Characterisation of 3D-GaN/InGaN nanostructured Light Emitting Diodes by Transmission Electron Microscopy

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Abstract. Transmission and scanning electron microscopy have been used to characterise GaN/InGaN 3D nanostructures grown on patterned GaN/sapphire substrates by MOVPE. It has been found that the growth of well ordered arrays of such nanostructures, containing multiple quantum wells on non-polar side-facets, can be achieved with a low density of defects. Growth changes and surface morphology play a major role in the nucleation of any defects present. The nanostructure morphology has been investigated and non-uniform growth on adjacent facets studied.

1. Introduction

The growth of 3D-GaN/InGaN nanostructures is of interest for the development of high efficiency Light Emitting Diodes (LEDs) for solid state lighting¹⁻³. Utilising a core-shell structure, as illustrated in Fig 1, should allow the active InGaN multiple quantum wells (MQW) to have not only a lower defect density, but also an increased area, compared to conventional (0001)-oriented thin films. Due to the non-polar orientation of the MQW, piezoelectric fields will also be reduced or eliminated, promising a further increase in light output and efficiency. In this paper, we examine patterned GaN/InGaN nanostructures of the type illustrated in Fig 1. To fully understand the structure, and any defects present, the nanostructures have been characterised using scanning and transmission electron microscopy (SEM and TEM).

2. Experimental

The GaN/InGaN nanostructures were grown on c-plane sapphire using continuous flow metal organic vapour phase epitaxy (MOVPE). A layer of n-type GaN was initially grown, followed by the deposition of a SiOₓ masking layer by plasma-enhanced chemical vapour deposition (PECVD). The mask was subsequently etched to produce an ordered array of circular holes with diameters from 250nm to 400nm, and pitches of 750nm to 2µm. Growth of the n-type doped GaN was then resumed under growth conditions, producing uniform arrays of high quality vertical-sided nanorods. The growth conditions were then changed to favour lateral growth, producing the MQW and p-type doped GaN growth. More details of the growth conditions can be found in ref. [4].

The nanostructures were initially studied using SEM, prior to the preparation of TEM samples. For TEM characterisation both cross-section and plan-view samples were prepared, allowing a better understanding of the structure and any defects present. To prepare the cross-section samples described...
here, the nanostructures were removed from the substrate, by sonication, and dispersed in an ethanol suspension. This was deposited onto holey carbon films and dried to create a dispersed film, where the nanorods can be studied free of contamination, Fig 2b, 4b.

Fig. 2. (a) SEM image showing nanostructures with partial overgrowth. (b) Sample prepared for cross-section analysis free of contamination.

To create plan-view samples, a novel preparation technique has been developed using dual-beam focused ion beam (FIB) thinning. Samples were prepared by attaching two pieces together with conductive epoxy resin. The FIB was then used to cut a section perpendicular through the nanostructure array, Fig 3. This technique allows the internal structure to be studied as a function of the height in the nanostructures. The prepared section was lifted from the remaining material and deposited on a conventional holey carbon film for imaging, Fig. 3c.

The samples were studied using a Philips EM430 TEM operating at 250kV and a JEOL 2010 operating at 200kV.

Fig. 3. (a) Schematic illustrating plan-view preparation. (b) SEM image of the sample taken during preparation. (c) The sample viewed in the TEM.

3. Results
Initial studies of the samples using SEM show the masking layer creates a regular array of nanostructures, with similar aspect ratios, which can be controlled during the growth process. It has been found that the switch to growing the MQW plays an important role in the overall crystalline quality of any subsequent growth of p-GaN. Fig 2a shows how the growth of p-GaN has only occurred at the very top of the nanostructure. In this sample the p-GaN layer has been found to contain a large density of both I₁ and I₂ type basal plane stacking faults (BSFs), Fig 5a, which are bounded by partial dislocations. The faults are dislocation half-loops seen both end on and in projection in Fig 5a. Further
studies of this layer show that the MQWs have grown with an island like morphology, Fig 5b. The rough island-like MQW can be seen to propagate under the p-GaN overgrowth, suggesting that the morphology is not a post-growth artefact. Examination of the n-GaN part of the nanostructure has shown that the initial growth is free of defects, both BSFs and threading dislocations. In contrast, studies of nanostructures where the MQW growth has formed smooth layers show a much reduced defect density. The few defects which have been observed are similar to those seen in the other samples. However it appears that the half-loop, bounding the defect, nucleates below the MQWs, Fig 5c. This suggests that the defects nucleate at the transition between the axial n-GaN growth, and the laterally grown material.

Plan-view analysis of the samples allows the radial sequence of growth to be examined more closely. From these samples it has been found that the growth rates of different facets are non-uniform, leading to not only non-uniform profiles across the rods, but also to non-uniform spacings within the
MQWs for different facets. This is clearly seen in Fig 6a, where the spacings of the QWs on one facet are roughly double that of the adjacent facet (see inset). Further studies of these samples allowed the quantum well thicknesses to be determined. Figure 6b shows three QWs which are buried within the bulk material. From the line plot we can see that the QWs have a thickness of ~2nm and a separation of ~4nm. The change in contrast across the MQWs indicates the presence of InGaN.

![Image](200 nm 50 nm 10 nm)

**Fig. 6.** (a) Plan view images showing the presence of QWs, along with the non-uniform growth of p-GaN on differing facets (inset). Closer examination of other MQWs shows that the InGaN layers have a thickness of ~2nm. A line plot (inset), taken where indicated.

### 4. Discussion

The growth of highly ordered GaN/InGaN 3D nanostructures on sapphire substrates by MOVPE has been achieved. The nanostructures have been found to consist of a core-shell structure with InGaN MQWs buried between the n-GaN core and the p-GaN shell. It has been found by TEM studies that the initial n-GaN core is free of both threading defects and basal plane stacking faults. The transition to lateral growth in preserving this crystalline quality was found to be important; growth leading to rough MQWs has been associated with the nucleation of BSFs in the subsequent p-GaN layer, whilst growth leading to smooth MQWs correlated with significantly fewer defects. Plan-view studies of the structures have revealed the non-uniform growth of p-GaN on differing facets, along with changes in QW thickness and spacing.

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