roughness and alloy disordering at the heterojunction.

This chapter deals with the fulfillment of the final criteria. First of all AlGaIn growth on the "optimized GaN" layers is investigated and optimized and then first AlGaIn/GaN heterojunctions produced. Resistivity control of the GaN buffer is addressed next. Finally the resulting HEMT structures are characterized and evaluated according to the requirement list.

## 8.1 AlGa<sub>N</sub> Growth in the inverted inlet

### 8.1.1 Thickness and Alloy Composition control

The first  $Al_xGa_{1-x}N$  growth experiments are directed toward the control of AlGa<sub>N</sub> thickness and the alloy composition. Due to the strong tendency of trimethylaluminum (TMAI) to form adducts with ammonia, the AlGa<sub>N</sub> layers are typically deposited at a reduced reactor pressure of 50 *mbar*. At this pressure, pre-reactions between TMAI and  $NH_3$  are largely suppressed in our reactor. Starting point for the investigations were growth and flow conditions with which AlGa<sub>N</sub> is suitably deposited in the conventional inlet. AlGa<sub>N</sub> layers with a thickness of 1  $\mu m$  were grown on GaN at a constant surface temperature of 1075 °C corresponding to a control temperature of 1100°C. The total flow (7.5 *slm*) and ammonia flow rate ( $5.8 \times 10^{-2} mole/min$ ) were kept constant whereas the TMAI and TMGa flow rates were varied to perform the growth at different aluminum contents in the gas phase. AlGa<sub>N</sub> layers were deposited on top of the "optimized GaN" layers as described in the last chapter. AlGa<sub>N</sub> growth conditions are summarized in table 8.1.

|                  | AlGa <sub>N</sub> growth |
|------------------|--------------------------|
| Temperature      | 1100°C                   |
| TMGa             | 25...5 <i>sccm</i>       |
| TMAI             | 10...32 <i>sccm</i>      |
| NH <sub>3</sub>  | 1.3 <i>slm</i>           |
| Carrier gas      | 8% $N_2$ 92% $H_2$       |
| Total flow       | 7.5 <i>slm</i>           |
| Reactor pressure | 50 <i>mbar</i>           |
| Thickness        | $\sim 1 \mu m$           |

Table 8.1: Growth parameters for the AlGa<sub>N</sub> growth.

The AlGa<sub>N</sub> solid-vapor relationship and AlGa<sub>N</sub> growth rate are shown in figure 8.1. The dashed line in the left inset marks the ideal vapor solid relationship, where all gas phase Al is incorporated. The real incorporation stays below this line, although, with increasing aluminum in the gas phase the solid content increases linearly, as determined by XRD and RBS. Accountable for the difference between real incorporation and the dashed line are loss mechanisms such as adduct formation. However, the aluminum incorporation efficiency is fairly high up to the Al content of about 60 %. The left inset displays the growth rate dependency on the gas phase aluminum. Here we observe a decrease (the dashed line represents an exponential fit) of the growth rate with increasing aluminum in the gas phase. Thus further increase of aluminum incorporation is limited by lower growth rates and enhanced Al losses.

For higher Al compositions from 70 to 100 %, the Al-containing species partial pressures

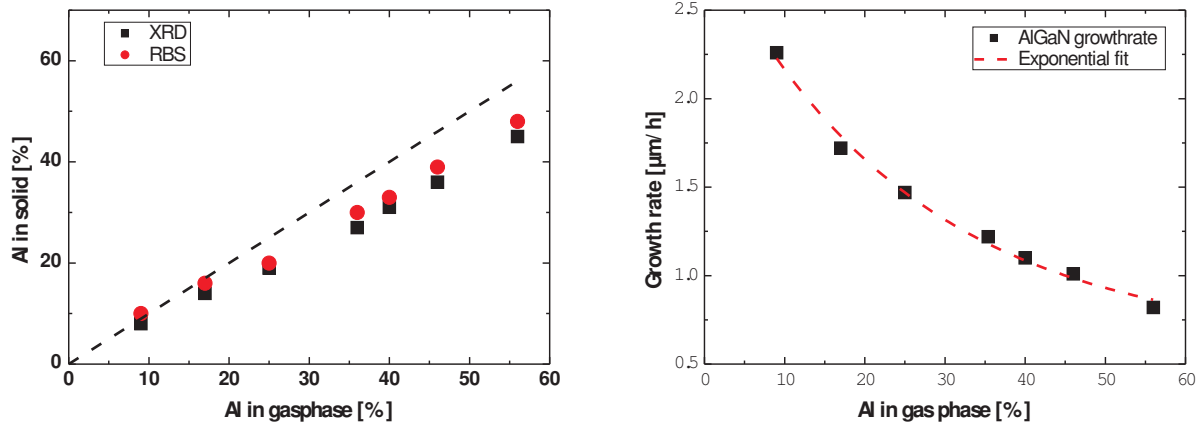


Figure 8.1: Al solid-vapor relationship (left) and AlGaIn growth rate for various Al gas phase concentrations (right). The dashed line in the left inset marks the ideal vapor solid relationship, whereas boxes and dots represent measurement results from XRD and RBS respectively. The growth rate (right) decreases with increasing aluminum in the gas phase.

and gas residence time is lowered to suppress the formation of particles via increasing the total flow in the reactor up to 10 *slm* and the total amount of Al precursor heightened (up to 98 *sccm*). For this growth regime additional process optimization is necessary to find the optimum precursor flow parameters in order to achieve efficient growth.

Figure 8.2 demonstrates the alloy composition distribution for several runs at different positions over the wafer in the inverted inlet configuration. Especially for the device relevant range around 30% of Al the absolute deviation over the wafer is *less* than one percent. Just the minimum and maximum aluminum containing wafers differ slightly above one percent in their composition.

Since our GaN layers are usually grown with a high nitrogen content (85% to 100%) and at a total flow of 9 *slm* it seems beneficial *not* to change the carrier gas composition or the total flow *abruptly* when growing the AlGaIn layer. Thus, the carrier gas dependent Al incorporation at 9 *slm* with an intended Al-content of 30% was investigated at conditions used for the GaN buffer except for the pressure. Growth parameters are displayed in table 8.2.

The growth rate in nitrogen is about 30% lower (absolute growth rate: 0.75  $\mu\text{m}/\text{h}$ ) than in hydrogen (1.1  $\mu\text{m}/\text{h}$ ). Furthermore, the Al incorporation is slightly more efficient in nitrogen than in hydrogen (33 % instead of 31 %) at otherwise identical TMAI and TMGa flows. However, the alloy uniformity over the wafer is equally good ( $< 0.5\%$

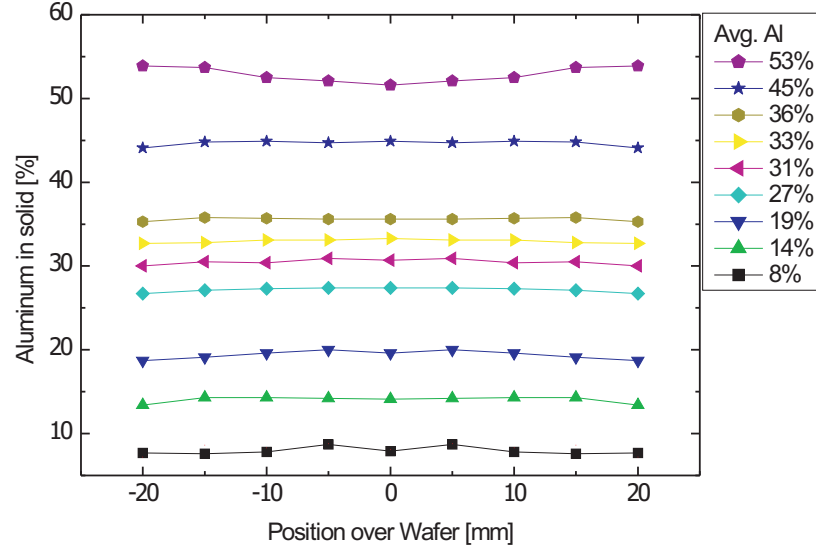


Figure 8.2: Alloy composition distribution for several runs at different positions over the wafer in the inverted inlet configuration.

|                                            | AlGaIn growth  |
|--------------------------------------------|----------------|
| TMGa                                       | 8 <i>sccm</i>  |
| TMAI                                       | 27 <i>sccm</i> |
| NH <sub>3</sub>                            | 1.3 <i>slm</i> |
| Carrier gas N <sub>2</sub> /H <sub>2</sub> | 8/92 or 85/15  |
| Total flow                                 | 9 <i>slm</i>   |

Table 8.2: Growth parameters for AlGaIn growth at 9 *slm* in two commonly applied carrier gas mixtures

absolute deviation), independent on the carrier gas. Whether there is an effect of the carrier gas on the transport properties (e.g. interface roughness) in the HEMT structure is investigated in subsection 8.1.2.

Thus far, thick AlGaIn layers of about 1  $\mu\text{m}$  were investigated to understand AlGaIn deposition behavior and to estimate alloy compositions. Next, device relevant thicknesses around 30 *nm* and AlGaIn compositions of around 30% Al are considered.

### 8.1.2 Thin AlGa<sub>N</sub>/Ga<sub>N</sub> layers

The first AlGa<sub>N</sub>/Ga<sub>N</sub> heterojunctions are grown on the "optimized Ga<sub>N</sub>" layer as described in the last chapter. Following device structure considerations in chapter 2 the layer stack is intended to consist of a 2  $\mu\text{m}$  Ga<sub>N</sub> base, an approximately 30  $\text{nm}$  thick  $\text{Al}_{0.3}\text{Ga}_{0.7}\text{N}$  layer with a 5  $\text{nm}$  Ga<sub>N</sub> cap. Within this structure the interface roughness is of primal importance for the transport properties of the HEMT, especially for high density 2DEGs. Higher deposition temperatures enhance the surface morphology, although in turn increase nitrogen desorption from the surface and change adduct formation in the gas phase. Also interface abruptness may be influenced detrimentally. In the last section thick AlGa<sub>N</sub> layers at a control temperature of 1100°C were analyzed, here we turn to thin layers and study the influence of temperature both on the layer stack and the aluminum content in the solid.

#### Temperature influence

| AlGa <sub>N</sub> growth in | hydrogen<br>(8% $\text{N}_2$ 92% $\text{H}_2$ ) | nitrogen<br>(85% $\text{N}_2$ 15% $\text{H}_2$ ) |
|-----------------------------|-------------------------------------------------|--------------------------------------------------|
| Temperature                 | 1100...1130 °C                                  | 1100...1130 °C                                   |
| TMGa                        | 8                                               | 9 <i>sccm</i>                                    |
| TMAI                        | 27                                              | 26 <i>sccm</i>                                   |
| NH <sub>3</sub>             | 1.3 <i>slm</i>                                  | 1.3 <i>slm</i>                                   |
| Thickness                   | ~ 30 <i>nm</i>                                  | ~ 30 <i>nm</i>                                   |

Table 8.3: Growth parameters for *thin* AlGa<sub>N</sub> growth at 9 *slm* in two commonly applied carrier gas mixtures

We studied the influence of growth temperature on the AlGa<sub>N</sub> layer, i.e. thickness and composition, in both hydrogen and nitrogen atmosphere. The temperature ranges from 1100 to 1130 °C. The basic parameters are summarized in table 8.3. Note, that the TMAI and TMGa flows in nitrogen ambient are slightly lowered, since thick layers exhibit a more efficient Al incorporation for nitrogen than hydrogen ambient (refer to subsection 8.1.1). Thickness and composition are determined by RBS.

The heterostructure stacks grown on "optimized Ga<sub>N</sub>", displayed in figure 8.3, exhibit almost identical features for each series: Structure, thickness and composition are temperature independent, however, different for hydrogen and nitrogen. In hydrogen the stack sequence consists of a 2  $\text{nm}$  Ga<sub>N</sub> cap and a 29  $\text{nm}$   $\text{Al}_{0.38}\text{Ga}_{0.62}\text{N}$  layer. In nitrogen we find 5  $\text{nm}$  Ga<sub>N</sub> cap and a 32  $\text{nm}$   $\text{Al}_{0.33}\text{Ga}_{0.67}\text{N}$  layer. Growth parameters for both nitrogen and hydrogen AlGa<sub>N</sub> layers are calculated from the discussed thick films. However, the AlGa<sub>N</sub> layer grown in hydrogen shows a distinctively higher aluminum percentage than

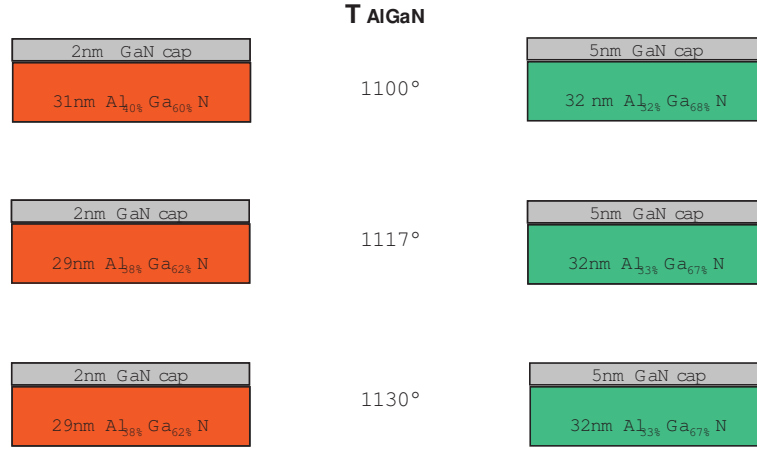


Figure 8.3: Heterostructure stacks grown on "optimized GaN" in hydrogen (left) or nitrogen (right) ambient. Structure, thickness and composition are temperature independent, however, different for hydrogen and nitrogen.

the thick layer. The reason for the higher Al content in hydrogen can only be speculated upon. It maybe related to the small thickness of the layer and thus to the start conditions of growth. Even in nitrogen the aluminum content is rather high although the precursor flows are already reduced in comparison to the thick layer.

AFM micrographs (figure 8.4) reveal similar surfaces with parallel steps in either case. Even growth in nitrogen demonstrates identical surface morphologies with samples in hydrogen. This might be due to the small thickness of the AlGaIn layers. The best RMS values of the  $15 \times 15 \mu m$  sized samples are achieved at a temperature of 1117 and 1100°C for hydrogen and nitrogen respectively. The RMS value range is quite comparable for both series from  $0.5 nm$  to  $\sim 0.8 nm$ . There is no clear temperature dependent trend.

Both series are electrically characterized by Hall measurements at room and liquid nitrogen temperature. Figure 8.5 exhibits the sheet carrier concentration for the AlGaIn temperature series in hydrogen (left) and nitrogen (right). In general, the nitrogen samples exhibit less free carriers ( $\sim 9 \times 10^{12} cm^{-2}$  at room temperature) in comparison to the hydrogen ones ( $\sim 1.2 \times 10^{13} cm^{-2}$  at room temperature) since the Al-content is lower. Hence, a lesser piezoelectric polarization is found. In addition it is found that within each series the sheet carrier concentration drops considerably at  $77K$ . Since these samples are grown on the "optimized GaN" layer with reduced dislocation density, residual donors -uncompensated by edge type dislocations- contribute to the carrier concentration at room temperature. This additional conductivity freezes out at low temperatures. In a device structure we would find considerable parallel conduction effects that will lead to unsatisfactory device pinch off.

Neglecting the inadequacy for devices the sheet conductivity ( $\mu \times n_s$ ), as seen in figure 8.6,

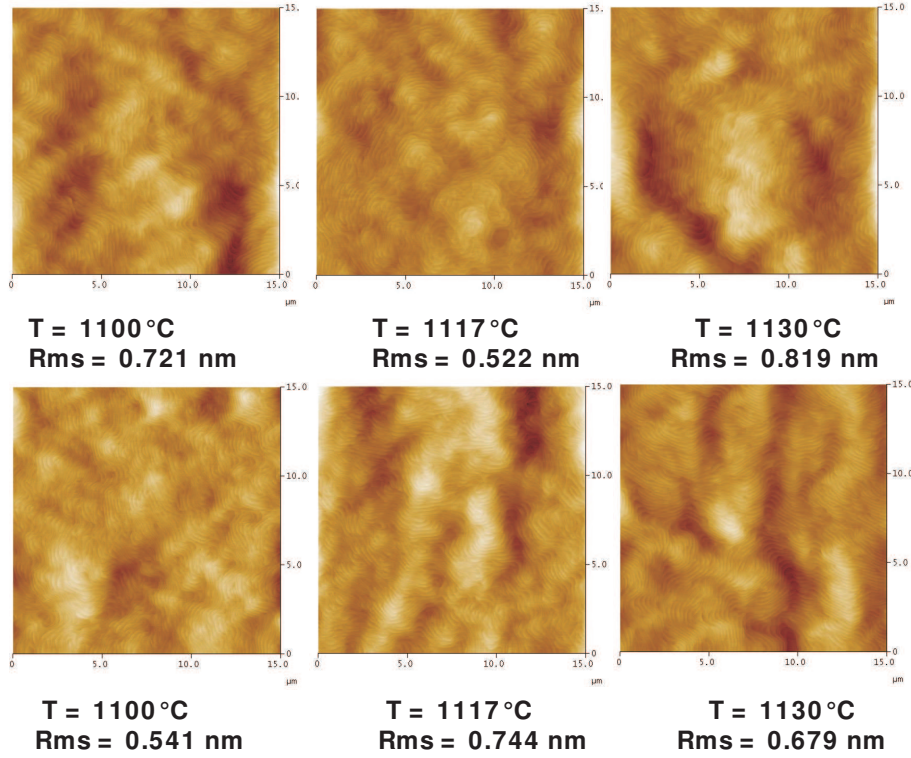


Figure 8.4: AFM images of AlGaIn layers grown in hydrogen (upper row) or nitrogen atmosphere (lower row).

indicates similar and state of the art values ( $> 1.5 \times 10^{16} V^{-1} s^{-1}$ , with  $\mu > 1400 cm^2/Vs$ ) for the growth series in hydrogen. In nitrogen, the sheet conductivity rises as a function of the deposition temperature (with  $\mu$  from 726 to  $1492 cm^2/Vs$ ), thus a further increase seems beneficial. The best respective hall mobilities are found for the samples grown at 1117 and  $1130^\circ C$  grown in hydrogen and nitrogen respectively. Mobilities of 1440 and  $1492 cm^2/Vs$  are measured at carrier concentrations of  $1.33 \times 10^{13} cm^{-2}$  and  $9.4 \times 10^{12} cm^{-2}$  for hydrogen and nitrogen respectively, resulting in sheet conductivity values of  $1.92$  and  $1.4 \times 10^{16} V^{-1} s^{-1}$  for hydrogen and nitrogen respectively. These results suggest that there is an optimum growth temperature for either atmosphere.

For the following discussion we will focus on AlGaIn grown in hydrogen ambient.

### 8.1.3 Conclusion

The inverted inlet combines the reproducibility of the growth process with a good growth control for  $Al_xGa_{1-x}N$ . Due to the high incorporation efficiency a minimal amount of adduct formation occurs. Tight alloy composition and thickness control is achieved both

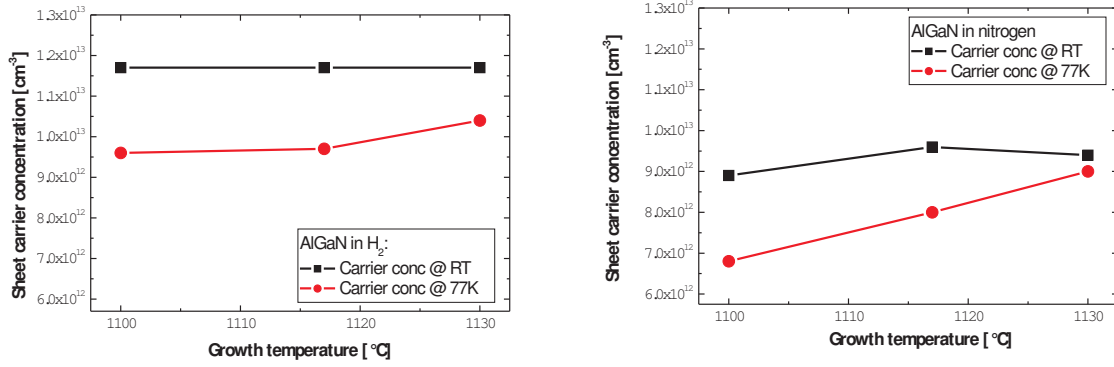


Figure 8.5: Sheet carrier concentration for the AlGaIn temperature series in hydrogen (left) and nitrogen (right) measured at room and liquid nitrogen temperature.

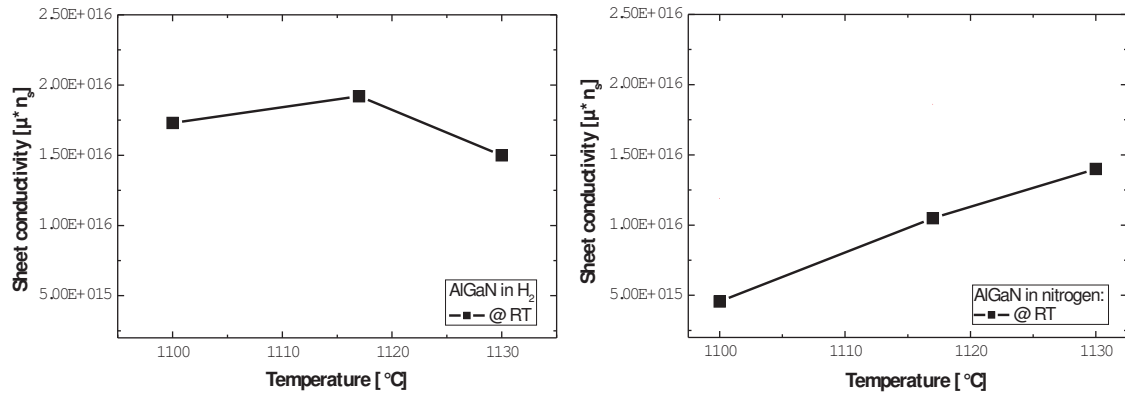


Figure 8.6: Sheet conductivity ( $\mu \times n_s$ ) values for each series (hydrogen left and nitrogen right).

for hydrogen and nitrogen as carrier gas. HEMT device relevant AlGaIn/GaN heterostructures were grown on the "optimized GaN". Despite state of the art sheet conductivity values the heterojunctions exhibit additional conductivity due to residual donors in the "optimized GaN" layer. In a device structure we would find considerable parallel conduction effects that will lead to unsatisfactory device pinch off.

Therefore resistance control is addressed next to achieve high quality HEMT devices.

## 8.2 Highly resistive GaN buffer

Undoped MOVPE grown GaN usually shows n-type conductivity. In the past, the n-type conductivity of GaN layers had been mainly attributed to the formation of nitrogen vacancies in the crystal. More detailed investigations, though, suggested that oxygen impurities, rather than nitrogen vacancies, are the major source of residual electrons [Kel95, Nak92]. Oxygen, if incorporated on the nitrogen site is a shallow donor in GaN. Water and oxygen are omnipresent impurities in the system. Both can either be present as impurities in the metal-organic precursors and gases or enter the system through leaks in the MOVPE system or during wafer loading. Another potential source is the sapphire substrate, since water forms during the sapphire ( $Al_2O_3$ ) pretreatment in hydrogen [Kel01].

AlGaIn/GaN HEMTs require a semi-insulating GaN buffer beneath the device structure to enable sharp current pinch off and low scatter in active device regions for high mobility, thus in semi-insulating material residual donors have to be compensated. Typical values of resistivity for undoped GaN layers, as reported in literature, range from 0.5 to  $10^{11} \Omega cm$  [Mul05]. In order to suppress current leakage and RF losses a buffer resistivity of at least  $10^2 \Omega cm$  (in our geometry corresponding to  $1 M\Omega/\square$ ) is intended.

### 8.2.1 Strategy for Semi-Insulating GaN

In the following discussion the previously obtained "optimized GaN layer" (chapter 7) is tested for its fulfilment of the resistivity requirements. The sheet resistance of the sample, as determined by contactless conductivity measurement with a Leighton LEI Eddy Current system, exhibits a value of  $3000 \Omega/\square$  ( $= 0.5 \Omega cm$ ). In this way, the "optimized GaN" layer does not meet the aforementioned requirements. Thus, the question arises how to control the resistivity of GaN.

To grow semi-insulating material the residual donors either have to be avoided or compensated by acceptor states, commonly done either by introducing high densities of threading dislocations, or –instead of extended defects– introducing point defects by choosing growth conditions that result in high levels of carbon impurities [Ogi80, Hwa97]. Recently a third possibility that renders GaN semi-insulating has been investigated: High concentrations of deep and shallow level acceptor states are introduced by doping GaN with Fe, Cr and Zn [Hei02, Ois03].

Summarizing there are three approaches:

- Choosing growth conditions that result in low levels of donor-impurity incorporation (mainly oxygen) in the lattice. However growth conditions that favor low oxygen levels, for example high growth pressures [Wic04], also decrease the number

of acceptor-like states such as dislocations and carbon impurities. Thus, residual donors still influence the layer resistivity.

- Changing growth conditions to introduce high densities of dislocations, potentially associated with undesirable effects on the surface morphology and consequently the interface roughness of the heterostructure [Hey99] and, moreover, the transport properties (dislocation scattering, refer to chapter 2).
- Incorporate transition metals, associated with memory effects on MOVPE growth, thus contaminating the reactor and subsequently grown layers. Moreover literature [Hei02] often reports that considerably higher levels of dopants ( $\sim 10^{19}$ ) were necessary to compensate the background carriers ( $\sim 10^{16}$ ).

In the discussion below we will solely consider growth approaches based on the first two items. Hence, our growth procedure and parameters aim at the avoidance of high impurity levels and the compensation of residual donors by dislocation related mechanisms. In chapter 7 the influence of nitrogen on the coalescence phase was investigated and it was found that with decreasing coalescence time electrically active edge type threading dislocations arise. Thus we have a tool at hand that allows the control of dislocation types and densities. Moreover, an early coalesced GaN layer avoids the migration of oxygen from the substrate into the bulk GaN [Wic04].

### Tuning the Insulation Level of "Optimized GaN"

Starting point for our resistivity improvements is the "optimized GaN" layer. Table 8.4 summarizes the growth conditions of the two-step-growth run. Nucleation layer growth and annealing take place in hydrogen atmosphere as found in table 7.1 in chapter 7. A coalescence growth (step 1) with lower precursor flows and lower V/III ratio is followed by the high temperature deposition (HT-GaN) with higher source flows and V/III ratio (step 2). In a second deposition run the step 1 is excluded from the growth run-recipe in order to shorten the coalescence.

The selective introduction of dislocations has been suggested to increase the resistance of the layer. From chapter 7 we know that a smaller "coalescence thickness" yields a higher density of edge type dislocations which are electrically active, acting as acceptor like traps in the layer [Grz05]. Figure 8.7 shows the transients of the two growth runs. Namely the "optimized GaN", with the two step growth, in comparison to the one step growth run. With a shorter coalescence we succeeded in increasing the buffer sheet resistance as summarized in table 8.5.

The sheet resistance for the one step growth run is twofold higher than for the two step growth run, however still not high enough to meet the  $10^2 \Omega \text{cm}$  criterion. Accompanied

|                 | Coalescence<br>"step 1" | HT-GaN<br>"step 2"  |
|-----------------|-------------------------|---------------------|
| Temperature     | 1080 °C                 | 1100 °C             |
| TMGa            | 12 <i>sccm</i>          | 25 <i>sccm</i>      |
| NH <sub>3</sub> | 0.8 <i>slm</i>          | 2.2 <i>slm</i>      |
| V/III ratio     | 674                     | 877                 |
| Total flow      | 9 <i>slm</i>            | 9 <i>slm</i>        |
| Thickness       |                         | ~ 2.5 $\mu\text{m}$ |

Table 8.4: Growth parameters for the "optimized GaN" template.

| Sample       | Sheet resistance [ $\Omega/\square$ ] | XRD FWHM [ $\text{arcsec}$ ] |
|--------------|---------------------------------------|------------------------------|
| <b>2step</b> | 3000<br>(~ 0.5 $\Omega\text{cm}$ )    | (002)= 268<br>(102)= 397     |
| <b>1step</b> | 6000<br>(~ 1 $\Omega\text{cm}$ )      | (002)= 319<br>(102)= 569     |

Table 8.5: Sheet resistance and structural quality of the "optimized GaN" layer (2step) versus a non optimized growth run in 75% nitrogen (1step).

with the sheet resistance is an increase in the FWHM of the (002) and (102) peaks, thus we find an increase in resistivity at the expense of structural quality.

### 8.2.2 Influence of Carrier Gas on Resistance

Control of dislocation types and densities is essential for obtaining highly resistive layers. As demonstrated in chapter 7 the usage of different carrier gas compositions allows control of the morphology development and hence dislocation evolution. Therefore *undoped* GaN layers with different nitrogen fractions were grown (similar to chapter 7) as summarized in table 8.6 and their sheet resistance determined, as seen in figure 8.8.

As expected, the sheet resistance rises as a function of the nitrogen amount in the gas phase, therefore as a function of the dislocation density. Above a certain nitrogen fraction (~ 80%) the resistance rises steeply and the sample grown in 100% nitrogen is not even measurable by the Leighton LEI Eddy Current System. Note, that the sheet resistance values are lower in comparison to those in table 8.5, but due to the fact that the layer thickness is twofold higher (4 instead of 2  $\mu\text{m}$ ) the resistivity of both series is quite comparable  $\leq 1\Omega\text{cm}$ . With ohmic contacts (Ti/Al/Ni/Au) to the nitrogen sample, with a contact resistance below  $1\Omega\text{mm}$ , I-V measurements at 5V in-between two contacts, give a sheet resistance of about  $100\text{ M}\Omega/\square$  corresponding to a resistivity of  $3 \times 10^4\Omega\text{cm}$ . Thus, solely GaN growth in pure nitrogen ambient fulfils the primary resistance requirements.

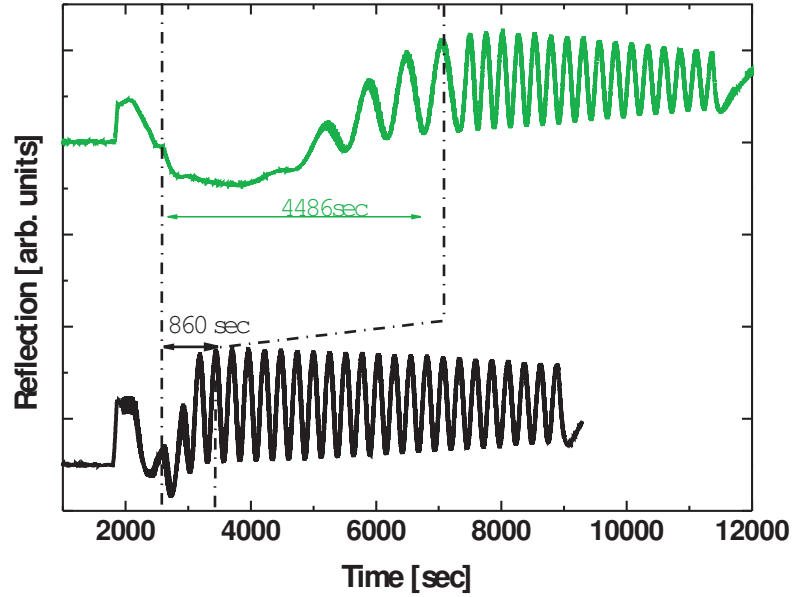


Figure 8.7: Comparison of two growth runs: The "optimized GaN" with the two step HT growth, consisting of the low growth rate and V/III ratio during coalescence and the higher growth rate and V/III ratio during 2D growth ("2step"), in comparison with GaN grown in 75% nitrogen ambient ("1step").

| HT-GaN          |                         |
|-----------------|-------------------------|
| Temperature     | 1100°C                  |
| TMGa            | 25 <i>sccm</i>          |
| NH <sub>3</sub> | 2.2 <i>slm</i>          |
| V/III ratio     | 877                     |
| Carrier gas     | 0...100% N <sub>2</sub> |
| Total flow      | 9 <i>slm</i>            |
| Thickness       | ≥ 4 μm                  |

Table 8.6: Parameters for *undoped* GaN growth with increasing nitrogen amount in the gas phase

### Comparison: "optimized GaN" vs. "N<sub>2</sub> GaN"

The question arises whether the layers grown in pure nitrogen atmosphere show a 2DEG and if the sheet conductance will suffer from the high dislocation densities in comparison

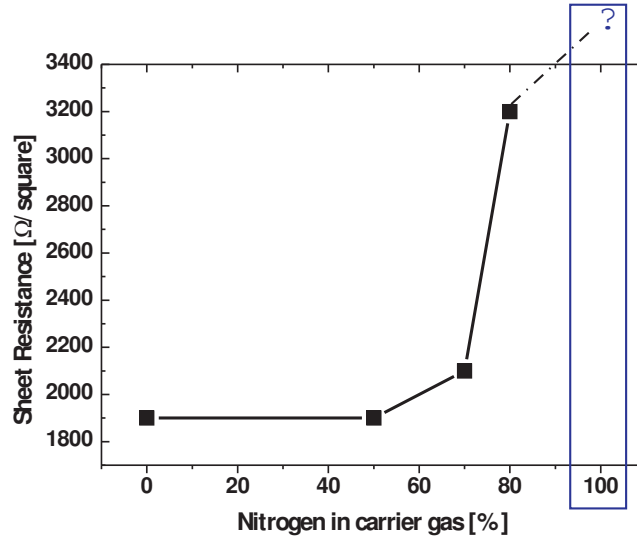


Figure 8.8: Sheet resistance as a function of nitrogen percentage in the carrier gas.

to AlGaN growth on top of the optimized GaN. First of all the surface on a nanometer scale is considered.

Figure 8.9 presents the AFM images. Both surface morphology and surface roughness differ. Where straight and parallel steps are observed for the heterostructure on "optimized GaN", the AlGaN on top of the nitrogen GaN reveals the familiar cauliflower like morphology. Thus, both (hydrogen grown) AlGaN layers adapt to the underlying GaN. Also the surface roughness increases from 0.52 to 0.74 nm for a  $15 \times 15 \mu m$  sized sample. Even though there is a distinct roughness difference, the channel conductivity of the HEMT structure using "nitrogen GaN" is still very high. An electron mobility at room temperature of  $1490 cm^2/Vs$  at sheet carrier concentrations of  $9.88 \times 10^{12} cm^{-2}$  demonstrates the electrical quality of HEMT structures grown on "nitrogen GaN". Nitrogen samples combine the advantage of tunable high resistivity with state of the art electronic properties.

Thus, the initially posed question whether the high numbers of dislocations are detrimental for the electrical properties can be denied. At least our heterojunctions are not yet sensitive to these effects, therefore we need to turn to HEMT device characterization (section 8.3) to give a final prove of the feasibility of our growth approach.

In chapter 7 the drawback of the epitaxial runs carried out in nitrogen atmosphere was already discussed. The surface exhibits hexagonal structures and "turrets" thus further processing becomes rather challenging. For instance pits or 3D structures on the surface complicate the necessary metallization steps due to inadequate sticking of the metal on

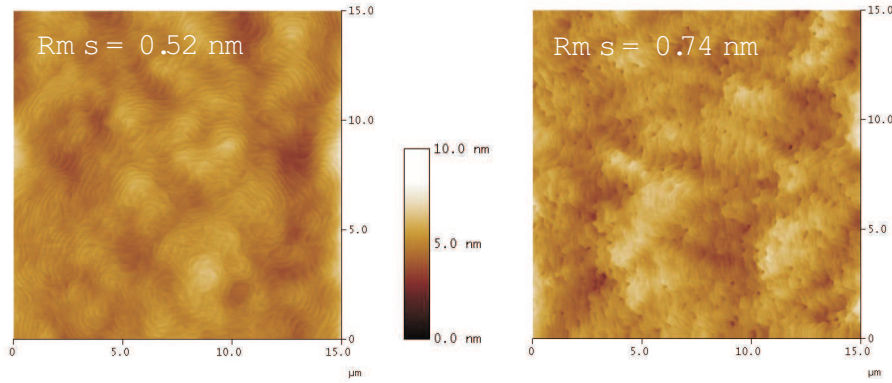


Figure 8.9: AFM images of the AlGaIn/GaN heterostructure on "optimized GaN" (left) and " $N_2$  GaN" (right).

the surface and therefore electrical characterization does not make sense. A strategy has to be developed which allows for resistance control on the one hand and an appropriate surface on the other. The morphology development using nitrogen strongly influences the structural and therefore electrical characteristics by changing the density and size of nucleation islands. This in turn can also influence the surface morphology and thus the quality of ohmic and Schottky contacts. In this way parameters will be investigated that promote early two dimensional growth. First of all the influence of the temperature in the HT-growth step is studied, since it enhances the adatom mobility on the surface and therefore should be beneficial for the surface morphology.

### Influence of Temperature on Resistivity and Morphology

Starting point for our considerations is growth of GaN in pure nitrogen with deposition parameters as summarized in table 8.6. The surface temperature in the HT-step is gradually varied run by run from 1095 to 1140°C. On top of the HT-GaN a 27 nm  $Al_{0.35}Ga_{0.65}N$  and a 5 nm GaN cap is deposited in order to directly test device functionality.

Figure 8.10 presents the Nomarski images of the surface. With increasing growth temperature the formation of bigger defects such as turrets accelerates. Temperatures beyond 1130°C result in hexagonal defects and pits on the surface.

Figure 8.11 demonstrates the ohmic characterization procedure for these samples. After the heterostructure is deposited the AlGaIn layer is etched through to the bare GaN by reactive ion etching (RIE). Then ohmic contacts are applied in order to solely measure the resistivity of the bulk GaN material. In this way, we grow the whole device structure but concurrently are able to measure the GaN characteristics.

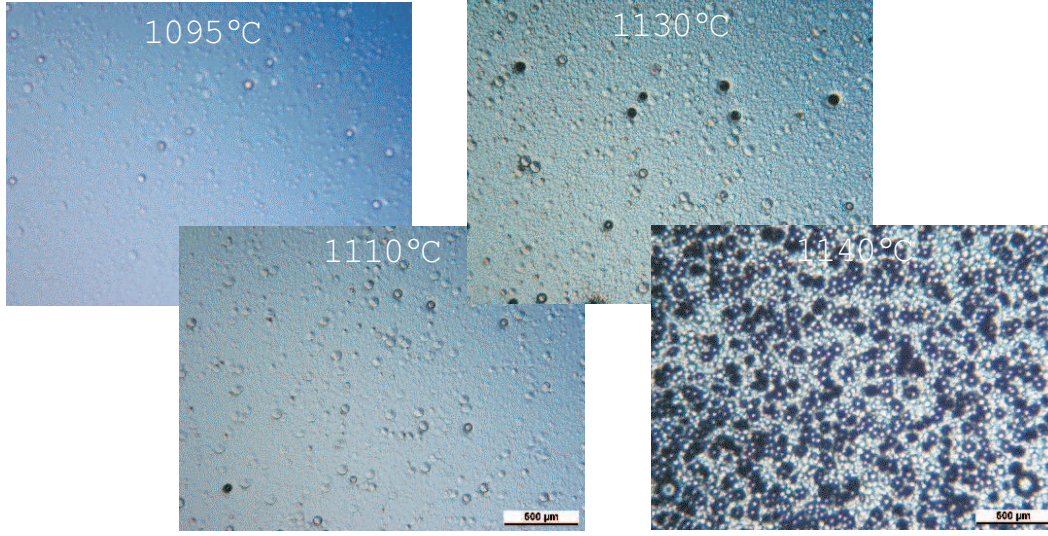


Figure 8.10: With increasing HT-step growth temperature the formation of bigger defects such as turrets accelerates. Surface temperatures beyond  $1130^{\circ}\text{C}$  result in hexagonal defects and pits on the surface.

Sheet resistance measurements of these layers require ohmic contacts, thus further processing steps become necessary, such as optical lithography to define the contact pads and different metallization steps for the contact itself. This effort is solely justified for continuous and relatively smooth surfaces. Due to the significant surface deterioration only the two lowest temperature samples are suggestive for further processing. For these samples the sheet resistance increases by one order of magnitude from  $1 \times 10^8$  to above  $1 \times 10^9 \Omega/\square$  and the resistivity from  $3 \times 10^4$  to  $3 \times 10^5 \Omega\text{cm}$ . The higher growth temperature additionally facilitates lateral growth [Kal03], promoting dislocations and point defects, which are the dominant structural feature responsible for the resistivity variation seen in the films. In terms of a reasonable compromise between surface quality and resistivity

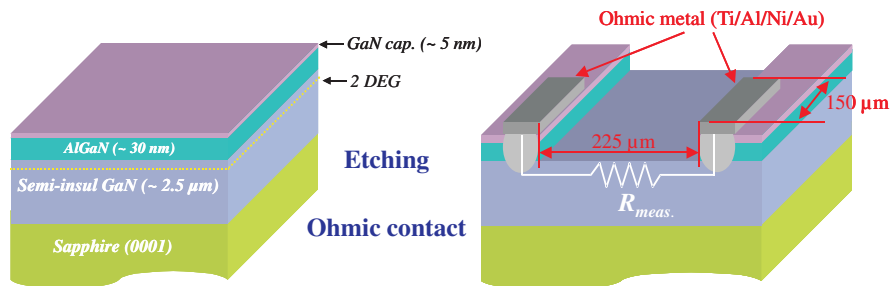


Figure 8.11: Resistance characterization procedure for the anneal time series. By locally etching the AlGaIn layer solely the GaN buffer is characterized.

the best morphology with a high insulating buffer of  $10^4 \Omega \text{cm}$  was achieved at  $1095^\circ \text{C}$ .

The insulation level should be increased, preferably not at the expense of surface quality. Therefore another approach to highly resistive GaN is investigated next: The annealing time influence on morphology development and the surface features is tested.

### Influence of Anneal Time on Resistivity and Morphology

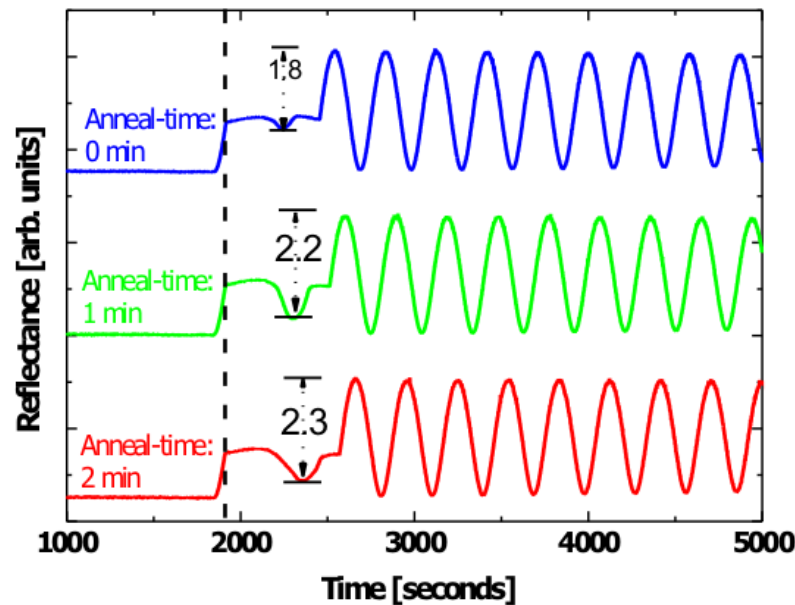


Figure 8.12: Reflectance transients for several growth runs with different annealing times. Values are given for the reflectance differences between oscillation minimum and maximum.

Another method to influence the distribution and size of nuclei is to tune the anneal time, thus promoting an early onset of two dimensional growth. During annealing the temperature is ramped, intentionally slow, from nucleation temperature to slightly below HT growth conditions, where it is fixed for a given period of time. During this recrystallization process some of the material decomposes and evaporates, resulting in an arbitrary distribution of islands. The most important parameter here is the annealing time. An extended annealing time will promote the reevaporation of GaN and etch the layer, decreasing the density of islands. Consequently, a shorter annealing time has the contrary effect. More small islands will cover the surface and therefore more dislocations arise when

growth is resumed. The normal annealing procedure consists of a 3 *min* recrystallization time at 970 °C followed by the ramping to HT growth conditions which takes another two minutes until all flows are adjusted.

In the last subsection it was shown that the sample grown at 1095°C demonstrates the comparably best surface in that temperature series, but also lower resistance values. Thus, for the investigation of the annealing time influence on morphology and resistivity we start with the sample with the best yet achieved resistance, which was grown at a temperature of 1110 °C, and modify the surface by shortening the anneal time from initially two minutes at 970°C stepwise (2,1,0) down to 0 minutes. After ramping, the flow conditions are stabilized in this series for an additional 2 minutes. On top of the HT-GaN a 27 nm  $Al_{0.35}Ga_{0.65}N$  and a 5 nm GaN cap is deposited in order to directly test device functionality.

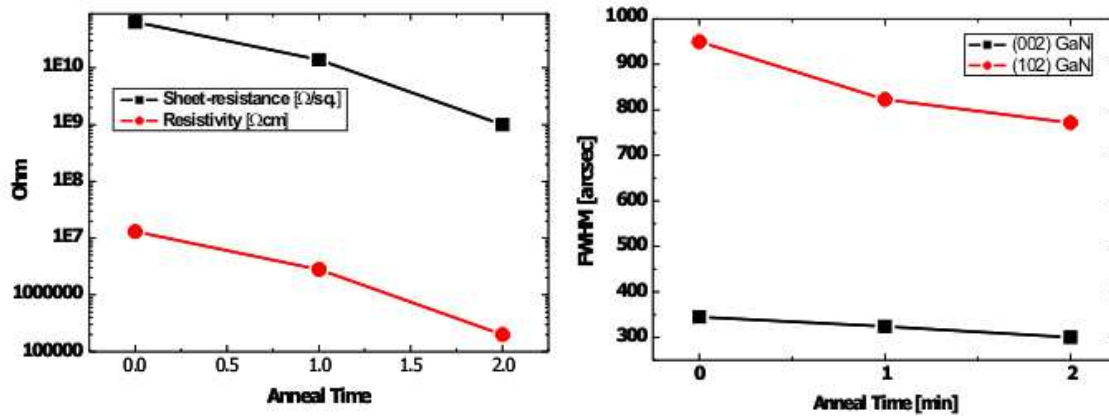


Figure 8.13: Broad increase in sheet resistance and resistivity over two orders of magnitude (left). Associated with the resistance is the rising FWHM value of the asymmetric (102) peak (right)

The reflectance transients for several growth runs with different annealing times are presented in figure 8.12. The low temperature growth step, as well as the annealing step, use hydrogen as carrier gas. The beginning of the annealing step is denoted by the dotted line. During the annealing the decrease in reflectivity is caused both by film etching and scattering effects which are due to film roughening as islands appear. Thus the following reflectance minimum is a qualitative measure of surface roughness, hence a measure of 3D island density. In the graph values are given for the reflectance differences between oscillation minimum and maximum, qualitatively describing the surface roughness. Thus the lower this number, the smoother the surface due to more islands and hence we expect to find a higher resistivity in the layer.

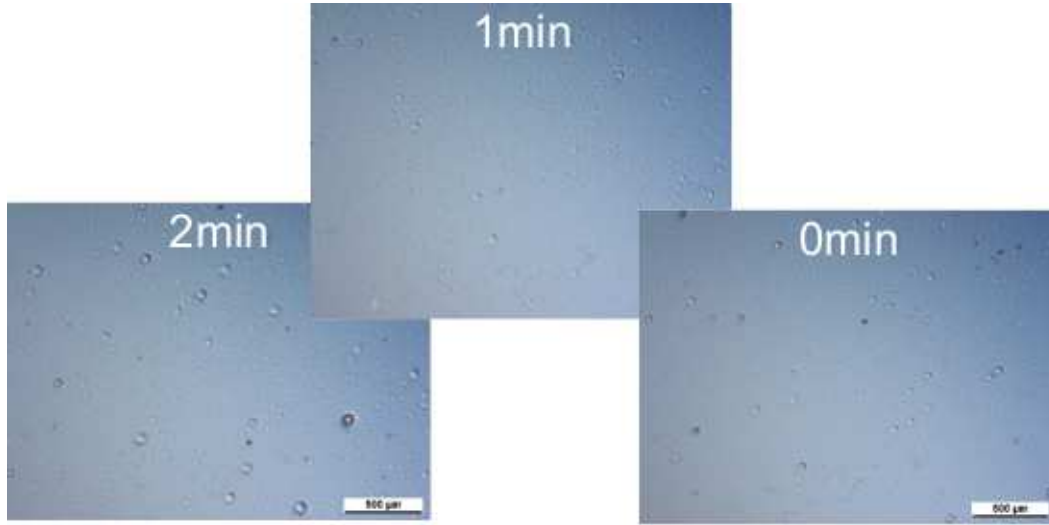


Figure 8.14: Nomarski images of the annealing series. The morphology on the micrometer scale smoothes with decreasing anneal time and also the density of defects clearly diminishes.

Determining the resistance values with ohmic contacts demonstrates a broad increase in resistivity from  $2 \times 10^5$  to  $1 \times 10^7 \Omega cm$  (figure 8.13 (left)). The lower the initial roughness (2.3 to 1.8 arb. units), the higher the resistance. The right inset of figure 8.13 demonstrates the (002) and (102) peak FWHM values. As expected, associated with the resistance is the rising FWHM value of the asymmetric (102) peak as demonstrated in figure 8.13 (right). According to the model proposed by Weimann et al. [Wei98], edge dislocations work as acceptor-like trap levels. Cho et al. [Cho01] experimentally confirmed this model using deep-level transient spectroscopy measurements. Simpkins et al. [Sim03] identified edge dislocation as negatively charged and not highly conductive.

Initially, the major obstacle hindering device efforts were the surface features. Nomarski microscopy is used to evaluate the surface morphology of the anneal series, as seen in figure 8.14. The morphology on the micrometer scale smoothes with decreasing anneal time and also the density of defects clearly diminishes. The best surface is achieved at one or zero minutes. Further processing now makes sense. On the nanometer scale (figure 8.15), the surface morphology is quite similar to the earlier grown  $N_2$  samples (Figure 7.8). Round steps and pits are found. To the eye it seems as if the surface becomes even more cauliflower like with decreasing anneal times. More round steps are found. In terms of surface roughness the sample with one minute annealing shows the lowest RMS value of  $0.82 nm$ . However all other samples have distinctly rougher surfaces with RMS values around  $1.3 nm$ <sup>1</sup>. This average roughness is significantly higher than for the sample grown

<sup>1</sup>In comparison to the micrographs from figure 7.8 the lateral scale differs (here:  $15 \times 15 \mu m$ ) to elucidate the influence of the shorter anneal times

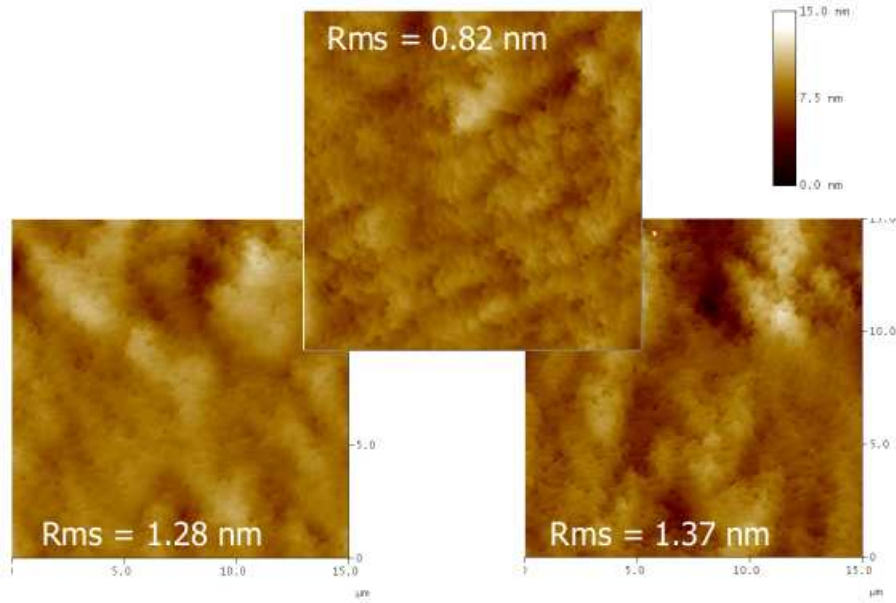


Figure 8.15: AFM images of the anneal series. Anneal times decrease from left (2 min) to right (0 min). The surface morphology is quite similar to the earlier grown  $N_2$  samples (Figure 7.8). Round steps and pits are found. It seems as if the surface becomes even more cauliflower like with decreasing anneal times.

at a surface temperature  $1095\text{ }^{\circ}\text{C}$  with an RMS of  $0.72\text{ nm}$ .

### 8.2.3 Conclusion

Undoped layers based on an "optimized GaN" layer do not meet the requirements for high HEMT performance since they show distinct parallel conductivity to the channel layer. A strategy for growing semi-insulating GaN was introduced using nitrogen as the carrier gas. The present results suggest that the increased number of edge dislocations compensate the donors present in GaN and makes it highly resistive. One key factor for varying the density of these dislocations is the morphology development in coalescence. Decreasing the coalescence time by using nitrogen in combination with shorter annealing times is beneficial for obtaining highly resistive layers ( $1 \times 10^7 \Omega\text{cm}$ ) with smooth and specular surfaces. Also the question whether the decreased structural properties and surface roughnesses influence the electrical properties of the 2DEG could be clarified. Hall measurements proof that our heterostructures are insensitive to these drawbacks. Electron mobilities at room temperature of  $1490\text{ cm}^2/\text{Vs}$  at sheet carrier concentrations of  $9.88 \times 10^{12}\text{ cm}^{-2}$  demonstrate the feasibility of HEMT structures grown on "nitrogen GaN". However, Hall measurements only give a first impression of the layer quality. We need to turn to HEMT device characterization (section 8.3) to give a final proof of the

feasibility of our growth approach.

### 8.3 HEMT results

The optimization of the GaN buffer and the AlGaN layer is successfully completed. However its applicability with respect to HEMT device characteristics needs still to be proved.

*RF-high-power HEMTs* represent the application aim of our work. In contrast to small signal devices power HEMTs have gate lengths of 0.5 to 1  $\mu\text{m}$  and cover areas of typically several square-millimeter. These devices have to withstand a larger voltage and current amplitude as compared to a small signal device, to provide high output power. Therefore the fulfillment of the -throughout this thesis- often mentioned requirements of reproducibility, uniformity and material quality is paramount. To give a final proof of the feasibility of our growth approach power HEMT devices were fabricated. In this section the DC and RF characteristics of our grown heterostructures are discussed.

The HEMT is a three terminal device (as seen in figure 8.16) and is geometrically characterized by the gate length  $L_g$ , the gate width  $W_g$ , and the Source-Drain distance S.D. The current flow in between the contacts is controlled by a bias voltage through the middle Schottky contact (Gate). The standard HEMT processing procedure requires several lithographic steps and consists of the mesa etching, the fabrication of ohmic and Schottky contacts for the source/drain contacts and the gate respectively. Further details about HEMT processing can be found in [Ber05]. It is worth noticing that the fabricated devices are unpassivated.

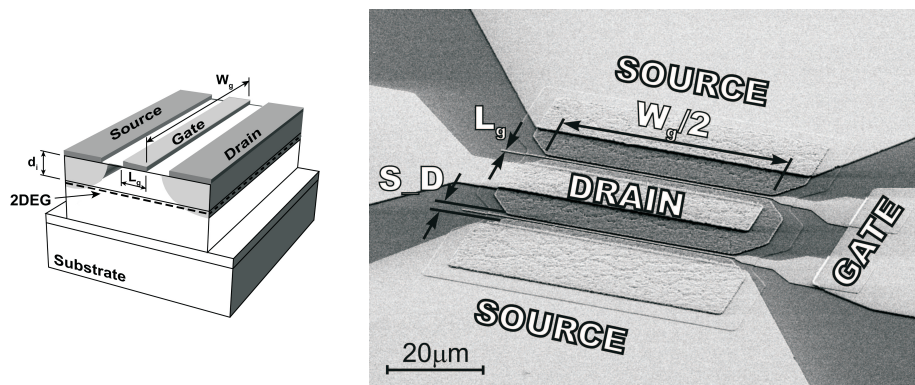


Figure 8.16: Schematic drawing of a HEMT structure (left) and a real processed HEMT (right).

The layer stack consists of a 2.5  $\mu\text{m}$  thick undoped GaN buffer followed by a 27 nm thin  $\text{Al}_{0.35}\text{Ga}_{0.65}\text{N}$  layer covered by a 4 nm GaN cap as determined via RBS. We compare

samples with different buffer resistivity values of  $3 \times 10^5$  and  $1 \times 10^7 \Omega\text{cm}$  and otherwise identical heterostructure. The latter sample originates from the "annealing time" series in section 8.2.2 with 0 minutes annealing at  $970^\circ\text{C}$ .

Hall measurements at room temperature reveal mobilities of 1405 and  $1093 \text{ cm}^2/\text{Vs}$  at sheet carrier concentrations of 8.2 and  $8.7 \times 10^{12} \text{ cm}^{-2}$ , respectively. The difference is not necessarily originating from the different layer characteristics, but due to the applied and rather simplistic alloying procedure based on indium contacts. Moreover the second measured sample is cut from near the wafer edge since the middle was used for the HEMT processing.

Measurements of the output and the transfer characteristics are one of the basic methods for HEMT characterization. Using these measurements the functionality of the device and the quality of the layer structure can be analyzed. All measurements are performed by means of an Agilent E5270A semiconductor analyzer. The measurement system allows to measure in a bias range of -200 to +200 V. The current range is up to 1A with femto-ampere accuracy. The gate-source and the drain-source biases are controlled simultaneously and the gate and drain currents are read subsequently.

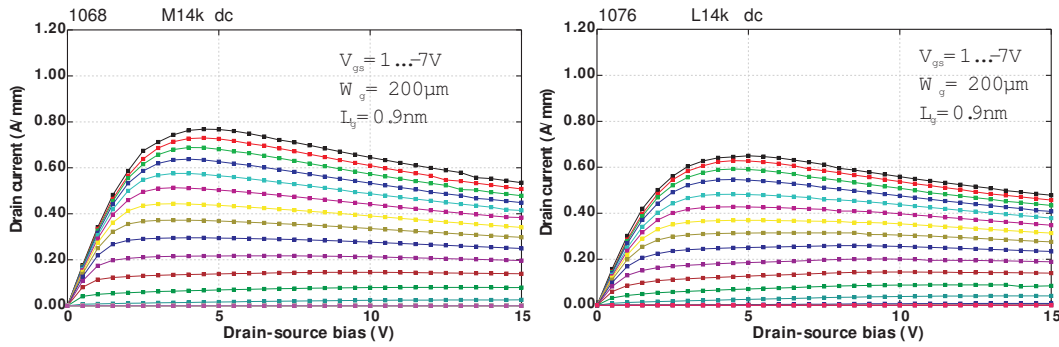


Figure 8.17: DC output characteristics for a gate bias from 1 to -7 V of two samples grown on different GaN buffers.  $L_g = 0.9 \mu\text{m}$ ;  $W_g = 200 \mu\text{m}$

From measurements one can extract the maximum drain current  $I_d^{sat}$ , the threshold voltage  $V_{th}$ , the extrinsic transconductance  $g_m$  and gate leakage currents. The typical output and transfer characteristics measured for both samples are shown in figure 8.17 for gate width of  $200 \mu\text{m}$ , a gate length of  $0.9 \mu\text{m}$  and a source drain distance of  $3 \mu\text{m}$  (two finger device). In either transistor structure a decreasing drain current is measured in the saturation region with increasing drain-source bias. The origin of this negative resistance effect has been attributed to self heating of the device due to the poor thermal conductivity of the sapphire substrate [Ber03]. This effect can thus only be reduced by using different substrates. The output characteristics shown in figure 8.17 exhibit a saturation drain current of 0.78 and 0.68 A/mm respectively. The origin of this slight difference can only be speculated upon. Further measurements will be necessary. It is reasonable to assume that

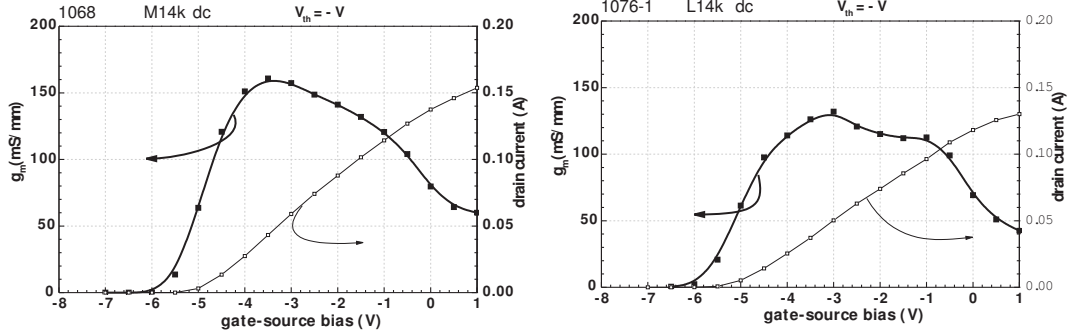


Figure 8.18: Transfer characteristics of the HEMT structures.

device manufacturing instead of intrinsic material properties accounts for the difference. Other origins of this difference may be the dependency of  $I_d$  on the mobility, electron velocity and carrier concentration. However, since RBS of these samples demonstrated identical structures and compositions at least a change in the carrier concentration can be excluded.

Extrinsic transconductances of 160 and 130  $mS/mm$  at a threshold voltage of  $-5.5 V$  are measured for the devices as seen in figure 8.18. These results are comparable with published results of AlGaIn/GaN/Sapphire HEMTs where  $I_d^{sat}$  reaches from 0.46A/mm to 0.95A/mm and  $g_m$  from 150  $mS/mm$  to 236  $mS/mm$  [Jav03, Gre00, Chi03, Dav04, Joh01]. The gate leakage currents were measured to be 1 and 0.1  $\mu A$  at  $V_{gs} = -6 V$  and  $V_{ds} = 0V$ , respectively.

Boundary frequencies are important for RF characterization. These parameters prove the device capability to operate at high frequencies or with high switching speeds. To characterize the frequency behavior two basic parameters are used in practice: The current gain cutoff frequency (or transit frequency)  $f_T$  and the maximum frequency of oscillation  $f_{max}$ , which define the frequency range where the HEMT shows amplifying behavior. The boundary frequencies were evaluated from  $s$ -parameter measurements in a frequency range up to 110GHz using a HP 8510C Network Analyzer. Devices were measured for gate source bias in a range from  $+1 V$  to  $V_{th}$  and drain source bias in a range from 0 to 20  $V$ . To be comparable to HEMT results from GaN on SiC, which were processed in our labs before, the GaN on sapphire devices were measured at comparable working points ( $V_{gs} = -2.5 V$  and  $V_{ds} = 20 V$ ). The devices with a gate length of  $L_g = 0.9 \mu m$  showed a cutoff frequency of  $f_T = 6.5$  and 11.2  $GHz$  respectively and a maximum frequency of oscillation of 20 and 18.9  $GHz$ . Our HEMTs on SiC substrates show similar cutoff frequencies ( $\sim 10 GHz$  for  $f_T$ ). Since the frequencies are working point and HEMT geometry dependent a working point more suitable for high transit frequencies ( $V_{gs} = -5 V$  and  $V_{ds} = 20 V$ ) shows a  $f_t$  value of 14.1 and 13.3  $GHz$  respectively. These results are also comparable to other published values of AlGaIn/GaN/Sapphire HEMTs [Jav03].

These findings finally prove the functionality and feasibility of our MOVPE growth process. Moreover the output and RF characteristics are comparable to HEMT results of GaN on SiC produced at our institute [Heid06]. Thus we are able to fabricate GaN-based HEMT devices with state of the art electrical characteristics using the new growth process approaches.



## Chapter 9

# Summary and Conclusions

Research aim of this work was to develop an MOVPE based growth process that allows the reliable and efficient deposition of high quality material for HEMT devices based on AlGaIn/GaN heterojunctions. The main task was to establish a broad comprehension of the epitaxial process based on numerical and experimental data and eventually to prove its functionality in terms of HEMT characteristics. On account of this, several requirements for suitable power HEMT material were derived in this work:

- Reproducible layer growth

Usage and development of adequate growth control and monitoring instruments is essential to meet these premises. It was shown that *direct wafer surface temperature determination is a must* during growth, though it is challenging for transparent substrates. Emissivity corrected pyrometry is a widely employed tool for surface temperature measurements. However, commonly applied calibration methods for absolute temperature determination of the wafer surface can be erroneous when based on phase transition or melting point observations. Therefore, *a new calibration method for transparent substrates has been demonstrated* by using the in-situ measured, temperature dependent and band-gap related reflectance shift of SiC. This new approach for temperature determination makes growth conditions on transparent substrates comparable, independent of the reactor type used. Moreover and more importantly for the following discussion, this technique allows to adjust the MOVPE system to identical run-by-run wafer temperatures. *A reliable basis for absolute temperature determination is established.* Using this new temperature determination approach the cause of irreproducibility in an horizontal reactor is investigated. Parasitic deposits that form on the cold reactor walls and insufficient temperature control (the one allowing for the other) are the two major origins of irreproducibility.

*A new deposition approach is introduced that reduces the formation of parasitic depositions on the reactor walls, by injecting the metalorganics near the substrate unlike the conventional process. Experiment and modelling verify the suitability of the new growth approach for enhanced reproducibility. Moreover the new approach leads to a fourfold reactor uptime without changing reactor quartz parts. The structural, morphological and electrical characteristics of the grown layers are comparable for both processes.*

- "Manufacturing uniformity" in terms of a high yield of homogenous layer characteristics

Homogeneity in terms of thickness and epilayer characteristics was addressed next. It has been found that inversion of the precursor supply has a drawback when considering the thickness uniformity. In order to achieve a homogeneous distribution of growth species over the substrate flow mechanical aspects in our reactor system are investigated. The gas velocity as well as the gas "nature" are therefore important optimization parameters. For that purpose the role of different carrier gases - nitrogen and hydrogen - on III-nitride growth is examined. *Using the inverted inlet configuration, uniformity can only be improved by changing the nature of the carrier gas*, thus, modifying the diffusive transport and decomposition of growth species. Thus, the growth species are distributed more uniformly above the susceptor. This way, in  $N_2$  atmosphere *a standard deviation in thickness of less than 3%* is achieved in comparison to 10% for  $H_2$  ambient. Further investigations are necessary to understand the influence of the nitrogen carrier gas on GaN epilayers characteristics.

- Structural quality with low dislocation densities and a smooth, defect free surface

Nitrogen is crucial for improved thickness homogeneity, but apparently nitrogen not only deteriorates the surface morphology but it affects the growth mode in the early stages of deposition. From the presented data *it is found that untimely recovery of the reflectance signal, i.e. start of 2D growth, leads to GaN layers which contain a larger number of edge type dislocations*. Dislocations, in turn, influence the strain in the layer, leading to more relaxed GaN layers when grown in nitrogen ambient. Dislocation scattering is the very probable source of the decline in electrical data when nitrogen is used as carrier gas. As a result a larger "coalescence thickness" is preferred, since it reduces the density of dislocations. Therefore a strategy was developed with which good quality material can be obtained despite the nitrogen ambient needed for uniform layers.

*State of the art electrical and structural quality material as well as uniform layers with smooth surfaces were achieved by using only small amounts of hydrogen ( $\sim 15\%$ ) during the coalescence phase in otherwise nitrogen ambient. We managed to even increase the Hall mobility at room temperature up to  $671 \text{ cm}^2/\text{Vs}$  with a carrier concentration of*

$9 \times 10^{16} \text{cm}^{-3}$  and a FWHM for the (002) and (102) reflections of 238 and 349 *arcsec* respectively at a standard deviation in thickness of 3%. In this way, a high quality GaN template is produced. Further investigation are carried out to check the suitability as a buffer for HEMTs.

- Tight alloy composition and thickness control in the heterostructure

AlGa<sub>N</sub> was grown on top of the high quality GaN template with the main focus on controlled Aluminium content and studies were performed regarding the abrupt- and smoothness of the heterostructure interface. *The inverted inlet combines the reproducibility of the growth process with a good growth control for  $\text{Al}_x\text{Ga}_{1-x}\text{N}$ .* Due to the high incorporation efficiency a minimal amount of adduct formation occurred. *Tight alloy composition and thickness control was achieved* with less than one percent deviation over the wafer for device relevant aluminum contents in either hydrogen and nitrogen as carrier gas. AlGa<sub>N</sub>/GaN heterostructures were grown on the "optimized GaN". Despite state of the art sheet conductivity values ( $1.92 \times 10^{16} \text{V}^{-1}\text{s}^{-1}$ ) the heterojunctions exhibit additional conductivity due to residual donors in the layer. That way we measured a resistivity of less than  $1 \Omega\text{cm}$  in the GaN. In a device structure we found considerable parallel conduction effects leading to unsatisfactory device pinch off. Therefore resistance control was addressed next to achieve high quality HEMT devices.

- Semi-insulating GaN layers

A strategy for growing semi-insulating GaN was introduced using nitrogen as the carrier gas. The present results suggest that *the increased number of edge dislocations compensates GaN and makes it highly resistive.* One key factor for varying the density of these dislocations is the morphology development in coalescence. Decreasing the coalescence time by *using nitrogen in combination with shorter annealing times is beneficial for obtaining high resistive layers* ( $1 \times 10^7 \Omega\text{cm}$ ) with smooth and specular surfaces. Also the question whether the decreased structural quality and surface roughnesses influence the electrical properties could be clarified. Electron mobilities at room temperature of  $1490 \text{cm}^2/\text{Vs}$  at a sheet carrier concentrations of  $9.88 \times 10^{12} \text{cm}^{-2}$  demonstrate the suitability of HEMT structures grown on "nitrogen GaN".

The output and RF characteristics of the produced HEMT structures are comparable to HEMT results of GaN on sapphire (and even GaN on SiC, produced at our institute), with values for  $I_d^{\text{sat}}$  of  $\sim 0.7 \text{ A/mm}$  and  $g_m$  of  $\sim 150 \text{ mS/mm}$  and cutoff frequencies  $\sim 11 \text{ GHz}$  for the chosen geometry and working point. These findings finally prove the functionality and feasibility of our MOVPE growth process.

Thus, new holistic approaches to III-nitride monitoring and growth are presented. High reliability, reproducibility and minimum downtimes of the equipment is proven, optimized

AlGaIn/GaN film deposition shown and transferability of process temperatures to any reactor demonstrated.

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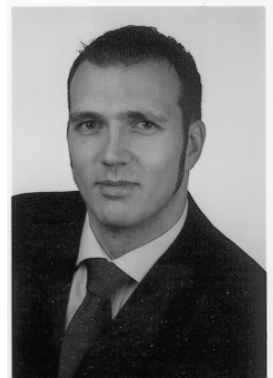
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# LEBENS LAUF

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## **PERSÖNLICHE INFORMATION**

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- 09/2002-03/2003 Diplomarbeit am Forschungszentrum Jülich
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- Französisch:          Grundkenntnisse

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- Computer Applications: Origin, Frontpage, Maple, LaTeX, Microsoft Office
- Operating Systems: Linux, Windows XP/2000/NT/95/98
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## **PERSONAL STATEMENT**

- Greift nur hinein ins volle Menschenleben! // Ein jeder lebt's, nicht vielen ist's bekannt, // Und wo Ihr's packt, da ist's interessant