Optical properties of mesoporous photonic crystals, filled with dielectrics, ferroelectrics and piezoelectrics*

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At present, it is very important to create new types of mirrors, nonlinear light frequency transformers and optical filters with controlled optical properties. In this connection, it is of great interest to study photonic crystals. Their dielectric permittivity varies periodically in space with a period permitting Bragg diffraction of light. In this paper, we have investigated the optical properties of mesoporous three-dimensional (3D) opal-type and one-dimensional (1D) anodic alumina photonic crystals, filled with different dielectrics, ferroelectrics and piezoelectrics. We have compared the optical properties of initial mesoporous photonic crystals and filled with different substances. The possibility of mesoporous photonic crystals using selective narrow-band light filters in Raman scattering experiments and nonlinear mirrors has been analyzed. The electromagnetic field enhancing in the case of exciting light frequency close to the stop band edges has been established. The optical harmonics and subharmonics generation in mesoporous crystals, filled with ferroelectrics and piezoelectrics was proposed.

Keywords: Optical properties; photonic crystal; dielectrics; optical filters; optical harmonics.

1. Introduction

Now there is an important task of composite dielectric media with controllable dielectric and optical properties creating. Mesoporous photonic crystals are convenient objects for resolving such task.^{1–3}

Mesoporous photonic crystals are space ordered micro-structure with lattice constant, comparable to the wave length of visible or near infrared electromagnetic emission. The known example of three-dimensional (3D) photonic crystals is the mesoporous opal matrices, constructed from highly packed amorphous silica nanoglobules with the same diameters at the range 200–400 nm.^{4–9}

Recently, the new technique has been developed for production of mesoporous one-dimensional (1D) photonic crystals by means of electrochemical etching of alumina.^{10–12} The lattice constant of obtained mesoporous 1D alumina photonic crystals depends on the etching regime and may vary in the range of 100–500 nm.

Mesoporous 3D or 1D photonic crystals may be saturated with different dielectrics, ferroelectrics and piezoelectrics.^{13,14} Thus, there is the opportunity of new kind materials obtaining, which dielectric and optical properties may be monitored by means of changing of photonic crystals lattice constant, the concentration of entered substances, etc.

The paper reports on the results of experimental studies of the optical properties of mesoporous opal matrices and 1D anodic alumina photonic crystals. The generation of harmonics, subharmonics and the lowering of the threshold of stimulated Raman scattering, substances introduced into the pores of photonic crystals have been specifically studied. The influence of the concentration of solutions introduced into the pores of photonic crystals on its optical properties was determined. The possibilities of such materials usage in laser science and spectroscopy were analyzed.

Initial samples for investigations were artificial amorphous quartz opals and photonic-crystalline films of anodic alumina.

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Fig. 1. Photographs of initial artificial opals samples for investigations.⁴

Fig. 2. The shape of opal-type photonic crystals (111) surface, obtained with the help of electronic microscope.

2. Structure of Opal Matrices

Figure 1 demonstrates the photographs of artificial opals. Electronic photographs of the surface (111) of artificial opals are shown in Fig. 2. Opal matrices consist of spherical nanoparticles with diameters from 200 to 600 nm. The volume fraction of cavities in artificial opals can reach 26% (for point contacts between globules of amorphous SiO_2 balls).⁴ Introduction of components such as nanoparticles in the pores of photonic crystals can change their optical properties.

The samples can be filled with insulators due to the impregnation of pure opals using hydrophilic liquids and saturated solutions of inorganic and organic salts. The lattice of the amorphous silica nanoglobules with the pores that contain insulators and conductors is formed in artificial opals due to technological procedures (drying, annealing, etc.).

3. Structure of 1D Alumina Photonic Crystals

An alumina photonic crystalline film is transparent in the visible spectral region in the case of frontal observation

(a)

(b)

Fig. 3. Photonic crystalline film of anodic alumina observed (a) from the front and at an angle of (b) 40° .¹²

(Fig. 3(a)) and changes in color (iridescent), when the angle of observation is changed (Fig. 3(b)).

The fundamental principles of the technology for the manufacturing of mesoporous anodic alumina photonic crystal with electrochemical etching are detailed in Refs. 10 and 15. The essence of this technology consists in alternating the degree of etching for thin conducting film layers during their anodization in an electrolyte. This results in the formation of a “binary” 1D photonic crystal constructed from a great number of double layers with a different degree of porosity. This technology was used in this work as the basis for the synthesis of 1D alumina photonic crystalline film samples with different lattice constants within the range 350–450 nm.

To study the parameters of samples, their electron microscope images were taken with a scanning electron microscope (Figs. 4 and 5). The microscopic structure of the frontal surface of a film for two variants of scanning-microscope

Fig. 4. Electron microscope images obtained for the surface film layer by scanning at a scale interval of (a) 0.5 and (b) 0.1 μm .¹²

Fig. 5. Electronic photographs obtained for the lateral sample surface by scanning at a scale interval of (a) 5 and (b) 0.1 μm .¹²

resolution is shown in Fig. 4. Frontal surface analysis resulted in a pore diameter of $d = 55\text{--}85\text{ nm}$ and a porosity of $\delta = 0.45$ for the surface layer of the sample. The porosity was determined via pixel analysis of the monochromatic image of the frontal surface of the studied film.

The electron microscope images of the lateral surface of the sample in Fig. 5 allowed us to determine the thickness and lattice constant of the studied photonic crystal: $H = 77\text{ }\mu\text{m}$ (Fig. 5(a)) and $a = 386\text{ nm}$ (Fig. 5(b)). Hence, the considered photonic crystalline film amounts to nearly 200 periods or 400 layers.

4. Experimental Schemes of Photonic Crystals Optical Properties Investigations

The principal scheme of the setup for the study of reflection spectra of photonic crystal at near-zero incidence angle is shown in Fig. 6. The photonic crystal 1 located at the center of an aluminum plate 2 is fixed to a fluoroplastic holder 3. Electromagnetic radiation is directed onto the sample through optical fiber probe 4. Halogen and xenon lamps, a “white” light-emitting diode, and a deuterium lamp are used as the

Fig. 6. Setup for recording the spectra of reflection from the surface of an alumina photonic crystal at near-zero incidence angle: (1) photonic crystal, (2) aluminum plate, (3) fluoroplastic holder, (4) optical-fiber probe, (5) quartz light guides, (6) radiation source, (7) minispectrometer with a multielement receiver, (8) computer.¹²

Fig. 7. Diagram of the apparatus for recording the transmission spectra of radiation through a photonic crystal; 1 — radiation source, 2 — lens, 3 — monochromator, 4 — photonic crystal, 5 — detector, 6 — computer.

broadband radiation source 6. The light from the radiation source propagates through a quartz light guide 5 with a diameter of $100\ \mu\text{m}$ and is incident onto the surface of the photonic crystal 1 in the near-normal direction. The reflected radiation is incident on the second quartz light guide, where it is analyzed with minispectrometer 7 and computer 8. The spectral range of the minispectrometer is 200–1000 nm at a resolution of 1 nm. The exposure times during spectral recording varied within a range of 0.1–100 ms.

Spectra of transmission of electromagnetic radiation by photonic crystals were obtained. The scheme of the experimental setup is shown in Fig. 7. The light from the broadband radiation source 1 passed through the collecting lens 2. The radiation from the lens was focused on the input slit of the monochromator 3. After the monochromator, the radiation with a fixed wavelength hit the lens 2. The monochromatic radiation in the form of a parallel beam collimated by the lens 2 was directed to a photonic crystal 4. The signal detected by the detector 5 was analyzed digitally in the form of a transmission spectrum by the computer 6.

Figure 8(a) demonstrates the layouts of the setups for the observation of the Stimulated Raman Scattering (SRS) of crystalline powders. For the observation of the stimulated Raman spectra of liquids infiltrating into pores of the photonic crystal was used the scheme shown in Fig. 8(b).

Stimulated Raman scattering was excited by the second optical harmonic (532 nm) of a YAG:Nd³⁺ laser 1, which was generated in the form of pulses with a duration of 10 ns, an energy of 7 mJ, and a repetition frequency of 10 Hz.

As Fig. 8(a) shows, that through the mirror 2, the laser beam was focused by the lens 3 to the sample 5 and then filtered in the photonic crystal 8 and entered the input of a spectrometer 10 with a multi-element receiver. Backward Stimulated Raman Scattering after mirror 2 fell to the second spectrometer 12. The computers 11 and 13 were designed for digital processing and accumulation of spectra.

In the study of SRS in liquids introduced into pores of the photonic crystal, pump laser radiation was focused by lens 2 on sample 3. The spectrum of radiation reflected from the (111) surface of the opal matrices was recorded by means of Ocean Optics spectrometer 5 and computer 6. The measurements were performed for various angles of incidence of

(a)

(b)

Fig. 8. Layouts of the experimental setups for the excitation of stimulated Raman scattering: (a) — in crystalline powders: 1 — laser; 2 — mirror; 3, 7, 9 — lens; 4 — holder; 5 — sample; 6 — windows; 8 — photonic crystal, 10, 12 — minispectrometers; 11, 13 — computers; (b) — in liquids introduced into pores of the photon crystal: (1) laser, (2) lens focusing radiation on the sample, (3) sample, (4) lens focusing reflected radiation on the slit of the spectrometer, (5) spectrometer, and (6) computer.¹⁶

radiation on the sample with the use of lenses 2 with different focal lengths (200 and 300 mm). The diameter of the spot of laser radiation on the surface of the sample was ~ 0.1 and $0.15\ \text{mm}$, respectively. At different (10° – 70°) angles θ (see Fig. 8(b)) incidence of radiation on the surface of opal matrices (both the opal matrices infiltrated by carbon disulfide or benzene), we recorded the SRS spectra. With an increase in the angle of incidence θ of pump radiation on the (111) surface of the opal matrices, the spectral position of the stop band was shifted toward short wavelengths. At a certain angle of incidence θ_s , this position approached the frequency of pump radiation. In this case, we have observed dramatic enhance of SRS spectra intensity.

Fig. 9. Schematic of the experimental setup: (1) femtosecond laser station PHAROS 4W, (2) deflecting mirror, (3) lens, (4) photonic crystal, (5) holder, (6) post, (7) light guide, (8) spectrometer, and (9) computer.¹⁷

The scheme of the used experimental setup for studying of the secondary emission spectra of a photonic crystal under action of high-power density ultrashort laser pulses on the crystal surface is shown in Fig. 9. Yb:KGW laser with a wavelength of 1026.0 nm was used as an exciting radiation source. At a pulse duration of 250 fs and a pulse repetition rate of 200 kHz, the peak radiation power of the fundamental harmonic at the femtosecond laser output was 0.7 GW. Due to laser radiation focusing to the sample surface, the intensity of 1.0 TW/cm^2 was reached. Secondary radiation from the sample was collected by a fiber probe and directed to a spectrometer based on a diffraction grating, which allowed measurements of spectral characteristics with a resolution not worse than 1.0 nm in an operating spectral range from 190.0 to 1000.0 nm. As samples to be studied, synthetic opal matrices structured as a close-packed cubic lattice of silica balls (globules) 220, 240, 260, and 290 nm in diameter were chosen. Pores of opal matrices with a globule diameter of 240 nm were filled with ferroelectrics sodium nitrite and barium titanate.

5. Results of Experimental Investigations

Figure 10 demonstrates the reflection and transmission spectra, respectively, of original opals (in the absence of the dielectric filling), obtained with experimental setup, shown in Figs. 6 and 7. When the globule diameter increases, the reflection spectra, corresponding to the band gap position, are red shifted. A similar effect is observed for the opals filled with different dielectric substances.

As a result of the performed experiments, transmittance spectra of broadband radiation of a xenon lamp of mesoporous 1D alumina photonic crystalline film were obtained. Figure 11(a) shows the transmission spectrum $T(\lambda)$ of the investigated photonic crystalline film in the wavelength range

Fig. 10. Spectra for the opal surface: (a) dependencies of the band gap position on the the globule diameter; (b) reflection and transmission spectra for the opal surface.

300–2000 nm. Figure 11(b) illustrates the corresponding reflection spectrum obtained by using the relation $R(\lambda) = 1 - T(\lambda)$ (dotted curve) and with the help of setup, shown in Fig. 6. As can be seen from Fig. 11(a), there are sharp minima in the transmission spectra of the electromagnetic radiation of the xenon lamp from the crystalline film of a 1D photonic crystal in the region of the first, second and third stop bands. Such minima correspond to wavelengths: 1070, 535, and 364 nm. Accordingly, in the reflection spectra (see Fig. 11(b)), we have observed the intense maxima due to the presence of stop bands in the spectrum of the photonic crystalline film under study.

Figure 12 shows the variation of the spectral distribution of radiation in the SRS spectra under the variation of the angle θ for opal matrices infiltrated by benzene and carbon disulfide. As is seen in Figs. 12(a) and 12(b), at certain (critical) angles of incidence θ_s (40° for benzene and 60° for carbon disulfide), the intensity of SRS increases sharply. In this case, several Stokes satellites and one anti-Stokes satellite appear in the spectrum.

The maximum intensities of SRS spectra were observed in the angular range of $|\Delta\theta_s| = 2^\circ$. The intensity of SRS spectra decreased sharply at angles different from the critical angle θ_s . The threshold pump intensity corresponding to the appearance of the first Stokes component in the radiation

Fig. 11. (a) Transmission and (b) reflection spectra of a mesoporous 1D alumina-based photonic crystal sample: the dashed curve was plotted on the basis of transmission data, and the solid curve was obtained by the scheme shown in Fig. 6.¹²

spectrum in both crystals at an angle of θ s was 0.09 GW/cm^2 in benzene and 0.07 GW/cm^2 in carbon disulfide. The divergence of the beam of the first Stokes component of stimulated Raman scattering was close to the divergence of pump radiation. The maximum energy efficiency of the transformation of pump radiation to radiation stimulated Raman scattering was 4% for benzene at an angle of 40° and was 9% for carbon disulfide at an angle of 60° .

Figure 13 shows (lines 1) the SRS spectra obtained at the critical angle of reflection for the opal matrices infiltrated by (a) benzene and (b) carbon disulfide in comparison with (lines 2) the corresponding reflection spectra of broadband radiation from the (111) surface of the opal matrices under study. According to these figures, the line of pump radiation is in the region of the corresponding stop band (Fig. 13(b)) or near its edge (Fig. 13(a)). Furthermore, the first Stokes component for benzene and the anti-Stokes component for carbon disulfide are located directly in the region of the corresponding stop bands. According to the theory,¹⁸ an anomalous increase in the spectral density of the effective electromagnetic field at the frequency of pump radiation and at the frequencies of SRS should be expected in these cases.

Fig. 12. Stimulated Raman spectra in opal matrices filled with (a) benzene and (b) carbon disulfide at various angles θ of mirror reflection from the (111) surface. The pump intensity was 0.12 and 0.14 GW/cm^2 for benzene and carbon disulfide, respectively.¹⁶

This in turn should increase the gain of generation of stimulated Raman scattering and, correspondingly, reduce the threshold of SRS observation.

Figure 14 shows the secondary emission spectra occurring near the surface of the initial (filled with air) opal matrices upon excitation by ultrashort (250 fs) pulses of the laser radiation focused on the (111) surface of this crystal.

As seen in Fig. 14, the secondary emission spectra of photonic crystals contain sharp peaks corresponding to the third optical harmonic with a wavelength of 342 nm and the bands corresponding to plasma resulting from intense laser radiation. We note that the second optical harmonic (513 nm) in the spectrum under consideration does not appear according to the selection rules for centrosymmetric structures. A similar spectrum was also recorded for air (curve 8 in Fig. 14). This spectrum contains sharp maxima (513 and 342 nm) corresponding to the second and third optical harmonics. We note that sample degradation was not observed during irradiation of the photonic crystal surface for several hours. This is explained by the transparency of used samples in the visible spectral region and by the low energy of each laser pulse ($5 \mu\text{J}$).

Fig. 13. (Lines 1) Stimulated Raman spectra in the opal matrices infiltrated by (a) benzene and (b) carbon disulfide in comparison with (lines 2) mirror reflection spectra of broadband radiation from the surface of the corresponding opal matrices at the critical angle of mirror reflection θ_s (40° for benzene and 60° for carbon disulfide).¹⁷

Fig. 14. Spectra of secondary emission occurring near the surface of the initial (filled with air) opal matrices with a globule diameter of 260 nm, upon excitation by laser radiation with a wavelength of 1026 nm, at average pumping powers of (1) 3.1, (2) 2.688, (3) 1.956, (4) 1.343, (5) 0.788, (6) 0.352, and (7) 0.093 W; (8) is the plasma spectrum.¹⁷

Fig. 15. Dependencies of the intensity of the third optical harmonic on the averaged pump power with wavelengths of (a) 1026 nm and (b) 513 nm for photonic crystals with various globule diameters: (1) 220, (2) 260, and (3) 290 nm.¹⁷

Figure 15 shows the dependencies of the intensity of the third optical harmonic on the pump power at a wavelength of 1026 nm (Fig. 15(a)) and 513 nm (Fig. 15(b)) for photonic crystals with various globule diameters. As seen in Fig. 15, the excitation efficiency of the third optical harmonic (see curves 1 and 3) for crystals with globule diameters of 220 and 290 nm significantly exceeds those at pumping with a wavelength of 1026 nm. This can be explained by the fact that the exciting radiation wavelength (513 nm) is close to the spectral position of stop bands in these photonic crystals (450 and 600 nm). The mechanism of the formation of enhanced generation of the third harmonic can be associated with the effect of a decrease in the group velocity of pump radiation propagation in the photonic crystal when operating near the band gap and with the effect of structural light focusing in the photonic crystal bulk.^{19–22} Structural light focusing in the opal matrices *l* leads to the formation of the regions with high intensity of optical radiation. Exactly when the pump wavelength coincides with the photonic crystal band gap position, high-intensity optical field regions are localized at the crystal globule center; in this case, the intensity of formed optical field caustics feature a maximum intensity exceeding the

radiation intensity in a homogeneous quartz medium by an order of magnitude. Due to the formation of local high-intensity regions, high-efficiency medium pumping can be achieved.

Figure 16 shows the spectra of secondary emission observed upon excitation of the surface layer of opal matrices filled with sodium nitrite nanoparticles by a femtosecond pulse-periodic laser radiation (1026 nm). We can see a strong peak corresponding to the second optical harmonic (513 nm); the third optical harmonic (342 nm) under these conditions is almost absent in the photonic crystal spectrum, but is present in the plasma spectrum (see curve 8 in Fig. 16).

A comparison of Figs. 14 and 16 shows that the plasma emission spectrum in the samples filled with ferroelectric sodium nitrite is significantly weakened relative to initial (filled with air) opal. This can be explained by a significant increase in the breakdown threshold of the photonic crystal whose pores are filled with ferroelectric.

Figure 17 shows the dependence of secondary emission spectra of opal matrices filled with barium titanate on the pump power. In this case, the maximum conversion efficiency to the second optical harmonic is about 0.2 percent. The high conversion efficiency of exciting radiation to the second optical harmonic is caused by the closeness of the spectral position (550 nm) of the stop band in the crystals under study to the second optical harmonic frequency (513 nm).^{23,24}

Figure 18 shows the dependencies of the second optical harmonic intensity in opal matrices filled with (a) sodium nitrite and (b) barium titanate on the pump power. We can see that this dependence is nonlinear and close to quadratic. In this case, the conversion efficiency to the second optical harmonic is about 0.5%.

Thus, it was shown that the secondary emission spectra obtained under the irradiation of the surface of opal matrices

Fig. 17. Secondary emission spectra of the photonic crystal with barium titanate upon excitation by femtosecond pulse-periodic laser radiation (1026 nm) at average pump powers of (1) 3.397, (2) 3.048, (3) 2.237, (4) 1.594, (5) 1.281, (6) 0.982, (7) 0.492, and (8) 0.066 W; (9) is the plasma spectrum.¹⁷

by intense (about 1 TW/cm^2) ultrashort (250 fs) laser pulses in the “reflection mode” contain plasma bands and sharp peaks corresponding to the second (in the photonic crystal filled with ferroelectrics) and the third (in the photonic crystal filled with air) optical harmonics.

Fig. 16. Secondary emission spectra of the photonic crystal with sodium nitrite, excited with femtosecond pulse-periodic laser radiation (1026 nm) at average pump powers of (1) 3.567, (2) 3.072, (3) 2.237, and (4) 1.581 W; (8) is the plasma spectrum.¹⁷

Fig. 18. Dependence of the second optical harmonic (513 nm) intensity in opal matrices filled with (a) sodium nitrite and (b) barium titanate on the average intensity of exciting radiation (1026 nm).¹⁷

6. Theory of Photonic Crystals Optical Properties

The optical properties of the periodic structures were described using the Kronig–Penney model.²⁵ The dispersion dependence $k(\omega)$ and, correspondingly, the law $\omega(k)$ of the dispersion of electromagnetic waves in the considered structure of a 1D photonic crystal can be found from the relationship

$$\cos(ka) = \cos(k_1a_1) \cos(k_2a_2) - \frac{1}{2} \left(\frac{n_1}{n_2} + \frac{n_2}{n_1} \right) \sin(k_1a_1) \sin(k_2a_2). \quad (1)$$

Here, $a = a_1 + a_2$ is the lattice constant of a structure formed from alternating layers with thicknesses a_1 and a_2 and refractive indices n_1 and n_2 , $k_1 = n_1\omega/c$, $k_2 = n_2\omega/c$ and $c = 3 \times 10^8$ m/s is the speed of light in vacuum. Since $|\cos(ka)| \leq 1$, the spectrum of the dispersion curves $\omega(k)$ contains both allowed and forbidden photonic bands (band gaps) according to Eq. (1).

Knowing the dependence $k(\omega)$, it is possible to calculate the coefficients of reflection $R(\omega)$ and transmission $T(\omega)$, the refraction-index dispersion $n(\omega)$, and the spectral dependencies for the group velocity $V(\omega)$ of electromagnetic waves in a photonic crystal and the effective mass of photons $m_{\text{eff}}(\omega)$ by the relationships

$$R(\omega) = \left| \left(\frac{ck(\omega)}{\omega} - 1 \right) / \left(\frac{ck(\omega)}{\omega} + 1 \right) \right|^2. \quad (2)$$

$$T(\omega) = 1 - R(\omega). \quad (3)$$

$$|n(\omega)| = \frac{ck(\omega)}{\omega}. \quad (4)$$

$$V_g(\omega) = \frac{d\omega}{dk}. \quad (5)$$

$$m_{\text{eff}}(\omega) = \frac{\hbar}{\frac{d^2\omega}{dk^2}}. \quad (6)$$

To calculate the dependence $k(\omega)$ within a specified frequency range using Eq. (1), it is necessary to know the layer refraction-index dispersions $n_1(\lambda)$ and $n_2(\lambda)$ determined by the Cauchy formula

$$n_{1,2}(\lambda) = A_{1,2} + \frac{B_{1,2}}{\lambda^2}. \quad (7)$$

Here, $A_{1,2}$ and $B_{1,2}$ are coefficients, which are found by substituting $n_{1,2}(\lambda_m)$ calculated in the region of band gaps with numbers $m = 1, 3$ into Eq. (7).

The refractive index of the first film layer $n_1(\lambda_m)$ in the region of the m th band gap is calculated as

$$n_1(\lambda_m) = \sqrt{(1 - \delta)n_{\text{Al}_2\text{O}_3}^2(\lambda_m) + \delta n_{\text{air}}^2}. \quad (8)$$

Here, δ is the layer porosity, and $n_{\text{Al}_2\text{O}_3}(\lambda_m)$ and n_{air} are the refractive indices of an alumina single crystal and air, respectively. The width $\Delta\lambda_m$ and spectral location λ_m of the m th band gap are related with the layer refractive indices via

the known correlation²⁵

$$\frac{\Delta\lambda_m}{\lambda_m} = \frac{4}{\pi} \frac{n_1(\lambda_m) - n_2(\lambda_m)}{n_1(\lambda_m) + n_2(\lambda_m)}. \quad (9)$$

Using Eq. (9), we obtain the formula for the calculation of the refractive index $n_2(\lambda_m)$ of the second layer:

$$n_2(\lambda_m) = n_1(\lambda_m) \frac{4\lambda_m - \pi\Delta\lambda_m}{4\lambda_m + \pi\Delta\lambda_m}. \quad (10)$$

The effective refractive index of a sample is related to the layer refractive indices as follows:

$$n_{\text{eff}}(\lambda_m) = \sqrt{n_1^2(\lambda_m) \frac{a_1}{a} + n_2^2(\lambda_m) \frac{a_2}{a}}. \quad (11)$$

Here, the thicknesses of the layers $a_1 = a_2 = a/2$ are equal to each other according to the film manufacturing technology. The central location λ_m of the band gaps is specified by the known equation¹²:

$$m\lambda_m = 2a\sqrt{n_{\text{eff}}^2(\lambda_m) - \sin^2(\theta)}, \quad m = 1, 2, 3, \dots \quad (12)$$

where m is the number of the band gap, λ_m is the wavelength corresponding to the central location of the band gap, n_{eff} is the effective refractive index of a sample, and θ is the angle of radiation incidence onto the sample. In the case of normal incidence, Eq. (12) can be reduced to the form

$$m\lambda_m = 2an_{\text{eff}}(\lambda_m). \quad (13)$$

From Eq. (13), we obtain the lattice constant of the structure

$$a = \frac{m\lambda_m}{2n_{\text{eff}}(\lambda_m)}. \quad (14)$$

In this work, the lattice constant of the studied photonic crystal was calculated on the basis of Eqs. (8), (10), (11), and (14), substituting the first layer porosity δ determined from the electronic photo of the frontal surface of the sample λ_m and $\Delta\lambda_m$ and determined for the first band gap ($m = 1$) from the experimental reflection spectrum (Fig. 5(b)). Knowing the sample structure lattice constant a and the spectral location λ_m and width $\Delta\lambda_m$ of the band gap, it is possible to calculate the refractive indices $n_1(\lambda_m)$ and $n_2(\lambda_m)$ of adjacent layers for the m th band gap by the formulas obtained using Eqs. (9), (11), and (13) as

$$n_1(\lambda_m) = \frac{m\lambda_m}{2a\sqrt{\eta + (1 - \eta) \left(\frac{4\lambda_m - \pi\Delta\lambda_m}{4\lambda_m + \pi\Delta\lambda_m} \right)^2}}. \quad (15)$$

$$n_2(\lambda_m) = \frac{m\lambda_m}{2a\sqrt{\frac{16\pi\eta\lambda_m\Delta\lambda_m}{(4\lambda_m - \pi\Delta\lambda_m)^2} + 1}}. \quad (16)$$

Here $\eta = a_1/a$.

Knowing the sample structure lattice constant a and the number m and spectral location λ_m of the band gap, it is

possible to express and calculate the effective refractive index $n_{\text{eff}}(\lambda_m)$ of a sample in the region of the corresponding band gap by Eq. (13). Calculating the refractive index $n_1(\lambda_m)$ of the first layer by Eq. (8), we find the refractive index $n_2(\lambda_m)$ of the second layer by the formula derived from Eq. (11) as

$$n_2(\lambda_m) = \sqrt{\frac{a}{a_2} n_{\text{eff}}^2(\lambda_m) - \frac{a_1}{a_2} n_1^2(\lambda_m)}. \quad (17)$$

Substituting λ_m , $n_1(\lambda_m)$, and $n_2(\lambda_m)$ for the m th band gap into Eq. (9), it is possible to determine its width.

The calculation in this work was carried out within the frequency range $\Delta\omega(1.57-10.5) \times 10^{15} \text{ s}^{-1}$, which corresponds to the wavelength range of 180–1200 nm and includes the near infrared, visible and, partially, ultraviolet spectral regions. This spectral region accommodates six primary band gaps of the studied photonic crystal. Table 1 contains $a = a_{\text{th}}$, $n_{\text{eff}}(\lambda_m)$, $n_1(\lambda_m)$, $n_2(\lambda_m)$, and $\Delta\lambda_{\text{th}}$ calculated by Eqs. (8)–(17) and the corresponding experimentally measured values of the spectral location $\lambda_m = \lambda_{\text{exp}}$ and width $\Delta\lambda_{\text{exp}}$ of band gaps (from analysis of the transmission and reflection spectra in Fig. 11) and the lattice constant $a = a_{\text{exp}}$ (from analysis of the electronic photograph in Fig. 5) of the discussed photonic crystal.

Based on the data of Table 1 (for the refractive indices $n_1(\lambda_m)$, $n_2(\lambda_m)$ and $n_{\text{eff}}(\lambda_m)$, corresponding to the first band gap ($m = 1$) and the third band gap ($m = 3$)), the coefficients $A_{1,2}$, $B_{1,2}$ and A_{eff} , B_{eff} for the layer refractive indices $n_1(\lambda)$ and $n_2(\lambda)$ and the effective refractive index $n_{\text{eff}}(\lambda)$ were calculated from Eq. (7), and the dispersion curve of the effective refractive index $n_{\text{eff}}(\lambda)$ of the alumina photonic crystalline film was plotted (Fig. 19).

The numerical values of the alumina single crystal refractive index²⁶ used to calculate the refractive index of the first layer by Eq. (8) in the region from the first to the sixth band gap are given in Table 2.

The experimental (dotted line) and theoretical (solid) dependencies for the spectra of the transmission and reflection

Fig. 19. Dispersion of the effective refractive index n_{eff} of the studied photonic crystalline film.¹²

of broadband radiation from the photonic crystalline film surface are plotted in Figs. 20(a) and 20(b).

The theoretical dependencies were obtained from Eqs. (1)–(3) with allowance for the dispersion of refractive indices (Table 1 and Fig. 19). As a result, the locations λ_{th} and widths $\Delta\lambda_{\text{th}}$ of band gaps were determined from the calculated spectra and also presented in Table 1. As can be seen from Fig. 20(a) and Table 1, the experimentally determined locations λ_{exp} of band gaps are observed to be in good agreement with the theoretical values λ_{th} . At the same time, there is an appreciable difference between the calculated and experimental spectral curves for the second band gap. The performed calculations provide the possibility to determine the spectral locations of the fourth, fifth, and sixth band gaps lying in the ultraviolet region (Table 1).

Figure 21(a) illustrates the dispersion curve $\omega(k)$ calculated by Eqs. (1)–(6) with branches 1–7 located in the allowed photonic bands and also the dispersion dependence $\omega(k)$ for electromagnetic radiation in vacuum (dashed curve 8). The spectral dependencies of the refractive index $n(\lambda)$, the

Table 1. Experimental (exp) and theoretical (th) characteristics of a photonic crystal sample (m is the number of the band gap, λ_{exp} and λ_{th} are the spectral location of the center of the band gap, $\Delta\lambda_{\text{exp}}$ and $\Delta\lambda_{\text{th}}$ are the width of the band gap, $n_{\text{eff}}(\lambda_m)$, $n_1(\lambda_m)$ and $n_2(\lambda_m)$ are the effective refractive index and the refractive indices of layers, and a_{exp} and a_{th} are the structure lattice constant).

m	1	2	3	4	5	6
λ_{exp} , nm	1070	535	364	—	—	—
λ_{th} , nm	1072	539	365	279	228	196
$\Delta\lambda_{\text{exp}}$, nm	78	41	—	—	—	—
$\Delta\lambda_{\text{th}}$, nm	78	40	24	16	13	9
$n_1(\lambda_m)$	1.464	1.475	1.491	1.510	1.541	1.579
$n_2(\lambda_m)$	1.305	1.314	1.344	1.380	1.411	1.467
$n_{\text{eff}}(\lambda_m)$	1.387	1.397	1.415	1.446	1/477	1.524
a_{exp} , nm	386	386	386	386	386	386
a_{th} , nm	385.7	385.7	385.7	385.7	385.7	385.7

Table 2. Dependence of the refractive index n of an alumina Al_2O_3 single crystal on the wavelength λ .

λ , nm	n	λ , nm	n
193	1.929	458	1.778
213	1.889	488	1.775
222	1.875	515	1.773
226	1.870	532	1.772
244	1.851	590	1.768
248	1.847	633	1.766
257	1.839	670	1.764
266	1.833	694	1.763
280	1.824	755	1.761
308	1.811	780	1.761
325	1.805	800	1.760
337	1.801	820	1.760
351	1.797	980	1.756
355	1.796	1064	1.754
442	1.780	1320	1.750

Fig. 20. (a) Transmission and (b) reflection spectra obtained experimentally (dashed) and theoretically (solid) for a mesoporous 1D alumina-based photonic crystal sample.¹²

group velocity $V_g(\lambda)$, and the effective mass of photons $m_{\text{eff}}(\lambda)$ for the studied mesoporous photonic crystalline film are shown in Figs. 21(b)–21(d), respectively. The spectral locations of the first–six band gaps of the photonic crystal

structure (see Figs. 21(b)–21(d)) are marked with thin vertical lines. Point A ($\lambda_A = 922$ nm) corresponds to the intersection between curves 2 and 8. The refractive index of the structure at this point is $n(\omega_A) = -1$ (Figs. 21(a) and 21(b)). According to Eq. (2), the reflection index for point A is $R(\omega_A) = 0$.²⁷

The analysis of the dependence of the electromagnetic waves group velocity on the wavelength (Fig. 21(c)) shows that its value is close to zero at the boundaries between the photonic-band gaps. In this case, the efficiency of Raman and nonlinear optical processes sharply increases due to an abnormal increase in the density of photon states near the boundary of the band gap.²³ From the dependence of the effective mass of photons on the wavelength of incident radiation (Fig. 21(d)) it follows that the resting mass of photons can take both positive or negative values. The highest absolute values of the effective mass of photons correspond to the inflection points in the dispersion curves. The effective mass of photons at these points is several orders of magnitude higher than the mass of photons in the other spectral regions.

7. Applications of Photonic Crystals

Mesoporous photonic crystal has specific reflection spectrum with distinct stop band. Reflection coefficient in stop band area is very high (up to 99.9%). Such properties are usable in selective optical filters and for splitting of multimode laser emission lines (see Fig. 22).

Total reflection of chosen emission line can be realized in three different methods: it is possible to specify crystalline period while anodization (by changing current–time periods); stop band area may be moved by adjusting of angle of incidence/reflection; stop band area may be transformed²⁸ by injection of dielectric material inside photonic crystalline film pores.

Fig. 21. Dispersion dependencies of different characteristics of electromagnetic waves, propagating in photonic crystal; (a) — $\omega(k)$ (or $\lambda(k)$), branches 1–7 (solid line) correspond to photonic crystal, dotted line to $\omega = c_0 k$ — vacuum dispersion law; (b) — refractive index $n(\lambda)$, (c) the group velocity $V_g(\lambda)$, and (d) the photonic effective mass $m_{\text{eff}}(\lambda)$ for branches 1–7 of the dispersion curves $\omega(k)$; point A corresponds to the wavelength, for which the film refractive index $n(\lambda_A) = -1$.¹²

Fig. 21. (Continued)

Fig. 22. Scheme of laser lines division: 1 — multimode laser, 2 — photonic crystals, which reflect wavelengths of 532 and 1064 nm.

Another probable application is to enhance spontaneous and stimulated Raman scattering spectra intensity and essentially decrease exciting emission intensity in recording Raman spectra device. The last opportunity is illustrated by Fig. 23. In this case, the exciting emission is filtered by photonic crystal but Raman scattered ones pass through it. Sensor sensitivity may be improved because exciting line does not interrupt to measure Stokes and anti-Stokes components.

Fig. 23. Scheme of multi-mode to single-mode modification of laser: 1 — high reflection mirror, 2 — pump source, 3 — gain medium, 4 — diaphragms, 5 — photonic crystal, 6 — output mirror.

Mesoporous photonic crystals may be used as detectors of different molecular structures, embedded into voids of such crystals. Molecular structures are injected into photonic crystal pores by using different techniques: in form of wetting solution, in the form of alloy, etc. Raman scattering cross-section increases anomalously when spontaneous Raman scattering spectra are registered under conditions of an anomalous increase in the density of the corresponding photon states.¹⁶ When pores of photonic crystal are filled with SRS active media, it is possible to generate low-threshold SRS due to an anomalous increase in the density of photonic states¹⁶ in the case when the frequencies of the exciting or generated radiation fall on the edges of the corresponding stop bands. The value of the angle θ at which the Wulff-Bragg's condition is satisfied is given by the well-known relation (12).

Group light velocity is significantly reduced in this case (Fig. 21(c)). This leads to increase of photonic density of states resulting sharp enhance of the third optical harmonic generation in initial (without filling with some substances) photonic crystals. For mesoporous photonic crystals, filled by ferroelectrics or piezoelectrics, the enhancement of the second harmonics or subharmonics efficiency is realized.

Photonic crystals are usable for filtering of laser generation. On Fig. 22 radiation from multimode laser 1 is filtered by photonic crystal 2. One film reflects 532 nm part and transmits 266 and 1064 nm parts, another film reflects 1064 nm part and only 266 nm band is in output.

It is possible to modify the optical cavity construction to allow only one frequency mode generation. Such scheme is illustrated by Fig. 23. In this case, one of the cavity mirrors is replaced by photonic crystal 5. This crystal reflects only one frequency radiation (based on incident angle). So the single frequency emission will be in output although there are some other allowed modes of the cavity.

Compact analyzer for Raman spectroscopy may be constructed by using photonic crystals (Fig. 24). Radiation from

Fig. 24. Compact analyzer for Raman spectroscopy: 1 — sample, 2 — photonic crystal, 3 — substrate, 4 — lens, 5 — optical fibers, 6 — laser, 7 — refocusator, 8 — minispectrometer with multielement radiation receiver, 9 — computer.

laser 6 incidents at an angle θ on the sample 1. Sample is placed in the pores of photonic crystalline film 2. Lens 4 converge scattered radiation which goes to refocusator 7 via optical fiber 5. Another photonic crystalline film is placed inside the refocusator at an angle θ to the beam. Laser component is reflected by this photonic crystalline film while Stokes and anti-Stokes components pass through. Beside this, the investigated substance may be embedded into the pores of another mesoporous photonic crystal (see Fig. 24). By means of the angle θ incidence of exciting ray, we can realize the condition, when the frequency of laser emission is close to the stop band edges and corresponding spontaneous Raman cross-section enhancing.

Figure 25 illustrates the setup for generation of the second optical harmonics emission ($\lambda' = \lambda_0/2$) by using of mesoporous photonic crystal 3, filled with nonlinear optical substances (LiIO_3 , NaBrO_3 , BaTiO_3) and rotated by an angle θ to the beam (see Fig. 25) for coinciding of exciting line (λ_0) or the second harmonic emission ($\lambda' = \lambda_0/2$) wavelengths with the edges of the photonic stop band wavelength.

SRS analyzers scheme, by using mesoporous 1D anodic alumina crystalline films, filled in SRS active media, is shown in Fig. 26. The photonic crystalline film 3 reflects laser beam while SRS is generated inside in this mesoporous film. The same mesoporous photonic crystal 3, filled with Raman active media, is used both for SRS light amplification

Fig. 25. Setup for exciting laser wavelength doubling (the second optical generation) by photonic crystal: 1 — laser, 2 — lens, 3 — photonic crystalline film, embedded by nonlinear substance, 4 — spectrometer, 5 — computer.

Fig. 26. Stimulated Raman scattering with mesoporous photonic crystals, filled with Raman active medium: 1 — laser, 2 — converging lens, 3 — photonic crystalline film, 4 — spectrometer, 5 — computer.

and for laser beam filtration. In this case, laser beam incidents angle θ to the film surface should correspond to the photonic density of states increase.

In Fig. 27, the experimental scheme for subharmonic generation and for entangled photonic states investigations is illustrated. Photonic crystalline film, filled by ferroelectric or piezoelectric substance, is rotated at angle θ , improving

Fig. 27. Experimental setup for generation of entangled photonic states during two-photon subharmonic emission excitation: 1 — laser, 2, 4, 5 — lenses, 3, 6 — photonic crystals, embedded by nonlinear substance, 7 — spectrometers, 8 — computer.

phonic density of states enhancing at frequency ω_L of exciting light radiation or subharmonic frequency ($\omega_L/2$). It is convenient when wavelength of exciting light emission from laser 1 is close to the edge the second stop band of photonic crystal at wave vector $q = 0$. In this case, the efficiency of subharmonic generation increases and subharmonic waves with opposite wave vectors ($k_1 = -k_2$) may be detected with the help of two spectrometers 4.

8. Conclusion

Thus, the optical properties of mesoporous 3D opal-type and 1D anodic alumina photonic crystals have been investigated. It is shown that the high reflection coefficient in narrow stop band range may be realized in mesoporous photonic crystals, transparent for wide frequency region: ultraviolet, visible and infrared. Stop band spectral position and its width may be optimized with the help of lattice constant and effective refractive index changing. The introduction of Raman active material into the pores of the photonic crystal as an opportunity to enhance the spontaneous Raman scattering intensity and to reduce the SRS threshold. Such properties open the wide field for applications.^{29–34} One way for it is the filling of mesoporous photonic crystals void by different substances, including liquids, ferroelectrics, etc. Mesoporous photonic crystals may be explored as selective filters in laser resonators and in devices for registration of spontaneous and stimulated Raman scattering. Initial (nonfilled by some substance) mesoporous photonic crystals open the opportunity for effective third harmonic generation realizing. Mesoporous photonic crystals, embedded by nonlinear optics substances, may be used as nonlinear optic elements for the second harmonics and subharmonics generation and for observation of low threshold Stimulated Raman Scattering.

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