

Doping Behavior of (11 $\bar{2}2$)-GaN Grown on Patterned Sapphire Substrates

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We present results of the investigation on the doping behavior of planar semipolar (11 $\bar{2}2$)-oriented GaN grown on (10 $\bar{1}2$) patterned sapphire substrates mainly focusing on the magnesium incorporation. We observed that Mg is incorporated with much lower efficiency into the (11 $\bar{2}2$) plane as compared to polar c-plane GaN. This problem could be decreased by varying the growth temperature. On the one hand, at reduced growth temperature we observed higher Mg concentrations while keeping the Mg flow constant. On the other hand, parasitic background charge carriers due to defects, O-doping etc. could be reduced simultaneously. Using these conclusions, a planar semipolar InGaN/GaN-LED on (11 $\bar{2}2$)-oriented GaN was grown. Electroluminescence measurements show a suitable electrical and optical performance.

1. Introduction

Most of the common optoelectronic devices based on group III nitrides emitting in the visible and ultraviolet range are grown in the well-known c-direction. Lots of techniques are investigated already to achieve excellent crystal quality and smooth surfaces. Nevertheless, the crystal symmetry of the group III nitrides causes strong piezoelectric fields in heterostructures like InGaN/GaN, leading to a bending of the valence and conduction band. The wave functions of electrons and holes get spatially separated and the recombination probability is significantly reduced [1]. Furthermore, the effective band gap decreases resulting in a redshift of the emission spectrum. This behavior is known as quantum confined Stark effect (QCSE). To reduce or even avoid these internal piezoelectric fields, the growth in non-c-directions has been proposed. The epitaxy of nonpolar GaN (perpendicular to the c-axis) is typically dominated by a poor crystal quality. Choosing semipolar directions like (11 $\bar{2}2$), (10 $\bar{1}1$), or (20 $\bar{2}1$) seems to be a good compromise between a reasonable crystal quality and a reduced QCSE. For these particular directions, the amount of the piezoelectric field is reduced to a third as compared to c-plane and additionally, the field direction is inverted. Therefore, an externally applied voltage counteracts the internal field and hence reduces the band bending. However, the epitaxy of (11 $\bar{2}2$) GaN seems to be a big challenge. Typically, (11 $\bar{2}2$) GaN templates are produced by cutting thick c-plane wafers grown by HVPE, which results in wafers limited in size to just a few square mm [2]. This limitation can be overcome by growing such GaN layers on foreign substrates like sapphire or silicon with uncommon orientations. Similar as reported by Okada et al. [3], we are able to grow (11 $\bar{2}2$) GaN layers on (10 $\bar{1}2$) patterned sapphire

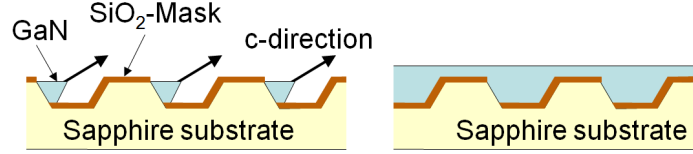


Fig. 1: Patterned sapphire substrate, schematically. All non-c-plane facets are covered with SiO_2 (brown) avoiding parasitic growth. The GaN nucleates on the c-plane sidewall, forms triangular-shaped stripes (left) and coalesces after a suitable growth time to a closed semipolar surface (right).

substrates with a reasonable crystal quality. By etching trenches into these wafers, c-plane-like sidewalls are formed on which the growth of GaN starts developing triangular shaped stripes. After a suitable growth time, these stripes coalesce and form a planar $(11\bar{2}2)$ oriented surface (see Fig. 1). By this procedure, we make use of the well-established growth in c-direction, eventually resulting in a semipolar surface. This approach offers some essential advantages: First, the growth of a full LED or laser diode structure in a single epitaxy run is possible. Second, the diameter of the template is just limited by the reactor size. In our studies, 2" diameter sapphire wafers were used. Well-known from the growth of c-plane GaN, a SiN mask can help to stop defects penetrating to the surface and therefore reduce the defect density in the subsequent layers significantly [4]. Most of the common materials acting as an acceptor in GaN like oxygen and silicon show a very high incorporation efficiency. Therefore, achieving $(11\bar{2}2)$ -oriented GaN layer with a suitable n-conductivity is not a big challenge. However, on the other side a parasitic n-doping (due to impure material sources, external leakages etc.) means a more challenging p-doping process. On the one hand, to achieve a reasonable hole concentration, one has to compensate all free electrons in the layer at first. On the other hand, magnesium acts in GaN indeed as an acceptor, but due to the comparably high activation energy of about 200 meV not very efficient [5]. For c-plane oriented InGaN/GaN-LEDs it is known, that for a suitable hole concentration in the range of $8 \cdot 10^{17} \text{ cm}^{-3}$, a Mg concentration of more than 10^{19} cm^{-3} is necessary [7]. However, it is reported that the Mg incorporation in $(11\bar{2}2)$ -oriented GaN is less efficient than in c-plane surfaces [6]. To establish a suitable p-doping process for our semipolar GaN structures, the doping behavior of Mg and related parasitic materials was investigated systematically.

2. Experimental

2.1 Structuring process and growth of $(11\bar{2}2)$ GaN

In order to achieve $(11\bar{2}2)$ GaN as described above, we have etched trenches into $(10\bar{1}2)$ oriented sapphire wafers. At the beginning of the structuring process, an about $1.7 \mu\text{m}$ thick layer of a negative photoresist is spin-coated on the substrate, which is patterned by optical lithography with a stripe shadow mask with an opening of $3 \mu\text{m}$ and a period of $6 \mu\text{m}$. Via reactive ion etching, the stripes get transferred into the sapphire substrate.

Covering all non-c-plane-like facets with SiO₂ (see Fig. 1) by directed sputtering prevents parasitic growth.

The MOVPE growth was done in a commercial Aixtron-200/4 RF-S HT reactor using the standard precursors ammonia (NH₃), trimethylgallium (TMGa) and trimethylaluminum (TMAI). The growth starts with our about 20 nm thick standard AlN nucleation layer at relatively low temperature of about 950 °C [8]. For the subsequent GaN growth a reactor temperature of about 1020 °C is chosen. The GaN gets pushed in c-direction and builds triangularly formed stripes, which coalesce after a suitable growth time to a planar, semipolar (11 $\bar{2}2$)-oriented surface. An in-situ deposited SiN interlayer helps to improve the crystal quality by stopping defects penetrating to the sample surface [4]. By decreasing the growth temperature of the topmost GaN layer to 970 °C, the growth gets pushed further in c-direction and the coalescence of the stripes gets improved. The total thickness of the GaN layer is about 5 μm. X-ray rocking curve measurements give a FWHM of about 200 arcsec for the (11 $\bar{2}2$) reflection indicating a suitable crystal quality. Atomic force microscopy (AFM) measurements show a surface roughness of 10 nm in an area of 70 μm × 70 μm.

From low temperature photoluminescence (PL) measurements (10 K), using a 1000 mm monochromator and a 1200 mm⁻¹ grating, we obtained more detailed information about defects and doping behavior close to the crystal surface.

2.2 n-doped GaN

The doping experiments have been performed in parallel on standard c-plane wafers and our semipolar (11 $\bar{2}2$) samples to investigate possible differences from our well-established c-plane results. Therefore, (0001) and (11 $\bar{2}2$) GaN were overgrown side by side with Si-doped GaN. The Si flow was systematically varied between 6 nmol/min and 17.5 nmol/min. The Si concentration was then analyzed by secondary ion mass spectrometry (SIMS)² (see Fig. 2). Obviously, the incorporation efficiency of silicon is independent of the surface orientation within reasonable error bars, similar as reported by other research groups like Xu et al. [9]. Standard InGaN/GaN-LEDs require an electron density of about 10¹⁹ cm⁻³ in the n-layer. Therefore, a molar Si flow of 12 nmol/min was established for the n-layer of the InGaN/GaN-LEDs grown on (11 $\bar{2}2$) oriented GaN (see below).

2.3 Parasitic background doping of GaN

To achieve a suitable hole concentration, the parasitic electron density due to O, Si, defects etc. has to be suppressed as much as possible. Hall measurements show a very high electron density of 7·10¹⁸ cm⁻³ in (11 $\bar{2}2$) layers grown at a temperature of 970 °C. A growth temperature variation of nominally undoped (11 $\bar{2}2$) GaN was done. Subsequent Hall measurements show a strong temperature dependence of the parasitic electron concentration (see Fig. 3). In the range of 910 °C there seems to be a minimum with 1.5·10¹⁸ cm⁻³. Compared to our standard c-plane oriented GaN layers with a parasitic

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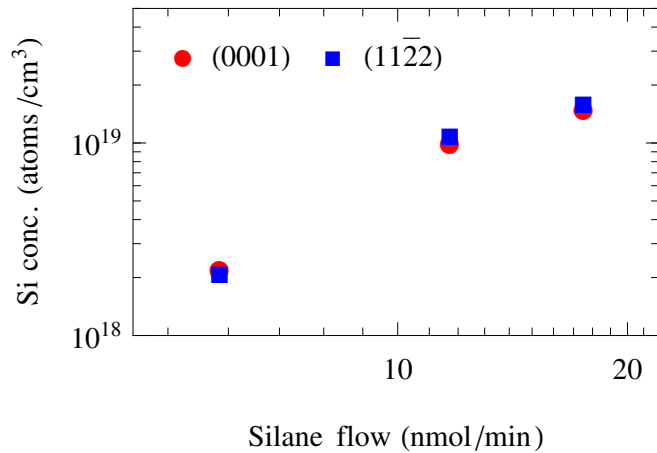


Fig. 2: Silicon concentration in GaN depending on the molar Si flow measured by SIMS.

electron density below 10^{16} cm^{-3} , it is still a very high value. A further reduction of the growth temperature leads to a poor crystal quality and therefore to an increasing electron density again.

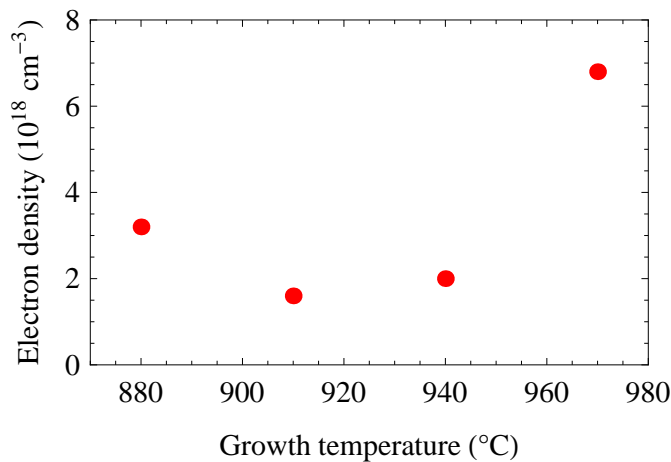


Fig. 3: Electron concentration in nominally undoped GaN grown at different temperatures.

2.4 p-doped GaN

Analog to the Si doping, (0001) and (112̄2) GaN were overgrown side by side also with Mg-doped GaN. The magnesium flow was systematically varied between 100 nmol/min and 350 nmol/min. Subsequent SIMS measurements (see Fig. 4, left) show that, grown at a temperature of 970 °C, (112̄2)-oriented GaN seems to incorporate just a tenth compared to a polar surface. This means that a 10 times larger Mg flow is needed to achieve the same Mg content on (112̄2) surfaces as compared to c-plane. Similar results were reported by Cruz *et al.* in 2009 [6]. Using these growth conditions, a maximum hole concentration

of $1 \cdot 10^{17} \text{ cm}^{-3}$ with a Mg flux of 390 nmol/min was achievable. Unfortunately, this value is still far away from desirable hole concentration in the range of $8 \cdot 10^{17} \text{ cm}^{-3}$, as mentioned before. Moreover, LEDs grown with this p-layer showed a fairly low output power. This may be caused by a degradation of the quantum wells during the subsequent p-GaN overgrowth at such comparably high temperatures. Emitting at a wavelength of 470 nm, the semipolar InGaN/GaN-LED had a fairly low optical output power of 50 μW at 20 mA driving current.

In order to reduce the parasitic background n-conductivity (cf. Sect. 2.3) and the thermal treatment of the InGaN/GaN-QWs due to the p-layer growth, its growth temperature should be reduced as much as possible. Corresponding investigations of the temperature dependence of the Mg incorporation show a significantly increased Mg incorporation efficiency at lower reactor temperatures (see Fig. 4, right). Keeping the Mg flow constant and reducing the temperature from 970 $^{\circ}\text{C}$ to 910 $^{\circ}\text{C}$, the Mg concentration gets more than doubled. A further reduction of the growth temperature leads, as already mentioned, to a poor crystal quality and an increasing electron density again. Therefore, the temperature of the p-doped layer was fixed at this temperature as well.

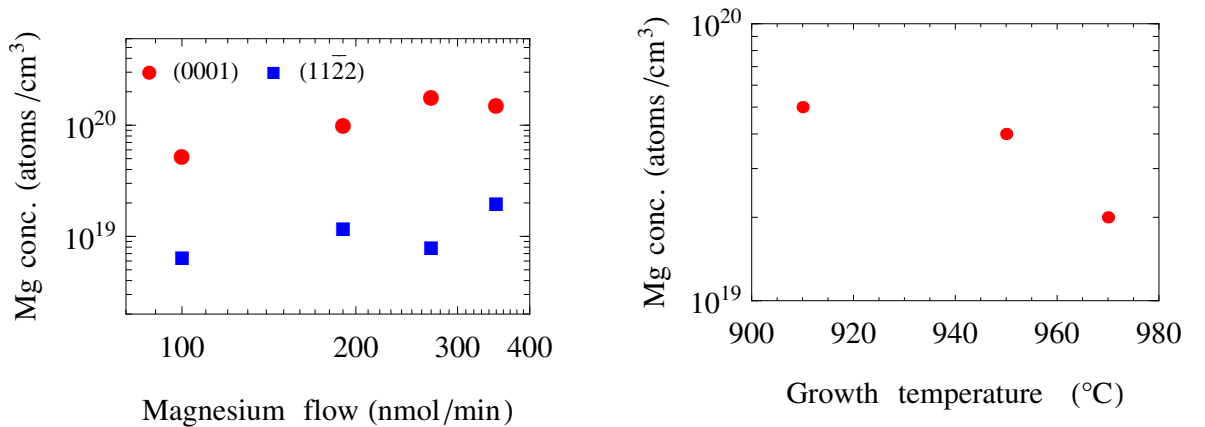


Fig. 4: Left: Magnesium concentration in GaN depending on the molar Mg flow measured by SIMS. Right: Growth temperature dependent Mg incorporation for a Mg flow of 350 nmol/min measured by SIMS.

Hall measurements are a standard method to investigate the carrier density in p-doped GaN. Unfortunately, a too low Mg doping is not able to compensate the parasitic electrons — the p-doped GaN layer is (semi-) isolating. A too high doping level leads to a high defect density, which increases the parasitic electron density as well — the p-doped GaN layer may be again (semi-) isolating. To distinguish between these two situations just by Hall measurements is difficult. However, a too high Mg content leads to a very strong defect related signal in low temperature photoluminescence (LTPL) measurements, whereas a too low p-doped layer gives just a weak donor-acceptor-pair (DAP) transition related signal. In Fig. 5 (left, top), the LTPL signal of the sample with a Mg flow of 390 nmol/min grown at 910 $^{\circ}\text{C}$ is shown. A very intense defect related signal at 3.1 eV is visible, which indicates, as already mentioned, a too high Mg content. Reducing the Mg flow to 140 nmol/min, a first DAP related signal is indicated. A further reduction of the Mg flow to 50 nmol/min

leads to a very clear DAP signal at 3.28 eV. The corresponding phonon replica at 3.19 eV, 3.10 eV and so on are clearly visible as well. Hall measurements on this sample show a hole density of $1.2 \cdot 10^{17} \text{ cm}^{-3}$, which is still, as already mentioned, far away from the desired value of $8 \cdot 10^{17} \text{ cm}^{-3}$ for an InGaN/GaN-LED.

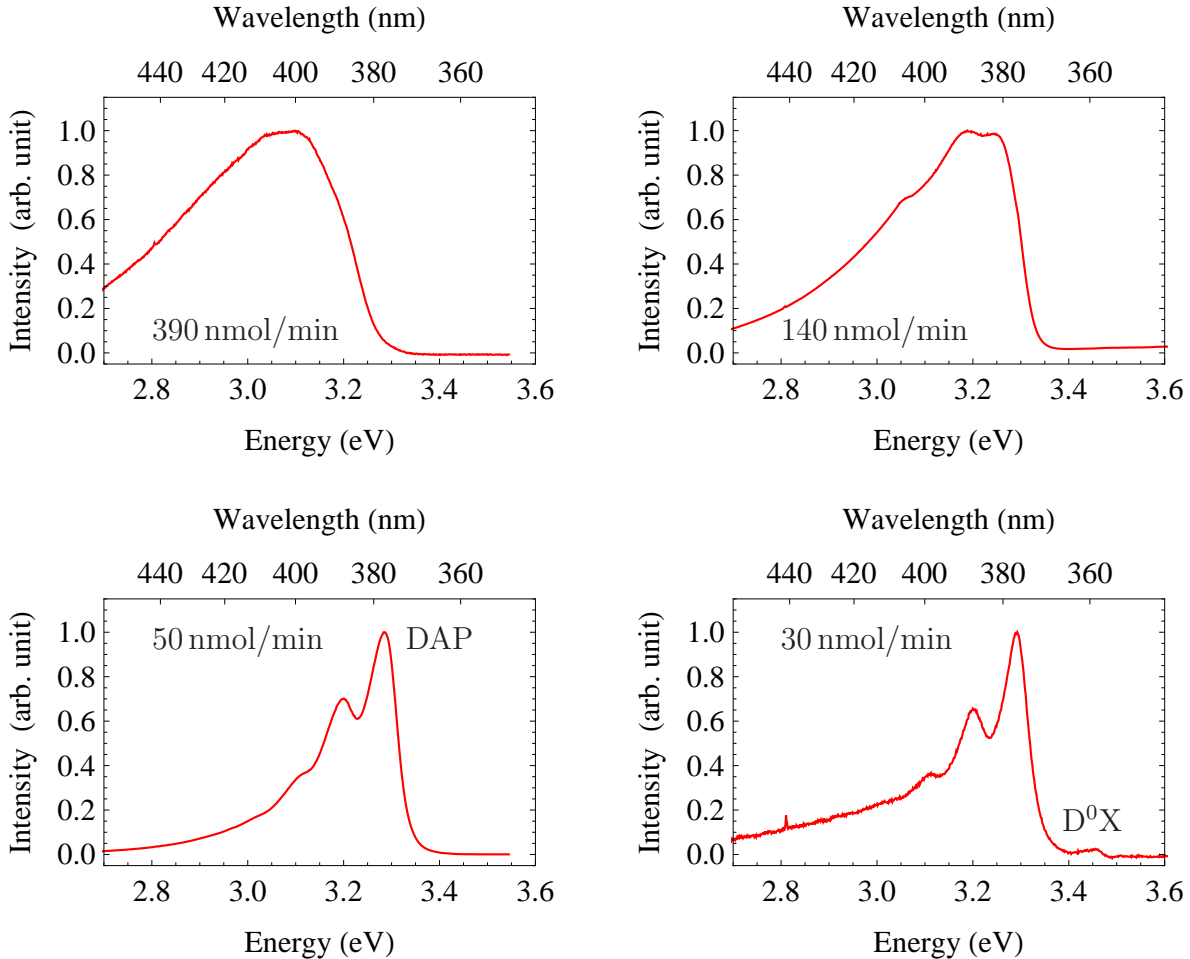


Fig. 5: Photoluminescence spectra of GaN grown with different Mg molar flows.

2.5 Semipolar InGaN/GaN-LED

After having optimized the n- and p-layers, we have integrated such layers together with InGaN/GaN-QWs to realize a semipolar LED structure. Therefore, five InGaN quantum wells (QW) with a thickness of 3 nm and 6 nm thick GaN barriers were grown on the top of an about 2 μm thick n-doped semipolar GaN layer. Between the p-doped layer and the QWs an about 30 nm thick intrinsic GaN spacer was grown in order to minimize the back-diffusion of Mg atoms into the InGaN layers. The p-layer itself has a thickness of about 100 nm. Corresponding electroluminescence (EL) measurements give a QW emission wavelength of 470 nm and an optical output power of 91 μW at a driving current of 20 mA measured with simple contact processing on the wafer (see Fig. 6, right). Thus, an optimized p-doping process and a reduced growth temperature of the layers subsequently

grown on the top of the InGaN-QWs improved the optical output power by 50 %. In Fig. 6 (left), the I-V-curve of this LED is shown. A reverse current of 5 μA at -10 V indicates a nice diode behavior of the LED. However, we observed a fairly large series resistance leading to a forward voltage of about 10 V at 20 mA. This may be a consequence of a not yet completely optimized p-doped GaN layer.

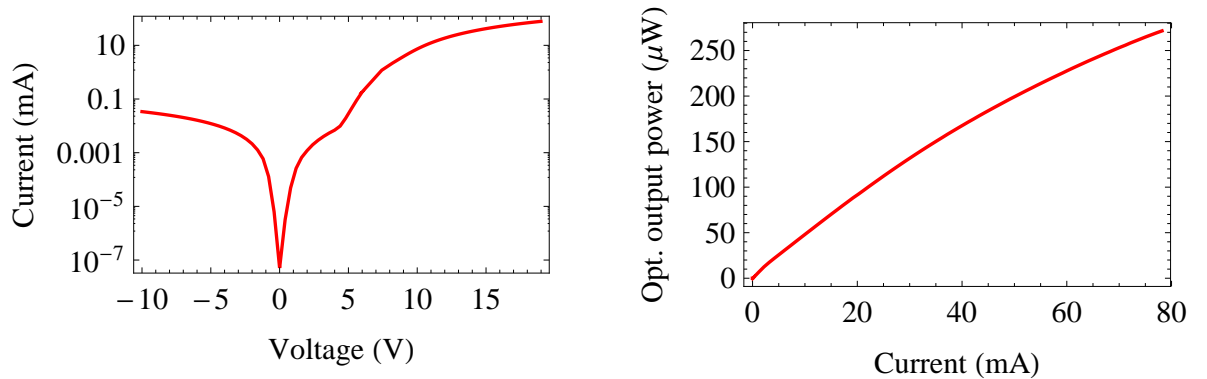


Fig. 6: Left: Voltage dependent current measurement of an InGaN/GaN LED based on the improved Mg doping process. Right: Optical output power versus drive current.

3. Conclusion

Using (10 $\bar{1}2$) patterned sapphire substrates, we were able to grow planar semipolar (11 $\bar{2}2$) GaN with a suitable crystal and surface quality. Systematic investigations of the Mg-doping behavior of (11 $\bar{2}2$)-oriented GaN showed a strong temperature dependent incorporation efficiency. The lower the growth temperature, the more Mg atoms were incorporated. However, in comparison to *c*-plane surfaces, the efficiency seems to be still significantly lower. Fortunately, a decreased growth temperature seems also to reduce the amount of parasitic background charge carriers. Choosing the optimized growth conditions, a LED with a suitable electrical and optical performance was grown. The LED shows a good diode behavior and the optical output power at a driving current of 20 mA was measured to 91 μW .

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