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Fabrication and characterization of subwavelength nanostructures on freestanding GaN slab

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Abstract: We develop a novel way to fabricate subwavelength nanostructures on the freestanding GaN slab using a GaN-on-silicon system by combining self-assemble technique and backside thinning method. Silicon substrate beneath the GaN slab is removed by bulk silicon micromachining, generating the freestanding GaN slab and eliminating silicon absorption of the emitted light. Fast atom beam (FAB) etching is conducted to thin the freestanding GaN slab from the backside, reducing the number of confined modes inside the GaN slab. With self-assembled silica nanospheres acting as an etching mask, subwavelength nanostructures are realized on the GaN surface by FAB etching. The reflection losses at the GaN interfaces are thus suppressed. When the InGaN/GaN multiple quantum wells (MQWs) active layers are excited, the light extraction efficiency is significantly improved for the freestanding nanostructured GaN slab. This work provides a very practical approach to fabricate freestanding nanostructures on the GaN-on-silicon system for further improving the light extraction efficiency.

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References and links
1. Introduction

Gallium nitride (GaN) is a relatively new direct band gap III-V semiconductor material for optoelectric applications, especially for light emission. In principle it is easy to tune the emission wavelength of the GaN system by changing the indium content [1, 2]. It thus can fabricate GaN-based light emitting devices that cover the entire visible and near- to deep-UV spectrum. From the fabrication point of view, the substrate used for GaN growth is still one bottleneck for developing GaN-based light emission. Sapphire and SiC are the most common substrates of choice due to the fact that bulk lattice-matched substrates are not readily available. However, both sapphire and SiC are difficult to manufacture. In recent years, high quality a GaN-on-silicon system has been developed by using an intermediate buffer layer to reduce the crack density [3–6]. Additionally, bulk silicon micromachining is a mature technique. It offers the potential to fabricate optical components on freestanding GaN slabs by combining bulk silicon micromachining and the GaN-on-silicon system [7–10].

On the other hand, nanostructures can break the total internal reflection condition at the GaN surface to improve the light extraction efficiency [11–14]. Self-assemble technique is a very competitive approach to define nanostructures. Chiu et al. fabricated GaN nanopillars using self-assembled Ni nanomasks and inductively coupled-plasma reactive ion etching process [15]. Ng et al. introduced photonic crystal structures with silica microspheres acting as an etching mask, where silica microspheres are self-assemble into a single layer hexagonal array on the GaN surface [16]. Chan et al. presented a method of fabricating GaN nanostructures using polystyrene spheres and its inverted structure as the etching mask [17]. Such nanostructures modify the light propagation direction at the GaN surface. The reflection losses at the GaN interfaces are reduced, and the light extraction efficiency is thus increased. Moreover, backside thinning of the freestanding GaN slab can reduce the number of confined modes inside the GaN slab. Wierer et al. demonstrated a laser lift-off and subsequent backside thinning technique to reduce the number of confined modes inside the GaN slab [18]. These methods are effective to enhance the light extraction efficiency when the InGaN/GaN multiple quantum wells (MQWs) active layers are excited.

The overall objective of this work is to fabricate subwavelength nanostructures on the freestanding GaN slab using the GaN-on-silicon system. The freestanding GaN slab is obtained by removing silicon substrate, and backside thinning of the freestanding GaN slab is realized by fast atom beam (FAB) etching. FAB etching uses energetic neutral particles for dry etching, and the FAB has good directionality of the beam flux [19]. As a neutral dry etching technique, FAB etching is advantageous for etching GaN layer with high lateral resolution. Subwavelength nanostructures are achieved on the GaN surface by FAB etching with self-assembled silica nanospheres acting as an etching mask. The light extraction efficiency is thus improved by reducing the number of confined modes inside the GaN slab, eliminating silicon absorption of the emitted light, and decreasing the losses caused by the total internal reflection.

2. Device fabrication

The starting material, consisting of 200nm GaN layer, 450nm AlGaN layer, 200nm AlN buffer layer and 200µm silicon handle layer, is a 20mm×20mm substrate which is cut from a commercial 4-inch GaN template grown on silicon substrate by metal organic chemical vapor deposition (MOCVD). The InGaN/GaN MQWs active layers are deposited on the GaN template by molecular beam epitaxy (MBE) with radio frequency nitrogen plasma as gas source [20–22]. A 90nm buffer layer, which incorporated 6nm AlN layer, twelve-period 3nm AlN/3nm GaN superlattice and 12nm GaN layer, was firstly deposited on the GaN template. A 950nm n-type GaN layer was then deposited, and the MQWs active layers consisting of three-period 3nm InGaN/9nm GaN were subsequently grown. A 6nm p-type AlGaN layer and a 50nm p-type GaN layer were finally deposited.

Fig. 1. Fabrication process of subwavelength nanostructures on the freestanding GaN slab

The schematic bottom-up process is illustrated in Fig. 1. The top GaN device layer was first protected by thick photoresist, and the silicon substrate was patterned from the backside by photolithography (steps a). The silicon substrate was then etched down to the AlN layer using deep reactive ion etching (DRIE), where the AlN layer acted as a definite etch stop (steps b). The freestanding GaN slab was thinned to a thickness of ~900nm from the backside by FAB etching, which was conducted with the Cl₂ flow of 7sccm at the high voltage of 3.0KV and the accelerated current of 10mA (steps c). After removing the residual resist, silica nanospheres were spin-coated onto the GaN top surface with the rotation speed of 3500rpm for 40sec, which makes the silica nanospheres self-assemble into a single layer close-packed array. In order to evaporate the solvent, the substrate was subsequently kept at 90°C for 10min and 145°C for 30min, respectively (steps d). With an etching mask of silica nanospheres, the nanostructure patterns were transferred to the GaN slab by FAB etching [22–24], and the residual silica particles were finally removed in buffered HF solution, generating subwavelength nanostructures on the freestanding GaN slab (steps e).
3. Experimental results and discussion

Figures 2(a) and 2(b) show the 30° tilt-view scanning electron microscope (SEM) images of silica nanospheres forming a close-packed single layer on the GaN surface. These silica nanospheres have a uniform diameter size of about 170 nm and display short-range order. Subwavelength nanostructures are transferred to the GaN surface by FAB etching, as illustrated in Figs. 2(c) and 2(d). The tilt views exhibit the well-defined subwavelength GaN nanostructures using self-assemble technique. As an etching mask, silica nanospheres are also etched during FAB etching. The fabricated subwavelength GaN nanostructures are tapered columns due to the silica nanosphere profiles and the good directionality of the neutral FAB etching. The diameters of the GaN nanocolumns are determined by the size of the silica nanospheres, ~170 nm, and the depth of the resultant GaN nanocolumns are about 170 nm, which can be controlled by the etching time. Figure 2(e) illustrates an optical micrograph of the freestanding GaN slab with subwavelength texturing. The size of square freestanding GaN slab is about 300 µm x 300 µm. Although the freestanding GaN slab has a small downward deflection due to the residual stress [22, 24], subwavelength texturing is well conducted in the whole device area.

Fig. 2. (a) and (b) the tilt-view SEM images of single-layer close-packed silica nanospheres; (c) and (d) the tilt-view SEM images of fabricated GaN nanostructures; (e) optical micrograph of nanostructures on the freestanding GaN slab.
The emission properties of fabricated samples are characterized using a microphotoluminescence (micro-PL) system at room temperature. The excitation source is a continuous wave He-Cd laser with a peak wavelength 325nm, and the pump light is focused on the GaN slab through a UV-compatible objective lens (20× and numerical aperture:0.36). The emitted light is collected by the same objective lens and measured using a multichannel Hamamatsu C10027 analyzer system.

Figure 3(a) shows the measured PL spectra of the GaN slabs, where the pump light is incident on the top GaN surface. The original GaN slab on silicon substrate is measured as reference, and the luminescence peak at 428nm is normalized to 1. A 3.3-fold enhancement in the peak intensity is observed at 415nm for the GaN slab with surface texturing, and the luminescence intensity integrated over the emission spectrum is increased by 3.1 times. Subwavelength nanostructures have a periodic distribution of the refractive index and modify the light-propagation direction at the GaN surface. Therefore, the total internal reflection condition is broken and the reflection losses are suppressed, which is able to help more photons leave the device when the MQWs active layers are excited by the pump laser beam. The measured PL intensity is thus improved.

Fig. 3. (a) PL spectra of fabricated samples obtained from the top GaN surface; (b) PL spectra of fabricated samples obtained from silicon substrate side.

After backside thinning of the freestanding GaN slab, silicon absorption of the emitted light is eliminated and the number of optical modes is reduced. Hence, the light extraction efficiency is increased. The measurements confirm the improvement in the light-extraction efficiency of the freestanding GaN slab. A 2.9-fold peak intensity enhancement and a 3.1-fold integrated intensity enhancement are obtained, and the intensity peak is observed at 423nm. The freestanding GaN slab with surface texturing can take all the mentioned advantages, i.e., reducing the number of confined modes inside the GaN slab, eliminating silicon absorption of the emitted light, and modifying the light propagation direction at the GaN surface. The experimental results provide solid evidences on significantly improving the light extraction efficiency. The luminescence peak at 416nm has a 14-fold enhancement in the peak intensity, and the integrated intensity is also increased by a factor of 14, as shown in Fig. 3(a).

Figure 3(b) illustrates a clear enhancement in PL intensity when the pump light is incident on the freestanding GaN slab from silicon substrate side. Compared with the PL intensities of the original GaN slab on silicon substrate, the luminescence peak is observed around 402nm for the freestanding GaN slab measured from the backside, and the enhancements of the integrated intensity and the peak intensity are about 2 times and 2.5 times, respectively. Regarding the freestanding GaN slab with surface texturing, subwavelength nanostructures work as patterned substrate to increase the light extraction efficiency [2, 25]. A 4.6-fold peak intensity enhancement and a 4.3-fold integrated intensity enhancement are measured, and the PL intensity peak is found at 407nm. These results indicate that the PL intensity form the backside is also significantly improved by introducing subwavelength texturing of the GaN surface.

Figure 4(a) shows the measured reflectivity from the top GaN surface, where the interference fringes are attributed to the multiple reflections of visible light at the GaN interfaces. Subwavelength nanostructures show excellent anti-reflective characteristics [12, 26]. The losses due to the total internal reflection are decreased after subwavelength texturing of the GaN surface. The measured reflectivity and the interference peaks of the nanostructured GaN slab on silicon
substrate are much smaller than those of the original GaN slab on silicon substrate. The subwavelength texturing technique can help more excited photon to escape out the GaN slab and thus enhance the extraction efficiency, as shown in Fig. 3(a). The blue curve illustrated in Fig. 4(a) represents the measured reflectivity of the freestanding nontextured GaN slab. The number of confined modes inside the GaN slab is decreased by backside thinning of the freestanding GaN slab, resulting in the reduction of the interference fringes. As to the freestanding nanostructured GaN slab, the reflectivity is decreased due to the reduction of the total internal reflection, and the number of confined modes is also reduced. These reflection measurements are in line with the above analysis on the improvement in the light extraction efficiency by reducing the number of optical modes inside the GaN slab, eliminating silicon absorption of the emitted light, and decreasing the reflection losses. Figure 4(b) illustrates the reflectivities of the freestanding GaN slab measured from the backside and it is shown that they behave very similarly to those obtained from the top side of the freestanding GaN slab.

![Graph](image)

**Fig. 4.** (a) Reflectivity of fabricated samples obtained from the top GaN surface; (b) Reflectivity of fabricated samples obtained from silicon substrate side.

### 4. Conclusions

We develop a novel way to fabricate subwavelength nanostructures on the freestanding GaN slab using the GaN-on-silicon system by combining self-assemble technique and backside thinning method. Bulk silicon micromachining can realize the freestanding GaN slab, and FAB etching is able to achieve backside thinning of the freestanding GaN slab and well-defined subwavelength GaN nanostructures. The light extraction efficiency is thus improved by reducing the number of confined modes inside the GaN slab, eliminating the silicon absorption of the emitted light, and decreasing the losses caused by the total internal reflection. This work provides a very practical approach to fabricate freestanding nanostructures on the GaN-on-silicon system for further improving the light extraction efficiency.

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