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# Lowering the density of dislocations in heteroepitaxial III-nitride layers: Effect of sapphire substrate treatment (review)

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**Abstract.** In this paper, different methods for lowering the dislocation density such as incorporation of the buffer layers, substrate patterning and nitridation, silan-ammonia treatment are reviewed and compared. Advantages and limitations of these methods as well as a specific mechanism to reduce the dislocation amount are discussed. Usually, high densities of threading dislocations within the range $10^{10}…10^{11}$ cm$^{-2}$ are present in typical thin nitride films that are directly grown on sapphire substrate. Using these methods for substrate preparation, the density of dislocations can be reduced to the value $1·10^{7}$ cm$^{-2}$. An important process that enables to obtain the high-quality GaN layers with the low dislocation density is patterning the sapphire substrate. The dislocation density of these substrates depends on the pattern shape and orientation of patterned strips on *c*-plane sapphire. Layers of GaN grown on a cone-shaped pattern have the lowest dislocation density. In addition, patterning the sapphire substrate increases the external quantum efficiency of radiative structures and reduces the mechanical stresses in the nitride layers.

**Keywords:** GaN layer, sapphire substrate, density of dislocations, reduction, patterning, treatment.

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## 1. Introduction

Wide bandgap semiconductors, such as GaN, are very promising for novel applications in solid state and vacuum nanoelectronic devices [1-3]. The large bandgap of group-III nitrides and as well as chemical and thermal stability allow new applications. Also important are also the advanced characteristics, namely: high saturation drift velocity of electrons close to $3·10^{7}$ cm/s [4] and extremely high breakdown fields $(3…5)·10^{6}$ V/cm [5]. Furthermore, these materials find their application for various sensor concepts because of their large piezoelectric coefficients and robustness in harsh environments.

Due to the lack of commercially available large size native substrates, lowering the density of threading dislocations remains one of the main challenges for group III-nitride-based technology. This paper reviews major methods aimed at producing high-quality heteroepitaxial nitride films with a reduced density of dislocations. The following methods, namely: incorporation of buffer layers, substrate nitridation, silane-ammonia treatment, substrate patterning, are reviewed. Advantages and drawbacks of these methods are discussed and compared.

One of the main problems in the group III-nitride-based technology is the lack of commercially available large-size native substrates. Sapphire or 6H-SiC substrates are typically chosen for most of nitride-based applications. These substrates, however, are poorly matched to nitride films. There is, for example, a lattice mismatch of 16 and 13% between *c*-plane sapphire substrate and GaN and AlN layers, respectively. As a result, the growth of nitride films directly on this substrate leads to formation of three-dimensional (3D) islands [6, 7]. At the initial stage of the growth, these slightly twisted/tilted islands coalesce, which leads to formation of threading dislocation (TD). Also, TDs are formed to reduce the biaxial compressive strains [8, 9] resulting from the lattice mismatch during the growth, as well as from the mismatch in thermal expansion coefficient between the layer and substrate during the post-growth cooling. As a consequence, high densities of threading dislocations, usually in the range $10^{10}\ldots10^{11}$ cm$^{-2}$, are present in typical thin nitride films, if no precautionary steps are taken.

In recent years, there have been many studies on the properties of dislocations in nitride films and their influence on the performance of nitride-based device structures. Many of these studies indicate that TDs are electrically active. Theoretical calculations predict that either dislocations themselves [10-14] or electrically active point defects and impurities accumulated at dislocation due to existing stress fields [15, 16] introduce electronic states in the bandgap. Therefore, if has been predicted that dislocations may act as nonradiative recombination centers [13, 14], and that scattering by charged dislocations can serve as an important mobility-limiting mechanism in two-dimensional (2D) electron gas [17]. Electrical activity of dislocations was also confirmed experimentally. For instance, it was shown by various techniques, including scanning capacitance microscopy [18], electron holography [19], and scanning Kelvin probe microscopy [20] that dislocations can be negatively charged. Also, a number of cathodoluminescence (CL) studies combined with transmission electron microscopy (TEM) have shown that dislocations are nonradiative recombination centers in GaN [21-24]. It was also shown by the correlation between the photoluminescence (PL) intensity and the number of pits at the surface of etched GaN layer [25]. Furthermore, the minority carrier diffusion length, measured by electron beam induced current in thick GaN layers, is directly related to the separation between dislocations determined using TEM studies of these layers [26].

Segregation [27] and diffusion of electrically active impurities and/or point defects along dislocation lines of TDs influence on the properties of nitride-based optoelectronic and electronic devices. TDs were found to serve as undesirable current pathways resulting in reverse-bias leakage current in *p-n* diodes [28], and in very high dark current densities in *p-i-n* structures [29]. They also were found to contribute to vertical leakage path in heterojunction bipolar transistors, where the leakage is caused by compensation of the base material near the dislocation, resulting in a punch-through from the collector to the emitter under bias [30]. As already mentioned, these TDs serve also as nonradiative recombination center limiting the carrier lifetime [21-26] and are responsible for scattering of carries and reduction of their mobility [17]. The high density of dislocations is also detrimental for the operation of nitride-based laser structures, as the potential fluctuations associated with electrically charged dislocations break up the excitons affecting the intensity and the linewidth of the exciton emission lines.

Limiting the scope of these undesired effects by reducing the density of threading dislocations became one of the major challenges for nitride-based technology. This challenge has been widely recognized and several approaches have been proposed in recent years. In this paper, various methods to reduce the dislocation density are reviewed and compared. Advantages and limitations of these methods as well as specific mechanisms responsible for lowering the density of dislocations are discussed.

## 2. Buffer layers

One of the first approaches for growing the high quality nitride heteroepitaxial layers with lowered dislocation densities was application of buffer layers. In the case of GaN films, this concept was used for the first time in 1983 by Yoshida *et al.* [31]. They observed the improvement of the optical and electrical properties of GaN films grown on sapphire substrates when AlN buffer layers were applied. Few years latter, Amano *et al.* reported the metalorganic chemical vapor deposition (MOCVD) growth of the high quality GaN films on sapphire substrate after low-temperature (LT) deposition (at ~800 °C) of thin AlN buffer layers [6]. It was followed by the publication of Nakamura *et al.*, where they reported that thin (~20 nm) LT GaN buffer layers deposited at temperatures between 450 and 600 °C promote high quality growth of subsequent high-temperature (HT) GaN layers [7]. Recently, the concept of using LT buffer layers for the growth of GaN epilayers was also extended on the case of InN, where it was shown that high quality GaN films with dislocation densities as low as $\sim6\cdot10^{8}$ cm$^{-2}$ can be grown on thin (20 to 30 nm) InN buffer layers deposited at approximately 600 °C [32].

A number of extensive studies on the effect of LT buffer layer deposition conditions, such as the layer thickness and deposition temperature, as well as the effect of LT buffer layer annealing conditions on the quality of subsequent HT-grown layer were carried out [7, 33-39]. It has been found for example that growth parameters such as the growth rate and temperature effect on the size and density of nucleation centers and the overall nucleation layer roughness [36]. Furthermore, the surface morphology and crystalline quality of the subsequent HT grown layer strongly depend on thermal effect during the temperature ramping process after growing the buffer layer [40].

Regarding the role of the thin LT buffer layer, it seems that its partially amorphous nature helps to decouple subsequently grown HT GaN from the lattice-mismatched substrate, what leads to the reduced stress in the epilayer. The models of growth mechanisms of GaN on LT AlN [33, 37] and on LT GaN [41] buffer layers have been proposed. According to them, in the initial stage of HT GaN growth isolated GaN islands grow laterally after geometric selection until they coalesce to achieve uniform growth. The number of islands, and subsequently the amount of threading dislocations arising for the coalescence of the island can be controlled by tuning initial growth conditions, since they determine the density of nucleation centers in HT growth. In fact, by selecting optimum growth conditions of the LT buffer layer, the density of dislocations in the HT layer can be reduced down to $5\cdot10^8\,\mathrm{cm}^{-2}$ [42].

Several modifications of the buffer layer approach were developed in recent years. For instance, double buffer layer structures consisting of two 10-nm thick GaN layers were studied, and it turned out that improved quality of the subsequent HT-grown epilayer can be achieved, when the deposition temperature of the first buffer layer is higher than that of the second one [43]. Also, the MOCVD growth of GaN layers on sapphire using HT-grown AlN buffer layers [44-46] and on 6H-SiC using HT-grown AlN and AlGaN buffer layers was realized [47, 48]. Studying the initial stage of this growth on HT AlN showed that 3D islands are formed and grow rapidly. Coalescence of the islands results in a non-uniform defect distribution. It is followed by the selective enlargement of the low defect parts of the layer and finally by lateral overgrowth of the uncovered regions resulting in the average dislocation density as low as $2\cdot10^8\,\mathrm{cm}^{-2}$ [49].

## 3. Substrate nitridation

Another important and well-established procedure for improving crystalline-quality of heteroepitaxial nitride layers is a high-temperature, prior-deposition sapphire substrate surface treatment with $NH_3$ or $N_2$ radicals [50-53]. This procedure known as substrate nitridation seems to be a necessary step in obtaining good quality layers. Poor quality nitride layers with rough surface morphologies and high densities of extended defects are obtained without surface nitridation [52, 54]. Substrate nitridation strongly modifies its surface and has also a strong influence on the surface morphology and polarity of the subsequent nitride epilayers. Mixed-polarity films (*i.e.*, films containing inversion domains (IDs) with a different polarity) of GaN [55] or AlN [56] are typically formed without sapphire nitridation. On the other hand, highly crystalline nitride layers with N-polarity are formed on the nitridated substrate [54, 56]. They still contain IDs with metal-polarity [57], which leads to formation of hexagonal facets at the surface. However, the density of these IDs can be lowered by several orders of magnitude when controlling the initial growth conditions [56].

The substrate nitridation and buffer layer concepts have been combined in the so-called three-step growth technique. In this process, LT buffer layer is deposited on the HT-nitridated substrate. It is followed by the HT growth of nitride layer. It was found that deposition of thicker buffer layers on the nitridated sapphire substrate improves surface morphology and leads to metal-polarity (*i.e.*, Ga-polarity in the case of GaN) of subsequent HT nitride layers [54].

Substrate nitridation enhances nucleation during buffer layer deposition, which leads to films with a small grain size. However, the annealing of these buffer layers results in formation of large isolated islands with (0001) facets, which promotes the lateral growth at high temperatures. Therefore, the 2D growth mode can be achieved on the buffer layers deposited on the nitridated sapphire substrates. It was found that, with increasing the nitridation time and reducing the buffer layer thickness, the buffer layers transform in the course of annealing into films with a lower density of islands resulting in lower dislocation densities in subsequent HT-grown layers [58].

The optimized use of the substrate nitridation being combined with a buffer layer reduces dislocation densities to the low $10^8\,\mathrm{cm}^{-2}$ range [59]. However, for many applications these densities are still unacceptably high and other techniques have been developed to reduce the number of dislocations even further. Very often, high quality GaN layers, with dislocation densities within the range $10^8\ldots10^9\,\mathrm{cm}^{-2}$, obtained in the course of optimized growth on nitridated substrates with deposited buffer layers are just starting materials for these more advanced methods. In the following sections some of the most promising methods will be described.

## 4. Silane-ammonia treatment

Another technique for lowering the dislocation amount is a pre-deposition surface treatment with a mixture of silane ($SiH_4$) and ammonia ($NH_3$). This treatment of the sapphire substrate before LT growth of GaN buffer layer enhances the 3D growth mode with the reduced density of nucleation sites, which leads to reduced densities of dislocations [60-62]. “Anti-surfactant” properties of the silane-ammonia mixture, which inhibits the GaN film from wetting the surface, seem to play an important role in this mechanism. It has been also postulated that, a few nanometer-thick $Si_xN_y$ mask is formed directly on the substrate and limits the density of GaN nucleation sites [61].

The silane-ammonia treatment leads to dislocation densities in the mid-$10^7\,\mathrm{cm}^{-2}$ range [63, 65]. The most serious drawback of this technique, however, can be unintentional *n*-type doping of GaN due to Si contamination.

## 5. The patterning of sapphire substrate

Patterned sapphire substrates can weaken the stress in nitride layers and increase the external quantum efficiency of LEDs that are based on GaN heterostructures. Schematically, the process of patterning

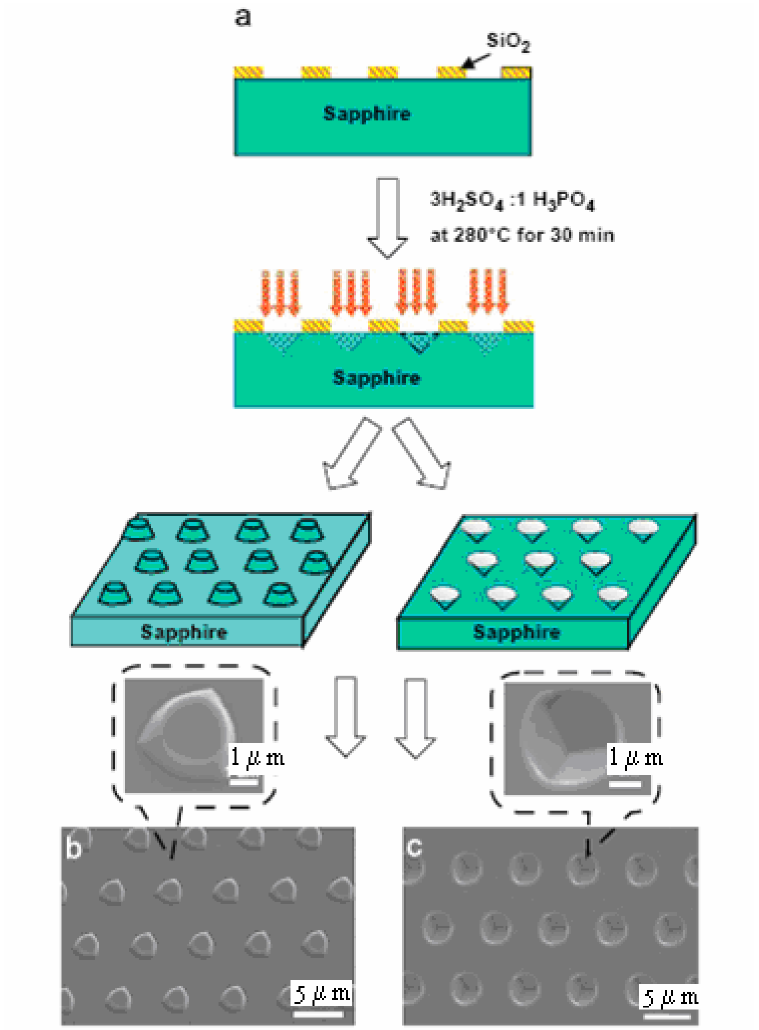

**Fig. 1.** (a) Schematic process flow of patterned sapphire substrate using a wet etching technique. SEM micrographs of various wet-etched patterned sapphire substrates (PSS) structures, (b) dot- array protruding PSS, and (c) dot-array recess PSS [66].

the sapphire substrate is shown in Fig. 1 [66]. A standard photolithography process using respective positive and negative photoresist was carried out to form a wet-chemical-etching mask with different $SiO_2$ patterns on the surface of sapphire substrate. The required $SiO_2$ patterns can be generated by plasma (*e.g.*, using $CF_4$ in inductively coupled plasma ICP) or wet etching.

A sapphire substrate with a positive or negative photoresist can be etched using wet etching (*e.g.*, a mixture $H_2SO_4$:$H_3PO_4$ = 3:1) or in $BCl_3$\$Cl_2$ inductively coupled plasma [67] and has the pattern with protruding or recess of different height\depth ratio.

It is shown that, by using the negative photoresist forming recess on sapphire surface, the dislocation density in GaN layers is lower [66]. The growth of GaN epilayers on patterned sapphire substrate is shown in Fig. 2 [68]. At the lower growth time, GaN grew as separate islands on the lens region and on the trench region, whereas the vertical growth takes place on the lens region, vertical and lateral growth GaN extending from the side walls of the pattern is on the trench region. The islands start to coalesce at the trench part with increasing the growth time [69]. Although the surface of this particular sample was flat, there exists a gap at the coalescence boundaries. From the width of the gap and the GaN film thickness, it was found that the lateral to vertical growth rate ratio was around 2 [70].

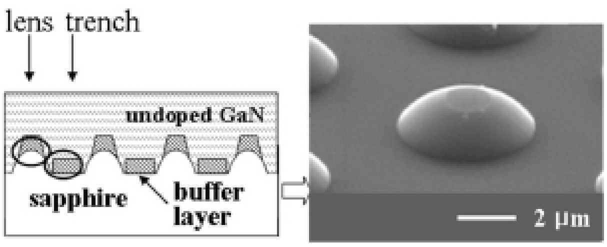

**Fig. 2.** Schematic diagram of GaN on patterned sapphire substrate. The SEM micrograph shows the patterned sapphire substrate after dry etching [68].

As the growth time increases, the GaN layer on the trench part grew separately covering the GaN pyramids during lateral growth, which led to TD bending toward GaN hexagonal pyramid [69]. Therefore, the TD density in the GaN epilayers can be effectively reduced. As the growth time is further increased, full coalescence takes place, which results in smooth surface with a less TD density.

The interface between the vertically grown GaN layer and the sapphire substrate decreases at the patterning of sapphire substrate, which leads to lowering the dislocation density in the grown film [71].

Low temperature nucleation layers with the thickness 3, 15, 30, 50 nm were grown on cone and 30 nm was grown on pyramid-shaped patterned sapphire, then annealed at the high temperature (1025 °C) in an environment of ammonia and investigated by scanning electron microscopy SEM [72]. Studies have shown that the cone surface is separated into six areas averagely. Three of them embrace a high density of nucleation as compared with the others. This phenomenon of selective nucleation does not depend on the thickness of the low-temperature LT layer and pattern shape on sapphire, and it is defined by orientation of the crystallographic planes [72]. Thus, having changed the low-temperature nucleation layer thickness, the annealing temperature and the pattern shape, one can control the number of the nucleus positions on the sapphire surface and, hence, properties of subsequent GaN epitaxial layer.

It is necessary to provide dominantion of high rate lateral growth from the trench sidewalls as compared to the vertical growth above the lens to reduce the dislocation density in GaN layers grown on patterned sapphire. This ratio depends on the orientation of patterned stripes on the sapphire *c*-surface. So, the stripes oriented along the lines $\langle 1\bar{1}20 \rangle$ and $\langle \bar{1}100 \rangle$ are the most suitable ones for growth of GaN films with a low density of dislocations [71].

The patterned sapphire substrate were prepared using the process of wet chemical etching with $SiO_2$ strip masks oriented along the two different directions $\langle \bar{0}110 \rangle$ and $\langle \bar{2}\bar{1}10 \rangle$ on *c*-plane sapphire. After etching in the mixture of $H_2SO_4$:$H_3PO_4$ = 3:1, the patterns on the sapphire substrate with V-shaped trenches were formed. After deposition of the nucleation layer, GaN epitaxial layer was grown in two stages. During the first stage of growth, the GaN layer with a triangular cross-section was prepared on selective surface areas of the patterned sapphire. At the second stage of the growth, GaN is laterally grown and integrated with the underlying GaN layer. The dislocation density was $(2\ldots4)\cdot10^7\,\text{cm}^{-2}$ on the surface of the integrated GaN layer [73].

The epitaxial GaN layer grown on hemispherical patterned sapphire showed the average dislocation density close to $6\cdot10^8\,\text{cm}^{-2}$ [74]. At the growth of GaN layer on pyramidal patterned sapphire obtained by wet etching in a mixture $H_2SO_4$ and $H_3PO_4$, the dislocation density was approximately $5.6\cdot10^7\,\text{cm}^{-2}$, and it was less than that after plasma etching of sapphire [75]. GaN layers grown on cone-shaped patterned sapphire obtained by means of inductively coupled plasma (ICP) had the dislocation density measured by TEM microscopy $\sim1\cdot10^7\,\text{cm}^{-2}$ [76], which increased with the pattern height [76-78].

## 6. Summary

In summary, a number of methods for lowering the density of threading dislocations have been developed in recent years for heteroepitaxial III-nitride layers. Each of these methods offers a different efficiency in lowering dislocation density, as well as the different degree of overall structural quality inherent to resulting nitride films. All the discussed methods have been listed in Table, which compares dislocation reduction

**Table. Comparison of the methods for lowering the dislocation density.**

| **Method** | **Mechanism for lowering the dislocation density** | **Achievable density of dislocations ($cm^{-2}$)** | **Remarks** |
|---|---|---|---|
| Three-step method | Reduction of stress via decoupling the layer from substrate +controlled density of nucleation sites | $\sim5\cdot10^8$ | Insufficient lowering the dislocation density |
| $SiH_4$-$NH_3$ treatment | Growth interruption and surface etching followed by 3D nucleation and lateral growth | $\sim5\cdot10^7$ | Unintentional *n*-type doping is possible due to Si contamination |
| Patterned sapphire substrate with various pattern shapes:<br>a) hemispherical pattern shapes<br>b) pyramidal pattern shapes<br>c) V-shaped trenches<br>d) cone-shaped pattern | Lateral growth from the pattern obtained using photolithography and strictly oriented on *c*-plane sapphire | $\sim6\cdot10^8$<br>$\sim5.6\cdot10^7$<br>$\sim(2\ldots4)\cdot10^7$<br>$\sim1\cdot10^7$ | Lowest dislocation density is attained on certain nitride layer areas |

mechanisms and achievable lowest densities reached using each of these methods. Important advantages and drawbacks of these methods are also listed here.

An important process enabling to obtain the high quality GaN layers with a low dislocation density is patterning the sapphire substrate. The dislocation density depends on the shape and orientation of the patterned strips on *c*-plane sapphire. The GaN layers grown on cone-shaped sapphire have the lowest dislocation density. In addition, the patterned sapphire substrate may increase the external quantum efficiency of the emissive structures and weaken the stress in the nitride layers.

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