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Nanoselective area growth of defect-free thick indium-rich InGaN nanostructures on sacrificial ZnO templates.

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Abstract: Nanoselective area growth by metal organic vapor phase epitaxy of high-quality InGaN nanopyramids on GaN-coated ZnO/c-sapphire is reported. Nanopyramids grown on epitaxial low-temperature GaN-on-ZnO are uniform and appear to be single crystalline, as well as free of dislocations and V-pits. They are also indium-rich (with homogeneous 22% indium incorporation) and relatively thick (100 nm). These properties make them comparable to nanostructures grown on GaN and AlN/Si templates, in terms of crystallinity, quality, morphology, chemical composition and thickness. Moreover, the ability to selectively etch away the ZnO allows for the potential lift-off and transfer of the InGaN / GaN nanopyramids onto alternative substrates, e.g. cheaper and/or flexible. This technology offers an attractive alternative to NSAG on AlN/Si as a platform for the fabrication of high quality, thick and indium-rich InGaN monocrystals suitable for cheap, flexible and tunable light-emitting diodes.

Keywords: InGaN, nano selective area growth (NSAG), metal organic vapour phase epitaxy (MOVPE)

The fundamental band gap of GaN can be tuned to cover the whole visible spectrum by alloying with indium[1]. This capacity has already proven to be highly successful for the fabrication of light emitting diodes (LEDs)[2] and can potentially lead to the production of phosphor-free true white LEDs made of red, green and blue solid-state spontaneous emitters. However, the strong tendencies to compositional phase instability and relaxation of the layer due to lattice mismatches with conventional epitaxial substrates lead to indium clusters and large dislocation densities at the higher indium contents and film thicknesses necessary for absorption of the solar spectrum[3–7]. Hence the growth of high-quality InGaN layers with over 15% of In incorporation and having suitable thickness remains challenging (even on GaN templates)[8,9]. These issues are even more pronounced in the case of InGaN growth on silicon, as reported by several authors who recently grew InGaN layers (with up to 40% of indium content) on AlN-buffered Si (111) substrates by plasma-assisted molecular beam epitaxy (PA-MBE) and metal organic vapor phase epitaxy (MOVPE)[10–13]. Moreover these silicon-based processes may have additional disadvantages, like a longer processing time due to the back-etching of the Si substrate, and the resulting impossibility to re-use the wafer[10–13]. Thus it is desirable to develop novel approaches to improve the quality of thick and indium-rich InGaN epilayers. One potential solution is in the growth of InGaN nano-structures using a non-adsorbing masking material which could accommodate the elastic strain without the generation of dislocations[14]. Significant work has been reported on self-assembled InGaN nanostructures over the entire composition range using HVPE, but this process leads to a random distribution of island sizes[15]. Others have reported MBE growth of InGaN nanostructures on GaN-nanocolumn-buffered silicon substrates emitting from the ultraviolet (3.2 eV) to the infrared (0.78 eV) and achieved an internal quantum efficiency of 36% for green-emitting nanocolumns[16]. Using MOVPE, low indium content InGaN nanostructures have been selectively grown only on GaN templates by pulsing the precursors[17]. Recently, using continuous flow MOVPE on GaN templates and an improved patterning process, the growth of thick, uniform, dense, single phase, indium-rich nanorod arrays was demonstrated on both GaN/c-sapphire[18,19] and AlN/Si wafers[20]. ZnO substrates offer a number of advantages over c-sapphire, AlN/Si and wurtzite SiC wafers[21]: a lower lattice mismatch with GaN (1.8% vs. resp. 16% and 4%), a lattice match with $In_{.22}Ga_{.78}N$, and a more similar c-axis thermal expansion coefficient[22]. Moreover, the compressive strain due to the growth on the ZnO underlayer promotes stabilisation of an InGaN compositional phase with enhanced indium content[23,24]. This approach also

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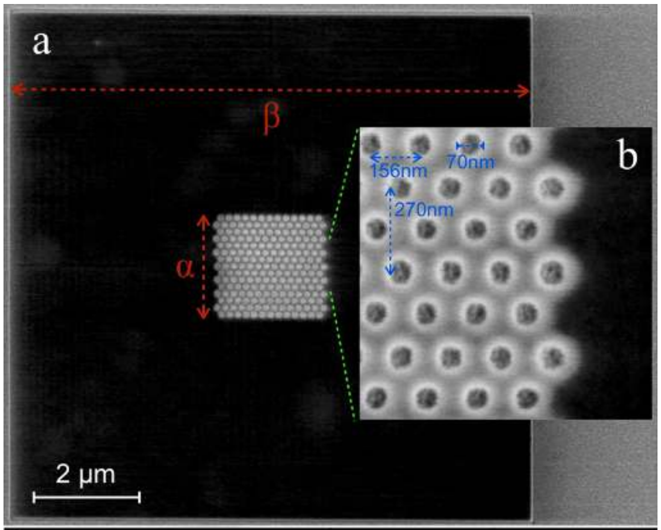

c Pattern	α (µm)	β (µm)	mask width (β - α)/2 (µm)
a1	2	10	4
a2	2	34	16
a3	2	66	32
a4	8	10	1

FIG. 1. Mask geometry. (a) Field-emission scanning electron microscope (SEM) image of a pattern of electron-beam-exposed HSQ (dark on image) on a GaN / ZnO / c-Al_2O_3 substrate. (b) SEM Zoom on the mask. (c) Definition of arrays (a1), (a2), (a3) and (a4) as a function of the variables α and β.

has the potential for transfer of nano InGaN-GaN structures onto alternative substrates (e.g. cheaper or flexible) through chemical liftoff via preferential etching of the ZnO underlayer[25].

In this work, we demonstrate NSAG as a potential route which mitigates the technological blocks resulting in high quality InGaN nanostructures on ZnO templates. ZnO template layers were grown on c-sapphire using pulsed laser deposition (PLD) from a sintered 5N ZnO target in a molecular oxygen ambient with a 248 nm KfF excimer laser[26]. Nanoselective area growth (NSAG) of InGaN was made by MOVPE through electron-beam-exposed hydrogen silsesquioxane (HSQ) masks which were fabricated using a process described in Refs[18,27]. Several mask formats were used, as shown in Fig.1.

We first tried to pattern the HSQ directly on the ZnO without GaN on top, and then 100 nanometers of InGaN were grown. However it turned out that for MOVPE of InGaN above 730°C ZnO starts decomposing[28]. Indeed at low growth temperature (around 730°C), due to adsorption and lower migration of growth species on the surface of the SiO_2, nucleation occurs on the exposed HSQ resulting in polycrystalline InGaN formation, leading to loss of selectivity, as seen in Fig.2. Furthermore both NH_3 (the standard precursor gas for atomic nitrogen N), and H_2 (the standard carrier gas) back-etch the

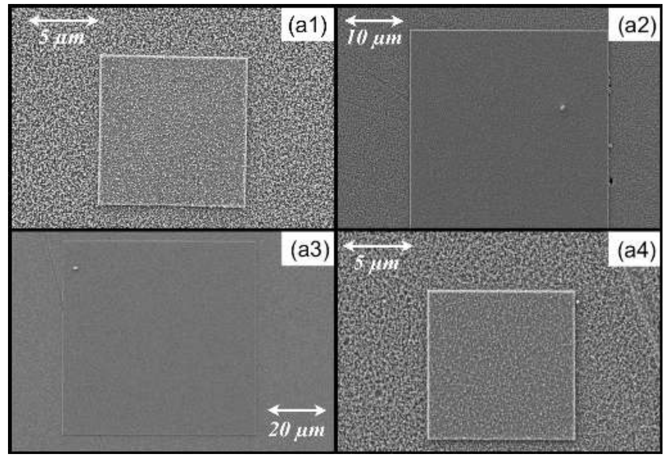

FIG. 2. SEM images of 100 nm of InGaN grown directly on patterned ZnO templates, showing motifs (a1) to (a4). As can be clearly observed, selectivity is lost, due to the relatively low temperature required for growth on the ZnO templates.

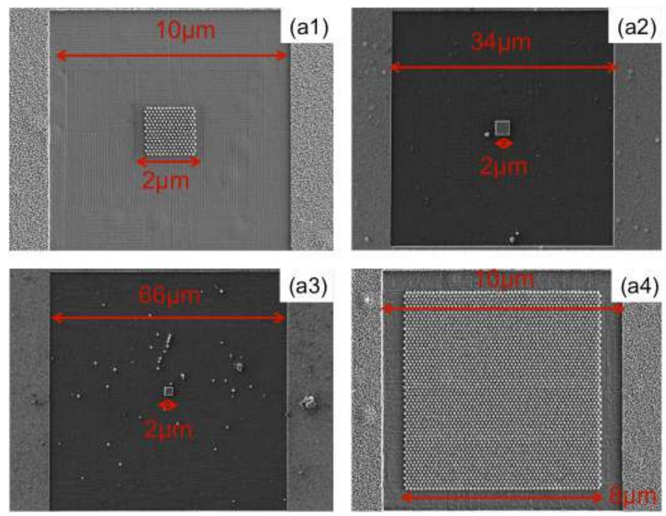

FIG. 3. SEM images of 100-nm-thick InGaN structures on a patterned GaN (100 nm) / ZnO template, showing arrays (a1) to (a4). The protective MOVPE-grown 100-nm GaN coating allows growth at higher temperature (800°C) without significant ZnO back-etching and thus restores selectivity.

surface of the ZnO at these temperatures, which can lead to delamination of the GaN layer[29–31].

In previous work, the authors addressed the back-etching of ZnO by growing high quality GaN by MOVPE under N_2 carrier gas (normally H_2) and replacing the ammonia with 1,1dimethylhydrazine (UDMH), cf Refs.[32,33]. The use of UDMH allows for a reduction of the GaN growth temperature from the conventional range of 1000-1100°C to a range of 550-800°C (henceforth referred to as low temperature, or LT).

Hence, in an attempt to restore selectivity, 100 nm of GaN were first grown epitaxially on the ZnO at LT and then an SiO_2 overlayer mask was patterned using e-beam lithography on HSQ, cf Fig.1. SEM analysis shows that using this method InGaN growth at 800°C became

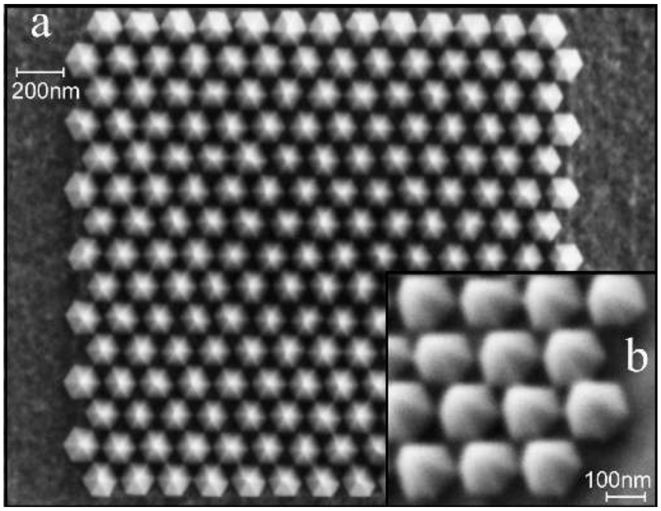

FIG. 4. SEM of pattern (a2), an array of 100-nanometer-thick $In_{.22}Ga_{.78}N$ defect-free wurtzite monocrystals. (a) Full array and (b) Zoom on the lower right corner at 37° tilt angle.

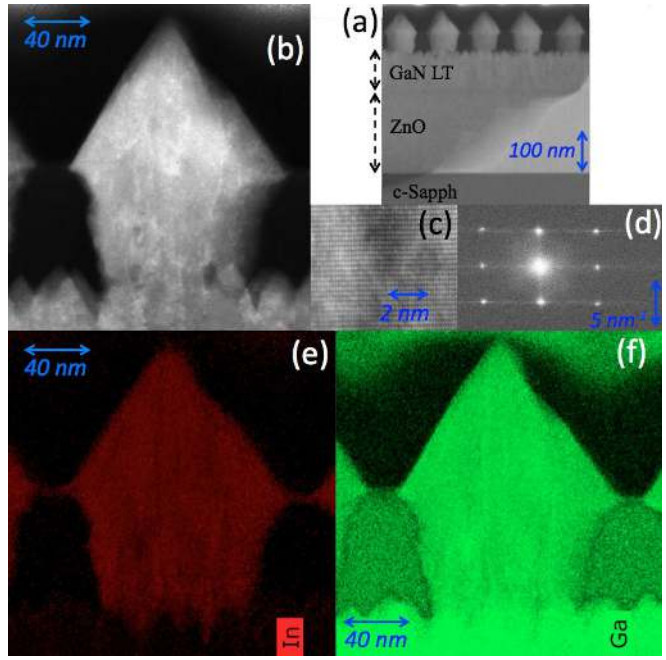

FIG. 5. STEM images of InGaN nanopyramids. (a) HAADF image of 5 consecutive defect-free monocrystalline InGaN nanopyramids grown by NSAG on top of 100 nm of low-temperature GaN, which was grown on 200nm-thick ZnO on c-sapphire. The FIB cut was realized along the $[1\bar{1}00]$ direction. (b) HAADF zoom on one InGaN nanopyramid, illustrating the single-crystal structure and absence of V-pits or threading dislocations which are common to all nanopyramids. (c) A further HAADF zoom of the center of the InGaN nanopyramid in Fig.5(b), revealing wurtzite structure. (d) FFT of Fig.5(c), confirming an hexagonal atomic arrangement in the InGaN. (e) EDX spectral map for the indium emission peak. (f) EDX image for the gallium emission peak.

perfectly selective on the $GaN/ZnO/Al_2O_3$ samples, as seen in Fig.3. Fig.4 shows that arrays of regularly-spaced InGaN nanopyramids have formed in the patterned areas. The nanopyramids exhibit aligned hexagonal faceting, and are homogenous in size, shape, and composition. The six smooth triangular semipolar $(1\bar{1}01)$ facets indicate epitaxial growth. Each nanopyramid appears to be free from intrinsic defects within the detection limits of the SEM. The SEM images on Fig.4 do not show any V-pit (V-pits are usually the result of propagation of threading dislocations[34,35]). Whereas the planar InGaN in the unpatterened area exhibits 3D growth with V-pits and other defects resulting in the rough morphological quality as vividly seen from the SEM image in Fig.3. As a remark we can notice evidence of micron-sized blisters in Figs.3(a1) and (a4) which may be due to the underlying ZnO starting to dissociate locally, but being blocked by the GaN. The main result here is the demonstration of a novel nano-patterning process that suits fragile ZnO templates, leading to the growth of InGaN nanopyramids on LT-GaN-covered ZnO templates with perfect selectivity.

Scanning transmission electron microscopy (STEM) images of the InGaN nanopyramids are displayed in Fig.5. The focused ion beam (FIB) lamellae were cut along the $[1\bar{1}00]$ direction to account for the six-fold symmetry of the InGaN nanopyramids and hence obtain a full description of their morphology. The high-angle annular dark field (HAADF) images Figs.5(a), (b) and (c) illustrate the monocrystallinity and the absence of threading dislocations or resulting V-defects[34,35] within the InGaN nanopyramids. The fast Fourier transform (FFT) shown in Fig.5(d) confirms their wurtzite atomic arrangement. The energy-dispersive X-ray (EDX) spectral maps shown in Figs.5(e) and (f) highlight the homogeneity of the nanopyramids in both indium and gallium compositions, indicating that there is a single compositional phase in the nanopyramids. Fig.5(a) also shows that the LT 100nm-thick GaN epitaxially-grown on ZnO has relatively small columnar grains, which would be expected to present many stacking faults. However the poor morphology of the LT GaN layer did not appear to affect the high quality of the InGaN nanopyramids, which were comparable with those already observed for GaN and InGaN grown on GaN-on-sapphire[18,19] and AlN-buffered silicon[20]. Indeed, at the nanoscale, the quality of InGaN on LT-GaN-covered ZnO templates is comparably simillar to that of GaN-on-sapphire or AlN-on-Si wafers thanks to our approach where the size of the opening favors formation of one crystal seed to grow regardless of the quality of substrate: as growth proceeds in the openings less than 100nm-wide diameter, the growth dynamics favor the formation of only one preferential crystal seed in the openings and all the growth in the nano-opening undergoes elastic strain relaxation after reaching critical thickness without generating any strain related degradations, due to the availability of free lateral surfaces; hence we typically have no dislocations, no cracks even if the buffer layer is polycrystalline, cf AlN-on-Si[20].

In addition, the optical properties of these nanostructures were studied using depth-resolved cathodolumines-

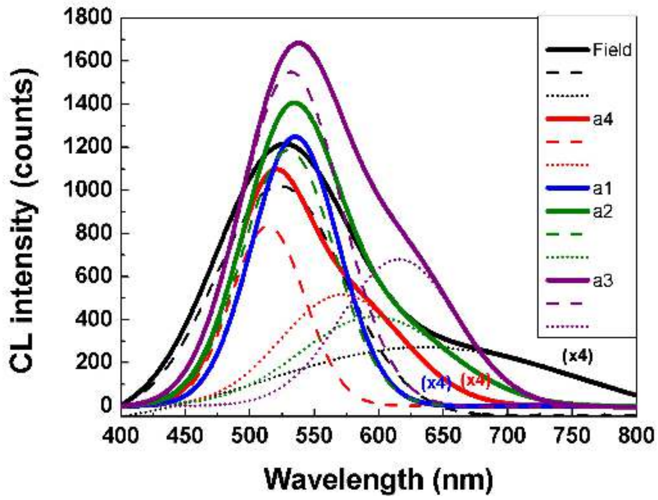

FIG. 6. Cathodoluminescence (CL) measurements at 77K on nanopyramids in patterns (a1) to (a4) and the field. All measurements come from one sample, meaning that all the patterned areas and the field experienced the same growth conditions.

cence spectroscopy (CL), see Fig.6. The spectra all show homogeneous main CL emission peaks in the green region of the visible spectrum, with centers between 535 nm (2.32 eV) and 519 nm (2.39 eV), respectively corresponding to relaxed high-quality $In_{.22}Ga_{.78}N$ and $In_{.20}Ga_{.78}N$ (calculated using Ref.[36]).There are red shifts in the emission peaks for arrays (a1), (a2) and (a3), confirming an increase in indium incorporation in these nanostructures compared to the unpatterned area. For masks (a2), (a3) and (a4), there is a distinct red-shifted shoulder which is very small, compared to the InGaN in the areas of conventional growth (a.k.a. the field, black curve on Fig.6). This shows that indium incorporation is significantly more homogeneous in the nanostructures than in the field. As a remark on the red-shifted shoulders we found in previous study[37], in the case of InGaN grown on AlN/Si substrate by NSAG, that depending on mask margin size and growth temperature, the relative and overall intensity of the two-peak emission can be controlled. The study suggests that the mask margin role in the color ratio is directly related to the delay of the transition between pseudomorphic and relaxed growth modes because of increased local TMIn concentration due to vapor phase diffusion and increased local free Indium atoms due to surface migration. The increased TMIn concentration has this effect even when it does not enhance growth rate or composition. Comparable studies by different groups have led to similar conclusions[38–40].

To conclude, NSAG of regular single-crystal InGaN nanopyramid arrays was achieved on GaN-coated ZnO epitaxial substrates by MOVPE. The relatively poor quality of the low-temperature GaN layer epitaxially grown on ZnO did not seem to impact the quality of the nanopyramids grown on top it by NSAG. Indeed they were thick (100 nm), indium-rich (up to 22%), single crystalline, dislocation-free, uniform, and thus comparable to high-quality InGaN nanostructures grown on GaN (see Refs.[18,19]) and AlN/Si (see Ref.[20]) templates. LT-GaN can also be n-doped with the ZnO templates with a high flow of silane, and optimized low temperature (800°C) lateral growth of p-doped (precursor Cp2Mg) GaN on the InGaN nanopyramid exposed semipolar r-plane facets, coupled with the selective etching of the ZnO, has the potential to transfer the optoelectronic PIN structure onto flexible and/or cheaper substrates.

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