Defect Reduction in GaN by Facet Assisted Lateral Overgrowth With Hexagonal Mask Geometry

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The goal of this project was the development of a complex growth procedure of a GaN layer on sapphire with minimized dislocation density, which can be used as template for the subsequent growth of a thick GaN layer by hydride vapor phase epitaxy. We extended a conventional stripe-based FACELO approach (FACELO = facet assisted epitaxial lateral overgrowth) [1] to a mask geometry where the stripes are arranged as a hexagonal honeycomb grid. We expect a more uniform defect reduction and more isotropic strain and curvature development in the GaN layer by such a mask geometry. By a careful optimization of each layer in a multi-layer growth procedure, GaN structures with excellent spectroscopic properties could be grown. Although some surface features visible by optical microscopy could not be completely suppressed, a very low dislocation density below 10^6 cm⁻² was evaluated from etch experiments performed in our hydride vapor phase reactor.

1. Introduction

GaN layers and device structures grown on foreign substrates like sapphire still dominate by far the current approaches for such structures, although the hetero-epitaxial approach leads to a comparably huge defect density. Various defect-reduction methods have been developed over the recent decades, mainly based on the idea of 'epitaxial lateral overgrowth' (ELO) [2]. A particularly effective method was originally proposed by Vennéguès *et al.* [3], called 'facet-assisted epitaxial lateral overgrowth' (FACELO) [4]: On a GaN template layer, a dielectric stripe mask is fabricated. In a second epitaxial process, GaN grows out of the mask openings forming GaN stripes with triangular cross section. By changing the growth conditions, lateral growth can be enhanced leading to a lateral bending of the originally vertically running threading dislocations. This potentially leads to a completely dislocation-free final surface, if the coalescence of neighboring stripes does not induce new defects. Besides the coalescence problem, this approach leads to an anisotropic strain situation in the final GaN layer with great differences for the strain parallel and perpendicular to the stripes.

Based on our previous work towards the conventional FACELO approach [1], we extended such a stripe-based approach to a mask geometry where the stripes are arranged as a hexagonal honeycomb grid. We expect a more uniform defect reduction and more isotropic strain and curvature development by such a mask geometry.

2. Experimental

For these studies, optimized c-plane GaN layers grown by metalorganic vapor phase epitaxy (MOVPE) [5] are used as templates, which contain a fairly low dislocation density of about $3-5 \cdot 10^8 \text{ cm}^{-2}$. On those templates, a 200 nm thick SiO₂ layer is deposited on top by plasma enhanced chemical vapor deposition (PECVD) as a mask for the subsequent selective area growth of GaN. A stripe pattern with periodicity of 11 µm (3 µm opening) is created in the SiO₂ layer.

In the second MOVPE process, a multi step procedure is applied based on our earlier findings [1]. This process needed careful adaption to the current MOVPE reactor conditions etc. which have been changed since our studies in 2004.

3. FACELO Multi-Step Growth of GaN With Stripe Mask

In the first overgrowth step, GaN stripes with a triangular cross section are developed at a temperature of 980 °C and pressure of 250 hPa. Then, lateral overgrowth is enhanced by lowering the reactor pressure and increasing the temperature. In the last step, very good coalescence is achieved at higher temperature and lower reactor pressure, leading to samples without wing-tilt, as observed in scanning electron microscopy (SEM, Fig. 1, left) and confirmed by only one peak in high-resolution X-ray diffraction (HRXRD) 0002 rocking curve measurements (Fig. 2, left). The still fairly large full width at half maximum (FWHM) of such peaks of about 280 arcsec may be due to some bowing of the samples. For the X-ray beam parallel to the stripes, the samples exhibit very narrow peaks with a FWHM of only 150 arcsec. However, the surface is not completely smooth (Fig. 1, right).



Fig. 1: SEM cross section (left) and top view image (right) of stripe mask sample. The dust seen in the cross section image is some contamination after cleaving of the sample.

The low-temperature photoluminescence (PL) spectra of this sample (Fig. 2, right) shows an extremely narrow peak at 3.487 eV related to the donor-bound excitonic transition (D°X) with a FWHM of 2 meV. The peak at 3.493 eV can be related to the free exciton (named FX) as confirmed by temperature dependent PL measurement.



Fig. 2: HRXRD rocking curve with the beam perpendicular to the stripes (left) and low temperature PL spectra (right) for the sample on SiO_2 mask.

To improve the surface morphology, we reduced the growth temperature in both, the lateral growth and coalescence steps to 1020 °C and 1065 °C respectively. Indeed, we successfully reduced the size of the round shaped defects in the surface (Fig. 3). By HRXRD rocking curve measurements we could confirm that this temperature reduction doesn't lead again to some wing-tilt, which is often observed in such structures [6]. For the 0002 reflection with the beam parallel and perpendicular to the stripes, only one peak with a FWHM of 300 and 375 arcsec, respectively, was observed.



Fig. 3: Optical microscope image for sample with stripe structure: Lateral and coalescence growth performed at high temperature (left) and at low temperature (right).

The low temperature PL measurements further confirmed the high quality of these samples (Fig. 4), featuring a narrow and intense signal at 3.486 eV related to the D°X transition with a FWHM of 3 meV and the free-exciton peak at 3.492 eV.

These optimized parameters formed a sound base for our further investigations using a honeycomb mask made of SiO_2 .



Fig. 4: Low temperature PL spectrum for the sample with stripe structure and improved surface morphology.

4. FACELO Multi-Step Growth of GaN With Hexagonal Mask

We extended our stripe-based approach to a mask geometry where the stripes are arranged as a hexagonal honeycomb grid with a periodicity of 13 μ m and an open stripe width of 3 μ m with the stripes running along the m-directions of GaN. In the first overgrowth step, GaN with a triangular cross section is developed at a temperature of 980 °C and pressure of 200 hPa (Fig. 5, left). Then, lateral overgrowth is enhanced by lowering the reactor pressure to 90 hPa and increasing the temperature to 1160 °C. As it is seen in the SEM cross section image (Fig. 5, right), no tilting angle in the top c-plane surface is observed.



Fig. 5: SEM bird's eye view after second growth step (left), cross section image after lateral growth step (right).

In the last step, the coalescence is achieved at a higher temperature of $1220 \,^{\circ}\text{C}$ and the same pressure of 90 hPa (Fig. 6a). However, again, the surface is not completely smooth (Fig. 6b and 8, right). We also have studied another mask orientation with a 30° rotation of the hexagonal structure resulting in $(10\overline{1}1)$ facets at the beginning of the growth procedure (sample B). The SEM cross section for this sample also confirms that the FACELO technique is working (Fig. 6c). However, the surface is rougher than on the sample A with the lines of the mask running along m-direction (Fig. 6d). The FWHMs of the HRXRD rocking curve peaks are determined to 325 and 370 arcsec for samples A and B, respectively. Therefore, sample A is considered as the desirable result. Furthermore, the PL spectrum for this sample exhibits an extremely intense and narrow peak (FWHM of 3 meV) related to the D°X transition (Fig. 7).



Fig. 6: SEM micrographs of the samples grown with the hexagonal masks: cross section (a, c) and top view (b, d) images of samples A and B, respectively.

The procedure to improve the surface morphology of stripe mask samples mentioned in Sect. 3 was then also applied successfully to our hexagon mask samples: We decreased again the temperature of the lateral and coalescence growth to $1080 \,^{\circ}\text{C}$ and $1150 \,^{\circ}\text{C}$, respectively (Fig. 8, right). Very narrow rocking curve peaks with FWHM of 190 and 240 arcsec for the beam parallel and perpendicular to the stripes, respectively, confirm their excellent quality with no indication for any wing-tilt, again confirmed by excellent PL spectra with an extremely intense and narrow D°X peak (FWHM of 2 meV) at 3.488 eV and a significant FX related signal at 3.492 eV (Fig. 9).

5. Etch Pit / Dislocation Density

In order to evaluate the dislocation density of our optimized samples, we have applied our vapor phase etching process where the samples are exposed to 50 sccm HCl gas diluted by nitrogen at an elevated temperature of 600 °C and a pressure of 940 hPa for 15 minutes in an Aixtron hydride vapor phase epitaxy (HVPE) reactor [7] to decorate the dislocations on the surface. The sample with the coalesced stripes and sample A and



Fig. 7: Low temperature PL spectrum of sample A.

Fig. 8: Optical microscope image for sample with hexagonal structure: Lateral and coalescence growth performed at high temperature (left) and at low temperature (right).

B with the coalesced hexagonal structures were etched by the described experiment. In addition, a standard c-plane GaN layer was etched to check the validity of the experiment. $10 \times 10 \,\mu\text{m}^2$ atomic force microscopy (AFM) scans were evaluated to determine the etch pit density (EPD; Fig. 10). On the standard c-plane sample, we found an EPD value of $5 \cdot 10^8 \,\text{cm}^{-2}$ (Fig. 10a), which is very reasonable for such a sample. However, no pits were observed on the other three samples (Fig. 10b-d) which obviously confirms the very low dislocation densities for the samples grown by the FACELO technique. Even on larger area AFM scans including about three periods of stripes and hexagons ($30 \times 30 \,\mu\text{m}^2$), we could not identify etch pits on the optimized FACELO samples. Hence, we estimate the EPD to be below $10^6 \,\text{cm}^{-2}$.

6. Summary

Based on our experience obtained in 2004, we could re-establish a multi-step FACELO process for GaN on sapphire by using a stripe mask pattern. By carefully improving this process, layers with excellent X-ray and photoluminescence properties have been



Fig. 9: Low temperature PL spectrum for the sample with hexagonal structure and improved surface morphology.

obtained. The very often observed wing-tilt could be efficiently suppressed. Also the surface morphology could be drastically improved, although some features still are present on the best wafers. Anyway, the density of threading dislocations at the surface could be decreased to values below 10^6 cm^{-2} according to EPD experiments performed in our HVPE reactor. These excellent properties have been also obtained on samples grown on full 2" sapphire wafers using a mask pattern with hexagonal (honeycomb) geometry in order to improve the lateral isotropy of our structures. Very similar data have been obtained on such wafers as on the stripe mask wafers including EPD-values below 10^6 cm^{-2} .

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Fig. 10: $10 \times 10 \,\mu\text{m}^2$ AFM image of etched samples: normal c-plane (a), coalesced stripes (b), sample A (c) and sample B (d) with the coalesced hexagonal structures.

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