Realization of Ultrahigh Quality InGaN Platelets to Be Used as Relaxed Templates for Red Micro-LEDs

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INTRODUCTION

Self-emissive displays based on red, green, and blue micro light-emitting diodes (LEDs) are drawing increasing attention. While micro-LEDs made of nitride semiconductors are chosen for green and blue colors, red micro-LEDs form a bottleneck for large-scale display applications.1 Red nitride micro-LEDs are expected to have more stable light output and less efficiency is still too low.2 In order to improve the performance of red LEDs, high quality relaxed InGaN platelets need to be developed for InGaN quantum wells (QWs, x < y), rather than using GaN templates.3–10 Compared with an InGaN QW grown on a GaN template, the same QW grown on an InGaN template has a much smaller compressive strain, leading to more efficient indium incorporation, lower density of defects, and a reduced quantum-confined Stark effect. InGaN layers with an indium content around 5% have previously been studied as a buffer layer or underlayer for the InGaN QW growth and the observed luminescence enhancement was attributed to the filtering of point defects by this InGaN layer.11–13 In order to achieve highly efficient green and red LEDs (indium contents in the QWs are 25 and >35%, respectively) which are important for ultrahigh resolution and high brightness display applications based on micro-LEDs, high-quality InGaN layers with indium contents well above 5% are clearly needed as a buffer layer. Solar cell applications were reported for thick InGaN layers with indium contents close to 40%.14 However, a breakthrough seems to be difficult to achieve without solving the growth issues, including the key control of the surface smoothness.15 A similar situation also occurs in the studies of p-type InGaN layers with Mg doping. P-InGaN can be obtained with an indium content of up to 35% with a low activation energy of Mg acceptors.16 However, the rough surface of such layers prevents the fabrication of devices with high performance.

GaN templates, either GaN films grown on dissimilar substrates or free-standing bulk GaN substrates, have been used to grow thick InGaN layers.11,12,17–21 The compressive strain from the lattice mismatch and dislocations from the underlying GaN films are responsible for the low-quality InGaN layer growth. The rough surface, caused by the strain relaxation, was found to improve by periodically inserting thin GaN layers during InGaN growth.20,21 In this way, thick InGaN layers with an indium content of up to 16% were obtained despite the pseudomorphic growth.21 Recently, bulk GaN substrates with low dislocation densities have been used to grow thick InGaN layers in order to control the dislocation-induced pit formation and spiral growth.17,19,22 However, the low density of dislocations prevents the InGaN layer from...
relaxing and features of step-meandering instabilities appear on the surface. Besides the growth on GaN templates, other substrates that can be lattice-matched to InGaN with a certain indium content were applied to realize strain-free InGaN growth. ScAlMgO₄ (0001), lattice-matched to In₀.₁₇Ga₀.₈₃N, was used to study the growth of thick InGaN layers and the corresponding applications on LEDs emitting red light.¹²³ Halide vapor phase epitaxy, which is used to grow free-standing bulk GaN substrates with a high growth rate, has also been applied to investigate the growth of thick InGaN layers.²⁴

In this work, we present a method to fabricate arrays of submicrometer-sized c-oriented InGaN platelets with indium contents of up to 18%. The InGaN platelets are predominantly relaxed, free from dislocations, and offer a top c-plane with single bilayer surface steps. Strong photoluminescence (PL) at room temperature (RT) was observed with a full-width at half maximum (FWHM) of about 140 meV at 2.63 eV (indium content: 17%).²⁵ The InGaN platelets were achieved by chemical mechanical polishing (CMP) of InGaN pyramids. Using metal–organic vapor phase epitaxy (MOVPE), InGaN layers can be grown on such a dome-like surface with thicknesses from a few tens of nanometers up to a few hundreds of nanometers. The dome-like InGaN templates are fabricated on GaN/sapphire substrates with a small contact area through 100–200 nm large openings in a growth mask.²⁶ In this way, the lattice mismatch strain has no significant effect on the growth and the InGaN platelets are predominantly relaxed. Compared with our previous work on InGaN platelets where dislocations were formed because of the large indium content discontinuity between different layers,²⁷ the direct InGaN growth on the CMP-formed InGaN templates with similar indium contents completely eliminates the formation of dislocations. Such high-quality InGaN platelets can be used as templates to grow green and red LEDs on the top c-plane with improved lattice match between the templates and InGaN QWs.

Experimental Section
InGaN Template Preparation Using CMP. A series of scanning electron microscopy (SEM) images given in Figure 1 show the procedures to fabricate the InGaN templates by polishing dislocation-free InGaN pyramids. The InGaN pyramids were grown selectively on substrates of GaN/sapphire from 100 to 200 nm large openings in a SiNₓ mask by MOVPE (Figure 1a). Such InGaN pyramids, defined by 6 equivalent {1011} planes, were realized with an indium content of up to 20% (PL peak around 2.48 eV at RT).²⁶ In order to prevent the pyramids from being detached during the CMP, a layer of SiO₂ was deposited on the samples by plasma-enhanced chemical vapor deposition (Figure 1b). Such samples (10 × 8 mm in size) were then polished with a PMS CMP machine from Logitech. A slurry (SF1) from Logitech with SiO₂ nanoparticles in it was used. Figure 1c shows the structures after CMP, where the top part of the pyramids is polished away, leading to the formation of a top surface. Depending on the polishing time, structures thinner than 100 nm can be obtained. In order to remove the remaining SiO₂ nanoparticles and SiO₂ layers between the polished structures, HF etching was conducted on the polished samples with the original SiNₓ growth mask as an etch stop layer (Figure 1d).

InGaN Growth by MOVPE. InGaN growth was carried out on the templates shown in Figure 1d with similar indium contents. An MOVPE system with a 3 × 2 in. close-coupled showerhead was used to grow InGaN. Triethylgallium (TEG), trimethylindium (TMI), and ammonia were used as Ga, In, and N precursors, respectively. For the InGaN growth with the indium contents of 9–11 and 17–18%, the same ratio (0.9) of TMI/(TEG + TMI) in the gas phase was used. The V/III ratio was also the same for both cases, which was about 4000. In order to increase the indium content from 9–11 to 17–18%, the growth temperature was lowered from 790 to 770 °C.

Characterization. A Hitachi SU8010 cold field emission SEM was used to record SEM images at an acceleration voltage of 15 kV. For atomic force microscopy (AFM) investigation, a Bruker Dimension Icon (300) microscope was operated in the peak force tapping mode with Si cantilevers. A 300 keV JEOL 3000F transmission electron microscope was used to characterize the sample using high-angle annular dark field. The transmission electron microscopy (TEM) specimens were prepared with an FEI Nova 600 dual-beam FIB/SEM system. Optical properties were characterized with micro-PL and cathodoluminescence (CL) at RT. A 375 nm UV laser was used for PL measurements, and the CL characterization was conducted with an acceleration voltage of 5 kV and a probe current of 20 pA. Time-resolved PL was measured in a backscattering configuration. A frequency-doubled Ti:Sa laser line having 100 fs pulses was used for excitation at 370 nm. PL decay was measured using a streak camera having a temporal resolution of around 2 ps.

Results and Discussion
Figure 1c,d shows top-view SEM images of the structures after CMP. The brighter area at the structure center is due to the InGaN growth on a local c-plane with less indium incorporation, in contrast to the major growth on {1011} planes during the pyramid formation.²⁸ Figure 1e shows a side-view SEM image of the polished structures, illustrating that the actual top surface has a dome shape, where the height offset is about 60 nm in contrast to the about 550 nm diagonal size of the top surface. The dome-like surface shows a larger curvature at the periphery than the central part. Different polishing rates between SiO₂ and InGaN may be the origin of the dome-like surface formation. However, the nonplanarized/three-dimen-
sional surface after the SiO$_2$ deposition cannot be excluded as the cause for the formation of this dome-like surface.

InGaN samples, as shown in Figure 1d, were loaded back into the MOVPE reactor for the additional InGaN growth in order to flatten the top c-plane. The conditions were adjusted to achieve PL emissions at a similar photon energy as the original pyramids, which in this case is 2.88 eV, corresponding to an indium content of 11%.25 Figure 2 shows top-view SEM images of samples grown for different times from 250 to 3500 s. After 250 s growth, steps are observed on the top surface from the center to the periphery. With continued growth, the number of steps on the top surface is reduced because the steps close to the periphery will reach the inclined {10\(\overline{1}1\)} facets and terminate there because of the extremely low growth rate on the {10\(\overline{1}1\)} planes. Meanwhile, the flat plateau, formed at the top surface center, expands to cover the whole top surface when the growth time is longer than 2500 s. Even though this plateau seems to expand isotropically on the top surface, a few notches are still observed around the plateau, as shown in Figure 2d−f. There are two types of step edges for nitride growth on the c-plane, denoted as type A and B, depending on the number of dangling bonds on the edge atoms.28 During the growth, type A and B step edges can form a zigzag-shaped step,19 which may be a reason for the notch formation. However, surface damage during CMP could not be ruled out for the formation of such notches. The pits shown at the periphery to the inclined {10\(\overline{1}1\)} facets in Figure 2f are caused by the notches, rather than by dislocations. With prolonged growth times, the pits are filled up and a flat top c-plane is obtained, as shown in Figure 2g, where the thickness of the grown InGaN layer at the center is 40–50 nm. Note that the base size of the platelets does not show any visible change for any of the samples in Figure 2 because the growth on the

Figure 2. Top-view SEM images of the samples with InGaN growth for different times on the as-polished InGaN templates: (a) 250, (b) 500, (c) 1000, (d) 1500, (e) 2000, (f) 2500, and (g) 3500 s. Scale bar: 200 nm.

Figure 3. AFM characterization of the top surface after InGaN growth for different times: (a) 250, (b) 1000, (c) 2000, and (d) 3500 s. Scale bar: 100 nm. Height profiles along the lines in (a−d) are presented in (e), and (f) shows the expanded height profiles for growth times of 2000 and 3500 s. The green arrow in (f) indicates nucleation of a surface step.
inclined \{10\overline{1}1\} facets is extremely slow due to N-termination of the surface.29

AFM was conducted on the samples grown for 250, 1000, 2000, and 3500 s. The results are presented in Figure 3. The surface steps that were observed by SEM, as shown in Figure 2, are also clearly visible in the AFM images shown in Figure 3a–c. According to the line profiles shown in Figure 3e, the step height is much larger than 0.26 nm which is the value corresponding to a single bilayer thickness on the c-plane for this indium content. The observed steps can be as high as 6−7 nm, corresponding to a step bunching of over 20 single bilayers. The surface polished by CMP, prior to InGaN growth, is expected to exhibit single bilayer steps.30 If we assume that the dome-like surface shown in Figure 1e is evenly decorated with single bilayer steps, there are over 200 such steps from the top surface center to the edge, and the average step width is about 1 nm based on the dome height and the top surface extension shown in Figure 1e. Such a step width is much smaller than the adatom diffusion length, which leads to a step instability during the growth. As a result of this, step bunching usually takes place, explaining the surface structures observed in Figures 2 and 3.12 In Figure 3c, a bump can be observed at the surface center, and the corresponding line profile in Figure 3f shows this to be one single bilayer high. This indicates that the nucleation of surface steps is from the center of the plateau before all the bunched steps are terminated by the inclined \{10\overline{1}1\} facets. Once the plateau expands over the top surface completely, the nucleation site moves to the six corners between the top c-plane surface and the inclined \{10\overline{1}1\} facets, as shown in Figure 3d. This is in agreement with what we found in our previous work, where the c-plane was formed by high-temperature annealing of InGaN pyramids.27 New surface steps initiated at the six corners propagate toward the center of the top surface and a low-lying “valley” is observed because of this (Figure 3d). The corresponding line profile in Figure 3f shows that the height of all the steps is 0.2−0.3 nm, in line with a single bilayer height of 0.26 nm.

The lateral surface development during the growth is illustrated in Figures 2 and 3. In order to better understand how the growth takes place in the vertical/c-direction, four thin GaN layers were inserted as markers during the InGaN growth and the first GaN layer was grown directly on the polished InGaN surface. Figure 4a shows a cross-sectional scanning TEM (STEM) image recorded using high-angle annular dark field, where GaN is dark and InGaN is bright in contrast. Schematics shown in Figure 4b–g are based on Figure 4a, showing how the growth develops for the first three GaN marker layers and the corresponding InGaN layers. Figure 4a shows that the growth mainly takes place at the curved periphery sides for the growth to the second GaN marker layer (also refer to Figure 4b–d). With the growth of the third InGaN layer, the dome-like surface is almost completely flattened. The first GaN marker layer, grown directly on the polished InGaN surface, shows step bunching which leads to separated sections in the area between the top surface center to the periphery. In the area close to the periphery, the first GaN marker layer seems to grow continuously on the curved surface due to the larger curvature. InGaN growth continues from the GaN step edges. During the same growth section of InGaN, the upper InGaN terrace can grow onto the bottom InGaN terrace and a dark interface is left. One such example is shown in Figure 4.
by the arrow in Figure 4a and this is marked accordingly in Figure 4e with a dashed black line. This could arise from indium desorption on the free InGaN terrace before it is covered by an upper one, leading to elemental contrast. Besides the growth on the small terraces at the periphery, the second and third InGaN layers at the surface center grow symmetrically on both sides from the center. This further confirms the nucleation at the plateau center before the dome-like surface is completely flattened, which was observed by the AFM characterization mentioned above. The dimensions of these two layers show much faster lateral than vertical growth, with a ratio of 60–70.

Once the InGaN growth flattens the dome-like surface, the optical quality of the InGaN layer is greatly enhanced as shown by the RT PL spectra in Figure 5a,b. The integrated PL intensity increases linearly with the growth time until 2500 s and then shows an abrupt increase for the 3500 s growth (Figure 5b). The integrated PL intensity of the sample grown for 3500 s is 80 times that of the sample grown for 250 s and 4 times that of the sample grown for 2500 s. The FWHM of the PL spectrum for the 3500 s growth is 140 meV, in contrast to 260 meV for the as-polished InGaN template. The volume of the as-polished InGaN template is much larger than that of the layer grown for 3500 s, as shown by the TEM image in Figure 4a. However, the as-polished InGaN template shows a much lower PL intensity. This is believed not to be due to the surface damage from the CMP, but rather due to C and O impurities incorporated during the InGaN pyramid growth on {1011} facets.

Figure 5c shows the time-resolved PL spectra measured at the peak energy for the samples grown for different times. The as-polished template is also included together with the setup response to the laser. For the samples with a growth time shorter than 3500 s, the spectra are dominated by a fast decay. For the sample grown for 1000 s, the intensity decays even faster than the as-polished InGaN template. This indicates one more mechanism contributing to the nonradiative recombination, which is most probably related to the bunched steps. With the continued growth, the fast decay becomes slower, consistent with the reduced number of the bunched steps and the increase in the PL intensity. For the growth from 2500 to 3500 s, the decay becomes much slower for both the fast and the slow components, explaining the abrupt increase in the PL intensity. This also indicates that the pits at the edges for the 2500 s growth, resulting from the notches, play the same role as the bunched steps on the carrier recombination.

With this method, we can also fabricate InGaN platelets with PL emissions at 2.63 eV, as shown in Figure S1 in the Supporting Information, corresponding to an indium content around 17%. In order to study the quality of the grown layer, we performed CL measurements on samples with the InGaN layers grown for 4 h (layer thickness: about 300 nm) with indium contents of about 9 and 18% (see Figures S2 and S3 in the Supporting Information for SEM and CL results). Because of the low growth rate of the inclined {1011} facets, the top c-plane shrinks with the thick layer growth. Even though the layer thickness is around 300 nm, the top surface is still smooth with both indium contents, indicating insignificant influence from the strain and the phase separation. For the platelets with the indium content of about 18%, dark regions were observed at two periphery sides in cross-sectional CL imaging, close to the interface between the polished InGaN template and the grown layer (Figure S3d–g). This originates from the local low quality of InGaN because of the fast growth through bunched steps as discussed above, rather than by dislocations because dark areas caused by dislocations are typically found to have a much larger extension. Otherwise, the grown InGaN layers show strong luminescence across the whole cleaved surface for both samples, indicating an absence of dislocations.

CONCLUSIONS

In summary, we present a method to fabricate arrays of high-quality submicrometer-sized InGaN platelets by InGaN growth on InGaN templates with similar indium contents. The InGaN platelets can be grown free from dislocation and with a smooth top c-plane surface decorated by single bilayer steps. InGaN platelets, with an indium content of up to 18%, are realized on a dome-like surface through the formation of bunched steps and complete termination of these, leading to an ultraflat top c-plane surface with the luminescence efficiency enhanced by a factor of about 80 (integrated PL intensity). This work shows a feasibility to grow high-quality InGaN material once the strain can controllably be kept low. Such predominantly relaxed InGaN platelets can be used as a platform to investigate highly efficient green and red nitride LEDs with improved lattice match between the platelet templates and the InGaN QWs, which are urgently needed for full-color micro-LED displays.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsami.0c00951.

Fabrication of InGaN platelets with an indium content of up to 17% and CL characterization of InGaN platelets with indium contents of 9 and 18%, showing a good uniformity of the indium content and an absence of dislocations

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Author Contributions
Z.B. designed and conducted the template preparation by CMP, MOVPE growth, and PL and SEM characterizations. T.L. joined the design of the MOVPE growth and discussion on the results. J.C. and R.T. conducted the AFM measurements. E.S. and F.L. prepared the TEM specimen and carried out the TEM characterization. R.W. helped with the TEM measurements. A.G. conducted the CL measurements. J.J. and B.M. joined the result discussion. L.S. supervised all experiments, discussion, and the write-up in this work. All authors contributed to the result discussion and the revision of this manuscript.

Notes
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