Microphotonics and Lithographic Structures
Three-Dimensional Yablonovite-like Photonic Crystals by Focused Ion Beam Etching of Macroporous Silicon

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ABSTRACT

We report on fabrication of three-dimensional photonic crystals by focused-ion-beam etching of macroporous silicon. The fabricated Yablonovite-like structures contain up to 25*25*5 lattice cells with a periodicity of 0.75 μm. The presence of the photonic bandgaps at wavelengths close to 3 μm is shown from reflection measurements and confirmed by numerical calculations. Further reduction of the periodicity is possible using the same technology.

INTRODUCTION

Since more than one decade, the focused ion beam (FIB) has been widely used for submicrometer patterning of various solid materials [1,2]. This technology offers the possibility of dry etching that is much more flexible than traditional etching methods. In particular it allows maskless etching of patterns at any angles with respect to the substrate surface. One of the major drawbacks of the FIB etching is that the etched patterns have low aspect ratio (<10), due to several secondary effects [3,4]. We show that these depth-limiting secondary effects are strongly reduced if, instead of bulk samples, the FIB etching is performed on a microscopically pre-structured (i.e., containing microscopic cavities) material. The etching of extremely deep high aspect ratio holes then become possible [5]. Following this approach, we were able to fabricate a three-dimensional (3D) photonic crystal in pre-structured macroporous silicon.

EXPERIMENTAL

We choose to fabricate a Yablonovite structure that consists of 3 series of holes with axes following the (110), (101) and (011) vectors of a fcc lattice [6]. A combination of photoelectrochemical [7] and FIB etchings is used for hole drilling. None of these two methods alone allows the fabrication of 3D structures. The chemical etching can only generate 2D lattices of holes that are perpendicular to the substrate surface. As far as the FIB etching is concerned, the holes have sloped sidewalls and, thus, limited depth for a given hole opening diameter [4]. The origins of these phenomena are attributed to the in-hole redeposition of the sputtered material, the so-called “self-focusing” of the beam as well as to the presence of gaussian wings in the ion beam transverse profile [3,4].

The redeposition and the self-focusing effects are intrinsic to the FIB etching in bulk samples. However, if the sample has a porous structure, these effects are strongly reduced [5]. Indeed, 1) the sputtered material is evacuated into the adjacent cavities, each wall between cavities is thus etched in an almost independent way, canceling the redeposition effects; 2) the self-focusing effect is reduced because the etched holes have discontinuous side-walls along the beam direction. Thus, the maximum aspect ratio of the FIB etched
patterns is dramatically increased and it is possible to drill deep and high aspect-ratio patterns in porous substrates using FIB.

The fabrication is divided into two stages. First, the macropores (submicrometer holes) situated following a two-dimensional triangular lattice are obtained by photo-electrochemical etching in low-doped monocrystalline silicon substrate. This structure is initially designed for two-dimensional photonic crystals. The macropores are about 50 \( \mu \text{m} \) deep, with a periodicity of 0.65 \( \mu \text{m} \) [8].

In the second stage, the two other sets of holes, crossing the macropores with predefined angles, are drilled using FIB to achieve the three-dimensional structure.

The arrangement of the three sets of holes (macropores and FIB etched holes) is presented in figure 1. The three-dimensional photonic crystal we fabricate is actually Yablonovite-like. The two sets of FIB etched holes (indicated by arrows) form an angle of about 51.3° between them (\( \gamma \)), and each of them forms an angle of 60° (\( \alpha \) and \( \beta \)) with the macropores. We made this choice to get a structure as close as possible to a Yablonovite, for which \( \alpha = \beta = \gamma = 60° \), since it is impossible to generate a strict Yablonovite from a two-dimensional triangle lattice (an oblique 2D lattice of macropores with an angle of 70.52° should to be used instead). The FIB etching positions on the sample surface are defined by the openings of the macropores. It is easy to check that to form a Yablonovite-like structure whose hole crisscross coincide with the nodes of a face-centered lattice, only one row of macropores out of two will be concerned [5].

![Figure 1. A schema showing the geometrical relation between the macropores and the FIB drilled holes. The later are indicated by the arrows](image)

The FIB is generated by a Canion ion gun, provided by Orsay Physics. It uses a hairpin type needle to form the liquid Ga+ metal ion source. The ion gun is connected to an ultrahigh vacuum chamber. The apparatus is designed for both micro machining and visualization of the samples. In the later function the scanning system can reach a high speed (up to 40 megapixel/s) to reduce the sample surface erosion [9].

For each hole to etch, a small portion of the sample surface around the corresponding macropore opening is scanned and visualized. The beam is subsequently positioned at the center of the macropore opening according to the scanned image. The use of the macropore openings to guide the etching allows the positioning of the ion beam with needed precision over large areas, as far as the sample surface remains sufficiently flat. The probe ion current used for both imaging and etching is about 30 pA, at the energy of 25 keV. The final ion current spot is focused to about 0.1 \( \mu \text{m} \). In order to drill larger holes the beam is scanned around its central position during the etching.
In order to maintain the beam positioning precision, it is necessary to reduce the sample surface damage due to the imaging and to the successive hole etching. Two etching sections, of a parallelogram shape, are defined on the sample surface. They are shown as A and B in the figure 2. The macropore openings in each section are etched only once, and in only one direction (shown with arrows). The etching direction in A forms an angle of 51.3° with that in B. The FIB etched holes cross each other and the macropores in the depth of the sample. The ion beam exits the sample side through the zone C. The Yablonovite-like structure is thus obtained inside the sample volume. In order to measure the optical response of the structure an optical interface is prepared by consequent FIB milling.

Figure 2. A sample after etching and milling. The pore openings in the sections A and B are etched. The holes cross each other in the depth of the sample and the beam exits through the zone C.

The exploded view of an area in the zone C is shown in figure 3 (left). The photonic crystal is 5 periods deep and 25 by 25 periods wide. The diameter of the photo-electrochemically etched macropores is of 0.45 μm and that of the FIB etched holes is of 0.35 μm. The distance between the "atoms" of the photonic crystal is of 0.75 μm. The precision of the FIB etch is high enough for the FIB etched holes to cross inside a sub-micrometer macropore after having run through approximately 25 photonic crystal periods (figure 3 right).

Figure 3. FIB etched holes on the sample side where the beam exits. An amplified view is displayed on the left.
The optical reflection spectra of the samples were obtained using a Fourier Transform Infrared Spectrometer coupled to an infrared achromatic microscope. A wide band thermal source and a liquid nitrogen cooled mercury-cadmium-telluride photo-detector allowed measurements at wavelengths from 1.5 μm to 15 μm. Since the measured structure is shape birefringent, the measurements were performed in a polarized light. One polarization is chosen to be parallel to the bisector of the angle formed by the two FIB etching directions (hereafter, polarization A). The other one is perpendicular to the polarisation A (hereafter, polarization B).

Figure 4 shows the measured spectra of the Yablonovite-like structure for the two polarizations (bottom), along with the calculated reflection (top). The calculations were performed for 5 layers of “atoms” using the finite-element electromagnetic simulator HFSS from Hewlett-Packard. The reflection maxima (one for each polarization) result from photonic bandgaps and appear in the reflection spectra close to 3 μm. A very good agreement between the two diagrams is observed concerning the reflection peak positions and the contrasts between the peak maximum and the pedestal. The bandgap positions were also well reproduced by plane-wave method calculations [10]. The discrepancy between the absolute reflection values can essentially be attributed to the optical diffraction losses inside the measuring system due to the small sample size (comparable to the wavelengths in the measurements).

**CONCLUSIONS**

In conclusion, we fabricated three-dimensional submicrometer Yablonovite-like photonic crystals directly in crystalline silicon. The structures are obtained by successive photo-electrochemical and FIB etchings of a silicon substrate. Submicrometer-period photonic crystals show the presence of the photonic bandgaps in agreement with the calculations. Further reduction in size as well as the fabrication of true Yablonovite photonic structures is possible using the described technology.
REFERENCES

Accurate Dry Etching with Fluorinated Gas for Two-dimensional Si Photonic Crystal

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ABSTRACT

Anisotropic Si dry etching is usually carried out with chlorinated gases for electronic devices such as Si-LSIs. We had another look at Si dry etching with fluorinated gases in order to obtain an ideal air hole for two-dimensional Si photonic crystal. We simulated vertical Si etching, and showed the possibility that single crystal Si can be etched vertically with high selectivity to the etching mask using fluorinated gases. We investigated ECR etching with an SF6-CF4 mixture, and vertical Si etching was achieved at room temperature. High Si/Ni selectivity above 100 was also obtained. Two-dimensional Si photonic crystal with a photonic band gap between 1.25 and 1.51 μm was produced using SF6-CF4 ECR plasma and a thin Ni mask.

INTRODUCTION

Silicon (Si) is attracting much attention as a photonic material because of its transparency and high dielectric constant in optical telecommunication wavelength regions [1]. One of the interesting applications is Si photonic crystal (Si-PhC) [2]. Recently, the observation of a clear photonic band gap (PBG) was reported for two-dimensional (2-D) Si PhC using silicon-on-insulator (SOI) substrate [3]. When a 2-D Si-PhC is fabricated, air holes with less than submicrometer diameters need to be periodically and densely arrayed in single-crystal Si (sc-Si). The processing technologies (lithography and dry etching) developed for electronic devices such as Si-LSIs are applicable. In the dry etching of Si-LSI fabrication, chlorinated gases are usually used for anisotropic sc-Si etching with a resist or SiO2 mask. However, in the fabrication of PhC for optical devices, it is ideally required that the periodicity and diameter of air holes be accurate, and the Si sidewall should be vertical and smooth.

In this study, we investigated Si dry etching characteristics with chlorinated gases and fluorinated gases in order to satisfy the above requirements for PhC fabrication. We also investigated a metal etching mask which makes Si-PhC fabrication accurate and simple.

EXPERIMENT

We simulated vertical Si etching with chlorinated and fluorinated gases. We used a topography simulator in which etched surface velocity against ion incidence angle (θ) is expressed as [4]

\[ V(\theta) = A + B \cos(\theta) + C \sin^2(\theta) \cos(\theta) + D \cos^3(\phi) \cos(\phi-\theta) d\phi + E \cos(\phi-\theta) d\phi. \]  (1)

Here, parameters A, B, and C are the contribution of isotropic etching due to radicals, vertical
etching due to ions, and sputtering, respectively. The fourth term with parameter D deals with anisotropic reactions due to particles with various incident angles. The fifth term with parameter E deals with indirect reactions due to particles reflected and desorbed from other surfaces. This simulator can accurately describe the reaction at the sloped surfaces near the pattern sidewall. We characterized the vertical etching by comparing the magnitude of these parameters.

For Si etching, we employed an ECR plasma apparatus because it can generate high-density plasma at low gas pressure and enables anisotropic etching with the assistance of ion bombardment at low energy of about 20 eV [5]. The chlorinated gas was Cl₂. The fluorinated gas was SF₆, CF₄, or a mixture of SF₆ and CF₄ [6]. Ni was patterned as the etching mask by the lift-off method, including electron beam writing and Ni evaporation [7].

Figure 1 shows sample structures. A SOI wafer with 0.2-μm-thick sc-Si and 3-μm-thick SiC was prepared. Si-PhC was formed at the center of a ridge Si waveguide. The transmission characteristics were measured by introducing light with wavelengths between 1.25 and 1.70 μm.

RESULTS AND DISCUSSION

Etched profile simulation of single crystal Si

Figure 2 shows typical sc-Si etched shapes with Cl₂ and SF₆. Etching with chlorinated gases usually produces a tapered shape without undercutting, and etching with fluorinated gases usually produces undercutting. From these shapes, we simulated vertical etching with both gases.

Figure 3 shows the simulation results. In the case of chlorinated gas, the fitting between SEM viewgraph and simulation profile can be done for the tapered shape by optimizing parameter B and C. The result that C is negative is important; it is the origin of the tapered shape in sc-Si etching with chlorinated gas. Positive C is related to normal sputtering, which causes preferential etching at the sloped surface. Negative C (negative sputtering) seems to refer to the phenomenon where chlorine radicals as etchant are removed by ion bombardment at the sloped Si surface. If we want to have sc-Si with a vertical etched shape, parameter B should be increased to 20, which is a very large value. In the case of fluorinated gas, the etched shape fitting can be done for undercutting by optimizing parameter A and B. Undercutting is caused by isotropic etching due to...