FABRICATION, TREATMENT, AND TESTING OF MATERIALS AND STRUCTURES

Fabrication Technology of Heterojunctions in the Lattice of a 2D Photonic Crystal Based on Macroporous Silicon

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Abstract—Design and fabrication technology of a microcavity structure based on a double heterojunction in macroporous silicon is suggested. The fabrication process of a strip of a 2D photonic crystal constituted by a finite number of lattice periods and the technique for defect formation by local opening of macropores on the substrate side, followed by filling of these macropores with a nematic liquid crystal, are considered.

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1. INTRODUCTION

Interest in photonic-crystal heterojunctions is a logical development of studies in the field of photonic crystals (PCs) [1]. Photonic-crystal heterojunctions can be used to create waveguides and photonic superlattices, widen the energy gap of PCs, etc. The most important achievements undoubtedly include cavities based on a double heterostructure, which have a record-breaking Q-factor [2, 3]. Microcavities of this kind are finding use in fabrication of low-threshold lasers, optical filters, quantum information systems, and sensors. Of particular interest is experimental fabrication of heterojunction device structures, most frequently fabricated by using 2D PCs formed by apertures in an insulator or a semiconductor. A double heterostructure (an analog of a quantum well in semiconductor heterostructures) is constituted by two PC1 regions between which is situated a thin PC2 region with an energy gap changed as compared with the outer layers. PC1 and PC2 are so designed that propagation of light with a prescribed frequency is possible in the central part of the structure, but is prohibited at its edges, so that photons are confined within the central PC2 region. If the frequency of incident light coincides with the natural frequency of the resulting well, then the electromagnetic wave resonantly tunnels through both barriers and the optical transmission of the whole structure reaches 100%.

In the first studies, PC2 was formed by changing the geometric lattice parameters of PC1 [2]. Somewhat later, it was suggested, instead, to change the refractive index by filling certain pores with various fluids [4, 5]. In the latter case, it is possible to use the

starting PC with the same period and diameter of apertures throughout the structure. Then, it becomes unnecessary to precisely vary the geometric lattice parameters. The method of pore filling with a micropipette controlled by a mechanical device with precise positioning used so far [6] seems overly complicated and labor consuming. The solution we suggest is based on photolithography and is devoid of the above shortcoming.

Photoelectrochemical etching of micropores in *n*-Si (100) is a convenient method for obtaining 2D PCs on silicon that is compatible with integrated circuit technology [7, 8]. This method has its own distinctive features and is sensitive to disruptions of the pattern periodicity because of the redistribution of the etching current density and the resulting change in the size and shape of pores. Therefore, the most convenient way to obtain heterojunction structures in the lattice of microporous silicon is to infiltrate certain pores with fluids. Use of a filler with a variable refractive index enables control over the resonance frequency of the allowed state in the energy gap of a PC.

The goal of our study was to develop design-related and technological solutions for fabrication of a microcavity based on a double heterostructure in a 2D silicon PC. The heterojunction is formed in a PC of finite size. For this purpose, windows that open pores in certain regions are etched out in the substrate of an oxidized macroporous sample. Some windows are used to remove the porous layers and form PC strips, and others are used to introduce a liquid crystal (LC) into the pores.

Fig. 1. Prototype 2D PC bar with locally opened pores filled with a liquid crystal (LC): (a) front view and (b) back side view.

2. DESIGN AND TECHNOLOGY

In accordance with published data, the problem of obtaining 2D PCs of finite size with vertical walls for input and output of light has been for the most part solved in two ways—by simultaneous etching of micropores and trenches [9, 10] and by fabrication of 3D structures from a uniform periodic array of macropores. In the latter case, the pattern is produced either by photolithography on the porous layer from the front side [11] or by local removal of the substrate [12]. In this study, we chose as the basis the variant of [12], which was updated and supplemented with a technique for defect formation. A prototype of the structure being developed is shown in Fig. 1.

Our experiments were performed on macroporous silicon with a triangular lattice of pores (lattice constant $a = 8 \mu\text{m}$). The process of microcavity fabrication is illustrated by Fig. 2. The technique is based on local removal of the substrate in an alkaline solvent (in the process, the inner surface of the pores is masked with a film of thermal oxide). Then the masking SiO_2 film is dissolved in opened pores with HF and, pene-

Fig. 2. Scheme of the technological process for fabrication of 2D PC bars with a double heterojunction: (1) macroporous silicon sample upon oxidation, (2) local pore opening for removal of the oxide from pores, (3) removal of silicon oxide from opened pores in the region of squares, (4) deposition of plasmochemical silicon oxide, (5) photolithography over the oxide on the back side of a sample to form a window for the defect, (6) dissolution of porous silicon in the squares and simultaneous pore opening in the region of the defect, (7) dissolution of silicon oxide, and (8) separation into chips and filling of opened pores with a liquid crystal.

trating into the macropores, the alkaline solvent dissolves Si-walls unprotected with the oxide layer. Thus, large square regions are etched through and form outer edges of a dumb-bell-shaped chip (Fig. 3). The narrow part of the chip is a PC strip, and the remaining parts improve the mechanical strength of the structure and enable experiments with a separate chip. Simultaneously with etching of squares, pores in the central part of the PC strip are opened through a preliminarily fabricated window in the oxide covering the substrate. The fluid poured into the resulting depression fills pores, thereby forming heterointerfaces between regions with air- and fluid-filled pores.

To fabricate the structures, we developed a set of four DR-1 photomasks: the first two are intended for fabrication of etch pits and register marks on both sides of a sample, and the third is intended for opening of large squares. The fourth mask enables opening of pores for a defect to be formed in the PC strip.

Fig. 3. (a) Principal directions in the reciprocal lattice of PC and (b) fragment of a photomask pattern, which illustrates how the microcavity structure is fabricated, back side view. (1) Bright squares are etched through, with chips remaining in the form of dumb-bells 2 (shown by gray), (3) opened pores to be filled with a fluid, and (4) scribing paths.

In the triangular lattice of the 2D PC, the principal directions Γ -M and Γ -K are mutually perpendicular [13] (Fig. 3a). The entire field of the photomask was divided into two zones, each containing elements differing in the width and orientation of bars along Γ -M and Γ -K. A fragment of the structure pattern is shown in Fig. 3.

The substrate thickness $h = t - l$, where t is the sample thickness and l is the pore depth (Fig. 4), is an important parameter in designing and fabricating our structures. Since the local pore opening is performed by anisotropic etching, it is necessary to take into account, when designing a photomask, that the depressions formed have sloping walls. These walls are formed by the (111) planes etched in alkaline etchants at the lowest rate [14]. The window w in the oxide mask should be larger than the width w_b of the depression at the bottom of the etched-out region: $w = w_b + 1.4h$. It is noteworthy that w_b , in turn, determines the number of opened pore rows.

3. SAMPLE FABRICATION

As the starting material for fabrication of macroporous silicon served n -Si (100) wafers with a diameter of 76 mm and resistivity of 15 Ω cm. The chosen resistivity of the starting material provides sta-

Fig. 4. Schematic representation illustrating local pore opening via anisotropic etching.

ble etching of macropores organized into a triangular lattice with a period of 8 μ m [7]. Square 30 $\times$ 30-mm samples were cut from wafers with a thickness of 380–400 μ m, and the thickness of these samples was reduced to 200 ± 5 μ m by grinding and polishing. An n^+ -contact was fabricated by ion implantation of phosphorus on the backside of the samples. Deep cylindrical macropores were produced by photoelectrochemical etching (PECE) with illumination of the sample backside [8]. The porous layer was created in a circular region with a diameter of 18 mm situated in the central part of a sample. The etchant was a 4% aqueous solution of HF with 5% ethanol. The process was carried out at a temperature of 25°C in a mode providing a constant diameter of pores along their depth. The initial current density was $j = 6$ mA cm⁻². A regular lattice of macropores was obtained owing to the preliminary formation of nucleation centers in the form of etch pits on the sample surface. The etch pits were produced by anisotropic etching in an aqueous solution of potassium hydroxide and isopropyl alcohol through a mask of a plasmochemical oxide SiO₂. The pattern was oriented so that seed rows were arranged along sides of a square sample that coincide with the $\langle 110 \rangle$ crystallographic directions.

3.1. Fabrication of a PC Bar

Let us first consider the process in which a PC bar is obtained (procedures 1–3 and 6–7 in Fig. 2). Macroporous silicon sample 20.05.10N with a substrate thickness $h = 4$ μ m was oxidized in water vapor (oxide thickness ~ 0.3 μ m) (procedure 1), and pores in square windows delimiting the PC bar were opened in the resulting oxide from the substrate side (procedure 2). In these windows, the inner surface of pores was freed from the masking oxide by etching in a dilute HF solu-

Fig. 5. SEM image of a fragment of a sample with a removed square (back side view with directions in the photonic crystal indicated).

tion (procedure 3). Attention should be given to the fact that, in the process, SiO_2 was etched off from both sides of a sample. Procedures 4 and 5 were skipped, and procedure 6, etching in an aqueous solution of KOH, followed immediately after procedure 3. To obtain smooth surfaces, isopropyl alcohol (IPA) was added to the 5% aqueous solution of KOH in a 1 : 2 volume ratio [15]. The etching was performed at a temperature of 70°C for 15 min. The removal of the

squares formed vertical walls of the PC bars. Figures 5 and 6 show images of the side walls of the resulting structures, furnished by scanning electron microscopy (SEM). Since the substrate of this sample is not protected by a silicon oxide layer and the substrate thickness is comparable with the thickness of walls between pores, pores were opened over the entire back side of structures in the course of squares' removal.

After the PECE, pores had slightly unequal diameters and cross-section shapes along their depths (the diameter became larger with increasing depth, and the shape became more square). Therefore, the walls between pores are thicker in the upper part (closer to the front side), with their remnants seen as projections, and are so thin in the lower part (closer to the backside) that they are etched through (Fig. 6d).

It can be seen in the figures that rectangular projections are formed on the wall of the structure along the Γ –M direction, and only remnants of silicon walls perpendicular to the interface are seen on the wall along Γ –K. The formation of such side walls is due to the anisotropic etching of macropores. The etching rate of the (110) plane in the KOH + IPA solution is lower than that of the (100) plane [15]. This results in the cross section of a round pore becoming square (Fig. 7a). The square sides are formed by slowly etching planes [110]. Attention should be given to the fact

Fig. 6. SEM images of PC edges upon the removal of the oxide from pores: bars with boundaries along (a, b) Γ –M and (c, d) Γ –K. A view from the back side of a sample.

Fig. 7. (a) SEM image of macroporous silicon treated in a KOH + IPA solution and (b) transformation of a round pore to a square-shaped one. The lengths of the arrows indicate the etching rates in different crystallographic directions.

that walls between square pores have different thicknesses for two perpendicular directions: they are thinner along Γ -K and thicker along Γ -M, which occurs because the lattice is triangular.

Figure 8 schematically shows how walls of macroporous silicon are dissolved in KOH + IPA and the structure edges are formed for two different directions of PC. As the etching time becomes longer, the thickness of the Si walls between the square pores decreases (Fig. 8b). The walls oriented along Γ -K are dissolved first, and the edges shown in Fig. 8c are formed. Upon more prolonged etching, the edges take the form shown in Fig. 8d.

3.2. Pore Opening in the Region of a Defect

Macroporous sample 24.05.10N had a thicker substrate than 20.05.10N and was protected by a plasmochemical oxide, which precluded pore opening over the entire surface when square regions were etched out in an alkali. In this sample, not only was a 160- μ m-wide PC bar was formed, pores were opened in a special window to enable their filling with a liquid filler (defect width 70 μ m). For this purpose, after the oxide

Fig. 8. Schematic representation of how the boundary is formed in the corner of a square being removed in dissolution of macroporous silicon: (a) initial structure upon removal of SiO_2 from pores in large squares; (b) initial stage of etching in an alkaline solution, in which the pore shape changes from round to square; (c) removal of a square; and (d) smoothing of structure borders in further etching and dissolution of the oxide.

Fig. 9. SEM image of the backside of chip no. 4. The PC bar and the defect are oriented along the Γ -K direction; the light is directed in spectroscopic studies in the perpendicular direction, along Γ -M.

was removed from pores in the region of large squares, the upper (front) and lower (back) sides of the sample were masked by deposition of an 0.42- μm -thick plasma-chemical oxide (procedure 4). A window for the defect was formed in this oxide by photolithography on the back side (stage 5). The etching of the sample in the alkaline solution simultaneously removed the large squares, with PC bars formed, and locally opened pores in the region of the defect. A chip fabricated in this way is shown in Fig. 9.

4. OPTICAL MEASUREMENTS

The spectral reflectance R was measured in the range 650–6500 cm^{-1} ($\lambda = 1.5$ –15 μm) with a Digilab FTS 6000 Fourier spectrophotometer coupled with a UMA 500 IR microscope (for more detail, see [16]). A glass plate coated with a thin film of gold served as reference with $R = 100\%$. The measurements were carried out with the 50×200 - μm rectangular aperture of the microscope, with a resolution of 8 cm^{-1} , and a polarizer placed before an MCT detector cooled with liquid nitrogen. The TM polarization corresponds to the light-wave electric-field vector directed along the axis of pores, and the TE polarization corresponds to the vector $\mathbf{E}$ directed perpendicularly to the pore axis (i.e., lying in the plane of the sample).

Figure 10 shows a reflectance spectrum of a PC bar with unfilled pores. The transmission of such wide chips could not be measured, because the output signal was too weak because of the heavy loss via scattering of light at the inner surface of pores. Estimates show that the fundamental energy gap of the PC obtained lies at $\nu \approx 350 \text{ cm}^{-1}$, outside the measurable spectral range, and the peaks in Fig. 10 lie at higher-order energy gaps. The shift of the interference bands for the TM polarization relative to those for the TE polarization is indicative of a positive anisotropy inherent in macroscopic silicon [17].

Fig. 10. Reflectance spectrum for chip no. 4 before pore filling with the liquid crystal.

5. INFILTRATION OF A LIQUID CRYSTAL

For infiltration of pores in the region of the defect, we chose an E7 nematic liquid crystal [18]. This LC has a high anisotropy of the refractive index in the IR spectral range, $\Delta n = 0.2$ [19] and is convenient in that it has a mesophase at room temperature. The calculations performed in [20] showed that a defect produced by filling a single row of pores with E7 LC creates a discrete level in the lower photonic band gap. The spectral position of the defect mode shifts as the refractive index of LC varies upon changing the director orientation. The shift of the resonance peak may be as large as $\Delta\nu/\nu = 4.7\%$.

For infiltration of opened macropores, a sample was placed with its frontal side down over the aperture in the table of the optical microscope, so that it could be displaced with micrometer screws in three dimensions. A drop of LC was deposited onto the chip surface near the place in which LC was to be introduced. The thin end of a feeder was dipped into this drop, and LC captured by the feeder was transferred into the depression of the defect with opened pores. Depending on the amount of transferred LC and on the volume of filled pores, this procedure was repeated several times. The LC was retained in through pores by the surface tension forces. The filling was monitored using the image of the front side. Figure 11 shows images of the front side of samples with locally filled pores, on which even edges of the heterointerfaces can be seen.

Using this technique, we filled pores in chip no. 4. The reflectance spectrum of this chip upon infiltration of LC is shown in Fig. 12. It can be seen that the absolute value of the reflectance somewhat decreased upon the infiltration. The spectrum shows bands characteristic of LC at $\nu = 817$, 1606, and 2226 cm^{-1} and groups of bands in the range 2780–3013 cm^{-1} . The bands at 1606 and 2226 cm^{-1} are associated with vibrations

Fig. 11. Micrographs of the front side of samples with pores filled with the liquid crystal through the window in the substrate: (a) broad window and (b) a single row of pores along different directions in the PC lattice.

Fig. 12. Reflectance spectrum for chip no. 4 with the defect filled with E7 liquid crystal.

directed along the axis of the LC director (or along the long axis of rodlike LC molecules), whereas bands in the region of the complex band at 817 cm^{-1} and a group of bands centered at $\sim 2910\text{ cm}^{-1}$ are related to vibrations directed perpendicularly to the long axis of the molecule [19, 21]. It can be seen that the depth of the bands at 1606 and 2226 cm^{-1} is smaller for the TM polarization, compared with the TE polarization, whereas, for the bands at 817 and $\sim 2970\text{ cm}^{-1}$, the situation is opposite. Hence there follows the conclusion that there exists a preferable orientation of the LC director along the axis of micropores. This conclusion agrees with the previously observed behavior of rodlike LCs (including E7 LC) in macroporous silicon [22, 23]. The degree of orientational ordering found for chip no. 4 from the dichroic relation [24] is low: $s = 0.15$.

6. CONCLUSIONS

Thus, a technology was suggested for fabrication of a microcavity structure based on a 2D photonic crystal with a double heterojunction. Implementation of the design-related and technological solutions was demonstrated for structures fabricated from macroporous silicon with a pore lattice period of $8\text{ }\mu\text{m}$. The penetration of the liquid crystal into pores of the defect was confirmed by optical microscopy and polarization spectrometry. A study of the optical properties of the microcavity based on the heterostructure obtained requires either a wider spectral range of studies or application of the developed technique to fabrication of photonic-crystal structures with a smaller period.

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