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Fabrication of 2D photonic crystals using block copolymer patterns on as grown LEDs

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ABSTRACT

Di-block copolymer polystyrene-block-polymethyl methacrylate (PS-b-PMMA) was used to make patterns over a large area of as grown LEDs. The polymer patterns on LEDs surface could be transferred to the underlying p-GaN, the topmost layer of as grown LEDs by both reactive ion etching (RIE) and photo-enhanced chemical (PEC) etching. Removal of remaining polymer chains results in patterned LEDs which shows higher light extraction efficiency. In our experiment, much higher intensity for patterned LEDs in both photoluminescence (PL) and electroluminescence (EL) data plot were found. Similar improvements were found in *I–V* and *L–I* curves for patterned LEDs.

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1. Introduction

Due to the limitations of conventional lithographic techniques, alternative methods for patterning the substrates on a nanoscale level have attracted considerable interest. Among those methods, block copolymer lithography appears very promising for creating regular periodic patterns. Successful nanopatterning has been reported using block copolymers with spherical [1–4] and cylindrical [3–9] morphologies. Self-assembly of block copolymers is a versatile way to prepare nanoparticles and to control their size, shape, and location [10–12]. One commonly studied diblock copolymer is polystyrene-block-methyl-methacrylate (PS-b-PMMA) where the blocks PS and PMMA are chemically and physically distinct.

Gallium nitride (GaN) has become a prominent material in the optoelectronics market, with numerous evolutionary products that have been commercialized such as light-emitting diodes (LEDs) and laser diodes [13]. Recently, as the brightness of GaN-based LEDs has increased, applications such as displays, traffic signals, backlights for cell phones, exterior automotive lighting, and printers have become possible. But due to the large difference in refractive indices between GaN materials and outer ambient air, only a limited fraction of extracted photons can escape from the LEDs to the air. Research into improving the light extraction efficiency (external quantum efficiency) and brightness in the LEDs has been intense. In particular, rapid progresses have been reported in the past several years to increase the light extraction from the LED by roughening or incorporating the photonic crystal structures into the emitting surface with the help of nanoscale patterning technology such as porous anodic alumina [14], laser interface lithography (LIL) [15–18], e-beam lithography (EBL) [19], nanoimprint lithography [20] and colloidal lithography [21]. In this paper, an approach is presented to fabricate photonic crystal structure using block copolymer self-assembly on as grown LEDs, resulting much higher light extraction efficiency.

2. Materials and methods

Diblock copolymer PS-b-PMMA with Mn: PS (46,100), PMMA (21,000) and Mw/Mn: 1.09, having a PS volume fraction of 0.69 (Polymer Source, Inc.) was used as polymer materials. One wt.% polymer solution in toluene was spin-coated onto as grown LEDs surface to form a thin polymer film. The samples were then baked for 48 h with a continuous flow of Ar gas at approximately 200 °C, which is well above the glass transition temperatures (T_g) of both PS (100 °C) and PMMA (115 °C). During baking, the polymer components self-assemble into a phase separated layer on the substrate surface. Being negative photoresist, PMMA chains get degraded upon exposure to UV irradiation and could be easily removed by rinsing in acetic acid followed by wishing with DI water to get PS cylindrical patterns. A certain depth of the top layer of as grown LEDs, p-GaN was etched along the gaps of PS cylinders by means of both reactive ion etching and photo enhanced chemical etching. Cl_2/Ar gas was used for reactive ion etching and the

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samples were stained with ruthenium tetroxide (RuO_4) vapor prior to the etching process [22]. For photo enhanced chemical etching, the samples were kept on 0.04 M KOH solution and exposed to UV exposure. Finally the remaining PS cylinders were removed using acetone which results in patterned LEDs.

The surface morphology was examined by field emission scanning electron microscopy (FE-SEM, JEOL JSM-7401F). The optical property of patterned LED was measured by photoluminescence spectroscopy (PL, Accent RPM 2000) with Nd-YAG laser as the excitation light source with wavelength of 266 nm at room temperature. The performance of patterned LED was made by electroluminescence measurement (Optel-Precision OPI-150) at the injected current of 20 mA.

3. Results and discussion

Diblock copolymers are macromolecular surfactants those are capable of spontaneous self-organization into several ordered microphases, depending principally on molecular composition, if the constituent (A and B) sequences are sufficiently incompatible [23,24]. With care, the spin coating method can give films with low surface roughness over areas of square of millimeters. The film thickness can be controlled through the spin speed, the concentration of the block copolymer solution or the volatility of the solvent, which also influences the surface roughness [25]. Polystyrene (PS) and poly methylmethacrylate (PMMA) have significantly different photodegradation properties. PMMA is known to be a negative photo resist, i.e., with UV or electron beam irradiation, the polymer chains get degraded via chain scission. The chemical processes taking place in PS upon exposure to deep UV radiation, on the other hand are less well defined, with cross linking, chain scission, and oxidation taking place [8]. Deep UV exposure of ordered PS-b-PMMA should, therefore, lead to a degradation of the PMMA block, whereas the PS matrix becomes insoluble. The degradation products from PMMA can then be rinsed away, leaving a patterned surface.

The PS cylindrical patterns after removing PMMA can be seen in the FE-SEM images (Fig. 1). In our experiment, to ensure the removal of unreacted polymer chains the samples were sonicated in toluene after baking and to ensure the complete removal of PMMA chains after UV exposure, the samples were sonicated in acetic acid for small times. The patterns could be made throughout the entire surface. Fig. 1a shows the high magnification SEM image of the surface patterns of PS cylinders and Fig. 1b is the low magnification SEM image where it shows the uniformity of the patterns over the whole surface.

The PS pattern on as grown LED could be transferred to underlying p-GaN BY reactive ion etching (RIE). Before so doing, the samples were exposed to ruthenium tetroxide vapor, which yields PS cylinders containing Ru atoms and therefore exhibits improved chemical and thermal stability. In our experiment, the RIE has been done with Cl_2/Ar gas having flow rates 10/25 sccm using an inductively coupled plasma (ICP) reactor. ICP/bias power = 300/100 W was used with a chamber pressure of 5 mTorr.

After etching, the PS polymer chains were removed using acetone. The removal of PMMA leaves the LEDs with p-GaN patterns on its surface. The patterns of p-GaN on LEDs surface can be seen

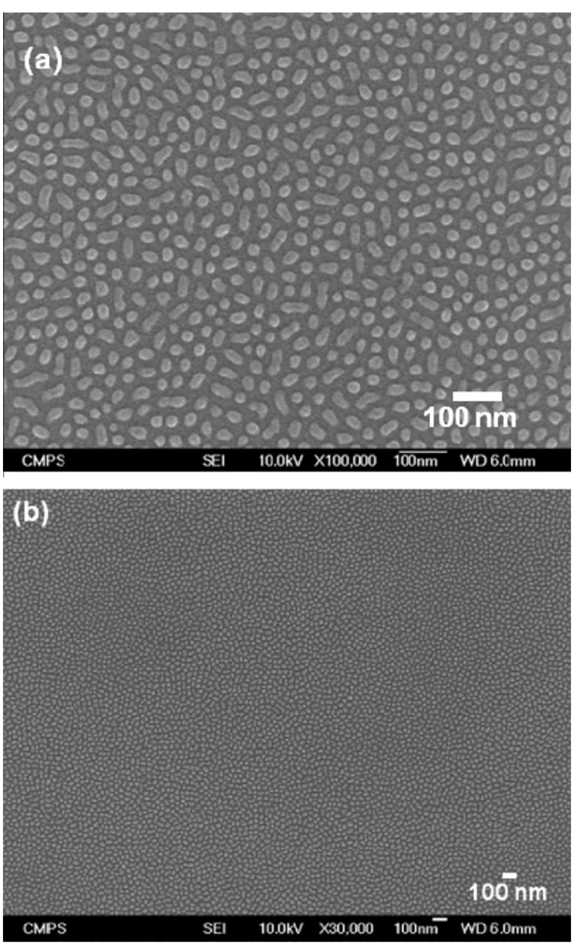

Fig. 1. (a) High magnification and (b) low magnification images of the PS patterns on as grown LEDs.

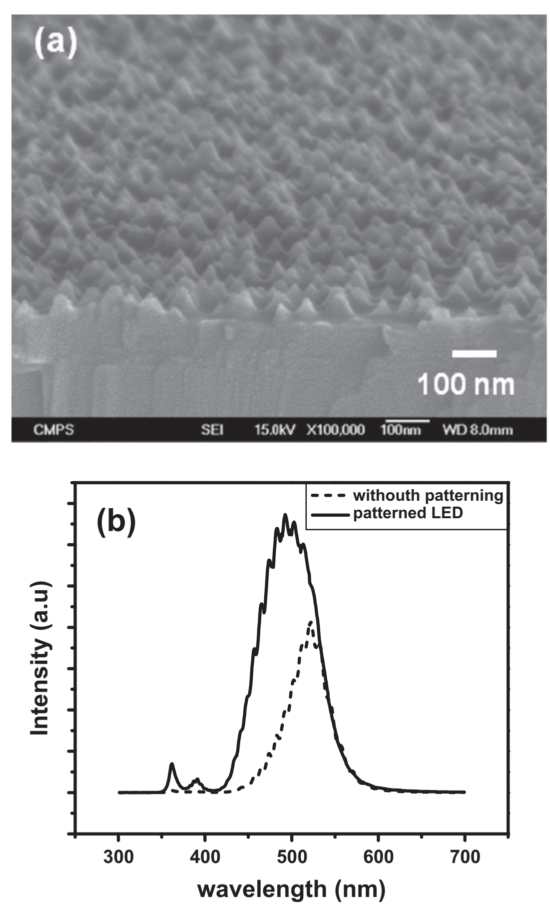

Fig. 2. (a) The patterned surface of LEDs after RIE and removing PMMA and (b) the comparison of PL intensity of as grown and patterned LEDs.

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