Hot-wall MOCVD of N-polar group-III nitride materials

Hengfang Zhang

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During the course of research underlying this thesis, Hengfang Zhang was enrolled in Agora Materiae, a multidisciplinary doctoral program at Linköping University, Sweden.

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ABSTRACT

Group III-Nitride semiconductors: indium nitride (InN), gallium nitride (GaN), aluminum nitride (AlN) and their alloys continue to attract significant scientific interest due to their unique properties and diverse applications in photonic and electronic applications. Group-III nitrides have direct bandgaps which cover the entire spectral range from the infrared (InN) to the ultraviolet (GaN) and to the deep ultraviolet (AlN). This makes III-nitride materials suitable for high-efficient and energy-saving optoelectronic devices, such as light-emitting diodes (LEDs) and laser diodes (LDs). The Nobel Prize in Physics 2014 was awarded for the invention of efficient GaN blue LEDs, which further accelerated the research in the field of group III-nitride materials. GaN and related alloys are also suitable for high-temperature, high-power and high-frequency electronic devices with performance that cannot be delivered by other semiconductor technologies such as silicon (Si) and gallium arsenide (GaAs). For example, GaN-based high electron mobility transistors (HEMTs) have been widely adopted for radio frequency (RF) communication and power amplifiers, high-voltage power switches in radars, satellites, and wireless base stations for 5G.

Recently, nitrogen (N)-polar group-III nitrides have drawn much attention due to their advantages over their metal-polar counterparts in e.g. HEMTs. These include feasibility to fabricate ohmic contacts with low resistance, an enhanced carrier confinement with a natural back barrier, and improved device scalability. Despite intensive research, the growth of micrometer-thick high-quality N-polar GaN based materials remains challenging. One of the major problems to develop device-quality N-polar nitrides is the high surface roughness, which results from the formation of hexagonal hillocks or step-bunching. Another significant hurdle is the unintentional polarity inversion, which reduces the crystalline quality and prohibits device fabrication.

This licentiate thesis focuses on the development of N-polar AlN and GaN heterostructures on SiC substrates for HEMT RF applications. The overall aim is to exploit the advantages of the hot-wall MOCVD concept to grow high-quality N-polar HEMT structures for higher operational frequencies and improved device performance. In order to achieve this goal, special effort is dedicated to understanding the effects of growth conditions and substrate orientation on the structural properties and polarity of AlN, GaN and AlGaIn grown by hot-wall MOCVD. N-polar AlN nucleation layers (NLs) with layer by layer growth mode and step-flow growth mode can be achieved on on-axis and 4° off-axis SiC (000 $\bar{1}$), respectively, by carefully controlling V/III ratio and growth temperature. Utilizing scanning transmission electron microscopy (STEM) we have established a comprehensive picture of the atomic arrangements, local polarity and polarity evolution in AlN,

GaN/AlN and AlGaN/GaN/AlN in the cases of low-temperature and high-temperature AlN NLs both for on-axis and off-axis substrates. We have shown that typically employed methods for polarity determination using potassium hydroxide wet etching could not provide conclusive results in the case of mixed-polar AlN as Al-polar domains may be easily over-etched and remain undetected. Atomic scale electron microscopy is therefore needed to accurately determine the polarity. We further have developed growth strategy and have optimized the epitaxial process for N-polar GaN, and have demonstrated high quality N-polar AlGaN/GaN/AlN heterostructures.

POPULÄRVETENSKAPLIG SAMMANFATTNING

Halvledare, likt kisel (Si), som är baserade på grupp III-nitrider, tex indiumnitrid (InN), galliumnitrid (GaN), aluminiumnitrid (AlN) och deras legeringar fortsätter att attrahera vetenskapligt intresse på grund av deras unika egenskaper väl anpassade för fotoniska och elektroniska tillämpningar. Grupp III-nitrider besitter direkta bandgap som sträcker sig över hela energispektrumet från infrarött (InN) till djup ultraviolett (GaN). Det innebär att III-nitrider är lämpliga som energieffektiva optoelektroniska komponenter, så som ljusdioder och laserdioder. Nobelpriset i fysik 2014 tilldelades innovationen för den effektiva blå ljusdioden och därefter har utvecklingen av III-nitrider ökat avsevärt. GaN med relaterade legeringar är också lämpliga inom högtemperatur-, högspännings- och högfrekvenselektronik med en prestanda som överstiger existerande teknologi, tex Si och galliumarsenid (GaAs). GaN-baserade högelektronmobilitetstransistorer (HEMTs) är ett exempel som är väletablerad på marknaden för radiofrekvenskommunikation (RF), högeffektsförstärkare samt även inom högspänningsväxlar i radar, satelliter och trådlösa basstationer för 5G.

Kvävepolära (N-polära) grupp III-nitrider har på kort tid fått ökad uppmärksamhet pga. deras fördelar i jämförelse med deras mycket vanligare metallpolära motsvarigheter i tex HEMTs. Det inkluderar möjligheten att realisera ohmiska kontakter med låg resistans, en förbättrad inneslutning av laddningsbärare med en naturlig ledningsbandsbarriär samt en förbättrad komponentuppskalningsförmåga. Grupp III-nitridlager och komponentstrukturer är till mestadels fabricerade genom metallorganisk kemisk ångdeposition (MOCVD) på separata substrat som kiselkarbid (SiC). Trots intensiva undersökningar har tillväxt av mikrometertjocka lager av hög kvalitet för N-polära GaN-baserade material visat sig mödosamt. En av de svåraste utmaningarna som återstår för N-polär tillväxt är ojämnheter på ytan i form av hexagonala kullar eller steghopning. Ytterligare en svår utmaning är spontana polaritetsinversioner som uppkommer i MOCVD tillväxt av grupp III-nitrider som påverkar både kristallkvaliteten och elektroniska egenskaper.

Den här licentiatavhandlingen fokuserar på utveckling av N-polära AlN- och GaN-heterostrukturer på SiC-substrat för HEMT-baserade RF-tillämpningar. Målet är att exploatera fördelarna med MOCVD-konceptet, specifikt varmvägs MOCVD, för att växa N-polära HEMT-strukturer för ökat frekvensomfång samt en förbättrad prestanda. I genomförandet av detta mål läggs speciell fokus på en ökad förståelse av tillväxsvillkor och substratriktningar och dess inverkan på strukturella egenskaper och polaritet av AlN, GaN samt AlGaIn. N-polära kärnbildningslager (NL) av AlN med lager-på-lager-tillväxt och stegflödetillväxt presenteras på på-axel-tillväxt samt för 4° avskärningstillväxt av kolterminerad SiC ($000\bar{1}$) genom noggrann kontroll av kväve/metall inblandning (V/III) och temperatur. Men

hjälp av sveptransmissionselektronmikroskopi (STEM) har en bred kunskapsbas etablerats gällande atomära arrangemang, lokala polaritetsdomäner samt deras utveckling under olika tillväxtsvillkor av AlN-, AlN/GaN- och även för AlGaN/GaN/AlN-strukturer vid låga och höga temperaturer. Det har visats att den huvudsakliga karaktäriseringsmetoden för att påvisa polaritet, KOH-etsning, har begränsningar när det gäller mixade polariteter. Atomupplöst STEM är därför ett nödvändigt komplement för att med säkerhet kunna särskilja mixade polariteter. Det har dessutom fokuserats på att utveckla en tillväxtstrategi för optimering av epitaxiella processer av N-polära AlGaN/GaN/AlN-heterostrukturer av hög kvalitet.

PREFACE

The licentiate is based on the knowledge and research results accumulated during the 2.5-years of graduate studies of Hengfang Zhang at the Swedish Center for III-Nitride Technology (C₃NiT), Department of Physics, Chemistry and Biology at Linköping University from September 2017 to May 2021. The licentiate contains two main parts: the first part provides a brief introduction to the research field and a brief summary of the thesis work, and the second part presents the main research results summarized in three scientific papers.

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Part I

1.1 Introduction

Group-III nitrides are regarded as materials for the third generation of semiconductor technology and attract significant research and commercial interests for electronic and optoelectronic applications. Group-III nitride semiconductors include *indium nitride* (InN), *gallium nitride* (GaN), *aluminum nitride* (AlN) and their ternary and quaternary alloys. The bandgap of these material span from 0.7 eV to 6.2 eV [1, 2] covering the entire spectral range from *infrared* (IR) to deep *ultraviolet* (UV). This makes group-III nitrides suitable materials for optoelectronic devices, such as *light-emitting diodes* (LEDs) [3–6], *laser diodes* (LDs) [7, 8], and UV *photodetectors* (PDs) [9, 10]. The 2014 Nobel Prize in Physics was awarded to Isamu Akasaki, Hiroshi Amano and Shuji Nakamura for the invention of the efficient blue GaN LEDs [11].

Another important area for application of group-III nitrides is the *high power* (HP) and *high frequency* (HF) electronic devices. Wide-bandgap semiconductors provide favorable properties like high breakdown electric field, high saturated electron velocity, and high thermal conductivity, and high saturation carrier velocity. The *two-dimensional electron gas* (2DEG) formed in *aluminum gallium nitride* (AlGaN)/GaN heterostructures offers a superior electronic transport allowing fabrication of *high electron mobility transistor* (HEMTs) with a high efficiency. Especially, the high 2DEG sheet electron density in AlGaN/GaN HEMTs enables an operation in the millimeter wave range (> 30 GHz). For example, AlGaN/GaN HEMTs with potential applications in *radio frequency* (RF) communication and power amplifiers for radars, missiles, satellites have been reported [12–15].

GaN and related group-III nitride crystals do not occur naturally. In early 1960s, the GaN compound was prepared by the reaction between gaseous *ammonia* (NH_3) and *gallium phosphide* (GaP) or *gallium arsenide* (GaAs) in fine powders[16]. Single crystalline GaN was demonstrated in the beginning of 1970s [17, 18]. One of the main challenge in

development of group-III nitride materials is the lack of native substrates. Commonly, *silicon carbide* (SiC), *silicon* (Si), or *sapphire* (α -Al₂O₃) substrates are used for epitaxial growth. The main techniques for growth of layers and device structures are metal-organic chemical vapor deposition (MOCVD) and molecular beam epitaxy (MBE).

For epitaxy of GaN layers, sapphire (0001) (c-plane) substrates, which have a hexagonal unit cell, are usually employed [4]. However, the large lattice mismatch (16%) together with the difference in the thermal expansion coefficients (27%) between sapphire and GaN (Table 1.1) results in a formation of high density of extended defects in heteroepitaxial layers. In order to mitigate these issues, substrate pretreatment such as thermal annealing [19] and nitridation [20] were employed. In addition, an AlN [21] or a low temperature GaN buffer layer [22] improves the crystalline quality of the layers.

Due to the low cost, large wafer size and compatibility with Si metal-oxide-semiconductor (MOS) technology, Si is widely used as a substrate for GaN epitaxy. However, there are some challenges in order to realize high quality material on Si substrates. As shown in Table 1.1, the lattice mismatch between Si and GaN is 16.9% causing a high dislocation density. The large difference (56%) in the thermal expansion coefficient between the two materials results in surface cracking which is a big obstacle for device applications [23]. Researchers have adopted different approaches in order to alleviate these problems and realize high quality GaN on Si substrates. For example, different interlayers such as Si_xN_y, low-temperature AlN, AlGN/GaN superlattices, and graded AlGaIn buffer layers were developed. Another approach employs patterned Si substrates in order to reduce stress in the overgrown GaN layers [23, 24].

Currently, SiC substrates (4H and 6H) are the most often employed for GaN-based HP and HF electronic devices. SiC and GaN both have wurtzite crystal structures. As shown in Table 1.1, the in-plane lattice constants of 4H-SiC and GaN are closer to each other resulting in a small lattice mismatch (3.5%). It is still difficult to achieve layer-by-layer two-dimensional growth of GaN directly on SiC due to wetting problem or because of the onset of Stranski-Krastanov (SK) growth mode in combination with two-dimensional and three-dimensional growth [25, 26]. The use of AlN nucleation layers [25, 27, 28] and pre-treatment of substrate surfaces [29–31], have been demonstrated to enable high-quality GaN layers on SiC. Applying these approaches, high quality GaN-based devices on SiC have been demonstrated [14, 32–34].

In this thesis, we focus on the developments of N-polar III-Nitride materials on SiC for HF HEMTs applications. Although, N-polar GaN-based HEMTs have been demonstrated on sapphire and Si substrates. However, due to the low thermal conductivities of the substrates, these devices are unsuitable for HP and HF applications. In addition, the dislocation density in layers grown on sapphire (10^8 cm^{-2}) [38, 39] and Si (10^9 cm^{-2})

Table 1.1: Material properties of Si, α -Sapphire, 4H-SiC, and Wurtzite GaN.

	Si ^{a,b,d}	α -Sapphire ^{a,c}	4H-SiC ^{b,d}	GaN ^{a,c,d}
Lattice constant				
a (@300 K) [nm]	0.54301	0.4758	0.307	0.3189
c (@300 K) [nm]		1.2991	1.008	0.5185
Thermal expansion coefficient				
α [10^{-6} K $^{-1}$]	3.59	7.5	3.30	5.59
β [10^{-6} K $^{-1}$]		8.5	3.16	3.17
Thermal conductivity [W (cm $\cdot$ $\text{\AA}$ $\cdot$ K) $^{-1}$]	1.3	0.46	4.9	1.1
Bandgap (@300 K) [eV]	1.12	8.1 – 8.6	3.2	3.44
Melting point [K]	1690	2303	3103	2846
Critical electric field [10^6 V/cm]	0.3		3.5	3.3

^a Ref. [35], ^b Ref. [36], ^c Ref. [4], ^d Ref. [37]

[40–42] is usually higher in comparison to material grown on SiC [42, 43]. Dislocations typically present leakage current paths limiting device performance. Free-standing GaN substrates are commercially available, but the high cost still limits their application in large-scale device fabrication and wide adoption.

1.2 Fundamental properties of group-III nitride semiconductors

1.2.1 Crystal structure of group-III nitrides

Group III-nitrides may crystallize in wurtzite, zinc blende or rock salt crystal structures as shown in Figure 1.1. The rocksalt or NaCl structure of III-nitrides (Fig. 1.1 (c)) has been experimentally observed at high pressure (> 12 GPa), but is hardly produced by any epitaxial growth methods [44]. The zinc blende structure of III-nitrides (Fig. 1.1 (b)) has a cubic unit cell with the position of the atoms identical to the diamond crystal structure. This unit cell is described by one lattice parameter. The thermodynamically stable crystal structure is wurtzite which is commonly realized for typical III-nitrides film growth. The wurtzite crystalline structure has a hexagonal unit cell with two lattice constants a and c as shown in Fig. 1.1 (a). The a lattice is along the $[1\bar{1}00]$ direction and the c lattice is along the $[0001]$ direction as described in the Miller-Bravais notation [45]. In both zinc blende and wurtzite crystal structures, each group-III atom is positioned at the center of a tetrahedron, with four nearest nitrogen neighbors in the corners, and vice versa. The wurtzite structure is composed of two interpenetrating *hexagonal close packed* (hcp) sublattices: one consisting of cations (Ga^{3+} , Al^{3+} or In^{3+}) and the other occupied by anions (N^{3-}). For the ideal hcp structure, the c/a ratio equals $\sqrt{3/8}$ and the cation lattice is shifted by $3c/8$ with respect

to the anion lattice along the c -axis (also refer as $[0001]$ or $[000\bar{1}]$ direction) [46]. However, for almost all the as-grown III-nitride films in reality, the wurtzite structure is not ideal leading to smaller bonding angles resulting in a c/a ratio smaller than $\sqrt{3}/8$.

Figure 1.1: Ball-and-stick stacking model representations of GaN crystal structures: (a) wurtzite GaN along the $[0001]$ direction, (b) cubic zincblende GaN along the $[111]$ direction, and (c) cubic rocksalt GaN along the $[001]$ direction. The respective top views are presented below each crystal structure. The bonds in the a -plane ($11\bar{2}0$) and the (111) plane are indicated with dashed lines to highlight the stacking sequences in the wurtzite and zincblende structures. The small green balls indicate Ga^{3+} while the large blue balls indicate N^{3-} .

The main difference between wurtzite and zinc blende structures is the stacking sequences of the closed-packed planes. The closed-packed plane of wurtzite structure is the (0001) c -plane. The stacking sequence of GaN (AlN) along the c -axis is written as ABABAB... as shown in Fig. 1.1 (a), which is similar to the sequence of hexagonal closed packed structures [45]. In zinc blende structure, a 60° in-plane (110) rotation leads to the stacking sequence for (111) close-packed planes of ABCABC... as illustrated in Fig. 1.1 (b). The bonds in the a -plane ($11\bar{2}0$) and the (110) plane are indicated with dashed lines to show the stacking sequences in the wurtzite and zinc blende crystal structures, respectively.

Figure 1.2: Schematic illustration of GaN wurtzite crystal structure by ball and stick models with: (a) Ga-polarity along the +c ([0001]) and (b) N-polarity along the -c ([000 $\bar{1}$]). The single unit cell is indicated. When the direction of three bonds of the metal-elements is towards the substrate, it is defined as metal-polar or +c polarity. On the other hand, when the three bonds of the metal-elements is upward against the substrate, it is defined as N-polar or -c polarity.

1.2.2 Polarity of group-III nitrides

The wurtzite III-nitride semiconductors are pyroelectric and piezoelectric materials, and are typically grown along the polar axis (c-axis). The [0001] direction is known as metal (Al-/Ga-)polar, while the [000 $\bar{1}$] direction is known as *nitrogen-polar* (N-polar). Figure 1.2 shows a schematic illustration of GaN wurtzite crystal with Ga-polarity along the +c ([0001]) and N-polarity along the -c ([000 $\bar{1}$]). The single unit cell is indicated in the figure. When the direction of the three bonds of the metal-elements (Fig. 1.2 (a)) is towards the substrate, it is defined as metal-polar or +c polarity. On the other hand, when the three bonds of the metal-elements is upward against the substrate, it is defined as N-polar or -c polarity. It is worth noting that the termination on the surface cannot define the polarity as the surface atoms can be either *gallium* (Ga) or *nitrogen* (N) for both polarity situations.

Wurtzite III-nitrides have large spontaneous polarization (P_{sp}), which is driven by the noncentrosymmetric crystal structure with the tetrahedral coordination along the c-axis. The spontaneous polarization of a wurtzite semiconductor can be theoretically calculated, e.g. by *density-functional theory* (DFT) and experimentally estimated from the emission energies of bound excitons [47]. The spontaneous polarization of GaN and AlN were found to be negative, indicating that the spontaneous polarization in N-polar heterostructures is pointing in the direction of growth, while it is in the opposite direction for the metal-polar structures [48]. In addition, the spontaneous polarization show quantitative differences due to the different crystalline structural parameters. III-nitrides are typically grown

heteroepitaxially on foreign substrates. The difference in lattice constants and thermal expansion coefficients between the substrate and the epitaxial layer create a strain in III-nitrides layers causing the strain-related piezoelectric effect in heterostructures [34, 49]. The piezoelectric polarization, P_{pz} , can be determined by [50]:

$$P_{pz} = e_{ij}\epsilon_j = d_{ij}c_{jk}\epsilon_k, j, k = xx, yy, zz, yz, zx, xy, \quad (1.1)$$

where ϵ_j is the strain tensor, e_{ij} are the strain piezoelectric coefficients e_{ij} , $c_{ij}\epsilon_k$ is the stress tensor, where c_{jk} is the elastic constants tensor, and d_{ij} are the stress piezoelectric coefficients. x, y, z are defined along the main crystallographic directions $[1\bar{1}00]$, $[11\bar{2}0]$ and $[0001]$, respectively.

Fig. 1.3 shows the orientations of the spontaneous polarization and piezoelectric polarization in strained and relaxed AlGaIn/GaN heterostructures in Ga-polar (indicated by Ga-face in Fig. 1.3) and N-polar (indicated by N-face Fig. 1.3) respectively [48]. If the heterostructure is relaxed (see Fig. 1.3 (a) and (d)), only spontaneous polarization plays a role at the AlGaIn/GaN interface. The piezoelectric polarization is negative and has the same direction as the spontaneous polarization for tensile strained AlGaIn barrier layers while for compressive strained AlGaIn, P_{pz} is positive and has the opposite direction than P_{sp} . The total polarization field in the heterostructure is the sum of the spontaneous polarization and the piezoelectric polarization. At the heterojunction, a sheet charge density (σ) is induced by the total polarization. This sheet charge density can be affected by e.g. the interface roughness, composition variations, or strain distribution. For N-polar heterostructures, the sheet charge density is negative (see Fig. 1.3 (d) and (e)), and holes will be accumulated at the interface forming two-dimensional hole gas (2DHG). In contrast, electrons will be confined forming 2DEG when the sheet charge is positive (see Fig. 1.3 (f)) in the case of N-polar GaN grown on top of AlGaIn. The polarization induced charges are the main factor to determine the carrier concentrations in AlGaIn/GaN transistor structures [34, 50].

Good control of the polarity is necessary to exploit the polarization effects in III Nitrides for developing optoelectronic and electronic devices with improved performance and new functionalities. Metal-polar III-nitride heterostructures and devices are well developed and widely utilized. On the other hand, the development of N-polar III-nitride heterostructures has been lagging behind. The reason is that it is challenging to grow high-quality N-polar films, which have typically very rough surfaces with large hexagonal hillocks. In contrast metal-polar films are atomically smooth and of good crystal quality. In addition, polarity control in N-polar nitride heterostructures has proven difficult with Me-polar domains forming and disrupting crystal quality or leading to complete polarity inversion. Nonetheless, N-polar III-nitrides have attracted great interest owing to their

Figure 1.3: Spontaneous polarization (P_{sp}) and piezoelectric polarization (P_{pz}) in strained and relaxed AlGaN/GaN heterostructures. (a), (b) and (c) show polarization of metal-polar (Ga (Al)-polar) heterostructures grown along the $+c$ ([0001]), while (d), (e) and (f) show polarization of N-polar heterostructures grown along the $-c$ ([000 $\bar{1}$]). σ indicates the polarization induced sheet charge density at the heterojunction. Arrows indicate the directions of corresponding polarizations [Reproduced from Ambercher, O. et al., *J. Appl. Phys.*, 85, 3222, 1999] [48]

superior optical and electrical properties [51]. N-polar LEDs with wide range of emission wavelengths have been demonstrated exhibiting superiority in suppressing the electron leakage and promoting carriers confinement capability due to the reversed polarization field over *gallium-polar* (Ga-polar) HEMTs [46, 52–54]. N-polar III-nitrides heterostructures provide a low resistivity with low ohmic contacts, a high carrier confinement with a nature back barriers, and an improved scalability with short channels [46, 55–57].

Figure 1.4: Equilibrium band diagrams for metal-polar AlGaIn/GaN and N-polar GaN/AlGaIn/GaN heterostructures showing polarization induced two-dimensional electron gas.

1.2.3 N-polar HEMTs

In AlGaIn/GaN HEMTs structures, the combined spontaneous and piezoelectric polarizations cause a polarization induced band offset at the AlGaIn/GaN interface resulting in the formation of 2DEG at the interface [34, 51, 55]. Figure 1.4 shows the equilibrium band diagrams of metal-polar AlGaIn/GaN and N-polar GaN/AlGaIn/GaN heterostructures with the 2DEG indicated at the respective interfaces. For Ga-polar HEMTs structures, the 2DEG is located below the AlGaIn barrier layer, while in the N-polar ones, the 2DEG is positioned above the AlGaIn layer, which acts as a natural back barrier for the 2DEG [34, 51]. As exemplified in Fig. 1.3, the spontaneous and piezoelectric polarizations at the interface of GaN and AlGaIn cause a positive or negative sheet charge ($+\sigma$ or $-\sigma$), thus attracting electrons or holes, respectively [48]. Therefore, free electrons are confined at the interface of GaN/AlGaIn (with GaN channel grown on top of AlGaIn barrier) compensating the positive sheet charge in the case of N-polar heterostructures. In contrast, free electrons compensate the positive sheet charge at the interface of AlGaIn/GaN (with AlGaIn barrier grown on top of GaN channel) for metal-polar heterostructures. Theoretical and experimental works have shown that the 2DEG density can reach 10^{13} cm^{-2} and the built-in electric field can be in the order of MV/cm [58]. The 2DEG mobility in AlGaIn HEMTs can reach very high values exceeding $2500 \text{ cm}^2/\text{V}^{-1}.\text{s}^{-1}$ for sharp and compositionally uniform AlGaIn/GaN interface. Sharp interface is also required to maintain large electric fields and avoid current leakage of conductive polarization induced sheet charge [59]. Any

variation in alloy composition or thickness uniformity of the AlGaN barrier layer causes variation of the polarization induced band offset [48, 59]. Consequently, the 2DEG mobility can be significantly reduced due to the increase in interface roughness scattering [48].

N-polar GaN-based HEMTs structures have been intensively investigated [33, 55, 60–64]. Initially, AlGaN/GaN epitaxial heterostructure was developed with an AlGaN back barrier layer and a GaN cap layer. Large-signal dispersion due to interfacial hole traps at the negatively polarized interface was observed. Later, a graded AlGaN-back-barrier with ionized donors above the GaN-buffer layer was introduced to separate the trap levels from the Fermi energy level thereby providing the compensating positive charges for the negatively polarized interface [60]. Due to the low Schottky barrier height of the top GaN layer which causes a gate leakage, N-polar GaN-based HEMTs structures are usually capped with Al(Ga)N or *indium aluminum nitride* (InAlN) layer [55]. The cap typically has a wider bandgap to reduce the tunneling. Recently, N-polar GaN HEMTs exhibiting a record breakdown voltage over 2000 V combined with low dynamic on-resistance fabricated on epi-layers grown by *metal-organic chemical vapor deposition* (MOCVD) technique on sapphire substrates were reported [33]. Furthermore, high-performance HEMTs are achieved by employing a heterostructure with GaN/InGaN composite channel and superlattice back barrier [65, 66].

1.3 Epitaxy of N-polar III-Nitrides

1.3.1 History and challenges

N-polar GaN-based III-nitrides have been less explored due to the difficulties to obtain good quality N-polar epitaxial films. For instance, surface roughness of N-polar layers is typically large due to the presence of hexagonal hillocks formed regardless of substrates used (sapphire [67], SiC [51, 68] or even native GaN [69]). Besides, the control of polarity in N-polar III-nitride films and heterostructures is still very challenging [70]. Previously, it was reported that a pre-flow of metal precursors (e.g. *trimethylaluminum* (TMAI), $\text{Al}(\text{CH}_3)_3$, *trimethylgallium* (TMGa), $\text{Ga}(\text{CH}_3)_3$) or a low V/III (NH_3 /TMAI)-ratio flow [68, 71] would result in metal-polarity layers on *carbon-face* (C-face) SiC substrates instead of expected N-polar layers. Polarity inversion from metal-polar to N-polar or mixed polarity could be triggered by e.g., substrate treatment or deposition of a low-temperature buffer layer [72]. In addition, incorporation of impurities, such as oxygen or magnesium, at high levels is also known to cause polarity inversion.

The first high quality N-polar GaN films were demonstrated on C-face SiC substrate by *molecular beam epitaxy* (MBE) [73] in year 2000. Further improvement of N-polar GaN film quality has been achieved later by the MOCVD method. Hexagonal hillocks were

Figure 1.5: A simple schematic diagram of the hot-wall MOCVD system.

suppressed by using off-cut substrates of sapphire, SiC and GaN. This stimulated active research on developing of high quality N-polar III-nitride films for device applications. However, the step bunching causing high roughness is still an issue faced by technologists when vicinal substrates are used for the epitaxial growth [51]. Furthermore, due to the active chemical nature of N-polar III-Nitrides, incorporation of impurities, e.g. oxygen and carbon, in N-polar material is higher compared to the Ga-polar case. This has a negative impact on the intended electrical properties [51, 58]. Despite intense investigations, device-grade high-quality N-polar GaN epitaxial layers remain challenging.

1.3.2 Hot-wall MOCVD

1.3.2.1 Reactor set-up

MOCVD is a commonly employed technique to grow III-nitride films and device heterostructures. In a typical MOCVD system, the metal-organic precursors in vapour phase are introduced with carrier gases (normally *nitrogen* (N_2), *hydrogen* (H_2)) and the reactive species are transported toward a hot substrates on which epitaxial growth takes place. In this thesis, we employ a hot-wall MOCVD system to explore its advantages in growth of III-nitride layers with desired polarity. Figure 1.5 gives a schematic presentation of the hot-wall MOCVD system. It consists of several main parts: the sources or precursors (ammonia and *metal-organic* (MO) compounds) with H_2 and N_2 carrier gases; the reactor chamber including susceptor heating and cooling with a hot-wall design; the vacuum pumping system to get rid of impurities and maintain low pressure during epitaxy; the scrubber system, which deals with the exhaust gases from the reactor, and a digital control system to monitor the whole reactor with feedback signals connected to different compo-

Figure 1.6: A schematic illustration of the horizontal hot-wall MOCVD growth zone. The small round yellow part on the susceptor indicates the substrate sample. Precursors flowing in gas phase go through one side of horizontal quartz tube to the reactor zone. Reprinted from Ref. [75]

nents. The gas flow in MOCVD system is controlled by thermal *mass flow controller* (MFC) [74]. The metal-organic sources in liquid phase are stored inside bubblers immersed in separate temperature controlled thermal baths. They are also of sufficient volatility so that their vapor pressure is higher than the atmospheric pressure at room temperature. The H_2 carrier gas is controlled by electric MFC and goes through the metal-organic bubbler carrying the TMAI or TMGa out to the metal-organic source line. Ammonia goes through hydride runline together with the N_2 carrier gas into the hot-wall reactor. Metal-organic precursors are carried out by H_2 via MFC and diluted through MO runline. The hot-wall reactor is designed horizontally with gases inlet from one side and the exhaust from the other side. Turbomolecular pump is employed to provide good vacuum for the epitaxial process. It is well-known that oxygen from the residual gas is an unintentional donor in III-nitrides and its incorporation should be minimized.

Figure 1.6 shows a schematic illustration of the horizontal hot-wall MOCVD growth zone. In order to reach high temperature, *radio frequency* (RF) heater is equipped in this hot-wall reactor which allows to reach 1400°C . The graphite susceptor is coated by a thin layer of *tantalum carbide* (TaC) in order to prevent the reaction between ammonia or its decomposition products with graphite [76]. A rotating satellite (substrate holder) locates in the susceptor. This rotation can compensate for the process gas depletion in a linear profile and also assist to provide homogeneous temperature in order to generate uniform epitaxial layers. There are several advantages of using hot-wall MOCVD compared with

commonly employed conventional cold-wall MOCVD, in which only the substrate is heated. The hot-wall MOCVD uses a heated susceptor all around the substrate providing highly uniform temperature distribution [30, 77]. It also provides better cracking efficiency of the precursors which prevents growth-limited species consumption through gas-phase adduct formation [75]. Furthermore, high quality of device applicable III-nitrides materials has been successfully developed by hot-wall MOCVD [14].

1.3.2.2 General processes in MOCVD

In general, MOCVD is highly complex with multiple chemical and physical reactions governed by thermodynamics and kinetics [78]. Surface configuration, substrate orientation, precursor concentration, gas flow speed together with the process pressure play important roles for different aspects in the growth process. The epitaxial growth rate in MOCVD is mainly influenced by the process temperature which can be divided into three different temperature regimes [78, 79]. At low temperature, the reaction rates limit the growth rate, termed the kinetic limited growth. The growth rate increases as the temperature increases in this regime. At medium temperature, the growth rate is limited by the atom diffusion rate through a boundary layer and nearly independent of substrate temperature, termed the mass-transport limited growth. The boundary layer is formed due to the reactants concentration gradient from the vapour phase to the substrate surface vertically in the horizontal reactor. At high temperature, the growth rate decreases with increasing temperature as the material deposition rate is similar to the desorption rate because of the rising equilibrium vapor pressure of constituent elements in the epitaxial film. There are several key processes during MOCVD epitaxy illustrated in Figure 1.7 [78–81]. Precursors are transported from laminar gas flow to the substrates surface through the boundary layer. Chemical reactions and mass transport happen all the way. Adsorption and then diffusion of atoms on the substrate surface lead to the formation of nucleation centers first and then solid films. Possible desorption occurs at the surface and some atoms leave the surface moving to the vapour phase. Often, MOCVD epitaxy of III-nitrides take place at low pressures and high temperatures, which ensure significant lifetime and high kinetic energies of the reactive species. At low pressure (e.g. 10 torr), both kinetics and mass transport play a significant role in deposition [82]. For MOCVD GaN growth, a sequence of gas-phase reactions between TMGa and NH_3 precursors occurs resulting in various reaction paths, which depend on carrier gas composition and temperature, as well as the geometry of the MOCVD reactor [83–85].

In a horizontal reactor, the deposition uniformity and growth efficiency are affected by the mass transport. Typically, a high gas-phase V/III ratio with mixed H_2 and N_2 carrier gases is employed for the epitaxy of III-Nitrides films. Ammonia is the most commonly

Figure 1.7: Illustration of typical physical processes and chemical reactions involved in the epitaxial MOCVD growth. Reprinted from Ref. [80]

employed group-V precursor. High temperature is required for ammonia to have efficient pyrolysis and produce active nitrogen species on the growth surface because of the high N-H bond energies (~ 4 eV) comparable with the Ga-N bond strength [86, 87]. As the decomposition rate of ammonia is normally low under MOCVD conditions, excess of ammonia is required to provide sufficient surface reactive nitrogen species [88]. Koleske et al. [89] reported that nitrogen species are incorporated into the GaN lattice via an adsorption/desorption cycle during epitaxial growth because of the large desorption rate of nitrogen species. Under high concentration of ammonia, the GaN decomposition rate is enhanced in H_2 compared with N_2 carrier gases, due to the H_2 dissociation on the surface followed by ammonia formation and desorption [87, 90].

Furthermore, gas-phase parasitic deposition also limits the III-Nitrides epitaxial growth. This gas-phase parasitic chemical reactions lead to the formation of particles as a by-product between metal-organic sources and ammonia [86, 88]. In order to prevent extensive parasitic deposition, metal-organic source supply lines are separated from the ammonia line before these sources are introduced to the growth zone. The heated reactor wall (hot-wall) allows to reverse the adduct formation of metal-organic and ammonia [91] and reduce thermophoretic forces [92], thus suppressing the gas-phase parasitic chemistry and enhancing the growth efficiency. Lower growth pressure was also reported to reduce parasitic chemistry and improve crystalline quality of AlGaIn films [93].

1.3.3 Vacuum in MOCVD

1.3.3.1 Pumping system in MOCVD

In our hot-wall MOCVD system, a turbo-molecular pump is used, which transports gas by momentum transfer through the high-speed rotating blades. A backing pump typically a mechanical dry pump is required as a transition stage to keep the output pressure of turbo-molecular pump in the molecular regime. The backing pump is used as a backup stage for the turbo-molecular pump and it connects to the exhaust via a check valve. It pumps the exhaust to sub atmospheric pressure maintaining the pressure in a lower pressure range at the outlet of another pump and helping to reduce the pumping time for the next run. Another operating pump is the processing pump consisting of multi-stage Roots pump with a throttle valve to regulate the pressure (e.g. 50 mbar) during the epitaxy process. The chamber (reactor) pressure is maintained at a constant value by controlling the throttle valve. This throttle valve locates in the pumping line between the chamber and the processing pump. The throttle conductance is adjustable via pressure changing in a certain range to maintain the processing pressure at the setting value.

1.3.3.2 Gas flow in MOCVD

In general, vacuum can be described by three different type of physical state and its surrounding, which are pressure, gas composition and mean free path [94, 95]. Pressure is the force applied to a unit area of surface. It depends on the number of gas particles and kinetic energy distribution [94]. According to pressures ranges, vacuum is divided into three different levels [94]. First is the low and medium vacuum with pressure range between 10^{-3} torr to 760 torr. Second is the high vacuum with the pressure range between 10^{-7} torr to 10^{-3} torr. Third is the ultra-high vacuum with the pressure less than 10^{-7} torr. The gas composition can be used to describe vacuum state and it is strongly dependent on the pressure in an artificial vacuum [94]. The gas composition is close to atmospheric in the low and medium vacuum. In comparison, gas composition is dominated by *water* (H_2O) in high vacuum, while it is dominated by H_2 in ultra-high vacuum. In the work presented in this thesis the pressure before growth is pumped down to or below 10^{-5} torr in high vacuum range, indicating that the main gas composition is H_2O . However, the residual water will be purified during baking the cell in the growth process. The operation setting pressure is 50 mbar in low vacuum range.

Under vacuum condition, three state of gas flows can be differentiated by the Knudsen number (K_N), which depends on the mean free path (λ) and the cross geometrical dimension of a tube (L), with the relation of $K_N = \frac{\lambda}{D}$. One state of gas flow is viscous flow when the Knudsen number $K_N < 0.01$ [95]. The particles experience collisions frequently

and the gas behaves as a continuum. In viscosity flow regime, two different flow motion, laminar and turbulent respectively, are distinguished by Reynolds number (R_e) [95]. R_e is proportional to the velocity (v_0) and density of the gas (ρ) as well as the tube diameter (L), and it is inversely proportional to the viscosity of the gas (η):

$$R_e = \frac{\rho v L}{\eta}, \quad (1.2)$$

At laminar flow ($R_e < 1200$), the gas in a state flows smoothly in parallel streamlines with no cross flow with each other. While at turbulent flow ($R_e > 2200$), the gas flows irregular with random motion, but towards a main direction flow.

In hot-wall MOCVD epitaxy of III Nitrides, the total gas flow of Reynolds number is less than 100 in the laminar flow regime. The thickness of boundary layer (δ_x) (shown in Figure 1.8) at substrates position (x) along the flow direction is estimated by [82]

$$\delta_x \sim \frac{x}{R_e^{1/2}} \sim x \left(\frac{\eta}{\rho v_0 L} \right)^{1/2}, \quad (1.3)$$

Figure 1.8 shows the laminar viscous flow model through a horizontal tube reactor. The gas flow contains carrier gases (e.g. N_2 and H_2), precursors and their relevant reactant species. These gases go through the horizontal tube reactor from one side to the other over the substrate along the flow direction x . The reactants that are diffused to the substrate surface will be consumed and contribute to the film growth, and thereby, the gas concentration near the substrate surface is close to zero. Therefore, a gradient concentration boundary layer (shown in Figure 1.8) is generated resulting in a mass diffusion of molecules from a high concentration in the gas flow to the near-zero concentration at the substrate surface forming a precursor molecules flux ($J(x)$). This precursor flux plays an import role for the uniformity of the deposition and it depends on the local boundary layer thickness (δ_x) and its concentration gradient.

As shown in Figure 1.8, from the deposition model in the boundary layer, the substrate edge close to the gas inlet is exposed to the highest concentration flow but has the thinnest boundary layer resulting in the fastest deposition rate. In our reactor, a rotation is applied in order to achieve a uniform deposition. The thickness of the boundary layer (Eq. 1.3) is very important for the uniformity of teh epitaxial growth. If the thickness is too small, the mass transportation by precursor flux ($J(x)$) from the gas phase to the boundary layer will be limited, which results in a low growth rate. From Eq. 1.3, it is evident that one way to increase the thickness of this boundary layer is to decrease the gas density or flow velocity, or to narrow the tube reactor dimension (e.g. L). Another way could be to reduce the operating pressure. As the precursors are mixed, it is important to minimize the turbulent flow but keep in laminar flow regime which can diminish molecular collisions in the

Figure 1.8: Schematic diagram of the laminar viscous flow model through a horizontal tube reactor.

vapour phase during mass transport.

1.3.4 MOCVD of N-polar AlN and GaN layers

Epitaxial N-polar GaN layers grown by MOCVD have been demonstrated on various substrates, GaN [69], sapphire [96] and SiC [97]. In early works, the presence of hexagonal hillocks was a persisting problem even in the case of homoepitaxial GaN. In 1999, Zauner et al. [69] showed that the hillock formation can be suppressed for homoepitaxial GaN by using misoriented substrates. In 2007, Keller et al. [96] reported that the density of hexagonal hillocks could be significantly decreased by increasing the misorientation angle of the substrate up to 4° for the case of sapphire. Brown et al. [97] observed the same phenomenon of suppressed hexagonal hillocks for the growth of N-polar GaN by using vicinal C-face SiC substrates. Although the formation of hexagonal hillocks could be suppressed by using vicinal substrates, the roughness of N-polar GaN surface still remains high due to the undulation/steps parallel to the misorientation direction [96]. Recently, in order to improve the N-polar GaN crystalline quality, various growth approaches have been explored. A two-step process for N-polar GaN film growth was adopted, in which a thin initial GaN layer was grown at a reduced temperature, and then followed by a thick high-temperature GaN buffer [97]. Through the experimental optimization, the formation of both large macrosteps and hexagonal hillocks was greatly suppressed by using this two-step GaN growth method resulting in an improved N-polar GaN quality and structural and morphological properties. Deng et al. further improved the N-polar

GaN films quality and surface morphology by using in-situ SiN_x interlayers [98] and optimization of the V/III ratio [99].

Another major challenge of N-polar III-nitride epitaxy is polarity inversion. It was reported that the polarities of GaN and AlN layers were highly dependent on the pre-treatment of C-face SiC substrates [71]. A pre-flow of TMAI on the surface of C-face SiC substrate before AlN nucleation layer growth, or a very low NH_3 /TMAI ratio at the initial growth stage of AlN result results in inversion to *aluminum-polar* (Al-polar) AlN or Ga-polar GaN. This effect is attributed to the formation of *aluminum* (Al) adlayers [71]. Similarly, Keller et al. [68] reported that for low NH_3 /TMAI ratio during AlN growth, polarities inversion in both AlN and GaN-on-AlN films takes place. Won et al. [100] also reported polarity inversion domains in AlN nucleation layers. The authors further argued that the hexagonal hillocks cannot be completely suppressed by using vicinal substrates, but instead their density can be significantly reduced by increasing the NH_3 /TMAI ratio and by decreasing the thickness of the AlN nucleation layers. MOCVD of N-polar III-nitrides epilayers on on-axis C-face SiC substrates, which is highly desirable for device fabrication on industrial scale, has not yet been achieved and it remains a significant challenge. Therefore, detailed understanding of the factors governing polarity control and polarity inversion on both on- and off-axis substrates is needed in order to further the progress in fabrication of high-quality device-relevant N-polar GaN epitaxial layers. Novel and superior growth methods, such as hot-wall MOCVD provide possibility to expand growth windows facilitating polarity control and enables high-quality N-polar epitaxial growth of III-Nitrides, as demonstrated in this thesis.

1.4 Characterization techniques

1.4.1 Optical Microscopy

Optical microscopy is a fast, convenient and efficient technique which can be used to evaluate the outcome of an epitaxial growth. It gives quick feedback on the surface morphology, films uniformity, and surface morphological defects of epitaxial films. *Nomarski differential interference contrast* (NDIC) microscopy is used to characterize the epitaxial films presented in this thesis. The NDIC microscope [101] employs dual-beam interference optics and special beam splitting prisms. It contains two polarizers with one polarizer in front of a condenser producing light polarized parallel to the plane and one analyzer located near the objective back aperture transmitting the light from the object. The transmitted and incoming beams interfere and generate an image in the image plane. One condenser Nomarski prism is mounted between a condenser and polarizer and one objective Nomarski prism is mounted between the objective and the analyzer. The condenser Nomarski

Figure 1.9: Typical principle of AFM. Tip is mounted at the end of cantilever acting as scanning probe. Enlarged view of tip reveal the atomic force between tip and surface atoms. A laser beam is aligned on top of tip monitoring the motion of cantilever. Reflected laser beam will be collected by photodetector and thus to the feedback control.

prism works as a beam splitter, which can generate two closely spaced parallel beams and pass through the object. The objective Nomarski prism located close to the back of the objective lens combines the two parallel beams for interference and image formation. This forms conjugate interference planes between the two Nomarski prisms. The differential interference between the two planar beams generates high contrast patterns between the edge of the object and the surrounding medium.

1.4.2 Atomic force Microscopy

Atomic force microscopy (AFM) is a widely adopted characterization tool by researchers to obtain surface images down to atomic resolution. As show in Fig. 1.9, there are several core components in AFM [102, 103]: a sharp tip (or probe) is attached to a cantilever operating like a spring which can be bent upward or downward towards the sample surface, a laser beam shining on the cantilever, which reflects bending of the cantilever and is fed back to a photo-detector. Through a feedback control loop, the tip keeps operating and the feedback signal is sent to a processing software which records a three-dimensional topography of the surface. There are three operation modes of AFM: contact mode, non-contact mode and tapping mode, which can be distinguished by the interaction forces between the tip and the surface atoms.

Figure 1.10: Schematic representation of X-ray diffraction experiment exemplifying Bragg's law. The X-rays are diffracted from crystal lattice planes with an interplanar distance d . The incident X-rays angle (θ) is equal to the angle of the diffracted X-rays beam. The path-length difference of two diffracted beams is $2d \cdot \sin\theta$.

In the work presented in this thesis, an Veeco Dimension 3100 AFM instrument is employed in tapping mode. In tapping mode, a set-point amplitude of cantilever oscillation is required to excite the probe. The probe amplitude can be adjusted from full oscillation to smaller oscillations by the intermittent contact with the surface structure. Due to the fluctuation of the surface structure, the amplitude of the probe will change and be stored through feedback control generating the structural information of the surface in a three-dimensional topography.

AFM gives quick and direct feedback on the topography and surface morphology of the fabricated III-nitrides films. This information is important for further understanding and optimization of growth process. For example, different crystal growth modes such as island growth mode, two-dimensional growth mode, and step-flow growth mode can be inferred based on AFM images. In addition, surface defects, dislocations, pits, voids, or hexagonal hillocks could also be easily identified by AFM.

1.4.3 High-resolution X-ray Diffraction

High-resolution X-ray diffraction (HR-XRD) is a non-destructive and efficient technique commonly employed for the characterization of epitaxial films. HR-XRD can be used to obtain information on crystallographic orientation, texture, thickness, lattice parameters, alloy composition, strain and defect densities, for example [104–109].

In HR-XRD x-ray radiation is generated by bombarding a metal (typically *cooper*

(Cu)) with electrons in an evacuated tube. Monochromator crystals are used to obtain monochromatic radiation and condition the beam. When the incident x-ray beam passes through the crystal, it is scattered by the electron cloud around each atom. Fig. 1.10 shows a schematic representation of the diffraction process in a crystal. When the path difference between the diffracted beams from a set of given planes with interplanar distance d , is equal to $n\lambda$, constructive interference occurs:

$$n\lambda = 2d \cdot \sin\theta, \quad (1.4)$$

where n is the order of the diffraction. This is the so-called Bragg's law. The wavelength of the incident monochromatic beam λ and the incident beam angle θ are known. In all experiments presented in this work monochromatic Cu K_α radiation with wavelength $\lambda = 1.5406 \text{ \AA}$ is used. For wurtzite hexagonal crystal system, a four Miller index notation $\{h k i l\}$ (with i equal to $-(h+k)$) is used to describe individual planes with interplanar distance d . Thus, by measuring the so called, $2\theta - \omega$ scan one can obtain experimentally the diffracted angle θ and calculate the lattice parameters. The angle 2θ refers to the angle between the incident and diffraction beams, while ω is the incident angle and it is equal to θ . During the measurement, the detector is rotated by 2θ while the incident angle, ω , varies with an angular ratio of 2:1. The $2\theta - \omega$ peak broadening could be related to finite film thickness and heterogeneous strain. Rocking curve measurement, or so called ω scan is performed by rocking the sample around the ω axis (incident beam) while the detector is kept stationary. The broadening of the rocking curve provides information on overall crystal quality in view of tilt, twist and dislocation density. In this thesis, symmetric (0002) and skew (1 0 $\bar{1}$ 2) or (1 0 $\bar{1}$ 5) rocking curves and $2\theta - \omega$ scans are used to evaluate the crystalline quality of the III-nitride epitaxial layers and heterostructures.

1.4.4 Scanning Electron Microscopy

Scanning electron microscopy (SEM) is a commonly employed technique for characterization of sample topography. Fig. 1.11 shows a schematic presentation of beam-sample interactions within SEM. Typically, the focused electron beam is emitted by electron gun and accelerated to a selected energy range from 0.1 to 30 keV. The interaction of the electron beam with the specimen produces two important signals for image recording which are *backscattered electrons* (BSE) and *secondary electrons* (SE). These two types of signals are produced and collected by corresponding detectors mounted at different positions above the surface of the specimen (see Fig. 1.11). The size of the interaction volume depends on the incident electron beam energy, the atomic weight, the atomic number and the density of the components in the sample.

Figure 1.11: Schematic presentation of beam-sample interactions within SEM. A focused incident electron beam scans (indicated in orange) on the surface of a specimen (indicated in green) mounted on the sample stage (indicated in grey). The backscattered electron detector (dark blue) is installed directly above the sample, while the secondary electron detector (light blue) is installed on the top side of the sample.

In this study, we use a low-voltage SEM operating at 3 keV with the beam incident perpendicular to the specimen surface (no tilt) imaging in-lens backscattered electrons. A certain fraction of the incident beam electrons go through sufficient scattering events and escape to the entrance surface with reversed direction comparing with their initial travel route into the specimen. The escaped electrons are referred to as backscattered electrons [110]. The BSE signal can give information on the specimen composition, topography, mass thickness and crystallography. The BSE signal is proportional to the atomic number from the specimen and also the incident electron energy. If higher incident electron energies (high-voltage mode) are used, the BSE signal will be enhanced but it will also contain backscattered electrons from deep in the sample.

Because of their wide bandgaps, both III-nitride epilayers and the SiC become significantly charged during an SEM experiment. Thus, we use liquid silver to connect the sample surface with the copper tape on the back of the sample in order to drag the excess electrons by providing an electrical discharge path. Cross-section images of multilayer specimens

can also be investigated for layer thickness estimation and evaluation of interface quality.

1.4.5 Transmission Electron Microscopy

Aberration corrected *transmission electron microscopy* (TEM) is a powerful technique which uses an electron beam passing through a thin specimen and forming images for sub-Ångström and atomic characterization of materials. The electrons are emitted in the electron gun and accelerated to the operating energies ranging typically from 30 keV to 300 keV. These accelerated electrons are focused by electromagnetic lenses and apertures to an electron beam interacting strongly with atoms inside the specimen by elastic and inelastic scattering. In general electromagnetic lenses are subject to severe electromagnetic aberrations, much more so than visible light lenses. Aberration correctors can however be used to apply a conjugate aberration to some extent cancel out the original aberrations and therefore provide atomic resolution. The specimen must however be very thin, typically between 5 nm to 100 nm for a 100 keV to be electron transparent, but it also depends on the elemental composition and the density of the object. Normally, a time-consuming sample preparation is needed for TEM investigation involving sample polishing and ion-beam milling procedures.

In the *scanning transmission electron microscopy* (STEM) mode, an atomic-scale electron probe is formed by the electromagnetic lens system. A variety of scattered signals can be detected in the STEM [111, 112] including (i) transmitted electrons through the specimen with smaller scattering angles compared with the incident beam convergence angle referred to as bright field; (ii) transmitted electrons through the specimen with larger scattering angles compared with the incident beam convergence angle referred to as annular dark field; (iii) the intensity of transmitted electrons with energy losses can be recorded leading to the *electron energy loss spectroscopy* (EELS); (iv) X-rays generated from core electron excitations can be detected and referred to as *energy-dispersive x-ray* (EDX). EDX and EELS are analytical techniques that gives information on elemental composition and bonding characteristics in the specimen. If the inner scattering angle of the annular dark field detector is increased to a high scattering angle ($> 3^\circ$ or > 50 mrad), it would enhance the compositional contrast from single atoms and coherent effects of elastic scattering could be neglected as the scattering is mostly incoherent and thermally diffuse [113]. Thus, this leads to the use of the *high-angle annular dark-field* (HAADF) detector. In HAADF STEM mode, Z-contrast (where the intensity of each column depends on the average atomic number of the atoms in the column) dominates the contributor to image contrast formation. The HAADF can give direct interpretation of atoms types, atomic positions and arrangement. Normally, STEM image simulations are required to compare with the quantitative analysis of HAADF STEM images to interpret the effect of detection angles

and thermal diffuse scattering.

1.5 Polarity determination

Various techniques have been used to determine the polarity of group III-nitrides. *X-ray photo-emission spectroscopy* (XPS) [72], which probes the surfaces of the films by studying the energy shifts of the Ga- and N- core-levels. Similarly, by *angle-resolved x-ray photo-electron diffraction* (ARXPD) [46], one measures the different angular distribution of the photoemission intensity for anion- and cation-polar layers in wurtzite crystals as the relative positions of the cation and anion sublattices are distinct. Thereby, different intensities can be observed from the forward elastically scattering of electrons by neighboring anions and cations atoms in crystalline structures resulting in differentiation between two polar orientations. *Coaxial impact collision ion scattering spectroscopy* (CAICISS) [46, 72] measures the intensity of the recorded ions (or neutral particles) scattered by atoms in the crystalline lattice as a function of the incidence detecting angle with respect to the surface plane. The CAICISS is able to analyze the atomic arrangement on the surface, determine the bond orientation and bond length between neighboring atoms. Therefore, the polarity can be identified with CAICISS, however comparison to a simulated spectra or a measured signal to a reference sample with known polarity is required. *Scanning Kelvin probe microscopy* (SKPM) [46] enables mapping of the polarity of the surface by providing the contact potential difference (work function) between the metallic tip above and the semiconductor surface. The observed band bending which relates to work function depends on the surface polarity charges and is also very sensitive to the measurement conditions due to adsorption or contamination, effectively screening the local electric fields. All these polarity determination techniques are non-destructive but very sensitive and highly dependent on the surface of the films. However, the information acquired from the techniques described above comes from the surface only, meaning that the polarity of the films below the surface is unknown. In case of the mix-polarity below the surface, it is also difficult to distinguish local polarities, especially for small inversion domains.

Some destructive techniques are also developed to assess the polarity of group III-nitride films. The *potassium hydroxide* (KOH) wet chemical etching [46] is widely employed as it is simple and fast method. The polarity can be determined from the change in surface morphology as a function of etchant, temperature, and etching time and concentration, because the chemical natures of the metal-polar and N-polar surfaces are substantially different [46, 114, 115]. The N-polar surface generally undergoes significant changes after KOH etching. The hexagonal hillocks, typically present in N-polar layers, are etched through their sidewalls resulting in very rough surface morphology. In contrast, metal-

Figure 1.12: An example of polarity determination via SEM and HR-XRD for an N-polar GaN epitaxial layer on AlN/ 4° off-axis SiC (000 $\bar{1}$) obtained in this work [unpublished]. SEM images of (a) as-grown GaN surface, (b) the same surface after etching in KOH solution for 10 seconds and (c) after 10 minutes of etching resulting in complete removal of the N-polar GaN. (d) HR-XRD radial scans in the vicinity of the AlN and GaN (0002) peaks for the as-grown GaN (solid line) and after 10 min of KOH etching (dashed line).

polar surfaces remain smooth after KOH etching [114–116].

For metal-polar film, the metal-(Al-, Ga-) atoms on the surface can be removed leaving the surface covered by nitrogen termination. The hydroxide ions (OH^-) in the KOH solution experience repulsion by the three nitrogen atoms with dangling bonds rendering the metal-polar surface resistant to the wet chemical etching. In contrast, for N-polar film, the OH^- attack the metal (Al-, Ga-) atoms in the three bonds with only a single dangling bond on the top. The released OH^- ions will be adsorbed on the surface and form gallium oxide which is subsequently dissolved by KOH solution. Therefore, continuous etching of N-polar surface will take place. Consequently, the N-polar surface has much higher etching rate compared with the metal-polar surface, for which the etching rate is extremely low [115, 116]. Typically, the etching rate of AlN is more than ten times higher than the etching rate of GaN under the same etching conditions. This is due to the difference in the activation energies of wet etching of GaN (0.59 eV) and AlN (0.67 eV) [115].

TEM is another destructive technique which can be employed to determine the polarity of III-nitride films with a high spatial resolution using *convergent-beam electron diffraction* (CBED), EDX and STEM. CBED is a well-established method to confirm the polarity of

Figure 1.13: Some examples of polarity determination for mix-polar GaN epitaxial films obtained in this work on on-axis and 4° off-axis SiC (000 $\bar{1}$) [unpublished]. SEM images of (a) GaN surface after 10 minutes etching for growth on on-axis SiC, (b) the same as in (a) but for growth on 4° off-axis SiC and (c) the same as in (b) but in the region from the edge of the sample. Respective HR-XRD radial scans in the vicinity of the AlN and GaN (0002) peaks for the as-grown GaN (solid lines) and after 10 min of KOH etching (dashed lines): (d) corresponding to the sample shown in (a); (e) corresponding to the sample shown in (b) and (f) corresponding to the sample shown in (c).

(Al,Ga)N epitaxial films [68, 96, 97, 100]. However, CBED has a relatively large probe size and therefore cannot provide local information, e.g., on inversion boundaries or mixed domains [46]. The scattered intensity is strongly dependent on the thickness, thus the simulated dynamic electron-diffraction pattern vs thickness needs to be recorded and compared with experiment and calculations in order to identify the polarity. Therefore, this technique becomes challenging for very small dimensions of the films. With a HAADF detector for z-contrast imaging, the STEM [46] can reach an angstrom resolution and observe a direct arrangement of individual atoms in the film. Therefore, this technique allows a direct polarity identification in the unit cell dimension and the corresponding simulation provides the positions of individual atoms and their bonding length.

We have proposed a combination of KOH wet chemical etching, HR-XRD and SEM for the polarity evaluation of III-Nitride epitaxial layers and heterostructures [117]. In this approach, sufficient etching conditions (e. g. time, temperature, concentration) are employed in order to etch away the N-polar layers [117] as contrasted to only observing change of morphology [99]. Our method has also the potential to verify metal-polar inclusions in mixed-polar layers, which could remain after the N-polar material is etched away and hence detected by XRD. This method is used throughout the work presented in

the thesis.

Examples of polarity determination via the combined KOH etching and HR-XRD approach described above are shown in Figure 1.12 for N-polar and in Figure 1.13 for mixed-polar GaN layers obtained in this work. The surface topography of the as-grown, N-polar GaN surface and after 10 s and 10 min of KOH etching are shown in Fig. 1.12 (a), (b) and (c), respectively. It is seen that after 10 s of KOH etching, the GaN surface becomes significantly rougher, which is consistent with the etching behavior reported previously for N-polar surfaces [114–116]. The 10 min etching conditions are selected to ensure complete removal of both the GaN and the AlN-NL, should they be N-polar. Indeed, the SEM image in Fig. 1.12 (c) reveals very smooth surface similar to bare SiC substrates, indicating that the layers have been etched away. This is confirmed by the respective HR-XRD $2\theta - \omega$ scans in the vicinity of the AlN and GaN (0002) peaks of the as-grown (black solid line) and KOH etched layer (Fig. 1.12 (d)). It is seen that neither the GaN nor the AlN (0002) diffraction peaks could be detected after 10 min KOH etching.

In the case of layers with mixed polarity, it becomes less straightforward to conclusively determine the polarity. This is exemplified in Figure 1.13 with three different GaN samples grown under different conditions. The GaN layers display different surface topographies after 10 min of KOH etching but all are of mixed-polarity. The surface of the GaN grown on on-axis SiC (000 $\bar{1}$) (Fig. 1.13 (a)) reveals large irregularly shaped GaN islands after etching. In contrast, after etching a GaN layer grown on 4° off-axis SiC (000 $\bar{1}$), only very tiny islands with tetragon and pentagon shapes could be seen [Fig. 1.13 (b)]. The sample in Fig. 1.13 (c) reflects a third situation of mix-polar GaN, in which the layers were completely etched away in the central area of the sample, while most of the GaN layer at the edge remain after 10 min of KOH etching. The respective HR-XRD $2\theta - \omega$ scans in the vicinity of the AlN and GaN (0002) peaks of the as-grown (black solid line) and KOH etched layers are shown Fig. 1.13 (d-f). Based on the combined HR-HRD and SEM we have concluded that the first GaN layer (Fig. 1.13 (a and d)) has mix-polarity, the second GaN layer (Fig. 1.13 (b and e)) has N-polar dominated mix-polarity, and the third GaN (Fig. 1.13 (c and f)) is in fact N-polar, since the Ga-polar regions could only be detected in the edge areas of the sample.

It is obvious, that although using KOH etching combined with HR-XRD provides improved method for polarity determination on a macro-scale, it is not sufficient for conclusive assignment. In particular, when striving to suppress IDs and control polarity via growth strategy optimization. Therefore, in our work we have extensively employed STEM for investigating the atomic arrangements and local polarity evolution (**Papers II and III**).

Figure 1.14: AFM images of the AlN NLs grown at different temperatures: (a), (b), (c) - 850 °C; (d), (e), (f) - 950 °C and (g), (h), (i) - 1050 °C. For each temperature the growth is performed simultaneously on three types of SiC substrates: (a), (d), (g) C-face SiC on-axis; (b), (e), (h) C-face SiC 4° off-axis; (c), (f), (i) Si-face SiC on-axis. The root-mean-square (RMS) roughness of the AlN NLs are indicated on each image. The scale bar of 1 μm indicated in (h) refers to all images.

1.6 Summary of main results

In this thesis, we focus on the development of (N)-polar group-III nitride epitaxy by hot-wall *metal-organic chemical vapor deposition* (MOCVD) for *high electron mobility transistor* (HEMTs) applications. As a key component for heteroepitaxy of GaN on SiC, first the growth and polarity control of AlN nucleation layers (NLs) was established. We have explored growth conditions and different substrate orientations and off-cut angles to build a comprehensive picture of respective growth mechanisms in connection with polarity, structural quality and surface morphology. We have shown that with increasing growth temperature the growth mode changes from island-like to step-flow on off-axis SiC (000 $\bar{1}$) and to layer-by-layer on SiC (0001), respectively. In contrast, island-like mode is preserved when AlN NLs are grown on on-axis C-face SiC independent of growth temperature up to 1050 °C (Fig.1.14). N-polar AlN nucleation layers with smooth surfaces and good crystalline quality that can be employed for subsequent growth of N-polar device heterostructures

Figure 1.15: AFM images of the AlN NLs grown at higher temperatures from 1100 °C to 1400 °C at constant V/III ratio of 800 on on-axis (upper row) and 4° off-axis (lower row) C-face SiC. The root-mean-square (RMS) roughness of the AlN NLs are indicated on each image. The scale bar of 1 μm refers to all images.

are demonstrated for the first time by hot-wall MOCVD in **Paper I**.

We further optimized growth conditions, by investigating higher growth temperatures (HT) and V/III ratios as illustrated in Figs. 1.15 and 1.16. As a result, smooth (RMS below 1 nm) HT N-polar AlN NLs with a step-flow growth mode are achieved at 1300 °C on 4° off-axis SiC (000 $\bar{1}$). On on-axis SiC (000 $\bar{1}$) growth at higher temperature of 1400 °C was needed to transition to layer-by-layer two dimensional growth mode. However the surface roughness in this case remained relatively high and detailed STEM investigation revealed that the AlN layer is of mixed polarity (**Paper III**).

As a next-step focused efforts have been directed to develop N-polar GaN with optimized surface morphology and crystalline quality on HT N-polar AlN NLs on 4° off-axis SiC (000 $\bar{1}$). Maintaining N-polarity in GaN requires much higher V/III ratios as compared to the case of AlN. This represent a particular challenge for preserving smooth interface between AlN and GaN, which is important in view of subsequent growth and device performance as a rough interface causes additional thermal boundary resistance and leakage. We have demonstrated that by employing HT AlN NLs with a step-flow growth mode smooth AlN/GaN interface can be realized (**Paper III**). However, the high V/III ratio for N-polar GaN buffer growth, results in a low growth rate of 1.4 nm/min, which is not optimal.

Minimizing impurity incorporation is another important aspect for achieving good device quality material. Typically, higher growth temperatures are required to reduce unintentional impurities such as, O, Si and C in MOCVD. Indeed we found that increasing growth temperature from 850 °C to 950 °C for typical growth condition of N-polar GaN,

Figure 1.16: AFM images of the AlN NLs grown at different V/III ratios of 800, 1600 and 3200 at constant temperature of 1300 °C on on-axis (upper row) and 4° off-axis (lower row) C-face SiC. The root-mean-square (RMS) roughness of the AlN NLs are indicated on each image. The scale bar of 1 μm refers to all images.

leads to reduction of C levels by one order of magnitude to $1 \times 10^{18} \text{ cm}^{-3}$. Even higher temperatures are required for the GaN channel layer to further reduce the impurities. However, this presents yet another challenge for N-polar GaN, as surface roughness increases significantly as the growth temperature increases, as exemplified in Fig. 1.17 for GaN grown at 850 °C, 900 °C and 1000 °C.

To address these conflicting demands, we have developed a two-step temperature growth strategy for N-polar GaN on LT-GaN buffer layers. Using this approach we succeeded in increasing the growth rate from 1.4 nm/min to 19 nm/min while preserving N-polarity and surface roughness, as demonstrated in Fig. 1.18. We further optimized the HT GaN main layer grown at 1000 °C with thickness up to 1.5 μm , with reduced roughness of 3.1 nm and improved crystalline quality.

A key to develop appropriate growth strategy for N-polar III-Nitrides is to reliably determine the polarity of epitaxial layers and device heterostructures. Therefore, we have critically evaluated the applicability of standard methods for determination of polarity, such as KOH wet chemical etching in combination with XRD and SEM by extensive STEM studies of varieties of polarity and surface morphology situations (**Paper II**). We have shown that there are limitations in solely relying on KOH etching for polarity determination, in particular mixed-polar layers consisting of metal-polar and N-polar domains. In particular, pyramidal N-polar domains enveloped in Al-polar regions could be easily over-etched and remain undetected by KOH etching. When mixed type of Ga-polar and N-polar GaN layers form under competitive growth, the resulting two different morphology types are easily detected by AFM and SEM. In such instances, KOH etching

Figure 1.17: AFM images of N-polar GaN buffer layers grown with HT AlN NLs on 4° off-axis SiC (0001) at different temperatures: (a) 850 °C, (b) 900 °C, and (c) 1000 °C. The values for the RMS roughness are indicated on the respective images.

Figure 1.18: AFM images of N-polar GaN using LT GaN buffer on 4° off-axis SiC (0001) with different growth rates (a) 1.4 nm/min (b) 7.5 nm/min and 12 nm/min in (c). The root-mean-square (RMS) roughness of GaN buffer layers are indicated in the respective images.

could provide adequate indication of polarity on a macro scale (**Paper II**).

Last but not least we have also developed N-polar AlGaIn and demonstrated high quality N-polar epitaxial AlGaIn/GaN/AlN structures (Fig.1.19) by suppressing polarity inversion domains in the nucleation layers (**Paper III**). We further established the detailed polarity evolution for GaN grown with low- and high-temperature NLs for both on- and 4° off-cut 4H-SiC (0001), which can provide guidelines for polarity control and further growth strategy optimization (**Paper III**). Future work will be dedicated to further improve surface roughness and optimize the properties of the top GaN channel for the demonstration of 2DEG and N-polar GaN HEMT device fabrication.

Figure 1.19: Cross-section STEM images of N-polar AlGaN/GaN/AlN structure on 4° off-axis SiC ($000\bar{1}$) with magnified interface regions revealing sharp AlN/SiC, GaN/AlN and AlGaN/GaN interfaces.

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List of abbreviations

AFM	<i>Atomic force microscopy</i>
Al	<i>aluminum</i>
AlN	<i>aluminum nitride</i>
AlGaN	<i>aluminum gallium nitride</i>
EELS	<i>electron energy loss spectroscopy</i>
Ga	<i>gallium</i>
N	<i>nitrogen</i>
GaN	<i>gallium nitride</i>
HEMTs	<i>high electron mobility transistor</i>
InAlN	<i>indium aluminum nitride</i>
InN	<i>indium nitride</i>
TEM	<i>transmission electron microscopy</i>
Si	<i>silicon</i>
SiC	<i>silicon carbide</i>
STEM	<i>scanning transmission electron microscopy</i>
2DEG	<i>two-dimensional electron gas</i>
LEDs	<i>light-emitting diodes</i>
LDs	<i>laser diodes</i>
IR	<i>infrared</i>
UV	<i>ultraviolet</i>
PDs	<i>photodetectors</i>
HP	<i>high power</i>

HF	<i>high frequency</i>
RF	<i>radio frequency</i>
GaP	<i>gallium phosphide</i>
GaAs	<i>gallium arsenide</i>
MBE	<i>molecular beam epitaxy</i>
MOCVD	<i>metal-organic chemical vapor deposition</i>
N-polar	<i>nitrogen-polar</i>
Ga-polar	<i>gallium-polar</i>
DFT	<i>density-functional theory</i>
hcp	<i>hexagonal close packed</i>
C-face	<i>carbon-face</i>
NH₃	<i>ammonia</i>
TMAI	<i>trimethylaluminum</i>
TMGa	<i>trimethylgallium</i>
N₂	<i>nitrogen</i>
H₂	<i>hydrogen</i>
MO	<i>metal-organic</i>
MFC	<i>mass flow controller</i>
TaC	<i>tantalum carbide</i>
H₂O	<i>water</i>
Al-polar	<i>aluminum-polar</i>
XPS	<i>X-ray photo-emission spectroscopy</i>
ARXPD	<i>angle-resolved x-ray photo-electron diffraction</i>
CAICISS	<i>Coaxial impact collision ion scattering spectroscopy</i>
SKPM	<i>Scanning Kelvin probe microscopy</i>
KOH	<i>potassium hydroxide</i>
CBED	<i>convergent-beam electron diffraction</i>
EDX	<i>energy-dispersive x-ray</i>
HAADF	<i>high-angle annular dark-field</i>
HR-XRD	<i>High-resolution X-ray diffraction</i>

SEM	<i>Scanning electron microscopy</i>
NDIC	<i>Nomarski differential interference contrast</i>
Cu	<i>cooper</i>
BSE	<i>backscattered electrons</i>
SE	<i>secondary electrons</i>

Part II

2.1 Publications included in the thesis

Paper I

Zhang, Hengfang, Plamen P. Paskov, Olof Kordina, Jr-Tai Chen, and Vanya Darakchieva
"N-polar AlN nucleation layers grown by hot-wall MOCVD on SiC: Effects of substrate orientation on the polarity, surface morphology and crystal quality."
 Physica B: Condensed Matter 580, 411819 (2020).

Contribution: I was involved in the experimental design and I have performed all epitaxial growth runs. I also performed the sample characterizations and data analysis presented in this paper. I wrote the first draft of the paper.

Paper II

Zhang, Hengfang, Ingemar Persson, Alexis Papamichail, Jr-Tai Chen, Per O. Å. Persson, Plamen P. Paskov, and Vanya Darakchieva
"On the polarity determination and polarity inversion in nitrogen-polar group III-nitride layers "
 Manuscript in final stage of preparation

Contribution: I was active in designing this study and I performed all epitaxial growth and XRD, AFM, SEM and KOH etching experiments. I was involved in the TEM data analysis and interpretation of the results. I wrote the first draft of the manuscript.

Paper III

Zhang, Hengfang, Ingemar Persson, Plamen P. Paskov, Per O. Å. Persson, Jr-Tai Chen, and Vanya Darakchieva
"Polarity control by inversion domain suppression in N-polar III-Nitride heterojunctions"

In draft manuscript

Contribution: I was active in the study plan and I performed the epitaxial growth and XRD, AFM, SEM and KOH etching experiments. I actively participated in the TEM data analysis and interpretation. I wrote the parts on growth, and XRD, KOH, AFM and SEM analysis in the manuscript.

2.2 Publications not included in the thesis

Dat Q. Tran, Nicholas Blumenschein, Alyssa Mock, Pitsiri Sukkaew, Zhang, Hengfang, John F. Muth, Tania Paskova, Plamen P. Paskov, and Vanya Darakchieva
"Thermal conductivity of ultra-wide bandgap thin layers - High Al-content AlGa_N and β -Ga₂O₃"
Physica B: Condensed Matter 579, 411810 (2020)

Papers

The papers associated with this thesis have been removed for copyright reasons. For more details about these see:

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