Wet chemical etching of GaN or InGaN nanowires on Si substrate for micro and nano-devices fabrication

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Abstract. In this work, it is experimentally shown that the etching of GaN or InGaN NWs in the KOH solution allows managing the morphology and optical properties of the nanowires array. Thinning rate of GaN nanowires is 5 times slower than the rate for InGaN nanowires along semi-polar and non-polar direction for nanowires with Ga-polar crystal structure. The diameter of the InGaN NWs decreases from 200-100 to 30-40 nm. After etching process, the intensity photoluminescence (PL) of nanowires and uniformity PL signal at all sample area increase.

1. Introduction

In the last few years, nanowires (NWs) based on III-N (in particular, GaN and InGaN) materials are widely used in nanophotonics and optoelectronics to create high-efficient light-emitting diodes (LEDs), solar cells and etc. Firstly, this is due, for example, to the fact that GaN LEDs achieve high efficiency (more 70%) in comparison with more common analogues (organic light emitting diodes, etc.) [1]. Transfer planar LED technologies on 1-D nanostructures such as nanowires, nanorods can make it possible to increase the efficiency of light extraction from the structure, occur precision control of LED structure parameters, create flexible NWs-based devices etc. [2]. GaN NWs can also be used to manufacture single photon emitters (SPEs) [3]. Secondly, InGaN has a direct band gap in the range from 0.7 to 3.43 eV depending on the In composition, that’s why it can emit light across the visible spectrum. Thirdly, nanowires can be grown practically defect-free on substrates whose lattice constants differ significantly from the NW lattice constants [4]. However, the synthesis NWs with lateral sizes of nanometers requires special preparation of the substrates, including electron lithography method. There are also methods for diameter thinning synthesized NWs arrays. For example, thermal decomposition of NWs [5] after growth process which can impair the optoelectronic properties of NWs. At the same time, wet chemical etching is a promising and simply method due to which it is possible to control the morphology [6] and, as a consequence, the electrical and optical properties of NWs. Specifically, wet chemical etching of III-N NWs by alkalis solution should target the non-polar and semi-polar planes (figure 1). Drawing an analogy with KOH etching of layers of III-
N materials [7] in the case of Ga-polar crystallographic structure of NWs etching process along the [0001] direction will be slow. On the contrary, in the case of an N-polar structure will be show a decrease in the NW length. These features make it possible to control the final size of NWs [8] and indelicate polarity of crystal structure of NWs what important for create devices. At the KOH etching process, solution etches N-polar, semi-polar and non-polar planes, while the Ga-polar plane is untouched [9]. The work presents results of the wet chemical etching in the KOH solution of InGaN and GaN nanowires.

**Figure 1.** Scheme of polar, semi-polar and non-polar planes of the GaN-nitride crystal structure.

### 2. Experiment and results

The NWs were grown by molecular-beam epitaxy (Riber Compact 12 MBE system) on Si (111) substrates. The surface morphology of the samples was studied using Supra 25 (Carl Zeiss, Germany) scanning electron microscope (SEM). Optical properties of the NWs arrays were examine by photoluminescence spectroscopy (Accent RPM Sigma equipment). Wet chemical etching of III-N materials is carried out in the solution KOH:H$_2$O (1:5) at a temperature of 75°C with a variation of etching time [7].

#### 2.1. Etching of GaN nanowires

The grown GaN NWs are hexagonal in cross-section, extending to the top from 100-150 to 200-400 nm (figure 2a) with an average length of about 2 µm. Figure 2b shows of sample morphology after etching within 5 min. The nanowires diameter decreases by 50-70 nm along its entire length. The SEM images and schematic illustrations of NWs shape demonstrate etching along non-polar or semi-polar directions. Note that the NWs height doesn’t change after etching.

**Figure 2(a, b).** Isometric view SEM images of GaN NWs and schematic illustration of NWs shape: (a) initial sample; (b) sample after 5 min. etching. The inset shows a top of single NW. The scale bars correspond to 200 nm.
2.2. Etching of InGaN nanowires

Morphology studies of InGaN NWs initial samples show (figure 3a) a pencil-like shape with an 80-100 nm truncated top. SEM studies show that at the first stage of etching (60 sec.) there is a decrease in the diameter of the NWs along a direction perpendicular to the NWs (figure 3b). The diameter of the NWs decreases from 100 to 30-40 nm and becomes approximately the same at the entire length of the NWs. With further etching (60-180 sec.), the NWs are etched at the tops, which provides to a decrease in the overall height of the structure and separation of the NWs from the substrate. It achieves a greater homogeneity of the photoluminescence map. Note that when etching the structures increases the intensity of photoluminescence signal. At the etching process of samples removal of existing surface defects and heterogeneity of the array NWs, oxide layer and defects on surface single NWs. This mechanism of etching will allow to formation an uniform surface morphology of the samples and homogeneity of the photoluminescence map samples. Potentially, changing the viscosity of the etching solution (for example, add a glycerol at the etching solution) allows precision controlling the rate of etching with high accuracy.

![Figure 3(a, b). Plane view SEM images of InGaN NWs (a) initial sample; (b) sample after 1 min. etching. The inset shows a top of single NW. The scale bars correspond to 100 nm.](image)

3. Conclusions

The etching of GaN and InGaN nanowires occurs along the semi-polar and no-polar crystallographic directions [10] perpendicular to the growth direction of the NWs. Since the height of the NWs has not changed during the etching process, it is possible that the synthesized GaN and InGaN NWs have a Ga-polar crystallographic structure [7]. The rate of lateral etching of GaN NWs is 10 nm per min. This is 5 times slower than the rate of etching of InGaN NWs (GaN more chemical-resistant material).

As a result, it is experimentally shown that the etching of GaN and InGaN NWs in the KOH solution allows controlling the morphology and optical properties of the NWs array, what promising for different application.

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