

# Fabrication of Photonic Crystal Rod by Hot Vacuum Stacking Method Using Multicomponent Glass

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## ホットバキューム法による多成分ガラスフォトニッククリスタルロッドの作製

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We have fabricated a photonic crystal rod (PCR), which is a novel optical connecting component with a two-dimensional photonic crystal structure, by drawing a preform made of a multicomponent alkali-borosilicate glass. A hot vacuum (725°C, 10 hPa) stacking method was developed for the fabrication of the preform, which enables self-organization of the inner structure of the preform. PCRs with sub-micrometer dimensional accuracy were successfully fabricated by drawing the preform at 820°C, which is a much lower temperature than that used for the drawing of silica glass. The PCRs showed single-mode optical propagation at both wavelengths of 633 and 1550 nm with mode field diameters (MFDs) of 23.1 and 33.4  $\mu\text{m}$ , respectively. The bending strength of the PCR reached 850 MPa after ion-exchange treatment. PCR is expected to be a candidate for precision, high-strength, endlessly single-mode novel optical connecting components with large MFDs.

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### 1. Introduction

In recent years, photonic crystal fibers (PCFs) have attracted much attention in the field of optical telecommunication, because of their unique optical propagation characteristics. PCFs are optical fibers with fine through-holes regularly arranged along their longitudinal axis, at the center of which light propagates on the basis of the total reflection principle.<sup>1)–4)</sup> It has been reported that many unique optical characteristics of PCFs, such as endless single-mode propagation and very large or small mode field diameters (MFDs), can be achieved by the controlling through-hole arrangement.<sup>1),5),6)</sup> In general, PCFs are fabricated by a preform drawing process. Since long-distance optical transmission is an interesting field of application of PCFs because of their unique dispersion or nonlinear optical properties, so far, silica glass with low transmission loss has been used as the base glass of PCFs.<sup>7),8)</sup> In the production of silica glass PCFs, stacking of silica glass tubes or drilling of silica glass ingots is usually employed for preform fabrication.<sup>3)–5)</sup> However, the efficiency of these processes is insufficient for the commercial production of PCFs. In addition, the high drawing temperature of silica glass ( $\sim 1900^\circ\text{C}$ ) gives rise to other difficulties in process control, leading to a high production cost of silica glass PCFs.

In this paper, in order to realize photonic crystal waveguides with high production efficiency, we demonstrate a novel preform fabrication method based on the hot vacuum stacking of multicomponent glass, and propose a new application of the waveguide as an optical connecting component. In optical connecting components, the optical loss of the base glass itself is less significant than in the case of long-distance optical transmission media, because of their much shorter optical path length (several millimeters). Therefore, in such cases, multicomponent glass can be used as the base glass instead of

silica glass. The use of multicomponent glass will make the fabrication of the preform and the waveguides much easier than in the case of using silica glass because of the lower process temperature and the wide selectivity of chemical composition. The base glass for the optical connecting components should have sufficient mechanical durability and a refractive index close to that of silica glass optical fibers, as well as thermal stability in high-temperature processes. We employed an alkali-borosilicate ( $\text{Na}_2\text{O}-\text{B}_2\text{O}_3-\text{SiO}_2$ ) glass as the base glass, and developed a new method for fabricating the preform, utilizing the thermal stability and low-temperature formability of that base glass. In this paper, we describe a hot vacuum process for preform fabrication that induces a self-organization effect on the formation of the inner structure of the preform, and the properties of a newly developed rod-like optical connecting component with a photonic crystal structure, the photonic crystal rod (PCR).

### 2. Experimental

The glass used in the present study contains 7% of  $\text{Na}_2\text{O}$ , 12% of  $\text{B}_2\text{O}_3$ , and 75% of  $\text{SiO}_2$  in molar ratio and small amounts of  $\text{K}_2\text{O}$ ,  $\text{CaO}$  and  $\text{Al}_2\text{O}_3$ . A mixture of the raw materials was melted in a platinum pot at  $1600^\circ\text{C}$  for 50 h in an electric furnace to form a homogeneous glass. The total iron content in the raw materials was controlled to be less than 10 ppm in order to suppress the optical absorption in the near-infrared region caused by  $\text{Fe}^{2+}$  ions. The transmittance of the specimens in the wavelength region between 200 to 1600 nm was measured using a spectrometer (Model: Shimadzu UV-3100PC). We calculated the optical loss of the base glass from the transmittances of polished specimens with different thicknesses (3, 5 and 10 mm) based on the Lambert-Beer law. The refractive index of the base glass was measured using a refrac-

tometer (Model: KPR-200, Kalnew). The softening temperature of the base glass was measured under the regulation of American society for testing and material (C338-73).

For preform fabrication, first, glass capillaries (inner diameter: 0.125 mm), a solid glass rod (diameter: 1.0 mm) and an outer glass tube (outer diameter: 50 mm) made of the alkali-borosilicate base glass were prepared. Both ends of the capillaries were sealed by heating with a gas burner in advance. The glass capillaries were then randomly loaded into the outer tube, and the solid rod was loaded into the center of the outer tube, and then the rod was fixed using an organic adhesive. The inside of the outer tube was evacuated until the pressure reached 10 hPa and the tube was subsequently heated at 725°C for 1 h in an electric furnace. After annealing to room temperature, the outer surface of the obtained preform was machined into a cylindrical shape with an accuracy of  $\pm 0.005$  mm for the diameter.

The PCR was drawn from the preform at 820°C by a conventional drawing process. To investigate the optical guidance properties of the PCR, we lead He-Ne laser light (wavelength  $\lambda = 633$  nm) and amplified spontaneous emission (ASE) light (1550 nm) to one end of a PCR sample 40 mm in length, using an objective lens. Near-field patterns (NFPs) of the guided mode at the output end of the PCR were observed using an optical analysis system (Model: LEPAS-11, Hamamatsu Photonics). A simulation of the optical propagation was also carried out by the beam propagation method (BPM, Model: BeamPROP, RSoft Design Group). The following parameters were used for the MFD calculation: period of air holes: 17; incident light diameter: 22.1  $\mu\text{m}$ ; optical length: 1024  $\mu\text{m}$ . In order to realize the high bending strength required for optical connecting components, the PCR was strengthened by an ion exchange process, in which the PCR was immersed in molten  $\text{KNO}_3$  at 380°C for 20 h. The bending strength was measured by the three-point bending method for 15 specimens.

### 3. Results and discussion

#### Preform fabrication

The base glass showed a softening temperature of 785°C, which is much lower than that of silica glass ( $\sim 1800^\circ\text{C}$ ), and is also lower than the crystallization temperature of this glass (900°C). Therefore, this glass could be thermally processed at a much lower temperature (725°C) than silica glass without any devitrification problems. The base glass showed transmission losses of 0.002 dB/cm and 0.06 dB/cm at 1550 nm and 1400 nm, which were sufficiently small for the use as optical connecting components. The reflection loss at 1550 nm was calculated to be -40 dB from the refractive indices of the base glass (1.475) and the silica glass (1.444).<sup>9)</sup> This value satisfies the requirement of reflection loss for conventional optical connectors.<sup>10)</sup> This small reflection loss was achieved due to the similarity of the refractive indices of the base glass and silica glass.

Figure 1 shows a schematic drawing of the hot vacuum process that induces the self-organization effect on the formation of the inner structure. A cross-sectional view of the fabricated preform is also shown in the figure. First, when the assembly (Fig. 1(a)) is held at 725°C, the outer glass tube softens and shrinks. Subsequently, the capillaries inside automatically stack on each other in such a way as to have minimum interface energy, i.e., to form an equilateral triangle arrangement (Fig. 1(b)). Then, the capillaries and the solid rod fuse with each other without an air gap, because residual air between the capillaries or the rod is evacuated (Fig. 1(c)). Since both ends of the capillaries are sealed in advance, the in-

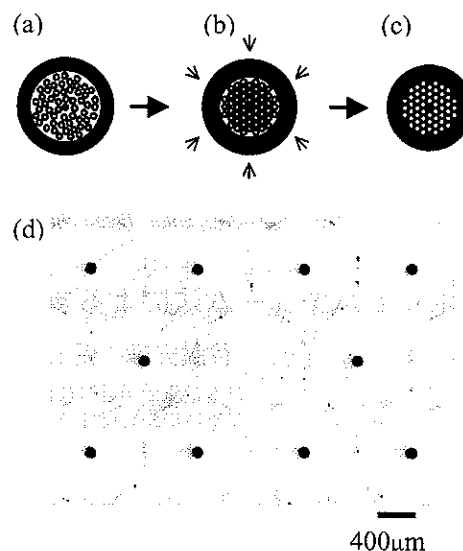


Fig. 1. Schematic diagram of the hot vacuum process. (a) Random loading of capillaries. (b) Shrinkage of outer tube and self-organization of capillaries (725°C, 10 hPa). (c) Fusion and close packing. (d) Cross-sectional view of the obtained preform.

ner diameter of the capillaries remains constant throughout the process. In Fig. 1(d), one can see that the capillaries are regularly organized in an equilateral triangle structure in spite of the random loading during the first stage (Fig. 1(a)). This spontaneous organization effect eliminates the requirement of the precise prearrangement of the capillaries by machining capillaries into a hexagonal shape and then piling them up, which has been commonly employed in conventional preform fabrication processes. This self-organization effect is a great advantage of this method. The dimensional accuracy of the diameter and pitch of the through-holes in the preform were within 4  $\mu\text{m}$  and 30  $\mu\text{m}$ , respectively. The stacking process at such a low temperature (725°C) has become feasible by employing the thermally stable, low-viscosity sodium borosilicate base glass. If the silica glass is applied to this process, it would require a very high temperature ( $\sim 1800^\circ\text{C}$ ), and would cause devitrification problems.

#### PCR

Figure 2 shows a cross section of the PCR fabricated by drawing the preform. The figure indicates that the PCR possesses an equilateral triangle structure, maintaining the same structure as that of the preform. The interfaces between the glass capillaries or the rod, which were observed in Fig. 1, were not observed in the PCR due to homogenization of the glass at the interface areas during drawing. The PCR had an outer diameter of 1.249 mm with the accuracy of 1.0  $\mu\text{m}$ , which satisfies the dimensional requirements for optical fiber connectors. The hole diameter ( $d$ ) and the pitch ( $\Lambda$ ) were  $3.9 \pm 0.2$   $\mu\text{m}$  and  $22.1 \pm 0.5$   $\mu\text{m}$ , respectively, and the eccentricity of the core was less than 3  $\mu\text{m}$ . These dimensions of the PCR are sufficiently accurate for the occurrence of photonic crystal functions.

Birks et al.<sup>11)</sup> reported that the photonic crystal fiber with a  $d/\Lambda$  ratio of less than 0.4 shows single-mode optical propagation at any wavelength (endless single-mode propagation). Since the PCR fabricated in this study has the  $d/\Lambda$  ratio of 0.18, endless single-mode propagation was expected. Figure 3 shows observed NFP and optical intensity profiles of the light emitted from the end of PCR with the length of 40 mm. In Figs. 3(a) and 3(b), one can see that the light is confined in

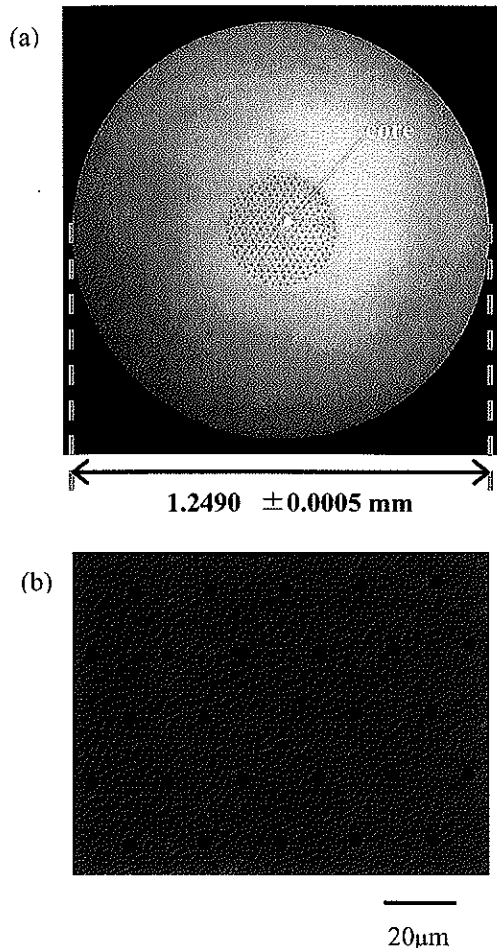


Fig. 2. (a) Optical microscope image of cross section of the PCR. (b) Laser microscope image of the core portion of the PCR.

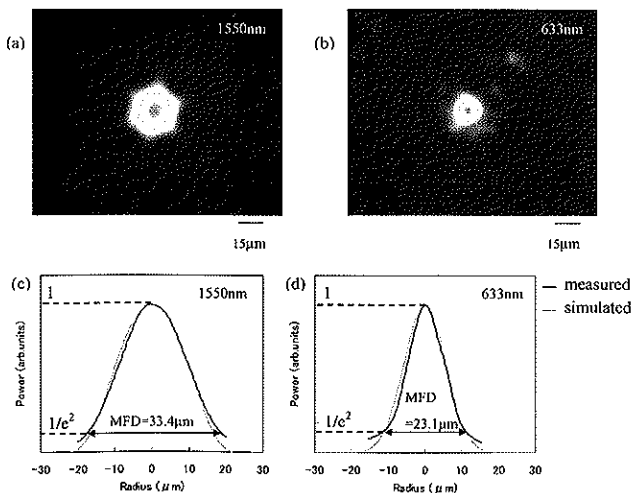


Fig. 3. Optical power distribution of the light emitted from the PCR: (a) NFP at 1550 nm, (b) NFP at 633 nm, (c) Power intensity profile at 1550 nm, (d) Power intensity profile at 633 nm.

the core of the PCR in both cases of wavelengths of 633 and 1550 nm. The optical intensity profiles showed single peak distributions, and were in fair agreement with the Gaussian fits in both cases. These results indicate that single-mode propagation was achieved at these wavelengths; namely, endless single-

mode propagation was realized. The measured MFDs were 23.1 and 33.4  $\mu\text{m}$  at the wavelengths of 633 and 1550 nm, as shown in Fig. 3. These values were in good agreement with the simulated values of 22.6 and 33.2  $\mu\text{m}$ , respectively. The MFD at the wavelength of 1550 nm is approximately three times greater than that of normal single-mode optical fibers.

The average bending strengths of the PCR before and after the ion exchange treatment were 540 and 850 MPa, respectively. The strength after the treatment is comparable to that of commercial optical connector ferrules.<sup>12)</sup> The marked increase in the bending strength realized after the treatment is also due to the use of the sodium borosilicate base glass that contains a large amount of sodium ions (7 mol%) that are replaced by potassium ions during the treatment, which leads to surface compressive stress.

#### 4. Conclusion

A novel rod-type photonic crystal waveguide, PCR, was fabricated using a sodium borosilicate base glass. A hot vacuum stacking method that induces a self-organization effect on the formation of the inner structure was developed for the preform fabrication. This method greatly simplifies the preform fabrication process, eliminating the requirement of the premachining and stacking of glass capillaries. The obtained PCR showed single-mode optical propagation with large MFDs at wavelengths of 633 and 1550 nm. The PCR showed excellent dimensional accuracy with sub-micrometer order and a high bending strength of 850 MPa. The PCR is expected to become a novel optical connecting component that provides endless single-mode propagation and large MFDs with high production efficiency.

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