Fabrication of GaN nanorods by focused ion beam

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A B S T R A C T

Ordered arrays of III-Nitride nanocolumns are excellent candidates for the fabrication of nano-optoelectronic devices. Different technologies such as e-beam lithography or colloidal lithography, have been used to obtain ordered arrays. All these technologies have in common several processing steps that can affect the crystalline growth of the nanocolumns. In this work, we present a single lithographic step that permits to grow ordered GaN nanocolumns with different geometries. The patterning is based in the use of a focused ion beam with different doses. With this method has been possible to create GaN nanopillars and nanocylinders.

1. Introduction

The III-Nitride nanocolumns (III-N NCs) are the subject of intense research since the past decade because of their unique properties and potential electronic and optoelectronic applications [1–5]. NCs are usually grown on Si(1 1 1), Si(1 0 0), SiC, and sapphire substrates by a self-assembly process using plasma-assisted molecular beam epitaxy (PA-MBE) [6,7]. Unlike continuous layers, NCs accommodate the lattice-mismatch with the substrate through a network of misfit dislocations localized at the hetero-interface. Therefore, they grow fully relaxed and free of extended defects such as basal plane stacking faults or threading dislocations. This fact makes III-N NCs excellent candidates to develop arrays of highly efficient nanolight-emitters in the infrared–visible-ultraviolet range.

The efficiency of a nanolight emitter will be increased if the NCs are aligned in a periodic pattern. Several research groups have tried different methods for the patterning [8–14]. The most used one has been e-beam lithography. In this case, periodic structures can be fabricated, but most of the times resist residues remains along the sample and in that case a plasma treatment is needed. This problem affect in the posterior growth of the NCs in the MBE system. Another approach has been the use of colloidal lithography with nanospheres. This technique requires less fabrication steps than the e-beam lithography and it is almost free from residues over the substrate. But the disadvantage of this technique is that is quite difficult to have a regular pattern along large areas.

In our approach, we decided to use a focused ion beam system (ionLine from Raith, Germany) in order to create the nanostructures. This technique will pattern our substrate in only one step and with high precision in the periodicity due to the use of laser interferometric stage. Several studies with different doses have been carried out in order to obtain the optimum conditions for the nanorods growth. A plasma assisted molecular beam epitaxy system allows us to grow the GaN nanorods. Finally, by scanning electron microscopy analysis (SEM), we have observed that the geometries of the nanopillars depend on the dose used.

2. Experimental and results

The substrates used in this work were 2 in.-wafers of GaN on sapphire (Lumilog, France). Each wafer had a 4 μm GaN (0 0 0 1) layer grown on sapphire by MOVPE. A cleaning procedure of the surface was carried out during 30 min using n-methyl-pyrrolidone (NMP) at 90 °C. The wafer was later rinsed in iso-propanol (IPA) and thoroughly cleaned with de-ionized water (DI water). A thin layer of 10 nm of Ti was deposited on top by e-beam evaporation in order to act as a mask material (Fig. 1). The GaN template was pre-heated before Ti deposition in order to desorb water molecules from the surface.

The metal layer was patterning with nano-holes by a focused ion beam system in a one-step process. The liquid–metal ion source used was Ga^+ at 30 keV (ionLine Raith GmbH, Germany). The process conditions were optimized to obtain ordered arrays of nanoholes with 100 nm diameter and 250 nm pitch (30 pA, ion dose 10^{17} cm^{-2}). A matrix of 64 elements was fabricated covering an area of 400 μm^2 in writing fields of 50 μm. We increased the dose in every writing field with a factor 0.1. No extra cleaning steps

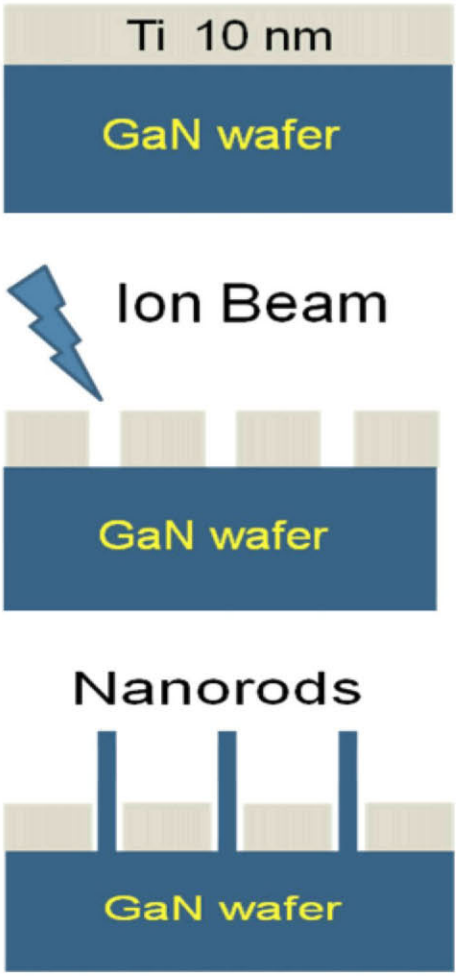

Fig. 1. Fabrication scheme of the GaN nanorods. A Titanium thin metal layer is deposited along the wafer with an e-beam evaporator. In a second step, a metal layer is patterned with an ion beam equipment in order to create nano-holes arrays. Finally, the sample in inserted in a MBE system and the nanorods grow inside the patterned areas.

were applied after the ion etching. The depths of the nanoholes were characterized by atomic force microscopy (Nanoscope III Multimode AFM, Veeco Instr., USA). AFM analysis revealed an etching depth between 10 and 15 nm (Fig. 2).

After this one step process, the sample was inserted inside a PA-MBE (Riber Compact 21) in order to grow the NCs. GaN NCs were grown on the hole-patterned mask using radio frequency (RF). In order to prevent the degradation of the Ti mask a nitridation step is required due to the high temperatures used in GaN NCs growth (860–900 °C). We followed the method proposed by Sekiguchi et al. [8,9] that consists in two nitridation steps: 10 min at 460 °C followed by 3 min at 880 °C. The plasma power was set to 580 W during nitridation and the nitrogen flux to 1.2 sccm. These conditions correspond to an equivalent stoichiometric GaN growth rate higher than 30 nm/min. In order to grow the NCs, the plasma power and the nitrogen flux have to be reduced to 150 W and 0.3 sccm, respectively. The GaN flux is maintained at a corresponding GaN growth rate of 16 nm/min. With these conditions, we obtained an ordered growth of GaN NCs inside the nano-holes, as widely illustrated in a previous publication [11].

After the growth process, the sample was inserted in a scanning electron microscopy (CABL-9500C SEM, Crestec, Japan) for inspection. From the SEM analysis one can observe that the NCs nucleate and grow selectively inside the nanoholes and not on the surface of the mask.

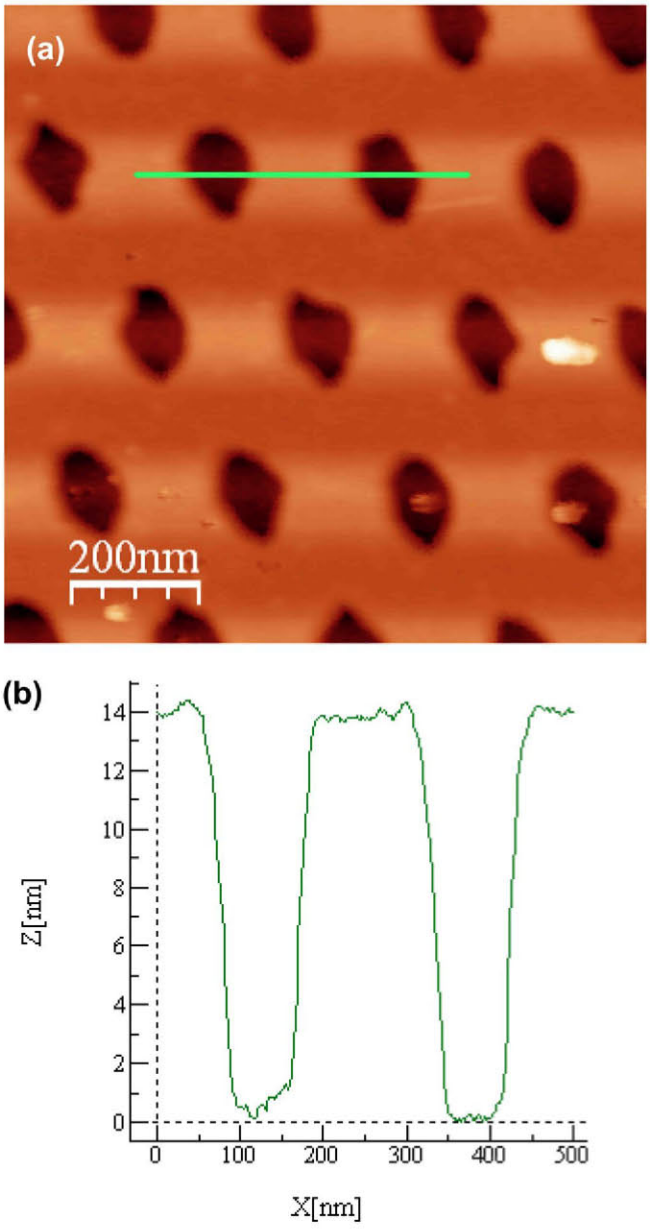

Fig. 2. (a) AFM image of the nano-patterned holes on the Ti metal layer. (b) Profile analysis of the nano-holes selected in the AFM image. The ion beam removes all the Ti inside the nanoholes.

Using an ion dose of 10^{17} cm^{-2}, (Fig. 3a) we obtained a single GaN NCs in every hole. In this case, there is a great level of selectivity but one can observe a few small NCs in the mask. This behavior will be expected also inside the holes patterning with bigger doses (above $6 \cdot 10^{17}$ cm^{-2}), but as one can observe in Fig. 3b the NCs are a vertical cylinder with a hole inside. This effect can be observed more clear when the samples are tilted 45° (Fig. 4). A possible explanation for these results is that in the patterning process at an ion dose of $6 \cdot 10^{17}$ cm^{-2} a small re-deposition of Ti (1 nm) appears at the edges of the holes. In these cases, the NCs nucleate just all around the hole leaving the core empty. In SEM pictures after growth revealed a single tubular column with no other NCs around.

Finally, in order to study the optical properties of the nanocolumns, we perform a photoluminescence analysis (PL). The PL was excited in backscattering configuration by using a He–Cd laser (λ = 325 nm). The PL signal was collected and dispersed through

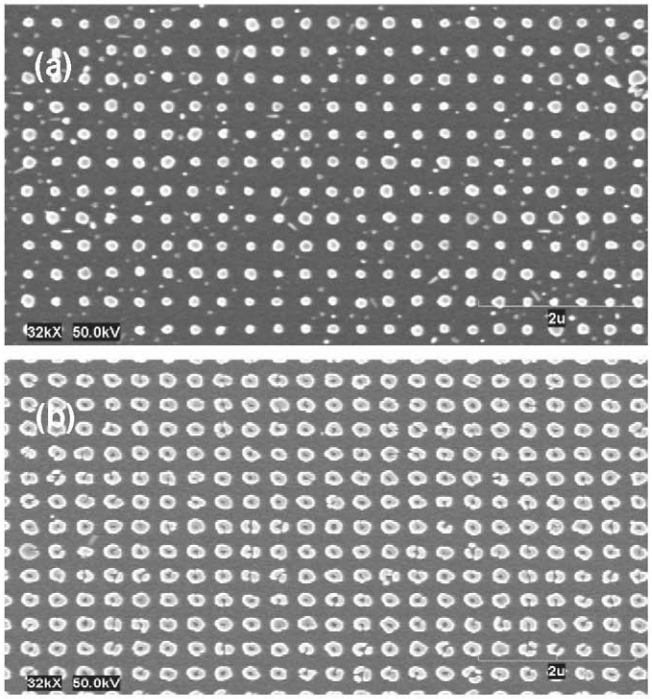

Fig. 3. SEM images of the selective area growth of GaN NCs by PA-MBE at different doses. (a) Single GaN nanocolumns grow inside every nano-hole at an ion dose of 10^{17} cm^{-2}. (b) Increasing the ion dose to $6 \cdot 10^{17}$ cm^{-2}, appear nanocylinders of GaN.

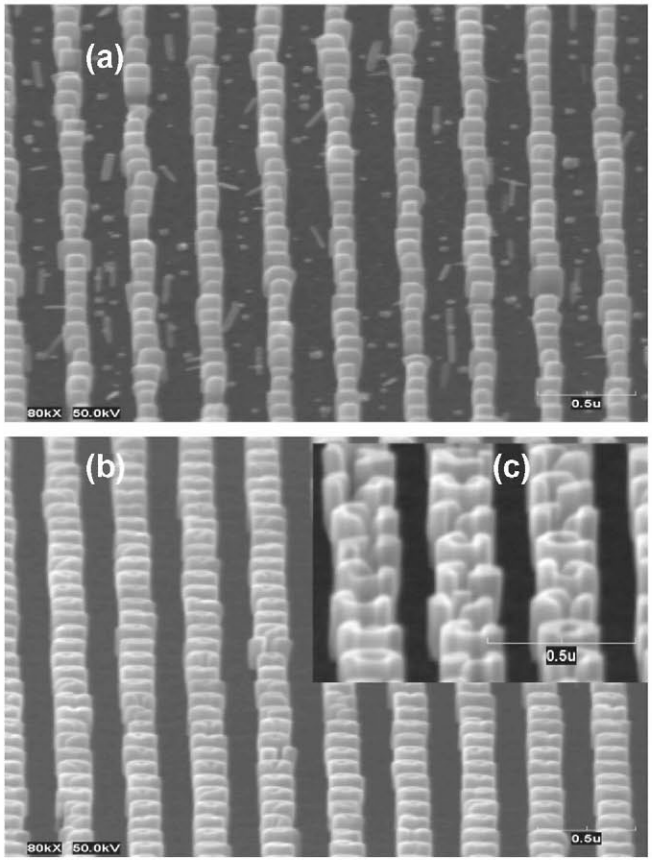

Fig. 4. SEM images tilted 45°. (a) Image of an ordered array of single GaN NCs from Fig. 3a. (b) Image of an ordered array of GaN nanocylinders from Fig. 3b. (c) Closer look to the GaN nanocylinders.

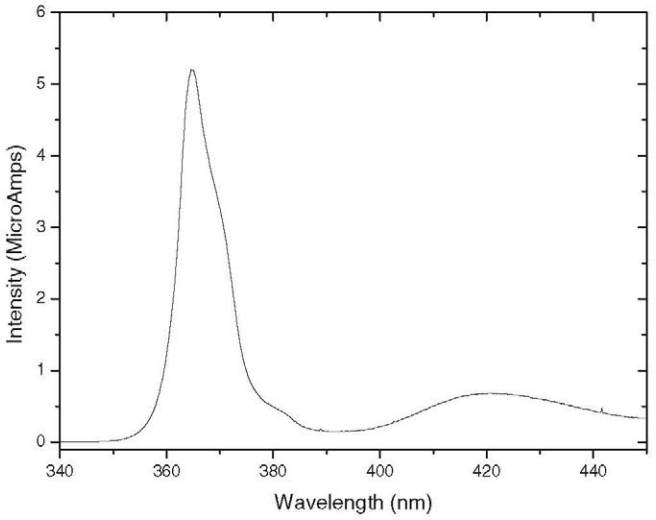

Fig. 5. PL spectra of the GaN nanocolumns from Fig. 3a. The peak emission is around 365 nm.

a spectrometer with grating optimized for ultraviolet–visible spectrum, and then detected by a photomultiplier tube. In Fig. 5, one can observe the PL spectra obtained from the GaN nanopillars showed in Fig. 4a at room temperature. In general, the photoluminescence on GaN nanowires depends on the growth direction of the GaN. The value of the peak at its maximum that we obtained is close to the one expected and has a similar value with respect to the results obtained by other research laboratories [15].

3. Conclusions

We propose a single step processing for the fabrication of GaN NCs. This technology is very efficient because there is no need of a resist layer and the system can pattern large areas. A focused ion beam system is used to pattern nano-holes and a PA-MBE system makes a selective nucleation and growth of GaN NCs inside the holes. The ion dose can control the geometry of the NCs, from single ones to circular ones. Finally with this method one can achieve selective growth and high crystal quality ordered GaN NCs.

Acknowledgements

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