## Performance testing of log pile photonic crystal fast-fabricated by direct femtosecond laser writing

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Received March 10, 2008

Great efforts has been made on fabricating photonic crystals (PCs) with photonic band gaps (PBGs) during the past decade. Three-dimensional (3D) log pile PC was fabricated fast by direct femtosecond laser writing in ORMOCER. Qualitative analysis of the errors of PC was investigated using the Image Pro Plus. Surface qualities such as bending, distortion, and surface roughness were shown, and the band gap in the infrared wavelength region was observed. Meanwhile, the theory was experimentally verified that the center of PBG diminishes as the crystal lattice period reduces. Therefore, it is possible to fabricate PCs whose band gap range is from the near-infrared to visible wave band.

OCIS codes: 060.4510, 250.2080, 250.5300.

doi: 10.3788/COL20080611.0864.

Photonic crystal (PC), a new photoelectron device, has a periodical structure containing photonic band gap (PBG)<sup>[1-5]</sup>. PBG effect can induce useful optical properties, such as inhibition of spontaneous emission and photon localization $^{[1,2]}$ . Thereby, PCs can provide a novel platform for the realization of photonic circuits. During the past twenty years, it have made plenty of efforts on the design and fabrication of PCs since the realization of the first three-dimensional (3D) PC in  $1989^{[6]}$ . As for different PCs, many methods were developed and proved successfully, such as colloidal self-assembled method, ion etching method, electrochemistry etching method, and molecule biology assembled method<sup>[7-9]</sup>. However, these fabrication methods have some disadvantages. Precision machine drilling method is merely used to fabricate PCs whose band gap lies in microwave band. Etching method has complicated process to introduce defects into PC. Colloid self-assembled method has difficulty in controlling the fabricating process. Direct femtosecond laser writing, as a unique fabrication technique, is thought to be the most promising.

In the process of direct femtosecond laser writing, an electron transforms from the ground state to an excited state by the simultaneous absorption of two laser photons<sup>[10]</sup>. By focusing femtosecond laser into transparent material, high optical density is obtained in the focal region. Due to the Gauss distribution of intensity and materials' quadratic dependence on the optical intensity, one can make the intensity exceeds the threshold. Thus, free carrier plasma is generated within focal region. The plasma creates free radicals, which subsequently trigger the propagation of polymerization reaction. This produces nearly spherical solid regions in liquid photosensitive resin, while the unexposed portion remains liquid and is removed by washing. It is possible to reduce the polymerized size to  $\lambda^{3[11]}$  by optimizing the focus condition.

In 1992, Webb brought two-photon technique into microfabrication field<sup>[12]</sup>. Sun *et al.*<sup>[13]</sup> reported that 3D PC structures had been fabricated through two-photon-absorption photopolymerization of resin and significant

band-gap effects in the infrared wavelength region were observed. Kaneko et al.<sup>[14]</sup> demonstrated that diamondlattice PC structures with a submicron feature size had been realized, from which single-period attenuation up to 35% had been achieved. In 2004, Deubel *et al.*<sup>[15]</sup> reported the fabrication through direct femtosecond laser writing and detailed characterization of high quality large-scale face-centred-cubic (f.c.c.) layer-by-layer structures, with fundamental stop bands ranging from 1.3 to 1.7  $\mu \mathrm{m.}$  In 2006, Chutinan  $et~al.^{[16]}$  demonstrated the design and fabrication of 3 + 1 dimensional integrated optics with localized light in a PBG. In addition, lots of application researches of PC have been developed using direct femtosecond laser writing<sup>[17-20]</sup>. With more investigation, less linewidth can be obtained by direct femtosecond laser writing of photosensitive resin. Therefore, it seems to be more and more important to investigate the error and surface roughness that can not be neglected under very small linewidth conditions. In this paper, the direct femtosecond laser writing system and fabrication of 3D log pile  $PC^{[21]}$  were discussed; the optimum parameters were investigated to improve resolution. Qualitative analyses were investigated with the Image Pro Plus. Surface qualities such as bending. distortion, and surface roughness of the PC were shown. The transmission spectrum was measured with nearinfrared spectrogram. In addition, direct femtosecond laser writing technique was proved experimentally to be a fast method in fabricating PCs.

The experimental setup was depicted in Fig. 1. A pump source (Coherent) and Ti:sapphire femtosecond laser pulse generation (Institute of Physics of CAS) system were used as the irradiation sources. Pulse duration was 30 fs measured by the auto-correlation. The output with the wavelength of 800 nm, repetition rate of 82 MHz, and single pulse energy of 8 nJ, was collimated and directed to an inverted biological microscope (Olympus IX-71). The average power is around 560 mW. The whole fabrication process was monitored by a charge coupled device (CCD) camera-monitor set. The sample, a drop of liquid resin on a glass substrate, was mounted



Fig. 1. Experimental setup of direct femtosecond laser writing system.

on a holder attached to a 3D piezoelectric translation (PZT) stage. By the controlling software which can adjust the speed of 3D stage and the action of shutter simultaneously, the microstructure designed by CAD software could be converted into the instructions accepted by D230 controller, which drives the 3D stage. Therefore, the laser focus could move in the resin. The position of the stage was computer-controlled according to a preprogrammed code with an accuracy of 20 nm. The material was ORMOCER, which is a type of inorganic-organic hybrid polymers provided by Fraunhofer-Institute for Silicate Research (Germany). The mass ratio of photoinitiator was 1 wt.-%, and free radical critical concentration of photopolymerization is 0.25 wt.-%. From the absorption spectrum of the resin (between 350 and 405 nm), it can be concluded that the laser of 800 nm can be absorbed barely unless through two-photon photopolymerization.

The light intensity and the scan speed can influence the fabrication precision and mechanical property when the numerical aperture of objective, solidified threshold, and breakage threshold of photosensitive resin are constant. Mechanical strength of the structures was decided by the cross linking degree of monomer. In fabricating, each voxel was solidified in a very short time. Polymer existed mostly in the form of line chain and branch chain, and the rest was cross  $linking^{[22]}$ . As the exposure time and laser intensity increase, the proportion of monomer cross linking increases. Therefore, the bond strength and mechanical property were improved correspondingly. The laser intensity fluctuated greatly under low laser power, which highly influences the properties of solidified voxel. Nevertheless, excessive laser intensity will lead to biggish linewidth, even destroy the resin. In addition, scan speed is also an important factor in fabrication because fast scan speed can reduce the cost of time and money. We have shown a scan speed of 120  $\mu$ m/s is faster than the others<sup>[23,24]</sup>. Accordingly, taking account of the factors above, we used oil objective lens with numerical aperture of 1.35 and laser power 100 mW to fabricate PCs. Under these conditions, high efficiency and few defects can be achieved.

The PBG of 3D log pile (see Fig. 2) PC was determined by medium filling ratio and lattice constants<sup>[13]</sup>. The PC with different linewidth and medium filling ratios can be fabricated by adjusting laser power and scan speed. By changing the distance between adjacent



Fig. 2. Diagram of the 3D log pile PC.



Fig. 3. 3D log pile PC (left) and partial enlarged view (right).

rods and that of adjacent layers to alter lattice constant in controlling software, different photonic crystals can also be achieved. The connection between the rods of adjacent layers is important for high binding strength. When laser power and scan speed are constant, the axiallateral dimensions ratio<sup>[25]</sup> of solidified medium rods is k = l/d = 1.64n/NA, where n is refractive index of photosensitive resin. NA is numerical aperture of objective. Therefore, we can calculate the axial dimension according to the lateral dimension by selecting proper laser power, scan speed, and numerical aperture. In addition, binding strength between adjacent layers can also be improved by fabricating wall around the PC and selecting reasonable baking process. Figure 3 shows a 3D PC with 12 layers fabricated by direct femtosecond laser writing in photosensitive resin. It is consisted of one-dimensional rods with a stacking sequence that repeats itself every four layers with a repeat distance of 4.00  $\mu$ m. Within each layer, the axes of the rods are parallel to each other with a pitch of 1.5  $\mu$ m. The orientations of the axes are rotated by  $90^{\circ}$  between adjacent layers. Between every other layer, the rods are shifted relative to each other by 0.75  $\mu$ m. The diameter of the rods is 0.86  $\mu$ m. The resulting structure has a face centred- tetragonal (f.c.t.) lattice symmetry. This layered structure can also be derived by replacing the (110) chains of atoms in the diamond rod structure. In order to increase the viscidity of liquid resin and avoid the excursion of solidified voxels in fabrication, we baked the liquid resin in oven at 80 °C before experiment. Meanwhile, a massive wall around the PC was fabricated by the same technique to improve bond strength and avoid the shrinkage and distortion. In addition, the wall can also avoid bending during the cleaning. As a result, the bending and distortion during cleaning and the excursion of solidified voxels can be avoided effectively.

Surface quality of the PC was investigated, such as bending, distortion, and surface roughness. Figure 4 shows the line profile of the topside layer (along the a



Fig. 4. Line profile of topside PC (perpendicular to topside solidified resin).



Fig. 5. Line profile of one solidified resin rod (parallel to topside solidified resin).

direction in Fig. 2) depicted by Image Pro Plus. The x-axis presents the distance in the a direction, y-axis denotes laser intensity, and line profile reflects the surface quality. From the line profile in Fig. 4, we can see that light intensity peak values are on a line and half-peak width of each wave is equal. This indicates that there is no bending and distortion in the sample and the diameters of every rod are equal basically. Therefore, every layer has the same surface quality because of the high accuracy of 3D stage. A few peak values are odd and some line profiles are coarse, which are mostly attributed to the erosion of solidified resin in the cleaning processing. This instability of line profile is just a reflection of the solidified resin's surface roughness. Figure 5 is a line profile of a single solidified rod in upper layer (along the b direction in Fig. 2). This picture shows that the line profile has slight fluctuation, which reflects the surface roughness. The parameters include scan speed and laser power, and concentration of cleaning solution will influence the surface roughness and band gap characteristic. Thereby, problems concerning fabrication parameters and cleaning techniques mentioned above are being studied at present to improve the structure and optical property of PC.

Figure 6 shows a transmission spectrum measured by



Fig. 6. Transmission characteristics of 3D log pile PC.

Fourier infrared spectrometer. We can conclude that the center wavelength, transmission ratio, band width, relative width, and total decline of the PBG are 4.3  $\mu$ m, 55%, 130 nm, 0.0302, and 0.94 dB, respectively. The medium filling ratio is 56.5% according to the correlative experimental parameters. Considering refractive indices of resin and air are 1.538  $(n_{resin})$  and 1  $(n_{air})$  respectively, we can calculate the efficient refractive index,  $N_{\rm eff}$ , as  $N_{\rm eff} = n_{\rm resin} \times 56.5\% + n_{\rm air} \times (1 - 56.5\%) \approx 1.30$ . Measurement indicates that the band gap lies in midinfrared wave band, which can control the propagation of mid-infrared light of 4.3  $\mu$ m. Comparing with our former experimental results with a pitch of 2.0  $\mu$ m and band gap of 4.7  $\mu m^{[26]}$ , we experimentally verify that the centre of PBG diminishes as the crystal lattice period reduces, while the other parameters of PC are  $constant^{[15]}$ . We can achieve small crystal lattice periods by controlling software and improving spatial resolution by selecting quick scan speed and low laser power. So it is possible to fabricate PC whose PBG ranges is from near infrared to visible light wave band. Theoretically, transmission spectrum should be a quite fair curve and a 100% transmission rate except near the band gap. Whereas, experimental results indicates that biggish noise occurs on both sides of band gap, and transmission rate is around 80% on the right and 65% on the left. The noise is related to the surface roughness of solidified rods, which has different scattering influence on different wavelength light. Meanwhile, Rayleigh scattering is dependent on the light wavelength, which leads to the different transmission rate on both sides of the PBG. We can also conclude that the transmission ratio is still high in mid-gap, that is, there is no complete PBG for photosensitive resin with refractive index of 1.55. Generally speaking, the PC has full PBG when the ratio of two refractive indices is above  $2.7^{[27]}$ . Therefore, in order to obtain PCs with full PBG, template method is being investigated currently by two-step process. Firstly, the PC will be infiltrated with  $SiO_2$ , followed by removing of the ORMOCER. Secondly, silicon will be infiltrated and the SiO<sub>2</sub> etched away. Under these conditions, the 3D PCs allow for full PBG in infrared wavelengths.

In summary, characteristic parameters of direct femto second laser writing were introduced, such as the laser intensity and scan speed. High-quality 3D log pile PC with mid-infrared PBG was fabricated fast by direct femtosecond laser writing in photosensitive resin and error analyzes were investigated with the Image Pro Plus. Meanwhile, we experimentally verified that the centre of PBG diminishes as the crystal lattice period reduces. We can achieve small crystal lattice periods and surface roughness by improving spatial resolution and post processing techniques. Under the conditions of fastfabricating, desired defects such as waveguide and microcavity can be introduced in these structures easily to realize the photonic circuits. All these indicate that direct femtosecond laser writing is not only an effective method to fabricate PCs at present but also a good foundation to develop the applications of PCs.

This work was supported by the National "863" Project of China (No. 2006AA04Z307), Foundation for the Author of National Excellent Doctoral Dissertation

of China (No. 2006039), the National Natural Science Foundation of China (No. 50775140), Natural Science Foundation of Jiangsu Province (No. BK2006507), and Jiangsu Province Research Innovation Program of College Graduate (No. CX07B\_086z). M. Zhou is the author to whom the correspondence should be addressed, his e-mail address is zm\_laser@126.com.

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