Pyramid Formation by Etching of In$_x$Ga$_{1-x}$N/GaN Quantum Well Structures Grown on N-face GaN for Nanooptical Light Emitters

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While growth processes of In$_x$Ga$_{1-x}$N/GaN quantum well (QW) structures on the Ga face of GaN buffer layers are already optimized to obtain high quantum efficiency, the growth on N-face has gained momentum only in the past years. Compared with Ga face In$_x$Ga$_{1-x}$N layers are more stable on N face, and the surface can easily be structured by wet chemical etching, which usually leads to the formation of pyramids on the surface. This allows a new way to realize nanooptical light emitters, which offers the possibility to produce structures with similar emission properties. First, In$_x$Ga$_{1-x}$N/GaN (single or multi-) QW structures on N-face GaN are grown. In a second step, pyramids are formed by KOH etching. Pyramids with smooth side facets of the type (110T) are demonstrated and sharp tips in the nanometer range can be achieved without any sign of damage. Transmission electron microscopy (TEM) reveals that In$_x$Ga$_{1-x}$N quantum dot-like structures are present in the pyramids and in photoluminescence narrow emission lines are observed. The etching process depends on electrolyte composition and temperature, defects at the surface, and surface morphology. A better control of this process is required to achieve reproducible nanostructures.

1. Introduction

Nanooptical light emitters based on semiconductors are very promising for a broad range of applications such as single-photon sources or optical sensors of nanoobjects. The group-III nitrides are especially interesting in this respect as light emitters in the visible range with high efficiency are nowadays commercially available. By changing the indium content, the bandgap of In$_x$Ga$_{1-x}$N can in principle be tuned over the whole wavelength range from the near-IR region to the near-UV region. In most studies, three routes are followed to produce nanooptical light emitters: 1) the self-assembled growth of quantum dots (QDs),[1,2] 2) the growth of pyramids by applying masks of, for example, SiO$_2$,[3] or 3) the growth[4,5] or top—down fabrication[6] of nanorods. The first one suffers from the high defect density in group-III nitride growth when using conventional sapphire, SiC, or silicon substrates,[7] that the sites of the QDs are unknown (unless further steps are taken), the properties are critically dependent on the growth conditions, and that it is difficult to stabilize the QDs in further growth. The second one has the problem that indium incorporation on different parts of the pyramid, side facets, edges, and tip, varies and is difficult to control, which typically result in an inhomogeneous emission. Furthermore, defects may form, induced by the mask, which reduce the efficiency. The insertion of QDs in nanorods during growth shows, up to now, low efficiency. A top—down approach to realize QDs in etched nanorods would be quite a challenge due to the length of the nanorods and the small required diameters.

Hence, for the generation of a large array of as similar as possible nanooptical light emitters, we propose another solution. A single or multiquantum well (SQW, MQW) structure is grown on N-face GaN and in a second step pyramids are formed by wet chemical etching. This has the advantage that the QW can be optimized independently of pyramid formation. However, in contrast to the N face, the Ga face is chemically inert and plasma etching processes may create damage, which reduces the efficiency. Another advantage of using N-face GaN is that the
indium incorporation efficiency is known to be higher compared with Ga face\cite{8,9} and therefore it should be easier to obtain high indium contents for long wavelength emission. However, in a different way, pyramids have been formed by etching processes after growth of Ga-face structures, and it is possible that such structures can also be used as nanooptical light emitters\cite{10,11}. The advantage is that the QW structures can be grown with high efficiency on Ga face. However, we believe that for the purpose of arrays of similar light emission properties, our approach is better suited.

In this publication, first, the process is described in some detail and then the results of the characterization of the various necessary steps to produce structures—eventually arrays—with similar emission properties. We demonstrate that it is possible to obtain pyramids with QDs close to the tip and identify the most critical step in this process, that is, the control of the pyramid etching.

2. The Process to Create In\(_x\)Ga\(_{1-x}\)N QDs in Pyramids

The proposed process to obtain pyramids with a QD in the top is shown in Figure 1 and contains the following steps. First, In\(_x\)Ga\(_{1-x}\)-N/GaN QW structures are grown on N-face GaN. As the lateral extension of the QDs depends on the depth with respect to the tip (or the starting surface), a single QW would be optimal. The growth conditions have to be chosen such that the indium content and thickness of the QW as well as the thicknesses of the subsequent layers are homogeneous. In the second step, the samples are etched by aqueous KOH and hexagonal pyramids with (1101) facets formed. As these facets are more stable than the (000\(\overline{1}\)) surface, the etch rate at the pyramids’ side facets drops and only their sizes may change in further etching\cite{12,13}.

The formation of the pyramids needs control on a scale of a few nm by, for example, optical in situ methods (e.g., light scattering or reflection-based techniques) to be able to stop the etching process when the proper depth is reached. This is a challenge and requires good knowledge of the etching process and we can expect lateral fluctuations of the etching depth.

While the two steps are very likely to be sufficient to produce single pyramids with QDs, the inhomogeneity of the etching process will not result in structures of pyramids of similar sizes. To achieve this, a further step is necessary. It is needed to define the locations where etching begins. Patterns of masks (metals or other suited materials) are usually used to accomplish this. If this approach works, the regions, where the tips of the pyramids are formed, are protected and the height of the pyramid (and from geometry thereby the lateral extension) is given by the overall etch depth. However, the etching process is not well understood and there is no clear picture in which way it proceeds. There are two obvious scenarios. 1) The metal may protect the forming pyramid against etching. In this case, for example, circular masks define the location of the pyramids and the distance—in this case the pyramids would be dense on the surface—between the circles defines the lateral extension. 2) Openings in the metal mask define locations where small pits are formed, which eventually coalesce and form the pyramids. The location and extension of the pyramids would also be given by the geometry of the used mask.

If the QW is located near the surface, a QD is formed by the formation of the pyramid. The lateral extensions of the QDs are given by that of the pyramid and would be similar, if the sizes of the pyramids can be controlled well enough. While the homogeneity of height and indium content is given by the starting QW structure, the lateral extensions depend mainly on the etching process.

3. Results and Demonstration of the Feasibility

3.1. QW Growth

Various samples have been grown, and in agreement with recent publications\cite{9}, we find that the indium incorporation efficiency is higher on N-face material and thus more than 50 K higher growth temperatures can be used to achieve the same indium content as in the case of Ga face (see Figure 2). However, the samples suffer from hillock formation and thus a bad surface morphology. For high growth temperatures and low V/III ratios, we were able to minimize the density of such hillocks with areas of a few 10\(\mu\)m

![Figure 1](image1.png)  
**Figure 1.** Process to obtain pyramids containing In\(_x\)Ga\(_{1-x}\)N QDs.

![Figure 2](image2.png)  
**Figure 2.** \(x_{\text{In}}\) for In\(_x\)Ga\(_{1-x}\)-N QW structures grown on N face compared with various substrate orientations (c-, m-, a-plane taken from H. Jönén et al. PSS (b)248, 600 (2011)).
in diameter free of hillocks. The arrangement of the hillocks indicates that they originate at defects, likely already present in the substrates. However, we cannot exclude that higher offcut angles, as used in the growth on sapphire, may result in hillock-free material. HRXRD shows less-pronounced superlattice peaks, indicating that layer quality is inferior to that of Ga-face samples. It is likely that hillocks attract Ga and In, thus disturbing the growth process in the vicinity, as we found in these locations much thicker QWs.

For most as-grown samples, we could observe PL at room and low temperatures. However, for the excitation with 334 nm laser lines, the spectra are dominated by GaN PL and GaN-related defects, which resemble in spectral position and shape the well-known stacking faults-related luminescence for semipolar GaN samples. The defect-related PL can also be observed after etching and in some cases becomes even stronger.

For the as-grown QW structures, we find low intensity of In$_x$Ga$_{1-x}$N-related PL at room and low temperatures (see Figure 3). Low efficiency is expected because suppression of nonradiative recombination by pits, as in the case of Ga face, is not possible. In addition, incorporation of oxygen is a problem for N-face material which also suppresses PL. Further optimization is required to achieve more efficient PL intensity.

### 3.2. Pyramid Etching

All grown samples are etched by 1 M aqueous KOH at 80 °C, which according to other studies should be favored conditions for a high density of pyramids. The etching time is varied between 20 s and 4 min.

For the first series of samples, pyramids with very sharp tips are formed after etching for 3 min...4 min but also irregular, etched structures, as shown in Figure 4. The length of the base edge of the pyramids for these samples is in the range 1–2 μm.

Figure 3. PL spectrum of a sample (first series) grown at 825 °C (5xQW, 23% indium) not etched at 15 K using the 380 nm line of an Ar$^+$-ion laser, which is essentially absorbed only in the In$_x$Ga$_{1-x}$N-containing regions. The as-grown N-face QW structures typically show a broad emission spectrum.

Figure 4. SEM image of a sample (5xQW, 16% indium) of the first series etched for 3 min in KOH (no mask). A few pyramids show quite sharp tips (curvature radius of a few nm, see Figure 8). Irregular structures are observed, which may be a consequence of the presence of surface defects.

As described earlier, control of the positions and sizes of the pyramids can only be achieved by the usage of suited masks. The mask material needs to resist the electrolyte and should have some stability during underetching of the mask. We conducted experiments with Ti masks (stripes and dots). Titanium is stable in an aqueous KOH electrolyte and may serve as metal contact in an electrically driven light-emitting nanostructure, if it is not completely removed by underetching of the mask. SEM pictures of an etched sample after 1 and 3 min etching time are shown in Figure 7. After about 1 min etching time, small pits are visible, after 3 min, pyramids are very well developed, and after 4 min, larger pyramids seem to have split in smaller ones. This indicates that etching starts very slowly but after small pits are formed etching progresses very fast. Some hillocks can be seen in the SEM images on the surface, which can still be observed after etching.

The overall etching process corresponds well to the model of Wang et al.: etching starts at dislocation sites and pits are formed, which coalesce, and truncated pyramids appear, which evolve to pyramids with sharp tips by further etching. However, a high density of threading dislocations is not expected for these kinds of substrates. Thus, it is more likely that etching starts at surface defects such as vacancies, contamination, scratches, or at locations which are microrough including step edges. In the beginning, those defects at the surface are attacked, depressions are formed, which may evolve into small pits which grow, and pyramids are generated like in the aforementioned model. The pits with (1T0T) facets may form by one or more intermediate facets, as mentioned in the study by Tautz et al., or be known from lateral growth experiments.

For the second series of samples, a different behavior in the etching process seems to occur, as shown in Figure 5. Small pyramids rapidly form within 30 s and after a longer etching time, the small pyramids vanish and few large pyramids remain (see Figure 6).

This suggests that in this case the etch nuclei are more dense and homogeneous. In earlier publications, it is stated that larger
pyramids are expected only after longer etching times of the order of 1 h.\textsuperscript{12,13} In contrast we observe large pyramids of heights in the range from 1 to 2 μm after already 3 min etching time. For the sample in Figure 6, a typical height value of the pyramids is 1.2 μm. This would give a lower bound of the vertical etch rate of about 400 nm min\textsuperscript{-1}. The real value is higher, as there is an additional overall etching taking place, which reduces also the height of the pyramids, as shown in, for example, Figure 7, or the missing QWs in the TEM image of the pyramid with sharp tips as discussed later (Figure 8). This value is in rough agreement with a value of 138 nm min\textsuperscript{-1} (also for 80 °C) for the etch rate reported in the study by Tautz et al.\textsuperscript{18} However, this value for the etch rate is obtained for a much higher concentration of the aqueous KOH (30 wt%) and it is well known to depend strongly on temperature and molarity.\textsuperscript{19}

Whether a better-defined surface morphology or less defects at the surface are the causes for the different behaviors of the two sample series cannot be decided from the conducted experiments and will be further investigated. Similar surface structures as obtained for the second series with a high density of small pyramids have been reported for photochemical etching and the role of defects in the initial state of the process.\textsuperscript{20} However, in both cases, large pyramids of lateral extensions of 1μm are obtained, which is sufficient for the intended applications.

### 3.3. TEM Analysis of Pyramids

A single pyramid of an etched sample (4 min) of the first series was analyzed by TEM in more detail (see Figure 8). The \{110\} facets are well developed and smooth. No facets of other orientations are visible. The tip is very sharp with curvature radius of a few nm. There is no sign of damage introduced by the etching. However, no QW is visible, it is very likely that the structure is etched too deep.

As mentioned earlier, for the second series of samples, small pyramids of the order of 100 nm are formed after a short etching time. It has been possible to cut (in a focused ion beam [FIB] system) through pyramids of such an etched 5xQW structure. The lower two QWs are shown in Figure 9 and possibly a third
one at the top. The orientation of the side facets remains the same and there is no sign of a changed etching process induced by the QWs or other damage like cracking. At the bottom of the left pyramid is an inclined facet visible, which supports the idea that intermediate facets are formed in the etching process, as discussed earlier, and if a (1T0T) facet is formed, it is conserved. Therefore, after a pyramid is formed, its shape remains essentially the same but eventually the etch depth becomes so large that all QWs are etched away.

In the TEM image, the first QW seems to be thicker than the upper ones and the upper and lower interfaces are rough (see Figure 9). This is in contrast to Ga-face MQW structures:

The QWs have essentially the same thickness and the lower interface GaN $\rightarrow$ In$_x$Ga$_{1-x}$N is atomically smooth. This requires further optimization.

3.4. Pyramid-Related PL

One of the etched samples (5xQW, $x_{In} = 17.6\%$, first series, pyramids like the ones in Figure 8) was investigated by PL at low temperatures in more detail. For low excitation powers, QDs are expected to show narrow emission lines. In Figure 11, a typical spectrum with high spatial resolution is shown with quite narrow emission lines (linewidth of a few meV). As discussed earlier, the samples suffer from inhomogeneity, the PL efficiency

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**Figure 8.** Bright-field TEM images of a single pyramid (sample of the first series). The (1T0T) facets are well developed and smooth. The tip is very sharp, and the curvature radius is estimated to be 1.5 nm in this case. However, no QW is visible, likely etched too deep (courtesy of Zeiss).

**Figure 9.** TEM image (HAADF, STEM) of an etched sample of the second series. It contains five QWs and the nominal thicknesses of the top four QWs are 1.0 nm and the nominal thickness of the first one indicated by the arrow is 1.5 nm, respectively. The inclined facet at the bottom, which is indicated by the dotted lines, deviates by $\approx12^\circ$ to the (1T0T) facet. The position of the two upper QWs is marked by the pink dashed line.

**Figure 10.** Same as Figure 9 with the first QW (marked by the arrow in Figure 9) at higher resolution.
Figure 11. μ-PL spectrum of an etched sample grown with a 5xQW structure of the first series with a morphology similar to that shown in Figure 4 (focus 2 μm, confocal setup, 320 s integration time) at low temperatures. The full width at half maximum (FWHM) is \( \approx 2 \) meV.

Figure 12. The surface was covered (for simplicity) with a 300 nm-thick carbon layer, which was patterned by FIB (dark spots in the image). Afterward, the sample was etched for 80 s in KOH (aq.). The carbon masks protect the surface from etching. Some underetching can be seen at the rim of the dots. The side facets are rough but have an angle close to that expected for \( \{1101\} \) facets.

is low, and due to the very long integration times, a detailed analysis at low temperatures is hardly possible. To facilitate the investigations, prior to etching, masks have to be applied to define the positions of the pyramids. This work is in progress.

4. Summary and Outlook

A new concept is proposed for the realization of structures with a large number of nanooptical light emitters based on pyramids with similar emission properties. In a first step, QW structures are grown on N-face GaN. In a second step, the layers are etched and pyramids are formed which contain \( \text{In}_{x}\text{Ga}_{1-x}\text{N} \) nanostructures or QDs. The feasibility of the concept is demonstrated. 1) Pyramids with smooth facets and very sharp tips with curvatures in the nm range have been produced. This allows a QW being located close to the tip. 2) The pyramids can be produced to contain InGaN close to the tip. The QWs do not prevent sharp tips to be formed and are not lost in the etching process by any kind of relaxation process or the like. 3) Some of the pyramids contain \( \text{In}_{x}\text{Ga}_{1-x}\text{N} \) QDs as indicated by narrow PL lines and TEM images. InGaN is not damaged or seems to be otherwise affected by the etching process. 4) Titanium masks have been applied and those masks are stable in the hot KOH-based electrolyte on N-face GaN. As the masks cover and protect the GaN in the region, where the tip of the pyramid is intended to be formed, the height of the pyramid is given by the etch depth, which could be measured with high precision and could be determined in advance by the etch rate and etching time. This is a quite conventional approach and various processes of such kinds are well established. However, this requires that some underetching of the masks occurs, which is known to be the case, and that in fact \( \{1101\} \) facets are formed. The latter is expected from the results in the study by Tautz et al.\[12\]. 5) By FIB and applying masks, we can influence and likely control the initial phase of the etching process. This allows pyramids being placed at special locations. A preliminary experiment applying carbon masks (dark discs, carbon for simplicity) demonstrates that this is possible (result shown in Figure 12). The side facets are quite rough due to the less-defined initial phase of etching or/and the nonperfect masks. However, the angles are about 60° ± 5° and thus are close to that expected for \( \{1101\} \) facets. These pyramids are still truncated but it should be possible to achieve sharp tips below the masks by optimizing the etching time and layered structure.

Future work aims toward an improvement of the efficiency of the QWs and consequently of the QDs. Furthermore, a better control of the etching process is required, which turned out to be a key issue. A homogeneous etching process will eventually allow arrays of single-photon emitters to be produced.

5. Experimental Section

In this study, we used commercially available low-offcut-angle (0.5°…2.0°) pseudobulk GaN substrates (from Kyma Technologies) with low nominal defect densities. The first series was undoped and the second series was n-type doped. The layers were grown by low-pressure metal–organic vapor-phase epitaxy (MOVPE) in a horizontal reactor (Aixtron AIX 200RF) with trimethylgallium (TMGa), triethylgallium (TEGa), trimethylindium (TMIIn), and ammonia (NH\(_3\)) as precursors and hydrogen or nitrogen as carrier gas. First, an undoped GaN layer with thickness of about 300 nm was grown using standard growth conditions: 6 slpm total flux, 1180 °C, 100 HPA, 500 sccm NH\(_3\), and TMIIn 71 \( \mu \)mol min\(^{-1}\). The \( \text{In}_{x}\text{Ga}_{1-x}\text{N} \) QW region was grown with total pressure of 200 HPA with nitrogen as carrier gas. The growth temperature \( T_G \) for \( \text{In}_{x}\text{Ga}_{1-x}\text{N} \) layers was varied between 770 and 855 °C. In all experiments, the TMIIn flux was 2.0 \( \mu \)mol min\(^{-1}\) and a TMIIn/TMGa flux ratio of about 4.5 was obtained. The fivefold multiquantum well (MQW), double QW, and single QW structures were grown in continuous mode—growth times and temperature ramping in barriers as for Ga face—with a GaN barrier thickness of nominally about 19 nm and a QW thickness of about 1.5 or 1.0 nm for all samples, respectively. The growth rate of the QWs under these growth conditions was expected to be 1.05 nm min\(^{-1}\) and that of
the GaN barrier 0.83 nm min\(^{-1}\).\(^{[21]}\) The V/III ratio was varied between that used for Ga-face growth and reduced by a factor 5 to improve the quality of the QW structure, which follows the results reported in Keller et al.\(^{[14]}\)

All temperatures were readings of a thermocouple placed inside a SiC-coated graphite susceptor. The sample was placed on a disc, which rotated in a circular opening of the susceptor. Due to the limited thermal contact between the disk and susceptor, the temperatures at the growth front were estimated to be 70 °C lower than the thermocouple readings for the indium-containing layers.

After growth, the surface morphology of the samples was characterized by atomic force microscopy (AFM) in noncontact mode and scanning electron microscopy (SEM, JEOL JSM 5600).

Transmission electron microscopy (TEM) images were taken at Zeiss in a bright-field mode at 200 kV and at our site using a Jem Neoarm 200F (200 kV) with an annular dark-field (ADF) detector (high-angle-annular dark-field [HAADF] images and scanning TEM [STEM] mode). Optical properties were obtained by conventional photoluminescence (PL) at room and low temperatures (15 K) using an argon-ion laser (lines at 380, 350, 334 nm) and a laser diode at 376 nm with a focus diameter of about 50 or 2 μm, respectively. For structural information, we applied high-resolution X-ray diffractometry (HRXRD) using a triple-axis Panalytical Xpert Pro system with Cu Kα1 radiation. From the spacing of the superlattice peaks measured by HRXRD, we obtained the period of the structure and from the zero-order peak the average indium concentration \(x_{\text{In,eff}}\). The indium concentrations of the QWs \(x_n\) were determined from the period, \(x_{\text{In,eff}}\), and the growth times, assuming that the surfaces were abrupt and growth rates depended linearly on the incorporated group-III elements. The thicknesses of wells and barriers were compared with those measured using TEM and HRXRD, which were usually in very good agreement (for Ga face).\(^{[22]}\) For N-face growth, the superlattice peaks were very weak for the grown MQW structures, which was caused by inhomogeneities in thickness and period. Therefore, the values of \(x_n\) were less accurate compared with Ga face.

Metal masks were evaporated by e-beam in a commercial system (Edwards Vacuum Auto 306). The samples were not intentionally heated during evaporation and not annealed after deposition. Shadow masks used to cover half of the sample during the deposition process. In other experiments, Ti plates of rectangular shape were used to cover half of the sample during the deposition process.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

Research data are not shared.

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