From InGaN pyramids to micro-LEDs characterized by cathodoluminescence

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Abstract
We present a study of the optical properties of various steps in the process of fabricating micro light-emitting diodes (μ-LEDs) based on quantum wells embedded in micron-sized InGaN platelets. In this study, we focus on structures for red emission, but the technology is equally suitable for the less technologically challenging blue and green emitting μ-LEDs. The starting point is growth of an InGaN pyramid with a sub-micron sized hexagonal base. The pyramid is flattened to create a flat top c-facet for the subsequent InGaN quantum well growth. We compare two approaches, reshaping of the pyramid by high-temperature annealing; and by chemical mechanical polishing. Their merits are discussed. The flattened platelets are used as templates to grow low-strain single quantum wells in both heterostructures and full LEDs including n- and p-barriers on either side of the quantum well. The structures are investigated in terms of homogeneity in peak energy position and intensity using hyperspectral cathodoluminescence imaging. We observe that the main contribution to inhomogeneity is the growth of the initial pyramid, that exhibits regular, facet driven variations in the In incorporation.

1. Background

III-Nitrides are a group of semiconductors with a large span of bandgap energies, ranging from InN (0.7 eV), via GaN (3.4 eV) to AlN (6.2 eV) at room temperature (RT, 293 K) [1]. Binary and ternary combinations of AlInGaN can therefore cover the full spectral emission range from infrared (InN at 1.7 μm) to ultraviolet (AlN at 200 nm) [2, 3]. One of the main technological issues is that there is large lattice mismatch between the binary components, 2.5% between AlN and GaN; and 11% between GaN and InN [2]. This has implications for growth of heterostructures with the large differences in composition needed for fabrication of many III-Nitride devices, especially green and red light-emitting diodes (LEDs). The large lattice mismatch potentially leads to the introduction of misfit dislocation and other growth defects [4–7]. The crystal structure of III-Nitrides is mostly wurtzite, and layers are typically grown in the polar c-direction (0001). The polar direction leads to a spontaneous polarization of charges at heterointerfaces [8, 9]. The strain in the structures also introduces a piezoelectric polarization of the carriers in quantum wells (QWs) [10, 11]. The two polarization effects introduce electric fields in the QW, leading to the quantum confined Stark effect (QCSE) [5, 6, 12]. This effect separates electrons and holes, reducing the overlap between them in the QW resulting in lower emission efficiency. This is more severe for wider QWs. It also leads to a blue shift of the emission peak with excitation power, for instance the driving current of an LED. As an example, blue shifts of 60–110 meV of the electroluminescence have been demonstrated as the current density was increased from 1–40 A cm⁻² for yellow and red [13]. Dislocations have a negative effect on device performance as they act as non-radiative recombination centres competing with the radiative recombination [14, 15]. Dislocations can also reduce device lifetime. Another issue is the lack of available affordable lattice-matched substrates for the growth. III-Nitrides are often grown on (111) silicon or sapphire covered with a few micron thick GaN buffer layer.
The growth of the LED structures is performed in several steps, as presented in a series of publications [2, 23, 24]. The starting point for the growth is a substrate with a suitable lattice matching for this range, growth on a GaN substrate will need to be modified. In order to improve the EQE of e.g. red LEDs, it is essential to reduce the strain between the barriers and QW while keeping the carriers confined. Though red LEDs can be fabricated using AlInGaP [17], there is a significant interest in developing III-Nitride structures for this spectral range, specifically since the red-emitting AlInGaP devices loose efficiency when the discreet emitters are processed to very small sizes, typically less than 50 μm [18]. Our long-term focus is on micro-LEDs (μ-LEDs) based on InGaN for display applications, using the direct emission from the LEDs for individual display pixels [19]. This would enable the use of similar driving circuits for all three colours, rather than a different design needed for the red AlGaInP. This would potentially reduce the production costs of displays. The QWs need to have an In concentration in the QW of about 25% for green LEDs and 35% for red LEDs [4, 20, 21]. Using the same GaN barriers as for the blue LEDs will inevitably lead to structural defects, as we show below. The barriers should therefore ideally be InGaN, with about 10% (20%) for green (red). Since there are no substrates with suitable lattice matching for this range, growth on a GaN substrate will need to be modified. This is typically done by growing a graded buffer or more complex layer structures to reach the desired In content in the barrier. This must be done without introducing any additional defects. Here, we present a different approach, using relaxed InGaN platelets grown directly out of nanometre-sized holes in a mask by selective area epitaxy on GaN. We present a cathodoluminescence (CL) study of the evolution of the structure from a six-sided InGaN pyramid with a hexagonal base to a flat topped, red μ-LED.

2. Preparation and growth of the structures

The growth of the LED structures is performed in several steps, as presented in a series of publications [22–24]. The starting point for the growth is a substrate with a (0001) GaN buffer layer on either (111) Si or Sapphire substrates. This is covered with a 30 nm thick film of SiN, into which a hexagonal array of circular holes is defined by either electron-beam lithography or imprint lithography. The holes are made by reactive ion etching, with a typical diameter of around 100 nm and the spacing is 1 μm. The mask effectively blocks a large portion of the threading dislocations in the substrate from propagating into the selective area growth, illustrated in figure 1 [25]. The growth is seeded by a short GaN nanowire, with a diameter defined by the hole diameter. This nanowire, just filling up the holes in SiN, is the starting point for growth of the InGaN pyramids. Rather than forming a nanowire during growth, like in that of the case of GaN, the InGaN growth develops inclined (1011) facets (s-facets) resulting in a pyramid with a hexagonal base, figure 1(a) [22].

In order to reshape the pyramid, figure 1(b), into a structure with a flat top defined by a large c-facet, we have used two different approaches. The initial approach is to perform an in-situ reshaping of the pyramid [23]. This is done through annealing at an elevated temperature, where InGaN decomposes from the top apex of the pyramids and the s-facets are intact under NH3 ambient [23]. By controlling the annealing time, a thin platelet with a top c-facet can be obtained. The second approach is to remove the tips of the pyramids mechanically, using chemical-mechanical polishing (CMP) [24]. This results in a smooth and domed-like top surface, in contrast to the rough but flat c-plane formed by annealing.

Figure 1. Illustration of the selected area growth steps for the array of InGaN structures. (a) The first step is to grow a hexagonal pyramid from a hole in mask. The pyramid is defined by the slow-growing s-facets. (b) The tips of the pyramid is removed by either in-situ annealing or CMP to leave a platelet with a flat c-facet. (c) After the flattening, a second layer is grown in order to ensure a flat top surface for the subsequent QW growth. This layer has the same nominal composition and doping as the pyramid. (d) The QW is grown with a higher In content than the barriers. (e) The final step is to grow the top barrier, which has the same composition as the lower barrier. For a full LED structure, the lower barrier layers are n-type and the top barrier is p-type. As the growth proceeds, the area of the fast-growing c-facet is reduced.

The large range of bandgap energies means that it is possible to design LEDs in the colour range from infrared to ultraviolet [1]. Whereas blue LEDs exhibit external quantum efficiency (EQE) of over 80%, the number drops rapidly as the energy is reduced, to less than 10% for red LEDs [13, 16]. The main causes of the drop are defects introduced by the mismatch and the QCSE. In order to improve the EQE of e.g. red LEDs, it is essential to reduce the strain between the barriers and QW while keeping the carriers confined. This can be done through annealing at an elevated temperature, where InGaN decomposes from the top apex of the pyramids and the s-facets are intact under NH3 ambient [23]. By controlling the annealing time, a thin platelet with a top c-facet can be obtained. The second approach is to remove the tips of the pyramids mechanically, using chemical-mechanical polishing (CMP) [24]. This results in a smooth and domed-like top surface, in contrast to the rough but flat c-plane formed by annealing.
After the c-facet is established, the subsequent growth starts with a lower barrier layer, figure 1(c), ensuring that the c-facet is smooth for the growth of the single InGaN QW, figure 1(d), or a number of identical InGaN QWs. This is an important step, as the composition of this barrier layer should match that of the base of the pyramid. A smooth upper surface is also crucial in order to have a homogenous thickness of the QW, which is essential for obtaining a narrow line width of the emission. For the LED structures, this layer, as well as the original pyramid is n-type, using Si as the dopant. For this study, we have aimed at a structure containing a single QW with a thickness of about 3 nm. After the growth of the QW, an upper barrier is grown with the same composition as the lower barrier, figure 1(e). In case of a full LED structure, the upper barrier is p-doped with Mg.

3. CL studies

This study focuses on characterizing the pyramids, platelets and LEDs using CL in a conventional scanning electron microscope (SEM, Zeiss EVO MA 15). The structures were studied as grown, both in top view and cleaved side view. The latter is possible as, the structures cleave through and stick to the substrate, unlike most nanostructures that tend to break away from the edge. It is possible to locate platelets that are cleaved through the centre as the stem that seeds the original nanowire is visible in SEM images due to the contrast between the GaN stem and the SiN mask. The stem is only visible when the pyramid is cleaved through the centre. The studies were mostly done at 293 K, but also at 8 K using a liquid helium cold stage (Gatan CF 302 M). The emission was collected by a parabolic mirror (Delmic Sparc - acceptance angle 1.46π Sr) and the light was focused on the entrance slit of a monochromator (Andor Shamrock 193i). Monochromatic images and spectra were recorded using a GaAs photomultiplier tube (PMT, Hamamatsu GaAs R943-2), and hyperspectral imaging was done using a Si charge-coupled device (CCD, Andor Newton DU970P-UVB). The SEM was operated at 5 keV, typically using a probe current of 10–50 pA. This gives a penetration depth of about 100 nm [26]. During the recording of the CL images, an SEM image was recorded simultaneously using an in chamber secondary electron (SE) detector. All data was recorded by Delmic’s ODEMS software. Images are described as SEM, when using the SE detector and monochromatic images are identified by the detection energy.

The detection conditions were chosen using several criteria. The grating was selected to cover a suitable spectral range and the input slit of the monochromator was chosen by balancing the need for spectral resolution and signal intensity. A third consideration is pixel density and dwell-time in the images; and the image recording time. The typical approach is to set the entrance slit width to where the GaN emission peak, which is the narrowest peak, is not limited by the spectral resolution. The pixel dwell-time is chosen to have a sufficient signal-to-noise ratio. The pixel density is given by the recording time for the hyperspectral image.

The data is presented as spectra and images. Average spectra are from the entire region of the images, in cases where there are several spectra in one plot, they are normalized to the maximum intensity in each spectrum. We also present spot mode spectra along lines in the images, presented here normalized and displaced. The CL images presented here are extracted from hyperspectral images or recorded as single energy images, and they are presented in grey scale. They are normalized, where black represents no emission and white the maximum intensity in the entire image in the range of the extracted energy. This way we present local variations in the emission intensity. To compare the intensity of the images, we include the normalization factor in the images, unless the images are presented with the same grey scale. The data is also presented as peak maps, where the energy of the most intense emission is shown for each pixel in the image.

4. Full pyramid

The full pyramids have been studied in both top view and side view on a cleaved substrate. Figure 2 shows an SEM image of an area of the pyramid array in (b and d) and a single pyramid in (e). The CL spectrum in (a) shows three different emissions, the near bandgap emission (NBE) of the GaN substrate at 3.35 eV and the high-energy part of the yellow luminescence (YL) also from the substrate [27]. The spatial origin was confirmed by imaging these emission peaks. The main emission at 2.6 eV is from the pyramid. When extracting a series monochromatic images from the hyperspectral data, the emission pattern changes. With increasing detection energy, the emission appears to shift towards the tip. These variations are consistent with variations in the composition in the pyramid, less In (higher energy) near the tip. The dark lines in the images, especially the low energy images, at the corners between the side facets and the tip could be an artefact of escaping secondary electrons (bright in the SEM images) leading to reduced excitation in these areas [28].

The top view imaging is not conclusive, as it does not account for variations in the emission with distance from the surface of the pyramid. In order to understand the variations, we have studied the same sample in side view, where some of the pyramids are cleaved through the centre. The position of the cleave can be confirmed by
the appearance of the seeding GaN NW above the substrate, visible in figure 3(a). The series of monochromatic images (b)–(f) shows a clear trend that the In content decreases from the centre of the structure to the surface of the pyramid. This is especially clear at the tip of the pyramid. By extracting a series of spot mode spectra from the base to the tip along the centre, it is clear that there is a blue shift towards the tip. The material at the tip is thin, and some of this emission comes from the surrounding pyramids, as electrons from the beam penetrate the tip and are scattered to the nearby pyramids. These last spectra in the series therefore appear to show a slight red shift. The variation in the composition is linked to the increased area of the structure tend reduce the In incorporation as the growth proceeds, revealed as a gradual expansion of the emission pattern with increasing detection energy, (b)–(f). At the centre of the base of the pyramid, the dark void is caused by the seeding GaN NW. The main emission from the seed is YL and is out of the detection range in these images and spectra. Even when recording images of the YL, it is difficult to isolate any emission from the NW, as the nearby substrate emits
non-native substrates. To understand the growth and the limitations, we grew a series of full QW structures.

One of the advantages of growing QWs on InGaN platelets is that they can be grown relaxed and defect free on terms of quality and homogeneity. Figure 5 shows a platelet after reshaping with an additional 50 nm-thick layer remains. This is presented in figure 4. An SEM image of one thin base of a pyramid, around 50–100 nm thick is shown in figure 4(a). The monochromatic CL images (b)–(f) show that CMP is quite gentle and does not appear to introduce any defects due to the mechanical polishing [29, 30]. This series of images show changes in the contrast, most likely related to the In incorporation. Similar patterns could be related to optical modes in the platelet, but we observe similar patterns in asymmetric platelets that would not support the optical modes. At the base of the pyramid, it is clear that the In content is higher at the corners between the side facets, the ones that were dark in the top view images of the full pyramid. It is also clear that the In content is lower in between the corners and at the edges of the platelet. This series of images also shows the dark void in the centre, corresponding to the GaN seed NW, as discussed above.

The combination of the three views of the pyramids is an important illustration that more data can be needed to form a complete picture of a complicated structure. It also illustrates that it is important to control the compositional variations in the pyramid to achieve an as homogeneous and flat platelet as possible for the subsequent layers.

5. Platelets formed via high temperature annealing

It is of importance to understand the high temperature reshaping process and especially the resulting platelet in terms of quality and homogeneity. Figure 5 shows a platelet after reshaping with an additional 50 nm-thick InGaN layer on top, emulating the lower barrier that the InGaN QW (or QWs) will be grown on. The image was recorded at 8 K in order to improve the spectral resolution. Figure 5(a) is an SEM image of the reshaped and regrown pyramid, and (g) shows a spectrum from the single platelet in the SEM image of (a). Compared with the full pyramid, the 8 K spectral width of the reshaped and regrown platelet is significantly reduced, from over 200 meV for a pyramid to about 150 meV for the platelet. This indicates an improved homogeneity when the top of the pyramid is removed. The emission also appears to be stronger in emission compared with the YL of the substrate. Figure 5(b) is an image of the integrated emission from the platelet (2.52–2.87 eV), showing the total intensity variations, and (c)–(e) show the emission from the platelet using different detection energies. Figures 5(f) is a peak map, where the colour scale is a map of the energy of the maximum intensity in each pixel. The CL images reveal some random variations in the emission, both in energy and total intensity. The energy variations do not resemble the patterns in figures 2–4, indicating that the regrown layer has some random fluctuations in it, apparently unrelated to the original pyramid. It is also worth pointing out that the edges of the platelet appears to have less In in it, consistent with the growth of the original pyramid, as seen in figure 4(f). An important conclusion from the images is that there does not appear to be any visible dislocations, as there are no dark spots or lines in the CL images [31]. Though there are variations in the monochromatic images, when extracting the peak position of each pixel in the image, figure 5(f), the variations in the composition equate to about ±1% [2]. The variation could be related to random clustering of In. These variations are manageable, except for the larger deviation at edges of the platelet. The emission from the edge can be related to the original pyramid as seen in figure 4(f). The emission is significantly weaker from the edges, compared with the centre, which could be an effect of surface damage of the s-facets during the high temperature reshaping.

6. Full QW structures

One of the advantages of growing QWs on InGaN platelets is that they can be grown relaxed and defect free on non-native substrates. To understand the growth and the limitations, we grew a series of full QW structures.
nominally with the same QW, but on platelets with different In content. The nominal compositions of all barrier layers (original pyramid, lower and upper barriers) in the platelets were 0, 9 and 18% In, and 3 nm thick QW with about 35% In. The compositions of the barriers were confirmed from energy dispersive x-ray analysis (EDX) in the TEM study [23]. Due to the lattice mismatch, the In incorporation in the QW decreases with decreasing In content in the barrier, despite the same growth parameters. Data from this series is presented in figure 6. Going from 18 to 9 %, as expected there is a small blueshift of the emission, from 2.00 to 2.15 eV. This is consistent with an increased barrier height and therefore more quantum confinement, or a lower In content.

From TEM studies of these samples [23], we identify that the QWs are of similar thicknesses, but they are too thin for their compositions to be determined by EDX. Qualitative EDX data indicates a reduced In content in sample with the lower In content. Therefore, we cannot rule out that there is a difference in the composition of the QWs, which seems to be true since larger lattice constant (higher indium content in the barrier) facilitates the indium incorporation into the QWs [32]. Like for the barrier platelets in figure 5, there are some variations in the emission patterns when the entire emission peak is imaged, see figures 5(c) and (e). The variations differ from platelet to platelet, but as the patterns do not vary with detection energy the variations cannot be related to different emission energies, caused by either thickness or compositional variations. There is no QW emission from the edges due to a very low growth rate on the side facets.
The QW on the pure GaN platelets shows a different emission pattern. It exhibits a blueshift to around 2.9 eV, significantly larger than expected from the shift from the 18% to the 9% sample. The emission intensity is also significantly lower that for the QWs on the InGaN platelets. The CL imaging reveals the reason. The emission only originates from the edges of the platelet, where the growth rate is expected to be appreciably lower than on the flat top facet. We can exclude QCSE due to higher strain as a cause for the absence of QW emission, as we cannot observe any emission below the GaN NBE emission from the c-facet even at a 100 times higher probe current. From this sample, we can conclude that there probably is a fundamental limit for the possible difference in the In content between the QW and the barrier material. When the difference is too large, the QW or top barrier becomes defective with dislocations or other structural or point defects. From this study, it is clear that the compositions of the barrier and QW must be matched in order to achieve strong QW emission.

The intensity variations in figures 6(c) and (e) are intriguing, and we have recorded monochromatic images over larger areas of the arrays. This way, we can observe the variations with more statistics than for single platelets. Figure 7 shows a series of monochromatic CL images of an array of the QW-containing platelets of figures 6(b) and (c). We make two important observations from these images. The shapes of the bright areas of the individual platelets are quite random, and very few are homogeneous in the emission pattern. When the detection energy is varied, the same emission patterns appear, where the same bright (dark) areas appear in all images irrespective of the detection energy. This leads us to conclude that the intensity variations are not related to any shift in the emission energy, either due to thickness or compositional variations. Another important observation is that all the platelets show the same peak position which means that they have similar thicknesses and compositions.

7. Complete LED structure

The aim of this study is to investigate a working red LED made from an array of flat-top platelets. The final example in this study is therefore a full LED structure, including the contacts. These structures can only be studied in side view as the 1–2 μm thick top contact prevents excitation in top view. Figure 8 shows a single platelet, cleaved through the centre of the structure. This study was performed at both RT and 8 K, the latter to be able to observe emission from the Mg of the p-type top barrier. This emission is expected to quench at RT, just like for Mg in GaN [33, 34]. Figure 8(a) is an SEM image and (b)-(f) are monochromatic images extracted from hyperspectral images recorded at 8 K. A dashed line is included in the images to indicate the position of the QW. The lower barrier (d), (c) and (e) shows variations in the emission with detection energy, similar to what is observed in figures 2–4. (d) shows the emission from the QW with some intensity variations, though not as severe as in figure 7. The upper barrier shows two distinctly different emission energies located at different spatial locations, in the 2.6 eV image (e) it appears near the QW, and slightly higher up in the 2.4 eV image (f).

The spot mode spectra along the middle of the structure shows a number of peaks at different energies with different spatial origins. From bottom to top, there is a peak at 2.8 eV, which disappears at the position of the QW, a peak at 1.85 eV appears only at the position of the QW, and finally a peak at 2.4 eV that comes up from the position of the QW. It has a small shoulder on the high energy side. For comparison, a similar RT series does not show the jump down in energy of the barrier emission across the QW region, shown in figure 8(h). This is consistent with the Mg-related emission from the top p-type barrier, emission that is quenched at RT, leaving only the bandgap emission from the top [34]. The RT scan shows a good match of the composition of the lower and upper barriers. The peak at the high energy side of the 8 K Mg peak has its origin in how the upper barrier was designed. The first part of the upper barrier has a low Mg concentration and the second part has an increased concentration in order to improve the contact. Assuming a similar temperature behaviour of the Mg emission from our InGaN platelets as from GaN [34], we can expect a redshift of the emission with increasing Mg concentration. The nature of this emission is presently not known, but the high Mg concentration makes it likely
to be related to either donor-acceptor pairs or acceptor related complexes [34]. We can identify the emission just above the QW in figure 8(e) as the p-region with a low Mg concentration, and figure 8(f) as the region with a high Mg concentration. In addition, figure 8(h) shows a slight increase in the In content towards the top. The doping of Mg will be the focus of a study in the near future.

The supplementary information (available online at stacks.iop.org/NANOX/2/014006/mmedia) shows a video sequence of monochromatic images over the entire spectral range similar to the CL images in figures 8(b)–(f). Each image is presented in colour scale, normalised to the maximum intensity at each intensity. The video also shows the average spectrum from the area, with a cursor running over the spectrum as the images are presented.

8. Discussion and conclusions

The results from this study shows that it is possible to create arrays of micron sized InGaN LEDs in the colour range from deep red to blue, including green. These would be suitable to direct emitting RGB-displays. The array means it is scalable, depending on the need for size and intensity. The advantage of the pyramid/platelet approach is that each platelet is essentially dislocation free due to the dislocation filtering by the mask and size of the holes. One condition to ensure that the structures are free from dislocations is that the combination of barriers and QW(s) has a low lattice mismatch, similar to what is used in blue LEDs, compare figures 6(e) and (g). This was illustrated by the series of samples with different barrier compositions, where an InGaN QW emits strong emission, until the difference in In content becomes too large and the QW does not emit any light from the c-facet, only from the side s-facets.

There are several issues that need to be improved. The transformation from a pyramid to a platelet is a critical step. The reshaping by in-situ annealing results in an almost perfect structure with a flat c-facet, but with potential facet damage. Although the CMP approach results in a dome-shaped c-facet, the remaining platelet is of better quality [24]. We have shown that the CMP process does not damage the top surface, as shown in figure 4. The original platelet (remaining part from high temperature reshaping or polishing of the pyramids) shows regular variations in the composition, which is a potential issue when growing the lower barrier. The solution may be to thin the platelet down to a minimum and let the growth of the lower barrier smooth the composition. There is also work to be done on the quality of the QW. The QWs on different platelets emit at identical energies and there is very little variation in the QW emission from the individual platelets. The issue is that there are, what appears to be random intensity variations. The emission patterns vary from platelet to platelet, unlike the original pyramids that show very similar variations in composition and intensity. Studies of the statistics of these variations within an ensemble of these relaxed platelets will be an important field of future investigation.

Finally, the full LED structure shows a number of important features. The RT study shows that the barriers are quite well matched in composition, whereas the low-temperature study shows that there is a regular pattern of compositional variations in the lower barrier, a mixture of the original pyramid and the regrown layer. The low-temperature study also identifies the Mg doping and confirms that the upper barrier consist of two layers of InGaN with different Mg concentrations. With suitable reference samples, it should be possible to use the energy position and shape of the emission to determine the concentration.
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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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