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Top–down fabrication of horizontally-aligned gallium nitride nanowire arrays for sensor development☆

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ABSTRACT

This paper demonstrates a high-throughput fabrication method of gallium nitride (GaN) nanowire (NW) and sub-micron wire (SMW) arrays using a combination of projection lithography, plasma etching, and post-plasma wet etching techniques. Photoluminescence (PL), field emission scanning electron microscopy (FESEM), and I–V measurements were used to characterize the GaN NW/SMW devices. These NWs/SMWs can be used to create highly-sensitive and selective conductometric chemical/bio-sensors.

The length and width of the wires can be precisely customized. The length of the NW/SMW varied from 5 μm to 5 mm and the width ranges from 100 nm to 500 nm. Such comprehensive control in the geometry of a wire is difficult to achieve with other fabrication methods. The post-plasma KOH wet etching greatly reduces the surface roughness of the GaN NW/SMW as well as the performance of devices. Complementary metal-oxide-semiconductor (CMOS) and micro-electro-mechanical system (MEMS) devices can be incorporated with GaN NW/SMW arrays on a single chip using this top–down fabrication method.

1. Introduction

Gallium nitride (GaN) is a versatile semiconductor used in optical devices (light-emitting diodes, laser diodes, UV sensors) and in power electronics (power switches, RF devices, and high power transistors) [1]. Due to its direct band gap and chemical and thermal stability, GaN nanowires (NWs) are gaining significant importance as chemical sensor elements [2–4].

To date, most of GaN NWs devices are fabricated using chemical vapor deposition growth with a follow-up detachment from the substrate and individual on-chip alignment. Due to the variation in morphology, dimensions, doping, and crystal quality, it is hard to control the quality of NWs grown via the bottom–up methods [5]. Besides, variations from a multi-step pick-and-place fabrication process result in a low yield of functioning devices. Thus, such a process has low large-area integration capabilities, rendering mass-manufacturing challenging. An alternative approach to overcome these drawbacks is the top–down fabrication, in which NWs are patterned on a uniform thin-film using standard lithography and etching. Vertically-aligned semiconductor NWs (nanorods, nanopillars or nanocolumns) have been demonstrated by such top–down methods [6–10]. The heights of these nanostructures are limited by the thickness of the starting thin-film material, and they often exhibit tapering as a result of etching. On the contrary, horizontally-aligned NWs can be fabricated without any length limitations. For photodetectors and photovoltaic devices, vertically-aligned nanowires represent a high efficiency platform, whereas for chemical sensors horizontally-aligned nanowires are the optimal choice [11–13]. Due to its inherent inertness to most wet chemical etches, GaN nanostructures are commonly produced using dry etch techniques [8]. However, high-aspect ratio GaN structures achieved by plasma etching are often associated with extensive side wall damage, result in lower performance of the device [14]. Therefore, producing damage-free nitride structures with a precisely-defined geometry over large area remains a challenge.

By combining deep-UV projection lithography and inductively-coupled plasma (ICP) etching, this paper demonstrates horizontally-aligned NWs etched from a GaN thin film grown on sapphire. Since the rough and tapered NW sidewalls due to dry etching create leakage current and limit NW performance [14], a post ICP potassium hydroxide (KOH) wet etching procedure was developed to smooth the NW walls.

☆ Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

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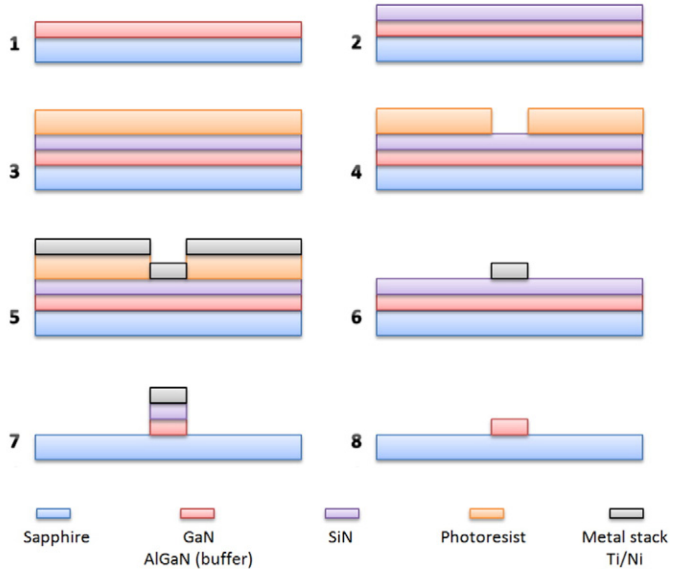


Fig. 1. Schematic representation of the nanowire fabrication process flow. (1) RCA cleaning of GaN on sapphire wafer, (2) SiN etch-mask deposited using PECVD, (3) spin-coating of photo-resist stack of LOR3A and Ultra-I, (4) lithography and development, (5) metal deposition by e-beam evaporator, (6) lift-off in 1165 photo-resist remover, (7) ICP etching to transfer the pattern on GaN, (8) HF etching, RIE etching to remove metal and SiN etch masks, followed by subsequent KOH wet etch treatment.

The KOH etching also allows us to control the final shape and dimensions of the NWs. The PL and current–voltage (I–V) results verify the improved performance resulting from the KOH treatment.

The top–down approach described here can potentially enable GaN NW and SMW to be integrated with LED, micro-pumps, and micro heaters for system-on-chip development [15–20]. Different types of sensors such as chemical, gas and bio sensors can be fabricated using the top–down method reported here.

2. Fabrication

Two different thicknesses of commercial intrinsic GaN epitaxial layers (NTT Advanced Technology) grown on c-plane sapphire were used in this study: 3 µm thick GaN films were used for characterization of the etch rate of ICP dry etching, optimization of wet etching and measurements of PL spectra; 1 µm thick GaN films were used in the optimization of the wet etching process as well as in I–V characterization. AlGaN is a buffer layer widely used to compensate lattice mismatch and reduce stress in the growing film, and thereby improves the crystalline quality of GaN on sapphire. In our experiment, a 500 nm AlGaN layer was used between the GaN and sapphire substrate. A silicon nitride (SiN) passivation layer with thickness of 50 nm was deposited by plasma enhanced chemical vapor deposition (PECVD). This layer prevents metal penetration into the bulk GaN. A bi-layer stack of photoresist (MicroChem LOR3A and Ultra-i) was spun onto the wafer for photolithography patterning. The 570 mJ/cm^2 stepper lithography dose was optimized for the fabrication process.

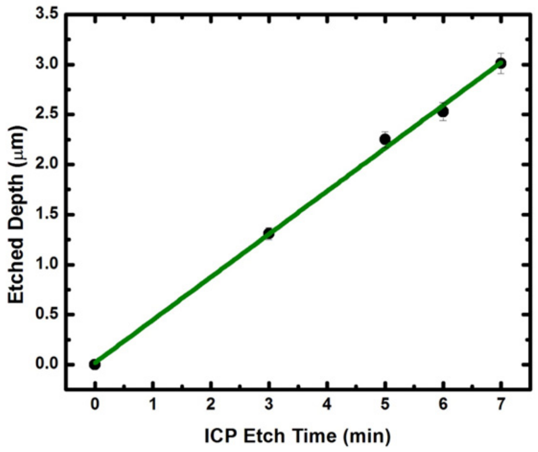
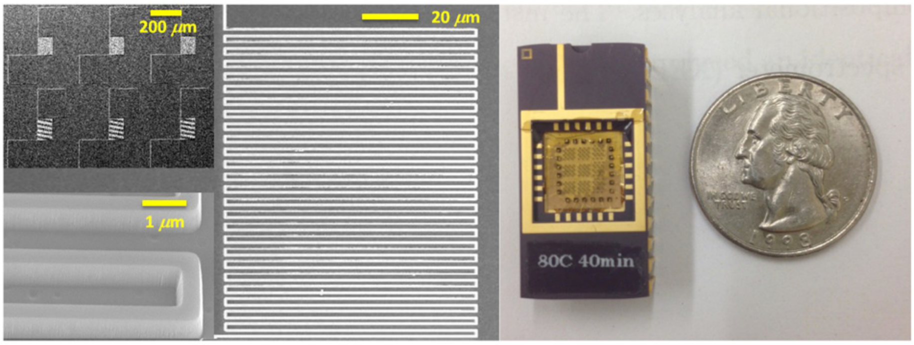


Fig. 3. ICP etch depth as a function of the etch time. The original data are shown by black dots, and a liner fit to the data shown by the solid green line.

A bi-layer metal stack (50 nm Ti and 120 nm Ni) was deposited by e-beam evaporator (Denton Infinity 22) and lifted-off. This was followed by ICP etching (Oxford Plasmalab 100) with a combination gases (Cl/N$_2$/Ar 25/5/1 sccm) at 40 °C temperature, 300 W RF power and 800 W ICP power. After removal of Ti/Ni metal shadow mask and SiN, potassium hydroxide (KOH) wet etching was studied at various temperatures (40 °C, 60 °C, and 80 °C) and in different solvents (deionized water, isopropanol, and ethylene glycol), and for different etching times from 10 min to 5 h. The fabrication steps for NW formation are shown in Fig. 1. PL measurements and SEM imaging were conducted before and after KOH etching as well as on the control sample (pristine GaN film). The optimized condition of KOH treatment (10% KOH in ethylene glycol etching for 2 h at 80 °C) was used on NWs. An ohmic contact metal stack (Ti/Al/Ti/Au) was deposited by e-beam evaporation for I–V characterization of the samples. The electrical properties of the no KOH treatment control group and a 2 h KOH etched group with different length NWs were studied. Due to the highly resistive property of intrinsic GaN, I–V measurements were conducted under UV (355 nm wavelength, 48 µW/cm^2 intensity) illumination. The width of both



(a) (b)

Fig. 2. (a) SEM image of single nanowire device. The total length is 5000 µm and width of 500 nm. The upper inset shows the identical NW arrays. The lower inset shows a higher magnification SEM image of the same nanowire with 30° tilt. (b) Final packaged device for photoconductance testing, a single die shown in the inset contains 81 NWs devices.