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Technical note

Lithographic sonication patterning of large area GaN nanopillar forests grown on a Si substrate☆

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ABSTRACT

This paper presents lithographic sonication patterning, a highly-scalable, material-independent method for patterning nanopillar forests. Through contact lithography, patterns with dimensions down to 3 μm were written across a 3-inch silicon wafer with a gallium nitride nanopillar forest grown through molecular beam epitaxy. Standard, ultraviolet lithography techniques were used to define a photoresist mask that protects covered nanopillars. Exposed nanopillars are removed via local cavitation in a deionized water ultrasonic bath. Sonication strips nanopillars 100 nm from their base, thus enabling further processing steps, including metal evaporation and substrate etching. As an example application, a four-point conductivity test device is demonstrated, where lithographic sonication patterning enables smooth, Ohmic contacts and successful dry etching of the silicon device layer. This method is compatible with commonly available cleanroom tools and provides a readily available alternative to more complicated fabrication approaches, such as selective nanopillar growth.

1. Introduction

Vertical nanowires, otherwise known as nanopillars, have found many potential applications across a wide range of disciplines, including energy harvesting [1,2], photovoltaics [3,4], integrated circuits [5,6], photonics [7–9], and sensing arrays [10]. Their high-aspect ratio enables dense, three-dimensional fabrication of unique structures with high surface-to-volume ratios from a variety of material systems. Industrial applications have been limited by the difficulty in controlling nanopillar location and orientation across large surface areas. Proof-of-concept nanopillar device fabrication and nanowire material property studies have often relied on dispersal into a solution before being deposited randomly or with modest control through dielectrophoresis [11]. Bottom-up selective growth techniques have been in development since the 1960s in an attempt to better control nanopillar location and diameter [12]. These efforts began with the vapor-liquid-solid (VLS) growth of nanostructures from catalyst droplets [13]. By controlling the diameter and placement of catalyst droplets, desired nanopillar arrays can be achieved. However, metal contamination from catalyst droplets during the growth phase has been shown to impair electrical properties [12]. Catalyst-free techniques have also been developed whereby a hard mask (typically silicon nitride or silicon dioxide) is used to confine nucleation during VLS or epitaxial growth [14]. Furthermore, work on top-down approaches has successfully enabled the fabrication of nanopillars etched out of the substrate with anisotropic wet or dry processing [15]. In all three cases, nanopillar diameters are governed by achievable resolution of patterning capabilities that include phase-shift photolithography, electron-beam lithography, nanoimprint lithography, and nanosphere lithography.

In applications where ordered nanopillar arrays are not needed but device operation benefits from having well-defined, micrometer-sized regions with and without nanopillars, a simplified approach can be used. Lithographic sonication patterning (LSP) is a wafer-scale, post-growth technique that involves traditional ultraviolet (UV) lithography to protect and remove selective regions of nanopillars. It is often compatible with most nanopillar-substrate material systems and provides definable resolutions down to several micrometers. After spin-depositing and developing a protective photoresist mask onto a nanopillar forest, a wafer or device can be sonicated in deionized water to remove unprotected nanopillars via local cavitation [16]. Because only standard UV lithography equipment and an ultrasonic bath are used, this technique is straightforward to implement in typical cleanrooms and is readily scalable.

2. Materials and methods

A gallium nitride (GaN) nanopillar forest was grown on a 3-inch Si ⟨111⟩ wafer by molecular beam epitaxy (MBE). GaN nanopillars grow along the c-axis, oriented perpendicular to the wafer surface [17]. These nanopillars are defect-free, possess a high mechanical

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Abbreviations: LSP, lithographic sonication patterning; MBE, molecular beam epitaxy.

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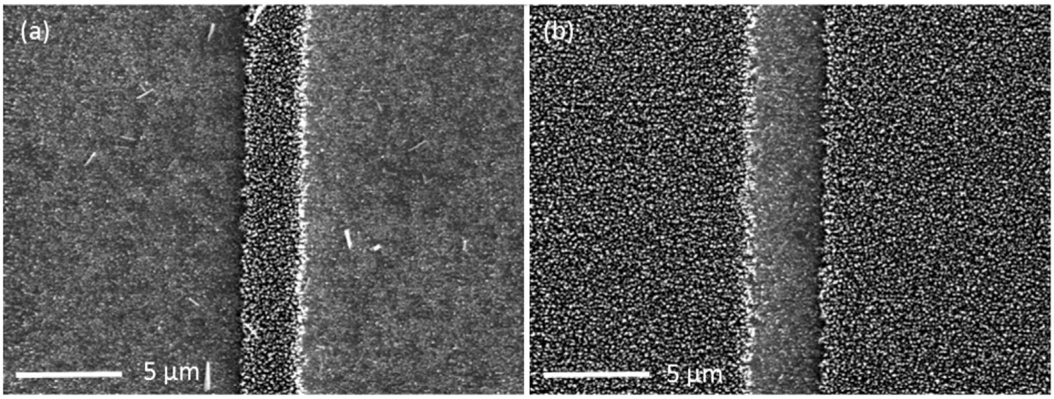

Fig. 1. (a) A positive line shape of protected GaN nanopillars with a width of 2.8 ± 0.4 µm. (b) A negative line shape of removed GaN nanopillars with a width of 3.4 ± 0.4 µm.

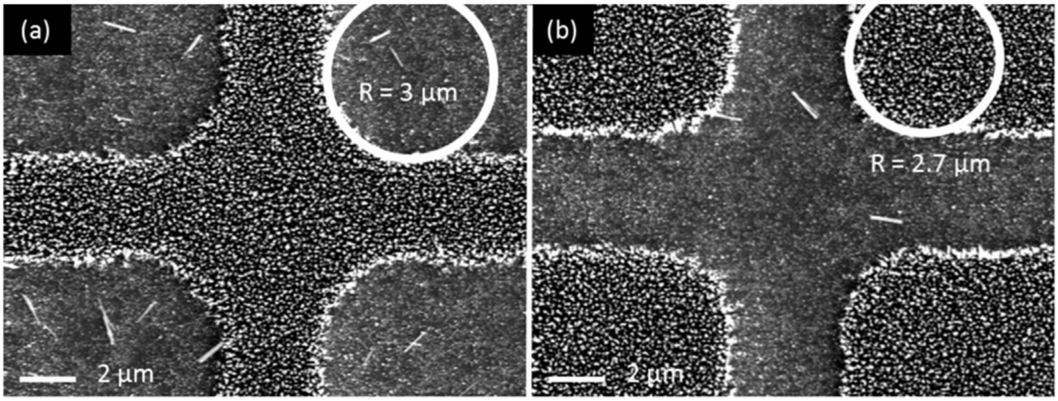

Fig. 2. (a) Positive cross shape with interior corners showing a radius of curvature of 3 µm after LSP and resist removal. (b) Negative cross shape with interior corners showing a radius of curvature of 2.7 µm after LSP, a 30% increase from the resist pattern. Stray, re-deposited NPs are present post-LSP. In the future, a low-power ultrasonic solvent clean on an inverted wafer may help further clean the sample.

quality factor, and can be doped for optoelectronic applications [18]. Characterization with a scanning electron microscope (SEM) showed nanopillar lengths to be about 1 µm with an average diameter of 50 nm. Post-growth, the wafer was plasma cleaned in O_2 with 60 W RF power for 5 min to improve resist adhesion. Microprime-P20[1] was spun at 3500 rpm for 40 s followed by positive resist SPR 220-7[1] at 1700 rpm for 60 s and a 240 s bake at 95 °C, yielding a mask thickness of ~6 µm. The wafer was then exposed for 48 s with a contact aligner using an 18 mW/cm^2 ultraviolet lamp before being developed in MF26A[1] for 180 s.

With the pattern prepared, the wafer was placed inside a 0.75 L beaker along with 100 mL of de-ionized water. The beaker was set inside a Branson CPXH[1] 3 L ultrasonic bath set to 50% power and the chamber filled with de-ionized water to a height of 1 cm. The wafer was checked at 2 min intervals to monitor the progress of the nanopillar patterning. After 20 min of sonication, the wafer was removed and the resist stripped via a solvent clean followed by a second 5 min O_2 plasma clean at 60 W.

3. Results and discussion

To determine the resolution limits and selectivity of nanopillar LSP, a wafer was patterned with an array of positive and negative lines and crosses with resolutions down to 3 µm (limited by the contact aligner and photoresist development). For this experiment, positive shapes are defined as those where nanopillars are protected while negative shapes are those where nanopillars are removed (Fig. 1). Scanning electron microscopy revealed that lines shapes with widths down to 3 µm survived the initial lithography step. However, the smallest positive line shapes after LSP was completed had a resulting nanopillar forest 2.8 ± 0.4 µm in width (Fig. 1a). By comparison, negative line shapes were also successfully developed down to widths of 3 µm with resulting trenches in the nanopillar forest resolved at 3.4 ± 0.4 µm (Fig. 1b). From these resolution experiments, it is evident that protective masking patterns are damaged on the order of 0.1 µm during sonication with desired positive and negative dimensions shrinking and expanding, respectively. Future experiments, in which patterns are defined by higher resolution lithography techniques such as electron beam lithography, are needed to determine the absolute limit of feature definition with LSP and establish the resolution as a function of nanopillar material, diameter, length, and masking resist.

In further experiments, positive and negative crosses yield identical minimum feature sizes as the lines. Analysis of the interior corners of the cross enabled quantification of any changes in the radius of curvature between resist pattern development in MF26A and nanopillar removal during sonication. Prior to sonication, interior corners of the positive resist cross features showed a radius of curvature of 3 µm. As is shown in Fig. 2a, this value remained effectively unchanged for the resulting NP pattern after LSP. By contrast, the interior corners of the negative resist cross feature had their radius of curvature increase by approximately 30% from 2.1 µm to 2.7 µm for the resulting NP pattern after LSP (Fig. 2b). This result indicates that positive convex resist

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[1] Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by NIST, nor does it imply that the materials are necessarily the best for the purpose.