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Fabrication of InGaN/GaN Multiple Quantum Wells Nanopillars by Focused Ion Beam Milling

Shang-En Wu[1], Tao-Hung Hsueh[2*], Chuan-Pu Liu[1,2], Jinn-Kong Sheu[3], Wei-Chih Lai[3], Shoou-Jinn Chang[2,4]

[1]Department of Materials Science & Engineering
National Cheng Kung University, Tainan 70101, Taiwan
[2*]Center for Micro/Nano Science and Technology
National Cheng Kung University, Tainan 70101, Taiwan
Phone: +886-6-2080013 E-mail: taohung@mail.mina.naku.edu.tw
[3]Institute of Electro-Optical Science and Engineering
National Cheng Kung University, Tainan 70101, Taiwan
[4]Institute of Microelectronics & Department of Electrical Engineering
National Cheng Kung University, Tainan 70101, Taiwan

1. Introduction

Focused ion beam milling (FIB) has been in use in many applications since the liquid metal ion source was developed in 1975. FIBs have advantages of large depth of focus, high resolution, and, the most important, the direct-writing capability. The recent applications have expanded into areas such as maskless lithography [1-3], deposition [4,5], doping [6,7], and even quantum dot growth [8,9]. Most studies conducted on III-nitride top-down fabrication were only devoted to some etching routes like inductively coupled plasma (ICP) etching and reactive ion etching (RIE) [12-14]. In this work, fabrication of InGaN/GaN multiple quantum wells (MQWs) nano-pillars are demonstrated by FIB using a modified beam-shape tuning [13]. The anomalous phenomenon of this approach leaving behind high-aspect-ratio InGaN/GaN MQWs nanopillars will also be discussed. High resolution cross-sectional transmission electron microscopy (XTEM) revealed the retained single-crystalline nature of these pillars while the emission image revealed the the InGaN/GaN MQWs emission.

2. Experiments

The samples used in this study have a light emitting diode structure grown by metal-organic chemical-vapor deposition which includes a 30-nm-thick GaN nucleation layer, a 4-μm-thick Si-doped GaN layer, 14-pair $In_xGa_{1-x}N$/GaN MQWs, a 560-nm-thick Mg-doped GaN. The FIB instrument we used here is SMI-3050SE dual beam system (SII Nanotechnology Inc). 30 keV Ga ions with a beam current of 300 pA have been applied to completely remove the III-V material around the pillars. Sharp-tip array and high-aspect-ratio InGaN/GaN MQWs pillars are achieved by stigmating and defocusing the ion beam simultaneously as reported in our previous work on a Si substrate [13]. With the ion doses of 2.48×1018 cm^{-2}, 99 arrayed tips were produced within 7 minutes. The pattern-defined areas did not suffer any ion scanning before the milling step was started. The XTEM investigations were performed by JOEL-2100F high resolution TEM system, operated at 200 keV.

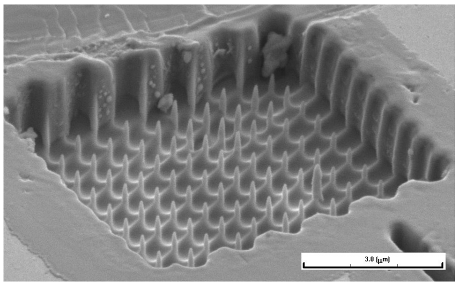

Fig. 1 SEM tilted-view image of the s-milled sample surface.

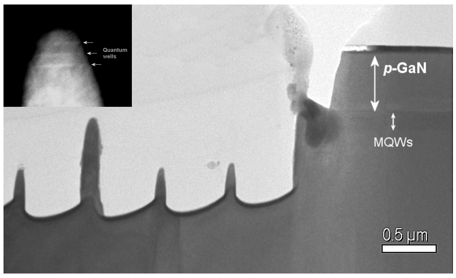

Fig. 2 XTEM image of the FIB-patterned pillars.

3. Results and Discussion

Figure 1 shows a scanning electron microscope bird-view image for the FIB-patterned pillars which typically have diameters of around 100~150 nm and heights of around 600 ~ 1000 nm. It has been shown that, volume swelling in GaN can be introduced by energetic ion implantation process [7]. The evidence of swelling was found in a series of SEM monitoring the pillar formation taken every 60 seconds during the ion beam milling process. The ratio of tips became pillars is estimated to be around 38/99 in figure 2. The insert in figure 2 is the corresponding high-angle annular dark field scanning-TEM image of a pillar tip, proving the existence of QWs MQWs structures embedded in the nanopillars. The single-crystal nature inside the taller pillar was demonstrated by electron diffraction pattern with the beam along the [11-13] zone

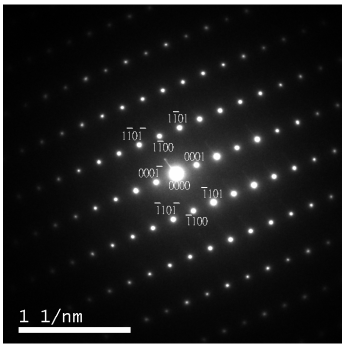

Fig. 3 Electron diffraction pattern confirming the single-crystal nature of one single pillar.

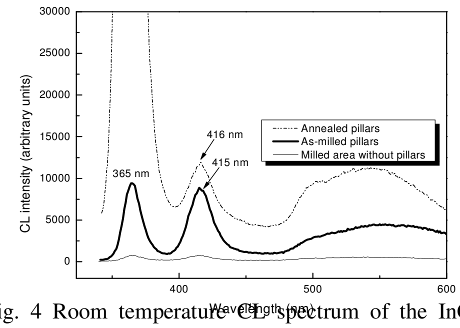

Fig. 4 Room temperature CL spectrum of the InGaN MQWs nanopillars.

axis, as shown in figure 3, conforming that the epitaxial relationship still remains.

Emission spectra of InGaN/GaN MQWs embedded in the pillars were observed around 2.98 eV by cathodoluminescence measurements in room temperature, as shown in figure 4. Two sharp and discrete emission peaks of near 365 nm and 415 nm were observed, originated from the exposed n-type GaN and InGaN/GaN MQWs embedded in nanopaillars, respectively. The emission peak of the InGaN/GaN MQWs embedded in nanopaillars showed a blue shift of about 35 meV (5nm) compared with that of the as-grown wafer. The blue-shift is attributed mainly to the strain relief in the well, since the quantum confinement effect is quite trivial (<< 10meV) for this case with quantum disc diameter of about 65 nm [14]. Moreover, rapid thermal annealing (RTA) at 800℃ for 1 min under nitrogen ambient was carried out. The n-GaN related emission intensity was increased up to 40 times, which may be due to the improvement on the crystal quality by the RTA, which are shown in figure 4. MQWs-related emission is almost unchanged and did not profit from the RTA treatment evidently in emission intensity. In addition, the peak position was blue-shifted to 362 nm, which reasonably can be attributed to the doping effect after RTA. Nevertheless, MQWs-related emission position is almost unchanged (416 nm) and did not profit from the RTA treatment evidently in peak intensity.

4. Conclusions

FIB direct milling of InGaN/GaN MQWs samples are performed with a beam-shape tuning process deliberately, and site-controlled high-aspect-ratio nano-pillars were anomalously produced, without any lithographies. The swelling of p-GaN tip induced by the ion beam live-irradiation served as low-milling-rate sites－the sites on which the resultant nanopillars form. The bump (or blister) formation mechanism is complicated but indeed plays an important role in heightening the nano-pillar. The retained QW discs inside the pillar emitted a sharp and discrete peak at 415 nm, showing a blue shift of ~35 meV with respect to the bulk sample.

Our results imply an alternative method for maskless and site-controlled nano-rod writing, and pave out another way for FIB prototyping III-nitride optical device.

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