Original Paper

Fabrication of large preforms for low-loss single-mode optical fibers by a hybridized process

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A hybridized fabrication process for single-mode optical fibers has been studied. The process consists of direct overcladding a VAD-derived core rod with a porous body made from commercial silica powder by means of the cold isostatic pressing technique. By using a mixture of main silica powder with an average particle diameter of 10 μm and 1 wt% of fine silica powder with an average particle diameter of 0.04 μm and a core rod having a core/cladding diameter ratio of 1/2.4 as starting materials, large preforms for 300 km fibers were fabricated, which were drawn into fibers with attenuation losses of 0.350 dB/km at 1.31 μm and 0.232 dB/km at 1.55 μm. In the process, a special rubber mold with a structure in which both ends of the core rod are pressed by hydrostatic pressure makes the fabrication of large hybridized soot preforms possible, plastic deformation of the porous body during degreasing being remarkably reduced by using a suitable content of glycerol.

Herstellung großer Vorformen für verlustarme optische Einmodenfasern durch ein Hybridverfahren

Es wurde ein Hybridverfahren zur Herstellung von optischen Einmodenfasern untersucht. Das Verfahren besteht aus dem direkten Ummanteln eines nach der VAD-Methode erzeugten Kernstabes mit einem porösen Körper aus handelsüblichem Kieselglaspulver nach der Methode des isostatischen Kaltpressens. Unter Verwendung einer Mischung, bestehend hauptsächlich aus Kieselglaspulver mit einer durchschnittlichen Korngröße von 10 μm und einem Massenanteil von 1 % feinem Kieselglaspulver mit einer durchschnittlichen Korngröße von 0,04 μm, und eines Kernstabes mit einem Kern/Mantel-Durchmesserverhältnis von 1/2,4 als Ausgangsmaterialien wurden große Vorformen für 300 km lange Fasern hergestellt, die zu Fasern mit Dämpfungsverlusten von 0,350 dB/km bei einer Cutoff-Wellenlänge von 1,31 μm und 0,232 dB/km bei einer solchen von 1,55 μm ausgezogen wurden. Bei diesem Verfahren ermöglicht eine spezielle Gummiform, in die mit hydrostatischem Druck beide Enden des Kernstabes gepreßt werden, die Herstellung großer Hybrid-Aufdampfvorformen, wobei die plastische Verformung des porösen Körpers während des Ausbrennens des organischen Bindemittels deutlich reduziert wird, wenn ein entsprechender Glycerinanteil verwendet wird.

1. Introduction

Silica optical fibers for optical telecommunications have been produced generally by Chemical Vapor Deposition (CVD) techniques such as Vapor phase Axial Deposition (VAD), Modified Chemical Vapor Deposition (MCVD) and Outside Vapor Deposition (OVD).

Recently new processes have been investigated for the fabrication of optical fibers, especially single-mode optical fibers. One is the all-powder-forming process, for example the Mechanically Shaped Preforms (MSP) method [1 and 2], in which the core and the cladding are made simultaneously. Although the minimum of attenuation in single-mode fibers fabricated by MSP was reduced to 0.27 dB/km at 1550 nm and 0.49 dB/km at 1310 nm, it is still considerably higher than with CVD fibers as Dorn et al. mentioned [2]. In addition, they also identified reboiling during preform stretching and fiber drawing as the last remaining problem of the process. The other is the hybridized process that consists of overcladding a core rod made by the CVD method with a silica glass produced from commercial silica powder by sintering. Using a CVD-derived core rod with low loss, this process may be able to make low-loss fibers.

Received December 21, 1995, revised manuscript April 4, 1996.

The hybridized processes reported so far consist of rod-in-tubing a CVD-derived core rod with either a tubular porous body made by the colloidal sol-gel technique [3] or a tubular porous body made from silica powder by the compression technique [4]. However, the two processes of rod-in-tube type could not make low-loss fibers that compete with CVD fibers although fibers with about 0.5 and with 0.392 dB/km at 1300 nm were fabricated by the colloidal sol-gel and the compression method, respectively.

Lower-loss fibers may be made successfully by a hybridized process if the waveguide imperfection loss is reduced. Thus, the authors tried to develop a hybridized process of direct type in which a CVD-derived core rod is directly covered by a porous body made from silica powder.

Another big target for the new fabrication process is to make large preforms aiming at low cost. Though Dorn et al. [2] demonstrated the fabrication of a preform corresponding to 150 km of fiber by the MSP method, a larger preform is more suitable for low-cost fiber fabri-

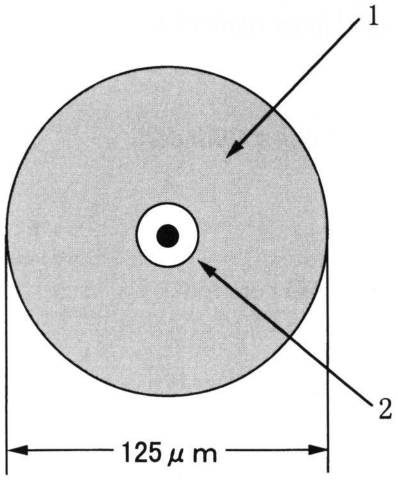

Figure 1. Cross-section of the optical fiber made by the hybridized process. The fiber consists of a core rod portion (1) with the core and a part of cladding made by the VAD method and an overcladding portion (2) made from commercial silica powder.

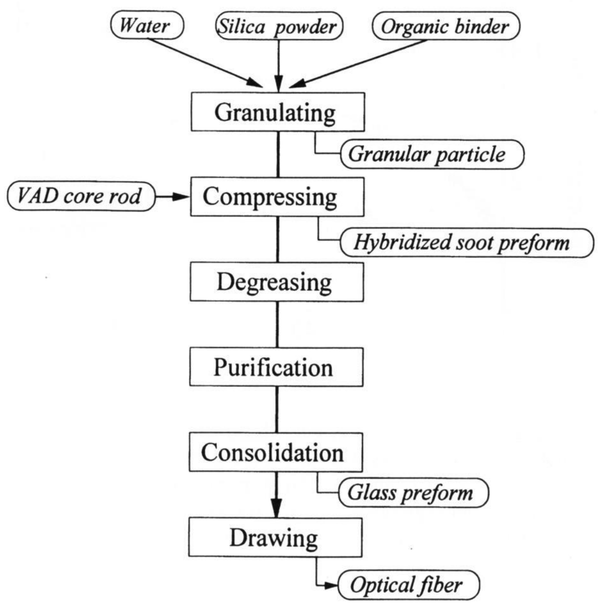

Figure 2. Fabrication stages for the direct hybridized process using compression forming.

cation. Against this background, the authors have studied a direct hybridized process using a compression technique for shape forming and have successfully made low-loss fibers drawn from large 300 km preforms. In this paper, some of these studies of making large preforms to be drawn into low-loss fibers are described.

2. Concept of the direct hybridized process for optical fibers

Figure 1 shows a cross-section of the optical fiber made by the hybridized process. The fiber consists of a core rod portion and an overcladding portion. The core rod portion consisting of the core and a part of the cladding is made by the VAD method and the overcladding portion is made from commercial silica powder by the process described in the following.

Figure 2 shows the fabrication stages of the direct hybridized process using the compression forming. The starting materials are commercial silica powder and a VAD-derived core rod. A core rod having a core/cladding diameter ratio of about 1/3 was used. Using the core rod, a single-mode optical fiber with 5% VAD-derived and 95% powder-derived silica in volume is made. The first step of the fabrication is granulating, in which granular silica particles are prepared from a slurry-containing silica powder, organic binder and water using the spray dryer technique. The granular particle has such a large fluidity that it is easily fed into a mold die cavity. The fluidity of particle is a very important factor to obtain a porous body with high homogeneity leading to low-loss and bubble-free glass. The next step is soot preform forming by Cold Isostatic Pressing (CIP). In this step, a hybridized soot preform consisting of a VAD core rod at the center and a porous glass body made from silica powder around the VAD rod is prepared. Compacting is done in a high-pressure vessel of the conventional CIP equipment. The following steps are degreasing, purification and consolidation. Degreasing means a step to remove the organic binder that is added at the first fabrication step. The organic binder is burned out from the porous body at high temperature in an air flow. In the purification, the hybridized soot preform is purified at high temperature in a helium gas atmosphere containing chlorine as MacChesney et al. [5] and Clasen [6] described. In this step, impurities such as OH radicals and transition metal elements are removed from the porous body. Consolidation is carried out in a helium gas atmosphere at high temperature to make a glass preform from the purified soot preform. Then, the glass preform prepared as mentioned above is drawn into an optical fiber.

3. Hybridized soot preform forming

In a previous paper [4], a method to make a tubular porous body from silica powder by compression forming is reported. At that time it was not possible to make any hybridized soot preform because the core rod broke during compression. In that study, it was found that breakage of the core rod occurred due to a stretching force that came from compressed silica powder during compression.

In the present paper the authors tried to use a rubber mold which is shown in figure 3. With this mold, both ends of the core rod are pressed onto the mold by the hydrostatic pressure during compression, and the stretching force to the core rod is likely to be compensated by the pressure on both ends, so that breakage is prevented. In fact, the use of the rubber mold makes a large preform possible. The preform is 85 mm in diameter and 1 m in length. It is turned into the transparent glass preform of 70 mm in diameter and 1 m in length which is drawn into about 300 km fibers.

4. Deformation of the hybridized soot preform

To make the porous body from silica powder, a binder to combine silica particles with each other is required. In this study, an organic binder such as a mixture of polyvinyl alcohol (PVA) and glycerol was used. As a porous body of soot preform containing organic binder before degreasing is elastic, its shape changes by the deadweight during the degreasing step. The deformation causes gaps between a core rod and a porous body and the gaps create bubbles in glass when consolidated. The problem is more serious in the case of larger soot preforms.

Figure 4 shows deformations of a soot preform which is laid horizontally on two wedges. Curve 1 shows the result with a soot preform prepared from granular particles containing 1.6 wt% PVA and 1.2 wt% glycerol. The deformation of the soot preform is as large as 0.4 mm at 75 min. There are some points, as at about 60 min, at which the deformation rate increases discontinuously. It is supposed that cracking in the soot preform is taking place at these discontinuous points of deformation rate. Curve 2 in figure 4 shows the result with a soot preform which is prepared from granular particles containing 1.6 wt% PVA and 1.0 wt% glycerol. As shown in this figure, the deformation of the soot preform is reduced to 0.15 mm at 75 min. The result shows that soot preforms with little deformation can be made by improving the binder.

5. Silica powder

Silica powder has impurities like OH radicals and transition metal elements, for example iron, chromium and nickel. To remove these impurities, the soot preform is purified by chlorine gas. In the purification process, chlorine diffuses into pores of the porous body and reacts with impurities. Thus, large pores are desirable for the purpose of purification which are more easily formed in the porous body fabricated from large silica particles.

In the other new fabrication processes reported, fused silica is used for the MSP method or the colloidal sol-gel method, and fine silica powder having a particle diameter of 0.3 μm is used for powder sintering of rod-in-tube method. In this study, it was tried to use large particle silica powder having an average particle diameter of 10 μm.

However, the strength of the porous body after the degreasing step decreases with increasing the diameter of silica particles. To improve the strength, fine silica powder was added having an average particle diameter of 0.04 μm to the 10 μm silica powder.

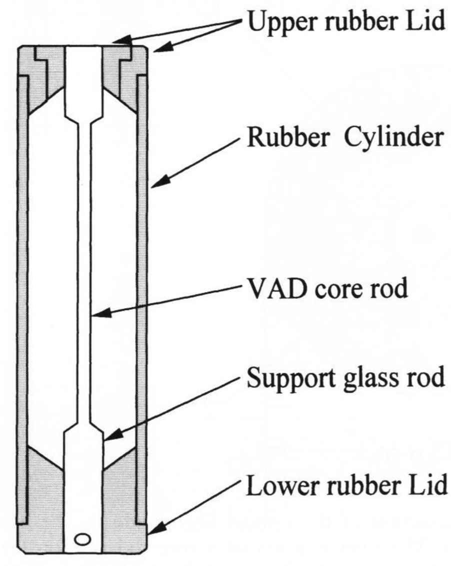

Figure 3. Rubber mold.

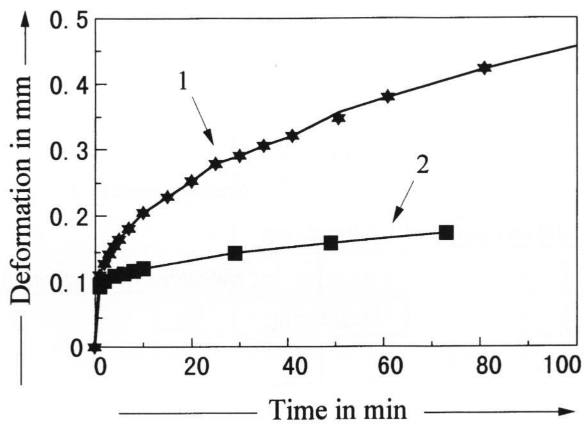

Figure 4. Deformations of a hybridized soot preform laid horizontally on two wedges. Curve 1 shows the result with a soot preform prepared from granular particles containing 1.6 wt% PVA and 1.2 wt% glycerol. Curve 2 shows the result with a soot preform prepared from granular particles containing 1.6 wt% PVA and 1.0 wt% glycerol.

Figure 5 shows the compression strength of a disc sample (diameter: 19 mm, thickness: 10 mm) of the porous body after the degreasing step. As shown in the figure, the addition of fine silica powder improves the strength of the porous body.

As already mentioned, the pore diameter is important for easy purification. Figure 6 shows pore size distributions measured on the same porous bodies as shown in figure 5. In figure 6, only a little increase in average pore diameter is found for 1 wt% addition of fine silica, but a small amount of generation of very fine pores is found for 5 or 15 wt% addition. The fine pore has a diameter of 0.05 μm and is likely to be formed from the fine silica powder. Because it is difficult to purify the porous body with 0.05 μm pores, the authors determined to use 1 wt% addition of fine silica.

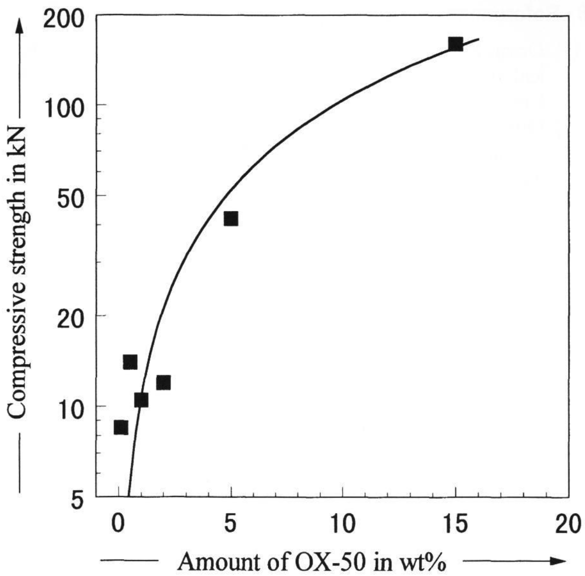

Figure 5. Compression strength of a disc sample (diameter: 19 and thickness: 10 mm) of the porous body after the degreasing step.

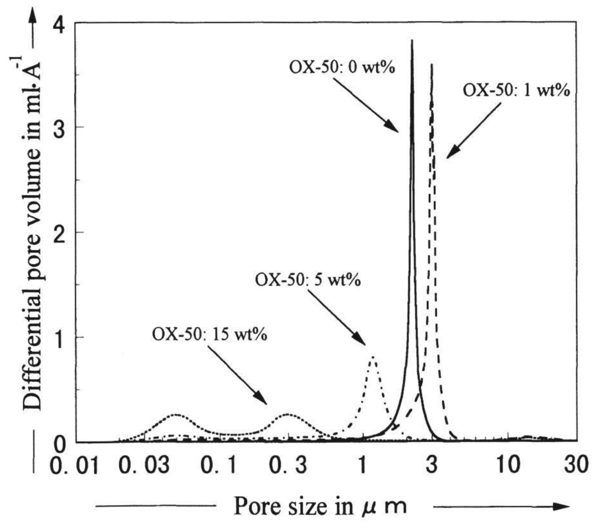

Figure 6. Distributions of pore size measured on the same porous bodies as in figure 5.

6. Fiber fabrication

Single-mode optical fibers shown in figure 1 were fabricated by the process schematically shown in figure 2. The silica powder having an average particle diameter of 10 μm was mixed with 1 wt% fine silica having an average particle diameter of 0.04 μm and the mixture was used for the starting silica powder. A core rod having a core/cladding diameter ratio of 1/2.4 and a refractive index difference of 0.393 % was used.

The first step of the fabrication is granulating where 100 weight parts of 10 μm silica powder, 1.0 weight part of the fine silica, 1.6 weight parts of PVA and 1.0 weight part of glycerol were mixed with 50 weight parts of pure water. The granular silica particles were made from the mixture so-called slurry by the spray dryer method. The granular particles consisting mainly of silica particles had an average diameter of about 100 μm. Next, the hybridized soot preform was formed by CIP using a rubber mold shown in figure 3. In this step, the core rod was placed at the center of the mold, and the granular particles were packed into the space between the mold and the rod. Then, the rubber mold filled with the granular particles was pressed from outside at 98 MPa of hydrostatic pressure in a high-pressure vessel of the conventional CIP equipment. A large hybridized soot preform of 85 mm in diameter and 1 m in length was fabricated.

The following steps were degreasing, purification and consolidation. For degreasing, the hybridized soot preform was treated for 5 h at 500 °C in a dry air flow. For purification, it was treated at 1200 °C in helium gas containing 1 vol.% chlorine. Consolidation was carried out in helium gas at 1600 °C to obtain a glass preform of 70 mm in diameter and 1 m in length. Then, the glass preform was drawn into an optical fiber of about 300 km.

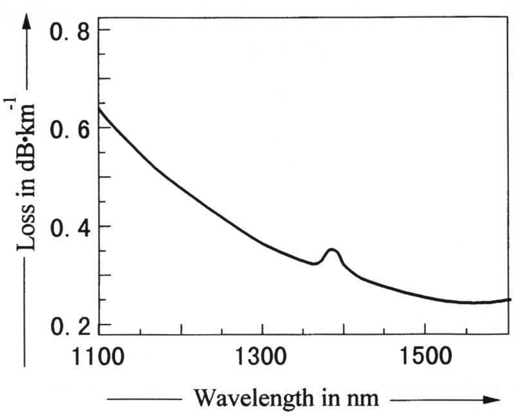

Figure 7. Loss spectrum of a single-mode optical fiber having a cutoff wavelength of 1.28 μm. (Fiber length: 30 km.)

Figure 7 shows a loss spectrum of the single-mode optical fiber having a cutoff wavelength of 1.28 μm. The losses were 0.350 dB/km at 1.31 and 0.232 dB/km at 1.55 μm. The peak height at 1.39 μm due to OH radicals was 0.037 dB/km. Further, the loss was confirmed to be uniform along the fiber length by Optical Time Domain Reflectometry (OTDR) measurement.

7. Conclusions

A hybridized process consisting of overcladding a VAD-derived core rod with a porous body made from commercial silica powder by the CIP technique has been studied. The results are summarized as follows.

a) A rubber mold with a structure in which both ends of the core rod are pressed by hydrostatic pressure makes a large hybridized soot preform possible.

b) Deformations of the porous body during degreasing are remarkably reduced by using a suitable content of glycerol.

c) The porous body of silica powder was strengthened by adding a small amount of fine silica powder into 10 μm silica powder. But fine pore which is not suitable for purification was generated at a high-level addition.

d) Large preforms of 300 km fibers were fabricated by the developed process.

e) Attenuation losses of fibers were 0.350 dB/km at 1.31 and 0.232 dB/km at 1.55 μm.

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