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Fabrication of transferrable, fully suspended silicon photonic crystal nanomembranes exhibiting vivid structural color and high-Q guided resonance

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The authors report the fabrication and optical characterization of fully suspended, transferrable, and deflectable silicon photonic crystal nanomembranes. Starting with a silicon-on-insulator wafer, the authors used electron beam lithography and inductively coupled plasma reactive ion etching (ICP-RIE) to introduce various photonic crystal patterns in silicon. A membrane containing the photonic crystal patterns was then defined by photolithography combined with ICP-RIE and released from the handle wafer by wet chemical etching. Finally, a free-standing photonic crystal membrane was obtained by a wet transfer and alignment process over a perforated foreign substrate. In the fabricated structures, the authors observed vivid structural colors in dark-field optical images of square lattice photonic crystals and measured a guided resonance mode with a quality factor as high as 5600 in a novel slot-graphite photonic crystal. © 2013 American Vacuum Society.

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I. INTRODUCTION

Photonic crystal slabs, dielectric slabs patterned with two-dimensional arrays of holes, are known to support guided resonance modes.1 Such modes strongly enhance the optical near field of the slab while simultaneously couple to external radiation. Their unique optical properties open up tremendous opportunities for realizing compact, surface-normal optical components such as filters, mirrors, and sensors.2,3 Quite recently, it has been demonstrated that guided-resonance modes can also be leveraged for guided self assembly of nanoparticles.4,5

Transfer of a photonic crystal slab to a foreign substrate can yield additional desirable properties, such as transparency and flexibility.6–9 If the slab is fully suspended in air instead (“air-bridged”),10–13 its deflectable nature enables numerous interesting optomechanical applications.14–16 Furthermore, the absence of semiconductor materials above or below the photonic crystal slab can improve filtering functionality13 and allow accurate optical measurements in the visible range.10,17

In this work, we developed a fabrication process for making silicon photonic crystal nanomembranes that are both transferrable and fully suspended, which is difficult using either an oxide under-etching technique10–13 or a dry-transfer process with Polydimethylsiloxane (PDMS) stamp alone.5,7 The photonic crystal pattern was introduced into the silicon device layer of a silicon-on-insulator (SOI) wafer, using electron beam lithography combined with inductively coupled plasma reactive ion etching (ICP-RIE). A membrane containing the photonic crystal pattern was then defined by photolithography and released from the handle wafer in hydrofluoric acid. Finally, we wet-transferred and positioned the membrane over an opening in a foreign substrate to obtain a fully suspended configuration. We observed vivid visible structural color in dark-field mode optical microscopy and measured a high quality factor (~5600) guided resonance mode in the near infrared, confirming the excellent optical quality of the fabricated structure.

II. FABRICATION

The fabrication process starts with a SOI wafer (SOITEC) consisting of a 340 nm device layer and a 2 μm buried oxide layer. We first spin coated the wafer with an approximately 260 nm-thick layer of electron beam resist (PMMA-A4 950K). The resist was prebaked in an oven at 170° for 70 min. We then performed electron beam lithography with an electron acceleration voltage of 30 kV on a Raith 150 e-Line system to define various photonic crystal patterns in the e-beam resist. Each photonic crystal pattern was defined in a circular region with 50 μm diameter. Proximity effect correction was performed by the NanoPECS module in the Raith software. After the e-beam exposure, we developed the e-beam resist in a 1:3 mixture of methyl isobutyl ketone and isopropyl alcohol (IPA) and then rinsed the sample in IPA. The sample was postbaked on a hot plate at 100° for 2 min. In order to transfer the photonic crystal pattern from the e-beam resist to the silicon device layer, we performed ICP-RIE (PlasmaPro100 ICP180, Oxford Instruments) with a mixed-mode Pseudo-Bosch process,18 in which the etching gas SF6 and the passivation gas C4F8 flow simultaneously into the etching chamber. We optimized the etching recipe to obtain a smooth, vertical sidewall profile, which is critical for the optical quality of the photonic crystals. The sidewall profile angle determined from scanning electron microscope (SEM) inspection is around 90°. The pseudo-Bosch process we use here is known to produce a much smoother sidewall than the traditional Bosch process.

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In the literature, a less than 5 nm surface roughness has been reported for nanoscale silicon nanowires etched with a pseudo-Bosch process. The parameters of the optimized recipe were 33 SCCM of SF$_6$ and 57 SCCM of C$_4$F$_8$, 20 mTorr pressure, 600 W ICP power, and 30 W RF forward power. These settings give a DC bias voltage of around 200 V. Under these conditions, the PMMA resist to silicon etching selectivity is around 1.8:1. After etching, the remnant e-beam resist was removed in acetone followed by oxygen plasma.

After the fabrication of the photonic crystal pattern, we performed standard ultraviolet (UV) photolithography to define a 6 mm $\times$ 6 mm square membrane region in the silicon device layer, as shown in Fig. 1(a). The central square part of the membrane with dimensions of 500 $\mu$m $\times$ 500 $\mu$m was aligned to overlap with the photonic crystal patterns [shown as 9 solid circles in Fig. 1(a)], while the rest of the membrane was patterned with an array of circular access holes (40 $\mu$m in diameter, 500 $\mu$m in pitch) to facilitate the wet chemical etching of the buried oxide layer in hydrofluoric acid. Specifically, we spin coated our sample with AZ5214 positive photoresist and soft-baked the resist at 100°C for 2 min. After being exposed to UV light in a mask aligner (Karl Suss MJB-3), the photoresist was developed in AZ400K (1:4 concentration) and postbaked at 170°C for 2 min. ICP-RIE etching was used to transfer the membrane pattern to the sample, using the same etch parameters as above. The remaining photoresist was stripped in acetone and oxygen plasma.

In order to release the silicon membrane from the handle silicon substrate, we immersed the sample in hydrofluoric acid (concentration, 48%–51%, Avantor-Macron Fine Chemicals) for a few hours to completely remove the buried oxide layer. The released nanomembrane weakly adheres to the underlying silicon handle wafer. When transferred to deionized water, mild agitation of the water causes the membrane to detach from the substrate and float, as shown in Fig. 1(b).

The final step of the process is to position the nanomembrane over a perforated window in a host substrate. The host substrate can be chosen from a wide variety of materials, including glass, plastic, and oxidized silicon. Here we focus on oxidized silicon. We start with a plain [100]-oriented silicon wafer and perforate it using either mechanical drilling or wet chemical etching in KOH solution. In the latter case, we first deposited silicon nitride on the wafer by low pressure chemical vapor deposition to serve as a wet-etching mask, and then defined a window pattern by UV photolithography and electron cyclotron resonance reactive ion etching (RIE). After the KOH solution etched through the entire silicon wafer, the silicon nitride mask was removed in hydrofluoric acid. Finally, a thin layer (~50 nm) of thermal oxide was grown on the surface of the perforated silicon wafer in a furnace. The purpose of this oxidation step is to ease the manipulation of the nanomembrane on surface of the host substrate.

The detailed wet transfer and positioning process is described as follows. First, the floating membrane was picked up by a glass slide and transferred from water to IPA. IPA has a lower surface tension and higher viscosity than water. We find that mild agitation of the IPA can be used to dislodge the membrane, allowing repositioning on the substrate. The membrane is extremely flexible, compared to a silicon wafer, and can be repositioned many times without damage. We can therefore first pick up the nanomembrane using the host substrate and then reposition the membrane until the central part containing the photonic crystals lies over the perforated window [Fig. 1(c)]. Finally, after the
evaporation of IPA, we obtain a fully suspended silicon photonic crystal nanomembrane.

A perspective SEM image of the fabricated silicon photonic crystal nanomembrane is shown in Fig. 2(a). In this case, the silicon membrane was positioned over a circular, mechanically perforated opening in an oxidized silicon wafer. The central square area of the membrane is patterned with nine different photonic crystal devices, as indicated by nine ellipses. We have fabricated several different photonic crystal patterns, including a square lattice [Fig. 2(b)] and a novel slot-graphite lattice [Fig. 2(c)]. The slot-graphite lattice is created by placing a rectangular slot in the center of each unit cell in the regular graphite photonic crystal structure. Our simulation results show that such a lattice supports a high-quality-factor Τ-point guided resonance mode (referred to as a “slot mode”), which can couple to an external plane wave in the vertical direction.

III. OPTICAL CHARACTERIZATION

In order to assess the optical quality of the transferred photonic crystal nanomembranes, we perform both dark-field reflection optical microscopy in the visible range and cross-polarization optical transmittance measurement in the near infrared range.

A. Structural color

Dark-field reflection optical microscopy was performed in a Nikon microscope with a 20× objective. In dark-field mode, the microscope objective provides oblique incidence light and collects scattered rather than directly reflected light. Figure 3 shows the dark-field optical microscope image of a free-standing silicon membrane consisting of nine different photonic crystal patterns. The patterns are square lattice photonic crystals with lattice constants ranging from 350 to 750 nm and a fixed radius to lattice constant ratio (r/a) of around 0.28. We can clearly observe that the color of the photonic crystal pattern gradually changes from violet to yellow with increasing lattice constant. The color originates from reflected diffraction orders supported by the periodic lattice.

We have also observed vivid structural colors in bright-field optical microscope images, with both front and back illumination (not shown here). The tunable structural colors observed here will be useful for applications such as color filters and refractive index sensors.

![Fig. 2. (Color online) (a) Perspective-view SEM picture of a fabricated silicon photonic crystal nanomembrane. Nine ellipses indicate device regions. Top-view SEM images of a square lattice (b) and slot-graphite lattice (c) photonic crystals.](image1)

![Fig. 3. (Color online) Dark-field optical microscope images of a silicon nanomembrane patterned with nine square lattice photonic crystals with varying lattice constants from 350 to 750 nm with 50 nm spacing. The scale bar is 100 μm.](image2)
B. High-Q guided resonance

The optical transmittance measurement was carried out in a custom cross-polarization transmission setup. In cross-polarization measurement mode, the nonresonant path through the photonic crystal membrane contributing to the Fabry–Perot background in the transmission spectrum is suppressed, while the resonant transmission at the resonance frequency is readily detected. As a result, guided resonance modes appear as peaks in the transmission spectrum. The incident light from a fiber-coupled tunable semiconductor laser in the near infrared range (1500–1620 nm) was first collimated and then focused by a microscope objective (Mitutoyo, 5×, N.A = 0.14) to illuminate the photonic crystal area of interest. The transmitted light through the photonic crystal nanomembrane was collected by an achromatic doublet lens (f = 30 mm, Thorlabs) and then focused into a single mode optical fiber connected to a photodetector. The transmittance spectra were obtained by synchronizing the laser source and the photodetector in a LABVIEW program. In order to perform the transmission measurement in cross-polarization mode, two polarizers were inserted before the illuminating objective and after the collecting lens to provide separate control on the polarization of the incident light and transmitted light.

We fabricated a slot-graphite lattice photonic crystal and adjusted the thickness of the membrane by ICP-RIE etching so that the slot mode was within the wavelength range of our laser. Figure 4 shows a representative cross-polarization transmission spectrum of the slot-graphite lattice photonic crystal pattern shown in Fig. 2(c). The resonance peak was fitted to a Fano lineshape and exhibited a high quality factor of around 5600. The extended nature of the mode makes it a good indicator of the optical quality of the fabricated device throughout the entire device area. The quality factor measured here was comparable to that of a slot-graphite device with the same lattice but on oxide.

IV. SUMMARY

In summary, we have successfully fabricated transferrable, fully suspended, and deflectable silicon photonic crystal nanomembranes with good optical quality. We observed vivid structural colors in a membrane patterned with square lattice photonic crystals in dark-field optical microscopy. We also measured a guided resonance mode with a quality factor up to 5,600 in a slot-graphite lattice photonic crystal. We expect the fabrication process reported here will be useful for the study of optomechanical interactions based on photonic crystal slabs, as well as characterizing the optical properties of semiconductor thin films with photonic crystal patterning in the visible range.

In future work, we will investigate how we can improve the precision of the wet positioning process, possibly with the assistance of an optical microscope (compared to the naked eye, used in the current process), as well as a more controlled liquid flow for membrane manipulation. In addition, the use of optical tweezers or the predefinition of embedded magnetic materials in the nano-membrane are interesting approaches for further research. Ultimately, we expect the wet transfer and positioning technique can be used for the stacking of nanomembranes with different photonic functionalities and enable a wide range of interesting applications.

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Fig. 4. (Color online) Representative cross-polarization transmission spectra of the silicon nanomembrane patterned with a slot-graphite photonic crystal.
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